Electronic Supplementary Information

Phosphine-Catalyzed Activation of Cyclopropenones: A Versatile C3 Synthon for (3+2) Annulations with Unsaturated Electrophiles

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1. General Information

Unless otherwise noted, all reactions were performed in nitrogen atmosphere under anhydrous conditions using standard Schlenk techniques. Diphenylcyclopropenone **1a** was purchased from commercial sources; other cyclopropenones were prepared according to reported methods.[1] Solvents were purified prior to use according to standard procedures. ^{1}H, ^{13}C and ^{19}F NMR spectra were recorded on a Bruker AV600/JEOL 400 spectrometer. Chemical shifts (δ values) were reported in ppm with TMS (^{1}H NMR) and $CDCl_3$ (^{13}C NMR) as internal standard, respectively. $CDCl_3$ was treated with anhydrous K_2CO_3 before using. Peak multiplicities are reported as follows: s = singlet, d = doublet, t = triplet, q = quartet, dd = doublet of doublets, hept = heptet, m = multiplet. High-resolution ESI mass spectra were determined on WATERS I-Class VION IMS Q Tof LC/MS. IR data were measured on a Nicolet iS10 FT-IR spectrometer. Melting points were measured on an SGW® X-4B apparatus and uncorrected. All reactions were monitored by thin layer chromatography (TLC) and visualized by UV irradiation under 254 nm or 365 nm, or stained with potassium permanganate. X-ray crystallographic analysis was performed at Bruker D8 Quest. Flash column chromatography was performed over silica gel or alumina, using petroleum ether (60–90 ºC)/ethyl acetate as the eluent.

2. Synthesis of Compounds 3, 4, 6, 8, and Analytical Data

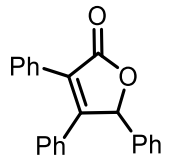
2.1 General Procedure for the Synthesis of Compounds 3, 4, 6, and 8

Under N_2 atmosphere, PMe_3 (1 M in THF, 20 μL, 0.02 mmol) was added to a dispersion of cyclopropenone (0.3 mmol), 4 Å molecular sieves (100 mg), and aldehyde (0.2 mmol) in anhydrous dichloromethane (DCM) (2.0 mL). The mixture was stirred at room temperature and the reaction was monitored by TLC. When the reaction finished, the solvent was evaporated in *vacuo* and the residue was purified by silica gel column chromatography with petroleum ether/ethyl acetate as eluent to afford the corresponding products **3**, **4**, **6**, and **8**. The analytical

data are provided as follows. Compounds **3a**–**d, 3g**–**h, 3l, 3q, 3w**–**y, 3ab, 4,** and **8a** are known, and the analytical data are consistent with those reported.[2–9]

2.2 Analytical Data for Compounds 3, 4, 6, and 8

3,4,5-triphenylfuran-2(5*H*)-one (3a)[2]

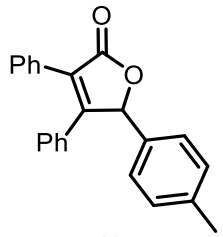


3a

Following the general procedure, the reaction of 2,3-diphenylcycloprop-2-en-1-one **1a** (62.1 mg, 0.3 mmol) and benzaldehyde **2a** (21 μL, 0.2 mmol) with PMe_3 (1 M in THF, 20 μL, 0.02 mmol) was conducted for 40 minutes which produced **3a** in 61.7 mg, 99% yield, as white solid.

^{1}H NMR (400 MHz, $CDCl_3$): δ = 7.50 – 7.46 (m, 2H), 7.36 – 7.32 (m, 3H), 7.31 – 7.27 (m, 5H), 7.26 – 7.22 (m, 1H), 7.21 – 7.15 (m, 2H), 7.12 – 7.09 (m, 2H), 6.26 (s, 1H).

3,4-diphenyl-5-(*p*-tolyl) furan-2(5*H*)-one (3b)[2]

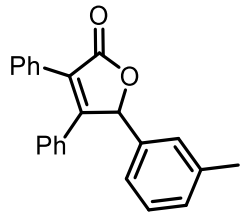


3b

Following the general procedure, the reaction of 2,3-diphenylcycloprop-2-en-1-one **1a** (62.0 mg, 0.3 mmol) and 4-methylbenzaldehyde **2b** (24 μL, 0.2 mmol) with PMe_3 (1 M in THF, 20 μL, 0.02 mmol) was conducted for 3 hours which produced **3b** in 62.1 mg, 95% yield, as pale-yellow solid.

^{1}H NMR (400 MHz, $CDCl_3$): δ = 7.50 – 7.43 (m, 2H), 7.38 – 7.30 (m, 3H), 7.27 – 7.16 (m, 5H), 7.13 – 7.07 (m, 4H), 6.23 (s, 1H), 2.29 (s, 3H).

3,4-diphenyl-5-(*m*-tolyl) furan-2(5*H*)-one (3c)[2]

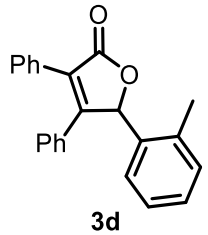


3c

Following the general procedure, the reaction of 2,3-diphenylcycloprop-2-en-1-one **1a** (62.1 mg, 0.3 mmol) and 3-methylbenzaldehyde **2c** (24 µL, 0.2 mmol) with PMe_3 (1 M in THF, 20 µL, 0.02 mmol) was conducted for 3 hours which produced **3c** in 59.5 mg, 91% yield, as pale-yellow solid.

^{1}H NMR (400 MHz, $CDCl_3$): δ = 7.53 – 7.45 (m, 2H), 7.37 – 7.32 (m, 3H), 7.28 – 7.24 (m, 1H), 7.22 – 7.16 (m, 3H), 7.13 – 7.05 (m, 5H), 6.21 (s, 1H), 2.29 (s, 3H).

3,4-diphenyl-5-(*o*-tolyl) furan-2(5*H*)-one (3d)[2]

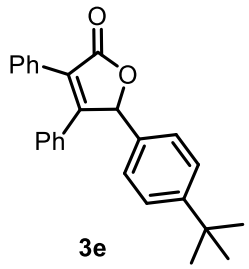


3d

Following the general procedure, the reaction of 2,3-diphenylcycloprop-2-en-1-one **1a** (61.8 mg, 0.3 mmol) and 2-methylbenzaldehyde **2d** (24 µL, 0.2 mmol) with PMe_3 (1 M in THF, 20 µL, 0.02 mmol) was conducted for 3 hours which produced **3d** in 60.7 mg, 93% yield, as pale-yellow solid.

^{1}H NMR (400 MHz, $CDCl_3$): δ = 7.50 – 7.46 (m, 2H), 7.37 – 7.32 (m, 3H), 7.27 – 7.23 (m, 1H), 7.22 – 7.16 (m, 4H), 7.12 – 7.06 (m, 4H), 6.54 (s, 1H), 2.44 (s, 3H).

5-(4-(*tert*-butyl) phenyl)-3,4-diphenylfuran-2(5*H*)-one (3e)

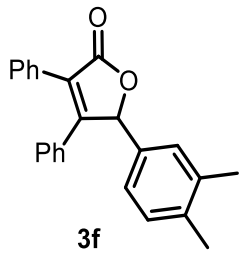


3e

Following the general procedure, the reaction of 2,3-diphenylcycloprop-2-en-1-one **1a** (61.8 mg, 0.3 mmol) and 4-(*tert*-butyl) benzaldehyde **2e** (32.5 mg, 0.2 mmol) with PMe_3 (1 M in THF, 20 µL, 0.02 mmol) was conducted for 3 hours which produced **3e** in 64.8 mg, 88% yield, as pale-yellow solid, m.p. 120 – 123 °C.

^{1}H NMR (400 MHz, $CDCl_3$): δ = 7.49 – 7.42 (m, 2H), 7.36 – 7.31 (m, 5H), 7.28 – 7.24 (m, 2H), 7.22 – 7.17 (m, 3H), 7.15 – 7.11 (m, 2H), 6.26 (s, 1H), 1.26 (s, 9H); **^{13}C NMR** (100 MHz, $CDCl_3$): δ = 172.6, 159.2, 152.5, 131.8, 131.3, 130.1, 130.0, 129.5, 128.9, 128.8, 128.7, 128.5, 127.5, 127.1, 126.0, 83.6, 34.7, 31.3; **FTIR** (neat): υ 2960, 1736, 1445, 1350, 1160, 1003, 783, 692 cm^{-1}; **HRMS** (ESI) calcd for $C_{26}H_{24}O_2[M + Na]^+$: 391.1668, found: 391.1668.

5-(3,4-dimethylphenyl)-3,4-diphenylfuran-2(5*H*)-one (3f)



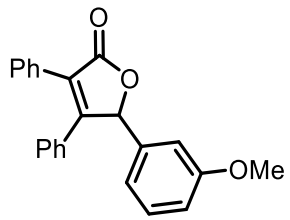
3f

Following the general procedure, the reaction of 2,3-diphenylcycloprop-2-en-1-one **1a** (62.0 mg, 0.3 mmol) and 3,4-dimethylbenzaldehyde **2f** (26.9 mg, 0.2 mmol) with PMe_3 (1 M in THF, 20 µL, 0.02 mmol) was conducted for 1 hour which produced **3f** in 61.9 mg, 91% yield, as white solid, m.p. 102 – 105 °C.

^{1}H NMR (400 MHz, $CDCl_3$): δ = 7.50 – 7.46 (m, 2H), 7.38 – 7.31 (m, 3H), 7.28 – 7.23 (m, 1H), 7.22 – 7.17 (m, 2H), 7.14 – 7.10 (m, 2H), 7.08 – 7.04 (m, 2H), 7.03 – 6.96 (m, 1H), 6.20 (s, 1H), 2.20 (s, 6H); **^{13}C NMR** (100 MHz, $CDCl_3$): δ = 172.7, 159.4, 138.1, 137.4, 132.1, 131.3, 130.2, 130.1, 129.9, 129.6, 128.9, 128.83, 128.75, 128.7, 128.5, 126.9, 125.3, 83.8, 19.9, 19.7;

FTIR (neat) υ 2918, 1745, 1444, 1297, 1155, 998, 692 cm^{-1}; **HRMS** (ESI) calcd for $C_{24}H_{20}O_2$[M + Na]$^+$: 363.1356, found: 363.1358.

5-(3-methoxyphenyl)-3,4-diphenylfuran-2(5*H*)-one (3g)[3]

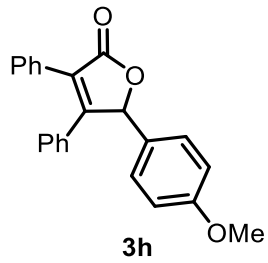


3g

Following the general procedure, the reaction of 2,3-diphenylcycloprop-2-en-1-one **1a** (62.0 mg, 0.3 mmol) and 3-methoxybenzaldehyde **2g** (25 µL, 0.2 mmol) with PMe_3 (1 M in THF, 20 µL, 0.02 mmol) was conducted for 2 hours which produced **3g** in 62.3 mg, 91% yield, as white solid.

^{1}H NMR (400 MHz, $CDCl_3$): δ = 7.50 – 7.45 (m, 2H), 7.38 – 7.32 (m, 3H), 7.30 – 7.26 (m, 1H), 7.24 – 7.18 (m, 3H), 7.14 – 7.09 (m, 2H), 6.91 – 6.79 (m, 3H), 6.22 (s, 1H), 3.74 (s, 3H).

5-(4-methoxyphenyl)-3,4-diphenylfuran-2(5*H*)-one (3h)[2]

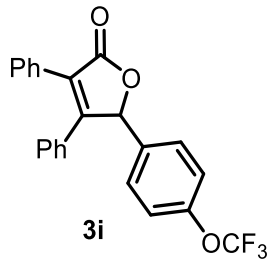


3h

Following the general procedure, the reaction of 2,3-diphenylcycloprop-2-en-1-one **1a** (62.0 mg, 0.3 mmol) and 4-methoxybenzaldehyde **2h** (25 µL, 0.2 mmol) with PMe_3 (1 M in THF, 20 µL, 0.02 mmol) was conducted for 2 hours which produced **3h** in 65.7 mg, 96% yield, as white solid.

^{1}H NMR (400 MHz, $CDCl_3$): δ = 7.47 (dd, *J* = 6.7, 3.0 Hz, 2H), 7.37 – 7.32 (m, 3H), 7.24 – 7.17 (m, 5H), 7.13 – 7.09 (m, 2H), 6.86 – 6.80 (m, 2H), 6.23 (s, 1H), 3.74 (s, 3H).

3,4-diphenyl-5-(4-(trifluoromethoxy) phenyl) furan-2(5*H*)-one (3i)

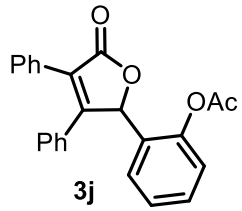


3i

Following the general procedure, the reaction of 2,3-diphenylcycloprop-2-en-1-one **1a** (61.9 mg, 0.3 mmol) and 4-(trifluoromethoxy) benzaldehyde **2i** (29 µL, 0.2 mmol) with PMe_3 (1 M in THF, 20 µL, 0.02 mmol) was conducted for 2 hours which produced **3i** in 55.5 mg, 70% yield, as pale-yellow solid, m.p. 135 – 138 °C.

^{1}H NMR (400 MHz, $CDCl_3$): δ = 7.51 – 7.43 (m, 2H), 7.37 – 7.26 (m, 6H), 7.25 – 7.20 (m, 2H), 7.19 – 7.07 (m, 4H), 6.30 (s, 1H); **^{13}C NMR** (100 MHz, $CDCl_3$): δ = 172.3, 159.1, 149.8, 133.6, 130.9, 130.2, 129.6, 129.5, 129.3, 129.1, 129.0, 128.7, 128.4, 127.2, 121.3, 120.4 (q, *J* = 257.9 Hz), 82.7; **^{19}F NMR** (376 MHz, $CDCl_3$): δ = -57.8; **FTIR** (neat) υ 1749, 1443, 1257, 1159, 1004, 692 cm^{-1}; **HRMS** (ESI) calcd for $C_{23}H_{15}F_3O_3[M + Na]^+$: 419.0866, found: 419.0870.

2-(5-oxo-3,4-diphenyl-2,5-dihydrofuran-2-yl) phenyl acetate (3j)

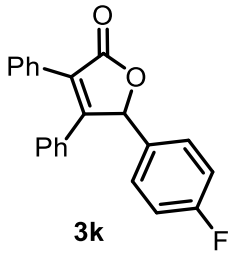


3j

Following the general procedure, the reaction of 2,3-diphenylcycloprop-2-en-1-one **1a** (62.1 mg, 0.3 mmol) and 2-formylphenyl acetate **2j** (32.8 mg, 0.2 mmol) with PMe_3 (1 M in THF, 20 µL, 0.02 mmol) was conducted for 40 minutes which produced **3j** in 43.7 mg, 59% yield, as pale-yellow solid, m.p. 123 – 126 °C.

^{1}H NMR (400 MHz, $CDCl_3$): δ = 7.50 – 7.45 (m, 2H), 7.38 – 7.33 (m, 4H), 7.30 – 7.26 (m, 1H), 7.24 – 7.14 (m, 5H), 7.12 – 7.08 (m, 2H), 6.42 (s, 1H), 2.28 (s, 3H); **^{13}C NMR** (100 MHz, $CDCl_3$): δ = 172.5, 168.9, 158.7, 149.6, 131.0, 130.6, 130.2, 129.9, 129.5, 129.4, 129.0, 128.9, 128.7, 128.3, 127.3, 126.4, 126.2, 123.6, 79.4, 21.2; **FTIR** (neat) υ 2942, 1716, 1510, 1246, 1170, 1028, 829 cm^{-1}; **HRMS** (ESI) calcd for $C_{24}H_{18}O_4[M + Na]^+$: 393.1097, found: 393.1103.

5-(4-fluorophenyl)-3,4-diphenylfuran-2(5*H*)-one (3k)

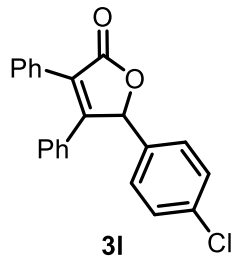


3k

Following the general procedure, the reaction of 2,3-diphenylcycloprop-2-en-1-one **1a** (61.8 mg, 0.3 mmol) and 4-fluorobenzaldehyde **2k** (22 µL, 0.2 mmol) with PMe_3 (1 M in THF, 20 µL, 0.02 mmol) was conducted for 2 hours which produced **3k** in 56.8 mg, 86% yield, as pale-yellow solid, m.p. 108 – 110 °C.

^{1}H NMR (400 MHz, $CDCl_3$): δ = 7.51 – 7.44 (m, 2H), 7.38 – 7.32 (m, 3H), 7.30 – 7.25 (m, 3H), 7.24 – 7.18 (m, 2H), 7.13 – 7.08 (m, 2H), 7.03 – 6.96 (m, 2H), 6.27 (s, 1H); **^{13}C NMR** (100 MHz, $CDCl_3$): δ = 172.4, 163.2 (d, *J* = 248.7 Hz), 159.2, 131.0, 130.8 (d, *J* = 3.7 Hz), 130.1, 129.7, 129.6 (d, *J* = 8.5 Hz), 129.5, 129.0, 128.9, 128.7, 128.4, 127.0, 116.1 (d, *J* = 21.8 Hz), 82.9; **^{19}F NMR** (376 MHz, $CDCl_3$): δ = -111.5; **FTIR** (neat) υ 1750, 1508, 1226, 1154, 1009, 837, 784, 697 cm^{-1}; **HRMS** (ESI) calcd for $C_{22}H_{15}FO_2[M + Na]^+$: 353.0948, found: 353.0946.

5-(4-chlorophenyl)-3,4-diphenylfuran-2(5*H*)-one (3l)[3]

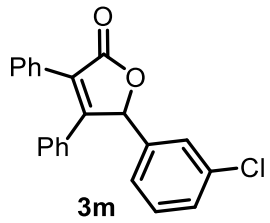


3l

Following the general procedure, the reaction of 2,3-diphenylcycloprop-2-en-1-one **1a** (61.7 mg, 0.3 mmol) and 4-chlorobenzaldehyde **2l** (28.3 mg, 0.2 mmol) with PMe_3 (1 M in THF, 20 μL, 0.02 mmol) was conducted for 2 hours which produced **3l** in 46.5 mg, 67% yield, as white solid.

^{1}H NMR (400 MHz, $CDCl_3$): δ = 7.50 – 7.44 (m, 2H), 7.38 – 7.33 (m, 3H), 7.31 – 7.27 (m, 3H), 7.25 – 7.20 (m, 4H), 7.14 – 7.08 (m, 2H), 6.25 (s, 1H).

5-(3-chlorophenyl)-3,4-diphenylfuran-2(5*H*)-one (3m)

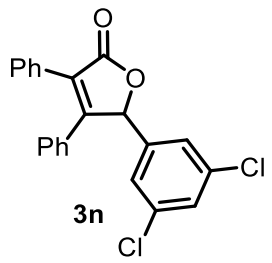


3m

Following the general procedure, the reaction of 2,3-diphenylcycloprop-2-en-1-one **1a** (62.2 mg, 0.3 mmol) and 3-chlorobenzaldehyde **2m** (23 μL, 0.2 mmol) with PMe_3 (1 M in THF, 20 μL, 0.02 mmol) was conducted for 1 hour which produced **3m** in 57.6 mg, 83% yield, as pale-yellow solid, m.p. 111 – 114 °C.

^{1}H NMR (400 MHz, $CDCl_3$): δ = 7.50 – 7.45 (m, 2H), 7.38 – 7.34 (m, 3H), 7.32 – 7.27 (m, 3H), 7.26 – 7.21 (m, 3H), 7.16 (dt, *J* = 7.2, 1.4 Hz, 1H), 7.14 – 7.09 (m, 2H), 6.23 (s, 1H); **^{13}C NMR** (100 MHz, $CDCl_3$): δ = 172.3, 159.0, 136.9, 134.9, 130.8, 130.3, 130.2, 129.7, 129.6, 129.5, 129.1, 129.0, 128.7, 128.4, 127.7, 127.1, 125.9, 82.8; **FTIR** (neat) υ 2978, 1751, 1399, 1241, 1067, 878, 692 cm^{-1}; **HRMS** (ESI) calcd for $C_{22}H_{15}ClO_2[M + Na]^+$: 369.0653, found: 369.0654.

5-(3,5-dichlorophenyl)-3,4-diphenylfuran-2(5*H*)-one (3n)

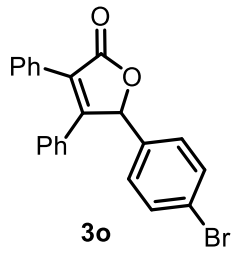


3n

Following the general procedure, the reaction of 2,3-diphenylcycloprop-2-en-1-one **1a** (61.9 mg, 0.3 mmol) and 3,5-dichlorobenzaldehyde **2n** (35.0 mg, 0.2 mmol) with PMe_3 (1 M in THF, 20 µL, 0.02 mmol) was conducted for 1 hour which produced **3n** in 41.9 mg, 55% yield, as pale-yellow solid, m.p. 130 – 133 °C.

^{1}H NMR (400 MHz, $CDCl_3$): δ = 7.49 – 7.45 (m, 2H), 7.38 – 7.26 (m, 7H), 7.17 – 7.11 (m, 4H), 6.20 (s, 1H); **^{13}C NMR** (100 MHz, $CDCl_3$): δ = 172.0, 158.5, 138.4, 135.6, 130.54, 130.48, 129.7, 129.5, 129.34, 129.28, 129.2, 128.8, 128.3, 127.3, 126.1, 82.0; **FTIR** (neat) υ 2982, 1750, 1571, 1432, 1150, 1023, 963, 691 cm^{-1}; **HRMS** (ESI) calcd for $C_{22}H_{14}Cl_2O_2[M + Na]^+$: 403.0263, found: 403.0260.

5-(4-bromophenyl)-3,4-diphenylfuran-2(5*H*)-one (3o)



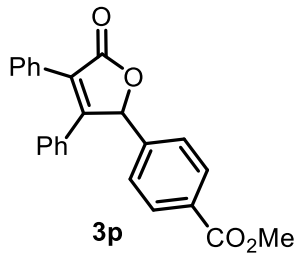
3o

Following the general procedure, the reaction of 2,3-diphenylcycloprop-2-en-1-one **1a** (62.4 mg, 0.3 mmol) and 4-bromobenzaldehyde **2o** (37.1 mg, 0.2 mmol) with PMe_3 (1 M in THF, 20 µL, 0.02 mmol) was conducted for 2 hours which produced **3o** in 58.7 mg, 75% yield, as white solid, m.p. 133 – 136 °C.

^{1}H NMR (400 MHz, $CDCl_3$): δ = 7.50 – 7.41 (m, 4H), 7.37 – 7.35 (m, 3H), 7.32 – 7.27 (m, 1H), 7.25 – 7.20 (m, 2H), 7.16 (dd, *J* = 8.8, 2.0 Hz, 2H), 7.12 – 7.08 (m, 2H), 6.24 (s, 1H); **^{13}C NMR** (100 MHz, $CDCl_3$): δ = 172.3, 159.1, 134.0, 132.3, 130.9, 130.2, 129.7, 129.5, 129.3, 129.1,

129.0, 128.7, 128.4, 127.1, 123.6, 82.9; **FTIR** (neat) υ 1748, 1487, 1299, 1154, 1002, 961, 691 cm^{-1}; **HRMS** (ESI) calcd for $C_{22}H_{15}BrO_2[M + Na]^+$: 413.0147, found: 413.0152.

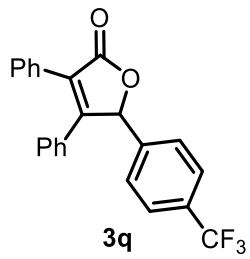
methyl 4-(5-oxo-3,4-diphenyl-2,5-dihydrofuran-2-yl) benzoate (3p)



Following the general procedure, the reaction of 2,3-diphenylcycloprop-2-en-1-one **1a** (61.8 mg, 0.3 mmol) and methyl 4-formylbenzoate **2p** (32.7 mg, 0.2 mmol) with PMe_3 (1 M in THF, 20 µL, 0.02 mmol) was conducted for 30 minutes which produced **3p** in 53.2 mg, 72% yield, as white solid, m.p. 128 – 131 °C.

^{1}H NMR (400 MHz, $CDCl_3$): δ = 8.00 – 7.96 (m, 2H), 7.50 – 7.45 (m, 2H), 7.38 – 7.33 (m, 5H), 7.31 – 7.26 (m, 1H), 7.25 – 7.19 (m, 2H), 7.12 – 7.06 (m, 2H), 6.31 (s, 1H), 3.89 (s, 3H); **^{13}C NMR** (100 MHz, $CDCl_3$): δ = 172.4, 166.5, 159.3, 139.8, 131.2, 130.9, 130.3, 130.2, 129.6, 129.5, 129.2, 129.0, 128.7, 128.3, 127.6, 127.0, 83.0, 52.4; **FTIR** (neat) υ 2980, 2372, 1755, 1402, 1250, 1068, 868 cm^{-1}; **HRMS** (ESI) calcd for $C_{24}H_{18}O_4[M + Na]^+$: 393.1097, found: 393.1104.

3,4-diphenyl-5-(4-(trifluoromethyl) phenyl) furan-2(5*H*)-one (3q)[2]

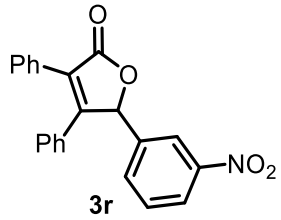


Following the general procedure, the reaction of 2,3-diphenylcycloprop-2-en-1-one **1a** (62.3 mg, 0.3 mmol) and 4-(trifluoromethyl) benzaldehyde **2q** (28 µL, 0.2 mmol) with PMe_3 (1 M in

THF, 20 μL, 0.02 mmol) was conducted for 30 minutes which produced **3q** in 44.2 mg, 58% yield, as white solid.

^{1}H NMR (400 MHz, $CDCl_3$): δ = 7.57 (d, *J* = 8.2 Hz, 2H), 7.49 – 7.45 (m, 2H), 7.40 (d, *J* = 8.1 Hz, 2H), 7.37 – 7.33 (m, 3H), 7.32 – 7.27 (m, 1H), 7.26 – 7.21 (m, 2H), 7.14 – 7.09 (m, 2H), 6.34 (s, 1H).

5-(3-nitrophenyl)-3,4-diphenylfuran-2(5*H*)-one (3r)

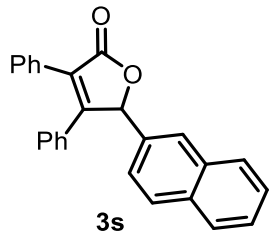


3r

Following the general procedure, the reaction of 2,3-diphenylcycloprop-2-en-1-one **1a** (61.9 mg, 0.3 mmol) and 3-nitrobenzaldehyde **2r** (30.4 mg, 0.2 mmol) with PMe_3 (1 M in THF, 10 μL, 0.01 mmol) was conducted for 30 minutes which produced **3r** in 46.6 mg, 65% yield, as pale-yellow solid, m.p. 122 – 125 °C.

^{1}H NMR (400 MHz, $CDCl_3$): δ = 8.20 – 8.13 (m, 2H), 7.62 (dt, J = 7.7, 1.4 Hz, 1H), 7.53 – 7.47 (m, 3H), 7.37 – 7.34 (m, 3H), 7.32 – 7.28 (m, 1H), 7.27 – 7.22 (m, 2H), 7.16 – 7.13 (m, 2H), 6.40 (s, 1H); **^{13}C NMR** (100 MHz, $CDCl_3$): δ = 172.0, 158.7, 148.5, 137.3, 133.4, 130.53, 130.49, 130.2, 129.5, 129.32, 129.27, 129.2, 128.8, 128.3, 127.4, 124.4, 122.8, 82.2; **FTIR** (neat) υ 2979, 1749, 1528, 1442, 1346, 1153, 962, 690 cm^{-1}; **HRMS** (ESI) calcd for $C_{22}H_{15}NO_4$[M + Na]$^+$: 380.0893, found: 380.0887.

5-(naphthalen-2-yl)-3,4-diphenylfuran-2(5*H*)-one (3s)

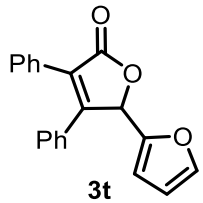


3s

Following the general procedure, the reaction of 2,3-diphenylcycloprop-2-en-1-one **1a** (61.7 mg, 0.3 mmol) and 2-naphthaldehyde **2s** (31.3 mg, 0.2 mmol) with PMe_3 (1 M in THF, 20 μL, 0.02 mmol) was conducted for 1 hour which produced **3s** in 63.8 mg, 88% yield, as white solid, m.p. 140 – 143 °C.

^{1}H NMR (400 MHz, $CDCl_3$): δ = 7.83 – 7.77 (m, 4H), 7.54 – 7.44 (m, 4H), 7.39 – 7.34 (m, 3H), 7.31 (dd, *J* = 8.6, 1.7 Hz, 1H), 7.23 – 7.10 (m, 5H), 6.42 (s, 1H); **^{13}C NMR** (100 MHz, $CDCl_3$): δ = 172.7, 159.5, 133.7, 133.2, 132.2, 131.2, 130.0, 129.9, 129.6, 129.2, 129.0, 128.8, 128.7, 128.4, 128.22, 128.21, 127.9, 127.1, 126.9, 126.7, 123.9, 84.0; **FTIR** (neat) υ 1759, 1444, 1270, 1149, 1009, 960, 695 cm^{-1}; **HRMS** (ESI) calcd for $C_{26}H_{18}O_2[M + Na]^+$: 385.1199, found: 385.1196.

3,4-diphenyl-[2,2'-bifuran]-5(2*H*)-one (3t)

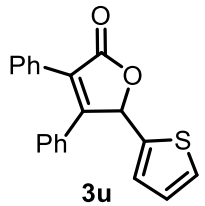


3t

Following the general procedure, the reaction of 2,3-diphenylcycloprop-2-en-1-one **1a** (62.0 mg, 0.3 mmol) and furan-2-carbaldehyde **2t** (17 μL, 0.2 mmol) with PMe_3 (1 M in THF, 10 μL, 0.01 mmol) was conducted for 40 minutes which produced **3t** in 59.3 mg, 98% yield, as pale-yellow solid, m.p. 112 – 115 °C.

^{1}H NMR (400 MHz, $CDCl_3$): δ = 7.49 – 7.43 (m, 2H), 7.37 – 7.33 (m, 4H), 7.32 – 7.27 (m, 1H), 7.25 – 7.20 (m, 2H), 7.17 – 7.13 (m, 2H), 6.39 (dd, *J* = 3.4, 0.7 Hz, 1H), 6.33 (s, 1H), 6.29 (dd, *J* = 3.3, 1.9 Hz, 1H); **^{13}C NMR** (100 MHz, $CDCl_3$): δ = 172.0, 156.0, 147.3, 143.9, 130.9, 130.2, 129.8, 129.5, 129.0, 128.8, 128.6, 128.2, 127.7, 111.8, 110.8, 76.3; **FTIR** (neat) υ 1748, 1443, 1287, 1153, 1063, 963, 742, 692 cm^{-1}; **HRMS** (ESI) calcd for $C_{20}H_{14}O_3[M + Na]^+$: 325.0835, found: 325.0839.

3,4-diphenyl-5-(thiophen-2-yl) furan-2(5*H*)-one (3u)

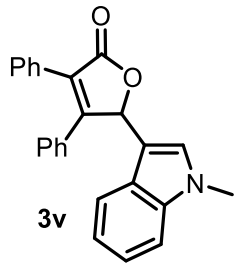


3u

Following the general procedure, the reaction of 2,3-diphenylcycloprop-2-en-1-one **1a** (62.1 mg, 0.3 mmol) and thiophene-2-carbaldehyde **2u** (19 μL, 0.2 mmol) with PMe_3 (1 M in THF, 10 μL, 0.01 mmol) was conducted for 2 hours which produced **3u** in 59.7 mg, 94% yield, as pale-yellow solid, m.p. 118 – 121 °C.

^{1}H NMR (400 MHz, $CDCl_3$): δ = 7.48 – 7.44 (m, 2H), 7.38 – 7.34 (m, 3H), 7.32 – 7.24 (m, 3H), 7.23 – 7.17 (m, 3H), 7.10 – 7.07 (m, 1H), 6.92 (dd, *J* = 5.1, 3.6 Hz, 1H), 6.57 (s, 1H); **^{13}C NMR** (100 MHz, $CDCl_3$): δ = 171.8, 158.3, 137.8, 131.0, 130.3, 129.8, 129.5, 129.1, 128.9, 128.7, 128.52, 128.46, 127.5, 127.1(2C), 78.5; **FTIR** (neat) υ 2981, 1739, 1401, 1283, 1152, 1055, 699 cm^{-1}; **HRMS** (ESI) calcd for $C_{20}H_{14}O_2S[M + Na]^+$: 341.0607, found: 341.0607.

5-(1-methyl-1*H*-indol-3-yl)-3,4-diphenylfuran-2(5*H*)-one (3v)



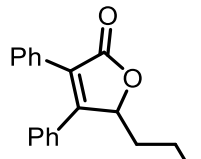
3v

Following the general procedure, the reaction of 2,3-diphenylcycloprop-2-en-1-one **1a** (61.8 mg, 0.3 mmol) and 1-methyl-1*H*-indole-3-carbaldehyde **2v** (32.0 mg, 0.2 mmol) with PMe_3 (1 M in THF, 40 μL, 0.04 mmol) was conducted for 2 hours which produced **3v** in 72.8 mg, 99% yield, as white solid, m.p. 152 – 155 °C.

^{1}H NMR (400 MHz, $CDCl_3$): δ = 7.64 – 7.59 (m, 1H), 7.53 – 7.49 (m, 2H), 7.39 – 7.34 (m, 3H), 7.22 – 7.17 (m, 5H), 7.15 – 7.06 (m, 3H), 6.94 (s, 1H), 6.58 (s, 1H), 3.57 (s, 3H); **^{13}C NMR** (100 MHz, $CDCl_3$): δ = 172.6, 158.8, 137.3, 131.6, 130.4, 129.9, 129.7, 129.5, 128.8, 128.7, 128.6, 128.5, 127.4, 126.4, 122.4, 120.2, 119.2, 109.8, 108.1, 78.2, 32.9; **FTIR** (neat) υ 2981,

1736, 1399, 1243, 1068, 740, 696 cm^{-1}; **HRMS** (ESI) calcd for $C_{25}H_{19}NO_2[M + Na]^+$: 388.1308, found: 388.1313.

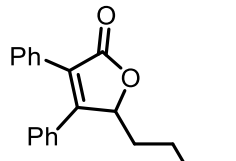
3,4-diphenyl-5-propylfuran-2(5*H*)-one (3w)[4]



3w

Following the general procedure, the reaction of 2,3-diphenylcycloprop-2-en-1-one **1a** (61.8 mg, 0.3 mmol) and butyraldehyde **2w** (18 μL, 0.2 mmol) with PMe_3 (1 M in THF, 20 μL, 0.02 mmol) was conducted for 7 hours which produced **3w** in 49.6 mg, 89% yield, as white solid, **^{1}H NMR** (400 MHz, $CDCl_3$): δ = 7.41 – 7.29 (m, 8H), 7.25 – 7.21 (m, 2H), 5.46 (dd, *J* = 7.4, 2.8 Hz, 1H), 1.88 – 1.74 (m, 1H), 1.56 – 1.43 (m, 3H), 0.90 (t, *J* = 7.2 Hz, 3H).

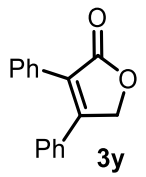
5-butyl-3,4-diphenylfuran-2(5*H*)-one (3x)[5]



3x

Following the general procedure, the reaction of 2,3-diphenylcycloprop-2-en-1-one **1a** (62.2 mg, 0.3 mmol) and pentanal **2x** (21 μL, 0.2 mmol) with PMe_3 (1 M in THF, 20 μL, 0.02 mmol) was conducted for 7 hours which produced **3x** in 51.0 mg, 87% yield, as pale-yellow solid.
^{1}H NMR (400 MHz, $CDCl_3$): δ = 7.41 – 7.29 (m, 8H), 7.25 – 7.20 (m, 2H), 5.46 (dd, *J* = 7.5, 3.3 Hz, 1H), 1.93 – 1.78 (m, 1H), 1.57 – 1.39 (m, 3H), 1.37 – 1.18 (m, 2H), 0.84 (t, *J* = 7.3 Hz, 3H).

3,4-diphenylfuran-2(5*H*)-one (3y)[6]

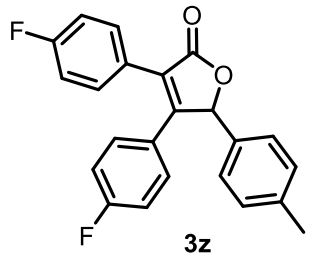


3y

Following the general procedure, the reaction of 2,3-diphenylcycloprop-2-en-1-one **1a** (62.3 mg, 0.3 mmol) and paraformaldehyde **2y** (6.1 mg, 0.2 mmol) with PMe_3 (1 M in THF, 20 μL, 0.02 mmol) was conducted for 12 hours which produced **3y** in 38.7 mg, 82% yield, as white solid.

^{1}H NMR (400 MHz, $CDCl_3$): δ = 7.47 – 7.29 (m, 10H), 5.18 (s, 2H).

3,4-*bis*(4-fluorophenyl)-5-(p-tolyl) furan-2(5*H*)-one (3z)

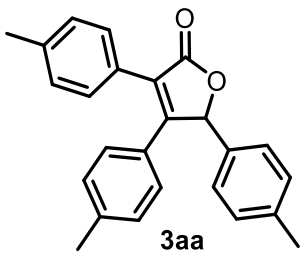


3z

Following the general procedure, the reaction of 2,3-bis(4-fluorophenyl) cycloprop-2-en-1-one **1b** (72.7 mg, 0.3 mmol) and 4-methylbenzaldehyde **2b** (24 μL, 0.2 mmol) with PMe_3 (1 M in THF, 20 μL, 0.02 mmol) was conducted for 3 hours which produced **3z** in 65.8 mg, 91% yield, as pale-yellow solid, m.p. 105 – 108 °C.

^{1}H NMR (400 MHz, $CDCl_3$): δ = 7.50 – 7.42 (m, 2H), 7.17 – 7.02 (m, 8H), 6.95 – 6.87 (m, 2H), 6.19 (s, 1H), 2.31 (s, 3H); **^{13}C NMR** (100 MHz, $CDCl_3$): δ = 172.4, 163.4 (d, *J* = 251.6 Hz), 163.1 (d, *J* = 249.5 Hz), 158.2, 139.7, 131.5 (d, *J* = 7.8 Hz), 131.4, 130.5 (d, *J* = 8.6 Hz), 129.9, 127.7, 127.2 (d, *J* = 3.8 Hz), 126.0, 125.8 (d, *J* = 3.6 Hz), 116.3 (d, *J* = 21.4 Hz), 116.0 (d, *J* = 22.1 Hz), 83.7, 21.4; **^{19}F NMR** (376 MHz, $CDCl_3$): δ = -109.1, -111.4; **FTIR** (neat) υ 2981, 2314, 1749, 1400, 1229, 1069, 868 cm^{-1}; **HRMS** (ESI) calcd for $C_{23}H_{16}F_2O_2[M + H]^+$: 363.1191, found: 363.1193.

3,4,5-tri-*p*-tolylfuran-2(5*H*)-one (3aa)

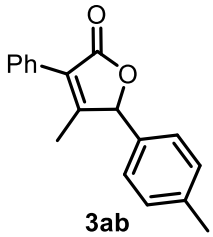


3aa

Following the general procedure, the reaction of 2,3-di-*p*-tolylcycloprop-2-en-1-one **1c** (70.3 mg, 0.3 mmol) and 4-methylbenzaldehyde **2b** (24 μL, 0.2 mmol) with PMe_3 (1 M in THF, 20 μL, 0.02 mmol) was conducted for 3 hours which produced **3aa** in 65.9 mg, 93% yield, as pale-yellow solid, m.p. 120 – 123 °C.

^{1}H NMR (400 MHz, $CDCl_3$): δ = 7.40 – 7.37 (m, 2H), 7.19 – 7.14 (m, 4H), 7.11 (d, *J* = 8.0 Hz, 2H), 7.06 – 6.97 (m, 4H), 6.20 (s, 1H), 2.36 (s, 3H), 2.29 (s, 3H), 2.25 (s, 3H); **^{13}C NMR** (100 MHz, $CDCl_3$): δ = 173.0, 158.7, 140.2, 139.3, 138.8, 132.2, 129.7, 129.5, 129.4(2C), 128.5, 128.4, 127.8, 127.3, 126.3, 83.6, 21.52, 21.50, 21.4; **FTIR** (neat) υ 2980, 1746, 1402, 1253, 1153, 1068, 816 cm^{-1}; **HRMS** (ESI) calcd for $C_{25}H_{22}O_2$[M + Na]$^+$: 377.1512, found: 377.1512.

4-methyl-3-phenyl-5-(*p*-tolyl) furan-2(5*H*)-one (3ab)[7]

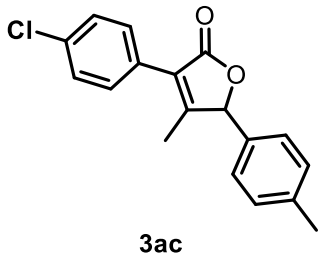


3ab

Following the general procedure, the reaction of 2-methyl-3-phenylcycloprop-2-en-1-one **1d** (43.4 mg, 0.3 mmol) and 4-methylbenzaldehyde **2b** (24 μL, 0.2 mmol) with PMe_3 (1 M in THF, 20 μL, 0.02 mmol) was conducted for 3 hours which produced **3ab** in 47.0 mg, 89% yield, as pale-yellow solid.

^{1}H NMR (400 MHz, $CDCl_3$): δ = 7.57 – 7.53 (m, 2H), 7.48 – 7.43 (m, 2H), 7.42 – 7.36 (m, 1H), 7.22 (d, *J* = 8.0 Hz, 2H), 7.17 (d, *J* = 8.2 Hz, 2H), 5.72 (s, 1H), 2.38 (s, 3H), 2.00 (s, 3H).

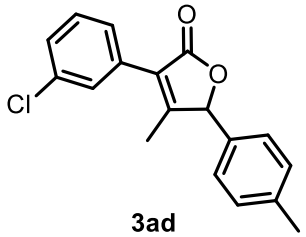
3-(4-chlorophenyl)-4-methyl-5-(*p*-tolyl)furan-2(*5H*)-one (3ac)



3ac

Following the general procedure, the reaction of 2-(4-chlorophenyl)-3-methylcycloprop-2-en-1-one **1e** (53.5 mg, 0.3 mmol) and 4-methylbenzaldehyde **2b** (24 μL, 0.2 mmol) with PMe_3 (1 M in THF, 20 μL, 0.02 mmol) was conducted for 6 hours which produced **3ac** in 54.4 mg, 91% yield, as pale-yellow solid, m.p. 126 – 128 °C.

^{1}H NMR (600 MHz, $CDCl_3$): δ = 7.50 (d, *J* = 8.3 Hz, 2H), 7.42 (d, *J* = 8.3 Hz, 2H), 7.21 (d, *J* = 7.8 Hz, 2H), 7.15 (d, *J* = 7.8 Hz, 2H), 5.71 (s, 1H), 2.37 (s, 3H), 1.99 (s, 3H); **^{13}C NMR** (150 MHz, $CDCl_3$): δ = 172.6, 161.1, 139.7, 134.7, 131.5, 130.4, 129.9, 128.9, 128.4, 127.1, 125.5, 85.0, 21.4, 13.6; **FTIR** (neat) υ 2921, 1750, 1490, 1137, 1089, 970 cm^{-1}; **HRMS** (ESI) calcd for $C_{18}H_{16}ClO_2[M + Na]^+$: 321.0653, found: 321.0652.

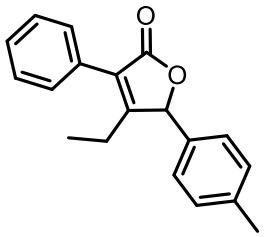


3ad

Following the general procedure, the reaction of 2-(3-chlorophenyl)-3-methylcycloprop-2-en-1-one **1f** (53.5 mg, 0.3 mmol) and 4-methylbenzaldehyde **2b** (24 μL, 0.2 mmol) with PMe_3 (1 M in THF, 20 μL, 0.02 mmol) was conducted for 4 hours which produced **3ad** in 55.0 mg, 92% yield, as yellow liquid.

^{1}H NMR (400 MHz, $CDCl_3$): δ = 7.54 (t, *J* = 1.8 Hz, 1H), 7.44 (dt, *J* = 7.0, 1.9 Hz, 1H), 7.41 – 7.36 (m, 2H), 7.22 (d, *J* = 8.1 Hz, 2H), 7.17 – 7.14 (m, 2H), 5.72 (s, 1H), 2.37 (s, 3H), 2.00 (d, *J* = 1.0 Hz, 3H); **^{13}C NMR** (100 MHz, $CDCl_3$): δ = 172.4, 161.7, 139.7, 134.6, 131.8, 131.5, 129.9(2C), 129.1, 128.8, 127.3, 127.1, 125.4, 85.0, 21.4, 13.5; **FTIR** (neat) υ 2921, 1751, 1412, 1210, 1139, 989, 731 cm^{-1}; **HRMS** (ESI) calcd for $C_{18}H_{16}ClO_2[M + H]^+$: 299.0842, found: 299.0833.

4-ethyl-3-phenyl-5-(*p*-tolyl)furan-2(*5H*)-one (3ae)

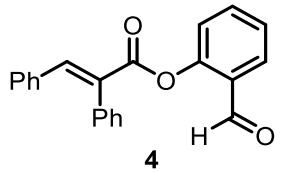


3ae and regioisomer (1.5:1)

Following the general procedure, the reaction of 2-ethyl-3-phenylcycloprop-2-en-1-one **1g** (47.5 mg, 0.3 mmol) and 4-methylbenzaldehyde **2b** (24 µL, 0.2 mmol) with PMe_3 (1 M in THF, 20 µL, 0.02 mmol) was conducted for 24 hours which produced **3ae** as slightly yellow liquid, in 28.5 mg, 51% yield as a pair of regioisomers (1.5:1).

^{1}H NMR (600 MHz, $CDCl_3$, major): δ = 7.53 – 7.49 (m, 2H), 7.45 (t, *J* = 7.6 Hz, 2H), 7.41 – 7.37 (m, 1H), 7.22 (d, *J* = 7.8 Hz, 2H), 7.18 (d, *J* = 7.8 Hz, 2H), 5.85 (s, 1H), 2.65 (dq, *J* = 15.0, 7.5 Hz, 1H), 2.37 (s, 3H), 2.14 (dq, *J* = 15.1, 7.6 Hz, 1H), 1.05 (t, *J* = 7.6 Hz, 3H); **^{1}H NMR** (600 MHz, $CDCl_3$, selected signals for the minor): 7.08 (s, 4H), 6.11 (s, 1H), 2.60 – 2.49 (m, 2H), 2.28 (s, 3H), 1.26 (t, *J* = 7.5 Hz, 3H); **^{13}C NMR** (150 MHz, $CDCl_3$, major): δ = 173.1, 165.8, 139.6, 131.8, 130.1, 129.9, 129.1, 128.70, 128.67, 127.3, 126.4, 83.5, 21.4, 20.6, 12.6; **^{13}C NMR** (150 MHz, $CDCl_3$, minor): δ = 174.2, 158.6, 139.2, 132.1, 131.7, 129.9, 129.7, 129.6, 128.9, 128.0, 127.5, 83.7, 21.3, 18.0, 13.1. **FTIR** (neat) υ 3020, 1747, 1214, 746 cm^{-1}; **HRMS** (ESI) calcd for $C_{19}H_{18}O_2[M + H]^+$: 279.1380, found: 279.1387.

2-formylphenyl (*E*)-2,3-diphenylacrylate (4)[8]

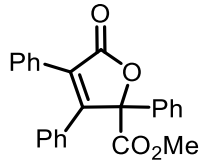


4

Following the general procedure, the reaction of 2,3-diphenylcycloprop-2-en-1-one **1a** (61.8 mg, 0.3 mmol) and salicylaldehyde **2z** (22 μL, 0.2 mmol) with PMe_3 (1 M in THF, 20 μL, 0.02 mmol) was conducted for 12 hours which produced **4** in 45.2 mg, 69% yield, as white solid.

^{1}H NMR (400 MHz, $CDCl_3$): δ = 10.06 (s, 1H), 8.08 (s, 1H), 7.88 (dd, *J* = 7.7, 1.7 Hz, 1H), 7.61 (td, *J* = 7.8, 1.8 Hz, 1H), 7.45 – 7.35 (m, 6H), 7.29 (d, *J* = 8.3 Hz, 2H), 7.19 (t, *J* = 7.6 Hz, 2H), 7.12 (d, *J* = 7.1 Hz, 2H).

methyl 5-oxo-2,3,4-triphenyl-2,5-dihydrofuran-2-carboxylate (6a)

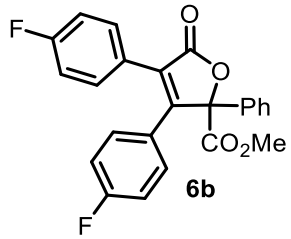


6a

Following the general procedure, the reaction of 2,3-diphenylcycloprop-2-en-1-one **1a** (62.0 mg, 0.3 mmol) and methyl 2-oxo-2-phenylacetate **5a** (29 μL, 0.2 mmol) with PMe_3 (1 M in THF, 20 μL, 0.02 mmol) was conducted for 2 hours which produced **6a** in 71.8 mg, 97% yield, as pale-yellow solid, m.p. 135 – 138 °C.

^{1}H NMR (400 MHz, $CDCl_3$): δ = 7.42 – 7.38 (m, 2H), 7.38 – 7.32 (m, 5H), 7.32 – 7.26 (m, 3H), 7.26 – 7.21 (m, 3H), 6.96 – 6.91 (m, 2H), 3.84 (s, 3H); **^{13}C NMR** (100 MHz, $CDCl_3$): δ = 171.2, 167.7, 159.7, 134.0, 131.2, 129.8, 129.5(2C), 129.2(2C), 128.6(3C), 128.5, 128.0, 127.1, 89.6, 53.7; **FTIR** (neat) υ 1745, 1442, 1260, 1162, 1004, 967, 692 cm^{-1}; **HRMS** (ESI) calcd for $C_{24}H_{18}O_4[M + Na]^+$: 393.1097, found: 393.1096.

methyl 3,4-*bis*(4-fluorophenyl)-5-oxo-2-phenyl-2,5-dihydrofuran-2-carboxylate (6b)



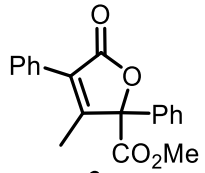
6b

Following the general procedure, the reaction of 2,3-*bis*(4-fluorophenyl) cycloprop-2-en-1-one **1b** (72.6 mg, 0.3 mmol) and methyl 2-oxo-2-phenylacetate **5a** (29 μL, 0.2 mmol) with PMe_3 (1

M in THF, 20 μL, 0.02 mmol) was conducted for 3 hours which produced **6b** in 73.9 mg, 91% yield, as pale-yellow solid, m.p. 145 – 148 °C.

^{1}H NMR (400 MHz, $CDCl_3$): δ = 7.45 – 7.31 (m, 7H), 7.04 – 6.90 (m, 6H), 3.84 (s, 3H); **^{13}C NMR** (100 MHz, $CDCl_3$): δ = 170.9, 167.6, 163.4 (d, *J* = 251.7 Hz), 163.1 (d, *J* = 250.4 Hz), 158.6, 133.8, 131.5 (d, *J* = 8.6 Hz), 131.3 (d, *J* = 8.6 Hz), 129.7, 128.8, 127.3, 127.0 (d, *J* = 3.9 Hz), 126.9, 125.0 (d, *J* = 3.7 Hz), 116.0 (d, *J* = 20.4 Hz), 115.8 (d, *J* = 21.1 Hz), 89.7, 53.8; **^{19}F NMR** (376 MHz, $CDCl_3$): δ = -109.8, -110.7; **FTIR** (neat) υ 1746, 1440, 1258, 1004, 969, 695 cm^{-1}; **HRMS** (ESI) calcd for $C_{24}H_{16}F_2O_4[M + Na]^+$: 429.0909, found: 429.0904.

methyl 3-methyl-5-oxo-2,4-diphenyl-2,5-dihydrofuran-2-carboxylate (6c)

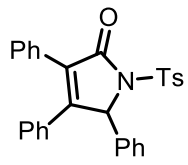


6c

Following the general procedure, the reaction of 2-methyl-3-phenylcycloprop-2-en-1-one **1d** (43.5 mg, 0.3 mmol) and methyl 2-oxo-2-phenylacetate **5a** (29 μL, 0.2 mmol) with PMe_3 (1 M in THF, 20 μL, 0.02 mmol) was conducted for 3 hours which produced **6c** in 60.4 mg, 98% yield, as pale-yellow solid, m.p. 105 – 108 °C.

^{1}H NMR (400 MHz, $CDCl_3$): δ = 7.54 – 7.50 (m, 2H), 7.47 – 7.36 (m, 8H), 3.88 (s, 3H), 2.25 (s, 3H); **^{13}C NMR** (100 MHz, $CDCl_3$): δ = 171.2, 167.9, 159.6, 134.6, 129.5, 129.3, 129.2, 129.1, 129.0, 128.6, 127.5, 126.0, 89.7, 53.6, 13.8; **FTIR** (neat) υ 1761, 1440, 1241, 1178, 1063, 970, 695 cm^{-1}; **HRMS** (ESI) calcd for $C_{19}H_{16}O_4[M + Na]^+$: 331.0941, found: 331.0947.

3,4,5-triphenyl-1-tosyl-1,5-dihydro-2*H*-pyrrol-2-one (8a)[9]

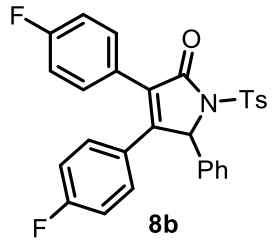


8a

Following the general procedure, the reaction of 2,3-diphenylcycloprop-2-en-1-one **1a** (62.2 mg, 0.3 mmol) and N-benzylidene-4-methylbenzenesulfonamide **7a** (51.8 mg, 0.2 mmol) with PMe_3 (1 M in THF, 40 µL, 0.04 mmol) was conducted for 20 hours which produced **8a** in 81.9 mg, 88% yield, as pale-yellow solid.

^{1}H NMR (400 MHz, $CDCl_3$): δ = 7.43 – 7.40 (m, 2H), 7.35 (dd, *J* = 6.7, 3.0 Hz, 2H), 7.29 – 7.26 (m, 3H), 7.25 – 7.16 (m, 6H), 7.14 – 7.07 (m, 4H), 7.03 – 6.99 (m, 2H), 6.12 (s, 1H), 2.34 (s, 3H).

3,4-*bis*(4-fluorophenyl)-5-phenyl-1-tosyl-1,5-dihydro-2*H*-pyrrol-2-one (8b)

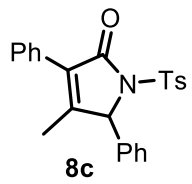


8b

Following the general procedure, the reaction of 2,3-*bis*(4-fluorophenyl) cycloprop-2-en-1-one **1b** (72.7 mg, 0.3 mmol) and N-benzylidene-4-methylbenzenesulfonamide **7a** (51.9 mg, 0.2 mmol) with PMe_3 (1 M in THF, 40 µL, 0.04 mmol) was conducted for 24 hours which produced **8b** in 71.3 mg, 71% yield, as pale-yellow solid, m.p. 163 – 165 °C.

^{1}H NMR (400 MHz, $CDCl_3$): δ = 7.40 (d, *J* = 8.4 Hz, 2H), 7.37 – 7.31 (m, 2H), 7.28 – 7.20 (m, 3H), 7.19 – 7.14 (m, 2H), 7.10 (d, *J* = 8.2 Hz, 2H), 7.03 – 6.94 (m, 4H), 6.89 – 6.81 (m, 2H), 6.10 (s, 1H), 2.35 (s, 3H); **^{13}C NMR** (100 MHz, $CDCl_3$): δ = 167.8, 163.2 (d, *J* = 251.5 Hz), 162.9 (d, *J* = 249.4 Hz), 154.6, 144.8, 135.7, 134.4, 131.6 (d, *J* = 8.2 Hz), 130.7 (d, *J* = 8.6 Hz), 130.0, 129.3, 129.0, 128.9, 128.4, 128.1, 127.6 (d, *J* = 3.6 Hz), 125.8 (d, *J* = 3.5 Hz), 116.1 (d, *J* = 21.5 Hz), 115.7 (d, *J* = 21.4 Hz), 66.7, 21.7; **^{19}F NMR** (376 MHz, $CDCl_3$): δ = -109.5, -111.6; **FTIR** (neat) υ 1703, 1504, 1352, 1227, 1166, 843, 668, 576 cm^{-1}; **HRMS** (ESI) calcd for $C_{29}H_{21}F_2NO_3S[M + Na]^+$: 524.1102, found: 524.1106.

4-methyl-3,5-diphenyl-1-tosyl-1,5-dihydro-2*H*-pyrrol-2-one (8c)



8c

Following the general procedure, the reaction of 2-methyl-3-phenylcycloprop-2-en-1-one **1d** (43.4 mg, 0.3 mmol) and N-benzylidene-4-methylbenzenesulfonamide **7a** (51.8 mg, 0.2 mmol) with PMe_3 (1 M in THF, 40 μL, 0.04 mmol) was conducted for 24 hours which produced **8c** in 69.4 mg, 86% yield, as white solid, m.p. 150 – 153 °C.

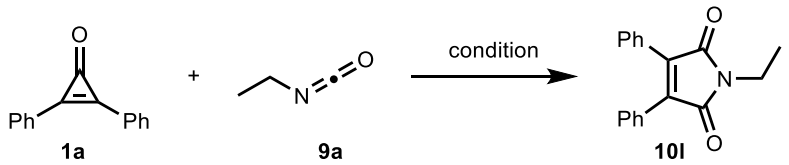
^{1}H NMR (400 MHz, $CDCl_3$): δ = 7.47 – 7.29 (m, 10H), 7.14 (d, *J* = 7.0 Hz, 2H), 7.09 (d, *J* = 8.0 Hz, 2H), 5.59 (s, 1H), 2.34 (s, 3H), 1.89 (s, 3H); **^{13}C NMR** (100 MHz, $CDCl_3$): δ = 168.3, 156.2, 144.6, 135.9, 134.9, 130.7, 129.8, 129.3, 129.2, 129.0(2C), 128.6, 128.4, 128.1, 128.0, 68.1, 21.7, 14.0; **FTIR** (neat) υ 2980, 2374, 1400, 1239, 1069, 870 cm^{-1}; **HRMS** (ESI) calcd for $C_{24}H_{21}NO_3S[M + Na]^+$: 426.1134, found: 426.1140.

3. Synthesis of Compounds 10, 12 and Analytical Data

3.1 Optimization of Reaction Conditions

Cyclopropenone **1a** and ethyl isocyanate **9a** were selected as the model substrate for the optimization of conditions (Table S1). Among various phosphine catalysts screened, only tributyl phosphine and trimethyl phosphine were effective to deliver the product maleimide **10l** in 10% and 36% yield, respectively (entries 1-8). Using PMe_3 as the catalyst, it was found that the addition of 4Å MS could upgrade the yield to 69% (entry 8). Examination of common solvents indicated that DCM remained the best (entries 9-13). Reducing the catalyst loading to 10 mol % decreased the yield to 49%, while increasing the amount of **1a** to 1.5 equiv could enhance the yield to 83% (entries 14 and 15). The reaction was found to be influenced by temperature, as running the reaction at 0 °C significantly reduced the yield (entry 16).

Table S1. Investigation of reaction conditions.[a]



entry	catalyst	additive	solvent	yield[b](%)
1	PPh_3		DCM	N.D.
2	$P(4\text{-}FC_6H_4)_3$		DCM	N.D.
3	$P(4\text{-}CH_3C_6H_4)_3$		DCM	N.D.
4	dppb		DCM	N.D.
5	P^nBu_3		DCM	10
6	P^tBu_3		DCM	trace
7	PCy_3		DCM	trace
8	PMe_3		DCM	36
9	PMe_3	4Å MS	DCM	69
10	PMe_3	4Å MS	CH_3CN	11
11	PMe_3	4Å MS	toluene	68
12	PMe_3	4Å MS	pentane	36
13	PMe_3	4Å MS	THF	49
14[c]	PMe_3	4Å MS	DCM	49
15[d]	PMe_3	4Å MS	DCM	83
16[e]	PMe_3	4Å MS	DCM	15

[a] Reaction condition: Under N_2 atmosphere, the catalyst (20 mol %) was added to a mixture of **1a** (0.2 mmol), **9a** (0.2 mmol) in specified solvent (2.0 mL) in a Schlenk tube, and then the mixture was stirred at room temperature for 24 hours. [b] Isolated yield. [c] 10 mol % catalyst loading. [d] 0.3 mmol **1a** was adopted. [e] The reaction was carried out at 0 °C.

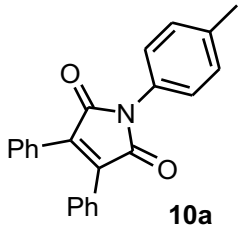
3.2 General Procedure for the Synthesis of Compounds 10 and 12

Under N_2 atmosphere, PMe_3 (1M in THF, 40 μL, 0.04 mmol) was added to a dispersion of cyclopropenone (0.3 mmol), 4 Å MS (100 mg), and isocyanates or carbodiimides (0.2 mmol) in anhydrous DCM (2 mL). The mixture was stirred at room temperature for 24 hours, and then the solvent was removed in *vacuo*. The residue was purified by column chromatography (for maleimides **10** and iminomaleimides **12**, silica gel and alumina were used as the stationary

phase, respectively) with petroleum ether/ethyl acetate (20/1) as eluent to provide the corresponding products **10** and **12**. Compounds **10a**–**c, 10f, 10h**–**q** are known, and the analytical data are consistent with those reported.[10–13]

3.2 Analytical Data for Compounds 10 and 12

3,4-diphenyl-1-(*p*-tolyl)-1*H*-pyrrole-2,5-dione (10a)[10]

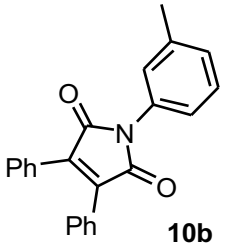


10a

Following the general procedure, the reaction of 2,3-diphenylcycloprop-2-en-1-one **1a** (62.2 mg, 0.3 mmol) and 1-isocyanato-4-methylbenzene **9a** (25 μL, 0.2 mmol) with PMe_3 (1 M in THF, 40 μL, 0.04 mmol) was conducted for 24 hours which produced **10a** in 40.7 mg, 60% yield, as yellow solid.

^{1}H NMR (400 MHz, $CDCl_3$): δ = 7.52 (dd, *J* = 7.9, 1.5 Hz, 4H), 7.42 – 7.31 (m, 7H), 7.29 – 7.22 (m, 2H), 7.18 (d, *J* = 7.5 Hz, 1H), 2.40 (s, 3H).

3,4-diphenyl-1-(*m*-tolyl)-1*H*-pyrrole-2,5-dione (10b)[10]

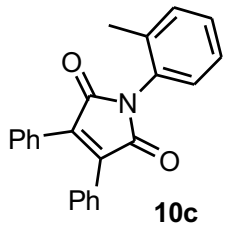


10b

Following the general procedure, the reaction of 2,3-diphenylcycloprop-2-en-1-one **1a** (61.8 mg, 0.3 mmol) and 1-isocyanato-3-methylbenzene **9b** (26 μL, 0.2 mmol) with PMe_3 (1 M in THF, 40 μL, 0.04 mmol) was conducted for 24 hours which produced **10b** in 41.5 mg, 61% yield, as yellow solid.

^{1}H NMR (400 MHz, $CDCl_3$): δ = 7.57 – 7.51 (m, 4H), 7.42 – 7.28 (m, 9H), 7.23 (d, *J* = 7.1 Hz, 1H), 2.26 (s, 3H).

3,4-diphenyl-1-(*o*-tolyl)-1*H*-pyrrole-2,5-dione (10c)[10]

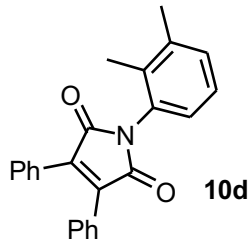


10c

Following the general procedure, the reaction of 2,3-diphenylcycloprop-2-en-1-one **1a** (62.0 mg, 0.3 mmol) and 1-isocyanato-2-methylbenzene **9c** (25 μL, 0.2 mmol) with PMe_3 (1 M in THF, 40 μL, 0.04 mmol) was conducted for 24 hours which produced **10c** in 55.1 mg, 81% yield, as yellow solid.

^{1}H NMR (400 MHz, $CDCl_3$): δ = 7.54 – 7.49 (m, 4H), 7.42 – 7.24 (m, 10H), 2.39 (s, 3H).

1-(2,3-dimethylphenyl)-3,4-diphenyl-1*H*-pyrrole-2,5-dione (10d)



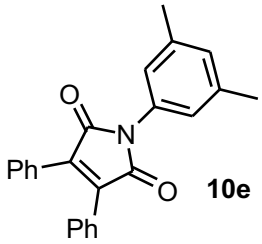
10d

Following the general procedure, the reaction of 2,3-diphenylcycloprop-2-en-1-one **1a** (62.0 mg, 0.3 mmol) and 1-isocyanato-2,3-dimethylbenzene **9d** (28 μL, 0.2 mmol) with PMe_3 (1 M in THF, 40 μL, 0.04 mmol) was conducted for 24 hours which produced **10d** in 62.9 mg, 89% yield, as yellow solid, m.p. 170 – 173 °C.

^{1}H NMR (400 MHz, $CDCl_3$): δ = 7.57 – 7.49 (m, 4H), 7.43 – 7.32 (m, 6H), 7.27 – 7.18 (m, 2H), 7.08 (d, *J* = 7.1 Hz, 1H), 2.35 (s, 3H), 2.13 (s, 3H); **^{13}C NMR** (100 MHz, $CDCl_3$): δ = 169.9, 138.5, 136.5, 135.3, 130.9, 130.8, 130.2, 130.1, 128.7(2C), 126.43, 126.35, 20.6, 15.0; **FTIR**

(neat) υ 2919, 1704, 1597, 1514, 1438, 1286, 1024, 699 cm^{-1}; **HRMS** (ESI) calcd for $C_{24}H_{19}NO_2[M + H]^+$: 354.1489, found: 354.1484.

1-(3,5-dimethylphenyl)-3,4-diphenyl-1*H*-pyrrole-2,5-dione (10e)

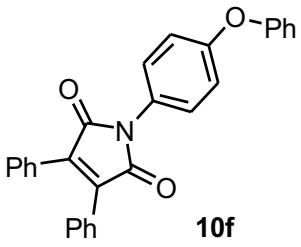


10e

Following the general procedure, the reaction of 2,3-diphenylcycloprop-2-en-1-one **1a** (61.9 mg, 0.3 mmol) and 1-isocyanato-3,5-dimethylbenzene **10e** (28 µL, 0.2 mmol) with PMe_3 (1 M in THF, 40 µL, 0.04 mmol) was conducted for 24 hours which produced **10e** in 60.1 mg, 85% yield, as yellow solid, m.p. 168 – 171 °C.

^{1}H NMR (400 MHz, $CDCl_3$): δ = 7.53 – 7.36 (m, 4H), 7.40 – 7.23 (m, 6H), 6.98 (s, 2H), 6.95 (s, 1H), 2.29 (s, 6H); **^{13}C NMR** (100 MHz, $CDCl_3$): δ = 169.9, 139.0, 136.4, 131.6, 130.2, 130.1, 129.9, 128.7(2C), 124.2, 21.5; **FTIR** (neat) υ 2929, 1703, 1639, 1446, 1373, 1348, 1180, 692 cm^{-1}; **HRMS** (ESI) calcd for $C_{24}H_{19}NO_2[M + H]^+$: 354.1489, found: 354.1478.

1-(4-phenoxyphenyl)-3,4-diphenyl-1H-pyrrole-2,5-dione (10f)[11]

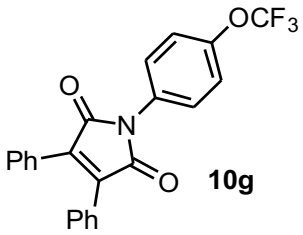


10f

Following the general procedure, the reaction of 2,3-diphenylcycloprop-2-en-1-one **1a** (61.9 mg, 0.3 mmol) and 1-isocyanato-4-phenoxybenzene **9f** (36 µL, 0.2 mmol) with PMe_3 (1 M in THF, 40 µL, 0.04 mmol) was conducted for 24 hours which produced **10f** in 71.8 mg, 86% yield, as yellow solid.

^{1}H NMR (400 MHz, $CDCl_3$): δ = 7.52 (dd, *J* = 7.8, 1.6 Hz, 4H), 7.44 – 7.32 (m, 10H), 7.17 – 7.04 (m, 5H).

3,4-diphenyl-1-(4-(trifluoromethoxy) phenyl)-1*H*-pyrrole-2,5-dione (10g)

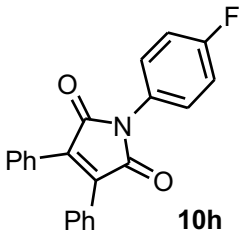


10g

Following the general procedure, the reaction of 2,3-diphenylcycloprop-2-en-1-one **1a** (62.2 mg, 0.3 mmol) and 1-isocyanato-4-(trifluoromethoxy) benzene **9g** (30 μL, 0.2 mmol) with PMe_3 (1 M in THF, 40 μL, 0.04 mmol) was conducted for 24 hours which produced **10g** in 63.8 mg, 78% yield, as yellow solid, m.p. 140 – 143 °C.

^{1}H NMR (400 MHz, $CDCl_3$): δ = 7.56 – 7.50 (m, 6H), 7.48 – 7.31 (m, 8H); **^{13}C NMR** (100 MHz, $CDCl_3$): δ = 169.4, 148.2, 136.6, 130.5, 130.3, 130.2, 128.8, 128.4, 127.5, 121.7, 120.6 (q, *J* = 257.9 Hz); **^{19}F NMR** (376 MHz, $CDCl_3$): δ = -57.8; **FTIR** (neat) υ 2920, 1761, 1694, 1374, 1259, 1026, 694 cm^{-1}; **HRMS** (ESI) calcd for $C_{23}H_{14}F_3NO_3[M + H]^+$: 410.0998, found: 410.0995.

1-(4-fluorophenyl)-3,4-diphenyl-1*H*-pyrrole-2,5-dione (10h)[10]

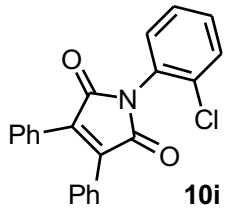


10h

Following the general procedure, the reaction of 2,3-diphenylcycloprop-2-en-1-one **1a** (62.0 mg, 0.3 mmol) and 1-fluoro-4-isocyanatobenzene **9h** (23 μL, 0.2 mmol) with PMe_3 (1 M in THF, 40 μL, 0.04 mmol) was conducted for 24 hours which produced **10h** in 46.7 mg, 68% yield, as yellow solid.

^{1}H NMR (400 MHz, $CDCl_3$): δ = 7.52 (d, *J* = 8.1 Hz, 4H), 7.46 – 7.41 (m, 2H), 7.41 – 7.34 (m, 6H), 7.17 (t, *J* = 8.6 Hz, 2H).

1-(2-chlorophenyl)-3,4-diphenyl-1*H*-pyrrole-2,5-dione (10i)[10]

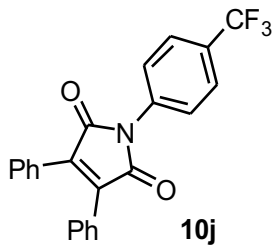


10i

Following the general procedure, the reaction of 2,3-diphenylcycloprop-2-en-1-one **1a** (61.9 mg, 0.3 mmol) and 1-chloro-2-isocyanatobenzene **9i** (25 µL, 0.2 mmol) with PMe_3 (1 M in THF, 40 µL, 0.04 mmol) was conducted for 24 hours which produced **10i** in 58.4 mg, 81% yield, as yellow solid.

^{1}H NMR (400 MHz, $CDCl_3$): δ = 7.61 – 7.51 (m, 5H), 7.45 – 7.34 (m, 9H).

3,4-diphenyl-1-(4-(trifluoromethyl) phenyl)-1*H*-pyrrole-2,5-dione (10j)[10]

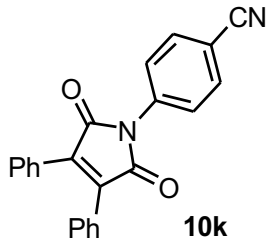


10j

Following the general procedure, the reaction of 2,3-diphenylcycloprop-2-en-1-one **1a** (61.8 mg, 0.3 mmol) and 1-isocyanato-4-(trifluoromethyl) benzene **9j** (29 µL, 0.2 mmol) with PMe_3 (1 M in THF, 40 µL, 0.04 mmol) was conducted for 24 hours which produced **10j** in 48.9 mg, 62% yield, as yellow solid.

^{1}H NMR (400 MHz, $CDCl_3$): δ = 7.76 (d, *J* = 8.4 Hz, 2H), 7.67 (d, *J* = 8.4 Hz, 2H), 7.57 – 7.50 (m, 4H), 7.46 – 7.34 (m, 6H).

4-(2,5-dioxo-3,4-diphenyl-2,5-dihydro-1*H*-pyrrol-1-yl) benzonitrile (10k)[10]

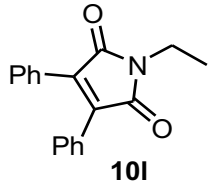


10k

Following the general procedure, the reaction of 2,3-diphenylcycloprop-2-en-1-one **1a** (62.3 mg, 0.3 mmol) and 4-isocyanatobenzonitrile **9k** (28.9 mg, 0.2 mmol) with PMe_3 (1 M in THF, 40 μL, 0.04 mmol) was conducted for 24 hours which produced **10k** in 30.9 mg, 44% yield, as yellow solid.

^{1}H NMR (400 MHz, $CDCl_3$): δ = 7.78 (d, *J* = 8.8 Hz, 2H), 7.71 (d, *J* = 8.8 Hz, 2H), 7.54 – 7.49 (m, 4H), 7.47 – 7.36 (m, 6H).

1-ethyl-3,4-diphenyl-1*H*-pyrrole-2,5-dione (10l)[12]

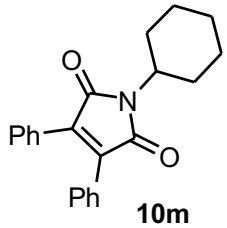


10l

Following the general procedure, the reaction of 2,3-diphenylcycloprop-2-en-1-one **1a** (62.0 mg, 0.3 mmol) and isocyanatoethane **9l** (17 μL, 0.2 mmol) with PMe_3 (1 M in THF, 40 μL, 0.04 mmol) was conducted for 24 hours which produced **10l** in 45.9 mg, 83% yield, as yellow solid.

^{1}H NMR (400 MHz, $CDCl_3$): δ = 7.50 – 7.44 (m, 4H), 7.38 – 7.31 (m, 6H), 3.72 (q, *J* = 7.2 Hz, 2H), 1.29 (t, *J* = 7.2 Hz, 3H).

1-cyclohexyl-3,4-diphenyl-1*H*-pyrrole-2,5-dione (10m)[13]

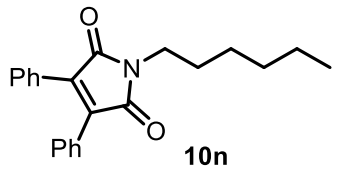


10m

Following the general procedure, the reaction of 2,3-diphenylcycloprop-2-en-1-one **1a** (62.1 mg, 0.3 mmol) and isocyanatocyclohexane **9m** (26 µL, 0.2 mmol) with PMe_3 (1 M in THF, 40 µL, 0.04 mmol) was conducted for 24 hours which produced **10m** in 40.5 mg, 61% yield, as yellow solid.

^{1}H NMR (400 MHz, $CDCl_3$): δ = 7.48 – 7.42 (m, 4H), 7.39 – 7.30 (m, 6H), 4.06 (tt, *J* = 12.3, 3.9 Hz, 1H), 2.17 (qd, *J* = 12.5, 3.2 Hz, 2H), 1.91 – 1.66 (m, 5H), 1.41 – 1.22 (m, 3H).

1-hexyl-3,4-diphenyl-1*H*-pyrrole-2,5-dione (10n)

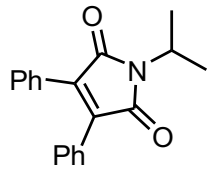


10n

Following the general procedure, the reaction of 2,3-diphenylcycloprop-2-en-1-one **1a** (61.9 mg, 0.3 mmol) and 1-isocyanatohexane **9n** (29 µL, 0.2 mmol) with PMe_3 (1 M in THF, 40 µL, 0.04 mmol) was conducted for 24 hours which produced **10n** in 40.2 mg, 60% yield, as yellow solid.

^{1}H NMR (400 MHz, $CDCl_3$): δ = 7.56 – 7.45 (m, 4H), 7.39 – 7.32 (m, 6H), 3.64 (t, *J* = 7.3 Hz, 2H), 1.68 (p, *J* = 7.4 Hz, 2H), 1.40 – 1.25 (m, 6H), 0.89 (t, *J* = 6.7 Hz, 3H); **^{13}C NMR** (100 MHz, $CDCl_3$): δ = 171.0, 136.2, 130.0, 129.9, 128.8, 128.6, 38.6, 31.5, 28.7, 26.6, 22.6, 14.1. **FTIR** (neat) υ 2987, 1704, 1495, 1396, 1087, 796, 688 cm^{-1}; **HRMS** (ESI) calcd for $C_{22}H_{23}NO_2$[M + H]$^+$: 334.1802, found: 334.1810.

1-isopropyl-3,4-diphenyl-1*H*-pyrrole-2,5-dione (10o)[12]

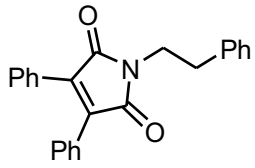


10o

Following the general procedure, the reaction of 2,3-diphenylcycloprop-2-en-1-one **1a** (61.8 mg, 0.3 mmol) and 2-isocyanatopropane **9o** (20 μL, 0.2 mmol) with PMe_3 (1 M in THF, 40 μL, 0.04 mmol) was conducted for 24 hours which produced **10o** in 45.5 mg, 78% yield, as yellow solid.

^{1}H NMR (400 MHz, $CDCl_3$): δ = 7.50 – 7.42 (m, 4H), 7.41 – 7.30 (m, 6H), 4.49 (hept, *J* = 6.9 Hz, 1H), 1.49 (d, *J* = 6.9 Hz, 6H).

1-phenethyl-3,4-diphenyl-1*H*-pyrrole-2,5-dione (10p)[12]

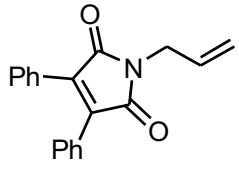


10p

Following the general procedure, the reaction of 2,3-diphenylcycloprop-2-en-1-one **1a** (62.4 mg, 0.3 mmol) and (2-isocyanatoethyl) benzene **9p** (28 μL, 0.2 mmol) with PMe_3 (1 M in THF, 40 μL, 0.04 mmol) was conducted for 24 hours which produced **10p** in 53.3 mg, 75% yield, as yellow solid.

^{1}H NMR (400 MHz, $CDCl_3$): δ = 7.44 (d, *J* = 6.5 Hz, 4H), 7.39 – 7.21 (m, 11H), 3.89 (t, *J* = 7.7 Hz, 2H), 3.00 (t, *J* = 7.7 Hz, 2H).

1-allyl-3,4-diphenyl-1H-pyrrole-2,5-dione (10q)[11]

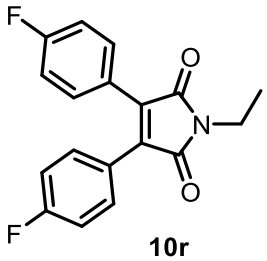


10q

Following the general procedure, the reaction of 2,3-diphenylcycloprop-2-en-1-one **1a** (62.1 mg, 0.3 mmol) and 3-isocyanatoprop-1-ene **9q** (18 μL, 0.2 mmol) with PMe_3 (1 M in THF, 40 μL, 0.04 mmol) was conducted for 24 hours which produced **10q** in 39.4 mg, 68% yield, as yellow solid.

^{1}H NMR (400 MHz, $CDCl_3$): δ = 7.52 – 7.44 (m, 4H), 7.39 – 7.31 (m, 6H), 5.90 (ddt, *J* = 16.4, 11.2, 5.8 Hz, 1H), 5.30 (d, *J* = 17.1 Hz, 1H), 5.22 (d, *J* = 10.2 Hz, 1H), 4.26 (d, *J* = 5.8 Hz, 2H).

1-ethyl-3,4-*bis*(4-fluorophenyl)-1*H*-pyrrole-2,5-dione (10r)

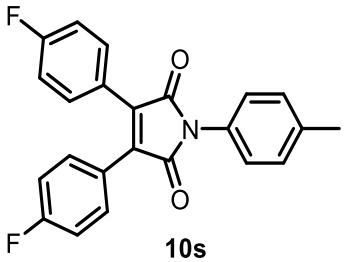


10r

Following the general procedure, the reaction of 2,3-*bis*(4-fluorophenyl) cycloprop-2-en-1-one **1b** (72.5 mg, 0.3 mmol) and isocyanatoethane **9l** (17 μL, 0.2 mmol) with PMe_3 (1 M in THF, 40 μL, 0.04 mmol) was conducted for 24 hours which produced **10r** in 45.1 mg, 72% yield, as yellow solid, m.p. 110 – 113 °C.

^{1}H NMR (400 MHz, $CDCl_3$): δ = 7.48 (m, 4H), 7.09 – 6.90 (m, 4H), 3.71 (q, *J* = 7.2 Hz, 2H), 1.28 (t, *J* = 7.2 Hz, 3H); **^{13}C NMR** (100 MHz, $CDCl_3$): δ = 170.6, 163.7 (d, *J* = 251.7 Hz), 135.1, 132.1 (d, *J* = 8.4 Hz), 124.7 (d, *J* = 3.4 Hz), 116.1 (d, *J* = 21.9 Hz), 33.5, 14.1; **^{19}F NMR** (376 MHz, $CDCl_3$): δ= -109.4; **FTIR** (neat) υ 2972, 1694, 1597, 1502, 1444, 1405, 1224, 1161, 845 cm^{-1}; **HRMS** (ESI) calcd for $C_{18}H_{13}F_2NO_2[M + H]^+$: 314.0987, found: 314.0981.

3,4-*bis*(4-fluorophenyl)-1-(*p*-tolyl)-1*H*-pyrrole-2,5-dione (10s)

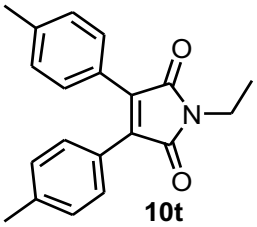


10s

Following the general procedure, the reaction of 2,3-*bis*(4-fluorophenyl) cycloprop-2-en-1-one **1b** (72.6 mg, 0.3 mmol) and 1-isocyanato-4-methylbenzene **9a** (25 μL, 0.2 mmol) with PMe_3 (1 M in THF, 40 μL, 0.04 mmol) was conducted for 24 hours which produced **10s** in 57.0 mg, 76% yield, as yellow solid, m.p. 166 – 168 °C.

^{1}H NMR (400 MHz, $CDCl_3$): δ = 7.58 – 7.47 (m, 4H), 7.33 – 7.26 (m, 4H), 7.15 – 7.00 (m, 4H), 2.40 (s, 3H); **^{13}C NMR** (100 MHz, $CDCl_3$): δ = 169.6, 163.8 (d, *J* = 252.1 Hz), 138.1, 135.1, 132.3 (d, *J* = 8.5 Hz), 129.9, 129.1, 126.2, 124.6 (d, *J* = 3.5 Hz), 116.2 (d, *J* = 21.8 Hz), 21.3; **^{19}F NMR** (376 MHz, $CDCl_3$): δ= -109.1; **FTIR** (neat) υ 2987, 1704, 1598, 1501, 1382, 1221, 1158, 841, 698 cm^{-1}; **HRMS** (ESI) calcd for $C_{23}H_{15}F_2NO_2[M + H]^+$: 376.1144, found: 376.1132.

1-ethyl-3,4-di-*p*-tolyl-1*H*-pyrrole-2,5-dione (10t)

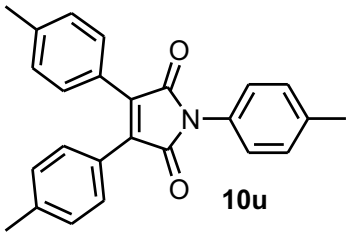


10t

Following the general procedure, the reaction of 2,3-di-*p*-tolylcycloprop-2-en-1-one **1c** (70.2 mg, 0.3 mmol) and isocyanatoethane **9l** (17 μL, 0.2 mmol) with PMe_3 (1 M in THF, 40 μL, 0.04 mmol) was conducted for 24 hours which produced **10t** in 48.2 mg, 79% yield, as yellow solid, m.p. 163 – 166 °C.

^{1}H NMR (400 MHz, $CDCl_3$): δ = 7.39 (d, *J* = 8.2 Hz, 4H), 7.15 (d, *J* = 8.0 Hz, 4H), 3.70 (q, *J* = 7.2 Hz, 2H), 2.36 (s, 6H), 1.27 (t, *J* = 7.2 Hz, 3H); **^{13}C NMR** (100 MHz, $CDCl_3$): δ = 171.1, 140.1, 135.6, 129.9, 129.4, 126.1, 33.4, 21.6, 14.2. **FTIR** (neat) υ 2979, 1698, 1400, 1348, 1049, 872 cm^{-1}; **HRMS** (ESI) calcd for $C_{20}H_{19}NO_2[M + H]^+$: 306.1489, found: 306.1482.

1,3,4-tri-*p*-tolyl-1*H*-pyrrole-2,5-dione (10u)

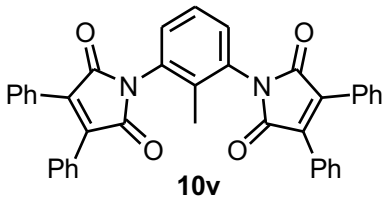


10u

Following the general procedure, the reaction of 2,3-di-*p*-tolylcycloprop-2-en-1-one **1c** (70.4 mg, 0.3 mmol) and 1-isocyanato-4-methylbenzene **9a** (25 μL, 0.2 mmol) with PMe_3 (1 M in THF, 40 μL, 0.04 mmol) was conducted for 24 hours which produced **10u** in 59.5 mg, 81% yield, as yellow solid, m.p. 115 – 118 °C.

^{1}H NMR (400 MHz, $CDCl_3$): δ = 7.43 (d, *J* = 7.9 Hz, 4H), 7.30 (q, *J* = 8.2 Hz, 4H), 7.17 (d, *J* = 7.9 Hz, 4H), 2.38 (s, 3H), 2.36 (s, 6H); **^{13}C NMR** (100 MHz, $CDCl_3$): δ = 170.0, 140.3, 137.7, 135.6, 130.0, 129.8, 129.4, 129.3, 126.2, 125.9, 21.6, 21.3; **FTIR** (neat) υ 2920, 1706, 1513, 1385, 811, 776 cm^{-1}; **HRMS** (ESI) calcd for $C_{25}H_{21}NO_2[M + H]^+$: 368.1645, found: 368.1640.

1,1'-(2-methyl-1,3-phenylene) *bis*(3,4-diphenyl-1*H*-pyrrole-2,5-dione) (10v)

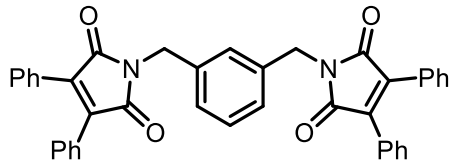


10v

Following the general procedure, the reaction of 2,3-diphenylcycloprop-2-en-1-one **1a** (103.4 mg, 0.5 mmol) and 1,3-diisocyanato-2-methylbenzene **9r** (29 μL, 0.2 mmol) with PMe_3 (1 M in THF, 40 μL, 0.04 mmol) was conducted for 24 hours which produced **10v** in 111.4 mg, 95% yield, as yellow solid, m.p. 289 – 292 °C.

^{1}H NMR (400 MHz, $CDCl_3$): δ = 7.59 – 7.51 (m, 8H), 7.49 – 7.34 (m, 15H), 2.15 (s, 3H); **^{13}C NMR** (100 MHz, $CDCl_3$): δ = 169.3, 136.5, 136.2, 132.3, 130.2(2C), 129.7, 128.8, 128.6, 127.1, 14.5; **FTIR** (neat) υ 2987, 1710, 1473, 1372, 1352, 1140, 689 cm^{-1}; **HRMS** (ESI) calcd for $C_{39}H_{26}N_2O_4[M + H]^+$: 587.1965, found: 587.1967.

1,1'-(1,3-phenylenebis(methylene))*bis*(3,4-diphenyl-1*H*-pyrrole-2,5-dione) (10w)

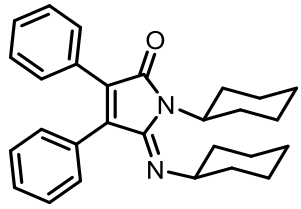


10w

Following the general procedure, the reaction of 2,3-diphenylcycloprop-2-en-1-one **1a** (103.3 mg, 0.5 mmol) and 1,3-*bis*(isocyanatomethyl)benzene **9s** (31 μL, 0.2 mmol) with PMe_3 (1 M in THF, 40 μL, 0.04 mmol) was conducted for 24 hours which produced **10w** in 112.9 mg, 94% yield, as yellow solid, m.p. 193 – 196 °C.

^{1}H NMR (400 MHz, $CDCl_3$): δ = 7.56 (s, 1H), 7.48 – 7.43 (m, 8H), 7.38 – 7.28 (m, 15H), 4.81 (s, 4H); **^{13}C NMR** (100 MHz, $CDCl_3$): δ = 170.5, 137.1, 136.4, 130.0, 129.9, 129.24, 129.18, 128.7, 128.6, 128.2, 41.9; **FTIR** (neat) υ 1695, 1429, 1399, 1316, 1082, 949, 691 cm^{-1}; **HRMS** (ESI) calcd for $C_{40}H_{28}N_2O_4[M + Na]^+$: 623.1941, found: 623.1944.

(*E*)-1-cyclohexyl-5-(cyclohexylimino)-3,4-diphenyl-1,5-dihydro-2*H*-pyrrol-2-one (12a)



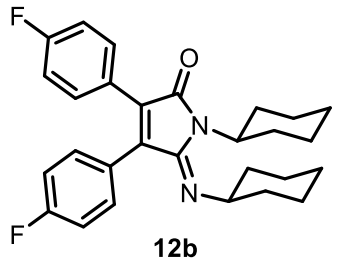
12a

Following the general procedure, the reaction of 2,3-diphenylcycloprop-2-en-1-one **1a** (61.9 mg, 0.3 mmol) and dicyclohexylmethanediimine **11a** (41.3 mg, 0.2 mmol) with PMe_3 (1 M in THF, 40 μL, 0.04 mmol) was conducted for 24 hours which produced **12a** in 71.8 mg, 87% yield, as pale-yellow solid, m.p. 112 – 115 °C.

^{1}H NMR (400 MHz, $CDCl_3$): δ = 7.38 – 7.29 (m, 3H), 7.35 – 7.26 (m, 2H), 7.29 – 7.20 (m, 2H), 7.23 – 7.12 (m, 3H), 4.29 (tt, *J* = 12.0, 3.7 Hz, 1H), 3.26 (td, *J* = 9.5, 4.8 Hz, 1H), 2.35 (qd, *J* = 12.5, 3.2 Hz, 2H), 1.83 (d, *J* = 13.1 Hz, 2H), 1.73 – 1.64 (m, 3H), 1.57 (dt, *J* = 13.1, 3.6 Hz, 2H), 1.47 – 1.18 (m, 8H), 1.11 (dt, *J* = 12.0, 3.2 Hz, 1H), 0.81 – 0.66 (m, 2H); **^{13}C NMR** (100

MHz, $CDCl_3$): δ = 169.2, 148.9, 138.1, 134.5, 134.4, 130.0, 129.8, 128.7, 128.6(2C), 128.4, 127.8, 56.5, 51.2, 34.9, 29.7, 26.4, 25.6, 25.5, 24.2; **FTIR** (neat) υ 2928, 1703, 1639, 1445, 1373, 1260, 1179, 1024, 692 cm^{-1}; **HRMS** (ESI) calcd for $C_{28}H_{32}N_2O$ $[M + H]^+$: 413.2587, found: 413.2582.

(*E*)-1-cyclohexyl-5-(cyclohexylimino)-3,4-*bis*(4-fluorophenyl)-1,5-dihydro-2*H*-pyrrol-2-one (12b)

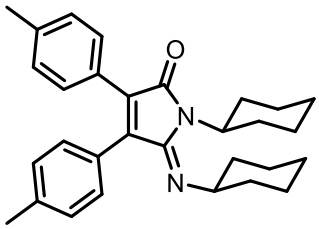


12b

Following the general procedure, the reaction of 2,3-*bis*(4-fluorophenyl) cycloprop-2-en-1-one **1b** (72.4 mg, 0.3 mmol) and dicyclohexylmethanediimine **11a** (41.2 mg, 0.2 mmol) with PMe_3 (1 M in THF, 40 µL, 0.04 mmol) was conducted for 24 hours which produced **12b** in 81.6 mg, 91% yield, as pale-yellow solid, m.p. 108 – 111 °C.

^{1}H NMR (400 MHz, $CDCl_3$): δ = 7.32 – 7.21 (m, 4H), 7.14 – 7.05 (m, 2H), 6.95 – 6.84 (m, 2H), 4.27 (tt, *J* = 12.3, 3.9 Hz, 1H), 3.28 (tt, J = 9.1, 4.2 Hz, 1H), 2.33 (qd, J = 12.5, 3.5 Hz, 2H), 1.91 – 1.77 (m, 2H), 1.75 – 1.57 (m, 3H), 1.46 – 1.12 (m, 11H), 0.80 (qt, *J* = 12.0, 3.9 Hz, 2H); **^{13}C NMR** (100 MHz, $CDCl_3$) δ = 168.9, 162.9 (d, *J* = 249.8 Hz), 162.8 (d, *J* = 249.0 Hz), 148.6, 137.8, 133.3, 132.0 (d, *J* = 8.2 Hz), 130.5 (d, *J* = 8.1 Hz), 130.2 (d, *J* = 3.7 Hz), 125.7 (d, *J* = 3.3 Hz), 116.2 (d, *J* = 21.6 Hz), 115.2 (d, *J* = 21.5 Hz), 56.6, 51.4, 34.9, 29.7, 26.5, 25.6, 25.5, 24.2; **^{19}F NMR** (376 MHz, $CDCl_3$): δ= -111.6, -112.2; **FTIR** (neat) υ 2929, 1701, 1639, 1501, 1400, 1226, 1158, 837, 671 cm^{-1}; **HRMS** (ESI) calcd for $C_{28}H_{30}F_2N_2O$ $[M + H]^+$: 449.2399, found: 449.2395.

(*E*)-1-cyclohexyl-5-(cyclohexylimino)-3,4-di-*p*-tolyl-1,5-dihydro-2*H*-pyrrol-2-one (12c)

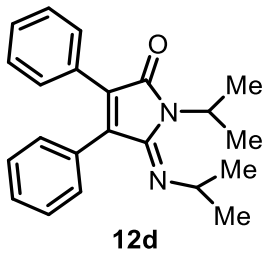


12c

Following the general procedure, the reaction of 2,3-di-*p*-tolylcycloprop-2-en-1-one **1c** (70.4 mg, 0.3 mmol) and dicyclohexylmethanediimine **11a** (41.3 mg, 0.2 mmol) with PMe_3 (1 M in THF, 40 μL, 0.04 mmol) was conducted for 24 hours which produced **12c** in 72.2 mg, 82% yield, as pale-yellow solid, m.p. 126 – 129 °C.

^{1}H NMR (400 MHz, $CDCl_3$): δ = 7.22 (d, *J* = 8.2 Hz, 2H), 7.18 – 7.10 (m, 4H), 6.99 (d, *J* = 8.1 Hz, 2H), 4.27 (tt, *J* = 12.3, 3.8 Hz, 1H), 3.31 (tt, *J* = 9.3, 3.9 Hz, 1H), 2.38 (s, 3H), 2.37 – 2.28 (m, 2H), 2.25 (s, 3H), 1.82 (d, *J* = 13.1 Hz, 2H), 1.74 – 1.64 (m, 3H), 1.58 (dt, *J* = 13.0, 3.6 Hz, 2H), 1.46 – 1.20 (m, 8H), 1.18 – 1.06 (m, 1H), 0.83 – 0.70 (m, 2H); **^{13}C NMR** (100 MHz, $CDCl_3$): δ = 169.4, 149.2, 138.6, 138.1, 137.9, 134.1, 131.5, 130.0, 129.4, 128.6, 128.5, 127.1, 56.3, 51.1, 34.9, 29.7, 26.5, 25.7, 25.6, 24.2, 21.5, 21.4; **FTIR** (neat) υ2924, 1701, 1638, 1448, 1369, 1184, 827 cm^{-1}; **HRMS** (ESI) calcd for $C_{30}H_{36}N_2O$ $[M + H]^+$: 441.2900, found: 441.2903.

(*E*)-1-isopropyl-5-(isopropylimino)-3,4-diphenyl-1,5-dihydro-2*H*-pyrrol-2-one (12d)



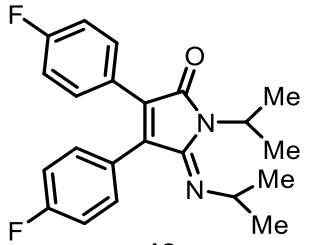
12d

Following the general procedure, the reaction of 2,3-diphenylcycloprop-2-en-1-one **1a** (62.0 mg, 0.3 mmol) and diisopropylmethanediimine **11b** (31 μL, 0.2 mmol) with PMe_3 (1 M in THF, 40 μL, 0.04 mmol) was conducted for 24 hours which produced **12d** in 60.5 mg, 91% yield, as pale-yellow solid, m.p. 103 – 106 °C.

^{1}H NMR (400 MHz, $CDCl_3$): δ = 7.37 – 7.32 (m, 3H), 7.29 – 7.24 (m, 4H), 7.21 – 7.14 (m, 3H), 4.72 (p, *J* = 6.9 Hz, 1H), 3.71 (p, *J* = 6.0 Hz, 1H), 1.49 (d, *J* = 7.0 Hz, 6H), 0.95 (d, *J* = 6.1 Hz,

6H); **^{13}C NMR** (100 MHz, $CDCl_3$): δ = 169.1, 148.7, 138.6, 134.7, 134.3, 130.0, 129.7, 128.8, 128.7(2C), 128.6, 127.9, 48.5, 43.2, 24.9, 20.1; **FTIR** (neat) υ 2969, 1708, 1639, 1404, 1353, 1260, 1021, 798, 690 cm^{-1}; **HRMS** (ESI) calcd for $C_{22}H_{24}N_2O$ $[M + H]^+$: 333.1961, found: 333.1957.

(*E*)-3,4-*bis*(4-fluorophenyl)-1-isopropyl-5-(isopropylimino)-1,5-dihydro-2*H*-pyrrol-2-one (12e)

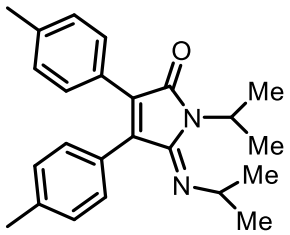


12e

Following the general procedure, the reaction of 2,3-*bis*(4-fluorophenyl) cycloprop-2-en-1-one **1b** (72.7 mg, 0.3 mmol) and diisopropylmethanediimine **11b** (31 µL, 0.2 mmol) with PMe_3 (1 M in THF, 40 µL, 0.04 mmol) was conducted for 24 hours which produced **12e** in 65.6 mg, 89% yield, as pale-yellow solid, m.p. 99 – 101 °C.

^{1}H NMR (400 MHz, $CDCl_3$): δ = 7.30 – 7.20 (m, 4H), 7.09 (t, *J* = 8.6 Hz, 2H), 6.90 (t, *J* = 8.7 Hz, 2H), 4.71 (hept, *J* = 6.7 Hz, 1H), 3.71 (hept, *J* = 6.1 Hz, 1H), 1.48 (d, *J* = 7.0 Hz, 6H), 0.96 (d, *J* = 6.1 Hz, 6H); **^{13}C NMR** (100 MHz, $CDCl_3$): δ = 168.8, 162.94 (d, *J* = 250.0 Hz), 162.89 (d, *J* = 249.0 Hz), 148.4, 138.2, 133.4, 132.0 (d, *J* = 8.3 Hz), 130.5 (d, *J* = 8.1 Hz), 130.1 (d, *J* = 3.7 Hz), 125.6 (d, *J* = 3.4 Hz), 116.3 (d, *J* = 21.7 Hz), 115.2 (d, *J* = 21.6 Hz), 48.5, 43.3, 24.8, 20.1; **^{19}F NMR** (376 MHz, $CDCl_3$): δ= -111.5, -112.0; FTIR (neat) υ 2964, 1701, 1444, 1398, 1260, 1018, 693 cm^{-1}; **HRMS** (ESI) calcd for $C_{22}H_{22}F_2N_2O$ $[M + H]^+$: 369.1773, found: 369.1775.

(*E*)-1-isopropyl-5-(isopropylimino)-3,4-di-*p*-tolyl-1,5-dihydro-2*H*-pyrrol-2-one (12f)



12f

Following the general procedure, the reaction of 2,3-di-*p*-tolylcycloprop-2-en-1-one **1c** (70.3 mg, 0.3 mmol) and diisopropylmethanediimine **11b** (31 μL, 0.2 mmol) with PMe_3 (1 M in THF, 40 μL, 0.04 mmol) was conducted for 24 hours which produced **12f** in 63.4 mg, 88% yield, as pale-yellow solid, m.p. 125 – 127 °C.

^{1}H NMR (400 MHz, $CDCl_3$): δ = 7.20 – 7.11 (m, 6H), 6.99 (d, *J* = 8.2 Hz, 2H), 4.70 (hept, *J* = 7.0 Hz, 1H), 3.76 (hept, *J* = 6.0 Hz, 1H), 2.37 (s, 3H), 2.25 (s, 3H), 1.48 (d, *J* = 7.0 Hz, 6H), 0.94 (d, *J* = 6.1 Hz, 6H); **^{13}C NMR** (100 MHz, $CDCl_3$): δ = 169.4, 148.9, 138.6, 138.4, 138.3, 134.2, 131.4, 129.9, 129.6, 128.7, 128.6, 127.0, 48.3, 43.1, 24.9, 21.5, 21.4, 20.1; **FTIR** (neat) υ 2970, 1700, 1633, 1400, 1352, 1260, 1024, 677 cm^{-1}; **HRMS** (ESI) calcd for $C_{24}H_{28}N_2O$ [M + H]$^+$: 361.2274, found: 361.2272.

4. X-ray Crystallographic Data of 3v, 10e and 12a

Single crystal of compound **3v** was obtained by recrystallization from ethyl acetate, while compounds **10e** and **12a** were both obtained from a mixture solvent of petroleum ether (60–90 ºC) and ethyl acetate. The structures of **3v**, **10e** and **12a** are shown in Figures S1–S3, and X-ray diffraction data and refinement are shown in Tables S2–S4, respectively. CIF files can be obtained from the Cambridge Crystallographic Data Center using deposition number (**3v**, CCDC 2118493; **10e**, CCDC 2118492; **12a**, CCDC 2118486).

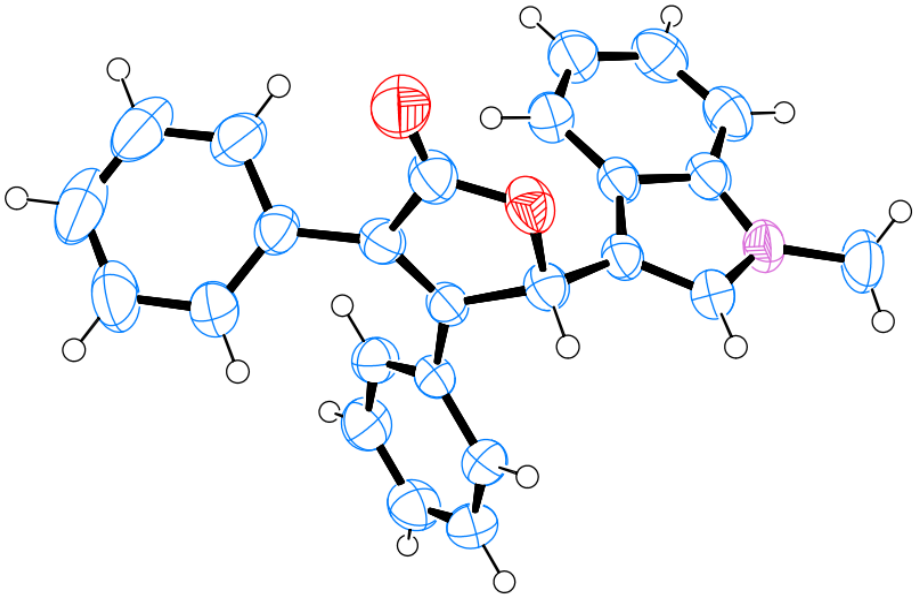


Figure S1. X-ray Single Crystal Structure of **3v**

Table S2. Crystal data and structure refinement for **3v**

Identification code	**3v**
Empirical formula	$C_{20}H_{17}NO_2$
Formula weight	303.13
Temperature/K	296.15
Crystal system	trigonal
Space group	R-3
a/Å	32.848(15)
b/Å	32.848(15)
c/Å	10.842(9)
α/°	90
β/°	90
γ/°	120
Volume/Å^3	10130(19)
Z	1

$\rho_{calc}g/cm^3$	1.078
μ/mm^{-1}	0.068
F(000)	3456.0
Crystal size/mm^3	0.24 × 0.22 × 0.2
Radiation	MoKα (λ = 0.71073)
2Θ range for data collection/°	4.02 to 50.87
Index ranges	-39 ≤ h ≤ 39, -39 ≤ k ≤ 35, -13 ≤ l ≤ 12
Reflections collected	22203
Independent reflections	4144 [Rint = 0.0948, Rsigma = 0.0548]
Data/restraints/parameters	4144/0/254
Goodness-of-fit on F^2	1.014
Final R indexes [I==2σ (I)]	R1 = 0.0504, wR2 = 0.1233
Final R indexes [all data]	R1 = 0.0767, wR2 = 0.1450
Largest diff. peak/hole / e $Å^{-3}$	0.14/-0.22

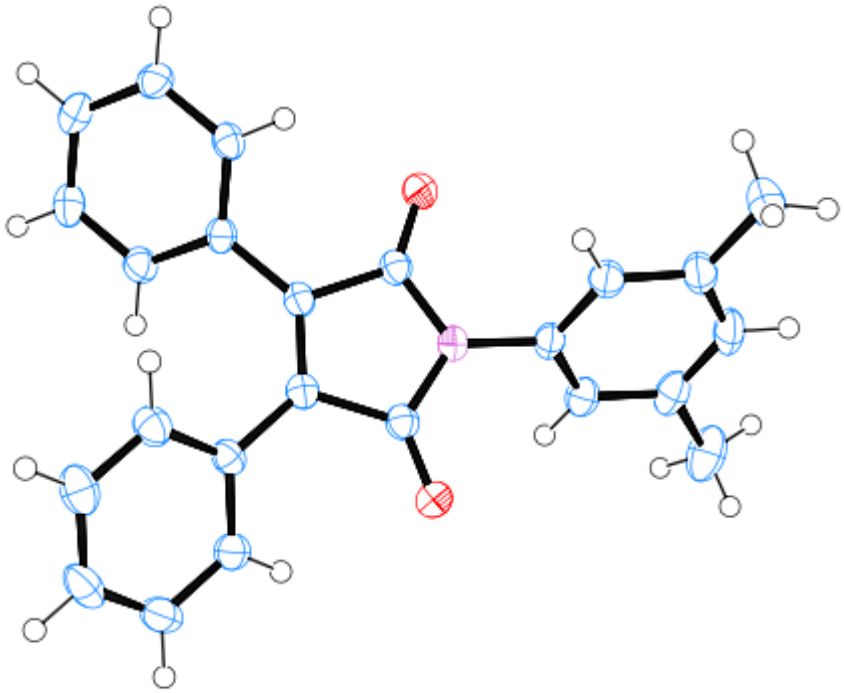


Figure S2. X-ray Single Crystal Structure of **10e**

Table S3. Crystal data and structure refinement for **10e**

Identification code	**10e**
Empirical formula	$C_{24}H_{19}NO_2$
Formula weight	353.14
Temperature/K	296.15
Crystal system	monoclinic
Space group	$P2_1/c$
a/Å	9.4143(14)
b/Å	14.690(2)
c/Å	13.927(2)
α/°	90
β/°	106.730(2)
γ/°	90
Volume/Å^3	1844.6(5)
Z	1
ρ_{calc}g/cm^3	1.273
μ/mm^{-1}	0.081
F(000)	744.0
Crystal size/mm^3	0.26 × 0.24 × 0.2
Radiation	MoKα (λ = 0.71073)
2Θ range for data collection/°	4.124 to 53.074
Index ranges	-11 ≤ h ≤ 11, -18 ≤ k ≤ 18, -17 ≤ l ≤ 17
Reflections collected	18618
Independent reflections	3822 [Rint = 0.0359, Rsigma = 0.0281]
Data/restraints/parameters	3822/0/246
Goodness-of-fit on F^2	1.021

Final R indexes [I==2σ (I)]	R1 = 0.0385, wR2 = 0.0906
Final R indexes [all data]	R1 = 0.0568, wR2 = 0.1002
Largest diff. peak/hole / e Å^{-3}	0.22/-0.21

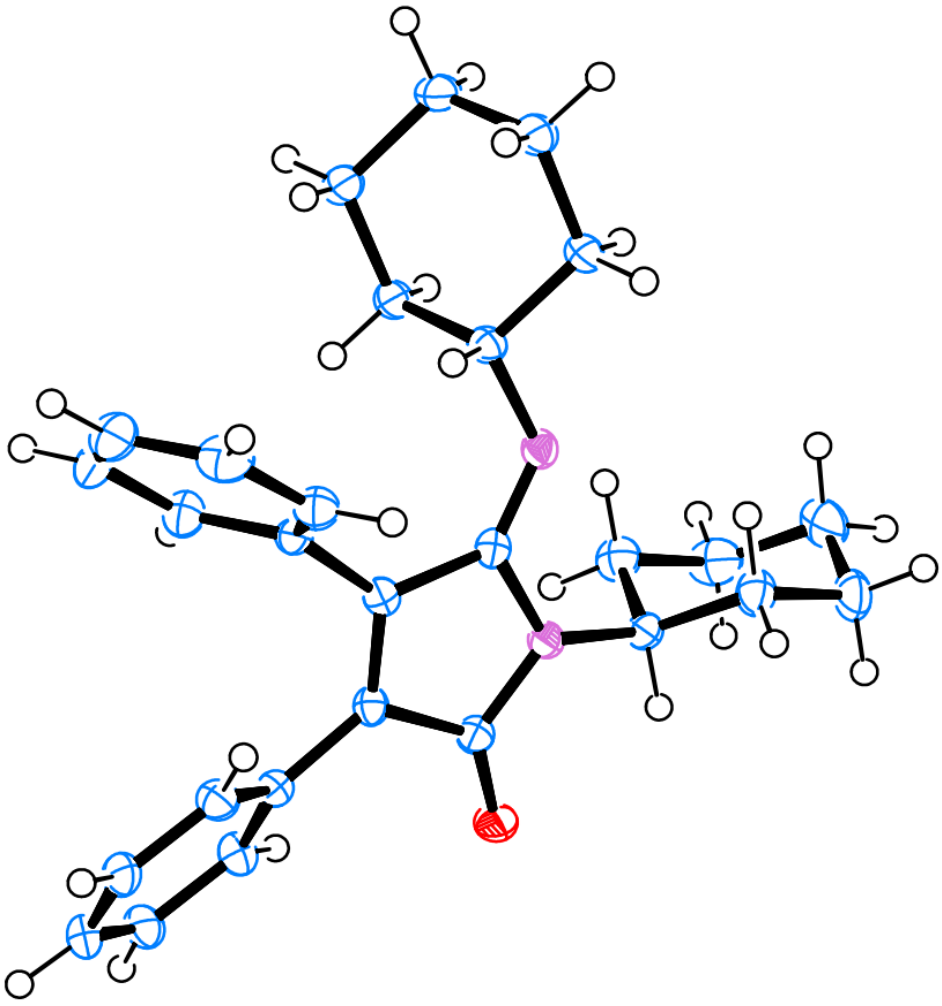


Figure S3. X-ray Single Crystal Structure of **12a**

Table S4. Crystal data and structure refinement for **12a**

Identification code	**12a**
Empirical formula	$C_{28}H_{32}N_2O$

Formula weight	412.25
Temperature/K	296.15
Crystal system	triclinic
Space group	P-1
a/Å	6.2055(17)
b/Å	11.319(3)
c/Å	32.886(9)
α/°	89.884(3)
β/°	85.258(4)
γ/°	89.848(4)
Volume/Å^3	2302.0(11)
Z	1
ρ_{calc}g/cm^3	1.193
μ/mm^{-1}	0.072
F(000)	892.0
Crystal size/mm^3	0.24 × 0.22 × 0.2
Radiation	MoKα (λ = 0.71073)
2Θ range for data collection/°	3.598 to 53.342
Index ranges	-7 ≤ h ≤ 7, -14 ≤ k ≤ 14, -41 ≤ l ≤ 41
Reflections collected	14200
Independent reflections	6186 [Rint = 0.0446, Rsigma = 0.0751]
Data/restraints/parameters	6186/0/559
Goodness-of-fit on F^2	1.066
Final R indexes [I==2σ (I)]	R1 = 0.0728, wR2 = 0.1952
Final R indexes [all data]	R1 = 0.0995, wR2 = 0.2062
Largest diff. peak/hole / e Å^{-3}	0.19/-0.29

5. HRMS and ^{31}P NMR Tracking Experiments

To clarify the possible mechanism of the PMe_3-catalyzed (3+2) annulation, ^{31}P NMR tracking and HRMS experiments were conducted to trap any possible reaction intermediates.

HRMS Detection:

Under N_2 atmosphere, PMe_3 (1M in THF, 20 μL, 0.02 mmol) was added to a dispersion of cyclopropenone **1a** (0.15 mmol), 4 Å MS (50 mg), and isocyanate **9a** (0.1 mmol) in dried CH_2Cl_2 (1.0 mL). The mixture was stirred at room temperature for 30 min, a small amount of the mixture was transferred for the HRMS detection. From the HRMS spectrum, a peak with mass 283.1248 (Calcd. for $C_{18}H_{19}OP[M + H]^+$ 283.1247) corresponding to the adduct of PMe_3 and **1a** was observed, which may support the formation of the α-ketenyl P-ylide intermediate.

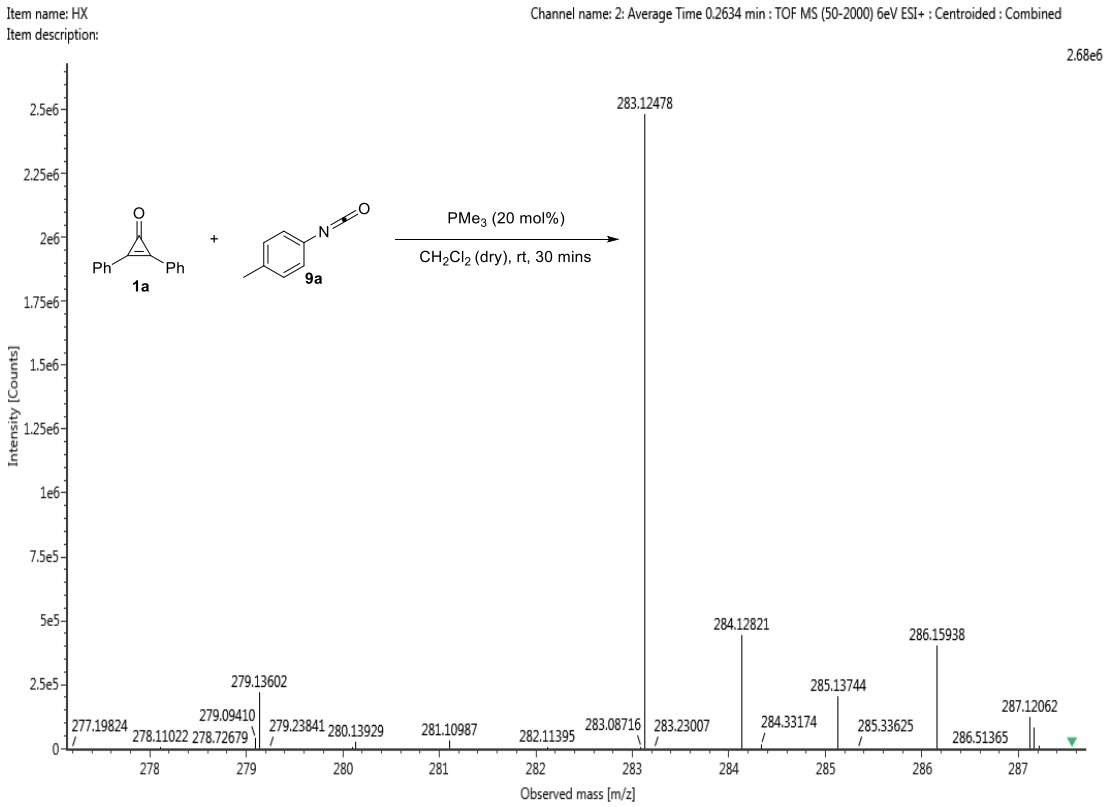


Figure S4. HRMS detection of PMe_3-catalyzed reaction of **1a** and **9a**

^{31}P NMR Tracking:

Under N_2 atmosphere, PMe_3 (1M in THF, 20 μL, 0.02 mmol) was added to a dispersion of cyclopropenone **1a** (0.15 mmol), 4 Å MS (50 mg), and isocyanate **9a** (0.1 mmol) in dried $CDCl_3$ (1.0 mL). After 3 hours, the reaction mixture was filtered through syringe filter and the filtrate was subjected to the ^{31}P NMR test. From the ^{31}P NMR spectrum, it was found that several new peaks with signals at 5.8, 15.6, 22.9, and 38.6 ppm, respectively, appeared, while the PMe_3 catalyst (signal at -61.8 ppm) disappeared (Figure S5). The result suggests the formation of several PMe_3-derived intermediates in the reaction, and implies that free phosphine is not the resting state of the catalytic cycle. One of the new signal presumably corresponds to the proposed α-ketenyl P-ylide intermediate as the chemical shift is compatible.

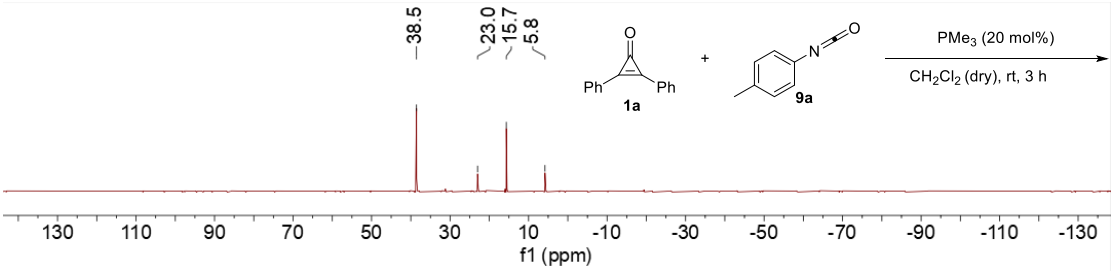


Figure S5. ^{31}P NMR tracking of the PMe_3-catalyzed reaction of **1a** and **9a**

6. Computational Details

All calculations were carried out by using the Gaussian 16 Rev. A.03.[14] Given the benchmark study done by Brinck and co-workers,[15] geometry optimizations and frequency calculations were performed at the ωB97X-D/6-31+G(d) level of theory.[16] Frequency calculations were conducted at the same level of theory to confirm the presence of local minima (no imaginary frequencies) and transition states (one imaginary frequency) on the PES. Subsequently, single point energies were computed at the ωB97X-D/def2TZVP level with SMD[17] solvation model. Conformational searches were conducted using the CREST conformer-rotamer ensemble sampling tool version 2.10.2 with xtb version 6.3.3. to ensure all structures showed are lowest

energy conformers.[18] Intrinsic reaction coordinate (IRC) calculations were performed to verify that the saddle points found were true TSs connecting the reactants and the products.[19] All thermodynamic quantities (1 mol/L, 298.15 K) were computed in the GoodVibes code[20] with quasiharmonic corrections.[21] 3D renderings of stationary points and Frontier Molecular Orbitals(FMOs) were generated using CYLview 1.0[22] and PyMol 2.7.[23]

Cartesian Coordinates of Calculated Structure

1a

C	-0.717978	-0.781079	1.013243
C	0.642618	-0.784657	1.023888
C	-1.944221	-1.312234	0.450709
C	-3.159535	-1.012869	1.078564
C	-1.940149	-2.097263	-0.708831
C	-4.350605	-1.508745	0.563388
H	-3.154440	-0.392639	1.969735
C	-3.133911	-2.583687	-1.225497
H	-1.001676	-2.303869	-1.214018
C	-4.338683	-2.294528	-0.587016
H	-5.290104	-1.278882	1.056173
H	-3.126993	-3.187714	-2.127555
H	-5.270738	-2.678684	-0.990492
C	1.874459	-1.344527	0.502960
C	3.085027	-0.708655	0.804606
C	1.880261	-2.509177	-0.274458
C	4.281238	-1.221105	0.318375
H	3.072200	0.186470	1.419006
C	3.079117	-3.023043	-0.751192
H	0.945377	-3.020344	-0.482608
C	4.279161	-2.376663	-0.459900

H	5.217071	-0.722177	0.549926
H	3.079918	-3.929054	-1.349316
H	5.215207	-2.777956	-0.836558
C	-0.042254	0.108700	1.904160
O	-0.046772	0.967854	2.757621

2a

C	2.590793	-0.480838	2.321275
H	1.565656	-0.887993	2.459755
C	3.602161	-1.476313	1.896241
C	4.928400	-1.085495	1.689632
C	3.226713	-2.806363	1.702538
C	5.870193	-2.023548	1.291454
H	5.195436	-0.045125	1.846933
C	4.171011	-3.746256	1.303285
H	2.192818	-3.103130	1.865413
C	5.491151	-3.352916	1.098520
H	6.901354	-1.724660	1.129936
H	3.880596	-4.781239	1.151951
H	6.230176	-4.085172	0.786752
O	2.820488	0.692108	2.513147

3a

C	-0.017145	0.172937	0.180737
C	0.259053	-1.137878	0.074958
C	-0.758114	1.063692	-0.727031
C	-1.973105	0.652371	-1.285977
C	-0.280077	2.346988	-1.010368
C	-2.688130	1.505213	-2.116775
H	-2.353499	-0.338819	-1.060724
C	-0.993828	3.197015	-1.848257
H	0.664269	2.678465	-0.587258
C	-2.199846	2.778401	-2.402030
H	-3.631741	1.175838	-2.540742
H	-0.607139	4.187615	-2.067256
H	-2.759924	3.443254	-3.052544
C	0.006433	-2.077095	-1.031548
C	0.128901	-1.660779	-2.362810
C	-0.350943	-3.403508	-0.765567
C	-0.121634	-2.545024	-3.404193
H	0.427629	-0.640079	-2.580142
C	-0.603517	-4.285232	-1.811126
H	-0.417533	-3.743691	0.261599
C	-0.493367	-3.859433	-3.131337
H	-0.020305	-2.208225	-4.431544
H	-0.882858	-5.311163	-1.590962

H	-0.688134	-4.550944	-3.945737
C	0.956402	-1.554517	1.325984
O	1.361401	-2.637935	1.651008
C	-2.709425	2.330454	3.864472
C	-1.777129	3.171812	3.267217
C	-0.727300	2.633073	2.528046
C	-0.608099	1.254561	2.376700
C	-1.546185	0.413514	2.974896
C	-2.589736	0.949519	3.719206
H	-3.526350	2.747744	4.445436
H	-1.861254	4.248440	3.379860
H	0.002805	3.292458	2.065407
H	-1.451799	-0.663066	2.866412
H	-3.312364	0.288327	4.187869
C	0.494036	0.675560	1.515854
H	1.283600	1.425125	1.376921
O	1.085488	-0.462286	2.129583

3a*

C	-0.706781	-0.605386	0.600954
C	-0.425515	-1.937786	0.001695
C	-1.819476	0.160924	-0.027168
C	-3.022093	-0.472890	-0.350697

C	-1.664588	1.515120	-0.341854
C	-4.058383	0.236981	-0.947727
H	-3.141445	-1.530346	-0.132264
C	-2.697411	2.223463	-0.943201
H	-0.727097	2.010655	-0.108011
C	-3.899650	1.587578	-1.244281
H	-4.989451	-0.268152	-1.186911
H	-2.561582	3.274116	-1.181883
H	-4.706216	2.140970	-1.715989
C	-0.338005	-2.179709	-1.458904
C	0.087216	-1.150977	-2.305948
C	-0.671915	-3.417969	-2.018581
C	0.171660	-1.356899	-3.677809
H	0.350590	-0.185586	-1.884990
C	-0.566317	-3.627229	-3.388729
H	-1.027491	-4.221957	-1.378266
C	-0.149001	-2.596029	-4.225534
H	0.497125	-0.544737	-4.321154
H	-0.826014	-4.596362	-3.804432
H	-0.078004	-2.755822	-5.297064
C	-0.255326	-2.943806	0.849921
O	-0.128279	-3.833117	1.593262
C	-0.565450	3.228025	4.184490

C	-1.670218	2.526979	3.705739
C	-1.497830	1.423502	2.879722
C	-0.213654	1.002157	2.509517
C	0.888382	1.701716	3.016607
C	0.716289	2.809041	3.839915
H	-0.703421	4.089831	4.830656
H	-2.673400	2.837346	3.982818
H	-2.364249	0.877666	2.520932
H	1.891446	1.374777	2.753435
H	1.584588	3.342322	4.215691
C	0.022437	-0.174331	1.651913
H	0.898490	-0.764266	1.919140

IM1

C	0.799525	0.765579	0.539914
C	1.230785	-0.326423	-0.376106
C	1.594819	0.816188	1.600541
O	2.340239	0.826935	2.503273
C	-0.360061	1.655975	0.343511
C	-0.611380	2.754819	1.177695
C	-1.234173	1.416495	-0.723117
C	-1.702765	3.583152	0.951185
H	0.057029	2.964658	2.010436

C	-2.324951	2.250020	-0.948715
H	-1.055568	0.554224	-1.359813
C	-2.565944	3.338330	-0.115542
H	-1.878112	4.429213	1.609777
H	-2.995126	2.041101	-1.778062
H	-3.417569	3.988273	-0.292031
C	0.638085	-1.661820	-0.298893
C	-0.499812	-1.889623	0.502414
C	1.139174	-2.774877	-1.004230
C	-1.101873	-3.137739	0.572550
H	-0.914454	-1.064427	1.073068
C	0.525925	-4.021108	-0.941534
H	2.044696	-2.683618	-1.599688
C	-0.603051	-4.217601	-0.153801
H	-1.978924	-3.266313	1.201634
H	0.949224	-4.849671	-1.503766
H	-1.078174	-5.192047	-0.098176
P	2.290275	0.130375	-1.646004
C	3.998272	-0.544018	-1.633796
H	3.981989	-1.635260	-1.687960
H	4.583445	-0.153918	-2.473193
H	4.475206	-0.257370	-0.692160
C	1.673870	-0.260177	-3.324725

H	0.803878	0.371889	-3.523067
H	2.439475	-0.081442	-4.085728
H	1.355368	-1.304286	-3.368638
C	2.536921	1.929369	-1.634342
H	1.567232	2.431182	-1.679493
H	3.044456	2.233648	-0.716563
H	3.144103	2.218291	-2.497010

TS1

C	-0.776945	-0.424249	0.397820
C	0.626516	-0.318750	0.494847
C	-1.881023	-1.345783	0.524326
C	-3.195572	-0.915393	0.293510
C	-1.657472	-2.676242	0.902912
C	-4.257215	-1.799058	0.435836
H	-3.369329	0.116148	0.001543
C	-2.723925	-3.555293	1.048994
H	-0.639464	-3.015711	1.071799
C	-4.026615	-3.120578	0.815959
H	-5.271267	-1.456089	0.251938
H	-2.538395	-4.584859	1.341598
H	-4.859201	-3.808659	0.928124
C	1.796173	-1.019623	-0.084642

C	2.242831	-2.254959	0.391463
C	2.469794	-0.414032	-1.148588
C	3.337042	-2.881484	-0.193860
H	1.733891	-2.717072	1.233999
C	3.563296	-1.043820	-1.736458
H	2.125107	0.551067	-1.508673
C	3.998817	-2.277456	-1.261486
H	3.674193	-3.843253	0.181644
H	4.078644	-0.565963	-2.564370
H	4.853752	-2.766815	-1.718426
C	-0.243601	0.812813	0.118686
O	-0.277332	2.005078	-0.159344
P	1.395050	0.297922	2.543204
C	2.448199	1.713490	2.055128
H	2.725394	2.340850	2.908512
H	3.355614	1.339207	1.571239
H	1.889067	2.306882	1.323760
C	-0.061993	1.077822	3.329214
H	-0.514517	1.772638	2.615833
H	-0.802198	0.311235	3.577588
H	0.213216	1.625047	4.236080
C	2.312056	-0.480948	3.939479
H	3.222250	-0.949026	3.552887

H	1.693907	-1.258587	4.397528
H	2.586822	0.253263	4.705389

TS2

C	-1.921746	0.139395	-0.532475
C	-2.306551	-1.284400	-0.323128
C	-2.929291	1.078580	-1.059064
C	-4.296775	0.790492	-0.887408
C	-2.617433	2.301444	-1.679966
C	-5.282227	1.683262	-1.277552
H	-4.574520	-0.149537	-0.422925
C	-3.608209	3.195394	-2.076216
H	-1.584230	2.582417	-1.866060
C	-4.950246	2.899105	-1.872745
H	-6.324312	1.425896	-1.110480
H	-3.320950	4.130355	-2.550183
H	-5.723638	3.596520	-2.179271
C	-2.569741	-2.207213	-1.472226
C	-2.876453	-1.685065	-2.740602
C	-2.477799	-3.606102	-1.360755
C	-3.075876	-2.517486	-3.837480
H	-2.978150	-0.613603	-2.868923
C	-2.685037	-4.433719	-2.457403

H	-2.237911	-4.060053	-0.404565
C	-2.982723	-3.898799	-3.707440
H	-3.317125	-2.074617	-4.800113
H	-2.603341	-5.509736	-2.330745
H	-3.141558	-4.547790	-4.563218
C	-2.372329	-1.916925	0.871017
O	-2.594659	-2.793752	1.613889
P	-0.261527	0.237967	-1.123329
C	-0.015968	0.209197	-2.936921
H	1.044539	0.307629	-3.189104
H	-0.402352	-0.737481	-3.323190
H	-0.578363	1.024174	-3.400397
C	0.678211	1.705299	-0.555975
H	0.061421	2.605408	-0.561073
H	1.027748	1.531621	0.464882
H	1.546728	1.861533	-1.202441
C	0.668533	-1.166617	-0.461307
H	0.317384	-2.094692	-0.918037
H	0.485052	-1.208154	0.617235
H	1.734929	-1.032984	-0.665670
C	-5.502597	2.385798	2.378791
C	-4.466031	3.156623	1.858358
C	-3.215949	2.585803	1.658290

C	-2.994747	1.242023	1.963572
C	-4.032544	0.476906	2.499526
C	-5.282614	1.048619	2.704144
H	-6.482452	2.828178	2.533814
H	-4.635490	4.196885	1.598434
H	-2.408536	3.179297	1.236585
H	-3.845992	-0.560822	2.757412
H	-6.088571	0.450764	3.119497
C	-1.661889	0.639185	1.716080
H	-0.881015	1.391263	1.513226
O	-1.339880	-0.495947	2.124320

TS2*

C	-4.824963	1.287038	0.879713
C	-5.188872	-0.181672	0.795785
C	-5.908987	2.236281	0.487678
C	-7.249992	1.839460	0.628817
C	-5.672038	3.575227	0.152531
C	-8.293919	2.727456	0.420434
H	-7.475807	0.824073	0.934823
C	-6.721574	4.463777	-0.067647
H	-4.664033	3.961324	0.124159
C	-8.039932	4.048107	0.057972

H	-9.315732	2.383521	0.551085
H	-6.495851	5.495691	-0.322292
H	-8.857614	4.742803	-0.108176
C	-5.810303	-0.902172	-0.357551
C	-6.177927	-0.223811	-1.526172
C	-6.029141	-2.288050	-0.310627
C	-6.736281	-0.905377	-2.602764
H	-6.044995	0.849303	-1.591429
C	-6.587203	-2.965254	-1.386578
H	-5.761592	-2.854833	0.578270
C	-6.944793	-2.278396	-2.543633
H	-7.015492	-0.348870	-3.492723
H	-6.742617	-4.037880	-1.317281
H	-7.382724	-2.806899	-3.384568
C	-4.889157	-0.927512	1.853936
O	-4.618888	-1.625848	2.748871
P	-3.161447	1.441384	0.146525
C	-3.017235	0.534919	-1.432898
H	-1.979683	0.594263	-1.777215
H	-3.296073	-0.513032	-1.308752
H	-3.668425	0.978850	-2.188976
C	-2.464788	3.069883	-0.269837
H	-2.982410	3.504961	-1.127219

H	-2.543510	3.706215	0.611715
H	-1.413097	2.916680	-0.534141
C	-1.930755	0.644277	1.220688
H	-2.246377	-0.362816	1.500951
H	-1.817397	1.274641	2.105038
H	-0.985408	0.578194	0.674427
C	-8.119416	2.851662	4.435750
C	-7.672310	1.533513	4.429966
C	-6.429426	1.222815	3.884869
C	-5.627863	2.223064	3.335696
C	-6.066663	3.546032	3.378682
C	-7.308342	3.859091	3.916472
H	-9.091700	3.095128	4.855131
H	-8.289425	0.746253	4.854193
H	-6.081779	0.192955	3.902000
H	-5.412730	4.310518	2.973932
H	-7.647583	4.891092	3.930262
C	-4.308321	1.917602	2.671235
H	-3.918203	0.934929	3.030098
O	-3.474361	2.859846	2.477840

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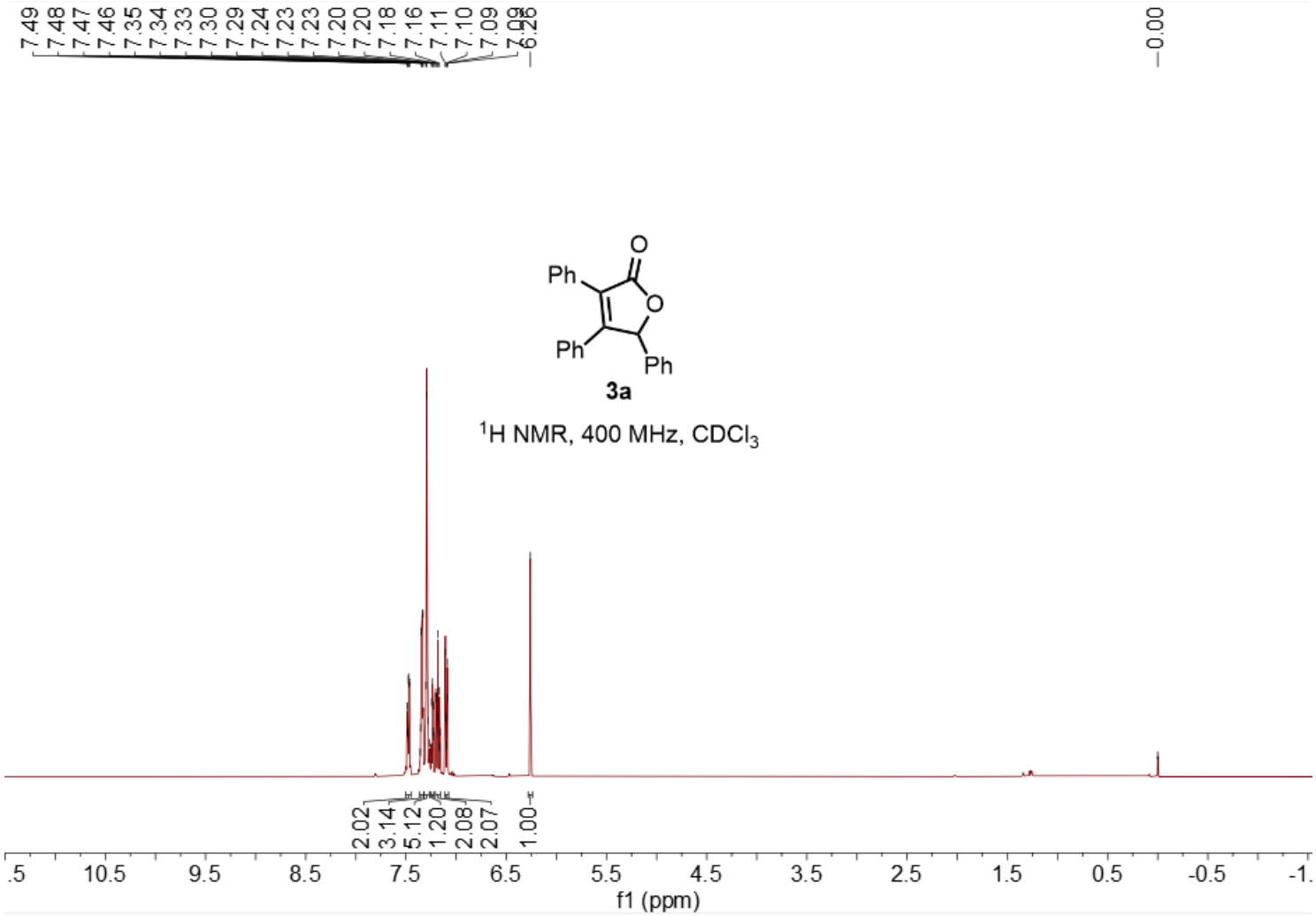
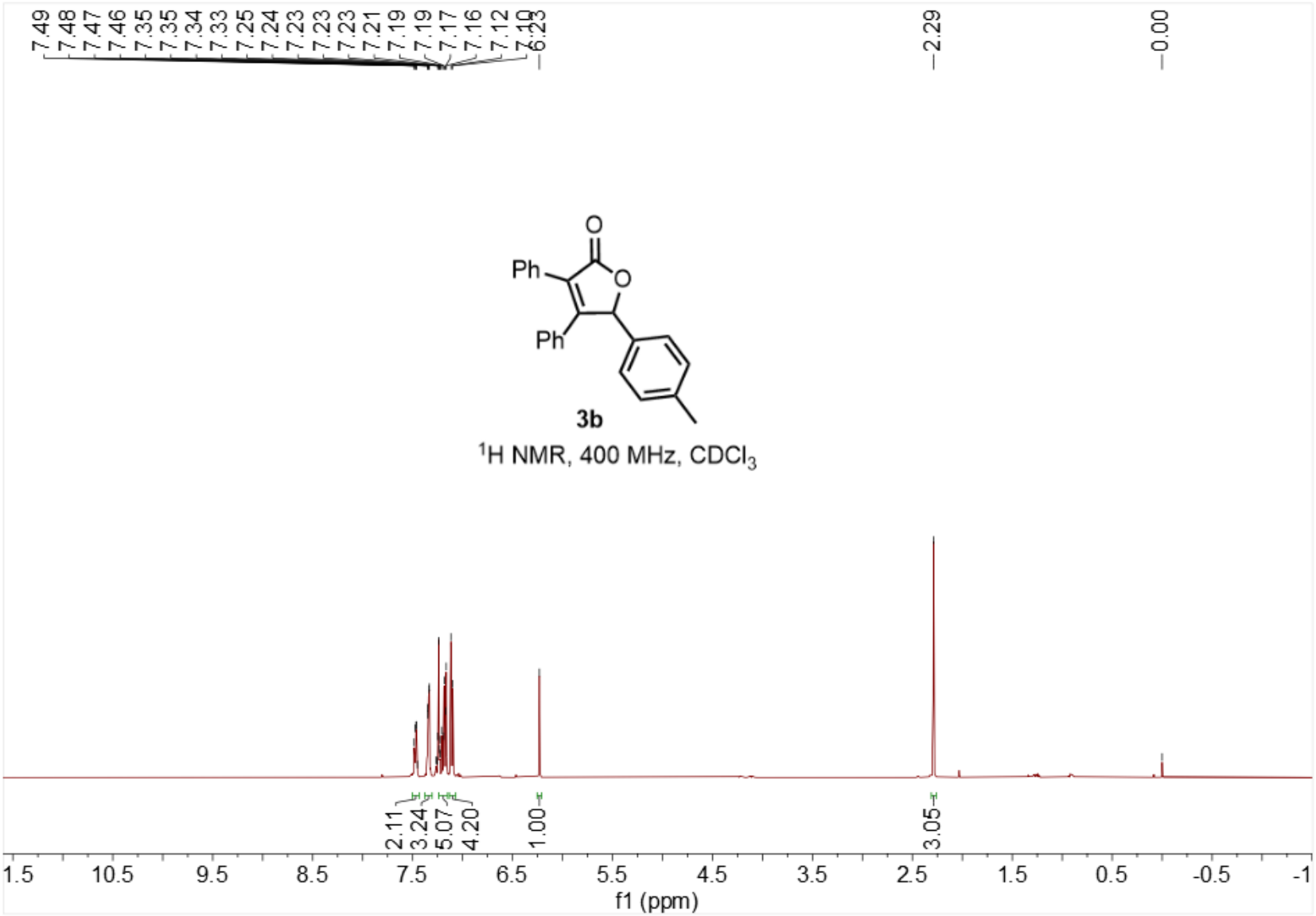
8. NMR Spectra Copies

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7.16
7.12
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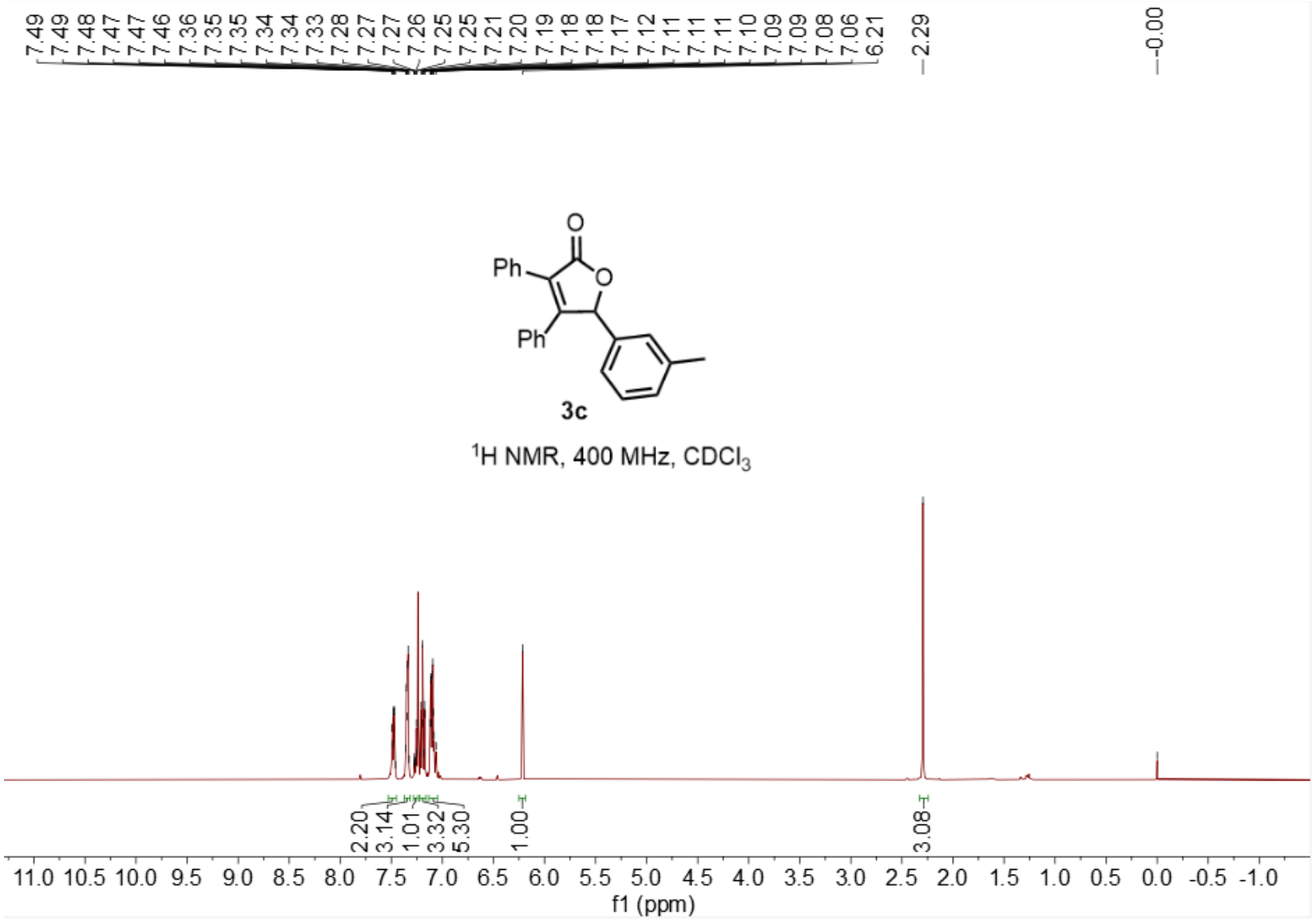
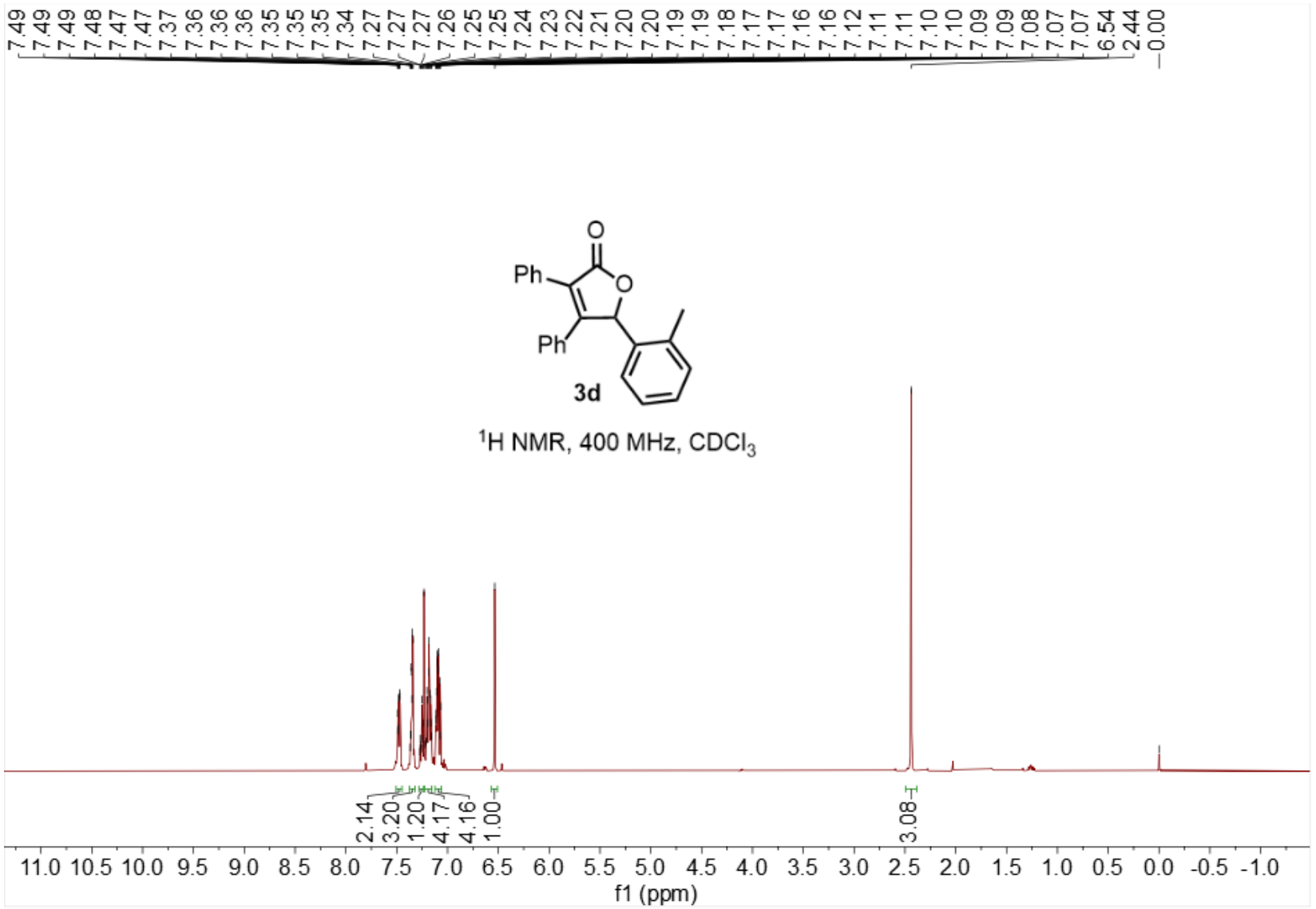
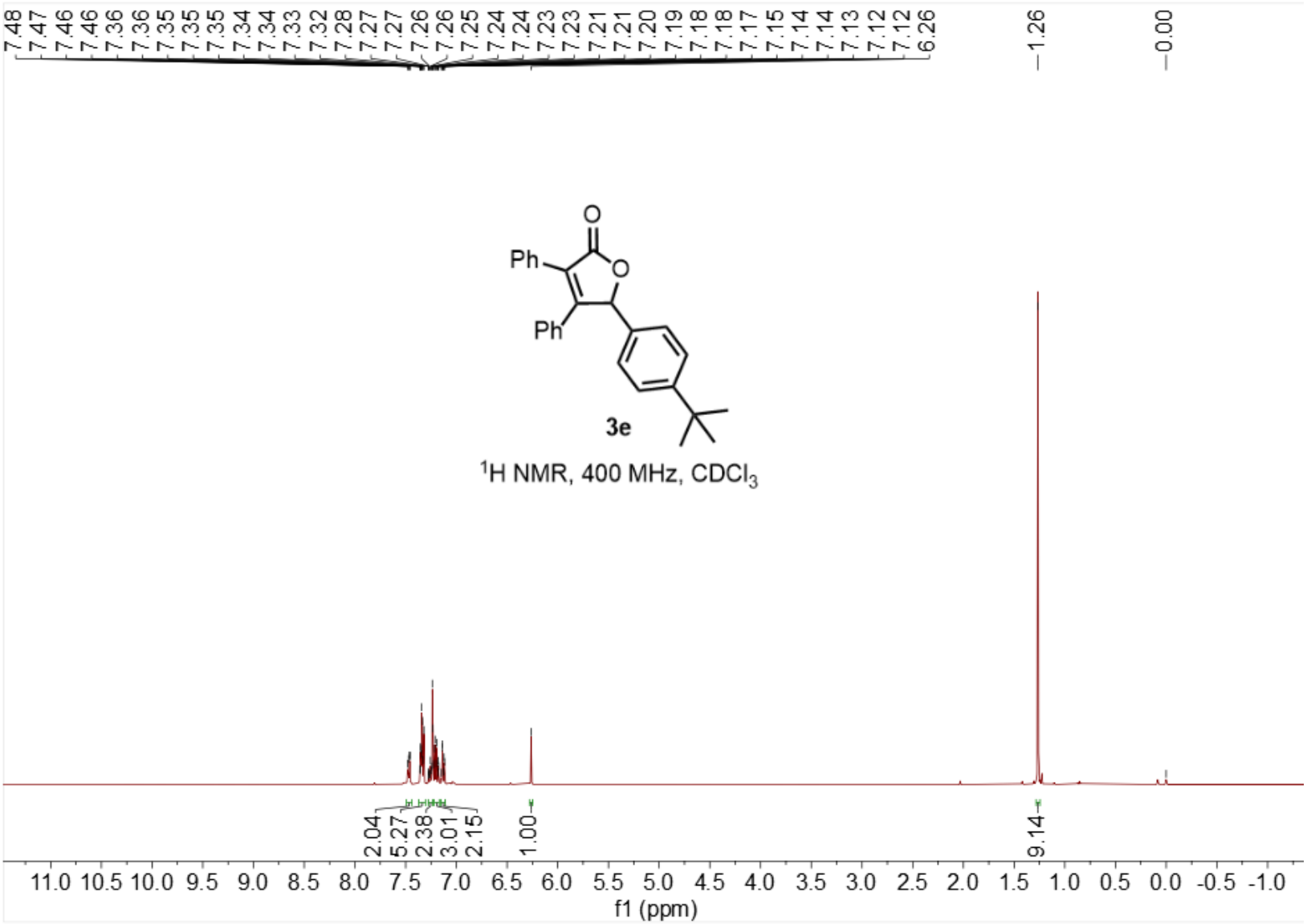
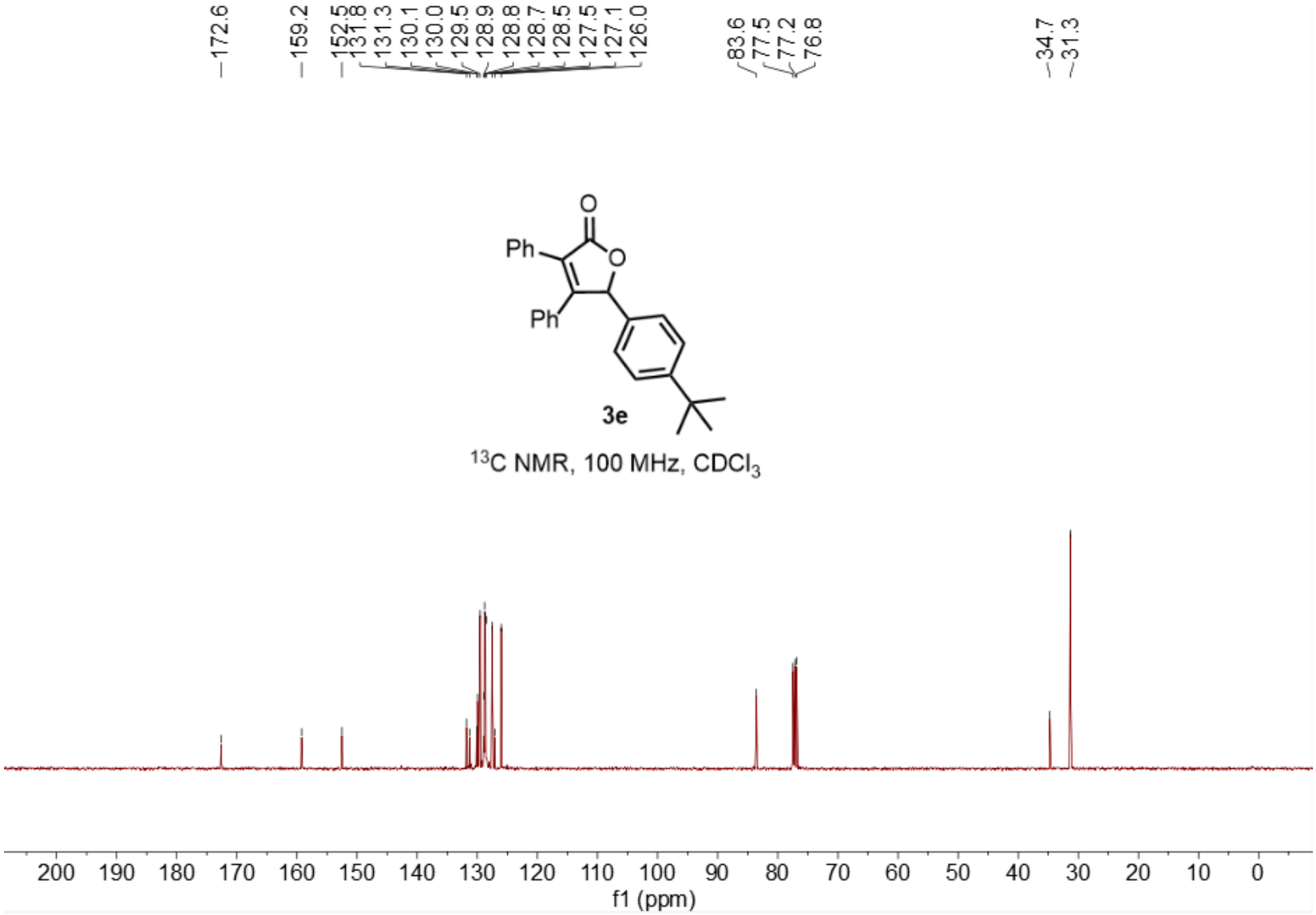
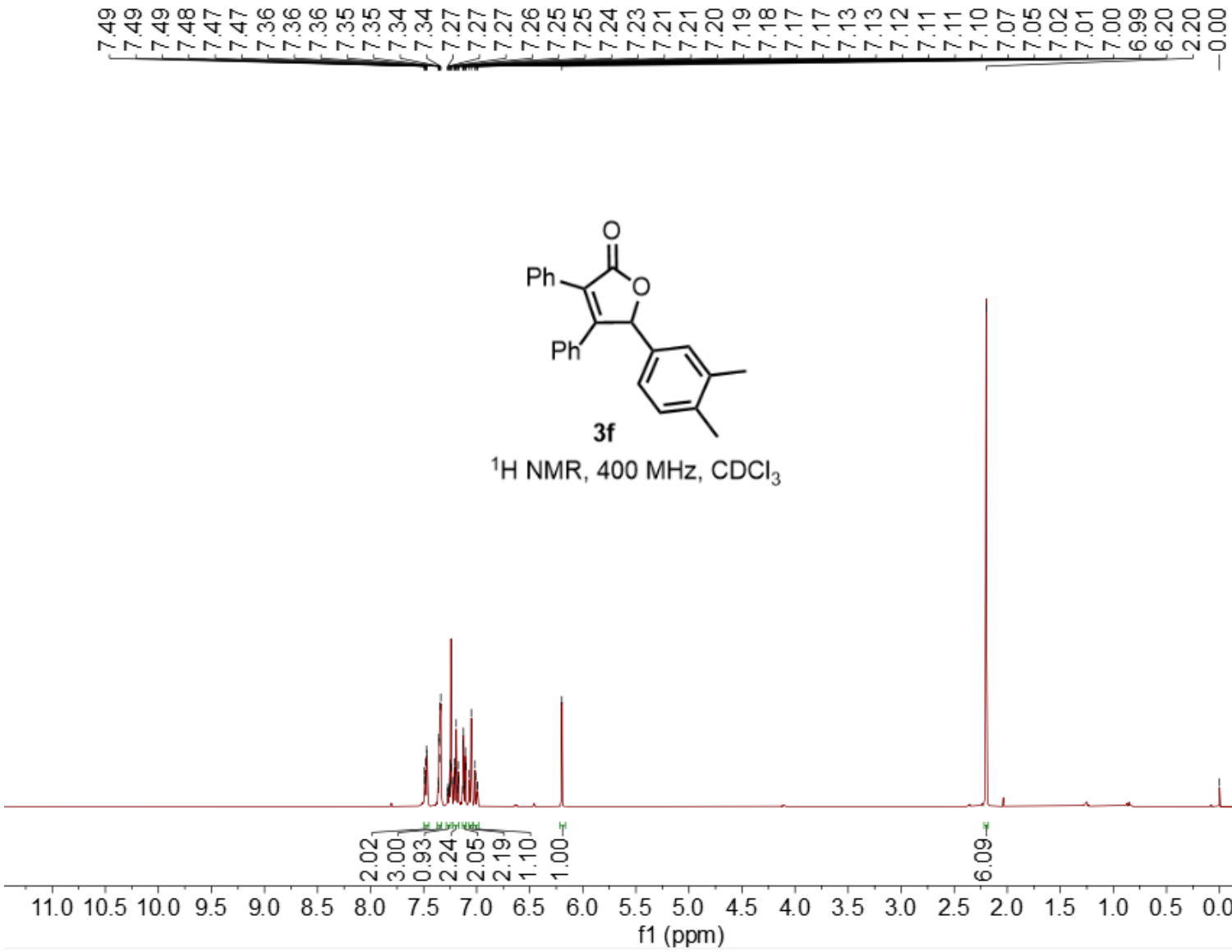
6.23
2.29
0.00
O
Ph
O
Ph
3b
1H NMR, 400 MHz, CDCl3
2.11
3.24
5.07
4.20
1.00
3.05
1.5
10.5
9.5
8.5
7.5
6.5
5.5
4.5
3.5
2.5
1.5
0.5
-0.5
-1
f1 (ppm)

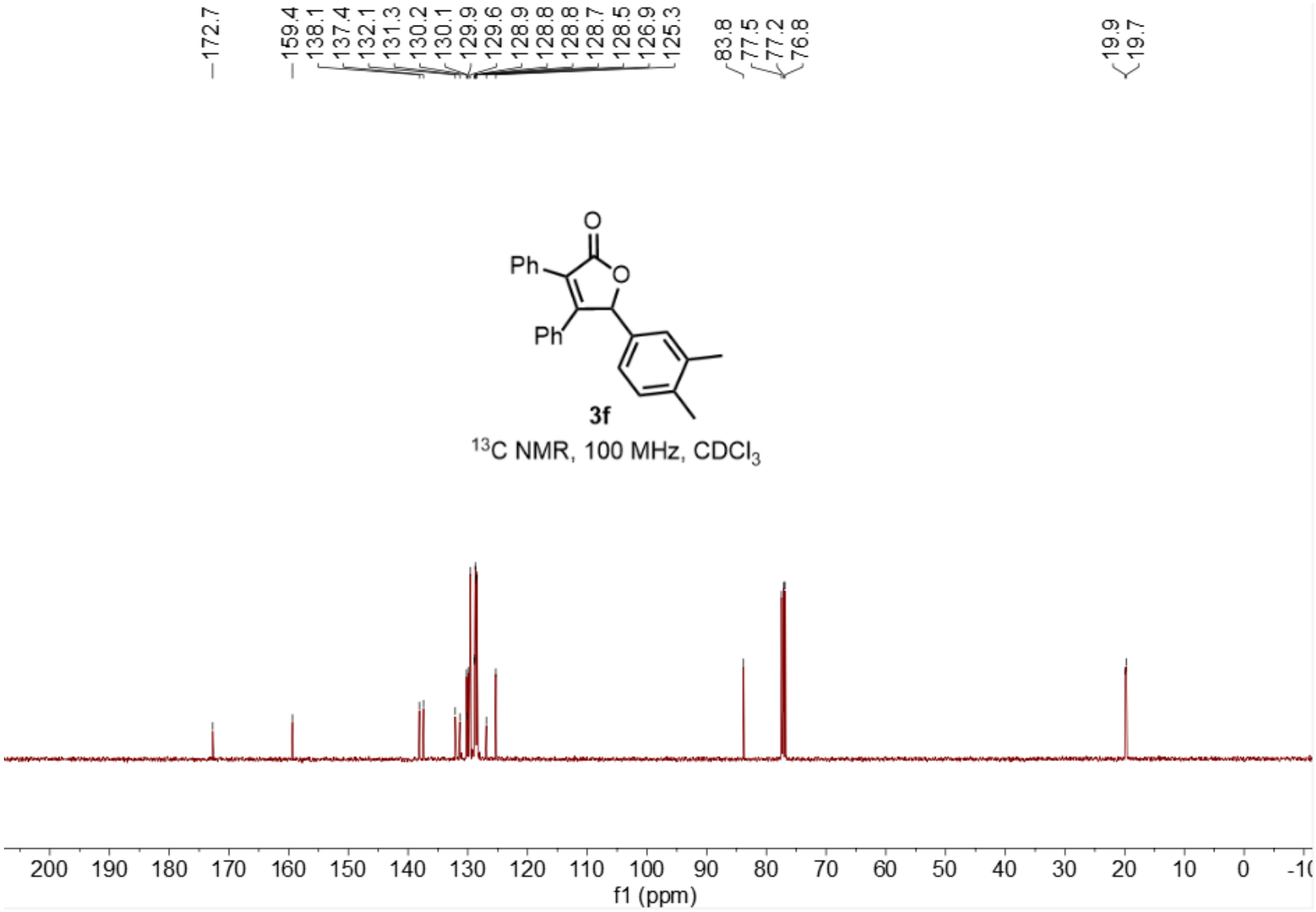

7.49
7.49
7.48
7.47
7.47
7.46
7.36
7.35
7.35
7.34
7.34
7.33
7.28
7.27
7.27
7.26
7.25
7.25
7.21
7.20
7.19
7.18
7.18
7.17
7.12
7.11
7.11
7.11
7.10
7.09
7.09
7.08
7.06
6.21
2.29
0.00
O
Ph
O
Ph
3c
1H NMR, 400 MHz, CDCl3
2.20
3.14
1.01
3.32
5.30
1.00
3.08
11.0 10.5 10.0 9.5 9.0 8.5 8.0 7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 0.5 0.0 -0.5 -1.0
f1 (ppm)


7.49 7.49 7.49 7.48 7.47 7.47 7.37 7.36 7.36 7.36 7.35 7.35 7.35 7.34 7.27 7.27 7.27 7.26 7.25 7.25 7.24 7.23 7.22 7.21 7.20 7.20 7.19 7.19 7.18 7.17 7.17 7.16 7.16 7.12 7.11 7.11 7.10 7.10 7.09 7.09 7.08 7.07 7.07 6.54 2.44 0.00
O
Ph
O
Ph
3d
1H NMR, 400 MHz, CDCl3
2.14 3.20 1.20 4.17 4.16 1.00 3.08
11.0 10.5 10.0 9.5 9.0 8.5 8.0 7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 0.5 0.0 -0.5 -1.0
f1 (ppm)


7.48 7.47 7.46 7.46 7.36 7.36 7.35 7.35 7.35 7.34 7.34 7.33 7.32 7.28 7.27 7.27 7.26 7.26 7.25 7.24 7.24 7.23 7.23 7.21 7.21 7.20 7.19 7.18 7.18 7.17 7.15 7.14 7.14 7.13 7.12 7.12 6.26
1.26
0.00
O
Ph
O
Ph
3e
^{1}H NMR, 400 MHz, $CDCl_3$
2.04 5.27 2.38 3.01 2.15 1.00 9.14
11.0 10.5 10.0 9.5 9.0 8.5 8.0 7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 0.5 0.0 -0.5 -1.0
f1 (ppm)


172.6
159.2
152.5
131.8
131.3
130.1
130.0
129.5
128.9
128.8
128.7
128.5
127.5
127.1
126.0
83.6
77.5
77.2
76.8
34.7
31.3
O
Ph
O
Ph
3e
13C NMR, 100 MHz, CDCl3
200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0
f1 (ppm)


7.49 7.49 7.49 7.48 7.47 7.47 7.36 7.36 7.36 7.35 7.35 7.34 7.34 7.27 7.27 7.27 7.26 7.25 7.25 7.24 7.23 7.21 7.21 7.20 7.19 7.18 7.17 7.17 7.13 7.13 7.12 7.11 7.11 7.10 7.07 7.05 7.02 7.01 7.00 6.99 6.20 2.20 0.00
O
Ph
O
Ph
3f
1H NMR, 400 MHz, CDCl3
2.02 3.00 0.93 2.24 2.05 2.19 1.10 1.00 6.09
11.0 10.5 10.0 9.5 9.0 8.5 8.0 7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 0.5 0.0 -0.5 -1.0
f1 (ppm)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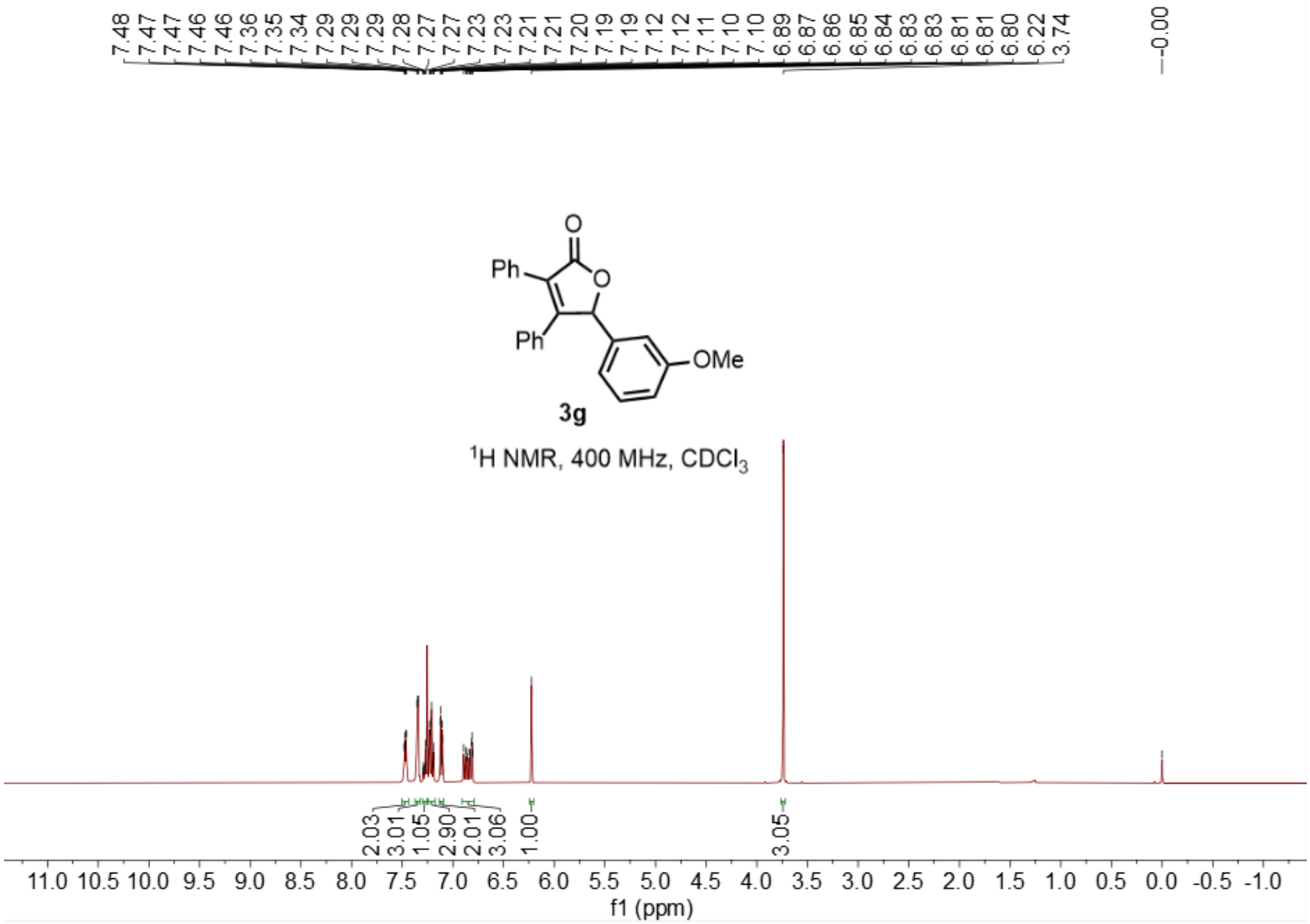
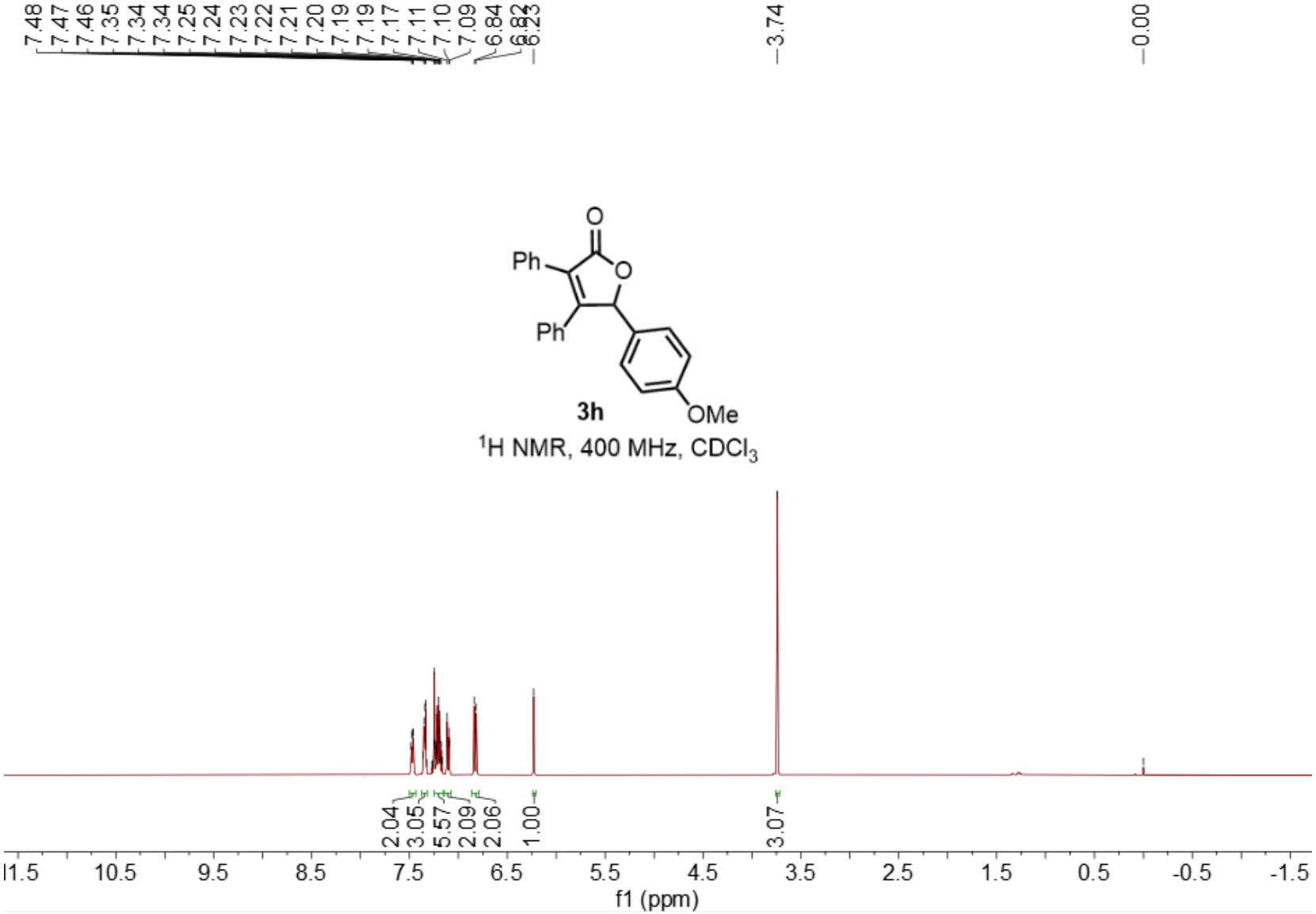
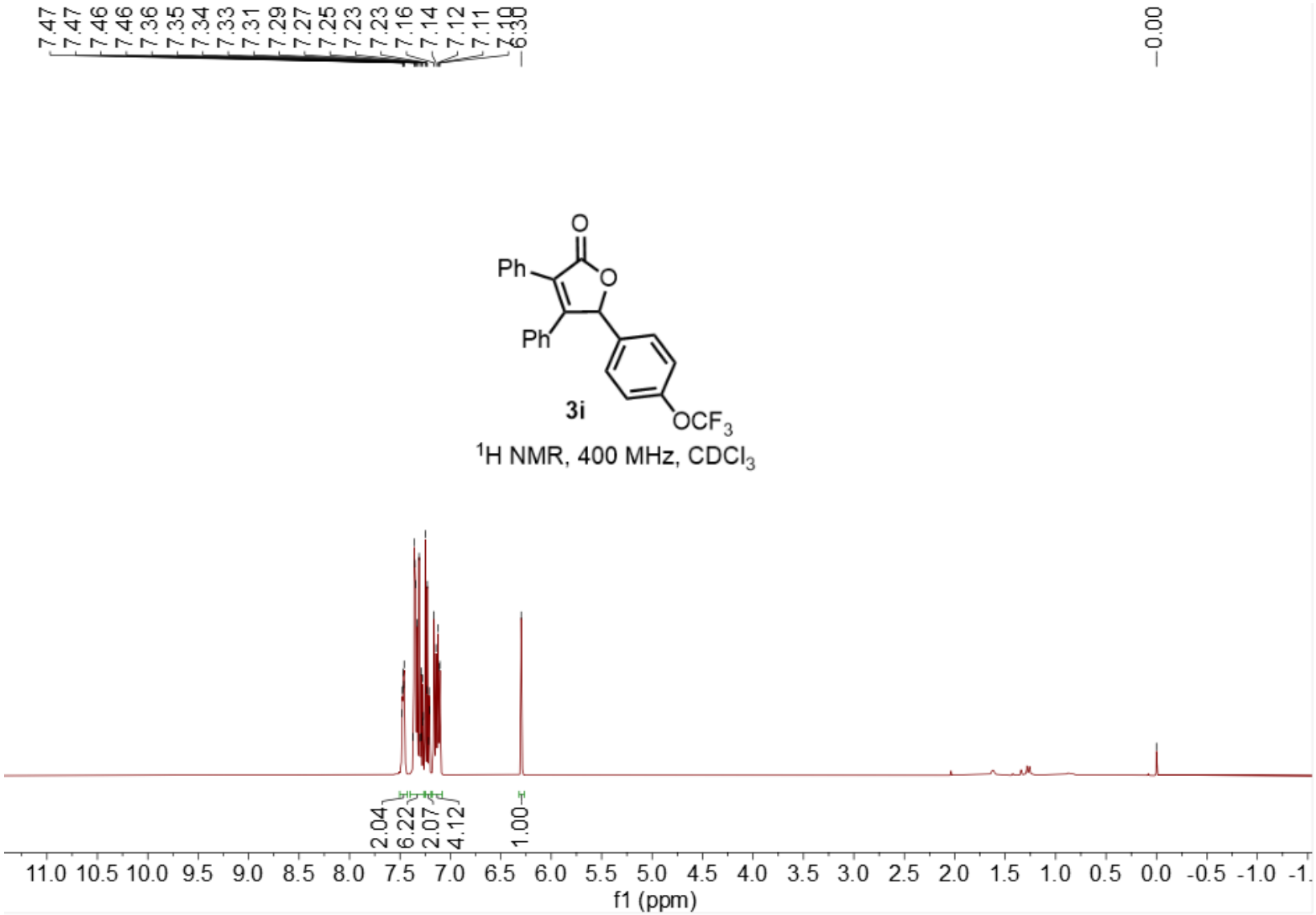
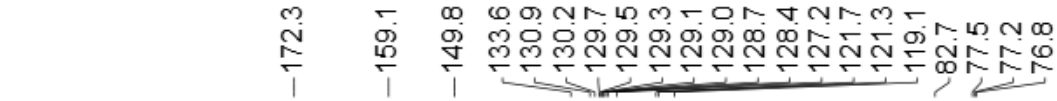
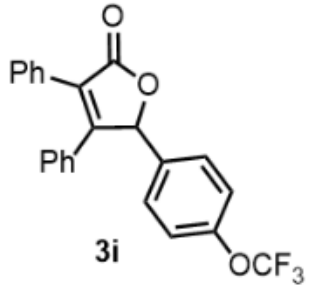
159.4
138.1
137.4
132.1
131.3
130.2
130.1
129.9
129.6
128.9
128.8
128.8
128.7
128.5
126.9
125.3
83.8
77.5
77.2
76.8
19.9
19.7
O
Ph
O
Ph
3f
13C NMR, 100 MHz, CDCl3
200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0 -10
f1 (ppm)


7.48
7.47
7.47
7.46
7.46
7.36
7.35
7.34
7.29
7.29
7.29
7.28
7.27
7.27
7.23
7.23
7.21
7.21
7.20
7.19
7.19
7.12
7.12
7.11
7.10
7.10
6.89
6.87
6.86
6.85
6.84
6.83
6.83
6.81
6.81
6.80
6.22
3.74
-0.00
O
Ph
O
Ph
OMe
3g
1H NMR, 400 MHz, CDCl3
2.03
3.01
1.05
2.90
2.01
3.06
1.00
3.05
11.0 10.5 10.0 9.5 9.0 8.5 8.0 7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 0.5 0.0 -0.5 -1.0
f1 (ppm)


7.48
7.47
7.46
7.35
7.34
7.34
7.25
7.24
7.23
7.22
7.21
7.20
7.19
7.19
7.17
7.11
7.10
7.09
6.84
6.82
6.23
3.74
0.00
O
Ph
O
Ph
OMe
3h
1H NMR, 400 MHz, CDCl3
2.04
3.05
5.57
2.09
2.06
1.00
3.07
11.5
10.5
9.5
8.5
7.5
6.5
5.5
4.5
3.5
2.5
1.5
0.5
-0.5
-1.5
f1 (ppm)

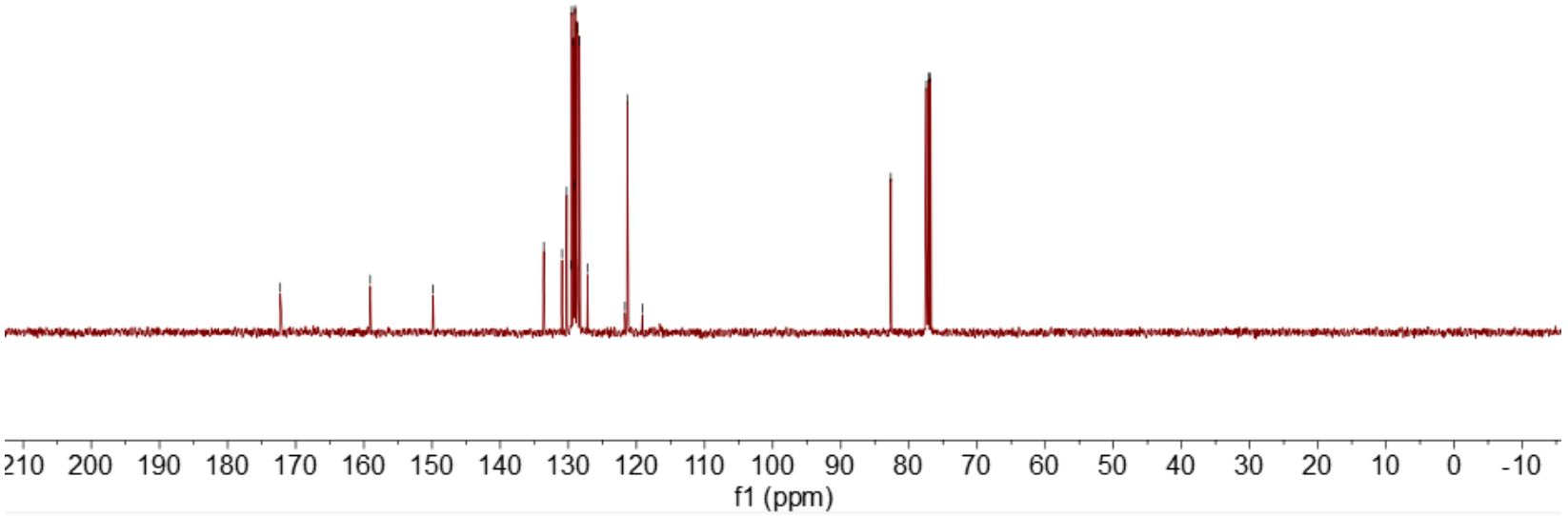

7.47
7.47
7.46
7.46
7.36
7.35
7.34
7.33
7.31
7.29
7.27
7.25
7.23
7.23
7.16
7.14
7.12
7.11
7.10
6.98
0.00
O
Ph
O
Ph
3i
OCF3
1H NMR, 400 MHz, CDCl3
2.04
6.22
2.07
4.12
1.00
11.0 10.5 10.0 9.5 9.0 8.5 8.0 7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 0.5 0.0 -0.5 -1.0 -1.
f1 (ppm)

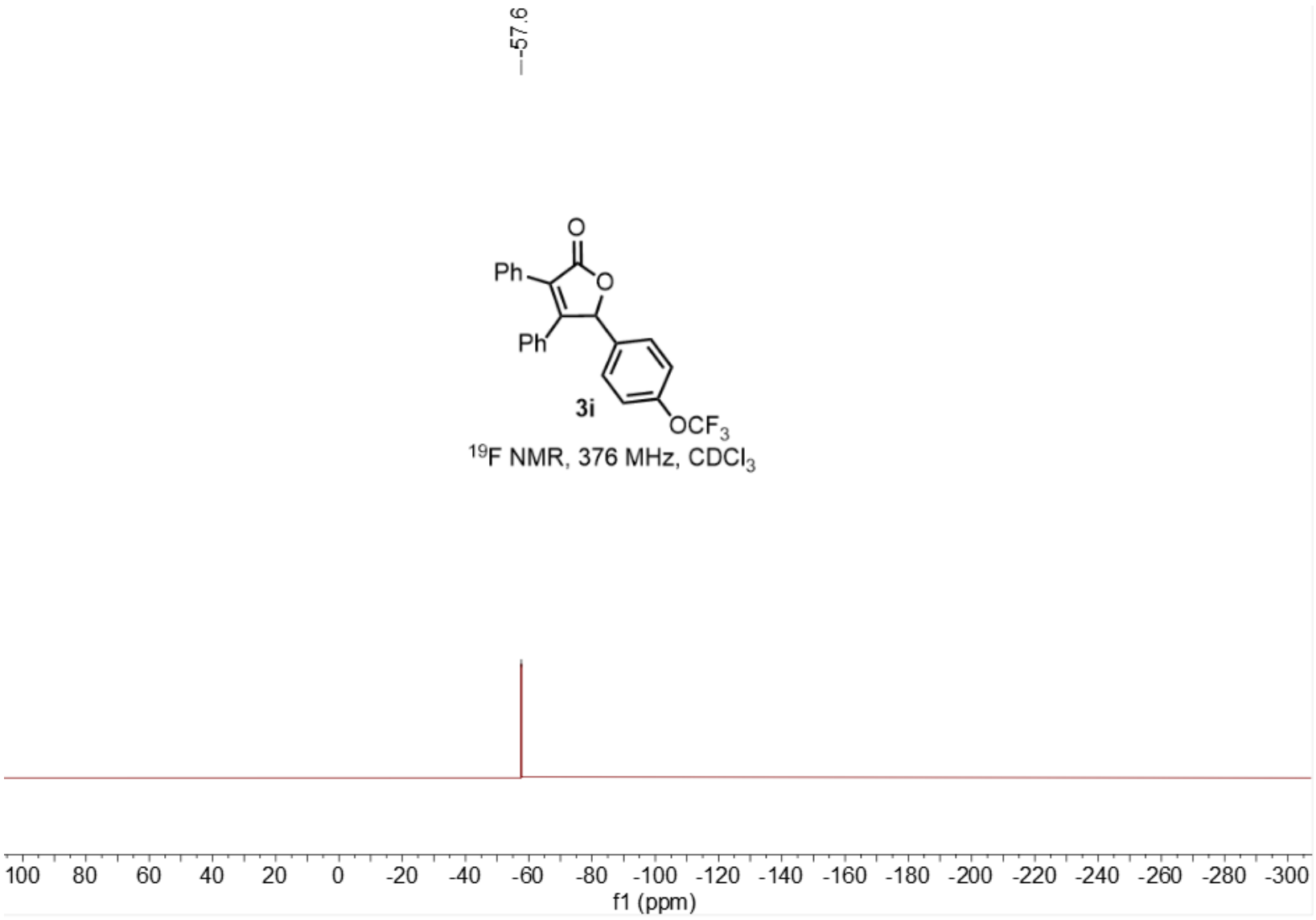




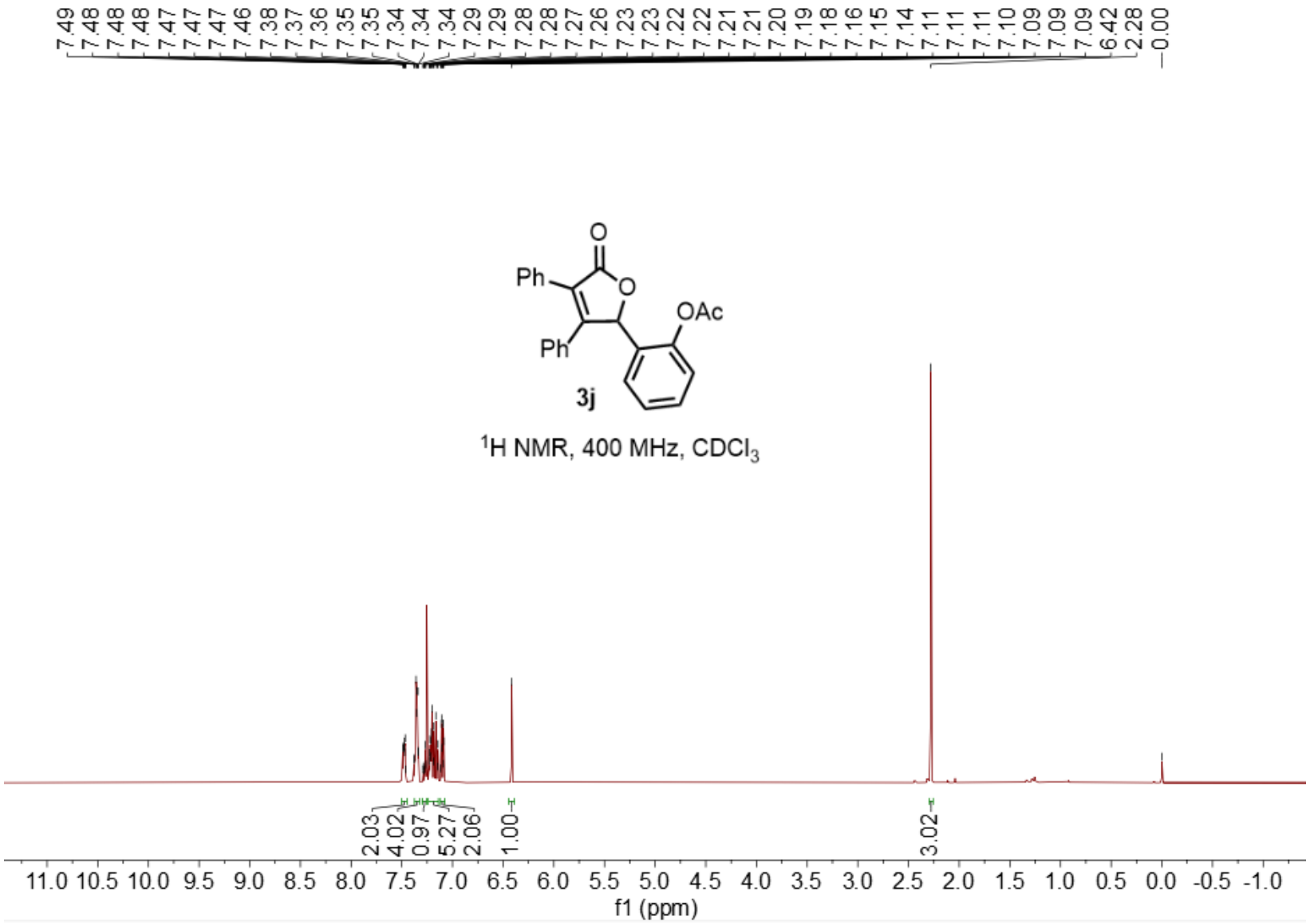
3i

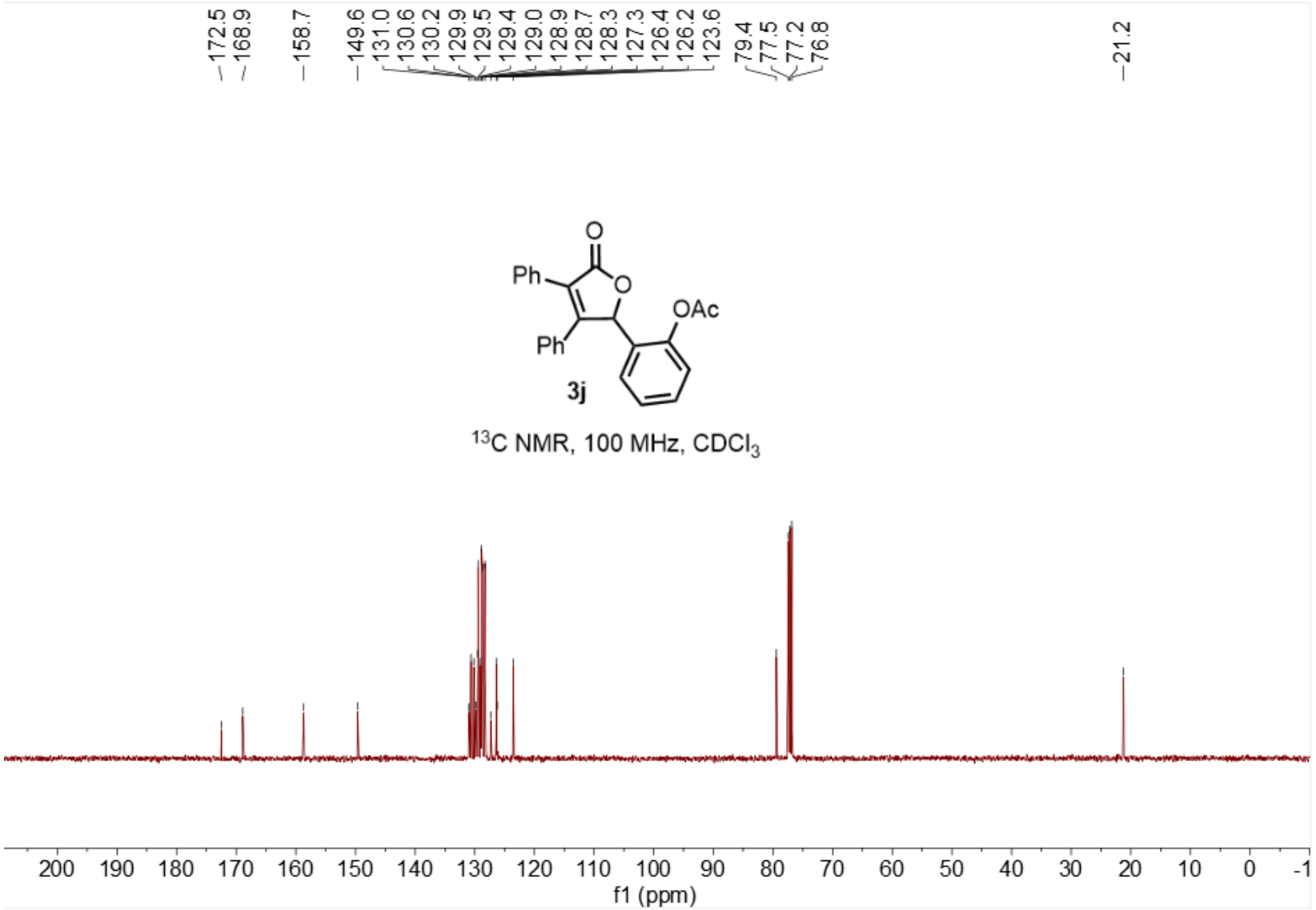
^{13}C NMR, 100 MHz, $CDCl_3$

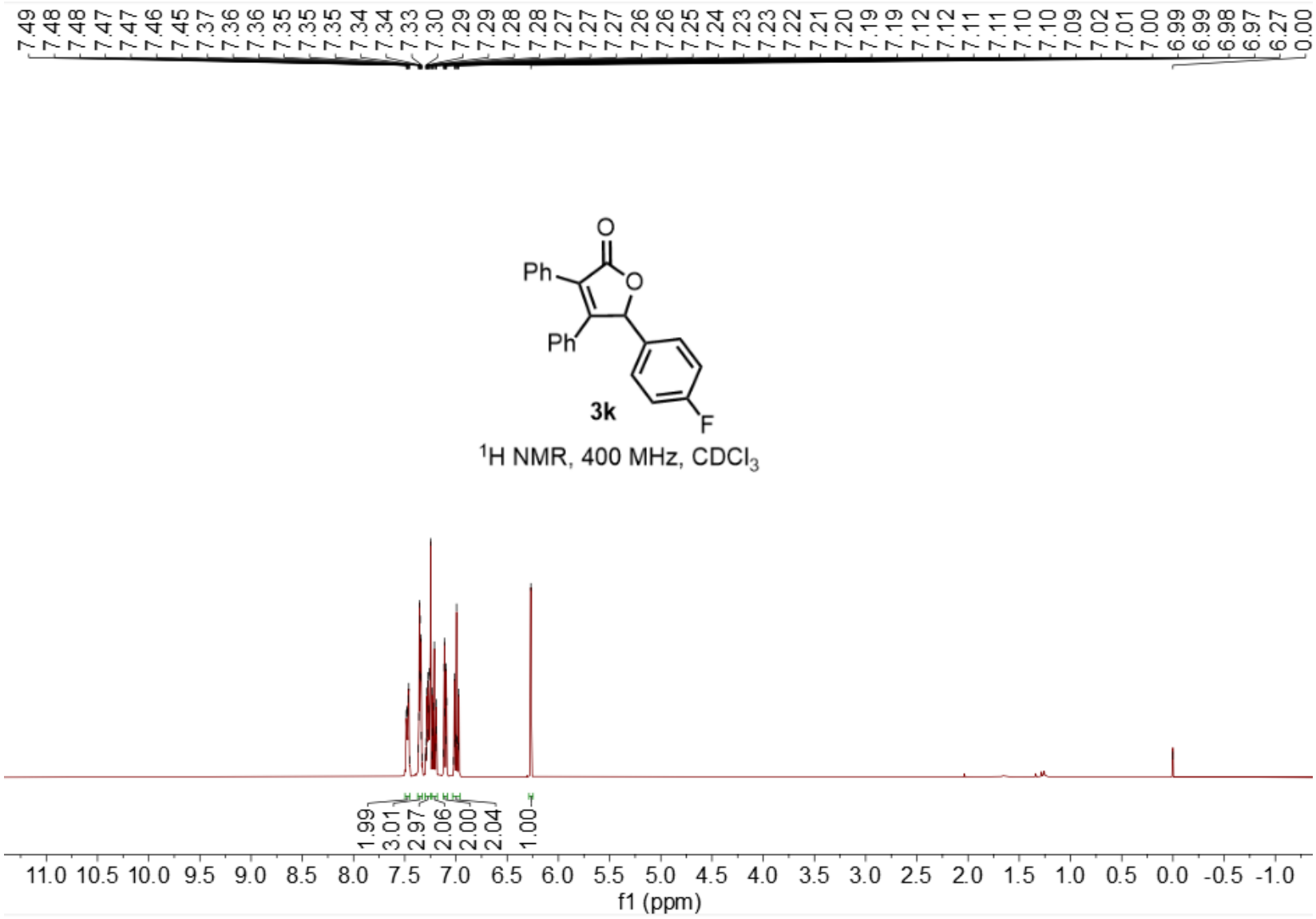




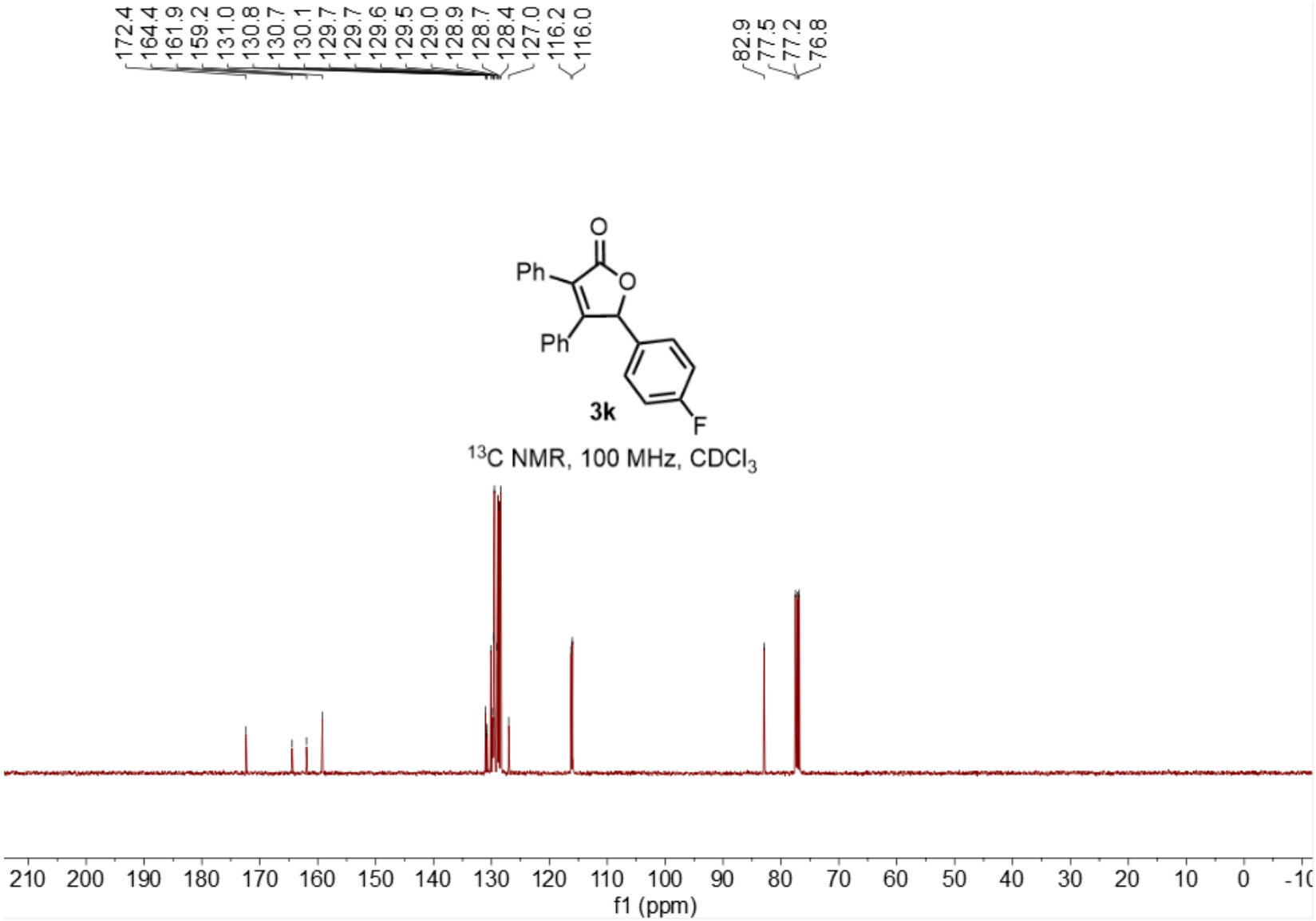
^{19}F NMR, 376 MHz, $CDCl_3$

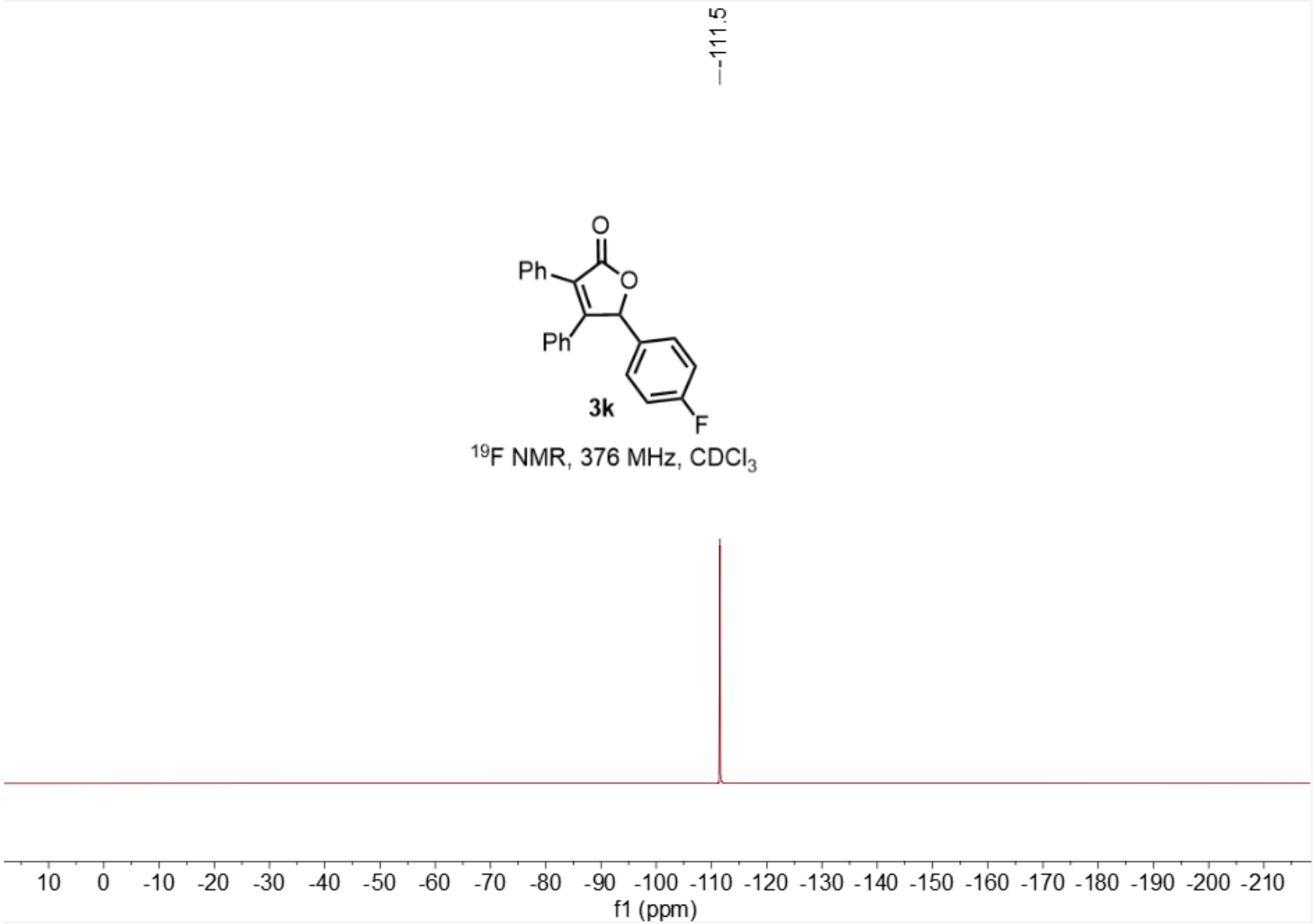

7.49 7.48 7.48 7.48 7.47 7.47 7.47 7.46 7.38 7.37 7.36 7.35 7.35 7.34 7.34 7.34 7.29 7.29 7.28 7.28 7.27 7.26 7.23 7.23 7.22 7.22 7.21 7.21 7.20 7.19 7.18 7.16 7.15 7.14 7.11 7.11 7.11 7.10 7.09 7.09 7.09 6.42 2.28 0.00
O
Ph
O
OAc
Ph
3j
1H NMR, 400 MHz, CDCl3
2.03 4.02 0.97 5.27 2.06 1.00 3.02
11.0 10.5 10.0 9.5 9.0 8.5 8.0 7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 0.5 0.0 -0.5 -1.0
f1 (ppm)

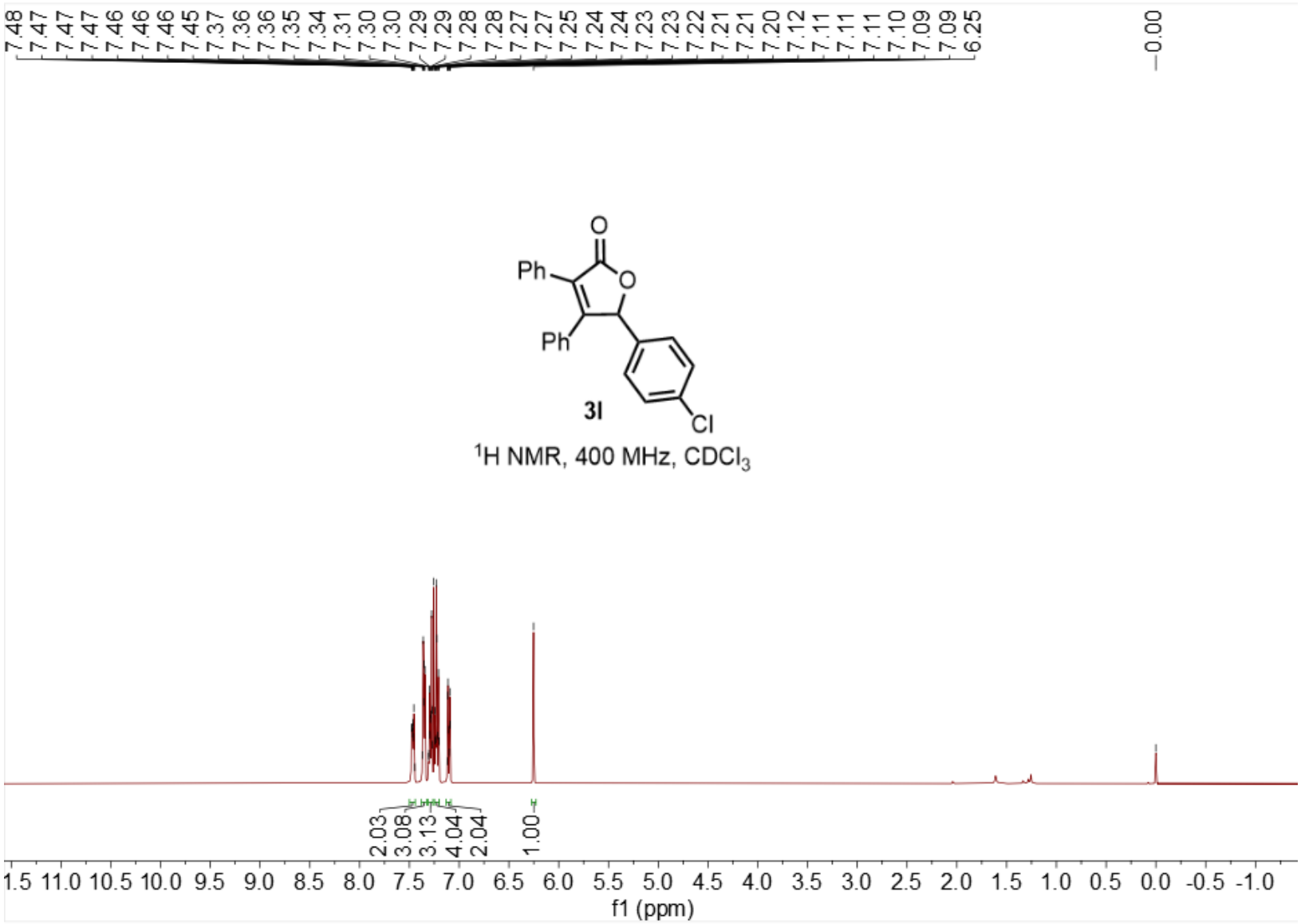

172.5
168.9
158.7
149.6
131.0
130.6
130.2
129.9
129.5
129.4
129.0
128.9
128.7
128.3
127.3
126.4
126.2
123.6
79.4
77.5
77.2
76.8
21.2
O
Ph
O
OAc
Ph
3j
^{13}C NMR, 100 MHz, $CDCl_3$
200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0 -1
f1 (ppm)

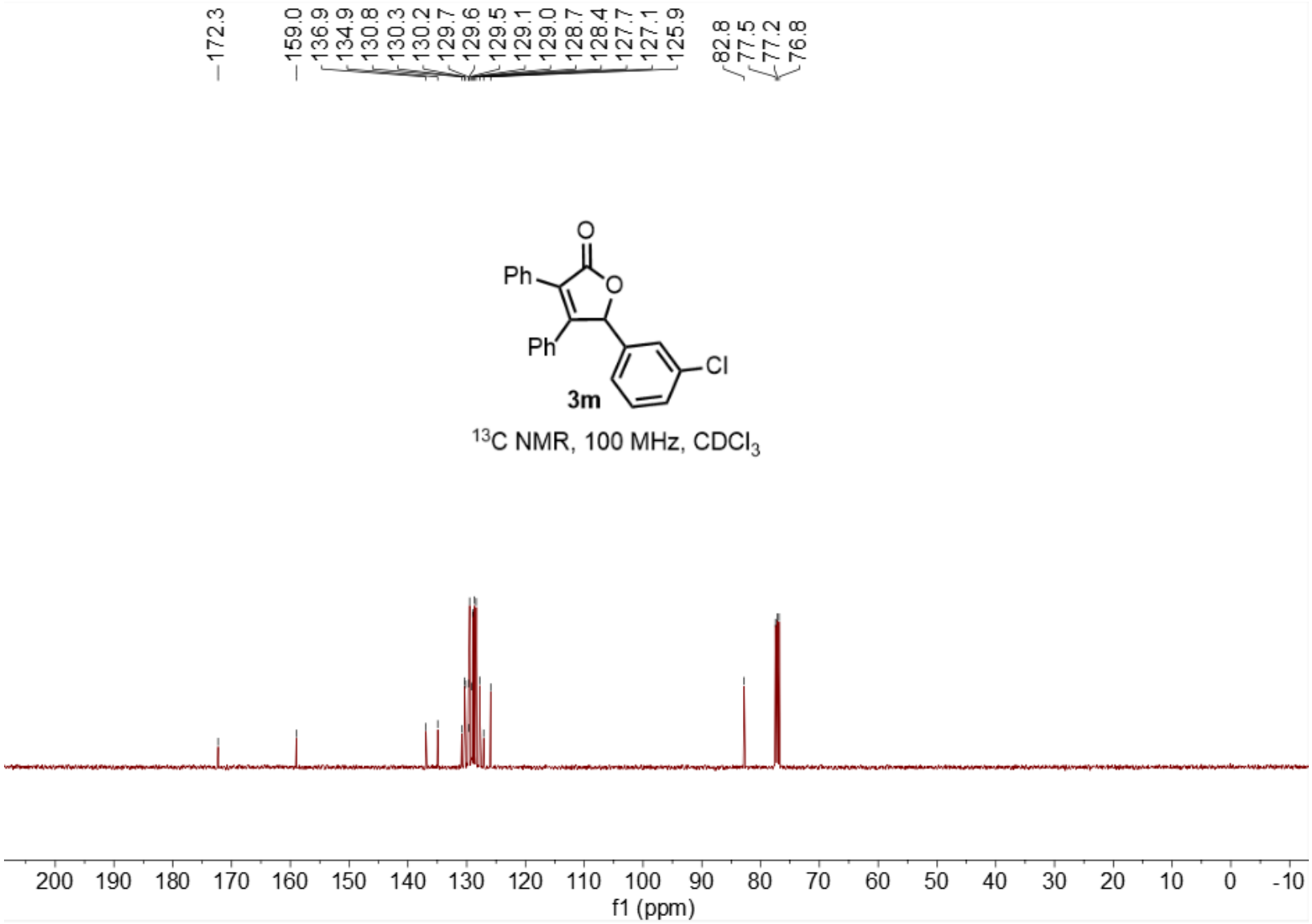


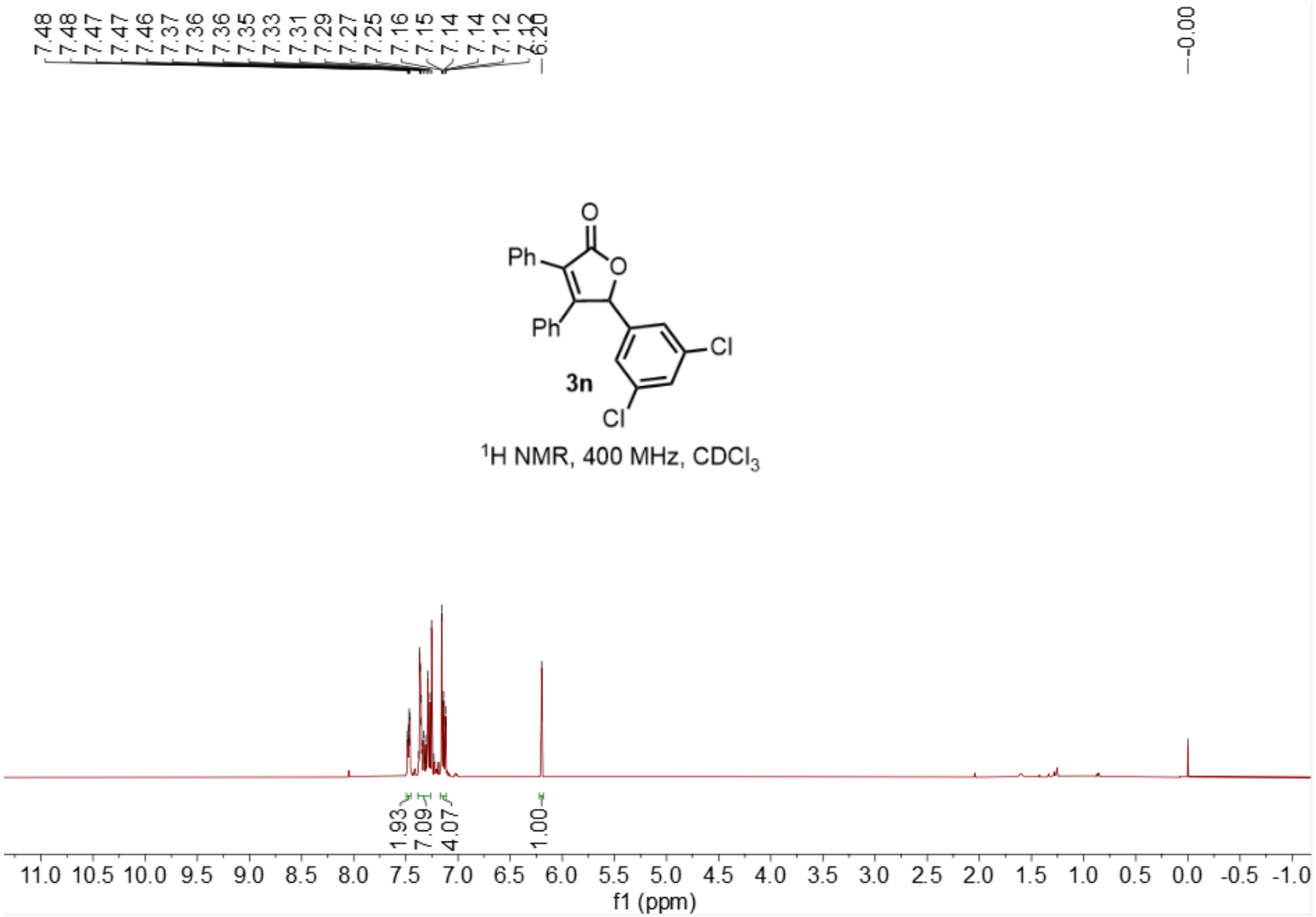
^{1}H NMR, 400 MHz, $CDCl_3$

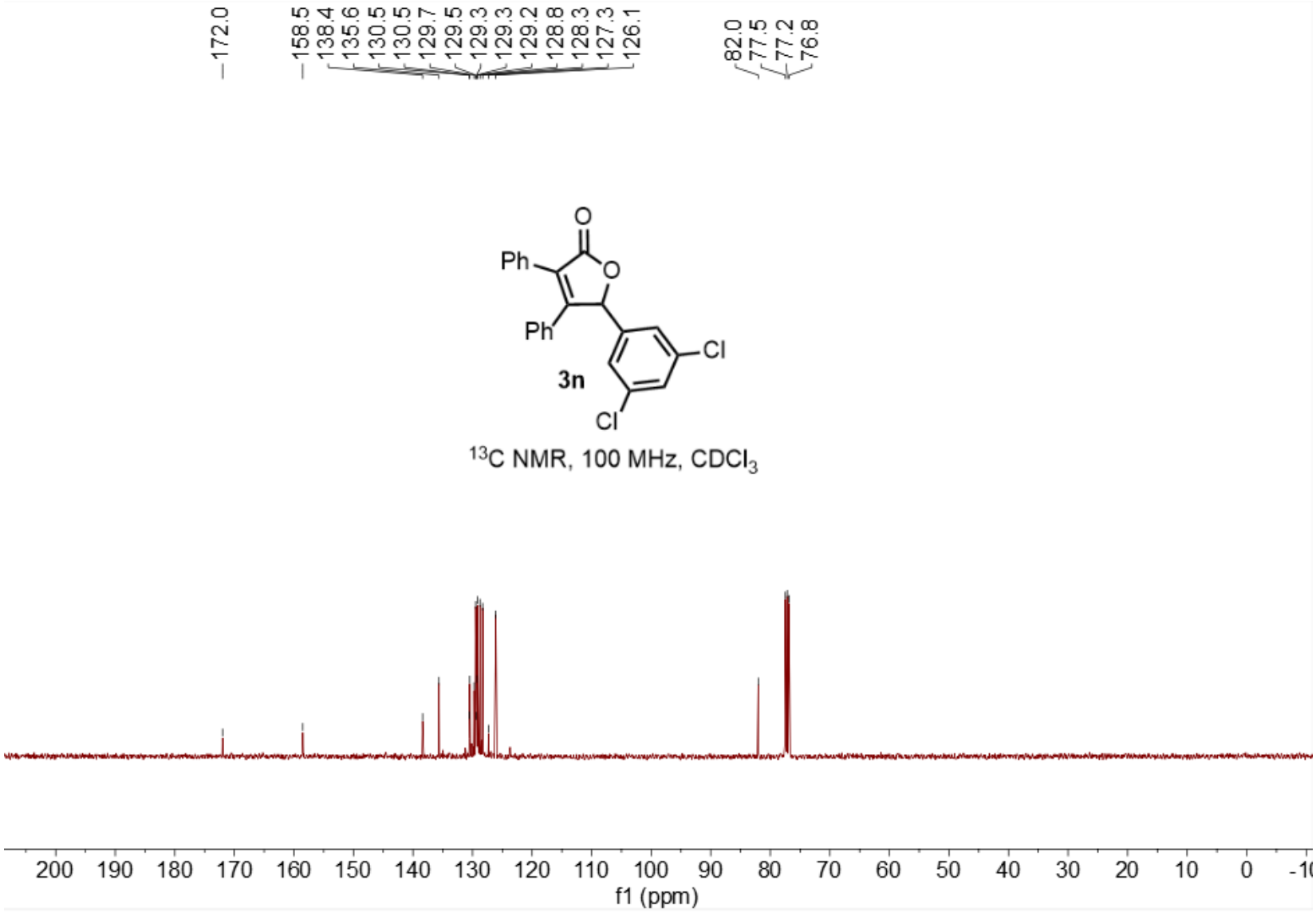

172.4
164.4
161.9
159.2
131.0
130.8
130.7
130.1
129.7
129.7
129.6
129.5
129.0
128.9
128.7
128.4
127.0
116.2
116.0
82.9
77.5
77.2
76.8
O
Ph
O
Ph
3k
F
13C NMR, 100 MHz, CDCl3
210 200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0 -10
f1 (ppm)


-111.5
O
Ph
O
Ph
3k
F
19F NMR, 376 MHz, CDCl3
10 0 -10 -20 -30 -40 -50 -60 -70 -80 -90 -100 -110 -120 -130 -140 -150 -160 -170 -180 -190 -200 -210
f1 (ppm)

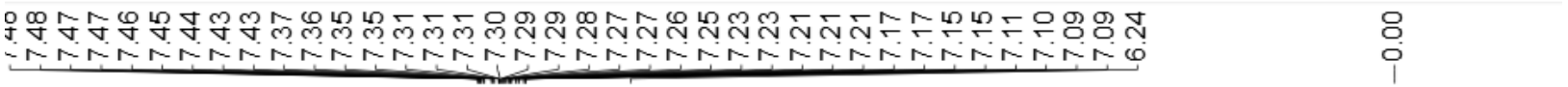

7.48 7.47 7.47 7.47 7.46 7.46 7.46 7.45 7.37 7.36 7.36 7.35 7.34 7.31 7.30 7.30 7.29 7.29 7.28 7.28 7.27 7.27 7.25 7.24 7.24 7.23 7.23 7.22 7.21 7.21 7.20 7.12 7.11 7.11 7.11 7.10 7.09 7.09 6.25
0.00
O
Ph
O
Ph
Cl
3l
1H NMR, 400 MHz, CDCl3
2.03 3.08 3.13 4.04 2.04 1.00
1.5 11.0 10.5 10.0 9.5 9.0 8.5 8.0 7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 0.5 0.0 -0.5 -1.0
f1 (ppm)

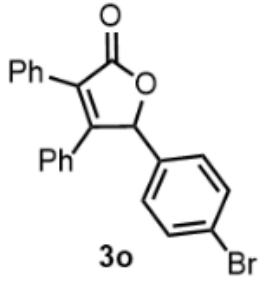

172.3
159.0
136.9
134.9
130.8
130.3
130.2
129.7
129.6
129.5
129.1
129.0
128.7
128.4
127.7
127.1
125.9
82.8
77.5
77.2
76.8
O
Ph
O
Ph
Cl
3m
^{13}C NMR, 100 MHz, $CDCl_3$
200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0 -10
f1 (ppm)


7.48
7.48
7.47
7.47
7.46
7.37
7.36
7.36
7.35
7.33
7.31
7.29
7.27
7.25
7.16
7.15
7.14
7.14
7.12
7.12
6.20
0.00
O
Ph
O
Ph
Cl
Cl
3n
1H NMR, 400 MHz, CDCl3
1.93
7.09
4.07
1.00
11.0 10.5 10.0 9.5 9.0 8.5 8.0 7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 0.5 0.0 -0.5 -1.0
f1 (ppm)



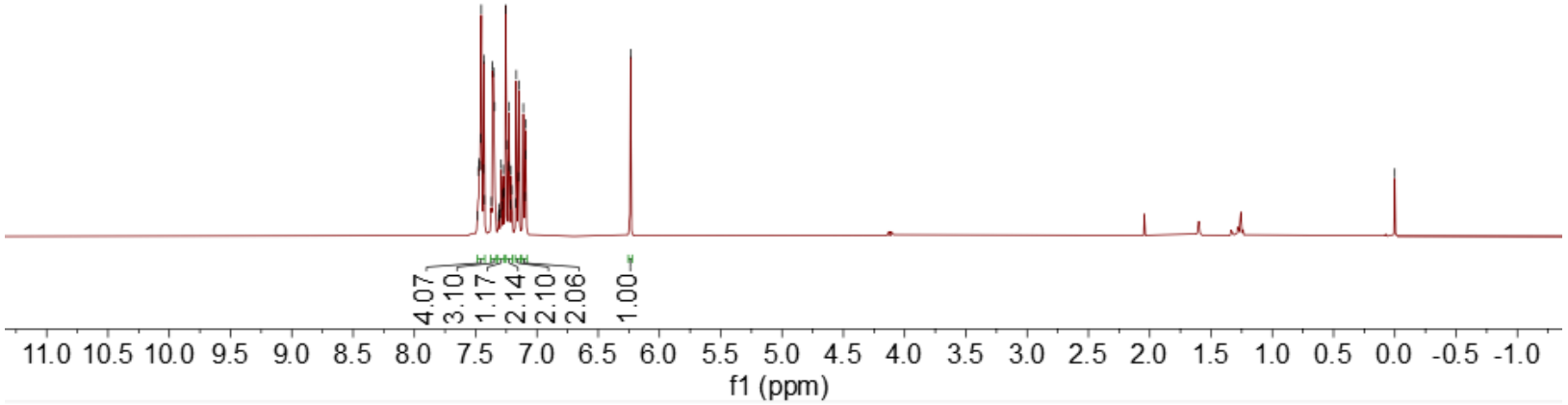
^{13}C NMR, 100 MHz, $CDCl_3$

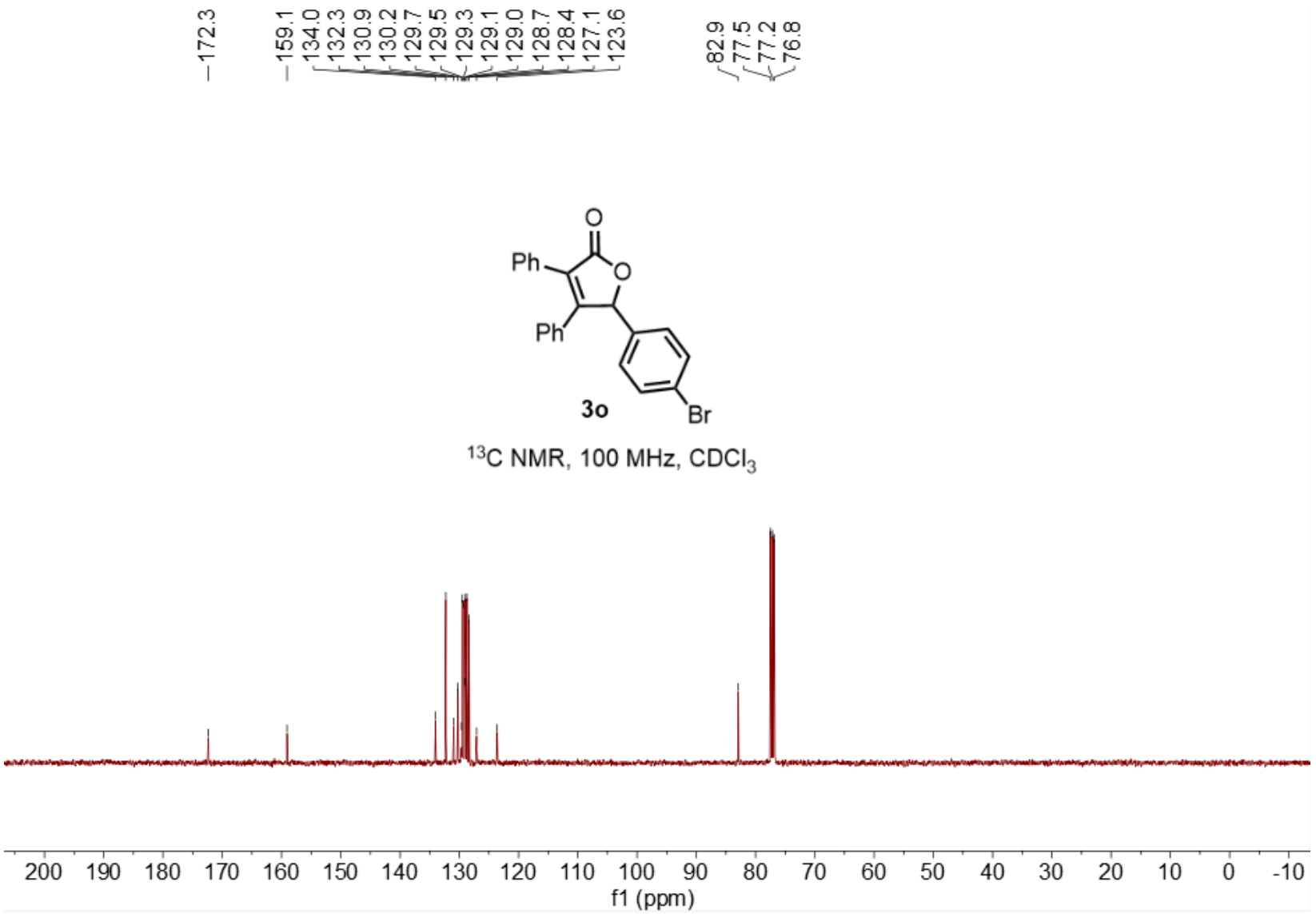


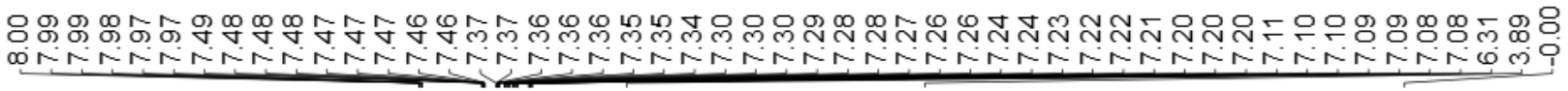


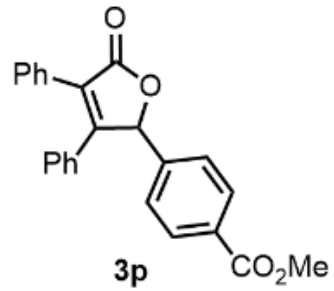
3o

^{1}H NMR, 400 MHz, $CDCl_3$



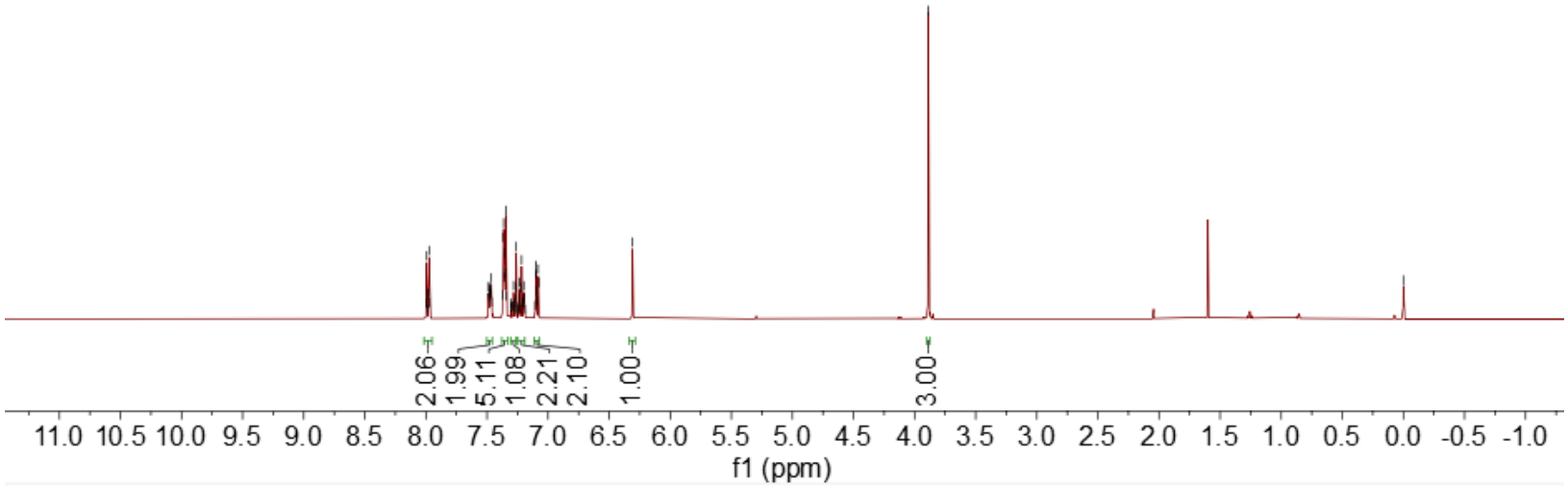

172.3
159.1
134.0
132.3
130.9
130.2
129.7
129.5
129.3
129.1
129.0
128.7
128.4
127.1
123.6
82.9
77.5
77.2
76.8
O
Ph
O
Ph
Br
3o
13C NMR, 100 MHz, CDCl3
200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0 -10
f1 (ppm)

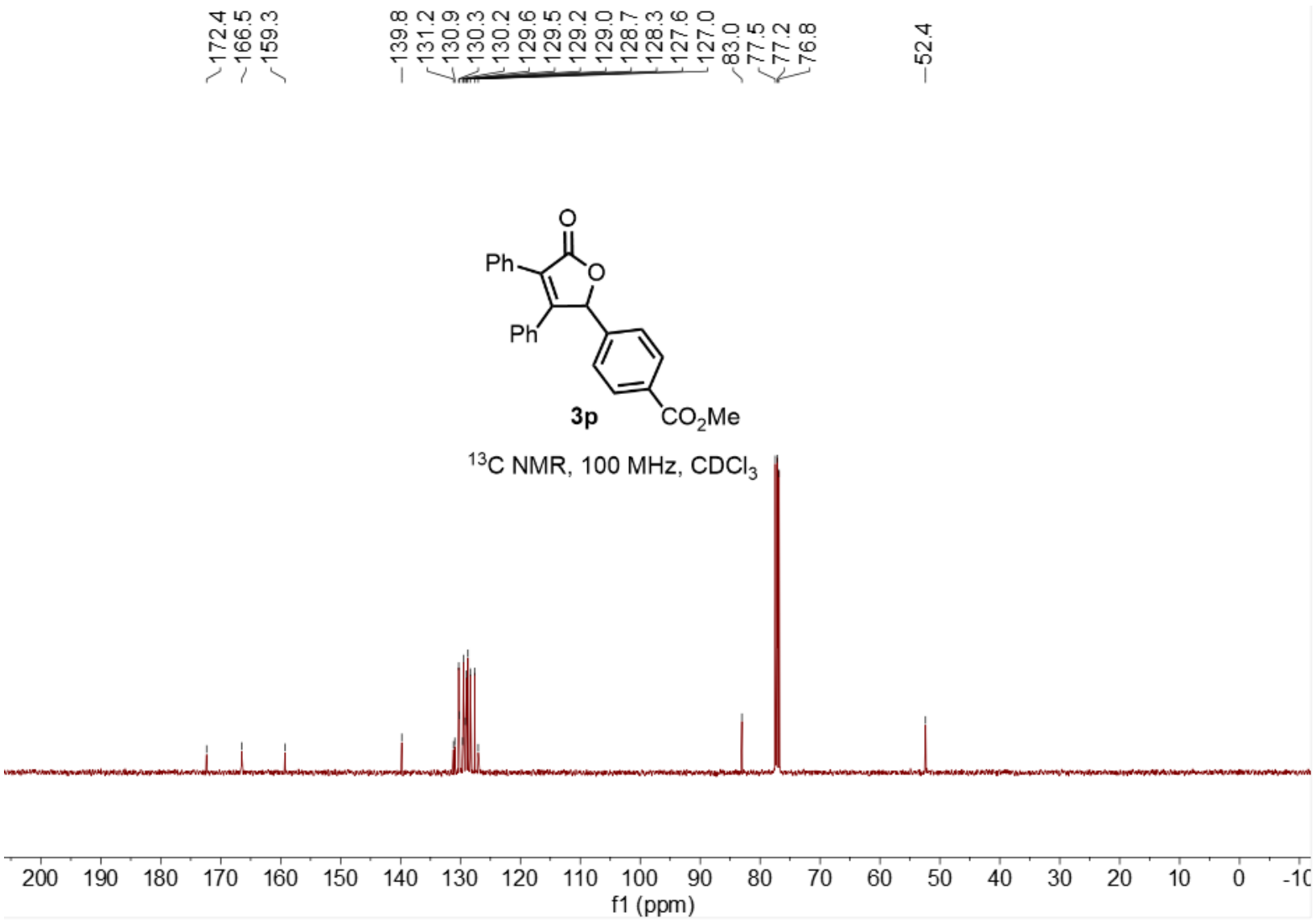


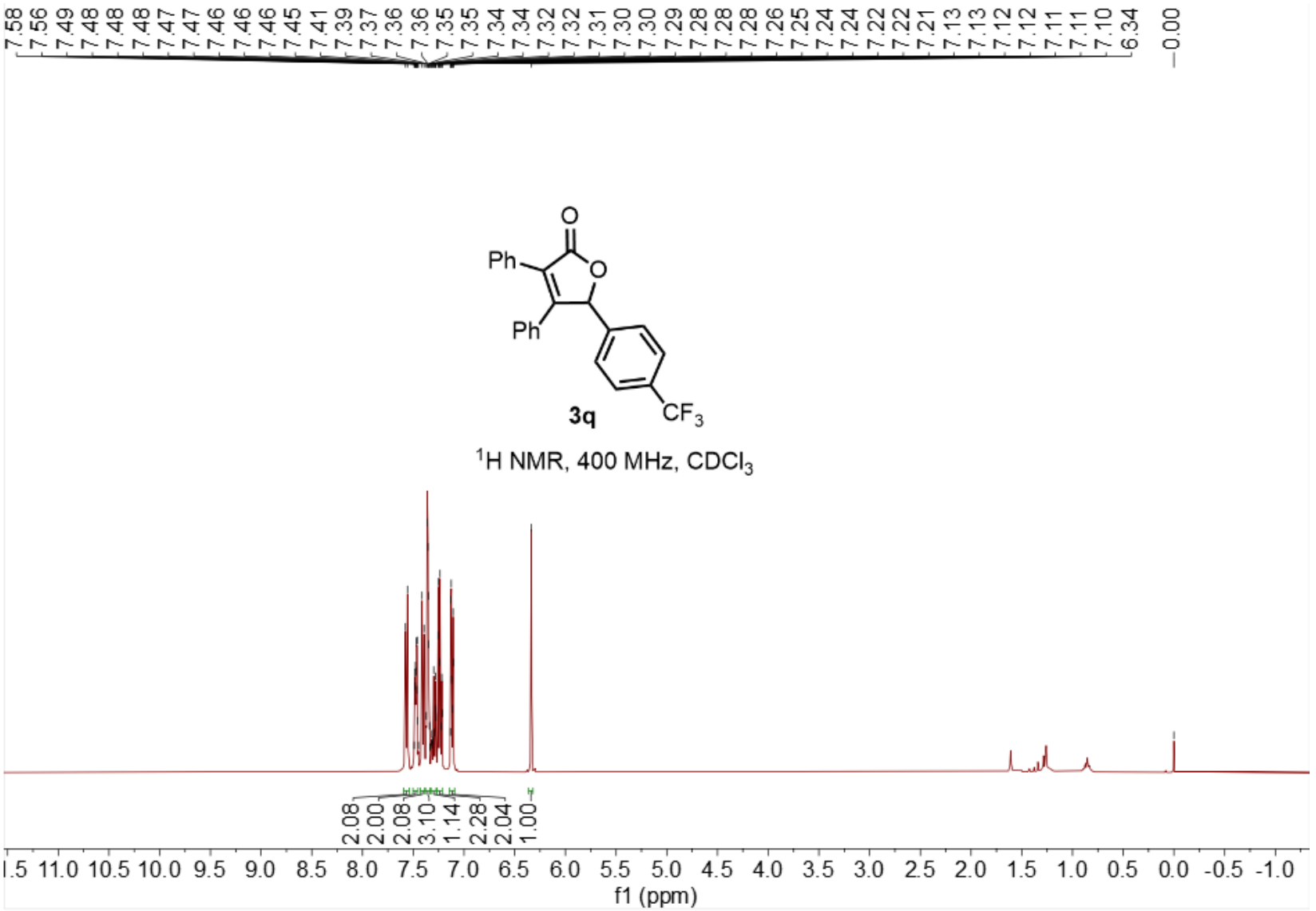


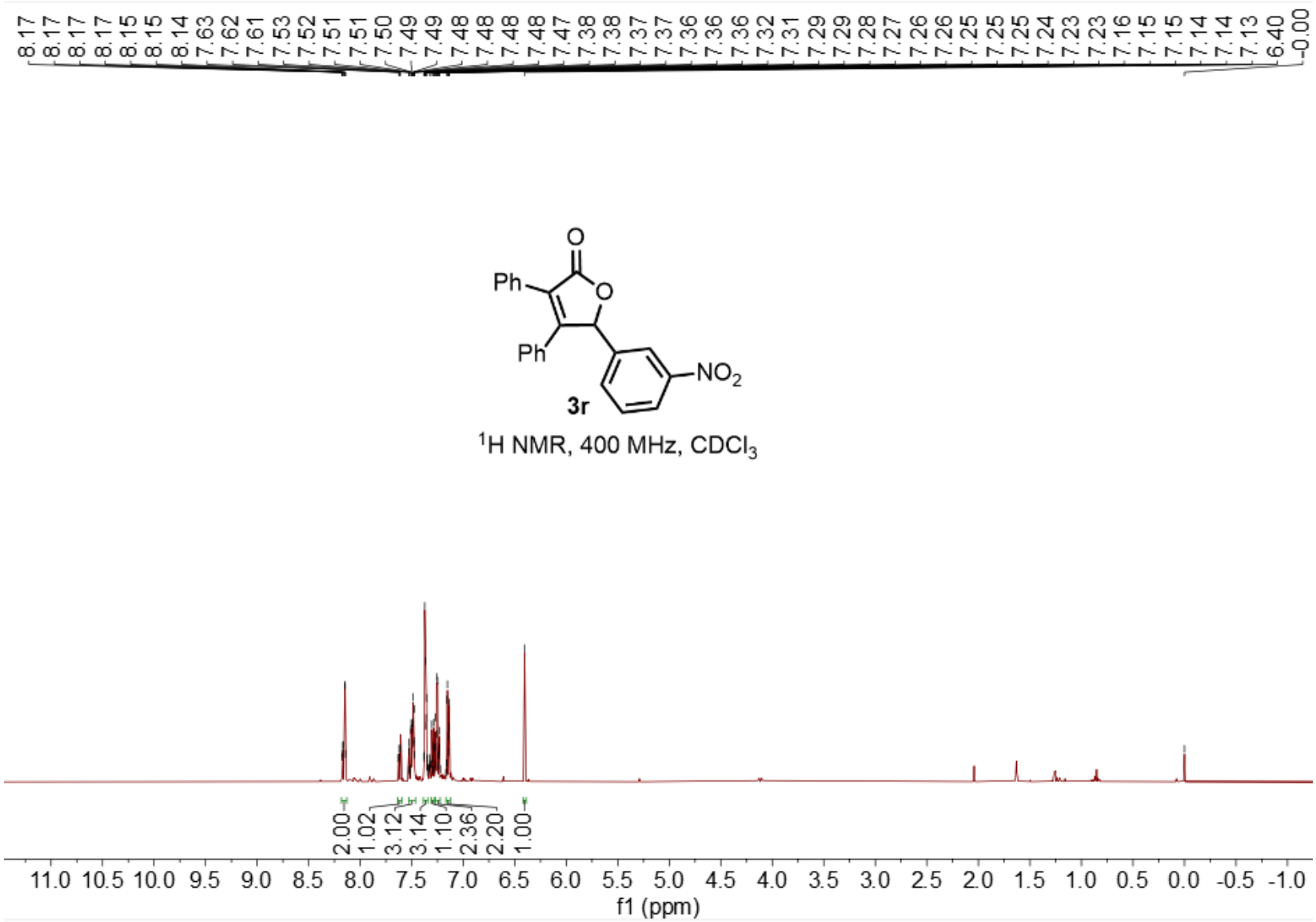
3p

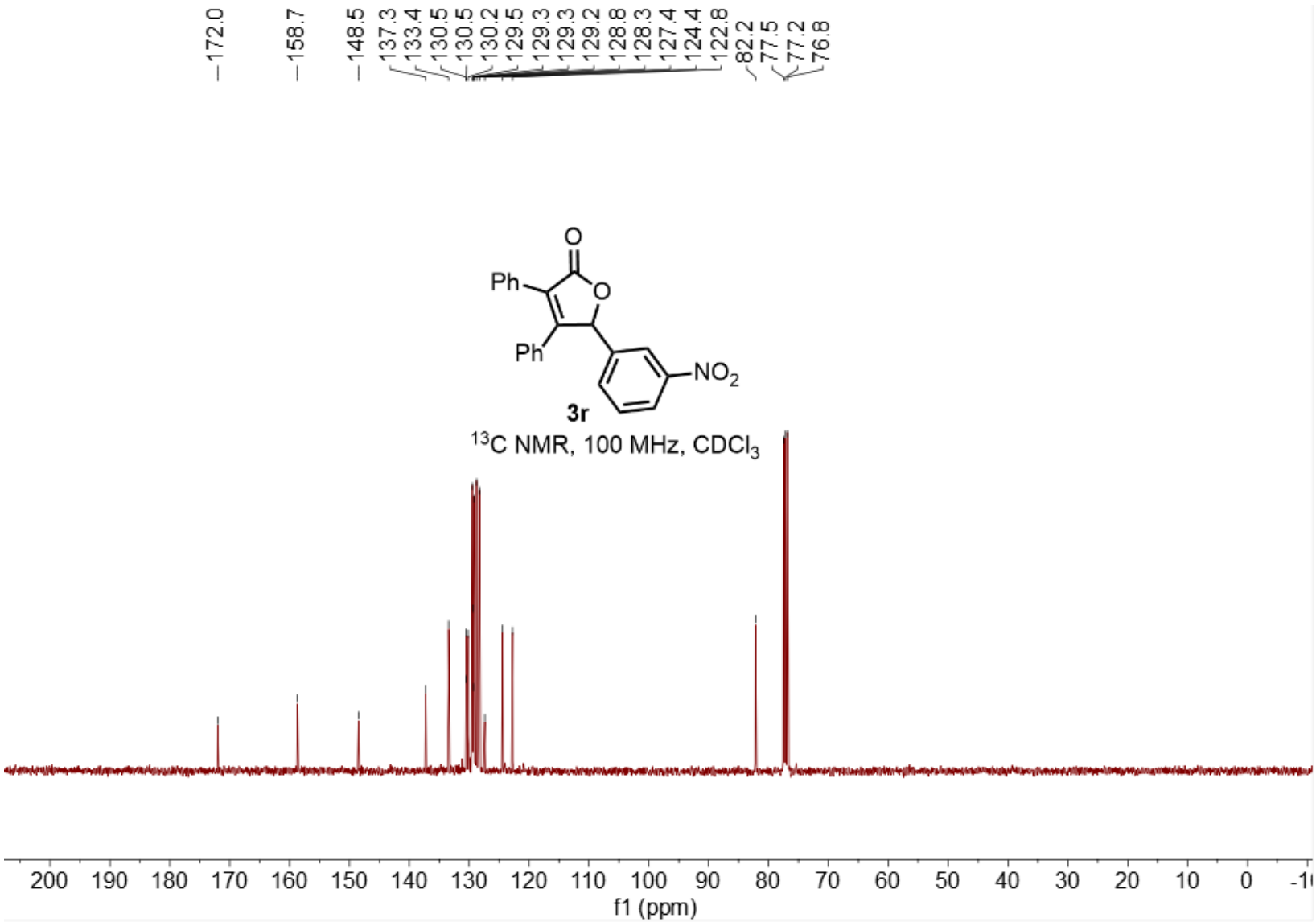
^{1}H NMR, 400 MHz, $CDCl_3$

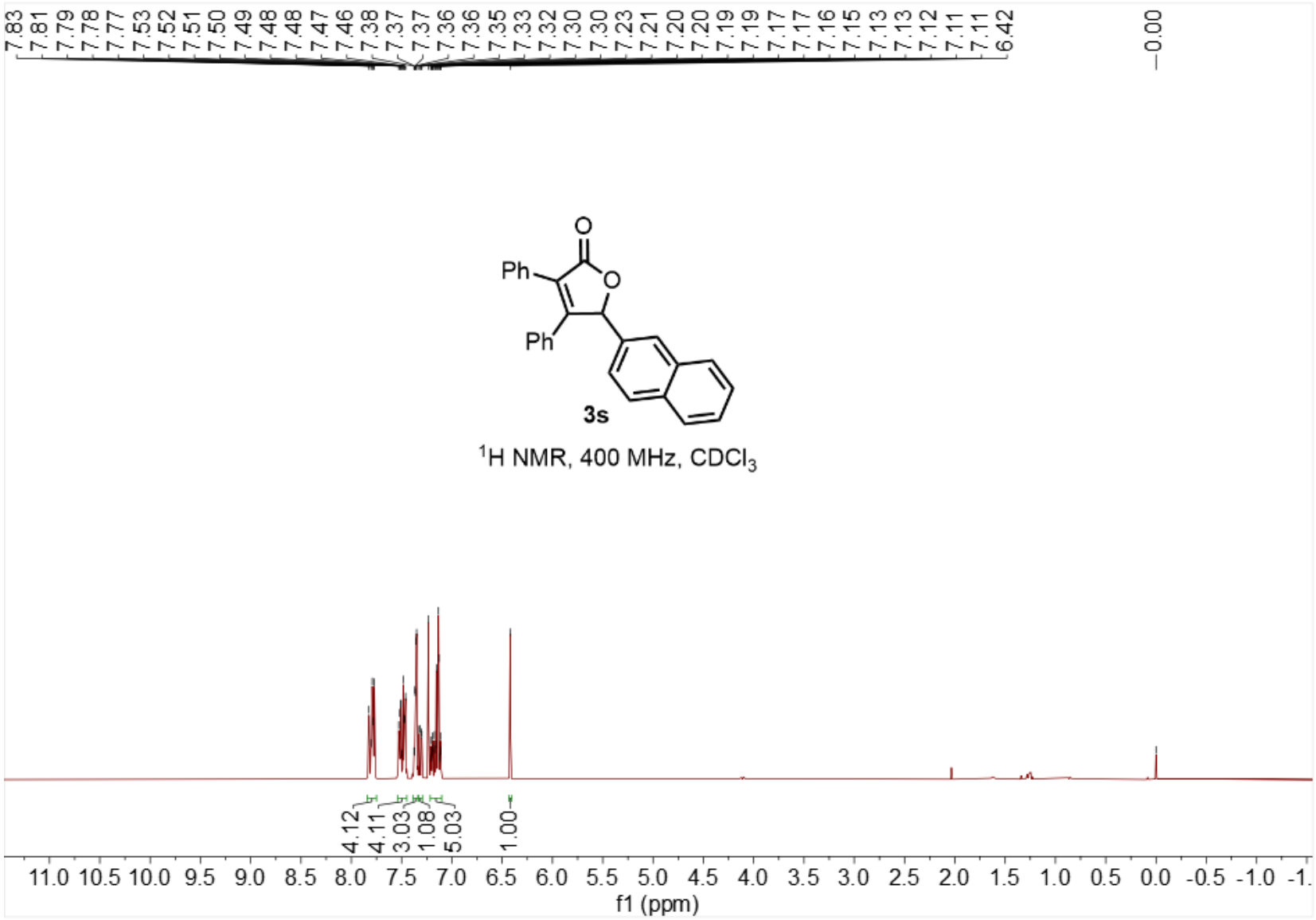


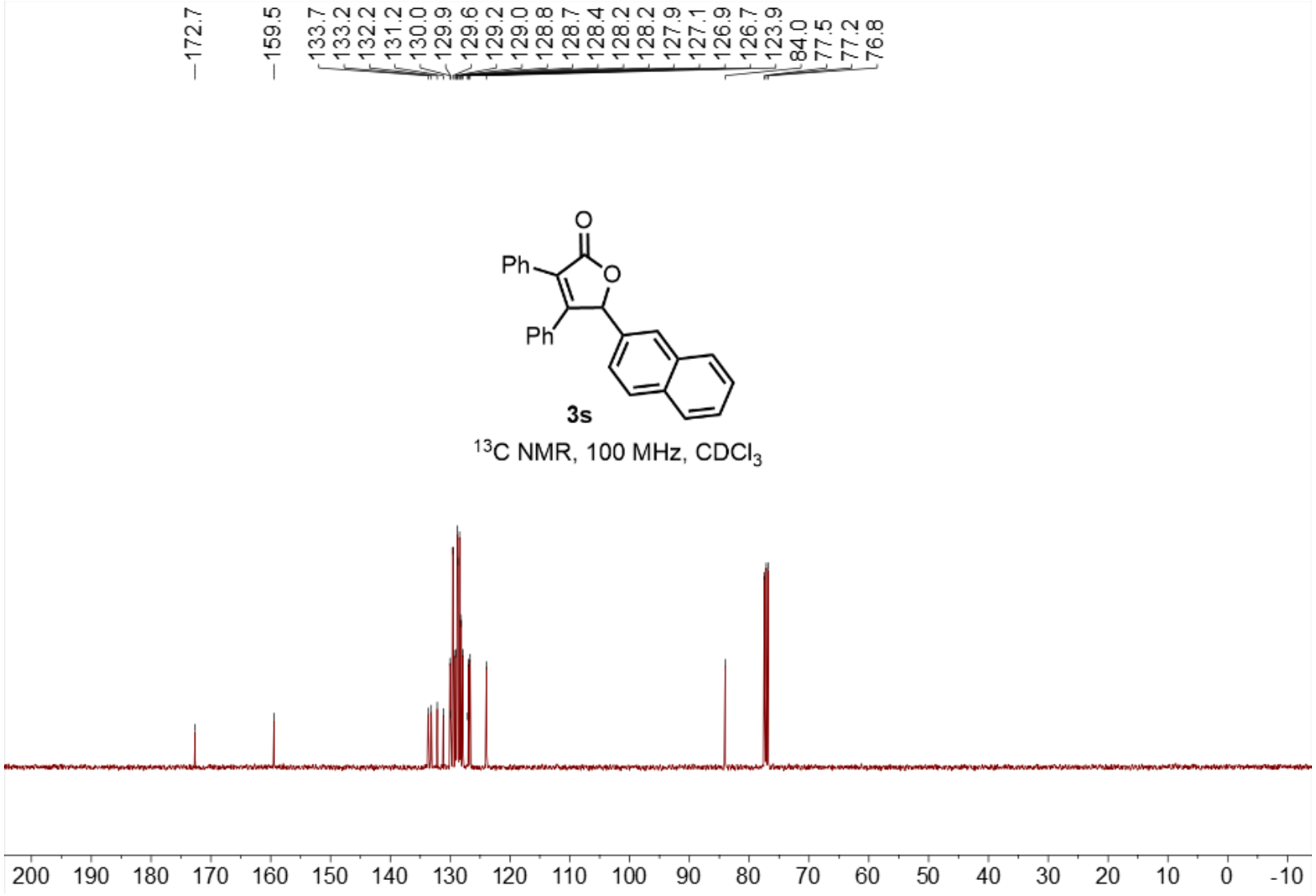

172.4
166.5
159.3
139.8
131.2
130.9
130.3
130.2
129.6
129.5
129.2
129.0
128.7
128.3
127.6
127.0
83.0
77.5
77.2
76.8
52.4
O
Ph
O
Ph
3p
CO2Me
13C NMR, 100 MHz, CDCl3
200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0 -10
f1 (ppm)


7.58 7.56 7.49 7.48 7.48 7.48 7.47 7.47 7.46 7.46 7.46 7.45 7.41 7.39 7.37 7.36 7.36 7.35 7.35 7.34 7.34 7.32 7.32 7.31 7.30 7.30 7.29 7.28 7.28 7.28 7.26 7.25 7.24 7.24 7.22 7.22 7.21 7.13 7.13 7.12 7.12 7.11 7.11 7.10 6.34 0.00
O
Ph
O
Ph
CF3
3q
1H NMR, 400 MHz, CDCl3
2.08 2.00 2.08 3.10 1.14 2.28 2.04 1.00
11.5 11.0 10.5 10.0 9.5 9.0 8.5 8.0 7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 0.5 0.0 -0.5 -1.0
f1 (ppm)


8.17 8.17 8.17 8.17 8.15 8.15 8.14 7.63 7.62 7.61 7.53 7.52 7.51 7.51 7.50 7.49 7.49 7.48 7.48 7.48 7.48 7.47 7.38 7.38 7.37 7.37 7.36 7.36 7.36 7.32 7.31 7.29 7.29 7.28 7.27 7.26 7.26 7.25 7.25 7.25 7.24 7.23 7.23 7.16 7.15 7.15 7.14 7.14 7.13 6.40 -0.00
O
Ph
O
Ph
NO2
3r
1H NMR, 400 MHz, CDCl3
2.00 1.02 3.12 3.14 1.10 2.36 2.20 1.00
11.0 10.5 10.0 9.5 9.0 8.5 8.0 7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 0.5 0.0 -0.5 -1.0
f1 (ppm)

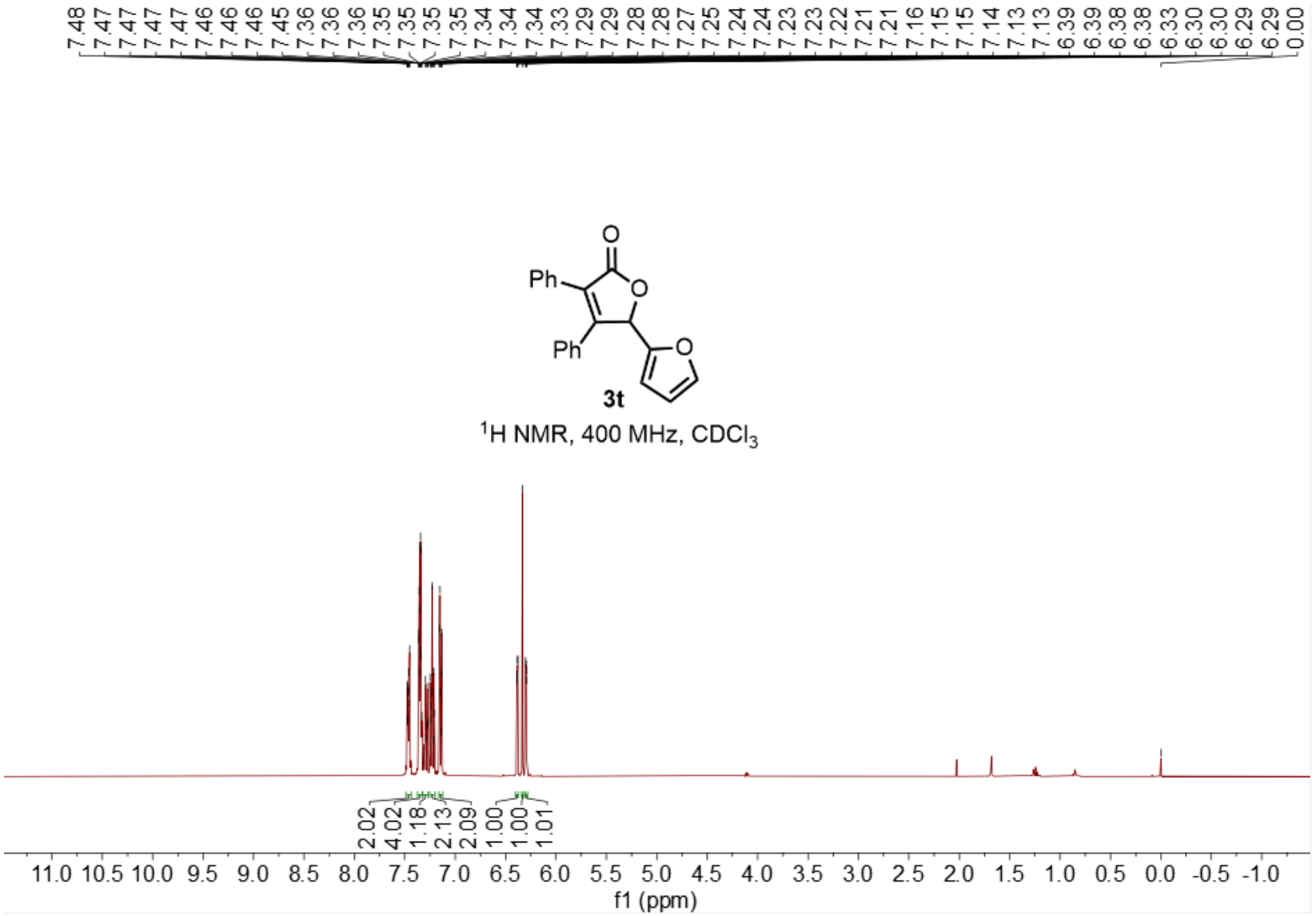

172.0
158.7
148.5
137.3
133.4
130.5
130.5
130.2
129.5
129.3
129.3
129.2
128.8
128.3
127.4
124.4
122.8
82.2
77.5
77.2
76.8
O
Ph
O
Ph
NO2
3r
13C NMR, 100 MHz, CDCl3
200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0
f1 (ppm)

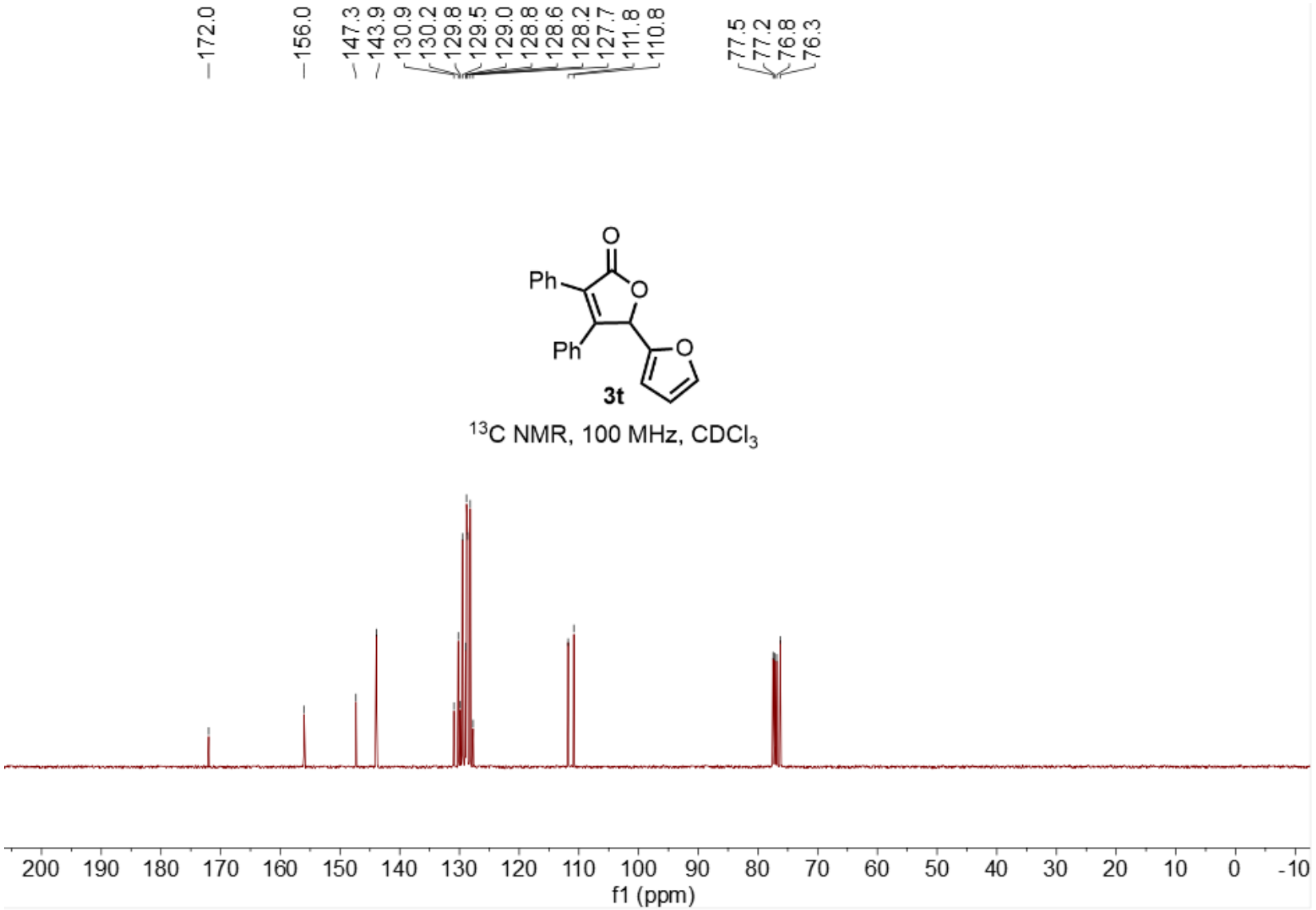

7.83
7.81
7.79
7.78
7.77
7.53
7.52
7.51
7.50
7.49
7.48
7.48
7.47
7.46
7.38
7.37
7.37
7.36
7.36
7.35
7.33
7.32
7.30
7.30
7.23
7.21
7.20
7.20
7.19
7.19
7.17
7.17
7.16
7.15
7.13
7.13
7.12
7.11
7.11
6.42
0.00
O
Ph
O
Ph
3s
1H NMR, 400 MHz, CDCl3
4.12
4.11
3.03
1.08
5.03
1.00
11.0 10.5 10.0 9.5 9.0 8.5 8.0 7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 0.5 0.0 -0.5 -1.0 -1.
f1 (ppm)

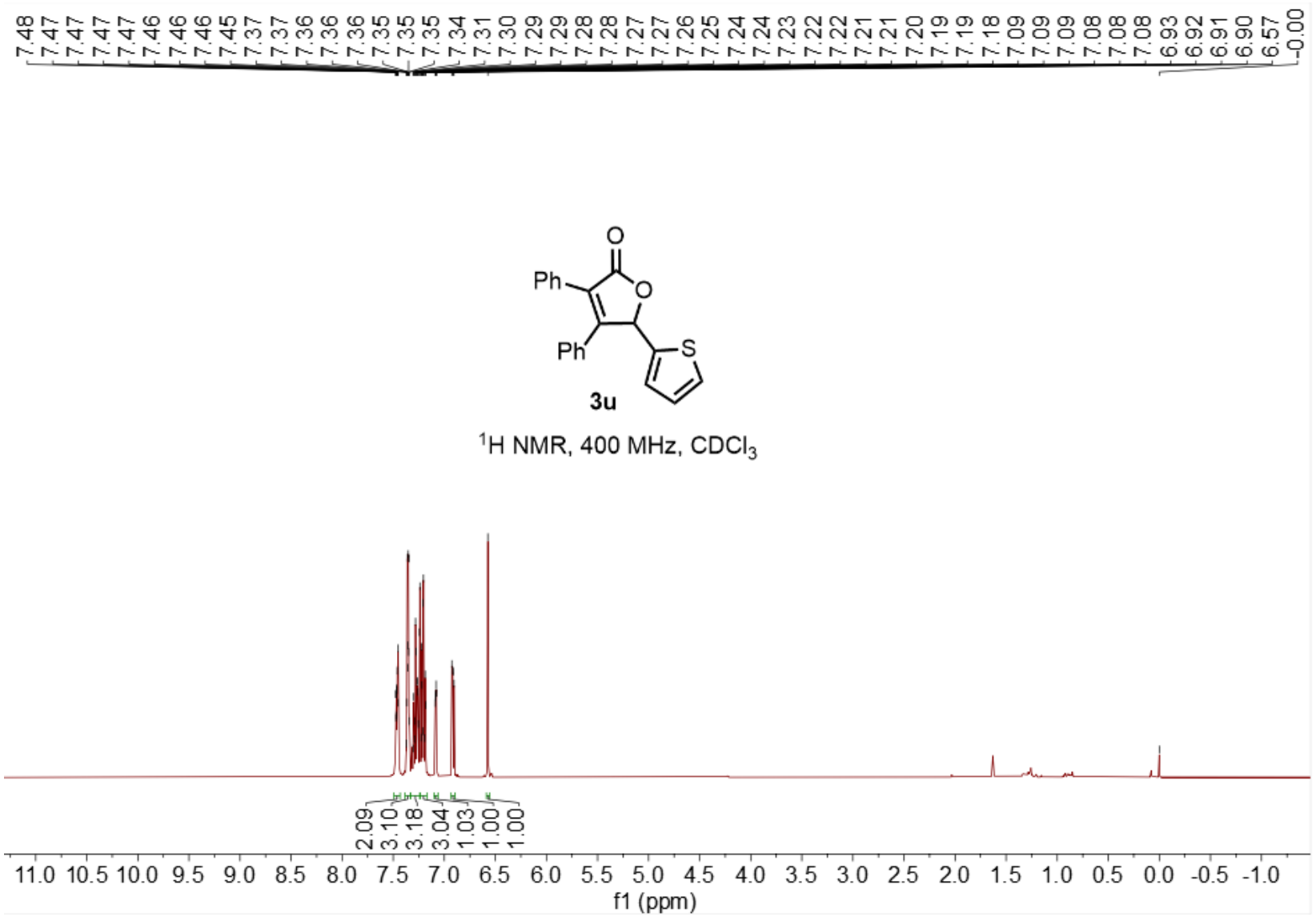


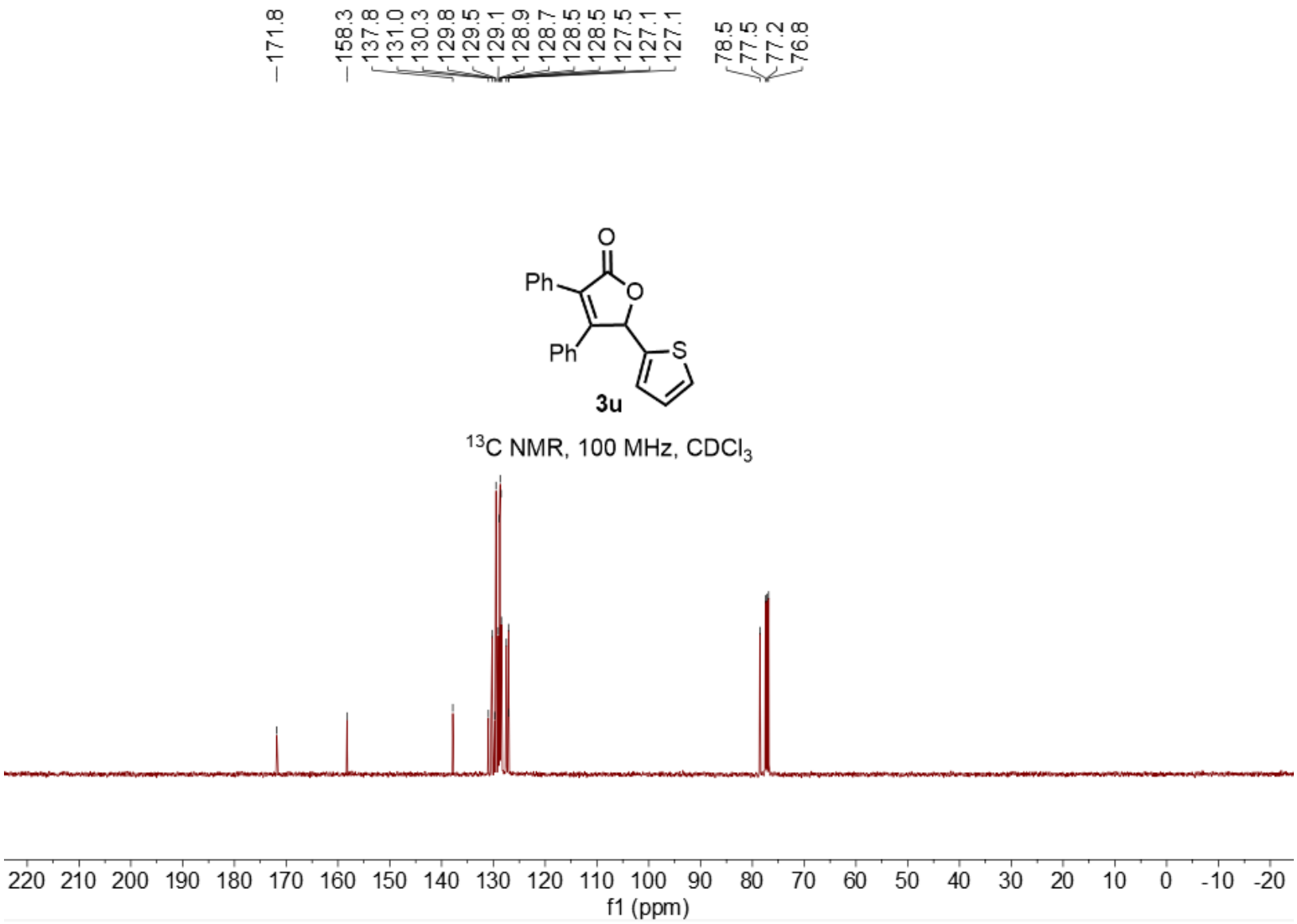
3s

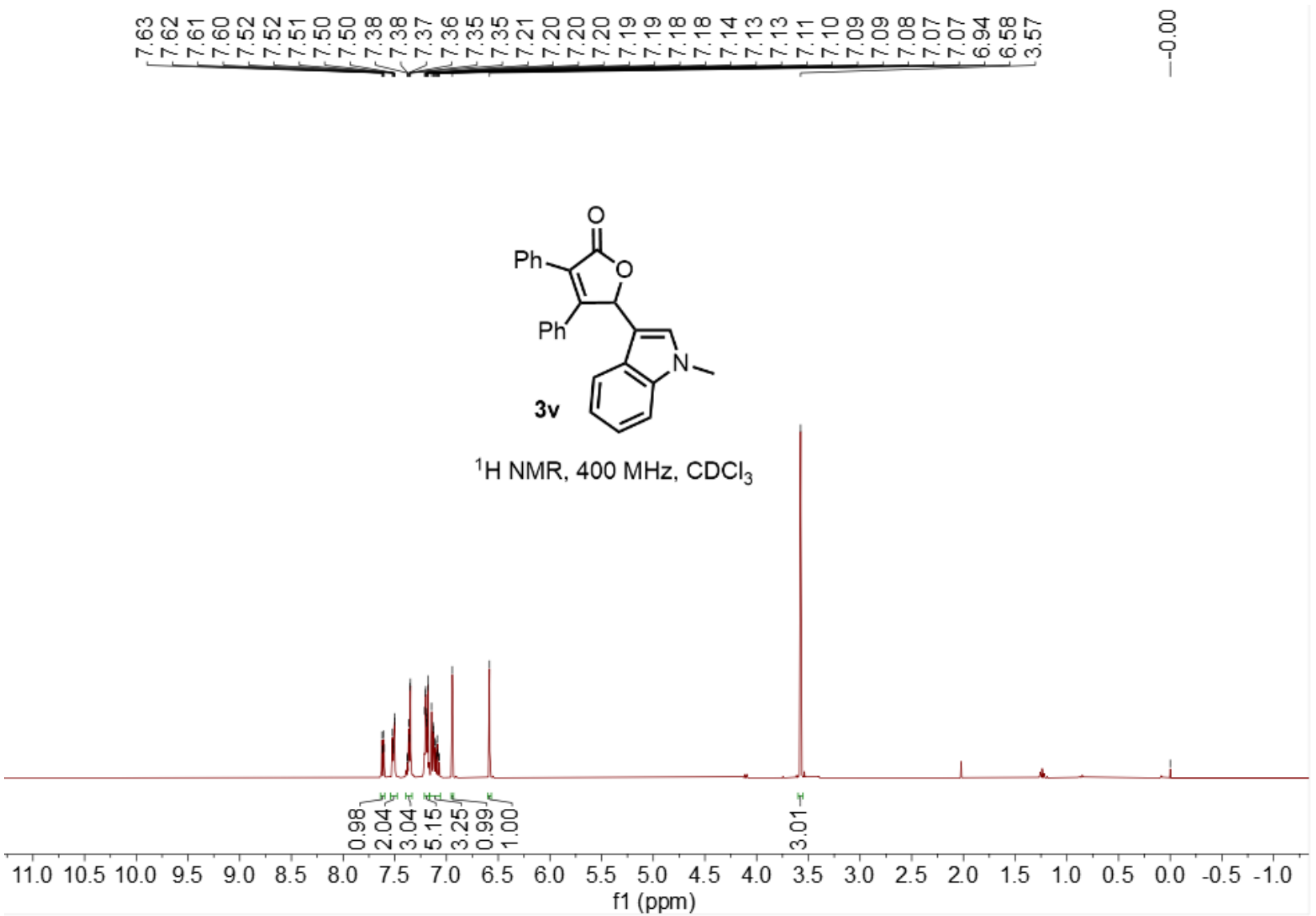
^{13}C NMR, 100 MHz, $CDCl_3$

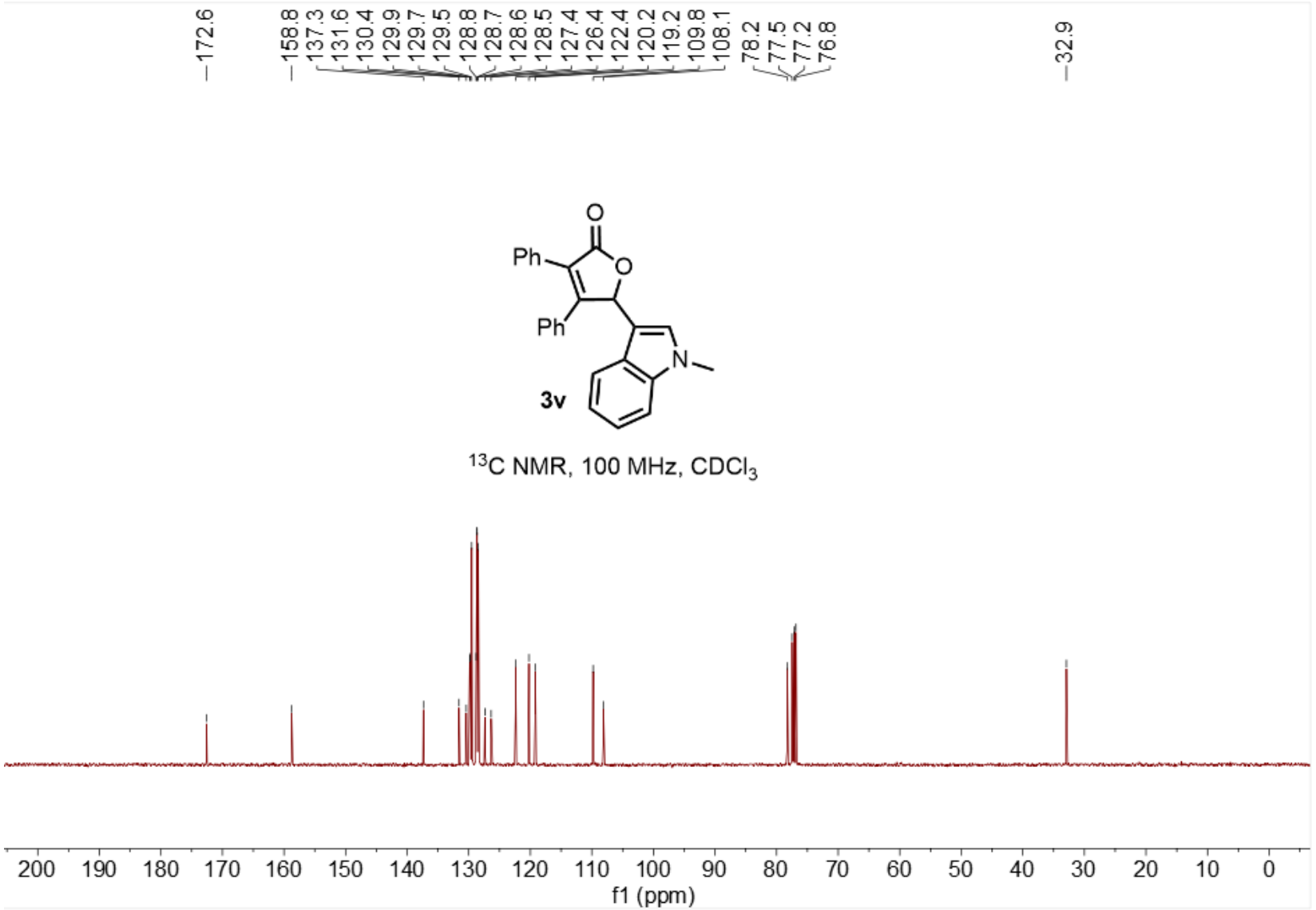

7.48 7.47 7.47 7.47 7.47 7.46 7.46 7.46 7.45 7.36 7.36 7.36 7.35 7.35 7.35 7.34 7.34 7.34 7.33 7.29 7.29 7.28 7.28 7.27 7.25 7.24 7.24 7.23 7.23 7.22 7.21 7.21 7.16 7.15 7.15 7.14 7.13 7.13 6.39 6.39 6.38 6.38 6.33 6.30 6.30 6.29 6.29 0.00
O
Ph
O
Ph
O
3t
1H NMR, 400 MHz, CDCl3
2.02 4.02 1.18 2.13 2.09 1.00 1.00 1.01
11.0 10.5 10.0 9.5 9.0 8.5 8.0 7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 0.5 0.0 -0.5 -1.0
f1 (ppm)

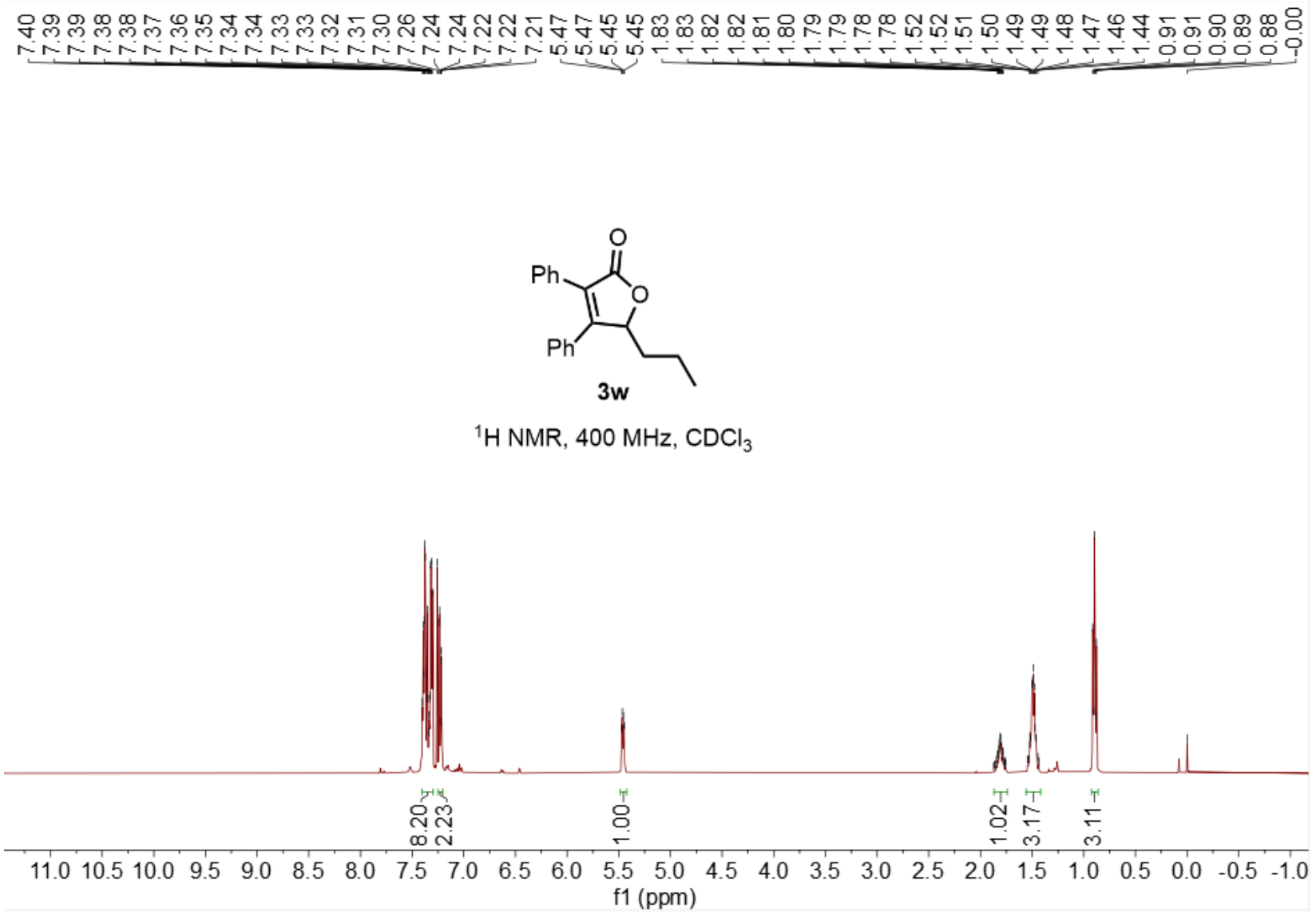

172.0
156.0
147.3
143.9
130.9
130.2
129.8
129.5
129.0
128.8
128.6
128.2
127.7
111.8
110.8
77.5
77.2
76.8
76.3
O
Ph
O
Ph
O
3t
13C NMR, 100 MHz, CDCl3
200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0 -10
f1 (ppm)

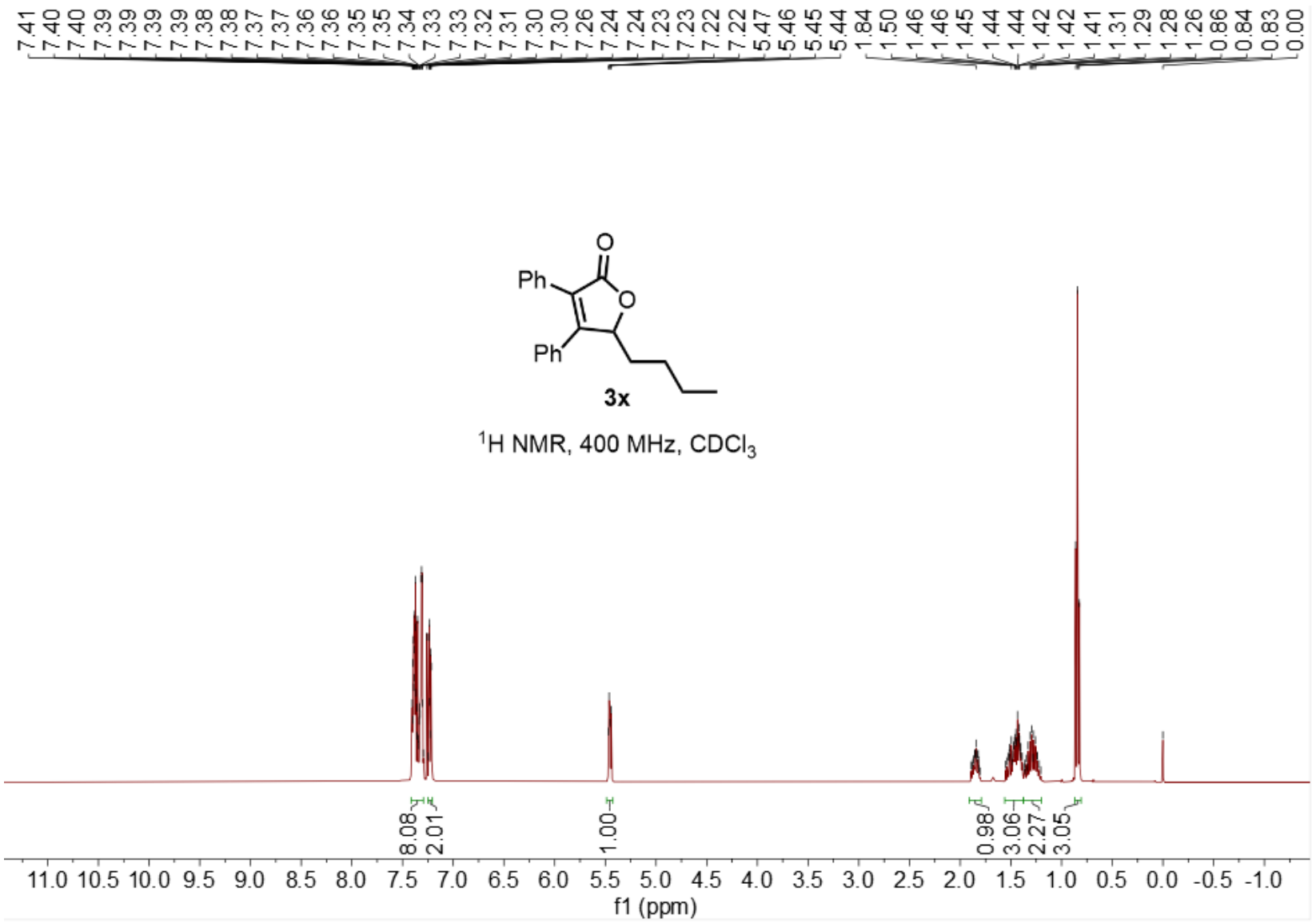

7.48 7.47 7.47 7.47 7.47 7.46 7.46 7.46 7.45 7.37 7.37 7.36 7.36 7.36 7.35 7.35 7.35 7.34 7.31 7.30 7.29 7.29 7.28 7.28 7.27 7.27 7.26 7.25 7.24 7.24 7.23 7.22 7.22 7.21 7.21 7.20 7.19 7.19 7.18 7.09 7.09 7.09 7.08 7.08 7.08 6.93 6.92 6.91 6.90 6.57 -0.00
O
Ph
O
Ph
S
3u
^{1}H NMR, 400 MHz, $CDCl_3$
2.09 3.10 3.18 3.04 1.03 1.00 1.00
11.0 10.5 10.0 9.5 9.0 8.5 8.0 7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 0.5 0.0 -0.5 -1.0
f1 (ppm)

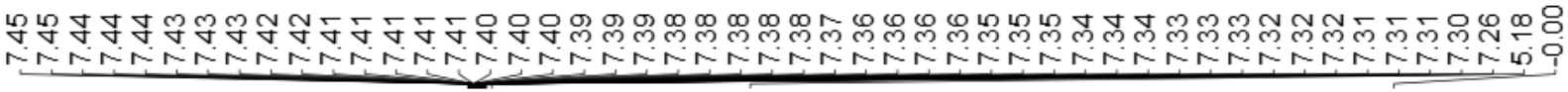

171.8
158.3
137.8
131.0
130.3
129.8
129.5
129.1
128.9
128.7
128.5
128.5
127.5
127.1
127.1
78.5
77.5
77.2
76.8
O
Ph
O
Ph
S
3u
13C NMR, 100 MHz, CDCl3
220 210 200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0 -10 -20
f1 (ppm)

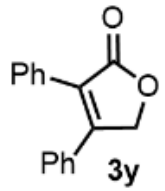

7.63
7.62
7.61
7.60
7.52
7.52
7.51
7.50
7.50
7.38
7.38
7.37
7.36
7.35
7.35
7.21
7.20
7.20
7.20
7.19
7.19
7.18
7.18
7.14
7.13
7.13
7.11
7.10
7.09
7.09
7.08
7.07
7.07
6.94
6.58
3.57
-0.00
O
Ph
O
Ph
N
3v
1H NMR, 400 MHz, CDCl3
0.98
2.04
3.04
5.15
3.25
0.99
1.00
3.01
11.0 10.5 10.0 9.5 9.0 8.5 8.0 7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 0.5 0.0 -0.5 -1.0
f1 (ppm)


172.6
158.8
137.3
131.6
130.4
129.9
129.7
129.5
128.8
128.7
128.6
128.5
127.4
126.4
122.4
120.2
119.2
109.8
108.1
78.2
77.5
77.2
76.8
32.9
O
Ph
O
Ph
N
3v
13C NMR, 100 MHz, CDCl3
200
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
f1 (ppm)


7.40 7.39 7.39 7.38 7.38 7.37 7.36 7.35 7.34 7.34 7.33 7.33 7.32 7.31 7.30 7.26 7.24 7.24 7.22 7.22 7.21
5.47 5.47 5.45 5.45
1.83 1.83 1.82 1.82 1.81 1.80 1.79 1.79 1.78 1.78 1.52 1.52 1.51 1.50 1.49 1.49 1.48 1.47 1.46 1.44
0.91 0.91 0.90 0.89 0.88 -0.00
O
Ph
O
Ph
3w
^{1}H NMR, 400 MHz, $CDCl_3$
8.20
2.23
1.00
1.02
3.17
3.11
11.0 10.5 10.0 9.5 9.0 8.5 8.0 7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 0.5 0.0 -0.5 -1.0
f1 (ppm)

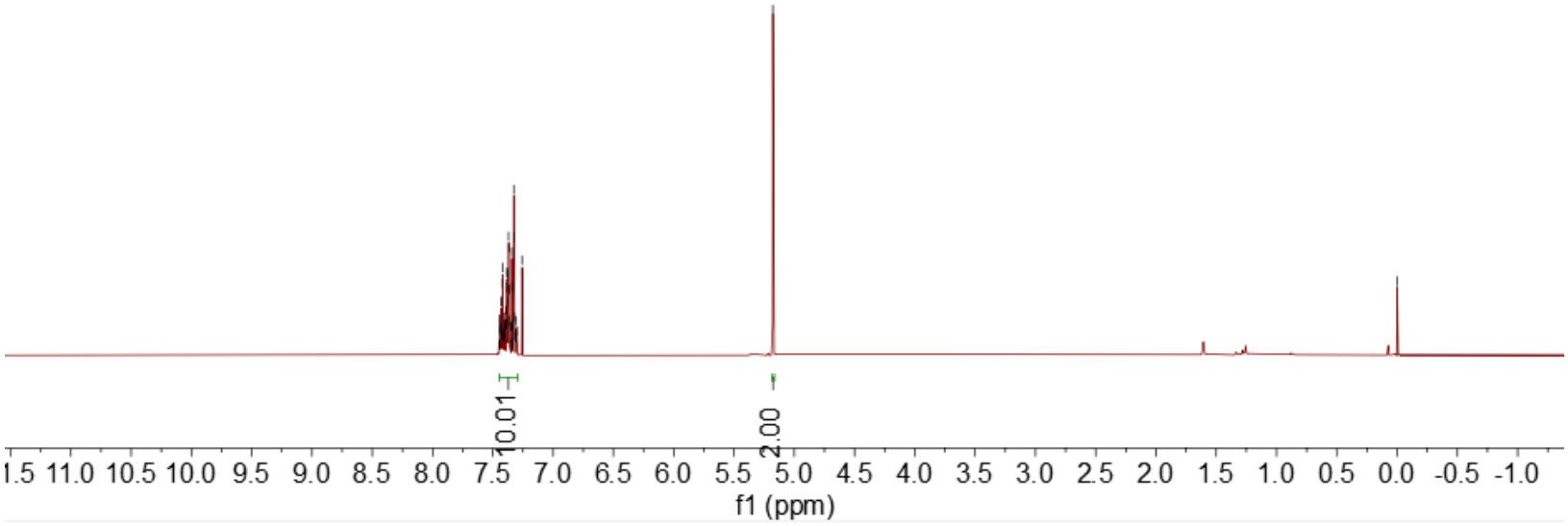

7.41 7.40 7.40 7.39 7.39 7.39 7.39 7.38 7.38 7.37 7.37 7.36 7.36 7.35 7.35 7.34 7.33 7.33 7.32 7.31 7.30 7.30 7.26 7.24 7.24 7.23 7.23 7.22 7.22 5.47 5.46 5.45 5.44 1.84 1.50 1.46 1.46 1.45 1.44 1.44 1.42 1.42 1.41 1.31 1.29 1.28 1.26 0.86 0.84 0.83 0.00
O
Ph
O
Ph
3x
1H NMR, 400 MHz, CDCl3
8.08
2.01
1.00
0.98
3.06
2.27
3.05
11.0 10.5 10.0 9.5 9.0 8.5 8.0 7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 0.5 0.0 -0.5 -1.0
f1 (ppm)

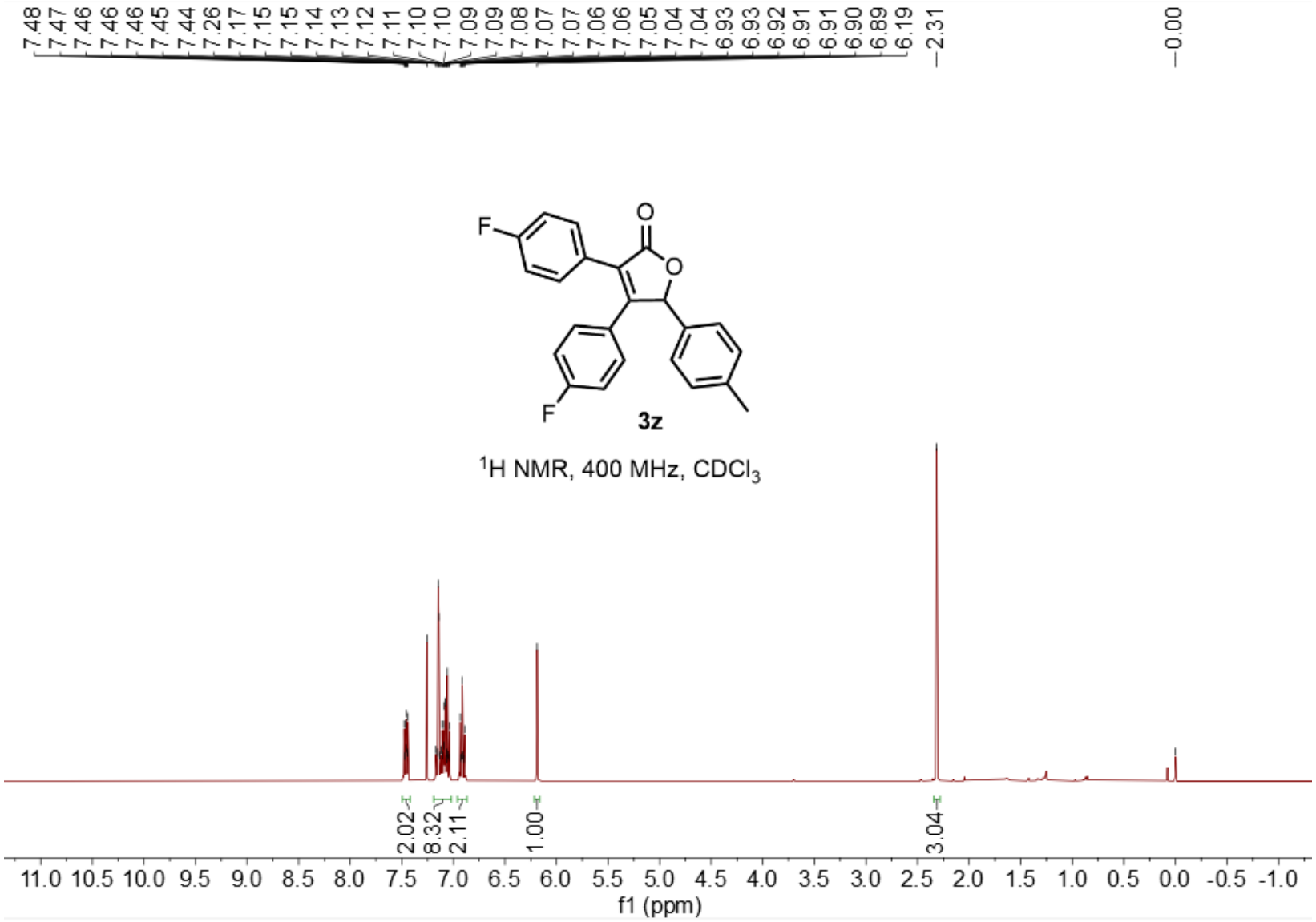


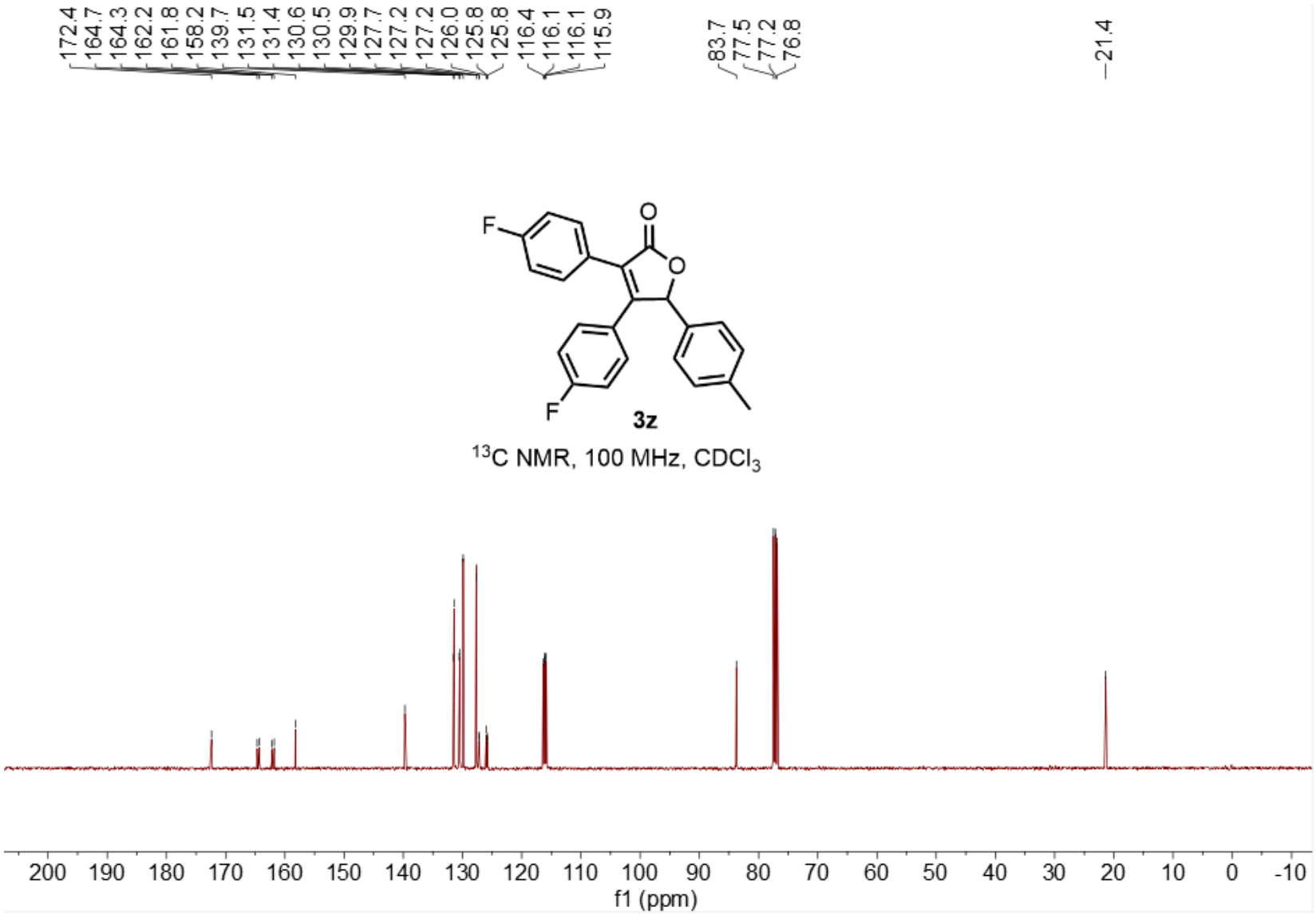


3y

^{1}H NMR, 400 MHz, $CDCl_3$




7.48
7.47
7.46
7.46
7.46
7.45
7.44
7.26
7.17
7.15
7.15
7.14
7.13
7.12
7.11
7.10
7.10
7.09
7.09
7.08
7.07
7.07
7.06
7.06
7.05
7.04
7.04
6.93
6.93
6.92
6.91
6.91
6.90
6.89
6.19
2.31
0.00
F
O
O
F
3z
1H NMR, 400 MHz, CDCl3
2.02
8.32
2.11
1.00
3.04
11.0 10.5 10.0 9.5 9.0 8.5 8.0 7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 0.5 0.0 -0.5 -1.0
f1 (ppm)


172.4
164.7
164.3
162.2
161.8
158.2
139.7
131.5
131.4
130.6
130.5
129.9
127.7
127.2
127.2
126.0
125.8
125.8
116.4
116.1
116.1
115.9
83.7
77.5
77.2
76.8
21.4
F
O
O
F
3z
13C NMR, 100 MHz, CDCl3
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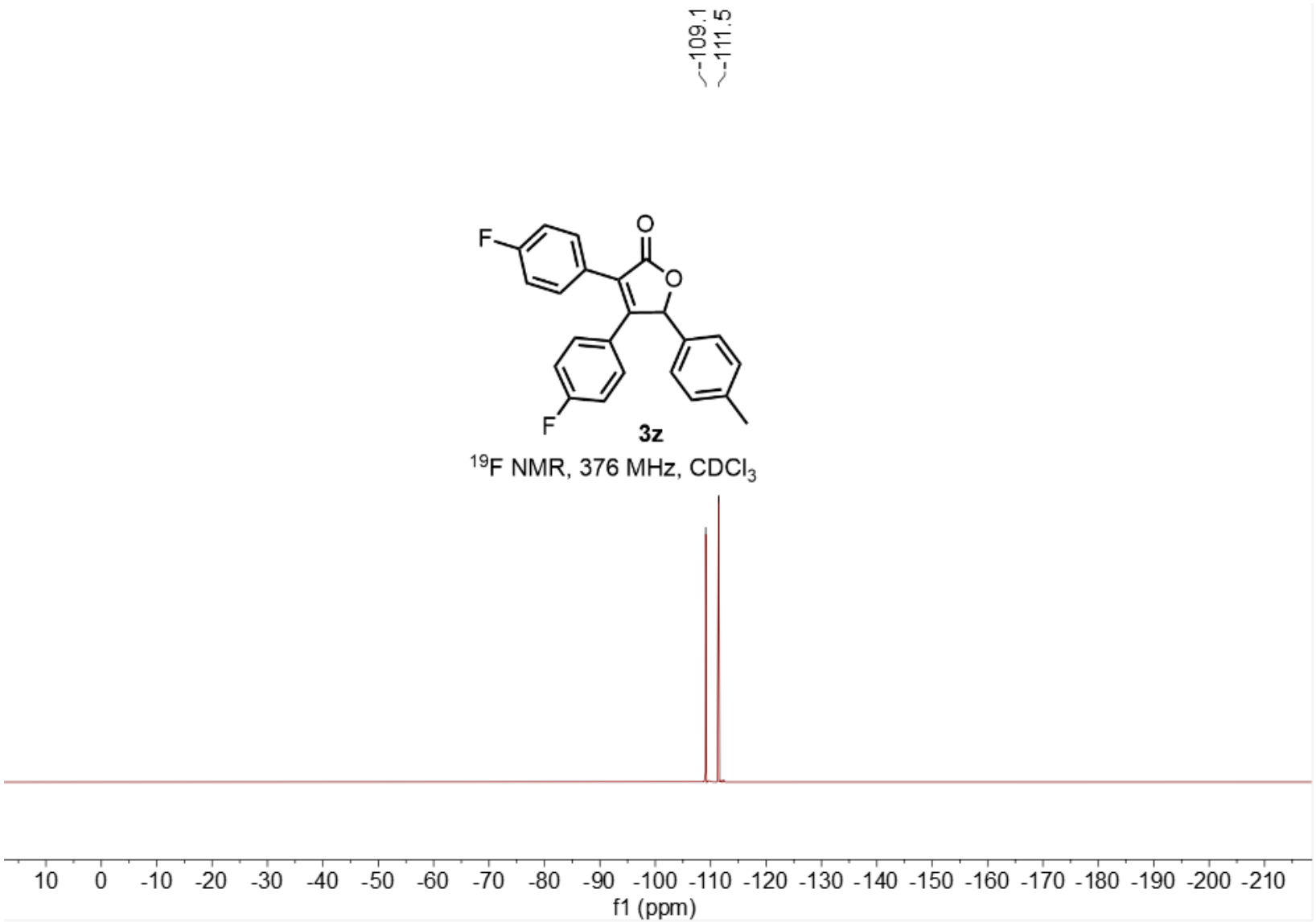
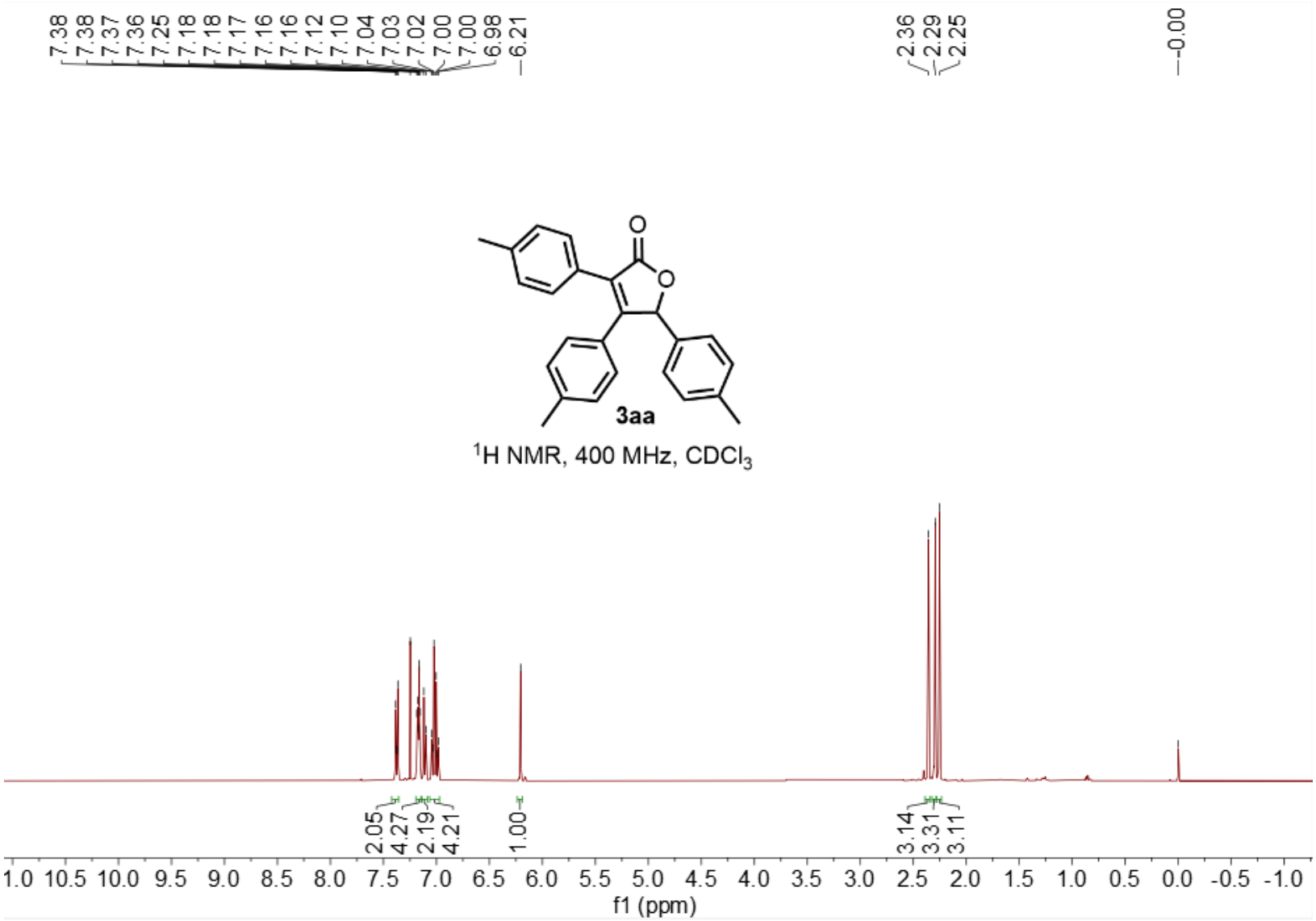
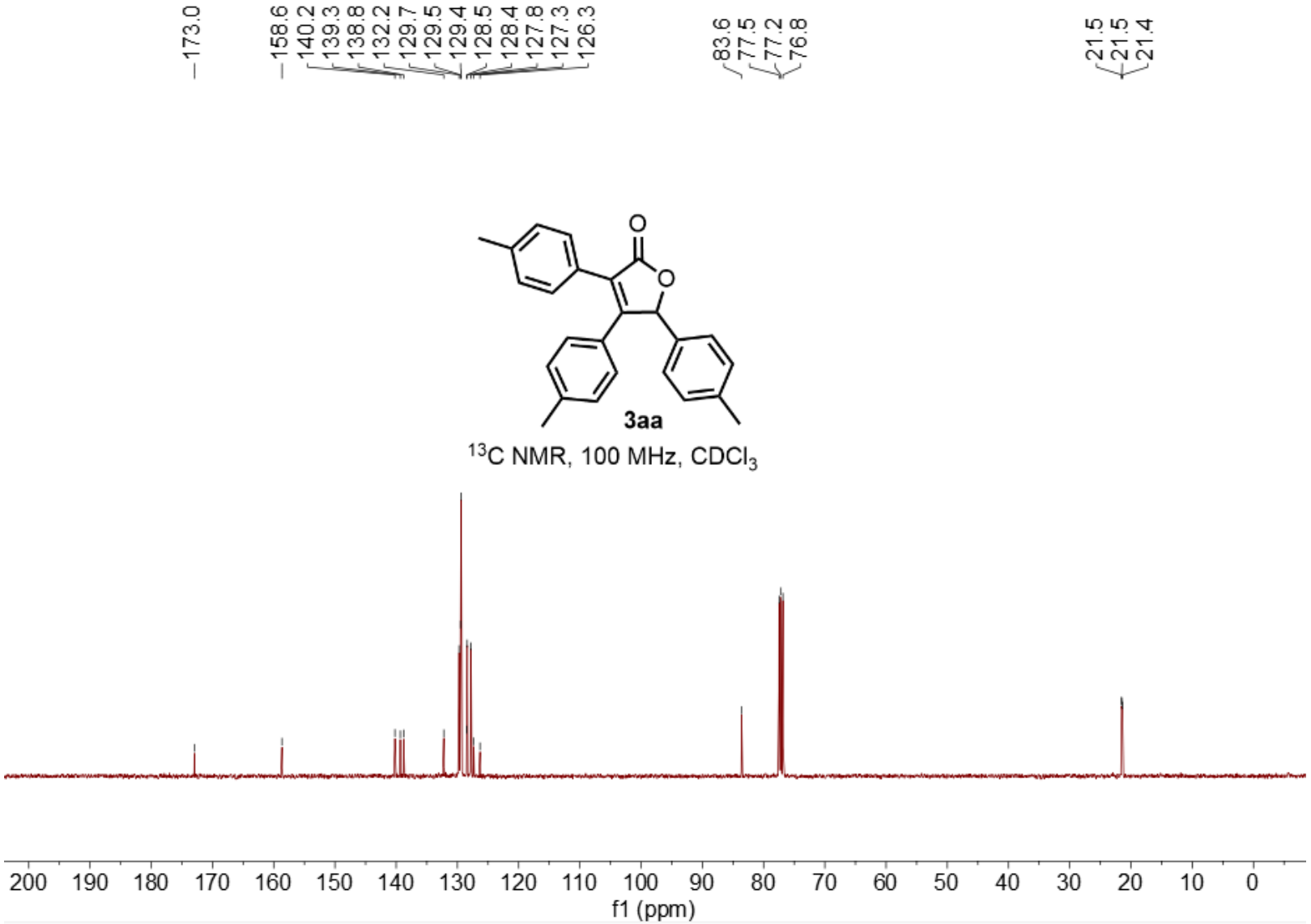
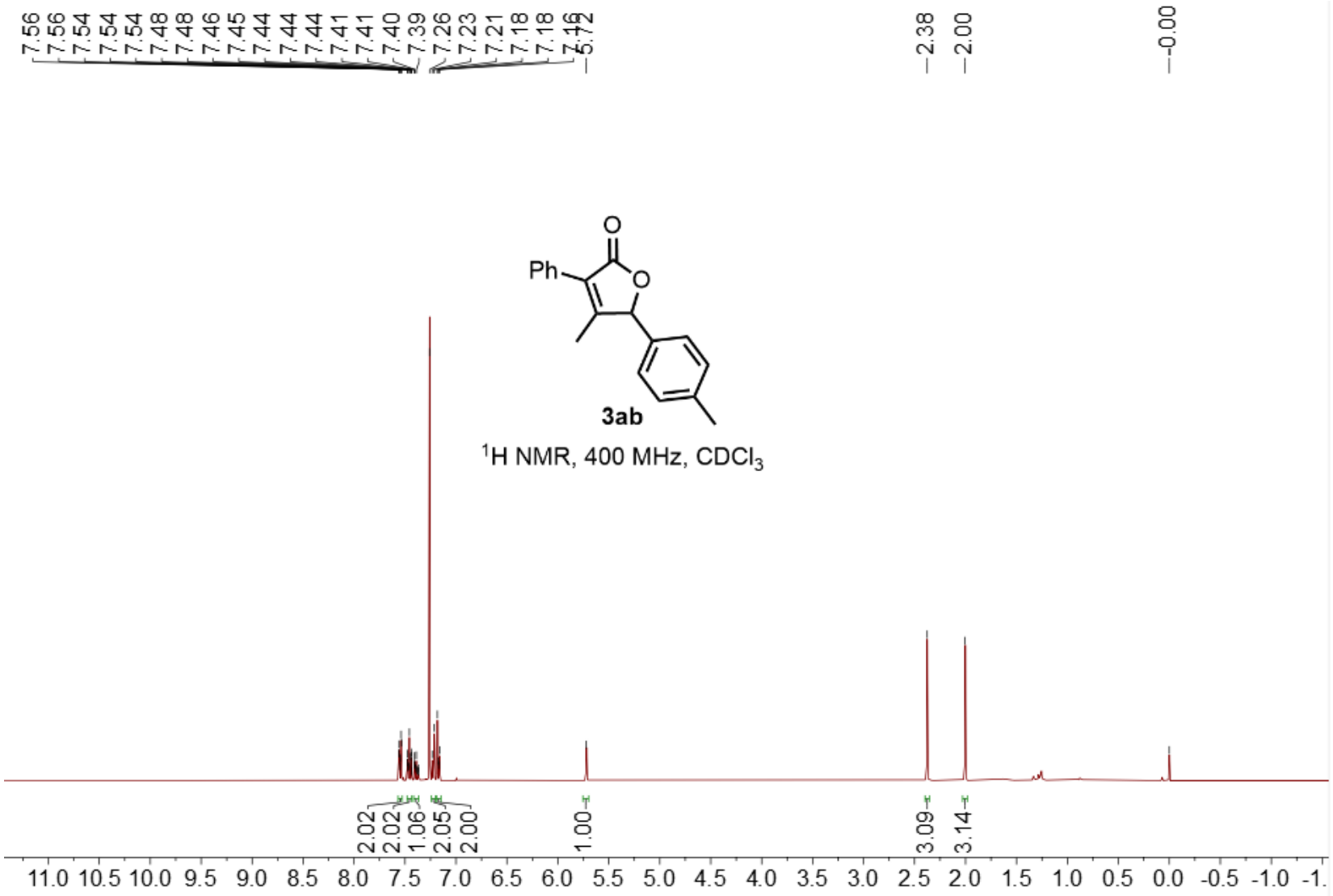
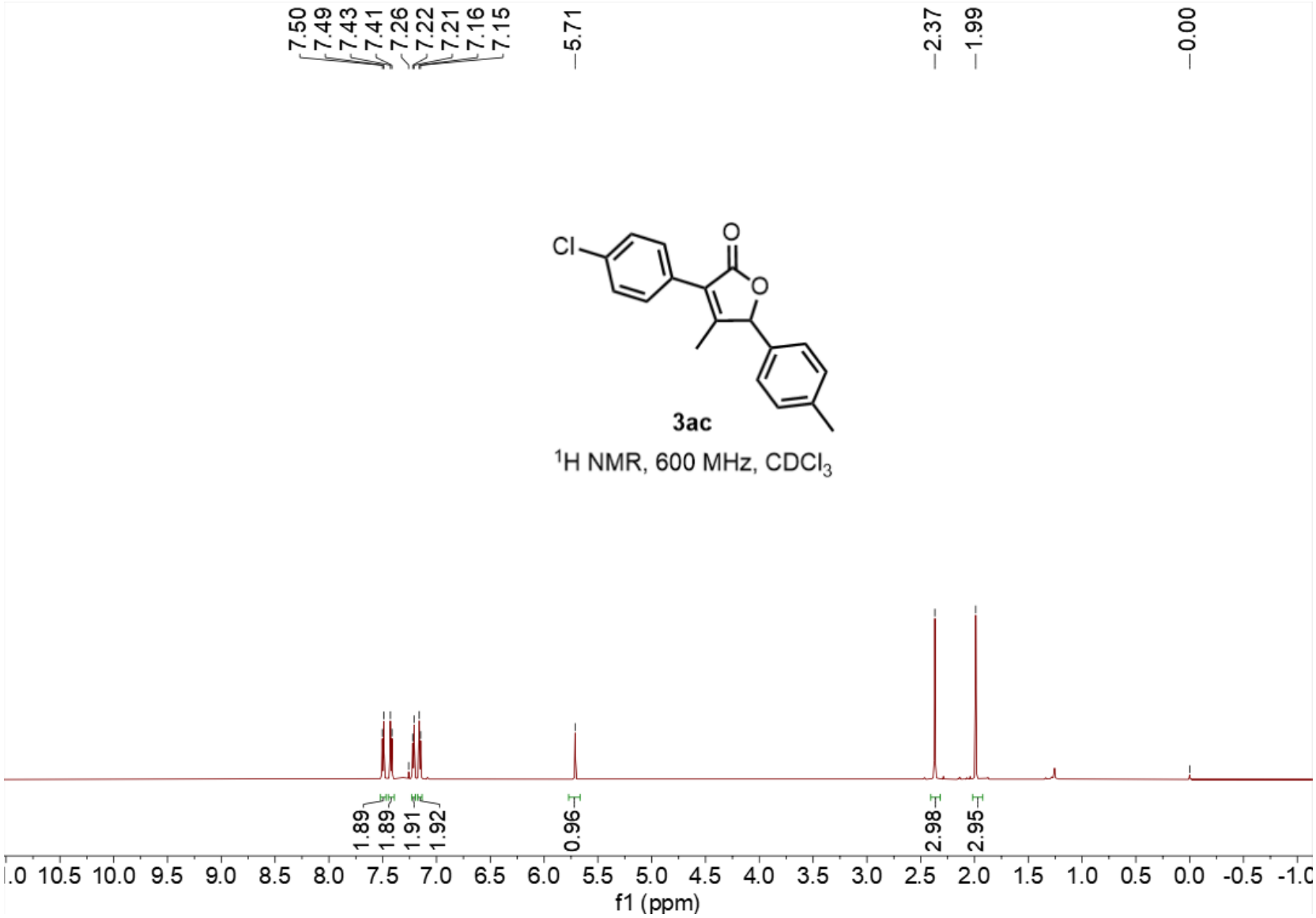
f1 (ppm)

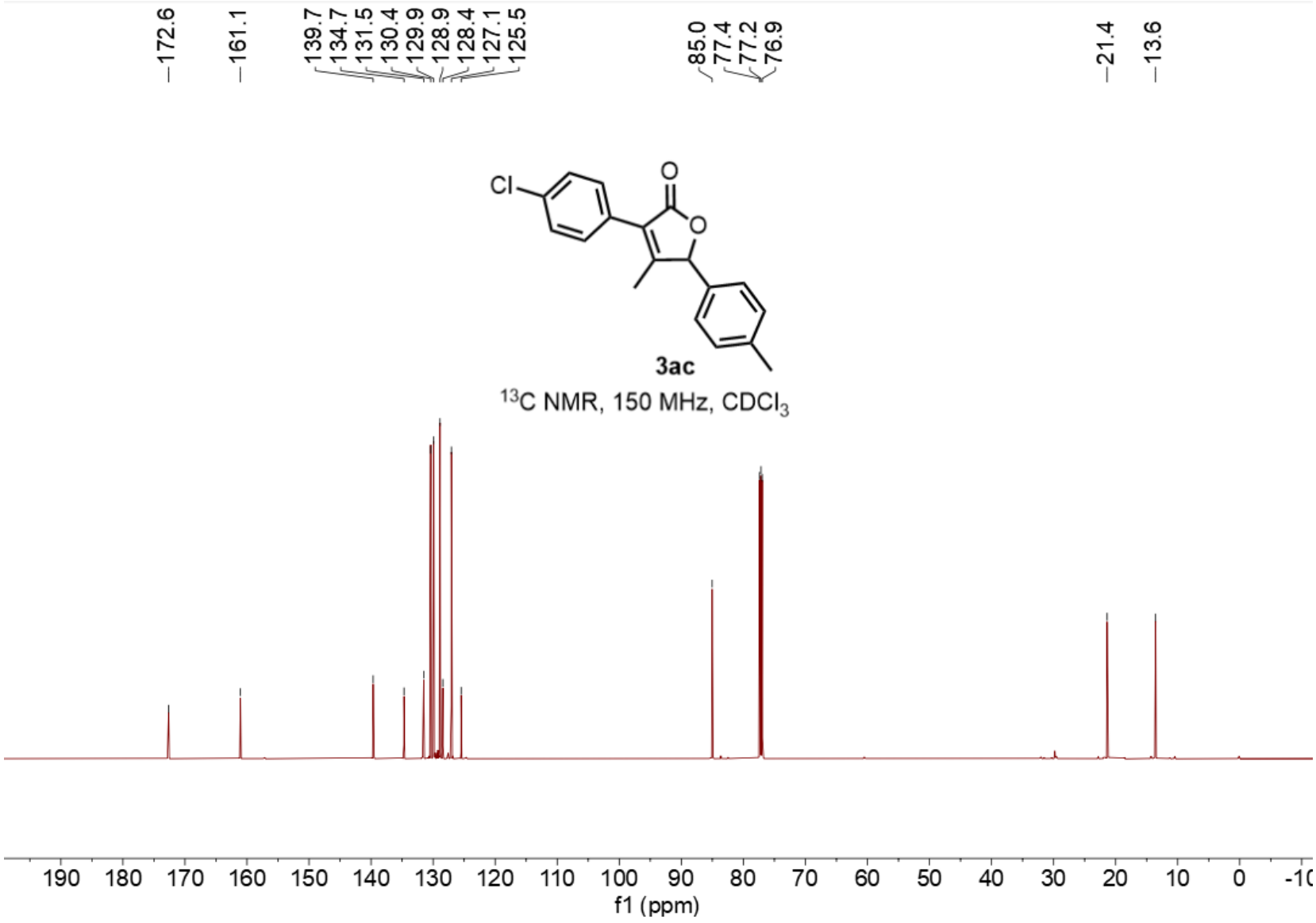

-109.1
-111.5
F
O
O
F
3z
^{19}F NMR, 376 MHz, $CDCl_3$
10 0 -10 -20 -30 -40 -50 -60 -70 -80 -90 -100 -110 -120 -130 -140 -150 -160 -170 -180 -190 -200 -210
f1 (ppm)

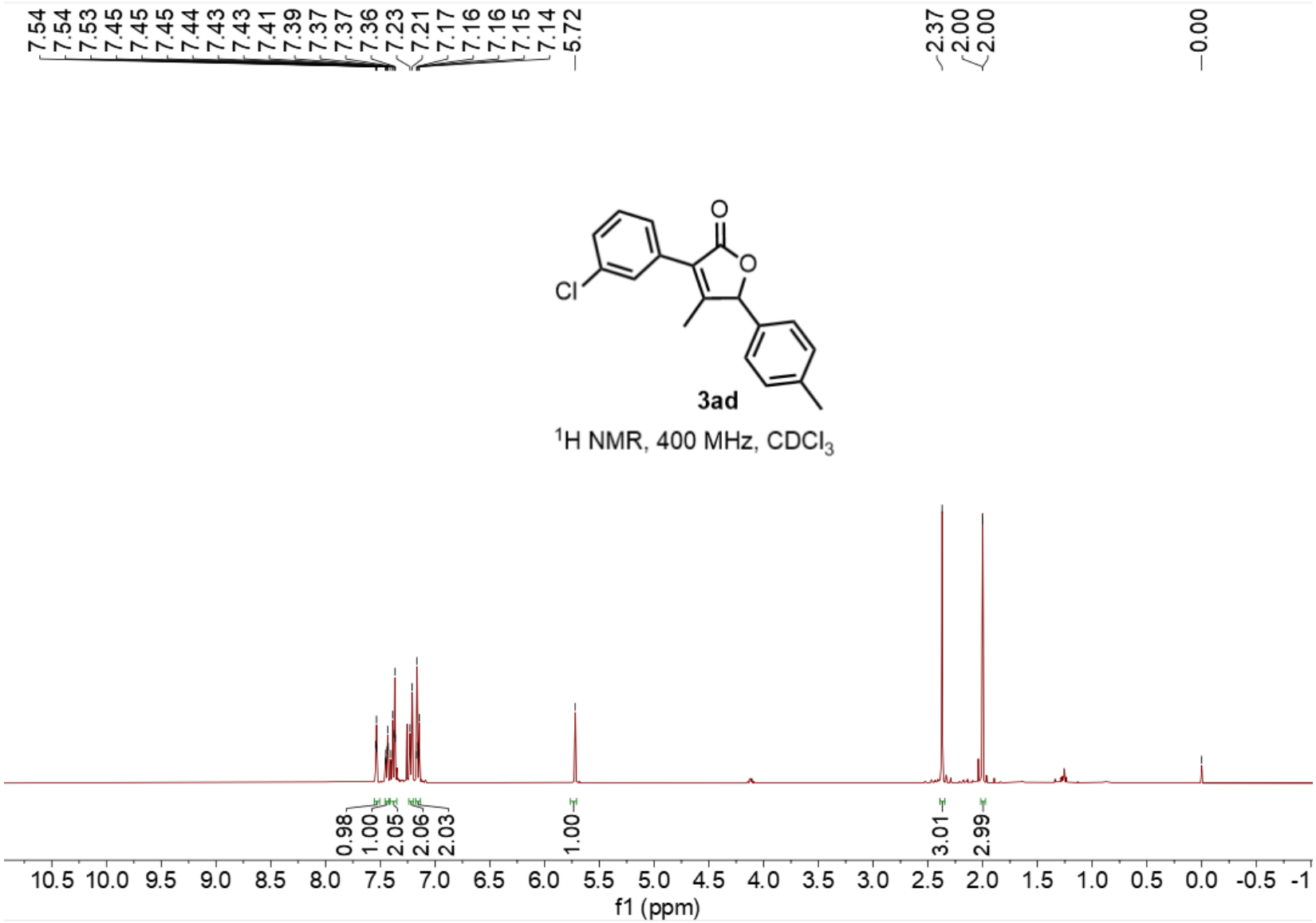

7.38
7.38
7.37
7.36
7.25
7.18
7.18
7.17
7.16
7.16
7.12
7.10
7.04
7.03
7.02
7.00
7.00
6.98
6.21
2.36
2.29
2.25
0.00
O
O
3aa
1H NMR, 400 MHz, CDCl3
2.05
4.27
2.19
4.21
1.00
3.14
3.31
3.11
1.0 10.5 10.0 9.5 9.0 8.5 8.0 7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 0.5 0.0 -0.5 -1.0
f1 (ppm)

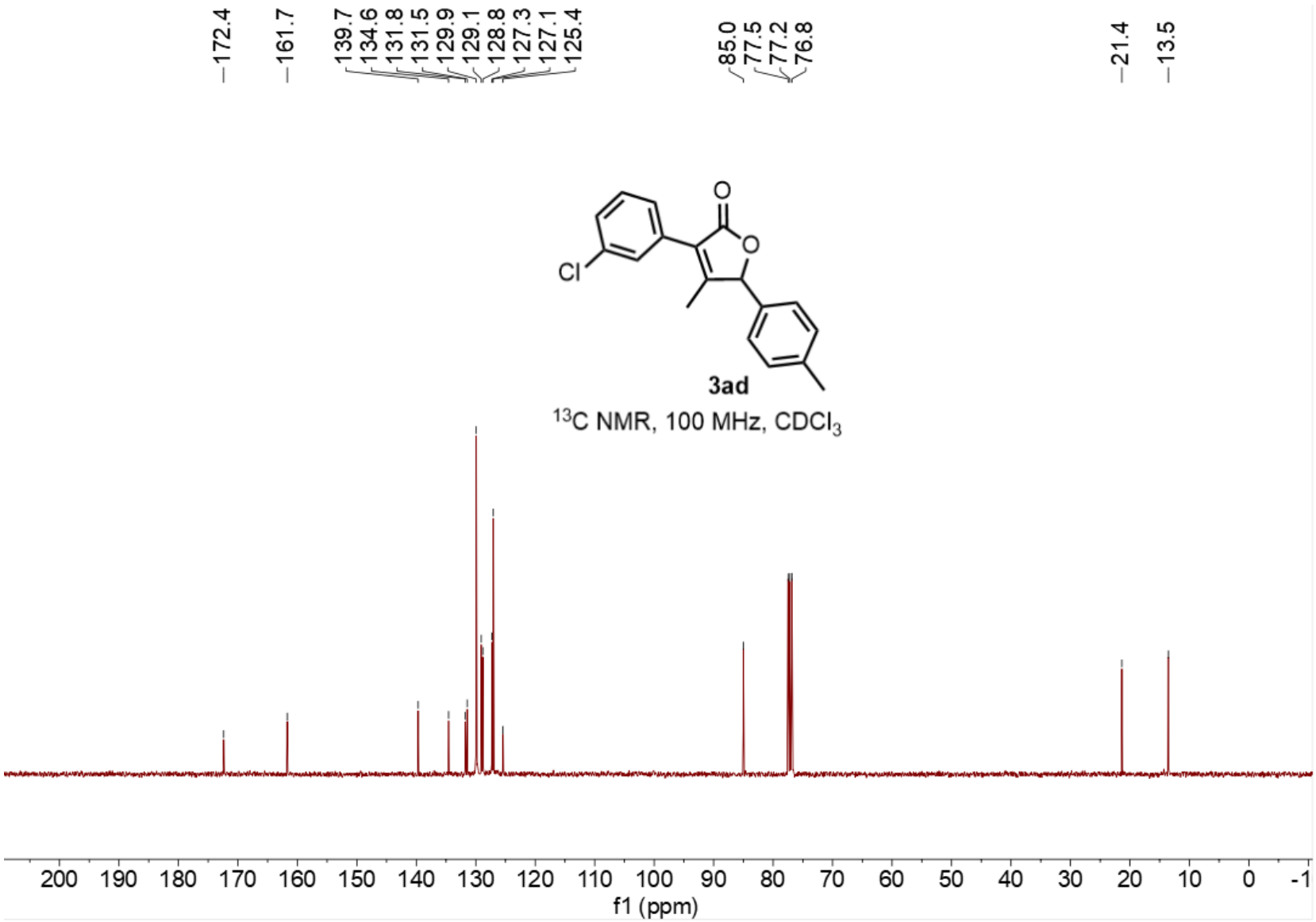

173.0
158.6
140.2
139.3
138.8
132.2
129.7
129.5
129.4
128.5
128.4
127.8
127.3
126.3
83.6
77.5
77.2
76.8
21.5
21.5
21.4
O
O
3aa
13C NMR, 100 MHz, CDCl3
200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0
f1 (ppm)

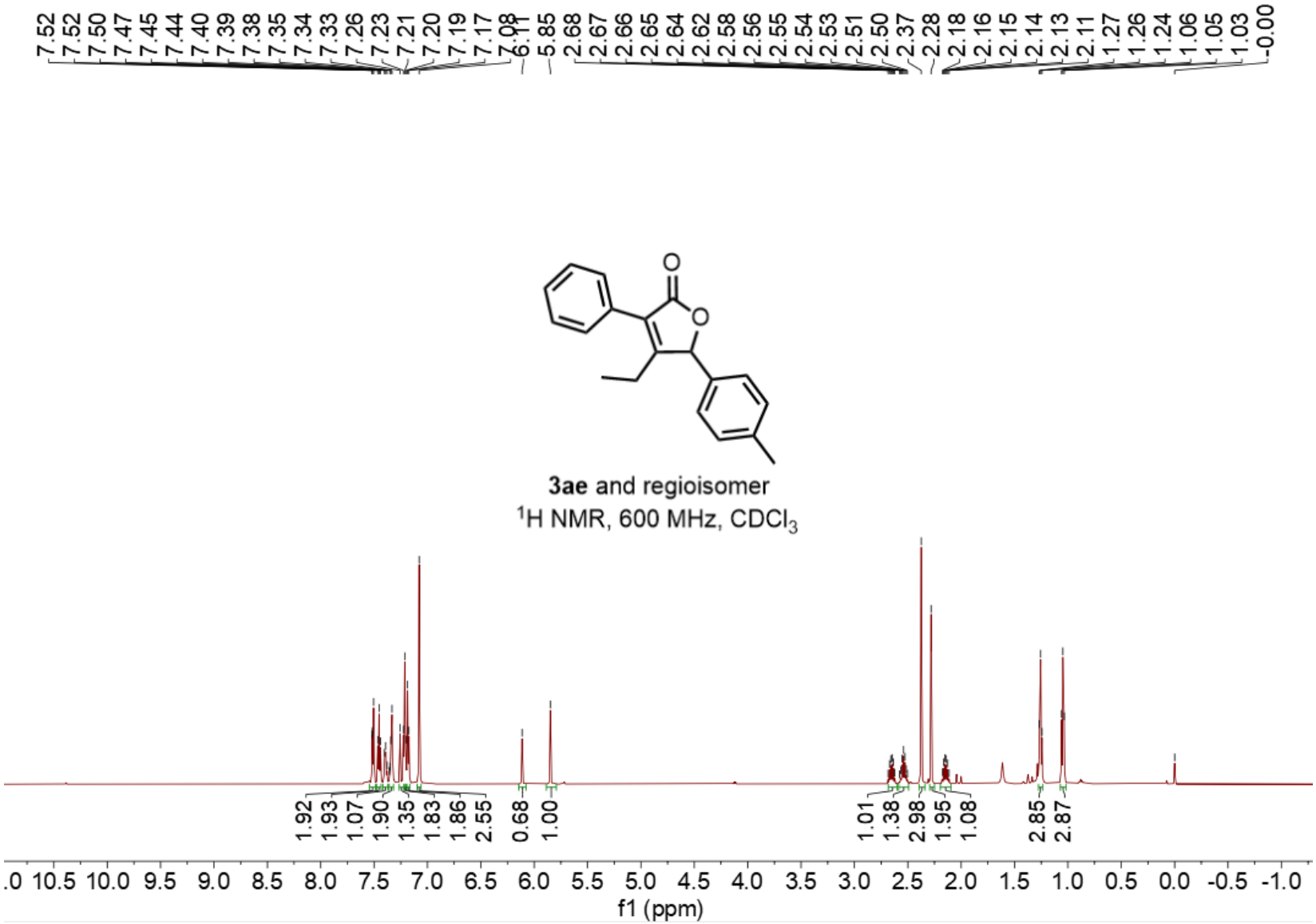

7.56
7.56
7.54
7.54
7.54
7.48
7.48
7.46
7.45
7.44
7.44
7.44
7.41
7.41
7.40
7.39
7.26
7.23
7.21
7.18
7.18
7.16
5.12
2.38
2.00
0.00
O
Ph
O
3ab
1H NMR, 400 MHz, CDCl3
2.02
2.02
1.06
2.05
2.00
1.00
3.09
3.14
11.0 10.5 10.0 9.5 9.0 8.5 8.0 7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 0.5 0.0 -0.5 -1.0 -1.

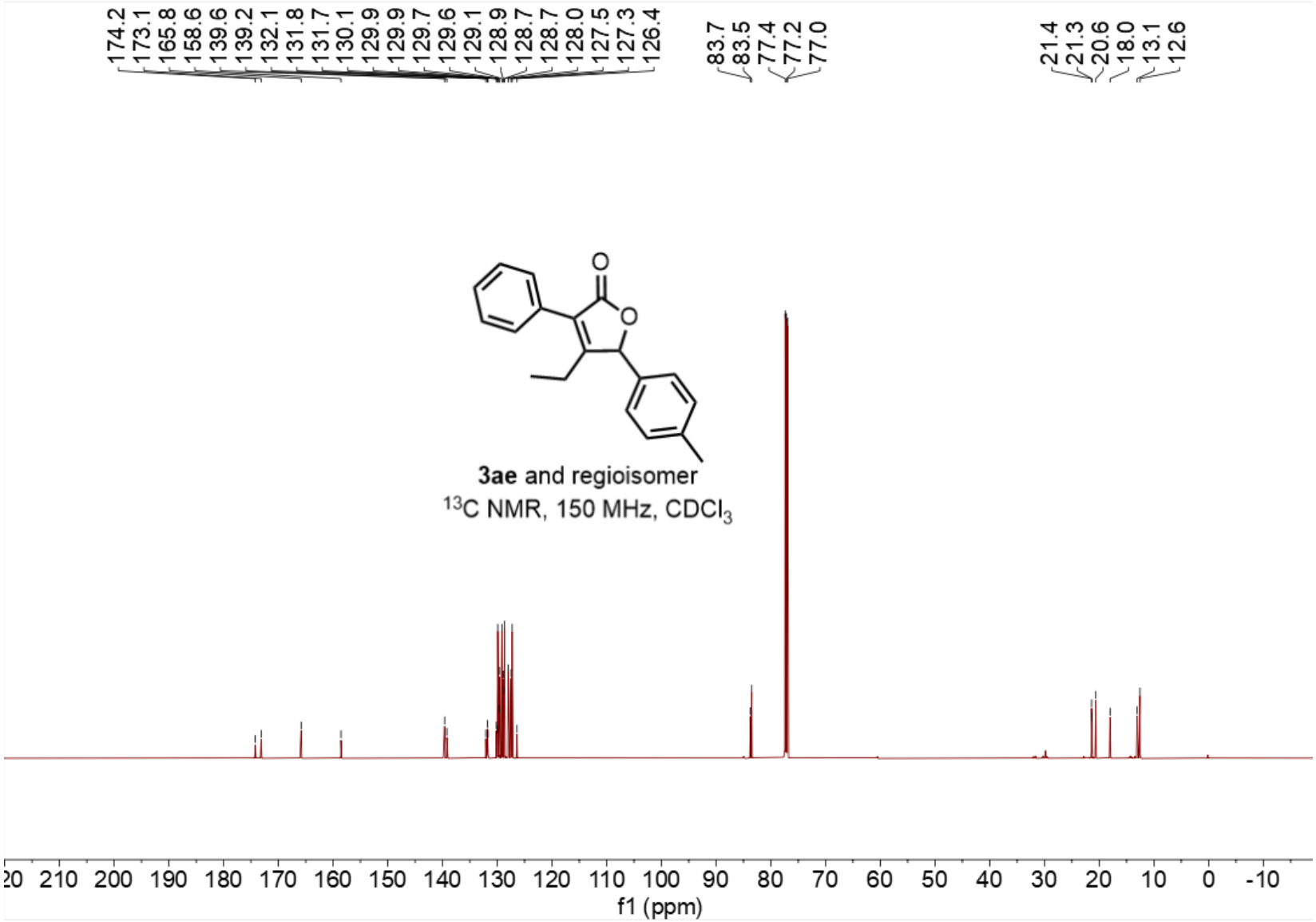

7.50
7.49
7.43
7.41
7.26
7.22
7.21
7.16
7.15
5.71
2.37
1.99
0.00
Cl
O
O
3ac
^{1}H NMR, 600 MHz, $CDCl_3$
1.89
1.89
1.91
1.92
0.96
2.98
2.95
f1 (ppm)

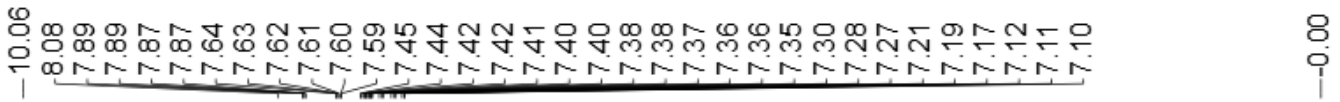

172.6
161.1
139.7
134.7
131.5
130.4
129.9
128.9
128.4
127.1
125.5
85.0
77.4
77.2
76.9
21.4
13.6
Cl
O
O
3ac
13C NMR, 150 MHz, CDCl3
190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0 -10
f1 (ppm)

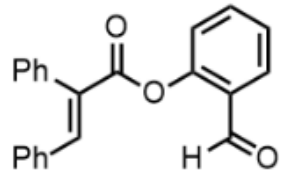

7.54
7.54
7.53
7.45
7.45
7.45
7.44
7.43
7.43
7.41
7.39
7.37
7.37
7.36
7.23
7.21
7.17
7.16
7.16
7.15
7.14
5.72
2.37
2.00
2.00
0.00
Cl
O
O
3ad
1H NMR, 400 MHz, CDCl3
0.98
1.00
2.05
2.06
2.03
1.00
3.01
2.99
10.5 10.0 9.5 9.0 8.5 8.0 7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 0.5 0.0 -0.5 -1
f1 (ppm)


172.4
161.7
139.7
134.6
131.8
131.5
129.9
129.1
128.8
127.3
127.1
125.4
85.0
77.5
77.2
76.8
21.4
13.5
Cl
O
O
3ad
13C NMR, 100 MHz, CDCl3
200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0 -1
f1 (ppm)


7.52
7.52
7.50
7.47
7.45
7.44
7.40
7.39
7.38
7.35
7.34
7.33
7.26
7.23
7.21
7.20
7.19
7.17
7.08
6.11
5.85
2.68
2.67
2.66
2.65
2.64
2.62
2.58
2.56
2.55
2.54
2.53
2.51
2.50
2.37
2.28
2.18
2.16
2.15
2.14
2.13
2.11
1.27
1.26
1.24
1.06
1.05
1.03
-0.00
O
O
3ae and regioisomer
1H NMR, 600 MHz, CDCl3
1.92
1.93
1.07
1.90
1.35
1.83
1.86
2.55
0.68
1.00
1.01
1.38
2.98
1.95
1.08
2.85
2.87
.0 10.5 10.0 9.5 9.0 8.5 8.0 7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 0.5 0.0 -0.5 -1.0
f1 (ppm)

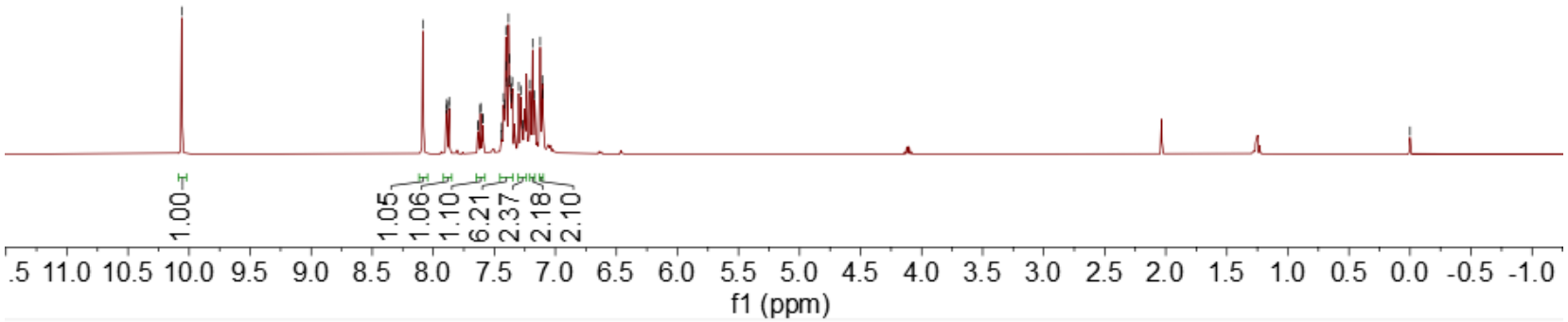

174.2
173.1
165.8
158.6
139.6
139.2
132.1
131.8
131.7
130.1
129.9
129.9
129.7
129.6
129.1
128.9
128.7
128.7
128.0
127.5
127.3
126.4
83.7
83.5
77.4
77.2
77.0
21.4
21.3
20.6
18.0
13.1
12.6
O
O
3ae and regioisomer
13C NMR, 150 MHz, CDCl3
f1 (ppm)

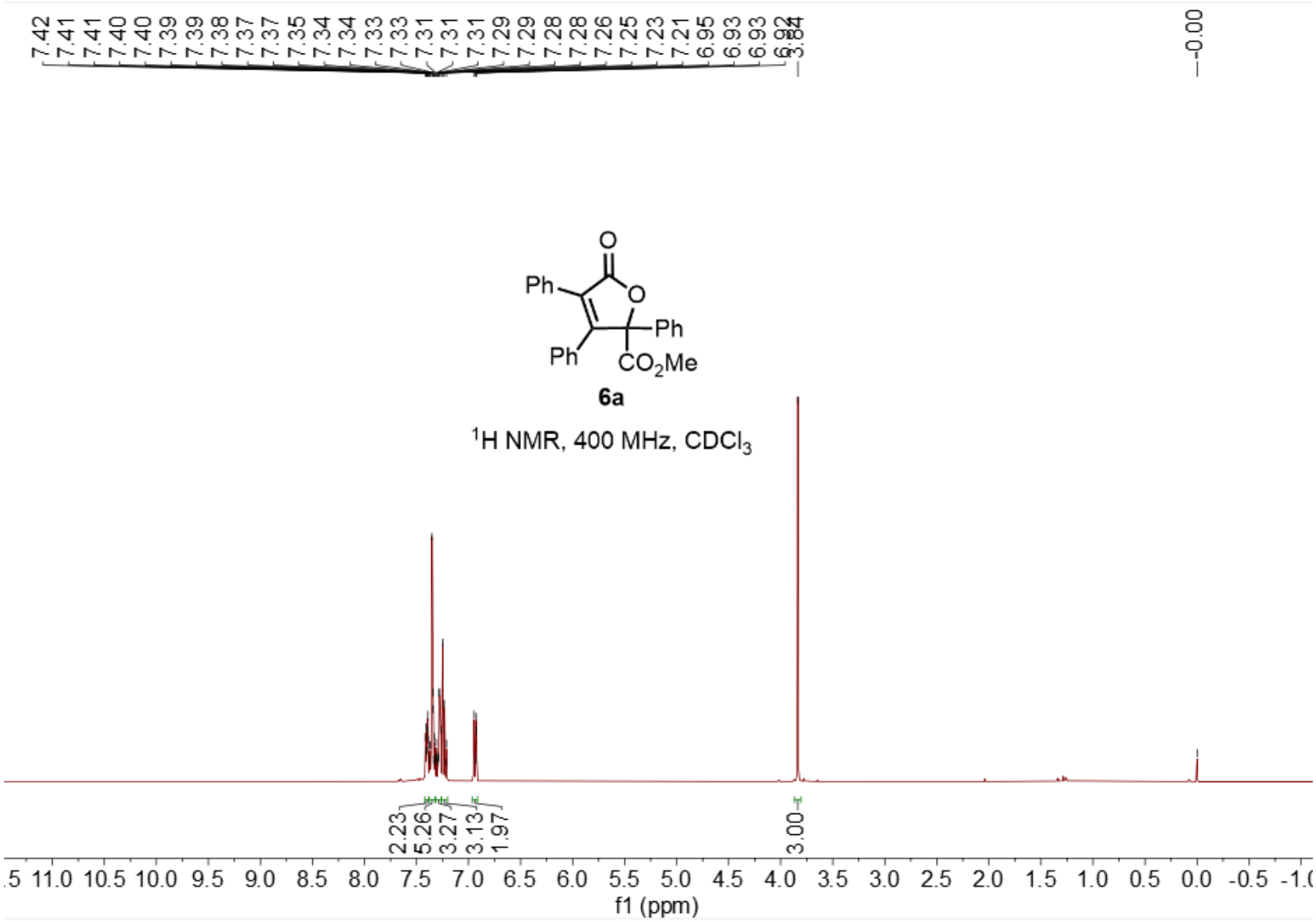


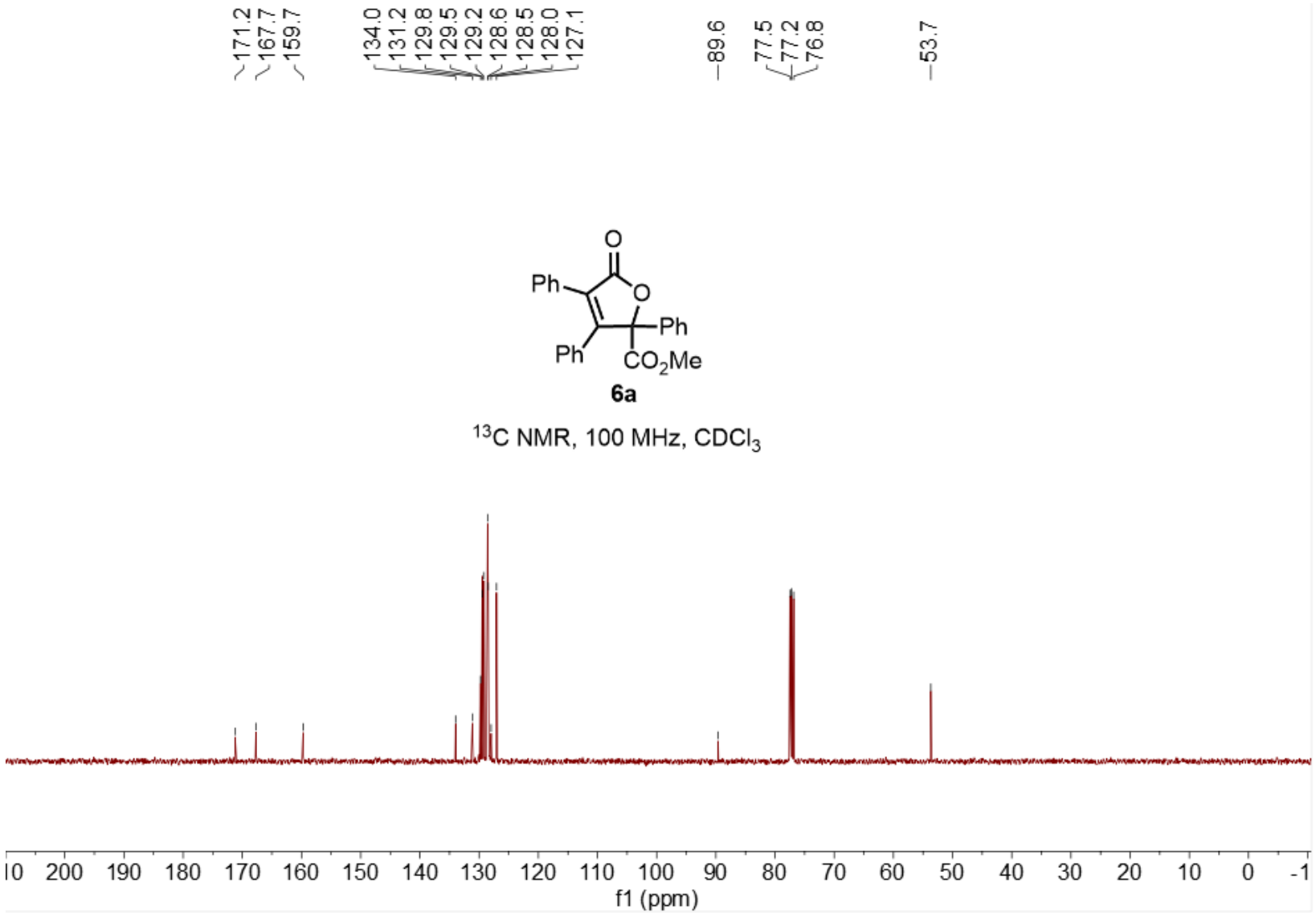


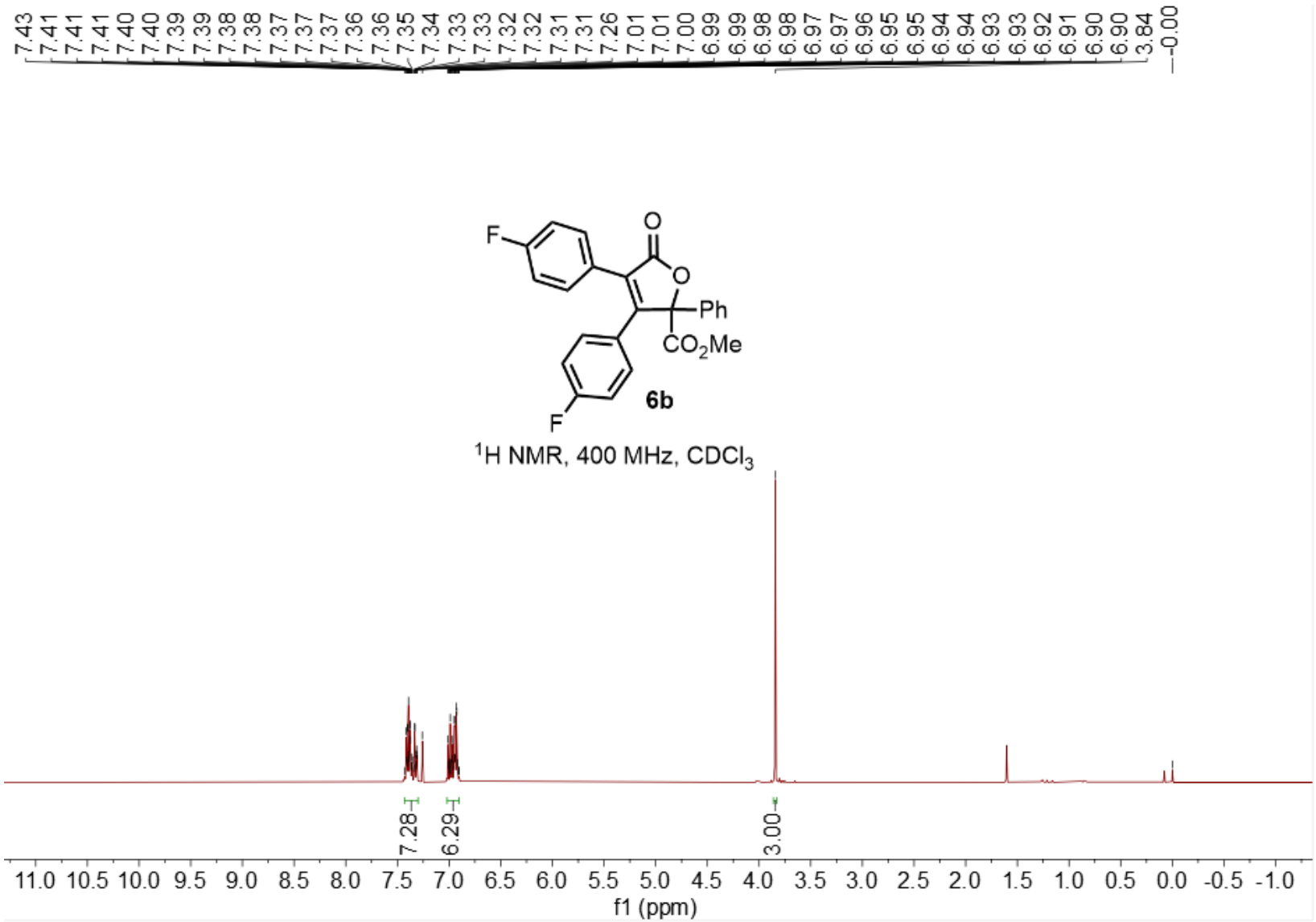
4

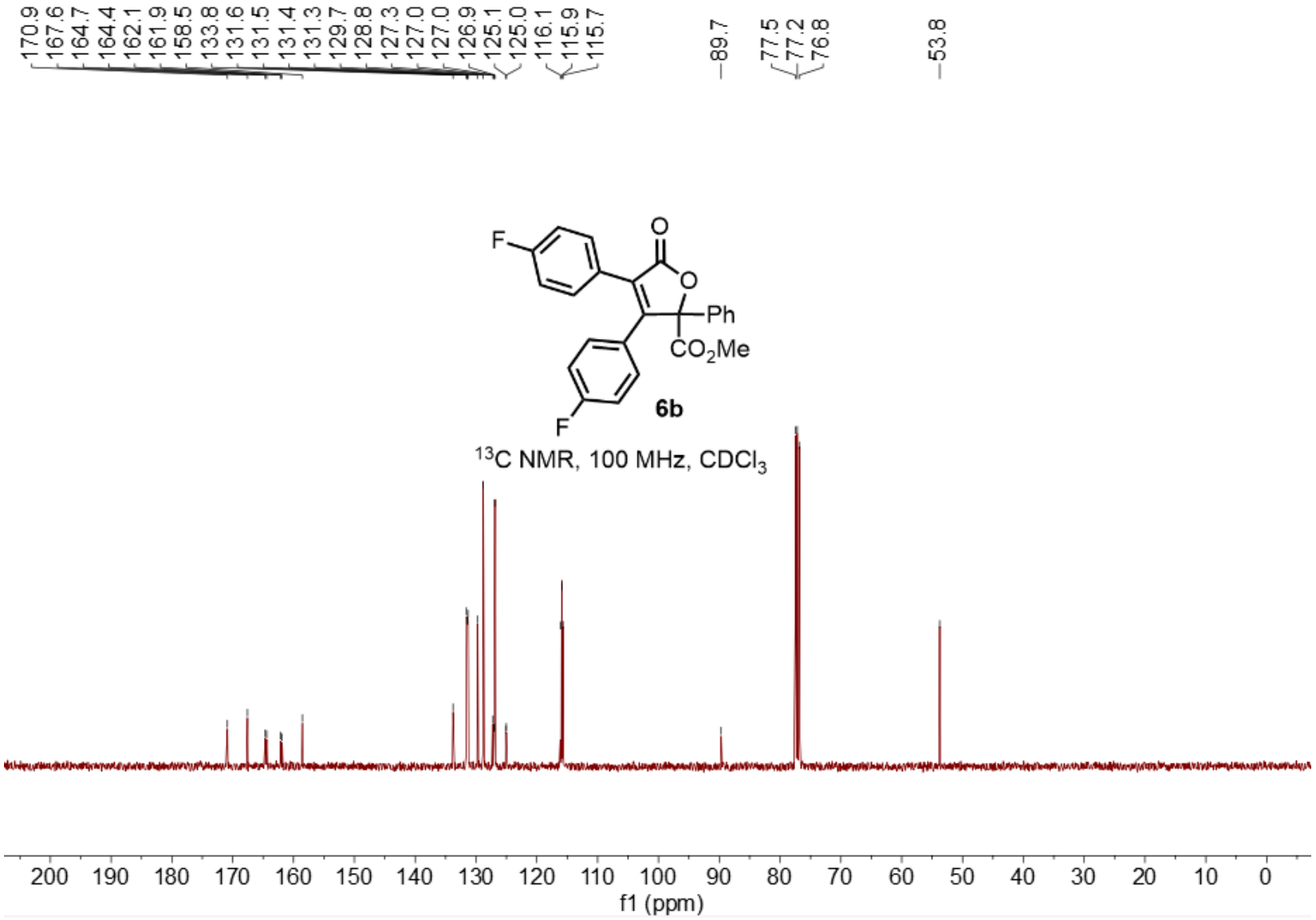
^{1}H NMR, 400 MHz, $CDCl_3$

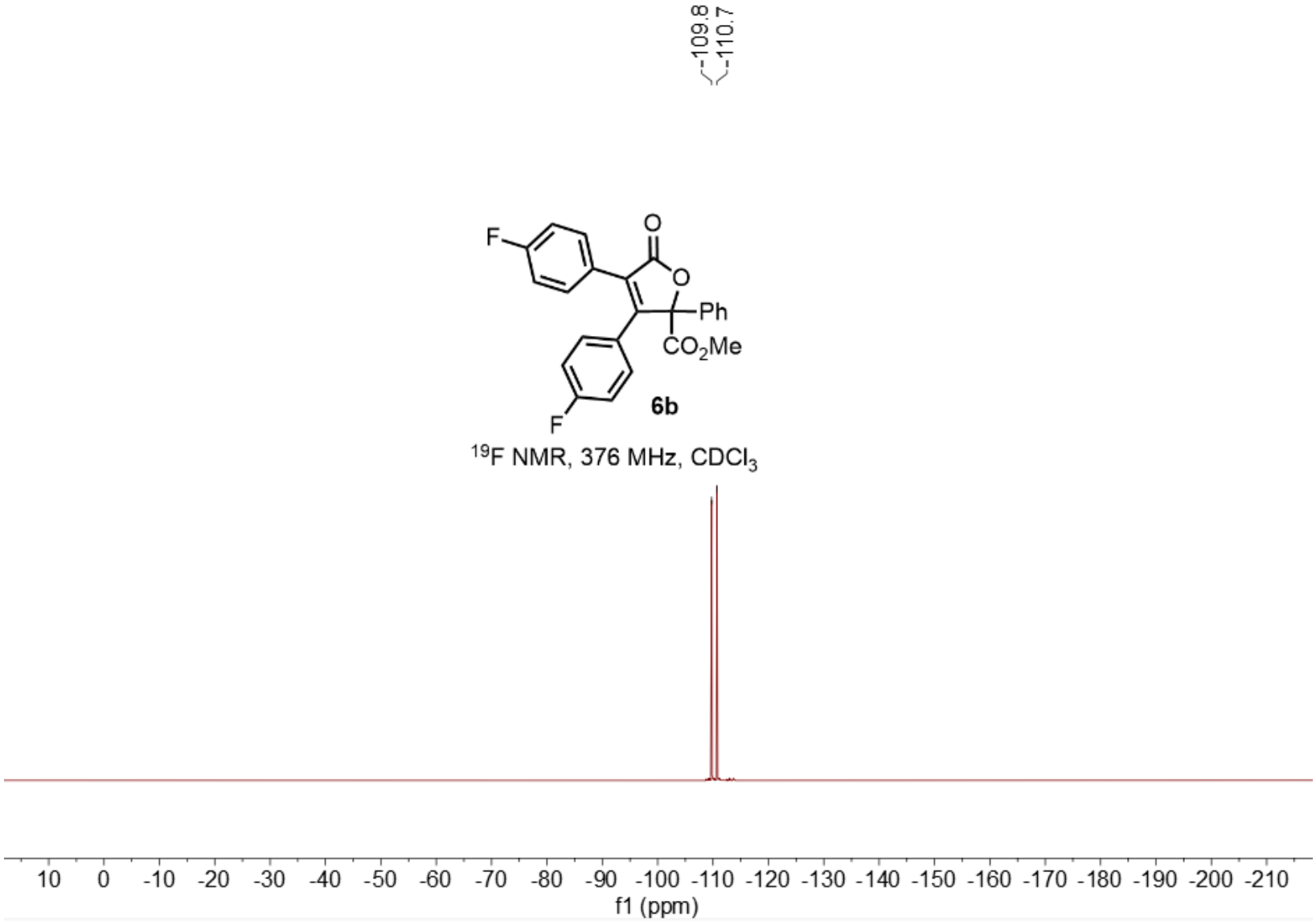


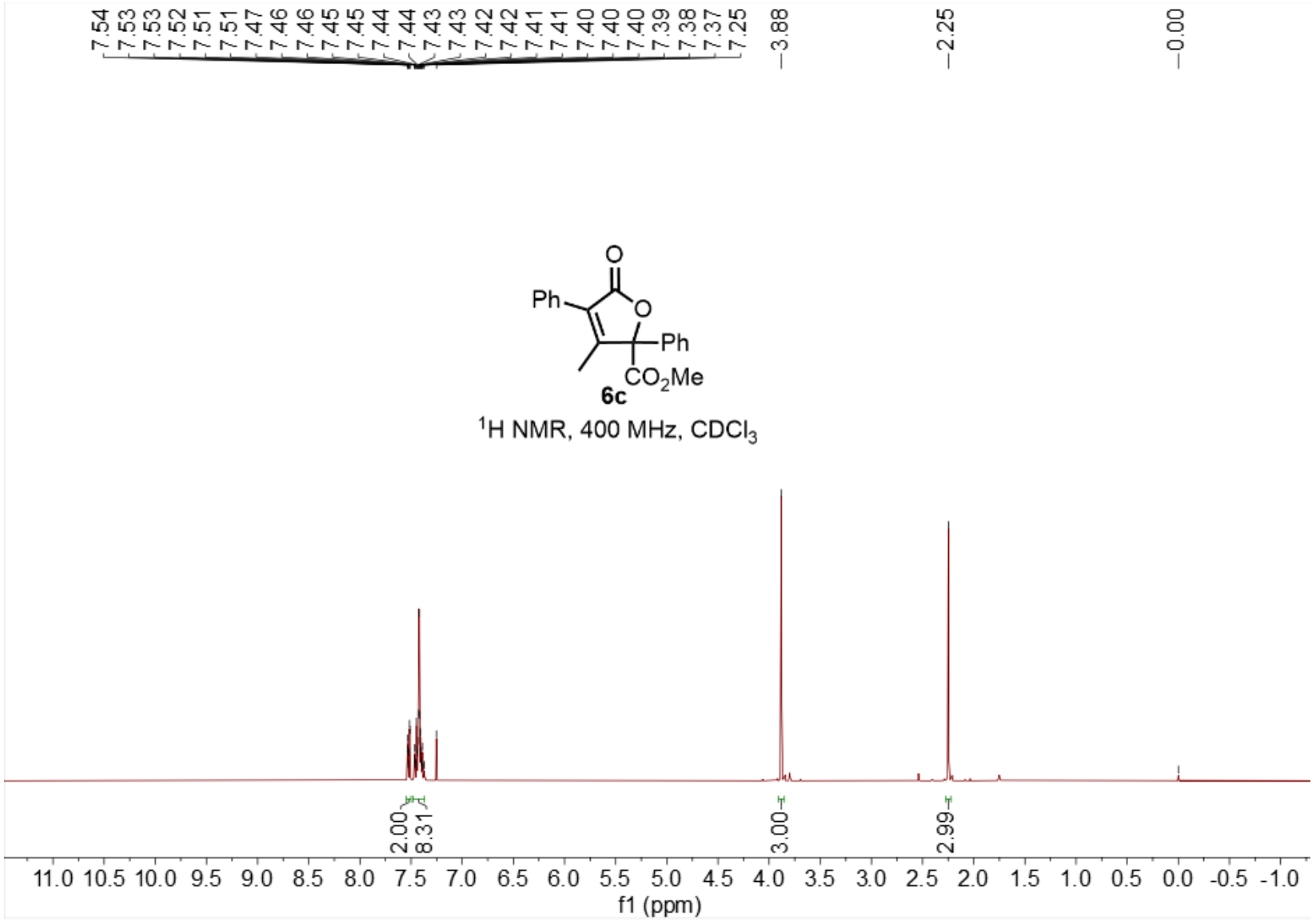

7.42
7.41
7.41
7.40
7.40
7.39
7.39
7.38
7.37
7.37
7.35
7.34
7.34
7.33
7.33
7.31
7.31
7.31
7.29
7.29
7.28
7.28
7.26
7.25
7.23
7.21
6.95
6.93
6.93
3.84
0.00
O
Ph
O
Ph
Ph
CO2Me
6a
1H NMR, 400 MHz, CDCl3
2.23
5.26
3.27
3.13
1.97
3.00
f1 (ppm)

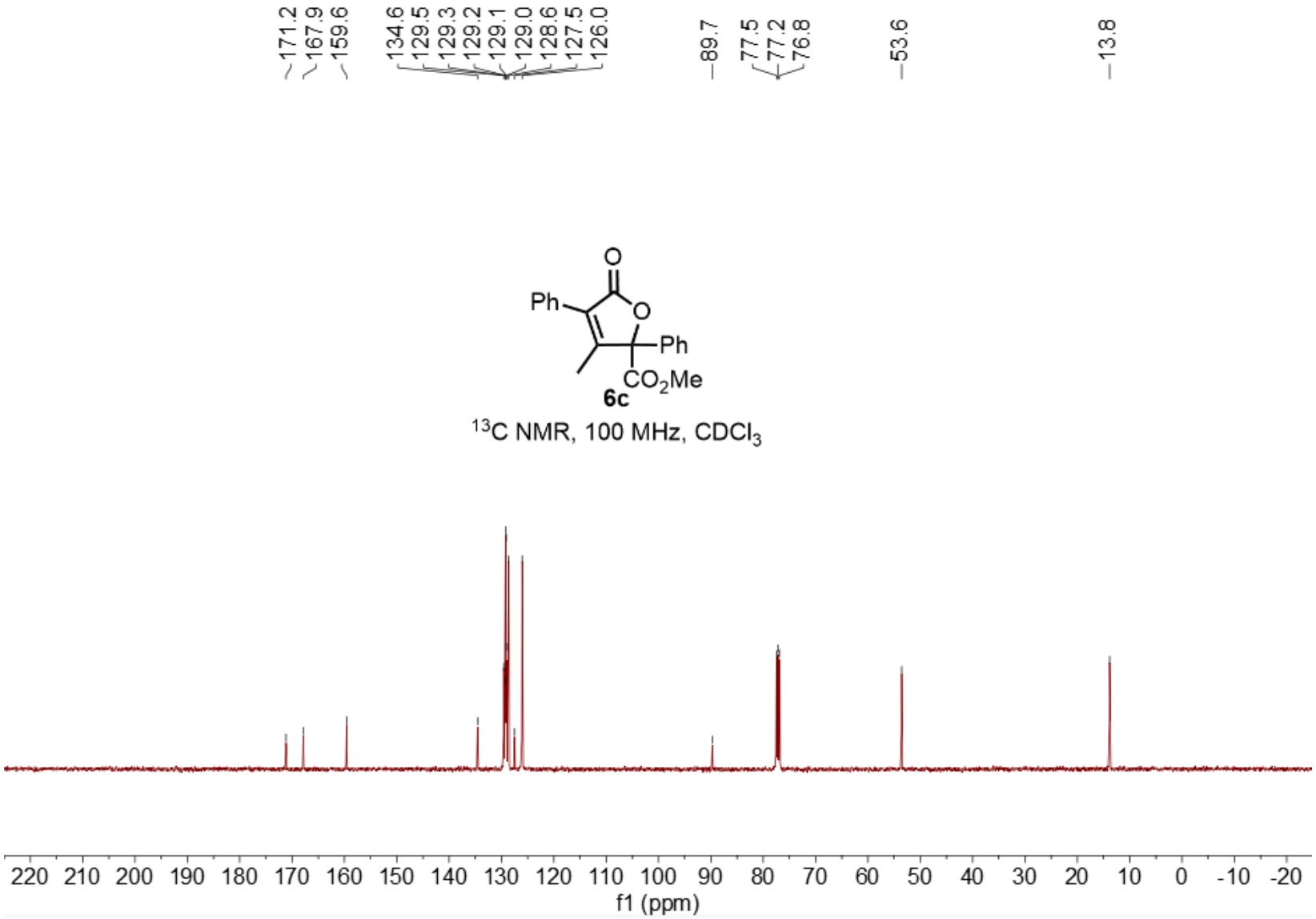

171.2
167.7
159.7
134.0
131.2
129.8
129.5
129.2
128.6
128.5
128.0
127.1
89.6
77.5
77.2
76.8
53.7
O
Ph
O
Ph
Ph
CO2Me
6a
13C NMR, 100 MHz, CDCl3
f1 (ppm)

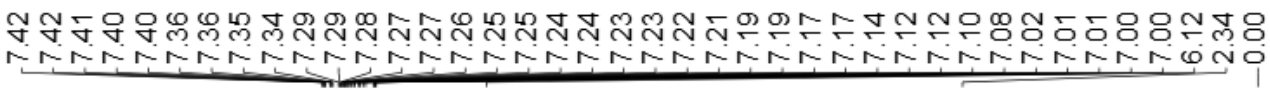

7.43
7.41
7.41
7.41
7.40
7.40
7.39
7.39
7.38
7.38
7.37
7.37
7.37
7.36
7.36
7.35
7.34
7.33
7.33
7.32
7.32
7.31
7.31
7.26
7.01
7.01
7.00
6.99
6.99
6.98
6.98
6.97
6.97
6.96
6.95
6.95
6.94
6.94
6.93
6.93
6.92
6.91
6.90
6.90
3.84
0.00
F
O
O
Ph
CO2Me
F
6b
1H NMR, 400 MHz, CDCl3
7.28
6.29
3.00
11.0 10.5 10.0 9.5 9.0 8.5 8.0 7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 0.5 0.0 -0.5 -1.0
f1 (ppm)

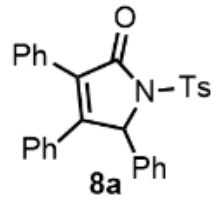

170.9
167.6
164.7
164.4
162.1
161.9
158.5
133.8
131.6
131.5
131.4
131.3
129.7
128.8
127.3
127.0
127.0
126.9
125.1
125.0
116.1
115.9
115.7
89.7
77.5
77.2
76.8
53.8
F
O
O
Ph
CO2Me
F
6b
13C NMR, 100 MHz, CDCl3
200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0
f1 (ppm)


-109.8
-110.7
F
O
O
Ph
CO2Me
F
6b
19F NMR, 376 MHz, CDCl3
10 0 -10 -20 -30 -40 -50 -60 -70 -80 -90 -100 -110 -120 -130 -140 -150 -160 -170 -180 -190 -200 -210
f1 (ppm)


7.54
7.53
7.53
7.52
7.51
7.51
7.47
7.46
7.46
7.45
7.45
7.44
7.44
7.43
7.43
7.42
7.42
7.41
7.41
7.40
7.40
7.40
7.39
7.38
7.37
7.25
3.88
2.25
0.00
Ph
O
O
Ph
CO2Me
6c
1H NMR, 400 MHz, CDCl3
2.00
8.31
3.00
2.99
11.0 10.5 10.0 9.5 9.0 8.5 8.0 7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 0.5 0.0 -0.5 -1.0
f1 (ppm)

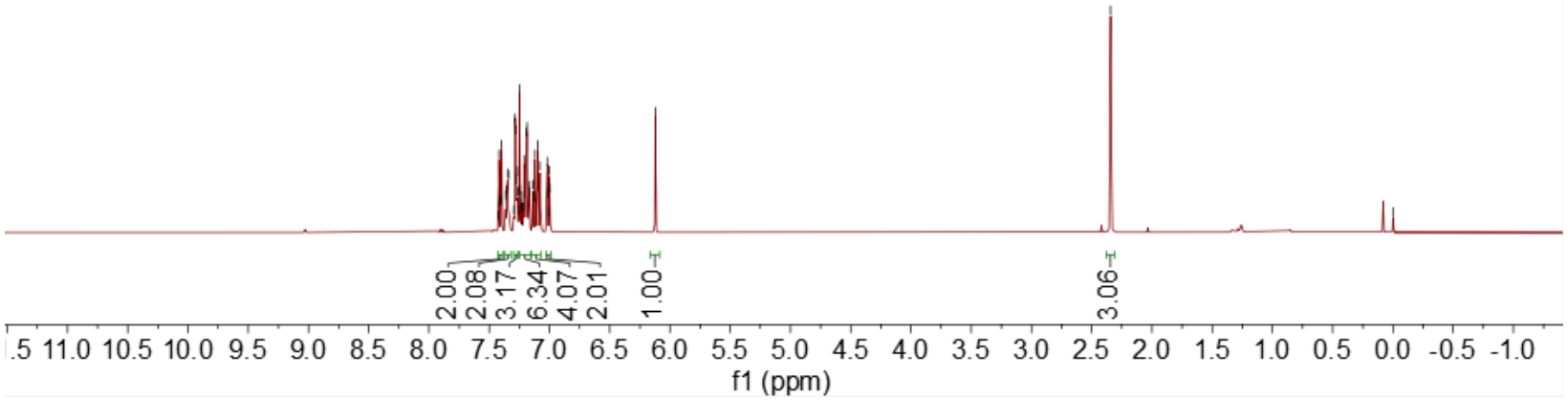

171.2
167.9
159.6
134.6
129.5
129.3
129.2
129.1
129.0
128.6
127.5
126.0
89.7
77.5
77.2
76.8
53.6
13.8
O
Ph
O
Ph
CO2Me
6c
13C NMR, 100 MHz, CDCl3
220 210 200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0 -10 -20
f1 (ppm)

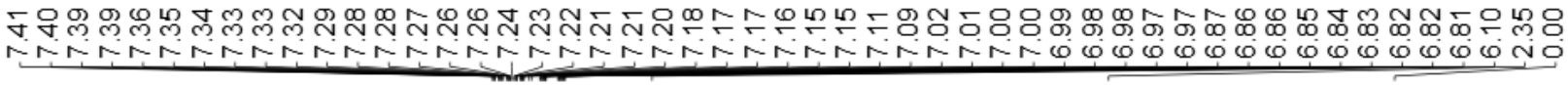


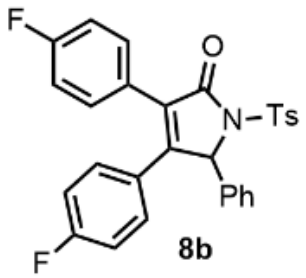


8a

^{1}H NMR, 400 MHz, $CDCl_3$

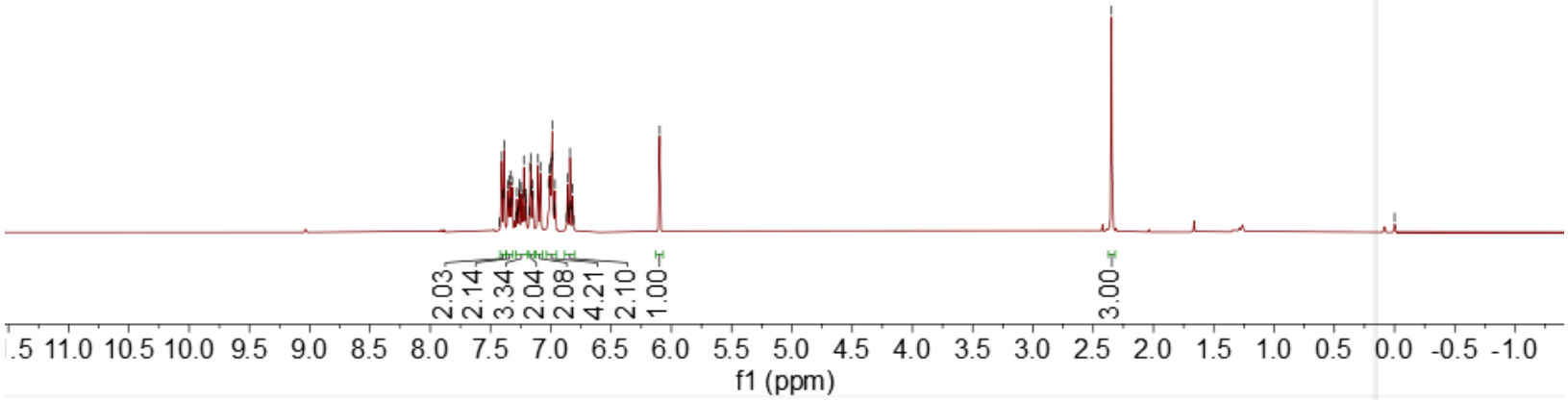


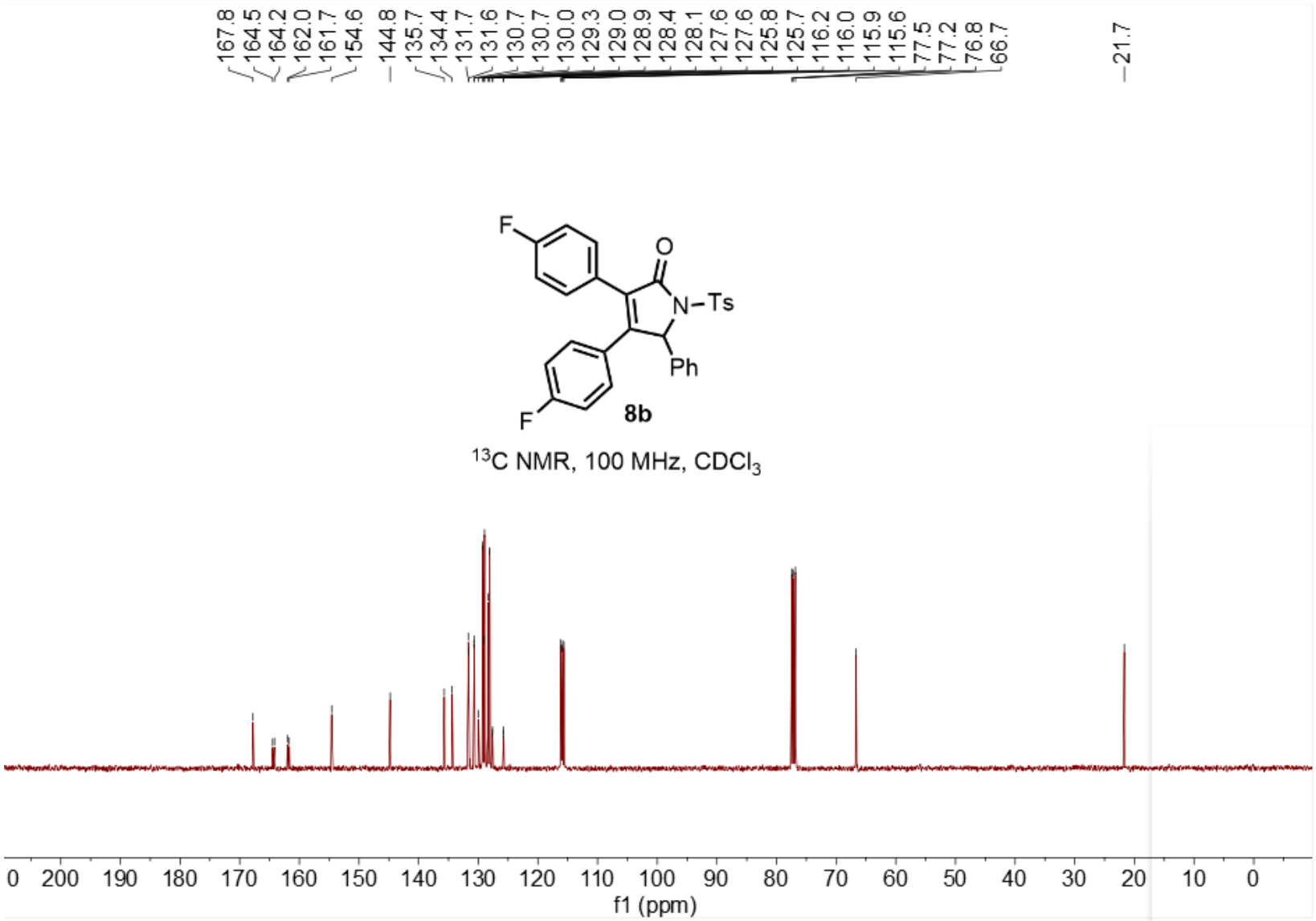


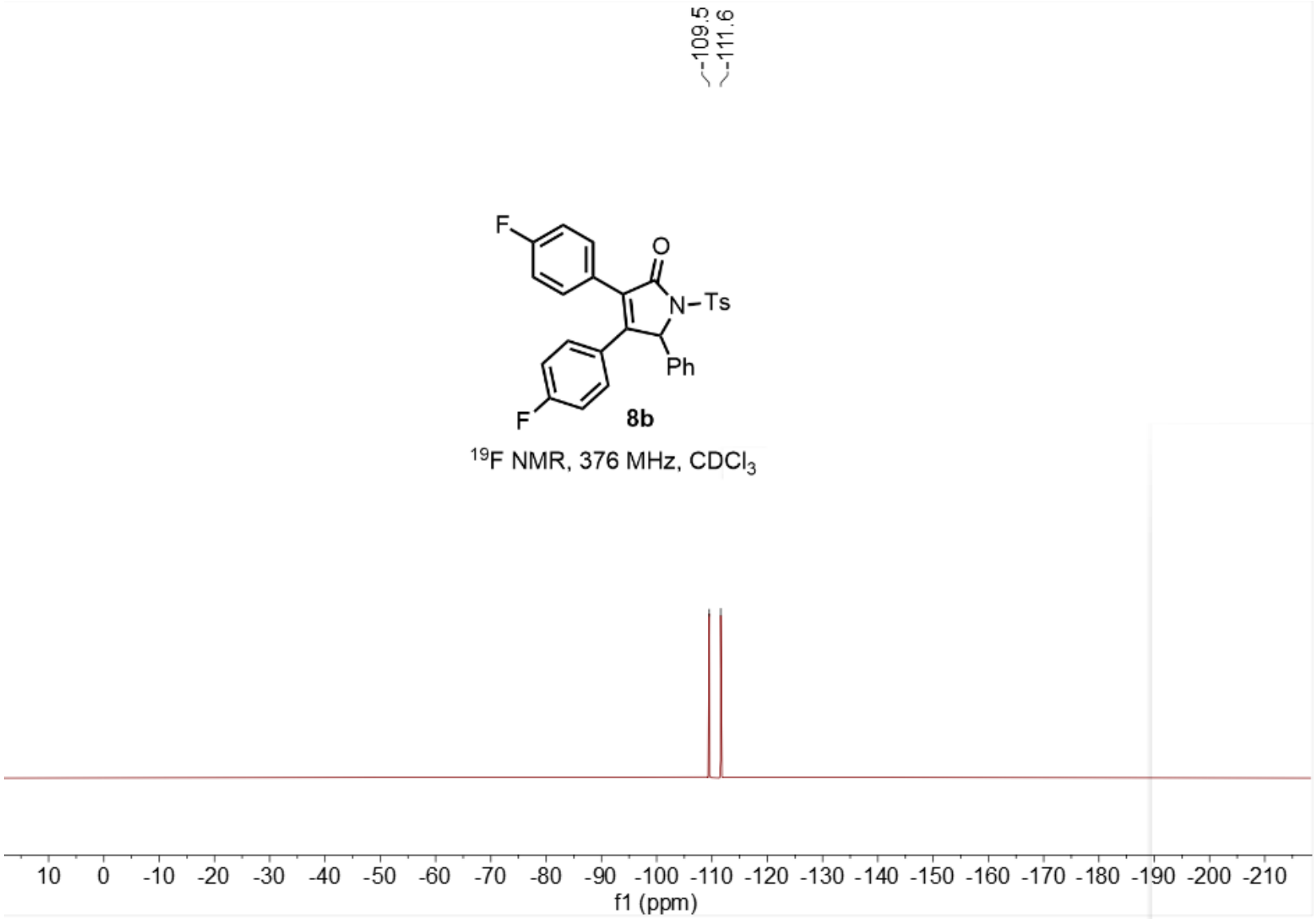


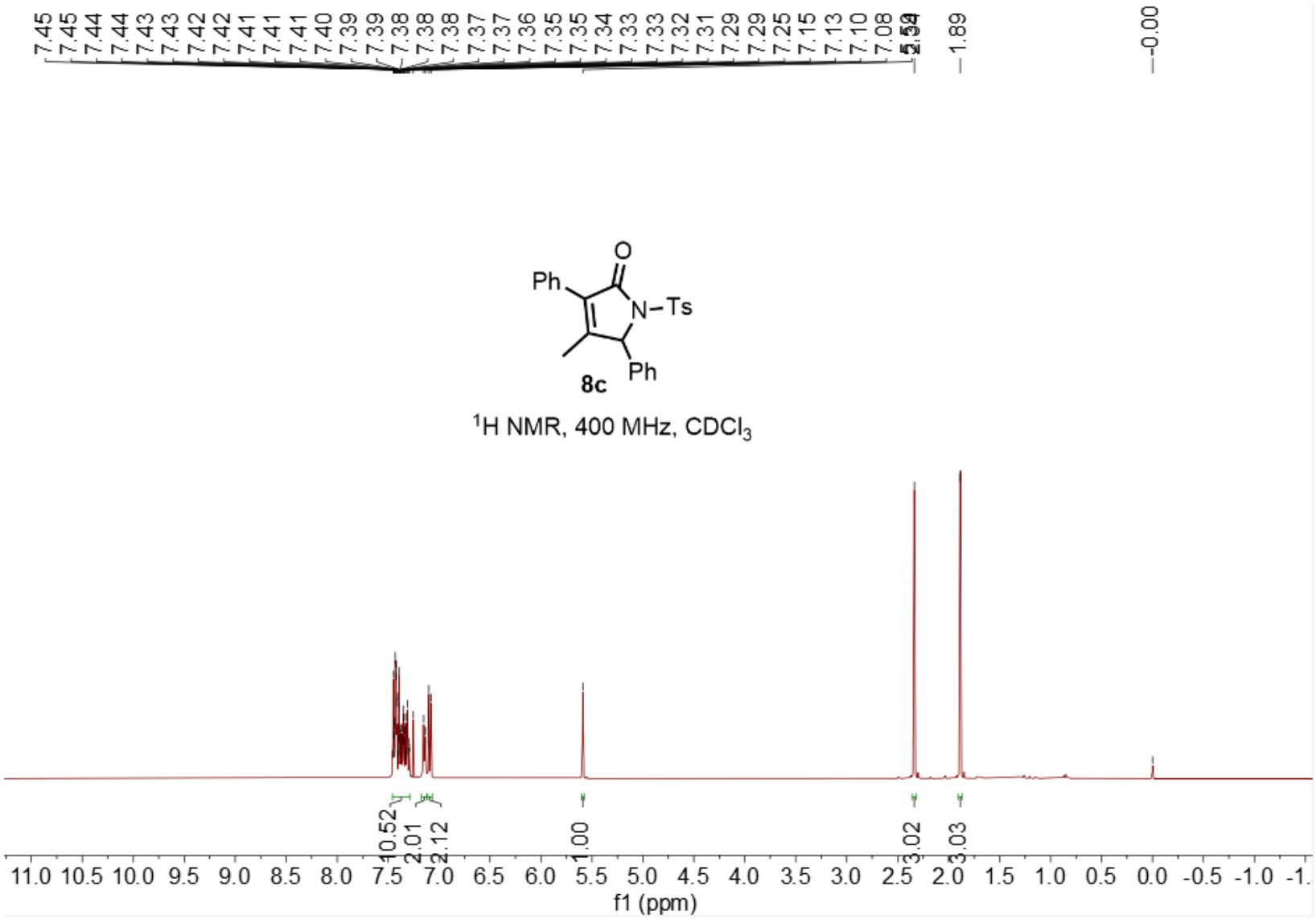
8b

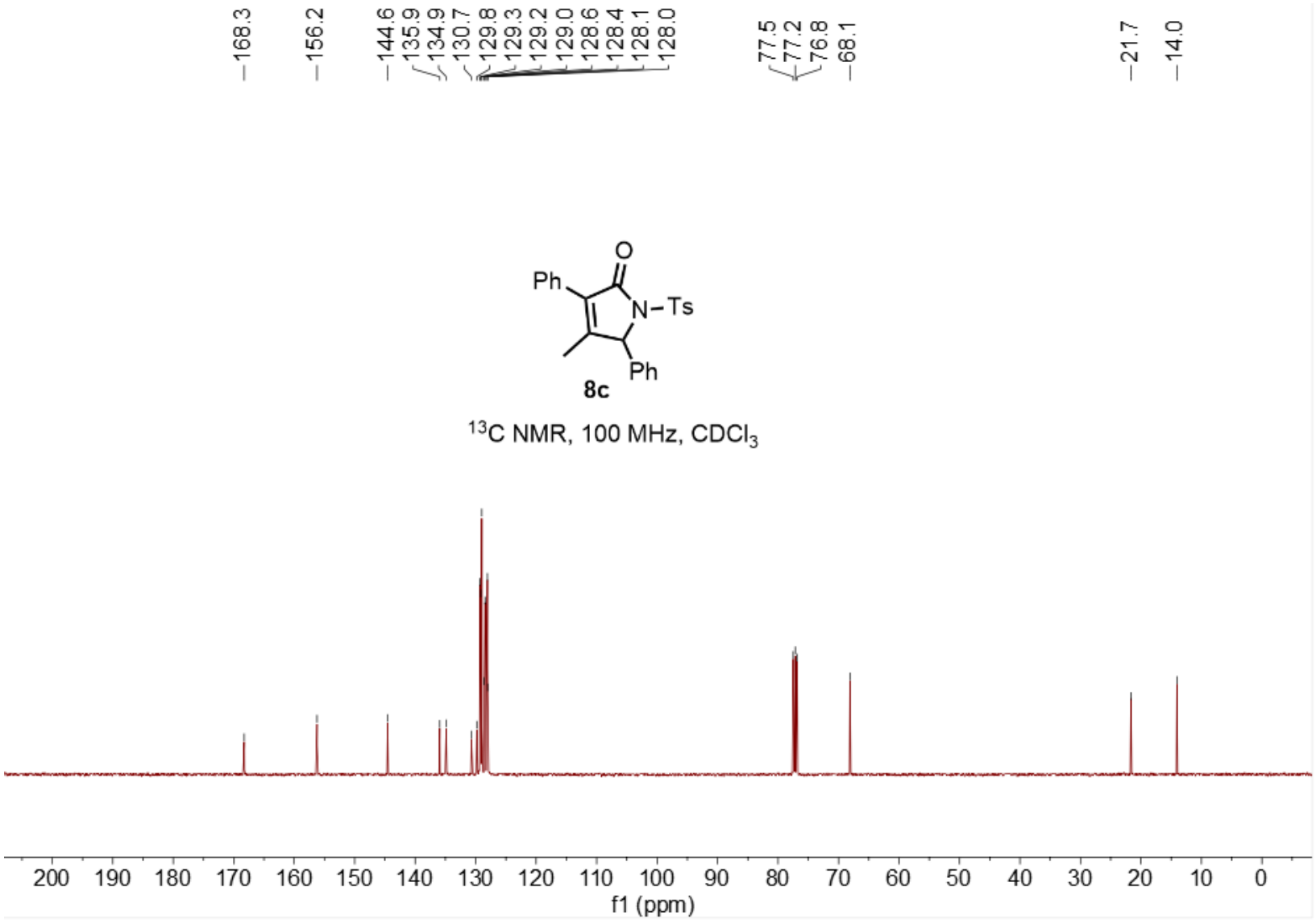
^{1}H NMR, 400 MHz, $CDCl_3$




167.8
164.5
164.2
162.0
161.7
154.6
144.8
135.7
134.4
131.7
131.6
130.7
130.7
130.0
129.3
129.0
128.9
128.4
128.1
127.6
127.6
125.8
125.7
116.2
116.0
115.9
115.6
77.5
77.2
76.8
66.7
21.7
F
O
N–Ts
Ph
F
8b
^{13}C NMR, 100 MHz, $CDCl_3$
0 200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0
f1 (ppm)

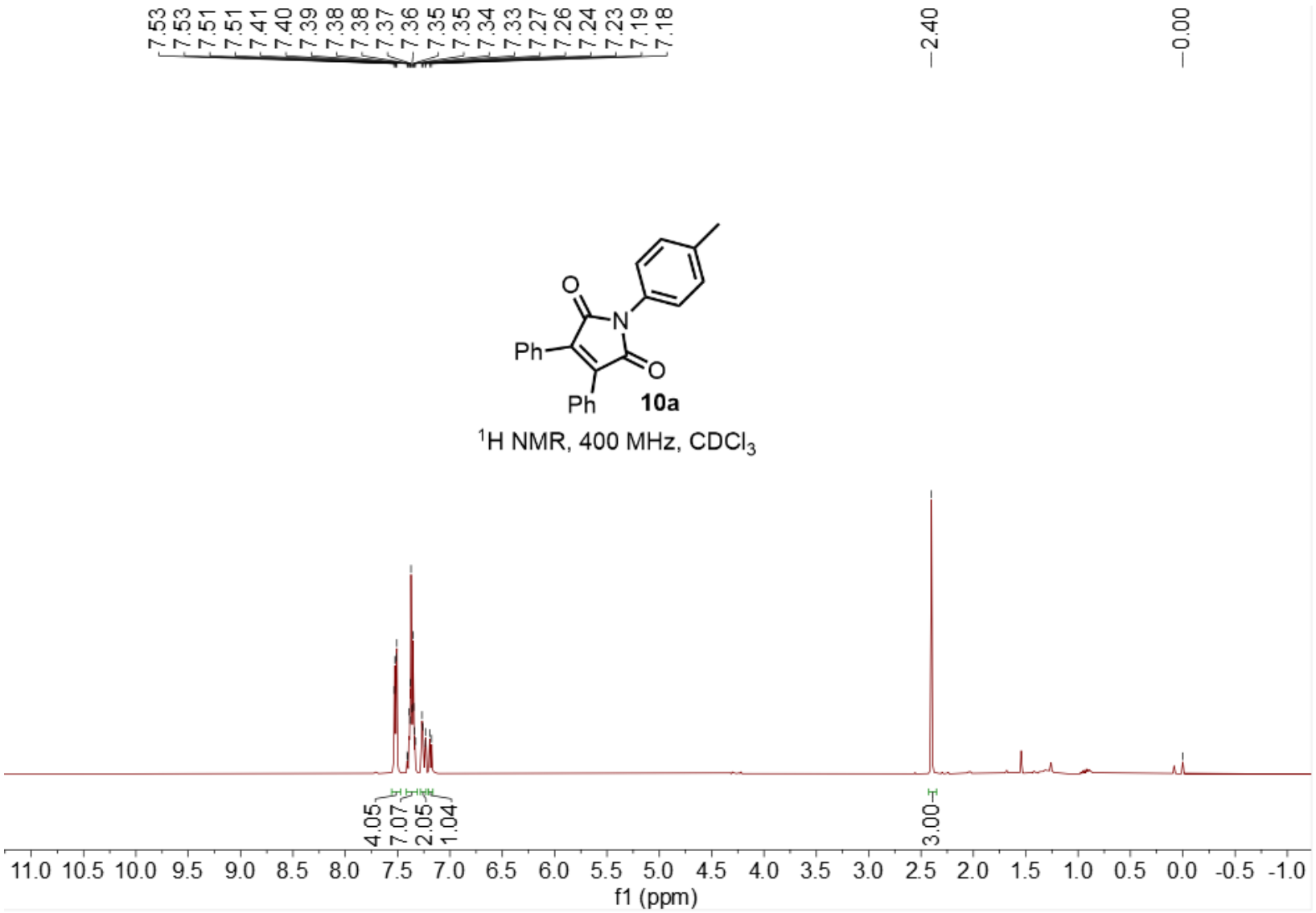

-109.5
-111.6
F
O
N–Ts
Ph
F
8b
^{19}F NMR, 376 MHz, $CDCl_3$
10 0 -10 -20 -30 -40 -50 -60 -70 -80 -90 -100 -110 -120 -130 -140 -150 -160 -170 -180 -190 -200 -210
f1 (ppm)

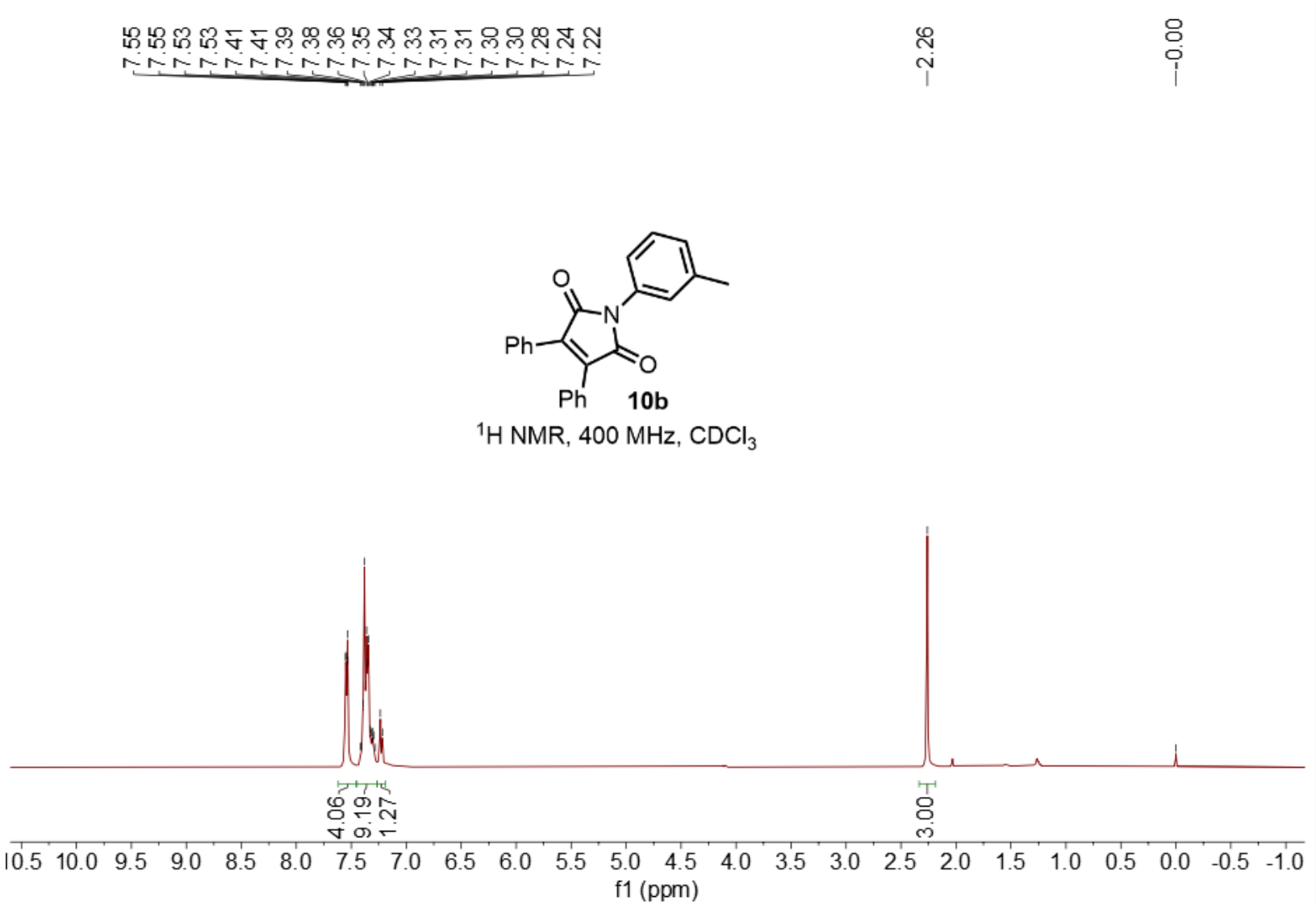

7.45 7.45 7.44 7.44 7.43 7.43 7.42 7.42 7.41 7.41 7.41 7.40 7.39 7.39 7.38 7.38 7.38 7.37 7.37 7.36 7.35 7.35 7.34 7.33 7.33 7.32 7.31 7.29 7.29 7.25 7.15 7.13 7.10 7.08
1.89
-0.00
O
Ph
N–Ts
Ph
8c
^{1}H NMR, 400 MHz, $CDCl_3$
10.52
2.01
2.12
1.00
3.02
3.03
11.0 10.5 10.0 9.5 9.0 8.5 8.0 7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 0.5 0.0 -0.5 -1.0 -1.
f1 (ppm)

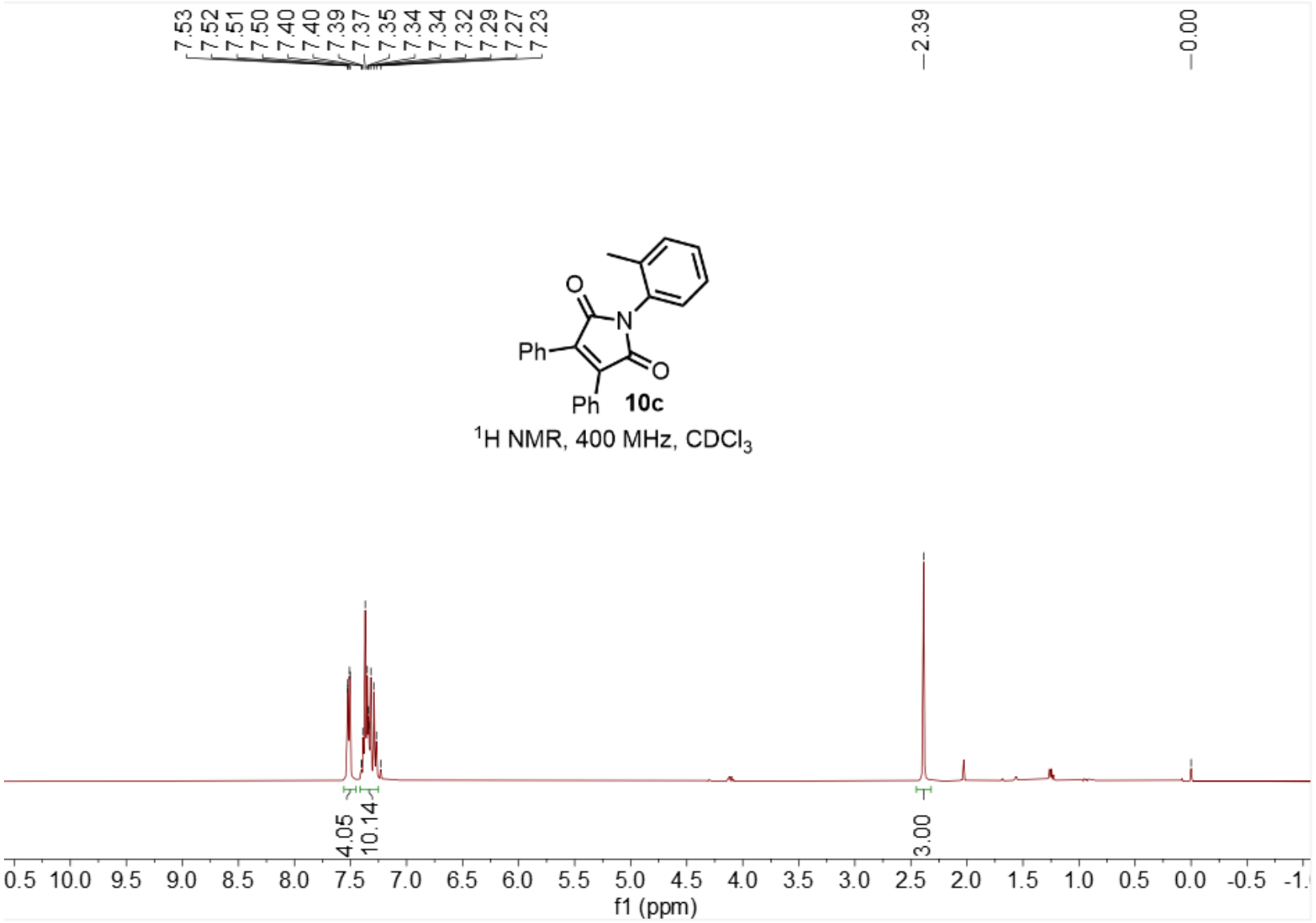


8c

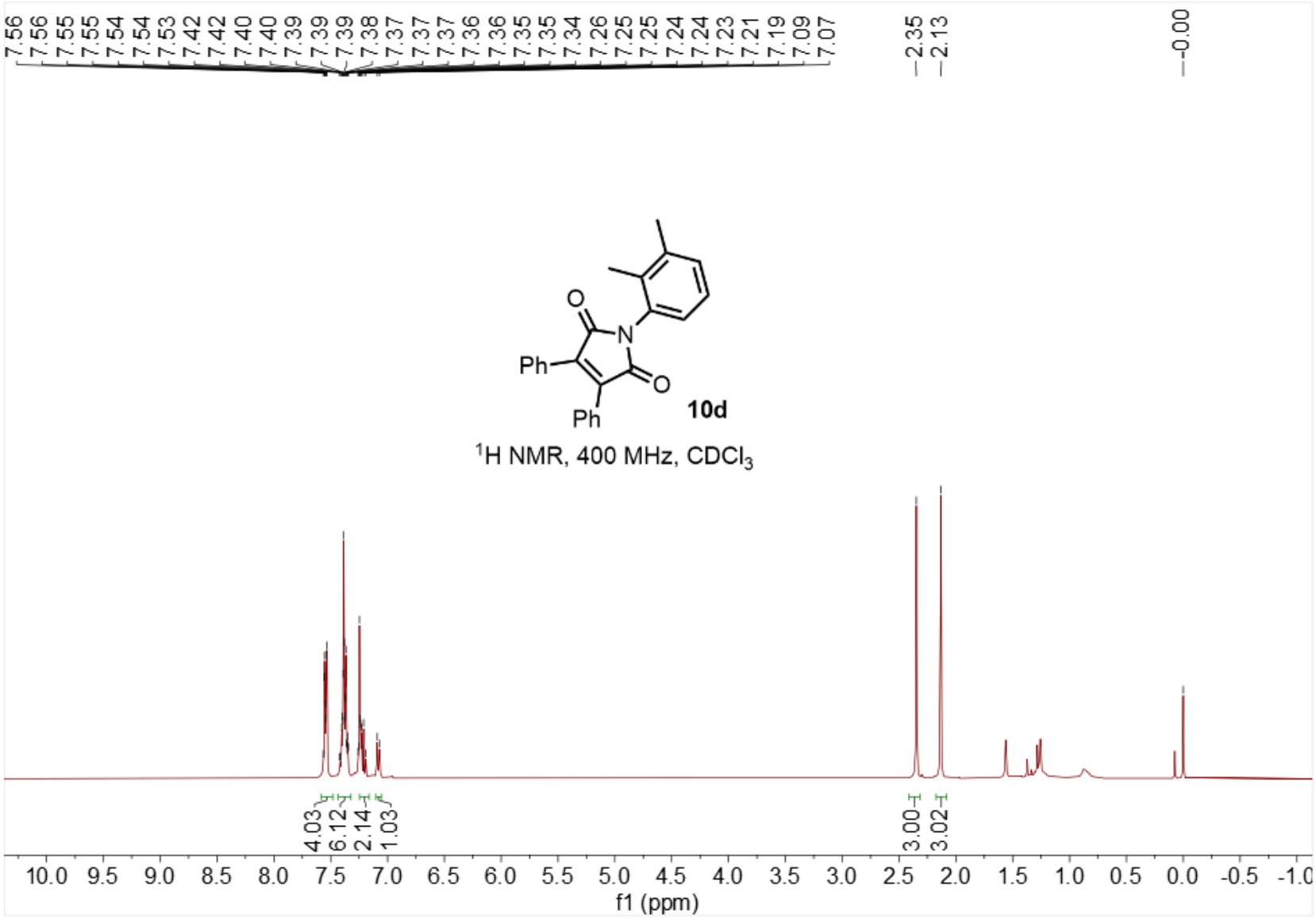
^{13}C NMR, 100 MHz, $CDCl_3$

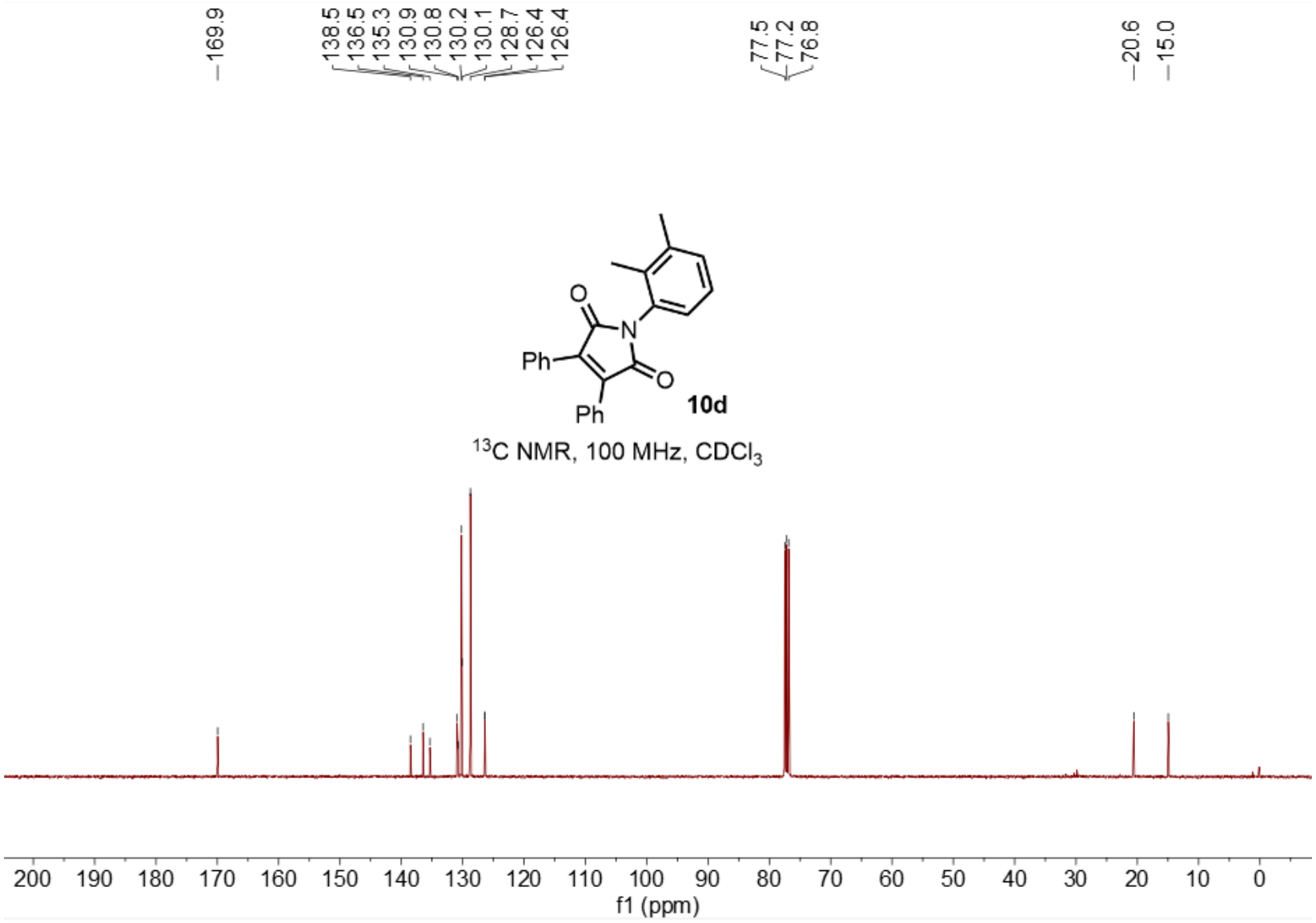

7.53
7.53
7.51
7.51
7.41
7.40
7.39
7.38
7.38
7.37
7.36
7.35
7.35
7.34
7.33
7.27
7.26
7.24
7.23
7.19
7.18
2.40
0.00
O
N
Ph
O
Ph
10a
1H NMR, 400 MHz, CDCl3
4.05
7.07
2.05
1.04
3.00
11.0 10.5 10.0 9.5 9.0 8.5 8.0 7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 0.5 0.0 -0.5 -1.0
f1 (ppm)

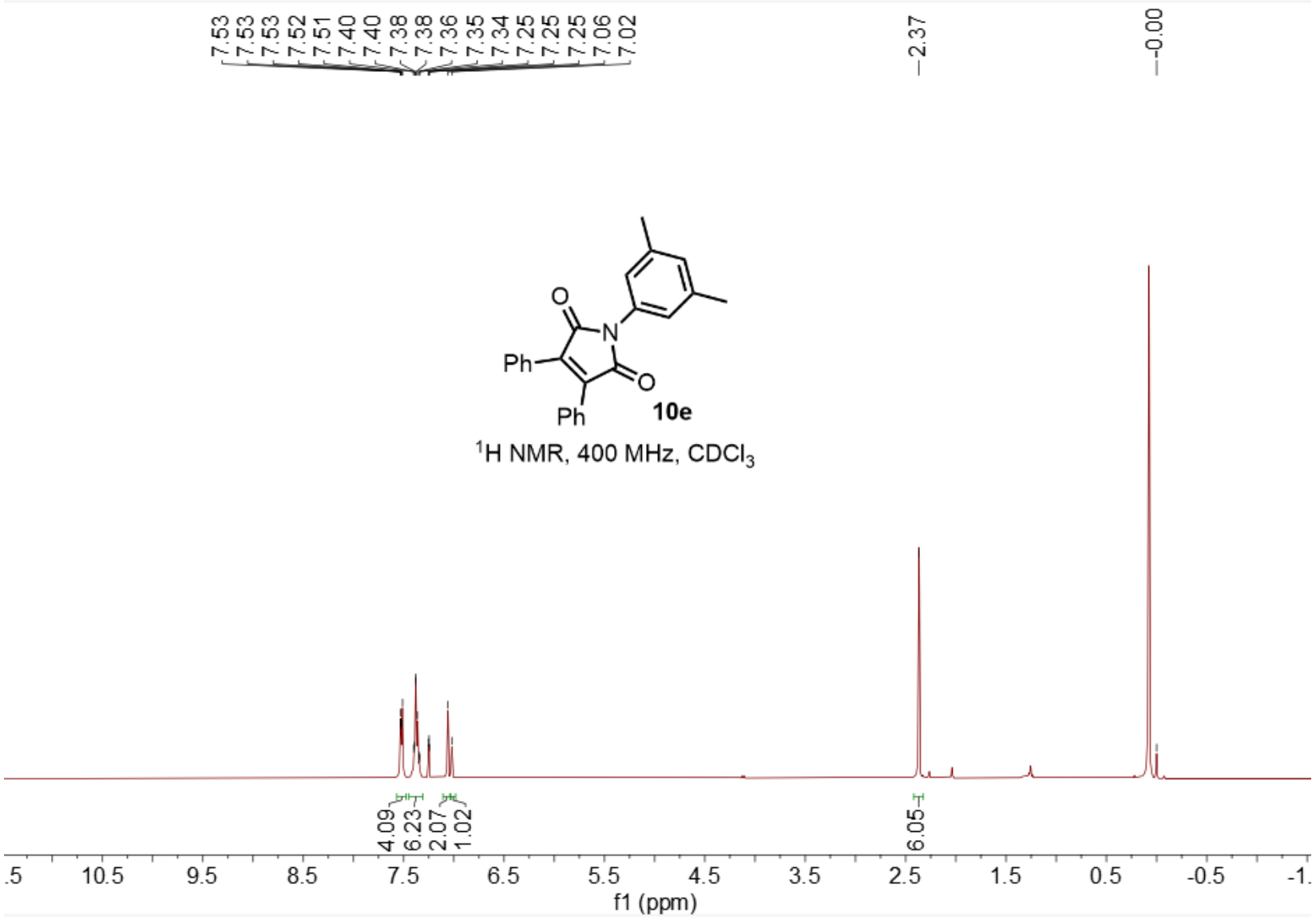

7.55
7.55
7.53
7.53
7.41
7.41
7.39
7.38
7.36
7.35
7.34
7.33
7.31
7.31
7.30
7.30
7.28
7.24
7.22
2.26
0.00
Ph
Ph
10b
1H NMR, 400 MHz, CDCl3
4.06
9.19
1.27
3.00
10.5 10.0 9.5 9.0 8.5 8.0 7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 0.5 0.0 -0.5 -1.0
f1 (ppm)

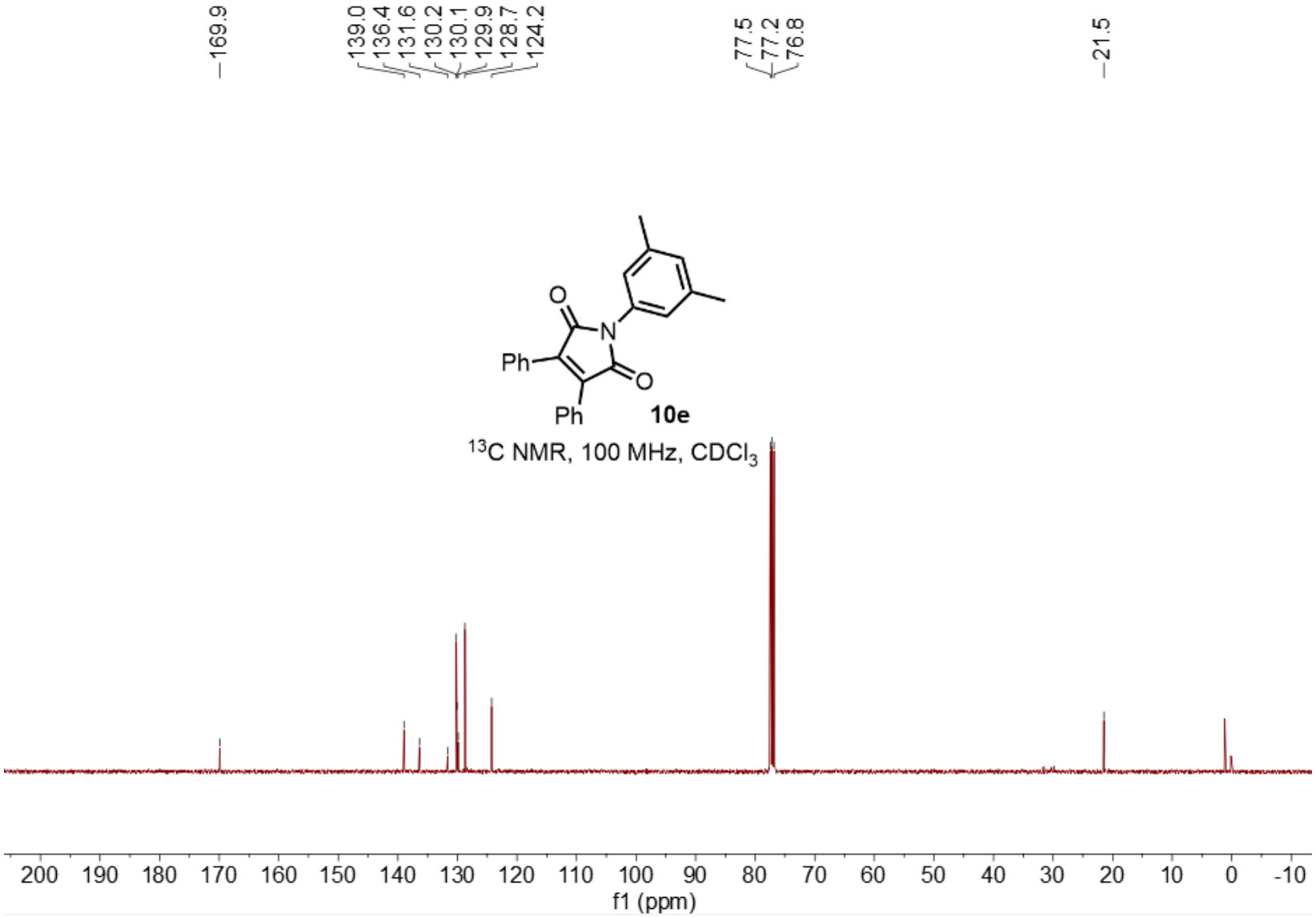


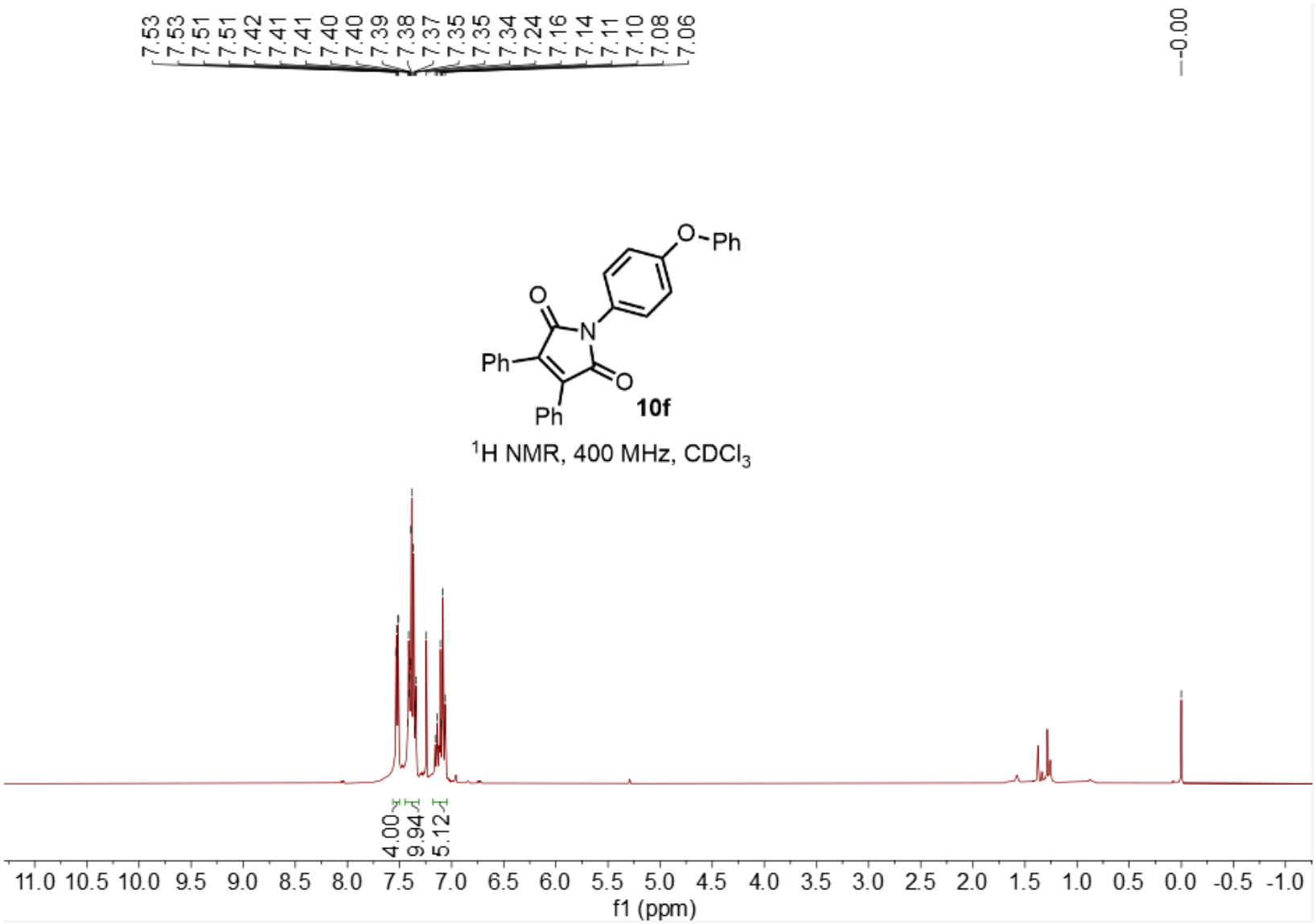
^{1}H NMR, 400 MHz, $CDCl_3$

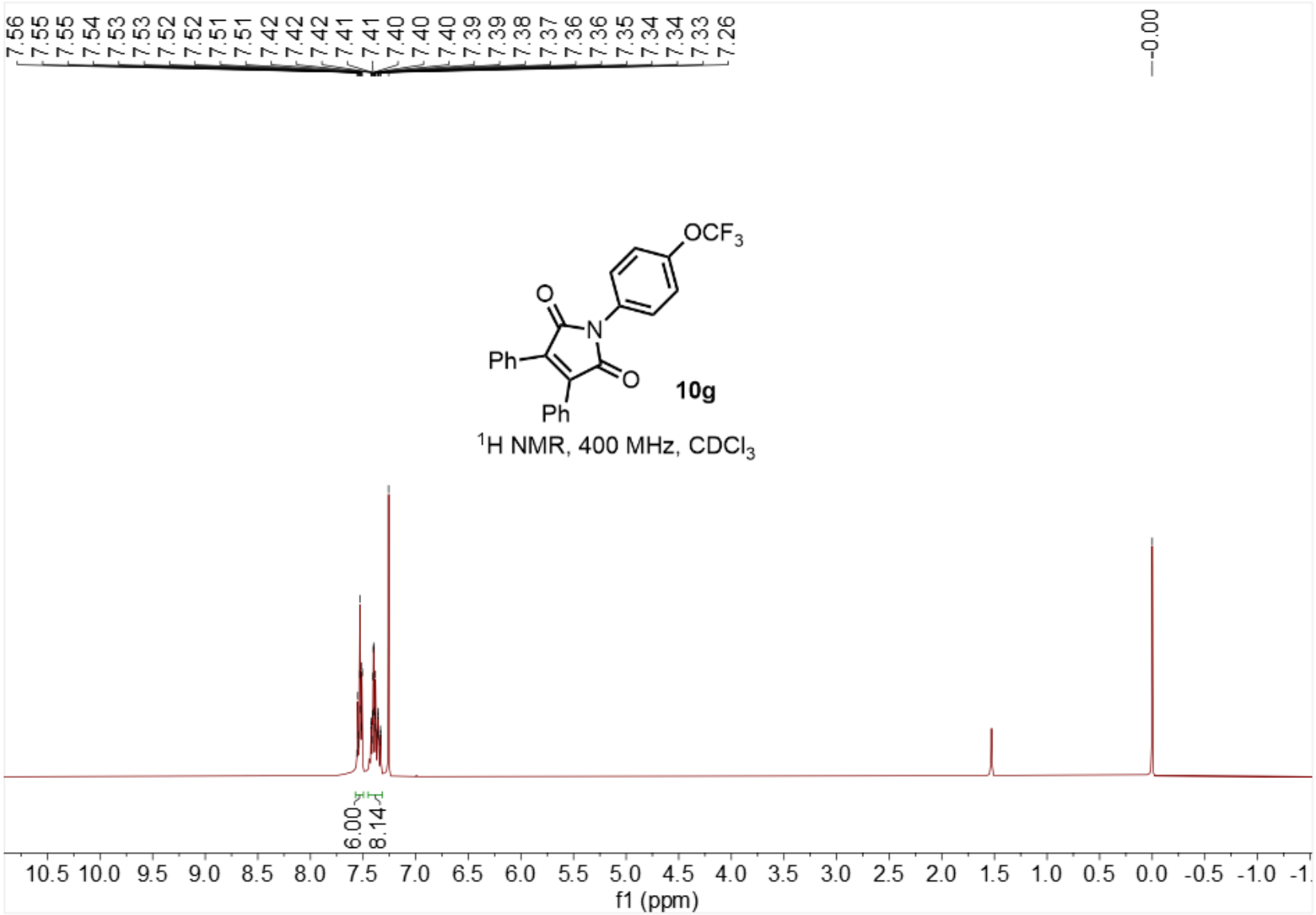

7.56 7.56 7.55 7.55 7.54 7.54 7.53 7.42 7.42 7.40 7.40 7.39 7.39 7.39 7.38 7.37 7.37 7.37 7.36 7.36 7.35 7.35 7.34 7.26 7.25 7.25 7.24 7.24 7.23 7.21 7.19 7.09 7.07
2.35
2.13
0.00
O
N
O
Ph
Ph
10d
^{1}H NMR, 400 MHz, $CDCl_3$
4.03
6.12
2.14
1.03
3.00
3.02
10.0 9.5 9.0 8.5 8.0 7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 0.5 0.0 -0.5 -1.0
f1 (ppm)


169.9
138.5
136.5
135.3
130.9
130.8
130.2
130.1
128.7
126.4
126.4
77.5
77.2
76.8
20.6
15.0
O
N
O
Ph
Ph
10d
13C NMR, 100 MHz, CDCl3
200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0
f1 (ppm)


7.53
7.53
7.53
7.52
7.51
7.40
7.40
7.38
7.38
7.36
7.35
7.34
7.25
7.25
7.25
7.06
7.02
2.37
0.00
O
N
O
Ph
Ph
10e
1H NMR, 400 MHz, CDCl3
4.09
6.23
2.07
1.02
6.05
10.5
9.5
8.5
7.5
6.5
5.5
4.5
3.5
2.5
1.5
0.5
-0.5
f1 (ppm)


169.9
139.0
136.4
131.6
130.2
130.1
129.9
128.7
124.2
77.5
77.2
76.8
21.5
O
N
O
Ph
Ph
10e
^{13}C NMR, 100 MHz, $CDCl_3$
200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0 -10
f1 (ppm)


7.53
7.53
7.51
7.51
7.42
7.41
7.41
7.40
7.40
7.39
7.38
7.37
7.35
7.35
7.34
7.24
7.16
7.14
7.11
7.10
7.08
7.06
0.00
O
Ph
N
O
Ph
O
Ph
10f
1H NMR, 400 MHz, CDCl3
4.00
9.94
5.12
11.0 10.5 10.0 9.5 9.0 8.5 8.0 7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 0.5 0.0 -0.5 -1.0
f1 (ppm)


7.56
7.55
7.55
7.54
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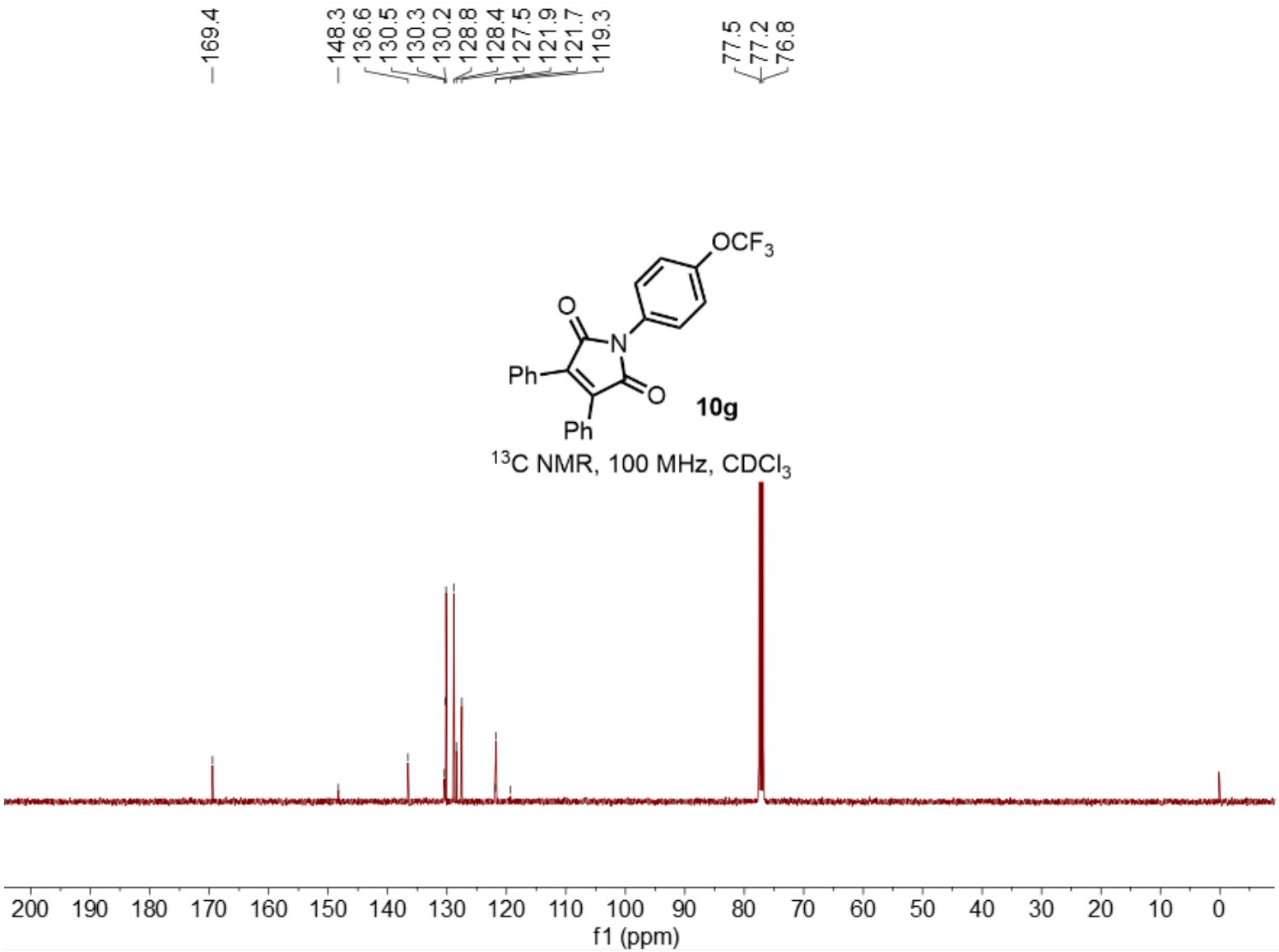
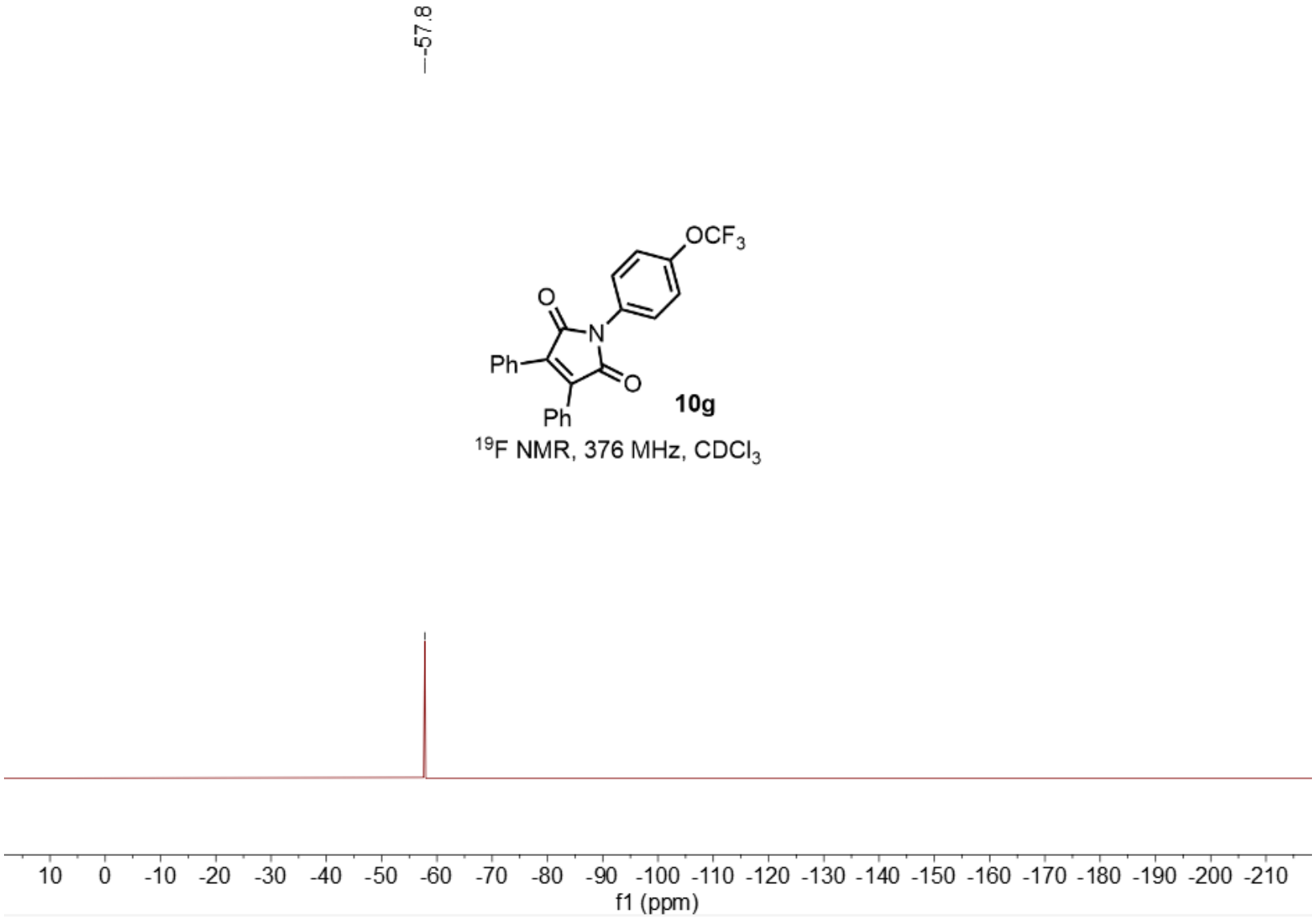
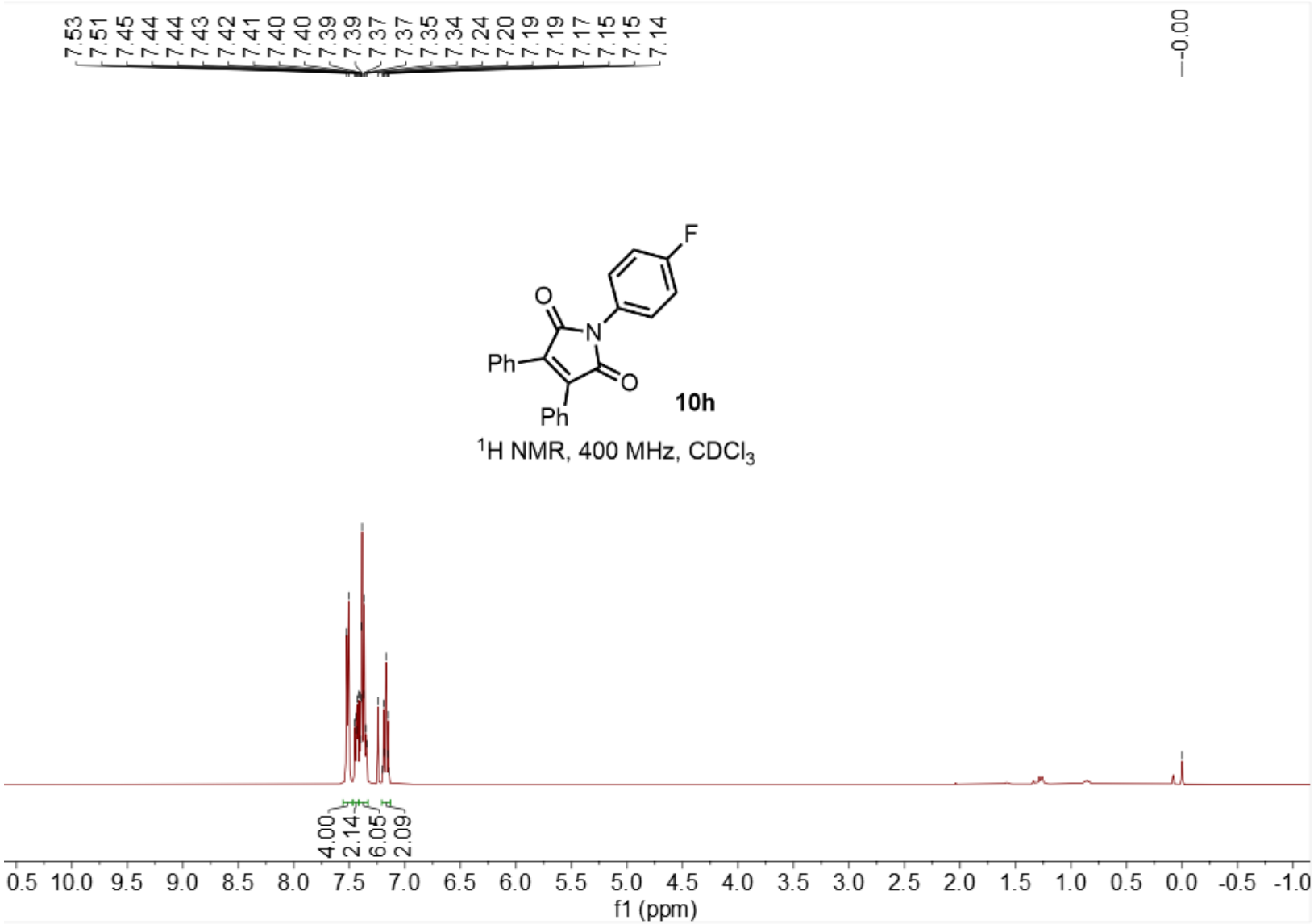
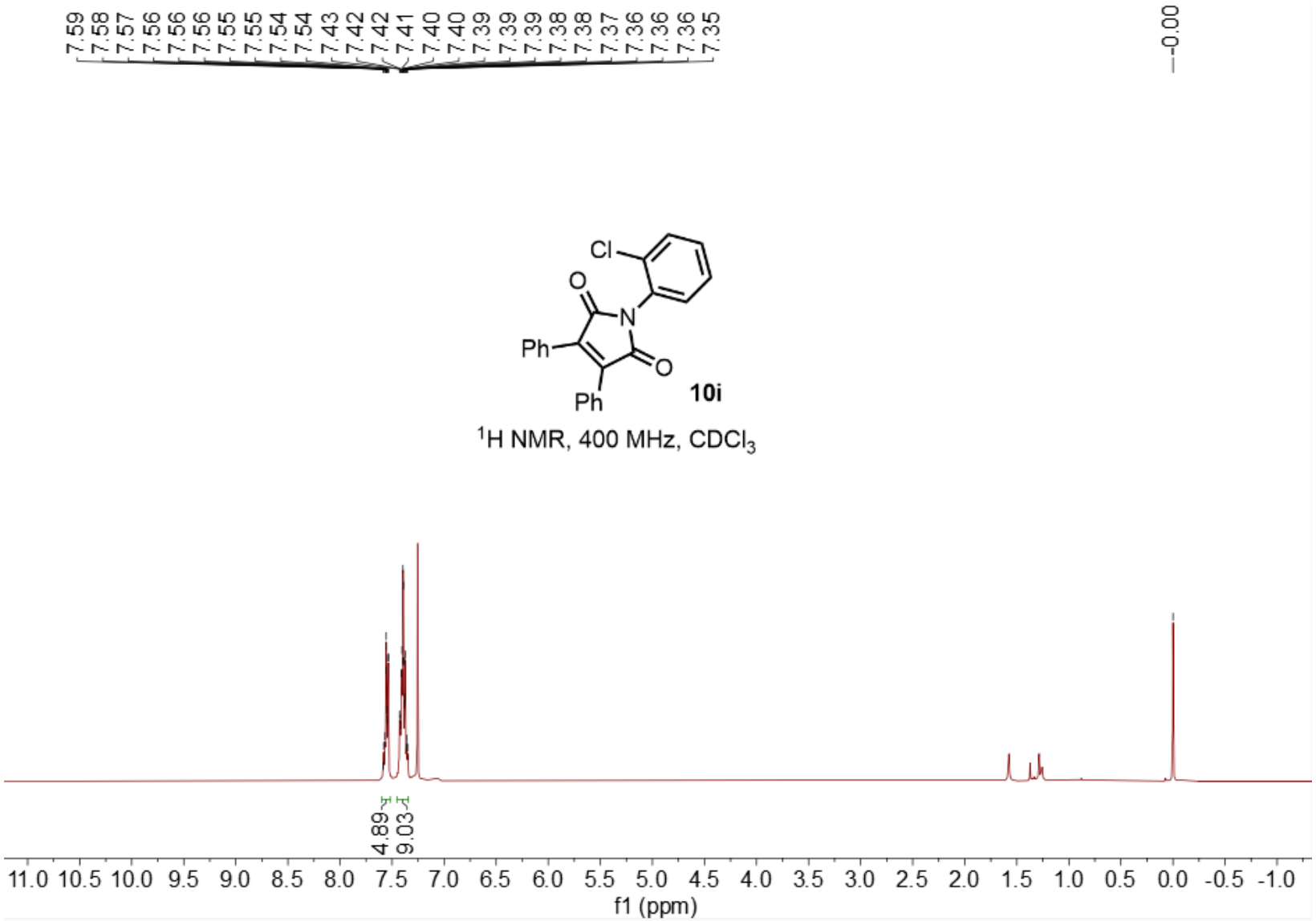
7.53
7.52
7.52
7.51
7.51
7.42
7.42
7.42
7.41
7.41
7.40
7.40
7.40
7.39
7.39
7.38
7.37
7.36
7.36
7.35
7.34
7.34
7.33
7.26
-0.00
OCF3
O
N
Ph
O
Ph
10g
1H NMR, 400 MHz, CDCl3
6.00
8.14
10.5 10.0 9.5 9.0 8.5 8.0 7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 0.5 0.0 -0.5 -1.0
f1 (ppm)


169.4
148.3
136.6
130.5
130.3
130.2
128.8
128.4
127.5
121.9
121.7
119.3
77.5
77.2
76.8
OCF3
O
N
O
Ph
Ph
10g
13C NMR, 100 MHz, CDCl3
200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0
f1 (ppm)


-57.8
OCF3
O
N
Ph
O
Ph
10g
19F NMR, 376 MHz, CDCl3
10 0 -10 -20 -30 -40 -50 -60 -70 -80 -90 -100 -110 -120 -130 -140 -150 -160 -170 -180 -190 -200 -210
f1 (ppm)


7.53
7.51
7.45
7.44
7.44
7.43
7.42
7.41
7.40
7.40
7.39
7.39
7.37
7.37
7.35
7.34
7.24
7.20
7.19
7.19
7.17
7.15
7.15
7.14
0.00
F
O
N
O
Ph
Ph
10h
1H NMR, 400 MHz, CDCl3
4.00
2.14
6.05
2.09
0.5 10.0 9.5 9.0 8.5 8.0 7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 0.5 0.0 -0.5 -1.0
f1 (ppm)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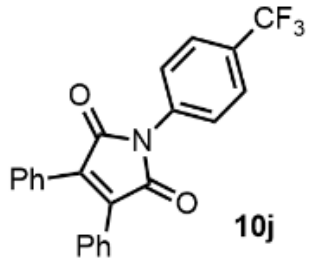
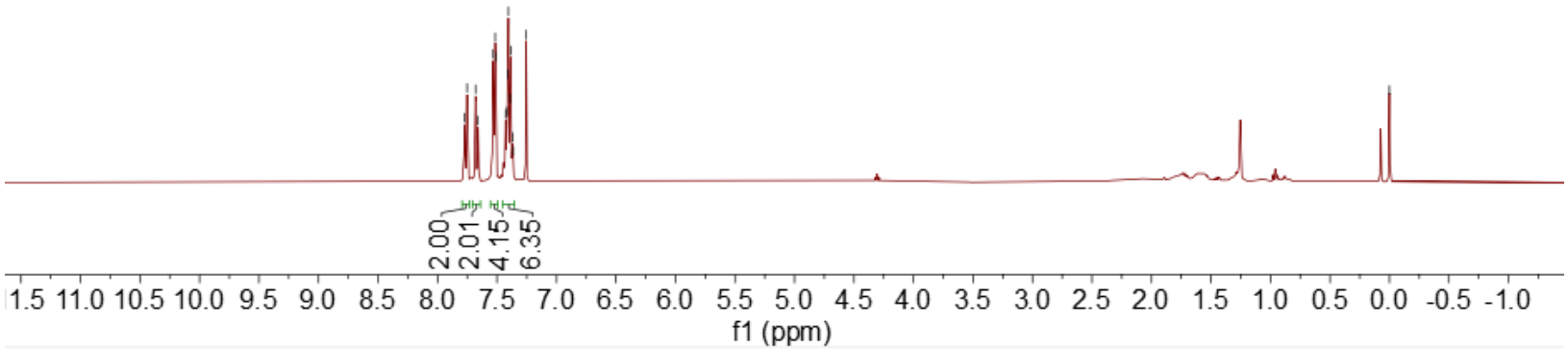
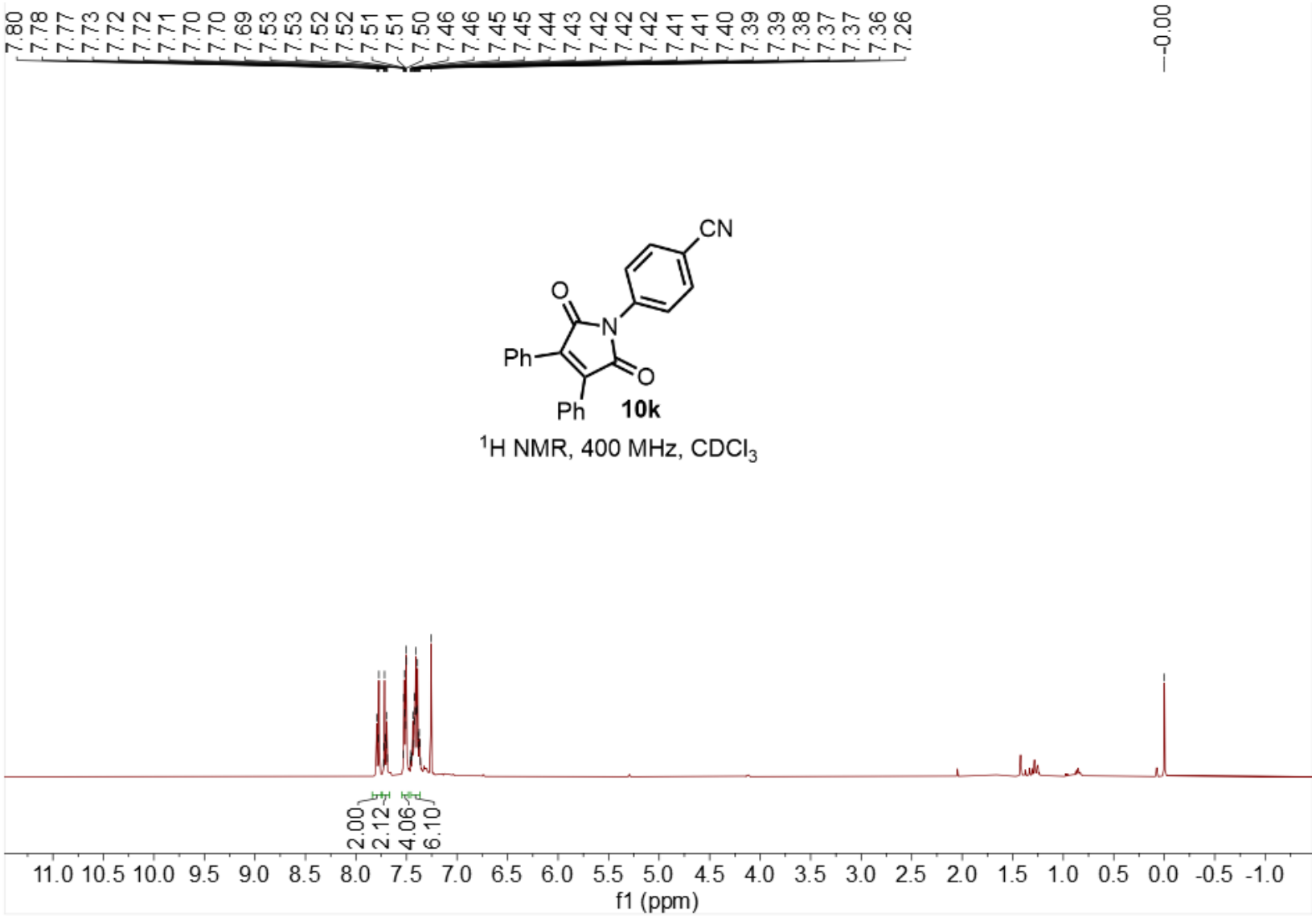
7.58
7.57
7.56
7.56
7.56
7.55
7.55
7.54
7.54
7.43
7.42
7.42
7.41
7.40
7.40
7.39
7.39
7.39
7.38
7.38
7.37
7.36
7.36
7.36
7.35
-0.00
Cl
O
N
Ph
O
Ph
10i
1H NMR, 400 MHz, CDCl3
4.89
9.03
11.0 10.5 10.0 9.5 9.0 8.5 8.0 7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 0.5 0.0 -0.5 -1.0
f1 (ppm)



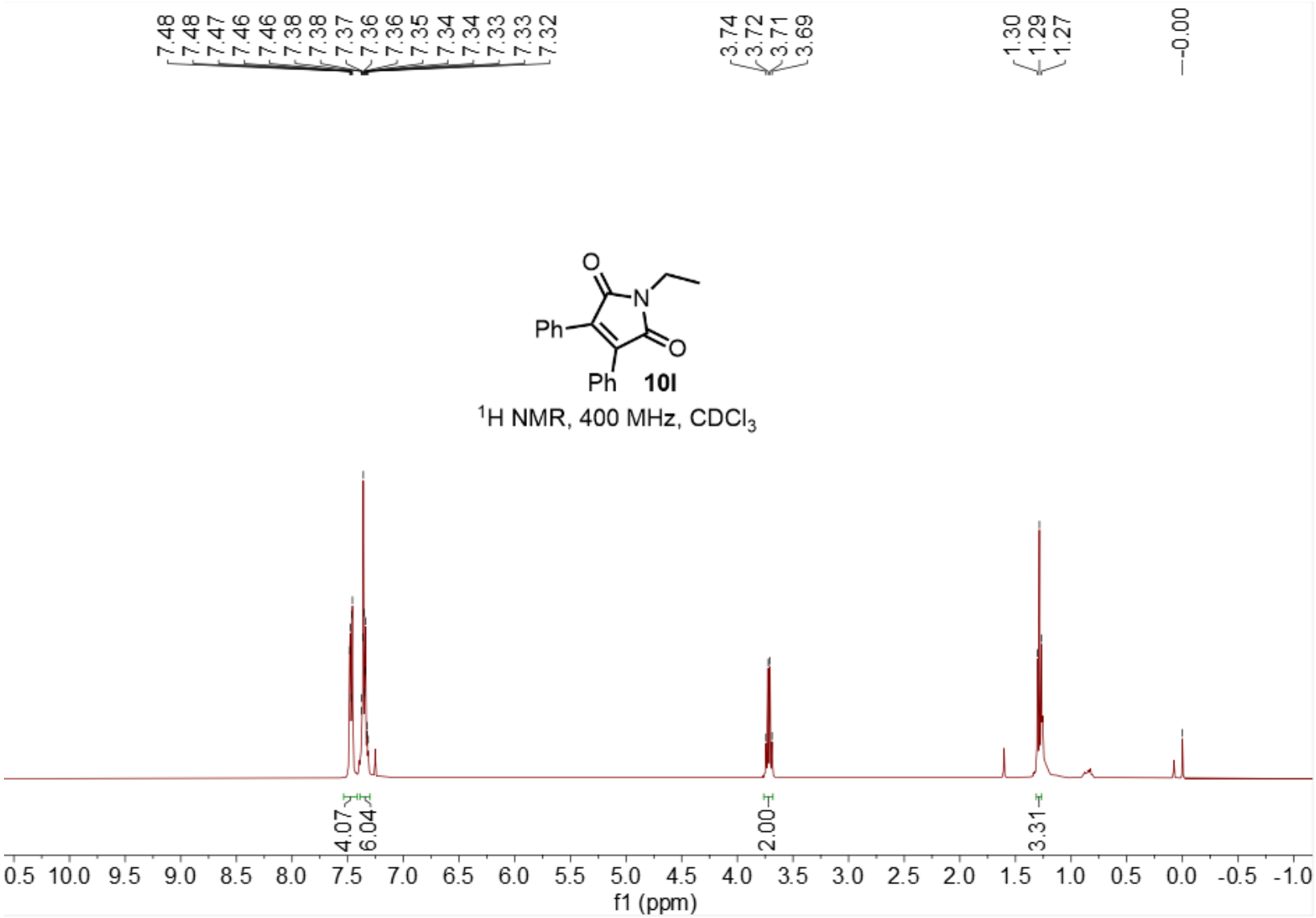
10j

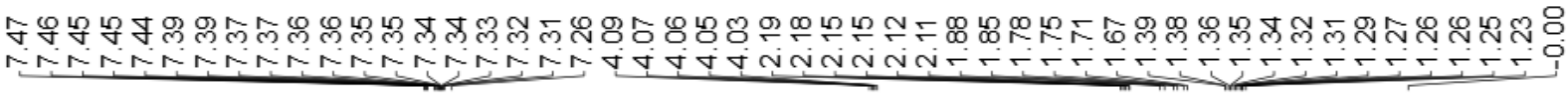
^{1}H NMR, 400 MHz, $CDCl_3$

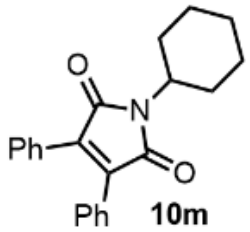




^{1}H NMR, 400 MHz, $CDCl_3$

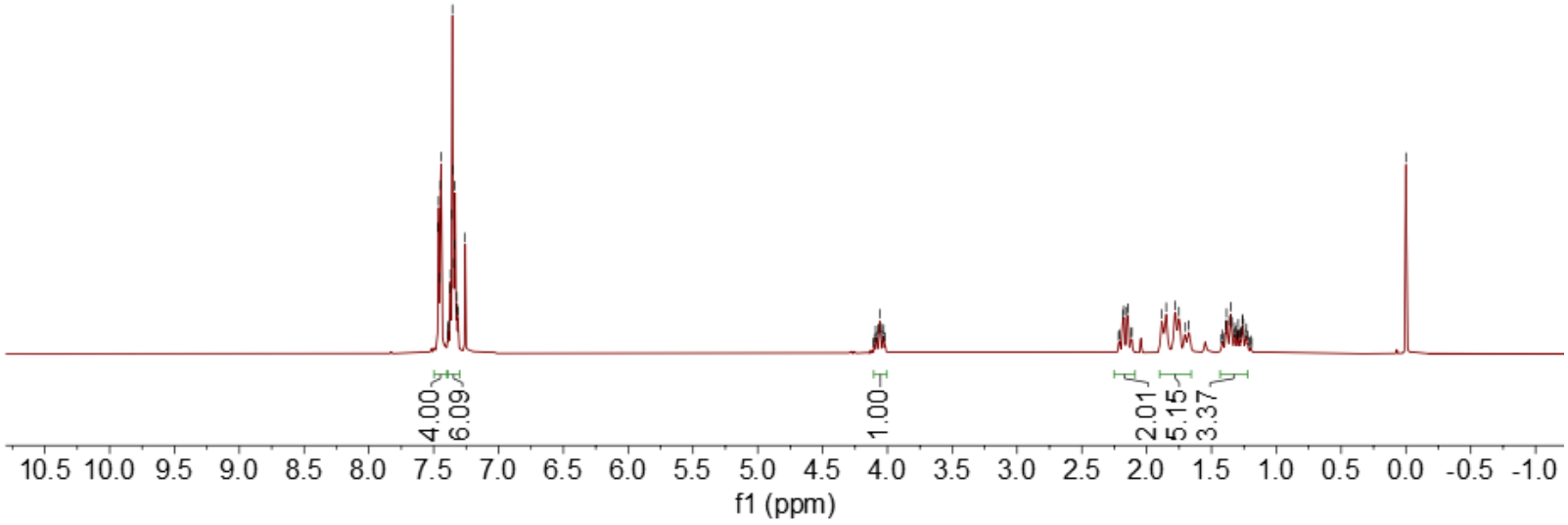

7.48
7.48
7.47
7.46
7.46
7.38
7.38
7.37
7.36
7.36
7.35
7.34
7.34
7.33
7.33
7.32
3.74
3.72
3.71
3.69
1.30
1.29
1.27
0.00
O
N
Ph
O
Ph
10l
1H NMR, 400 MHz, CDCl3
4.07
6.04
2.00
3.31
0.5 10.0 9.5 9.0 8.5 8.0 7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 0.5 0.0 -0.5 -1.0
f1 (ppm)

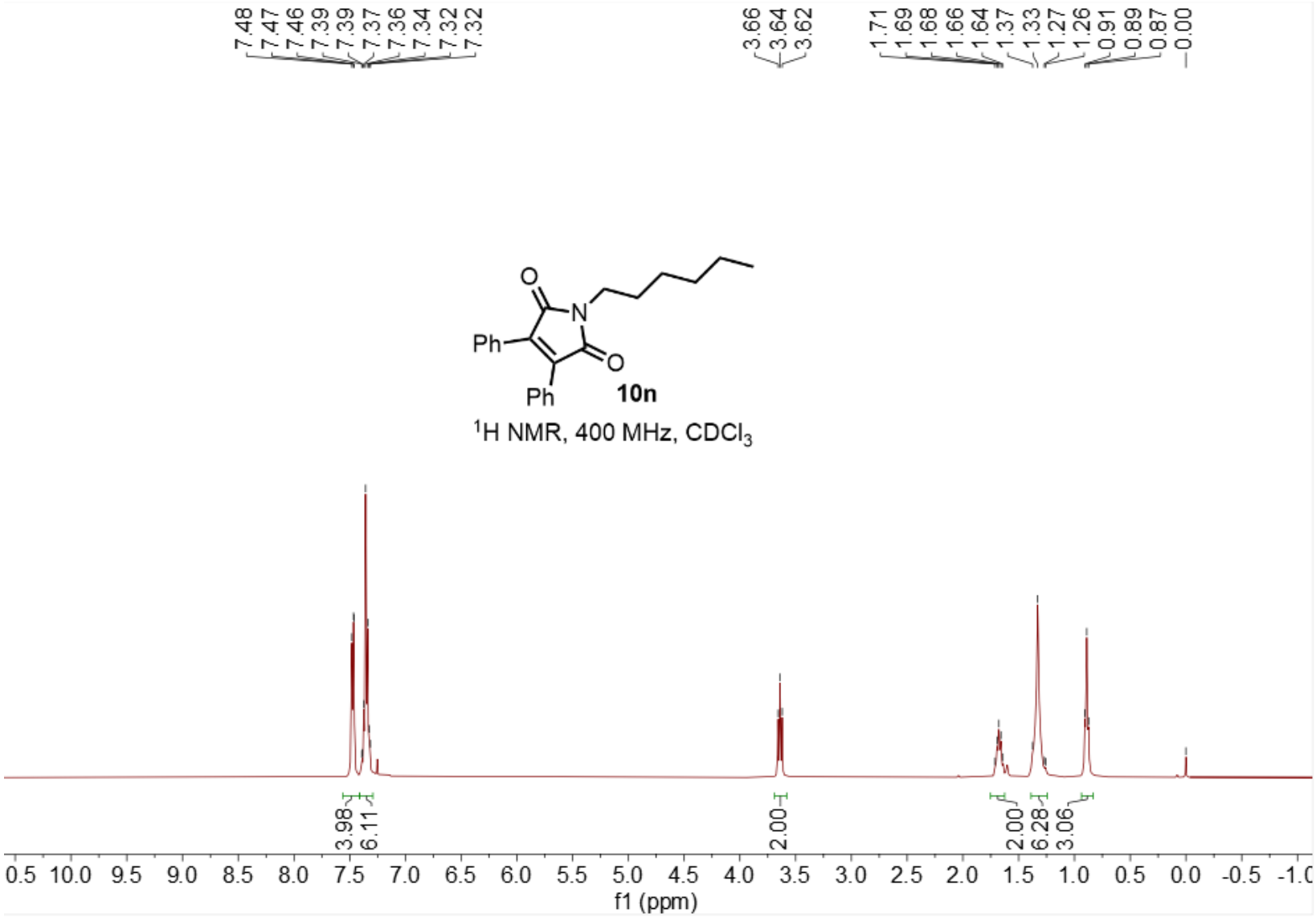


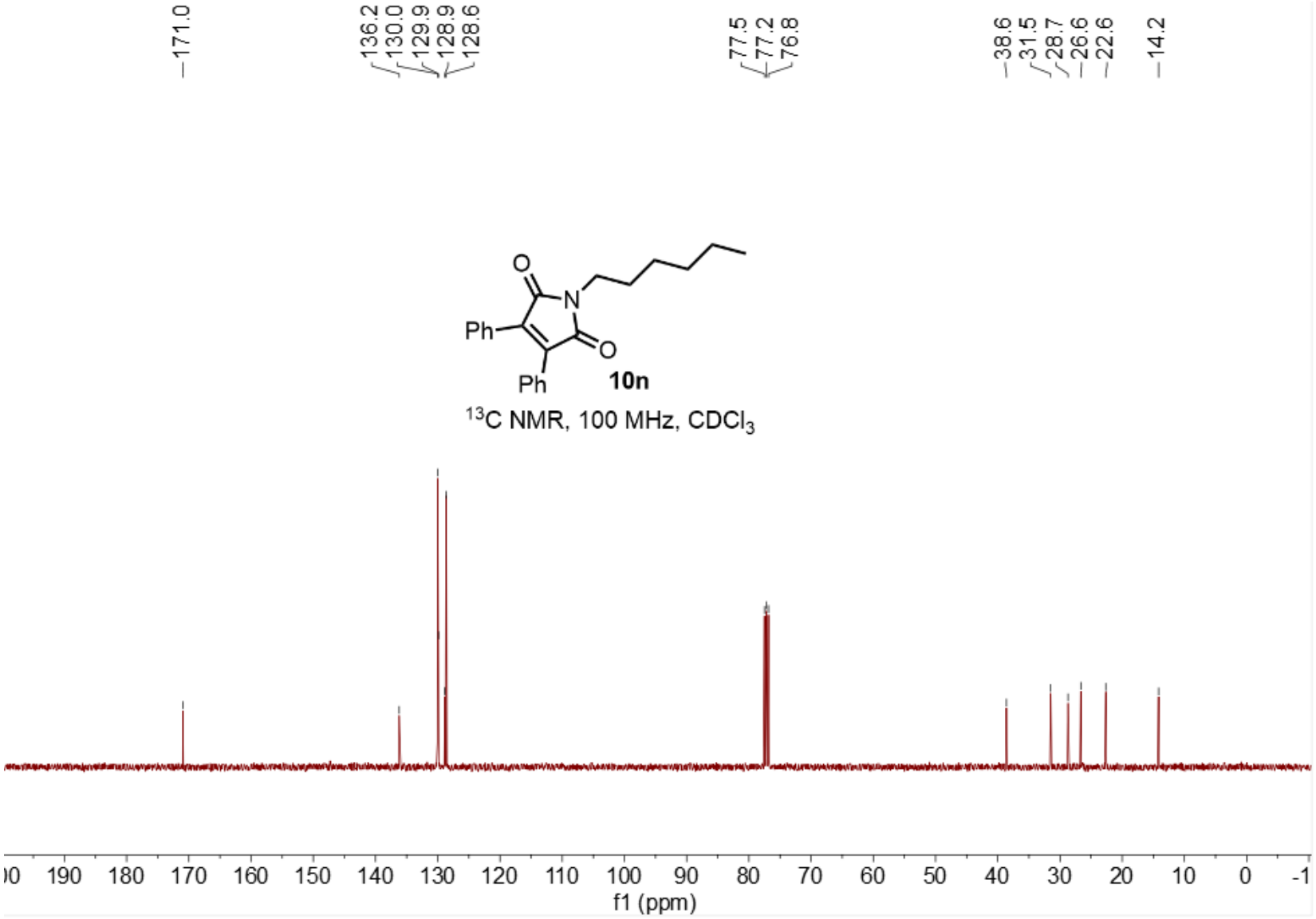


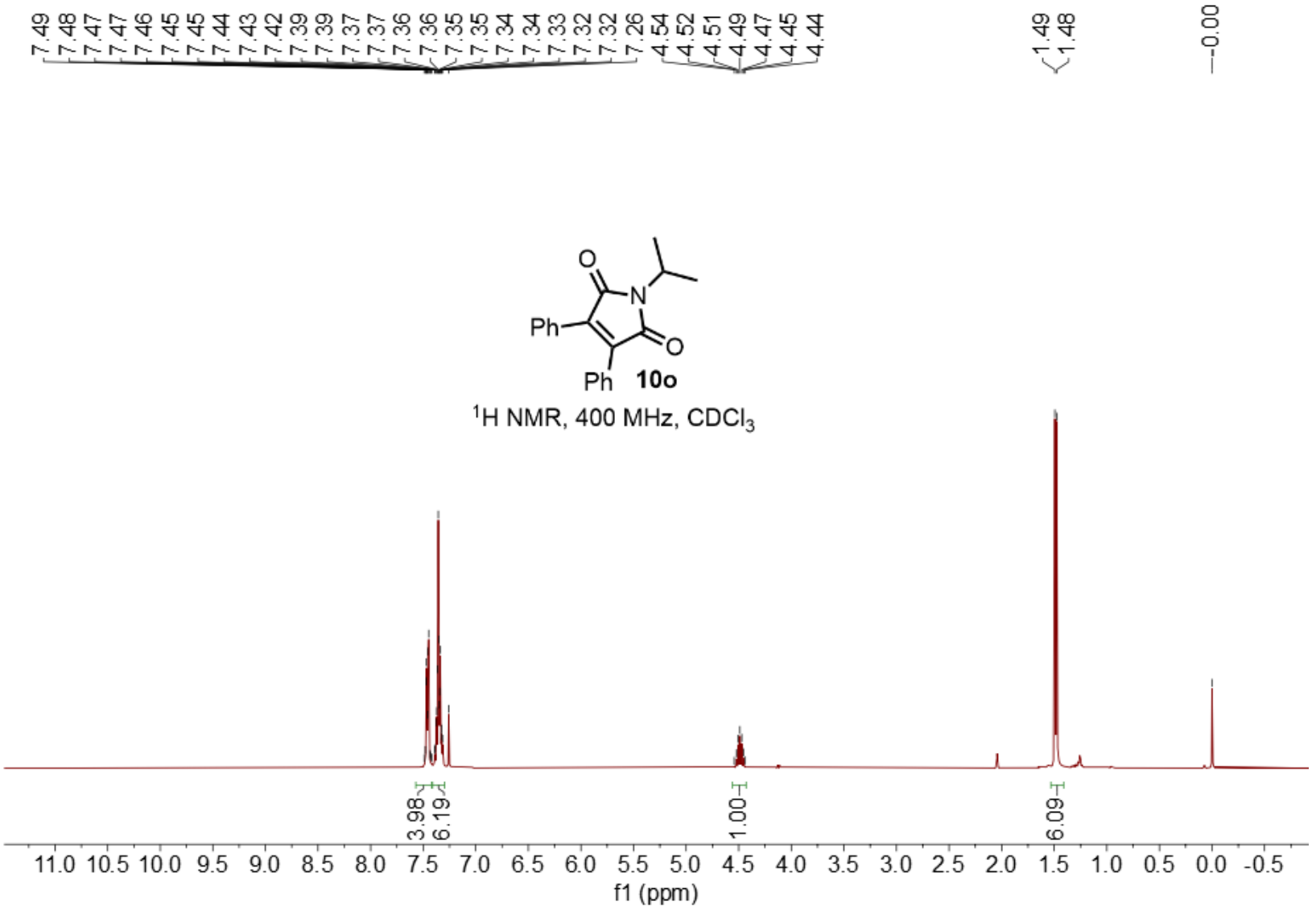
10m

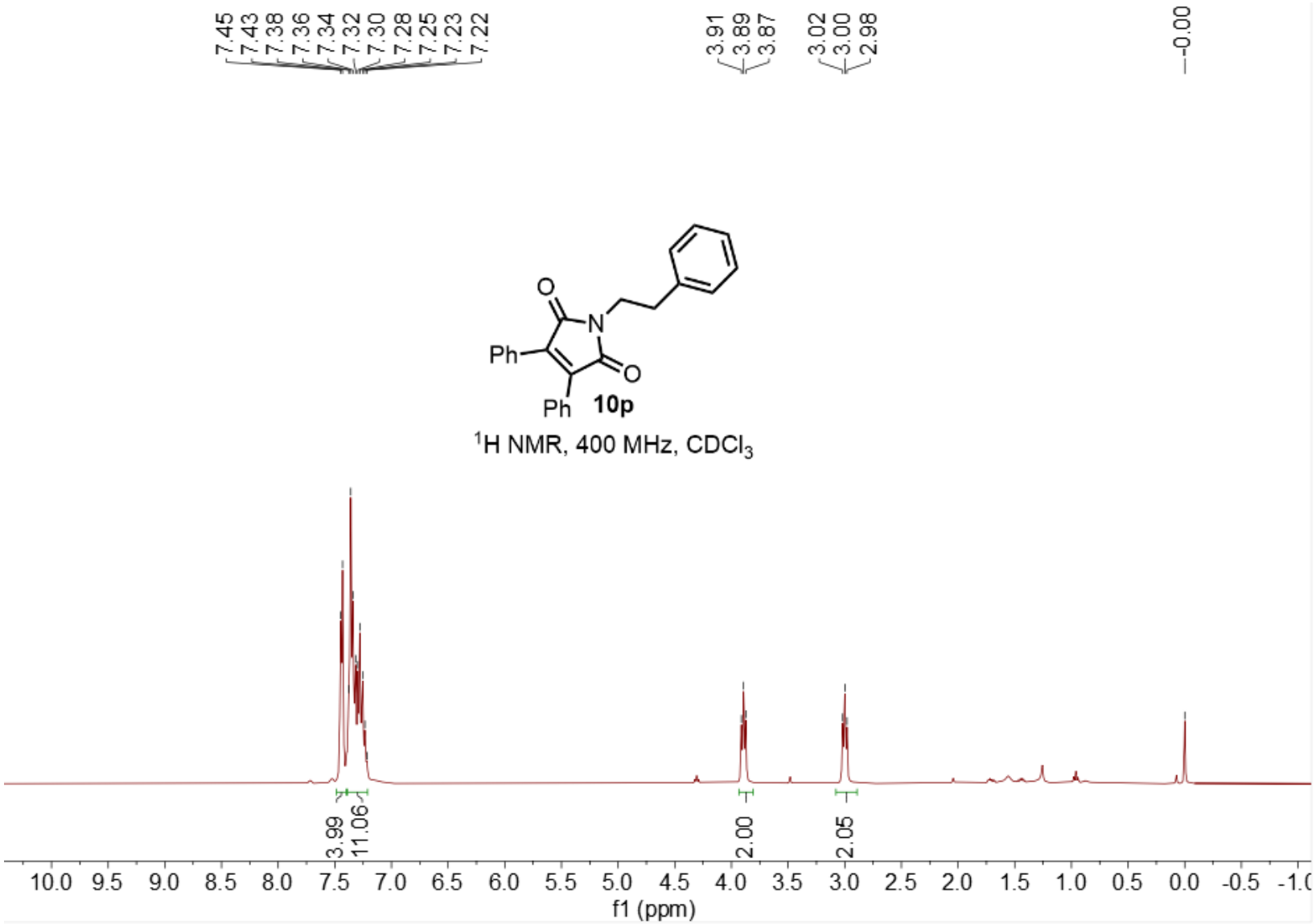
^{1}H NMR, 400 MHz, $CDCl_3$

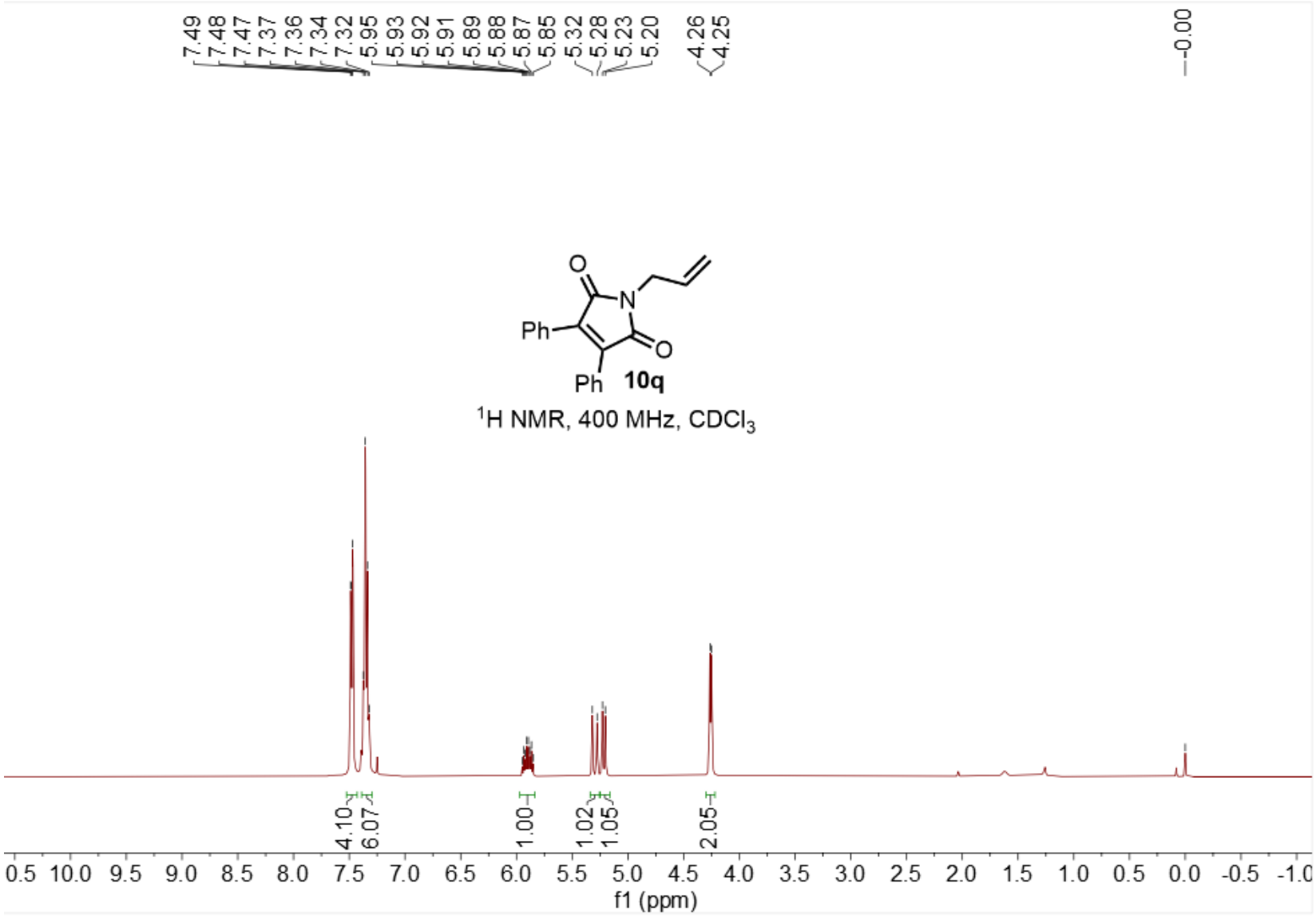



7.48
7.47
7.46
7.39
7.39
7.37
7.36
7.34
7.32
7.32
3.66
3.64
3.62
1.71
1.69
1.68
1.66
1.64
1.37
1.33
1.27
1.26
0.91
0.89
0.87
0.00
O
N
Ph
O
Ph
10n
1H NMR, 400 MHz, CDCl3
3.98
6.11
2.00
2.00
6.28
3.06
f1 (ppm)


171.0
136.2
130.0
129.9
128.9
128.6
77.5
77.2
76.8
38.6
31.5
28.7
26.6
22.6
14.2
O
N
Ph
O
Ph
10n
13C NMR, 100 MHz, CDCl3
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
f1 (ppm)


7.49
7.48
7.47
7.47
7.46
7.45
7.45
7.44
7.43
7.42
7.39
7.39
7.37
7.37
7.36
7.36
7.35
7.35
7.34
7.34
7.33
7.32
7.32
7.26
4.54
4.52
4.51
4.49
4.47
4.45
4.44
1.49
1.48
0.00
O
N
Ph
O
Ph
10o
1H NMR, 400 MHz, CDCl3
3.98
6.19
1.00
6.09
11.0 10.5 10.0 9.5 9.0 8.5 8.0 7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 0.5 0.0 -0.5
f1 (ppm)


7.45
7.43
7.38
7.36
7.34
7.32
7.30
7.28
7.25
7.23
7.22
3.91
3.89
3.87
3.02
3.00
2.98
0.00
O
N
Ph
O
Ph
10p
1H NMR, 400 MHz, CDCl3
3.99
11.06
2.00
2.05
10.0 9.5 9.0 8.5 8.0 7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 0.5 0.0 -0.5
f1 (ppm)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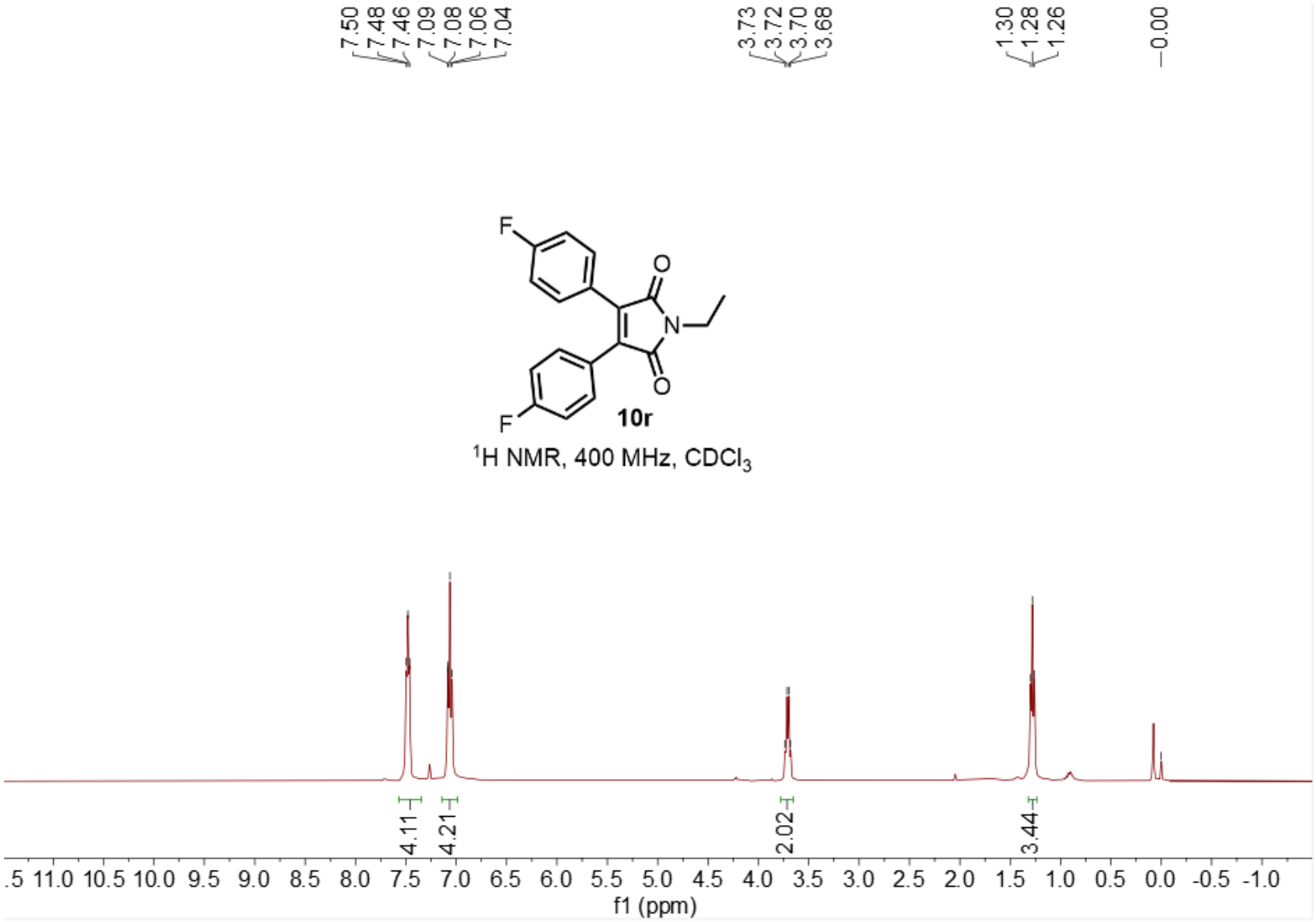
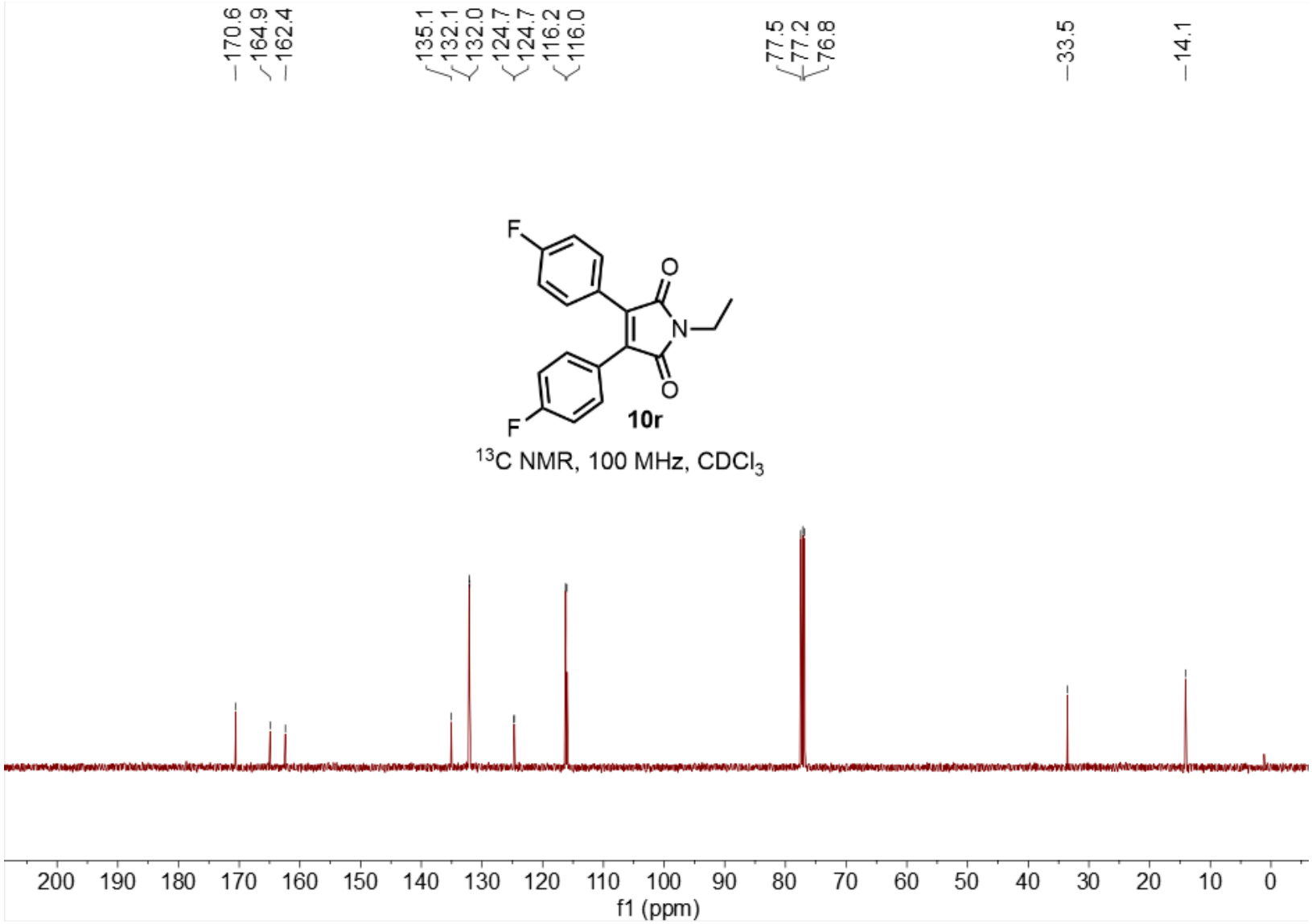
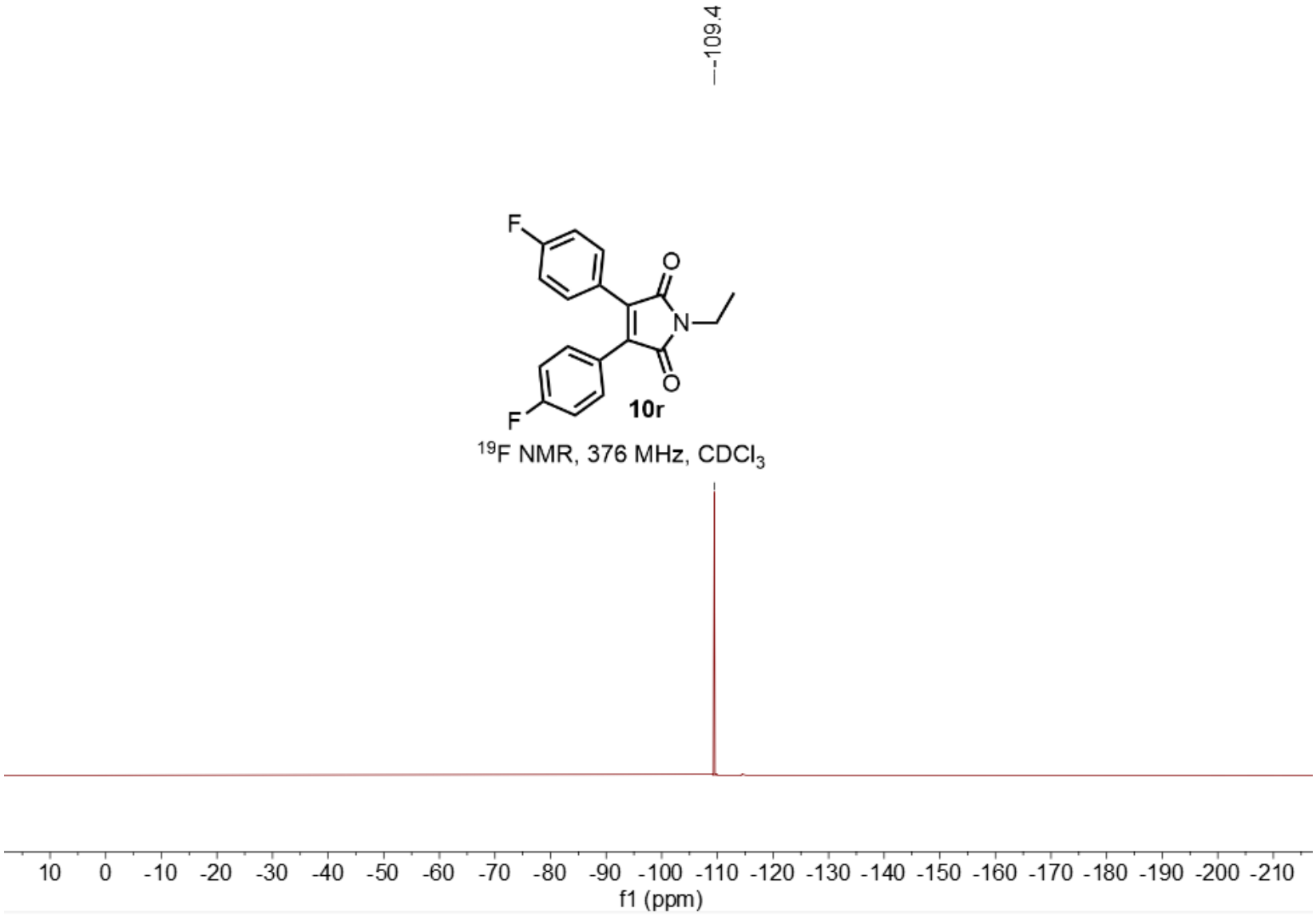
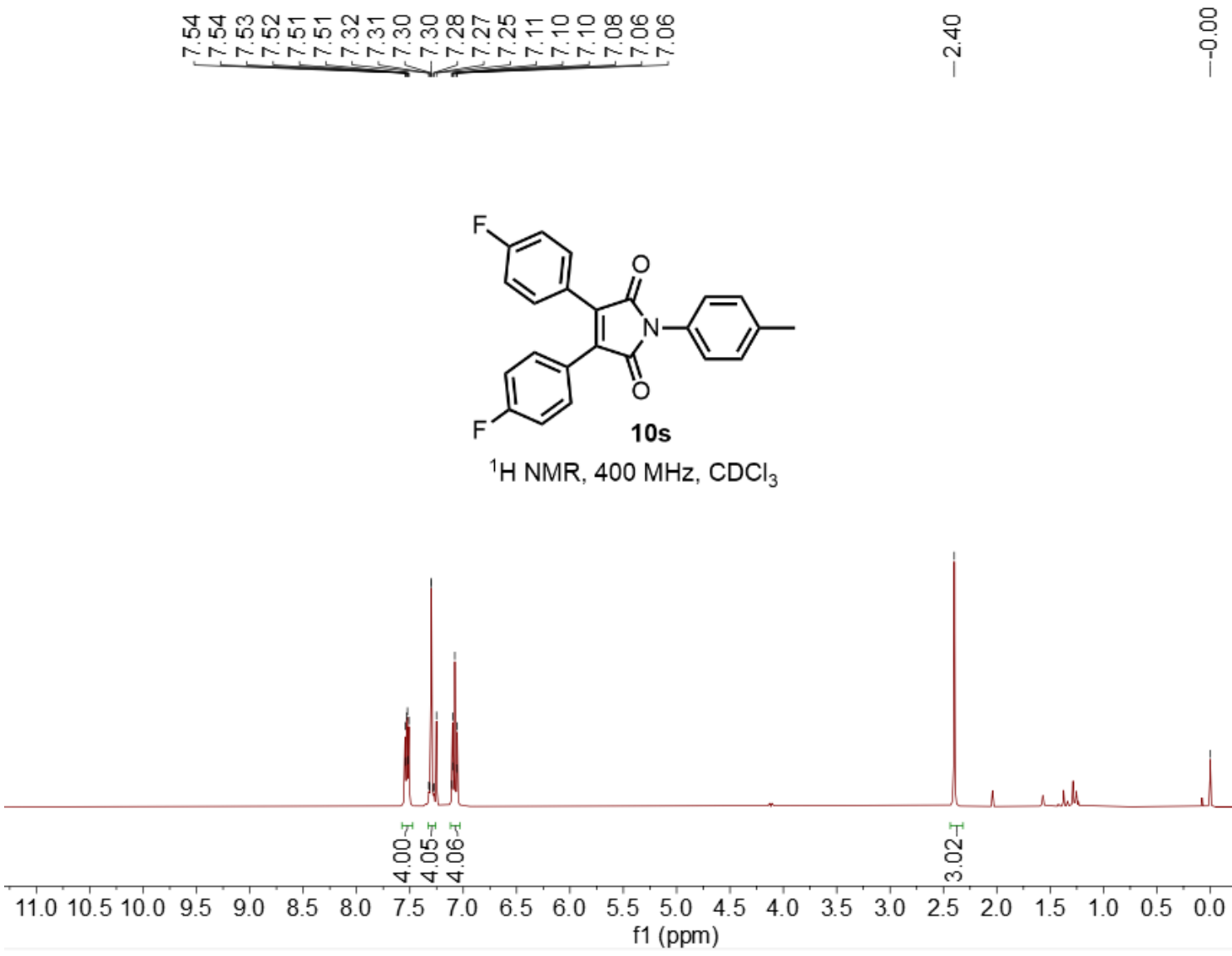
7.48
7.47
7.37
7.36
7.34
7.32
5.95
5.93
5.92
5.91
5.89
5.88
5.87
5.85
5.32
5.28
5.23
5.20
4.26
4.25
0.00
O
N
O
Ph
Ph
10q
1H NMR, 400 MHz, CDCl3
4.10
6.07
1.00
1.02
1.05
2.05
f1 (ppm)



^{1}H NMR, 400 MHz, $CDCl_3$


170.6
164.9
162.4
135.1
132.1
132.0
124.7
124.7
116.2
116.0
77.5
77.2
76.8
33.5
14.1
F
O
N
O
F
10r
13C NMR, 100 MHz, CDCl3
200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0
f1 (ppm)


-109.4
F
O
N
O
F
10r
^{19}F NMR, 376 MHz, $CDCl_3$
10 0 -10 -20 -30 -40 -50 -60 -70 -80 -90 -100 -110 -120 -130 -140 -150 -160 -170 -180 -190 -200 -210
f1 (ppm)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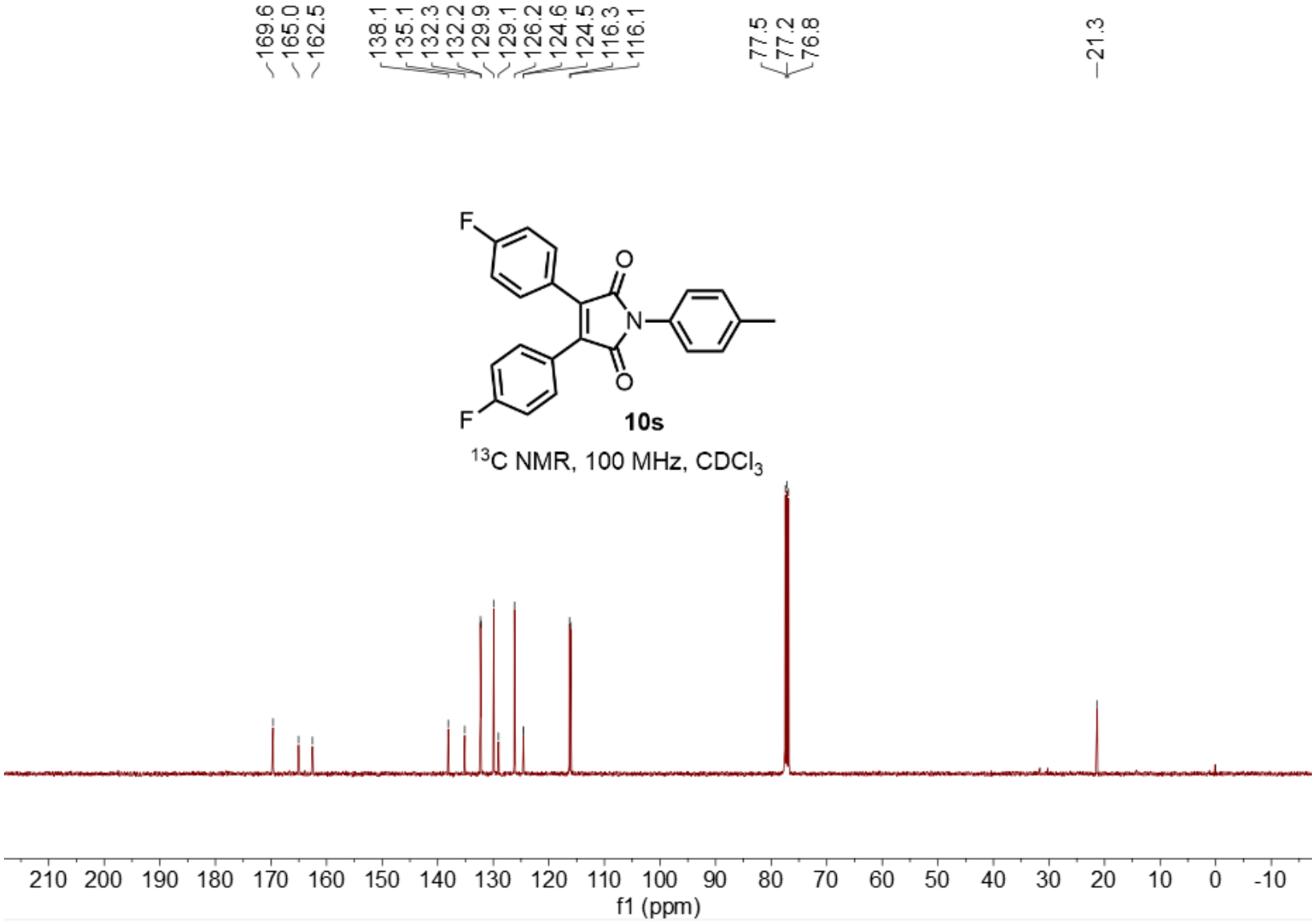
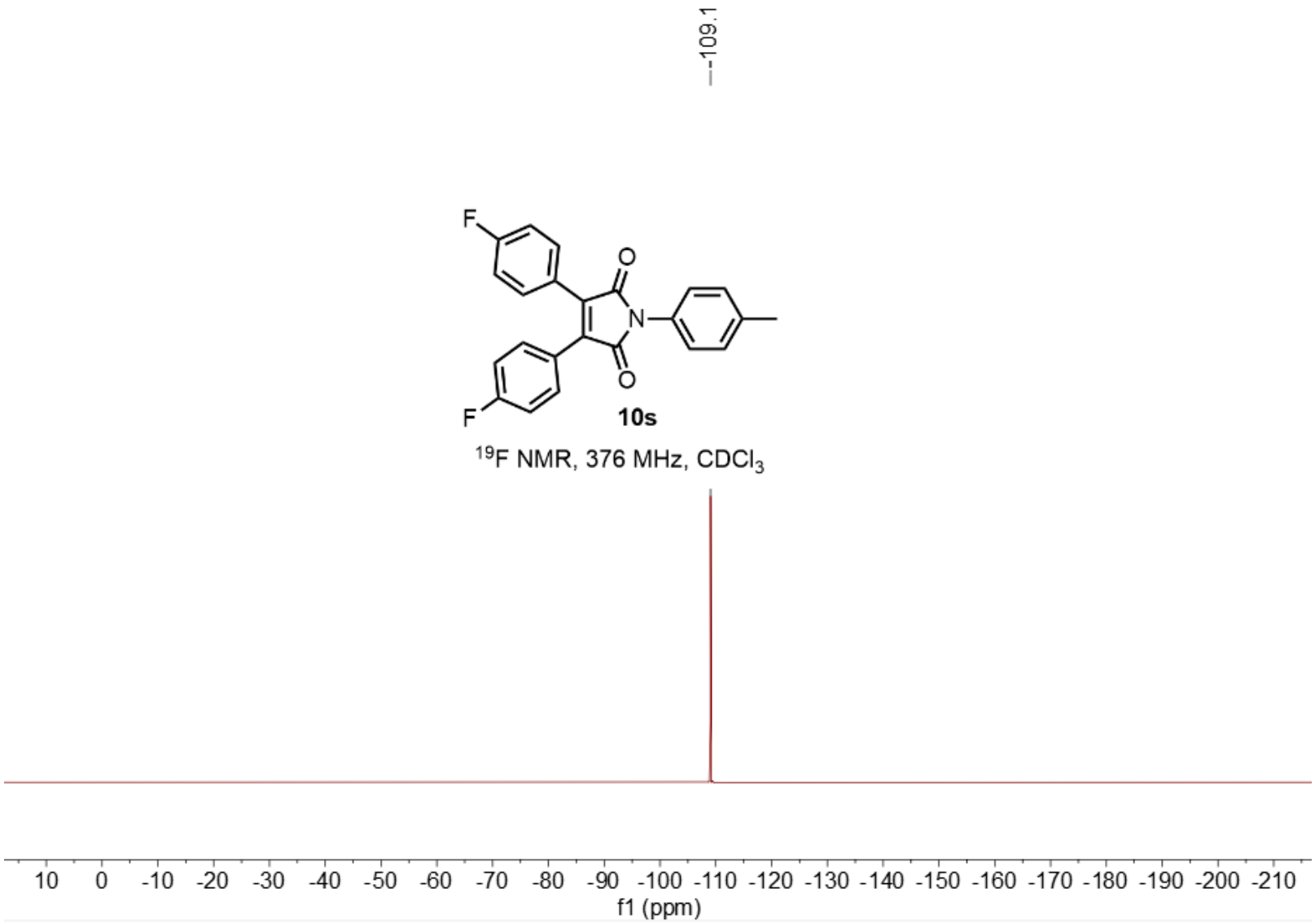
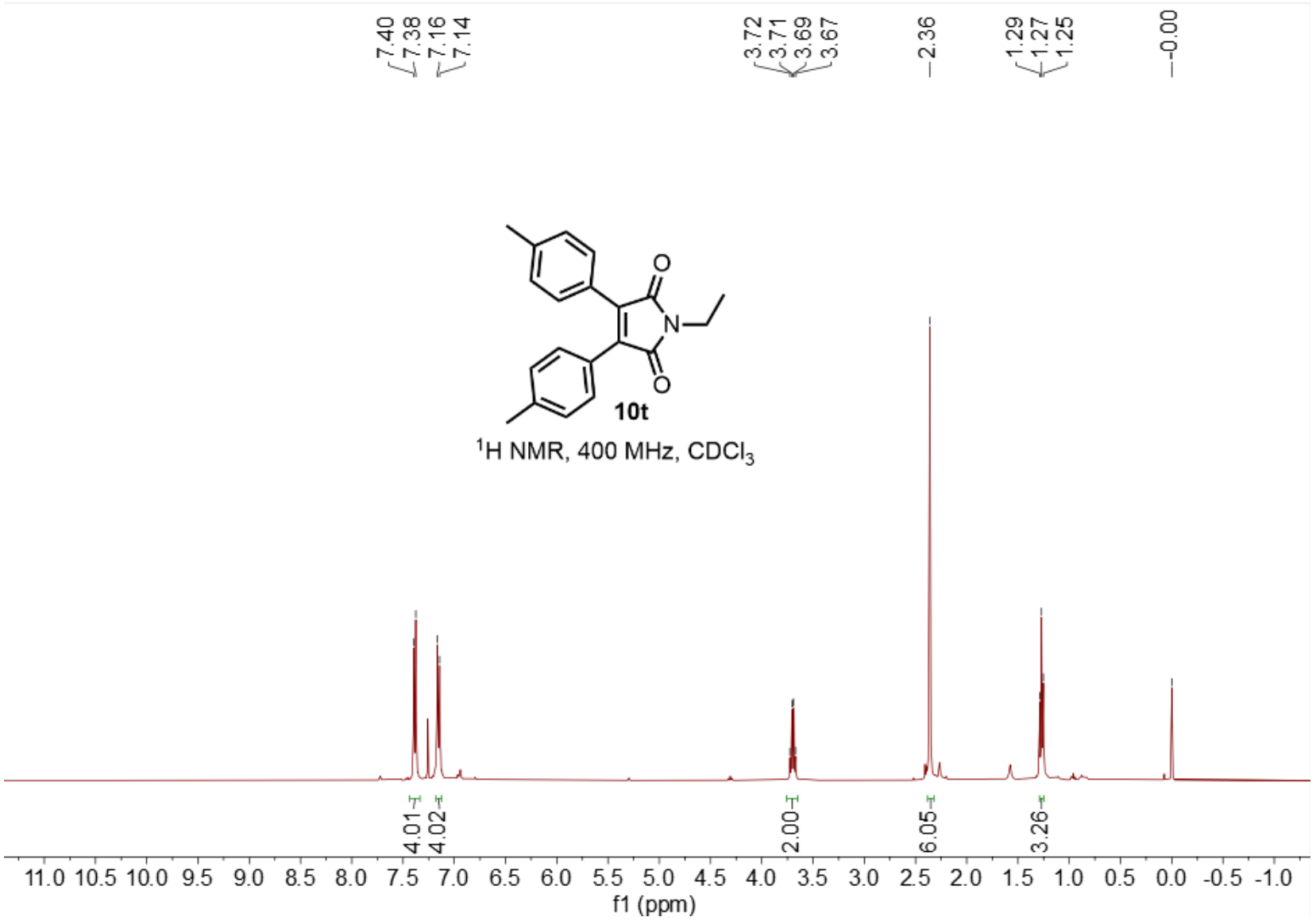
7.54
7.53
7.52
7.51
7.51
7.32
7.31
7.30
7.30
7.28
7.27
7.25
7.11
7.10
7.10
7.08
7.06
7.06
2.40
0.00
F
O
N
O
F
10s
1H NMR, 400 MHz, CDCl3
4.00
4.05
4.06
3.02
11.0 10.5 10.0 9.5 9.0 8.5 8.0 7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 0.5 0.0 -0.5 -1.0
f1 (ppm)


169.6
165.0
162.5
138.1
135.1
132.3
132.2
129.9
129.1
126.2
124.6
124.5
116.3
116.1
77.5
77.2
76.8
21.3
F
O
N
O
F
10s
^{13}C NMR, 100 MHz, $CDCl_3$
210 200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0 -10
f1 (ppm)


-109.1
F
O
N
O
F
10s
^{19}F NMR, 376 MHz, $CDCl_3$
10 0 -10 -20 -30 -40 -50 -60 -70 -80 -90 -100 -110 -120 -130 -140 -150 -160 -170 -180 -190 -200 -210
f1 (ppm)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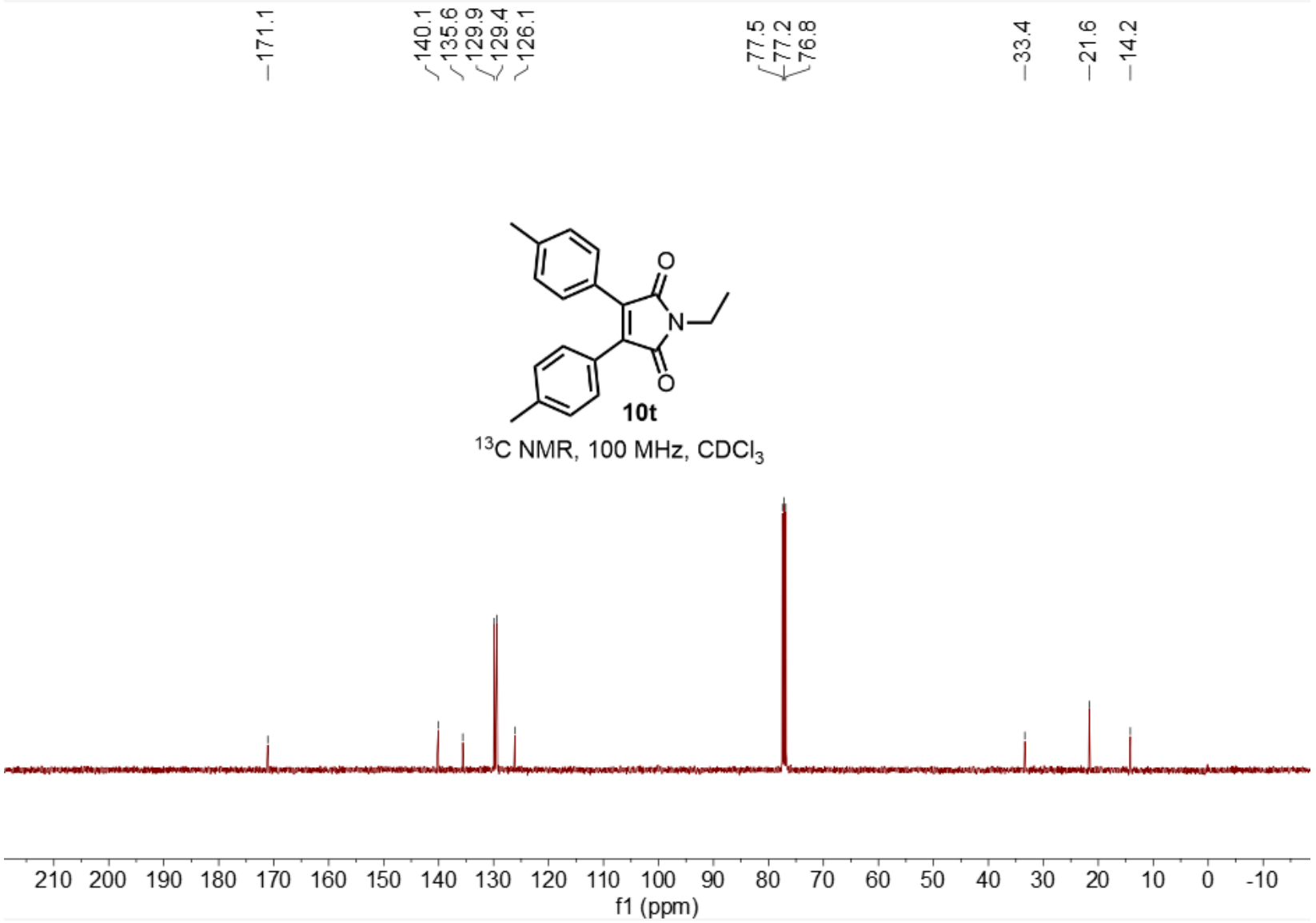
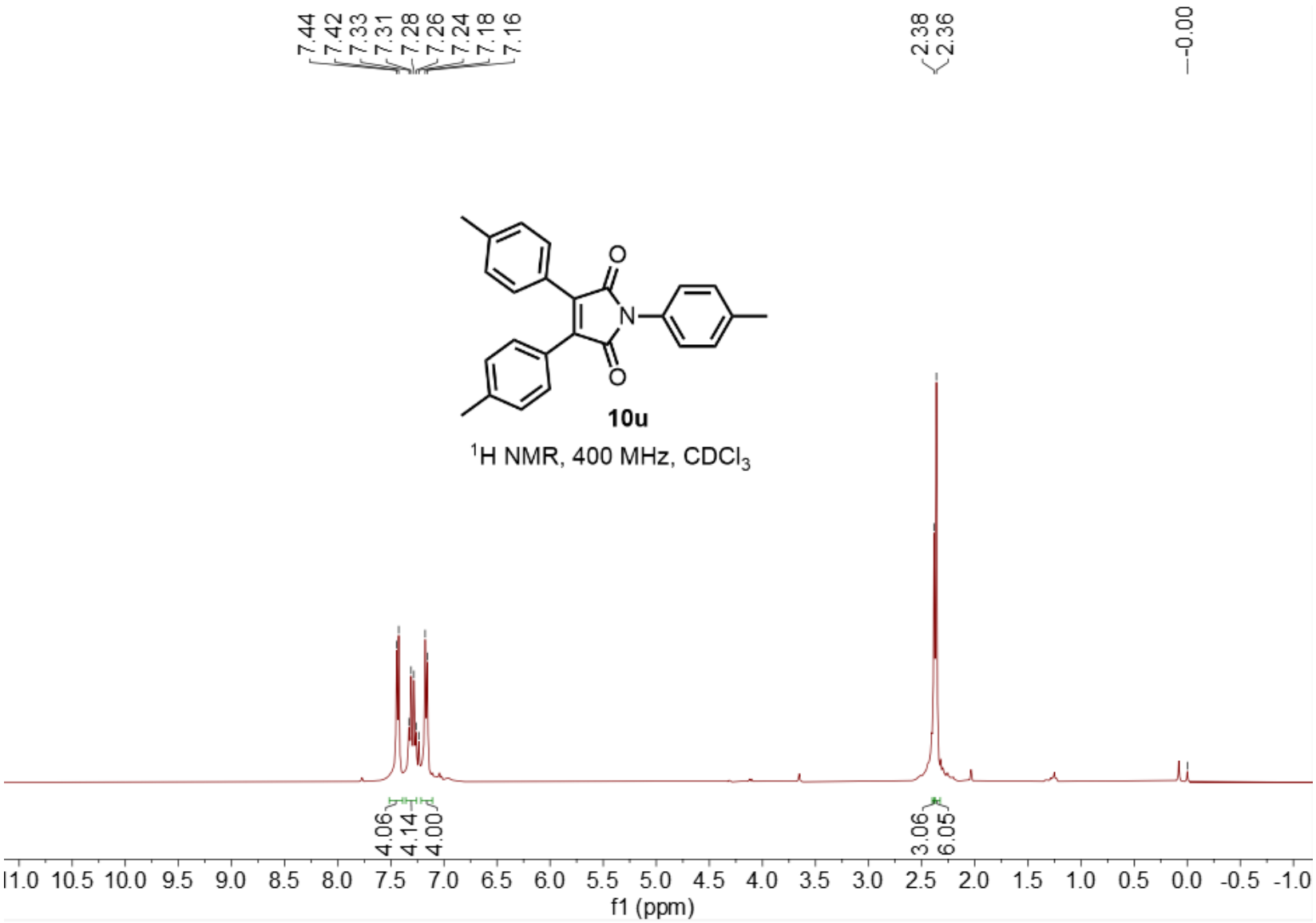
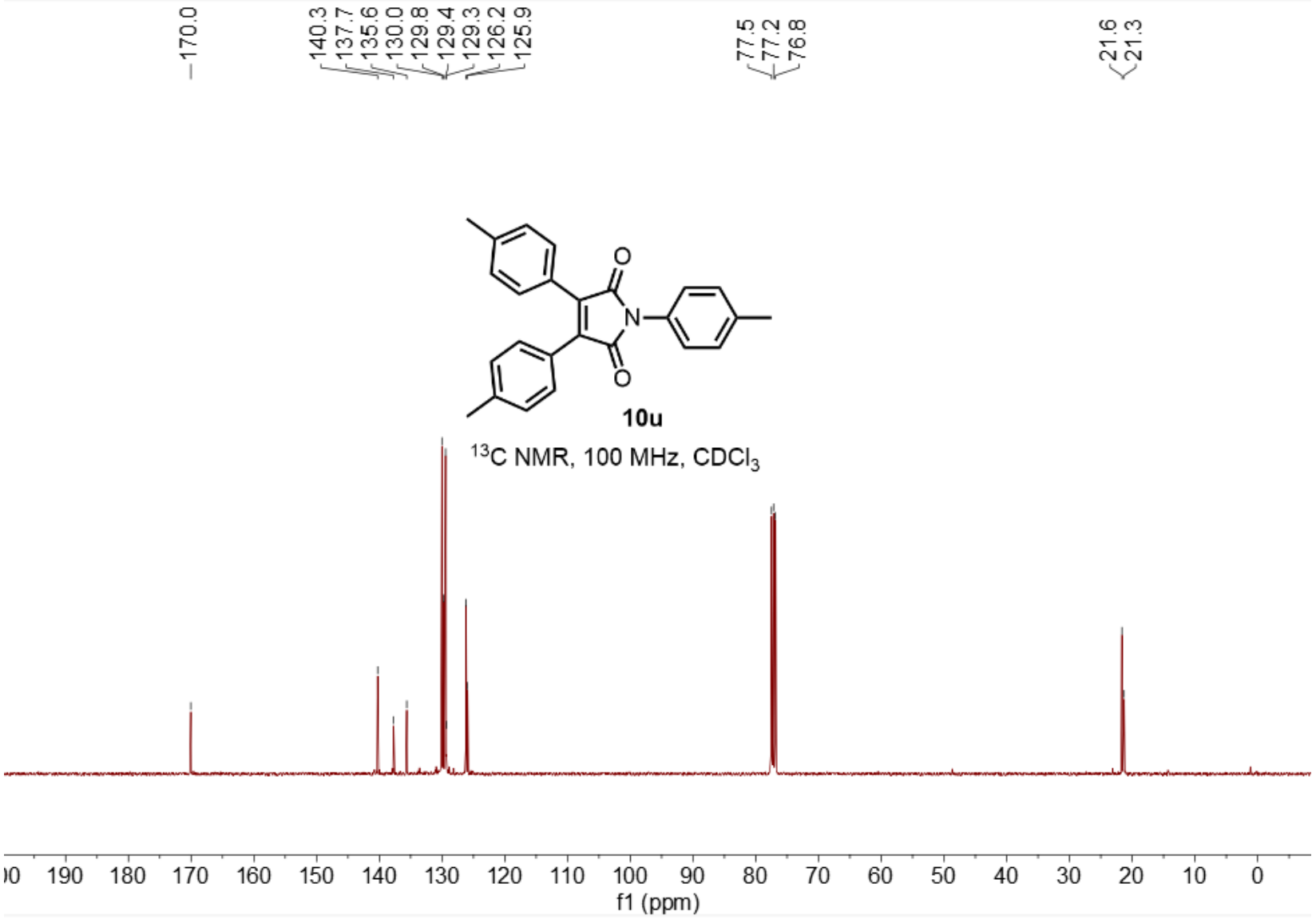
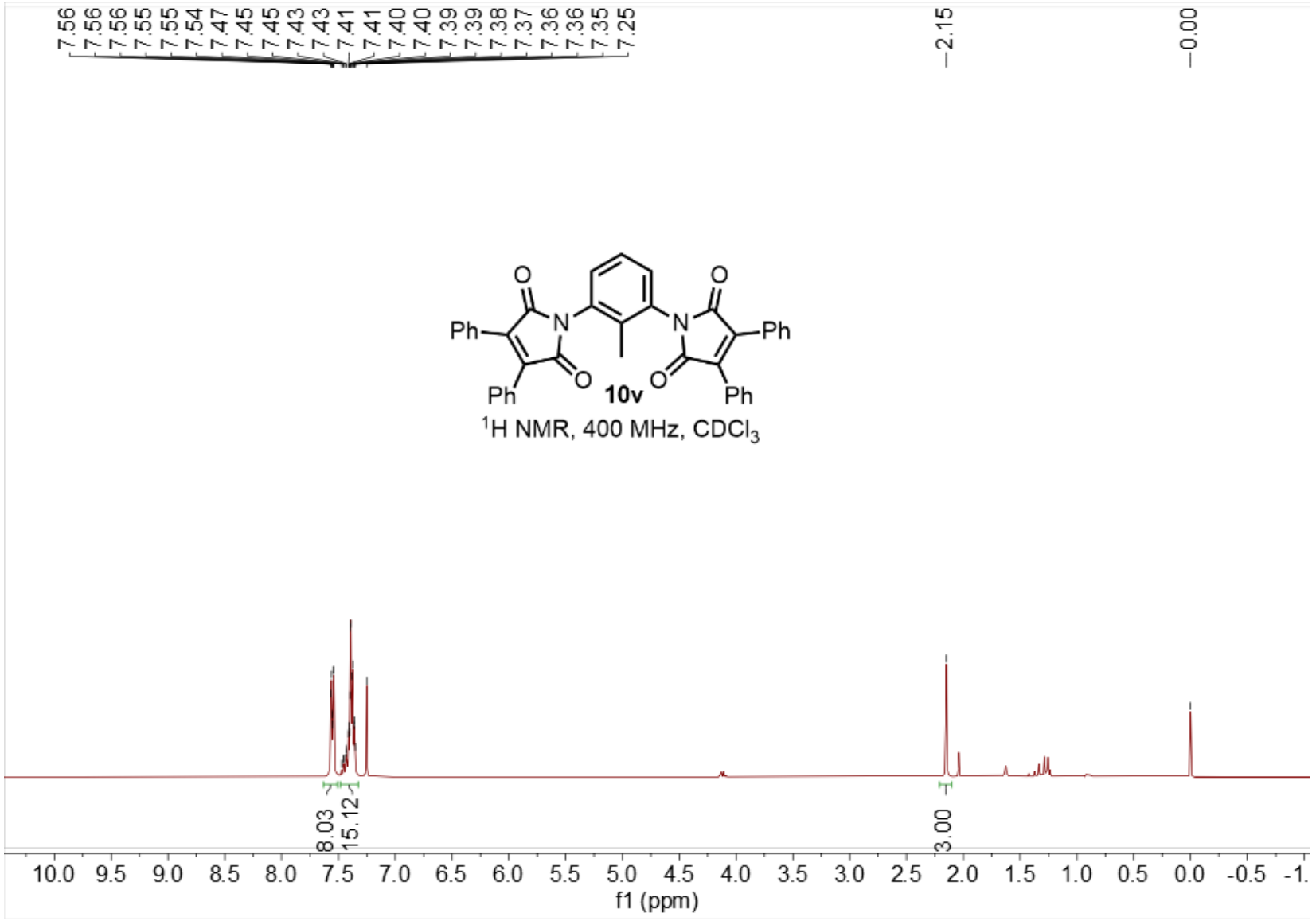
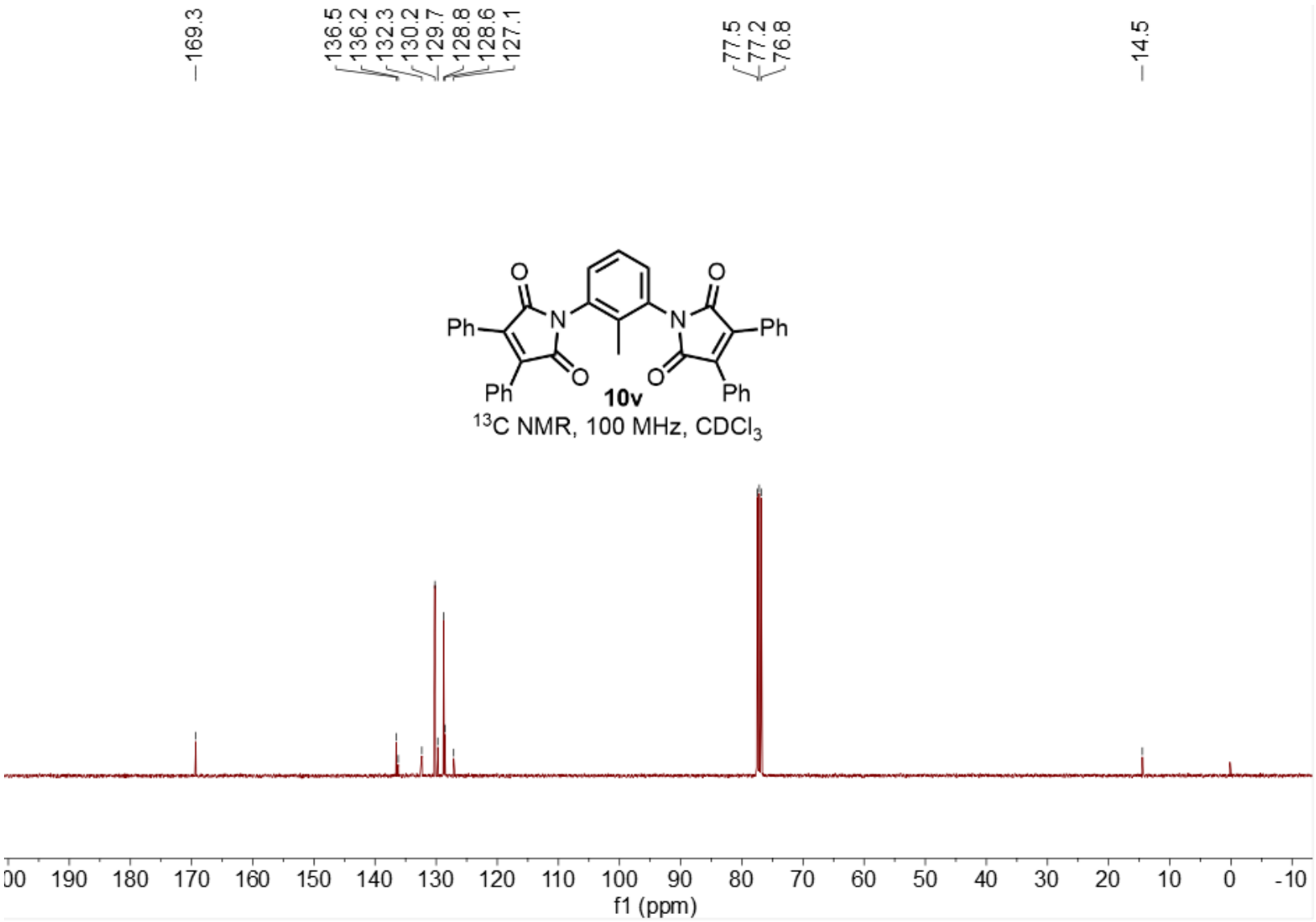
7.38
7.16
7.14
3.72
3.71
3.69
3.67
2.36
1.29
1.27
1.25
0.00
O
N
O
10t
^{1}H NMR, 400 MHz, $CDCl_3$
4.01
4.02
2.00
6.05
3.26
11.0 10.5 10.0 9.5 9.0 8.5 8.0 7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 0.5 0.0 -0.5 -1.0
f1 (ppm)

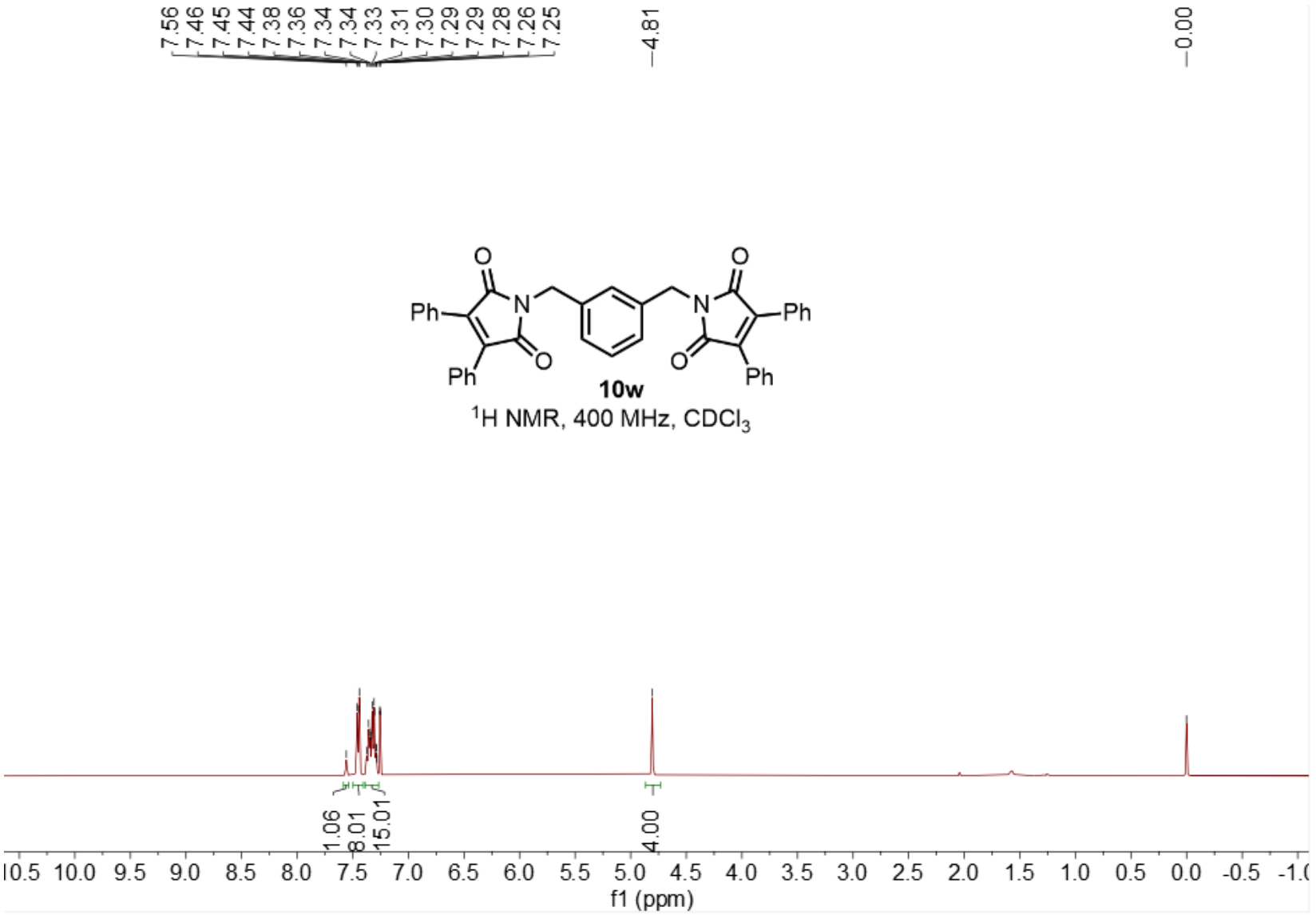

171.1
140.1
135.6
129.9
129.4
126.1
77.5
77.2
76.8
33.4
21.6
14.2
O
N
O
10t
13C NMR, 100 MHz, CDCl3
210 200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0 -10
f1 (ppm)

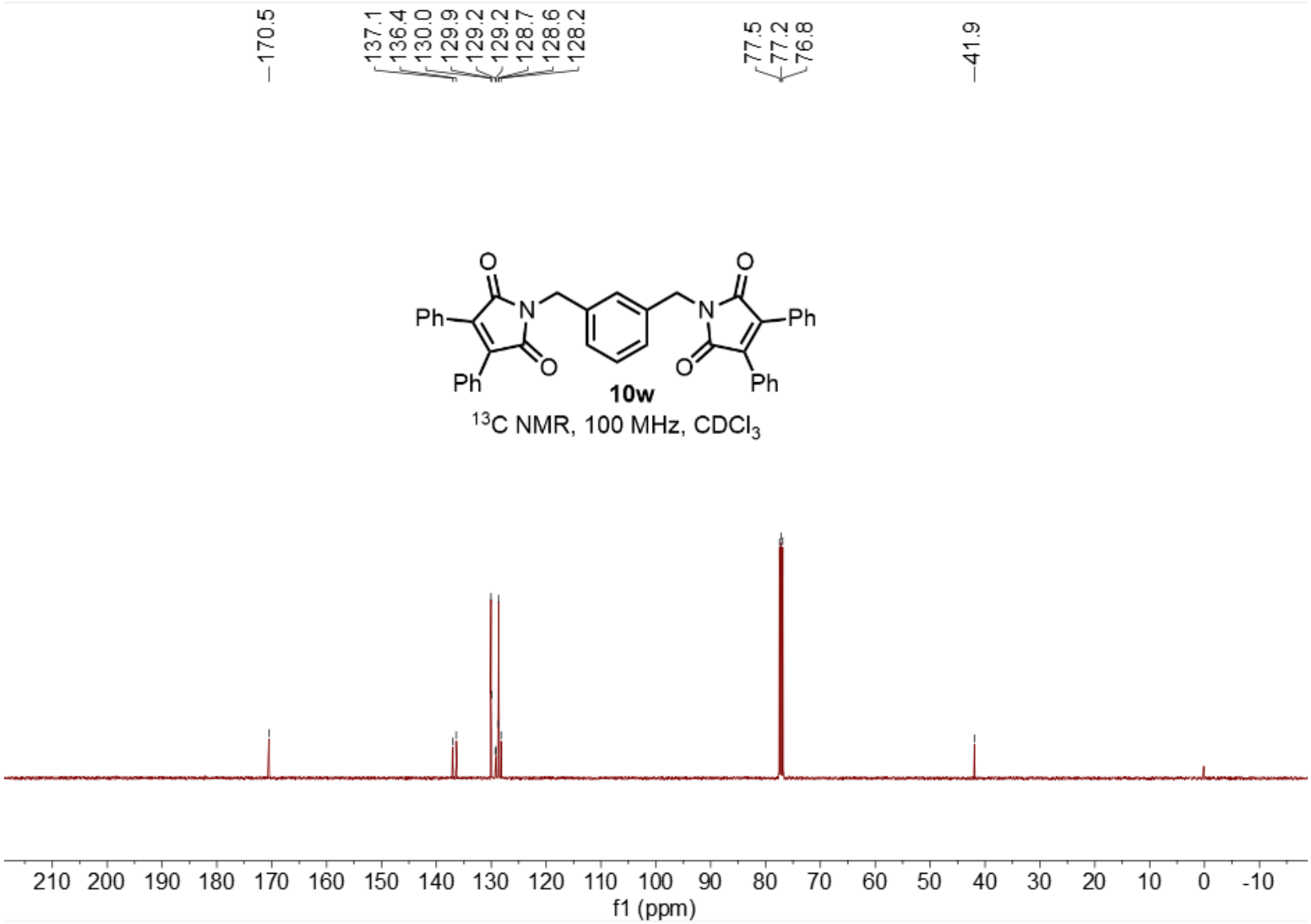

7.44
7.42
7.33
7.31
7.28
7.26
7.24
7.18
7.16
2.38
2.36
0.00
O
N
O
10u
1H NMR, 400 MHz, CDCl3
4.06
4.14
4.00
3.06
6.05
11.0 10.5 10.0 9.5 9.0 8.5 8.0 7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 0.5 0.0 -0.5 -1.0
f1 (ppm)

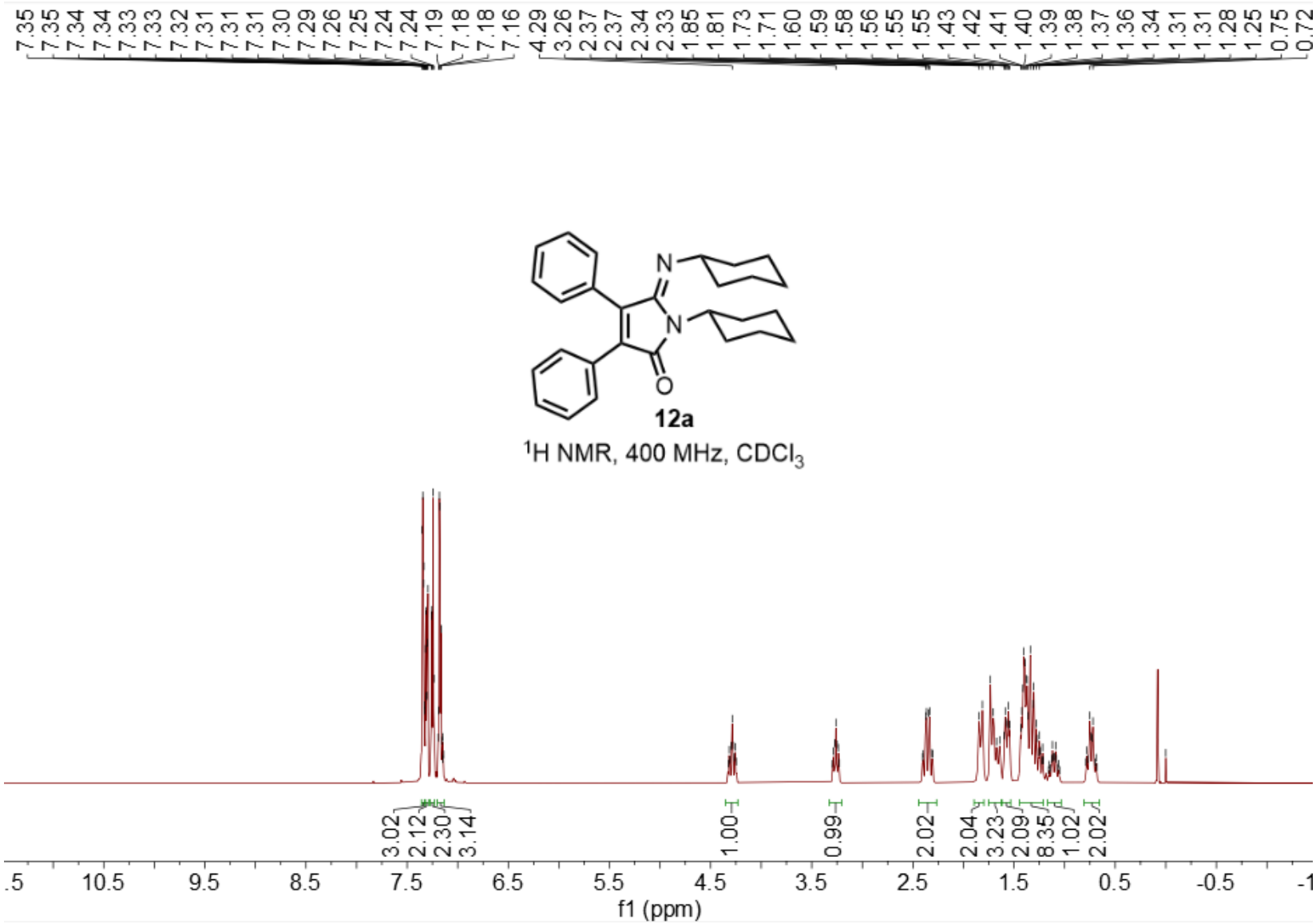

170.0
140.3
137.7
135.6
130.0
129.8
129.4
129.3
126.2
125.9
77.5
77.2
76.8
21.6
21.3
O
N
O
10u
13C NMR, 100 MHz, CDCl3
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
f1 (ppm)

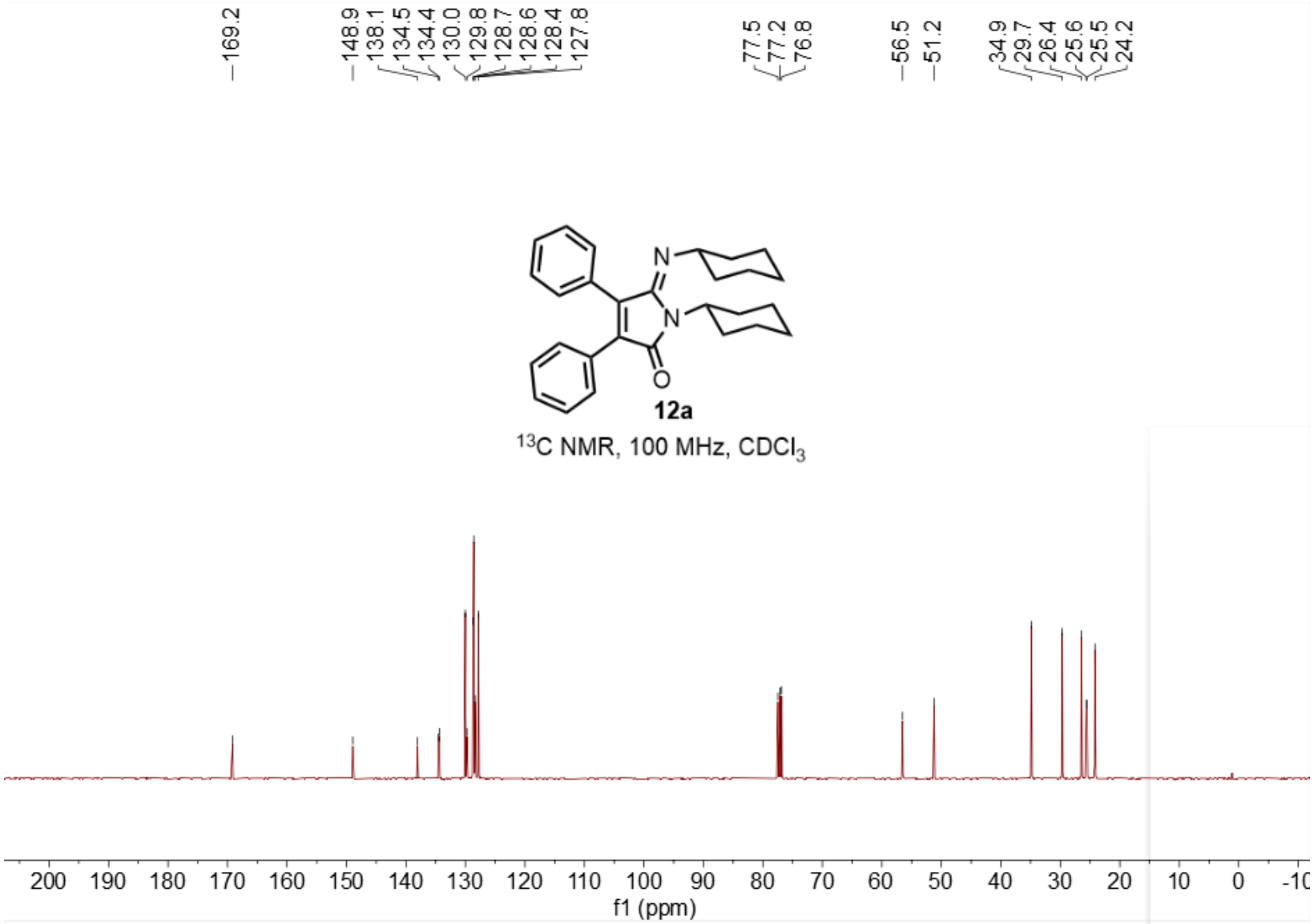

7.56
7.56
7.56
7.55
7.55
7.54
7.47
7.45
7.45
7.43
7.43
7.41
7.41
7.40
7.40
7.39
7.39
7.38
7.37
7.36
7.36
7.35
7.25
2.15
0.00
Ph
Ph
Ph
Ph
10v
1H NMR, 400 MHz, CDCl3
8.03
15.12
3.00
10.0 9.5 9.0 8.5 8.0 7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 0.5 0.0 -0.5 -1.
f1 (ppm)

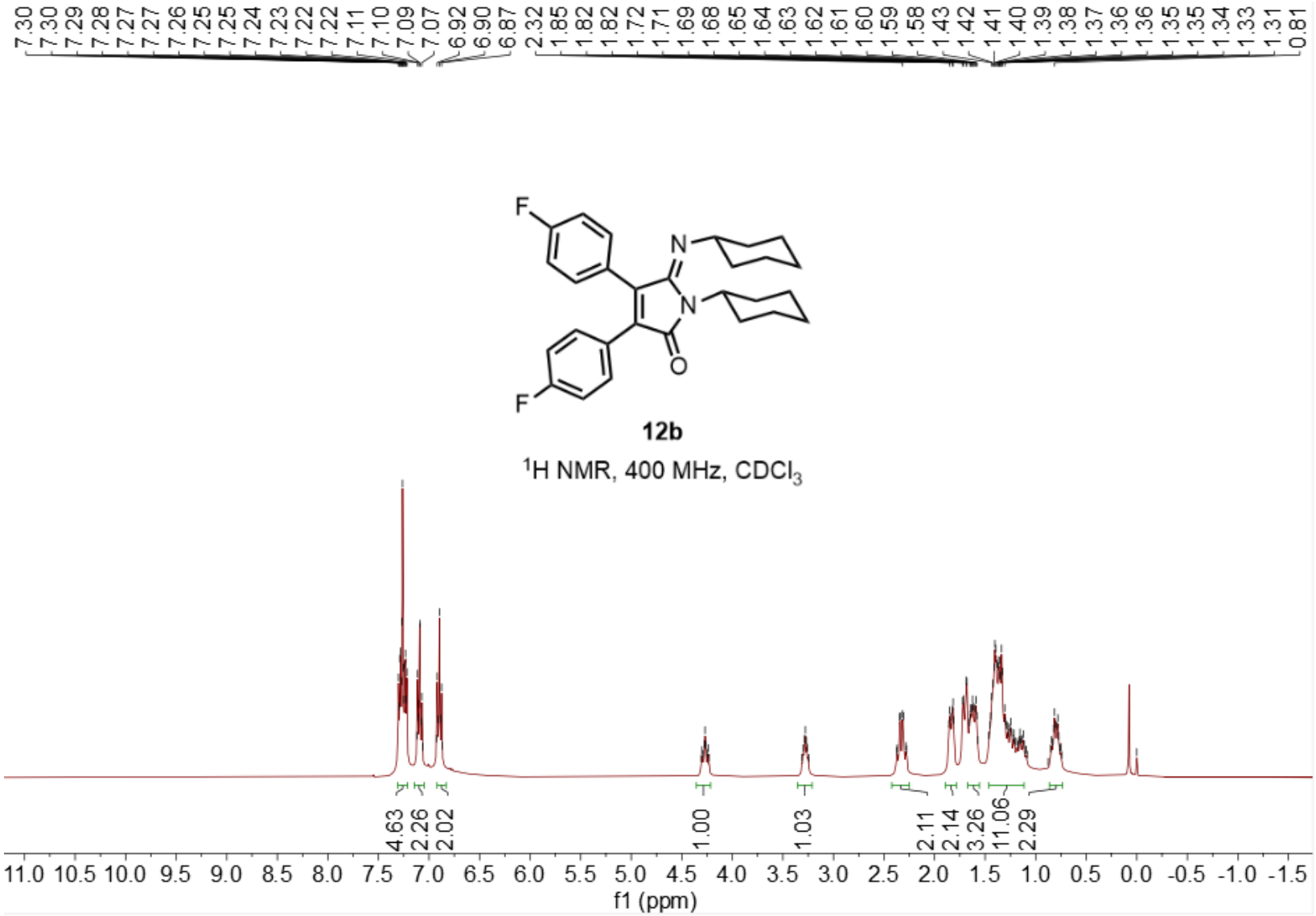

169.3
136.5
136.2
132.3
130.2
129.7
128.8
128.6
127.1
77.5
77.2
76.8
14.5
Ph
Ph
Ph
Ph
10v
13C NMR, 100 MHz, CDCl3
f1 (ppm)

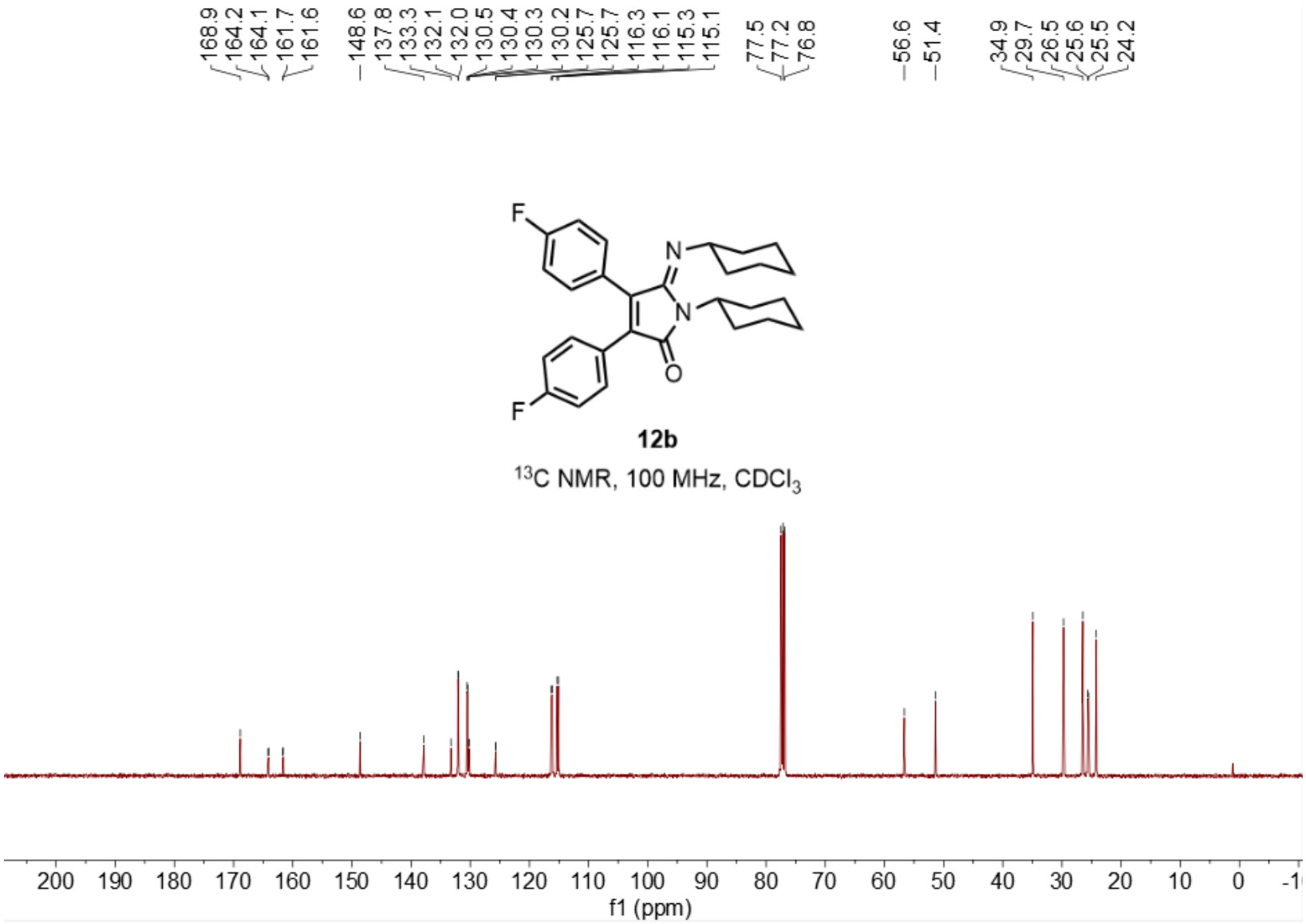

7.56
7.46
7.45
7.44
7.38
7.36
7.34
7.34
7.33
7.31
7.30
7.29
7.29
7.28
7.26
7.25
4.81
0.00
Ph
N
O
Ph
Ph
N
O
Ph
10w
1H NMR, 400 MHz, CDCl3
1.06
8.01
15.01
4.00
10.5 10.0 9.5 9.0 8.5 8.0 7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 0.5 0.0 -0.5 -1.0
f1 (ppm)

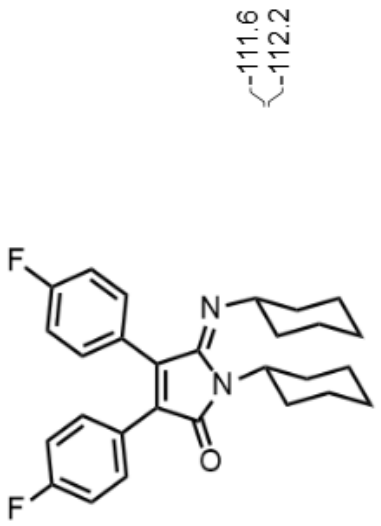

170.5
137.1
136.4
130.0
129.9
129.2
129.2
128.7
128.6
128.2
77.5
77.2
76.8
41.9
Ph
Ph
Ph
Ph
10w
13C NMR, 100 MHz, CDCl3
210 200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0 -10
f1 (ppm)


7.35 7.35 7.34 7.34 7.33 7.33 7.32 7.31 7.31 7.31 7.30 7.29 7.26 7.25 7.24 7.24 7.19 7.18 7.18 7.16
4.29 3.26 2.37 2.37 2.34 2.33 1.85 1.81 1.73 1.71 1.60 1.59 1.58 1.56 1.55 1.55 1.43 1.42 1.41 1.40 1.39 1.38 1.37 1.36 1.34 1.31 1.31 1.28 1.25 0.75 0.72
N
N
O
12a
^{1}H NMR, 400 MHz, $CDCl_3$
3.02 2.12 2.30 3.14 1.00 0.99 2.02 2.04 3.23 2.09 8.35 1.02 2.02
.5 10.5 9.5 8.5 7.5 6.5 5.5 4.5 3.5 2.5 1.5 0.5 -0.5 -1
f1 (ppm)


169.2
148.9
138.1
134.5
134.4
130.0
129.8
128.7
128.6
128.4
127.8
77.5
77.2
76.8
56.5
51.2
34.9
29.7
26.4
25.6
25.5
24.2
N
N
O
12a
13C NMR, 100 MHz, CDCl3
200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0 -10
f1 (ppm)

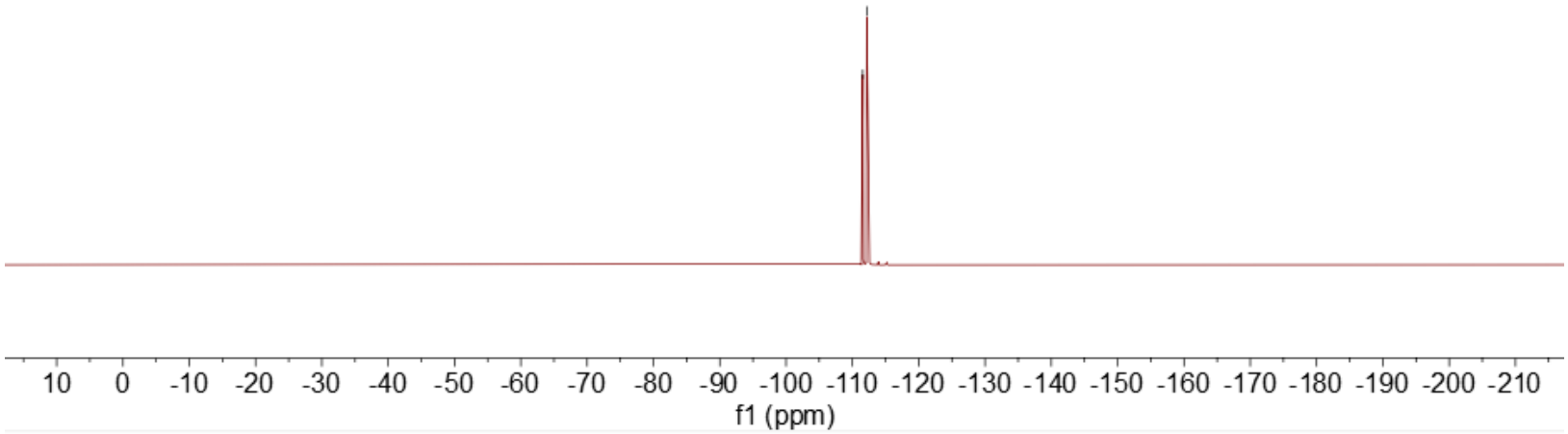

7.30
7.30
7.29
7.28
7.27
7.27
7.26
7.25
7.25
7.24
7.23
7.22
7.22
7.11
7.10
7.09
7.07
6.92
6.90
6.87
2.32
1.85
1.82
1.82
1.72
1.71
1.69
1.68
1.65
1.64
1.63
1.62
1.61
1.60
1.59
1.58
1.43
1.42
1.41
1.40
1.39
1.38
1.37
1.36
1.36
1.35
1.35
1.34
1.33
1.31
0.81
F
N
N
O
F
12b
1H NMR, 400 MHz, CDCl3
4.63
2.26
2.02
1.00
1.03
2.11
2.14
3.26
11.06
2.29
11.0 10.5 10.0 9.5 9.0 8.5 8.0 7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 0.5 0.0 -0.5 -1.0 -1.5
f1 (ppm)

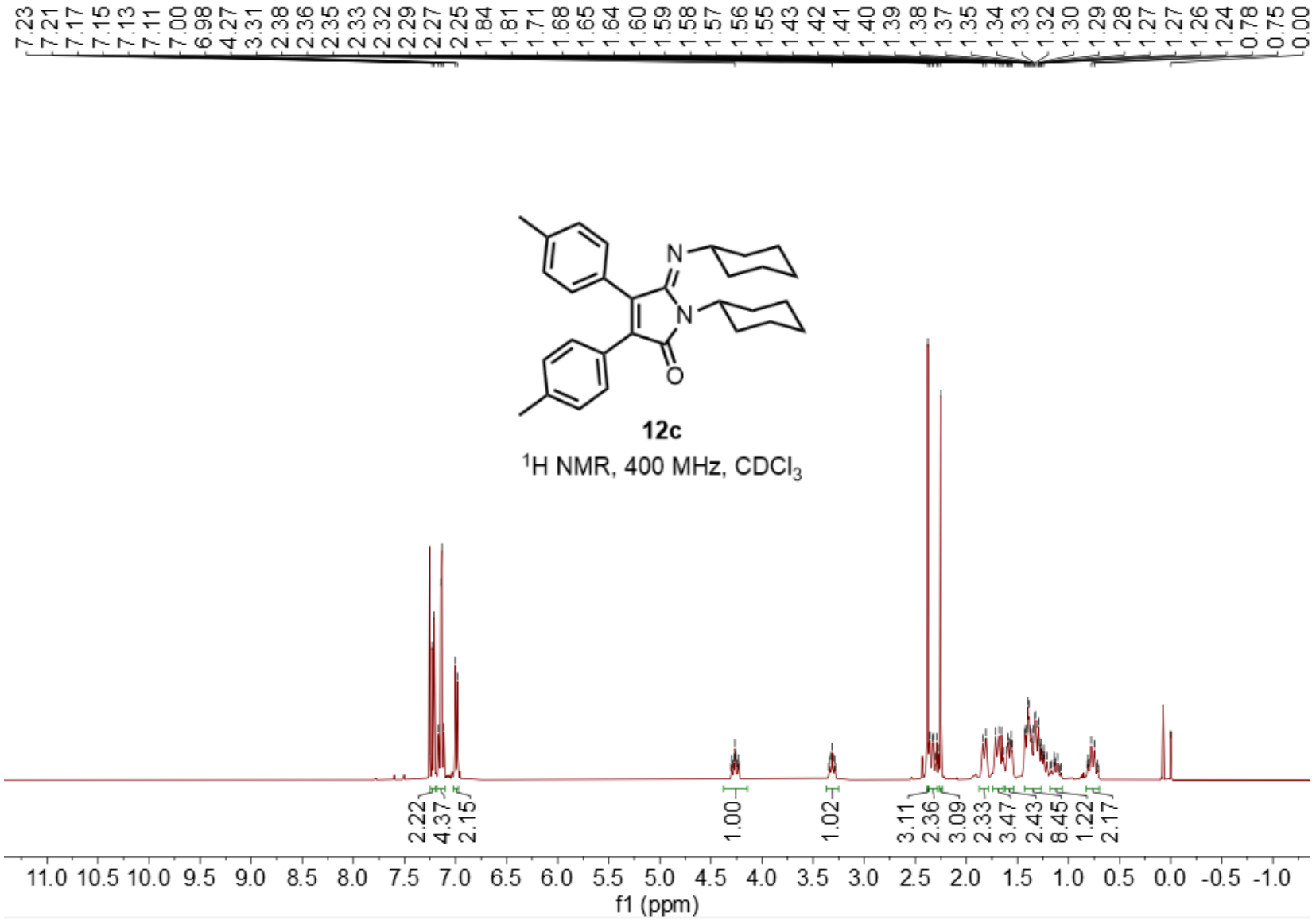

168.9
164.2
164.1
161.7
161.6
148.6
137.8
133.3
132.1
132.0
130.5
130.4
130.3
130.2
125.7
125.7
116.3
116.1
115.3
115.1
77.5
77.2
76.8
56.6
51.4
34.9
29.7
26.5
25.6
25.5
24.2
F
N
N
O
F
12b
13C NMR, 100 MHz, CDCl3
200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0 -1
f1 (ppm)

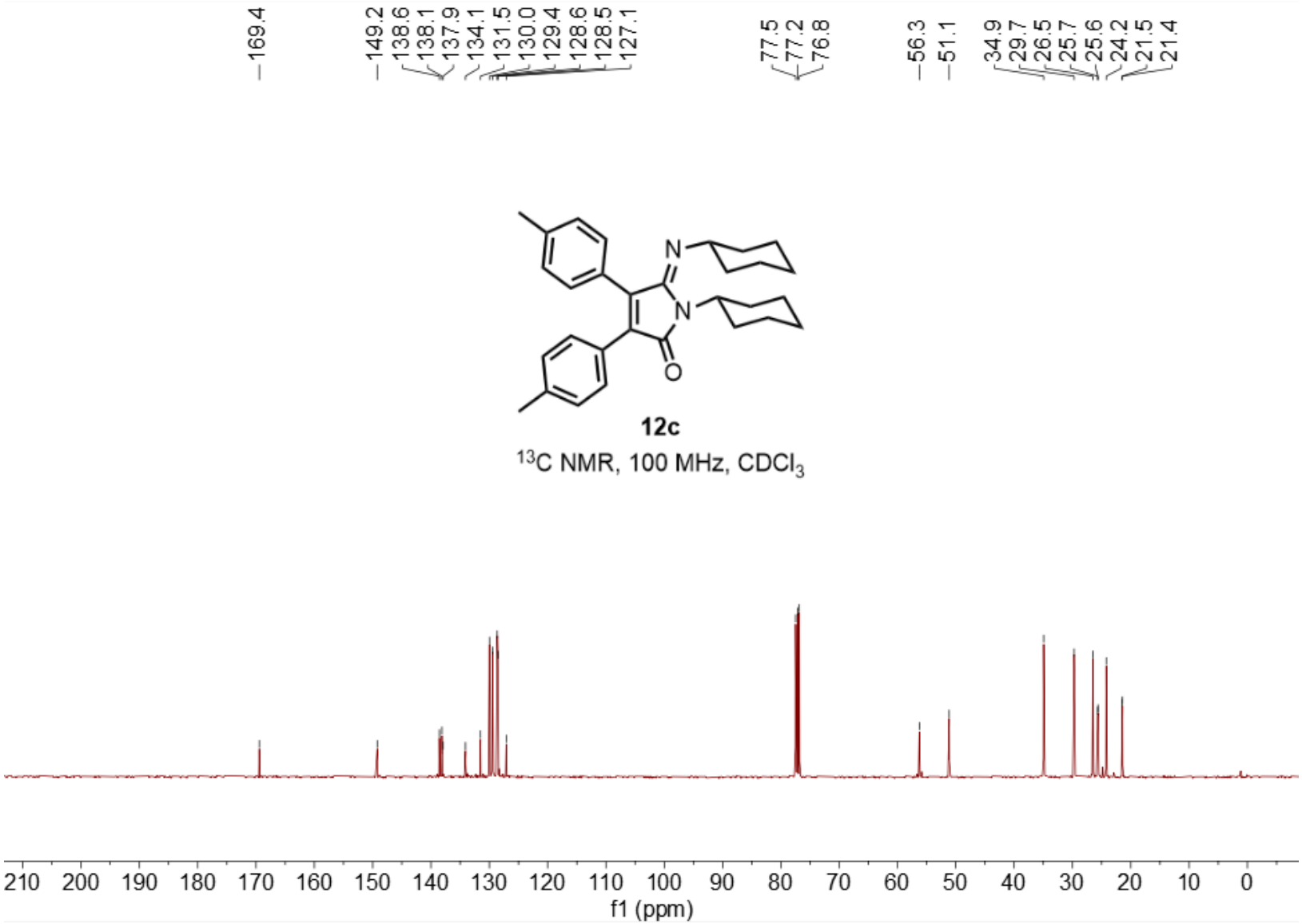


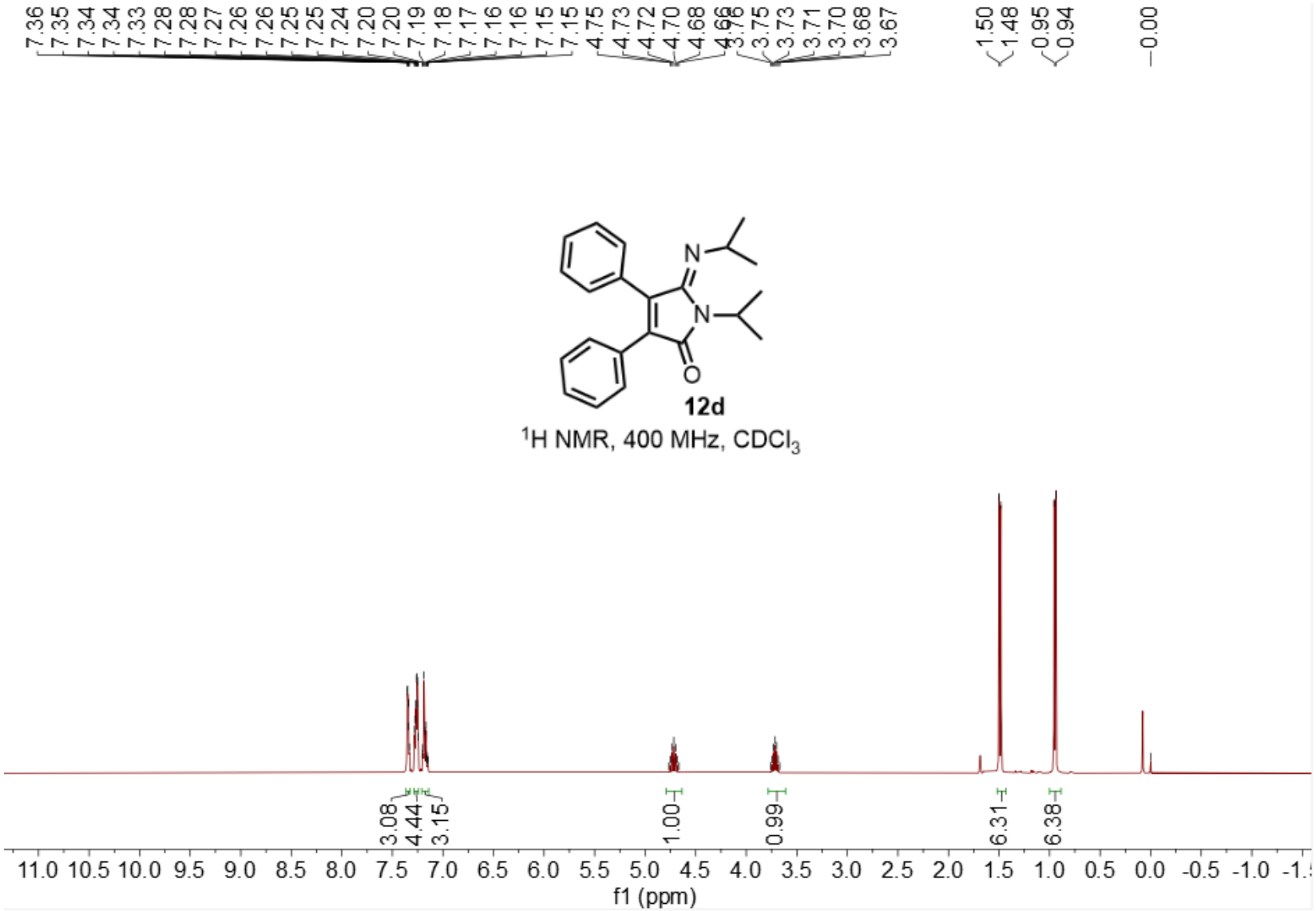
12b

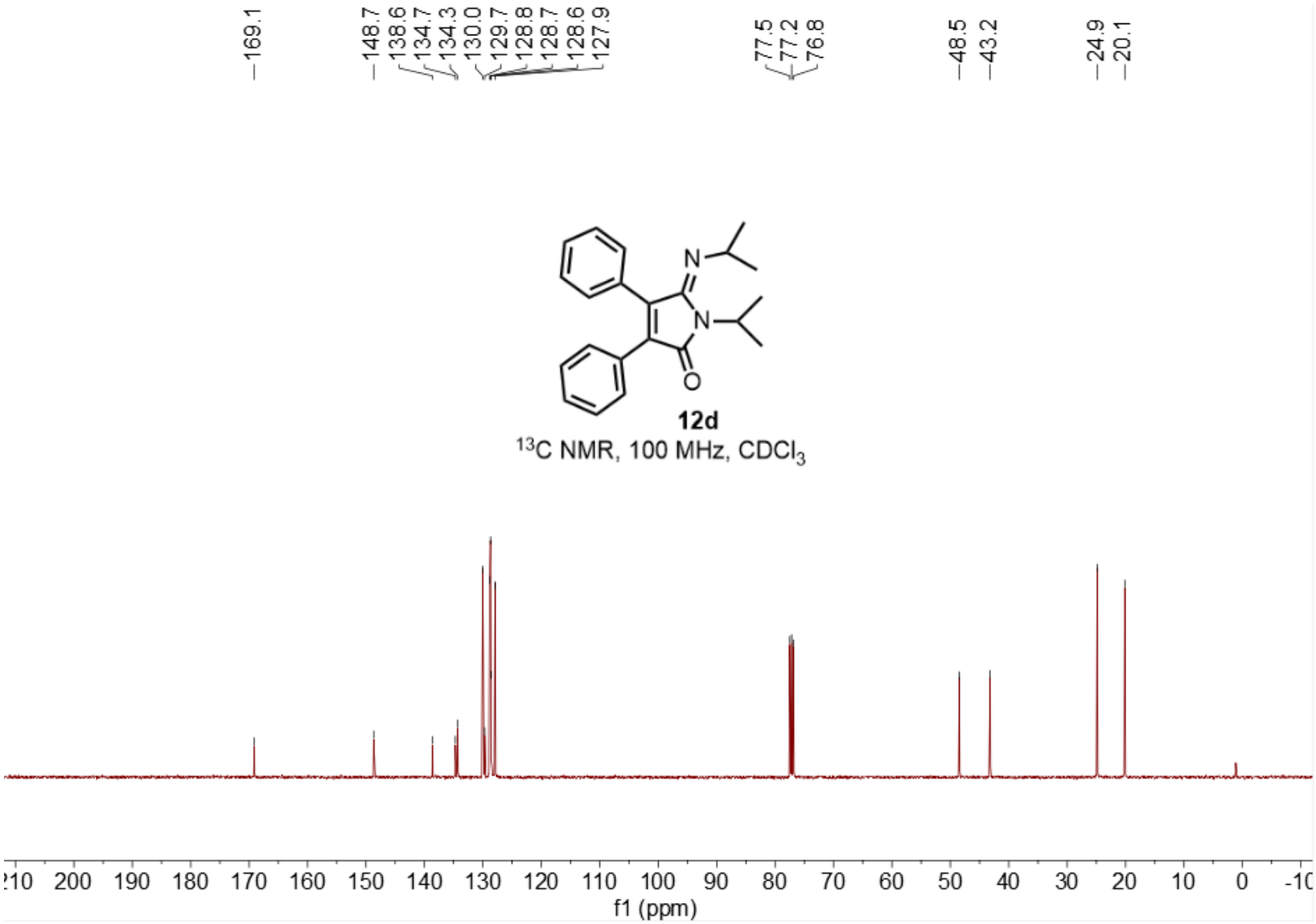
^{19}F NMR, 376 MHz, $CDCl_3$




7.23 7.21 7.17 7.15 7.13 7.11 7.00 6.98 4.27 3.31 2.38 2.36 2.35 2.33 2.32 2.29 2.27 2.25 1.84 1.81 1.71 1.68 1.65 1.64 1.60 1.59 1.58 1.57 1.56 1.55 1.43 1.42 1.41 1.40 1.39 1.38 1.37 1.35 1.34 1.33 1.32 1.30 1.29 1.28 1.27 1.27 1.26 1.24 0.78 0.75 0.00
N
N
O
12c
1H NMR, 400 MHz, CDCl3
2.22 4.37 2.15 1.00 1.02 3.11 2.36 3.09 2.33 3.47 2.43 8.45 1.22 2.17
11.0 10.5 10.0 9.5 9.0 8.5 8.0 7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 0.5 0.0 -0.5 -1.0
f1 (ppm)

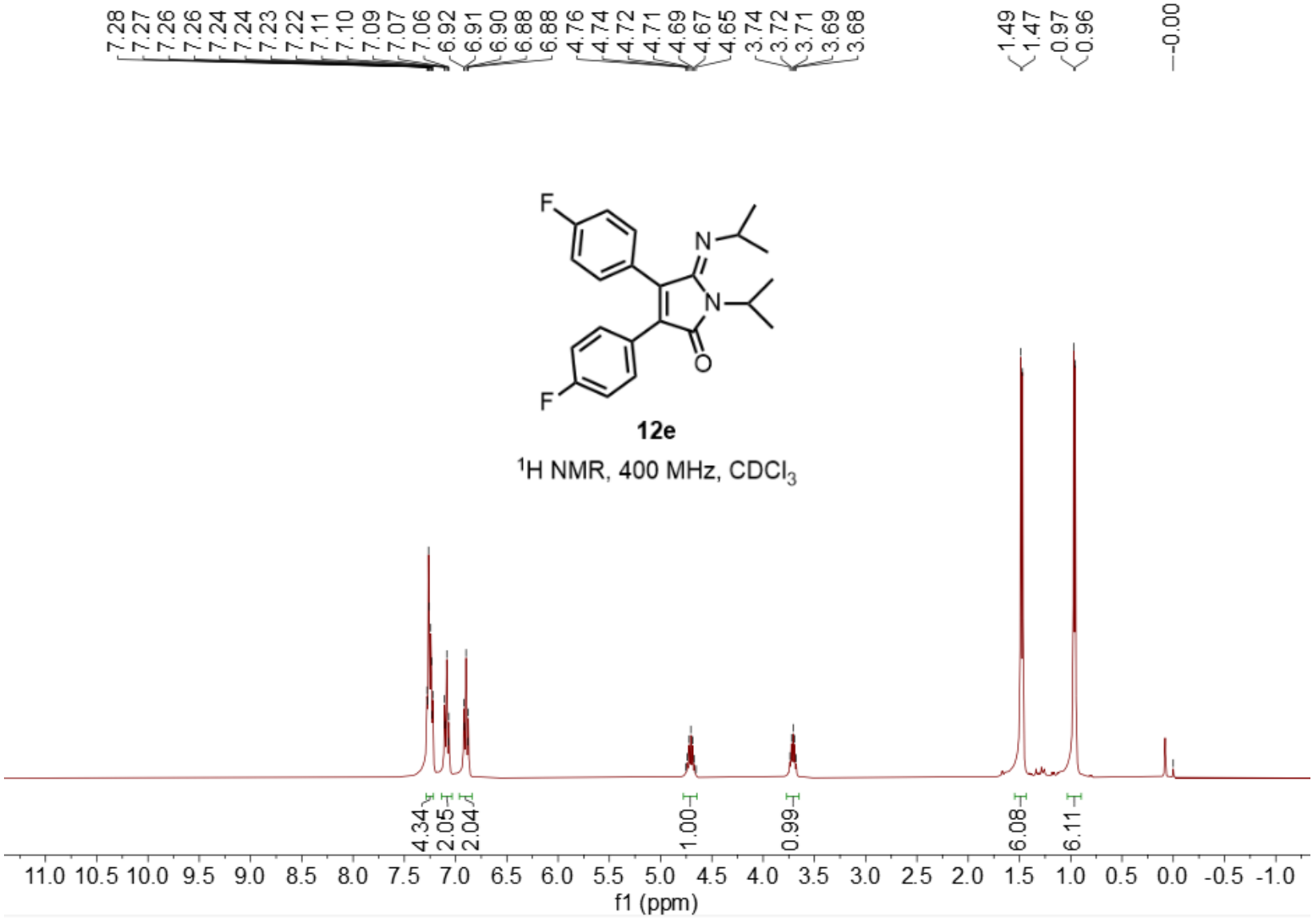

169.4
149.2
138.6
138.1
137.9
134.1
131.5
130.0
129.4
128.6
128.5
127.1
77.5
77.2
76.8
56.3
51.1
34.9
29.7
26.5
25.7
25.6
24.2
21.5
21.4
N
N
O
12c
13C NMR, 100 MHz, CDCl3
210 200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0
f1 (ppm)

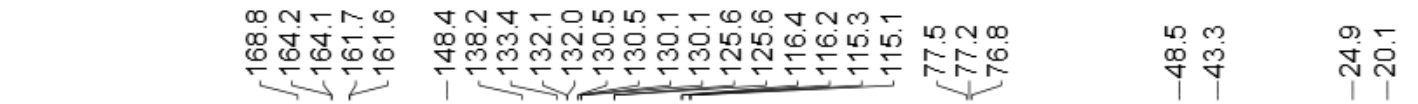

7.36
7.35
7.34
7.34
7.33
7.28
7.28
7.27
7.26
7.26
7.25
7.25
7.24
7.20
7.20
7.19
7.18
7.17
7.16
7.16
7.15
7.15
4.75
4.73
4.72
4.70
4.68
3.75
3.73
3.71
3.70
3.68
3.67
1.50
1.48
0.95
0.94
0.00
N
N
O
12d
1H NMR, 400 MHz, CDCl3
3.08
4.44
3.15
1.00
0.99
6.31
6.38
11.0 10.5 10.0 9.5 9.0 8.5 8.0 7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 0.5 0.0 -0.5 -1.0
f1 (ppm)

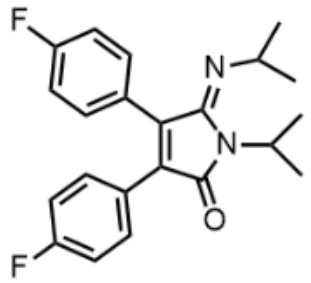


12d

^{13}C NMR, 100 MHz, $CDCl_3$

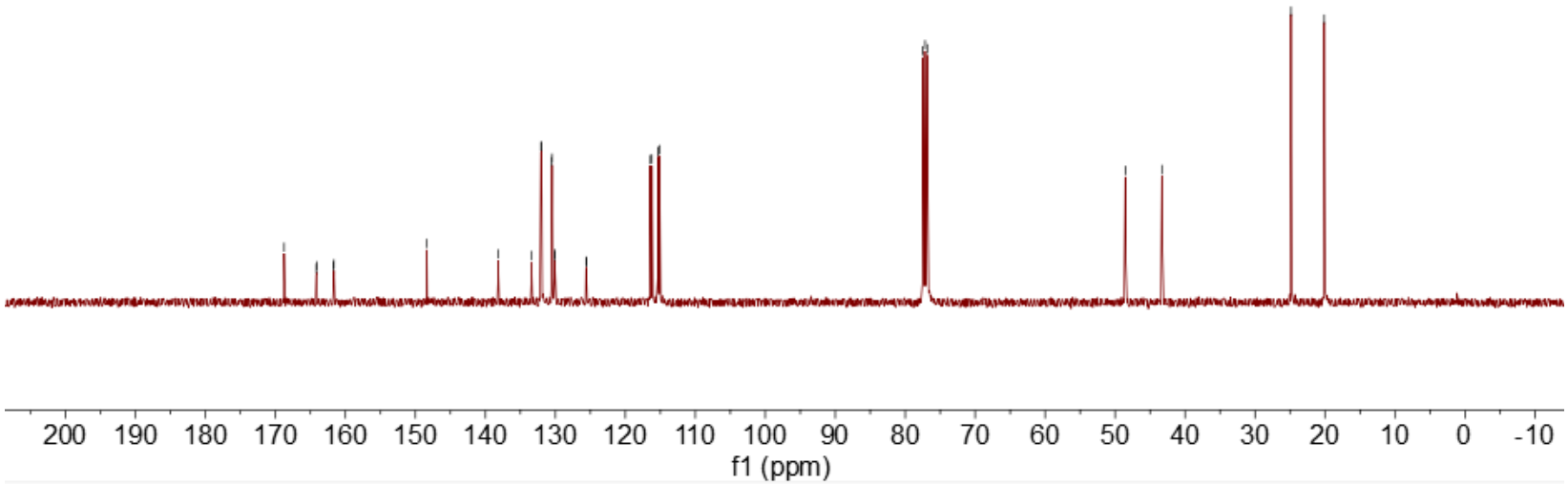

7.28
7.27
7.26
7.26
7.24
7.24
7.23
7.22
7.11
7.10
7.09
7.07
7.06
6.92
6.91
6.90
6.88
6.88
4.76
4.74
4.72
4.71
4.69
4.67
4.65
3.74
3.72
3.71
3.69
3.68
1.49
1.47
0.97
0.96
0.00
F
N
N
O
F
12e
1H NMR, 400 MHz, CDCl3
4.34
2.05
2.04
1.00
0.99
6.08
6.11
11.0 10.5 10.0 9.5 9.0 8.5 8.0 7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 0.5 0.0 -0.5 -1.0
f1 (ppm)

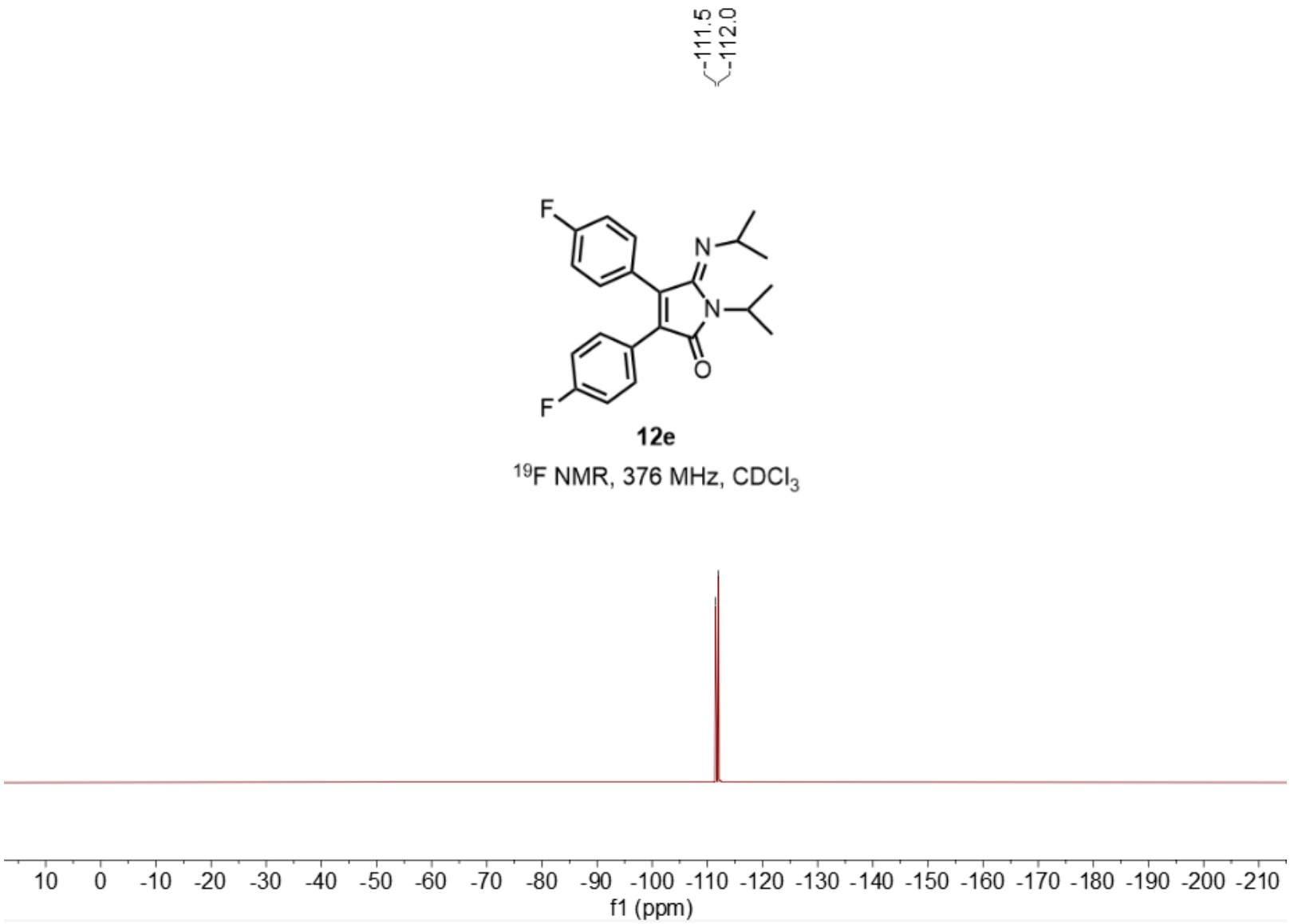


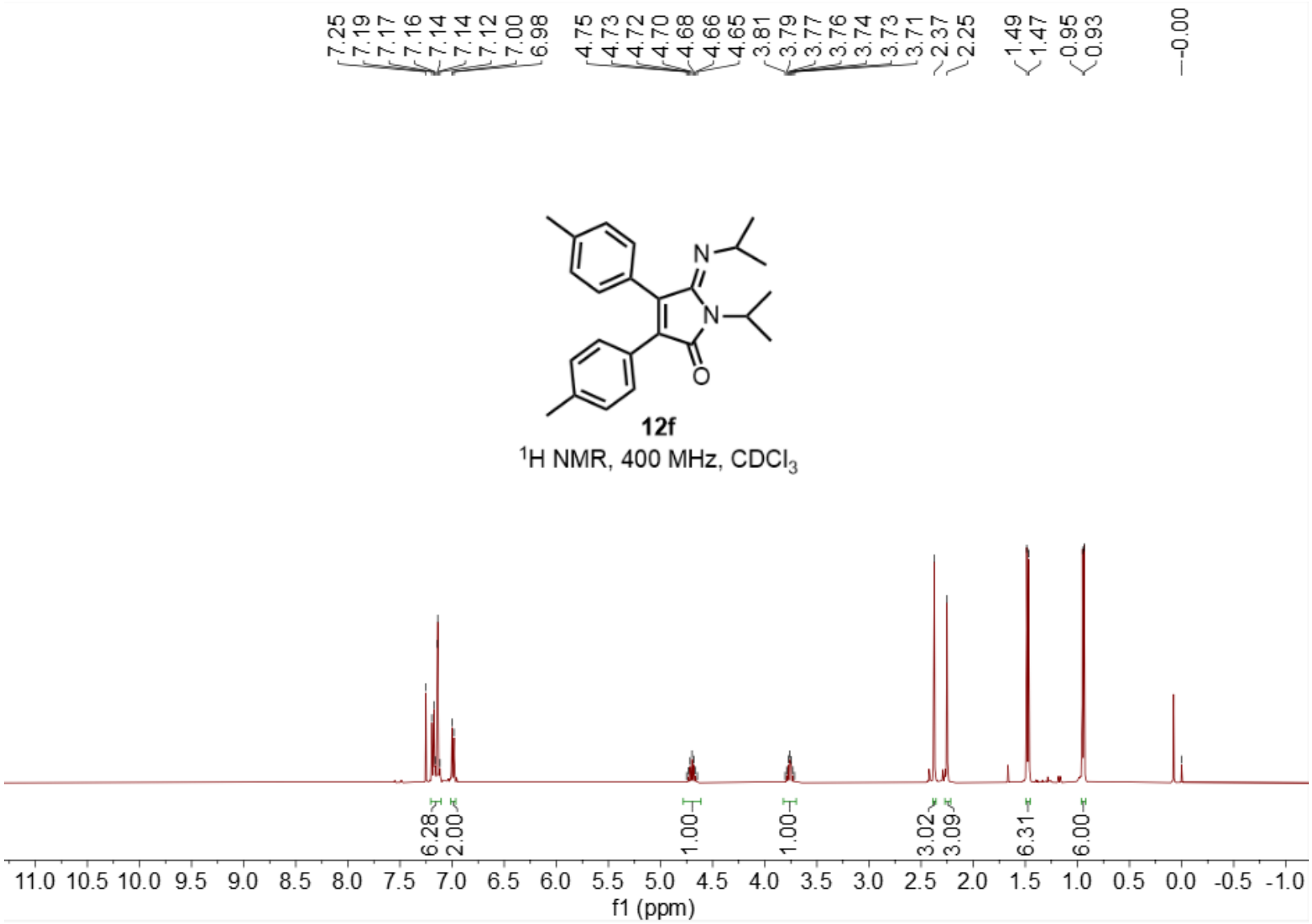


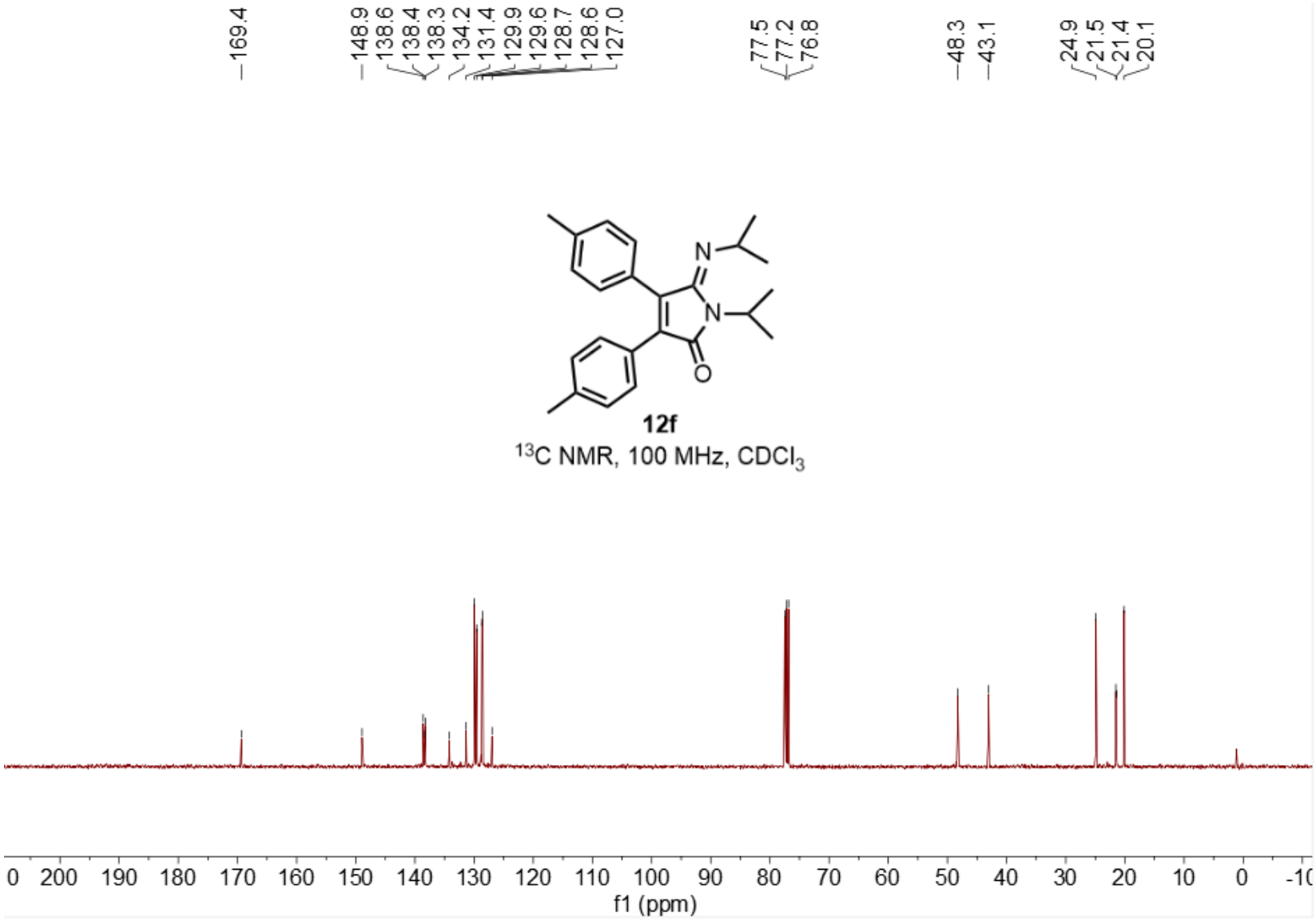
12e

^{13}C NMR, 100 MHz, $CDCl_3$




-111.5
-112.0
F
N
N
O
F
12e
19F NMR, 376 MHz, CDCl3
10 0 -10 -20 -30 -40 -50 -60 -70 -80 -90 -100 -110 -120 -130 -140 -150 -160 -170 -180 -190 -200 -210
f1 (ppm)


7.25
7.19
7.17
7.16
7.14
7.14
7.12
7.00
6.98
4.75
4.73
4.72
4.70
4.68
4.66
4.65
3.81
3.79
3.77
3.76
3.74
3.73
3.71
2.37
2.25
1.49
1.47
0.95
0.93
0.00
N
N
O
12f
1H NMR, 400 MHz, CDCl3
6.28
2.00
1.00
1.00
3.02
3.09
6.31
6.00
11.0 10.5 10.0 9.5 9.0 8.5 8.0 7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 0.5 0.0 -0.5 -1.0
f1 (ppm)


169.4
148.9
138.6
138.4
138.3
134.2
131.4
129.9
129.6
128.7
128.6
127.0
77.5
77.2
76.8
48.3
43.1
24.9
21.5
21.4
20.1
N
N
O
12f
13C NMR, 100 MHz, CDCl3
0 200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0 -10
f1 (ppm)