Electronic Supplementary Material (ESI) for Green Chemistry.

A nitrogen-ligated nickel-catalyst enables selective intermolecular cyclisation of β- and γ-amino alcohols with ketones: access to five and six-membered N-heterocycles

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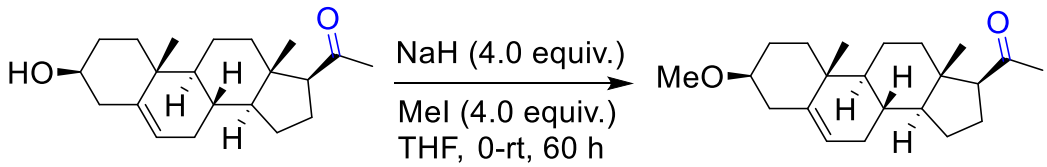
1.1 General Experimental Details:

All solvents and reagents were used, as received from the suppliers. TLC was performed on Merck Kiesel gel 60, F_{254} plates with the layer thickness of 0.25 mm. Column chromatography was performed on silica gel (100-200 mesh) using a gradient of ethyl acetate and hexane as mobile phase. ^{1}H NMR spectral data were collected at, 400 (JEOL), 500 (Bruker) MHz, while ^{13}C NMR were recorded at 100, 125 MHz. ^{1}H NMR spectral data are given as chemical shifts in ppm followed by multiplicity (s- singlet; d- doublet; t- triplet; q- quartet; m- multiplet), number of protons and coupling constants. ^{13}C NMR chemical shifts are expressed in ppm. HRMS (ESI) spectral data were collected using Bruker High Resolution Mass. GC-MS were recorded using Perkin-Elmer Mass Spectrometer. All the reactions were performed in a closed system using Schlenk tube. All nickel salts were purchased from Sigma Aldrich. Potassium *tert*-butoxide was purchased from Avra Synthesis Pvt. Ltd., India. (Purity-98%, CAS No: 865-47-4, Catalog No- ASP2012).

1.2 Preparation of starting materials:



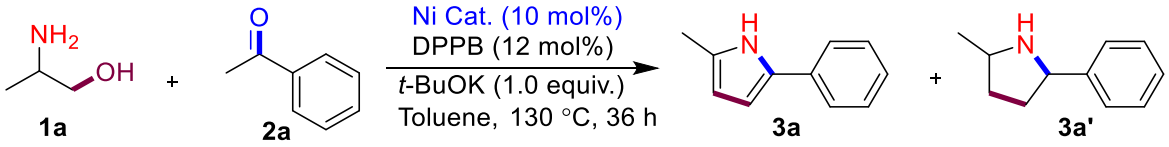
Procedure:

In a 50 mL oven dried RB flask, 5-pregnen-3β-ol-20-one (633 mg, 2.0 mmol), NaH (4.0 equiv.) in THF 10 mL were taken under an atmosphere of N_2 at 0 °C and stirred for 1 h. Then MeI (4.0 equiv.) was added to the reaction mixture at 0 °C and continued reaction at RT for 60 h. The reaction mixture was quenched with NH_4Cl solution and was partitioned between ethyl acetate (25.0 mL) and water (25.0 mL) in a separatory funnel. The organic layer was washed with water, brine and dried over anhydrous Na_2SO_4 (s) and concentrated *in vacuo*. The residue was purified by column chromatography using a gradient of hexane and ethyl acetate (eluent system) to afford the pure product as colourless solid (430 mg, 65% yield). **NMR DATA**: ^{1}H NMR (400 MHz, $CDCl_3$) δ 5.35 – 5.31 (m, 1H), 3.33 (d, J = 0.8 Hz, 3H), 3.09 – 2.99 (m, 1H), 2.51 (t, J = 8.9 Hz, 1H), 2.37 (ddd, J = 13.1, 4.6, 2.1 Hz, 1H), 2.16 (ddd, J = 12.9, 10.3, 7.8 Hz, 2H), 2.10 (s, 3H), 2.06-1.82 (m, 4H), 1.69 – 1.36 (m, 8H), 1.26 -1.01 (m, 3H), 1.01 – 0.95 (m, 4H), 0.61 (s, 3H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 209.70, 140.93, 121.37, 80.30, 63.78, 56.99,

55.72, 50.09, 44.09, 38.91, 38.70, 37.24, 36.96, 31.91, 31.87, 31.66, 28.04, 24.56, 22.86, 21.15, 19.44, 13.31.

1.3 Nickel catalysed synthesis of pyrroles, quinolines and pyridines from amino alcohols with ketones.

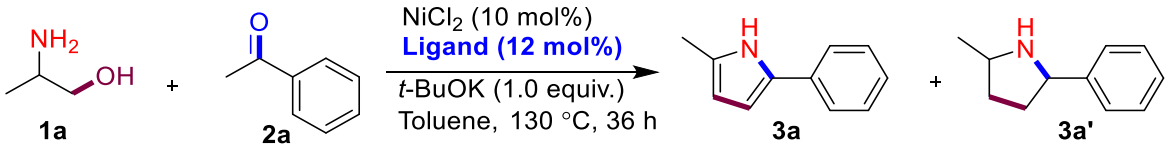
Table S1: Screening of Nickel catalysts

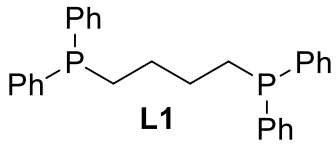


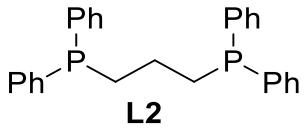
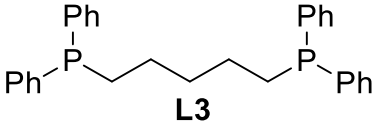
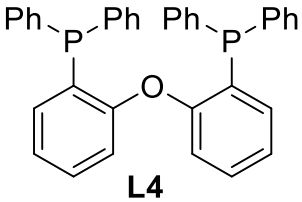
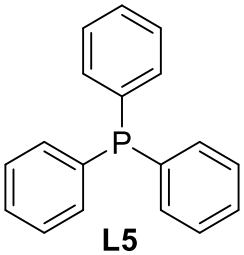
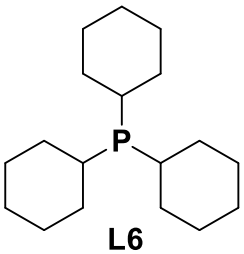
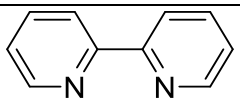
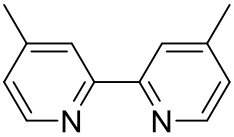
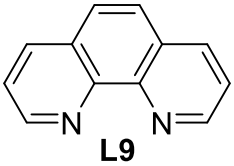
Entry	Ni-catalyst	Base	GC-MS Conversion	
			3a	**3a'**
1.	$NiBr_2$	*t*-BuOK	12	13
2.	**$NiCl_2$**	***t*-BuOK**	**30**	**8**
3.	$NiCl_2$.DME	*t*-BuOK	25	0
4.	$Ni(acac)_2$	*t*-BuOK	18	9
5.	$Ni(COD)_2$	*t*-BuOK	17	12
6[b].	$NiBr_2$	*t*-BuOK	28	20

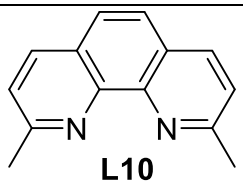
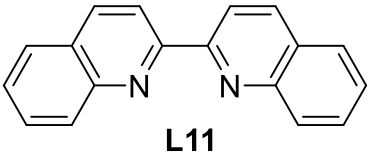
Reaction condition: [a] DL-Alaninol (0.5 mmol), Acetophenone (1.0 mmol), **Ni-cat (10 mol%)**, DPPB (12 mol%), *t*-BuOK (0.5 mmol), toluene (2.0 mL), Schlenk tube under nitrogen atmosphere, 130 °C oil bath, 36 h reaction time. DPPB = 1,4-Bis(diphenylphosphino)butane. [b] 1,10-phenthroline was used as ligand.

Table S2: Screening of Ligands



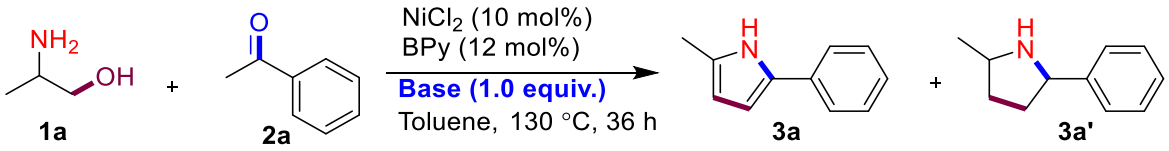
Entry	Ligand	GC-MS Conversion	
		3a	**3a'**
1.	 L1	30 %	8%

2.	 **L2**	28 %	19 %
3.	 **L3**	15 %	2 %
4.	 **L4**	0 %	0 %
5.	 **L5**	15 %	0 %
6.	 **L6**	24 %	0 %
7.	 **L7**	**72 (64)[b] %**	**0%**
8.	 **L8**	10 %	25 %
9.	 **L9**	15 %	0 %

10.	 L10	24 %	0 %
11.	 L11	0 %	0 %
12.	No Ligand	15 %	18 %

Reaction condition: [a] DL-Alaninol (0.5 mmol), Acetophenone (1.0 mmol), $NiCl_2$ (10 mol%), **Ligand (12 mol%)**, *t*-BuOK (0.5 mmol), toluene (2.0 mL), Schlenk tube under nitrogen atmosphere, 130 °C in oil bath, 36 h reaction time. [b] Isolated yield

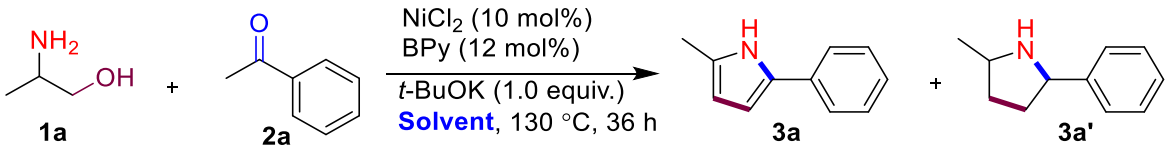
Table S3: Screening of Base



Entry	Base	GC-MS Conversion	
		3a	**3a'**
1.	*t*-BuONa	50 %	0%
2.	***t*-BuOK**	**72 (64)[b] %**	**0%**
3.	K_2CO_3	0 %	0 %
4.	Na_2CO_3	0 %	0 %
5.	K_3PO_4	0%	0 %
6.	Cs_2CO_3	0 %	0 %

Reaction condition: [a] DL-Alaninol (0.5 mmol), Acetophenone (1.0 mmol), $NiCl_2$ (10 mol%), BPy (12 mol%), **Base (0.5 mmol)**, Toluene (2.0 mL), Schlenk tube under nitrogen atmosphere, 130 °C in oil bath, 36 h reaction time. [b] Isolated yield

Table S4: Screening of Solvents



Entry	Solvents	Base	GC-MS Conversion	
			3a	**3a'**
1.	**Toluene**	***t*-BuOK**	**72 (64)[b] %**	**0%**
2.	Xylene	*t*-BuOK	31 %	3%
3.	Dioxane	*t*-BuOK	39 %	8 %
4.	DMA	*t*-BuOK	14 %	10 %
5.	DMF	*t*-BuOK	9 %	10 %

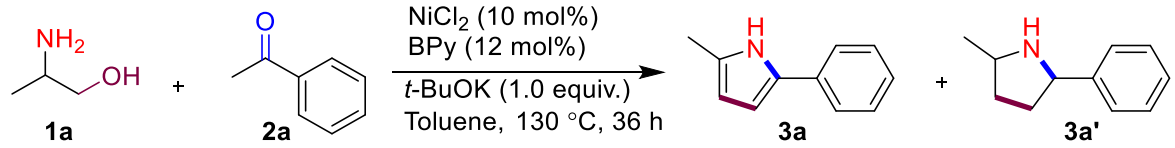
Reaction condition: [a] DL-Alaninol **(**0.5 mmol), Acetophenone (1.0 mmol), $NiCl_2$ (10 mol%), BPy (12 mol%), *t*-BuOK (0.5 mmol), **Solvent (2.0 mL)**, Schlenk tube under nitrogen atmosphere, 130 °C in oil bath, 36 h reaction time. [b] Isolated yield

Table S5: Screening of base equivalents [a]

Entry	Base equivalents	GC-MS Conversion	
		3a	**3a'**
1.	***t*-BuOK (1.0 equiv.)**	**72 (64)[b] %**	**0%**
2.	*t*-BuOK (0.75 equiv.)	41 %	0%
3.	*t*-BuOK (0.50 equiv.)	29 %	0%
4.	-	0 %	0 %

Reaction condition: [a] DL-Alaninol (0.5 mmol), Acetophenone (1.0 mmol), $NiCl_2$ (10 mol%), BPy (12 mol%), ***t*-BuOK** (**X equiv.**), Toluene (2.0 mL), Schlenk tube under nitrogen atmosphere, 130 °C in oil bath, 36 h reaction time. [b] Isolated yield

Table S6: Screening of Alcohol and Ketone equivalents [a]



Entry	**1a** X mmol	**2a** Y mmol	GC-MS Conversion	
			3a	**3a'**
1.	**0.5**	**1.0**	**72 (64)[b] %**	**0%**
2.	0.5	0.5	46 %	8 %
3.	0.75	0.5	17 %	43 %
4.	1.0	0.5	14 %	55 %

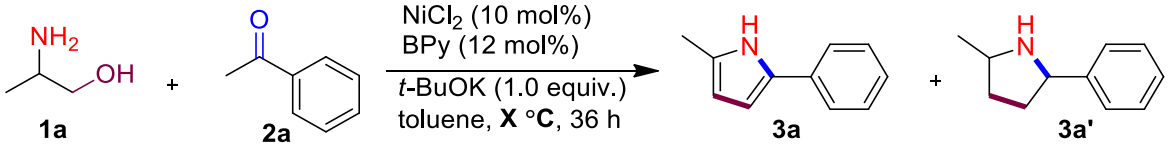
Reaction condition: [a] DL-Alaninol **1a** (**X mmol**), Acetophenone **2a** (**Y mmol**), $NiCl_2$ (10 mol%), BPy (12 mol%), *t*-BuOK (0.5 mmol), Toluene (2.0 mL), Schlenk tube under nitrogen atmosphere, 130 °C in oil bath, 36 h reaction time. [b] Isolated yield.

Table S7: Screening of catalyst/ligand loading [a]

Entry	Catalyst loading	Ligand Loading	GC-MS Conversion	
			3a	**3a'**
1.	**$NiBr_2$ (10 mol%)**	**BPy (12 mol%)**	**72 (64)[b] %**	**0%**
2.	$NiBr_2$ (7.5 mol%)	BPy (9 mol%)	54 %	0 %
3.	$NiBr_2$ (5.0 mol%)	BPy (6 mol%)	42 %	0 %
4.	-	-	5 %	0 %

Reaction condition: [a] DL-Alaninol (0.5 mmol), Acetophenone (1.0 mmol), **$NiCl_2$ (X mol%)**, **BPy (Y mol%)**, *t*-BuOK (0.5 mmol), Toluene (2.0 mL), Schlenk tube under nitrogen atmosphere, 130 °C in oil bath, 36 h reaction time. [b] Isolated yield.

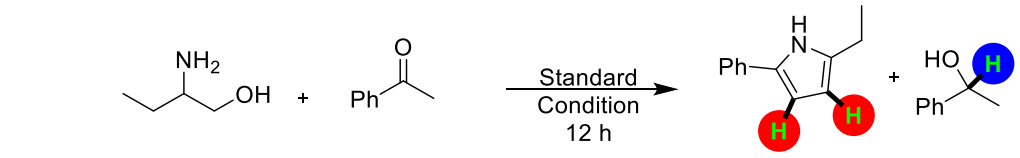
Table S8: Screening of temperature[a]

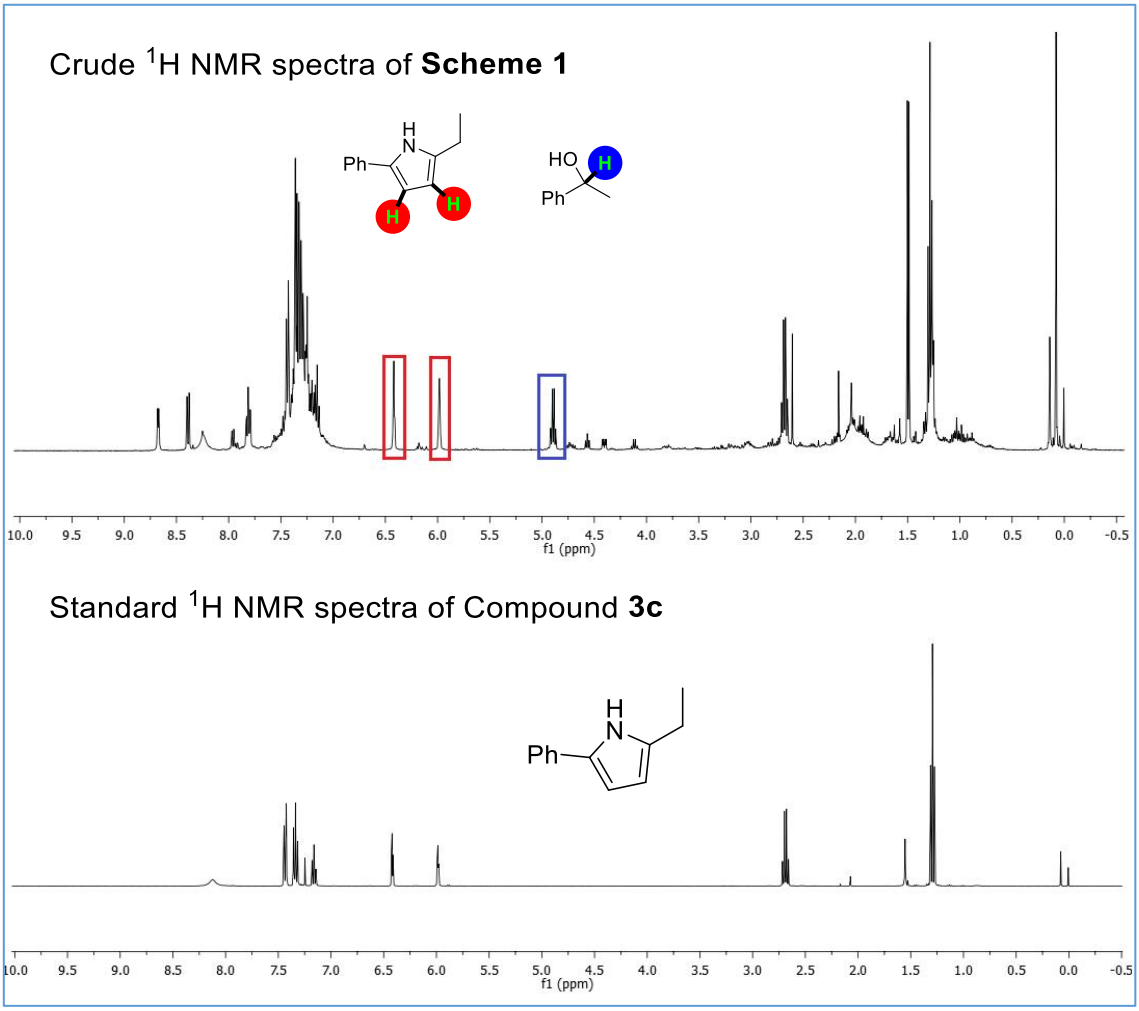


Entry	Temperature (X °C)	GC-MS Conversion	
		3a	**3a'**
1.	**130 °C**	**72 (64)[b] %**	**0%**
2.	120 °C	49 %	0%
3.	110 °C	36 %	0%
4.	100 °C	25 %	0 %
5.	80 °C	1 %	0 %

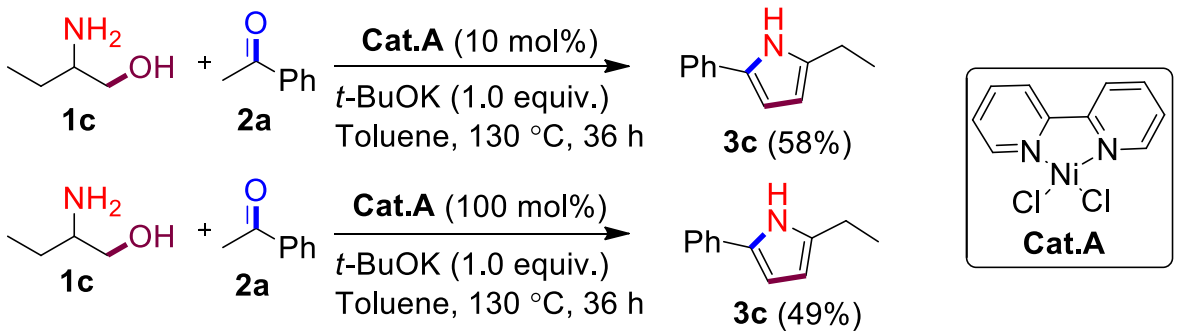
Reaction condition: [a] DL-Alaninol (0.5 mmol), Acetophenone (1.0 mmol), $NiCl_2$ (10 mol%), BPy (12 mol%), *t*-BuOK (0.5 mmol), Toluene (2.0 mL), Schlenk tube under nitrogen atmosphere, **X °C** oil bath, 36 h reaction time. [b] Isolated yield.

Scheme S1 [1]**:**

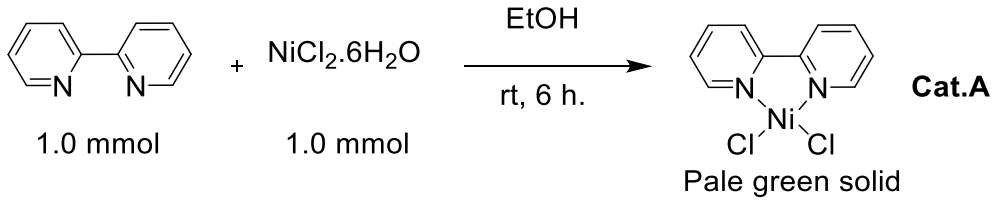




Scheme S2: Stoichiometric and catalytic studies using **Cat.A**



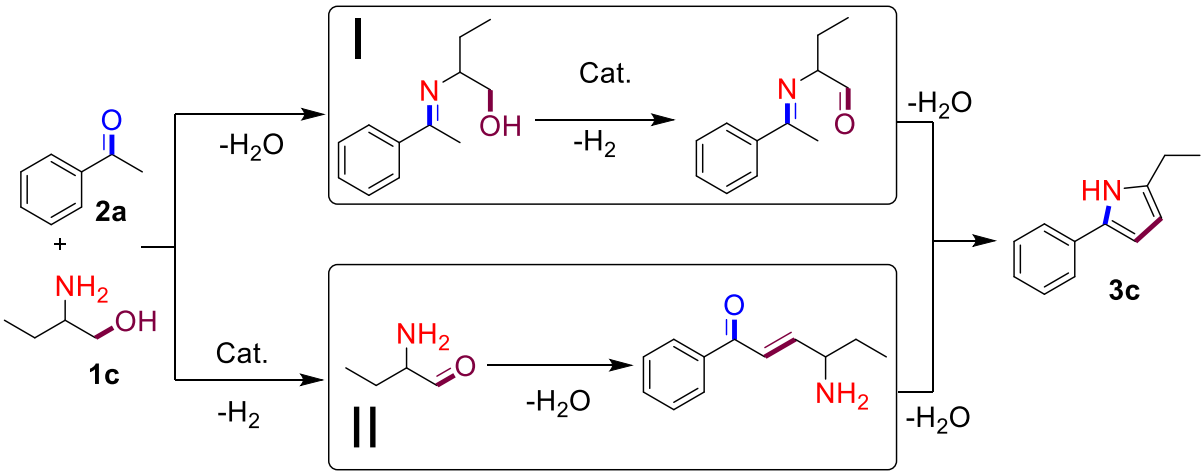
Preparation: Synthesis of [$NiCl_2$(bpy)] complex:[1]



A solution of bpy (78 mg, 0.5 mmol) in EtOH (2 mL) was added to a solution of $NiCl_2$•$6H_2O$ (119 mg, 0.5 mmol) in EtOH (2 mL) at rt, after stirring for 6 h, a pale green precipitate formed and was filtered off, washed with EtOH (3×3 mL), and dried *in vacuo* to afford pale green solid 114 mg (80%) yield. *Elemental Analysis* calculated: C, 42.03; H, 2.82; Cl, 24.81; N, 9.80; Ni, 20.54; Found: C, 41.75; H, 2.76; N, 9.61.

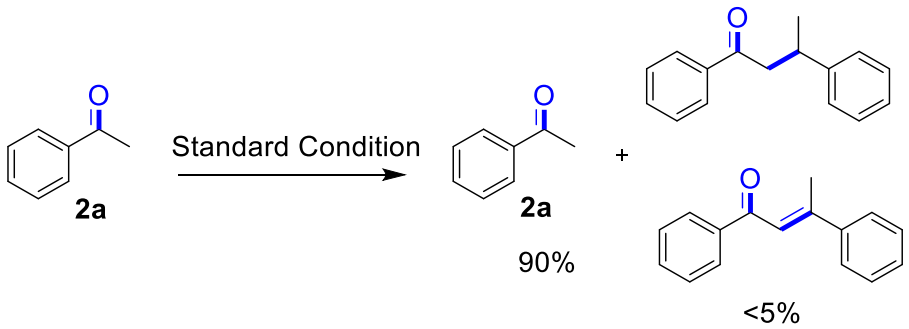
Reference: 1. D. G. Yakhvarov, E. H. Hawkins, R. M. Kagirov, Y. H. Budnikova, Y. S. Ganushevich, O. G. Sinyashin, *Russian Chemical Bulletin, Int. Ed.*, **2007**, *56*, 935 - 942.

Scheme S3: Possible reaction pathways



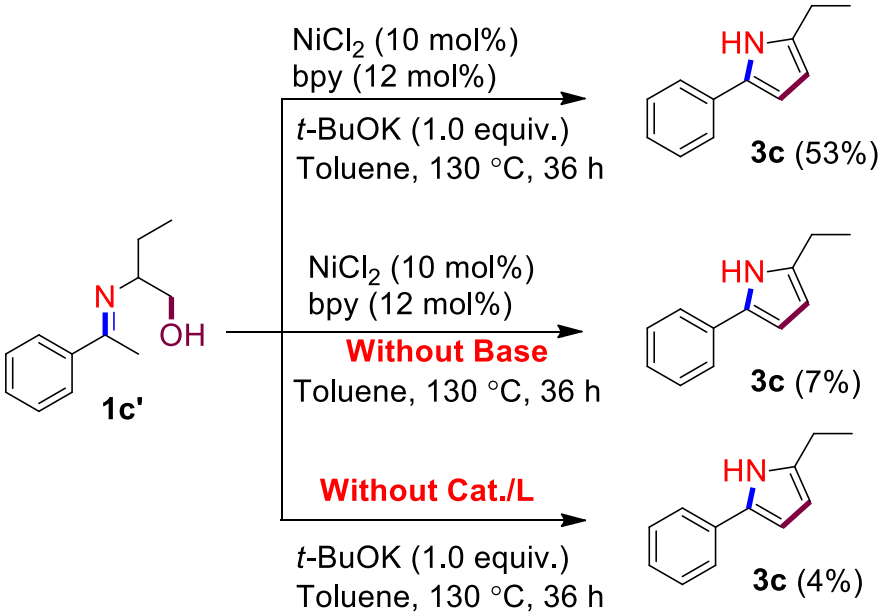
Reactions which confirm Pathway I:

Scheme S4:



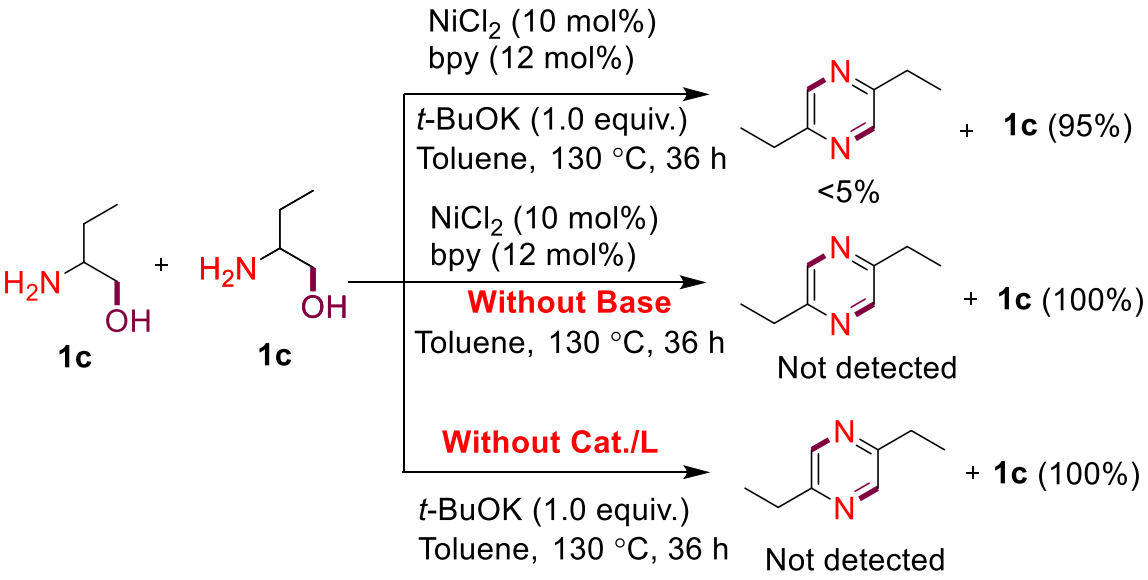
Acetophenone was treated with standard conditions and we observed <5% of aldol condensation product.

Scheme S5: Conversion of 2-(1-phenylethylideneamino)butan-1-ol to 2-ethyl-5-phenyl-1*H*-pyrrole (**3c**)



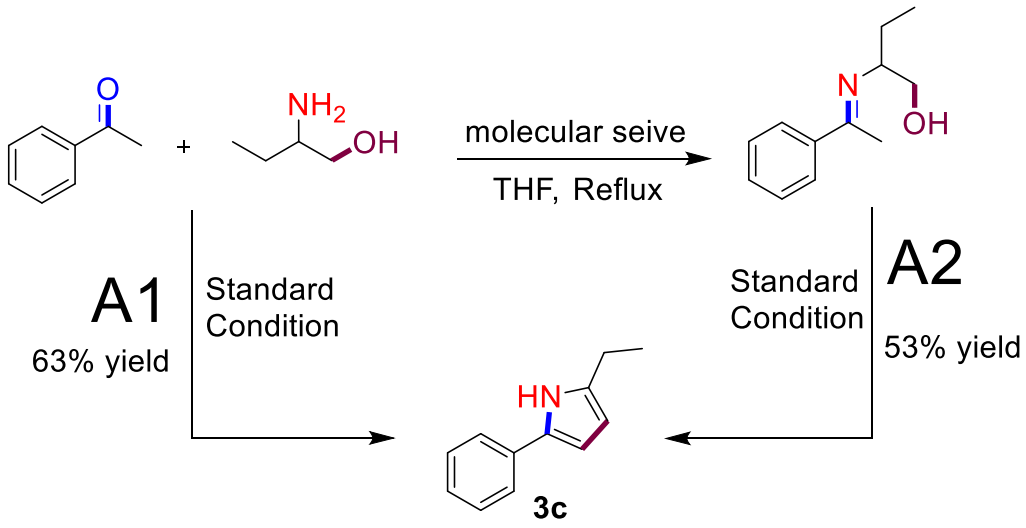
Under standard conditions 2-(1-phenylethylideneamino)butan-1-ol **1c'** is converted to 2-ethyl-5-phenyl-1*H*-pyrrole in 53% yield. If no catalyst or base was used under standard conditions, we observed only 4-7% of product conversion and unreacted imine. These experiments evident the potential role of catalyst and base for intramolecular cyclization to pyrrole.

Scheme S6: Dehydrogenation and condensation reaction of 2-amino-1-butanol to 2,5-diethylpyrazine



Dehydrogenation and condensation reaction of amino alcohol to 2,5-diethylpyrazine is very slow under the standard reaction conditions. In absence of catalyst or base following standard conditions, we did not observe any product conversion.

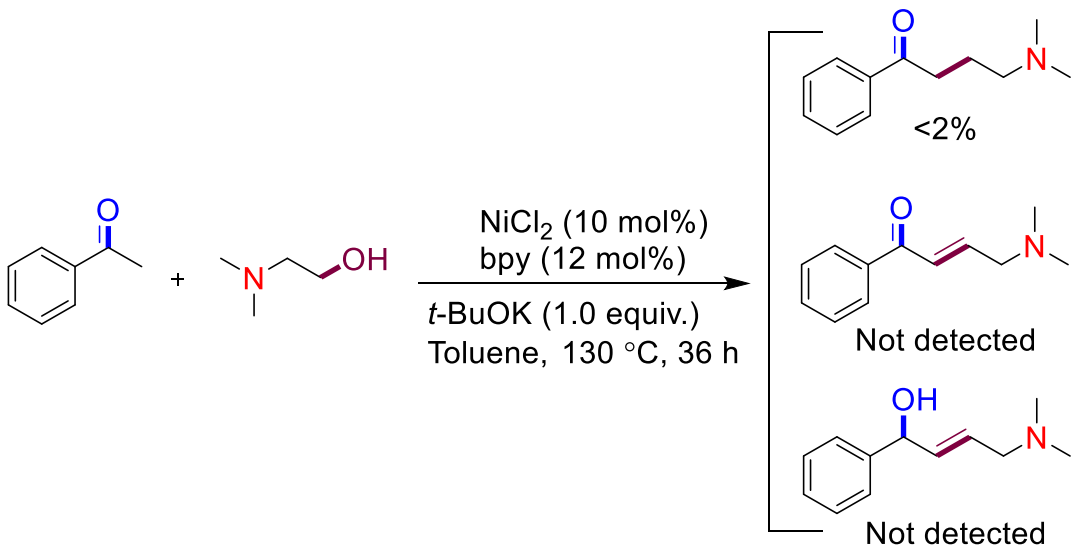
Scheme S7:



Under standard catalytic conditions, the reaction of ketone and amino alcohol (path **A1**), or the reaction of preformed Schiff base (path **A2**), resulted similar efficiency.

Reactions which contradict Pathway II:

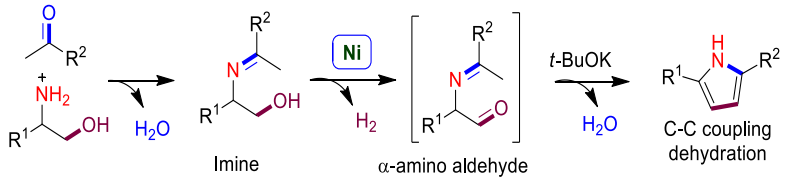
Scheme S8:



The reaction between acetophenone and the N-protected amino alcohol using standard catalytic conditions, we observed only <2% of the α-alkylation product. This experiment indicated that

the α-alkylation is very slow and unlikely be first step during pyrrole synthesis (pathway B, Scheme S3).

Scheme S9. Proposed mechanism for synthesis of pyrroles



[1.4] General Procedure for synthesis of pyrroles, quinolines and pyridines:

Procedure A:

In a 15 mL oven dried Schlenk tube amino alcohols (0.5 mmol), *t*-BuOK (0.5 mmol), $NiCl_2$ (0.05 mmol), BPy (0.06 mmol) and ketones (1.0 mmol) were added followed by toluene 2.0 mL under an atmosphere of N_2 and after sealing the Schlenk tube the reaction mixture was heated at 130 °C for 36 h. The reaction mixture was cooled to room temperature and 3.0 mL of ethyl acetate was added and concentrated *in vacuo*. The residue was purified by column chromatography using a gradient of hexane and ethyl acetate (eluent system) to afford the pure products.

Procedure B:

In a 15 mL oven dried Schlenk tube, amino alcohols (0.5 mmol), *t*-BuOK (0.75 mmol), $NiCl_2$ (0.05 mmol), BPy (0.06 mmol) and ketones (1.0 mmol) were added followed by toluene 2.0 under an atmosphere of N_2 and after sealing the Schlenk tube the reaction mixture was heated at 130 °C for 36 h. The reaction mixture was cooled to room temperature and 3.0 mL of ethyl acetate was added and concentrated *in vacuo*. The residue was purified by column chromatography using a gradient of hexane and ethyl acetate (eluent system) to afford the pure products.

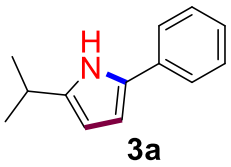
Procedure C:

In a 15 mL oven dried Schlenk tube, amino alcohol (0.2 mmol), *t*-BuOK (0.2 mmol), $NiCl_2$ (0.02 mmol), BPy (0.024 mmol), ketone (0.25 mmol) were added followed by toluene 1.5 mL under an atmosphere of N_2 and after sealing the Schlenk tube the reaction mixture was heated at 130 °C for 36 h. The reaction mixture was cooled to room temperature and 3.0 mL of ethyl acetate was added and concentrated *in vacuo*. The residue was purified by column

chromatography using a gradient of hexane and ethyl acetate (eluent system) to afford the pure product.

[1.5] Analytical Data:

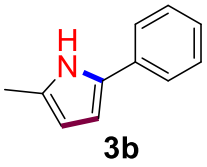
2-Isopropyl-5-phenyl-1*H*-pyrrole (3a) [1]:



3a

Following the general procedure A, the title product was obtained as a colourless solid (70% yield); ^{1}H NMR (400 MHz, $CDCl_3$) δ 8.12 (br s, 1H), 7.44 (dd, J = 8.2, 0.8 Hz, 2H), 7.33 (t, J = 7.7 Hz, 2H), 7.16 (t, J = 7.4 Hz, 1H), 6.41 (t, J = 3.1 Hz, 1H), 5.98 (t, J = 3.0 Hz, 1H), 2.97 (dt, J = 13.8, 6.9 Hz, 1H), 1.30 (d, J = 7.0 Hz, 6H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 135.90, 133.18, 131.00, 128.94, 125.82, 123.56, 106.47, 105.93, 27.29, 22.76.

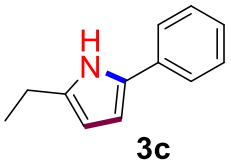
2-Methyl-5-phenyl-1*H*-pyrrole (3b) [1]:



3b

Following the general procedure A, the title product was obtained as a colourless oil (64% yield); ^{1}H NMR (400 MHz, $CDCl_3$) δ 8.13 (br s, 1H), 7.43 (dd, J = 8.2, 1.1 Hz, 2H), 7.36 – 7.32 (m, 2H), 7.19 – 7.13 (m, 1H), 6.40 (t, J = 3.0 Hz, 1H), 5.96 (t, J = 2.5 Hz, 1H), 2.33 (s, 3H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 133.05, 130.88, 128.91, 128.60, 125.74, 123.44, 108.03, 106.27, 29.82.

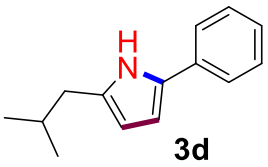
2-Ethyl-5-phenyl-1*H*-pyrrole (3c) [1]:



3c

Following the general procedure A, the title product was obtained as a colourless oil (63% yield); ^{1}H NMR (400 MHz, $CDCl_3$) δ 8.12 (br s, 1H), 7.46 – 7.42 (m, 2H), 7.36 – 7.31 (m, 2H), 7.16 (t, J = 7.3 Hz, 1H), 6.42 (t, J = 3.0 Hz, 1H), 5.99 (t, J = 3.0 Hz, 1H), 2.69 (q, J = 7.6 Hz, 2H), 1.29 (t, J = 7.6 Hz, 3H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 135.71, 133.10, 130.68, 128.89, 125.75, 123.49, 106.32, 106.10, 21.09, 13.68.

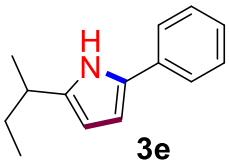
2-Isobutyl-5-phenyl-1*H*-pyrrole (3d) [1]:



3d

Following the general procedure A, the title product was obtained as a colourless solid (65% yield); ^{1}H NMR (400 MHz, $CDCl_3$) δ 8.08 (br s, 1H), 7.46 – 7.42 (m, 2H), 7.35 (dd, J = 10.6, 5.0 Hz, 2H), 7.20 – 7.13 (m, 1H), 6.44 (t, J = 3.0 Hz, 1H), 5.98 (t, J = 3.0 Hz, 1H), 2.51 (d, J = 7.1 Hz, 2H), 1.90 (dp, J = 13.5, 6.7 Hz, 1H), 0.98 (d, J = 6.6 Hz, 6H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 133.35, 133.09, 130.54, 128.92, 125.70, 123.42, 108.10, 106.15, 37.48, 29.40, 22.59.

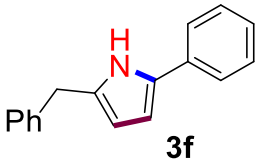
2-(*sec*-Butyl)-5-phenyl-1*H*-pyrrole (3e) [1]:



3e

Following the general procedure A, the title product was obtained as a colourless oil (58% yield); ^{1}H NMR (400 MHz, $CDCl_3$) δ 8.12 (br s, 1H), 7.46 (d, J = 7.7 Hz, 2H), 7.35 (t, J = 7.7 Hz, 2H), 7.17 (t, J = 7.3 Hz, 1H), 6.45 (t, J = 2.9 Hz, 1H), 6.00 (t, J = 2.7 Hz, 1H), 2.79 – 2.69 (m, 1H), 1.66 (tdd, J = 20.7, 13.7, 7.1 Hz, 2H), 1.31 (d, J = 7.0 Hz, 3H), 0.95 (t, J = 7.4 Hz, 3H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 139.30, 133.13, 130.35, 128.91, 125.72, 123.46, 105.93, 105.75, 34.52, 30.36, 20.19, 11.97.

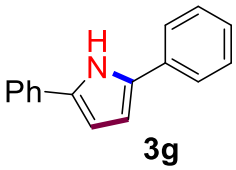
2-Benzyl-5-phenyl-1*H*-pyrrole (3f) [1]:



3f

Following the general procedure A, the title product was obtained as a colourless solid (46% yield); ^{1}H NMR (400 MHz, $CDCl_3$) δ 8.03 (br s, 1H), 7.41 – 7.37 (m, 2H), 7.36 – 7.29 (m, 4H), 7.28 – 7.23 (m, 3H), 7.16 (t, J = 7.3 Hz, 1H), 6.45 (t, J = 3.0 Hz, 1H), 6.06 (t, J = 3.0 Hz, 1H), 4.03 (s, 2H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 139.38, 132.92, 132.10, 131.61, 128.89, 128.80, 128.76, 126.65, 125.92, 123.56, 108.72, 106.21, 34.35.

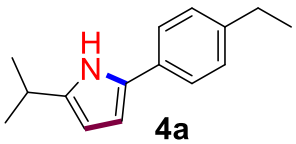
2,5-Diphenyl-1*H*-pyrrole (3g) [1]:



3g

Following the general procedure A, the title product was obtained as a colourless solid (37% yield); ^{1}H NMR (400 MHz, $CDCl_3$) δ 8.59 (br s, 1H), 7.56 – 7.51 (m, 4H), 7.42 – 7.36 (m, 4H), 7.23 (dd, J = 10.7, 4.3 Hz, 2H), 6.59 (d, J = 2.6 Hz, 2H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 133.21, 132.57, 129.05, 126.48, 123.88, 108.01.

2-(4-Ethylphenyl)-5-isopropyl-1*H*-pyrrole (4a):

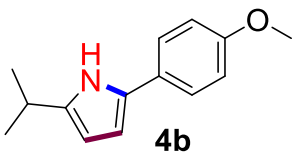


4a

Following the general procedure A, the title product was obtained as a pale brown oil (47% yield); ^{1}H NMR (400 MHz, $CDCl_3$) δ 8.12 (br s, 1H), 7.37 (d, J = 8.3 Hz, 2H), 7.18 (d, J = 8.2 Hz, 2H), 6.39 – 6.35 (m, 1H), 5.98 (t, J = 3.4 Hz, 1H), 3.03 – 2.92 (m, 1H), 2.65 (q, J = 7.6 Hz, 2H), 1.31 (d, J = 6.9 Hz, 6H), 1.25 (t, J = 7.6 Hz, 3H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 141.89, 140.00, 130.79, 130.70, 128.38, 123.68, 105.32, 104.84, 28.60, 27.31, 22.80, 15.68; HRMS (ESI): Calculated for $[C_{15}H_{19}N]^{+}$ 213.1512; Found 213.1534.

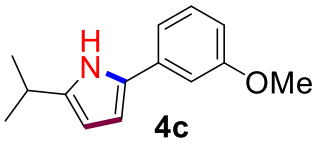
2-Isopropyl-5-(4-methoxyphenyl)-1*H*-pyrrole (4b) [5]:

Following the general procedure A, the title product was obtained as a white solid (49% yield);



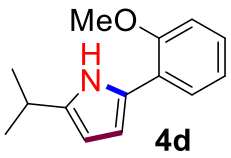
^{1}H NMR (400 MHz, $CDCl_3$) δ 8.04 (br s, 1H), 7.37 (d, J = 8.8 Hz, 2H), 6.89 (d, J = 8.7 Hz, 2H), 6.29 (t, J = 3.0 Hz, 1H), 5.96 (t, J = 2.8 Hz, 1H), 3.81 (s, 3H), 3.01 – 2.88 (m, 1H), 1.30 (d, J = 6.9 Hz, 6H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 158.00, 139.70, 130.59, 126.32, 125.03, 124.96, 114.31, 104.77, 55.45, 27.38, 22.79.

2-Isopropyl-5-(3-methoxyphenyl)-1*H*-pyrrole (4c) [2b]:



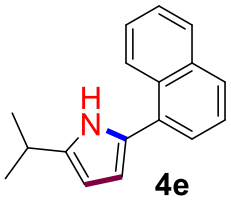
Following the general procedure A, the title product was obtained as a pale brown oil (68% yield); ^{1}H NMR (400 MHz, $CDCl_3$) δ 8.10 (br s, 1H), 7.24 – 7.21 (m, 1H), 7.02 (d, J = 7.6 Hz, 1H), 6.98 – 6.95 (m, 1H), 6.73 – 6.70 (m, 1H), 6.40 (t, J = 3.4 Hz, 1H), 5.97 (t, J = 3.4 Hz, 1H), 3.83 (s, 3H), 2.96 (dt, J = 13.8, 6.8 Hz, 1H), 1.29 (d, J = 6.9 Hz, 6H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 160.14, 140.51, 134.58, 130.44, 129.95, 116.19, 111.13, 109.52, 106.24, 105.06, 55.40, 27.33, 22.79.

2-Isopropyl-5-(2-methoxyphenyl)-1*H*-pyrrole (4d):



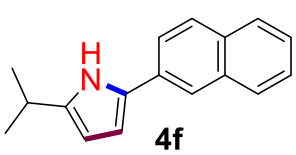
Following the general procedure A, the title product was obtained as a pale brown oil (35% yield); ^{1}H NMR (400 MHz, $CDCl_3$) δ 9.49 (br s, 1H), 7.61 (dd, J = 7.7, 1.6 Hz, 1H), 7.14 – 7.07 (m, 1H), 6.95 (ddd, J = 10.7, 5.9, 2.1 Hz, 2H), 6.52 – 6.48 (m, 1H), 5.97 (t, J = 3.5 Hz, 1H), 3.95 (s, 3H), 3.03 – 2.93 (m, 1H), 1.30 (d, J = 6.9 Hz, 6H); ^{13}C NMR (125 MHz, $CDCl_3$) δ 154.45, 139.13, 128.02, 126.20, 126.09, 121.49, 111.75, 106.13, 103.81, 55.79, 27.23, 22.61; HRMS (ESI): Calculated for $[C_{14}H_{17}NONa]^{+}$ 238.1202; Found 238.1204.

2-Isopropyl-5-(naphthalen-1-yl)-1*H*-pyrrole (4e) [5]:



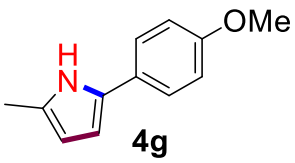
Following the general procedure A, the title product was obtained as a colourless solid (43% yield); ^{1}H NMR (400 MHz, $CDCl_3$) δ 8.28 (br s, 1H), 7.81 – 7.76 (m, 4H), 7.64 (dd, J = 8.5, 1.8 Hz, 1H), 7.47 – 7.36 (m, 2H), 6.54 (t, J = 3.1 Hz, 1H), 6.03 (t, J = 3.0 Hz, 1H), 3.07 – 2.95 (m, 1H), 1.34 (d, J = 6.9 Hz, 6H); ^{13}C NMR (125 MHz, $CDCl_3$) δ 140.83, 133.88, 131.90, 130.46, 128.48, 127.73, 127.55, 126.38, 125.11, 123.16, 120.21, 106.65, 105.25, 27.32, 22.71.

2-Isopropyl-5-(naphthalen-2-yl)-1*H*-pyrrole (4f) [2b]:



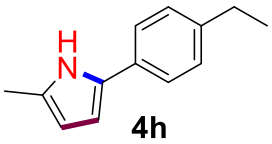
Following the general procedure A, the title product was obtained as a colourless solid (55% yield); ^{1}H NMR (400 MHz, $CDCl_3$) δ 8.35 (dt, J = 7.3, 3.3 Hz, 1H), 8.14 (br s, 1H), 7.92 – 7.83 (m, 1H), 7.77 (dd, J = 7.0, 3.2 Hz, 1H), 7.49 (dt, J = 7.2, 4.0 Hz, 4H), 6.43 – 6.39 (m, 1H), 6.12 – 6.07 (m, 1H), 3.10 – 2.96 (m, 1H), 1.35 (d, J = 7.1 Hz, 6H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 139.93, 134.19, 131.93, 131.37, 128.99, 128.48, 127.15, 126.29, 125.96, 125.93, 125.72, 125.55, 109.43, 104.42, 27.27, 22.76.

2-(4-Methoxyphenyl)-5-methyl-1*H*-pyrrole (4g) [2a]:



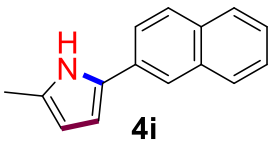
Following the general procedure A, the title product was obtained as a pale brown oil (48% yield); ^{1}H NMR (400 MHz, $CDCl_3$) δ 8.04 (br s, 1H), 7.35 (d, J = 9.1 Hz, 2H), 6.88 (d, J = 9.0 Hz, 2H), 6.30 – 6.24 (m, 1H), 5.95 – 5.88 (m, 1H), 3.81 (s, 3H), 2.32 (s, 3H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 157.97, 130.90, 128.39, 126.26, 124.88, 114.35, 107.69, 105.09, 55.38, 13.30.

2-(4-Ethylphenyl)-5-methyl-1*H*-pyrrole (4h):



Following the general procedure A, the title product was obtained as a light brown oil (54% yield); ^{1}H NMR (400 MHz, $CDCl_3$) δ 8.06 (br s, 1H), 7.34 (d, J = 8.2 Hz, 2H), 7.16 (d, J = 8.2 Hz, 2H), 6.34 (t, J = 3.0 Hz, 1H), 5.93 (t, J = 3.3 Hz, 1H), 2.63 (q, J = 7.6 Hz, 2H), 2.32 (s, 3H), 1.23 (t, J = 7.6 Hz, 3H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 141.84, 131.06, 130.59, 128.66, 128.37, 123.52, 107.82, 105.70, 28.56, 15.62, 13.27; HRMS (ESI): Calculated for $[C_{13}H_{16}N]^+$ 185.1199; Found 185.1206.

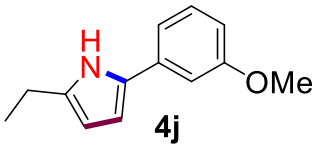
2-Methyl-5-(naphthalen-2-yl)-1*H*-pyrrole (4i) [2a]:



Following the general procedure A, the title product was obtained as a colourless solid (52% yield); ^{1}H NMR (400 MHz, $CDCl_3$) δ 8.29 (br s, 1H), 7.81 – 7.76 (m, 4H), 7.62 (dd, J = 8.6, 1.7 Hz, 1H), 7.46 – 7.35 (m, 2H), 6.52 (t, J = 3.0 Hz, 1H), 6.00 (t, J = 5.8 Hz, 1H), 2.37 (s, 3H); ^{13}C NMR (125 MHz, $CDCl_3$) δ 133.88, 131.88, 130.79, 130.35, 129.55, 128.48, 127.72, 127.55, 126.38, 125.11, 123.06, 120.13, 108.20, 106.99, 13.27.

2-Ethyl-5-(3-methoxyphenyl)-1*H*-pyrrole (4j):

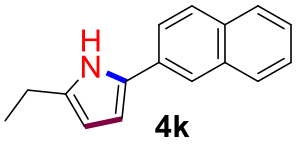
Following the general procedure A, the title product was obtained as a pale brown oil (61% yield); ^{1}H NMR (400 MHz, $CDCl_3$) δ 8.12 (br s, 1H), 7.25 (t, J = 8.0 Hz, 1H), 7.02 (d, J = 7.7 Hz, 1H), 6.99 – 6.95 (m, 1H), 6.72 (dd, J = 8.2, 2.5 Hz, 1H), 6.41 (t, J = 3.0 Hz, 1H), 5.98 (t, J



4j

= 2.9 Hz, 1H), 3.83 (s, 3H), 2.68 (q, *J* = 7.6 Hz, 2H), 1.29 (t, *J* = 7.6 Hz, 3H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 160.11, 135.77, 134.49, 130.53, 129.91, 116.10, 111.09, 109.39, 106.36, 106.31, 55.32, 21.09, 13.68; HRMS (ESI): Calculated for $[C_{13}H_{16}NO]^+$ 202.1226; Found 202.1219.

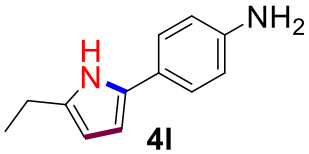
2-Ethyl-5-(naphthalen-2-yl)-1*H*-pyrrole (4k) [2a]:



4k

Following the general procedure A, the title product was obtained as a colourless solid (71% yield); ^{1}H NMR (400 MHz, $CDCl_3$) δ 8.28 (br s, 1H), 7.83 – 7.76 (m, 4H), 7.64 (dd, *J* = 8.6, 1.8 Hz, 1H), 7.48 – 7.36 (m, 2H), 6.55 (t, *J* = 3.1 Hz, 1H), 6.04 (t, *J* = 3.0 Hz, 1H), 2.73 (q, *J* = 7.6 Hz, 2H), 1.33 (t, *J* = 7.6 Hz, 3H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 136.21, 133.96, 131.97, 130.68, 130.48, 128.56, 127.81, 127.63, 126.46, 125.19, 123.20, 120.25, 106.90, 106.57, 21.16, 13.69.

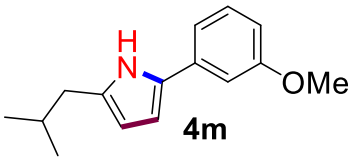
4-(5-Ethyl-1*H*-pyrrol-2-yl)aniline (4l)[5]:



4l

Following the general procedure A, the title product was obtained as a brown oil (42% yield); ^{1}H NMR (500 MHz, $CDCl_3$) δ 8.17 (br s, 1H), 7.29 (d, *J* = 8.5 Hz, 2H), 6.70 (d, *J* = 8.5 Hz, 2H), 6.28 (t, *J* = 3.0 Hz, 1H), 5.97 (t, *J* = 2.9 Hz, 1H), 3.66 (s, 2H), 2.69 (q, *J* = 7.5 Hz, 2H), 1.30 (t, *J* = 7.6 Hz, 3H); ^{13}C NMR (125 MHz, $CDCl_3$) δ 144.51, 134.49, 130.88, 124.92, 124.30, 115.63, 105.77, 104.08, 26.03, 21.00.

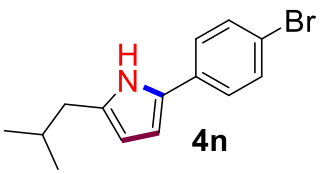
2-Isobutyl-5-(3-methoxyphenyl)-1*H*-pyrrole (4m)[2b]:



4m

Following the general procedure A, the title product was obtained as a pale brown oil (63% yield); ^{1}H NMR (500 MHz, $CDCl_3$) δ 8.11 (br s, 1H), 7.16 – 7.09 (m, 1H), 7.08 – 7.03 (m, 1H), 7.01 – 6.99 (m, 1H), 6.75 (dd, *J* = 8.2, 1.9 Hz, 1H), 6.45 (t, *J* = 3.0 Hz, 1H), 5.99 (t, *J* = 2.9 Hz, 1H), 3.87 (s, 3H), 2.53 (d, *J* = 7.1 Hz, 2H), 1.92 (dp, *J* = 13.5, 6.8 Hz, 1H), 0.99 (d, *J* = 6.6 Hz, 6H); ^{13}C NMR (125 MHz, $CDCl_3$) δ 160.04, 136.70, 134.41, 130.30, 129.85, 115.93, 110.98, 109.23, 106.40, 106.29, 55.28, 37.39, 29.31, 22.49.

2-(4-Bromophenyl)-5-isobutyl-1*H*-pyrrole (4n):

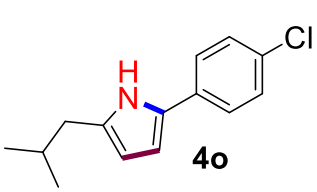


4n

Following the general procedure A, the title product was obtained as a colourless oil (30% yield); ^{1}H NMR (400 MHz, $CDCl_3$) δ 8.04 (br s, 1H), 7.43 (d, *J* = 8.6 Hz, 2H), 7.28 (d, *J* = 8.6 Hz, 2H), 6.42 – 6.38 (m, 1H), 5.95 (t, *J* = 3.0 Hz, 1H), 2.48 (d, *J* = 7.1 Hz, 2H), 1.94

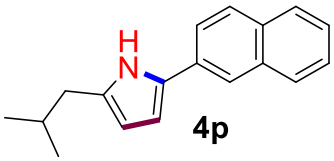
– 1.82 (m, 1H), 0.95 (d, J = 6.6 Hz, 6H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 133.90, 131.92, 128.90, 124.83, 123.38, 118.99, 108.35, 106.74, 37.43, 29.34, 22.56; HRMS (ESI): Calculated for $[C_{14}H_{17}NBr]^+$ 278.0539; Found 278.0546.

2-(4-Chlorophenyl)-5-isobutyl-1*H*-pyrrole (4o):



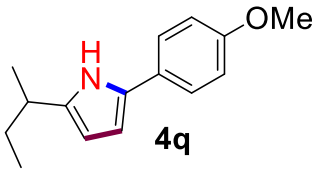
Following the general procedure A, the title product was obtained as a colourless oil (35% yield); ^{1}H NMR (400 MHz, $CDCl_3$) δ 8.02 (br s, 1H), 7.34 (d, J = 8.7 Hz, 2H), 7.28 (d, J = 8.7 Hz, 2H), 6.39 (t, J = 3.1 Hz, 1H), 5.95 (t, J = 3.0 Hz, 1H), 2.49 (d, J = 7.1 Hz, 2H), 1.94 – 1.82 (m, 1H), 0.95 (d, J = 6.6 Hz, 6H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 133.79, 131.59, 131.09, 129.00, 128.31, 124.52, 108.30, 106.68, 37.44, 29.33, 22.54; HRMS (ESI): Calculated for $[C_{14}H_{17}NCl]^+$ 234.1044; Found 234.1041.

2-Isobutyl-5-(naphthalen-2-yl)-1*H*-pyrrole (4p)[5]:



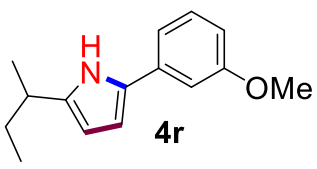
Following the general procedure A, the title product was obtained as a colourless solid (53% yield); ^{1}H NMR (400 MHz, $CDCl_3$) δ 8.25 (br s, 1H), 7.79 (dd, J = 10.7, 6.5 Hz, 4H), 7.67 – 7.60 (m, 1H), 7.49 – 7.35 (m, 2H), 6.58 – 6.53 (m, 1H), 6.05 – 6.00 (m, 1H), 2.55 (d, J = 7.5 Hz, 2H), 2.00 – 1.88 (m, 1H), 1.00 (d, J = 6.8 Hz, 6H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 133.97, 133.85, 131.93, 130.55, 130.50, 128.55, 127.80, 127.60, 126.47, 125.16, 123.17, 120.14, 108.32, 106.96, 37.54, 29.39, 22.60.

2-(*sec*-Butyl)-5-(4-methoxyphenyl)-1*H*-pyrrole (4q):



Following the general procedure A, the title product was obtained as a pale brown oil (50% yield); ^{1}H NMR (400 MHz, $CDCl_3$) δ 8.01 (br s, 1H), 7.37 (d, J = 8.8 Hz, 2H), 6.90 (d, J = 8.8 Hz, 2H), 6.30 (t, J = 3.0 Hz, 1H), 5.95 (t, J = 3.0 Hz, 1H), 3.82 (s, 3H), 2.77 – 2.66 (m, 1H), 1.72 – 1.57 (m, 2H), 1.28 (d, J = 7.0 Hz, 3H), 0.93 (t, J = 7.4 Hz, 3H); ^{13}C NMR (125 MHz, $CDCl_3$) δ 157.89, 138.50, 130.31, 126.31, 124.84, 114.30, 105.38, 104.68, 55.34, 34.42, 30.28, 20.10, 11.87; HRMS (ESI): Calculated for $[C_{15}H_{19}NONa]^+$ 252.1359; Found 252.1363.

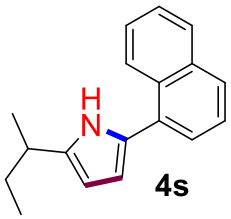
2-(*sec*-Butyl)-5-(3-methoxyphenyl)-1*H*-pyrrole (4r):



Following the general procedure A, the title product was obtained as a light brown oil (65% yield); ^{1}H NMR (400 MHz, $CDCl_3$) δ 8.08 (br s, 1H), 7.25 (t, J = 8.0 Hz, 1H), 7.02 (ddd, J = 7.7, 1.5, 0.9 Hz, 1H), 6.98 – 6.95 (m, 1H), 6.71 (ddd, J = 8.2, 2.5, 0.6 Hz, 1H), 6.43 – 6.38 (m, 1H), 5.96 (t, J = 2.9 Hz, 1H), 3.83 (s, 3H), 2.75 – 2.66 (m, 1H), 1.71 – 1.56 (m, 2H),

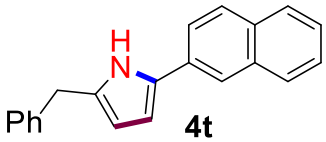
1.28 (d, J = 7.0 Hz, 3H), 0.92 (t, J = 7.4 Hz, 3H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 160.12, 139.33, 134.56, 130.19, 129.90, 116.07, 110.98, 109.44, 106.22, 105.71, 55.35, 34.50, 30.31, 20.14, 11.90; HRMS (ESI): Calculated for $[C_{15}H_{19}NONa]^+$ 252.1359; Found 252.1351.

2-(*sec*-Butyl)-5-(naphthalen-1-yl)-1*H*-pyrrole (4s):



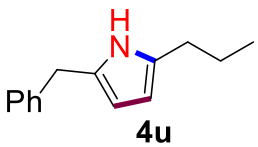
Following the general procedure A, the title product was obtained as a colourless solid (63% yield); ^{1}H NMR (400 MHz, $CDCl_3$) δ 8.39 – 8.34 (m, 1H), 8.12 (br s, 1H), 7.88 (dd, J = 6.1, 3.4 Hz, 1H), 7.78 (dd, J = 6.0, 3.4 Hz, 1H), 7.53 – 7.47 (m, 4H), 6.44 (t, J = 3.0 Hz, 1H), 6.10 (t, J = 3.0 Hz, 1H), 2.84 – 2.73 (m, 1H), 1.79 – 1.58 (m, 2H), 1.34 (d, J = 7.0 Hz, 3H), 0.98 (t, J = 7.4 Hz, 3H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 138.82, 134.19, 131.98, 131.36, 128.79, 128.48, 127.09, 126.27, 125.94, 125.70, 125.55, 109.44, 105.08, 34.43, 30.43, 20.11, 12.00; HRMS (ESI): Calculated for $[C_{18}H_{20}N]^+$ 250.1590; Found 250.1598.

2-Benzyl-5-(naphthalen-2-yl)-1*H*-pyrrole (4t):



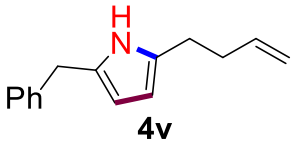
Following the general procedure A, the title product was obtained as a colourless solid (61% yield); ^{1}H NMR (500 MHz, $CDCl_3$) δ 8.25 (br s, 1H), 7.81 (dd, J = 13.1, 5.7 Hz, 3H), 7.76 (s, 1H), 7.64 (dd, J = 8.5, 1.7 Hz, 1H), 7.49 (d, J = 6.8 Hz, 1H), 7.45 – 7.42 (m, 1H), 7.39 (t, J = 7.4 Hz, 2H), 7.34 – 7.30 (m, 3H), 6.61 (t, J = 3.0 Hz, 1H), 6.14 (t, J = 2.9 Hz, 1H), 4.10 (s, 2H); ^{13}C NMR (125 MHz, $CDCl_3$) δ 139.28, 133.81, 132.52, 131.94, 131.56, 130.19, 128.76, 128.73, 128.52, 127.74, 127.59, 126.63, 126.43, 125.22, 123.09, 120.39, 108.95, 106.89, 34.34; HRMS (ESI): Calculated for $[C_{21}H_{18}N]^+$ 284.1434; Found 284.1442.

2-Benzyl-5-propyl-1*H*-pyrrole (4u):



Following the general procedure A, the title product was obtained as a colourless oil (45% yield); ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.48 (br s, 1H), 7.32 – 7.26 (m, 2H), 7.21 (t, J = 6.7 Hz, 3H), 5.85 (t, J = 2.9 Hz, 1H), 5.79 (t, J = 2.9 Hz, 1H), 3.93 (s, 2H), 2.50 – 2.45 (m, 2H), 1.59 (dt, J = 15.0, 7.4 Hz, 2H), 0.93 (t, J = 7.4 Hz, 3H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 139.93, 132.27, 129.00, 128.73, 128.62, 126.38, 106.45, 104.86, 34.28, 29.97, 22.99, 14.00; HRMS (ESI): Calculated for $[C_{14}H_{17}NNa]^+$ 222.1253; Found 222.1261.

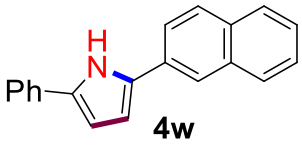
2-Benzyl-5-(but-3-en-1-yl)-1*H*-pyrrole (4v) [7]**:**



4v

Following the general procedure A, the title product was obtained as a pale brown oil (48% yield); ^{1}H NMR (500 MHz, $CDCl_3$) δ 7.61 (br s, 1H), 7.32 (t, J = 7.4 Hz, 2H), 7.26 – 7.22 (m, 3H), 5.90 – 5.76 (m, 1H), 5.60 (dd, J = 16.0, 6.5 Hz, 1H), 5.51 – 5.34 (m, 1H), 5.08 – 4.93 (m, 2H), 3.96 (s, 2H), 2.64 – 2.58 (m, 2H), 2.36 (q, J = 7.2 Hz, 2H); ^{13}C NMR (125 MHz, $CDCl_3$) δ 140.36, 138.86, 130.59, 129.75, 129.26, 129.23, 126.33, 115.00, 106.00, 105.04, 34.94, 34.44, 28.13.

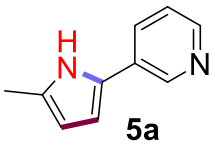
2-(Naphthalen-2-yl)-5-phenyl-1*H*-pyrrole (4w)[2a]**:**



4w

Following the general procedure A, the title product was obtained as a colourless solid (57% yield); ^{1}H NMR (500 MHz, $CDCl_3$) δ 8.34 (br s, 1H), 7.94 (s, 1H), 7.76 (d, J = 6.20 Hz, 1H), 7.56 (d, J = 8.5, 2H), 7.50 (d, J = 8.4 Hz, 1H), 7.40 (d, J = 6.19 Hz, 2H), 7.38 (d, J = 6.6 Hz, 4H), 7.19 (t, J = 5.0 Hz, 1H), 6.66 (t, J = 3.1 Hz, 1H), 6.56 (t, J = 3.0 Hz, 1H); ^{13}C NMR (125 MHz, $CDCl_3$) δ 143.48, 132.81, 132.20, 128.96, 128.13, 127.82, 127.74, 127.34, 127.14, 126.65, 125.42, 125.16, 124.62, 123.50, 123.12, 120.67, 107.77, 107.29.

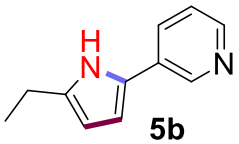
3-(5-Methyl-1*H*-pyrrol-2-yl)pyridine (5a):



5a

Following the general procedure B, the title product was obtained as a pale yellow solid (49% yield); ^{1}H NMR (400 MHz, CDCl*3*) δ 8.72 (d, J = 2.0 Hz, 1H), 8.42 (s, 1H), 8.37 (dd, J = 4.8, 1.5 Hz, 1H), 7.72 – 7.67 (m, 1H), 7.26 – 7.22 (m, 1H), 6.46 (t, J = 3.0 Hz, 1H), 5.98 (t, J= 2.5 Hz, 1H), 2.34 (s, 3H); ^{13}C NMR (125 MHz, $CDCl_3$) δ 146.43, 144.75, 130.39, 128.99, 127.31, 123.64, 115.00, 108.44, 107.67, 13.14; HRMS (ESI): Calculated for $[C_{10}H_{10}N_2Na]^+$ 181.0736; Found 181.0738.

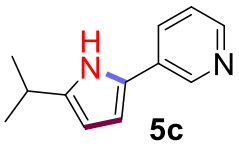
3-(5-Ethyl-1*H*-pyrrol-2-yl)pyridine (5b):



5b

Following the general procedure B, the title product was obtained as a pale brown solid (53% yield); ^{1}H NMR (400 MHz, $CDCl_3$) δ 8.73 (d, J = 1.8 Hz, 1H), 8.48 (s, 1H), 8.37 (dd, J = 4.8, 1.3 Hz, 1H), 7.74 – 7.67 (m, 1H), 7.26 – 7.23 (m, 1H), 6.47 (t, J = 3.1 Hz, 1H), 6.01 (t, J = 3.0 Hz, 1H), 2.74 – 2.63 (m, 2H), 1.29 (t, J = 7.6 Hz, 3H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 146.52, 144.88, 137.08, 130.50, 129.08, 127.22, 123.72, 107.51, 106.77, 21.08, 13.67; HRMS (ESI): Calculated for $[C_{11}H_{13}N_2]^+$ 173.1073; Found 173.1076.

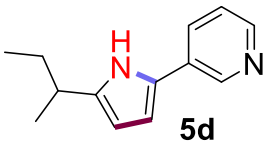
3-(5-Isopropyl-1*H*-pyrrol-2-yl)pyridine (5c):



5c

Following the general procedure B, the title product was obtained as a pale brown solid (52% yield); ^{1}H NMR (400 MHz, $CDCl_3$) δ 8.74 (d, J = 2.3 Hz, 1H), 8.47 (s, 1H), 8.37 (dd, J = 4.8, 1.5 Hz, 1H), 7.73 – 7.68 (m, 1H), 7.27 – 7.22 (m, 1H), 6.49 – 6.45 (m, 1H), 6.01 (t, J = 3.0 Hz, 1H), 3.03 – 2.92 (m, 1H), 1.30 (d, J = 6.9 Hz, 6H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 146.51, 144.97, 141.82, 130.60, 129.14, 127.10, 123.73, 107.36, 105.48, 27.37, 22.75; HRMS (ESI): Calculated for $[C_{12}H_{14}N_2]^+$ 186.1151; Found 186.1116.

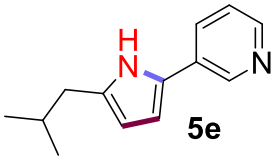
3-(5-(*sec*-Butyl)-1*H*-pyrrol-2-yl)pyridine (5d):



5d

Following the general procedure B, the title product was obtained as a colourless solid (58% yield); ^{1}H NMR (400 MHz, $CDCl_3$) δ 8.72 (d, J = 1.7 Hz, 1H), 8.37 (dd, J = 4.8, 1.5 Hz, 1H), 8.31 (s, 1H), 7.73 – 7.67 (m, 1H), 7.25 – 7.21 (m, 1H), 6.51 – 6.46 (m, 1H), 6.00 (t, J = 3.0 Hz, 1H), 2.77 – 2.68 (m, 1H), 1.68 – 1.58 (m, 2H), 1.28 (d, J = 7.0 Hz, 3H), 0.91 (d, J = 7.4 Hz, 3H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 146.57, 144.90, 140.65, 130.47, 129.08, 126.91, 123.70, 107.37, 106.18, 34.54, 30.29, 20.16, 11.91; HRMS (ESI): Calculated for $[C_{13}H_{17}N_2]^+$ 201.1386; Found 201.1338.

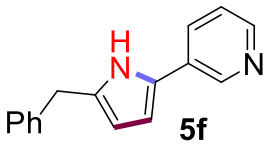
3-(5-Isobutyl-1*H*-pyrrol-2-yl)pyridine (5e):



5e

Following the general procedure B, the title product was obtained as a pale yellow solid (56% yield); ^{1}H NMR (400 MHz, $CDCl_3$) δ 8.73 (d, J = 2.1 Hz, 1H), 8.42 (s, 1H), 8.36 (dd, J = 4.8, 1.5 Hz, 1H), 7.73 – 7.67 (m, 1H), 7.26 – 7.21 (m, 1H), 6.48 (t, J = 3.1 Hz, 1H), 5.99 (t, J = 3.0 Hz, 1H), 2.51 (d, J = 7.1 Hz, 2H), 1.95 – 1.83 (m, 1H), 0.95 (d, J = 6.6 Hz, 6H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 146.49, 144.89, 134.68, 130.42, 129.08, 127.11, 123.71, 108.50, 107.56, 37.41, 29.32, 22.52; HRMS (ESI): Calculated for $[C_{13}H_{17}N_2]^+$ 201.1386; Found 201.1354.

3-(5-Benzyl-1*H*-pyrrol-2-yl)pyridine (5f):

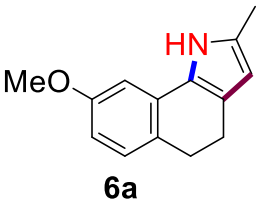


5f

Following the general procedure B, the title product was obtained as a colourless solid (45% yield); ^{1}H NMR (400 MHz, $CDCl_3$) δ 8.65 (ddd, J = 3.2, 2.4, 1.0 Hz, 1H), 8.44 – 8.35 (m, 1H), 8.33 (dd, J = 2.7, 1.8 Hz, 1H), 7.68 – 7.61 (m, 1H), 7.7734 – 7.28 (m, 2H), 7.25 – 7.18 (m, 4H), 6.51 – 6.46 (m, 1H), 6.10 – 6.04 (m, 1H), 4.02 (s, 2H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 146.79,

145.03, 139.06, 133.44, 130.55, 128.86, 128.83, 128.73, 128.16, 126.74, 123.68, 109.09, 107.56, 34.29; HRMS (ESI): Calculated for $[C_{16}H_{15}N_2]^+$ 235.1230; Found 235.1247.

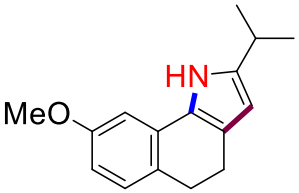
8-Methoxy-2-methyl-4,5-dihydro-1*H*-benzo[*g*]indole (6a):



6a

Following the general procedure A, the title product was obtained as a pale brown oil (69% yield); ^{1}H NMR (400 MHz, $CDCl_3$) δ 8.01 (s, 1H), 7.09 (d, J = 8.2 Hz, 1H), 6.67 (d, J = 2.5 Hz, 1H), 6.57 (dd, J = 8.2, 2.5 Hz, 1H), 5.81 (d, J = 1.4 Hz, 1H), 3.81 (s, 3H), 2.84 (t, J = 7.6 Hz, 2H), 2.67 (t, J = 7.6 Hz, 2H), 2.32 (s, 3H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 158.58, 130.54, 128.95, 126.85, 126.54, 121.54, 108.88, 106.46, 106.43, 104.45, 55.44, 29.33, 22.24, 13.41; HRMS (ESI): Calculated for $[C_{14}H_{16}NO]^+$ 214.1226; Found 214.1243.

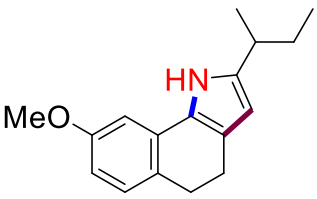
2-Isopropyl-8-methoxy-4,5-dihydro-1*H*-benzo[*g*]indole (6b):



6b

Following the general procedure A, the title product was obtained as a pale brown oil (70% yield); ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.98 (s, 1H), 7.06 (d, J = 8.1 Hz, 1H), 6.66 (d, J = 2.5 Hz, 1H), 6.54 (dd, J = 8.2, 2.6 Hz, 1H), 5.83 (d, J = 2.4 Hz, 1H), 3.81 (s, 3H), 2.83 (t, J = 7.6 Hz, 2H), 2.66 (t, J = 7.6 Hz, 2H), 2.17 – 2.01 (m, 1H), 1.29 (d, J = 6.9 Hz, 6H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 158.56, 140.24, 130.51, 128.92, 126.87, 126.21, 121.00, 108.86, 104.42, 103.44, 55.46, 29.27, 27.46, 22.78, 22.24; HRMS (ESI): Calculated for $[C_{16}H_{20}NO]^+$ 242.1539; Found 242.1548.

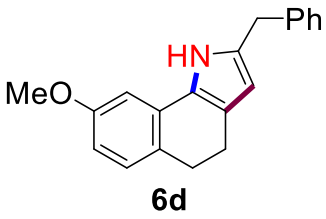
2-(*sec*-Butyl)-8-methoxy-4,5-dihydro-1*H*-benzo[*g*]indole (6c):



6c

Following the general procedure A, the title product was obtained as a pale brown oil (65% yield); ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.92 (s, 1H), 7.06 (d, J = 8.2 Hz, 1H), 6.64 (d, J = 2.5 Hz, 1H), 6.53 (dd, J = 8.2, 2.5 Hz, 1H), 5.81 (d, J = 2.3 Hz, 1H), 3.80 (s, 3H), 2.83 (t, J = 7.6 Hz, 2H), 2.72 – 2.62 (m, 3H), 1.70 – 1.57 (m, 2H), 1.27 (d, J = 7.0 Hz, 3H), 0.92 (t, J = 7.4 Hz, 3H); ^{13}C NMR (125 MHz, $CDCl_3$) δ 158.51, 139.07, 130.50, 128.84, 126.75, 125.95, 120.99, 108.73, 104.31, 104.05, 55.38, 34.60, 30.29, 29.18, 22.20, 20.11, 11.90: HRMS (ESI): Calculated for $[C_{17}H_{22}NO]^+$ 256.1696; Found 256.1634.

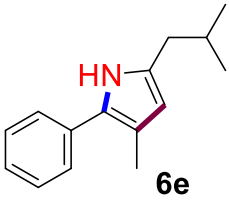
2-Benzyl-8-methoxy-4,5-dihydro-1*H*-benzo[*g*]indole (6d):



6d

Following the general procedure A, the title product was obtained as a pale brown oil (64% yield); ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.89 (s, 1H), 7.36 – 7.28 (m, 2H), 7.28 – 7.23 (m, 3H), 7.06 (d, J = 8.1 Hz, 1H), 6.59 – 6.52 (m, 2H), 5.87 (d, J = 2.1 Hz, 1H), 4.00 (s, 2H), 3.77 (s, 3H), 2.83 (t, J = 7.6 Hz, 2H), 2.66 (t, J = 7.6 Hz, 2H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 167.17, 139.45, 131.84, 128.95, 128.80, 128.76, 126.88, 126.64, 121.25, 109.81, 109.06, 107.05, 107.02, 104.54, 55.41, 34.48, 29.21, 22.17; HRMS (ESI): Calculated for $[C_{20}H_{19}NONa]^+$ 312.1359; Found 312.1327.

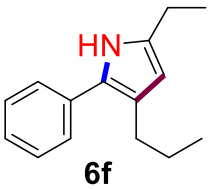
5-Isobutyl-3-methyl-2-phenyl-1*H*-pyrrole (6e) [3]:



6e

Following the general procedure A, the title product was obtained as a pale brown oil (53% yield); ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.76 (s, 1H), 7.41 – 7.34 (m, 4H), 7.21 – 7.16 (m, 1H), 5.83 (d, J = 2.8 Hz, 1H), 2.45 (d, J = 7.0 Hz, 2H), 2.24 (s, 3H), 1.92 – 1.80 (m, 1H), 0.96 (d, J = 6.6 Hz, 6H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 134.06, 131.74, 128.72, 126.49, 125.90, 125.43, 116.38, 110.40, 37.35, 29.34, 22.63, 12.72.

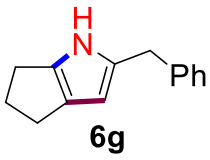
5-Ethyl-2-phenyl-3-propyl-1*H*-pyrrole (6f) [6]:



6f

Following the general procedure A, the title product was obtained as a pale brown oil (48% yield); ^{1}H NMR (500 MHz, $CDCl_3$) δ 7.74 (s, 1H), 7.43 – 7.35 (m, 4H), 7.25 – 7.17 (m, 1H), 5.92 (d, J = 2.7 Hz, 1H), 2.72 – 2.63 (m, 2H), 2.63 – 2.56 (m, 2H), 1.70 – 1.62 (m, 2H), 1.29 (t, J = 7.6 Hz, 3H), 0.89 (t, J = 7.1 Hz, 3H); ^{13}C NMR (125 MHz, $CDCl_3$) δ 134.16, 134.13, 128.40, 126.53, 126.47, 125.51, 121.60, 106.83, 28.91, 24.40, 20.91, 14.37, 13.55.

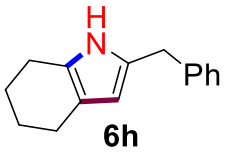
2-Benzyl-1,4,5,6-tetrahydrocyclopenta[*b*]pyrrole (6g) [4]:



6g

Following the general procedure A, the title product was obtained as a pale brown oil (45% yield); ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.42 (s, 1H), 7.33 – 7.18 (m, 5H), 5.70 (s, 1H), 3.92 (s, 2H), 2.79 – 2.67 (m, 4H), 2.34 – 2.21 (m, 2H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 141.43, 135.83, 134.59, 128.72, 126.38, 126.32, 102.35, 35.37, 29.43, 26.98, 25.03.

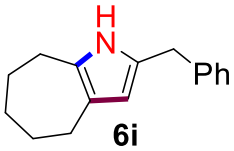
2-Benzyl-4,5,6,7-tetrahydro-1*H*-indole (6h) [4]**:**



6h

Following the general procedure A, the title product was obtained as a pale brown oil (30% yield); ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.34 – 7.19 (m, 6H), 5.69 (s, 1H), 3.90 (s, J = 7.3 Hz, 2H), 2.53 – 2.36 (m, 4H), 2.05 – 1.99 (m, 4H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 140.14, 128.88, 128.61, 128.09, 126.37, 126.26, 116.93, 105.78, 34.41, 23.90, 22.92, 22.75, 22.56.

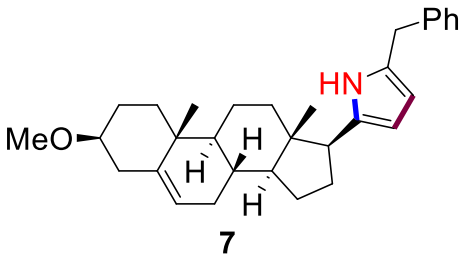
2-Benzyl-1,4,5,6,7,8-hexahydrocyclohepta[*b*]pyrrole (6i) [4]**:**



6i

Following the general procedure A, the title product was obtained as a pale brown oil (35% yield); ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.31 (s, 1H), 7.33 – 7.17 (m, 5H), 5.67 (d, J = 3.0 Hz, 1H), 3.86 (s, J = 7.4 Hz, 2H), 2.54 – 2.48 (m, 4H), 1.82 – 1.77 (m, 2H), 1.68 – 1.64 (m, 4H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 140.36, 129.89, 128.81, 128.76, 126.41, 126.34, 121.52, 108.60, 34.30, 32.91, 30.96, 29.53, 28.24, 28.13.

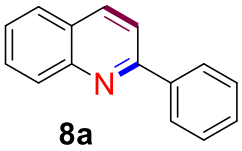
2-Benzyl-5-((3*S*,8*S*,9*S*,10*R*,13*S*,14*S*,17*S*)-3-methoxy-10,13-dimethyl-2,3,4,7,8,9,10,11, 12, 13,14,15,16,17-tetradecahydro-1*H*-cyclopenta[*a*]phenanthren-17-yl)-1*H*-pyrrole (7):



7

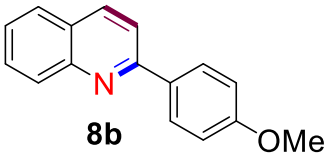
Following the general procedure C, the title product was obtained as a pale yellow oil (35% yield); ^{1}H NMR (500 MHz, $CDCl_3$) δ 7.53 (s, 1H), 7.33 (dd, J = 10.2, 4.6 Hz, 2H), 7.23 (dd, J = 15.3, 7.7 Hz, 3H), 5.90 (t, J = 2.8 Hz, 1H), 5.87 (t, J = 2.9 Hz, 1H), 5.40 – 5.37 (m, 1H), 3.97 (s, 2H), 3.38 (s, 3H), 3.09 (ddd, J = 11.3, 6.8, 4.6 Hz, 1H), 2.55 (t, J = 9.8 Hz, 1H), 2.44 – 2.40 (m, 1H), 2.22 – 2.15 (m, 2H), 2.04 – 1.94 (m, 3H), 1.92 – 1.87 (m, 2H), 1.76 – 1.70 (m, 2H), 1.61 – 1.56 (m, 2H), 1.47 (ddd, J = 12.4, 9.7, 4.8 Hz, 3H), 1.31 – 1.27 (m, 1H), 1.26 – 1.21 (m, 1H), 1.14 – 1.07 (m, 2H), 1.02 (s, 3H), 0.52 (s, 3H); ^{13}C NMR (125 MHz, $CDCl_3$) δ 140.95, 139.94, 132.28, 128.80, 128.55, 128.51, 126.27, 121.42, 106.23, 105.39, 80.31, 55.84, 55.61, 50.41, 50.15, 43.58, 38.70, 37.98, 37.23, 37.00, 34.17, 32.32, 31.89, 28.00, 26.26, 24.27, 20.92, 19.42, 12.97; HRMS (ESI): Calculated for $[C_{31}H_{42}NO]^{+}$ 444.3261; Found 444.3160.

2-Phenylquinoline (8a) [5]**:**



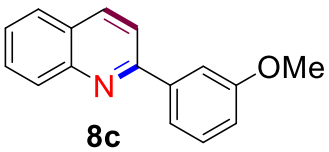
Following the general procedure A, the title product was obtained as a colourless solid (75% yield); ^{1}H NMR (500 MHz, $CDCl_3$) δ 8.26 (d, J = 8.6 Hz, 1H), 8.22 – 8.18 (m, 3H), 7.91 (d, J = 8.6 Hz, 1H), 7.86 (d, J = 7.9 Hz, 1H), 7.76 (t, J = 4.9 Hz, 1H), 7.58 – 7.54 (m, 3H), 7.50 (t, J = 2.1 Hz, 1H); ^{13}C NMR (125 MHz, $CDCl_3$) δ 157.41, 148.31, 139.72, 136.75, 129.69, 129.34, 129.33, 128.87, 127.66, 127.49, 127.20, 126.31, 119.07.

2-(4-Methoxyphenyl)quinoline (8b)[8]**:**



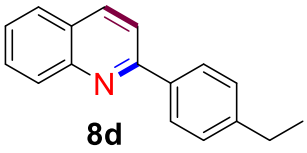
Following the general procedure A, the title product was obtained as a colourless solid (70% yield); ^{1}H NMR (400 MHz, $CDCl_3$) δ 8.20 – 8.11 (m, 4H), 7.81 (dd, J = 13.1, 8.4 Hz, 2H), 7.70 (t, J = 7.7 Hz, 1H), 7.49 (t, J = 7.5 Hz, 1H), 7.04 (d, J = 8.8 Hz, 2H), 3.88 (s, 3H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 160.89, 157.00, 148.37, 136.71, 132.35, 129.64, 128.96, 127.51, 126.99, 125.97, 118.64, 114.26, 55.48.

2-(3-Methoxyphenyl)quinoline (8c) [5]**:**



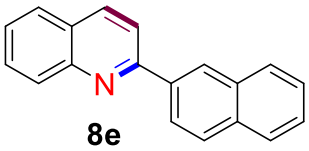
Following the general procedure A, the title product was obtained as a colourless oil (90% yield); ^{1}H NMR (500 MHz, $CDCl_3$) δ 8.23 (t, J = 8.5 Hz, 2H), 7.87 (dd, J = 18.2, 8.3 Hz, 2H), 7.81 (s, 1H), 7.75 (dd, J = 14.8, 7.9 Hz, 2H), 7.56 (t, J = 7.4 Hz, 1H), 7.47 (t, J = 7.9 Hz, 1H), 7.05 (dd, J = 8.1, 2.3 Hz, 1H), 3.96 (s, 3H); ^{13}C NMR (125 MHz, $CDCl_3$) δ 160.16, 157.17, 148.24, 141.19, 136.70, 129.69, 127.49, 127.29, 126.36, 120.04, 119.16, 119.11, 115.40, 112.76, 112.70, 55.48.

2-(4-Ethylphenyl)quinoline (8d) [8]**:**



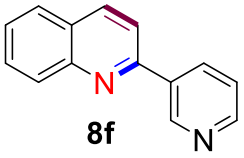
Following the general procedure A, the title product was obtained as a colourless solid (74% yield); ^{1}H NMR (400 MHz, $CDCl_3$) δ 8.17 (t, J = 8.0 Hz, 2H), 8.09 (d, J = 6.8 Hz, 2H), 7.85 (d, J = 8.7 Hz, 1H), 7.81 (d, J = 8.2 Hz, 1H), 7.71 (t, J = 7.7 Hz, 1H), 7.50 (t, J = 7.5 Hz, 1H), 7.36 (d, J = 7.8 Hz, 2H), 2.73 (q, J = 7.6 Hz, 2H), 1.29 (t, J = 7.6 Hz, 3H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 157.50, 148.41, 145.83, 137.26, 136.73, 129.77, 129.65, 128.45, 127.64, 127.53, 127.19, 126.15, 119.00, 28.82, 15.66.

2-(Naphthalen-2-yl)quinoline (8e)[8]:



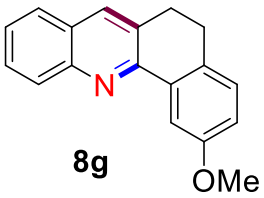
Following the general procedure A, the title product was obtained as a colourless solid (70% yield); ^{1}H NMR (500 MHz, $CDCl_3$) δ 8.65 (s, 1H), 8.41 (dd, J = 8.6, 1.7 Hz, 1H), 8.27 (dd, J = 12.5, 8.6 Hz, 2H), 8.05 (dd, J = 15.8, 8.2 Hz, 3H), 7.93 (dd, J = 5.9, 3.4 Hz, 1H), 7.88 (d, J = 7.9 Hz, 1H), 7.79 (ddd, J = 8.3, 6.9, 1.3 Hz, 1H), 7.57 (dd, J = 6.4, 2.9 Hz, 3H); ^{13}C NMR (125 MHz, $CDCl_3$) δ 157.20, 148.39, 136.99, 136.91, 133.88, 133.52, 129.78, 128.86, 128.62, 128.46, 127.76, 127.54, 127.25, 127.13, 126.75, 126.37, 119.19, 115.00.

2-(Pyridin-3-yl)quinoline (8f)[9]:



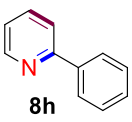
Following the general procedure A, the title product was obtained as a colourless solid (58% yield); ^{1}H NMR (400 MHz, $CDCl_3$) δ 9.34 (d, J = 1.6 Hz, 1H), 8.69 (dd, J = 4.8, 1.5 Hz, 1H), 8.53 – 8.48 (m, 1H), 8.26 (d, J = 8.5 Hz, 1H), 8.17 (d, J = 8.5 Hz, 1H), 7.86 (dd, J = 12.8, 8.4 Hz, 2H), 7.75 (ddd, J = 8.4, 6.9, 1.4 Hz, 1H), 7.55 (ddd, J = 8.1, 6.9, 1.1 Hz, 1H), 7.45 (dd, J = 7.3, 4.8 Hz, 1H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 154.72, 150.29, 148.90, 148.45, 137.29, 135.23, 135.07, 130.10, 129.83, 127.65, 127.46, 126.89, 123.76, 118.62.

2-Methoxy-5,6-dihydrobenzo[*c*]acridine (8g):



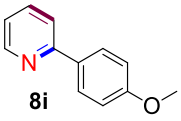
Following the general procedure A, the title product was obtained as a colourless solid (71% yield); ^{1}H NMR (400 MHz, $CDCl_3$) δ 8.17 – 8.11 (t, 2H), 7.90 (s, 1H), 7.73 (d, J = 8.1 Hz, 1H), 7.64 (dd, J = 8.2, 7.1 Hz, 1H), 7.47 (dd, J = 8.0, 7.0 Hz, 1H), 7.18 (d, J = 8.3 Hz, 1H), 6.94 (dd, J = 8.3, 2.8 Hz, 1H), 3.96 (s, 3H), 3.12 – 3.06 (t, 2H), 2.96 – 2.90 (t, 2H): ^{13}C NMR (100 MHz, $CDCl_3$) δ 159.16, 153.37, 147.63, 135.76, 133.80, 131.96, 130.77, 129.50, 129.09, 128.71, 128.00, 127.00, 126.18, 117.03, 109.73, 55.67, 29.19, 27.64; HRMS (ESI): Calculated for $[C_{18}H_{16}NO]^{+}$ 262.1226; Found 262.1219.

2-phenylpyridine (8h)[5]:



Following the general procedure A, the title product was obtained as a colourless oil (55% yield); ^{1}H NMR (500 MHz, $CDCl_3$) δ 8.73 (d, J = 4.5 Hz, 1H), 8.04 (dd, J = 6.1, 1.4 Hz, 2H), 7.77 (t, J = 8.6 Hz, 2H), 7.54 – 7.47 (m, 4H); ^{13}C NMR (125 MHz, $CDCl_3$) δ 157.51, 145.19, 139.43, 136.79, 128.81, 128.54, 126.94, 122.13, 120.62.

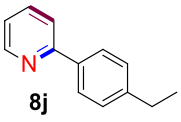
2-(4-Methoxyphenyl)pyridine (8i) [5]**:**



8i

Following the general procedure A, the title product was obtained as a colourless oil (49% yield); ^{1}H NMR (500 MHz, $CDCl_3$) δ 8.68 (d, J = 4.1 Hz, 1H), 8.00 – 7.95 (m, 2H), 7.73 (ddd, J = 20.6, 13.4, 4.9 Hz, 1H), 7.20 (ddd, J = 7.2, 4.8, 1.2 Hz, 1H), 7.03 (d, J = 8.9 Hz, 2H), 6.96 (d, J = 8.9 Hz, 1H), 3.89 (s, 3H); ^{13}C NMR (125 MHz, $CDCl_3$) δ 160.45, 157.14, 149.50, 136.71, 132.03, 128.18, 121.43, 119.85, 114.12, 55.36.

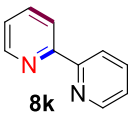
2-(4-Ethylphenyl)pyridine (8j) [10]**:**



8j

Following the general procedure A, the title product was obtained as a colourless oil (53% yield); ^{1}H NMR (500 MHz, $CDCl_3$) δ 8.70 (d, J = 4.6 Hz, 1H), 7.94 (d, J = 8.2 Hz, 1H), 7.79 – 7.72 (m, 1H), 7.34 (d, J = 8.2 Hz, 1H), 7.31 – 7.14 (m, 4H), 2.67 (dd, J = 15.6, 7.8 Hz, 2H), 1.32 – 1.29 (m, 3H); ^{13}C NMR (125 MHz, $CDCl_3$) δ 157.55, 145.30, 136.87, 128.31, 126.86, 125.93, 121.79, 120.32, 115.00, 28.66, 15.53.

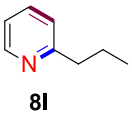
2,2'-Bipyridine (8k) [11]**:**



8k

Following the general procedure A, the title product was obtained as a colourless solid (48% yield);^{1}H NMR (500 MHz, $CDCl_3$) δ 8.70 (d, J = 4.0 Hz, 2H), 8.41 (t, J = 6.5 Hz, 2H), 7.83 (td, J = 7.8, 1.8 Hz, 2H), 7.32 (ddd, J = 7.3, 4.7, 1.0 Hz, 2H); ^{13}C NMR (125 MHz, $CDCl_3$) δ 156.17, 149.33, 136.97, 123.74, 121.12.

2-Propylpyridine (8l) [12]**:**



8l

Following the general procedure A, the title product was obtained as a colourless oil (50% yield);^{1}H NMR (500 MHz, $CDCl_3$) δ 8.54 (d, J = 4.1 Hz, 1H), 7.60 (td, J = 7.7, 1.8 Hz, 1H), 7.16 (d, J = 7.8 Hz, 1H), 7.11 (dd, J = 6.9, 5.4 Hz, 1H), 2.81 – 2.77 (m, 2H), 1.81 – 1.72 (m, 2H), 0.90 (t, J = 6.9 Hz, 3H); ^{13}C NMR (125 MHz, $CDCl_3$) δ 162.30, 149.17, 136.19, 122.75, 120.88, 40.40, 23.13, 13.84.

[1.6] References:

1. S. Michlik and R. Kempe, *Nature Chemistry*, 2013, **5**, 140.
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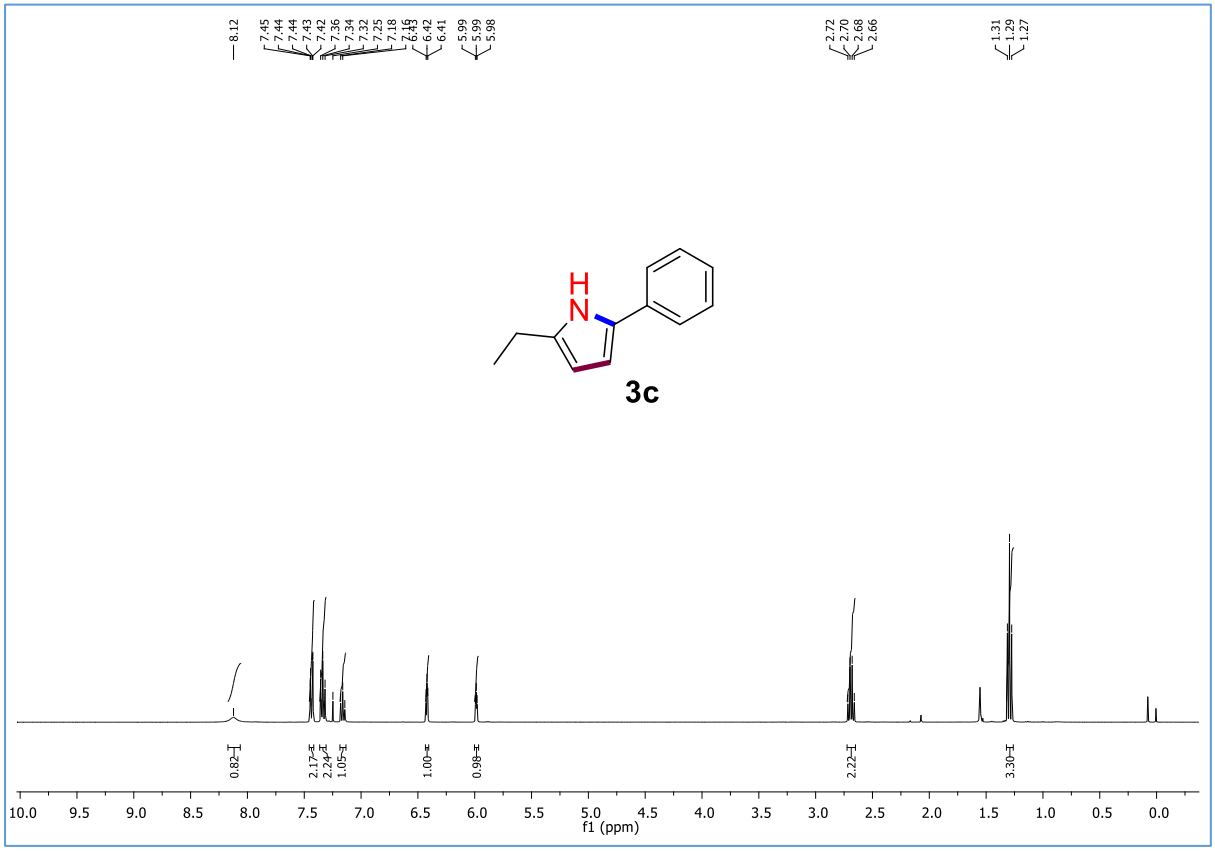
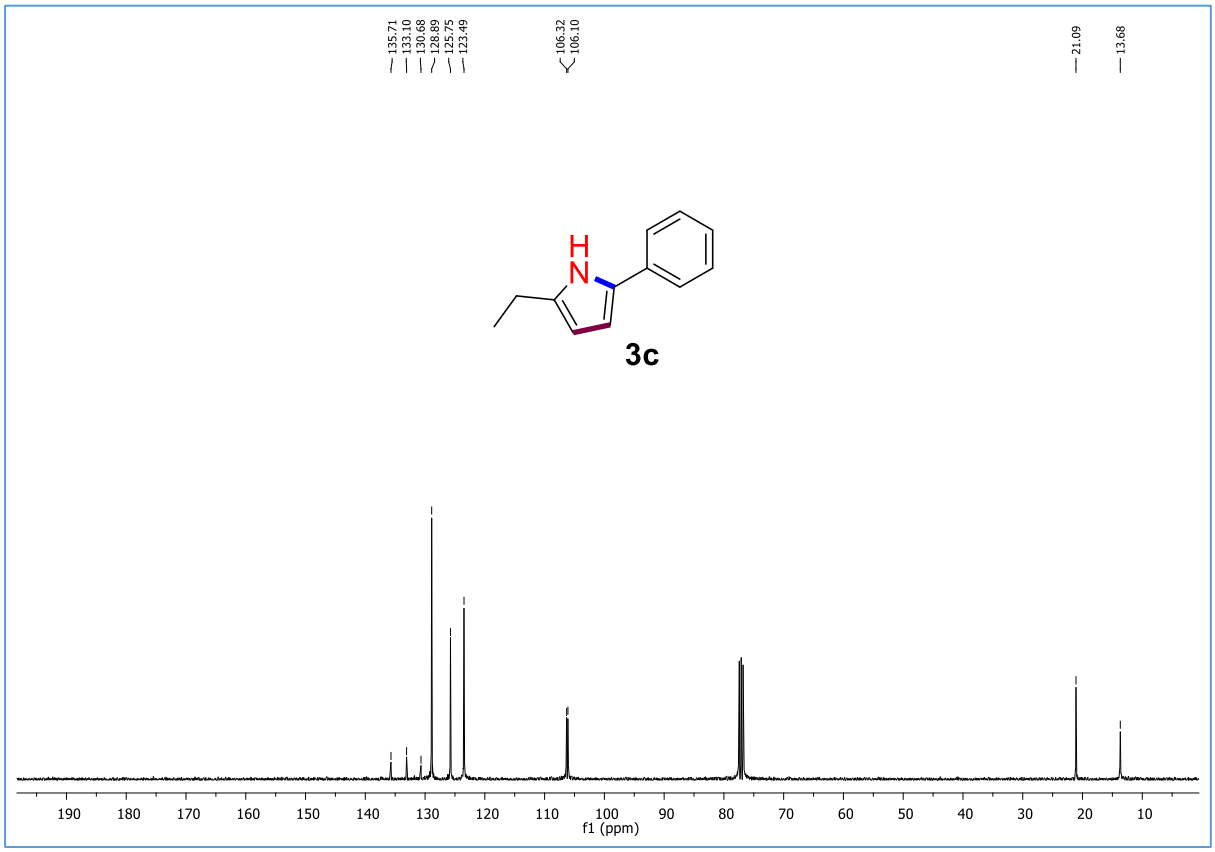
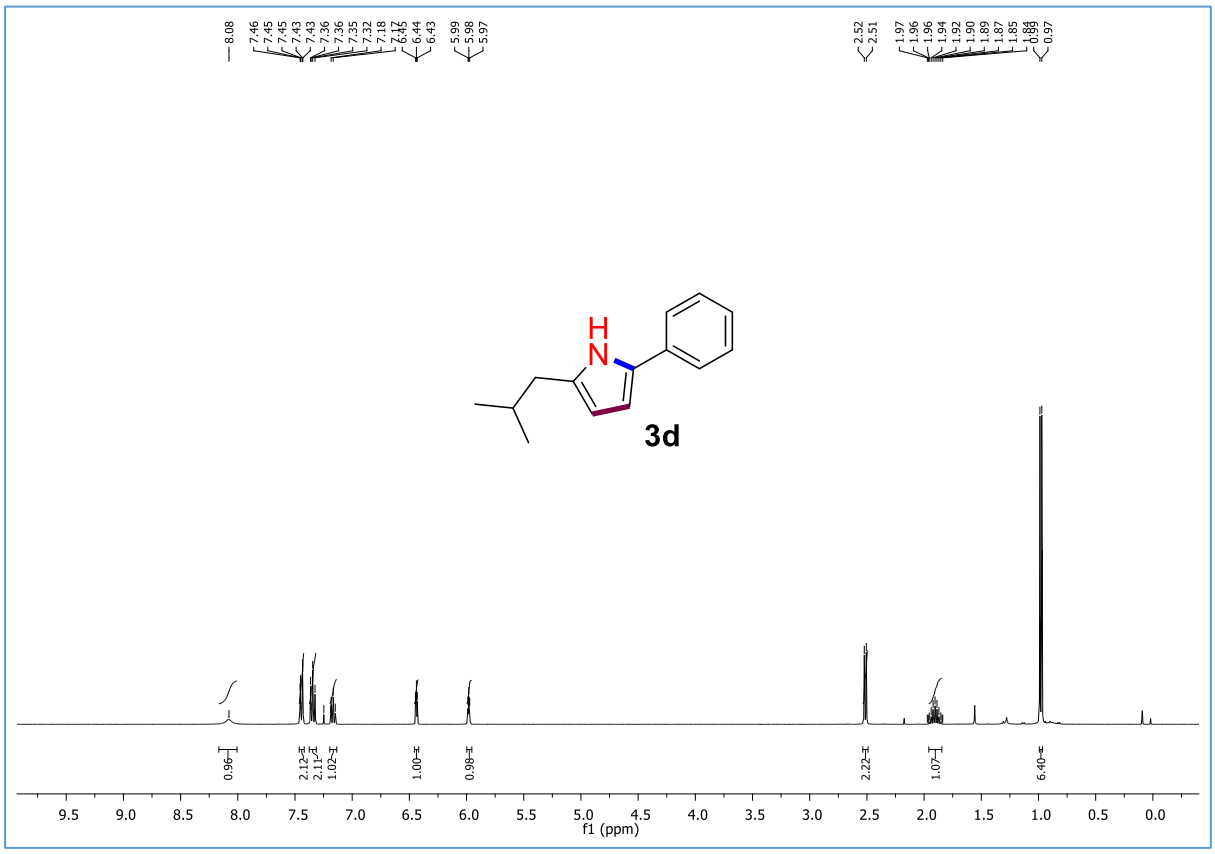
[1.7] Copies of ^{1}H and ^{13}C NMR spectra for selected compounds.



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1.96
1.94
1.92
1.90
1.89
1.87
1.85
0.99
0.97
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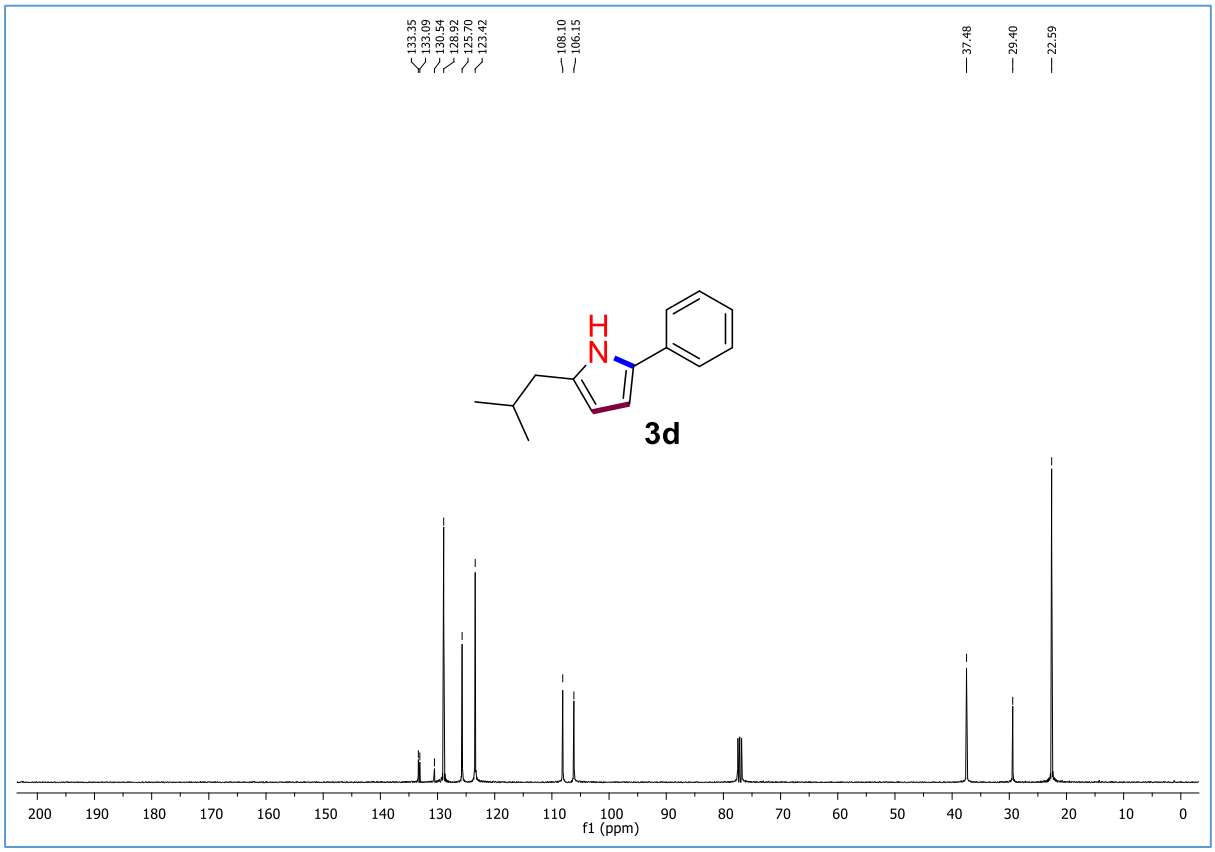
f1 (ppm)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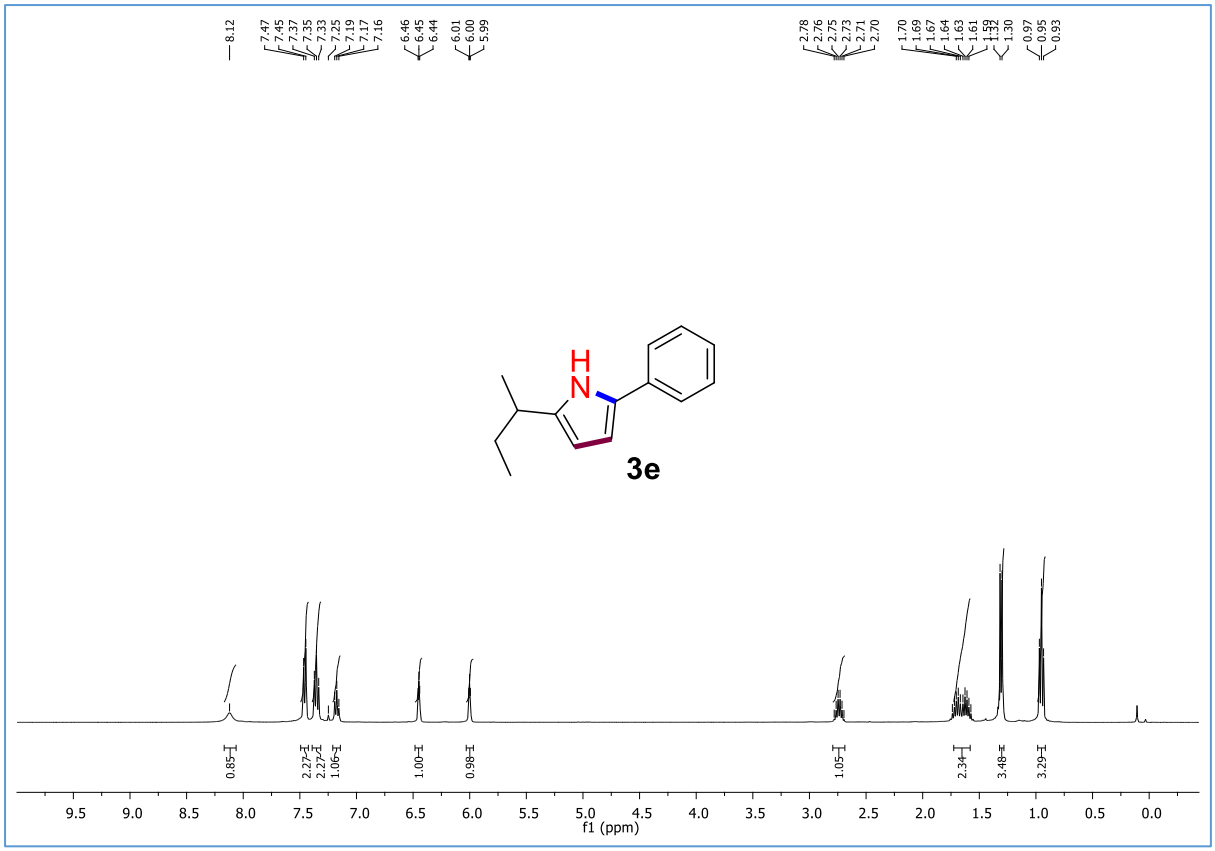
3d
f1 (ppm)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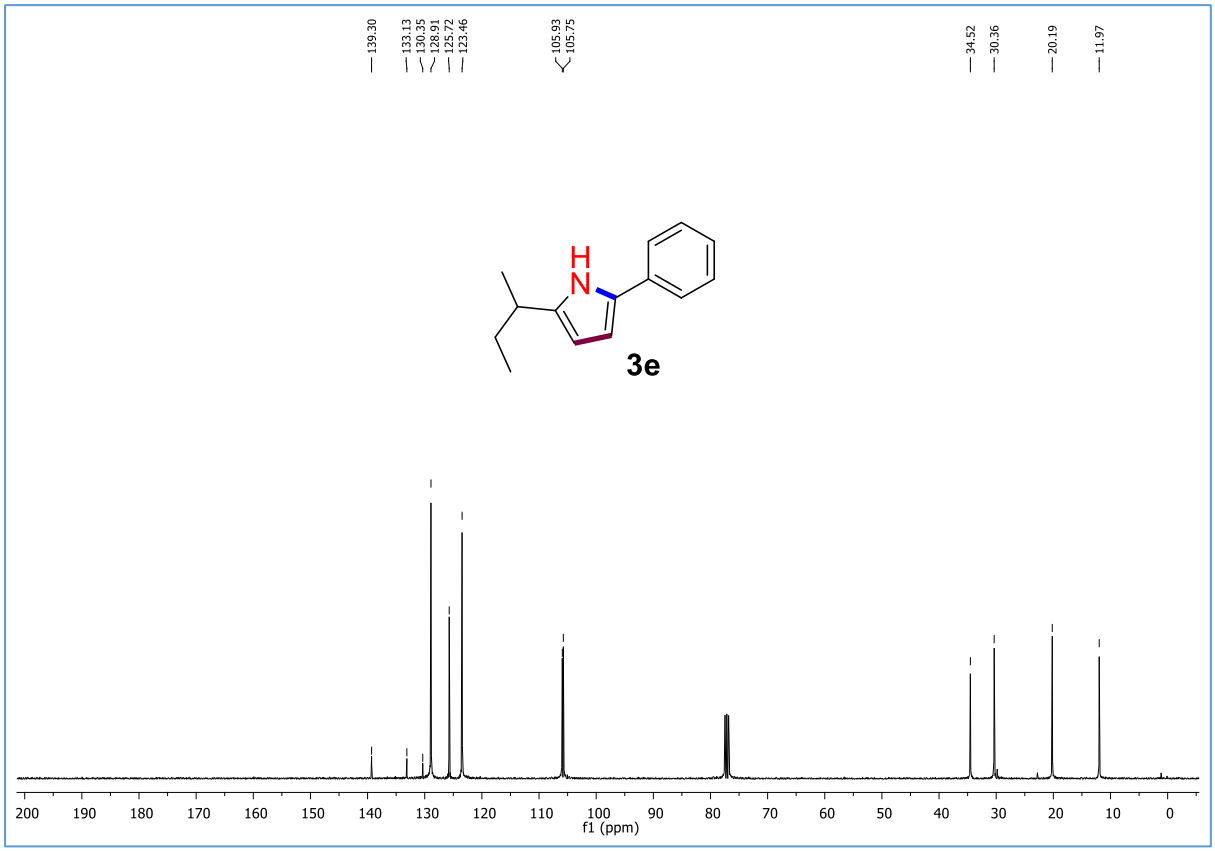
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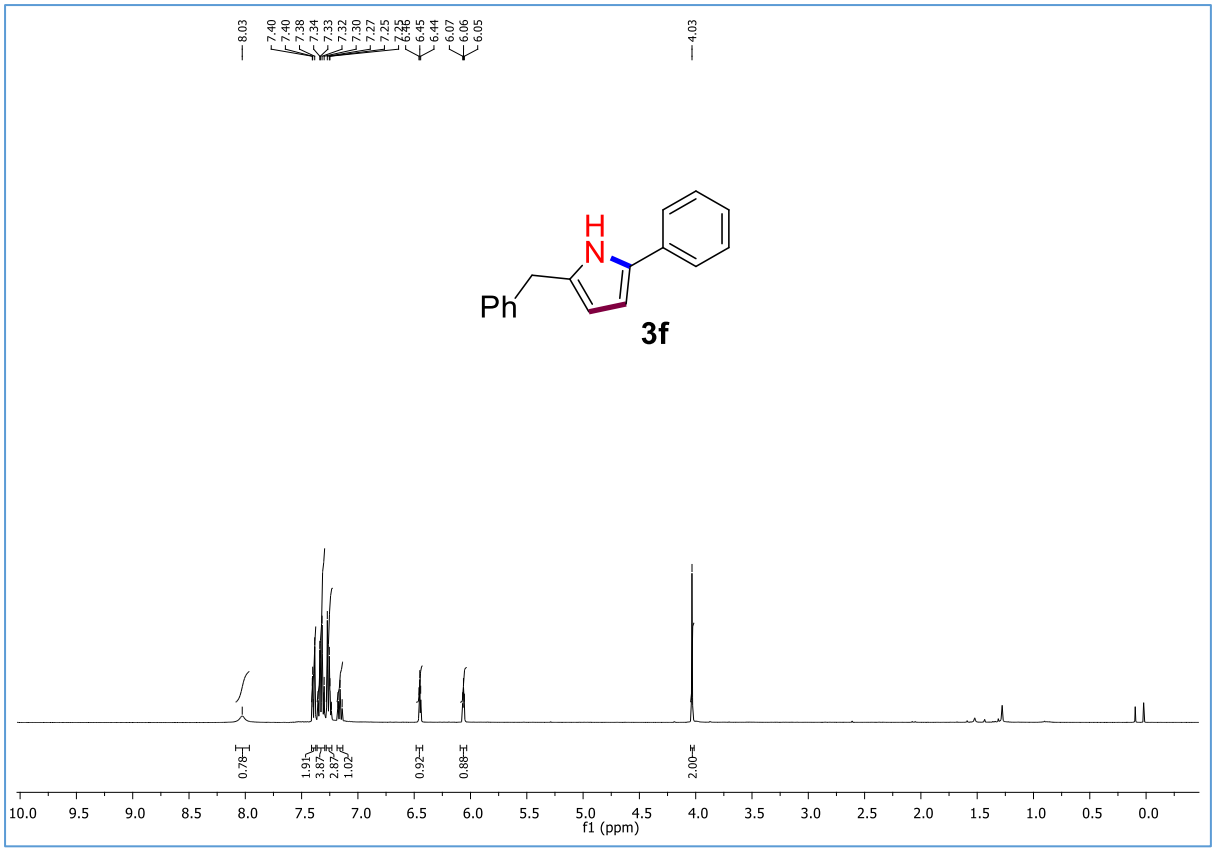
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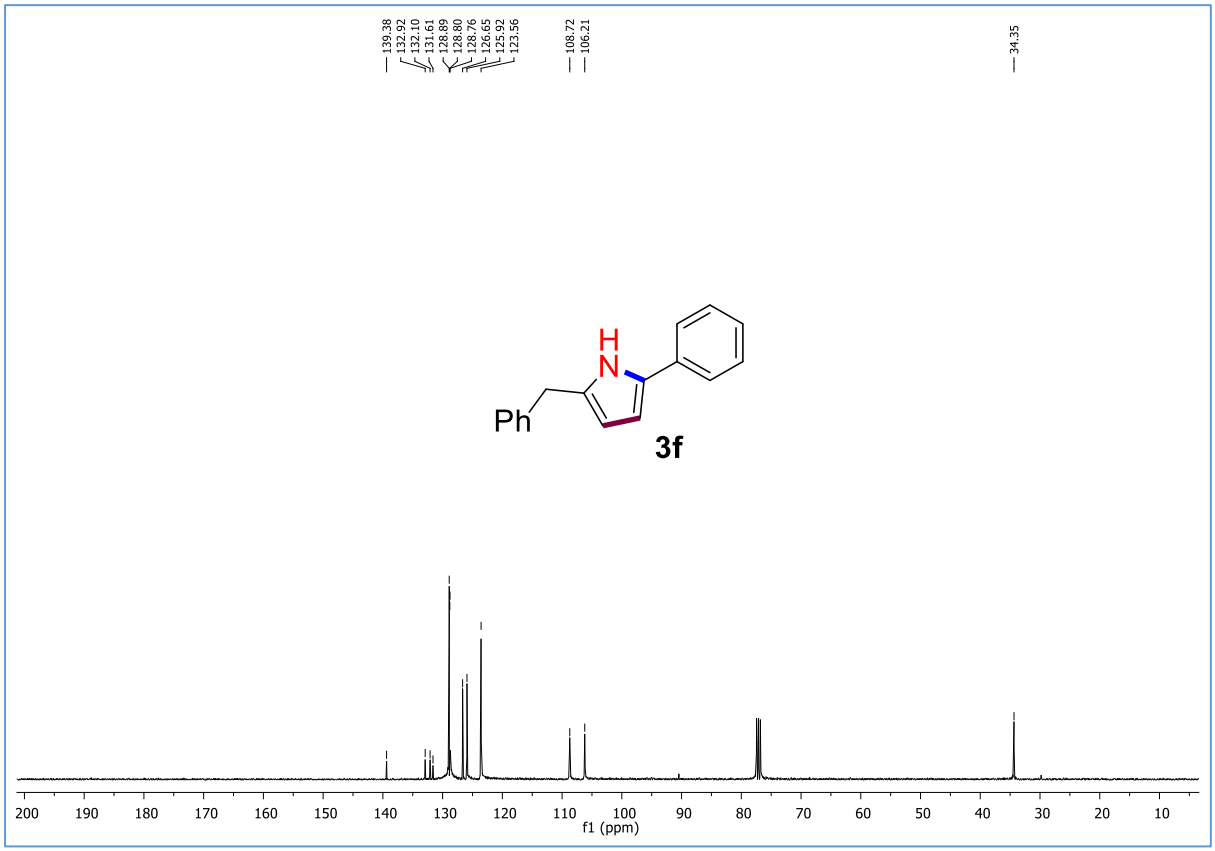
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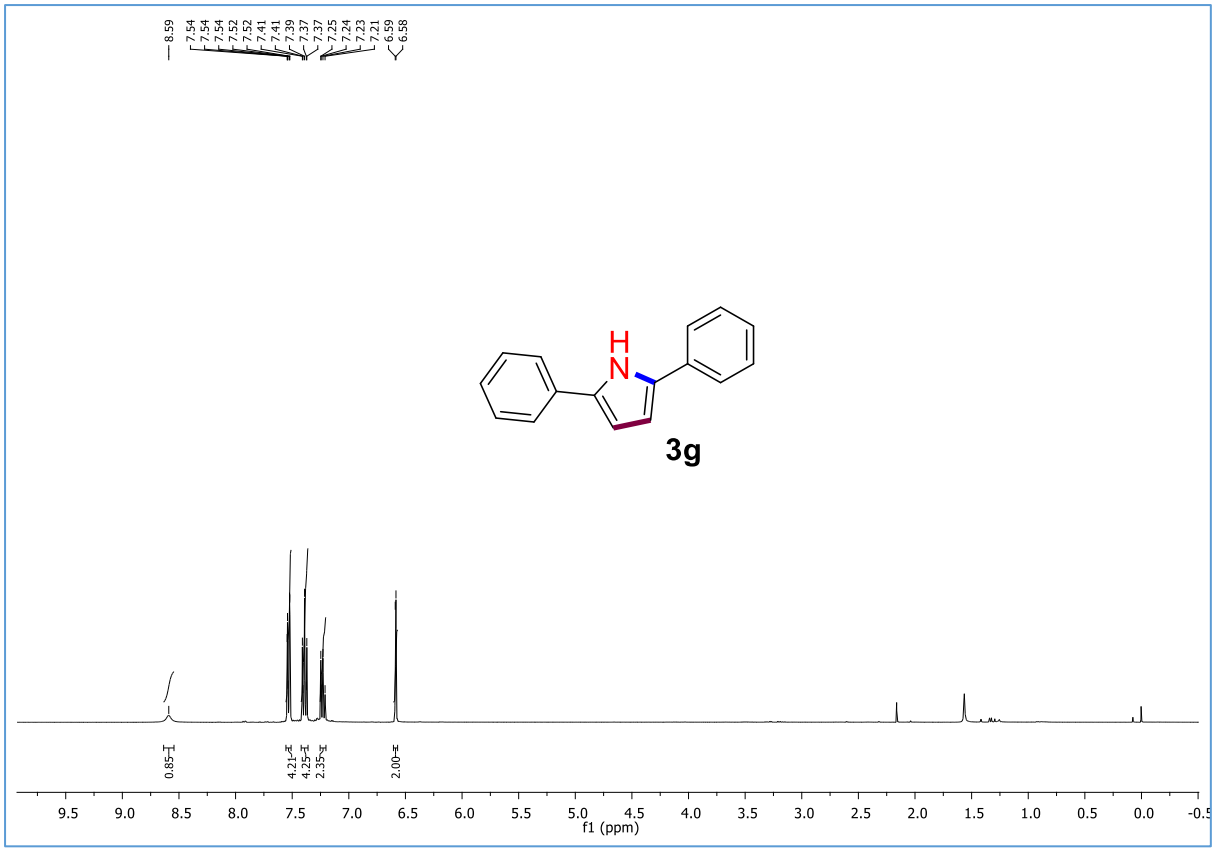
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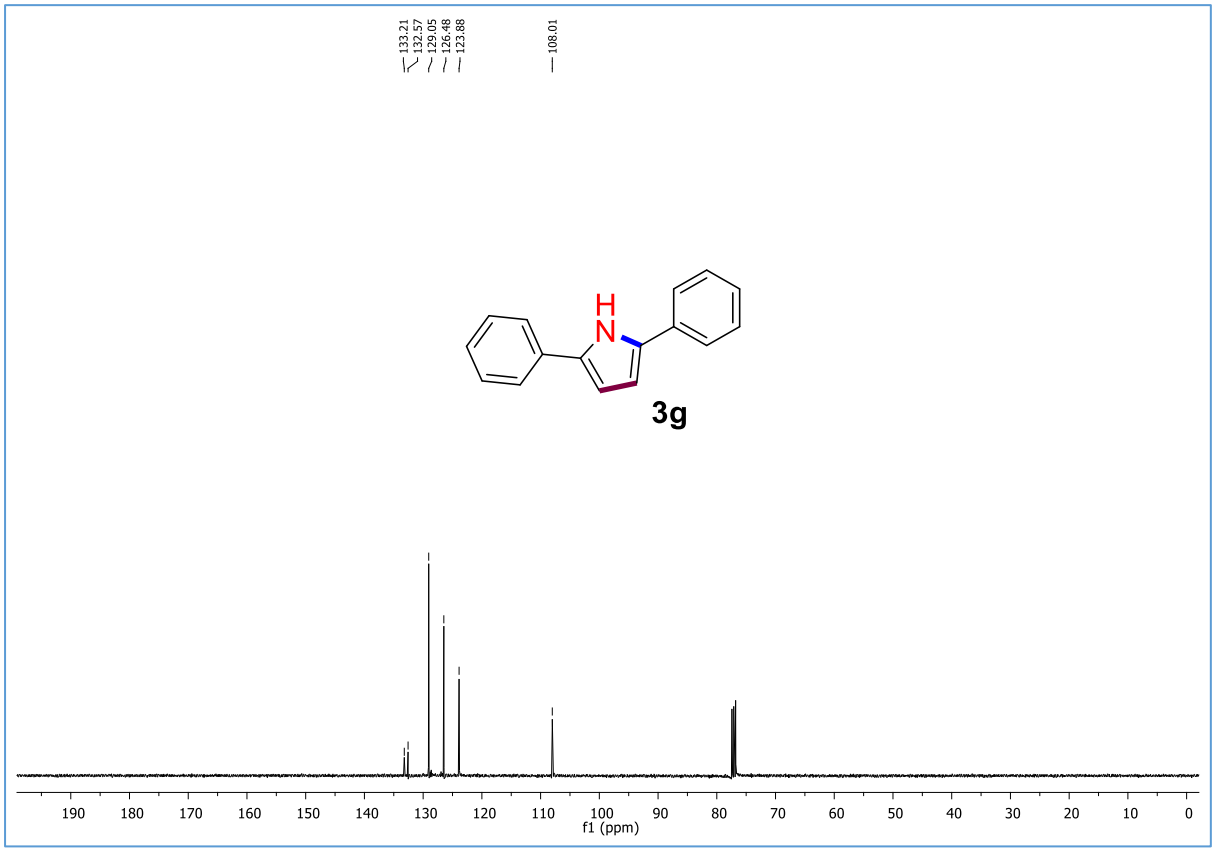
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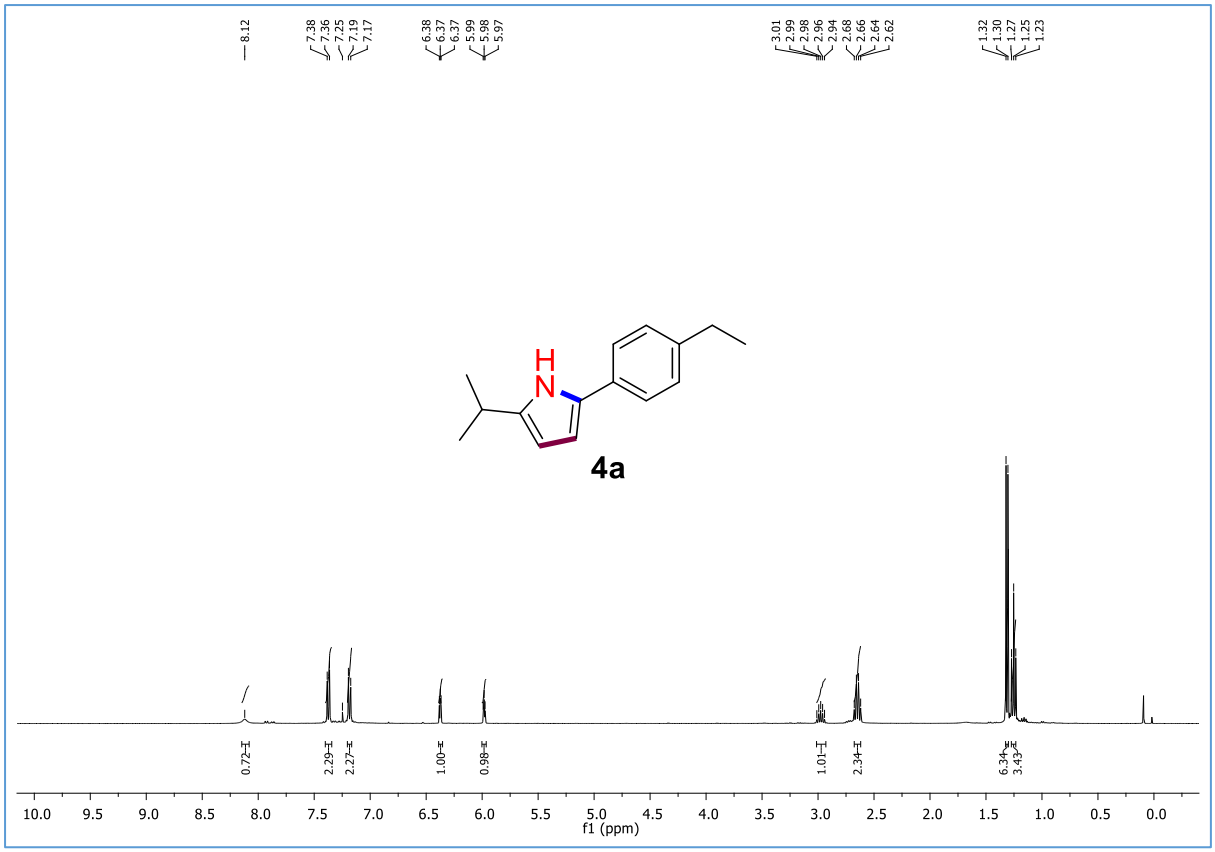
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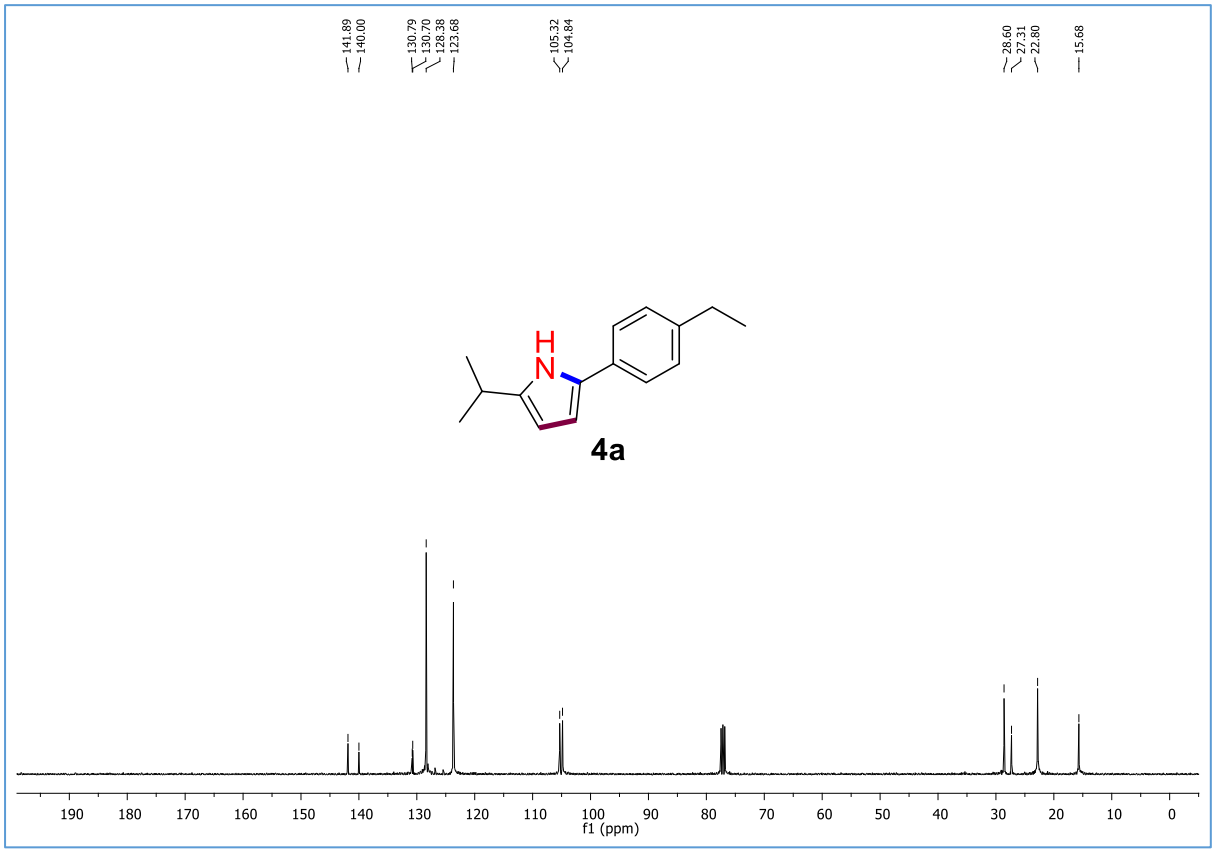
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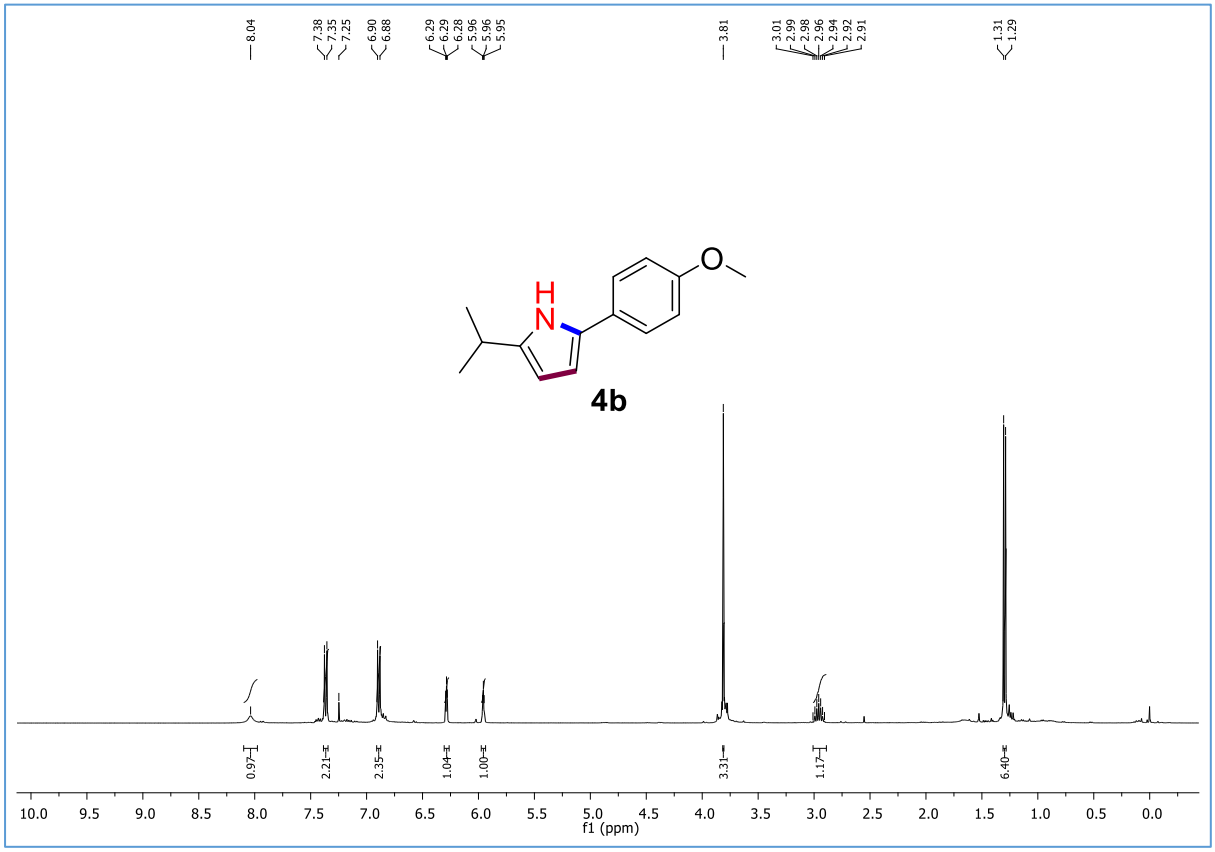
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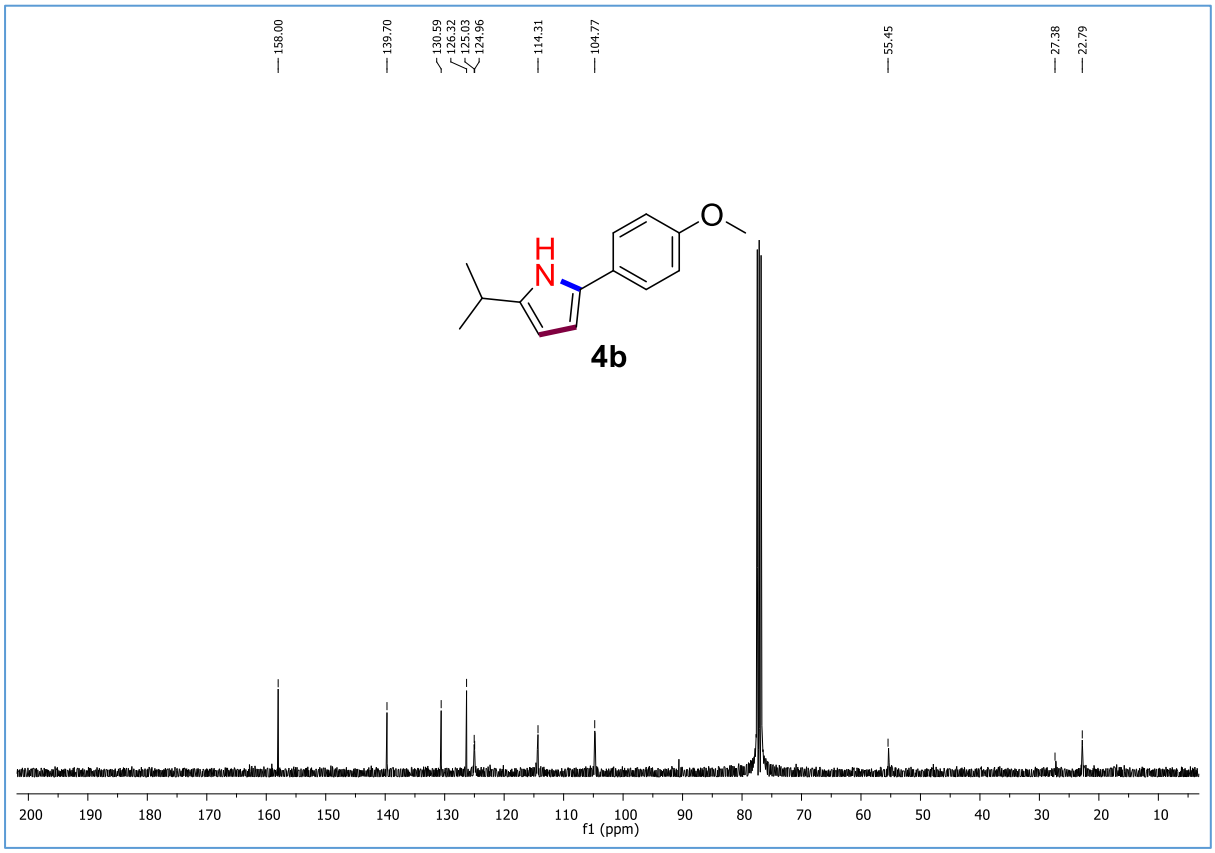
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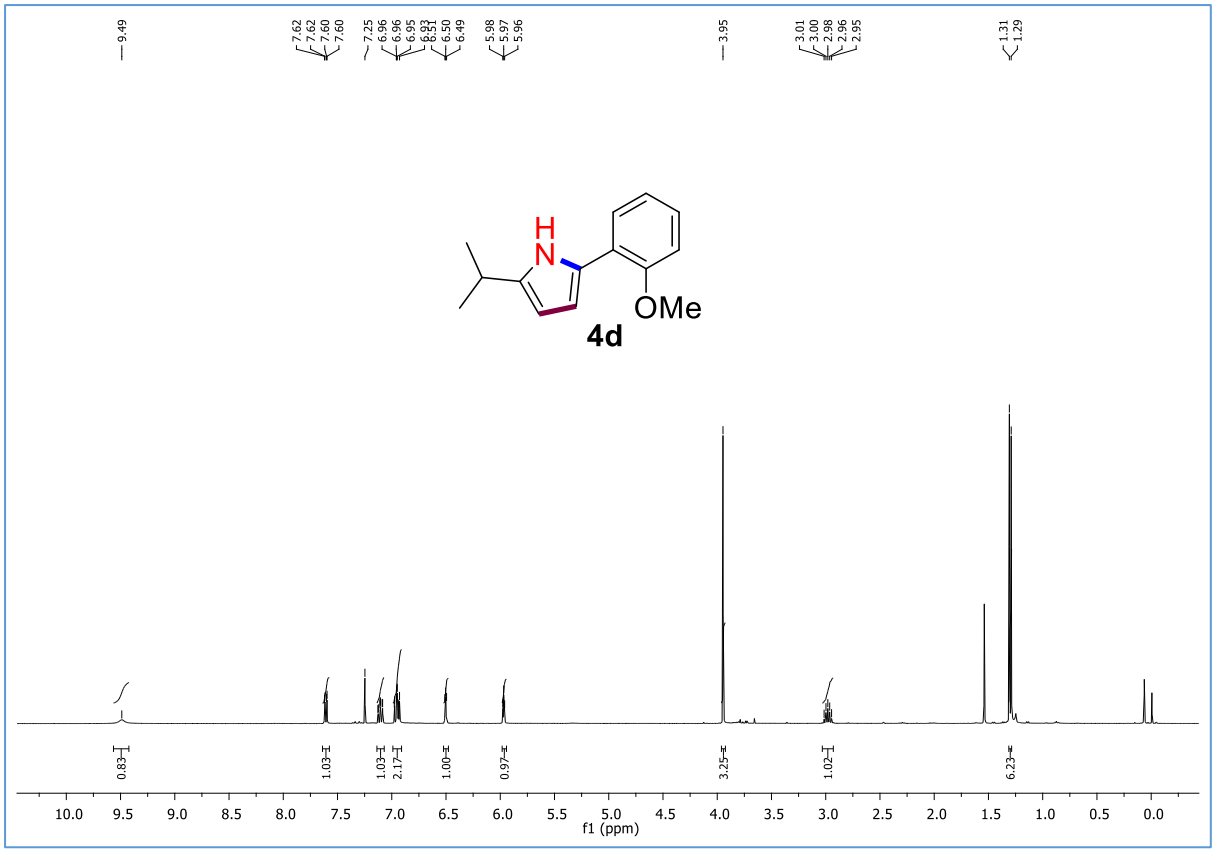
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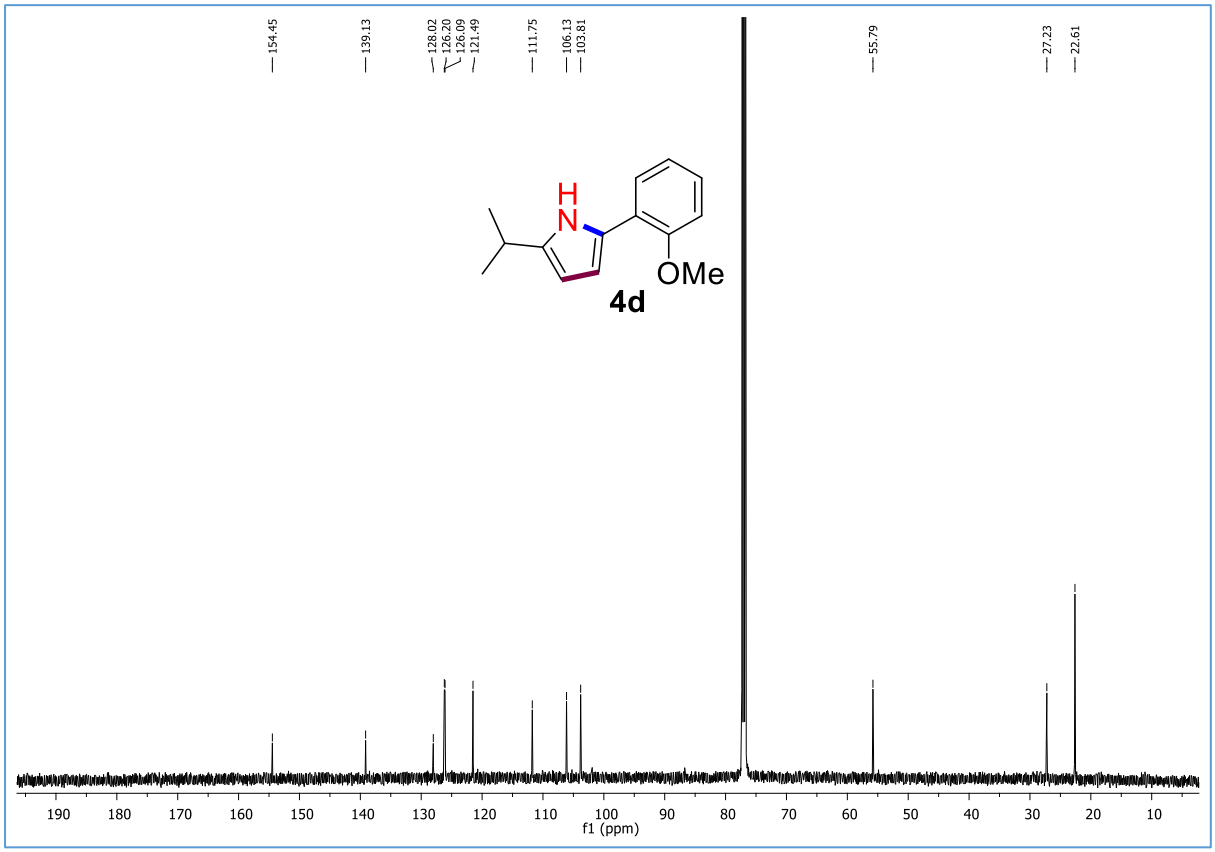
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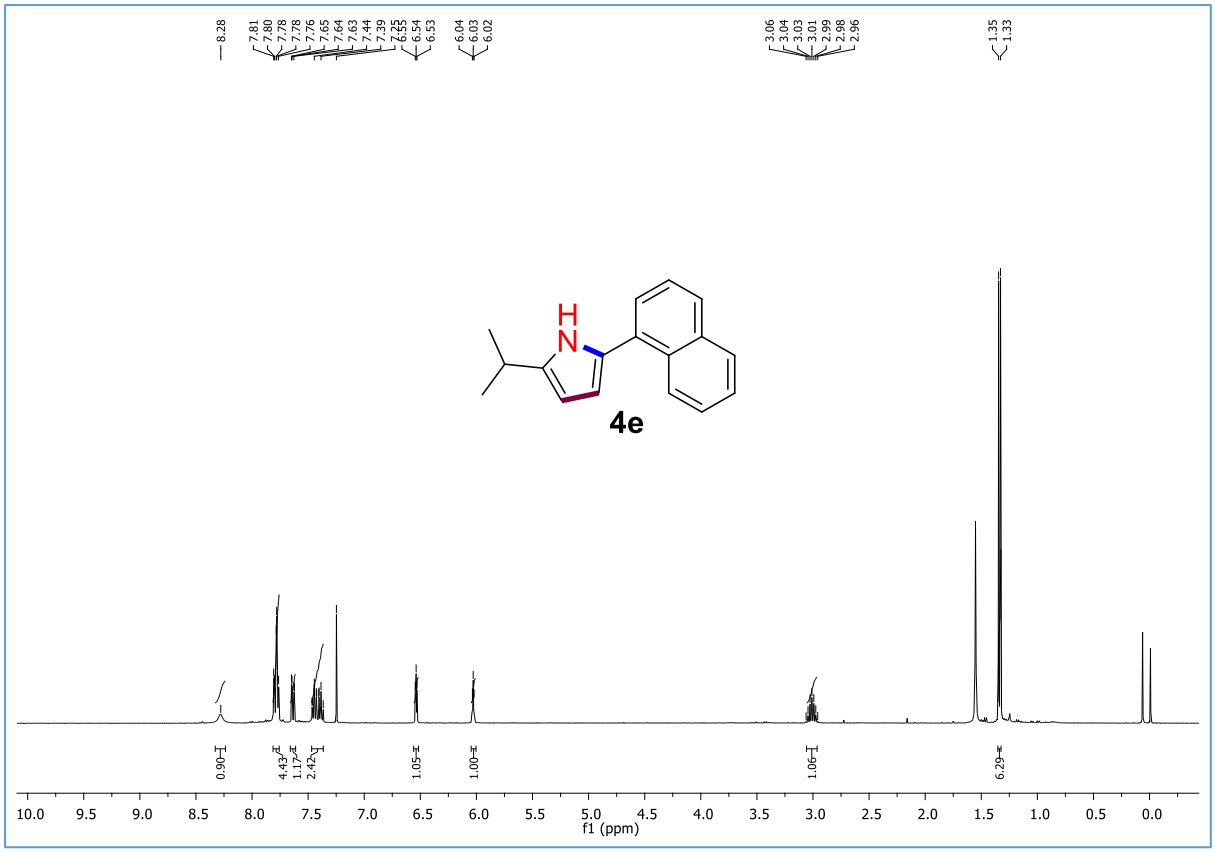
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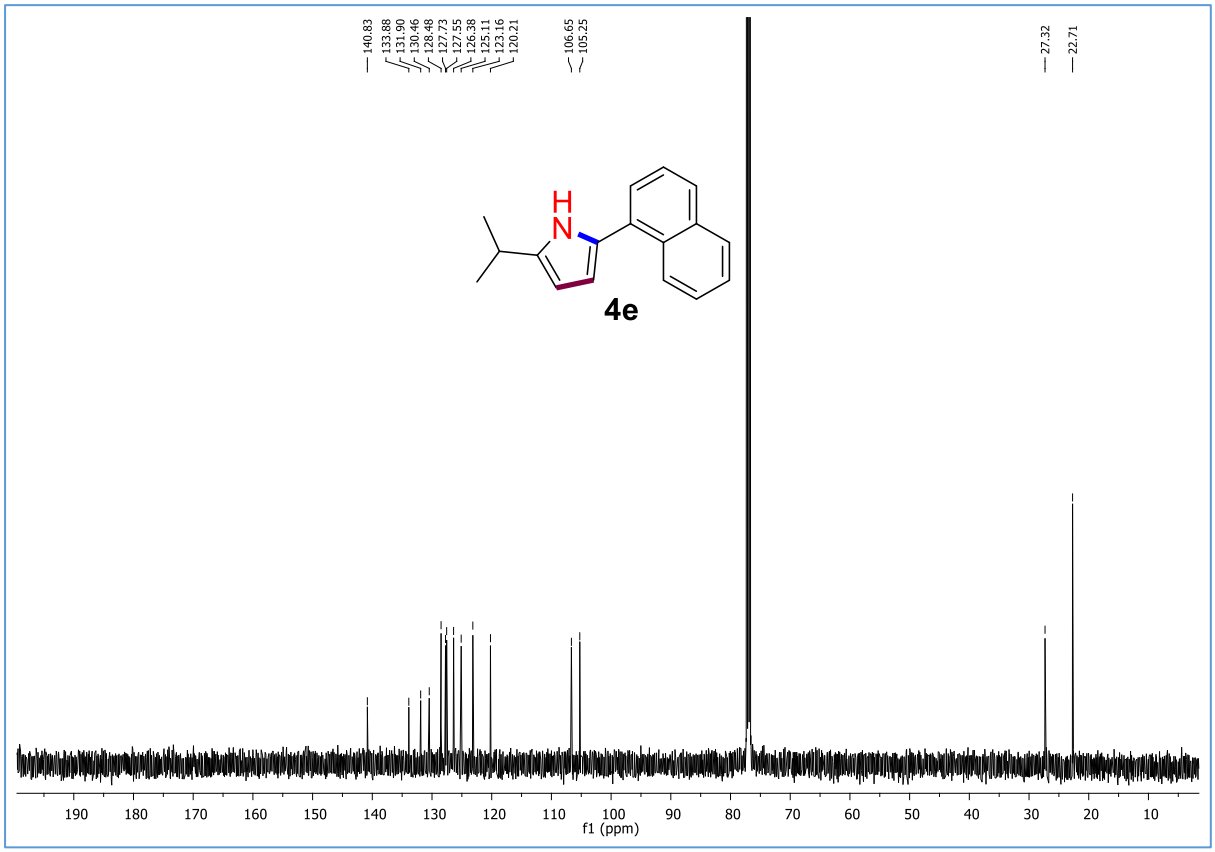
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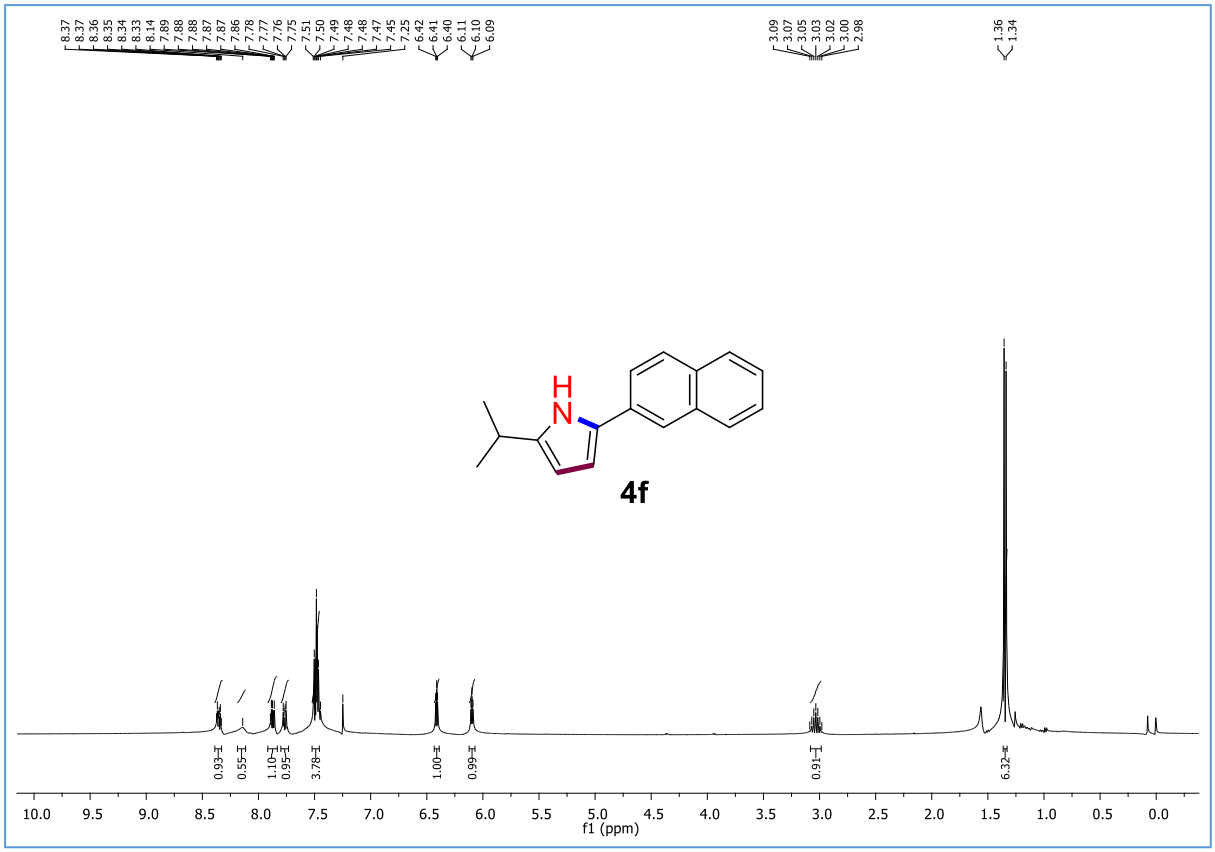
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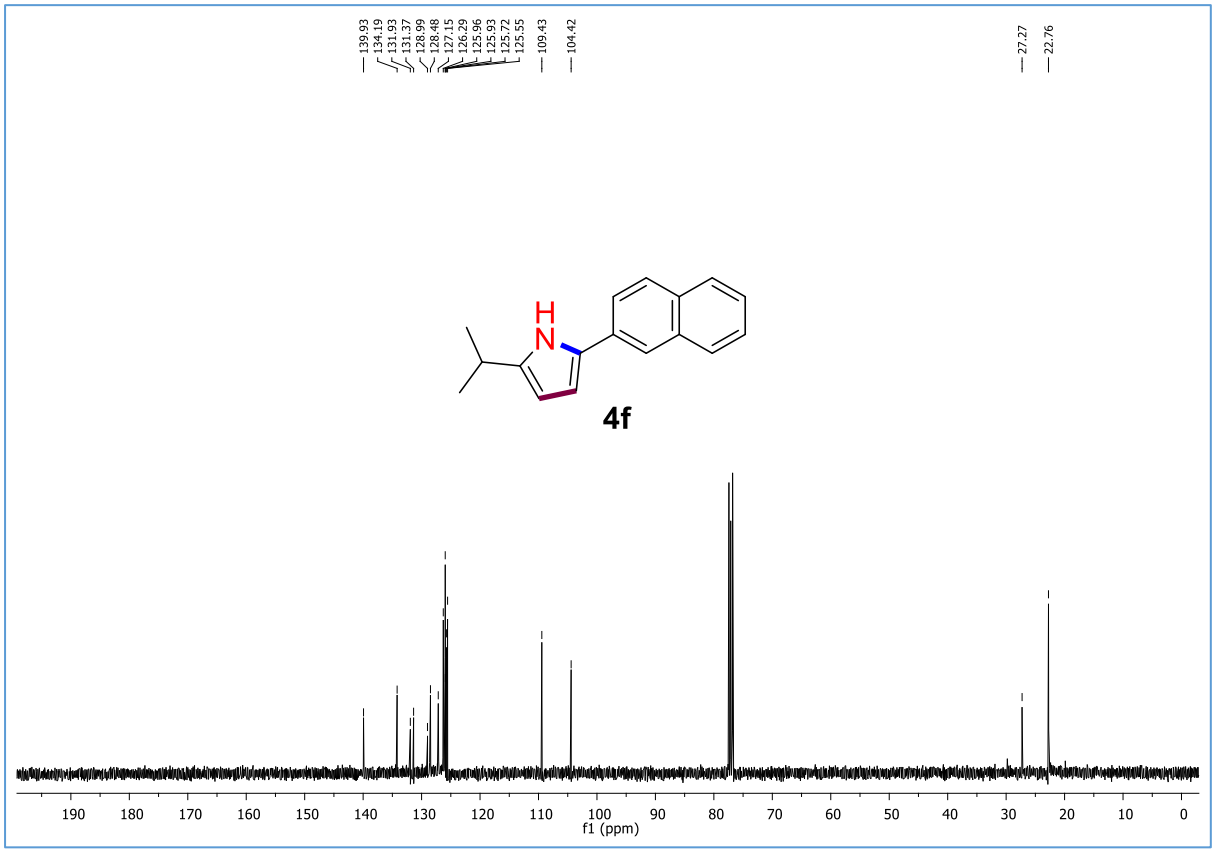
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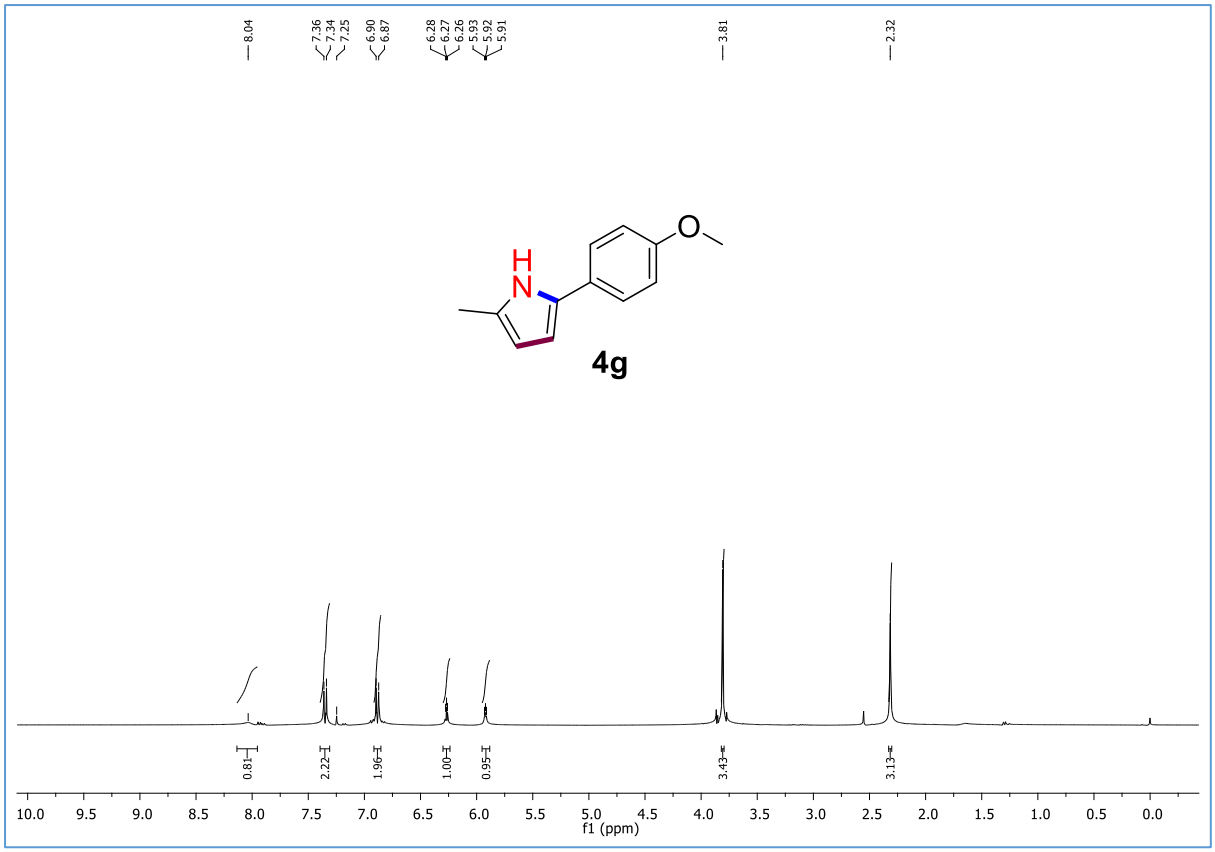
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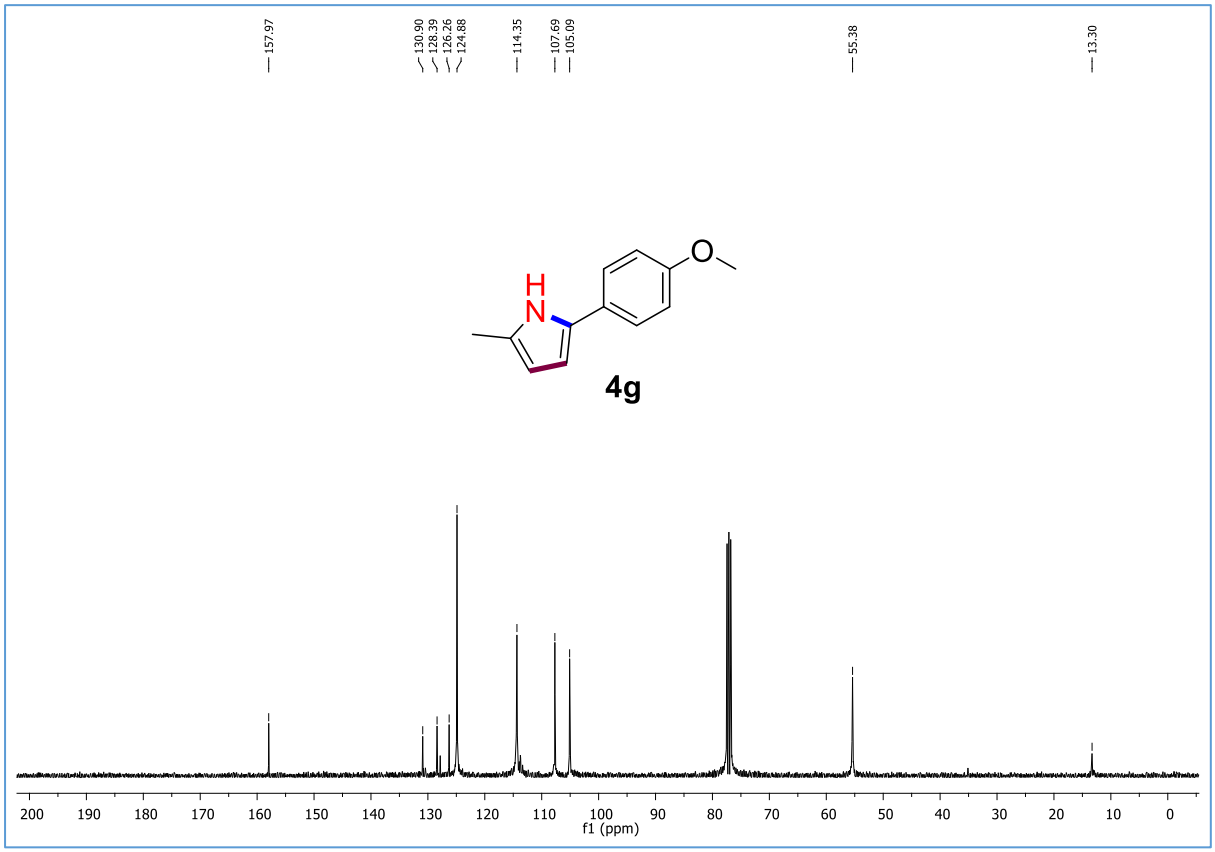
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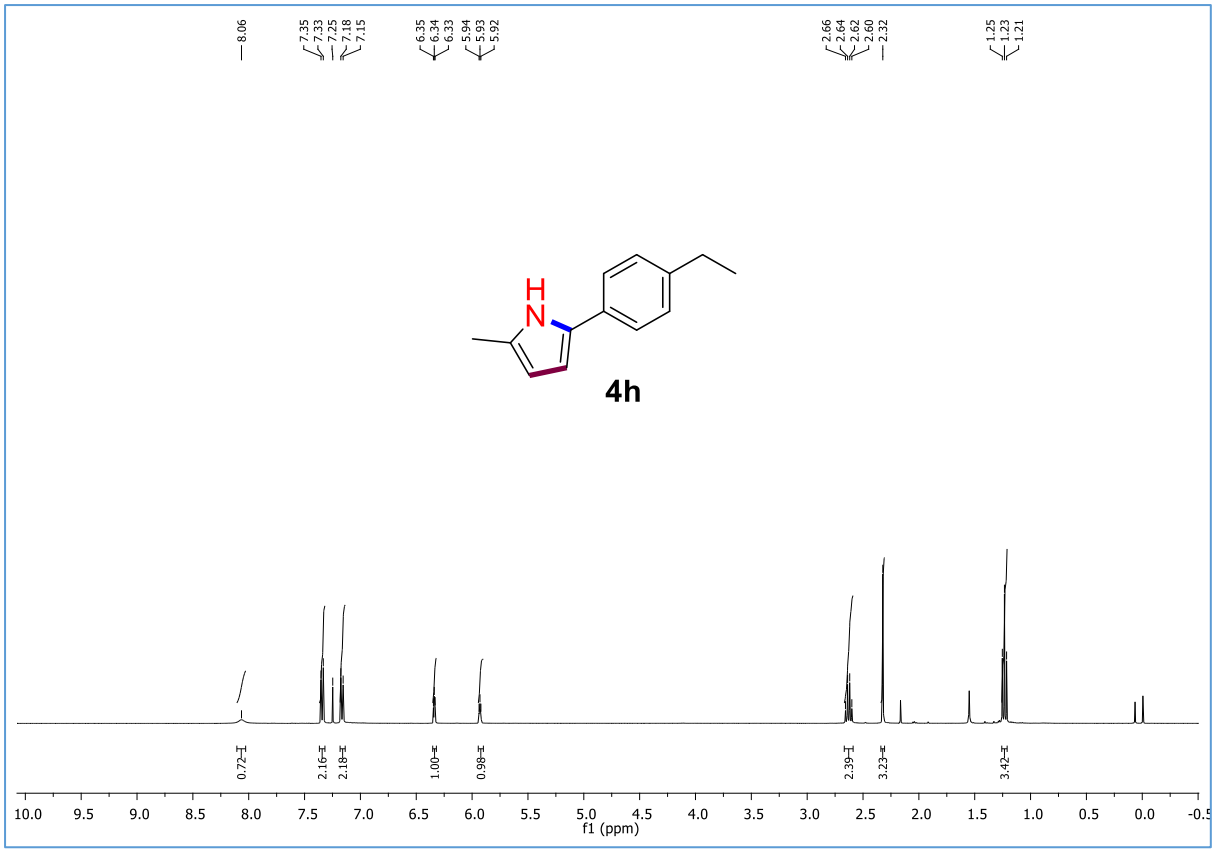
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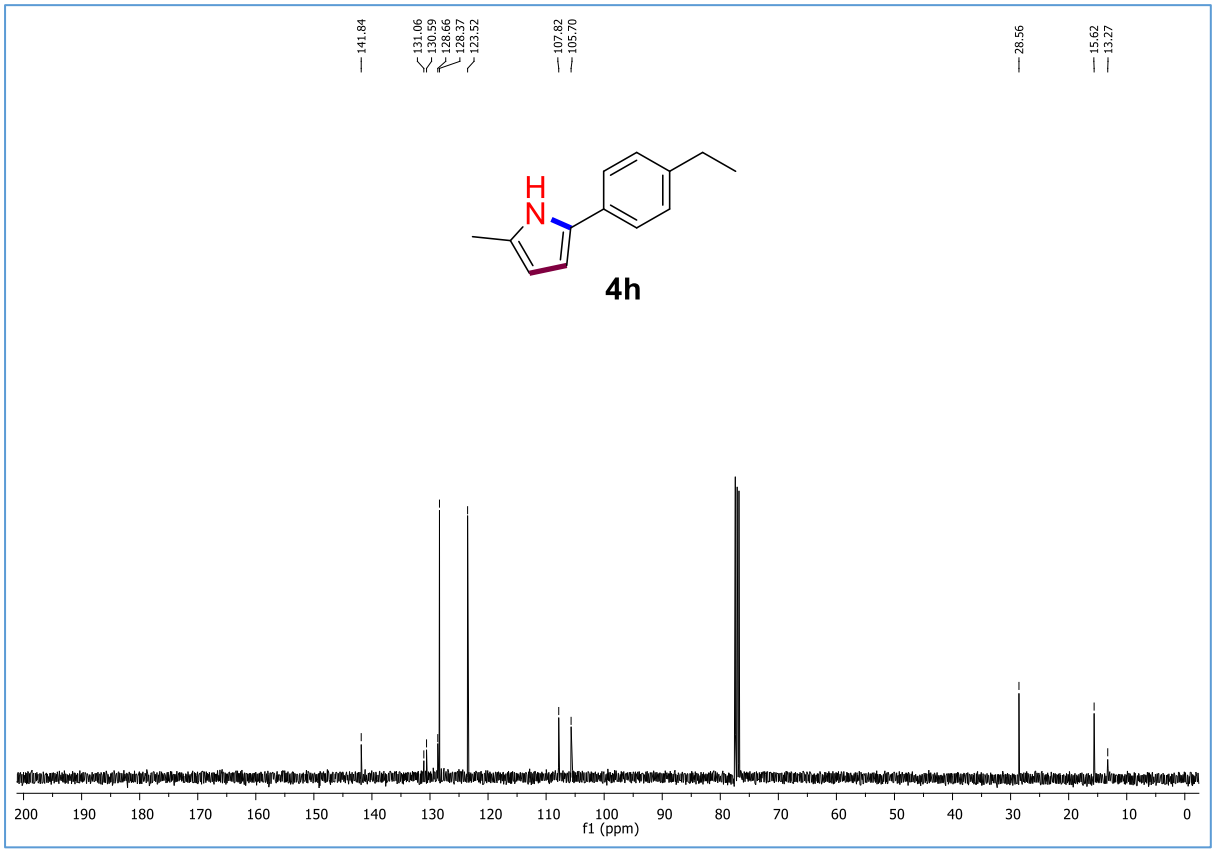
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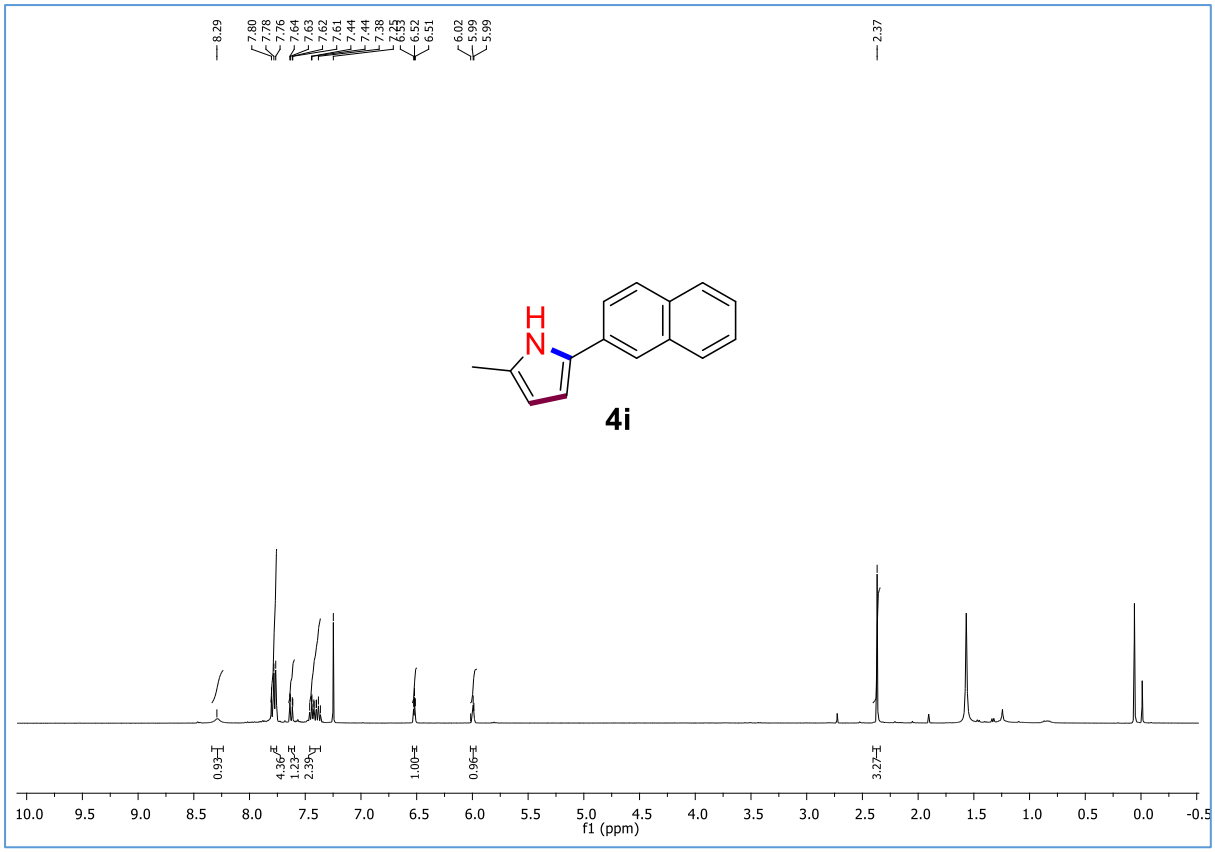
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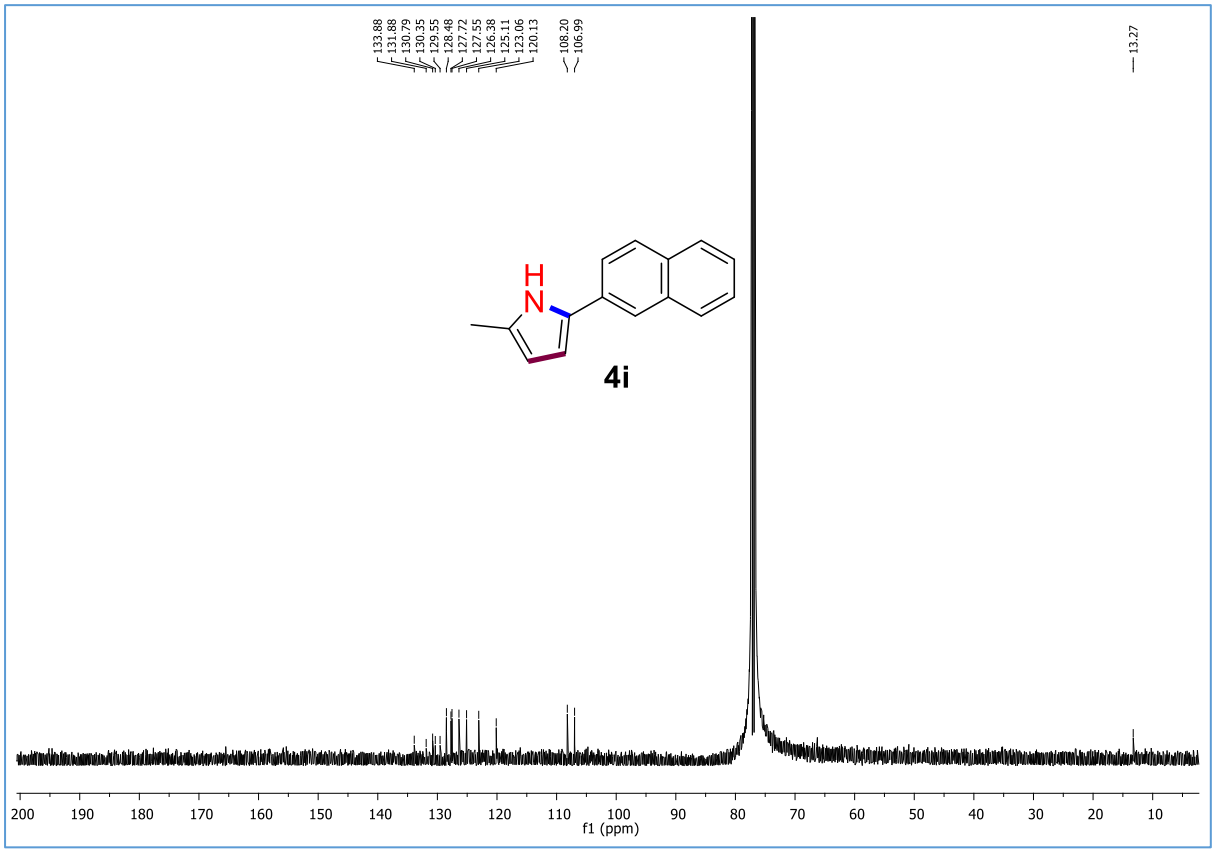
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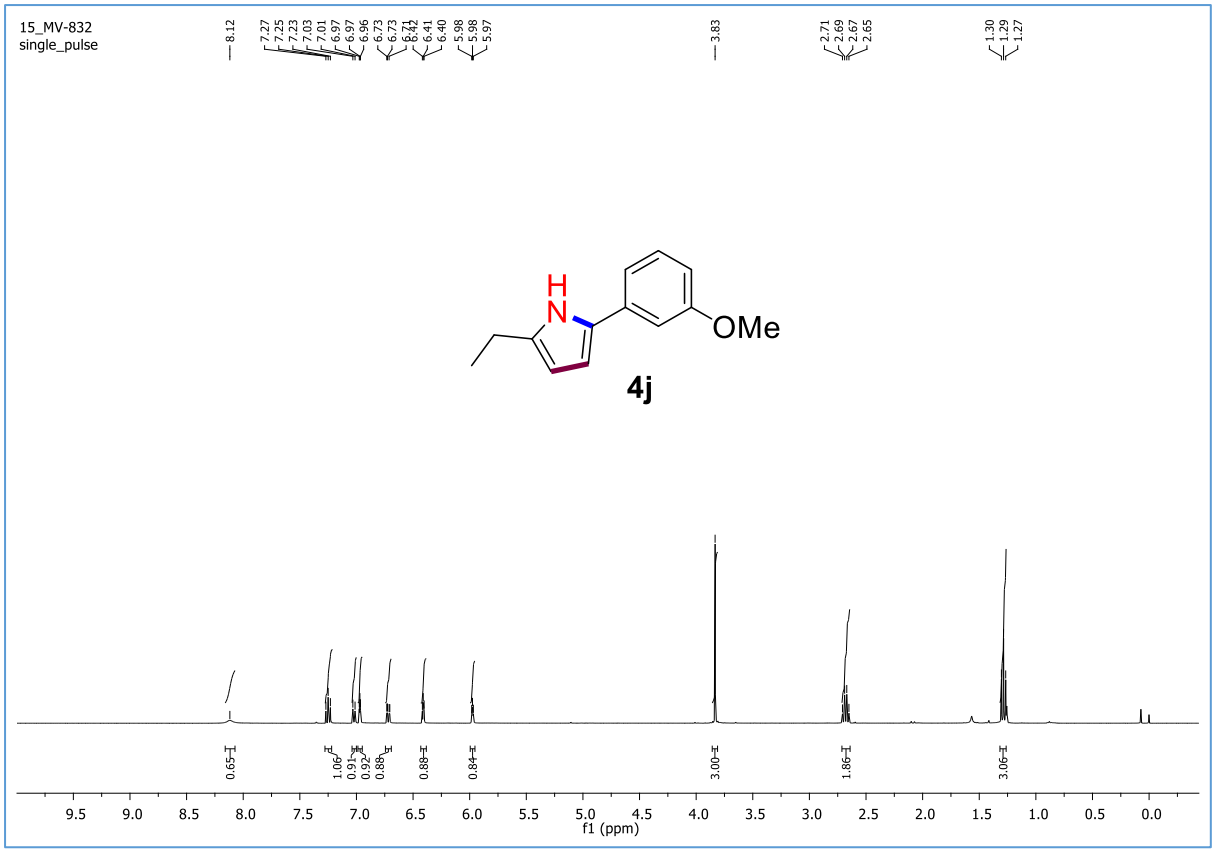
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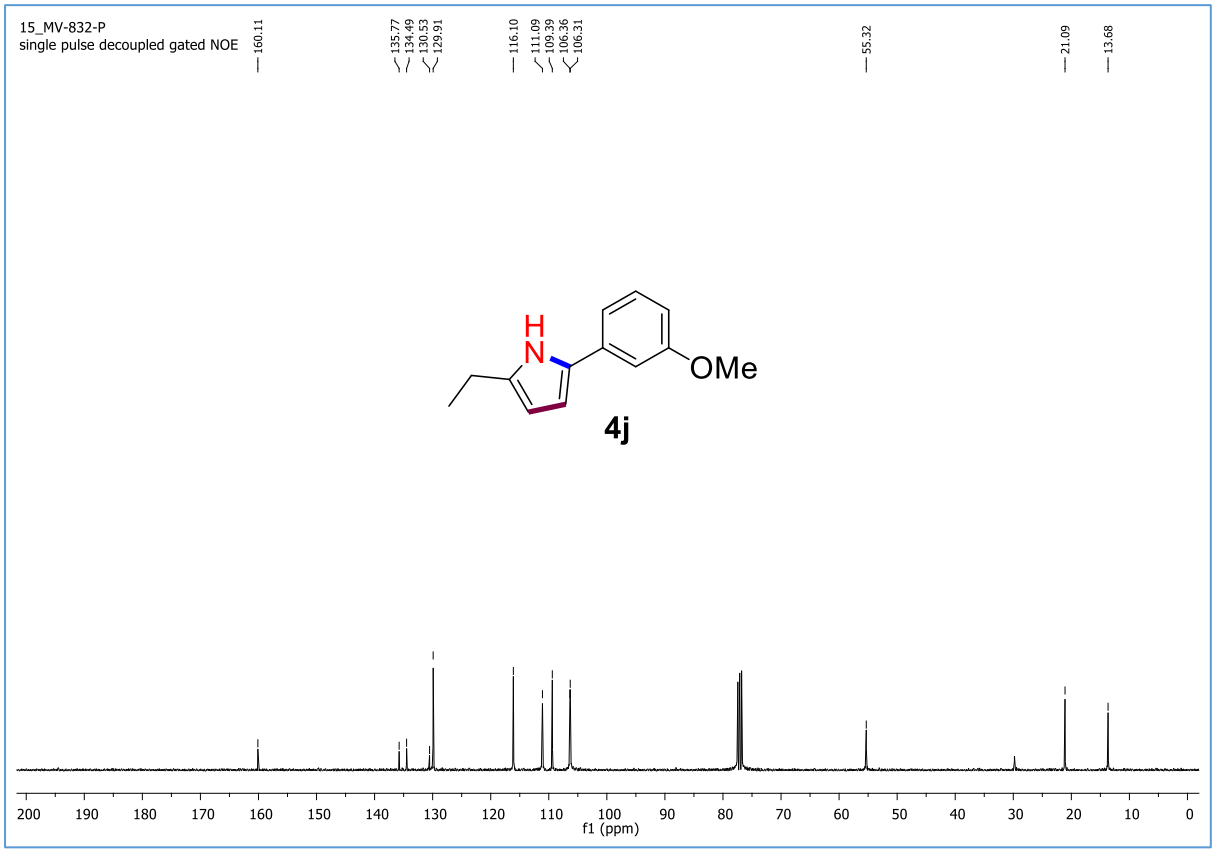
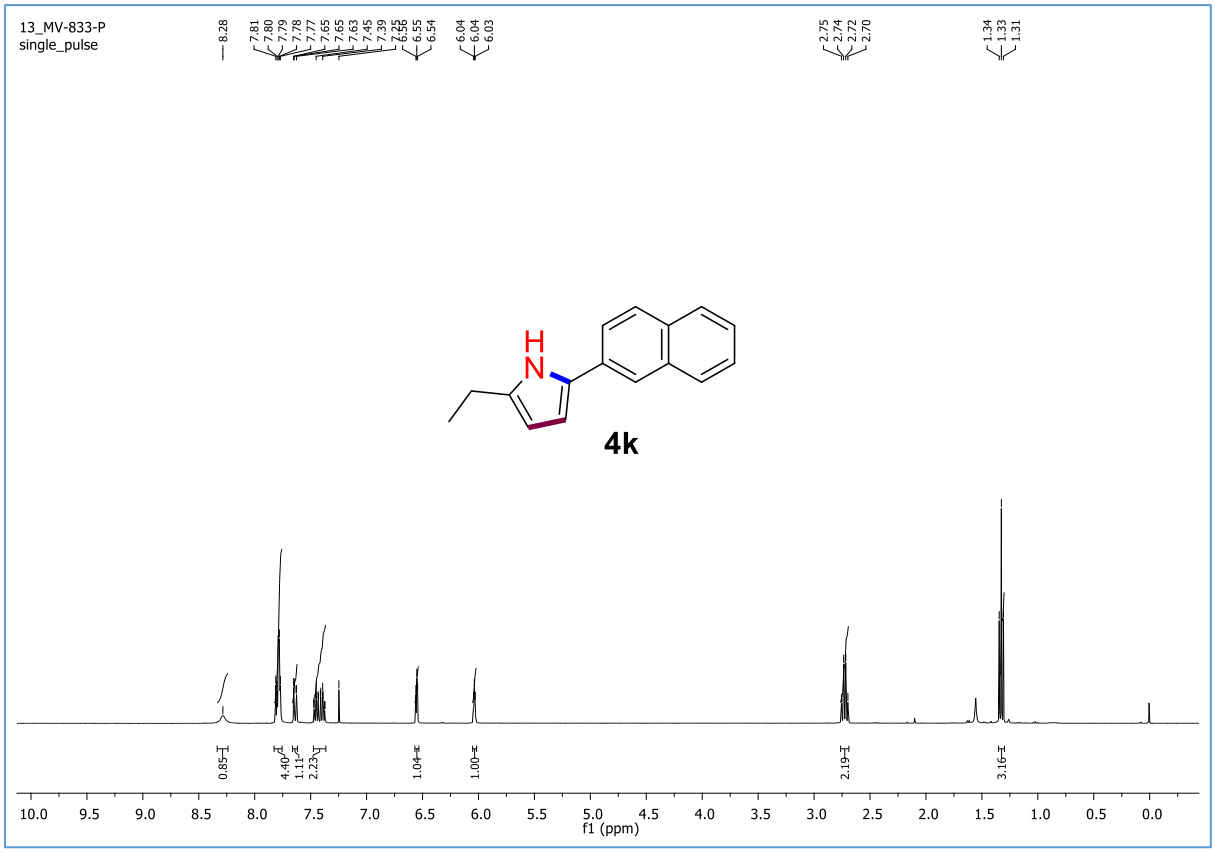
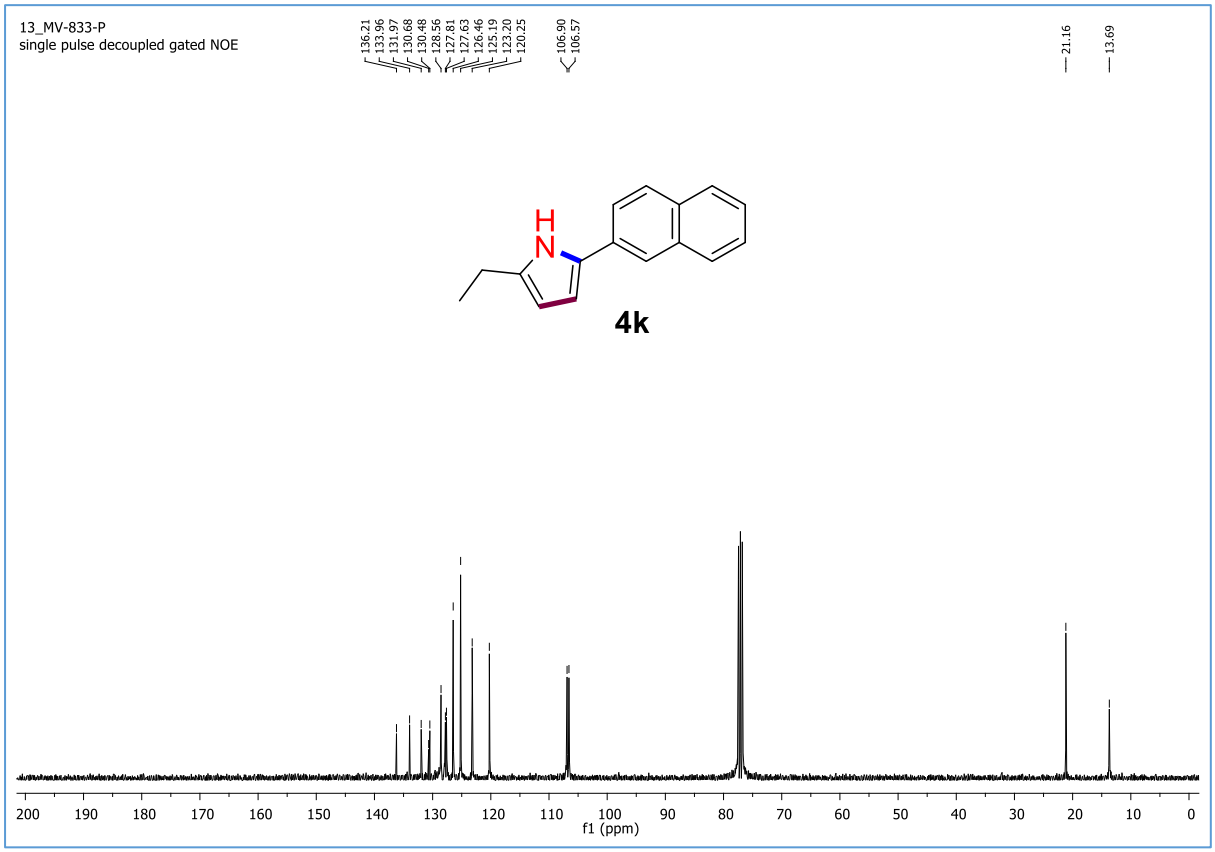
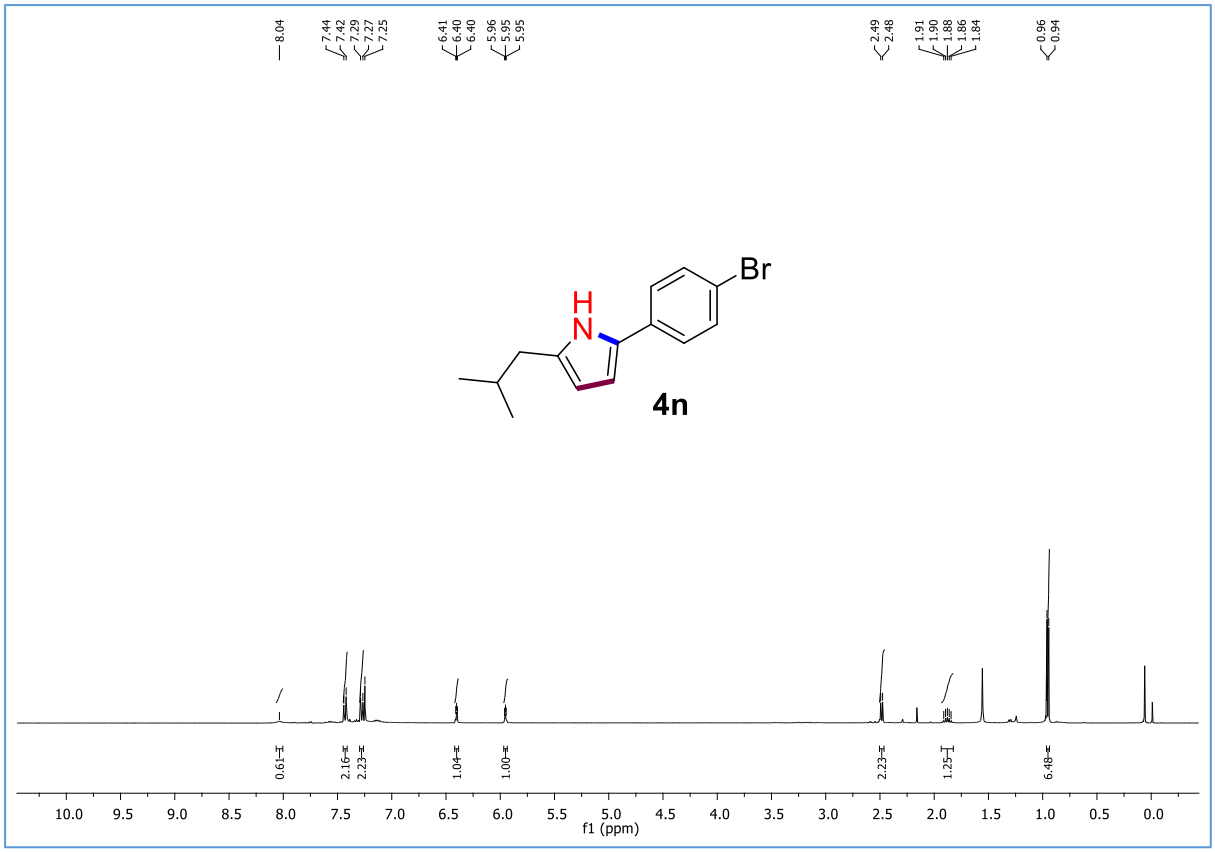
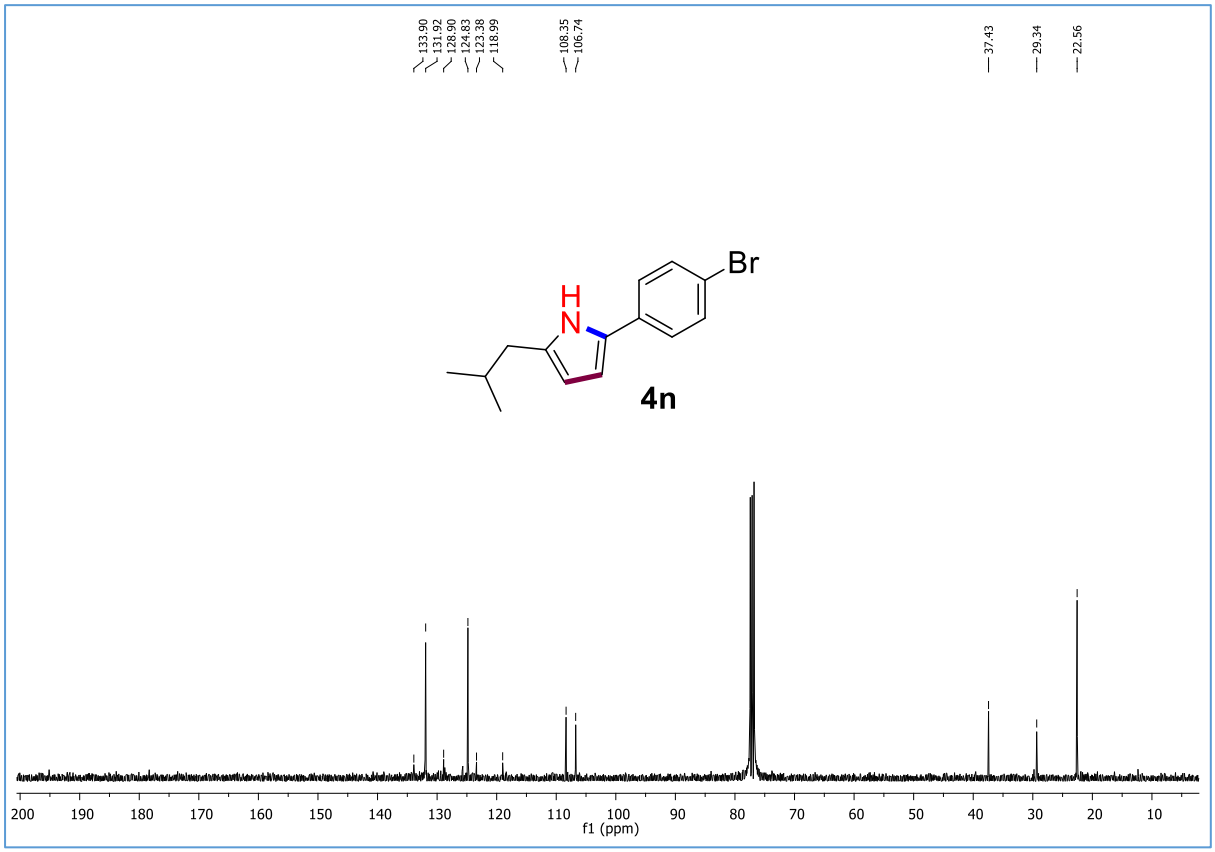
f1 (ppm)

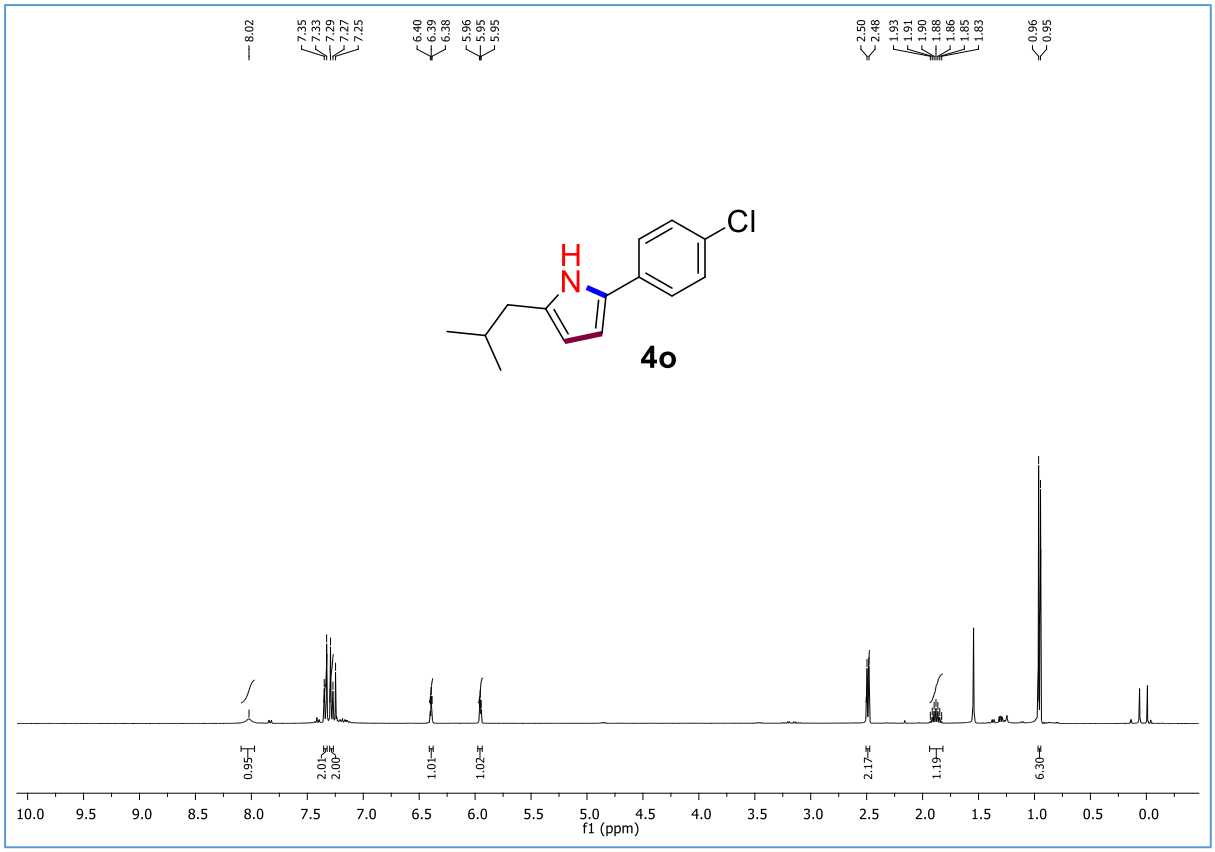

15_MV-832-P
single pulse decoupled gated NOE
160.11
135.77
134.49
130.53
129.91
116.10
111.09
109.39
106.36
106.31
55.32
21.09
13.68
H
N
OMe
4j
200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0
f1 (ppm)

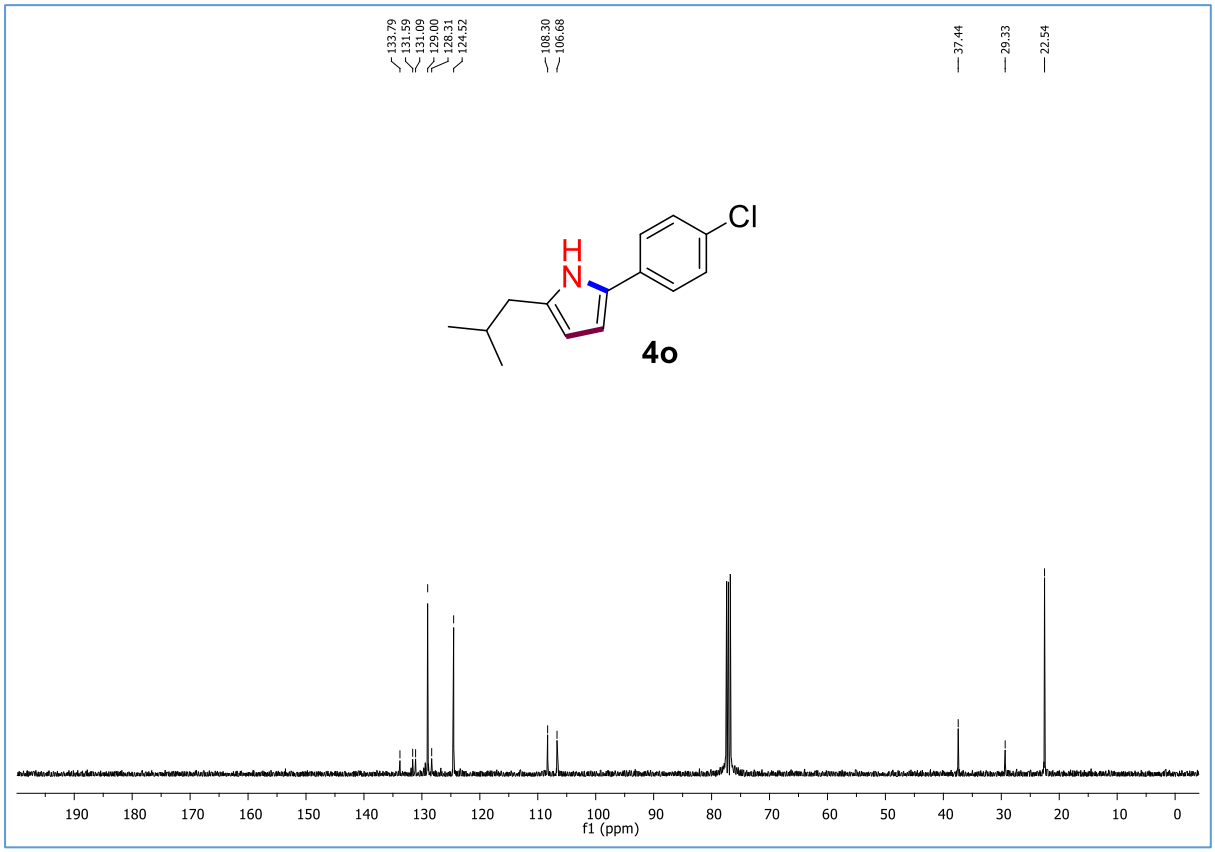

13_MV-833-P
single_pulse
8.28
7.81
7.80
7.79
7.78
7.77
7.65
7.63
7.45
7.39
7.25
6.56
6.55
6.54
6.04
6.04
6.03
2.75
2.74
2.72
2.70
1.34
1.33
1.31
H
N
4k
0.85
4.40
1.11
2.23
1.04
1.00
2.19
3.16
10.0
9.5
9.0
8.5
8.0
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
0.5
0.0
f1 (ppm)


13_MV-833-P
single pulse decoupled gated NOE
136.21
133.96
131.97
130.68
130.48
128.56
127.81
127.63
126.46
125.19
123.20
120.25
106.90
106.57
21.16
13.69
H
N
4k
200
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
f1 (ppm)


8.04
7.44
7.42
7.29
7.27
7.25
6.41
6.40
6.40
5.96
5.95
5.95
2.49
2.48
1.91
1.90
1.88
1.86
1.84
0.96
0.94
Br
H
N
4n
0.61
2.16
2.23
1.04
1.00
2.23
1.25
6.48
f1 (ppm)


133.90
131.92
128.90
124.83
123.38
118.99
108.35
106.74
37.43
29.34
22.56
Br
H
N
4n
f1 (ppm)


8.02
7.35
7.33
7.29
7.27
7.25
6.40
6.39
6.38
5.96
5.95
5.95
2.50
2.48
1.93
1.91
1.90
1.88
1.86
1.85
1.83
0.96
0.95
Cl
H
N
4o
0.95
2.01
2.00
1.01
1.02
2.17
1.19
6.30
10.0 9.5 9.0 8.5 8.0 7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 0.5 0.0
f1 (ppm)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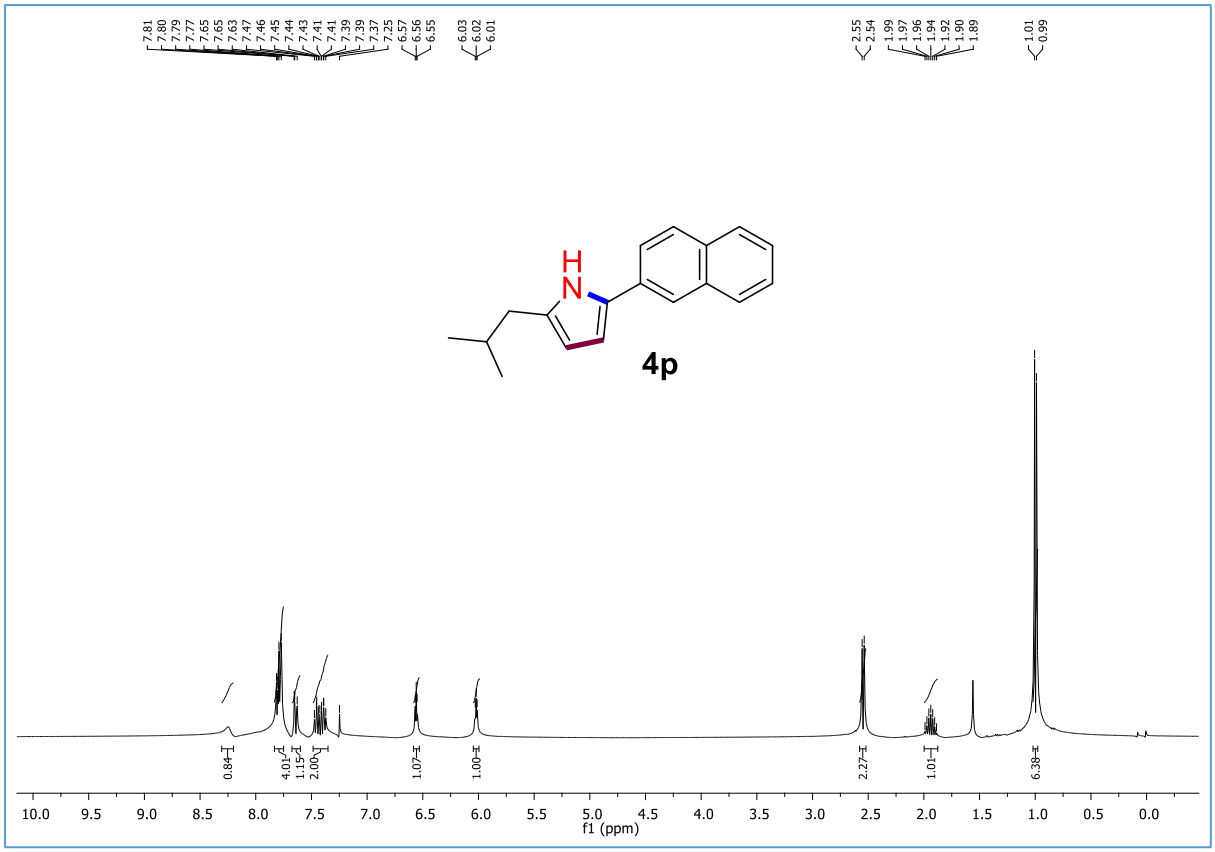
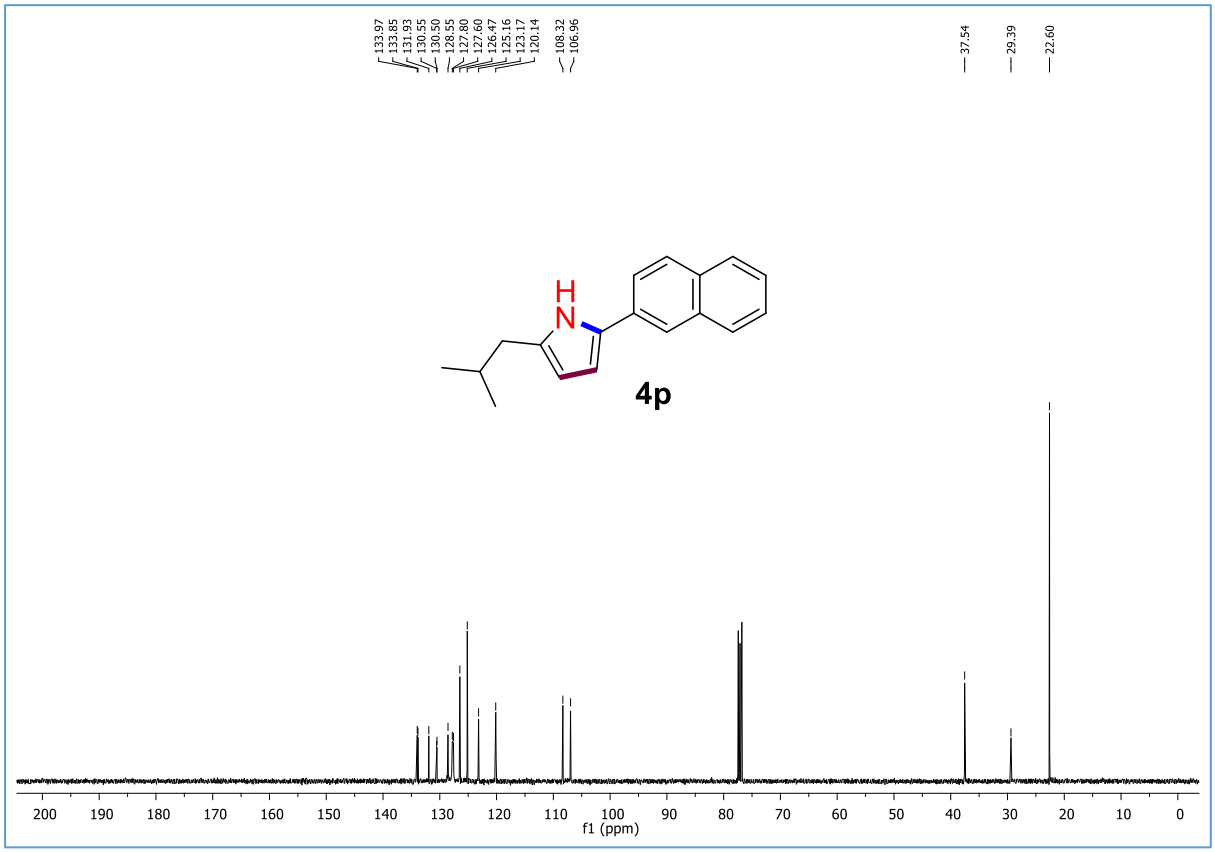
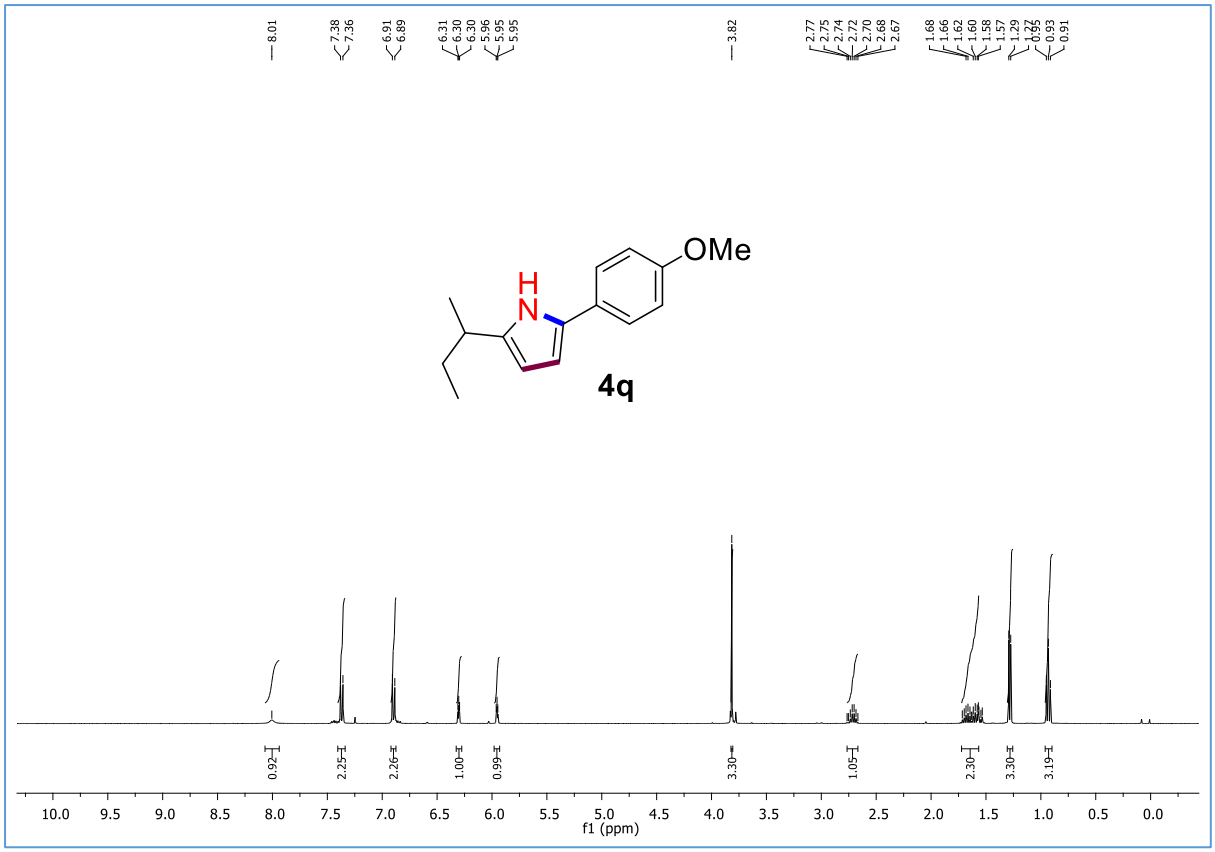
131.59
131.09
129.00
128.31
124.52
108.30
106.68
37.44
29.33
22.54
Cl
H
N
4o
190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0
f1 (ppm)


7.81
7.80
7.79
7.77
7.65
7.63
7.47
7.46
7.45
7.44
7.43
7.41
7.41
7.39
7.39
7.37
7.25
6.57
6.56
6.55
6.03
6.02
6.01
2.55
2.54
1.99
1.97
1.96
1.94
1.92
1.90
1.89
1.01
0.99
H
N
4p
0.84
4.01
1.15
2.00
1.07
1.00
2.27
1.01
6.38
10.0 9.5 9.0 8.5 8.0 7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 0.5 0.0
f1 (ppm)


133.97
133.85
131.93
130.55
130.50
128.55
127.80
127.60
126.47
125.16
123.17
120.14
108.32
106.96
37.54
29.39
22.60
H
N
4p
200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0
f1 (ppm)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7.38
7.36
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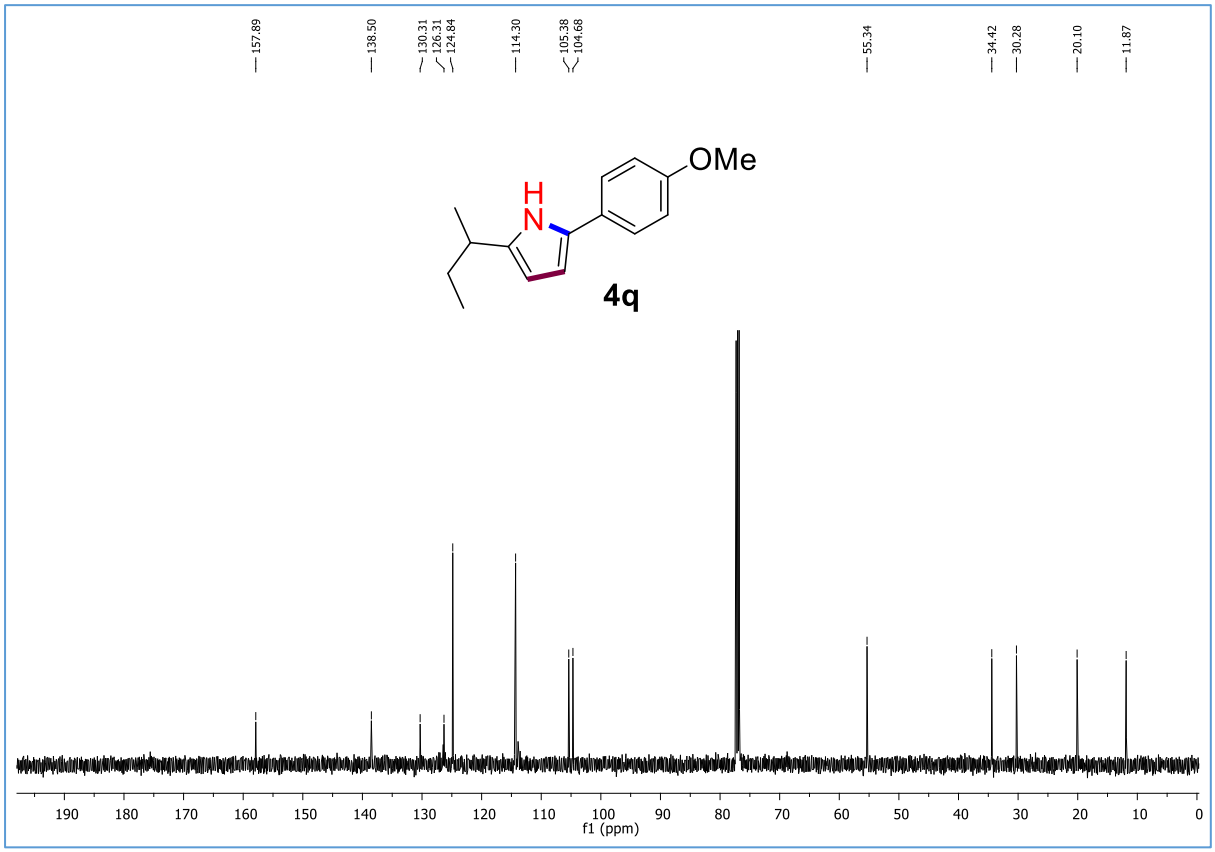
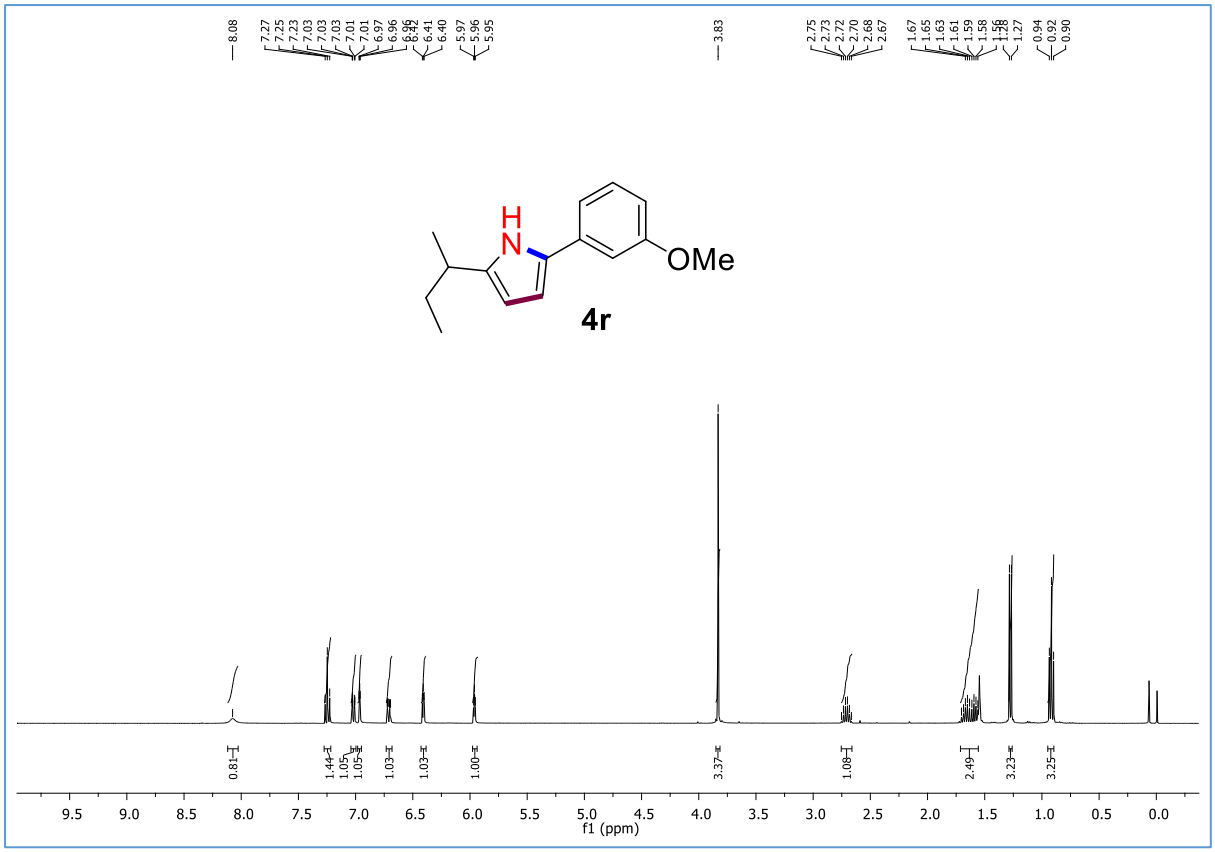
6.89
6.31
6.30
6.30
5.96
5.95
5.95
3.82
2.77
2.75
2.74
2.72
2.70
2.68
2.67
1.68
1.66
1.62
1.60
1.58
1.57
1.29
1.25
0.92
0.93
0.91
OMe
H
N
4q
0.92
2.25
2.26
1.00
0.99
3.30
1.05
2.30
3.30
3.19
10.0 9.5 9.0 8.5 8.0 7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 0.5 0.0
f1 (ppm)


157.89
138.50
130.31
126.31
124.84
114.30
105.38
104.68
55.34
34.42
30.28
20.10
11.87
OMe
H
N
4q
190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0
f1 (ppm)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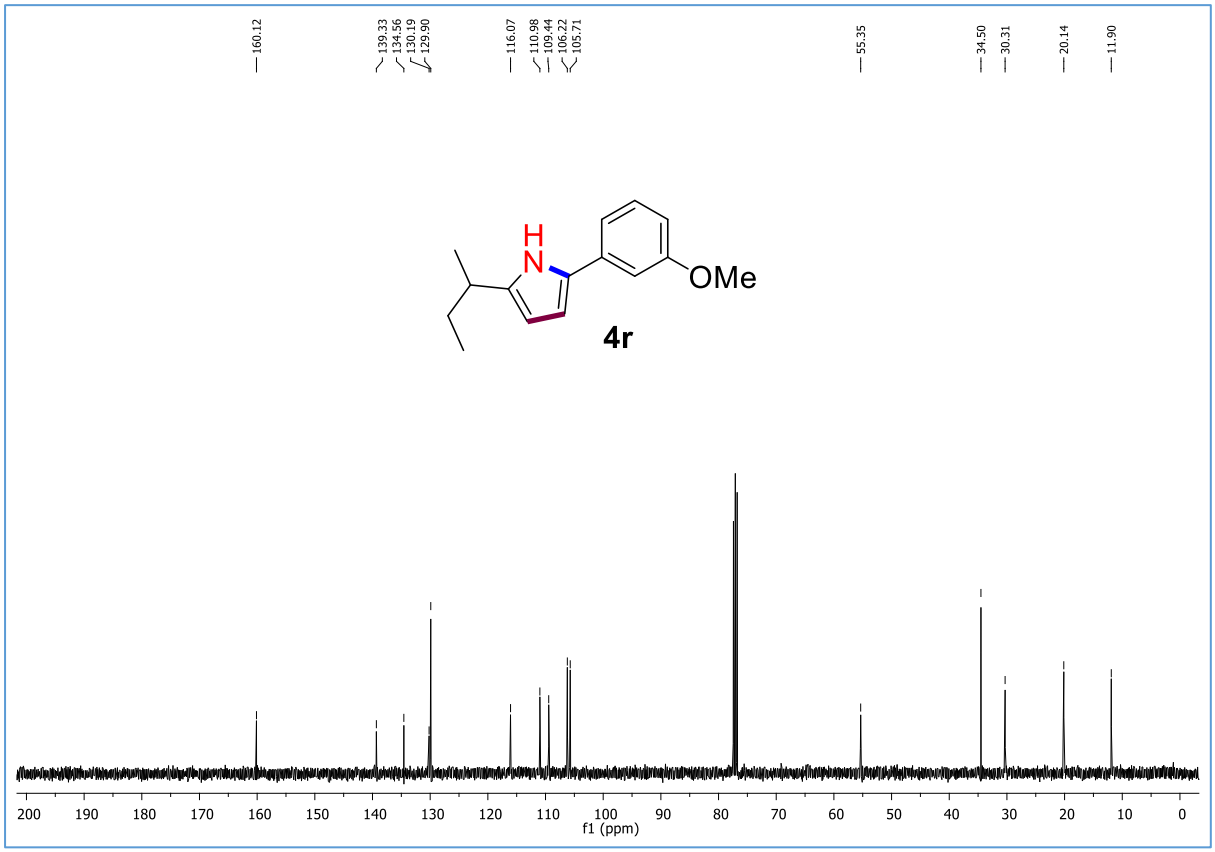
7.27
7.25
7.23
7.03
7.03
7.03
7.01
7.01
6.97
6.96
6.41
6.40
5.97
5.96
5.95
3.83
2.75
2.73
2.72
2.70
2.68
2.67
1.67
1.65
1.63
1.61
1.59
1.58
1.27
0.94
0.92
0.90
H
N
OMe
4r
0.81
1.44
1.05
1.05
1.03
1.03
1.00
3.37
1.08
2.49
3.23
3.25
f1 (ppm)


160.12
139.33
134.56
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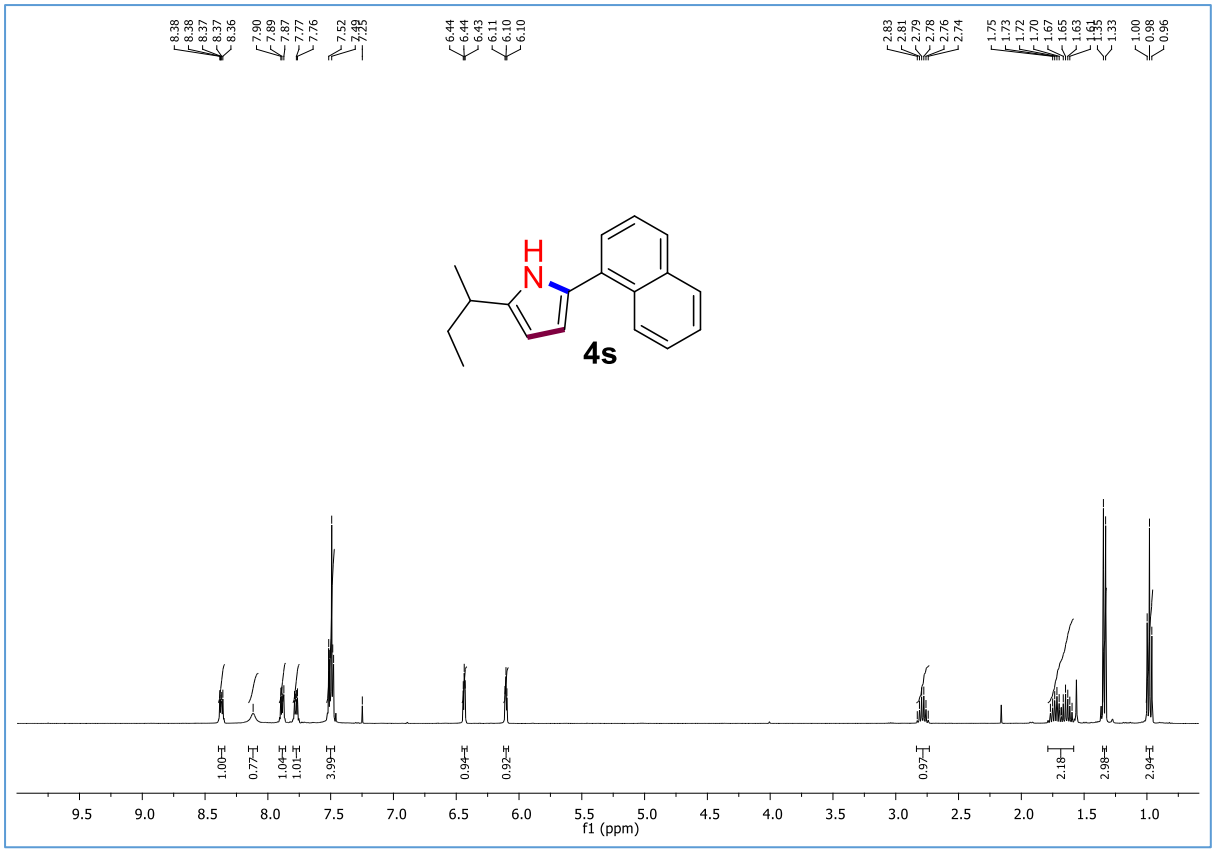
129.90
116.07
110.98
109.44
106.22
105.71
55.35
34.50
30.31
20.14
11.90
H
N
OMe
4r
f1 (ppm)


8.38
8.38
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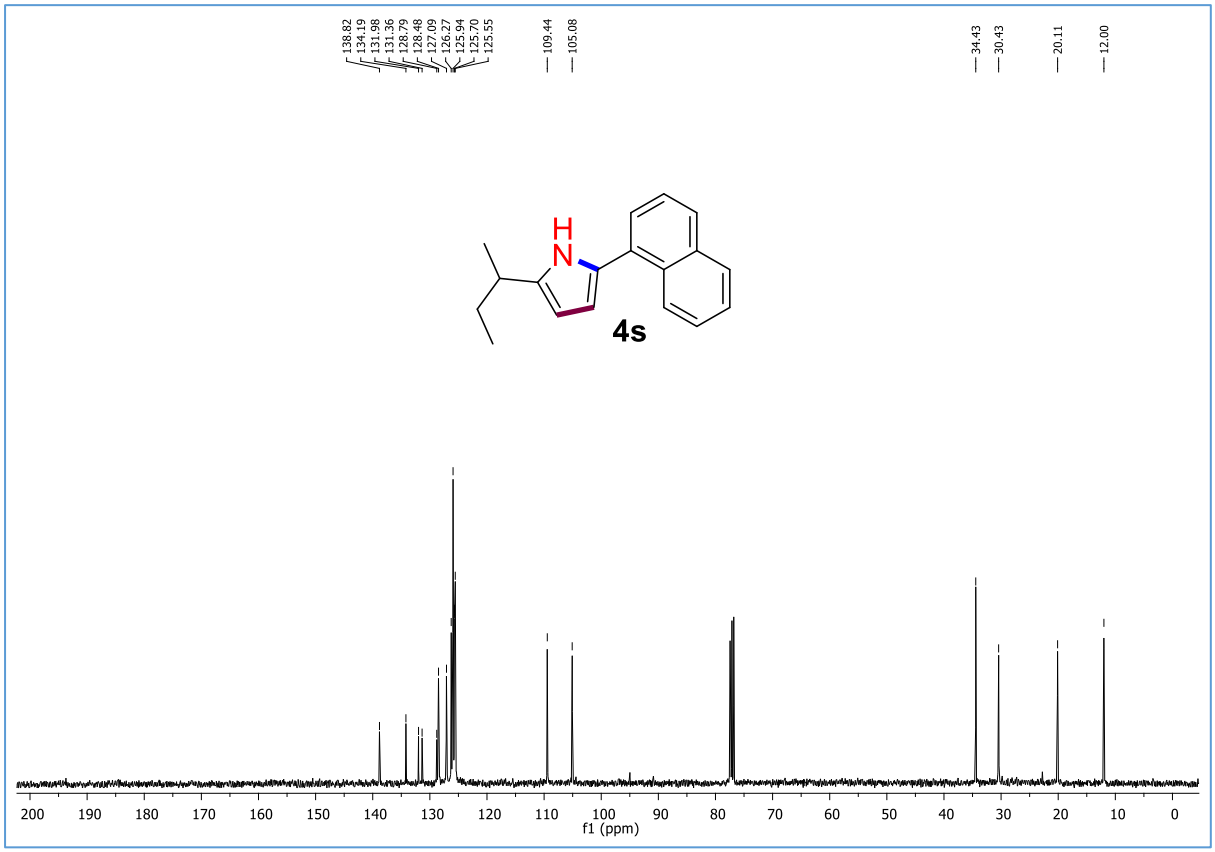
8.37
8.36
7.90
7.89
7.87
7.77
7.76
7.52
6.44
6.44
6.43
6.11
6.10
6.10
2.83
2.81
2.79
2.78
2.76
2.74
1.75
1.73
1.72
1.70
1.67
1.65
1.63
1.33
1.00
0.98
0.96
H
N
4s
1.00
0.77
1.04
1.01
3.99
0.94
0.92
0.97
2.18
2.98
2.94
9.5
9.0
8.5
8.0
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
f1 (ppm)


138.82
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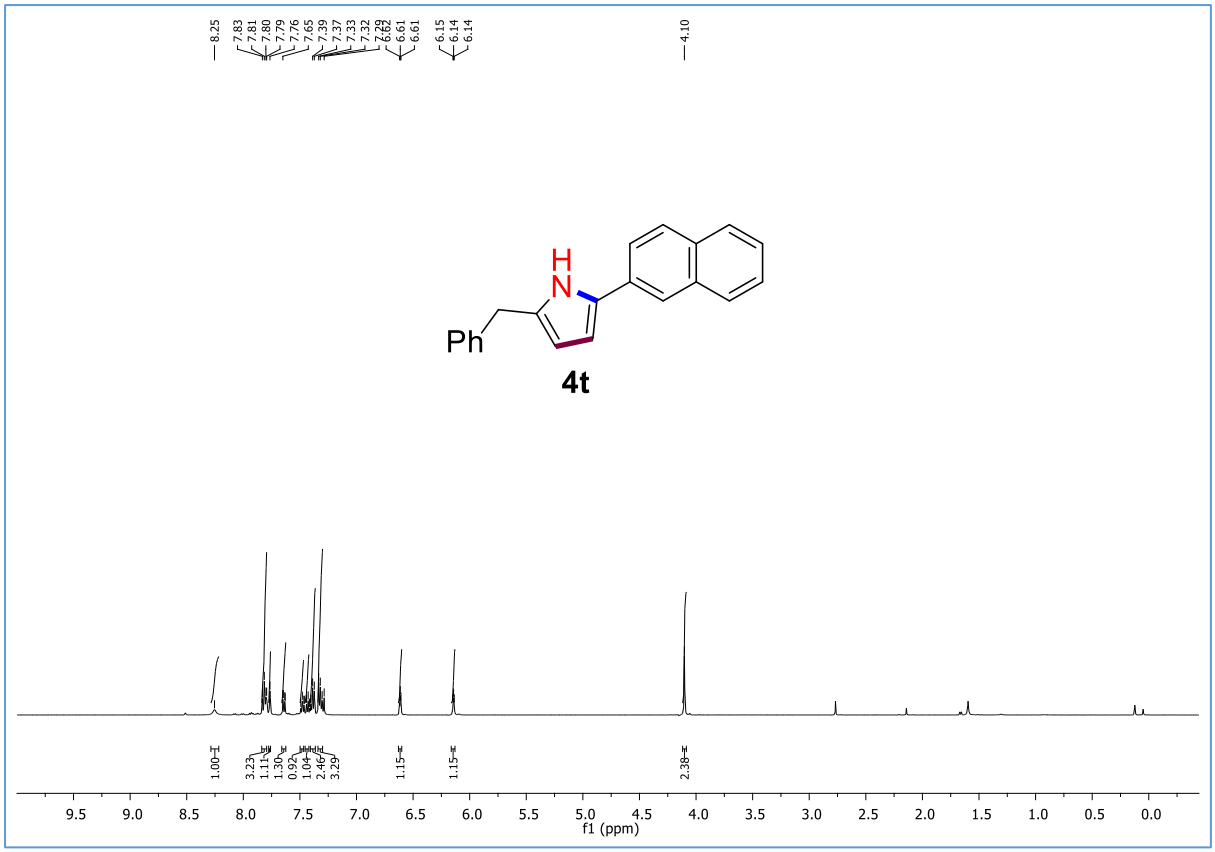
128.48
127.09
126.27
125.94
125.70
125.55
109.44
105.08
34.43
30.43
20.11
12.00
H
N
4s
200
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
f1 (ppm)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7.81
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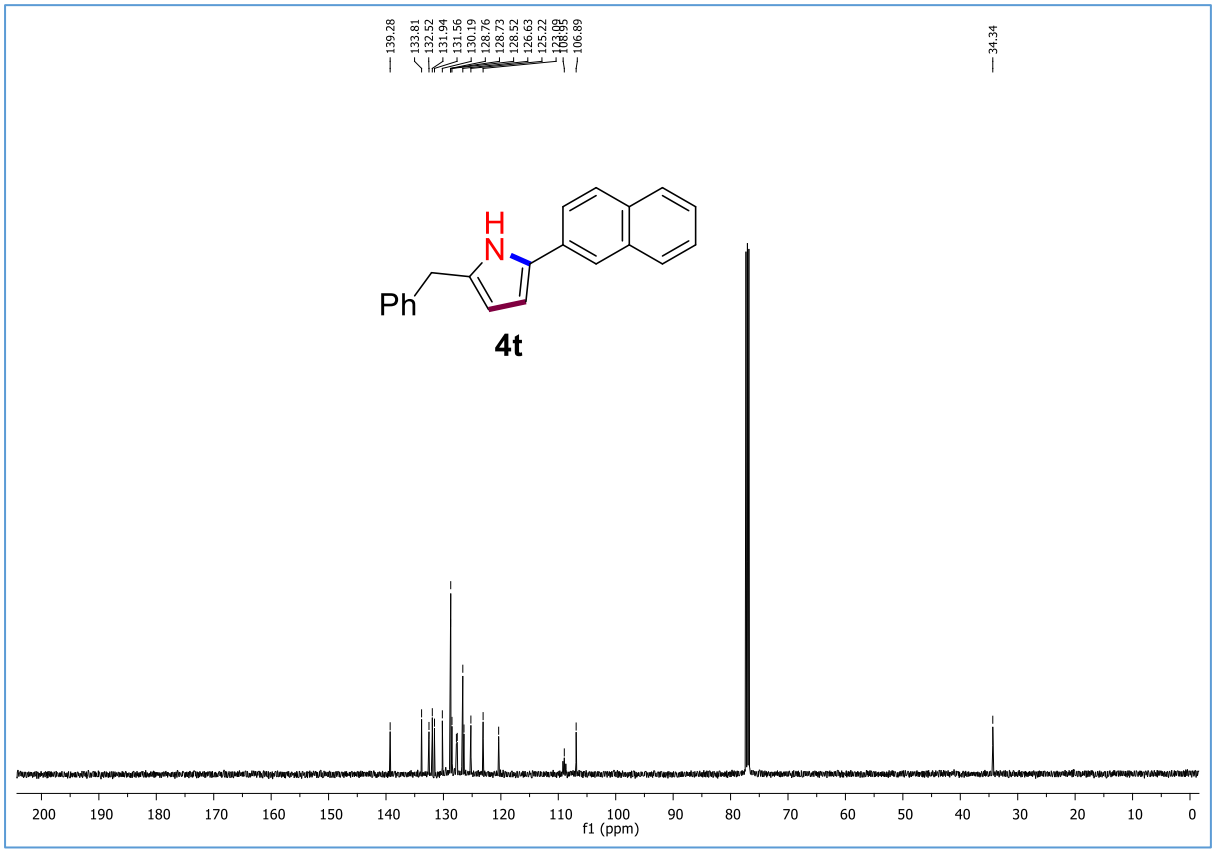
7.79
7.76
7.65
7.39
7.37
7.33
7.32
7.29
6.62
6.61
6.61
6.15
6.14
6.14
4.10
H
N
Ph
4t
1.00
3.23
1.11
1.30
0.92
1.04
2.46
3.29
1.15
1.15
2.38
f1 (ppm)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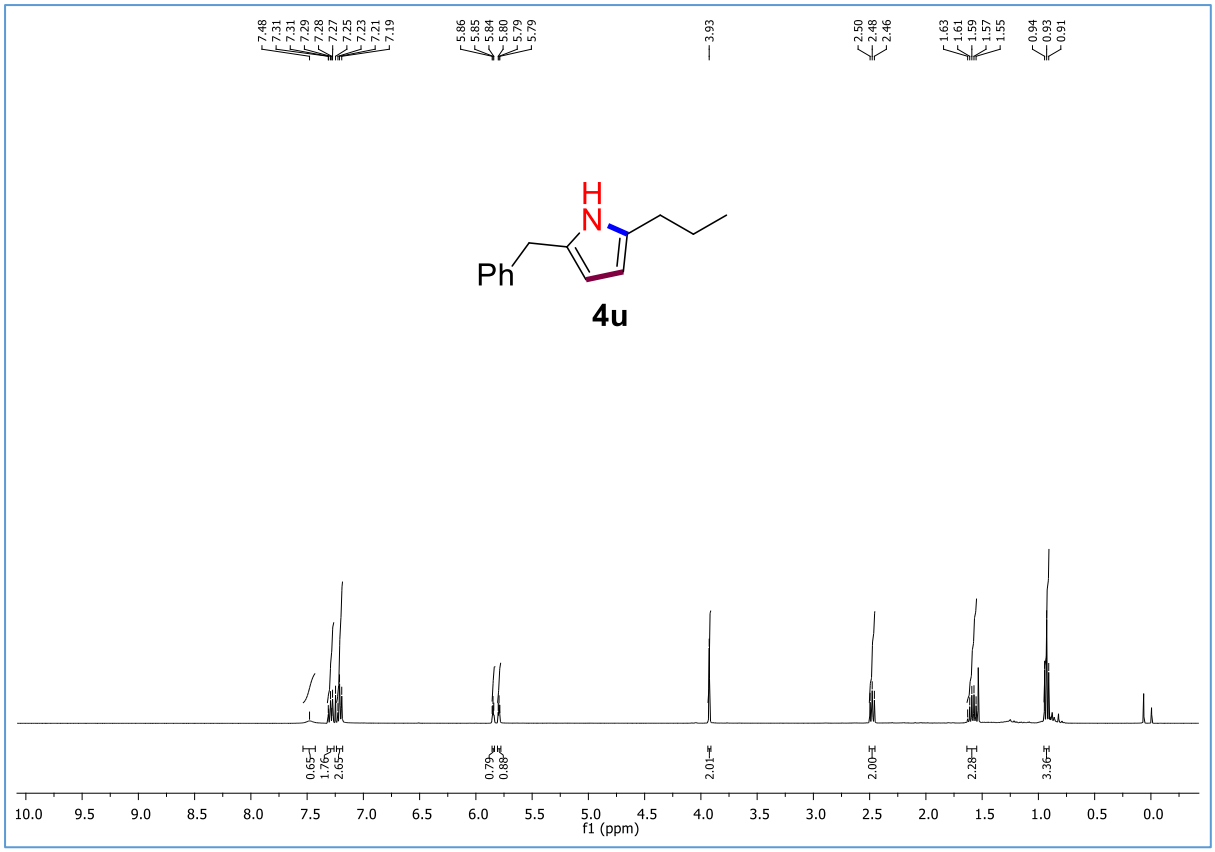
128.52
126.63
125.22
106.89
34.34
H
N
Ph
4t
f1 (ppm)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7.23
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7.19
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5.85
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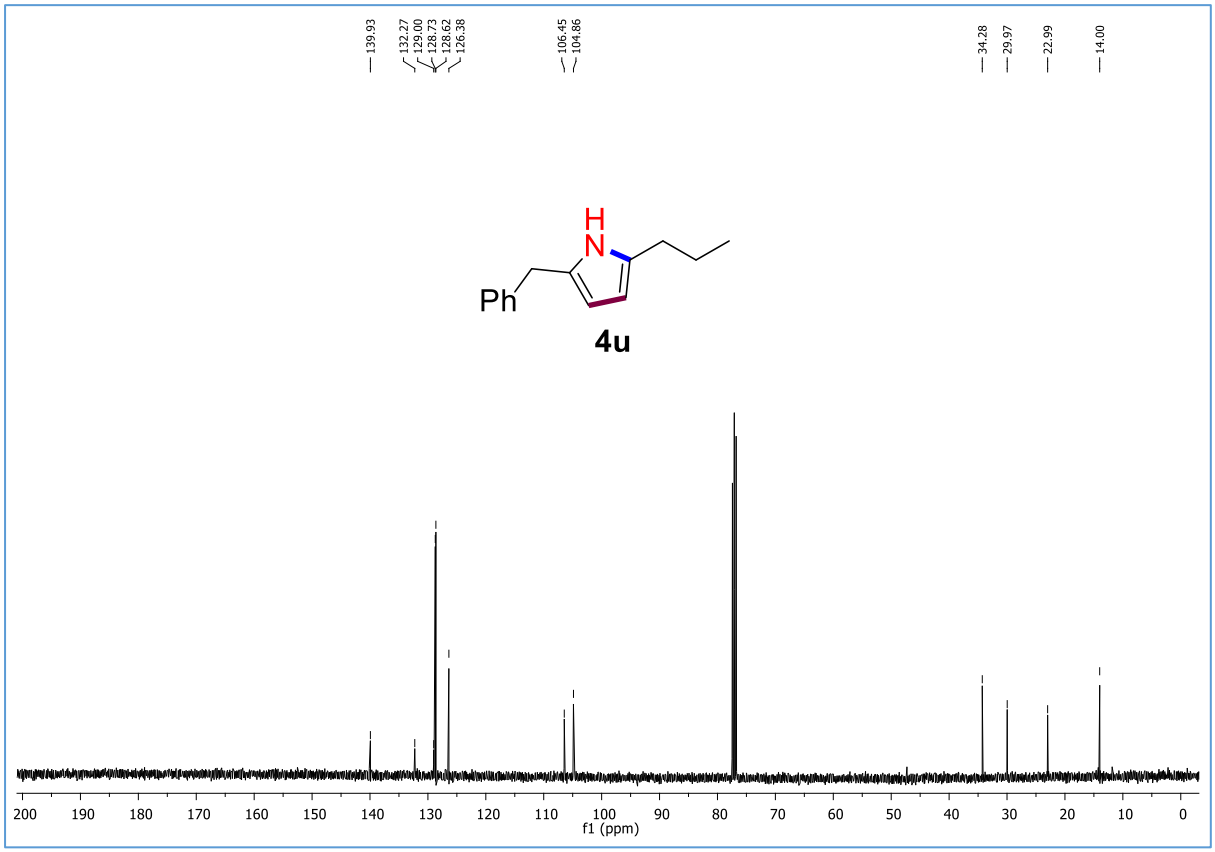
5.80
5.79
5.79
3.93
2.50
2.48
2.46
1.63
1.61
1.59
1.57
1.55
0.94
0.93
0.91
H
N
Ph
4u
0.65
1.76
2.65
0.79
0.88
2.01
2.00
2.28
3.36
10.0 9.5 9.0 8.5 8.0 7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 0.5 0.0
f1 (ppm)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106.45
104.86
34.28
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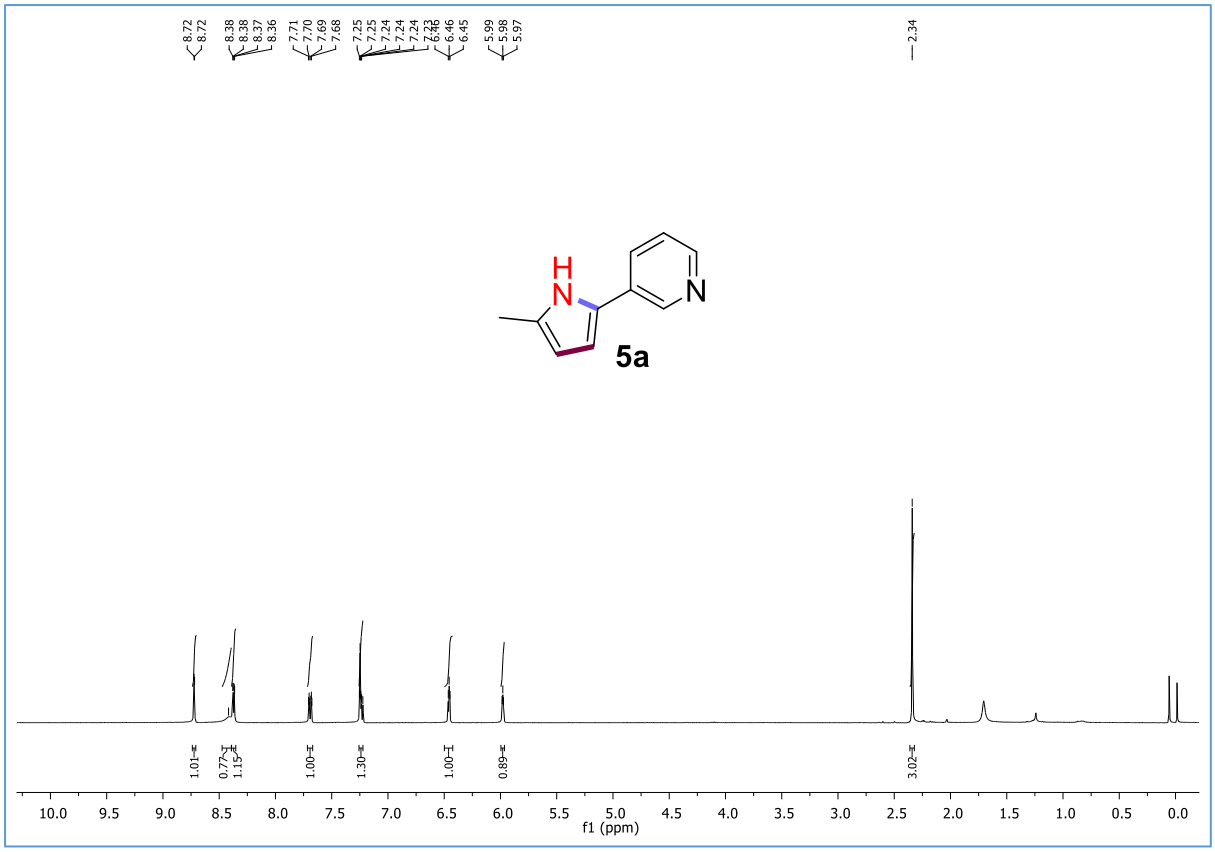
14.00
H
N
Ph
4u
200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0
f1 (ppm)


8.72
8.72
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7.71
7.70
7.69
7.68
7.25
7.25
7.24
7.24
7.24
6.46
6.45
5.99
5.98
5.97
2.34
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N
N
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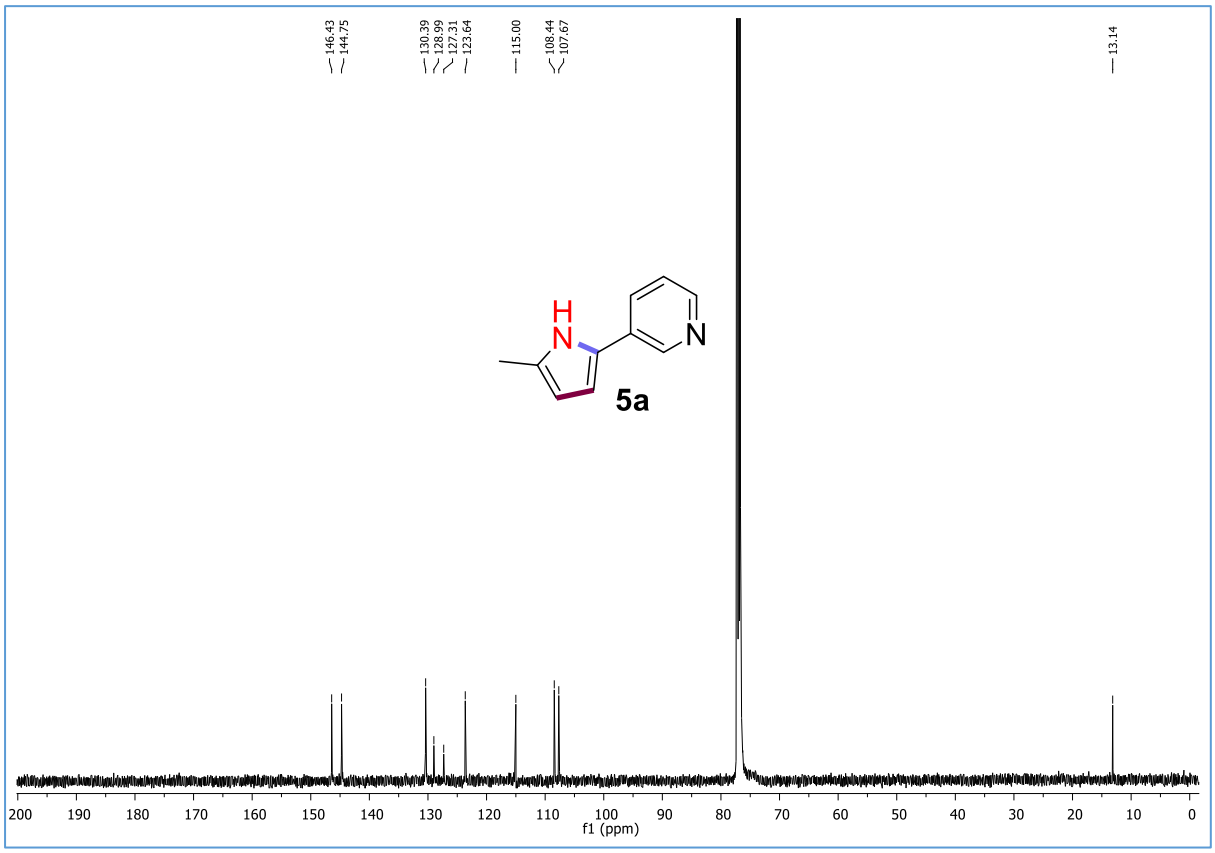
1.00
1.30
1.00
0.89
3.02
f1 (ppm)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107.67
13.14
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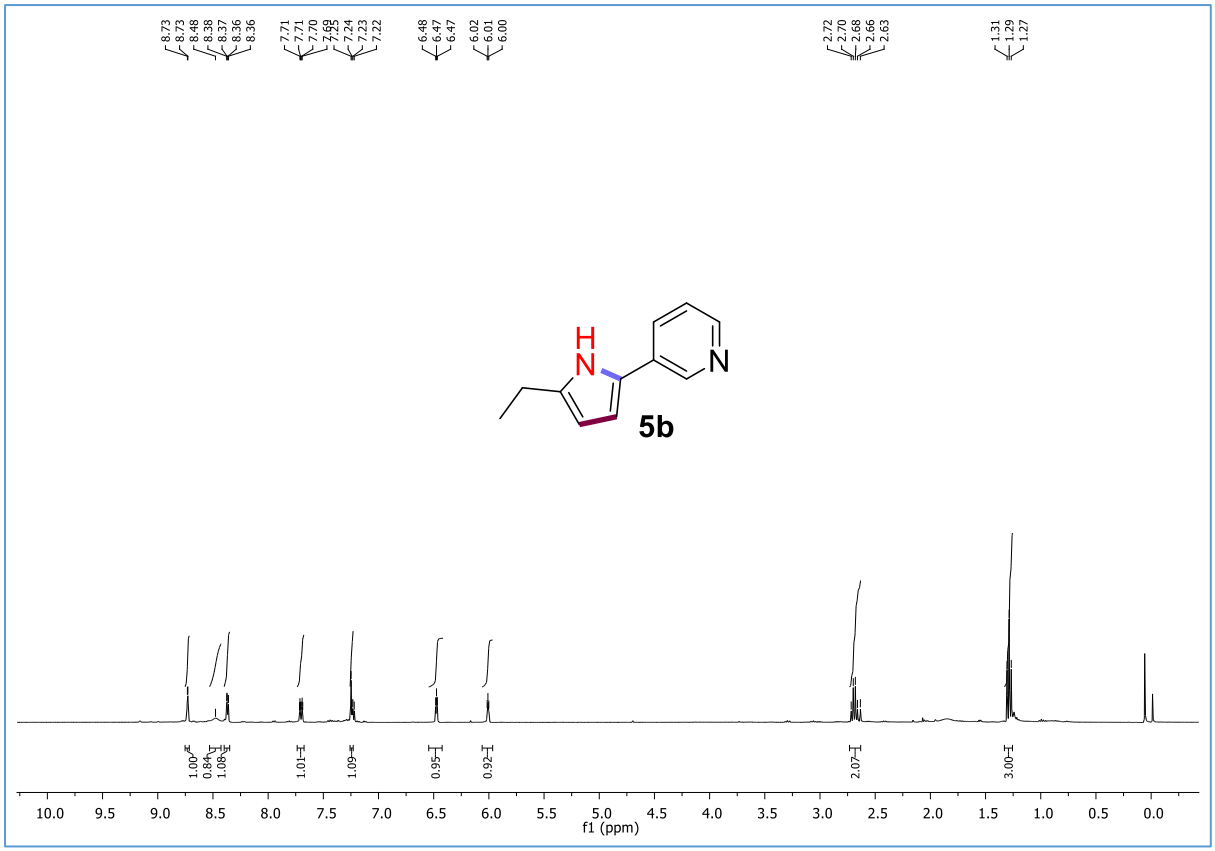
N
N
5a
f1 (ppm)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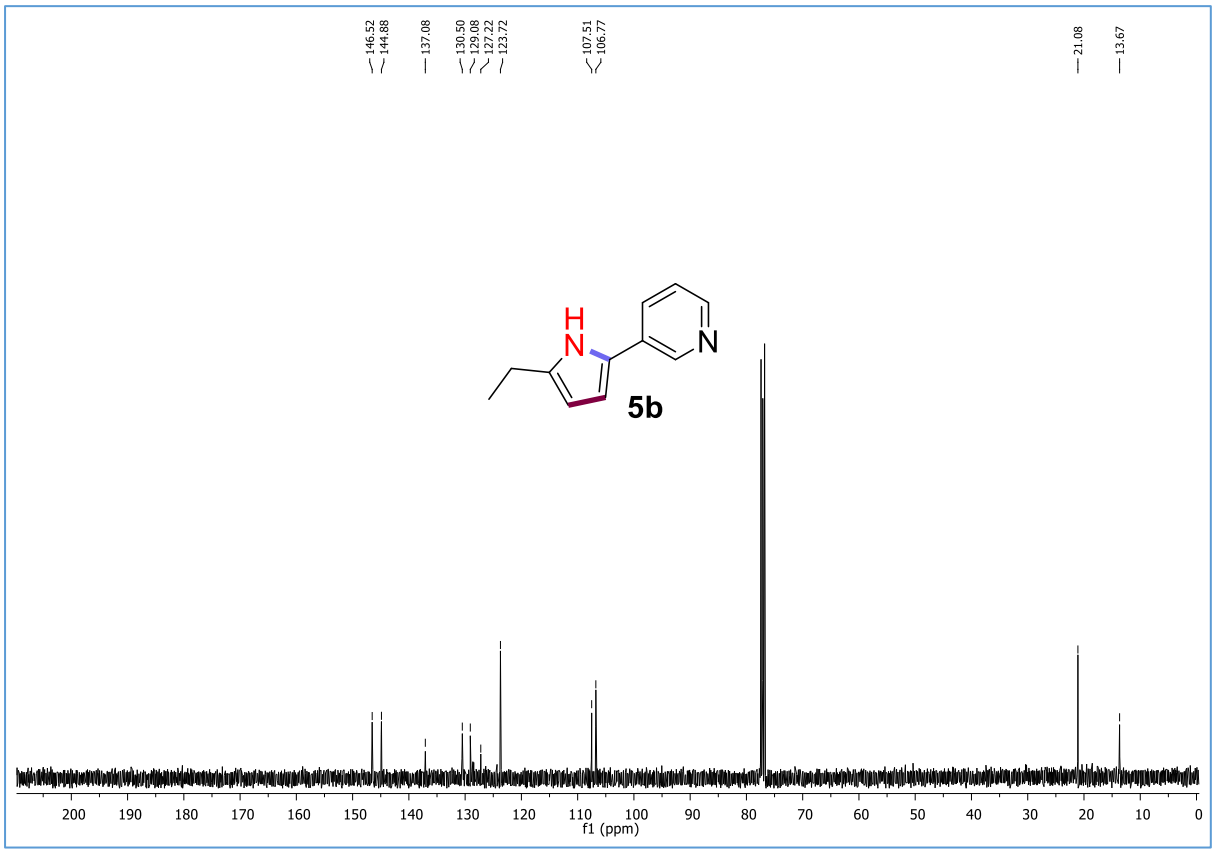
7.71
7.70
7.68
7.25
7.24
7.23
7.22
6.48
6.47
6.47
6.02
6.01
6.00
2.72
2.70
2.68
2.66
2.63
1.31
1.29
1.27
H
N
N
5b
1.00
0.84
1.08
1.01
1.09
0.95
0.92
2.07
3.00
10.0
9.5
9.0
8.5
8.0
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
0.5
0.0
f1 (ppm)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137.08
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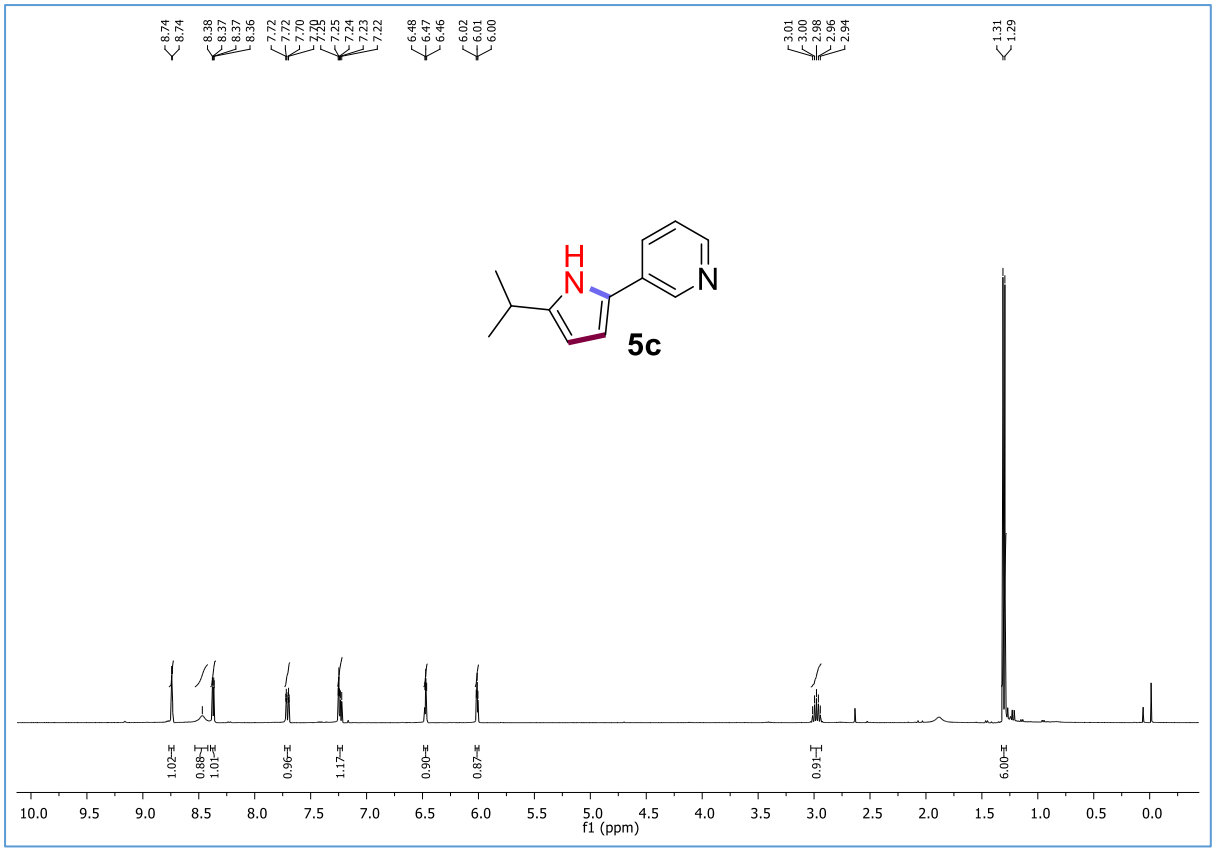
129.08
127.22
123.72
107.51
106.77
21.08
13.67
H
N
N
5b
200
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
f1 (ppm)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8.74
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7.72
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7.25
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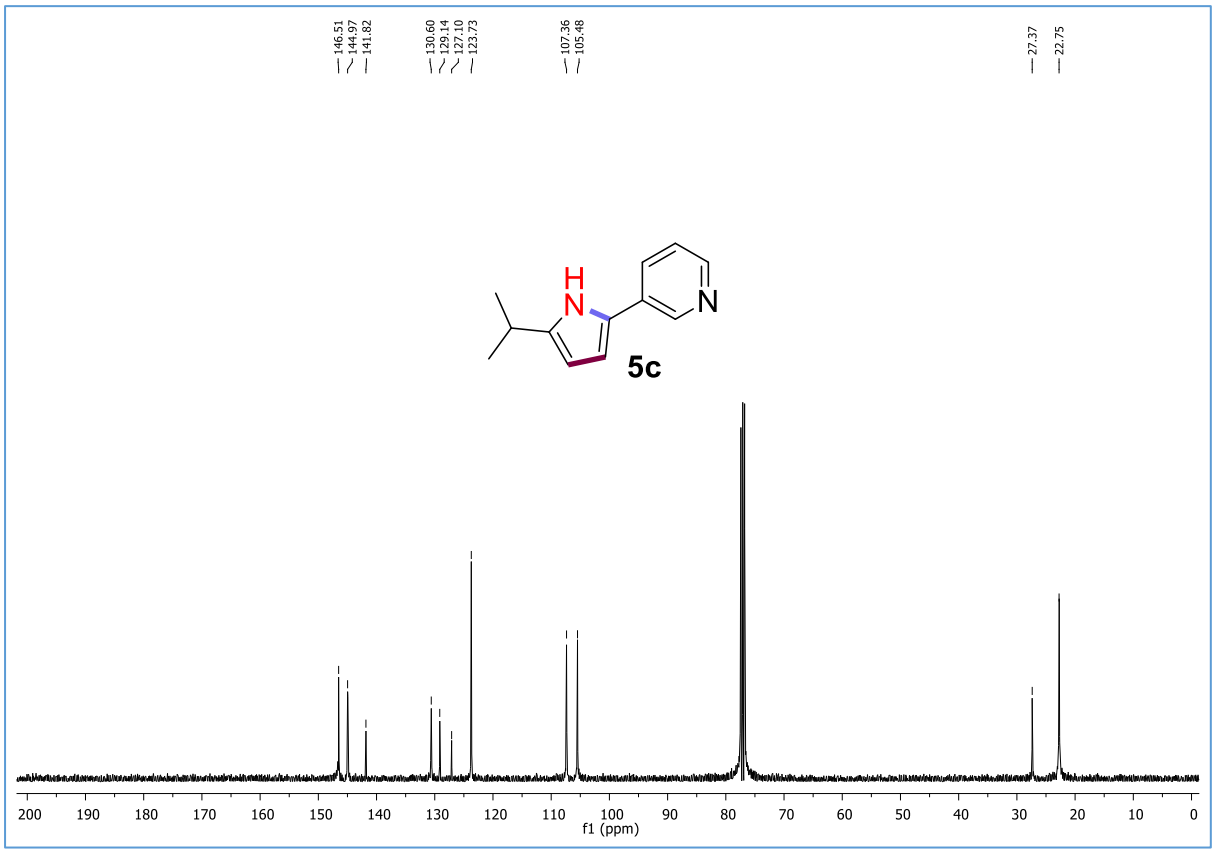
7.23
7.22
6.48
6.47
6.46
6.02
6.01
6.00
3.01
3.00
2.98
2.96
2.94
1.31
1.29
H
N
N
5c
1.02
0.88
1.01
0.96
1.17
0.90
0.87
0.91
6.00
10.0 9.5 9.0 8.5 8.0 7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 0.5 0.0
f1 (ppm)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129.14
127.10
123.73
107.36
105.48
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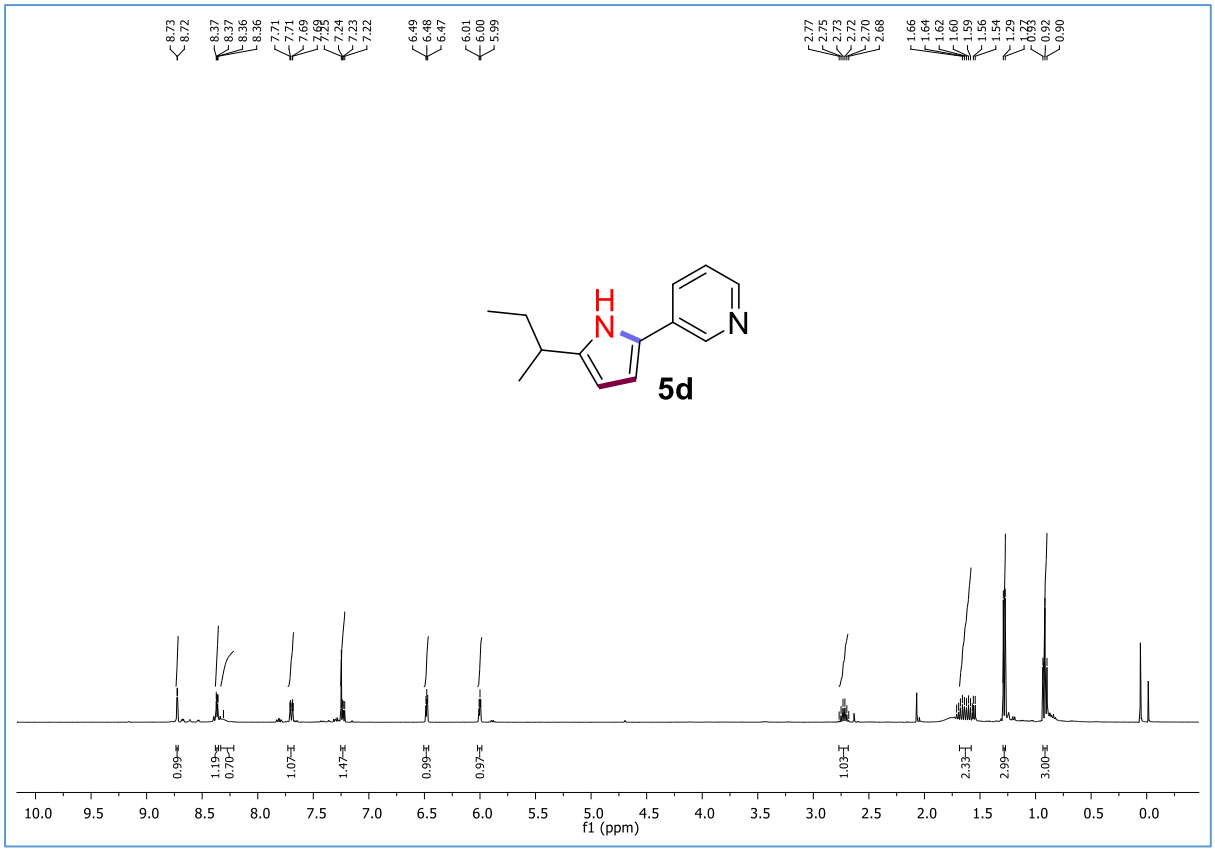
22.75
H
N
N
5c
200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0
f1 (ppm)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8.72
8.37
8.37
8.36
8.36
7.71
7.71
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7.69
7.24
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7.22
6.49
6.48
6.47
6.01
6.00
5.99
2.77
2.75
2.73
2.72
2.70
2.68
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1.64
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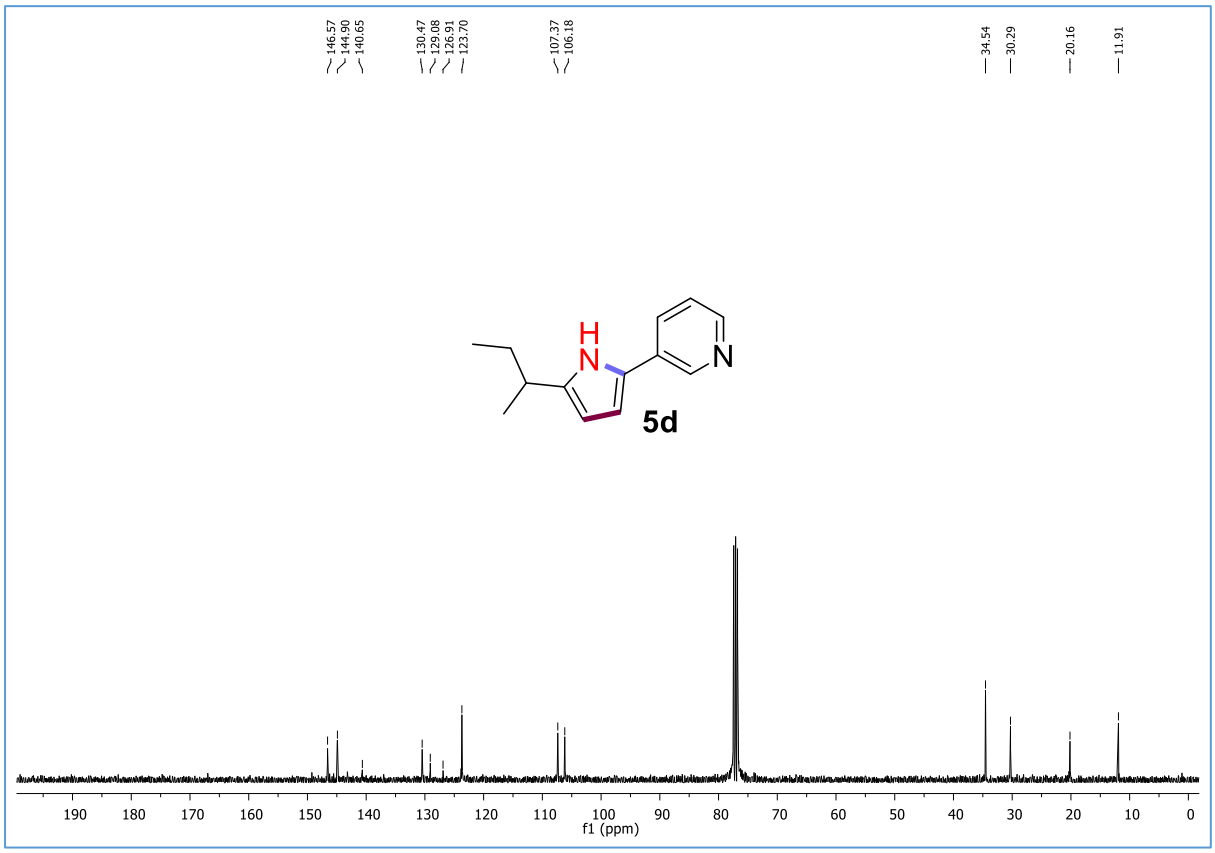
1.60
1.59
1.56
1.54
1.29
0.93
0.92
0.90
H
N
N
5d
0.99
1.19
0.70
1.07
1.47
0.99
0.97
1.03
2.33
2.99
3.00
10.0 9.5 9.0 8.5 8.0 7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 0.5 0.0
f1 (ppm)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11.91
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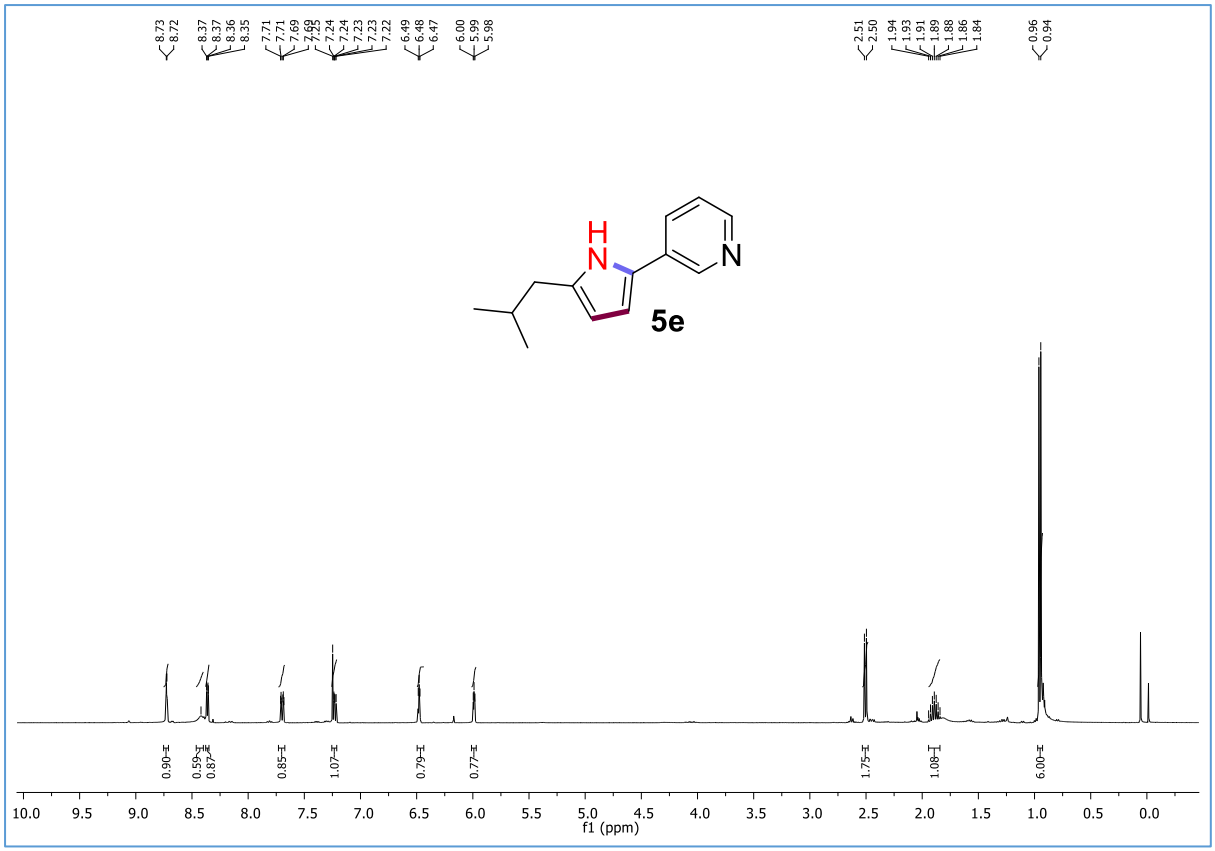
N
N
5d
190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0
f1 (ppm)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7.24
7.23
7.23
7.22
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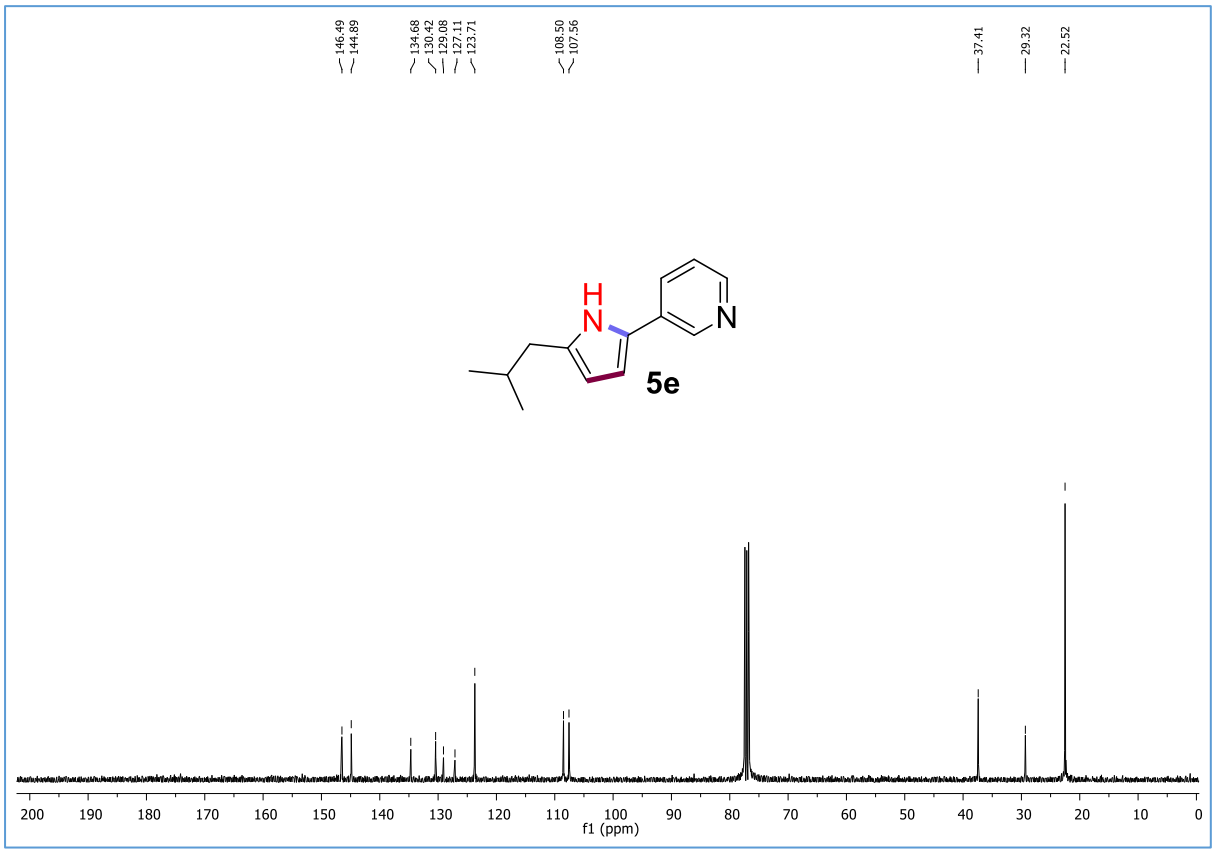
6.48
6.47
6.00
5.99
5.98
2.51
2.50
1.94
1.93
1.91
1.89
1.88
1.86
1.84
0.96
0.94
H
N
N
5e
0.90
0.59
0.87
0.85
1.07
0.79
0.77
1.75
1.08
6.00
10.0 9.5 9.0 8.5 8.0 7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 0.5 0.0
f1 (ppm)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22.52
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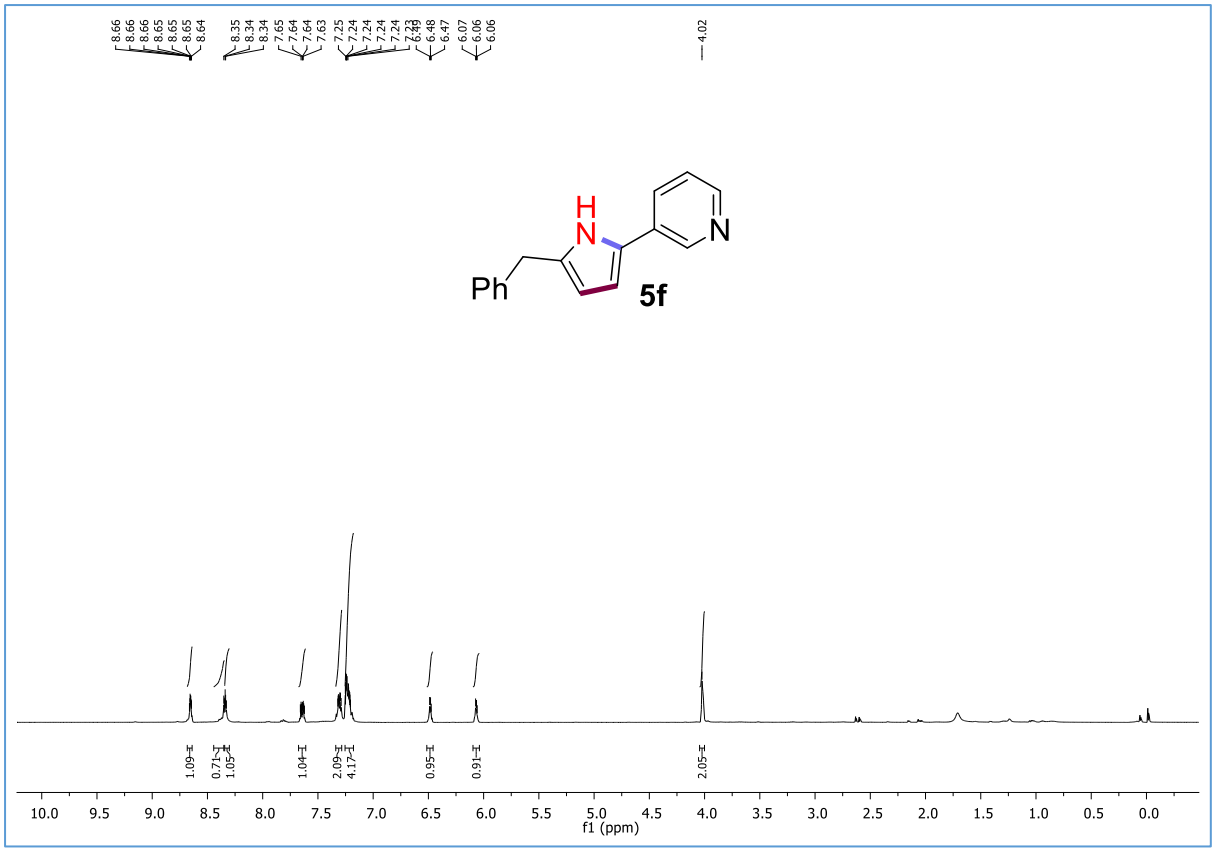
N
N
5e
200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0
f1 (ppm)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7.63
7.25
7.24
7.24
7.24
7.24
7.23
6.49
6.48
6.47
6.07
6.06
6.06
4.02
H
N
N
Ph
5f
1.09
0.71
1.05
1.04
2.09
4.17
0.95
0.91
2.05
10.0
9.5
9.0
8.5
8.0
7.5
7.0
6.5
6.0
5.5
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2.5
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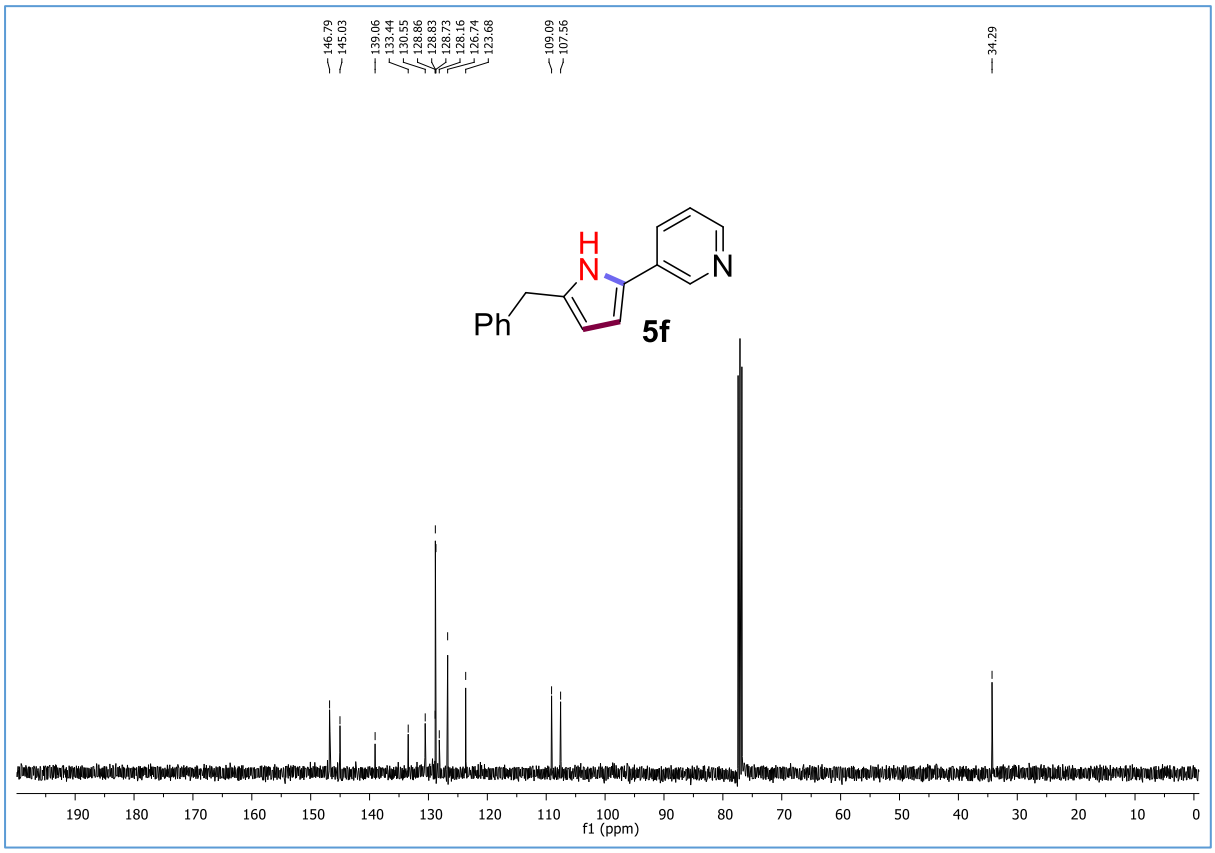
0.0
f1 (ppm)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128.83
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128.16
126.74
123.68
109.09
107.56
34.29
H
N
N
Ph
5f
190
180
170
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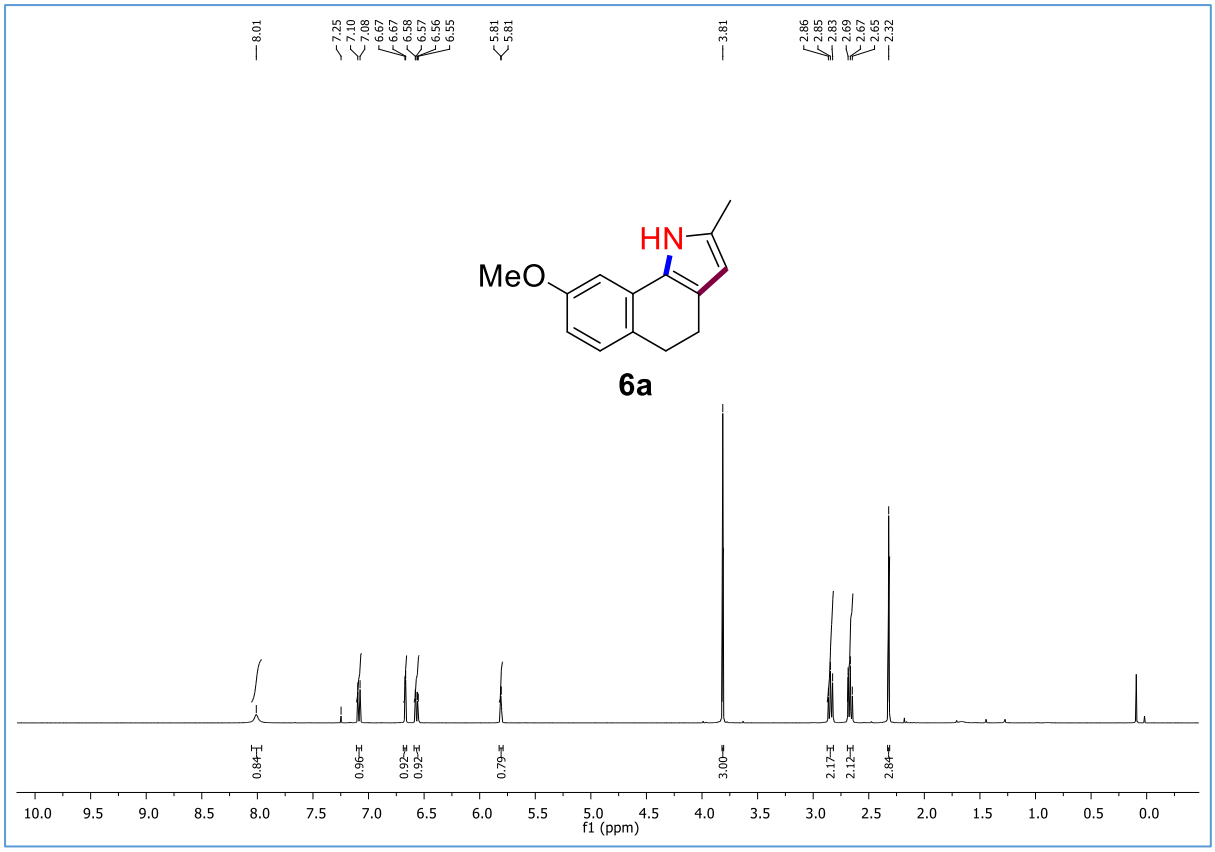
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
f1 (ppm)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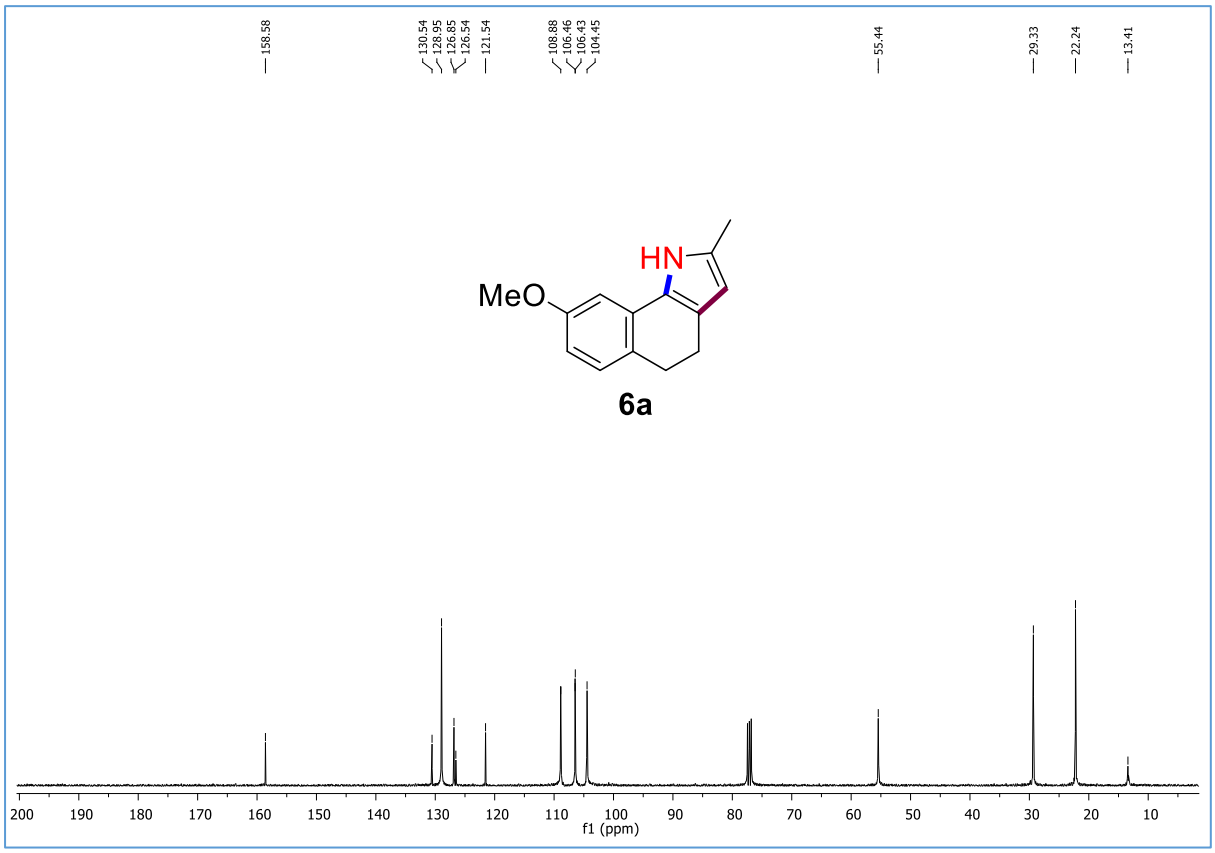
6.56
6.55
5.81
5.81
3.81
2.86
2.85
2.83
2.69
2.67
2.65
2.32
HN
MeO
6a
0.84
0.96
0.92
0.92
0.79
3.00
2.17
2.12
2.84
10.0 9.5 9.0 8.5 8.0 7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 0.5 0.0
f1 (ppm)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126.85
126.54
121.54
108.88
106.46
106.43
104.45
55.44
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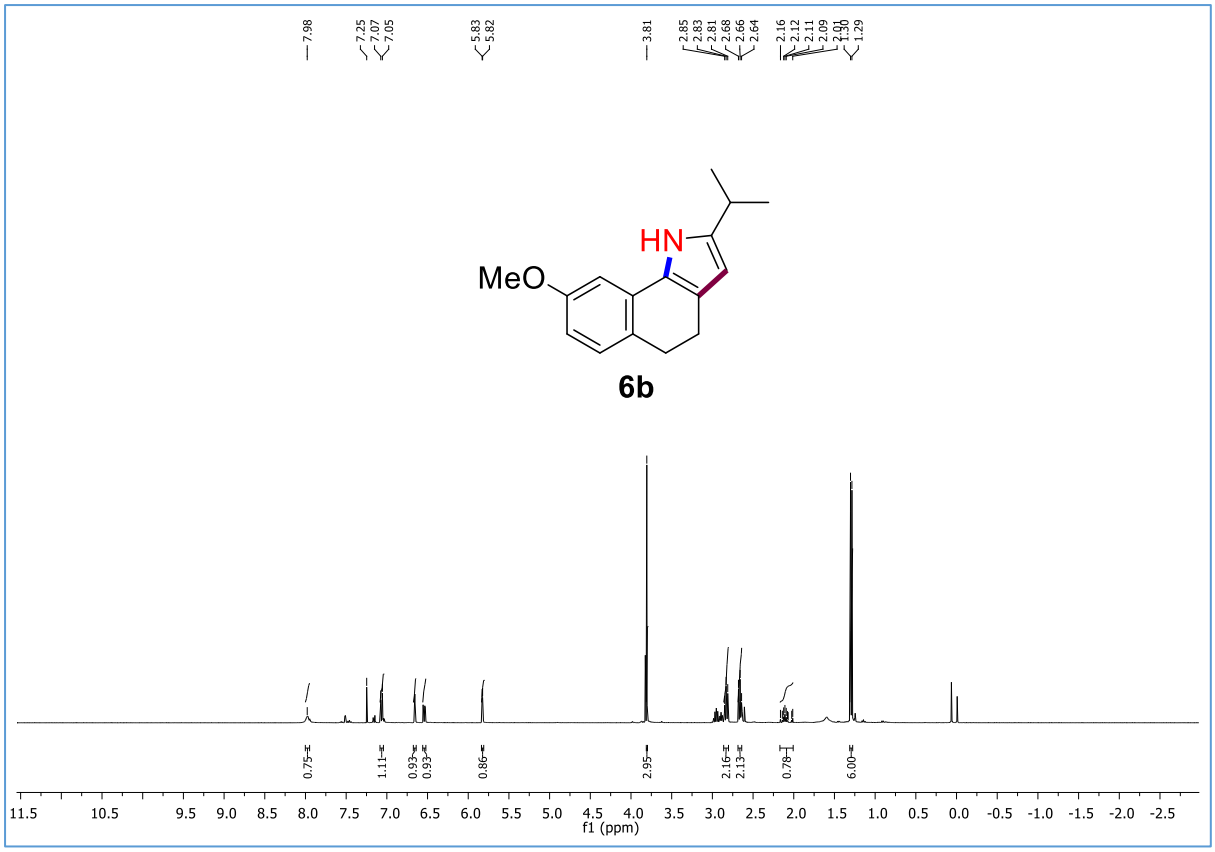
22.24
13.41
HN
MeO
6a
200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10
f1 (ppm)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5.82
3.81
2.85
2.83
2.81
2.68
2.66
2.64
2.16
2.12
2.11
2.09
1.30
1.29
HN
MeO
6b
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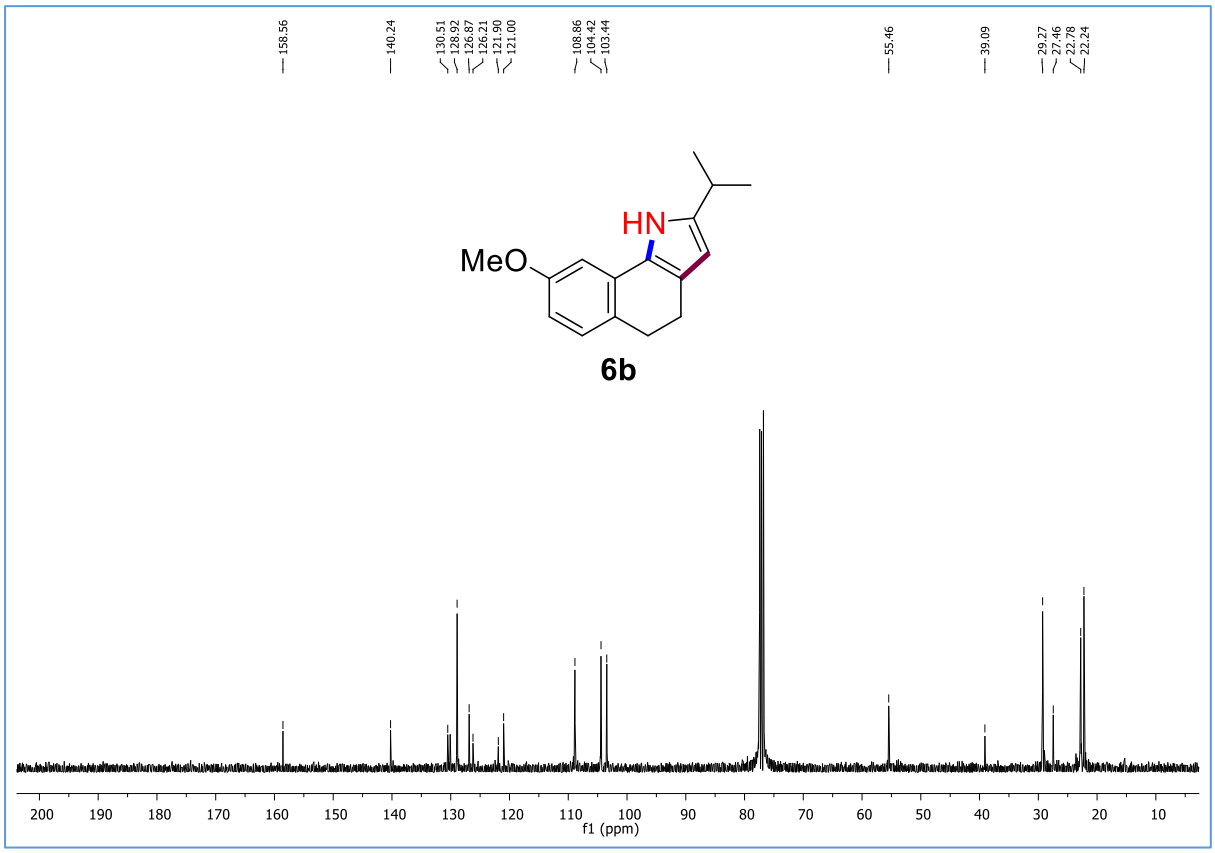
1.11
0.93
0.93
0.86
2.95
2.16
2.13
0.78
6.00
f1 (ppm)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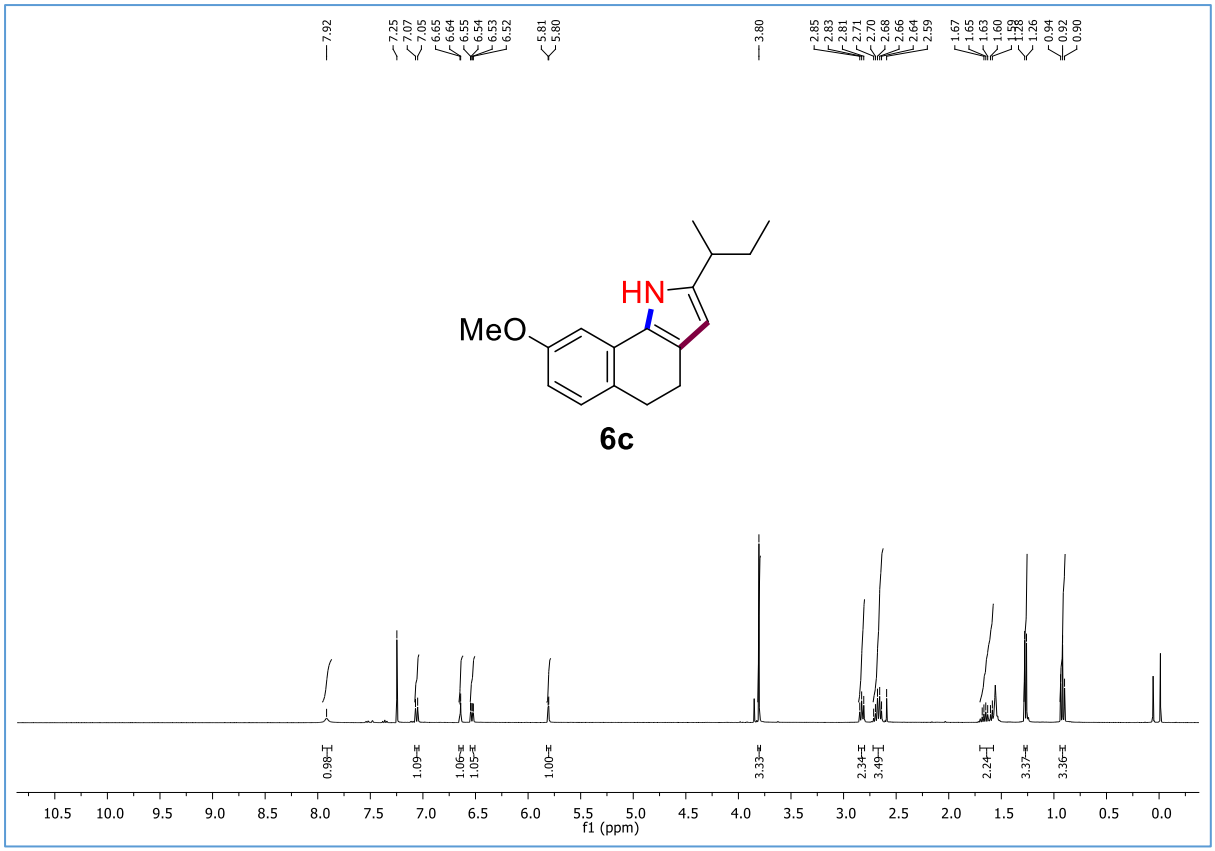
126.21
121.90
121.00
108.86
104.42
103.44
55.46
39.09
29.27
27.46
22.78
22.24
HN
MeO
6b
f1 (ppm)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6.64
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6.54
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6.52
5.81
5.80
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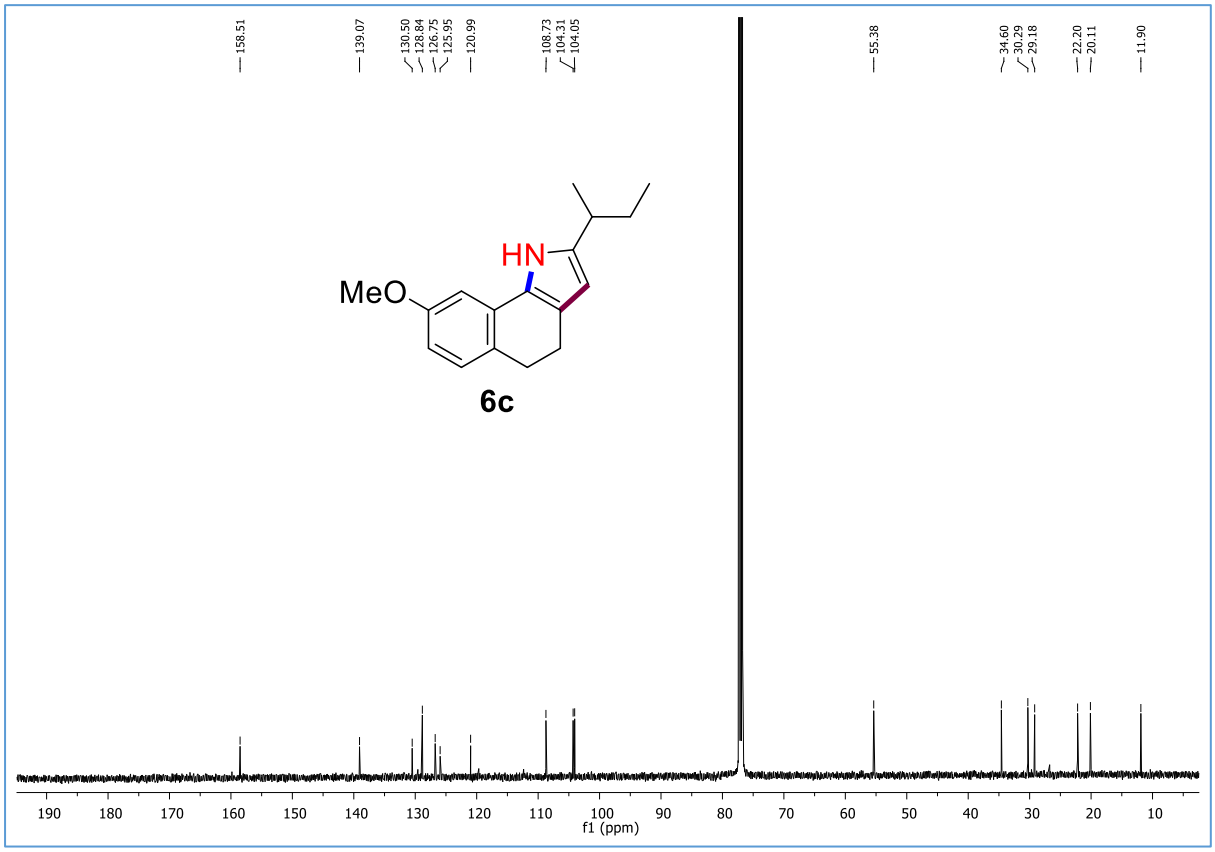
2.85
2.83
2.81
2.71
2.70
2.68
2.66
2.64
2.59
1.67
1.65
1.63
1.60
1.59
1.28
1.26
0.94
0.92
0.90
HN
MeO
6c
0.98
1.09
1.06
1.05
1.00
3.33
2.34
3.49
2.24
3.37
3.36
f1 (ppm)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108.73
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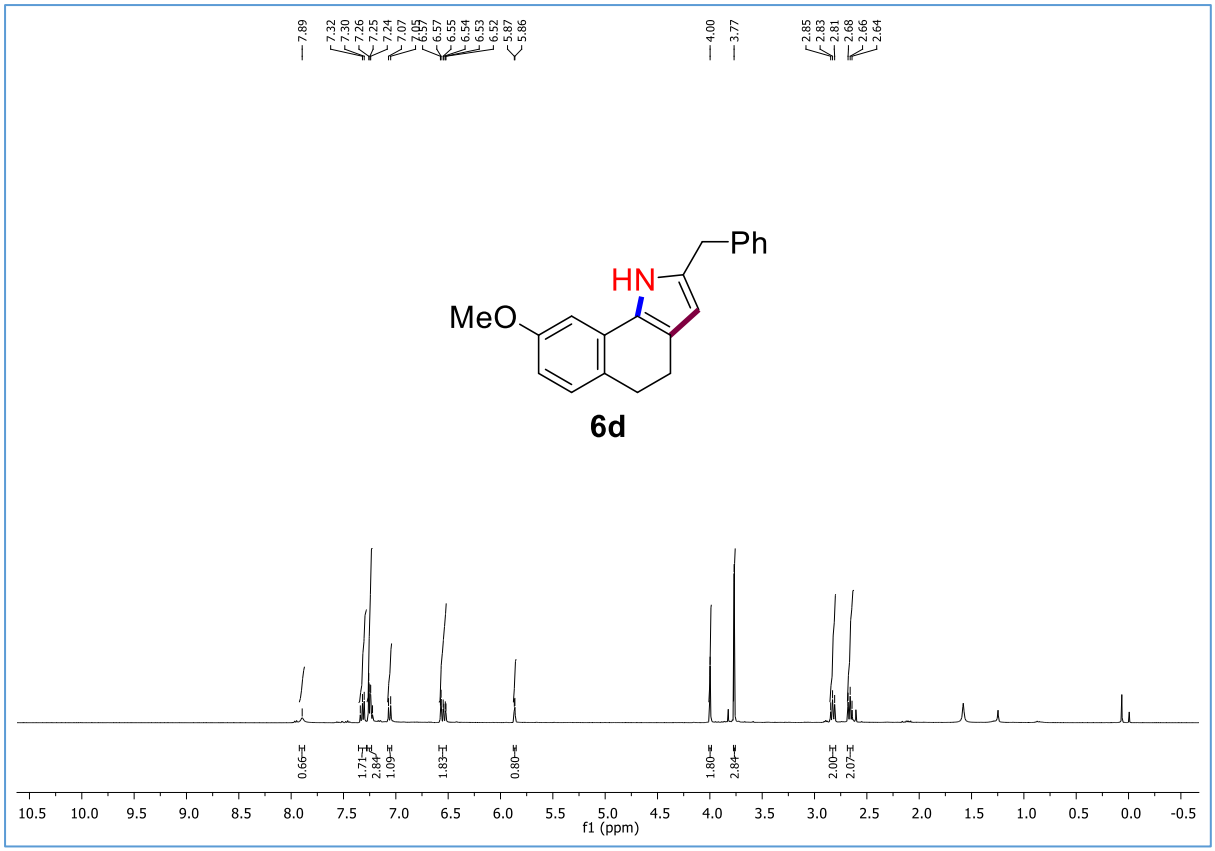
30.29
29.18
22.20
20.11
11.90
HN
MeO
6c
f1 (ppm)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7.32
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7.24
7.07
6.57
6.55
6.54
6.53
6.52
5.87
5.86
4.00
3.77
2.85
2.83
2.81
2.68
2.66
2.64
Ph
HN
MeO
6d
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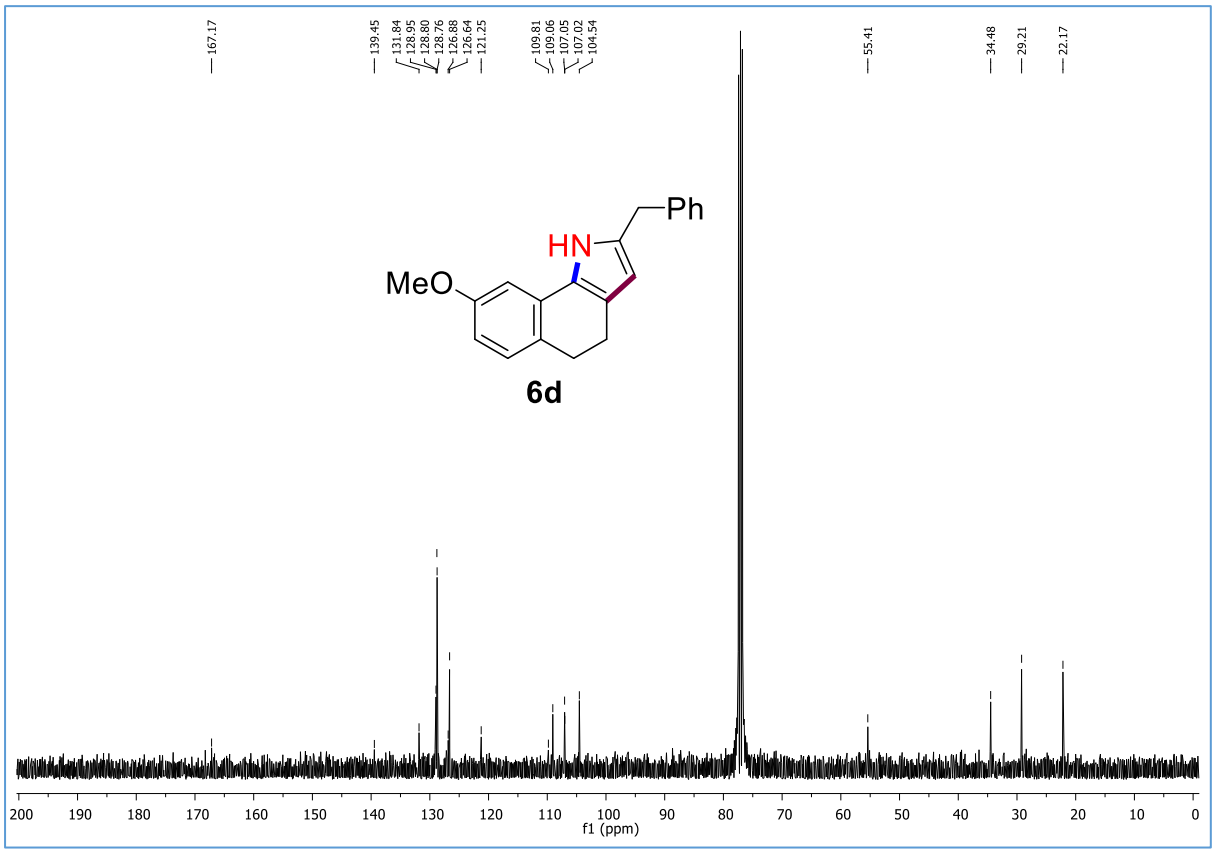
2.84
1.09
1.83
0.80
1.80
2.84
2.00
2.07
f1 (ppm)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126.88
126.64
121.25
109.81
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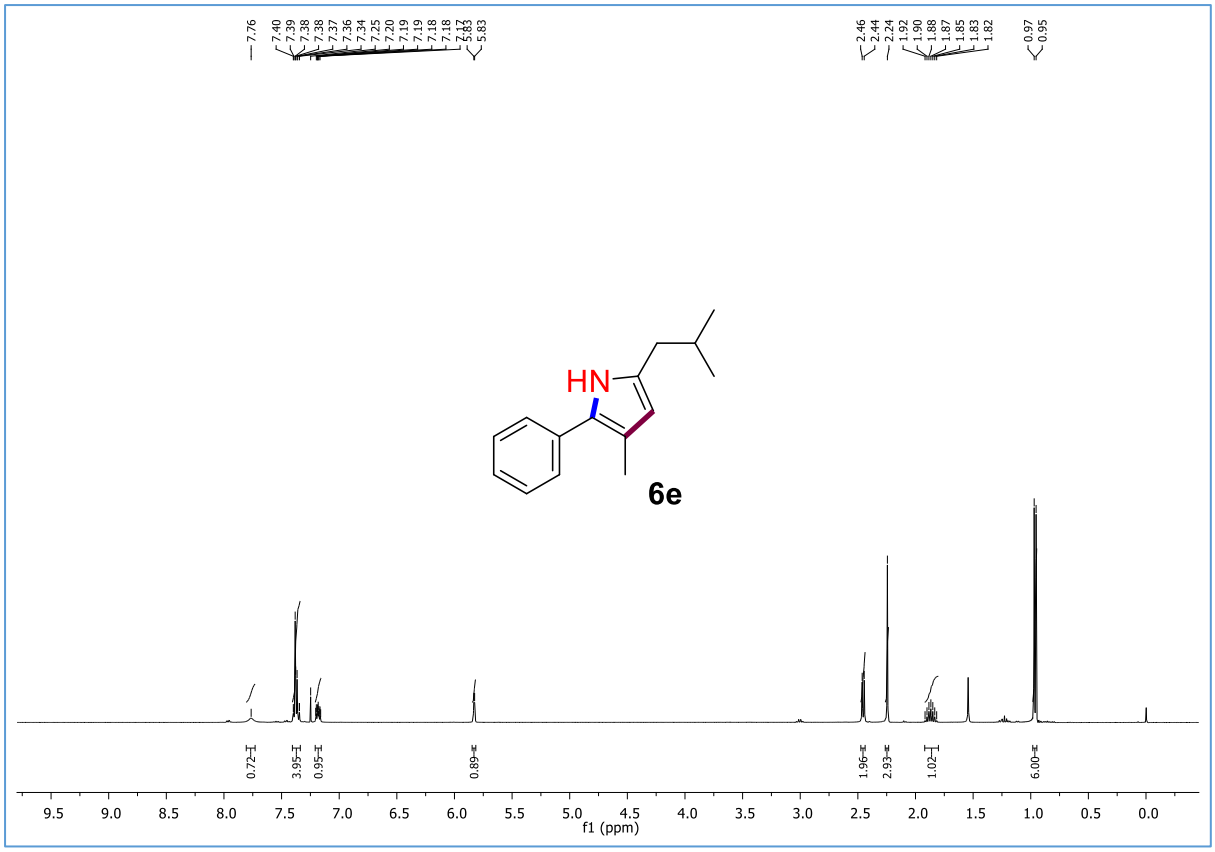
107.02
104.54
55.41
34.48
29.21
22.17
Ph
HN
MeO
6d
f1 (ppm)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7.34
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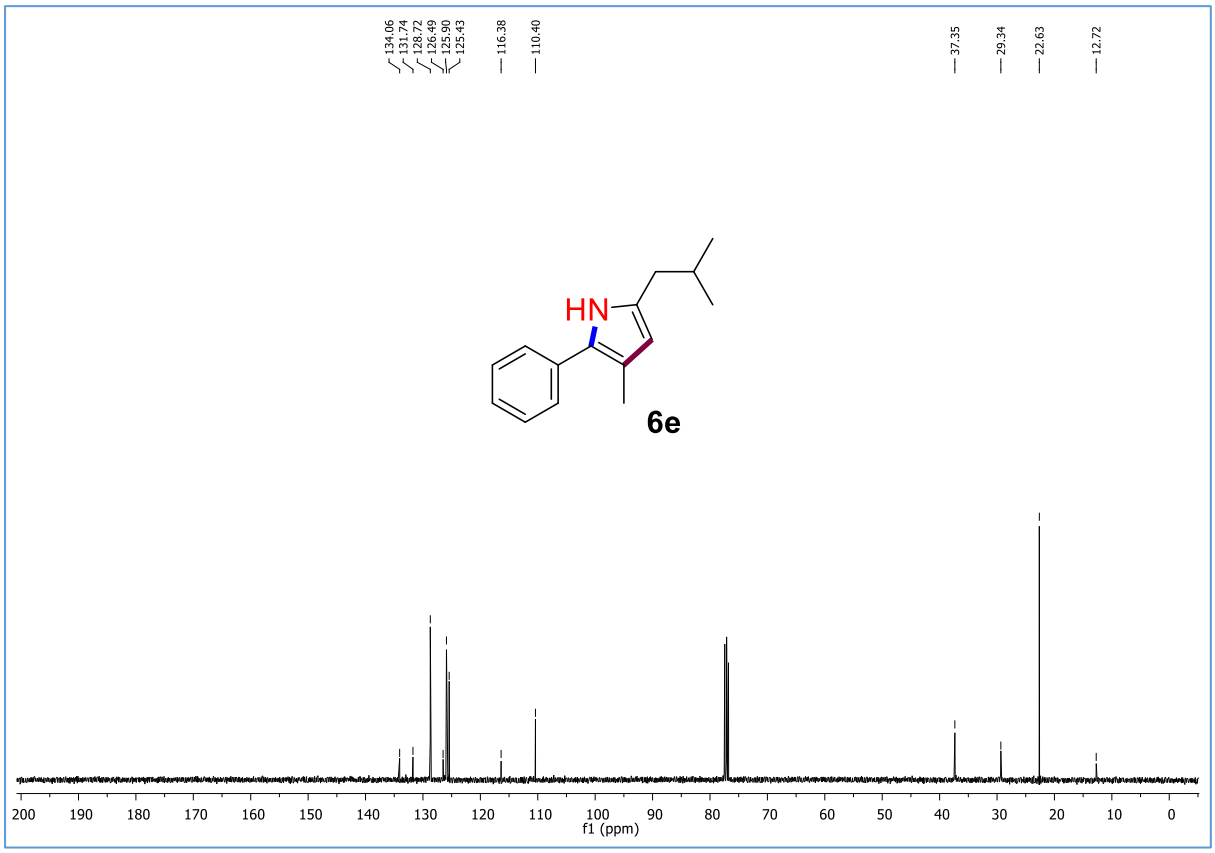
7.20
7.19
7.19
7.18
7.18
5.83
2.46
2.44
2.24
1.92
1.90
1.88
1.87
1.85
1.83
1.82
0.97
0.95
HN
6e
0.72
3.95
0.95
0.89
1.96
2.93
1.02
6.00
f1 (ppm)


134.06
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125.43
116.38
110.40
37.35
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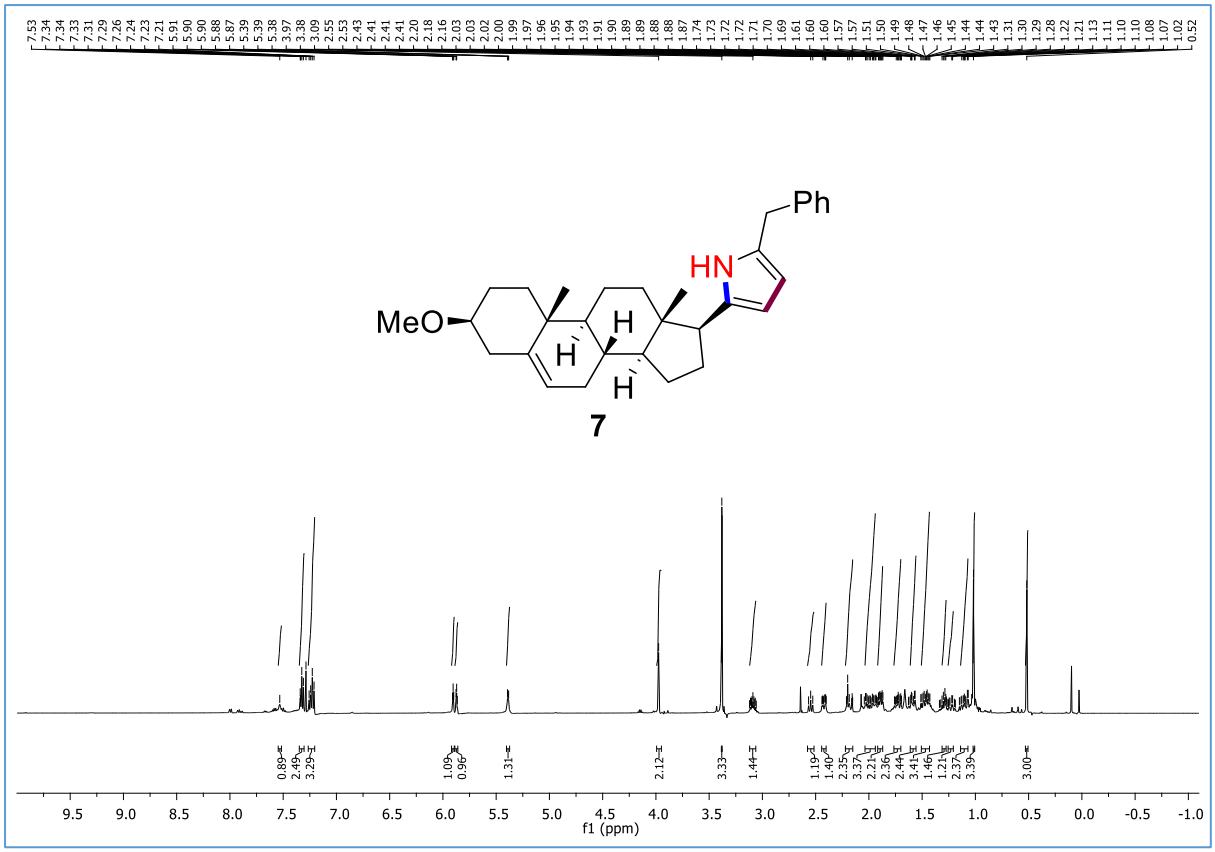
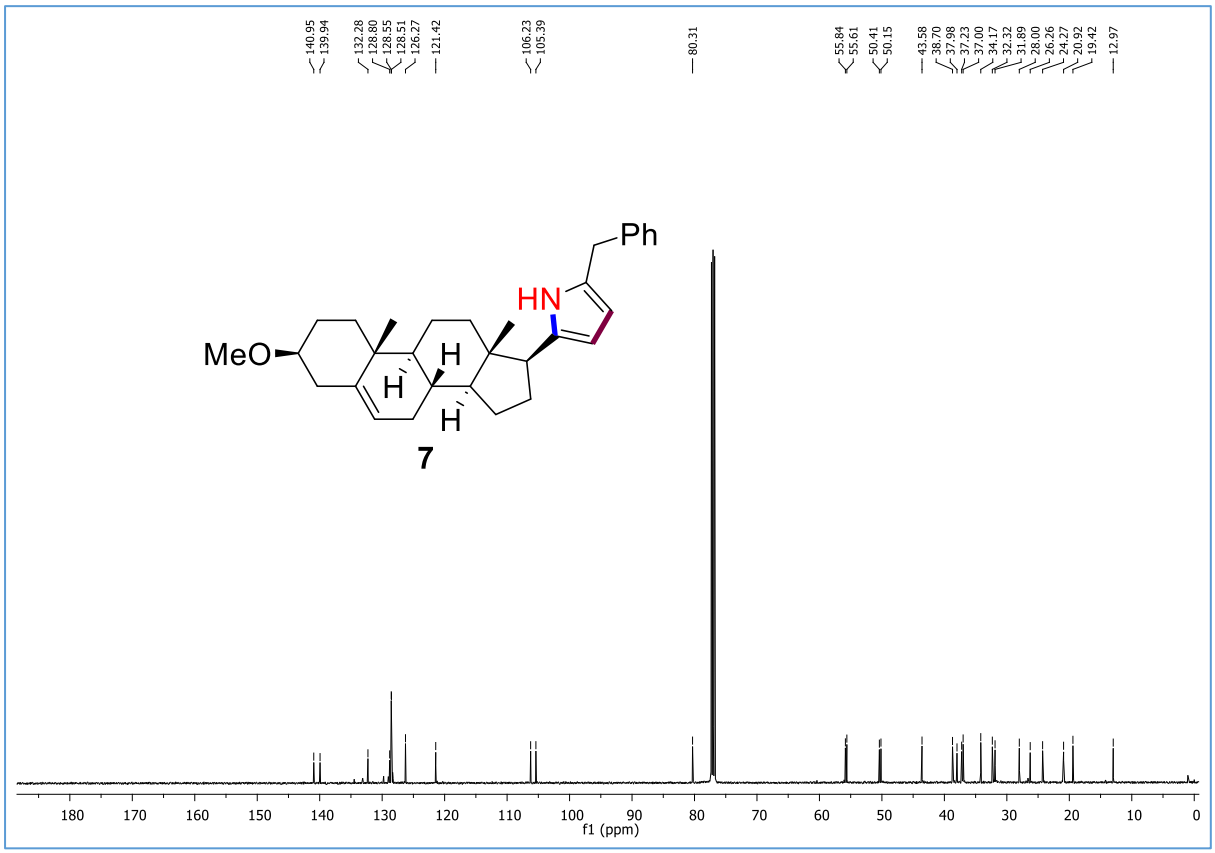
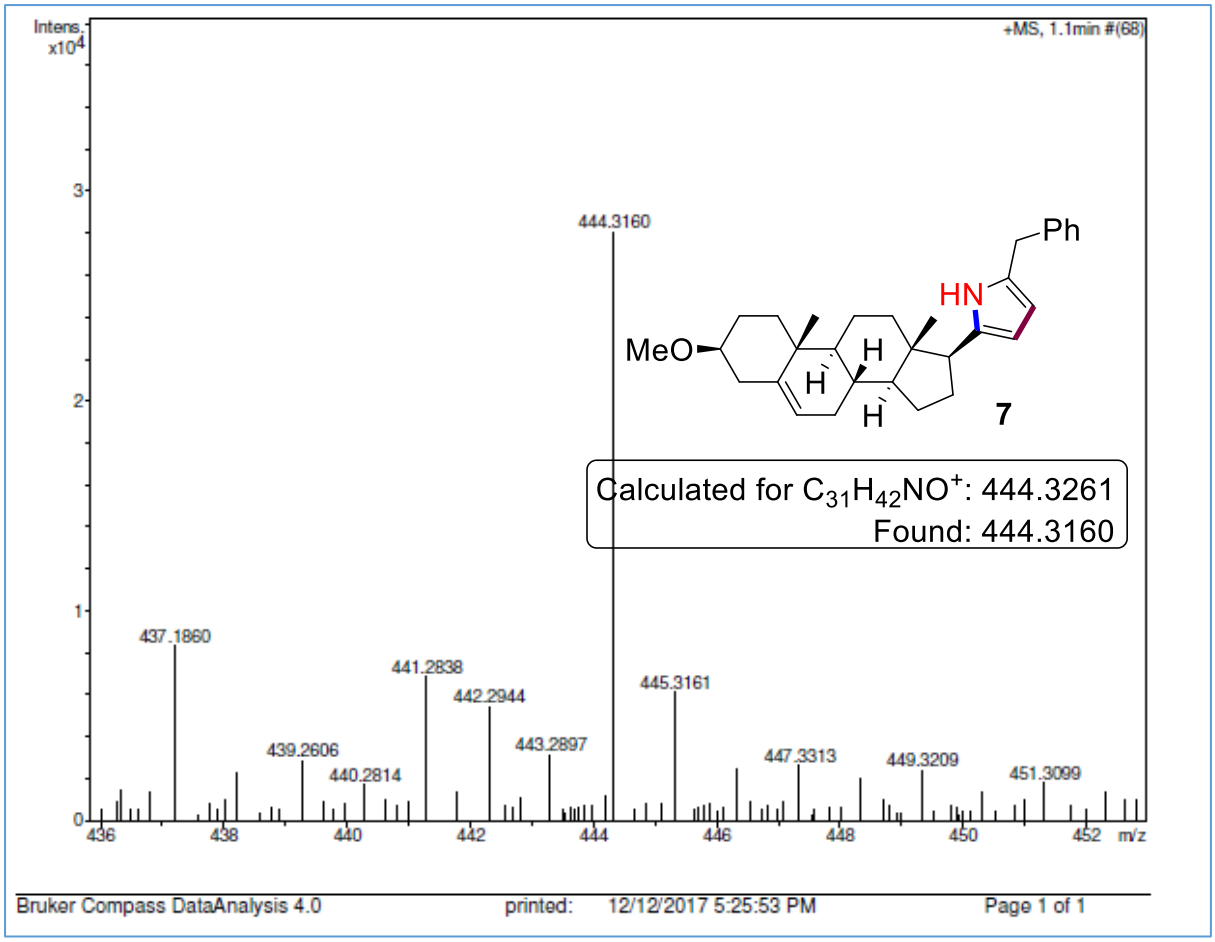
22.63
12.72
HN
6e
f1 (ppm)


Ph
HN
MeO
H
H
H
7
f1 (ppm)


Ph
HN
MeO
H
H
H
7
f1 (ppm)


Intens.
x10^4
+MS, 1.1min #(68)
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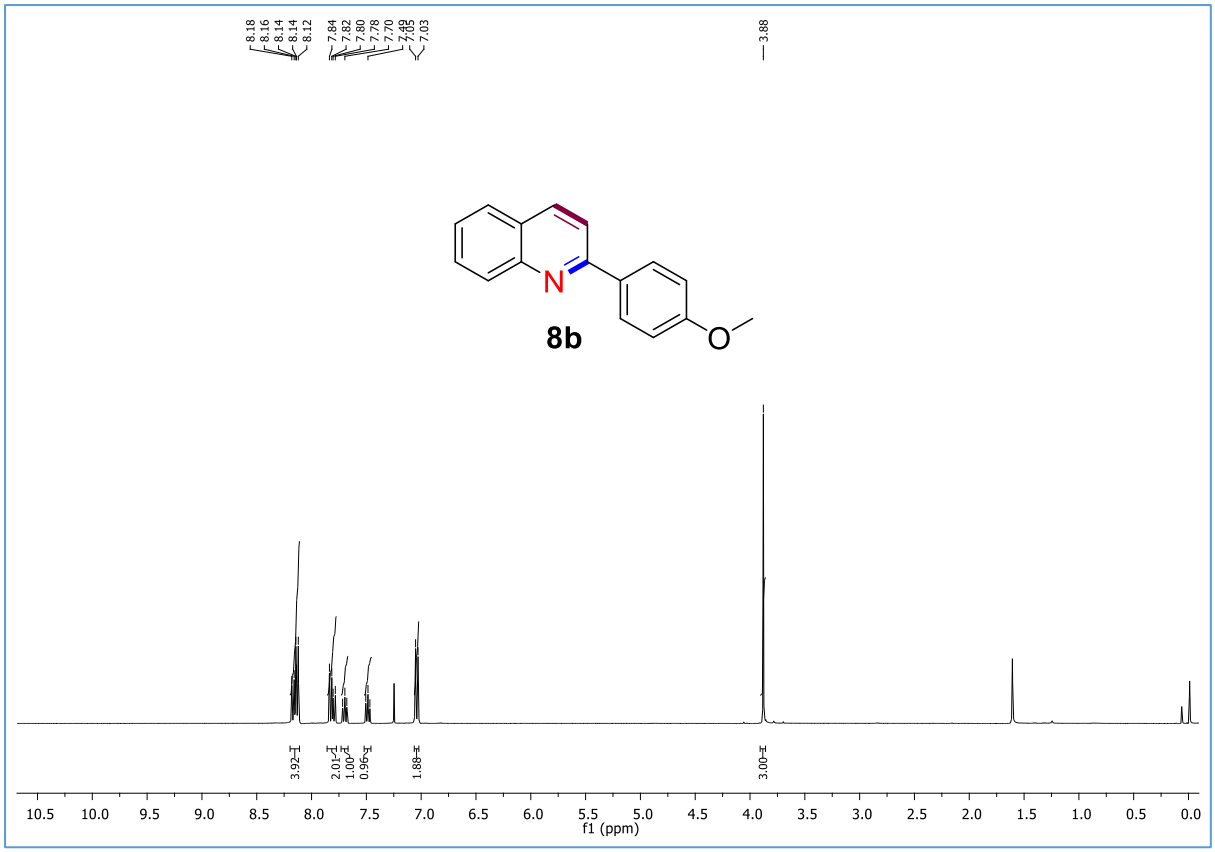
437.1860
441.2838
442.2944
445.3161
439.2606
443.2897
440.2814
447.3313
449.3209
451.3099
m/z
Ph
HN
MeO
H
H
H
7
Calculated for C31H42NO+: 444.3261
Found: 444.3160
Bruker Compass DataAnalysis 4.0
printed: 12/12/2017 5:25:53 PM
Page 1 of 1


8.18
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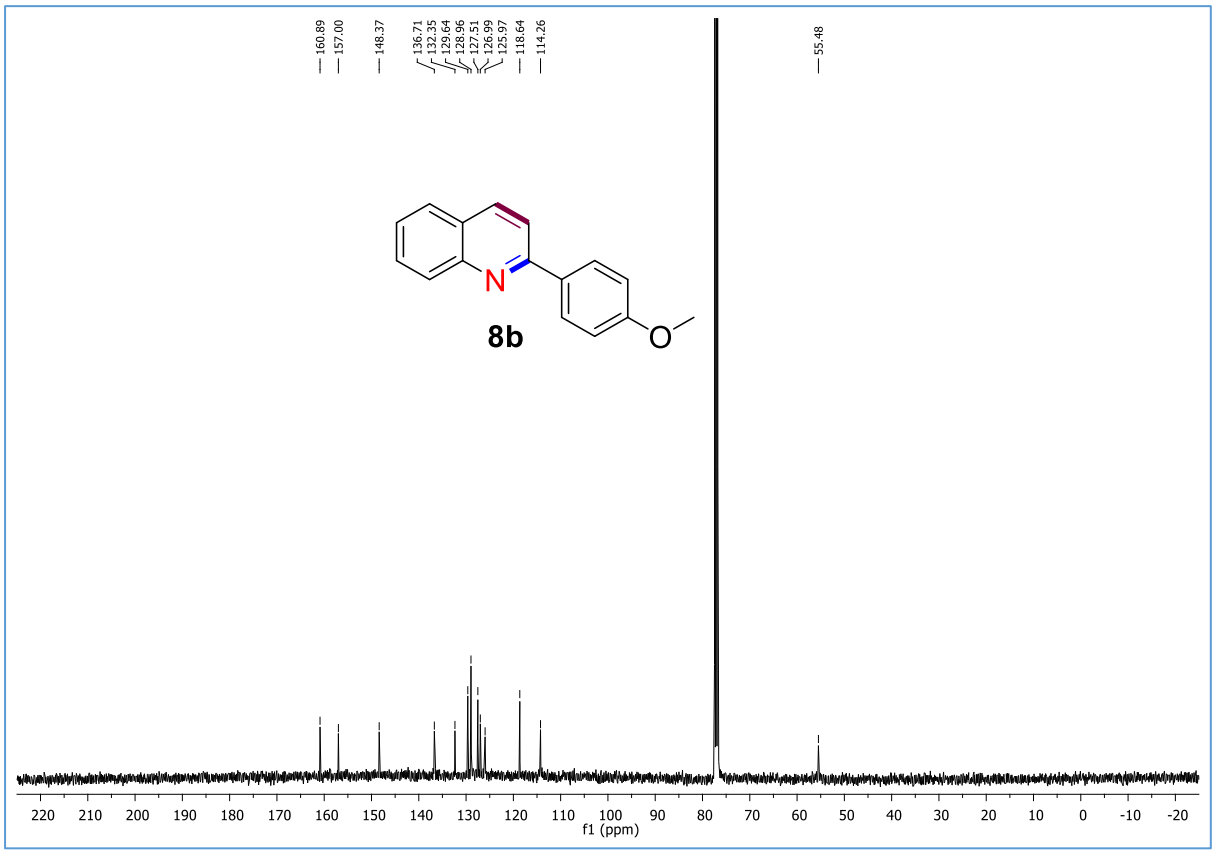
8.14
8.12
7.84
7.82
7.80
7.78
7.70
7.49
7.45
7.03
3.88
8b
3.92
2.01
1.00
0.96
1.88
3.00
f1 (ppm)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118.64
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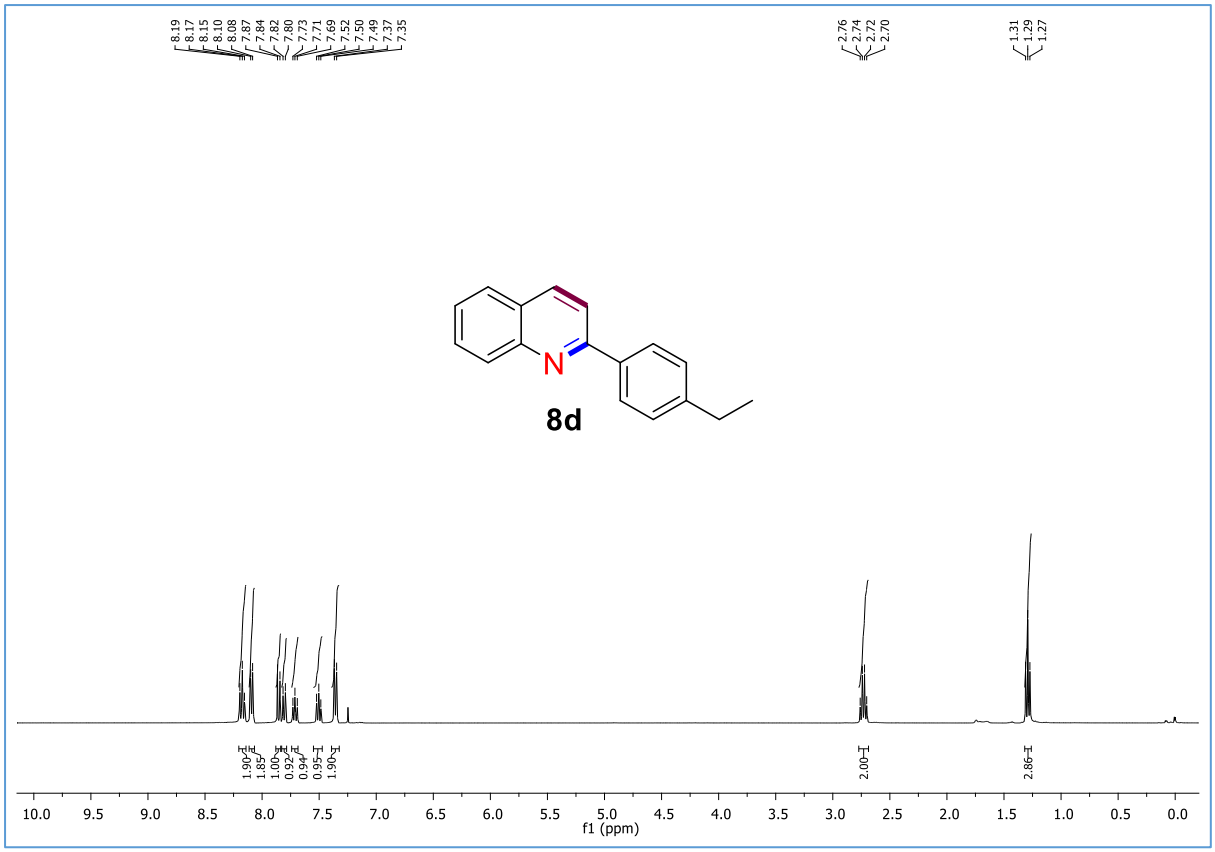
55.48
8b
f1 (ppm)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8.08
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7.84
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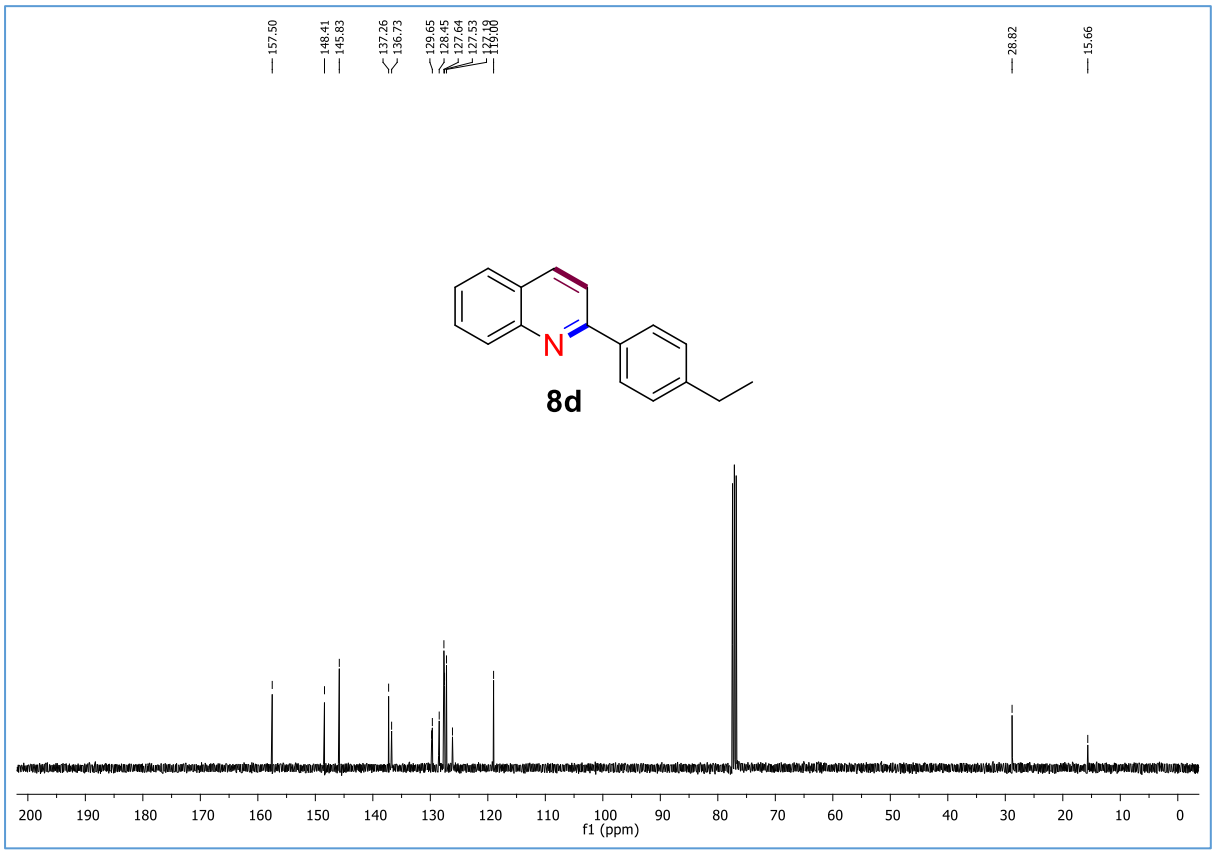
7.80
7.73
7.71
7.69
7.52
7.50
7.49
7.37
7.35
2.76
2.74
2.72
2.70
1.31
1.29
1.27
N
8d
1.96
1.85
1.00
0.92
0.94
0.99
1.90
2.00
2.86
10.0 9.5 9.0 8.5 8.0 7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 0.5 0.0
f1 (ppm)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127.64
127.53
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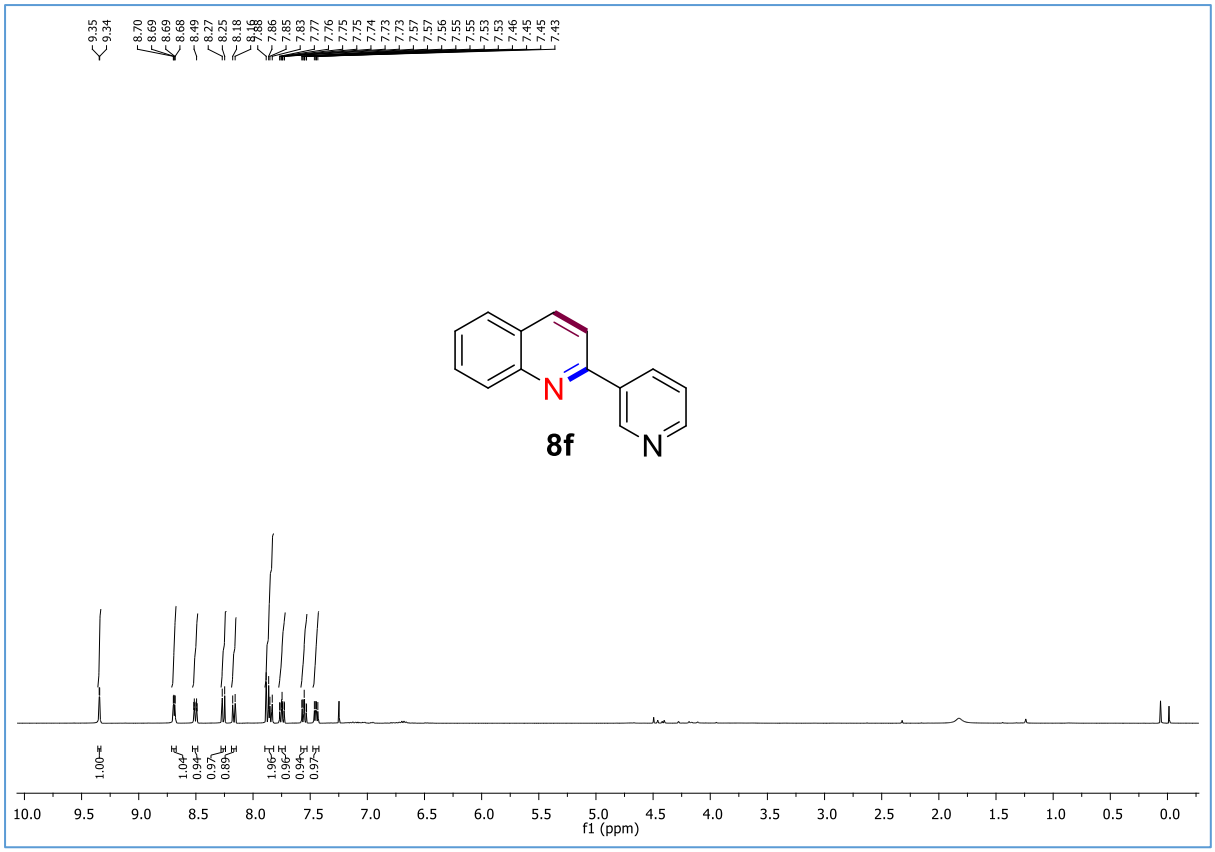
119.08
28.82
15.66
N
8d
200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0
f1 (ppm)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8.69
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8.27
8.25
8.18
7.86
7.85
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7.53
7.53
7.46
7.45
7.45
7.43
N
8f
N
1.00
1.04
0.94
0.97
0.89
1.96
0.96
0.94
0.97
10.0
9.5
9.0
8.5
8.0
7.5
7.0
6.5
6.0
5.5
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4.5
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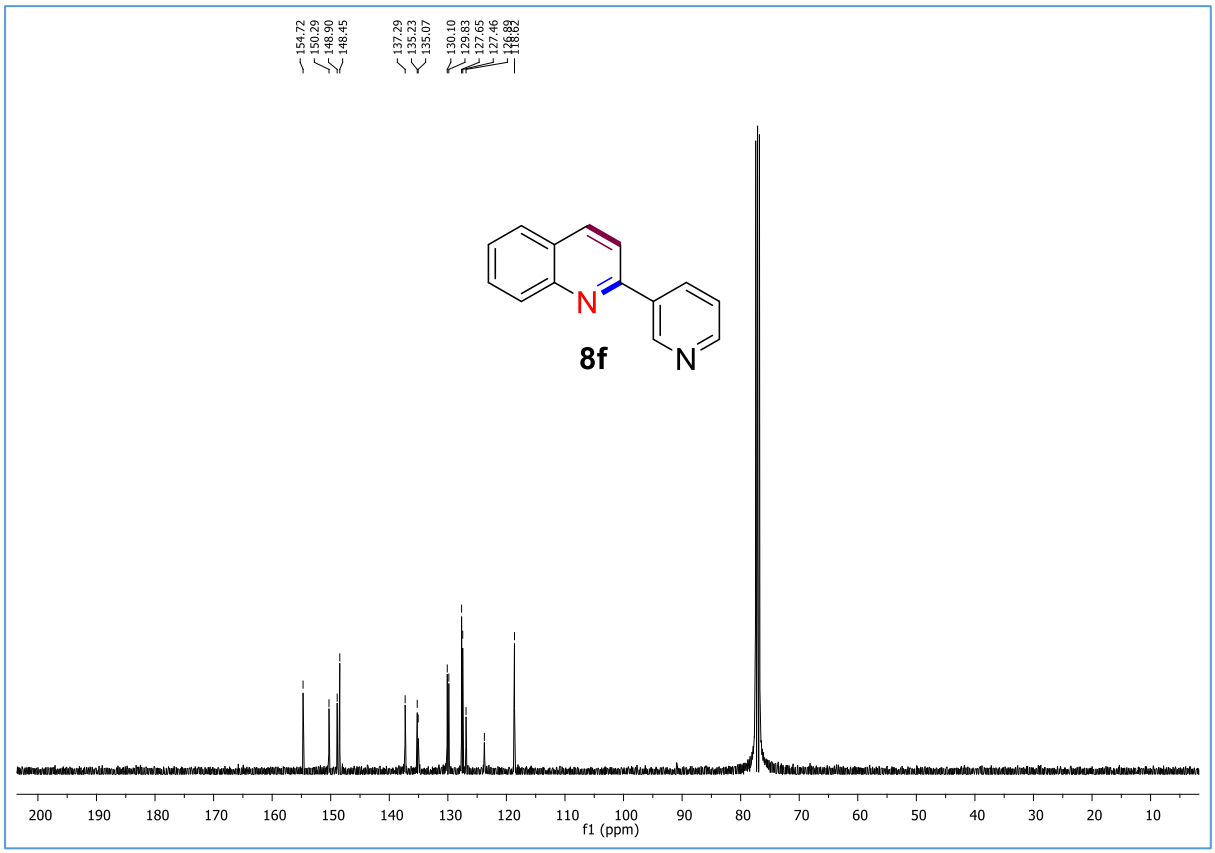
3.5
3.0
2.5
2.0
1.5
1.0
0.5
0.0
f1 (ppm)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135.07
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129.83
127.65
127.46
126.89
126.82
N
8f
N
200
190
180
170
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150
140
130
120
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100
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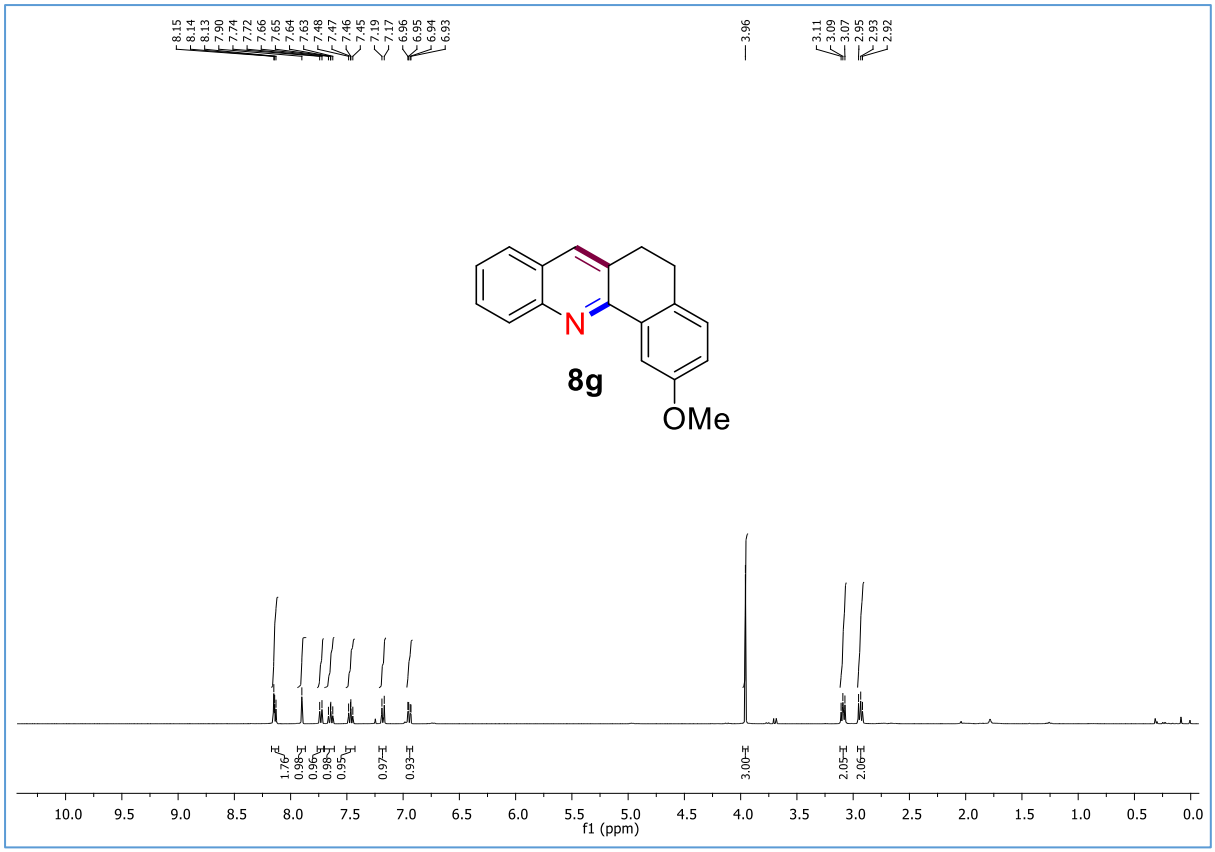
30
20
10
f1 (ppm)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7.48
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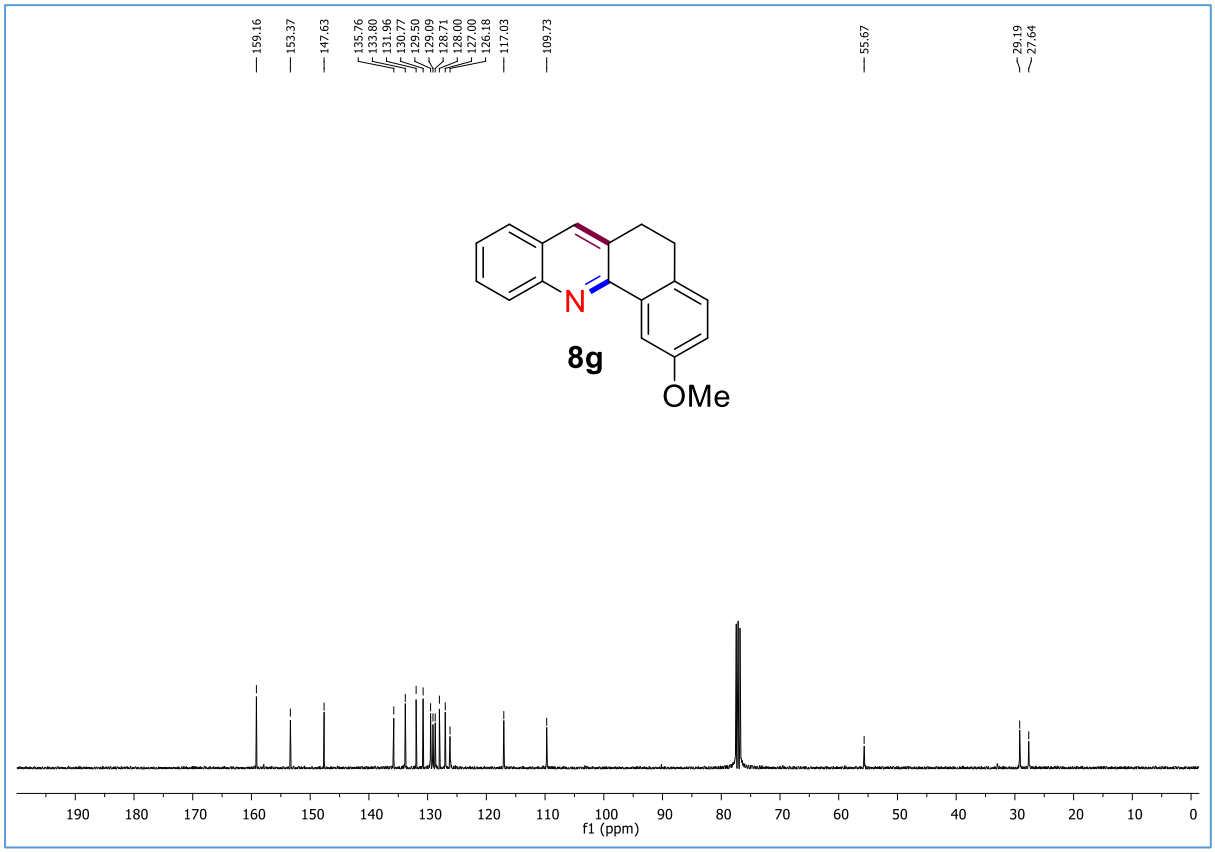
7.46
7.45
7.19
7.17
6.96
6.95
6.94
6.93
3.96
3.11
3.09
3.07
2.95
2.93
2.92
N
8g
OMe
1.76
0.98
0.96
0.98
0.95
0.97
0.93
3.00
2.05
2.06
10.0 9.5 9.0 8.5 8.0 7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 0.5 0.0
f1 (ppm)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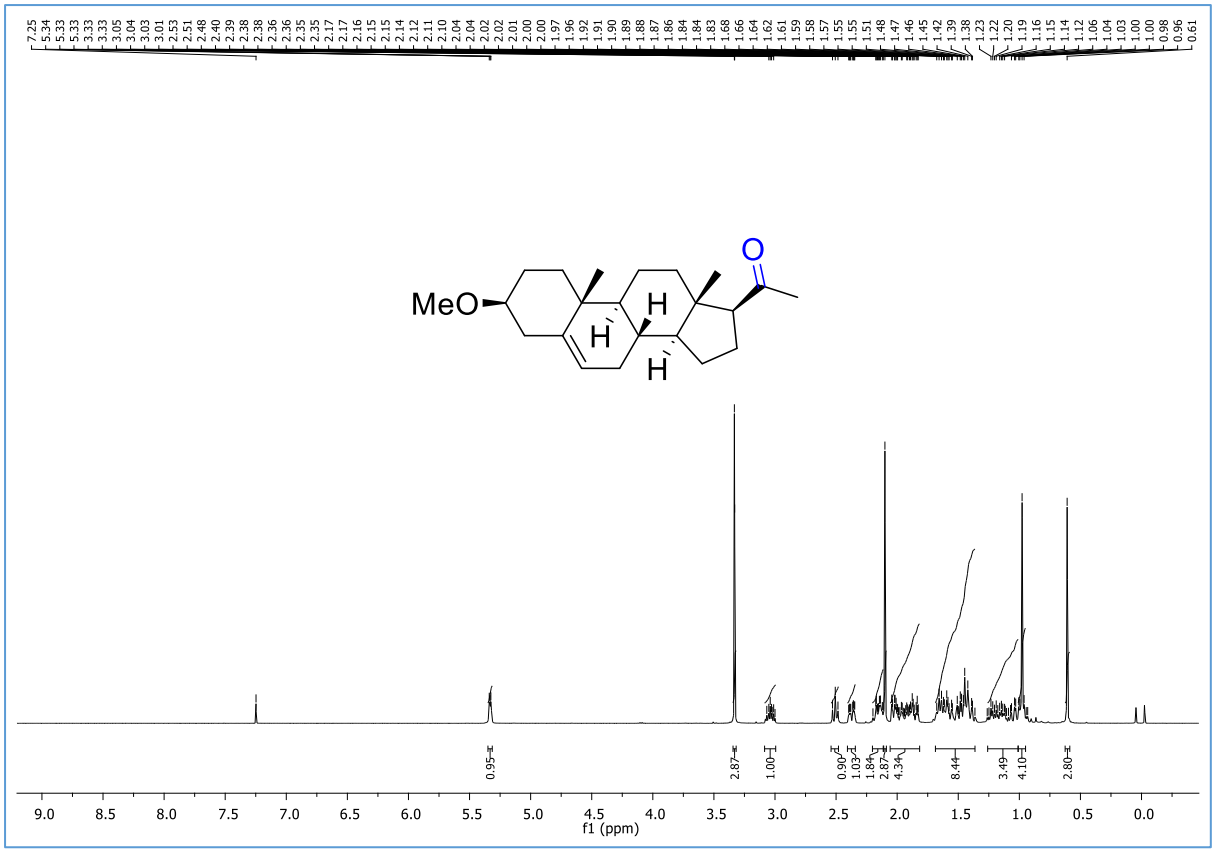
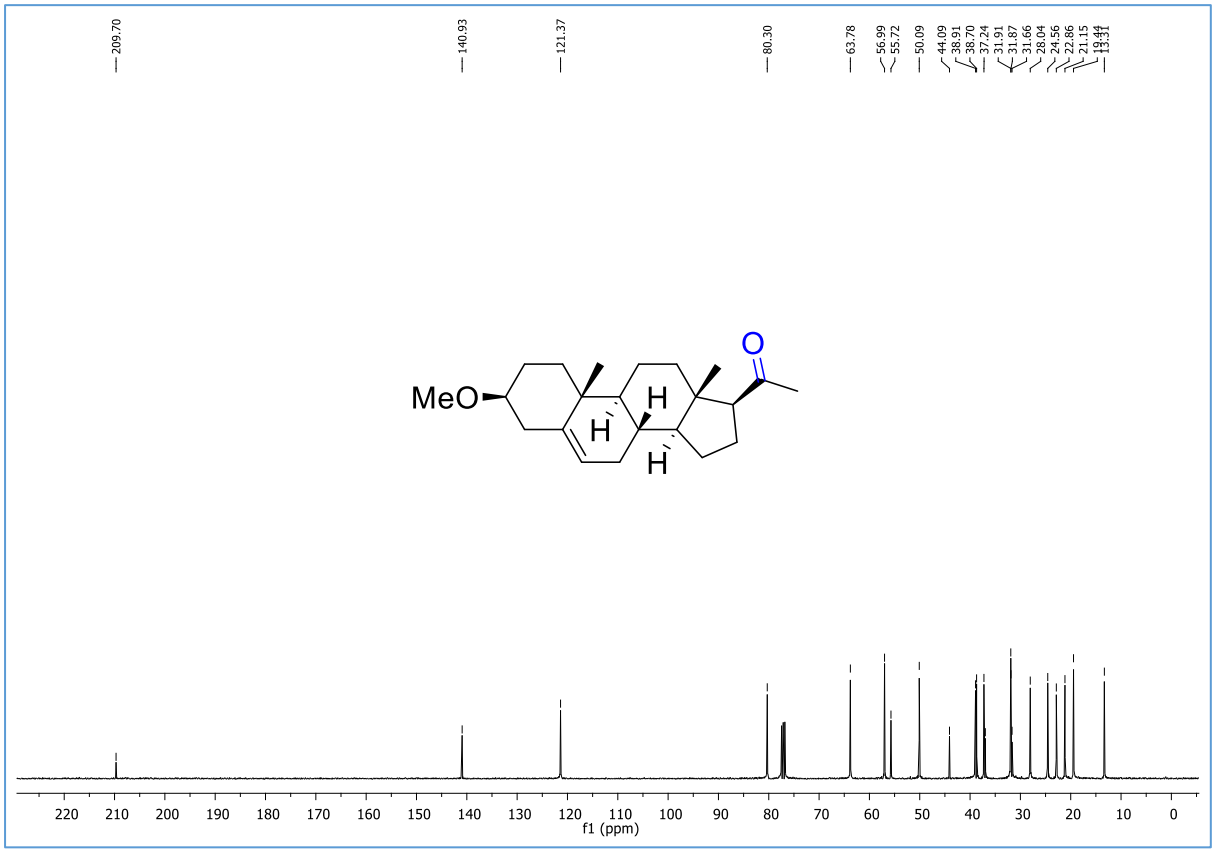
131.96
130.77
129.50
129.09
128.71
128.00
127.00
126.18
117.03
109.73
55.67
29.19
27.64
N
8g
OMe
190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0
f1 (ppm)


MeO
O
H
H
H
f1 (ppm)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56.99
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50.09
44.09
38.91
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37.24
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31.87
31.66
28.04
24.56
22.86
21.15
19.44
13.31
MeO
O
H
H
H
f1 (ppm)