Electronic Supplementary Material (ESI) for ChemComm.

Nickel-Catalyzed Direct α-Olefination of Alkyl Substituted *N*-Heteroarenes with Alcohols

Jagadish Das, Mari Vellakkaran and Debasis Banerjee*

Department of Chemistry
Laboratory of Catalysis and Organic Synthesis
Indian Institute of Technology Roorkee
Roorkee-247667, India
Email: dbane.fcy@iitr.ac.in

Table of Contents

[1.1] General Experimental Details:

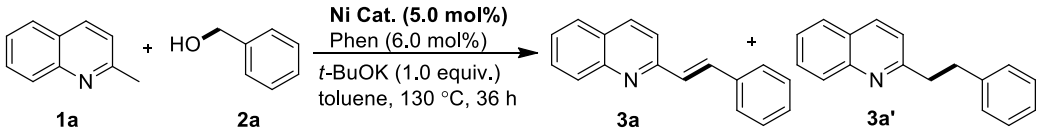
All solvents and reagents were used, as received from the suppliers. TLC was performed on Merck Kiesel gel 60, F_{254} plates with the layer thickness of 0.25 mm. Column chromatography was performed on silica gel (100-200 mesh) using a gradient of ethyl acetate and hexane as mobile phase. ^{1}H NMR spectral 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. All the reactions were performed in a close system using Schlenk tube. All nickel salts were purchased from Sigma Aldrich. Nickel(II) bromide (Assay- 98%; CAS Number 13462-88-9; EC Number 236-665-0; Pack Size- No 217891-10G). 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 Nickel Catalysed α-Olefination of Methylquinolines with Primary Alcohols:

In a 15 mL oven dried Schlenk tube, quinaldine (0.25 mmol), $NiBr_2$ (5 mol%), Phen (6 mol%), alcohols (0.50 mmol) and KOH (0.25 mmol), were added followed by toluene 2.0 mL under an atmosphere of N_2 and the reaction mixture was heated at 140 °C for 24 h in close 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] α-Olefination of 2-Methylquinolines with Alcohols:

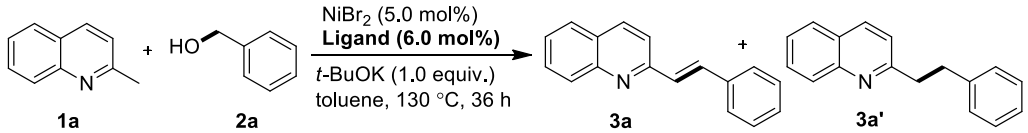
Table S1: Screening of catalyst[a]

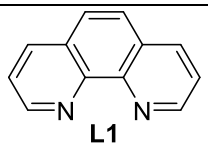
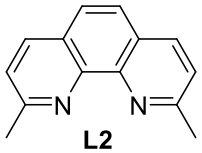
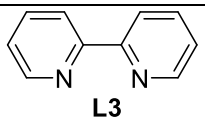
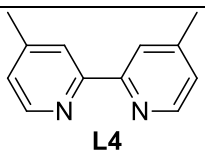


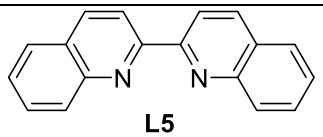
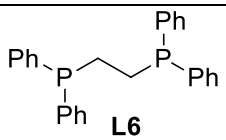
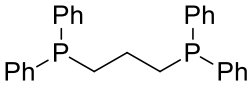
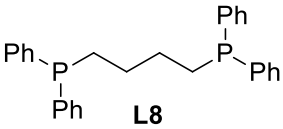
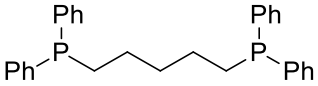
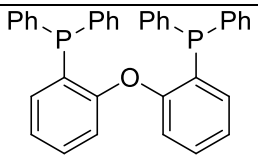
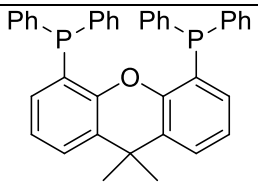
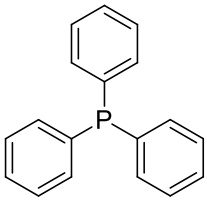
Entry	**Ni-Catalyst**	GC-MS Conversion **3a** (%)	Ratio **3a/3a'**
1	$NiCl_2$	49	1.5 : 1
2	**$NiBr_2$**	**65**	**13 : 1**
2	$Ni(acac)_2$	21	1.3 : 1
3	$NiCl_2(DME)$	49	5.4 : 1
4	$Ni(COD)_2$	21	3.5 : 1

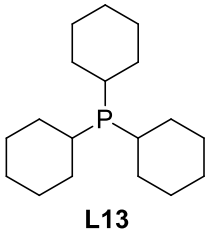
Reaction conditions: [a] Quinaldine **1a** (0.25 mmol), Benzyl alcohol **2a** (0.50 mmol), **Ni Cat. (5.0 mol%)**, Phen (6.0 mol%), *t*-BuOK (0.25 mmol), toluene (2.0 mL), Schlenk tube under nitrogen atmosphere, 130 ºC oil bath, 36 h reaction time.

Table S2: Screening of ligands [a]



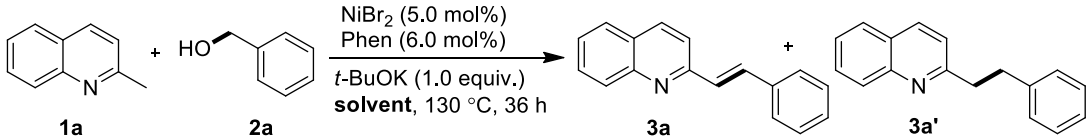
Entry	**Ligand**	GC-MS Conversion **3a** (%)	Ratio **3a/3a'**
1	 **L1**	**65**	**13 : 1**
2	 **L2**	15	7.5 : 1
3	 **L3**	15	-
4	 **L4**	43	-

5	 **L5**	10	-
6	 **L6**	12	-
7	 **L7**	30	15 : 1
8	 **L8**	28	-
9	 **L9**	34	17 : 1
10	 **L10**	17	8.5 : 1
11	 **L11**	15	15 : 1
12[b]	 **L12**	7	7 : 1

13[b]	 L13	20	4 : 1
14	No Ligand	22	2.8 : 1

Reaction conditions: [a] Quinaldine **1a** (0.25 mmol), Benzyl alcohol **2a** (0.50 mmol), $NiBr_2$ (5.0 mol%), **Ligand (6.0 mol%)**, *t*-BuOK (0.25 mmol), toluene (2.0 mL), Schlenk tube under nitrogen atmosphere, 130 ºC oil bath, 36 h reaction time. [b] 10 mol% of Ligand was used.

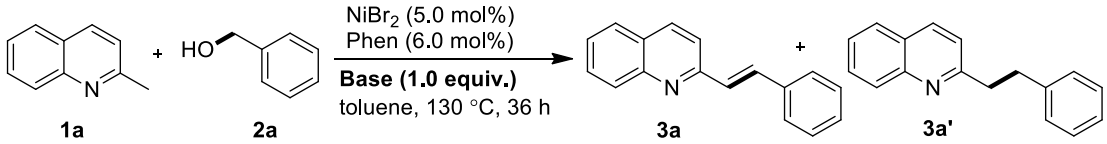
Table S3: Screening of solvents [a]



Entry	**Solvent**	GC-MS Conversion **3a** (%)	Ratio **3a/3a'**
1	**Toluene**	**65**	**13 : 1**
2	*p*-Xylene	40	1.2 : 1
3	1,4-Dioxane	5	5 : 1
4	*n*-BuOH	10	10 : 1
5	*t*-Amylalcohol	11	11 : 1

Reaction conditions: [a] Quinaldine **1a** (0.25 mmol), Benzyl alcohol **2a** (0.50 mmol), $NiBr_2$ (5.0 mol%), Phen (6.0 mol%), *t*-BuOK (0.25 mmol), **solvent (2.0 mL)**, Schlenk tube under nitrogen atmosphere, 130 ºC oil bath, 36 h reaction time.

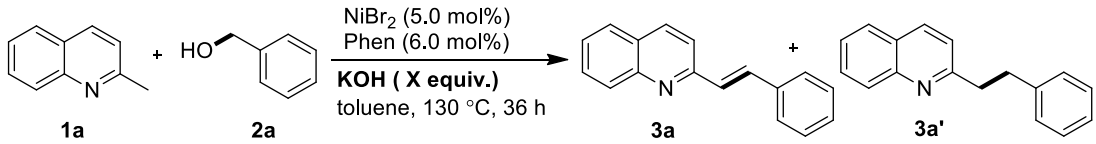
Table S4: Screening of base [a]



Entry	**Base**	GC-MS Conversion **3a** (%)	Ratio **3a/3a'**
1	***t*-BuOK**	**65**	**13 : 1**
2	*t*-BuONa	62	15 : 1
3	Cs_2CO_3	1	1 : 1
4	Na_2CO_3	6	3 : 1
5	NaOH	70	17 : 1
6	**KOH**	**81 (78)**[b]	**5.7 : 1**

Reaction conditions: [a] Quinaldine **1a** (0.25 mmol), Benzyl alcohol **2a** (0.50 mmol), $NiBr_2$ (5.0 mol%), Phen (6.0 mol%), **Base (0.25 mmol)**, toluene (2.0 mL), Schlenk tube under nitrogen atmosphere, 130 ºC oil bath, 36 h reaction time. [b] Isolated yield average of two run.

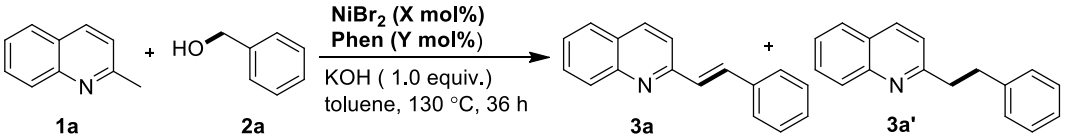
Table S5: Screening of base equivalents [a]



Entry	**Base Equivalent (X equiv.)**	GC-MS Conversion **3a** (%)	Ratio **3a/3a'**
1	**KOH (1.0 equiv.)**	**81 (78)**[b]	**5.7 : 1**
2	KOH (0.75 equiv.)	65	7.2 : 1
3	KOH (0.50 equiv.)	41	-
4	-	0	-

Reaction conditions: [a] Quinaldine **1a** (0.25 mmol), Benzyl alcohol **2a** (0.50 mmol), $NiBr_2$ (5.0 mol%), Phen (6.0 mol%), KOH (**X equiv.**), toluene (2.0 mL), Schlenk tube under nitrogen atmosphere, 130 ºC oil bath, 36 h reaction time. [b] Isolated yield average of two run.

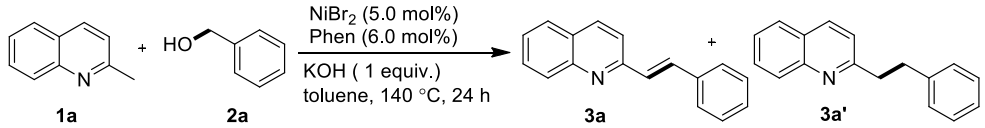
Table S6: Screening of catalyst and ligand loading [a]



Entry	**Cat. (X mol%)**	**Ligand (Y mol%)**	GC-MS Conversion **3a** (%)	Ratio **3a/3a'**
1	**$NiBr_2$ (5.0)**	**Phen (6.0)**	**81 (78)**[b]	**5.7 : 1**
2	$NiBr_2$ (2.5)	Phen (3.0)	76 (74)[b]	>20 : 1
3[c]	**$NiBr_2$ (5.0)**	**Phen (6.0)**	**85 (83)**[b]	**>20 : 1**
4	-	-	20	-

Reaction conditions: [a] Quinaldine **1a** (0.25 mmol), Benzyl alcohol **2a** (0.50 mmol), **$NiBr_2$ (X mol%), Phen (Y mol%)**, KOH (0.25 mmol), toluene (2.0 mL), Schlenk tube under nitrogen atmosphere, 130 ºC oil bath, 36 h reaction time. [b] Isolated yield average of two run. [c] 140 ºC, 24 h reaction time.

Table S7: Screening of alcohol equivalents [a]

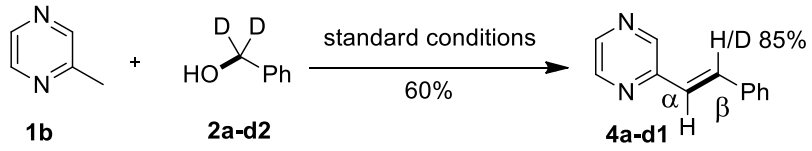


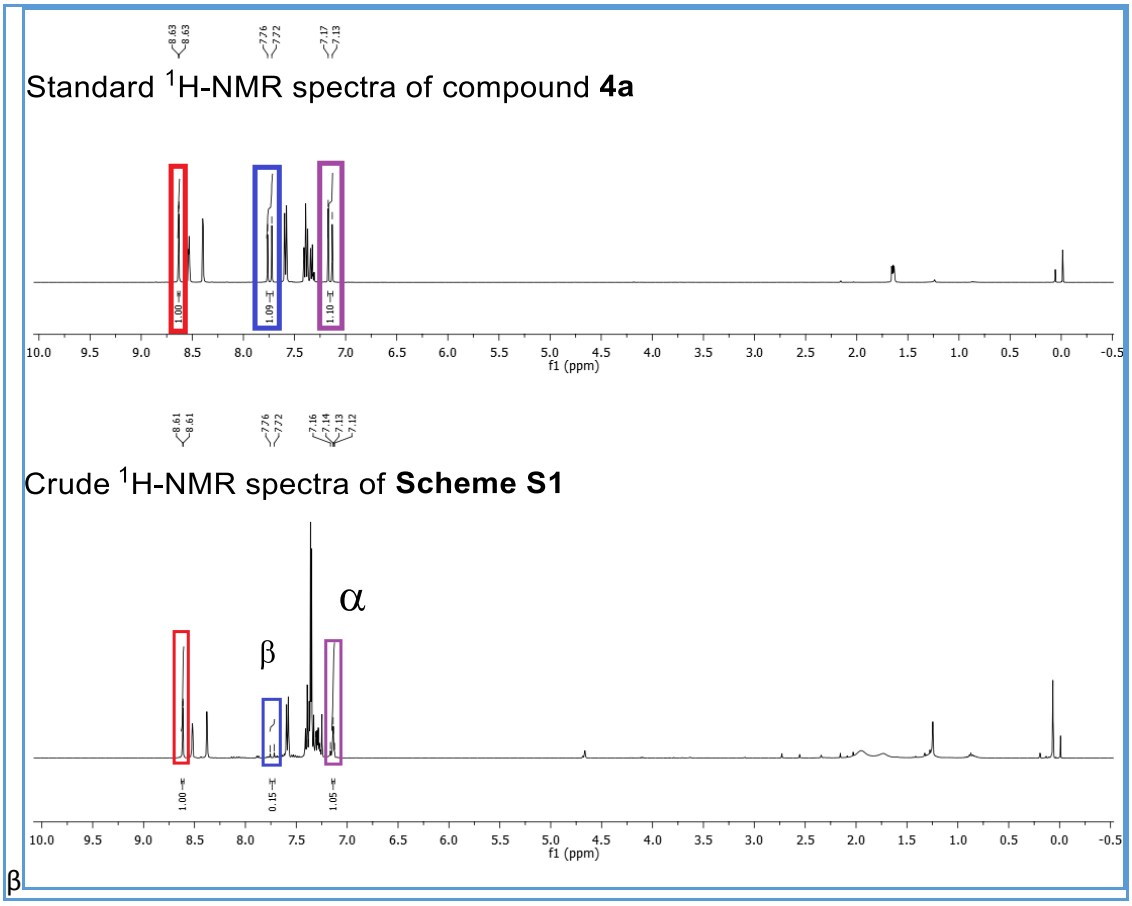
Entry	**Benzyl Alcohol Equivalent (X equiv.)**	GC-MS Conversion **3a** (%)	Ratio **3a/3a'**
1	**2.0 equiv.**	**85 (83)**[b]	**>20 : 1**
2	1.5 equiv.	65	-
3	1.0 equiv.	40	-

Reaction conditions: [a] Quinaldine **1a** (0.25 mmol), Benzyl alcohol **2a** (x mmol), $NiBr_2$ (5.0 mol%), Phen (6.0 mol%), KOH (1.0 equiv.), toluene (2.0 mL), Schlenk tube under nitrogen atmosphere, 140 ºC oil bath, 24 h reaction time. [b] Isolated yield average of two run.

Deuterium incorporation experiments

Scheme S1:

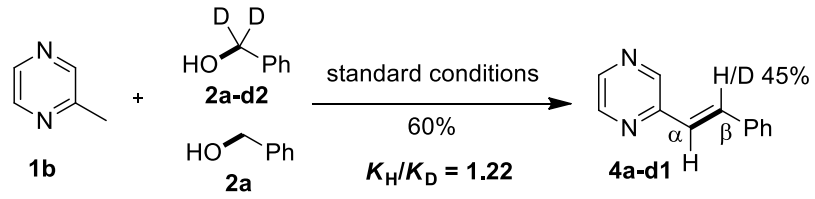


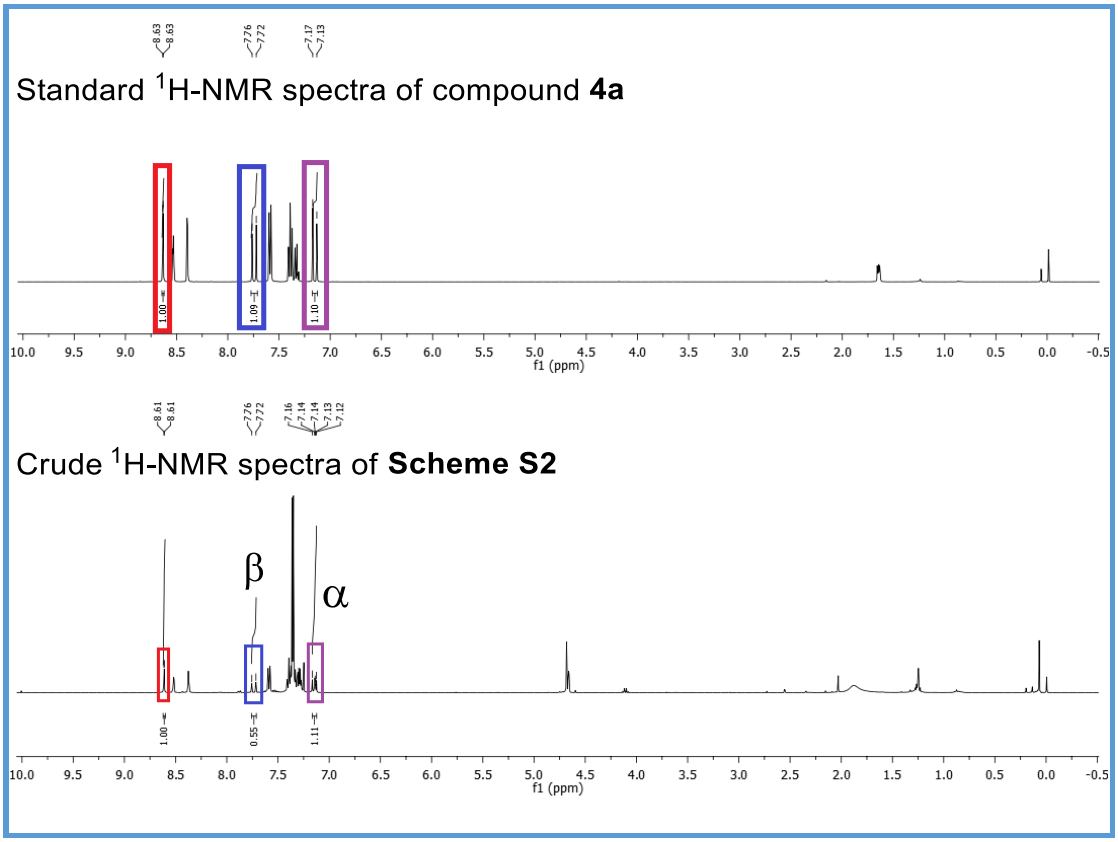


Conversion was calculated by ^{1}H-NMR integration value

		Deuterium incorporation in β position	Deuterium incorporation in α position
Signal δ ppm	8.63 [1H)]	7.74 (1H)	7.15 (1H)
Integral Value	1.0	0.15	1.05
Calculated ratio		{(1-0.15)/1}×100 = **85%**	{(1-1)/1}×100 = **0%**

Scheme S2:

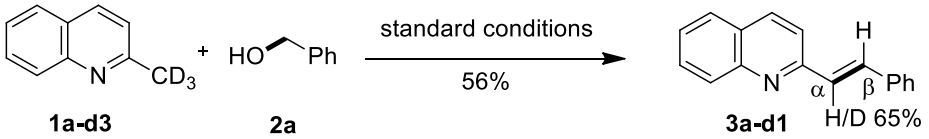


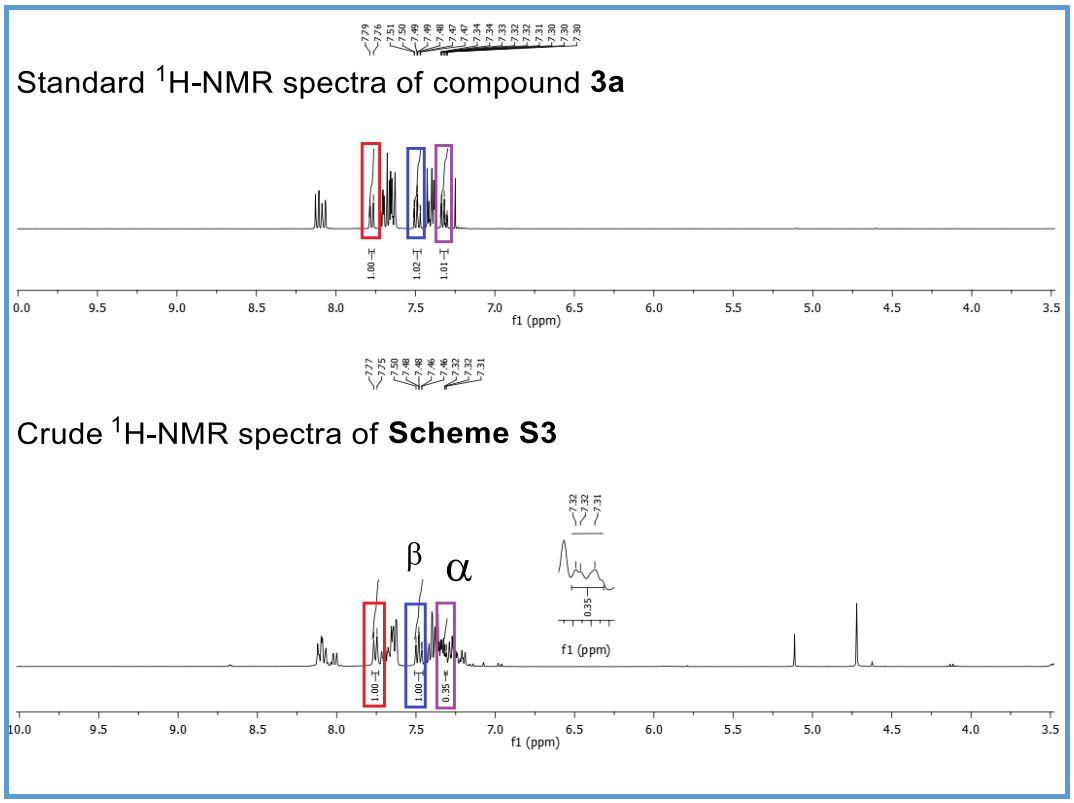


Conversion was calculated by ^{1}H-NMR integration value

		Deuterium incorporation in β position	Deuterium incorporation in α position
Signal δ ppm	8.63 [1H)]	7.74 (1H)	7.15 (1H)
Integral Value	1.0	0.55	1.11
Calculated ratio		{(1-0.55)/1}×100 = **45%**	{(1-1)/1}×100 = **0%**

Scheme S3:

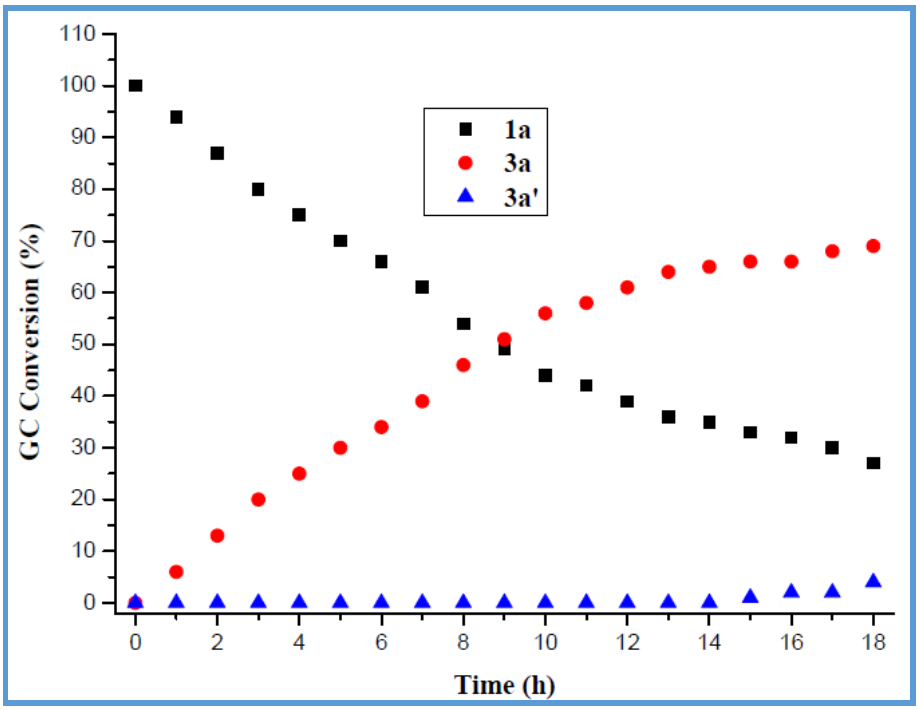




Conversion was calculated by ^{1}H-NMR integration value

		Deuterium incorporation in β position	Deuterium incorporation in α position
Signal δ ppm	7.76 [1H)]	7.49 (1H)	7.32 (1H)
Integral Value	1.0	1.0	0.35
Calculated ratio		{(1-1)/1}×100 = **0%**	{(1-0.35)/1}×100 = **65%**

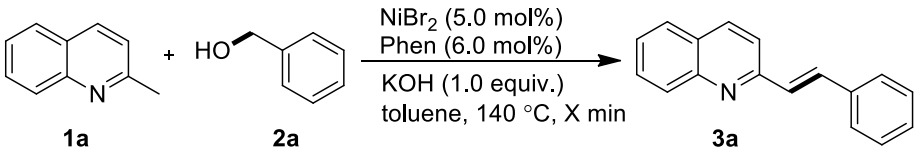
Scheme S4: Time-conversion-plot for the reaction of 2-methylquinoline (**1a**) with benzyl alcohol (**2a**)



Reaction conditions: Quinaldine **1a** (0.25 mmol), Benzyl alcohol **2a** (0.50 mmol), $NiBr_2$ (5.0 mol%), Phen (6.0 mol%), KOH (0.25 mmol), toluene (2.0 mL), Schlenk tube under nitrogen atmosphere, 140 °C oil bath.

Scheme S5: Determination of rate and order of reaction

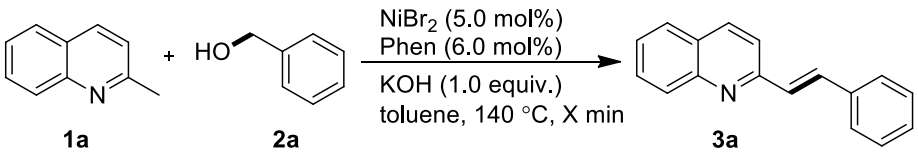
Run 1: Reaction was carried out in 2 mL of toluene and yield was calculated by GC



No.	**1a** (mmol)	**2a** (mmol)	$NiBr_2$ (mmol)	Phen (mmol)	KOH (mmol)	toluene (mL)
Run 1	0.2	0.4	0.01	0.012	0.2	2.0

Sl. No.	Time (min)	Concentration of **3a** (mM)
1	0	0
2	30	2
3	60	5
4	90	8
5	120	9.5
6	150	12
7	180	15
8	210	18
9	240	20
10	270	22
11	300	24

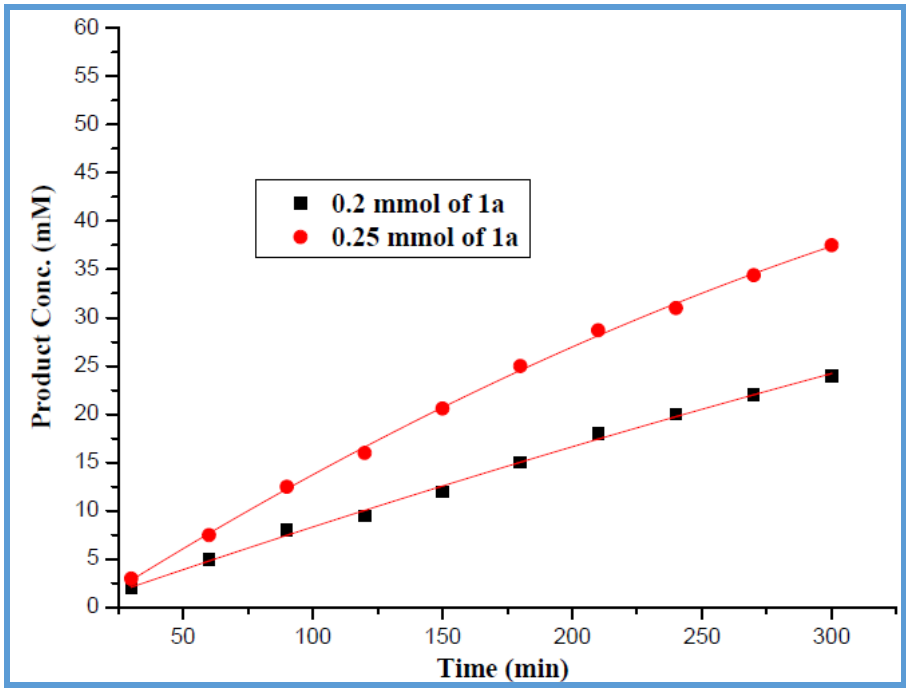
Run 2: Reaction was carried out in 2 mL of toluene and yield was calculated by GC



1a **2a** **3a**

No.	**1a** (mmol)	**2a** (mmol)	$NiBr_2$ (mmol)	Phen (mmol)	KOH (mmol)	toluene (mL)
Run 2	0.25	0.5	0.0125	0.015	0.25	2.0

Sl. No.	Time (min)	Concentration of **3a** (mM)
1	0	0
2	30	3
3	60	7.5
4	90	12.5
5	120	16
6	150	20.6
7	180	25
8	210	28.7
9	240	31
10	270	34.4
11	300	37.5



Graphical representation for determination of rate and order of reaction

Considering steady state approximation for benzyl alcohol

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

$0.082 = k\,[0.20]^{x}$

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

$0.129 = k\,[0.25]^{x}$

$0.129/0.082 = [0.25]^{x}/\,[0.2]^{x}$

$1.57 = [1.25]^{x}$

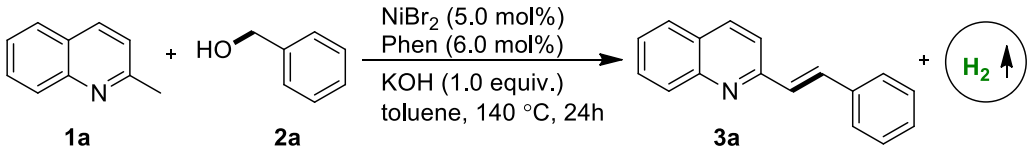
$\text{Log}\,(1.57) = x.\ \text{Log}\,(1.25)$

$x = 0.195 / 0.0969$

$= 2.01 \approx 2$

Rate = k [1a] ^{2}

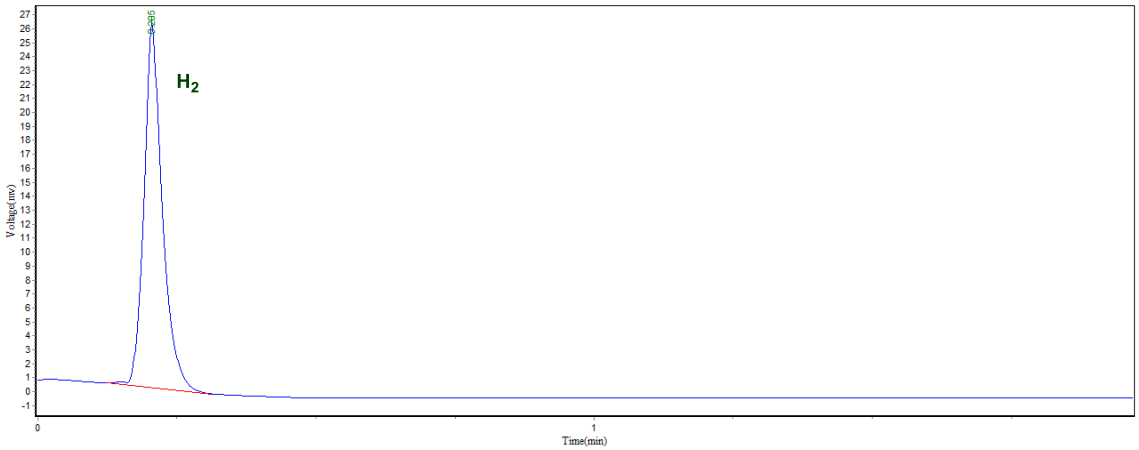
Scheme S6: Detection of H_2 gas liberation.



In a 100 mL oven dried Ace Pressure tube, quinaldine (3.0 mmol), $NiBr_2$ (5 mol%), Phen (6 mol%), alcohols (6.0 mmol) and KOH (3.0 mmol), were added followed by toluene 10.0 mL under an atmosphere of N_2 and the reaction mixture was sealed with septum and heated at 140 °C for 24 h. After completion of reaction H_2 gas was detected by Centurion Scientific Gas Chromatograph (CS-5700$^+$) through TCD Detector.

Date/Time: 2018-09-27, 12:50:42 PM
Data File: D:\CS200\TCD\genet\MARI.org
Method File: D:\CS200\TCD\genet\TCD NEW.mtd

Analyst:
Date/Time: 2018-09-27, 12:51:05 PM



Results

Peak No.	Peak ID	Ret Time	Height	Area	Conc.
1	H2	0.205	24767.684	56138.602	100.0000
Total					100.0000

Fig. 1: <u>Crystallographic data for compound 3b</u>

Identification code:	UPS3491DB_MVB110_0m_a		
CCDC	1871614		
Bond precision:	C-C = 0.0076 A		Wavelength = 0.71073
Cell:	a = 5.9624(3)	b = 8.0092(4)	c = 28.9234(16)
	alpha = 90	beta = 90	gamma = 90
Temperature:	296 K		

	Calculated	Reported
Volume	1381.21(12)	1381.21(12)
Space group	P 21 21 21	P 21 21 21
Hall group:	P 2ac 2ab	P 2ac 2ab
Moiety formula	C_{18} H_{15} N	
Sum formula	C_{18} H_{15} N	C_{18} H_{15} N
Mr	245.31	245.31
Dx, g cm-3	1.180	1.180
Z	4	4
Mu (mm-1)	0.068	0.068
F000	520.0	520.0
F000'	520.18	
h,k,l max	7,10,38	7,10,38
Nref	3436[2018]	3417
Tmin,Tmax	0.985, 0.985	0.985,0.985
Tmin'	0.985	

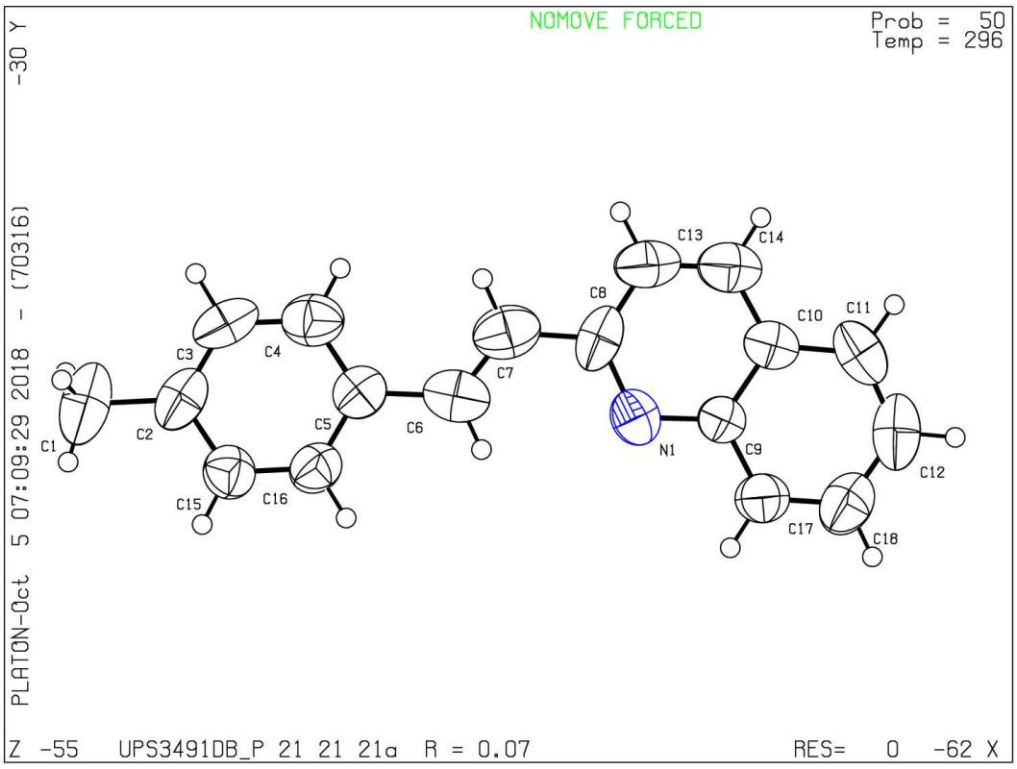
Correction method = # Reported T Limits: Tmin = 0.985 Tmax = 0.985

AbsCorr = MULTI-SCAN

Data completeness = 1.69/0.99 Theta (max) = 28.315

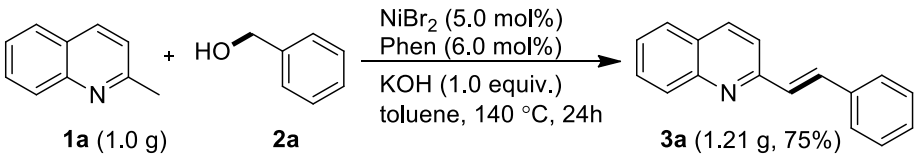
R (reflections) = 0.0684 (1657) wR2 (reflections) = 0.2304 (3417)

S = 0.955 Npar = 173



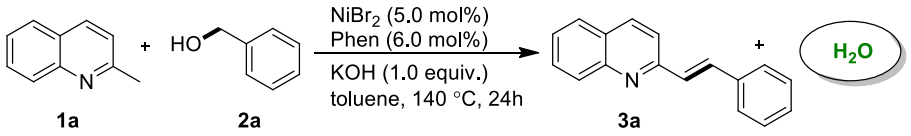
Platon-ellipsoid plot for compound 3b

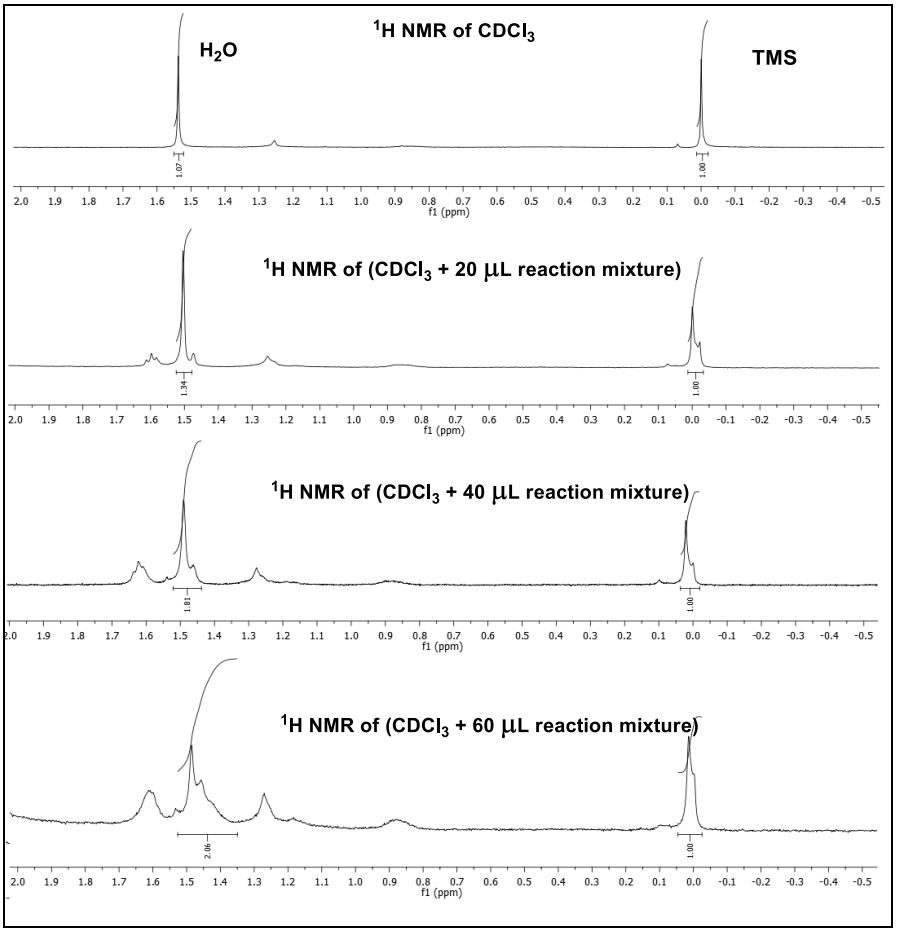
Scheme S7: Gram Scale Reaction.



Gram Scale reaction was performed using quinaldine (1.0 g, 6.99 mmol), benzyl alcohol (1.509 g, 13.98 mmol), $NiBr_2$ (76 mg, 5 mol%), Phen (76 mg, 6 mol%), KOH (391 mg, 6.99 mmol), toluene (15.0 mL) in a 100 mL pressure tube under nitrogen atmosphere at 140 ºC in oil bath for 24 h. The reaction mixture was cooled to room temperature and 15.0 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 **3a** (1.21 g, 75% yield).

Scheme S8: Detection of water in reaction mixture by ^{1}H-NMR

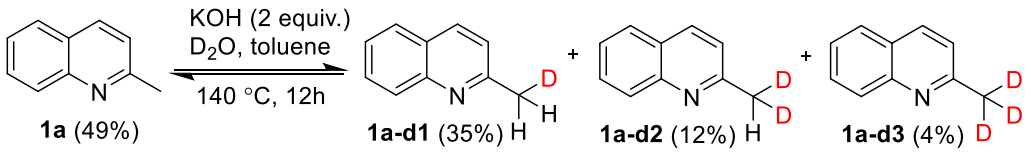




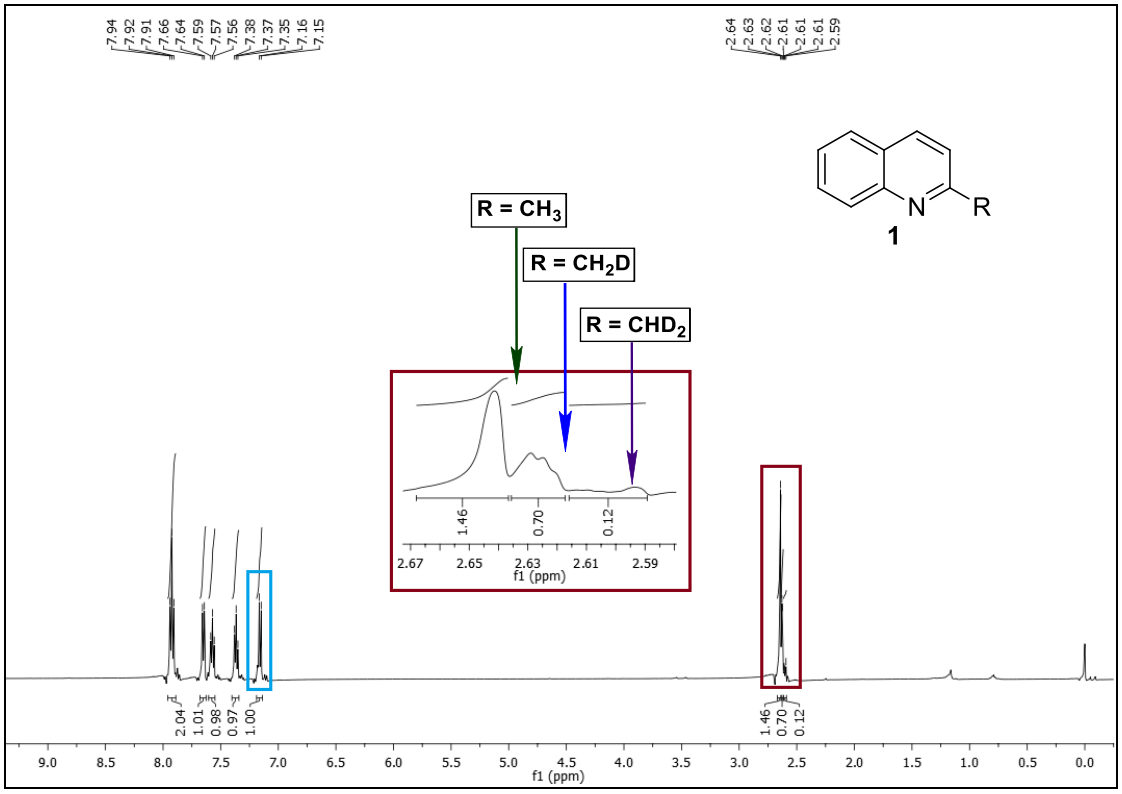
Reaction conditions: Quinaldine **1a** (0.25 mmol), Benzyl alcohol **2a** (0.50 mmol), $NiBr_2$ (5.0 mol%), Phen (6.0 mol%), KOH (0.25 mmol), toluene (2.0 mL), Schlenk tube under nitrogen atmosphere, 140 °C oil bath, 24 h.

In a 15 mL oven dried Schlenk tube, quinaldine **1a** (0.25 mmol), $NiBr_2$ (5 mol%), Phen (6 mol%), benzyl alcohol **2a** (0.50 mmol) and KOH (0.25 mmol), were added followed by toluene (dry) 2.0 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 1:1 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 S9: Evidence for the enamine intermediate formation



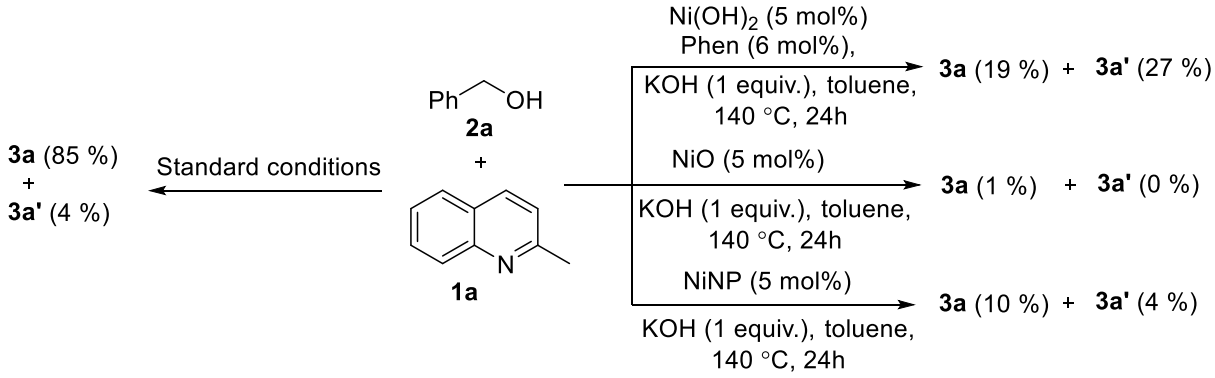
Reaction conditions: Quinaldine **1a** (0.25 mmol), **D_2O** (0.2 mL), KOH (0.5 mmol), toluene (2.0 mL), Schlenk tube under nitrogen atmosphere, 140 °C oil bath, 12 h.



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

		1a	**1a-d1**	**1a-d2**	**1a-d3**
Signal δ ppm	7.15 (1H)	2.64 (3H)	2.62 (2H)	2.59-2.61 (1H)	
Integral Value	1.0	1.46	0.70	0.12	
Calculated ratio		(1.46 / 3)×100 = **49%**	(0.70 / 2)×100 = **35%**	(0.12 / 1)×100 = **12%**	100-(49+35+12) = **4%**

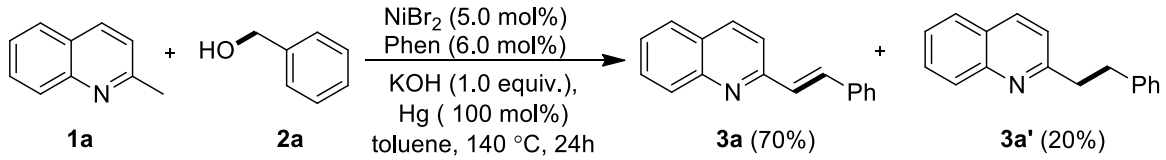
Scheme S10: Test for homogeneity experiments.



Reaction conditions: Quinaldine **1a** (0.25 mmol), Benzyl alcohol **2a** (0.50 mmol), Ni-Cat. (5.0 mol%), Phen (6.0 mol%), KOH (0.25 mmol), toluene (2.0 mL), Schlenk tube under nitrogen atmosphere, 140 °C oil bath, 24 h.

Moreover, to gain the additional proof for the homogeneous nature of the nickel-catalyst and exclude the involvement of the heterogeneous nickel-catalysts, such as, $Ni(OH)_2$, NiO and Ni-based nanoparticles, we conducted several experiments using model reactions and observed only trace amount or poor product conversion to **3a**. Notably, it is also evident that, presence of KOH base responsible for such poor product formation and there is no involvement of the heterogeneous Ni-catalysts. Thereafter, when the reaction was performed using mercury, commonly known as poison for heterogeneous catalysts, we observed 70% conversion to product **3a** (Scheme S11). These experiments strongly support the homogeneous nature of the present catalytic system.

Scheme S11: Test for catalyst poisoning experiment



In a 15 mL oven dried Schlenk tube, quinaldine (0.25 mmol), $NiBr_2$ (5 mol%), Phen (6 mol%), benzyl alcohol (0.50 mmol) and KOH (0.25 mmol), were added followed by toluene 2.0 mL. Then Hg (50 mg, 100 mol%) was added to the mixture and flushed with N_2 four times, the reaction mixture was heated at 140 °C for 24 h in close system. The reaction mixture was cooled to room temperature and 3.0 mL of ethyl acetate was added and analyzed by GC-MS. Product **3a** (70%) and reduced product **3a'**(20%) was observed in GC-MS

analysis of crude reaction mixture which eliminates the probability of a heterogeneous reaction.

Scheme S12: Quantitative determination of hydrogen gas produced in the reaction

In a 10 mL oven dried Schlenk tube, quinaldine (0.5 mmol), $NiBr_2$ (5 mol%), Phen (6 mol%), benzyl alcohol (1.0 mmol) and KOH (0.5 mmol), were added followed by toluene 4.0 mL and connected to the gas burette as shown in below figure. Then the reaction mixture was heated at 140 °C until the production of hydrogen gas ceased. The procedure was repeated three times to get concordant reading.

Total volume of water displaced, V = 0.0176 L

Vapor pressure of water at 298K, P_{H2O} = 23.7695 Torr

Atmospheric pressure at 298K, P_{atm} = 758.3124 Torr

Pressure of H_2 gas, $P_{H2} = P_{atm} - P_{H2O}$ = (758.3124 - 23.7695) Torr = 734.5429 Torr

$P_{H2} * V = nH_2 * R * T$

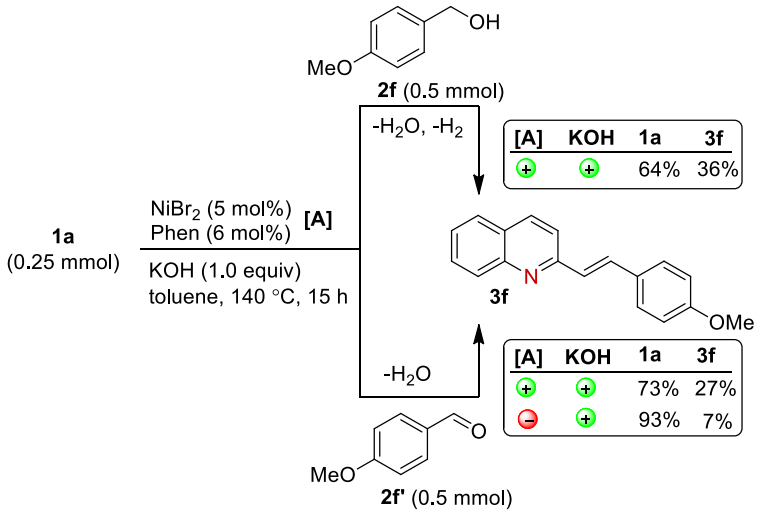
$nH_2 = P_{H2} * V / R * T$

= 734.5429 Torr * 0.0176 L / 62.3635 L Torr K^{-1} mol^{-1} * 298K

= 0.000696 mol

≈ 0.70 mmol

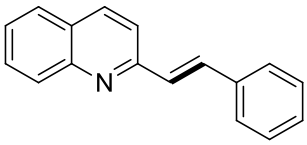
Scheme S13: Control experiments for α-olefination.



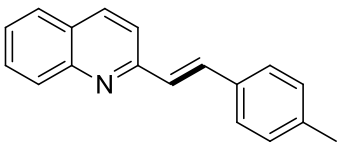
We explored our interests towards the reaction mechanism for the olefination process. Therefore, a series of experiments were performed using **1a** with 4-methoxy benzaldehyde as well as 4-methoxy benzylalcohol **2f** in presence and absence of nickel catalyst for 15 h (Scheme S13). When 4-methoxybenzaldehyde subjected to olefination with **1a** under standard conditions using nickel resulted **3f** in 27% yield. However, under identical conditions in absence of nickel, **3f** was obtained in 7% yield. Interestingly, under optimized conditions, similar reaction using 4-methoxybenzylalcohol **2f** gave 36% of the product **3f**. These experimental outcomes are in agreement with the participation of nickel catalyst for alcohol dehydrogenation as well as crucial for C-C bond forming condensation process. Nevertheless, either in absence of catalyst and KOH or in absence of KOH, 4-methoxybenzaldehyde did not result any desired product.

[1.4] Spectroscopic and Analytical Data:

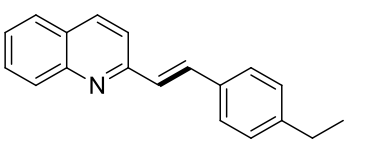
(*E*)-2-Styrylquinoline (3a)[1]: Following the general procedure, the title compound was isolated as a white solid (48 mg, Yield: 83%). ^{1}H NMR (400 MHz, $CDCl_3$) δ 8.10 (dd, J = 16.4, 8.6 Hz, 2H), 7.78 (dd, J = 8.1, 1.1 Hz, 1H), 7.71 (dd, J = 6.5, 2.0 Hz, 1H), 7.65 (ddd, J = 8.0, 7.3, 3.0 Hz, 4H), 7.49 (ddd, J = 8.1, 6.9, 1.1 Hz, 1H), 7.43 – 7.38 (m, 3H), 7.34 – 7.30 (m, 1H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 156.09, 148.36, 136.61, 136.43, 134.52, 129.83, 129.30, 129.12, 128.88, 128.72, 127.58, 127.44, 127.35, 126.26, 119.35.



(*E*)-2-(4-Methylstyryl)quinoline (3b)[1]: Following the general procedure, the title compound was isolated as a white solid (53 mg, Yield: 86%). ^{1}H NMR (400 MHz, $CDCl_3$) δ 8.08 (dd, J = 16.0, 8.5 Hz, 2H), 7.77 (d, J = 8.2 Hz, 1H), 7.68 (ddd, J = 23.1, 11.5, 4.1 Hz, 3H), 7.54 (d, J = 8.1 Hz, 2H), 7.48 (ddd, J = 8.0, 7.0, 1.1 Hz, 1H), 7.36 (d, J = 16.3 Hz, 1H), 7.20 (d, J = 7.9 Hz, 2H), 2.37 (s, 3H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 156.30, 148.31, 138.88, 136.42, 134.53, 133.80, 129.83, 129.64, 129.19, 128.09, 127.60, 127.37, 127.31, 126.17, 119.28, 21.49.



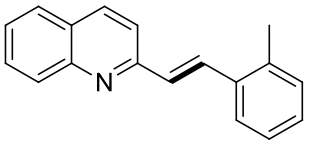
(*E*)-2-(4-Ethylstyryl)quinoline (3c): Following the general procedure, the title compound was isolated as a white solid (36 mg, Yield: 56%). ^{1}H NMR (400 MHz, $CDCl_3$) δ 8.09 (dd, J = 15.3, 8.6 Hz, 2H), 7.77 (d, J = 8.1 Hz, 1H), 7.72 – 7.63 (m, 3H), 7.56 (dd, J = 8.2, 2.0 Hz, 2H), 7.50 – 7.46 (m, 1H), 7.37 (dd, J = 16.3, 2.3 Hz, 1H), 7.23 (dd, J = 8.1, 1.8 Hz, 2H), 2.70 – 2.64 (m, 2H), 1.26 (t, J = 7.6 Hz, 3H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 155.22, 147.25, 144.09, 135.25, 133.42, 132.99, 128.68, 128.13, 127.32, 127.09, 126.47, 126.28, 126.27, 125.03, 118.16, 27.71, 14.41. HRMS (ESI): Calculated for $[C_{19}H_{18}N]^{+}$ 260.1434; Found 260.1429.



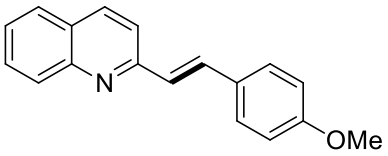
(*E*)-2-(4-Isopropylstyryl)quinoline (3d): Following the general procedure, the title compound was isolated as a white solid (46 mg, Yield: 67%). ^{1}H NMR (400 MHz, $CDCl_3$) δ 8.09 (dd, J = 17.8, 8.5 Hz, 2H), 7.77 (dd, J = 8.1, 1.2 Hz, 1H), 7.71 – 7.63 (m, 3H), 7.57 (d, J = 8.2 Hz, 2H), 7.48 (ddd, J = 8.0, 6.9, 1.2 Hz, 1H), 7.37 (d, J = 16.4 Hz, 1H), 7.26 (d, J = 8.8 Hz, 2H), 2.93 (dt, J = 13.9, 6.9 Hz, 1H), 1.27 (d, J = 6.9 Hz, 6H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 156.26, 149.73, 148.30, 136.28, 134.44, 134.18, 129.71,

129.17, 128.18, 127.51, 127.34, 127.31, 126.93, 126.07, 119.18, 34.02, 23.90. HRMS (ESI): Calculated for $[C_{20}H_{20}N]^+$ 274.1590; Found 274.1582.

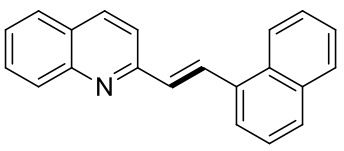
(*E*)-2-(2-Methylstyryl)quinoline (3e)[2]: Following the general procedure, the title compound was isolated as a colorless oil (48 mg, Yield: 78%). ^{1}H NMR (400 MHz, $CDCl_3$) δ 8.10 (dd, J = 13.8, 8.6 Hz, 2H), 7.93 (d, J = 16.2 Hz, 1H), 7.79 – 7.76 (m, 1H), 7.75 – 7.66 (m, 3H), 7.49 (ddd, J = 8.1, 7.0, 1.1 Hz, 1H), 7.32 (d, J = 16.2 Hz, 1H), 7.27 – 7.20 (m, 3H), 2.52 (s, 3H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 156.30, 148.32, 136.69, 136.47, 135.56, 132.18, 130.69, 130.26, 129.86, 129.31, 128.60, 127.61, 127.43, 126.44, 126.28, 125.89, 119.41, 20.16.



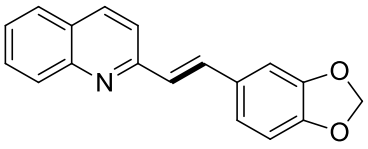
(*E*)-2-(4-Methoxystyryl)quinoline (3f)[1]: Following the general procedure, the title compound was isolated as a white solid (49.5 mg, Yield: 76%). ^{1}H NMR (400 MHz, $CDCl_3$) δ 8.07 (t, J = 9.4 Hz, 2H), 7.76 (dd, J = 8.1, 1.2 Hz, 1H), 7.71 – 7.64 (m, 2H), 7.62 (d, J = 4.0 Hz, 1H), 7.60 – 7.56 (m, 2H), 7.47 (ddd, J = 8.1, 7.0, 1.1 Hz, 1H), 7.28 (d, J = 16.3 Hz, 1H), 6.95 – 6.91 (m, 2H), 3.84 (s, 3H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 160.18, 156.43, 148.33, 136.38, 134.14, 129.81, 129.34, 129.13, 128.77, 127.60, 127.29, 126.87, 126.03, 119.21, 114.30, 55.45.



(*E*)-2-(2-(Naphthalen-1-yl)vinyl)quinoline (3g)[2]: Following the general procedure, the title compound was isolated as a yellow oil (50 mg, Yield: 71%). ^{1}H NMR (400 MHz, $CDCl_3$) δ 8.52 (d, J = 16.0 Hz, 1H), 8.34 (d, J = 8.4 Hz, 1H), 8.14 (dd, J = 12.5, 8.5 Hz, 2H), 7.91 – 7.85 (m, 3H), 7.80 (d, J = 8.0 Hz, 1H), 7.76 – 7.70 (m, 2H), 7.60 – 7.45 (m, 5H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 156.13, 148.38, 136.57, 134.12, 133.84, 131.83, 131.57, 131.46, 129.92, 129.39, 129.09, 128.80, 127.65, 127.52, 126.46, 126.36, 126.08, 125.84, 124.31, 123.85, 119.67.

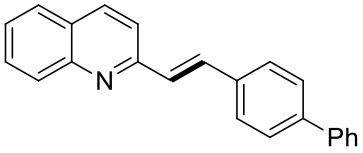


(*E*)-2-(2-(Benzo[*d*][1,3]dioxol-5-yl)vinyl)quinoline (3h)[1]: Following the general procedure, the title compound was isolated as a white solid (40 mg, Yield: 58%). ^{1}H NMR (500 MHz, $CDCl_3$) δ 8.14 (d, J = 8.6 Hz, 1H), 8.09 (d, J = 8.5 Hz, 1H), 7.80 (d, J = 7.9 Hz, 1H), 7.75 – 7.71 (m, 1H), 7.65 (t, J = 12.3 Hz, 2H), 7.53 – 7.49 (m, 1H), 7.29 (s, 1H), 7.22 (d, J = 1.5 Hz, 1H), 7.11 (dd, J = 8.0, 1.4 Hz, 1H), 6.87 (d, J = 8.0 Hz, 1H), 6.03 (s, 2H); ^{13}C NMR (125 MHz,

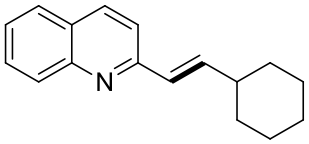


$CDCl_3$) δ 156.12, 148.29, 136.30, 134.15, 131.10, 129.72, 129.13, 127.49, 127.27, 127.25, 126.03, 122.81, 119.26, 115.00, 108.53, 106.06, 101.30.

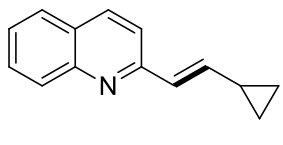
(*E*)-2-(2-([1,1'-Biphenyl]-4-yl)vinyl)quinoline (3i)[2]: Following the general procedure, the title compound was isolated as a white solid (34.5 mg, Yield: 45%). ^{1}H NMR (400 MHz, $CDCl_3$) δ 8.13 (d, J = 8.6 Hz, 1H), 8.08 (d, J = 8.4 Hz, 1H), 7.78 (d, J = 8.1 Hz, 1H), 7.75 – 7.68 (m, 5H), 7.66 – 7.62 (m, 3H), 7.60 – 7.56 (m, 1H), 7.48 – 7.42 (m, 4H), 7.36 (ddd, J = 8.2, 4.6, 1.8 Hz, 1H); ^{13}C NMR (125 MHz, $CDCl_3$) δ 156.01, 148.32, 141.37, 140.53, 136.37, 135.58, 133.97, 129.78, 129.23, 129.02, 128.85, 128.82, 127.74, 127.52, 127.48, 127.15, 126.99, 126.20, 119.34.



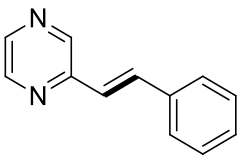
(*E*)-2-(2-Cyclohexylvinyl)quinoline (3l)[1]: : Following the general procedure, the title compound was isolated as a pale-yellow oil (18 mg, Yield: 31%). ^{1}H NMR (400 MHz, $CDCl_3$) δ 8.03 (dd, J = 13.4, 8.6 Hz, 2H), 7.73 (d, J = 8.1 Hz, 1H), 7.68 – 7.62 (m, 1H), 7.53 (d, J = 8.6 Hz, 1H), 7.44 (dd, J = 11.0, 4.1 Hz, 1H), 6.76 (dd, J = 16.1, 6.4 Hz, 1H), 6.66 (d, J = 16.3 Hz, 1H), 2.30 – 2.19 (m, 1H), 1.93 – 1.66 (m, 6H), 1.36 – 1.23 (m, 4H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 156.88, 148.16, 143.48, 136.20, 129.57, 129.18, 128.74, 127.50, 127.21, 125.88, 118.80, 41.23, 32.63, 26.23, 26.10.



(*E*)-2-(2-Cyclopropylvinyl)quinoline (3m)[1]: Following the general procedure, the title compound was isolated as a pale-yellow oil (14.5 mg, Yield: 30%). ^{1}H NMR (400 MHz, $CDCl_3$) δ 8.01 (dd, J = 16.4, 8.5 Hz, 2H), 7.72 (d, J = 8.0 Hz, 1H), 7.64 (ddd, J = 8.4, 6.9, 1.3 Hz, 1H), 7.46 – 7.40 (m, 2H), 6.76 (d, J = 15.7 Hz, 1H), 6.37 (dd, J = 15.7, 9.3 Hz, 1H), 1.75 – 1.65 (m, 1H), 0.94 – 0.89 (m, 2H), 0.70 – 0.63 (m, 2H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 156.28, 151.74, 148.20, 142.15, 136.23, 129.57, 129.10, 128.32, 127.49, 125.72, 118.93, 15.00, 8.13.

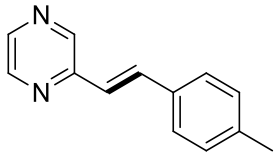


(*E*)-2-Styrylpyrazine (4a)[1]: Following the general procedure, the title compound was isolated as a white solid (35 mg, Yield: 77%). ^{1}H NMR (400 MHz, $CDCl_3$) δ 8.63 (d, J = 1.4 Hz, 1H), 8.54 – 8.53 (m, 1H), 8.39 (d, J = 2.5 Hz, 1H), 7.74 (d, J = 16.1 Hz, 1H), 7.59 (d, J = 7.4 Hz, 2H), 7.41 – 7.37 (m, 2H), 7.34 – 7.31 (m, 1H), 7.15 (d, J = 16.1 Hz, 1H); ^{13}C NMR (100



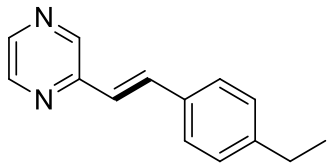
MHz, $CDCl_3$) δ 151.33, 144.45, 143.89, 142.86, 136.08, 135.27, 129.11, 128.95, 127.43, 124.06.

(*E*)-2-(4-Methylstyryl)pyrazine (4b)[1]: Following the general procedure, the title compound



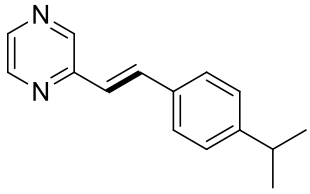
was isolated as a white solid (33.3 mg, Yield: 68%). ^{1}H NMR (400 MHz, $CDCl_3$) δ 8.61 (d, J = 1.4 Hz, 1H), 8.52 – 8.51 (m, 1H), 8.37 (d, J = 2.5 Hz, 1H), 7.71 (d, J = 16.1 Hz, 1H), 7.48 (d, J = 8.1 Hz, 2H), 7.19 (d, J = 8.0 Hz, 2H), 7.10 (d, J = 16.1 Hz, 1H), 2.37 (s, 3H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 151.57, 144.38, 143.76, 142.60, 139.27, 135.23, 133.36, 129.67, 127.37, 123.09, 21.48.

(*E*)-2-(4-Ethylstyryl)pyrazine (4c): Following the general procedure, the title compound



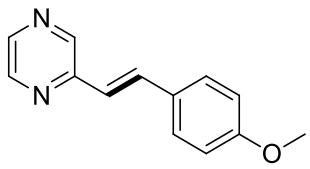
was isolated as a white solid (38 mg, Yield: 72%). ^{1}H NMR (400 MHz, $CDCl_3$) δ 8.62 (d, J = 1.4 Hz, 1H), 8.52 – 8.51 (m, 1H), 8.37 (d, J = 2.5 Hz, 1H), 7.72 (d, J = 16.1 Hz, 1H), 7.51 (d, J = 8.1 Hz, 2H), 7.22 (d, J = 8.1 Hz, 2H), 7.10 (d, J = 16.1 Hz, 1H), 2.66 (q, J = 7.6 Hz, 2H), 1.24 (t, J = 7.6 Hz, 3H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 151.59, 145.62, 144.41, 143.73, 142.63, 135.27, 133.61, 128.42, 127.44, 123.12, 28.83, 15.50. HRMS (ESI): Calculated for $[C_{14}H_{15}N_2]^+$ 211.1230; Found 211.1233.

(*E*)-2-(4-Isopropylstyryl)pyrazine (4d)[1]: Following the general procedure, the title



compound was isolated as a white solid (40 mg, Yield: 71%). ^{1}H NMR (400 MHz, $CDCl_3$) δ 8.62 (d, J = 1.5 Hz, 1H), 8.52 (dd, J = 2.4, 1.6 Hz, 1H), 8.36 (d, J = 2.5 Hz, 1H), 7.72 (d, J = 16.1 Hz, 1H), 7.53– 7.51 (m, 2H), 7.26-7.24 (m, 2H), 7.11 (d, J = 16.1 Hz, 1H), 2.92 (dt, J = 13.8, 6.9 Hz, 1H), 1.26 (d, J = 7.0 Hz, 6H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 151.57, 150.25, 144.40, 143.79, 142.61, 135.22, 133.72, 127.48, 127.06, 123.15, 34.11, 23.97.

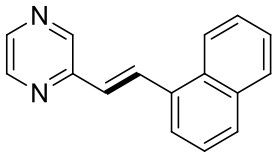
(*E*)-2-(4-Methoxystyryl)pyrazine (4e)[3]: Following the general procedure, the title



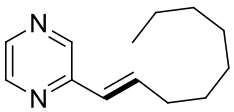
compound was isolated as a white solid (41.3 mg, Yield: 78%). ^{1}H NMR (400 MHz, $CDCl_3$) δ 8.60 (d, J = 1.7 Hz, 1H), 8.51 (d, J = 3.8 Hz, 1H), 8.36 (d, J = 2.5 Hz, 1H), 7.69 (dd, J = 16.0, 2.4 Hz,

1H), 7.55 – 7.52 (m, 2H), 7.05 – 6.98 (m, 1H), 6.93 – 6.91 (m, 2H), 3.83 (s, 3H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 160.45, 151.73, 144.34, 143.65, 142.36, 134.85, 128.91, 128.85, 121.86, 114.38, 55.45.

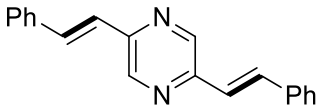
(*E*)-2-(2-(Naphthalen-1-yl)vinyl)pyrazine (4g)[4]: Following the general procedure, the title compound was isolated as a white solid (38 mg, Yield: 65%). ^{1}H NMR (400 MHz, $CDCl_3$) δ 8.55 (d, J = 5.2 Hz, 1H), 8.49 (d, J = 3.7 Hz, 1H), 8.29 (d, J = 8.2 Hz, 1H), 8.09 (d, J = 7.3 Hz, 1H), 7.87 (d, J = 7.7 Hz, 2H), 7.79 (d, J = 7.8 Hz, 2H), 7.58 – 7.52 (m, 3H), 7.15 (d, J = 15.8 Hz, 1H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 151.41, 144.47, 143.78, 142.72, 136.51, 133.86, 132.41, 131.32, 128.76, 128.56, 126.40, 125.96, 125.51, 125.35, 124.27, 123.79.



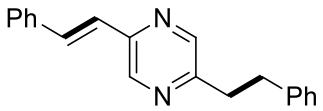
(*E*)-2-(Non-1-en-1-yl)pyrazine (4h): Following the general procedure, the title compound was isolated as a white solid (13 mg, Yield: 25%). ^{1}H NMR (400 MHz, $CDCl_3$) δ 8.50 (d, J = 1.4 Hz, 1H), 8.46 – 8.45 (m, 1H), 8.34 (d, J = 2.5 Hz, 1H), 6.87 (dt, J = 15.7, 7.0 Hz, 1H), 6.47 (dt, J = 15.8, 1.4 Hz, 1H), 2.28 (ddd, J = 14.8, 7.3, 1.5 Hz, 2H), 1.53 – 1.46 (m, 2H), 1.37 – 1.27 (m, 8H), 0.86 (d, J = 7.1 Hz, 3H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 150.90, 144.05, 142.86, 142.22, 139.15, 126.27, 33.02, 31.77, 29.19, 29.13, 28.75, 22.64, 14.10. HRMS (ESI): Calculated for $[C_{13}H_{21}N_2]^+$ 205.1699; Found 205.1696.



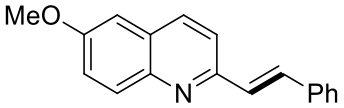
2,5-Di((*E*)-styryl)pyrazine (4i)[5]: Following the general procedure, the title compound was isolated as a white solid (32 mg, Yield: 45%). ^{1}H NMR (400 MHz, $CDCl_3$) δ 8.59 (s, 2H), 7.73 (d, J = 16.1 Hz, 2H), 7.60 (d, J = 8.5 Hz, 4H), 7.39 (d, J = 7.2 Hz, 3H), 7.33 (d, J = 7.2 Hz, 2H), 7.18 (d, J = 16.1 Hz, 3H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 154.98, 149.17, 143.39, 136.33, 134.45, 128.94, 127.37, 124.15.



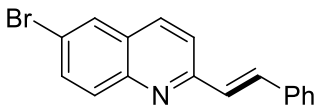
(*E*)-2-Phenethyl-5-styrylpyrazine (4i')[1]: Following the general procedure, the title compound was isolated as a white solid (22 mg, Yield: 31%). ^{1}H NMR (400 MHz, $CDCl_3$) δ 8.57 (s, 1H), 8.30 (s, 1H), 7.67 (d, J = 16.1 Hz, 1H), 7.58 (d, J = 7.4 Hz, 2H), 7.39 (t, J = 7.4 Hz, 2H), 7.33 – 7.26 (m, 3H), 7.21 – 7.12 (m, 4H), 3.14 – 3.04 (m, 4H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 154.57, 148.71, 143.94, 142.77, 140.84, 136.27, 134.07, 128.82, 128.75, 128.52, 128.45, 127.20, 126.23, 124.10, 37.02, 35.48.



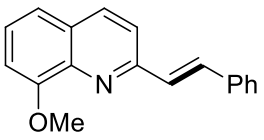
(*E*)-6-Methoxy-2-styrylquinoline (5a)[1]: Following the general procedure, the title compound was isolated as a white solid (50 mg, Yield: 77%). ^{1}H NMR (400 MHz, $CDCl_3$) δ 8.00 (dd, J = 17.6, 8.9 Hz, 2H), 7.64 – 7.58 (m, 4H), 7.40 – 7.28 (m, 5H), 7.05 (d, J = 2.8 Hz, 1H), 3.92 (s, 3H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 157.72, 153.80, 144.34, 136.76, 135.21, 133.30, 130.71, 129.13, 128.87, 128.50, 128.38, 127.22, 122.44, 119.65, 105.31, 55.65.



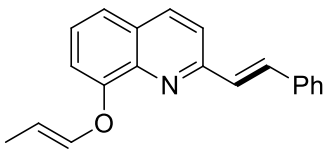
(*E*)-6-bromo-2-styrylquinoline (5b)[8]: Following the general procedure, the title compound was isolated as a white solid (25.5 mg, Yield: 33%). ^{1}H NMR (400 MHz, $CDCl_3$) δ 8.02 (d, J = 8.6 Hz, 1H), 7.95 – 7.91 (m, 2H), 7.75 (dd, J = 9.0, 2.2 Hz, 1H), 7.72 – 7.61 (m, 4H), 7.43 – 7.30 (m, 4H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 156.43, 146.92, 136.39, 135.43, 135.10, 133.28, 130.95, 129.66, 129.18, 128.94, 128.59, 128.47, 127.42, 120.31, 120.00.



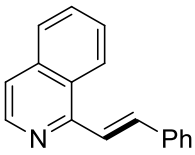
(*E*)-8-Methoxy-2-styrylquinoline (5c)[6]: Following the general procedure, the title compound was isolated as a white solid (38 mg, Yield: 58%). ^{1}H NMR (400 MHz, $CDCl_3$) δ 8.07 (d, J = 8.5 Hz, 1H), 7.74 (d, J = 8.6 Hz, 1H), 7.64 – 7.61 (m, 2H), 7.56 (d, J = 11.0 Hz, 2H), 7.41 – 7.33 (m, 5H), 7.04 (d, J = 7.4 Hz, 1H), 4.09 (s, 3H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 155.23, 155.18, 140.10, 136.66, 136.39, 134.06, 129.77, 128.89, 128.62, 128.49, 128.46, 127.33, 126.48, 119.53, 119.27, 108.02, 56.20.



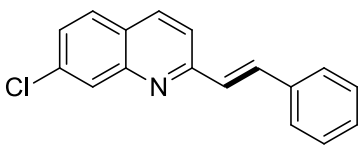
8-((*E*)-Prop-1-en-1-yloxy)-2-((*E*)-styryl)quinoline (5d): Following the general procedure, the title compound was isolated as a white solid (53 mg, Yield: 74%). ^{1}H NMR (500 MHz, $CDCl_3$) δ 8.12 (d, J = 8.6 Hz, 1H), 7.76 (d, J = 8.6 Hz, 1H), 7.71 – 7.64 (m, 3H), 7.56 (d, J = 16.4 Hz, 1H), 7.48 – 7.42 (m, 4H), 7.35 (dd, J = 10.5, 4.1 Hz, 1H), 7.23 (dd, J = 7.5, 1.3 Hz, 1H), 6.61 (dq, J = 5.7, 1.6 Hz, 1H), 5.17 – 5.12 (m, 1H), 1.91 (dd, J = 6.9, 1.7 Hz, 3H); ^{13}C NMR (125 MHz, $CDCl_3$) δ 155.47, 153.32, 141.43, 140.18, 136.65, 136.20, 134.25, 129.66, 128.79, 128.67, 128.55, 127.32, 126.09, 121.29, 119.45, 112.95, 109.55, 9.87. Elemental Analysis calculated: C, 83.59; H, 5.96; Found: C, 83.13; H, 6.07.



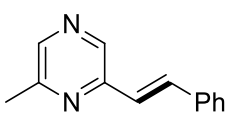
(*E*)-1-Styrylisoquinoline (5e)[2]: Following the general procedure, the title compound was isolated as a white solid (26 mg, Yield: 45%). ^{1}H NMR (400 MHz, $CDCl_3$) δ 8.56 (d, J = 5.6 Hz, 1H), 8.37 (d, J = 8.5 Hz, 1H), 8.00 (t, J = 8.9 Hz, 2H), 7.82 (d, J = 8.2 Hz, 1H), 7.71 – 7.60 (m, 4H), 7.56 (d, J = 5.6 Hz, 1H), 7.43 – 7.39 (m, 2H), 7.35 – 7.31 (m, 1H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 154.62, 142.55, 136.98, 136.82, 135.91, 130.03, 128.88, 128.73, 127.55, 127.43, 127.31, 126.84, 124.56, 122.89, 120.10.



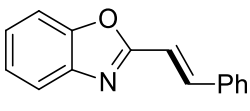
***(E)*-7-Chloro-2-styrylquinoline (5f)**[9]: Following the general procedure, the title compound was isolated as a yellow solid (39 mg, Yield: 58%). ^{1}H NMR (400 MHz, $CDCl_3$) δ 8.01 (d, J = 8.3 Hz, 2H), 7.64 (d, J = 8.6 Hz, 1H), 7.61 (s, 1H), 7.56 (dd, J = 7.9, 2.8 Hz, 3H), 7.34 (dd, J = 15.2, 7.2 Hz, 3H), 7.27 (t, J = 3.7 Hz, 2H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 155.89, 147.65, 135.30, 135.09, 134.51, 134.15, 127.81, 127.64, 127.46, 127.38, 127.18, 126.33, 126.06, 124.64, 118.61.



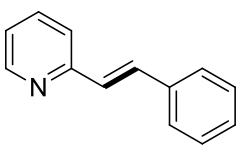
(*E*)-2-Methyl-6-styrylpyrazine (5g)[2]: Following the general procedure, the title compound was isolated as a white solid (22.5 mg, Yield: 46%). ^{1}H NMR (400 MHz, $CDCl_3$) δ 8.44 (s, 1H), 8.28 (s, 1H), 7.71 (d, J = 16.1 Hz, 1H), 7.59 (d, J = 7.3 Hz, 2H), 7.38 (t, J = 7.4 Hz, 2H), 7.33 (d, J = 7.3 Hz, 1H), 7.13 (d, J = 16.1 Hz, 1H), 2.58 (s, 3H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 153.43, 150.16, 142.66, 140.60, 136.27, 134.73, 128.91, 128.90, 127.36, 124.49, 21.84.



(*E*)-2-Styrylbenzo[*d*]oxazole (5h)[2]: Following the general procedure, the title compound was isolated as a white solid (25 mg, Yield: 45%). ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.80 (dd, J = 16.3, 11.9 Hz, 1H), 7.75 – 7.66 (m, 1H), 7.63 – 7.59 (m, 2H), 7.56 – 7.51 (m, 1H), 7.41 (dd, J = 11.5, 4.0 Hz, 2H), 7.37 – 7.25 (m, 3H), 7.09 (dd, J = 16.4, 11.6 Hz, 1H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 162.90, 150.51, 142.27, 139.57, 135.24, 129.87, 129.07, 127.65, 125.31, 124.61, 119.97, 114.05, 110.42.

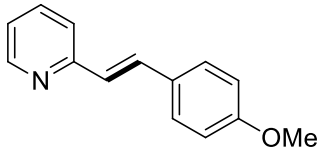


(*E*)-2-Styrylpyridine (5i)[1]: Following the general procedure, the title compound was isolated as a white solid (41 mg, Yield: 90%). ^{1}H NMR (400 MHz, $CDCl_3$) δ 8.61 – 8.59 (m, 1H), 7.66 – 7.64 (m, 1H), 7.63 – 7.61 (m, 1H), 7.59 – 7.56 (m, 2H), 7.38 – 7.35 (m, 3H), 7.29 (ddd, J = 7.2, 3.7, 1.2 Hz, 1H),



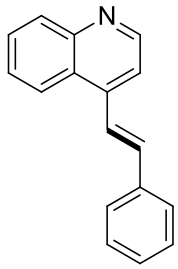
7.18 (s, 1H), 7.12 (ddd, *J* = 4.8, 2.4, 1.2 Hz, 1H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 155.71, 149.76, 136.74, 136.63, 132.82, 128.82, 128.43, 128.04, 127.20, 122.18, 122.15.

(*E*)-2-(4-Methoxystyryl)pyridine (5j)[7]: Following the general procedure, the title compound



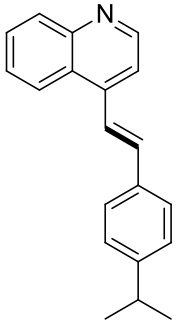
was isolated as a white solid (48 mg, Yield: 91%). ^{1}H NMR (400 MHz, $CDCl_3$) δ 8.58 – 8.56 (m, 1H), 7.64 – 7.55 (m, 2H), 7.53 – 7.49 (m, 2H), 7.33 (d, *J* = 7.9 Hz, 1H), 7.09 (ddd, *J* = 7.3, 4.8, 1.0 Hz, 1H), 7.03 (d, *J* = 16.1 Hz, 1H), 6.91 – 6.88 (m, 2H), 3.81 (s, 3H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 159.93, 156.01, 149.67, 136.62, 132.35, 129.48, 128.53, 125.86, 121.90, 121.79, 114.26, 55.42.

(*E*)-4-Styrylquinoline (5k)[1]: Following the general procedure, the title compound was



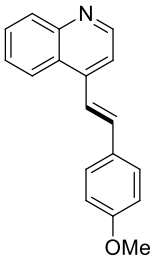
isolated as a yellow oil (32 mg, Yield: 55%). ^{1}H NMR (400 MHz, $CDCl_3$) δ 8.93 (d, *J* = 4.4 Hz, 1H), 8.24 (d, *J* = 8.4 Hz, 1H), 8.17 (d, *J* = 8.4 Hz, 1H), 7.84 (d, *J* = 16.1 Hz, 1H), 7.76 (t, *J* = 7.5 Hz, 1H), 7.68 – 7.60 (m, 4H), 7.46 (t, *J* = 7.3 Hz, 2H), 7.38 (t, *J* = 12.9 Hz, 2H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 150.23, 148.75, 142.97, 136.61, 135.16, 130.17, 129.31, 128.91, 128.80, 127.13, 126.51, 126.45, 123.49, 122.96, 117.10.

***(E)*-4-(4-Isopropylstyryl)quinoline (5l)**[10]: Following the general procedure, the title



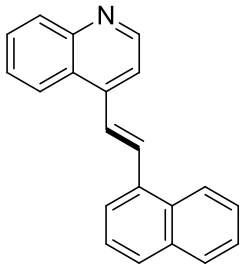
compound was isolated as a yellow oil (20 mg, Yield: 29%). ^{1}H NMR (400 MHz, $CDCl_3$) δ 8.88 (d, *J* = 4.6 Hz, 1H), 8.21 (dd, *J* = 8.5, 0.9 Hz, 1H), 8.12 (dd, *J* = 8.5, 0.6 Hz, 1H), 7.74 (ddd, *J* = 12.5, 10.8, 8.5 Hz, 2H), 7.57 (ddd, *J* = 8.2, 3.3, 1.7 Hz, 4H), 7.30 (dd, *J* = 13.9, 12.1 Hz, 3H), 2.95 (dq, *J* = 13.8, 6.9 Hz, 1H), 1.28 (d, *J* = 6.9 Hz, 6H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 150.30, 150.04, 148.78, 143.25, 135.18, 135.16, 134.31, 130.19, 129.37, 127.27, 127.09, 126.53, 123.58, 122.02, 117.02, 34.10, 23.99.

***(E)*-4-(4-Methoxystyryl)quinoline (5m)**[10]: Following the general procedure, the title

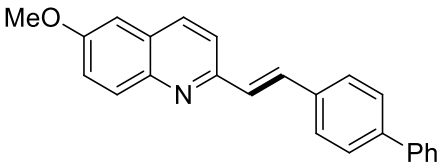


compound was isolated as a yellow oil (23 mg, Yield: 35%). ^{1}H NMR (400 MHz, $CDCl_3$) δ 8.87 (d, *J* = 4.6 Hz, 1H), 8.21 (dd, *J* = 8.5, 0.9 Hz, 1H), 8.12 (dd, *J* = 8.5, 0.6 Hz, 1H), 7.74 – 7.64 (m, 2H), 7.59 – 7.55 (m, 4H), 7.29 (d, *J* = 16.1 Hz, 1H), 6.95 (d, *J* = 8.8 Hz, 2H), 3.85 (s, 3H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 160.31, 150.23, 148.73, 143.39, 134.78, 130.10, 129.46, 129.36, 128.62, 126.51, 126.47, 123.57, 120.56, 116.80, 114.43, 55.48.

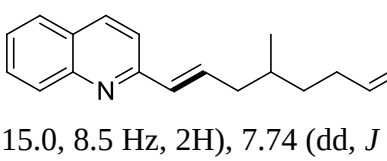
***(E)*-4-(2-(Naphthalen-1-yl)vinyl)quinoline (5n)**[8]: Following the general procedure, the title compound was isolated as a yellow solid (42 mg, Yield: 60%). ^{1}H NMR (400 MHz, $CDCl_3$) δ 8.87 (d, J = 4.6 Hz, 1H), 8.15 (t, J = 8.2 Hz, 2H), 8.06 (dd, J = 20.3, 12.2 Hz, 2H), 7.84 – 7.75 (m, 4H), 7.67 (ddd, J = 8.3, 6.8, 1.4 Hz, 1H), 7.62 (d, J = 4.5 Hz, 1H), 7.53 – 7.46 (m, 4H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 150.38, 148.83, 143.24, 134.38, 133.84, 132.52, 131.47, 130.24, 129.48, 129.26, 128.86, 126.71, 126.65, 126.55, 126.21, 125.75, 124.49, 123.67, 123.65, 117.47. GC-MS (EI) m/z = 281.1



(*E*)-2-(2-([1,1'-Biphenyl]-4-yl)vinyl)-6-methoxyquinoline (6a)[1]: **(*E*)-2-(2-([1,1'-Biphenyl]-4-yl)vinyl)-6-methoxyquinoline (6a)**[1]: Following the general procedure, the title compound was isolated as a white solid (34 mg, Yield: 40%). ^{1}H NMR (400 MHz, $CDCl_3$) δ 8.00 (dd, J = 16.5, 8.9 Hz, 2H), 7.66 (ddd, J = 6.6, 5.5, 2.6 Hz, 8H), 7.49 – 7.40 (m, 3H), 7.37 (ddd, J = 7.6, 5.3, 3.9 Hz, 2H), 7.06 (d, J = 2.8 Hz, 1H), 3.93 (s, 3H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 156.63, 152.68, 143.27, 140.06, 139.52, 134.71, 134.10, 131.70, 129.61, 128.01, 127.81, 127.29, 126.55, 126.44, 126.41, 125.94, 121.34, 118.60, 104.23, 54.54.



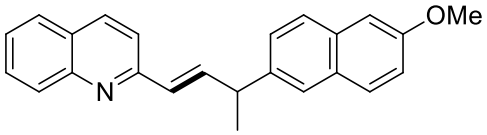
(*E*)-2-(4,8-Dimethylnona-1,7-dien-1-yl)quinoline (6b)[2]: Following the general procedure, the title compound was isolated as a colorless oil (32 mg, Yield: 46%). ^{1}H NMR (400 MHz, $CDCl_3$) δ 8.03 (dd, J = 15.0, 8.5 Hz, 2H), 7.74 (dd, J = 8.1, 1.1 Hz, 1H), 7.66 (ddd, J = 8.4, 6.9, 1.5 Hz, 1H), 7.53 (d, J = 8.6 Hz, 1H), 7.45 (ddd, J = 8.0, 7.0, 1.1 Hz, 1H), 6.77 (ddd, J = 36.6, 21.8, 11.5 Hz, 2H), 5.10 (dddd, J = 7.1, 5.7, 2.7, 1.4 Hz, 1H), 2.36 – 2.31 (m, 1H), 2.16 (ddd, J = 11.0, 8.1, 4.0 Hz, 1H), 2.15 – 1.97 (m, 2H), 1.71 – 1.68 (m, 1H), 1.67 (d, J = 1.0 Hz, 3H), 1.60 (s, 3H), 1.49 – 1.38 (m, 1H), 1.23 (dddd, J = 13.7, 9.3, 7.0, 5.0 Hz, 1H), 0.96 (d, J = 6.7 Hz, 3H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 156.52, 148.13, 136.75, 136.25, 132.37, 131.41, 129.63, 129.17, 127.51, 127.23, 125.94, 124.76, 118.75, 40.72, 36.91, 32.78, 25.84, 25.71, 19.72, 17.78.



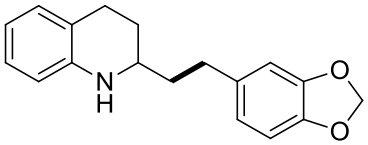
2-((1*E*,10*Z*)-Nonadeca-1,10-dien-1-yl)quinoline (6c): Following the general procedure, the title compound was isolated as a colorless oil (51 mg, Yield: 52%). ^{1}H NMR (400 MHz, $CDCl_3$) δ 8.03 (dd, J = 13.4, 8.5 Hz, 2H), 7.75 (t, J = 8.8 Hz, 1H), 7.66 (ddd, J = 8.3, 5.3, 1.2 Hz, 1H), 7.51 (d, J = 8.6 Hz, 1H), 7.45 (t, J = 7.2 Hz, 1H), 6.81 (dt, J = 15.8, 6.6 Hz, 1H),

6.70 (d, *J* = 16.0 Hz, 1H), 5.34 (dd, *J* = 9.5, 4.7 Hz, 2H), 2.32 (p, *J* = 7.3 Hz, 2H), 2.00 (d, *J* = 2.9 Hz, 4H), 1.54 (dt, *J* = 14.9, 7.3 Hz, 2H), 1.28 (dd, *J* = 14.9, 9.8 Hz, 20H), 0.86 (t, *J* = 6.8 Hz, 3H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 156.62, 148.17, 138.12, 136.20, 131.11, 130.04, 129.90, 129.57, 129.20, 127.47, 127.22, 125.88, 118.77, 33.13, 31.97, 29.85, 29.83, 29.59, 29.48, 29.40, 29.38, 29.34, 29.30, 28.98, 27.30, 27.28, 22.75, 14.17. Elemental Analysis calculated: C, 85.87; H, 10.55; Found: C, 85.52; H, 10.27.

(*E*)-2-(3-(6-Methoxynaphthalen-2-yl)but-1-en-1-yl)quinoline (6d): Following the general procedure, the title compound was isolated as a pale blue oil (38 mg, Yield: 45%). ^{1}H NMR (400 MHz, $CDCl_3$) δ 8.40 (d, *J* = 6.2 Hz, 1H), 8.00 (d, *J* = 8.5 Hz, 2H), 7.85 (d, *J* = 8.8 Hz, 2H), 7.76 (d, *J* = 8.6 Hz, 1H), 7.68 (dd, *J* = 17.9, 9.8 Hz, 1H), 7.36 – 7.25 (m, 4H), 7.16 (dd, *J* = 26.9, 13.1 Hz, 3H), 3.94 (s, 3H), 3.93 – 3.89 (m, 1H), 2.69 (d, *J* = 1.3 Hz, 3H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 160.01, 159.78, 137.30, 132.65, 131.13, 130.08, 129.18, 127.84, 127.11, 124.69, 119.78, 119.75, 115.00, 105.77, 55.44, 29.71, 26.56. Elemental Analysis calculated: C, 84.92; H, 6.22; Found: C, 84.47; H, 5.97.

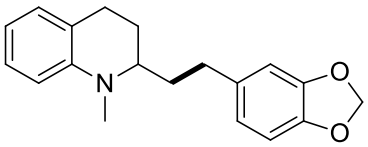


Procedure for the synthesis of 2-(2-(benzo[d][1,3]dioxol-5-yl)ethyl)-1,2,3,4-tetrahydroquinoline (3ha)[11]: Compound **3h** (0.073 mmol) and $NiCl_2.6H_2O$ (0.0146 mmol) were taken in a 50 mL RB and dissolved in 3 mL of methanol. Then $NaBH_4$ (0.3 mmol) was added in portion at 0 °C and stirred for 30 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 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 (19.5 mg, 95% yield).



^{1}H NMR (400 MHz, $CDCl_3$) δ 6.96 (t, *J* = 7.4 Hz, 2H), 6.72 (dd, *J* = 14.1, 4.7 Hz, 2H), 6.66 – 6.58 (m, 2H), 6.46 (d, *J* = 7.6 Hz, 1H), 5.92 (s, 2H), 3.74 (s, 1H), 3.28 (dtd, *J* = 9.4, 6.3, 3.0 Hz, 1H), 2.77 (tdd, *J* = 16.2, 11.0, 4.9 Hz, 2H), 2.68 – 2.63 (m, 2H), 2.01-1.95 (m, 1H), 1.78 (ddd, *J* = 8.7, 8.1, 3.3 Hz, 2H), 1.70 – 1.61 (m, 1H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 147.76, 145.80, 144.52, 135.72, 129.35, 126.83, 121.43, 121.11, 117.20, 114.27, 108.87, 108.32, 100.90, 51.07, 38.54, 31.96, 28.04, 26.29.

Procedure for the synthesis of 2-(2-(benzo[d][1,3]dioxol-5-yl)ethyl)-1-methyl-1,2,3,4-tetrahydroquinoline (3hb)[11]: In a 25 mL RB compound **3ha** (



0.0391 mmol), K_2CO_3 (0.06 mmol), MeI (0.235 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 products **3hb** (10 mg, 85% yield).

^{1}H NMR (400 MHz, $CDCl_3$) δ 6.96 (dd, J = 36.2, 7.2 Hz, 2H), 6.64 (t, J = 9.2 Hz, 2H), 6.52 (dd, J = 32.1, 19.1 Hz, 3H), 5.85 (s, 2H), 3.21 (t, J = 8.1 Hz, 1H), 2.84 (s, 3H), 2.76 (dd, J = 17.5, 10.1 Hz, 1H), 2.65 – 2.54 (m, 2H), 2.44 (dd, J = 19.4, 10.3 Hz, 1H), 2.27 (dd, J = 15.7, 8.1 Hz, 1H), 1.90 – 1.83 (m, 2H), 1.63 (dd, J = 12.0, 7.0 Hz, 2H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 147.35, 145.33, 144.97, 135.96, 127.94, 127.20, 121.31, 120.86, 115.71, 110.26, 107.84, 107.68, 100.94, 58.50, 38.07, 33.25, 31.17, 24.56, 23.05.

[1.5] References:

(1) M. K. Barman, S. Waiba and B. Maji, *Angew. Chem. Int. Ed.* 2018, **57**, 9126.

(2) G. Zhang, T. Irrgang, T. Dietel, F. Kallmeier and R. Kempe, *Angew. Chem. Int. Ed.* 2018, **57**, 9131.

(3) K. Hoffert, R. J. Durand, S. Gauthier, F. R. Guen and S. Achelle, *Eur. J. Org. Chem.* 2017, 523–529.

(4) A. Ohta, K. Hasegawa, K. Amano, C. Mori, A. Ohsawa, K. Ikeda and T. Watanabe, Chem. *Pharm. Bull.* 1979, **27**, 2596.

(5) R. W. Hogue, S. Dhers, R. M. Hellyer, J. Luo, G. S. Hanan, D. S. Larsen, A. L. Garden and S. Brooker, *Chem. Eur. J.* 2017, **23**, 14193.

(6) D. Pi, K. Jiang, H. Zhou, Y. Sui, Y. Uozumi, K. Zou, *RSC Adv.* 2014, **4**, 57875.

(7) H. B. Hepburna and P. Melchiorre, *Chem. Commun.* 2016, **52**, 3520.

(8) Y. Yan, K. Xu, Y. Fang and Z. Wang, *J. Org. Chem.* 2011, **76**, 6849.

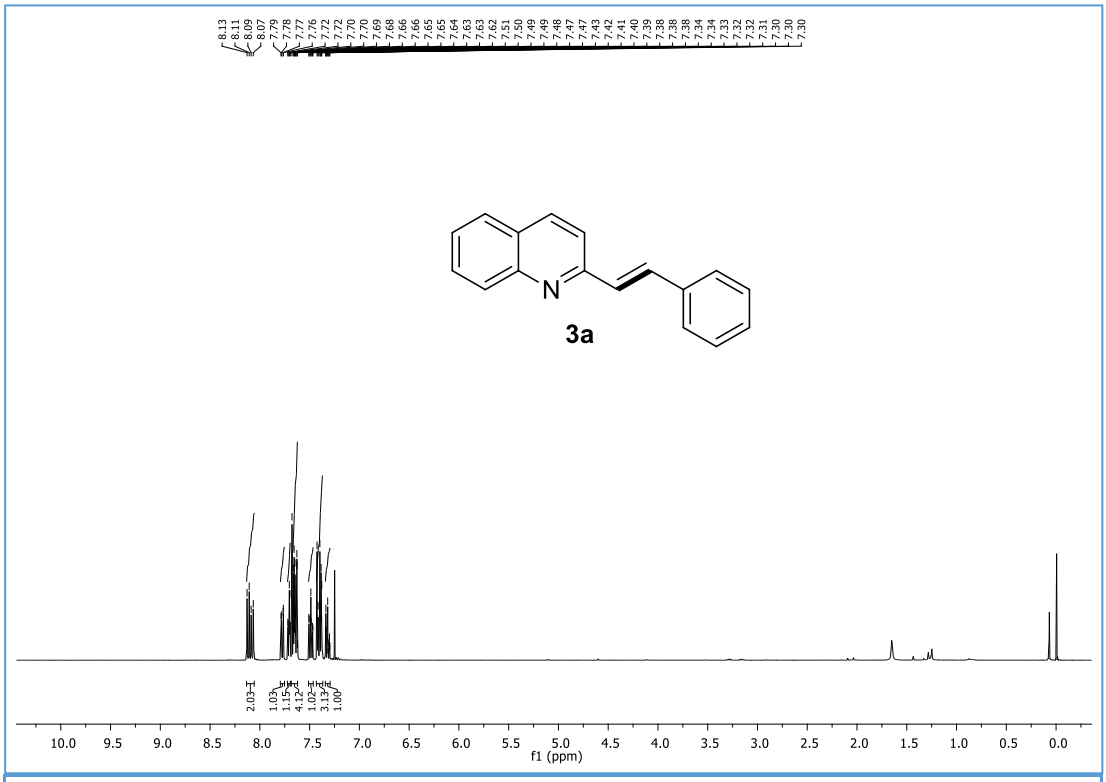
(9) K. N. Campbell, C. H. Helbing and J. F. Kerwin, *J. Am. Chem. Soc*. 1946, **68**, 1840.

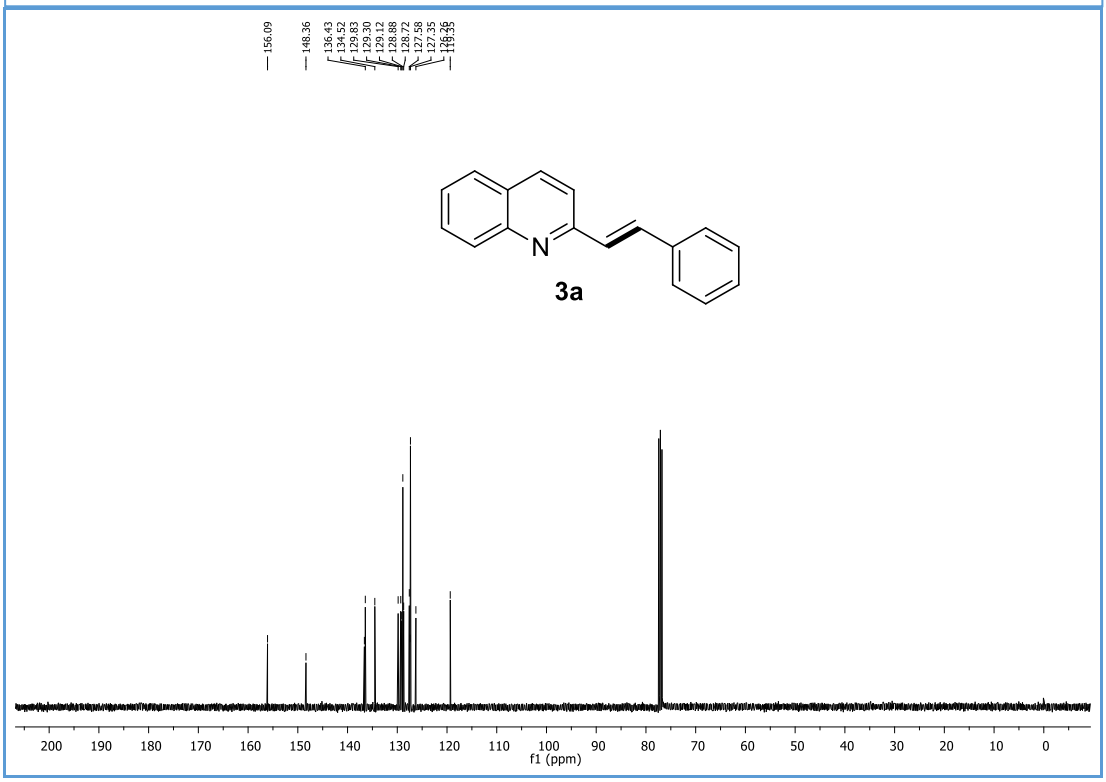
(10) S. A. Ahmed, T. Hartmann, Huch, Volker, H. Durr and A. -M. A. Abdel-Wahab, J. *Phys. Org. Chem.* 2000, **13**, 539.

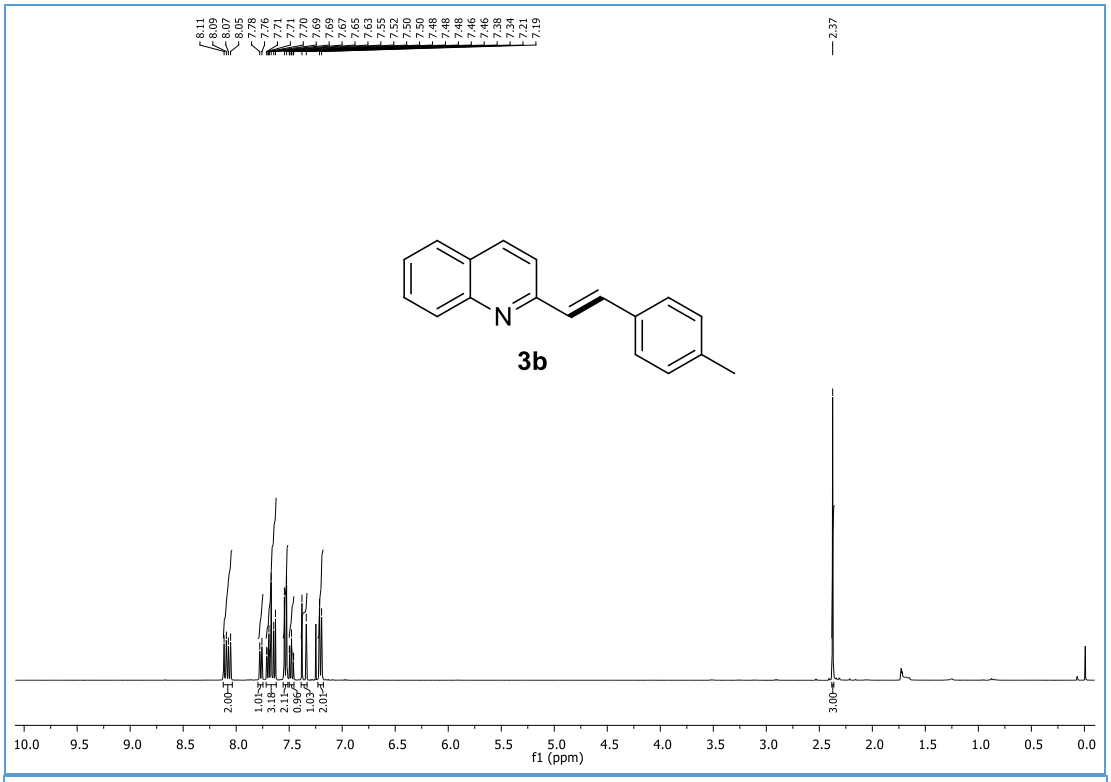
(11) W. Liu, B. Sahoo, K. Junge and M. Beller, *Acc. Chem. Res.* 2018, **51**, 1858.

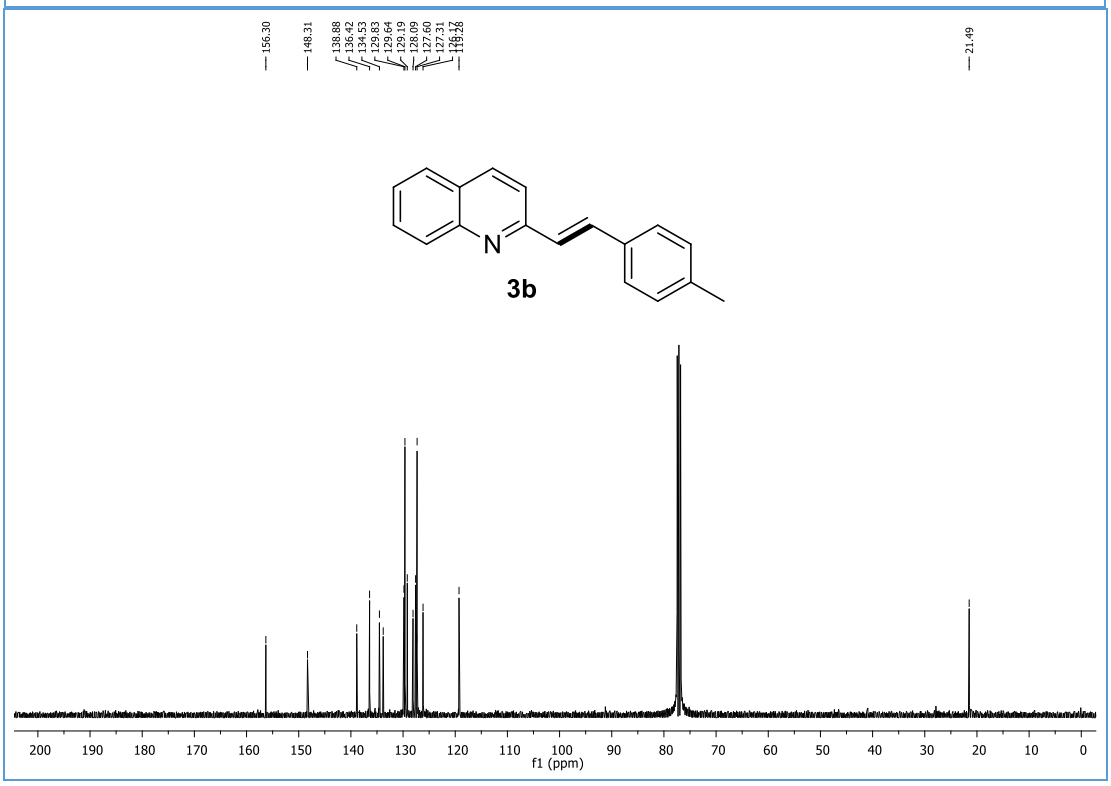
.

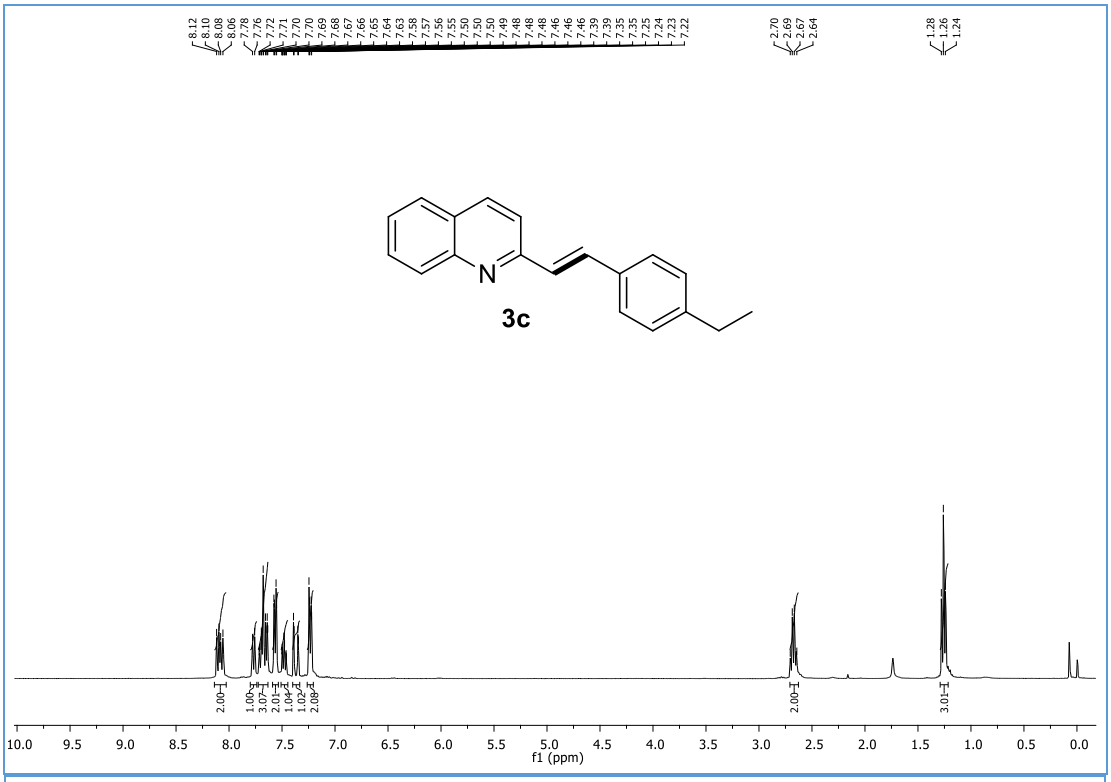
[1.6] Copies of ^{1}H NMR, ^{13}C NMR and HRMS Spectra for selected compounds

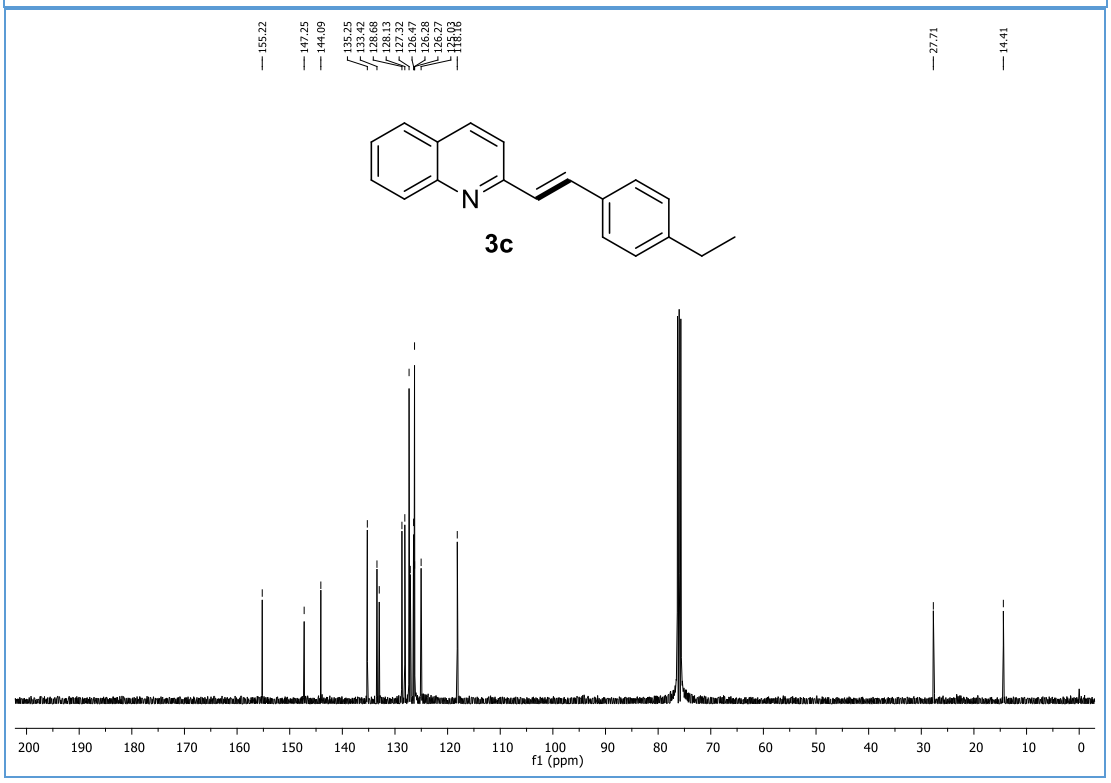


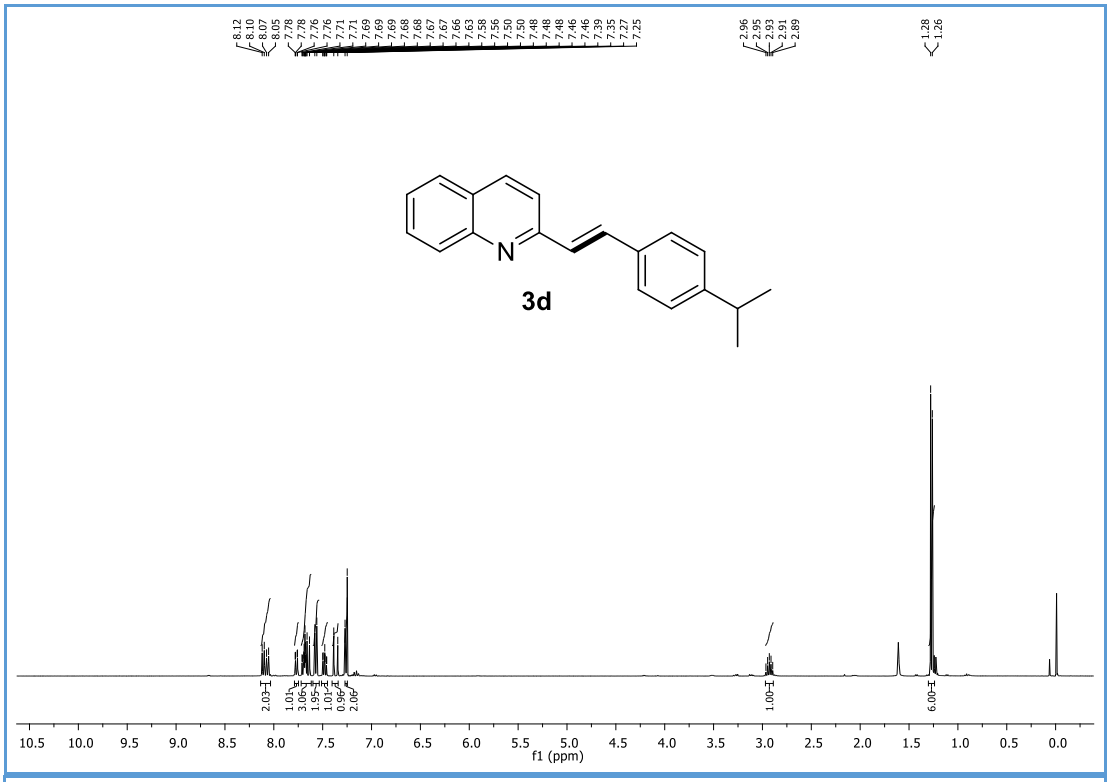


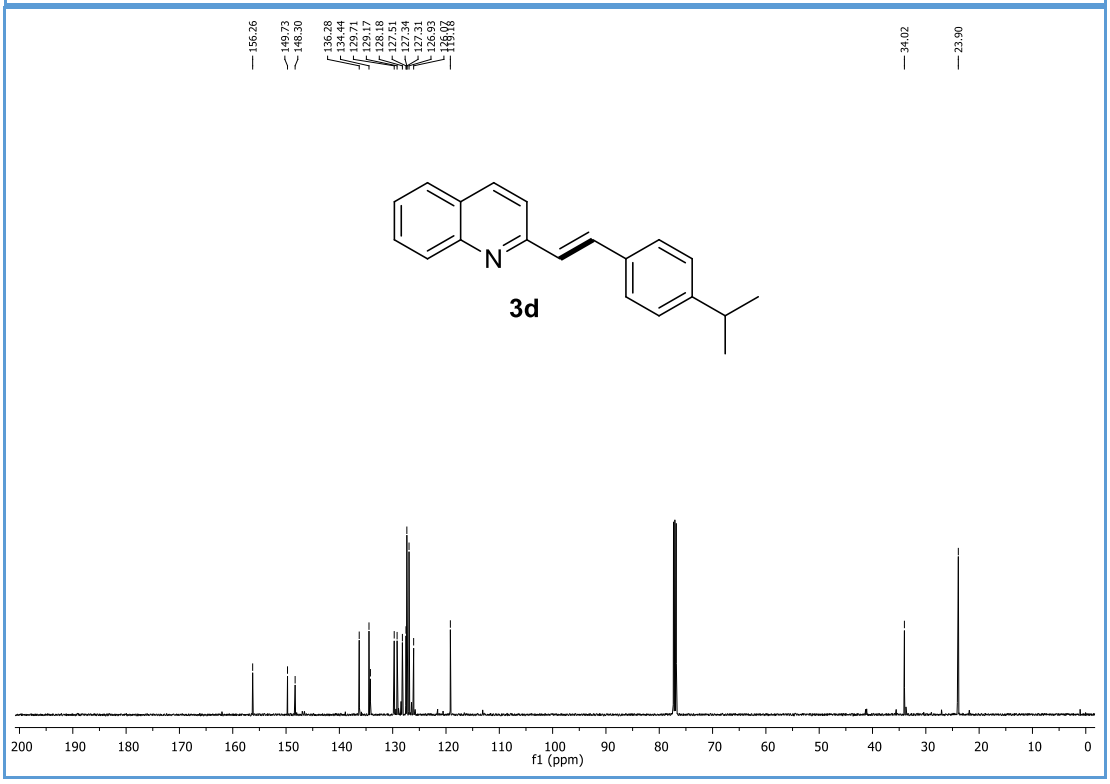

8.11
8.09
8.07
8.05
7.78
7.76
7.71
7.71
7.70
7.69
7.69
7.67
7.65
7.63
7.55
7.52
7.50
7.50
7.48
7.48
7.46
7.46
7.38
7.34
7.21
7.19
2.37
N
3b
2.00
1.01
3.18
2.11
0.96
1.03
2.01
3.00
10.0 9.5 9.0 8.5 8.0 7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 0.5 0.0
f1 (ppm)

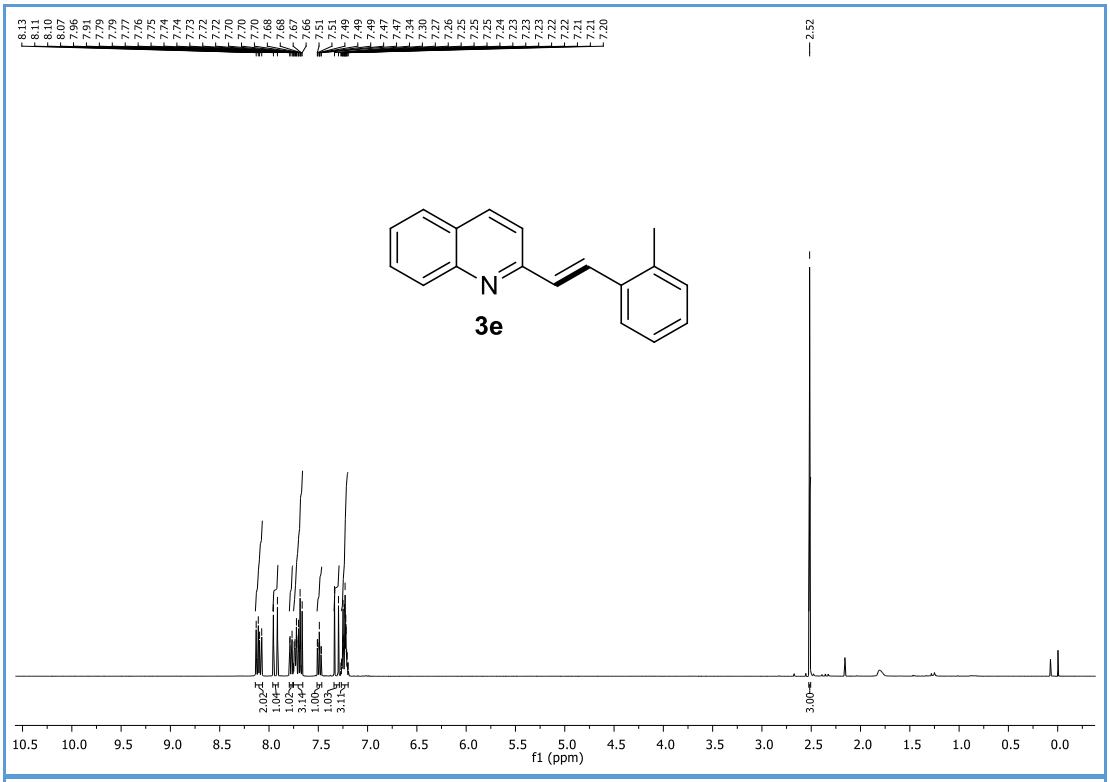

156.30
148.31
138.88
136.42
134.53
129.83
129.64
129.19
128.09
127.60
127.31
21.49
N
3b
200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0
f1 (ppm)

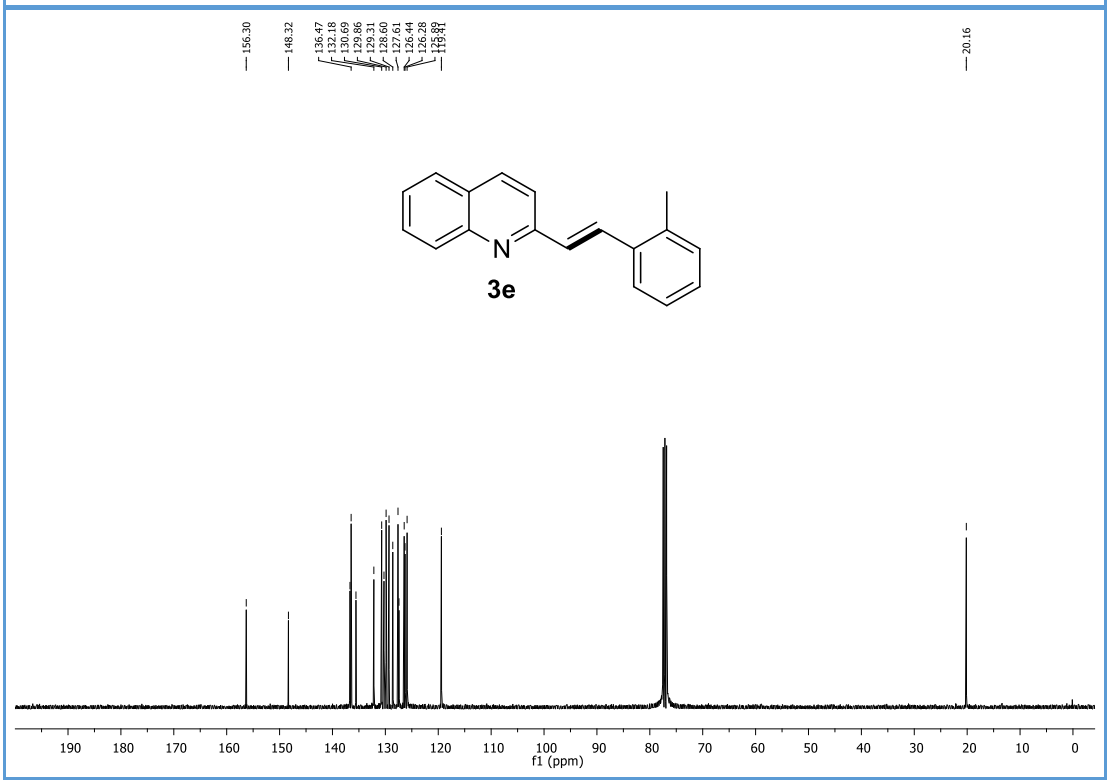

8.12
8.10
8.08
8.06
7.78
7.76
7.72
7.71
7.70
7.70
7.69
7.68
7.67
7.66
7.65
7.64
7.63
7.58
7.57
7.56
7.55
7.50
7.50
7.50
7.49
7.48
7.48
7.48
7.46
7.46
7.46
7.39
7.39
7.35
7.35
7.25
7.24
7.23
7.22
2.70
2.69
2.67
2.64
1.28
1.26
1.24
N
3c
2.00
1.00
3.07
2.01
1.04
1.02
2.08
2.00
3.01
10.0 9.5 9.0 8.5 8.0 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)

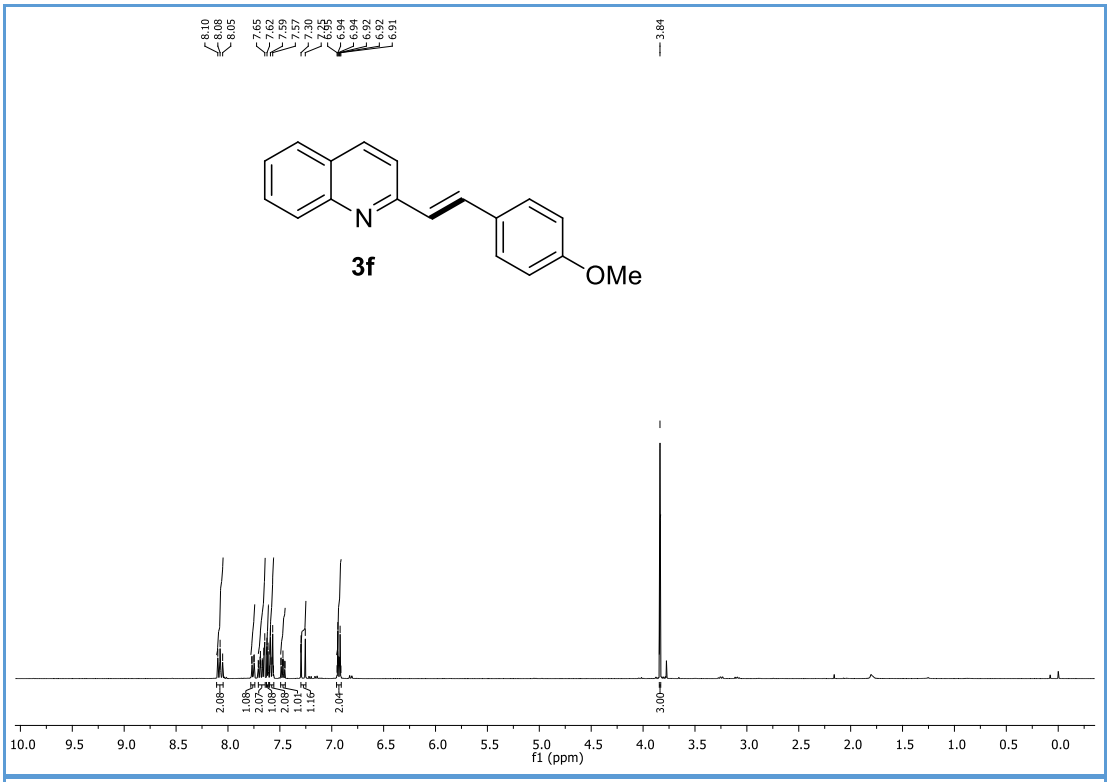

155.22
147.25
144.09
135.25
133.42
128.68
128.13
127.32
126.47
126.28
126.27
27.71
14.41
N
3c
200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0
f1 (ppm)

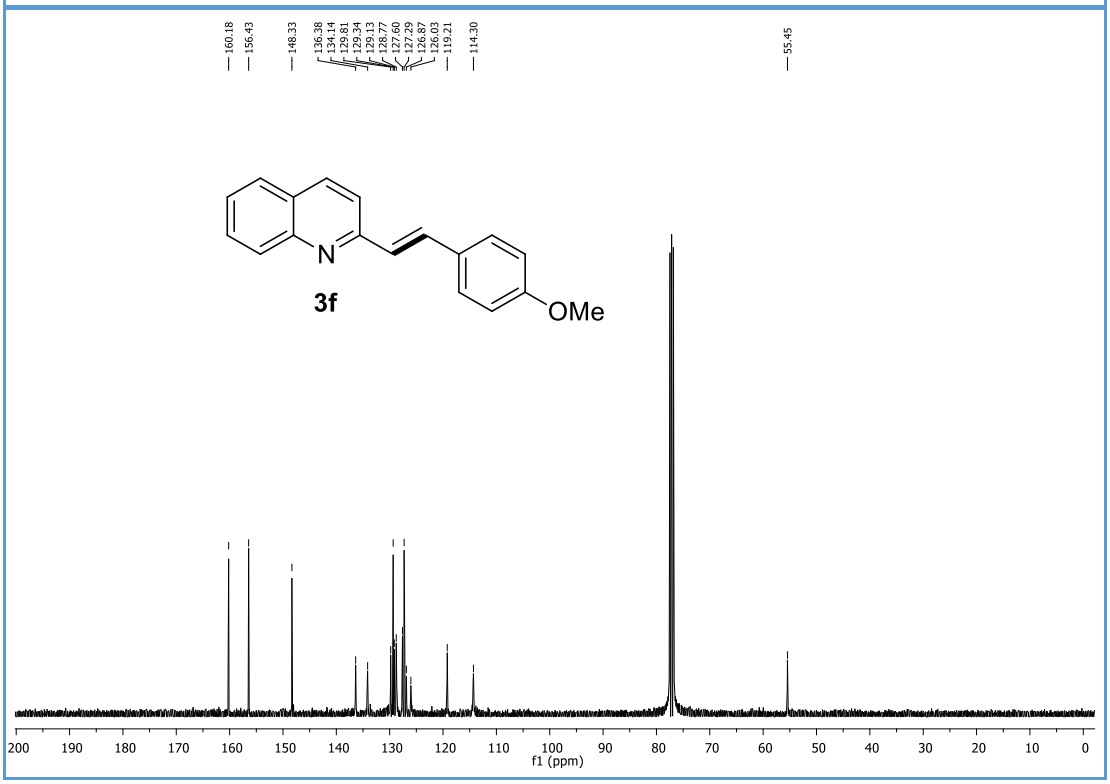

8.12
8.10
8.07
8.05
7.78
7.76
7.71
7.69
7.68
7.67
7.66
7.63
7.58
7.56
7.50
7.48
7.46
7.39
7.35
7.27
7.25
2.96
2.93
2.91
2.89
1.28
1.26
N
3d
2.03
1.01
3.06
1.95
1.01
0.96
2.06
1.00
6.00
10.5 10.0 9.5 9.0 8.5 8.0 7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 0.5 0.0
f1 (ppm)

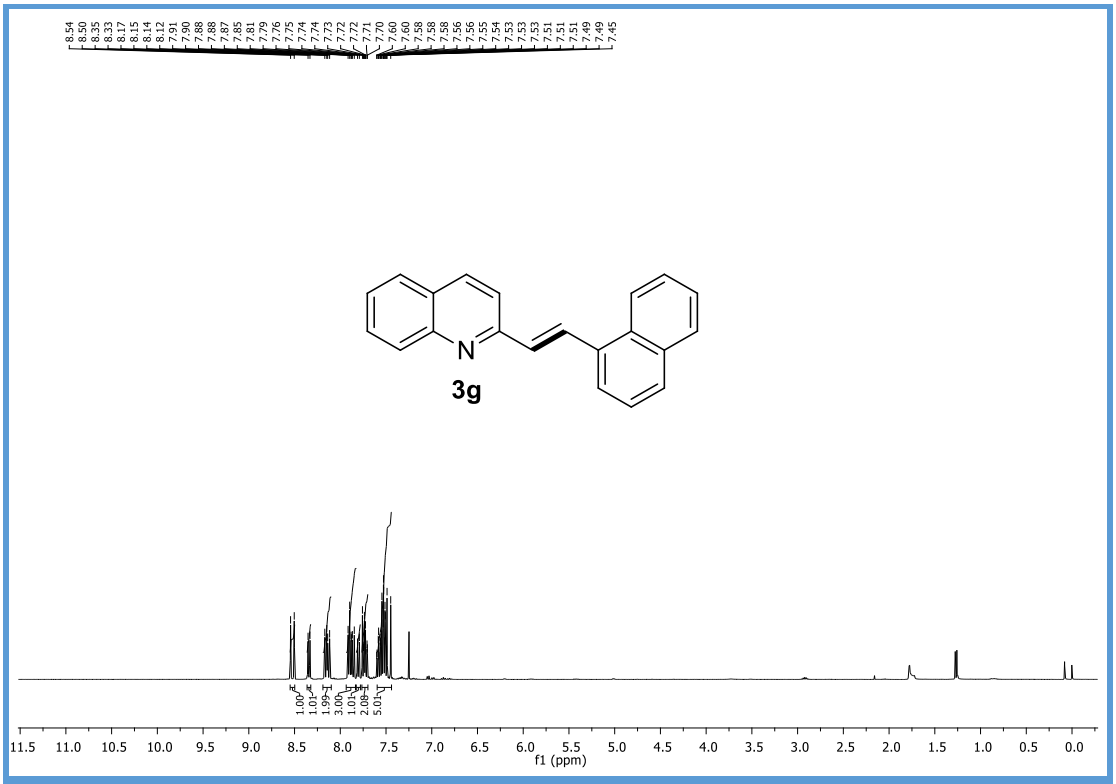

156.26
149.73
148.30
136.28
134.44
129.71
129.17
128.18
127.51
127.34
127.31
126.93
126.02
119.18
34.02
23.90
N
3d
200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0
f1 (ppm)

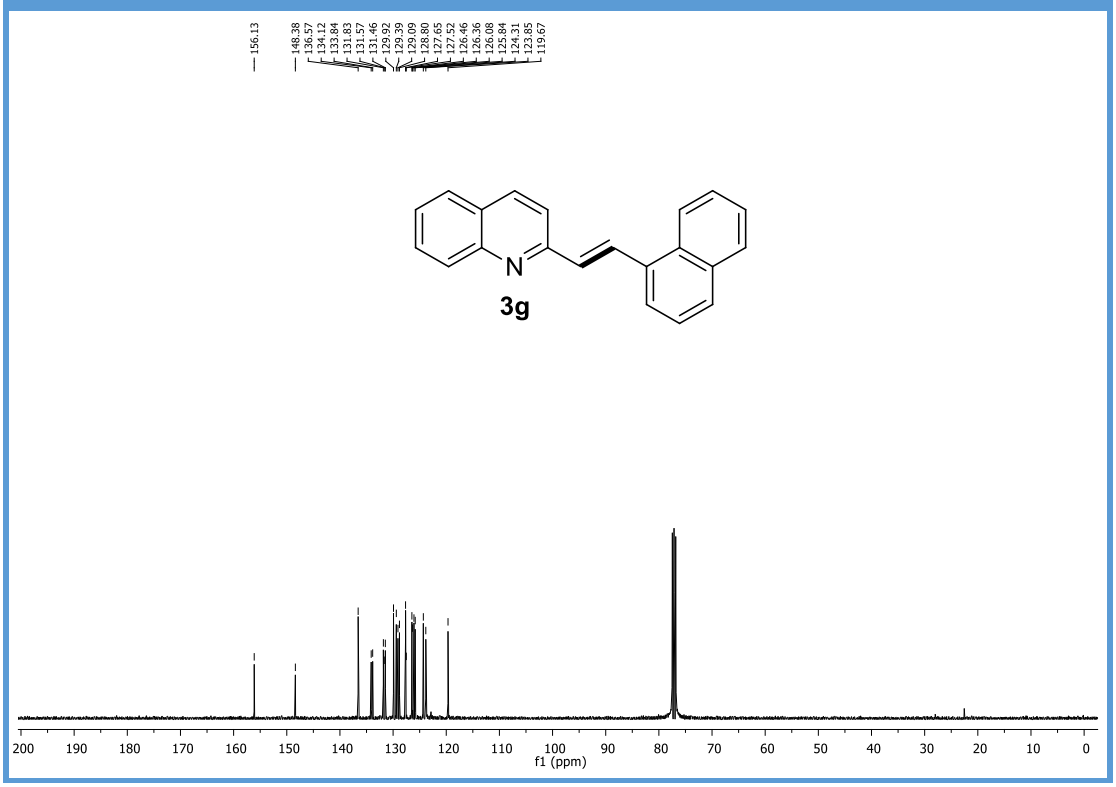

3e
f1 (ppm)

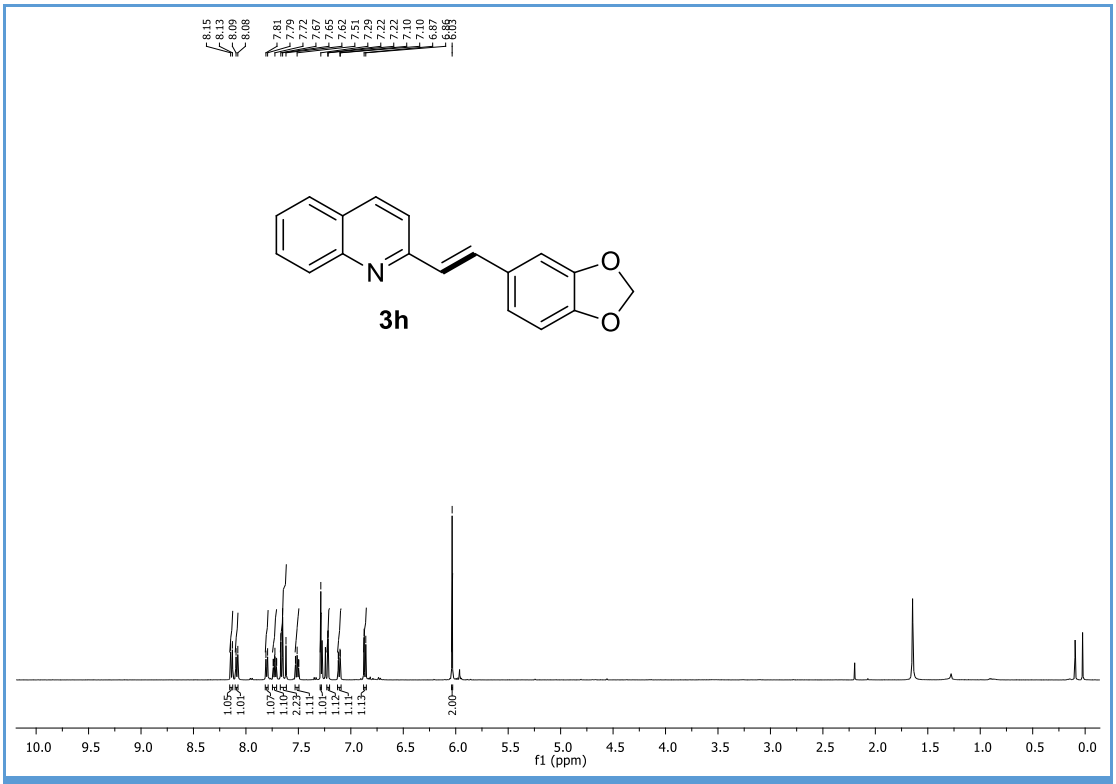

3e
f1 (ppm)

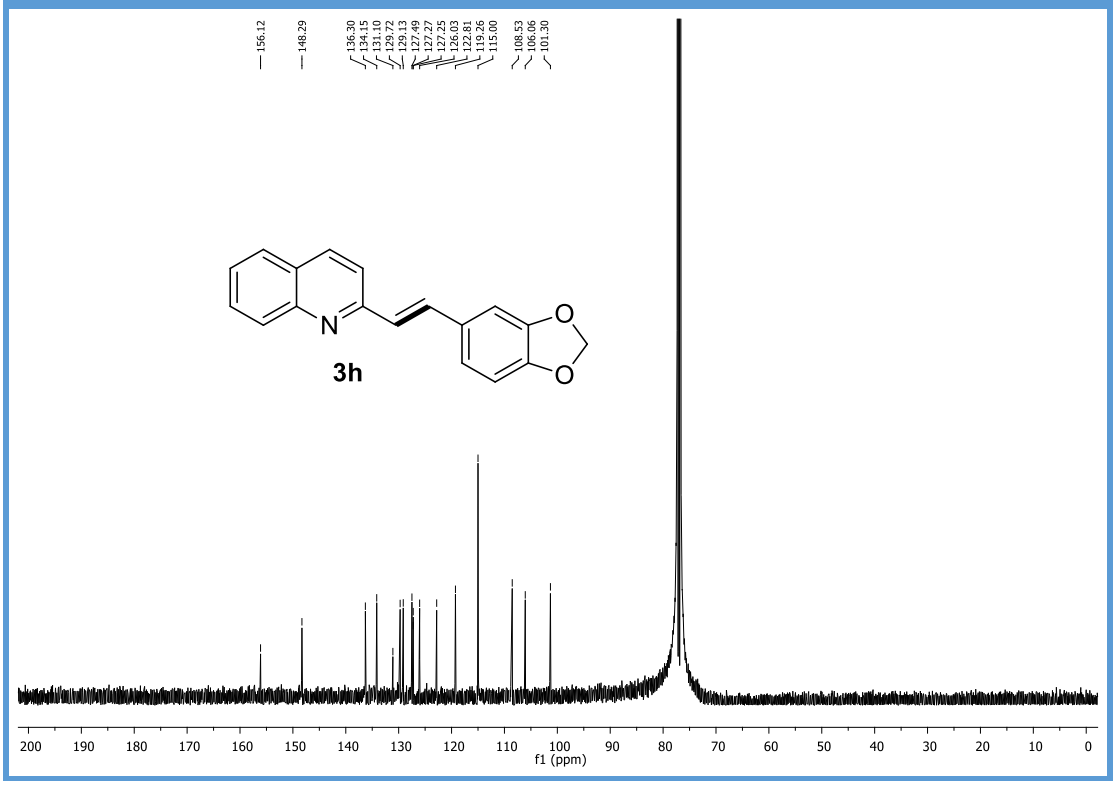

8.10
8.08
8.05
7.65
7.62
7.59
7.57
7.30
7.25
6.94
6.92
6.92
6.91
3.84
N
3f
OMe
2.08
1.08
2.07
1.08
2.09
1.01
1.16
2.04
3.00
10.0 9.5 9.0 8.5 8.0 7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 0.5 0.0
f1 (ppm)

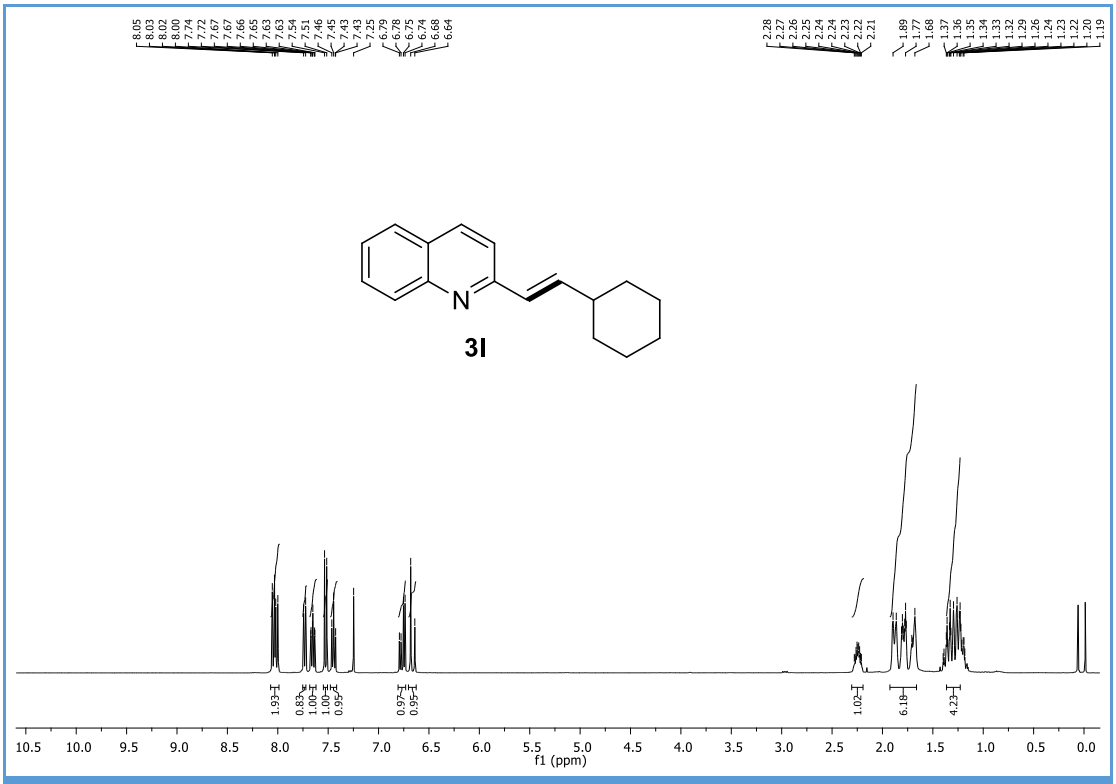

160.18
156.43
148.33
136.38
134.14
129.81
129.34
129.13
128.77
127.60
127.29
126.87
126.03
119.21
114.30
55.45
N
3f
OMe
200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0
f1 (ppm)

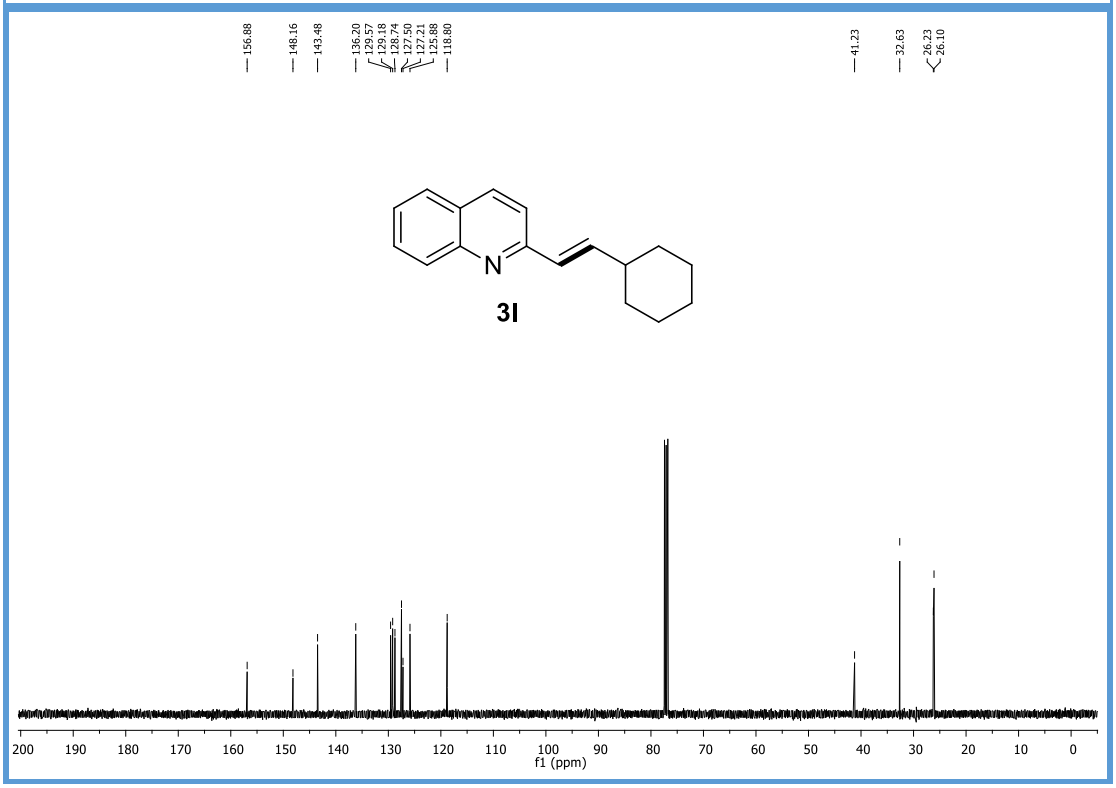

8.54
8.50
8.35
8.33
8.17
8.15
8.14
8.12
7.91
7.90
7.88
7.88
7.87
7.85
7.81
7.79
7.76
7.75
7.74
7.74
7.73
7.72
7.72
7.71
7.70
7.60
7.60
7.58
7.58
7.56
7.56
7.55
7.54
7.53
7.53
7.51
7.51
7.51
7.49
7.49
7.45
N
3g
1.00
1.01
1.99
3.00
1.05
2.09
5.01
11.5 11.0 10.5 10.0 9.5 9.0 8.5 8.0 7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 0.5 0.0
f1 (ppm)

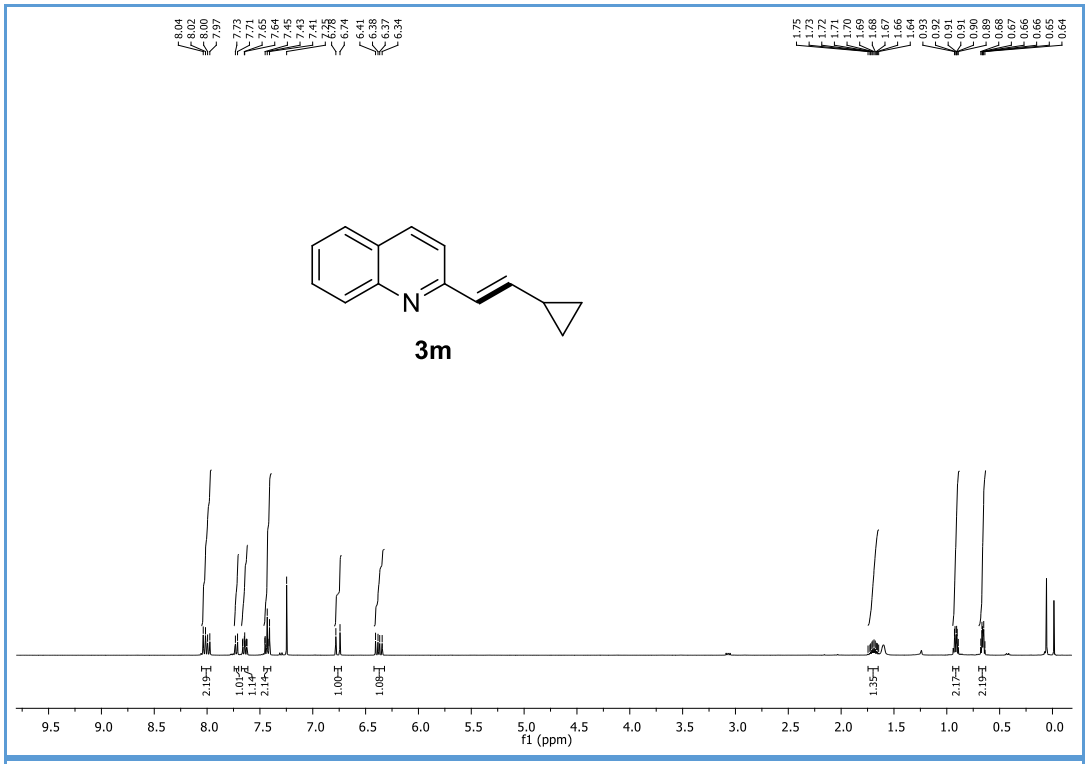

156.13
148.38
136.57
134.12
133.84
131.83
131.57
131.46
129.92
129.39
129.09
128.80
127.65
127.52
126.46
126.36
126.08
125.84
124.31
123.85
119.67
N
3g
200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0
f1 (ppm)

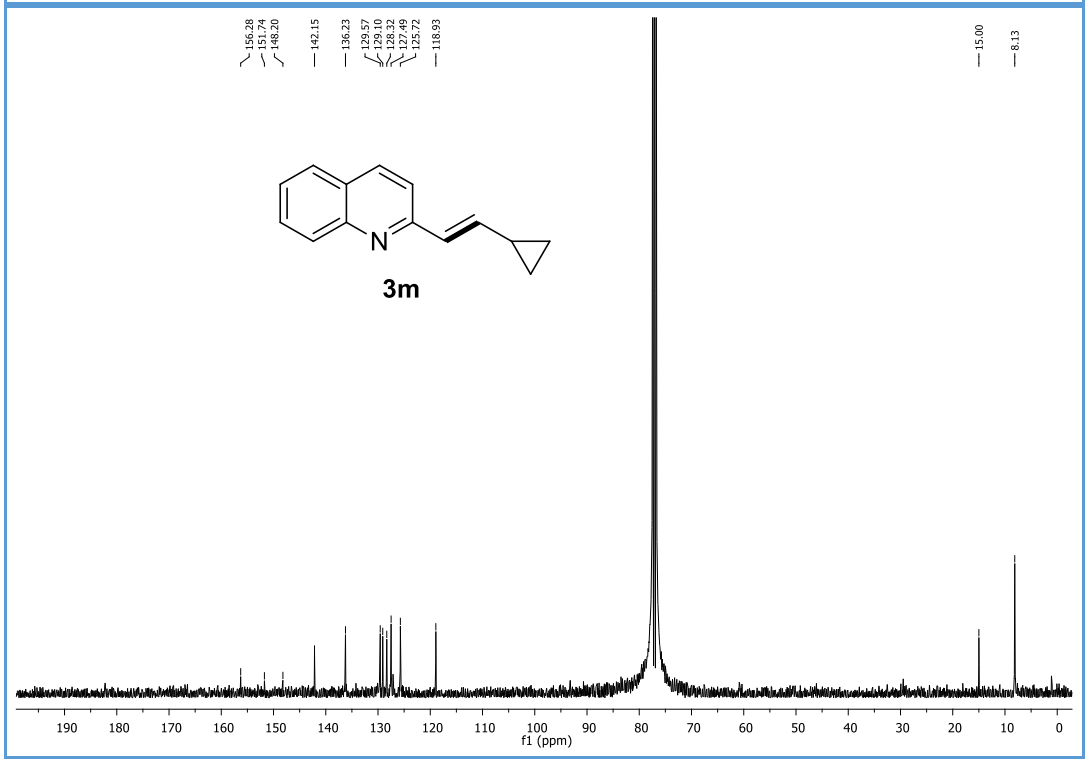

8.15
8.13
8.09
8.08
7.81
7.79
7.72
7.67
7.65
7.62
7.51
7.29
7.22
7.22
7.10
6.87
6.86
6.83
N
O
O
3h
1.05
1.01
1.07
1.10
2.29
1.14
1.01
1.12
1.11
1.13
2.00
10.0
9.5
9.0
8.5
8.0
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
0.5
0.0
f1 (ppm)

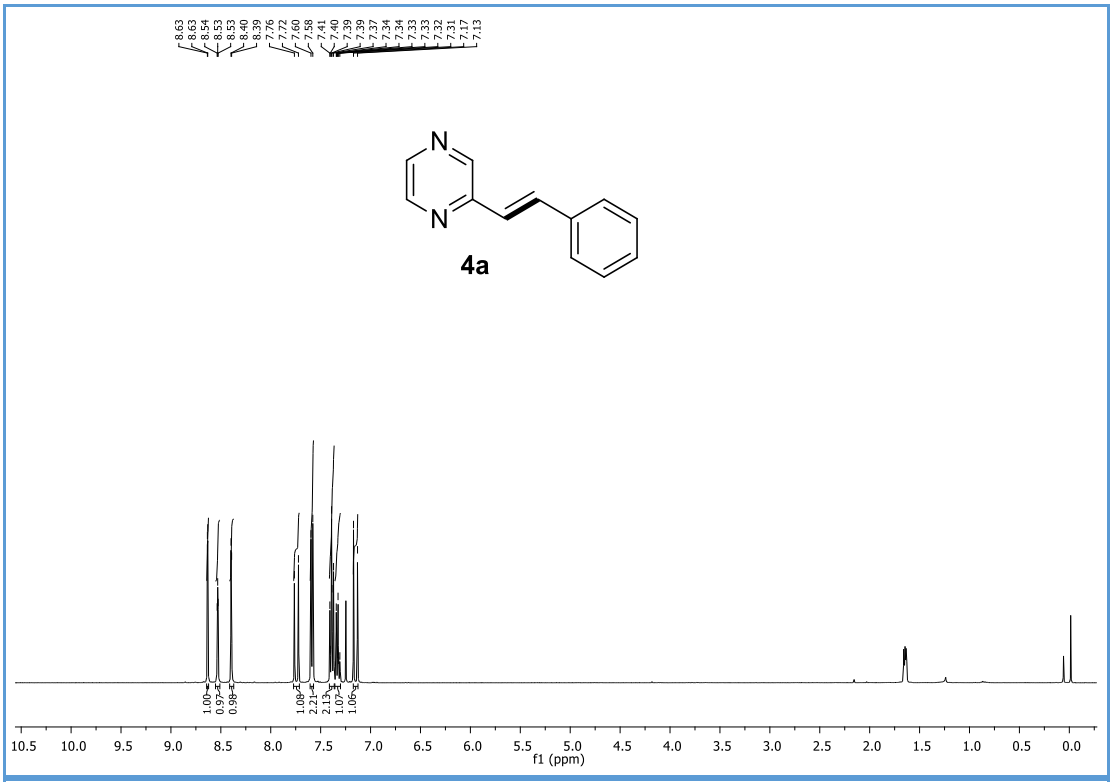

156.12
148.29
136.30
134.15
131.10
129.72
129.13
127.49
127.27
127.25
126.03
122.81
119.26
115.00
108.53
106.06
101.30
N
O
O
3h
200
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
f1 (ppm)

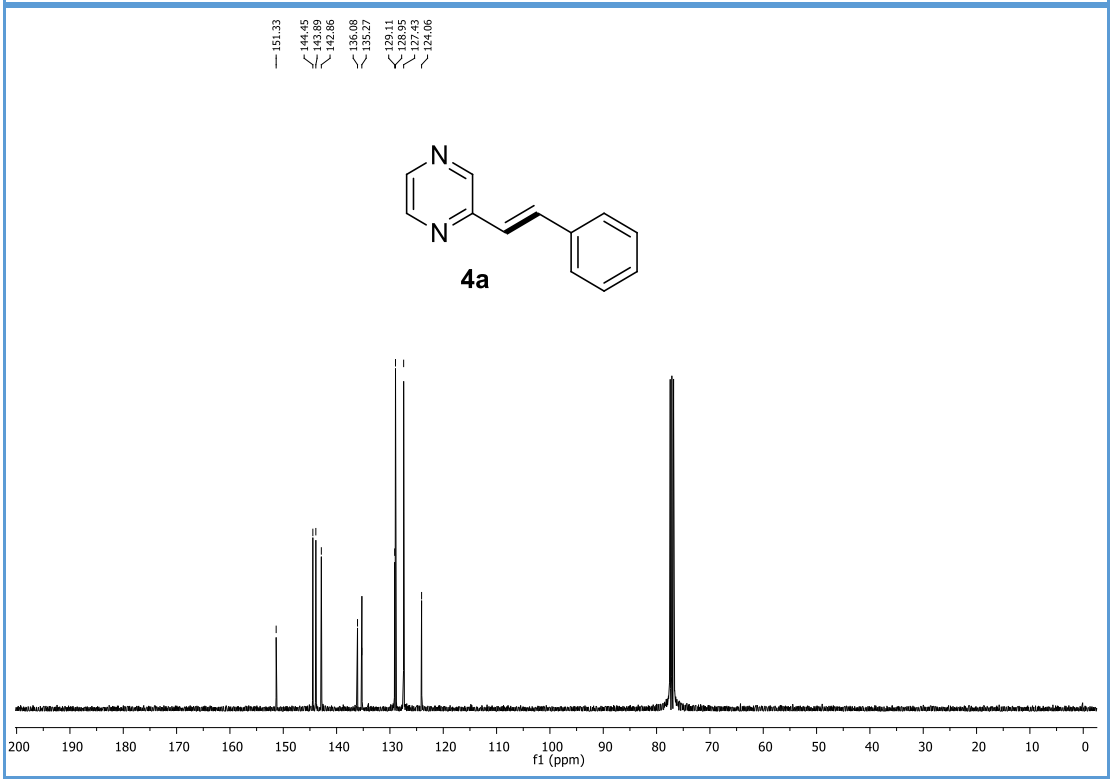

8.05
8.03
8.02
8.00
7.74
7.72
7.67
7.67
7.66
7.65
7.63
7.54
7.51
7.46
7.45
7.43
7.43
7.25
6.79
6.78
6.75
6.74
6.68
6.64
2.28
2.27
2.26
2.25
2.24
2.24
2.23
2.22
2.21
1.89
1.77
1.68
1.37
1.36
1.35
1.34
1.33
1.32
1.29
1.26
1.24
1.23
1.22
1.20
1.19
N
3l
1.93
0.83
1.00
1.00
0.95
0.97
0.95
1.02
6.19
4.23
10.5 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)

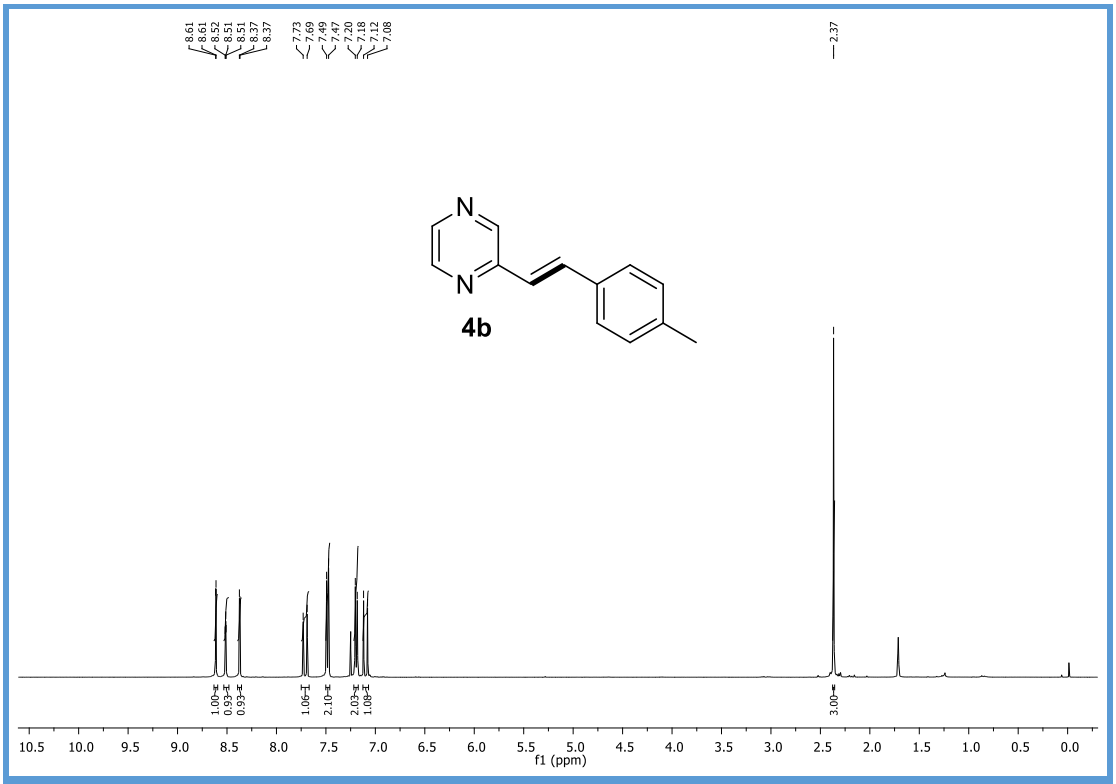

156.88
148.16
143.48
136.20
129.57
129.18
128.74
127.50
127.21
125.88
118.80
41.23
32.63
26.23
26.10
N
3l
200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0
f1 (ppm)

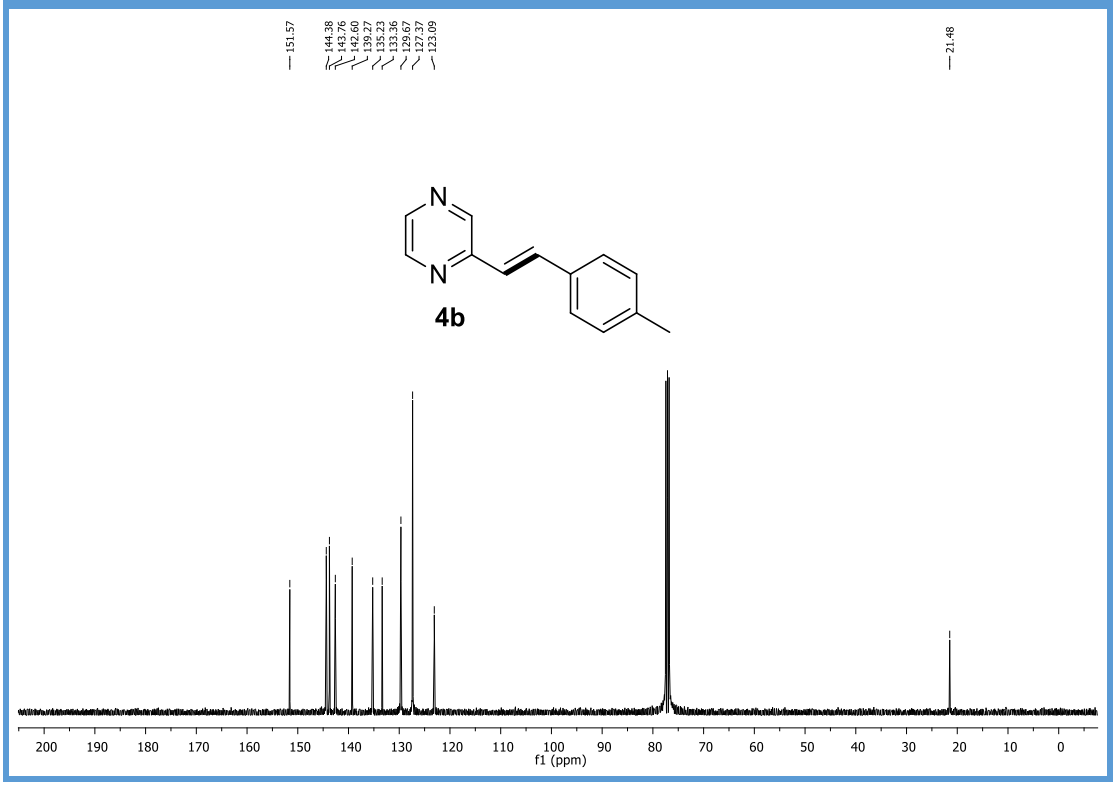

8.04
8.02
8.00
7.97
7.73
7.71
7.65
7.64
7.45
7.43
7.41
6.78
6.74
6.41
6.38
6.37
6.34
1.75
1.73
1.72
1.71
1.70
1.69
1.68
1.67
1.66
1.64
0.93
0.92
0.91
0.91
0.90
0.89
0.68
0.67
0.66
0.66
0.65
0.64
N
3m
2.19
1.01
1.14
2.14
1.00
1.08
1.35
2.17
2.19
9.5 9.0 8.5 8.0 7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 0.5 0.0
f1 (ppm)

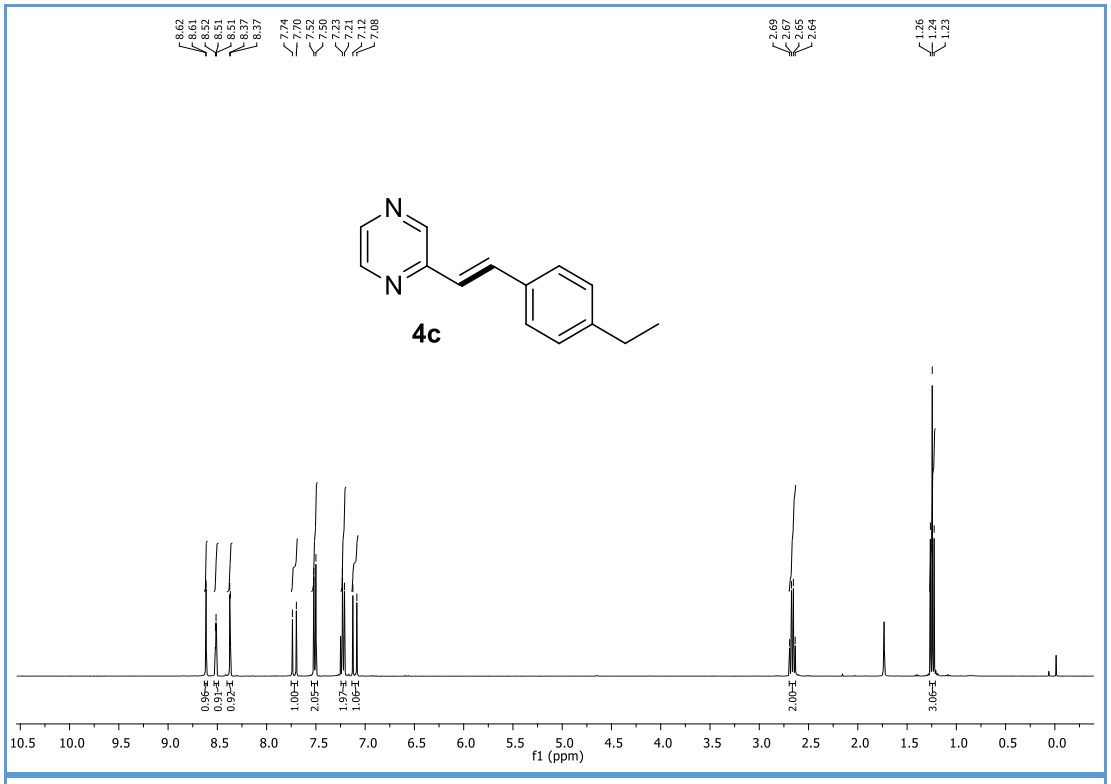

156.28
151.74
148.20
142.15
136.23
129.57
129.10
128.32
127.49
125.72
118.93
15.00
8.13
N
3m
190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0
f1 (ppm)

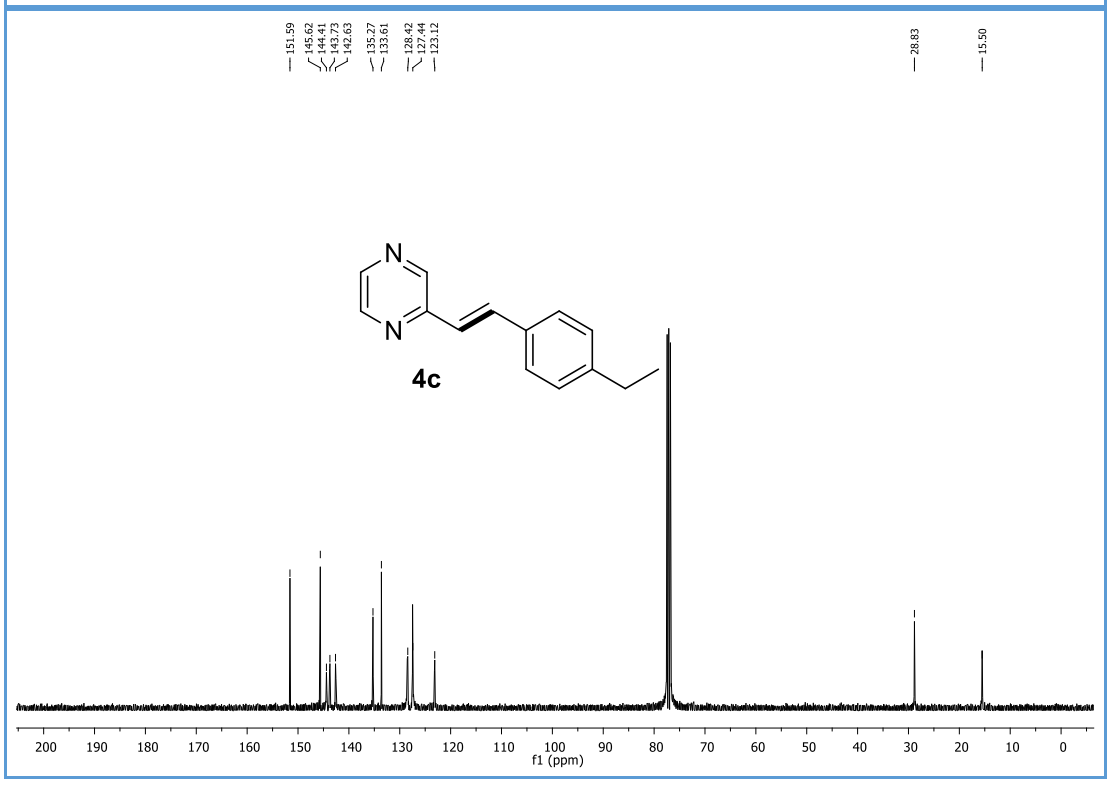

8.63
8.63
8.54
8.53
8.53
8.40
8.39
7.76
7.72
7.60
7.58
7.41
7.40
7.39
7.39
7.37
7.34
7.34
7.33
7.32
7.31
7.17
7.13
N
N
4a
1.00
0.97
0.98
1.09
2.21
2.13
1.07
1.06
10.5 10.0 9.5 9.0 8.5 8.0 7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 0.5 0.0
f1 (ppm)

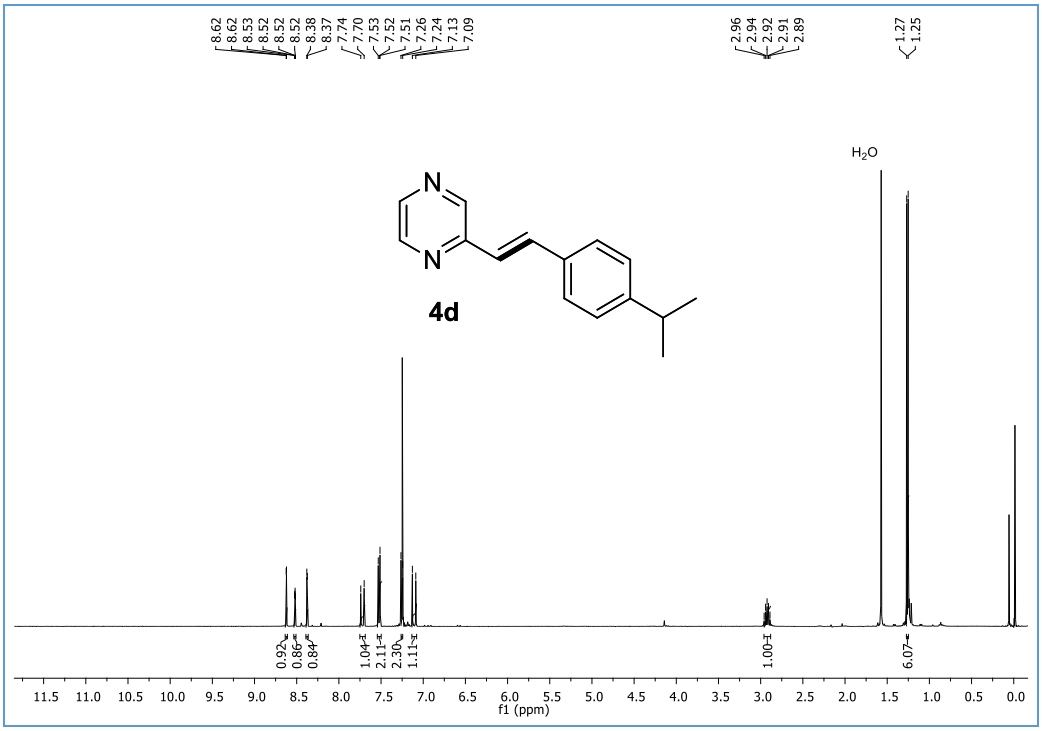

151.33
144.45
143.89
142.86
136.08
135.27
129.11
128.95
127.43
124.06
N
N
4a
200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0
f1 (ppm)

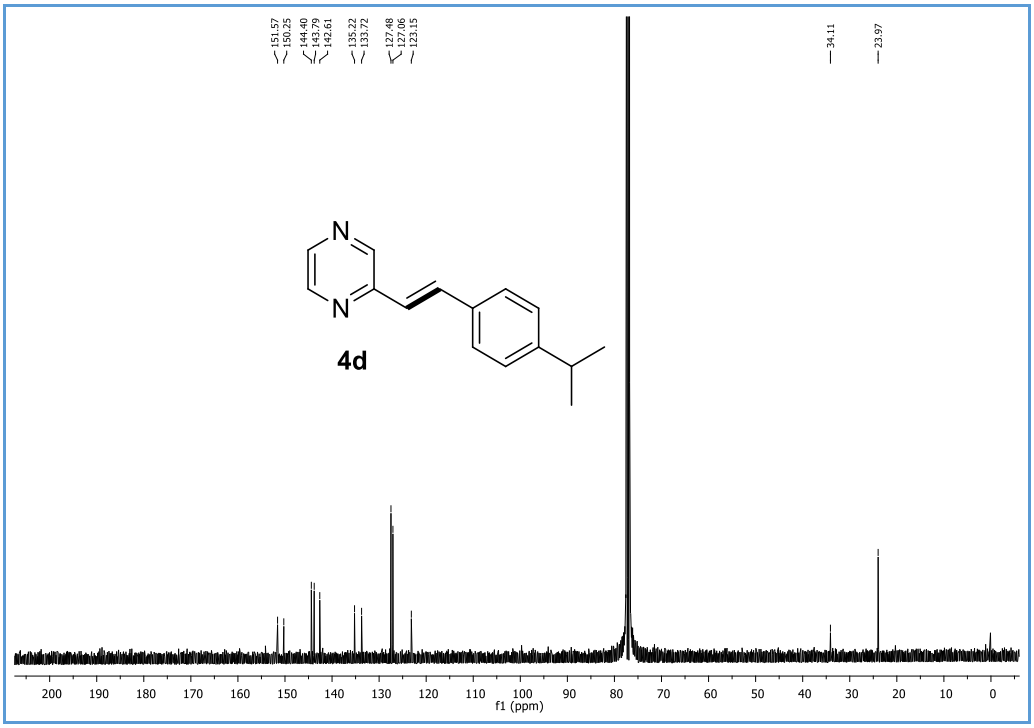

8.61
8.61
8.52
8.51
8.51
8.37
8.37
7.73
7.69
7.49
7.47
7.20
7.18
7.12
7.08
2.37
N
N
4b
1.00
0.93
0.93
1.06
2.10
2.03
1.08
3.00
10.5 10.0 9.5 9.0 8.5 8.0 7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 0.5 0.0
f1 (ppm)

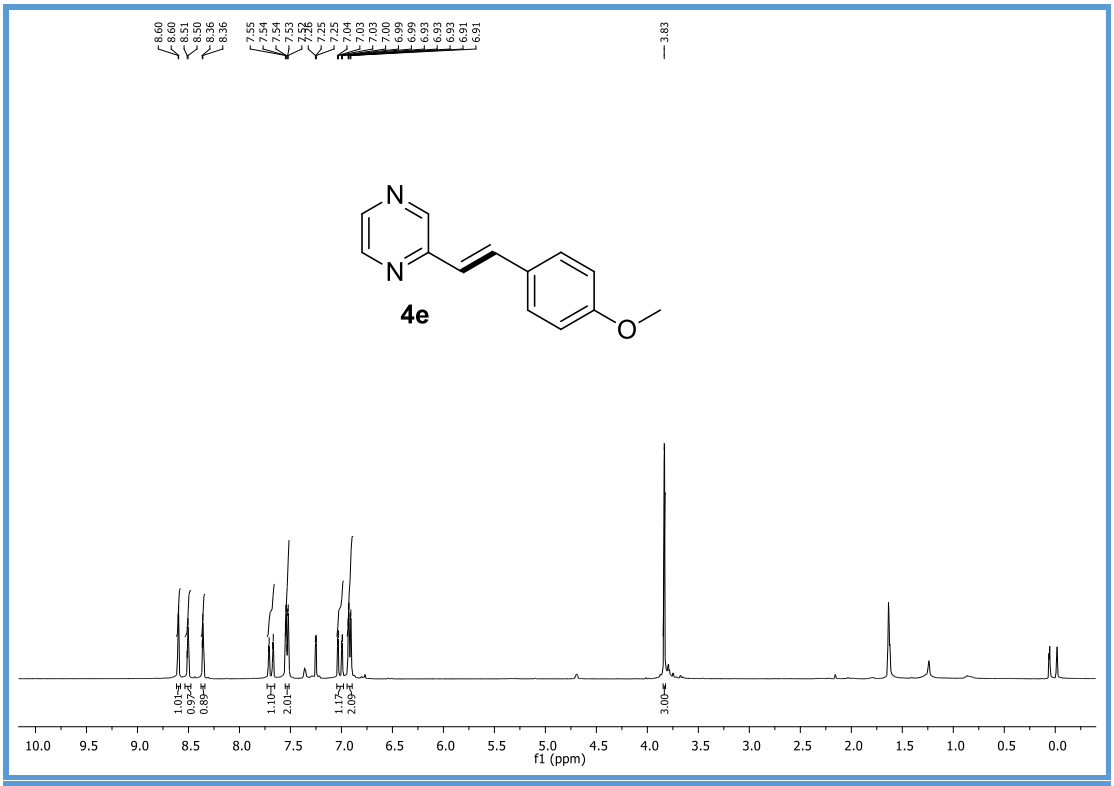

151.57
144.38
143.76
142.60
139.27
135.23
133.36
129.67
127.37
123.09
21.48
N
N
4b
200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0
f1 (ppm)

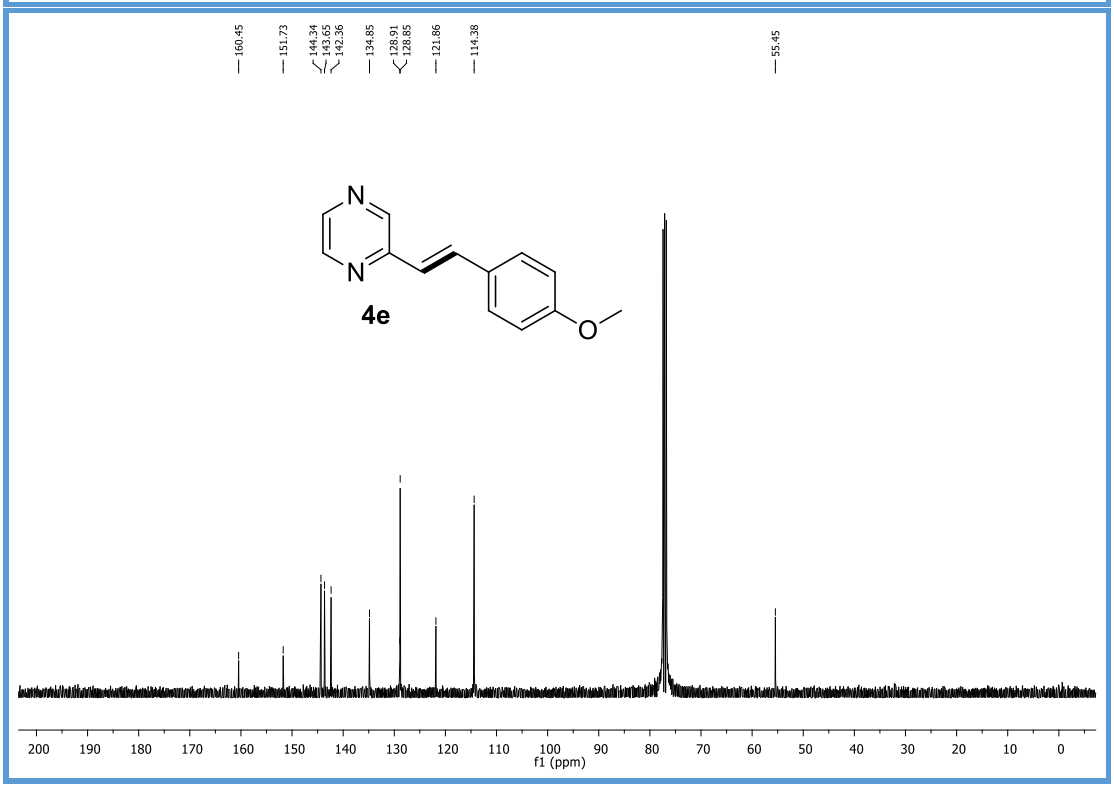

8.62
8.61
8.52
8.51
8.51
8.37
8.37
7.74
7.70
7.52
7.50
7.23
7.21
7.12
7.08
2.69
2.67
2.65
2.64
1.26
1.24
1.23
N
N
4c
0.96
0.91
0.92
1.00
2.05
1.97
1.06
2.00
3.06
10.5 10.0 9.5 9.0 8.5 8.0 7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 0.5 0.0
f1 (ppm)

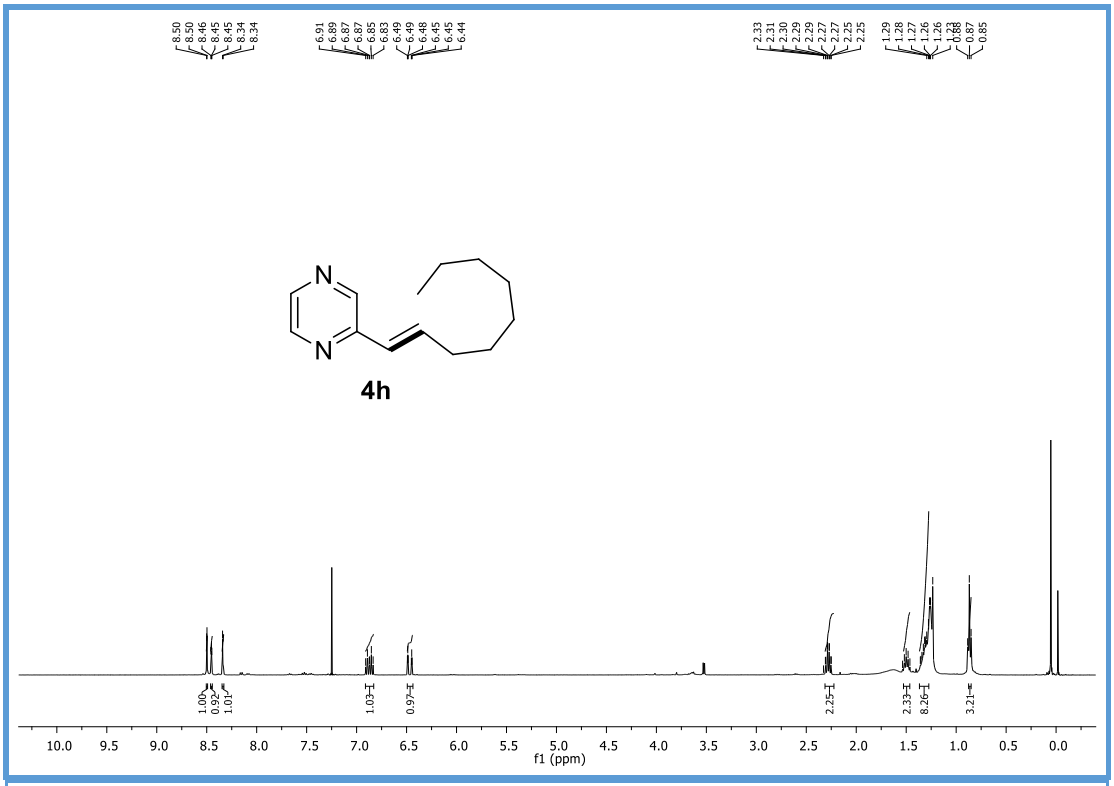

151.59
145.62
144.41
143.73
142.63
135.27
133.61
128.42
127.44
123.12
28.83
15.50
N
N
4c
200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0
f1 (ppm)

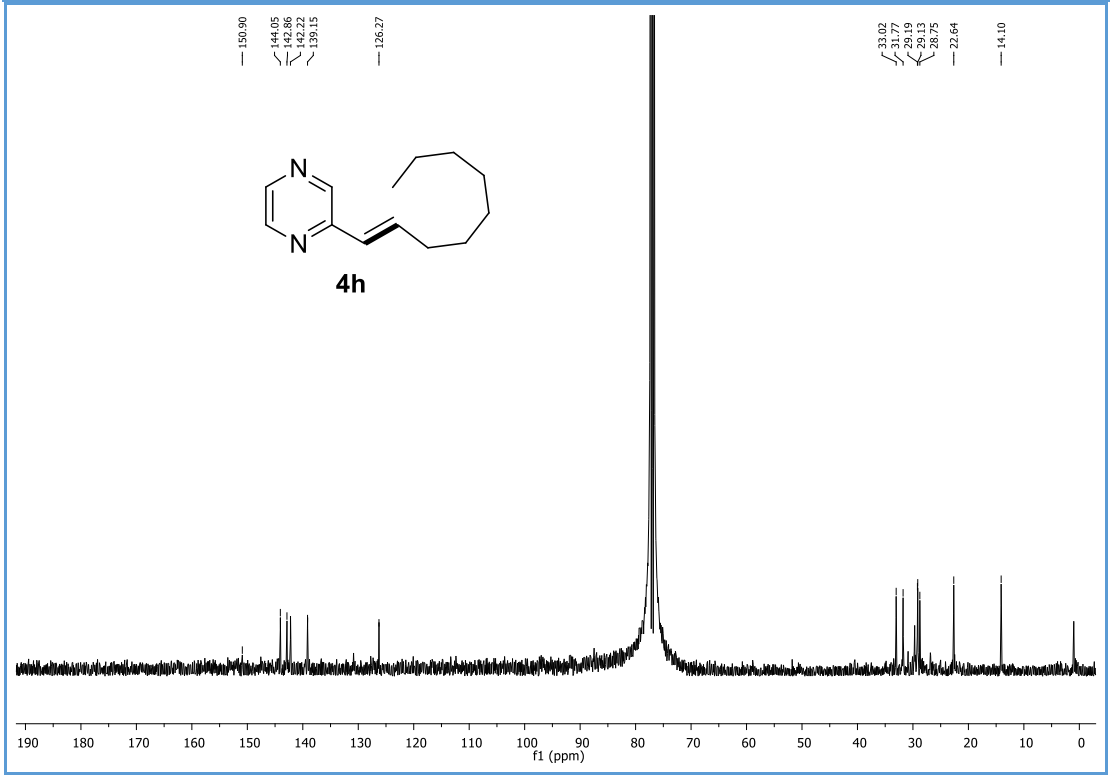

8.62
8.62
8.53
8.52
8.52
8.52
8.38
8.37
7.74
7.70
7.53
7.52
7.51
7.26
7.24
7.13
7.09
2.96
2.94
2.92
2.91
2.89
1.27
1.25
H2O
4d
0.92
0.86
0.84
1.04
2.11
2.30
1.11
1.00
6.07
f1 (ppm)

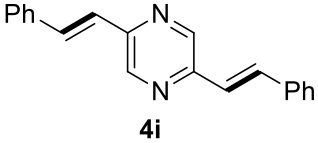

151.57
150.25
144.40
143.79
142.61
135.22
133.72
127.48
127.06
123.15
34.11
23.97
4d
f1 (ppm)


8.60
8.60
8.51
8.50
8.36
8.36
7.55
7.54
7.54
7.53
7.52
7.52
7.25
7.25
7.04
7.03
7.03
7.00
6.99
6.93
6.93
6.91
6.91
3.83
4e
1.01
0.97
0.89
1.10
2.01
1.17
2.09
3.00
10.0 9.5 9.0 8.5 8.0 7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 0.5 0.0
f1 (ppm)

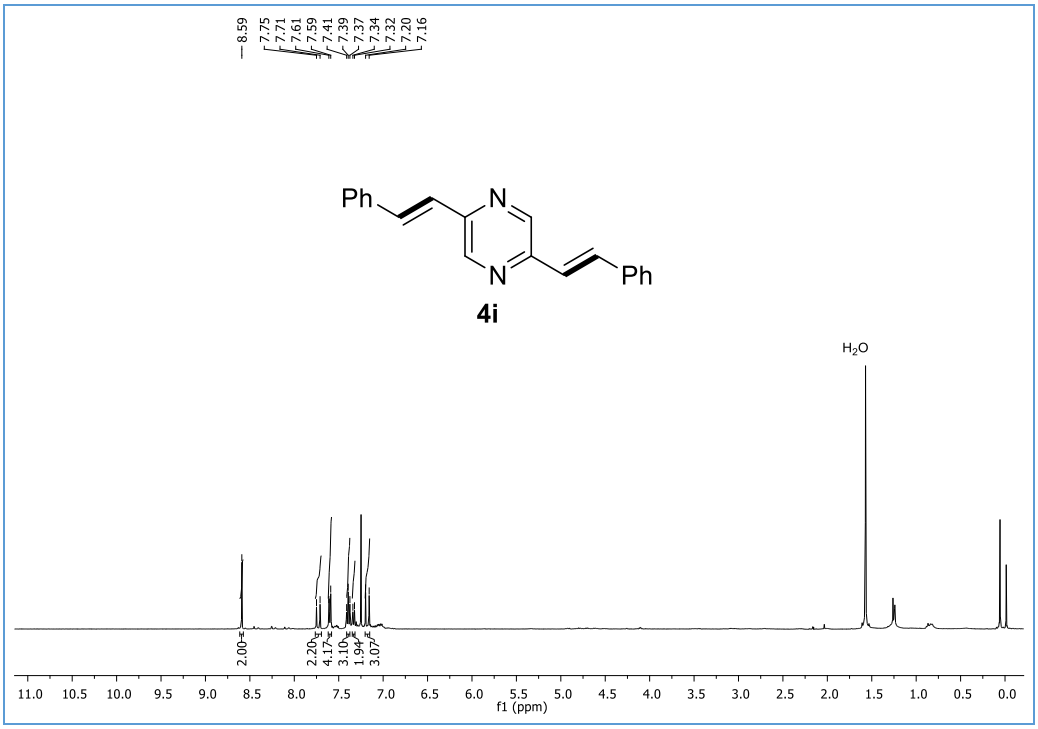

160.45
151.73
144.34
143.65
142.36
134.85
128.91
128.85
121.86
114.38
55.45
4e
200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0
f1 (ppm)

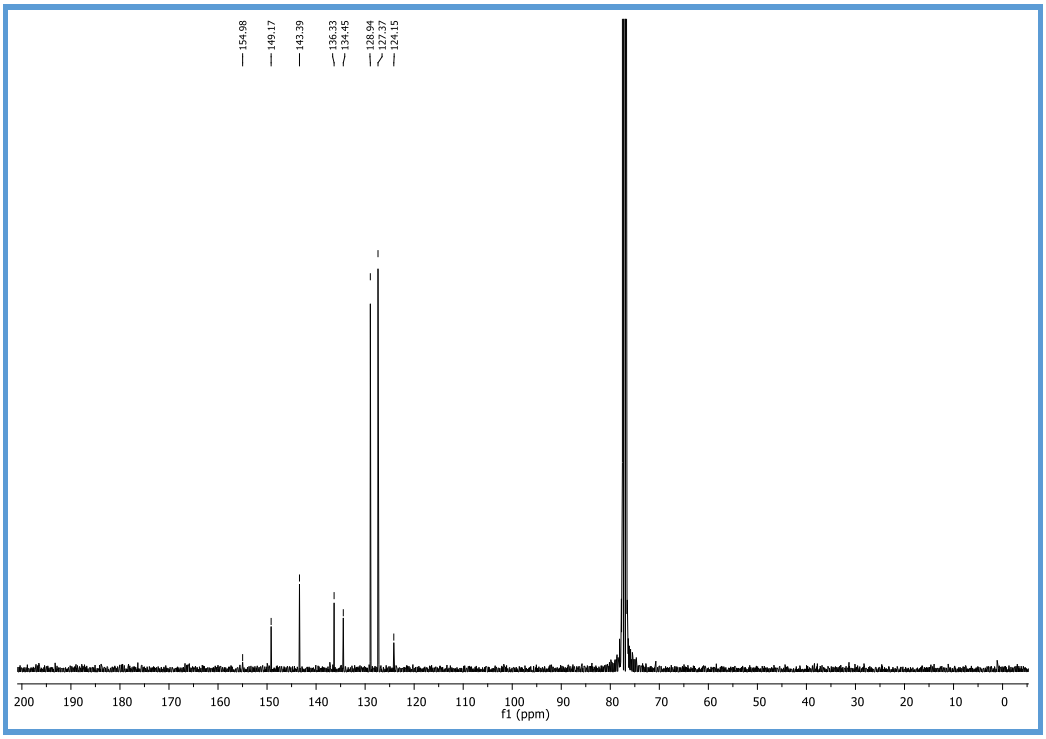

8.50
8.50
8.46
8.45
8.45
8.34
8.34
6.91
6.89
6.87
6.87
6.85
6.83
6.49
6.49
6.48
6.45
6.45
6.44
2.33
2.31
2.30
2.29
2.29
2.27
2.27
2.25
2.25
1.29
1.28
1.27
1.26
1.26
1.23
0.88
0.87
0.85
N
N
4h
1.00
0.92
1.01
1.03
0.97
2.25
2.33
8.26
3.21
10.0 9.5 9.0 8.5 8.0 7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 0.5 0.0
f1 (ppm)

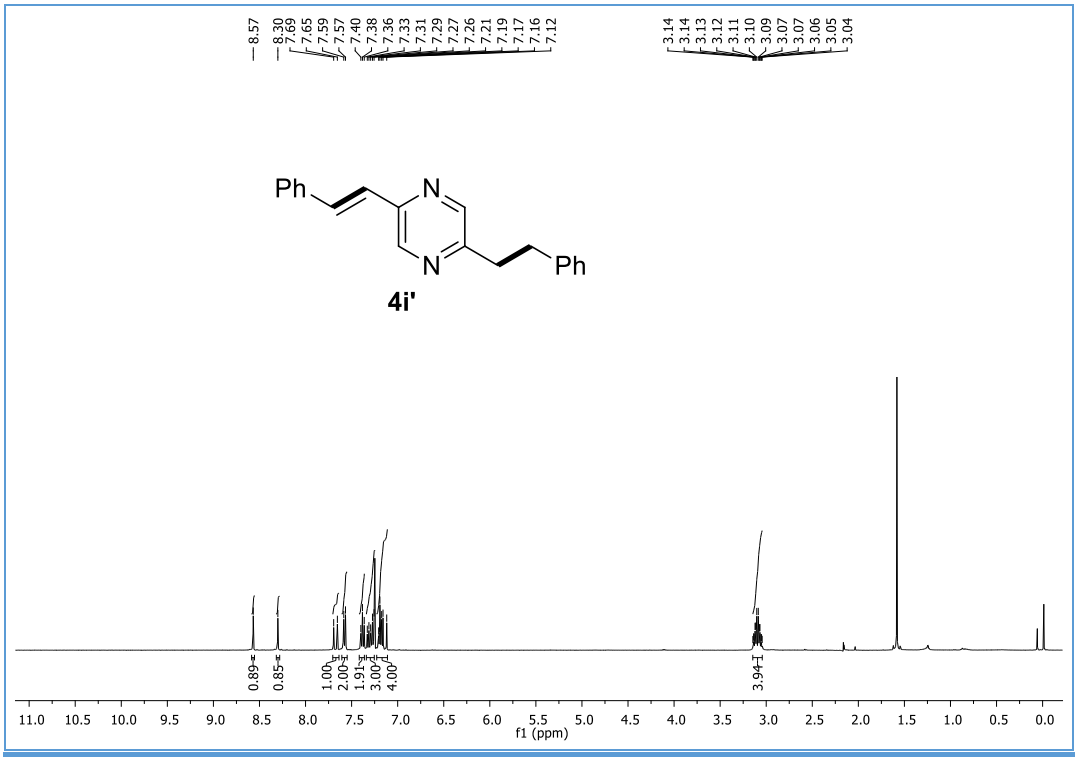

150.90
144.05
142.86
142.22
139.15
126.27
33.02
31.77
29.19
29.13
28.75
22.64
14.10
N
N
4h
190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0
f1 (ppm)

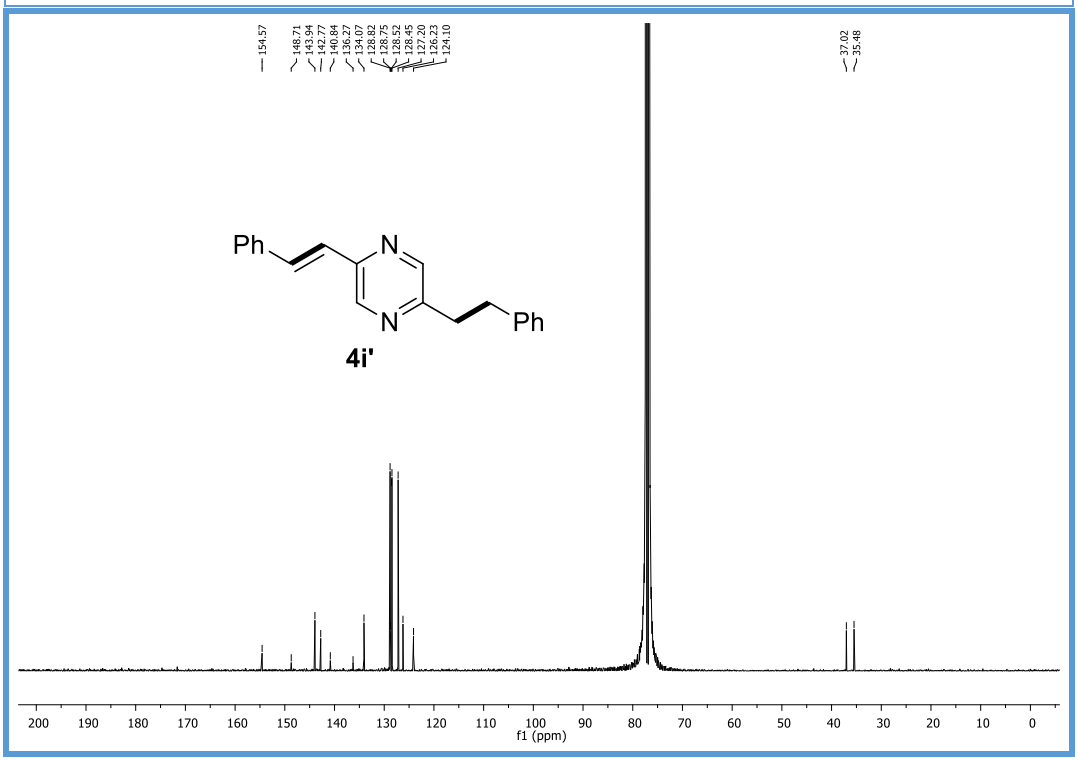

Ph
N
N
Ph

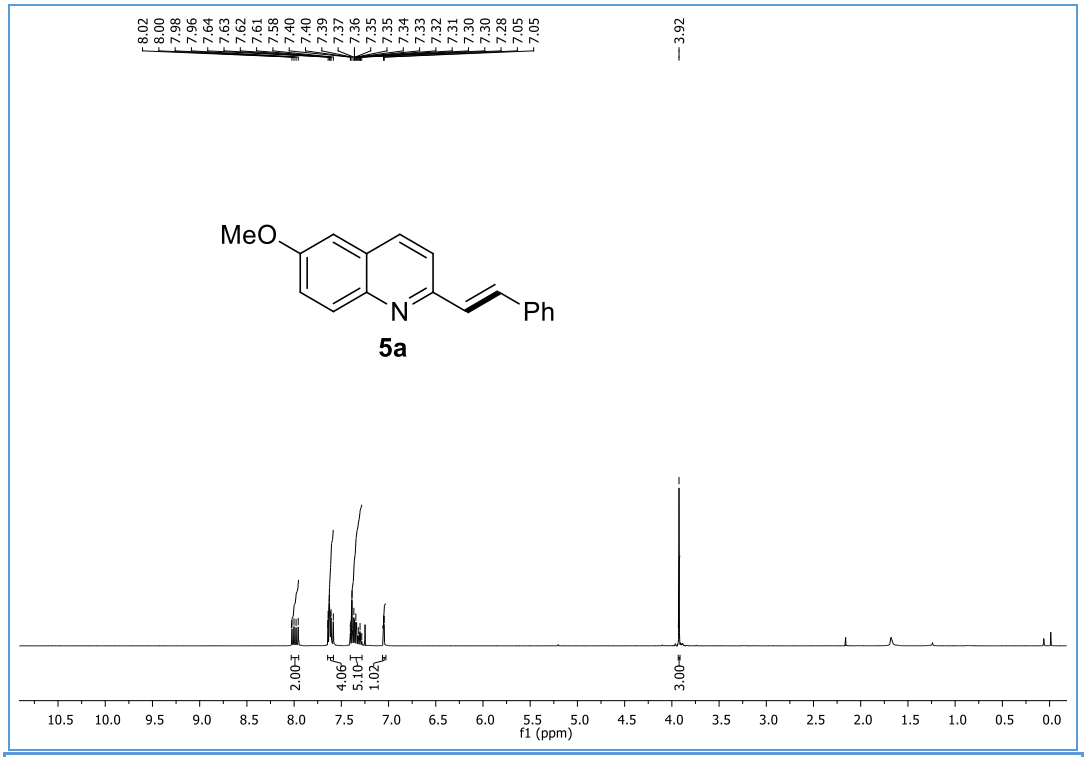
4i

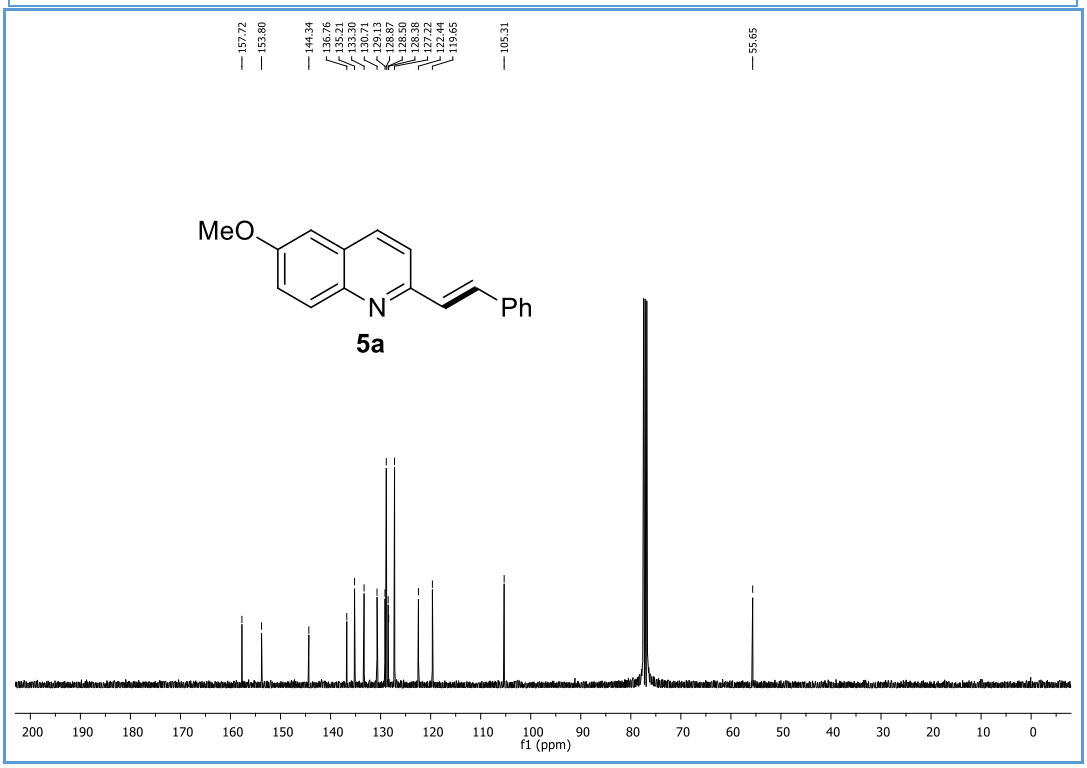

8.59
7.75
7.71
7.61
7.59
7.41
7.39
7.37
7.34
7.32
7.20
7.16
Ph
N
N
Ph
4i
H2O
2.00
2.20
4.17
3.10
1.94
3.07
11.0 10.5 10.0 9.5 9.0 8.5 8.0 7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 0.5 0.0
f1 (ppm)

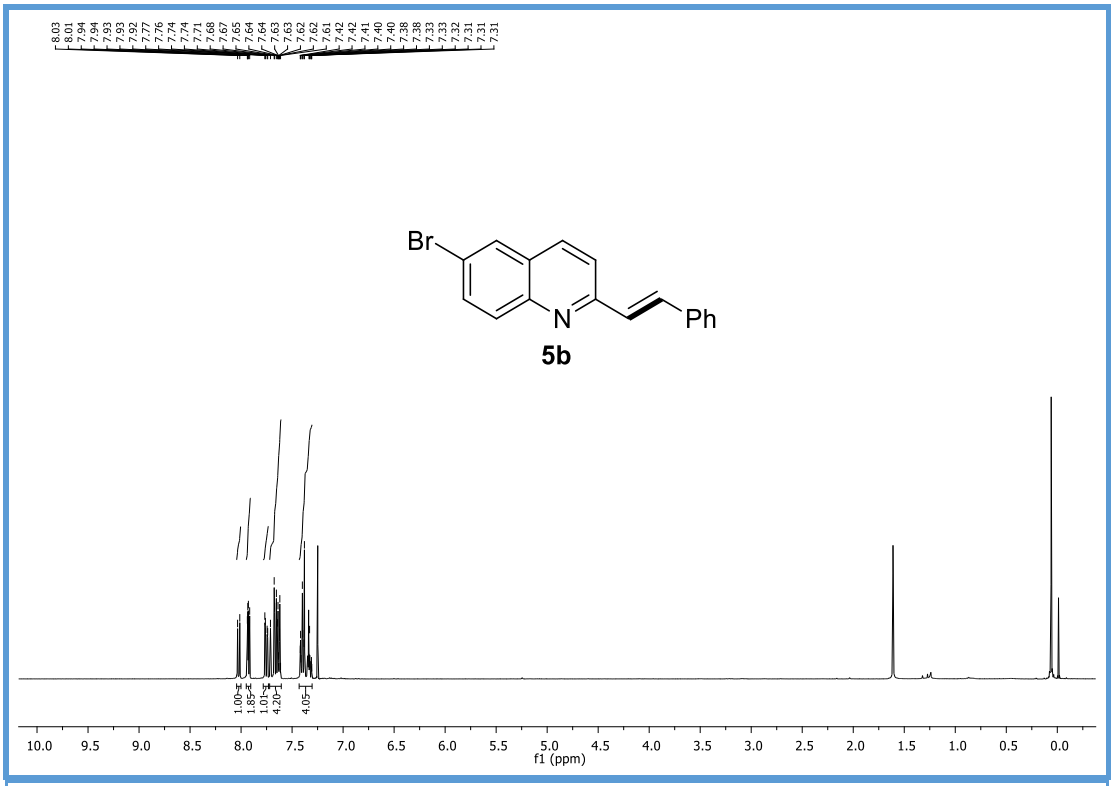

154.98
149.17
143.39
136.33
134.45
128.94
127.37
124.15
200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0
f1 (ppm)

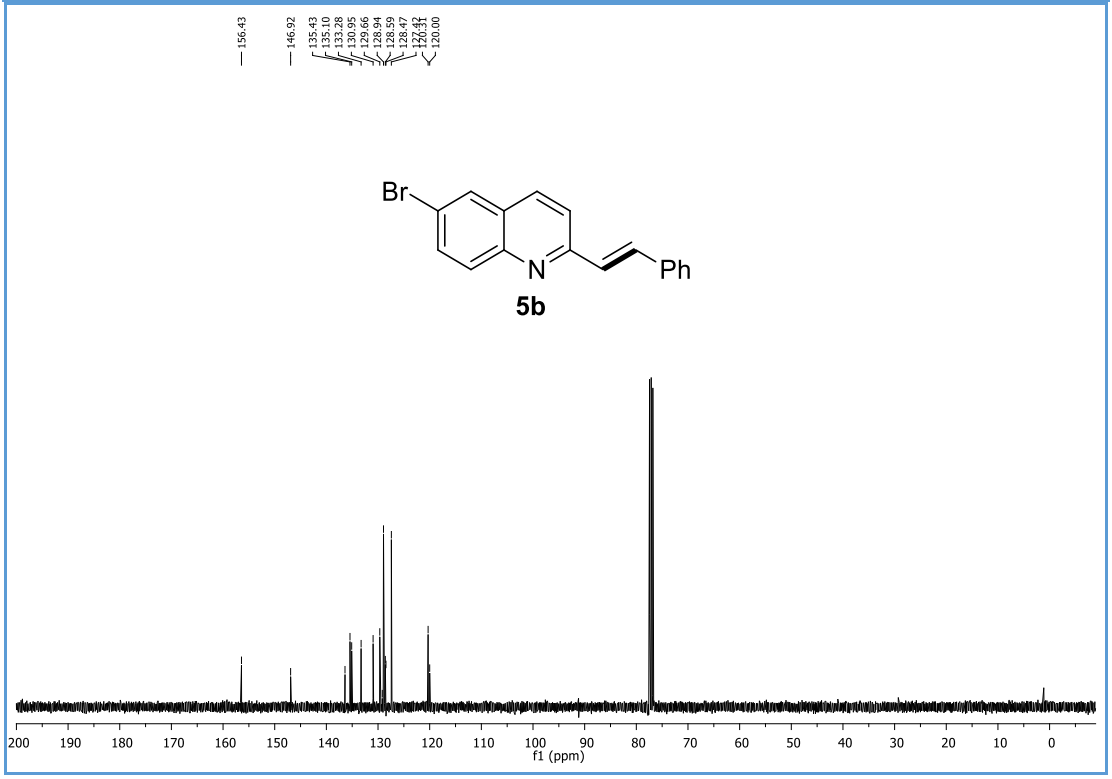

8.57
8.30
7.69
7.65
7.59
7.57
7.40
7.38
7.36
7.33
7.31
7.29
7.27
7.26
7.21
7.19
7.17
7.16
7.12
3.14
3.14
3.13
3.12
3.11
3.10
3.09
3.07
3.07
3.06
3.05
3.04
Ph
N
N
Ph
4i'
0.89
0.85
1.00
2.00
1.91
3.00
4.00
3.94
11.0 10.5 10.0 9.5 9.0 8.5 8.0 7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 0.5 0.0
f1 (ppm)

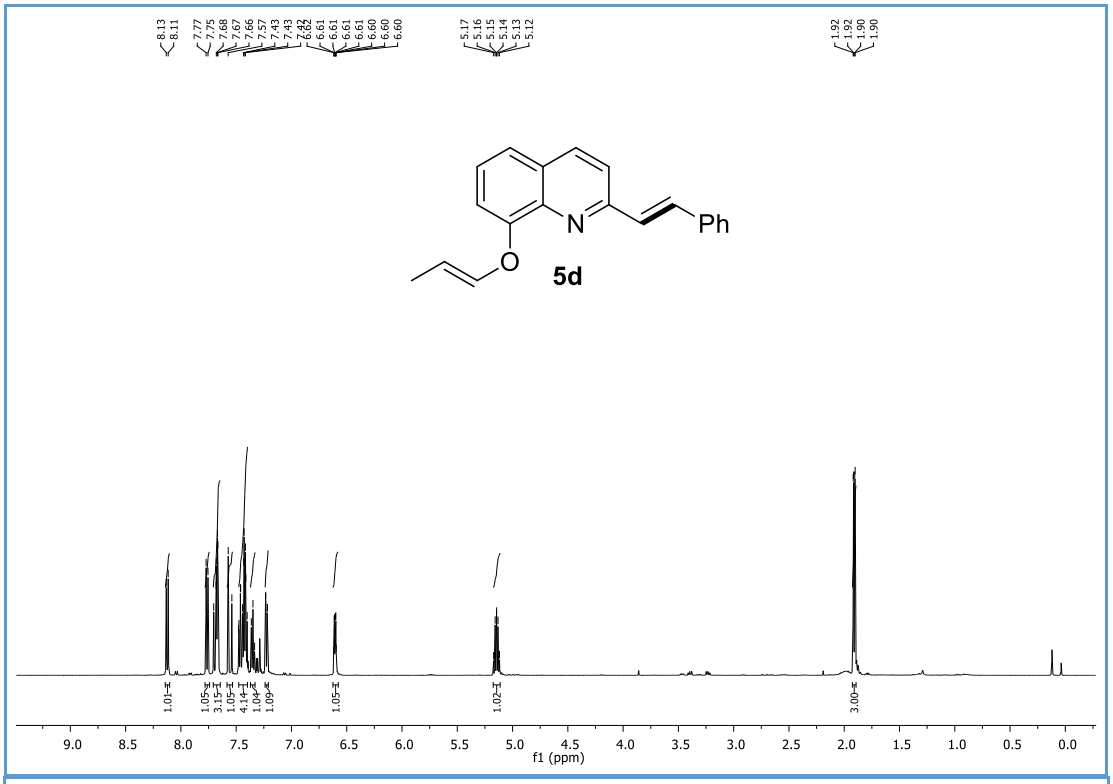

154.57
148.71
143.94
142.77
140.84
136.27
134.07
128.82
128.75
128.52
128.45
127.20
126.23
124.10
37.02
35.48
Ph
N
N
Ph
4i'
200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0
f1 (ppm)

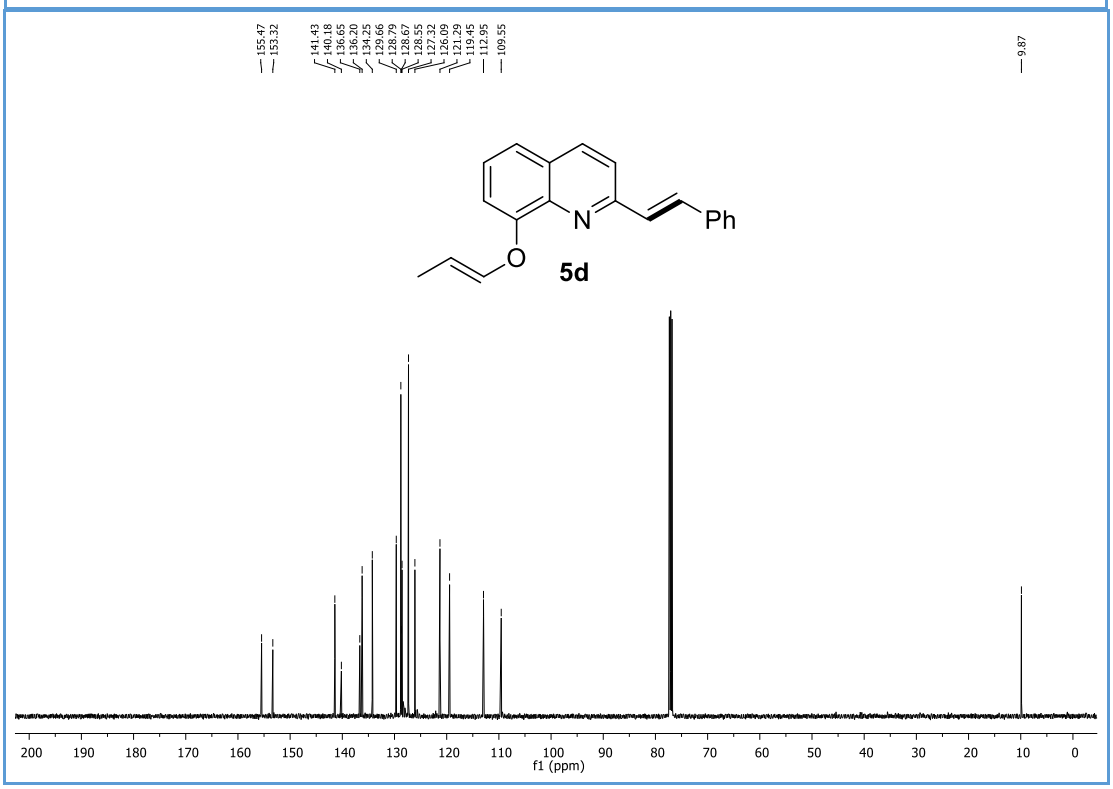

8.02
8.00
7.98
7.96
7.64
7.63
7.62
7.61
7.58
7.40
7.40
7.39
7.37
7.36
7.35
7.35
7.34
7.33
7.32
7.31
7.30
7.30
7.28
7.05
7.05
3.92
MeO
N
Ph
5a
2.00
4.06
5.10
1.02
3.00
10.5 10.0 9.5 9.0 8.5 8.0 7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 0.5 0.0
f1 (ppm)

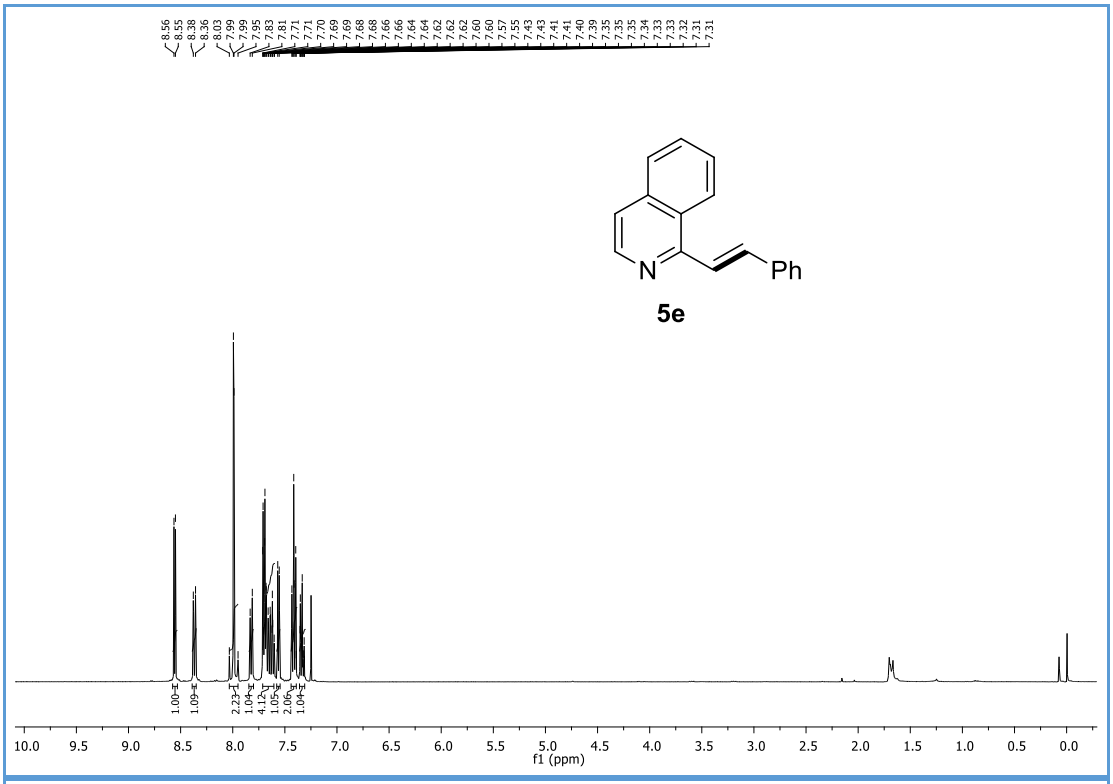

157.72
153.80
144.34
136.76
135.21
132.30
130.71
129.13
128.87
128.50
128.38
127.22
122.44
119.65
105.31
55.65
MeO
N
Ph
5a
200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0
f1 (ppm)

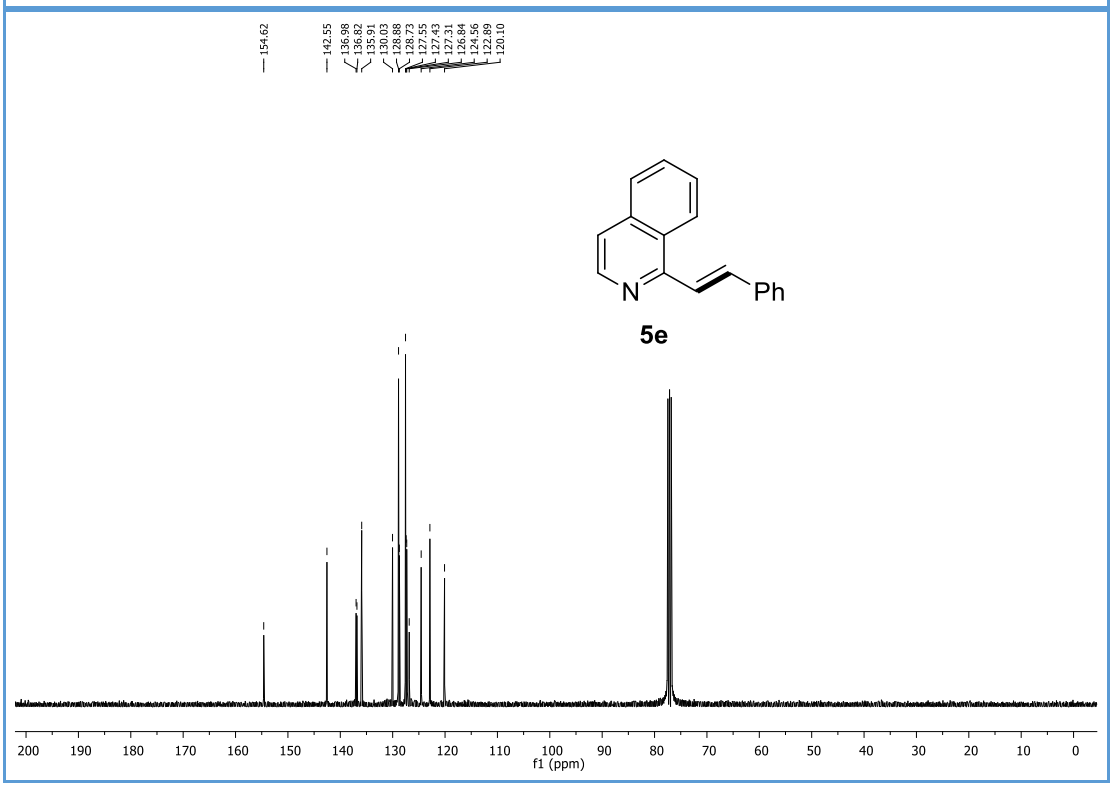

8.03
8.01
7.94
7.94
7.93
7.93
7.92
7.77
7.76
7.74
7.74
7.71
7.68
7.67
7.65
7.64
7.64
7.63
7.63
7.62
7.62
7.61
7.42
7.42
7.41
7.40
7.38
7.33
7.33
7.32
7.31
7.31
7.31
Br
N
Ph
5b
1.00
1.85
1.01
4.20
4.05
10.0
9.5
9.0
8.5
8.0
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
0.5
0.0
f1 (ppm)

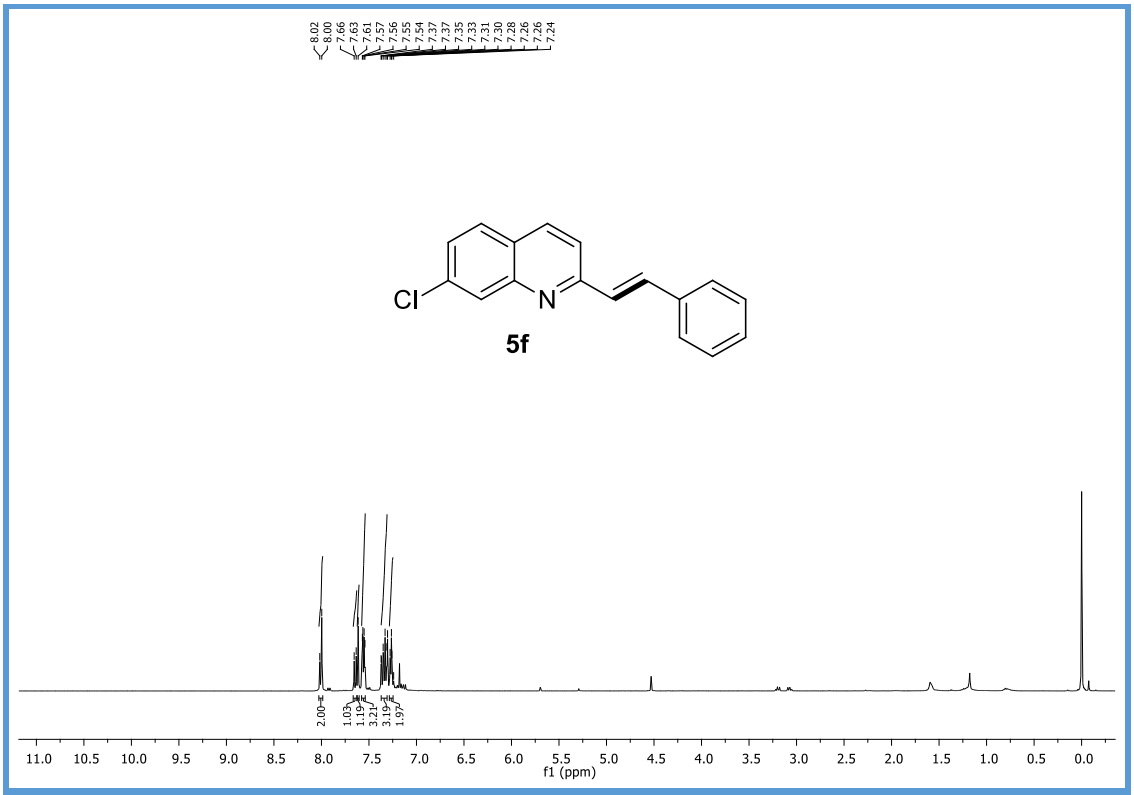

156.43
146.92
135.43
135.10
133.28
130.95
129.66
129.04
128.59
128.47
127.42
120.31
120.00
Br
N
Ph
5b
200
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
f1 (ppm)

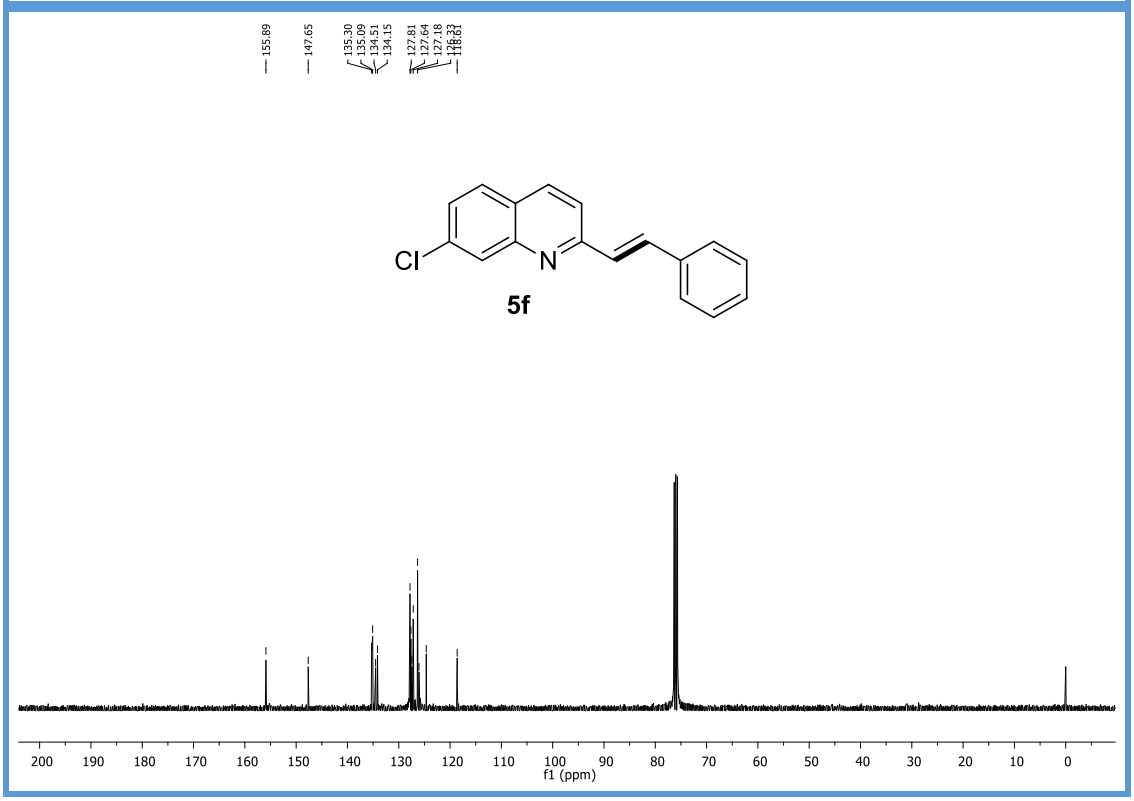

8.13
8.11
7.77
7.75
7.68
7.67
7.66
7.57
7.43
7.42
6.61
6.61
6.61
6.60
6.60
6.60
5.17
5.16
5.15
5.14
5.13
5.12
1.92
1.92
1.90
1.90
N
O
Ph
5d
1.01
1.05
3.15
1.05
4.14
1.04
1.09
1.05
1.02
3.00
9.0
8.5
8.0
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
0.5
0.0
f1 (ppm)

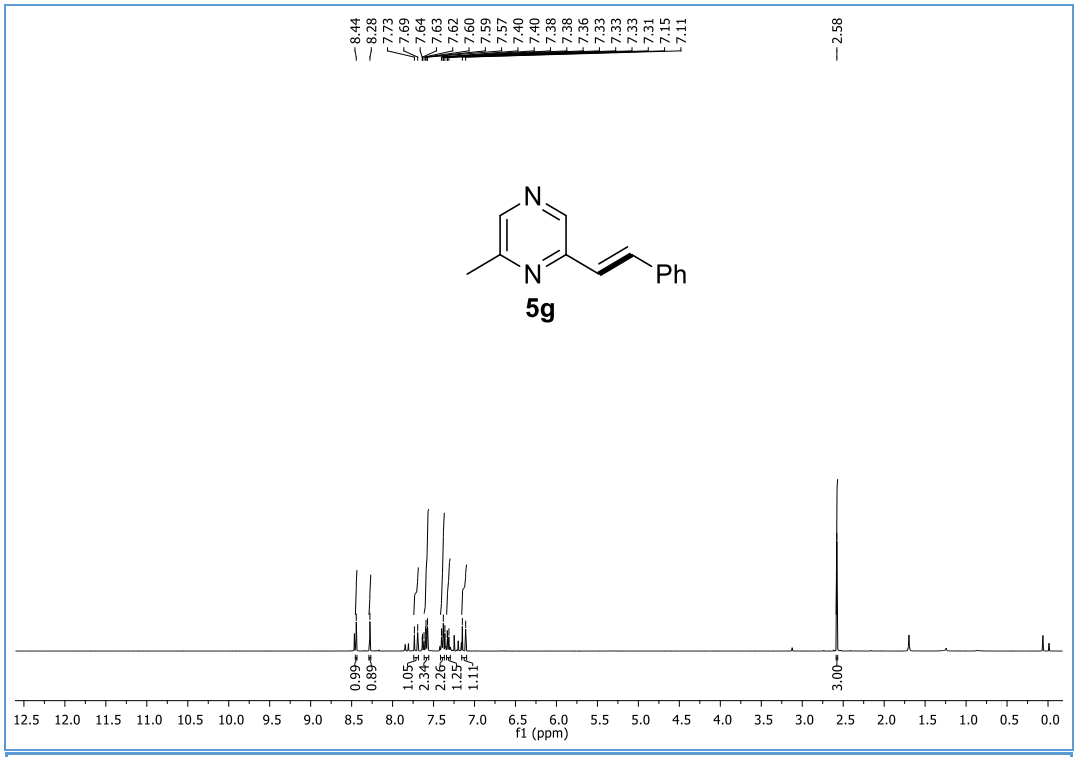

155.47
153.32
141.43
140.18
136.65
136.20
134.25
129.66
128.79
128.67
128.55
127.32
126.09
121.29
119.45
112.95
109.55
9.87
N
O
Ph
5d
200
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
f1 (ppm)

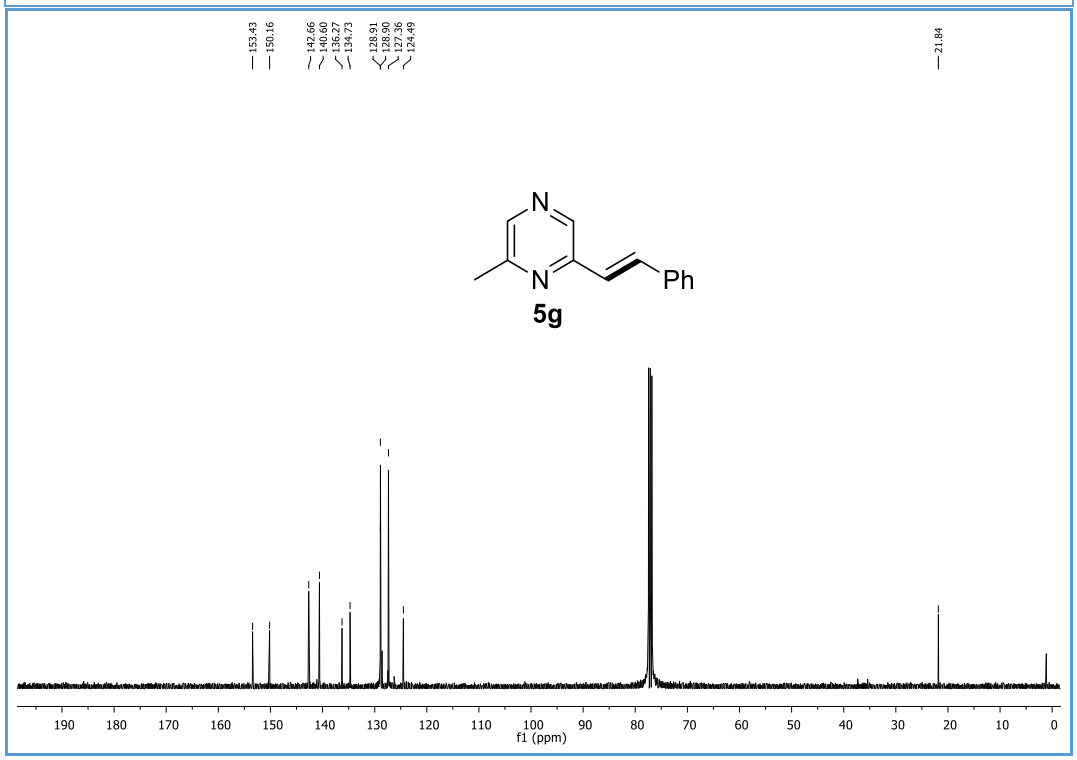

8.56
8.55
8.38
8.36
8.03
7.99
7.99
7.95
7.83
7.81
7.71
7.71
7.70
7.69
7.69
7.68
7.68
7.66
7.66
7.64
7.64
7.62
7.62
7.62
7.60
7.60
7.57
7.55
7.43
7.43
7.41
7.41
7.40
7.39
7.35
7.35
7.35
7.34
7.33
7.33
7.32
7.31
7.31
N
Ph
5e
1.00
1.09
2.23
1.04
4.12
1.05
2.06
1.04
10.0 9.5 9.0 8.5 8.0 7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 0.5 0.0
f1 (ppm)

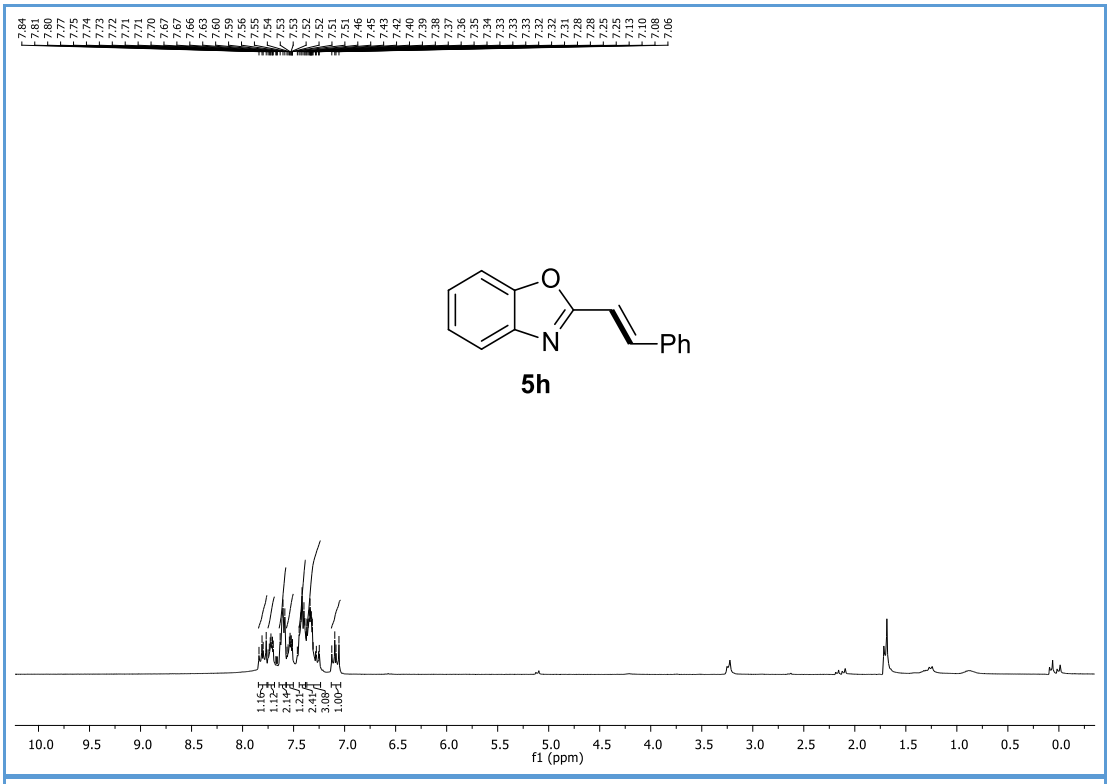

154.62
142.55
136.98
136.82
135.91
130.03
128.88
128.73
127.55
127.43
127.31
126.84
124.56
122.89
120.10
N
Ph
5e
200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0
f1 (ppm)

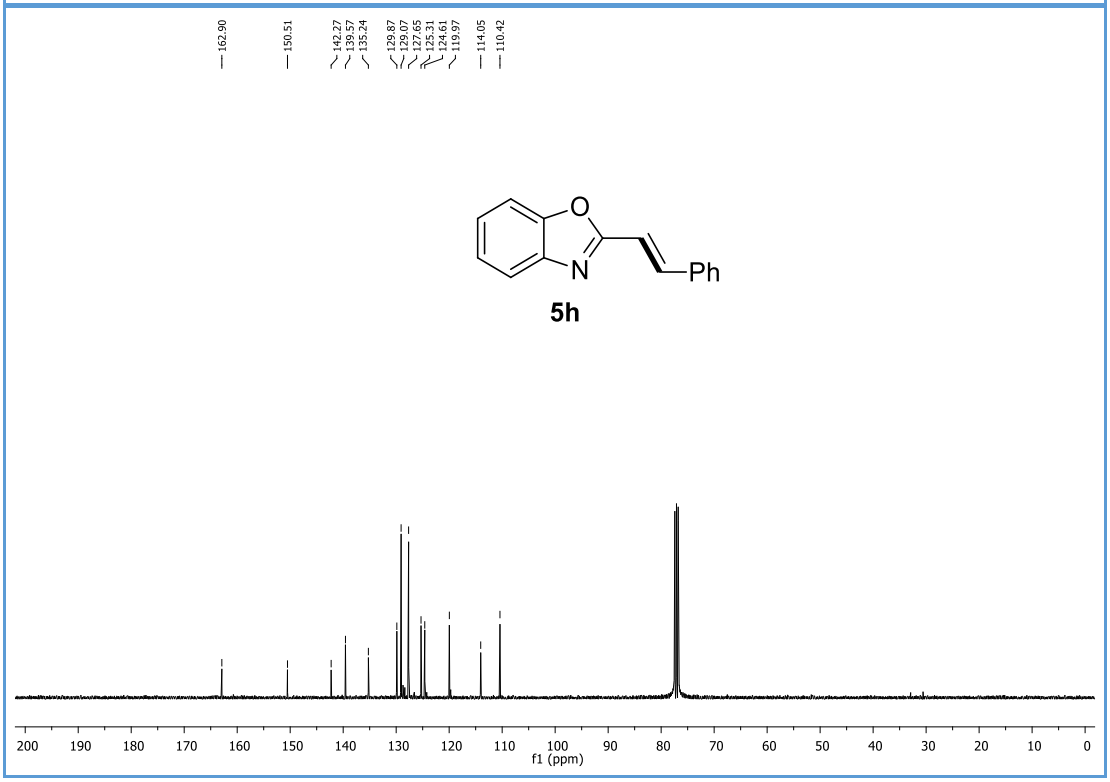

8.02
8.00
7.66
7.63
7.61
7.57
7.56
7.55
7.54
7.37
7.37
7.35
7.33
7.31
7.30
7.28
7.26
7.26
7.24
Cl
N
5f
2.00
1.03
1.19
3.21
3.19
1.97
11.0 10.5 10.0 9.5 9.0 8.5 8.0 7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 0.5 0.0
f1 (ppm)

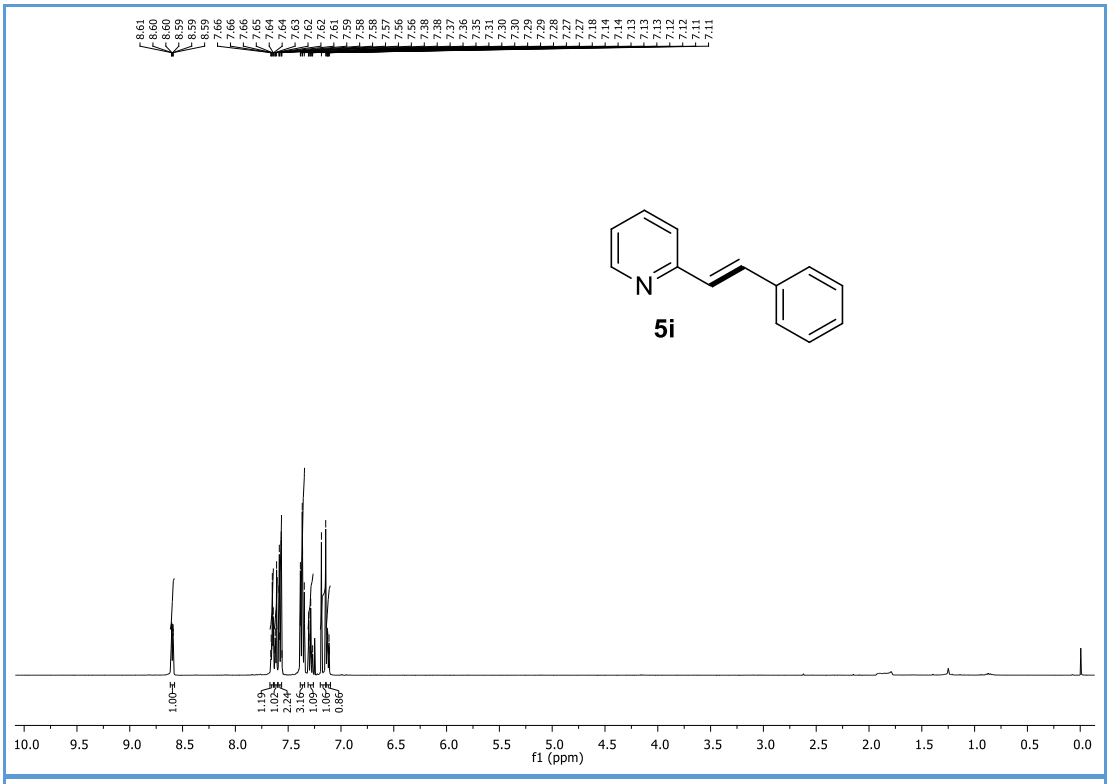

155.89
147.65
135.30
135.09
134.51
134.15
127.81
127.64
127.18
126.33
118.81
Cl
N
5f
200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0
f1 (ppm)

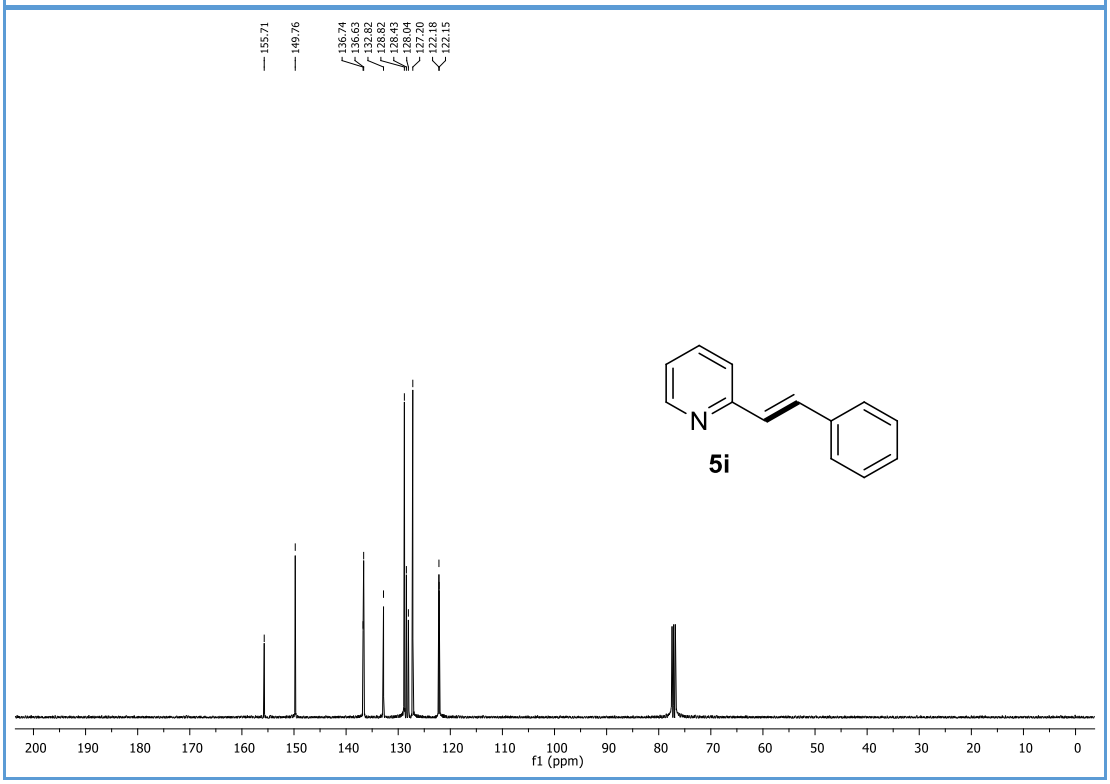

8.44
8.28
7.73
7.69
7.64
7.63
7.62
7.60
7.59
7.57
7.40
7.40
7.38
7.38
7.36
7.33
7.33
7.33
7.31
7.15
7.11
2.58
N
N
Ph
5g
0.99
0.89
1.05
2.34
2.26
1.25
1.11
3.00
12.5 12.0 11.5 11.0 10.5 10.0 9.5 9.0 8.5 8.0 7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 0.5 0.0
f1 (ppm)

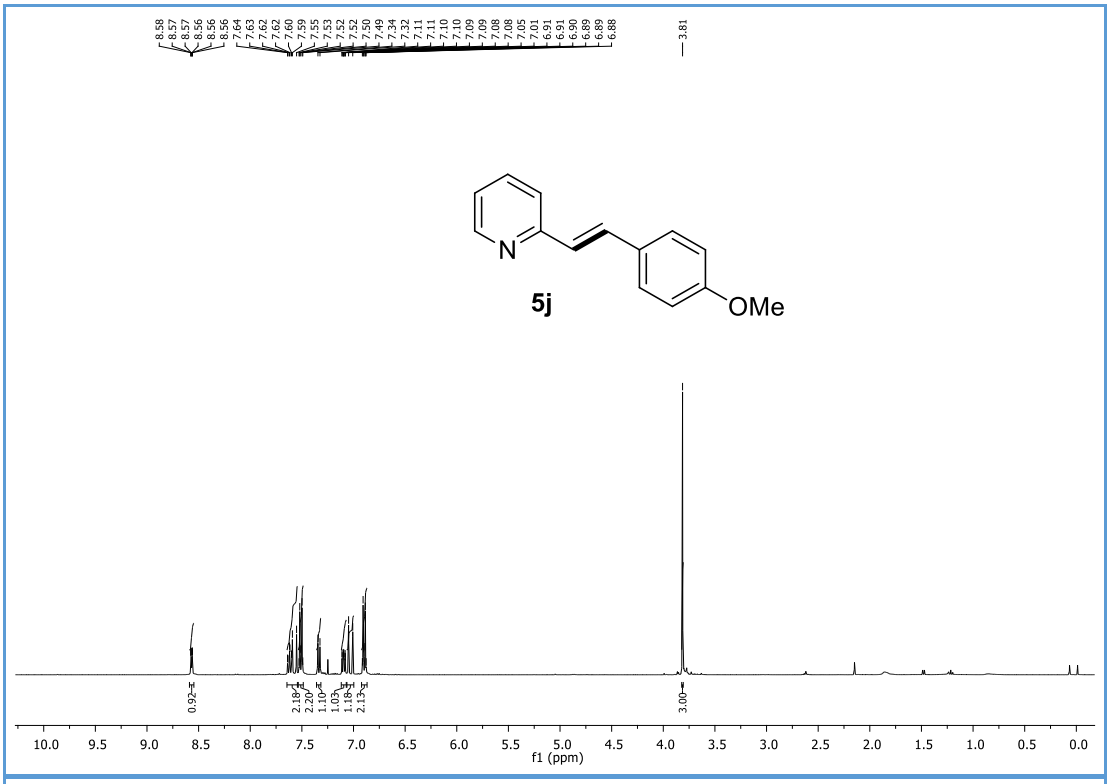

153.43
150.16
142.66
140.60
136.27
134.73
128.91
128.90
127.36
124.49
21.84
N
N
Ph
5g
190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0
f1 (ppm)

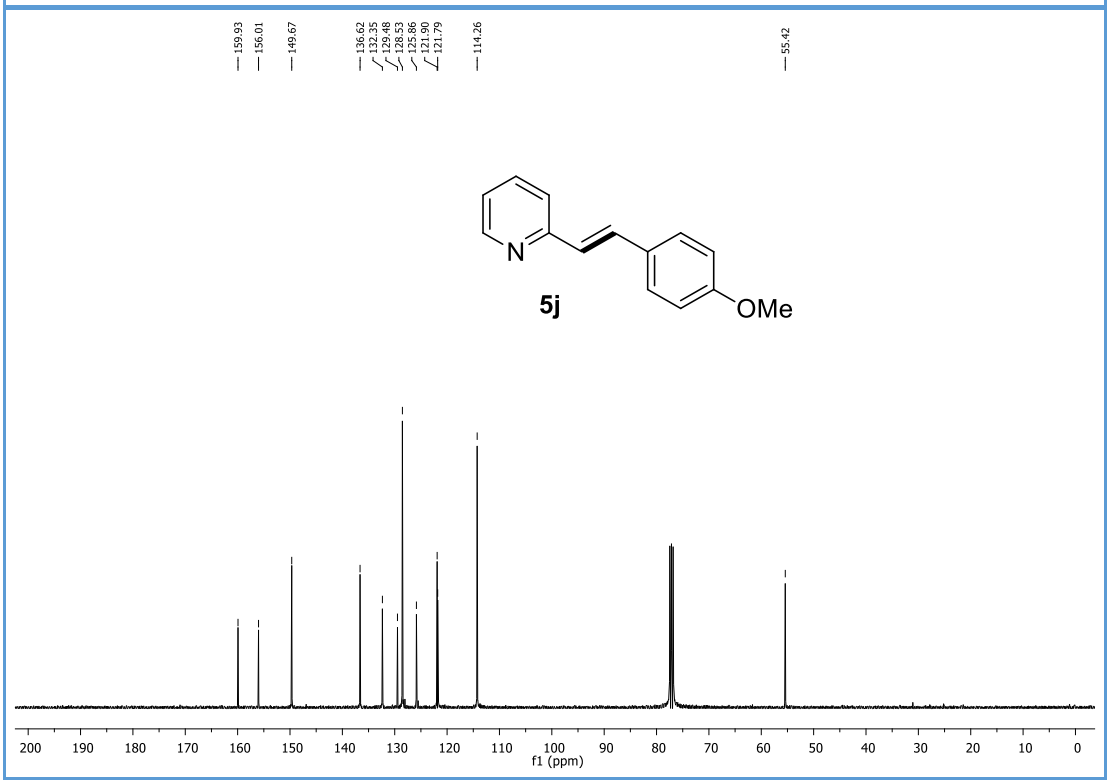

7.84 7.81 7.80 7.77 7.75 7.74 7.73 7.72 7.71 7.71 7.70 7.67 7.67 7.66 7.63 7.60 7.59 7.56 7.55 7.54 7.53 7.53 7.52 7.52 7.51 7.51 7.46 7.45 7.43 7.42 7.40 7.39 7.38 7.37 7.36 7.35 7.34 7.33 7.33 7.32 7.32 7.31 7.28 7.28 7.25 7.25 7.13 7.10 7.08 7.06
O
N
Ph
5h
1.16 1.12 2.14 1.21 2.41 3.08 1.00
10.0 9.5 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)

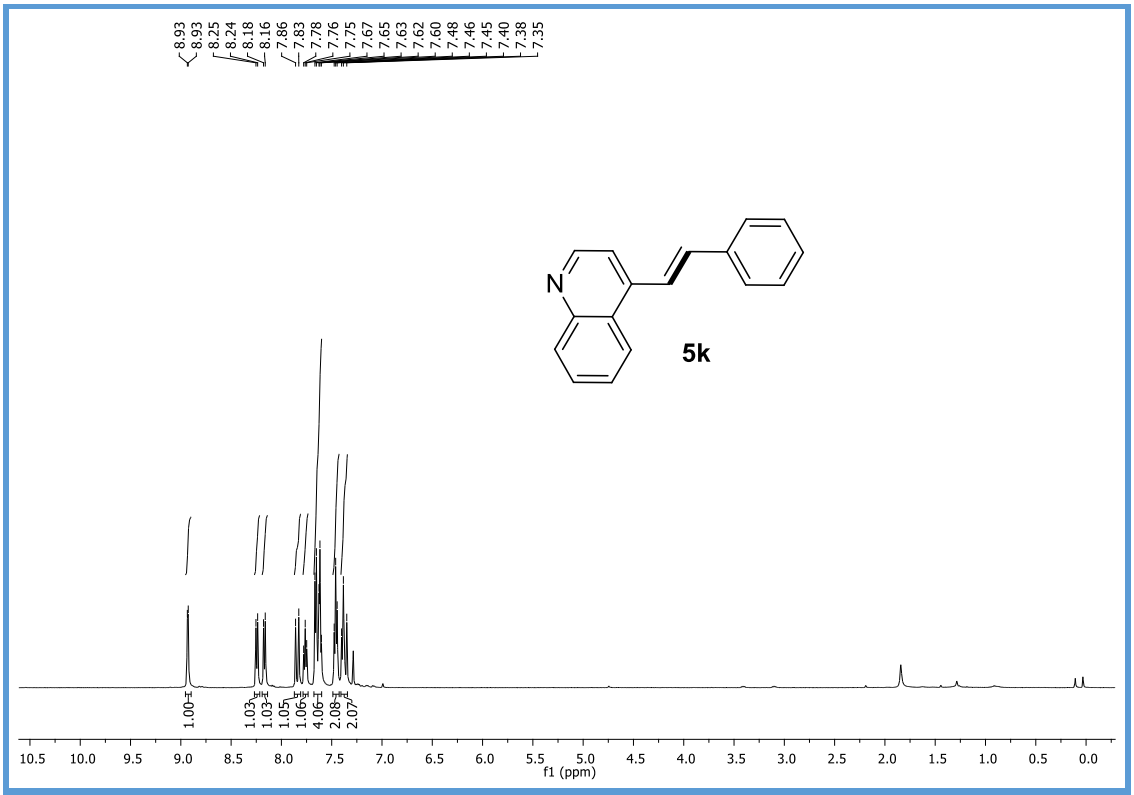

162.90 150.51 142.27 139.57 135.24 129.87 129.07 127.65 125.31 124.61 119.97 114.05 110.42
O
N
Ph
5h
200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0
f1 (ppm)

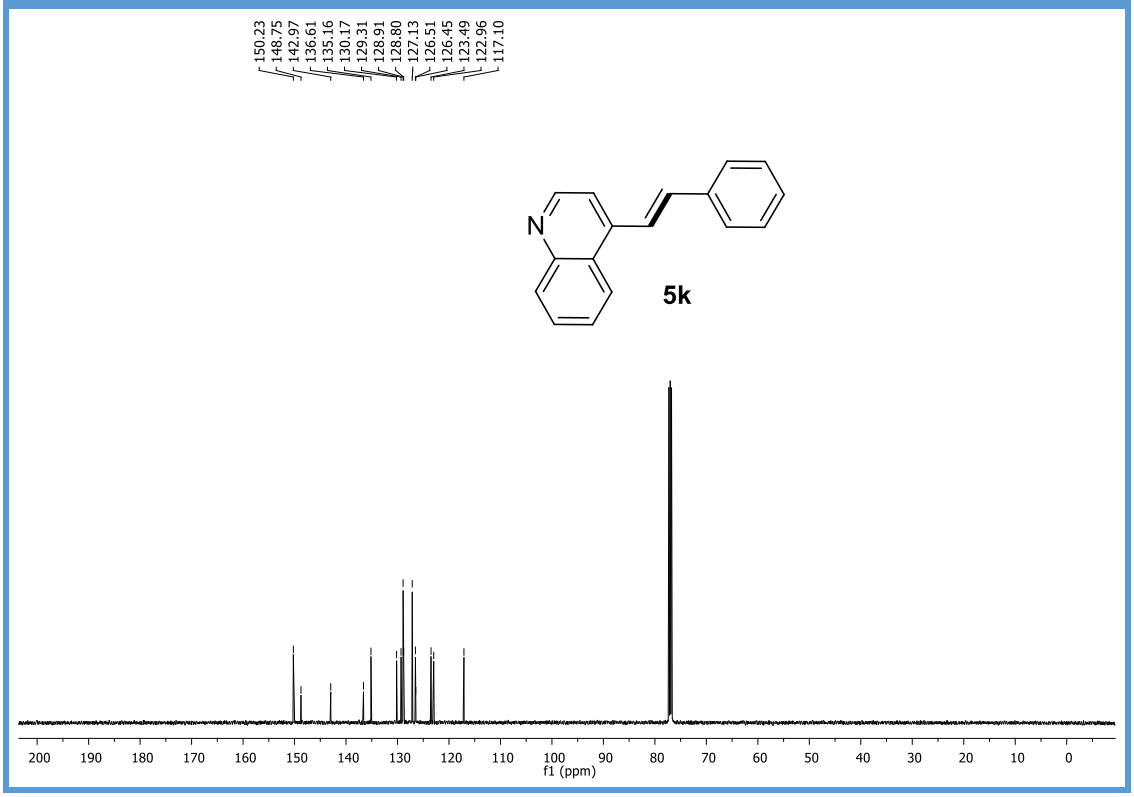

8.61
8.60
8.60
8.59
8.59
7.66
7.66
7.65
7.64
7.64
7.63
7.62
7.62
7.61
7.59
7.58
7.58
7.57
7.56
7.56
7.38
7.37
7.36
7.35
7.31
7.30
7.30
7.29
7.29
7.28
7.27
7.27
7.18
7.14
7.14
7.13
7.13
7.13
7.12
7.12
7.11
7.11
N
5i
1.00
1.19
1.02
2.24
3.16
1.09
1.06
0.86
10.0
9.5
9.0
8.5
8.0
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
0.5
0.0
f1 (ppm)

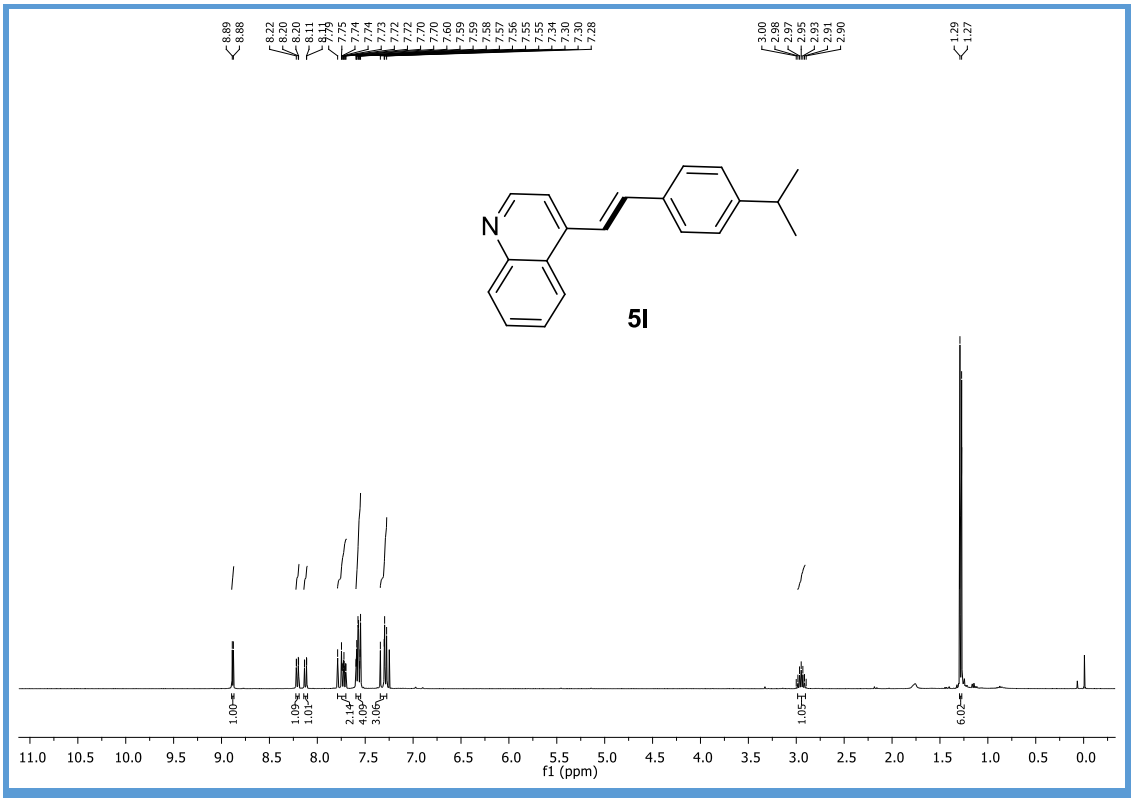

155.71
149.76
136.74
136.63
132.82
128.82
128.43
128.04
127.20
123.18
122.15
N
5i
200
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
f1 (ppm)

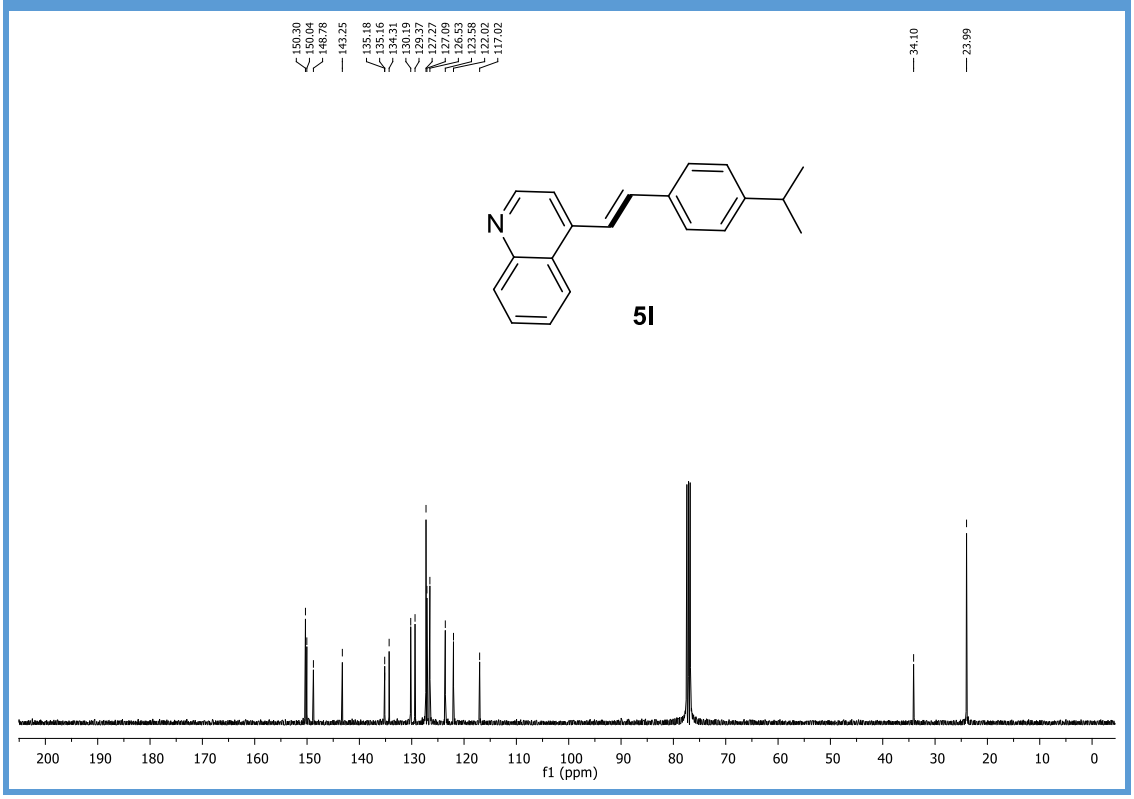

8.58
8.57
8.57
8.56
8.56
8.56
7.64
7.63
7.62
7.62
7.60
7.59
7.55
7.53
7.52
7.52
7.50
7.49
7.34
7.32
7.11
7.11
7.10
7.10
7.09
7.09
7.08
7.08
7.05
7.01
6.91
6.91
6.90
6.89
6.89
6.88
3.81
N
5j
OMe
0.92
2.18
2.20
1.10
1.05
1.18
2.13
3.00
10.0 9.5 9.0 8.5 8.0 7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 0.5 0.0
f1 (ppm)

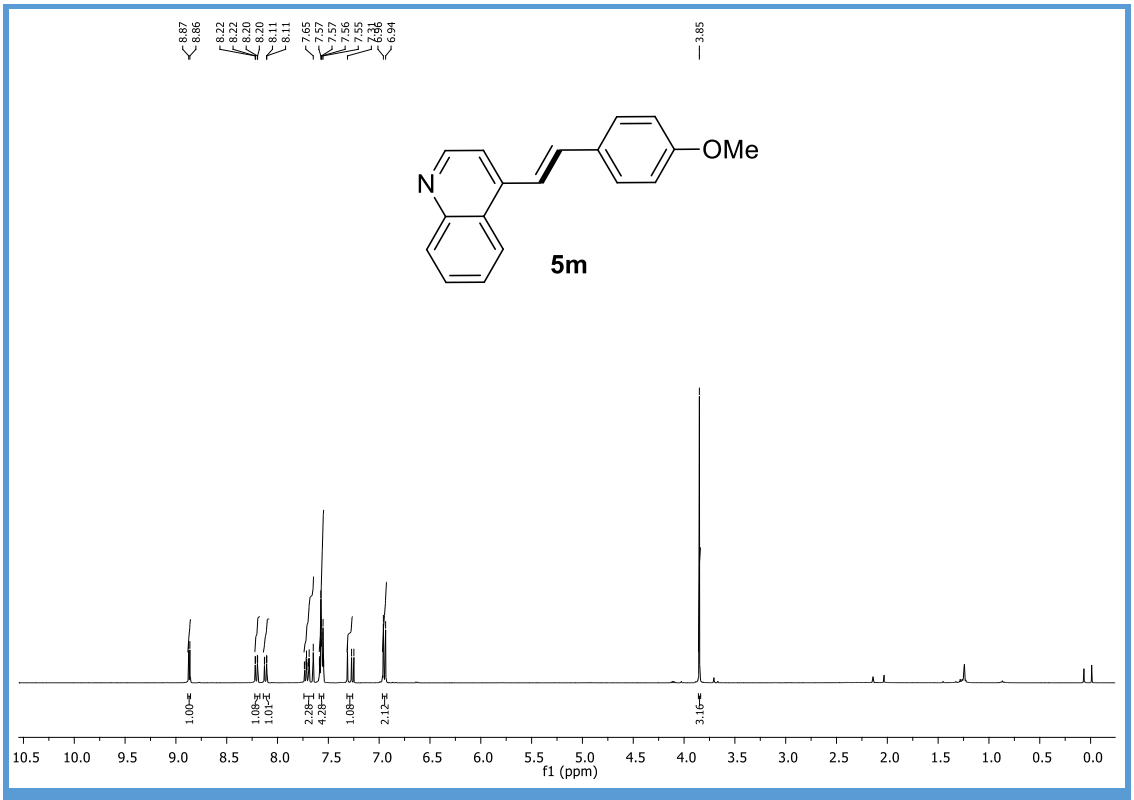

159.93
156.01
149.67
136.62
132.35
129.48
128.53
125.86
121.90
121.79
114.26
55.42
N
5j
OMe
200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0
f1 (ppm)

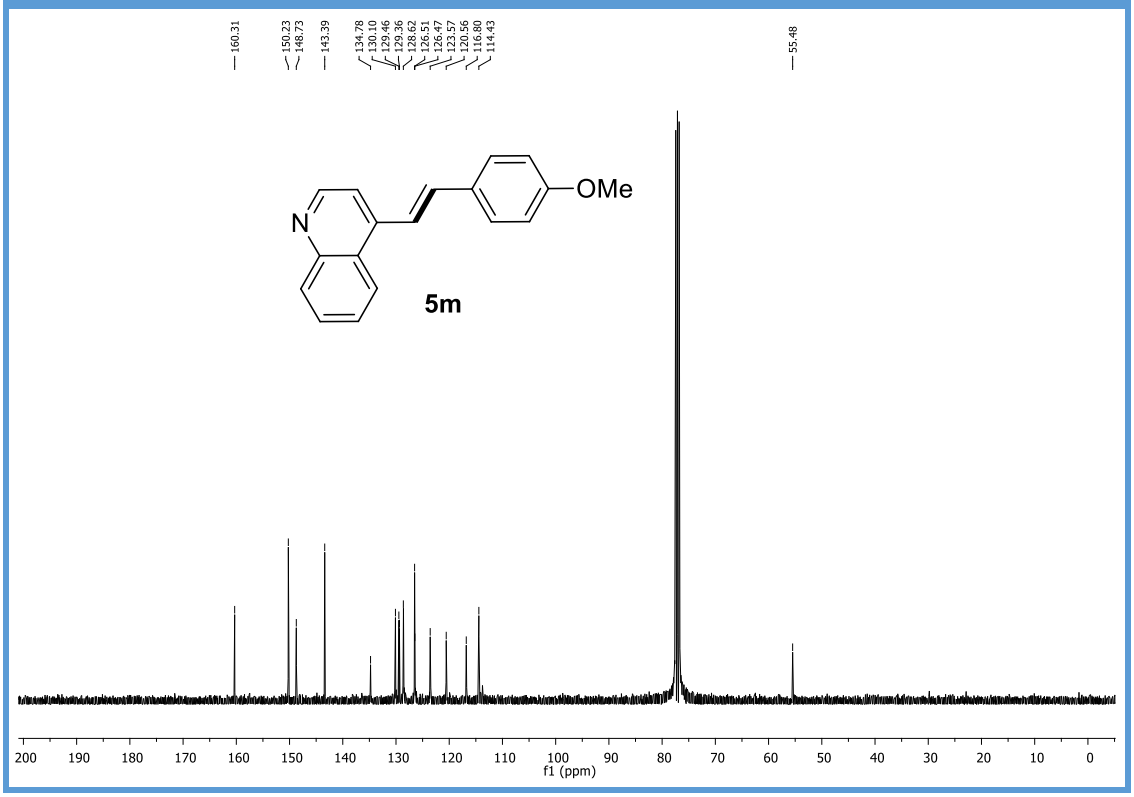

8.93
8.93
8.25
8.24
8.18
8.16
7.86
7.83
7.78
7.76
7.75
7.67
7.65
7.63
7.62
7.60
7.48
7.46
7.45
7.40
7.38
7.35
N
5k
1.00
1.03
1.03
1.05
1.06
4.06
2.09
2.07
10.5 10.0 9.5 9.0 8.5 8.0 7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 0.5 0.0
f1 (ppm)

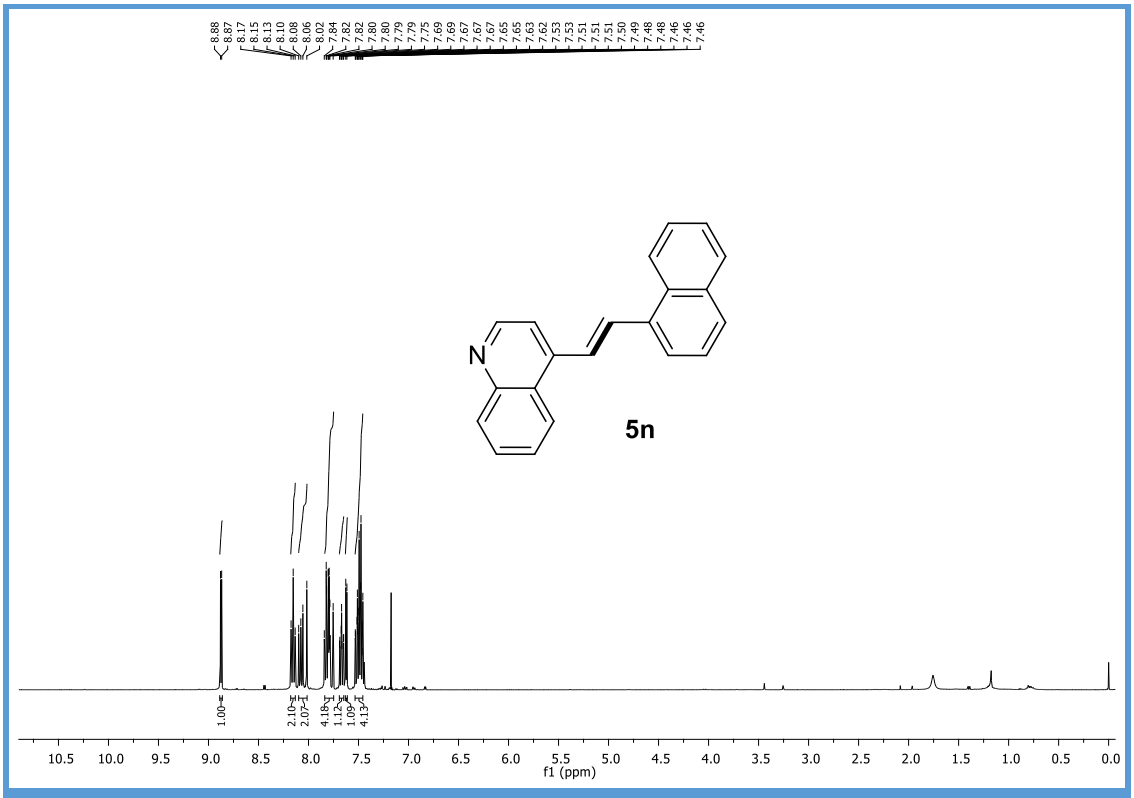

150.23
148.75
142.97
136.61
135.16
130.17
129.31
128.91
128.80
127.13
126.51
126.45
123.49
122.96
117.10
N
5k
200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0
f1 (ppm)

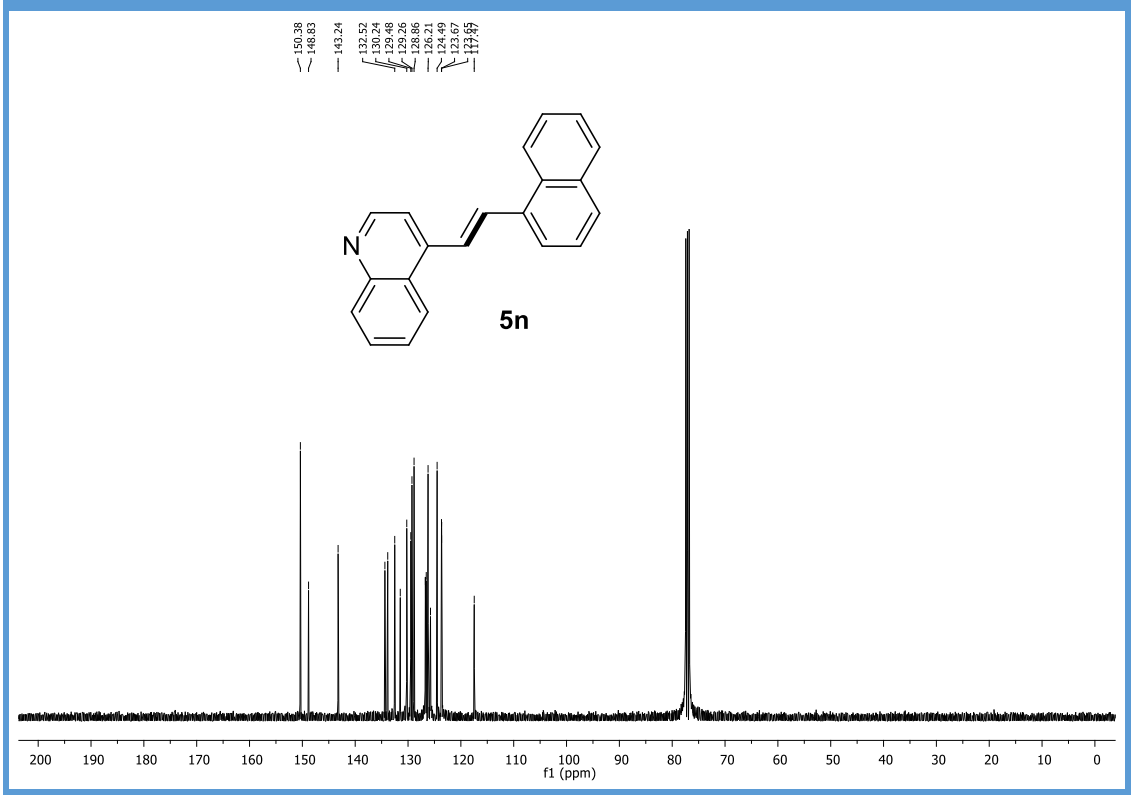

8.89
8.88
8.22
8.20
8.20
8.11
7.75
7.74
7.74
7.73
7.72
7.72
7.70
7.70
7.60
7.59
7.59
7.58
7.57
7.56
7.55
7.55
7.34
7.30
7.30
7.28
3.00
2.98
2.97
2.95
2.93
2.91
2.90
1.29
1.27
N
5l
1.00
1.09
1.01
2.14
4.09
3.06
1.05
6.02
11.0 10.5 10.0 9.5 9.0 8.5 8.0 7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 0.5 0.0
f1 (ppm)

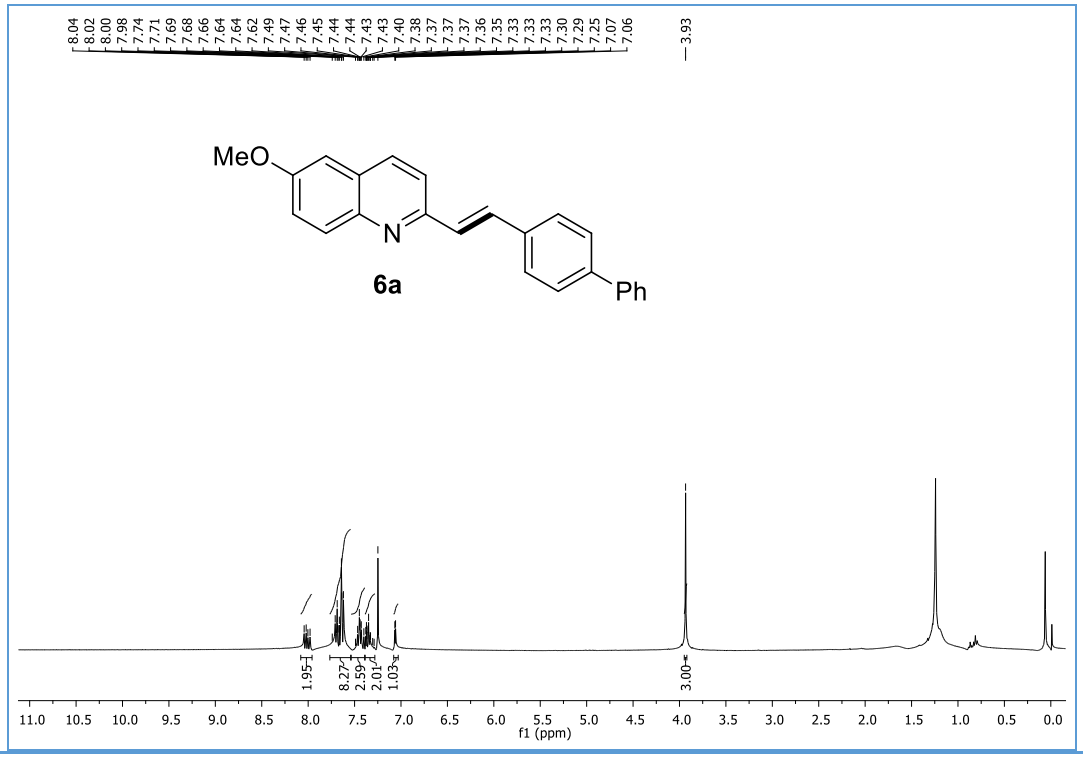

150.30
150.04
148.78
143.25
135.18
135.16
134.31
130.19
129.37
127.27
127.09
126.53
123.58
122.02
117.02
34.10
23.99
N
5l
200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0
f1 (ppm)

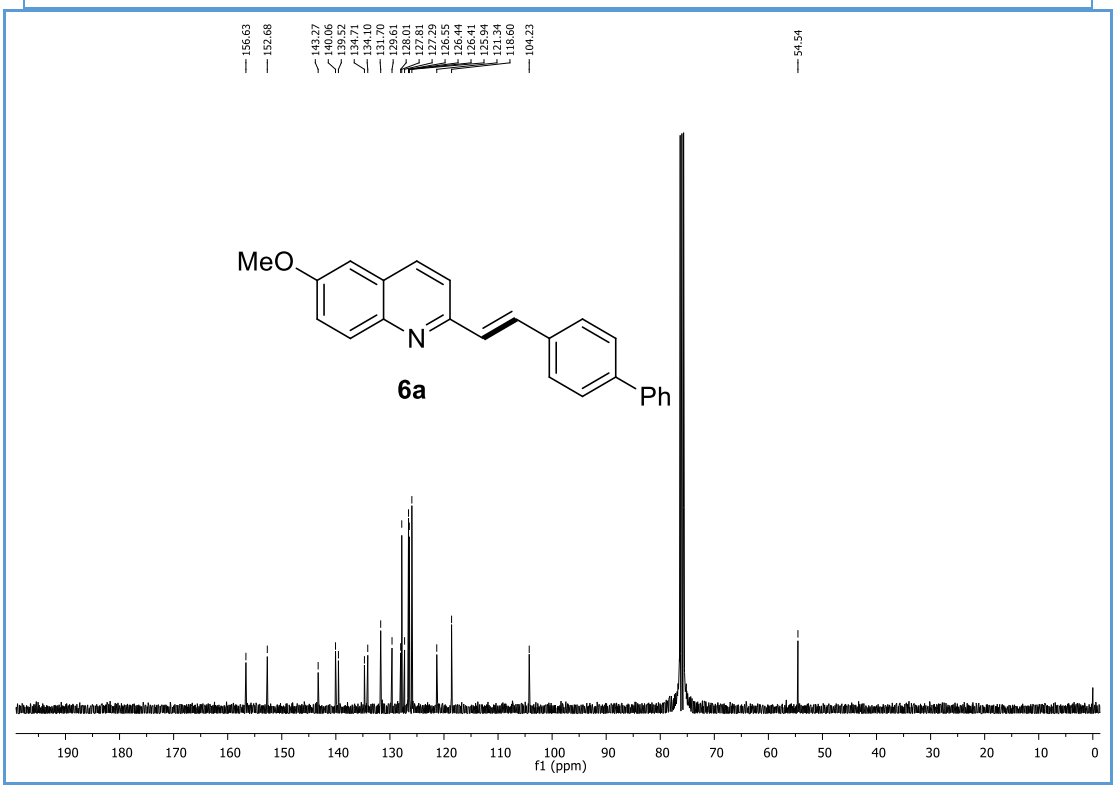

8.87
8.86
8.22
8.22
8.20
8.20
8.11
8.11
7.65
7.57
7.57
7.56
7.55
7.31
6.96
6.94
3.85
OMe
N
5m
1.00
1.08
1.01
2.28
4.26
1.08
2.12
3.16
10.5 10.0 9.5 9.0 8.5 8.0 7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 0.5 0.0
f1 (ppm)

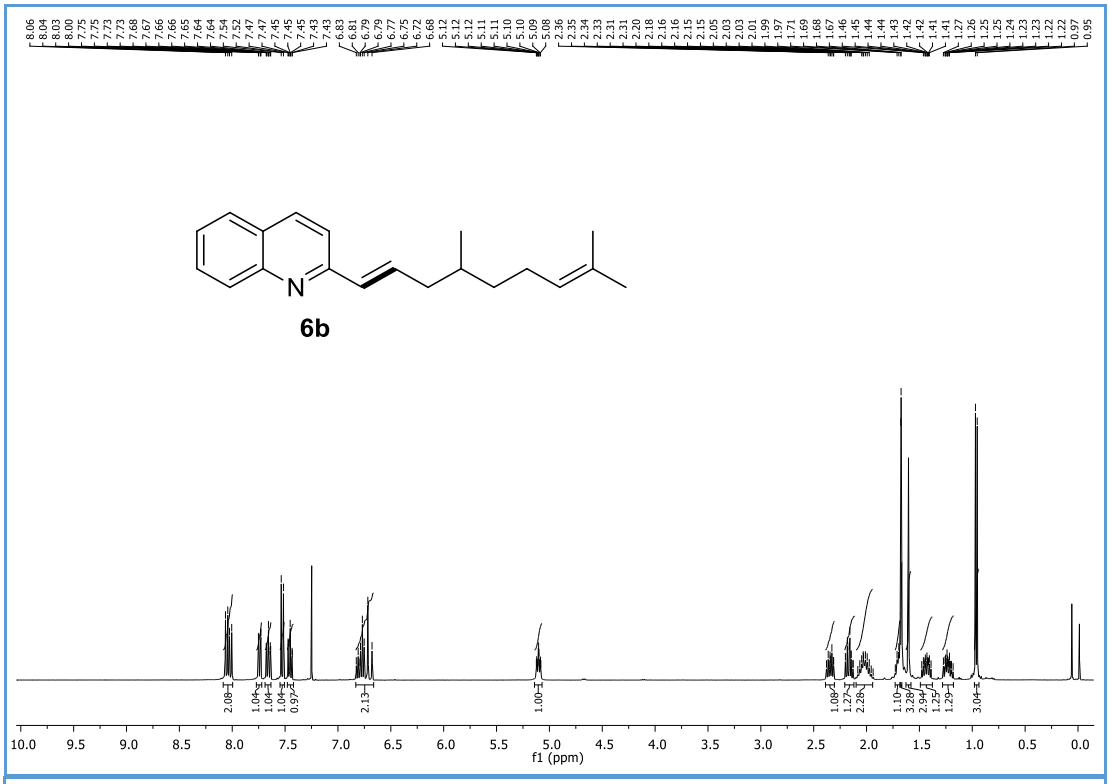

160.31
150.23
148.73
143.39
134.78
130.10
129.46
129.36
128.62
126.51
126.47
123.57
120.56
116.80
114.43
55.48
OMe
N
5m
200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0
f1 (ppm)

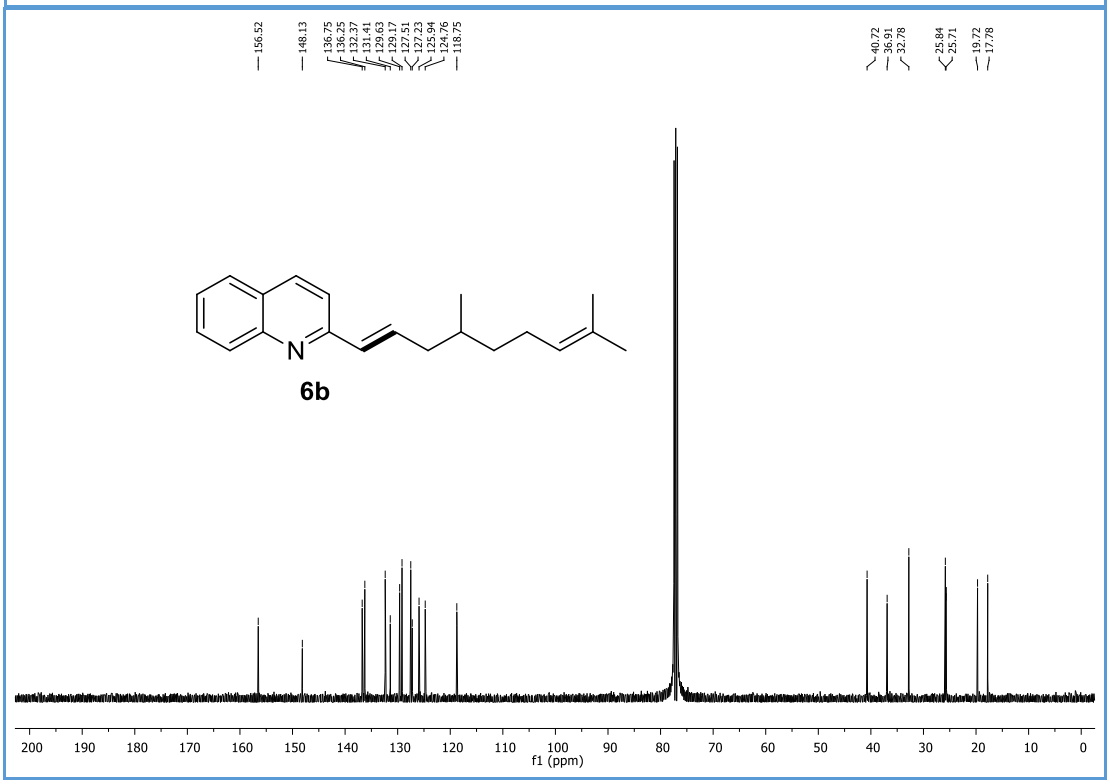

8.88
8.87
8.17
8.15
8.13
8.10
8.08
8.06
8.02
7.84
7.82
7.80
7.79
7.75
7.69
7.67
7.65
7.63
7.62
7.53
7.51
7.50
7.49
7.48
7.46
N
5n
1.00
2.10
2.07
4.18
1.12
1.09
4.13
10.5 10.0 9.5 9.0 8.5 8.0 7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 0.5 0.0
f1 (ppm)

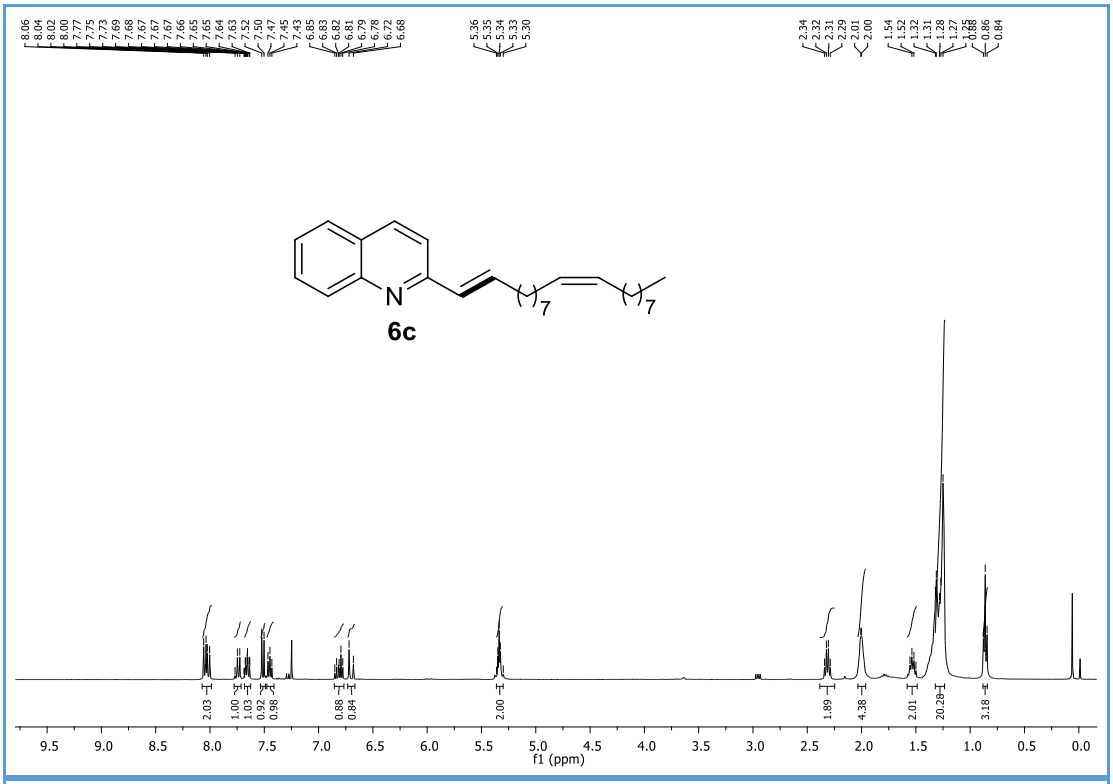

150.38
148.83
143.24
132.52
130.24
129.98
129.36
128.86
126.21
124.49
123.67
N
5n
200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0
f1 (ppm)

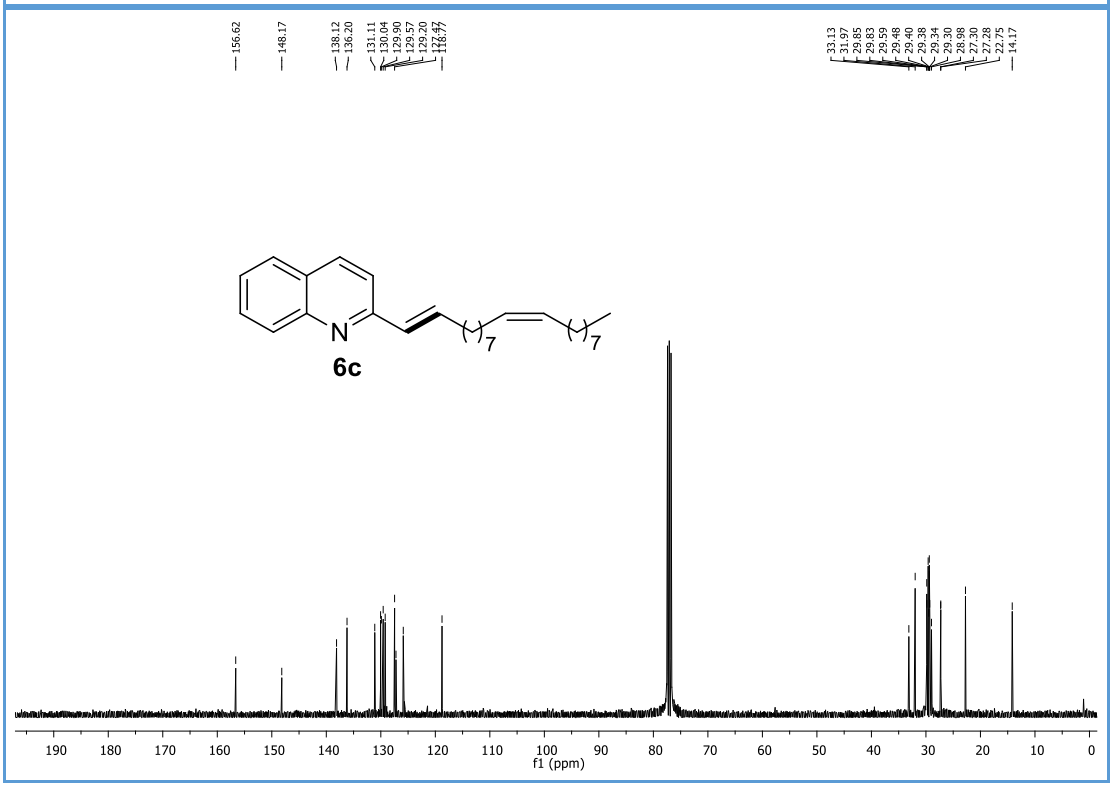

8.04
8.02
8.00
7.98
7.74
7.71
7.69
7.68
7.66
7.64
7.64
7.62
7.49
7.47
7.46
7.45
7.44
7.44
7.43
7.43
7.40
7.38
7.37
7.37
7.37
7.36
7.35
7.33
7.33
7.33
7.30
7.29
7.25
7.07
7.06
3.93
MeO
N
6a
Ph
1.95
8.27
2.59
2.01
1.03
3.00
11.0 10.5 10.0 9.5 9.0 8.5 8.0 7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 0.5 0.0
f1 (ppm)

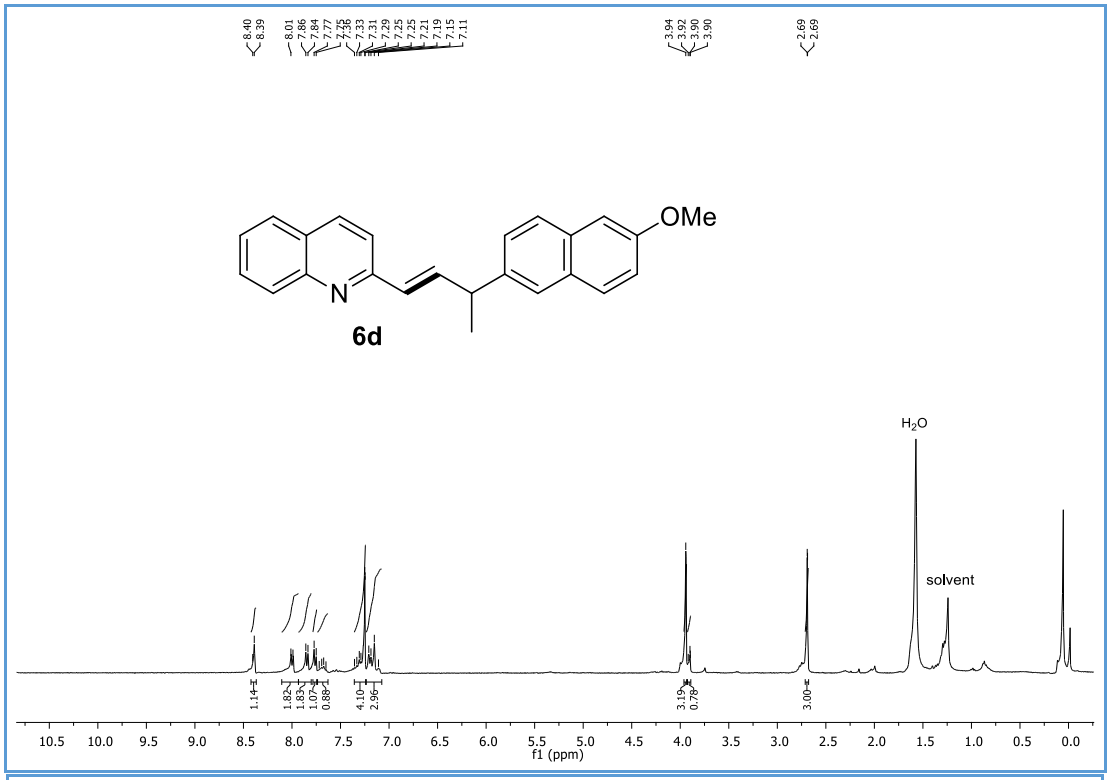

156.63
152.68
143.27
140.06
139.52
134.71
134.10
131.70
129.61
128.01
127.81
127.29
126.55
126.44
126.41
125.94
121.34
118.60
104.23
54.54
MeO
N
6a
Ph
190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0
f1 (ppm)

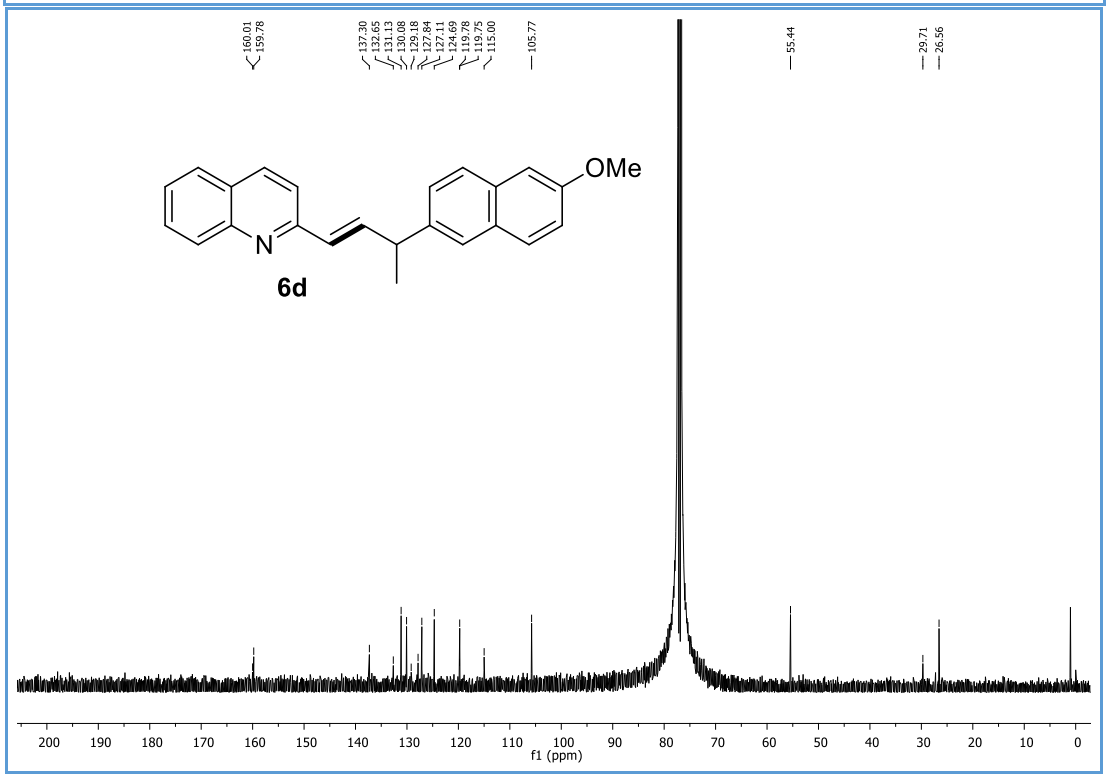

8.06 8.04 8.03 8.00 7.75 7.75 7.73 7.68 7.67 7.66 7.66 7.65 7.64 7.64 7.54 7.52 7.47 7.47 7.45 7.45 7.45 7.43 7.43 6.83 6.81 6.79 6.79 6.77 6.75 6.72 6.68 5.12 5.12 5.11 5.11 5.10 5.09 5.08 2.36 2.35 2.34 2.33 2.31 2.31 2.20 2.18 2.16 2.16 2.15 2.15 2.05 2.03 2.01 1.99 1.97 1.71 1.69 1.68 1.67 1.46 1.45 1.44 1.44 1.43 1.42 1.42 1.41 1.41 1.27 1.26 1.25 1.25 1.24 1.23 1.23 1.22 1.22 0.97 0.95
N
6b
2.08 1.04 1.04 1.04 0.97 2.13 1.00 1.08 1.27 2.28 1.10 3.28 2.64 1.29 3.04
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)

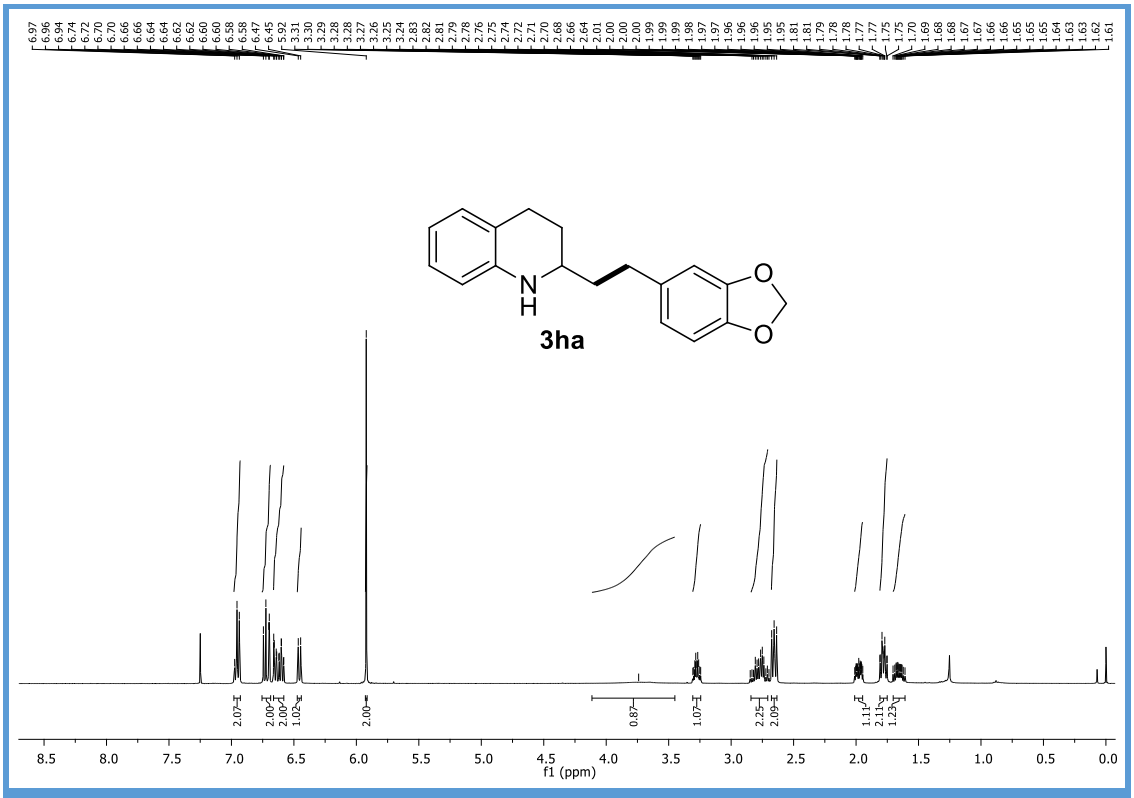

156.52 148.13 136.75 136.25 132.37 131.41 129.63 129.17 127.51 127.23 125.94 124.76 118.75
40.72 36.91 32.78 25.84 25.71 19.72 17.78
N
6b
200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0
f1 (ppm)

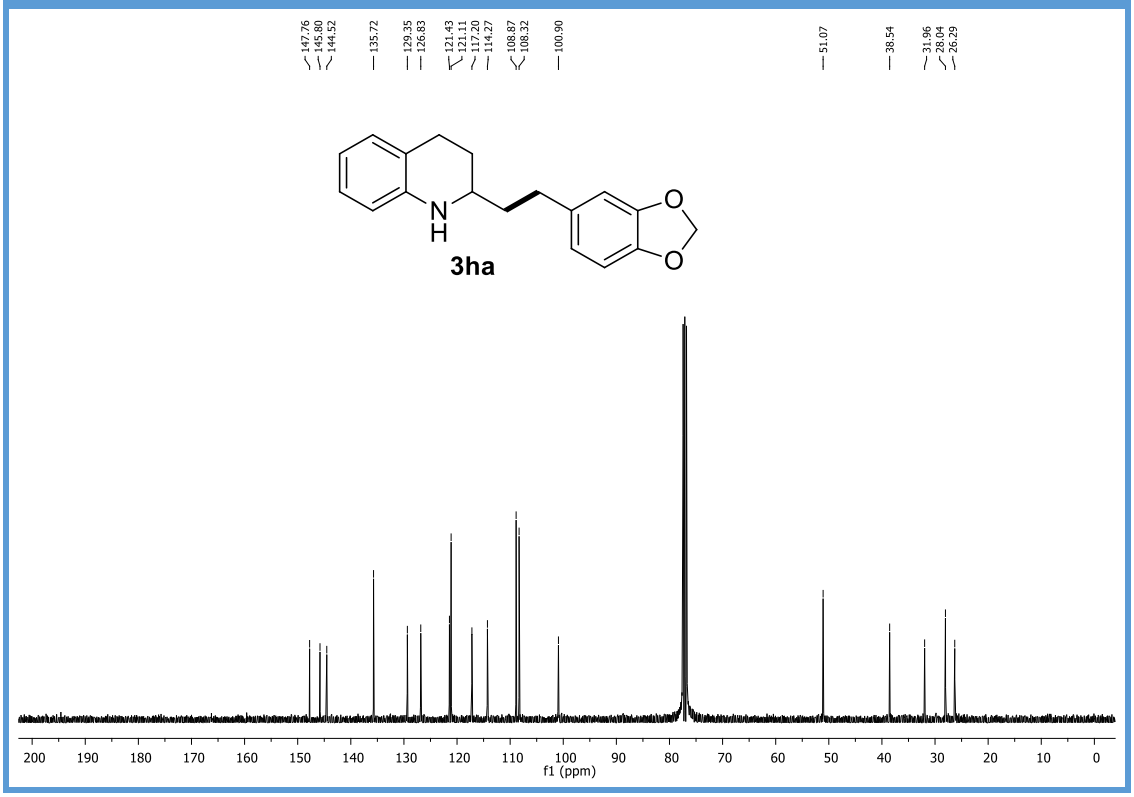

8.06
8.04
8.02
8.00
7.77
7.75
7.73
7.69
7.68
7.67
7.67
7.67
7.66
7.65
7.65
7.64
7.63
7.52
7.50
7.47
7.45
7.43
6.85
6.83
6.82
6.81
6.79
6.78
6.72
6.68
5.36
5.35
5.34
5.33
5.30
2.34
2.32
2.31
2.29
2.01
2.00
1.54
1.52
1.32
1.31
1.28
1.27
0.88
0.86
0.84
N
7
7
6c
2.03
1.00
1.03
0.92
0.98
0.88
0.84
2.00
1.89
4.38
2.01
20.28
3.18
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)

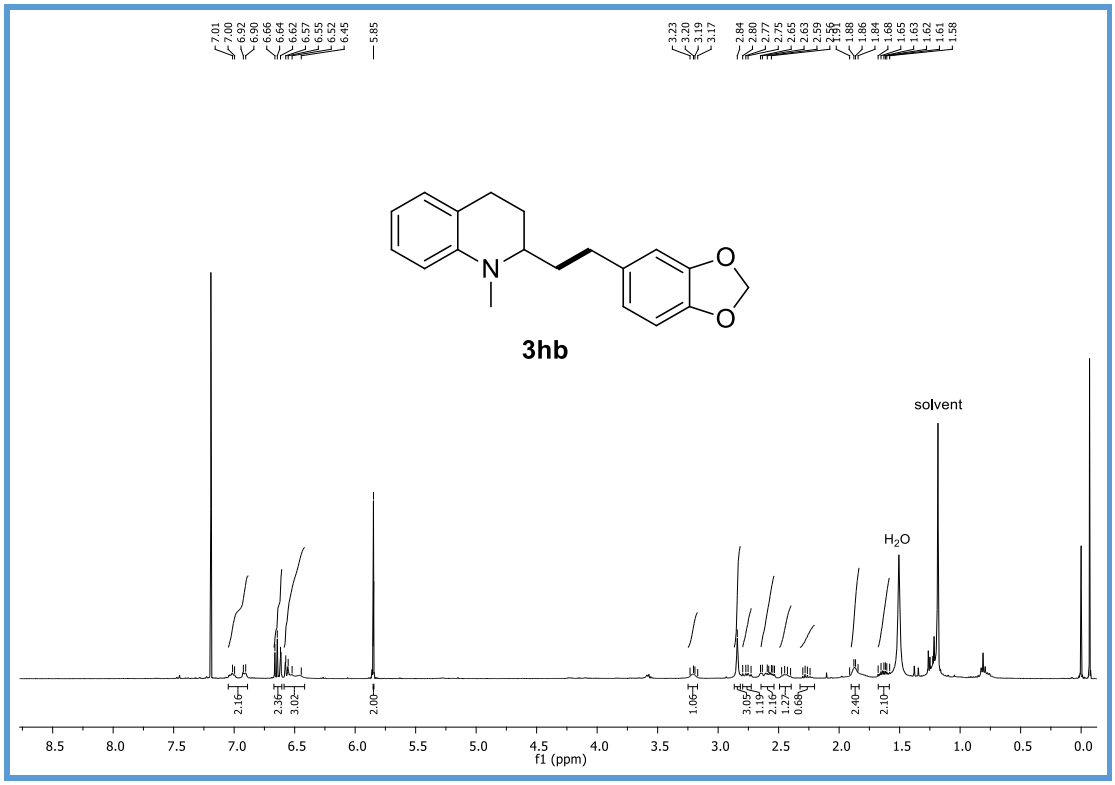

156.62
148.17
138.12
136.20
131.11
130.04
129.90
129.57
129.20
33.13
31.97
29.85
29.83
29.59
29.46
29.38
29.34
29.30
28.98
27.30
27.28
22.75
14.17
N
7
7
6c
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
f1 (ppm)


8.40
8.39
8.01
7.86
7.84
7.77
7.35
7.33
7.31
7.29
7.25
7.25
7.21
7.19
7.15
7.11
3.94
3.92
3.90
3.90
2.69
2.69
OMe
N
6d
H2O
solvent
1.14
1.82
1.83
1.07
0.88
4.10
2.96
3.19
0.78
3.00
10.5 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)


160.01
159.78
137.30
132.65
131.13
130.08
129.18
127.84
127.11
124.69
119.78
119.75
115.00
105.77
55.44
29.71
26.56
OMe
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)


3ha
f1 (ppm)


3ha
f1 (ppm)


7.01
7.00
6.92
6.90
6.66
6.64
6.62
6.57
6.55
6.52
6.45
5.85
3.23
3.20
3.19
3.17
2.84
2.80
2.77
2.75
2.65
2.63
2.59
1.88
1.86
1.84
1.68
1.65
1.63
1.62
1.61
1.58
N
O
O
3hb
solvent
H2O
2.16
2.36
3.02
2.00
1.06
3.05
1.19
2.15
1.25
0.68
2.40
2.10
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)