Iron-Catalysed Alkylation of 2-Methyl and 4-Methylazaarenes with Alcohols via C-H Bond Activation

Lalit Mohan Kabadwal, Sourajit Bera, and Debasis Banerjee*

Department of Chemistry
Laboratory of Catalysis and Organic Synthesis
Indian Institute of Technology Roorkee, Roorkee-247667, India
E-mail: debasis.banerjee@cy.iitr.ac.in

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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 MHz (JEOL), 500 MHz (Bruker) and ^{13}C NMR were recorded at 100 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. Elemental analysis data were recorded in Vario Micro Cube. GC-MS were recorded using Agilent GC Mass Spectrometer. High-resolution mass spectra (HRMS) were obtained on a Bruker micro TOF-Q II mass spectrometer (ESI-MS). All the reactions were performed in a closed system using Schlenk tube or pressure tube under inert atmosphere. $Fe(OAc)_2$ was purchased from TCI Chemicals (India) Pvt. Ltd (Purity >90%, CAS No: 3094-87-9, Product Number : I0765). 1,10-Phenanthroline was purchased from Sigma-Aldrich ((Assay- >99%; CAS Number- 66-71-7; EC Number 200-629-2; Pack Size- 131377-25G). Potassium *tert*-butoxide was purchased from Avra Synthesis Pvt. Ltd., India. (Purity-98%, CAS No: 865-47-4, Catalog No- ASP2012).

[1.2] General procedure for Fe-catalyzed alkylation of methylheteroarenes with primary alcohols:

Procedure A: In a 25 mL oven dried ace pressure tube, **1** (0.25 mmol), $Fe(OAc)_2$ (5 mol%), Phen (10 mol%), alcohols (0.50 mmol) and *t*-BuOK (0.25 mmol), were added followed by 1,4-dioxane 1.0 mL under an atmosphere of N_2 and the reaction mixture was heated at 140 °C for 24 h in a closed system. 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.

Procedure B: In a 25 mL oven dried ace pressure tube, **1** (0.25 mmol), $Fe(OAc)_2$ (10 mol%), Phen (30 mol%), alcohols (0.75 mmol) and *t*-BuOK (0.375 mmol), were added followed by 1,4-dioxane 1.0 mL under an atmosphere of N_2 and the reaction mixture was heated at 140 °C for 36 h in closed system. 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.

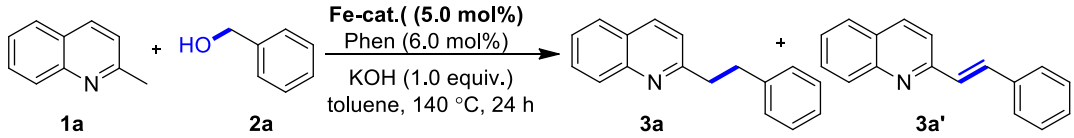
Procedure C: In a 25 mL oven dried ace pressure tube, **1** (0.25 mmol), $Fe(OAc)_2$ (10 mol%), Phen (30 mol%), alcohols (0.50 mmol) and *t*-BuOK (0.375 mmol), were added followed by 1,4-dioxane 1.0 mL under an atmosphere of N_2 and the reaction mixture was heated at 140 °C for 36 h in closed system. 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.

Procedure D: In a 25 mL oven dried ace pressure tube, **1** (0.25 mmol), $Fe(OAc)_2$ (10 mol%), Phen (30 mol%), alcohols (0.75 mmol) and *t*-BuOK (0.50 mmol), were added followed by 1,4-dioxane 1.0 mL under an atmosphere of N_2 and the reaction mixture was heated at 150 °C for 36 h in closed system. 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.

Procedure E: In a 25 mL oven dried ace pressure tube, **1** (0.25 mmol), $Fe(OAc)_2$ (10 mol%), Phen (20 mol%), alcohols (0.75 mmol) and *t*-BuOK (0.50 mmol), were added followed by 1,4-dioxane 1.0 mL under an atmosphere of N_2 and the reaction mixture was heated at 140 °C for 36 h in closed system. 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.

[1.3] Alkylation of methylquinoline with alcohols:

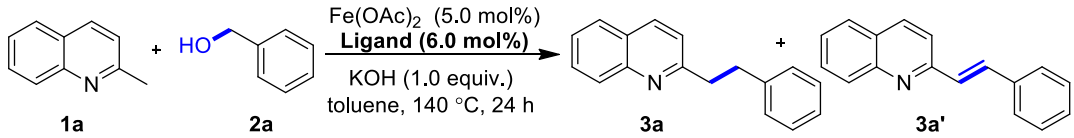
Table S1: Screening of catalyst[a]

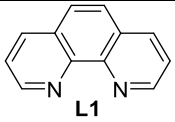
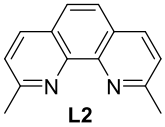
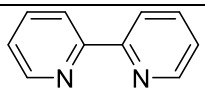
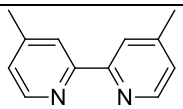
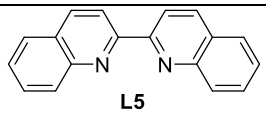


Entry	**Fe-Catalyst**	GC-MS Conversion, **3a** (%)	GC-MS Conversion, **3a'** (%)
1	**$Fe(OAc)_2$**	**71**	**29**
2	$Fe(acac)_3$	6	0
3	$Fe_2(CO)_9$[b]	16	35
4	$FeCl_2$	10	70
5	-	-	-

Reaction conditions: [a] 2-methyl quinoline **1a** (0.25 mmol), Benzyl alcohol **2a** (0.50 mmol), **Fe-Cat. (5.0 mol%)**, Phen (6.0 mol%), KOH (0.25 mmol), toluene (1.0 mL), ace pressure tube under N_2 atmosphere, 140 ºC oil bath, 24 h reaction time. [b] $Fe_2(CO)_9$ (2.5 mol%), Phen (3.0 mol%) were used. Conversions were calculated based on **1a.**

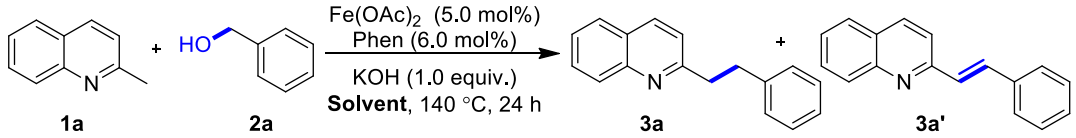
Table S2: Screening of ligands[a]



Entry	**Ligand**	GC-MS Conversion **3a** (%)	GC-MS Conversion **3a'** (%)
1	 **L1**	**71**	**29**
2	 **L2**	-	-
3	 **L3**	10	56
4	 **L4**	-	-
5	 **L5**	40	60
6	No Ligand	-	-

Reaction conditions: [a] 2-methyl quinoline **1a** (0.25 mmol), Benzyl alcohol **2a** (0.50 mmol), $Fe(OAc)_2$ (5.0 mol%), **Ligand (6.0 mol%)**, KOH (0.25 mmol), toluene (1.0 mL), ace pressure tube under N_2 atmosphere, 140 ºC oil bath, 24 h reaction time. Conversions were calculated based on **1a.**

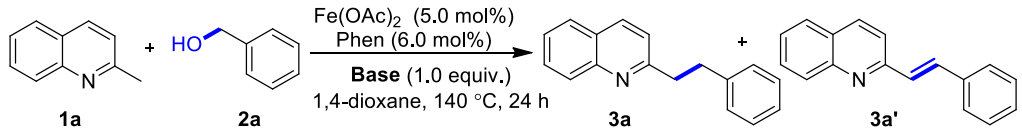
Table S3: Screening of solvents[a]



Entry	**Solvent**	GC-MS Conversion **3a** (%)	GC-MS Conversion **3a'** (%)
1	Toluene	71	29
2	*p*-Xylene	5	-
3	**1,4-Dioxane**	**75**	**25**
4	DMA	1	-
5	*t*-Amylalcohol	16	44

Reaction conditions: [a] 2-methyl quinoline **1a** (0.25 mmol), Benzyl alcohol **2a** (0.50 mmol), $Fe(OAc)_2$ (5.0 mol%), Phen (6.0 mol%), KOH (0.25 mmol), **solvent (1.0 mL)**, ace pressure tube under N_2 atmosphere, 140 ºC oil bath, 24 h reaction time. Conversions were calculated based on **1a.**

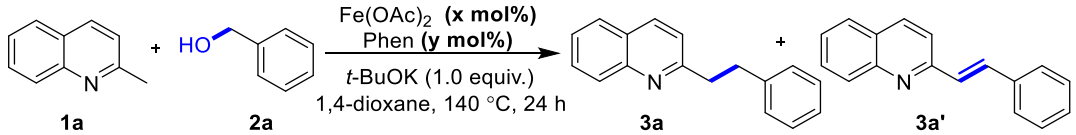
Table S4: Screening of base[a]



Entry	**Base**	GC-MS Conversion **3a** (%)	GC-MS Conversion **3a'** (%)
1	***t*-BuOK**	**76**	**14**
2	*t*-BuONa	47	11
3	K_2CO_3	1	-
4	NaOH	11	41
5	KOH	75	25
6	-	0	0

Reaction conditions: [a] 2-methyl quinoline **1a** (0.25 mmol), Benzyl alcohol **2a** (0.50 mmol), $Fe(OAc)_2$ (5.0 mol%), Phen (6.0 mol%), **Base (0.25 mmol)**, 1,4-dioxane (1.0 mL), ace pressure tube under N_2 atmosphere, 140 ºC oil bath, 24 h reaction time. Conversions were calculated based on **1a.**

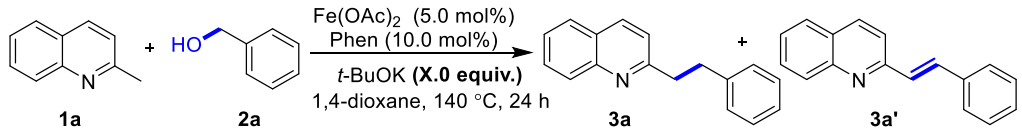
Table S5: Screening of ligand loading[a]



Entry	**$Fe(OAc)_2$ (x mol%)**	**Phen (y mol%)**	GC-MS Conversion **3a** (%)	GC-MS Conversion **3a'** (%)
1	5	6	76	14
2	**5**	**10**	**93 (91)**[b]	**3**
3[c]	5	10	70	10
4	5	15	90	3
5	5	20	91	3
6	5	30	93 (91)[b]	3
7	10	12	91	3
8	10	20	94	3
9	3	6	51	17

Reaction conditions: [a] 2-methyl quinoline **1a** (0.25 mmol), Benzyl alcohol **2a** (0.50 mmol), $Fe(OAc)_2$ (5.0 mol%), **Phen (x mol%)**, *t*-BuOK (0.25 mmol), 1,4-dioxane (1.0 mL), ace pressure tube under N_2 atmosphere, 140 ºC oil bath, 24 h reaction time. [b]Isolated yield. [c]reaction temp. 130 ºC. Conversions were calculated based on **1a.**

Table S6: Screening of base equivalence[a]

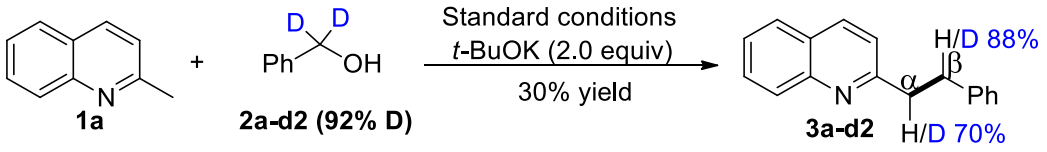


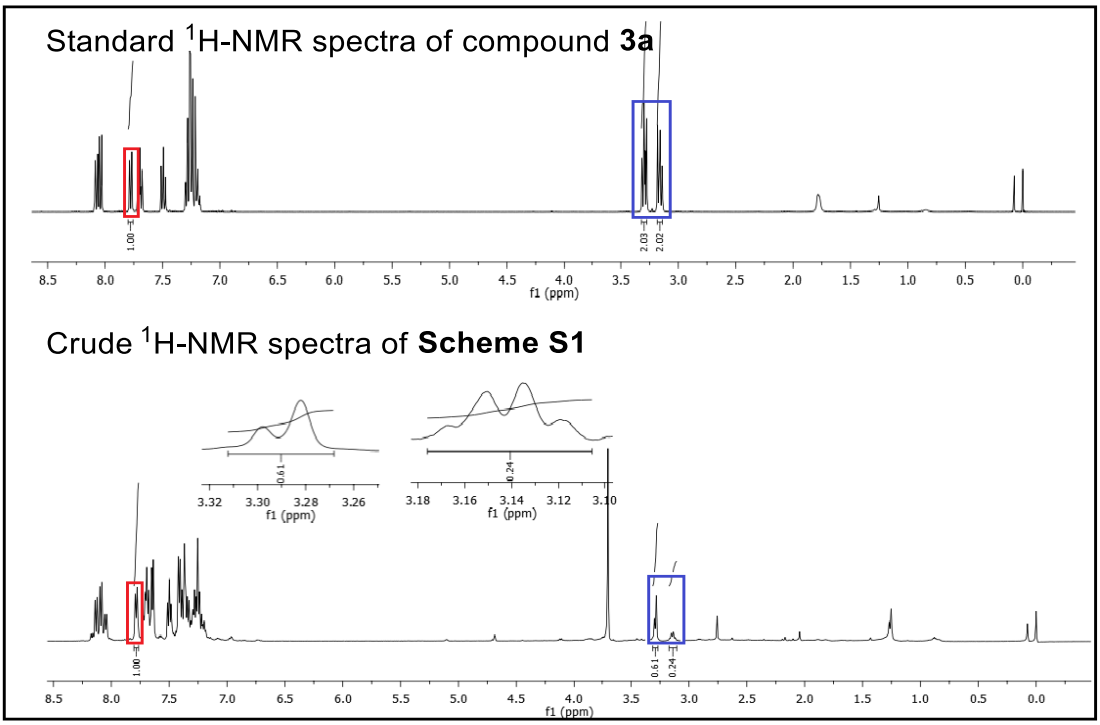
Entry	*t*-BuOK **(X.0 equiv.)**	GC-MS Conversion **3a** (%)	GC-MS Conversion **3a'** (%)
1	0.5	57	23
2	0.75	72	15
3	1.0	93	3

Reaction conditions: [a] 2-methyl quinoline **1a** (0.25 mmol), Benzyl alcohol **2a** (0.50 mmol), $Fe(OAc)_2$ (5.0 mol%), Phen (10.0 mol%), *t*-BuOK **(X equiv.)**, 1,4-dioxane (1.0 mL), ace pressure tube under N_2 atmosphere, 140 ºC oil bath, 24 h reaction time. Conversions were calculated based on **1a.**

Deuterium incorporation studies:

Scheme S1:

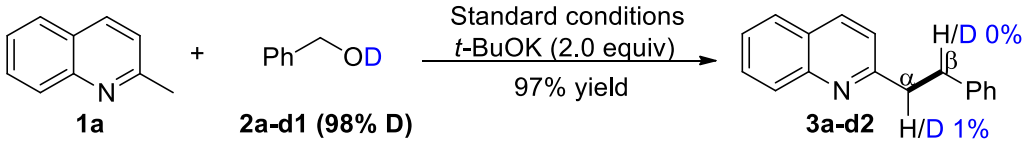


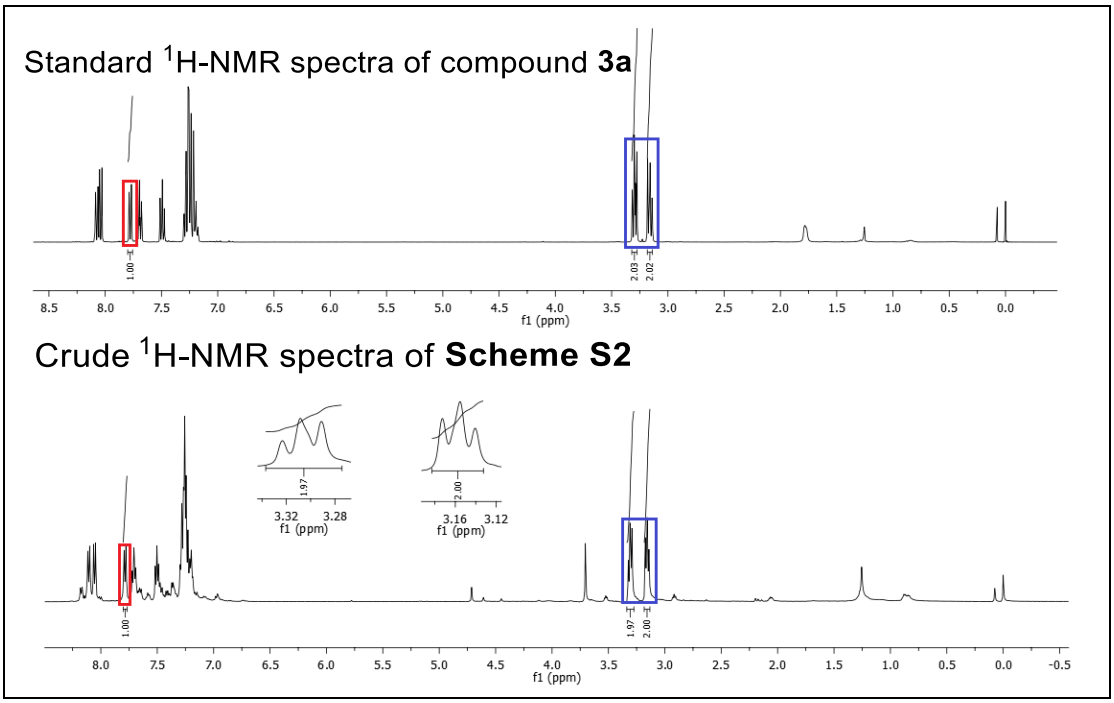


Conversion was calculated by ^{1}H-NMR integration value

		Deuterium incorporation in α position	Deuterium incorporation in β position
Signal δ ppm	7.78 (1H)	3.29 (2H)	3.14 (2H)
Integral Value	1.0	0.61	0.24
Calculated ratio		{(2-0.61)/2}×100 = **70%**	{(2-0.24)/2}×100 = **88%**

Scheme S2:

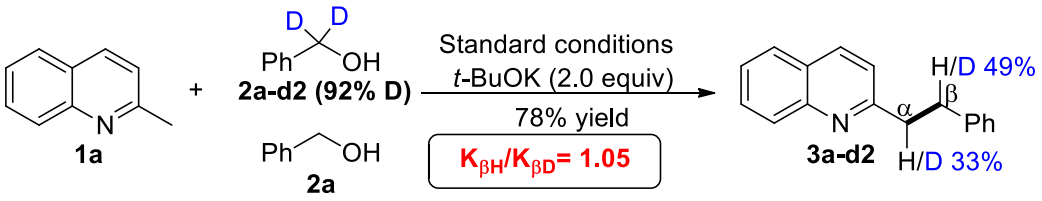


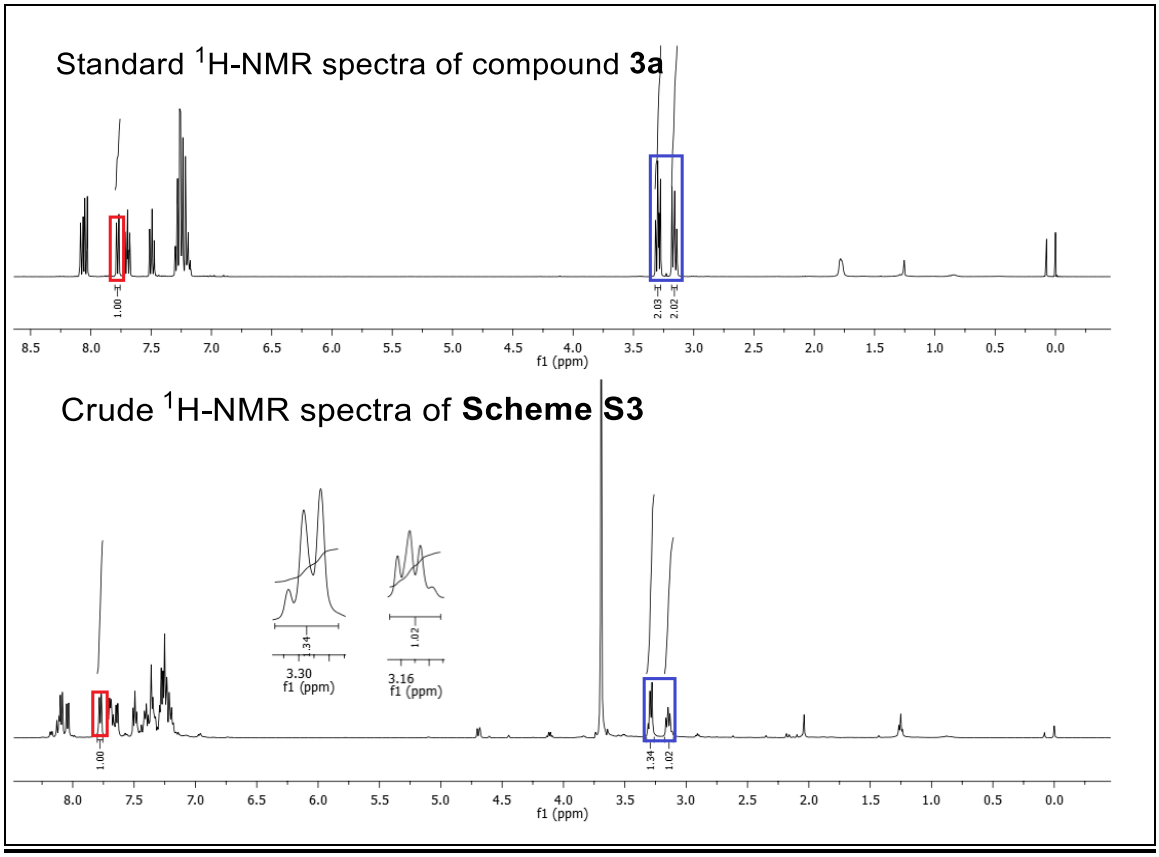


Conversion was calculated by ^{1}H-NMR integration value

		Deuterium incorporation in **α** position	Deuterium incorporation in **β** position
Signal δ ppm	7.78 (1H)	3.29 (2H)	3.14 (2H)
Integral Value	1.0	1.97	2.00
Calculated ratio		{(2-1.97)/2}×100 = **1%**	{(2-2.00)/2}×100 = **0%**

Scheme S3:

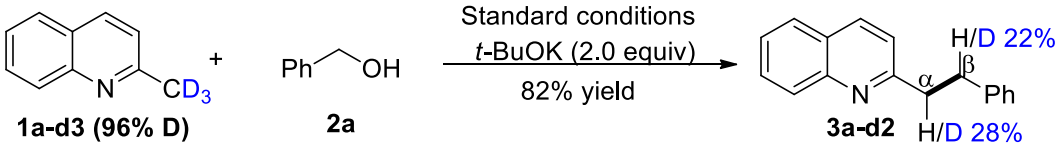


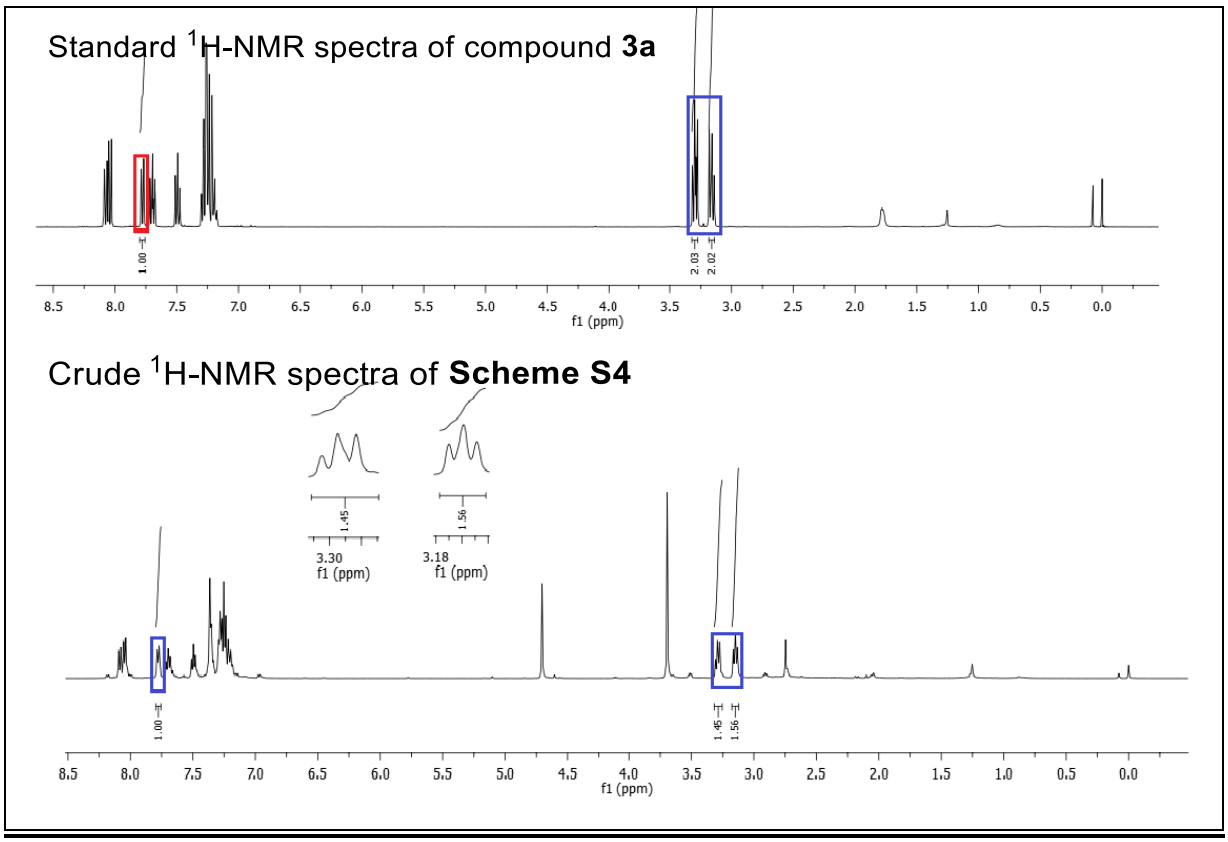


Conversion was calculated by ^{1}H-NMR integration value

		Deuterium incorporation in **α** position	Deuterium incorporation in **β** position
Signal δ ppm	7.78 (1H)	3.29 (2H)	3.14 (2H)
Integral Value	1.0	1.34	1.02
Calculated ratio		{(2-1.34)/2}×100 = **33%**	{(2-1.02)/2}×100 = **49%**

Scheme S4:

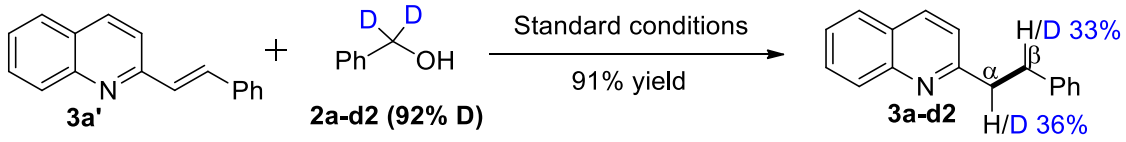


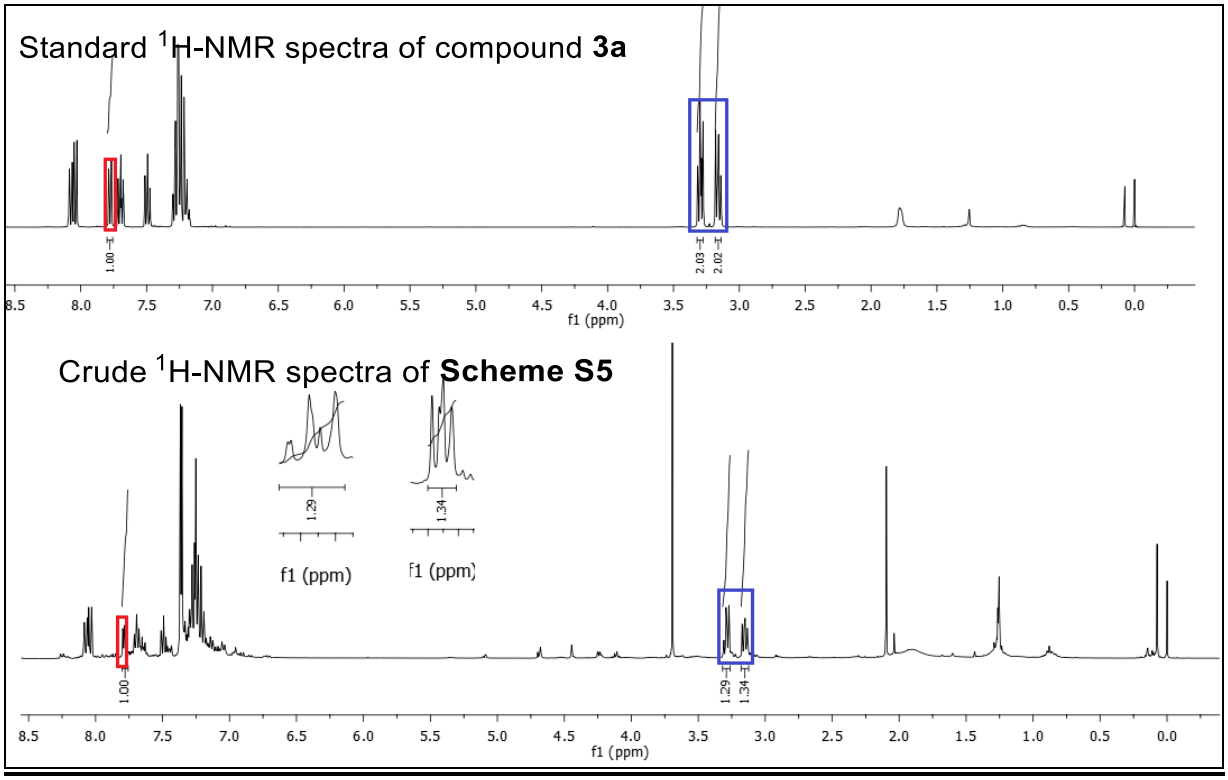


Conversion was calculated by ^{1}H-NMR integration value

		Deuterium incorporation in **α** position	Deuterium incorporation in **β** position
Signal δ ppm	7.78 (1H)	3.29 (2H)	3.14 (2H)
Integral Value	1.0	1.45	1.56
Calculated ratio		{(2-1.45)/2}×100 = **28%**	{(2-1.56)/2}×100 = **22%**

Scheme S5:



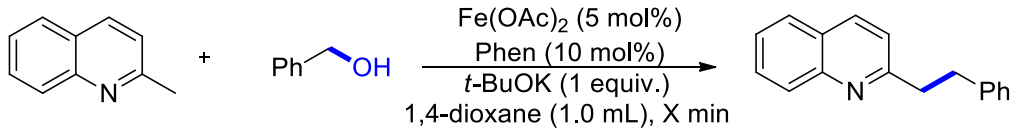


Conversion was calculated by ^{1}H-NMR integration value

		Deuterium incorporation in **α** position	Deuterium incorporation in **β** position
Signal δ ppm	7.78 (1H)	3.29 (2H)	3.14 (2H)
Integral Value	1.0	1.29	1.34
Calculated ratio		{(2-1.29)/2}×100 = **36%**	{(2-1.34)/2}×100 = **33%**

Scheme S6: Determination of rate and order of reaction

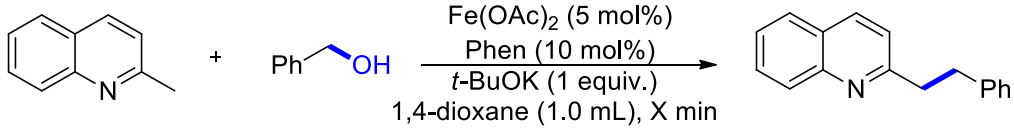
Run 1: Reaction was carried out in 1 mL of 1,4-dioxane and yield was calculated by GC



No.	**1a** (mmol)	**2a** (mmol)	$Fe(OAc)_2$ (mmol)	Phen (mmol)	*t*-BuOK (mmol)	dioxane (mL)
Run 1	0.2	0.40	0.01	0.02	0.2	1.0

Sl. No.	Time (min)	Concentration of **1a** (mM)
1	60	175
2	120	165
3	180	148
4	240	130
5	300	116
6	360	102
7	420	80
8	480	61
9	540	45

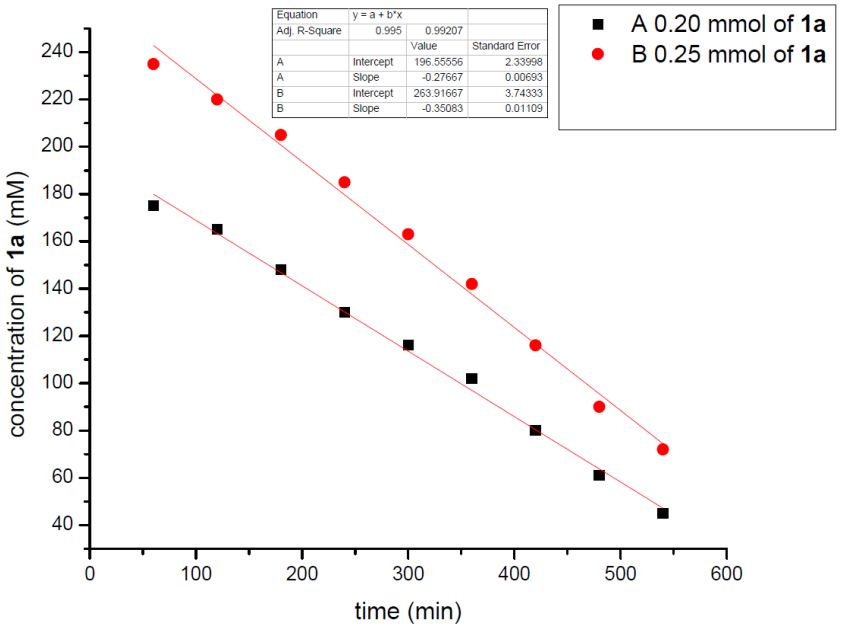
Run 2: Reaction was carried out in 1 mL of 1,4-dioxane and yield was calculated by GC



No.	**1a** (mmol)	**2a** (mmol)	$Fe(OAc)_2$ (mmol)	Phen (mmol)	*t*-BuOK (mmol)	dioxane (mL)
Run 2	0.25	0.50	0.0125	0.0250	0.25	1.0

Sl. No.	Time (min)	Concentration of **1a** (mM)
1	60	235
2	120	220
3	180	205
4	240	185
5	300	163
6	360	142
7	420	116
8	480	90
9	540	72

Graphical representation for determination of rate and order of reaction



Considering steady state approximation for benzyl alcohol

From Run 1: Slope = k [1a] ^{x}

$-0.276 = k\,[0.20]^{x}$

From Run 2: Slope = k [1a] ^{x}

$$-0.350 = k\,[0.25]^{x}$$

$$-0.350 \,/\!-\, 0.276 = [0.25]^{x} / [0.2]^{x}$$

$$1.26 = [1.25]^{x}$$

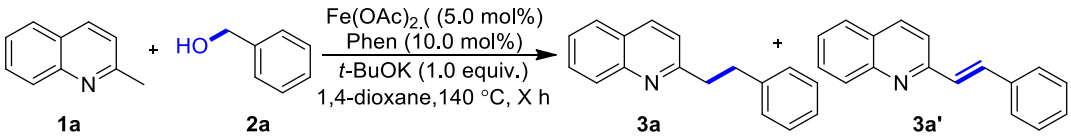
$$\text{Log}\,(1.26) = x.\ \text{Log}\,(1.25)$$

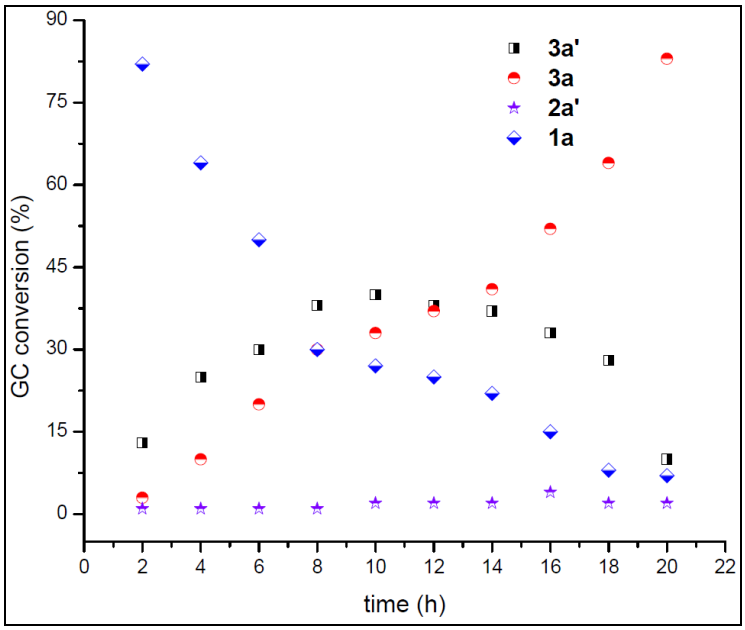
$$x = 0.100 / 0.0969$$

$$= 1.03 \approx 1$$

$$\text{Rate} = k\,[1a]^{1}$$

Scheme S7: Time-conversion-plot for the reaction profile:

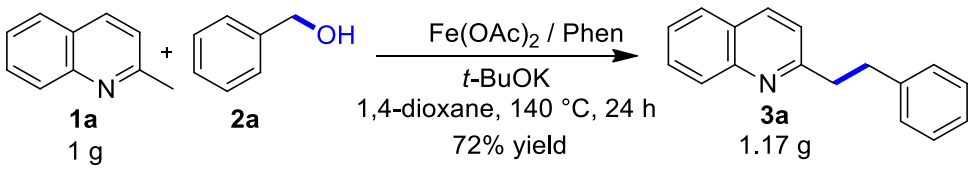




Time-conversion-plot for the reaction of quinaldine (**1a**) with benzyl alcohol (**2a**).

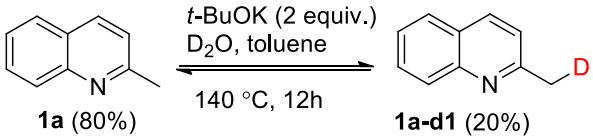
Reaction conditions: [a] 2-methyl quinoline **1a** (0.20 mmol), Benzyl alcohol **2a** (0.40 mmol), $Fe(OAc)_2$ (5.0 mol%), Phen (10 mol%), *t*-BuOK (0.20 mmol), 1,4-dioxane (1.0 mL), ace pressure tube under nitrogen atmosphere, 140 ºC oil bath, X h reaction time.

Scheme S8: Gram scale reaction: Practical utility

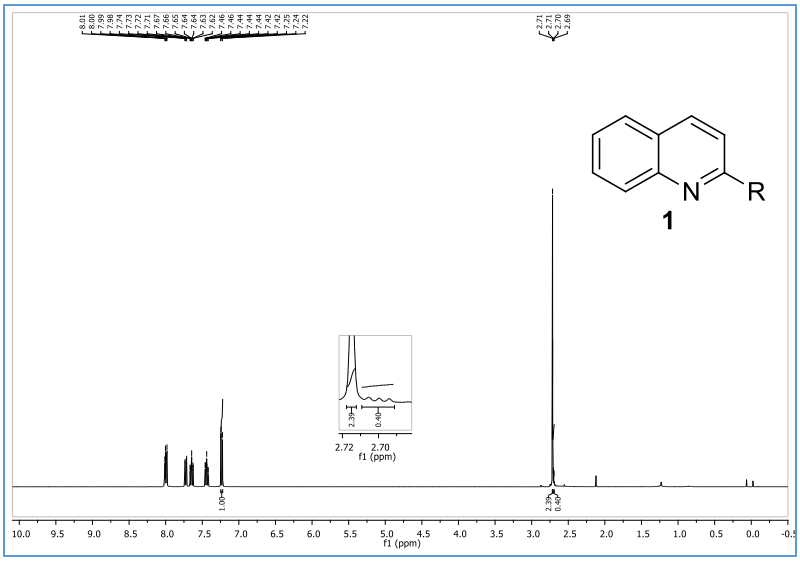


In a 100 mL oven dried ace pressure tube, 2-methyl quinoline **1a** (6.99 mmol, 1.0 g), benzyl alcohol **2a** (13.98 mmol, 1.51 g), $Fe(OAc)_2$ (5 mol%, 61 mg), Phen (10 mol%, 126 mg) and *t*-BuOK (6.99 mmol, 784 mg), were added followed by 1,4-dioxane (7.0 mL) under an atmosphere of N_2 and the reaction mixture was heated at 140 °C for 24 h in a closed system. The reaction mixture was cooled to room temperature and 12.0 mL of ethyl acetate was added and concentrated *in vacuo*. The residue was purified by column chromatography using ethyl acetate/hexane (1:99) (eluent system) to afford the desired product **3a** in 72% yield (1.17 g).

Scheme S9 : Evidence for the enamine intermediate



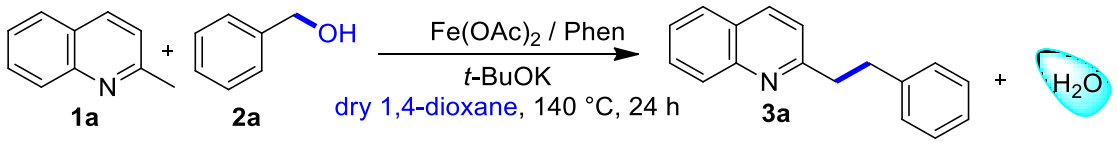
Reaction conditions: Quinaldine **1a** (0.25 mmol), **D_2O** (0.2 mL), *t*-BuOK (0.5 mmol), toluene (1.0 mL), Ace Pressure tube under nitrogen atmosphere, 140 °C oil bath, 12 h.



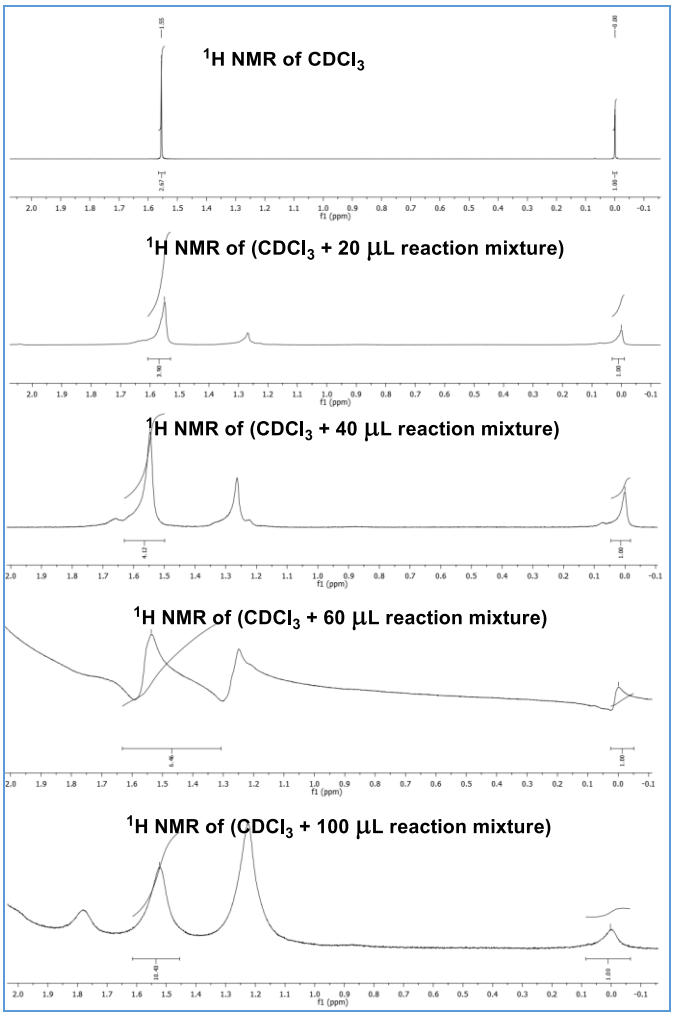
Conversion was calculated by ^{1}H-NMR integration value

		1a	1a-d1
Signal δ ppm	7.23 (1H)	2.71 (3H)	2.70 (2H)
Integral Value	1.0	2.39	0.40
Calculated ratio		(2.39 / 3)×100 = **80%**	(0.40 / 2)×100 = **20%**

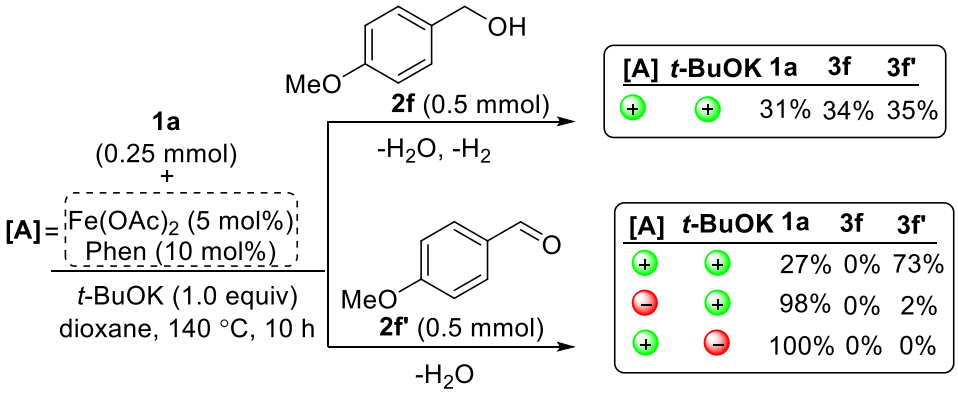
Scheme S10: Detection of water in the reaction mixture by ^{1}H-NMR



In a 15 mL oven dried Schlenk tube, quinaldine **1a** (0.25 mmol), $Fe(OAc)_2$ (5 mol%), Phen (10 mol%), benzyl alcohol **2a** (0.50 mmol) and *t*-BuOK (0.25 mmol), were added followed by dry 1,4-dioxane 1.0 mL under an atmosphere of N_2 and the reaction mixture was heated at 140 °C for 24 h in a closed system. Then the reaction mixture was cooled to room temperature. Initially ^{1}H NMR of $CDCl_3$ was measured and 2.67:1 ratio of H_2O and TMS was found. Afterwards 20 µL of reaction mixture was added to the nmr tube and ^{1}H NMR was measured which shows increment in the ratio of H_2O. Further addition of reaction mixture shows enhancement in the ratio of H_2O which proves that water was produced in the reaction.



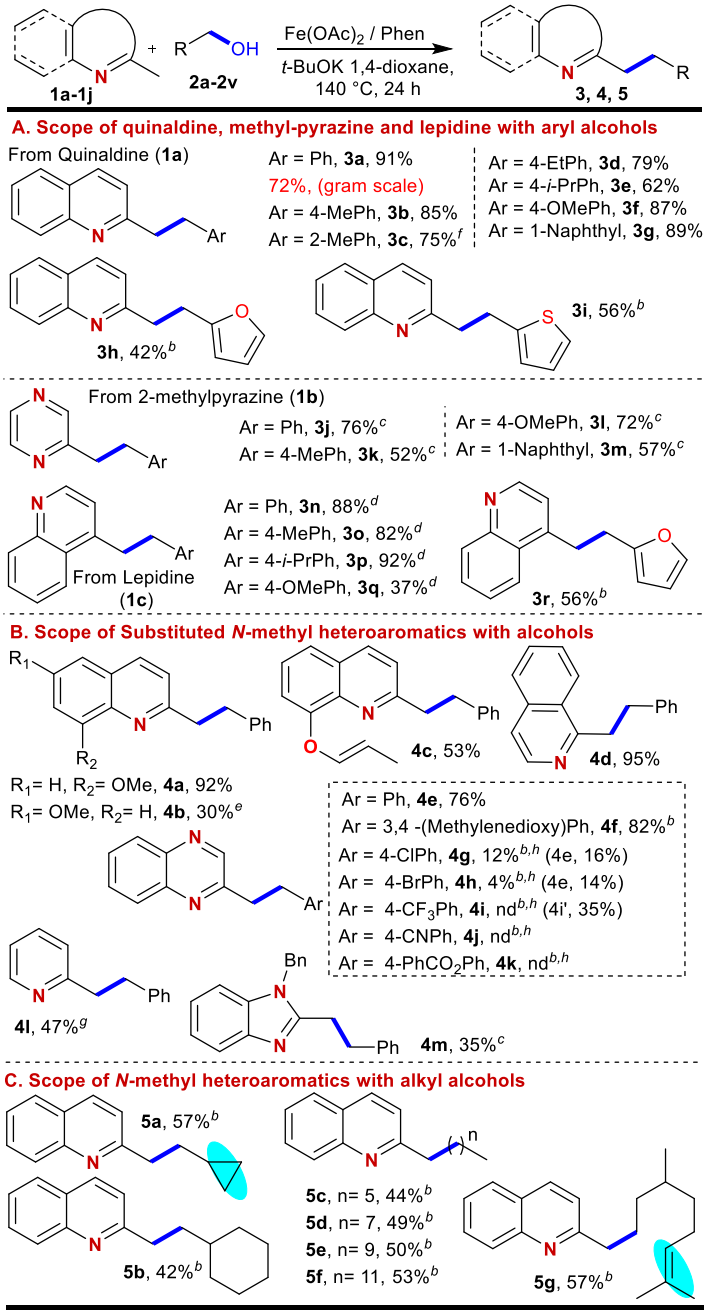
Scheme S11: Control experiment



Nevertheless, a series of control experiments were performed using **1a** with 4-methoxybenzaldehyde **2f'** and 4-methoxybenzylalcohol **2f** in presence and absence of iron-

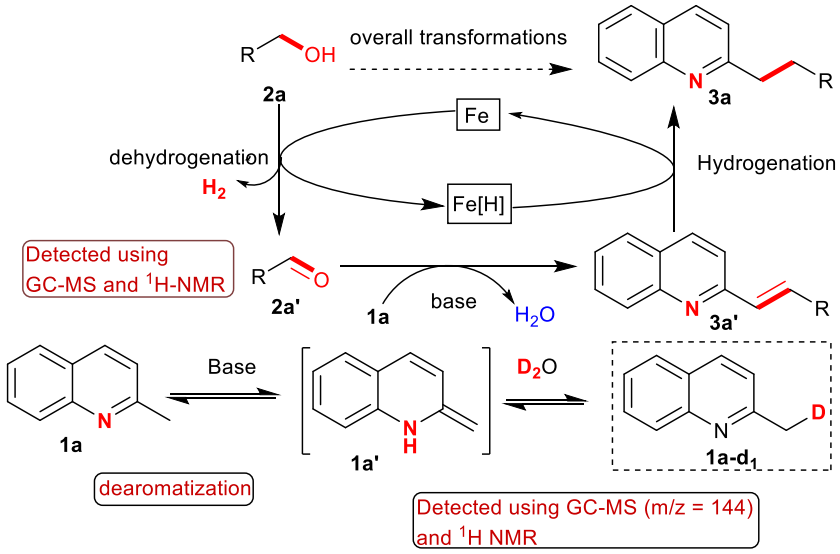
catalyst. The catalytic conditions presented as **A**. These experiments revealed the significant role of the Fe-catalyst for the alkylation process. The presence of a base and catalysts represent in green color, whereas the absence of these reagents represented in red color.

Scheme S12: Synthesis of chain-elongated heteroarenes[a]



Reaction Conditions: [a]1a (0.25 mmol), primary alcohol 2a (0.50 mmol), $Fe(OAc)_2$ (0.0125 mmol), phen (0.025 mmol), *t*-BuOK (0.25 mmol) using 1,4-dioxane (1.0 mL) in pressure tube under N_2 atmosphere at 140 °C in a pre-heated oil bath for 24 h. [b]2 (0.75 mmol), $Fe(OAc)_2$ (0.025 mmol), phen (0.075 mmol), *t*-BuOK (0.375 mmol), 36 h. [c]36 h. [d]*t*-BuOK (0.25 mmol), 24 h. [e]*t*-BuOK (0.375 mmol), 36 h. [f] 2 (0.75 mmol), $Fe(OAc)_2$ (0.025 mmol), phen (0.075 mmol), *t*-BuOK (0.375 mmol), 36 h, 150 °C. [g]2-methylpyridine *N*-oxide is used (0.25 mmol), Fe(OAc)2 (0.025 mmol), phen (0.075 mmol), *t*-BuOK (0.50 mmol), 36 h. [h]GC-MS conversion based on 1h.

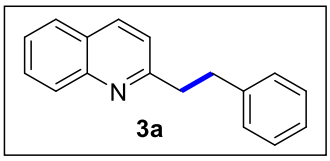
Scheme S13: Mechanistic rationale for Fe-catalysed alkylation of methyl heteroarenes



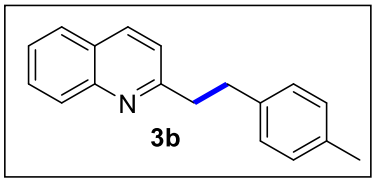
On the basis of our initial findings and literature report, we anticipated that, initially, alcohol **2a** undergoes dehydrogenation to aldehyde **2a'** catalyzed by iron and transient Fe-H species is formed. Afterward, aldehyde coupled with enamine **1a'** to the intermediate **3a'**. Successive hydrogenation of **3a'** by transient Fe-H species gave alkylated product **3a** with the elimination of water. Notably, chemo-selective alkylation was achieved when 1,4-dioxane was used as solvent and facilitate the hydrogenation of **3a'** by Fe-H species. Nevertheless, Meerwein-Ponndorf mechanism for such process is another possibility.

[1.4] Spectroscopic and analytical data:

2-Phenethylquinoline (3a)[1]**:** Following the general procedure A the title compound was isolated as a light brown oil eluting with 1% ethyl acetate in hexane (Yield 91%, 53 mg). ^{1}H NMR (400 MHz, $CDCl_3$) δ 8.08-8.02 (m, 2H), 7.77 (d, J = 8.1 Hz, 1H), 7.72-7.67 (m, 1H), 7.51-7.47 (m, 1H), 7.30-7.17 (m, 6H), 3.32-3.27 (m, 2H), 3.18-3.14 (m, 2H); ^{13}C{^{1}H} NMR (100 MHz, $CDCl_3$) δ 161.8, 148.0, 141.5, 136.2, 129.4, 128.9, 128.5, 128.4, 127.5, 126.8, 126.0, 125.8, 121.5, 41.0, 35.9.

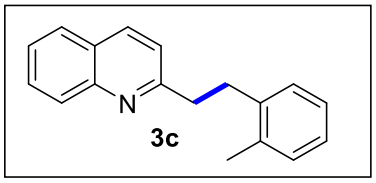


2-(4-Methylphenethyl)quinoline (3b)[1]**:** Following the general procedure A the title compound was isolated as a light brown oil eluting with 1% ethyl acetate in hexane (Yield 85%, 52 mg). ^{1}H NMR (400 MHz, $CDCl_3$) δ 8.06 (dd, J = 12.3, 8.5 Hz, 2H), 7.78 (dd, J = 8.1, 1.1 Hz, 1H), 7.72-7.67 (m, 1H), 7.51-7.47 (m, 1H), 7.25-7.22 (m, 1H), 7.15 (d, J =

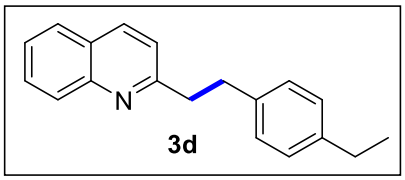


8.0 Hz, 2H), 7.09 (d, *J* = 8.1 Hz, 2H), 3.27 (dd, *J* = 10.0, 6.2 Hz, 2H), 3.11 (dd, *J* = 9.8, 6.4 Hz, 2H), 2.32 (s, 3H); $^{13}C\{^{1}H\}$ NMR (100 MHz, $CDCl_3$) δ 161.9, 147.9, 138.4, 136.2, 135.4, 129.4, 129.1, 128.8, 128.3, 127.9, 127.5, 126.8, 121.6, 41.1, 35.5, 21.0.

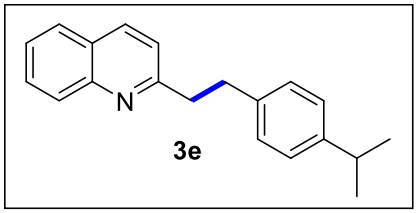
2-(2-methylphenethyl)quinoline (3c)[1]: Following the general procedure D the title compound was isolated as a light brown oil eluting with 1% ethyl acetate in hexane (Yield 75%, 46 mg). ^{1}H NMR (400 MHz, $CDCl_3$) δ 1H NMR (400 MHz, $CDCl_3$) δ 8.01-7.94 (m, 2H), 7.69 (dd, *J* = 8.4, 1.7 Hz, 1H), 7.61 (ddd, *J* = 8.6, 7.0, 1.7 Hz, 1H), 7.41 (ddd, *J* = 8.3, 7.0, 1.3 Hz, 1H), 7.16-7.09 (m, 2H), 7.08-7.02 (m, 3H), 3.17 (dd, *J* = 10.9, 6.3 Hz, 2H), 3.05 (dd, *J* = 11.0, 6.4 Hz, 2H), 2.26 (s, 3H). $^{13}C\{^{1}H\}$ NMR (100 MHz, $CDCl_3$) δ 162.1, 148.1, 139.8, 136.3, 136.1, 130.3, 129.5, 129, 129, 127.6, 126.9, 126.3, 126.1, 125.9, 121.6, 39.8, 33.4, 19.4.



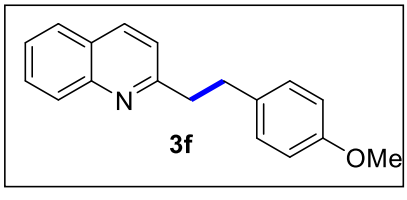
2-(4-Ethylphenethyl)quinolone (3d)[1]: Following the general procedure A the title compound was isolated as a light brown oil eluting with 1% ethyl acetate in hexane (Yield 79%, 51 mg). ^{1}H NMR (500 MHz, $CDCl_3$) δ 8.09 (t, *J* = 8.7 Hz, 2H), 7.81 (d, *J* = 8.0 Hz, 1H), 7.73 (t, *J* = 7.6 Hz, 1H), 7.53 (t, *J* = 7.5 Hz, 1H), 7.28 (t, *J* = 4.1 Hz, 2H), 7.21 (d, *J* = 7.7 Hz, 2H), 7.15 (d, *J* = 7.7 Hz, 2H), 3.31 (dd, *J* = 9.7, 6.6 Hz, 2H), 3.18-3.13 (m, 2H), 2.65 (d, *J* = 7.6 Hz, 2H), 1.29-1.25 (m, 3H); $^{13}C\{^{1}H\}$ NMR (100 MHz, $CDCl_3$) δ 161.9, 147.9, 141.9, 138.7, 136.2, 129.4, 128.8, 128.4, 127.9, 127.5, 126.8, 125.7, 121.5, 41.1, 35.5, 28.4, 15.6.



2-(4-Isopropylphenethyl)quinolone (3e)[1]: Following the general procedure A the title compound was isolated as a light brown oil eluting with 1% ethyl acetate in hexane (Yield 62%, 43 mg). ^{1}H NMR (400 MHz, $CDCl_3$) δ 8.06 (dd, *J* = 15.3, 8.4 Hz, 2H), 7.77 (dd, *J* = 8.1, 1.1 Hz, 1H), 7.71-7.67 (m, 1H), 7.50-7.46 (m, 1H), 7.25 (d, *J* = 8.4 Hz, 1H), 7.17 (td, *J* = 8.2, 6.1 Hz, 4H), 3.31-3.24 (m, 2H), 3.12 (dd, *J* = 10.0, 6.3 Hz, 2H), 2.93-2.82 (m, 1H), 1.24 (d, *J* = 6.9 Hz, 6H); $^{13}C\{^{1}H\}$ NMR (100 MHz, $CDCl_3$) δ 162.0, 147.9, 146.5, 138.8, 136.2, 129.3, 128.8, 128.4, 127.5, 126.7, 126.4, 125.8, 121.5, 41.0, 35.5, 33.7, 24.0.

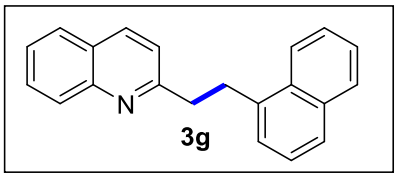


2-(4-Methoxyphenethyl)quinolone (3f)[1]: Following the general procedure A the title compound was isolated as a light brown oil eluting with 3% ethyl acetate

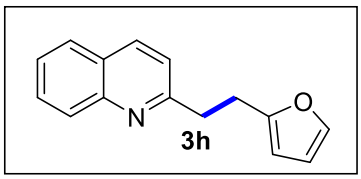


in hexane (Yield 87%, 57 mg). ^{1}H NMR (400 MHz, $CDCl_3$) δ 8.05 (dd, J = 14.2, 8.5 Hz, 2H), 7.77 (d, J = 8.1 Hz, 1H), 7.70-7.68 (m, 1H), 7.50 (dd, J = 11.1, 4.1 Hz, 1H), 7.22 (d, J = 8.4 Hz, 1H), 7.16 (d, J = 8.5 Hz, 2H), 6.85-6.80 (m, 2H), 3.78 (s, 3H), 3.26 (dd, J = 9.6, 6.2 Hz, 2H), 3.10 (dd, J = 9.6, 6.5 Hz, 2H); ^{13}C{^{1}H} NMR (100 MHz, $CDCl_3$) δ 161.9, 157.8, 147.9, 136.2, 133.5, 129.4, 129.4, 128.8, 127.5, 126.7, 125.7, 121.6, 113.7, 55.2, 41.3, 35.1.

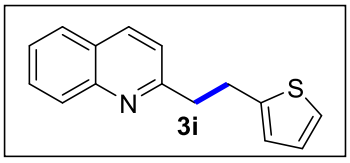
2-(2-(Naphthalen-2-yl)ethyl)quinoline (3g)[1]: Following the general procedure A the title compound was isolated as a light brown oil eluting with 1% ethyl acetate in hexane (Yield 89%, 63 mg). ^{1}H NMR (400 MHz, $CDCl_3$) δ 8.18 (d, J = 8.3 Hz, 1H), 8.12 (d, J = 8.5 Hz, 1H), 7.99 (d, J = 8.4 Hz, 1H), 7.89-7.83 (m, 1H), 7.78-7.67 (m, 3H), 7.54-7.45 (m, 3H), 7.39-7.30 (m, 2H), 7.17 (d, J = 8.4 Hz, 1H), 3.62 (dd, J = 9.6, 6.5 Hz, 2H), 3.42 (dd, J = 9.6, 6.5 Hz, 2H); ^{13}C{^{1}H} NMR (100 MHz, $CDCl_3$) δ 161.9, 148.0, 137.5, 136.2, 133.8, 131.8, 129.4, 128.8, 128.8, 127.5, 126.8, 126.8, 126.1, 125.9, 125.8, 125.5, 125.5, 123.7, 121.5, 40.0, 32.9.



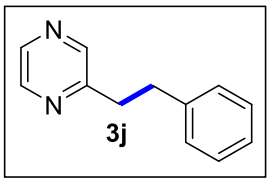
2-(2-(Furan-2-yl)ethyl)quinolone (3h)[2]: Following the general procedure B the title compound was isolated as a light brown oil eluting with 1% ethyl acetate in hexane (Yield 42%, 23 mg). ^{1}H NMR (400 MHz, $CDCl_3$) δ 8.05 (dd, J = 8.4, 3.3 Hz, 2H), 7.81-7.76 (m, 1H), 7.71-7.67 (m, 1H), 7.49 (dd, J = 11.2, 4.3 Hz, 1H), 7.32 (d, J = 1.6 Hz, 1H), 7.25 (d, J = 3.1 Hz, 1H), 6.28-6.24 (m, 1H), 5.99 (d, J = 3.0 Hz, 1H), 3.35-3.30 (m, 2H), 3.21-3.15 (m, 2H); ^{13}C{^{1}H} NMR (100 MHz, $CDCl_3$) δ 161.2, 155.1, 148.0, 141.0, 136.3, 129.4, 128.9, 127.5, 126.8, 125.8, 121.3, 110.1, 105.4, 37.4, 27.9.



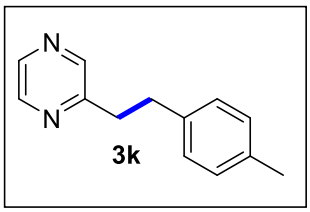
2-(2-(Thiophen-2-yl)ethyl)quinoline (3i)[3]: Following the general procedure B the title compound was isolated as a light brown oil eluting with 1% ethyl acetate in hexane (Yield 56%, 33mg). ^{1}H NMR (400 MHz, $CDCl_3$) δ 8.06 (dd, J = 8.3, 1.9 Hz, 2H), 7.78 (dd, J = 8.1, 1.3 Hz, 1H), 7.72-7.68 (m, 1H), 7.52-7.50 (m, 1H), 7.26 (t, J = 4.2 Hz, 1H), 7.11 (dd, J = 5.1, 1.2 Hz, 1H), 6.90 (dd, J = 5.1, 3.4 Hz, 1H), 6.84 -6.79 (m, 1H), 3.43-3.38 (m, 2H), 3.37-3.32 (m, 2H); ^{13}C{^{1}H} NMR (100 MHz, $CDCl_3$) δ 167.2, 161.1, 148.0, 144.2, 136.3, 129.4, 128.9, 127.5, 126.8, 126.7, 125.9, 124.6, 123.2, 121.5, 41.0, 29.7.



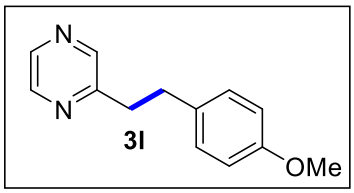
2-Phenethylpyrazine (3j)[1]: Following the general procedure A the title compound was isolated as a light brown oil eluting with 4% ethyl acetate in hexane (Yield 76%, 35 mg). ^{1}H NMR (400 MHz, $CDCl_3$) δ 8.51 (dd, J = 2.5, 1.6 Hz, 1H), 8.39 (d, J = 2.6 Hz, 1H), 8.35 (d, J = 1.5 Hz, 1H), 7.30-7.24 (m, 3H), 7.22-7.15 (m, 2H), 3.14-3.04 (m, 4H). ^{13}C{^{1}H} NMR (100 MHz, $CDCl_3$) δ 156.8, 144.8, 144.8, 144.3, 144.2, 142.5, 142.4, 140.8, 128.5, 126.4, 37.3, 35.5.



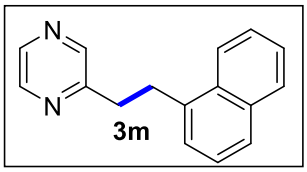
2-(4-Methylphenethyl)pyrazine (3k): Following the general procedure A the title compound was isolated as a light brown oil eluting with 4% ethyl acetate in hexane (Yield 52%, 26 mg). ^{1}H NMR (500 MHz, $CDCl_3$) δ 8.52 (d, J = 1.6 Hz, 1H), 8.41 (d, J = 2.1 Hz, 1H), 8.36 (s, 1H), 7.08 (d, J = 6.3 Hz, 4H), 3.11 (t, J = 8.0 Hz, 2H), 3.06-3.00 (m, 2H), 2.32 (s, 4H); ^{13}C{^{1}H} NMR (100 MHz, $CDCl_3$) δ 156.9, 144.7, 144.1, 142.3, 137.7, 135.7, 129.2, 128.3, 37.4, 35.0, 21.0. HRMS (ESI-TOF) m/z: $[M+H]^+$ Calcd for $C_{13}H_{14}N_2$ 199.1230; Found 199.1296.



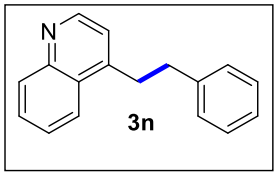
2-(4-Methoxyphenethyl)pyrazine (3l)[1]: Following the general procedure A the title compound was isolated as a light brown oil eluting with 5% ethyl acetate in hexane (Yield 72%, 39 mg). ^{1}H NMR (500 MHz, $CDCl_3$) δ 8.55-8.52 (m, 1H), 8.42 (d, J = 2.4 Hz, 1H), 8.37 (d, J = 1.0 Hz, 1H), 7.10 (d, J = 8.5 Hz, 2H), 6.84 (d, J = 8.6 Hz, 2H), 3.80 (s, 3H), 3.11 (dd, J = 8.7, 6.0 Hz, 2H), 3.04 (dd, J = 8.7, 6.0 Hz, 2H); ^{13}C{^{1}H} NMR (100 MHz, $CDCl_3$) δ 158.1, 156.9, 144.8, 144.2, 142.4, 132.8, 129.4, 114.0, 55.3, 37.6, 34.7.



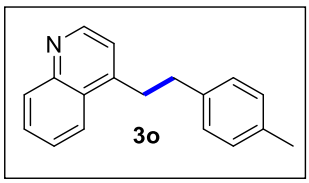
2-(2-(Naphthalen-1-yl)ethyl)pyrazine (3m): Following the general procedure A the title compound was isolated as a light brown oil eluting with 4% ethyl acetate in hexane (Yield 57%, 33 mg). ^{1}H NMR (500 MHz, $CDCl_3$) δ 8.56 (s, 1H), 8.41 (s, 1H), 8.33 (s, 1H), 8.12 (dd, J = 16.5, 8.3 Hz, 1H), 7.87 (d, J = 7.8 Hz, 1H), 7.74 (d, J = 8.1 Hz, 1H), 7.52 (dd, J = 15.7, 7.5 Hz, 2H), 7.37 (t, J = 7.5 Hz, 1H), 7.29-7.24 (m, 1H), 3.53 (t, J = 7.8 Hz, 2H), 3.25 (t, J = 7.9 Hz, 2H); ^{13}C{^{1}H} NMR (100 MHz, $CDCl_3$) δ 156.9, 144.6, 144.2, 142.4, 136.8, 133.9, 131.6, 128.9, 127.1, 126.1, 126.0, 125.6, 125.5, 123.5, 36.4, 32.6. HRMS (ESI-TOF) m/z: $[M+H]^+$ Calcd for $C_{16}H_{14}N_2$ 235.1230; Found 235.1306.



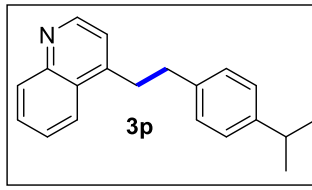
4-Phenethylquinoline (3n)[3]**:** Following the general procedure A the title compound was isolated as a colorless oil eluting with 7% ethyl acetate in hexane (Yield 88%, 51 mg). ^{1}H NMR (400 MHz, $CDCl_3$) δ 8.81 (d, J = 4.4 Hz, 1H), 8.16 (d, J = 8.9 Hz, 1H), 8.09 (d, J = 7.8 Hz, 1H), 7.74 (t, J = 7.0 Hz, 1H), 7.59 (t, J = 7.6 Hz, 1H), 7.33 (t, J = 7.2 Hz, 2H), 7.27-7.19 (m, 4H), 3.44-3.37 (m, 2H), 3.13-3.06 (m, 2H).^{13}C{^{1}H} NMR (100 MHz, $CDCl_3$) δ 150.3, 148.4, 147.5, 141.0, 130.4, 129.2, 128.6, 128.5, 127.5, 126.5, 126.4, 123.5, 121.0, 77.4, 77.1, 76.8, 36.2, 34.1.



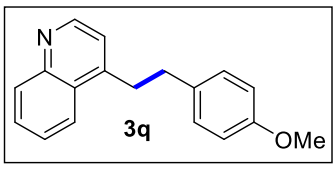
4-(4-Methylphenethyl)quinoline (3o): Following the general procedure A the title compound was isolated as a yellow solid eluting with 7% ethyl acetate in hexane (Yield 85%, 55 mg). ^{1}H NMR (400 MHz, $CDCl_3$) δ 8.78 (d, J = 4.5 Hz, 1H), 8.12 (d, J = 8.4 Hz, 1H), 8.07 (d, J = 8.0 Hz, 1H), 7.74-7.67 (m, 1H), 7.58-7.57 (m, 1H), 7.17 (d, J = 4.4 Hz, 1H), 7.13-7.08 (m, 4H), 3.35 (dd, J = 9.4, 6.8 Hz, 2H), 3.03 (dd, J = 9.4, 6.8 Hz, 2H), 2.33 (s, 3H). ^{13}C{^{1}H} NMR (100 MHz, $CDCl_3$) δ 150.3, 148.4, 147.6, 138.0, 135.9, 130.4, 129.3, 129.1, 128.3, 127.5, 126.5, 123.5, 121.0, 35.8, 34.3, 21.1. HRMS (ESI-TOF) m/z: $[M+H]^+$ Calcd for $C_{18}H_{17}N$ 248.1434; Found 248.1435.



4-(4-Isopropylphenethyl)quinoline (3p): Following the general procedure A the title compound was isolated as a yellow oil eluting with 7% ethyl acetate in hexane (Yield 92%, 64 mg). ^{1}H NMR (400 MHz, $CDCl_3$) δ 8.79 (d, J = 4.4 Hz, 1H), 8.12 (d, J = 8.1 Hz, 1H), 8.08-8.03 (m, 1H), 7.73-7.67 (m, 1H), 7.58-7.53 (m, 1H), 7.20 (t, J = 3.9 Hz, 1H), 7.18-7.16 (m, 2H), 7.16-7.13 (m, 2H), 3.37 (dd, J = 9.5, 6.8 Hz, 2H), 3.03 (dd, J = 9.6, 6.8 Hz, 2H), 2.89-2.86 (m, 1H), 1.26 (s, 3H), 1.24 (s, 3H). ^{13}C{^{1}H} NMR (100 MHz, $CDCl_3$) δ 150.3, 148.4, 147.7, 147.0, 138.4, 130.4, 129.1, 128.3, 127.6, 126.7, 126.5, 123.5, 120.9, 35.8, 34.2, 33.8, 24.2. HRMS (ESI-TOF) m/z: $[M+H]^+$ Calcd for $C_{20}H_{21}N$ 276.1747; Found 276.1786.

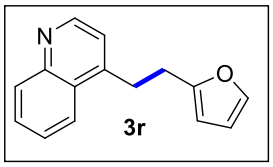


4-(4-Methoxyphenethyl)quinoline (3q): Following the general procedure A the title compound was isolated as a yellow solid eluting with 10% ethyl acetate in hexane (Yield 37%, 24 mg). ^{1}H NMR (400 MHz, $CDCl_3$) δ 8.76 (d, J = 4.6 Hz, 1H), 8.12 (dd, J = 8.9, 1.4 Hz, 1H), 8.05 (dd, J = 8.8, 1.6 Hz, 1H), 7.70-7.68 (m, 1H), 7.56-7.54 (m, 1H), 7.14 (t, J = 4.7 Hz, 1H), 7.12-7.06 (m, 2H), 6.85-6.80 (m, 2H), 3.78 (s, 3H), 3.33 (dd, J = 9.7, 6.9 Hz, 2H), 3.00 (dd,



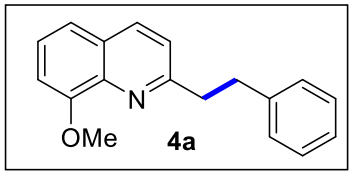
J = 9.6, 7.0 Hz, 2H). $^{13}C\{^{1}H\}$ NMR (100 MHz, $CDCl_3$) δ 158.2, 150.2, 148.4, 147.6, 133.1, 130.4, 129.4, 129.1, 127.5, 126.5, 123.5, 121.0, 114.0, 55.4, 35.4, 34.4. HRMS (ESI-TOF) m/z: $[M+H]^+$ Calcd for $C_{18}H_{17}NO$ 264.1383; Found 264.1387.

4-(2-(Furan-2-yl)ethyl)quinoline (3r): Following the general procedure A the title compound



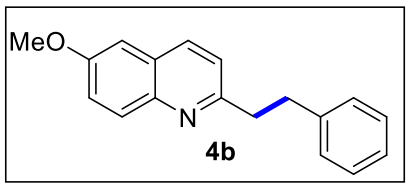
was isolated as a yellow oil eluting with 10% ethyl acetate in hexane (Yield 56%, 30 mg). ^{1}H NMR (400 MHz, $CDCl_3$) δ 8.78 (dd, *J* = 8.5, 4.5 Hz, 1H), 8.16-8.08 (m, 1H), 8.07-7.99 (m, 1H), 7.75-7.66 (m, 1H), 7.58-7.51 (m, 1H), 7.35-7.33 (m, 1H), 7.18 (t, *J* = 5.8 Hz, 1H), 6.27 (dd, *J* = 3.1, 1.9 Hz, 1H), 5.98 (dd, *J* = 3.1, 0.6 Hz, 1H), 3.44-3.36 (m, 2H), 3.11-3.03 (m, 2H). $^{13}C\{^{1}H\}$ NMR (100 MHz, $CDCl_3$) δ 154.3, 150.1, 148.3, 146.9, 141.2, 130.2, 129.1, 127.3, 126.5, 123.3, 120.7, 110.2, 105.7, 30.6, 28.4. HRMS (ESI-TOF) m/z: $[M+H]^+$ Calcd for $C_{18}H_{17}NO$ 224.1070; Found 224.1076.

8-Methoxy-2-phenethylquinoline (4a)[1]: Following the general procedure A the title compound



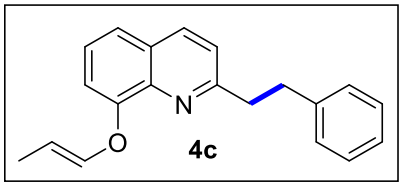
was isolated as a light brown oil eluting with 2% ethyl acetate in hexane (Yield 92%, 60 mg). ^{1}H NMR (400 MHz, $CDCl_3$) δ 8.01 (d, *J* = 8.4 Hz, 1H), 7.43-7.38 (m, 1H), 7.35 (dd, *J* = 8.1, 0.9 Hz, 1H), 7.32-7.23 (m, 5H), 7.21-7.19 (m, 1H), 7.04 (d, *J* = 7.5 Hz, 1H), 4.08 (s, 3H), 3.39-3.33 (m, 2H), 3.15 (dd, *J* = 9.9, 6.6 Hz, 2H); $^{13}C\{^{1}H\}$ NMR (125 MHz, $CDCl_3$) δ 157.7, 151.8, 138.3, 136.6, 132.9, 125.2, 125.1, 124.7, 122.7, 118.7, 116.2, 111.7, 104.5, 52.8, 37.7, 32.8.

6-Methoxy-2-phenethylquinoline (4b)[1]: Following the general procedure A the title compound



was isolated as a light brown oil eluting with 2% ethyl acetate in hexane (Yield 30%, 20 mg). ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.95 (t, *J* = 9.0 Hz, 2H), 7.34 (dd, *J* = 9.1, 2.9 Hz, 1H), 7.29-7.15 (m, 6H), 7.04 (d, *J* = 2.8 Hz, 1H), 3.91 (s, 3H), 3.28-3.20 (m, 2H), 3.12 (dd, *J* = 9.8, 6.0 Hz, 2H); $^{13}C\{^{1}H\}$ NMR (125 MHz, $CDCl_3$) δ 157.6, 155.7, 142.4, 140.0, 133.4, 128.7, 126.9, 126.7, 126.0, 124.3, 120.3, 120.2, 103.6, 53.9, 39.1, 34.4.

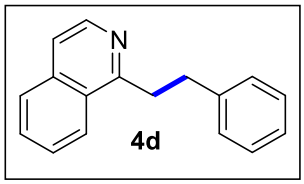
8-(Allyloxy)-2-phenethylquinoline (4c)[1]: Following the general procedure A the title compound



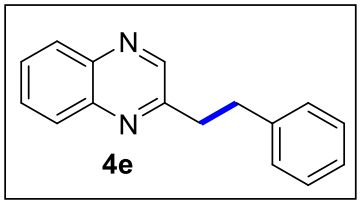
was isolated as a pale yellow oil eluting with 2% ethyl acetate in hexane (Yield 53%, 41 mg). ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.94 (d, *J* = 8.5 Hz, 1H), 7.37-7.34 (m, 1H), 7.29 (dd, *J* = 4.1,

2.2 Hz, 1H), 7.19 (s, 2H), 7.13-7.10 (m, 1H), 7.08-7.04 (m, 2H), 7.01-6.96 (m, 2H), 6.49-6.46 (m, 1H), 5.03-4.95 (m, 1H), 3.30-3.26 (m, 2H), 3.12 (dd, *J* = 9.6, 6.3 Hz, 2H), 1.79-1.75 (m, 3H); $^{13}C\{^{1}H\}$ NMR (100 MHz, $CDCl_3$) δ 159.9, 151.7, 140.4, 140.0, 138.4, 134.7, 128.1, 127.3, 127.0, 126.7, 124.6, 120.0, 111.3, 109.9, 108.2, 39.5, 34.3, 8.5.

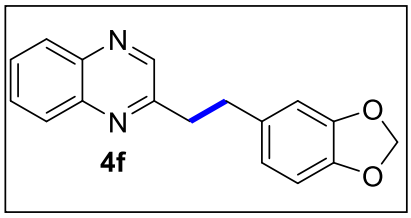
1-Phenethylisoquinoline (4d)[1]: Following the general procedure A the title compound was isolated as a light brown oil eluting with 1% ethyl acetate in hexane (Yield 95%, 55 mg). ^{1}H NMR (400 MHz, $CDCl_3$) δ 8.47 (d, *J* = 5.7 Hz, 1H), 8.14 (d, *J* = 8.5 Hz, 1H), 7.81 (d, *J* = 8.2 Hz, 1H), 7.67-7.63 (m, 1H), 7.59-7.53 (m, 1H), 7.52 (d, *J* = 5.7 Hz, 1H), 7.31 (d, *J* = 4.3 Hz, 4H), 7.24-7.19 (m, 1H), 3.62-3.57 (m, 2H), 3.22-3.17 (m, 2H); $^{13}C\{^{1}H\}$ NMR (125 MHz, $CDCl_3$) δ 161.0, 141.9, 136.2, 129.7, 128.4, 128.4, 127.4, 127.0, 126.9, 126.0, 125.0, 119.3, 37.2, 35.4.



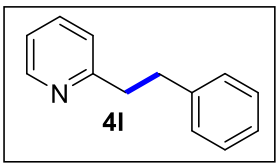
2-Phenethylquinoxaline (4e)[2]: Following the general procedure A the title compound was isolated as a pale yellow oil eluting with 4% ethyl acetate in hexane (Yield 76%, 41 mg). ^{1}H NMR (400 MHz, $CDCl_3$) δ 8.61 (s, 1H), 8.09-8.04 (m, 2H), 7.75-7.72 (m, 2H), 7.29-7.18 (m, 5H), 3.35-3.30 (m, 2H), 3.18 (dd, *J* = 9.4, 6.4 Hz, 2H). $^{13}C\{^{1}H\}$ NMR (100 MHz, $CDCl_3$) δ 156.4, 145.7, 142.2, 141.2, 140.7, 130.0, 129.2, 129.0, 128.9, 128.8, 128.4, 128.1, 38.2, 35.5.



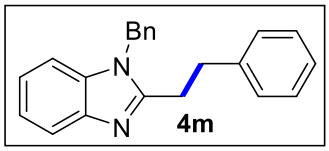
2-Phenethylquinoxaline (4f)[2]: Following the general procedure B the title compound was isolated as a brown solid eluting with 4% ethyl acetate in hexane (Yield 82%, 57 mg). ^{1}H NMR (400 MHz, $CDCl_3$) δ 8.54 (s, 1H), 8.00-7.98 (m, 2H), 7.69-7.66 (m, 2H), 6.66-6.64 (m, 1H), 6.57 (dd, *J* = 7.9, 1.2 Hz, 1H), 5.84 (s, 1H), 3.21 (dd, *J* = 9.0, 6.6 Hz, 2H), 3.03 (dd, *J* = 9.0, 6.6 Hz, 2H). $^{13}C\{^{1}H\}$ NMR (100 MHz, $CDCl_3$) δ 163.1, 147.8, 146.0, 142.3, 141.4, 134.6, 130.1, 129.3, 129.2, 129, 121.4, 109, 109, 108.4, 100.92, 38.4, 35.1. HRMS (ESI-TOF) m/z: $[M+H]^+$ Calcd for $C_{17}H_{14}N_2O_2$ 279.1128; Found 279.1132.



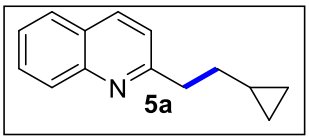
2-Phenethylpyridine (4m)[3]: Following the general procedure B the title compound was isolated as a light brown oil eluting with 3% ethyl acetate in hexane (Yield 47%, 21 mg). ^{1}H NMR (400 MHz, $CDCl_3$) δ 8.57-8.55 (m, 1H), 7.58-7.55 (m, 1H), 7.27 (dd, J = 4.3, 3.5 Hz, 2H), 7.20 (dd, J = 7.7, 2.1 Hz, 3H), 7.13-7.06 (m, 2H), 3.13-3.08 (m, 2H), 3.07-3.02 (m, 2H); ^{13}C{^{1}H} NMR (100 MHz, $CDCl_3$) δ 161.2, 149.3, 141.5, 136.3, 128.4, 128.3, 125.9, 123.0, 121.1, 40.2, 36.0.



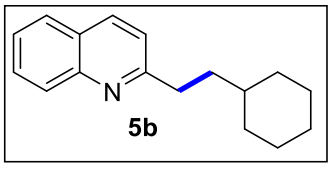
1-Benzyl-2-phenethyl-1H-benzo[d]imidazole (4l)[4]: Following the general procedure A, the title compound was isolated as a light brown oil eluting with 15% ethyl acetate in hexane (Yield 35%, 27 mg). ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.80 (d, J = 7.9 Hz, 1H), 7.28-7.24 (m, 6H), 7.21 (d, J = 4.2 Hz, 3H), 7.17-7.14 (m, 2H), 7.01-6.96 (m, 2H), 5.18 (s, 2H), 3.18-3.09 (m, 4H); ^{13}C{^{1}H} NMR (100 MHz, $CDCl_3$) δ 154.5, 142.7, 140.8, 135.9, 135.3, 129.0, 128.5, 128.4, 127.9, 126.3, 126.1, 122.4, 122.1, 119.3, 109.5, 46.7, 34.0, 29.7.



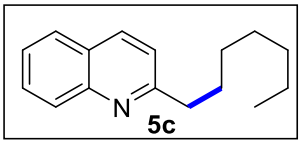
2-(2-Cyclopropylethyl)quinoline (5a)[2]: Following the general procedure C the title compound was isolated as a pale yellow oil eluting with 1% ethyl acetate in hexane (Yield 57%, 28mg).^{1}H NMR (400 MHz, $CDCl_3$) δ 8.06 (t, J = 8.2 Hz, 2H), 7.78-7.66 (m, 2H), 7.50-7.42 (m, 1H), 7.32-7.30 (m, 1H), 3.10-3.06 (m, 2H), 1.76-1.70 (m, 2H), 0.80-0.76 (m, 1H), 0.45-0.41 (m, 2H), 0.11-0.07 (m, 2H); ^{13}C{^{1}H} NMR (100 MHz, $CDCl_3$) δ 162.9, 148.0, 136.2, 129.4, 128.9, 127.6, 126.8, 125.7, 121.6, 39.5, 35.2, 11.0, 8.1, 4.7.



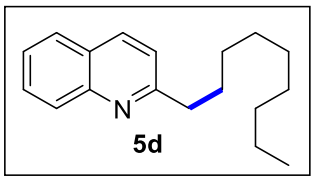
2-(2-Cyclohexylethyl)quinoline (5b)[2]: Following the general procedure C the title compound was isolated as a pale yellow oil eluting with 1% ethyl acetate in hexane (Yield 42%, 25 mg). ^{1}H NMR (500 MHz, $CDCl_3$) δ 8.10-8.05 (m, 2H), 7.79 (d, J = 8.1 Hz, 1H), 7.70 (t, J = 7.4 Hz, 1H), 7.50 (t, J = 7.5 Hz, 1H), 7.34-7.28 (m, 1H), 3.02-2.99 (m, 2H), 1.84 (d, J = 12.0 Hz, 2H), 1.72 (t, J = 12.0 Hz, 4H), 1.37 (d, J = 9.9 Hz, 1H), 1.30-1.16 (m, 4H), 1.00 (q, J = 12.1 Hz, 2H); ^{13}C{^{1}H} NMR (100 MHz, $CDCl_3$) δ 163.6, 148.0, 136.3, 129.4, 128.9, 127.6, 126.8, 125.7, 121.5, 37.9, 37.0, 33.4, 32.6, 26.7, 26.4.



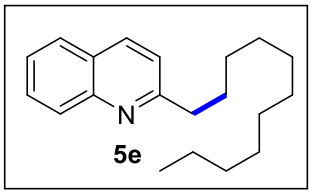
2-Heptylquinoline (5c)[2]: Following the general procedure C the title compound was isolated as a pale yellow oil eluting with 1% ethyl acetate in hexane (Yield 44%, 25 mg). ^{1}H NMR (400 MHz, $CDCl_3$) δ 8.06 (t, J = 8.2 Hz, 2H), 7.78-7.74 (m, 1H), 7.68 (dd, J = 7.7, 6.2 Hz, 1H), 7.50 -7.43 (m, 1H), 6.83 (dd, J = 14.6, 7.9 Hz, 1H), 3.00-2.96 (m, 1H), 2.34 (d, J = 6.9 Hz, 1H), 1.90-1.75 (m, 1H), 1.57-1.54 (m, 1H), 1.38-1.36 (m, 6H), 0.93-0.86 (m, 5H); ^{13}C{^{1}H} NMR (100 MHz, $CDCl_3$) δ 156.5, 136.2, 131.0, 129.5, 129.1, 127.4, 127.1, 125.8, 118.7, 33.0, 31.5, 30.1, 29.5, 28.6, 22.6, 14.0.



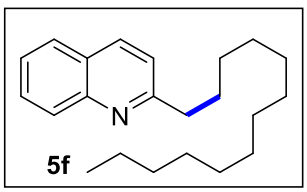
2-Nonylquinoline (5d)[1]: Following the general procedure C the title compound was isolated as a pale yellow oil eluting with 1% ethyl acetate in hexane (Yield 49%, 31 mg). ^{1}H NMR (400 MHz, $CDCl_3$) δ 8.04 (t, J = 8.0 Hz, 2H), 7.76 (dd, J = 8.1, 1.2 Hz, 1H), 7.69-7.65 (m, 1H), 7.51-7.43 (m, 1H), 7.29 (d, J = 8.4 Hz, 1H), 2.99-2.92 (m, 2H), 1.86-1.74 (m, 2H), 1.43-1.22 (m, 12H), 0.86 (t, J = 6.9 Hz, 3H); ^{13}C{^{1}H} NMR (100 MHz, $CDCl_3$) δ 163.1, 147.9, 136.2, 129.3, 128.8, 127.5, 126.7, 125.6, 121.4, 39.4, 31.9, 30.1, 29.6, 29.5, 29.5, 29.3, 22.6, 14.1.



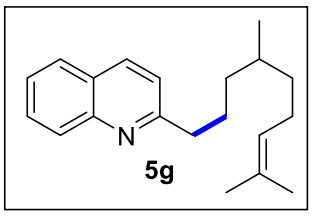
2-Undecylquinoline (5e)[1]: Following the general procedure C the title compound was isolated as a pale yellow oil eluting with 1% ethyl acetate in hexane (Yield 50%, 35 mg). ^{1}H NMR (400 MHz, $CDCl_3$) δ 8.07-8.00 (m, 2H), 7.79-7.72 (m, 1H), 7.69-7.62 (m, 1H), 7.52 (d, J = 8.6 Hz, 1H), 7.48-7.42 (m, 1H), 2.99-2.92 (m, 1H), 2.35-2.29 (m, 1H), 1.85-1.73 (m, 1H), 1.53 (dd, J = 15.1, 7.3 Hz, 1H), 1.29-1.21 (m, 16H), 0.86 (dd, J = 4.3, 2.7 Hz, 3H); ^{13}C{^{1}H} NMR (100 MHz, $CDCl_3$) δ 161.3, 147.0, 140.8, 136.0, 130.7, 129.2, 127.4, 120.6, 113.2, 40.6, 32.0, 31.0, 30.2, 27.0, 22.8, 14.2.



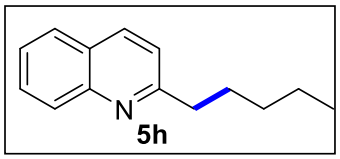
2-Tridecylquinoline (5f)[1]: Following the general procedure C the title compound was isolated as a pale yellow oil eluting with 1% ethyl acetate in hexane (Yield 53%, 39 mg). ^{1}H NMR (400 MHz, $CDCl_3$) δ 8.03 (dd, J = 15.8, 8.0 Hz, 2H), 7.75 (t, J = 8.7 Hz, 1H), 7.66 (dd, J = 9.9, 5.3 Hz, 1H), 7.53-7.42 (m, 1H), 7.29 (d, J = 8.4 Hz, 1H), 2.97-2.93 (m, 2H), 1.83-1.75 (m, 2H), 1.28-1.16 (s, 20H), 0.86 (t, J = 6.7 Hz, 3H); ^{13}C{^{1}H} NMR (100 MHz, $CDCl_3$) δ 163.2, 147.9, 136.2, 129.3, 128.8, 127.5, 126.7, 125.6, 121.4, 39.4, 31.9, 30.1, 29.7, 29.6, 29.6, 29.6, 29.6, 29.5, 29.5, 29.3, 22.7, 14.1.



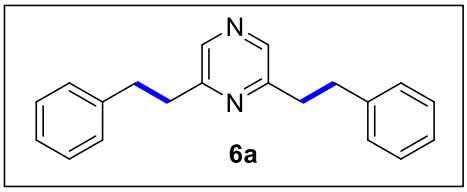
2-(4,8-Dimethylnon-7-en-1-yl)quinoline (5g)[1]: Following the general procedure C the title compound was isolated as a pale yellow oil eluting with 1% ethyl acetate in hexane (Yield 57%, 40 mg). ^{1}H NMR (500 MHz, $CDCl_3$) δ 8.70 (dd, J = 4.1, 1.4 Hz, 1H), 8.03 (dd, J = 8.2, 1.4 Hz, 1H), 7.34-7.26 (m, 2H), 7.18 (d, J = 8.2 Hz, 1H), 7.00 (d, J = 8.2 Hz, 1H), 5.94 (t, J = 32.0 Hz, 1H), 3.58-3.53 (m, 2H), 2.95 (t, J = 6.3 Hz, 2H), 2.20 (s, 3H), 2.09 (dd, J = 11.6, 6.0 Hz, 2H), 1.72-1.58 (m, 5H), 1.28 (s, 3H), 0.90 (dd, J = 11.6, 5.3 Hz, 3H); ^{13}C{^{1}H} NMR (125 MHz, $CDCl_3$) δ 146.9, 140.7, 137.5, 135.9, 129.1, 127.4, 121.2, 120.5, 116.6, 113.1, 41.3, 31.9, 30.9, 29.7, 29.4, 27.0, 22.7, 21.8, 14.1.



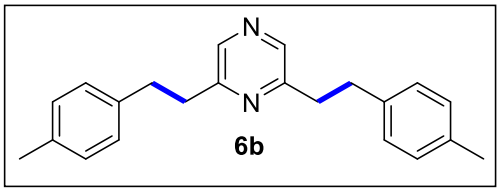
2-Pentyl quinoline (5h)[6]: Following the general procedure C the title compound was isolated as a pale yellow oil eluting with 1% ethyl acetate in hexane (Yield 57%, 28mg). ^{1}H NMR (400 MHz, $CDCl_3$) δ 8.04 (dd, J = 8.1, 6.3 Hz, 2H), 7.80-7.74 (m, 1H), 7.71-7.63 (m, 1H), 7.52-7.44 (m, 1H), 7.29 (d, J = 8.4 Hz, 1H), 3.00-2.92 (m, 2H), 2.31-2.25 (m, 1H), 1.86-1.78 (m, 2H), 1.40-1.36 (m, 2H), 1.03-0.94 (m, 1H), 0.93-0.87 (m, 3H). ^{13}C{^{1}H} NMR (100 MHz, $CDCl_3$) δ 163.2, 148.0, 136.3, 129.4, 128.9, 127.6, 126.8, 125.7, 121.5, 121.4, 39.4, 31.8, 29.8, 22.6, 14.1.



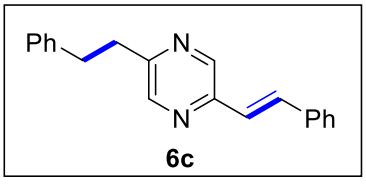
2,6-Diphenethylpyrazine (6a)[1]: Following the general procedure E the title compound was isolated as a colorless oil eluting with 3% ethyl acetate in hexane (Yield 77%, 55 mg). ^{1}H NMR (400 MHz, $CDCl_3$) δ 8.15 (s, 2H), 7.28 (dd, J = 11.1, 3.9 Hz, 4H), 7.19 (dd, J = 12.7, 7.2 Hz, 6H), 3.13-3.05 (m, 8H); ^{13}C{^{1}H} NMR (125 MHz, $CDCl_3$) δ 155.7, 141.8, 140.9, 128.5, 128.4, 126.1, 37.1, 35.4.



2,6-Bis(4-methylphenethyl)pyrazine (6b)[1]: Following the general procedure E the title compound was isolated as a colorless oil eluting with 3% ethyl acetate in hexane (Yield 65%, 51 mg). ^{1}H NMR (400 MHz, $CDCl_3$) δ 8.14 (s, 2H), 7.08-7.06 (m, 8H), 3.10-3.06 (m, 4H), 3.04-2.99 (m, 4H), 2.31 (s, 6H); ^{13}C{^{1}H} NMR (125 MHz, $CDCl_3$) δ 155.7, 141.7, 137.8, 135.6, 129.1, 128.3, 37.2, 35.0, 21.0.

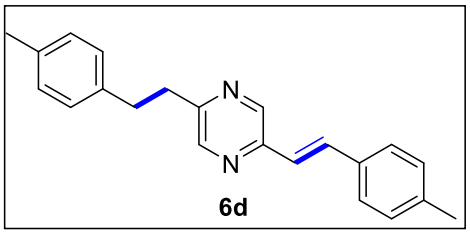


(*E*)-2-Phenethyl-5-styrylpyrazine (6c)[5]: Following the general procedure E the title compound was isolated as a colorless solid eluting with 3% ethyl acetate in



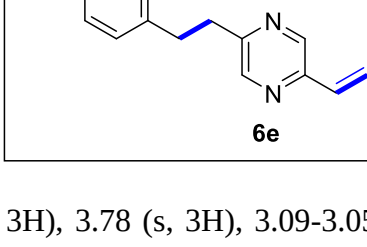
hexane (Yield 85%, 61 mg). ^{1}H NMR (400 MHz, $CDCl_3$) δ 8.57 (s, 1H), 8.30 (s, 1H), 7.67 (d, *J* = 16.1 Hz, 1H), 7.58 (d, *J* = 7.4 Hz, 2H), 7.38 (t, *J* = 7.4 Hz, 2H), 7.34-7.25 (m, 3H), 7.23-7.11 (m, 4H), 3.15-3.04 (m, 4H).; $^{13}C\{^{1}H\}$ NMR (100 MHz, $CDCl_3$) *δ* 154.6, 148.7, 143.9, 142.8, 140.8, 136.3, 134.1, 128.8, 128.8, 128.5, 128.4, 127.2, 126.2, 124.1, 37.0, 35.5.

(*E*)-2-(4-methylphenethyl)-5-(4-methylstyryl)pyrazine (6d): Following the general procedure E the title compound was isolated as a colorless solid eluting with 4% ethyl acetate in hexane (Yield 70%, 55 mg). ^{1}H NMR (400 MHz, $CDCl_3$) *δ* 8.56 (d, *J* = 1.4 Hz, 1H), 8.30 (d, *J* = 1.4 Hz, 1H), 7.66 (d, *J* = 16.1 Hz, 1H), 7.49 (d, *J* = 8.1 Hz, 2H), 7.20 (d, *J* = 8.0 Hz, 2H), 7.14-7.10 (m, 1H), 7.09 (d, *J* = 1.6 Hz, 4H), 3.13-3.01 (m, 4H), 2.38 (s, 3H), 2.32 (s, 3H); $^{13}C\{^{1}H\}$ NMR (100 MHz, $CDCl_3$) *δ* 154.4, 148.8, 143.9, 142.6, 138.9, 137.7, 135.7, 133.9, 133.5, 129.5, 129.2, 128.3, 127.1, 123.1, 37.1, 35.1, 21.4, 21.0. HRMS (ESI-TOF) m/z: $[M+H]^+$ Calcd for $C_{22}H_{22}N_2$ 315.1856; Found 315.1896.



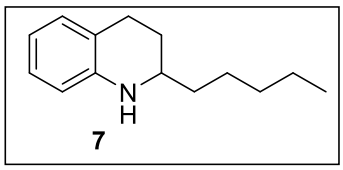
6d

(*E*)-2-(4-Methoxyphenethyl)-5-(4-methoxystyryl)pyrazine (6e): Following the general procedure E the title compound was isolated as a greenish solid eluting with 6% ethyl acetate in hexane (Yield 62%, 54 mg). ^{1}H NMR (400 MHz, $CDCl_3$) *δ* 8.54 (d, *J* = 1.4 Hz, 1H), 8.26 (d, *J* = 1.4 Hz, 1H), 7.64 (d, *J* = 16.1 Hz, 1H), 7.53 (d, *J* = 8.1 Hz, 1H), 7.11-7.06 (m, 3H), 7.03 (d, *J* = 1.61 Hz, 1H), 6.93-6.91 (m, 1H), 6.83-6.80 (m, 3H), 3.84 (s, 3H), 3.78 (s, 3H), 3.09-3.05 (m, 2H), 3.02-2.97 (m, 2H); $^{13}C\{^{1}H\}$ NMR (100 MHz, $CDCl_3$) *δ* 160.3, 158.1, 154.2, 153.9, 149.1, 143.9, 142.6, 133.7, 133.0, 133.0, 129.5, 128.7, 122.0, 114.3, 114.0, 55.4, 55.3, 37.3, 34.7. HRMS (ESI-TOF) m/z: $[M+H]^+$ Calcd for $C_{22}H_{22}N_2O_2$ 347.1754; Found 347.1755.



6e

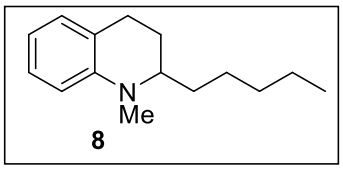
2-Pentyl-1,2,3,4-tetrahydroquinoline (7)[7]: Compound **6a** (50 mg, 0.25 mmol) and $NiCl_2.6H_2O$ (6 mg, 0.044 mmol) were taken in a 50 mL RB and dissolved in 3 mL of methanol. Then $NaBH_4$ (38 mg, 1.0 mmol) was added in portion at 0 °C and stirred for 30 min at RT. After completion of the reaction methanol was evaporated and black ppt. was dissolved in 10% HCl, the acidic solution was basified by adding conc. ammonium hydroxide solution and



7

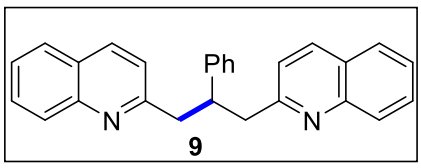
then extracted with ether. The extract was dried over $MgSO_4$, evaporated and purified by column chromatography to yield the desired product as yellow oil (46 mg, 90% yield). ^{1}H NMR (400 MHz, $CDCl_3$) δ 6.95 (t, J = 7.2 Hz, 2H), 6.61-6.57 (m, 1H), 6.47 (dd, J = 8.4, 1.0 Hz, 1H), 3.24-3.20 (m, 1H), 2.81-20.79 (m, 2H), 1.99-1.92 (m, 1H), 1.64-1.57 (m, 1H), 1.47 (t, J = 6.7 Hz, 2H), 1.38-1.31 (m, 4H), 1.27-1.18 (m, 2H), 0.90 (t, J = 6.8 Hz, 3H).^{13}C{H} NMR (100 MHz, $CDCl_3$) δ ^{13}C{^{1}H} NMR (100 MHz, $CDCl_3$) δ 143.9, 128.4, 125.9, 120.6, 116.1, 113.2, 50.8, 35.9, 31.2, 27.3, 25.6, 24.6, 21.8, 13.2.

1-Methyl-2-pentyl-1,2,3,4-tetrahydroquinoline (8)[7]: In a 25 mL RB compound **6aa** (46 mg 0.23 mmol), K_2CO_3 (32 mg, 0.23 mmol), MeI (1.36 mmol) and THF (3 mL) were taken, sealed and refluxed for 20h. The reaction mixture was cooled to rt, then H_2O (3 mL) was added and the aqueous phase was extracted with EtOAc. The combined organic extracts were washed with brine, dried over $MgSO_4$ then concentrated in vacuo. Purification afforded the desired product **6ab** (42 mg, 85% yield). ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.07 (t, J = 7.6 Hz, 1H), 6.96 (d, J = 7.3 Hz, 1H), 6.57 (t, J = 7.3 Hz, 1H), 6.51 (d, J = 8.2 Hz, 1H), 3.22 (dd, J = 8.6, 4.3 Hz, 1H), 2.91 (s, 3H), 2.85-2.74 (m, 1H), 2.67-2.62 (m, 1H), 1.90-1.86 (m, 2H), 1.46-1.20 (m, 8H), 0.88 (t, J = 6.8 Hz, 3H). ^{13}C{^{1}H} NMR (100 MHz, $CDCl_3$) δ 128.8, 127.4, 127.1, 121.9, 115.3, 110.4, 59.8, 32.1, 31.2, 25.8, 24.4, 23.6, 22.8, 14.2.



8

2,2'- (2-phenylpropane-1,3-diyl)diquinoline (9): Following the general procedure A the title compound was isolated as a colorless liquid eluting with 4% ethyl acetate in hexane (Yield 50%, 47 mg). ^{1}H NMR (400 MHz, $CDCl_3$) δ 8.05 (dd, J = 16.3, 5.8 Hz, 2H), 7.84 (t, J = 10.2 Hz, 2H), 7.71-7.60 (m, 4H), 7.51-7.41 (m, 2H), 7.20-7.12 (m, 2H), 7.17-7.12 (m, 2H), 7.10-7.04 (m, 3H), 4.03-3.93 (m, 1H), 3.53-3.31 (m, 4H); ^{13}C{^{1}H} NMR (100 MHz, $CDCl_3$) δ 160.8, 147.6, 143.7, 136.1, 129.4, 128.6, 128.4, 128.0, 127.5, 126.8, 126.4, 125.9, 122.3, 46.4, 45.5. HRMS (ESI-TOF) m/z: $[M+H]^+$ Calcd for $C_{27}H_{23}N_2$ 375.1861; Found 375.1891.

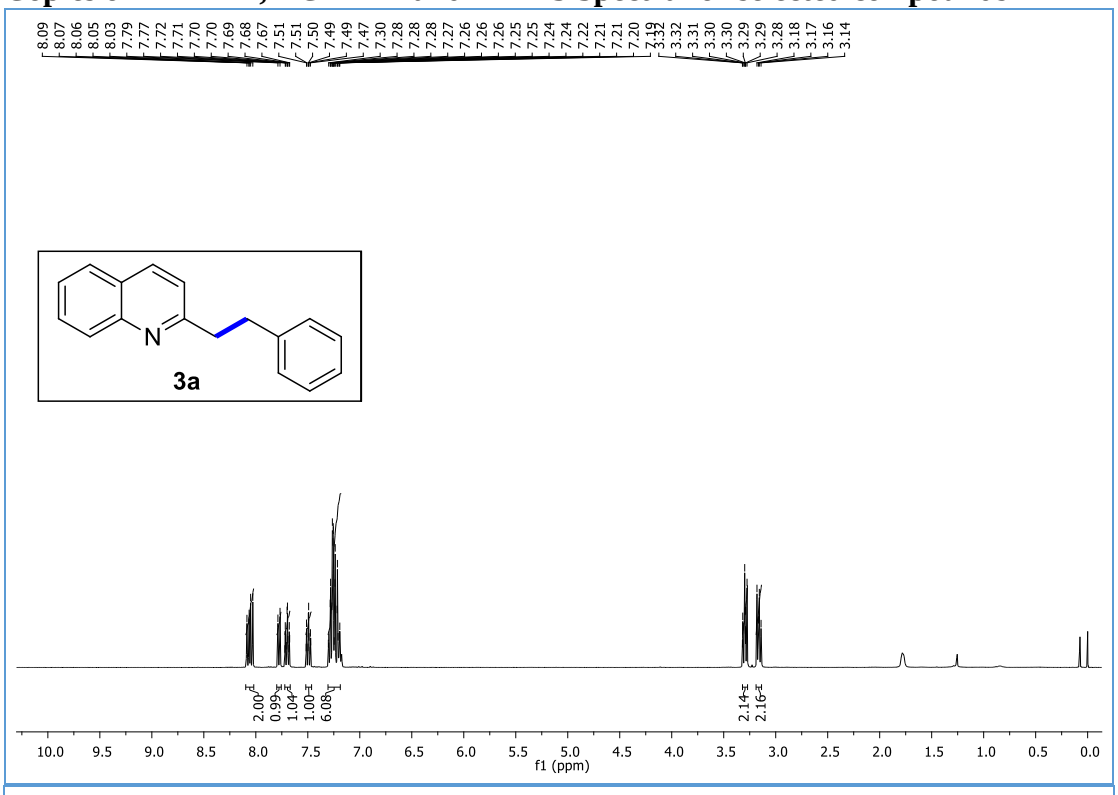


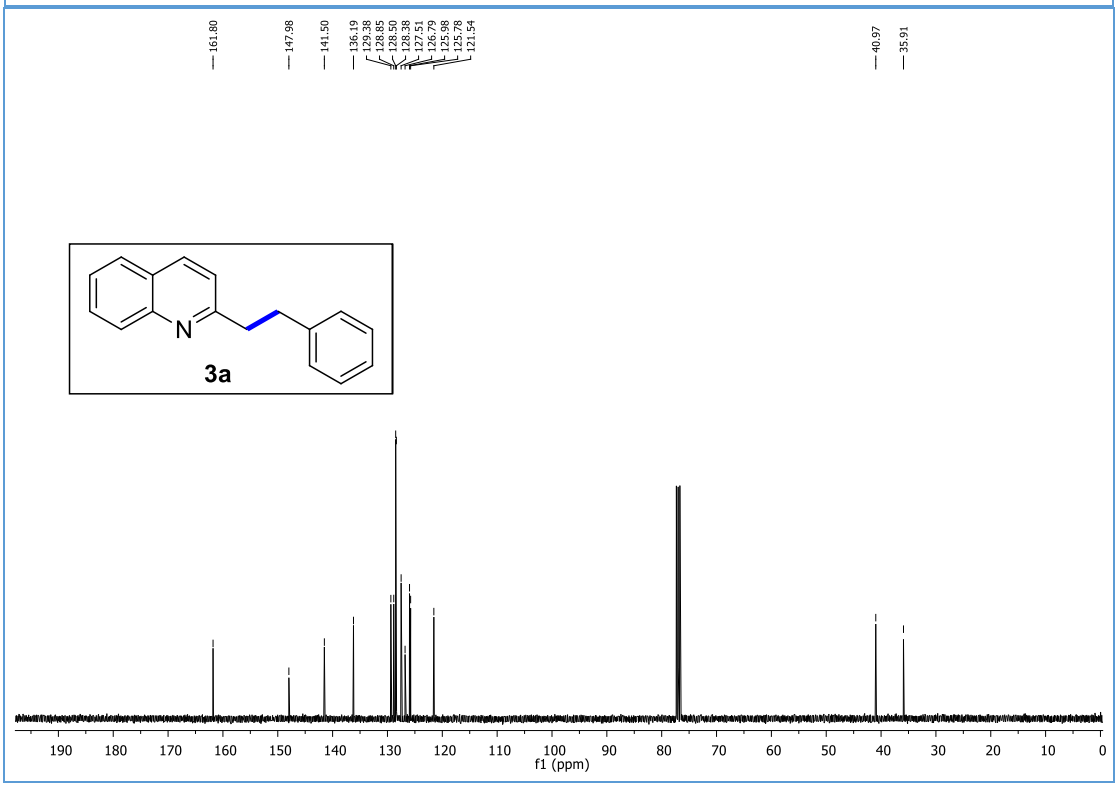
9

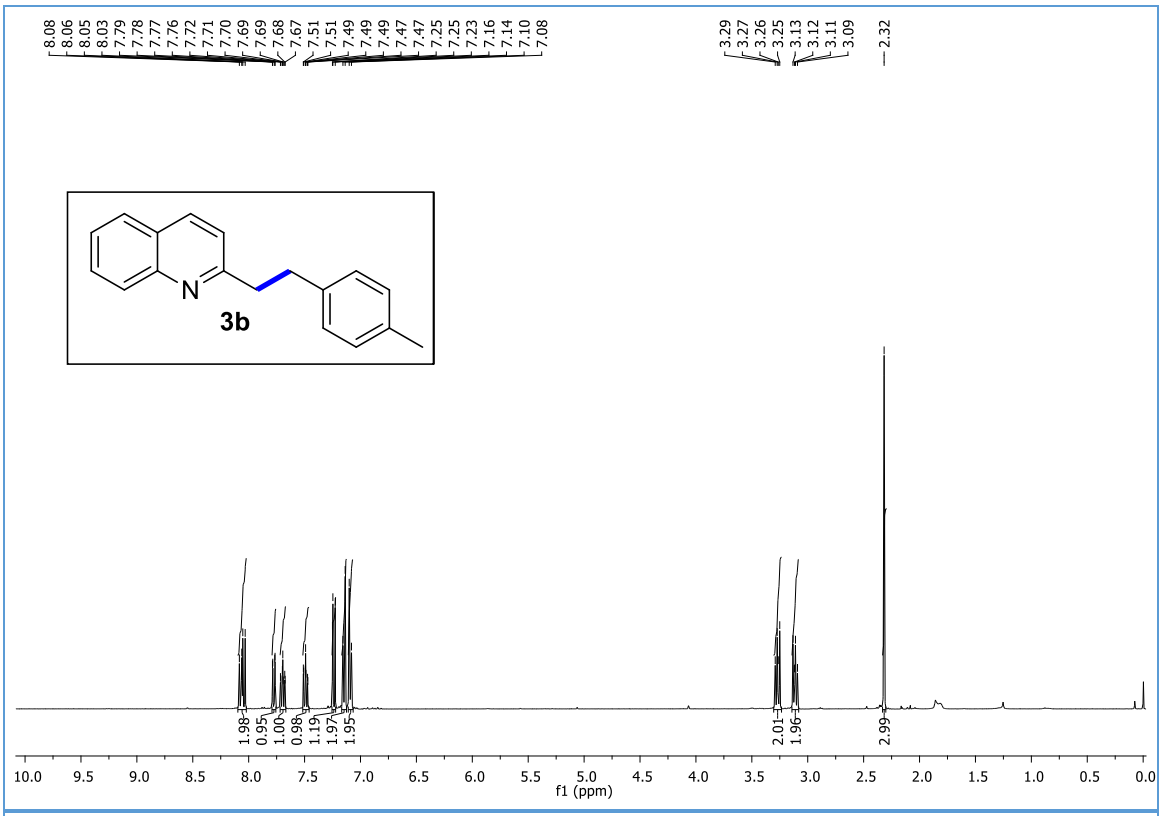
[1.5] References:

1. M. Vellakkaran, J. Das, S. Bera and D. Banerjee, *Chem. Commun.* 2018, ***54***, 12369-12372.
2. J. Rana, R. Babu, M. Subaramanian and E. Balaraman, *Org. Chem. Front*. 2018, **5**, 3250-3255.
3. T.-Y. Feng, H.-X. Li, D. J. Young and J.-P. Lang, *J. Org. Chem.* 2017, ***82***, 4113-4120.
4. W.-C. Shih, W.-C. Chen, Y.-C. Lai, M.-S. Yu, J.-J. Ho, G. P. A. Yap, and T.-G. Ong, *Org. Lett.* 2012, ***14***, 2046-2049.
5. J. Das, M. Vellakkaran and D. Banerjee, *Chem. Commun.* 2019, ***55,*** 7530-7533.
6. S. Genc, B. Arslan, S. Gulcemal, S. Gunnaz, B. Cetinkaya and D. Gulcemal, *J. Org. Chem.* 2019, ***84***, 6286-6297.
7. T. Wang, L.-G. Zhuo, Z. Li, F. Chen, Z. Ding, Y. He, Q.-H. Fan, J. Xiang, Z.-X. Yu and A. S. C. Chan, *J. Am. Chem. Soc*. 2011, ***133***, 9878-9891.

[1.6] Copies of ^{1}H NMR, ^{13}C NMR and HRMS Spectra for selected compounds



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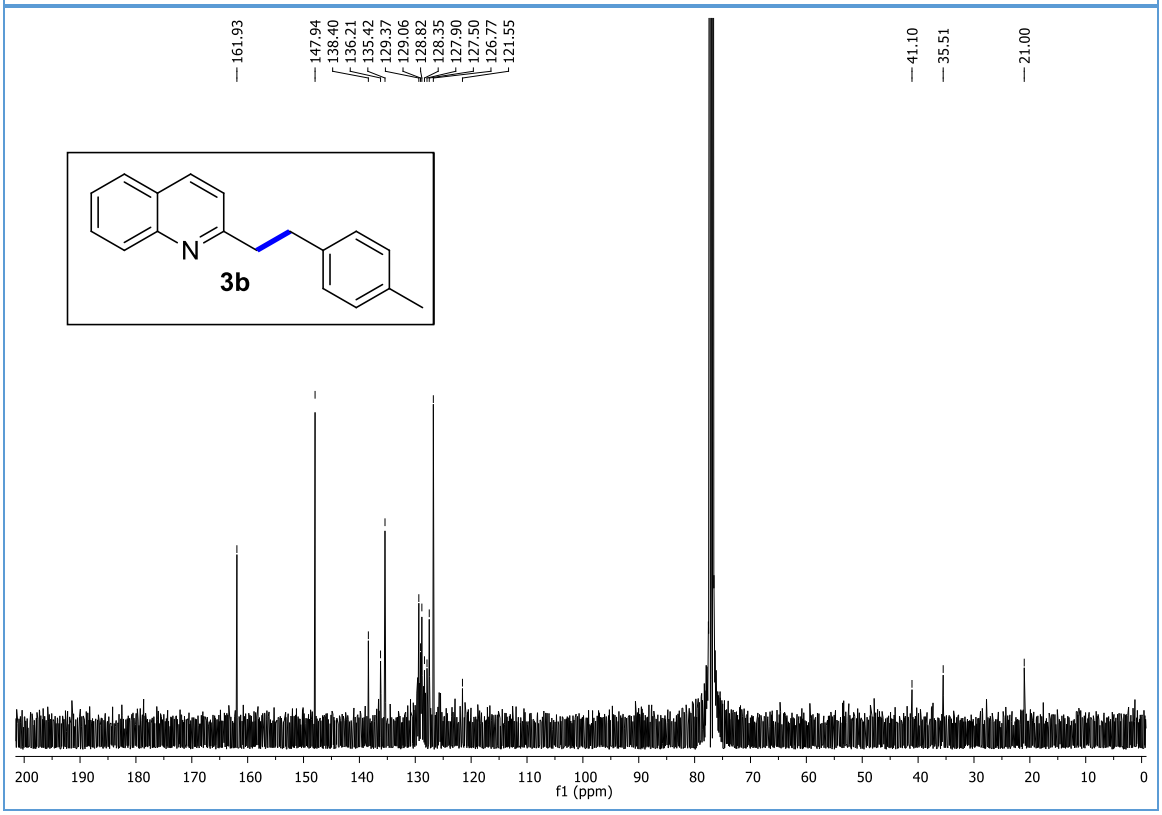
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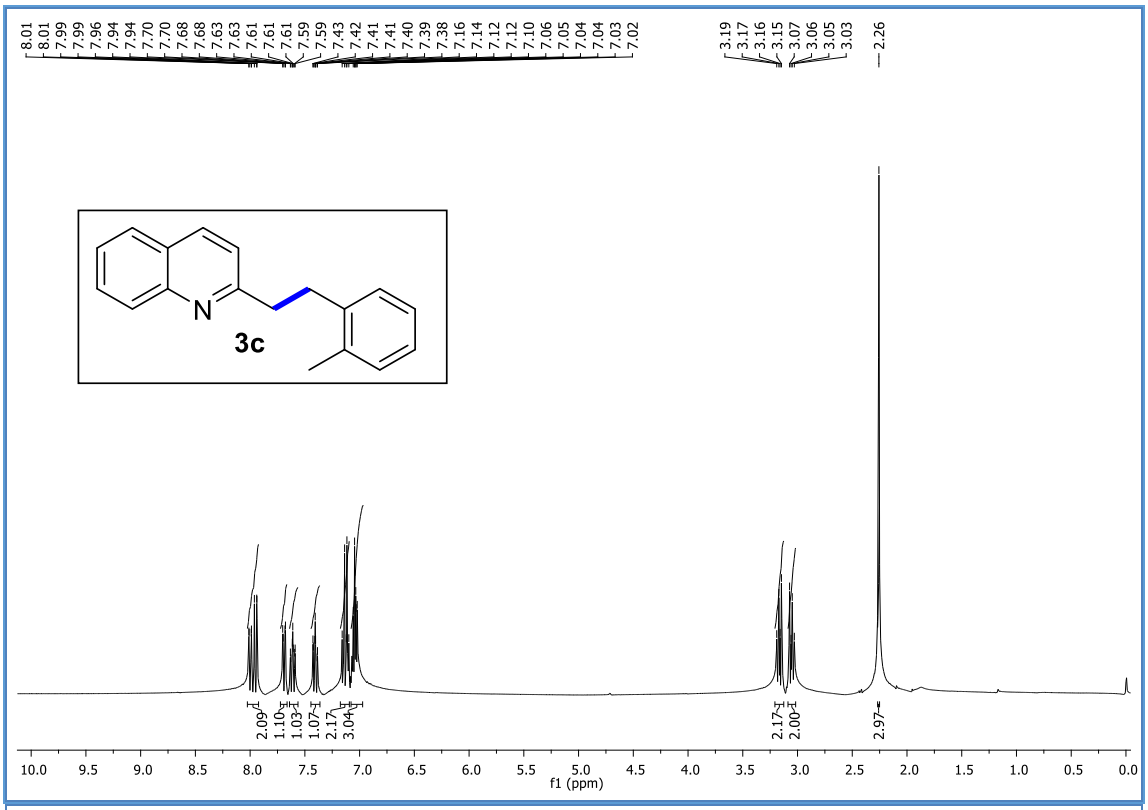
f1 (ppm)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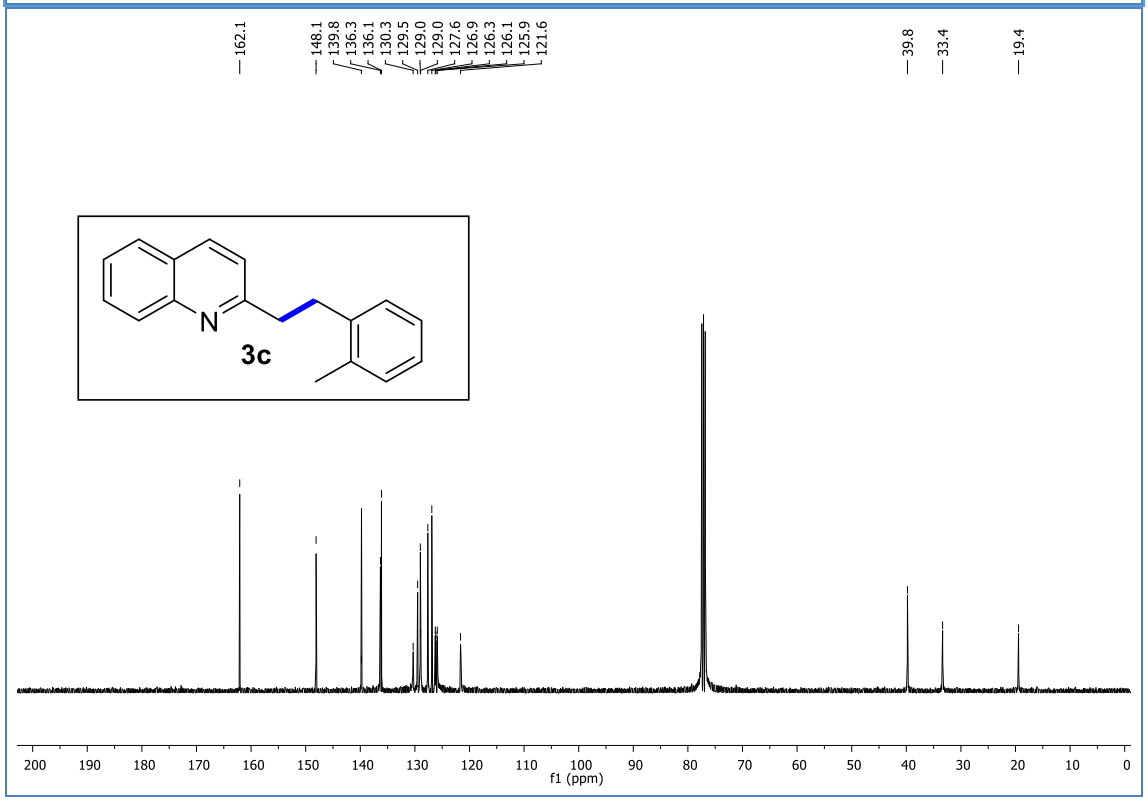
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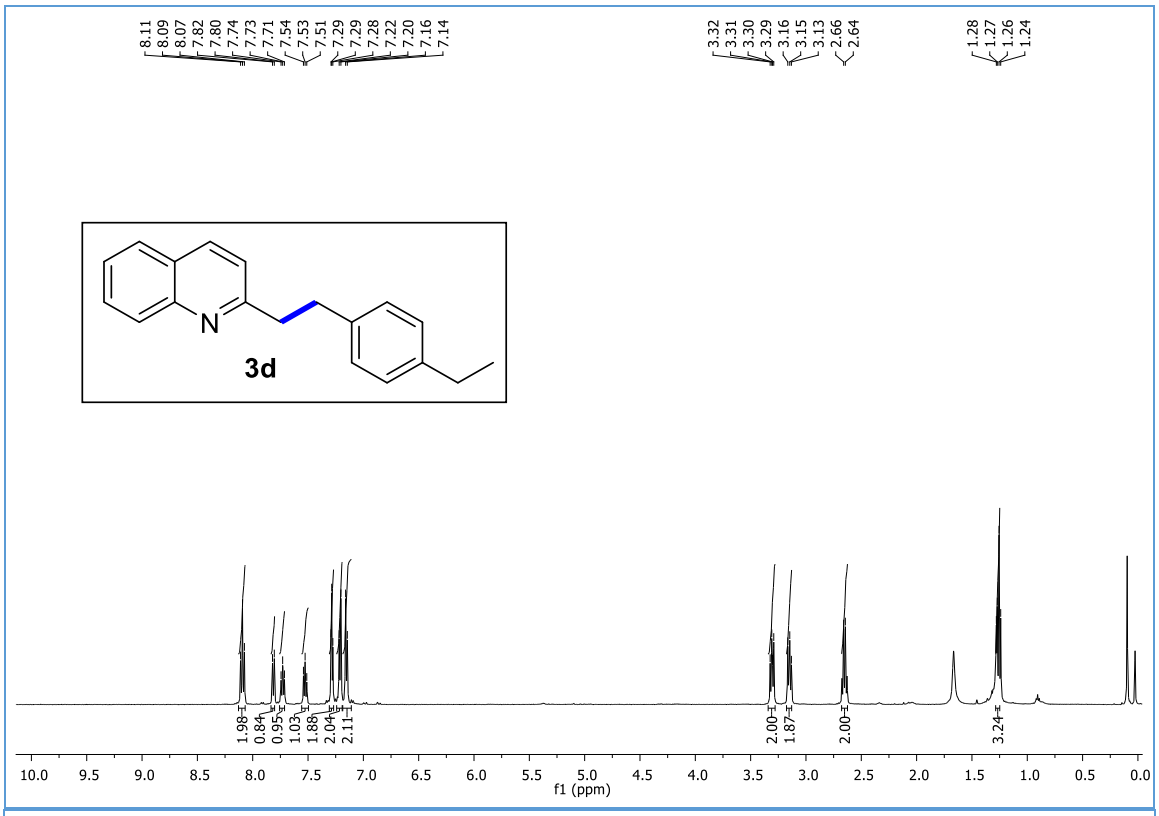
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8.0
7.5
7.0
6.5
6.0
5.5
5.0
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3.5
3.0
2.5
2.0
1.5
1.0
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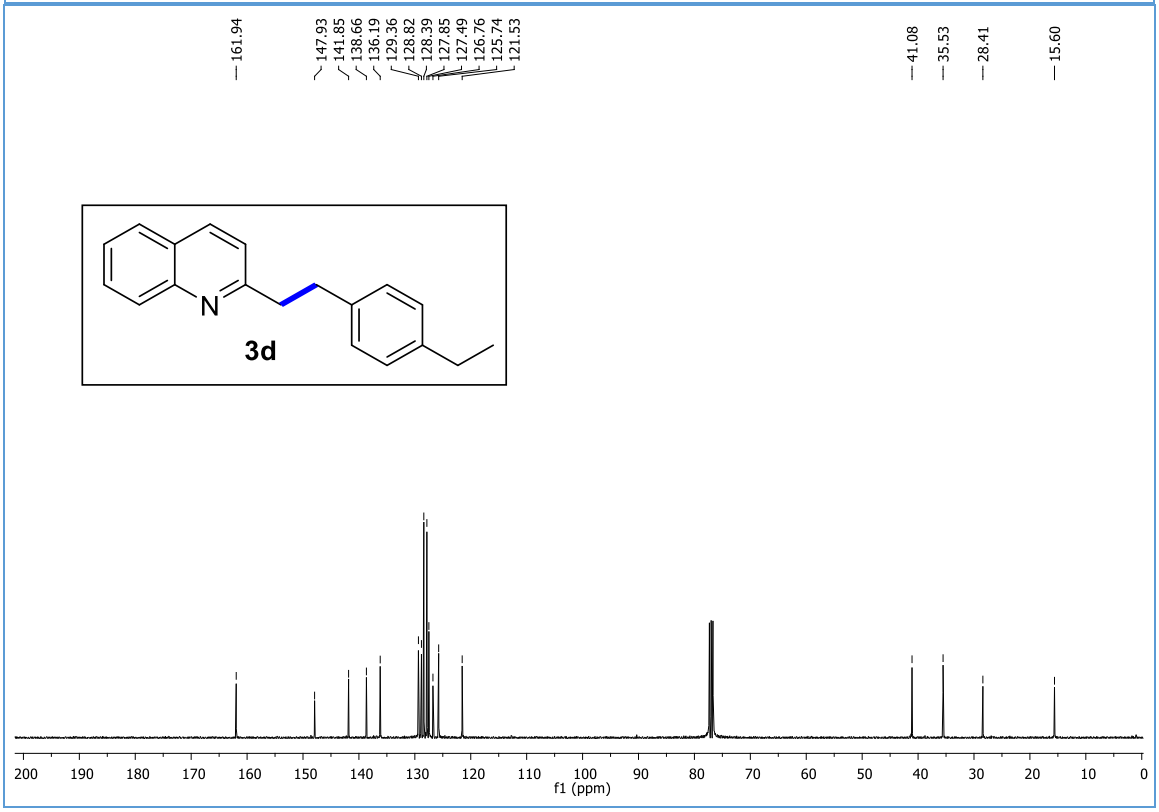
0.0
f1 (ppm)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128.39
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127.49
126.76
125.74
121.53
41.08
35.53
28.41
15.60
N
3d
200
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
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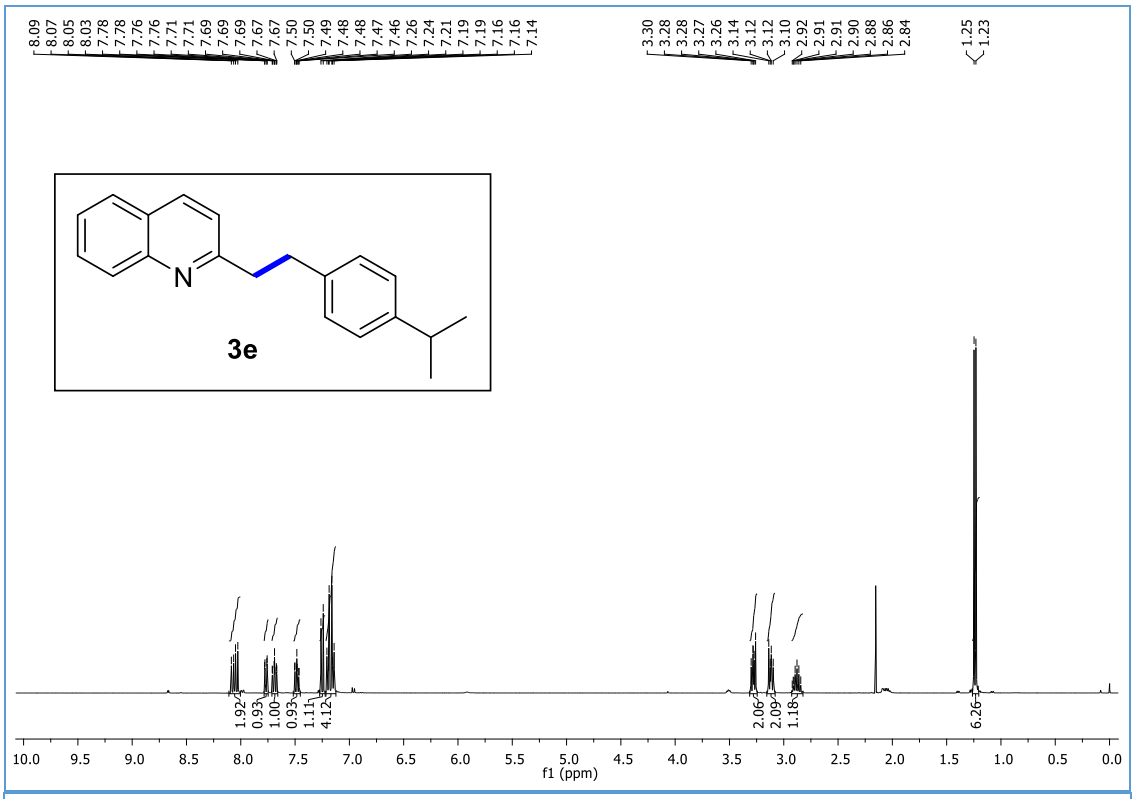
f1 (ppm)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7.69
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7.67
7.67
7.50
7.50
7.49
7.48
7.48
7.47
7.46
7.26
7.24
7.21
7.19
7.16
7.16
7.14
3.30
3.28
3.28
3.27
3.26
3.14
3.12
3.12
3.10
2.92
2.91
2.91
2.90
2.88
2.86
2.84
1.25
1.23
N
3e
1.92
0.93
1.00
0.93
1.11
4.12
2.06
2.09
1.18
6.26
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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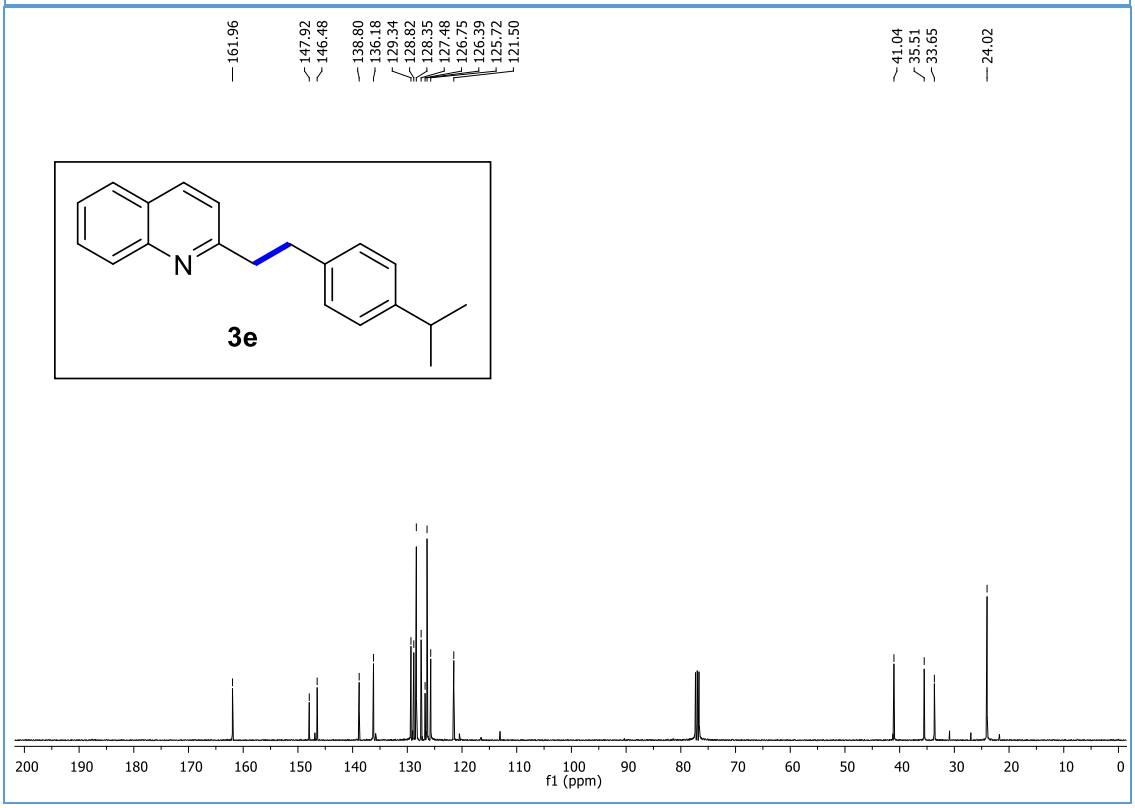
3.5
3.0
2.5
2.0
1.5
1.0
0.5
0.0
f1 (ppm)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126.75
126.39
125.72
121.50
41.04
35.51
33.65
24.02
N
3e
200
190
180
170
160
150
140
130
120
110
100
90
80
70
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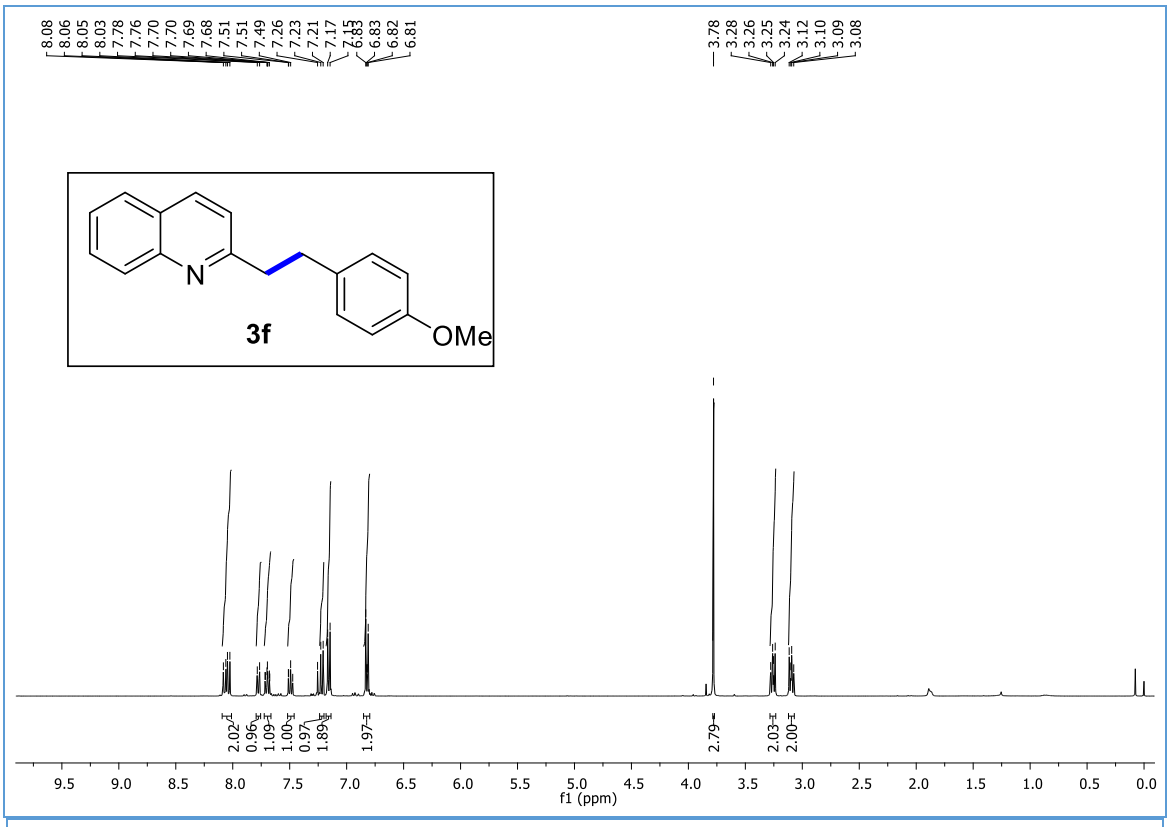
50
40
30
20
10
0
f1 (ppm)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8.06
8.05
8.03
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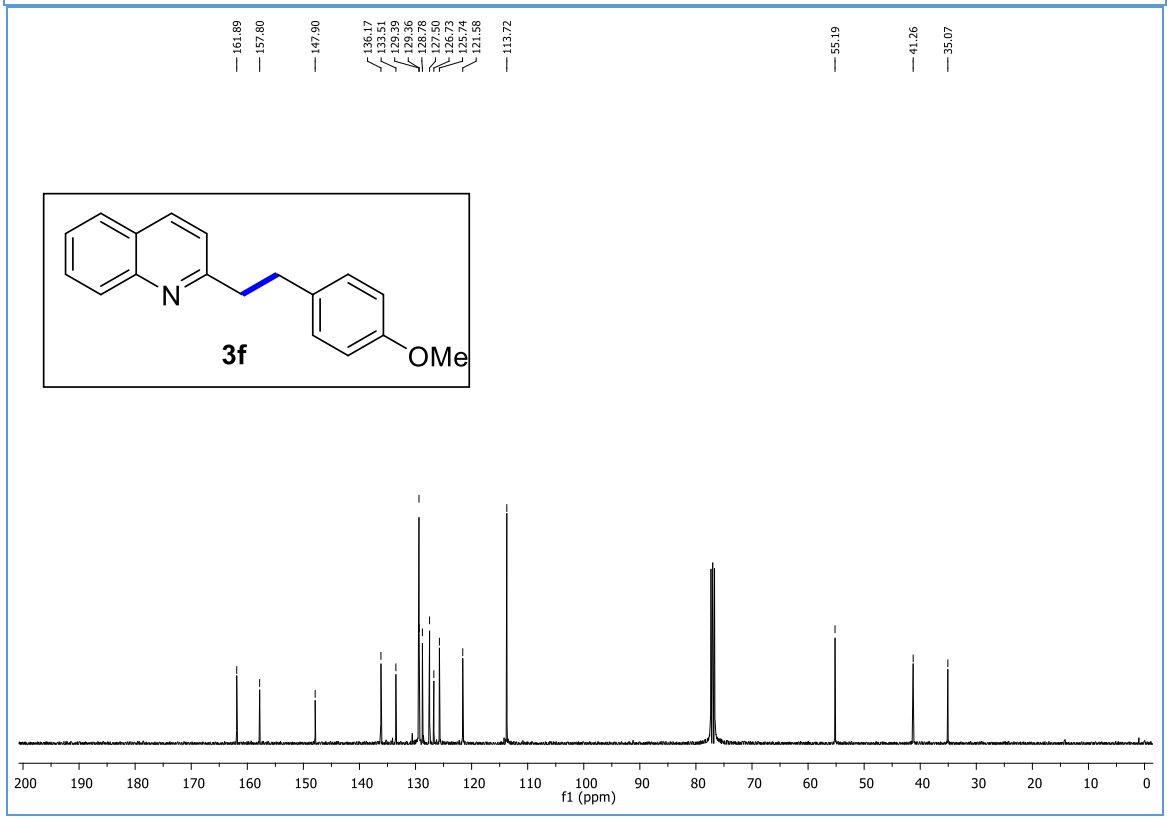
7.76
7.70
7.70
7.69
7.68
7.51
7.51
7.49
7.26
7.23
7.21
7.17
6.83
6.82
6.81
3.78
3.28
3.26
3.25
3.24
3.12
3.10
3.09
3.08
N
3f
OMe
2.02
0.96
1.09
1.00
0.97
1.89
1.97
2.79
2.03
2.00
9.5 9.0 8.5 8.0 7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 0.5 0.0
f1 (ppm)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157.80
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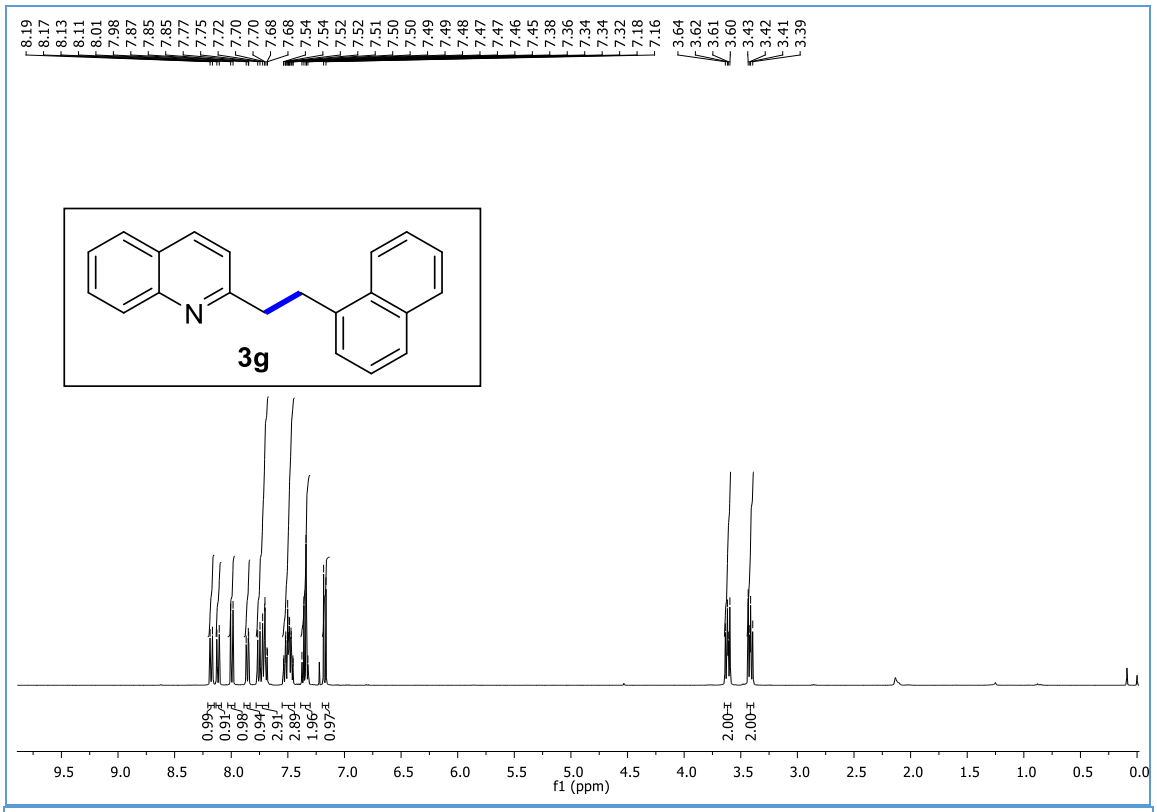
129.36
128.78
127.50
126.73
125.74
121.58
113.72
55.19
41.26
35.07
N
3f
OMe
200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0
f1 (ppm)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7.85
7.77
7.75
7.72
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7.70
7.68
7.68
7.54
7.54
7.52
7.52
7.51
7.50
7.50
7.49
7.49
7.48
7.47
7.47
7.46
7.45
7.38
7.36
7.34
7.34
7.32
7.18
7.16
3.64
3.62
3.61
3.60
3.43
3.42
3.41
3.39
N
3g
0.99
0.91
0.98
0.94
2.91
2.89
1.96
0.97
2.00
2.00
9.5
9.0
8.5
8.0
7.5
7.0
6.5
6.0
5.5
5.0
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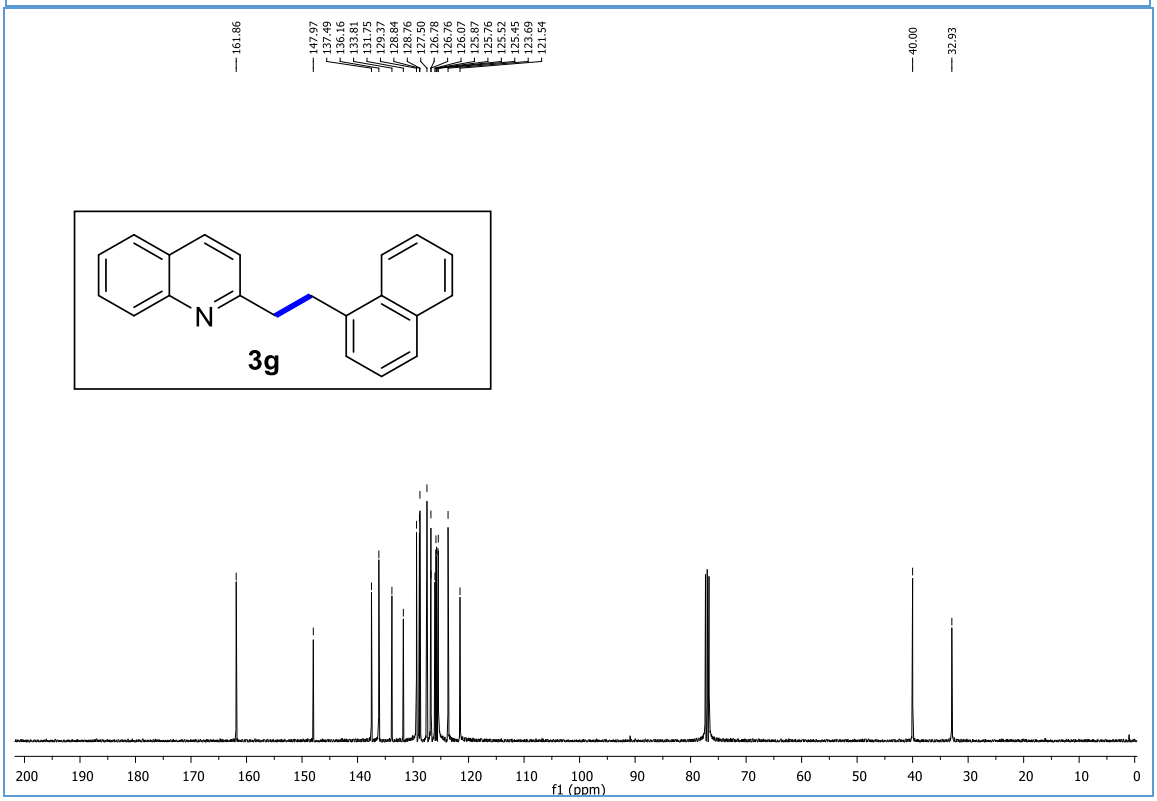
3.5
3.0
2.5
2.0
1.5
1.0
0.5
0.0
f1 (ppm)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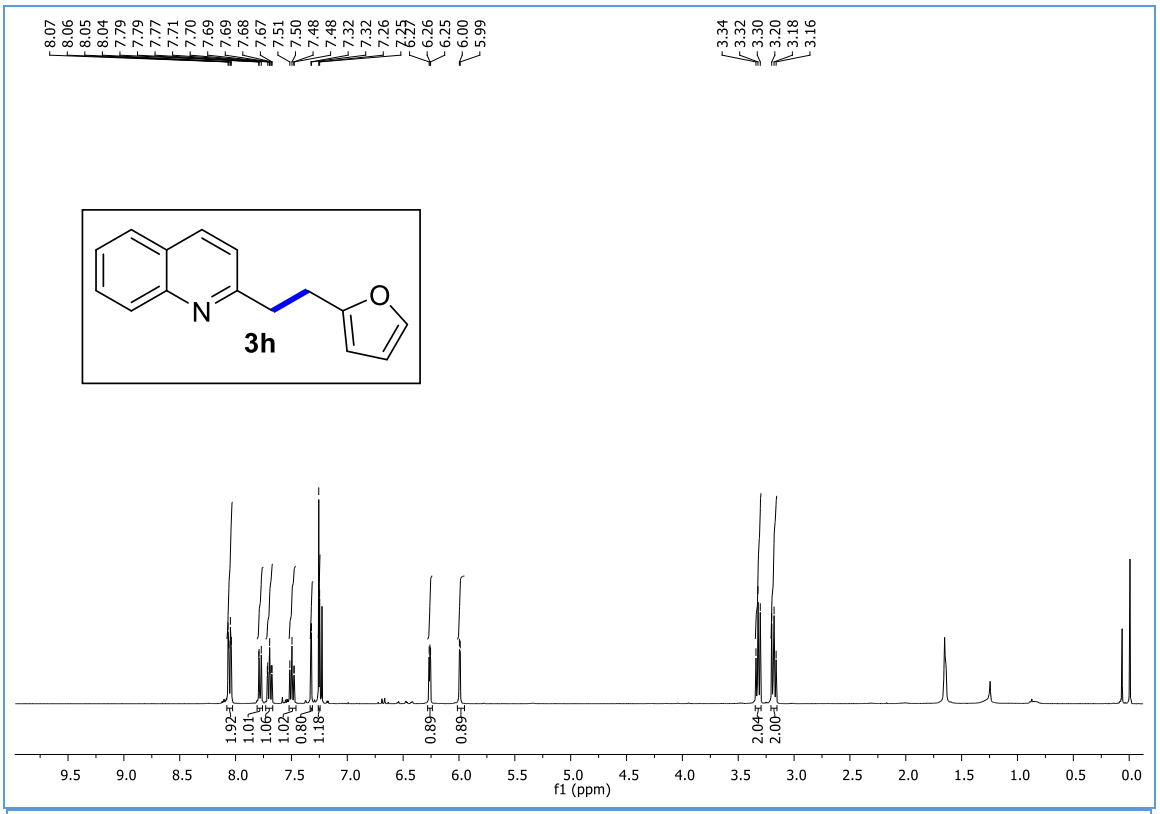
131.75
129.37
128.84
128.76
127.50
126.78
126.76
126.07
125.87
125.76
125.52
125.45
123.69
121.54
40.00
32.93
N
3g
200
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
f1 (ppm)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8.06
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7.79
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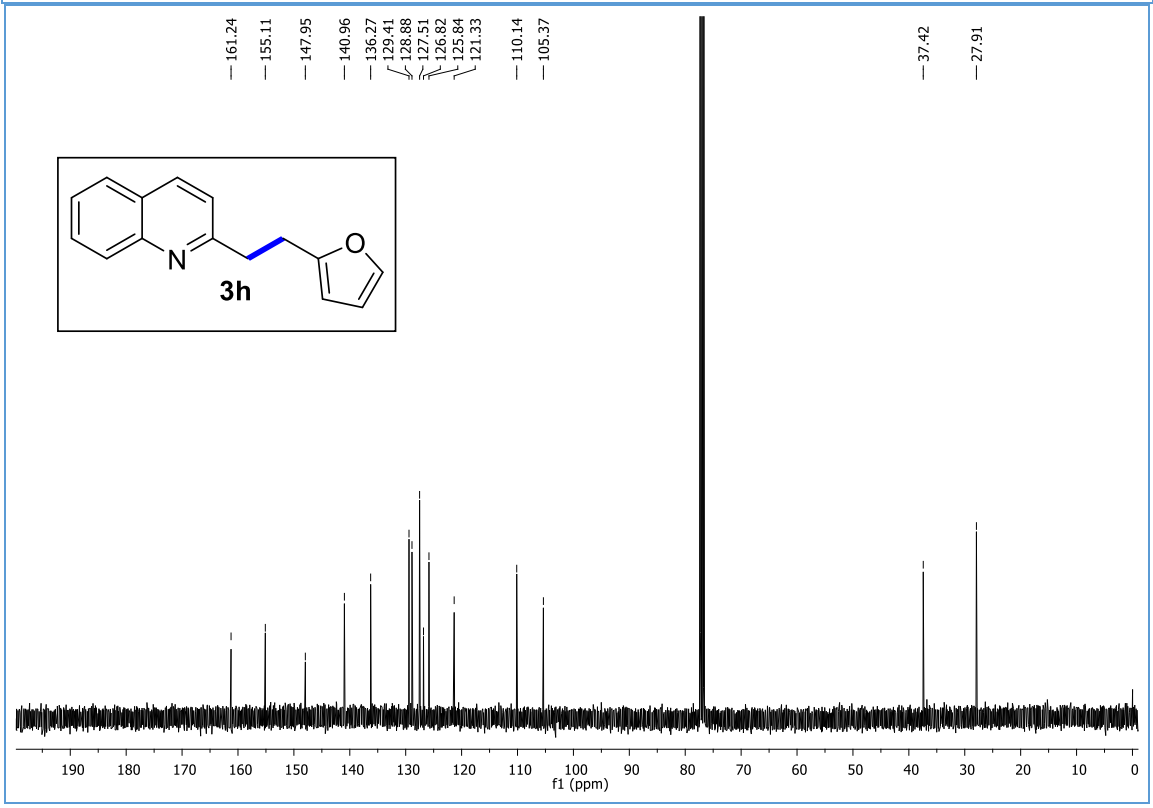
7.69
7.69
7.68
7.67
7.51
7.50
7.48
7.48
7.32
7.32
7.26
6.27
6.26
6.25
6.00
5.99
3.34
3.32
3.30
3.20
3.18
3.16
N
O
3h
1.92
1.01
1.05
1.02
0.80
1.18
0.89
0.89
2.04
2.00
f1 (ppm)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155.11
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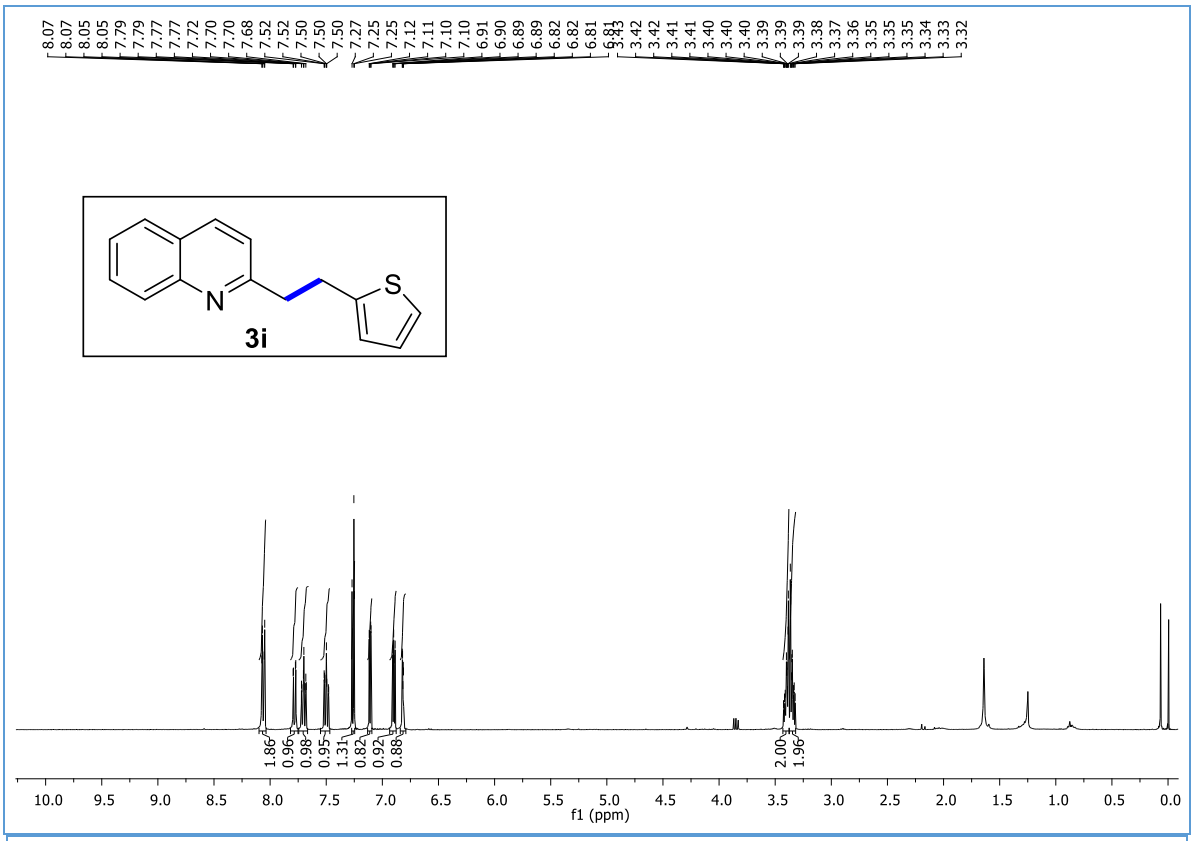
140.96
136.27
129.41
128.88
127.51
126.82
125.84
121.33
110.14
105.37
37.42
27.91
N
O
3h
f1 (ppm)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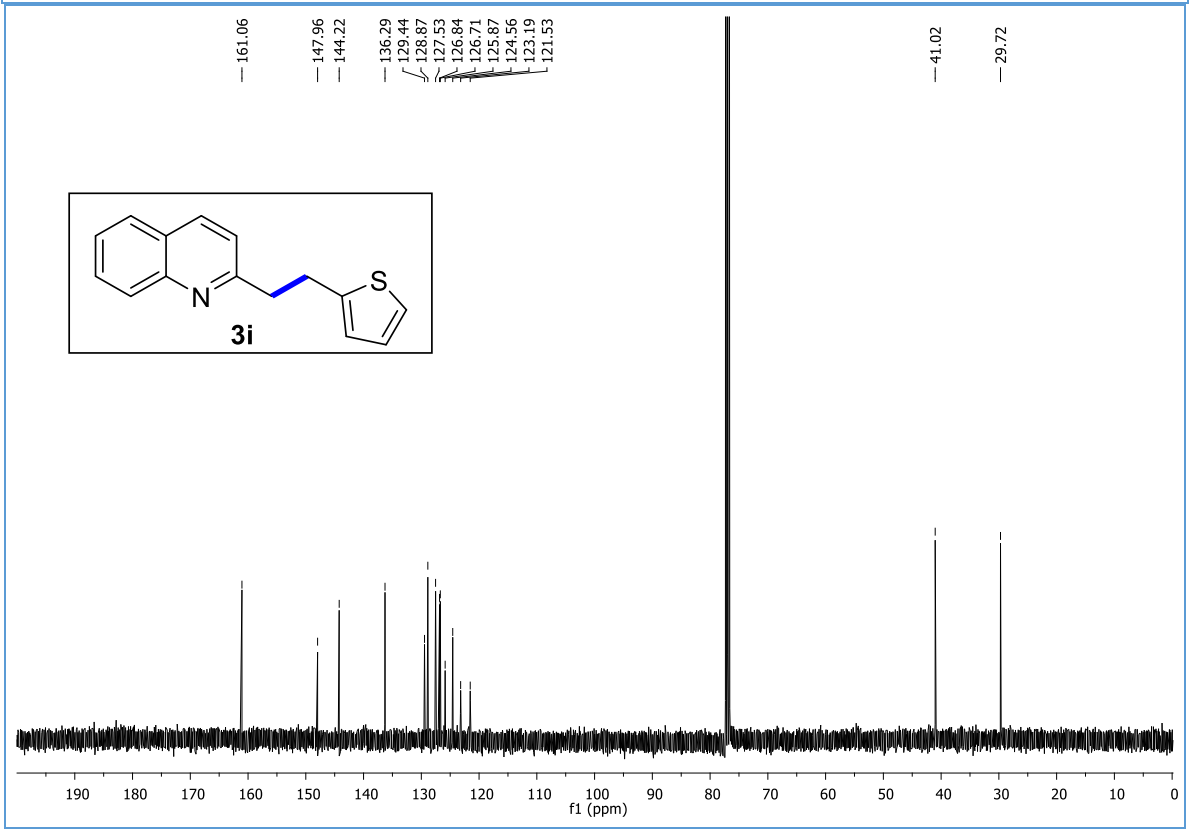
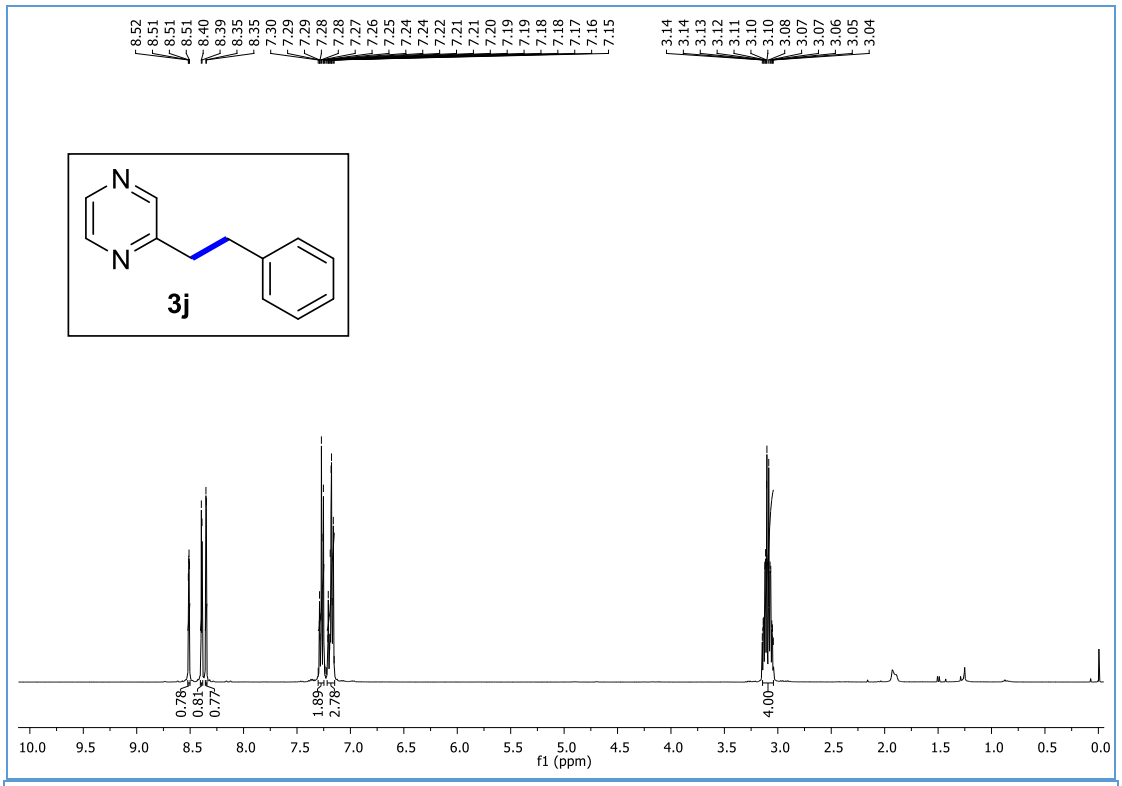
N
S
3i
1.86 0.96 0.98 0.95 1.31 0.82 0.92 0.88 2.00 1.96
10.0 9.5 9.0 8.5 8.0 7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 0.5 0.0
f1 (ppm)


161.06 147.96 144.22 136.29 129.44 128.87 127.53 126.84 126.71 125.87 124.56 123.19 121.53 41.02 29.72
N
S
3i
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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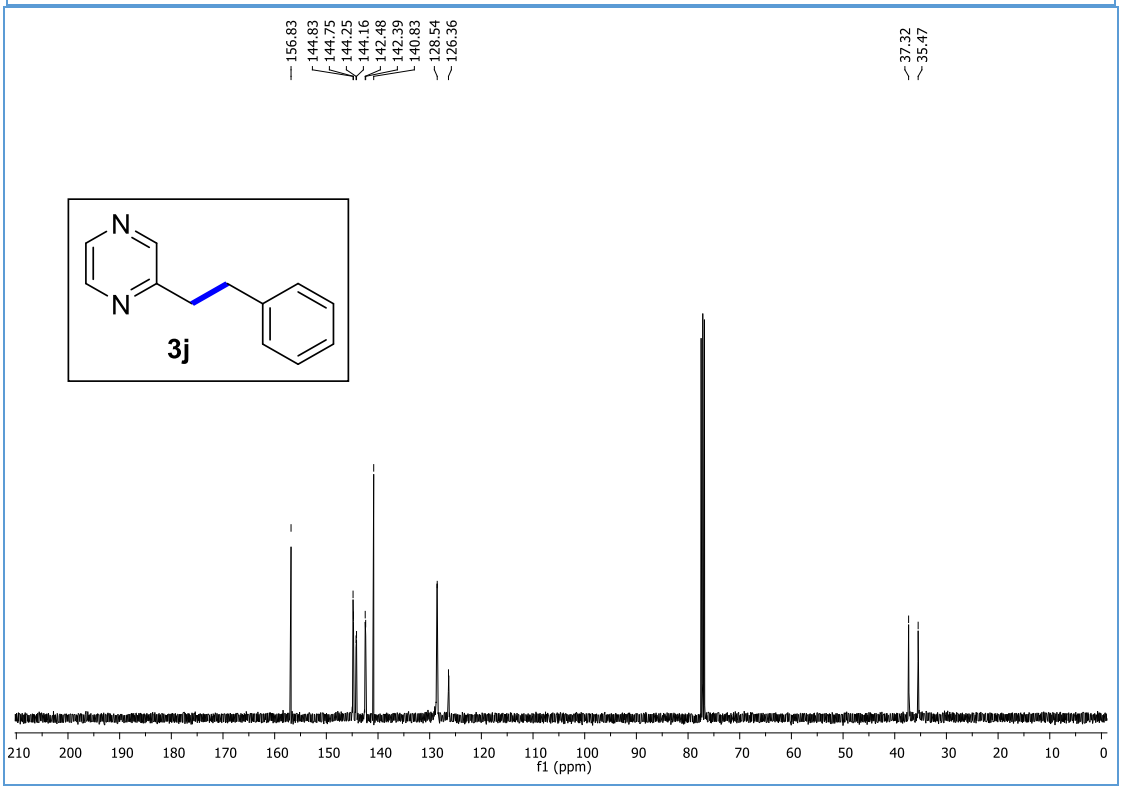
7.28
7.27
7.26
7.25
7.24
7.24
7.22
7.21
7.21
7.20
7.19
7.19
7.18
7.18
7.17
7.16
7.15
3.14
3.14
3.13
3.12
3.11
3.10
3.10
3.08
3.07
3.07
3.06
3.05
3.04
N
N
3j
0.78
0.81
0.77
1.89
2.78
4.00
10.0 9.5 9.0 8.5 8.0 7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 0.5 0.0
f1 (ppm)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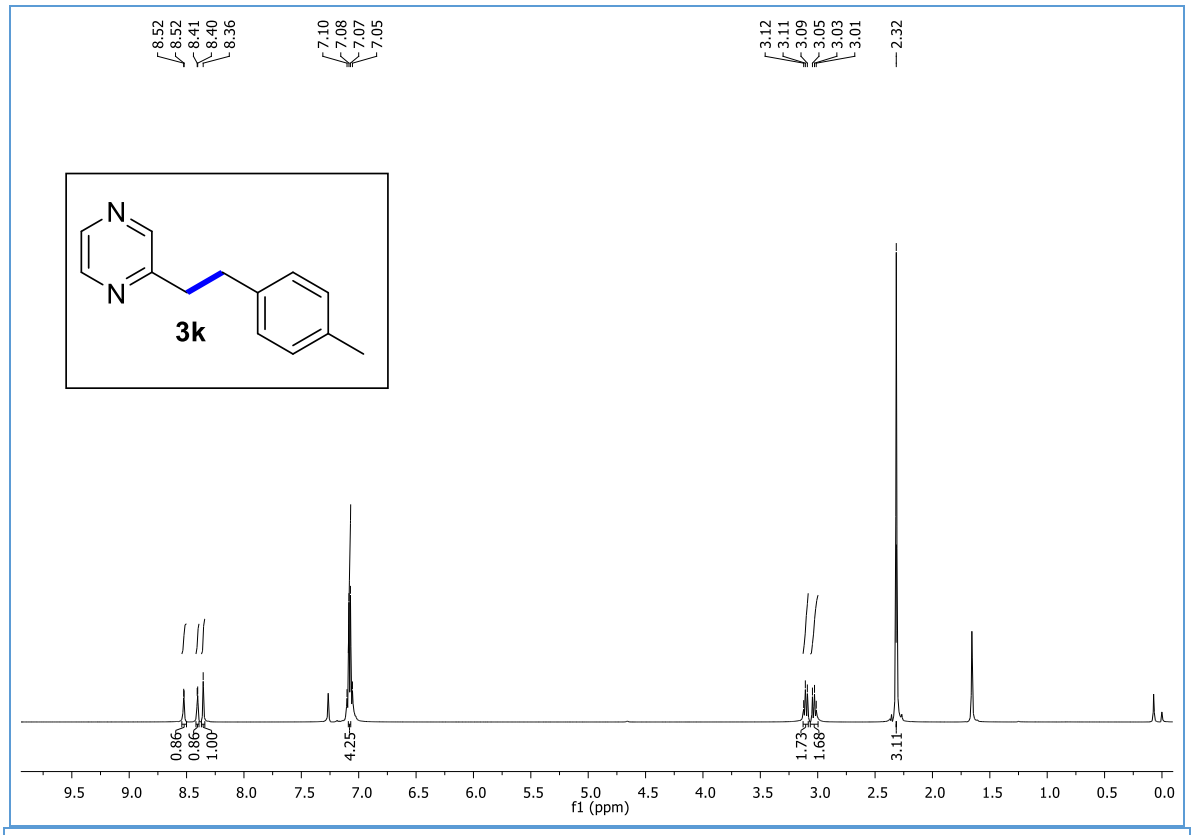
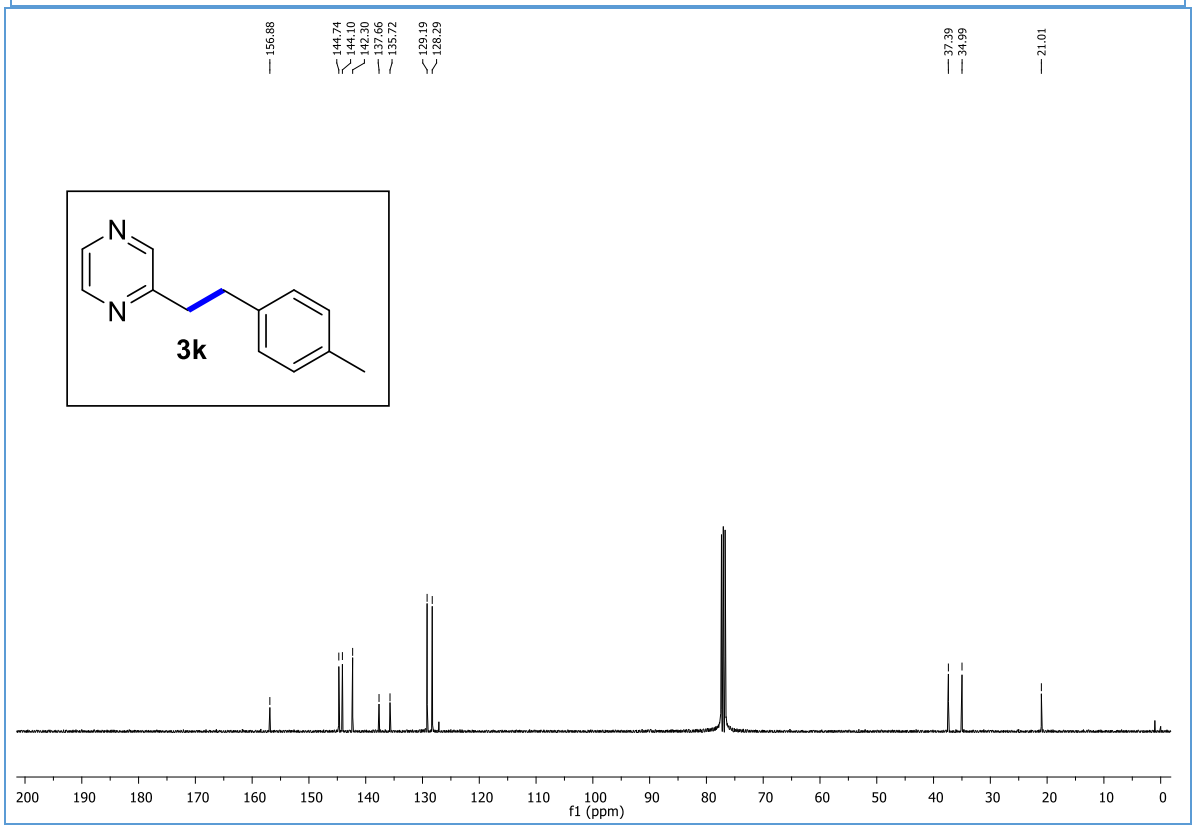
144.83
144.75
144.25
144.16
142.48
142.39
140.83
128.54
126.36
37.32
35.47
N
N
3j
210 200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0
f1 (ppm)


8.52
8.52
8.41
8.40
8.36
7.10
7.08
7.07
7.05
3.12
3.11
3.09
3.05
3.03
3.01
2.32
N
N
3k
0.86
0.86
1.00
4.25
1.73
1.68
3.11
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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144.74
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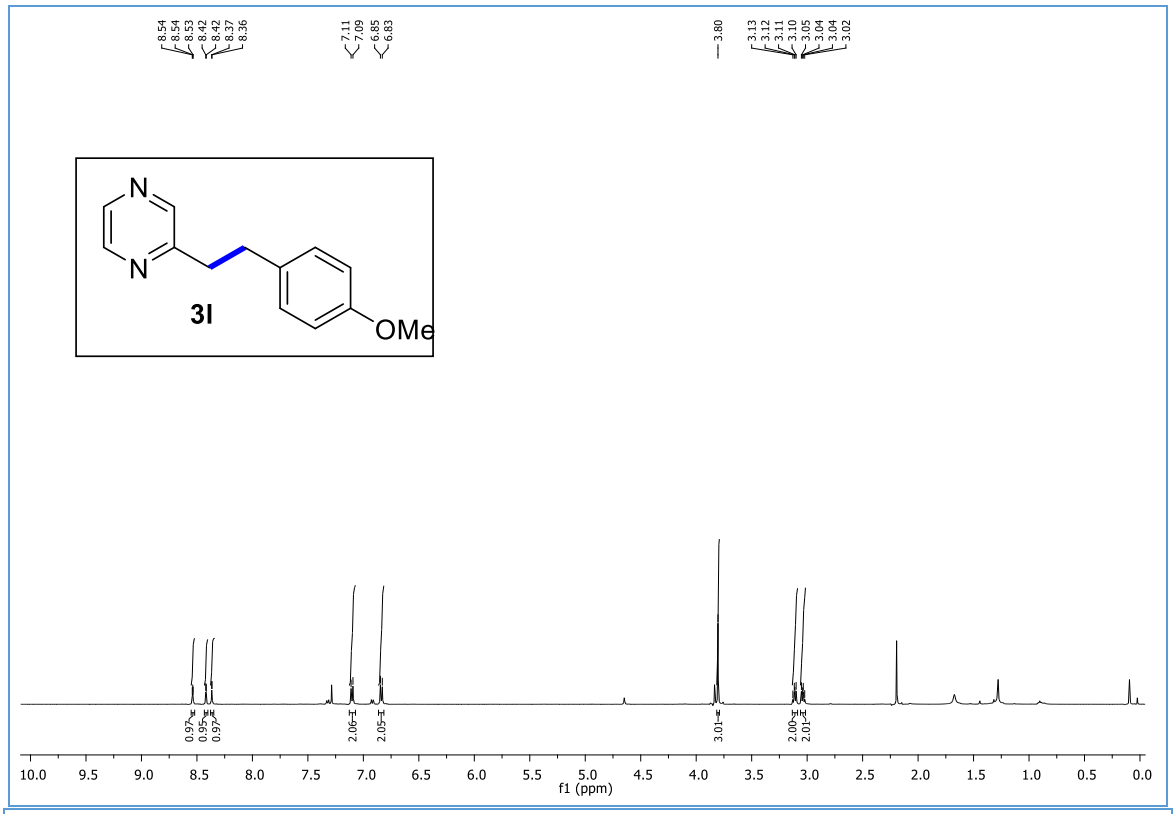
128.29
37.39
34.99
21.01
N
N
3k
200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0
f1 (ppm)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8.54
8.53
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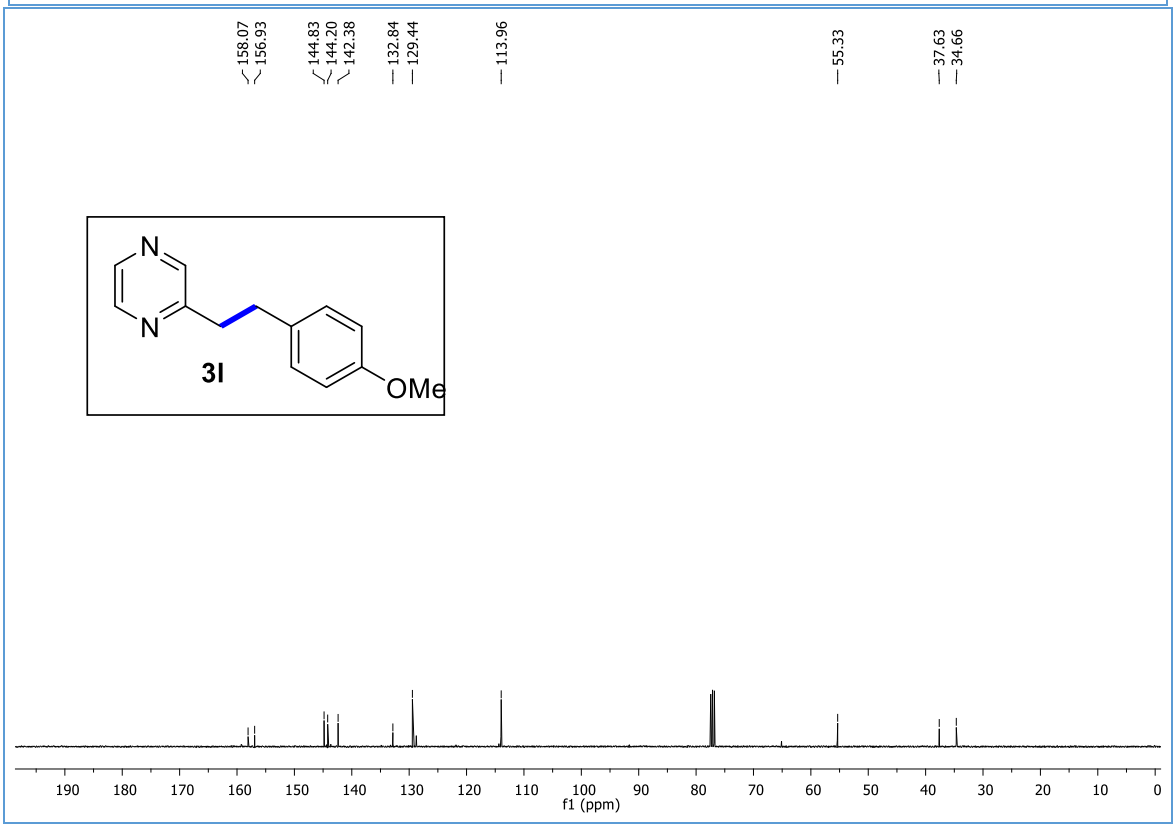
8.42
8.37
8.36
7.11
7.09
6.85
6.83
3.80
3.13
3.12
3.11
3.10
3.05
3.04
3.04
3.02
N
N
3l
OMe
0.97
0.95
0.97
2.06
2.05
3.01
2.00
2.01
10.0 9.5 9.0 8.5 8.0 7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 0.5 0.0
f1 (ppm)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156.93
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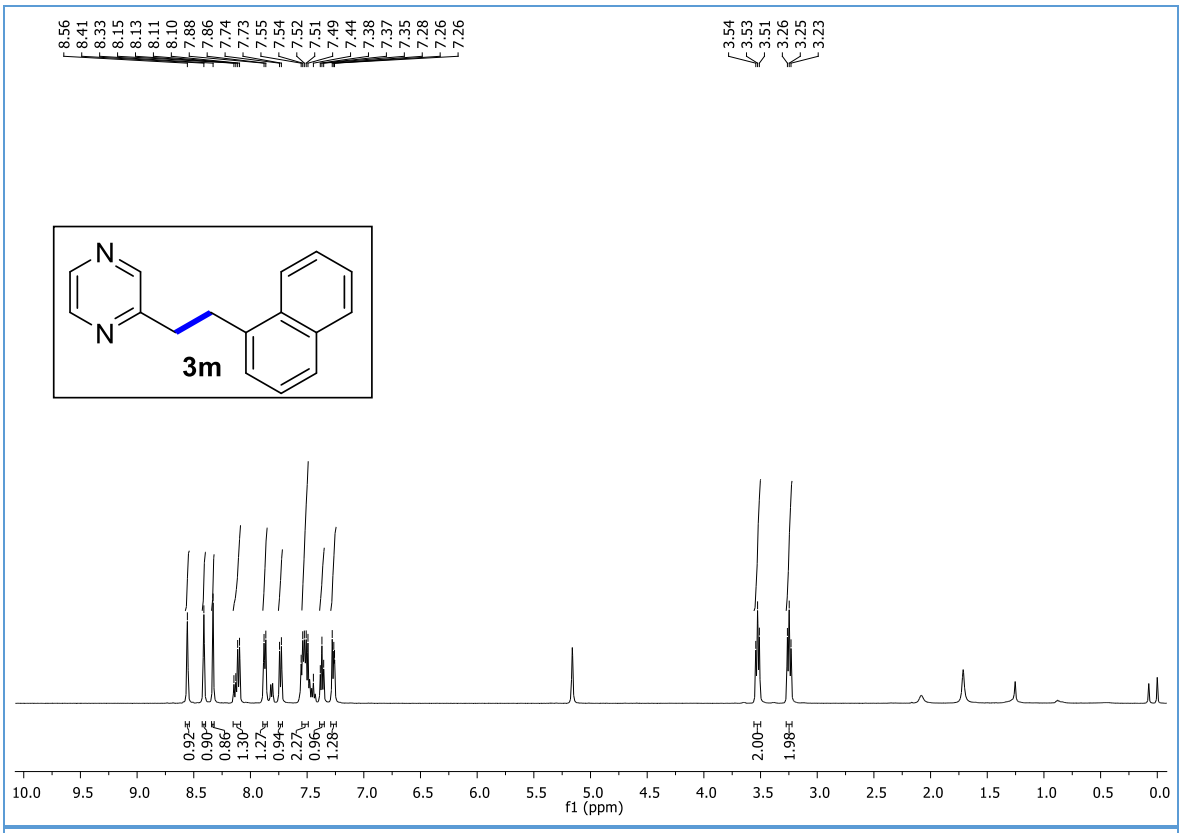
142.38
132.84
129.44
113.96
55.33
37.63
34.66
N
N
3l
OMe
190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0
f1 (ppm)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7.74
7.73
7.55
7.54
7.52
7.51
7.49
7.44
7.38
7.37
7.35
7.28
7.26
7.26
3.54
3.53
3.51
3.26
3.25
3.23
N
N
3m
0.92
0.90
0.86
1.30
1.27
0.94
2.27
0.96
1.28
2.00
1.98
10.0
9.5
9.0
8.5
8.0
7.5
7.0
6.5
6.0
5.5
5.0
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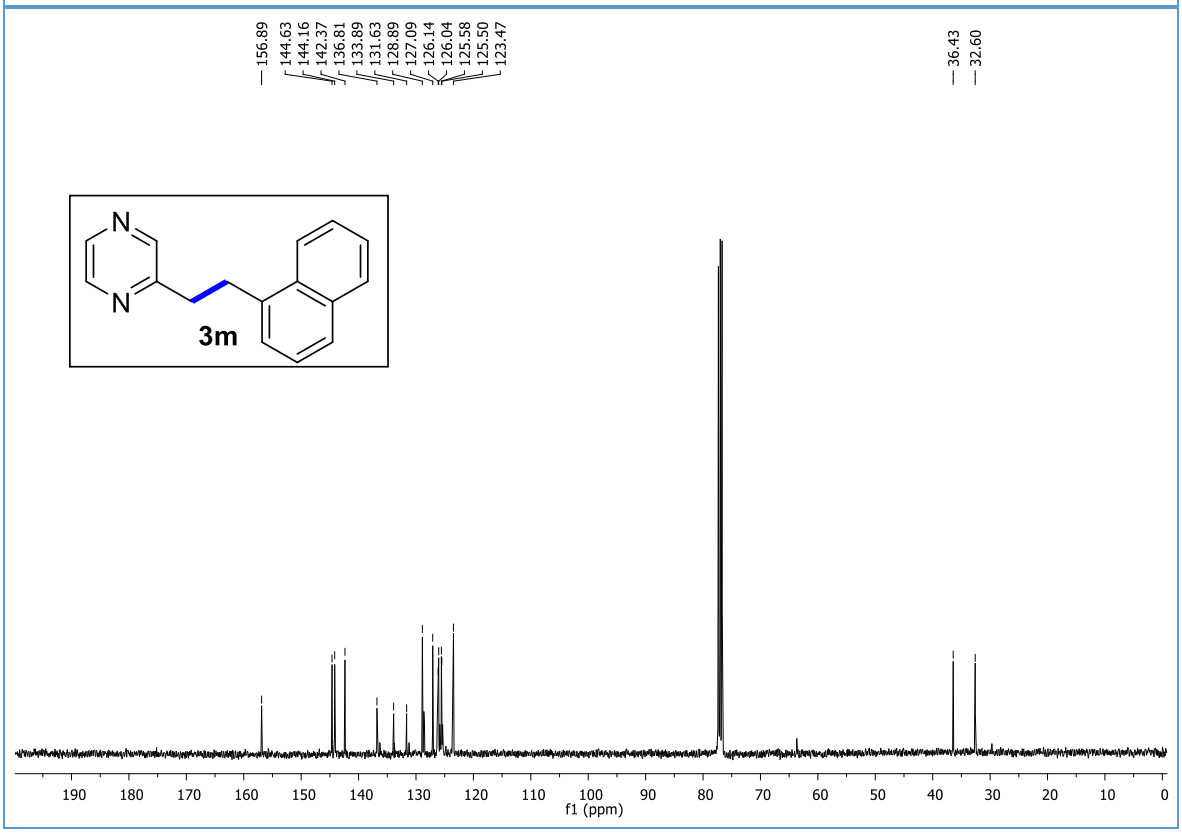
4.0
3.5
3.0
2.5
2.0
1.5
1.0
0.5
0.0
f1 (ppm)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128.89
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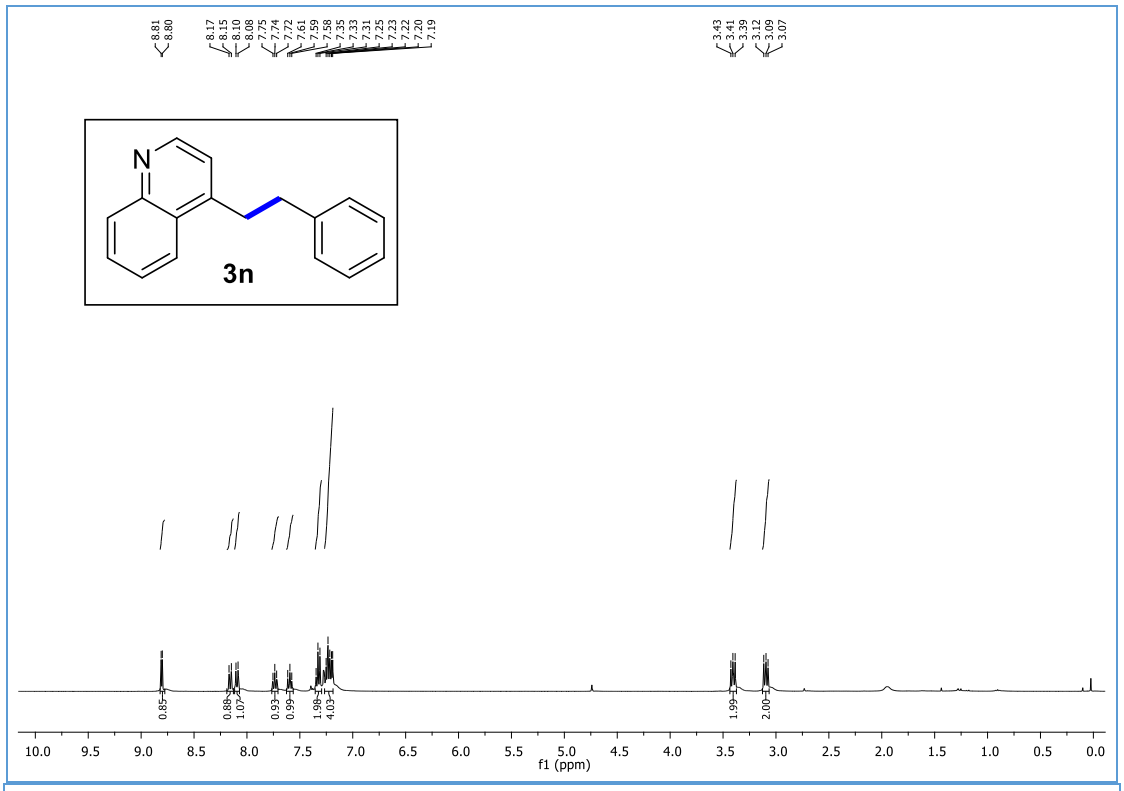
126.14
126.04
125.58
125.50
123.47
36.43
32.60
N
N
3m
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
f1 (ppm)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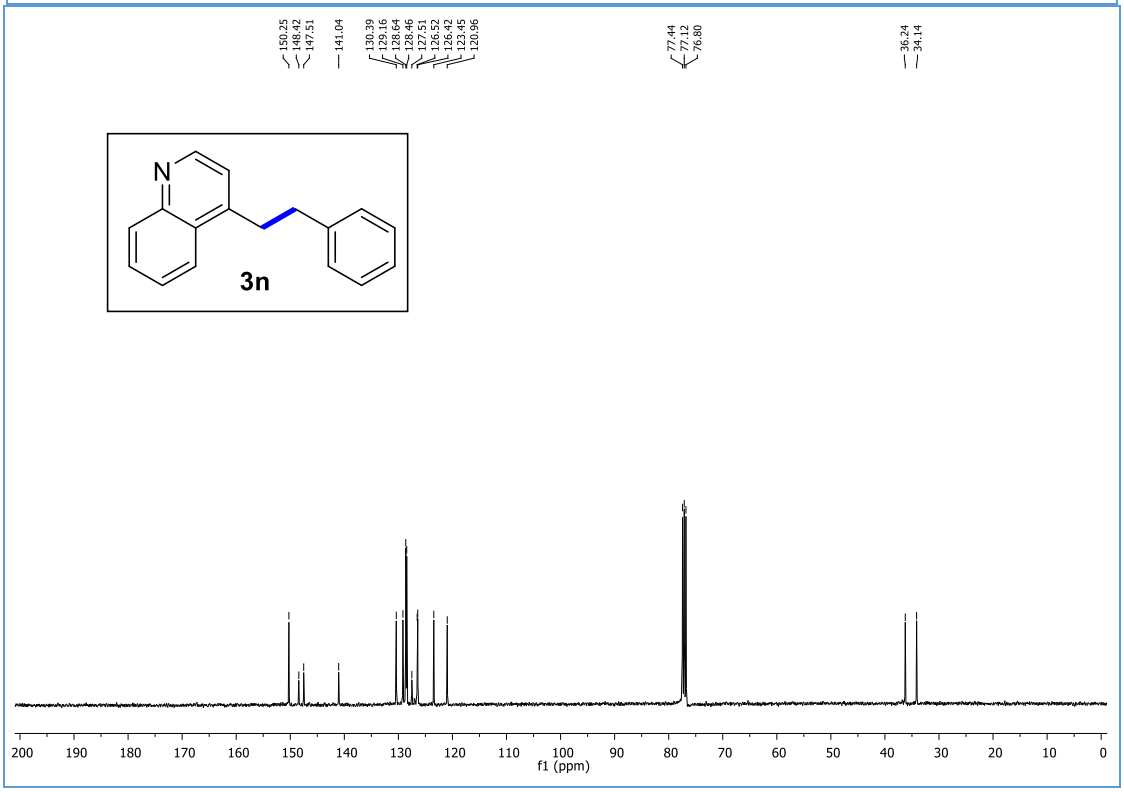
7.35
7.33
7.31
7.25
7.23
7.22
7.20
7.19
3.43
3.41
3.39
3.12
3.09
3.07
N
3n
0.85
0.88
1.07
0.93
0.99
1.98
4.03
1.99
2.00
10.0 9.5 9.0 8.5 8.0 7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 0.5 0.0
f1 (ppm)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129.16
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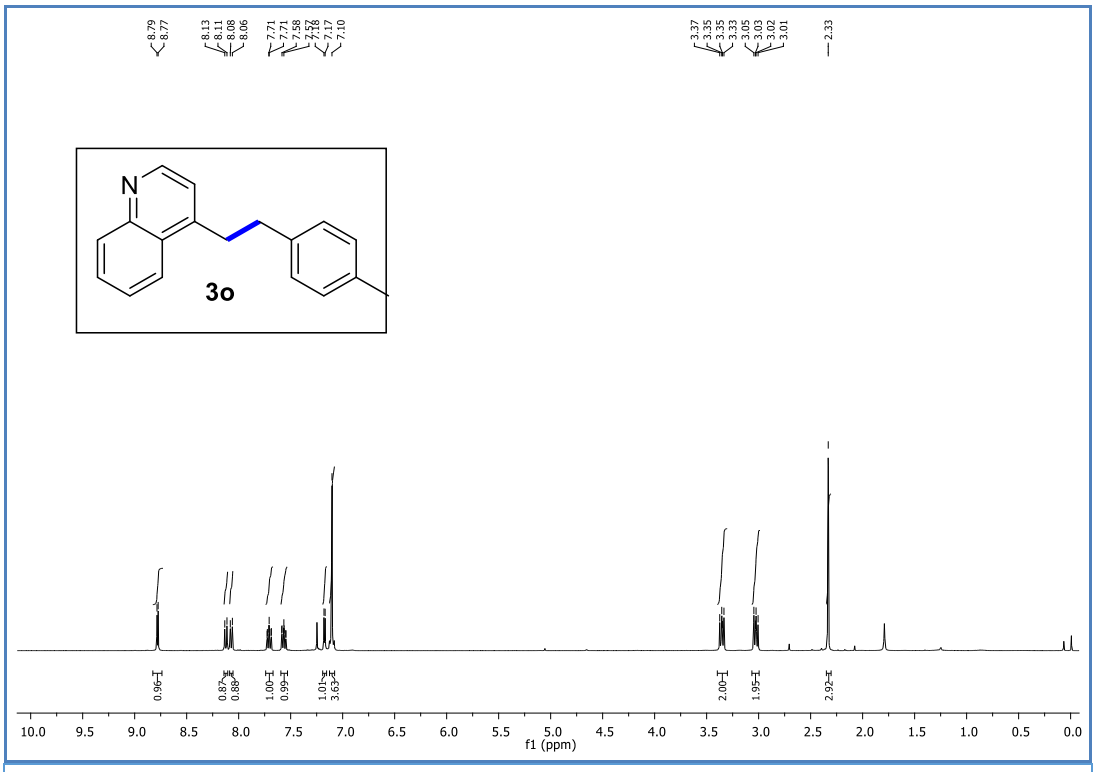
128.46
127.51
126.52
126.42
123.45
120.96
77.44
77.12
76.80
36.24
34.14
N
3n
200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0
f1 (ppm)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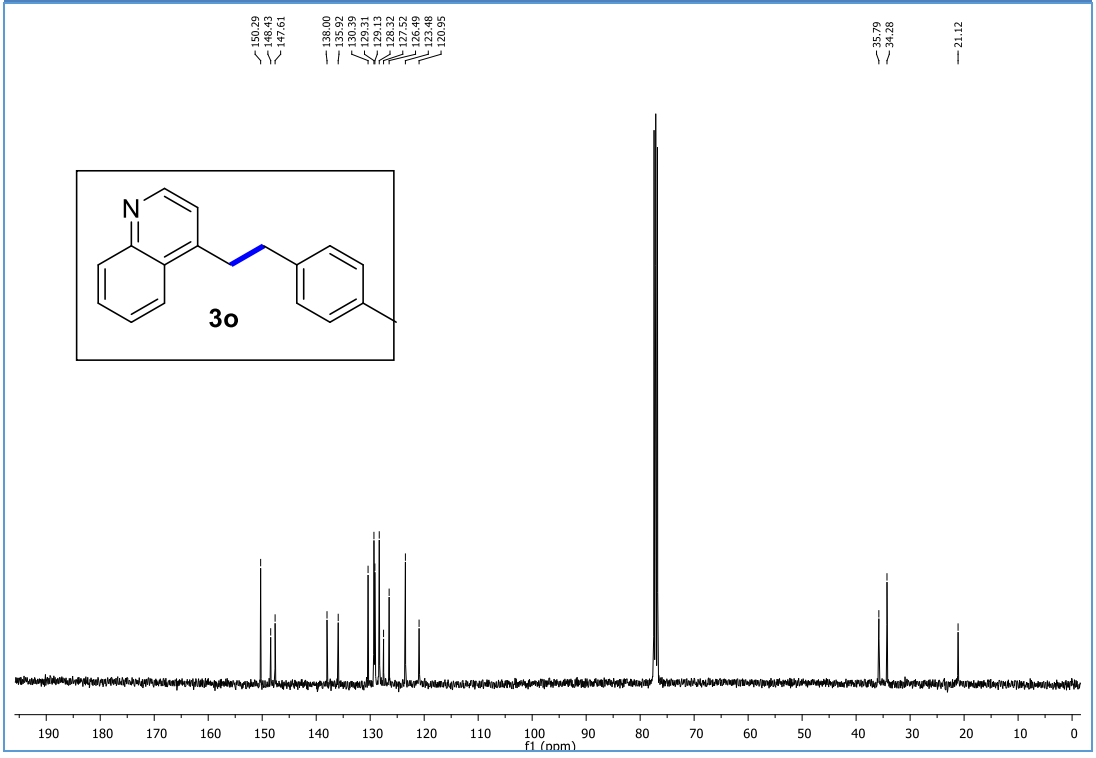
7.71
7.58
7.56
7.17
7.10
3.37
3.35
3.35
3.33
3.05
3.03
3.02
3.01
2.33
N
3o
0.96
0.87
0.88
1.00
0.99
1.01
3.63
2.00
1.95
2.92
10.0 9.5 9.0 8.5 8.0 7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 0.5 0.0
f1 (ppm)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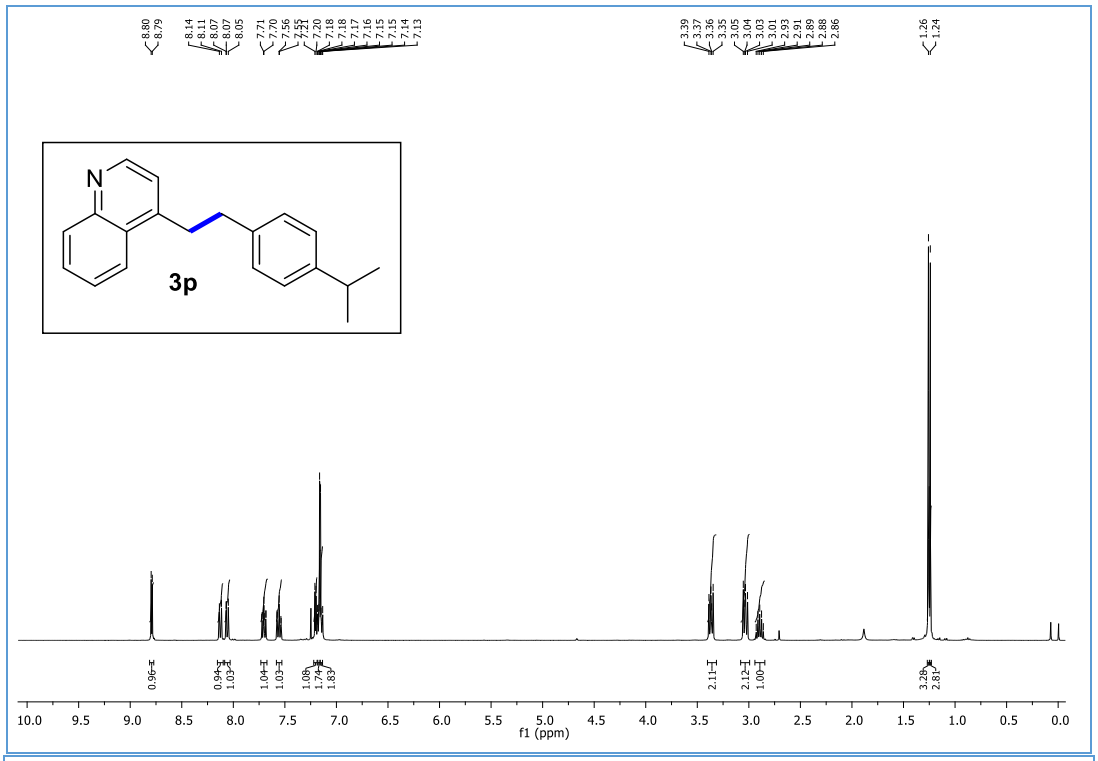
130.39
129.31
129.13
128.32
127.52
126.49
123.48
120.95
35.79
34.28
21.12
N
3o
190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0
f1 (ppm)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8.07
8.05
7.71
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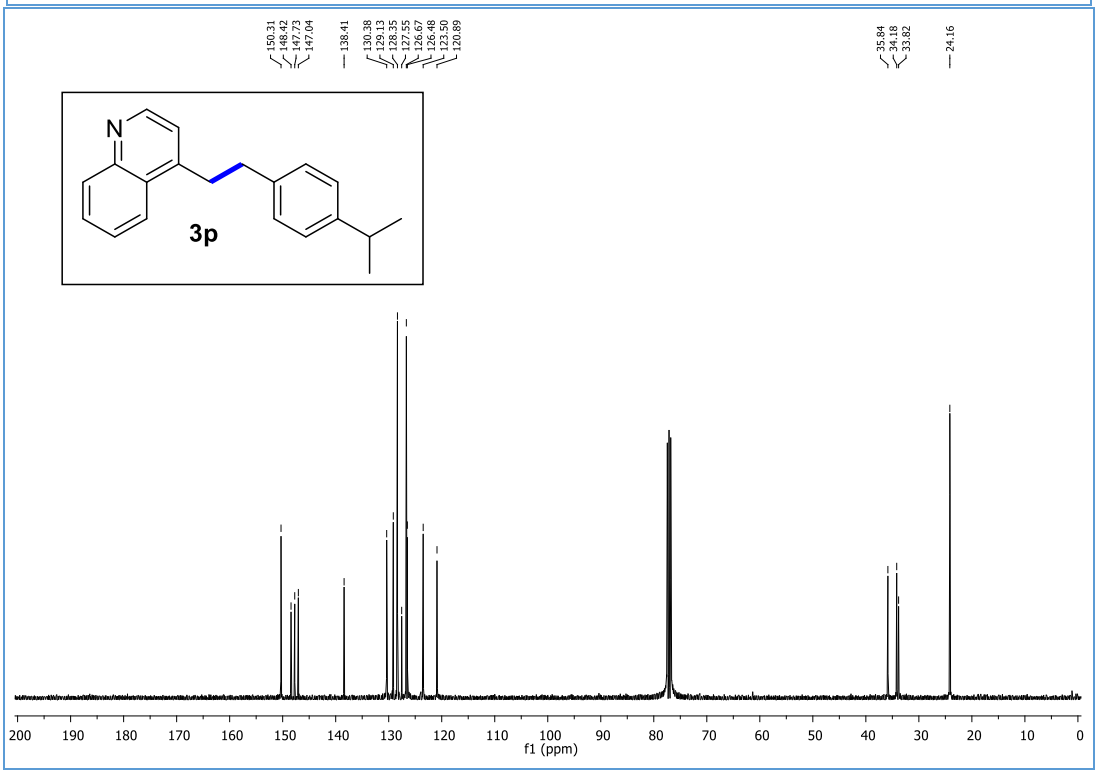
7.55
7.51
7.20
7.18
7.18
7.17
7.16
7.15
7.15
7.14
7.13
3.39
3.37
3.36
3.35
3.05
3.04
3.03
3.01
2.93
2.91
2.89
2.88
2.86
1.26
1.24
N
3p
0.96
0.94
1.03
1.04
1.03
1.08
1.74
1.83
2.11
2.12
1.00
3.28
2.81
10.0 9.5 9.0 8.5 8.0 7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 0.5 0.0
f1 (ppm)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148.42
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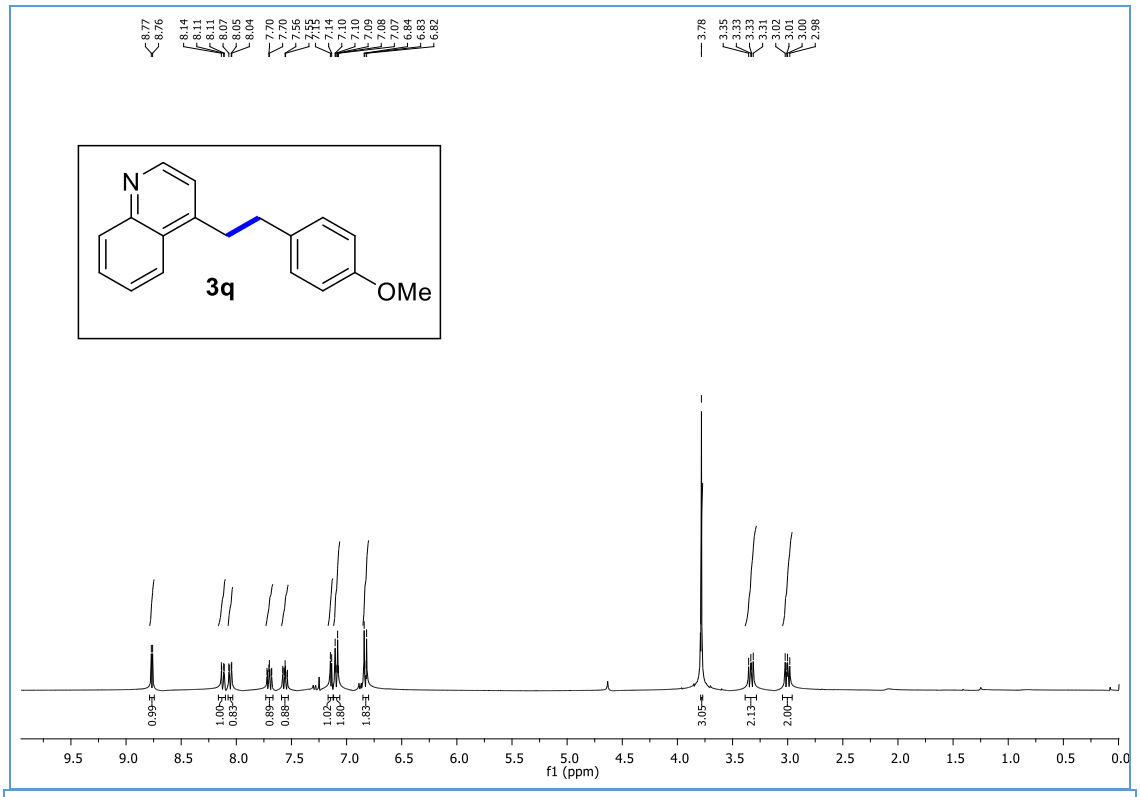
147.04
138.41
130.38
129.13
128.35
127.55
126.67
126.48
123.50
120.89
35.84
34.18
33.82
24.16
N
3p
200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0
f1 (ppm)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8.76
8.14
8.11
8.11
8.07
8.05
8.04
7.70
7.70
7.56
7.55
7.15
7.14
7.10
7.09
7.08
7.07
6.84
6.83
6.82
3.78
3.35
3.33
3.33
3.31
3.02
3.01
3.00
2.98
N
3q
OMe
0.99
1.00
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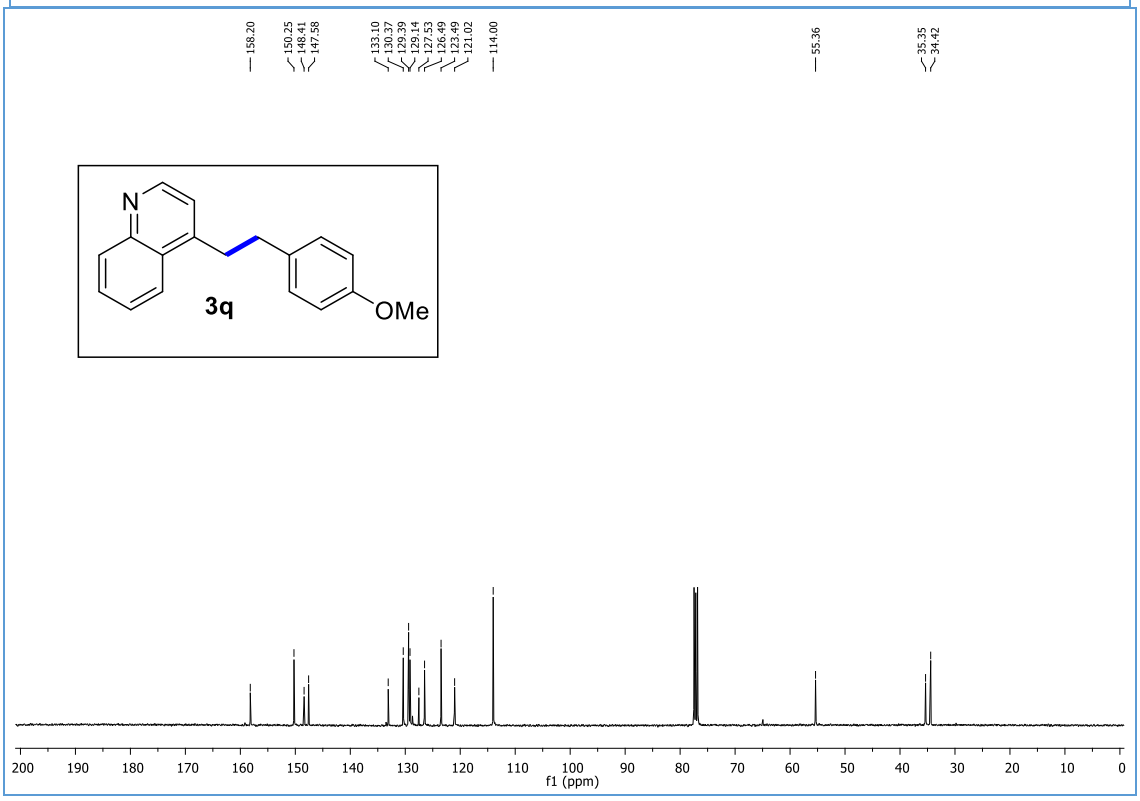
0.89
0.88
1.02
1.80
1.83
3.05
2.13
2.00
f1 (ppm)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150.25
148.41
147.58
133.10
130.37
129.39
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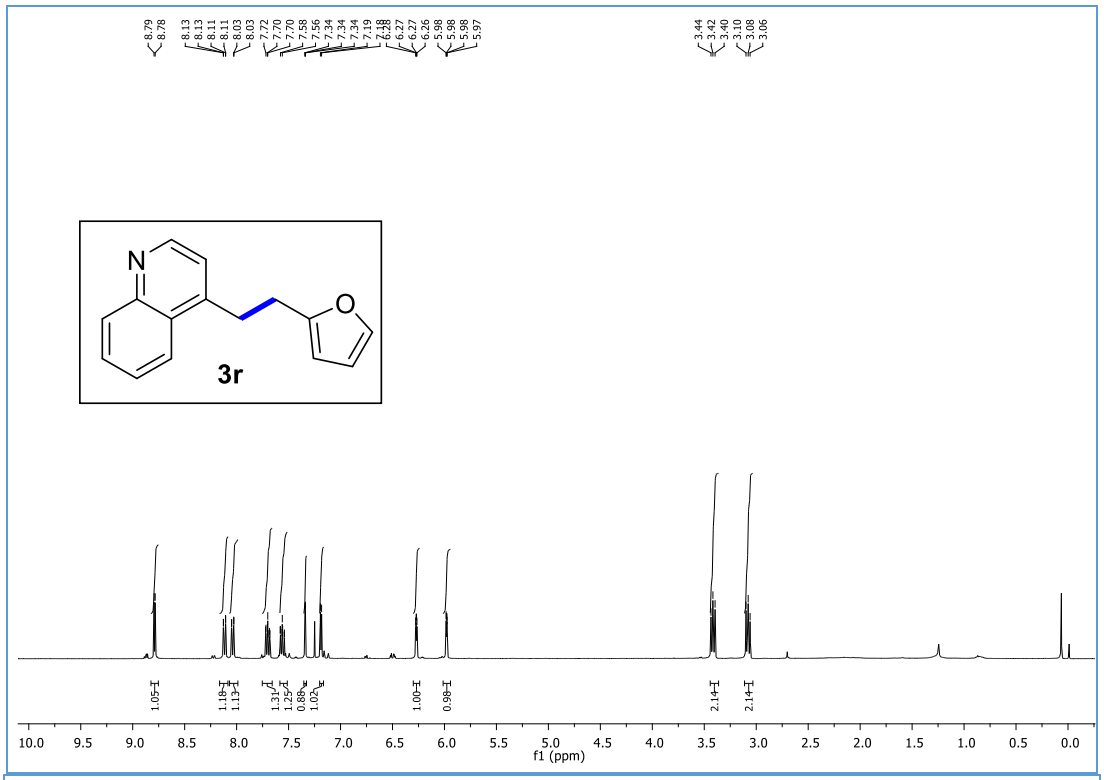
123.49
121.02
114.00
55.36
35.35
34.42
N
3q
OMe
f1 (ppm)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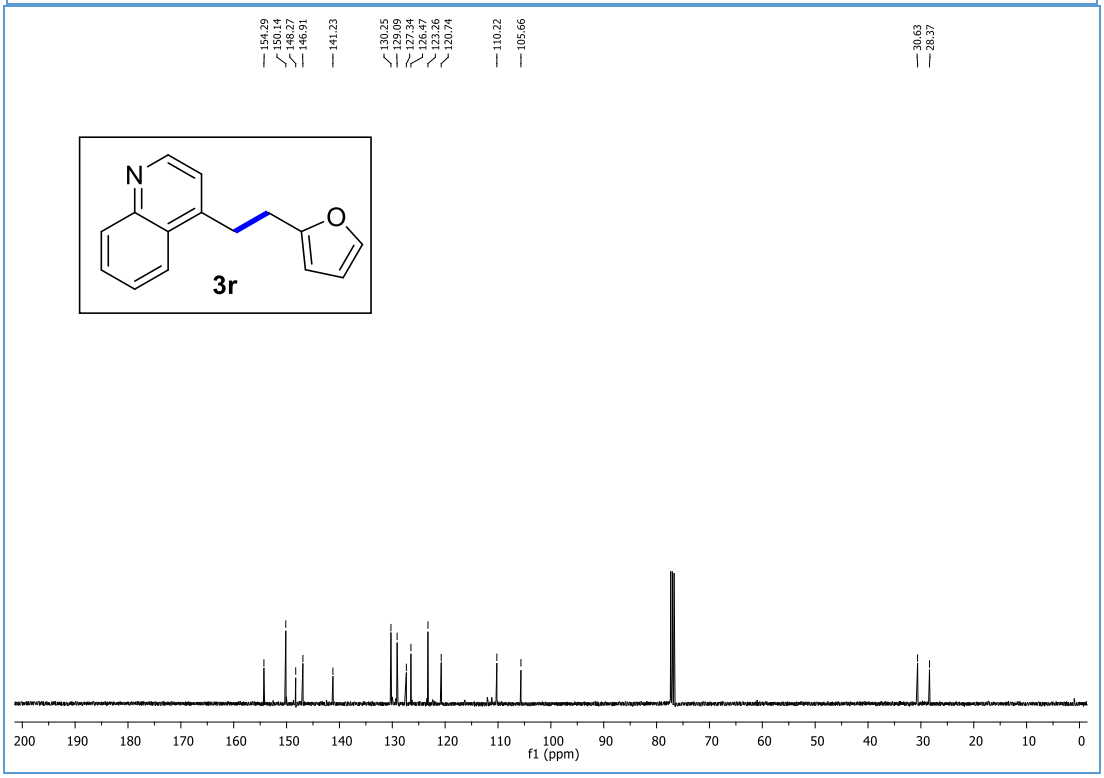
8.78
8.13
8.13
8.11
8.11
8.03
8.03
7.72
7.70
7.70
7.58
7.56
7.34
7.34
7.34
7.19
6.28
6.27
6.27
6.26
5.98
5.98
5.98
5.97
3.44
3.42
3.40
3.10
3.08
3.06
N
O
3r
1.05
1.10
1.13
1.31
1.25
0.88
1.02
1.00
0.99
2.14
2.14
10.0 9.5 9.0 8.5 8.0 7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 0.5 0.0
f1 (ppm)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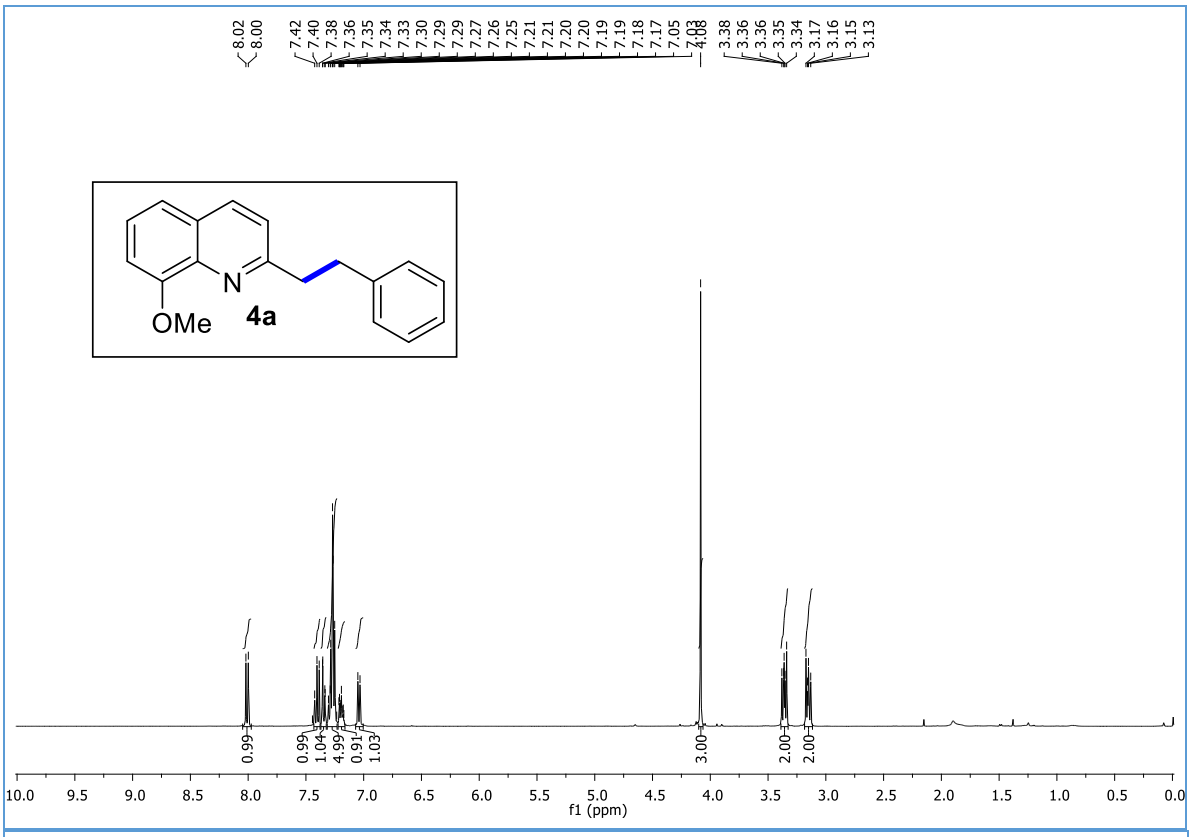
150.14
148.27
146.91
141.23
130.25
129.09
127.34
126.47
123.26
120.74
110.22
105.66
30.63
28.37
N
O
3r
200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0
f1 (ppm)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8.00
7.42
7.40
7.38
7.36
7.35
7.34
7.33
7.30
7.29
7.29
7.27
7.26
7.25
7.21
7.21
7.20
7.20
7.19
7.19
7.18
7.17
7.05
7.03
4.08
3.38
3.36
3.36
3.35
3.34
3.17
3.16
3.15
3.13
N
OMe
4a
0.99
0.99
1.04
4.99
0.91
1.03
3.00
2.00
2.00
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
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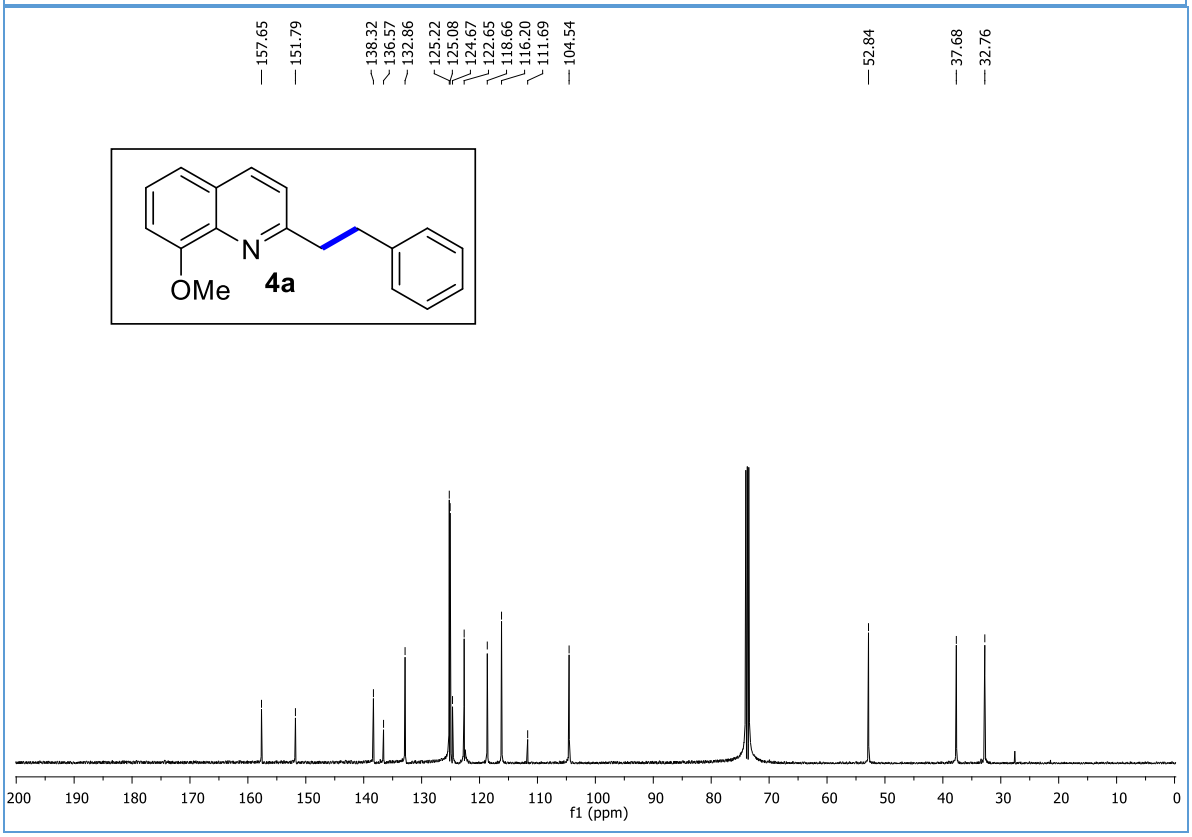
0.0
f1 (ppm)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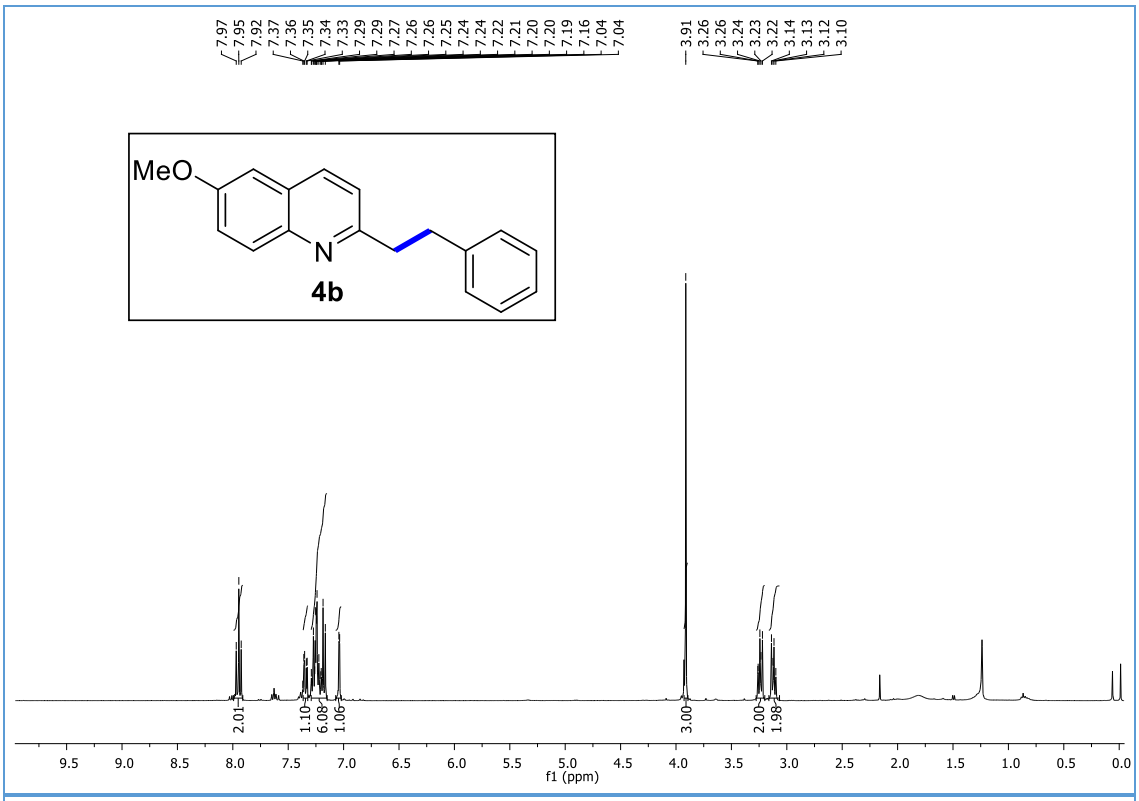
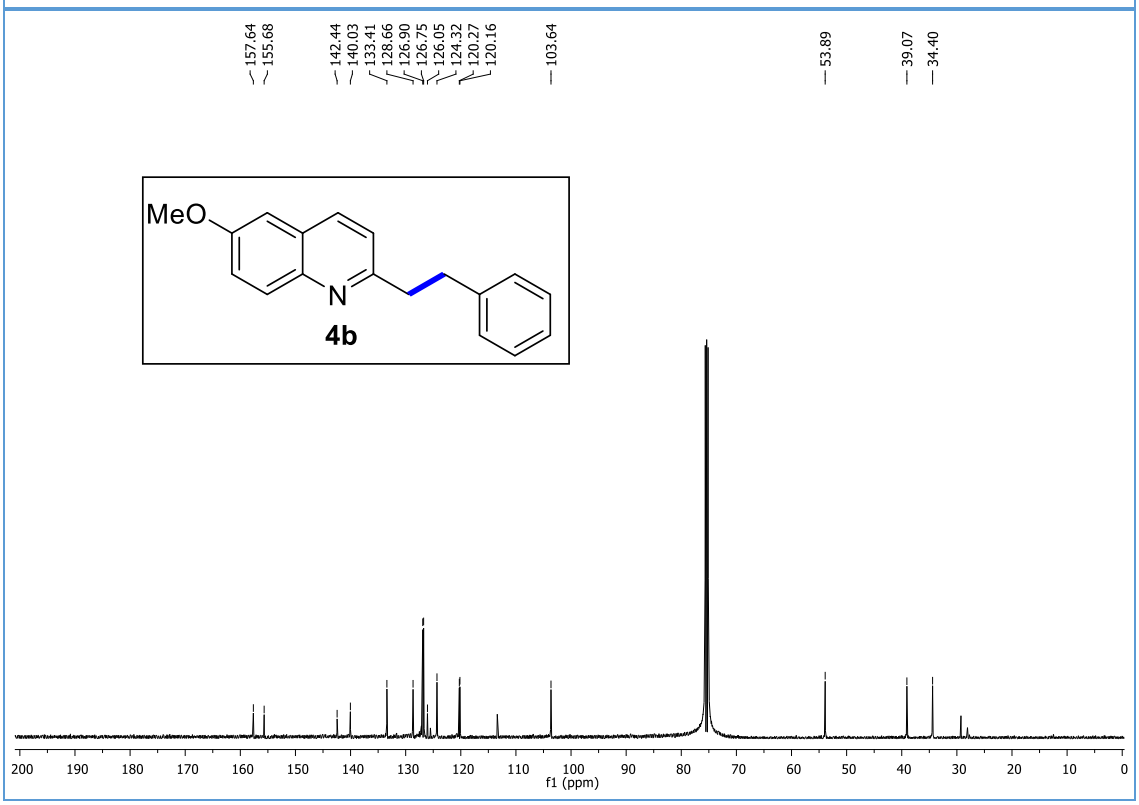
138.32
136.57
132.86
125.22
125.08
124.67
122.65
118.66
116.20
111.69
104.54
52.84
37.68
32.76
N
OMe
4a
200
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
f1 (ppm)


7.97
7.95
7.92
7.37
7.36
7.35
7.34
7.33
7.29
7.29
7.27
7.26
7.26
7.25
7.24
7.24
7.22
7.21
7.20
7.20
7.19
7.16
7.04
7.04
3.91
3.26
3.26
3.24
3.23
3.22
3.14
3.13
3.12
3.10
MeO
N
4b
2.01
1.10
6.08
1.06
3.00
2.00
1.98
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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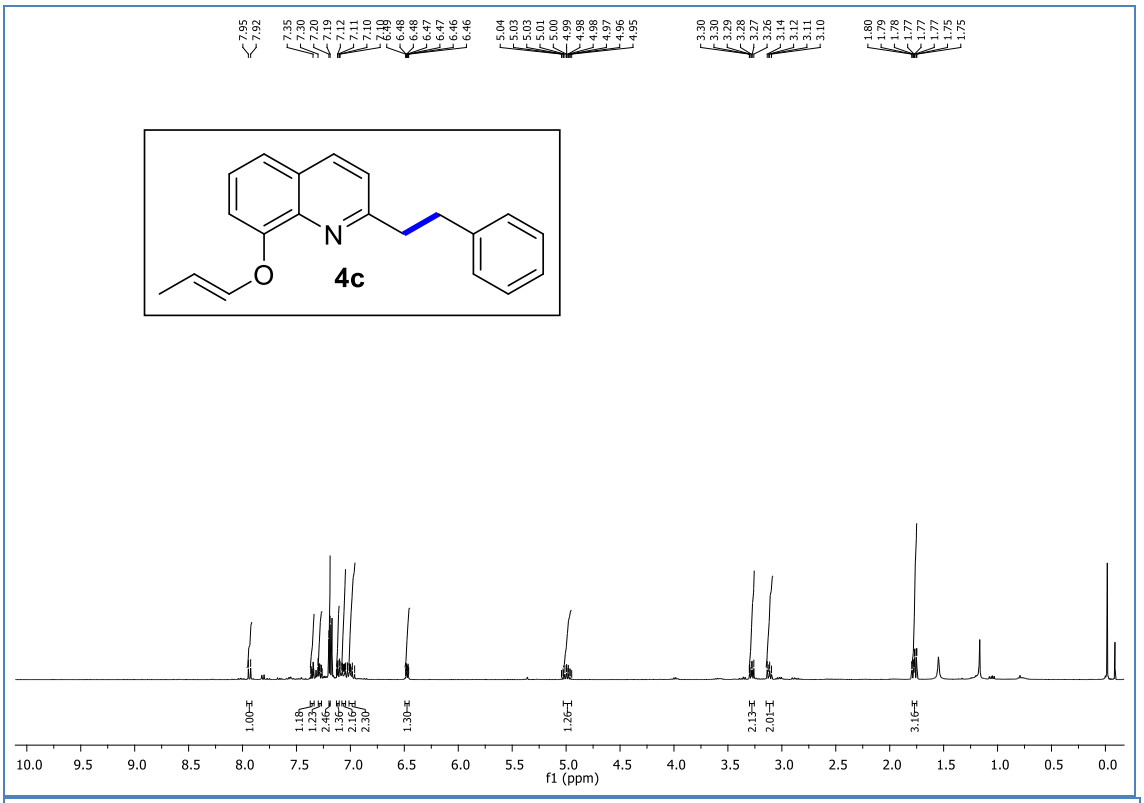
140.03
133.41
128.66
126.90
126.75
126.05
124.32
120.27
120.16
103.64
53.89
39.07
34.40
MeO
N
4b
200
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
f1 (ppm)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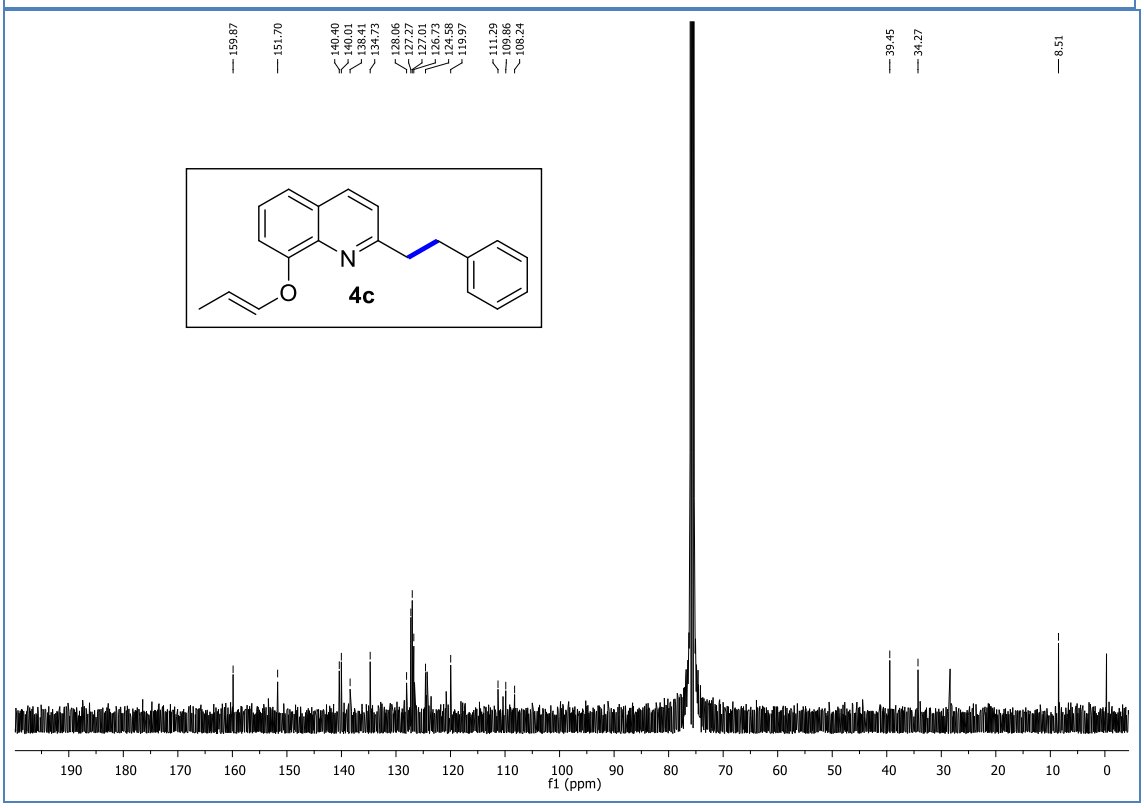
7.92
7.35
7.30
7.20
7.19
7.12
7.11
7.10
6.49
6.48
6.48
6.47
6.47
6.46
6.46
5.04
5.03
5.03
5.01
5.00
4.99
4.98
4.98
4.97
4.96
4.95
3.30
3.30
3.29
3.28
3.27
3.26
3.14
3.12
3.11
3.10
1.80
1.79
1.78
1.77
1.77
1.77
1.75
1.75
N
O
4c
1.00
1.18
1.23
2.46
1.36
2.16
2.30
1.30
1.26
2.13
2.01
3.16
10.0
9.5
9.0
8.5
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)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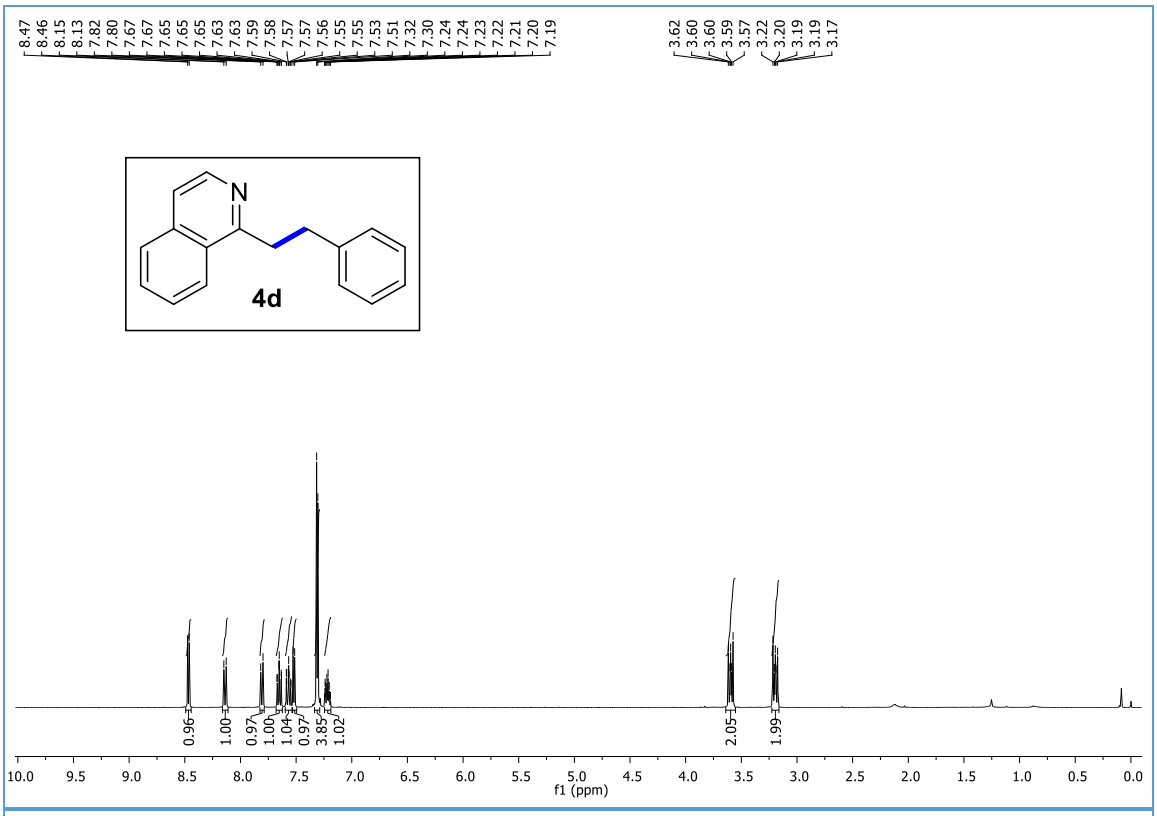
134.73
128.06
127.27
127.01
126.73
124.58
119.97
111.29
109.86
108.24
39.45
34.27
8.51
N
O
4c
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
f1 (ppm)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8.46
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7.80
7.67
7.67
7.65
7.65
7.65
7.63
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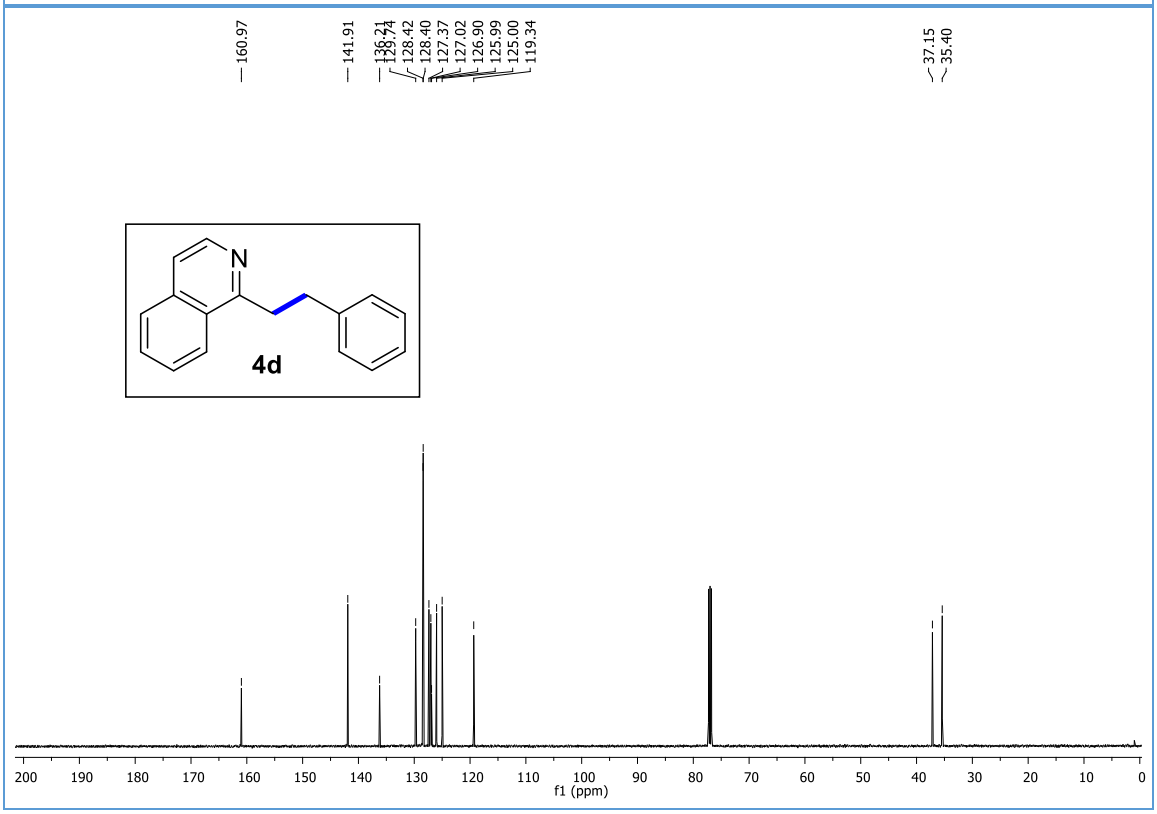
7.59
7.58
7.57
7.57
7.56
7.55
7.55
7.53
7.51
7.32
7.30
7.24
7.24
7.23
7.22
7.21
7.20
7.19
3.62
3.60
3.60
3.59
3.57
3.22
3.20
3.19
3.19
3.17
N
4d
0.96
1.00
0.97
1.00
1.04
0.97
3.85
1.02
2.05
1.99
10.0
9.5
9.0
8.5
8.0
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
0.5
0.0
f1 (ppm)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141.91
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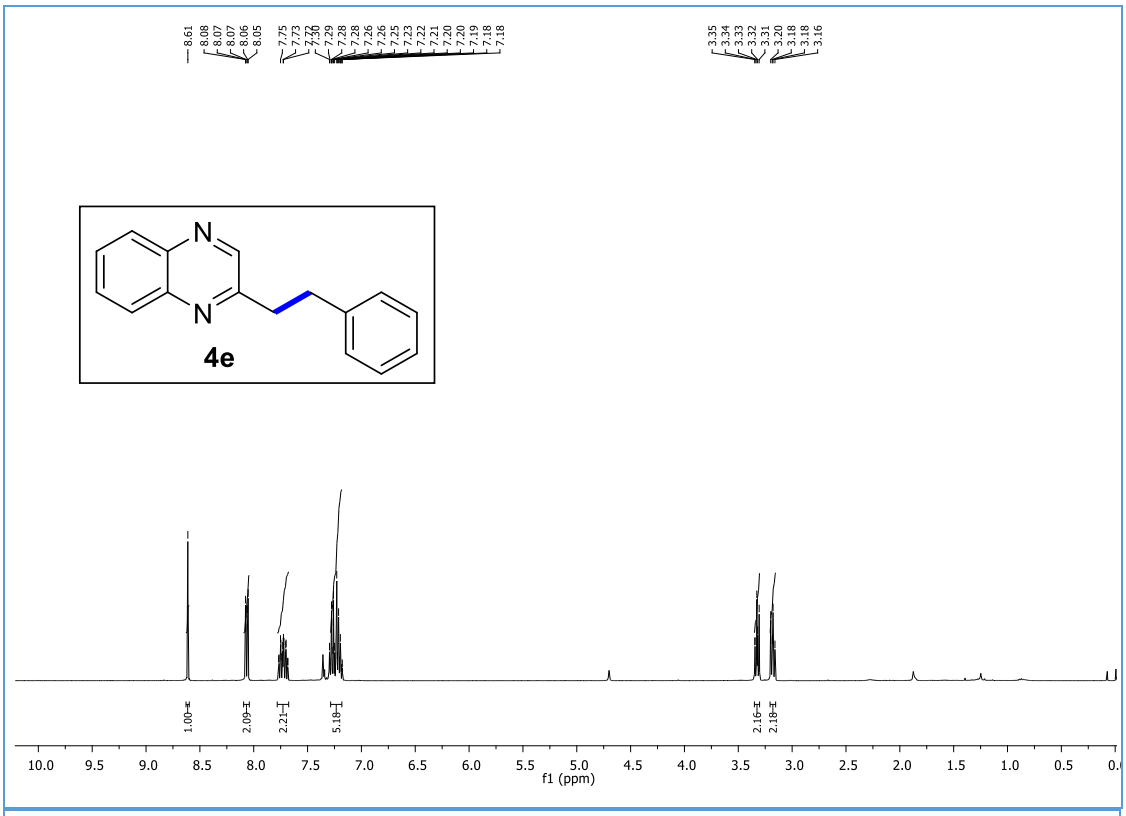
129.74
128.42
128.40
127.37
127.02
126.90
125.99
125.00
119.34
37.15
35.40
N
4d
200
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
f1 (ppm)


8.61
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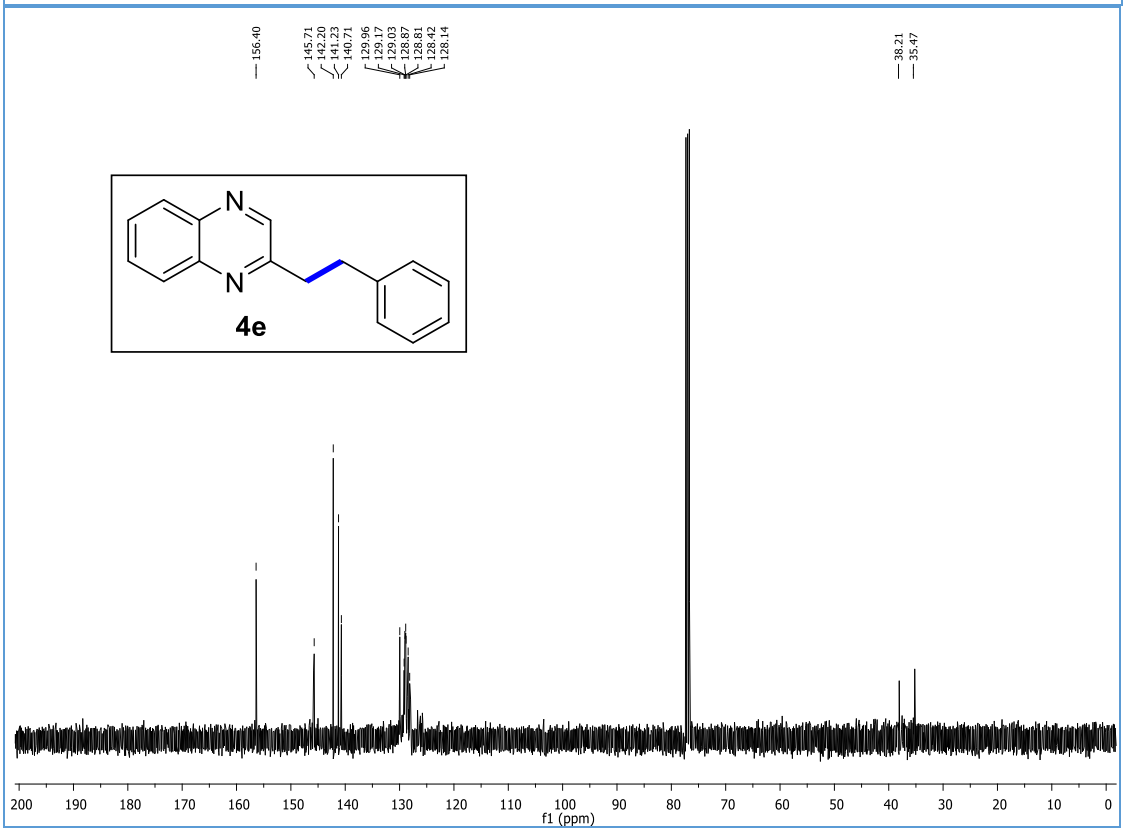
8.07
8.06
8.05
7.75
7.73
7.30
7.29
7.28
7.26
7.26
7.25
7.23
7.22
7.21
7.20
7.20
7.19
7.18
7.18
3.35
3.34
3.33
3.32
3.31
3.20
3.18
3.18
3.16
N
N
4e
1.00
2.09
2.21
5.18
2.16
2.18
10.0
9.5
9.0
8.5
8.0
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
0.5
f1 (ppm)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145.71
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141.23
140.71
129.96
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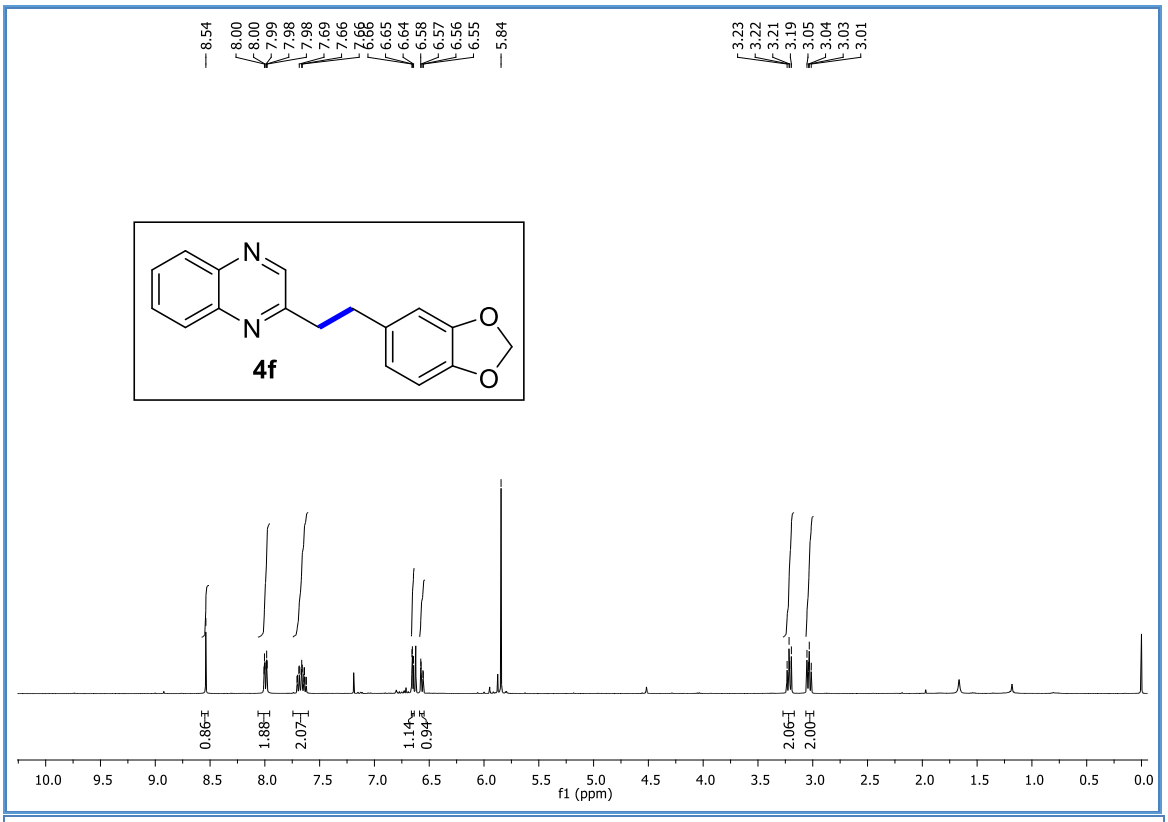
129.03
128.87
128.81
128.42
128.14
38.21
35.47
N
N
4e
200
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
f1 (ppm)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8.00
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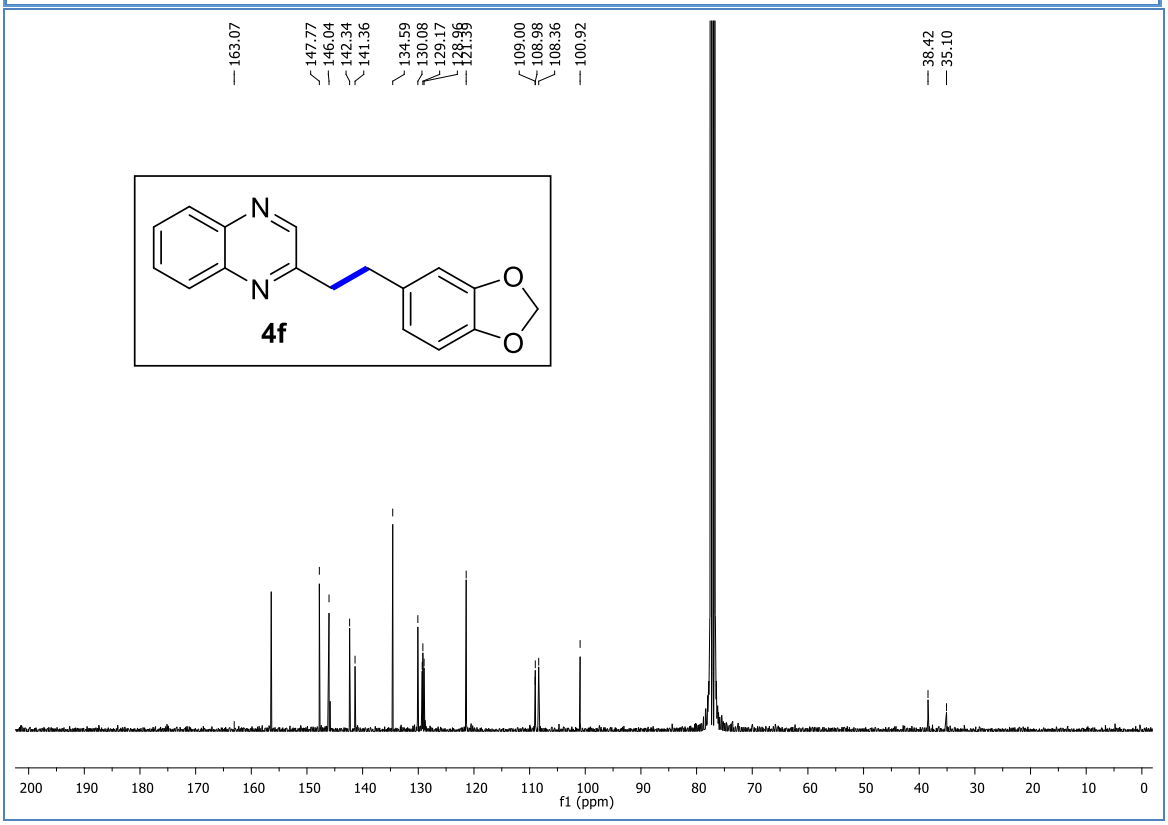
7.98
7.98
7.69
7.66
6.65
6.64
6.58
6.57
6.56
6.55
5.84
3.23
3.22
3.21
3.19
3.05
3.04
3.03
3.01
4f
0.86
1.88
2.07
1.14
0.94
2.06
2.00
f1 (ppm)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142.34
141.36
134.59
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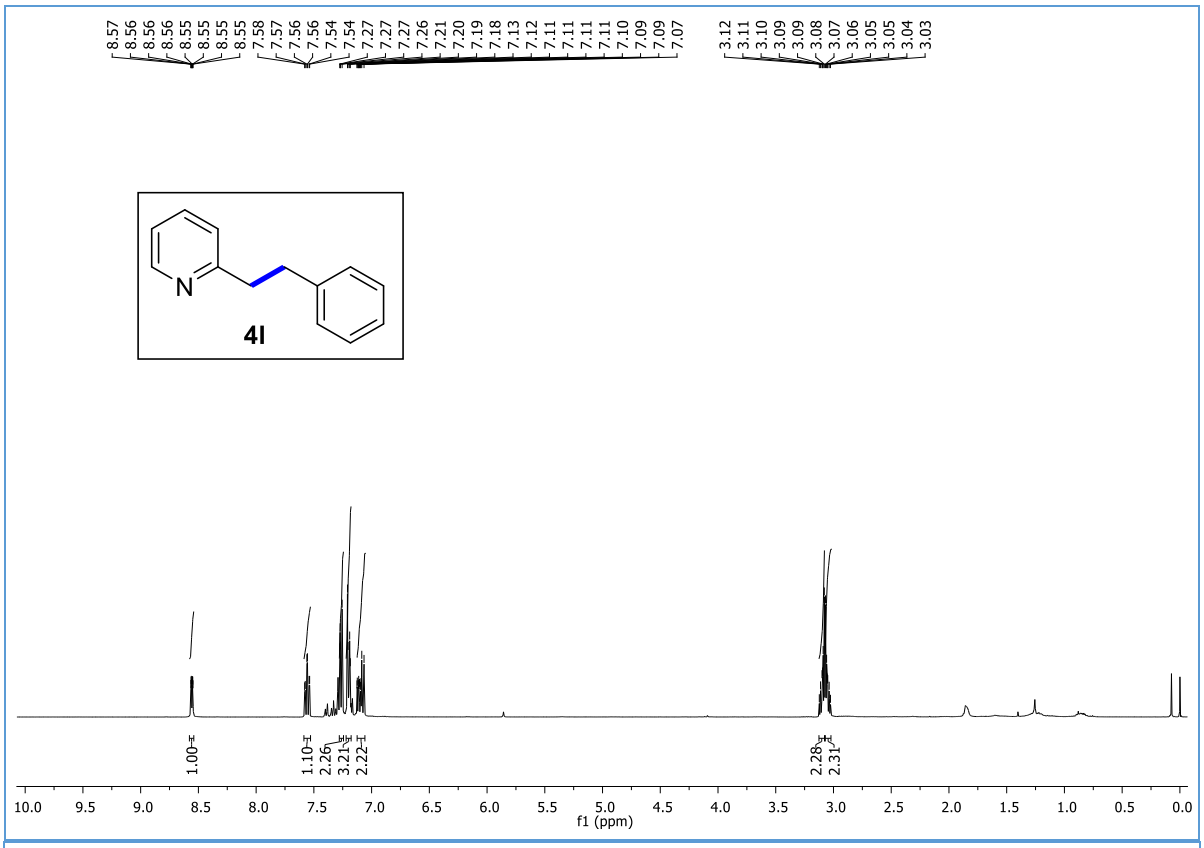
129.17
109.00
108.98
108.36
100.92
38.42
35.10
4f
f1 (ppm)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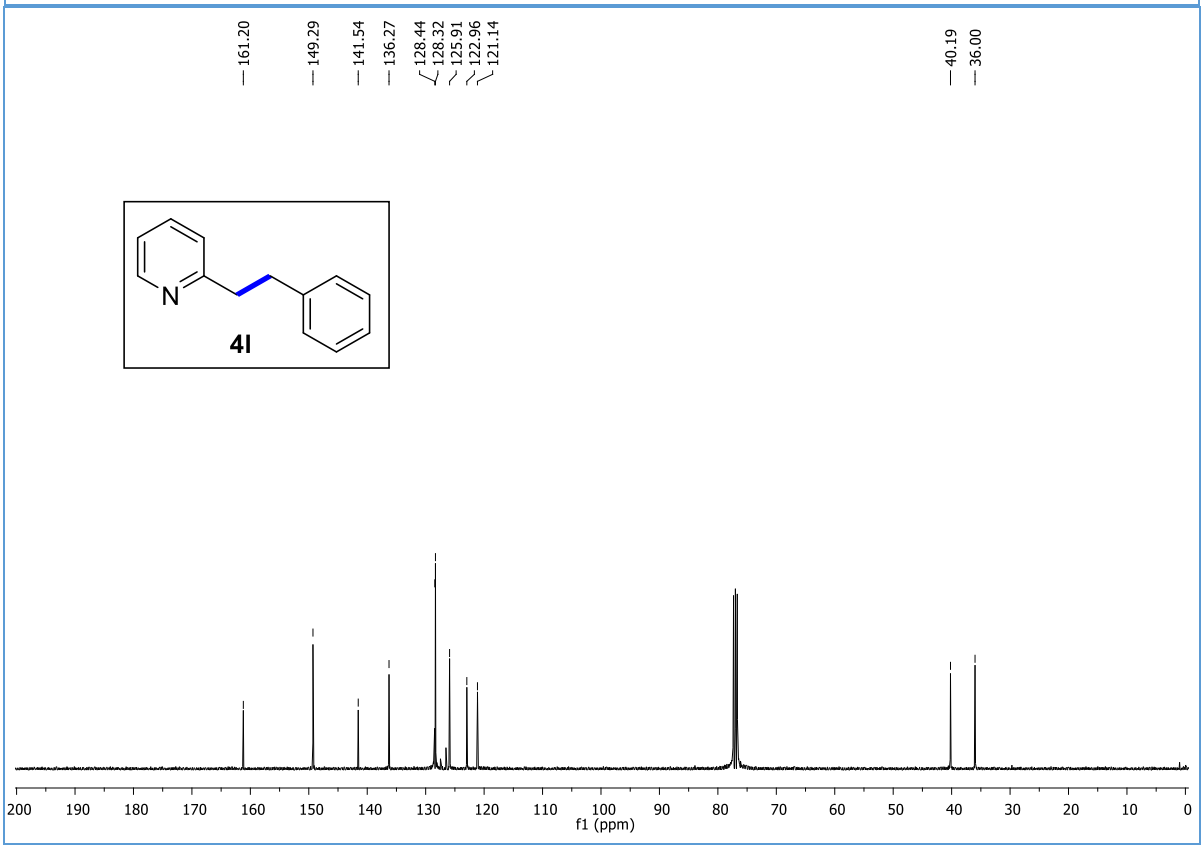
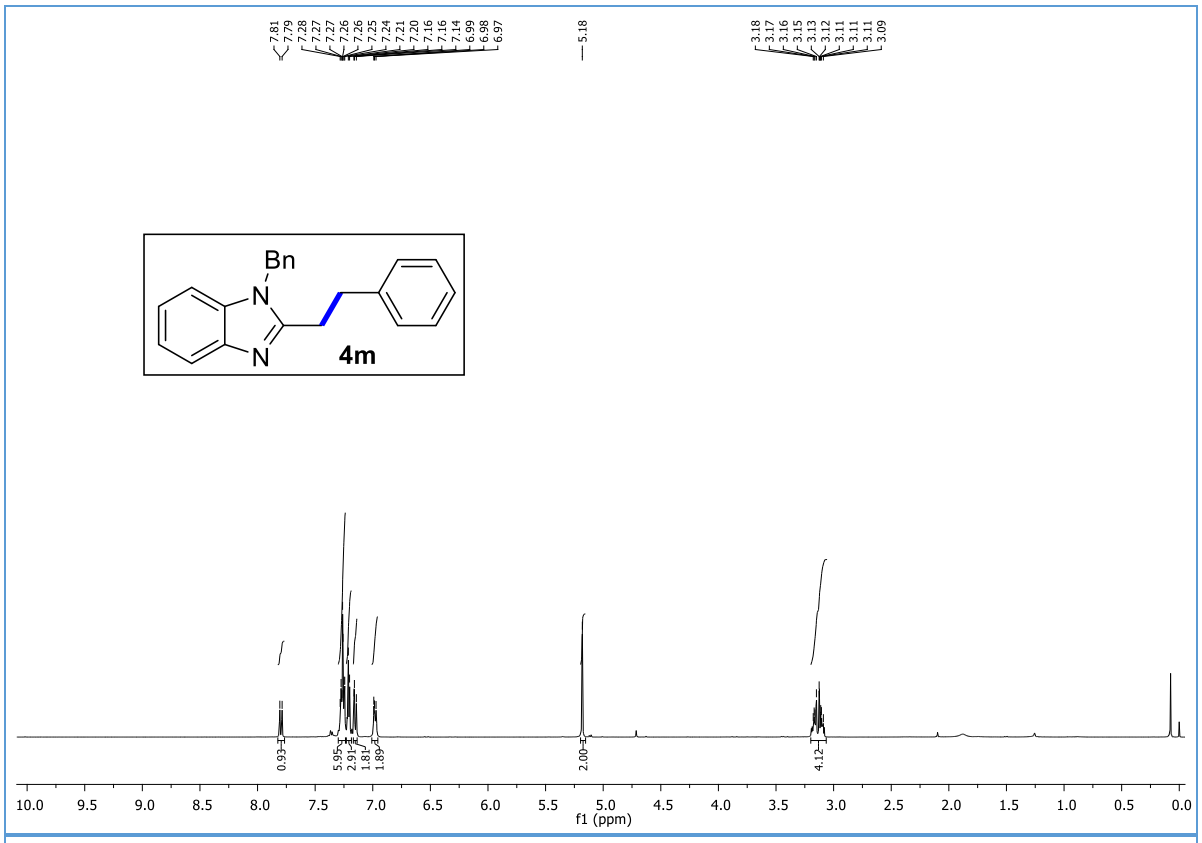
8.56
8.56
8.55
8.55
8.55
8.55
7.58
7.57
7.56
7.56
7.54
7.54
7.27
7.27
7.27
7.26
7.21
7.20
7.19
7.18
7.13
7.12
7.11
7.11
7.11
7.11
7.10
7.09
7.09
7.07
3.12
3.11
3.10
3.09
3.09
3.08
3.07
3.06
3.05
3.05
3.04
3.03
N
4l
1.00
1.10
2.26
3.21
2.22
2.28
2.31
10.0
9.5
9.0
8.5
8.0
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
0.5
0.0
f1 (ppm)


161.20
149.29
141.54
136.27
128.44
128.32
125.91
122.96
121.14
40.19
36.00
N
4l
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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7.26
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7.24
7.21
7.20
7.16
7.14
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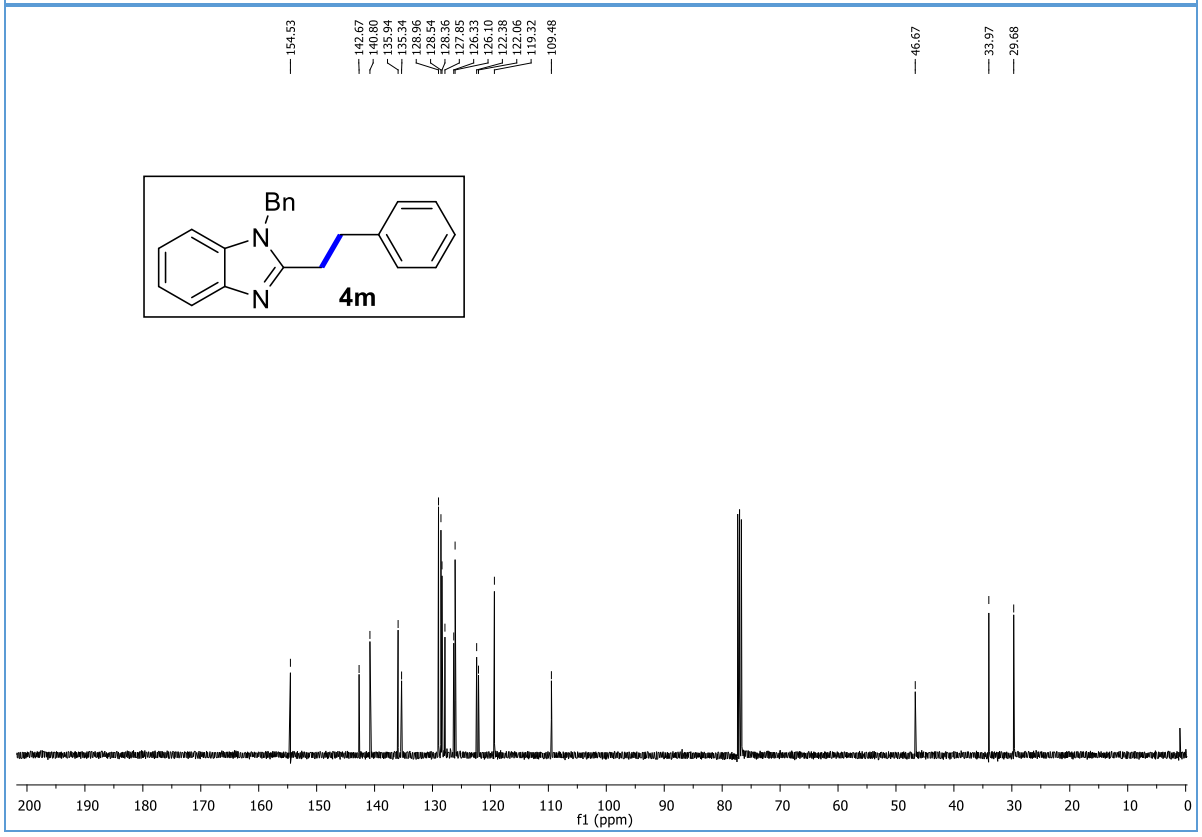
6.98
6.97
5.18
3.18
3.17
3.16
3.15
3.13
3.12
3.11
3.11
3.11
3.09
Bn
N
N
4m
0.93
5.95
2.91
1.81
1.89
2.00
4.12
10.0 9.5 9.0 8.5 8.0 7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 0.5 0.0
f1 (ppm)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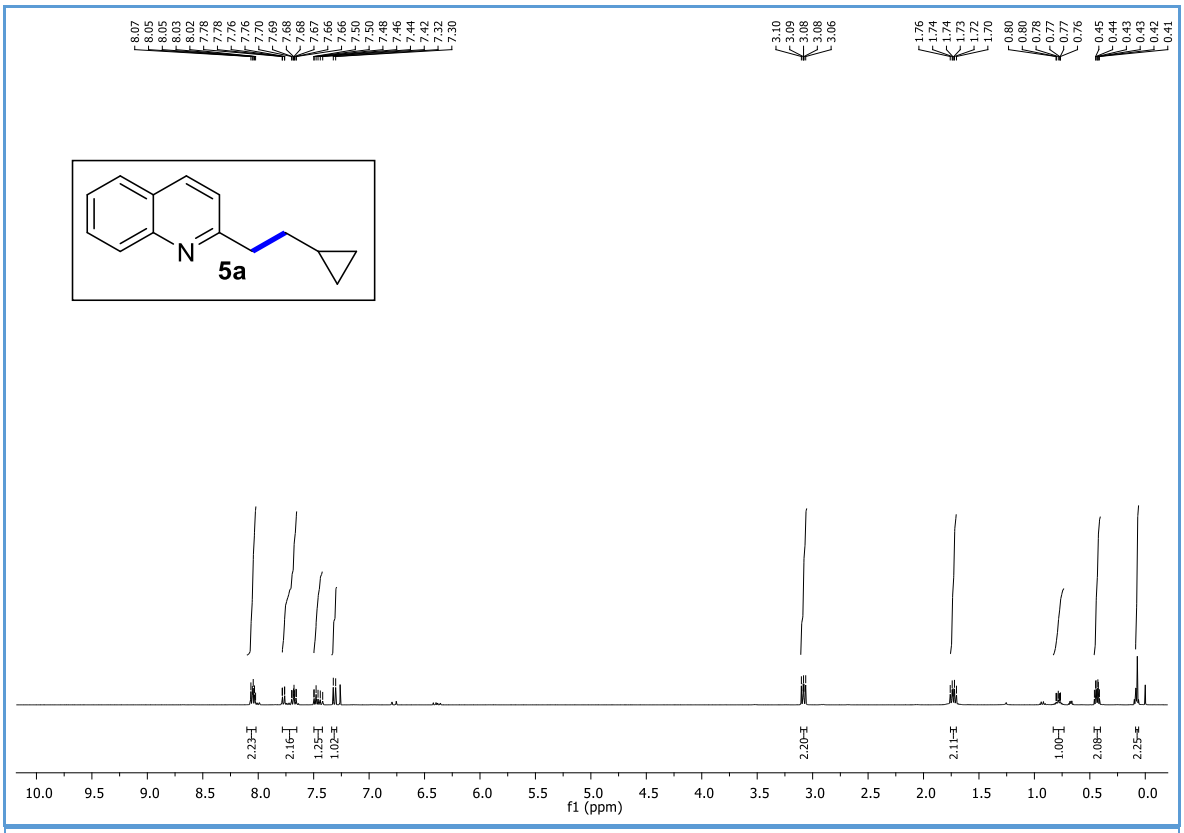
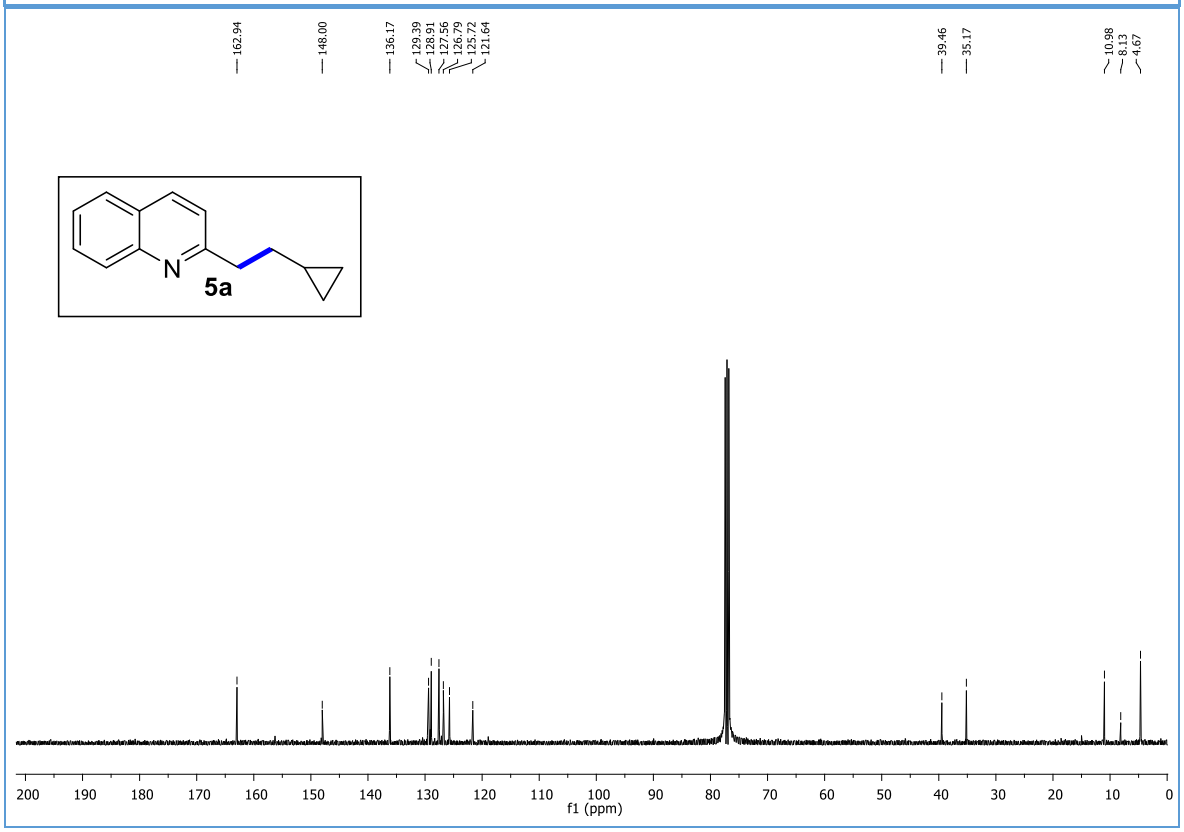
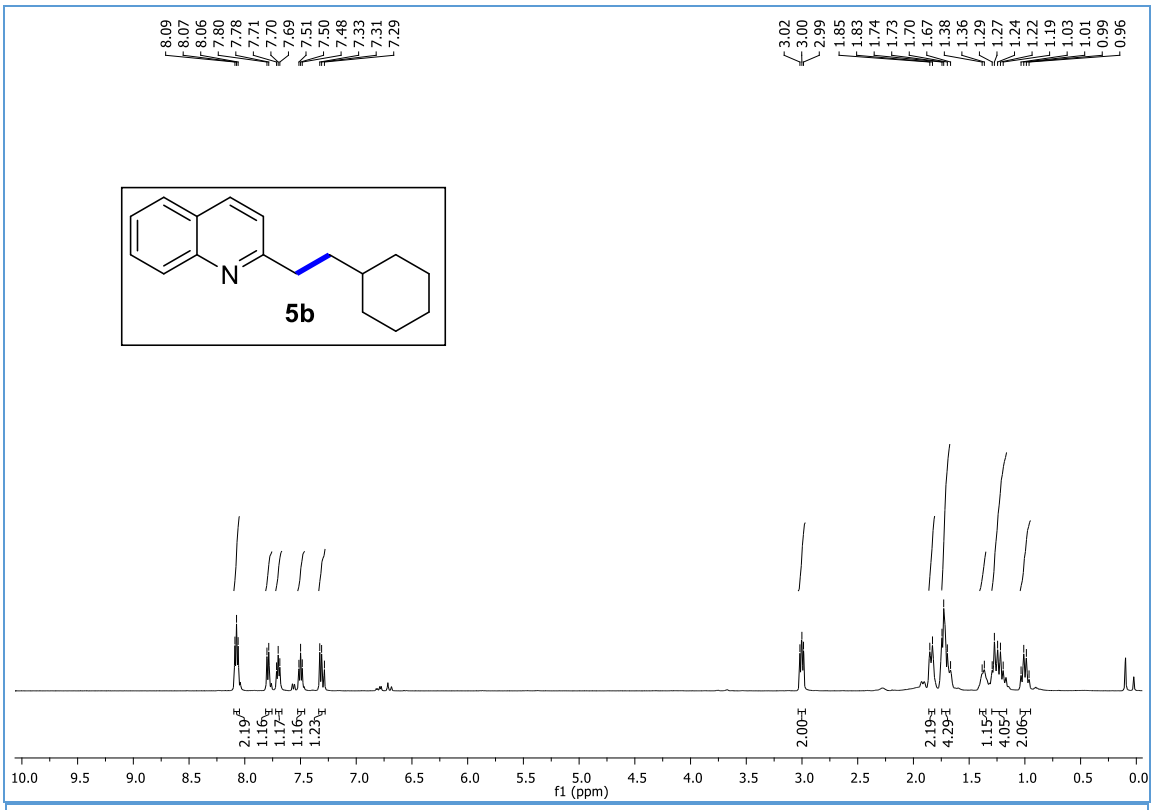
135.34
128.96
128.54
128.36
127.85
126.33
126.10
122.38
122.06
119.32
109.48
46.67
33.97
29.68
Bn
N
N
4m
200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0
f1 (ppm)


8.07 8.05 8.05 8.03 8.02 7.78 7.78 7.76 7.76 7.70 7.69 7.68 7.68 7.67 7.66 7.66 7.50 7.50 7.48 7.46 7.44 7.42 7.32 7.30
3.10 3.09 3.08 3.08 3.06
1.76 1.74 1.74 1.73 1.72 1.70
0.80 0.80 0.78 0.77 0.77 0.76
0.45 0.44 0.43 0.43 0.42 0.41
N
5a
2.23 2.16 1.25 1.02 2.20 2.11 1.00 2.08 2.25
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)


162.94 148.00 136.17 129.39 128.91 127.56 126.79 125.72 121.64
39.46 35.17
10.98 8.13 4.67
N
5a
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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8.07
8.06
7.80
7.78
7.71
7.70
7.69
7.51
7.50
7.48
7.33
7.31
7.29
3.02
3.00
2.99
1.85
1.83
1.74
1.73
1.70
1.67
1.38
1.36
1.29
1.27
1.24
1.22
1.19
1.03
1.01
0.99
0.96
N
5b
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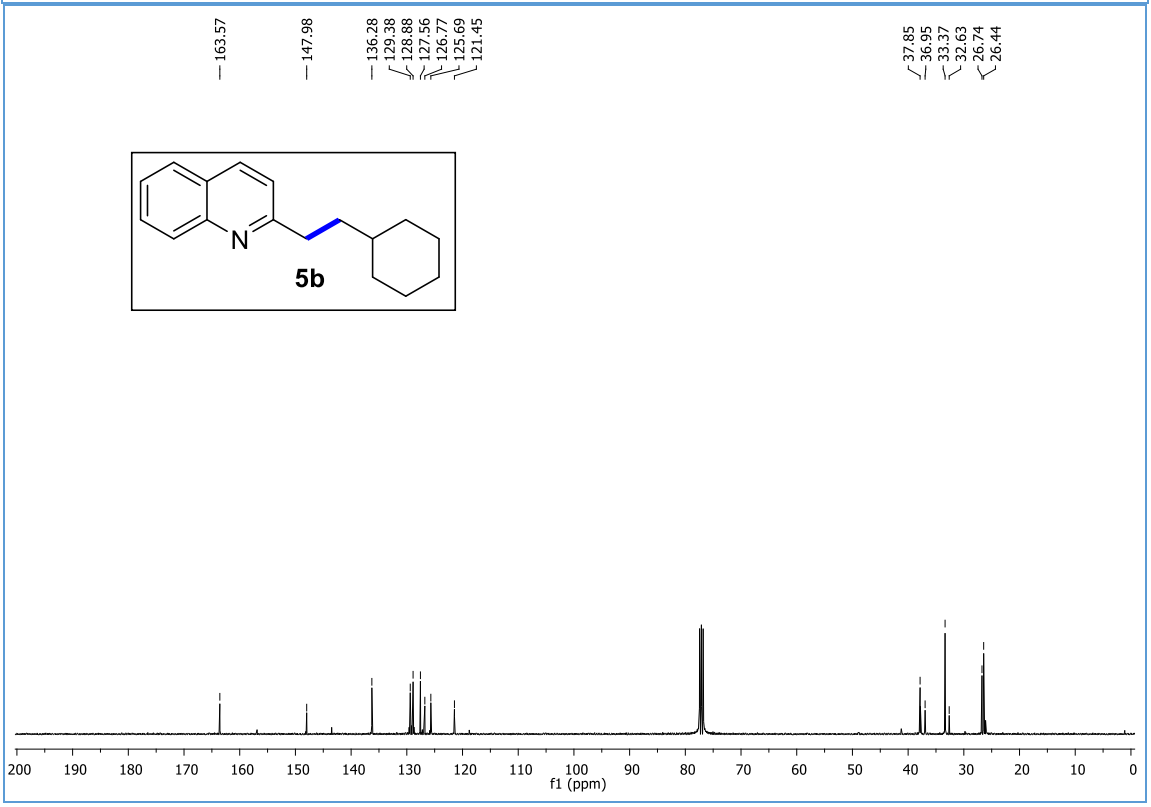
4.05
2.06
f1 (ppm)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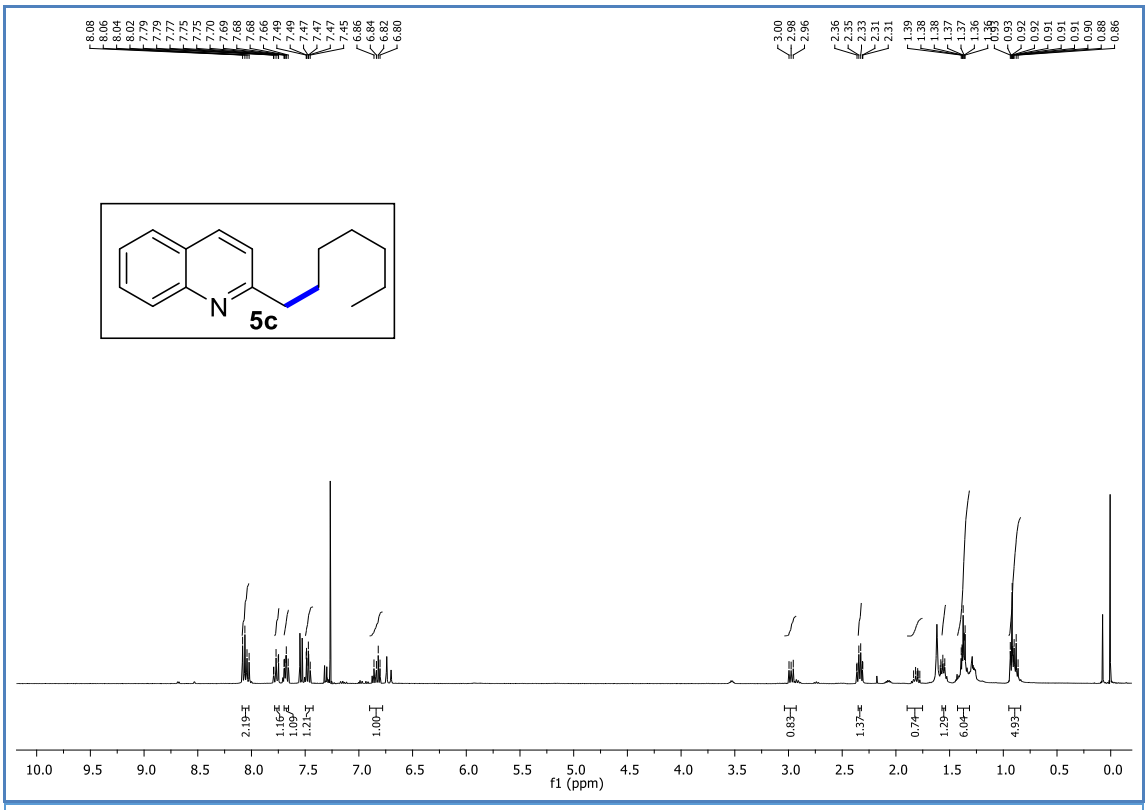
26.44
N
5b
f1 (ppm)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7.49
7.47
7.47
7.45
6.86
6.84
6.82
6.80
3.00
2.98
2.96
2.36
2.35
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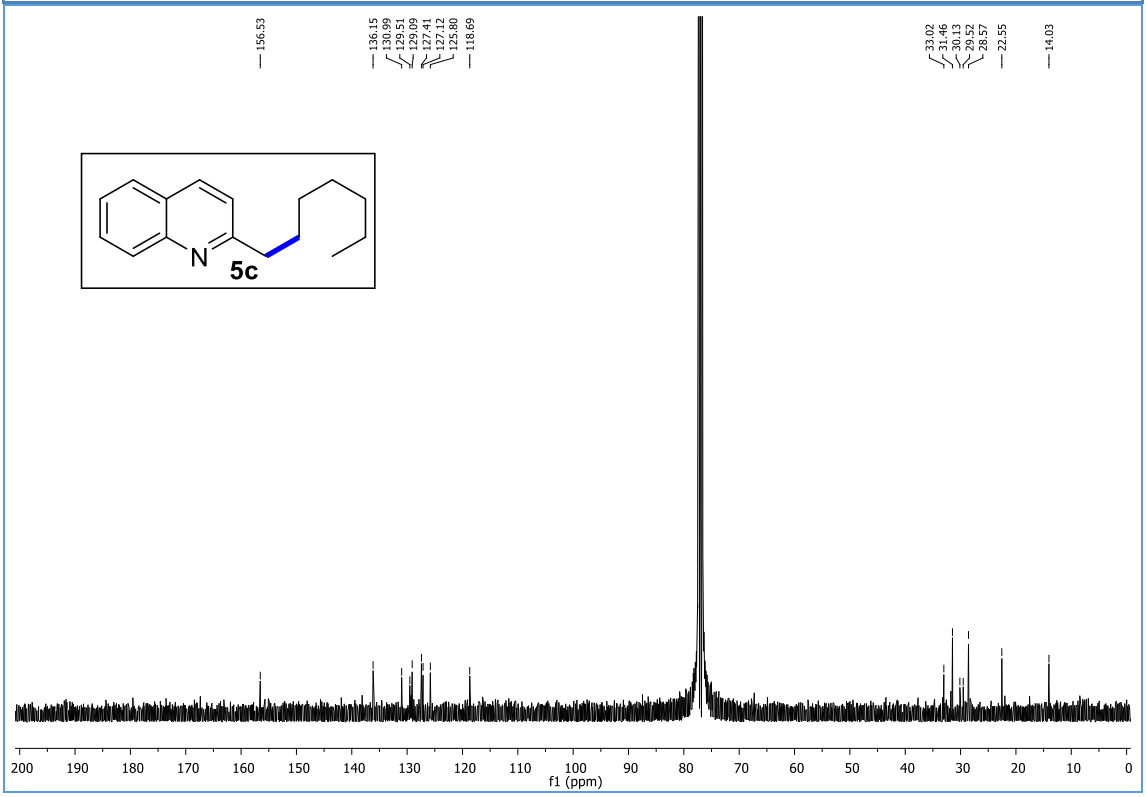
2.31
2.31
1.39
1.38
1.38
1.37
1.37
1.36
1.36
0.93
0.93
0.92
0.92
0.91
0.91
0.91
0.90
0.88
0.86
N
5c
2.19
1.16
1.09
1.21
1.00
0.83
1.37
0.74
1.29
6.04
4.93
10.0 9.5 9.0 8.5 8.0 7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 0.5 0.0
f1 (ppm)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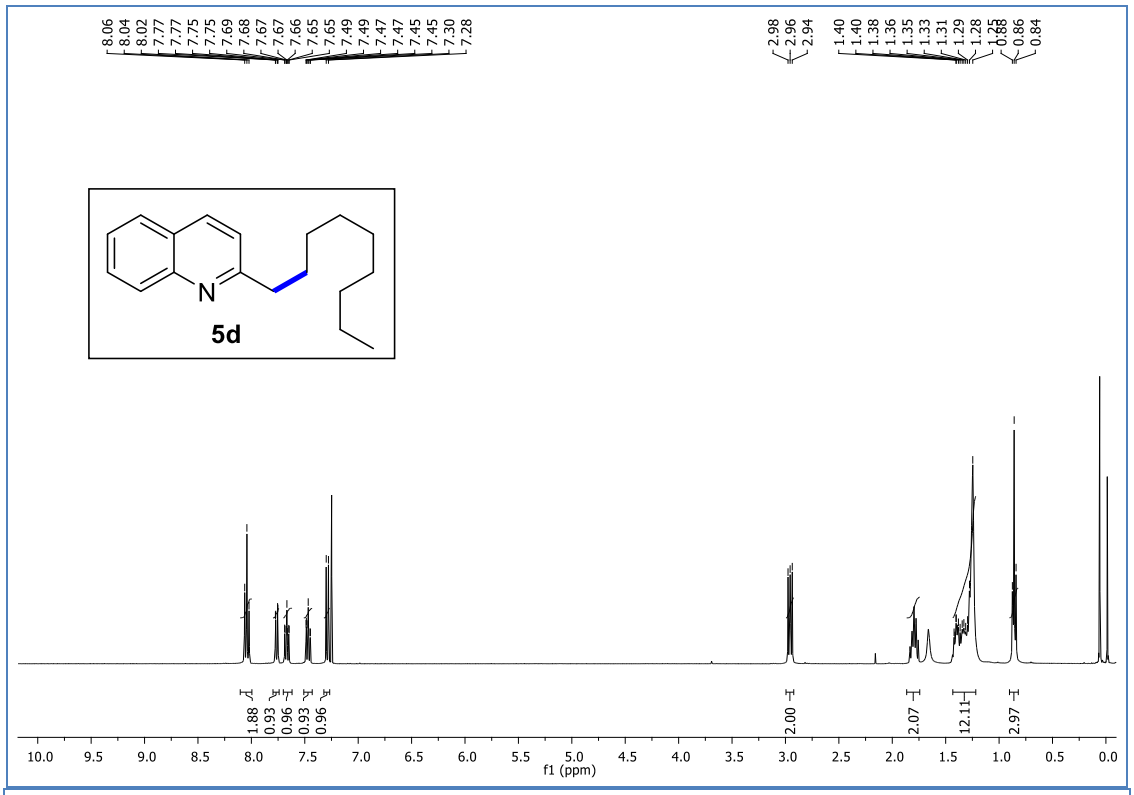
f1 (ppm)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7.49
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7.47
7.45
7.45
7.30
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2.96
2.94
1.40
1.40
1.38
1.36
1.35
1.33
1.31
1.29
1.28
1.25
0.88
0.86
0.84
N
5d
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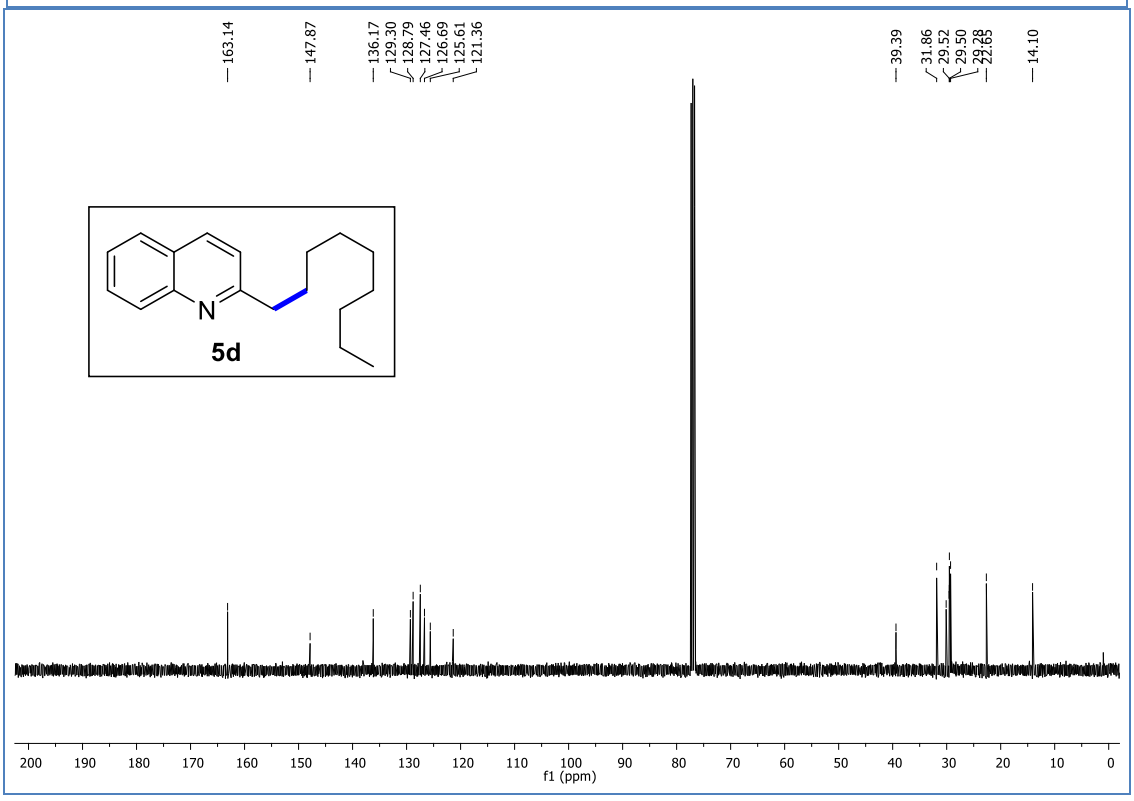
0.93
0.96
0.93
0.96
2.00
2.07
12.11
2.97
10.0 9.5 9.0 8.5 8.0 7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 0.5 0.0
f1 (ppm)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14.10
N
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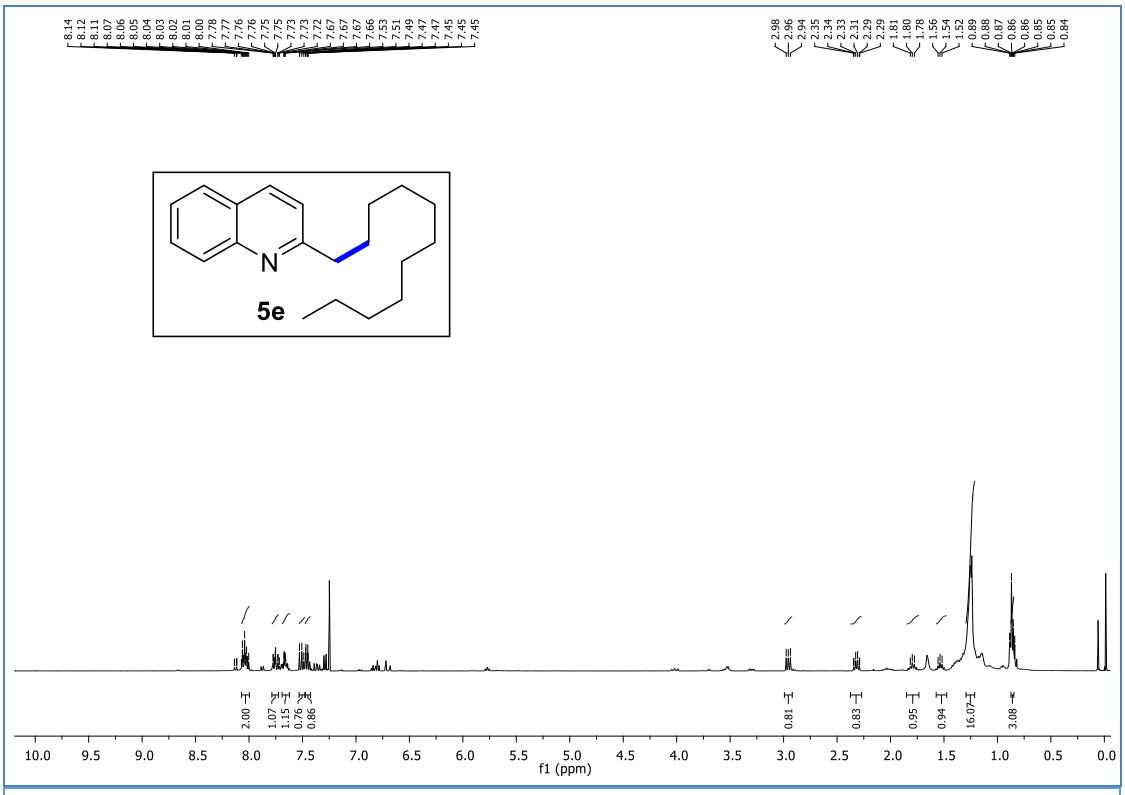
f1 (ppm)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7.76
7.76
7.75
7.75
7.73
7.73
7.72
7.67
7.67
7.66
7.53
7.51
7.49
7.47
7.47
7.45
7.45
7.45
2.98
2.96
2.94
2.35
2.34
2.33
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2.29
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1.80
1.78
1.56
1.54
1.52
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0.86
0.85
0.85
0.84
N
5e
2.00
1.07
1.15
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0.86
0.81
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0.95
0.94
16.07
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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
4.0
3.5
3.0
2.5
2.0
1.5
1.0
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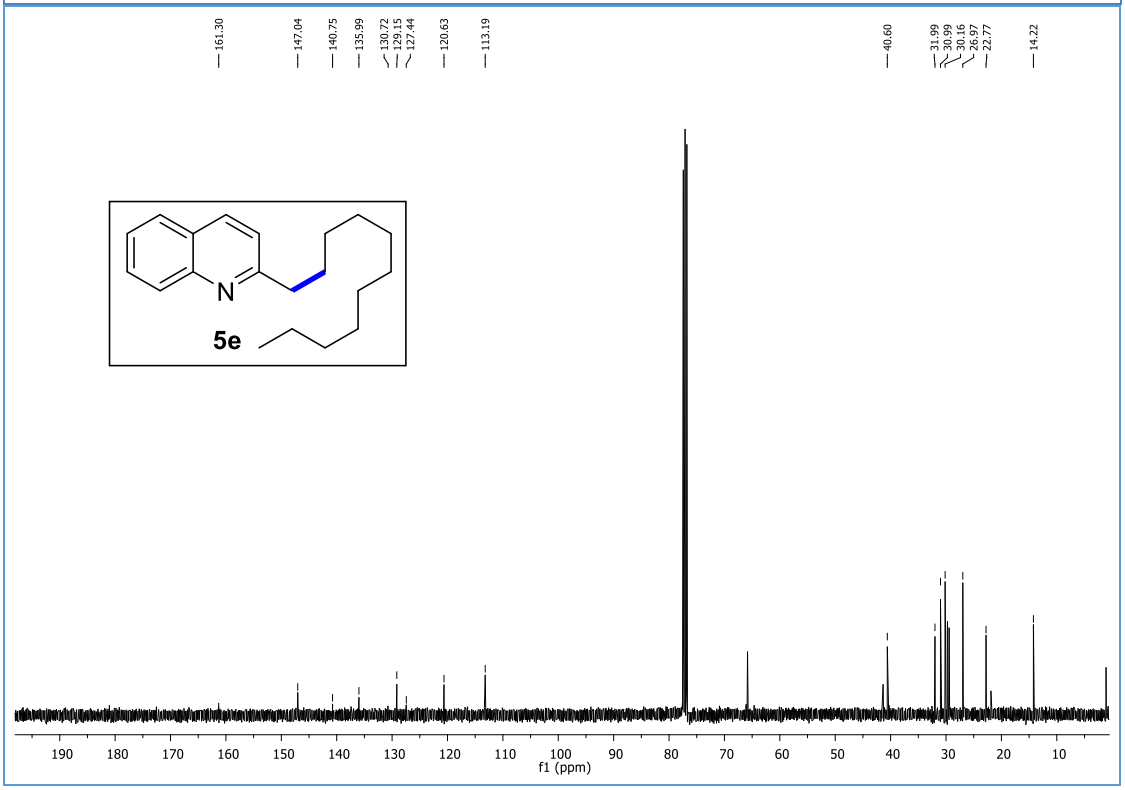
0.0
f1 (ppm)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113.19
40.60
31.99
30.99
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26.97
22.77
14.22
N
5e
190
180
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140
130
120
110
100
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80
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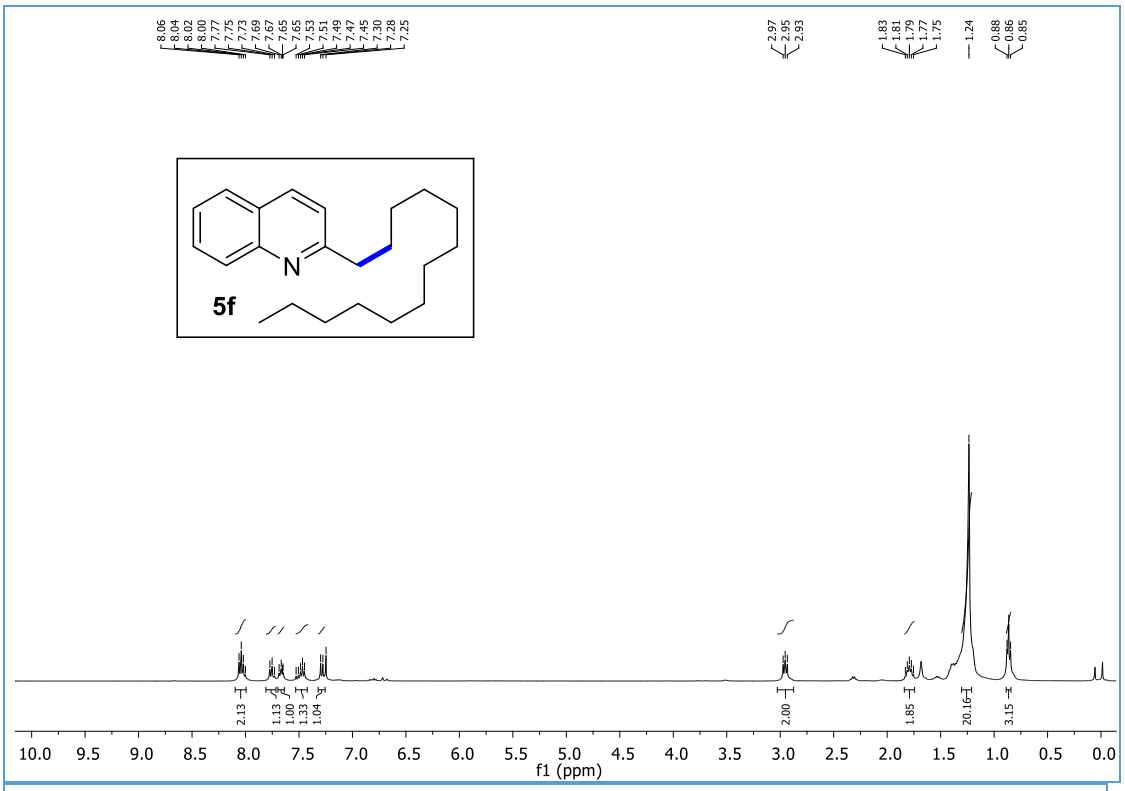
60
50
40
30
20
10
f1 (ppm)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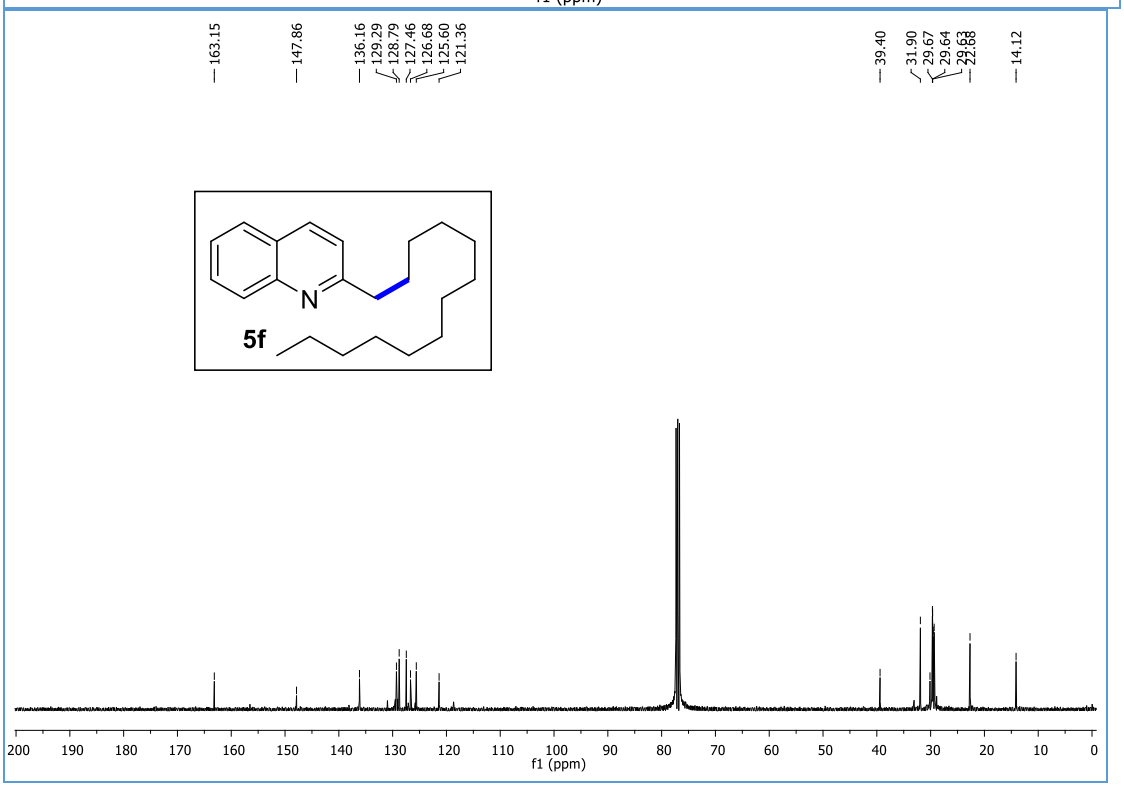
7.65
7.53
7.51
7.49
7.47
7.45
7.30
7.28
7.25
2.97
2.95
2.93
1.83
1.81
1.79
1.77
1.75
1.24
0.88
0.86
0.85
N
5f
2.13
1.13
1.00
1.33
1.04
2.00
1.85
20.16
3.15
10.0 9.5 9.0 8.5 8.0 7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 0.5 0.0
f1 (ppm)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22.68
14.12
N
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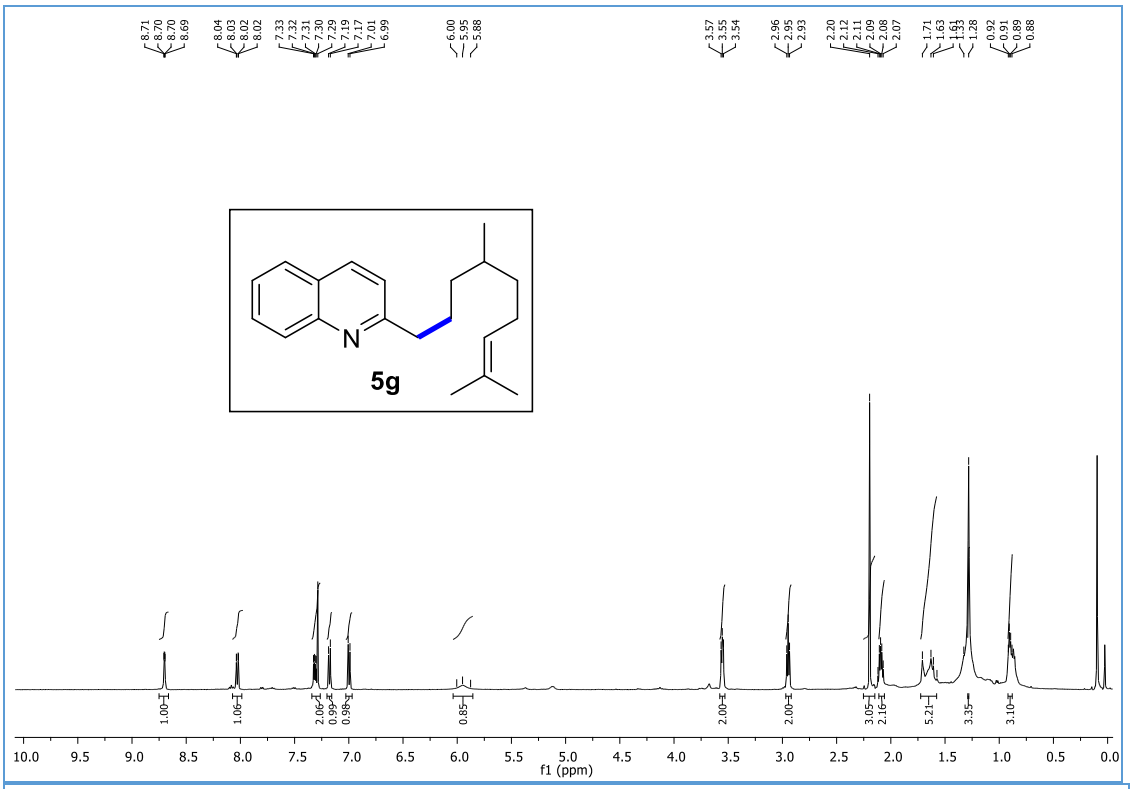
f1 (ppm)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7.29
7.19
7.17
7.01
6.99
6.00
5.95
5.88
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3.55
3.54
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2.95
2.93
2.20
2.12
2.11
2.09
2.08
2.07
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1.63
1.61
1.53
1.28
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0.91
0.89
0.88
N
5g
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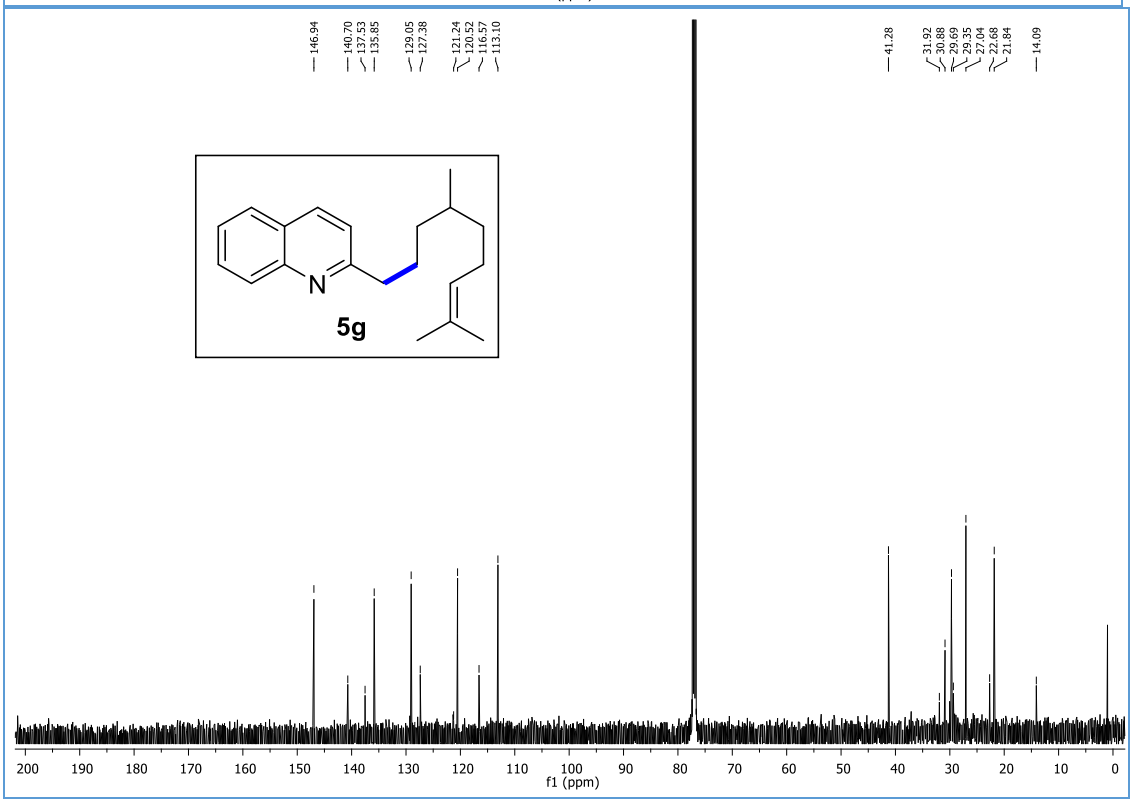
1.06
2.06
0.99
0.98
0.85
2.00
2.00
3.05
2.16
5.21
3.35
3.10
10.0 9.5 9.0 8.5 8.0 7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 0.5 0.0
f1 (ppm)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113.10
41.28
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30.88
29.69
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27.04
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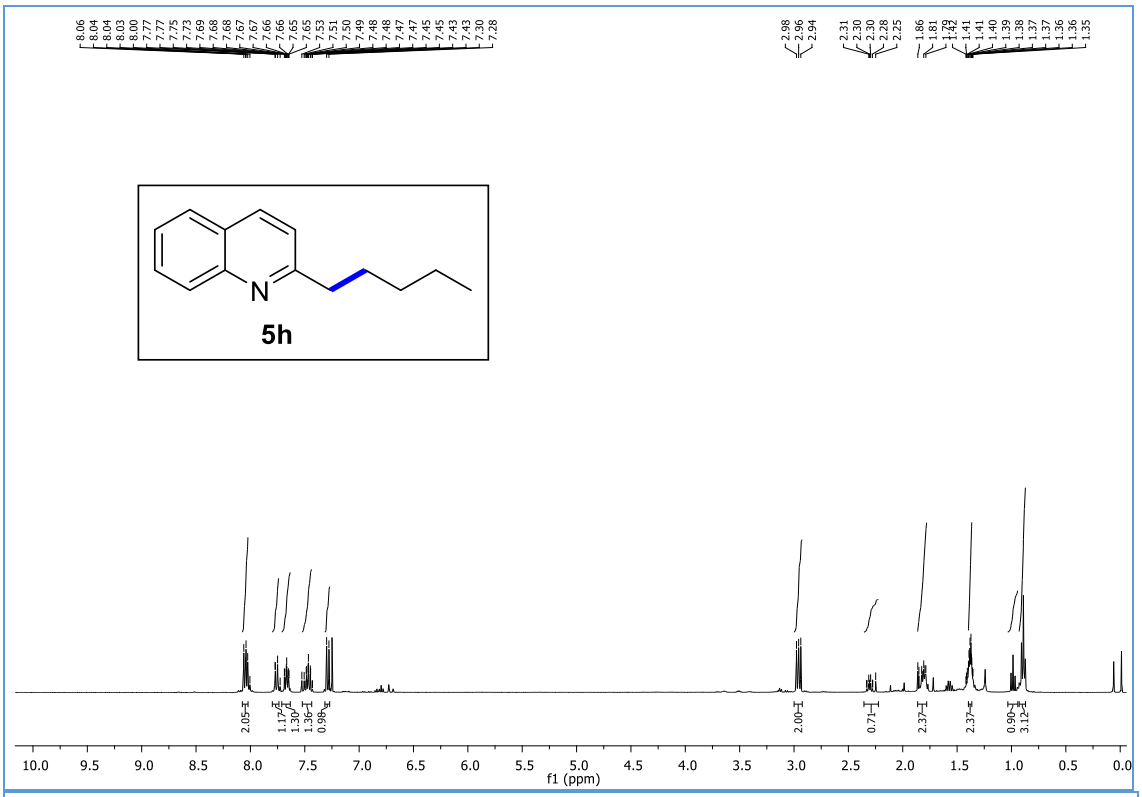
21.84
14.09
N
5g
200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0
f1 (ppm)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7.65
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7.45
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2.30
2.28
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1.41
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1.36
1.35
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8.0
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7.0
6.5
6.0
5.5
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3.0
2.5
2.0
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1.0
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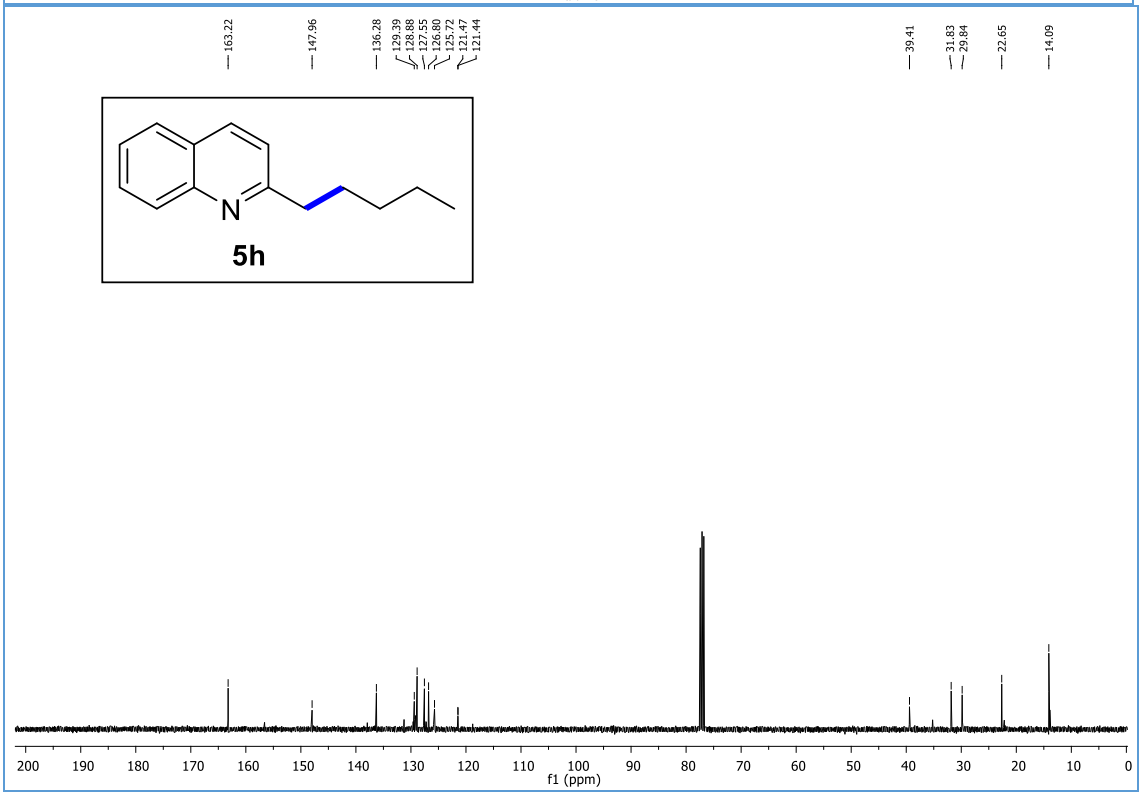
0.0
f1 (ppm)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31.83
29.84
22.65
14.09
N
5h
200
190
180
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110
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50
40
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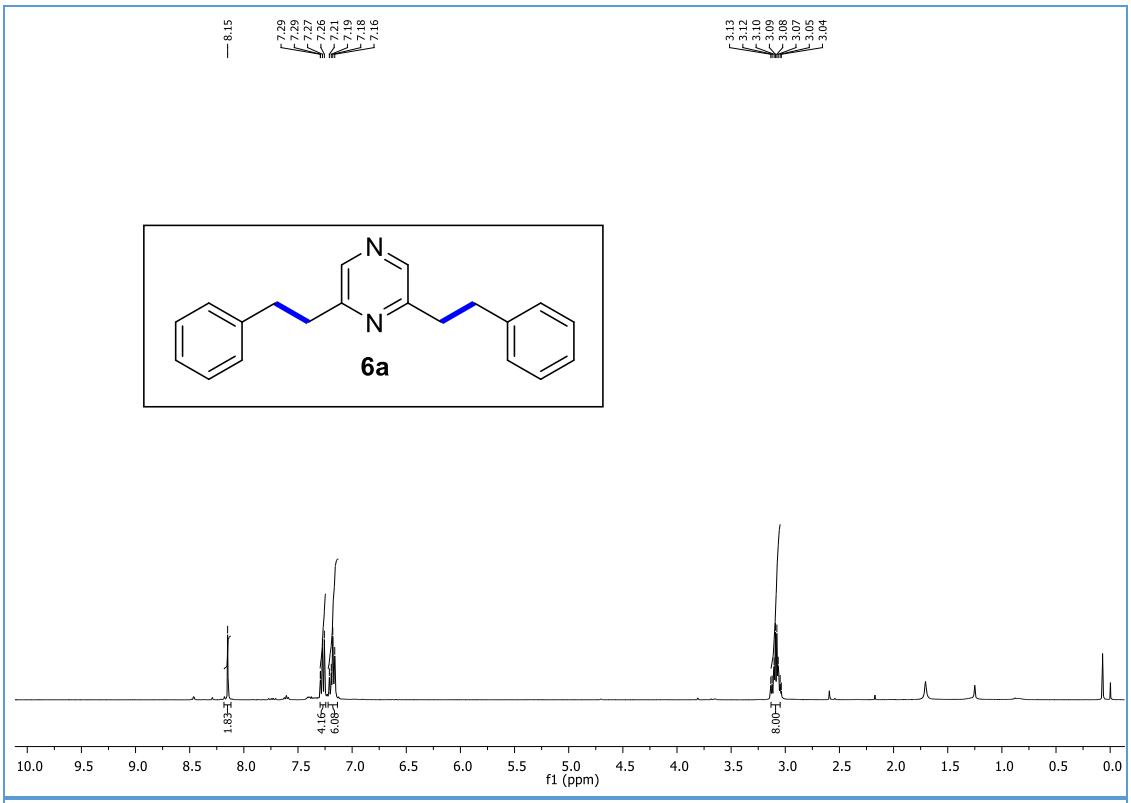
f1 (ppm)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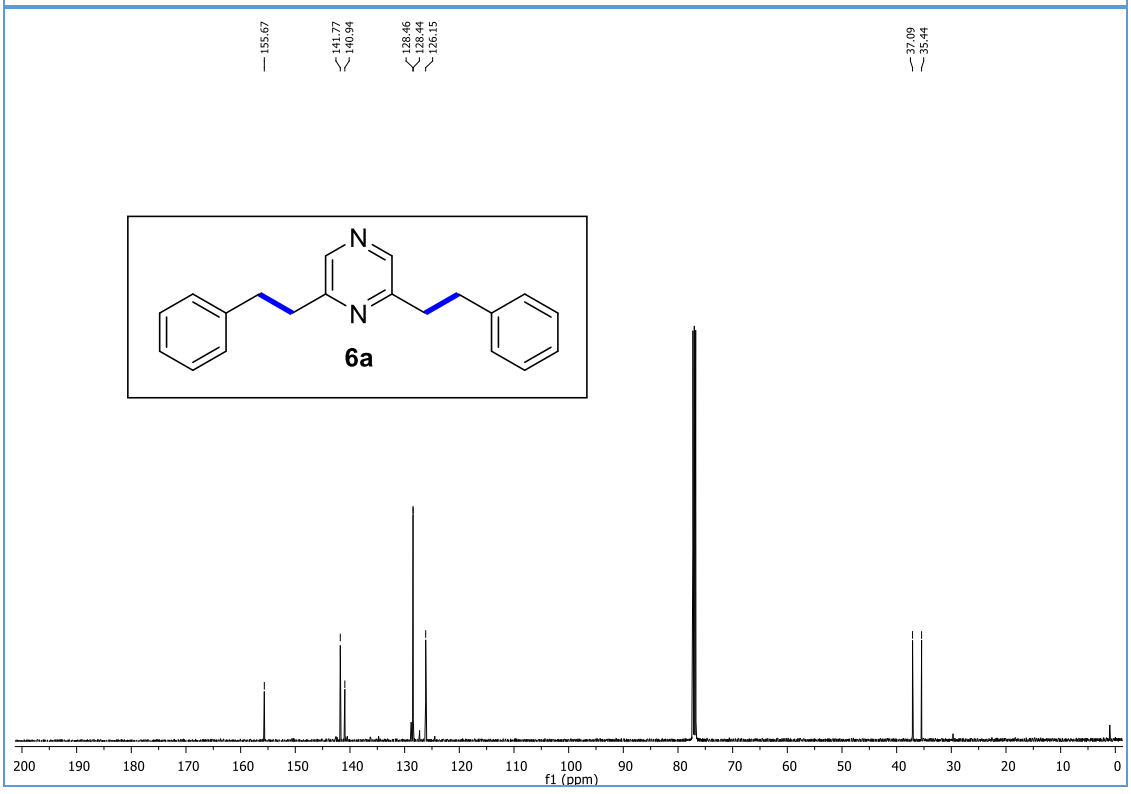
3.12
3.10
3.09
3.08
3.07
3.05
3.04
N
N
6a
1.83
4.16
6.08
8.00
10.0 9.5 9.0 8.5 8.0 7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 0.5 0.0
f1 (ppm)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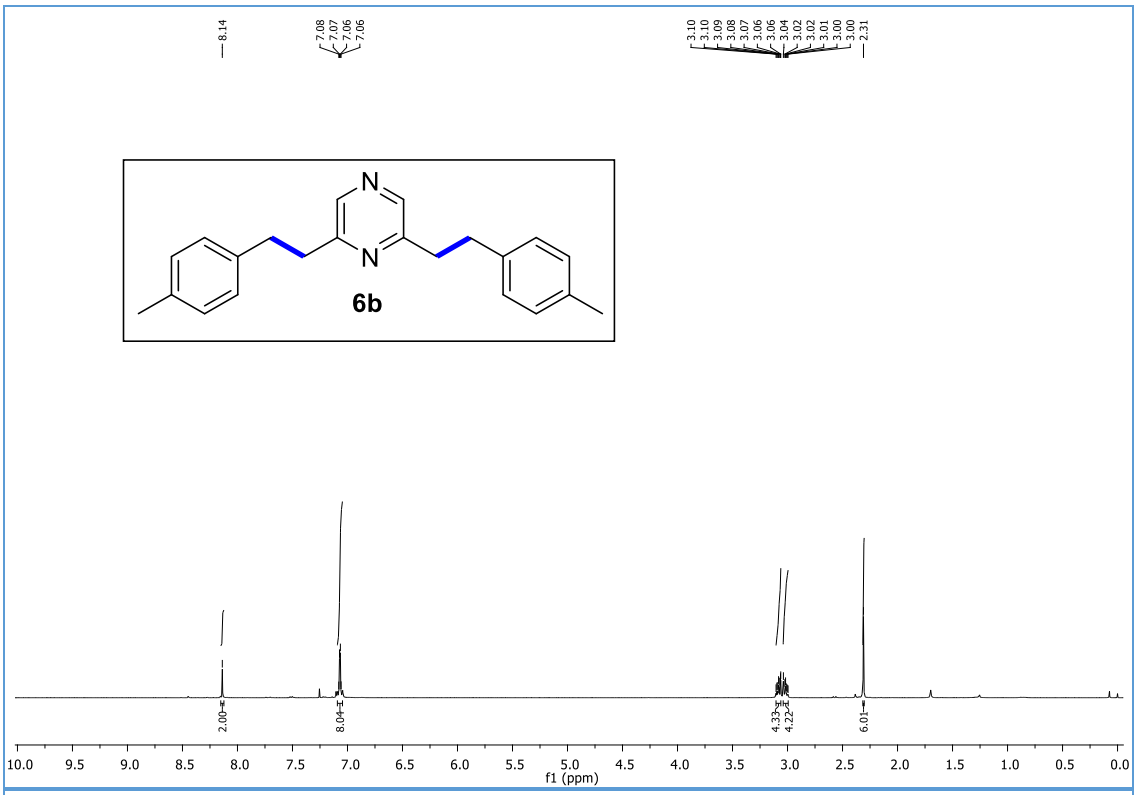
f1 (ppm)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3.06
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3.02
3.02
3.01
3.00
3.00
2.31
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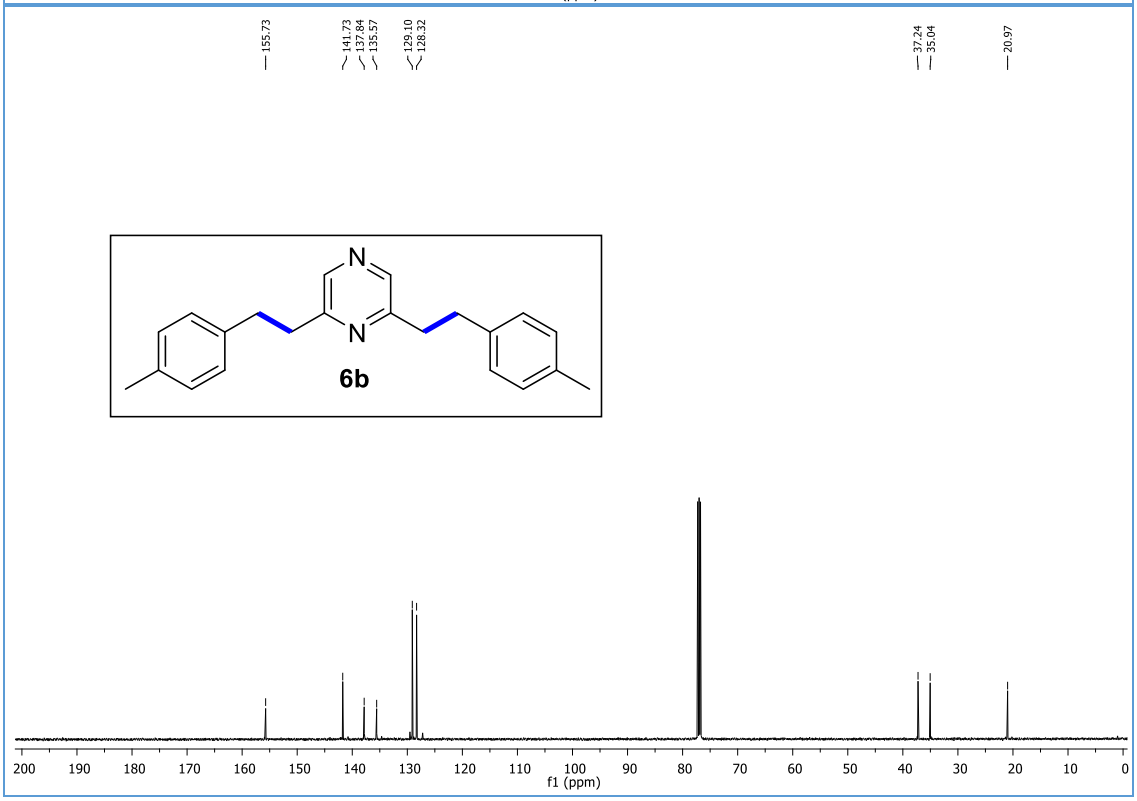
N
6b
2.00
8.04
4.33
4.22
6.01
10.0 9.5 9.0 8.5 8.0 7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 0.5 0.0
f1 (ppm)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N
6b
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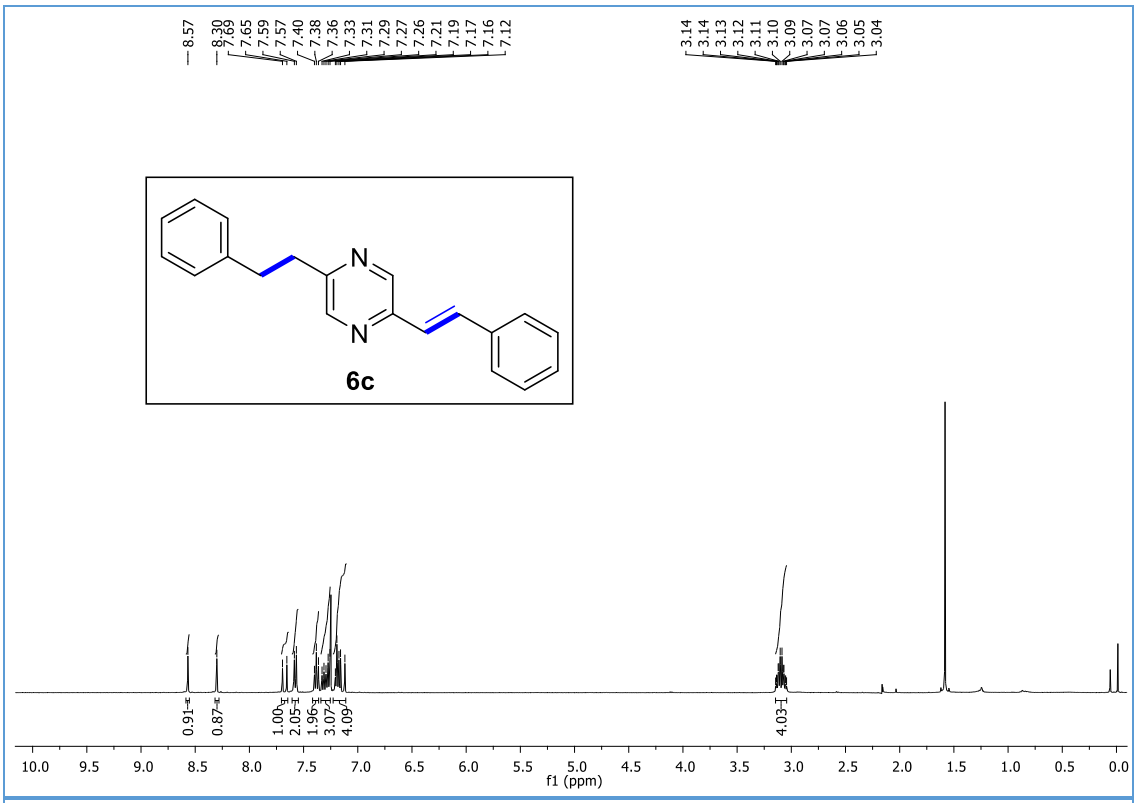
f1 (ppm)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7.19
7.17
7.16
7.12
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3.14
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3.12
3.11
3.10
3.09
3.07
3.07
3.06
3.05
3.04
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N
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0.87
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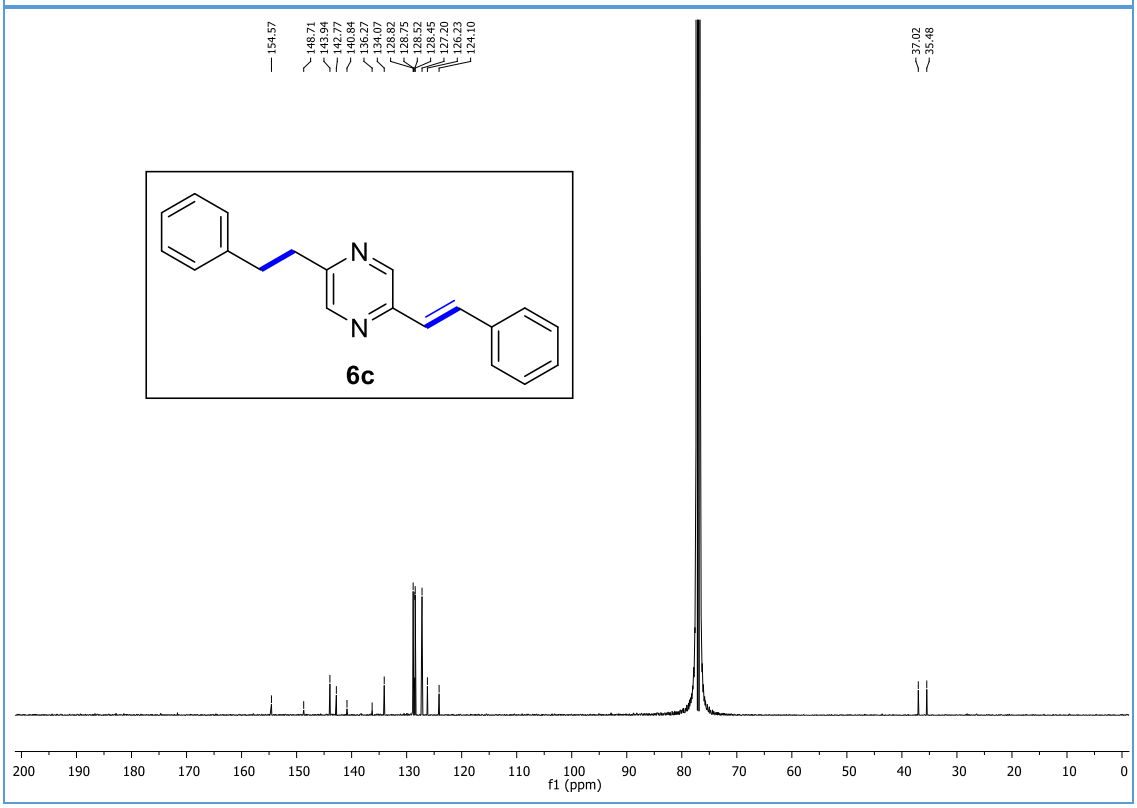
2.05
1.96
3.07
4.09
4.03
10.0 9.5 9.0 8.5 8.0 7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 0.5 0.0
f1 (ppm)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127.20
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N
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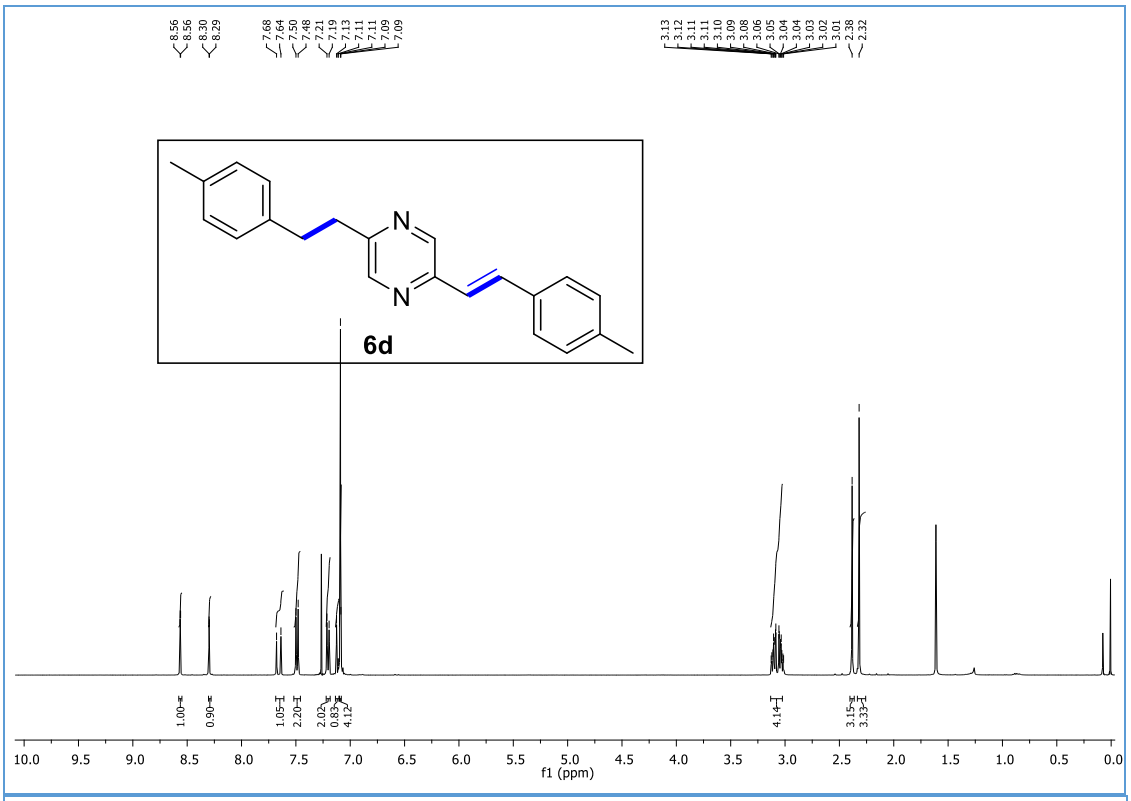
f1 (ppm)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7.09
7.09
3.13
3.12
3.11
3.11
3.10
3.09
3.08
3.06
3.05
3.04
3.04
3.03
3.02
3.01
2.38
2.32
N
N
6d
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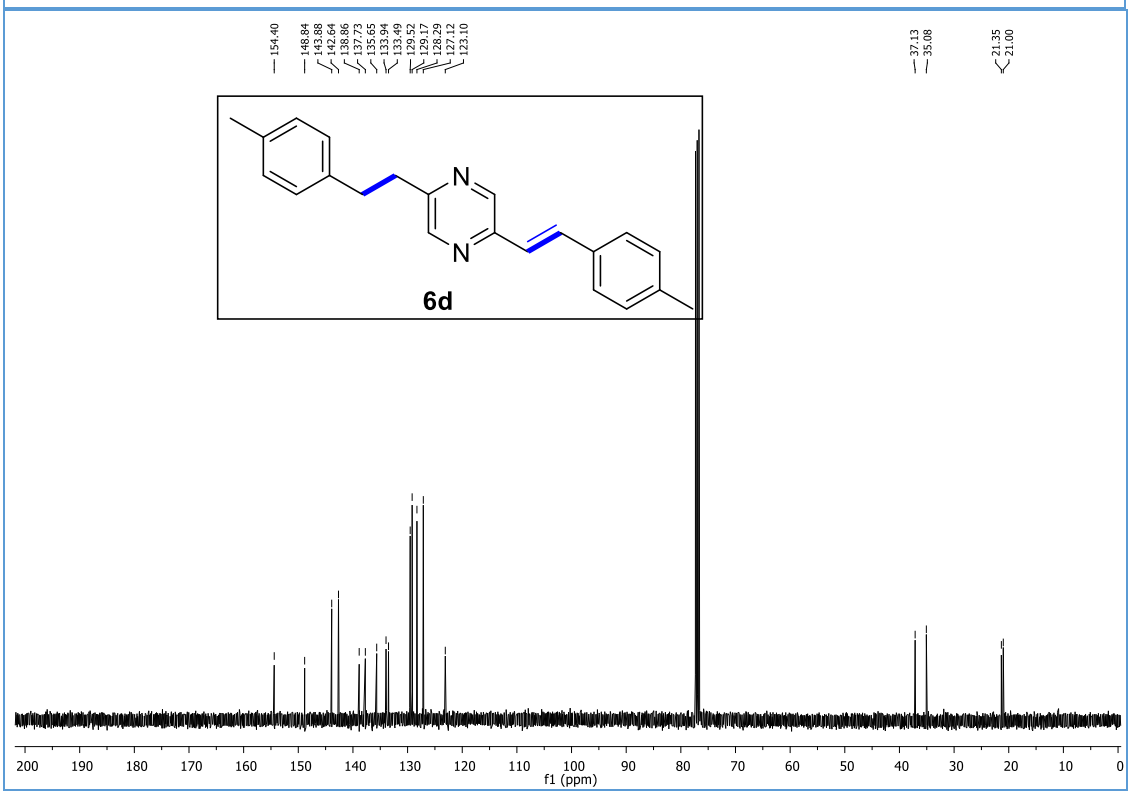
0.90
1.05
2.20
2.02
0.83
4.12
4.14
3.15
3.33
10.0 9.5 9.0 8.5 8.0 7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 0.5 0.0
f1 (ppm)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127.12
123.10
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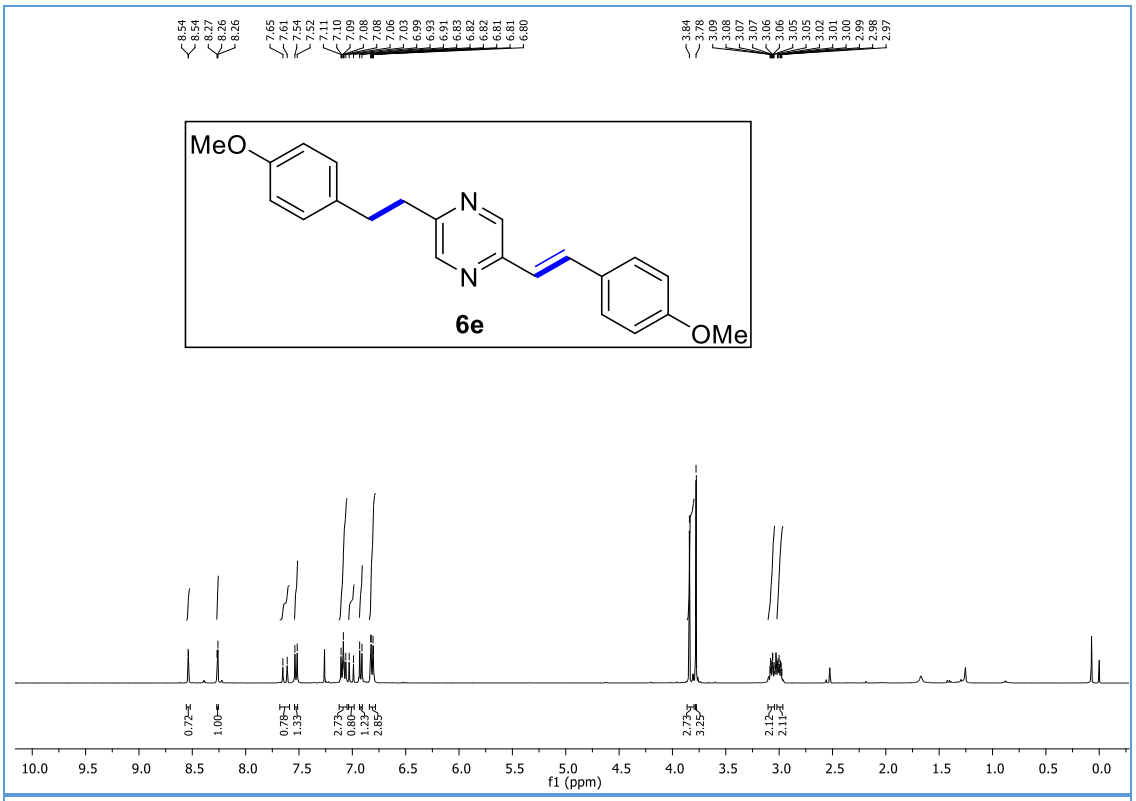
21.35
21.00
N
N
6d
200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0
f1 (ppm)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N
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OMe
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6.0
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3.0
2.5
2.0
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1.0
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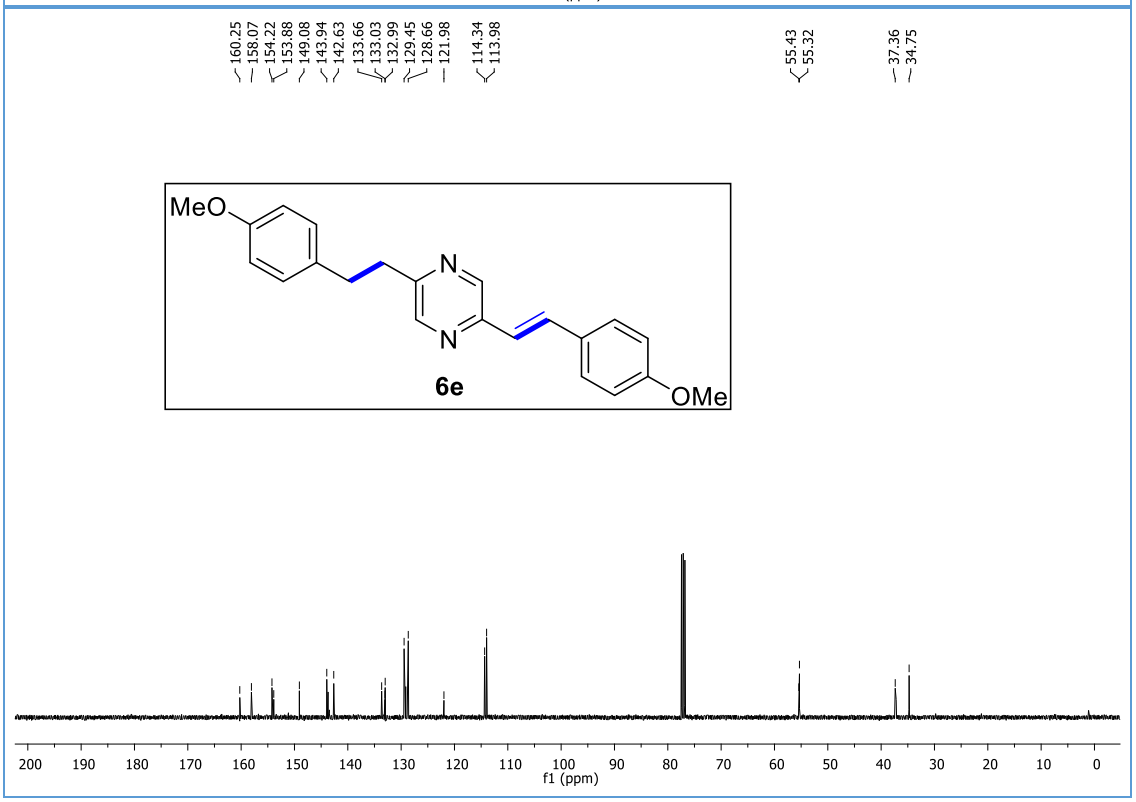
f1 (ppm)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133.03
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128.66
121.98
114.34
113.98
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55.32
37.36
34.75
MeO
N
N
6e
OMe
200
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180
170
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150
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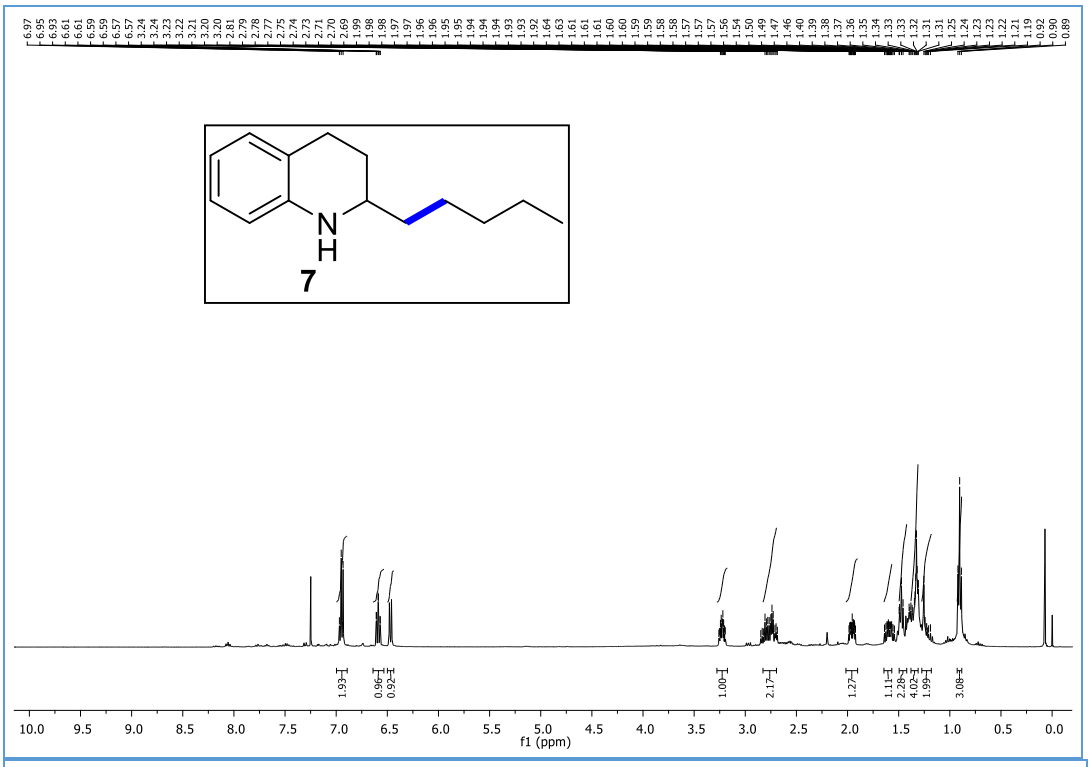
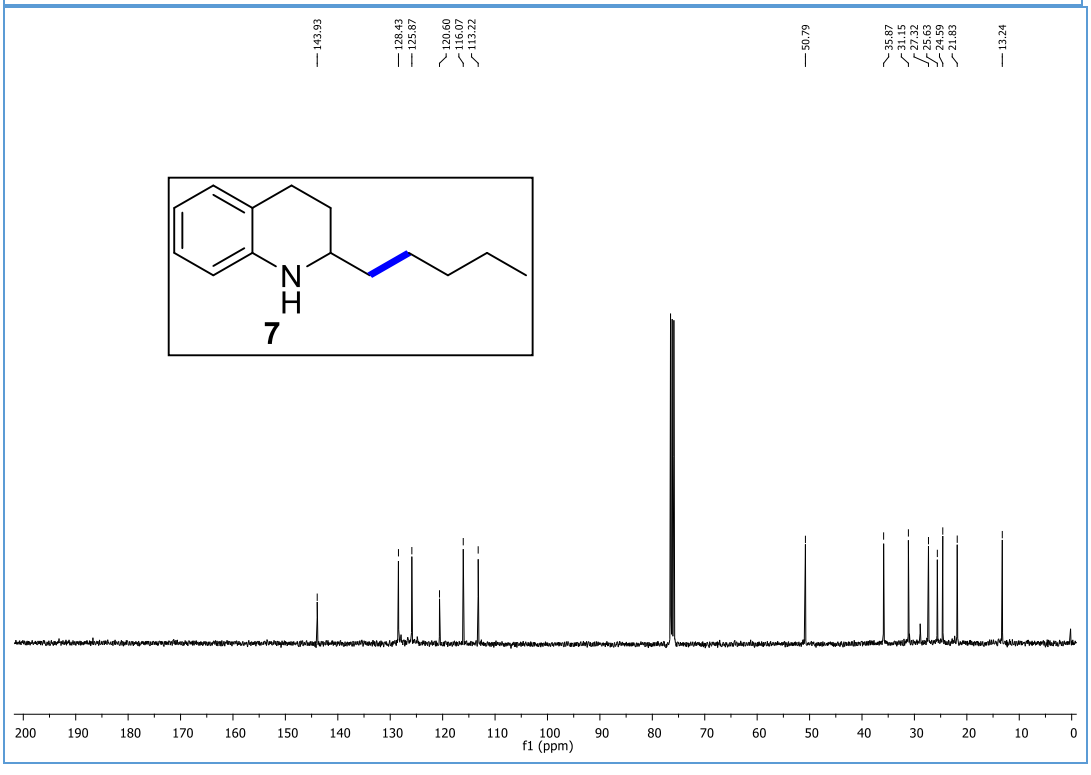
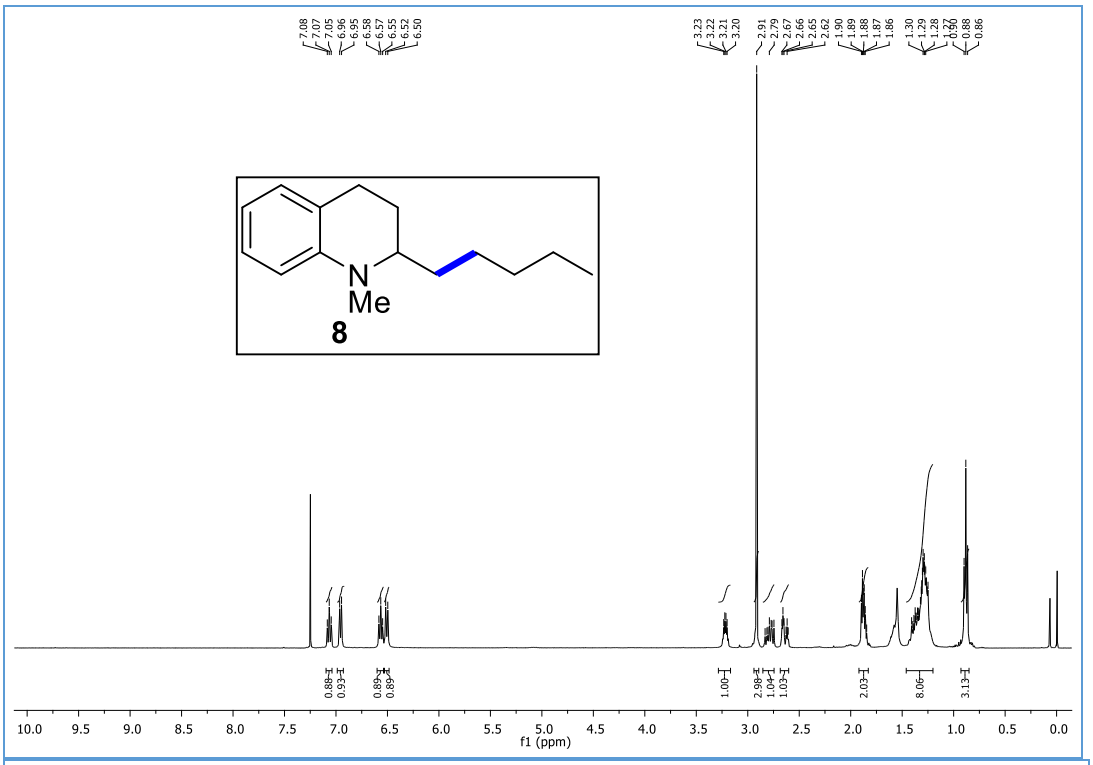
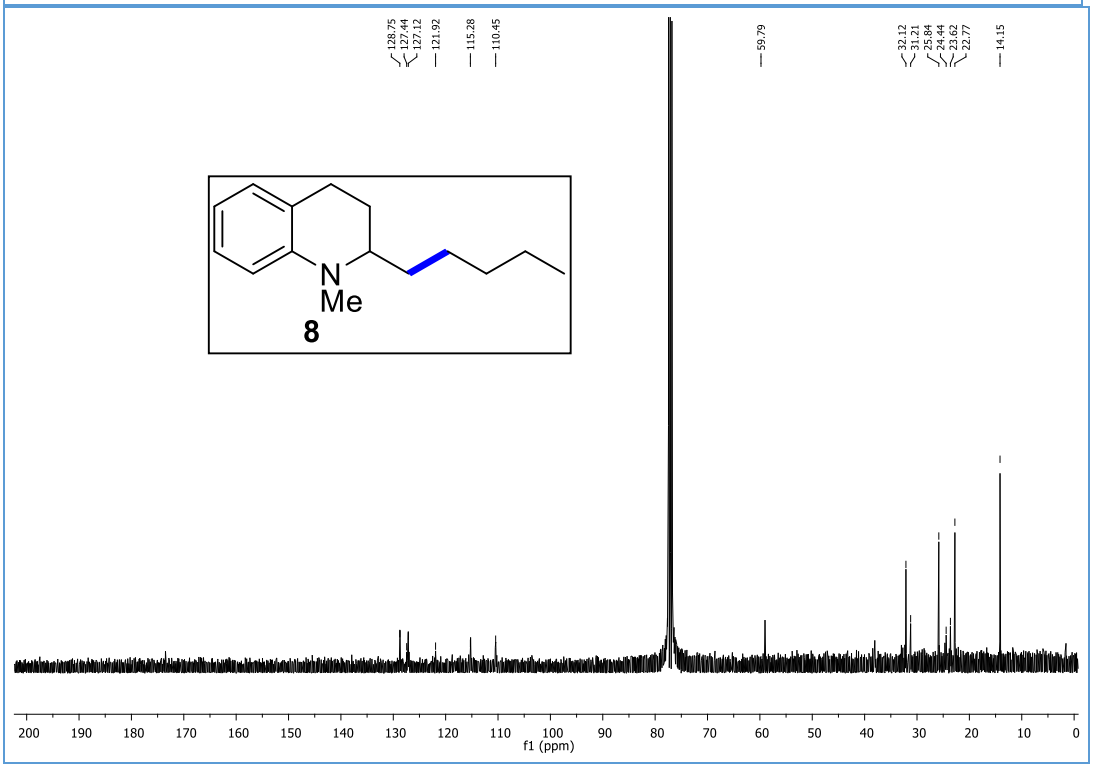
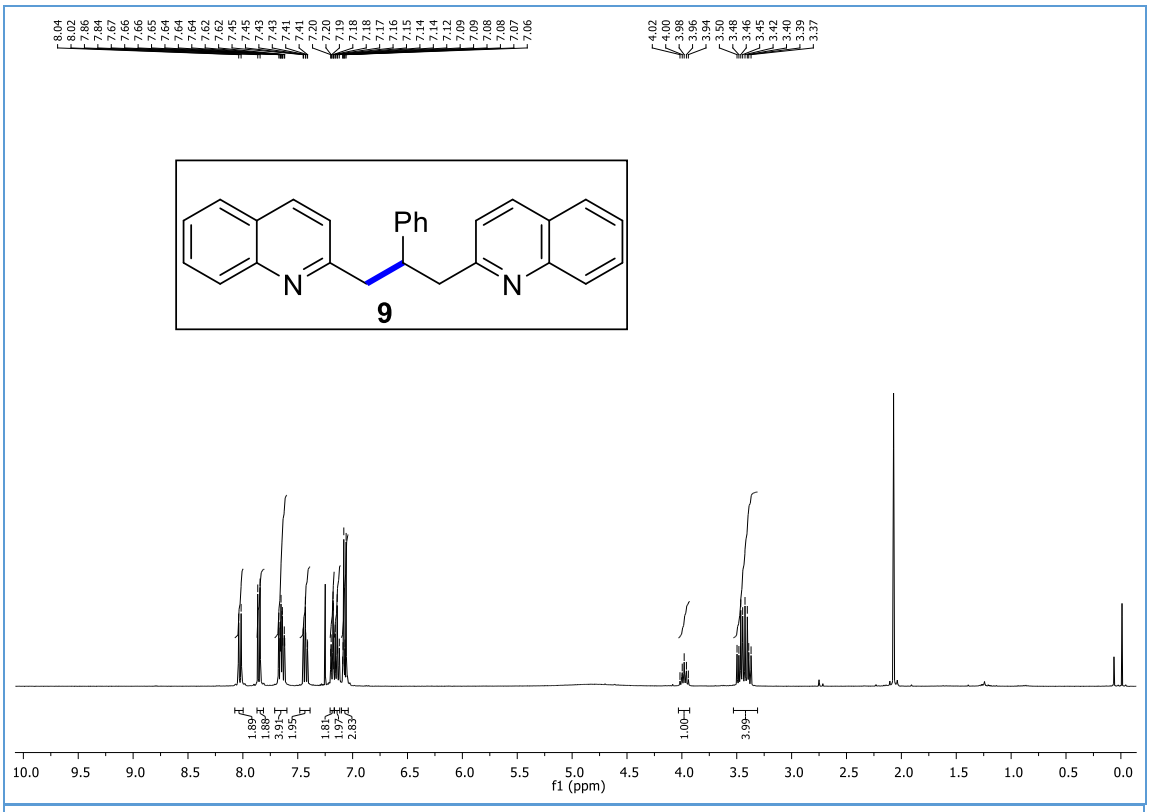
130
120
110
100
90
80
70
60
50
40
30
20
10
0
f1 (ppm)

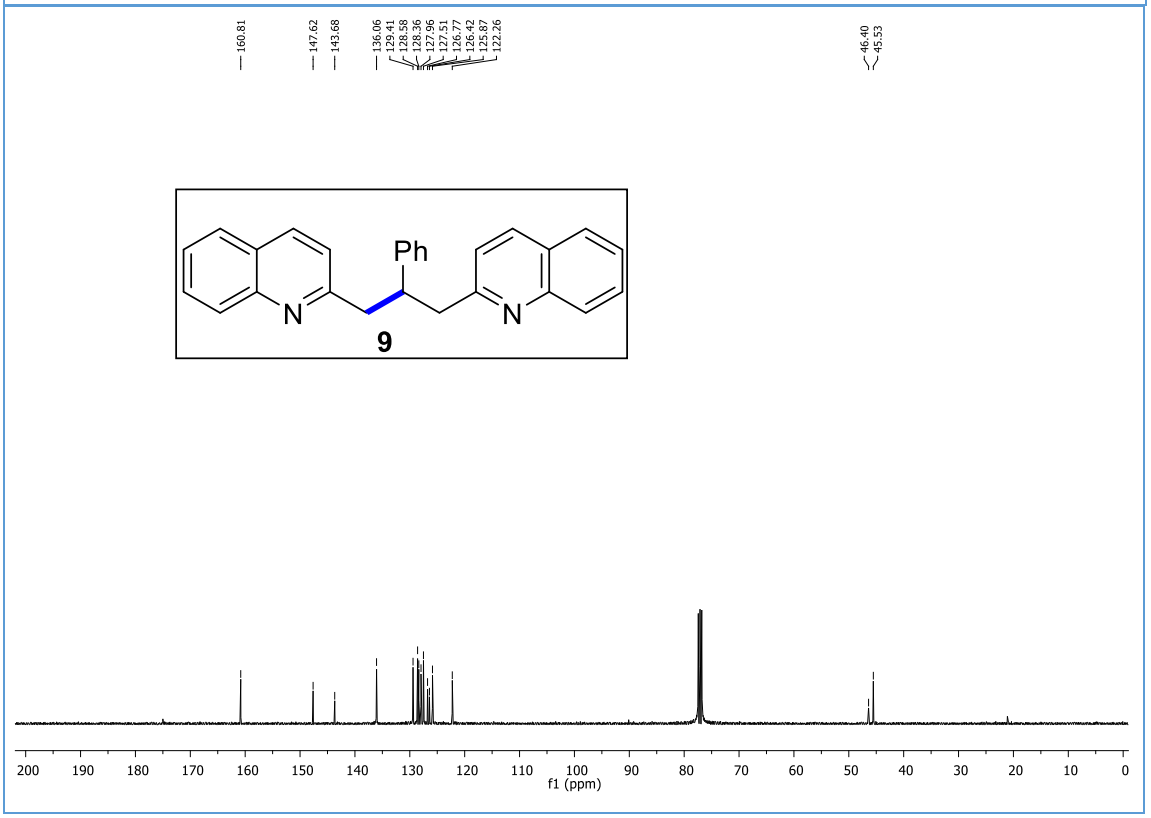

6.97 6.95 6.93 6.61 6.61 6.59 6.59 6.57 6.57 3.24 3.24 3.23 3.22 3.21 3.20 3.20 2.81 2.79 2.78 2.77 2.75 2.74 2.73 2.71 2.70 2.69 2.69 1.99 1.98 1.97 1.97 1.96 1.96 1.95 1.95 1.94 1.94 1.94 1.93 1.93 1.92 1.64 1.63 1.61 1.61 1.61 1.60 1.60 1.59 1.59 1.58 1.58 1.57 1.57 1.57 1.56 1.54 1.50 1.49 1.47 1.46 1.40 1.39 1.38 1.37 1.36 1.35 1.34 1.33 1.33 1.32 1.31 1.31 1.25 1.24 1.23 1.23 1.22 1.21 1.19 0.92 0.90 0.89
N
H
7
1.93 0.96 0.92 1.00 2.17 1.27 1.11 2.28 4.02 1.99 3.08
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)


143.93 128.43 125.87 120.60 116.07 113.22 50.79 35.87 31.15 27.32 25.63 24.59 21.83 13.24
N
H
7
200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0
f1 (ppm)


7.08
7.07
7.05
6.96
6.95
6.58
6.57
6.55
6.52
6.50
3.23
3.22
3.21
3.20
3.91
2.79
2.67
2.66
2.65
2.62
1.90
1.89
1.88
1.87
1.86
1.30
1.29
1.28
0.90
0.88
0.86
N
Me
8
0.88
0.93
0.89
0.89
1.00
2.98
1.04
1.03
2.03
8.06
3.13
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)


128.75
127.44
127.12
121.92
115.28
110.45
59.79
32.12
31.21
25.84
24.44
23.62
22.77
14.15
N
Me
8
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
8.02
7.86
7.84
7.67
7.66
7.66
7.65
7.64
7.64
7.62
7.62
7.45
7.45
7.43
7.43
7.41
7.41
7.20
7.19
7.18
7.18
7.17
7.16
7.15
7.14
7.12
7.09
7.09
7.08
7.08
7.07
7.06
4.02
4.00
3.98
3.96
3.94
3.50
3.48
3.46
3.45
3.42
3.40
3.39
3.37
Ph
N
N
9
1.89
1.88
3.91
1.95
1.81
1.97
2.83
1.00
3.99
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)


160.81
147.62
143.68
136.06
129.41
128.58
128.36
127.96
127.51
126.77
126.42
125.87
122.26
46.40
45.53
Ph
N
N
9
200
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
f1 (ppm)