HOTf-catalyzed Dehydrative Coupling Reaction of 3-Hydroxyisoindolinones with Alkyl Ketones

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Table of Contents

	Page
1. General information	**2**
2. General procedure for the Mannich-type reaction between 3-hydroxyisoindoliones with alkyl ketones to C3-alkyl 3,3-disubstituted isoindolinones.	**3-13**
3. Scaled synthesis and product elaboration	**14**
4. ^{1}H, ^{13}C and ^{19}F NMR spectra	**15-78**

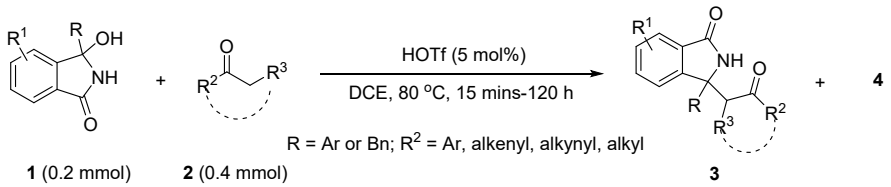
1. General information:

Reactions were monitored by thin layer chromatography using UV light to visualize the reaction course. Purification of reaction products were carried out by flash chromatography on silica gel H. Chemical yields refer to pure isolated substances. ^{1}H and ^{13}C NMR spectra were obtained using a Bruker DPX-600 or DPX-400 spectrometer. The ^{19}F NMR spectra was recorded at JEOL 565 MHz. HRMS data were collected on a on a Thermo Scientific LTQ Orbitrap Discovery (Bremen, Germany). The linear ion trap (LTQ) part of the hybrid MS system was equipped with electrospray ionization (ESI) probe and operated in both positive and negative ion modes. Chemical shifts are reported in ppm from tetramethylsilane with the solvent resonance as the internal standard. The following abbreviations were used to designate chemical shift multiplicities: s = singlet, d = doublet, t = triplet, q = quartet, h = heptet, m = multiplet, br = broad.

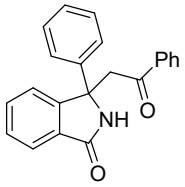
Unless noted, reactions were run under an atmosphere of air. Anhydrous THF, toluene and 1,4-dioxane were prepared by distillation over sodium-benzophenone ketyl prior to use. Anhydrous acetone was distilled over anhydrous $CaSO_4$ and stored over MS 4Å. Anhydrous halogenated solvents and CH_3CN were prepared by first distillation over P_2O_5 and then from CaH_2. Anhydrous ethyl acetate was prepared by first dried in anhydrous Na_2SO_4 and then distilled over P_2O_5 and stored over MS 4Å. Anhydrous CH_3NO_2 was prepared by first dried in anhydrous Na_2SO_4 and then distilled under reduced pressure. 3-Hydroxyisoindoliones **1** were prepared according to the literature report.[1]

1 J. Sućˊ, Josipa, I. Dokli and M. Grdičak, *Chem. Commun*., 2016, **52**, 2071.

2. General procedure for the Mannich-type reaction between 3-aryl-3-hydroxyisoindoliones with alkyl ketones to C3-alkyl 3,3-disubstituted isoindolinones.

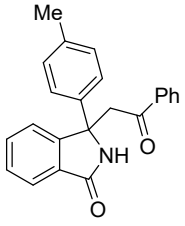


To a 10 mL vial were added 3-hydroxyisoindoliones **1** (0.2 mmol, 1.0 equiv), alkyl ketones **2** (0.4 mmol, 2.0 equivs) and 2.0 mL of anhydrous DCE. After adding HOTf (1.5 mg, 5 mol%), the reaction mixture was stirred at 80 °C till almost full conversion of **1** by TLC analysis. The reaction mixture was directly subjected to column chromatography using petroleum ether/ethyl acetate as the eluent to afford products **3** or **4**.



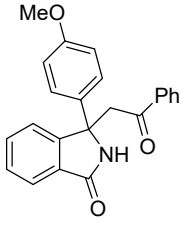
3aa

Column chromatography afforded the desired product **3aa**[2] in 96% yield (62.8 mg) as white solid; Mp: 173-175 °C; ^{1}H NMR (600 MHz, $CDCl_3$): δ = 7.95-7.94 (m, 2H), 7.88 (d, *J* = 7.2 Hz, 1H), 7.63-7.60 (m, 2H), 7.53-7.44 (m, 4H), 7.41-7.39 (m, 2H), 7.35 (d, *J* = 7.8 Hz, 1H), 7.30-7.27 (m, 2H), 7.23-7.21 (m, 1H), 4.71 (d, *J* = 18.0 Hz, 1H), 3.26 (d, *J* = 18.0 Hz, 1H).



3ba

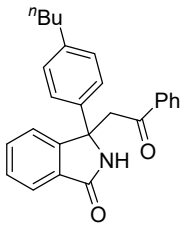
Column chromatography afforded **3ba**[2] in 74% yield (50.9 mg) as white solid; Mp: 162-164 °C; ^{1}H NMR (600 MHz, $CDCl_3$): δ = 7.88 (d, *J* =7.2 Hz, 1H), 7.84-7.83 (m, 2H), 7.67 (s, 1H), 7.52-7.50 (m, 1H), 7.45-7.43 (m, 1H), 7.40-7.39 (m, 2H), 7.36-7.34 (m, 1H), 7.28-7.26 (m, 4H), 7.22-7.19 (m, 1H), 4.69 (d, *J* = 18.0 Hz, 1H), 3.21 (d, *J* = 18.0 Hz, 1H), 2.42 (s, 3H).



3ca

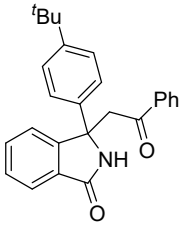
Column chromatography afforded **3ca**[2] in 93% yield (66.4 mg) as white solid; Mp: 154-156 °C; ^{1}H NMR (600 MHz, $CDCl_3$): δ = 7.95-7.94 (m, 2H), 7.88 (d, *J* = 7.2 Hz, 1H), 7.62-7.60 (m, 2H), 7.53-7.50 (m, 1H), 7.49-7.47 (m, 2H), 7.46-7.43 (m, 1H), 7.33 (d, *J* = 7.8 Hz, 1H), 7.31-7.28 (m, 2H), 6.81-6.79 (m, 2H), 4.66 (d, *J* = 18.0 Hz, 1H), 3.73 (s, 3H), 3.23 (d, *J* = 18.0 Hz, 1H).

[2] M. Matišić and M. Gredičak, *Chem. Commun.*, 2021, **57**, 13546.



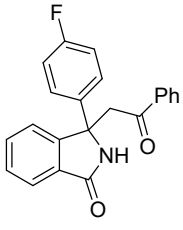
3da

Column chromatography afforded the desired product **3da** in 72% yield (58.2 mg) as white solid; Mp: 95-97 °C; ^{1}H NMR (600 MHz, $CDCl_3$): δ = 7.95-7.94 (m, 2H), 7.87 (d, *J* = 7.8 Hz, 1H), 7.62-7.59 (m, 2H), 7.52-7.43 (m, 4H), 7.35 (d, *J* = 7.8 Hz, 1H), 7.29-7.28 (m, 2H), 7.09-7.07 (m, 2H), 4.69 (d, *J* = 18.0 Hz, 1H), 3.23 (d, *J* = 18.0 Hz, 1H), 2.52 (t, *J* = 7.8 Hz, 2H), 1.54-1.49 (m, 2H), 1.32-1.28 (m, 2H), 0.88 (t, *J* = 7.2 Hz, 3H); ^{13}C{^{1}H} NMR (150 MHz, $CDCl_3$): δ = 197.2, 169.6, 151.8, 142.3, 137.6, 136.4, 133.8, 132.5, 129.9, 129.0, 128.8, 128.4, 128.0, 124.5, 124.4, 122.1, 64.4, 46.8, 35.1, 33.3, 22.4, 13.9; IR (neat, cm^{-1}): 3227, 2957, 1700, 1682, 1349, 1215, 757, 723, 692; HRMS (ESI): Exact mass calcd for $C_{26}H_{25}NO_2$ [M+Na]$^+$: 406.1778, Found: 406.1782.



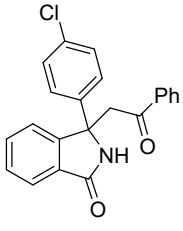
3ea

Column chromatography afforded **3ea** in 80% yield (64.2 mg) as white solid; Mp: 223-225 °C; ^{1}H NMR (600 MHz, $CDCl_3$): δ = 7.95-7.94 (m, 2H), 7.87 (d, *J* = 7.2 Hz, 1H), 7.65 (s, 1H), 7.62-7.59 (m, 1H), 7.53-7.50 (m, 1H), 7.49-7.47 (m, 2H), 7.46-7.43 (m, 1H), 7.37 (d, *J* = 7.8 Hz, 1H), 7.31-7.30 (m, 2H), 7.28-7.26 (m, 2H), 4.70 (d, *J* = 18.0 Hz, 1H), 3.23 (d, *J* = 18.0 Hz, 1H), 1.24 (s, 9H); ^{13}C{^{1}H} NMR (150 MHz, $CDCl_3$): δ = 197.2, 169.7, 137.3, 136.4, 133.9, 132.4, 129.9, 128.8, 128.4, 128.0, 125.9, 124.4, 124.3, 122.2, 64.3, 46.8, 34.4, 31.2; IR (neat, cm^{-1}): 3227, 2958, 1700, 1683, 1349, 1214, 758, 726, 692; HRMS (ESI): Exact mass calcd for $C_{26}H_{25}NO_2$ [M+Na]$^+$: 406.1778, Found: 406.1782.



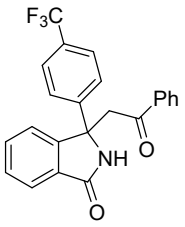
3fa

Column chromatography afforded the desired product **3fa**[2] in 99% yield (68.7 mg) as white solid; Mp: 217-219 °C; ^{1}H NMR (600 MHz, $CDCl_3$): δ = 7.94-7.93 (m, 2H), 7.89 (d, *J* = 7.2 Hz, 1H), 7.63-7.61 (m, 2H), 7.55-7.52 (m, 1H), 7.50-7.46 (m, 3H), 7.37-7.35 (m, 2H), 7.32 (d, *J* = 7.2 Hz, 1H), 6.98-6.95 (m, 2H), 4.67 (d, *J* = 18.0 Hz, 1H), 3.27 (d, *J* = 18.0 Hz, 1H).



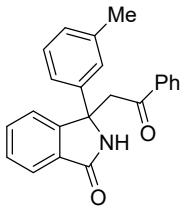
3ga

Column chromatography afforded **3ga**[2] in 89% yield (64.4 mg) as white solid; Mp: 209-211 °C; ^{1}H NMR (600 MHz, $CDCl_3$): δ = 7.94-7.93 (m, 2H), 7.89 (d, *J* = 7.2 Hz, 1H), 7.63-7.61 (m, 1H), 7.58 (s, 1H), 7.55-7.52 (m, 1H), 7.50-7.46 (m, 3H), 7.34-7.31 (m, 3H), 7.26-7.25 (m, 1H), 4.65 (d, *J* = 18.0 Hz, 1H), 3.28 (d, *J* = 18.6 Hz, 1H).



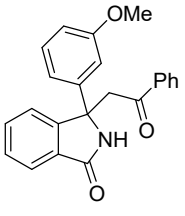
3ha

Column chromatography afforded **3ha**[2] in 79% yield (65.2 mg) as white solid; Mp: 127-129 °C; ^{1}H NMR (600 MHz, $CDCl_3$): δ = 7.95-7.94 (m, 2H), 7.90 (d, *J* = 7.8 Hz, 1H), 7.64-7.62 (m, 1H), 7.60 (s, 1H), 7.56-7.53 (m, 5H), 7.51-7.48 (m, 3H), 7.33 (d, *J* = 7.8 Hz, 1H), 4.72 (d, *J* = 18.6 Hz, 1H), 3.34 (d, *J* = 18.0 Hz, 1H).



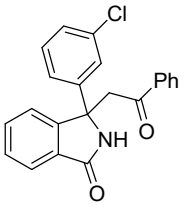
3ia

Column chromatography afforded **3ia**[2] in 94% yield (64.2 mg) as white solid; Mp: 178-180 °C; ^{1}H NMR (600 MHz, $CDCl_3$): δ = 7.95-7.93 (m, 2H), 7.88 (d, *J* = 7.2 Hz, 1H), 7.62-7.59 (m, 2H), 7.53-7.43 (m, 4H), 7.36 (d, *J* = 7.8 Hz, 1H), 7.21-7.19 (m, 2H), 7.18-7.15 (m, 1H), 7.03 (d, *J* =7.2 Hz, 1H), 4.70 (d, *J* =18.0 Hz, 1H), 3.24 (d, *J* = 18.0 Hz, 1H), 2.26 (s, 3H).



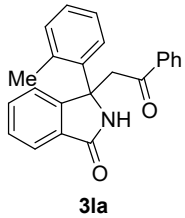
3ja

Column chromatography afforded the desired product **3ja**[2] in 88% yield (63.0 mg) as white solid; Mp: 89-91 °C; ^{1}H NMR (600 MHz, $CDCl_3$): δ = 7.94-7.93 (m, 2H), 7.88 (d, *J* = 7.8 Hz, 1H), 7.63-7.59 (m, 2H), 7.53-7.51 (m, 1H), 7.49-7.44 (m, 3H), 7.37 (d, *J* = 7.8 Hz, 1H), 7.22-7.19 (m, 1H), 7.00-6.99 (m, 1H), 6.95-6.94 (m, 1H), 6.76-6.74 (m, 1H), 4.69 (d, *J* = 18.0 Hz, 1H), 3.72 (s, 3H), 3.23 (d, *J* = 18.0 Hz, 1H).



3ka

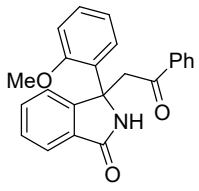
Column chromatography afforded the desired product **3ka** in 85% yield (61.7 mg) as white solid; Mp: 182-184 °C; ^{1}H NMR (600 MHz, $CDCl_3$): δ = 7.94-7.92 (m, 2H), 7.88 (d, *J* = 7.2 Hz, 1H), 7.62-7.60 (m, 2H), 7.55-7.52 (m, 1H), 7.49-7.45 (m, 3H), 7.40 (s, 1H), 7.35 (d, *J* = 7.2 Hz, 1H), 7.30 (d, *J* = 7.8 Hz, 1H), 7.23-7.18 (m, 2H), 4.65 (d, *J* = 18.6 Hz, 1H), 3.28 (d, *J* = 18.6 Hz, 1H); ^{13}C{^{1}H} NMR (150 MHz, $CDCl_3$): δ = 196.9, 169.5, 150.8, 143.0, 136.1, 135.0, 134.1, 132.7, 130.2, 129.8, 128.9, 128.8, 128.0, 127.9, 125.1, 124.6, 122.7, 122.0, 64.1, 46.9; IR (neat, cm^{-1}): 3416, 2924, 1688, 1673, 1377, 1217, 762, 753, 690, 681; HRMS (ESI): Exact mass calcd for $C_{22}H_{16}{}^{35}ClNO_2$ $[M+Na]^+$: 384.0762, Found: 384.0756.



3la

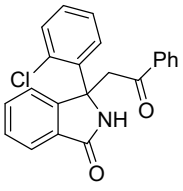
Column chromatography afforded the desired product **3la**[2] in 71% yield (48.4 mg) as white solid; Mp: 171-173 °C; ^{1}H NMR (600 MHz, $CDCl_3$): δ = 7.99-7.98 (m, 2H), 7.92 (d, *J* = 7.2 Hz, 1H), 7.64-7.62 (m, 1H), 7.58 (s, 1H), 7.54-7.48 (m, 4H), 7.19-7.13 (m, 3H), 7.11-7.08 (m, 1H), 7.05 (d, *J* = 7.2 Hz, 1H), 4.73 (d, *J* = 18.6 Hz, 1H), 3.02 (d, *J* =

18.6 Hz, 1H), 1.90 (s, 3H).



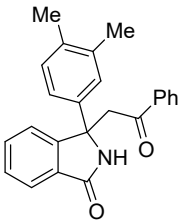
3ma

Column chromatography afforded the desired product **3ma** in 87% yield (62.1 mg) as white solid; Mp: 207-209 °C; ^{1}H NMR (600 MHz, $CDCl_3$): δ = 7.89-7.88 (m, 2H), 7.86 (d, *J* = 7.2 Hz, 1H), 7.75 (d, *J* = 7.2 Hz, 1H), 7.57 (s, 1H), 7.56-7.53 (m, 2H), 7.48-7.40 (m, 4H), 7.24-7.21 (m, 1H), 6.90-6.86 (m, 2H), 4.91 (d, *J* = 17.4 Hz, 1H), 3.76 (s, 3H), 3.33 (d, *J* = 17.4 Hz, 1H); ^{13}C{^{1}H} NMR (150 MHz, $CDCl_3$): δ = 197.5, 169.2, 156.3, 150.7, 132.1, 128.64, 128.56, 128.0, 127.6, 124.2, 123.6, 121.1, 111.7, 64.6, 55.2, 47.1; IR (neat, cm^{-1}): 3230, 2940, 1691, 1680, 1349, 1243, 751, 722, 692; HRMS (ESI): Exact mass calcd for $C_{23}H_{19}NO_3$ [M+Na]$^+$: 380.1257, Found: 380.1255.



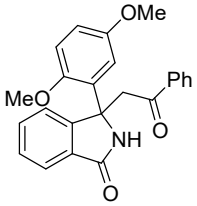
3na

Column chromatography afforded the desired product **3na** in 66% yield (47.8 mg) as white solid; Mp: 209-211 °C; ^{1}H NMR (600 MHz, $CDCl_3$): δ = 7.95-7.94 (m, 2H), 7.90 (d, *J* = 7.2 Hz, 1H), 7.62-7.54 (m, 3H), 7.51-7.46 (m, 5H), 7.33-7.31 (m, 1H), 7.20-7.18 (m, 2H), 5.01 (d, *J* = 18.6 Hz, 1H), 3.39 (d, *J* = 18.6 Hz, 1H); ^{13}C{^{1}H} NMR (150 MHz, $CDCl_3$): δ = 197.1, 169.5, 150.1, 136.5, 136.1, 133.9, 132.5, 132.4, 132.0, 131.3, 129.4, 128.9, 128.84, 128.80, 128.1, 127.2, 124.4, 122.8, 65.0, 48.4; IR (neat, cm^{-1}): 3225, 2920, 1683, 1681, 1350, 1218, 756, 730, 691; HRMS (ESI): Exact mass calcd for $C_{22}H_{16}{}^{35}ClNO_2$ [M+Na]$^+$: 384.0762, Found: 384.0764.



3oa

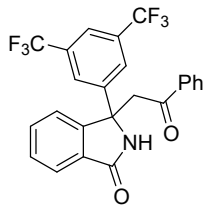
Column chromatography afforded the desired product **3oa** in 93% yield (66.1 mg) as white solid; Mp: 154-156 °C; ^{1}H NMR (600 MHz, $CDCl_3$): δ = 7.95-7.94 (m, 2H), 7.87 (d, *J* = 7.2 Hz, 1H), 7.62-7.59 (m, 2H), 7.52-7.46 (m, 3H), 7.45-7.42 (m, 1H), 7.36 (d, *J* = 7.8 Hz, 1H), 7.15 (s, 1H), 7.12 (d, *J* = 8.4 Hz, 1H), 7.03 (d, *J* = 7.8 Hz, 1H), 4.69 (d, *J* = 18.6 Hz, 1H), 3.22 (d, *J* = 18.0 Hz, 1H), 2.165 (s, 3H), 2.162 (s, 3H); ^{13}C{^{1}H} NMR (150 MHz, $CDCl_3$): δ = 197.3, 169.7, 151.8, 137.9, 137.3, 136.4, 136.1, 133.8, 132.5, 130.2, 129.9, 128.8, 128.4, 128.0, 125.8, 124.3, 122.0, 121.8, 64.3, 46.7, 20.0, 19.3; IR (neat, cm^{-1}): 3309, 2918, 1697, 1676, 1345, 1222, 990, 784, 763, 696, 597; HRMS (ESI): Exact mass calcd for $C_{24}H_{21}NO_2$ [M+Na]$^+$: 378.1465, Found: 378.1474.



3pa

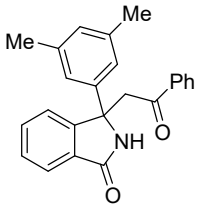
Column chromatography afforded the desired product **3pa** in 78% yield (60.7 mg) as

colorless sticky oil; ^{1}H NMR (600 MHz, $CDCl_3$): δ = 7.89-7.88 (m, 2H), 7.85 (d, *J* = 7.2 Hz, 1H), 7.73 (d, *J* = 7.8 Hz, 1H), 7.57-7.53 (m, 3H), 7.47-7.42 (m, 3H), 7.31 (d, *J* = 9.0 Hz, 1H), 6.419-6.415 (m, 1H), 6.40-6.38 (m, 1H), 4.86 (d, *J* = 17.4 Hz, 1H), 3.74 (s, 3H), 3.72 (s, 3H), 3.28 (d, *J* = 17.4 Hz, 1H); ^{13}C{^{1}H} NMR (150 MHz, $CDCl_3$): δ = 197.8, 169.3, 160.5, 157.4, 151.1, 137.0, 133.4, 132.2, 128.7, 128.6, 128.4, 128.1, 124.2, 123.5, 120.4, 104.4, 99.8, 64.4, 55.41, 55.35, 47.1; IR (neat, cm^{-1}): 3203, 2922, 1694, 1681, 1308, 1261, 1208, 1026, 752, 726, 690; HRMS (ESI): Exact mass calcd for $C_{24}H_{21}NO_4$ [M+Na]$^+$: 410.1363, Found: 410.1370.



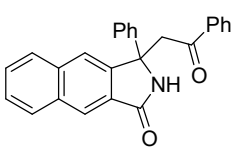
3qa

Column chromatography afforded **3qa**[2] in 75% yield (70.3 mg) as white solid; Mp: 143-145 °C; ^{1}H NMR (600 MHz, $CDCl_3$): δ = 7.94-7.91 (m, 3H), 7.86 (s, 2H), 7.76 (s, 1H), 7.66-7.63 (m, 1H), 7.60-7.57 (m, 2H), 7.54-7.49 (m, 3H), 7.33 (d, *J* = 7.8 Hz, 1H), 4.68 (d, *J* = 18.0 Hz, 1H), 3.43 (d, *J* = 18.0 Hz, 1H).



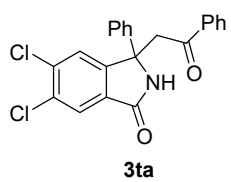
3ra

Column chromatography afforded the desired product **3ra**[2] in 82% yield (58.1 mg) as white solid; Mp: 206-208 °C; ^{1}H NMR (600 MHz, $CDCl_3$): δ = 7.95-7.94 (m, 2H), 7.87 (d, *J* = 7.2 Hz, 1H), 7.62-7.60 (m, 2H), 7.53-7.50 (m, 1H), 7.49-7.47 (m, 2H), 7.45-7.43 (m, 1H), 7.38 (d, *J* = 7.8 Hz, 1H), 7.01 (s, 2H), 6.85 (s, 1H), 4.69 (d, *J* = 18.0 Hz, 1H), 3.23 (d, *J* = 18.0 Hz, 1H), 2.22 (s, 6H).



3sa

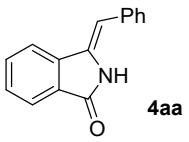
Column chromatography afforded the desired product **3sa** in 74% yield (56.5 mg) as white solid; Mp: 218-220 °C; ^{1}H NMR (600 MHz, $CDCl_3$): δ = 8.43 (s, 1H), 8.01 (d, *J* = 8.4 Hz, 1H), 7.97-7.96 (m, 2H), 7.84 (d, *J* = 7.8 Hz, 1H), 7.75 (s, 1H), 7.74 (s, 1H), 7.63-7.60 (m, 1H), 7.58-7.52 (m, 2H), 7.50-7.47 (m, 4H), 7.32-7.30 (m, 2H), 7.24-7.22 (m, 1H), 4.83 (d, *J* = 18.6 Hz, 1H), 3.36 (d, *J* = 18.6 Hz, 1H); ^{13}C{^{1}H} NMR (150 MHz, $CDCl_3$): δ = 197.2, 169.4, 146.7, 141.4, 136.4, 135.5, 133.9, 133.0, 129.6, 129.0, 128.9, 128.2, 128.05, 128.01, 127.9, 127.6, 126.7, 125.0, 124.6, 121.3, 64.4, 47.5; IR (neat, cm^{-1}): 3432, 2919, 1695, 1677, 1376, 1220, 763, 748, 697, 688, 667; HRMS (ESI): Exact mass calcd for $C_{26}H_{19}NO_2$ [M+Na]$^+$: 400.1308, Found: 400.1315



3ta

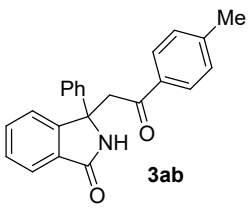
Column chromatography afforded the desired product **3ta** in 75% yield (59.6 mg) as

pale yellow solid; Mp: 242-244 °C; ^{1}H NMR (600 MHz, $CDCl_3$): δ = 7.945-7.935 (m, 3H), 7.70 (s, 1H), 7.64-7.62 (m, 1H), 7.51-7.48 (m, 2H), 7.43 (s, 1H), 7.37-7.36 (m, 2H), 7.33-7.30 (m, 2H), 7.27-7.25 (m, 1H), 4.68 (d, *J* = 18.0 Hz, 1H), 3.26 (d, *J* = 18.0 Hz, 1H); ^{13}C{^{1}H} NMR (150 MHz, $CDCl_3$): δ = 196.6, 167.4, 150.5, 139.5, 137.0, 136.1, 134.2 133.6, 129.9, 129.3, 128.9, 128.10, 128.06, 126.3, 124.5, 64.2, 46.4; IR (neat, cm^{-1}): 3426, 2921, 1699, 1670, 1396, 1227, 1003, 748, 682, 669, 626; HRMS (ESI): Exact mass calcd for $C_{22}H_{15}{}^{35}Cl_2NO_2$ $[M+Na]^+$: 418.0372, Found: 418.0381.



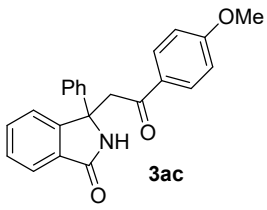
4aa

Column chromatography afforded the desired product **4aa**[3] in 87% yield (38.4 mg) as pale white solid; Mp: 177-179 °C; ^{1}H NMR (600 MHz, $CDCl_3$): δ = 8.37 (s, 1H), 7.88 (d, *J* = 7.8 Hz, 1H), 7.79 (d, *J* = 7.8 Hz, 1H), 7.65-7.62 (m, 1H), 7.53-7.51 (m, 1H), 7.47-7.43 (m, 4H), 7.33-7.30 (m, 1H), 6.56 (s, 1H).



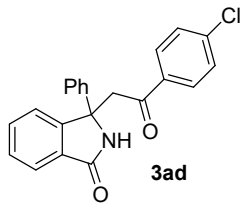
3ab

Column chromatography afforded the desired product **3ab**[4] in 99% yield (68.2 mg) as white solid; Mp: 198-200 °C; ^{1}H NMR (600 MHz, $CDCl_3$): δ = 7.88 (d, *J* = 7.8 Hz, 1H), 7.85-7.84 (m, 2H), 7.66 (s, 1H), 7.53-7.50 (m, 1H), 7.46-7.43 (m, 1H), 7.40-7.39 (m, 2H), 7.35 (d, *J* = 7.2 Hz, 1H), 7.29-7.26 (m, 4H), 7.22-7.20 (m, 1H), 4.69 (d, *J* = 18.0 Hz, 1H), 3.21 (d, *J* = 18.0 Hz, 1H), 2.42 (s, 3H).



3ac

Column chromatography afforded **3ac**[4] in 93% yield (66.2 mg) as white solid; Mp: 174-176 °C; ^{1}H NMR (600 MHz, $CDCl_3$): δ = 7.93-7.92 (m, 2H), 7.88 (d, *J* = 7.2 Hz, 1H), 7.68 (s, 1H), 7.52-7.50 (m, 1H), 7.46-7.43 (m, 1H), 7.40-7.39 (m, 2H), 7.34 (d, *J* = 7.8 Hz, 1H), 7.29-7.26 (m, 2H), 7.22-7.20 (m, 1H), 6.95-6.93 (m, 2H), 4.67 (d, *J* = 18.0 Hz, 1H), 3.88 (s, 3H), 3.19 (d, *J* = 18.0 Hz, 1H).

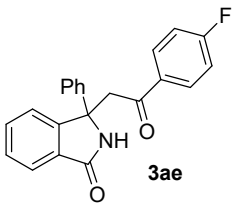


3ad

Column chromatography afforded product **3ad** in 99% yield (72.3 mg) as white solid; Mp: 274-276 °C; ^{1}H NMR (600 MHz, $CDCl_3$): δ = 7.89-7.87 (m, 3H), 7.60 (s, 1H), 7.53-7.51 (m, 1H), 7.46-7.44 (m, 3H), 7.39-7.38 (m, 2H), 7.35 (d, *J* = 7.8 Hz, 1H), 7.30-7.28 (m, 2H), 7.24-7.22 (m, 1H), 4.66 (d, *J* = 18.0 Hz, 1H), 3.24 (d, *J* = 18.0 Hz, 1H); ^{13}C{^{1}H} NMR (150 MHz, $CDCl_3$): δ = 196.0, 169.7, 151.5, 140.6, 134.7, 132.6, 130.0 129.5,

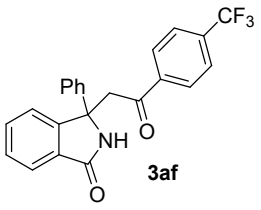
[3] M. Hellal and G. D. Cuny, *Tetrahedron Lett.*, 2011, **52**, 5508.
[4] F.-F. Feng, J.-S. Li, S. Li and J.-A. Ma, *Adv. Synth. Catal.* 2019, ***361***, 4222.

129.3, 129.1, 128.7, 127.8, 124.65, 124.56, 122.2, 64.5, 46.9; IR (neat, cm^{-1}): 3417, 2923, 1691, 1667, 1376, 1223, 1090, 1002, 790, 753, 695; HRMS (ESI): Exact mass calcd for $C_{22}H_{16}{}^{35}ClNO_2$ $[M+Na]^+$: 384.0762, Found: 384.0768.



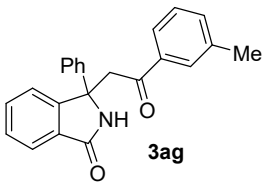
3ae

Column chromatography afforded the desired product **3ae**[4] in 99% yield (69.0 mg) as white solid; Mp: 194-196 °C; ^1H NMR (600 MHz, $CDCl_3$): δ = 7.98-7.96 (m, 2H), 7.88 (d, *J* = 7.8 Hz, 1H), 7.61 (s, 1H), 7.53-7.51 (m, 1H), 7.46-7.44 (m, 1H), 7.40-7.39 (m, 2H), 7.35 (d, *J* = 7.8 Hz, 1H), 7.30-7.28 (m, 2H), 7.24-7.21 (m, 1H), 7.16-7.13 (m, 2H), 4.67 (d, *J* = 18.0 Hz, 1H), 3.24 (d, *J* = 18.0 Hz, 1H).



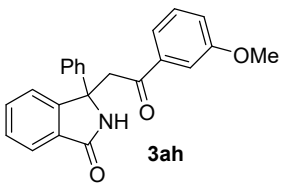
3af

Column chromatography afforded the desired product **3af**[4] in 77% yield (60.8 mg) as white solid; Mp: 224-226 °C; ^1H NMR (600 MHz, $CDCl_3$): δ = 8.04 (AB, *J* = 8.4 Hz, 2H), 7.89 (d, *J* = 7.2 Hz, 1H), 7.75 (AB, *J* = 8.4 Hz, 2H), 7.56 (s, 1H), 7.54-7.52 (m, 1H), 7.48-7.45 (m, 1H), 7.40-7.39 (m, 2H), 7.36 (d, *J* = 7.8 Hz, 1H), 7.31-7.29 (m, 2H), 7.25-7.23 (m, 1H), 4.70 (d, *J* = 18.0 Hz, 1H), 3.31 (d, *J* = 18.0 Hz, 1H).



3ag

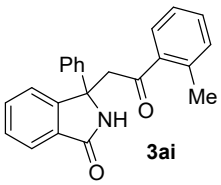
Column chromatography afforded the desired product **3ag**[4] in 93% yield (64.1 mg) as white solid; Mp: 198-200 °C; ^1H NMR (600 MHz, $CDCl_3$): δ = 7.88 (d, *J* = 7.8 Hz, 1H), 7.75-7.74 (m, 2H), 7.64 (s, 1H), 7.53-7.51 (m, 1H), 7.46-7.44 (m, 1H), 7.43-7.39 (m, 3H), 7.38-7.35 (m, 2H), 7.29-7.27 (m, 2H), 7.23-7.20 (m, 1H), 4.70 (d, *J* = 18.0 Hz, 1H), 3.24 (d, *J* = 18.0 Hz, 1H), 2.41 (s, 3H).



3ah

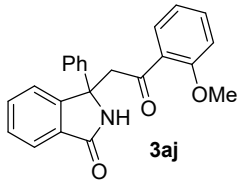
Column chromatography afforded the desired product **3ah** in 80% yield (57.9 mg) as white solid; Mp: 201-203 °C; ^1H NMR (600 MHz, $CDCl_3$): δ = 7.88 (d, *J* = 7.2 Hz, 1H), 7.63 (s, 1H), 7.54-7.50 (m, 2H), 7.46-7.44 (m, 2H), 7.40-7.37 (m, 3H), 7.35 (d, *J* = 7.8 Hz, 1H), 7.30-7.27 (m, 2H), 7.23-7.21 (m, 1H), 7.16-7.14 (m, 1H), 4.70 (d, *J* = 18.0 Hz, 1H), 3.83 (s, 3H), 3.24 (d, *J* = 18.0 Hz, 1H); $^{13}C\{^1H\}$ NMR (150 MHz, $CDCl_3$): δ = 197.0, 169.7, 160.0, 151.5, 140.6, 137.7, 132.5 129.9, 129.8, 129.0, 128.5, 127.6, 125.5, 124.6, 124.4, 122.1, 120.6, 120.4, 112.2, 64.5, 55.5, 46.9; IR (neat, cm^{-1}): 3407, 2990, 1691, 1674, 1373, 1256, 1202, 1039, 852, 791, 754, 704, 686, 601; HRMS (ESI): Exact mass calcd for $C_{23}H_{19}NO_3$ $[M+Na]^+$:

380.1257, Found: 380.1267.



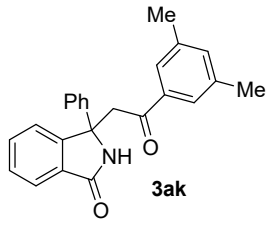
3ai

Column chromatography afforded the desired product **3ai** in 80% yield (54.9 mg) as white solid; Mp: 125-127 °C; ^{1}H NMR (600 MHz, $CDCl_3$): δ = 7.88 (d, *J* = 7.2 Hz, 1H), 7.70 (s, 1H), 7.66 (d, *J* = 7.8 Hz, 1H), 7.50-7.48 (m, 1H), 7.45-7.39 (m, 4H), 7.32-7.27 (m, 4H), 7.25-7.22 (m, 2H), 4.57 (d, *J* = 18.0 Hz, 1H), 3.24 (d, *J* = 17.4 Hz, 1H), 2.30 (s, 3H); ^{13}C{^{1}H} NMR (150 MHz, $CDCl_3$): δ = 201.0, 169.7, 151.4, 140.7, 138.7, 137.2, 132.5, 132.3, 132.1, 129.9, 129.0, 128.5, 128.3, 127.6, 125.8, 124.7, 124.4, 122.1, 64.9, 49.4, 21.2; IR (neat, cm^{-1}): 3204, 2923, 1688, 1686, 1349, 1215, 983, 906, 774, 753, 735, 703; HRMS (ESI): Exact mass calcd for $C_{23}H_{19}NO_2$ $[M+Na]^+$: 364.1308, Found: 364.1306.



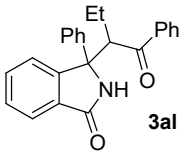
3aj

Column chromatography afforded **3aj** in 82% yield (58.8 mg) as white solid; Mp: 195-197 °C; ^{1}H NMR (600 MHz, $CDCl_3$): δ = 7.87 (d, *J* = 7.2 Hz, 1H), 7.64 (s, 1H), 7.60-7.59 (m, 1H), 7.52-7.48 (m, 2H), 7.44-7.42 (m, 1H), 7.41-7.40 (m, 2H), 7.30-7.26 (m, 3H), 7.22-7.20 (m, 1H), 7.00-6.98 (m, 2H), 4.72 (d, *J* = 18.6 Hz, 1H), 3.92 (s, 3H), 3.29 (d, *J* = 18.6 Hz, 1H); ^{13}C{^{1}H} NMR (150 MHz, $CDCl_3$): δ = 199.2, 169.9, 158.8, 151.9, 132.5, 130.7, 129.0, 128.4, 127.5, 124.9, 122.3, 121.1, 111.6, 65.1, 55.7, 52.0; IR (neat, cm^{-1}): 3314, 2922, 1694, 1652, 1594, 1492, 1434, 1348, 1239, 1012, 771, 751, 699; HRMS (ESI): Exact mass calcd for $C_{23}H_{19}NO_3$ $[M+Na]^+$: 380.1257, Found: 380.1265.



3ak

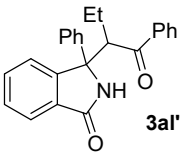
Column chromatography afforded **3ak** in 91% yield (64.7 mg) as white solid; Mp: 198-200 °C; ^{1}H NMR (600 MHz, $CDCl_3$): δ = 7.88 (d, *J* = 7.8 Hz, 1H), 7.66 (s, 1H), 7.54 (s, 2H), 7.53-7.50 (m, 1H), 7.46-7.43 (m, 1H), 7.40-7.39 (m, 2H), 7.37 (d, *J* = 7.8 Hz, 1H), 7.29-7.26 (m, 2H), 7.24 (s, 1H), 7.22-7.20 (m, 1H), 4.69 (d, *J* = 18.6 Hz, 1H), 3.22 (d, *J* = 18.0 Hz, 1H), 2.36 (s, 6H); ^{13}C{^{1}H} NMR (150 MHz, $CDCl_3$): δ = 197.4, 169.7, 151.6, 140.7, 138.5, 136.4, 135.6 132.5, 129.9, 129.0, 128.5, 127.6, 125.8, 124.6, 124.4, 122.2, 64.5, 46.8, 21.2; IR (neat, cm^{-1}): 3426, 2917, 1689, 1662, 1373, 1294, 1190, 1159, 853, 749, 693; HRMS (ESI): Exact mass calcd for $C_{24}H_{21}NO_2$ $[M+Na]^+$: 378.1465, Found: 378.1476.



3al

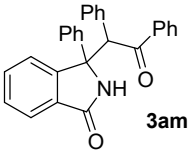
Column chromatography afforded **3al** in 31% yield (22.1 mg) as white solid; Mp: 222-224 °C; ^{1}H NMR (600 MHz, $CDCl_3$): δ = 7.91-7.90 (m, 2H), 7.86 (d, *J* = 7.8 Hz, 1H), 7.50 (s,

1H), 7.59-7.56 (m, 1H), 7.56-7.52 (m, 2H), 7.49-7.48 (m, 2H), 7.47-7.44 (m, 3H), 7.15-7.13 (m, 2H), 7.09-7.07 (m, 1H), 4.74 (dd, *J* = 9.0 Hz, 4.2 Hz, 1H), 1.70-1.63 (m, 2H), 0.70 (t, *J* = 7.8 Hz, 3H); $^{13}C\{^{1}H\}$ NMR (150 MHz, $CDCl_3$): δ = 204.0, 170.2, 149.9, 141.6, 138.0, 133.6, 132.4, 131.0, 128.80, 128.76, 128.6, 128.2, 127.6, 124.7, 124.2, 122.0, 68.8, 53.2, 22.1, 13.0; IR (neat, cm^{-1}): 3413, 2967, 1691, 1661, 1446, 1373, 1220, 770, 748, 694; HRMS (ESI): Exact mass calcd for $C_{24}H_{21}NO_2$ $[M+Na]^+$: 378.1465, Found: 378.1476.



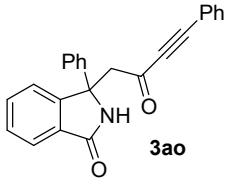
3al'

Column chromatography afforded the desired product **3al'** in 34% yield (24.2 mg) as white solid; Mp: 155-157 °C; ^{1}H NMR (600 MHz, $CDCl_3$): δ = 7.67 (d, *J* = 7.8 Hz, 1H), 7.64-7.63 (m, 2H), 7.51-7.50 (m, 2H), 7.41-7.35 (m, 4H), 7.31 (d, *J* = 7.8 Hz, 1H), 7.28-7.25 (m, 3H), 7.19-7.16 (m, 1H), 7.08-7.05 (m, 1H), 4.68 (dd, *J* = 11.4 Hz, 1.8 Hz, 1H), 2.01-1.93 (m, 1H), 1.62-1.56 (m, 1H), 0.90 (t, *J* = 7.2 Hz, 3H); $^{13}C\{^{1}H\}$ NMR (150 MHz, $CDCl_3$): δ = 200.9, 170.5, 148.0, 140.8, 140.0 132.4, 131.4 130.6, 129.1, 128.3, 128.2, 128.0, 127.2, 125.4, 123.9, 123.7, 68.8, 54.4, 22.4, 12.9; IR (neat, cm^{-1}): 3058, 2936, 2858, 1693, 1678, 1350, 1267, 766, 744, 699; HRMS (ESI): Exact mass calcd for $C_{24}H_{21}NO_2$ $[M+Na]^+$: 378.1465, Found: 378.1476.



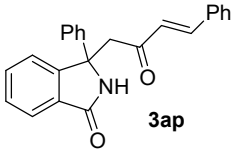
3am

Column chromatography afforded the desired product **3am**[2] in 57% yield (46.0 mg) as white solid; Mp: 259-261 °C; ^{1}H NMR (600 MHz, $CDCl_3$): 7.89-7.88 (m, 3H), 7.56-7.55 (m, 2H), 7.51-7.46 (m, 4H), 7.39-7.36 (m, 2H), 7.29-7.25 (m, 3H), 7.22-7.20 (m, 1H), 7.07-7.03 (m, 3H), 7.01-7.00 (m, 2H), 6.02 (s, 1H).



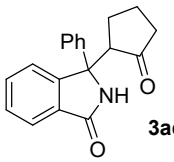
3ao

Column chromatography afforded the desired product **3ao** in 82% yield (57.7 mg) as pale yellow solid; Mp: 135-137 °C; ^{1}H NMR (600 MHz, $CDCl_3$): δ = 7.87 (d, *J* = 7.8 Hz, 1H), 7.56-7.55 (m, 2H), 7.52-7.47 (m, 2H), 7.45-7.42 (m, 3H), 7.41-7.38 (m, 3H), 7.35-7.32 (m, 3H), 7.27-7.25 (m, 1H), 4.29 (d, *J* = 18.0 Hz, 1H), 3.12 (d, *J* = 18.0 Hz, 1H); $^{13}C\{^{1}H\}$ NMR (150 MHz, $CDCl_3$): δ = 183.8, 169.6, 150.8, 133.2, 132.6, 131.3, 129.8, 129.1, 128.7, 128.6, 127.9, 124.8, 124.4, 122.1, 119.2, 92.8, 87.8, 64.4, 53.3; IR (neat, cm^{-1}): 3197, 2933, 2205, 1695, 1655, 1315, 1274, 749, 701, 686; HRMS (ESI): Exact mass calcd for $C_{24}H_{17}NO_2$ $[M+Na]^+$: 374.1151, Found: 374.1159.



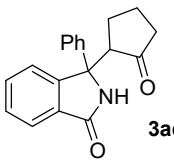
3ap

Column chromatography afforded the desired product **3ap**[2] in 99% yield (70.6 mg) as white solid; Mp: 224-226 °C; ^{1}H NMR (600 MHz, $CDCl_3$): δ = 7.87 (d, *J* = 7.8 Hz, 1H), 7.66 (s, 1H), 7.58 (d, *J* = 16.2 Hz, 1H), 7.54-7.53 (m, 2H), 7.52-7.50 (m, 1H), 7.45-7.38 (m, 6H), 7.35 (d, *J* = 7.8 Hz, 1H), 7.31-7.29 (m, 2H), 7.24-7.21 (m, 1H), 6.75 (d, *J* = 16.2 Hz, 1H), 4.35 (d, *J* = 18.0 Hz, 1H), 3.00 (d, *J* = 18.0 Hz, 1H).



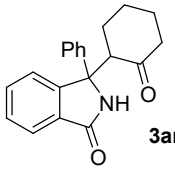
3aq

Column chromatography afforded product **3aq** in 61% yield (35.8 mg) as white solid; Mp: 160-162 °C; ^{1}H NMR (600 MHz, $CDCl_3$): δ = 7.81 (d, *J* = 7.8 Hz, 1H), 7.50-7.47 (m, 1H), 7.46-7.44 (m, 2H), 7.42-7.39 (m, 1H), 7.35-7.32 (m, 2H), 7.30-7.26 (m, 2H), 6.55 (s, 1H), 3.71 (dd, *J* = 12.6 Hz, 7.8 Hz, 1H), 2.41-2.37 (m, 1H), 2.17-2.10 (m, 1H), 1.96-1.92 (m, 1H), 1.83-1.78 (m, 1H), 1.63-1.59 (m, 1H), 1.38-1.33 (m, 1H); ^{13}C{^{1}H} NMR (150 MHz, $CDCl_3$): δ = 216.8, 170.5, 151.0, 140.7, 132.7, 129.9, 128.8, 128.3, 127.7, 125.1, 124.1, 122.3, 66.0, 54.6, 38.1, 25.5, 19.4; IR (neat, cm^{-1}): 3308, 2908, 1720, 1696, 1469, 1306, 1273, 1150, 746, 701; HRMS (ESI): Exact mass calcd for $C_{19}H_{17}NO_2$ $[M+Na]^+$: 314.1151, Found: 314.1163.



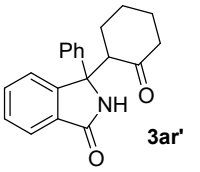
3aq'

Column chromatography afforded **3aq'** in 27% yield (15.7 mg) as white solid; Mp: 178-180 °C; ^{1}H NMR (600 MHz, $CDCl_3$): ^{1}H NMR (600 MHz, $CDCl_3$): δ = 8.55 (s, 1H), 7.83 (d, *J* = 7.2 Hz, 1H), 7.57-7.54 (m, 1H), 7.52-7.51 (m, 1H), 7.48-7.45 (m, 1H), 7.44-7.42 (m, 2H), 7.34-7.31 (m, 2H), 7.27-7.25 (m, 1H), 3.13 (dd, *J* = 11.4 Hz, 9.0 Hz, 1H), 2.35-2.31 (m, 1H), 2.29-2.25 (m, 1H), 2.15-2.10 (m, 1H), 2.03-1.98 (m, 1H), 1.94-1.87 (m, 1H), 1.76-1.68 (m, 1H); ^{13}C{^{1}H} NMR (150 MHz, $CDCl_3$): δ = 216.6, 170.3, 150.0, 139.8, 132.1, 131.2, 128.8 128.4, 127.8, 126.2, 124.1, 123.3, 67.5, 55.9, 39.5, 27.3, 20.2; IR (neat, cm^{-1}): 3301, 2935, 1711, 1670, 1480, 1331, 1260, 1130, 738, 698; HRMS (ESI): Exact mass calcd for $C_{19}H_{17}NO_2$ $[M+Na]^+$: 314.1151, Found: 314.1163.



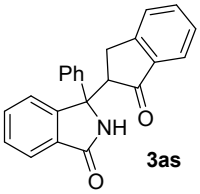
3ar

Column chromatography afforded the desired product **3ar**[2] in 81% yield (50.0 mg) as white solid; Mp: 240-242 °C; ^{1}H NMR (600 MHz, $CDCl_3$): δ = 7.82 (d, *J* = 7.2 Hz, 1H), 7.48-7.46 (m, 1H), 7.44-7.42 (m, 2H), 7.40-7.38 (m, 1H), 7.31-7.29 (m, 2H), 7.26-7.24 (m, 1H), 7.22-7.20 (m, 1H), 7.15 (s, 1H), 3.88 (dd, *J* = 12.0 Hz, 4.8 Hz, 1H), 2.50-2.45 (m, 1H), 2.40-2.38 (m, 1H), 2.15-2.12 (m, 1H), 1.81-1.79 (m, 1H), 1.72-1.60 (m, 2H), 1.51-1.42 (m, 2H).



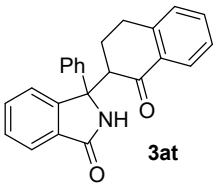
3ar'

Column chromatography afforded the desired product **3ar'**[2] in 13% yield (8.1 mg) as white solid; Mp: 205-207 °C; ^{1}H NMR (600 MHz, $CDCl_3$): ^{1}H NMR (600 MHz, $CDCl_3$): δ = 7.85 (d, *J* = 7.8 Hz, 1H), 7.77 (s, 1H), 7.57 (d, *J* = 7.8 Hz, 1H), 7.55-7.52 (m, 1H), 7.48-7.45 (m, 1H), 7.36-7.34 (m, 2H), 7.30-7.27 (m, 2H), 7.24-7.22 (m, 1H), 3.46 (dd, *J* = 12.6 Hz, 4.8 Hz, 1H), 2.35-2.29 (m, 2H), 2.19-2.16 (m, 1H), 2.05-2.02 (m, 1H), 1.87-1.85 (m, 1H), 1.69-1.58 (m, 2H), 1.42-1.35 (m, 1H).



3as

Column chromatography afforded product **3as** as a mixture with a 2:1 dr value in 77% yield (52.1 mg) as white solid; Mp: 240-242°C; ^{1}H NMR (600 MHz, $CDCl_3$): δ = 7.87 (d, *J* = 7.2 Hz, 0.57H), 7.84 (d, *J* = 7.2 Hz, 1H), 7.73 (s, 0.55H), 7.68 (d, *J* = 7.2 Hz, 0.56H), 7.64 (d, *J* = 7.8 Hz, 1H), 7.58-7.48 (m, 6.49H), 7.46-7.44 (m, 2H), 7.42 (d, *J* = 8.4 Hz, 0.60H), 7.39-7.37 (m, 2H), 7.34-7.28 (m, 6H), 6.58 (s, 0.92H), 4.19 (dd, *J* = 7.8 Hz, 5.4 Hz, 1H), 3.60 (s, 0.55H), 3.45 (dd, *J* = 17.4 Hz, 8.4 Hz, 0.57H), 3.15 (dd, *J* = 17.4 Hz, 3.6 Hz, 0.51H), 2.95 (dd, *J* = 17.4 Hz, 7.8 Hz, 1H), 2.43 (dd, *J* = 16.8 Hz, 5.4 Hz, 1H); ^{13}C{^{1}H} NMR (150 MHz, $CDCl_3$): δ = 204.0, 203.8, 170.4, 169.8, 152.6, 152.1, 150.7, 150.0, 135.2, 132.8, 129.0, 128.9, 128.5, 127.80, 127.77, 126.3, 126.1, 125.3, 124.25, 124.22, 124.0, 122.4, 68.1, 67.2, 54.0, 52.5, 31.2, 29.6; IR (neat, cm^{-1}): 3225, 2922, 1687, 1607, 1466, 1343, 1211, 1151, 738, 699, 642; HRMS (ESI): Exact mass calcd for $C_{23}H_{17}NO_2$ $[M+Na]^+$: 362.1151, Found: 362.1157.

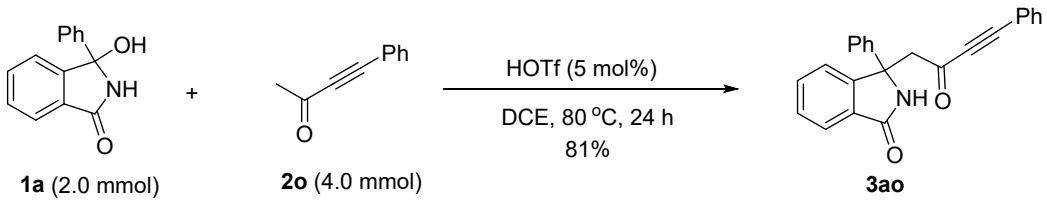


3at

Column chromatography afforded **3at**[4] in 70% yield (48.8 mg) as white solid; Mp: 240-242°C; ^{1}H NMR (600 MHz, $CDCl_3$): δ = 7.85-7.84 (m, 2H), 7.53-7.51 (m, 3H), 7.49-7.47 (m, 1H), 7.44-7.42 (m, 1H), 7.35-7.31 (m, 3H), 7.29-7.26 (m, 1H), 7.26-7.23 (m, 1H), 7.22 (d, *J* = 7.8 Hz, 1H), 6.97 (s, 1H), 4.09 (dd, *J* = 13.2 Hz, 3.6 Hz, 1H), 3.15-3.09 (m, 1H), 2.94-2.91 (m, 1H), 1.95-1.87 (m, 1H), 1.54-1.52 (m, 1H).

3. Scaled synthesis and product elaboration

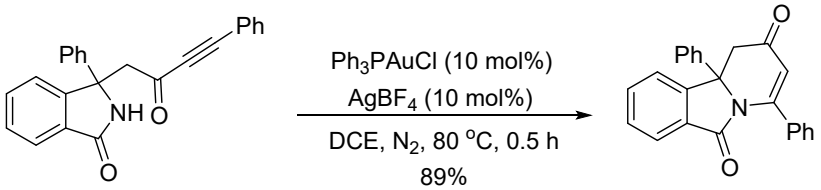
3.1 Scaled synthesis



1a (2.0 mmol) **2o** (4.0 mmol) **3ao**

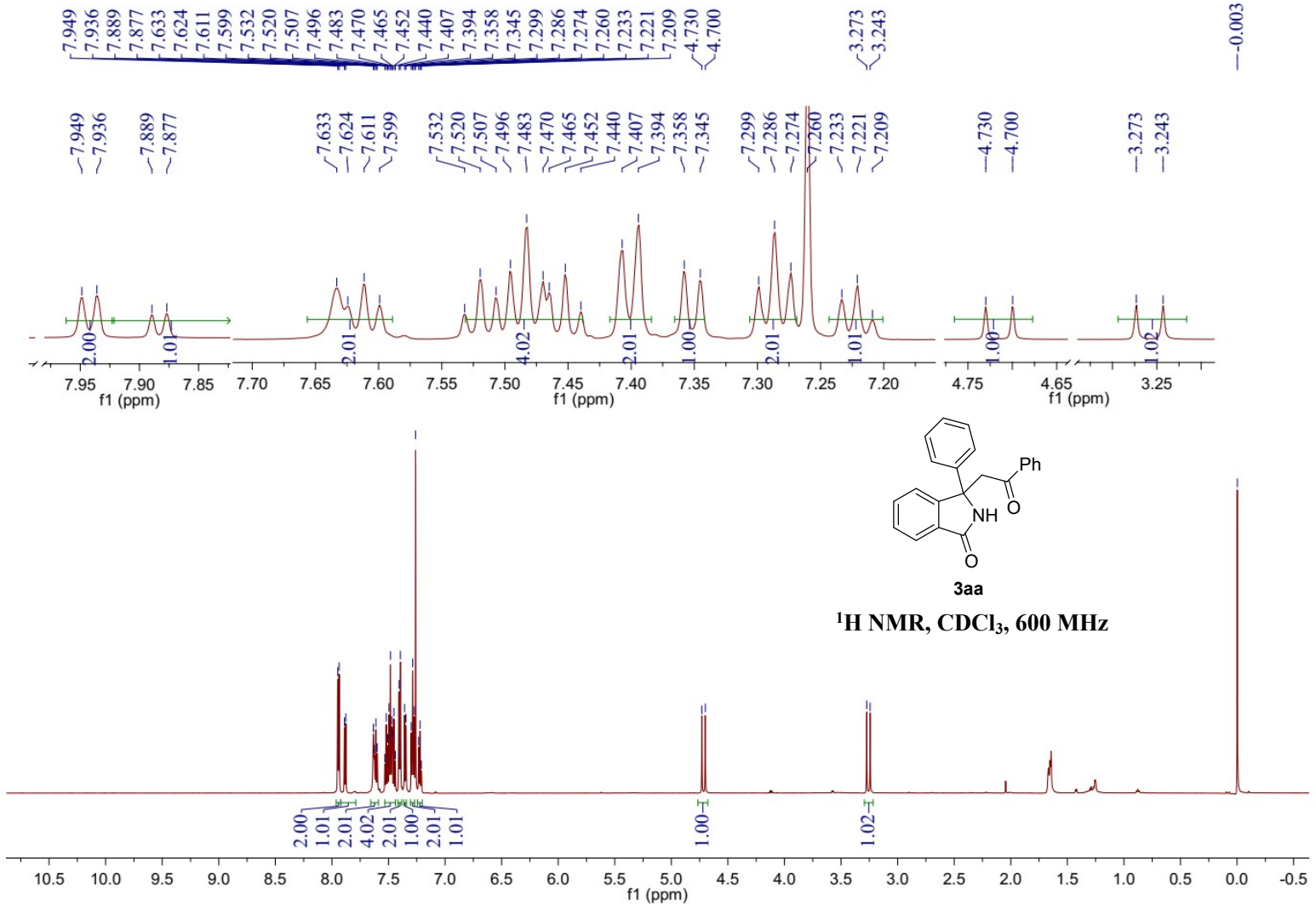
To a 100 mL of sealed tube were added **1a** (0.45 g, 2.0 mmol, 1.0 equiv), **2o** (0.58 mL, 4.0 mmol, 2.0 equivs) and 20.0 mL of anhydrous DCE. After adding HOTf (15 mg, 5 mol%), the reaction mixture was stirred at 80 °C till almost full conversion of **1a** by TLC analysis. The residue was directly subjected to column chromatography using petroleum ether/ethyl acetate (10:1, v:v) as the eluent to afford the desired product **3ao** in 81% yield (0.57 g) as pale yellow solid.

3.2 Product elaboration



3ao **5**

Under the N_2 atmosphere, to a 25 mL dried Schlenk tube were sequentially added **3ao** (35.1 mg, 0.1 mmol), Ph_3PAuCl (5.0 mg, 0.01 mmol), $AgBF_4$ (2.0 mg, 0.01 mmol) and 1.0 mL of anhydrous DCE. Then the reaction mixture was stirred at 80 °C till almost full conversion of **3ao** by TLC analysis. The reaction mixture was directly subjected to column chromatography using petroleum ether/ethyl acetate (generally 8:1, v:v) as the eluent to afford **5** in 89% yield (31.4 mg) as white solid; Mp: 184-186°C; ^{1}H NMR (600 MHz, $CDCl_3$): δ = 7.94 (d, *J* = 7.2 Hz, 1H), 7.61-7.58 (m, 1H), 7.54-7.51 (m, 3H), 7.50-7.47 (m, 1H), 7.44-7.40 (m, 4H), 7.38-7.35 (m, 2H), 7.32-7.30 (m, 1H), 7.20 (d, *J* = 7.8 Hz, 1H), 5.77 (s, 1H), 3.75 (d, *J* = 16.2 Hz, 1H), 2.87 (d, *J* = 16.2 Hz, 1H); ^{13}C{^{1}H} NMR (150 MHz, $CDCl_3$): δ = 193.5, 165.9, 152.7, 150.8, 131.1, 129.3, 129.1 128.4, 127.6, 126.3, 122.4, 113.4, 70.2, 46.7; IR (neat, cm^{-1}): 2918, 1721, 1664, 1568, 1446, 1375, 1254, 1183, 753, 729, 702, 681; HRMS (ESI): Exact mass calcd for $C_{24}H_{17}NO_2$ $[M+H]^+$: 374.1151, Found: 374.1161.


7.949
7.936
7.889
7.877
7.633
7.624
7.611
7.599
7.532
7.520
7.507
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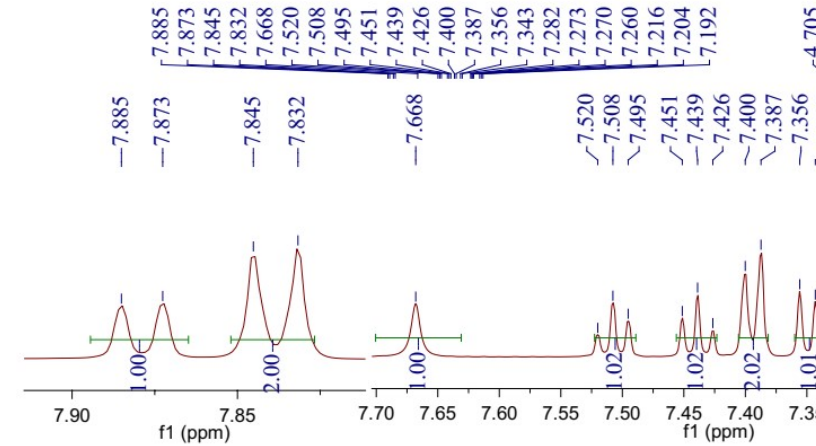
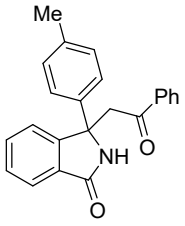
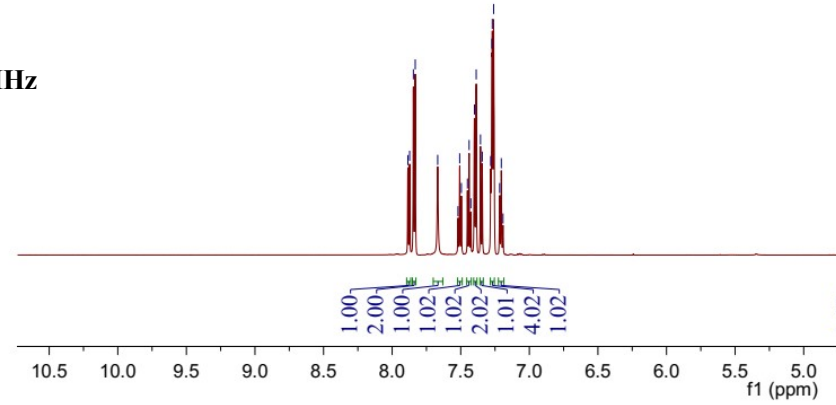
7.470
7.465
7.452
7.440
7.407
7.394
7.358
7.345
7.299
7.286
7.274
7.260
7.233
7.221
7.209
4.730
4.700
3.273
3.243
-0.003
f1 (ppm)
Ph
O
NH
O
3aa
1H NMR, CDCl3, 600 MHz

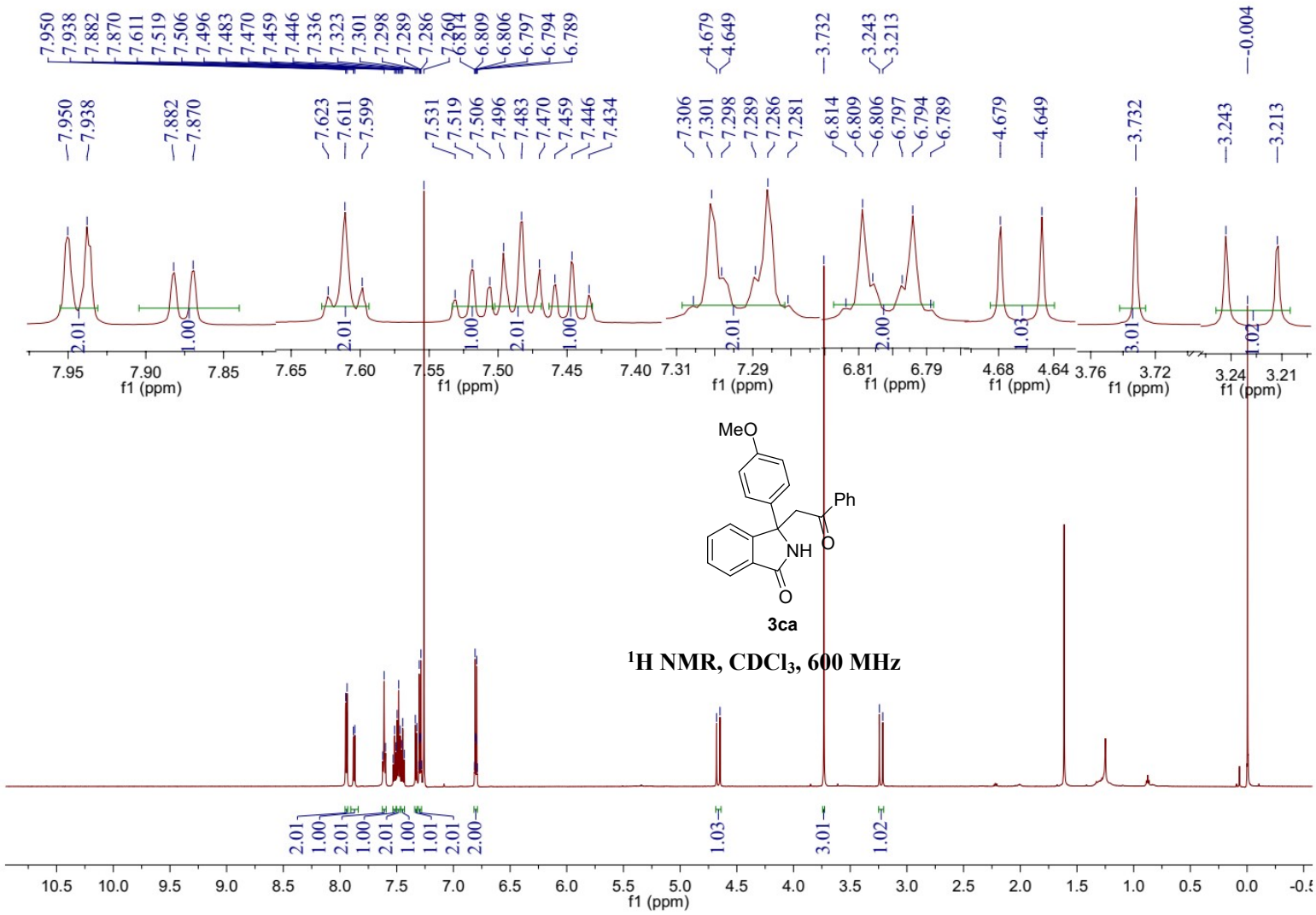


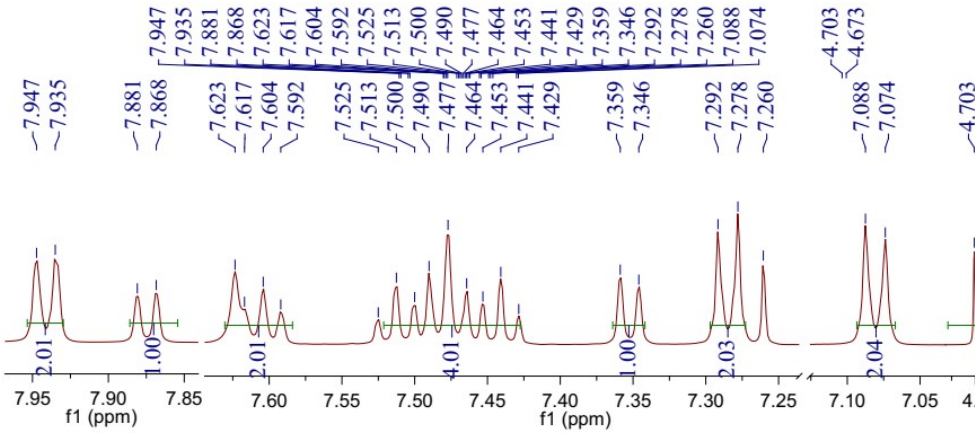


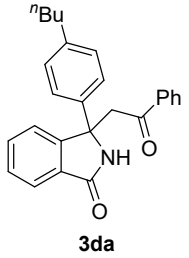
3ba

^{1}H NMR, $CDCl_3$, 600 MHz



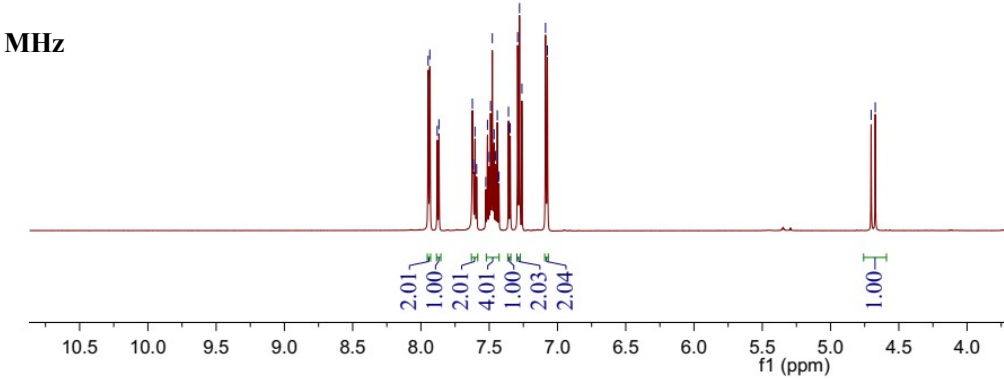

MeO
Ph
O
NH
O
3ca
^{1}H NMR, $CDCl_3$, 600 MHz
f1 (ppm)

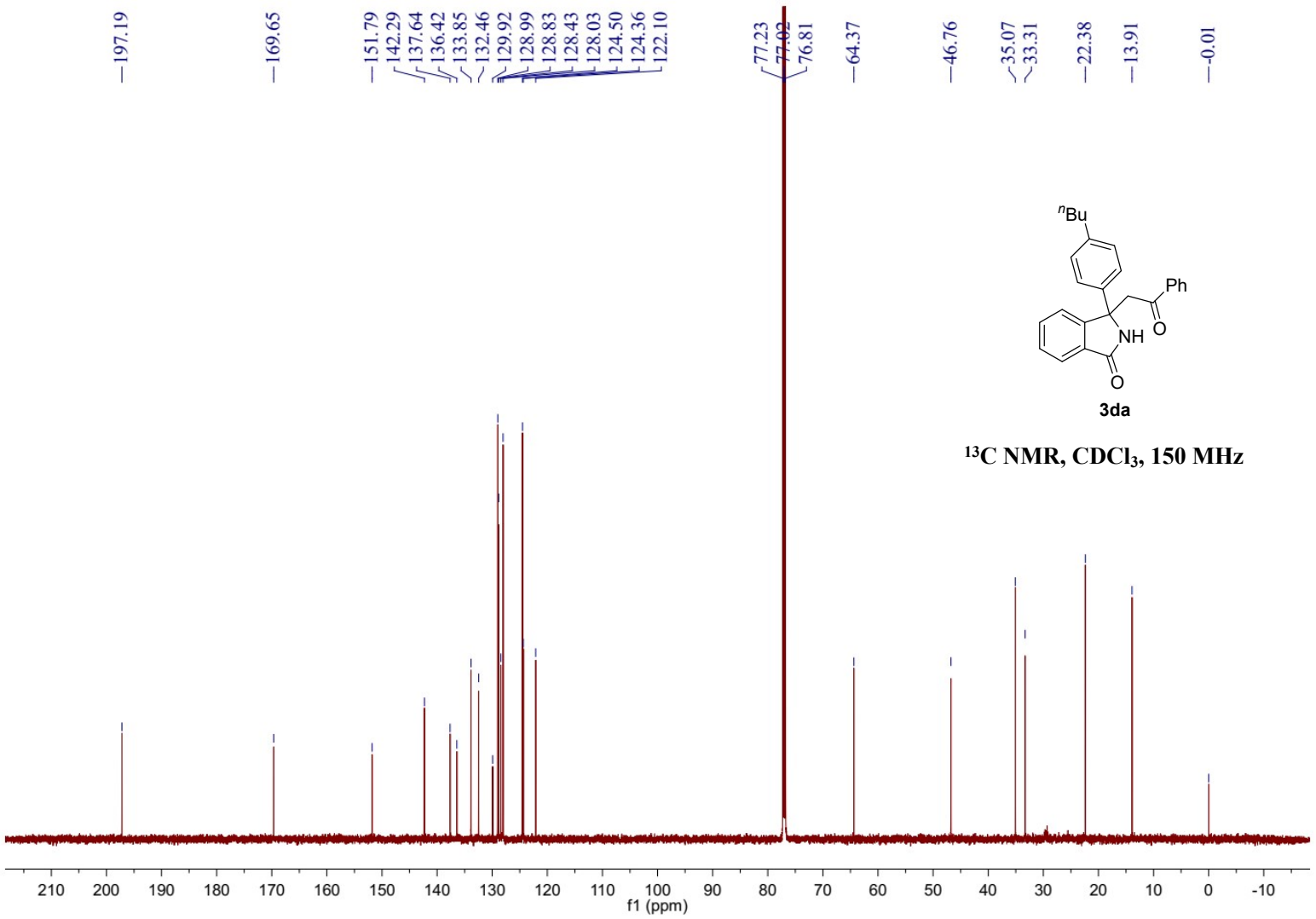




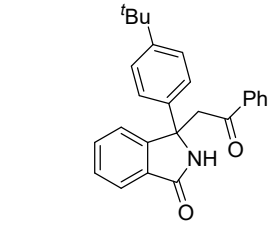
3da

^{1}H NMR, $CDCl_3$, 600 MHz



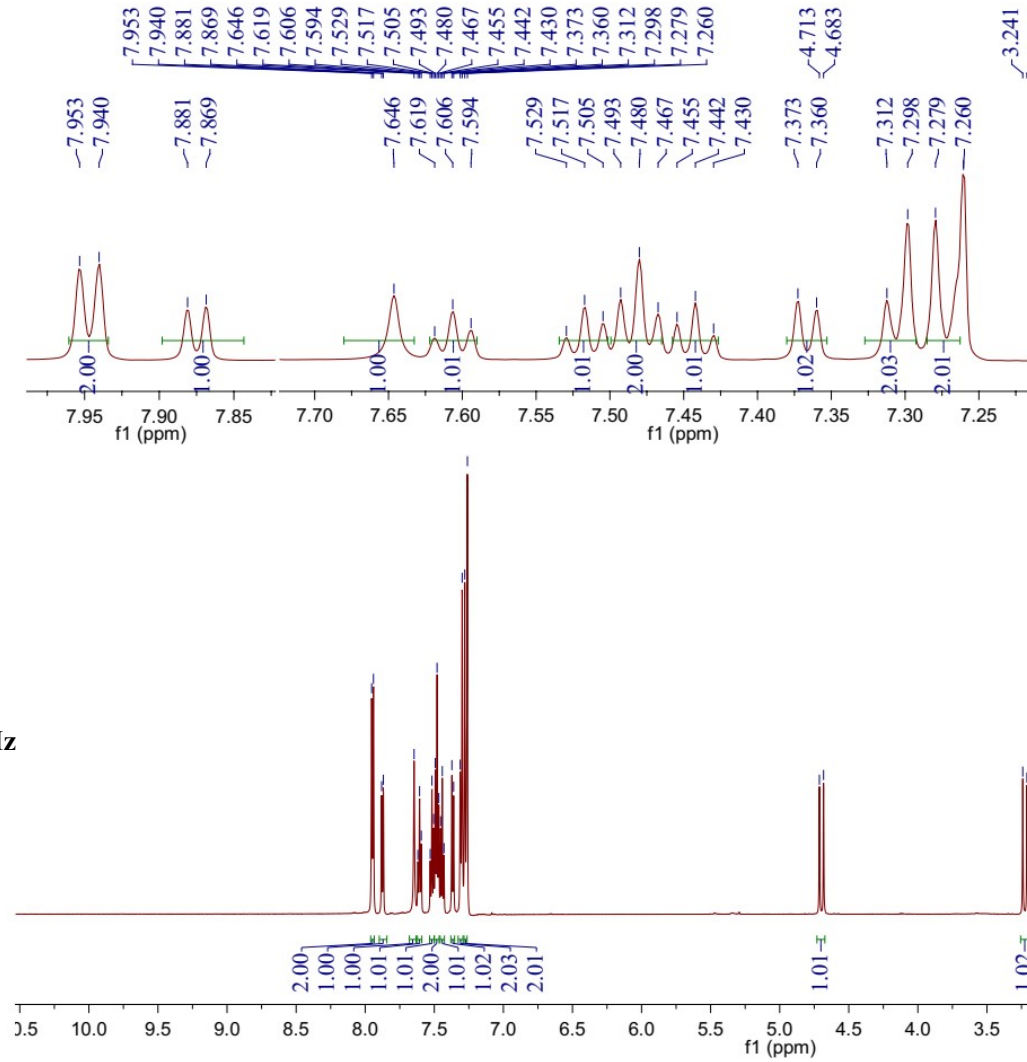


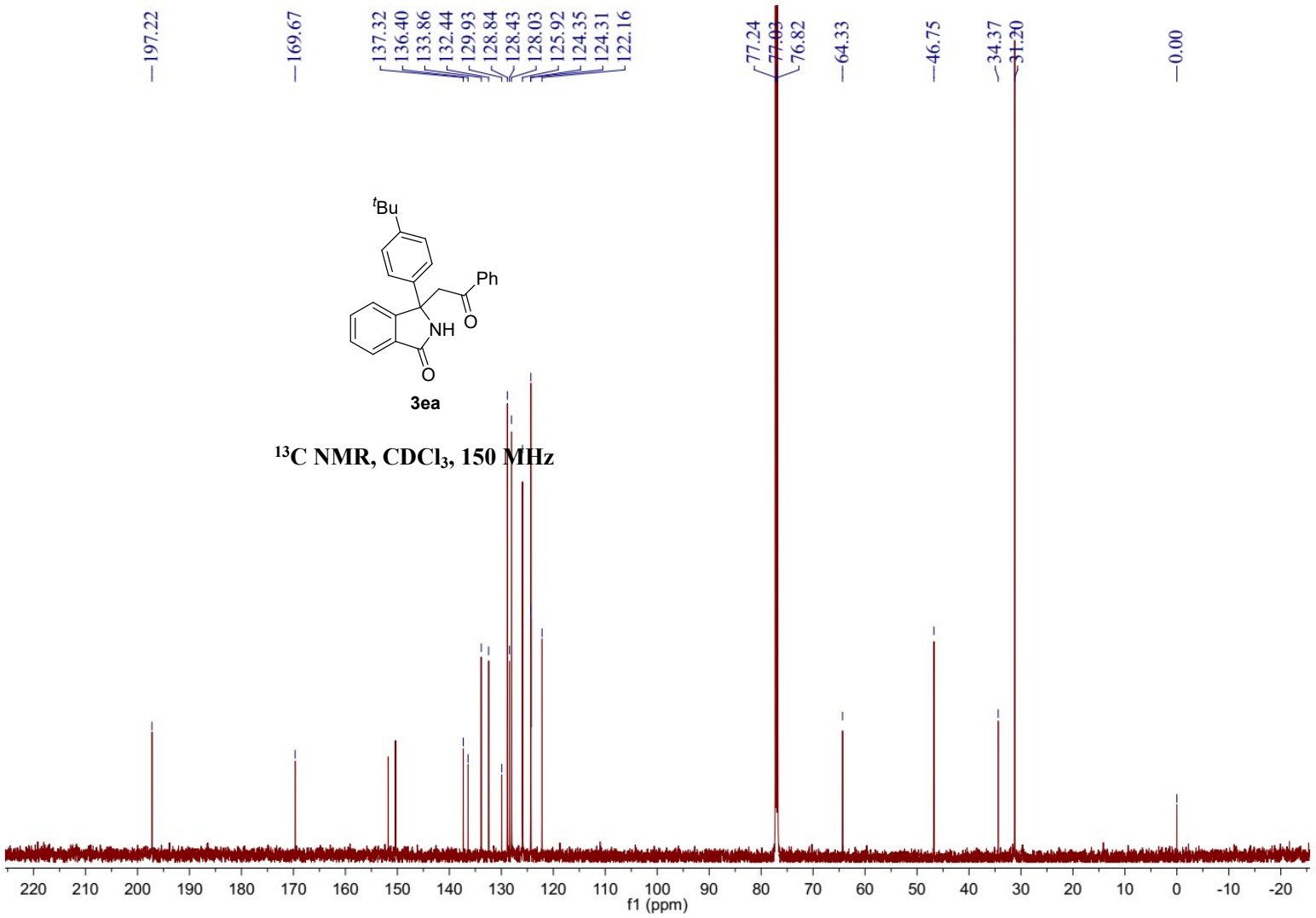
^{13}C NMR, $CDCl_3$, 150 MHz



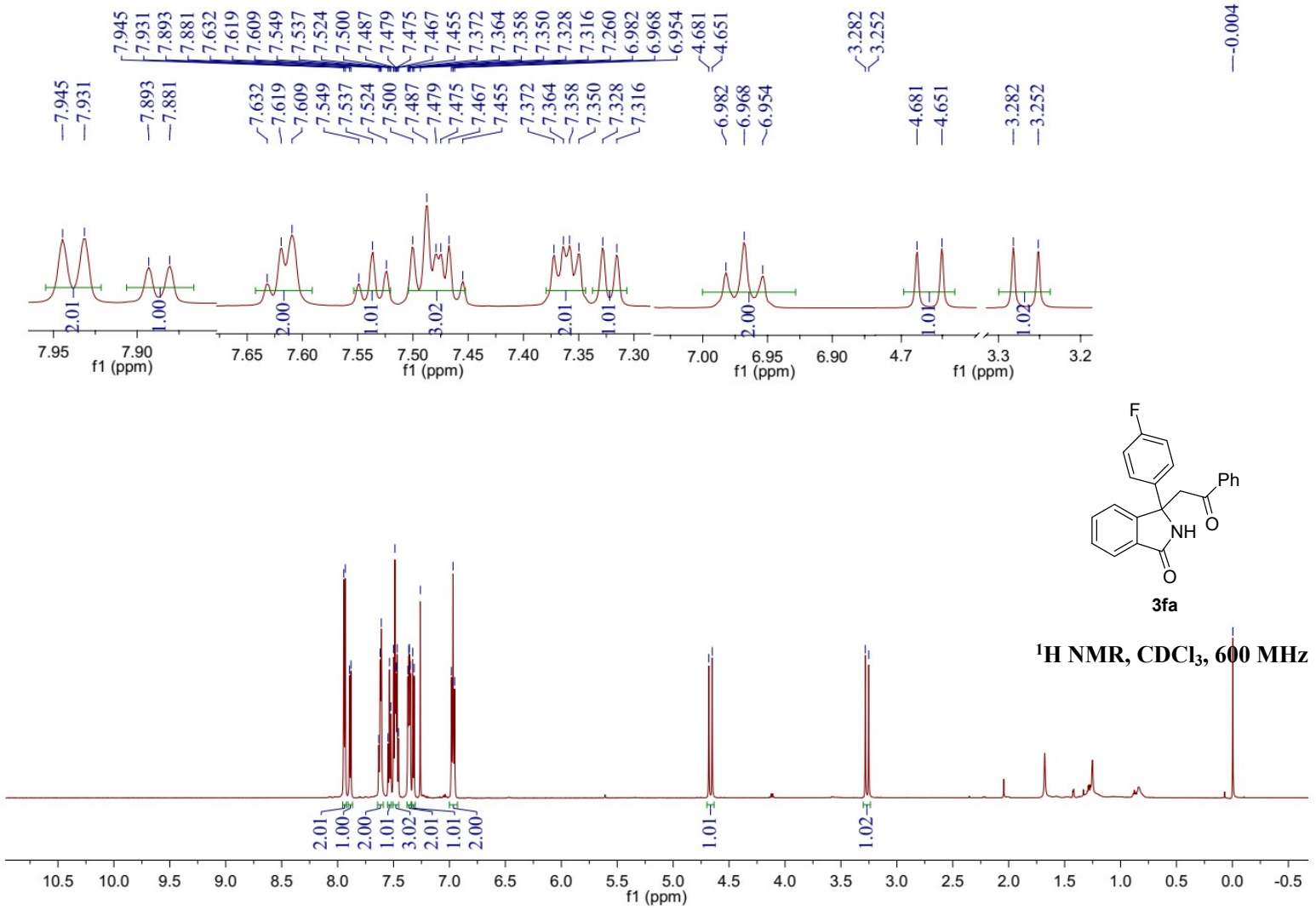
3ea

^{1}H NMR, $CDCl_3$, 600 MHz





^{13}C NMR, $CDCl_3$, 150 MHz


7.945
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7.881
7.632
7.619
7.609
7.549
7.537
7.524
7.500
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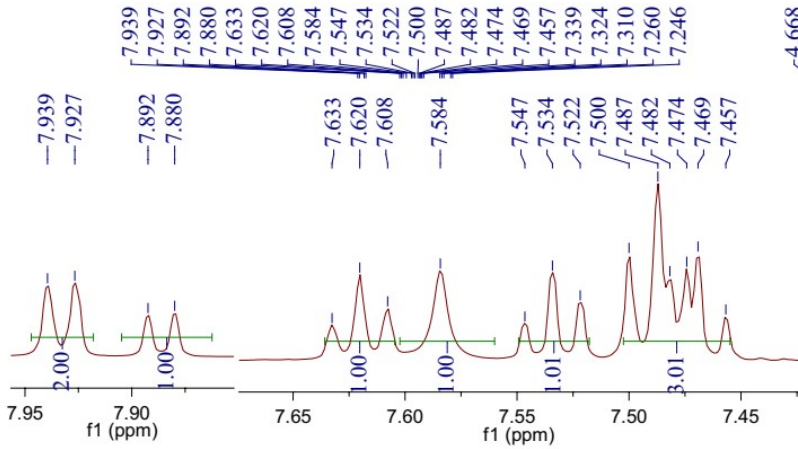
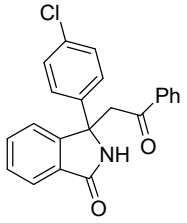
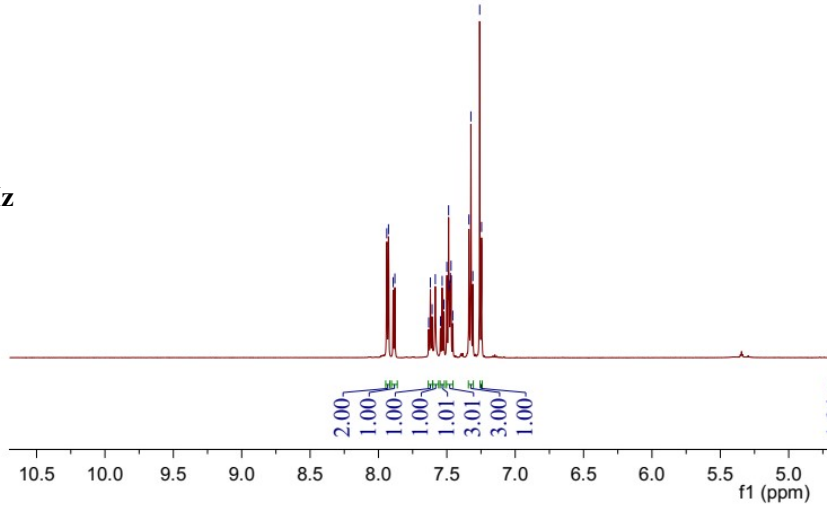
7.479
7.475
7.467
7.455
7.372
7.364
7.358
7.350
7.328
7.316
7.260
6.982
6.968
6.954
4.681
4.651
3.282
3.252
-0.004
2.01
1.00
2.00
1.01
3.02
2.01
1.01
2.00
1.01
1.02
f1 (ppm)
F
Ph
O
NH
O
3fa
1H NMR, CDCl3, 600 MHz

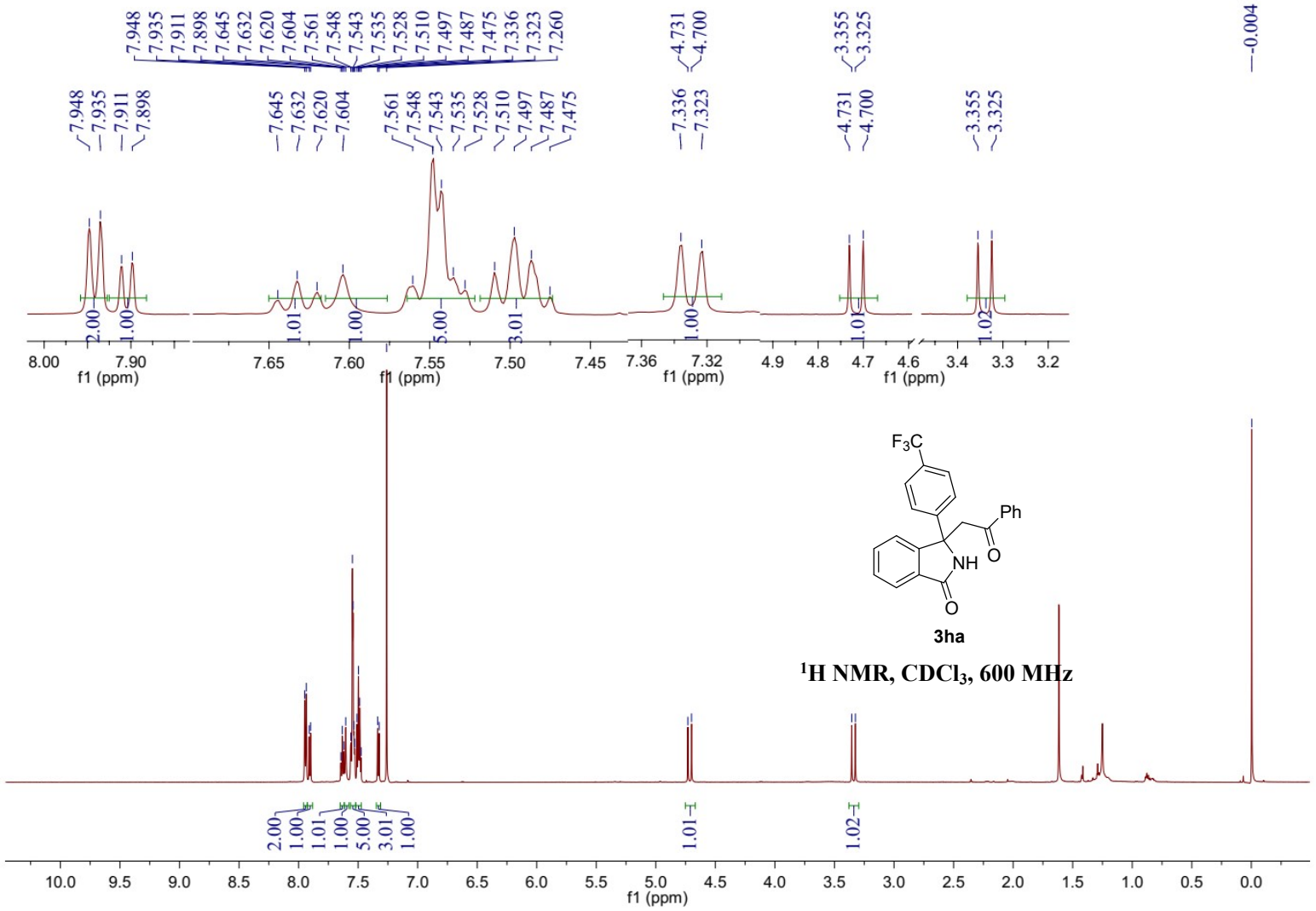




3ga

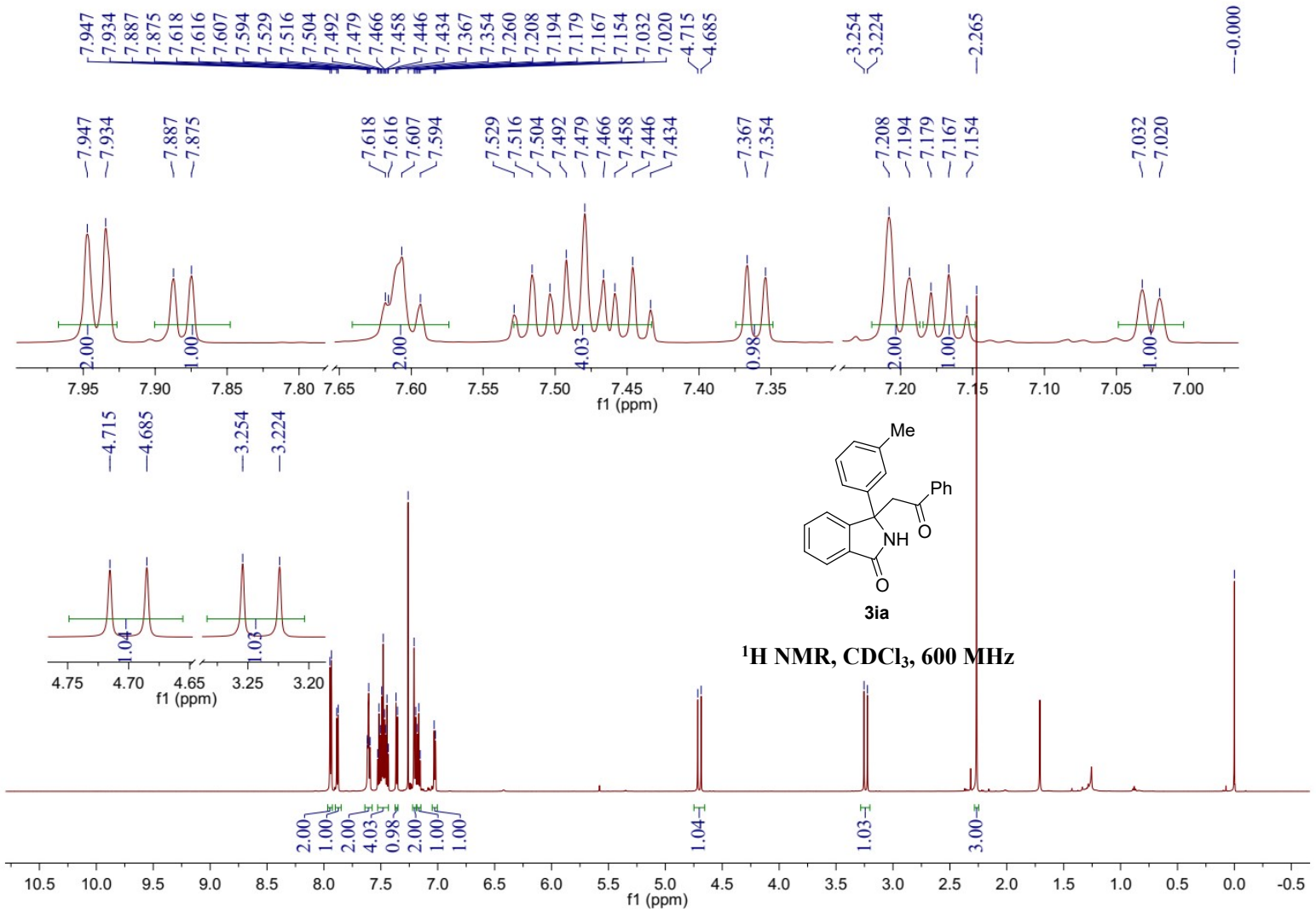
^{1}H NMR, $CDCl_3$, 600 MHz

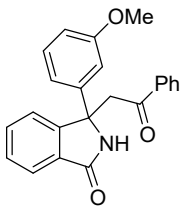




3ha

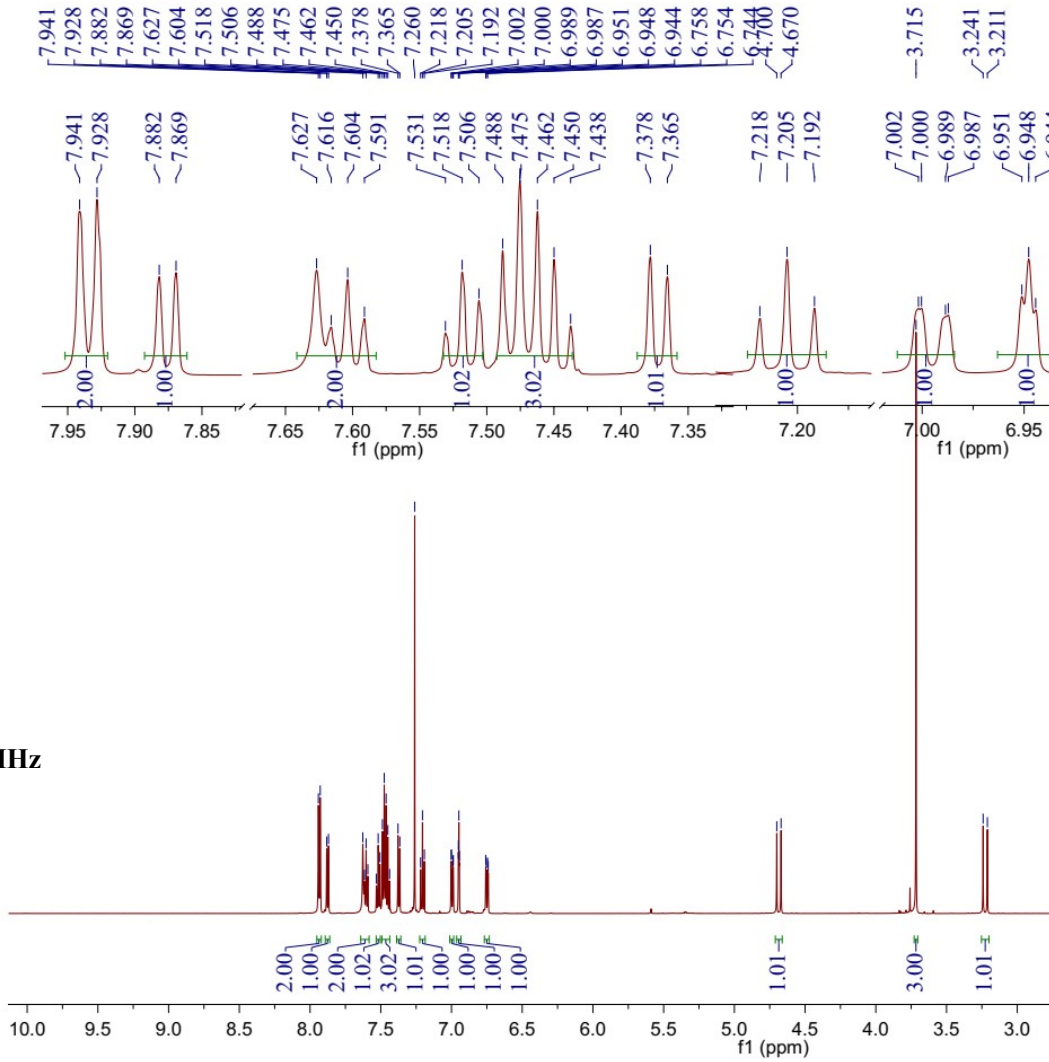
^{1}H NMR, $CDCl_3$, 600 MHz

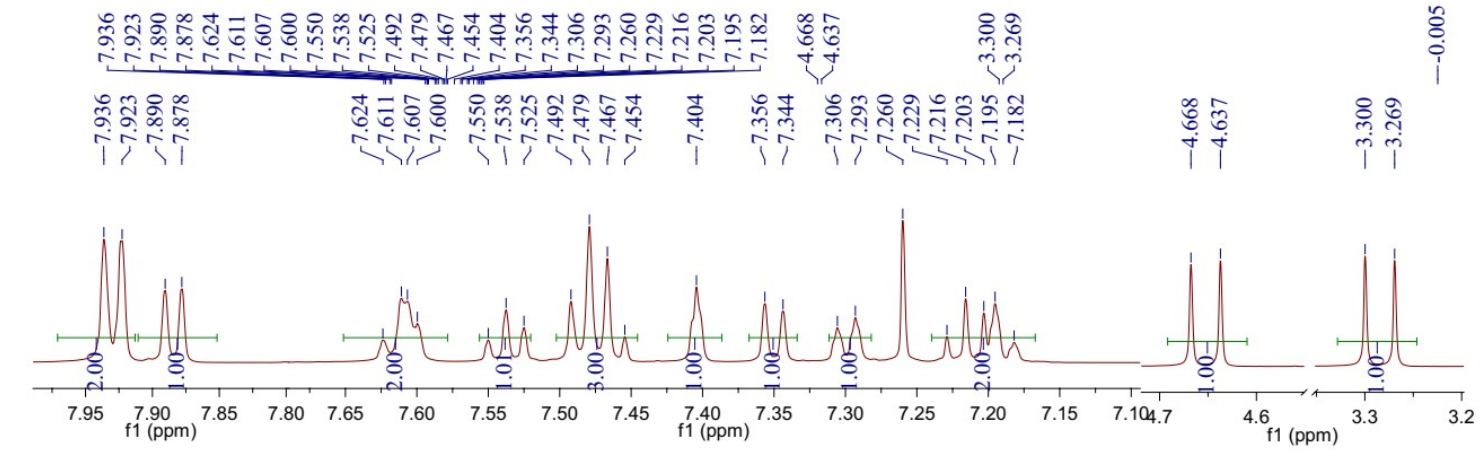

Me
Ph
O
NH
O
3ia
1H NMR, CDCl3, 600 MHz
f1 (ppm)

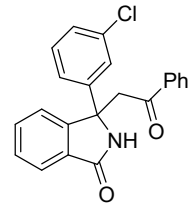


3ja

^{1}H NMR, $CDCl_3$, 600 MHz

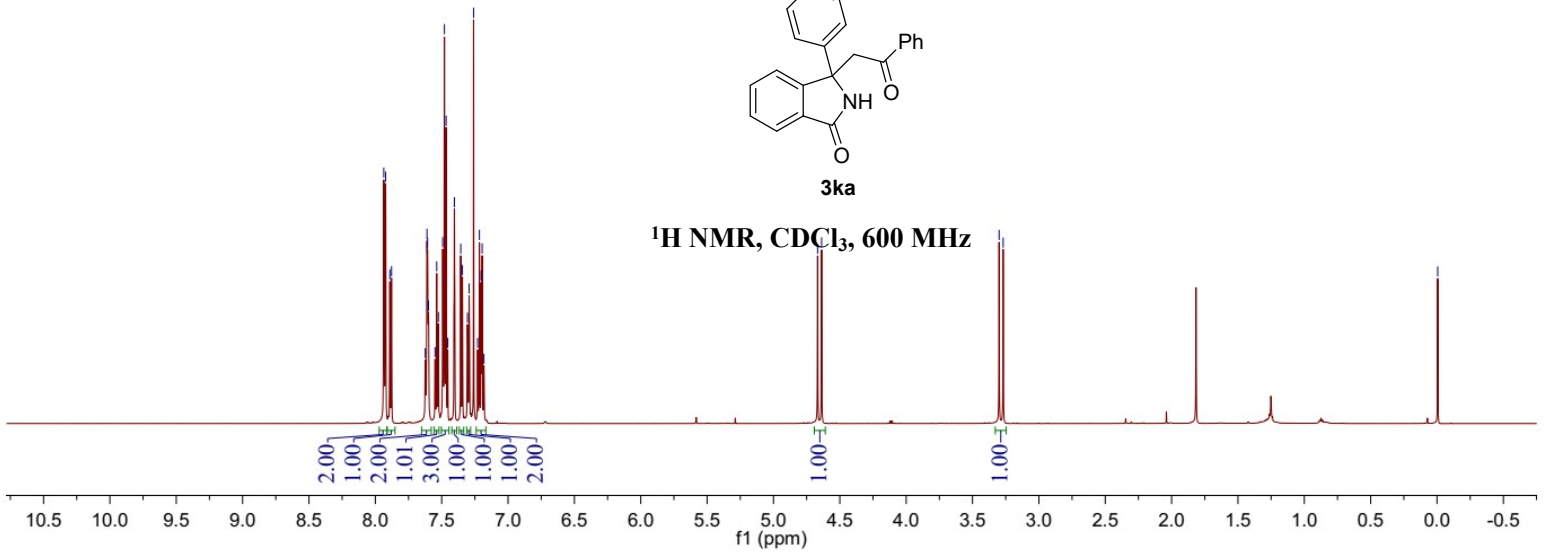


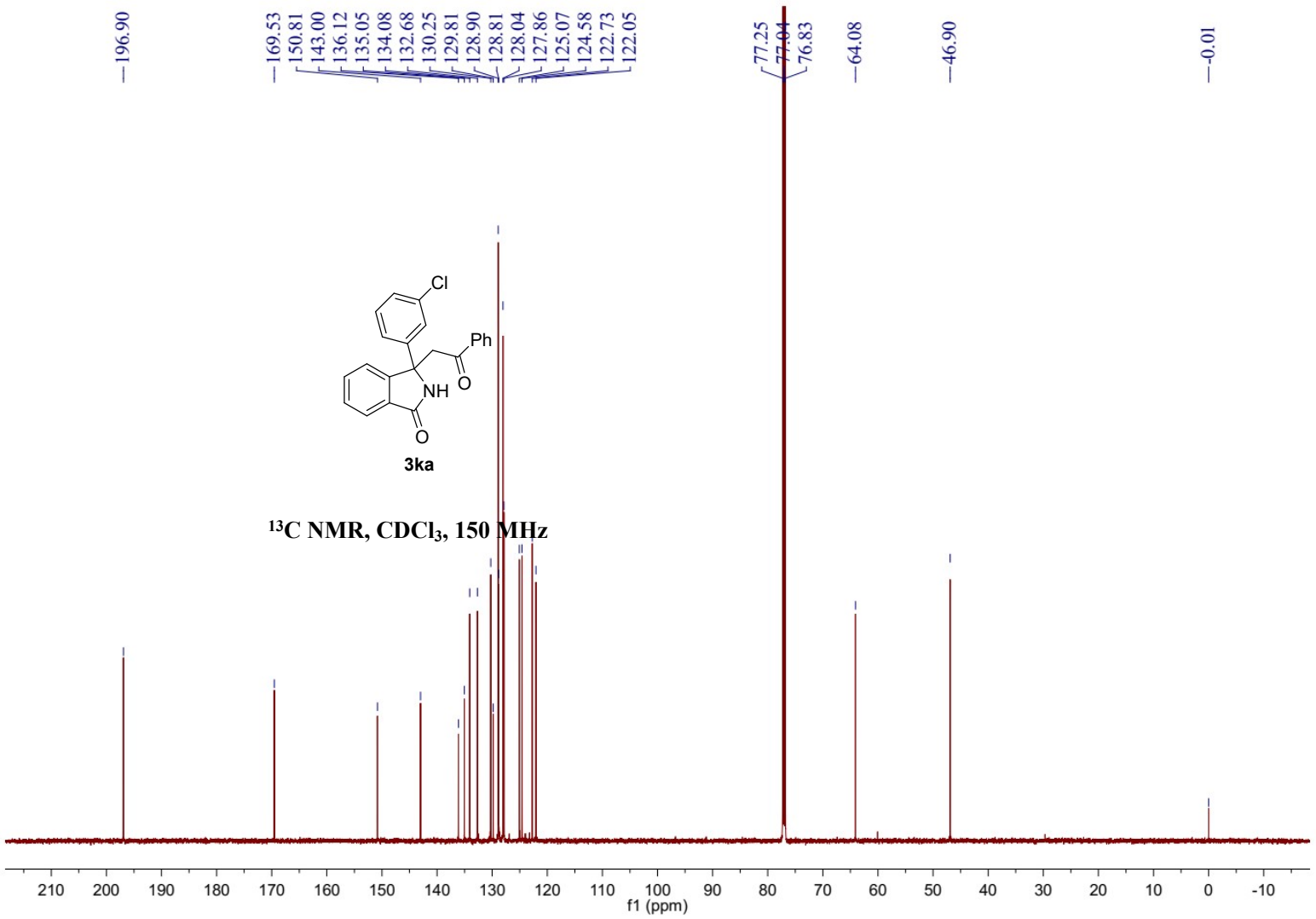




3ka

^{1}H NMR, $CDCl_3$, 600 MHz

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169.53
150.81
143.00
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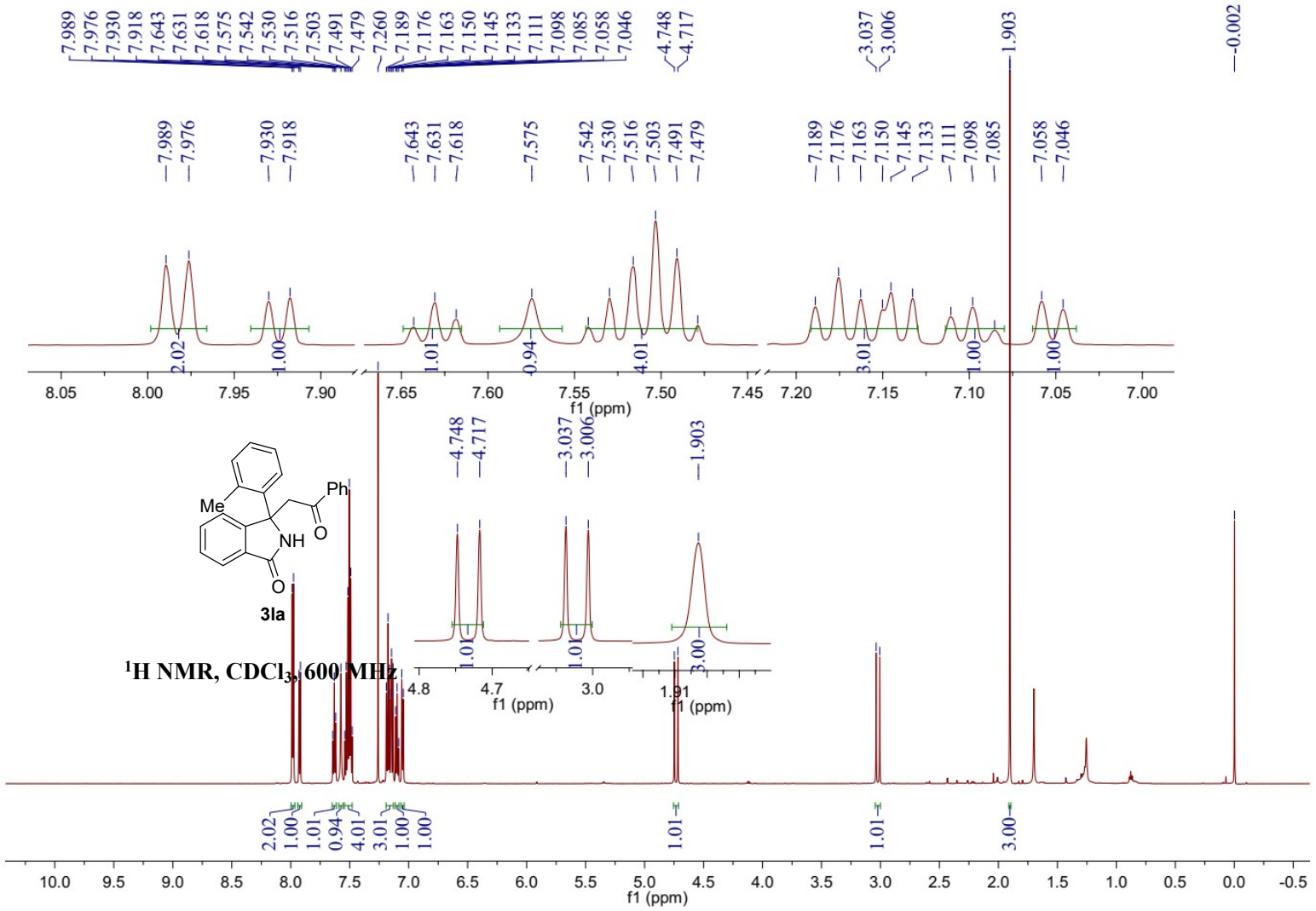
135.05
134.08
132.68
130.25
129.81
128.90
128.81
128.04
127.86
125.07
124.58
122.73
122.05
77.25
77.04
76.83
64.08
46.90
-0.01
Cl
Ph
O
NH
O
3ka
13C NMR, CDCl3, 150 MHz
210
200
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
-10
f1 (ppm)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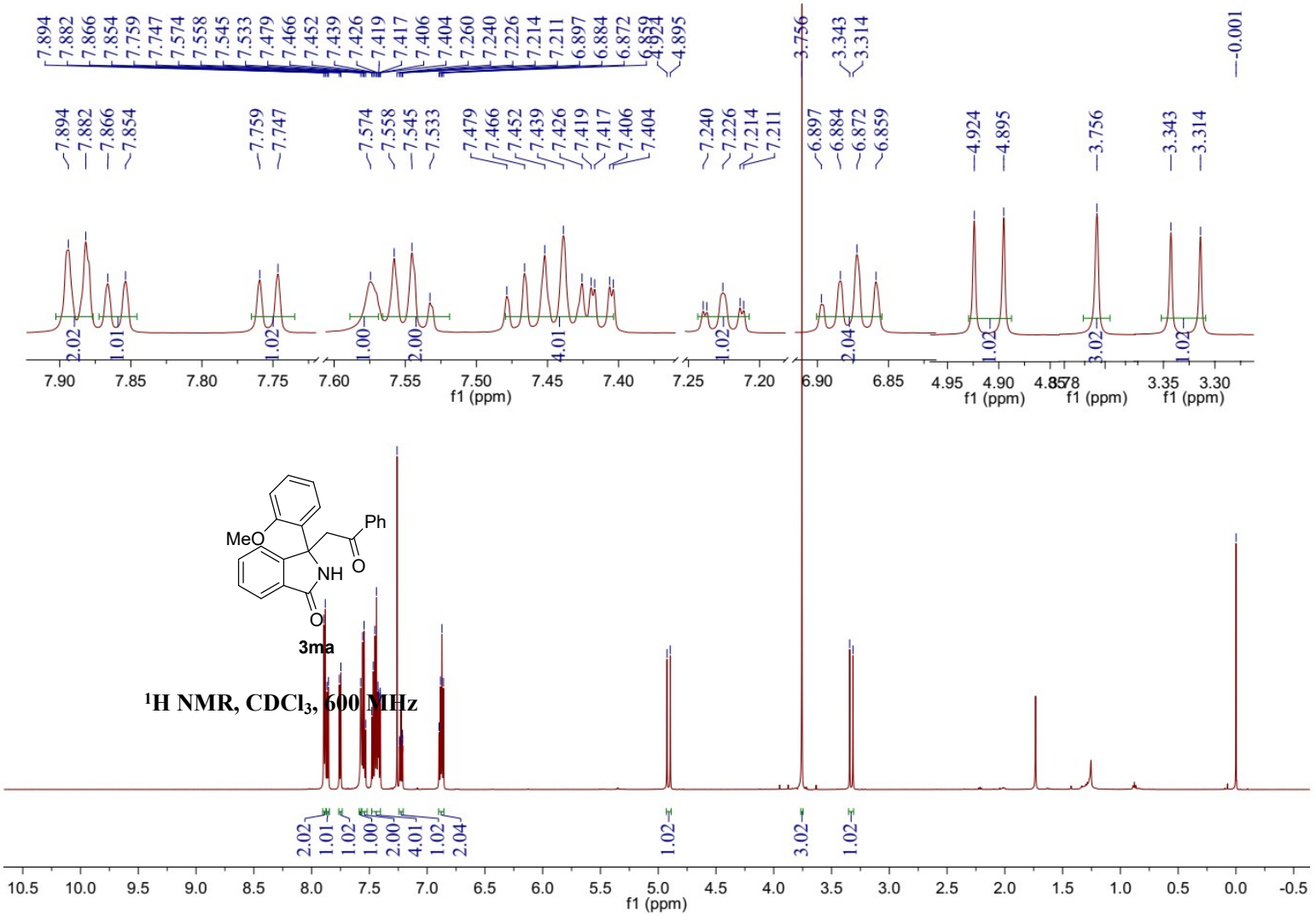
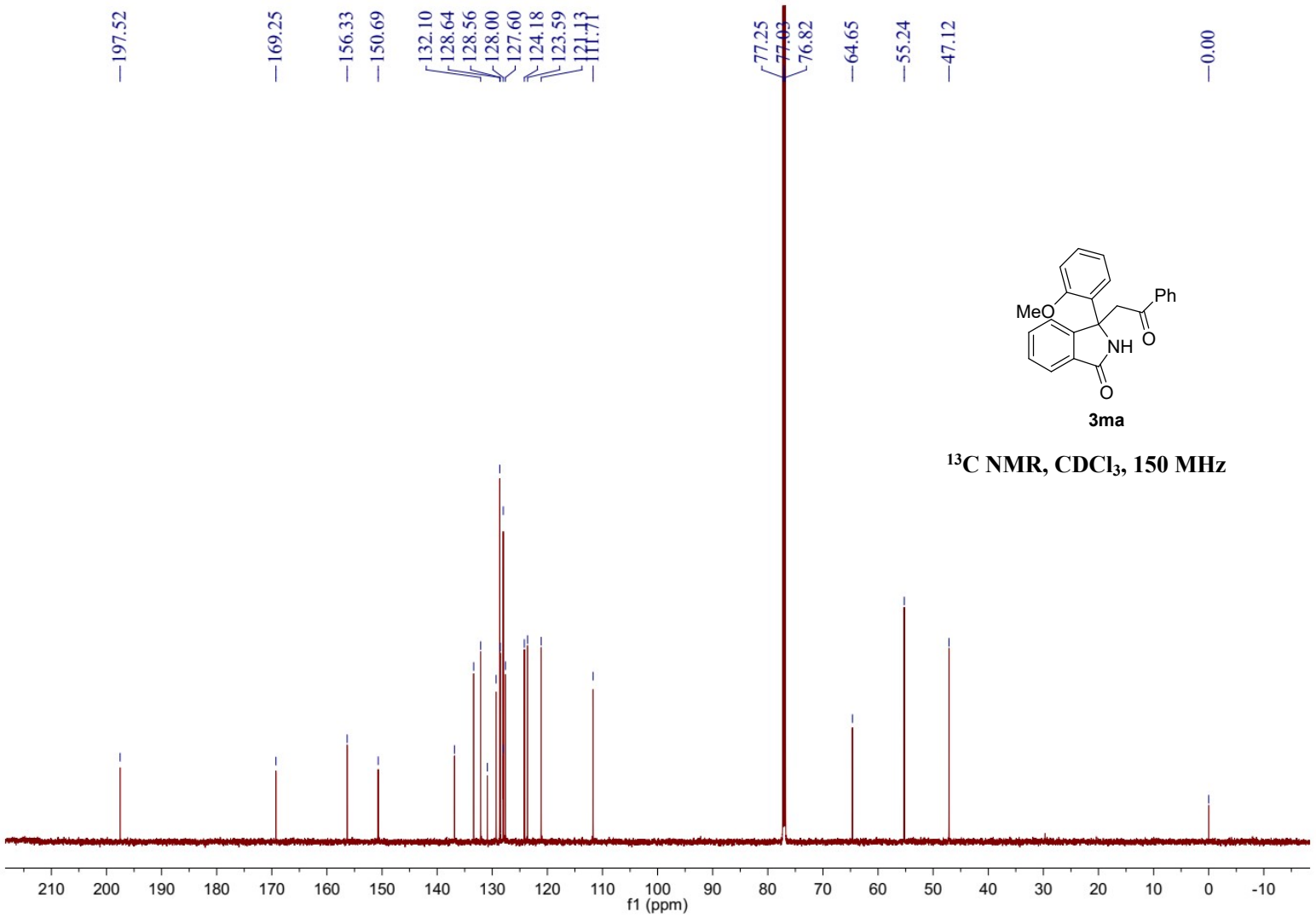
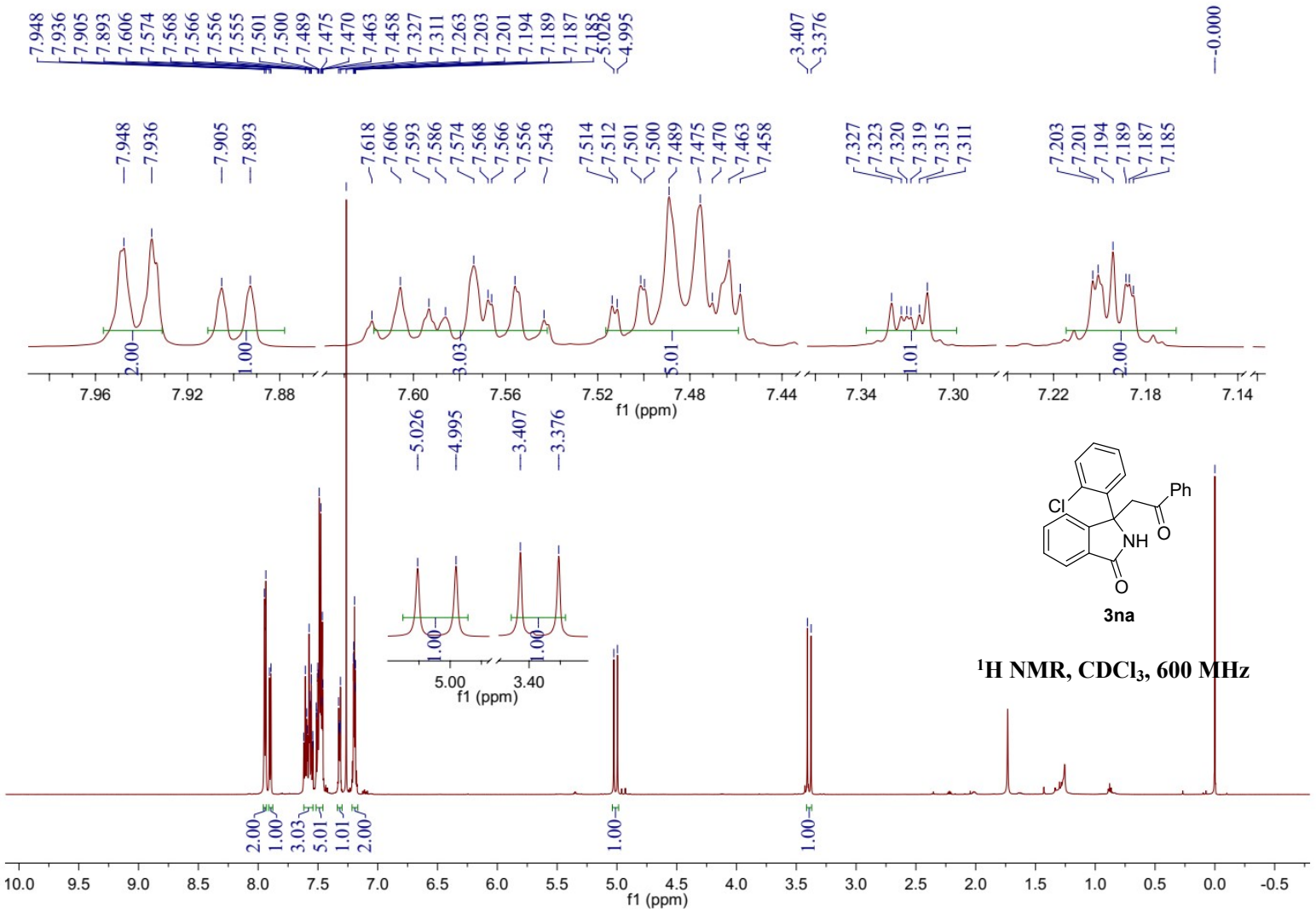
7.516
7.503
7.491
7.479
7.260
7.189
7.176
7.163
7.150
7.145
7.133
7.111
7.098
7.085
7.058
7.046
4.748
4.717
3.037
3.006
1.903
-0.002
2.02
1.00
1.01
0.94
4.01
3.01
1.00
1.00
1.01
3.00
f1 (ppm)
Me
NH
O
Ph
O
O
3la
^{1}H NMR, CDCl$_3$, 600 MHz


MeO
Ph
O
NH
O
3ma
1H NMR, CDCl3, 600 MHz
f1 (ppm)



3ma

^{13}C NMR, $CDCl_3$, 150 MHz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7.893
7.606
7.574
7.568
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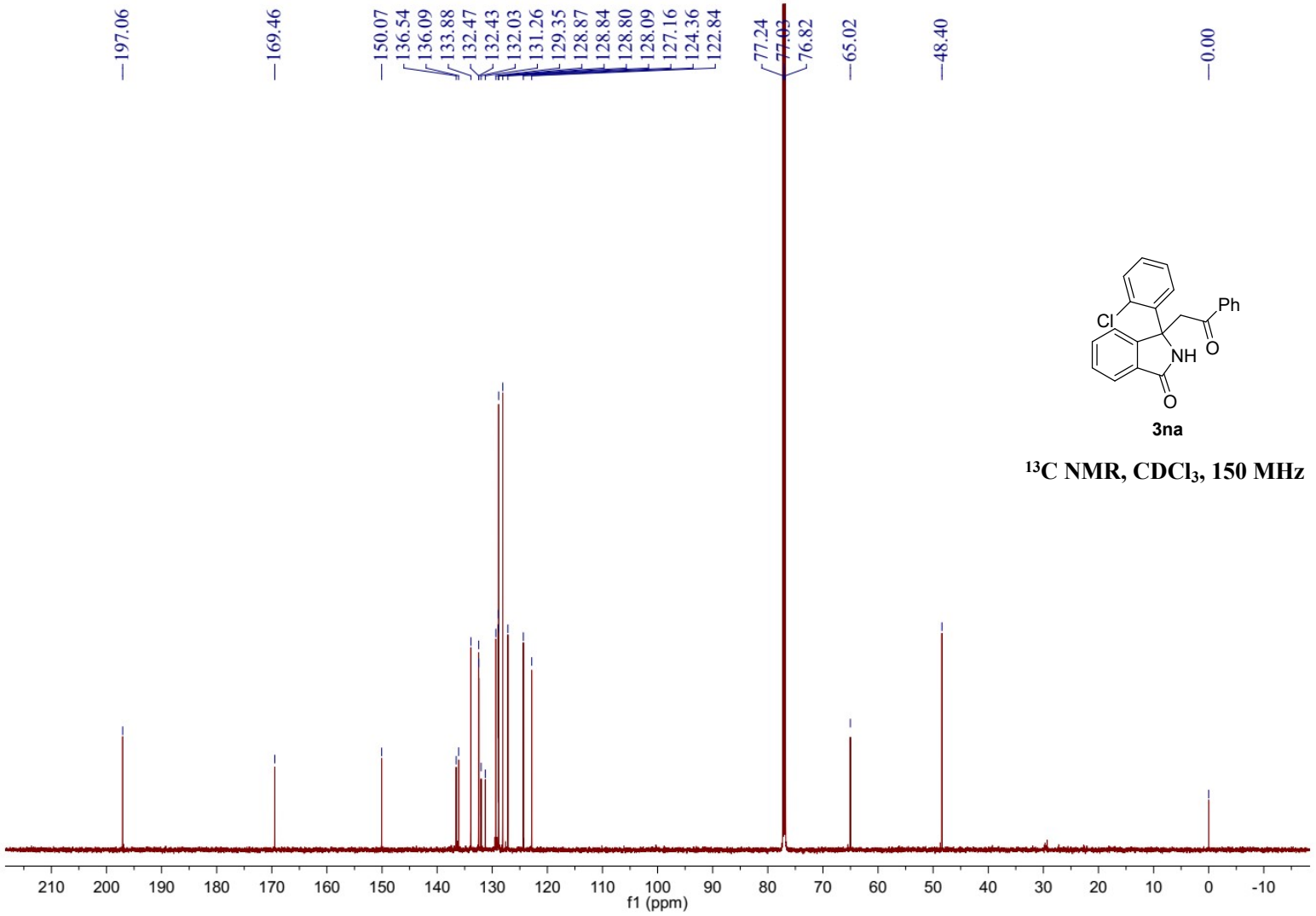
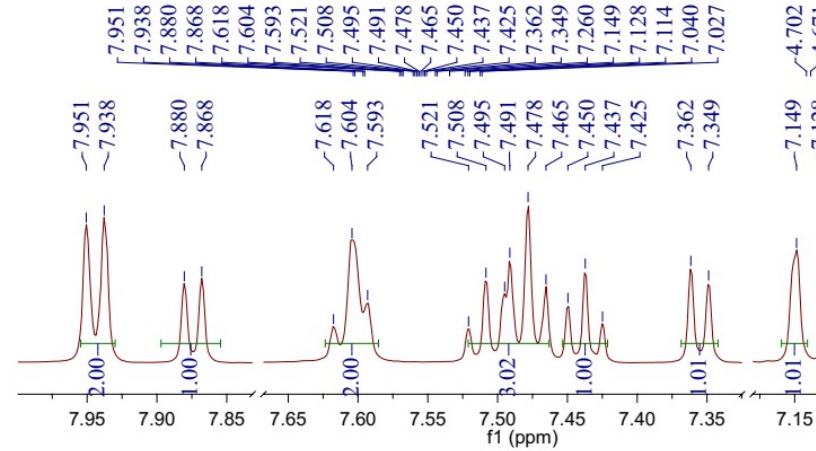
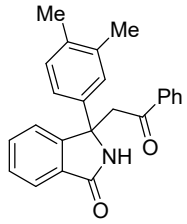
7.556
7.555
7.501
7.500
7.489
7.475
7.470
7.463
7.458
7.327
7.311
7.263
7.203
7.201
7.194
7.189
7.187
7.185
5.026
4.995
3.407
3.376
-0.000
2.00
1.00
3.03
5.01
1.01
2.00
f1 (ppm)
Cl
NH
O
Ph
3na
1H NMR, CDCl3, 600 MHz



3na

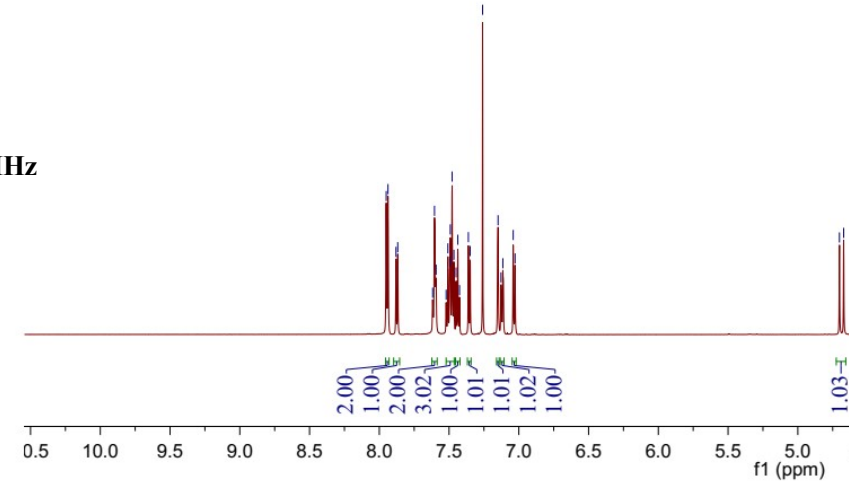
^{13}C NMR, $CDCl_3$, 150 MHz

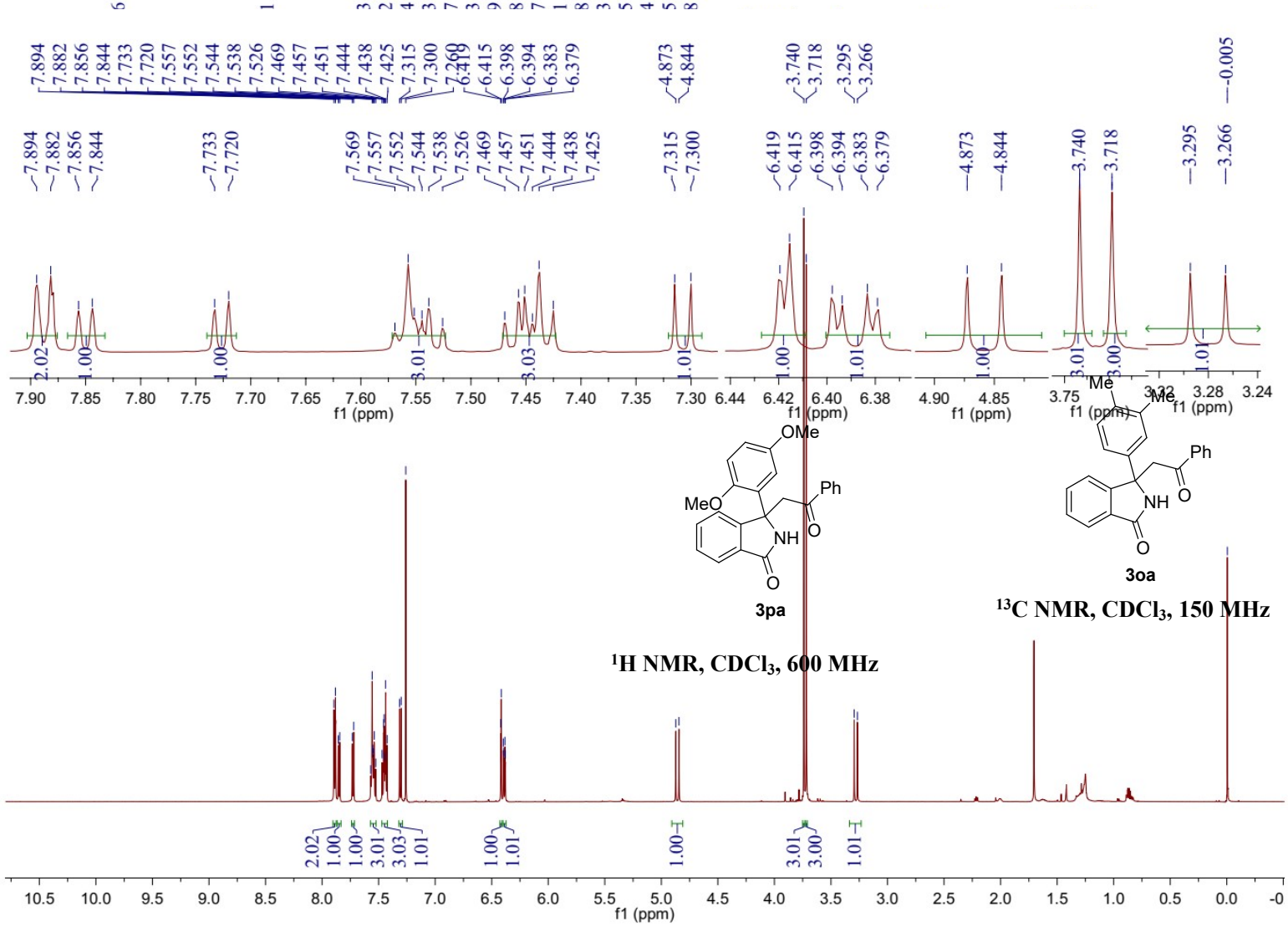


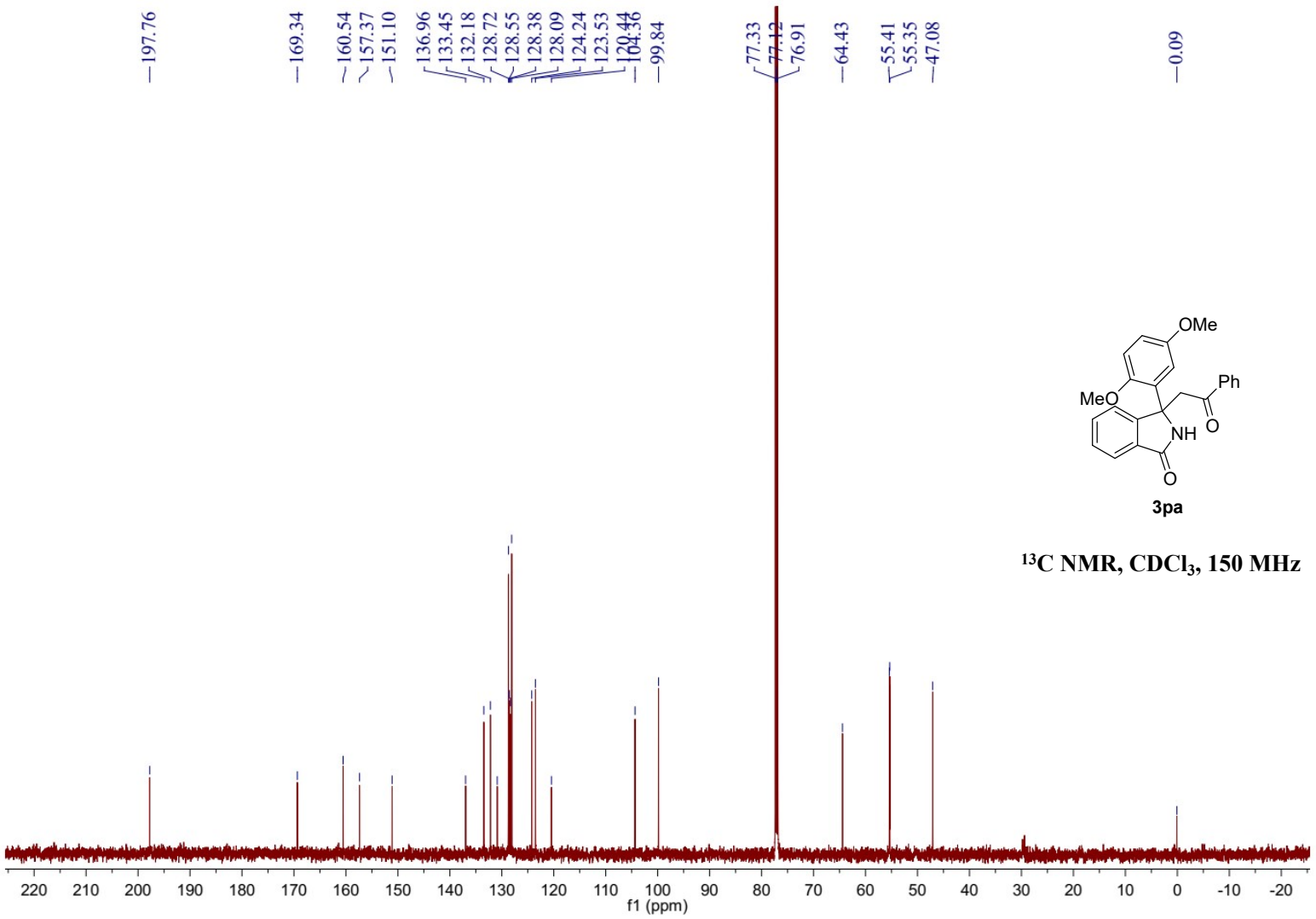


3oa

^{1}H NMR, $CDCl_3$, 600 MHz




OMe
MeO
Ph
O
NH
O
O
3pa
Me
Me
Ph
O
NH
O
O
3oa
^{1}H NMR, CDCl$_3$, 600 MHz
^{13}C NMR, CDCl$_3$, 150 MHz
f1 (ppm)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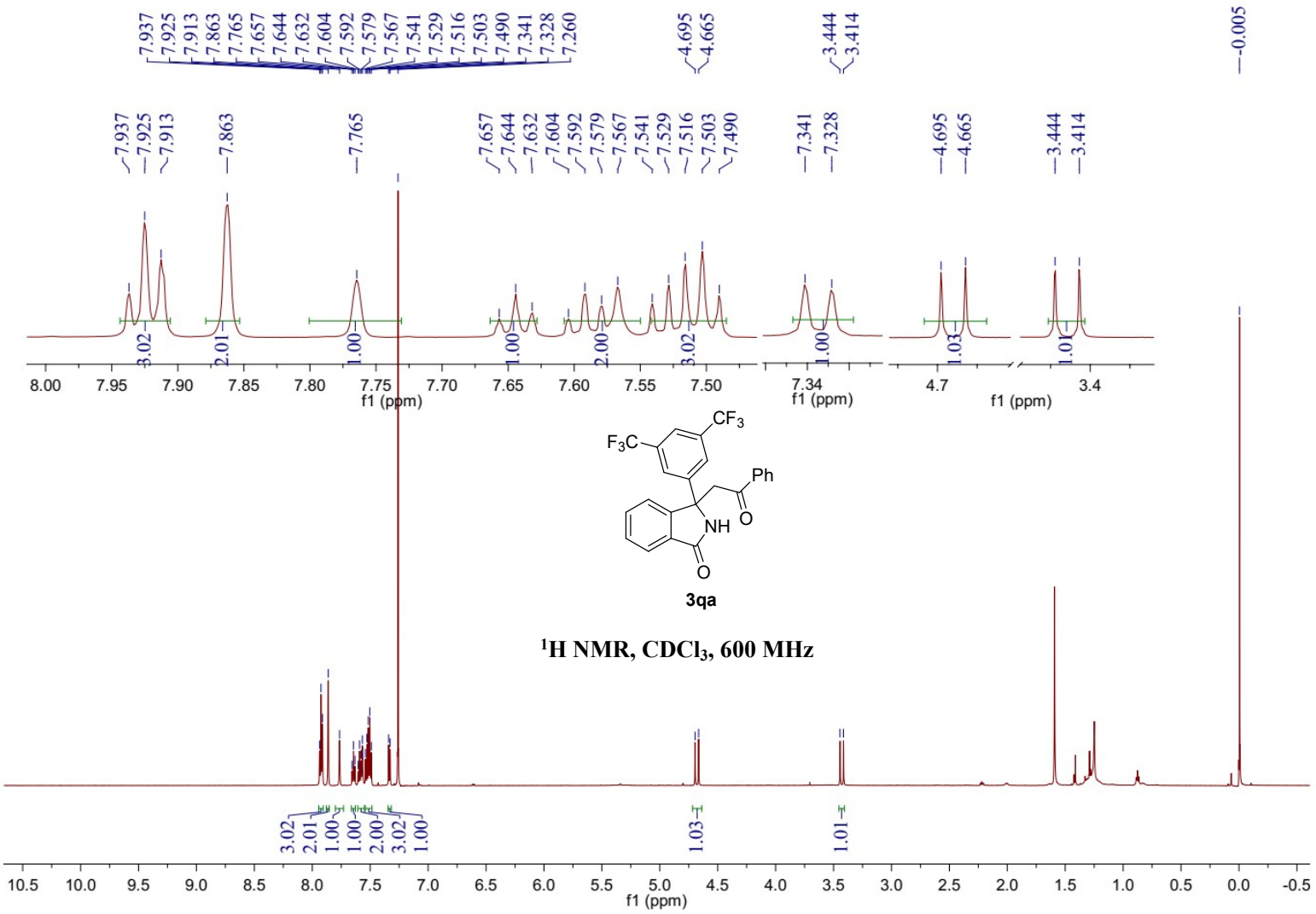
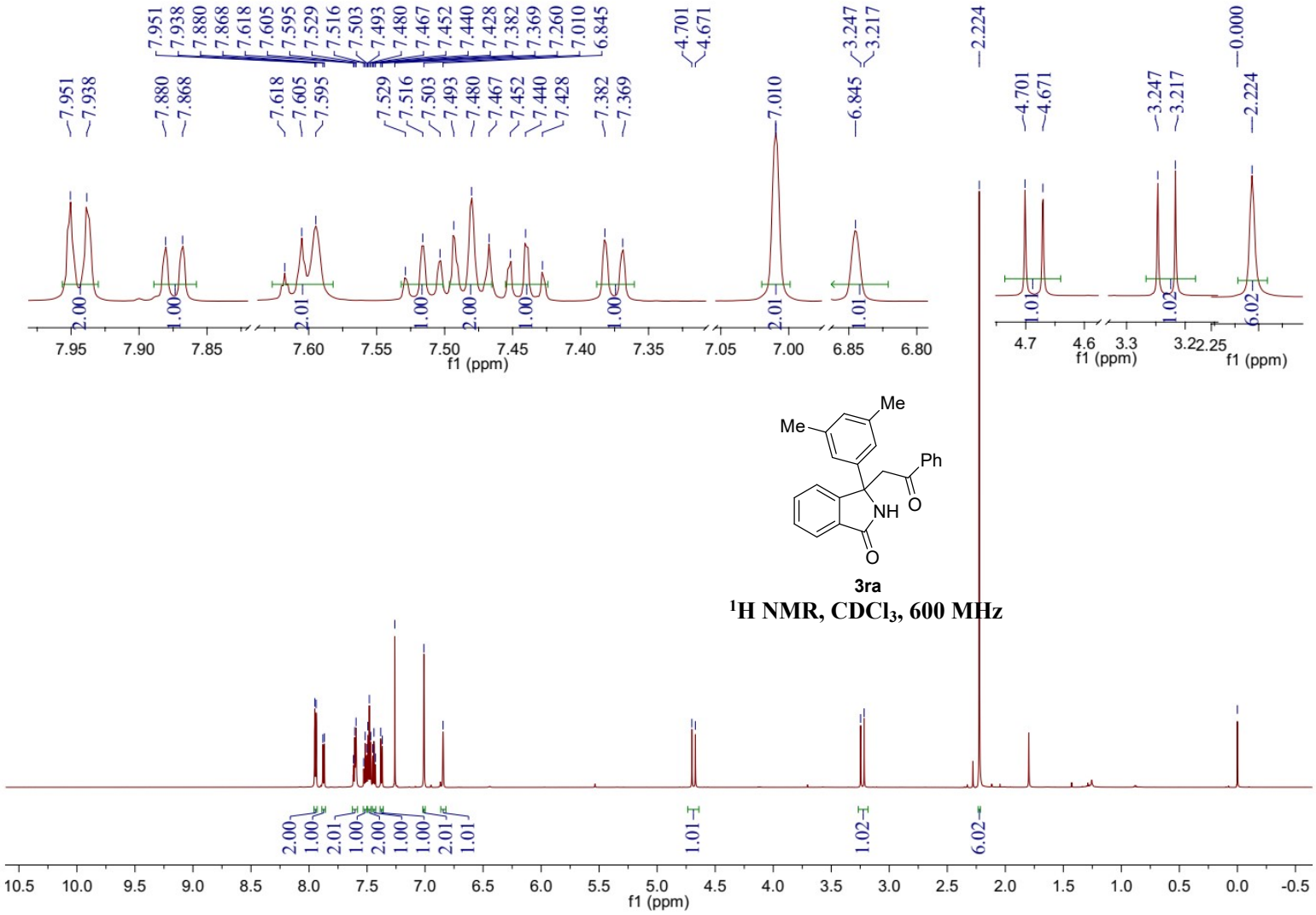
160.54
157.37
151.10
136.96
133.45
132.18
128.72
128.55
128.38
128.09
124.24
123.53
120.44
104.36
99.84
77.33
77.12
76.91
64.43
55.41
55.35
47.08
0.09
OMe
MeO
Ph
O
NH
O
3pa
13C NMR, CDCl3, 150 MHz
f1 (ppm)



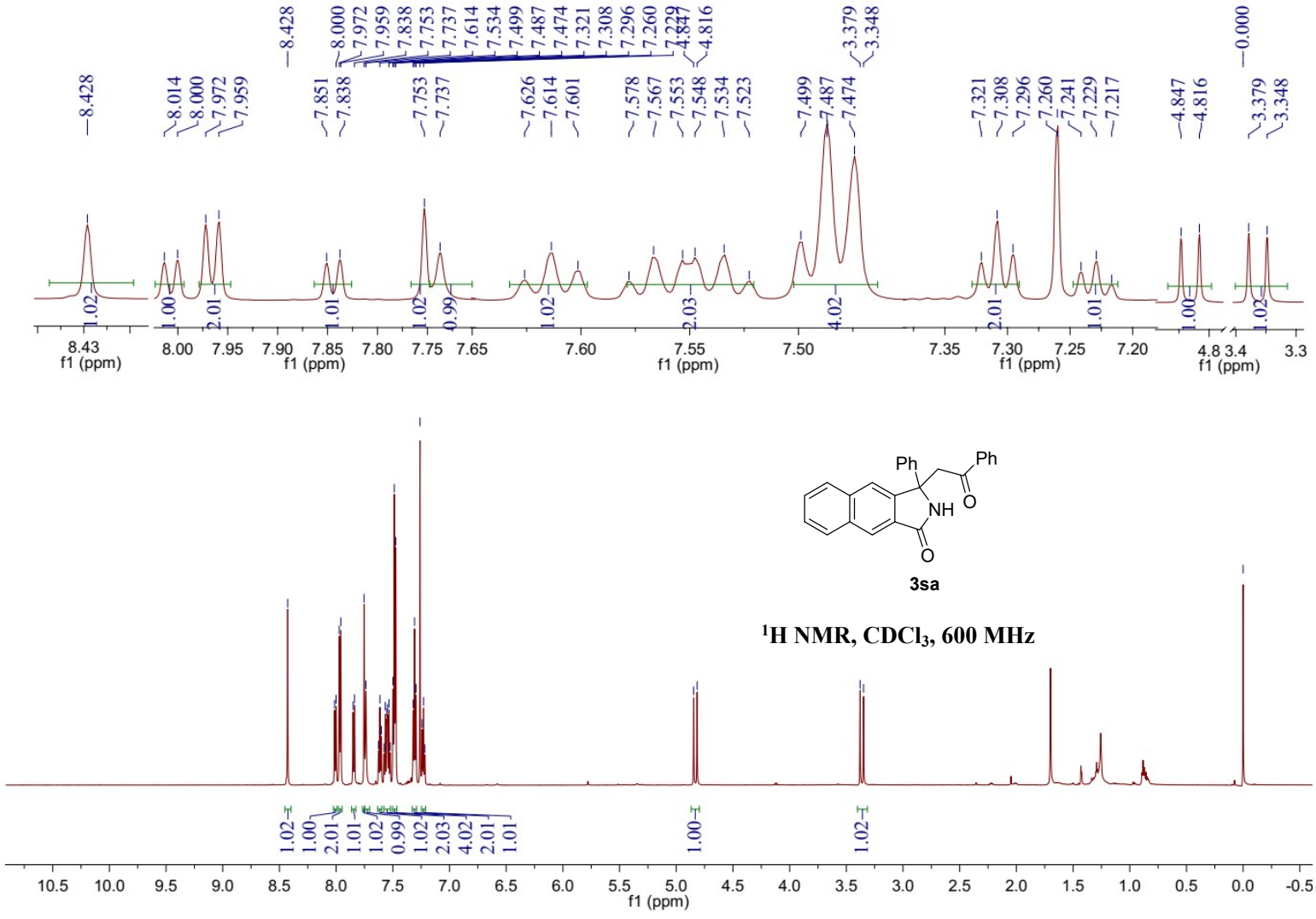
3qa

^{1}H NMR, $CDCl_3$, 600 MHz



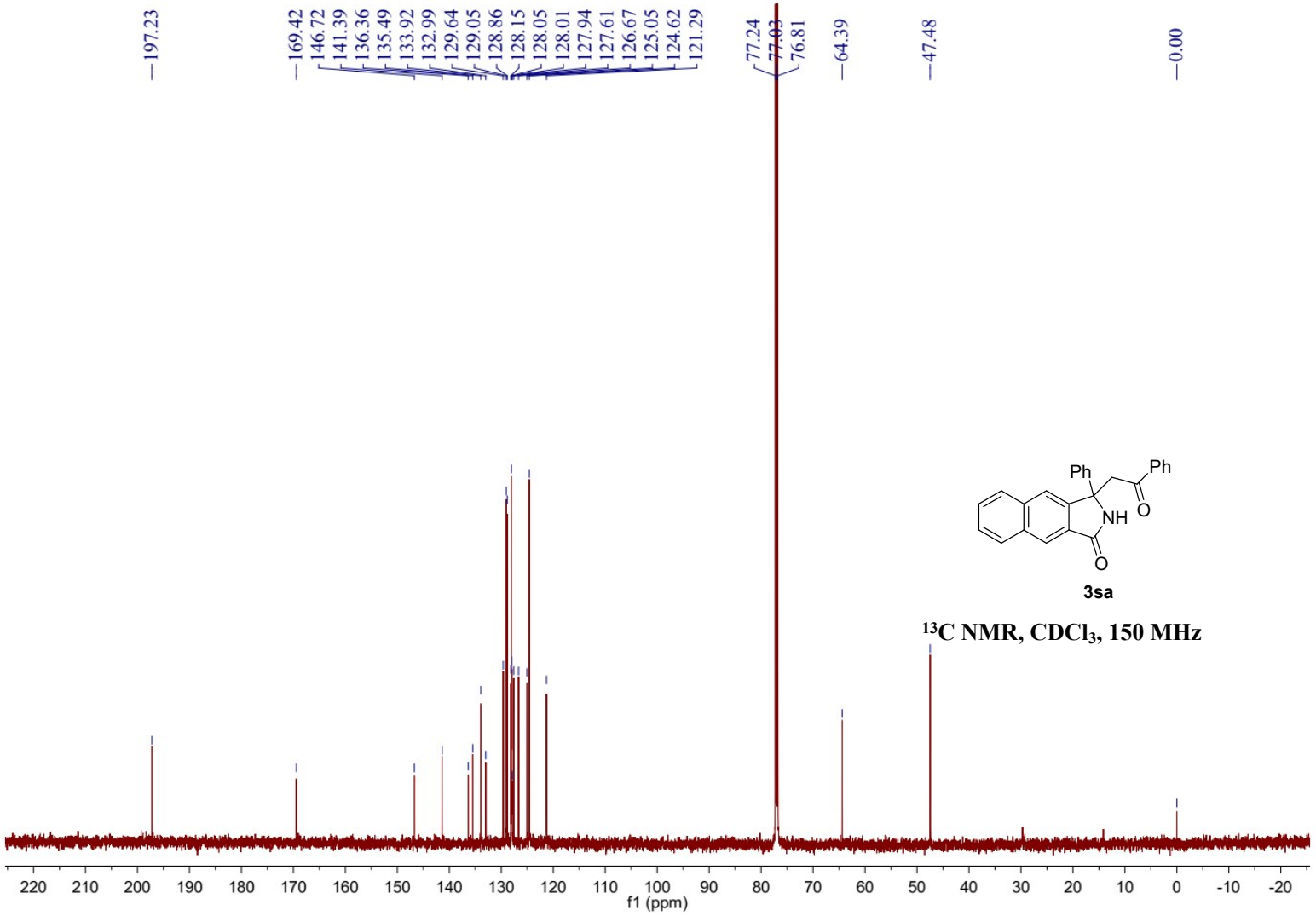
3ra

^{1}H NMR, $CDCl_3$, 600 MHz



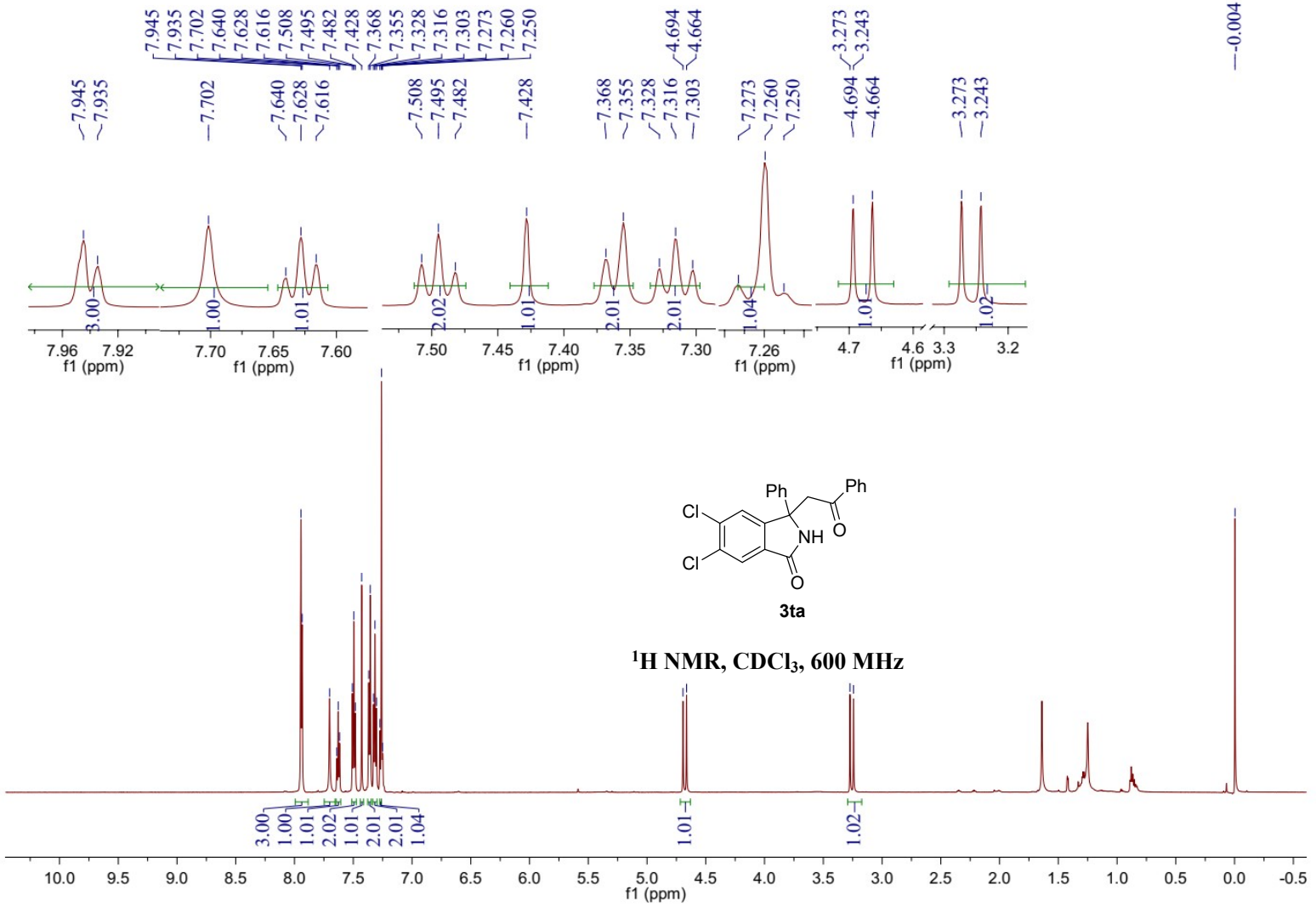
3sa

^{1}H NMR, $CDCl_3$, 600 MHz

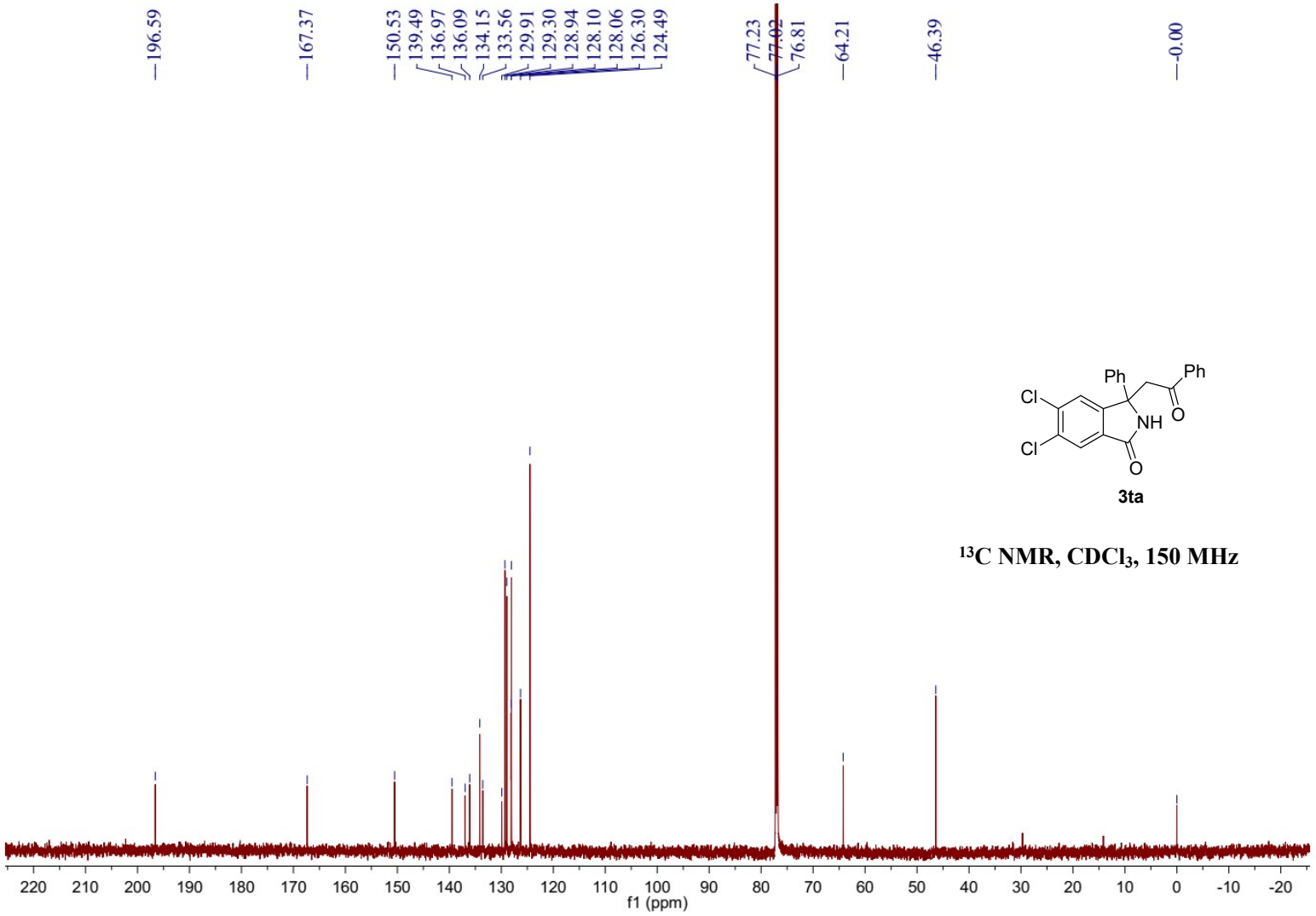


3sa

^{13}C NMR, $CDCl_3$, 150 MHz

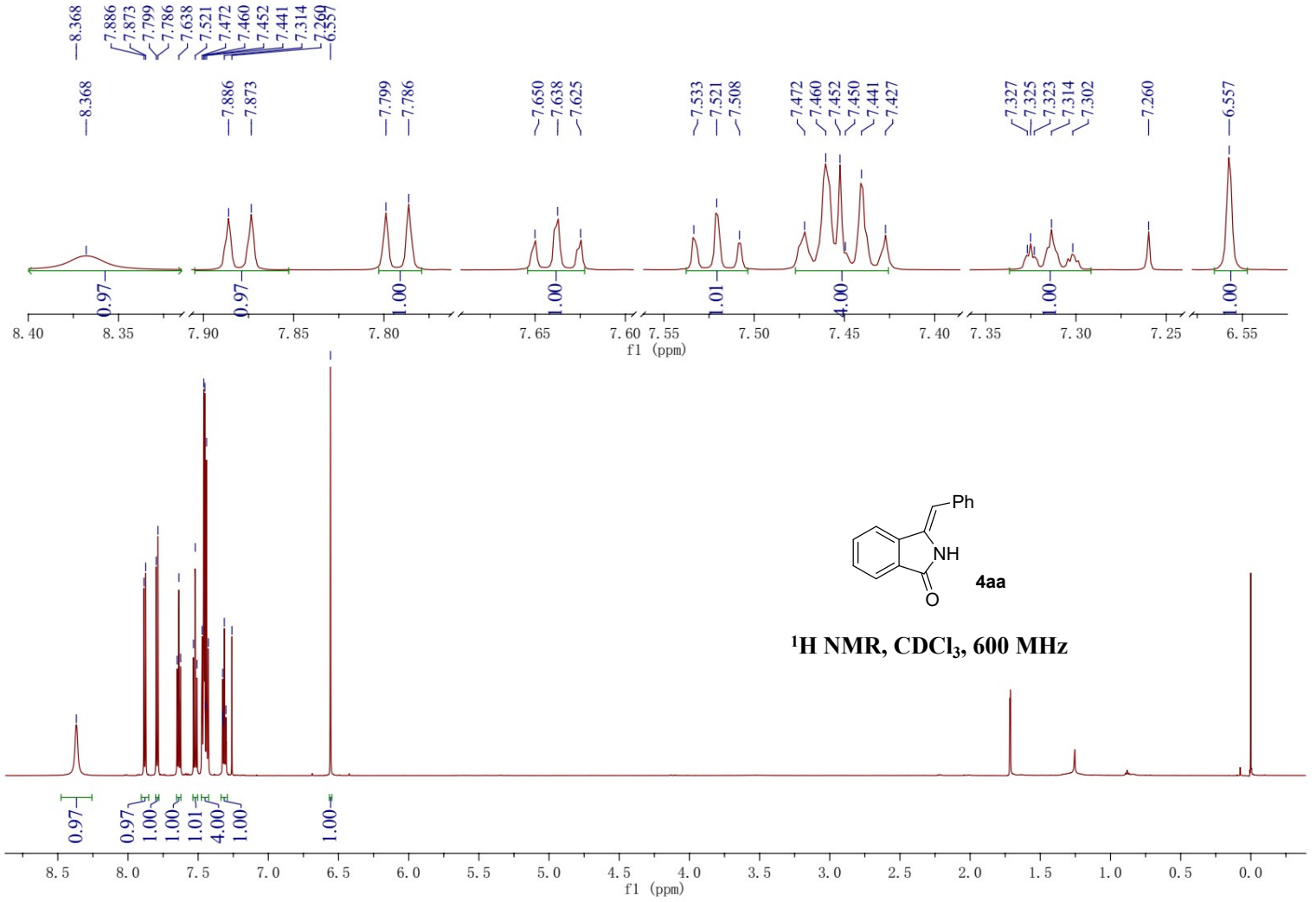


^{1}H NMR, $CDCl_3$, 600 MHz

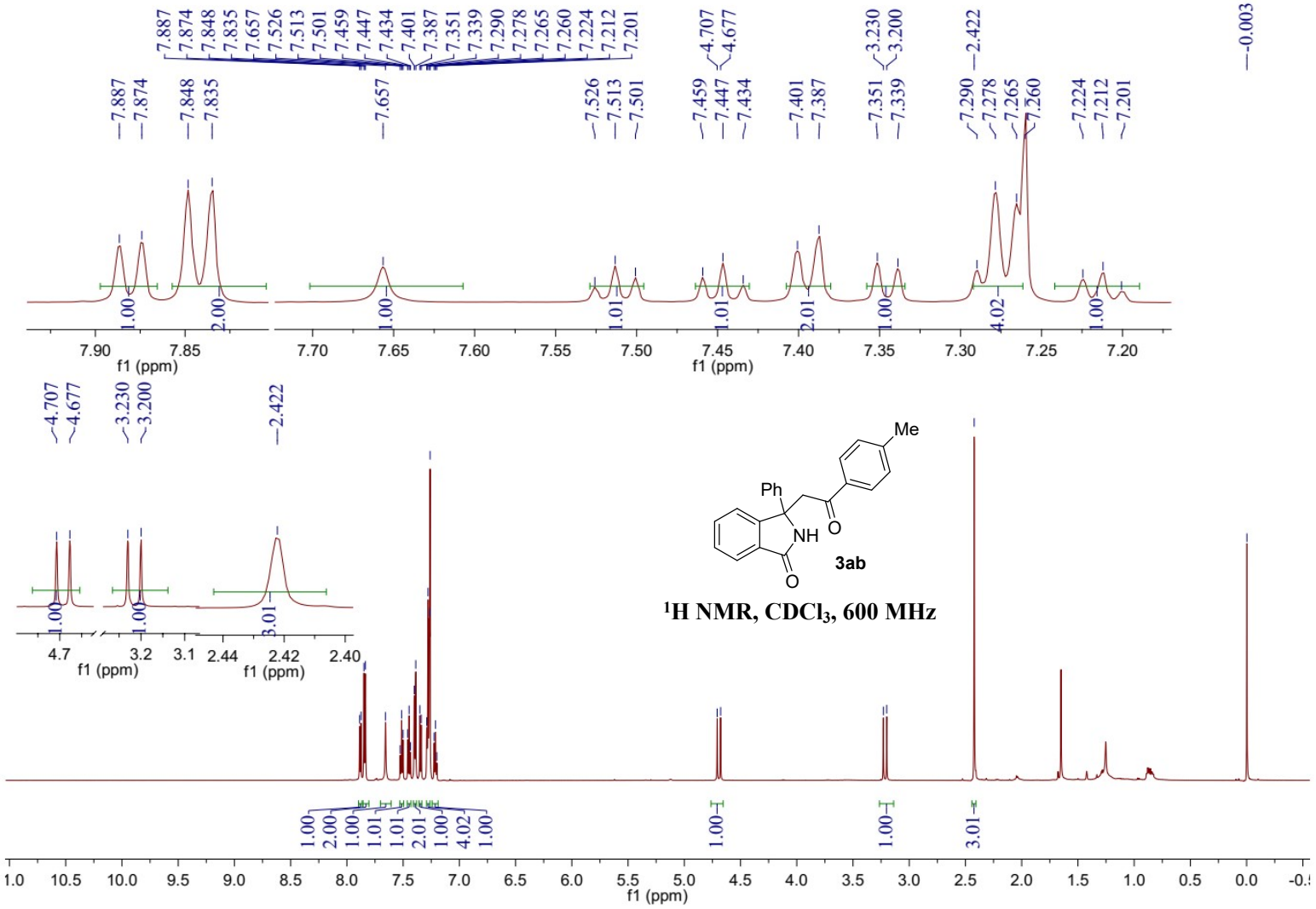


3ta

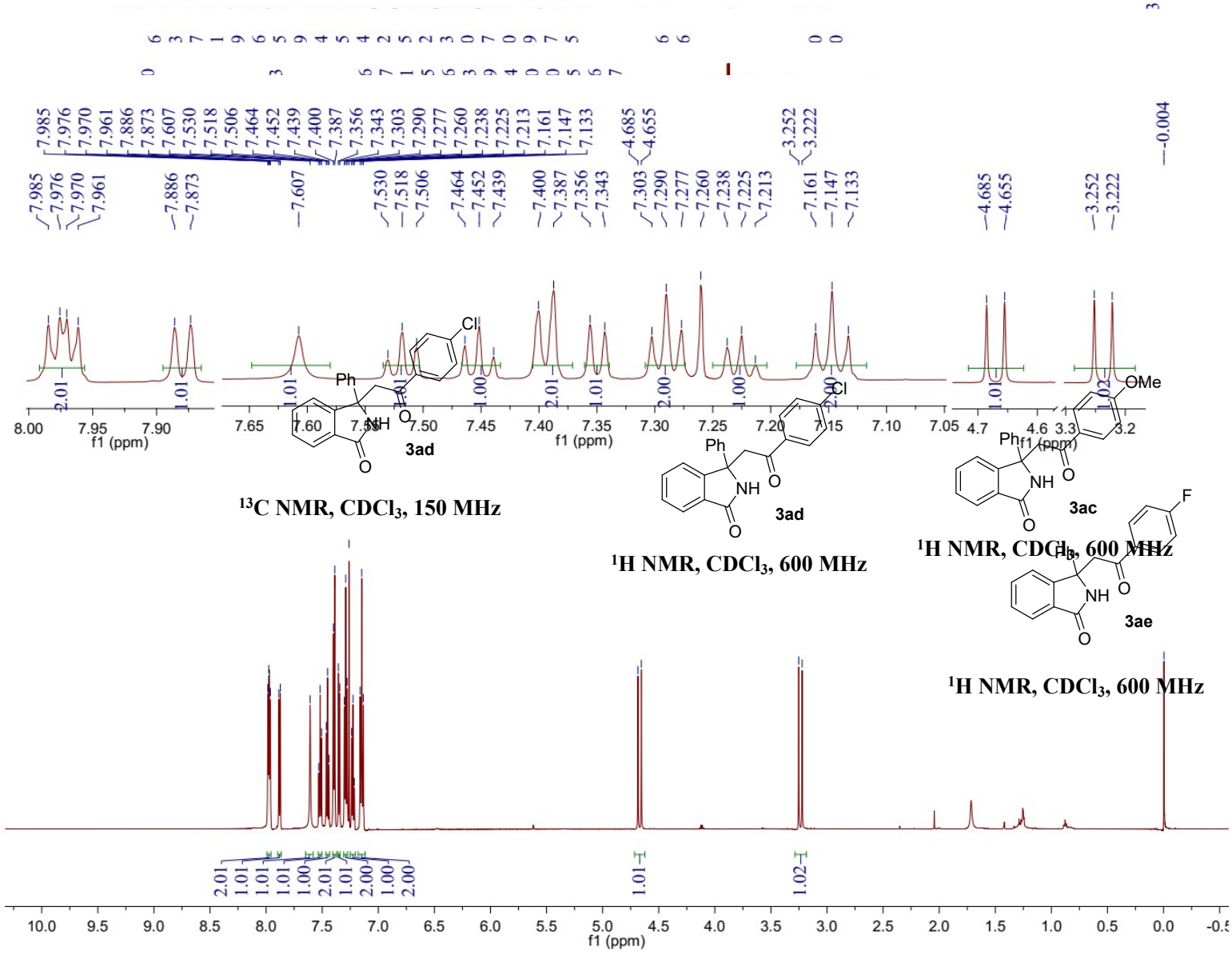
^{13}C NMR, $CDCl_3$, 150 MHz



^{1}H NMR, $CDCl_3$, 600 MHz



^{1}H NMR, $CDCl_3$, 600 MHz

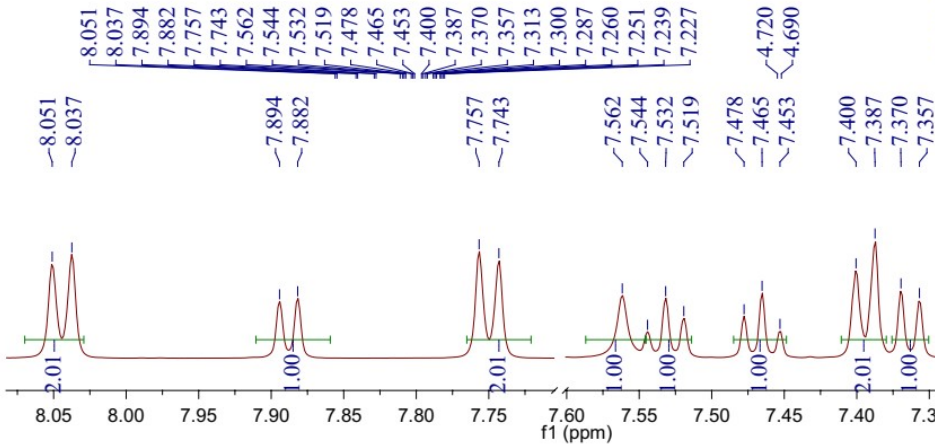


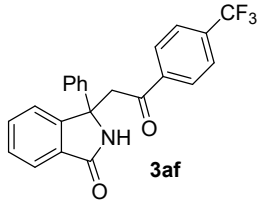
^{13}C NMR, $CDCl_3$, 150 MHz

^{1}H NMR, $CDCl_3$, 600 MHz

^{1}H NMR, $CDCl_3$, 600 MHz

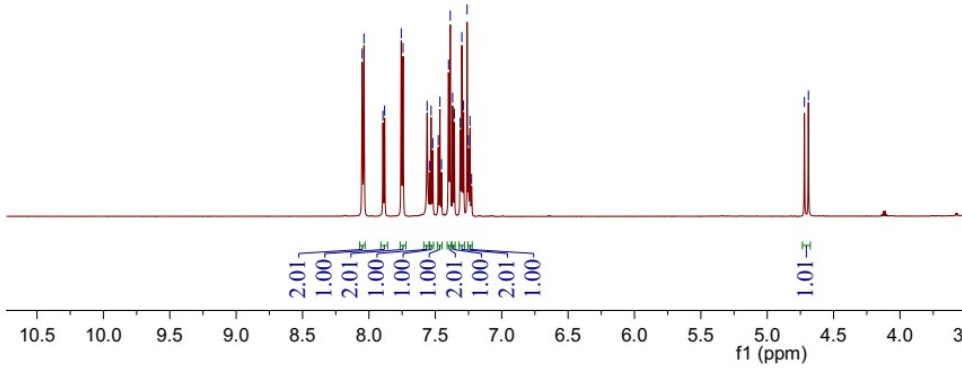
^{1}H NMR, $CDCl_3$, 600 MHz

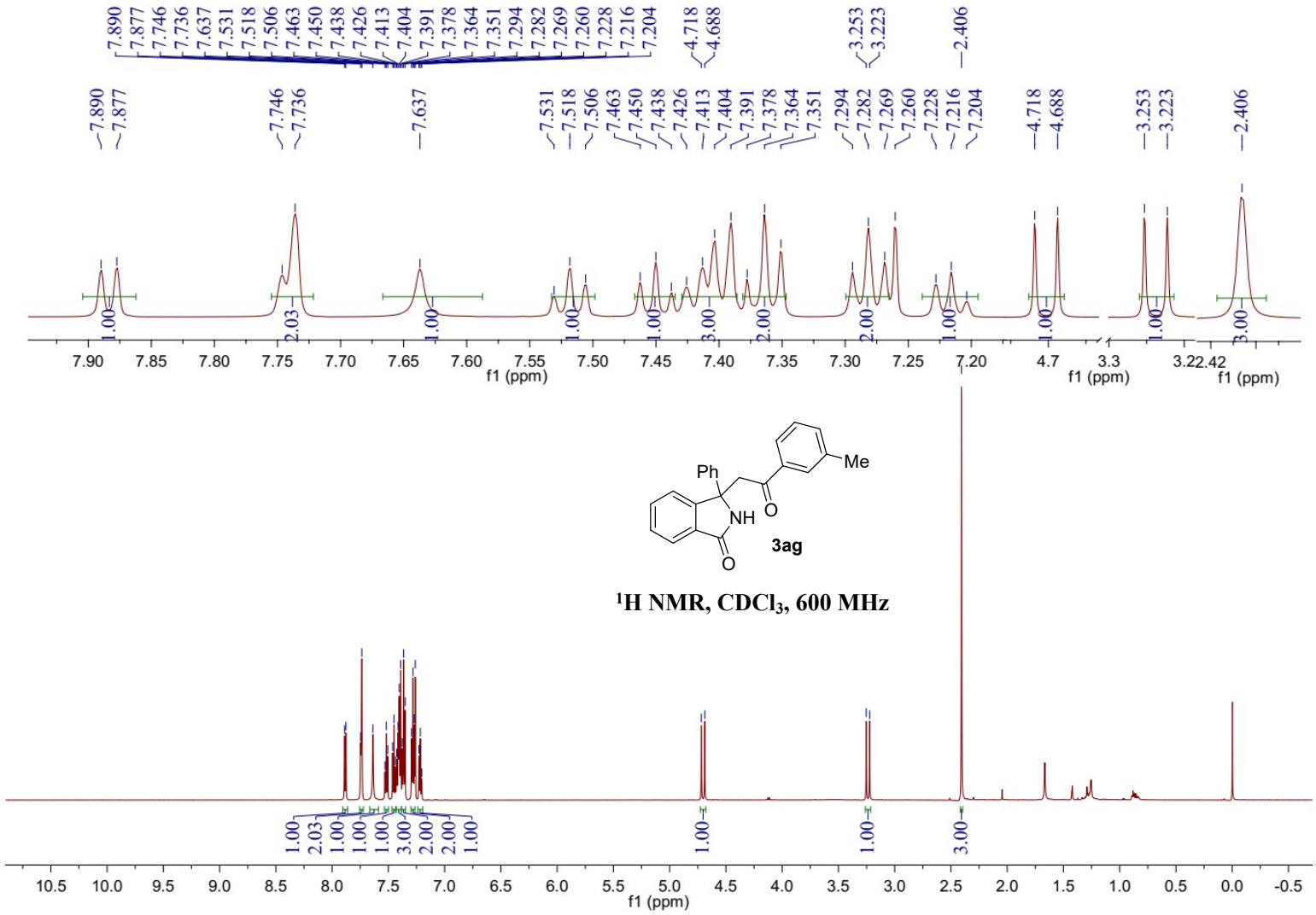




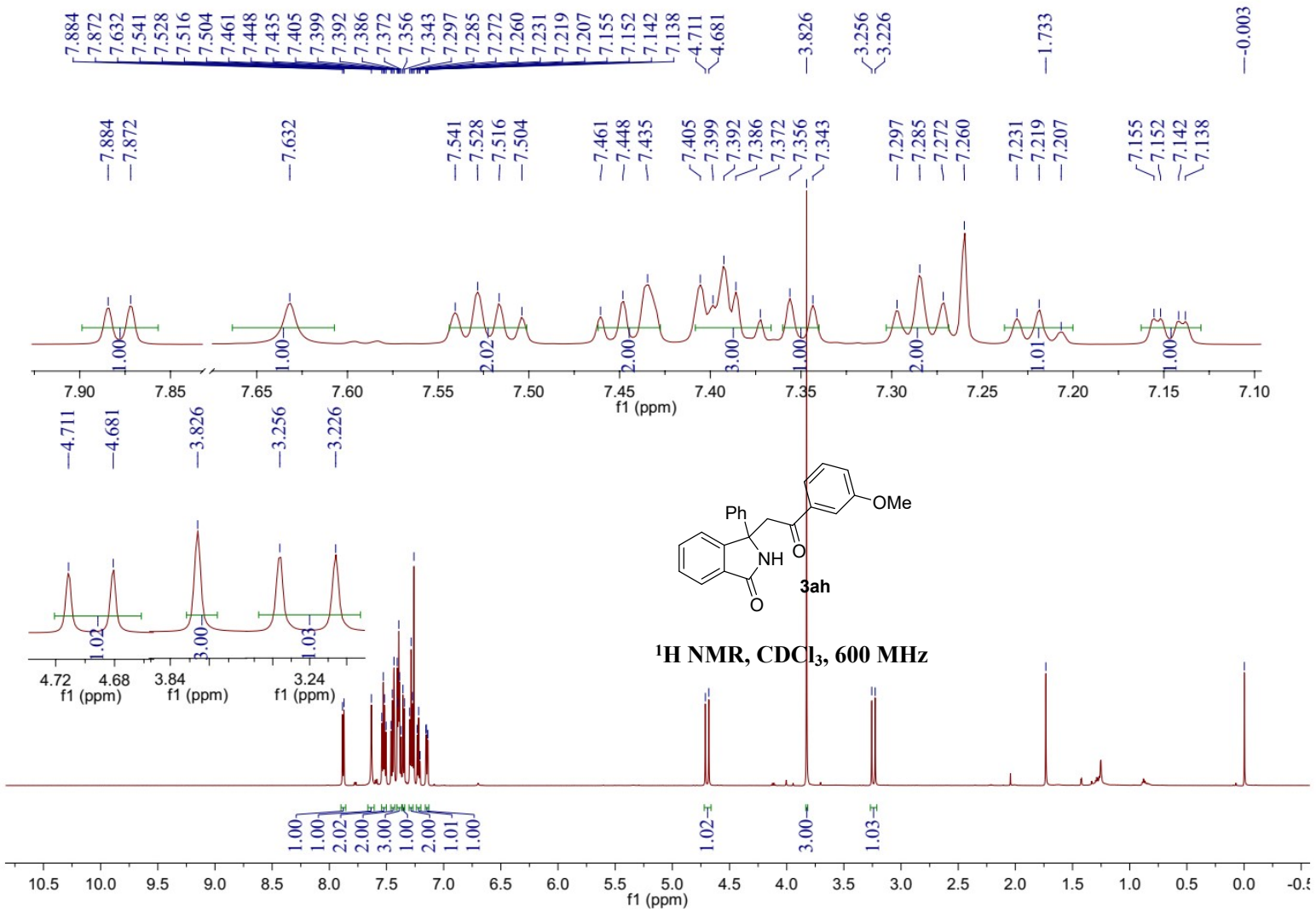
3af

^{1}H NMR, $CDCl_3$, 600 MHz

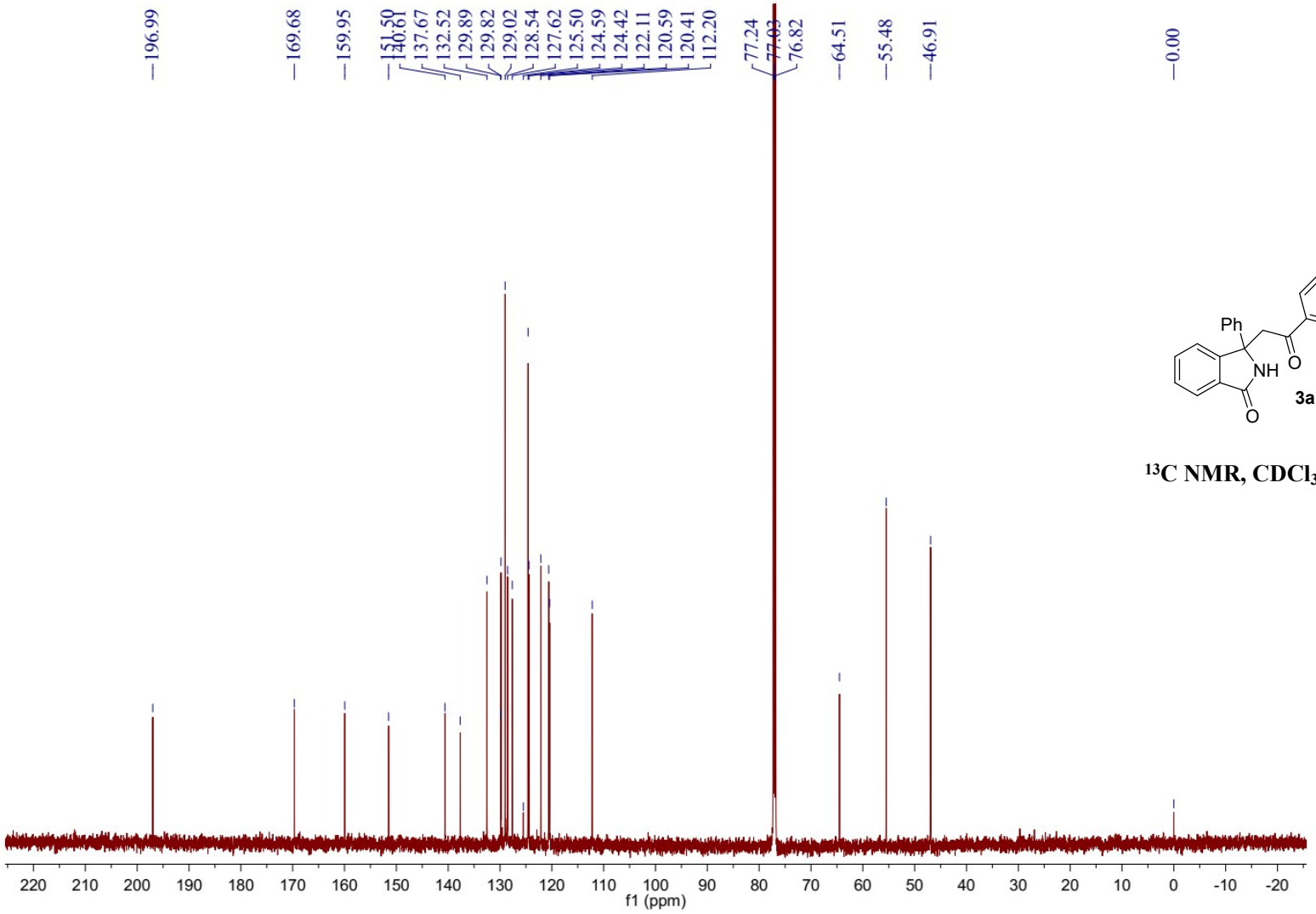


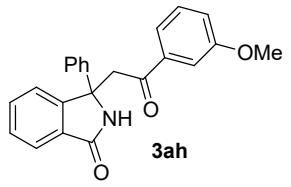


^{1}H NMR, $CDCl_3$, 600 MHz

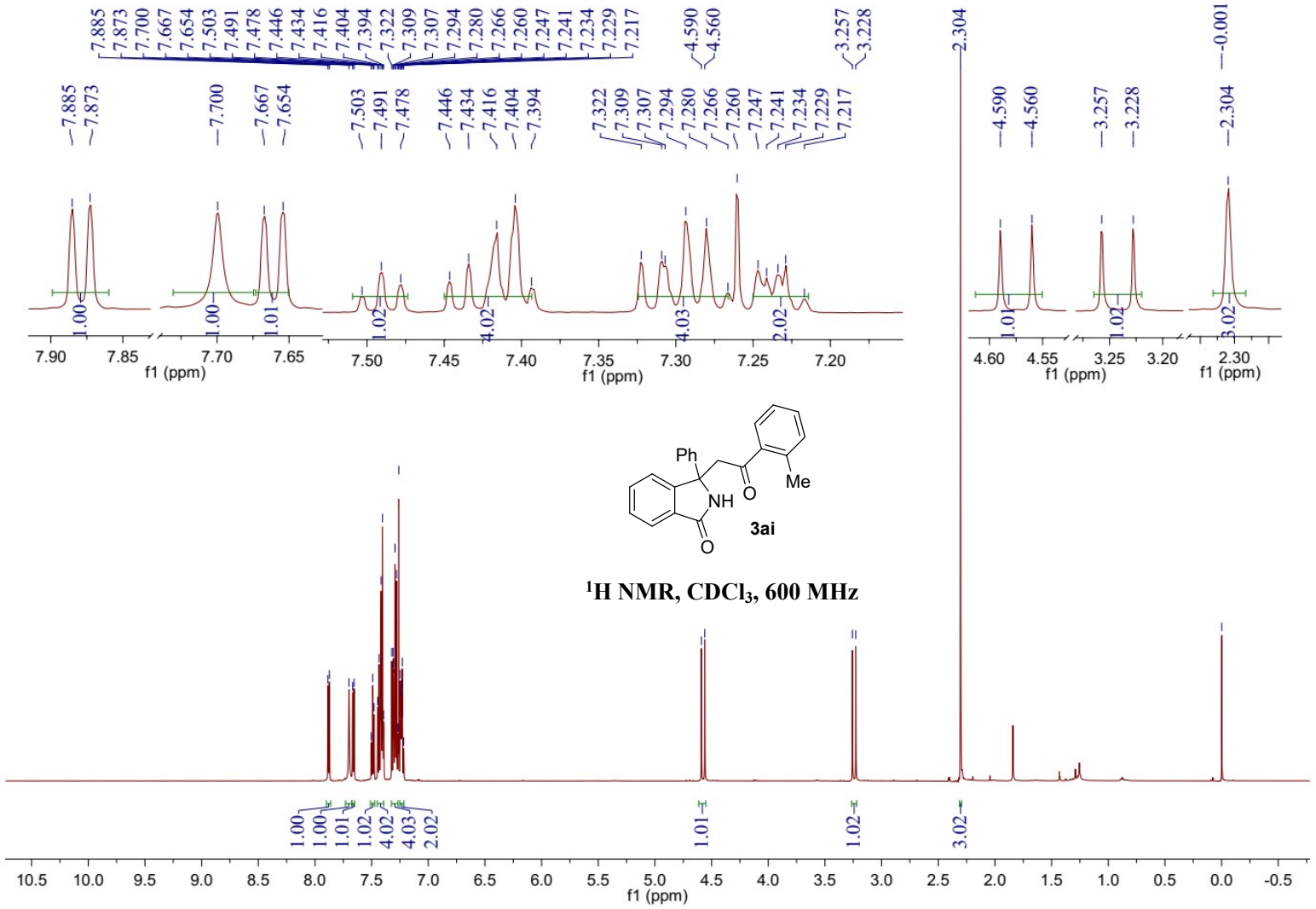


^{1}H NMR, $CDCl_3$, 600 MHz

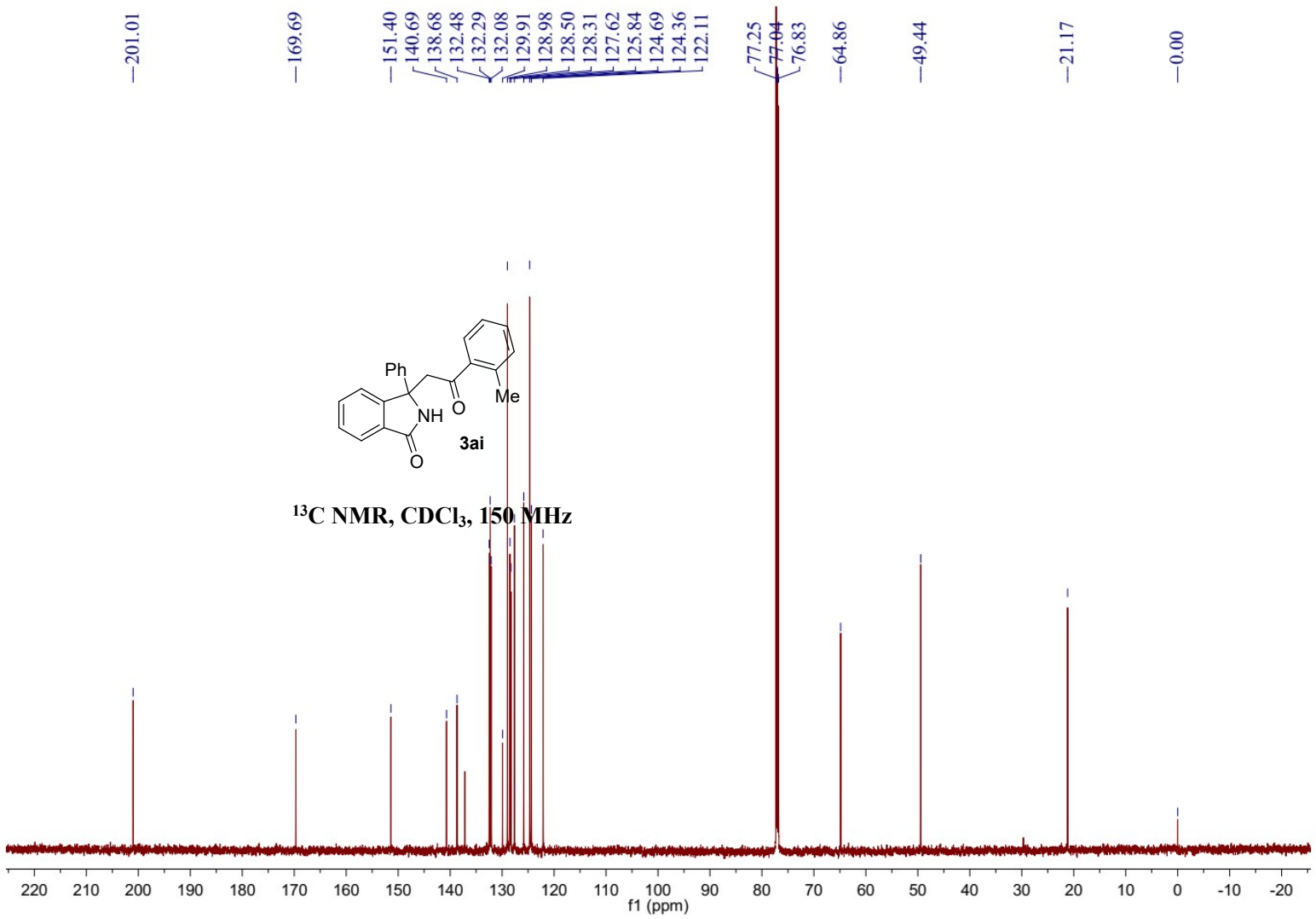




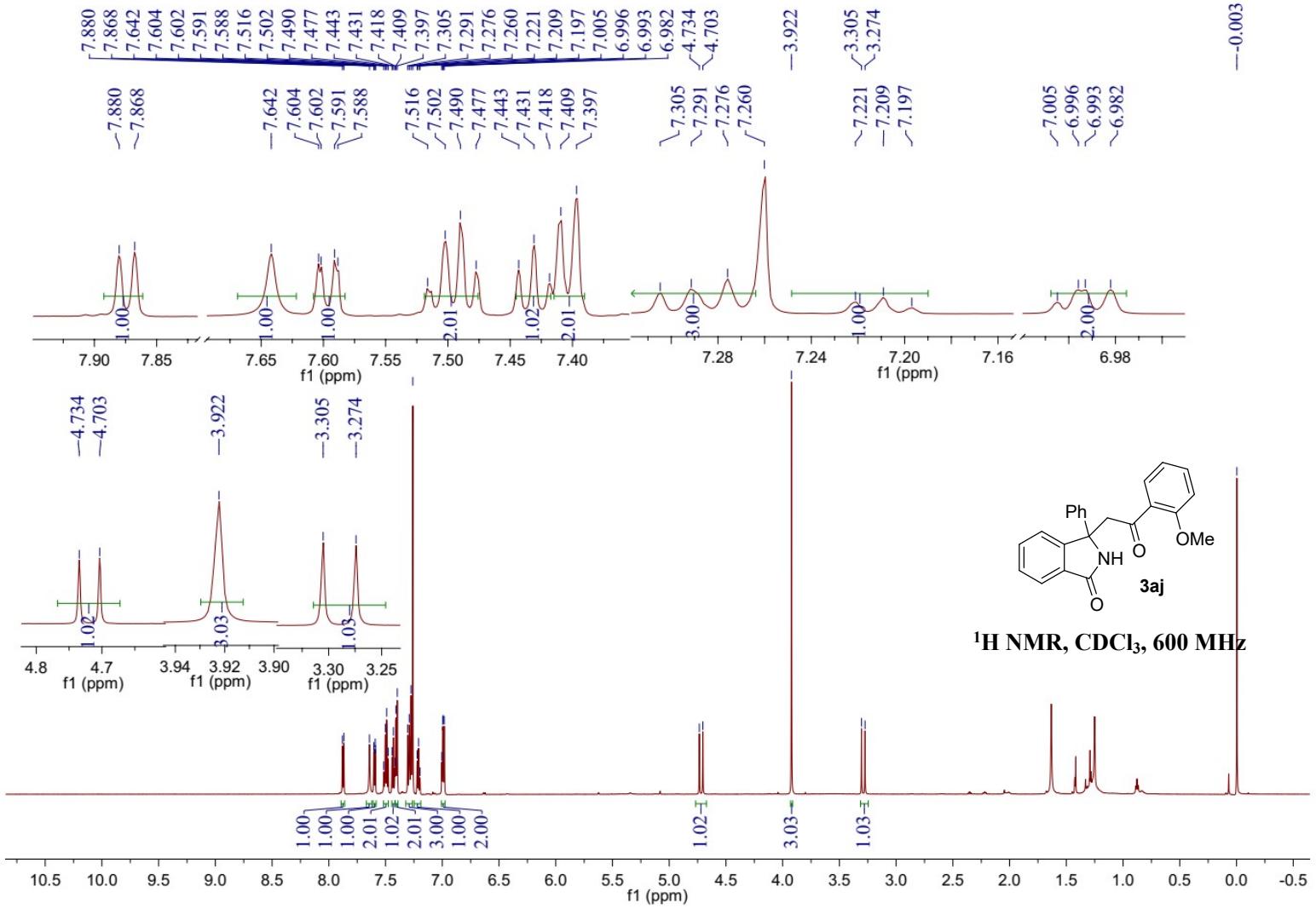
^{13}C NMR, $CDCl_3$, 150 MHz



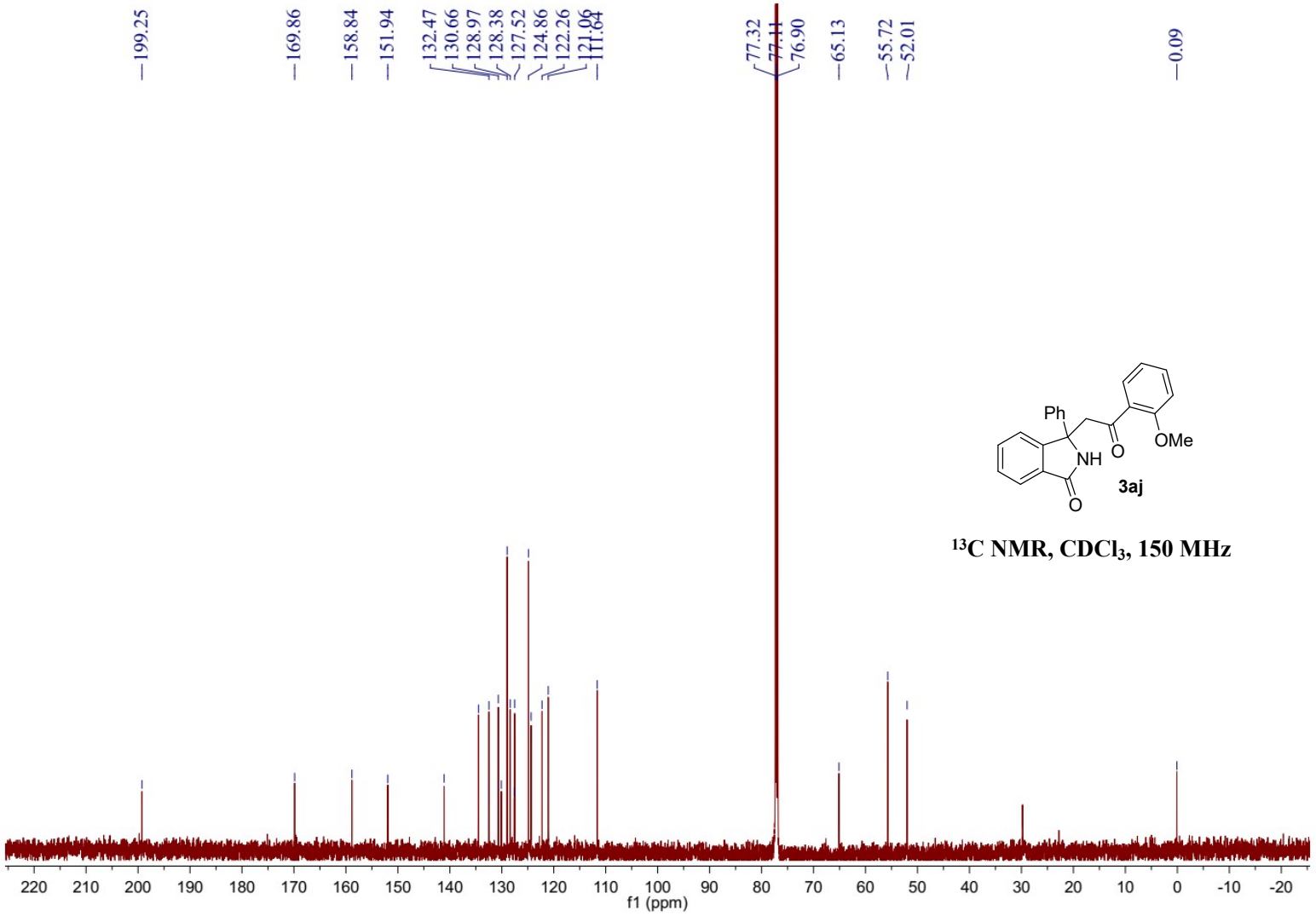
^{1}H NMR, $CDCl_3$, 600 MHz



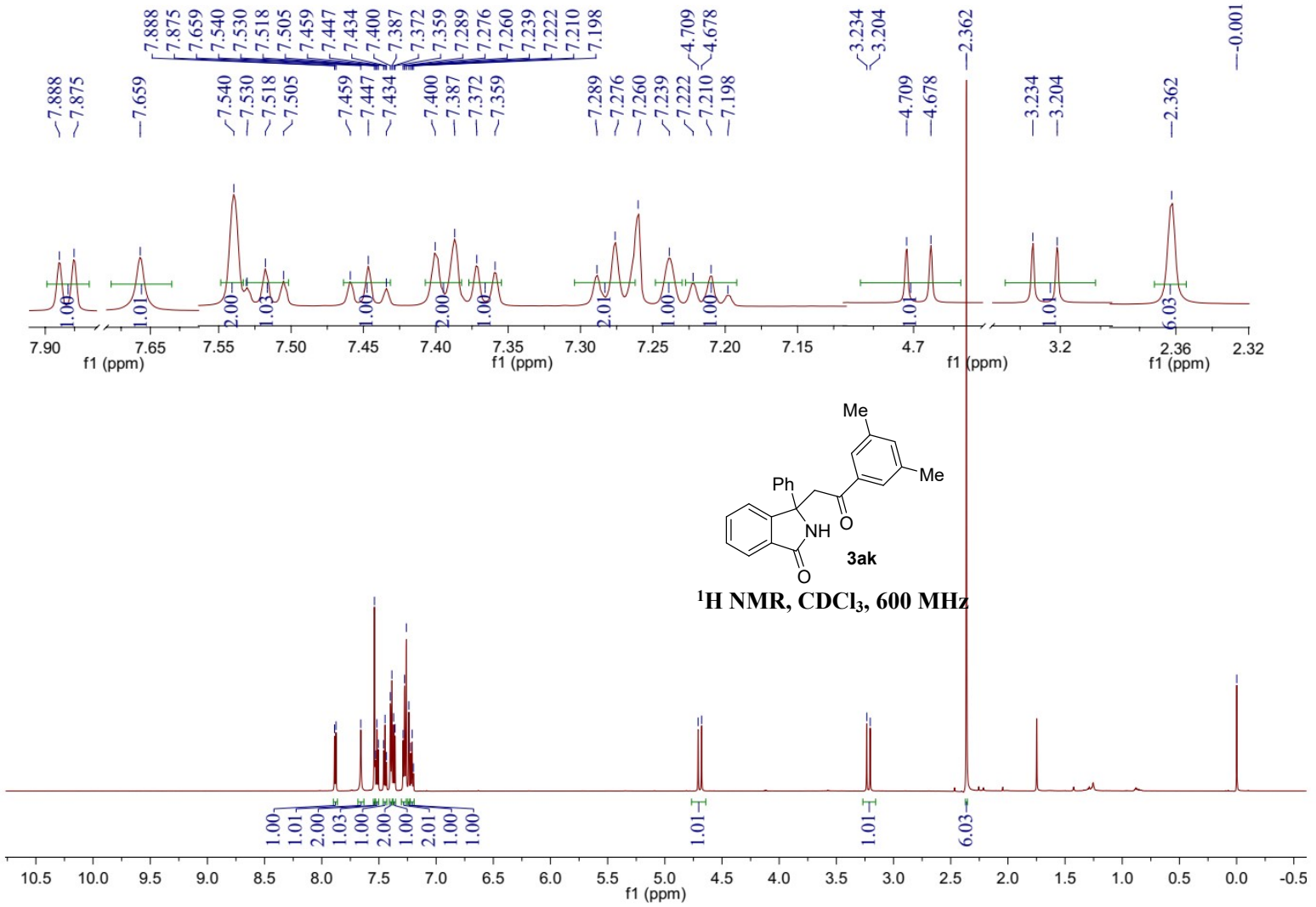
^{13}C NMR, $CDCl_3$, 150 MHz



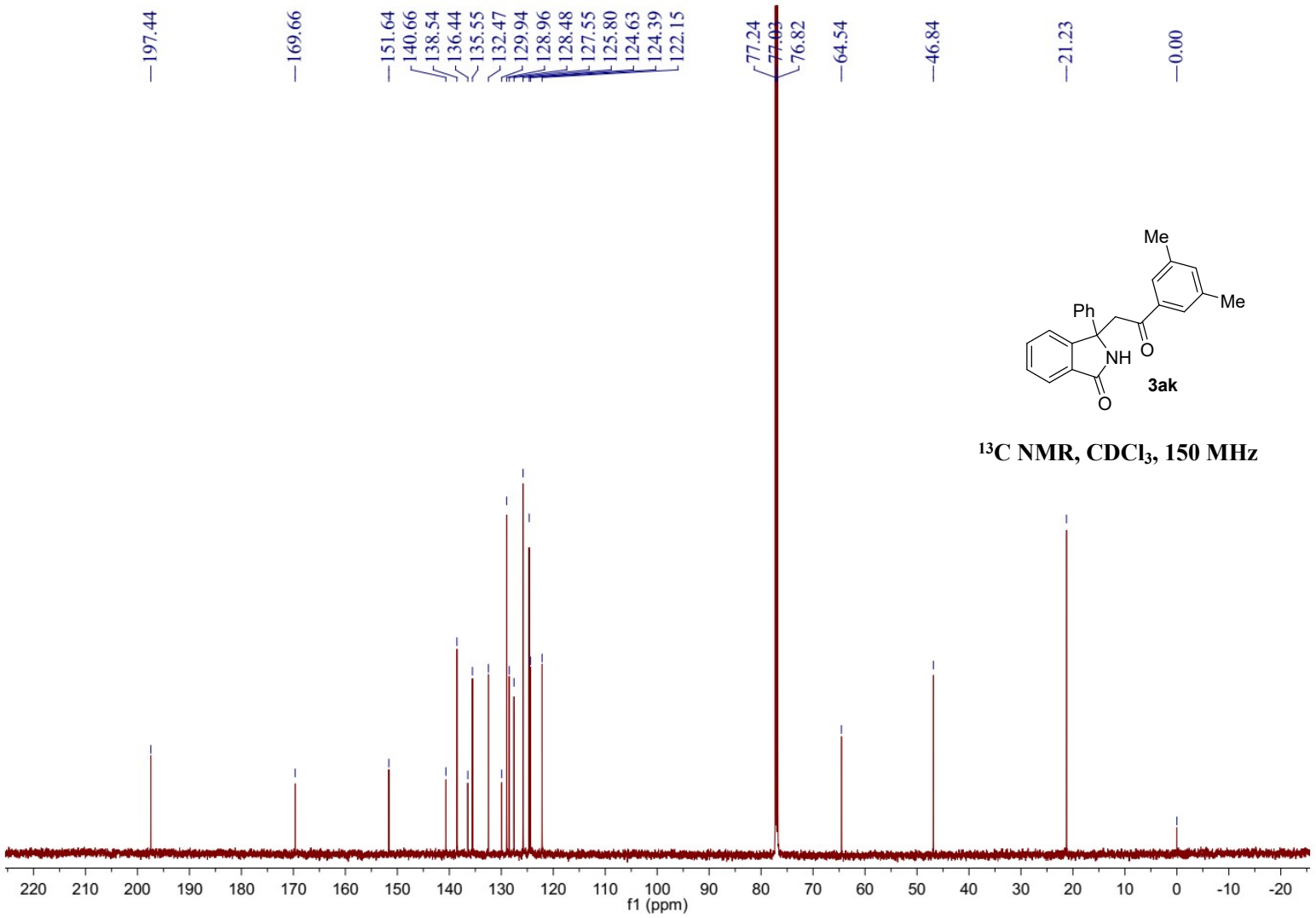
^{1}H NMR, $CDCl_3$, 600 MHz



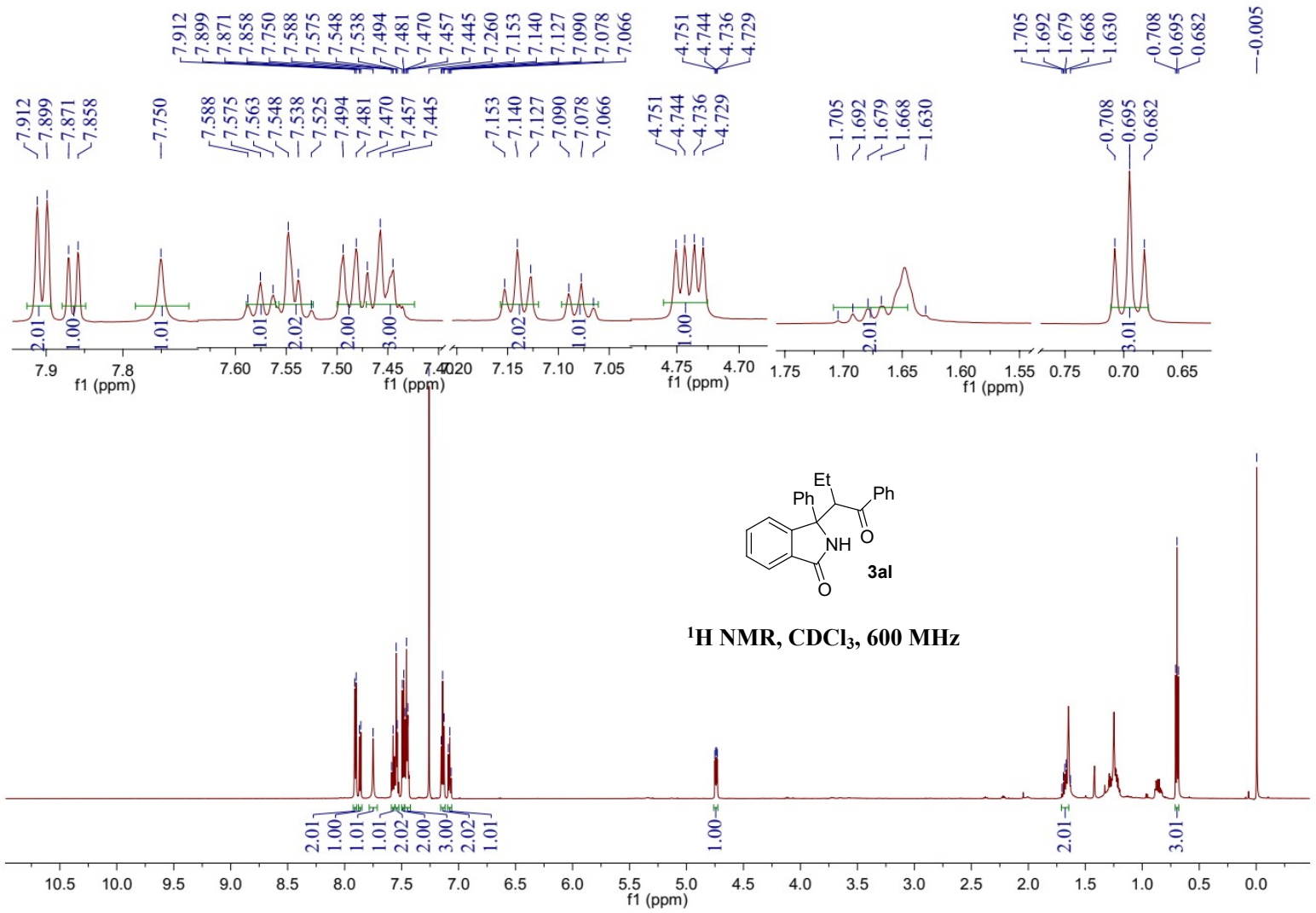
^{13}C NMR, $CDCl_3$, 150 MHz



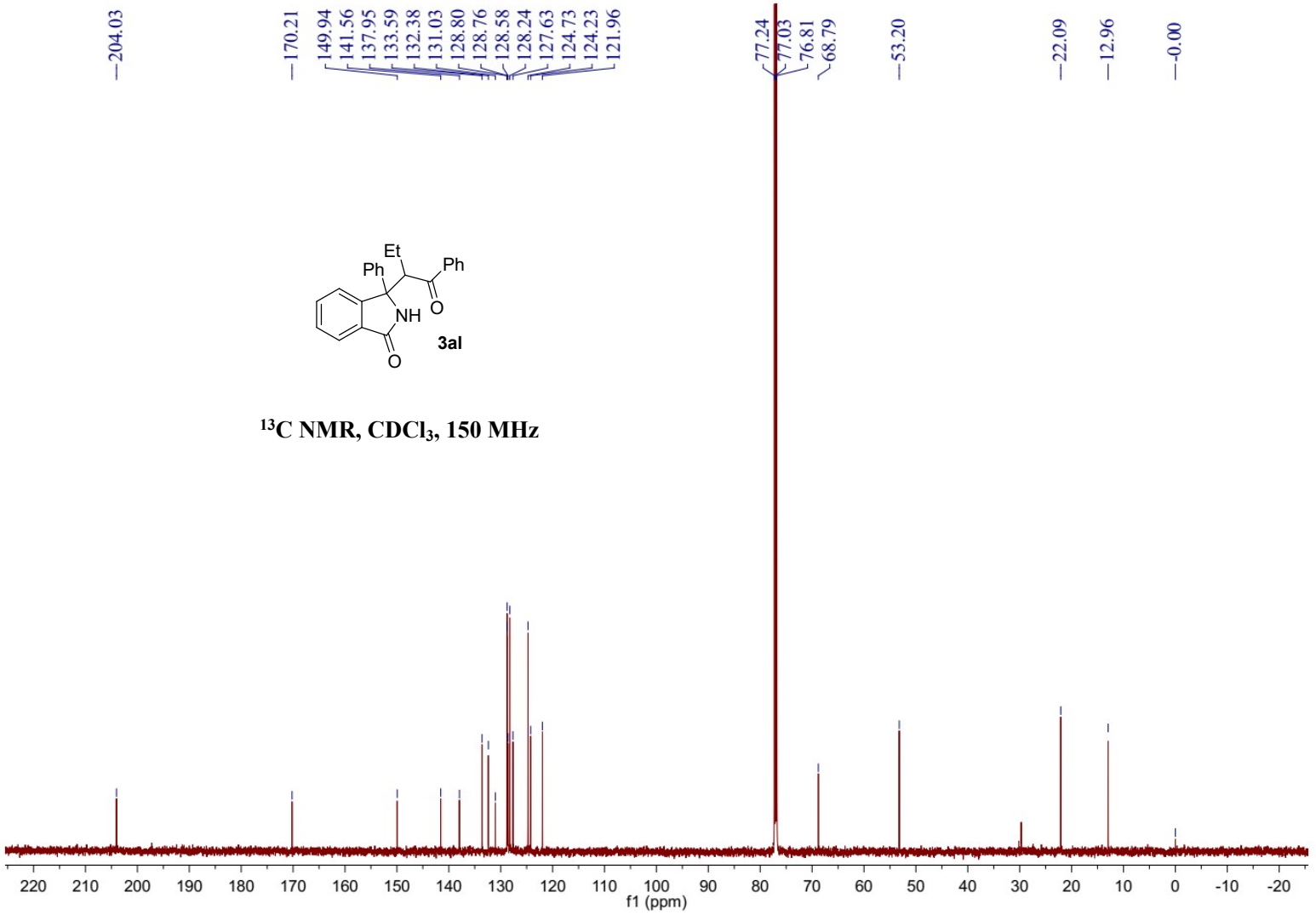
^{1}H NMR, $CDCl_3$, 600 MHz



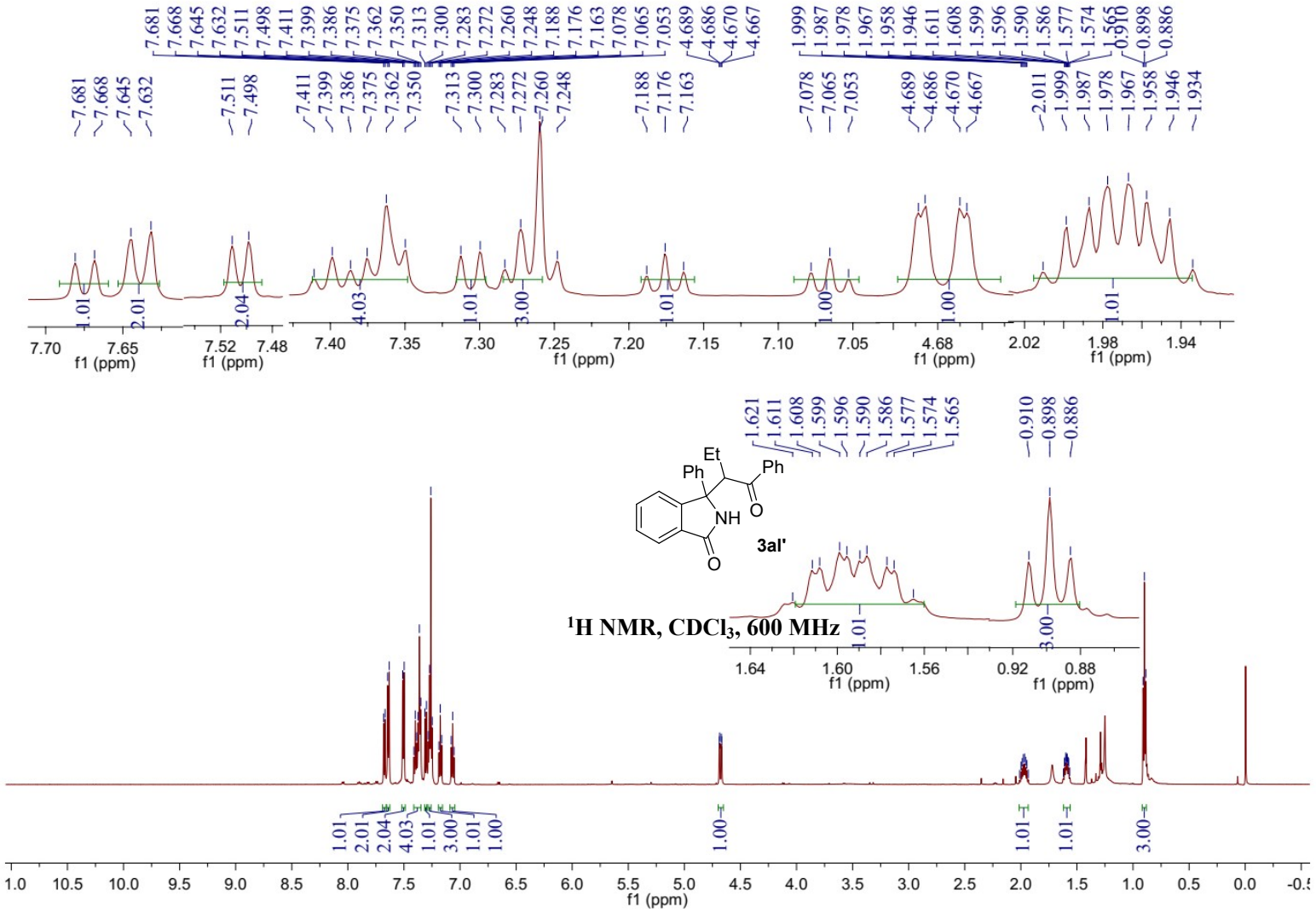
^{13}C NMR, $CDCl_3$, 150 MHz

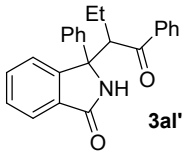


^{1}H NMR, $CDCl_3$, 600 MHz



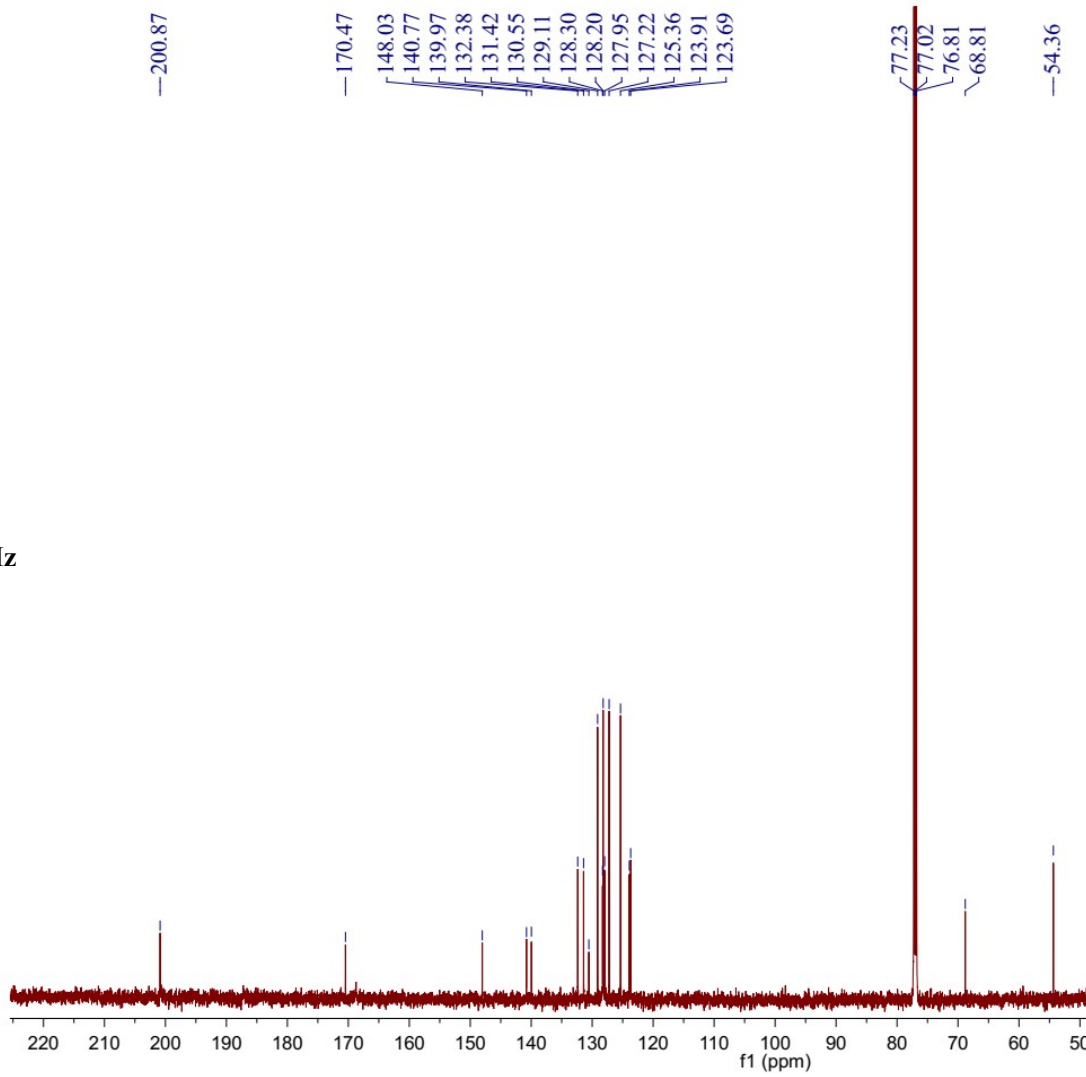
^{13}C NMR, $CDCl_3$, 150 MHz

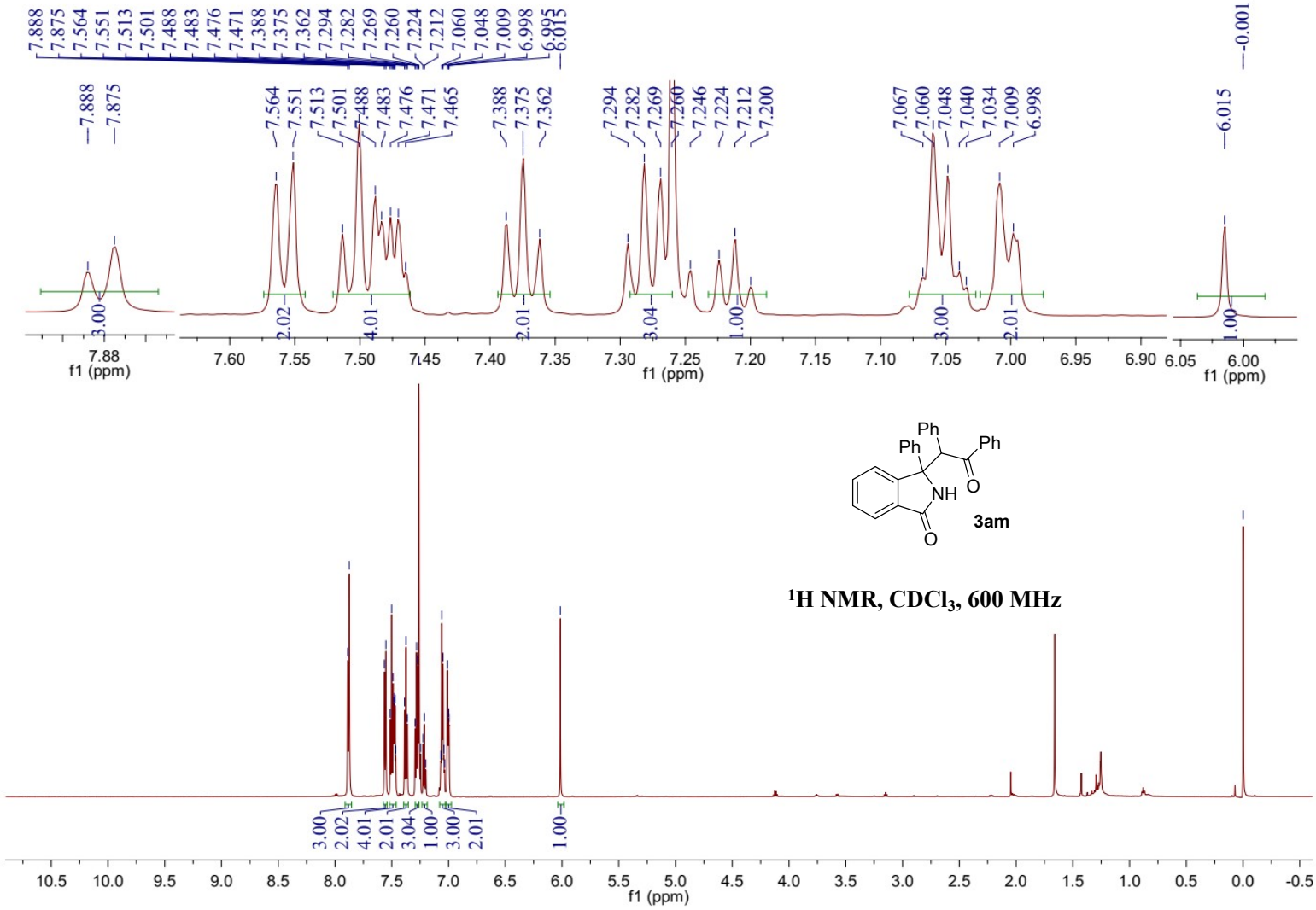

Et
Ph
Ph
O
NH
O
3al'
1H NMR, CDCl3, 600 MHz
f1 (ppm)



3al'

^{13}C NMR, $CDCl_3$, 150 MHz

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7.282
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7.260
7.224
7.212
7.060
7.048
7.009
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6.995
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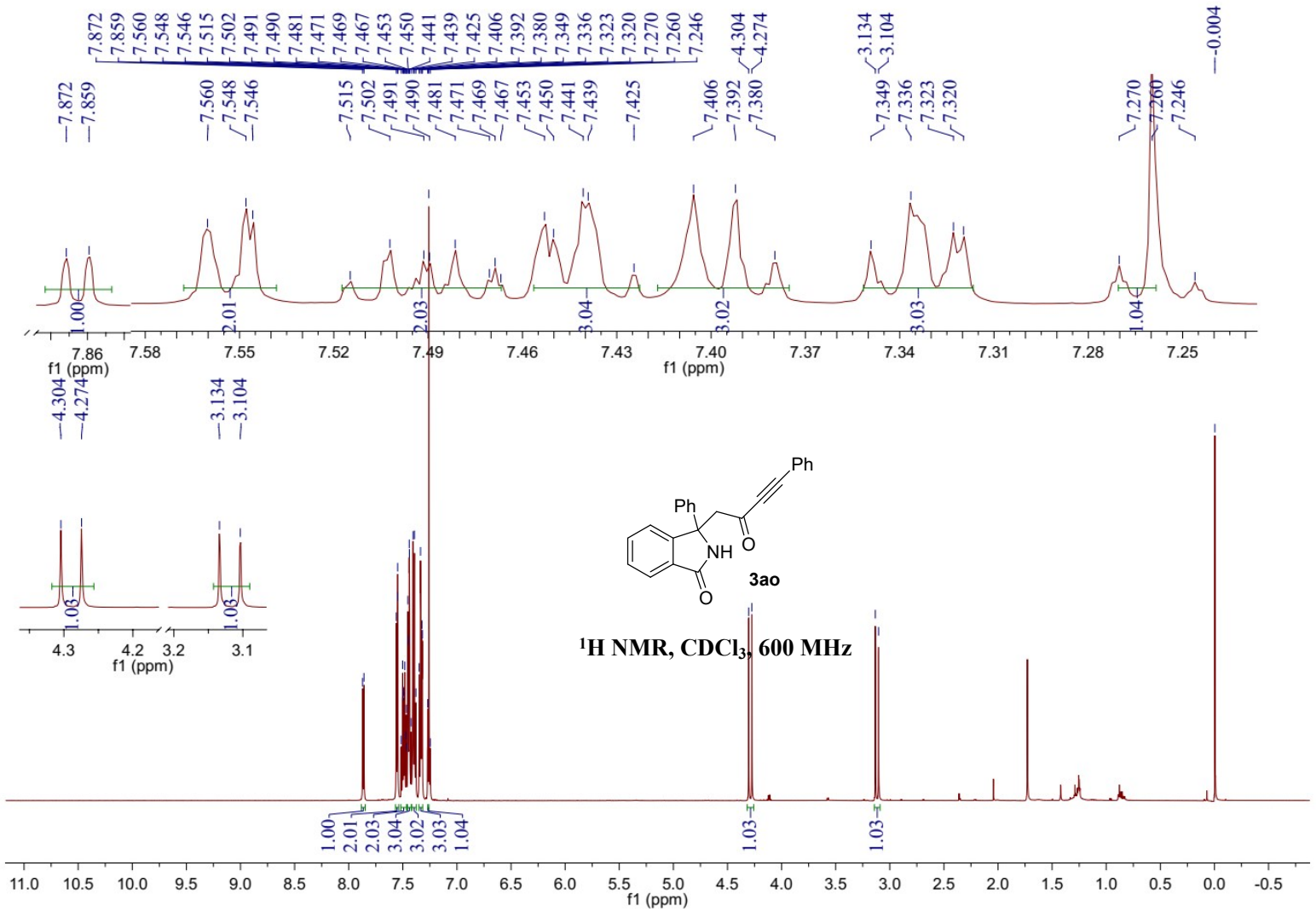
-0.001
f1 (ppm)
Ph
Ph
Ph
O
NH
O
3am
1H NMR, CDCl3, 600 MHz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7.260
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4.274
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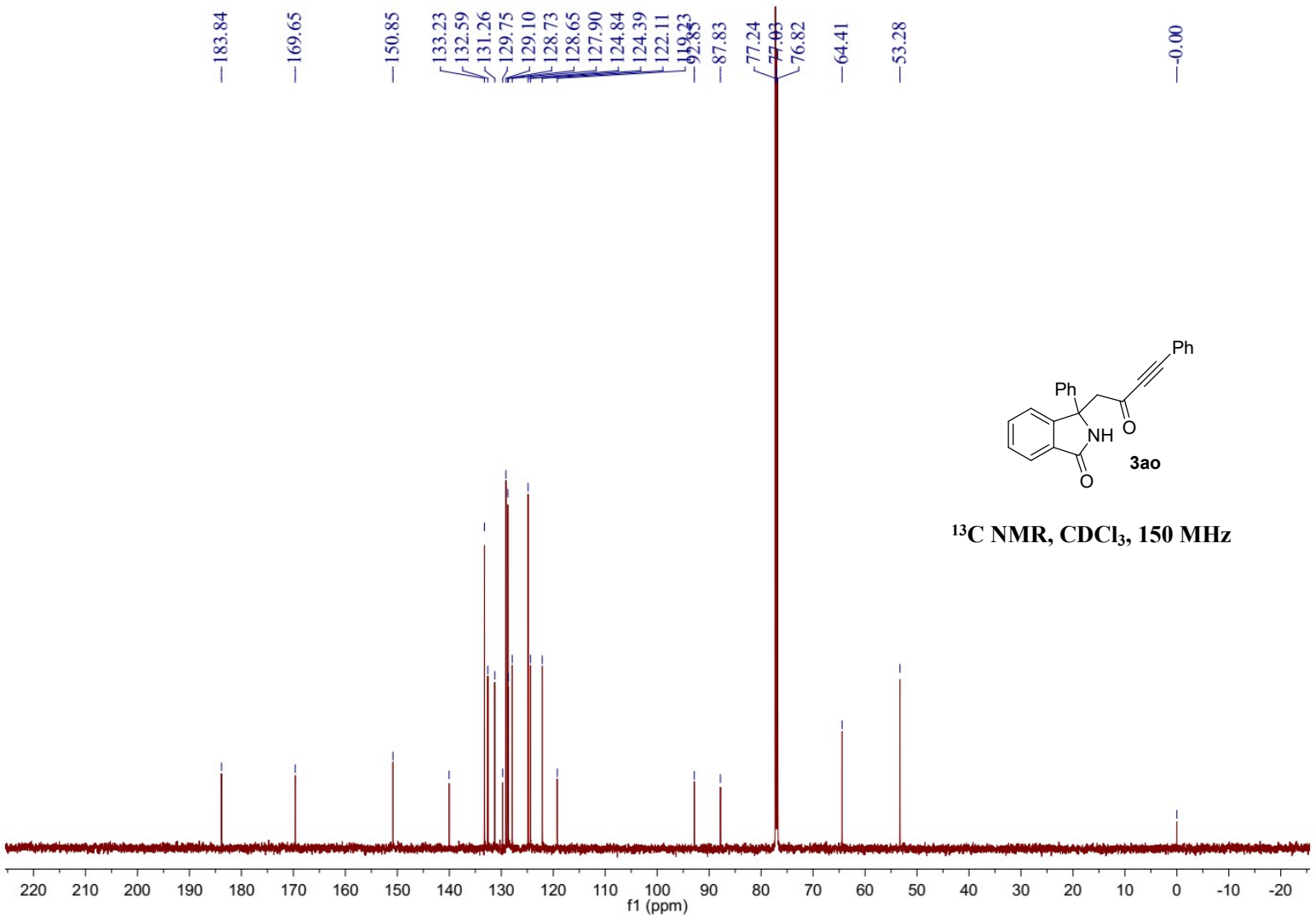
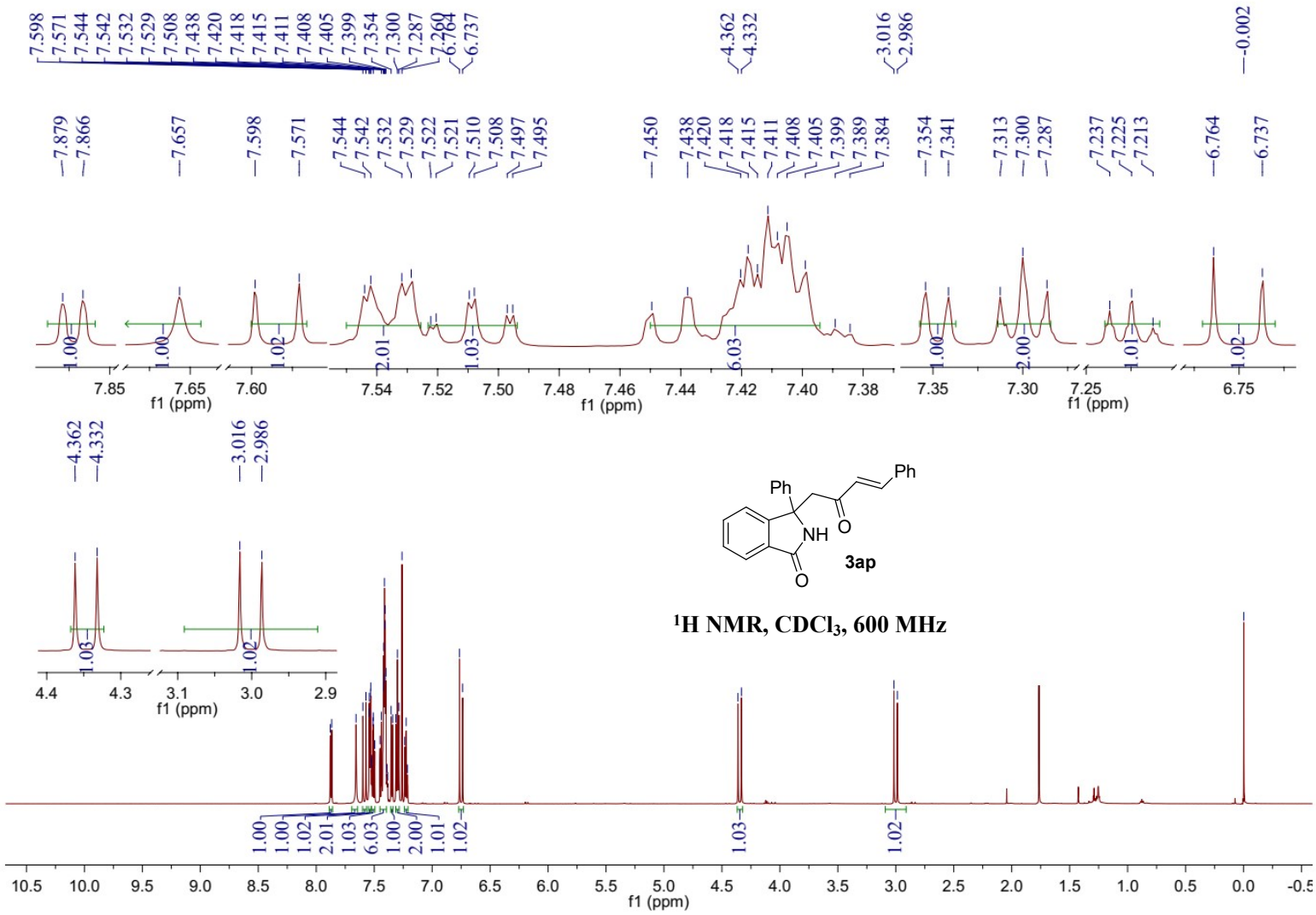
2.03
3.04
3.02
3.03
1.04
1.03
1.03
f1 (ppm)
Ph
Ph
NH
O
O
3ao
^{1}H NMR, CDCl$_3$, 600 MHz



^{13}C NMR, $CDCl_3$, 150 MHz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6.737
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4.332
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7.521
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7.508
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7.420
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7.415
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7.408
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7.313
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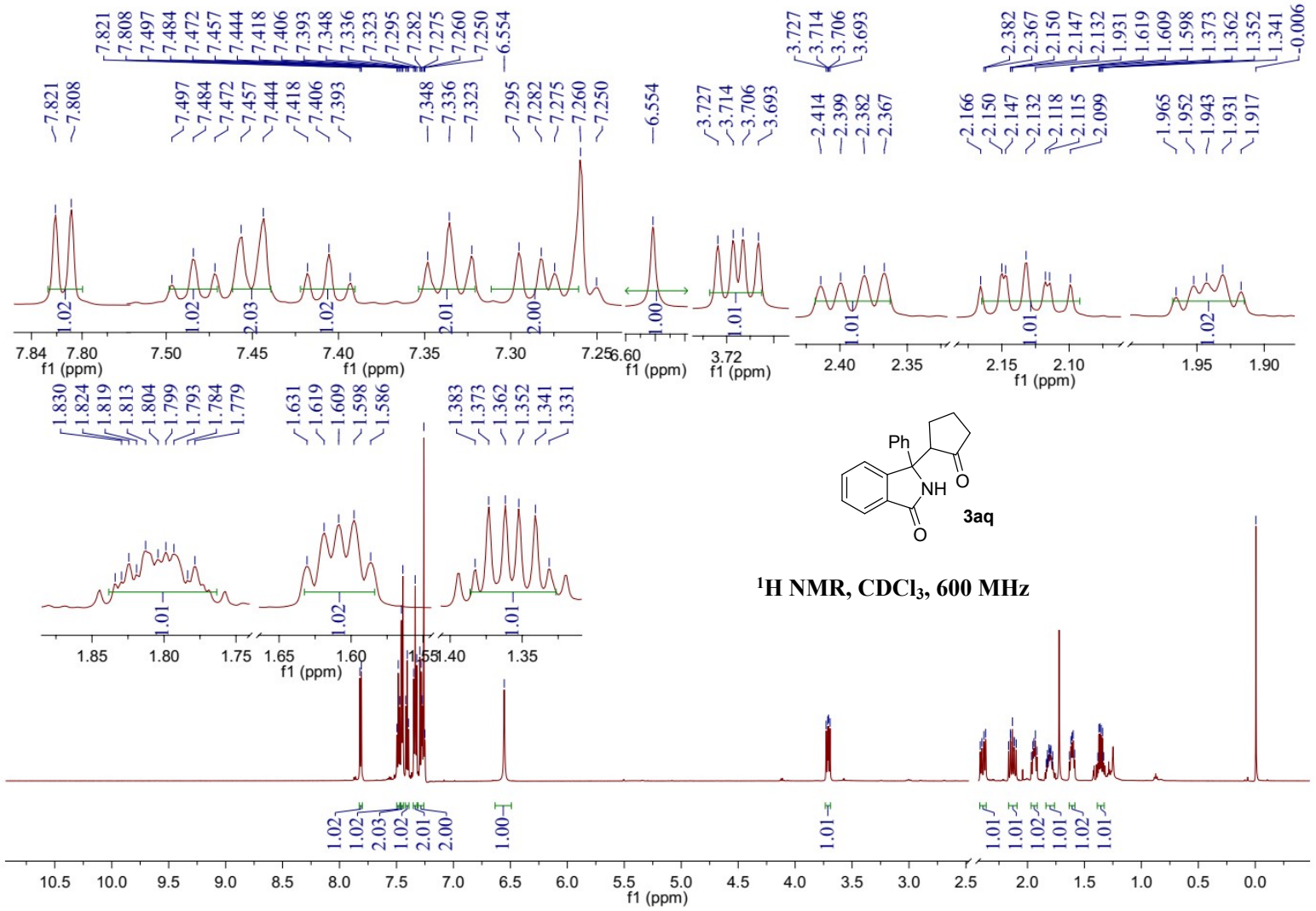
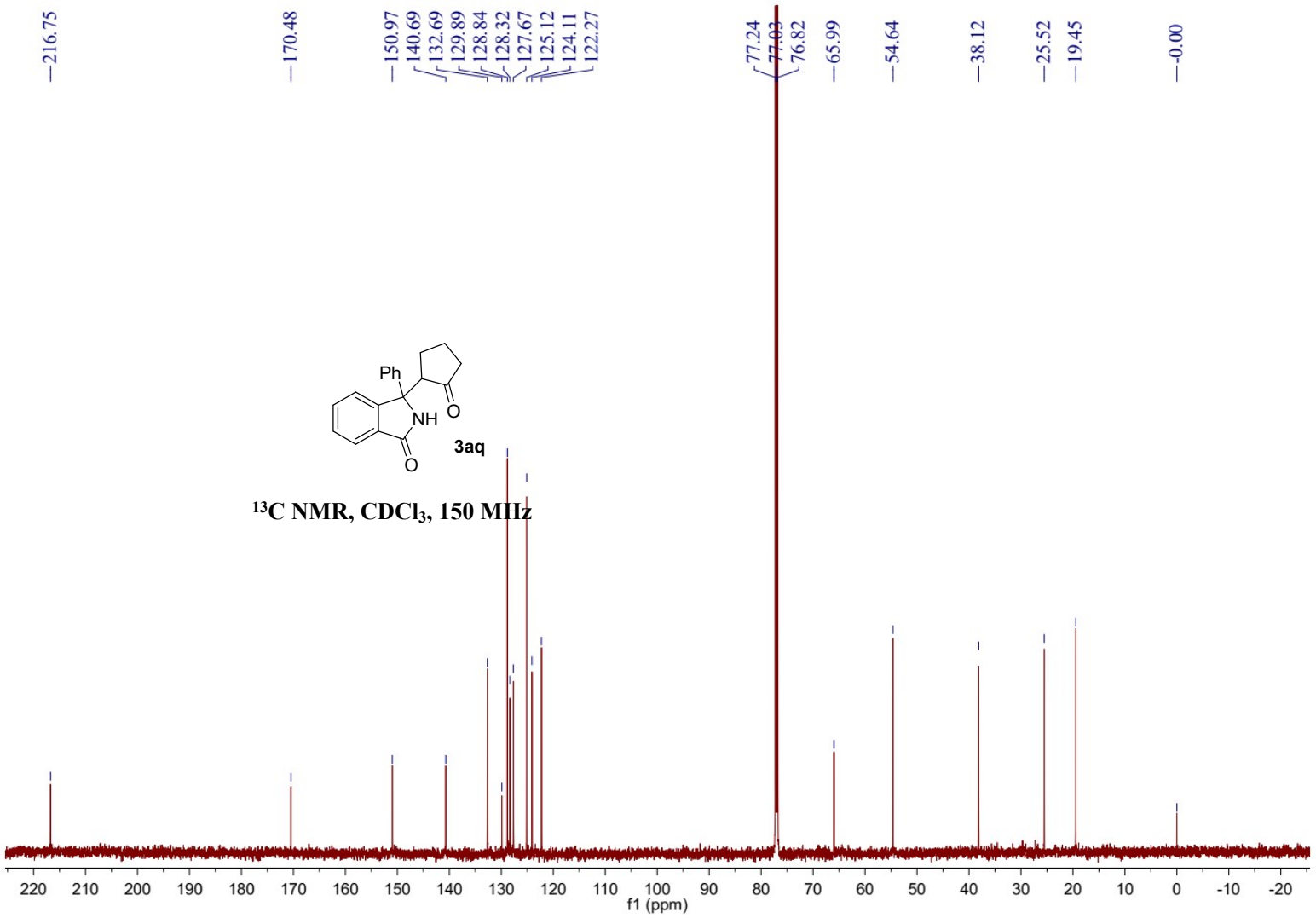
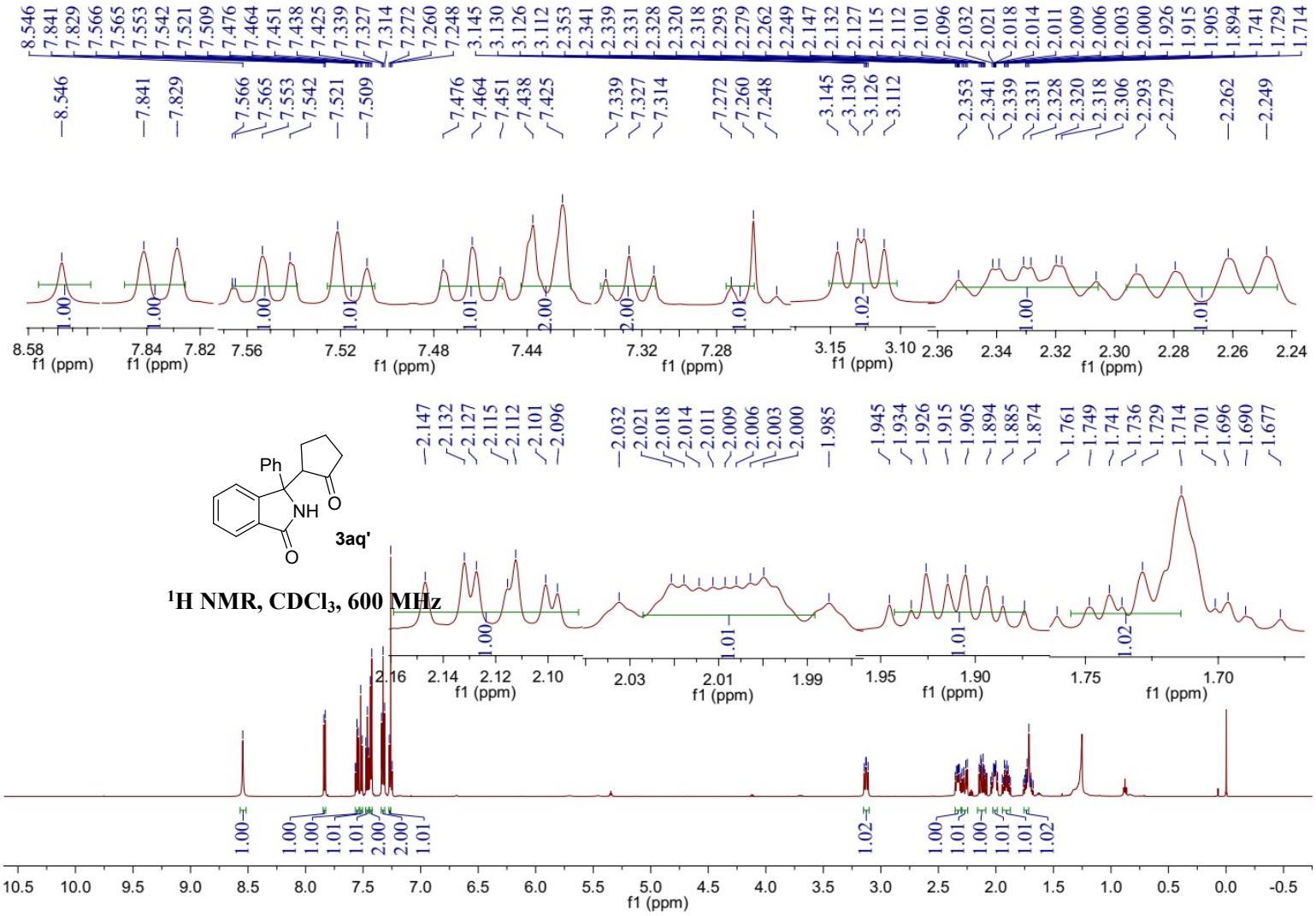
7.237
7.225
7.213
6.764
6.737
f1 (ppm)
Ph
Ph
NH
O
O
3ap
1H NMR, CDCl3, 600 MHz

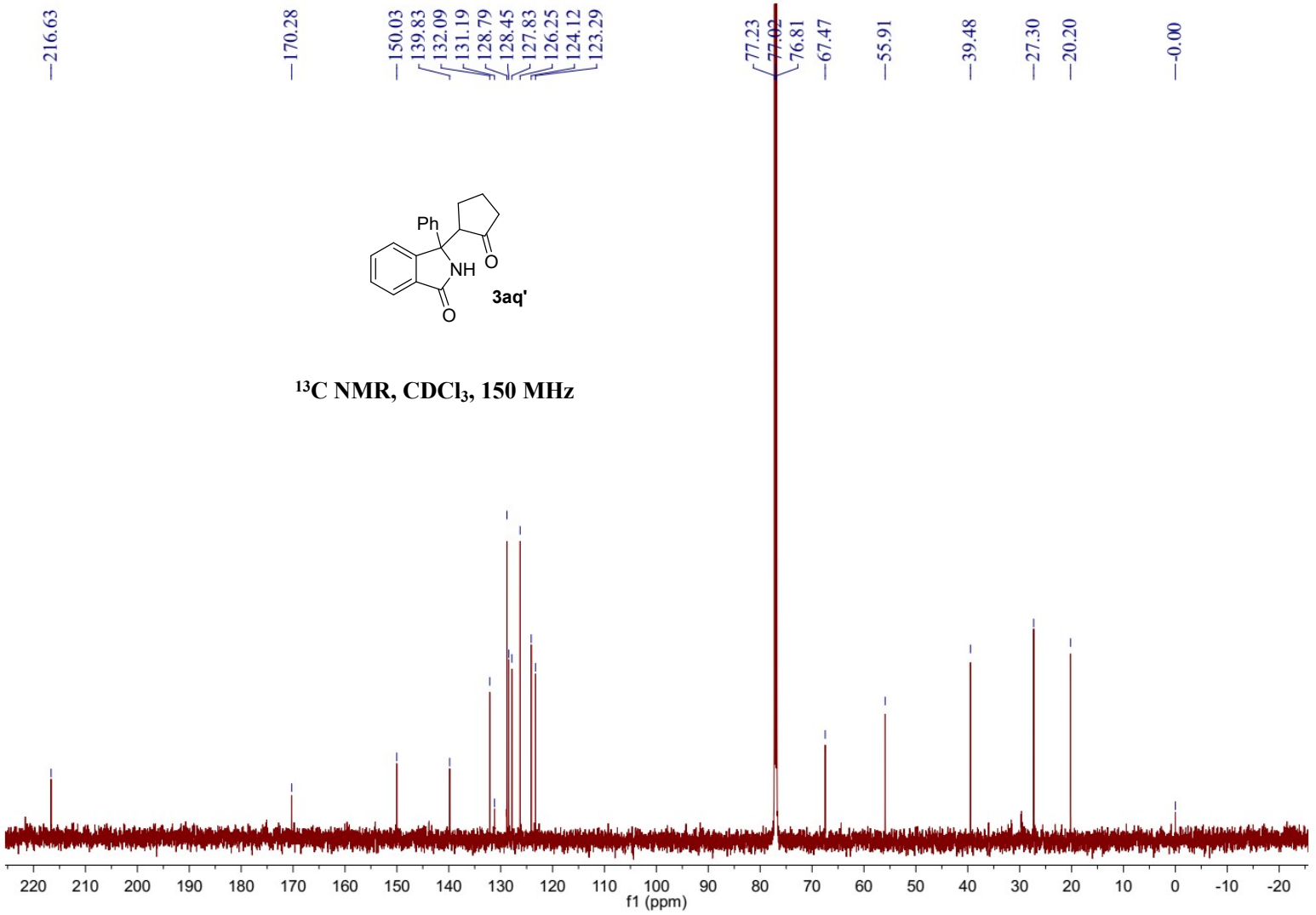


^{1}H NMR, $CDCl_3$, 600 MHz

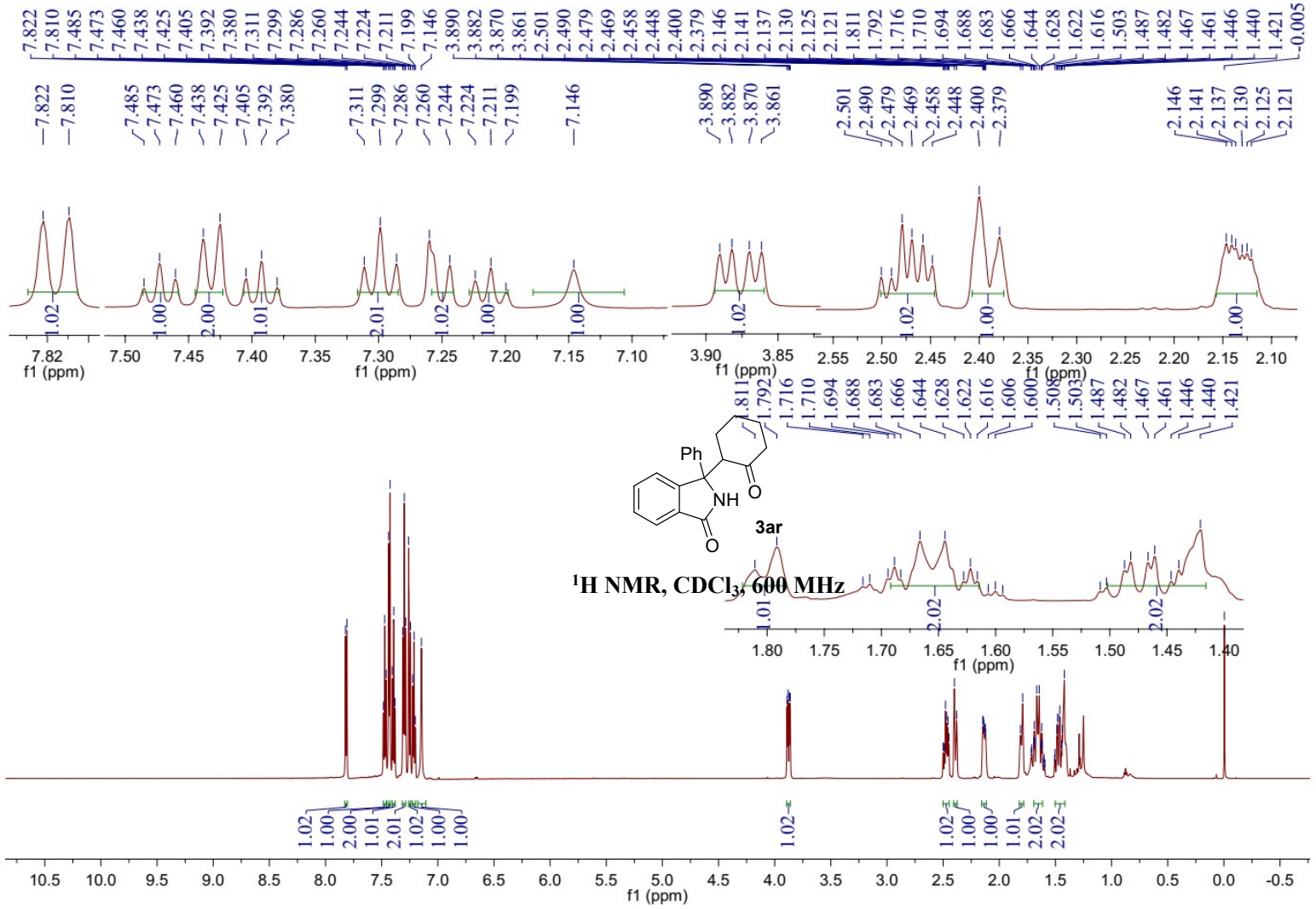


^{13}C NMR, $CDCl_3$, 150 MHz

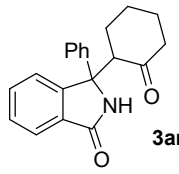

Ph
NH
O
O
3aq'
1H NMR, CDCl3, 600 MHz
f1 (ppm)



^{13}C NMR, $CDCl_3$, 150 MHz

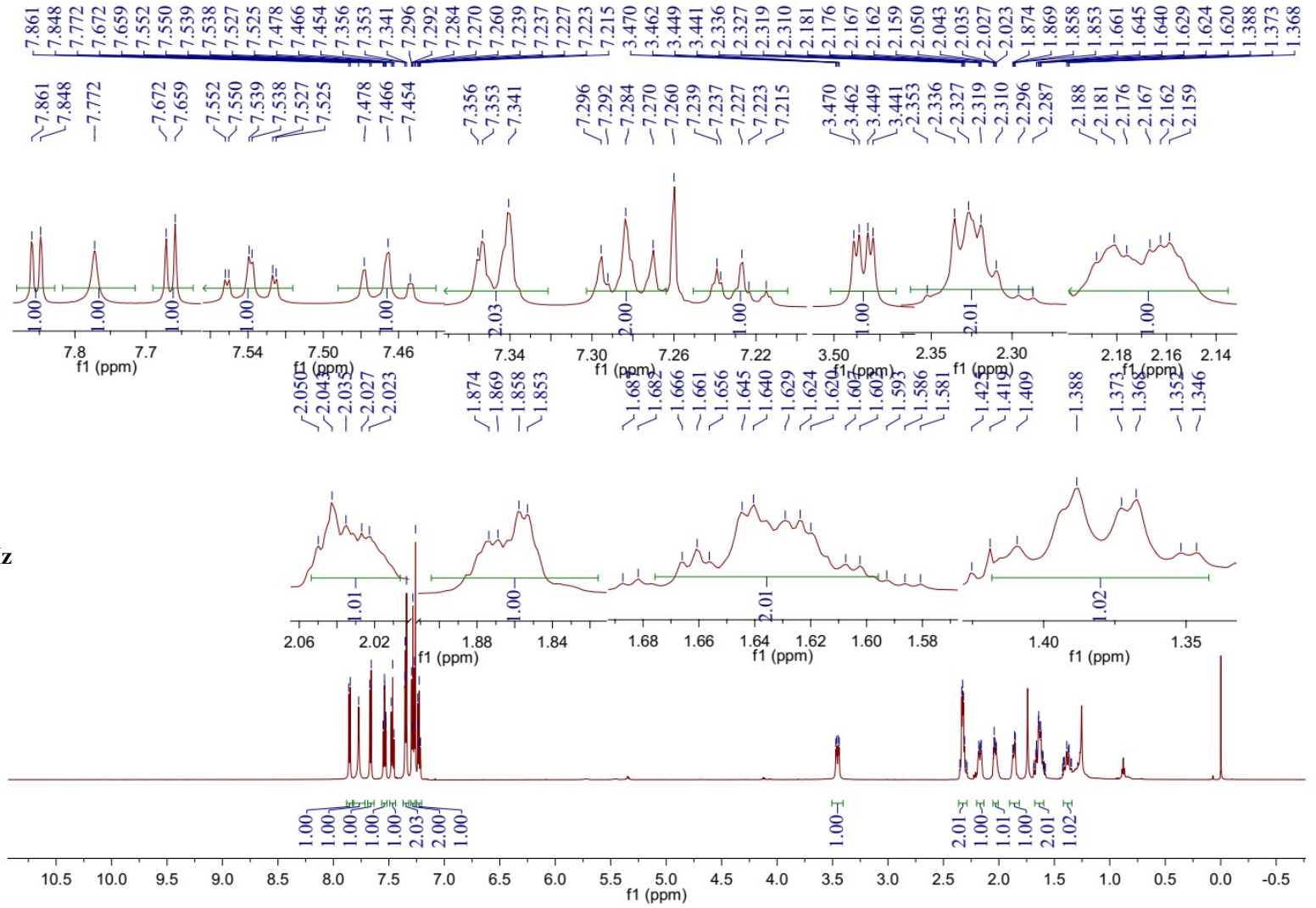


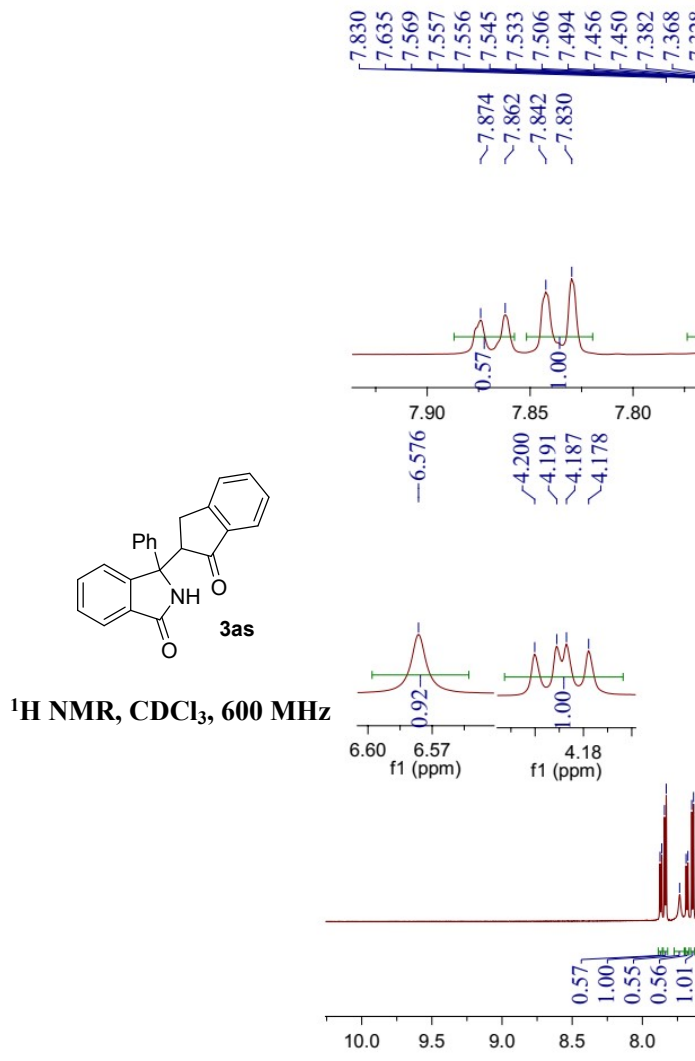
^{1}H NMR, $CDCl_3$, 600 MHz



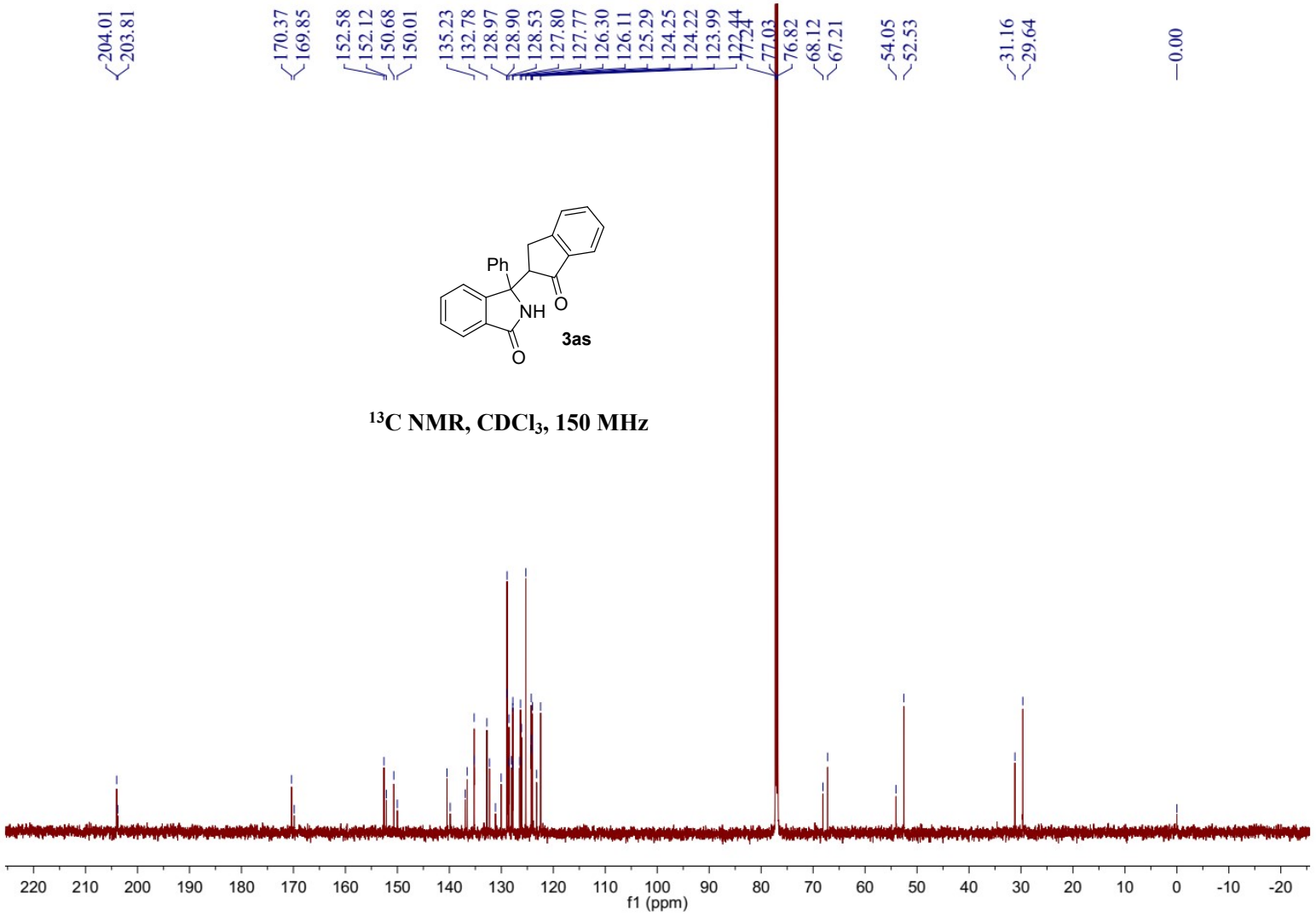
3ar'

^{1}H NMR, $CDCl_3$, 600 MHz

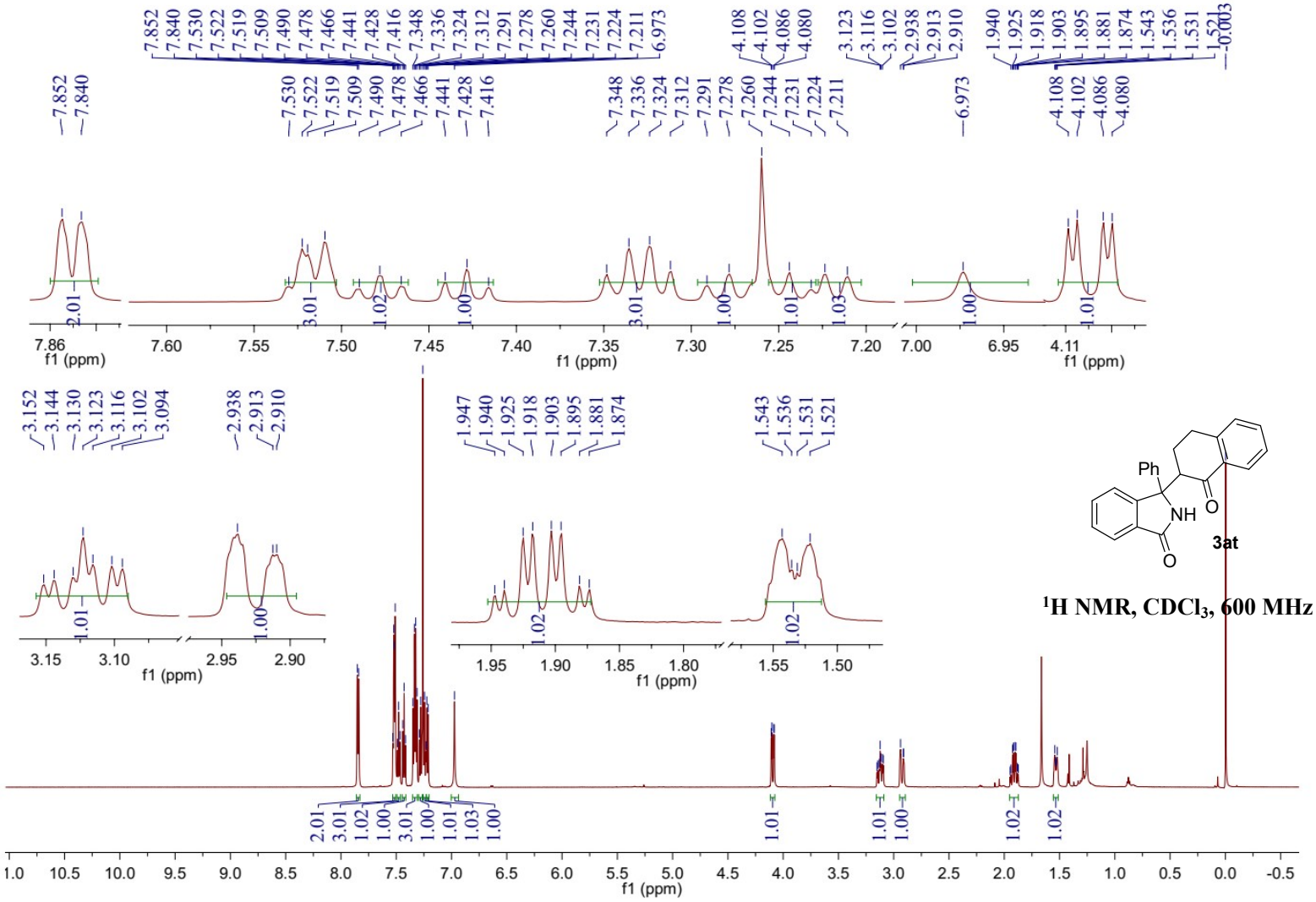




^{1}H NMR, $CDCl_3$, 600 MHz

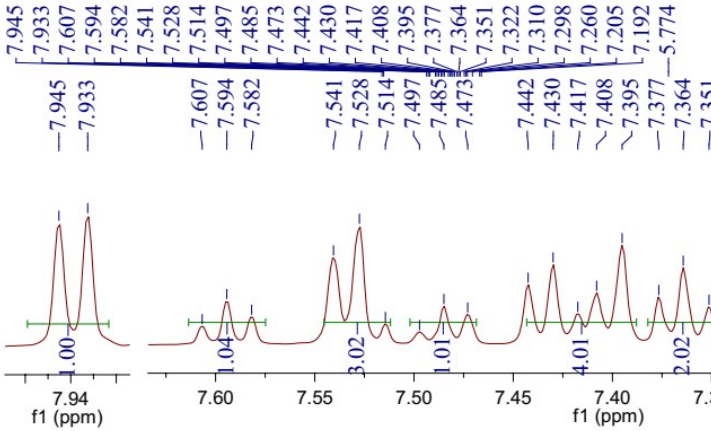


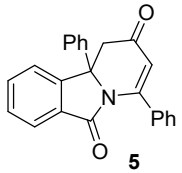
^{13}C NMR, $CDCl_3$, 150 MHz



3at

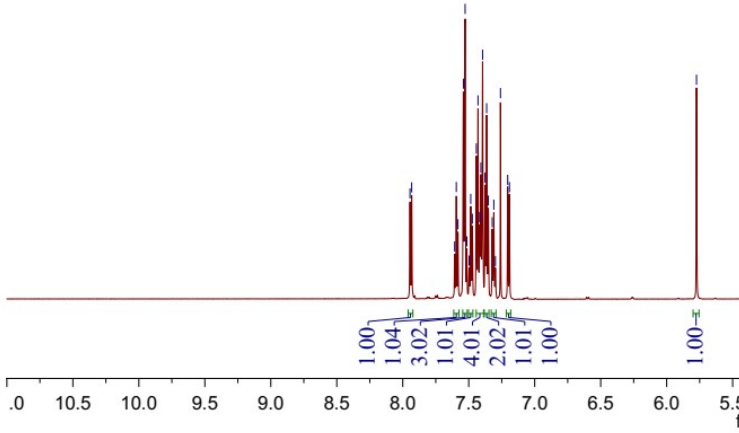
^{1}H NMR, $CDCl_3$, 600 MHz

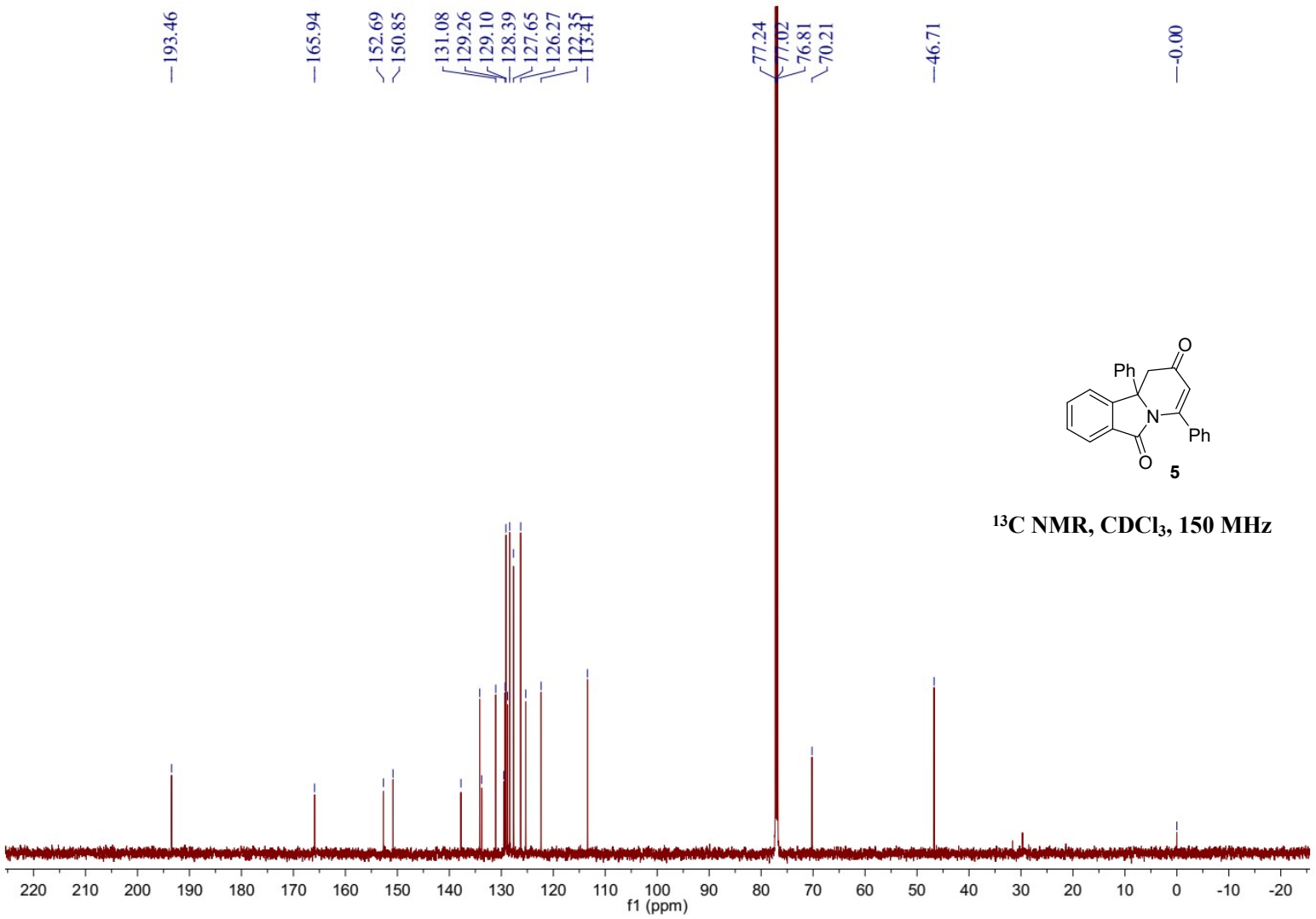




5

^{1}H NMR, $CDCl_3$, 600 MHz





^{13}C NMR, $CDCl_3$, 150 MHz