Electronic Supplementary Material (ESI) for Chemical Communications.

Supplementary Information

Supramolecular Bidentate Phosphine Ligand Scaffolds from Deconstructed Hamilton Receptors

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Experimental Section

General. All commercially-available reagents were used as received. Anhydrous, deoxygenated solvents were collected from a Pure Process Technologies solvent purification system. Triethylamine was dried and distilled over CaH_2 under nitrogen. Reactions were monitored using Merck F_{254} silica gel 60 TLC plates and visualized using UV light or a $KMnO_4$ stain. Reactions conducted under an inert atmosphere were performed by either using standard Schlenk techniques or a N_2-filled glove box. Chromatographic purification was performed using a Biotage automated flash chromatography purification system. ^1H and $^{13}C\{^1H\}$ NMR spectra were recorded at the reported frequencies, and chemical shifts are reported in ppm (δ) and referenced to the residual solvent resonance. $^{31}P\{^1H\}$ chemical shifts are referenced to H_3PO_4. The following naming conventions were used to describe NMR couplings: (s) singlet, (d) doublet, (t) triplet, (q) quartet, (dd) doublet of doublets, (m) multiplet, (b) broad.

General Procedure Binding Constant Determination. Binding studies were performed in $CDCl_3$ in duplicate or CD_3CN in triplicate for host molecules **4b-c** and were monitored by ^1H NMR spectroscopy at 25 °C. In a typical H_2O sat. $CDCl_3$ titration, 10.00 mL of a 1.0 mM barbiturate guest solution was prepared. The guest solution was then divided such that 600 μL was placed into an NMR tube and 1.50 mL was used to create a second solution containing 16 mM host. An initial spectrum of the guest was recorded using the following parameters: nt=16 and d_1=1s, after which aliquots (5-250 μL) of the host solution were added until the N-H resonance of barbiturate no longer shifted. The resultant curves were fit using a 1:1 model and the K_{assoc} obtained. In a typical CD_3CN titration, 3.0 mL of a 1.0 mM Pt host complex solution was prepared. The host solution was then divided such that 600 μL was placed into an NMR tube and the remaining 2.4 mL was used to create a second solution containing 30-60 mM synthetic

barbiturate guest. An initial spectrum of the host was recorded using the following parameters: nt=16 and d_1=1s, after which aliquots (5-250 µL) of the guest solution were added until the N-H resonance of the host no longer shifted. The resultant curves were fit using a 1:1 model and the K_{assoc} obtained.

General Procedure for Job Plot Analysis. Stoichiometric binding analysis was performed in H_2O-saturated $CDCl_3$ or 1% DMSO-d_6:$CDCl_3$ and was monitored by ^1H NMR spectroscopy at 25 °C. Total (host + guest) concentrations of 4.0 mM were used for all Job plots. For a typical Job plot, 2.0 mM stock solutions of guest **5a** and host **4b** were divided amongst 10 NMR tubes in 10 mol% increments to a total volume of 600 µL. A pure guest sample was also prepared. A d_1 of 2.0 s and nt=8 were during NMR data collection. Both the shift in host proximal N-H peak and guest N-H peaks were recorded.

Syntheses

***N*-(6-Aminopyridin-2-yl)-3,3-dimethylbutanamide (1)**. An oven dried flask containing 2,6-diaminopyridine (10.0 g, 91.6 mmol) was charged with anhydrous THF (300 mL) and cooled to 0 °C. A separate solution of 3,3-dimethylbutyryl chloride (6.0 mL, 43 mmol) in anhydrous THF (50 mL) was then added dropwise over 2.5 hours via addition funnel. The reaction mixture was warmed to room temperature and allowed to stir overnight. The crude reaction mixture was then filtered, concentrated via rotary evaporation, and purified via column chromatography (SiO_2, 1:1 EtOAc:Hex, R_f= 0.33) to yield a white solid (6.13 g, 69%). Spectroscopic data matched previously reported results.[1] ^1H NMR (500 MHz, $CDCl_3$) δ: 7.55 (d, *J* = 7.3 Hz, 2H), 7.44 (t, *J* = 7.9 Hz, 1H), 6.23 (d, *J* = 7.9 Hz, 1H), 4.28 (s, 2H), 2.19 (d, *J* = 2.0 Hz, 2H), 1.08 (s, 9H). $^{13}C\{^1H\}$ NMR (126 MHz, $CDCl_3$) δ: 170.29, 157.16, 149.93, 140.24, 104.31, 103.37, 51.89, 31.43, 29.94.

***N*-(6-(3,3-Dimethylbutanamido)pyridin-2-yl)-2-iodobenzamide (2a).** Excess thionyl chloride (3.0 mL, 41 mmol) was added to a scintillation vial containing 2-iodobenzoic acid (1.01 g, 4.05 mmol). Three drops of anhydrous DMF was added to the reaction mixture, and the reaction was heated to 65 °C, vented through a bubbler containing 1 M KOH, and stirred for 1.5 hours. The excess thionyl chloride was removed under vacuum. The resultant residue was dissolved in anhydrous THF (10 mL) and slowly added to a solution of **1** (0.763 g, 3.68 mmol) and anhydrous triethylamine (770 μL, 5.50 mmol) in THF (150 mL) at 0 °C. The resulting turbid mixture was warmed to room temperature and stirred overnight. The mixture was filtered, diluted with EtOAc, and washed with saturated $NaHCO_3$ (4 x 50 mL) and then with brine (2 x 50 mL). The organic layer was separated, dried over $MgSO_4$, filtered, and concentrated under reduced pressure. The crude product was purified via column chromatography (SiO_2, 1:3 EtOAc:Hex, $R_f = 0.18$) to yield a white solid (0.987 g, 62%).^{1}H NMR (500 MHz, $CDCl_3$) δ: 8.21 (s, 1H), 8.01 (d, *J* = 8.0 Hz, 1H), 7.97 (d, *J* = 8.1 Hz, 1H), 7.84 – 7.80 (m, 2H), 7.73 (t, *J* = 8.1 Hz, 1H), 7.60 – 7.57 (m, 3H), 2.23 (s, 2H), 1.10 (s, 9H). ^{13}C NMR (126 MHz, $CDCl_3$) δ: 170.42, 167.26, 149.76, 149.19, 141.53, 141.02, 140.33, 131.88, 128.45, 110.19, 109.81, 92.34, 51.76, 31.46, 29.90. (ES-TOF) *m/z:* $[M + H]^+$ Calcd for $C_{18}H_{21}IN_3O_2$, 438.0678; found 438.0673.

***N*-(6-(3,3-Dimethylbutanamido)pyridin-2-yl)-3-iodobenzamide (2b).** Excess thionyl chloride (3.0 mL, 41 mmol) was added to an oven-dried flask containing 3-iodobenzoic acid (0.503 g, 2.03 mmol). A drop of anhydrous DMF was added to the reaction mixture, and the mixture was heated to 65 °C, vented through a bubbler containing 1 M KOH, and stirred for 3 hours. The excess thionyl chloride was removed under vacuum. The resultant residue was dissolved in THF (10 mL) and slowly added to a solution of **1** (0.380 g, 1.83 mmol) and anhydrous triethylamine (380 μL, 2.72 mmol) in THF (50 mL) at 0 °C. The resulting turbid

mixture was warmed to room temperature and stirred overnight. The mixture was filtered, concentrated under reduced pressure, and purified via column chromatography (SiO_2, 1:2 EtOAc:Hex, R_f = 0.46) to yield a white solid (0.670 g, 84%). ^{1}H NMR (500 MHz, $CDCl_3$) δ: 8.22 (s, 1H), 8.15 (s, 1H), 8.03 (d, *J* = 8.0 Hz, 1H), 7.99 (d, *J* = 8.0 Hz, 1H), 7.90 (d, *J* = 7.9 Hz, 1H), 7.85 (d, *J* = 7.8 Hz, 1H), 7.76 (t, *J* = 8.0 Hz, 1H), 7.52 (s, 1H), 7.25 (t, *J* = 8.5 Hz, 1H), 2.25 (s, 2H), 1.12 (s, 9H). ^{13}C{^{1}H} NMR (126 MHz, $CDCl_3$) δ: 170.35, 163.90, 149.70, 149.37, 141.29, 141.14, 136.33, 136.25, 130.65, 126.44, 110.05, 109.74, 94.61, 52.00, 31.53, 29.96. (ES-TOF) *m/ z:* $[M + H]^+$ Calcd for $C_{18}H_{21}IN_3O_2$, 438.0678; found 438.0674.

***N*-(6-(3,3-Dimethylbutanamido)pyridin-2-yl)-4-iodobenzamide (2c).** Excess thionyl chloride (3.0 mL, 41.3 mmol) was added to scintillation vial containing 3-iodobenzoic acid (0.50 g, 2.0 mmol). A drop of anhydrous DMF was added to the reaction mixture, and the mixture was heated to 65 °C, vented through a bubbler containing 1 M KOH, and stirred for 4 hours. The excess thionyl chloride was removed under vacuum. The resultant residue was dissolved in anhydrous THF (10 mL) and slowly added to solution of **1** (0.381 g, 1.84 mmol) and anhydrous triethylamine (380 µL, 2.72 mmol) in THF (100 mL) at 0 °C. The resulting turbid mixture was warmed to room temperature and stirred overnight. The mixture was filtered, concentrated and purified via column chromatography (SiO_2, 1:3 EtOAc:Hex, R_f = 0.42) to yield a white solid (0.668 g, 83%).^{1}H NMR (500 MHz, $CDCl_3$) δ: 8.21 (s, 1H), 8.01 (d, *J* = 8.0 Hz, 1H), 7.97 (d, *J* = 8.1 Hz, 1H), 7.84 – 7.80 (m, 2H), 7.73 (t, *J* = 8.1 Hz, 1H), 7.60 – 7.57 (m, 3H), 2.23 (s, 2H), 1.10 (s, 9H). ^{13}C{^{1}H} NMR (126 MHz, $CDCl_3$) δ: 170.37, 164.74, 149.68, 149.41, 141.02, 138.20, 133.66, 128.74, 109.98, 109.73, 99.66, 51.85, 31.47, 29.92. (ES-TOF) *m/ z:* $[M + H]^+$ Calcd for $C_{18}H_{21}IN_3O_2$, 438.0678; found 438.0674.

***N*-(6-(3,3-Dimethylbutanamido)pyridin-2-yl)-2-(diphenylphosphino)benzamide (3a).** Reactants **2a** (0.152 g, 0.348 mmol), diphenylphosphine (0.081 g, 0.43 mmol), $Pd(OAc)_2$ (4.8 mg, 21 μmol), triethylamine (70 μL, 0.50 mmol, and CH_3CN (20 mL) were combined in a dry flask under N_2. The resulting dark red solution was heated to reflux and stirred overnight. The crude solution was loaded onto dry silica, in air, and purified via column chromatography (dry SiO_2, 1:3 EtOAc:Hex, $R_f = 0.29$) to yield an off white solid (0.095 g, 55%). ^{1}H NMR (500 MHz, $CDCl_3$) δ: 8.18 (bs, 1H), 7.91 (d, *J* = 8.1 Hz, 1H), 7.81 (d, *J* = 8.1 Hz, 1H), 7.72 (bm, 1H), 7.66 (t, *J* = 8.1 Hz, 1H), 7.43 (t, *J* = 7.4 Hz, 1H), 7.38 (t, *J* = 7.5 Hz, 1H), 7.31 (m, 9H), 7.05 (dd, *J* = 7.7, 4.1 Hz, 1H), 2.25 (s, 2H), 1.11 (s, 9H). ^{31}P{^{1}H} NMR (202 MHz, $CDCl_3$) δ: -9.32. ^{13}C{^{1}H} NMR (126 MHz, $CDCl_3$) δ: 166.95, 136.57, 136.49, 134.32, 134.09, 133.93, 130.89, 128.95, 128.86, 128.63, 128.57, 127.92, 127.89, 109.59, 109.51, 51.73, 31.38, 29.81.. HRMS (ESI-TOF) *m/z:* $[M + Na]^+$ Calcd for $C_{30}H_{30}N_3O_2PNa$, 518.1973; found 518.1972.

***N*-(6-(3,3-Dimethylbutanamido)pyridin-2-yl)-3-(diphenylphosphino)benzamide (3b).** Reactants **2b** (0.750 g, 1.72 mmol), diphenylphosphine (0.382 g, 2.05 mmol), $Pd(OAc)_2$ (22.7 mg, 101 μmol), triethylamine (340 μL, 2.44 mmol), and CH_3CN (35 mL) were combined in a dry flask under N_2. The resulting dark red solution was heated to reflux and stirred overnight. The crude solution was loaded onto dry silica, in air, and purified via column chromatography (dry SiO_2, 1:3 EtOAc:Hex, $R_f = 0.41$) to yield a white solid (0.690 g, 81%). ^{1}H NMR (500 MHz, $CDCl_3$) δ: 8.17 (s, 1H), 8.01 (d, *J* = 8.0 Hz, 1H), 7.96 (d, *J* = 8.0 Hz, 1H), 7.89 (d, *J* = 8.1 Hz, 1H), 7.85 (d, *J* = 7.5 Hz, 1H), 7.73 (t, *J* = 8.1 Hz, 1H), 7.54 (s, 1H), 7.45 (t, *J* = 7.4 Hz, 1H), 7.41 (t, *J* = 6.8 Hz, 1H), 7.39 – 7.28 (m, 10H), 2.24 (s, 2H), 1.11 (s, 9H). ^{13}C{^{1}H} NMR (126 MHz, $CDCl_3$) δ: 170.24, 165.14, 149.47 (d, *J* = 12.9 Hz), 140.87, 139.07 (d, *J* = 13.9 Hz), 137.06 (d, *J* = 13.9 Hz), 136.22 (d, *J* = 10.5 Hz), 134.44 (d, *J* = 7.7 Hz), 133.79 (d, *J* = 19.8 Hz), 132.23 (d, *J*

= 25.8 Hz), 129.15, 129.11, 129.07, 128.75 (d, *J* = 7.2 Hz), 127.58, 109.65 (d, *J* = 14.5 Hz), 51.80, 31.37, 29.82. $^{31}P\{^{1}H\}$ NMR (202 MHz, $CDCl_3$) δ: -5.23. HRMS (ESI-TOF) *m/z:* $[M + Na]^+$ Calcd for $C_{30}H_{30}N_3O_2PNa$, 518.1973; found 518.1964.

***N*-(6-(3,3-Dimethylbutanamido)pyridin-2-yl)-4-(diphenylphosphino)benzamide (3c).** Reactants **2c** (0.495 g, 1.13 mmol), diphenylphosphine (0.241 g, 1.30 mmol), $Pd(OAc)_2$ (15.7 mg, 69.9 μmol), triethylamine (230 μL, 1.65 mmol), and CH_3CN (30 mL) were combined in a dry flask under N_2. The resulting dark red solution was heated to reflux and stirred overnight. The crude solution was loaded onto dry silica, in air, and purified via column chromatography (dry SiO_2, 1:3 EtOAc:Hex, R_f = 0.32) to yield a white solid (0.437 g, 78%). ^{1}H NMR (500 MHz, $CDCl_3$) δ: 8.20 (s, 1H), 8.05 (d, *J* = 8.0 Hz, 1H), 7.97 (d, *J* = 8.0 Hz, 1H), 7.84 – 7.80 (m, 2H), 7.77 (s, 1H), 7.42 – 7.29 (m, 12H), 2.25 (s, 2H), 1.11 (s, 9H). $^{13}C\{^{1}H\}$ NMR (126 MHz, $CDCl_3$) δ: 170.33, 165.20, 149.63 (d, *J* = 6.8 Hz), 143.63 (d, *J* = 14.6 Hz), 141.05, 136.20 (d, *J* = 10.6 Hz), 134.17, 134.01, 133.88, 133.73, 129.36, 128.87 (d, *J* = 7.3 Hz), 127.05 (d, *J* = 6.4 Hz), 109.79 (d, *J* = 13.2 Hz), 51.95, 31.50, 29.94. $^{31}P\{^{1}H\}$ NMR (202 MHz, $CDCl_3$) δ: -5.26. HRMS (ESI-TOF) *m/z:* $[M + Na]^+$ Calcd for $C_{30}H_{30}N_3O_2PNa$, 518.1973; found 518.1957.

5,7-dihydro-1'H-Spiro[dibenzo[a,c][7]annulene-6,5'-pyrimidine]-2',4',6'(3'H)-trione (5a). Barbituric acid (76.9 mg, 6.00 mmol), 2,2'-bis(bromomethyl)-1,1'-biphenyl (198 mg, 5.83 mmol), triethylamine (170 μL , 1.23 mmol), and DMF (3 mL) were added to a scintillation vial. The reaction mixture was stirred overnight at room temperature. The reaction mixture changed from a clear and colorless solution to cloudy and white and then finally to clear and yellow solution upon completion of the reaction with some precipitate present. The DMF was removed under vacuum with gentle heating. The resultant residue was purified using column chromatography (SiO_2, 3:1 EtOAc:hexanes, R_f = 0.58) to afford an white solid (123 mg, 69%).

^{1}H NMR (500 MHz, DMSO-d_6) δ: 11.12 (s, 2H), 7.42 – 7.35 (m, 4H), 7.29 (ddd, *J* = 7.5, 5.6, 3.1 Hz, 2H), 7.21 (d, *J* = 7.4 Hz, 2H), 2.93 (bd, *J* = 5.5 Hz, 4H). ^{13}C{^{1}H} NMR (126 MHz, DMSO-d_6) δ: 172.24, 150.38, 139.60, 134.95, 131.09, 127.87, 127.55, 127.25, 61.71.

General Procedure for the synthesis of cis-PtL$_2$Cl$_2$ complexes. In an inert atmosphere, a solution of **3b** (66.2 mg, 134 μmol) in CH_2Cl_2 (2 mL) was added dropwise to a stirring solution of Pt(COD)Cl_2 (25.3 mg, 67.6 μmol) in CH_2Cl_2 (1 mL). The reaction was stirred at room temperature for one hour, after which the solvent was removed under vacuum. The resulting solids were triturated three times with hexanes, in air, and filtered to obtain **4b** as an off white solid (63 mg, 75%). ^{1}H NMR (500 MHz, $CDCl_3$) δ: 8.39 (s, 2H), 8.03 (d, *J* = 11.0 Hz, 2H), 7.99 (d, *J* = 8.1 Hz, 2H), 7.95 (d, *J* = 8.0 Hz, 2H), 7.89 (s, 2H), 7.79 (d, *J* = 7.8 Hz, 2H), 7.73 (t, *J* = 8.1 Hz, 2H), 7.60 – 7.46 (m, 8H), 7.43 – 7.31 (m, 6H), 7.30 – 7.23 (m, 2H), 7.21 (td, *J* = 7.9, 2.1 Hz, 8H), 2.26 (s, 4H), 1.10 (s, 18H). ^{31}P{^{1}H} NMR (202 MHz, $CDCl_3$) δ: 14.55.

Compound 4c. (white solid, 85 mg, 85%) ^{1}H NMR (500 MHz, $CDCl_3$) δ 8.40 (s, 2H), 7.99 (t, *J* = 7.5 Hz, 4H), 7.75 (t, *J* = 8.1 Hz, 3H), 7.62 (d, *J* = 8.1 Hz, 5H), 7.55 (dd, *J* = 11.4, 7.7 Hz, 8H), 7.48 – 7.35 (m, 8H), 7.25 – 7.18 (m, 8H), 2.23 (s, 4H), 1.09 (s, 18H). ^{31}P{^{1}H} NMR (202 MHz, $CDCl_3$) δ: 14.32

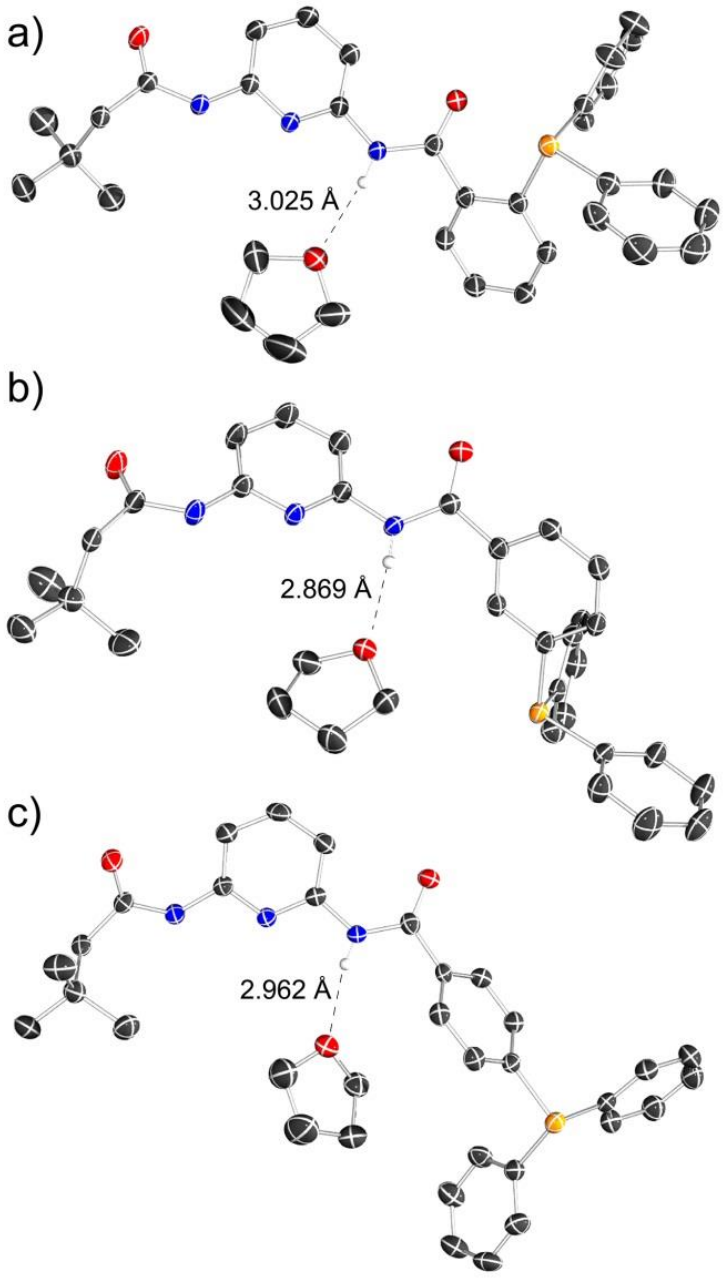


Figure S1. ORTEP representation of a) *o*- isomer, **3a**, b) *m*-isomer, **3b**, c) *p*-isomer, **3c**, co-crystallized with a molecule of THF. Thermal ellipsoids drawn at 50% probability with non-hydrogen bonding hydrogens omitted for clarity.

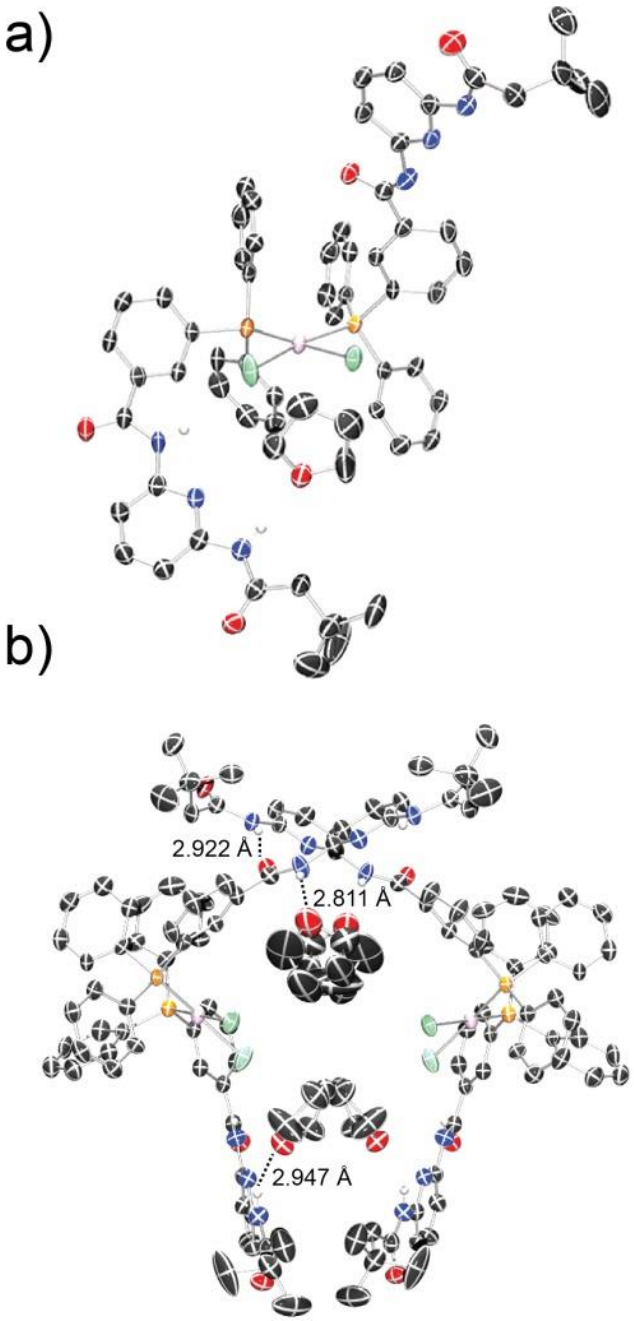


Figure 4. ORTEP representations of **4b** with thermal ellipsoids drawn at 50% probability. a) Structure viewed face-on with one molecule of THF shown and non-hydrogen bonding hydrogens omitted for clarity. b) Dimeric form of structure showing intra- and inter-molecular hydrogen bonds with non-hydrogen bonding hydrogens omitted for clarity.

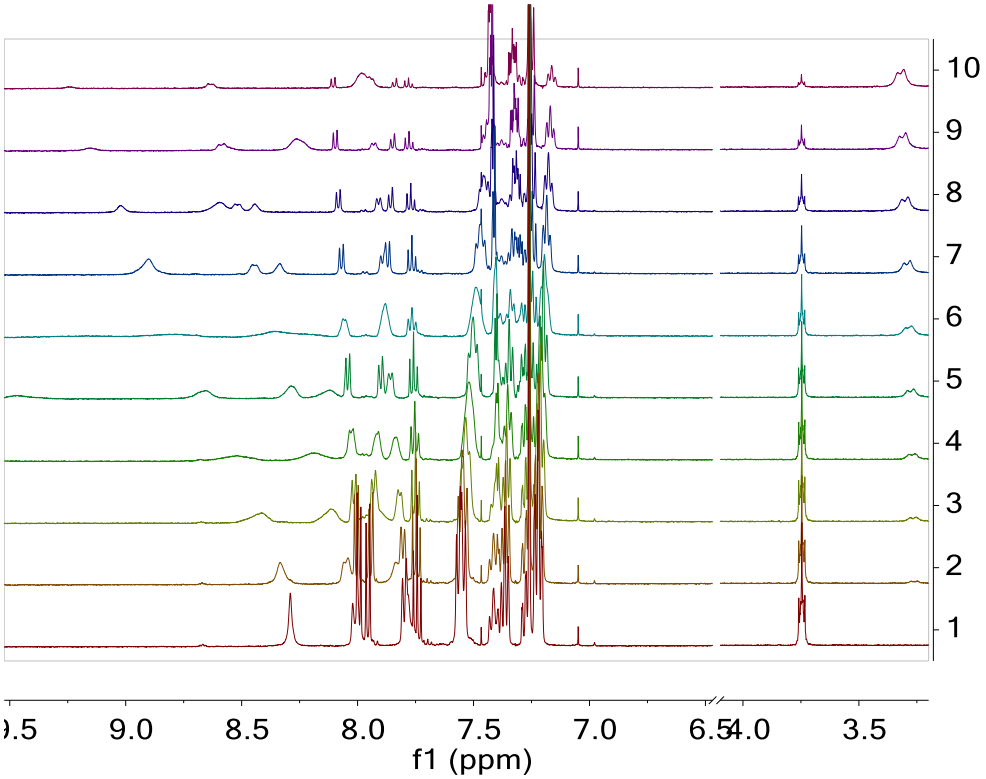


Figure S3. ^{1}H NMR data for Job Plot of **4b** and **5a** in $CDCl_3$

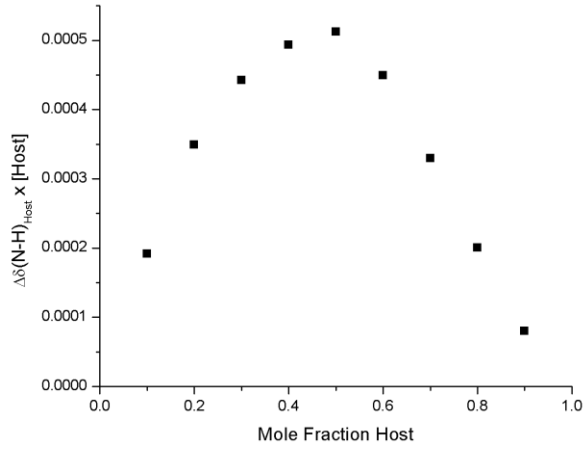


Figure S4. Job Plot of **4b** and **5a** in $CDCl_3$.

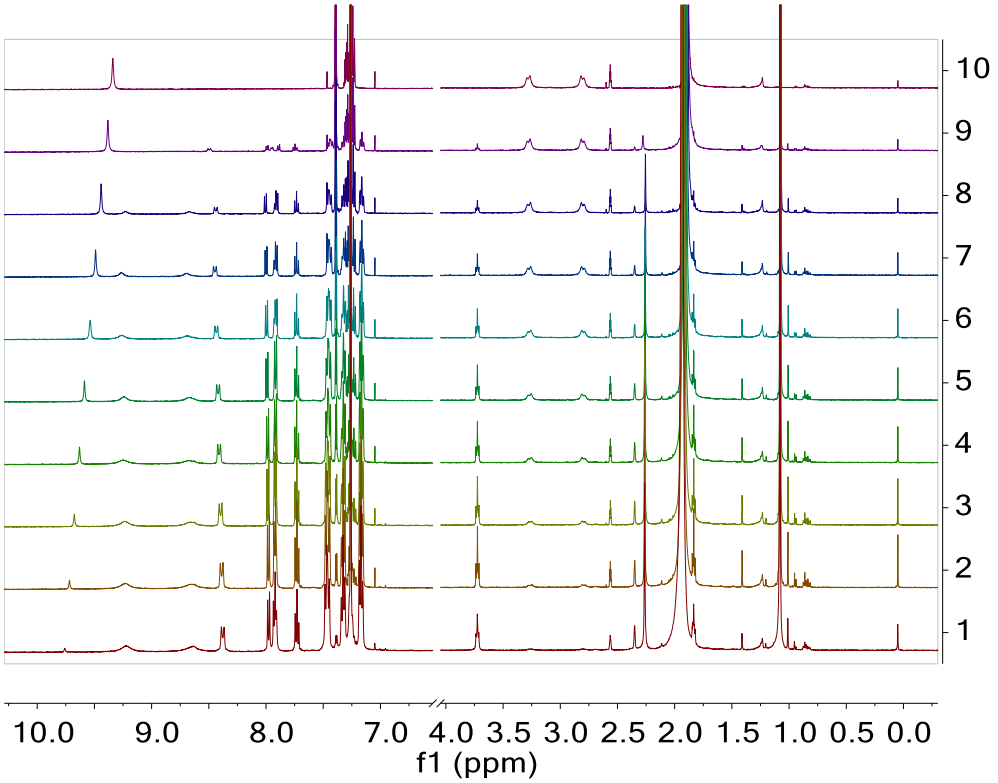


Figure S5. ^{1}H NMR data for Job Plot of **4b** and **5a** in 1% DMSO-d_6:$CDCl_3$.

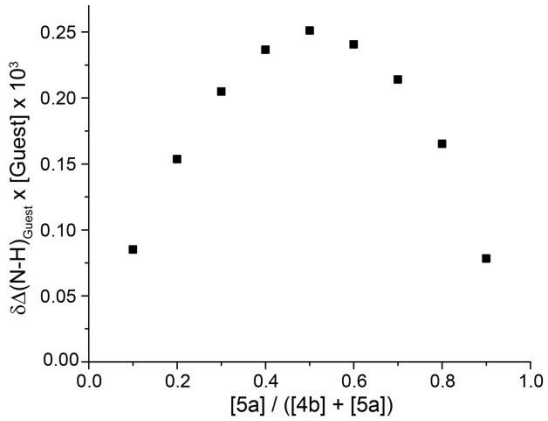


Figure S6. Job Plot of **4b** and **5a** in 1% DMSO-d_6:$CDCl_3$

NMR Spectra.

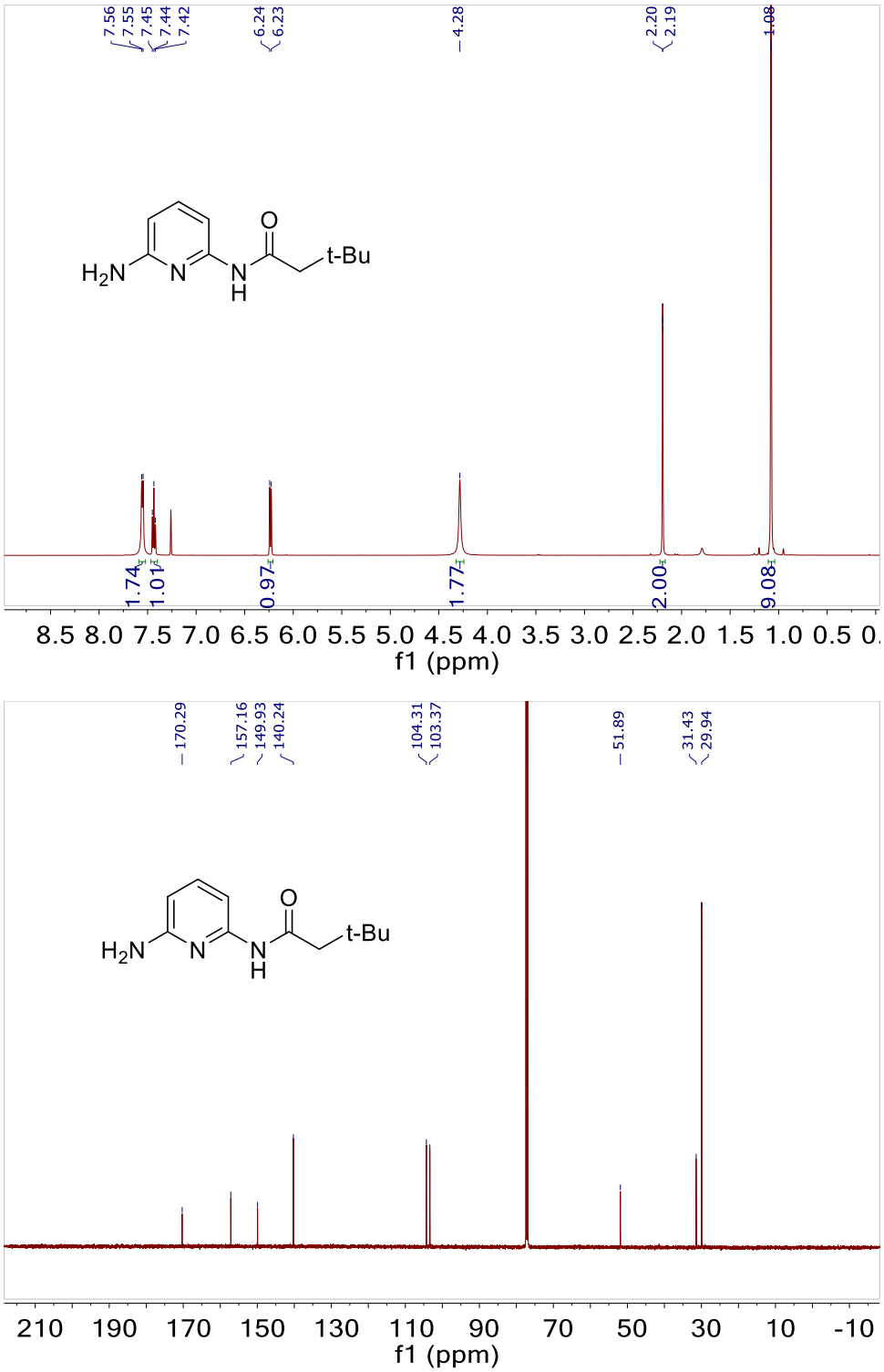


Figure S4. ^{1}H (500 MHz) and $^{13}C\{^{1}H\}$ (126 MHz) NMR spectra of **1** in $CDCl_3$.

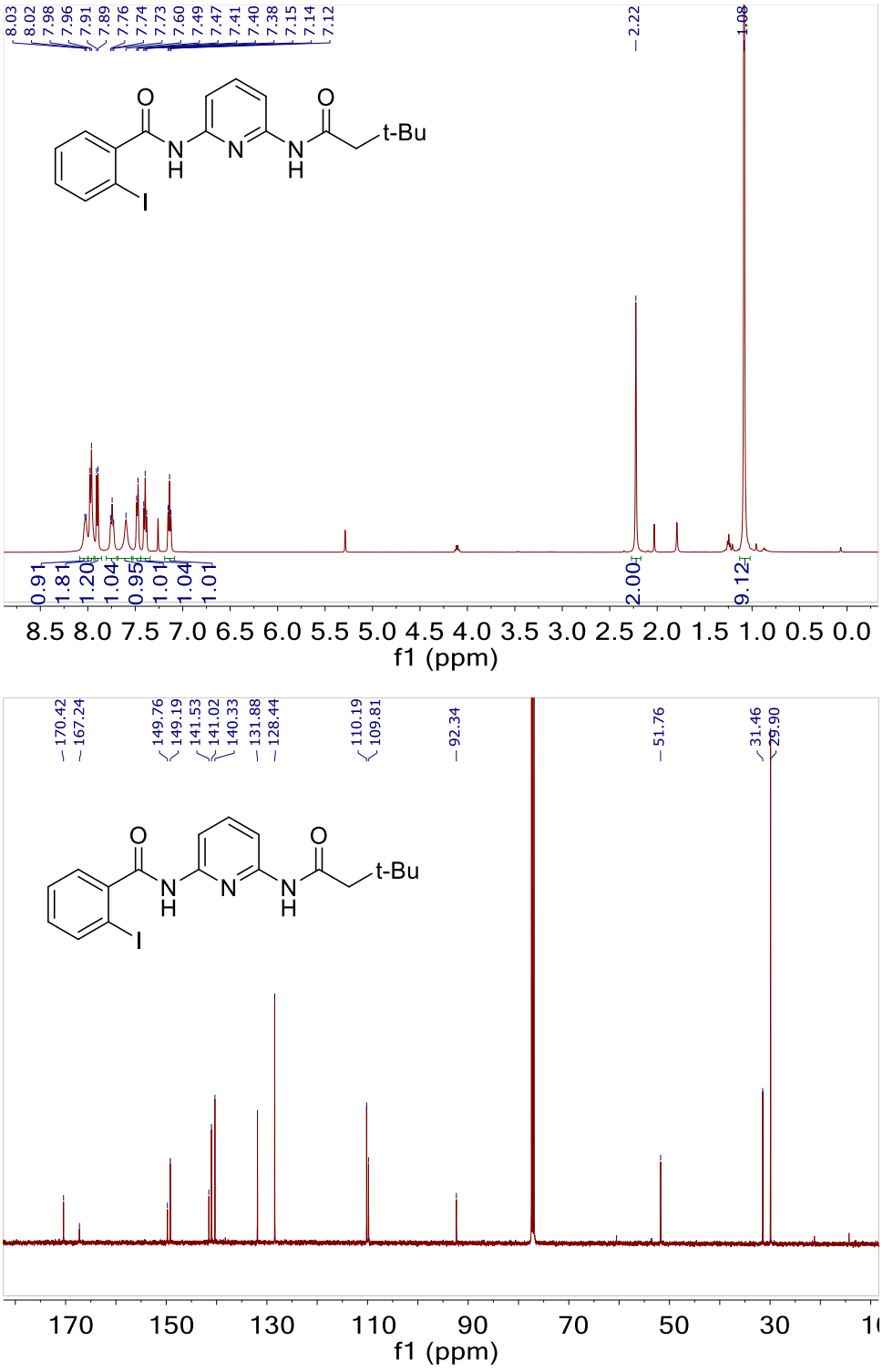


Figure S5. ^{1}H (500 MHz) and $^{13}C\{^{1}H\}$ (126 MHz) NMR spectra of **2a** in $CDCl_3$.

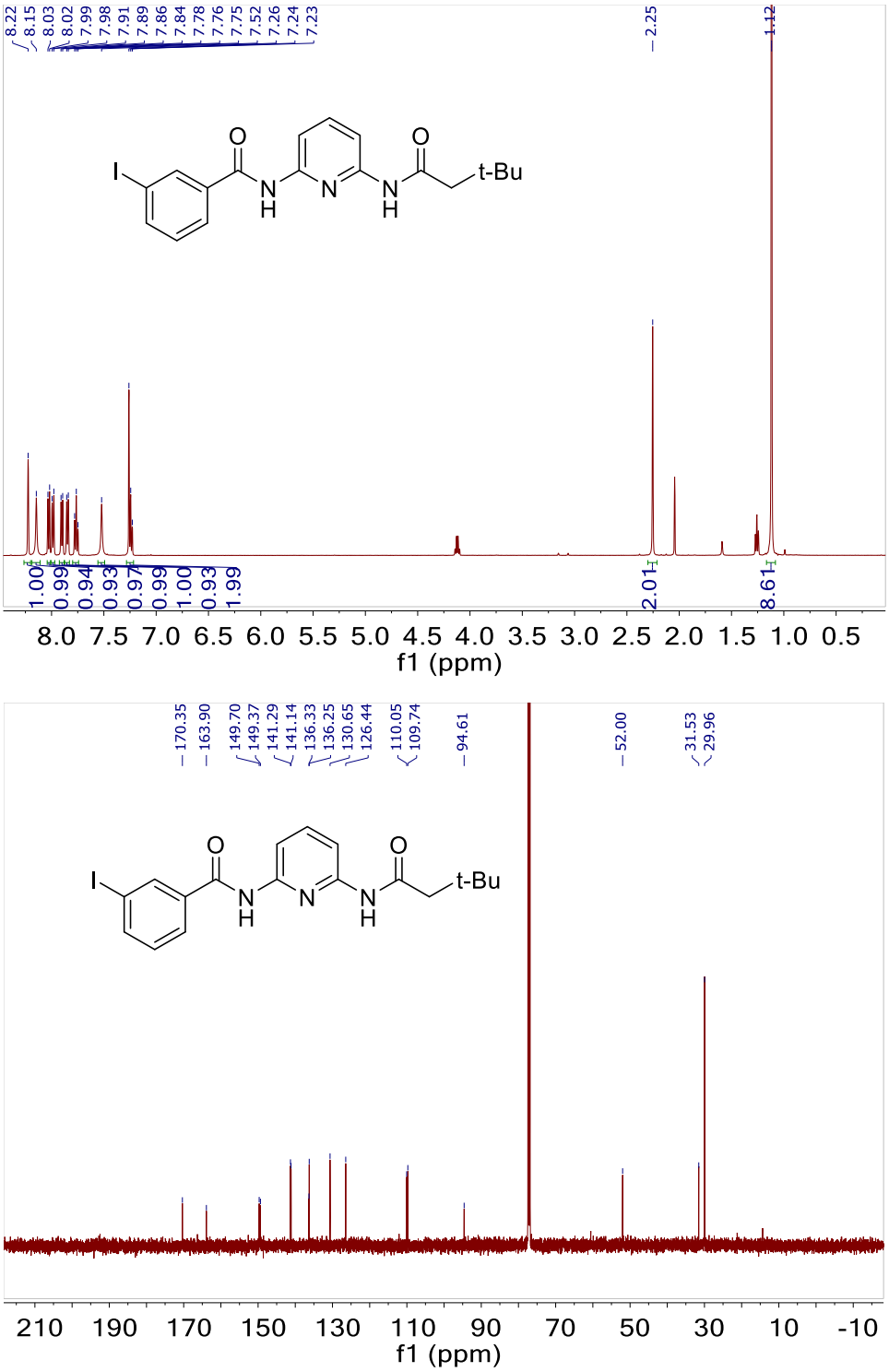


Figure S6. ^{1}H (500 MHz) and $^{13}C\{^{1}H\}$ (126 MHz) NMR spectra of **2b** in $CDCl_3$.

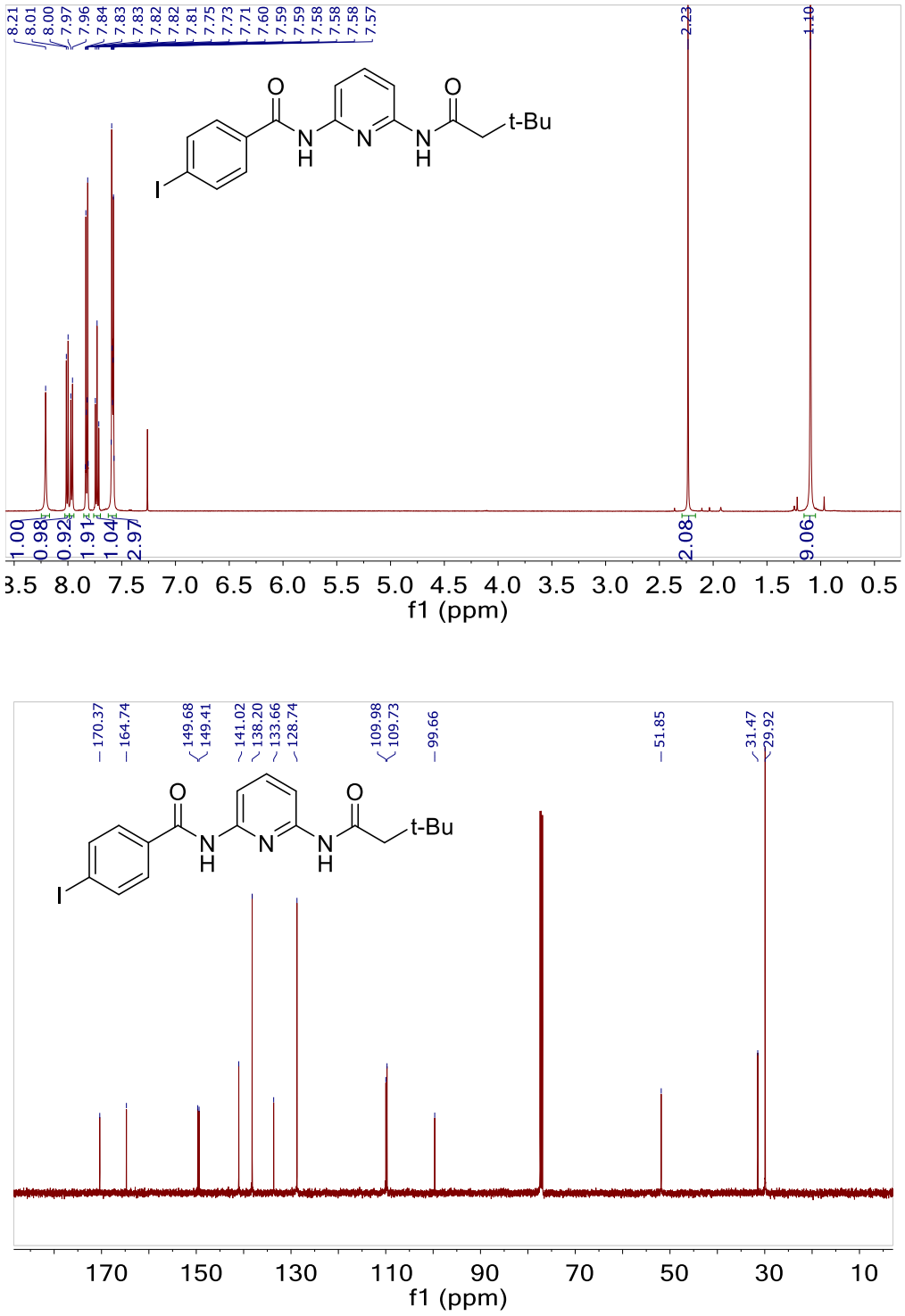
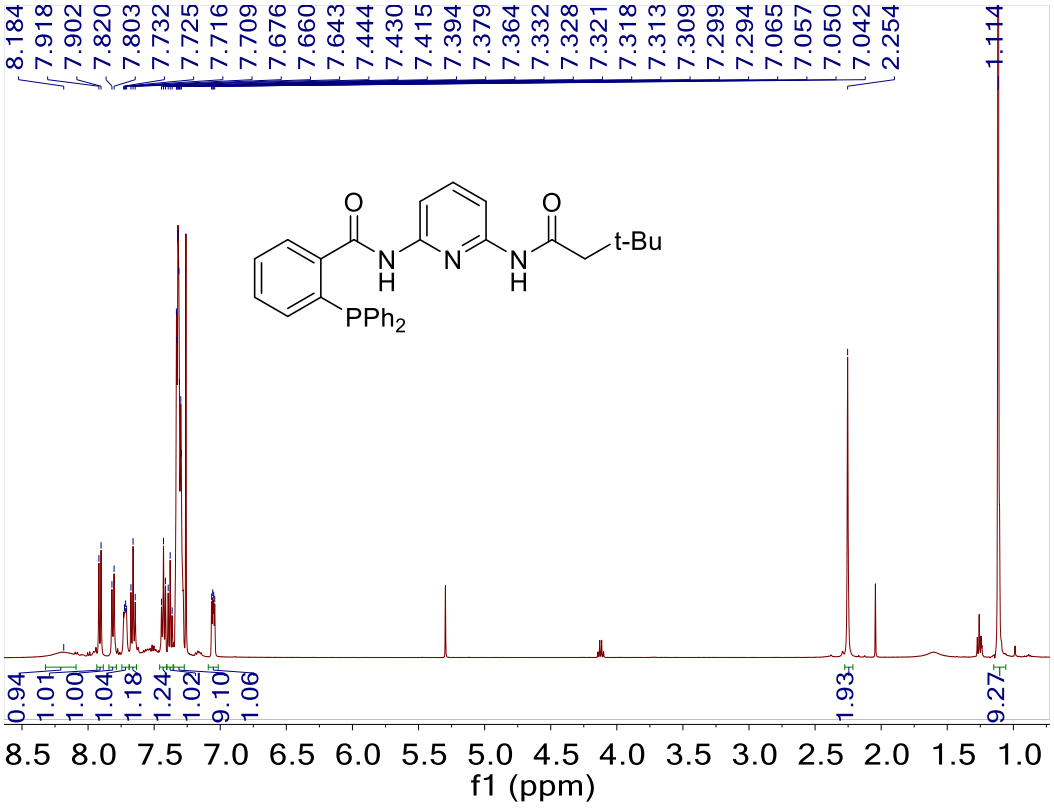


Figure S7. ^{1}H (500 MHz) and $^{13}C\{^{1}H\}$ (126 MHz) NMR spectra of **2c** in $CDCl_3$.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7.065
7.057
7.050
7.042
2.254
1.114
O
N
H
N
O
N
H
t-Bu
PPh2
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1.01
1.00
1.04
1.18
1.24
1.02
9.10
1.06
1.93
9.27
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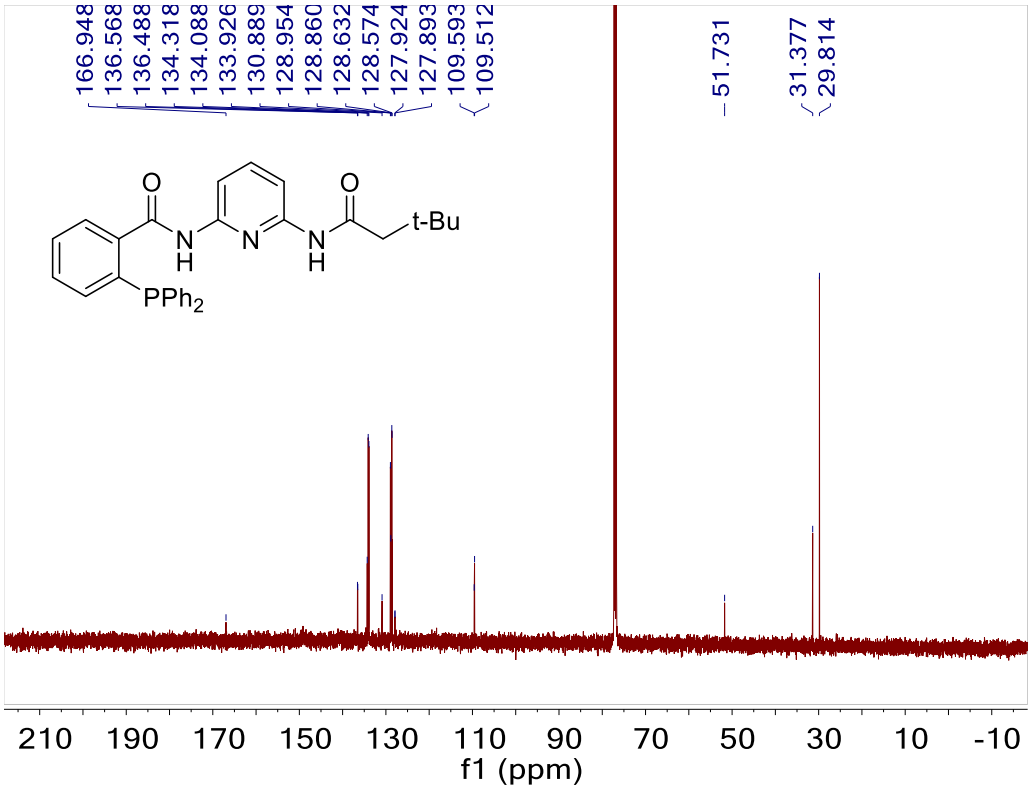
f1 (ppm)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f1 (ppm)

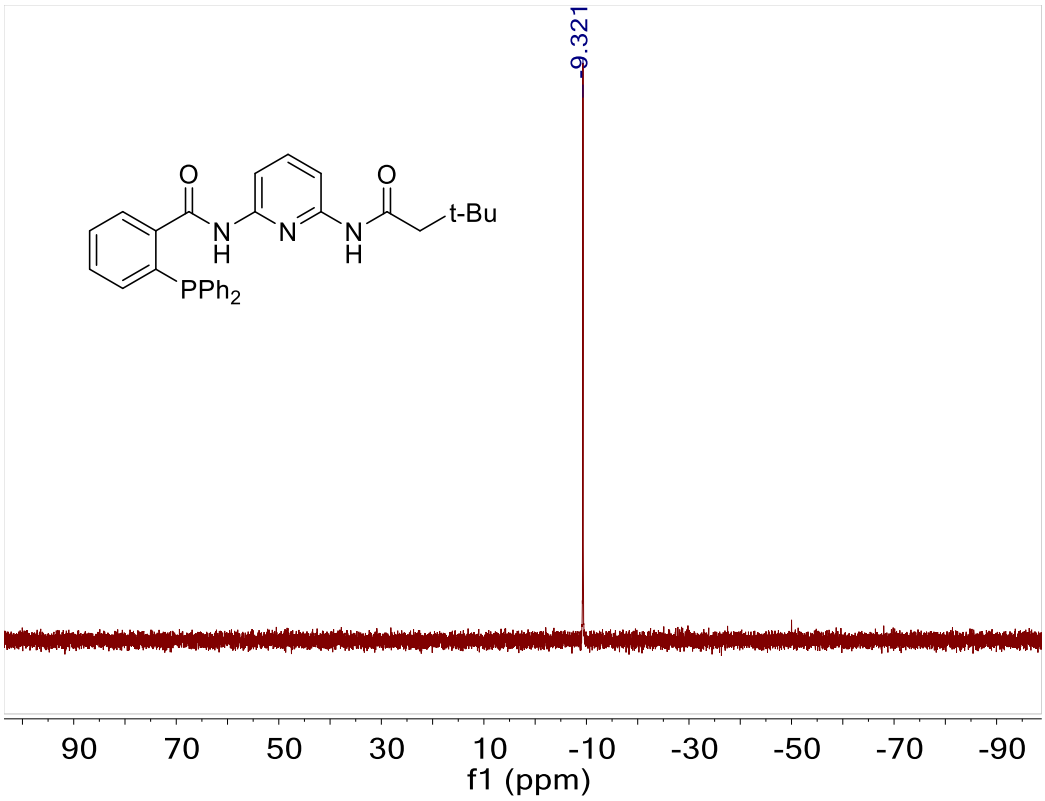
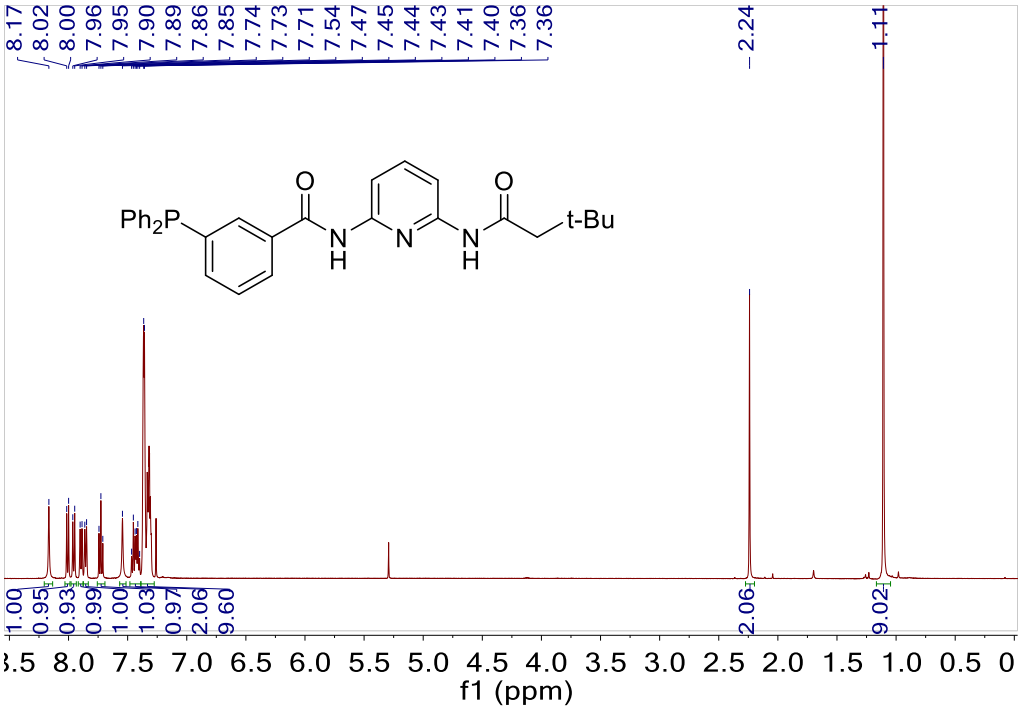


Figure S8. ^{1}H (500 MHz), $^{13}C\{^{1}H\}$ NMR (126 MHz), and $^{31}P\{^{1}H\}$ (202 MHz) NMR spectra of **3a** in $CDCl_3$



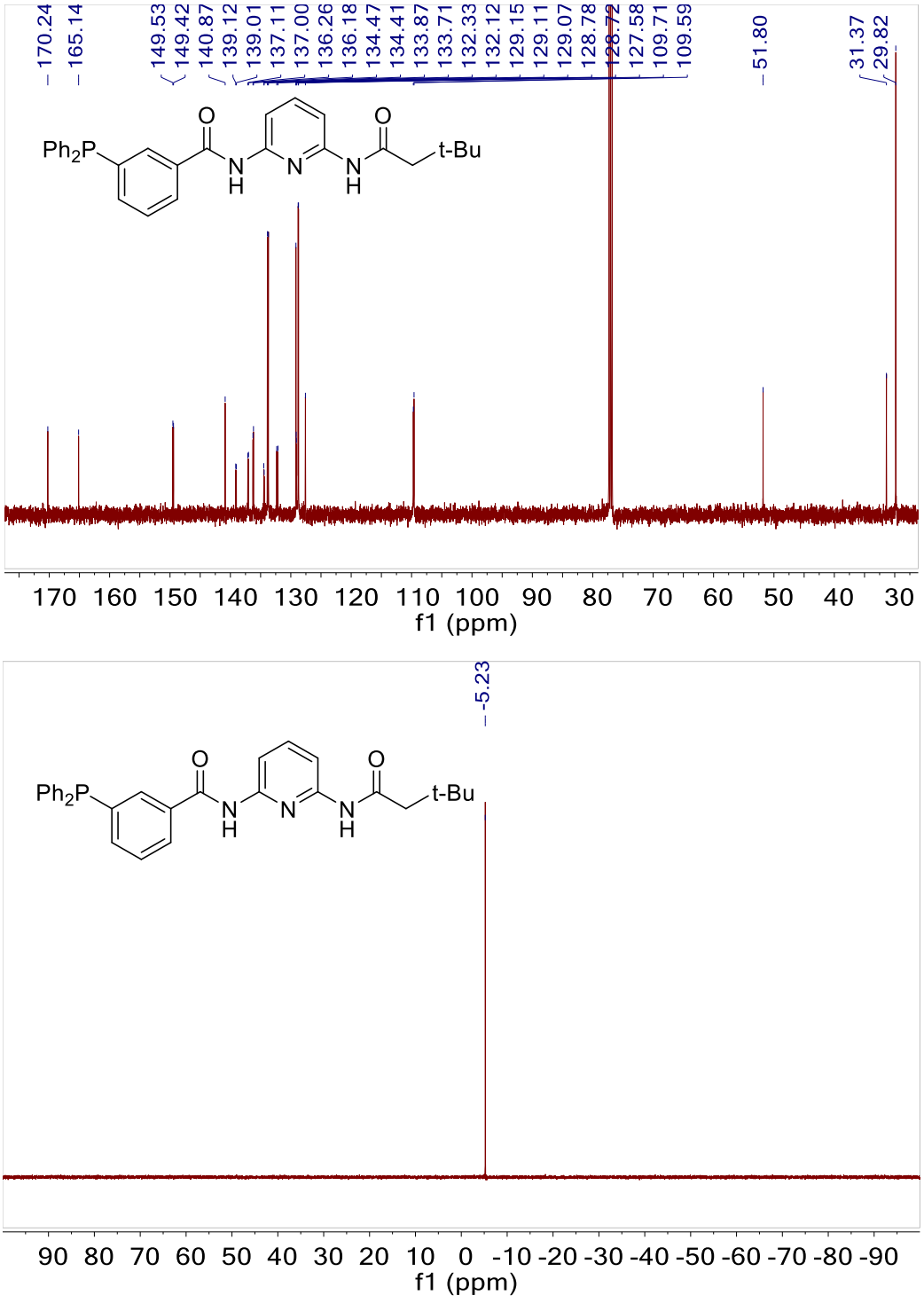
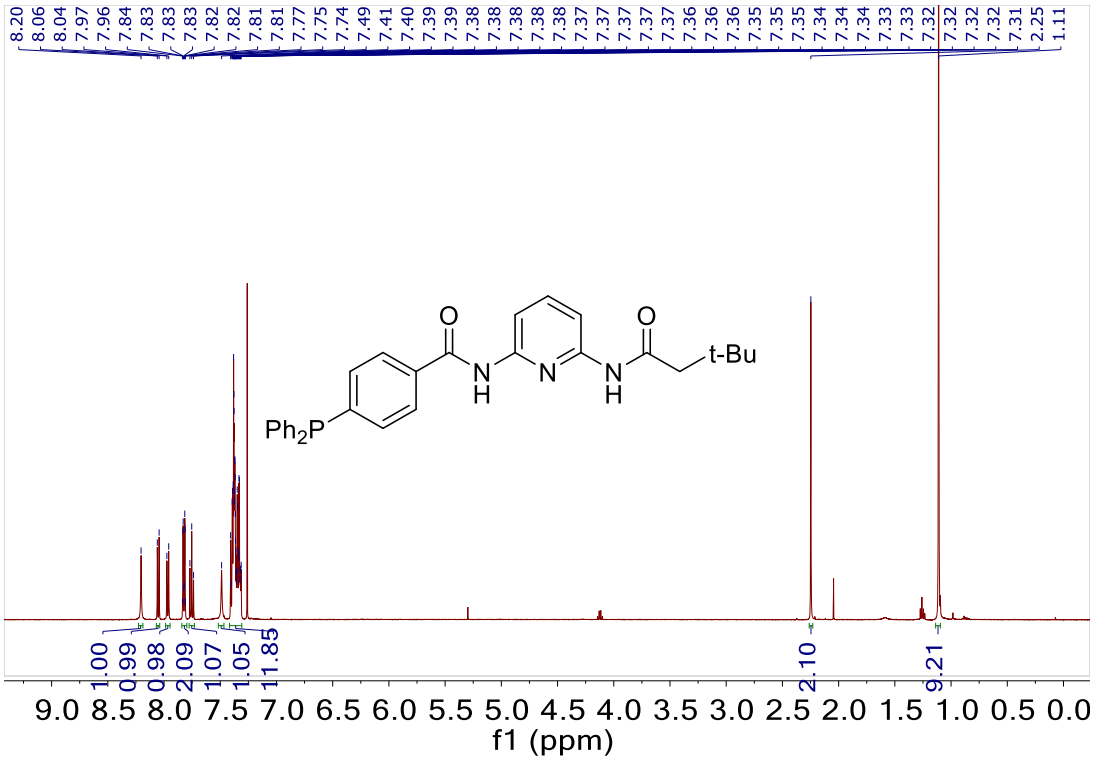
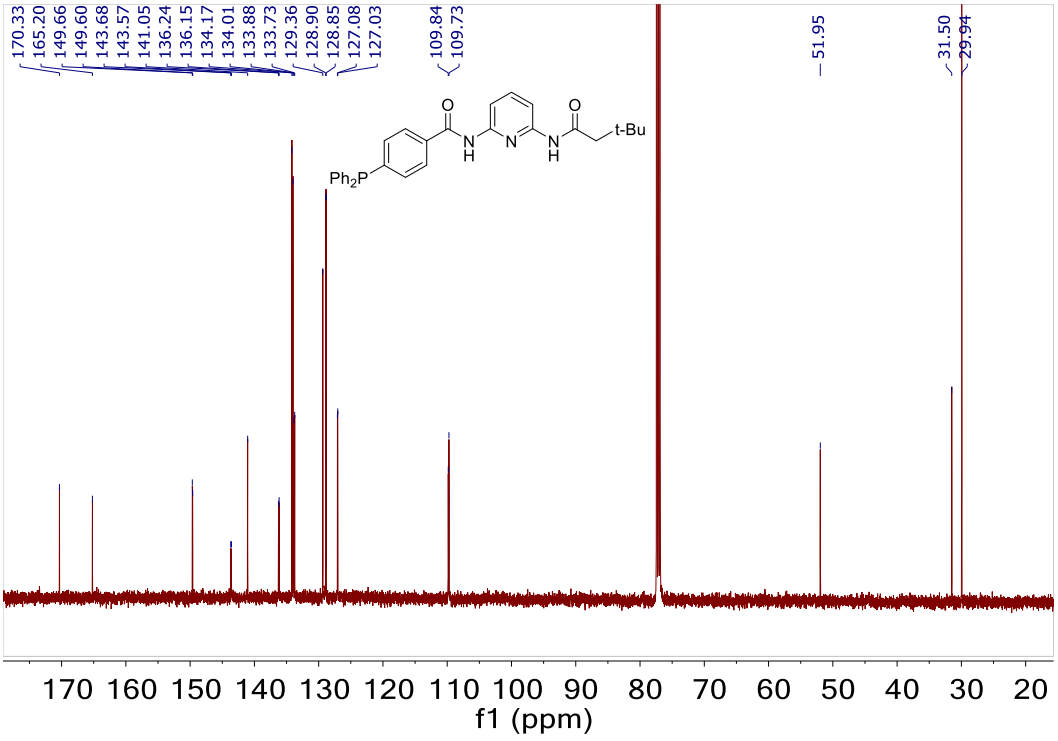


Figure S9. ^{1}H (500 MHz), $^{13}C\{^{1}H\}$ NMR (126 MHz), and $^{31}P\{^{1}H\}$ (202 MHz) NMR spectra of **3b** in $CDCl_3$


8.20 8.06 8.04 7.97 7.96 7.84 7.83 7.83 7.83 7.82 7.82 7.81 7.81 7.77 7.75 7.74 7.49 7.41 7.40 7.39 7.39 7.38 7.38 7.38 7.38 7.38 7.37 7.37 7.37 7.37 7.37 7.36 7.36 7.36 7.35 7.35 7.35 7.34 7.34 7.34 7.33 7.33 7.32 7.32 7.32 7.32 7.31 2.25 1.11
O
N
H
N
N
H
O
t-Bu
Ph2P
1.00 0.99 0.98 2.09 1.07 1.05 11.85 2.10 9.21
9.0 8.5 8.0 7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 0.5 0.0
f1 (ppm)


170.33 165.20 149.66 149.60 143.68 143.57 141.05 136.24 136.15 134.17 134.01 133.88 133.73 129.36 128.90 128.85 127.08 127.03 109.84 109.73 51.95 31.50 29.94
O
N
H
N
N
H
O
t-Bu
Ph2P
170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20
f1 (ppm)

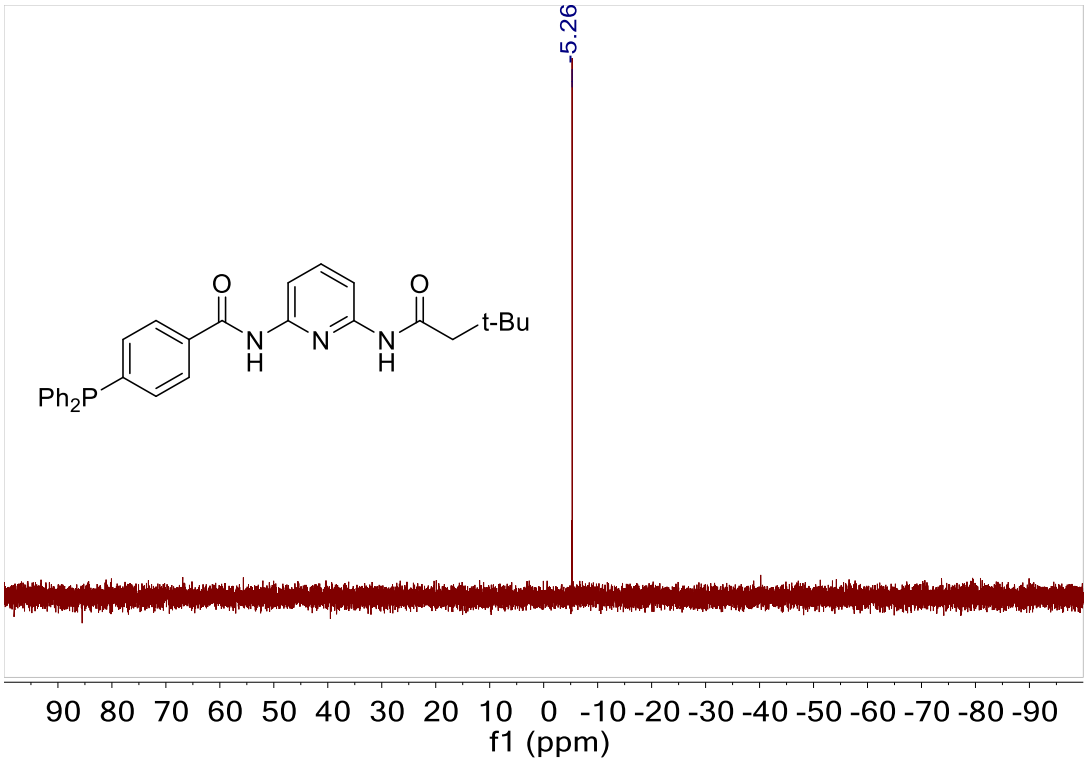
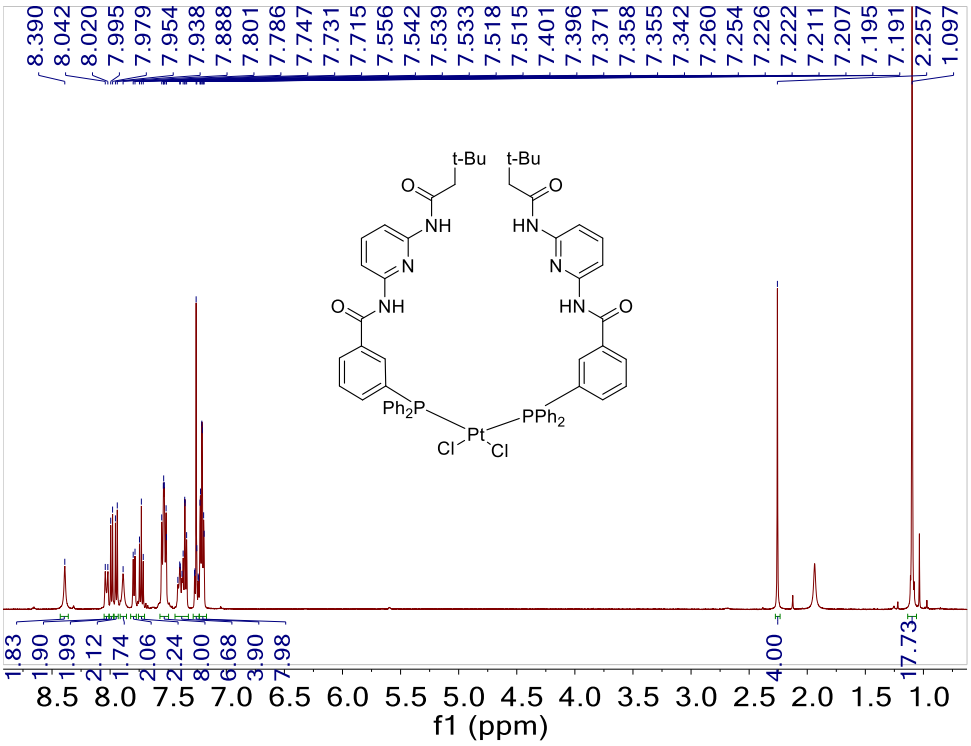
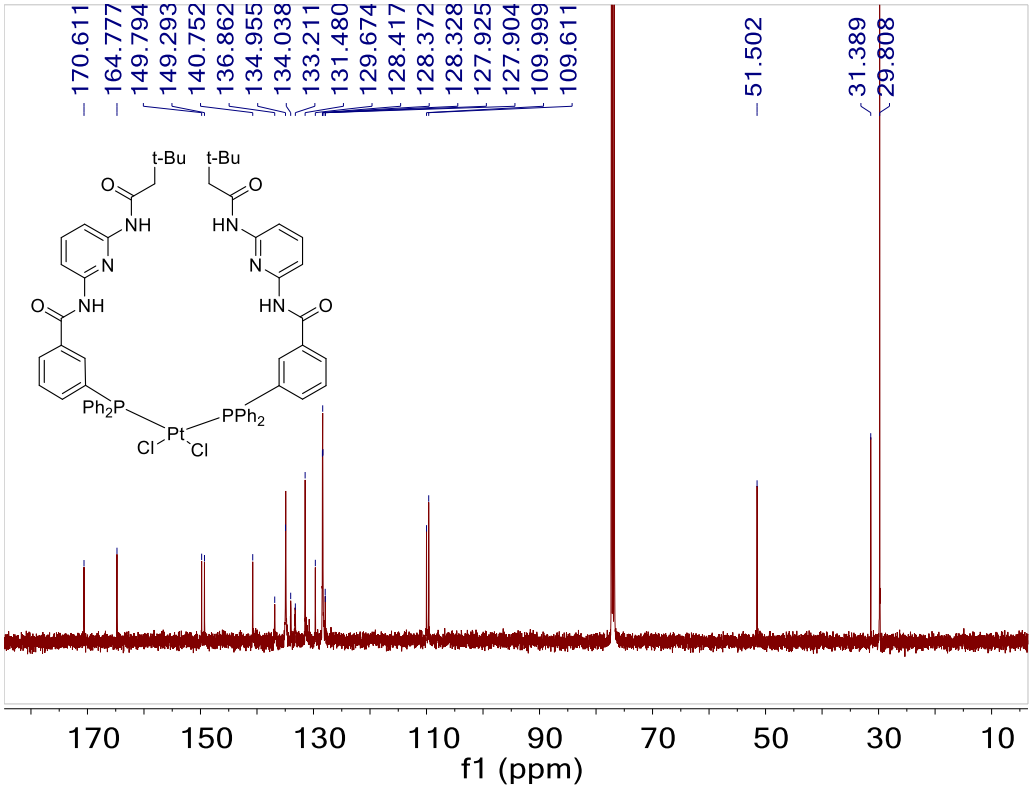


Figure S10. ^{1}H (500 MHz), $^{13}C\{^{1}H\}$ (126 MHz), and $^{31}P\{^{1}H\}$ (202 MHz) NMR spectra of **3c** in $CDCl_3$.




170.611
164.777
149.794
149.293
140.752
136.862
134.955
134.038
133.211
131.480
129.674
128.417
128.372
128.328
127.925
127.904
109.999
109.611
51.502
31.389
29.808
t-Bu
t-Bu
O
NH
HN
N
Ph2P
PPh2
Pt
Cl
170
150
130
110
90
70
50
30
10
f1 (ppm)

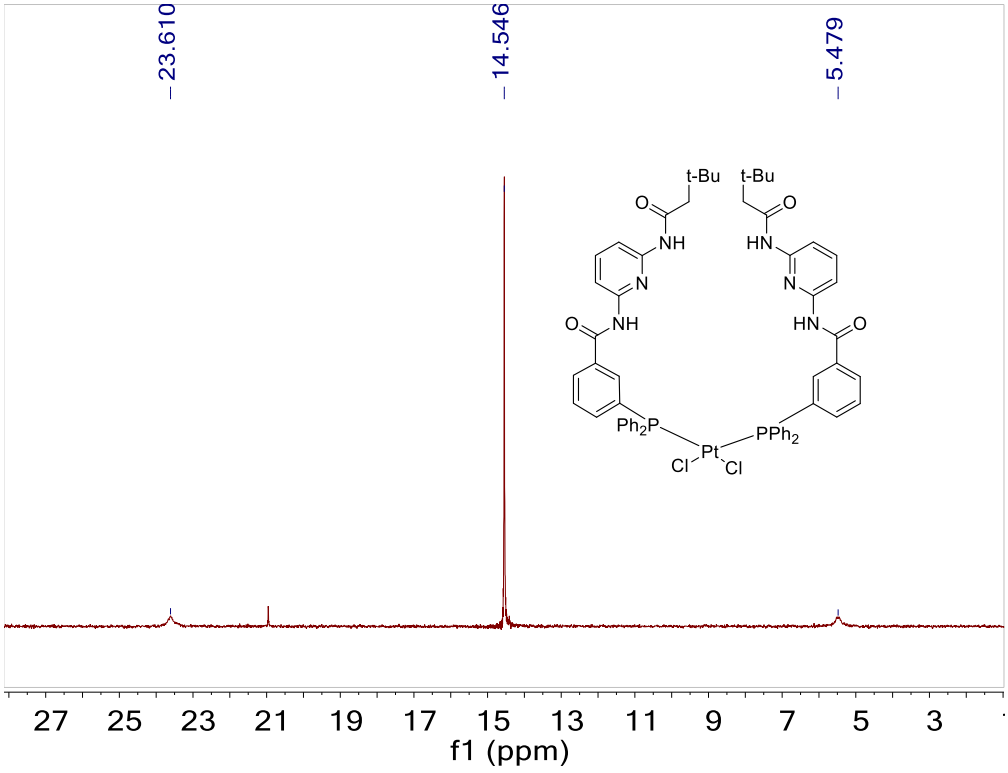
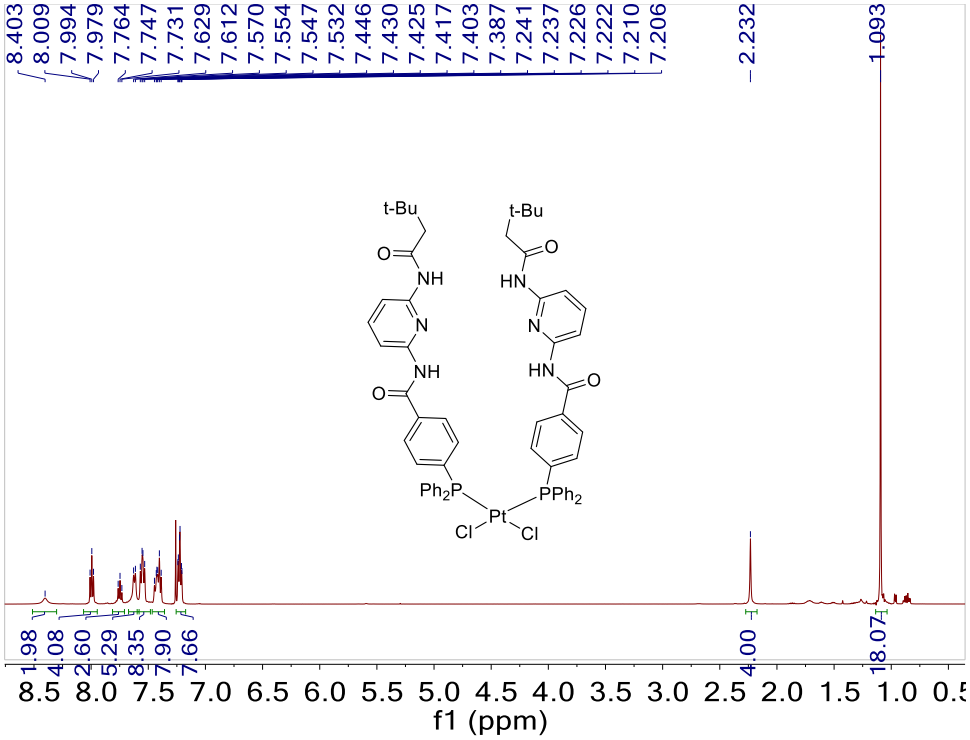
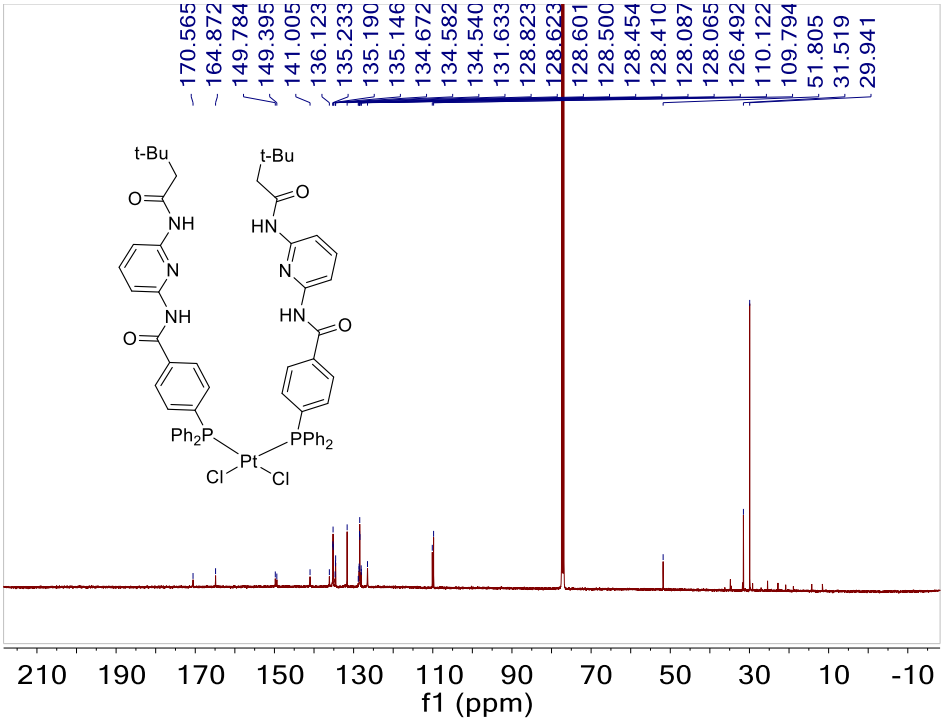

23.610
14.546
5.479
t-Bu
t-Bu
O
NH
HN
N
Ph2P
PPh2
Pt
Cl
27
25
23
21
19
17
15
13
11
9
7
5
3
f1 (ppm)

Figure S11. ^{1}H (500 MHz), $^{13}C\{^{1}H\}$ (126 MHz), and $^{31}P\{^{1}H\}$ (202 MHz) NMR spectra of **4b** in $CDCl_3$





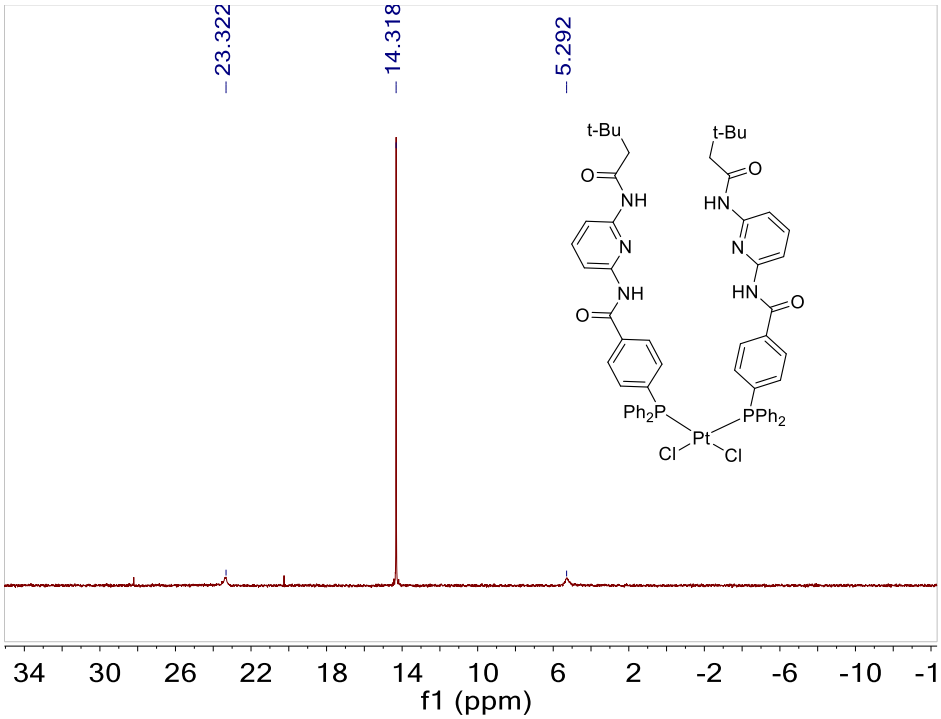
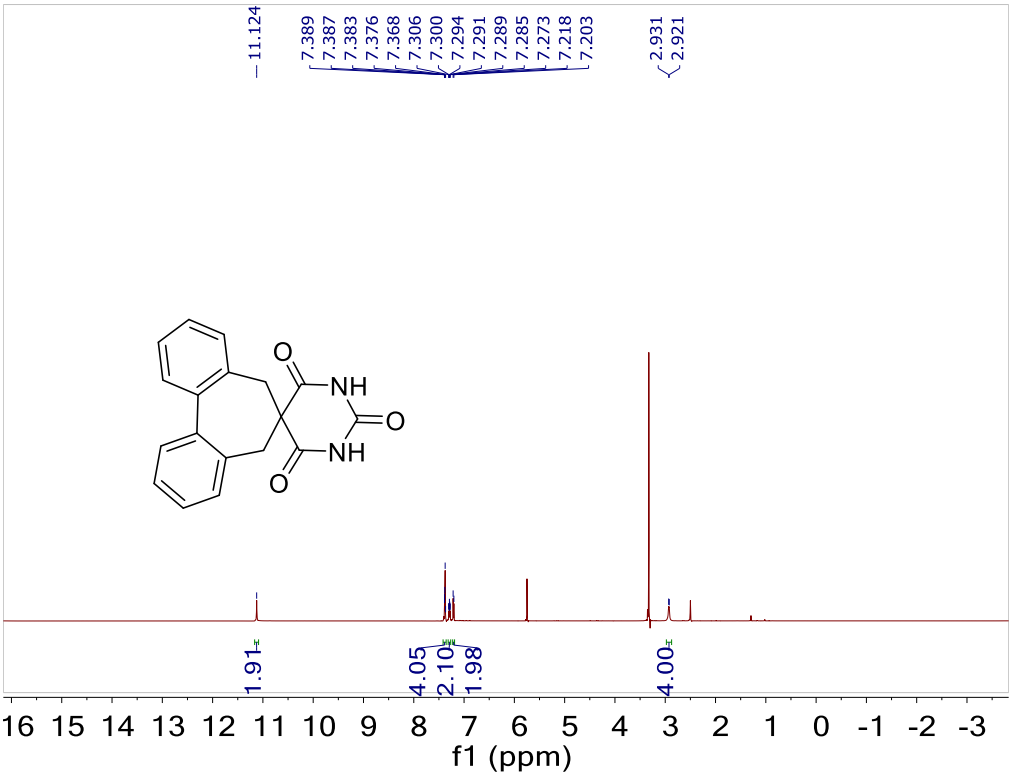


Figure S12. ^{1}H (500 MHz), $^{13}C\{^{1}H\}$ (126 MHz), and $^{31}P\{^{1}H\}$ (202 MHz) NMR spectra of **4c** in $CDCl_3$



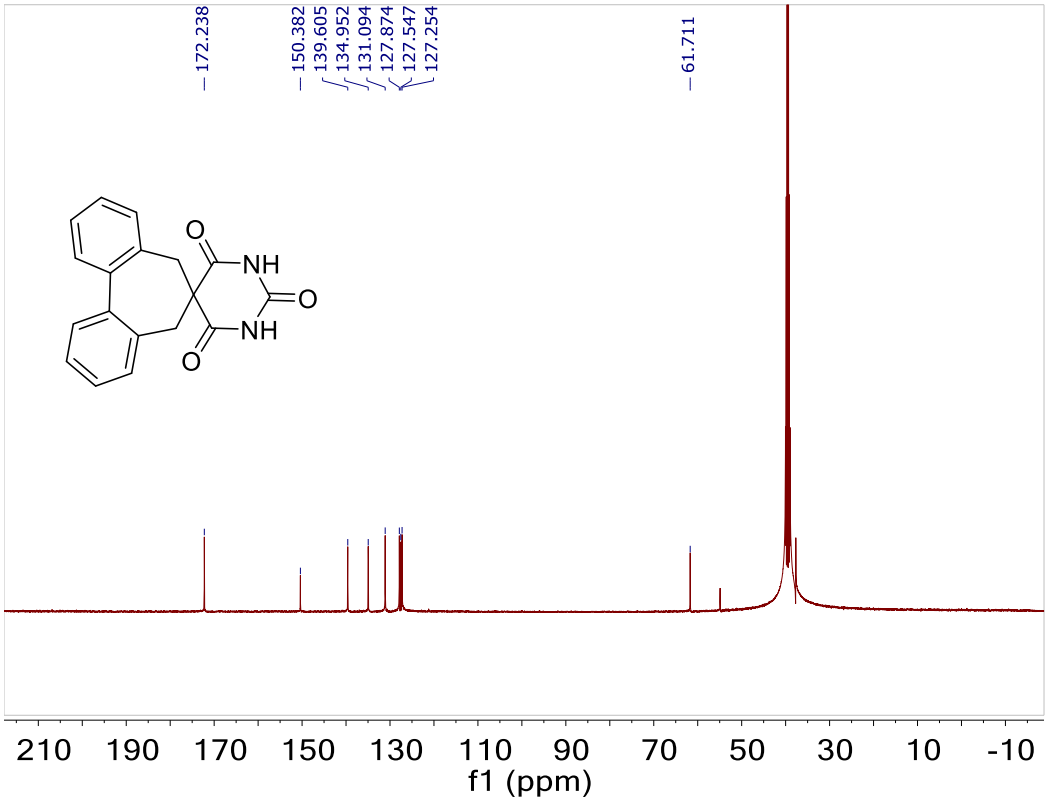


Figure S13. ^{1}H (500 MHz) and ^{13}C{^{1}H} (126 MHz), NMR spectra of **5a** in DMSO-d_6.

Example Binding Isotherms

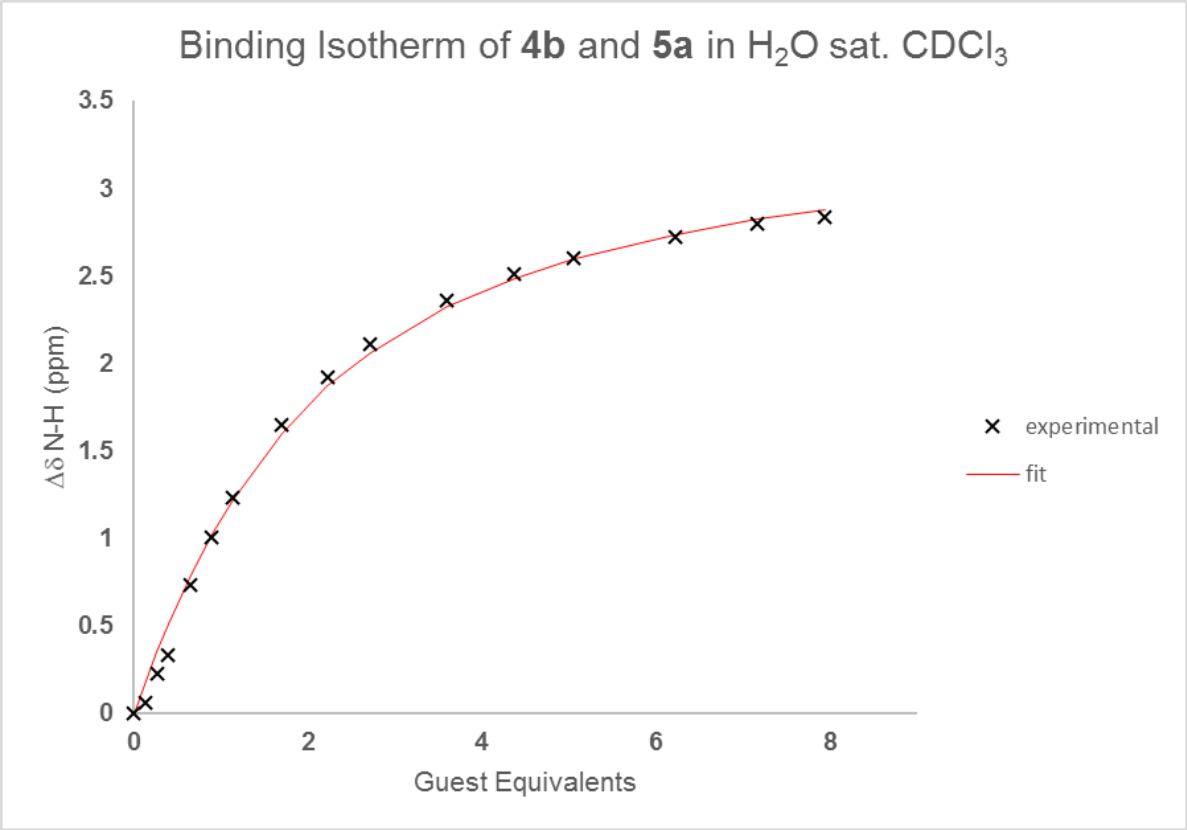


Figure S14: Binding isotherm from NMR titration of **4b** and **5a** in H_2O sat. $CDCl_3$ at 25 °C

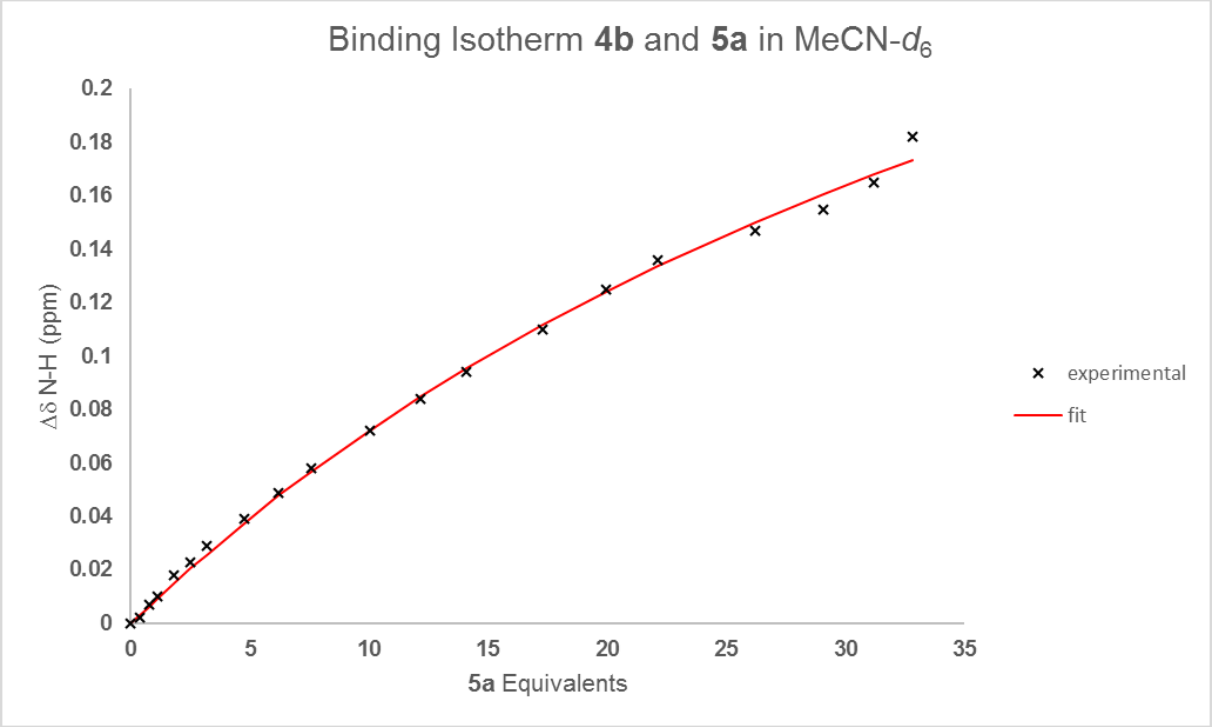


Figure S15: Binding isotherm from NMR titration of **4b** and **5a** in MeCN-d_6 at 25 °C

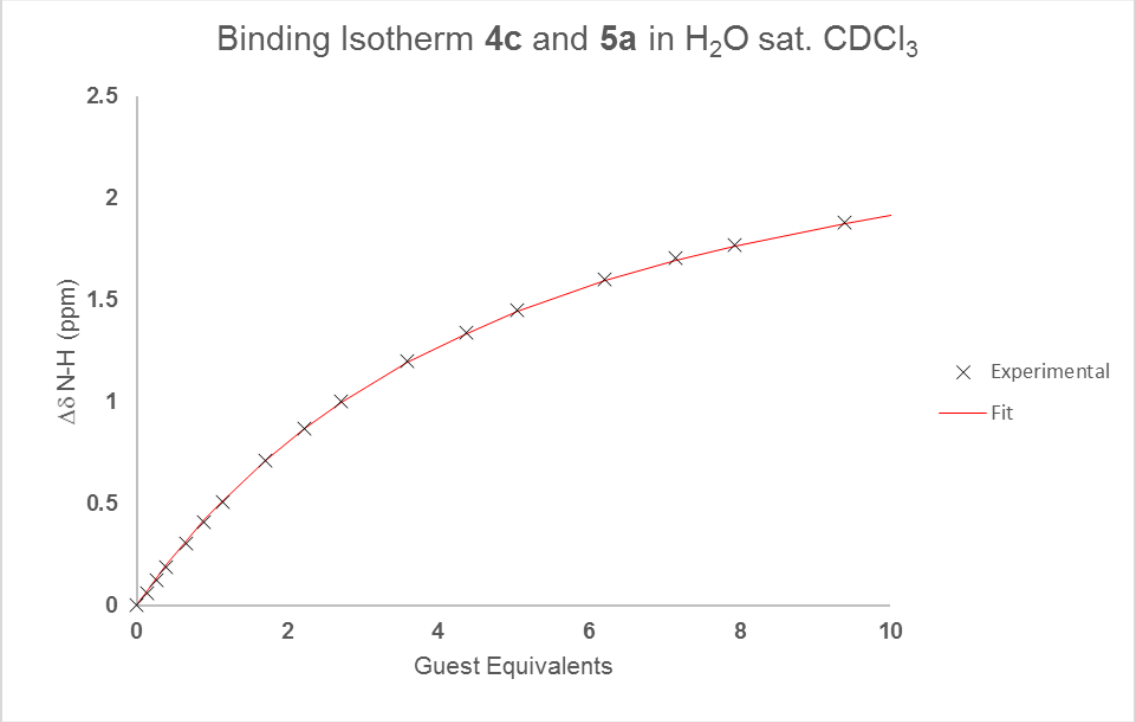


Figure S16: Binding isotherm from NMR titration of **4c** and **5a** in H_2O sat. $CDCl_3$ at 25 °C

References

1) McGrath, J. M.; Pluth, M. D. *J. Org. Chem.* **2014**, *79*, 11797–11801.