Rhodium(III) Catalyzed Arylation of Boc-Imines via C-H Bond Functionalization

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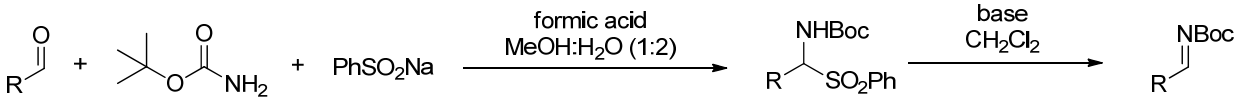
Supporting Information

I. General Methods. Unless otherwise noted, all reagents were obtained from commercial suppliers and used without further purification. Substituted 2-phenylarenes were synthesized according to published procedures.[1,2] Benzene, dichloromethane, and tetrahydrofuran were passed through a column of activated alumina under nitrogen. All reactions of air- and moisture-sensitive materials were carried out using syringe, cannula and/or inert atmosphere box techniques. All glassware was dried overnight at 150 ˚C or flame-dried under vacuum immediately prior to use. During workup procedures, organic extracts were dried over Na_2SO_4 and concentrated under reduced pressure with a rotary evaporator. Chromatography was performed on Merck 60 230-240 mesh silica gel. NMR chemical shifts are reported in ppm relative to $CHCl_3$ (7.26 ppm for ^1H, and 77.23 ppm for ^{13}C NMR) or CD_2Cl_2 (5.32 ppm for ^1H, and 53.84 ppm for ^{13}C NMR). IR spectra were recorded on a Nicolet Avatar 360 FTIR spectrometer equipped with an attenuated total reflectance accessory or a Nicolet 6700 FT-IR equipped with an attenuated total reflectance accessory, and only partial data are listed. Melting points were determined on a Mel-Temp apparatus and are reported uncorrected. Mass spectra (HRMS) were obtained by the University of California at Berkeley Mass Spectrometry Facility or the Keck Center of Yale University.

II. Synthesis of Starting Materials

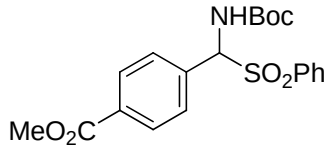
IIa. Synthesis of Sulfonyl Carbamates



In a 25 mL round bottom flask was combined aldehyde (13.5 mmol, 2 equiv), *tert*-butyl carbamate (0.850 g, 7.25 mmol, 1 equiv), sodium benzenesulfinic acid (2.90 g, 18.1 mmol, 2.5 equiv), formic acid (0.52 mL, 13.5 mmol, 2 equiv), MeOH (8 mL), and water (15 mL). The reaction mixture was stirred at rt for 24 h during which time it becomes heterogeneous. The

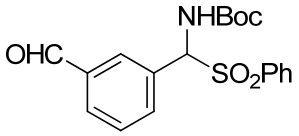
reaction mixturewas filtered and the precipitate was washed with water and Et_2O to yield the sulfonyl carbamate as a white powder.

The synthetic procedures and characterization have previously been reported for the following sulfonyl carbarmates: *N-tert*-butyl((2-methylphenyl)(phenylsulfonyl)methyl)-carbamate (R = 2-MeC_6H_4),[3] *N-tert*-butyl ((4-methoxyphenyl)(phenylsulfonyl)methyl)carbamate (R = 4-$OMeC_6H_4$),[3] *N-tert*-butyl((2-thienyl)(phenylsulfonyl)methyl)carbamate (R = 2-thienyl),[3] *N-tert*-butyl(4-(trifluoromethyl)phenyl(phenylsulfonyl)methyl)carbamate (R = 4-$CF_3C_6H_4$),[4] *N-tert*-butyl(4-chlorophenyl(phenylsulfonyl)methyl)carbamate (R = 4-ClC_6H_4),[5] *N-tert*-butyl(2-chlorophenyl(phenylsulfonyl)methyl)carbamate (R = 2-ClC_6H_4),[5] *N-tert*-butyl *N*-(4-nitrophenyl-(phenylsulfonyl)methyl)carbamate (R = 4-$NO_2C_6H_4$),[4] *N-tert*-butyl(4-methylphenyl(phenyl-sulfonyl)methyl)carbamate (R = 4-MeC_6H_4),[5] *N-tert*-butylphenyl(phenylsulfonyl)methyl)-carbamate (R = C_6H_5).[5]

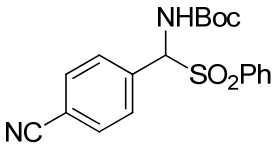


***N-tert*-Butyl(4-methoxycarbonylphenyl(phenylsulfonyl)methyl)carbamate** The synthesis was performed according to the general procedure using 4-methoxycarbonylbenzaldehyde (2.79 g, 17.0 mmol, 2 equiv), *tert*-butyl carbamate (1.02 g, 8.71 mmol, 1 equiv), sodium benzenesulfinic acid (3.55 g, 21.6 mmol, 2.5 equiv), formic acid (0.60 mL, 16 mmol, 2 equiv), MeOH (10 mL), and water (18 mL) yielded the sulfonyl carbamate as a white powder (1.37 g, 3.38 mmol, 39% yield). mp: 176 ºC (decomp.). IR (neat): 1726 (s), 1694 (s), 1506 (m), 1505 (m), 1308 (s), 1270 (s), 1248 (m), 1161 (w), 1142 (s), 1104 (m), 1085 (m), 1018 (w), 764 (w), 726 (m), 710 (m), 689 (m) cm^{-1}. ^1H NMR (600 MHz, $CDCl_3$) δ 8.09 (d, *J* =

8.1 Hz, 2H), 7.94 (d, *J* = 7.5 Hz, 2H), 7.68 (t, *J* = 7.4 Hz, 1H), 7.61 – 7.50 (m, 4H), 6.04 (d, *J* = 10.7 Hz, 1H), 5.97 (d, *J* = 10.6 Hz, 1H), 3.96 (s, 3H), 1.28 (s, 9H). $^{13}C\{^{1}H\}$ NMR (151 MHz, $CDCl_3$) δ 166.36, 153.50, 136.66, 134.72, 134.19, 131.38, 129.84, 129.48, 129.17, 129.01, 81.48, 73.63, 52.34, 28.01. HRMS (ESI+) Calcd for $C_{20}H_{23}NO_6NaS$ $[M+Na]^+$ 428.1138; Found 428.1131.



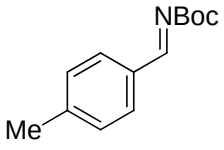
***N-tert*-Butyl(3-carbonylphenyl(phenylsulfonyl)methyl)carbamate** The synthesis was performed according to the general procedure using isophthalaldehyde (1.15 g, 8.57 mmol, 2 equiv), *tert*-butyl carbamate (0.50 g, 4.3 mmol, 1 equiv), sodium benzenesulfinic acid (1.76 g, 10.7 mmol, 2.5 equiv), formic acid (0.3 mL, 8 mmol, 2 equiv), MeOH (5 mL), and water (9 mL). The obtained solid was heated at reflux for 10 min in Et_2O (20 mL) and filtered hot to remove excess isophthalaldehyde. The sulfonyl carbarmate was isolated as a white solid (0.67 g, 1.8 mmol, 42% yield). mp: 160 ºC (decomp). IR (neat): 1715 (m), 1688 (s), 1526 (m), 1448 (w), 1307 (m), 1169 (m), 1145 (s), 1084 (m), 800 (m), 715 (m), 686 (s) cm^{-1}. ^{1}H NMR (500 MHz, $CDCl_3$) δ 10.08 (s, 1H), 8.06 – 7.93 (m, 4H), 7.82 (d, *J* = 7.5 Hz, 1H), 7.73 (t, *J* = 7.1 Hz, 1H), 7.69 – 7.58 (m, 3H), 6.09 (d, *J* = 10.5 Hz, 1H), 5.92 (d, *J* = 10.1 Hz, 1H), 1.30 (s, 9H). $^{13}C\{^{1}H\}$ NMR (126 MHz, $CDCl_3$) δ 191.53, 153.56, 153.40, 136.64, 136.50, 135.07, 134.30, 131.20, 131.01, 129.85, 129.57, 129.31, 81.66, 73.21, 28.03. HRMS (ESI+) Calcd for $C_{19}H_{21}NO_5NaS$ $[M+Na]^+$ 398.1033; Found 398.1024.



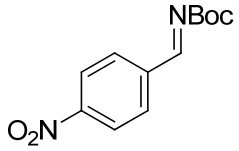
***N-tert*-Butyl(4-cyanophenyl(phenylsulfonyl)methyl)carbamate** The synthesis was performed according to the general procedure using 4-cyanobenzaldehyde (1.19 g, 9.08 mmol, 2 equiv), *tert*-butyl carbamate (0.51 g, 4.3 mmol, 1 equiv), sodium benzenesulfinic acid (1.73 g, 10.5 mmol, 2.5 equiv), formic acid (0.3 mL, 8 mmol, 2 equiv), MeOH (5 mL), and water (9 mL). The obtained solid was heated at refluxed for 10 min in Et_2O (20 mL) and filtered hot to remove excess 4-cyanobenzaldehyde. The sulfonyl carbamate was isolated as a white solid (0.665 g, 1.79 mmol, 41% yield). mp: 158 ºC (decomp.). IR (neat): 2231 (w), 1695 (m), 1498 (m), 1447 (w), 1305 (m), 1247 (m), 1138 (s), 1078 (m), 797 (m) cm^{-1}. ^1H NMR (600 MHz, $CDCl_3$) δ 7.95 (d, *J* = 7.2 Hz, 2H), 7.77 – 7.66 (m, 3H), 7.65 – 7.55 (m, 4H), 6.04 (d, *J* = 10.7 Hz, 1H), 5.95 (d, *J* = 10.5 Hz, 1H), 1.27 (s, 9H). $^{13}C\{^1H\}$ NMR (151 MHz, $CDCl_3$) δ 153.41, 136.35, 135.12, 134.44, 132.34, 129.75, 129.46, 129.30, 118.09, 113.71, 81.76, 73.29, 27.98. HRMS (ESI+) Calcd for $C_{19}H_{20}N_2O_4NaS$ $[M+Na]^+$ 395.1036; Found 395.1034.

IIb. General Synthesis Procedure I for the Preparation of Boc-Imines

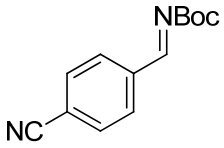
In a typical experiment sulfonyl carbamate (0.5 mmol) was dissolved in CH_2Cl_2 (8 mL) and 1.4 M K_2CO_3 (8 mL) was added. The biphasic solution was stirred vigorously at rt for 4 h. The organic layer was separated and the aqueous layer was washed two times with 10 mL of CH_2Cl_2. The combined organics were concentrated *in vacuo* at rt to yield the Boc-imine.



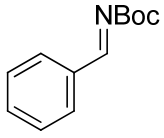
***N-tert*-Butyl-4-methylbenzylidenecarbamate** was synthesized according to the general synthesis procedure I using 1 mmol of sulfonyl carbamate and was obtained as a colorless oil (182 mg, 0.830 mmol, 83% yield). Characterization of this compound has been reported previously.[3]



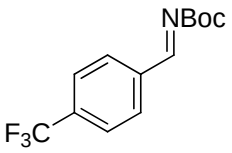
***N-tert*-Butyl-4-nitrobenzylidenecarbamate** was synthesized according to the general synthesis procedure I and was obtained as a white solid (109 mg, 0.437 mmol, 87% yield). Characterization of this compound has been reported previously.[4]



***N-tert*-Butyl-4-cyanobenzylidenecarbamate** was synthesized according to the general synthesis procedure I and was obtained as a white solid (73 mg, 0.32 mmol, 63% yield). mp: 85-87 ˚C. IR (film): 2230, 1698, 1631 cm^{-1}. ^{1}H NMR (400 MHz, $CDCl_3$): δ 8.83 (s, 1H), 8.02 (d, *J* = 8.6 Hz, 2H), 7.78 (d, *J* = 8.6 Hz, 2H), 1.59 (s, 9H). $^{13}C\{^{1}H\}$ NMR (100 MHz, $CDCl_3$): δ 166.7, 161.9, 137.7, 132.5, 130.2, 117.9, 116.4, 83.1, 27.8. HRMS (ESI+) Calcd for $C_{13}H_{15}N_2O_2$ $[MH]^+$ 231.1129; Found 231.1128.



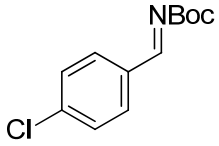
***N-tert*-Butyl-benzylidenecarbamate** was synthesized according to the general synthesis procedure I and was obtained as a colorless oil (57 mg, 0.28 mmol, 51% yield). Characterization of this compound has been reported previously.[5]



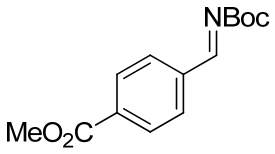
***N-tert*-Butyl-4-(trifluoromethyl)benzylidenecarbamate** was synthesized according to the general synthesis procedure I and was obtained as a white solid (80 mg, 0.30 mmol, 59% yield). Characterization of this compound has been reported previously.[4]

IIc. General Synthesis Procedure II for the Preparation of Boc-Imines II

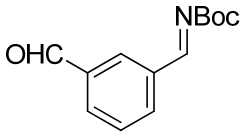
In a typical experiment the sulfonyl carbamate (0.75 mmol) was dissolved in CH_2Cl_2 (10 mL) and dry Cs_2CO_3 (2.4 g, 7.5 mmol, 10 equiv) was added. The mixture was stirred vigorously at rt for 1 h. The slurry was filtered and the organic phase concentrated *in vacuo* to afford the desired pure Boc-imine.



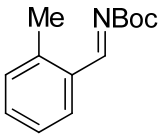
***N-tert*-Butyl-4-chlorobenzylidenecarbamate** was synthesized according to the general synthesis procedure II using of 0.37 mmol sulfonyl carbamate and was obtained as a white solid (54 mg, 0.225 mmol, 72% yield). Characterization of this compound has been reported previously.[5]



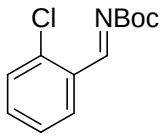
***N-tert*-Butyl-4-methoxycarbonylbenzylidenecarbamate** was synthesized according to the general synthesis procedure II and was obtained as a white solid (129 mg, 0.430 mmol, 55% yield). Characterization of this compound has been reported previously.[6]



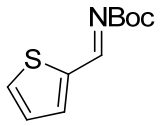
***N-tert*-Butyl-3-carbonylbenzylidenecarbamate** was synthesized according to the general synthesis procedure II and was obtained as a colorless oil (90 mg, 0.39 mmol, 50% yield). IR (neat): 1699 (s), 1633 (m), 1603 (w), 1368 (m), 1240 (s), 1138 (s), 984 (w), 851 (w), 802 (w), 682 (m) cm^{-1}. ^{1}H NMR (500 MHz, $CDCl_3$) δ 10.08 (s, 1H, CHO), 8.92 (s, 1H, CHN), 8.47 – 8.34 (m, 1H), 8.25 – 8.14 (m, 1H), 8.09 (dt, *J* = 7.7, 1.4 Hz, 1H), 7.66 (t, *J* = 7.7 Hz, 1H), 1.60 (s, 9H). $^{13}C\{^{1}H\}$ NMR (151 MHz, $CDCl_3$) δ 191.18, 167.78, 162.12, 136.96, 135.29, 135.10, 133.41, 131.51, 129.71, 82.84, 27.93. HRMS (ESI+) Calcd for $C_{13}H_{15}NO_3Na$ $[M+Na]^+$ 256.0944; Found 256.0946.



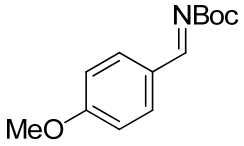
***N-tert*-Butyl-2-methylbenzylidenecarbamate was** synthesized according to the general synthesis procedure II using 0.29 mmol of sulfonyl carbamate and was obtained as a colorless oil (11 mg, 0.050 mmol, 17% yield). Characterization of this compound has been reported previously.[3]



***N-tert*-Butyl-2-chlorobenzylidenecarbamate** was synthesized according to the general synthesis procedure II using 0.6 mmol of the sulfonyl carbamate and was obtained as a colorless oil (132 mg, 0.551 mmol, 92% yield). Characterization of this compound has been reported previously.[5]

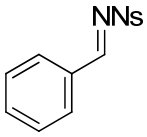


***N-tert*-Butyl-(thiophen-2-ylmethylene)carbamate** was synthesized according to the general synthesis procedure II using 0.58 mmol of the sulfonyl carbamate and was obtained as a slightly orange solid (112 mg, 0.530 mmol, 92% yield). Characterization of this compound has been reported previously.[3]

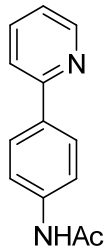


***N-tert*-Butyl-4-methoxybenzylidenecarbamate** was synthesized according to the general synthesis procedure II using 0.99 mmol of the sulfonyl carbamate and was obtained as a white solid (87 mg, 0.38 mmol, 37% yield). Characterization of this compound has been reported previously.[5]

IId. Synthesis of Other Starting Materials.



***N*-Benzylidene-4-nitrobenzenesulfonamide.** In a 50 mL round flask equipped with a magnetic stir bar, $FeCl_3$ (15 mg, 0.092 mmol, 0.04 equiv) was dissolved in EtOH (25 mL) and 4-nitrobenzenesulfonamide (506 mg, 2.50 mmol, 1 equiv) and freshly distilled benzaldehyde (0.51 mL, 5.0 mmol, 2 equiv) were subsequently added. The solution was stirred vigorously at rt for 16 h. All volatiles were then removed *in vacuo* and the resulting solid was crystallized from hot AcOEt/hexanes (1:3). The product was obtained as an off-white powder (360 mg, 1.24 mmol, 50%). Characterization of this compound has been reported previously.[7]

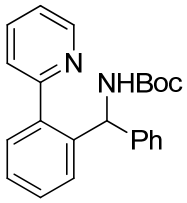


***N*-(4-(Pyridin-2-yl)phenyl)acetamide.** In a 10 mL round bottom flask was combined 4-(pyridin-2-yl)aniline[1] (200 mg, 1.18 mmol, 1 equiv), K_2CO_3 (490 mg, 3.5 mmol, 3 equiv), and CH_2Cl_2 (5 mL). A solution of acetyl chloride in dichloromethane (1 M, 1.7 mL, 1.7 mmol, 1.5 equiv) was added dropwise. The reaction mixture was stirred at rt for 20 h. The reaction was quenched with sat. $NaHCO_3$ and the resulting mixtue was extracted 3 x with CH_2Cl_2. The combined organics were concentrated and purified by chromatography (1:1 hex:EtOAc) to yield a yellow solid (220 mg, 1.03 mmol, 88% yield) mp: 130-132 ˚C. IR (film): 3249, 1664, 1590, 1532, 1466 cm^{-1}. ^1H NMR (400 MHz, C_6D_6): δ 8.71-8.64 (m, 1H), 8.53 (d, J = 4.5 Hz, 1H), 7.80

(d, J = 8.8 Hz, 2H), 7.62-7.57 (m, 1H), 7.57-7.51 (m, 3H), 7.11-7.05 (m, 1H), 1.99 (s, 3H). $^{13}C\{^{1}H\}$ NMR (100 MHz, $CDCl_3$): δ 169.2, 156.9, 149.4, 139.2, 136.9, 134.9, 127.5, 121.9, 120.3, 120.0, 24.4. HRMS (ESI+) Calcd for $C_{13}H_{13}N_2O$ $[MH]^+$ 213.1028; Found 213.1023.

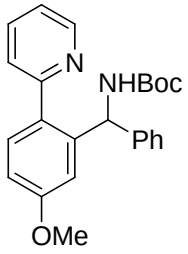
III. General Procedure for C–H Activation Experiments Monitored by NMR. In a nitrogen-filled inert atmosphere box, in a vial was combined $[Cp^*RhCl_2]_2$, $AgSbF_6$, 2-phenyl-pyridine, *tert*-butyl benzylidenecarbamate, 2,6-dimethoxytoluene and solvent (0.5 mL). The reaction mixture was transferred to thin-walled NMR tube. The tube was fitted with a Cajon adapter, frozen with liquid nitrogen, and flame-sealed under vacuum. The tube was then placed in an oil bath set to the desired temperature. Periodically, the tube was removed from the bath, cooled to room temperature, and analyzed by ^{1}H-NMR spectroscopy to monitor the progress of the reaction based on integration relative to 2,6-dimethoxytoluene as an internal standard. All optimization reactions were carried out via this procedure by varying temperature, ligand, and catalyst loading

IV. Rh(III) Catalyzed Hydroarylation of Imines

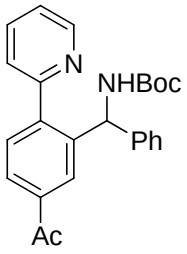


N-***tert*-Butyl (phenyl(2-(pyridin-2-yl)phenyl)methyl)carbamate (3a)**. To a vial in a glovebox was combined $[Cp^*RhCl_2]_2$ (15.5 mg, 0.0250 mmol, 0.1 equiv), $AgSbF_6$ (34.3 mg, 0.100 mmol, 0.4 equiv), 2-phenylpyridine (77.5 mg, 0.500 mmol, 2 equiv), *tert*-butyl benzylidene carbamate (51 mg, 0.25 mmol, 1 equiv), and CH_2Cl_2 (2.5 mL). The reaction mixture was transferred to a 5 mL Schlenk tube, removed from the glovebox, and heated at 75 ˚C for 20 h. Reaction mixture was then cooled to rt, concentrated *in vacuo*, and purified by

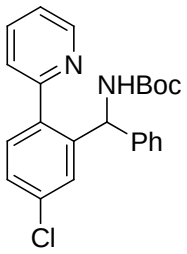
chromatography (5:1 hex:EtOAc) to yield a white solid (73.8 mg, 0.205 mmol, 82% yield) mp: 146-148 ˚C. IR (film): 3253, 1690 cm^{-1}. ^1H NMR (400 MHz, $CDCl_3$): δ 8.27 (m, 1H), 7.49-7.36 (m, 2H), 7.36-7.19 (m, 3H), 7.07-6.99 (m, 1H), 6.99-6.85 (m, 4H), 6.85-6.75 (m, 2H), 6.72-6.59 (m, 1H), 6.13 (d, J = 8.5 Hz, 1H), 1.36 (s, 9H). $^{13}C\{^1H\}$ NMR (100 MHz, $CDCl_3$): δ 160.0, 155.4, 148.4, 142.2, 141.2, 140.6, 136.5, 130.9, 130.1, 128.9, 127.8, 127.7, 126.4, 126.3, 124.3, 121.9, 79.3, 57.2, 28.6. HRMS (ESI+) Calcd for $C_{23}H_{25}N_2O_2$ $[MH]^+$ 361.1916; Found 361.1909.



***N-tert*-Butyl ((5-methoxy-2-(pyridin-2-yl)phenyl)(phenyl)methyl)carbamate (3b).** To a vial in a glovebox was combined $[Cp^*RhCl_2]_2$ (9.3 mg, 0.015 mmol, 0.1 equiv), $AgSbF_6$ (20.6 mg, 0.060 mmol, 0.4 equiv), 2-(4-methoxyphenyl)pyridine (55.5 mg, 0.300 mmol, 2 equiv), *tert*-butylbenzylidenecarbamate (31 mg, 0.15 mmol, 1 equiv), and CH_2Cl_2 (1.5 mL). The reaction mixture was transferred to a 5 mL Schlenk tube, removed from the glovebox, and heated at 75 ˚C for 20 h. Reaction mixture was then cooled to rt, concentrated *in vacuo*, and purified by chromatography (5:1 hex:EtOAc) to yield a white solid (37 mg, 0.095 mmol, 63% yield) mp: 135-137 ˚C. IR (film): 3335, 1703 cm^{-1}. ^1H NMR (400 MHz, $CDCl_3$): δ 8.46 (d, J = 4.3 Hz, 1H), 7.43-7.36 (m, 1H), 7.26-7.15 (m, 1H), 7.04-6.97 (m, 2 H), 6.97-6.87 (m, 4H), 6.87-6.80 (m, 3H), 6.80-6.73 (m, 1H), 6.11 (d, J = 8.6 Hz, 1H), 3.78 (s, 3H), 1.39 (s, 9H). $^{13}C\{^1H\}$ NMR (100 MHz, $CDCl_3$): δ 158.8, 154.3, 147.2, 141.5, 140.8, 135.3, 132.0, 131.2, 127.2, 126.6, 125.1, 123.1, 120.3, 114.7, 113.3, 111.5, 78.2, 56.3, 54.4, 27.5. HRMS (ESI+) Calcd for $C_{24}H_{27}N_2O_3$ $[MH]^+$ 391.2022; Found 391.2003.

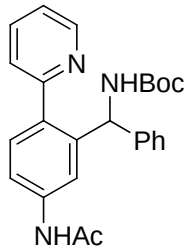


***N-tert*-Butyl ((5-acetyl-2-(pyridin-2-yl)phenyl)(phenyl)methyl)carbamate (3c).** To a vial in a glovebox was combined $[Cp^*RhCl_2]_2$ (15.5 mg, 0.0250 mmol, 0.1 equiv), $AgSbF_6$ (34.3 mg, 0.100 mmol, 0.4 equiv), 1-(4-(pyridin-2-yl)phenyl)ethanone (106 mg, 0.500 mmol, 2 equiv), *tert*-butyl benzylidenecarbamate (51 mg, 0.25 mmol, 1 equiv), and CH_2Cl_2 (2.5 mL). The reaction mixture was transferred to a 5 mL Schlenk tube, removed from the glovebox, and heated at 75 ˚C for 20h. Reaction mixture was then cooled to rt, concentrated *in vacuo*, and purified by chromatography (2:1 hex:EtOAc) to yield a white solid (87 mg, 0.22 mmol, 87% yield) mp: 47-49 ˚C. IR (film): 3350, 1690, 1681 cm^{-1}. ^1H NMR (400 MHz, $CDCl_3$): δ 8.50 (d, J = 4.3 Hz, 1H), 8.08-8.02 (m, 1H), 7.88 (dd, J = 7.8, 1.7 Hz, 1H), 7.44 (t, J = 7.4 Hz, 1H), 7.34 (d, J = 9.0 Hz, 1H), 7.07 (dd, J = 6.9, 5.1 Hz, 1H), 6.99 (d, J = 8.1Hz, 1H), 6.97-6.89 (m, 3H), 6. 75 (m, 2H), 6.55-6.49 (m, 1H), 6.23 (d, J = 8.7Hz, 1H), 2.57 (s, 3H), 1.38 (s, 9H). $^{13}C\{^1H\}$ NMR (100 MHz, $CDCl_3$): δ 197.7, 158.7, 155.1, 148.5, 144.7, 141.6, 141.4, 137.0, 136.5, 131.2, 129.2, 127.9, 127.4, 126.5, 126.3, 124.1, 122.3, 79.5, 56.8, 28.4, 26.8. HRMS (ESI+) Calcd for $C_{25}H_{27}N_2O_3$ $[MH]^+$ 403.2022; Found 403.2006.



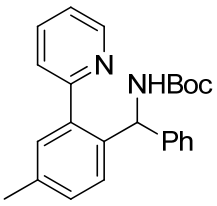
***N-tert*-Butyl ((5-chloro-2-(pyridin-2-yl)phenyl)(phenyl)methyl)carbamate (3d).** To a vial in a glovebox was combined $[Cp^*RhCl_2]_2$ (9.3 mg, 0.015 mmol, 0.1 equiv), $AgSbF_6$ (20.6

mg, 0.060 mmol, 0.4 equiv), 2-(4-chlorophenyl)pyridine (57 mg, 0.30 mmol, 2 equiv), *tert*-butyl benzylidenecarbamate (31 mg, 0.15 mmol, 1 equiv), and CH_2Cl_2 (1.5 mL). The reaction mixture was transferred to a 5 mL Schlenk tube, removed from the glovebox, and heated at 75 ˚C for 20 h. Reaction mixture was then cooled to rt, concentrated *in vacuo*, and purified by chromatography (5:1 hex:EtOAc) to yield a white solid (30 mg, 0.075 mmol, 50% yield) mp: 145-147 ˚C. IR (film): 3347, 1703, 1500, 1020 cm^{-1}. ^1H NMR (400 MHz, $CDCl_3$): δ 8.49 (d, *J* = 4.7 Hz, 1H), 7.48-7.41 (m, 2H), 7.28 (dd, *J* = 8.3, 2.3 Hz, 1H), 7.21-7.17 (m, 1H), 7.07-7.03 (m, 1H), 7.00-6.93 (m, 4H), 6.85-6.78 (m, 2H), 6.49-6.38 (m, 1H), 6.14 (d, *J* = 8.1 Hz, 1H), 1.38 (s, 9H). $^{13}C\{^1H\}$ NMR (100 MHz, $CDCl_3$): δ 159.0, 155.5, 148.9, 143.1, 141.6, 139.1, 136.8, 134.9, 132.4, 129.8, 128.2, 127.9, 126.8, 126.6, 124.5, 122.3, 79.8, 56.9, 28.8. HRMS (ESI+) Calcd for $C_{23}H_{24}ClN_2O_2$ $[MH]^+$ 395.1526; Found 395.1507.

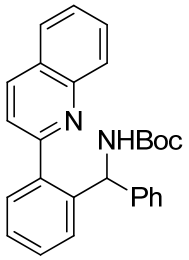


***N*-*tert*-Butyl ((5-acetamido-2-(pyridin-2-yl)phenyl)(phenyl)methyl)carbamate (3e).** To a vial in a glovebox was combined $[Cp^*RhCl_2]_2$ (15.5 mg, 0.0250 mmol, 0.1 equiv), $AgSbF_6$ (34.3 mg, 0.100 mmol, 0.4 equiv), *N*-(4-(pyridin-2-yl)phenyl)acetamide (106 mg, 0.500 mmol, 2 equiv), *tert*-butyl benzylidenecarbamate (51 mg, 0.25 mmol, 1 equiv), and CH_2Cl_2 (2.5 mL). The reaction mixture was transferred to a 5 mL Schlenk tube, removed from the glovebox, and heated at 75 ˚C for 20 h. Reaction mixture was then cooled to rt, concentrated *in vacuo*, and purified by chromatography (1:2 hex:EtOAc) to yield a white solid (70 mg, 0.17 mmol, 67% yield) mp: 127-130 ˚C. IR (film): 3281, 1690, 1671 cm^{-1}. ^1H NMR (400 MHz, $CDCl_3$): δ 8.45

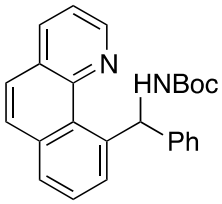
(d, J = 4.5 Hz, 1H), 8.29-8.25 (m, 1H), 7.71-7.74 (m, 1H), 7.44-7.36 (m, 1H), 7.31-7.25 (m, 1H), 7.19-7.15 (m, 1H), 7.04-6.98 (m, 1H), 6.98-6.83 (m, 5H), 6.83-6.74 (m, 2H), 6.08 (d, J = 8.5 Hz, 1H). $^{13}C\{^{1}H\}$ NMR (100 MHz, $CDCl_3$): δ 169.0, 159.3, 155.5, 148.2, 141.6, 141.3, 138.6, 136.5, 135.8, 131.7, 127.7, 126.3, 126.2, 124.2, 121.6, 120.8, 119.2, 79.4, 57.0, 28.5, 24.4 . HRMS (ESI+) Calcd for $C_{25}H_{28}N_3O_3$ $[MH]^+$ 418.2131; Found 418.2125.



***N-tert*-Butyl ((4-methyl-2-(pyridin-2-yl)phenyl)(phenyl)methyl)carbamate (3f).** To a vial in a glovebox was combined $[Cp^*RhCl_2]_2$ (9.3 mg, 0.015 mmol, 0.1 equiv), $AgSbF_6$ (20.6 mg, 0.060 mmol, 0.4 equiv), 2-(*m*-tolyl)pyridine (50.7 mg, 0.300 mmol, 2 equiv), *tert*-butyl benzylidenecarbamate (31 mg, 0.15 mmol, 1 equiv), and CH_2Cl_2 (1.5 mL). The reaction mixture was transferred to a 5 mL Schlenk tube, removed from the glovebox, and heated at 75 ˚C for 20 h. Reaction mixture was then cooled to rt, concentrated *in vacuo*, and purified by chromatography (5:1 hex:EtOAc) to yield a white solid (50 mg, 0.13 mmol, 89% yield) mp: 53-56 ˚C. IR (film): 3312, 1697 cm^{-1}. ^{1}H NMR (400 MHz, $CDCl_3$): δ 8.52-8.42 (m, 1H), 7.47-7.35 (m, 1H), 7.35-7.22 (m, 1H), 7.15-7.10 (m, 1H), 7.10-6.76 (m, 8H), 6.76-6.54 (m, 1H), 6.07 (d, J = 8.3 Hz, 1H), 2.29 (s, 3H), 1.37 (s, 9H) . $^{13}C\{^{1}H\}$ NMR (100 MHz, $CDCl_3$): δ 160.3, 155.6, 148.8, 142.7, 140.7, 138.5, 137.7, 136.7, 132.0, 130.3, 129.8, 128.1, 126.6, 126.5, 124.6, 122.1, 79.5, 57.2, 28.9, 21.4 . HRMS (ESI+) Calcd for $C_{24}H_{27}N_2O_2$ $[MH]^+$ 375.2073; Found 375.2066.

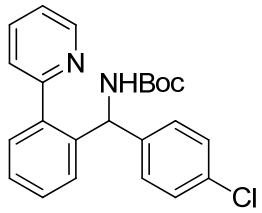


***N-tert*-Butyl (phenyl(2-(quinolin-2-yl)phenyl)methyl)carbamate (3g).** To a vial in a glovebox was combined $[Cp^*RhCl_2]_2$ (9.3 mg, 0.015 mmol, 0.1 equiv), $AgSbF_6$ (20.6 mg, 0.060 mmol, 0.4 equiv), 2-phenylquinoline (61.5 mg, 0.300 mmol, 2 equiv), *tert*-butyl benzylidene-carbamate (31 mg, 0.15 mmol, 1 equiv), and CH_2Cl_2 (1.5 mL). The reaction mixture was transferred to a 5 mL Schlenk tube, removed from the glovebox, and heated at 75 ˚C for 20 h. Reaction mixture was then cooled to rt, concentrated *in vacuo*, and purified by chromatography (5:1 hex:EtOAc) to yield a white solid (46 mg, 0.11 mmol, 75% yield) mp: 61-63 ˚C. IR (film): 3286, 1704, 1487 cm^{-1}. ^1H NMR (400 MHz, $CDCl_3$): δ 8.03 (d, J = 8.8 Hz, 1H), 7.86-7.81 (m, 1H), 7.71-7.63 (m, 3H), 7.58-7.53 (m, 1H), 7.49-7.44 (m, 1H), 7.42-7.36 (m, 1H), 7.36-7.30 (m, 2H), 7.02-6.96 (m, 1H), 6.95-6.80 (m, 1H) 6.80-6.62 (m, 4H), 6.1 (d, J = 8.8 Hz, 1H), 1.41 (s, 9H). $^{13}C\{^1H\}$ NMR (100 MHz, $CDCl_3$): δ 160.5, 155.8, 147.0, 142.5, 142.2, 141.1, 136.9, 131.5, 131.1, 130.3, 129.3, 128.1, 127.9, 127.8, 127.0, 126.9, 126.3, 126.2, 126.1, 122.5, 79.4, 58.4, 28.9. HRMS (ESI+) Calcd for $C_{27}H_{27}N_2O_2$ $[MH]^+$ 411.2073; Found 411.2061.



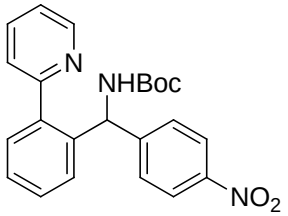
***N-tert*-Butyl (benzo[*h*]quinolin-10-yl(phenyl)methyl)carbamate (3h).** To a vial in a glovebox was combined $[Cp^*RhCl_2]_2$ (6.4 mg, 0.010 mmol, 0.1 equiv), $AgSbF_6$ (15.9 mg, 0.0463 mmol, 0.4 equiv), benzo[*h*]quinolin (37.9 mg, 0.189 mmol, 1.9 equiv), *N-tert*-butyl-

benzylidenecarbamate (21.7 mg, 0.106 mmol, 1 equiv), and CH_2Cl_2 (1.4 mL). The slurry was transferred to a reaction tube, which was sealed and then placed into an oil bath preheated to 75 ˚C for 20 h. The reaction mixture was then cooled to rt, concentrated *in vacuo*, and purified by chromatography (85:15 hex:EtOAc) to yield a white solid (28.2 mg, 0.0733 mmol, 70% yield). mp: 140 ˚C. IR (neat): 1707 (s), 1475 (m), 1454 (m), 1430 (m), 1362 (m), 1161 (s), 1119 (w), 1043 (w), 1019 (m), 839 (s), 729 (s), 696 (m) cm^{-1}. ^1H NMR (500 MHz, 328 K, $CDCl_3$) δ 8.73 (s, 1H), 8.11 (dd, *J* = 8.0, 1.7 Hz, 1H), 7.98 (d, *J* = 7.8 Hz, 1H), 7.96 – 7.92 (m, 1H), 7.89 (d, *J* = 8.7 Hz, 1H), 7.76 (t, *J* = 7.6 Hz, 1H), 7.69 (d, *J* = 8.7 Hz, 1H), 7.39 (dd, *J* = 7.7, 4.3 Hz, 1H), 7.24 (s, 2H), 7.15 (s, 2H), 7.10 (s, 1H), 1.52 (s, 9H). $^{13}C\{^1H\}$ NMR (126 MHz, 328 K, $CDCl_3$) δ 155.92, 146.90, 146.84, 146.63, 144.07, 136.01, 135.54, 129.38, 129.00, 128.39 – 127.75 (broad signal), 127.67, 127.61, 125.66, 120.89, 108.00 – 117.00, 79.01, 28.53. HRMS (ESI+) Calcd for $C_{25}H_{25}N_2O_2$ $[MH]^+$ 385.1911; Found 385.1908.



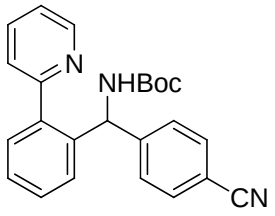
***N-tert*-Butyl ((4-chlorophenyl)(2-(pyridin-2-yl)phenyl)methyl)carbamate (3i).** To a vial in a glovebox was combined $[Cp^*RhCl_2]_2$ (3.3 mg, 0.0053 mmol, 0.1 equiv), $AgSbF_6$ (6.8 mg, 0.020 mmol, 0.4 equiv), 2-phenylpyridine (15.4 mg, 0.0992 mmol, 2 equiv), 2,4-dimethoxytoluene (15.4 mg, 0.101 mmol, 2 equiv) *N-tert*-butyl-4-chlorobenzylidenecarbamate (12.8 mg, 0.0530 mmol, 1 equiv), and CH_2Cl_2 (0.5 mL). The slurry was transferred to an NMR tube equipped with a sealed capillary containing C_6D_6. The tube was closed with a Cajon adapter, removed from the glovebox, flame sealed and then placed into an oil bath preheated to 75 ˚C for 20 h. The reaction mixture was then cooled to rt, concentrated *in vacuo*, and purified by

chromatography (85:15 hexanes:EtOAc) to yield a white solid (16.0 mg, 0.0410 mmol, 77% yield) mp: 60 ˚C. IR (neat): 1699 (s), 1588 (w), 1488 (s), 1365 (m), 1247 (m), 1163 (s), 1090 (m), 1043 (w), 1014 (s), 752 (s) cm^{-1}. ^{1}H NMR (600 MHz, $CDCl_3$): δ 8.56 (s, 1H), 7.51 (m, 2H), 7.44 (td, 1H, *J* = 7.5, 1.5 Hz), 7.41 (td, 1H, *J* = 7.2, 1.5 Hz), 7.33 (m, 1H), 7.16 (d, *J* = 5.3 Hz, 1H), 7.06 (d, *J* = 7.5 Hz, 1H), 6.99 (d, *J* = 7.7 Hz, 2H), 6.95 (d, *J* = 8.1 Hz, 1H), 6.86 (d, *J* = 7.6 Hz, 2H), 6.15 (d, *J* = 7.6 Hz, 1H, C*H*NBoc), 1.46 (s, 9H, C*Me*$_3$). $^{13}C\{^{1}H\}$ NMR (151 MHz, $CDCl_3$) δ 159.88, 155.39, 148.34, 140.92, 140.73, 140.46, 136.72, 131.92, 131.12, 130.39, 128.99, 128.00, 127.82, 127.41, 124.31, 122.05, 79.49 (Ar_2*C*HN), 57.04 (*C*Me_3), 28.61 (C*Me*$_3$). HRMS (ESI+) Calcd for $C_{23}H_{24}N_2O_2Cl$ $[MH]^+$ 395.1521; Found 395.1530.

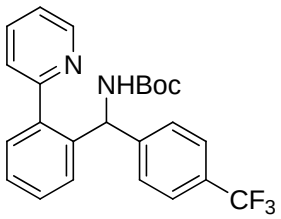


***N-tert*-Butyl ((4-nitrophenyl)(2-(pyridin-2-yl)phenyl)methyl)carbamate (3j).** To a vial in a glovebox was combined $[Cp^*RhCl_2]_2$ (15.5 mg, 0.0250 mmol, 0.1 equiv), $AgSbF_6$ (34.3 mg, 0.100 mmol, 0.4 equiv), 2-phenylpyridine (77.5 mg, 0.500 mmol, 2 equiv), *tert*-butyl 4-nitrobenzylidenecarbamate (62.5 mg, 0.250 mmol, 1 equiv), and CH_2Cl_2 (2.5 mL). The reaction mixture was transferred to a 5 mL Schlenk tube, removed from the glovebox, and heated at 75 ˚C for 20 h. Reaction mixture was then cooled to rt, concentrated *in vacuo*, and purified by chromatography (5:1 hex:EtOAc) to yield a white solid (78 mg, 0.19 mmol, 77% yield) mp: 51-53 ˚C. IR (film): 3306, 1701, 1514 1342 cm^{-1}. ^{1}H NMR (400 MHz, $CDCl_3$): δ 8.55 (d, *J* = 4.4 Hz, 1H), 7.85 (d, *J* = 8.1 Hz, 2H), 7.60-7.53 (m, 1H), 7.53-7.39 (m, 3H), 7.39-7.29 (m, 2H), 7.16-7.05 (m, 3H), 7.05-6.98 (m, 1H), 6.25 (d, *J* = 9.1 Hz, 1H), 1.48 (s, 9H). $^{13}C\{^{1}H\}$ NMR (100 MHz, $CDCl_3$): δ 159.5, 155.4, 150.2, 148.1, 146.1, 140.0, 139.8, 136.9, 131.3, 131.0, 121.1,

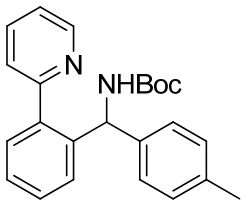
128.3, 126.7, 124.2, 122.6, 122.1, 79.7, 57.7, 28.4. HRMS (ESI+) Calcd for $C_{23}H_{24}N_3O_4$ $[MH]^+$ 406.1762; Found 406.1768.



***N-tert*-Butyl ((4-cyanophenyl)(2-(pyridin-2-yl)phenyl)methyl)carbamate (3k).** To a vial in a glovebox was combined $[Cp^*RhCl_2]_2$ (25.5 mg, 0.0413 mmol, 0.1 equiv), $AgSbF_6$ (56.7 mg, 0.165 mmol, 0.4 equiv), 2-phenylpyridine (128 mg, 0.826 mmol, 2 equiv), *tert*-butyl 4-cyanobenzylidenecarbamate (95.0 mg, 0.413 mmol, 1 equiv), and CH_2Cl_2 (4.1 mL). The reaction mixture was transferred to a 5 mL Schlenk tube, removed from the glovebox, and heated at 75 ˚C for 20 h. Reaction mixture was then cooled to rt, concentrated *in vacuo*, and purified by chromatography (3:1 hex:EtOAc) to yield a white solid (85 mg, 0.21 mmol, 50% yield) mp: 55-57 ˚C. IR (film): 3299, 2226, 1695, 1472 cm^{-1}. ^1H NMR (400 MHz, $CDCl_3$): δ 8.53 (d, J = 4.4 Hz, 1H), 7.56-7.47 (m, 2H), 7.47-7.39 (m, 2H), 7.36-7.31 (m, 1H), 7.31-7.24 (m, 3H), 7.14-7.09 (m, 1H), 7.06-7.01 (m, 2H), 7.01-6.96 (m, 1H), 6.21 (d, J = 8.2 Hz), 1.47 (s, 9H). $^{13}C\{^1H\}$ NMR (100 MHz, $CDCl_3$): δ 159.5, 155.3, 148.1, 148.0, 140.1, 140.0, 136.7, 131.3, 131.2, 131.0, 129.1, 128.3, 126.6, 124.1, 122.0, 118.9, 109.7, 79.6, 57.7, 28.5 HRMS (ESI+) Calcd for $C_{24}H_{24}N_3O_2$ $[MH]^+$ 386.1864; Found 386.1866.

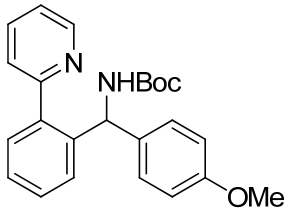


***N-tert*-Butyl ((2-(pyridin-2-yl)phenyl)(4-(trifluoromethyl)phenyl)methyl)carbamate (3l).** To a vial in a glovebox was combined $[Cp^*RhCl_2]_2$ (13.6 mg, 0.022 mmol, 0.1 equiv), $AgSbF_6$ (30.2 mg, 0.088 mmol, 0.4 equiv), 2-phenylpyridine (68 mg, 0.44 mmol, 2 equiv), *tert*-butyl 4-(trifluoromethyl)benzylidenecarbamate (60 mg, 0.22 mmol, 1 equiv), and CH_2Cl_2 (2.2 mL). The reaction mixture was transferred to a 5 mL Schlenk tube, removed from the glovebox, and heated at 75 ˚C for 20 h. Reaction mixture was then cooled to rt, concentrated *in vacuo*, and purified by chromatography (5:1 hex:EtOAc) to yield a white solid (89 mg, 0.21 mmol, 95% yield) mp: 52-54 ˚C. IR (film): 3260, 1693, 1325 cm^{-1}. ^1H NMR (400 MHz, $CDCl_3$): δ 8.55 (d, J = 4.9 Hz, 1H), 7.56-7.37 (m, 4H), 7.35-7.29 (m, 1H), 7.26-7.22 (d, 2H), 7.14-7.08 (m, 1H), 7.08-6.94 (m, 4H), 6.3 (d, J = 9.4 Hz, 1H), 9.25 (s, 9H) $^{13}C\{^1H\}$ NMR (100 MHz, $CDCl_3$): δ 159.7, 155.3, 148.1, 146.4, 140.4, 140.3, 136.6, 131.0, 130.7, 128.9, 128.4, 128.3 (q, J_{C-F} = 31.1 Hz) 126.3, 124.5 (q, J_{C-F} = 3.1 Hz), 124.2, 124.1 (q, J_{C-F} = 270.2 Hz) 121.9, 79.5, 57.4, 28.5. ^{19}F NMR (375 Mhz, $CDCl_3$): δ −60.2 HRMS (ESI+) Calcd for $C_{24}H_{24}F_3N_2O_2$ $[MH]^+$ 429.1785; Found 429.1790.



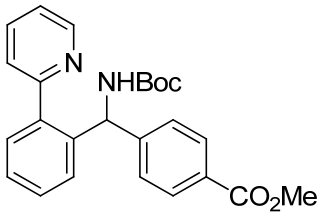
***N-tert*-Butyl ((2-(pyridin-2-yl)phenyl)(p-tolyl)methyl)carbamate (3m).** To a vial in a glovebox was combined $[Cp^*RhCl_2]_2$ (15.5 mg, 0.0250 mmol, 0.1 equiv), $AgSbF_6$ (34.3 mg, 0.100 mmol, 0.4 equiv), 2-phenylpyridine (77.5 mg, 0.499 mmol, 2 equiv), *tert*-butyl 4-methyl-benzylidenecarbamate (54.8 mg, 0.250 mmol, 1 equiv), and CH_2Cl_2 (2.5 mL). The reaction mixture was transferred to a 5 mL Schlenk tube, removed from the glovebox, and heated at 75 ˚C for 20 h. Reaction mixture was then cooled to rt, concentrated *in vacuo*, and purified by

chromatography (5:1 hex:EtOAc) to yield a white solid (65 mg, 0.18 mmol, 70% yield) mp: 49-51 ˚C. IR (film): 3299, 1696, 1472 cm^{-1}. ^1H NMR (400 MHz, $CDCl_3$): δ 8.58 (d, *J* = 4.5 Hz, 1H), 7.58-7.50 (m, 1H), 7.50-7.30 (m, 4 H), 7.18-7.09 (m, 2H), 6.92-6.72 (m, 4 H), 6.53-6.34 (m, 1H), 6.16 (d, *J* = 8.3 Hz, 1H), 2.19 (s, 3H), 1.44 (s, 9H). $^{13}C\{^1H\}$ NMR (100 MHz, $CDCl_3$): δ 159.7, 155.1, 148.4, 140.9, 140.3, 139.1, 136.2, 135.7, 130.7, 129.4, 128.7, 128.4, 127.5, 126.2, 124.2, 121.7, 79.1, 56.5, 28.4, 20.9. HRMS (ESI+) Calcd for $C_{23}H_{27}N_2O_2$ $[MH]^+$ 375.2068; Found 375.2076



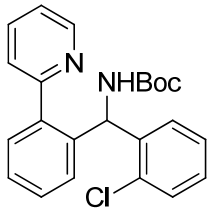
***N-tert*-Butyl((4-methoxyphenyl)(2-(pyridin-2-yl)phenyl)methyl)carbamate (3n).** To a vial in a glovebox was combined $[Cp^*RhCl_2]_2$ (3.0 mg, 0.005 mmol, 0.1 equiv), $AgSbF_6$ (3.7 mg, 0.011 mmol, 0.2 equiv), 2-phenylpyridine (15.5 mg, 0.100 mmol, 2 equiv), 2,4-dimethoxytoluene (15.4 mg, 0.101 mmol, 2 equiv) a solution of *N-tert*-butyl-4-methoxybenzylidenecarbamate in CH_2Cl_2 (0.13 mL, 0.42 M, 0.055 mmol, 1 equiv), and CH_2Cl_2 (0.5 mL). The slurry was transferred to an NMR tube equipped with a sealed capillary containing C_6D_6. The tube was closed with a Cajon adapter, removed from the glovebox, flame sealed and then placed into an oil bath preheated to 75 ˚C for 20 h. The reaction mixture was then cooled to rt, concentrated *in vacuo*, and purified by chromatography (85:15 hexanes:EtOAc) to yield a white solid (15.1 mg, 0.039 mmol, 70% yield). mp: 50 ˚C. IR (neat): 2160 (m, broad), 2027 (m, broad), 1698 (s), 1586 (w), 1508 (m), 1468 (w), 1364 (m), 1245 (s), 1161 (s), 1025 (m), 752 (s) cm^{-1}. ^1H NMR (600 MHz, $CDCl_3$) δ 8.61 (s, 1H), 7.57 (t, *J* = 6.9 Hz, 1H), 7.51 (d, *J* = 6.5 Hz, 1H), 7.44 (t, *J* = 7.8 Hz, 1H), 7.40 (t, *J* = 7.8 Hz, 1H), 7.35 (s, 1H), 7.16 (s, 1H), 7.13 (m,

1H), 6.85 (m, 2H), 6.61 (m, 2H), 6.53 (m, 1H), 6.18 (d, *J* = 8.0 Hz, 1H, C*H*NBoc), 3.72 (s, 3H, O*Me*), 1.47 and 1.48 (rotamers of C*Me*$_3$, 9H, coalescence at 323K). $^{13}C\{^1H\}$ NMR (151 MHz, $CDCl_3$) δ 159.83, 157.94, 155.13, 148.41, 141.04, 140.42, 136.34, 134.36, 130.76, 129.29, 128.66, 127.47, 124.17, 121.75, 113.95, 113.22, 79.27 (Ar_2CHN), 56.37 (*C*Me$_3$), 55.21 (O*Me*), 28.47 and 28.24 (rotamers of C*Me*$_3$, coalescence at 323 K). HRMS (ESI+) Calcd for $C_{24}H_{27}N_2O_3$ $[MH]^+$ 391.2016; Found 391.2030.

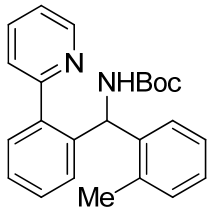


***N-tert*-Butyl ((4-methylbenzoato)(2-(pyridin-2-yl)phenyl)methyl)carbamate (3o).** To a vial in a glovebox was combined $[Cp^*RhCl_2]_2$ (3.6 mg, 0.006 mmol, 0.1 equiv), $AgSbF_6$ (8.5 mg, 0.025 mmol, 0.5 equiv), 2-phenylpyridine (15.4 mg, 0.099 mmol, 2 equiv), 2,4-dimethoxytoluene (15.4 mg, 0.101 mmol, 2 equiv) *N-tert*-butyl-4-methoxycarbonylbenzylidene-carbamate (13.2 mg, 0.050 mmol, 1 equiv), and CH_2Cl_2 (0.5 mL). The slurry was transferred to an NMR tube equipped with a sealed capillary containing C_6D_6. The tube was closed with a Cajon adapter, removed from the glovebox, flame sealed and then placed into an oil bath preheated to 75 ˚C for 20 h. The reaction mixture was then cooled to rt, concentrated *in vacuo*, and purified by chromatography (85:15 hexanes:EtOAc) to yield a white solid (14.6 mg, 0.035 mmol, 70% yield). mp: 71 ˚C. IR (neat): 1705 (s), 1610 (w), 1470 (m), 1365 (w), 1276 (s), 1161 (s), 1103 (m), 1043 (w), 1017 (m), 753 (s) cm^{-1}. ^1H NMR (500 MHz, $CDCl_3$) δ 8.60 (d, *J* = 4.2 Hz, 1H), 7.72 (d, *J* = 7.8 Hz, 2H), 7.59 (d, *J* = 6.9 Hz, 1H), 7.54 – 7.43 (m, 3H), 7.38 (d, *J* = 7.1 Hz, 1H), 7.22 – 7.11 (m, 2H), 7.03 (m, 2H), 6.27 (d, *J* = 9.0 Hz, 1H, C*H*NBoc), 3.88 (s, 3H, CO_2*Me*), 1.51 (s, 9H, C*Me*$_3$). $^{13}C\{^1H\}$ NMR (126 MHz, $CDCl_3$) δ 167.01, 159.73, 155.40,

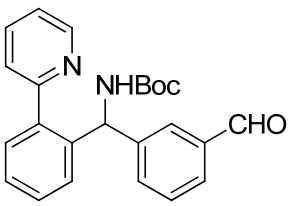
148.17, 147.68, 140.57, 140.51, 140.33, 136.67, 131.14, 131.09, 130.80, 128.95, 128.02, 125.99, 124.21, 121.98, 79.65 (Ar_2CHN), 57.62 (CMe_3), 51.98 (O*Me*), 28.51 (CMe_3). HRMS (ESI+) Calcd for $C_{25}H_{27}N_2O_4$ $[MH]^+$ 419.1965; Found 419.1969.



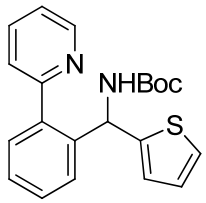
***N-tert*-Butyl((2-chlorophenyl)(2-(pyridin-2-yl)phenyl)methyl)carbamate (3p).** To a vial in a glovebox was combined $[Cp^*RhCl_2]_2$ (3.0 mg, 0.005 mmol, 0.1 equiv), $AgSbF_6$ (4.8 mg, 0.014 mmol, 0.2 equiv), 2-phenylpyridine (16.1 mg, 0.104 mmol, 2 equiv), 2,4-dimethoxy-toluene (15.8 mg, 0.104 mmol, 2 equiv) a solution of *N-tert*-butyl-2-chlorobenzylidenecarbamate in CH_2Cl_2 (0.25 mL, 0.20 M, 0.05 mmol, 1 equiv), and CH_2Cl_2 (0.45 mL). The slurry was transferred to an NMR tube equipped with a sealed capillary containing C_6D_6. The tube was closed with a Cajon adapter, removed from the glovebox, flame sealed and then placed into an oil bath preheated to 75 ˚C for 20 h. The reaction mixture was then cooled to rt, concentrated *in vacuo*, and purified by chromatography (85:15 hexanes:EtOAc) to yield a white solid (17.7 mg, 0.047 mmol, 76% yield). mp: 82 ˚C. IR (neat): 1697 (s), 1468 (m), 1364 (m), 1255 (m), 1160 (s), 1017 (m), 749 (s) cm^{-1}. ^1H NMR (500 MHz, $CDCl_3$) δ 8.67 (s, 1H), 7.67 (t, *J* = 7.5 Hz, 1H), 7.39 (m, 3H), 7.35 - 7.30 (m, 2H), 7.26 - 7.23 (m, 1H), 7.21 (d, *J* = 5.5 Hz, 1H), 7.17 (s, 1H), 7.10 (m, 2H), 6.38 (d, *J* = 7.6 Hz, 1H), 5.85 (s, 1H), 1.47 and 1.48 (rotamers of CMe_3, 9H). $^{13}C\{^1H\}$ NMR (126 MHz, $CDCl_3$) δ 154.56, 148.94, 140.77, 139.44, 138.60, 136.46, 133.14, 130.45, 129.62, 129.15, 128.22, 128.53, 128.12, 127.87, 127.74, 126.26, 123.79, 121.98, 79.50 (Ar_2CHN), 54.65 (CMe_3), 28.41 and 28.33 (rotamers of CMe_3). HRMS (ESI+) Calcd for $C_{23}H_{23}N_2O_2Cl$ $[MH]^+$: 395.1521; Found 395.1527.



***N-tert*-Butyl((2-methylphenyl)(2-(pyridin-2-yl)phenyl)methyl)carbamate (3q).** To a vial in a glovebox was combined $[Cp^*RhCl_2]_2$ (3.1 mg, 0.005 mmol, 0.1 equiv), $AgSbF_6$ (3.8 mg, 0.011 mmol, 0.2 equiv), 2-phenylpyridine (16.7 mg, 0.108 mmol, 2 equiv), 2,4-dimethoxy-toluene (15.4 mg, 0.101 mmol, 2 equiv) a solution of *N-tert*-butyl-2-methylbenzylidene-carbamate in CH_2Cl_2 (0.17 mL, 0.30 M, 0.051 mmol, 1 equiv), and CH_2Cl_2 (0.5 mL). The slurry was transferred to an NMR tube equipped with a sealed capillary containing C_6D_6. The tube was closed with a Cajon adapter, removed from the glovebox, flame sealed and then placed into an oil bath preheated to 75 ˚C for 20 h. The reaction mixture was then cooled to rt, concentrated *in vacuo*, and purified by chromatography (85:15 hexanes:EtOAc) to yield a white solid (17.7 mg, 0.047 mmol, 92% yield). mp: 55 ˚C. IR (neat): 1696 (s), 1485 (m), 1426 (w), 1364 (m), 1250 (m), 1162 (s), 1017 (m), 751 (s) cm^{-1}. ^1H NMR (400 MHz, $CDCl_3$) δ 8.67 (d, J = 4.1 Hz, 1H), 7.64 (t, J = 7.2 Hz, 1H), 7.47 – 7.31 (m, 4H), 7.21 (m, 2H), 7.04 (m, 4H), 6.33 (d, J = 7.8 Hz, 1H, C*H*NHBoc), 5.55 (d, J = 7.4 Hz, 1H, N*H*Boc), 2.01 (s, 3H, Ar*Me*), 1.54 – 1.37 (rotamers, 9H, C*Me*$_3$). $^{13}C\{^1H\}$ NMR (126 MHz, $CDCl_3$) δ 154.51, 149.07, 140.72, 140.11, 139.55, 136.26, 135.78, 130.37, 130.32, 128.55, 128.38, 127.50, 126.84, 126.08, 125.56, 124.26, 123.75, 121.88, 79.30 (Ar_2CHN), 53.61 (CMe_3), 28.44 and 28.26 (rotamers C*Me*$_3$), 19.43 (Ar*Me*). HRMS (ESI+) Calcd for $C_{24}H_{27}N_2O_2$ $[MH]^+$ 375.2067; Found 375.2072.

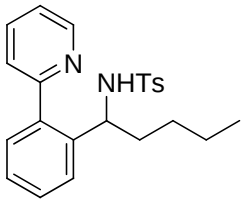


***N-tert*-Butyl ((3-carbonylphenyl)(2-(pyridin-2-yl)phenyl)methyl)carbamate (3r).** To a vial in a glovebox was combined $[Cp^*RhCl_2]_2$ (3.5 mg, 0.006 mmol, 0.1 equiv), $AgSbF_6$ (7.7 mg, 0.022 mmol, 0.4 equiv), 2-phenylpyridine (15.5 mg, 0.100 mmol, 2 equiv), 2,4-dimethoxytoluene (15.4 mg, 0.101 mmol, 2 equiv) *N-tert*-butyl-3-carbonylbenzylidenecarbamate (14.5 mg, 0.062 mmol, 1 equiv), and CH_2Cl_2 (0.5 mL). The slurry was transferred to an NMR tube equipped with a sealed capillary containing C_6D_6. The tube was closed with a Cajon adapter, removed from the glovebox, flame sealed and then placed into an oil bath preheated to 75 ˚C for 20 h. The reaction mixture was then cooled to rt, concentrated *in vacuo*, and purified by chromatography (85:15 hexanes:EtOAc) to yield a white solid (16.9 mg, 0.050 mmol, 81% yield). mp: 52 ˚C. IR (neat): 1694 (s), 1488 (m), 1365 (w), 1246 (m), 1157 (s), 1044 (w), 1018 (m), 794 (m), 752 (s) cm^{-1}. ^1H NMR (600 MHz, $CDCl_3$) δ 9.79 (s, 1H, ArC*H*O), 8.56 (d, *J* = 4.8 Hz, 1H), 7.56 (d, *J* = 7.0 Hz, 1H), 7.49 (d, *J* = 6.6 Hz, 1H), 7.47 – 7.39 (m, 4H), 7.32 (d, *J* = 7.2 Hz, 1H), 7.23 – 7.14 (m, 3H), 7.11 – 7.06 (m, 1H), 6.96 (d, *J* = 7.5 Hz, 1H), 6.25 (d, *J* = 8.9 Hz, 1H), 1.48 (bs, 9H, C*Me*$_3$). $^{13}C\{^1H\}$ NMR (151 MHz, $CDCl_3$) δ 192.24 (Ar*C*HO), 159.78, 155.40, 148.17, 143.56, 140.49, 140.27, 136.58, 135.75, 132.36, 131.11, 130.75, 129.01, 128.29, 128.06, 127.61, 127.01, 124.09, 121.89, 79.52 (Ar_2CHN), 57.39 (*C*Me_3), 28.48 (C*Me*$_3$). HRMS (ESI+) Calcd for $C_{24}H_{25}N_2O_3$ $[MH]^+$ 389.1860; Found 389.1866.



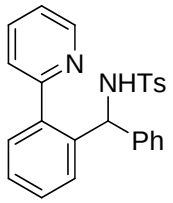
***N-tert*-Butyl(thiophene-2-yl)(2-(pyridin-2-yl)phenyl)methyl)carbamate (3s).** To a vial in a glovebox was combined $[Cp^*RhCl_2]_2$ (3.3 mg, 0.005 mmol, 0.1 equiv), $AgSbF_6$ (9.0 mg, 0.026 mmol, 0.5 equiv), 2-phenylpyridine (15.8 mg, 0.102 mmol, 1 equiv), 2,4-dimethoxy-

toluene (15.5 mg, 0.102 mmol, 1 equiv) a solution of *N-tert*-butyl-(thiophen-2-ylmethylene)-carbamate in CH_2Cl_2 (0.30 mL, 0.27 M, 0.08 mmol, 1 equiv), and CH_2Cl_2 (0.40 mL). The slurry was transferred to an NMR tube equipped with a sealed capillary containing C_6D_6. The tube was closed with a Cajon adapter, removed from the glovebox, flame sealed and then placed into an oil bath preheated to 75 ˚C for 20 h. The reaction mixture was then cooled to rt, concentrated *in vacuo*, and purified by chromatography (85:15 hexanes:EtOAc) to yield an orange solid (21.1 mg, 0.057 mmol, 71% yield). mp: 110 ˚C. IR (neat): 1694 (s), 1470 (s), 1364 (m), 1246 (m), 1160 (s), 1043 (w), 1014 (m), 751 (s), 694 (s) cm^{-1}. ^1H NMR (500 MHz, $CDCl_3$) δ 8.58 (d, J = 4.1 Hz, 1H), 7.59 (t, J = 7.3 Hz, 1H), 7.55 (d, J = 6.7 Hz, 1H), 7.47 – 7.34 (m, 3H), 7.24 – 7.18 (m, 1H), 7.14 (dd, J = 6.0, 1H), 6.95 (m, 2H), 6.65 (s, 1H), 6.42 (s, 1H), 6.37 (d, J = 8.5 Hz, 1H), 1.46 (bs, 9H, C*Me*$_3$). $^{13}C\{^1H\}$ NMR (126 MHz, $CDCl_3$) δ 159.71, 155.03, 148.47, 147.32, 140.42, 136.53, 130.98, 129.31, 128.85, 128.03, 126.26, 124.48, 124.30, 124.22, 123.97, 121.82, 79.45 (Ar_2CHN), 54.11 (CMe_3), 28.47 and 28.34 (rotamers of C*Me*$_3$). HRMS (ESI+) Calcd for $C_{21}H_{23}N_2O_2S$ $[MH]^+$: 367.1475; Found 367.1480.



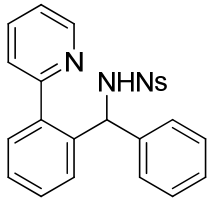
4-Methyl-*N*-(1-(2-(pyridin-2-yl)phenyl)pentyl)benenesulfonamide (3u). To a vial in a glovebox was combined $[Cp^*RhCl_2]_2$ (6.5 mg, 0.011 mmol, 0.1 equiv), $AgSbF_6$ (8.9 mg, 0.026 mmol, 0.3 equiv), 2-phenylpyridine (14.8 mg, 0.0953 mmol, 1 equiv), 2,4-dimethoxytoluene (14.6 mg, 0.0959 mmol, 1 equiv), 4-methyl-*N*-pentylidenebenzenesulfonamide[8] and CH_2Cl_2 (0.70 mL). The slurry was transferred to an NMR tube equipped with a sealed capillary containing C_6D_6. The tube was closed with a Cajon adapter, removed from the glovebox, flame

sealed and then placed into an oilbath preheated to 75 ˚C for 20 h. The reaction mixture was then cooled to rt, concentrated *in vacuo*, and purified by chromatography (75:25 hexanes:EtOAc) to yield a white solid (26.4 mg, 0.069 mmol, 72% yield). mp: 105 ˚C. IR (neat): 3060 (w), 1588 9w), 1469 (m), 1323 (s), 1157 (s), 1147 (s), 1092 (s), 1053 (m), 997 (w), 951 (m), 754 (s), 658 (s) cm^{-1}. ^1H NMR (300 MHz, $CDCl_3$) δ 8.72 (d, *J* = 4.1 Hz, 1H), 8.37 (s, 1H), 7.84 (td, *J* = 7.7, 1.8 Hz, 1H), 7.54 (d, *J* = 8.3 Hz, 2H), 7.42 (d, *J* = 7.9 Hz, 1H), 7.38 – 7.33 (m, 1H), 7.26 – 7.16 (m, 2H), 7.04 – 6.95 (m, 3H), 6.83 (d, *J* = 7.4 Hz, 1H), 4.33 (ddd, *J* = 7.7 Hz, 1H, Ar*CH*(NHTs)Bu), 2.31 (s, 3H), 1.48 – 1.34 (m, 1H), 1.23 – 1.10 (m, 2H, CH_2Pr), 1.11 – 0.98 (m, 2H, CH_2Et), 1.00 – 0.84 (m, 1H, $CH_2CH_2CH_3$), 0.68 (t, *J* = 7.1 Hz, 3H, CH_2CH_3). $^{13}C\{^1H\}$ NMR (151 MHz, $CDCl_3$) δ 160.61, 148.29, 142.21, 139.53, 138.82, 138.69, 137.65, 131.48, 130.41, 129.04, 128.37, 127.30, 127.03, 124.82, 122.46, 59.59 (*n*Bu*CH*NTs), 35.17 (CH_2Pr), 28.69 (CH_2Et), 22.14 (Ar-*Me*), 21.49 (CH_2Me), 13.87 (CH_2*Me*). HRMS (ESI+) Calcd for $C_{23}H_{27}N_2O_2S$ $[MH]^+$: 395.1788; Found 395.1786.



4-Methyl-N-(phenyl(2-(pyridin-2-yl)phenyl)methyl)benzenesulfonamide (3v). To a vial in a glovebox was combined $[Cp^*RhCl_2]_2$ (15.5 mg, 0.0250 mmol, 0.1 equiv), $AgSbF_6$ (34.3 mg, 0.100 mmol, 0.4 equiv), 2-phenylpyridine (39 mg, 0.25 mmol, 1 equiv), *N*-benzylidene-4-methylbenzenesulfonamide (65 mg, 0.25 mmol, 1 equiv), and CH_2Cl_2 (2.5 mL). The reaction was transferred to a 5 mL schlenk tube, removed from the glovebox, and heated at 75 ˚C for 20 h. Reaction was then cooled to rt, concentrated *in vacuo*, and purified by chromatography (5:1 hex:EtOAc) to yield a white solid (42 mg, 0.10 mmol, 40% yield) mp: 152-154 ˚C. IR (film):

3260, 1330, 1148, 1048 cm^{-1}. ^1H NMR (400 MHz, $CDCl_3$): δ 8.78 (d, *J* = 10.5 Hz, 1H), 8.45 (d, *J* = 4.6 Hz, 1H), 7.54 (d, *J* = 7.9 Hz, 2H), 7.42-7.33 (m, 1H), 7.23-7.10 (m, 2H), 7.06-6.90 (m, 5H), 6.90-6.78 (m, 6H), 5.63 (d, *J* = 9.9 Hz, 1H), 2.25 (s, 3H). $^{13}C\{^1H\}$ NMR (100 MHz, $CDCl_3$): δ 159.8, 147.5, 142.3, 140.7, 139.9, 139.4, 138.7, 137.0, 131.4, 131.1, 129.1, 128.1, 127.7, 127.3, 126.9, 126.2, 125.9, 124.4, 121.9, 61.4, 21.4. HRMS (ESI+) Calcd for $C_{25}H_{23}N_2O_2S$ $[MH]^+$ 415.1480; Found 415.1478.



4-Nitro-*N*-(phenyl-(2-(pyridin-2-yl)phenyl)methyl)benenesulfonamide (3w). To a vial in a glovebox was combined $[Cp^*RhCl_2]_2$ (1.6 mg, 0.003 mmol, 0.05 equiv), $AgSbF_6$ (5.0 mg, 0.015 mmol, 0.3 equiv), 2-phenylpyridine (15.8 mg, 0.102 mmol, 2 equiv), 2,4-dimethoxytoluene (15.5 mg, 0.102 mmol, 2 equiv), 4-nitro-*N*-benzylidenebenzenesulfonamide, and CH_2Cl_2 (0.60 mL). The slurry was transferred to an NMR tube equipped with a sealed capillary containing C_6D_6. The tube was closed with a Cajon adapter, removed from the glovebox, flame sealed and then placed into an oilbath preheated to 75 ˚C for 20 h. The reaction mixture was then cooled to rt, concentrated *in vacuo*, and purified by chromatography (85:15 hexanes:EtOAc) to yield a white solid (16.6 mg, 0.028 mmol, 51% yield). mp: 130 ˚C. IR (neat): 2160 (m, broad), 2028 (m, broad), 1526 (s), 1347 (s), 1301 (m), 1162 (s), 1091 (m), 1027 (w), 854 (m) 794 (w), 745 (s), 734 (s), 697 (m), 685 (s) cm^{-1}. ^1H NMR (600 MHz, $CDCl_3$) δ 9.74 (d, *J* = 7.5 Hz, 1H), 8.55 (d, *J* = 4.8 Hz, 1H), 8.07 (d, *J* = 8.8 Hz, 2H), 7.90 (d, *J* = 8.8 Hz, 2H), 7.52 (td, *J* = 7.7, 1.6 Hz, 1H), 7.28 – 7.23 (m, 2H), 7.16 – 7.12 (m, 1H), 7.07 (td, *J* = 7.4, 1.5 Hz, 1H), 7.04 – 7.01 (m, 1H), 7.00 – 6.95 (m, 6H), 5.77 (d, *J* = 6.5 Hz, 1H). $^{13}C\{^1H\}$ NMR (151 MHz,

$CDCl_3$) δ 159.59, 149.31, 147.70, 147.35, 139.74, 139.24, 139.18, 137.44, 131.91, 131.20, 128.30, 128.29, 128.05, 127.52, 126.53, 125.75, 124.79, 123.63, 122.24, 62.17. HRMS (ESI+) Calcd for $C_{24}H_{20}N_3O_4S$ $[MH]^+$: 446.116; Found 446.116

V. References:

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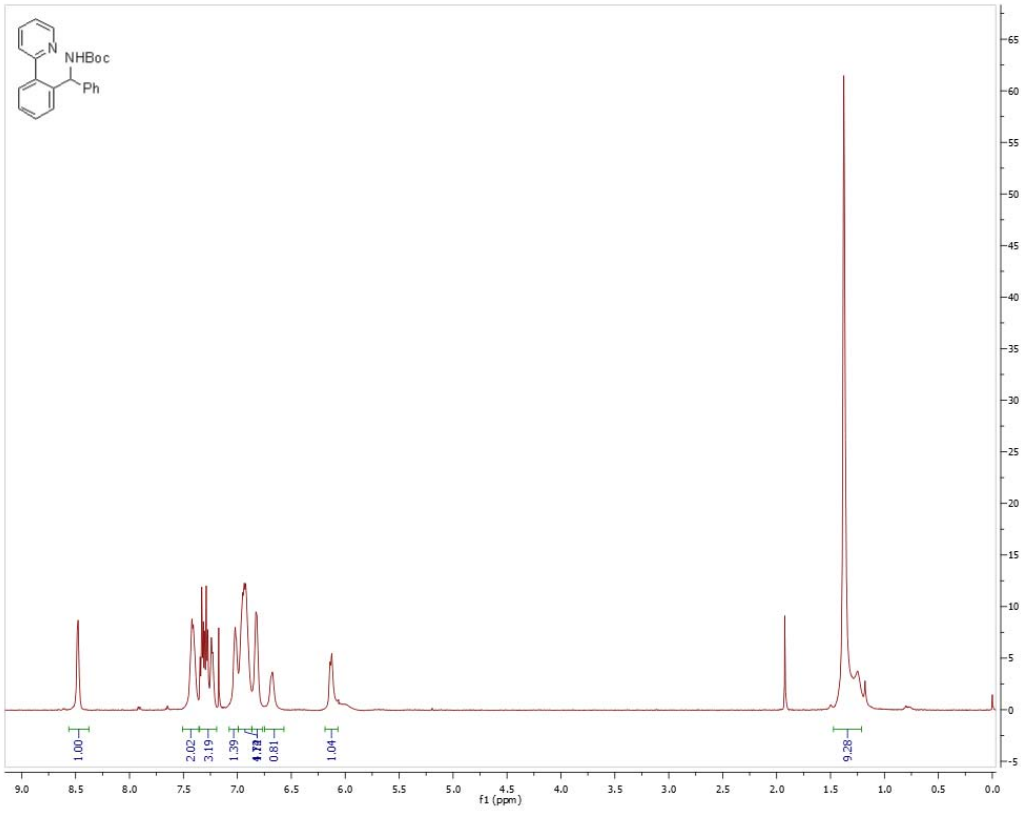
8. Chemla, F.; Hebbe, V.; and Normant, J. F. *Synthesis* **2000**, *2000*, 75-77.

VI. Spectral Data:



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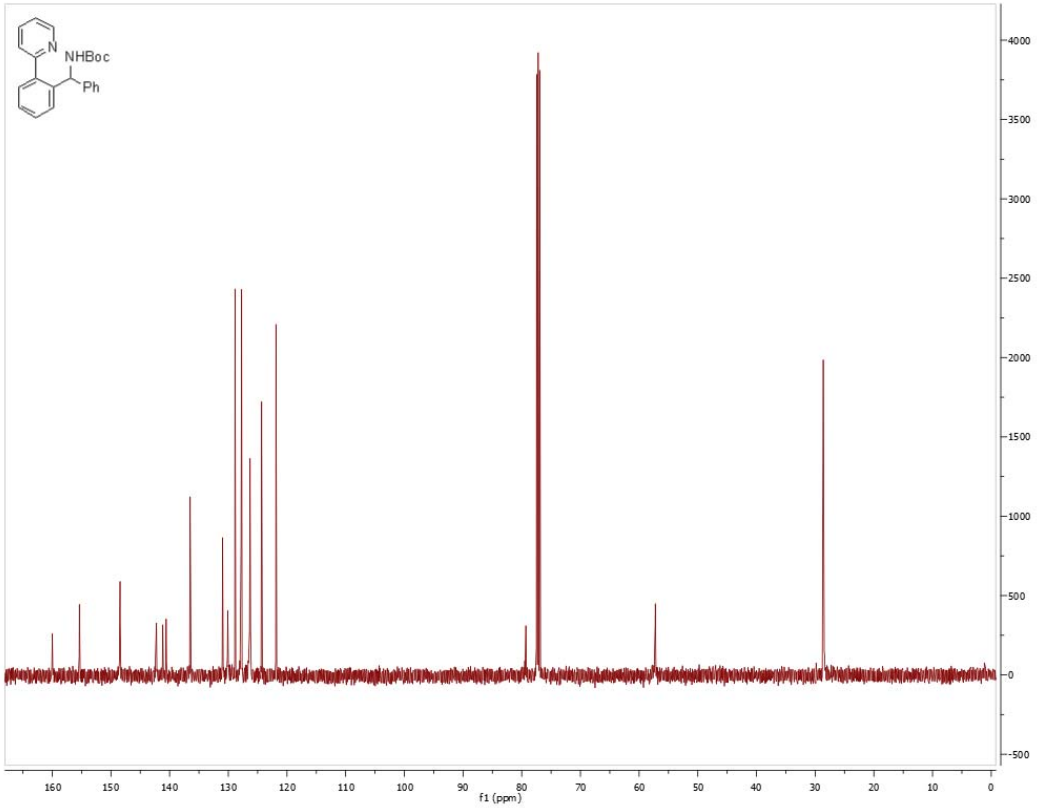
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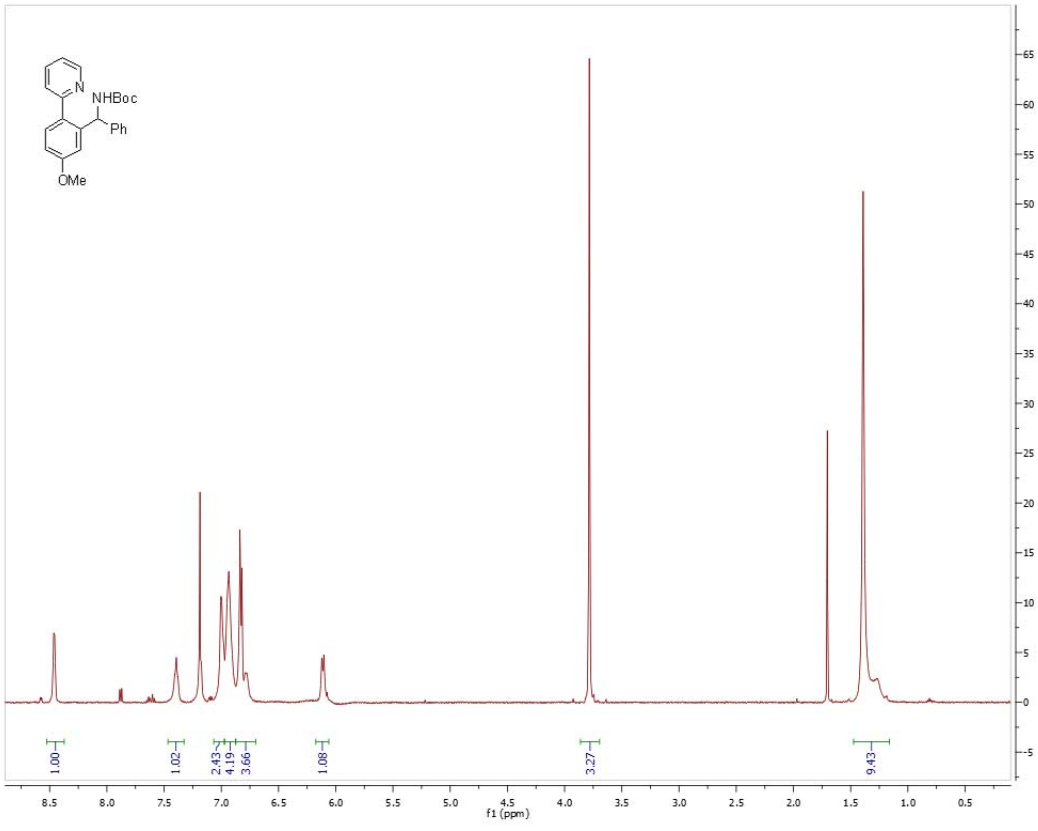
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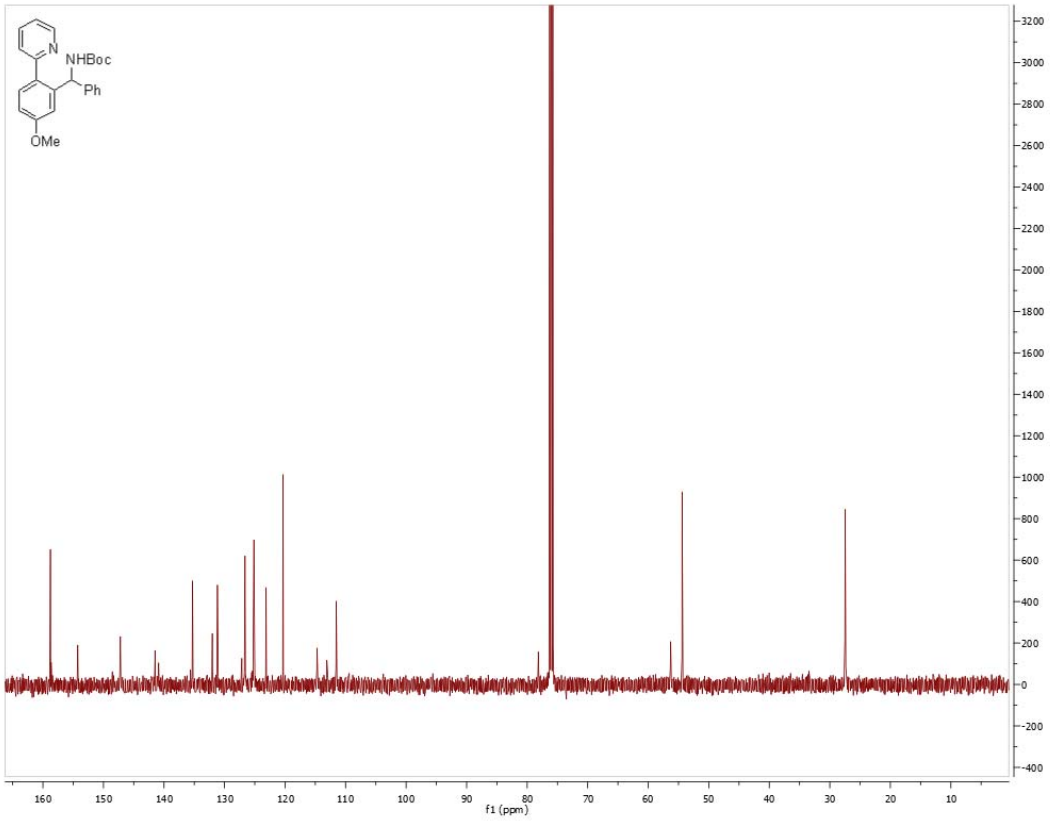
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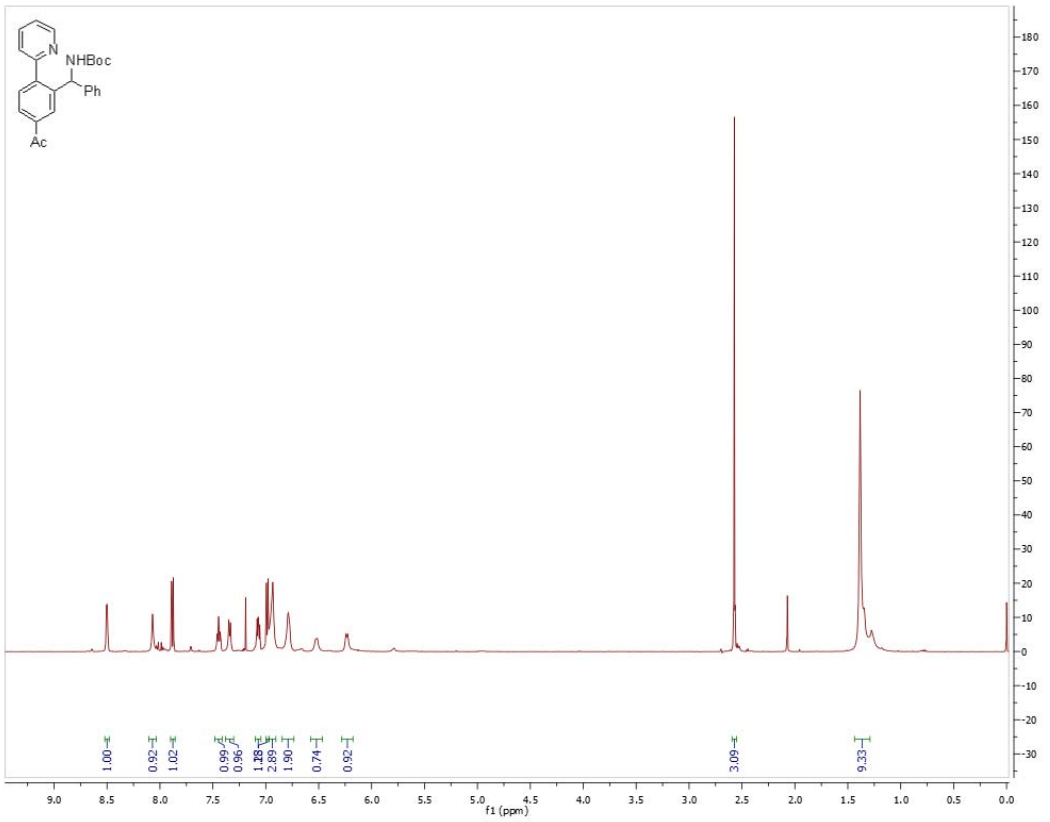
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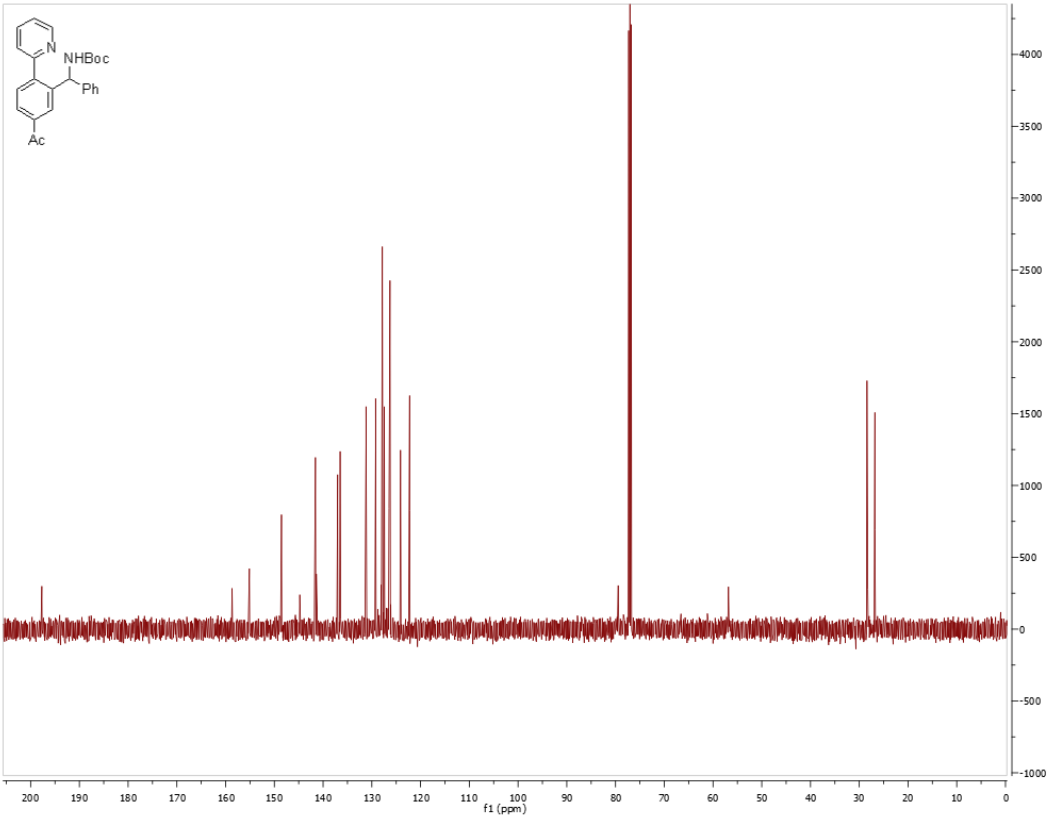
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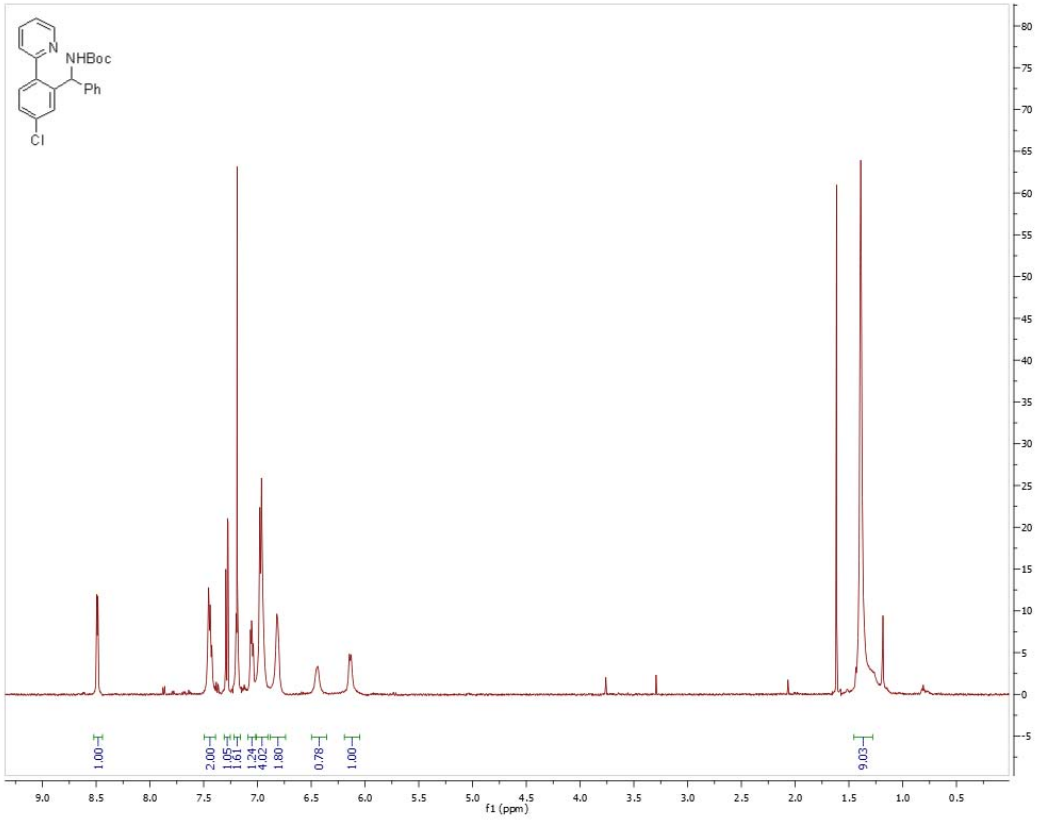
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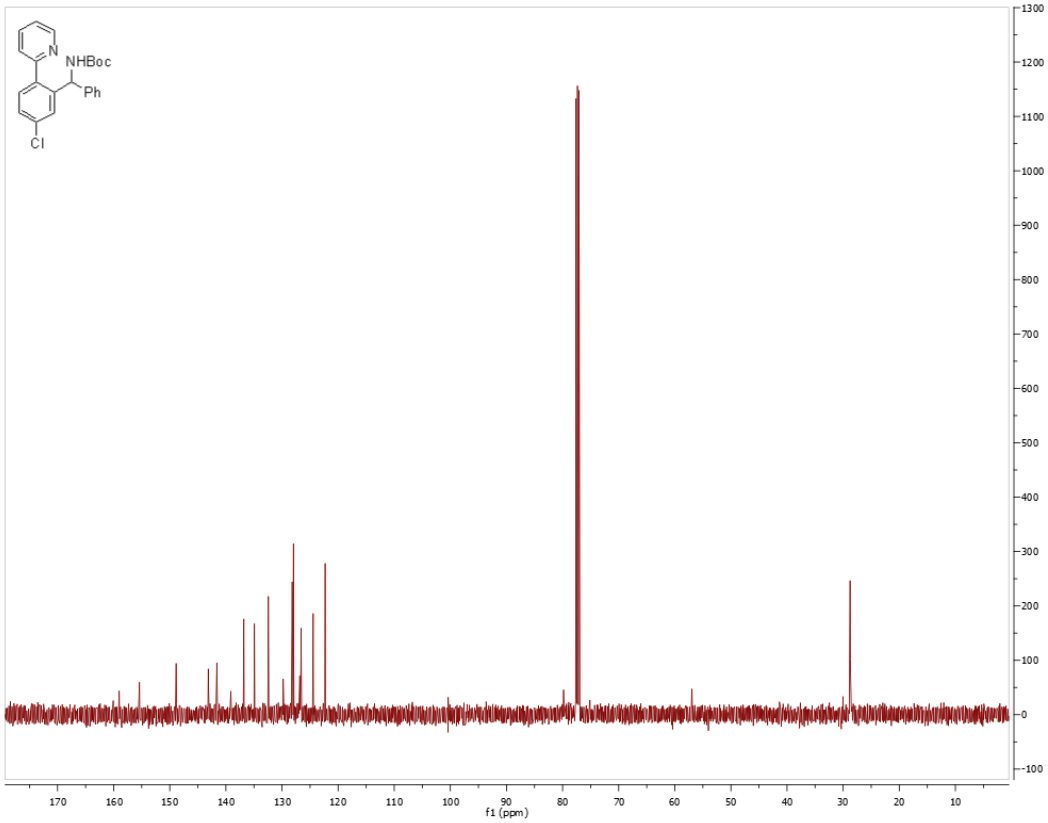
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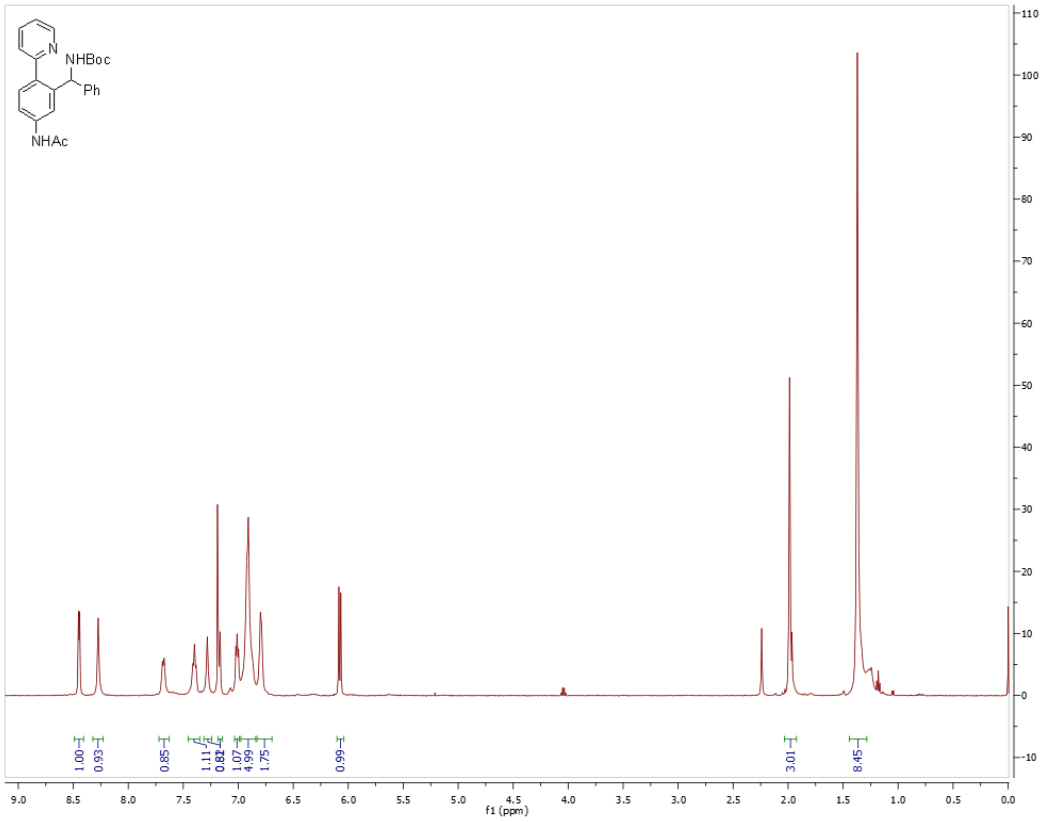
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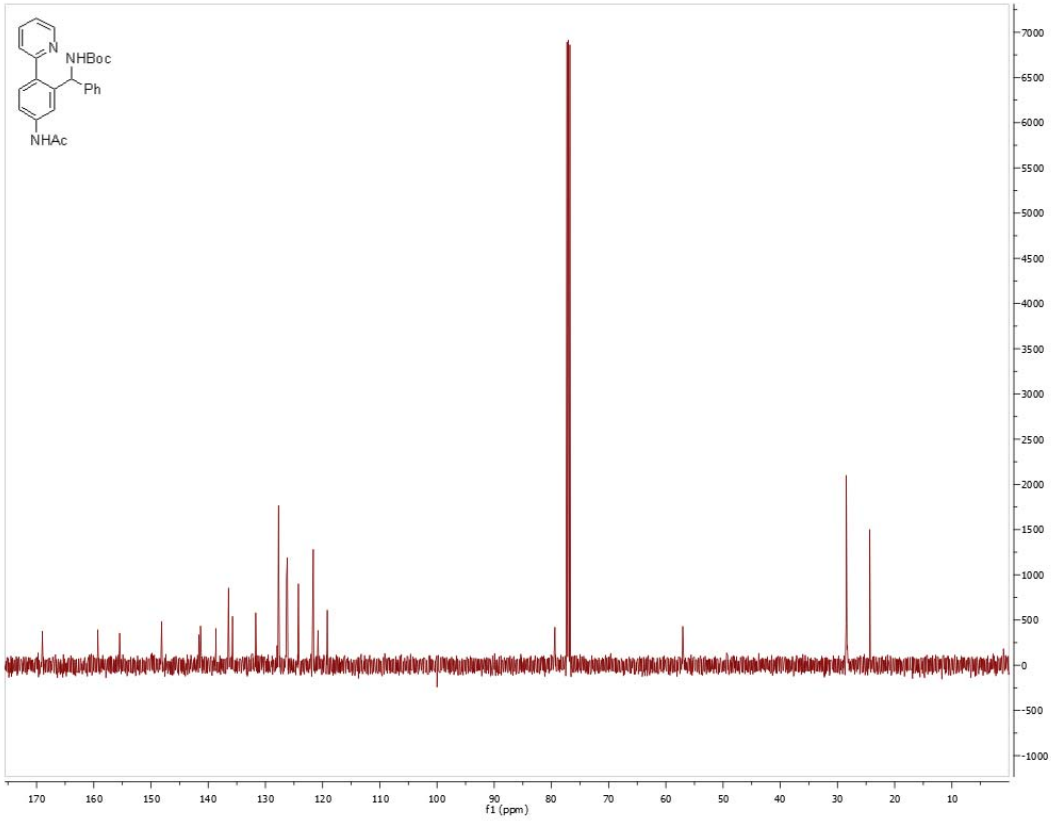
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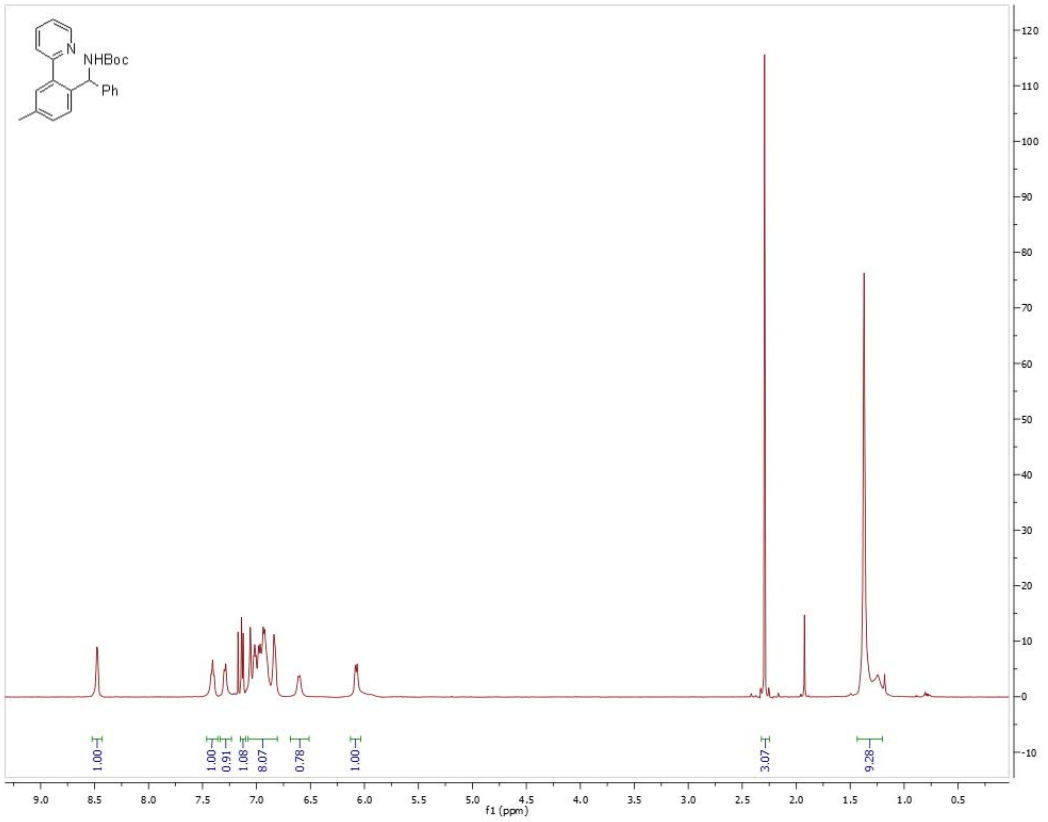
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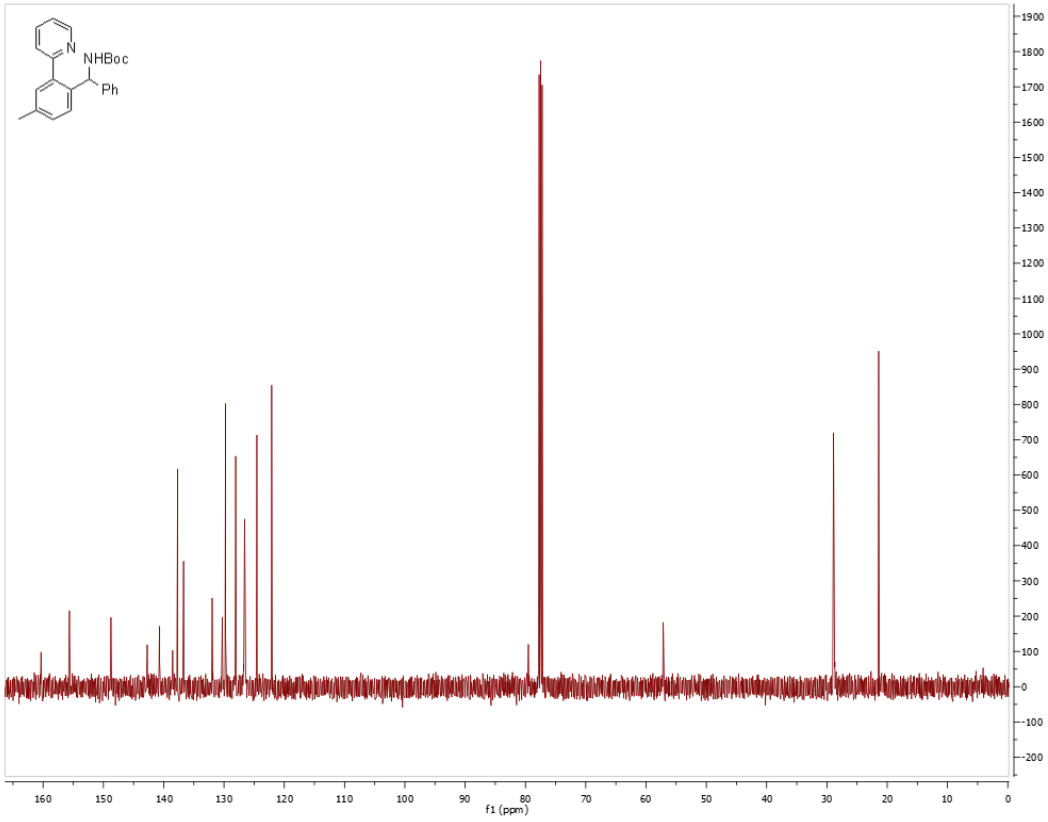
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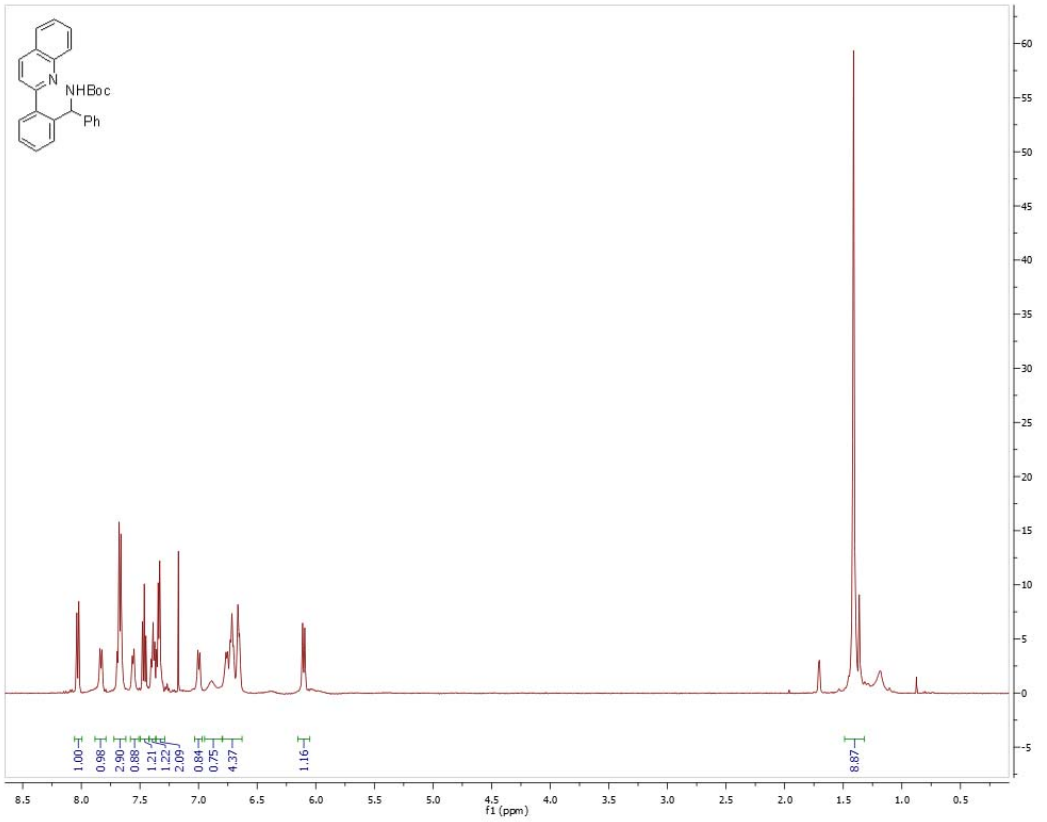
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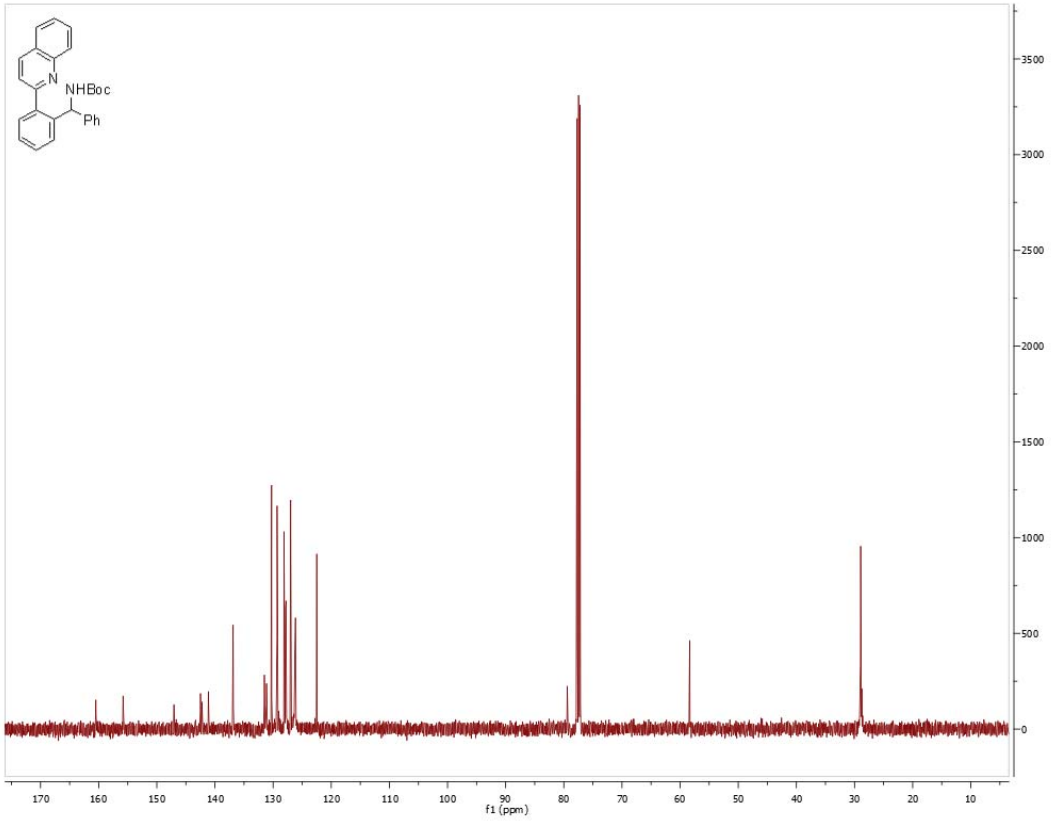
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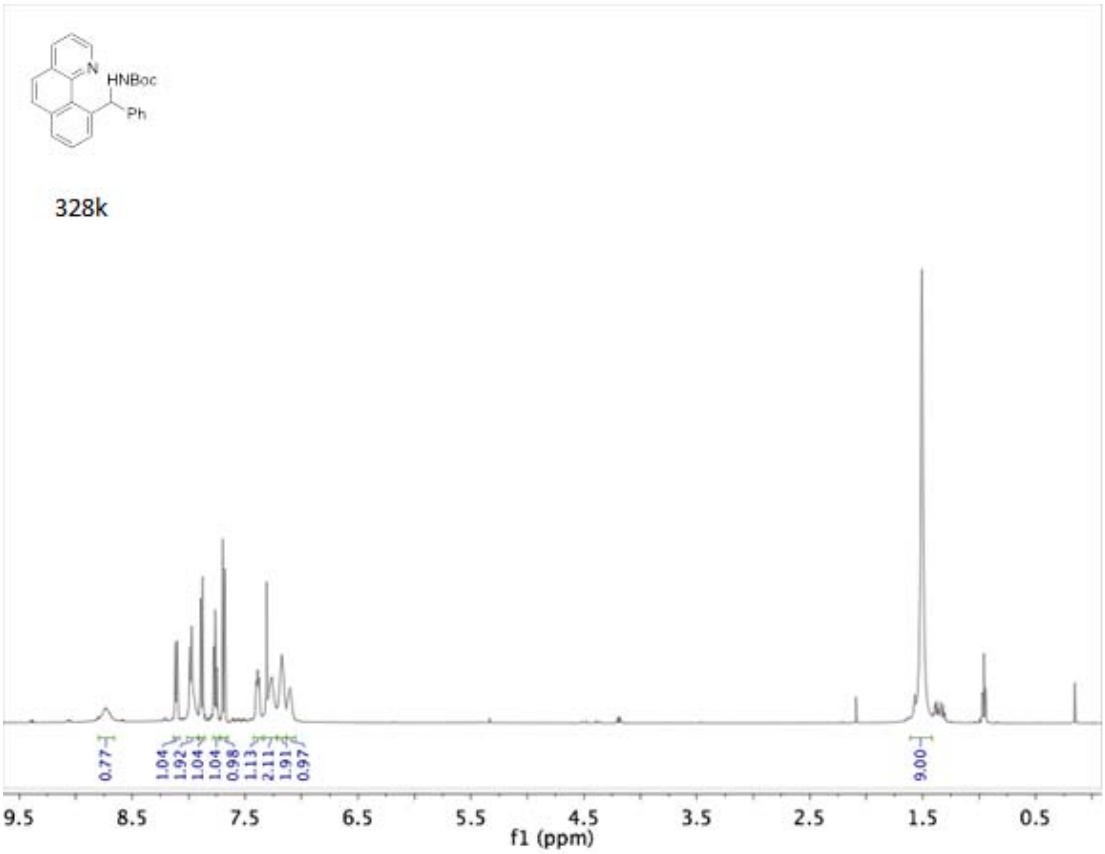
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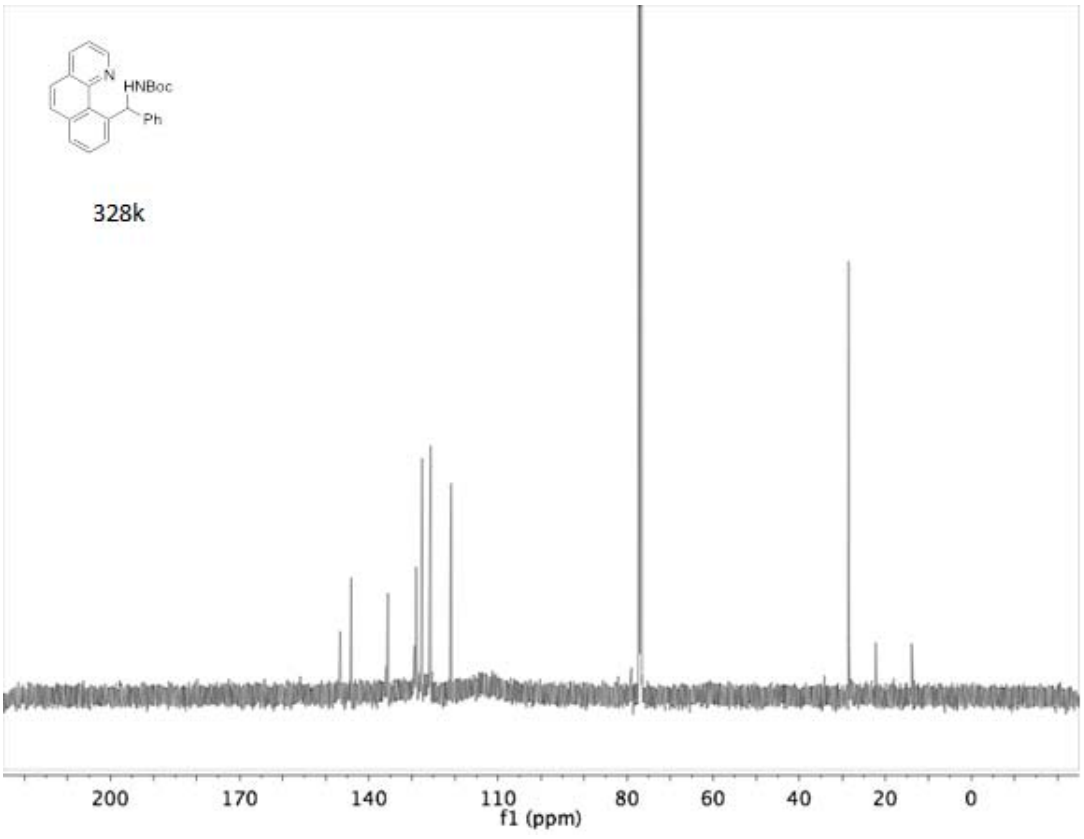
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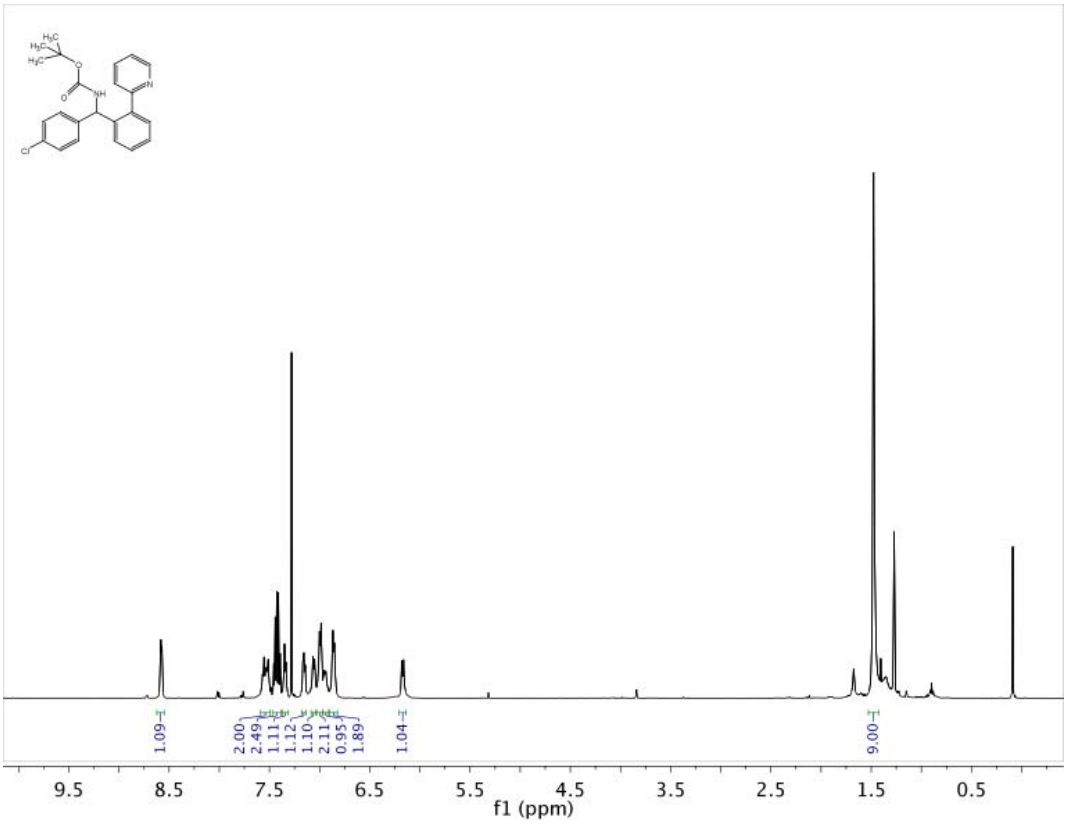
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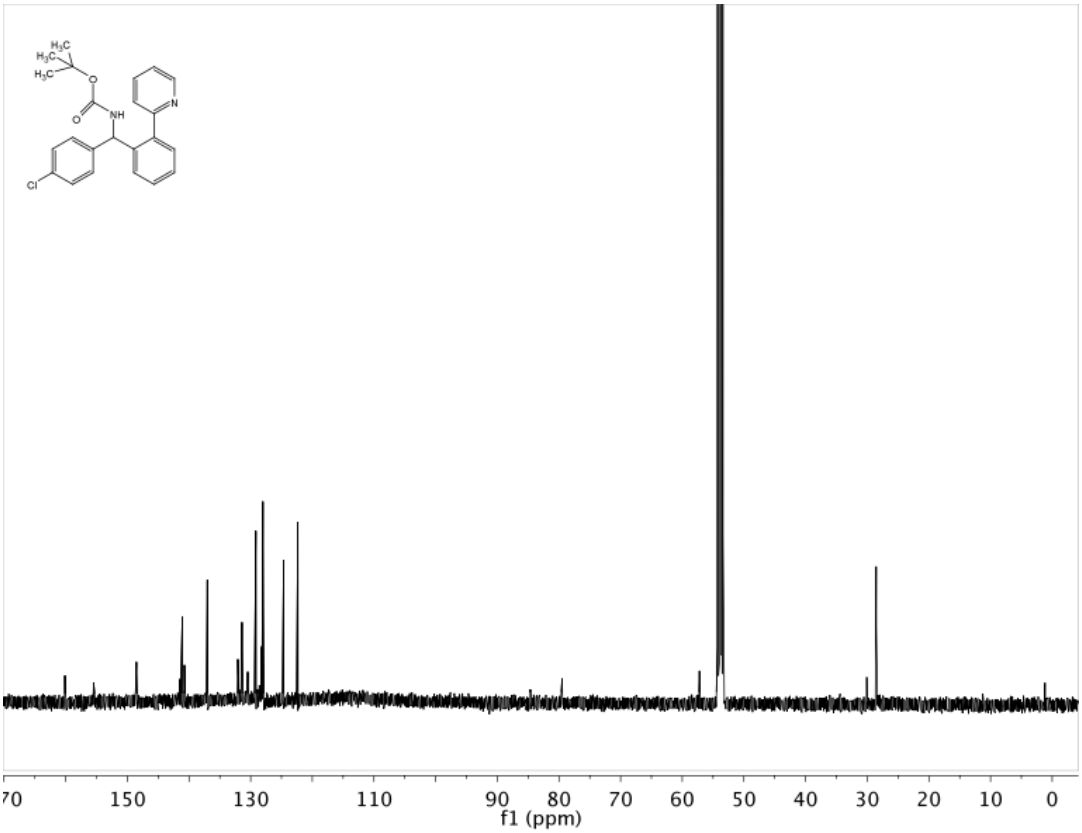
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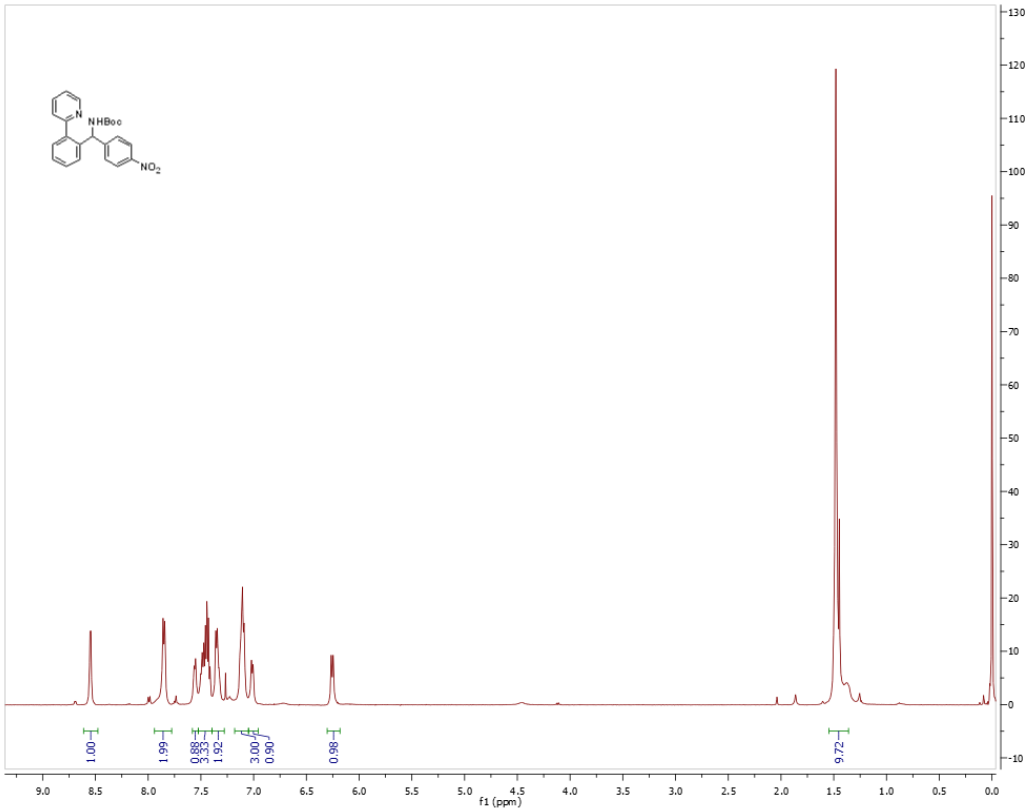
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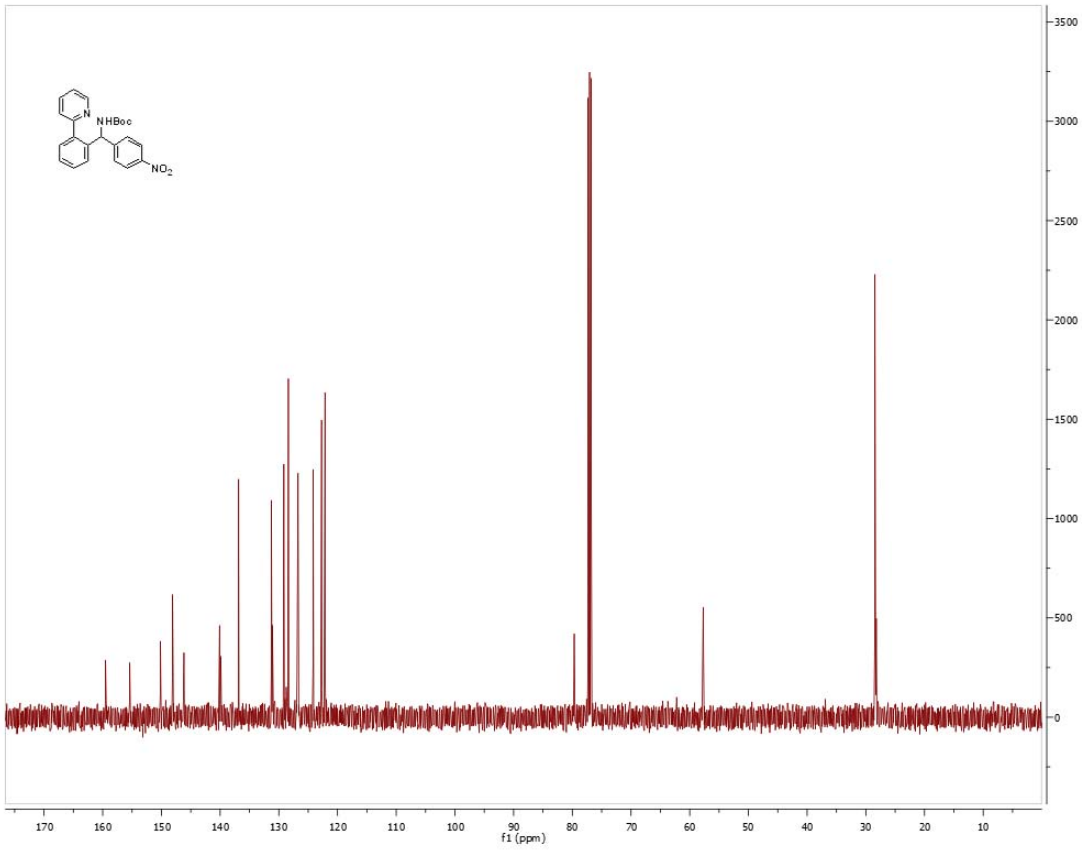
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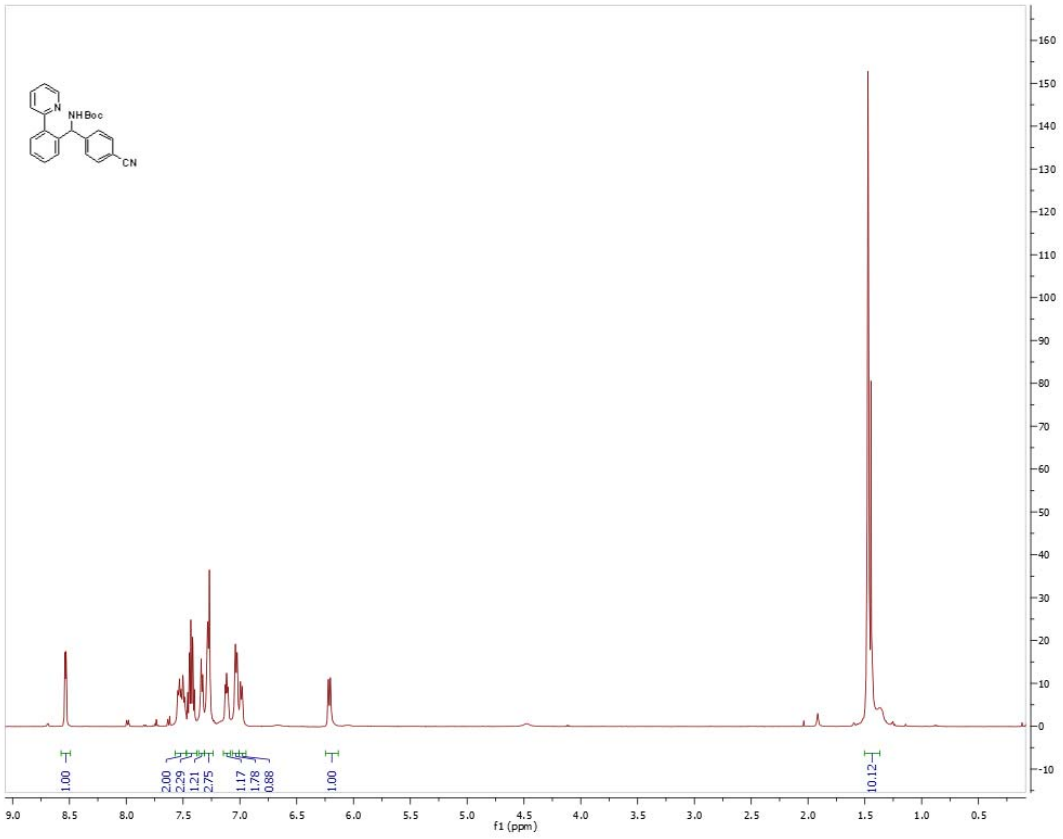
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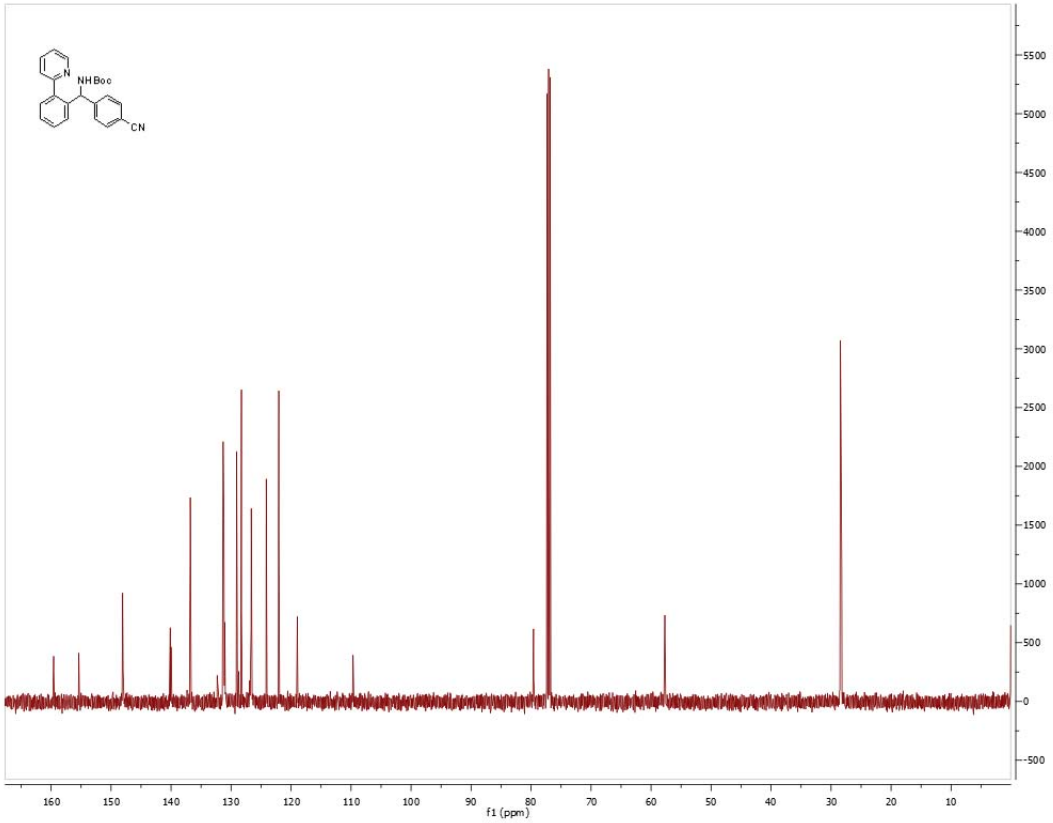
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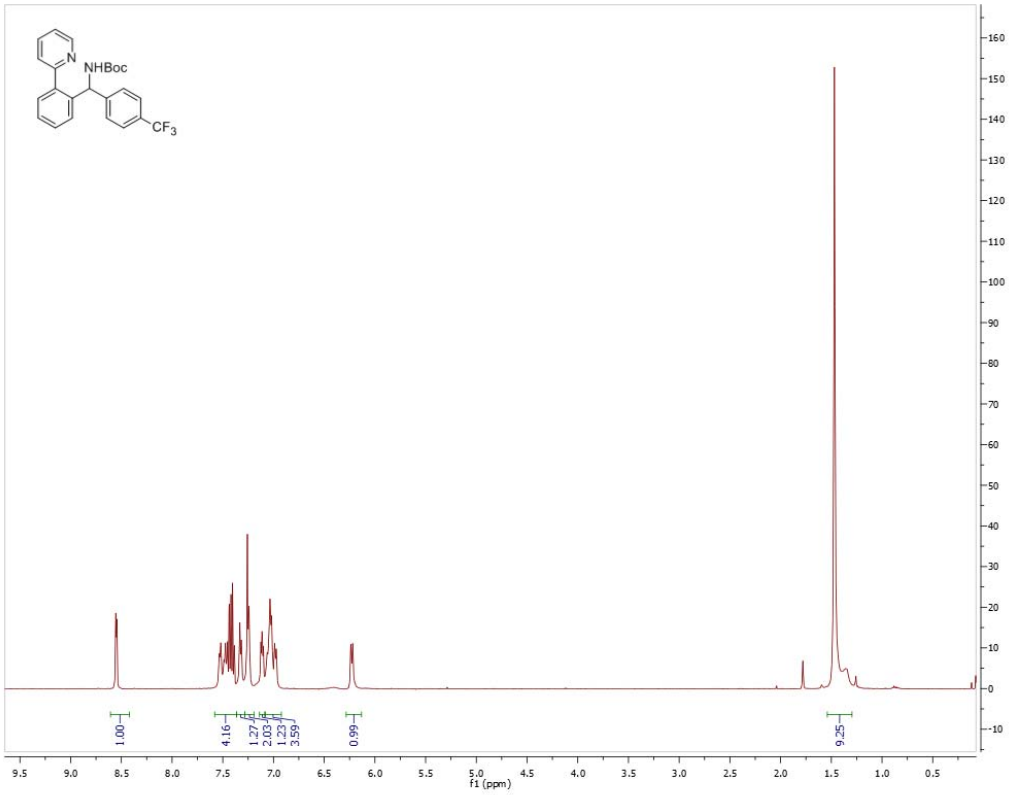
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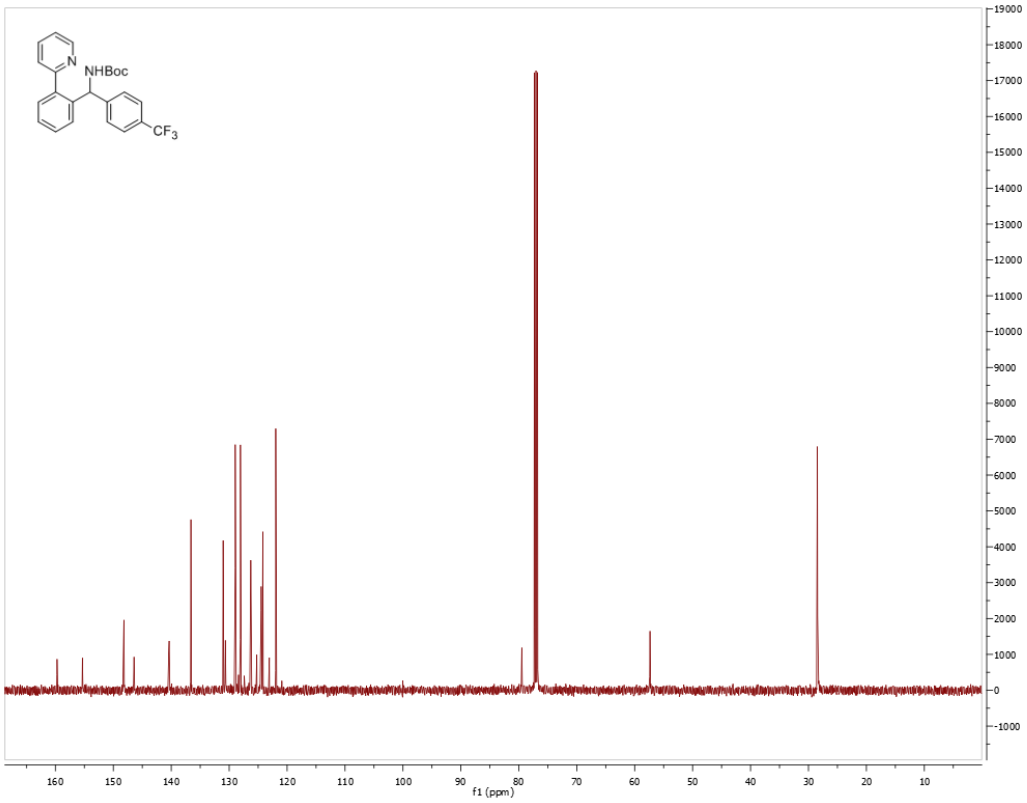
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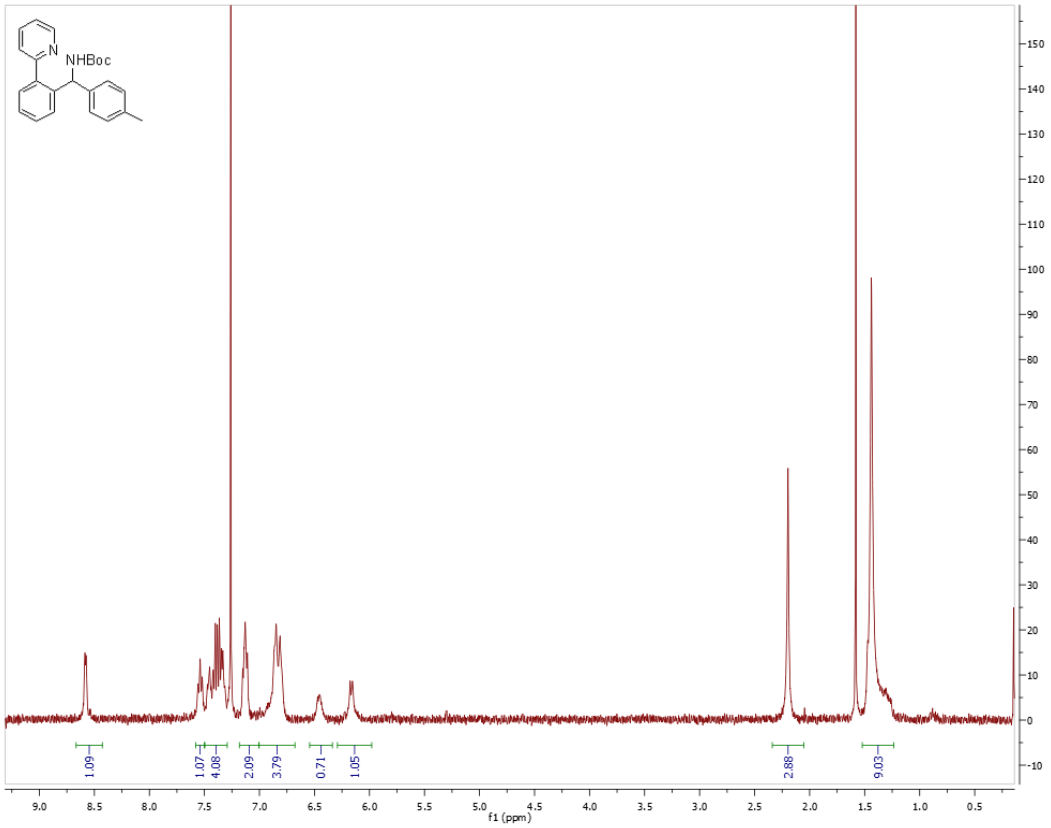
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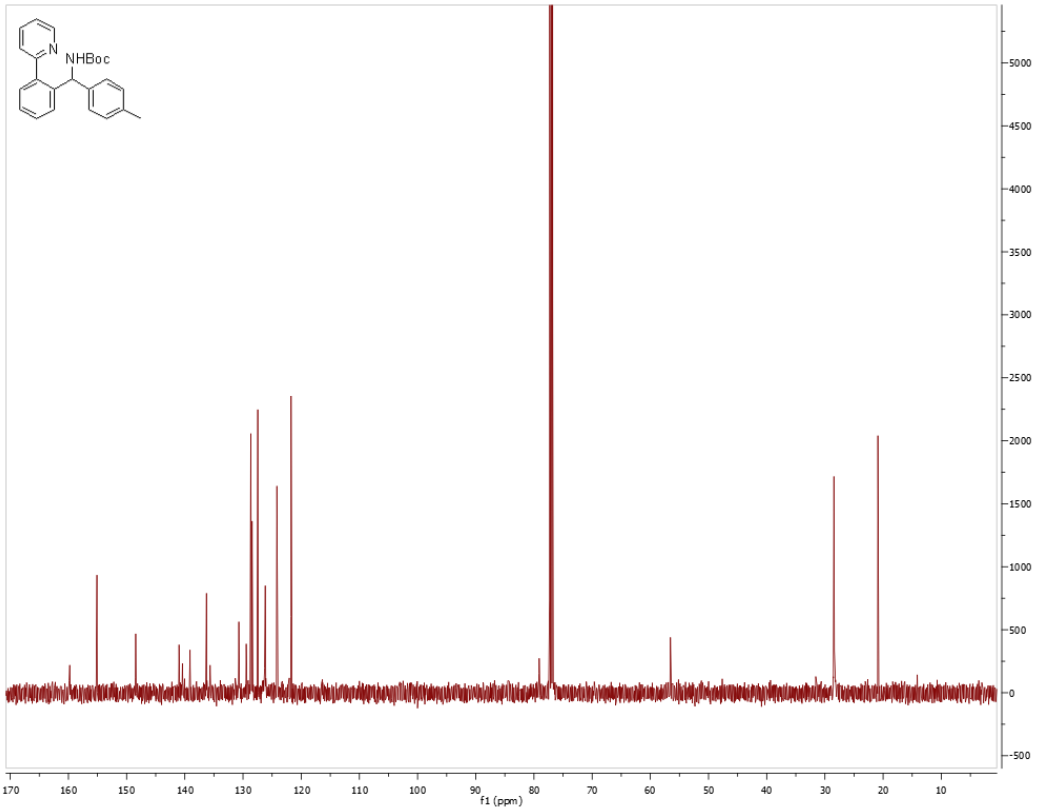
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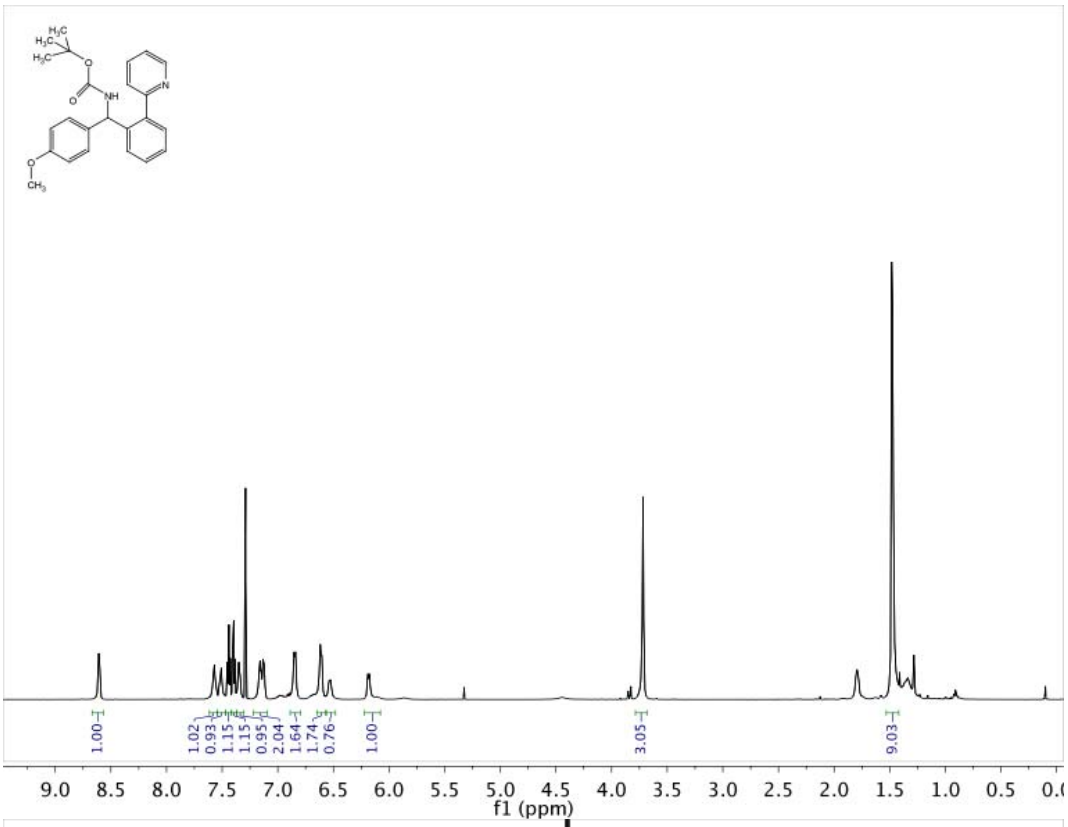
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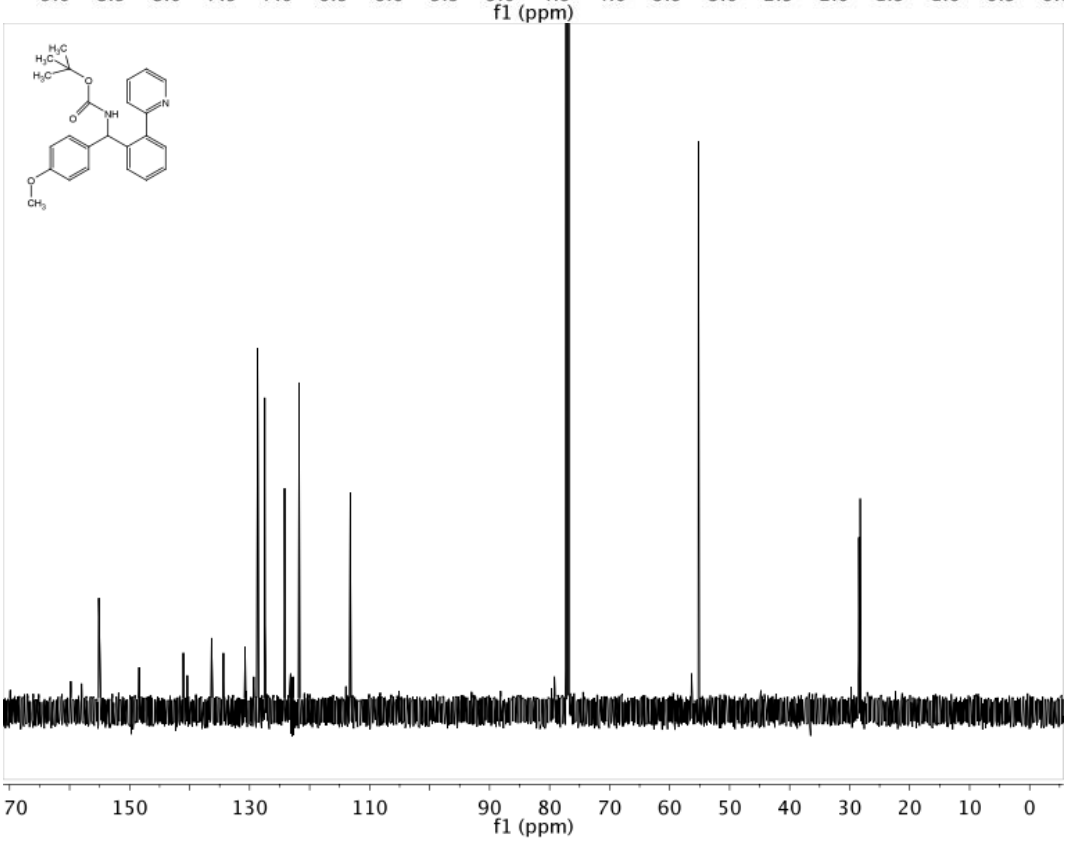
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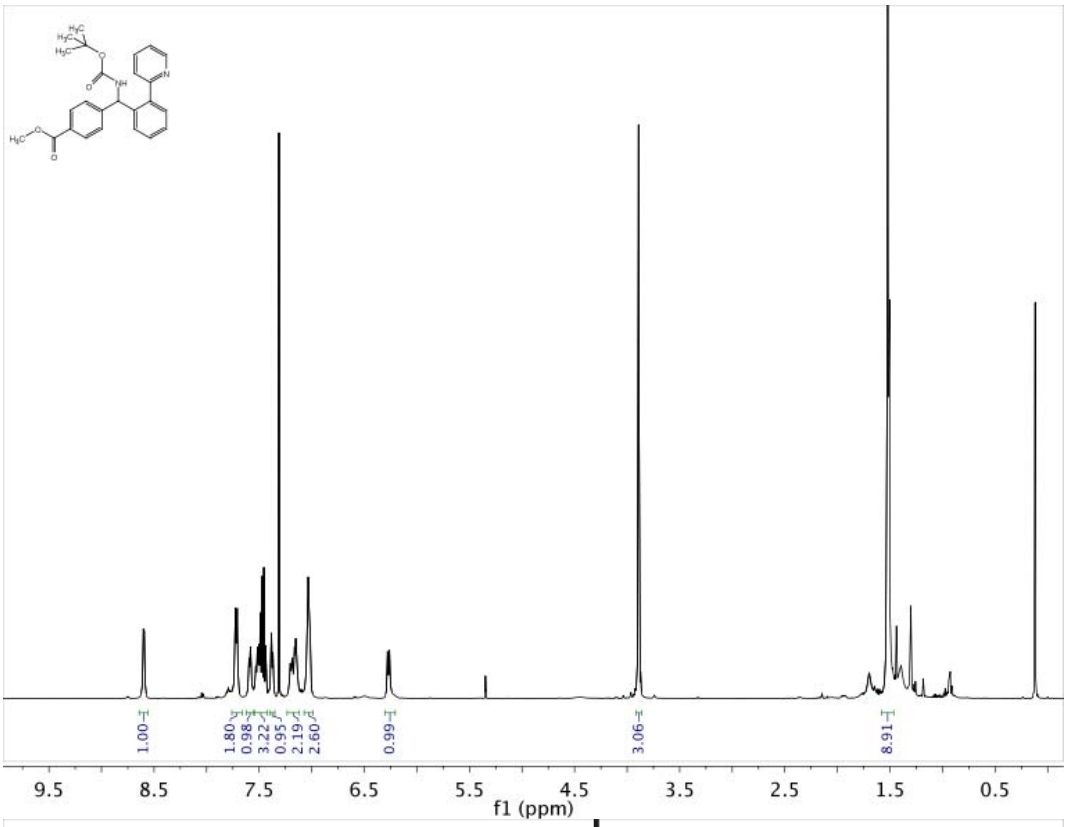
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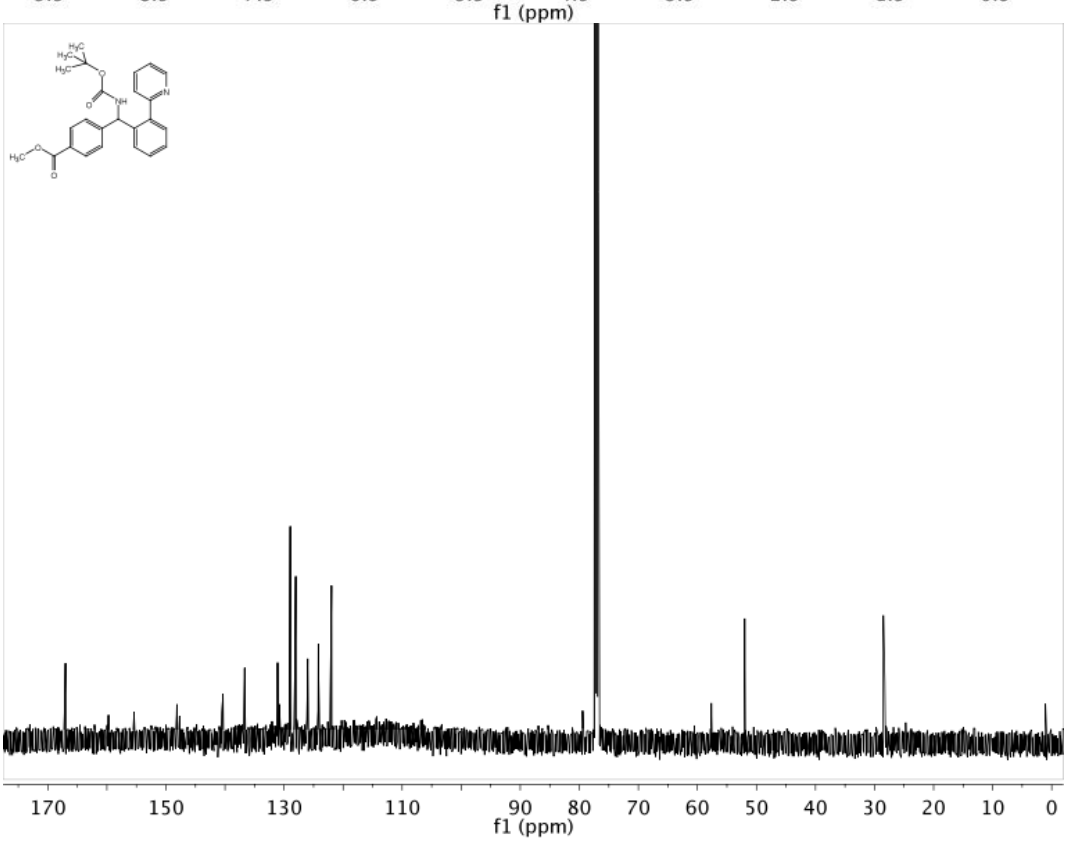
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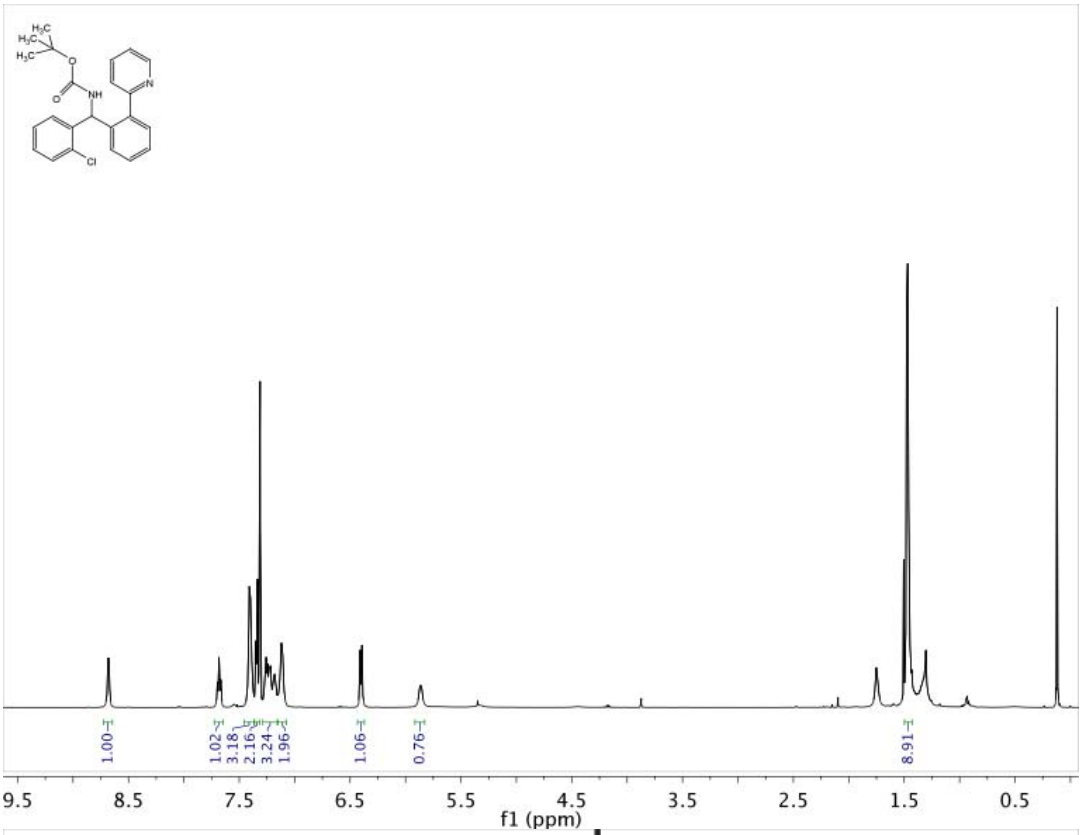
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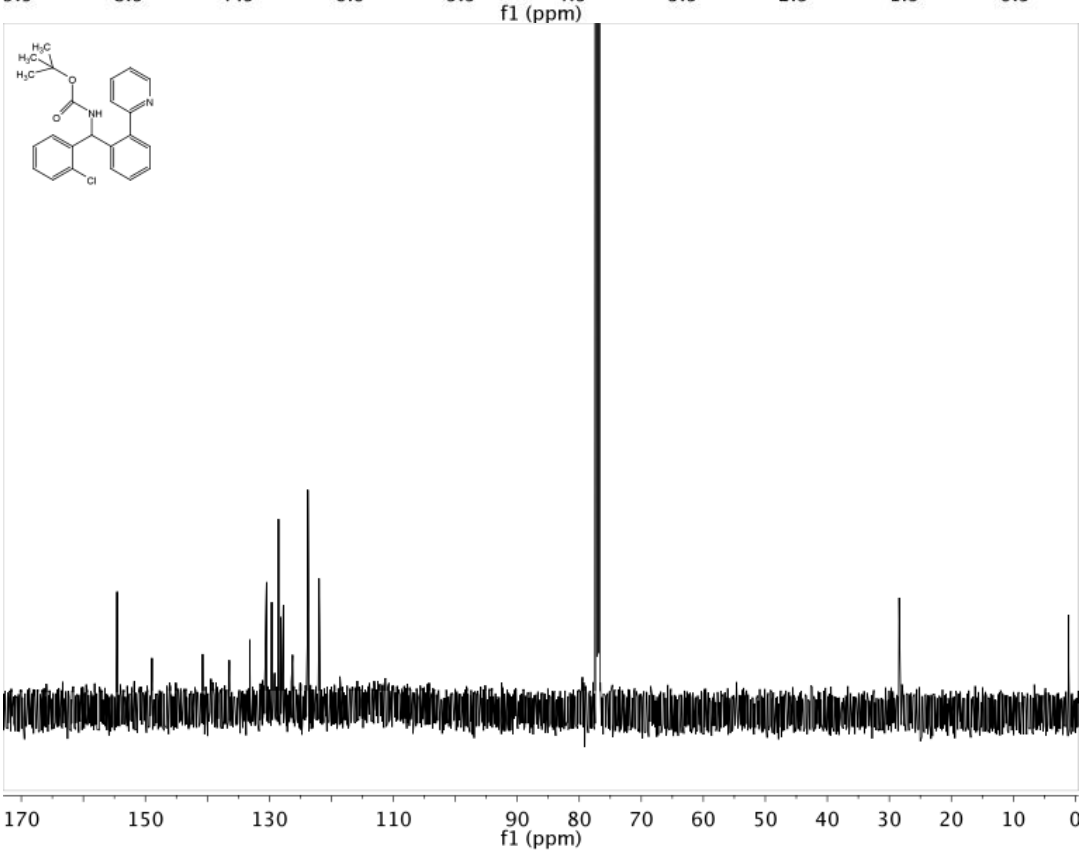
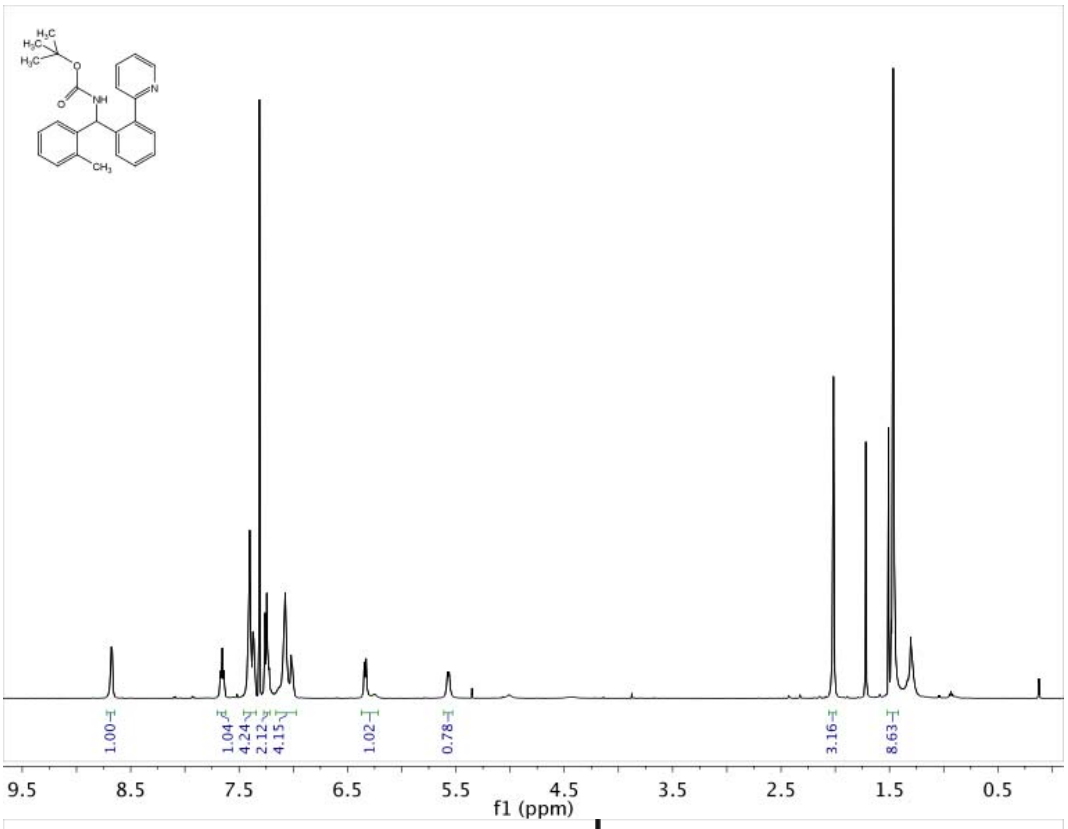
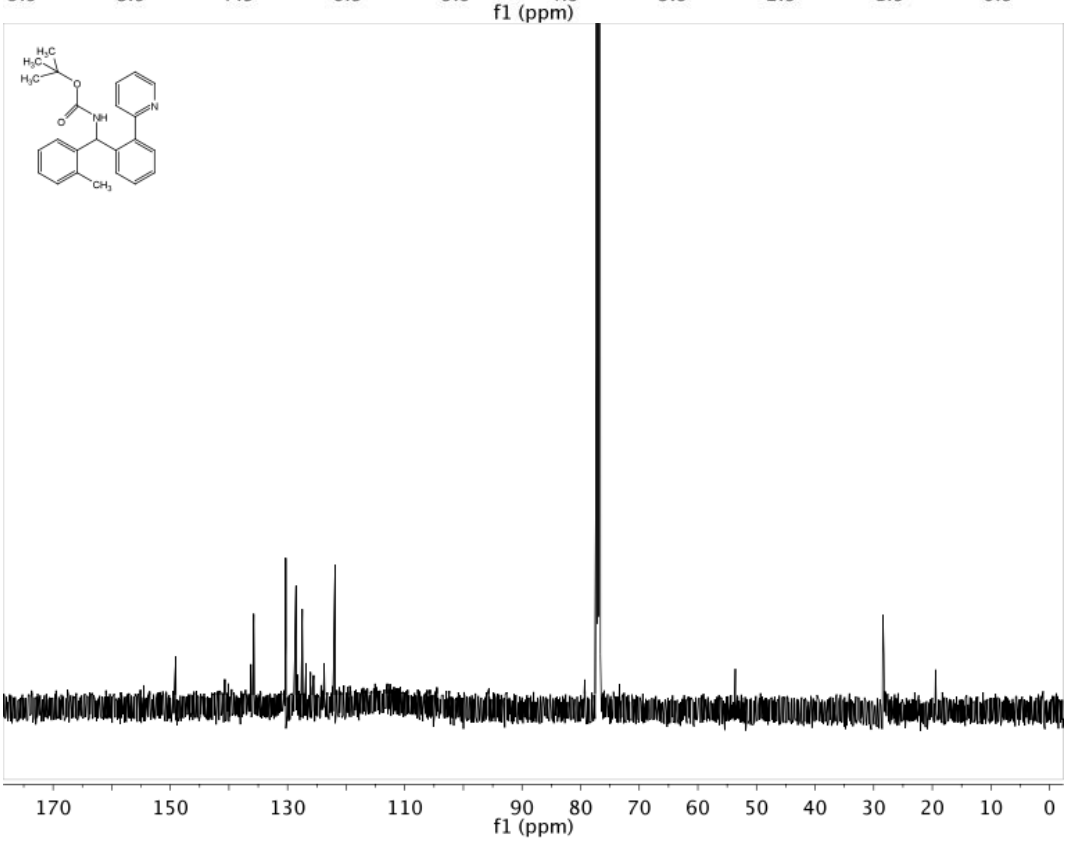
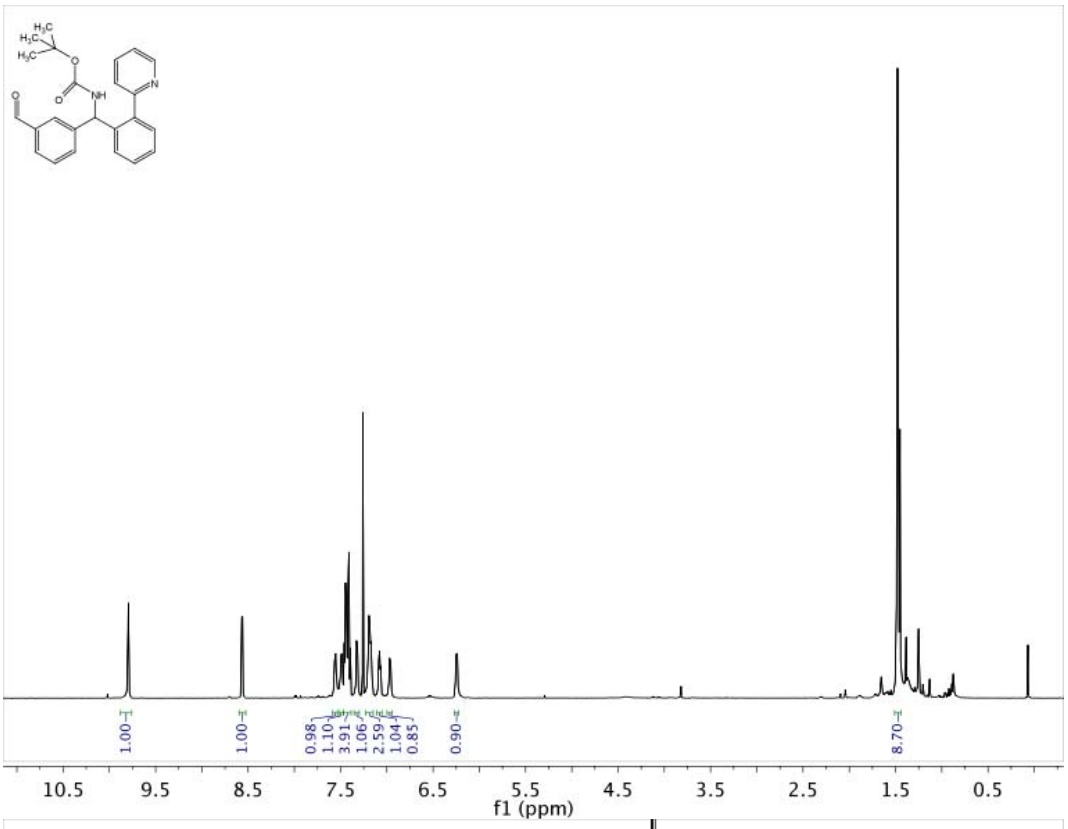
1.02
3.18
2.16
3.24
1.96
1.06
0.76
8.91
9.5
8.5
7.5
6.5
5.5
4.5
3.5
2.5
1.5
0.5
f1 (ppm)


170
150
130
110
90
80
70
60
50
40
30
20
10
0
f1 (ppm)


1.00
1.04
4.24
2.12
4.15
1.02
0.78
3.16
8.63
9.5
8.5
7.5
6.5
5.5
4.5
3.5
2.5
1.5
0.5
f1 (ppm)


170
150
130
110
90
80
70
60
50
40
30
20
10
0
f1 (ppm)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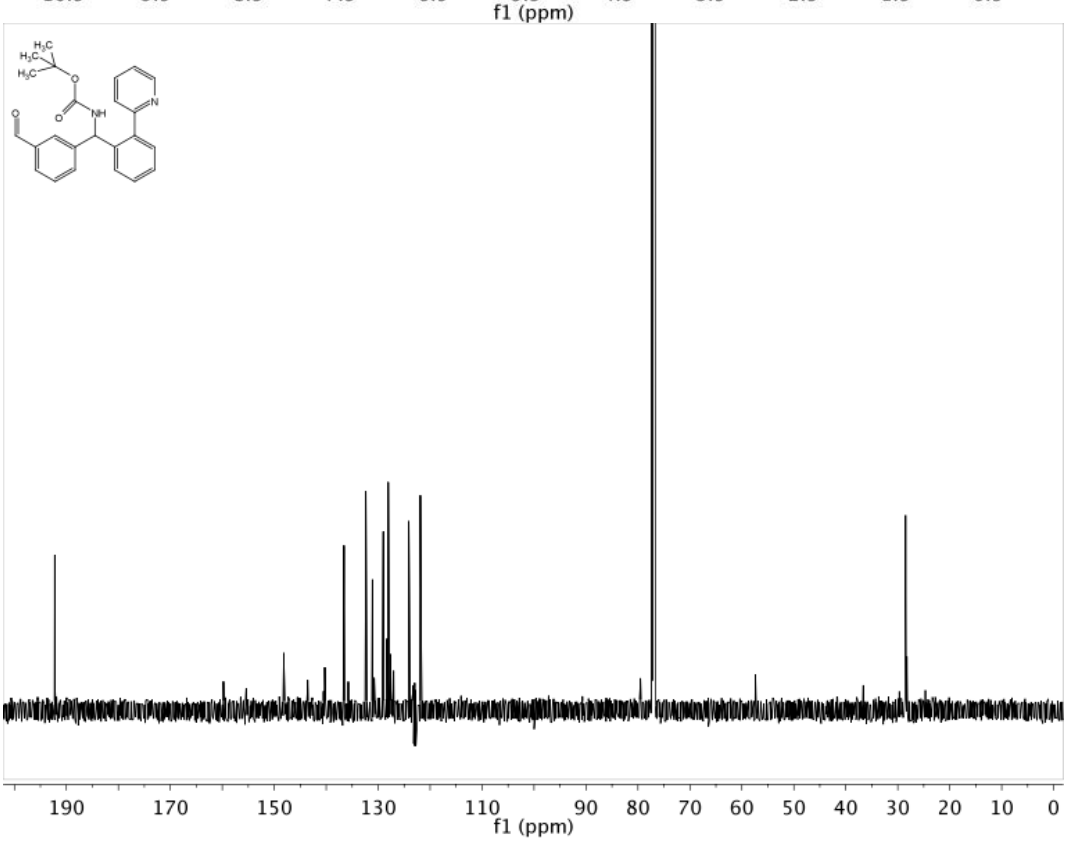
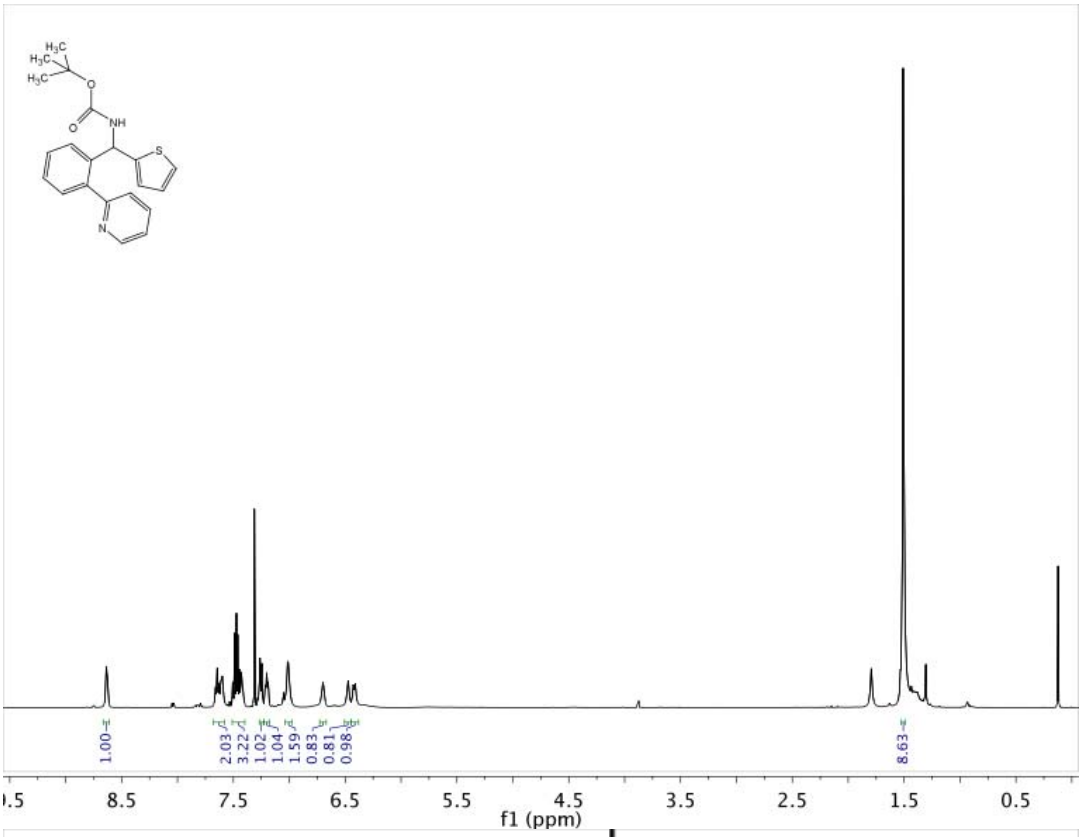
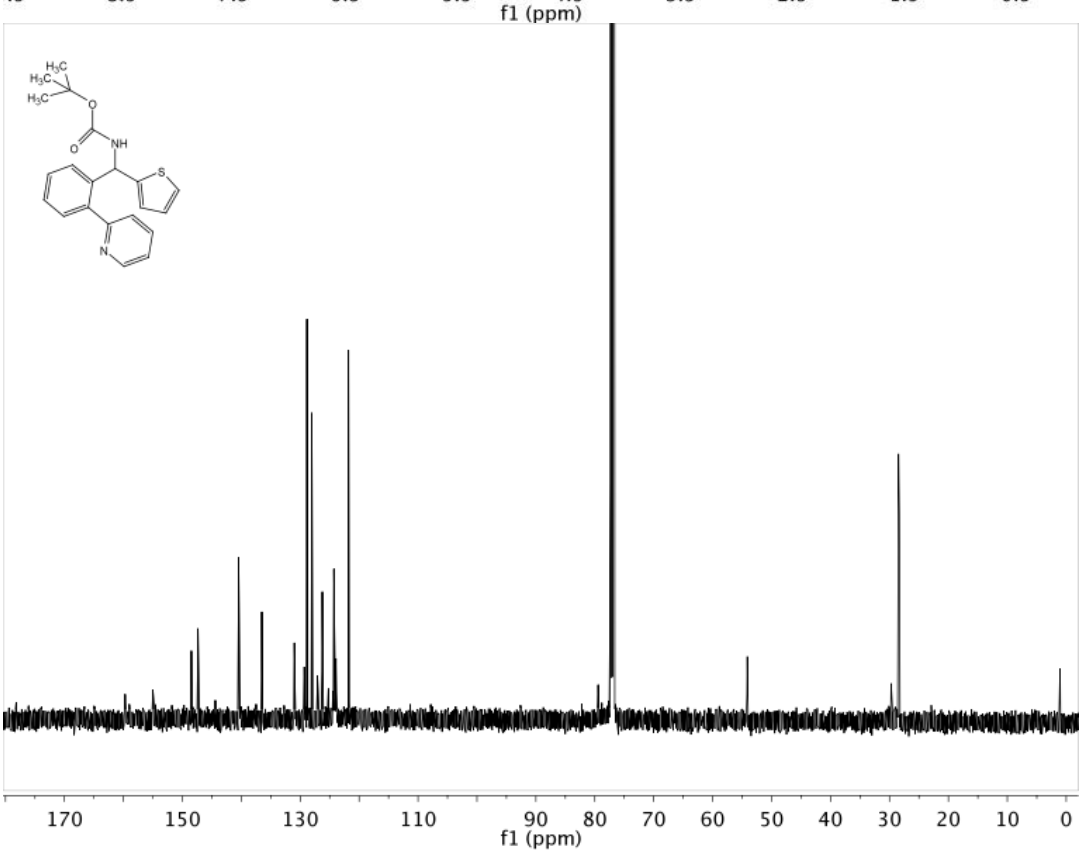
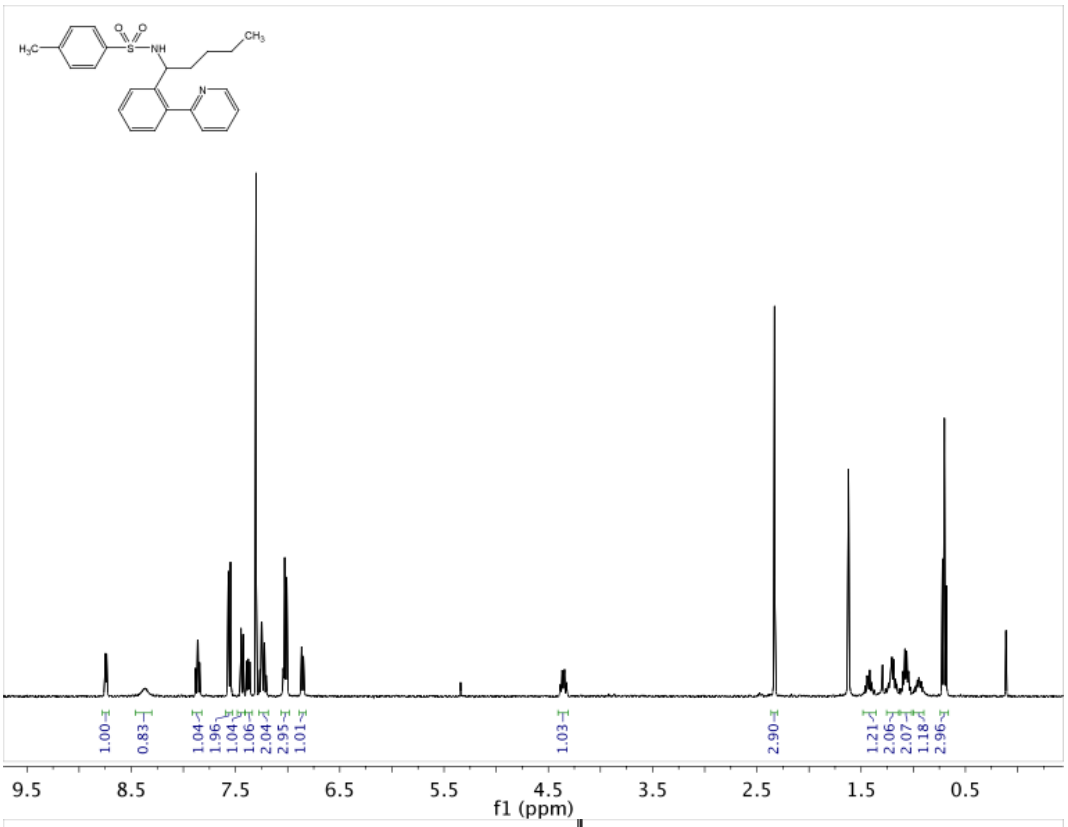
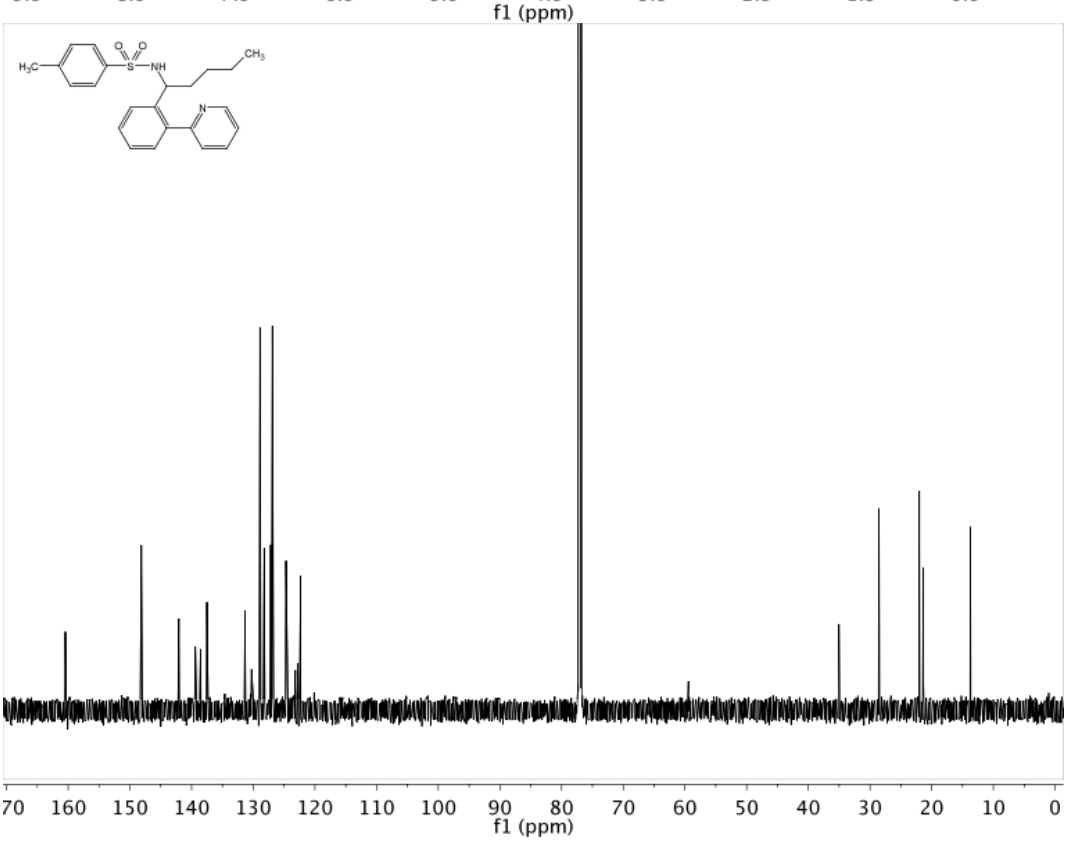
1.00
0.98
1.10
3.91
1.06
2.59
1.04
0.85
0.90
8.70
10.5
9.5
8.5
7.5
6.5
5.5
4.5
3.5
2.5
1.5
0.5
f1 (ppm)

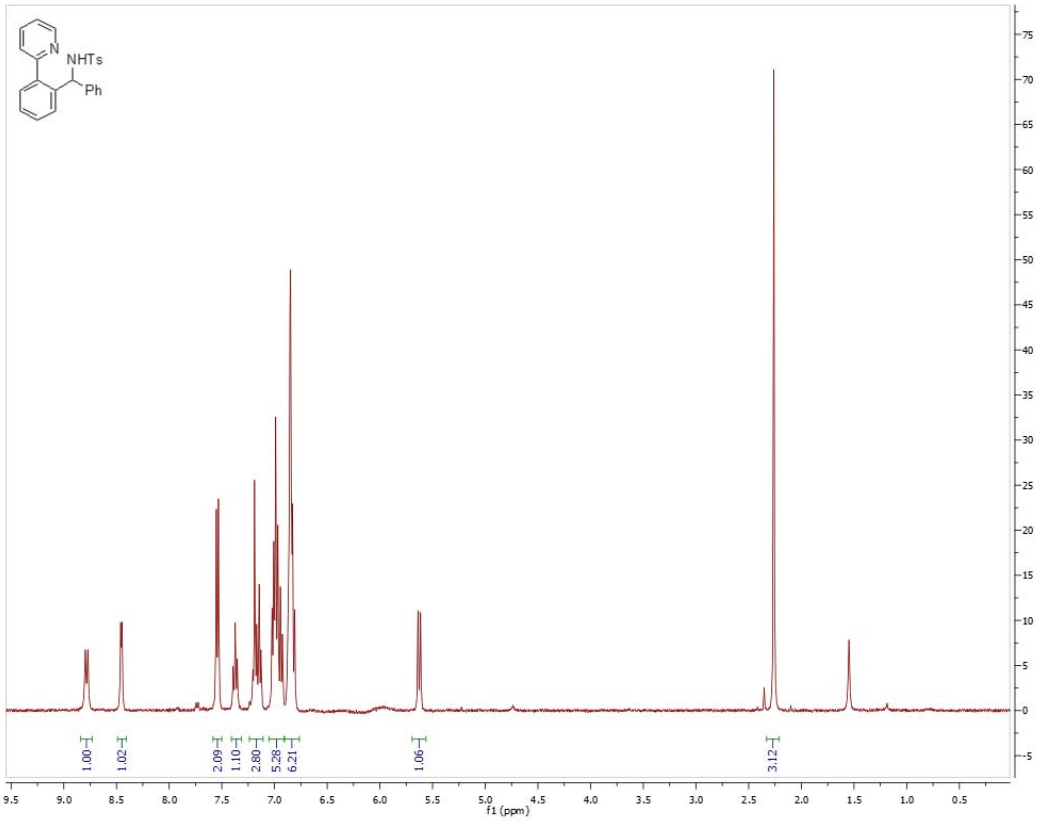

190
170
150
130
110
90
80
70
60
50
40
30
20
10
0
f1 (ppm)


1.00
2.03
3.22
1.02
1.04
1.59
0.83
0.81
0.98
8.63
8.5
7.5
6.5
5.5
4.5
3.5
2.5
1.5
0.5
f1 (ppm)


170
150
130
110
90
80
70
60
50
40
30
20
10
0
f1 (ppm)


1.00
0.83
1.04
1.96
1.04
1.06
2.04
2.95
1.01
1.03
2.90
1.21
2.06
2.07
1.18
2.96
9.5
8.5
7.5
6.5
5.5
4.5
3.5
2.5
1.5
0.5
f1 (ppm)


70
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
f1 (ppm)


NHTs
Ph
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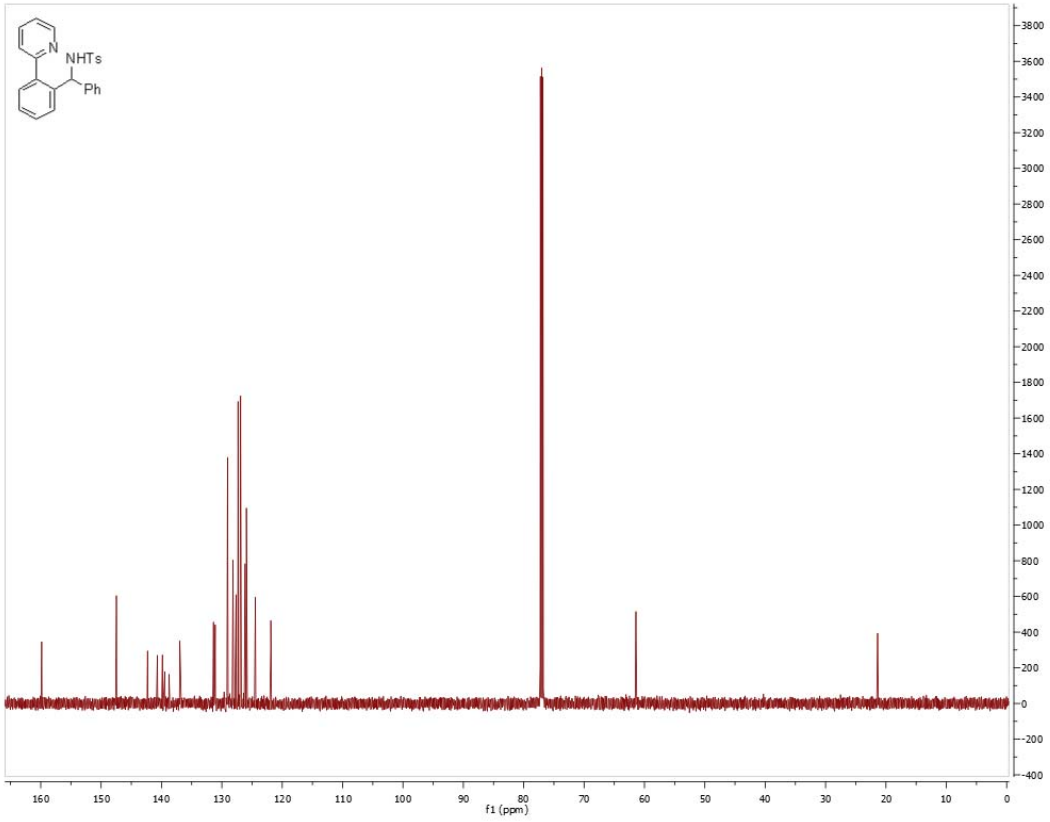
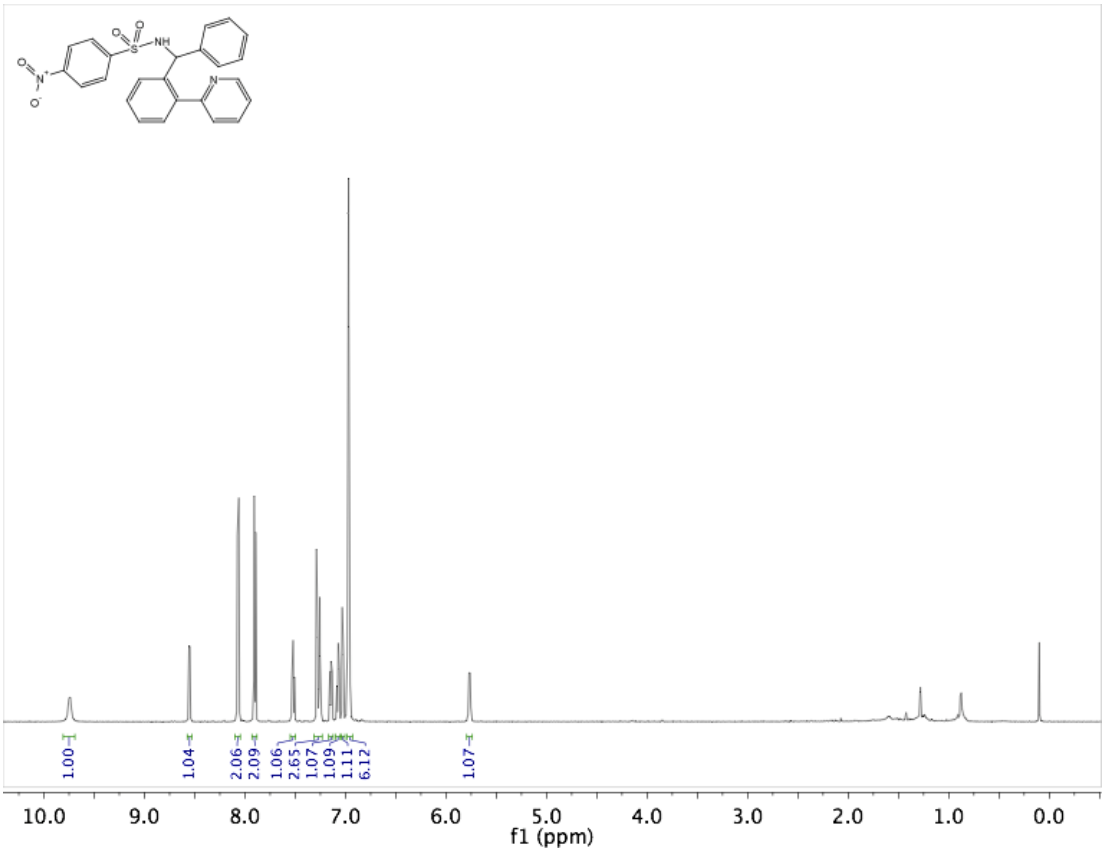
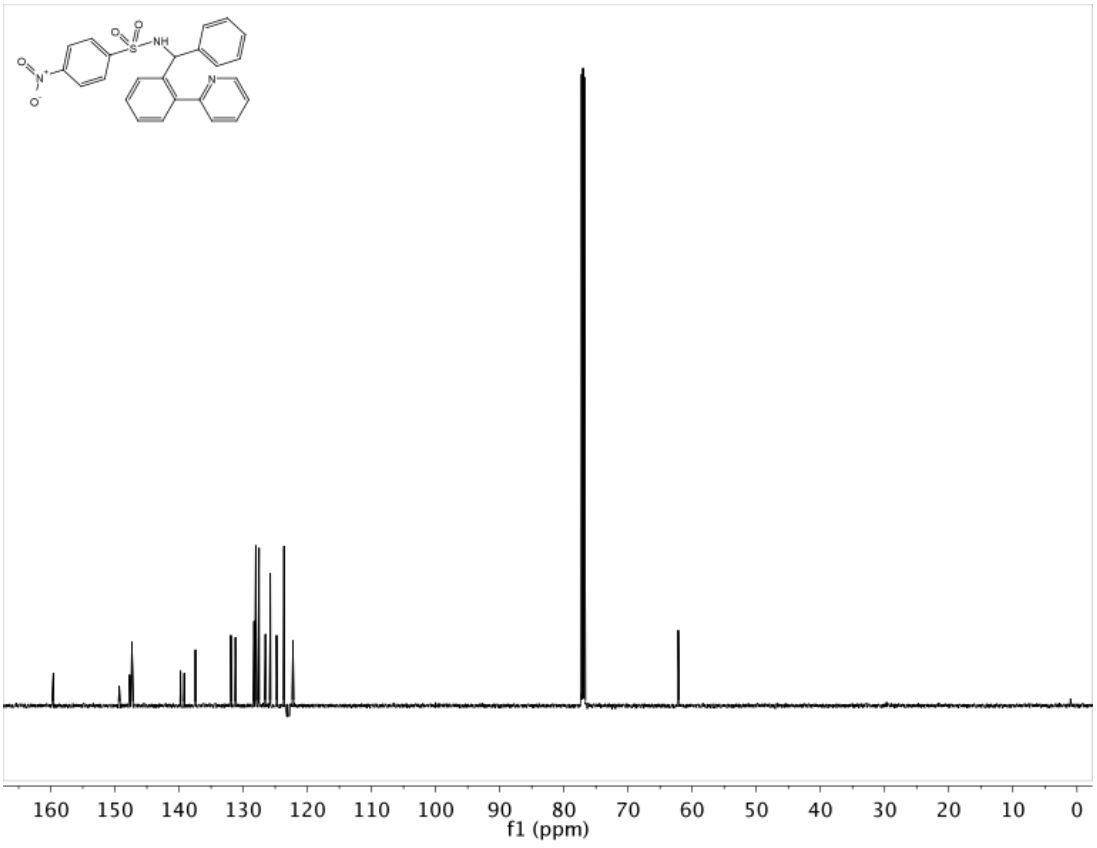
2.09
1.10
2.80
5.28
6.21
1.06
3.12
f1 (ppm)


NHTs
Ph
f1 (ppm)


1.00
1.04
2.06
2.09
1.06
2.65
1.07
1.09
1.11
6.12
1.07
10.0
9.0
8.0
7.0
6.0
5.0
4.0
3.0
2.0
1.0
0.0
f1 (ppm)


160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
f1 (ppm)