Electronic Supplementary Material (ESI) for Organic Chemistry Frontiers.
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Part I Experimental Part

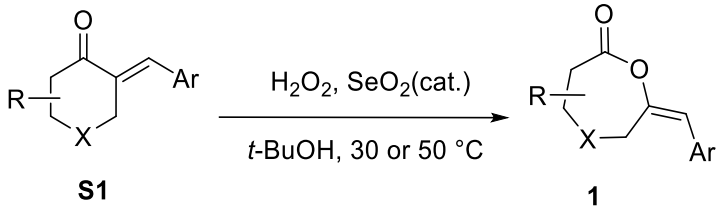
1. General information

Unless otherwise indicated, all reactions were carried out under N_2 protection with magnetic stirring. Anhydrous THF, toluene and 1,4-dioxane were distilled from sodium and benzophenone. Anhydrous CH_3CN and CH_2Cl_2 were distilled from CaH_2. Chiral triazolium salts **A-E** were synthesized according to literatures[1]. Column chromatography was performed on silica gel 200~300 mesh. All ^{1}H NMR (400 MHz), ^{13}C NMR (101 MHz) ;^{1}H NMR (500 MHz), ^{13}C NMR (126 MHz); ^{19}F NMR (376 MHz); ^{19}F NMR (658 MHz) spectra were recorded on a Bruker-DMX 400, 500 and 700 spectrometers in $CDCl_3$, with tetramethylsilane as an internal standard and reported in parts per million (ppm, δ). ^{1}H NMR and ^{19}F NMR spectroscopy splitting patterns were designated as singlet (s), doublet (d), triplet (t), quartet (q). Splitting patterns that could not be interpreted or easily visualized were designated as multiplet (m) or broad (br). Infrared spectra were recorded on a Nicolet 6700 spectrophotometer and reported as wave number (cm^{-1}). Optical rotations were

measured on Rudolph-Autop VI digital polarimeter operating at the sodium D line with a 100 mm path cell, and are reported as follows: $[\alpha]_D^{26}$ (concentration (g/100 mL), solvent).

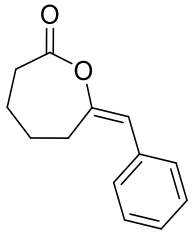
2. Preparation of enol lactones.

2.1 Preparation of *exo*-enol lactones



General procedure[2]: To a mixture of cycloketone **S1**[3] (1.0 equiv), SeO_2 (0.05 equiv) in *t*-BuOH (3mL/mmol) was added aqueous 30% H_2O_2 (3.0 equiv) at 30 °C. The reaction mixture was stirred at 30 °C or 50 °C in open air. After consumption of the starting material (monitored by TLC, typically in 24-36 h), the reaction mixture was quenched by addition of sat. Na_2SO_3. The solvent was removed under reduced pressure. To the residue, water was added and extracted with DCM for three times. The combined organic extracts were dried over anhydrous $MgSO_4$ and concentrated under reduced pressure. Purification by flash column chromatography afforded the desired *exo*-enol lactone **1**.

Characterization Data of *exo*-enol lactones **1**



1a (QY-299)

(*E*)-7-(3-chlorobenzylidene)oxepan-2-one

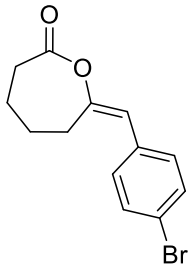
0.95 g, 47% yield. White solid, m.p. 65-67 °C. R_f = 0.19 (petroleum ether/ethyl acetate, 10:1).

^{1}H NMR (500 MHz, $CDCl_3$) δ 7.37 (t, *J* = 7.5 Hz, 2H), 7.30 – 7.26 (m, 3H), 6.45 (s, 1H), 2.69 – 2.67 (m, 2H), 2.60 – 2.58 (m, 2H), 1.94 – 1.90 (m, 4H).

^{13}C NMR (126 MHz, $CDCl_3$) δ 173.1, 151.7, 134.4, 128.6, 128.4, 127.4, 119.2, 33.8, 29.7, 27.8, 23.2.

IR (KBr) v 2940, 1756, 1658, 1345, 1210, 1167, 1114, 1007, 786, 691.

HRMS (APCI) *m/z*: Calcd for $C_{13}H_{15}O_2$ ($[M+H]^+$) 203.1067, found 203.1068.



1b (QY-437-2)

(*E*)-7-(4-bromobenzylidene)oxepan-2-one

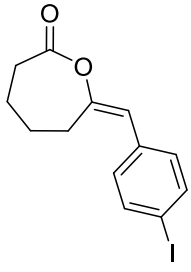
1.48 g, 81% yield. White solid, m.p. 105-106 ºC. R_f = 0.19 (petroleum ether/ethyl acetate, 10:1).

^{1}H NMR (500 MHz, $CDCl_3$) δ 7.49 (d, *J* = 8.5 Hz, 2H), 7.13 (d, *J* = 8.4 Hz, 2H), 6.37 (s, 1H), 2.68 – 2.66 (m, 2H), 2.57 – 2.55 (m, 2H), 1.92 – 1.90 (m, 4H).

^{13}C NMR (126 MHz, $CDCl_3$) δ 172.7, 152.2, 133.3, 131.8, 130.0, 121.4, 118.1, 33.8, 29.7, 27.7, 23.2.

IR (KBr) v 2946, 1750, 1659, 1166, 1117, 1009, 893, 861, 516.

HRMS (APCI) *m/z*: Calcd for $C_{13}H_{14}O_2Br$ ($[M+H]^+$) 281.0172, found 281.0172.



1c (QY-409-2)

(*E*)-7-(4-iodobenzylidene)oxepan-2-one

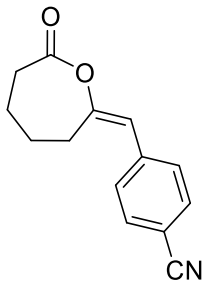
0.82 g, 50% yield. Yellowish solid, m.p. 125-126 ºC. R_f = 0.17 (petroleum ether/ethyl acetate, 10:1).

^{1}H NMR (500 MHz, $CDCl_3$) δ 7.69 (d, *J* = 8.3 Hz, 2H), 7.00 (d, *J* = 8.2 Hz, 2H), 6.35 (s, 1H), 2.68 – 2.66 (m, 2H), 2.57 – 2.55 (m, 2H), 1.91 – 1.90 (m, 4H).

^{13}C NMR (126 MHz, $CDCl_3$) δ 172.7, 152.3, 137.7, 133.9, 130.2, 118.2, 92.8, 33.8, 29.7, 27.7, 23.1.

IR (KBr) v 2939, 1746, 1661, 1164, 1115, 1006, 893, 814, 517.

HRMS (APCI) *m/z*: Calcd for $C_{13}H_{14}O_2I$ ($[M+H]^+$) 329.0033, found 329.0034.



1d (QY-419-2)

(*E*)-4-((7-oxooxepan-2-ylidene)methyl)benzonitrile

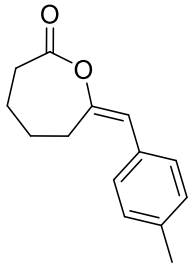
1.34 g, 57% yield. White solid, m.p. 168-169 ºC. R_f = 0.33 (petroleum ether/ethyl acetate, 3:1).

^{1}H NMR (500 MHz, $CDCl_3$) δ 7.66 (d, *J* = 8.2 Hz, 2H), 7.36 (d, *J* = 8.2 Hz, 2H), 6.45 (s, 1H), 2.69 – 2.66 (m, 2H), 2.61 – 2.58 (m, 2H), 1.94 – 1.93 (m, 4H).

^{13}C NMR (126 MHz, $CDCl_3$) δ 172.1, 154.1, 139.3, 132.4, 129.0, 118.6, 117.8, 111.0, 33.8, 29.9, 27.6, 23.1.

IR (KBr) v 2950, 2224, 1748, 1213, 1162, 1107, 1003, 896, 827.

HRMS (APCI) *m/z*: Calcd for $C_{14}H_{14}O_2N$ ($[M+H]^+$) 228.1019, found 228.1020.



1e (QY-374)

(*E*)-7-(4-methylbenzylidene)oxepan-2-one

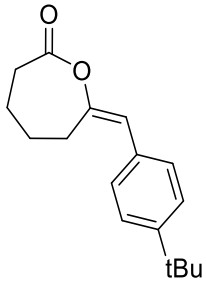
0.82 g, 63% yield. White solid, m.p. 98-99 ºC. R_f = 0.21 (petroleum ether/ethyl acetate, 10:1).

^{1}H NMR (400 MHz, $CDCl_3$) δ 7.19 – 7.14 (m, 4H), 6.40 (s, 1H), 2.71 – 2.63 (m, 2H), 2.60 – 2.57 (m, 2H), 2.35 (s, 3H), 1.93 – 1.86 (m, 4H).

^{13}C NMR (101 MHz, $CDCl_3$) δ 173.3, 151.1, 137.3, 131.4, 129.3, 128.3, 119.1, 33.8, 29.7, 27.8, 23.3, 21.2.

IR (KBr) v 2938, 1742, 1666, 1162, 1111, 1009, 865, 808, 526.

HRMS (APCI) *m/z*: Calcd for $C_{14}H_{17}O_2$ ($[M+H]^+$) 217.1223, found 217.1225.



1f (QY-452-2)

(*E*)-7-(4-(tert-butyl)benzylidene)oxepan-2-one

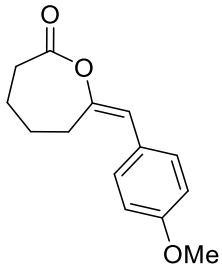
1.17 g, 50% yield. White solid, m.p. 83-84 ºC. R_f = 0.10 (petroleum ether/ethyl acetate, 20:1).

^{1}H NMR (500 MHz, $CDCl_3$) δ 7.39 (d, *J* = 8.3 Hz, 2H), 7.21 (d, *J* = 8.2 Hz, 2H), 6.41 (s, 1H), 2.68 – 2.66 (m, 2H), 2.62 – 2.60 (m, 2H), 1.94 – 1.90 (m, 4H), 1.33 (s, 9H).

^{13}C NMR (126 MHz, $CDCl_3$) δ 173.3, 151.1, 150.5, 131.4, 128.2, 125.5, 119.0, 34.6, 33.8, 31.3, 29.8, 27.8, 23.3.

IR (KBr) v 2961, 1761, 1659, 1268, 1166, 1111, 1106, 896, 563.

HRMS (APCI) *m/z*: Calcd for $C_{17}H_{23}O_2$ ($[M+H]^+$) 259.1693, found 259.1693.



1g (QY-403-2)

(*E*)-7-(4-methoxybenzylidene)oxepan-2-one

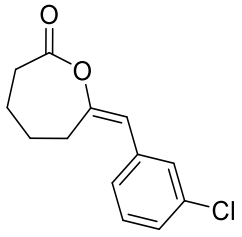
1.75 g, 75% yield. White solid, m.p. 77-78 ºC. R_f = 0.31 (petroleum ether/ethyl acetate, 5:1).

^{1}H NMR (500 MHz, $CDCl_3$) δ 7.19 (d, *J* = 8.6 Hz, 2H), 6.90 (d, *J* = 8.7 Hz, 2H), 6.38 (s, 1H), 3.82 (s, 3H), 2.68 – 2.66 (m, 2H), 2.59 – 2.57 (m, 2H), 1.92 – 1.89 (m, 4H).

^{13}C NMR (126 MHz, $CDCl_3$) δ 173.4, 158.9, 150.3, 129.6, 126.7, 118.7, 114.1, 55.3, 33.7, 29.7, 27.7, 23.3.

IR (KBr) v 2963, 1751, 1512, 1252, 1165, 1117, 894, 820.

HRMS (APCI) *m/z*: Calcd for $C_{14}H_{17}O_3$ ($[M+H]^+$) 233.1172, found 233.1174.



1h (QY-376-2)

(*E*)-7-(3-chlorobenzylidene)oxepan-2-one

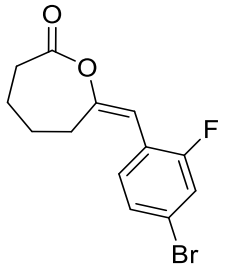
0.35 g, 68% yield. White solid, m.p. 43-45 ºC. R_f = 0.18 (petroleum ether/ethyl acetate, 10:1).

^{1}H NMR (500 MHz, $CDCl_3$) δ 7.32 – 7.24 (m, 3H), 7.14 (d, *J* = 7.4 Hz, 1H), 6.39 (s, 1H), 2.69 – 2.66 (m, 2H), 2.59 – 2.57 (m, 2H), 1.92 – 1.91 (m, 4H).

^{13}C NMR (126 MHz, $CDCl_3$) δ 172.7, 152.7, 136.2, 134.5, 129.8, 128.4, 127.5, 126.6, 118.0, 33.7, 29.7, 27.7, 23.1.

IR (KBr) v 2940, 1756, 1658, 1345, 1210, 1167, 1114, 1007, 786, 691.

HRMS (APCI) *m/z*: Calcd for $C_{13}H_{14}O_2Cl$ ($[M+H]^+$) 237.0677, found 237.0679.



1i (QY-372)

(*E*)-7-(4-bromo-2-fluorobenzylidene)oxepan-2-one

0.84 g, 64% yield. White solid, m.p. 97-98 ºC. R_f = 0.14 (petroleum ether/ethyl acetate, 10:1).

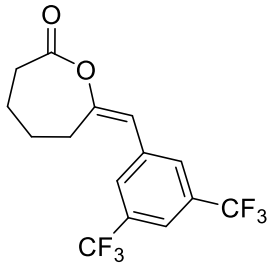
^{1}H NMR (500 MHz, $CDCl_3$) δ 7.31 – 7.27 (m, 2H), 7.14 (t, *J* = 8.2 Hz, 1H), 6.35 (s, 1H), 2.69 – 2.67 (m, 2H), 2.50 – 2.48 (m, 2H), 1.88 – 1.86 (m, 4H).

^{13}C NMR (101 MHz, $CDCl_3$) δ 172.4, 159.9 (d, $^1J_{CF}$ = 252.6 Hz), 153.6, 130.7 (d, $^3J_{CF}$ = 3.6 Hz), 127.5 (d, $^4J_{CF}$ = 3.6 Hz), 121.6 (d, $^3J_{CF}$ = 9.5 Hz), 121.4 (d, $^2J_{CF}$ = 14.9 Hz), 119.4 (d, $^2J_{CF}$ = 25.3 Hz), 111.2 (d, $^3J_{CF}$ = 3.6 Hz), 33.70, 29.63, 27.34, 23.01.

^{19}F NMR (658 MHz, $CDCl_3$) δ -111.1 (s).

IR (KBr) v 2957, 1751, 1657, 1170, 1135, 1010, 862.

HRMS (APCI) *m/z*: Calcd for $C_{13}H_{13}O_2BrF$ ($[M+H]^+$) 299.0077, found 299.0079.



1j (QY-394-2)

(*E*)-7-(3,5-bis(trifluoromethyl)benzylidene)oxepan-2-one

0.34 g, 37% yield. White solid, m.p. 87-88 ºC. R_f = 0.24 (petroleum ether/ethyl acetate, 10:1).

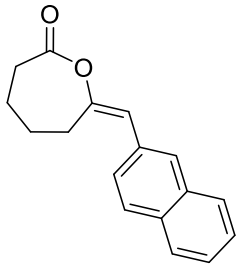
^{1}H NMR (500 MHz, $CDCl_3$) δ 7.80 (s, 1H), 7.69 (s, 2H), 6.50 (s, 1H), 2.71 – 2.69 (m, 2H), 2.59 – 2.57 (m, 2H), 1.96 – 1.94 (m, 4H).

^{13}C NMR (126 MHz, $CDCl_3$) δ 171.9, 154.6, 136.7, 132.1 (q, $^2J_{CF}$ = 33.3 Hz), 128.4 – 128.3 (m), 121.1 (q, $^1J_{CF}$ = 272.9 Hz), 121.1 – 121.0 (m), 116.7, 33.8, 29.8, 27.5, 23.0.

^{19}F NMR (658 MHz, $CDCl_3$) δ -111.1 (s).

IR (KBr) v 2953, 1767, 1653, 1281, 1128, 901, 825, 714, 682.

HRMS (APCI) *m/z*: Calcd for $C_{15}H_{13}O_2F_6$ ($[M+H]^+$) 339.0814, found 339.0817.



1k (QY-442-2)

(*E*)-7-(naphthalen-2-ylmethylene)oxepan-2-one

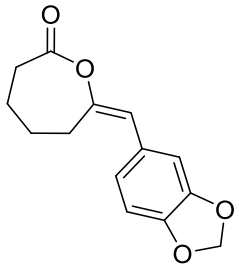
0.80 g, 29% yield. White solid, m.p. 131-132 ºC. R_f = 0.17 (petroleum ether/ethyl acetate, 10:1).

^{1}H NMR (500 MHz, $CDCl_3$) δ 7.84 – 7.81 (m, 3H), 7.71 (s, 1H), 7.51 – 7.46 (m, 2H), 7.38 (dd, *J* = 8.4, 1.0 Hz, 1H), 6.59 (s, 1H), 2.74 – 2.71 (m, 2H), 2.68 – 2.65 (m, 2H), 2.01 – 1.90 (m, 4H).

^{13}C NMR (126 MHz, $CDCl_3$) δ 173.1, 152.0, 133.3, 132.4, 131.9, 128.2, 127.9, 127.7, 127.3, 126.46, 126.43, 126.2, 119.3, 33.8, 29.8, 27.9, 23.2.

IR (KBr) v 2954, 1747, 1655, 1110, 1005, 910, 829, 745.

HRMS (APCI) *m/z*: Calcd for $C_{17}H_{17}O_2$ ($[M+H]^+$) 253.1223, found 253.1223.



1l (QY-454-2)

(*E*)-7-(benzo[d][1,3]dioxol-5-ylmethylene)oxepan-2-one

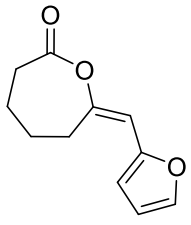
0.49 g, 37% yield. Yellowish solid, m.p. 121-122 ºC. R_f = 0.16 (petroleum ether/ethyl acetate, 10:1).

^{1}H NMR (500 MHz, $CDCl_3$) δ 6.81 (d, *J* = 7.9Hz, 1H), 6.75 – 6.72 (m, 2H), 6.35 (s, 1H), 5.98 (s, 2H), 2.67 – 2.65 (m, 2H), 2.59 – 2.57 (m, 2H), 1.91 – 1.89 (m, 4H).

^{13}C NMR (126 MHz, $CDCl_3$) δ 173.2, 150.7, 147.9, 146.9, 128.2, 122.3, 118.9, 108.5, 108.5, 101.2, 33.7, 29.7, 27.7, 23.2.

IR (KBr) v 2933, 1748, 1659, 1438, 1238, 1118, 1007, 933, 875.

HRMS (APCI) *m/z*: Calcd for $C_{14}H_{15}O_4$ ($[M+H]^+$) 247.0965, found 247.0966.



1m (QY-380-2)

(*E*)-7-(naphthalen-2-ylmethylene)oxepan-2-one

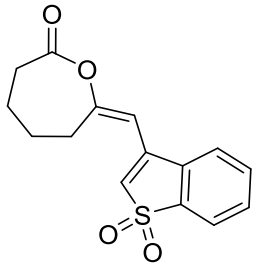
0.45 g, 46% yield. Yellow oil. R_f = 0.28 (petroleum ether/ethyl acetate, 10:1).

^{1}H NMR (400 MHz, $CDCl_3$) δ 7.43 (br, 1H), 6.44 – 6.42 (m, 1H), 6.29 (d, *J* = 3.3 Hz, 1H), 6.20 (s, 1H), 2.80 – 2.77 (m, 2H), 2.65 – 2.62 (m, 2H), 1.93 – 1.87 (m, 4H).

^{13}C NMR (101 MHz, $CDCl_3$) δ 173.1, 150.9, 149.3, 142.5, 111.4, 110.0, 108.0, 33.7, 30.7, 26.9, 23.5.

IR (KBr) v 2939, 1751, 1668, 1345, 1168, 1111, 1005, 886, 739.

HRMS (APCI) *m/z*: Calcd for $C_{11}H_{13}O_3$ ($[M+H]^+$) 193.0859, found 193.0861.



1n (QY-455-2)

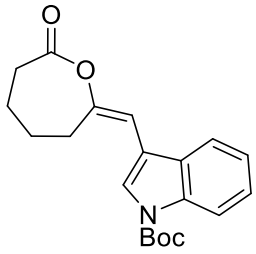
(*E*)-7-((1,1-dioxidobenzo[b]thiophen-3-yl)methylene)oxepan-2-one

1.06 g, 33% yield. White solid, m.p. 134-135 ºC. R_f = 0.31 (petroleum ether/ethyl acetate, 5:1).

^{1}H NMR (500 MHz, $CDCl_3$) δ 7.88 – 7.87 (m, 1H), 7.74 (dd, *J* = 7.5 Hz, 1.2Hz, 1H), 7.44 – 7.38 (m, 2H), 7.30 (s, 1H), 6.60 (s, 1H), 2.73 – 2.71 (m, 2H), 2.65 – 2.63 (m, 2H), 1.93 – 1.91 (m, 4H).

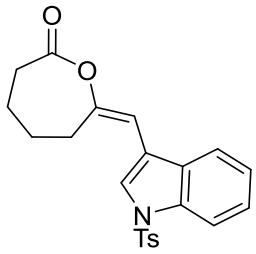
^{13}C NMR (126 MHz, $CDCl_3$) δ 172.9, 153.3, 139.6, 138.6, 129.6, 124.9, 124.5, 123.0, 122.8, 121.9, 111.1, 33.8, 30.2, 27.9, 23.3.

IR (KBr) v 2940, 1739, 1657, 1346, 1177, 1119, 1006, 858, 762.
HRMS (APCI) *m/z*: Calcd for $C_{15}H_{15}O_4S$ ($[M+H]^+$) 291.0686, found 291.0689.



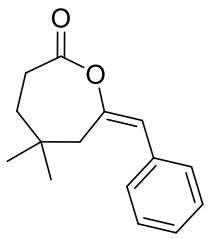
1o (QY-425-2)

tert-butyl (*E*)-3-((7-oxooxepan-2-ylidene)methyl)-1H-indole-1-carboxylate
0.74 g, 43% yield. Brown solid, m.p. 127-128 ºC. R_f = 0.18 (petroleum ether/ethyl acetate, 10:1).
^{1}H NMR (500 MHz, $CDCl_3$) δ 8.09 (d, *J* = 6.2 Hz, 1H), 7.57 – 7.53 (m, 2H), 7.38 – 7.34 (m, 1H), 7.28 (dd, *J* = 13.4, 5.9 Hz, 1H), 6.48 (s, 1H), 2.71 – 2.65 (m, 4H), 1.93 – 1.91 (m, 4H), 1.70 (s, 9H).
^{13}C NMR (126 MHz, $CDCl_3$) δ 173.1, 152.1, 149.7, 134.8, 130.1, 125.0, 123.0, 122.4, 118.9, 115.3, 114.5, 108.4, 84.3, 33.7, 30.6, 28.2, 27.0, 23.4.
IR (KBr) v 2937, 1733, 1454, 1373, 1271, 1155, 999, 761, 747.
HRMS (APCI) *m/z*: Calcd for $C_{20}H_{22}O_4N$ ($[M-H]^-$) 340.1554, found 247.0966.



1p (QY-456-2)

(*E*)-7-((1-tosyl-1H-indol-3-yl)methylene)oxepan-2-one
2.16 g, 68% yield. Light yellow solid, m.p. 158-159 ºC. R_f = 0.29 (petroleum ether/ethyl acetate, 3:1).
^{1}H NMR (500 MHz, $CDCl_3$) δ 7.98 (d, *J* = 8.3 Hz, 1H), 7.77 (d, *J* = 8.3 Hz, 2H), 7.49 (d, *J* = 8.8 Hz, 2H), 7.35 (t, *J* = 7.7 Hz, 1H), 7.29 – 7.23 (m, 3H), 6.41 (s, 1H), 2.70 – 2.68 (m, 2H), 2.64 – 2.61 (m, 2H), 2.35 (s, 3H), 1.94 – 1.93 (m, 4H).
^{13}C NMR (126 MHz, $CDCl_3$) δ 172.7, 152.7, 145.2, 135.0, 134.6, 130.3, 130.0, 126.8, 125.4, 123.6, 122.4, 119.4, 116.3, 113.7, 107.8, 33.6, 30.5, 27.0, 23.3, 21.6.
IR (KBr) v 2942, 1750, 1662, 1371, 1174, 1115, 1105, 760, 673, 587.
HRMS (APCI) *m/z*: Calcd for $C_{22}H_{22}O_4NS$ ($[M+H]^+$) 396.1264, found 396.1268.



1q (QY-416-2)

(*E*)-7-benzylidene-5,5-dimethyloxepan-2-one

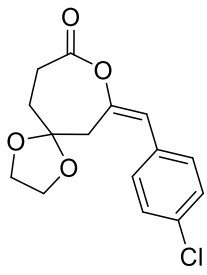
2.02 g, 78% yield. White solid, m.p. 123-124 ºC. R_f = 0.14 (petroleum ether/ethyl acetate, 20:1).

^{1}H NMR (500 MHz, $CDCl_3$) δ 7.37 – 7.33 (m, 2H), 7.28 – 7.25 (m, 3H), 6.54 (s, 1H), 2.64 – 2.61 (m, 2H), 2.47 (s, 2H), 1.63 – 1.61 (m, 2H), 0.91 (s, 6H).

^{13}C NMR (126 MHz, $CDCl_3$) δ 173.3, 149.4, 134.5, 128.60, 128.55, 127.3, 120.9, 40.8, 36.5, 34.0, 30.0, 28.5.

IR (KBr) v 2965, 1742, 1671, 1228, 1120, 745, 701.

HRMS (APCI) *m/z*: Calcd for $C_{15}H_{19}O_2$ ($[M+H]^+$) 231.1380, found 231.1379.



1r (QY-426-2)

(*E*)-7-(4-chlorobenzylidene)-1,4,8-trioxaspiro[4.6]undecan-9-one

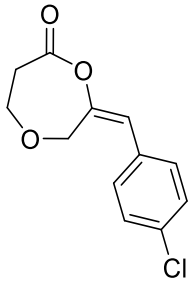
1.74 g, 74% yield. White solid, m.p. 130-131 ºC. R_f = 0.18 (petroleum ether/ethyl acetate, 5:1).

^{1}H NMR (500 MHz, $CDCl_3$) δ 7.39 (d, *J* = 8.5 Hz, 2H), 7.34 – 7.31 (m, 2H), 6.56 (s, 1H), 4.01 – 3.91 (m, 4H), 2.76 – 2.72 (m, 4H), 2.03 – 2.01 (m, 2H).

^{13}C NMR (126 MHz, $CDCl_3$) δ 172.3, 146.2, 133.5, 132.5, 130.1, 128.6, 121.4, 107.9, 64.9, 39.2, 33.6, 29.4.

IR (KBr) v 2959, 1750, 1663, 1170, 1141, 1114, 1039, 956, 888, 546.

HRMS (APCI) *m/z*: Calcd for $C_{15}H_{16}O_4Cl$ ($[M+H]^+$) 295.0732, found 295.0732.

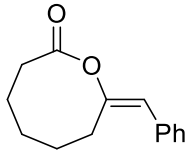


1s (QY-469-2)

(*E*)-3-(4-chlorobenzylidene)-1,4-dioxepan-5-one

0.44 g, 29% yield. White solid, m.p. 116-117 ºC. R_f = 0.20 (petroleum ether/ethyl acetate, 20:1).

^{1}H NMR (500 MHz, $CDCl_3$) δ 7.37 (d, *J* = 8.4 Hz, 2H), 7.31 (d, *J* = 8.4 Hz, 2H), 6.64 (s, 1H), 4.25 (s, 2H), 4.01 – 3.99 (m, 2H), 2.97 – 2.95 (m, 2H).

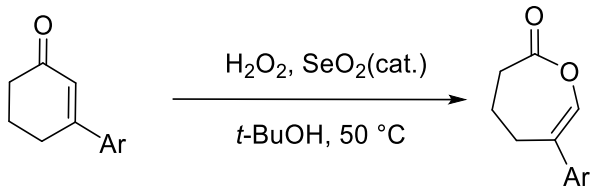


(*E*)-8-benzylideneoxocan-2-one

1.19 g, 24% yield. Colorless oil. R_f = 0.20 (petroleum ether/ethyl acetate, 20:1).

^{1}H NMR (300 MHz, $CDCl_3$) δ 7.39 – 7.35 (m, 2H), 7.30 – 7.25 (m, 3H), 6.38 (s, 1H), 2.73 – 2.69 (m, 2H), 2.63 – 2.59 (m, 2H), 2.97 – 2.95 (m, 2H), 1.89 – 1.81 (m, 2H), 1.78 – 1.71 (m, 2H), 1.68 – 1.61 (m, 2H).

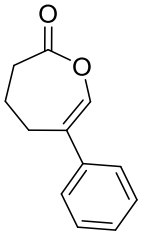
2.2 Preparation of *endo*-enol lactones



S2 **3**

General procedure: To a mixture of cycloketone **S2**[4] (1.0 equiv), SeO_2 (0.05 equiv) in *t*-BuOH (3mL/mmol) was added aqueous 30% H_2O_2 (3.0 equiv) at 30 °C. The reaction mixture was stirred at 50 °C in open air. After consumption of the starting material (monitored by TLC, typically 24-36 h), the reaction mixture was quenched by addition of sat. Na_2SO_3. The solvent was removed under reduced pressure. To the residue, water was added and extracted with DCM for three times. The combined organic extracts were dried over anhydrous $MgSO_4$ and concentrated under reduced pressure. Purification by flash column chromatography afforded the desired *endo*-enol lactone **3**.

Characterization Data of *endo*-enol lactones **3**



3a (QY-472)

6-phenyl-4,5-dihydrooxepin-2(3H)-one

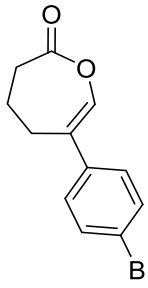
0.74 g, 39% yield. White solid, m.p. 82-84 °C. R_f = 0.22 (petroleum ether/ethyl acetate, 10:1).

^{1}H NMR (500 MHz, $CDCl_3$) δ 7.39 – 7.30 (m, 5H), 6.76 (s, 1H), 2.74 (t, *J* = 7.1 Hz, 2H), 2.71 (t, *J* = 7.4 Hz, 2H), 2.29 (p, *J* = 7.3 Hz, 2H).

^{13}C NMR (126 MHz, $CDCl_3$) δ 172.2, 137.7, 137.4, 129.0, 128.8, 127.9, 126.0, 32.3, 26.80, 26.76.

IR (KBr) v 2936, 1776, 1456, 1215, 1124, 834, 762, 696.

HRMS (APCI) *m/z*: Calcd for $C_{12}H_{13}O_2$ ($[M+H]^+$) 189.0910, found 189.0913.



3b (QY-505-2)

6-(4-chlorophenyl)-4,5-dihydrooxepin-2(3H)-one

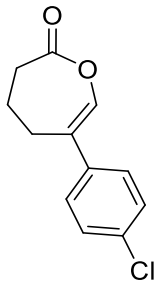
0.42 g, 21% yield. White solid, m.p. 87-89 ºC. R_f = 0.24 (petroleum ether/ethyl acetate, 7:1).

^{1}H NMR (500 MHz, $CDCl_3$) δ 7.50 – 7.48 (m, 2H), 7.21 – 7.20 (m, 2H), 6.75 (s, 1H), 2.74 (t, *J* = 7.1 Hz, 2H), 2.68 (t, *J* = 7.4 Hz, 2H), 2.28 (p, *J* = 7.3 Hz, 2H).

^{13}C NMR (126 MHz, $CDCl_3$) δ 171.8, 137.9, 136.4, 131.9, 128.1, 127.6, 121.8, 32.3, 26.7, 26.7.

IR (KBr) v 2940, 2872, 1757, 1634, 1207, 1134, 1007, 822.

HRMS (APCI) *m/z*: Calcd for $C_{12}H_{12}O_2$ ($[M+H]^+$) 267.0015, found 267.0017.



3c (QY-488-2)

6-(4-chlorophenyl)-4,5-dihydrooxepin-2(3H)-one

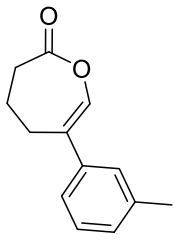
0.57 g, 23% yield. Colorless crystalline solid, m.p. 61-63 ºC. R_f = 0.27 (petroleum ether/ethyl acetate, 7:1).

^{1}H NMR (500 MHz, $CDCl_3$) δ 7.34 (d, *J* = 8.4 Hz, 2H), 7.27 – 7.25 (m, 2H), 6.75 (s, 1H), 2.74 (t, *J* = 7.1 Hz, 2H), 2.68 (t, *J* = 7.4 Hz, 2H), 2.28 (p, *J* = 7.2 Hz, 2H).

^{13}C NMR (126 MHz, $CDCl_3$) δ 171.9, 137.9, 135.9, 133.8, 129.0, 128.0, 127.3, 32.3, 26.8, 26.7.

IR (KBr) v 2939, 1761, 1635, 1493, 1212, 1134, 974, 827, 519.

HRMS (APCI) *m/z*: Calcd for $C_{12}H_{12}O_2Cl$ ($[M+H]^+$) 223.0520, found 223.0522.



3d (QY-482-2)

6-(m-tolyl)-4,5-dihydrooxepin-2(3H)-one

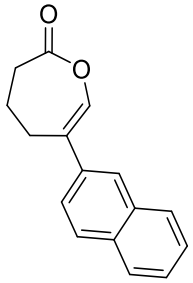
1.21 g, 42% yield. Colorless oil. R_f = 0.27 (petroleum ether/ethyl acetate, 10:1).

^{1}H NMR (500 MHz, $CDCl_3$) δ 7.27 – 7.24 (m, 1H), 7.14 – 7.12 (m, 3H), 6.74 (s, 1H), 2.74 (t, *J* = 7.1 Hz, 2H), 2.69 (t, *J* = 7.4 Hz, 2H), 2.37 (s, 3H), 2.28 (p, *J* = 7.3 Hz, 2H).

^{13}C NMR (126 MHz, $CDCl_3$) δ 172.3, 138.5, 137.5, 137.4, 129.1, 128.7, 128.6, 126.7, 123.1, 32.3, 26.8, 26.8, 21.5.

IR (KBr) v 2939, 1761, 1345, 1213, 1127, 1056, 783, 700.

HRMS (APCI) *m/z*: Calcd for $C_{13}H_{15}O_2$ ($[M+H]^+$) 203.1067, found 203.1068.



3e (QY-498-2)

6-(naphthalen-2-yl)-4,5-dihydrooxepin-2(3H)-one

1.05 g, 49% yield. White solid, m.p. 139-140 ºC. R_f = 0.21 (petroleum ether/dichloromethane/ethyl acetate, 30:10:1).

^{1}H NMR (500 MHz, $CDCl_3$) δ 7.83 – 7.81 (m, 3H), 7.76 (br, 1H), 7.51 – 7.44 (m, 3H), 6.88 (s, 1H), 2.82 – 2.76 (m, 4H), 2.33 (p, *J* = 7.3 Hz, 2H).

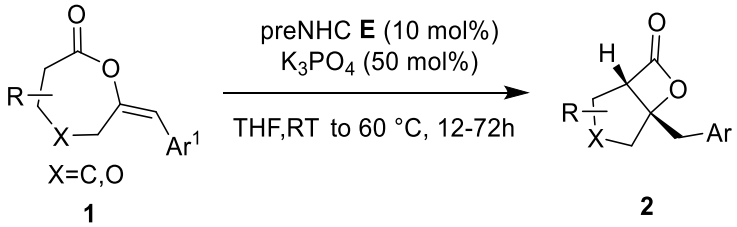
^{13}C NMR (126 MHz, $CDCl_3$) δ 172.2, 138.1, 134.6, 133.4, 132.8, 129.0, 128.6, 127.9, 127.6, 126.6, 126.3, 124.8, 123.9, 32.4, 26.8, 26.7.

IR (KBr) v 2928, 2867, 1784, 1452, 1217, 1122, 852, 827, 753.

HRMS (APCI) *m/z*: Calcd for $C_{16}H_{15}O_2$ ($[M+H]^+$) 239.1067, found 239.1067.

3. Synthesis of bicyclic β-lactones.

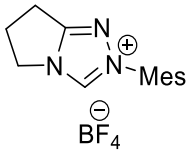
3.1 Synthesis from *exo*-enol lactones



Typical procedure: To an oven-dried 25 mL Schlenk tube equipped with a stir bar was charged with enol lactone **1a** (Ar^1 = Ph, X = C, 60.6 mg, 0.3 mmol), preNHC **E** (15.1 mg, 10 mol%) and K_3PO_4 (31.8 mg, 50 mol%). This tube was closed with a septum, evacuated, back-filled with nitrogen for three times. To this mixture was added freshly distilled dry THF (3 mL). The reaction mixture was stirred at room temperature until the full consumption of enol lactone **1a**. The reaction mixture was concentrated under reduced pressure, and the residue was purified by column chromatography on silica gel (petroleum ether/EtOAc = 10:1 as the eluent) to give the product **2a** (43 mg, 71% yield, 96% ee).

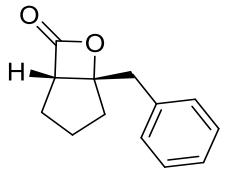
Racemic samples for the chiral phase HPLC analysis were prepared using achiral NHC precursor **F**

under the same conditions.



F

Characterization Data of β-lactones **2**



2a (QY-343-1)

(1*S*,5*S*)-5-benzyl-6-oxabicyclo[3.2.0]heptan-7-one

43 mg, 71% yield. Reaction time: 48 h. Colorless oil. R_f = 0.19 (petroleum ether/ethyl acetate, 10:1). $[\alpha]_D^{26}$ +13.0 (*c* = 1.29, $CHCl_3$).

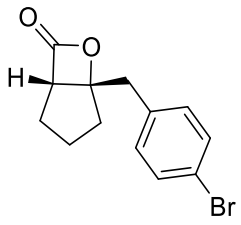
HPLC analysis: 96% ee (Daicel CHIRALPAK IC column, 25 °C, 210 nm, hexane/*i*-PrOH = 98:2, 1.0 mL/min, 21.28 min (minor), 24.78 min (major)).

^{1}H NMR (400 MHz, $CDCl_3$) δ 7.33 – 7.22 (m, 5H), 3.50 (d, *J* = 7.9 Hz, 1H), 3.23 (s, 2H), 2.10 – 2.02 (m, 2H), 1.93 – 1.77 (m, 2H), 1.65 – 1.52 (m, 2H).

^{13}C NMR (101 MHz, $CDCl_3$) δ 171.0, 135.8, 129.8, 128.6, 127.1, 89.6, 57.6, 41.3, 34.1, 27.0, 23.6.

IR (KBr) v 3030, 2964, 1825, 1455, 1169, 1082, 801, 703.

HRMS (ESI) *m/z*: Calcd for $C_{13}H_{13}O_2$ ($[M-H]^-$) 201.0921, found 201.0919.



2b (QY-462)

(1*S*,5*S*)-5-(4-bromobenzyl)-6-oxabicyclo[3.2.0]heptan-7-one

34 mg (0.2mmol scale), 60% yield. Reaction time: 48 h. White solid, m.p. 59-61 °C. R_f = 0.16 (petroleum ether/ethyl acetate, 10:1). $[\alpha]_D^{26}$ -0.1 (*c* = 0.81, $CHCl_3$).

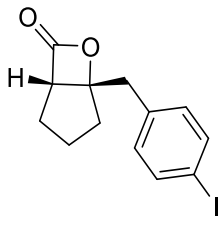
HPLC analysis: 93% ee (Daicel CHIRALPAK IC column, 25 °C, 210 nm, hexane/*i*-PrOH = 98:2, 1.0 mL/min, 24.36 min (minor), 28.08 min (major)).

^{1}H NMR (500 MHz, $CDCl_3$) δ 7.44 (d, *J* = 8.3 Hz, 2H), 7.12 (d, *J* = 8.2 Hz, 2H), 3.47 (d, *J* = 7.9 Hz, 1H), 3.18 (s, 2H), 2.11 – 2.02 (m, 2H), 1.95 – 1.89 (m, 1H), 1.87 – 1.78 (m, 1H), 1.67 – 1.50 (m, 2H).

^{13}C NMR (126 MHz, $CDCl_3$) δ 170.6, 134.7, 131.7, 131.5, 121.2, 89.1, 57.7, 40.8, 34.2, 27.0, 23.6.

IR (KBr) v 2963, 2924, 1820, 1489, 1102, 1071, 1012, 843, 798.

HRMS (ESI) *m/z*: Calcd for $C_{13}H_{12}O_2Br$ ($[M-H]^-$) 279.0026, found 279.0030.



2c (QY-435)

(1*S*,5*S*)-5-(4-iodobenzyl)-6-oxabicyclo[3.2.0]heptan-7-one

48 mg (0.2mmol scale), 73% yield. Reaction time: 36 h. Colorless crystalline solid, m.p. 53-56 ºC. R_f = 0.14 (petroleum ether/ethyl acetate, 10:1). $[\alpha]_D^{26}$ -0.3 (*c* = 0.30, $CHCl_3$).

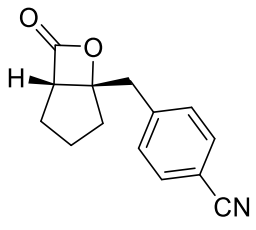
HPLC analysis: 93% ee (Daicel CHIRALPAK IC column, 25 ºC, 210 nm, hexane/*i*-PrOH = 98:2, 1.0 mL/min, 24.30 min (minor), 28.29 min (major)).

^{1}H NMR (500 MHz, $CDCl_3$) δ 7.65 – 7.63 (m, 2H), 7.00 – 6.98 (m, 2H), 3.47 (d, *J* = 7.9 Hz, 1H), 3.17 (s, 2H), 2.11 – 2.02 (m, 2H), 1.95 – 1.89 (m, 1H), 1.87 – 1.77 (m, 1H), 1.67 – 1.50 (m, 2H).

^{13}C NMR (126 MHz, $CDCl_3$) δ 170.6, 137.7, 135.4, 131.7, 92.7, 89.1, 57.7, 40.8, 34.2, 27.0, 23.54.

IR (KBr) v 2918, 2849, 1820, 1484, 1166, 1101, 1008, 802.

HRMS (ESI) *m/z*: Calcd for $C_{13}H_{12}O_2I$ ($[M-H]^-$) 326.9887, found 326.9891.



2d (QY-398)

4-(((1*S*,5*S*)-7-oxo-6-oxabicyclo[3.2.0]heptan-5-yl)methyl)benzonitrile

32 mg, 47% yield. Reaction time: 24 h. Colorless crystalline solid, m.p. 88-89 ºC. R_f = 0.28 (petroleum ether/ethyl acetate, 3:1). $[\alpha]_D^{26}$ -15.1 (*c* = 0.27, $CHCl_3$).

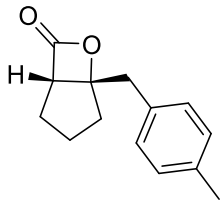
HPLC analysis: 96% ee (Daicel CHIRALPAK IA column, 25 ºC, 210 nm, hexane/*i*-PrOH = 95:5, 1.0 mL/min, 26.89 min (minor), 32.65 min (major)).

^{1}H NMR (500 MHz, $CDCl_3$) δ 7.63 – 7.62 (m, 2H), 7.39 – 7.37 (m, 2H), 3.50 (d, *J* = 7.7 Hz, 1H), 3.33 – 3.26 (m, 2H), 2.12 (dd, *J* = 13.1, 5.6 Hz, 1H), 2.04 (dd, *J* = 14.2, 5.8 Hz, 1H), 1.98 – 1.93 (m, 1H), 1.87 – 1.79 (m, 1H), 1.72 – 1.64 (m, 1H), 1.58 – 1.51 (m, 1H).

^{13}C NMR (126 MHz, $CDCl_3$) δ 170.2, 141.3, 132.4, 130.6, 118.6, 111.3, 88.6, 58.1, 41.5, 34.37, 27.0, 23.6.

IR (KBr) v 2962, 2921, 2228, 1817, 1609, 1167, 1102, 849, 802, 550.

HRMS (ESI) *m/z*: Calcd for $C_{14}H_{12}O_2N$ ($[M-H]^-$) 226.0873, found 226.0872.



2e (QY-443)

(1*S*,5*S*)-5-(4-methylbenzyl)-6-oxabicyclo[3.2.0]heptan-7-one

27 mg, 42% yield. Reaction time: 36 h. Colorless oil. R_f = 0.31 (petroleum ether/ethyl acetate, 10:1).

$[\alpha]_D^{26}$ +17.6 (*c* = 0.55, $CHCl_3$).

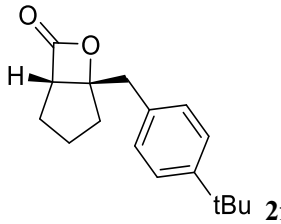
HPLC analysis: 90% ee (Daicel CHIRALPAK IC column, 25 ºC, 210 nm, hexane/*i*-PrOH = 98:2, 1.0 mL/min, 21.00 min (minor), 25.01 min (major)).

^{1}H NMR (400 MHz, $CDCl_3$) δ 7.11 (s, 4H), 3.48 (d, *J* = 7.8 Hz, 1H), 3.23 – 3.15 (m, 2H), 2.32 (s, 3H), 2.09 – 2.02 (m, 2H), 1.93 – 1.75 (m, 2H), 1.64 – 1.52 (m, 2H).

^{13}C NMR (101 MHz, $CDCl_3$) δ 171.1, 136.7, 132.7, 129.6, 129.3, 89.7, 57.5, 40.8, 34.1, 27.0, 23.6, 21.1.

IR (KBr) v 2938, 1800, 1515, 1254, 1157, 1105, 942, 801, 776, 645.

HRMS (ESI) *m/z*: Calcd for $C_{14}H_{15}O_2$ ($[M-H]^-$) 215.1077, found 215.1074.



2f (QY-461)

(1*S*,5*S*)-5-(4-(tert-butyl)benzyl)-6-oxabicyclo[3.2.0]heptan-7-one

41 mg, 53% yield. Reaction time: 48 h. White solid, m.p. 49-50 ºC. R_f = 0.29 (petroleum ether/ethyl acetate, 10:1). $[\alpha]_D^{26}$ +8.6 (*c* = 0.80, $CHCl_3$).

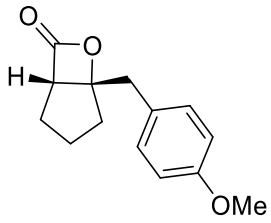
HPLC analysis: 93% ee (Daicel CHIRALPAK IC column, 25 ºC, 210 nm, hexane/*i*-PrOH = 98:2, 1.0 mL/min, 16.19 min (minor), 19.47 min (major)).

^{1}H NMR (500 MHz, $CDCl_3$) δ 7.33 (d, *J* = 8.1 Hz, 2H), 7.16 (d, *J* = 8.1 Hz, 2H), 3.50 (d, *J* = 7.9 Hz, 1H), 3.23 – 3.17 (d, *J* = 2.9 Hz, 2H), 2.09 – 2.04 (m, 2H), 1.93 – 1.76 (m, 2H), 1.65 – 1.54 (m, 2H), 1.31 (s, 9H).

^{13}C NMR (126 MHz, $CDCl_3$) δ 170.1, 148.9, 131.6, 128.4, 124.5, 88.75, 56.5, 39.7, 33.4, 33.1, 30.3, 26.0, 22.5.

IR (KBr) v 2962, 2868, 1799, 1363, 1268, 1154, 1107, 1036, 802.

HRMS (ESI) *m/z*: Calcd for $C_{17}H_{23}O_2$ ($[M+H]^+$) 259.1693, found 259.1694.



2g (QY-413-4)

(1*S*,5*S*)-5-(4-methoxybenzyl)-6-oxabicyclo[3.2.0]heptan-7-one

44 mg, 64% yield. Reaction time: 36 h. Colorless oil. R_f = 0.33 (petroleum ether/ethyl acetate, 5:1). $[\alpha]_D^{26}$ +9.1 (*c* = 0.29, $CHCl_3$).

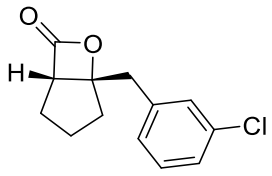
HPLC analysis: 92% ee (Daicel CHIRALPAK IC column, 25 ºC, 210 nm, hexane/*i*-PrOH = 97:3, 1.0 mL/min, 33.05 min (minor), 33.69 min (major)).

^{1}H NMR (500 MHz, $CDCl_3$) δ 7.15 – 7.14 (m, 2H), 6.85 – 6.84 (m, 2H), 3.79 (s, 3H), 3.47 (d, *J* = 7.9 Hz, 1H), 3.20 – 3.13 (m, 2H), 2.09 – 2.02 (m, 2H), 1.93 – 1.76 (m, 2H), 1.64 – 1.52 (m, 2H).

^{13}C NMR (126 MHz, $CDCl_3$) δ 171.1, 158.7, 130.7, 127.8, 114.0, 89.8, 57.4, 55.2, 40.4, 34.0, 27.0, 23.6.

IR (KBr) v 2929, 1819, 1718, 1611, 1513, 1248, 1177, 1034, 803.

HRMS (ESI) *m/z*: Calcd for $C_{14}H_{15}O_3$ ([M-H]$^-$) 231.1027, found 231.1029.



2h (QY-389)

(1*S*,5*S*)-5-(3-chlorobenzyl)-6-oxabicyclo[3.2.0]heptan-7-one

44 mg, 62% yield. Reaction time: 12 h. Colorless oil. R_f = 0.17 (petroleum ether/ethyl acetate, 10:1). $[\alpha]_D^{26}$ +2.5 (*c* = 0.33, $CHCl_3$).

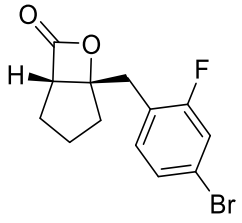
HPLC analysis: 96% ee (Daicel CHIRALPAK IC column, 25 ºC, 210 nm, hexane/*i*-PrOH = 98:2, 1.0 mL/min, 19.56 min (minor), 21.73 min (major)).

^{1}H NMR (400 MHz, $CDCl_3$) δ 7.26 – 7.24 (m, 3H), 7.14 – 7.12 (m, 1H), 3.50 (d, *J* = 7.8 Hz, 1H), 3.24 – 3.16 (m, 2H), 2.12 – 2.02 (m, 2H), 1.96 – 1.76 (m, 2H), 1.69 – 1.51 (m, 2H).

^{13}C NMR (101 MHz, $CDCl_3$) δ 170.6, 137.8, 134.3, 129.9, 129.8, 128.0, 127.4, 89.1, 57.9, 41.0, 34.2, 27.0, 23.6.

IR (KBr) v 2925, 1822, 1598, 1574, 1475, 1432, 1101, 801, 785, 713.

HRMS (ESI) *m/z*: Calcd for $C_{13}H_{12}O_2Cl$ ([M-H]$^-$) 235.0531, found 235.0533.



2i (QY-395)

(1*S*,5*S*)-5-(4-bromo-2-fluorobenzyl)-6-oxabicyclo[3.2.0]heptan-7-one

58 mg, 65% yield. Reaction time: 10 h. Colorless oil. R_f = 0.20 (petroleum ether/ethyl acetate, 10:1). $[\alpha]_D^{26}$ -0.2 (*c* = 0.46, $CHCl_3$).

HPLC analysis: 94% ee (Daicel CHIRALPAK IC column, 25 ºC, 210 nm, hexane/*i*-PrOH = 98:2, 1.0 mL/min, 16.81 min (minor), 17.87 min (major)).

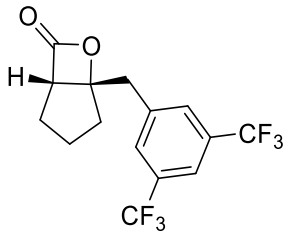
^{1}H NMR (400 MHz, $CDCl_3$) δ 7.27 – 7.24 (m, 2H), 7.19 – 7.15 (m, 1H), 3.51 (d, *J* = 7.8 Hz, 1H), 3.28 (d, *J* = 14.5Hz, 1H), 3.21 (d, *J* = 14.5Hz, 1H), 2.11 – 2.04 (m, 2H), 1.97 – 1.76 (m, 2H), 1.71 – 1.54 (m, 2H).

^{13}C NMR (101 MHz, $CDCl_3$) δ 170.51, 160.8 (d, $^1J_{CF}$ = 249.8 Hz), 133.4 (d, $^3J_{CF}$ = 4.7 Hz), 127.7 (d, $^4J_{CF}$ = 3.7 Hz), 121.9 (d, $^2J_{CF}$ = 15.8 Hz), 121.4 (d, $^3J_{CF}$ = 9.5 Hz), 119.2 (d, $^2J_{CF}$ = 25.7 Hz), 88.74, 57.9, 34.16, 33.6, 27.02, 23.59.

^{19}F NMR (376 MHz, $CDCl_3$) δ -114.4 (s).

IR (KBr) v 2923, 1822, 1577, 1486, 1404, 1218, 1166, 1101, 879, 804.

HRMS (ESI) *m/z*: Calcd for $C_{13}H_{11}O_2BrF$ ([M-H]$^-$) 296.9932, found 296.9935.



2j (QY-407)

(1*S*,5*S*)-5-(3,5-bis(trifluoromethyl)benzyl)-6-oxabicyclo[3.2.0]heptan-7-one

35 mg (0.2mmol scale), 51% yield. Reaction time: 12 h. Colorless oil. R_f = 0.10 (petroleum ether/ethyl acetate, 10:1). $[\alpha]_D^{26}$ -20.7 (*c* = 0.60, $CHCl_3$).

HPLC analysis: 96% ee (Daicel CHIRALPAK IB column, 25 ºC, 210 nm, hexane/*i*-PrOH = 99.5:0.5, 1.0 mL/min, 16.68 min (minor), 17.91 min (major)).

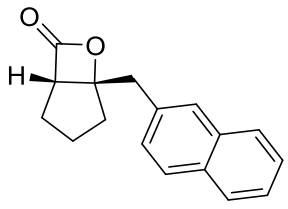
^{1}H NMR (400 MHz, $CDCl_3$) δ 7.82 (s, 1H), 7.73 (s, 2H), 3.54 (d, *J* = 7.7 Hz, 1H), 3.42 (d, *J* = 14.6 Hz, 1H), 3.31 (d, *J* = 14.6 Hz, 1H), 2.18 – 2.13 (m, 1H), 2.06 – 1.95 (m, 2H), 1.92 – 1.79 (m, 1H), 1.77 – 1.66 (m, 1H), 1.59 – 1.51 (m, 1H).

^{13}C NMR (101 MHz, $CDCl_3$) δ 169.9, 138.3, 132.0 (q, $^2J_{CF}$ = 33.5 Hz), 129.8 (m), 123.2 (q, $^1J_{CF}$ = 272.6 Hz), 121.4 (m), 88.2, 58.5, 41.2, 34.3, 27.0, 23.6.

^{19}F NMR (658 MHz, $CDCl_3$) δ -62.8 (d, *J* = 7.7 Hz).

IR (KBr) v 2923, 1824, 1380, 1336, 1279, 1171, 1132, 1106, 1076, 899, 840, 805, 709.

HRMS (ESI) *m/z*: Calcd for $C_{15}H_{11}O_2F_6$ ($[M-H]^-$) 337.0669, found 337.0673.



2k (QY-459)

(1*S*,5*S*)-5-(naphthalen-2-ylmethyl)-6-oxabicyclo[3.2.0]heptan-7-one

49 mg, 65% yield. Reaction time: 72 h. White solid, m.p. 58-60 ºC. R_f = 0.16 (petroleum ether/ethyl acetate, 10:1). $[\alpha]_D^{26}$ -2.2 (*c* = 1.14, $CHCl_3$).

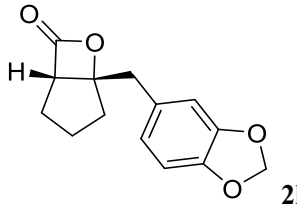
HPLC analysis: 87% ee (Daicel CHIRALPAK IC column, 25 ºC, 210 nm, hexane/*i*-PrOH = 98:2, 1.0 mL/min, 31.85 min (minor), 38.23 min (major)).

^{1}H NMR (500 MHz, $CDCl_3$) δ 7.82 – 7.78 (m, 3H), 7.67 (s, 1H), 7.48 – 7.44 (m, 2H), 7.36 (dd, *J* = 8.4, 1.2 Hz, 1H), 3.55 (d, *J* = 7.9 Hz, 1H), 3.40 – 3.34 (m, 2H), 2.08 – 2.03 (m, 2H), 1.90 – 1.76 (m, 2H), 1.63 – 1.55 (m, 2H).

^{13}C NMR (126 MHz, $CDCl_3$) δ 171.0, 133.44, 133.39, 132.5, 128.5, 128.3, 127.9, 127.7, 127.7, 126.27, 125.9, 89.7, 57.8, 41.5, 34.2, 27.0, 23.6.

IR (KBr) v 2961, 1821, 1509, 1167, 1102, 824, 804, 752, 476.

HRMS (ESI) *m/z*: Calcd for $C_{17}H_{15}O_2$ ($[M-H]^-$) 251.1077, found 251.1078.



2l (QY-466)

(1*S*,5*S*)-5-(benzo[d][1,3]dioxol-5-ylmethyl)-6-oxabicyclo[3.2.0]heptan-7-one

48 mg, 65% yield. Reaction time: 36 h. White solid, m.p. 78-79 ºC. R_f = 0.28 (petroleum ether/ethyl acetate, 5:1). $[\alpha]_D^{26}$ +10.4 (*c* = 1.37, $CHCl_3$).

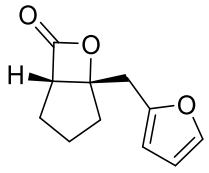
HPLC analysis: 92% ee (Daicel CHIRALPAK IC column, 25 ºC, 210 nm, hexane/*i*-PrOH = 95:5, 1.0 mL/min, 34.82 min (minor), 39.18 min (major)).

^{1}H NMR (500 MHz, $CDCl_3$) δ 6.76 – 6.72 (m, 2H), 6.68 – 6.66 (m, 1H), 5.94 (s, 2H), 3.48 (d, *J* = 7.8 Hz, 1H), 3.14 (s, 2H), 2.09 – 2.02 (m, 2H), 1.94 – 1.88 (m, 1H), 1.87 – 1.77 (m, 1H), 1.66 – 1.52 (m, 2H).

^{13}C NMR (126 MHz, $CDCl_3$) δ 171.0, 147.7, 146.7, 129.4, 122.8, 110.1, 108.3, 101.0, 89.7, 57.5, 41.0, 34.1, 27.0, 23.6.

IR (KBr) v 2962, 1812, 1490, 1443, 1248, 1038, 927, 803.

HRMS (ESI) *m/z*: Calcd for $C_{14}H_{13}O_4$ ($[M-H]^-$) 245.0819, found 245.0820.



2m (QY-460)

(1*S*,5*S*)-5-(furan-2-ylmethyl)-6-oxabicyclo[3.2.0]heptan-7-one

39 mg, 68% yield. Reaction time: 36 h. Yellow oil. R_f = 0.29 (petroleum ether/ethyl acetate, 10:1). $[\alpha]_D^{26}$ +40.9 (*c* = 1.06, $CHCl_3$).

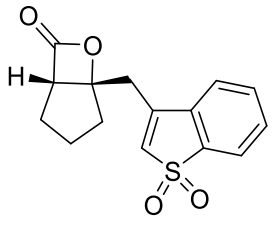
HPLC analysis: 93% ee (Daicel CHIRALPAK IC column, 25 ºC, 210 nm, hexane/*i*-PrOH = 98:2, 1.0 mL/min, 23.10 min (minor), 25.92 min (major)).

^{1}H NMR (500 MHz, $CDCl_3$) δ 7.34 (br, 1H), 6.32 (br, 1H), 6.17 (d, J = 3.0 Hz, 1H), 3.65 (d, J = 7.9 Hz, 1H), 3.34 – 3.27 (m, 2H), 2.14 – 2.07 (m, 2H), 1.95 – 1.80 (m, 2H), 1.67 – 1.56 (m, 2H).

^{13}C NMR (126 MHz, $CDCl_3$) δ 170.9, 149.9, 142.1, 110.5, 108.3, 88.0, 58.1, 33.9, 33.7, 27.0, 23.6.

IR (KBr) v 2964, 1822, 1167, 1147, 1104, 1011, 926, 805, 745.

HRMS (ESI) *m/z*: Calcd for $C_{11}H_{11}O_3$ ($[M-H]^-$) 191.0714, found 191.0711.



2n (QY-473)

(1*S*,5*S*)-5-((1,1-dioxidobenzo[b]thiophen-3-yl)methyl)-6-oxabicyclo[3.2.0]heptan-7-one

28 mg (0.2mmol scale), 49% yield. Colorless oil. R_f = 0.32 (petroleum ether/ethyl acetate, 5:1). $[\alpha]_D^{26}$ +19.0 (*c* = 0.51, $CHCl_3$).

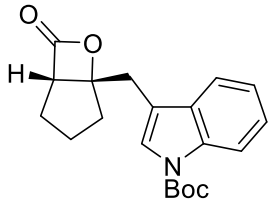
HPLC analysis: 91% ee (Daicel CHIRALPAK IC column, 25 ºC, 210 nm, hexane/*i*-PrOH = 95:5, 1.0 mL/min, 20.55 min (minor), 27.99 min (major)).

^{1}H NMR (400 MHz, $CDCl_3$) δ 7.88 – 7.86 (m, 1H), 7.76 – 7.74 (m, 1H), 7.43 – 7.35 (m, 2H), 7.28 (s, 1H), 3.55 – 3.50 (m, 3H), 2.17 – 2.06 (m, 2H), 1.95 – 1.77 (m, 2H), 1.68 – 1.56 (m, 2H).

^{13}C NMR (101 MHz, $CDCl_3$) δ 170.9, 140.3, 138.9, 130.3, 124.9, 124.4, 124.3, 123.0, 121.7, 89.3, 58.0, 34.5, 33.6, 27.0, 23.7.

IR (KBr) v 2922, 1810, 1427, 1166, 1102, 802, 760, 735.

HRMS (ESI) *m/z*: Calcd for $C_{15}H_{13}O_4S$ ($[M-H]^-$) 289.0533, found 289.0540.



2o (QY-448)

tert-butyl 3-(((1*S*,5*S*)-7-oxo-6-oxabicyclo[3.2.0]heptan-5-yl)methyl)-1H-indole-1-carboxylate

27 mg (0.2mmol scale), 55% yield. Reaction time: 72 h. Wax. R_f = 0.32 (petroleum ether/ethyl acetate, 7:1). $[\alpha]_D^{26}$ +2.1 (*c* = 0.87, $CHCl_3$).

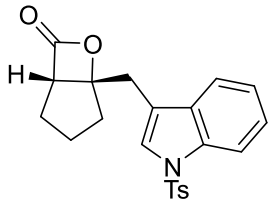
HPLC analysis: 88% ee (Daicel CHIRALPAK IC column, 25 ºC, 210 nm, hexane/*i*-PrOH = 96:4, 1.0 mL/min, 24.25 min (minor), 29.48 min (major)).

^{1}H NMR (500 MHz, $CDCl_3$) δ 8.13 (br, 1H), 7.53 (d, *J* = 7.8 Hz, 1H), 7.49 (s, 1H), 7.34 – 7.31 (m, 1H), 7.27 – 7.24 (m, 1H), 3.56 (d, *J* = 7.9 Hz, 1H), 3.37 – 3.31 (m, 2H), 2.14 (dd, *J* = 14.4, 6.0 Hz, 1H), 2.08 (dd, *J* = 13.4, 5.9 Hz, 1H), 1.94 – 1.79 (m, 2H), 1.67 – 1.60 (s, 11H).

^{13}C NMR (126 MHz, $CDCl_3$) δ 171.0, 149.6, 135.3, 130.5, 124.7, 124.6, 122.7, 119.0, 115.3, 114.7, 89.4, 83.9, 57.9, 34.4, 30.6, 28.2, 27.0, 23.7.

IR (KBr) v 2976, 2933, 1823, 1733, 1453, 1369, 1256, 1157, 1081, 748.

HRMS (ESI) *m/z*: Calcd for $C_{20}H_{24}O_4N$ ($[M+H]^+$) 342.1700, found 342.1696.



2p (QY-458')

(1*S*,5*S*)-5-((1-tosyl-1H-indol-3-yl)methyl)-6-oxabicyclo[3.2.0]heptan-7-one

45 mg (0.2mmol scale), 57% yield. Reaction time: 72 h. Yellow solid, m.p. 121-122 ºC. R_f = 0.36 (petroleum ether/dichloromethane/ethyl acetate, 18:12:1). $[\alpha]_D^{26}$ -13.4 (*c* = 0.79, $CHCl_3$).

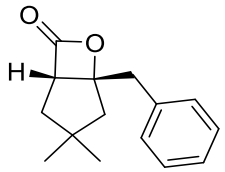
HPLC analysis: 96% ee (Daicel CHIRALPAK IC column, 25 ºC, 254 nm, hexane/*i*-PrOH = 80:20, 1.0 mL/min, 38.26 min (minor), 44.77 min (major)).

^{1}H NMR (500 MHz, $CDCl_3$) δ 7.97 (d, *J* = 8.3 Hz, 1H), 7.72 (d, *J* = 8.3 Hz, 2H), 7.49 (d, *J* = 8.5 Hz, 2H), 7.33 – 7.30 (m, 1H), 7.26 – 7.20 (m, 3H), 3.45 (d, *J* = 7.9 Hz, 1H), 3.31 (s, 2H), 2.33 (s, 3H), 2.09 – 2.04 (m, 2H), 1.93 – 1.76 (m, 2H), 1.63 – 1.52 (m, 2H).

^{13}C NMR (126 MHz, $CDCl_3$) δ 170.7, 145.0, 135.04, 135.02, 130.8, 129.9, 126.8, 125.1, 125.0, 123.4, 119.6, 116.8, 113.8, 89.1, 57.9, 34.4, 30.6, 27.0, 23.7, 21.6.

IR (KBr) v 2962, 2926, 1822, 1448, 1366, 1174, 1121, 804, 748.

HRMS (ESI) *m/z*: Calcd for $C_{22}H_{22}O_4NS$ ($[M+H]^+$) 396.1264, found 396.1261.

 **2q** (QY-467)

(1*S*,5*S*)-5-benzyl-3,3-dimethyl-6-oxabicyclo[3.2.0]heptan-7-one

47 mg, 61% yield. Reaction time: 36 h. Colorless oil. R_f = 0.28 (petroleum ether/ethyl acetate, 10:1). $[\alpha]_D^{26}$ +0.0 (*c* = 0.21, $CHCl_3$).

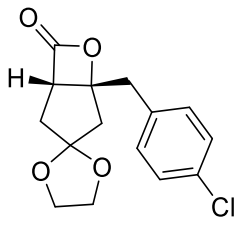
HPLC analysis: 92% ee (Daicel CHIRALPAK IB column, 25 ºC, 210 nm, hexane/*i*-PrOH = 99.5:0.5, 1.0 mL/min, 15.93 min (major), 17.35 min (minor)).

^{1}H NMR (500 MHz, $CDCl_3$) δ 7.33 – 7.23 (m, 5H), 3.50 (d, *J* = 9.1 Hz, 1H), 3.19 (s, 2H), 2.00 – 1.97 (m, 2H), 1.71 – 1.66 (m, 2H), 1.18 (s, 3H), 1.07 (s, 3H).

^{13}C NMR (126 MHz, $CDCl_3$) δ 172.3, 135.7, 129.8, 128.6, 127.1, 91.4, 58.3, 47.6, 42.4, 41.0, 40.7, 31.6, 28.7.

IR (KBr) v 2922, 1818, 1456, 1341, 1116, 1030, 957, 809, 701.

HRMS (ESI) *m/z*: Calcd for $C_{15}H_{17}O_2$ ($[M-H]^-$) 229.1234, found 229.1232.

 **2r** (QY-439)

(1*S*,5*R*)-5-(4-chlorobenzyl)-6-oxaspiro[bicyclo[3.2.0]heptane-3,2'-[1,3]dioxolan]-7-one

47 mg (0.2mmol scale), 80% yield. Reaction time: 36 h. White solid, m.p. 139-140 ºC. R_f = 0.14 (petroleum ether/ethyl acetate, 3:1). $[\alpha]_D^{26}$ -0.9 (*c* = 0.33, $CHCl_3$).

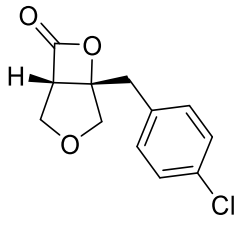
HPLC analysis: 87% ee (Daicel CHIRALPAK IC column, 25 ºC, 210 nm, hexane/*i*-PrOH = 90:10, 1.0 mL/min, 41.70 min (major), 44.39 min (minor)).

^{1}H NMR (500 MHz, $CDCl_3$) δ 7.29 (d, *J* = 8.3 Hz, 2H), 7.17 (d, *J* = 8.2 Hz, 2H), 4.02 – 3.94 (m, 2H), 3.89 – 3.81 (m, 2H), 3.57 (d, *J* = 8.9 Hz, 1H), 3.18 (s, 2H), 2.29 – 2.21 (m, 2H), 2.05 – 2.01 (m, 2H).

^{13}C NMR (126 MHz, $CDCl_3$) δ 169.7, 133.5, 133.4, 131.0, 128.9, 115.2, 85.2, 65.0, 64.8, 55.4, 42.8, 41.3, 37.3.

IR (KBr) v 2917, 1809, 1113, 1097, 1051, 1009, 807, 729.

HRMS (ESI) *m/z*: Calcd for $C_{15}H_{16}O_4Cl$ ($[M+H]^+$) 295.0732, found 295.0731.

 **2s** (QY-478)

(1*S*,5*S*)-5-(4-chlorobenzyl)-3,6-dioxabicyclo[3.2.0]heptan-7-one

40 mg, 56% yield. Reaction time: 36 h. White solid, m.p. 80-81 °C. R_f = 0.23 (petroleum ether/tetrahydrofuran/
dichloromethane, 20:5:1). $[\alpha]_D^{26}$ -21.6 (*c* = 0.45, $CHCl_3$).

HPLC analysis: 69% ee (Daicel CHIRALPAK IC column, 25 °C, 210 nm, hexane/*i*-PrOH = 90:10, 1.0 mL/min, 28.69 min (minor), 38.64 min (major)).

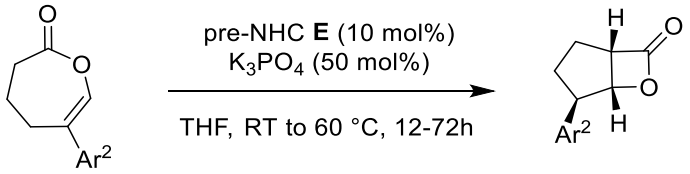
^{1}H NMR (500 MHz, $CDCl_3$) δ 7.32 (d, *J* = 8.2 Hz, 2H), 7.19 (d, *J* = 8.3 Hz, 2H), 4.34 (d, *J* = 10.1 Hz, 1H), 4.10 (d, *J* = 11.3 Hz, 1H), 3.68 (d, *J* = 5.3 Hz, 1H), 3.61 (dd, *J* = 10.1, 5.3 Hz, 1H), 3.50 (d, *J* = 11.3 Hz, 1H), 3.29 (s, 2H).

^{13}C NMR (126 MHz, $CDCl_3$) δ 167.9, 133.7, 133.0, 130.9, 129.1, 87.2, 72.8, 67.9, 58.7, 36.8.

IR (KBr) v 2921, 1824, 1492, 1168, 1064, 818, 762.

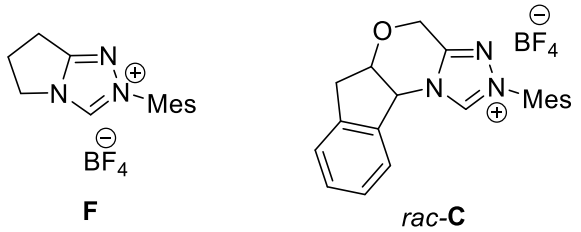
HRMS (ESI) *m/z*: Calcd for $C_{12}H_{10}O_3Cl$ ($[M-H]^-$) 237.0324, found 237.0323.

3.2 Synthesis from *endo*-enollactones

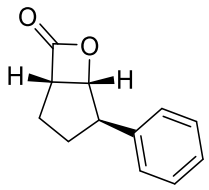


Typical procedure: To an oven-dried 25 mL Schlenk tube equipped with a stir bar was charged with enollactone **3a** (Ar^2 = Ph, 56.4 mg, 0.3 mmol), preNHC **E** (15.1 mg, 10 mol%) and K_3PO_4 (31.8 mg, 50 mol%). This tube was closed with a septum, evacuated, back-filled with nitrogen for three times. To this mixture was added freshly distilled dry THF (3 mL). The reaction mixture was stirred at room temperature until the full consumption of enol lactone **3a**. The reaction mixture was concentrated under reduced pressure, and the residue was purified by column chromatography on silica gel (petroleum ether/EtOAc = 10:1 as the eluent) to give the product **2a** (34 mg, 60% yield, 89% ee).

For compound **4a**, **4d** and **4e** racemic samples for the chiral phase HPLC analysis were prepared using achiral NHC precursor **F** under the same conditions. For compound **4b** and **4c**, racemic samples were prepared using achiral NHC precursor **F** (*rac*-1) and racemic NHC precursor **C** (*rac*-2).



Characterization Data of β-lactones **4**



4a (QY-476)

(1*S*,4*R*,5*R*)-4-phenyl-6-oxabicyclo[3.2.0]heptan-7-one

34 mg, 12:1 dr, 60% yield. Reaction time: 24 h. Yellow oil. R_f = 0.30 (petroleum ether/ethyl acetate, 10:1). $[\alpha]_D^{26}$ +17.1 (*c* = 0.68, $CHCl_3$).

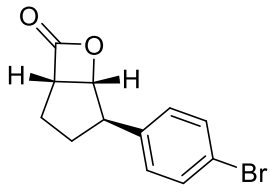
HPLC analysis: 89% ee (Daicel CHIRALPAK IC column, 25 ºC, 210 nm, hexane/*i*-PrOH = 98:2, 1.0 mL/min, 29.79 min (minor), 36.54 min (major)).

^{1}H NMR (400 MHz, $CDCl_3$) δ 7.35 – 7.31 (m, 2H), 7.27 – 7.23 (m, 1H), 7.07 – 7.05 (m, 2H), 5.10 (d, *J* = 3.7 Hz, 1H), 4.06 (dd, *J* = 8.2, 3.7 Hz, 1H), 3.64 (d, *J* = 7.2 Hz, 1H), 2.40 – 2.30 (m, 1H), 2.20 (dd, *J* = 13.7, 7.0 Hz, 1H), 2.10 (dd, *J* = 13.5, 6.9 Hz, 1H), 2.02 – 1.91 (m, 1H).

^{13}C NMR (101 MHz, $CDCl_3$) δ 171.0, 140.3, 128.9, 127.0, 127.0, 81.4, 56.6, 46.8, 29.9, 25.1.

IR (KBr) v 2920, 1829, 1112, 963, 899, 747, 700.

HRMS (ESI) *m/z*: Calcd for $C_{12}H_{11}O_2$ ($[M-H]^-$) 187.0765, found 187.0763.



4b (QY-510)

(1*S*,4*R*,5*R*)-4-(4-bromophenyl)-6-oxabicyclo[3.2.0]heptan-7-one

21 mg (0.2mmol scale), > 20:1 dr, 40% yield. Reaction time: 24 h. White solid, m.p. 88-89 ºC. R_f = 0.29 (petroleum ether/ethyl acetate, 10:1). $[\alpha]_D^{26}$ +14.9 (*c* = 0.32, $CHCl_3$).

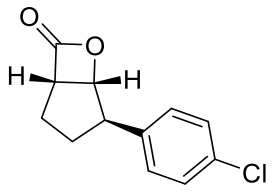
HPLC analysis: 84% ee (Daicel CHIRALPAK IC column, 25 ºC, 210 nm, hexane/*i*-PrOH = 98:2, 1.0 mL/min, 37.14 min (minor), 47.50 min (major)).

^{1}H NMR (500 MHz, $CDCl_3$) δ 7.45 (d, *J* = 8.4 Hz, 2H), 6.94 (d, *J* = 8.3 Hz, 2H), 5.05 (d, *J* = 3.6 Hz, 1H), 4.06 (dd, *J* = 8.1, 3.6 Hz, 1H), 3.60 (d, *J* = 7.2 Hz, 1H), 2.40 – 2.31 (m, 1H), 2.21 (dd, *J* = 13.8, 7.0 Hz, 1H), 2.06 (dd, *J* = 13.6, 6.9 Hz, 1H), 1.96 – 1.88 (m, 1H).

^{13}C NMR (126 MHz, $CDCl_3$) δ 170.6, 139.2, 132.0, 128.7, 120.9, 80.9, 56.6, 46.3, 29.8, 25.0.

IR (KBr) v 2921, 1833, 1817, 1490, 1110, 1009, 963, 890, 825.

HRMS (ESI) *m/z*: Calcd for $C_{12}H_{10}O_2Br$ ($[M-H]^-$) 264.9870, found 264.9871.



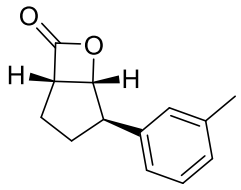
4c (QY-500)

(1*S*,4*R*,5*R*)-4-(4-chlorophenyl)-6-oxabicyclo[3.2.0]heptan-7-one

28 mg, 9:1 dr, 41% yield. Reaction time: 24 h. Orange solid, m.p. 59-62 ºC. R_f = 0.38 (petroleum ether/ethyl acetate, 7:1). $[\alpha]_D^{26}$ +20.2 (*c* = 0.66, $CHCl_3$).

HPLC analysis: 83% ee (Daicel CHIRALPAK IC column, 25 ºC, 210 nm, hexane/*i*-PrOH = 98:2, 1.0 mL/min, 34.88 min (minor), 44.52 min (major)).

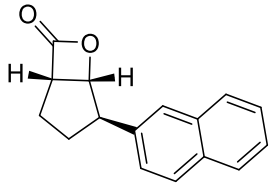
^{1}H NMR (500 MHz, $CDCl_3$) δ 7.30 (d, *J* = 8.3 Hz, 2H), 6.99 (d, *J* = 8.3 Hz, 2H), 5.05 (d, *J* = 3.6 Hz, 1H), 4.06 (dd, *J* = 8.1, 3.6 Hz, 1H), 3.61 (d, *J* = 7.2 Hz, 1H), 2.39 – 2.31 (m, 1H), 2.21 (dd, *J* = 13.8, 7.0 Hz, 1H), 2.06 (dd, *J* = 13.6, 6.9 Hz, 1H), 1.96 – 1.88 (m, 1H).
^{13}C NMR (126 MHz, $CDCl_3$) δ 170.7, 138.7, 132.9, 129.0, 128.3, 81.0, 56.5, 46.3, 29.9, 25.0.
IR (KBr) v 2977, 1844, 1493, 1116, 965, 919, 839, 827.
HRMS (ESI) *m/z*: Calcd for $C_{12}H_{11}O_2Cl$ ($[M-H]^-$) 221.0375, found 221.0375.



4d (QY-491)

(1*S*,4*R*,5*R*)-4-(m-tolyl)-6-oxabicyclo[3.2.0]heptan-7-one

39 mg, 11:1 dr, 64% yield. Reaction time: 36 h. yellow oil. R_f = 0.33 (petroleum ether/ethyl acetate, 10:1). $[\alpha]_D^{26}$ +20.1 (*c* = 0.78, $CHCl_3$).
HPLC analysis: 88% ee (Daicel CHIRALPAK IC column, 25 °C, 210 nm, hexane/*i*-PrOH = 98:2, 1.0 mL/min, 25.86 min (minor), 30.54 min (major)).
^{1}H NMR (400 MHz, $CDCl_3$) δ 7.21 (t, *J* = 7.6 Hz, 1H), 7.07 (d, *J* = 7.6 Hz, 1H), 6.87 – 6.83 (m, 2H), 5.09 (d, *J* = 3.6 Hz, 1H), 4.05 (dd, *J* = 8.2, 3.7 Hz, 1H), 3.60 (d, *J* = 7.2 Hz, 1H), 2.39 – 2.28 (m, 4H), 2.19 (dd, *J* = 13.6, 7.0 Hz, 1H), 2.09 (dd, *J* = 13.5, 6.9 Hz, 1H), 2.02 – 1.91 (m, 1H).
^{13}C NMR (101 MHz, $CDCl_3$) δ 171.1, 140.2, 138.6, 128.7, 127.9, 127.7, 123.9, 81.4, 56.6, 46.7, 29.9, 25.2, 21.5.
IR (KBr) v 2963, 1834, 1133, 1113, 966, 916, 847, 790, 709.
HRMS (ESI) *m/z*: Calcd for $C_{13}H_{15}O_2$ ($[M+H]^+$) 203.1067, found 203.1066.

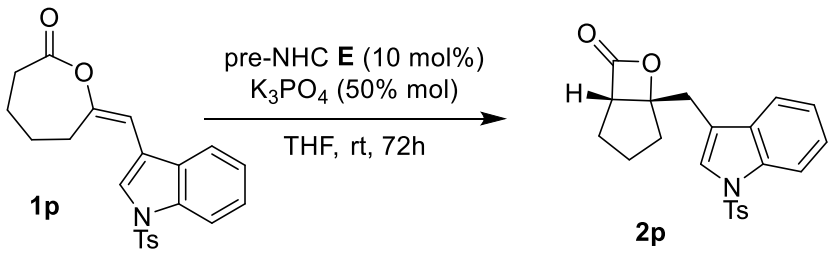


4e (QY-509)

(1*S*,4*R*,5*R*)-4-(naphthalen-2-yl)-6-oxabicyclo[3.2.0]heptan-7-one

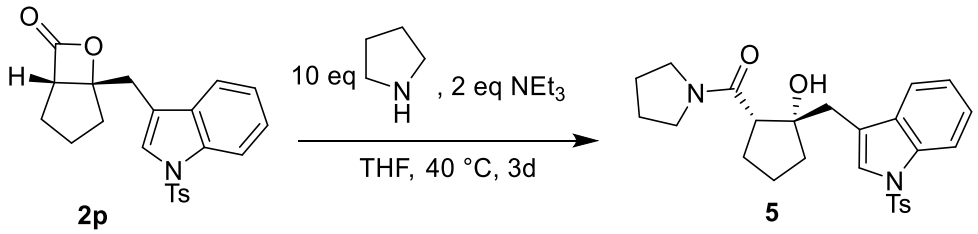
38 mg, > 20:1 dr, 53% yield. Reaction time: 24 h. White solid, m.p. 129-130 °C. R_f = 0.30 (petroleum ether/dichloromethane/ethyl acetate, 35:7:1). $[\alpha]_D^{26}$ +8.5 (*c* = 0.36, $CHCl_3$).
HPLC analysis: 81% ee (Daicel CHIRALPAK IC column, 25 °C, 210 nm, hexane/*i*-PrOH = 98:2, 1.0 mL/min, 42.05 min (minor), 48.97 min (major)).
^{1}H NMR (500 MHz, $CDCl_3$) δ 7.83 – 7.77 (m, 3H), 7.51 – 7.46 (m, 2H), 7.41 (s, 1H), 7.26 – 7.24 (m, 1H), 5.22 (d, *J* = 3.6 Hz, 1H), 4.12 (dd, *J* = 8.1, 3.6 Hz, 1H), 3.80 (d, *J* = 7.2 Hz, 1H), 2.46 – 2.38 (m, 1H), 2.26 – 2.18 (m, 2H), 2.07 – 1.99 (m, 1H).
^{13}C NMR (126 MHz, $CDCl_3$) δ 171.0, 137.5, 133.3, 132.2, 128.7, 127.6, 127.6, 126.5, 126.0, 126.0, 124.7, 81.2, 56.6, 46.9, 29.8, 25.1.
IR (KBr) v 2928, 1839, 1272, 1106, 972, 910, 898, 847, 773.
HRMS (ESI) *m/z*: Calcd for $C_{16}H_{15}O_2$ ($[M+H]^+$) 239.1067, found 239.1067.

4. Gram-scale synthesis.



To an oven-dried 100 mL Schlenk tube equipped with a stir bar was charged with (*E*)-7-((1-tosyl-1H-indol-3-yl)methylene)oxepan-2-one **1n** (1.98 g, 5 mmol), preNHC **E** (0.25 g, 10 mol%) and K_3PO_4 (0.53 g, 50 mol%). This tube was closed with a septum, evacuated, back-filled with nitrogen for three times. To this mixture was added freshly distilled dry THF (75 mL). The reaction was complete after stirring at room temperature for 72 h, as indicated by TLC. The reaction mixture was concentrated under reduced pressure, and the residue was purified by column chromatography on silica gel (petroleum ether/DCM/EtOAc = 18:12:1 as the eluent) to give the product **2a** (1.17 g, 59% yield, 96% ee).

5. Chemical transformations of bicyclic β-lactones.



To a 10 mL Schlenk flask was added (1S,5S)-5-((1-tosyl-1H-indol-3-yl)methyl)-6-oxabicyclo[3.2.0]heptan-7-one **2n** (79 mg, 0.2 mmol), pyrrolidine (83 μL, 5 eq), triethylamine (28 μL, 1 eq) and dry THF (3 mL) under N_2 atmosphere. After stirring at 40 °C for 41 h, the reaction mixture was added a further pyrrolidine (83 μL, 5 eq) and triethylamine (28 μL, 1 eq). The reaction mixture was stirred at 40 °C for 34 h. The reaction mixture was concentrated under reduced pressure. The residue was purified by column chromatography (DCM/MeOH = 50:1) to give **5** (QY-477) as colorless wax (89 mg, 95% yield, 96% ee).

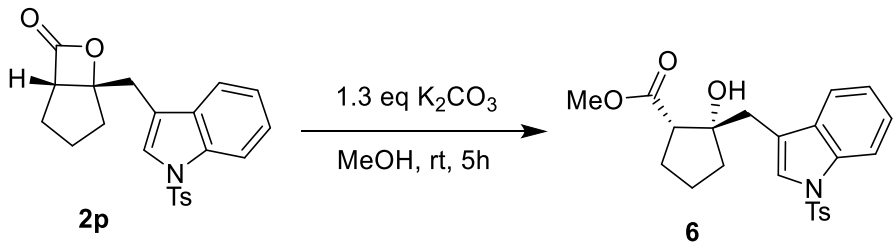
R_f = 0.29 (dichloromethane/methanol, 50:1). $[\alpha]_D^{26}$ -20.2 (*c* = 1.26, $CHCl_3$).

HPLC analysis: 96% ee (Daicel CHIRALPAK IC column, 25 °C, 254 nm, hexane/*i*-PrOH = 50:50, 1.0 mL/min, 20.18 min (major), 26.21 min (minor)).

^{1}H NMR (500 MHz, $CDCl_3$) δ 7.95 (d, *J* = 8.3 Hz, 1H), 7.73 (d, *J* = 8.2 Hz, 2H), 7.48 (d, *J* = 7.8 Hz, 1H), 7.37 (s, 1H), 7.28 (t, *J* = 7.7 Hz, 1H), 7.20 – 7.17 (m, 3H), 3.30 – 3.26 (m, 1H), 3.20 – 3.15 (m, 1H), 3.05 – 2.97 (m, 3H), 2.65 – 2.60 (m, 1H), 2.32 (s, 3H), 2.20 – 2.16 (m, 1H), 1.98 – 1.57 (m, 10H).

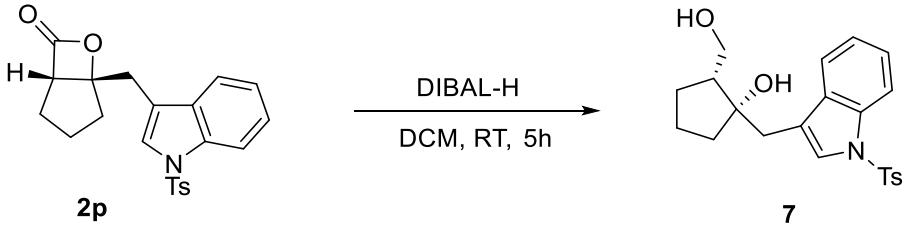
^{13}C NMR (126 MHz, $CDCl_3$) δ 174.6, 144.8, 135.1, 135.0, 131.7, 129.8, 126.8, 124.5, 124.4, 122.8, 119.8, 119.6, 113.6, 83.0, 47.1, 46.1, 45.3, 38.1, 34.6, 27.6, 25.6, 24.0, 21.5, 21.4.

IR (KBr) v 2971, 1609, 1448, 1362, 1173, 1120, 750, 670, 578.
HRMS (ESI) *m/z*: Calcd for $C_{26}H_{31}O_4N_2S$ ($[M+H]^+$) 467.1999, found 467.1992.



To a 10 mL Schlenk flask was added (1S,5S)-5-((1-tosyl-1H-indol-3-yl)methyl)-6-oxabicyclo[3.2.0]heptan-7-one **2n** (79 mg, 0.2 mmol), K_2CO_3 (36 mg, 1.3 eq) and MeOH (3 mL) under N_2 atmosphere. The reaction mixture was stirred at room temperature for 5 h. TLC showed that the reduction reaction was complete. The reaction mixture was concentrated under reduced pressure. The residue was purified by column chromatography (petroleum ether/acetone = 5:1) to give **6** (QY-515') as colorless wax (74 mg, 86% yield, 96% ee).

R_f = 0.29 (petroleum ether/acetone, 5:1). $[\alpha]_D^{26}$ -8.6 (*c* = 1.83, $CHCl_3$).
HPLC analysis: 96% ee (Daicel CHIRALPAK AD-H column, 25 ºC, 254 nm, hexane/*i*-PrOH = 90:10, 1.0 mL/min, 22.74 min (major), 27.16 min (minor)).
^{1}H NMR (500 MHz, $CDCl_3$) δ 7.96 (d, *J* = 8.3 Hz, 1H), 7.74 (d, *J* = 8.3 Hz, 2H), 7.52 (d, *J* = 7.8 Hz, 1H), 7.45 (s, 1H), 7.28 (t, *J* = 7.6 Hz, 1H), 7.23 – 7.18 (m, 3H), 3.76 (s, 1H), 3.47 (s, 3H), 3.05 – 2.98 (m, 2H), 2.59 (t, *J* = 9.6 Hz, 1H), 2.31 (s, 3H), 2.06 – 1.83 (m, 3H), 1.78 – 1.73 (m, 1H), 1.67 – 1.55 (m, 2H).
^{13}C NMR (126 MHz, $CDCl_3$) δ 175.9, 144.8, 135.2, 135.0, 131.7, 129.8, 126.8, 125.0, 124.6, 123.1, 120.0, 118.8, 113.6, 82.4, 51.6, 50.8, 38.7, 35.0, 28.5, 21.7, 21.5.
IR (KBr) v 2953, 1714, 1447, 1363, 1173, 747, 670, 571.
HRMS (ESI) *m/z*: Calcd for $C_{23}H_{25}O_5NNaS$ ($[M+Na]^+$) 450.1346, found 450.1339.



To a (1S,5S)-5-((1-tosyl-1H-indol-3-yl)methyl)-6-oxabicyclo[3.2.0]heptan-7-one **2n** (79 mg, 0.2 mmol) solution in DCM (0.067 M) was added DIBAL-H (0.67 mL, 1.5 M solution in toluene, 5 eq) dropwise under N_2 atmosphere at 0 °C. Then, the reaction mixture was stirred at room temperature for 5 h. TLC showed that the reduction reaction was complete. The reaction mixture was quenched with an aqueous Rochelle's salt solution. The resulting suspension was stirred vigorously for 2 h. The precipitate was filtered off with celite. The filtrate was poured into a separatory funnel. The layers were separated and aqueous layer was extracted with DCM (2 x 20 mL) and the combined organic layers were dried over$MgSO_4$, filtered, and concentrated under reduced pressure. The

residue was purified by column chromatography (petroleum ether/acetone = 3:1) to give **7** (QY-517) as colorless wax (67 mg, 83% yield, 93% ee).

R_f = 0.31 (petroleum ether/acetone, 3:1). $[\alpha]_D^{26}$ -11.7 (*c* = 1.35, $CHCl_3$).

HPLC analysis: 93% ee (Daicel CHIRALPAK AD-H column, 25 ºC, 254 nm, hexane/*i*-PrOH = 70:30, 1.0 mL/min, 7.98 min (minor), 9.82 min (major)).

^{1}H NMR (400 MHz, $CDCl_3$) δ 7.98 (d, *J* = 8.2 Hz, 1H), 7.74 (d, *J* = 8.3 Hz, 2H), 7.55 (d, *J* = 7.8 Hz, 1H), 7.47 (s, 1H), 7.32 – 7.18 (m, 4H), 3.85 (dd, *J* = 11.2, 3.4 Hz, 1H), 3.72 (dd, *J* = 11.1, 6.0 Hz, 1H), 3.08 (d, *J* = 14.4 Hz, 1H), 2.83 (d, *J* = 14.3 Hz, 1H), 2.60 (br, 2H), 2.31 (s, 3H), 1.95 – 1.88 (m, 1H), 1.78 – 1.67 (m, 4H), 1.56 – 1.45 (m, 2H).

^{13}C NMR (101 MHz, $CDCl_3$) δ 144.9, 135.2, 135.1, 131.8, 129.8, 126.8, 124.9, 124.7, 123.2, 120.1, 119.1, 113.7, 83.5, 62.7, 48.0, 40.4, 35.9, 26.6, 22.0, 21.5.

IR (KBr) v 2926, 1447, 1363, 1173, 1120, 747, 671, 572, 538.

HRMS (APCI) *m/z*: Calcd for $C_{22}H_{24}O_4NS$ ($[M-H]^-$) 398.1431, found 398.1433.

6. X-ray Structure of compound 2c and 4b.

The crystal suitable for X-ray analysis was prepared by slow evaporation of the solvent of the solution of **2c** or **4b** in dichloromethane/hexane at room temperature (Figure S1 & S2).

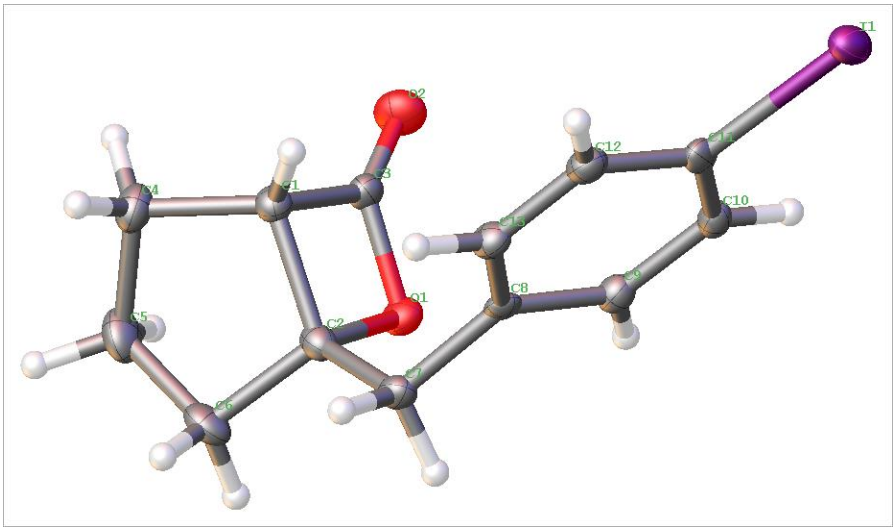


Figure S1. The absolute structure of **2c**.

Table S2. Crystal data and structure refinement for **2c**.

Empirical formula	C13H13IO2
Formula weight	328.13
Temperature/K	169.99(11)
Crystal system	monoclinic
Space group	P21

a/Å	5.72921(5)
b/Å	19.8664(2)
c/Å	16.68090(13)
α/°	90
β/°	92.5198(8)
γ/°	90
Volume/Å3	1896.76(3)
Z	6
ρcalcg/cm3	1.724
μ/mm-1	19.766
F(000)	960.0
Crystal size/mm3	0.15 × 0.12 × 0.06
Radiation	Cu Kα (λ = 1.54184)
2Θ range for data collection/°	5.302 to 150.6
Index ranges	-7 ≤ h ≤ 7, -24 ≤ k ≤ 21, -20 ≤ l ≤ 20
Reflections collected	33429
Independent reflections	7200 [Rint = 0.0404, Rsigma = 0.0290]
Data/restraints/parameters	7200/1/433
Goodness-of-fit on F2	1.064
Final R indexes [I>=2σ (I)]	R1 = 0.0403, wR2 = 0.1085
Final R indexes [all data]	R1 = 0.0410, wR2 = 0.1092
Largest diff. peak/hole / e Å-3	0.80/-1.59
Flack parameter	-0.011(7)

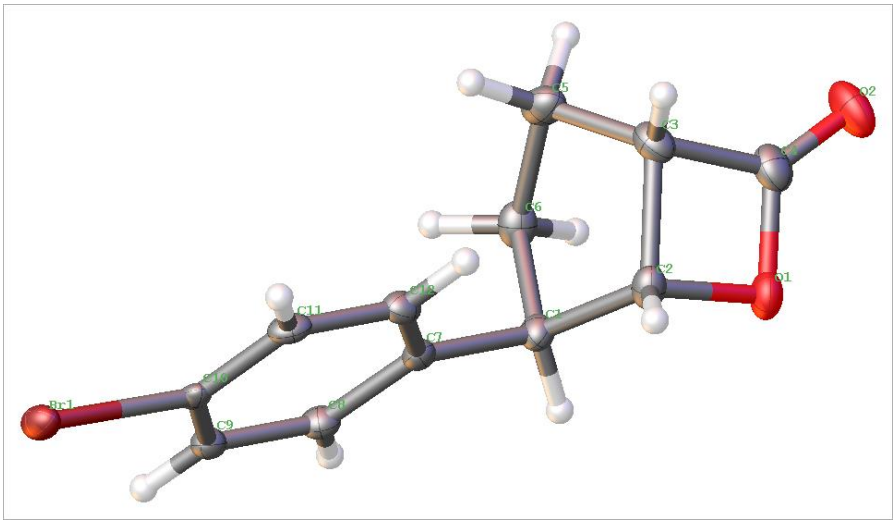


Figure S2. The absolute structure of **4b**.

Table S3 Crystal data and structure refinement for **4b**.

Empirical formula	C12H11BrO2
Formula weight	267.12
Temperature/K	170.00(10)
Crystal system	orthorhombic
Space group	P212121
a/Å	6.0227(3)
b/Å	6.9189(3)
c/Å	25.7398(17)
α/°	90
β/°	90
γ/°	90
Volume/Å3	1072.59(10)
Z	4
ρcalcg/cm3	1.654
μ/mm-1	3.808
F(000)	536.0
Crystal size/mm3	0.23 × 0.11 × 0.01
Radiation	Mo Kα (λ = 0.71073)
2Θ range for data collection/°	3.164 to 62.104
Index ranges	-8 ≤ h ≤ 7, -9 ≤ k ≤ 9, -36 ≤ l ≤ 30
Reflections collected	19510
Independent reflections	2996 [Rint = 0.0767, Rsigma = 0.0588]
Data/restraints/parameters	2996/0/136
Goodness-of-fit on F2	1.053
Final R indexes [I>=2σ (I)]	R1 = 0.0555, wR2 = 0.1105
Final R indexes [all data]	R1 = 0.0773, wR2 = 0.1165
Largest diff. peak/hole / e Å-3	0.92/-0.70
Flack parameter	-0.009(11)

7. References

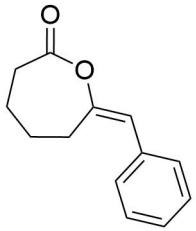
1 (a) M. S. Kerr, J. Read de Alaniz and T. Rovis, A highly enantioselective catalytic intramolecular Stetter

reaction, *J. Am. Chem. Soc.*, 2002, **124**, 10298; (b) M. He, J. R. Struble and J. W. Bode, Highly enantioselective azadiene Diels-Alder reactions catalyzed by chiral N-heterocyclic carbenes, *J. Am. Chem. Soc.*, 2006, **128**, 8418; (c) S. Ye, L. He, Y.-R. Zhang and X.-L. Huang, Chiral Bifunctional N-Heterocyclic Carbenes: Synthesis and Application in the Aza-Morita-Baylis-Hillman Reaction, *Synthesis*, 2008, **2008**, 2825; (d) P. C. Chiang, M. Rommel and J. W. Bode, Alpha'-hydroxyenones as mechanistic probes and scope-expanding surrogates for alpha,beta-unsaturated aldehydes in N-heterocyclic carbene-catalyzed reactions, *J. Am. Chem. Soc.*, 2009, **131**, 8714; (e) C. Zhao, F. Li and J. Wang, N-Heterocyclic Carbene Catalyzed Dynamic Kinetic Resolution of Pyranones, *Angew. Chem. Int. Ed.*, 2016, **55**, 1820; (f) Z. Wu, F. Li and J. Wang, Intermolecular dynamic kinetic resolution cooperatively catalyzed by an N-heterocyclic carbene and a Lewis acid, *Angew. Chem. Int. Ed.*, 2015, **54**, 1629.

2 J. A. Guzmán, V. Mendoza, E. García, C. F. Garibay, L. Z. Olivares and L. A. Maldonado, Baeyer-Villiger Oxidation of β-Aryl Substituted Unsaturated Carbonyl Compounds with Hydrogen Peroxide and Catalytic Selenium Dioxide, *Synth. Commun.*, 1995, **25**, 2121.

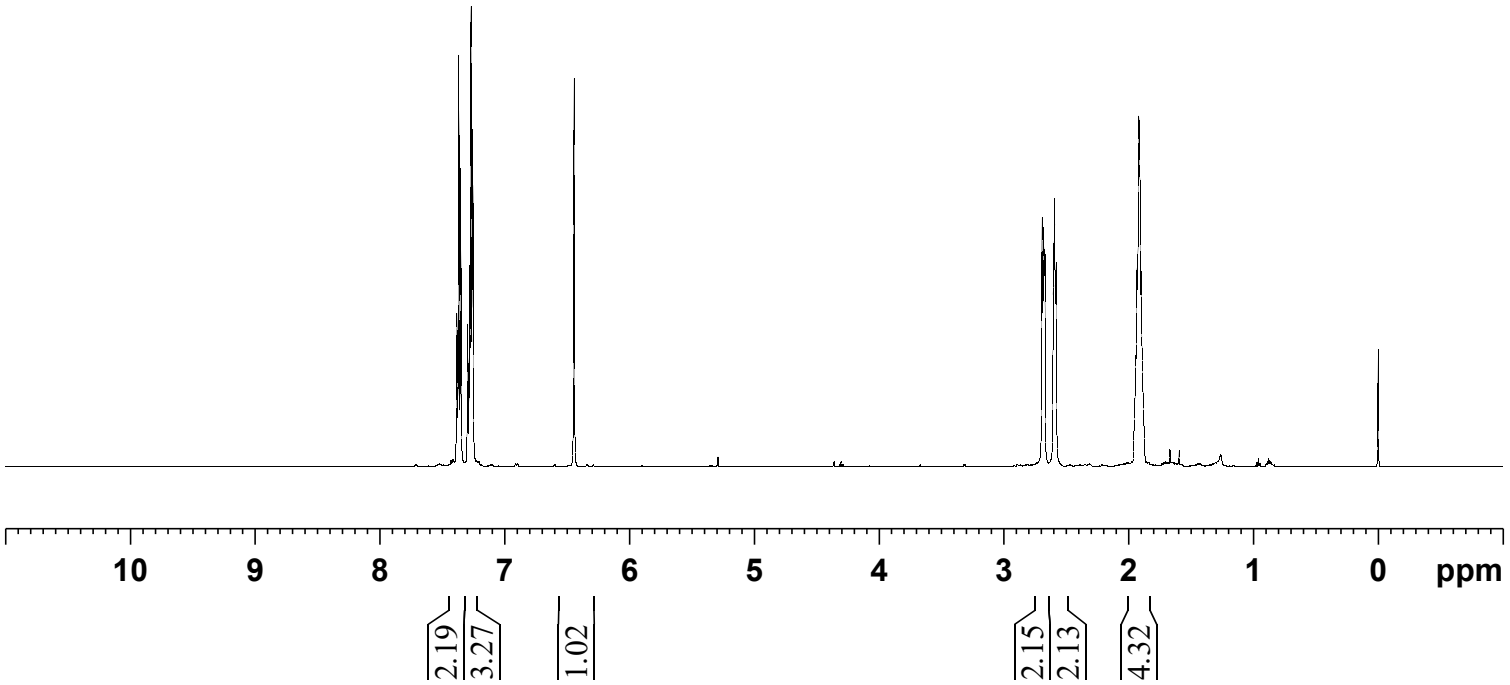
3 J. B. Xie, J. H. Xie, X. Y. Liu, W. L. Kong, S. Li and Q. L. Zhou, Highly enantioselective hydrogenation of alpha-arylmethylene cycloalkanones catalyzed by iridium complexes of chiral spiro aminophosphine ligands, *J. Am. Chem. Soc.*, 2010, **132**, 4538.

4 (a) E. D. Beaulieu, L. Voss and D. Trauner, Conjugate addition of allyl stannanes with concomitant triflation, *Org. Lett.*, 2008, **10**, 869; (b) C. J. Scheuermann née Taylor and C. Jaekel, Enantioselective Hydrogenation of Enones with a Hydroformylation Catalyst, *Adv. Synth. Catal.*, 2008, **350**, 2708; (c) G. Saini, A. Mondal and M. Kapur, Palladium-Mediated Remote Functionalization in gamma- and epsilon-Arylations and Alkenylations of Unblocked Cyclic Enones, *Org. Lett.*, 2019, **21**, 9071.



1a

500 MHz, $CDCl_3$




```
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NAME          500M-2020
EXPNO                 7
PROCNO                1

F2 - Acquisition Parameters
Date_          20191207
Time               9.29
INSTRUM           spect
PROBHD   5 mm CPPBBO BB
PULPROG            zg30
TD                65536
SOLVENT           CDCl3
NS                   16
DS                    2
SWH           10000.000 Hz
FIDRES         0.152588 Hz
AQ            3.2767999 sec
RG                31.72
DW               50.000 usec
DE                6.50 usec
TE               298.2 K
D1           1.00000000 sec
D11      0 sec
TD0                   1

======== CHANNEL f1 ========
SFO1        500.1330885 MHz
NUC1                   1H
P1                 10.59 usec
PLW1        20.00000000 W

======== CHANNEL f2 ========
SFO2        500.1330885 MHz
NUC2                  off
CPDPRG[2
PCPD2     0 usec
PLW2      0 W
PLW12     0 W
PLW13     0 W

F2 - Processing parameters
SI                 65536
SF          500.1300118 MHz
WDW                   EM
SSB       0
LB                 0.30 Hz
GB        0
PC                 1.00
```

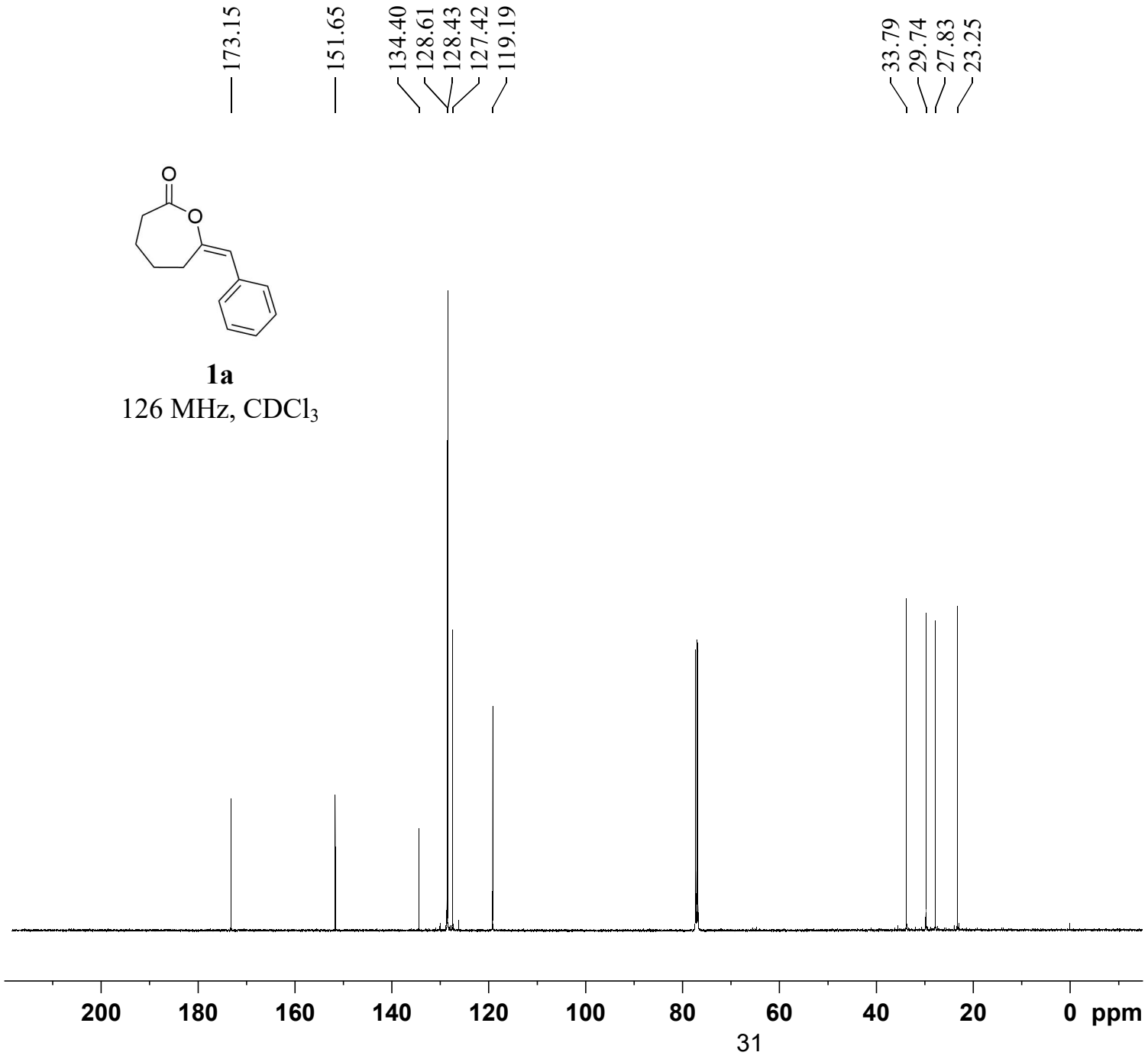


```
Current Data Parameters
NAME          500M-2020
EXPNO                 8
PROCNO                1

F2 - Acquisition Parameters
Date_          20191207
Time               9.51
INSTRUM           spect
PROBHD   5 mm CPPBBO BB
PULPROG          zgpg30
TD                65536
SOLVENT           CDCl3
NS                  400
DS                    4
SWH           29761.904 Hz
FIDRES         0.454131 Hz
AQ            1.1010048 sec
RG               192.89
DW               16.800 usec
DE               18.00 usec
TE               298.2 K
D1           2.00000000 sec
D11          0.03000000 sec
TD0                   1

======== CHANNEL f1 ========
SFO1        125.7703637 MHz
NUC1                13C
P1                 9.80 usec
PLW1        57.00000000 W

======== CHANNEL f2 ========
SFO2        500.1320005 MHz
NUC2                1H
CPDPRG[2         waltz16
PCPD2             80.00 usec
PLW2        20.00000000 W
PLW12        0.35778001 W
PLW13        0.22898000 W

F2 - Processing parameters
SI                 32768
SF          125.7577885 MHz
WDW                   EM
SSB       0
LB                 1.00 Hz
GB        0
PC                 1.40
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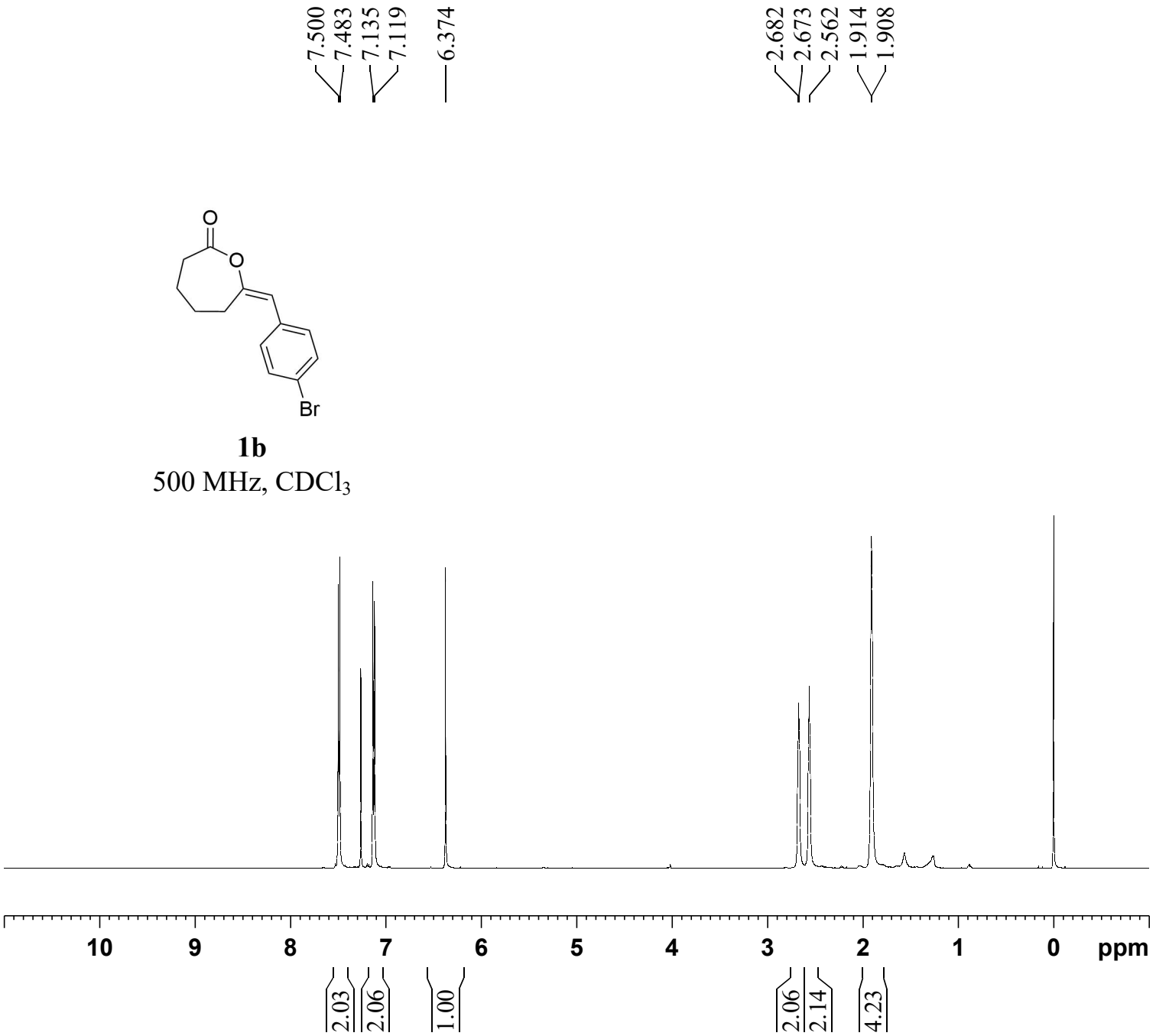


1b

500 MHz, $CDCl_3$

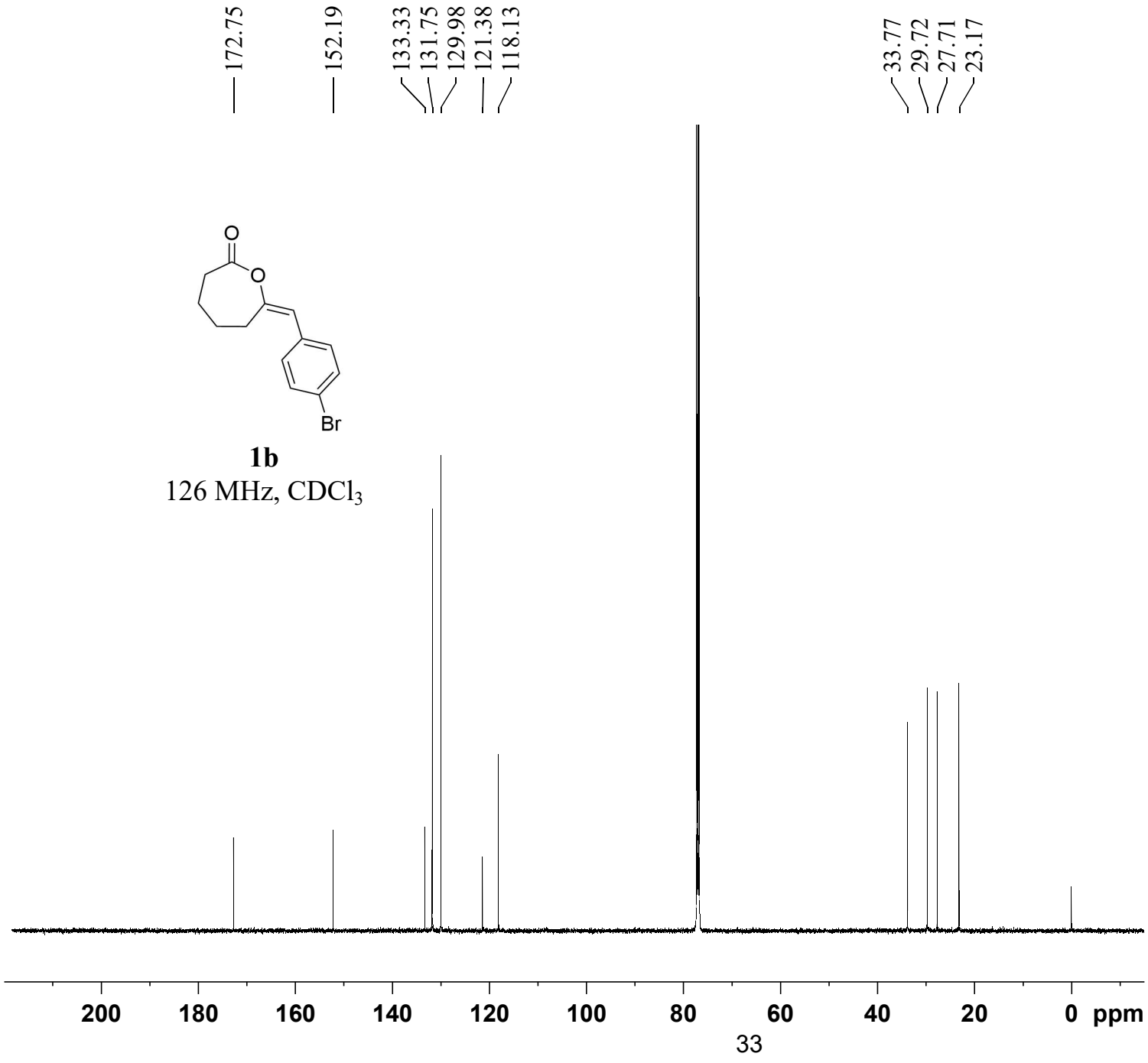
```
Current Data Parameters
NAME        500M-2020xia
EXPNO                 30
PROCNO                 1

F2 - Acquisition Parameters
Date_           20201210
Time              21.56
INSTRUM           spect
PROBHD   5 mm CPPBBO BB
PULPROG             zg30
TD                65536
SOLVENT           CDCl3
NS                   16
DS                    2
SWH           10000.000 Hz
FIDRES         0.152588 Hz
AQ            3.2767999 sec
RG                31.72
DW               50.000 usec
DE                6.50 usec
TE               298.2 K
D1            1.00000000 sec
D11      0 sec
TD0                   1

======== CHANNEL f1 ========
SFO1         500.1330885 MHz
NUC1                 1H
P1                10.59 usec
PLW1         20.00000000 W

======== CHANNEL f2 ========
SFO2         500.1330885 MHz
NUC2                off
CPDPRG[2
PCPD2     0 usec
PLW2      0 W
PLW12     0 W
PLW13     0 W

F2 - Processing parameters
SI                 65536
SF           500.1300098 MHz
WDW                   EM
SSB       0
LB                 0.30 Hz
GB        0
PC                 1.00
```



1b

126 MHz, $CDCl_3$

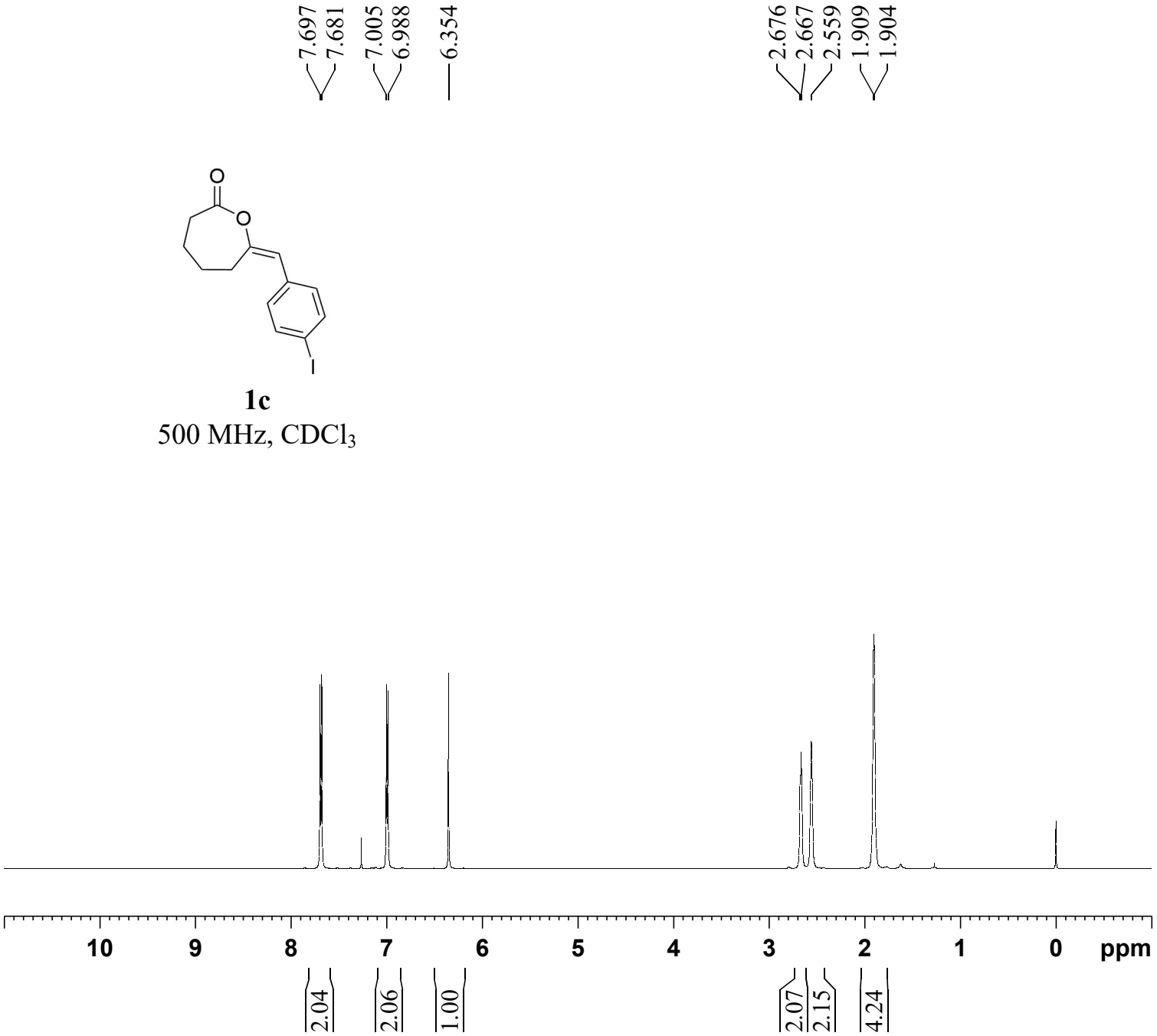
```
Current Data Parameters
NAME        500M-2020xia
EXPNO                 31
PROCNO                 1

F2 - Acquisition Parameters
Date_           20201210
Time              22.51
INSTRUM           spect
PROBHD   5 mm CPPBBO BB
PULPROG          zgpg30
TD                65536
SOLVENT           CDCl3
NS                 1024
DS                    4
SWH           29761.904 Hz
FIDRES         0.454131 Hz
AQ            1.1010048 sec
RG               192.89
DW               16.800 usec
DE               18.00 usec
TE               298.2 K
D1           2.00000000 sec
D11          0.03000000 sec
TD0                   1

======== CHANNEL f1 ========
SFO1        125.7703637 MHz
NUC1                13C
P1                 9.80 usec
PLW1        57.00000000 W

======== CHANNEL f2 ========
SFO2        500.1320005 MHz
NUC2                1H
CPDPRG[2         waltz16
PCPD2             80.00 usec
PLW2        20.00000000 W
PLW12        0.35778001 W
PLW13        0.22898000 W

F2 - Processing parameters
SI                 32768
SF          125.7577897 MHz
WDW                   EM
SSB       0
LB                 1.00 Hz
GB        0
PC                 1.40
```



1c

500 MHz, $CDCl_3$

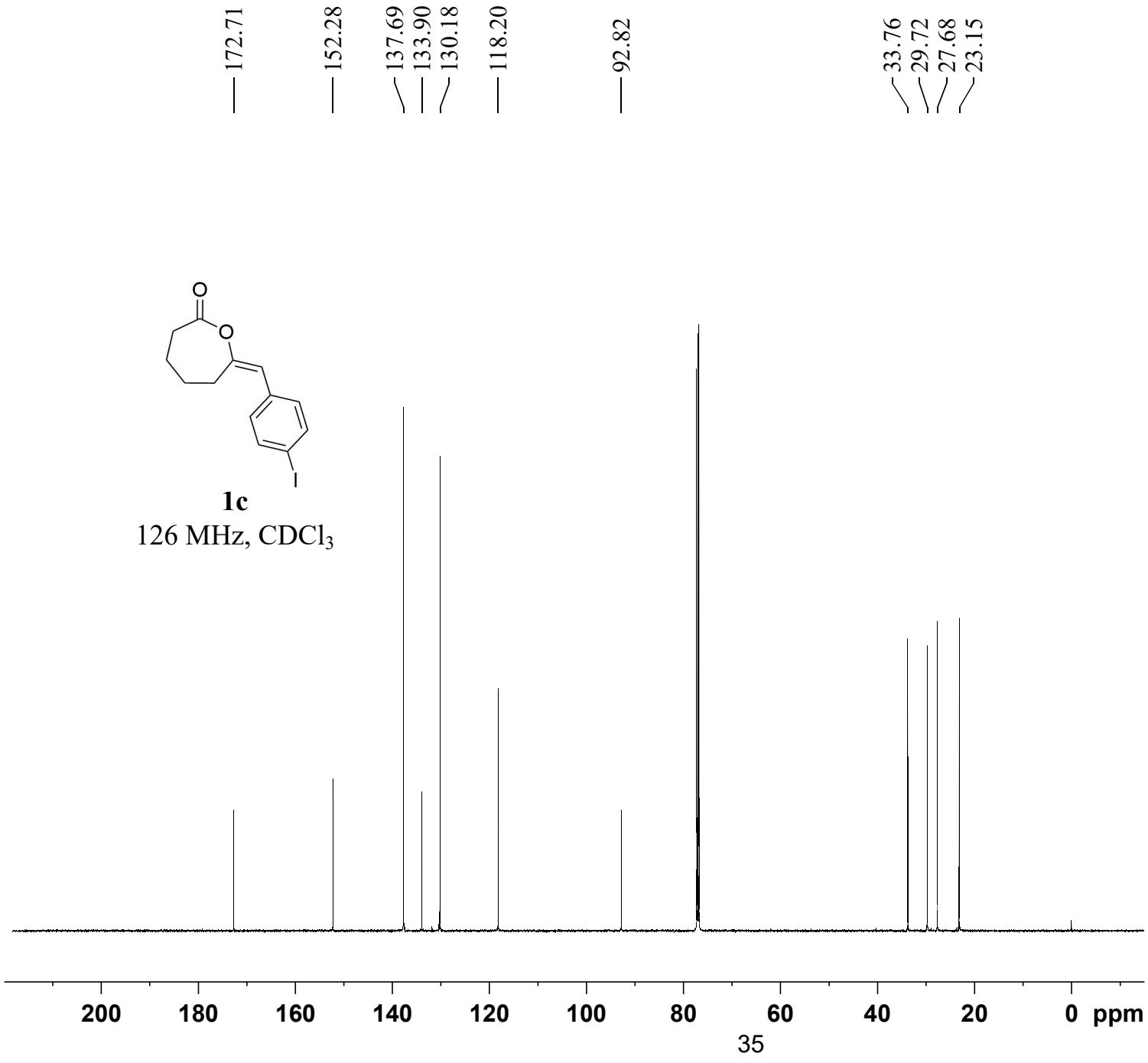
```
Current Data Parameters
NAME        500M-2020xia
EXPNO                 77
PROCNO                 1

F2 - Acquisition Parameters
Date_           20210423
Time              20.11
INSTRUM           spect
PROBHD   5 mm CPPBBO BB
PULPROG             zg30
TD                65536
SOLVENT           CDCl3
NS                   16
DS                    2
SWH           10000.000 Hz
FIDRES         0.152588 Hz
AQ            3.2767999 sec
RG                55.37
DW               50.000 usec
DE                6.50 usec
TE               298.2 K
D1            1.00000000 sec
D11      0 sec
TD0                   1

======== CHANNEL f1 ========
SFO1        500.1330885 MHz
NUC1                   1H
P1                 10.59 usec
PLW1        20.00000000 W

======== CHANNEL f2 ========
SFO2        500.1330885 MHz
NUC2                  off
CPDPRG[2
PCPD2     0 usec
PLW2      0 W
PLW12     0 W
PLW13     0 W

F2 - Processing parameters
SI                 65536
SF          500.1300089 MHz
WDW                   EM
SSB       0
LB                 0.30 Hz
GB        0
PC                 1.00
```



1c

126 MHz, $CDCl_3$

```
Current Data Parameters
NAME        500M-2020xia
EXPNO                 78
PROCNO                 1

F2 - Acquisition Parameters
Date_           20210423
Time              20.43
INSTRUM           spect
PROBHD   5 mm CPPBBO BB
PULPROG          zgpg30
TD                65536
SOLVENT           CDCl3
NS                  600
DS                    4
SWH           29761.904 Hz
FIDRES         0.454131 Hz
AQ             1.1010048 sec
RG                192.89
DW                16.800 usec
DE                18.00 usec
TE                298.2 K
D1            2.00000000 sec
D11           0.03000000 sec
TD0                   1

======== CHANNEL f1 ========
SFO1         125.7703637 MHz
NUC1                 13C
P1                  9.80 usec
PLW1         57.00000000 W

======== CHANNEL f2 ========
SFO2         500.1320005 MHz
NUC2                 1H
CPDPRG[2         waltz16
PCPD2               80.00 usec
PLW2         20.00000000 W
PLW12         0.35778001 W
PLW13         0.22898000 W

F2 - Processing parameters
SI                  32768
SF           125.7577919 MHz
WDW                  EM
SSB       0
LB                  1.00 Hz
GB        0
PC                  1.40
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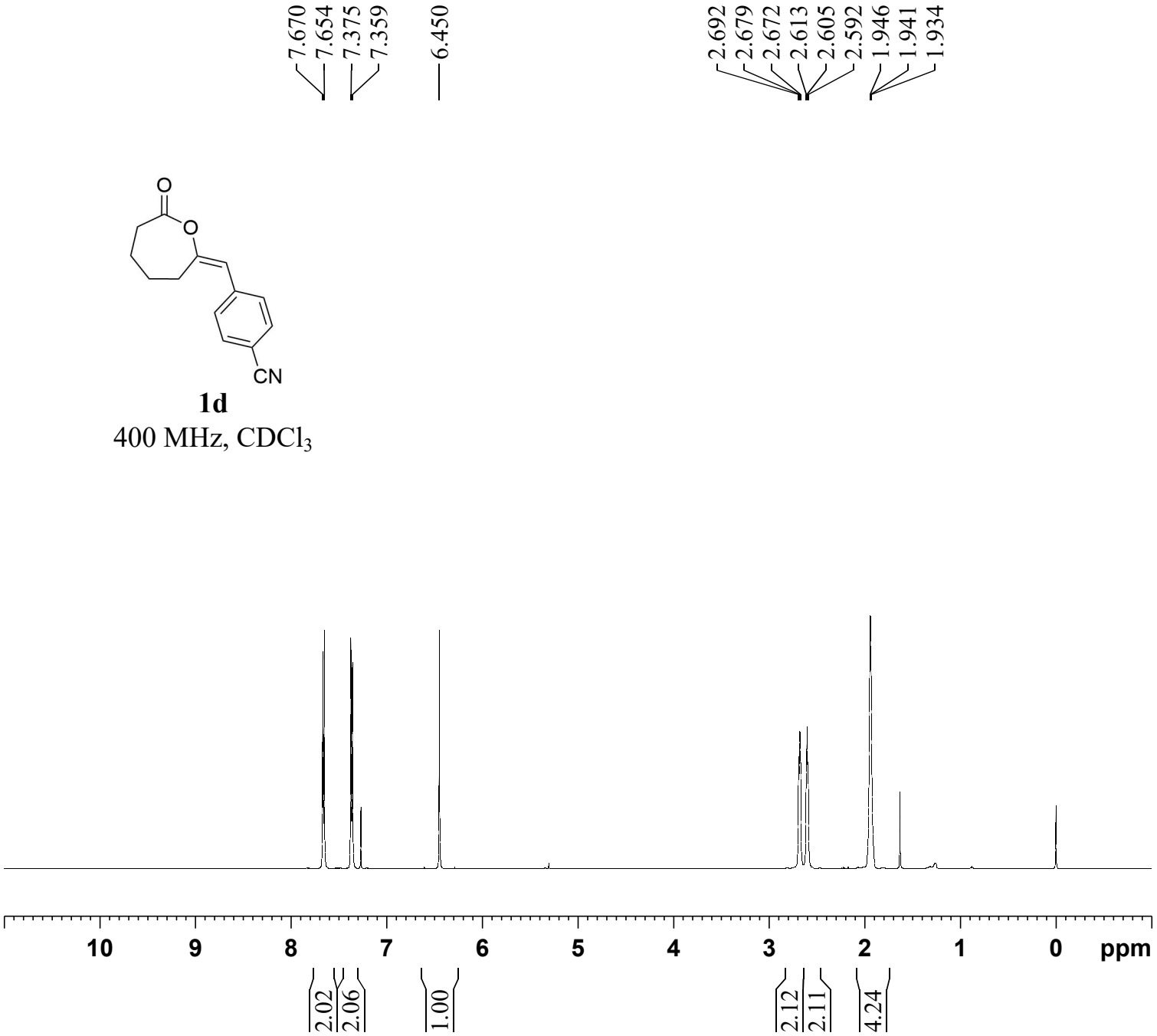
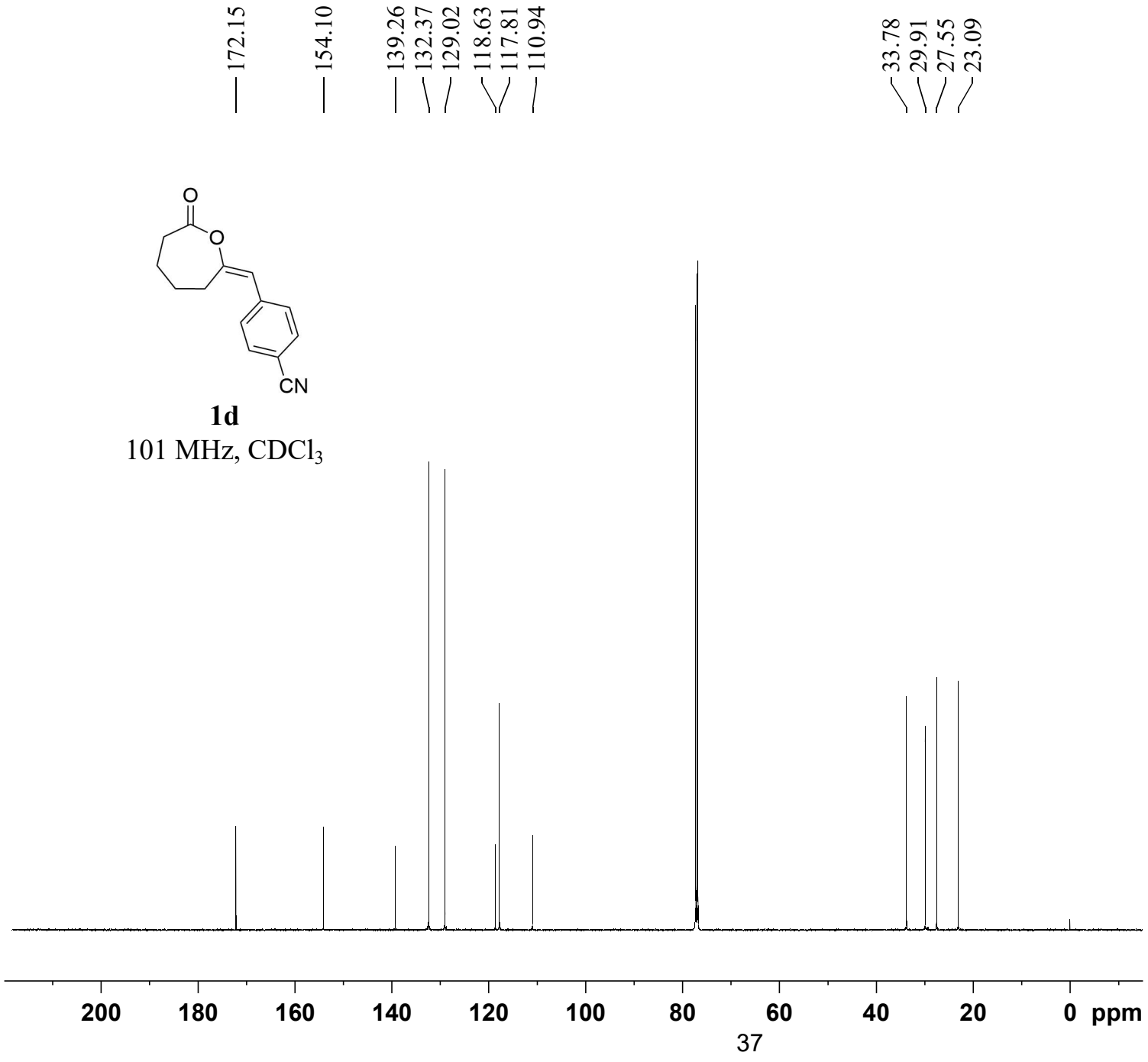


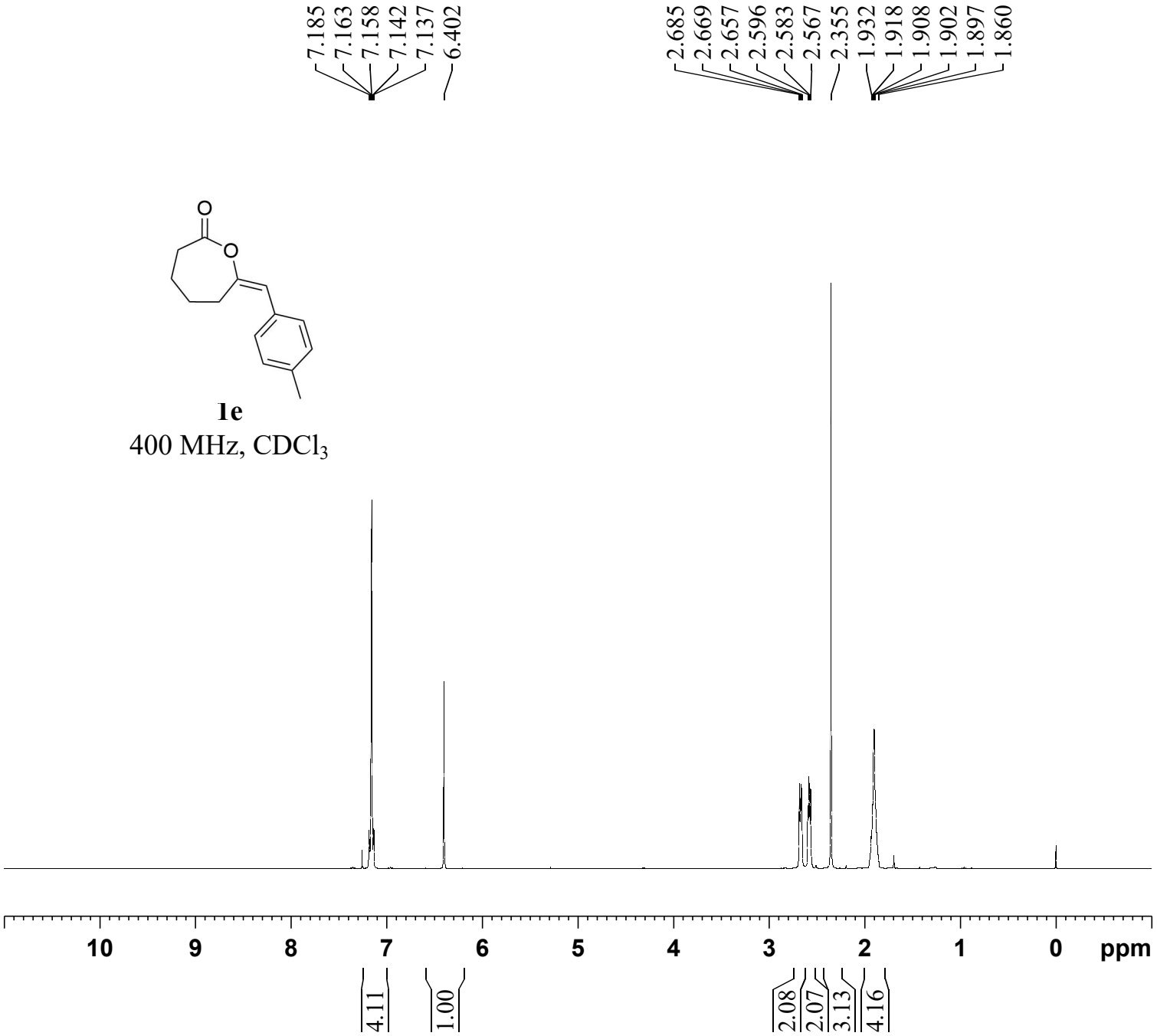
1d

400 MHz, $CDCl_3$

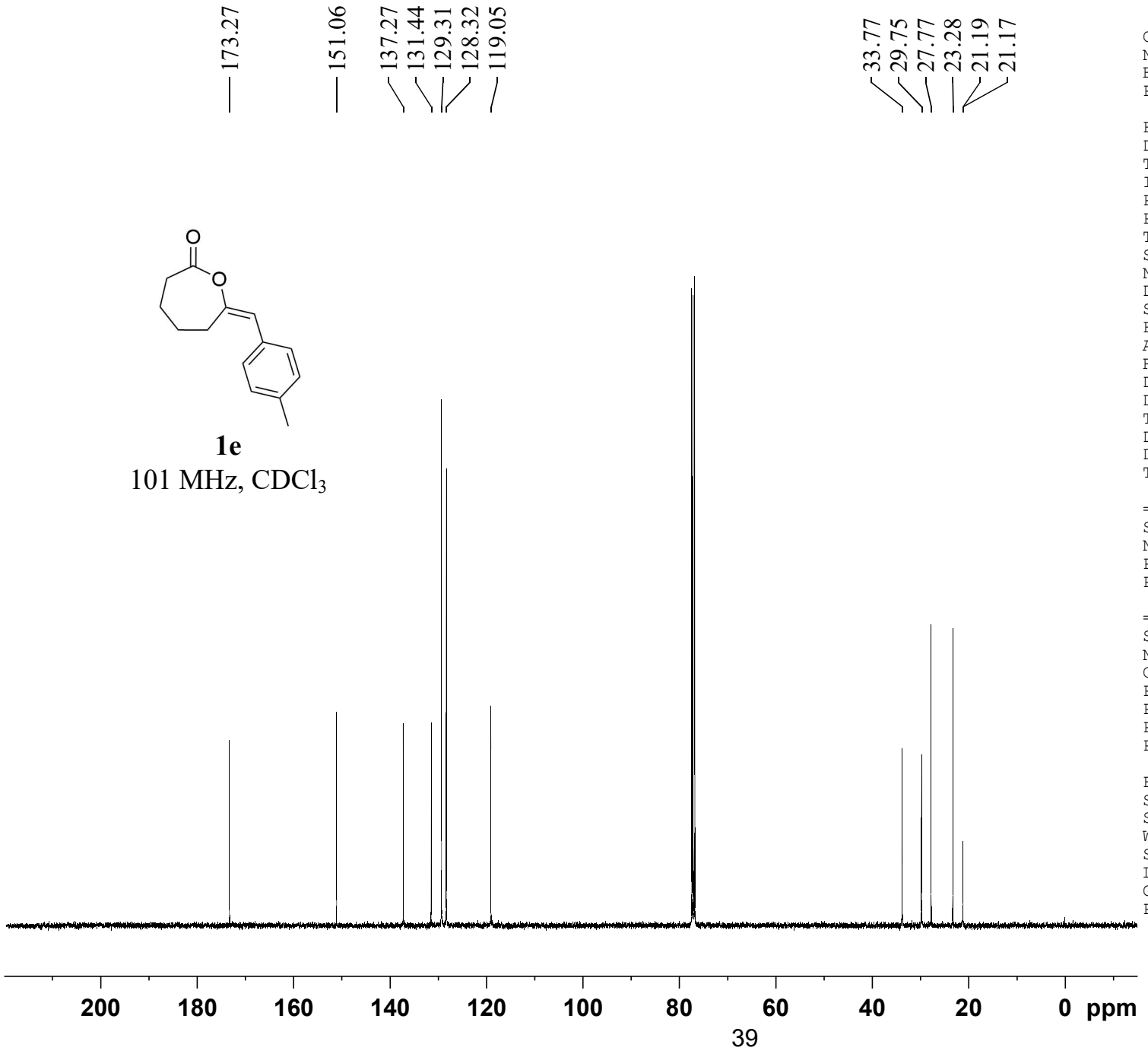
```
Current Data Parameters
NAME        500M-2020xia
EXPNO                  5
PROCNO                 1

F2 - Acquisition Parameters
Date_           20201118
Time                5.30
INSTRUM            spect
PROBHD   5 mm CPPBBO BB
PULPROG             zg30
TD                 65536
SOLVENT            CDCl3
NS                    16
DS                     2
SWH           10000.000 Hz
FIDRES         0.152588 Hz
AQ            3.2767999 sec
RG                 31.72
DW                50.000 usec
DE                 6.50 usec
TE                298.2 K
D1           1.00000000 sec
D11      0 sec
TD0                    1

======== CHANNEL f1 ========
SFO1         500.1330885 MHz
NUC1                   1H
P1                 10.59 usec
PLW1         20.00000000 W

======== CHANNEL f2 ========
SFO2         500.1330885 MHz
NUC2                  off
CPDPRG[2
PCPD2     0 usec
PLW2      0 W
PLW12     0 W
PLW13     0 W

F2 - Processing parameters
SI                 65536
SF           500.1300059 MHz
WDW                   EM
SSB       0
LB                 0.30 Hz
GB        0
PC                 1.00
```



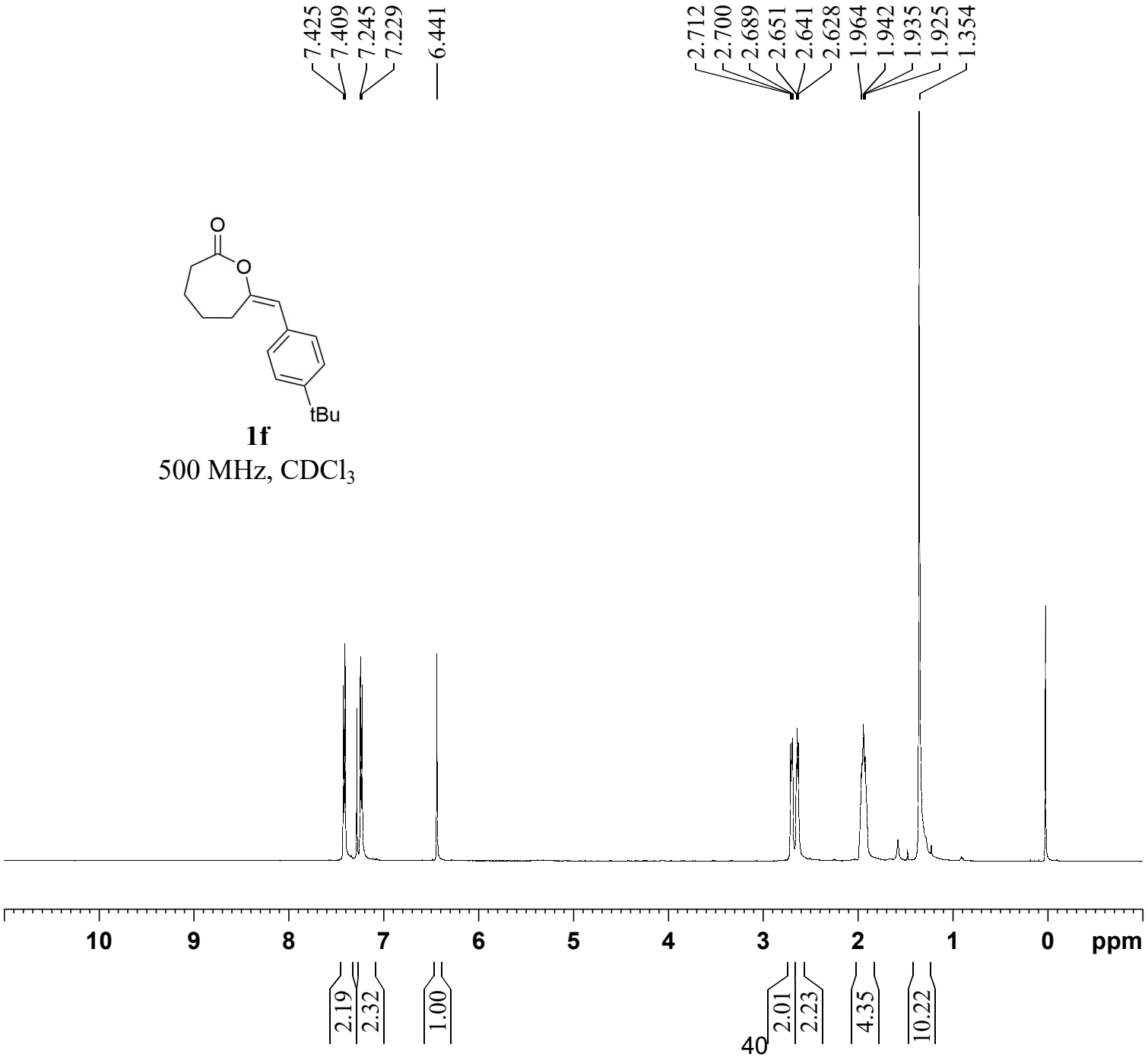

```
Current Data Parameters
NAME        500M-2020xia
EXPNO                  6
PROCNO                 1

F2 - Acquisition Parameters
Date_           20201118
Time               6.25
INSTRUM           spect
PROBHD   5 mm CPPBBO BB
PULPROG          zgpg30
TD                65536
SOLVENT           CDCl3
NS                 1024
DS                    4
SWH           29761.904 Hz
FIDRES         0.454131 Hz
AQ            1.1010048 sec
RG               192.89
DW               16.800 usec
DE               18.00 usec
TE               298.2 K
D1           2.00000000 sec
D11          0.03000000 sec
TD0                   1

======== CHANNEL f1 ========
SFO1        125.7703637 MHz
NUC1                13C
P1                 9.80 usec
PLW1        57.00000000 W

======== CHANNEL f2 ========
SFO2        500.1320005 MHz
NUC2                1H
CPDPRG[2         waltz16
PCPD2             80.00 usec
PLW2        20.00000000 W
PLW12        0.35778001 W
PLW13        0.22898000 W

F2 - Processing parameters
SI                 32768
SF          125.7577895 MHz
WDW                   EM
SSB       0
LB                 1.00 Hz
GB        0
PC                 1.40
```




```
Current Data Parameters
NAME                 400xia
EXPNO                     3
PROCNO                    1

F2 - Acquisition Parameters
Date_              20200929
Time                 10.11
INSTRUM              spect
PROBHD   5 mm PABBO BB/
PULPROG               zg30
TD                   32768
SOLVENT              CDCl3
NS                      16
DS                       0
SWH               8012.820 Hz
FIDRES            0.244532 Hz
AQ               2.0447233 sec
RG                   55.55
DW                  62.400 usec
DE                   6.50 usec
TE                  301.2 K
D1              2.00000000 sec
D11       0 sec
TD0                       1

======== CHANNEL f1 ========
SFO1           400.2424716 MHz
NUC1                    1H
P1                   14.30 usec
PLW1           12.00000000 W

======== CHANNEL f2 ========
SFO2           400.2424716 MHz
NUC2                   off
CPDPRG[2
PCPD2     0 usec
PLW2      0 W
PLW12     0 W
PLW13     0 W

F2 - Processing parameters
SI                    65536
SF           400.2400104 MHz
WDW                     EM
SSB       0
LB                   0.30 Hz
GB        0
PC                   1.00
```



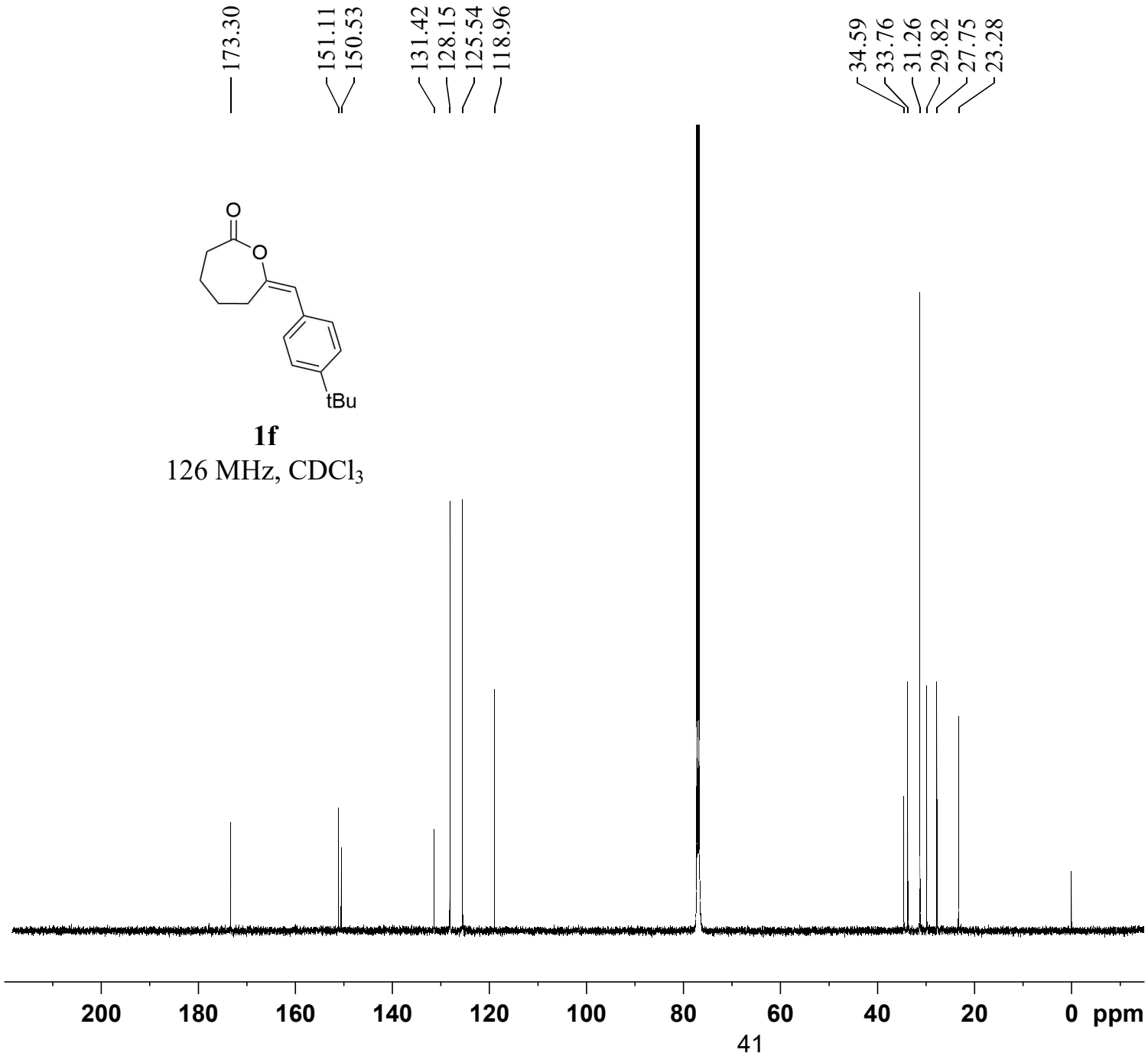

```
Current Data Parameters
NAME                 400xia
EXPNO                     4
PROCNO                    1

F2 - Acquisition Parameters
Date_             20200929
Time                 11.10
INSTRUM              spect
PROBHD   5 mm PABBO BB/
PULPROG             zgpg30
TD                   65536
SOLVENT              CDCl3
NS                    1024
DS                       4
SWH              24038.461 Hz
FIDRES            0.366798 Hz
AQ               1.3631488 sec
RG                  206.33
DW                  20.800 usec
DE                   6.50 usec
TE                  300.8 K
D1              2.00000000 sec
D11             0.03000000 sec
TD0                      1

======== CHANNEL f1 ========
SFO1           100.6504916 MHz
NUC1                   13C
P1                  10.00 usec
PLW1           54.00000000 W

======== CHANNEL f2 ========
SFO2           400.2416010 MHz
NUC2                   1H
CPDPRG[2           waltz16
PCPD2               90.00 usec
PLW2           12.00000000 W
PLW12           0.30294999 W
PLW13           0.24539000 W

F2 - Processing parameters
SI                    32768
SF             100.6404280 MHz
WDW                     EM
SSB       0
LB                   1.00 Hz
GB        0
PC                   1.40
```



1f

500 MHz, $CDCl_3$

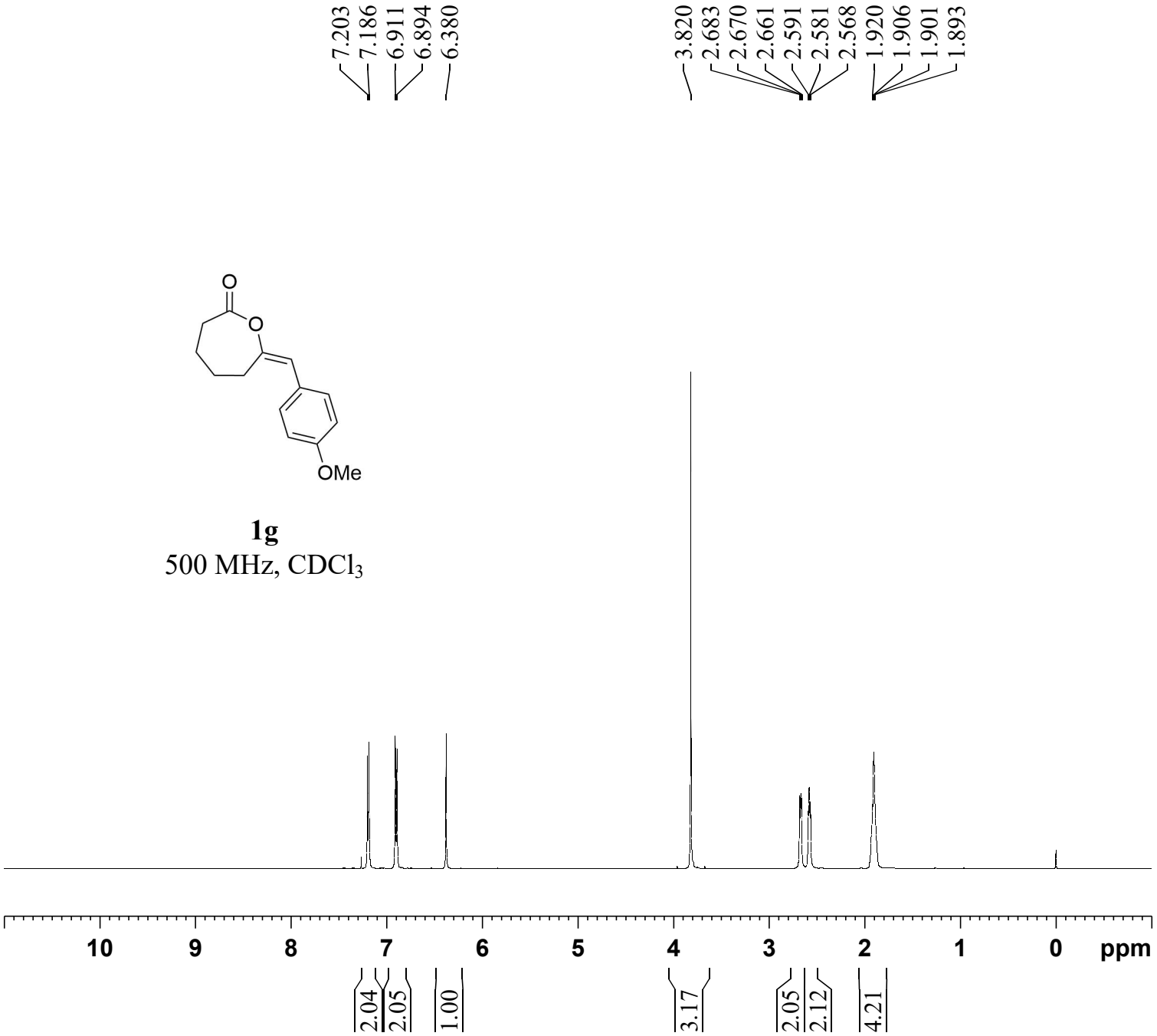
```
Current Data Parameters
NAME        500M-2020xia
EXPNO                 62
PROCNO                 1

F2 - Acquisition Parameters
Date_           20210112
Time              11.43
INSTRUM           spect
PROBHD   5 mm CPPBBO BB
PULPROG             zg30
TD                65536
SOLVENT           CDCl3
NS                   16
DS                    2
SWH           10000.000 Hz
FIDRES         0.152588 Hz
AQ            3.2767999 sec
RG                62.06
DW               50.000 usec
DE                6.50 usec
TE               298.2 K
D1           1.00000000 sec
D11      0 sec
TD0                    1

======== CHANNEL f1 ========
SFO1        500.1330885 MHz
NUC1                   1H
P1                 10.59 usec
PLW1        20.00000000 W

======== CHANNEL f2 ========
SFO2        500.1330885 MHz
NUC2                  off
CPDPRG[2
PCPD2     0 usec
PLW2      0 W
PLW12     0 W
PLW13     0 W

F2 - Processing parameters
SI                 65536
SF          500.1300000 MHz
WDW                   EM
SSB       0
LB                 0.30 Hz
GB        0
PC                 1.00
```



1f

126 MHz, $CDCl_3$

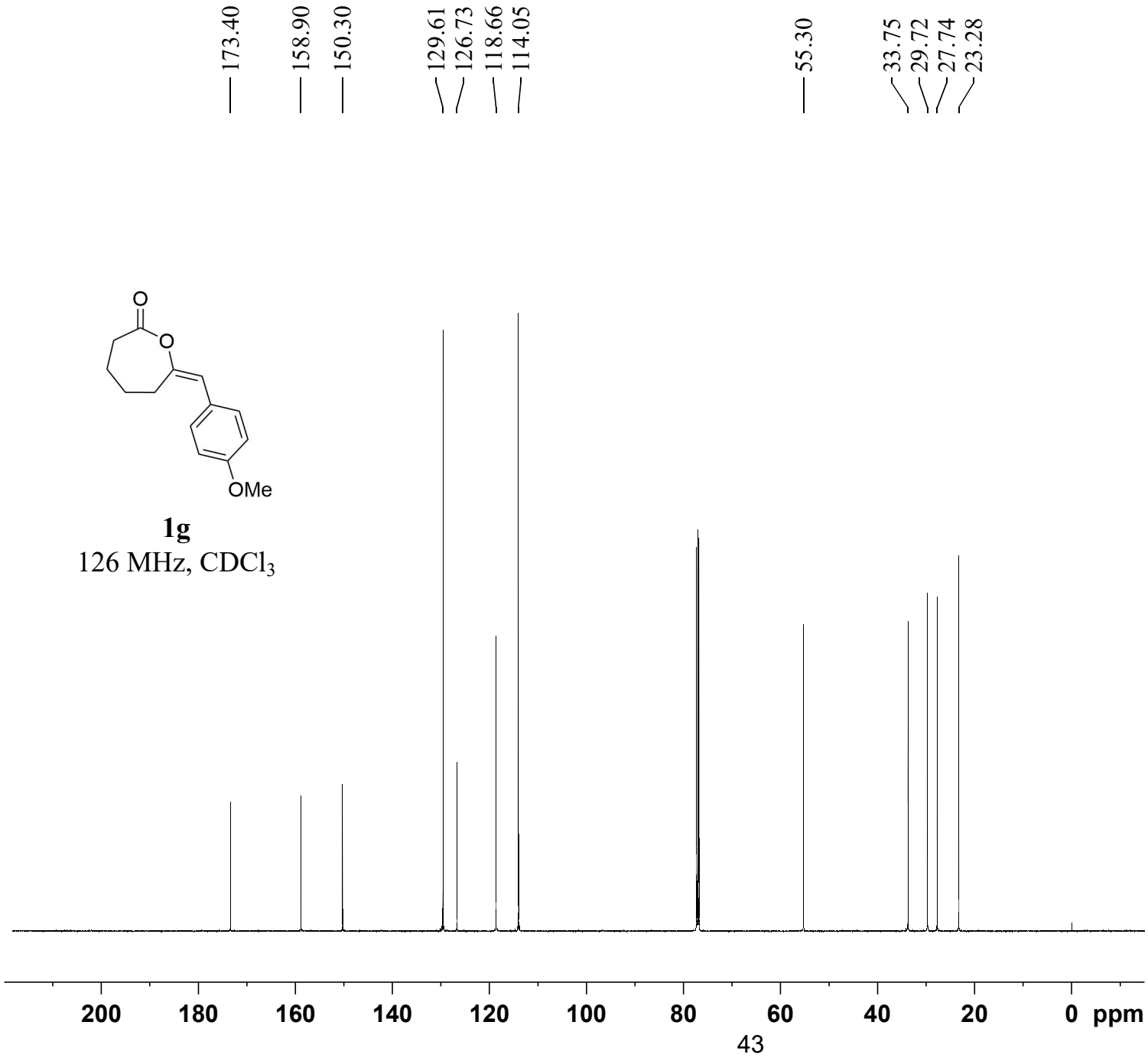
```
Current Data Parameters
NAME        500M-2020xia
EXPNO                 39
PROCNO                 1

F2 - Acquisition Parameters
Date_           20210111
Time                0.21
INSTRUM            spect
PROBHD   5 mm CPPBBO BB
PULPROG          zgpg30
TD                65536
SOLVENT           CDCl3
NS                  800
DS                    4
SWH           29761.904 Hz
FIDRES         0.454131 Hz
AQ            1.1010048 sec
RG               192.89
DW               16.800 usec
DE               18.00 usec
TE               298.2 K
D1           2.00000000 sec
D11          0.03000000 sec
TD0                   1

======== CHANNEL f1 ========
SFO1        125.7703637 MHz
NUC1                13C
P1                 9.80 usec
PLW1        57.00000000 W

======== CHANNEL f2 ========
SFO2        500.1320005 MHz
NUC2                1H
CPDPRG[2         waltz16
PCPD2             80.00 usec
PLW2        20.00000000 W
PLW12        0.35778001 W
PLW13        0.22898000 W

F2 - Processing parameters
SI                 32768
SF          125.7577898 MHz
WDW                   EM
SSB       0
LB                 1.00 Hz
GB        0
PC                 1.40
```



1g

500 MHz, $CDCl_3$

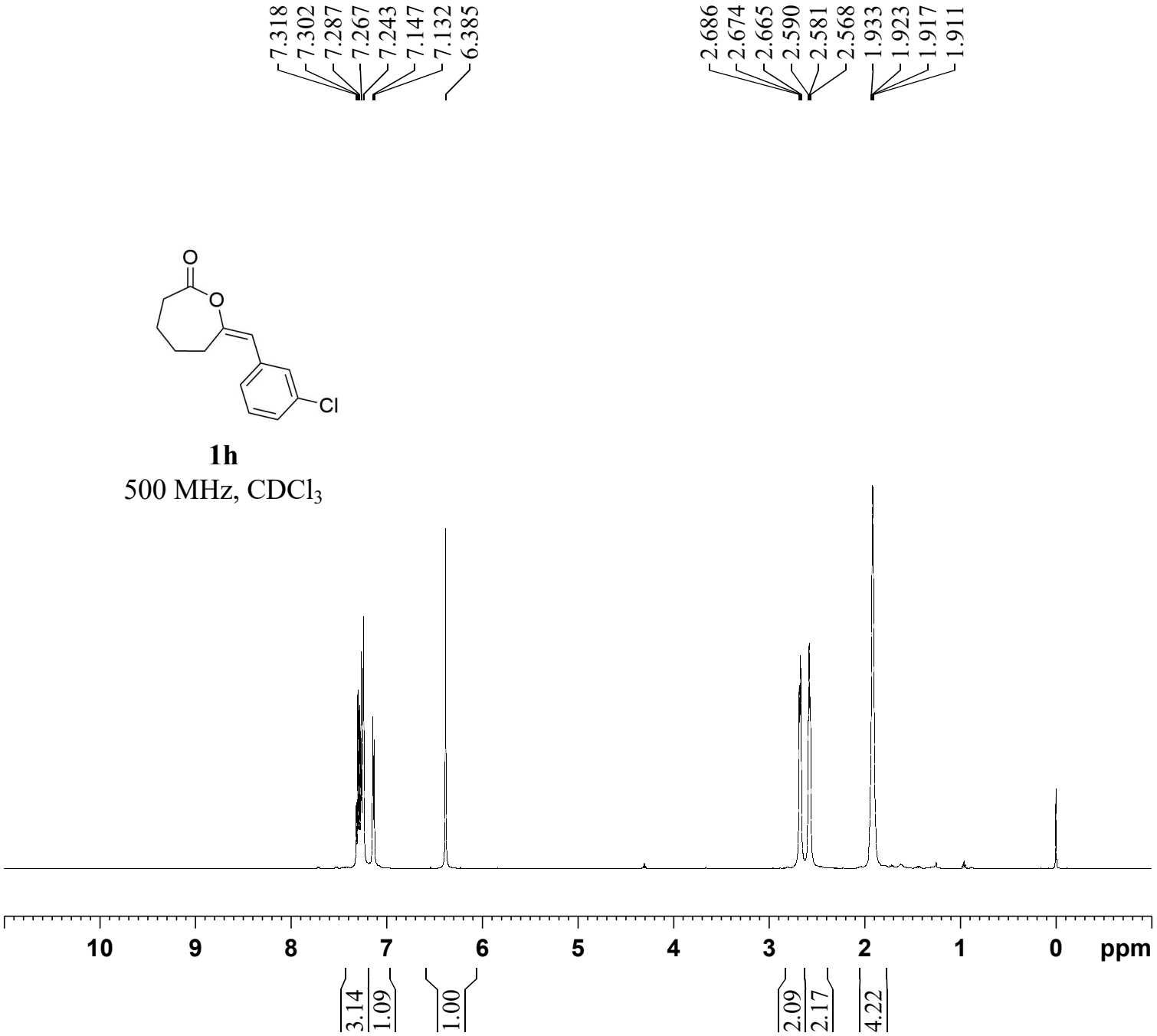
```
Current Data Parameters
NAME        500M-2020xia
EXPNO                 75
PROCNO                 1

F2 - Acquisition Parameters
Date_           20210423
Time              19.35
INSTRUM           spect
PROBHD   5 mm CPPBBO BB
PULPROG             zg30
TD                65536
SOLVENT           CDCl3
NS                   16
DS                    2
SWH           10000.000 Hz
FIDRES         0.152588 Hz
AQ            3.2767999 sec
RG                31.72
DW               50.000 usec
DE                6.50 usec
TE               298.2 K
D1           1.00000000 sec
D11      0 sec
TD0                   1

======== CHANNEL f1 ========
SFO1        500.1330885 MHz
NUC1                  1H
P1                10.59 usec
PLW1        20.00000000 W

======== CHANNEL f2 ========
SFO2        500.1330885 MHz
NUC2                 off
CPDPRG[2
PCPD2     0 usec
PLW2      0 W
PLW12     0 W
PLW13     0 W

F2 - Processing parameters
SI                  65536
SF          500.1300084 MHz
WDW                    EM
SSB       0
LB                  0.30 Hz
GB        0
PC                  1.00
```



1g

126 MHz, $CDCl_3$

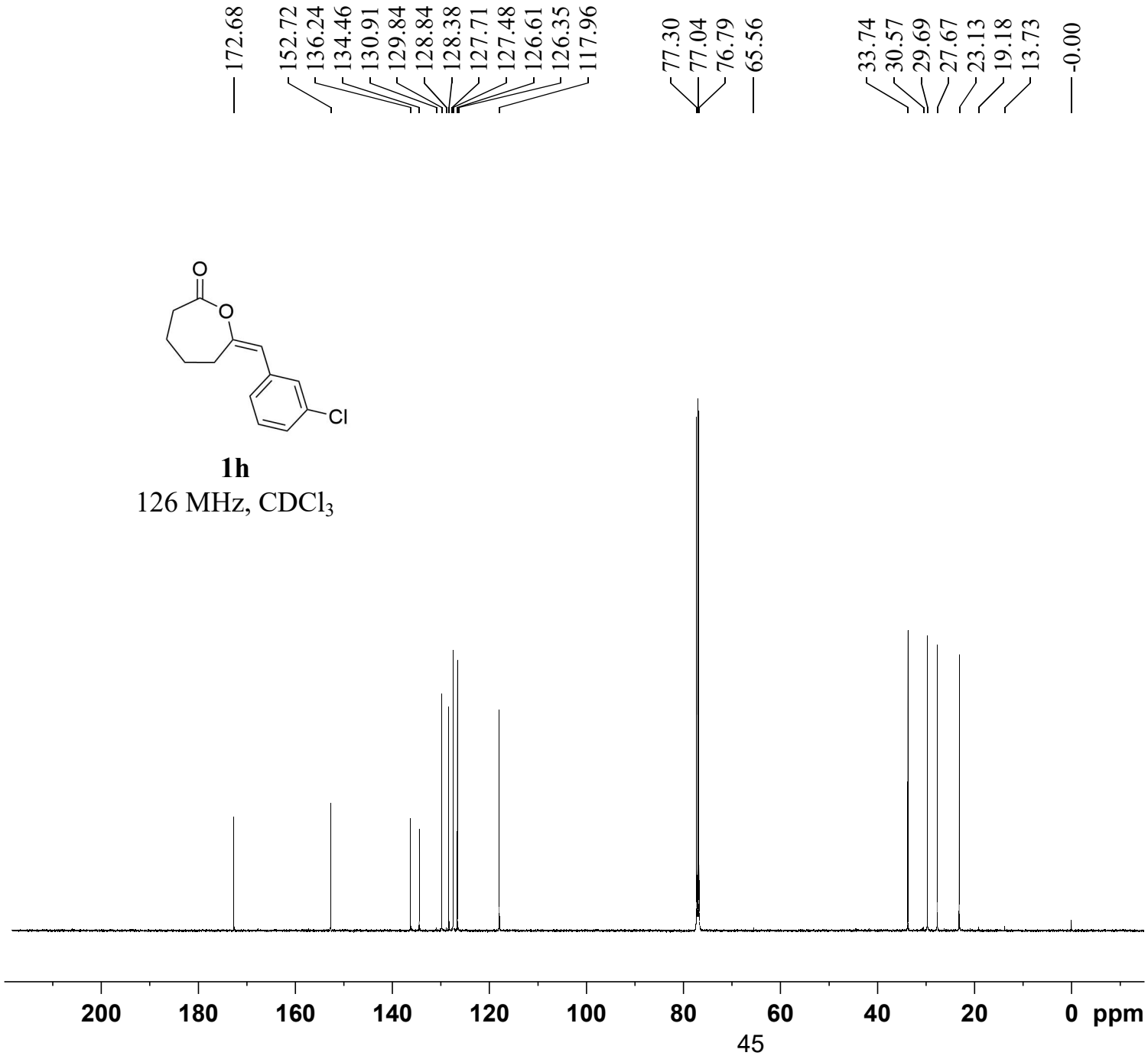
```
Current Data Parameters
NAME        500M-2020xia
EXPNO                 76
PROCNO                 1

F2 - Acquisition Parameters
Date_           20210423
Time              20.07
INSTRUM           spect
PROBHD   5 mm CPPBBO BB
PULPROG          zgpg30
TD                65536
SOLVENT           CDCl3
NS                  600
DS                    4
SWH           29761.904 Hz
FIDRES         0.454131 Hz
AQ             1.1010048 sec
RG                192.89
DW                16.800 usec
DE                18.00 usec
TE                298.2 K
D1            2.00000000 sec
D11           0.03000000 sec
TD0                   1

======== CHANNEL f1 ========
SFO1         125.7703637 MHz
NUC1                 13C
P1                  9.80 usec
PLW1         57.00000000 W

======== CHANNEL f2 ========
SFO2         500.1320005 MHz
NUC2                 1H
CPDPRG[2          waltz16
PCPD2               80.00 usec
PLW2         20.00000000 W
PLW12         0.35778001 W
PLW13         0.22898000 W

F2 - Processing parameters
SI                  32768
SF           125.7577906 MHz
WDW                  EM
SSB       0
LB                  1.00 Hz
GB        0
PC                  1.40
```



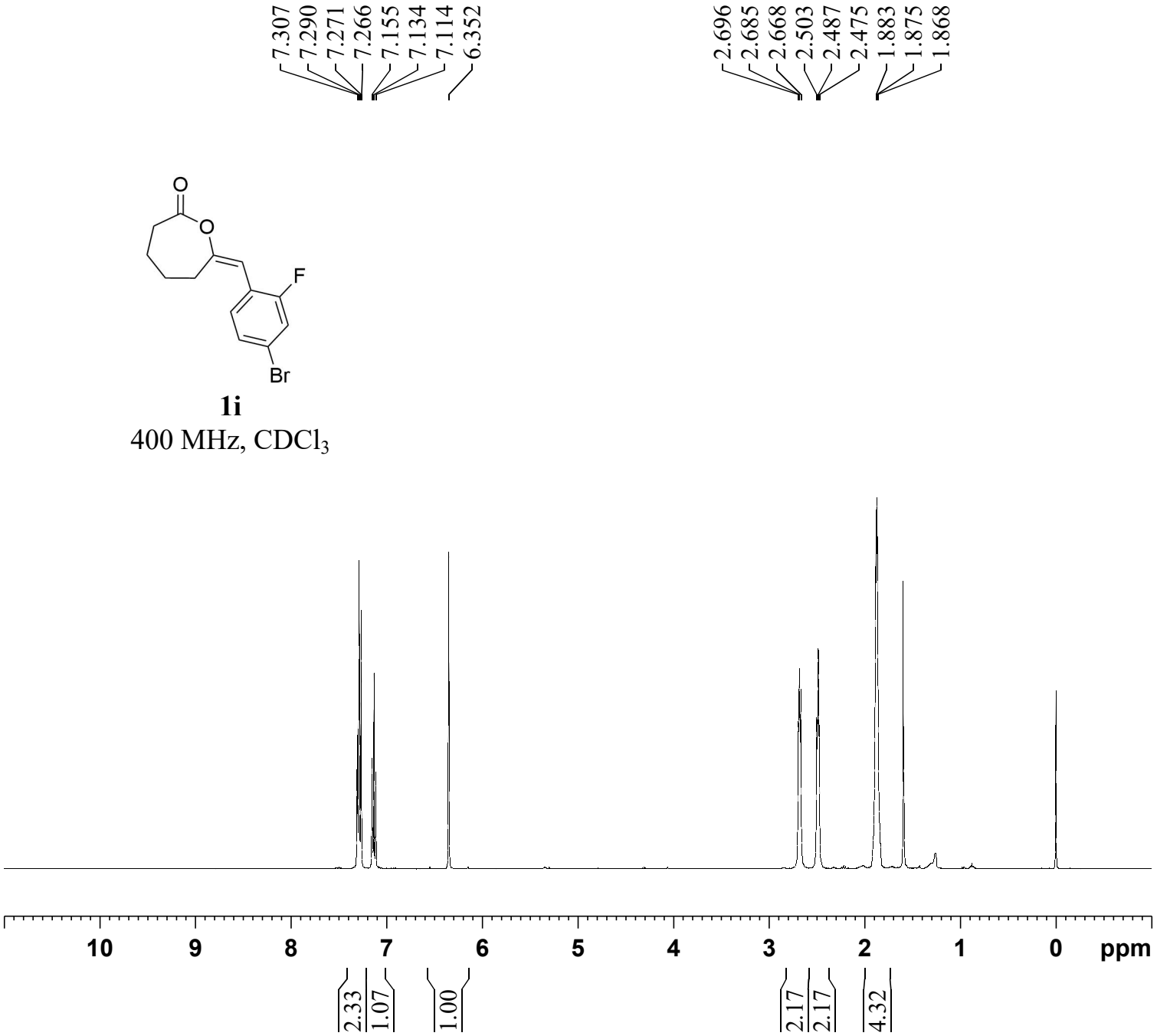
1h

500 MHz, $CDCl_3$

```
Current Data Parameters
NAME        500M-2020xia
EXPNO                 71
PROCNO                 1

F2 - Acquisition Parameters
Date_           20210423
Time              18.23
INSTRUM           spect
PROBHD   5 mm CPPBBO BB
PULPROG             zg30
TD                65536
SOLVENT           CDCl3
NS                   16
DS                    2
SWH           10000.000 Hz
FIDRES         0.152588 Hz
AQ            3.2767999 sec
RG                31.72
DW               50.000 usec
DE                6.50 usec
TE               298.2 K
D1           1.00000000 sec
D11      0 sec
TD0                   1

======== CHANNEL f1 ========
SFO1        500.1330885 MHz
NUC1                  1H
P1                10.59 usec
PLW1        20.00000000 W

======== CHANNEL f2 ========
SFO2        500.1330885 MHz
NUC2                 off
CPDPRG[2
PCPD2     0 usec
PLW2      0 W
PLW12     0 W
PLW13     0 W

F2 - Processing parameters
SI                  65536
SF          500.1300092 MHz
WDW                    EM
SSB       0
LB                  0.30 Hz
GB        0
PC                  1.00
```



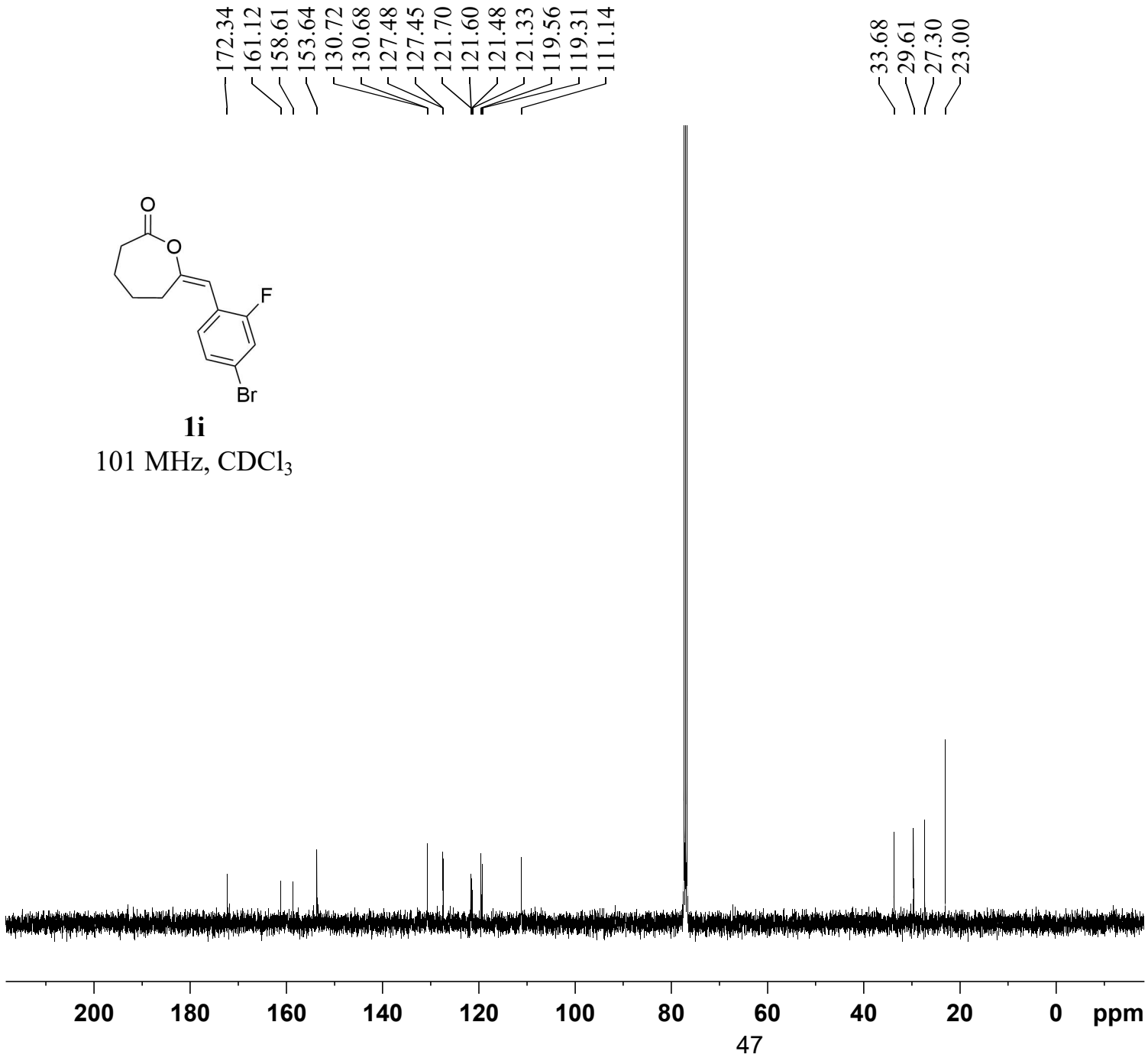

```
Current Data Parameters
NAME        500M-2020xia
EXPNO                 72
PROCNO                 1

F2 - Acquisition Parameters
Date_           20210423
Time              18.55
INSTRUM           spect
PROBHD   5 mm CPPBBO BB
PULPROG          zgpg30
TD                65536
SOLVENT           CDCl3
NS                  600
DS                    4
SWH           29761.904 Hz
FIDRES         0.454131 Hz
AQ             1.1010048 sec
RG                192.89
DW                16.800 usec
DE                18.00 usec
TE                298.2 K
D1            2.00000000 sec
D11           0.03000000 sec
TD0                   1

======== CHANNEL f1 ========
SFO1         125.7703637 MHz
NUC1                 13C
P1                  9.80 usec
PLW1         57.00000000 W

======== CHANNEL f2 ========
SFO2         500.1320005 MHz
NUC2                 1H
CPDPRG[2         waltz16
PCPD2               80.00 usec
PLW2         20.00000000 W
PLW12         0.35778001 W
PLW13         0.22898000 W

F2 - Processing parameters
SI                  32768
SF           125.7577899 MHz
WDW                  EM
SSB       0
LB                  1.00 Hz
GB        0
PC                  1.40
```



1i

400 MHz, $CDCl_3$

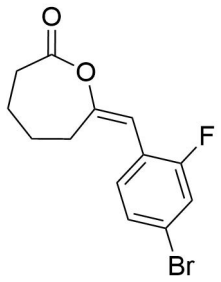
```
Current Data Parameters
NAME                 400xia
EXPNO                     1
PROCNO                    1

F2 - Acquisition Parameters
Date_             20200929
Time                  8.59
INSTRUM              spect
PROBHD   5 mm PABBO BB/
PULPROG               zg30
TD                   32768
SOLVENT              CDCl3
NS                      16
DS                       0
SWH               8012.820 Hz
FIDRES            0.244532 Hz
AQ               2.0447233 sec
RG                  206.33
DW                  62.400 usec
DE                   6.50 usec
TE                  299.7 K
D1              2.00000000 sec
D11       0 sec
TD0                       1

======== CHANNEL f1 ========
SFO1          400.2424716 MHz
NUC1                     1H
P1                   14.30 usec
PLW1          12.00000000 W

======== CHANNEL f2 ========
SFO2          400.2424716 MHz
NUC2                   off
CPDPRG[2
PCPD2     0 usec
PLW2      0 W
PLW12     0 W
PLW13     0 W

F2 - Processing parameters
SI                    65536
SF          400.2400075 MHz
WDW                     EM
SSB       0
LB                   0.30 Hz
GB        0
PC                   1.00
```



1i

101 MHz, $CDCl_3$

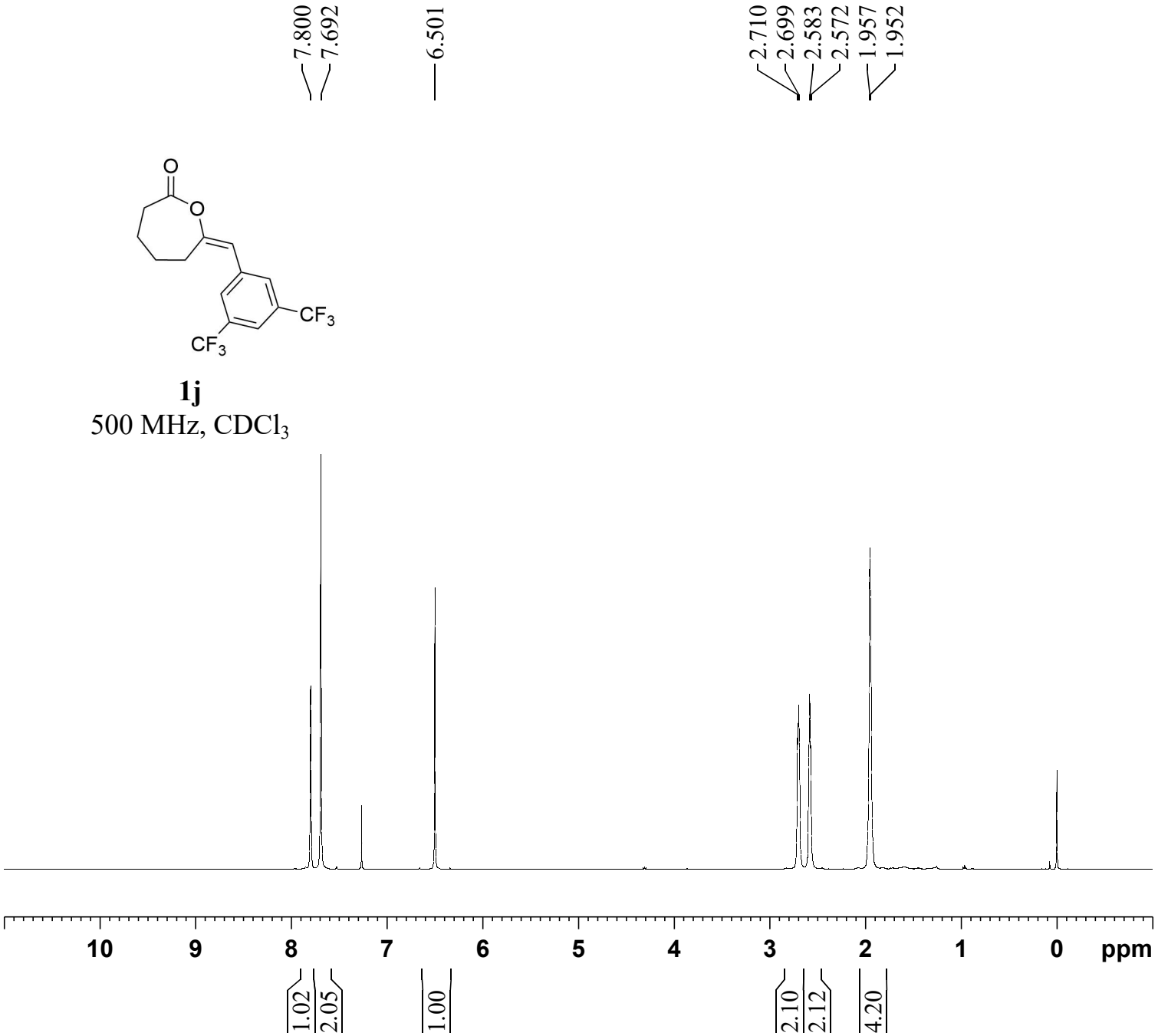
```
Current Data Parameters
NAME                 400xia
EXPNO                     2
PROCNO                    1

F2 - Acquisition Parameters
Date_             20200929
Time                 10.07
INSTRUM              spect
PROBHD   5 mm PABBO BB/
PULPROG             zgpg30
TD                   65536
SOLVENT              CDCl3
NS                     500
DS                       4
SWH              24038.461 Hz
FIDRES            0.366798 Hz
AQ                1.3631488 sec
RG                   206.33
DW                  20.800 usec
DE                   6.50 usec
TE                  301.9 K
D1               2.00000000 sec
D11              0.03000000 sec
TD0                       1

======== CHANNEL f1 ========
SFO1           100.6504916 MHz
NUC1                    13C
P1                   10.00 usec
PLW1           54.00000000 W

======== CHANNEL f2 ========
SFO2           400.2416010 MHz
NUC2                    1H
CPDPRG[2           waltz16
PCPD2                90.00 usec
PLW2           12.00000000 W
PLW12           0.30294999 W
PLW13           0.24539000 W

F2 - Processing parameters
SI                    32768
SF             100.6404280 MHz
WDW                      EM
SSB       0
LB                    1.00 Hz
GB        0
PC                    1.40
```



1i

658 MHz, $CDCl_3$

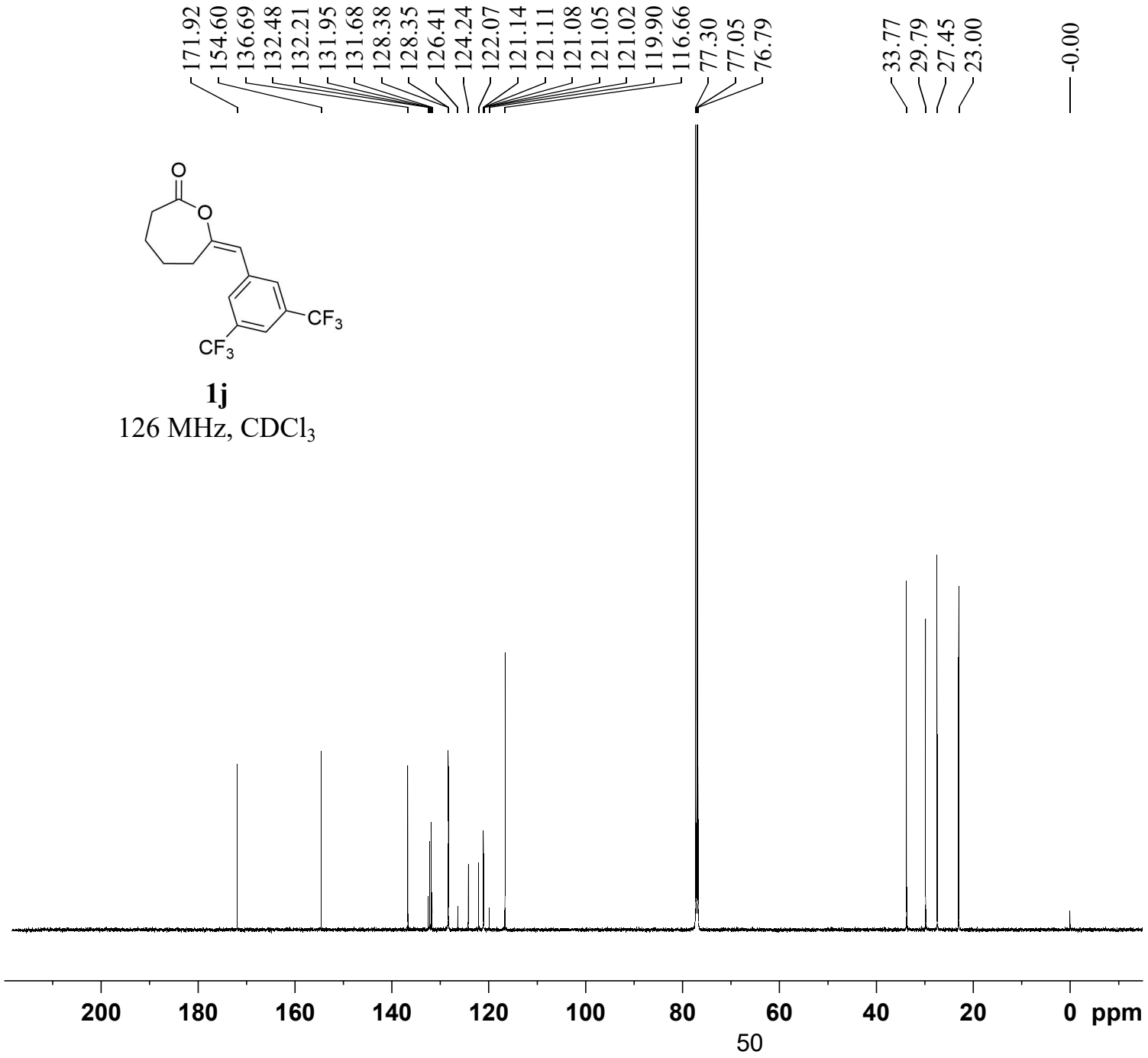

```
Current Data Parameters
NAME        400M-2022-F
EXPNO                 4
PROCNO                1

F2 - Acquisition Parameters
Date_          20221128
Time              14.33 h
INSTRUM           Avance
PROBHD   Z149054_0001 (
PULPROG            zgig
TD               131072
SOLVENT           CDCl3
NS                   64
DS                    4
SWH          156250.000 Hz
FIDRES         2.384186 Hz
AQ            0.4194304 sec
RG                  101
DW                3.200 usec
DE                6.50 usec
TE                298.1 K
D1           1.00000000 sec
D11          0.03000000 sec
TD0                   1
SFO1         658.7615635 MHz
NUC1                19F
P1                25.00 usec
PLW1         21.74500084 W
SFO2         700.1828007 MHz
NUC2                1H
CPDPRG[2        waltz16
PCPD2             65.00 usec
PLW2          8.61509991 W
PLW12         0.39965999 W

F2 - Processing parameters
SI                65536
SF           658.8274462 MHz
WDW                  EM
SSB      0
LB                5.00 Hz
GB       0
PC                1.00
```



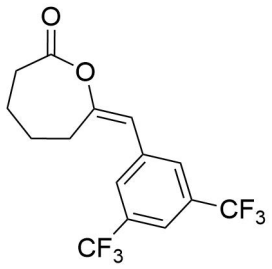
1j

500 MHz, $CDCl_3$

```
Current Data Parameters
NAME        500M-2020xia
EXPNO                 73
PROCNO                 1

F2 - Acquisition Parameters
Date_           20210423
Time              18.59
INSTRUM           spect
PROBHD   5 mm CPPBBO BB
PULPROG             zg30
TD                65536
SOLVENT           CDCl3
NS                   16
DS                    2
SWH           10000.000 Hz
FIDRES         0.152588 Hz
AQ            3.2767999 sec
RG                55.37
DW               50.000 usec
DE                6.50 usec
TE               298.2 K
D1           1.00000000 sec
D11      0 sec
TD0                   1

======== CHANNEL f1 ========
SFO1        500.1330885 MHz
NUC1                  1H
P1                10.59 usec
PLW1        20.00000000 W

======== CHANNEL f2 ========
SFO2        500.1330885 MHz
NUC2                 off
CPDPRG[2
PCPD2     0 usec
PLW2      0 W
PLW12     0 W
PLW13     0 W

F2 - Processing parameters
SI                  65536
SF          500.1300088 MHz
WDW                    EM
SSB       0
LB                  0.30 Hz
GB        0
PC                  1.00
```



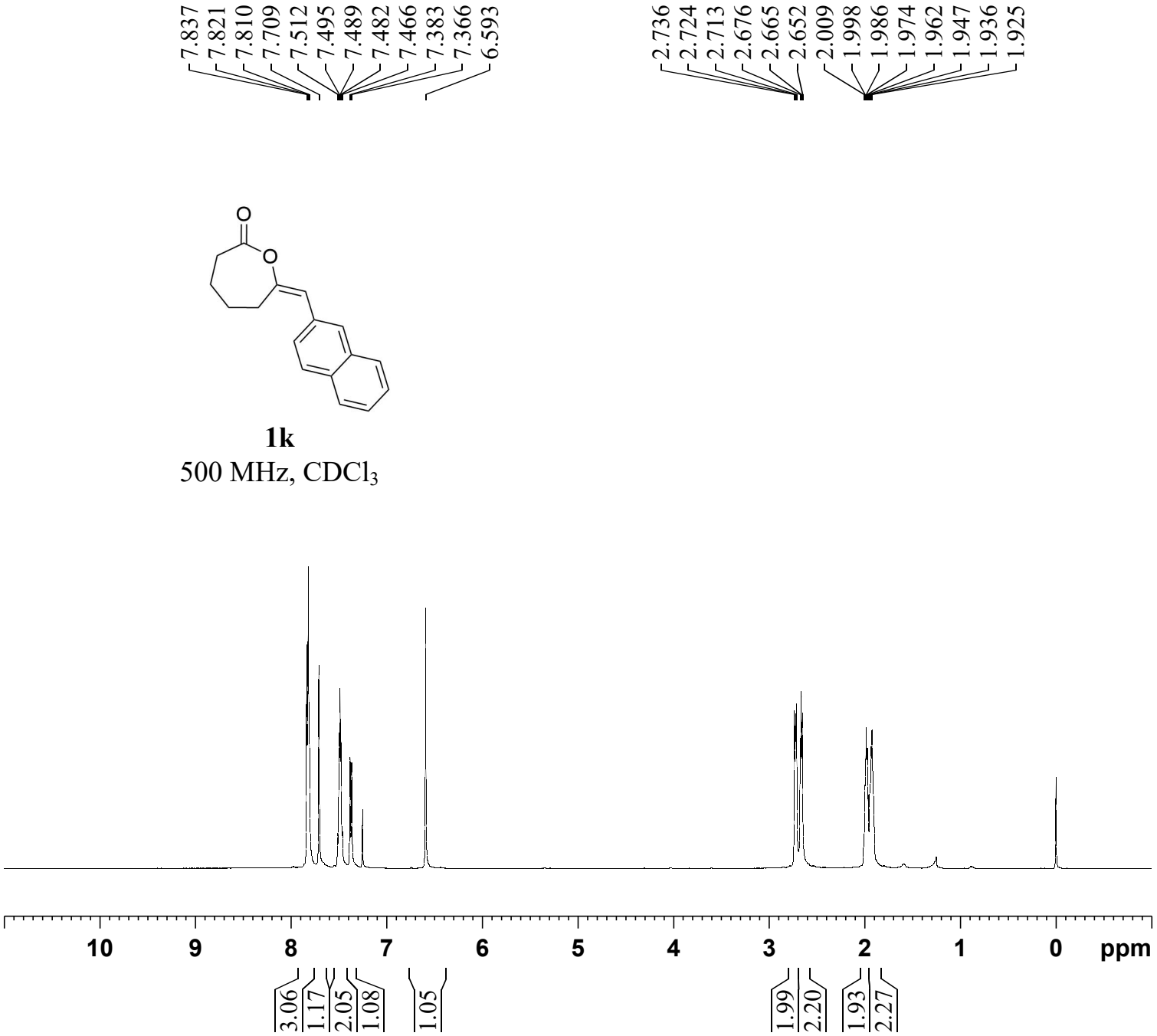

```
Current Data Parameters
NAME        500M-2020xia
EXPNO                 74
PROCNO                 1

F2 - Acquisition Parameters
Date_           20210423
Time              19.18
INSTRUM           spect
PROBHD   5 mm CPPBBO BB
PULPROG          zgpg30
TD                65536
SOLVENT           CDCl3
NS                  600
DS                    4
SWH           29761.904 Hz
FIDRES         0.454131 Hz
AQ             1.1010048 sec
RG                192.89
DW                16.800 usec
DE                18.00 usec
TE                298.2 K
D1            2.00000000 sec
D11           0.03000000 sec
TD0                   1

======== CHANNEL f1 ========
SFO1         125.7703637 MHz
NUC1                13C
P1                 9.80 usec
PLW1         57.00000000 W

======== CHANNEL f2 ========
SFO2         500.1320005 MHz
NUC2                1H
CPDPRG[2         waltz16
PCPD2             80.00 usec
PLW2         20.00000000 W
PLW12         0.35778001 W
PLW13         0.22898000 W

F2 - Processing parameters
SI                 32768
SF           125.7577858 MHz
WDW                  EM
SSB       0
LB                1.00 Hz
GB        0
PC                1.40
```



1i

658 MHz, $CDCl_3$

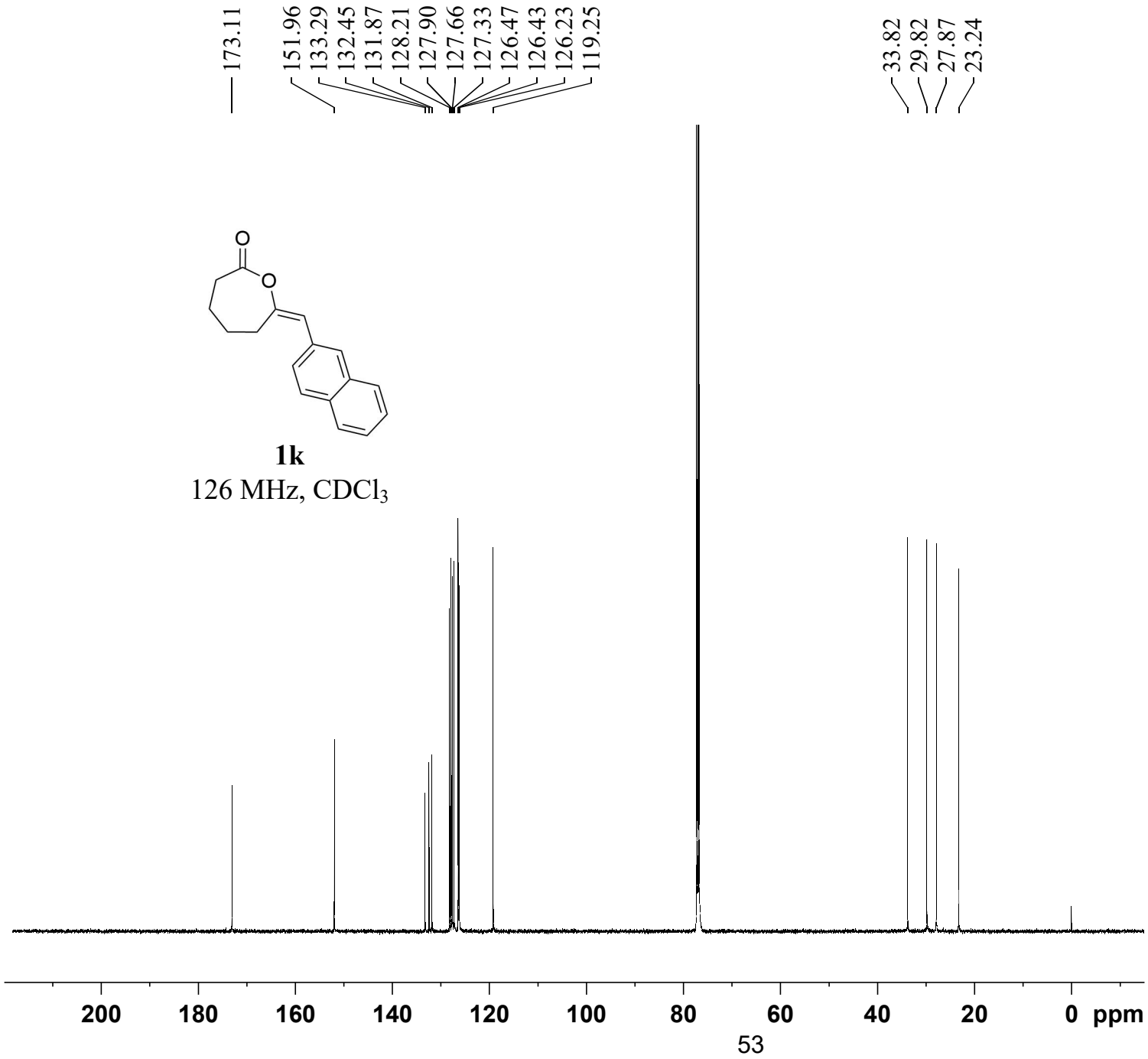

```
Current Data Parameters
NAME         400M-2022-F
EXPNO                  5
PROCNO                 1

F2 - Acquisition Parameters
Date_           20221128
Time               14.39 h
INSTRUM            Avance
PROBHD   Z149054_0001 (
PULPROG             zgig
TD                131072
SOLVENT            CDCl3
NS                    64
DS                     4
SWH           156250.000 Hz
FIDRES          2.384186 Hz
AQ             0.4194304 sec
RG                   101
DW                 3.200 usec
DE                 6.50 usec
TE                 298.0 K
D1             1.00000000 sec
D11            0.03000000 sec
TD0                    1
SFO1         658.7615635 MHz
NUC1                 19F
P1                25.00 usec
PLW1         21.74500084 W
SFO2         700.1828007 MHz
NUC2                 1H
CPDPRG[2         waltz16
PCPD2              65.00 usec
PLW2          8.61509991 W
PLW12         0.39965999 W

F2 - Processing parameters
SI                 65536
SF           658.8274462 MHz
WDW                   EM
SSB       0
LB                 0.30 Hz
GB        0
PC                 1.00
```



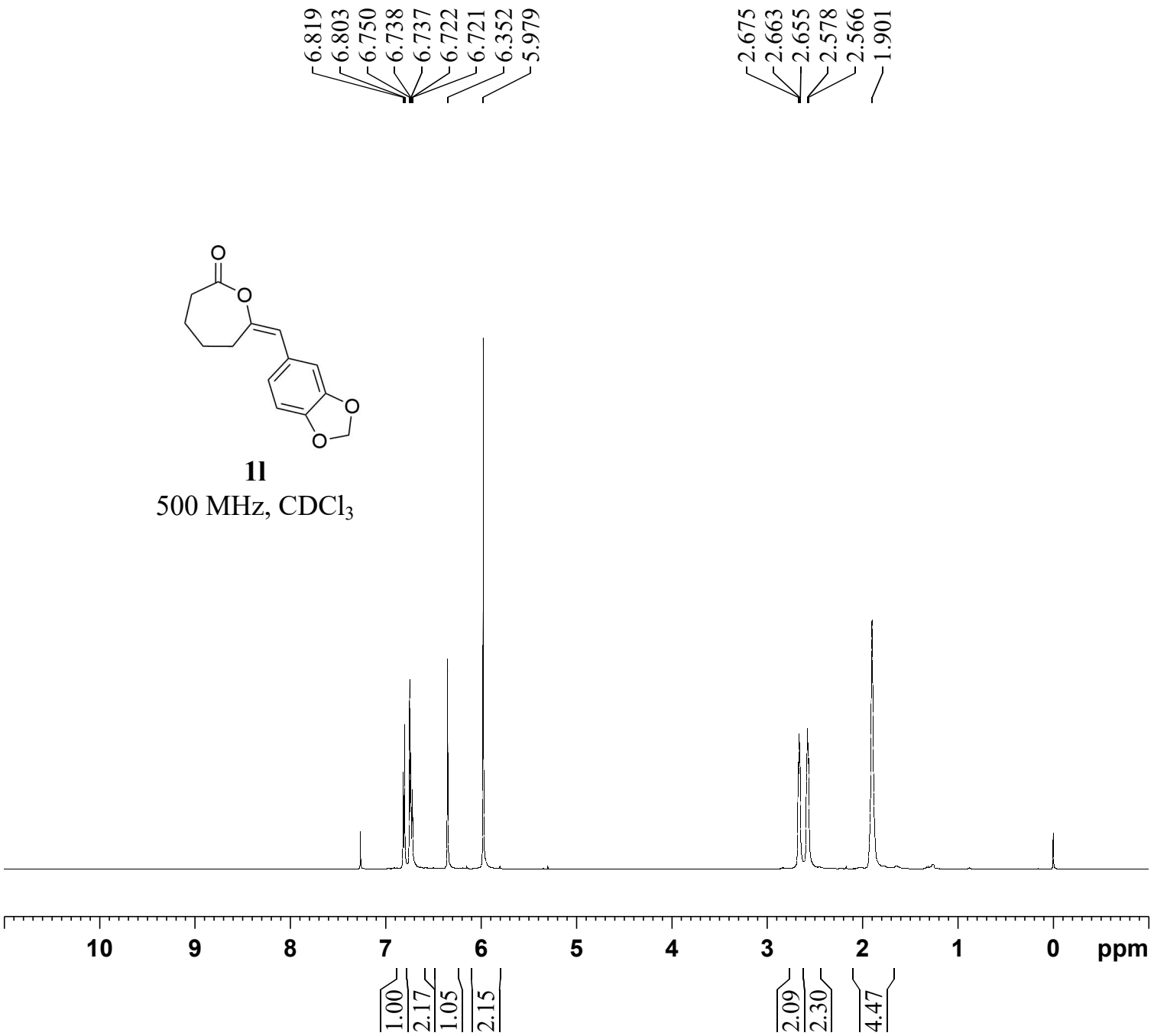
1k

500 MHz, $CDCl_3$

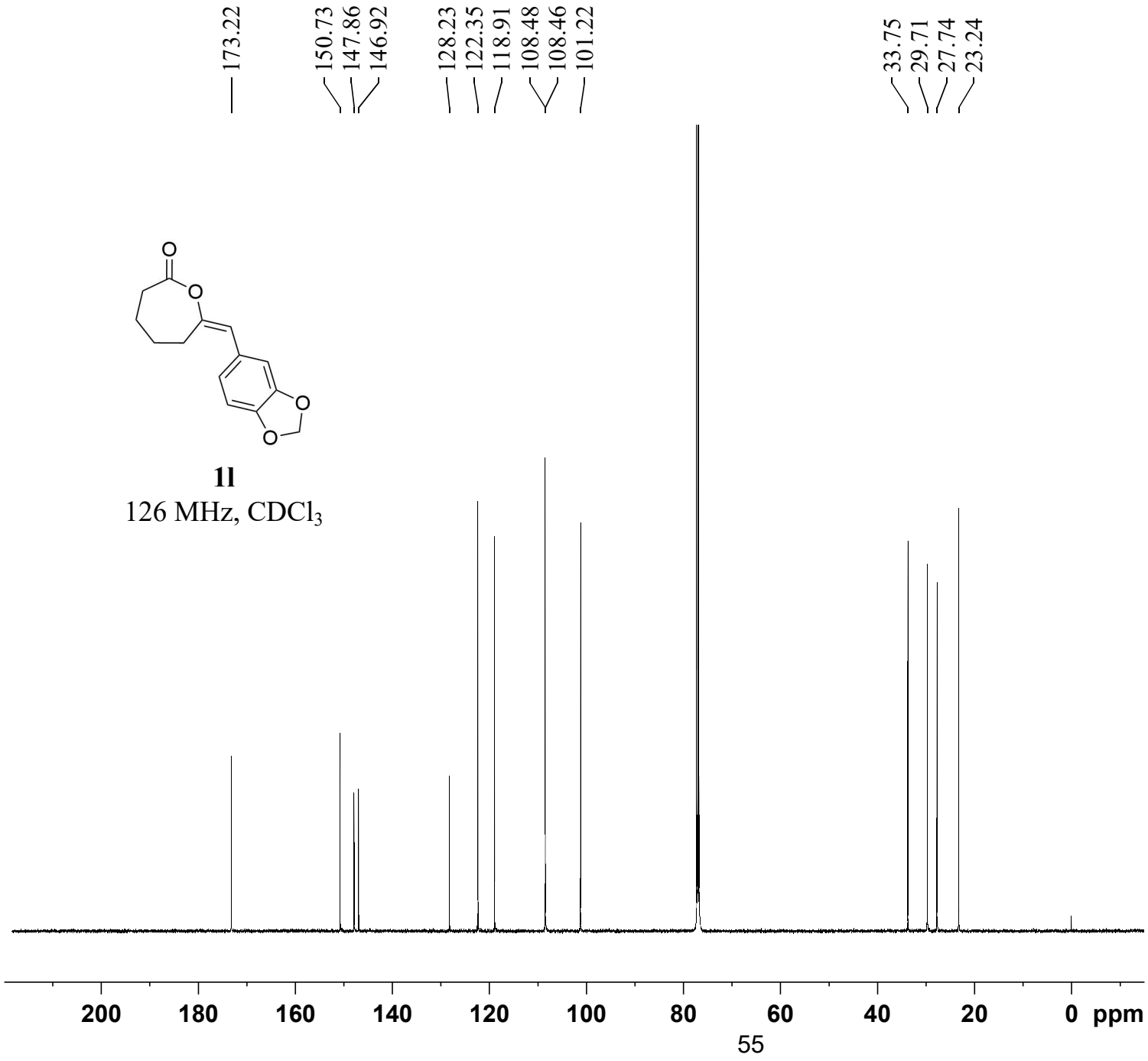
```
Current Data Parameters
NAME        500M-2020xia
EXPNO                 36
PROCNO                 1

F2 - Acquisition Parameters
Date_           20210110
Time               22.29
INSTRUM            spect
PROBHD   5 mm CPPBBO BB
PULPROG             zg30
TD                 65536
SOLVENT            CDCl3
NS                    16
DS                     2
SWH           10000.000 Hz
FIDRES         0.152588 Hz
AQ            3.2767999 sec
RG                 49.27
DW                50.000 usec
DE                 6.50 usec
TE                298.2 K
D1           1.00000000 sec
D11      0 sec
TD0                    1

======== CHANNEL f1 ========
SFO1         500.1330885 MHz
NUC1                   1H
P1                 10.59 usec
PLW1         20.00000000 W

======== CHANNEL f2 ========
SFO2         500.1330885 MHz
NUC2                  off
CPDPRG[2
PCPD2     0 usec
PLW2      0 W
PLW12     0 W
PLW13     0 W

F2 - Processing parameters
SI                 65536
SF           500.1300159 MHz
WDW                   EM
SSB       0
LB                 0.30 Hz
GB        0
PC                 1.00
```




```
Current Data Parameters
NAME        500M-2020xia
EXPNO                 37
PROCNO                 1

F2 - Acquisition Parameters
Date_           20210110
Time              23.12
INSTRUM           spect
PROBHD   5 mm CPPBBO BB
PULPROG          zgpg30
TD                65536
SOLVENT           CDCl3
NS                  800
DS                    4
SWH           29761.904 Hz
FIDRES         0.454131 Hz
AQ             1.1010048 sec
RG                192.89
DW                16.800 usec
DE                18.00 usec
TE                298.2 K
D1            2.00000000 sec
D11           0.03000000 sec
TD0                   1

======== CHANNEL f1 ========
SFO1         125.7703637 MHz
NUC1                 13C
P1                  9.80 usec
PLW1         57.00000000 W

======== CHANNEL f2 ========
SFO2         500.1320005 MHz
NUC2                 1H
CPDPRG[2          waltz16
PCPD2               80.00 usec
PLW2         20.00000000 W
PLW12         0.35778001 W
PLW13         0.22898000 W

F2 - Processing parameters
SI                  32768
SF           125.7577916 MHz
WDW                  EM
SSB       0
LB                  1.00 Hz
GB        0
PC                  1.40
```



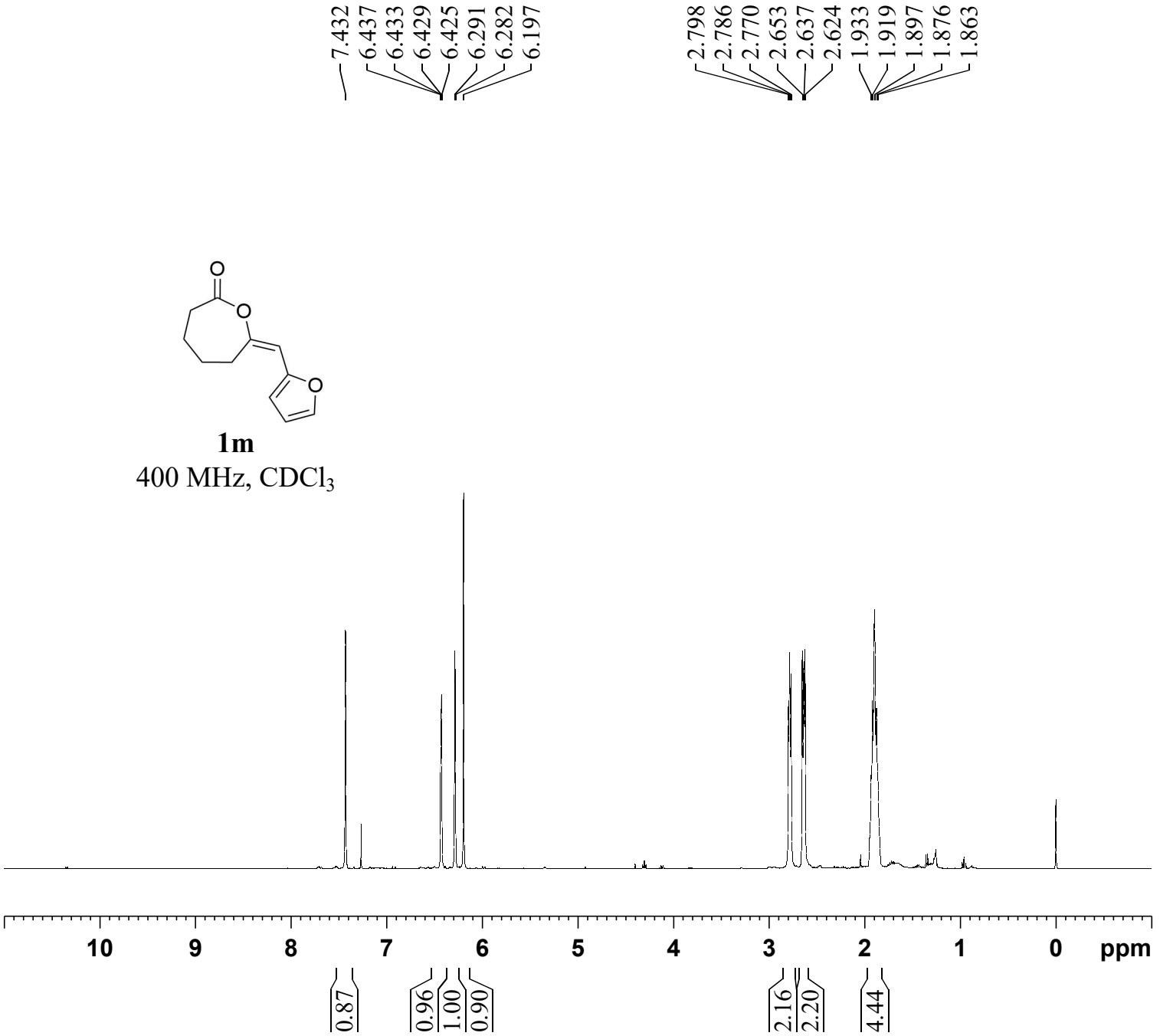

```
Current Data Parameters
NAME        500M-2020xia
EXPNO                 58
PROCNO                 1

F2 - Acquisition Parameters
Date_           20210111
Time              21.57
INSTRUM           spect
PROBHD   5 mm CPPBBO BB
PULPROG             zg30
TD                65536
SOLVENT           CDCl3
NS                   16
DS                    2
SWH           10000.000 Hz
FIDRES         0.152588 Hz
AQ            3.2767999 sec
RG                31.72
DW               50.000 usec
DE                6.50 usec
TE               298.2 K
D1          1.00000000 sec
D11      0 sec
TD0                   1

======== CHANNEL f1 ========
SFO1        500.1330885 MHz
NUC1                  1H
P1                10.59 usec
PLW1        20.00000000 W

======== CHANNEL f2 ========
SFO2        500.1330885 MHz
NUC2                 off
CPDPRG[2
PCPD2     0 usec
PLW2      0 W
PLW12     0 W
PLW13     0 W

F2 - Processing parameters
SI                  65536
SF          500.1300093 MHz
WDW                    EM
SSB       0
LB                  0.30 Hz
GB        0
PC                  1.00
```



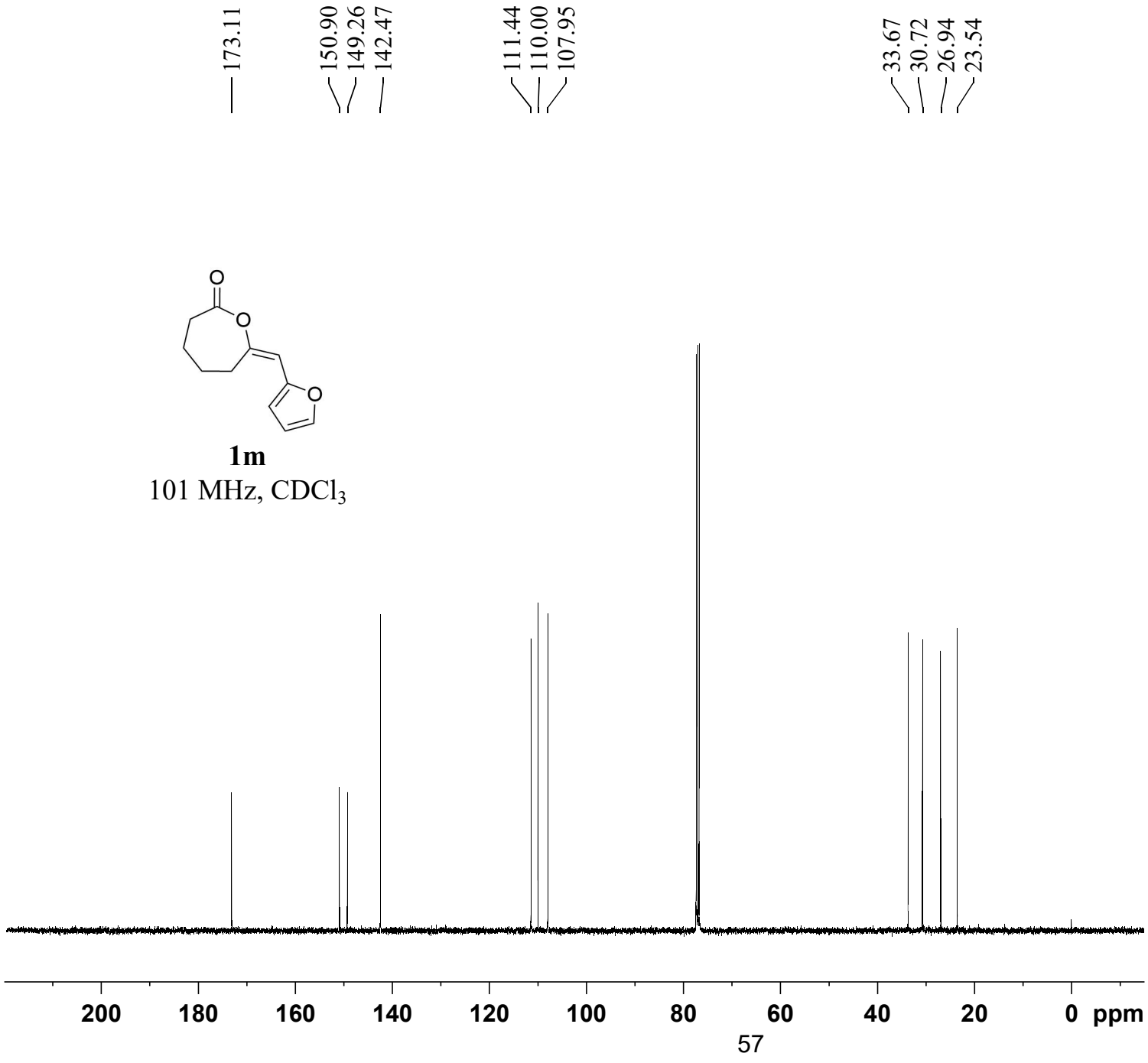
1l

126 MHz, $CDCl_3$

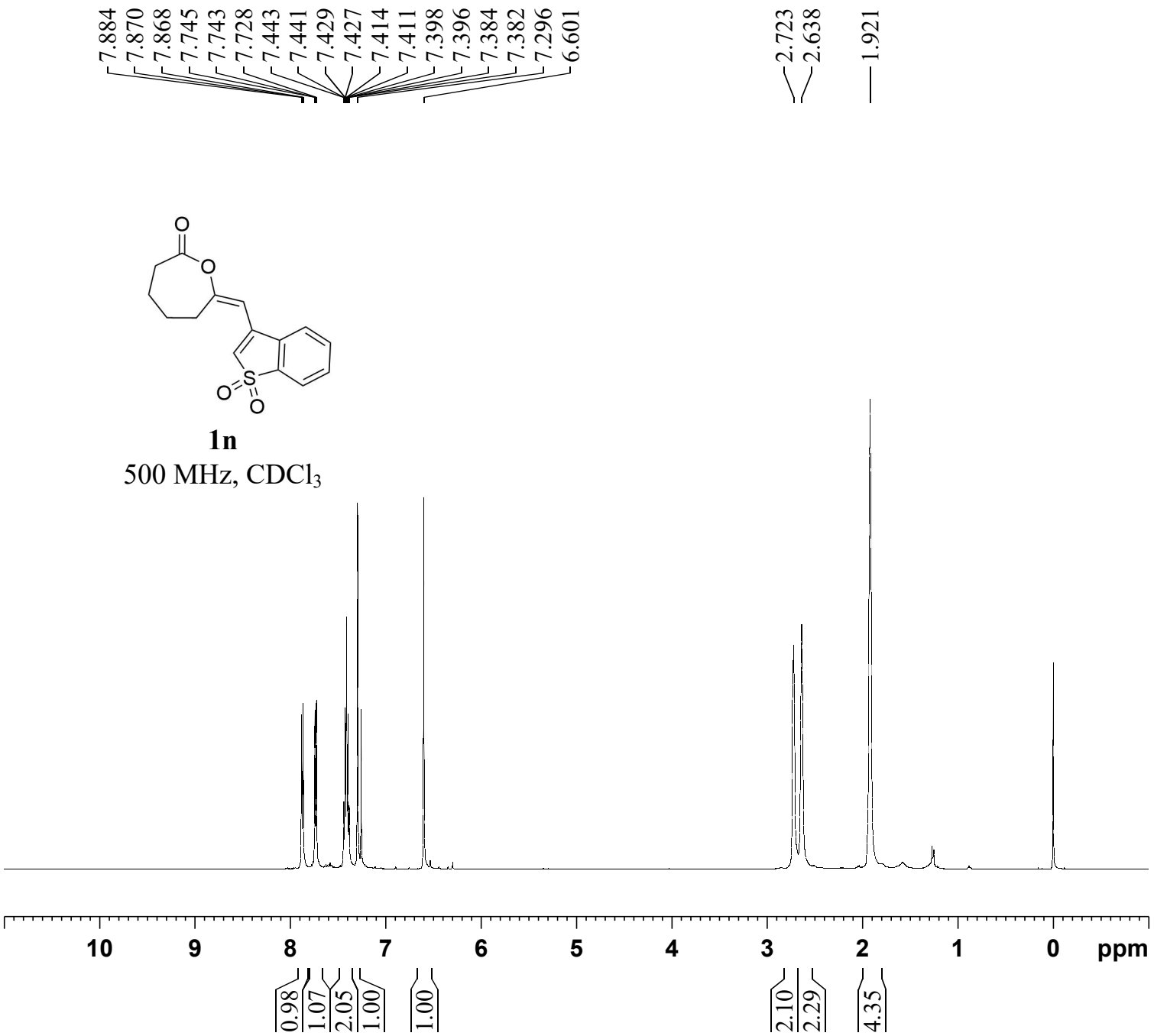
```
Current Data Parameters
NAME        500M-2020xia
EXPNO                 40
PROCNO                 1

F2 - Acquisition Parameters
Date_           20210111
Time               1.06
INSTRUM           spect
PROBHD   5 mm CPPBBO BB
PULPROG          zgpg30
TD                65536
SOLVENT           CDCl3
NS                  800
DS                    4
SWH           29761.904 Hz
FIDRES         0.454131 Hz
AQ            1.1010048 sec
RG               192.89
DW               16.800 usec
DE               18.00 usec
TE               298.2 K
D1           2.00000000 sec
D11          0.03000000 sec
TD0                   1

======== CHANNEL f1 ========
SFO1        125.7703637 MHz
NUC1                13C
P1                 9.80 usec
PLW1        57.00000000 W

======== CHANNEL f2 ========
SFO2        500.1320005 MHz
NUC2                1H
CPDPRG[2         waltz16
PCPD2             80.00 usec
PLW2        20.00000000 W
PLW12        0.35778001 W
PLW13        0.22898000 W

F2 - Processing parameters
SI                 32768
SF          125.7577898 MHz
WDW                   EM
SSB       0
LB                 1.00 Hz
GB        0
PC                 1.40
```



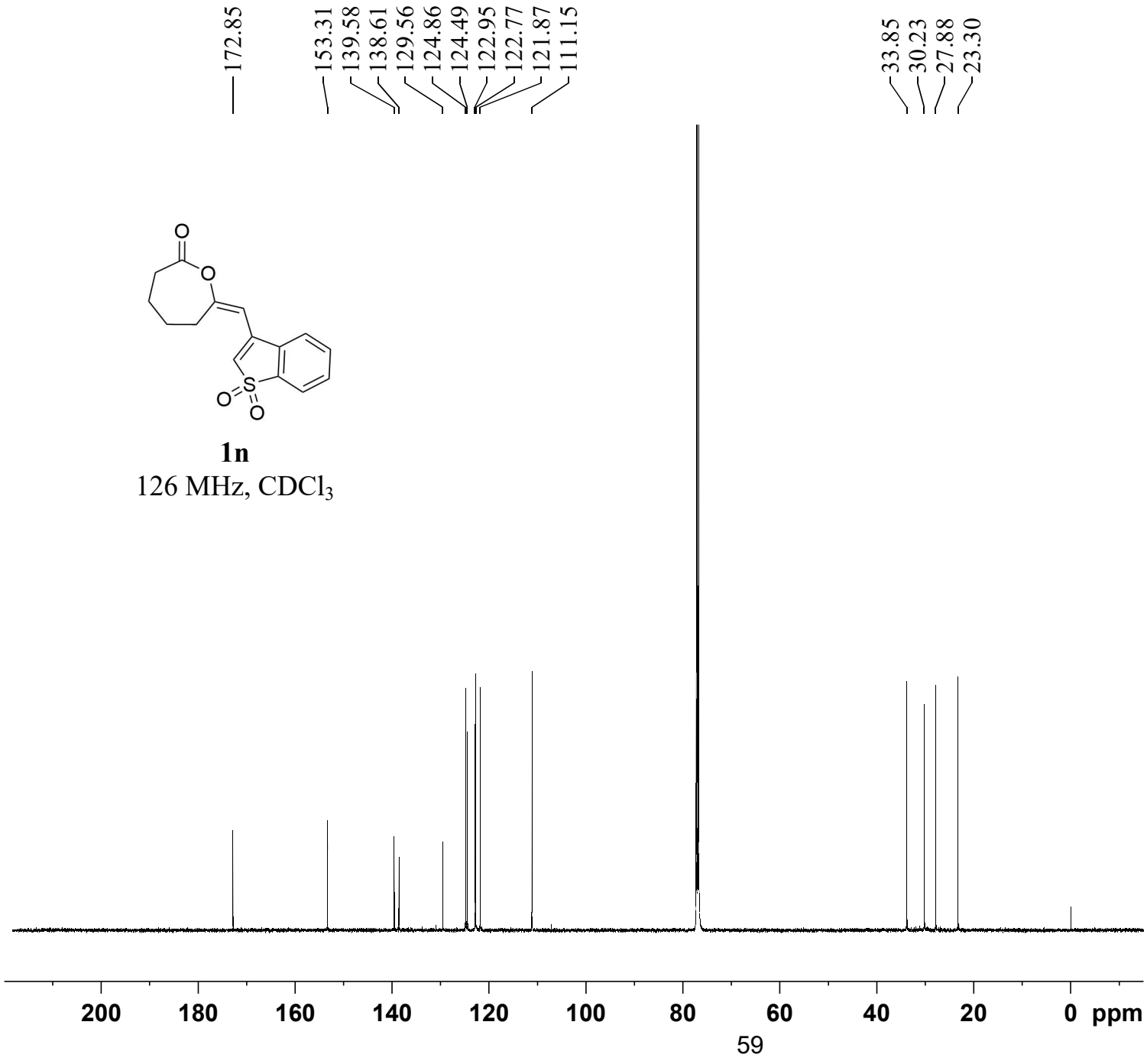

```
Current Data Parameters
NAME                 400xia
EXPNO                     7
PROCNO                    1

F2 - Acquisition Parameters
Date_             20201017
Time                  1.25
INSTRUM              spect
PROBHD   5 mm PABBO BB/
PULPROG               zg30
TD                   32768
SOLVENT              CDCl3
NS                      16
DS                       0
SWH               8012.820 Hz
FIDRES            0.244532 Hz
AQ               2.0447233 sec
RG                  125.02
DW                  62.400 usec
DE                   6.50 usec
TE                  298.5 K
D1              2.00000000 sec
D11       0 sec
TD0                       1

======== CHANNEL f1 ========
SFO1           400.2424716 MHz
NUC1                    1H
P1                   14.30 usec
PLW1           12.00000000 W

======== CHANNEL f2 ========
SFO2           400.2424716 MHz
NUC2                   off
CPDPRG[2
PCPD2     0 usec
PLW2      0 W
PLW12     0 W
PLW13     0 W

F2 - Processing parameters
SI                    65536
SF           400.2400062 MHz
WDW                     EM
SSB       0
LB                   0.30 Hz
GB        0
PC                   1.00
```




```
Current Data Parameters
NAME                 400xia
EXPNO                     8
PROCNO                    1

F2 - Acquisition Parameters
Date_             20201017
Time                  2.24
INSTRUM              spect
PROBHD   5 mm PABBO BB/
PULPROG             zgpg30
TD                   65536
SOLVENT              CDCl3
NS                    1024
DS                       4
SWH              24038.461 Hz
FIDRES            0.366798 Hz
AQ               1.3631488 sec
RG                  206.33
DW                  20.800 usec
DE                   6.50 usec
TE                  299.1 K
D1              2.00000000 sec
D11             0.03000000 sec
TD0                       1

======== CHANNEL f1 ========
SFO1           100.6504916 MHz
NUC1                   13C
P1                   10.00 usec
PLW1           54.00000000 W

======== CHANNEL f2 ========
SFO2           400.2416010 MHz
NUC2                   1H
CPDPRG[2           waltz16
PCPD2                90.00 usec
PLW2           12.00000000 W
PLW12           0.30294999 W
PLW13           0.24539000 W

F2 - Processing parameters
SI                    32768
SF             100.6404280 MHz
WDW                      EM
SSB       0
LB                    1.00 Hz
GB        0
PC                    1.40
```



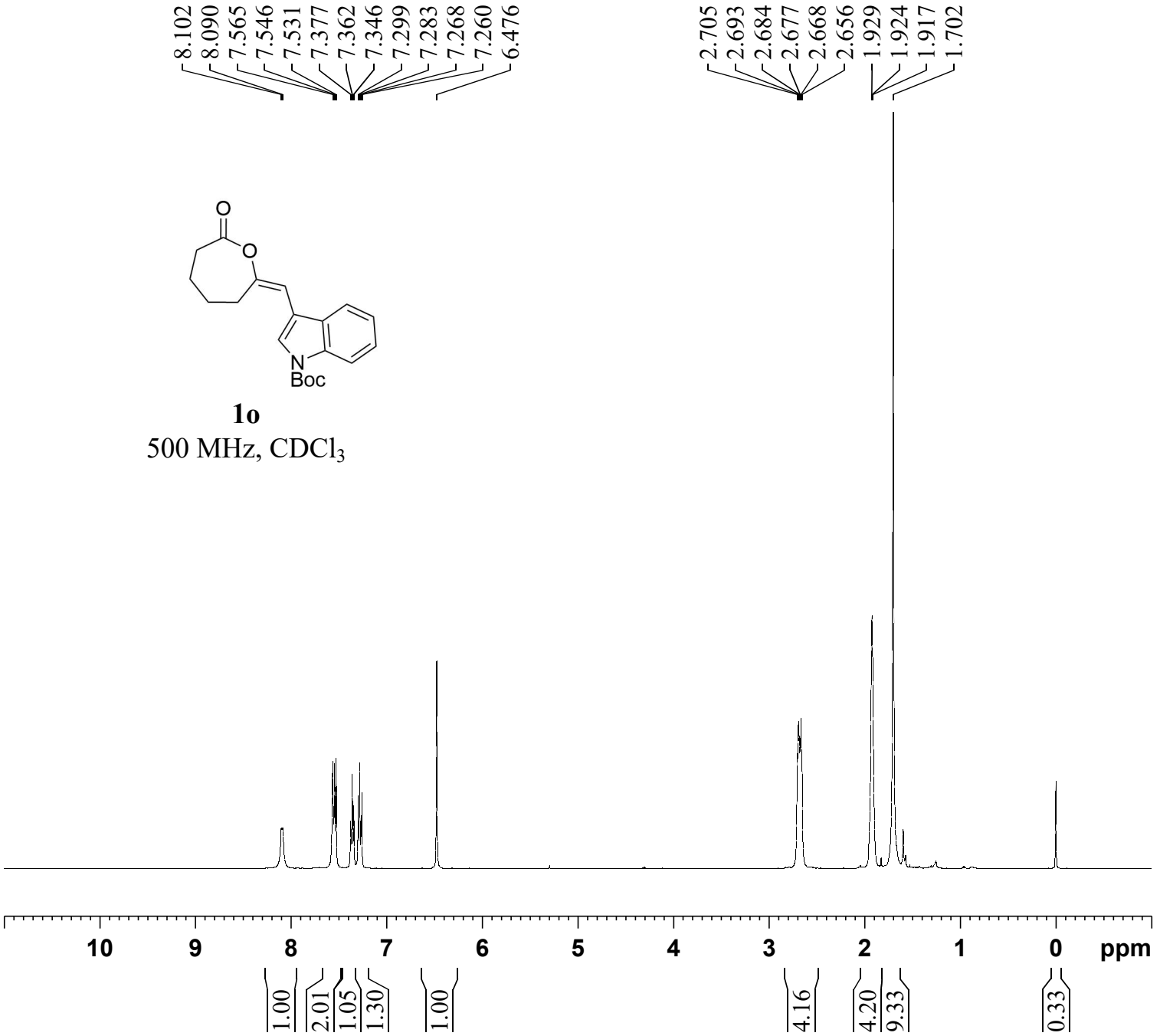

```
Current Data Parameters
NAME        500M-2020xia
EXPNO                 59
PROCNO                 1

F2 - Acquisition Parameters
Date_           20210111
Time              22.02
INSTRUM           spect
PROBHD   5 mm CPPBBO BB
PULPROG             zg30
TD                65536
SOLVENT           CDCl3
NS                   16
DS                    2
SWH           10000.000 Hz
FIDRES         0.152588 Hz
AQ            3.2767999 sec
RG                31.72
DW               50.000 usec
DE                6.50 usec
TE               298.2 K
D1          1.00000000 sec
D11      0 sec
TD0                   1

======== CHANNEL f1 ========
SFO1        500.1330885 MHz
NUC1                  1H
P1                10.59 usec
PLW1        20.00000000 W

======== CHANNEL f2 ========
SFO2        500.1330885 MHz
NUC2                 off
CPDPRG[2
PCPD2     0 usec
PLW2      0 W
PLW12     0 W
PLW13     0 W

F2 - Processing parameters
SI                  65536
SF          500.1300134 MHz
WDW                    EM
SSB       0
LB                  0.30 Hz
GB        0
PC                  1.00
```



1n

126 MHz, $CDCl_3$

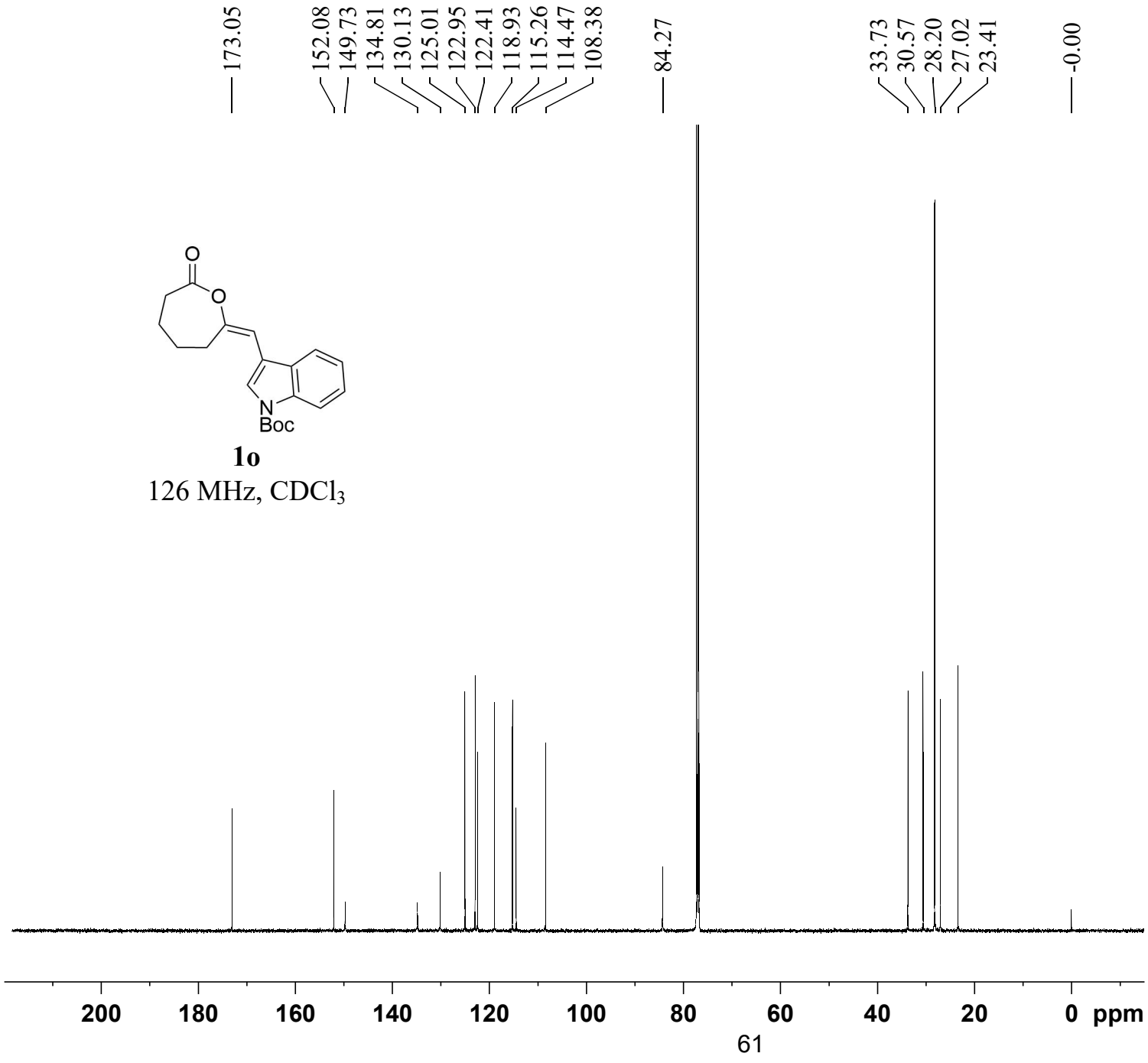
```
Current Data Parameters
NAME        500M-2020xia
EXPNO                 41
PROCNO                 1

F2 - Acquisition Parameters
Date_           20210111
Time                2.03
INSTRUM            spect
PROBHD   5 mm CPPBBO BB
PULPROG          zgpg30
TD                65536
SOLVENT           CDCl3
NS                 1024
DS                    4
SWH           29761.904 Hz
FIDRES         0.454131 Hz
AQ            1.1010048 sec
RG               192.89
DW               16.800 usec
DE               18.00 usec
TE               298.2 K
D1           2.00000000 sec
D11          0.03000000 sec
TD0                   1

======== CHANNEL f1 ========
SFO1        125.7703637 MHz
NUC1                13C
P1                 9.80 usec
PLW1        57.00000000 W

======== CHANNEL f2 ========
SFO2        500.1320005 MHz
NUC2                1H
CPDPRG[2         waltz16
PCPD2             80.00 usec
PLW2        20.00000000 W
PLW12        0.35778001 W
PLW13        0.22898000 W

F2 - Processing parameters
SI                 32768
SF          125.7577906 MHz
WDW                   EM
SSB       0
LB                 1.00 Hz
GB        0
PC                 1.40
```



1o

500 MHz, $CDCl_3$

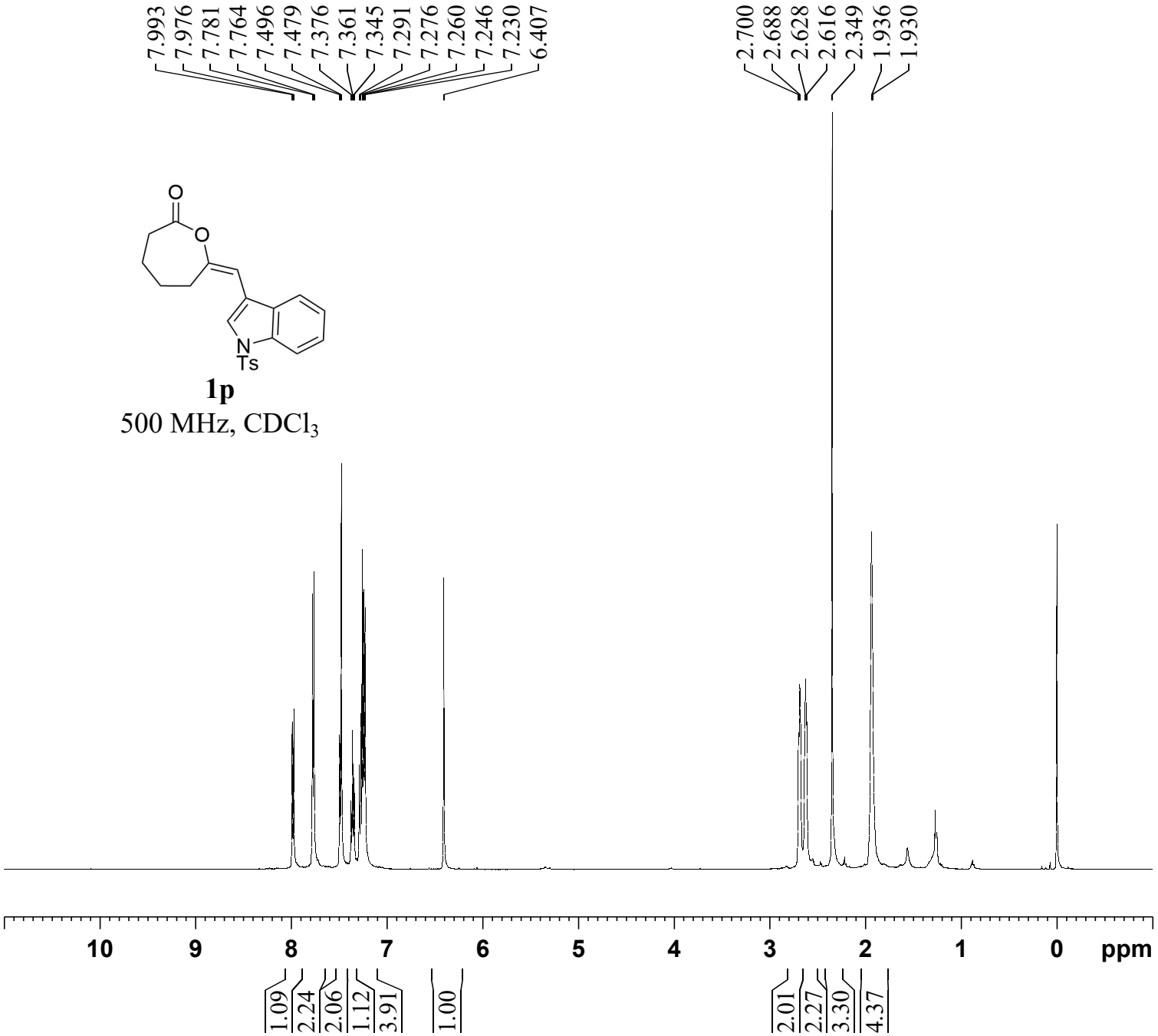
```
Current Data Parameters
NAME        500M-2020xia
EXPNO                 11
PROCNO                 1

F2 - Acquisition Parameters
Date_           20201118
Time                8.27
INSTRUM            spect
PROBHD   5 mm CPPBBO BB
PULPROG             zg30
TD                 65536
SOLVENT            CDCl3
NS                    16
DS                     2
SWH           10000.000 Hz
FIDRES         0.152588 Hz
AQ            3.2767999 sec
RG                 31.72
DW                50.000 usec
DE                 6.50 usec
TE                298.2 K
D1           1.00000000 sec
D11      0 sec
TD0                    1

======== CHANNEL f1 ========
SFO1         500.1330885 MHz
NUC1                   1H
P1                 10.59 usec
PLW1         20.00000000 W

======== CHANNEL f2 ========
SFO2         500.1330885 MHz
NUC2                  off
CPDPRG[2
PCPD2     0 usec
PLW2      0 W
PLW12     0 W
PLW13     0 W

F2 - Processing parameters
SI                 65536
SF           500.1300118 MHz
WDW                   EM
SSB       0
LB                 0.30 Hz
GB        0
PC                 1.00
```



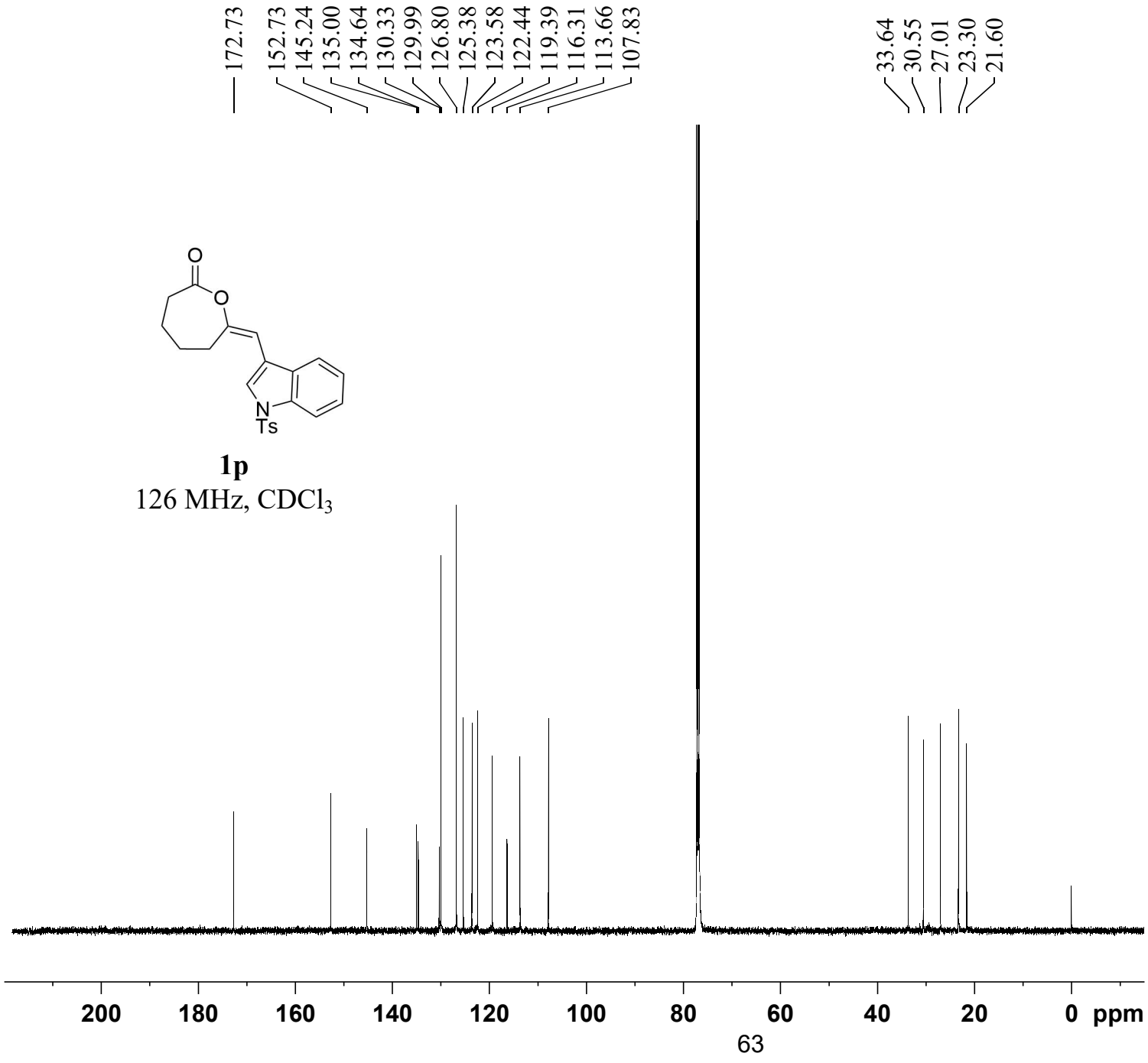
1o

126 MHz, $CDCl_3$

```
Current Data Parameters
NAME        500M-2020xia
EXPNO                 12
PROCNO                 1

F2 - Acquisition Parameters
Date_           20201118
Time                9.21
INSTRUM            spect
PROBHD   5 mm CPPBBO BB
PULPROG          zgpg30
TD                65536
SOLVENT           CDCl3
NS                 1024
DS                    4
SWH           29761.904 Hz
FIDRES         0.454131 Hz
AQ            1.1010048 sec
RG               192.89
DW               16.800 usec
DE               18.00 usec
TE               298.2 K
D1           2.00000000 sec
D11          0.03000000 sec
TD0                   1

======== CHANNEL f1 ========
SFO1        125.7703637 MHz
NUC1                13C
P1                9.80 usec
PLW1        57.00000000 W

======== CHANNEL f2 ========
SFO2        500.1320005 MHz
NUC2                1H
CPDPRG[2         waltz16
PCPD2             80.00 usec
PLW2        20.00000000 W
PLW12        0.35778001 W
PLW13        0.22898000 W

F2 - Processing parameters
SI                 32768
SF          125.7577900 MHz
WDW                   EM
SSB       0
LB                1.00 Hz
GB        0
PC                1.40
```



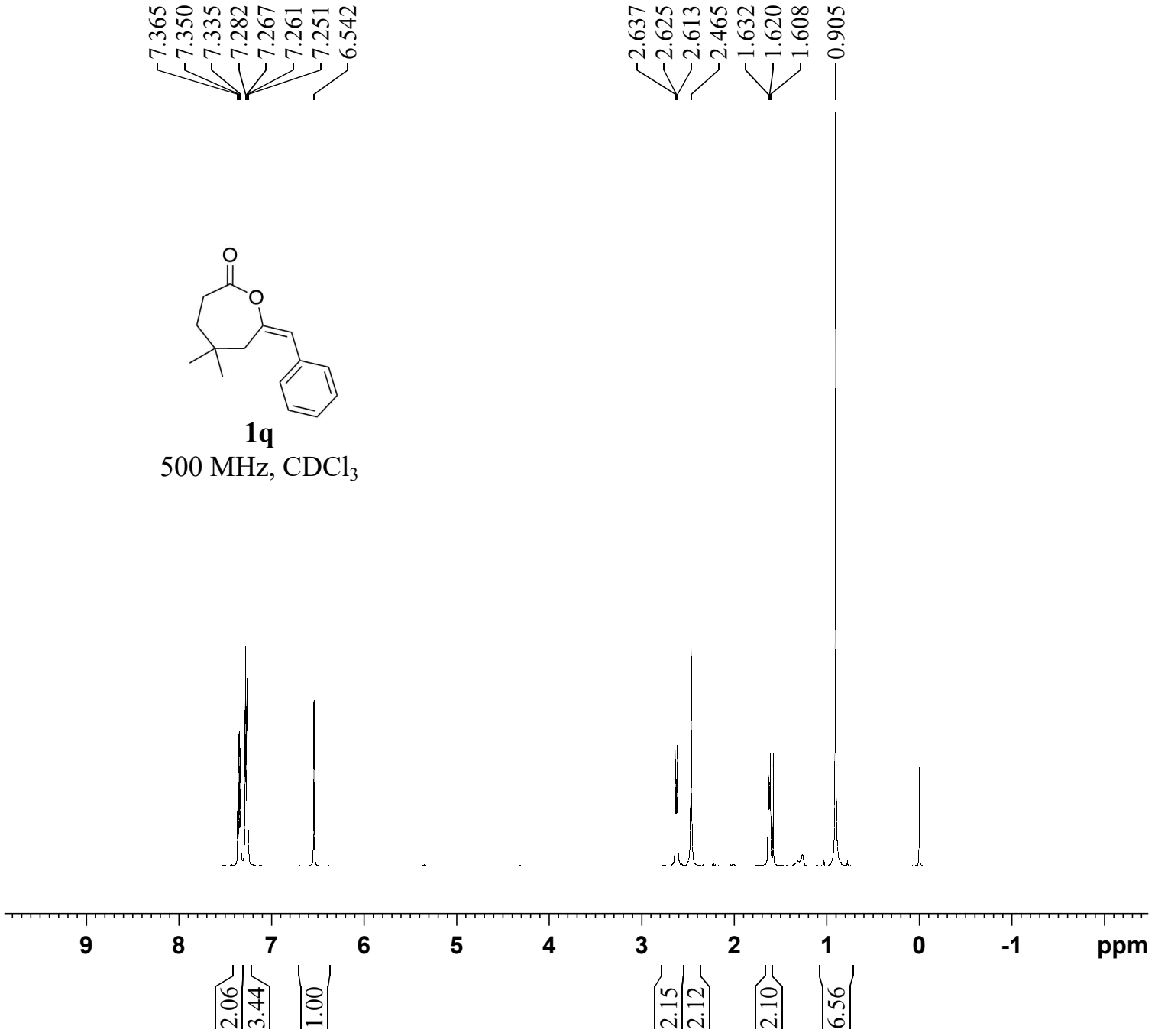

```
Current Data Parameters
NAME        500M-2020xia
EXPNO                 42
PROCNO                 1

F2 - Acquisition Parameters
Date_           20210111
Time                2.07
INSTRUM            spect
PROBHD   5 mm CPPBBO BB
PULPROG             zg30
TD                 65536
SOLVENT            CDCl3
NS                    16
DS                     2
SWH           10000.000 Hz
FIDRES         0.152588 Hz
AQ            3.2767999 sec
RG                 62.06
DW                50.000 usec
DE                 6.50 usec
TE                298.2 K
D1           1.00000000 sec
D11      0 sec
TD0                    1

======== CHANNEL f1 ========
SFO1         500.1330885 MHz
NUC1                   1H
P1                 10.59 usec
PLW1         20.00000000 W

======== CHANNEL f2 ========
SFO2         500.1330885 MHz
NUC2                  off
CPDPRG[2
PCPD2     0 usec
PLW2      0 W
PLW12     0 W
PLW13     0 W

F2 - Processing parameters
SI                 65536
SF          500.1300125 MHz
WDW                   EM
SSB       0
LB                 0.30 Hz
GB        0
PC                 1.00
```



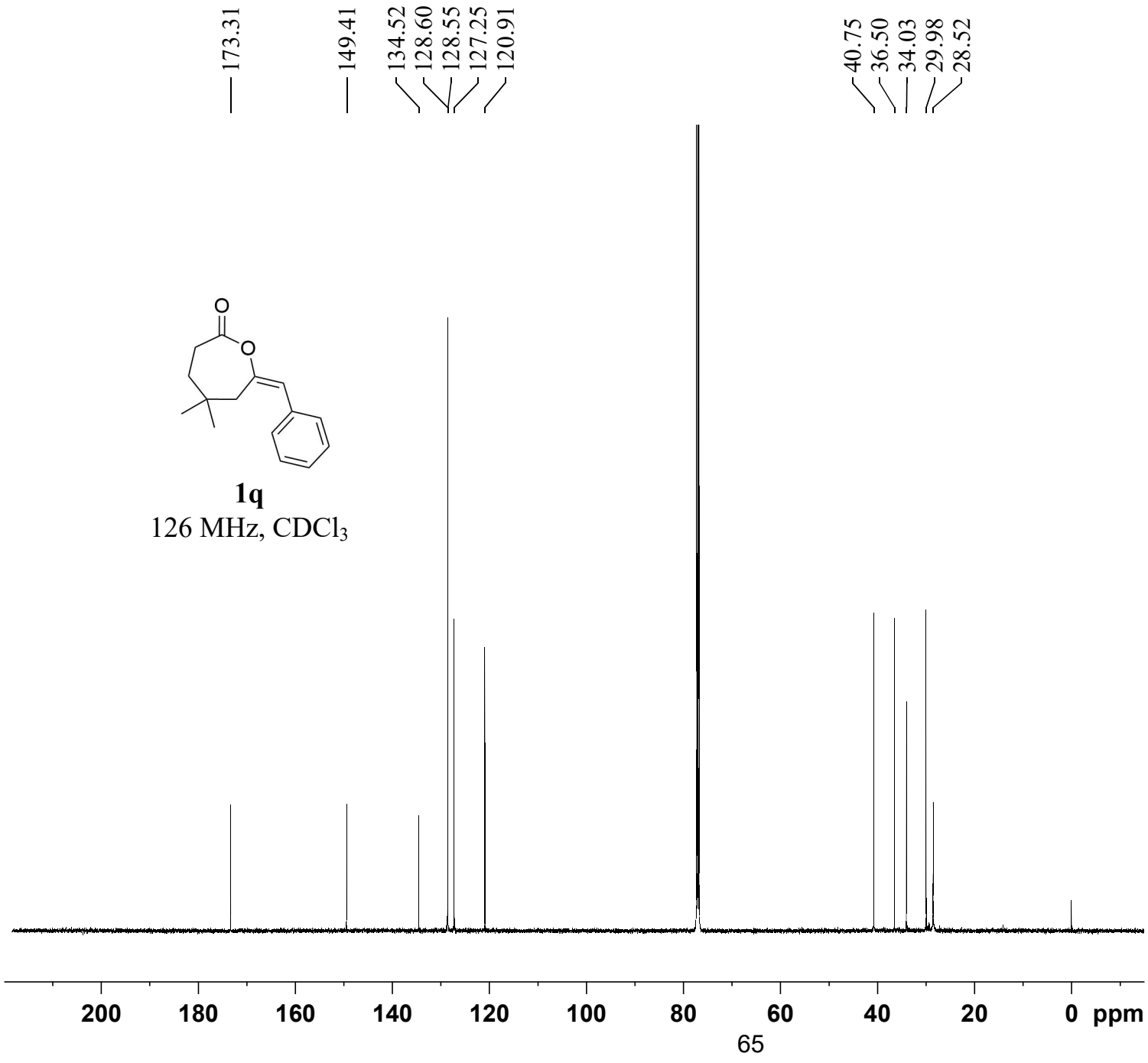
1p

126 MHz, $CDCl_3$

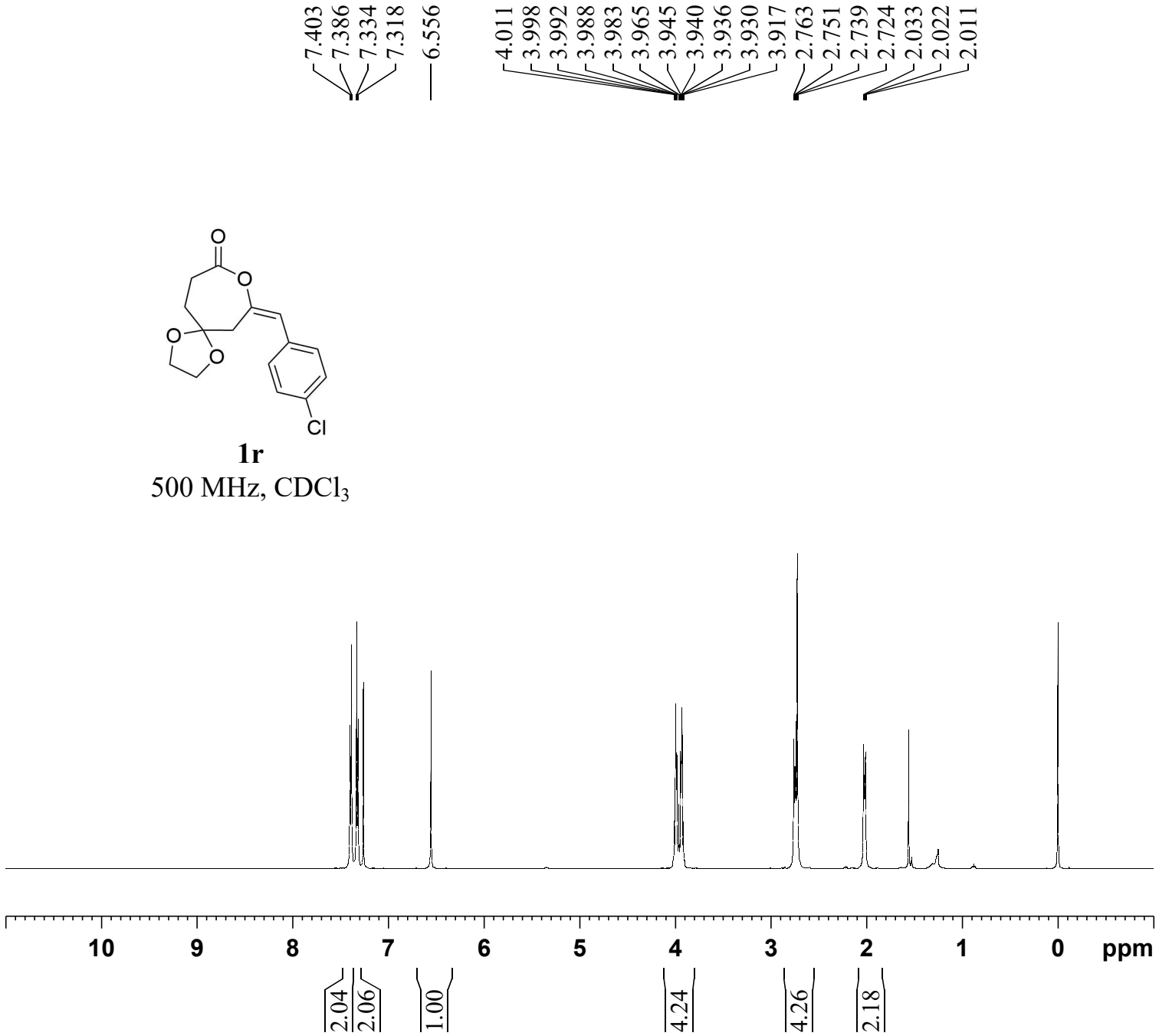
```
Current Data Parameters
NAME        500M-2020xia
EXPNO                 43
PROCNO                 1

F2 - Acquisition Parameters
Date_           20210111
Time                3.01
INSTRUM            spect
PROBHD   5 mm CPPBBO BB
PULPROG          zgpg30
TD                65536
SOLVENT           CDCl3
NS                 1024
DS                    4
SWH           29761.904 Hz
FIDRES         0.454131 Hz
AQ            1.1010048 sec
RG               192.89
DW               16.800 usec
DE               18.00 usec
TE               298.2 K
D1           2.00000000 sec
D11          0.03000000 sec
TD0                   1

======== CHANNEL f1 ========
SFO1        125.7703637 MHz
NUC1                13C
P1                 9.80 usec
PLW1        57.00000000 W

======== CHANNEL f2 ========
SFO2        500.1320005 MHz
NUC2                1H
CPDPRG[2         waltz16
PCPD2             80.00 usec
PLW2        20.00000000 W
PLW12        0.35778001 W
PLW13        0.22898000 W

F2 - Processing parameters
SI                 32768
SF          125.7577899 MHz
WDW                   EM
SSB       0
LB                 1.00 Hz
GB        0
PC                 1.40
```




```
Current Data Parameters
NAME       500M-2020xia
EXPNO                 3
PROCNO                1

F2 - Acquisition Parameters
Date_          20201118
Time               4.32
INSTRUM           spect
PROBHD   5 mm CPPBBO BB
PULPROG             zg30
TD                 65536
SOLVENT            CDCl3
NS                    16
DS                     2
SWH           10000.000 Hz
FIDRES         0.152588 Hz
AQ            3.2767999 sec
RG                 31.72
DW                50.000 usec
DE                 6.50 usec
TE                298.2 K
D1           1.00000000 sec
D11      0 sec
TD0                    1

======== CHANNEL f1 ========
SFO1        500.1330885 MHz
NUC1                   1H
P1                 10.59 usec
PLW1        20.00000000 W

======== CHANNEL f2 ========
SFO2        500.1330885 MHz
NUC2                  off
CPDPRG[2
PCPD2     0 usec
PLW2      0 W
PLW12     0 W
PLW13     0 W

F2 - Processing parameters
SI                 65536
SF          500.1300113 MHz
WDW                   EM
SSB       0
LB                 0.30 Hz
GB        0
PC                 1.00
```



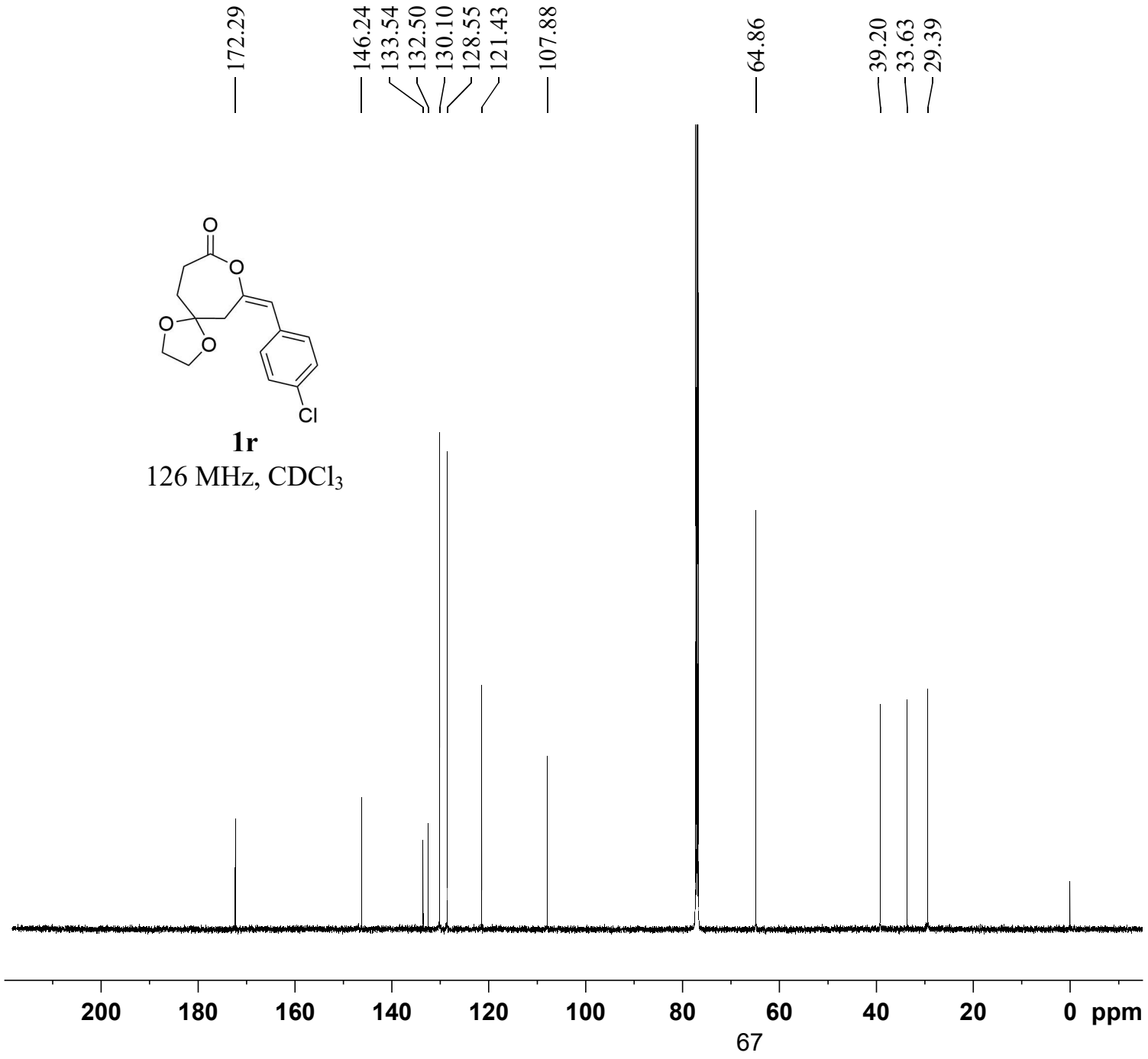

```
Current Data Parameters
NAME        500M-2020xia
EXPNO                  4
PROCNO                 1

F2 - Acquisition Parameters
Date_           20201118
Time               5.26
INSTRUM           spect
PROBHD   5 mm CPPBBO BB
PULPROG          zgpg30
TD                65536
SOLVENT           CDCl3
NS                 1024
DS                    4
SWH           29761.904 Hz
FIDRES         0.454131 Hz
AQ             1.1010048 sec
RG                192.89
DW                16.800 usec
DE                18.00 usec
TE                298.2 K
D1            2.00000000 sec
D11           0.03000000 sec
TD0                    1

======== CHANNEL f1 ========
SFO1         125.7703637 MHz
NUC1                 13C
P1                  9.80 usec
PLW1         57.00000000 W

======== CHANNEL f2 ========
SFO2         500.1320005 MHz
NUC2                 1H
CPDPRG[2         waltz16
PCPD2              80.00 usec
PLW2         20.00000000 W
PLW12         0.35778001 W
PLW13         0.22898000 W

F2 - Processing parameters
SI                  32768
SF           125.7577898 MHz
WDW                    EM
SSB       0
LB                  1.00 Hz
GB        0
PC                  1.40
```



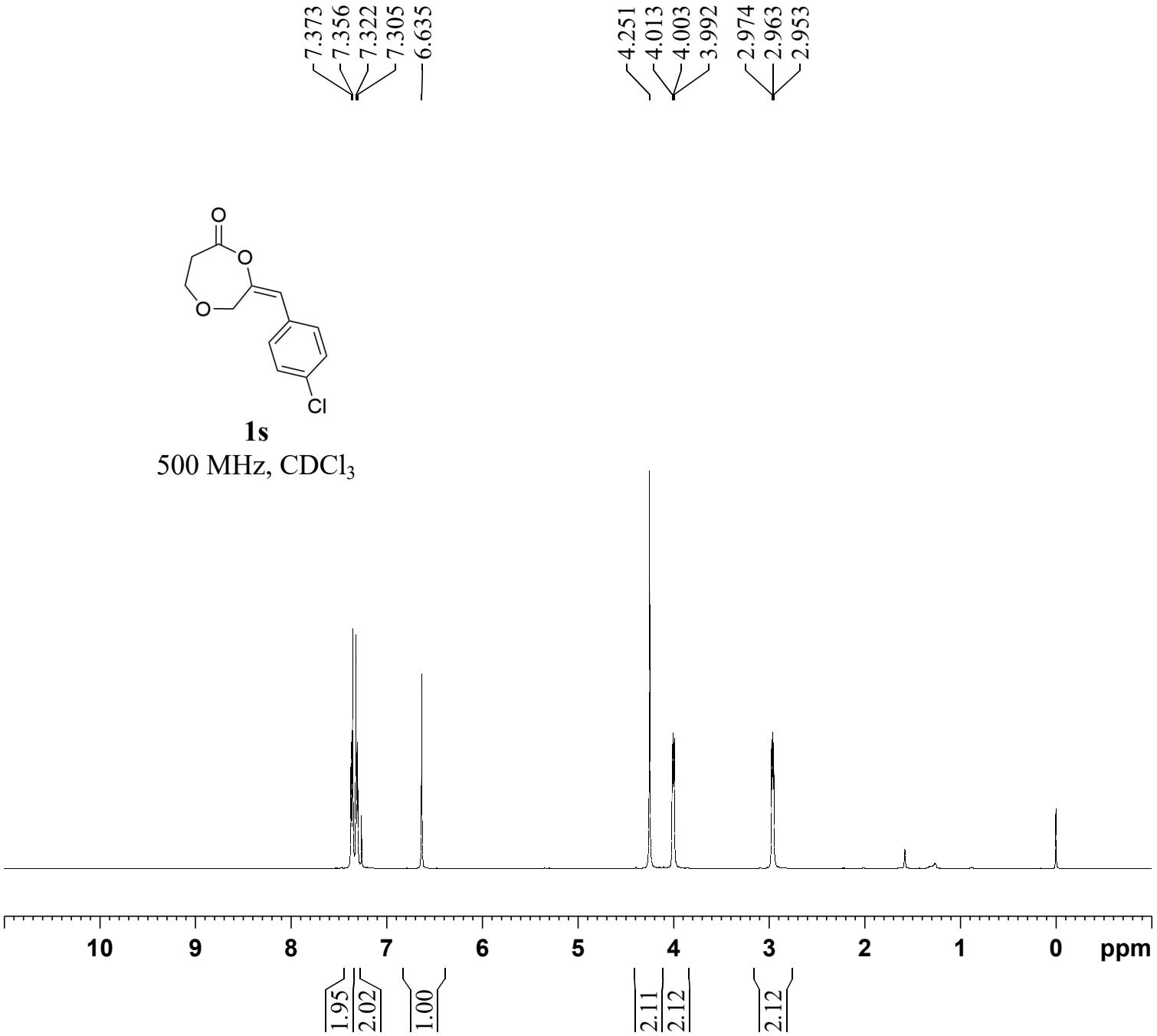
1r

500 MHz, $CDCl_3$

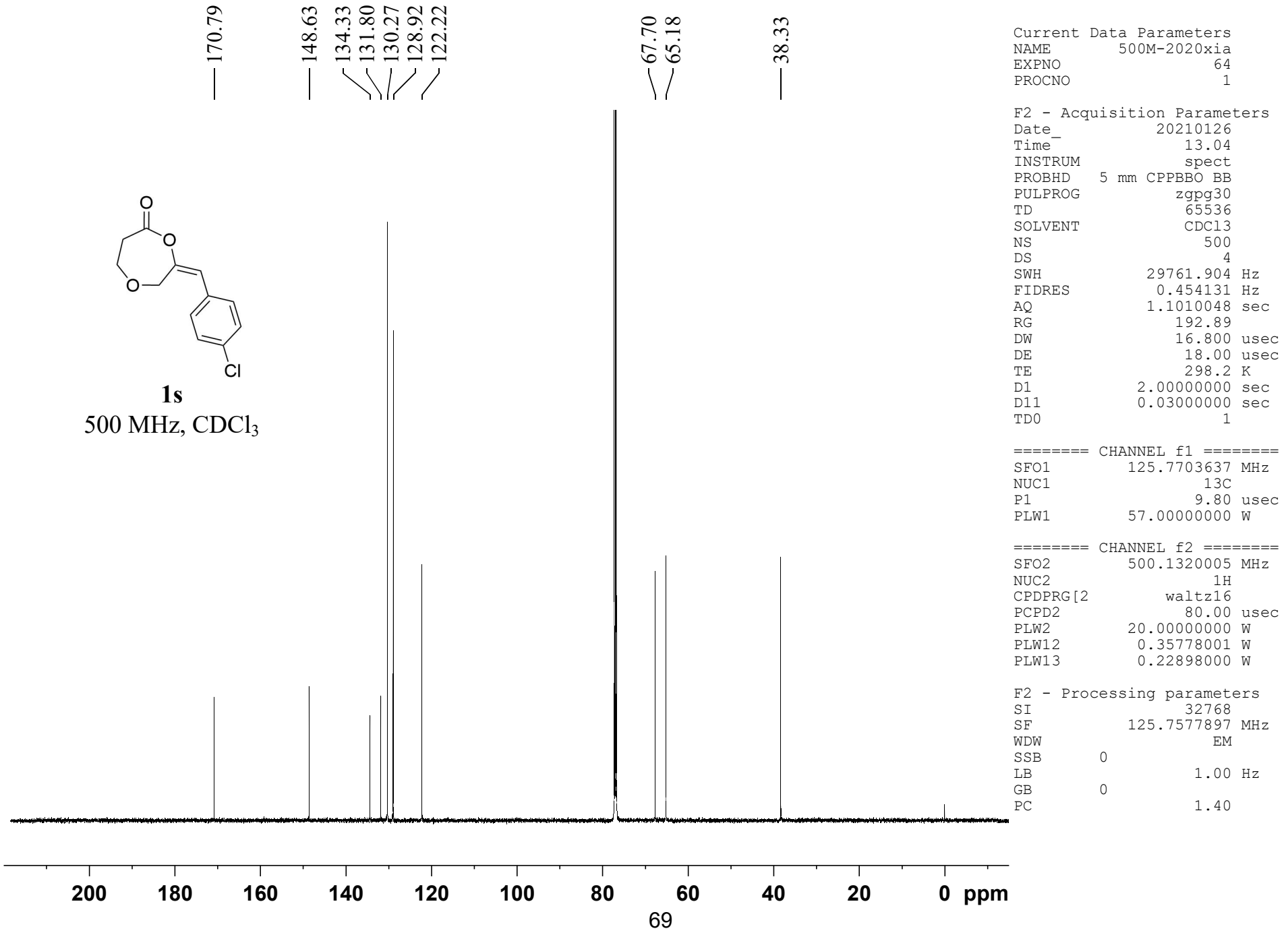
```
Current Data Parameters
NAME       500M-2020xia
EXPNO                15
PROCNO                1

F2 - Acquisition Parameters
Date_          20201118
Time              10.24
INSTRUM           spect
PROBHD   5 mm CPPBBO BB
PULPROG             zg30
TD                65536
SOLVENT           CDCl3
NS                   16
DS                    2
SWH           10000.000 Hz
FIDRES         0.152588 Hz
AQ            3.2767999 sec
RG                31.72
DW               50.000 usec
DE                6.50 usec
TE               298.2 K
D1          1.00000000 sec
D11      0 sec
TD0                   1

======== CHANNEL f1 ========
SFO1        500.1330885 MHz
NUC1                   1H
P1                 10.59 usec
PLW1        20.00000000 W

======== CHANNEL f2 ========
SFO2        500.1330885 MHz
NUC2                  off
CPDPRG[2
PCPD2     0 usec
PLW2      0 W
PLW12     0 W
PLW13     0 W

F2 - Processing parameters
SI                 65536
SF          500.1300109 MHz
WDW                   EM
SSB       0
LB                 0.30 Hz
GB        0
PC                 1.00
```



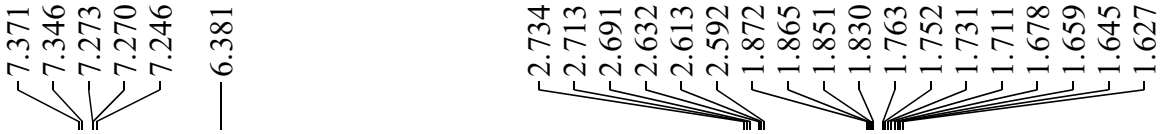

```
Current Data Parameters
NAME        500M-2020xia
EXPNO                 16
PROCNO                 1

F2 - Acquisition Parameters
Date_           20201118
Time               11.19
INSTRUM            spect
PROBHD   5 mm CPPBBO BB
PULPROG          zgpg30
TD                65536
SOLVENT           CDCl3
NS                 1024
DS                    4
SWH           29761.904 Hz
FIDRES         0.454131 Hz
AQ            1.1010048 sec
RG               192.89
DW               16.800 usec
DE               18.00 usec
TE               298.2 K
D1           2.00000000 sec
D11          0.03000000 sec
TD0                   1

======== CHANNEL f1 ========
SFO1        125.7703637 MHz
NUC1                13C
P1                 9.80 usec
PLW1        57.00000000 W

======== CHANNEL f2 ========
SFO2        500.1320005 MHz
NUC2                1H
CPDPRG[2         waltz16
PCPD2             80.00 usec
PLW2        20.00000000 W
PLW12        0.35778001 W
PLW13        0.22898000 W

F2 - Processing parameters
SI                 32768
SF          125.7577896 MHz
WDW                   EM
SSB       0
LB                 1.00 Hz
GB        0
PC                 1.40
```

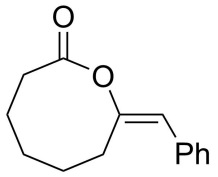



```
Current Data Parameters
NAME       500M-2020xia
EXPNO                63
PROCNO                1

F2 - Acquisition Parameters
Date_          20210126
Time              12.37
INSTRUM           spect
PROBHD   5 mm CPPBBO BB
PULPROG             zg30
TD                65536
SOLVENT           CDCl3
NS                   16
DS                    2
SWH           10000.000 Hz
FIDRES         0.152588 Hz
AQ            3.2767999 sec
RG                62.06
DW               50.000 usec
DE                6.50 usec
TE               298.2 K
D1           1.00000000 sec
D11      0 sec
TD0                   1

======== CHANNEL f1 ========
SFO1        500.1330885 MHz
NUC1                  1H
P1                10.59 usec
PLW1        20.00000000 W

======== CHANNEL f2 ========
SFO2        500.1330885 MHz
NUC2                 off
CPDPRG[2
PCPD2     0 usec
PLW2      0 W
PLW12     0 W
PLW13     0 W

F2 - Processing parameters
SI                  65536
SF          500.1300106 MHz
WDW                    EM
SSB       0
LB                  0.30 Hz
GB        0
PC                  1.00
```



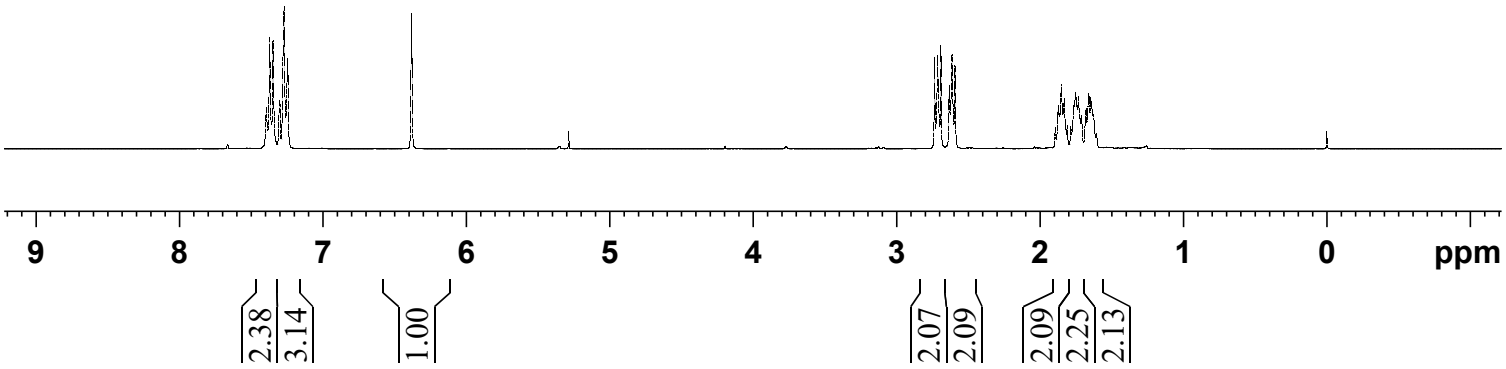

```
Current Data Parameters
NAME        500M-2020xia
EXPNO                 64
PROCNO                 1

F2 - Acquisition Parameters
Date_           20210126
Time              13.04
INSTRUM           spect
PROBHD   5 mm CPPBBO BB
PULPROG         zgpg30
TD                65536
SOLVENT           CDCl3
NS                  500
DS                    4
SWH           29761.904 Hz
FIDRES         0.454131 Hz
AQ             1.1010048 sec
RG                192.89
DW                16.800 usec
DE                18.00 usec
TE                298.2 K
D1            2.00000000 sec
D11           0.03000000 sec
TD0                   1

======== CHANNEL f1 ========
SFO1         125.7703637 MHz
NUC1                 13C
P1                  9.80 usec
PLW1         57.00000000 W

======== CHANNEL f2 ========
SFO2         500.1320005 MHz
NUC2                 1H
CPDPRG[2         waltz16
PCPD2               80.00 usec
PLW2         20.00000000 W
PLW12         0.35778001 W
PLW13         0.22898000 W

F2 - Processing parameters
SI                  32768
SF           125.7577897 MHz
WDW                  EM
SSB       0
LB                  1.00 Hz
GB        0
PC                  1.40
```



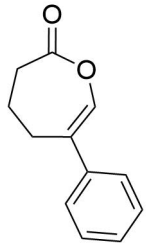


1t

300 MHz, $CDCl_3$

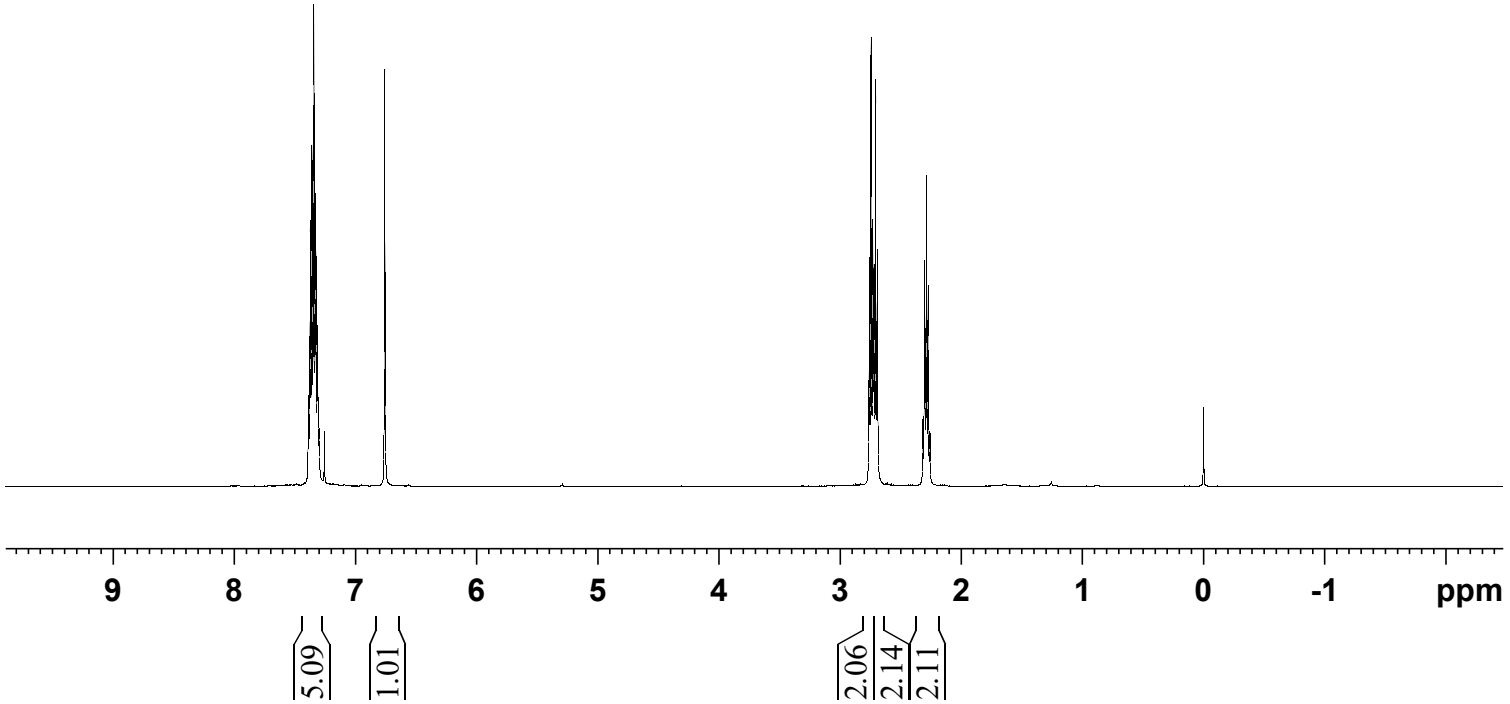



```
Current Data Parameters
NAME      300M-2020shang
EXPNO                106
PROCNO                 1

F2 - Acquisition Parameters
Date_           20200916
Time                9.09
INSTRUM            spect
PROBHD   5 mm DUL 13C-1
PULPROG             zg30
TD                 65536
SOLVENT            CDCl3
NS                    16
DS                     2
SWH             6009.615 Hz
FIDRES          0.091699 Hz
AQ              5.4525952 sec
RG                 142.81
DW                 83.200 usec
DE                   6.50 usec
TE                 298.2 K
D1            1.00000000 sec
D11      0 sec
TD0                    1

======== CHANNEL f1 ========
SFO1         300.1318534 MHz
NUC1                   1H
P1                  8.00 usec
PLW1         18.00000000 W

======== CHANNEL f2 ========
SFO2         300.1318534 MHz
NUC2                  off
CPDPRG[2
PCPD2     0 usec
PLW2      0 W
PLW12     0 W
PLW13     0 W

F2 - Processing parameters
SI                 65536
SF          300.1300060 MHz
WDW                   EM
SSB       0
LB                 0.30 Hz
GB        0
PC                 1.00
```

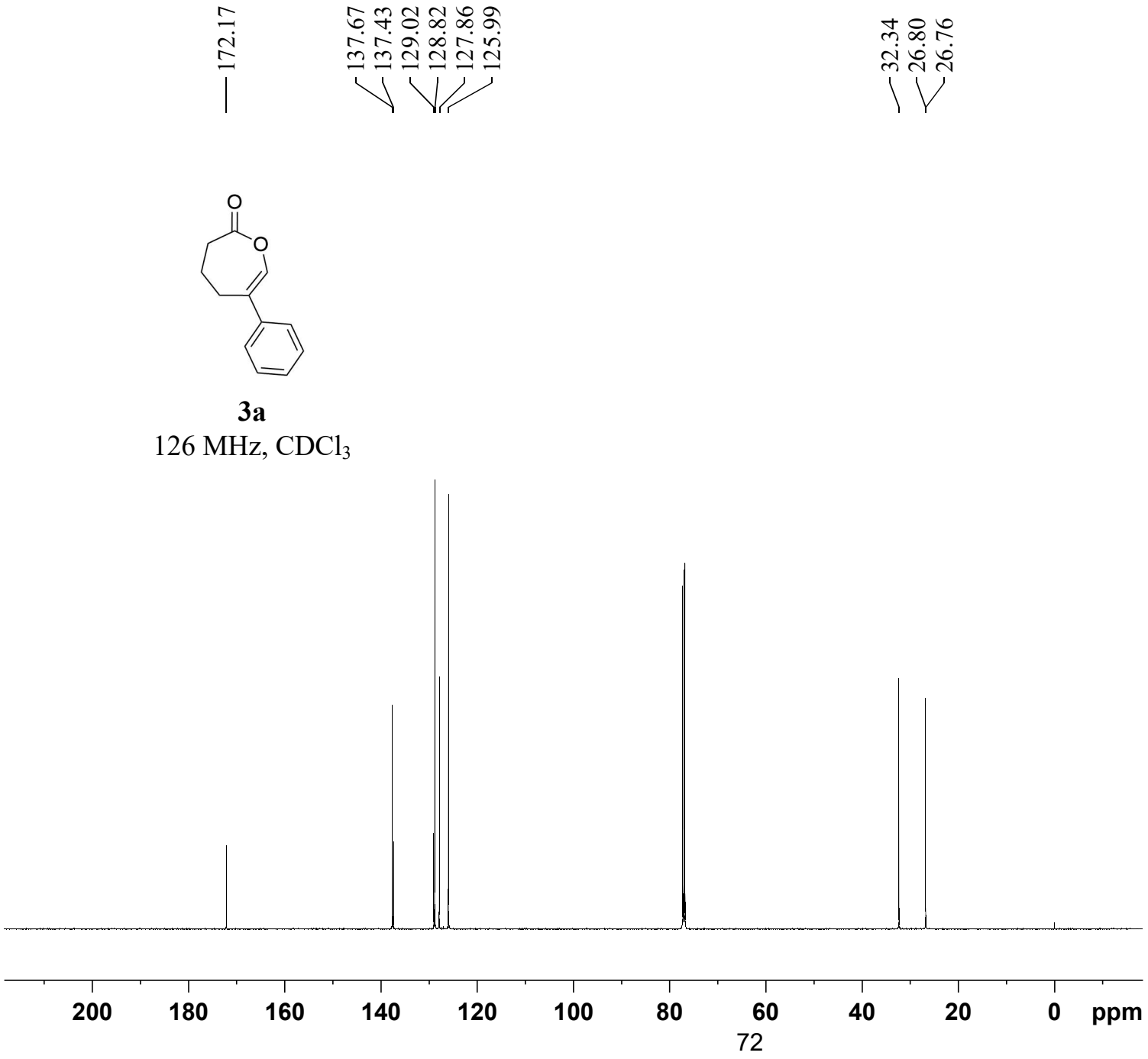


3a

500 MHz, $CDCl_3$



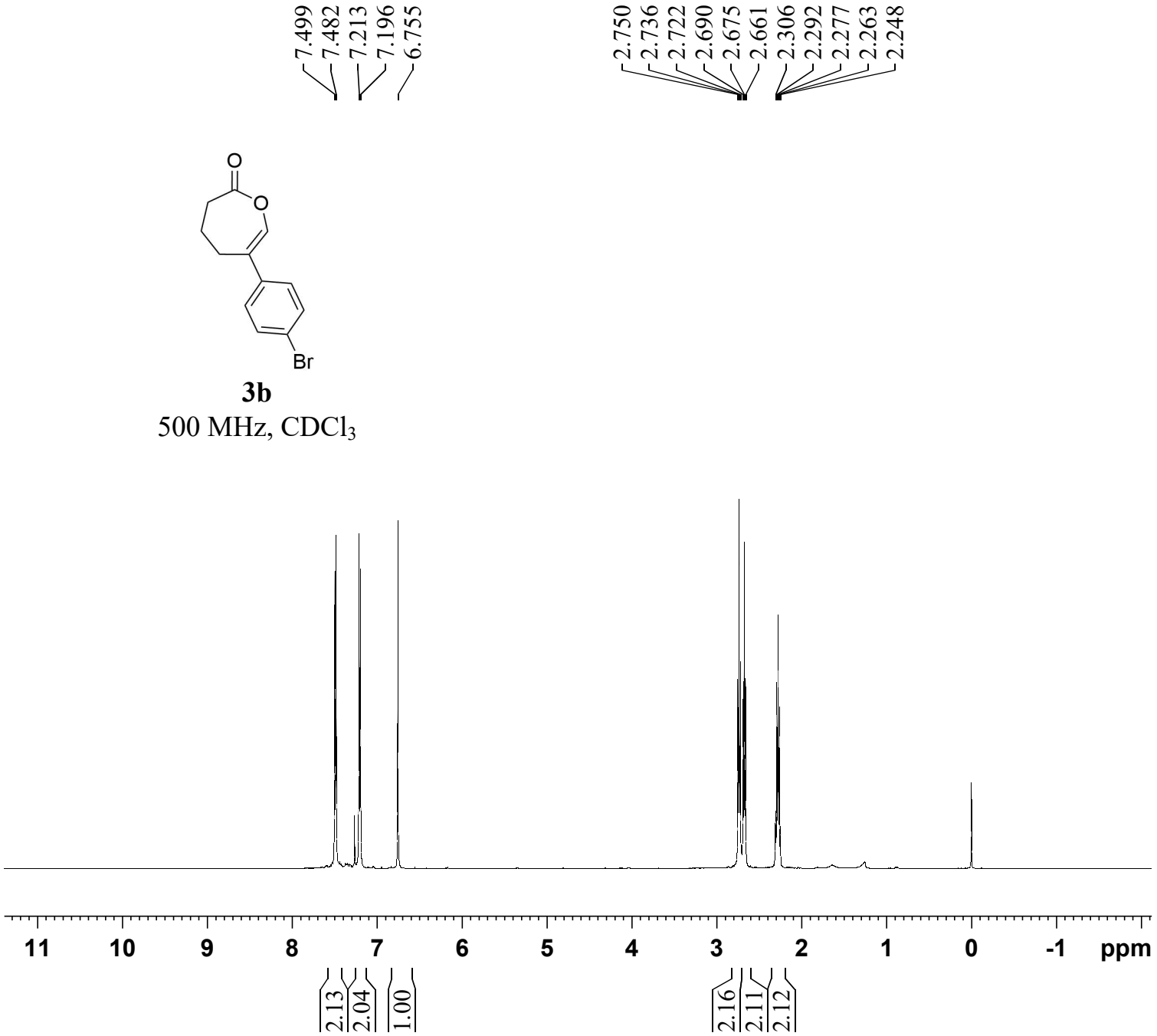

```
Current Data Parameters
NAME        500M-2020xia
EXPNO                 81
PROCNO                 1

F2 - Acquisition Parameters
Date_           20210423
Time              21.46
INSTRUM           spect
PROBHD   5 mm CPPBBO BB
PULPROG             zg30
TD                65536
SOLVENT           CDCl3
NS                   16
DS                    2
SWH           10000.000 Hz
FIDRES         0.152588 Hz
AQ            3.2767999 sec
RG                49.27
DW               50.000 usec
DE                6.50 usec
TE               298.2 K
D1           1.00000000 sec
D11      0 sec
TD0                   1

======== CHANNEL f1 ========
SFO1        500.1330885 MHz
NUC1                  1H
P1                10.59 usec
PLW1        20.00000000 W

======== CHANNEL f2 ========
SFO2        500.1330885 MHz
NUC2                 off
CPDPRG[2
PCPD2     0 usec
PLW2      0 W
PLW12     0 W
PLW13     0 W

F2 - Processing parameters
SI                  65536
SF          500.1300135 MHz
WDW                    EM
SSB       0
LB                 0.30 Hz
GB        0
PC                 1.00
```



3a

126 MHz, $CDCl_3$

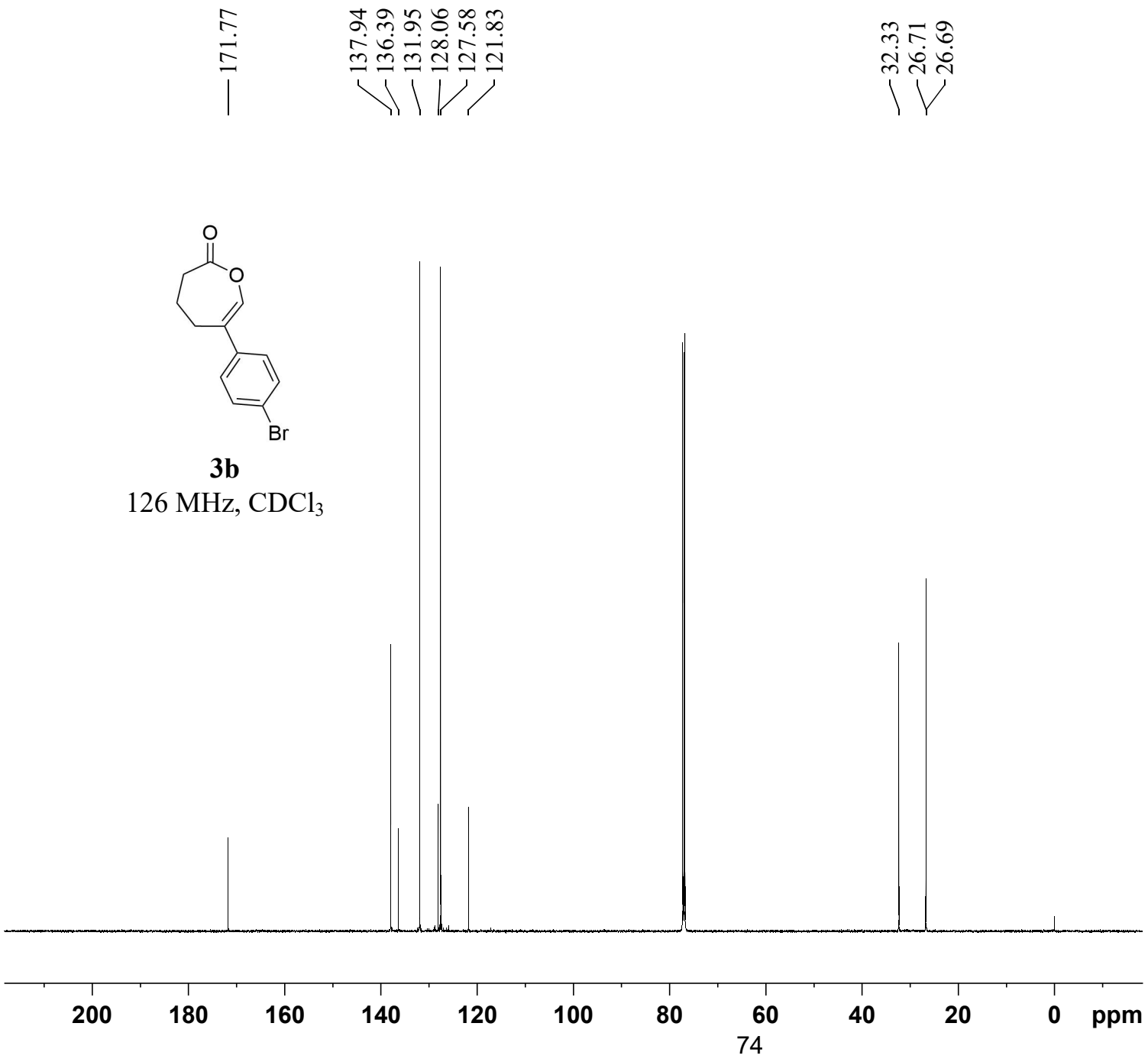
```
Current Data Parameters
NAME        500M-2020xia
EXPNO                 82
PROCNO                 1

F2 - Acquisition Parameters
Date_           20210423
Time              22.40
INSTRUM           spect
PROBHD   5 mm CPPBBO BB
PULPROG          zgpg30
TD                65536
SOLVENT           CDCl3
NS                 1024
DS                    4
SWH           29761.904 Hz
FIDRES         0.454131 Hz
AQ            1.1010048 sec
RG               192.89
DW               16.800 usec
DE               18.00 usec
TE               298.2 K
D1           2.00000000 sec
D11          0.03000000 sec
TD0                   1

======== CHANNEL f1 ========
SFO1        125.7703637 MHz
NUC1                13C
P1                 9.80 usec
PLW1        57.00000000 W

======== CHANNEL f2 ========
SFO2        500.1320005 MHz
NUC2                1H
CPDPRG[2         waltz16
PCPD2             80.00 usec
PLW2        20.00000000 W
PLW12        0.35778001 W
PLW13        0.22898000 W

F2 - Processing parameters
SI                 32768
SF          125.7577912 MHz
WDW                   EM
SSB       0
LB                 1.00 Hz
GB        0
PC                 1.40
```



3b

500 MHz, $CDCl_3$

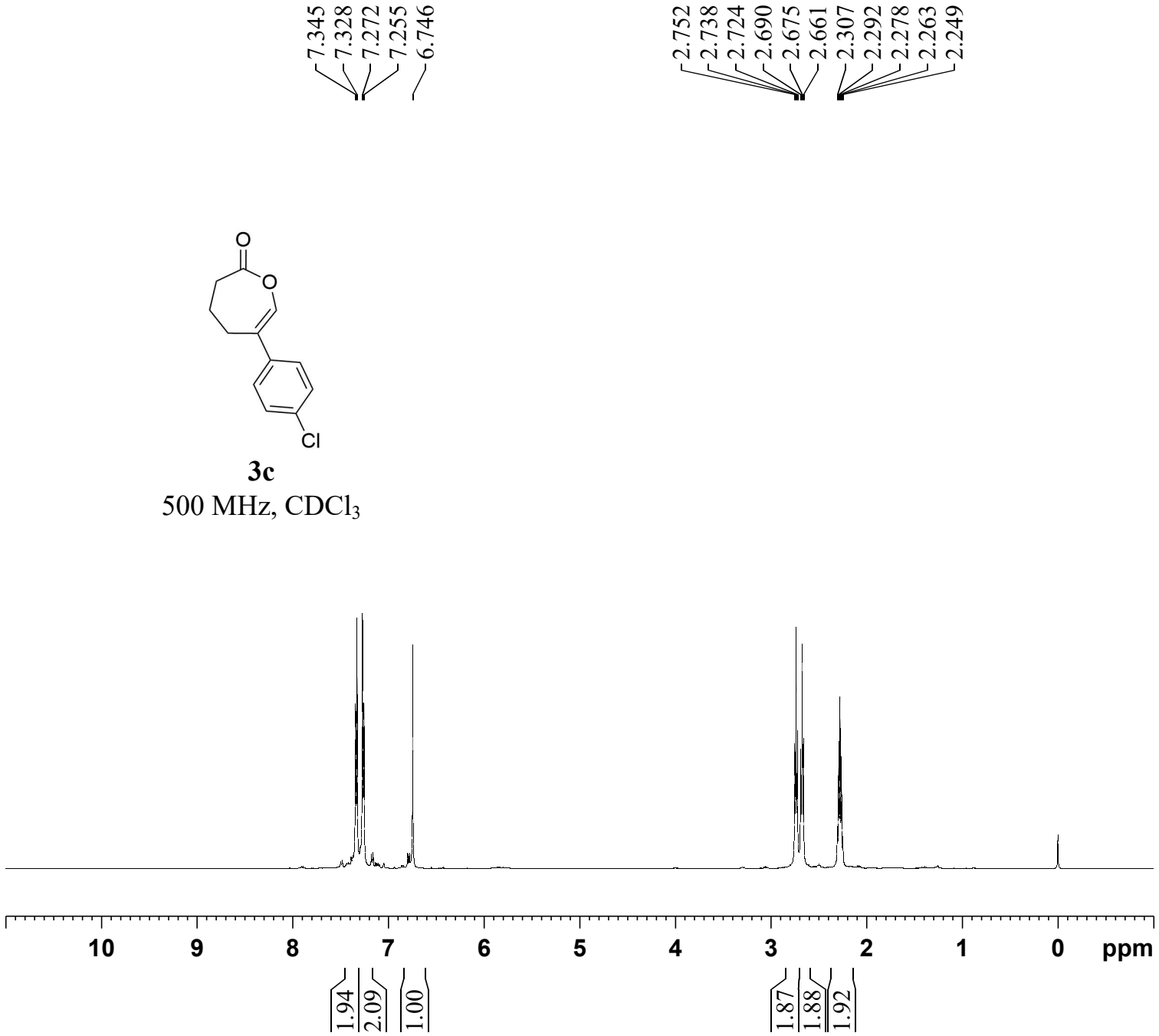
```
Current Data Parameters
NAME       500M-2020xia
EXPNO                93
PROCNO                1

F2 - Acquisition Parameters
Date_          20210424
Time               1.31
INSTRUM           spect
PROBHD   5 mm CPPBBO BB
PULPROG             zg30
TD                65536
SOLVENT           CDCl3
NS                   16
DS                    2
SWH           10000.000 Hz
FIDRES         0.152588 Hz
AQ            3.2767999 sec
RG                55.37
DW               50.000 usec
DE                6.50 usec
TE               298.2 K
D1           1.00000000 sec
D11      0 sec
TD0                   1

======== CHANNEL f1 ========
SFO1        500.1330885 MHz
NUC1                   1H
P1                 10.59 usec
PLW1        20.00000000 W

======== CHANNEL f2 ========
SFO2        500.1330885 MHz
NUC2                  off
CPDPRG[2
PCPD2     0 usec
PLW2      0 W
PLW12     0 W
PLW13     0 W

F2 - Processing parameters
SI                 65536
SF          500.1300097 MHz
WDW                   EM
SSB       0
LB                 0.30 Hz
GB        0
PC                 1.00
```



3b

126 MHz, $CDCl_3$

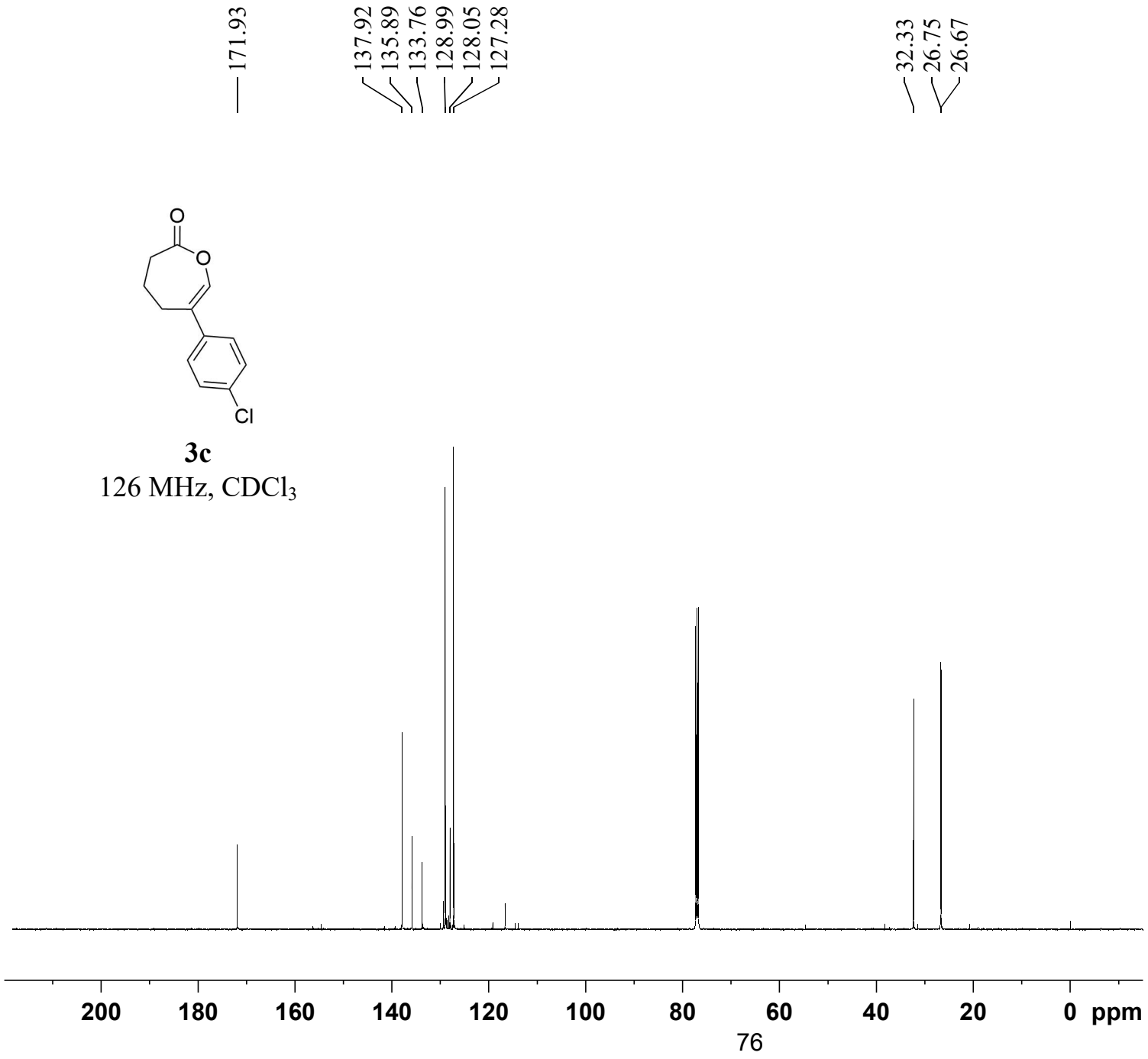
```
Current Data Parameters
NAME        500M-2020xia
EXPNO                 94
PROCNO                 1

F2 - Acquisition Parameters
Date_           20210424
Time                2.03
INSTRUM            spect
PROBHD   5 mm CPPBBO BB
PULPROG          zgpg30
TD                65536
SOLVENT           CDCl3
NS                  600
DS                    4
SWH           29761.904 Hz
FIDRES         0.454131 Hz
AQ             1.1010048 sec
RG                192.89
DW                16.800 usec
DE                18.00 usec
TE                298.2 K
D1            2.00000000 sec
D11           0.03000000 sec
TD0                   1

======== CHANNEL f1 ========
SFO1         125.7703637 MHz
NUC1                 13C
P1                  9.80 usec
PLW1         57.00000000 W

======== CHANNEL f2 ========
SFO2         500.1320005 MHz
NUC2                 1H
CPDPRG[2         waltz16
PCPD2              80.00 usec
PLW2         20.00000000 W
PLW12         0.35778001 W
PLW13         0.22898000 W

F2 - Processing parameters
SI                  32768
SF           125.7577907 MHz
WDW                    EM
SSB       0
LB                  1.00 Hz
GB        0
PC                  1.40
```



3c

500 MHz, $CDCl_3$

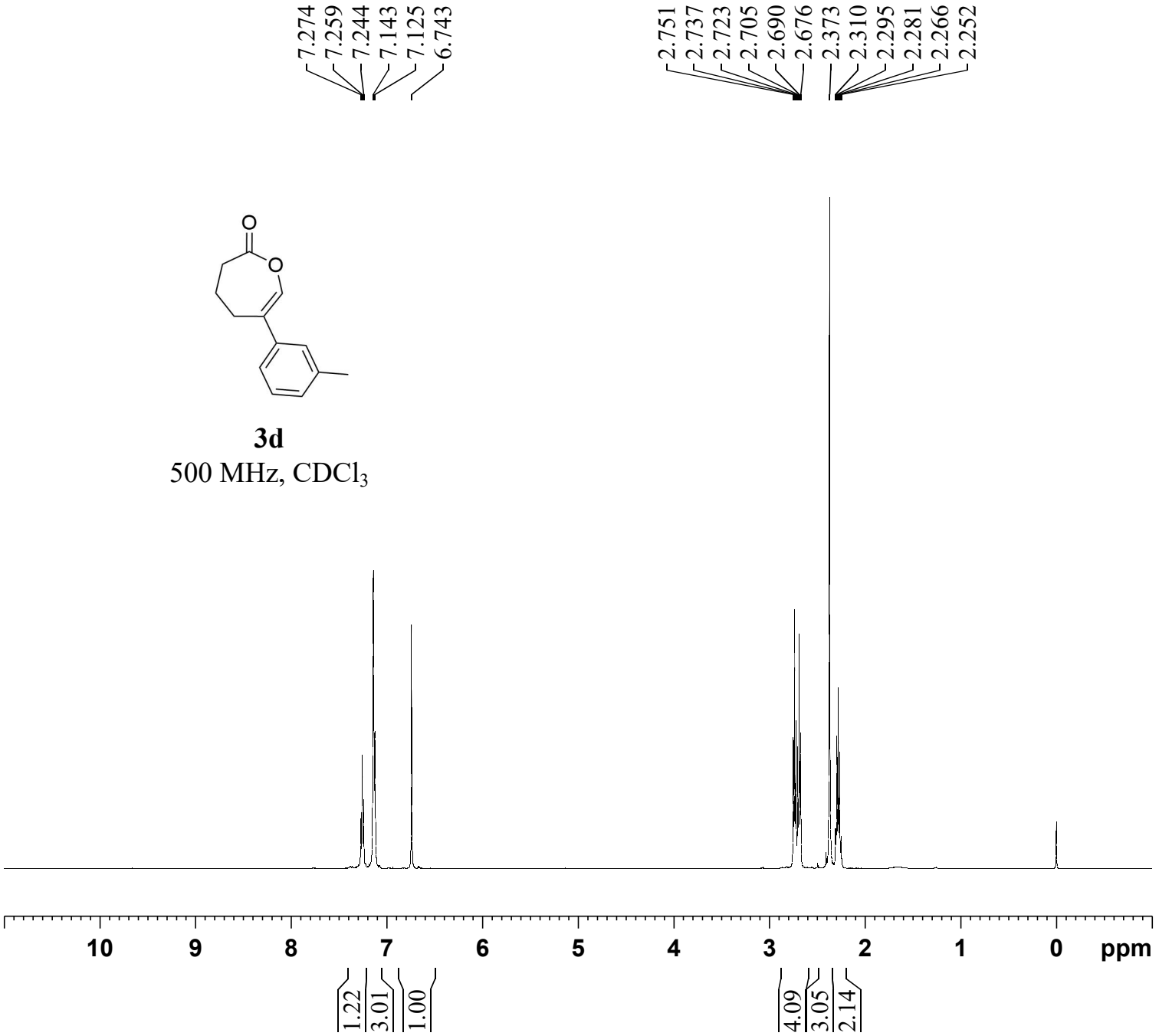
```
Current Data Parameters
NAME        500M-2020xia
EXPNO                 87
PROCNO                 1

F2 - Acquisition Parameters
Date_           20210424
Time                0.19
INSTRUM            spect
PROBHD   5 mm CPPBBO BB
PULPROG             zg30
TD                 65536
SOLVENT            CDCl3
NS                    16
DS                     2
SWH           10000.000 Hz
FIDRES         0.152588 Hz
AQ            3.2767999 sec
RG                 31.72
DW                50.000 usec
DE                 6.50 usec
TE                298.2 K
D1           1.00000000 sec
D11      0 sec
TD0                    1

======== CHANNEL f1 ========
SFO1         500.1330885 MHz
NUC1                   1H
P1                 10.59 usec
PLW1         20.00000000 W

======== CHANNEL f2 ========
SFO2         500.1330885 MHz
NUC2                  off
CPDPRG[2
PCPD2     0 usec
PLW2      0 W
PLW12     0 W
PLW13     0 W

F2 - Processing parameters
SI                 65536
SF           500.1300095 MHz
WDW                   EM
SSB       0
LB                 0.30 Hz
GB        0
PC                 1.00
```



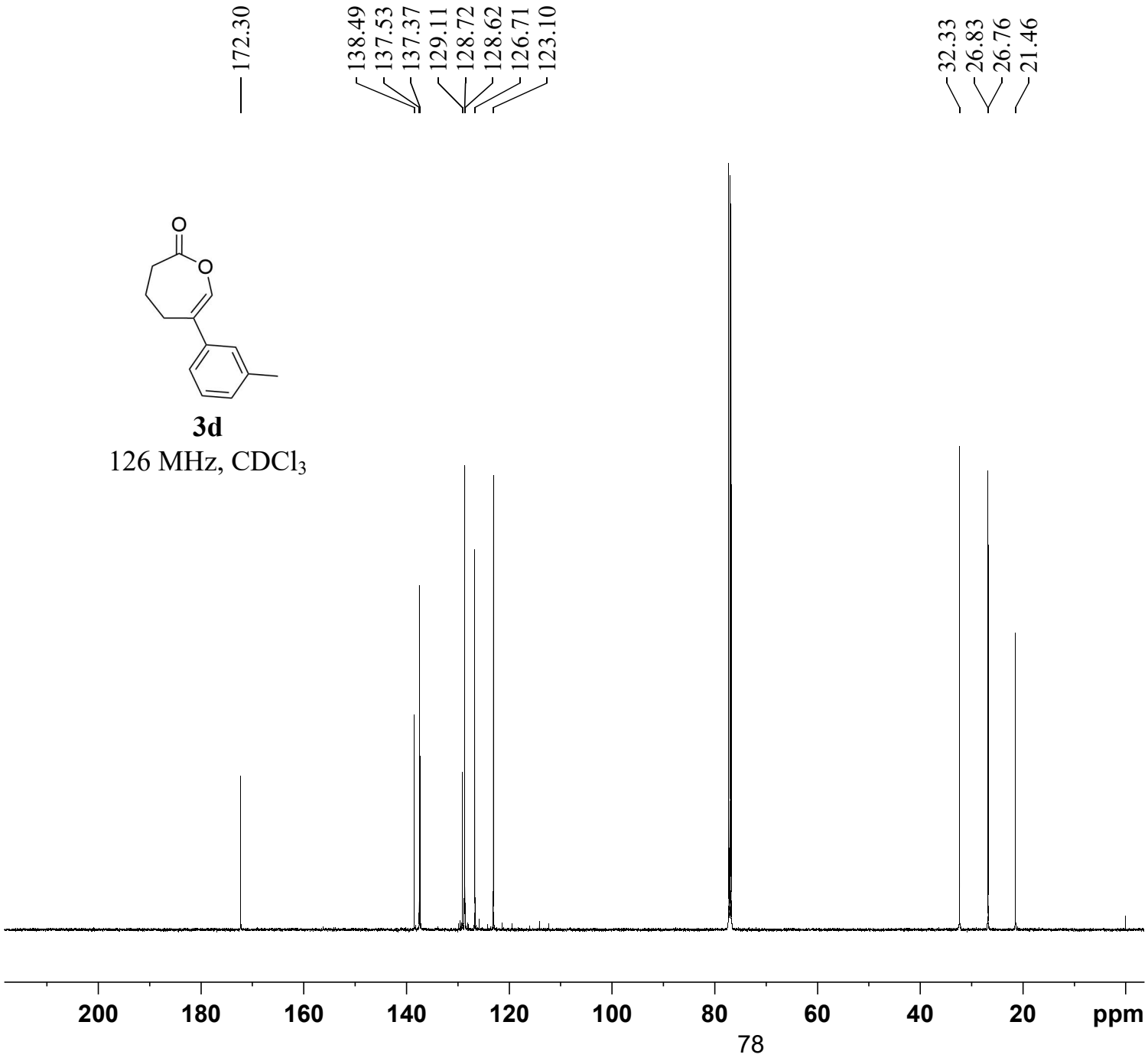
3c

126 MHz, $CDCl_3$

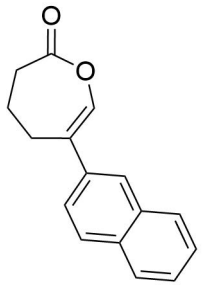
```
Current Data Parameters
NAME        500M-2020xia
EXPNO                 88
PROCNO                 1

F2 - Acquisition Parameters
Date_           20210424
Time                0.51
INSTRUM            spect
PROBHD   5 mm CPPBBO BB
PULPROG           zgpg30
TD                 65536
SOLVENT            CDCl3
NS                   600
DS                     4
SWH            29761.904 Hz
FIDRES          0.454131 Hz
AQ             1.1010048 sec
RG                192.89
DW                16.800 usec
DE                18.00 usec
TE                298.2 K
D1            2.00000000 sec
D11           0.03000000 sec
TD0                    1

======== CHANNEL f1 ========
SFO1         125.7703637 MHz
NUC1                 13C
P1                  9.80 usec
PLW1         57.00000000 W

======== CHANNEL f2 ========
SFO2         500.1320005 MHz
NUC2                 1H
CPDPRG[2         waltz16
PCPD2              80.00 usec
PLW2         20.00000000 W
PLW12         0.35778001 W
PLW13         0.22898000 W

F2 - Processing parameters
SI                  32768
SF           125.7577907 MHz
WDW                    EM
SSB       0
LB                  1.00 Hz
GB        0
PC                  1.40
```




```
Current Data Parameters
NAME        500M-2020xia
EXPNO                 85
PROCNO                 1

F2 - Acquisition Parameters
Date_           20210423
Time              23.43
INSTRUM           spect
PROBHD   5 mm CPPBBO BB
PULPROG             zg30
TD                65536
SOLVENT           CDCl3
NS                   16
DS                    2
SWH           10000.000 Hz
FIDRES         0.152588 Hz
AQ            3.2767999 sec
RG                31.72
DW               50.000 usec
DE                6.50 usec
TE               298.2 K
D1           1.00000000 sec
D11      0 sec
TD0                   1

======== CHANNEL f1 ========
SFO1        500.1330885 MHz
NUC1                   1H
P1                 10.59 usec
PLW1        20.00000000 W

======== CHANNEL f2 ========
SFO2        500.1330885 MHz
NUC2                  off
CPDPRG[2
PCPD2     0 usec
PLW2      0 W
PLW12     0 W
PLW13     0 W

F2 - Processing parameters
SI                 65536
SF          500.1300145 MHz
WDW                   EM
SSB       0
LB                 0.30 Hz
GB        0
PC                 1.00
```

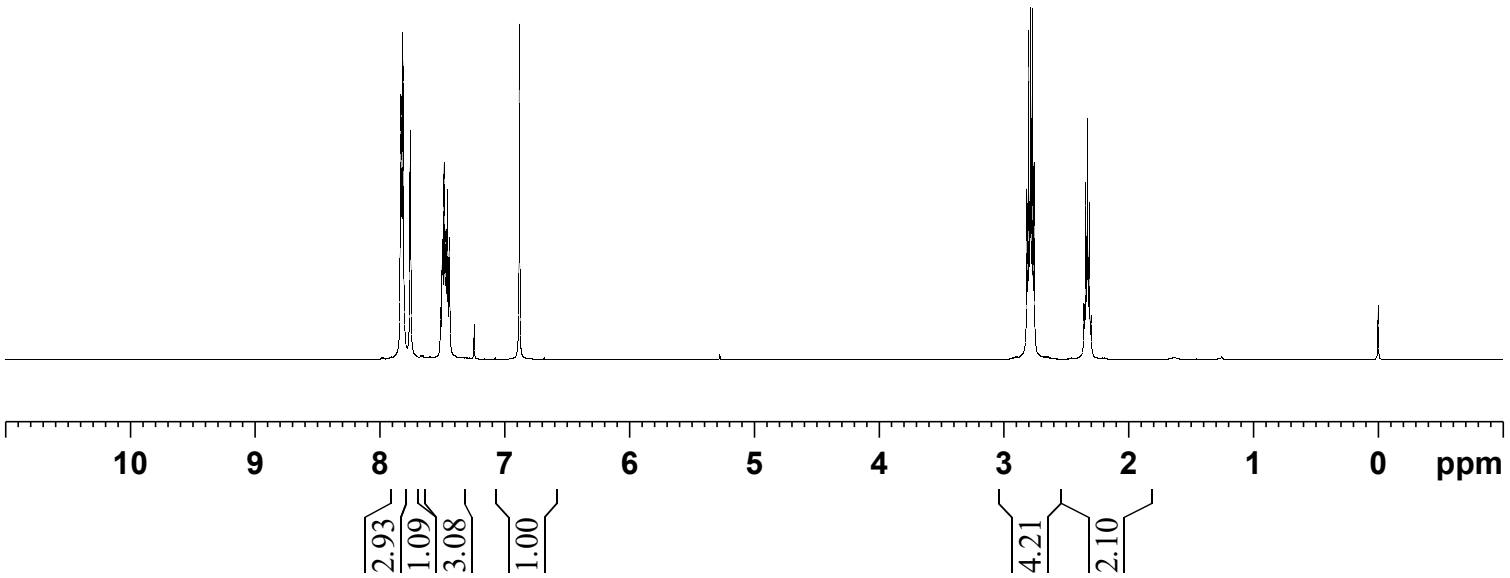



```
Current Data Parameters
NAME        500M-2020xia
EXPNO                 86
PROCNO                 1

F2 - Acquisition Parameters
Date_           20210424
Time                0.15
INSTRUM            spect
PROBHD   5 mm CPPBBO BB
PULPROG          zgpg30
TD                65536
SOLVENT           CDCl3
NS                  600
DS                    4
SWH           29761.904 Hz
FIDRES         0.454131 Hz
AQ             1.1010048 sec
RG                192.89
DW                16.800 usec
DE                18.00 usec
TE                298.2 K
D1            2.00000000 sec
D11           0.03000000 sec
TD0                   1

======== CHANNEL f1 ========
SFO1         125.7703637 MHz
NUC1                 13C
P1                  9.80 usec
PLW1         57.00000000 W

======== CHANNEL f2 ========
SFO2         500.1320005 MHz
NUC2                 1H
CPDPRG[2         waltz16
PCPD2              80.00 usec
PLW2         20.00000000 W
PLW12         0.35778001 W
PLW13         0.22898000 W

F2 - Processing parameters
SI                  32768
SF           125.7577911 MHz
WDW                    EM
SSB       0
LB                  1.00 Hz
GB        0
PC                  1.40
```

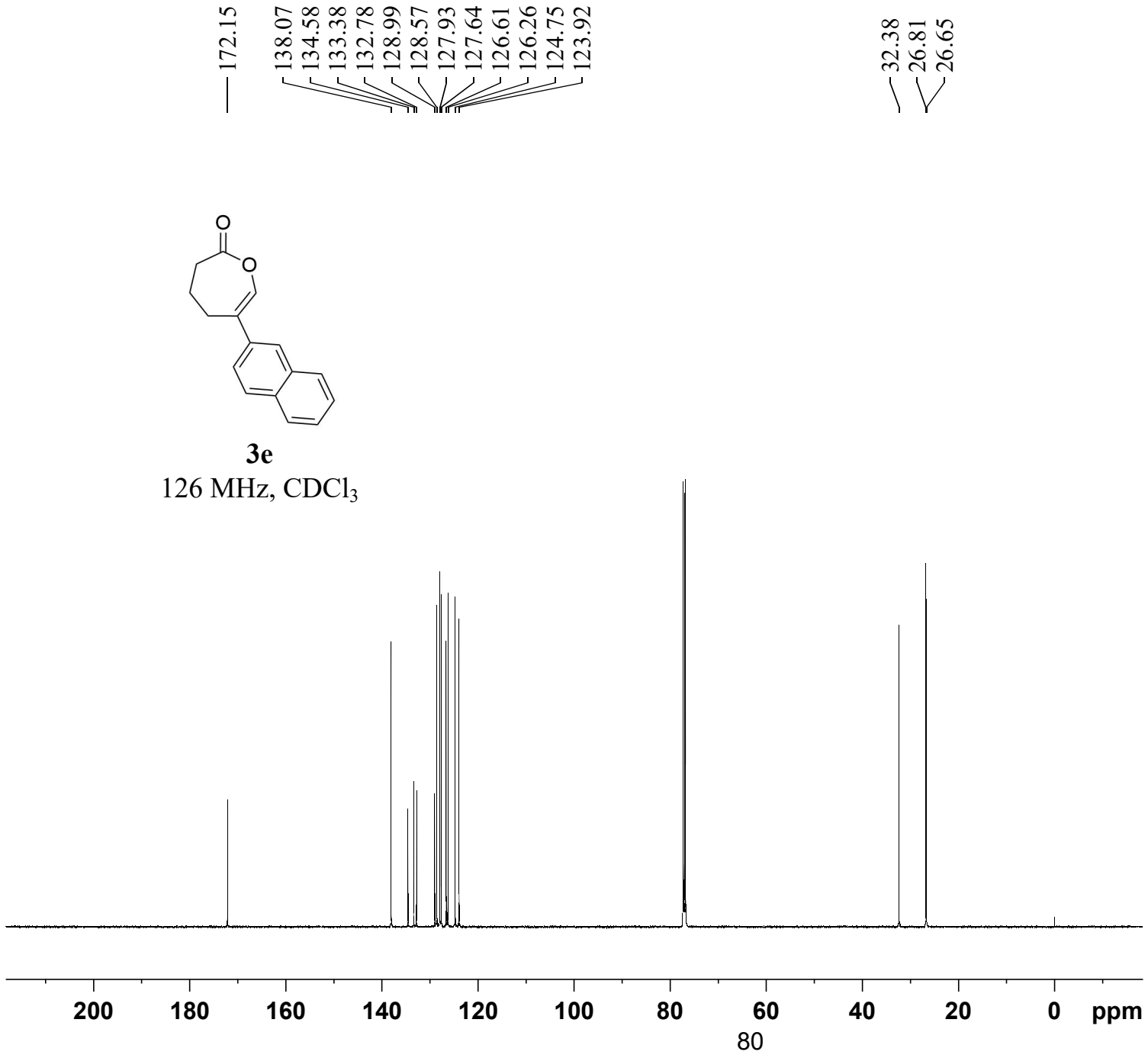


3e

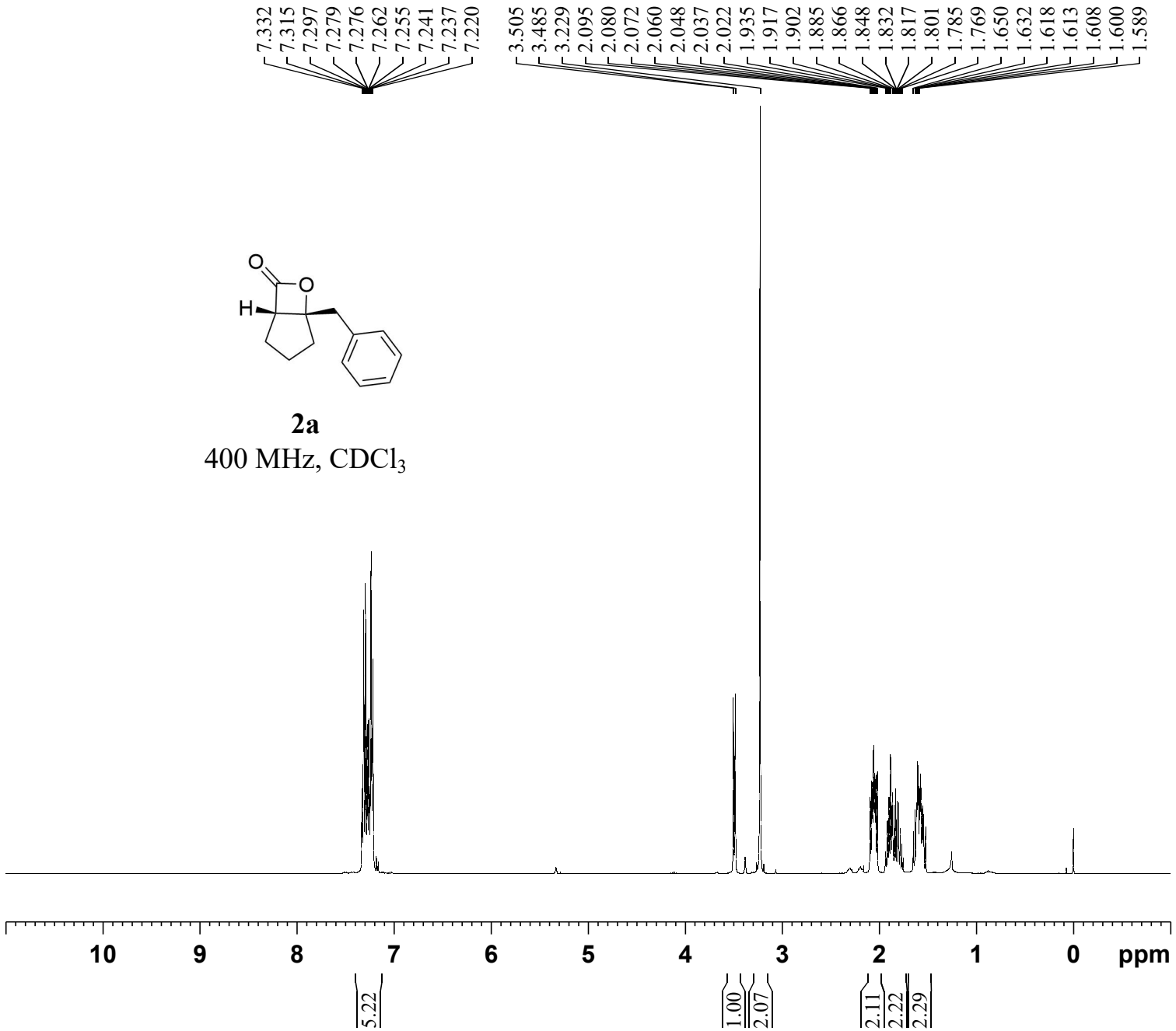
500 MHz, $CDCl_3$




```
Current Data Parameters
NAME        500M-2020xia
EXPNO                 89
PROCNO                 1

F2 - Acquisition Parameters
Date_           20210424
Time                0.55
INSTRUM            spect
PROBHD   5 mm CPPBBO BB
PULPROG             zg30
TD                 65536
SOLVENT            CDCl3
NS                    16
DS                     2
SWH           10000.000 Hz
FIDRES         0.152588 Hz
AQ            3.2767999 sec
RG                 31.72
DW                50.000 usec
DE                 6.50 usec
TE                298.2 K
D1           1.00000000 sec
D11      0 sec
TD0                    1

======== CHANNEL f1 ========
SFO1         500.1330885 MHz
NUC1                   1H
P1                 10.59 usec
PLW1         20.00000000 W

======== CHANNEL f2 ========
SFO2         500.1330885 MHz
NUC2                  off
CPDPRG[2
PCPD2     0 usec
PLW2      0 W
PLW12     0 W
PLW13     0 W

F2 - Processing parameters
SI                 65536
SF           500.1300196 MHz
WDW                   EM
SSB       0
LB                 0.30 Hz
GB        0
PC                 1.00
```



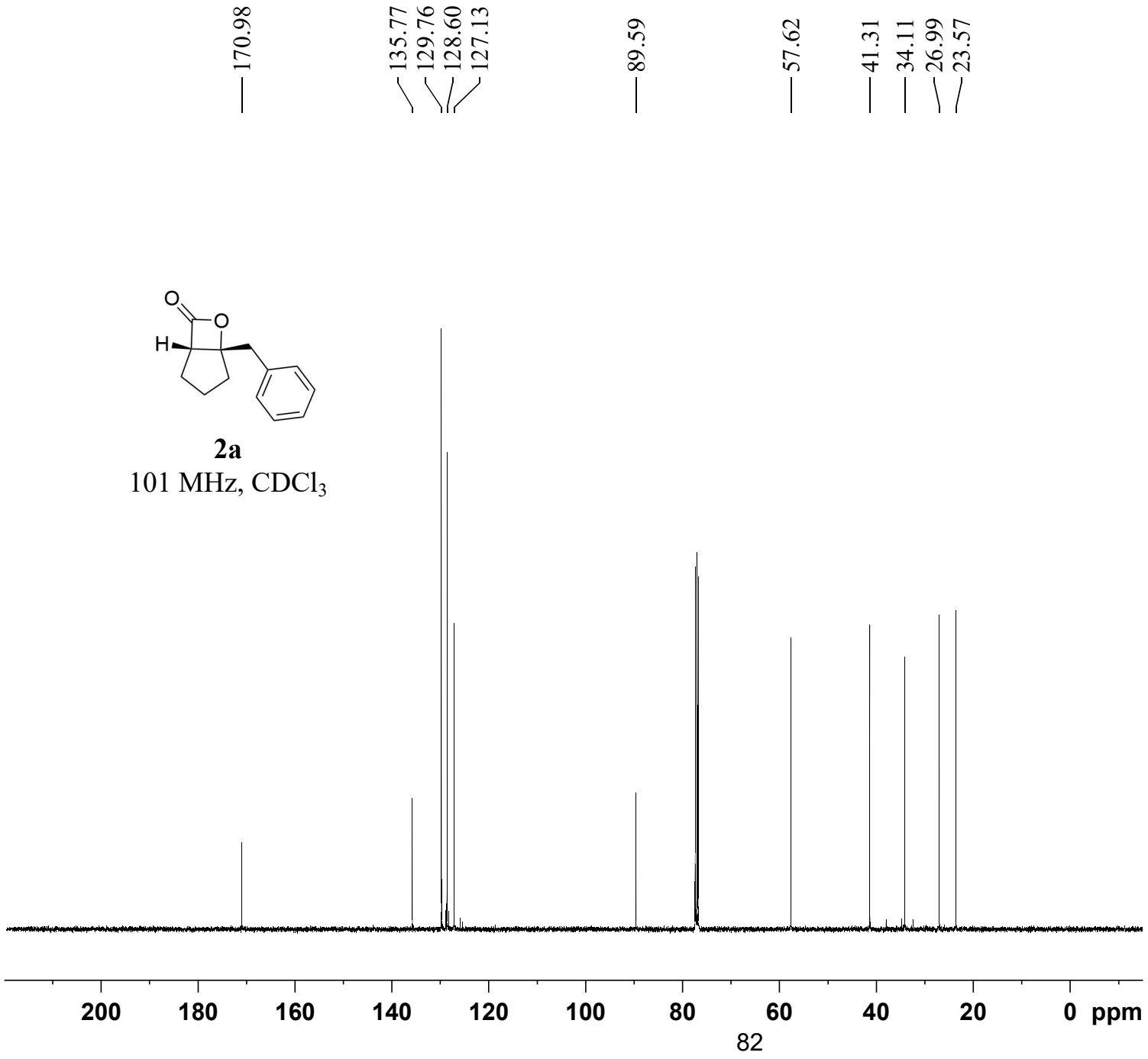

```
Current Data Parameters
NAME        500M-2020xia
EXPNO                 90
PROCNO                 1

F2 - Acquisition Parameters
Date_           20210424
Time               1.27
INSTRUM           spect
PROBHD   5 mm CPPBBO BB
PULPROG          zgpg30
TD                65536
SOLVENT           CDCl3
NS                  600
DS                    4
SWH           29761.904 Hz
FIDRES         0.454131 Hz
AQ            1.1010048 sec
RG               192.89
DW               16.800 usec
DE               18.00 usec
TE               298.2 K
D1           2.00000000 sec
D11          0.03000000 sec
TD0                   1

======== CHANNEL f1 ========
SFO1        125.7703637 MHz
NUC1                13C
P1                 9.80 usec
PLW1        57.00000000 W

======== CHANNEL f2 ========
SFO2        500.1320005 MHz
NUC2                1H
CPDPRG[2         waltz16
PCPD2             80.00 usec
PLW2        20.00000000 W
PLW12        0.35778001 W
PLW13        0.22898000 W

F2 - Processing parameters
SI                 32768
SF          125.7577936 MHz
WDW                   EM
SSB       0
LB                 1.00 Hz
GB        0
PC                 1.40
```



2a

400 MHz, $CDCl_3$

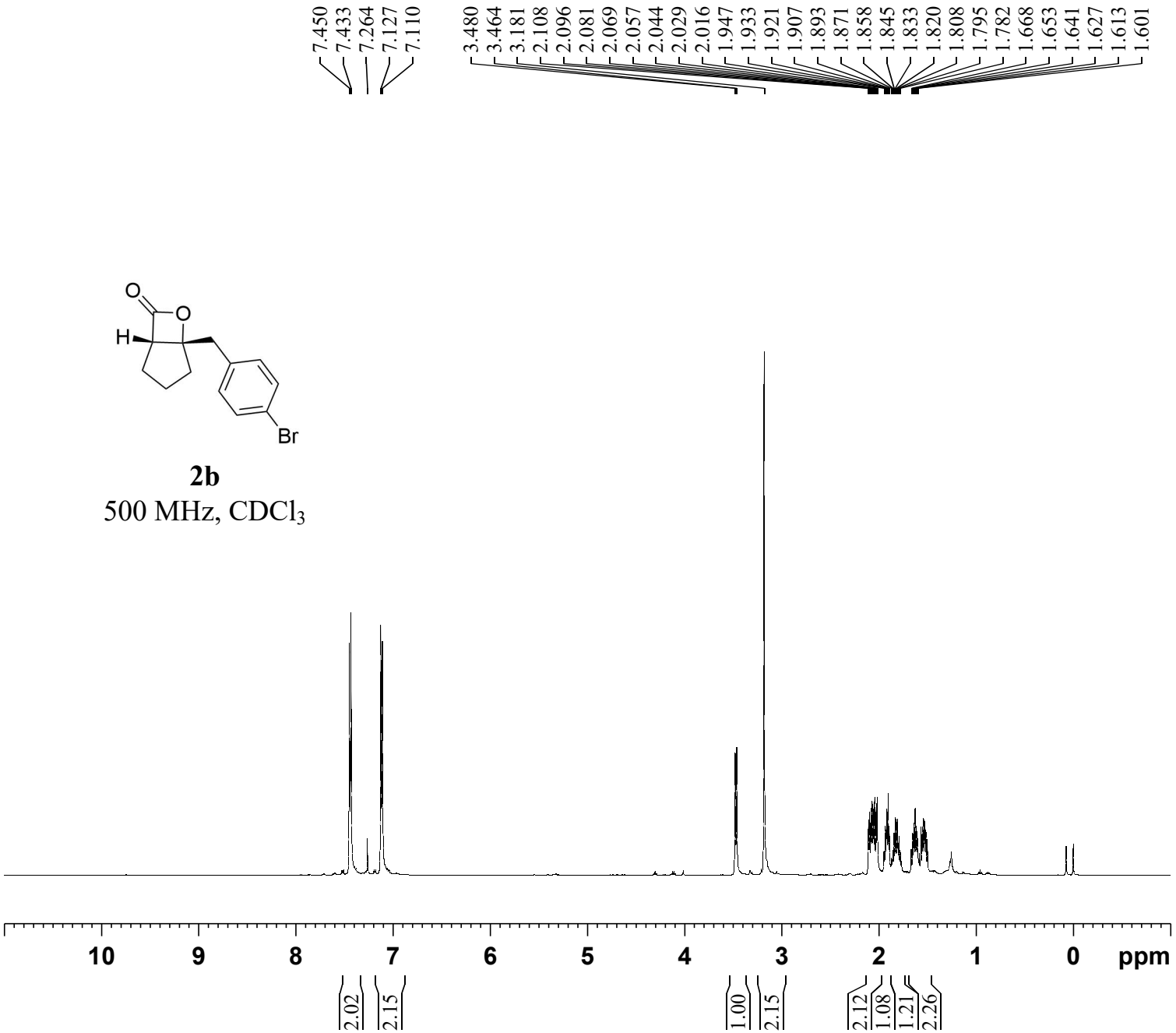
```
Current Data Parameters
NAME                 400xia
EXPNO                    38
PROCNO                    1

F2 - Acquisition Parameters
Date_              20210519
Time                  21.57
INSTRUM               spect
PROBHD    5 mm PABBO BB/
PULPROG                zg30
TD                    32768
SOLVENT               CDCl3
NS                       16
DS                        0
SWH                8012.820 Hz
FIDRES             0.244532 Hz
AQ                2.0447233 sec
RG                     73.9
DW                   62.400 usec
DE                    6.50 usec
TE                   299.5 K
D1              2.00000000 sec
D11       0 sec
TD0                       1

======== CHANNEL f1 ========
SFO1           400.2424716 MHz
NUC1                      1H
P1                    14.30 usec
PLW1           12.00000000 W

======== CHANNEL f2 ========
SFO2           400.2424716 MHz
NUC2                    off
CPDPRG[2
PCPD2     0 usec
PLW2      0 W
PLW12     0 W
PLW13     0 W

F2 - Processing parameters
SI                     65536
SF           400.2400118 MHz
WDW                       EM
SSB       0
LB                    0.30 Hz
GB        0
PC                    1.00
```



2a

101 MHz, $CDCl_3$

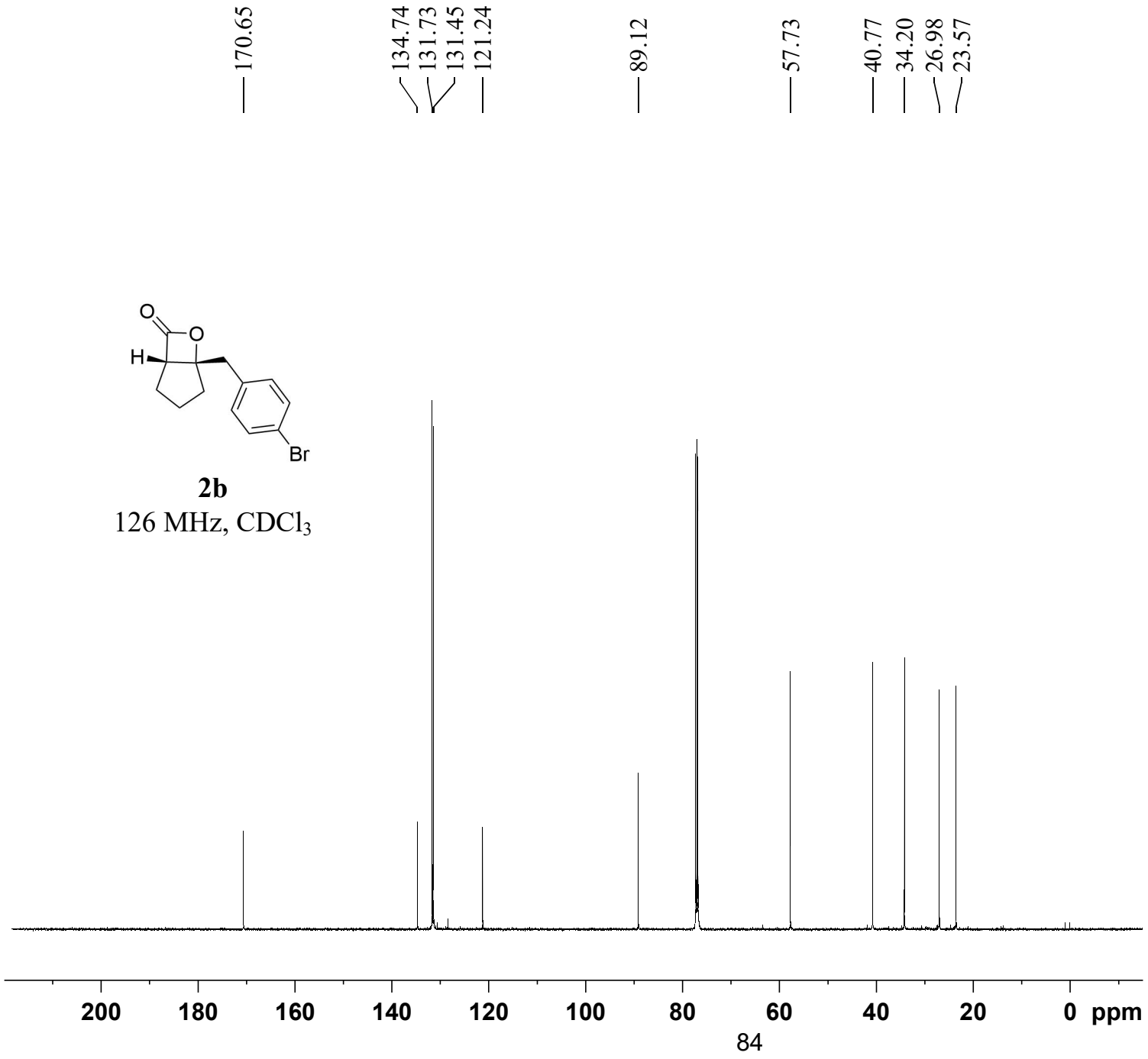
```
Current Data Parameters
NAME                 400xia
EXPNO                    39
PROCNO                    1

F2 - Acquisition Parameters
Date_              20210519
Time                 22.19
INSTRUM              spect
PROBHD   5 mm PABBO BB/
PULPROG             zgpg30
TD                   65536
SOLVENT              CDCl3
NS                     512
DS                       4
SWH              24038.461 Hz
FIDRES            0.366798 Hz
AQ                1.3631488 sec
RG                   206.33
DW                  20.800 usec
DE                   6.50 usec
TE                  300.3 K
D1               2.00000000 sec
D11              0.03000000 sec
TD0                      1

======== CHANNEL f1 ========
SFO1           100.6504916 MHz
NUC1                   13C
P1                  10.00 usec
PLW1           54.00000000 W

======== CHANNEL f2 ========
SFO2           400.2416010 MHz
NUC2                   1H
CPDPRG[2           waltz16
PCPD2               90.00 usec
PLW2           12.00000000 W
PLW12           0.30294999 W
PLW13           0.24539000 W

F2 - Processing parameters
SI                     32768
SF             100.6404280 MHz
WDW                     EM
SSB       0
LB                   1.00 Hz
GB        0
PC                   1.40
```



2b

500 MHz, $CDCl_3$

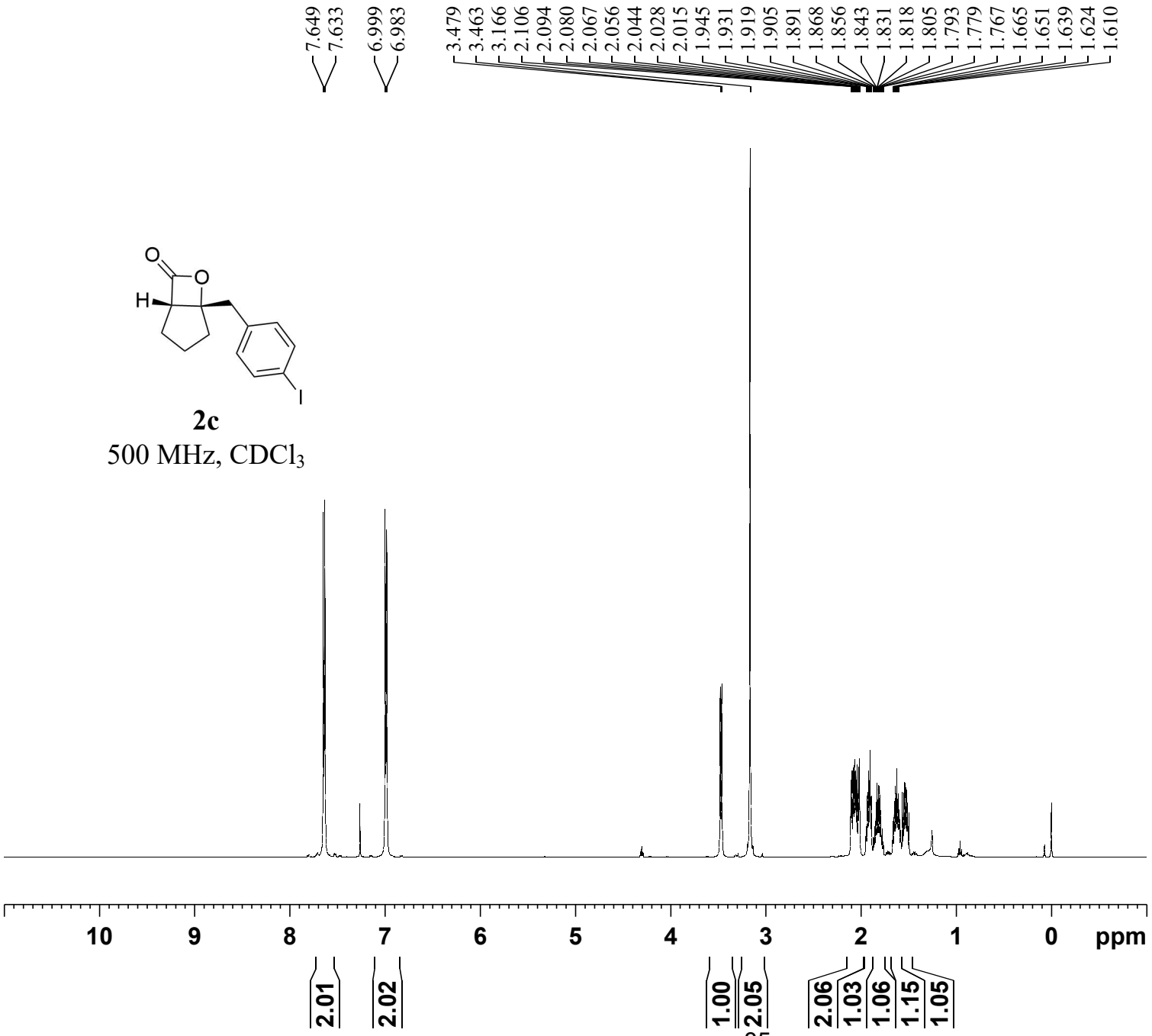
```
Current Data Parameters
NAME        500M-2020xia
EXPNO                 61
PROCNO                 1

F2 - Acquisition Parameters
Date_           20210111
Time               22.11
INSTRUM            spect
PROBHD   5 mm CPPBBO BB
PULPROG             zg30
TD                 65536
SOLVENT            CDCl3
NS                    16
DS                     2
SWH           10000.000 Hz
FIDRES         0.152588 Hz
AQ            3.2767999 sec
RG                 31.72
DW                50.000 usec
DE                 6.50 usec
TE                298.2 K
D1           1.00000000 sec
D11      0 sec
TD0                    1

======== CHANNEL f1 ========
SFO1         500.1330885 MHz
NUC1                   1H
P1                 10.59 usec
PLW1         20.00000000 W

======== CHANNEL f2 ========
SFO2         500.1330885 MHz
NUC2                  off
CPDPRG[2
PCPD2     0 usec
PLW2      0 W
PLW12     0 W
PLW13     0 W

F2 - Processing parameters
SI                 65536
SF           500.1300101 MHz
WDW                   EM
SSB       0
LB                 0.30 Hz
GB        0
PC                 1.00
```



2b

126 MHz, $CDCl_3$

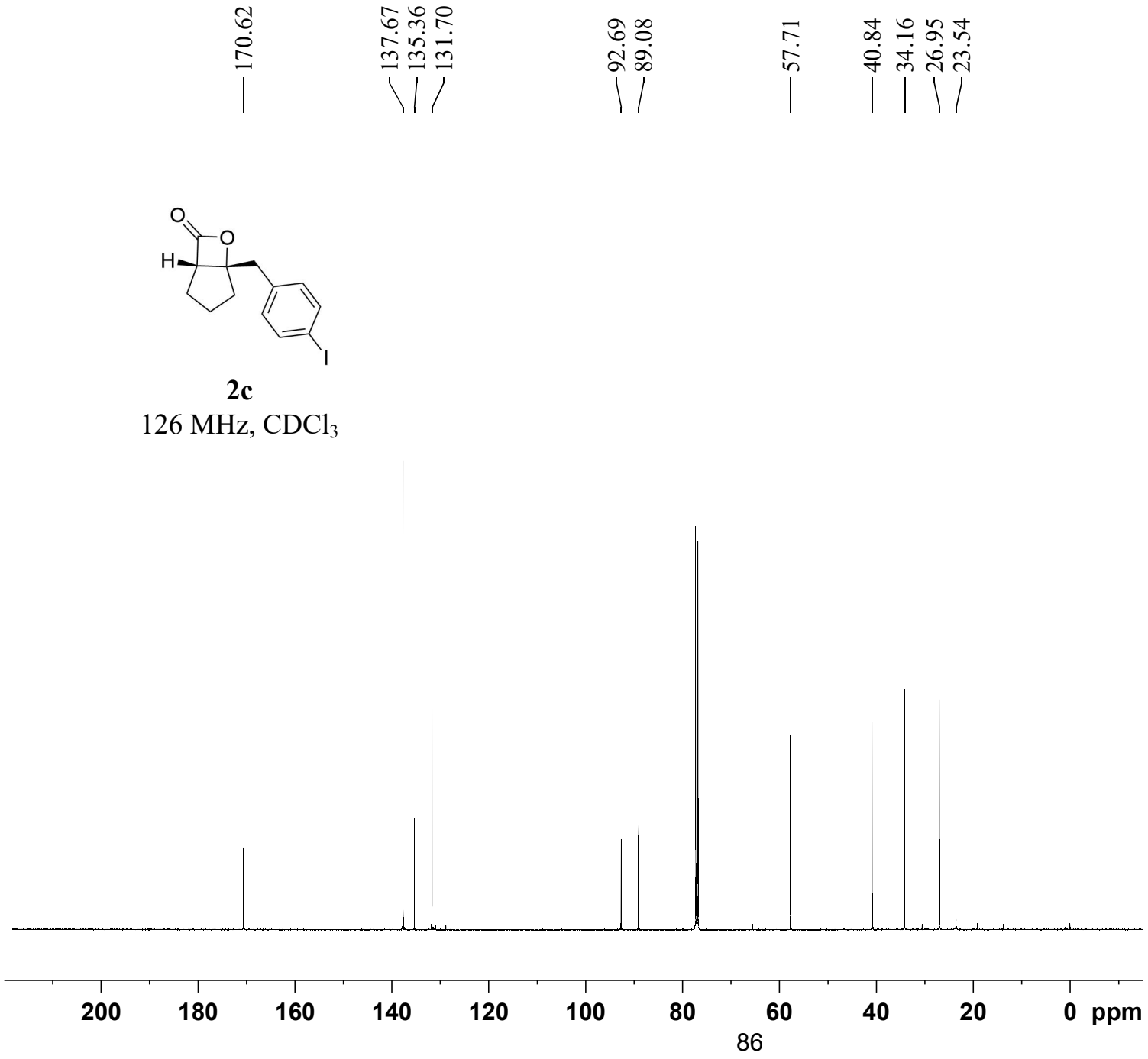
```
Current Data Parameters
NAME        500M-2020xia
EXPNO                 53
PROCNO                 1

F2 - Acquisition Parameters
Date_           20210111
Time               6.25
INSTRUM           spect
PROBHD   5 mm CPPBBO BB
PULPROG          zgpg30
TD                65536
SOLVENT           CDCl3
NS                  500
DS                    4
SWH           29761.904 Hz
FIDRES         0.454131 Hz
AQ             1.1010048 sec
RG                192.89
DW                16.800 usec
DE                18.00 usec
TE                298.2 K
D1            2.00000000 sec
D11           0.03000000 sec
TD0                   1

======== CHANNEL f1 ========
SFO1         125.7703637 MHz
NUC1                 13C
P1                  9.80 usec
PLW1         57.00000000 W

======== CHANNEL f2 ========
SFO2         500.1320005 MHz
NUC2                 1H
CPDPRG[2         waltz16
PCPD2              80.00 usec
PLW2         20.00000000 W
PLW12         0.35778001 W
PLW13         0.22898000 W

F2 - Processing parameters
SI                  32768
SF           125.7577885 MHz
WDW                  EM
SSB       0
LB                 1.00 Hz
GB        0
PC                 1.40
```



2c

500 MHz, $CDCl_3$

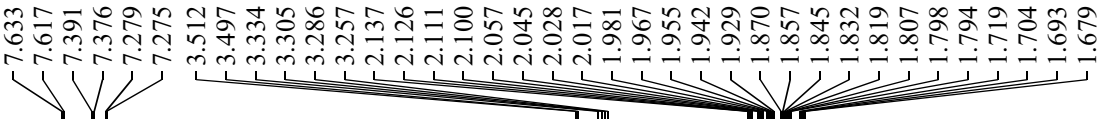
```
Current Data Parameters
NAME        500M-2020xia
EXPNO                 18
PROCNO                 1

F2 - Acquisition Parameters
Date_           20201121
Time                5.02
INSTRUM            spect
PROBHD   5 mm CPPBBO BB
PULPROG             zg30
TD                 65536
SOLVENT            CDCl3
NS                    16
DS                     2
SWH           10000.000 Hz
FIDRES         0.152588 Hz
AQ            3.2767999 sec
RG                 31.72
DW                50.000 usec
DE                 6.50 usec
TE                298.2 K
D1           1.00000000 sec
D11      0 sec
TD0                    1

======== CHANNEL f1 ========
SFO1         500.1330885 MHz
NUC1                   1H
P1                 10.59 usec
PLW1         20.00000000 W

======== CHANNEL f2 ========
SFO2         500.1330885 MHz
NUC2                  off
CPDPRG[2
PCPD2     0 usec
PLW2      0 W
PLW12     0 W
PLW13     0 W

F2 - Processing parameters
SI                 65536
SF            500.1300103 MHz
WDW                   EM
SSB       0
LB                 0.30 Hz
GB        0
PC                 1.00
```

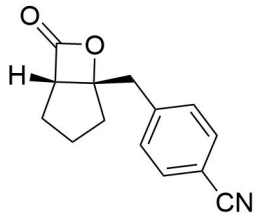


2c

126 MHz, $CDCl_3$

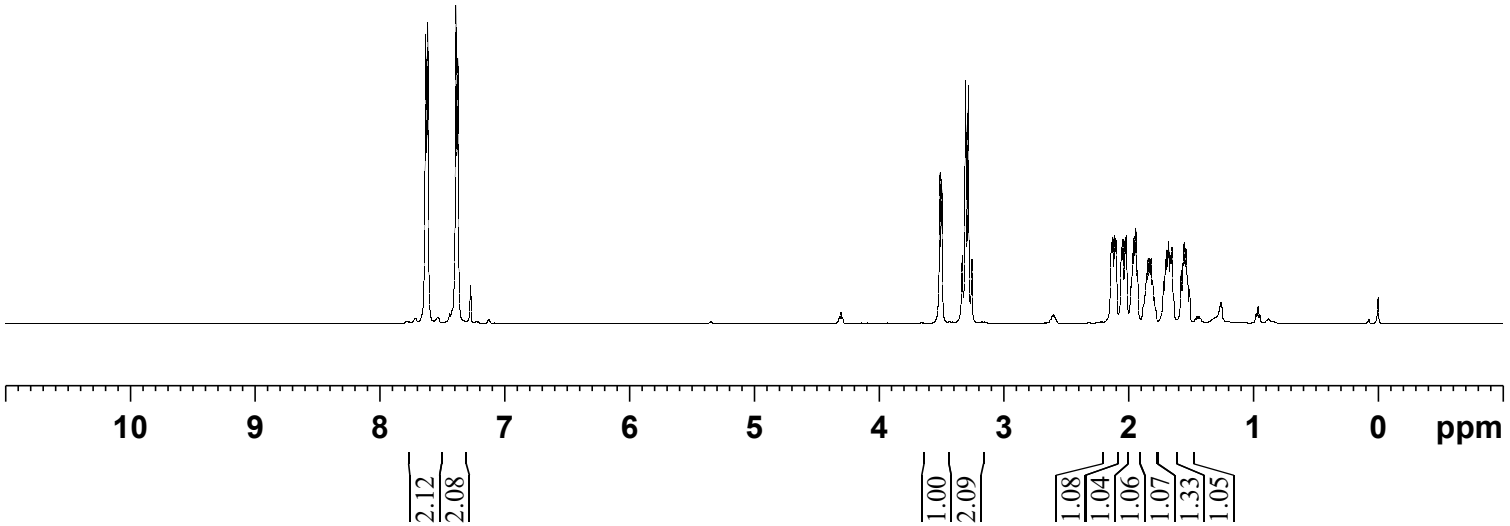
```
Current Data Parameters
NAME        500M-2020xia
EXPNO                 19
PROCNO                 1

F2 - Acquisition Parameters
Date_           20201121
Time                5.57
INSTRUM            spect
PROBHD   5 mm CPPBBO BB
PULPROG           zgpg30
TD                 65536
SOLVENT            CDCl3
NS                  1024
DS                     4
SWH           29761.904 Hz
FIDRES         0.454131 Hz
AQ            1.1010048 sec
RG               192.89
DW               16.800 usec
DE               18.00 usec
TE               298.2 K
D1            2.00000000 sec
D11           0.03000000 sec
TD0                    1

======== CHANNEL f1 ========
SFO1         125.7703637 MHz
NUC1                 13C
P1                 9.80 usec
PLW1         57.00000000 W

======== CHANNEL f2 ========
SFO2         500.1320005 MHz
NUC2                 1H
CPDPRG[2         waltz16
PCPD2              80.00 usec
PLW2         20.00000000 W
PLW12         0.35778001 W
PLW13         0.22898000 W

F2 - Processing parameters
SI                  32768
SF           125.7577926 MHz
WDW                    EM
SSB       0
LB                 1.00 Hz
GB        0
PC                 1.40
```



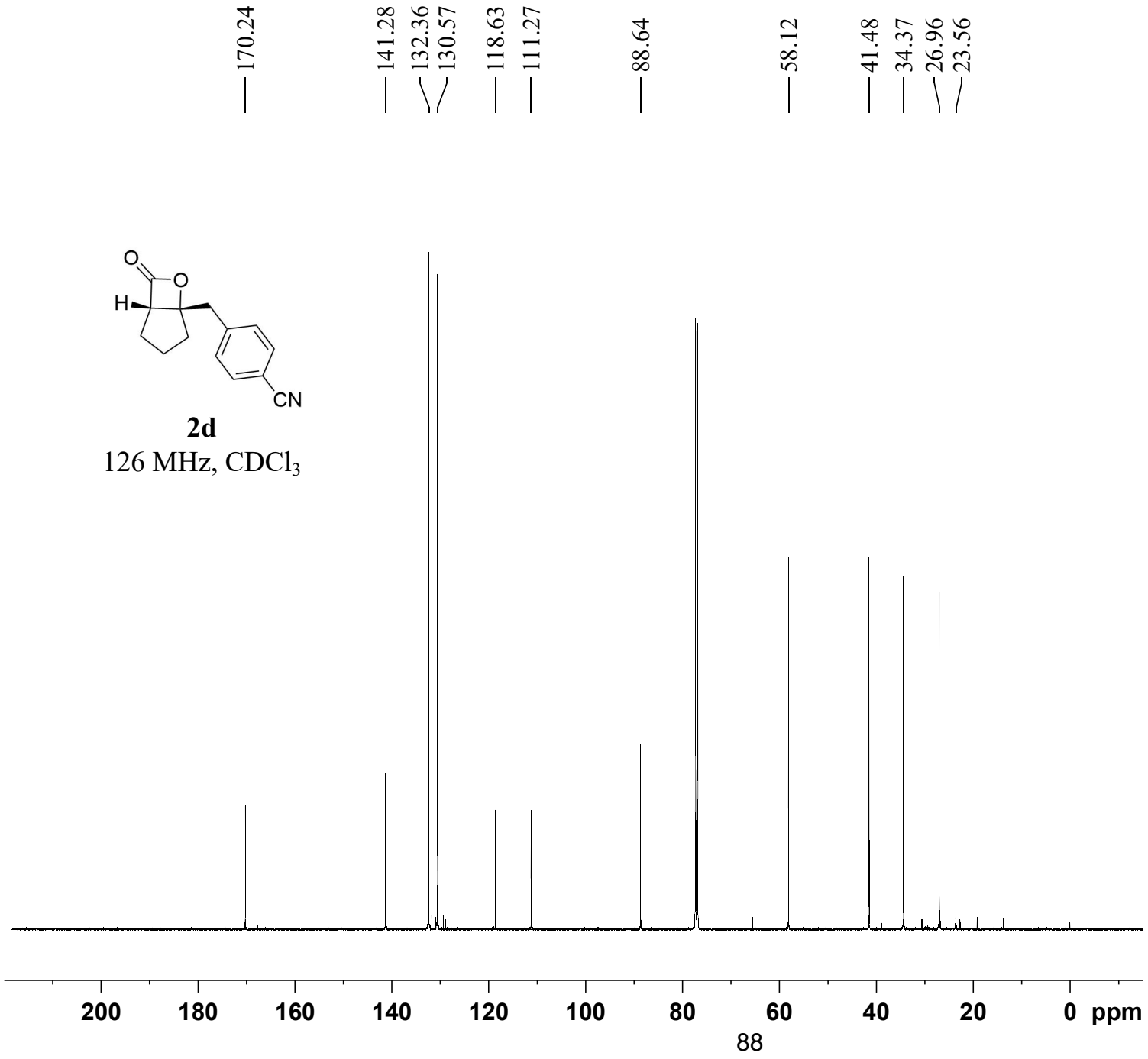


2d

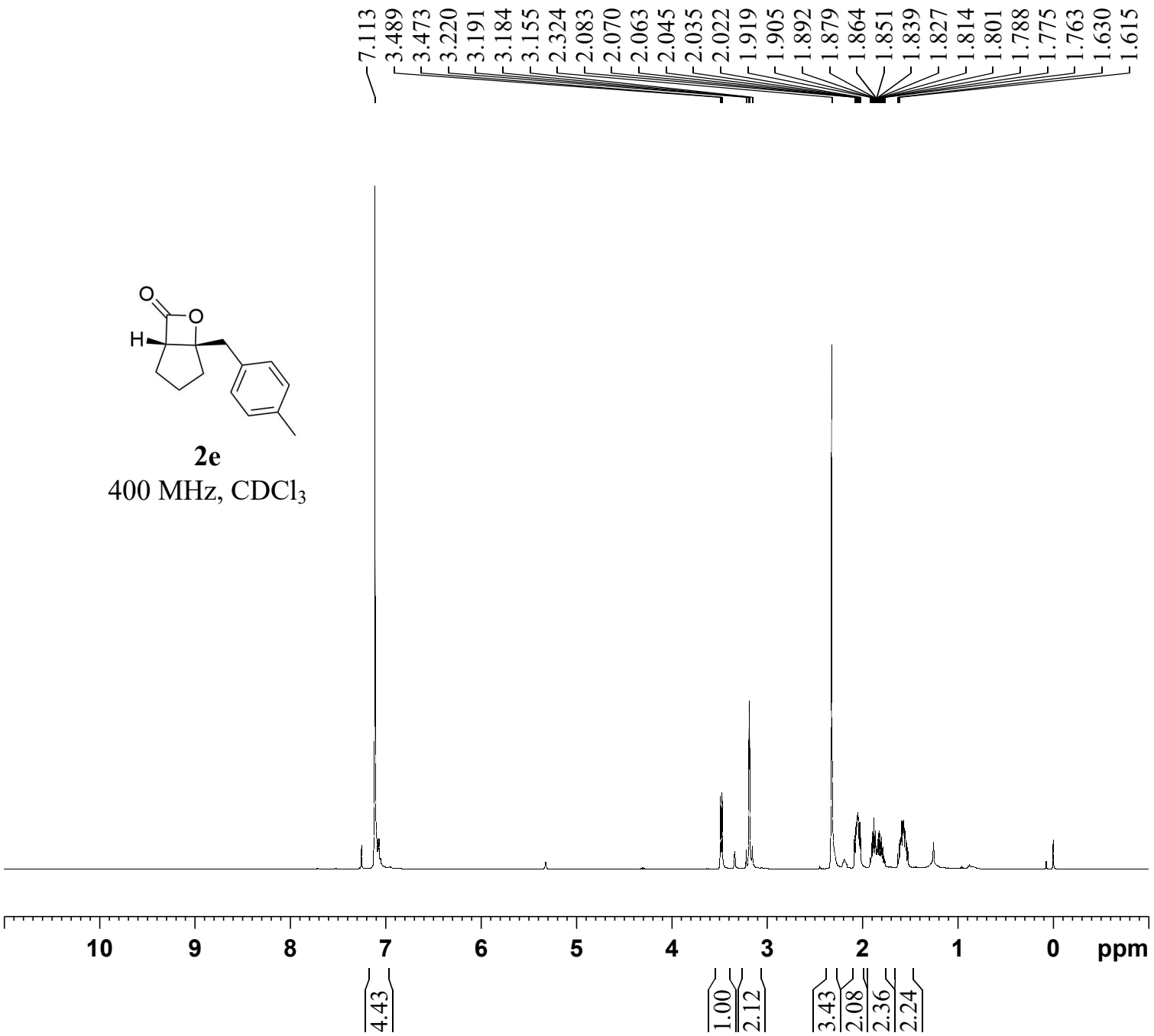
500 MHz, $CDCl_3$




```
Current Data Parameters
NAME        500M-2020xia
EXPNO                  1
PROCNO                 1

F2 - Acquisition Parameters
Date_           20201118
Time                4.01
INSTRUM            spect
PROBHD   5 mm CPPBBO BB
PULPROG             zg30
TD                 65536
SOLVENT            CDCl3
NS                    16
DS                     2
SWH           10000.000 Hz
FIDRES         0.152588 Hz
AQ            3.2767999 sec
RG                 31.72
DW                50.000 usec
DE                 6.50 usec
TE                298.2 K
D1           1.00000000 sec
D11      0 sec
TD0                    1

======== CHANNEL f1 ========
SFO1         500.1330885 MHz
NUC1                   1H
P1                 10.59 usec
PLW1         20.00000000 W

======== CHANNEL f2 ========
SFO2         500.1330885 MHz
NUC2                  off
CPDPRG[2
PCPD2     0 usec
PLW2      0 W
PLW12     0 W
PLW13     0 W

F2 - Processing parameters
SI                 65536
SF         500.1300041 MHz
WDW                   EM
SSB       0
LB                 0.30 Hz
GB        0
PC                 1.00
```



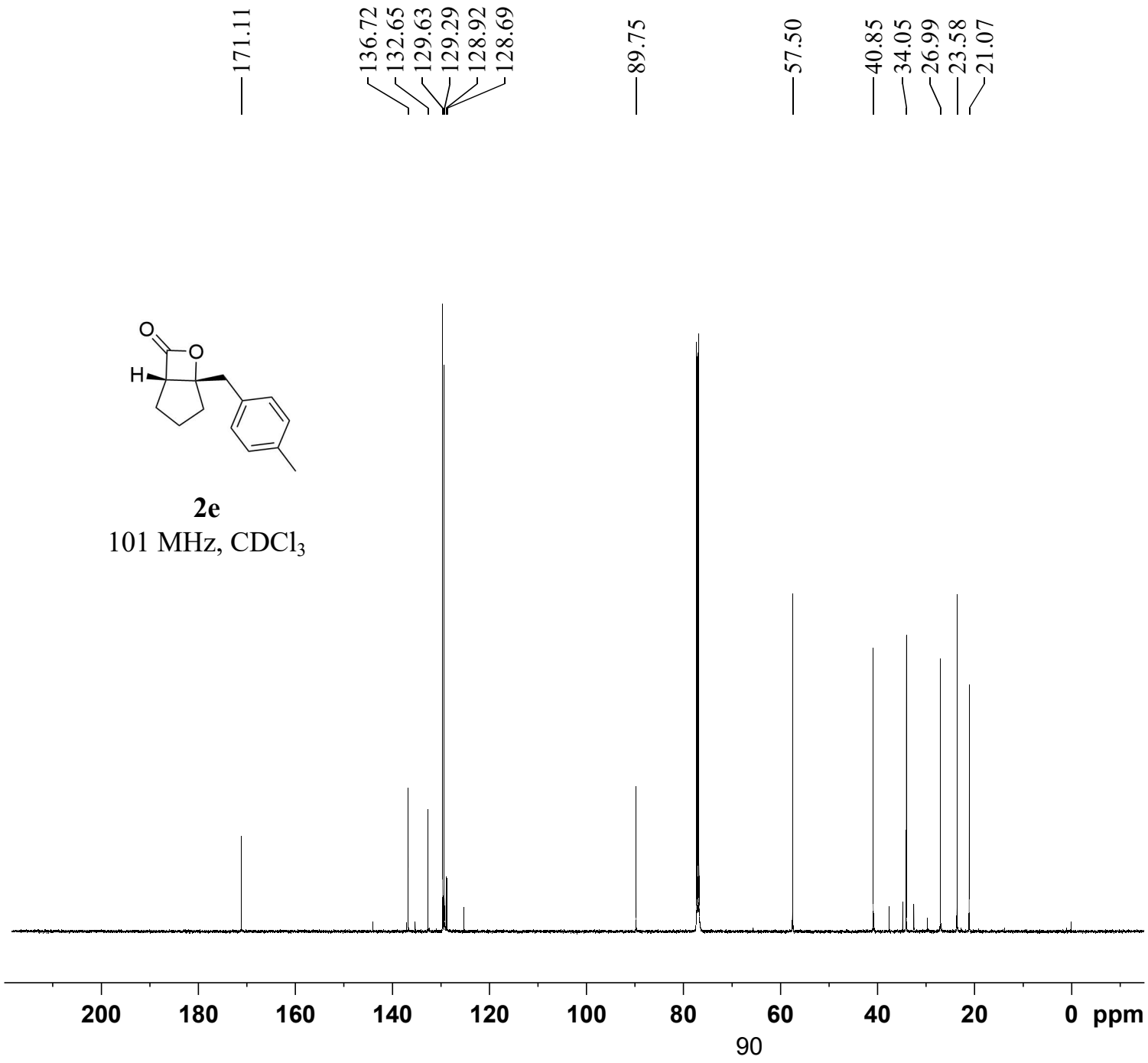

```
Current Data Parameters
NAME        500M-2020xia
EXPNO                  2
PROCNO                 1

F2 - Acquisition Parameters
Date_           20201118
Time               4.28
INSTRUM           spect
PROBHD   5 mm CPPBBO BB
PULPROG          zgpg30
TD                65536
SOLVENT           CDCl3
NS                  500
DS                    4
SWH           29761.904 Hz
FIDRES         0.454131 Hz
AQ            1.1010048 sec
RG               192.89
DW               16.800 usec
DE               18.00 usec
TE               298.2 K
D1           2.00000000 sec
D11          0.03000000 sec
TD0                   1

======== CHANNEL f1 ========
SFO1        125.7703637 MHz
NUC1                13C
P1                 9.80 usec
PLW1        57.00000000 W

======== CHANNEL f2 ========
SFO2        500.1320005 MHz
NUC2                1H
CPDPRG[2         waltz16
PCPD2             80.00 usec
PLW2        20.00000000 W
PLW12        0.35778001 W
PLW13        0.22898000 W

F2 - Processing parameters
SI                 32768
SF          125.7577885 MHz
WDW                   EM
SSB       0
LB                 1.00 Hz
GB        0
PC                 1.40
```



2e

400 MHz, $CDCl_3$

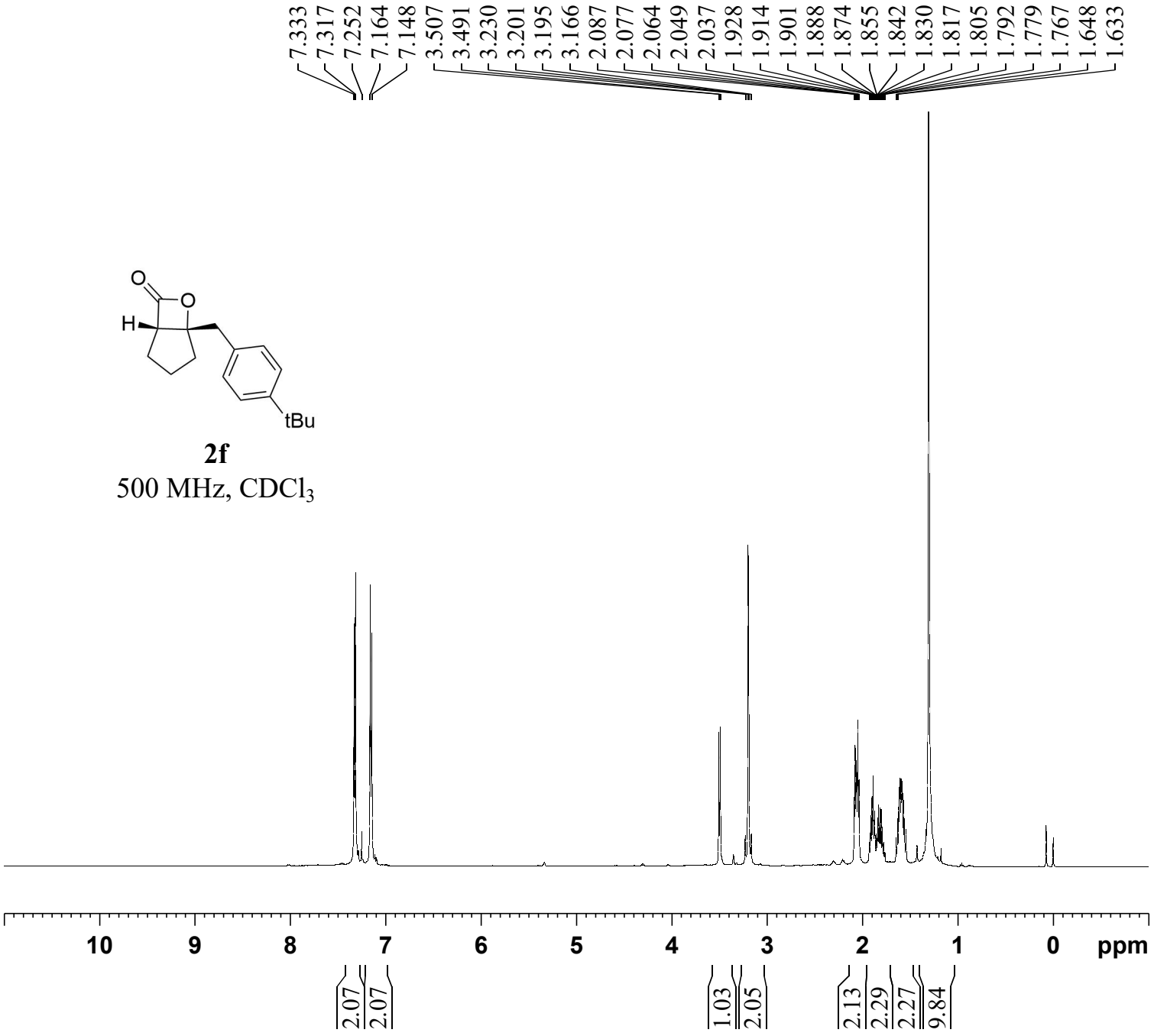
```
Current Data Parameters
NAME        500M-2020xia
EXPNO                 56
PROCNO                 1

F2 - Acquisition Parameters
Date_           20210111
Time              21.49
INSTRUM           spect
PROBHD   5 mm CPPBBO BB
PULPROG             zg30
TD                65536
SOLVENT           CDCl3
NS                   16
DS                    2
SWH           10000.000 Hz
FIDRES         0.152588 Hz
AQ            3.2767999 sec
RG                31.72
DW               50.000 usec
DE                6.50 usec
TE               298.2 K
D1            1.00000000 sec
D11      0 sec
TD0                   1

======== CHANNEL f1 ========
SFO1         500.1330885 MHz
NUC1                 1H
P1                10.59 usec
PLW1         20.00000000 W

======== CHANNEL f2 ========
SFO2         500.1330885 MHz
NUC2                off
CPDPRG[2
PCPD2     0 usec
PLW2      0 W
PLW12     0 W
PLW13     0 W

F2 - Processing parameters
SI                  65536
SF           500.1300154 MHz
WDW                    EM
SSB       0
LB                  0.30 Hz
GB        0
PC                  1.00
```



2e

101 MHz, $CDCl_3$

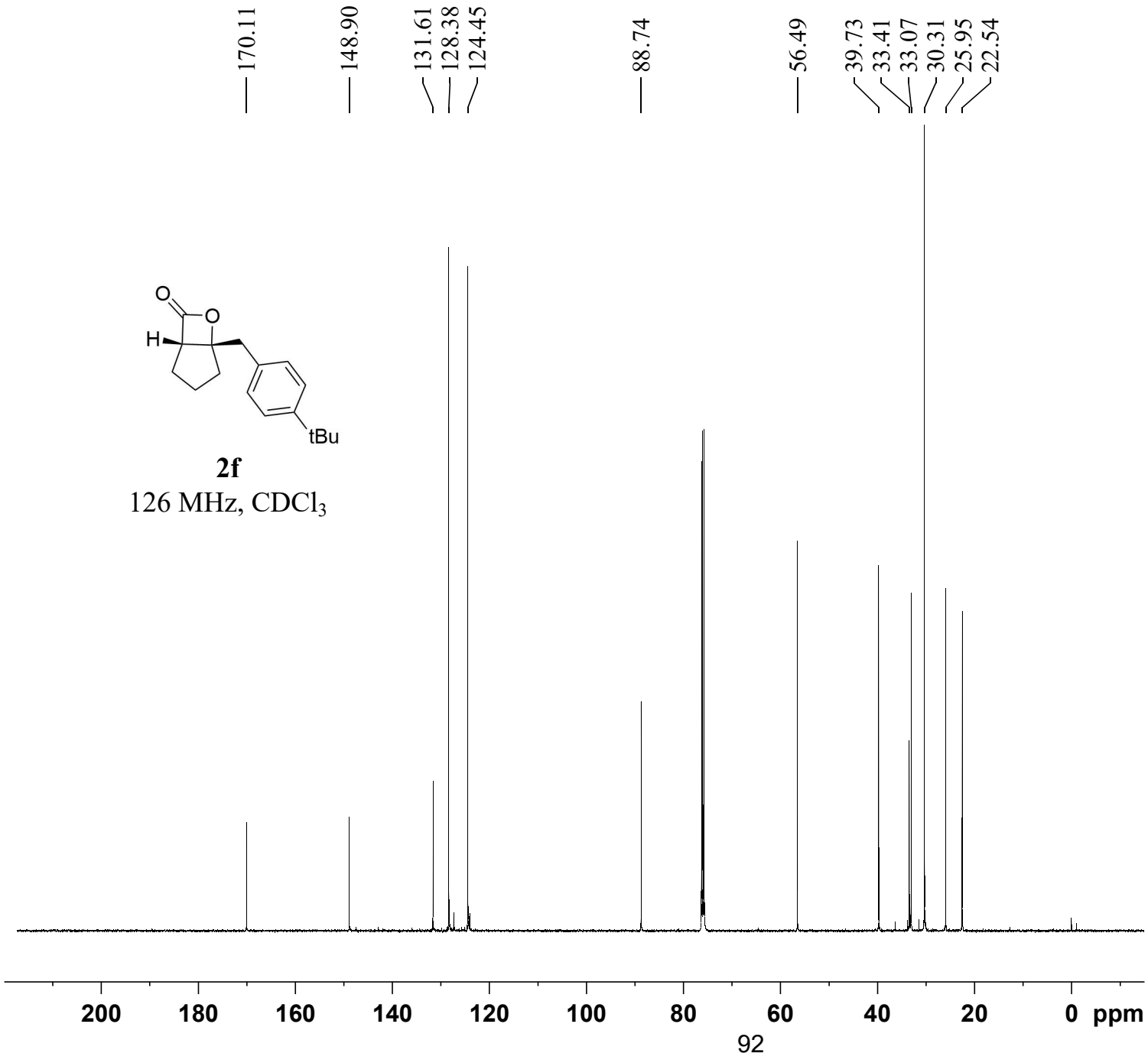
```
Current Data Parameters
NAME        500M-2020xia
EXPNO                 38
PROCNO                 1

F2 - Acquisition Parameters
Date_           20210110
Time              23.36
INSTRUM           spect
PROBHD   5 mm CPPBBO BB
PULPROG          zgpg30
TD                65536
SOLVENT           CDCl3
NS                  400
DS                    4
SWH           29761.904 Hz
FIDRES         0.454131 Hz
AQ             1.1010048 sec
RG                192.89
DW                16.800 usec
DE                18.00 usec
TE                298.2 K
D1            2.00000000 sec
D11           0.03000000 sec
TD0                   1

======== CHANNEL f1 ========
SFO1         125.7703637 MHz
NUC1                 13C
P1                  9.80 usec
PLW1         57.00000000 W

======== CHANNEL f2 ========
SFO2         500.1320005 MHz
NUC2                 1H
CPDPRG[2          waltz16
PCPD2               80.00 usec
PLW2         20.00000000 W
PLW12         0.35778001 W
PLW13         0.22898000 W

F2 - Processing parameters
SI                  32768
SF           125.7577885 MHz
WDW                  EM
SSB       0
LB                  1.00 Hz
GB        0
PC                  1.40
```



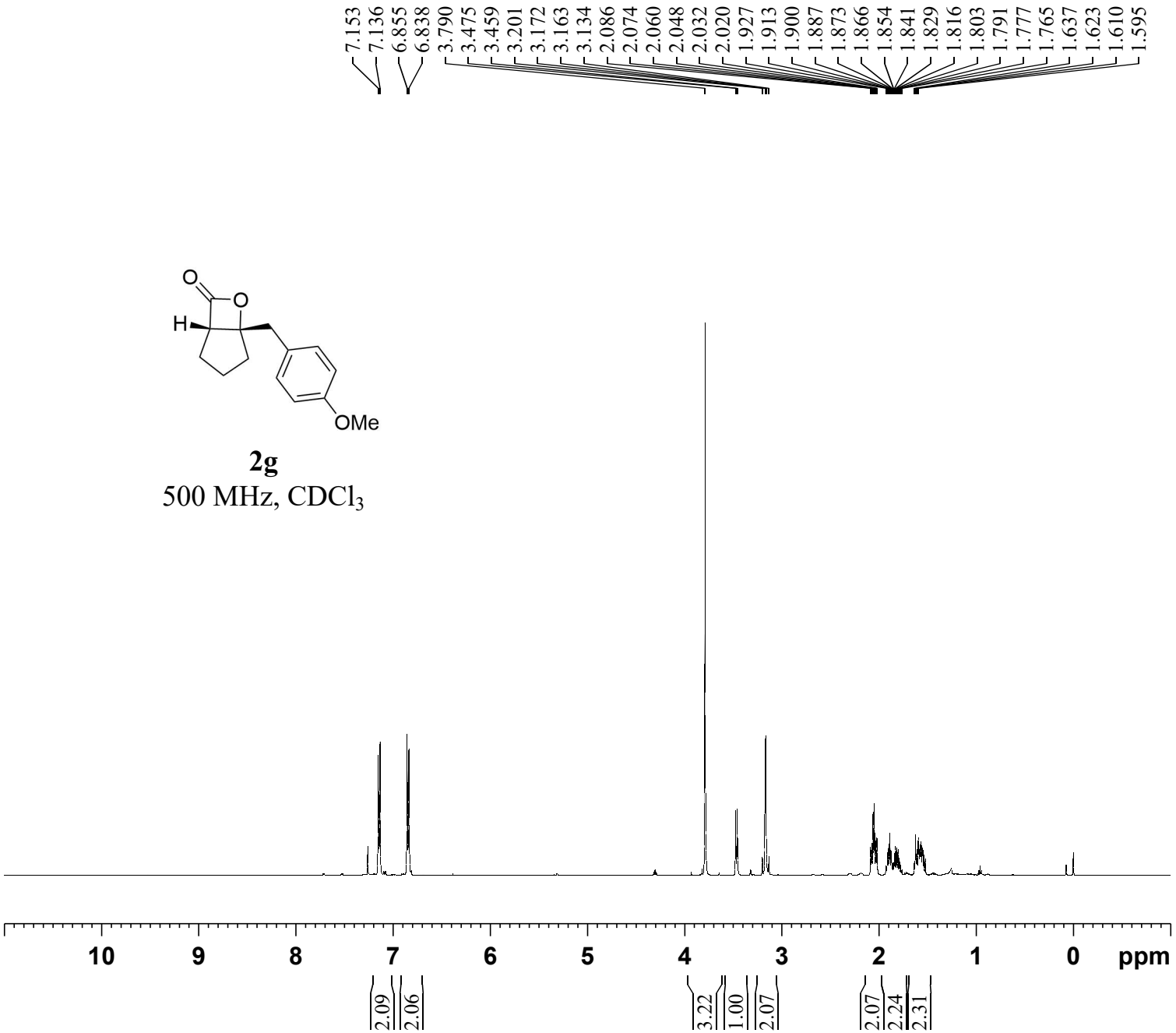
2f

500 MHz, $CDCl_3$

```
Current Data Parameters
NAME        500M-2020xia
EXPNO                 60
PROCNO                 1

F2 - Acquisition Parameters
Date_           20210111
Time              22.06
INSTRUM           spect
PROBHD   5 mm CPPBBO BB
PULPROG             zg30
TD                65536
SOLVENT           CDCl3
NS                   16
DS                    2
SWH           10000.000 Hz
FIDRES         0.152588 Hz
AQ            3.2767999 sec
RG                31.72
DW               50.000 usec
DE                6.50 usec
TE               298.2 K
D1            1.00000000 sec
D11      0 sec
TD0                   1

======== CHANNEL f1 ========
SFO1         500.1330885 MHz
NUC1                 1H
P1                10.59 usec
PLW1         20.00000000 W

======== CHANNEL f2 ========
SFO2         500.1330885 MHz
NUC2                off
CPDPRG[2
PCPD2     0 usec
PLW2      0 W
PLW12     0 W
PLW13     0 W

F2 - Processing parameters
SI                  65536
SF           500.1300163 MHz
WDW                   EM
SSB       0
LB                 0.30 Hz
GB        0
PC                 1.00
```



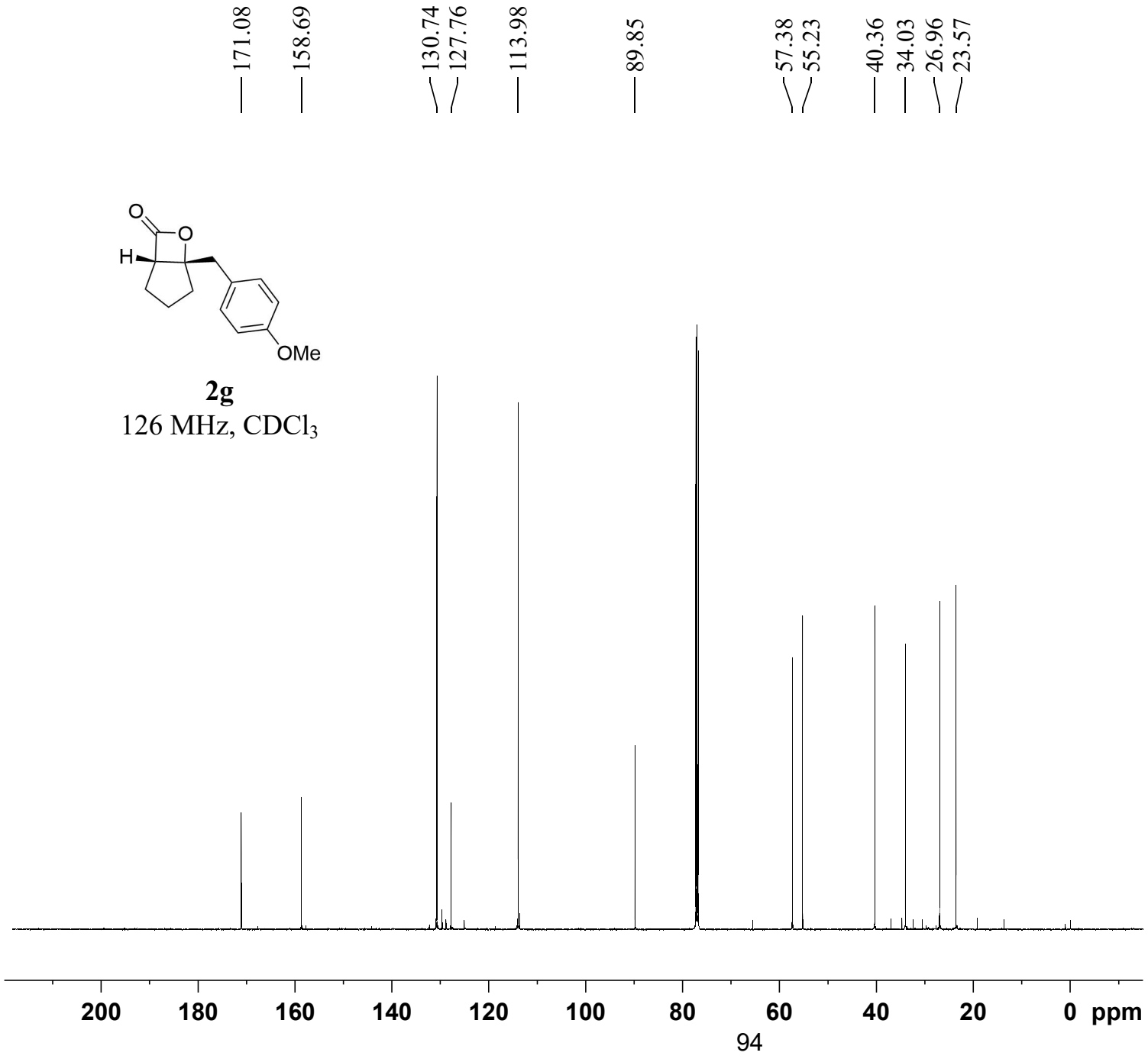

```
Current Data Parameters
NAME        500M-2020xia
EXPNO                 52
PROCNO                 1

F2 - Acquisition Parameters
Date_           20210111
Time               5.56
INSTRUM           spect
PROBHD   5 mm CPPBBO BB
PULPROG          zgpg30
TD                65536
SOLVENT           CDCl3
NS                  500
DS                    4
SWH           29761.904 Hz
FIDRES         0.454131 Hz
AQ            1.1010048 sec
RG               192.89
DW               16.800 usec
DE               18.00 usec
TE               298.2 K
D1           2.00000000 sec
D11          0.03000000 sec
TD0                   1

======== CHANNEL f1 ========
SFO1        125.7703637 MHz
NUC1                13C
P1                 9.80 usec
PLW1        57.00000000 W

======== CHANNEL f2 ========
SFO2        500.1320005 MHz
NUC2                1H
CPDPRG[2         waltz16
PCPD2             80.00 usec
PLW2        20.00000000 W
PLW12        0.35778001 W
PLW13        0.22898000 W

F2 - Processing parameters
SI                 32768
SF          125.7579205 MHz
WDW                   EM
SSB       0
LB                 1.00 Hz
GB        0
PC                 1.40
```



2g

500 MHz, $CDCl_3$

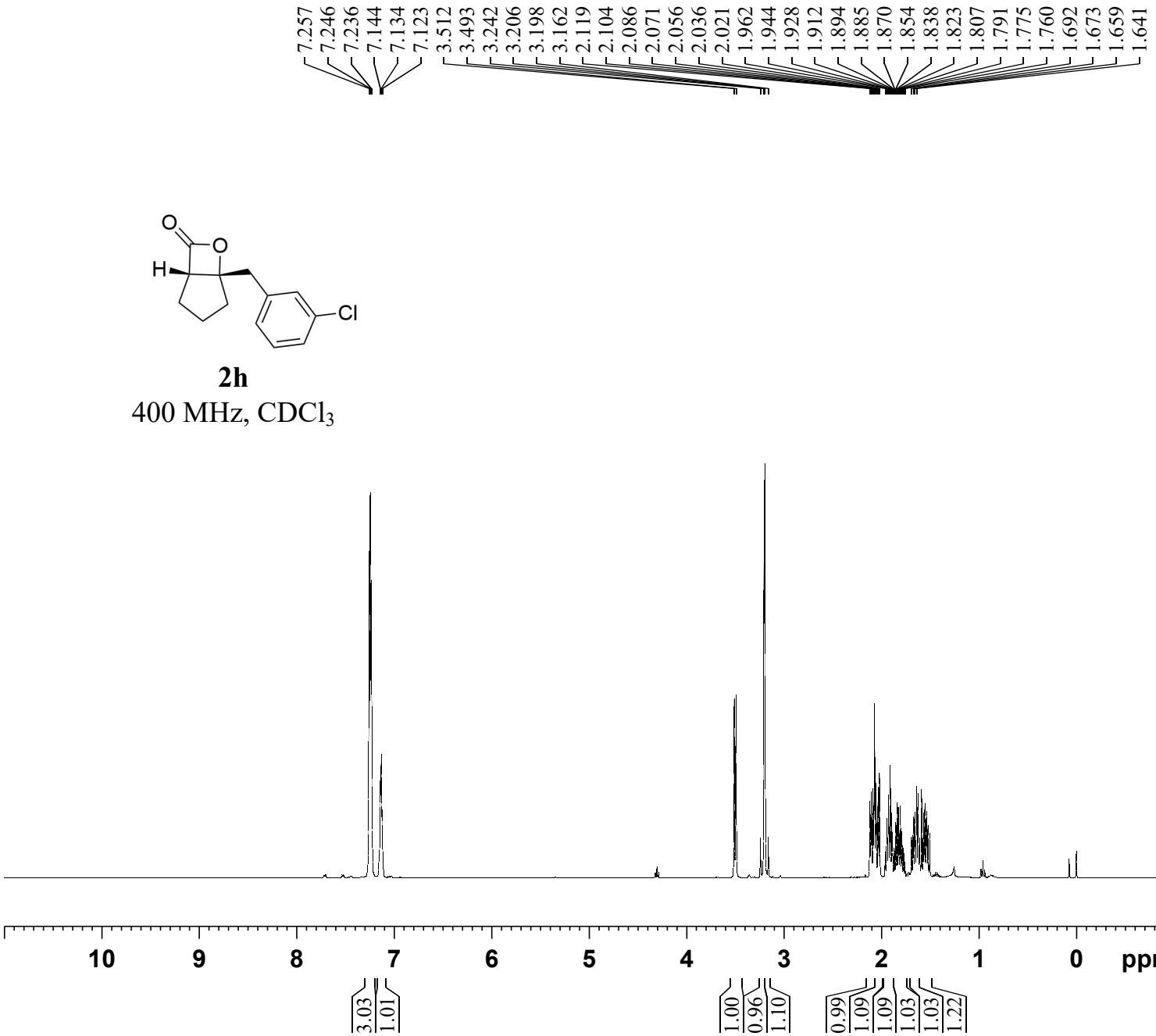
```
Current Data Parameters
NAME        500M-2020xia
EXPNO                  7
PROCNO                 1

F2 - Acquisition Parameters
Date_           20201118
Time                6.30
INSTRUM            spect
PROBHD   5 mm CPPBBO BB
PULPROG             zg30
TD                 65536
SOLVENT            CDCl3
NS                    16
DS                     2
SWH           10000.000 Hz
FIDRES         0.152588 Hz
AQ             3.2767999 sec
RG                 31.72
DW                50.000 usec
DE                 6.50 usec
TE                298.2 K
D1           1.00000000 sec
D11      0 sec
TD0                    1

======== CHANNEL f1 ========
SFO1         500.1330885 MHz
NUC1                   1H
P1                 10.59 usec
PLW1         20.00000000 W

======== CHANNEL f2 ========
SFO2         500.1330885 MHz
NUC2                  off
CPDPRG[2
PCPD2     0 usec
PLW2      0 W
PLW12     0 W
PLW13     0 W

F2 - Processing parameters
SI                 65536
SF           500.1300113 MHz
WDW                   EM
SSB       0
LB                 0.30 Hz
GB        0
PC                 1.00
```



2g

126 MHz, $CDCl_3$

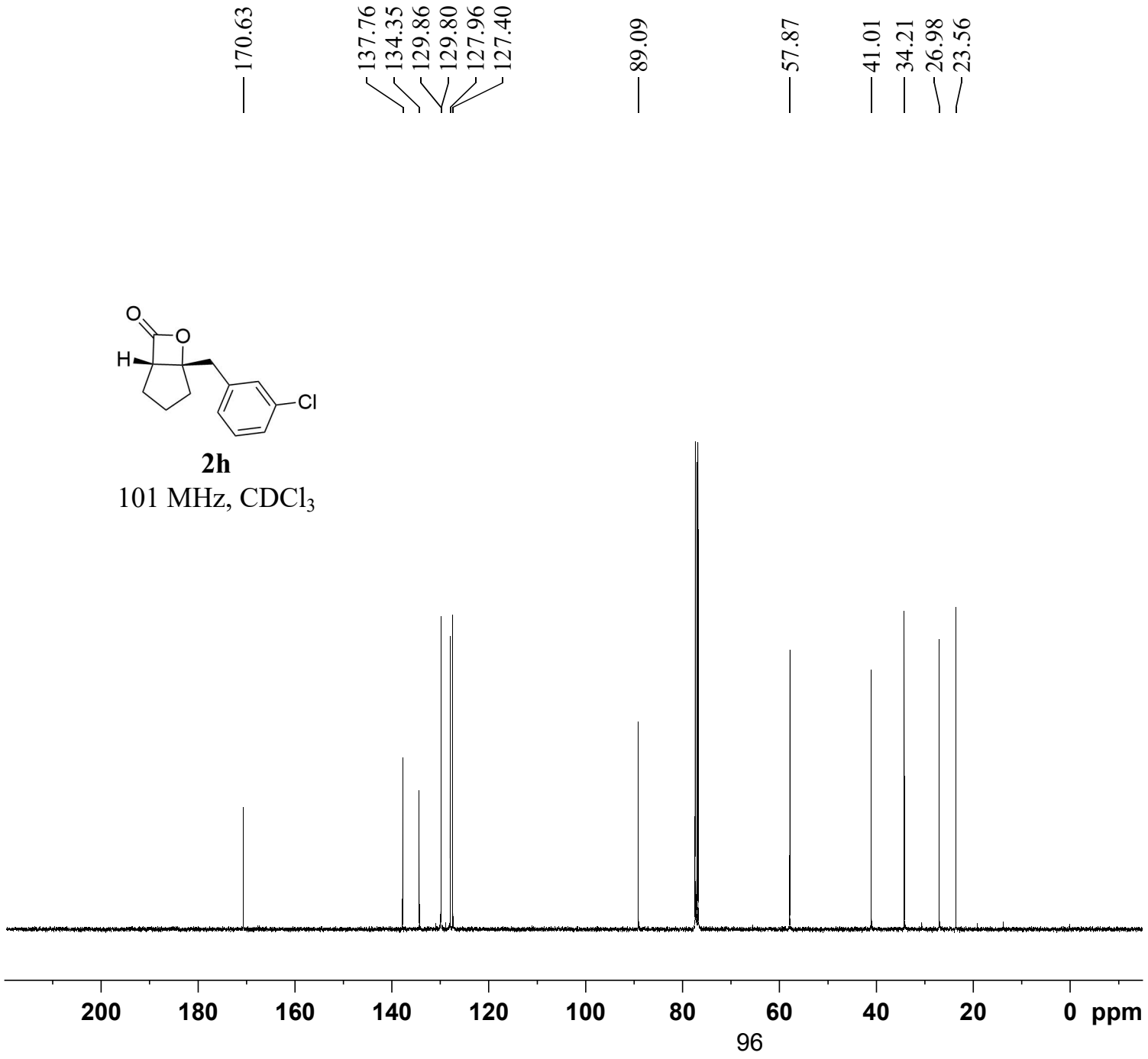
```
Current Data Parameters
NAME        500M-2020xia
EXPNO                  8
PROCNO                 1

F2 - Acquisition Parameters
Date_           20201118
Time               7.24
INSTRUM           spect
PROBHD   5 mm CPPBBO BB
PULPROG          zgpg30
TD                65536
SOLVENT           CDCl3
NS                 1024
DS                    4
SWH           29761.904 Hz
FIDRES         0.454131 Hz
AQ            1.1010048 sec
RG               192.89
DW               16.800 usec
DE               18.00 usec
TE               298.2 K
D1           2.00000000 sec
D11          0.03000000 sec
TD0                   1

======== CHANNEL f1 ========
SFO1        125.7703637 MHz
NUC1                13C
P1                9.80 usec
PLW1        57.00000000 W

======== CHANNEL f2 ========
SFO2        500.1320005 MHz
NUC2                1H
CPDPRG[2         waltz16
PCPD2            80.00 usec
PLW2        20.00000000 W
PLW12        0.35778001 W
PLW13        0.22898000 W

F2 - Processing parameters
SI                 32768
SF          125.7577906 MHz
WDW                   EM
SSB       0
LB                1.00 Hz
GB        0
PC                1.40
```



2h

400 MHz, $CDCl_3$

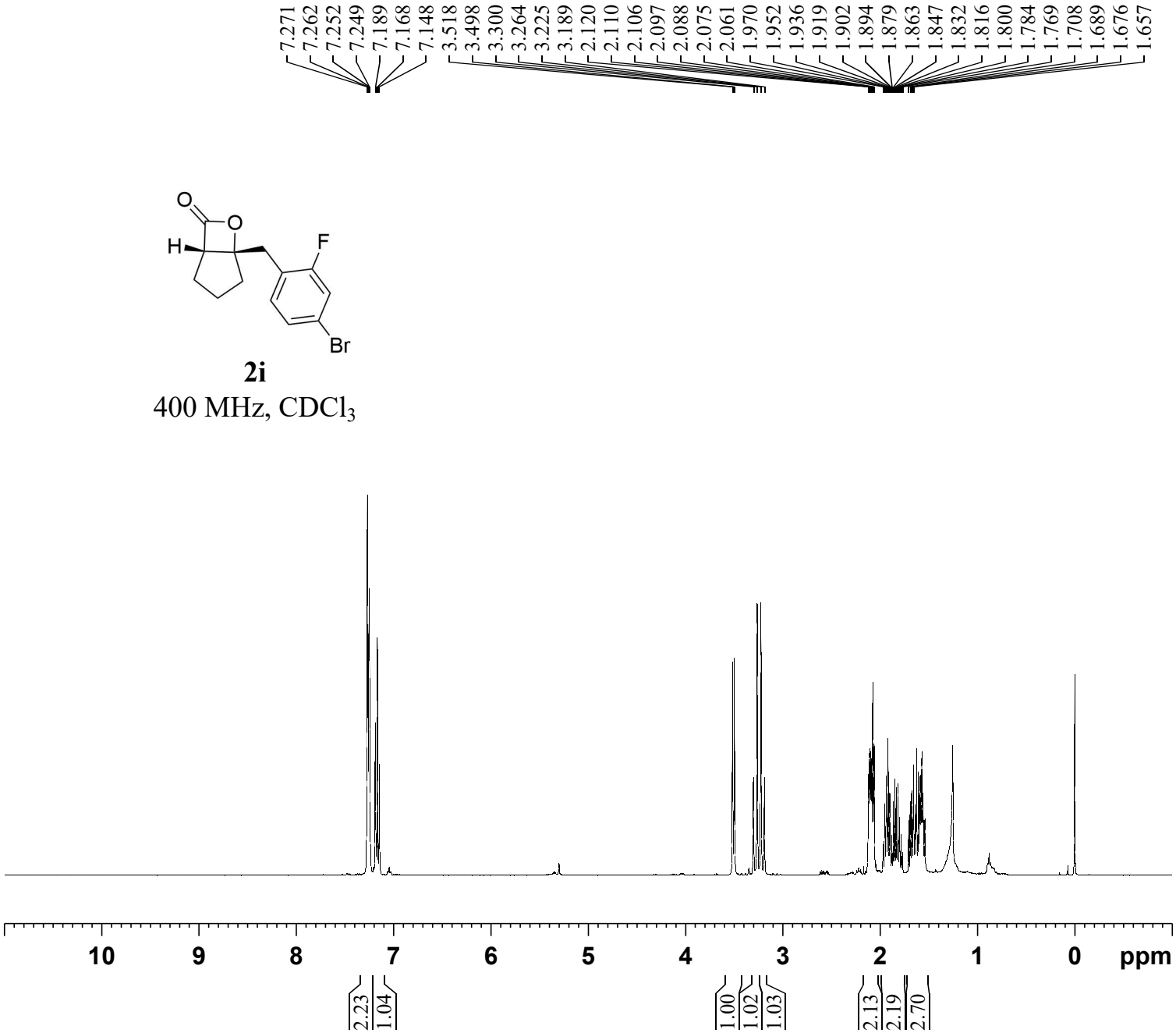
```
Current Data Parameters
NAME                 400xia
EXPNO                    11
PROCNO                    1

F2 - Acquisition Parameters
Date_              20201017
Time                   3.31
INSTRUM               spect
PROBHD   5 mm PABBO BB/
PULPROG                zg30
TD                    32768
SOLVENT               CDCl3
NS                       16
DS                        0
SWH                8012.820 Hz
FIDRES            0.244532 Hz
AQ               2.0447233 sec
RG                   102.73
DW                   62.400 usec
DE                    6.50 usec
TE                   298.4 K
D1              2.00000000 sec
D11       0 sec
TD0                       1

======== CHANNEL f1 ========
SFO1           400.2424716 MHz
NUC1                      1H
P1                    14.30 usec
PLW1           12.00000000 W

======== CHANNEL f2 ========
SFO2           400.2424716 MHz
NUC2                    off
CPDPRG[2
PCPD2     0 usec
PLW2      0 W
PLW12     0 W
PLW13     0 W

F2 - Processing parameters
SI                     65536
SF           400.2400077 MHz
WDW                       EM
SSB       0
LB                    0.30 Hz
GB        0
PC                    1.00
```



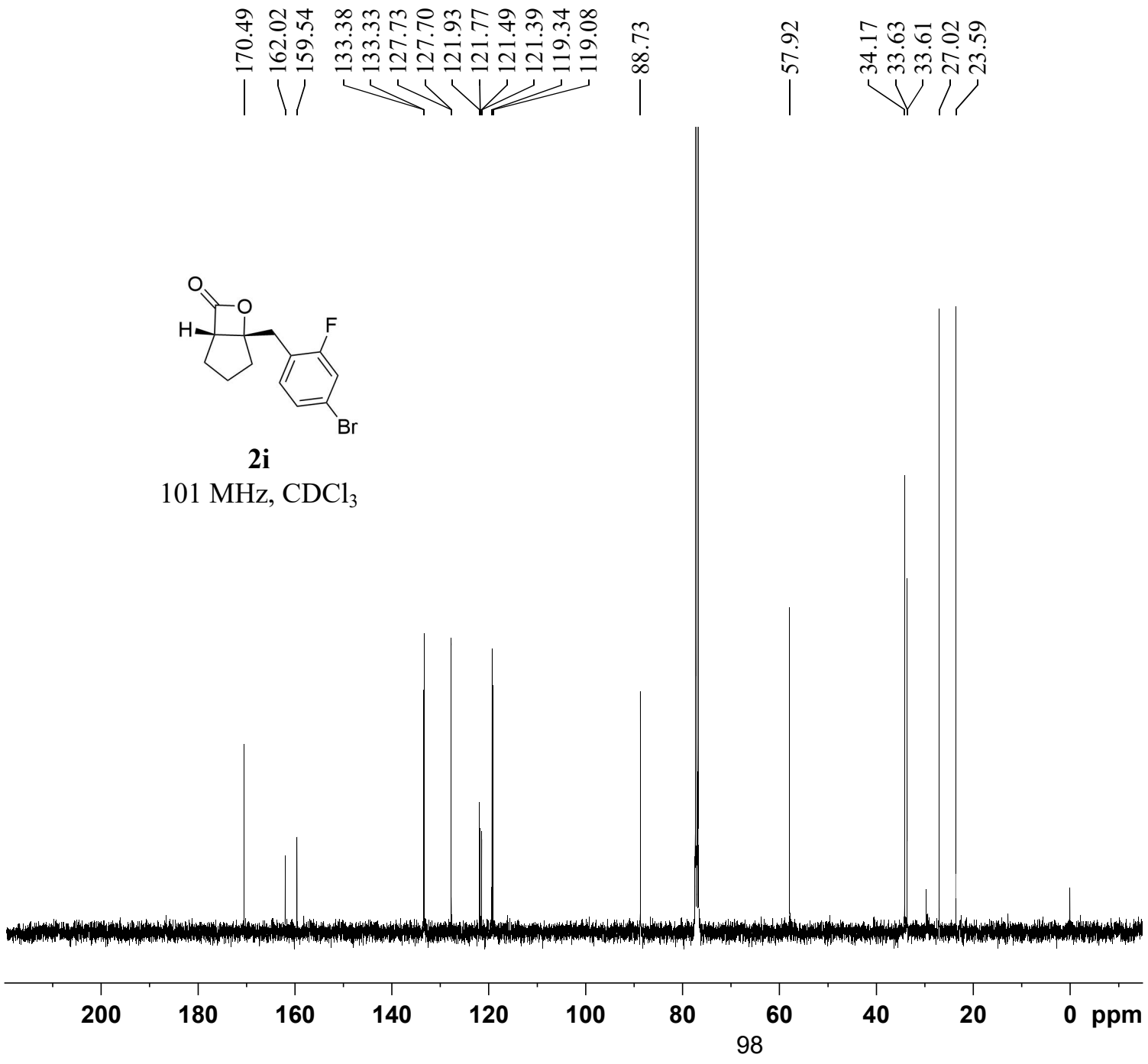
2h

101 MHz, $CDCl_3$

```
Current Data Parameters
NAME                 400xia
EXPNO                    12
PROCNO                    1

F2 - Acquisition Parameters
Date_              20201017
Time                   4.30
INSTRUM               spect
PROBHD   5 mm PABBO BB/
PULPROG              zgpg30
TD                    65536
SOLVENT               CDCl3
NS                     1024
DS                        4
SWH               24038.461 Hz
FIDRES             0.366798 Hz
AQ                1.3631488 sec
RG                   206.33
DW                   20.800 usec
DE                    6.50 usec
TE                   299.1 K
D1               2.00000000 sec
D11              0.03000000 sec
TD0                       1

======== CHANNEL f1 ========
SFO1            100.6504916 MHz
NUC1                    13C
P1                    10.00 usec
PLW1            54.00000000 W

======== CHANNEL f2 ========
SFO2            400.2416010 MHz
NUC2                    1H
CPDPRG[2             waltz16
PCPD2                 90.00 usec
PLW2            12.00000000 W
PLW12            0.30294999 W
PLW13            0.24539000 W

F2 - Processing parameters
SI                      32768
SF              100.6404280 MHz
WDW                        EM
SSB          0
LB                     1.00 Hz
GB           0
PC                     1.40
```




```
Current Data Parameters
NAME                 400xia
EXPNO                    46
PROCNO                    1

F2 - Acquisition Parameters
Date_              20210520
Time                 16.00
INSTRUM              spect
PROBHD    5 mm PABBO BB/
PULPROG               zg30
TD                   32768
SOLVENT              CDCl3
NS                      16
DS                       0
SWH               8012.820 Hz
FIDRES            0.244532 Hz
AQ               2.0447233 sec
RG                  206.33
DW                  62.400 usec
DE                   6.50 usec
TE                  299.3 K
D1              2.00000000 sec
D11       0 sec
TD0                      1

======== CHANNEL f1 ========
SFO1           400.2424716 MHz
NUC1                    1H
P1                   14.30 usec
PLW1           12.00000000 W

======== CHANNEL f2 ========
SFO2           400.2424716 MHz
NUC2                   off
CPDPRG[2
PCPD2     0 usec
PLW2      0 W
PLW12     0 W
PLW13     0 W

F2 - Processing parameters
SI                    65536
SF             400.2400087 MHz
WDW                      EM
SSB       0
LB                    0.30 Hz
GB        0
PC                    1.00
```

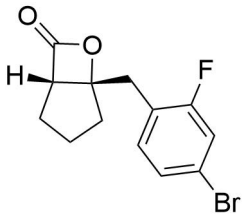


2i

101 MHz, $CDCl_3$

```
Current Data Parameters
NAME                 400xia
EXPNO                    47
PROCNO                    1

F2 - Acquisition Parameters
Date_              20210520
Time                 16.59
INSTRUM              spect
PROBHD   5 mm PABBO BB/
PULPROG             zgpg30
TD                   65536
SOLVENT              CDCl3
NS                    1024
DS                       4
SWH              24038.461 Hz
FIDRES            0.366798 Hz
AQ               1.3631488 sec
RG                  206.33
DW                  20.800 usec
DE                   6.50 usec
TE                  300.4 K
D1              2.00000000 sec
D11             0.03000000 sec
TD0                      1

======== CHANNEL f1 ========
SFO1           100.6504916 MHz
NUC1                   13C
P1                  10.00 usec
PLW1           54.00000000 W

======== CHANNEL f2 ========
SFO2           400.2416010 MHz
NUC2                   1H
CPDPRG[2           waltz16
PCPD2               90.00 usec
PLW2           12.00000000 W
PLW12           0.30294999 W
PLW13           0.24539000 W

F2 - Processing parameters
SI                    32768
SF             100.6404280 MHz
WDW                      EM
SSB       0
LB                   1.00 Hz
GB        0
PC                   1.40
```

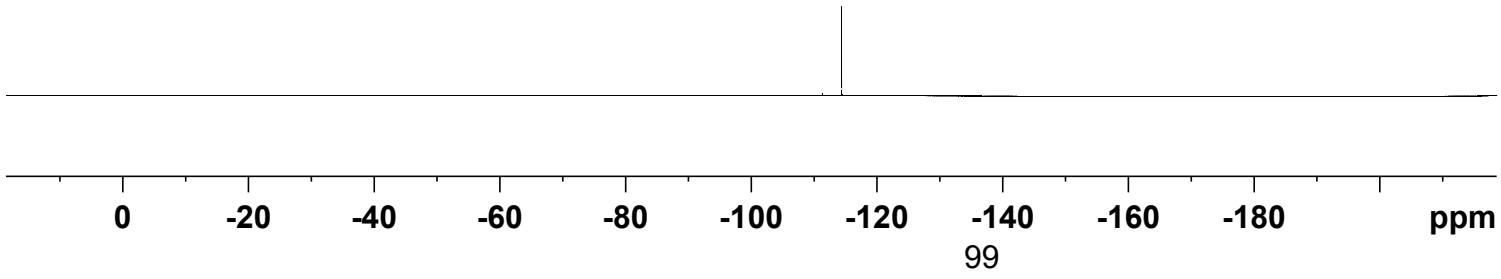
-114.43

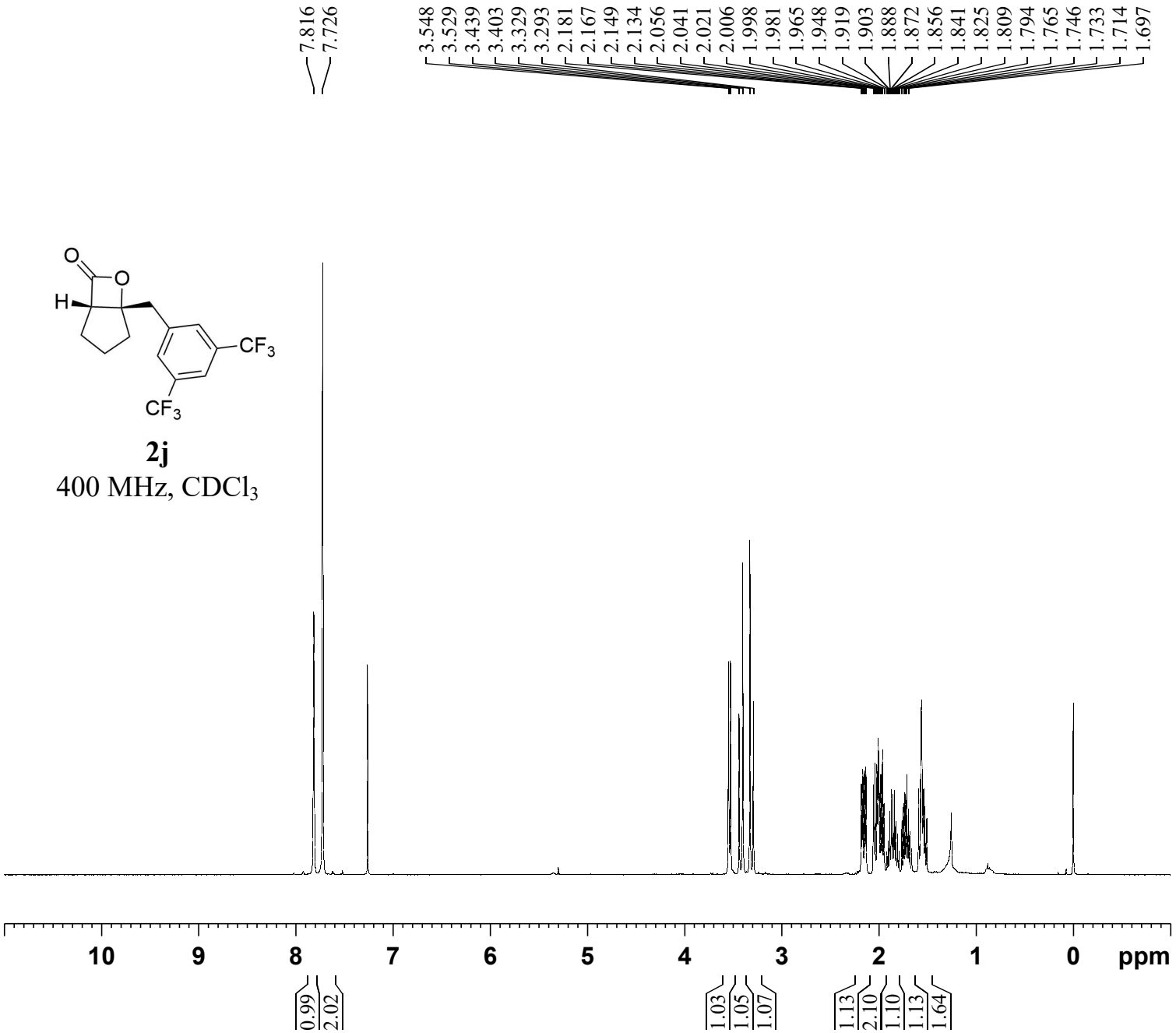


2i

376 MHz, $CDCl_3$

```
Current Data Parameters
NAME         400M-2022-F
EXPNO                  7
PROCNO                 1

F1 - Acquisition parameters
TD                   128
SFO1          400.242 MHz
FIDRES       59.637405 Hz
SW             9.536 ppm
FnMODE              QF

F2 - Processing parameters
SI                 65536
SF          376.6018696 MHz
WDW                  EM
SSB       0
LB                0.30 Hz
GB        0
PC                1.00
```

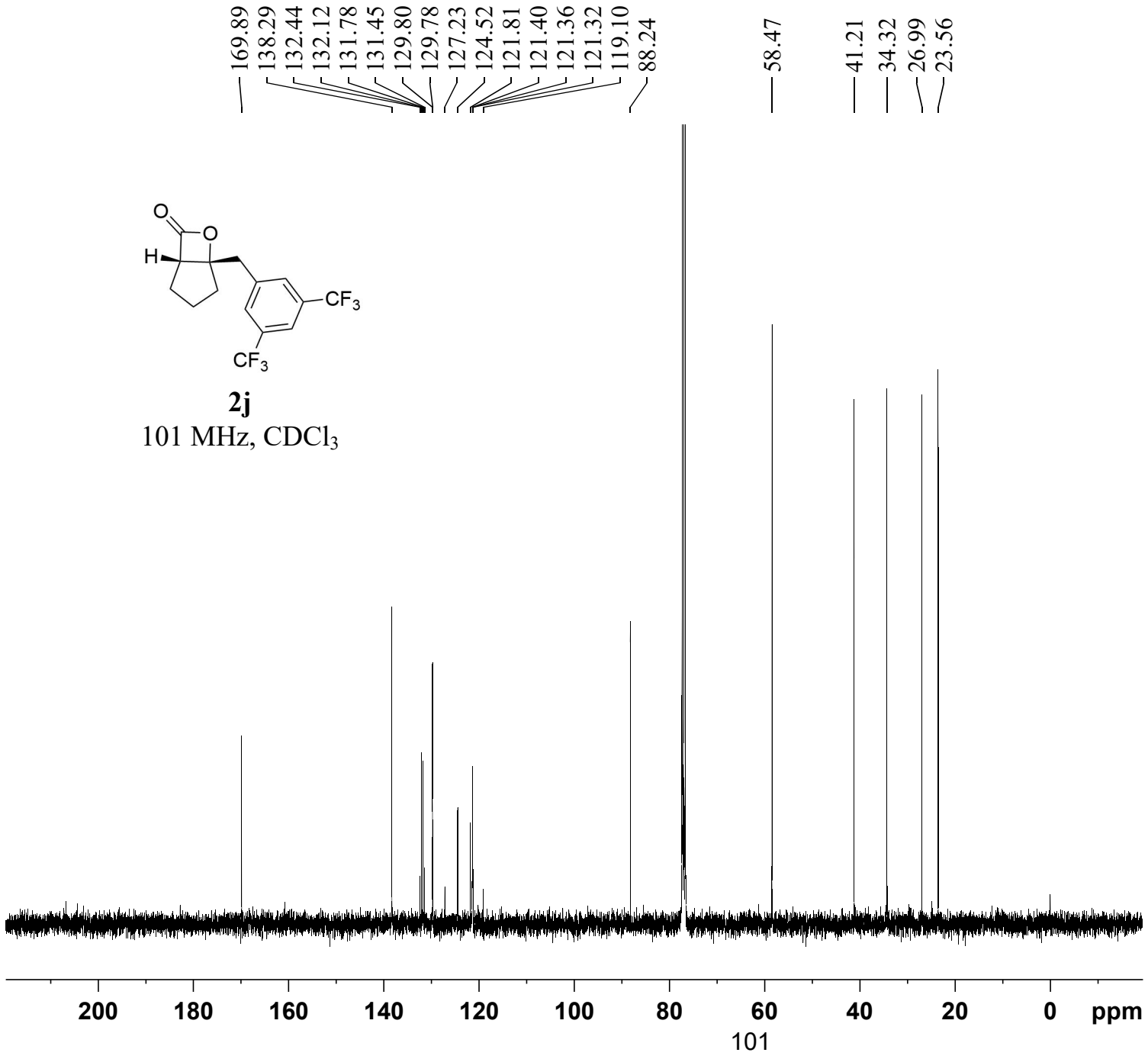




2j

400 MHz, $CDCl_3$

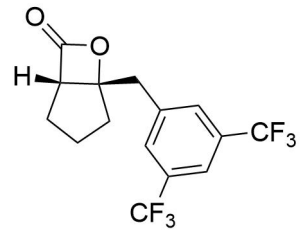
```
Current Data Parameters
NAME                 400xia
EXPNO                    40
PROCNO                    1

F2 - Acquisition Parameters
Date_              20210519
Time                  22.37
INSTRUM               spect
PROBHD   5 mm PABBO BB/
PULPROG                zg30
TD                    32768
SOLVENT               CDCl3
NS                       16
DS                        0
SWH                8012.820 Hz
FIDRES            0.244532 Hz
AQ               2.0447233 sec
RG                   206.33
DW                   62.400 usec
DE                    6.50 usec
TE                   299.3 K
D1              2.00000000 sec
D11      0 sec
TD0                       1

======== CHANNEL f1 ========
SFO1           400.2424716 MHz
NUC1                     1H
P1                    14.30 usec
PLW1           12.00000000 W

======== CHANNEL f2 ========
SFO2           400.2424716 MHz
NUC2                    off
CPDPRG[2
PCPD2     0 usec
PLW2      0 W
PLW12     0 W
PLW13     0 W

F2 - Processing parameters
SI                    65536
SF           400.2400087 MHz
WDW                      EM
SSB       0
LB                    0.30 Hz
GB        0
PC                    1.00
```



2j

101 MHz, $CDCl_3$

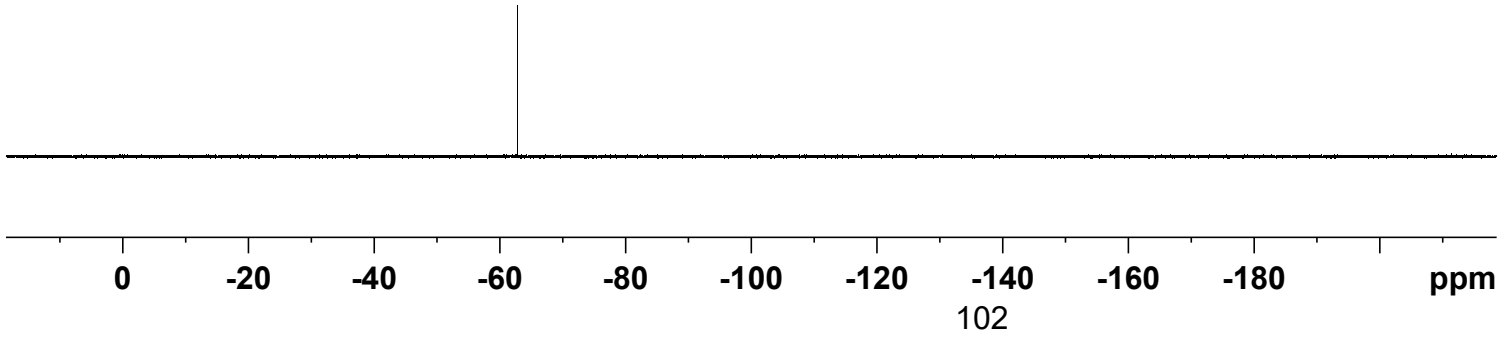
```
Current Data Parameters
NAME                 400xia
EXPNO                    41
PROCNO                    1

F2 - Acquisition Parameters
Date_              20210519
Time                 23.36
INSTRUM              spect
PROBHD   5 mm PABBO BB/
PULPROG             zgpg30
TD                   65536
SOLVENT              CDCl3
NS                    1024
DS                       4
SWH              24038.461 Hz
FIDRES            0.366798 Hz
AQ                1.3631488 sec
RG                  206.33
DW                  20.800 usec
DE                   6.50 usec
TE                  300.4 K
D1               2.00000000 sec
D11              0.03000000 sec
TD0                      1

======== CHANNEL f1 ========
SFO1            100.6504916 MHz
NUC1                    13C
P1                   10.00 usec
PLW1            54.00000000 W

======== CHANNEL f2 ========
SFO2            400.2416010 MHz
NUC2                    1H
CPDPRG[2            waltz16
PCPD2                90.00 usec
PLW2            12.00000000 W
PLW12            0.30294999 W
PLW13            0.24539000 W

F2 - Processing parameters
SI                      32768
SF              100.6404280 MHz
WDW                        EM
SSB          0
LB                    1.00 Hz
GB           0
PC                    1.40
```

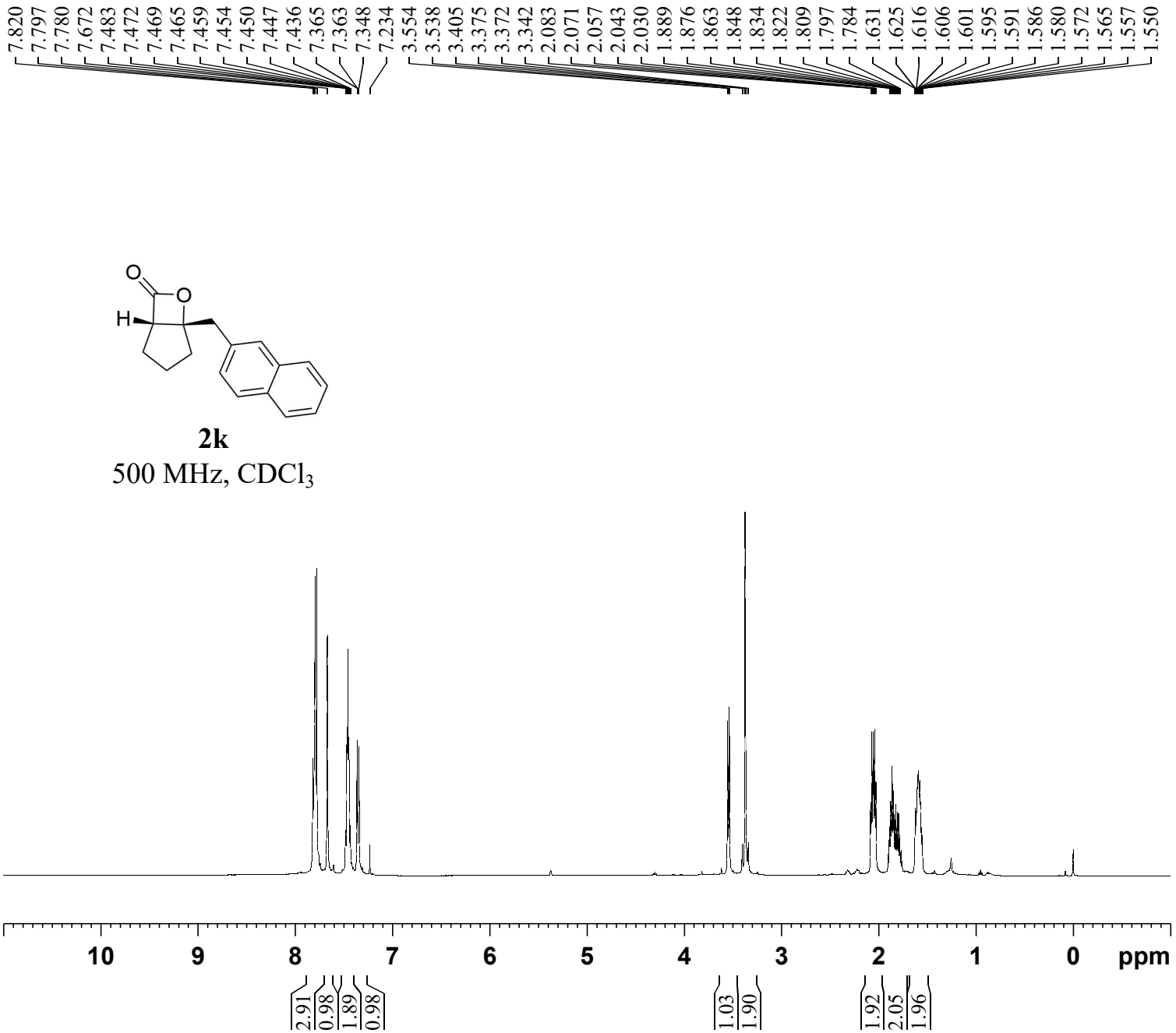


2j

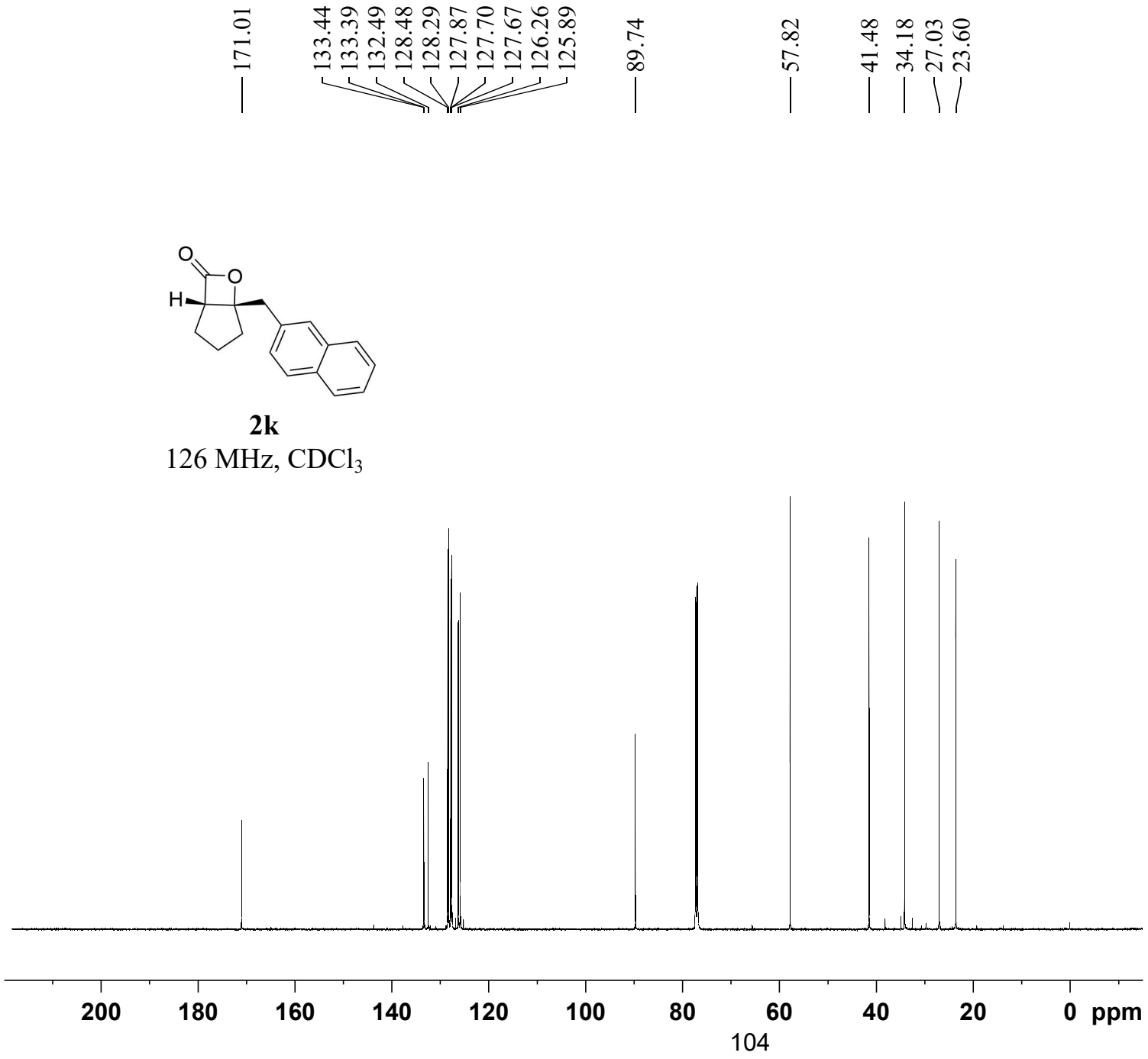
658 MHz, $CDCl_3$

```
Current Data Parameters
NAME         400M-2022-F
EXPNO                  6
PROCNO                 1

F2 - Acquisition Parameters
Date_           20221128
Time              14.46 h
INSTRUM           Avance
PROBHD   Z149054_0001 (
PULPROG             zgig
TD                131072
SOLVENT            CDCl3
NS                    64
DS                     4
SWH           156250.000 Hz
FIDRES          2.384186 Hz
AQ             0.4194304 sec
RG                   101
DW                 3.200 usec
DE                 6.50 usec
TE                 298.0 K
D1            1.00000000 sec
D11           0.03000000 sec
TD0                    1
SFO1         658.7615635 MHz
NUC1                 19F
P1                25.00 usec
PLW1         21.74500084 W
SFO2         700.1828007 MHz
NUC2                 1H
CPDPRG[2         waltz16
PCPD2              65.00 usec
PLW2          8.61509991 W
PLW12         0.39965999 W

F2 - Processing parameters
SI                 65536
SF           658.8274462 MHz
WDW                   EM
SSB       0
LB                 0.30 Hz
GB        0
PC                 1.00
```





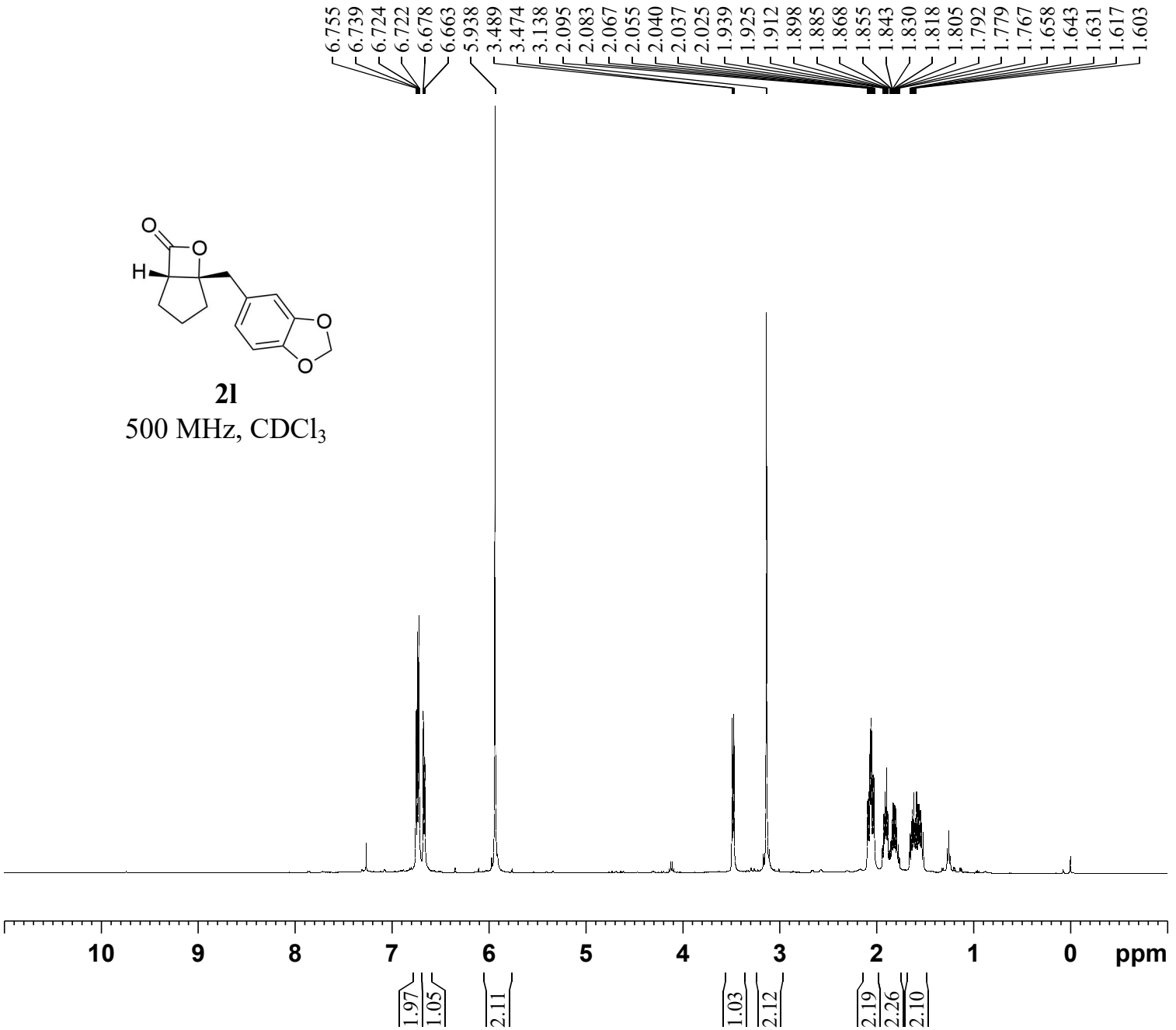

```
Current Data Parameters
NAME        500M-2020xia
EXPNO                 48
PROCNO                 1

F2 - Acquisition Parameters
Date_           20210111
Time                4.29
INSTRUM            spect
PROBHD   5 mm CPPBBO BB
PULPROG             zg30
TD                 65536
SOLVENT            CDCl3
NS                    16
DS                     2
SWH           10000.000 Hz
FIDRES         0.152588 Hz
AQ            3.2767999 sec
RG                 31.72
DW                50.000 usec
DE                 6.50 usec
TE                298.2 K
D1           1.00000000 sec
D11      0 sec
TD0                    1

======== CHANNEL f1 ========
SFO1         500.1330885 MHz
NUC1                   1H
P1                 10.59 usec
PLW1         20.00000000 W

======== CHANNEL f2 ========
SFO2         500.1330885 MHz
NUC2                  off
CPDPRG[2
PCPD2     0 usec
PLW2      0 W
PLW12     0 W
PLW13     0 W

F2 - Processing parameters
SI                 65536
SF           500.1300249 MHz
WDW                   EM
SSB       0
LB                 0.30 Hz
GB        0
PC                 1.00
```



2k

126 MHz, $CDCl_3$

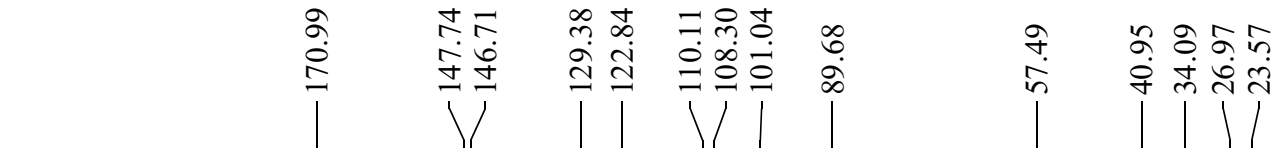
```
Current Data Parameters
NAME        500M-2020xia
EXPNO                 49
PROCNO                 1

F2 - Acquisition Parameters
Date_           20210111
Time               4.56
INSTRUM           spect
PROBHD   5 mm CPPBBO BB
PULPROG          zgpg30
TD                65536
SOLVENT           CDCl3
NS                  500
DS                    4
SWH           29761.904 Hz
FIDRES         0.454131 Hz
AQ            1.1010048 sec
RG               192.89
DW               16.800 usec
DE               18.00 usec
TE               298.2 K
D1           2.00000000 sec
D11          0.03000000 sec
TD0                   1

======== CHANNEL f1 ========
SFO1        125.7703637 MHz
NUC1                13C
P1                 9.80 usec
PLW1        57.00000000 W

======== CHANNEL f2 ========
SFO2        500.1320005 MHz
NUC2                1H
CPDPRG[2         waltz16
PCPD2             80.00 usec
PLW2        20.00000000 W
PLW12        0.35778001 W
PLW13        0.22898000 W

F2 - Processing parameters
SI                 32768
SF          125.7577885 MHz
WDW                   EM
SSB       0
LB                 1.00 Hz
GB        0
PC                 1.40
```

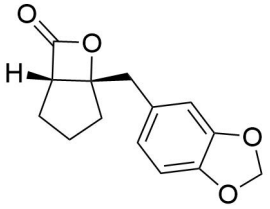


2l

500 MHz, $CDCl_3$

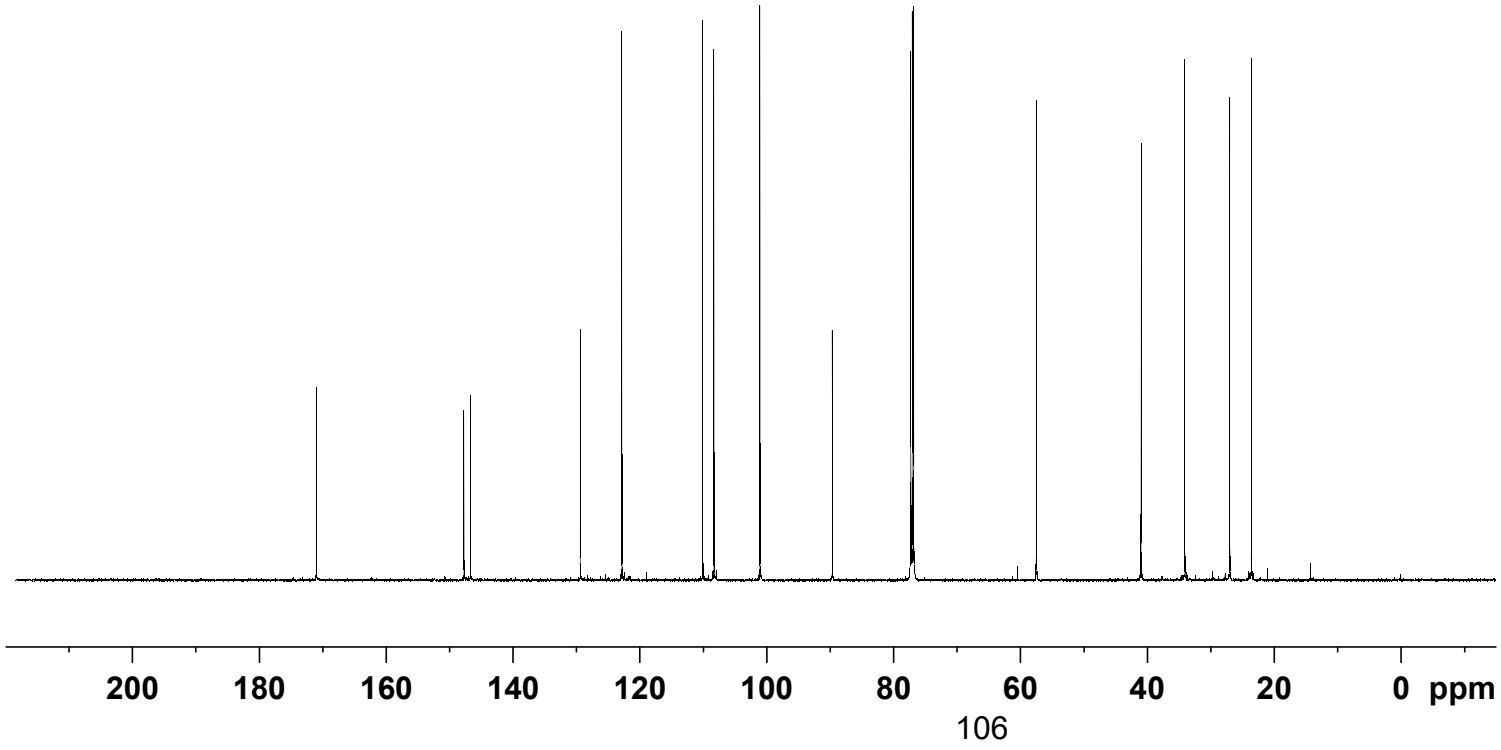
```
Current Data Parameters
NAME        500M-2020xia
EXPNO                 54
PROCNO                 1

F2 - Acquisition Parameters
Date_           20210111
Time                6.29
INSTRUM            spect
PROBHD   5 mm CPPBBO BB
PULPROG             zg30
TD                 65536
SOLVENT            CDCl3
NS                    16
DS                     2
SWH           10000.000 Hz
FIDRES         0.152588 Hz
AQ            3.2767999 sec
RG                 31.72
DW                50.000 usec
DE                 6.50 usec
TE                298.2 K
D1           1.00000000 sec
D11      0 sec
TD0                    1

======== CHANNEL f1 ========
SFO1         500.1330885 MHz
NUC1                   1H
P1                 10.59 usec
PLW1         20.00000000 W

======== CHANNEL f2 ========
SFO2         500.1330885 MHz
NUC2                  off
CPDPRG[2
PCPD2     0 usec
PLW2      0 W
PLW12     0 W
PLW13     0 W

F2 - Processing parameters
SI                 65536
SF          500.1300083 MHz
WDW                   EM
SSB       0
LB                 0.30 Hz
GB        0
PC                 1.00
```



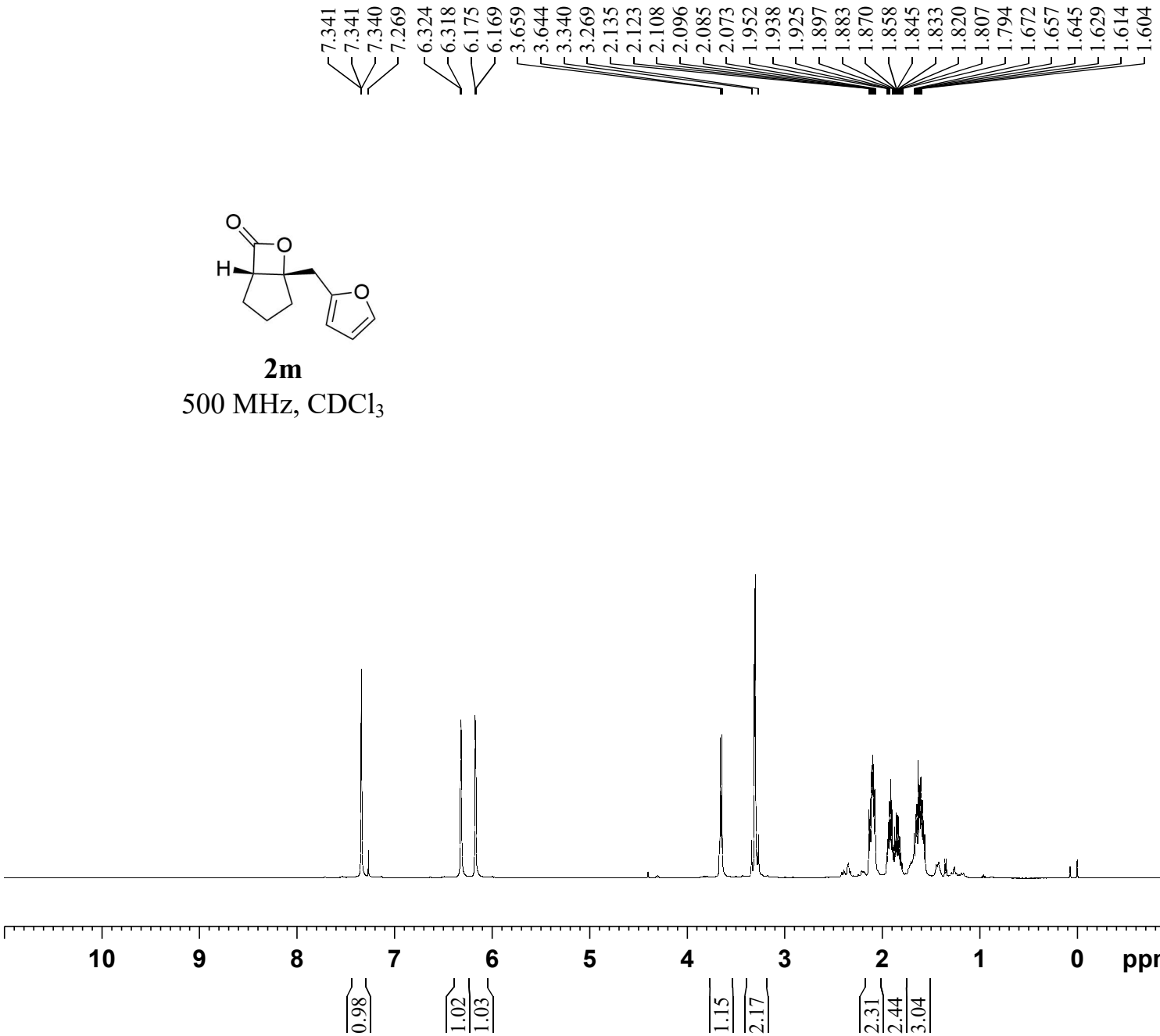


2l

126 MHz, $CDCl_3$



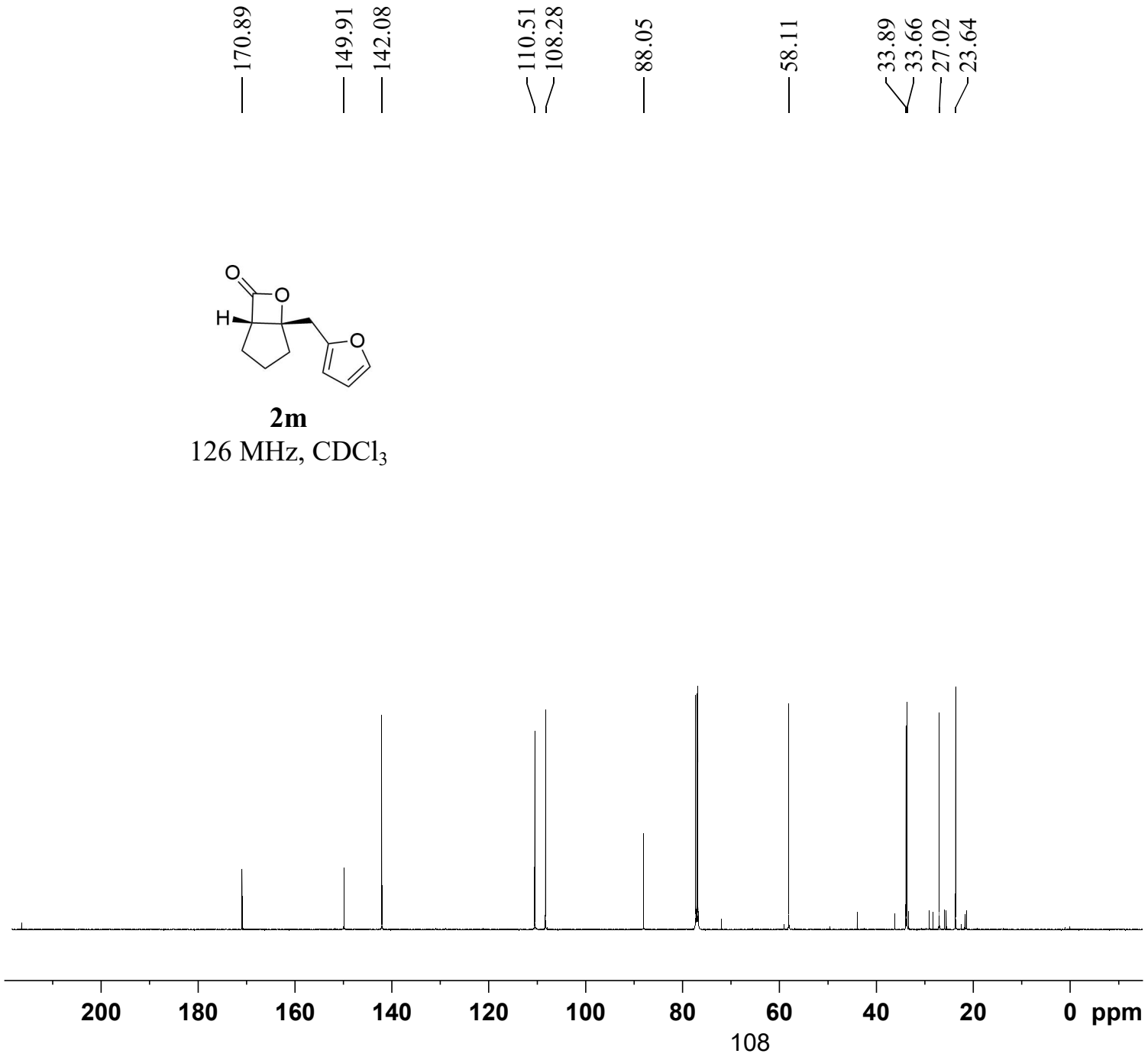

```
Current Data Parameters
NAME        500M-2020xia
EXPNO                 55
PROCNO                 1

F2 - Acquisition Parameters
Date_           20210111
Time               6.56
INSTRUM           spect
PROBHD   5 mm CPPBBO BB
PULPROG          zgpg30
TD                65536
SOLVENT           CDCl3
NS                  500
DS                    4
SWH           29761.904 Hz
FIDRES         0.454131 Hz
AQ            1.1010048 sec
RG               192.89
DW               16.800 usec
DE               18.00 usec
TE               298.2 K
D1            2.00000000 sec
D11           0.03000000 sec
TD0                   1

======== CHANNEL f1 ========
SFO1         125.7703637 MHz
NUC1                13C
P1                 9.80 usec
PLW1         57.00000000 W

======== CHANNEL f2 ========
SFO2         500.1320005 MHz
NUC2                1H
CPDPRG[2         waltz16
PCPD2             80.00 usec
PLW2         20.00000000 W
PLW12         0.35778001 W
PLW13         0.22898000 W

F2 - Processing parameters
SI                 32768
SF           125.7577885 MHz
WDW                  EM
SSB       0
LB                1.00 Hz
GB        0
PC                1.40
```



2m

500 MHz, $CDCl_3$

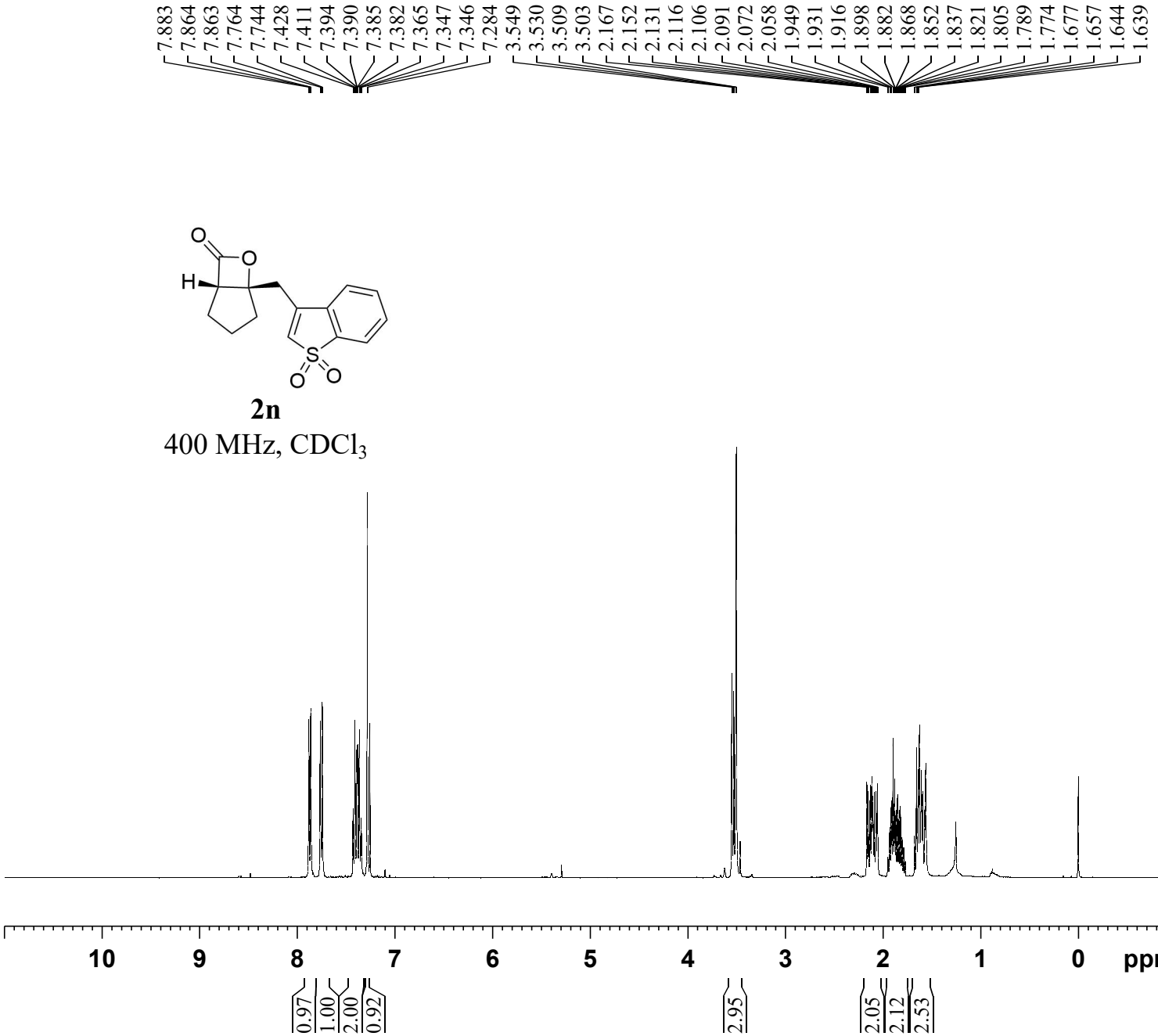
```
Current Data Parameters
NAME        500M-2020xia
EXPNO                 50
PROCNO                 1

F2 - Acquisition Parameters
Date_           20210111
Time                5.00
INSTRUM            spect
PROBHD   5 mm CPPBBO BB
PULPROG             zg30
TD                 65536
SOLVENT            CDCl3
NS                    16
DS                     2
SWH           10000.000 Hz
FIDRES         0.152588 Hz
AQ            3.2767999 sec
RG                 31.72
DW                50.000 usec
DE                 6.50 usec
TE                298.2 K
D1           1.00000000 sec
D11      0 sec
TD0                    1

======== CHANNEL f1 ========
SFO1         500.1330885 MHz
NUC1                   1H
P1                 10.59 usec
PLW1         20.00000000 W

======== CHANNEL f2 ========
SFO2         500.1330885 MHz
NUC2                  off
CPDPRG[2
PCPD2     0 usec
PLW2      0 W
PLW12     0 W
PLW13     0 W

F2 - Processing parameters
SI                 65536
SF          500.1300079 MHz
WDW                   EM
SSB       0
LB                 0.30 Hz
GB        0
PC                 1.00
```



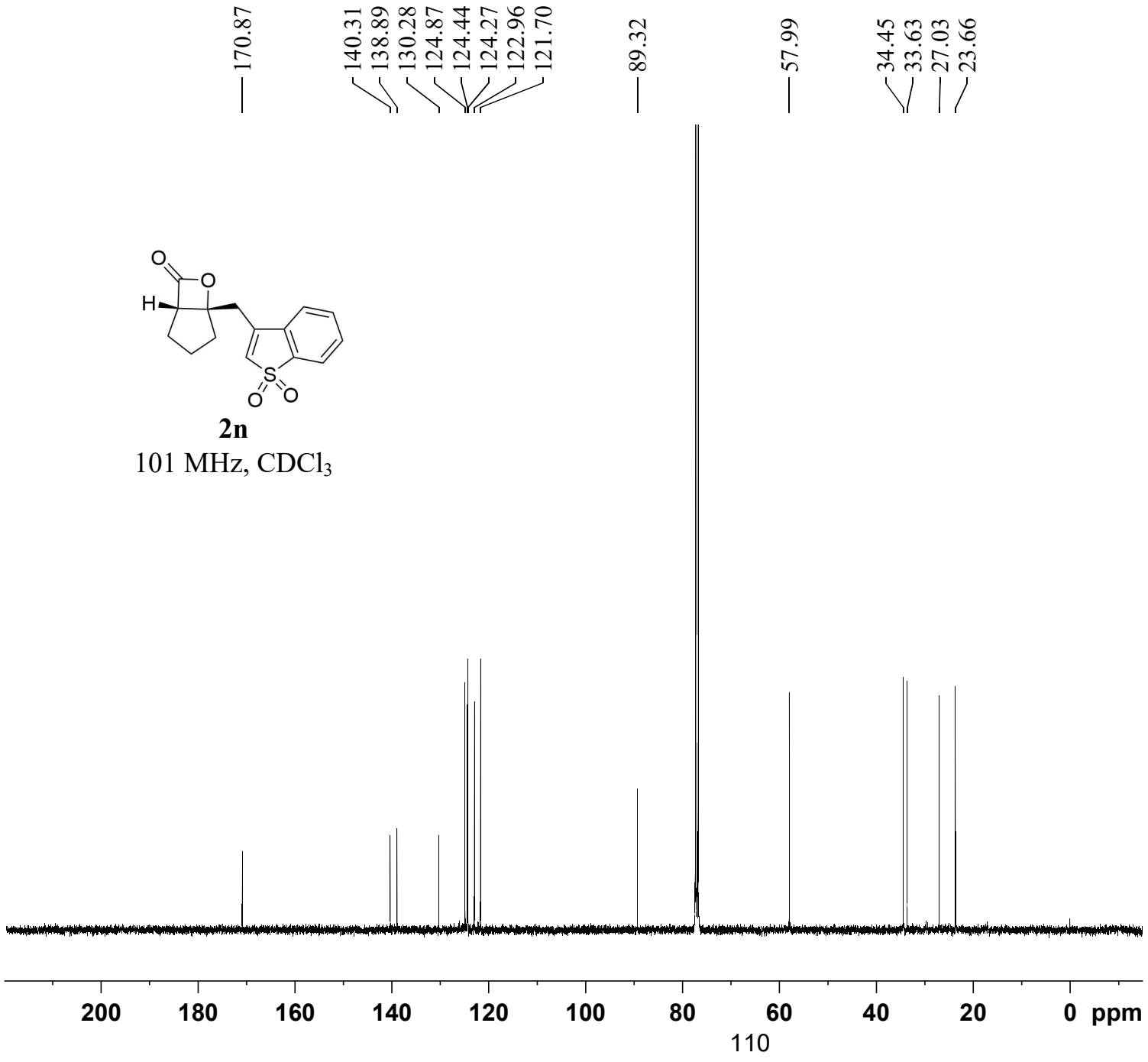
2m

126 MHz, $CDCl_3$

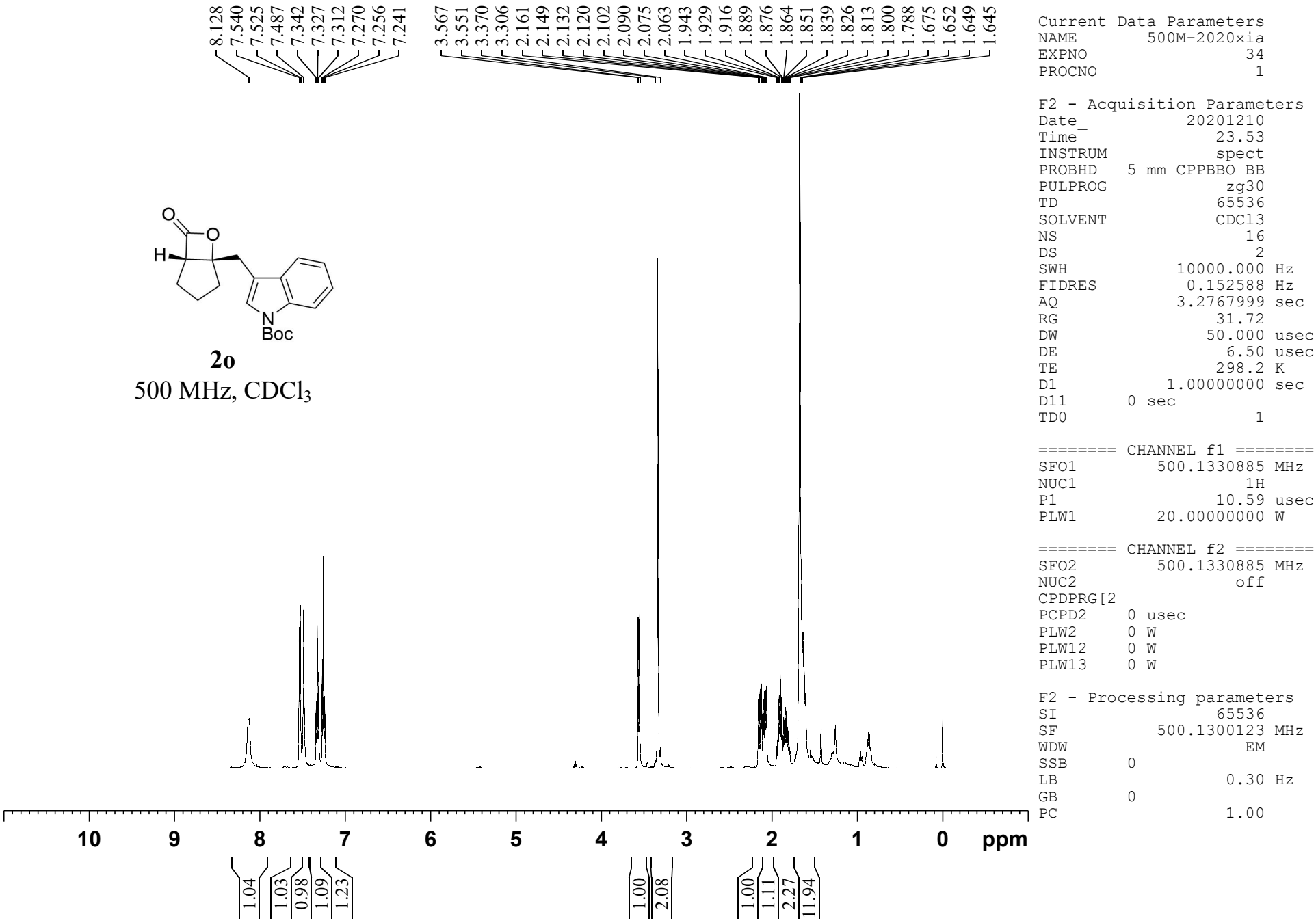
```
Current Data Parameters
NAME        500M-2020xia
EXPNO                 51
PROCNO                 1

F2 - Acquisition Parameters
Date_           20210111
Time                5.27
INSTRUM            spect
PROBHD   5 mm CPPBBO BB
PULPROG          zgpg30
TD                65536
SOLVENT           CDCl3
NS                  500
DS                    4
SWH           29761.904 Hz
FIDRES         0.454131 Hz
AQ            1.1010048 sec
RG               192.89
DW               16.800 usec
DE               18.00 usec
TE               298.2 K
D1           2.00000000 sec
D11          0.03000000 sec
TD0                   1

======== CHANNEL f1 ========
SFO1        125.7703637 MHz
NUC1                13C
P1                 9.80 usec
PLW1        57.00000000 W

======== CHANNEL f2 ========
SFO2        500.1320005 MHz
NUC2                1H
CPDPRG[2         waltz16
PCPD2             80.00 usec
PLW2        20.00000000 W
PLW12        0.35778001 W
PLW13        0.22898000 W

F2 - Processing parameters
SI                 32768
SF          125.7577885 MHz
WDW                   EM
SSB       0
LB                 1.00 Hz
GB        0
PC                 1.40
```



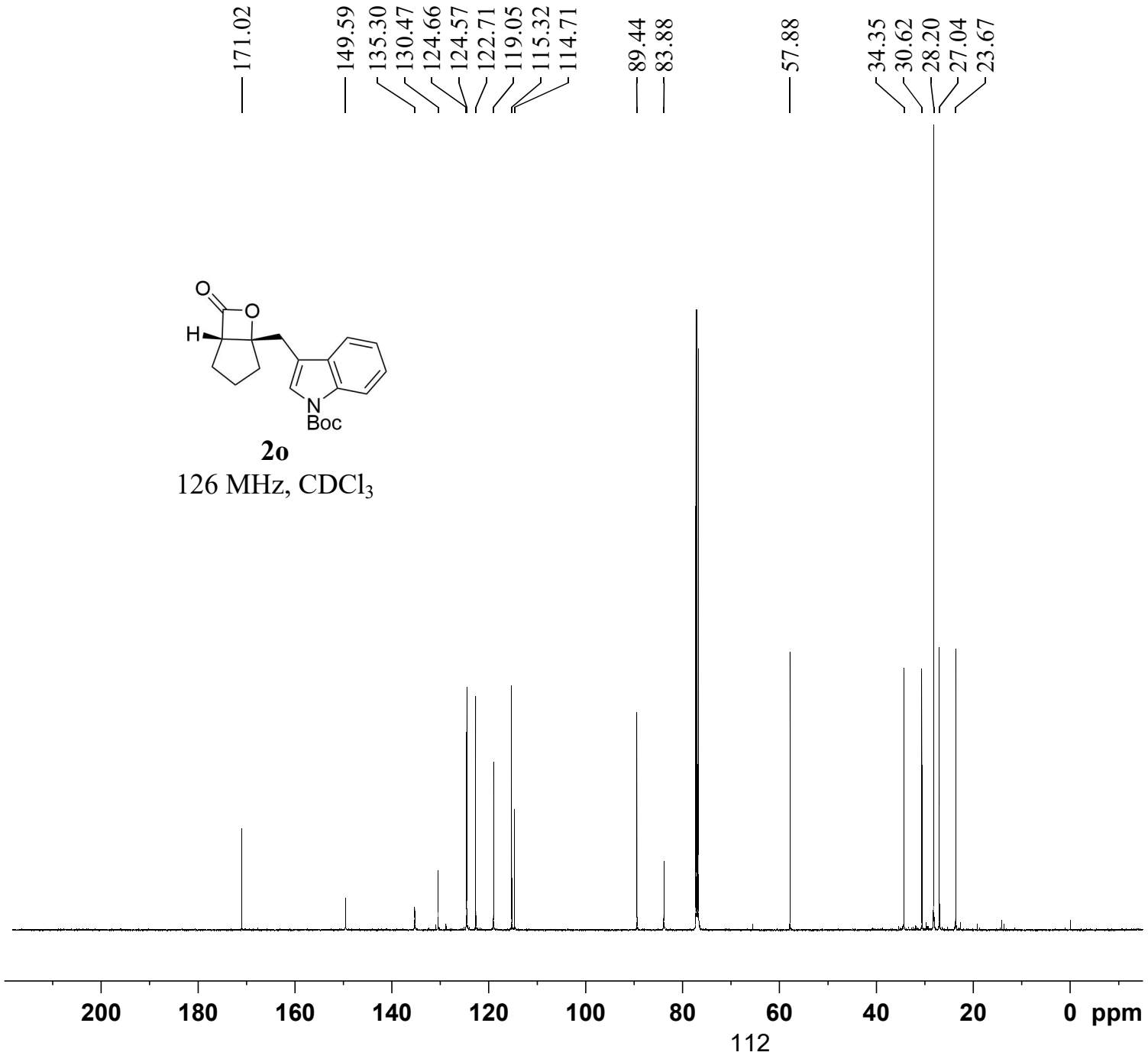

```
Current Data Parameters
NAME                  400xia
EXPNO                     44
PROCNO                     1

F2 - Acquisition Parameters
Date_              20210520
Time                   0.13
INSTRUM               spect
PROBHD    5 mm PABBO BB/
PULPROG                zg30
TD                    32768
SOLVENT               CDCl3
NS                       16
DS                        0
SWH                8012.820 Hz
FIDRES            0.244532 Hz
AQ               2.0447233 sec
RG                  206.33
DW                  62.400 usec
DE                   6.50 usec
TE                  299.4 K
D1               2.00000000 sec
D11       0 sec
TD0                       1

======== CHANNEL f1 ========
SFO1           400.2424716 MHz
NUC1                      1H
P1                    14.30 usec
PLW1           12.00000000 W

======== CHANNEL f2 ========
SFO2           400.2424716 MHz
NUC2                    off
CPDPRG[2
PCPD2     0 usec
PLW2      0 W
PLW12     0 W
PLW13     0 W

F2 - Processing parameters
SI                     65536
SF           400.2400113 MHz
WDW                       EM
SSB       0
LB                    0.30 Hz
GB        0
PC                    1.00
```




```
Current Data Parameters
NAME                 400xia
EXPNO                    45
PROCNO                    1

F2 - Acquisition Parameters
Date_              20210520
Time                   1.12
INSTRUM               spect
PROBHD   5 mm PABBO BB/
PULPROG              zgpg30
TD                    65536
SOLVENT               CDCl3
NS                     1024
DS                        4
SWH              24038.461 Hz
FIDRES            0.366798 Hz
AQ               1.3631488 sec
RG                   206.33
DW                 20.800 usec
DE                  6.50 usec
TE                  300.4 K
D1              2.00000000 sec
D11             0.03000000 sec
TD0                       1

======== CHANNEL f1 ========
SFO1           100.6504916 MHz
NUC1                    13C
P1                  10.00 usec
PLW1           54.00000000 W

======== CHANNEL f2 ========
SFO2           400.2416010 MHz
NUC2                    1H
CPDPRG[2           waltz16
PCPD2               90.00 usec
PLW2           12.00000000 W
PLW12           0.30294999 W
PLW13           0.24539000 W

F2 - Processing parameters
SI                      32768
SF             100.6404280 MHz
WDW                        EM
SSB         0
LB                   1.00 Hz
GB          0
PC                   1.40
```



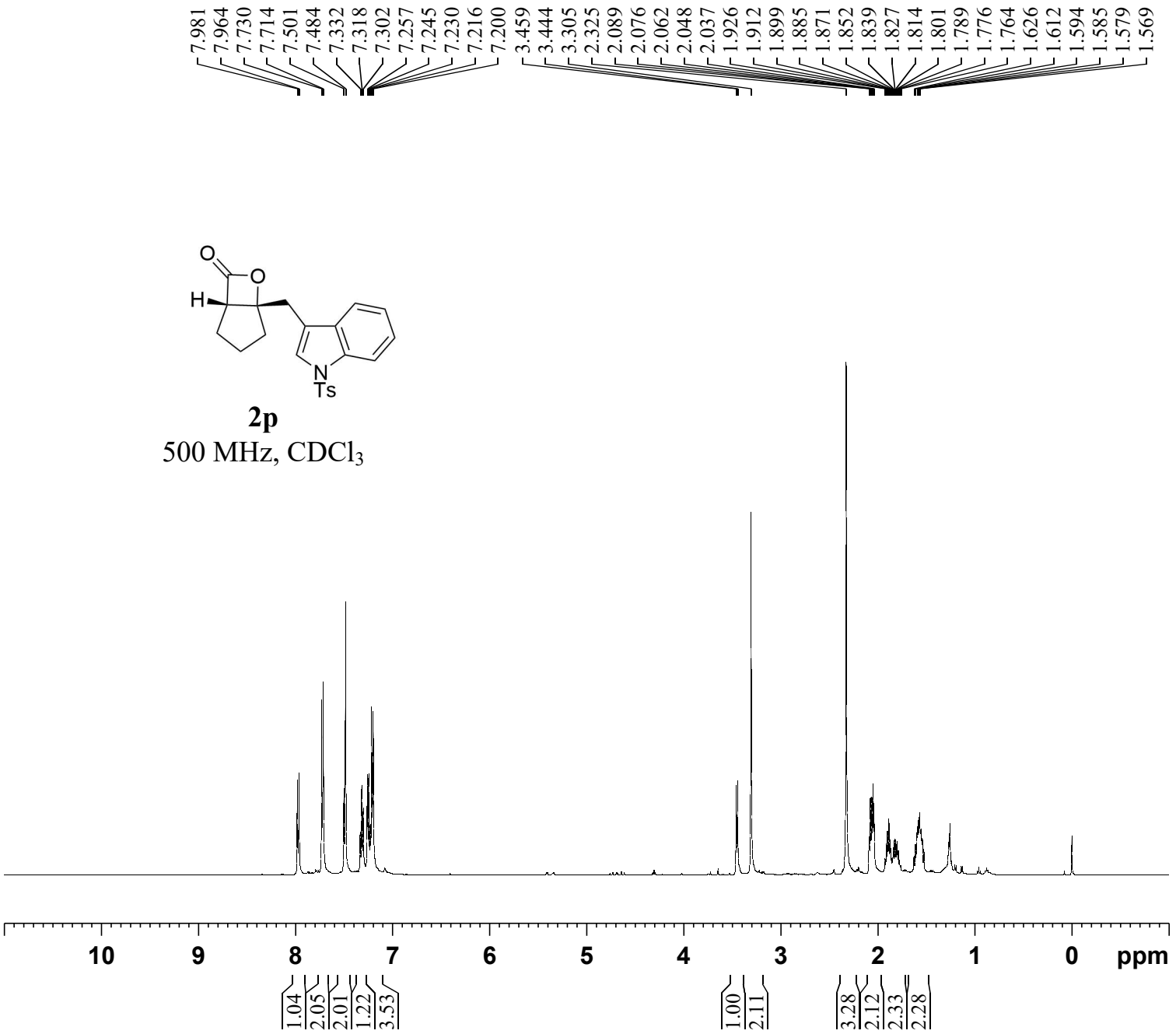

```
Current Data Parameters
NAME        500M-2020xia
EXPNO                 34
PROCNO                 1

F2 - Acquisition Parameters
Date_           20201210
Time               23.53
INSTRUM            spect
PROBHD   5 mm CPPBBO BB
PULPROG             zg30
TD                 65536
SOLVENT            CDCl3
NS                    16
DS                     2
SWH          10000.000 Hz
FIDRES        0.152588 Hz
AQ           3.2767999 sec
RG                 31.72
DW                50.000 usec
DE                 6.50 usec
TE                298.2 K
D1           1.00000000 sec
D11      0 sec
TD0                    1

======== CHANNEL f1 ========
SFO1        500.1330885 MHz
NUC1                   1H
P1                 10.59 usec
PLW1        20.00000000 W

======== CHANNEL f2 ========
SFO2        500.1330885 MHz
NUC2                  off
CPDPRG[2
PCPD2     0 usec
PLW2      0 W
PLW12     0 W
PLW13     0 W

F2 - Processing parameters
SI                 65536
SF          500.1300123 MHz
WDW                   EM
SSB       0
LB                 0.30 Hz
GB        0
PC                 1.00
```



2o

126 MHz, $CDCl_3$

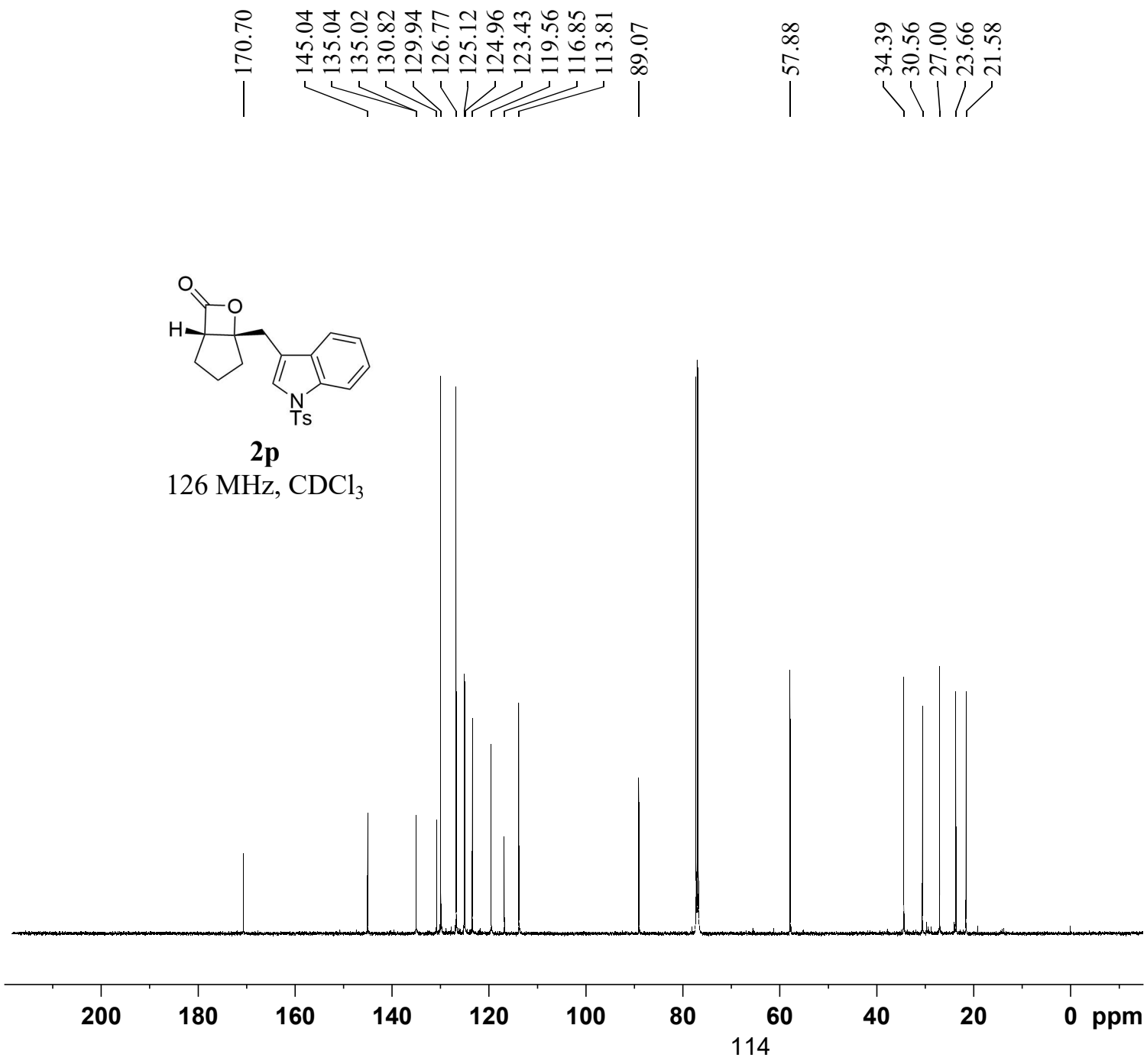
```
Current Data Parameters
NAME        500M-2020xia
EXPNO                 35
PROCNO                 1

F2 - Acquisition Parameters
Date_           20201211
Time                0.48
INSTRUM            spect
PROBHD   5 mm CPPBBO BB
PULPROG          zgpg30
TD                65536
SOLVENT           CDCl3
NS                 1024
DS                    4
SWH           29761.904 Hz
FIDRES         0.454131 Hz
AQ            1.1010048 sec
RG               192.89
DW               16.800 usec
DE               18.00 usec
TE               298.2 K
D1           2.00000000 sec
D11          0.03000000 sec
TD0                   1

======== CHANNEL f1 ========
SFO1        125.7703637 MHz
NUC1                13C
P1                 9.80 usec
PLW1        57.00000000 W

======== CHANNEL f2 ========
SFO2        500.1320005 MHz
NUC2                1H
CPDPRG[2         waltz16
PCPD2             80.00 usec
PLW2        20.00000000 W
PLW12        0.35778001 W
PLW13        0.22898000 W

F2 - Processing parameters
SI                 32768
SF          125.7577907 MHz
WDW                   EM
SSB       0
LB                 1.00 Hz
GB        0
PC                 1.40
```



2p

500 MHz, $CDCl_3$

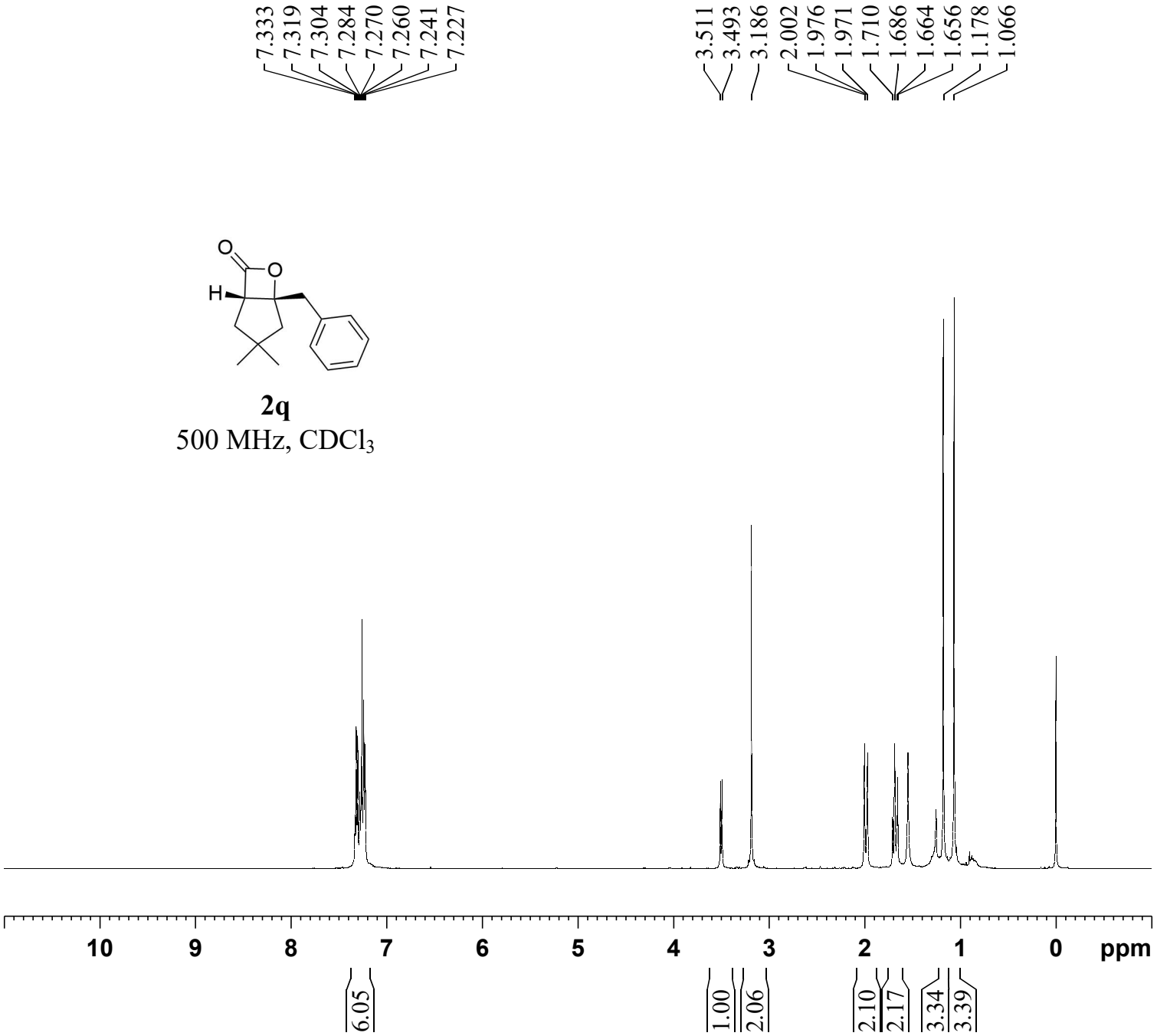
```
Current Data Parameters
NAME        500M-2020xia
EXPNO                 44
PROCNO                 1

F2 - Acquisition Parameters
Date_           20210111
Time                3.05
INSTRUM            spect
PROBHD   5 mm CPPBBO BB
PULPROG             zg30
TD                 65536
SOLVENT            CDCl3
NS                    16
DS                     2
SWH           10000.000 Hz
FIDRES         0.152588 Hz
AQ            3.2767999 sec
RG                 31.72
DW                50.000 usec
DE                 6.50 usec
TE                298.2 K
D1           1.00000000 sec
D11      0 sec
TD0                    1

======== CHANNEL f1 ========
SFO1         500.1330885 MHz
NUC1                   1H
P1                 10.59 usec
PLW1         20.00000000 W

======== CHANNEL f2 ========
SFO2         500.1330885 MHz
NUC2                  off
CPDPRG[2
PCPD2     0 usec
PLW2      0 W
PLW12     0 W
PLW13     0 W

F2 - Processing parameters
SI                 65536
SF           500.1300136 MHz
WDW                   EM
SSB       0
LB                 0.30 Hz
GB        0
PC                 1.00
```



2p

126 MHz, $CDCl_3$

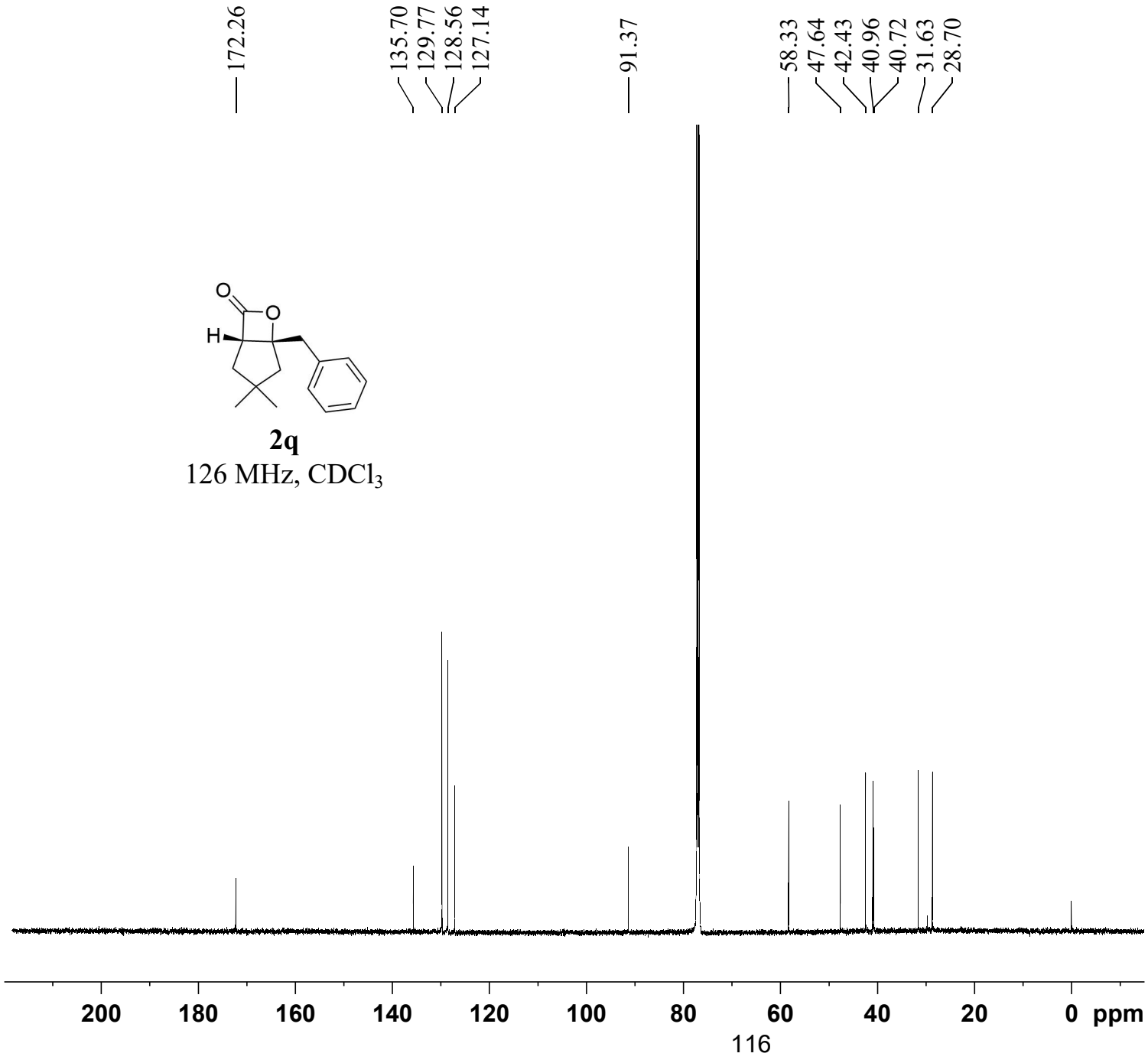
```
Current Data Parameters
NAME        500M-2020xia
EXPNO                 45
PROCNO                 1

F2 - Acquisition Parameters
Date_           20210111
Time                3.27
INSTRUM            spect
PROBHD   5 mm CPPBBO BB
PULPROG          zgpg30
TD                65536
SOLVENT           CDCl3
NS                  400
DS                    4
SWH           29761.904 Hz
FIDRES         0.454131 Hz
AQ            1.1010048 sec
RG               192.89
DW               16.800 usec
DE               18.00 usec
TE               298.2 K
D1           2.00000000 sec
D11          0.03000000 sec
TD0                   1

======== CHANNEL f1 ========
SFO1        125.7703637 MHz
NUC1                13C
P1                 9.80 usec
PLW1        57.00000000 W

======== CHANNEL f2 ========
SFO2        500.1320005 MHz
NUC2                1H
CPDPRG[2         waltz16
PCPD2             80.00 usec
PLW2        20.00000000 W
PLW12        0.35778001 W
PLW13        0.22898000 W

F2 - Processing parameters
SI                 32768
SF          125.7577885 MHz
WDW                   EM
SSB       0
LB                 1.00 Hz
GB        0
PC                 1.40
```



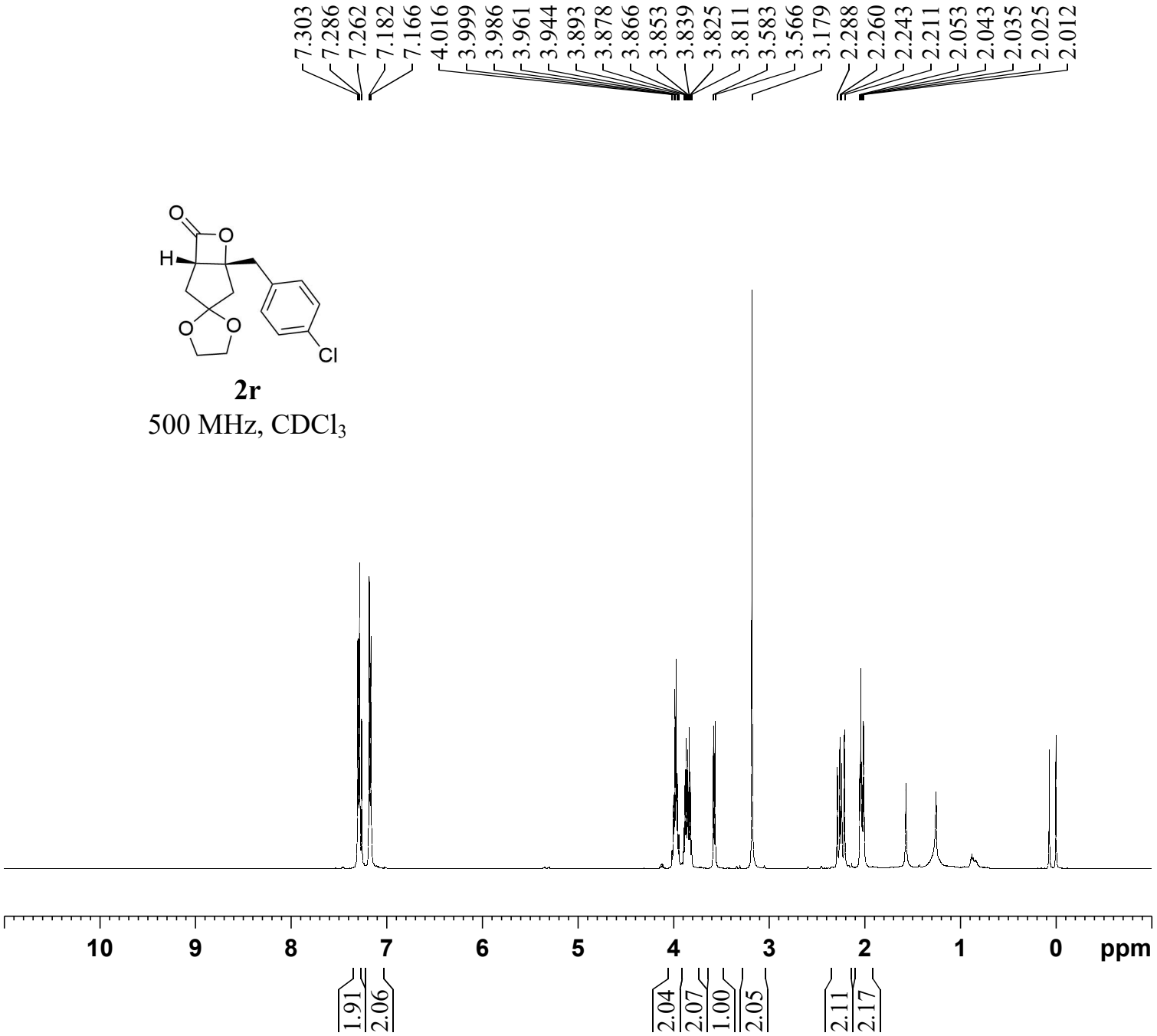
2q

500 MHz, $CDCl_3$

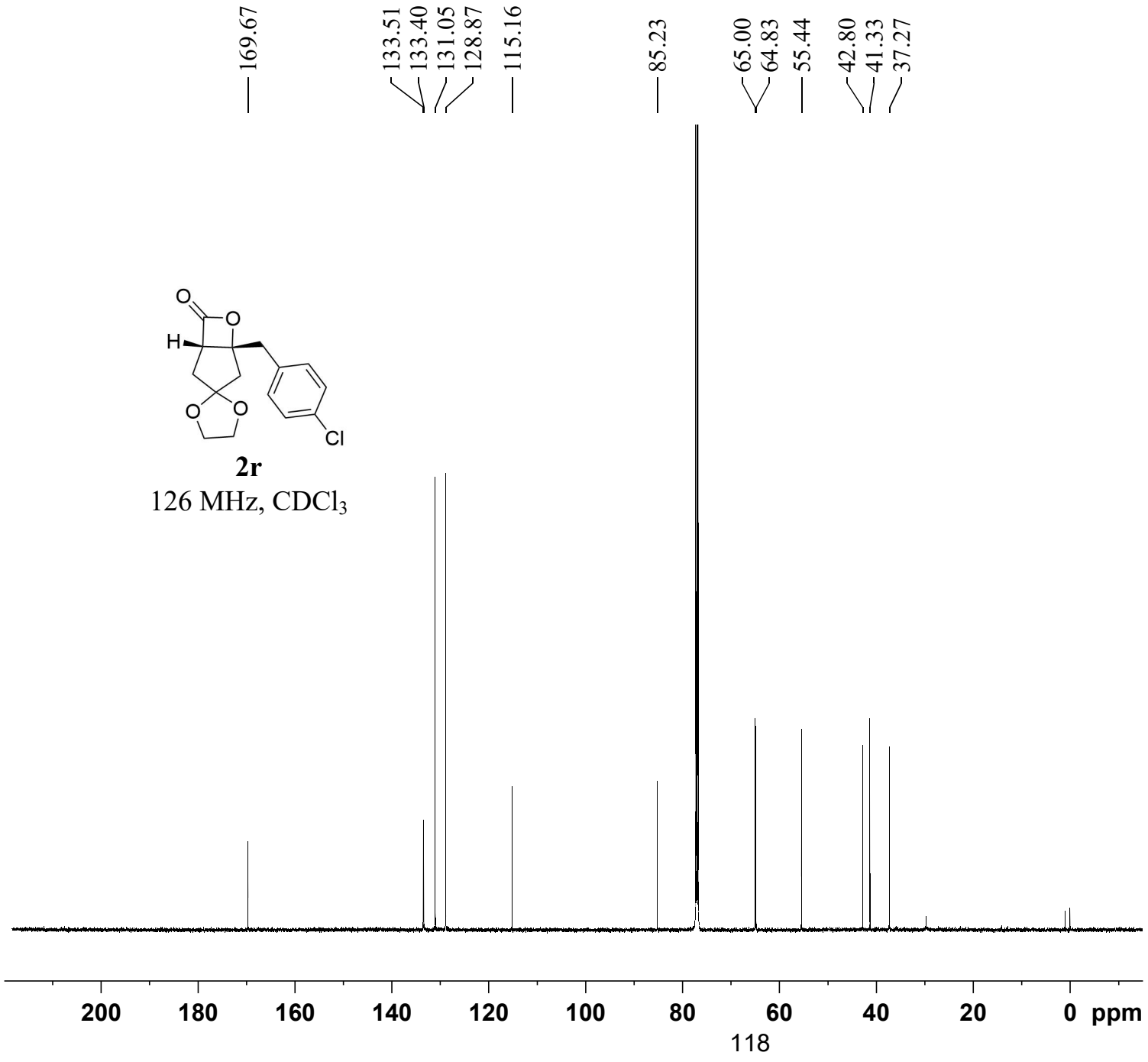
```
Current Data Parameters
NAME        500M-2020xia
EXPNO                118
PROCNO                 1

F2 - Acquisition Parameters
Date_           20210516
Time               14.42
INSTRUM            spect
PROBHD   5 mm CPPBBO BB
PULPROG             zg30
TD                 65536
SOLVENT            CDCl3
NS                    16
DS                     2
SWH           10000.000 Hz
FIDRES         0.152588 Hz
AQ            3.2767999 sec
RG                 55.37
DW                50.000 usec
DE                 6.50 usec
TE                298.2 K
D1           1.00000000 sec
D11      0 sec
TD0                    1

======== CHANNEL f1 ========
SFO1         500.1330885 MHz
NUC1                   1H
P1                 10.59 usec
PLW1         20.00000000 W

======== CHANNEL f2 ========
SFO2         500.1330885 MHz
NUC2                  off
CPDPRG[2
PCPD2     0 usec
PLW2      0 W
PLW12     0 W
PLW13     0 W

F2 - Processing parameters
SI                 65536
SF           500.1300124 MHz
WDW                   EM
SSB       0
LB                 0.30 Hz
GB        0
PC                 1.00
```




```
Current Data Parameters
NAME        500M-2020xia
EXPNO                119
PROCNO                 1

F2 - Acquisition Parameters
Date_           20210516
Time              16.24
INSTRUM           spect
PROBHD   5 mm CPPBBO BB
PULPROG          zgpg30
TD                65536
SOLVENT           CDCl3
NS                 1925
DS                    4
SWH           29761.904 Hz
FIDRES         0.454131 Hz
AQ            1.1010048 sec
RG               192.89
DW               16.800 usec
DE               18.00 usec
TE               298.2 K
D1           2.00000000 sec
D11          0.03000000 sec
TD0                   1

======== CHANNEL f1 ========
SFO1        125.7703637 MHz
NUC1                13C
P1                 9.80 usec
PLW1        57.00000000 W

======== CHANNEL f2 ========
SFO2        500.1320005 MHz
NUC2                1H
CPDPRG[2         waltz16
PCPD2             80.00 usec
PLW2        20.00000000 W
PLW12        0.35778001 W
PLW13        0.22898000 W

F2 - Processing parameters
SI                 32768
SF          125.7577889 MHz
WDW                   EM
SSB       0
LB                 1.00 Hz
GB        0
PC                 1.40
```



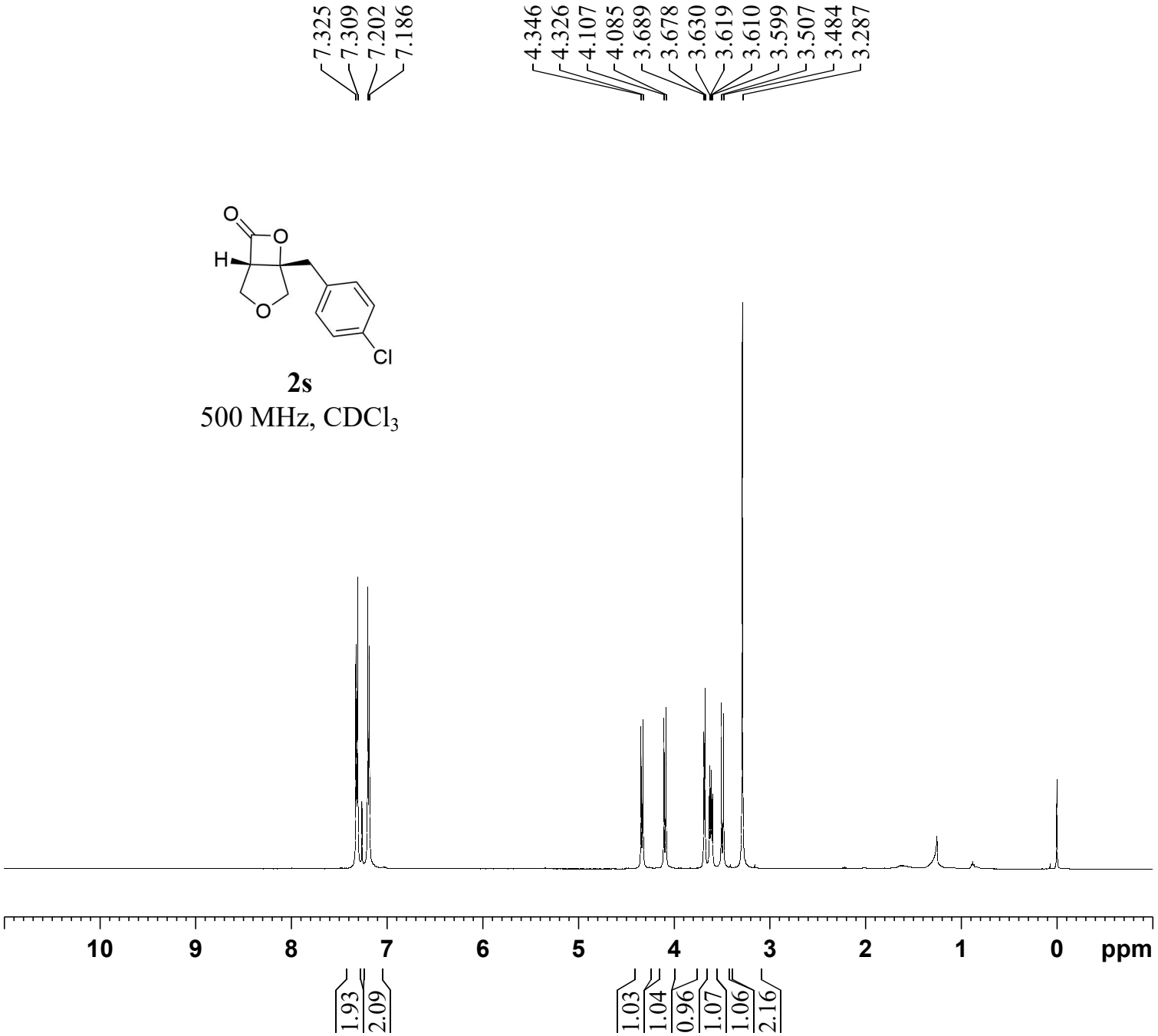

```
Current Data Parameters
NAME        500M-2020xia
EXPNO                116
PROCNO                 1

F2 - Acquisition Parameters
Date_           20210516
Time               13.43
INSTRUM            spect
PROBHD   5 mm CPPBBO BB
PULPROG             zg30
TD                 65536
SOLVENT            CDCl3
NS                    16
DS                     2
SWH           10000.000 Hz
FIDRES         0.152588 Hz
AQ            3.2767999 sec
RG                 55.37
DW                50.000 usec
DE                 6.50 usec
TE                298.2 K
D1           1.00000000 sec
D11      0 sec
TD0                    1

======== CHANNEL f1 ========
SFO1         500.1330885 MHz
NUC1                   1H
P1                 10.59 usec
PLW1         20.00000000 W

======== CHANNEL f2 ========
SFO2         500.1330885 MHz
NUC2                  off
CPDPRG[2
PCPD2     0 usec
PLW2      0 W
PLW12     0 W
PLW13     0 W

F2 - Processing parameters
SI                 65536
SF         500.1300112 MHz
WDW                   EM
SSB       0
LB                 0.30 Hz
GB        0
PC                 1.00
```



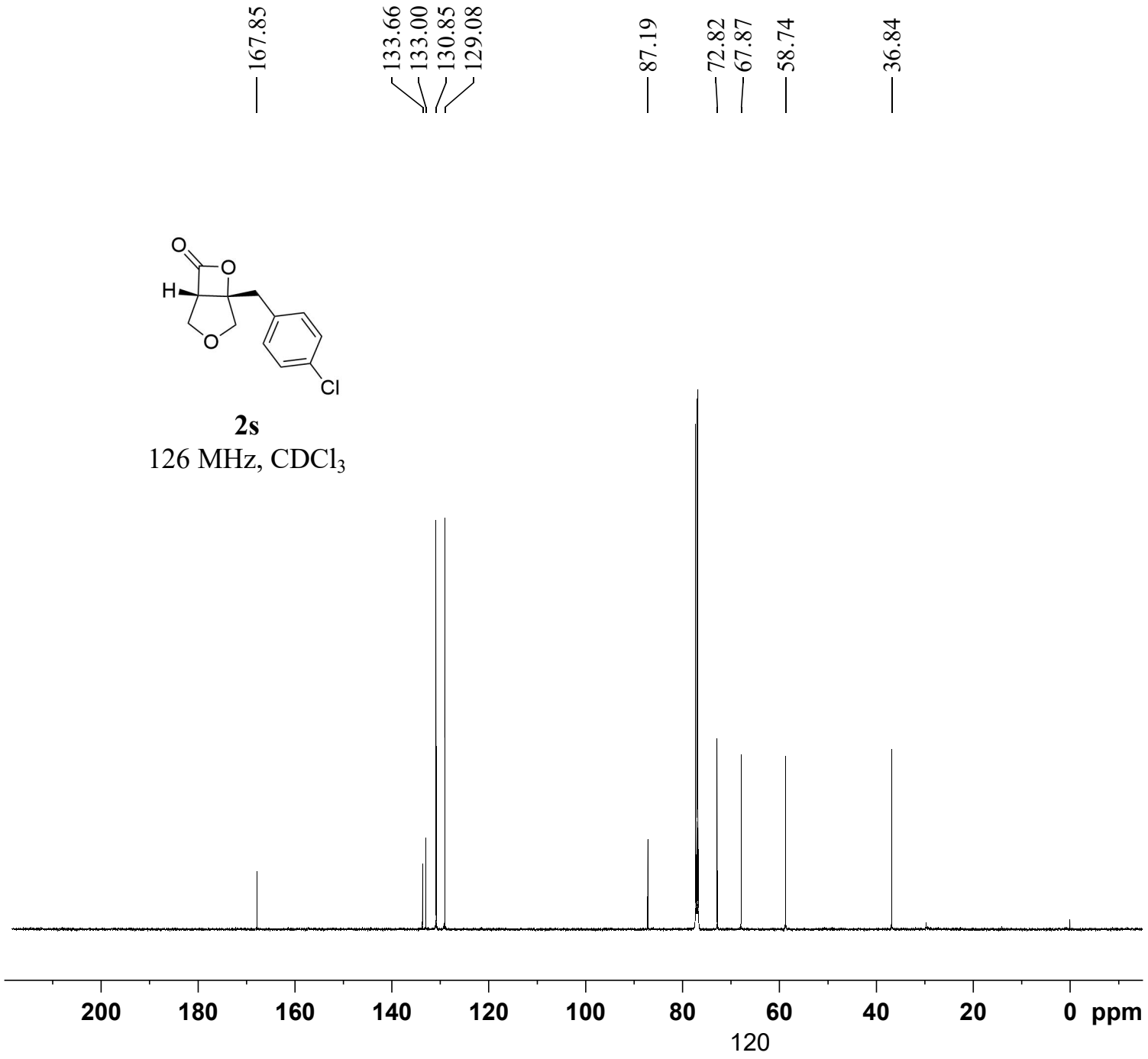
2r

126 MHz, $CDCl_3$

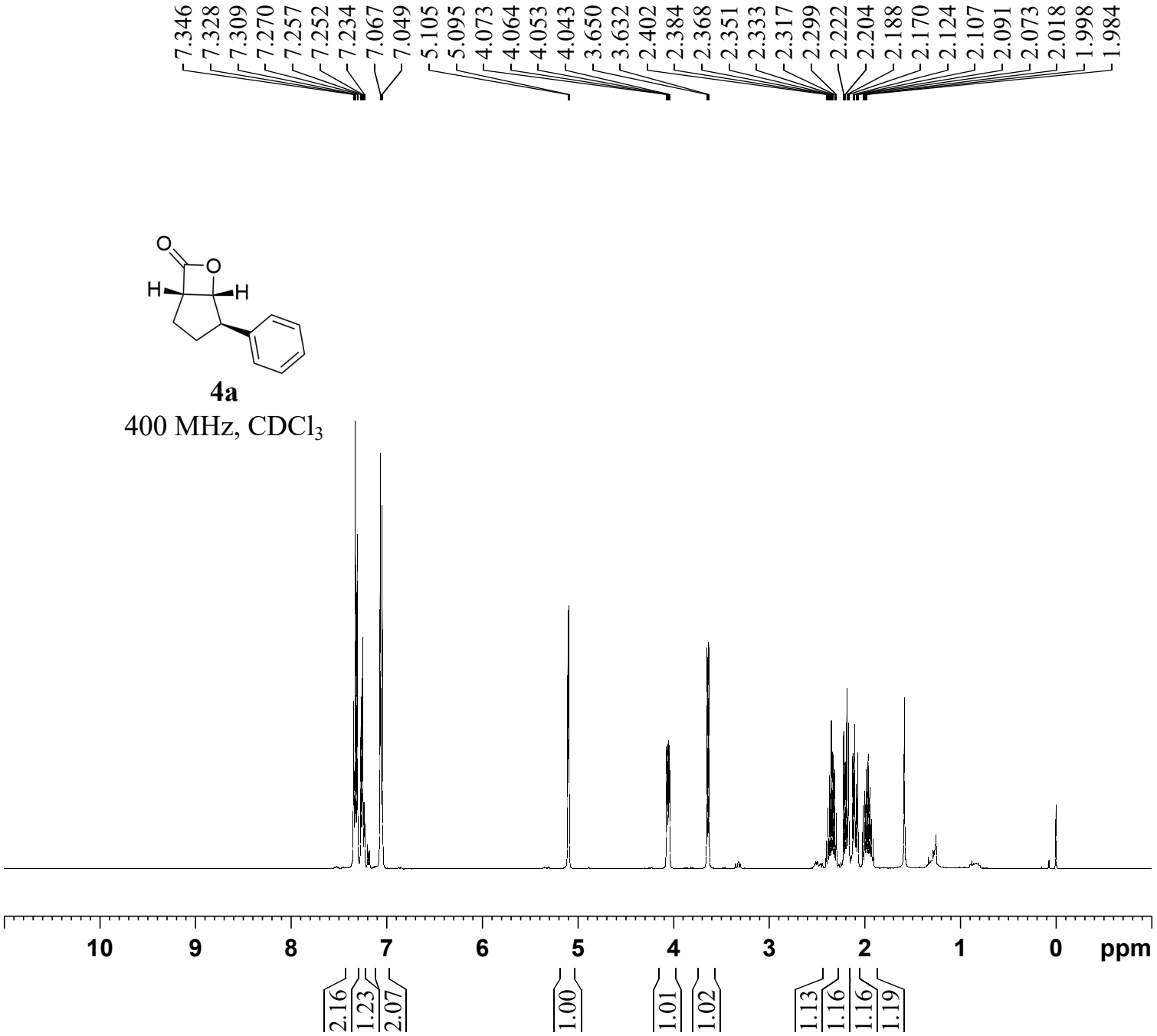
```
Current Data Parameters
NAME        500M-2020xia
EXPNO                117
PROCNO                 1

F2 - Acquisition Parameters
Date_           20210516
Time              14.38
INSTRUM           spect
PROBHD   5 mm CPPBBO BB
PULPROG          zgpg30
TD                65536
SOLVENT           CDCl3
NS                 1024
DS                    4
SWH           29761.904 Hz
FIDRES         0.454131 Hz
AQ            1.1010048 sec
RG               192.89
DW               16.800 usec
DE               18.00 usec
TE               298.2 K
D1           2.00000000 sec
D11          0.03000000 sec
TD0                   1

======== CHANNEL f1 ========
SFO1        125.7703637 MHz
NUC1                13C
P1                 9.80 usec
PLW1        57.00000000 W

======== CHANNEL f2 ========
SFO2        500.1320005 MHz
NUC2                1H
CPDPRG[2         waltz16
PCPD2             80.00 usec
PLW2        20.00000000 W
PLW12        0.35778001 W
PLW13        0.22898000 W

F2 - Processing parameters
SI                 32768
SF          125.7577897 MHz
WDW                   EM
SSB       0
LB                 1.00 Hz
GB        0
PC                 1.40
```



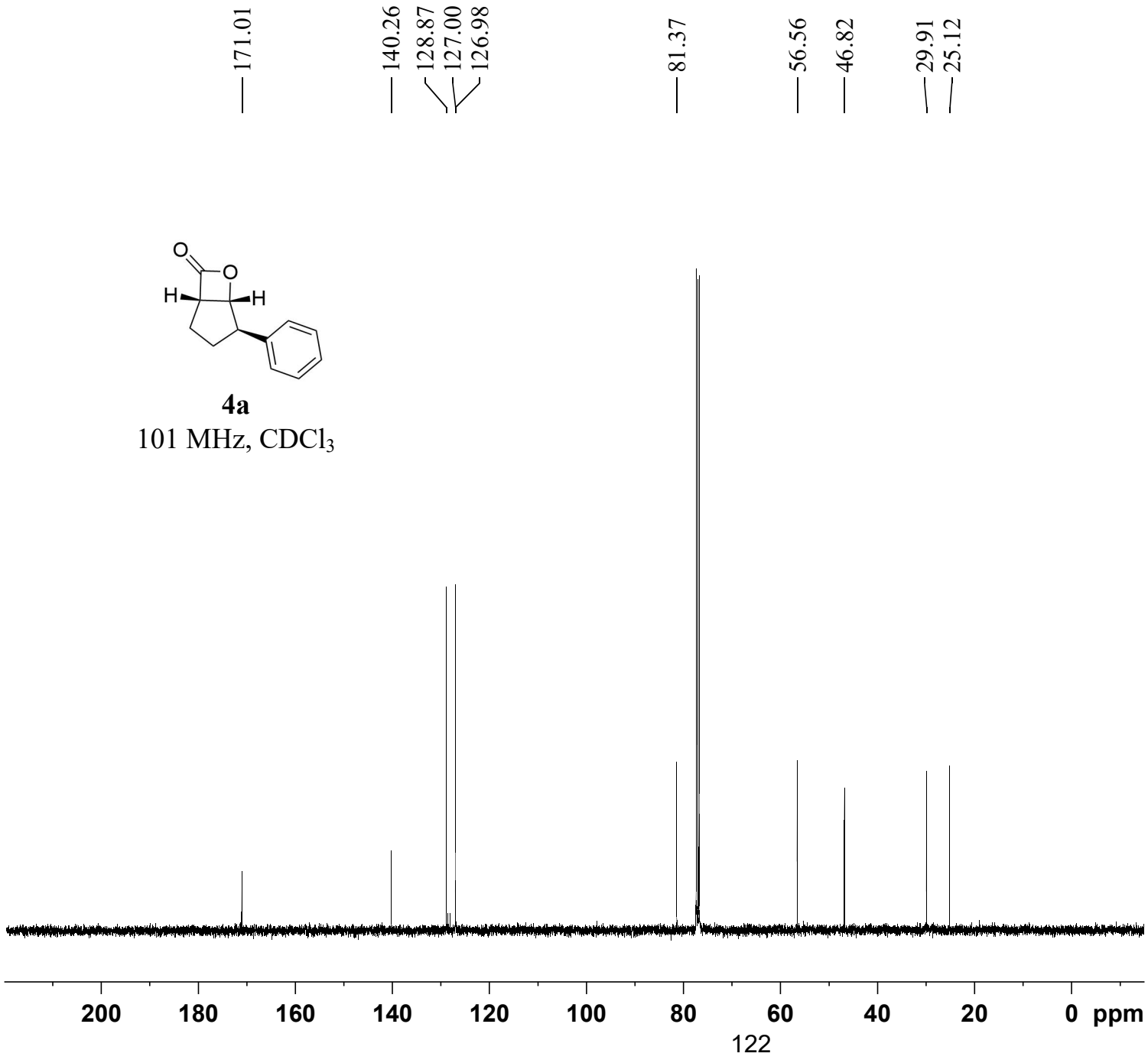

```
Current Data Parameters
NAME        500M-2020xia
EXPNO                122
PROCNO                 1

F2 - Acquisition Parameters
Date_           20210516
Time              17.12
INSTRUM           spect
PROBHD   5 mm CPPBBO BB
PULPROG             zg30
TD                65536
SOLVENT           CDCl3
NS                   16
DS                    2
SWH           10000.000 Hz
FIDRES         0.152588 Hz
AQ            3.2767999 sec
RG                49.27
DW               50.000 usec
DE                6.50 usec
TE               298.2 K
D1          1.00000000 sec
D11      0 sec
TD0                   1

======== CHANNEL f1 ========
SFO1         500.1330885 MHz
NUC1                 1H
P1                10.59 usec
PLW1         20.00000000 W

======== CHANNEL f2 ========
SFO2         500.1330885 MHz
NUC2                 off
CPDPRG[2
PCPD2     0 usec
PLW2      0 W
PLW12     0 W
PLW13     0 W

F2 - Processing parameters
SI                  65536
SF           500.1300109 MHz
WDW                    EM
SSB       0
LB                  0.30 Hz
GB        0
PC                  1.00
```




```
Current Data Parameters
NAME        500M-2020xia
EXPNO                123
PROCNO                 1

F2 - Acquisition Parameters
Date_           20210516
Time              17.40
INSTRUM           spect
PROBHD   5 mm CPPBBO BB
PULPROG          zgpg30
TD                65536
SOLVENT           CDCl3
NS                  512
DS                    4
SWH           29761.904 Hz
FIDRES         0.454131 Hz
AQ            1.1010048 sec
RG               192.89
DW               16.800 usec
DE               18.00 usec
TE               298.2 K
D1           2.00000000 sec
D11          0.03000000 sec
TD0                   1

======== CHANNEL f1 ========
SFO1        125.7703637 MHz
NUC1                13C
P1                 9.80 usec
PLW1        57.00000000 W

======== CHANNEL f2 ========
SFO2        500.1320005 MHz
NUC2                1H
CPDPRG[2         waltz16
PCPD2             80.00 usec
PLW2        20.00000000 W
PLW12        0.35778001 W
PLW13        0.22898000 W

F2 - Processing parameters
SI                 32768
SF          125.7577902 MHz
WDW                  EM
SSB       0
LB                1.00 Hz
GB        0
PC                1.40
```



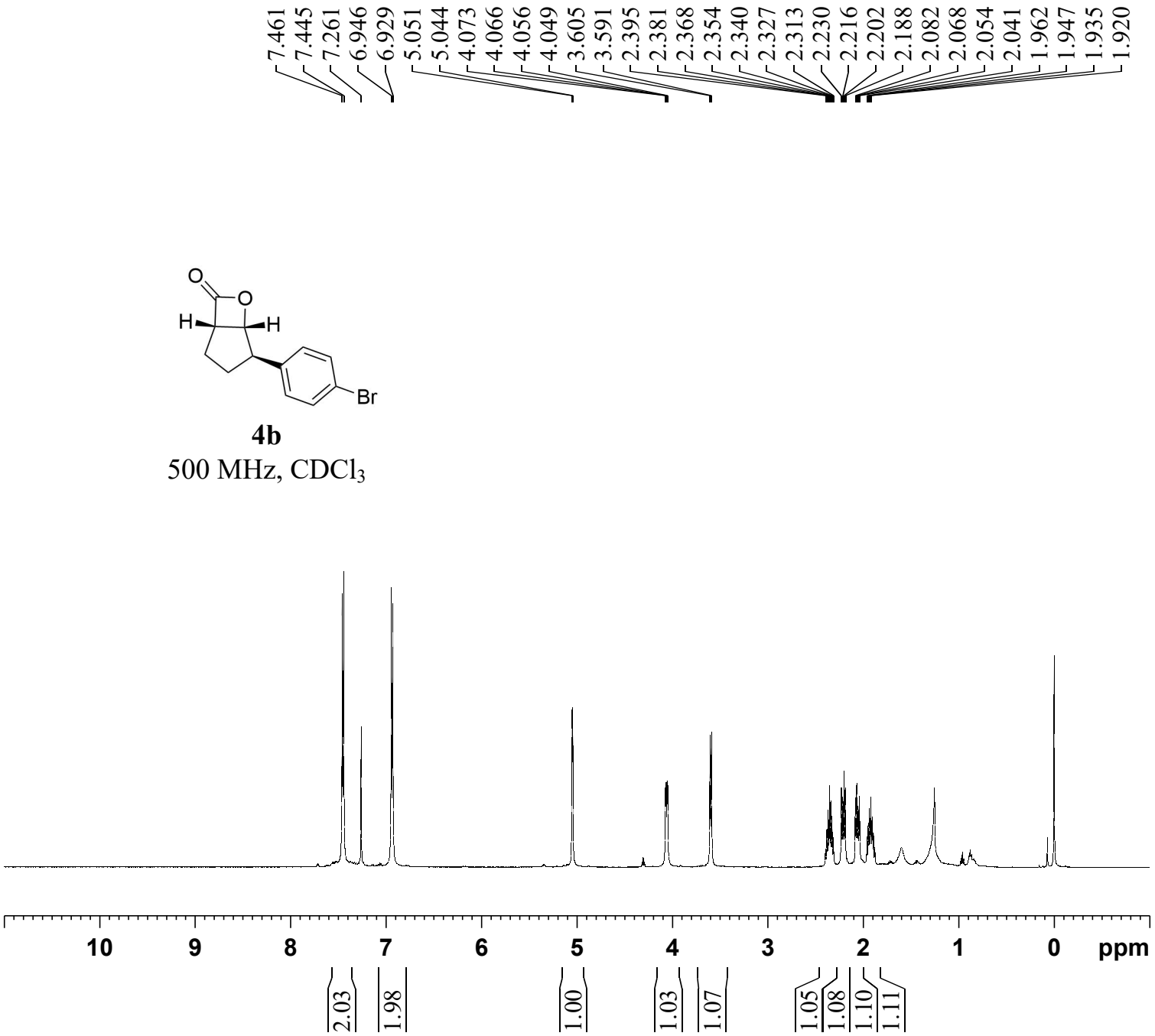

```
Current Data Parameters
NAME                  400xia
EXPNO                     48
PROCNO                     1

F2 - Acquisition Parameters
Date_               20210602
Time                   15.04
INSTRUM                spect
PROBHD    5 mm PABBO BB/
PULPROG                zg30
TD                    32768
SOLVENT               CDCl3
NS                       16
DS                        0
SWH                8012.820 Hz
FIDRES             0.244532 Hz
AQ                2.0447233 sec
RG                   162.77
DW                   62.400 usec
DE                    6.50 usec
TE                   299.3 K
D1                2.00000000 sec
D11       0 sec
TD0                        1

======== CHANNEL f1 ========
SFO1            400.2424716 MHz
NUC1                       1H
P1                    14.30 usec
PLW1           12.00000000 W

======== CHANNEL f2 ========
SFO2            400.2424716 MHz
NUC2                     off
CPDPRG[2
PCPD2     0 usec
PLW2      0 W
PLW12     0 W
PLW13     0 W

F2 - Processing parameters
SI                     65536
SF              400.2400111 MHz
WDW                       EM
SSB       0
LB                    0.30 Hz
GB        0
PC                    1.00
```



4a

101 MHz, $CDCl_3$

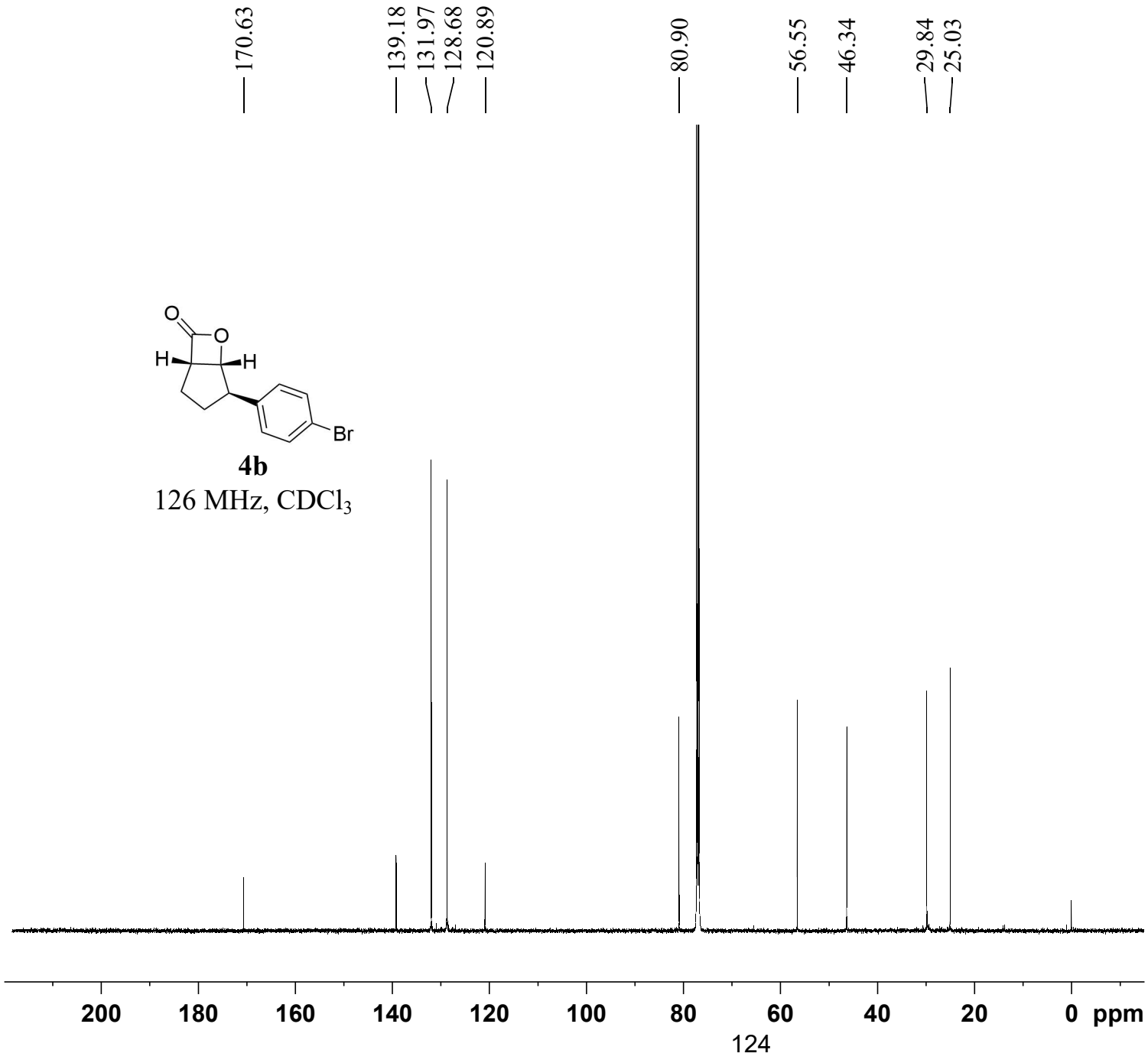
```
Current Data Parameters
NAME                 400xia
EXPNO                    49
PROCNO                    1

F2 - Acquisition Parameters
Date_              20210602
Time                  15.19
INSTRUM               spect
PROBHD   5 mm PABBO BB/
PULPROG              zgpg30
TD                    65536
SOLVENT               CDCl3
NS                      256
DS                        4
SWH               24038.461 Hz
FIDRES             0.366798 Hz
AQ                1.3631488 sec
RG                   206.33
DW                   20.800 usec
DE                    6.50 usec
TE                   300.1 K
D1               2.00000000 sec
D11              0.03000000 sec
TD0                       1

======== CHANNEL f1 ========
SFO1            100.6504916 MHz
NUC1                    13C
P1                    10.00 usec
PLW1            54.00000000 W

======== CHANNEL f2 ========
SFO2            400.2416010 MHz
NUC2                    1H
CPDPRG[2             waltz16
PCPD2                 90.00 usec
PLW2            12.00000000 W
PLW12            0.30294999 W
PLW13            0.24539000 W

F2 - Processing parameters
SI                      32768
SF              100.6404280 MHz
WDW                        EM
SSB          0
LB                     1.00 Hz
GB           0
PC                     1.40
```



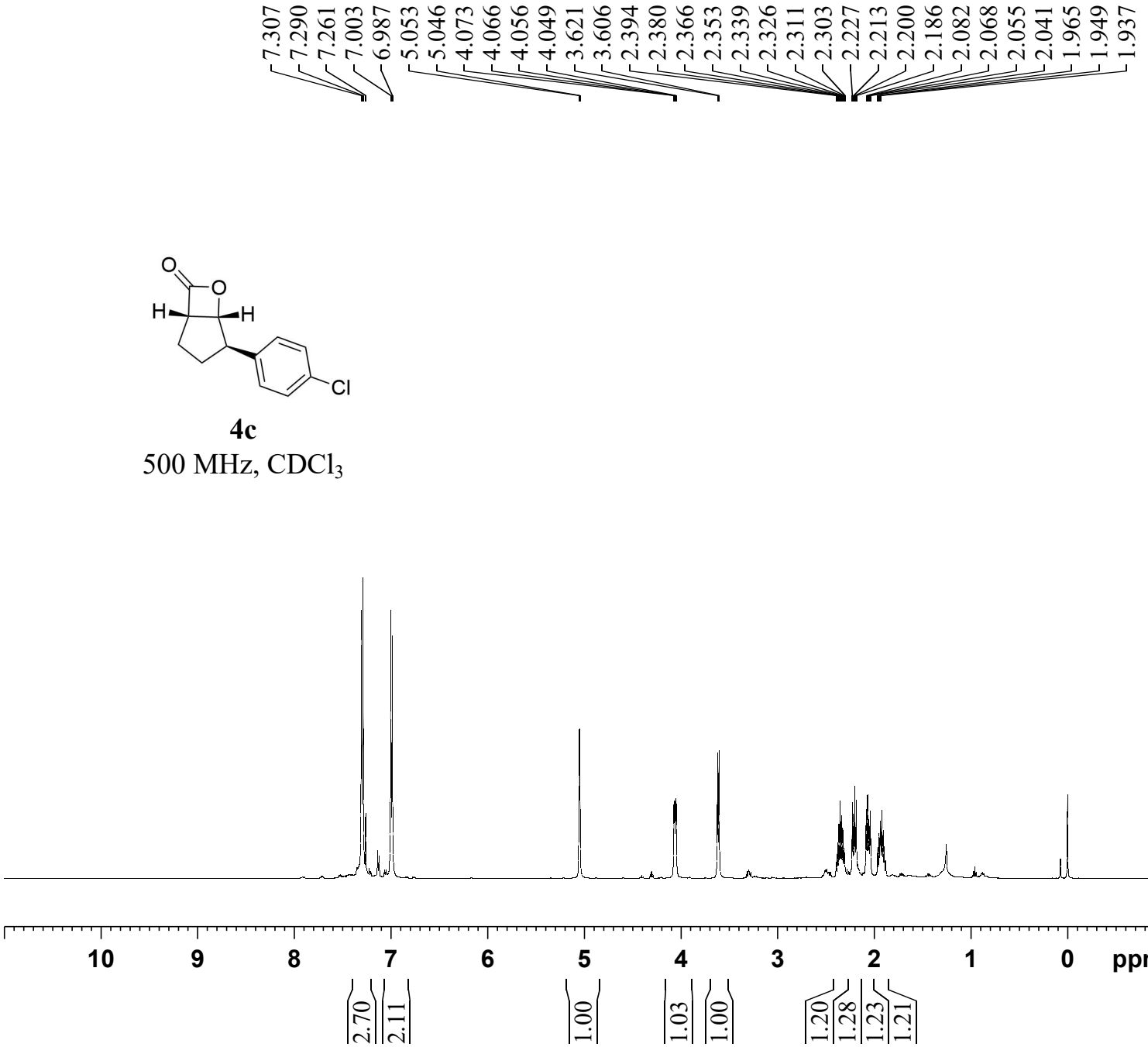
4b

500 MHz, $CDCl_3$

```
Current Data Parameters
NAME        500M-2020xia
EXPNO                110
PROCNO                 1

F2 - Acquisition Parameters
Date_           20210424
Time                8.59
INSTRUM            spect
PROBHD   5 mm CPPBBO BB
PULPROG             zg30
TD                 65536
SOLVENT            CDCl3
NS                    16
DS                     2
SWH           10000.000 Hz
FIDRES         0.152588 Hz
AQ            3.2767999 sec
RG                 62.06
DW                50.000 usec
DE                 6.50 usec
TE                298.2 K
D1           1.00000000 sec
D11      0 sec
TD0                    1

======== CHANNEL f1 ========
SFO1         500.1330885 MHz
NUC1                   1H
P1                 10.59 usec
PLW1         20.00000000 W

======== CHANNEL f2 ========
SFO2         500.1330885 MHz
NUC2                  off
CPDPRG[2
PCPD2     0 usec
PLW2      0 W
PLW12     0 W
PLW13     0 W

F2 - Processing parameters
SI                 65536
SF           500.1300118 MHz
WDW                   EM
SSB       0
LB                 0.30 Hz
GB        0
PC                 1.00
```



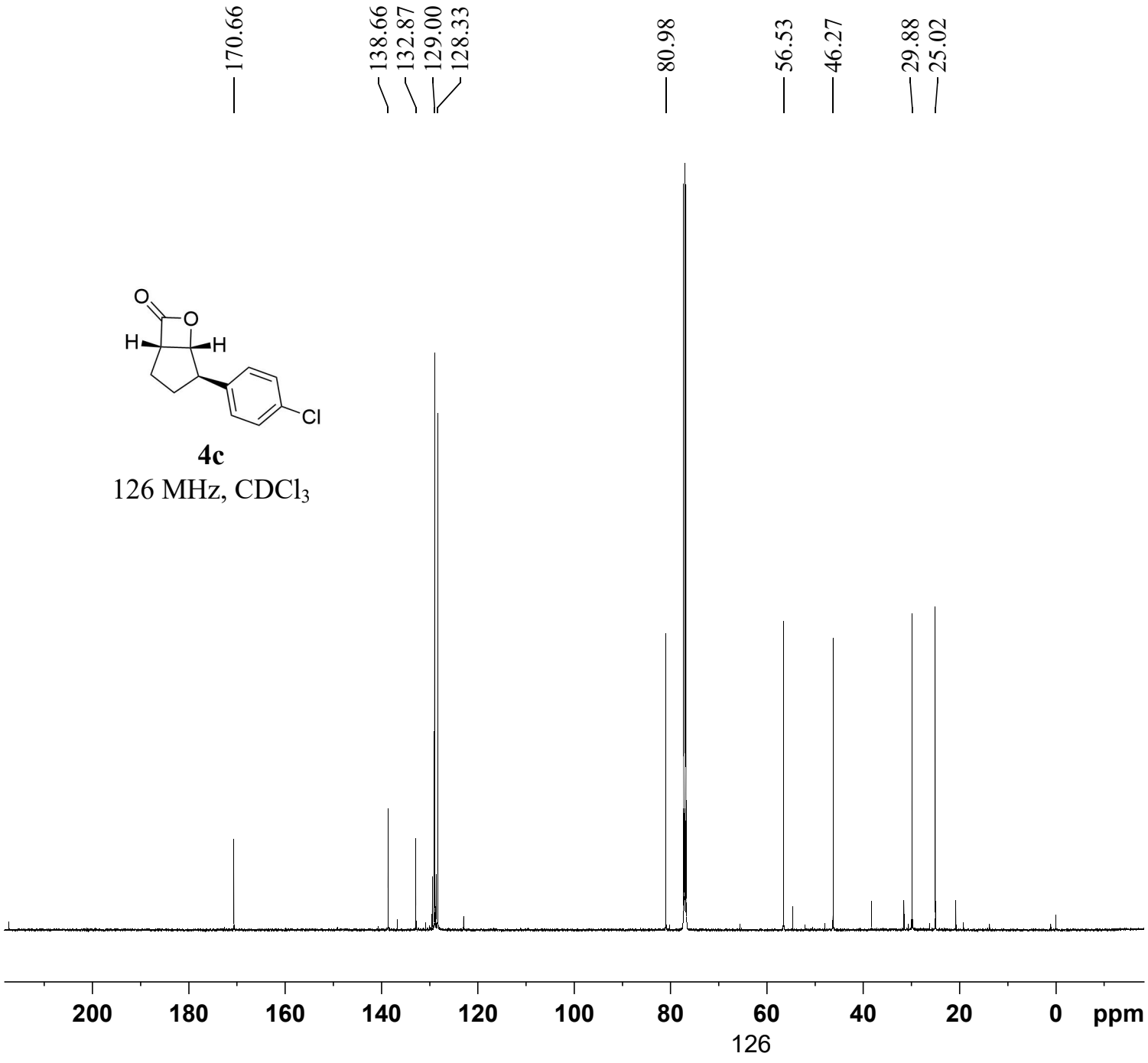

```
Current Data Parameters
NAME        500M-2020xia
EXPNO                111
PROCNO                 1

F2 - Acquisition Parameters
Date_           20210424
Time                9.53
INSTRUM            spect
PROBHD   5 mm CPPBBO BB
PULPROG          zgpg30
TD                65536
SOLVENT           CDCl3
NS                 1024
DS                    4
SWH           29761.904 Hz
FIDRES         0.454131 Hz
AQ            1.1010048 sec
RG               192.89
DW               16.800 usec
DE               18.00 usec
TE               298.2 K
D1           2.00000000 sec
D11          0.03000000 sec
TD0                   1

======== CHANNEL f1 ========
SFO1         125.7703637 MHz
NUC1                 13C
P1                 9.80 usec
PLW1         57.00000000 W

======== CHANNEL f2 ========
SFO2         500.1320005 MHz
NUC2                 1H
CPDPRG[2         waltz16
PCPD2              80.00 usec
PLW2         20.00000000 W
PLW12         0.35778001 W
PLW13         0.22898000 W

F2 - Processing parameters
SI                  32768
SF           125.7577899 MHz
WDW                    EM
SSB       0
LB                 1.00 Hz
GB        0
PC                 1.40
```



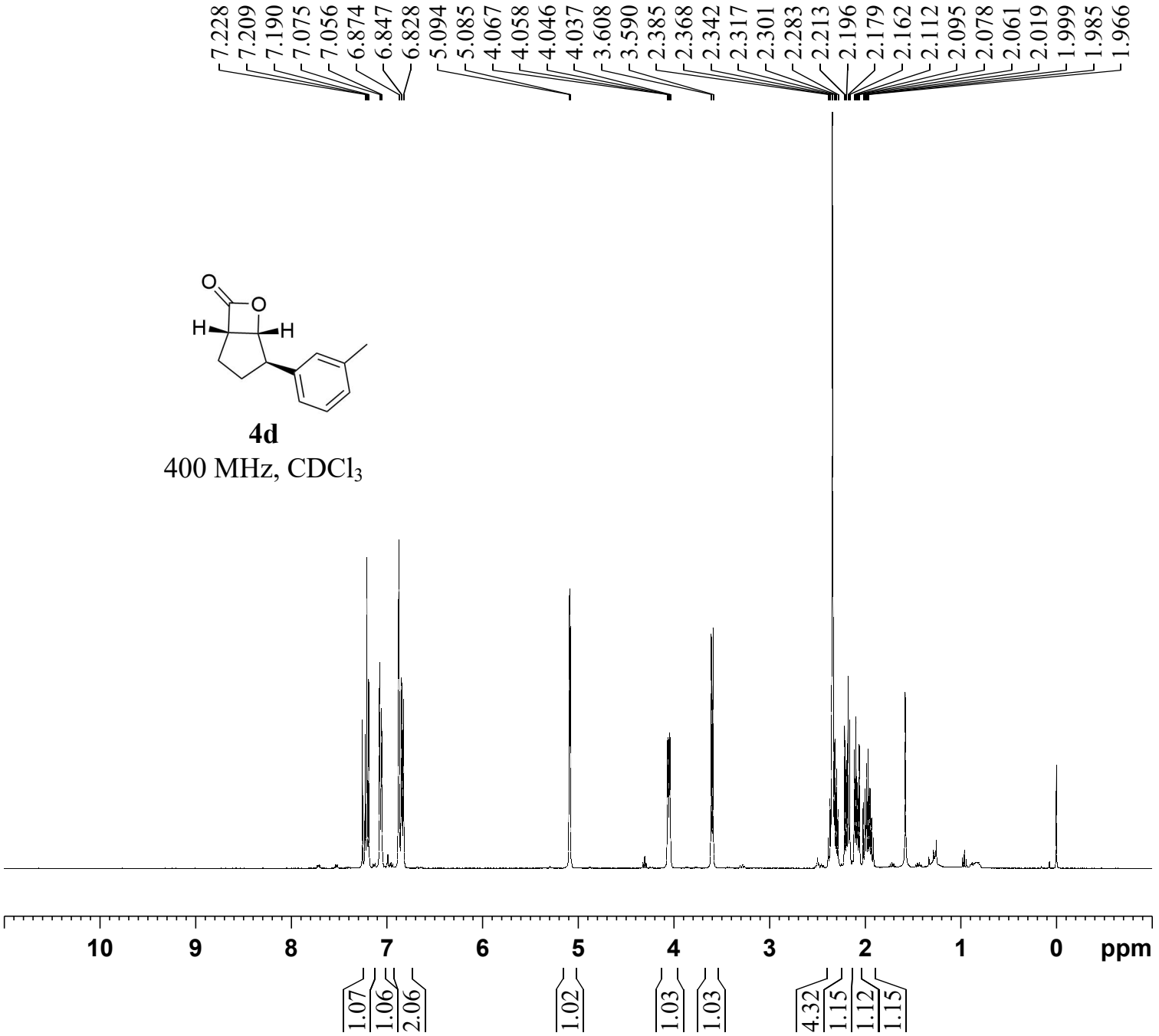
4c

500 MHz, $CDCl_3$

```
Current Data Parameters
NAME        500M-2020xia
EXPNO                100
PROCNO                 1

F2 - Acquisition Parameters
Date_           20210424
Time                4.05
INSTRUM            spect
PROBHD   5 mm CPPBBO BB
PULPROG             zg30
TD                 65536
SOLVENT            CDCl3
NS                    16
DS                     2
SWH           10000.000 Hz
FIDRES         0.152588 Hz
AQ            3.2767999 sec
RG                 55.37
DW                50.000 usec
DE                 6.50 usec
TE                298.2 K
D1           1.00000000 sec
D11      0 sec
TD0                    1

======== CHANNEL f1 ========
SFO1         500.1330885 MHz
NUC1                   1H
P1                 10.59 usec
PLW1         20.00000000 W

======== CHANNEL f2 ========
SFO2         500.1330885 MHz
NUC2                  off
CPDPRG[2
PCPD2     0 usec
PLW2      0 W
PLW12     0 W
PLW13     0 W

F2 - Processing parameters
SI                 65536
SF           500.1300116 MHz
WDW                   EM
SSB       0
LB                 0.30 Hz
GB        0
PC                 1.00
```



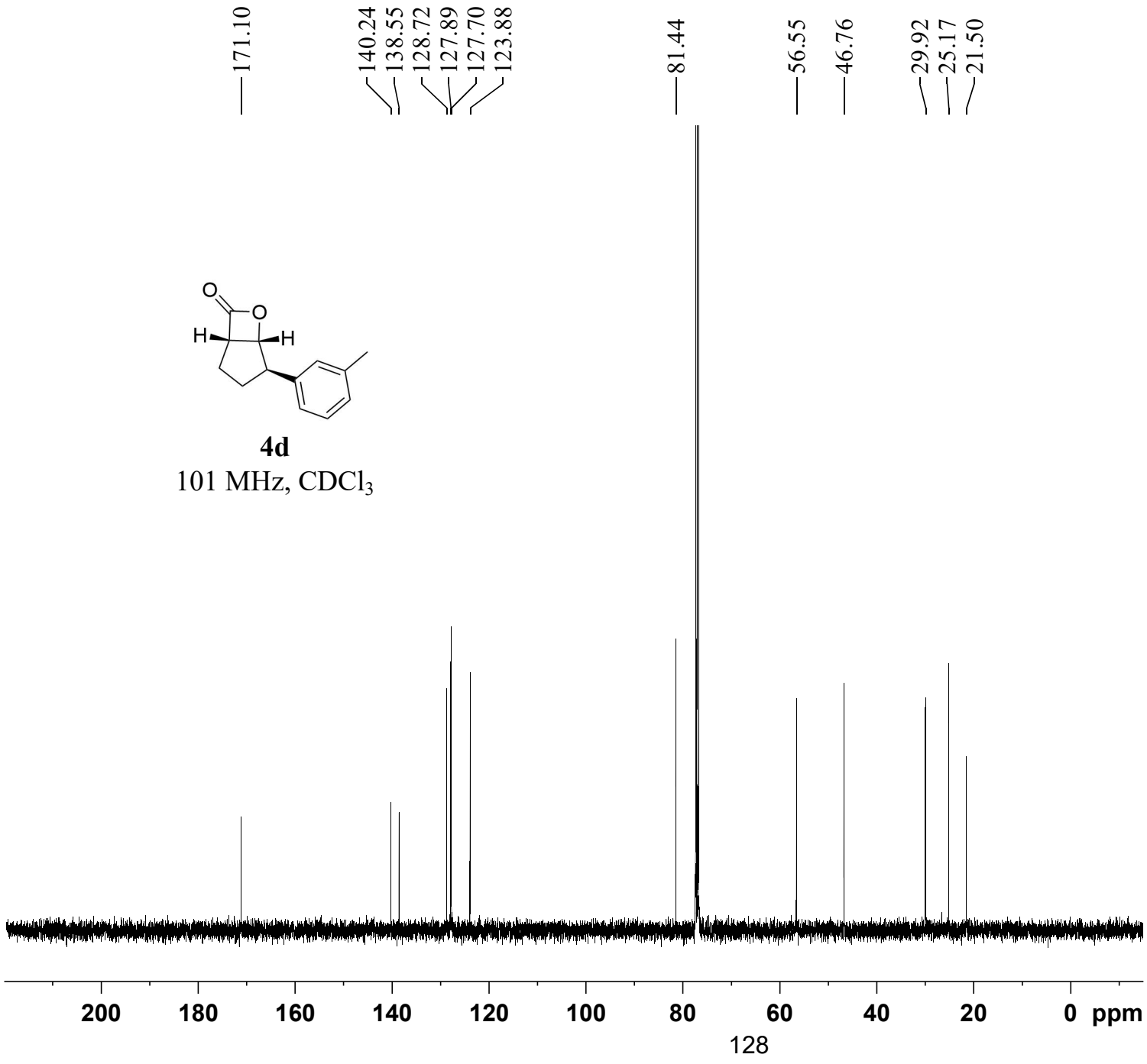

```
Current Data Parameters
NAME        500M-2020xia
EXPNO                101
PROCNO                 1

F2 - Acquisition Parameters
Date_           20210424
Time                5.00
INSTRUM            spect
PROBHD   5 mm CPPBBO BB
PULPROG           zgpg30
TD                 65536
SOLVENT            CDCl3
NS                  1024
DS                     4
SWH            29761.904 Hz
FIDRES          0.454131 Hz
AQ             1.1010048 sec
RG                192.89
DW                16.800 usec
DE                18.00 usec
TE                298.2 K
D1            2.00000000 sec
D11           0.03000000 sec
TD0                    1

======== CHANNEL f1 ========
SFO1         125.7703637 MHz
NUC1                 13C
P1                  9.80 usec
PLW1         57.00000000 W

======== CHANNEL f2 ========
SFO2         500.1320005 MHz
NUC2                 1H
CPDPRG[2         waltz16
PCPD2              80.00 usec
PLW2         20.00000000 W
PLW12         0.35778001 W
PLW13         0.22898000 W

F2 - Processing parameters
SI                  32768
SF           125.7577905 MHz
WDW                    EM
SSB       0
LB                  1.00 Hz
GB        0
PC                  1.40
```



4d

400 MHz, $CDCl_3$

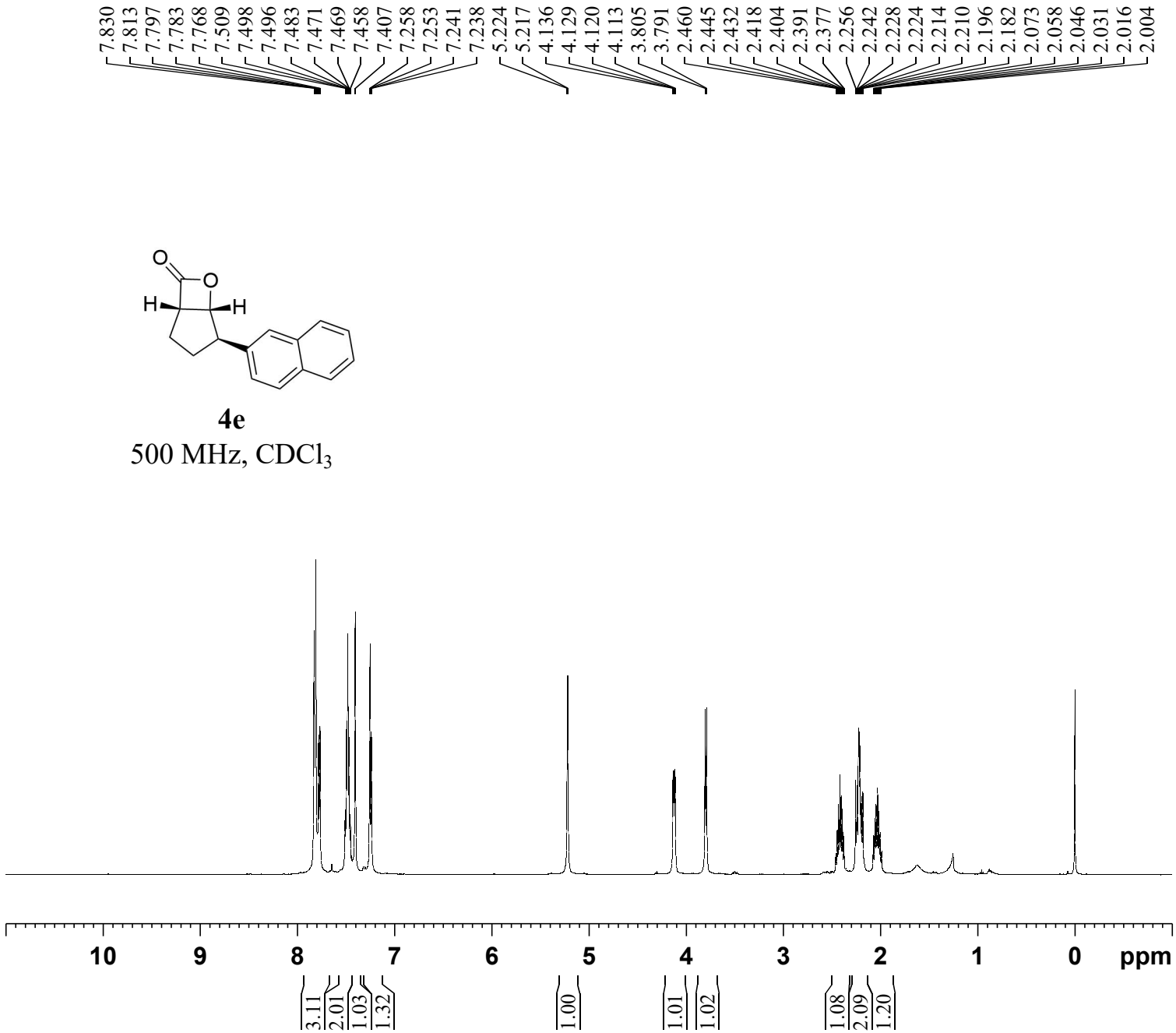
```
Current Data Parameters
NAME                 400xia
EXPNO                    50
PROCNO                    1

F2 - Acquisition Parameters
Date_              20210602
Time                 15.23
INSTRUM              spect
PROBHD    5 mm PABBO BB/
PULPROG               zg30
TD                   32768
SOLVENT              CDCl3
NS                      16
DS                       0
SWH               8012.820 Hz
FIDRES            0.244532 Hz
AQ               2.0447233 sec
RG                  206.33
DW                  62.400 usec
DE                   6.50 usec
TE                  299.5 K
D1              2.00000000 sec
D11       0 sec
TD0                      1

======== CHANNEL f1 ========
SFO1           400.2424716 MHz
NUC1                    1H
P1                   14.30 usec
PLW1           12.00000000 W

======== CHANNEL f2 ========
SFO2           400.2424716 MHz
NUC2                   off
CPDPRG[2
PCPD2     0 usec
PLW2      0 W
PLW12     0 W
PLW13     0 W

F2 - Processing parameters
SI                    65536
SF             400.2400118 MHz
WDW                     EM
SSB       0
LB                   0.30 Hz
GB        0
PC                   1.00
```



4d

101 MHz, $CDCl_3$

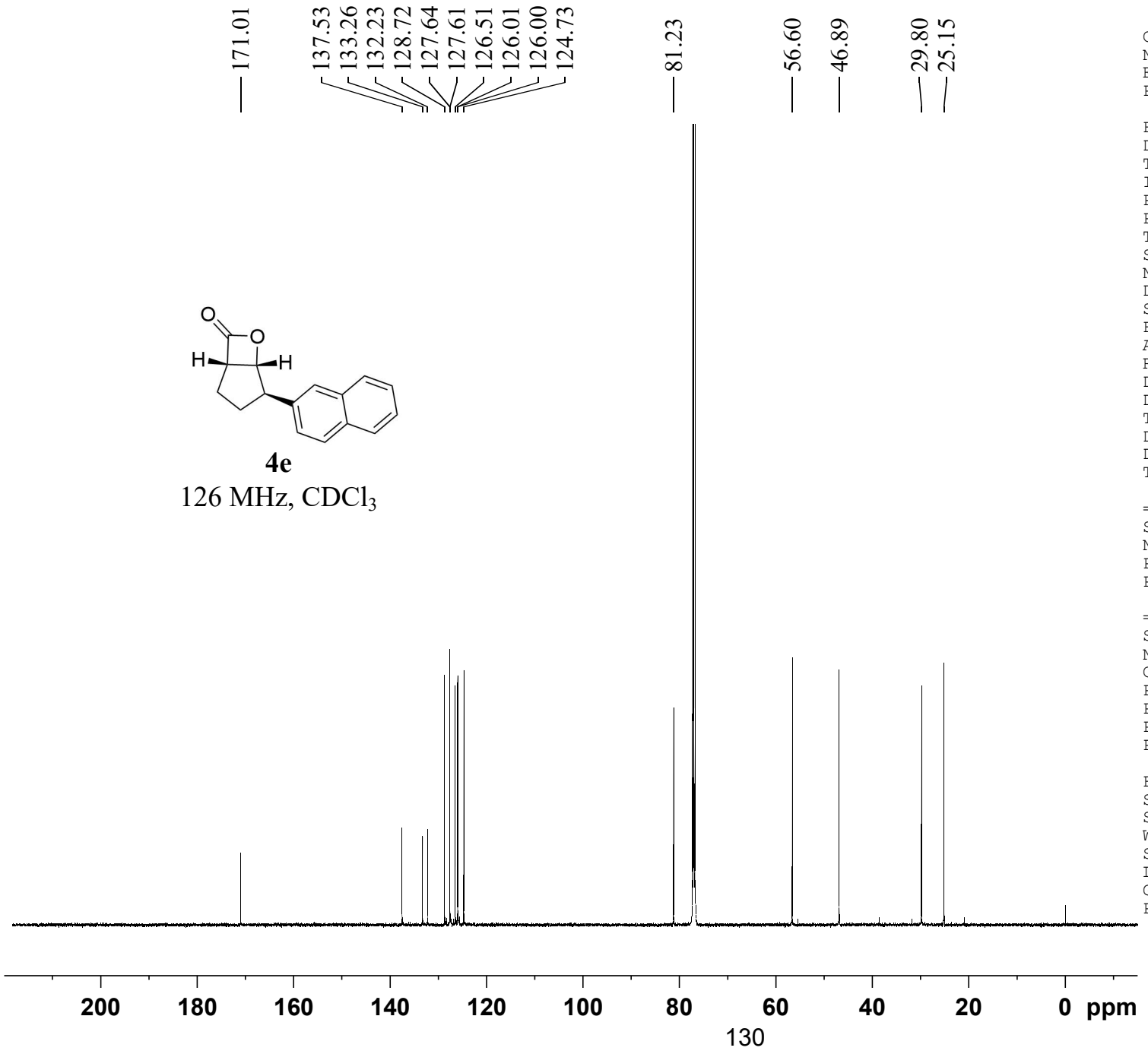
```
Current Data Parameters
NAME                 400xia
EXPNO                    51
PROCNO                    1

F2 - Acquisition Parameters
Date_             20210602
Time                 15.38
INSTRUM              spect
PROBHD   5 mm PABBO BB/
PULPROG             zgpg30
TD                   65536
SOLVENT              CDCl3
NS                     256
DS                       4
SWH              24038.461 Hz
FIDRES            0.366798 Hz
AQ                1.3631488 sec
RG                   206.33
DW                  20.800 usec
DE                   6.50 usec
TE                  300.0 K
D1               2.00000000 sec
D11              0.03000000 sec
TD0                       1

======== CHANNEL f1 ========
SFO1           100.6504916 MHz
NUC1                    13C
P1                   10.00 usec
PLW1           54.00000000 W

======== CHANNEL f2 ========
SFO2           400.2416010 MHz
NUC2                    1H
CPDPRG[2           waltz16
PCPD2                90.00 usec
PLW2           12.00000000 W
PLW12           0.30294999 W
PLW13           0.24539000 W

F2 - Processing parameters
SI                   32768
SF             100.6404280 MHz
WDW                     EM
SSB       0
LB                    1.00 Hz
GB        0
PC                    1.40
```



4e

500 MHz, $CDCl_3$

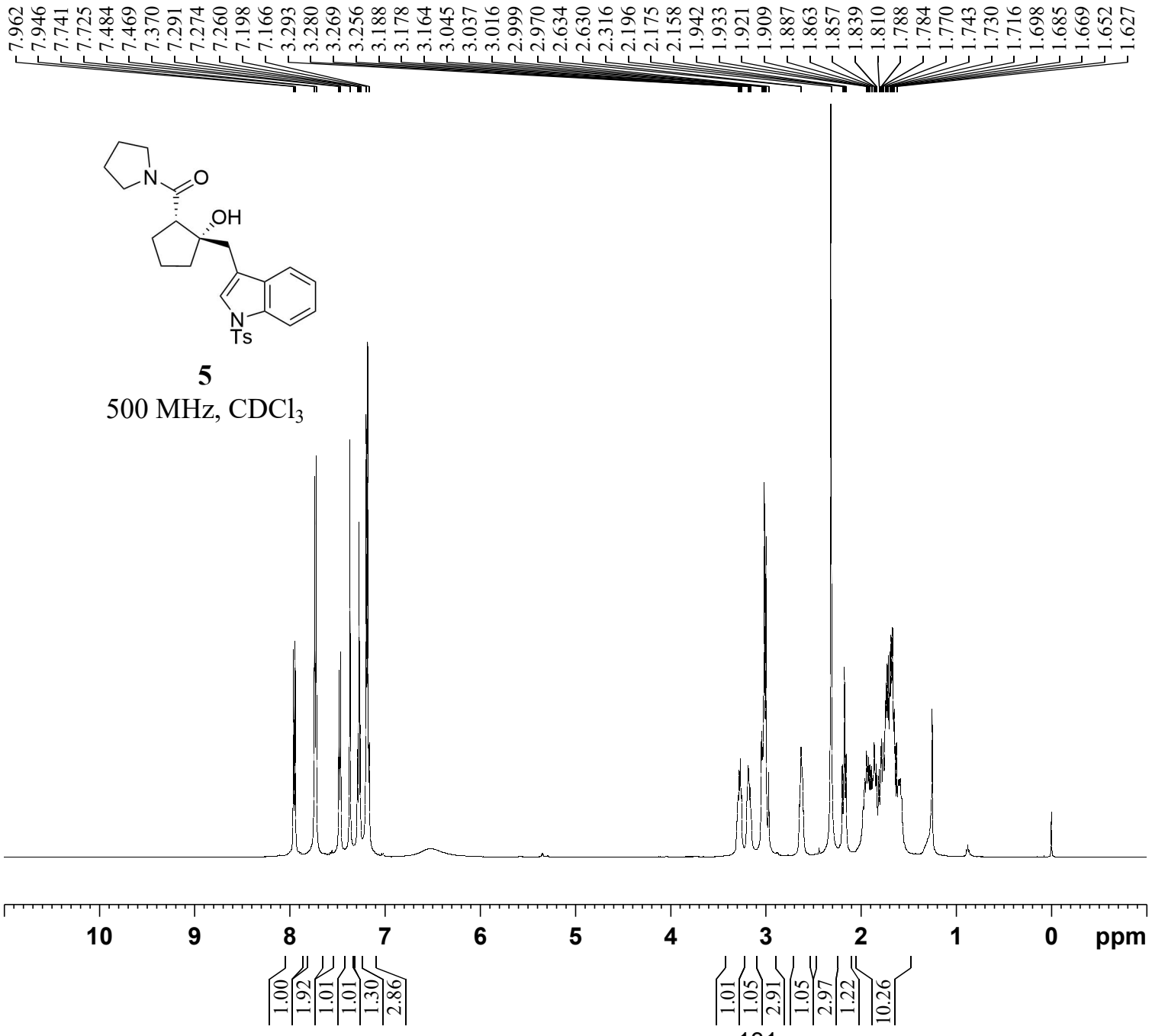
```
Current Data Parameters
NAME        500M-2020xia
EXPNO                108
PROCNO                 1

F2 - Acquisition Parameters
Date_           20210424
Time                8.00
INSTRUM            spect
PROBHD   5 mm CPPBBO BB
PULPROG             zg30
TD                 65536
SOLVENT            CDCl3
NS                    16
DS                     2
SWH           10000.000 Hz
FIDRES         0.152588 Hz
AQ            3.2767999 sec
RG                 62.06
DW                50.000 usec
DE                 6.50 usec
TE                298.2 K
D1           1.00000000 sec
D11      0 sec
TD0                    1

======== CHANNEL f1 ========
SFO1         500.1330885 MHz
NUC1                   1H
P1                 10.59 usec
PLW1         20.00000000 W

======== CHANNEL f2 ========
SFO2         500.1330885 MHz
NUC2                  off
CPDPRG[2
PCPD2     0 usec
PLW2      0 W
PLW12     0 W
PLW13     0 W

F2 - Processing parameters
SI                 65536
SF           500.1300157 MHz
WDW                   EM
SSB       0
LB                 0.30 Hz
GB        0
PC                 1.00
```



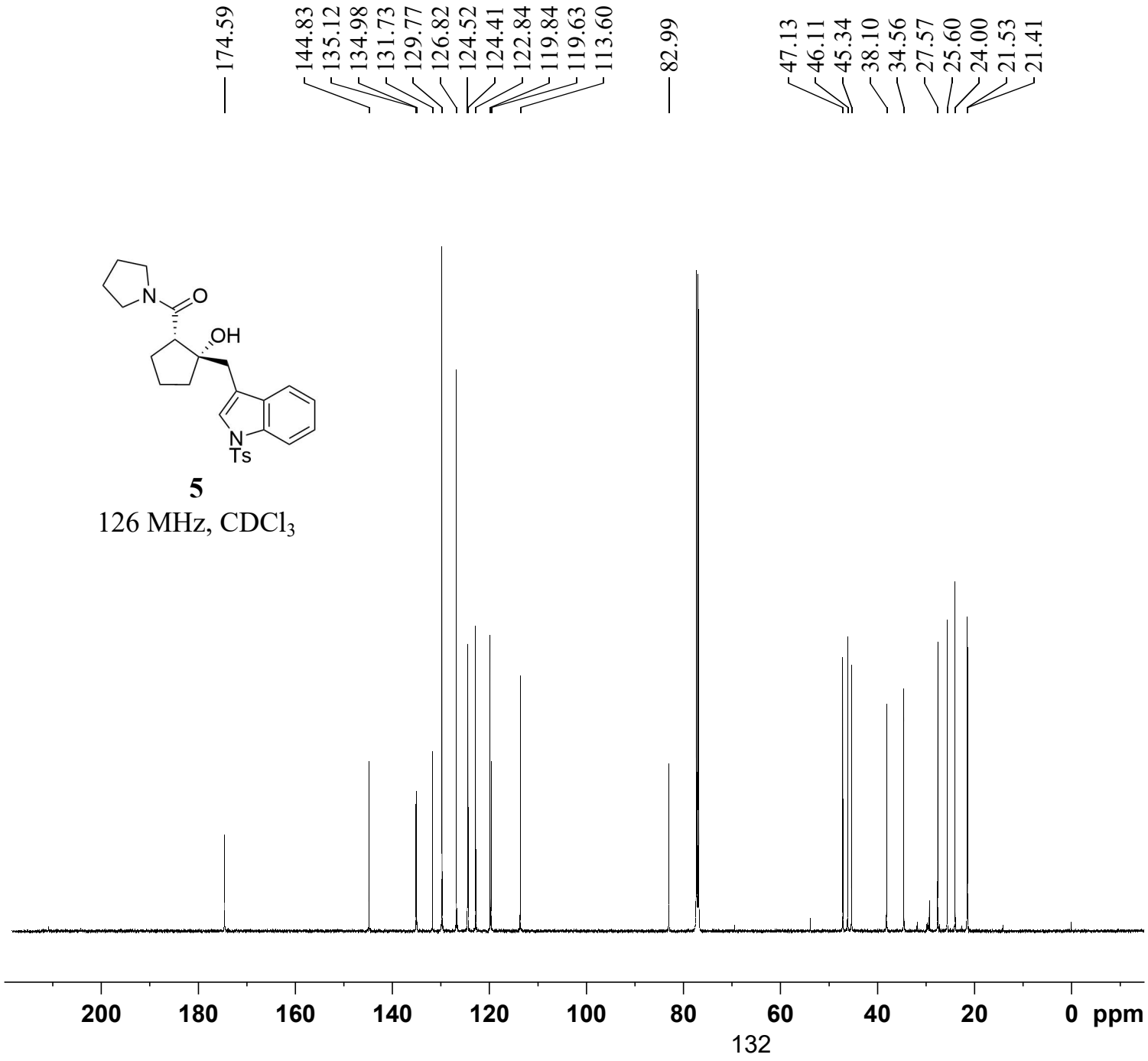
4e

126 MHz, $CDCl_3$

```
Current Data Parameters
NAME        500M-2020xia
EXPNO                109
PROCNO                 1

F2 - Acquisition Parameters
Date_           20210424
Time                8.55
INSTRUM            spect
PROBHD   5 mm CPPBBO BB
PULPROG           zgpg30
TD                 65536
SOLVENT            CDCl3
NS                  1024
DS                     4
SWH            29761.904 Hz
FIDRES          0.454131 Hz
AQ             1.1010048 sec
RG                192.89
DW                16.800 usec
DE                18.00 usec
TE                298.2 K
D1            2.00000000 sec
D11           0.03000000 sec
TD0                    1

======== CHANNEL f1 ========
SFO1         125.7703637 MHz
NUC1                 13C
P1                  9.80 usec
PLW1         57.00000000 W

======== CHANNEL f2 ========
SFO2         500.1320005 MHz
NUC2                 1H
CPDPRG[2         waltz16
PCPD2              80.00 usec
PLW2         20.00000000 W
PLW12         0.35778001 W
PLW13         0.22898000 W

F2 - Processing parameters
SI                  32768
SF           125.7577913 MHz
WDW                    EM
SSB       0
LB                  1.00 Hz
GB        0
PC                  1.40
```



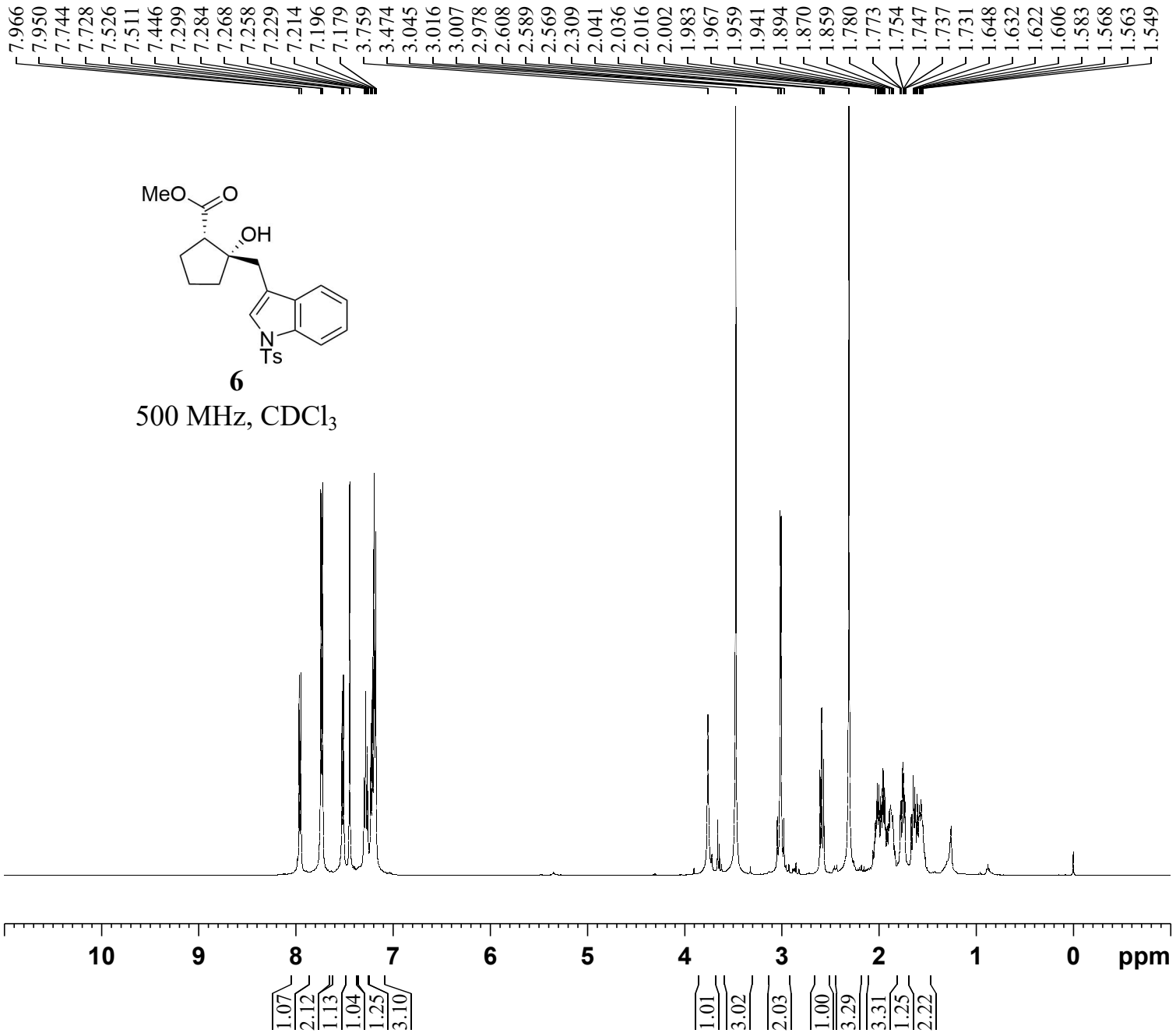

```
Current Data Parameters
NAME        500M-2020xia
EXPNO                124
PROCNO                 1

F2 - Acquisition Parameters
Date_           20210527
Time                4.52
INSTRUM            spect
PROBHD   5 mm CPPBBO BB
PULPROG             zg30
TD                 65536
SOLVENT            CDCl3
NS                    16
DS                     2
SWH           10000.000 Hz
FIDRES         0.152588 Hz
AQ            3.2767999 sec
RG                 31.72
DW                50.000 usec
DE                 6.50 usec
TE                298.2 K
D1           1.00000000 sec
D11      0 sec
TD0                    1

======== CHANNEL f1 ========
SFO1         500.1330885 MHz
NUC1                   1H
P1                 10.59 usec
PLW1         20.00000000 W

======== CHANNEL f2 ========
SFO2         500.1330885 MHz
NUC2                  off
CPDPRG[2
PCPD2     0 usec
PLW2      0 W
PLW12     0 W
PLW13     0 W

F2 - Processing parameters
SI                 65536
SF           500.1300053 MHz
WDW                   EM
SSB       0
LB                 0.30 Hz
GB        0
PC                 1.00
```



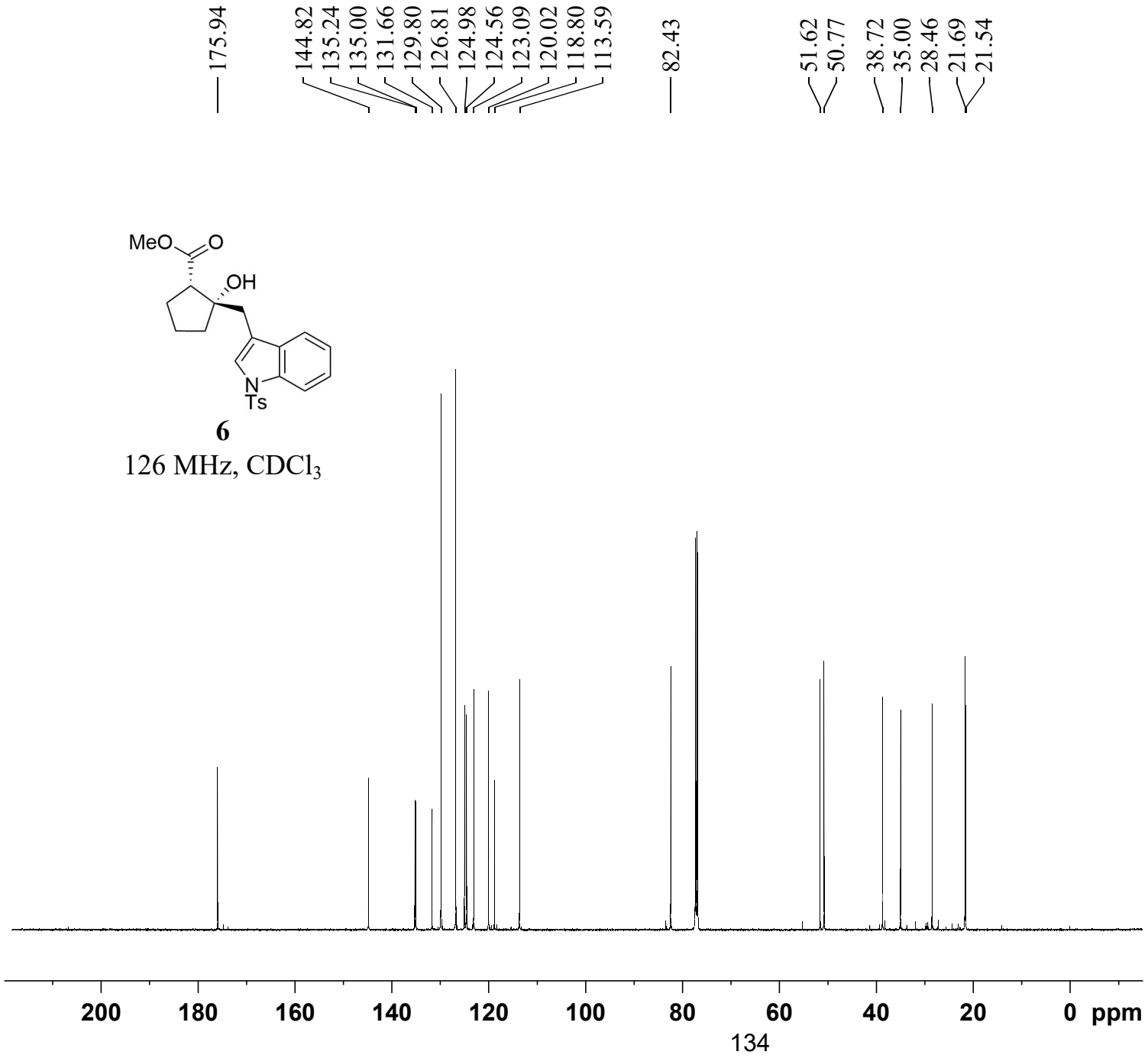
5

126 MHz, $CDCl_3$

```
Current Data Parameters
NAME        500M-2020xia
EXPNO                125
PROCNO                 1

F2 - Acquisition Parameters
Date_           20210527
Time                5.20
INSTRUM            spect
PROBHD   5 mm CPPBBO BB
PULPROG          zgpg30
TD                65536
SOLVENT           CDCl3
NS                  512
DS                    4
SWH           29761.904 Hz
FIDRES         0.454131 Hz
AQ             1.1010048 sec
RG                192.89
DW                16.800 usec
DE                18.00 usec
TE                298.2 K
D1            2.00000000 sec
D11           0.03000000 sec
TD0                   1

======== CHANNEL f1 ========
SFO1         125.7703637 MHz
NUC1                 13C
P1                  9.80 usec
PLW1         57.00000000 W

======== CHANNEL f2 ========
SFO2         500.1320005 MHz
NUC2                 1H
CPDPRG[2         waltz16
PCPD2              80.00 usec
PLW2         20.00000000 W
PLW12         0.35778001 W
PLW13         0.22898000 W

F2 - Processing parameters
SI                  32768
SF           125.7577885 MHz
WDW                    EM
SSB       0
LB                  1.00 Hz
GB        0
PC                  1.40
```



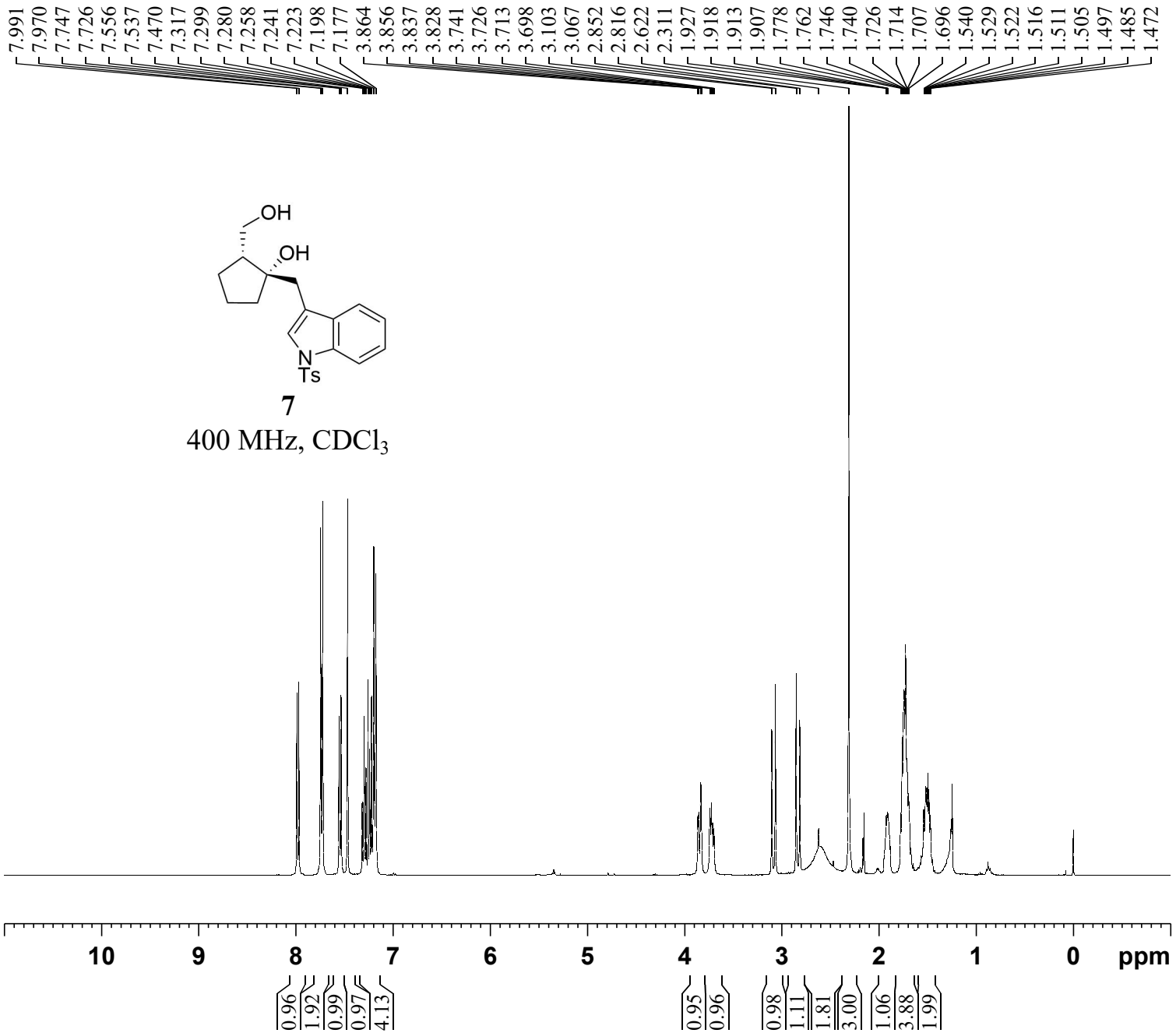

```
Current Data Parameters
NAME        500M-2020xia
EXPNO                128
PROCNO                 1

F2 - Acquisition Parameters
Date_           20210527
Time                6.22
INSTRUM            spect
PROBHD   5 mm CPPBBO BB
PULPROG             zg30
TD                 65536
SOLVENT            CDCl3
NS                    16
DS                     2
SWH           10000.000 Hz
FIDRES         0.152588 Hz
AQ            3.2767999 sec
RG                 31.72
DW                50.000 usec
DE                 6.50 usec
TE                298.2 K
D1           1.00000000 sec
D11      0 sec
TD0                    1

======== CHANNEL f1 ========
SFO1         500.1330885 MHz
NUC1                   1H
P1                 10.59 usec
PLW1         20.00000000 W

======== CHANNEL f2 ========
SFO2         500.1330885 MHz
NUC2                  off
CPDPRG[2
PCPD2     0 usec
PLW2      0 W
PLW12     0 W
PLW13     0 W

F2 - Processing parameters
SI                 65536
SF         500.1300129 MHz
WDW                   EM
SSB       0
LB                 0.30 Hz
GB        0
PC                 1.00
```



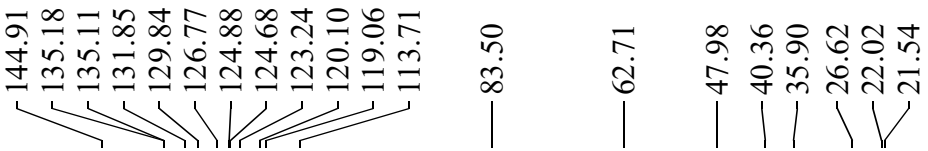
6

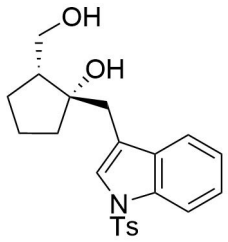
126 MHz, $CDCl_3$

```
Current Data Parameters
NAME        500M-2020xia
EXPNO                129
PROCNO                 1

F2 - Acquisition Parameters
Date_           20210527
Time                6.50
INSTRUM            spect
PROBHD   5 mm CPPBBO BB
PULPROG          zgpg30
TD                65536
SOLVENT           CDCl3
NS                  512
DS                    4
SWH           29761.904 Hz
FIDRES         0.454131 Hz
AQ            1.1010048 sec
RG               192.89
DW               16.800 usec
DE               18.00 usec
TE               298.2 K
D1           2.00000000 sec
D11          0.03000000 sec
TD0                   1

======== CHANNEL f1 ========
SFO1        125.7703637 MHz
NUC1                13C
P1                 9.80 usec
PLW1        57.00000000 W

======== CHANNEL f2 ========
SFO2        500.1320005 MHz
NUC2                1H
CPDPRG[2         waltz16
PCPD2             80.00 usec
PLW2        20.00000000 W
PLW12        0.35778001 W
PLW13        0.22898000 W

F2 - Processing parameters
SI                 32768
SF          125.7577885 MHz
WDW                   EM
SSB       0
LB                 1.00 Hz
GB        0
PC                 1.40
```



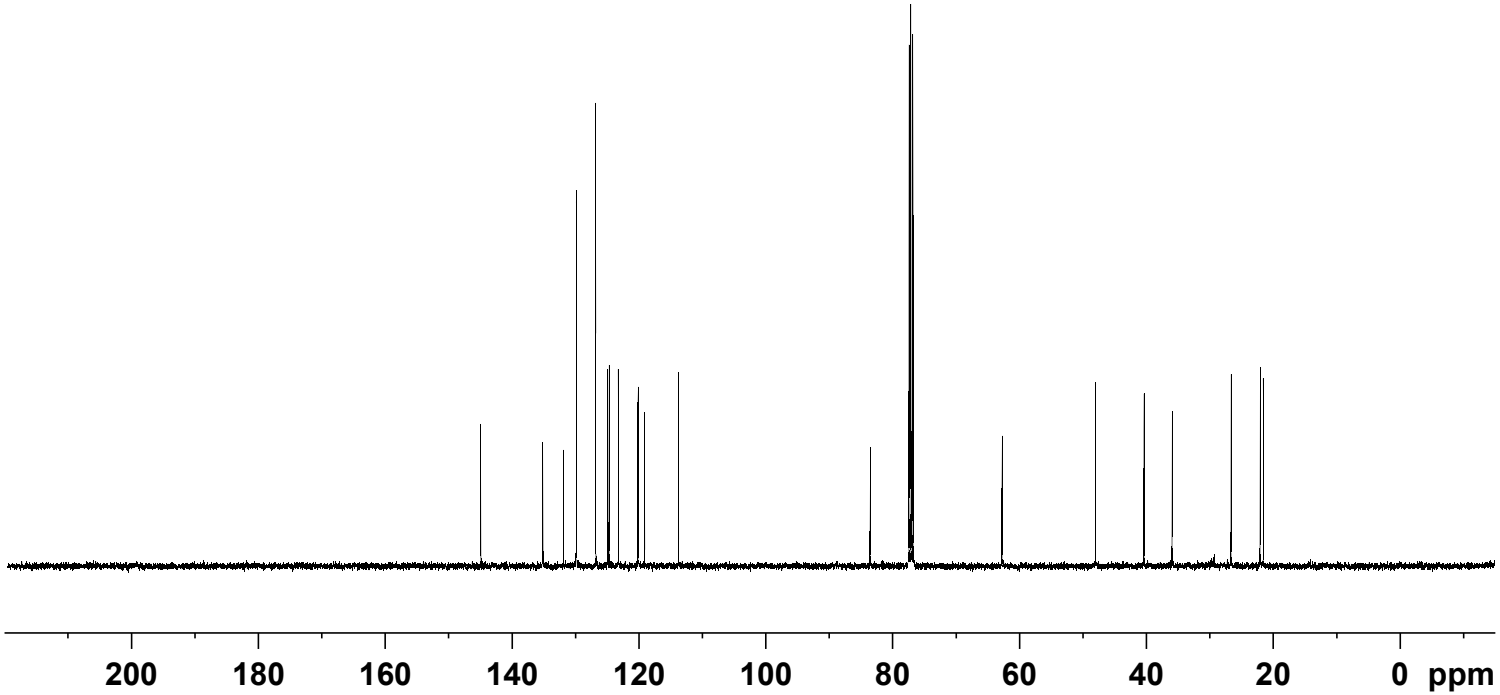

```
Current Data Parameters
NAME                 400xia
EXPNO                    54
PROCNO                    1

F2 - Acquisition Parameters
Date_              20210602
Time                 17.03
INSTRUM              spect
PROBHD    5 mm PABBO BB/
PULPROG              zg30
TD                  32768
SOLVENT              CDCl3
NS                      16
DS                       0
SWH               8012.820 Hz
FIDRES            0.244532 Hz
AQ               2.0447233 sec
RG                   55.55
DW                  62.400 usec
DE                   6.50 usec
TE                  299.6 K
D1              2.00000000 sec
D11       0 sec
TD0                      1

======== CHANNEL f1 ========
SFO1           400.2424716 MHz
NUC1                    1H
P1                   14.30 usec
PLW1           12.00000000 W

======== CHANNEL f2 ========
SFO2           400.2424716 MHz
NUC2                   off
CPDPRG[2
PCPD2      0 usec
PLW2       0 W
PLW12      0 W
PLW13      0 W

F2 - Processing parameters
SI                    65536
SF             400.2400103 MHz
WDW                      EM
SSB        0
LB                    0.30 Hz
GB         0
PC                    1.00
```





7

101 MHz, $CDCl_3$




```
Current Data Parameters
NAME                 400xia
EXPNO                    55
PROCNO                    1

F2 - Acquisition Parameters
Date_             20210602
Time                 17.32
INSTRUM              spect
PROBHD   5 mm PABBO BB/
PULPROG             zgpg30
TD                   65536
SOLVENT              CDCl3
NS                     512
DS                       4
SWH              24038.461 Hz
FIDRES            0.366798 Hz
AQ                1.3631488 sec
RG                   206.33
DW                  20.800 usec
DE                   6.50 usec
TE                  300.0 K
D1               2.00000000 sec
D11              0.03000000 sec
TD0                       1

======== CHANNEL f1 ========
SFO1           100.6504916 MHz
NUC1                    13C
P1                    10.00 usec
PLW1           54.00000000 W

======== CHANNEL f2 ========
SFO2           400.2416010 MHz
NUC2                    1H
CPDPRG[2           waltz16
PCPD2                 90.00 usec
PLW2           12.00000000 W
PLW12           0.30294999 W
PLW13           0.24539000 W

F2 - Processing parameters
SI                    32768
SF             100.6404280 MHz
WDW                      EM
SSB       0
LB                     1.00 Hz
GB        0
PC                     1.40
```