Supporting Information for

DNDMH Enabled C(sp^3)−H Nitration of Aryl Alkenes

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1. General Information

For Solvent and Reagent: Anhydrous tetrahydrofuran (THF) and other reagents were purchased from the market without further purification.

For Reaction Operation: Reactions were generally performed in round-bottom flask unless other specific illuminations, and monitored by thin-layer chromatography (TLC, 254 nm silica gel 60-F plates) with fluorescence upon 254 nm irradiation, potassium permanganate ($KMnO_4$) stain, and phosphomolybdic acid (PMA) stain. Flash chromatographies were applied for the purification of reaction products with silica gel 200-300 mesh.

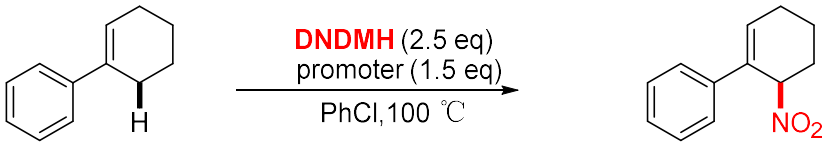
For NMR Spectroscopy: All NMR spectra were obtained at ambient temperature using Bruker AVANCE III-400MHz, JEOL-ZETA 400MHz or JEOL-ZETA 600MHz spectrometers. ^1H NMR and ^{13}C NMR spectra were recorded with $CDCl_3$ unless other specific illuminations. Spectra were referenced internally to the residual proton resonance of $CDCl_3$ (δ 7.26 ppm ^1H NMR, δ 77.16 ppm ^{13}C NMR) with tetramethylsilane (TMS, δ 0.00 ppm) as the standard. Chemical shifts (δ) were reported as part per million (ppm) in δ scale downfield from TMS. ^1H NMR data were recorded as follows: multiplicity (s = singlet, d = doublet, t = triplet, m = multiplet, septet or unresolved), coupling constant (Hz), integration. ^{13}C NMR spectra were recorded with complete ^1H decoupling.

For Mass Spectrometry: High resolution mass spectrometry (HRMS) data were obtained on a MicrOTOF-Q II (hybrid quadrupolar/time-of-flight) API US system by electrospray ionization (ESI) in the positive or negative ion mode from Bruker Corporation.

For IR Spectroscopy: Infrared spectra were recorded on an INVENIO spectrometer from Bruker Corporation as thin films. Absorptions are given in wavenumbers (cm^{-1}).

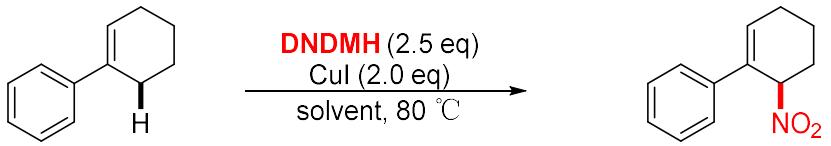
2. Explorations on Optimization of C(sp^3)-H Nitration of 1a with DNDMH

Table S1: Optimization of reductant using PhCl as the solvent.



Entry	promoter	Yield/%
1	CuCl	trace
2	$Cu(OTf)_2$	decompose
3	Ag_2O	No reaction
4	$NiCl_2$	No reaction
5	$ZnCl_2$	decompose
6	$CoCl_2$	No reaction
7	CuI	54
8	CuBr	31
9	$(Cu(CH_3CN)_4)PF_6$	16
10	CuTC	No reaction

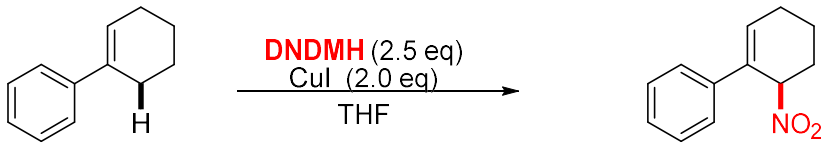
Table S2: Optimization of solvent using CuI as the reductant.



Entry	solvent	Yield/%
1	DCE	47
2	PhMe	40
3	PhCl	47
4	*t*-BuOH	50
5	MeCN	10
6	DMF	No reaction
7	MeOH	No reaction
8	EtOAc	No reaction
9	CCl_4	20
10	DMSO	No reaction
11	m-xylene	5
12	1,4-Dioxane	40
13	DME	69
14	DCM	20
15	Acetone	No reaction

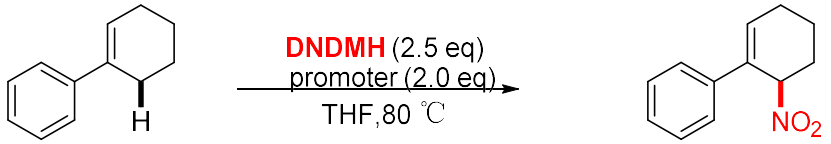
16	THF	91
17	$MeNO_2$	No reaction
18	MTBE	3

Table S3: Optimization of the reaction temperature.



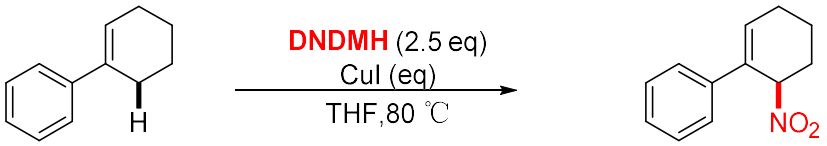
Entry	T/℃	Yield/%
1	60	Trace
2	70	46
3	80	91
4	90	78
5	100	54

Table S4: Optimization of reductants using THF as the solvent.



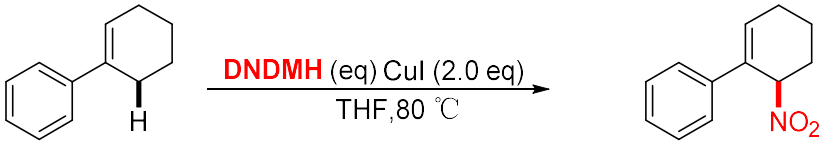
Entry	promoter	Yield/%
1	CuCl	No reaction
2	$Cu(OTf)_2$	decompose
3	Ag_2O	No reaction
4	$NiCl_2$	No reaction
5	$ZnCl_2$	decompose
6	$CoCl_2$	No reaction
7	CuI	91
8	CuBr	10
9	$(Cu(CH_3CN)4)PF_6$	trace
10	$(CF_3SO_3Cu)_2$·PhMe	25
11	CuTC	No reaction
12	$[Cu(MeCN)_4](CF_3SO_3)$	10
13	$Cu(BH_4)[P(C_6H_5)_3]_2$	No reaction
14	$[Cu(MeCN)_4]BF_4$	No reaction
15	CuBr·DMS	Trace
16	CuI(TMPhen)	No reaction

Table S5: Optimization of the loading of CuCl.



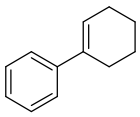
Entry	CuI	Yield/%
1	0.1 eq	No reaction
2	0.5 eq	52
3	1.0 eq	66
4	1.5 eq	77
5	2.0 eq	91
6	2.5 eq	70

Table S6: Optimization of the loading of DNDMH.

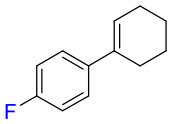


Entry	DNDMH	Yield/%
1	1.1 eq	50
2	1.5 eq	71
3	2.0 eq	75
4	2.5 eq	91
5	3.5 eq	52

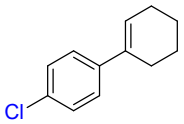
3. Synthesis and Characterization of Substrates



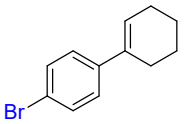
1a



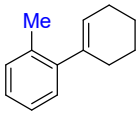
1b



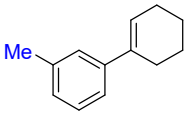
1c



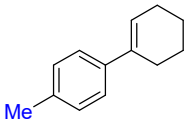
1d



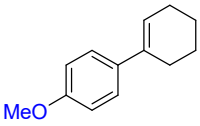
1e



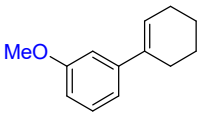
1f



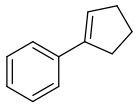
1g



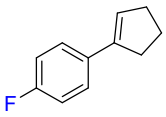
1h



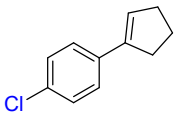
1i



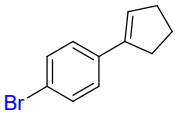
1j



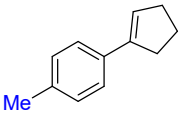
1k



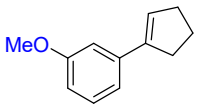
1l



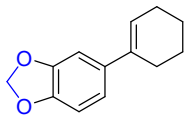
1m



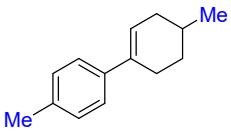
1n



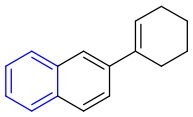
1o



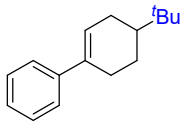
1p



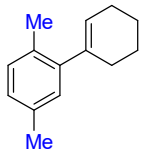
1q



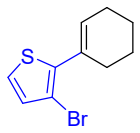
1r



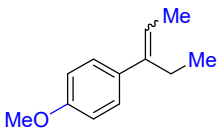
1s



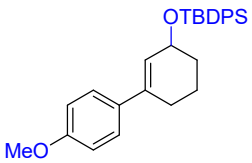
1t



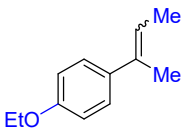
1u



1v



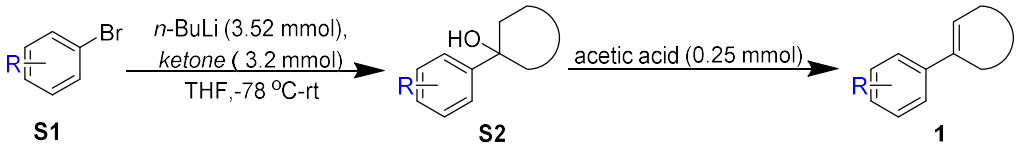
1w



1x

3.1. Substrate **1a** is commercially available.

3.2. Substrates **1b, 1c, 1d, 1e, 1f, 1g, 1h, 1i, 1j, 1k, 1l, 1m, 1n, 1o, 1p, 1q, 1r, 1s, 1t,** and **1u** are known compounds and prepared according to the general procedure as below.

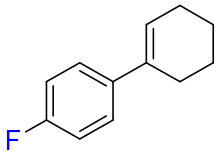


Arylbromide (**S1**, 3.20 mmol) was added into a 50 mL flame dried round bottom flask containing a magnetic stir bar. The bromide was then dissolved in tetrahydrofuran. The reaction mixture was purged with nitrogen and cooled to –78 °C then *n*-BuLi (3.52 mmol, 1.4mL) was added slowly into the solution. The reaction mixture was stirred at -78 °C for 30 minutes and the related ketone (3.20 mmol) was added. The reaction was allowed to warm up to room temperature and the consumption of starting material was monitored by TLC. When the reaction was completed, the reaction mixture was warmed to 0 °C and quenched by slow addition of 10 mL ice-cold ammonium chloride solution. The aqueous layer was extracted with ethyl acetate. The combined organic layers were washed with brine, dried over anhydrous Na_2SO_4, concentrated, and the crude extracts were purified by silica gel column chromatography to obtain the pure alcohol **S2**.

Alcohol (**S2**, 1.0 mmol) was added into a 50 mL round bottom flask charged with a magnetic stir bar and dissolved in acetic acid (0.25 mmol). The reaction mixture was stirred at 110 °C for 1 h. The reaction progress was monitored by TLC. When the starting material was fully consumed, the reaction was cooled to room temperature and quenched by addition of 1M NaOH until neutral pH. Organic phase was separated. And the aqueous phase was extracted three times with ethyl acetate. The combined organic layers were washed with brine, dried over anhydrous Na_2SO_4, and concentrated under reduced pressure. The crude extracts were purified by silica gel column chromatography to obtain the aryl alkenes **1**.

Characteristic Data of Synthesized 1:

4'-fluoro-2,3,4,5-tetrahydro-1,1'-biphenyl (1b)

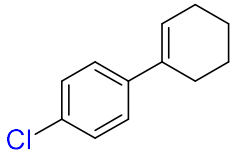


^{1}H NMR (400 MHz, $CDCl_3$): δ 7.37 – 7.30 (m, 2H), 7.03 – 6.94 (m, 2H),6.07 – 6.05 (m, 1H), 2.39 – 2.36 (m, 2H), 2.21 – 2.19 (m, 2H), 1.82 – 1.74 (m, 2H), 1.70 – 1.62 (m, 2H) ppm.

^{13}C NMR (100 MHz, $CDCl_3$): δ161.90(d, J= 244 Hz), 138.89, 135.78, 126.51(d, J= 8 Hz), 124.77, 115.01(d, J= 21 Hz), 27.67, 25.95, 23.15, 22.21 ppm.

1b is a known compound.[S1]

4'-chloro-2,3,4,5-tetrahydro-1,1'-biphenyl (1c)

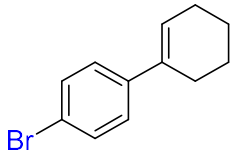


^{1}H NMR (400 MHz, $CDCl_3$): δ 7.32 – 7.25 (m, 4H), 6.15 – 6.13(m, 1H), 2.41 – 2.37 (m, 2H), 2.24 – 2.22 (m, 2H), 1.81 – 1.74 (m, 2H), 1.69 – 1.62 (m, 2H) ppm.

^{13}C NMR (100 MHz, $CDCl_3$): δ 141.21, 135.67, 132.25, 128.37, 126.32, 125.49, 27.43, 25.98, 23.08, 22.15 ppm.

1c is a known compound. [S2]

4'-bromo-2,3,4,5-tetrahydro-1,1'-biphenyl (1d)

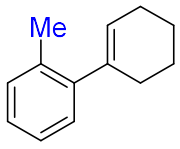


^{1}H NMR (400 MHz, $CDCl_3$): δ 7.51 – 7.40 (m, 2H), 7.35 – 7.22 (m, 2H), 6.16 – 6.14 (m, 1H), 2.42 – 2.38 (m, 2H), 2.25 – 2.22 (m, 2H), 1.83 – 1.80 (m, 2H), 1.74 – 1.65 (m, 2H) ppm.

^{13}C NMR (100 MHz, $CDCl_3$): δ 141.63, 135.68, 131.30, 126.68, 125.56, 120.37, 27.35, 25.98, 23.05, 22.13 ppm.

1d is a known compound. [S3]

2'-methyl-2,3,4,5-tetrahydro-1,1'-biphenyl (1e)

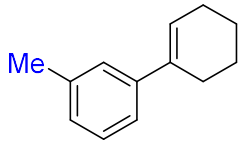


^{1}H NMR (400 MHz, $CDCl_3$): δ 7.19 – 7.12 (m, 3H), 7.10 – 7.06 (m, 1H), 5.57 – 5.55 (m, 1H), 2.29 (s, 3H), 2.21 – 2.16 (m, 4H), 1.78 – 1.75 (m, 2H), 1.71 – 1.69 (m, 2H) ppm.

^{13}C NMR (100 MHz, $CDCl_3$): δ 144.80, 138.98, 135.14, 130.08, 128.42, 126.51, 125.77, 125.60, 30.23, 25.53, 23.25, 22.35, 19.91 ppm.

1e is a known compound. [S3]

3'-methyl-2,3,4,5-tetrahydro-1,1'-biphenyl (1f)

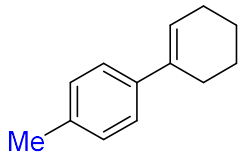


^{1}H NMR (400 MHz, $CDCl_3$): δ 7.23 – 7.16 (m, 3H), 7.07 – 7.02 (m, 1H), 6.11 – 6.09 (m, 1H), 2.44 – 2.38 (m, 2H), 2.35 (s, 3H), 2.21 – 2.19(m, 2H), 1.83 – 1.75 (m, 2H), 1.71 – 1.62 (m, 2H) ppm.

^{13}C NMR (100 MHz, $CDCl_3$): δ 142.90, 137.77, 136.83, 128.22, 127.40, 125.93, 124.75, 122.22, 27.62, 26.01, 23.23, 22.33, 21.68 ppm.

1f is a known compound. [S3]

4'-methyl-2,3,4,5-tetrahydro-1,1'-biphenyl (1g)

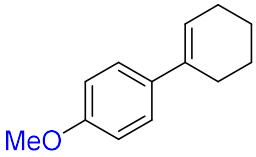


^{1}H NMR (400 MHz, $CDCl_3$): δ 7.30 – 7.26 (m, 2H), 7.11 (dd, *J* = 7.7, 1.1 Hz, 2H), 6.09 – 6.07 (m, 1H), 2.40 – 2.38 (m, 2H), 2.33 (s, 3H), 2.21 – 2.19 (m, 2H), 1.79 – 1.76 (m, 2H), 1.67 – 1.64 (m, 2H) ppm.

^{13}C NMR (100 MHz, $CDCl_3$): δ 140.00, 136.52, 136.24, 129.01, 124.94, 124.07, 27.56, 25.99, 23.24, 22.35, 21.17 ppm.

1g is a known compound.[S1]

4'-methoxy-2,3,4,5-tetrahydro-1,1'-biphenyl (1h)

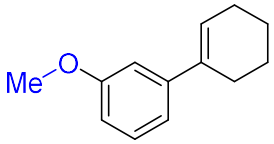


^{1}H NMR (400 MHz, $CDCl_3$): δ 7.35 – 7.30 (m, 2H), 6.90 – 6.81 (m, 2H), 6.05 – 6.02 (m, 1H), 3.81 (s, 3H), 2.40 – 2.36 (m, 2H), 2.21 – 2.18 (m, 2H), 1.79 – 1.76 (m, 2H), 1.68 – 1.64 (m, 2H) ppm.

^{13}C NMR (100 MHz, $CDCl_3$): δ 158.52, 136.01, 135.48, 126.05, 123.30, 113.67, 55.41, 27.60, 25.97, 23.25, 22.34 ppm.

1h is a known compound. [S1]

3'-methoxy-2,3,4,5-tetrahydro-1,1'-biphenyl (1i)

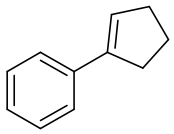


^{1}H NMR (400 MHz, $CDCl_3$): δ 7.23 (t, *J* = 7.9 Hz, 1H), 7.00 – 6.98 (m, 1H), 6.93 (dd, *J* = 2.6, 1.7 Hz, 1H), 6.79 – 6.76 (m, 1H), 6.14 – 6.12 (m, 1H), 3.82 (s, 3H), 2.43 – 2.38 (m, 2H), 2.24 – 2.19 (m, 2H), 1.82 – 1.76 (m, 2H), 1.70 – 1.64 (m, 2H) ppm.

^{13}C NMR (100 MHz, $CDCl_3$): δ 159.68, 144.45, 136.63, 129.21, 125.20, 117.69, 111.95, 110.98, 55.32, 27.60, 25.99, 23.18, 22.28 ppm.

1i is a known compound. [S4]

cyclopent-1-en-1-ylbenzene (1j)

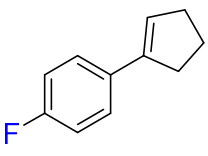


^{1}H NMR (400 MHz, $CDCl_3$): δ 7.48 – 7.42 (m, 2H), 7.32 (dd, *J* = 8.4, 6.9 Hz, 2H), 7.22 (t, *J* = 7.3 Hz, 1H), 6.21 – 6.19 (m, 1H), 2.75 – 2.70 (m, 2H), 2.56 – 2.52 (m, 2H), 2.07 – 2.00 (m, 2H) ppm.

^{13}C NMR (100 MHz, $CDCl_3$): δ 142.57, 136.95, 128.40, 126.95, 126.24, 125.69, 33.49, 33.31, 23.50 ppm.

1j is a known compound. [S3]

1-(cyclopent-1-en-1-yl)-4-fluorobenzene (1k)

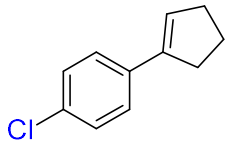


^{1}H NMR (400 MHz, $CDCl_3$): δ 7.40 (dd, *J* = 8.6, 5.6 Hz, 2H), 7.00 (t, *J* = 8.8 Hz, 2H), 6.11 (t, *J* = 2.3 Hz, 1H), 2.73 – 2.65 (m, 2H), 2.56 – 2.50 (m, 2H), 2.07 – 1.98 (m, 2H) ppm.

^{13}C NMR (100 MHz, $CDCl_3$): δ 160.78, 141.51, 133.18, 127.18(d, J= 8 Hz), 125.90(d, J= 2 Hz), 115.20(d, J= 22 Hz), 33.51, 33.50, 23.52 ppm.

1k is a known compound. [S5]

1-chloro-4-(cyclopent-1-en-1-yl)benzene (1l)

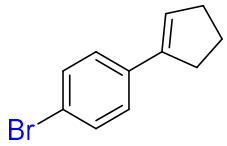


^{1}H NMR (400 MHz, $CDCl_3$): δ 7.40 – 7.35 (m, 2H), 7.32 – 7.27 (m, 2H), 6.21 – 6.19(m, 1H), 2.74 – 2.66 (m, 2H), 2.58 – 2.52 (m, 2H), 2.08 – 2.01 (m, 2H) ppm.

^{13}C NMR (100 MHz, $CDCl_3$): δ 141.49, 135.42, 132.51, 128.50, 127.01, 126.94, 33.54, 33.31, 23.46 ppm.

1l is a known compound. [S3]

1-bromo-4-(cyclopent-1-en-1-yl)benzene (1m)

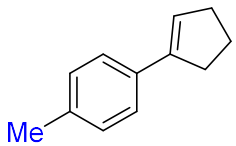


^{1}H NMR (400 MHz, $CDCl_3$): δ 7.46 – 7.40 (m, 2H), 7.34 – 7.27 (m, 2H), 6.20 – 6.17 (m, 1H), 2.70 – 2.65 (m, 2H), 2.54 – 2.50 (m, 2H), 2.07 – 1.98 (m, 2H) ppm.

^{13}C NMR (100 MHz, $CDCl_3$): δ 141.54, 135.86, 131.44, 127.29, 127.18, 120.65, 33.56, 33.26, 23.45 ppm.

1m is a known compound. [S3]

1-(cyclopent-1-en-1-yl)-4-methylbenzene (1n)

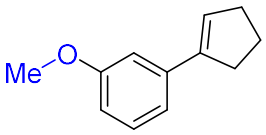


^{1}H NMR (400 MHz, $CDCl_3$): δ 7.35 – 7.32 (m, 2H), 7.12 (d, *J* = 8.0 Hz, 2H), 6.14 – 6.11 (m, 1H), 2.72 – 2.66 (m, 2H), 2.54 – 2.49 (m, 2H), 2.33 (s, 3H), 2.05 – 1.96 (m, 2H) ppm.

^{13}C NMR (100 MHz, $CDCl_3$): δ 142.41, 136.62, 134.16, 129.08, 125.59, 125.19, 33.44, 33.35, 23.48, 21.29 ppm.

1n is a known compound. [S6]

1-(cyclopent-1-en-1-yl)-3-methoxybenzene (1o)

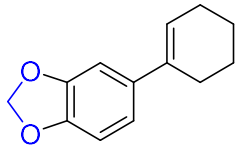


^{1}H NMR (400 MHz, $CDCl_3$): δ 7.23 (t, *J* = 7.9 Hz, 1H), 7.05 (dt, *J* = 7.8, 1.3 Hz, 1H), 6.97 (t, *J* = 2.1 Hz, 1H), 6.79 – 6.75 (m, 1H), 6.18 (t, *J* = 2.3 Hz, 1H), 3.82 (s, 3H), 2.72 – 2.67 (m, 2H), 2.55 – 2.50 (m, 2H), 2.06 – 1.96 (m, 2H) ppm.

^{13}C NMR (100 MHz, $CDCl_3$): δ 159.69, 142.48, 138.45, 129.33, 126.74, 118.36, 112.28, 111.48, 55.33, 33.46, 33.40, 23.47 ppm.

1o is a known compound. [S6]

5-(cyclohex-1-en-1-yl)benzo[d][1,3]dioxole (1p)

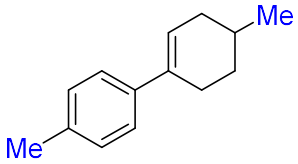


^{1}H NMR (400 MHz, $CDCl_3$): δ 6.90 (d, *J* = 1.8 Hz, 1H), 6.85 (dd, *J* = 8.1, 1.8 Hz, 1H), 6.75 (d, *J* = 8.1 Hz, 1H), 6.02 – 5.99 (m, 1H), 5.93 (s, 2H), 2.37 – 2.33 (m, 2H), 2.20 – 2.16 (m, 2H), 1.81 – 1.73 (m, 2H), 1.68 – 1.60 (m, 2H) ppm.

^{13}C NMR (100 MHz, $CDCl_3$): δ 147.71, 146.34, 137.44, 136.23, 123.95, 118.30, 108.04, 105.81, 100.98, 27.83, 25.95, 23.21, 22.28 ppm.

1p is a known compound. [S3]

4,4'-dimethyl-2,3,4,5-tetrahydro-1,1'-biphenyl (1q)

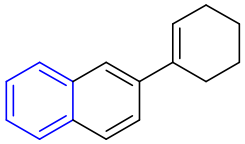


^{1}H NMR (400 MHz, $CDCl_3$): δ 7.31 – 7.27 (m, 2H), 7.12 (d, *J* = 7.9 Hz, 2H), 6.10 – 6.03 (m, 1H), 2.47 – 2.43 (m, 2H), 2.34 (s, 3H), 2.32 – 2.25 (m, 1H), 1.90 – 1.70 (m, 3H), 1.40 – 1.37 (m, 1H), 1.01 (d, *J* = 6.3 Hz, 3H) ppm.

^{13}C NMR (100 MHz, $CDCl_3$): δ 139.68, 136.24, 136.09, 129.00, 124.96, 123.59, 34.57, 31.47, 28.30, 27.58, 21.88, 21.17 ppm.

1q is a known compound. [S3]

2-(cyclohex-1-en-1-yl)naphthalene (1r)

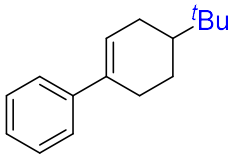


^{1}H NMR (400 MHz, $CDCl_3$): δ 7.85 – 7.76 (m, 4H), 7.61 (dd, *J* = 8.7, 1.8 Hz, 1H), 7.46 – 7.42 (m, 2H), 6.33-6.30 (m, 1H), 2.57 – 2.55 (m, 2H), 2.30 – 2.28 (m, 2H), 1.87 – 1.84 (m, 2H), 1.74 – 1.71 (m, 2H) ppm.

^{13}C NMR (100 MHz, $CDCl_3$): δ 139.93, 136.46, 133.68, 132.54, 128.17, 127.71, 127.61, 126.07, 125.66, 125.46, 123.94, 123.20, 27.52, 26.17, 23.24, 22.35 ppm.

1r is a known compound. [s1]

4-(tert-butyl)-2,3,4,5-tetrahydro-1,1'-biphenyl (1s)

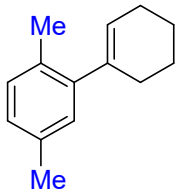


^{1}H NMR (400 MHz, $CDCl_3$): δ 7.40 (m, 2H), 7.35 – 7.29 (m, 2H), 7.25 – 7.20 (m, 1H), 6.17 – 6.13 (m, 1H), 2.58 – 2.40 (m, 2H), 2.32 – 2.23 (m, 1H), 2.04 – 1.96 (m, 2H), 1.40 – 1.29 (m, 2H), 0.93 (d, *J* = 1.2 Hz, 9H) ppm.

^{13}C NMR (100 MHz, $CDCl_3$): δ 142.41, 136.50, 128.32, 126.62, 125.08, 125.06, 43.94, 32.36, 28.99, 27.64, 27.38, 24.55 ppm.

1s is a known compound. [S7]

2',5'-dimethyl-2,3,4,5-tetrahydro-1,1'-biphenyl (1t)

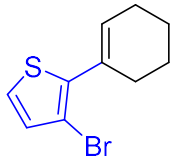


^{1}H NMR (400 MHz, $CDCl_3$): δ 7.05 (d, *J* = 7.7 Hz, 1H), 6.95 (dd, *J* = 7.7, 1.9 Hz, 1H), 6.90 (d, *J* = 2.0 Hz, 1H), 5.55 – 5.52 (m, 1H), 2.30 (s, 3H), 2.24 (s, 3H), 2.21 – 2.14 (m, 4H), 1.79 – 1.64 (m, 4H) ppm.

^{13}C NMR (100 MHz, $CDCl_3$): δ 144.63, 139.09, 134.95, 131.93, 130.01, 129.11, 127.21, 125.56, 30.25, 25.53, 23.28, 22.38, 21.02, 19.40 ppm.

1t is a known compound. [S3]

3-bromo-2-(cyclohex-1-en-1-yl)thiophene (1u)

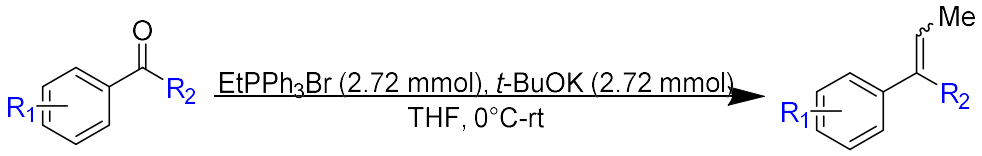


^{1}H NMR (400 MHz, $CDCl_3$): δ 7.09 (d, *J* = 5.3 Hz, 1H), 6.92 (d, *J* = 5.3 Hz, 1H), 6.22 – 6.20 (m, 1H), 2.45 – 2.39 (m, 2H), 2.22 – 2.19 (m, 2H), 1.78 – 1.75 (m, 2H), 1.70 – 1.63 (m, 2H) ppm.

^{13}C NMR (100 MHz, $CDCl_3$): δ 140.98, 131.42, 130.27, 130.15, 123.06, 105.91, 29.59, 25.78, 22.96, 21.84 ppm.

1u is a known compound. [S3]

3. Substrates **1v, 1x** are known compounds and prepared according to the general procedure as below.



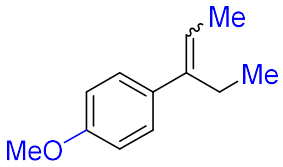
S3 **1v/1x**

To a stirred suspension of $EtPPh_3Br$ (2.72 mmol, 1.0 g) in anhydrous THF, potassium *tert*-butoxide (2.72 mmol, 305mg) was added under argon at 0 ºC and stirred for 2 h. Then the solution of related ketone (1.70 mmol) in anhydrous THF was added slowly and stirred for 30 minutes. The mixture was mixed with water and the aqueous layer was extracted with ethyl acetate (3 x 15 mL). The combined organic extracts were

dried over anhydrous Na_2SO_4, concentrated and purified by silica gel column chromatography using petroleum ether and ethyl acetate as the diluent to afford expected **1v** and **1x**, respectively.

Characteristic Data of Synthesized 1:

1-methoxy-4-(pent-2-en-3-yl)benzene (1v)

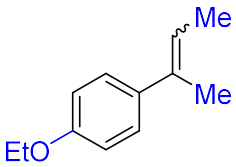


^{1}H NMR (400 MHz, $CDCl_3$ mixture of two inseparable diastereomers at ratio of 5:1) (**major**) δ 7.13 – 7.11 (m, 2H), 6.93 – 6.89 (m, 2H), 5.55 – 5.50 (m, 1H), 3.83 (s, 3H), 2.38 – 2.32 (m, 2H), 1.61 – 1.58 (m, 3H), 1.00 – 0.96 (m, 3H). (**minor**) δ 7.32 – 7.28 (m, 2H), 6.89 – 6.85 (m, 2H), 5.71 – 5.66 (m, 1H), 3.82 (s, 3H), 2.55 – 2.49 (m, 2H), 1.80 (d, J = 6.9 Hz, 3H), 1.02 – 1.01 (m, 3H) ppm.

^{13}C NMR (100 MHz, $CDCl_3$): (**major**) δ 158.15, 142.96, 133.61, 129.65, 119.62, 113.49, 55.28, 32.20, 14.76, 13.30. (**minor**) δ 158.44, 141.83, 135.77, 127.25, 120.68, 113.65, 55.33, 22.75, 14.00, 13.36 ppm.

1v is a known compound. [S8]

1-(but-2-en-2-yl)-4-ethoxybenzene (1x)

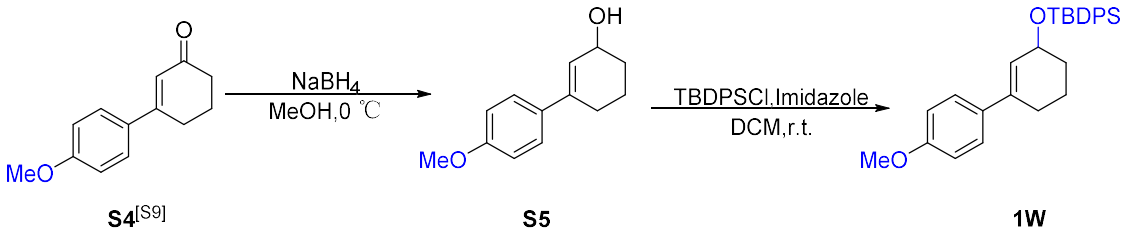


^{1}H NMR (400 MHz, $CDCl_3$ mixture of two inseparable diastereomers at ratio of 6:1): (**major**) δ 7.17 – 7.15 (m, 2H), 6.91 – 6.89 (m, 2H), 5.56 – 5.54 (m, 1H), 4.09 – 4.04 (m, 2H), 2.04 – 2.03(m, 3H), 1.66 – 1.63 (m, 3H), 1.44 (t, 3H). (**minor**) δ 7.35 – 7.30 (m, 2H), 6.88 – 6.83 (m, 2H), 5.82 – 5.80 (m, 1H), 4.08 – 4.03 (m, 2H), 2.04 – 2.03 (m, 3H), 1.81 (dd, J = 6.9, 1.3 Hz, 3H), 1.43 (t, 3H) ppm.

^{13}C NMR (100 MHz, $CDCl_3$): (**major**) δ 157.56, 136.32, 134.08, 129.24, 121.15, 114.04, 63.46, 25.59, 15.07, 15.03. (**minor**) δ 157.81, 136.78, 135.42, 126.57, 120.82, 114.20, 63.51, 25.59, 15.61, 14.39 ppm.

1x is a known compound. [S8]

***tert*-butyl((4'-methoxy-3,4,5,6-tetrahydro-[1,1'-biphenyl]-3-yl)oxy)diphenylsilane (1w)**[S6]



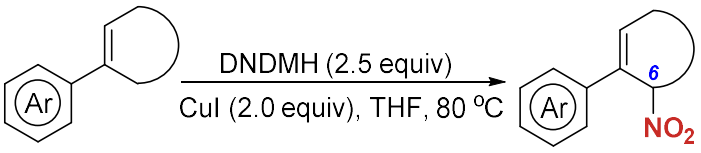
S4[S9] **S5** **1W**

S4 was obtained from literature [9] and prepared according to the general procedure of **1w**.

^{1}H NMR (400 MHz, $CDCl_3$): δ 7.79 – 7.73 (m, 4H), 7.49 – 7.38 (m, 6H), 7.29 – 7.25 (m, 2H), 6.86 (d, *J* = 8.9 Hz, 2H), 5.93 – 5.88 (m, 1H), 4.48 – 4.40 (m, 1H), 3.83 (s, 3H), 2.42 (m, 1H), 2.29 (d, *J* = 17.2 Hz, 1H), 1.96 (q, *J* = 5.6 Hz, 1H), 1.80 – 1.71 (m, 2H), 1.11 (s, 9H) ppm.

^{13}C NMR (100 MHz, $CDCl_3$): δ 158.97, 137.95, 136.05, 135.98, 134.88, 134.75, 134.34, 129.65, 129.63, 127.67, 126.47, 126.21, 113.66, 68.11, 55.36, 31.97, 27.57, 27.18, 19.87, 19.37 ppm.

4. General Procedure for the Direct Nitration of Alkyl Alkenes 1 with DNDMH



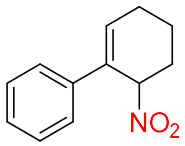
1a **3a**

To an oven-dried reaction glass tube equipped with a magnetic stir-bar, alkyl alkenes **1a** (0.5 mmol, 79 mg), DNDMH (1.25 mmol, 272 mg), and CuI (1.0 mmol, 190.45 mg) were added. Under a nitrogen atmosphere, 3 mL of tetrahydrofuran (THF) was introduced using a laboratory syringe. The mixture was then stirred in an oil bath at 80 °C for 5 hours. The progress of the reaction was monitored by thin-layer chromatography (TLC) analysis. Once the reaction was completed, the solvent was

evaporated under reduced pressure. The residue was purified by silica gel column chromatography, with petroleum ether/ethyl acetate (EtOAc) serving as the eluent.

Characteristic Data of Synthesized 3:

2-nitro-2,3,4,5-tetrahydro-1,1'-biphenyl (3a)



Yield of **3a** 91%, 92.41 mg; yellow oil; R_f = 0.7 (silica, PE:EtOAc = 10:1);

^{1}H NMR (400 MHz, $CDCl_3$): δ 7.39 – 7.27 (m, 5H), 6.52 – 6.47 (m, 1H), 5.67 – 5.61 (m, 1H), 2.56 – 2.41 (m, 2H), 2.36 – 2.17 (m, 2H), 1.82 – 1.76 (m, 2H) ppm.

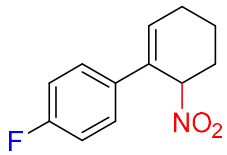
^{13}C NMR (100 MHz, $CDCl_3$): δ 138.89, 133.91, 131.40, 128.76, 127.84, 125.61, 83.10, 29.07, 25.51, 17.38 ppm.

HRMS (ESI) m/z: $[M+Na]^+$ calculated for $C_{12}H_{13}NNaO_2^+$ 226.0838, found 226.0843;

IR (film) νmax =3050, 2950, 1546, 1260, 740 cm^{-1}.

3a is a known compound.[S10]-[S12]

4'-fluoro-2-nitro-2,3,4,5-tetrahydro-1,1'-biphenyl (3b)



Yield of **3b** 80%, 88.43 mg; orange oil; R_f = 0.7 (silica, PE:EtOAc = 10:1);

^{1}H NMR (400 MHz, $CDCl_3$): δ 7.29 – 7.23 (m, 2H), 7.03 – 6.96 (m, 2H), 6.40 – 6.38 (m, 1H), 5.55 – 5.53 (m, 1H), 2.50 – 2.36 (m, 2H), 2.30 – 2.11 (m, 2H), 1.85 – 1.71 (m, 2H) ppm.

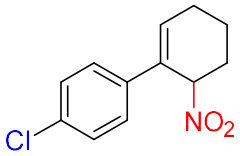
^{13}C NMR (100 MHz, $CDCl_3$): δ162.46 (d, J= 246 Hz), 135.11, 134.06, 130.60, 127.42 (d, J= 8 Hz), 115.64 (d, J=21 Hz), 83.24, 28.98, 25.46, 17.31 ppm.

^{19}F NMR (376 MHz, $CDCl_3$): δ -114.37 ppm.

HRMS (ESI) m/z: $[M+Na]^+$ calculated for $C_{12}H_{12}FNNaO_2^+$ 244.0744, found 244.0721;

IR (film) νmax = 3050,1940,1540,1270,840,810 cm^{-1}.

4'-chloro-2-nitro-2,3,4,5-tetrahydro-1,1'-biphenyl (3c)



Yield of **3c** 60%, 71.11 mg; yellow oil; R_f = 0.7 (silica, PE:EtOAc = 10:1);

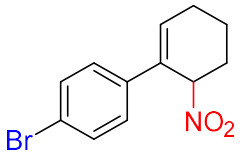
^{1}H NMR (400 MHz, $CDCl_3$): δ 7.31 – 7.27 (m, 2H), 7.26 – 7.22 (m, 2H), 6.42 – 6.40 (m, 1H), 5.53 – 5.50 (m, 1H), 2.48 (d, *J* = 18.6 Hz, 2H), 2.32 – 2.12 (m, 2H), 1.84 – 1.74 (m, 2H) ppm.

^{13}C NMR (100 MHz, $CDCl_3$): δ 137.40, 134.57, 133.71, 130.50, 128.92, 126.98, 82.95, 28.95, 25.52, 17.31 ppm.

HRMS (ESI) m/z: $[M+Na]^+$ calculated for $C_{12}H_{12}ClNNaO_2^+$ 260.0448, found 260.0436;

IR (film) νmax = 3050, 1550, 1260, 730 cm^{-1}.

4'-bromo-2-nitro-2,3,4,5-tetrahydro-1,1'-biphenyl (3d)



Yield of **3d** 81%, 113.80 mg; yellow oil; R_f = 0.7 (silica, PE:EtOAc = 10:1);

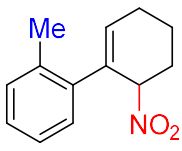
^{1}H NMR (400 MHz, $CDCl_3$): δ 7.47 – 7.42 (m, 2H), 7.20 – 7.15 (m, 2H), 6.47 – 6.43 (m, 1H), 5.56 – 5.52 (m, 1H), 2.52 – 2.37 (m, 2H), 2.32 – 2.12 (m, 2H), 1.87 – 1.73 (m, 2H) ppm.

^{13}C NMR (100 MHz, $CDCl_3$): δ 137.89, 134.64, 131.91, 130.61, 127.33, 121.89, 82.91, 28.96, 25.56, 17.32 ppm.

HRMS (ESI) m/z: $[M+Na]^+$ calculated for $C_{12}H_{12}BrNNaO_2^+$ 303.9943, found 303.9924;

IR (film) νmax = 3056, 1548, 1263, 732 cm^{-1}.

2'-methyl-2-nitro-2,3,4,5-tetrahydro-1,1'-biphenyl (3e)



Yield of **3e** 32%, 34.73 mg; yellow oil; R_f = 0.7 (silica, PE:EtOAc = 10:1);

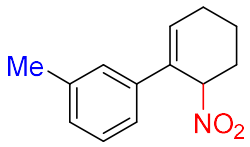
^{1}H NMR (400 MHz, $CDCl_3$): δ 7.20 – 7.12 (m, 3H), 7.11 – 7.07 (m, 1H), 6.06 (m, 1H), 5.36 – 5.32 (m, 1H), 2.50 – 2.37 (m, 2H), 2.31 (s, 3H), 2.29 – 2.20 (m, 2H), 1.97 – 1.85 (m, 1H), 1.81 – 1.77 (m, 1H) ppm.

^{13}C NMR (100 MHz, $CDCl_3$): δ 139.13, 135.76, 135.63, 131.75, 130.58, 129.03, 127.80, 125.88, 85.00, 28.75, 25.11, 19.76, 17.55 ppm.

HRMS (ESI) m/z: $[M+Na]^+$ calculated for $C_{13}H_{15}NNaO_2^+$ 240.0995, found 240.0989;

IR (film) νmax = 2930, 1550, 1454, 1369, 756 cm^{-1}.

3'-methyl-2-nitro-2,3,4,5-tetrahydro-1,1'-biphenyl (3f)



Yield of **3f** 82%, 89.01 mg; yellow oil; R_f = 0.7 (silica, PE:EtOAc = 10:1);

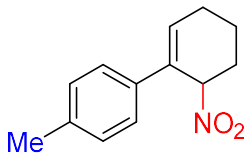
^{1}H NMR (400 MHz, $CDCl_3$): δ 7.22 (t, *J* = 7.6 Hz, 1H), 7.16 – 7.08 (m, 3H), 6.47 – 6.45 (m, 1H), 5.63 – 5.60 (m, 1H), 2.52 – 2.39 (m, 2H), 2.35 (s, 3H), 2.33 – 2.14 (m, 2H), 1.87 – 1.73 (m, 2H) ppm.

^{13}C NMR (100 MHz, $CDCl_3$): δ 138.87, 138.34, 133.67, 131.46, 128.64, 128.62, 126.42, 122.66, 83.13, 29.10, 25.51, 21.56, 17.41 ppm.

HRMS (ESI) m/z: $[M+Na]^+$ calculated for $C_{13}H_{15}NNaO_2^+$ 240.0995, found 240.0989;

IR (film) νmax = 2937, 1542, 1442, 1369, 1267, 784, 700 cm^{-1}.

4'-methyl-2-nitro-2,3,4,5-tetrahydro-1,1'-biphenyl (3g)



Yield of **3g** 90%, 97.69 mg; yellow oil; R_f = 0.7 (silica, PE:EtOAc = 10:1);

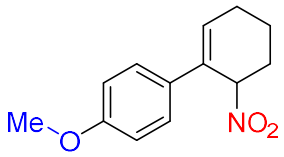
^{1}H NMR (400 MHz, $CDCl_3$): δ 7.24 – 7.20 (m, 2H), 7.14 (d, *J* = 8.1 Hz, 2H), 6.45 – 6.43(m, 1H), 5.62 – 5.58 (m, 1H), 2.51 – 2.38 (m, 2H), 2.33 (s, 3H), 2.31 – 2.14 (m, 2H), 1.88 – 1.73 (m, 2H) ppm.

^{13}C NMR (100 MHz, $CDCl_3$): δ 137.65, 136.02, 133.05, 131.23, 129.46, 125.45, 83.15, 29.10, 25.50, 21.15, 17.42 ppm.

HRMS (ESI) m/z: $[M+Na]^+$ calculated for $C_{13}H_{15}NNaO_2^+$ 240.0995, found 240.0989;

IR (film) νmax = 2923, 1542, 1282, 1373, 804 cm^{-1}.

4'-methoxy-2-nitro-2,3,4,5-tetrahydro-1,1'-biphenyl (3h)



Yield of **3h** 70%,81.58 mg; yellow oil; R_f = 0.5 (silica, PE:EtOAc = 10:1);

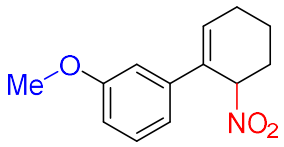
^{1}H NMR (400 MHz, $CDCl_3$): δ 7.27 (d, *J* = 8.8 Hz, 2H), 6.88 (d, *J* = 8.8 Hz, 2H), 6.44 – 6.37 (m, 1H), 5.61 – 5.59 (m, 1H), 3.81 (s, 3H), 2.52 – 2.38 (m, 2H), 2.32 – 2.13 (m, 2H), 1.88 – 1.74 (m, 2H) ppm.

^{13}C NMR (100 MHz, $CDCl_3$): δ 159.31, 132.37, 131.38, 130.76, 126.71, 114.11, 83.23, 55.35, 29.09, 25.45, 17.37 ppm.

HRMS (ESI) m/z: $[M+Na]^+$ calculated for $C_{13}H_{15}NNaO_3^+$ 256.0944, found 256.0932;

IR (film) νmax = 2937, 1546, 1265, 732, 703 cm^{-1}.

3'-methoxy-2-nitro-2,3,4,5-tetrahydro-1,1'-biphenyl (3i)



Yield of **3i** 90%, 104.89 mg; yellow oil; R_f = 0.5 (silica, PE:EtOAc = 10:1);

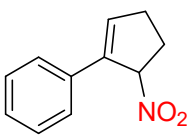
^{1}H NMR (400 MHz, $CDCl_3$): δ 7.26 (t, *J* = 7.9 Hz, 1H), 6.94 – 6.81 (m, 3H), 6.51 – 5.49(m, 1H), 5.64 – 5.59 (m, 1H), 3.82 (s, 3H), 2.53 – 2.41 (m, 2H), 2.35 – 2.16 (m, 2H), 1.84 – 1.78 (m, 2H) ppm.

^{13}C NMR (100 MHz, $CDCl_3$): δ 159.91, 140.38, 134.06, 131.33, 129.74, 117.96, 113.22, 111.65, 83.14, 55.32, 29.09, 25.51, 17.42 ppm.

HRMS (ESI) m/z: $[M+Na]^+$ calculated for $C_{13}H_{15}NNaO_3^+$ 256.0944, found 256.0937;

IR (film) νmax = 2937, 1542, 1286, 1050, 856, 781, 694 cm^{-1}.

(5-nitrocyclopent-1-en-1-yl)benzene (3j)



Yield of **3j** 75%, 70.90 mg; yellow oil; R_f = 0.7 (silica, PE:EtOAc = 10:1);

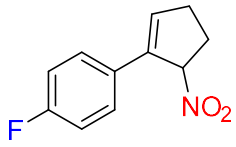
^{1}H NMR (400 MHz, $CDCl_3$): δ 7.47 – 7.42 (m, 2H), 7.35 (t, *J* = 7.4 Hz, 2H), 7.29 (d, *J* = 7.2 Hz, 1H), 6.70 (t, *J* = 2.7 Hz, 1H), 5.98 – 5.93 (m, 1H), 2.97 – 2.88 (m, 1H), 2.69 – 2.62 (m, 1H), 2.57 – 2.53 (m, 2H) ppm.

^{13}C NMR (100 MHz, $CDCl_3$): δ 138.64, 136.66, 133.04, 128.90, 128.33, 125.78, 92.41, 32.09, 31.39 ppm.

HRMS (ESI) m/z: $[M+Na]^+$ calculated for $C_{11}H_{11}NNaO_2^+$ 212.0682, found 212.0668;

IR (film) νmax = 3060, 1544, 1367, 765, 692 cm^{-1}.

1-fluoro-4-(5-nitrocyclopent-1-en-1-yl)benzene (3k)



Yield of **3k** 75%, 77.64 mg; orange oil; R_f = 0.7 (silica, PE:EtOAc = 10:1);

^{1}H NMR (400 MHz, $CDCl_3$): δ 7.43 – 7.39 (m, 2H), 7.06 – 7.00 (m, 2H), 6.62 – 6.61 (m, 1H), 5.92 – 5.89 (m, 1H), 2.93 – 2.87 (m, 1H), 2.70 – 2.60 (m, 1H), 2.57 – 2.52 (m, 2H) ppm.

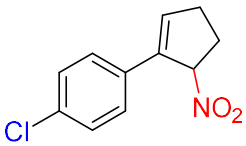
^{13}C NMR (100 MHz, $CDCl_3$): δ162.65 (d, J= 247 Hz), 137.67, 136.49, 129.28, 127.58(d, J= 8 Hz), 115.87 (d, J= 21 Hz), 92.53, 32.09, 31.31 ppm.

^{19}F NMR (376 MHz, $CDCl_3$): δ -113.05 ppm.

HRMS (ESI) m/z: $[M+Na]^+$ calculated for $C_{11}H_{10}FNNaO_2^+$ 230.0587, found 230.0591;

IR (film) νmax = 3062, 2923, 1544, 1510, 1363, 1232, 833, 750 cm^{-1}.

1-chloro-4-(5-nitrocyclopent-1-en-1-yl)benzene (3l)



Yield of **3l** 50%, 55.76 mg; yellow oil; R_f = 0.7 (silica, PE:EtOAc = 10:1);

^{1}H NMR (400 MHz, $CDCl_3$): δ 7.36 (d, *J* = 8.7 Hz, 2H), 7.30 (d, *J* = 8.6 Hz, 2H), 6.68 – 6.67 (m, 1H), 5.91 – 5.88 (m, 1H), 2.97 – 2.87 (m, 1H), 2.70 – 2.61 (m, 1H), 2.58 – 2.53 (m, 2H) ppm.

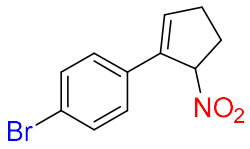
^{13}C NMR (100 MHz, $CDCl_3$): δ 137.64, 137.38, 134.14, 131.54, 129.09, 127.10,

92.30, 32.16, 31.30 ppm.

HRMS (ESI) m/z: $[M+Na]^+$ calculated for $C_{11}H_{10}ClNNaO_2^+$ 246.0292, found 246.0279;

IR (film) νmax = 2927, 1542, 1492, 1365, 1093, 827 cm^{-1}.

1-bromo-4-(5-nitrocyclopent-1-en-1-yl)benzene (3m)



Yield of **3m** 67%, 89.43 mg; orange oil; R_f = 0.7 (silica, PE:EtOAc = 10:1);

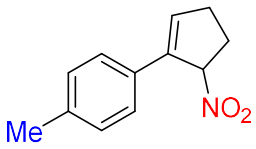
^{1}H NMR (400 MHz, $CDCl_3$): δ 7.46 (d, *J* = 8.7 Hz, 2H), 7.30 (d, *J* = 8.5 Hz, 2H), 6.69 (t, *J* = 2.7 Hz, 1H), 5.93 – 5.86 (m, 1H), 2.96 – 2.85 (m, 1H), 2.69 – 2.60 (m, 1H), 2.59 – 2.52 (m, 2H) ppm.

^{13}C NMR (100 MHz, $CDCl_3$): δ 137.69, 137.52, 132.03, 131.98, 127.38, 122.31, 92.23, 32.17, 31.29 ppm.

HRMS (ESI) m/z: $[M+Na]^+$ calculated for $C_{11}H_{10}BrNNaO_2^+$ 289.9787, found 289.9789;

IR (film) νmax = 3064, 2923, 1542, 1365, 798, 761 cm^{-1}.

1-methyl-4-(5-nitrocyclopent-1-en-1-yl)benzene (3n)



Yield of **3n** 73%, 74.12 mg; yellow oil; R_f = 0.7 (silica, PE:EtOAc = 10:1);

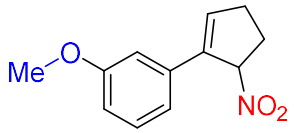
^{1}H NMR (400 MHz, $CDCl_3$): δ 7.36 – 7.32 (m, 2H), 7.15 (d, *J* = 7.9 Hz, 2H), 6.64 – 6.63 (m, 1H), 5.95 – 5.92(m, 1H), 2.93 – 2.88 (m, 1H), 2.69 – 2.59 (m, 1H), 2.56 – 2.51 (m, 2H), 2.34 (s, 3H) ppm.

^{13}C NMR (100 MHz, $CDCl_3$): δ 138.56, 138.25, 135.57, 130.26, 129.58, 125.69, 92.50, 32.03, 31.38, 21.29 ppm.

HRMS (ESI) m/z: $[M+Na]^+$ calculated for $C_{12}H_{13}NNaO_2^+$ 226.0838, found 226.0832;

IR (film) νmax = 2923, 1546, 1367, 1278, 750 cm^{-1}.

1-methoxy-3-(5-nitrocyclopent-1-en-1-yl)benzene (3o)



Yield of **3o** 83%, 90.88 mg; yellow oil; R_f = 0.5 (silica, PE:EtOAc = 10:1);

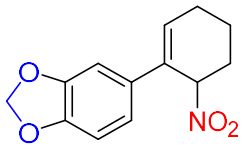
^{1}H NMR (400 MHz, $CDCl_3$): δ 7.28 (t, *J* = 8.0 Hz, 1H), 7.06 – 7.03 (m, 1H), 7.00 (t, *J* = 2.1 Hz, 1H), 6.87 – 6.84 (m, 1H), 6.71 (t, *J* = 2.7 Hz, 1H), 5.96 – 5.95 (m, 1H), 3.83 (s, 3H), 2.99 – 2.88 (m, 1H), 2.71 – 2.61 (m, 1H), 2.56 (d, *J* = 7.3 Hz, 2H) ppm.

^{13}C NMR (100 MHz, $CDCl_3$): δ 159.94, 138.56, 137.01, 134.42, 129.92, 118.25, 113.78, 111.54, 92.46, 55.34, 32.06, 31.38 ppm.

HRMS (ESI) m/z: $[M+Na]^+$ calculated for $C_{12}H_{13}NNaO_3^+$ 242.0787, found 242.0780;

IR (film) νmax = 2937, 1542, 1363, 1282, 1043, 777, 688 cm^{-1}.

5-(6-nitrocyclohex-1-en-1-yl)benzo[d][1,3]dioxole (3p)



Yield of **3p** 72%, 88.94 mg; yellow oil; R_f = 0.5 (silica, PE:EtOAc = 10:1);

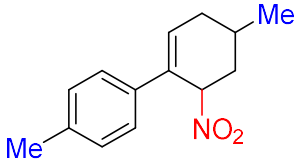
^{1}H NMR (400 MHz, $CDCl_3$): δ 6.82 (s, 1H), 6.78 – 6.73 (m, 2H), 6.38 – 6.32 (m, 1H), 5.94 (d, *J* = 0.9 Hz, 2H), 5.56 – 5.50 (m, 1H), 2.49 – 2.35 (m, 2H), 2.30 – 2.11 (m, 2H), 1.85 – 1.70 (m, 2H) ppm.

^{13}C NMR (100 MHz, $CDCl_3$): δ 148.11, 147.38, 133.23, 133.03, 131.04, 119.08, 108.40, 106.50, 101.28, 83.38, 29.10, 25.47, 17.38 ppm.

HRMS (ESI) m/z: $[M+Na]^+$ calculated for $C_{13}H_{13}NNaO_4^+$ 270.0736, found 270.0723;

IR (film) νmax = 2914, 1541, 1242, 1035, 867, 802 cm^{-1}.

4,4'-dimethyl-2-nitro-2,3,4,5-tetrahydro-1,1'-biphenyl (3q)



Yield of **3q** 82%, 94.75 mg; yellow oil; R_f = 0.7 (silica, PE:EtOAc = 10:1);

^{1}H NMR (400 MHz, $CDCl_3$): δ 7.24 – 7.20 (m, 2H), 7.14 (d, J = 8.1 Hz, 2H), 6.46 – 6.44 (m, 1H), 5.60 – 5.57 (d, J = 5.4 Hz, 1H), 2.54 – 2.41 (m, 2H), 2.33 (s, 3H), 2.07 –

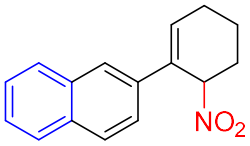
1.96 (m, 1H), 1.93 – 1.78 (m, 2H), 1.04 (d, J = 6.5 Hz, 3H) ppm.

^{13}C NMR (100 MHz, $CDCl_3$): δ 137.72, 135.78, 133.22, 130.61, 129.50, 125.49, 83.59, 36.76, 34.33, 23.32, 21.19, 20.99 ppm.

HRMS (ESI) m/z: $[M+Na]^+$ calculated for $C_{14}H_{17}NNaO_2^+$ 254.1151, found 254.1140;

IR (film) νmax = 2956, 2923, 1546, 1274, 806, 750 cm^{-1}.

2-(6-nitrocyclohex-1-en-1-yl)naphthalene (3r)



Yield of **3r** 90%, 113.89 mg; yellow solid; R_f = 0.7 (silica, PE:EtOAc = 10:1);

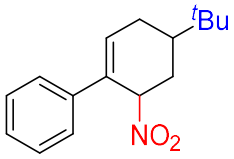
^{1}H NMR (400 MHz, $CDCl_3$): δ 7.86–7.78 (m, 3H), 7.73 (d, *J* = 1.9 Hz, 1H), 7.54 – 7.43 (m, 3H), 6.63 – 6.61 (m, 1H), 5.79 – 5.73 (m, 1H), 2.58 – 2.44 (m, 2H), 2.38 – 2.18 (m, 2H), 1.87 – 1.79 (m, 2H) ppm.

^{13}C NMR (100 MHz, $CDCl_3$): δ 136.13, 134.46, 133.40, 132.89, 131.19, 128.50, 128.26, 127.64, 126.48, 126.20, 124.13, 123.89, 83.03, 29.13, 25.66, 17.38 ppm.

HRMS (ESI) m/z: $[M+Na]^+$ calculated for $C_{16}H_{15}NNaO_2^+$ 276.0995, found 276.0983;

IR (film) νmax = 2921, 1542, 1359, 1278, 854, 810, 750 cm^{-1}.

4-(tert-butyl)-2-nitro-2,3,4,5-tetrahydro-1,1'-biphenyl (3s)



Yield of **3s** 65%, 84.22 mg; yellow oil; R_f = 0.7 (silica, PE:EtOAc = 10:1);

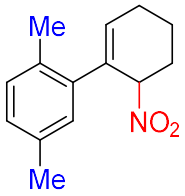
^{1}H NMR (400 MHz, $CDCl_3$): δ 7.33 – 7.30 (m, 4H), 7.28 – 7.25 (m, 1H), 6.54 (dd, *J* = 5.6, 2.6 Hz, 1H), 5.64 – 5.60 (m, 1H), 2.63 – 2.56 (m, 1H), 2.47 – 2.41 (m, 1H), 2.11 – 2.01 (m, 1H), 1.85 – 1.76 (m, 1H), 1.69 – 1.60 (m, 1H), 0.92 (s, 9H) ppm.

^{13}C NMR (100 MHz, $CDCl_3$): δ 138.68, 134.57, 130.60, 128.80, 127.86, 125.62, 83.86, 37.82, 31.79, 30.42, 27.66, 27.06 ppm.

HRMS (ESI) m/z: $[M+Na]^+$ calculated for $C_{16}H_{21}NNaO_2^+$ 282.1464, found 282.1455;

IR (film) νmax = 2960, 1544, 1365, 1265, 754 cm^{-1}.

2',5'-dimethyl-2-nitro-2,3,4,5-tetrahydro-1,1'-biphenyl (3t)



Yield of **3t** 53%, 61.24 mg; yellow oil; R_f = 0.7 (silica, PE:EtOAc = 10:1);

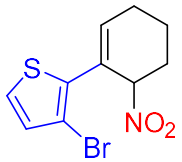
^{1}H NMR (400 MHz, $CDCl_3$): δ 7.07 (d, *J* = 7.7 Hz, 1H), 6.99 (dd, *J* = 7.8, 1.9 Hz, 1H), 6.91 (d, *J* = 1.8 Hz, 1H), 6.06 – 6.04 (m, 1H), 5.36 – 5.33 (m, 1H), 2.49 – 2.37 (m, 2H), 2.29 (s, 3H), 2.27 (s, 3H), 2.25 – 2.15 (m, 2H), 1.93 – 1.88 (m, 1H), 1.83 – 1.73 (m, 1H) ppm.

^{13}C NMR (100 MHz, $CDCl_3$): δ 138.98, 135.44, 135.26, 132.53, 131.84, 130.46, 129.64, 128.53, 84.96, 28.76, 25.10, 20.98, 19.28, 17.51 ppm.

HRMS (ESI) m/z: $[M+Na]^+$ calculated for $C_{14}H_{17}NNaO_2^+$ 254.1151, found 254.1156;

IR (film) νmax = 2927, 1544, 1369, 1265, 858, 811 cm^{-1}.

3-bromo-2-(6-nitrocyclohex-1-en-1-yl)thiophene (3u)



Yield of **3u** 80%, 115.58 mg; yellow oil; R_f = 0.7 (silica, PE:EtOAc = 10:1);

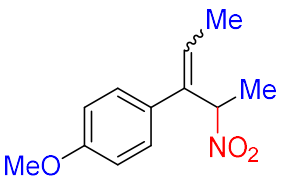
^{1}H NMR (400 MHz, $CDCl_3$): δ 7.19 (d, *J* = 5.4 Hz, 1H), 6.92 (d, *J* = 5.3 Hz, 1H), 6.50 – 6.46 (m, 1H), 5.60 – 5.59(m, 1H), 2.56 – 2.38 (m, 2H), 2.35 – 2.12 (m, 2H), 1.82 – 1.72 (m, 2H) ppm.

^{13}C NMR (100 MHz, $CDCl_3$): δ 140.08, 136.10, 130.88, 125.35, 124.45, 108.38, 83.16, 28.47, 25.52, 16.87 ppm.

HRMS (ESI) m/z: $[M+K]^+$ calculated for $C_{10}H_{10}BrSNKO_2^+$ 325.9247, found 325.9230;

IR (film) νmax = 2921, 1542, 1371, 1278, 864, 752, 713 cm^{-1}.

1-methoxy-4-(4-nitropent-2-en-3-yl)benzene (3v)



Yield of **3v** 48%, 53.06mg; yellow oil; R_f = 0.7 (silica, PE:EtOAc = 10:1);

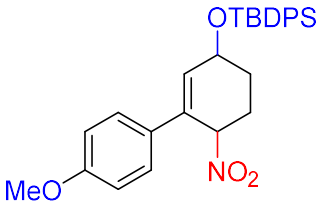
^{1}H NMR (400 MHz, $CDCl_3$ mixture of two inseparable diastereomers at ratio of 10:7): (**major**) δ 7.02 – 7.00 (m, 2H), 6.89 – 6.87 (m, 2H), 6.03 (q, 1H), 5.26 (q, 1H), 3.81 (s, 3H), 1.60 (d, 6H). (**minor**) δ 7.09 – 7.07 (m, 2H), 6.85 – 6.81 (m, 2H), 5.90 (q, 1H), 5.65 (q, 1H), 3.80 (s, 3H), 1.92 (d, 3H), 1.57 (d, 3H) ppm.

^{13}C NMR (100 MHz, $CDCl_3$): (**major**) δ 159.19, 137.01, 132.00, 130.34, 129.45, 114.01, 88.58, 55.34, 17.92, 14.99. (**minor**) δ 159.19, 136.60, 131.86, 129.88, 128.46, 113.72, 81.09, 55.36, 17.46, 14.19 ppm.

HRMS (ESI) m/z: $[M+Na]^+$ calculated for $C_{12}H_{15}NNaO_2^+$ 244.0944, found 244.0942;

IR (film) νmax = 2990, 1550, 1518, 1375, 1280, 1055, 830 cm^{-1}.

***tert*-butyl((4'-methoxy-6-nitro-3,4,5,6-tetrahydro-[1,1'-biphenyl]-3-yl)oxy)diphenylsilane (3w)**



Yield of **3w** 20%, 48.71 mg; yellow oil; R_f = 0.5 (silica, PE:EtOAc = 10:1);

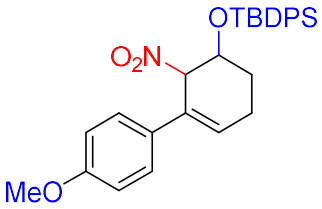
^{1}H NMR (400 MHz, $CDCl_3$): δ 7.76-7.71 (m, 4H), 7.48 – 7.37 (m, 6H), 7.14 (d, *J* = 8.8 Hz, 2H), 6.82 (d, *J* = 8.8 Hz, 2H), 6.14 (d, *J* = 3.0 Hz, 1H), 5.45 (s, 1H), 4.37 (s, 1H), 3.79 (s, 3H), 2.55 – 2.45 (m, 1H), 2.13 – 1.89 (m, 2H), 1.83 – 1.76 (m, 1H), 1.10 (s, 9H) ppm.

^{13}C NMR (100 MHz, $CDCl_3$): δ 159.71, 136.02, 135.92, 135.21, 133.95, 131.57, 130.10, 129.95, 127.88, 127.08, 114.17, 82.72, 67.56, 55.42, 27.31, 27.03, 26.84, 19.35 ppm.

HRMS (ESI) m/z: $[M+Na]^+$ calculated for $C_{29}H_{33}NSiNaO_4^+$ 510.2071,found 510.2082;

IR (film) νmax = 2929, 2856, 1550, 1514, 1257, 1107, 833, 744, 702 cm^{-1}.

***tert*-butyl((4'-methoxy-2-nitro-2,3,4,5-tetrahydro-[1,1'-biphenyl]-3-yl)oxy)diphenylsilane (3w')**



Yield of **3w'** 50%, 120 mg; yellow oil; R_f = 0.7 (silica, PE:EtOAc = 10:1);

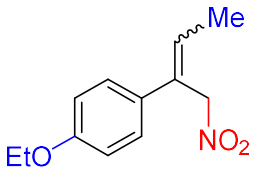
^{1}H NMR (400 MHz, $CDCl_3$): δ 7.71 – 7.63 (m, 8H), 7.49 – 7.34 (m, 12H), 7.18 – 7.12 (m, 4H), 6.86 – 6.78 (m, 4H), 6.21 – 6.17 (m, 1H), 6.12 (dd, *J* = 4.1, 0.8 Hz, 1H), 5.61 – 5.57 (m, 1H), 5.54 – 5.50 (m, 1H), 4.52-4.50 (m, 1H), 4.47-4.44 (m, 1H), 3.78 (s, 3H), 3.78 (s, 3H), 2.62 – 2.52 (m, 1H), 2.42-2.36 (m, 1H), 2.27 – 2.11 (m, 2H), 1.91 – 1.82 (m, 1H), 1.81-1.73 (m, 3H), 1.05 (s, 9H) , 1.04 (s, 9H) ppm.

^{13}C NMR (100 MHz, $CDCl_3$): δ159.95, 159.08, 135.90, 133.42, 131.26, 130.79, 130.27, 128.04, 127.90, 127.16, 127.03, 114.21, 114.07, 90.37, 83.51, 71.48, 65.83, 55.42, 27.79, 27.25, 27.08, 26.91, 25.64, 23.03, 19.41, 19.33 ppm.

HRMS (ESI) m/z: $[M+Na]^+$ calculated for $C_{29}H_{33}NSiNaO_4^+$ 510.2071,found 510.2080;

IR (film) νmax = 2929, 2856, 1550, 1512, 1253, 1110, 832, 742, 703 cm^{-1}.

1-ethoxy-4-(1-nitrobut-2-en-2-yl)benzene (3x)



Yield of **3x** 55%, 60.80mg; yellow oil; R_f = 0.7 (silica, PE:EtOAc = 10:1);

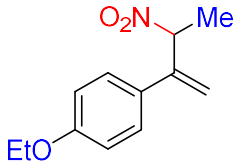
^{1}H NMR (400 MHz, $CDCl_3$ mixture of two inseparable diastereomers at ratio of 1:1): δ 7.29 – 7.25 (m, 2H), 7.12 (d, *J* = 8.7 Hz, 2H), 6.91 – 6.83 (m, 4H), 6.25 (q, *J* = 7.1 Hz, 1H), 6.03 (q, *J* = 6.9 Hz, 1H), 5.38 (s, 2H), 5.12 (s, 2H), 4.03 (q, 2H), 4.02 (q, 2H), 1.94 (d, *J* = 7.1 Hz, 3H), 1.73 (d, *J* = 6.9 Hz, 3H), 1.41 (t, 3H), 1.40 (t, 3H) ppm.

^{13}C NMR (100 MHz, $CDCl_3$): δ 158.74, 158.62, 133.38, 132.16, 131.81, 131.57, 130.21, 129.82, 128.43, 127.13, 114.71, 114.58, 83.29, 74.47, 63.61, 63.54, 15.31, 14.96, 14.94, 14.85 ppm.

HRMS (ESI) m/z: $[M+Na]^+$ calculated for $C_{12}H_{15}NNaO_2^+$ 244.0944, found 244.0933

IR (film) νmax = 2993, 2922, 1548, 1518, 1374, 1268, 1183, 1058, 832 cm^{-1}.

1-ethoxy-4-(3-nitrobut-1-en-2-yl)benzene (3x')



Yield of **3x'** 15%, 16.58 mg; yellow oil; R_f = 0.5 (silica, PE:EtOAc = 10:1);

^{1}H NMR (400 MHz, $CDCl_3$): δ 7.32 – 7.27 (m, 2H), 6.90 – 6.84 (m, 2H), 5.60 – 5.54 (m, 1H), 5.52 (s, 1H), 5.43 (d, *J* = 1.0 Hz, 1H), 4.03 (q, *J* = 7.0 Hz, 2H, 2H), 1.75 (d, *J* = 6.9 Hz, 3H), 1.41 (t, *J* = 7.0 Hz, 3H) ppm.

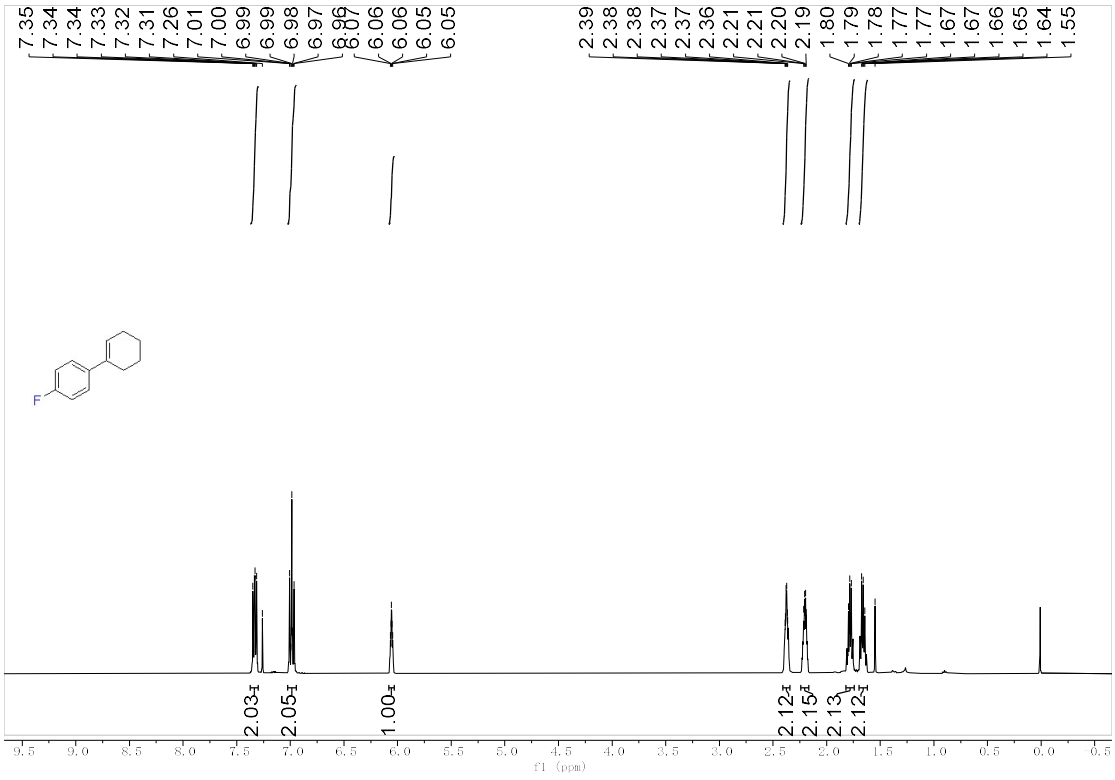
^{13}C NMR (100 MHz, $CDCl_3$): δ 159.28, 143.78, 130.54, 127.84, 116.31, 114.64, 85.27, 63.59, 18.58, 14.89 ppm.

HRMS (ESI) m/z: $[M+Na]^+$ calculated for $C_{12}H_{15}NNaO_2^+$ 244.0944, found 244.0939;

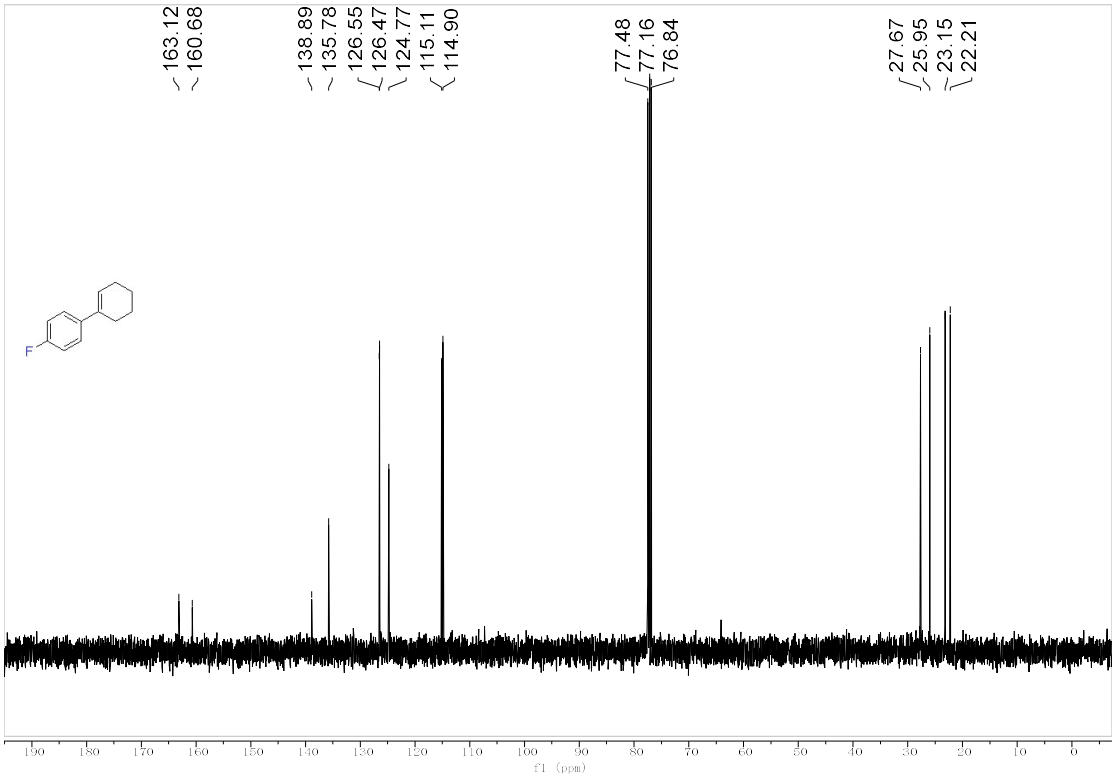
IR (film) νmax = 2981, 1544, 1510, 1240, 1182, 1043, 919, 837, 748 cm^{-1}.

5. Copies of NMR spectra

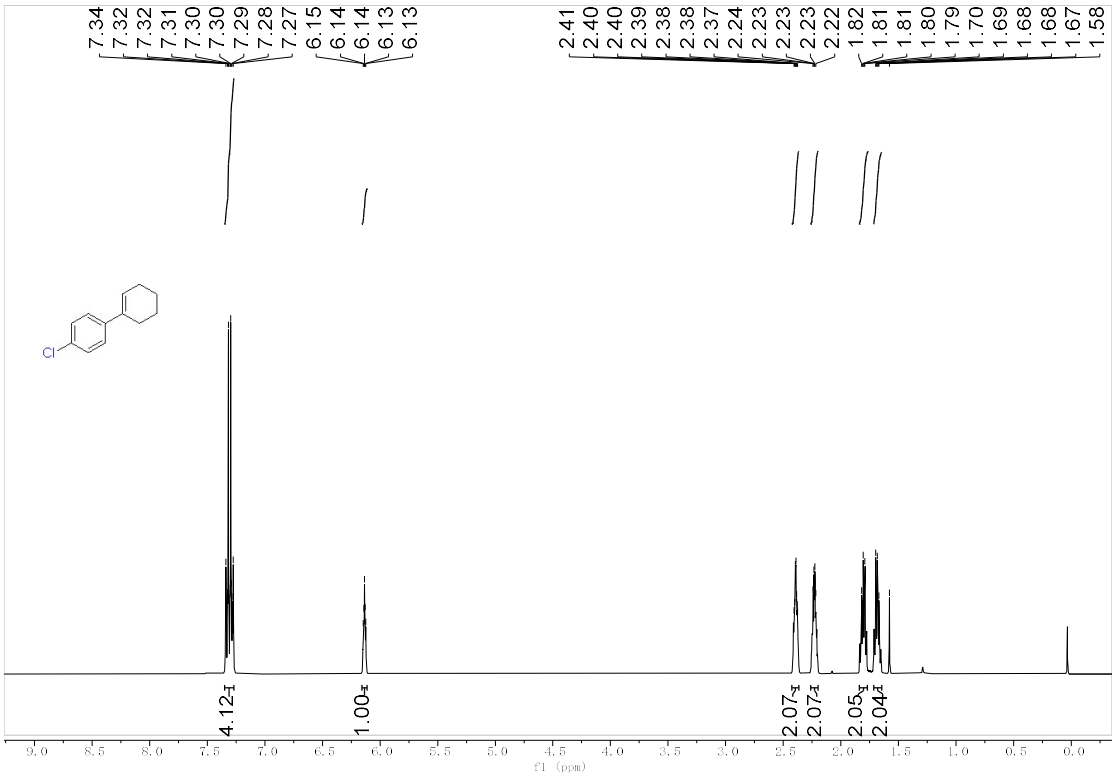
^{1}H NMR of 1b (400 MHz, $CDCl_3$)



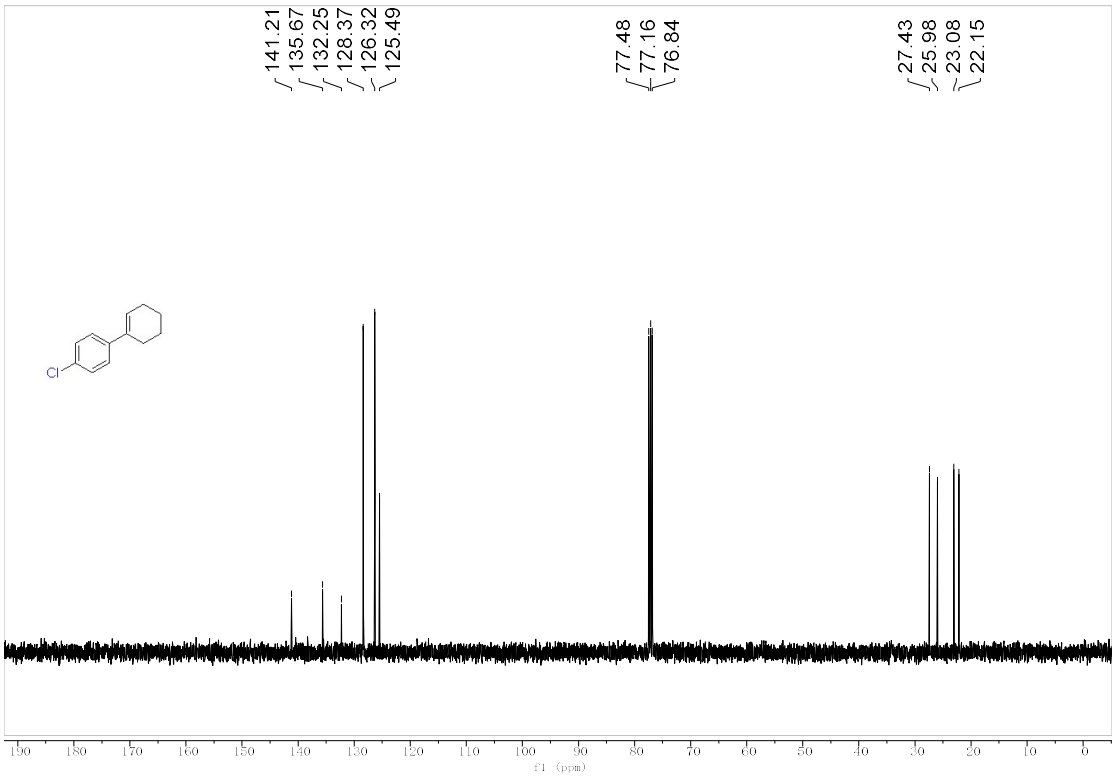
^{13}C NMR of 1b (100 MHz, $CDCl_3$)



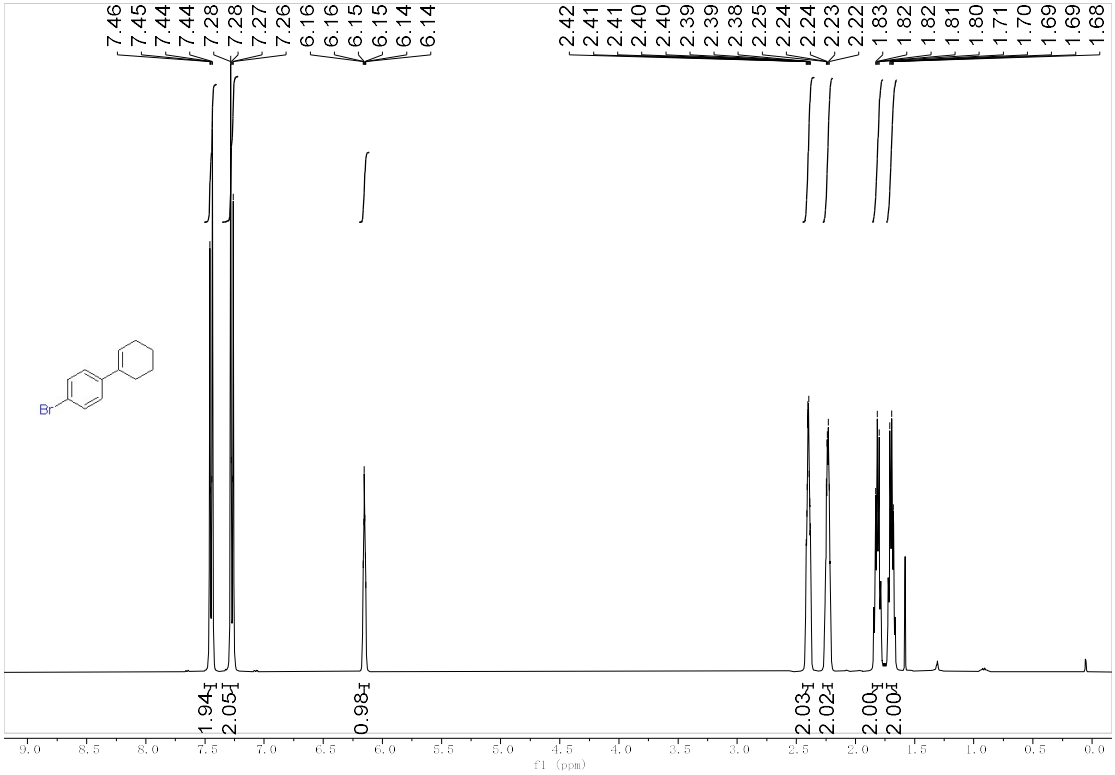
^{1}H NMR of 1c (400 MHz, $CDCl_3$)



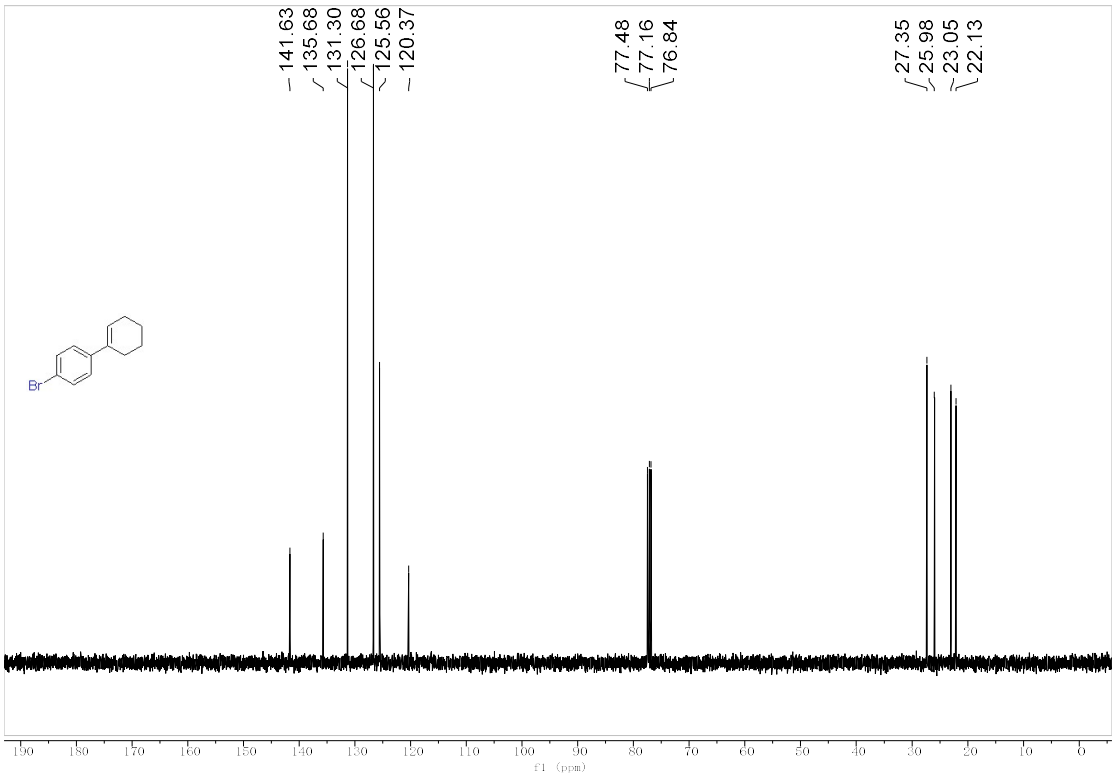
^{13}C NMR of 1c (100 MHz, $CDCl_3$)



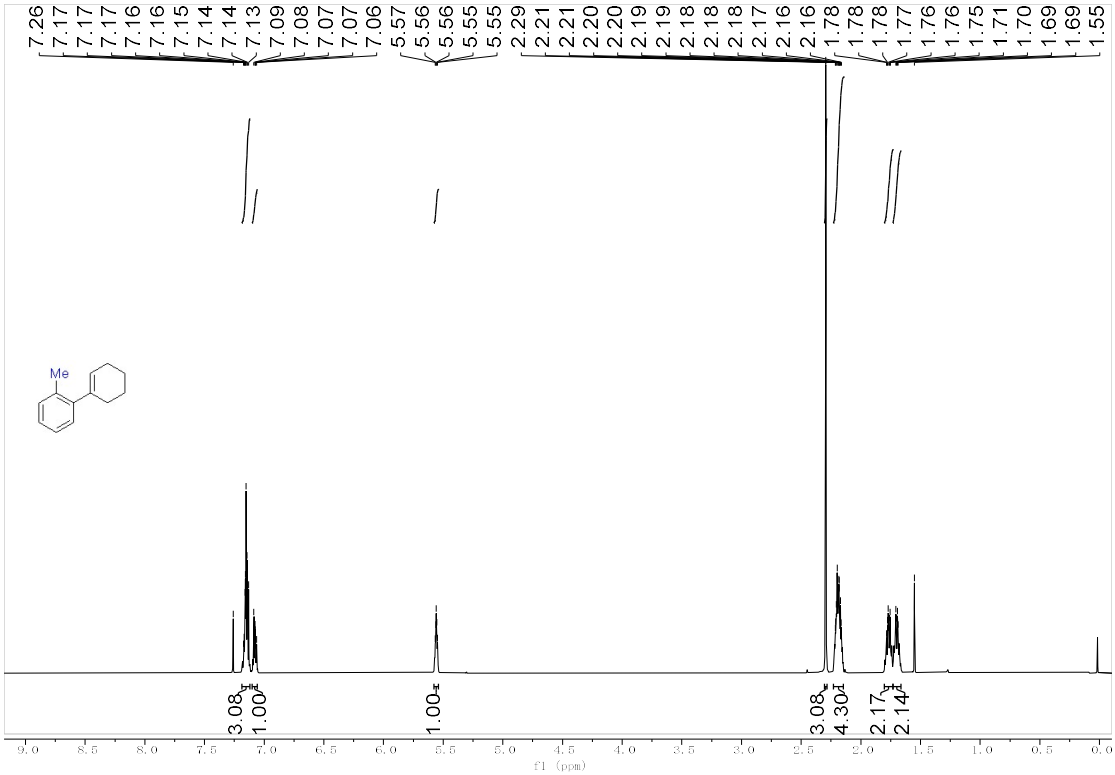
^{1}H NMR of 1d (400 MHz, $CDCl_3$)



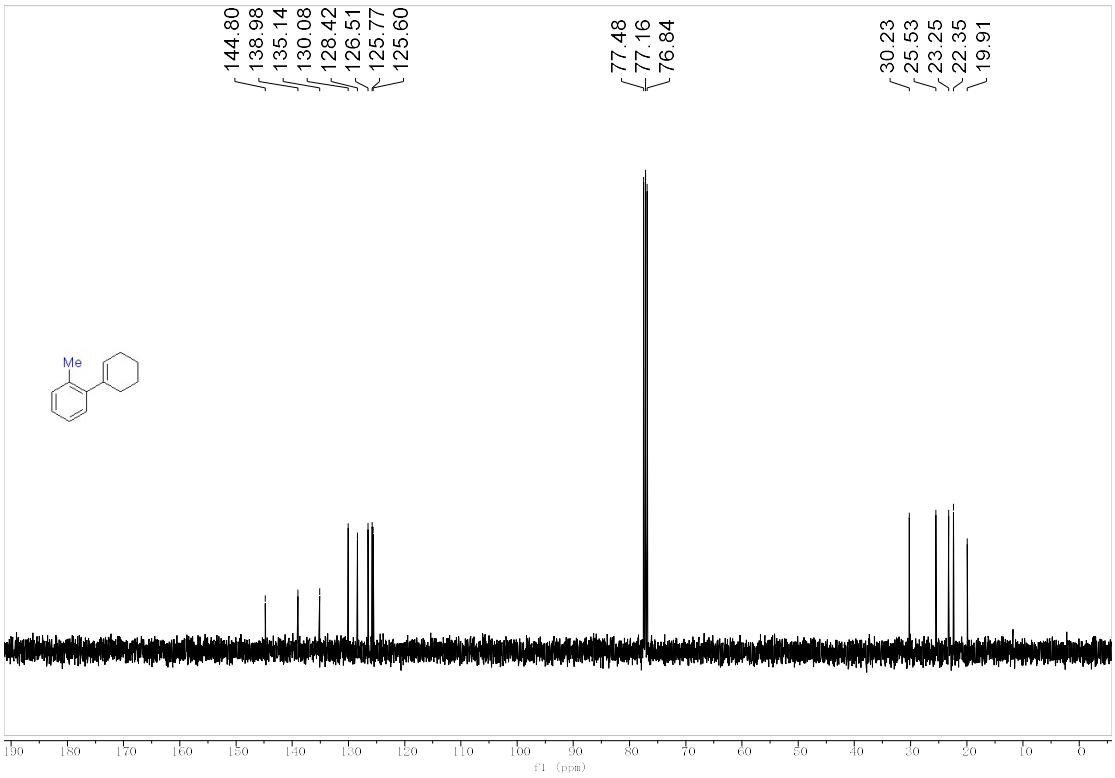
^{13}C NMR of 1d (100 MHz, $CDCl_3$)



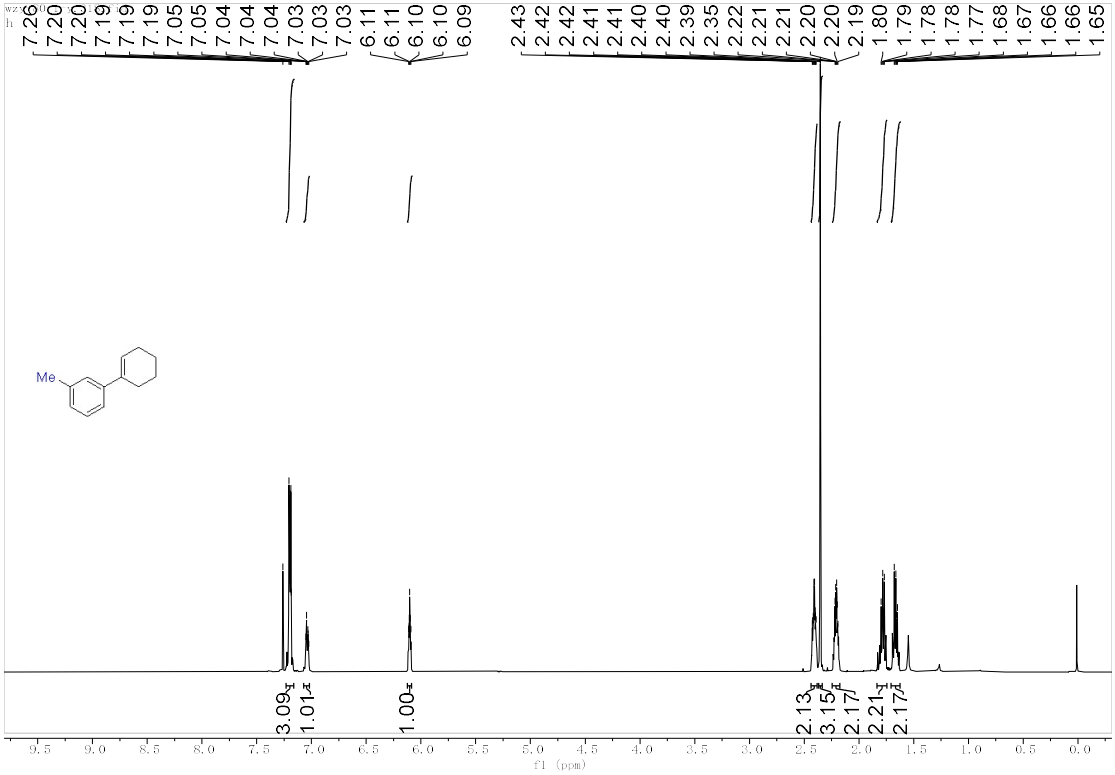
^{1}H NMR of 1e (400 MHz, $CDCl_3$)



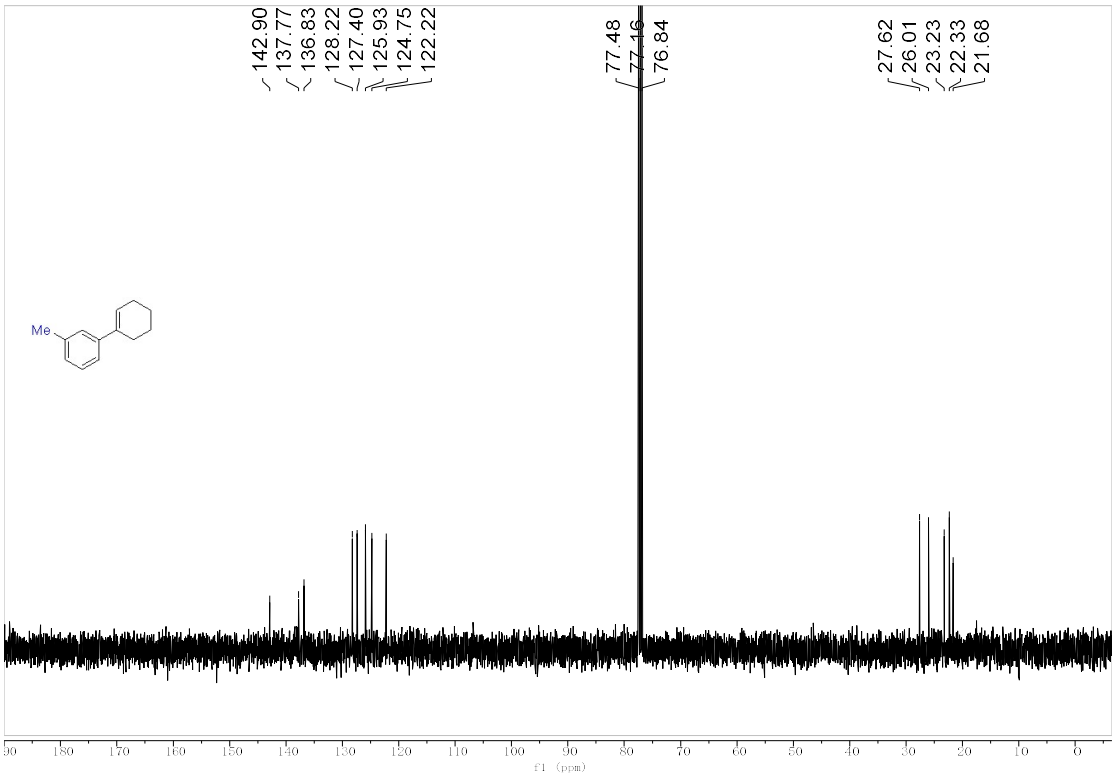
^{13}C NMR of 1e (100 MHz, $CDCl_3$)



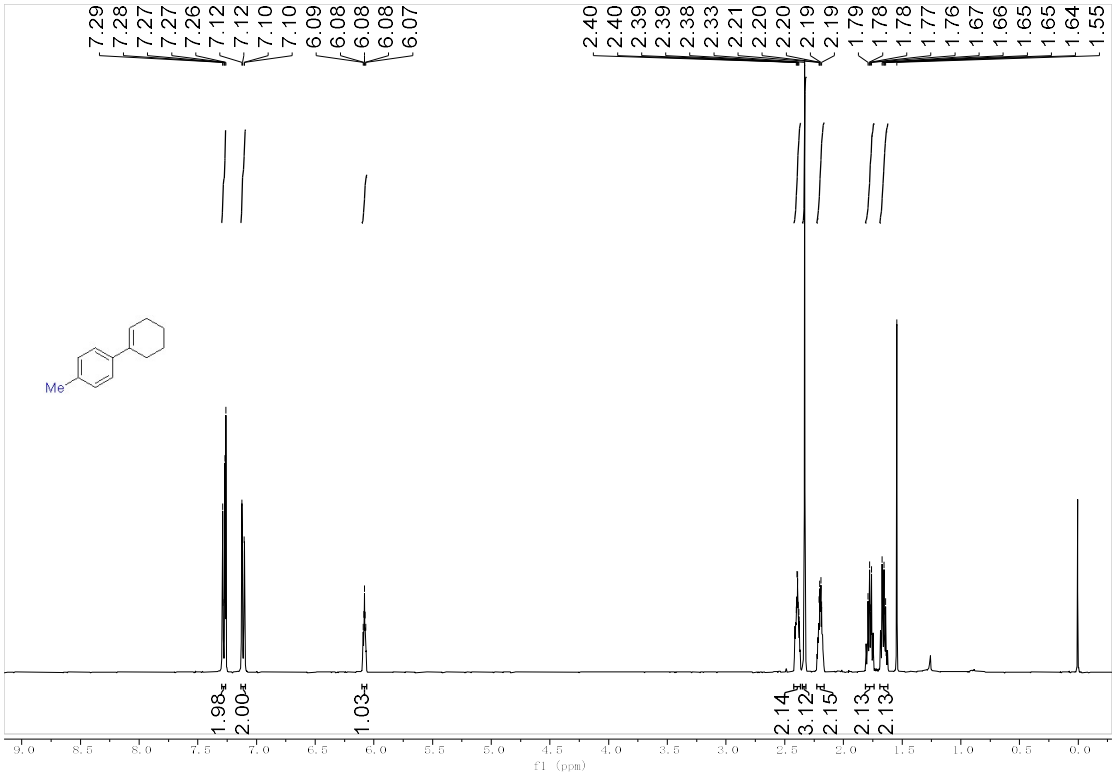
^{1}H NMR of 1f (400 MHz, $CDCl_3$)



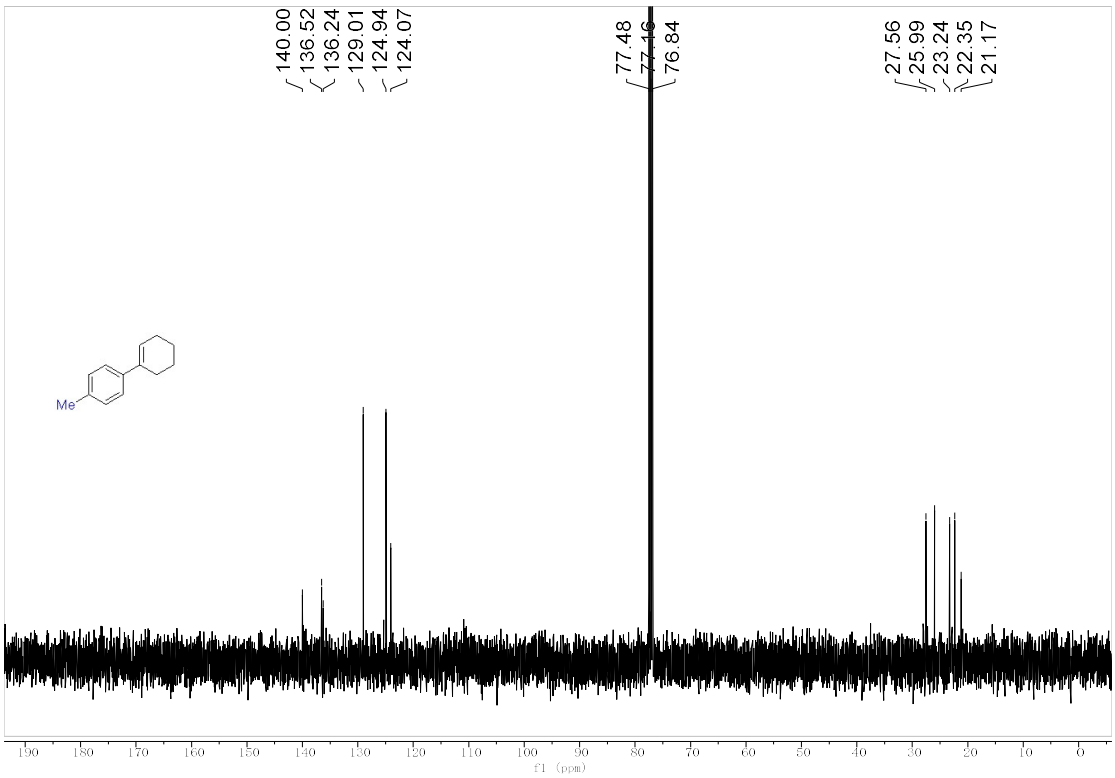
^{13}C NMR of 1f (100 MHz, $CDCl_3$)



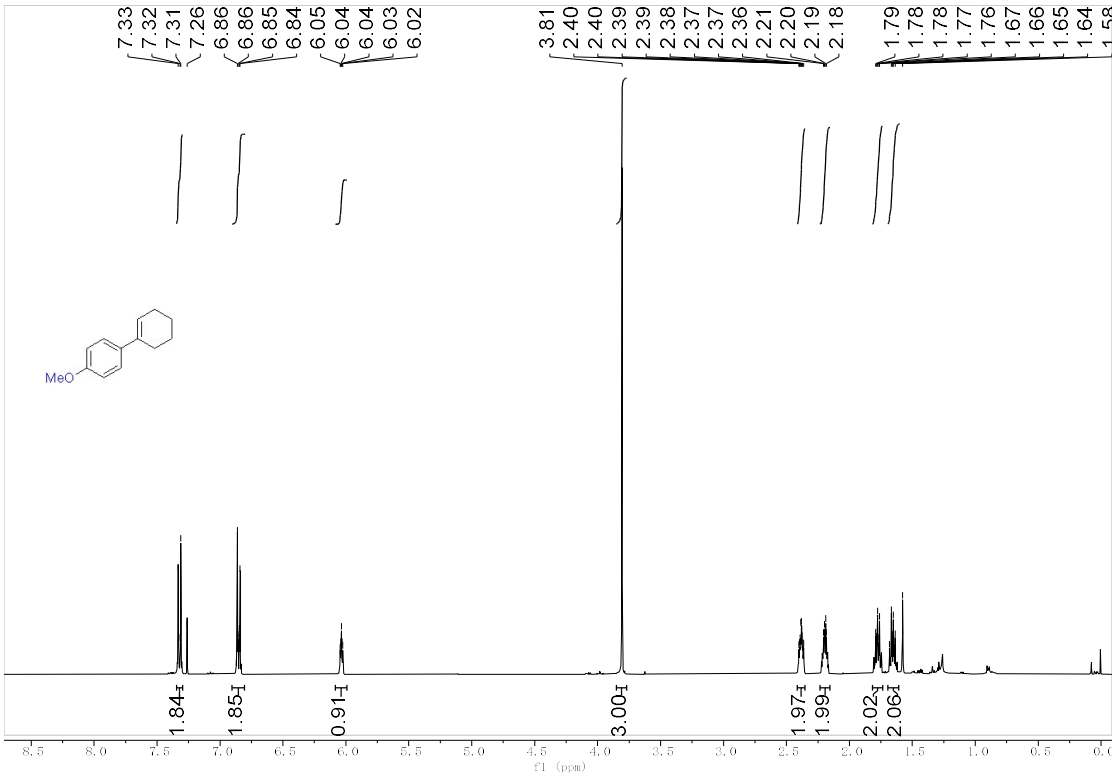
^{1}H NMR of 1g (400 MHz, $CDCl_3$)



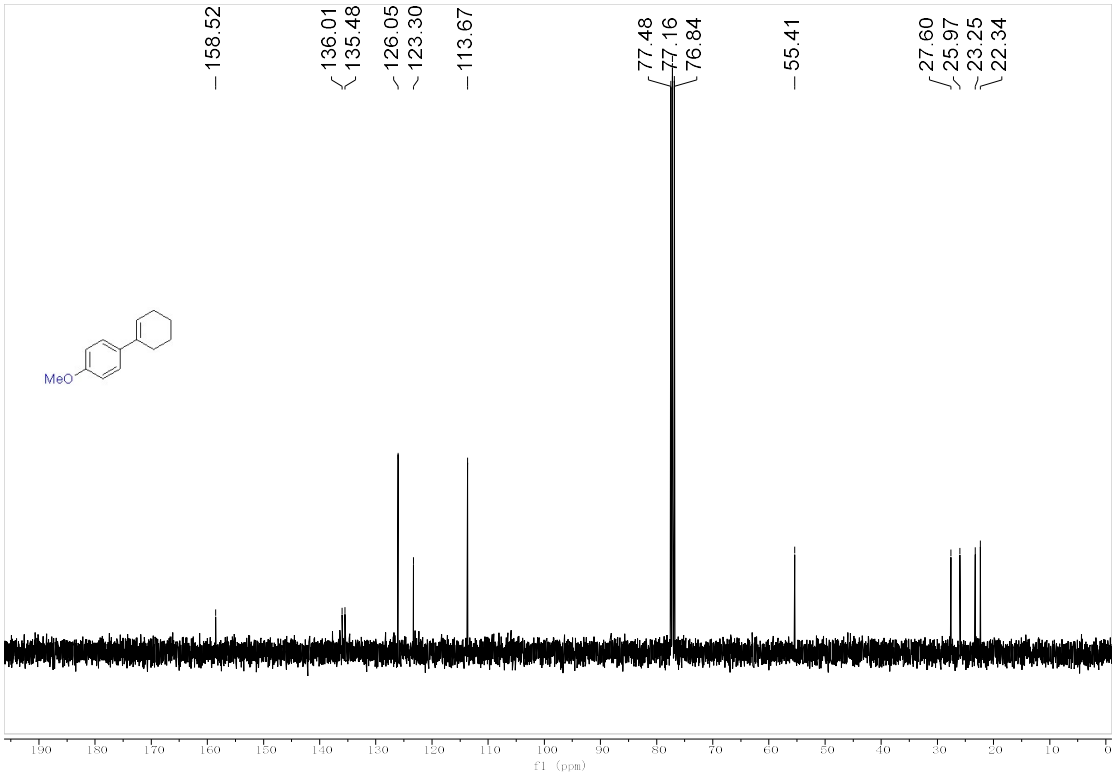
^{13}C NMR of 1g (100 MHz, $CDCl_3$)



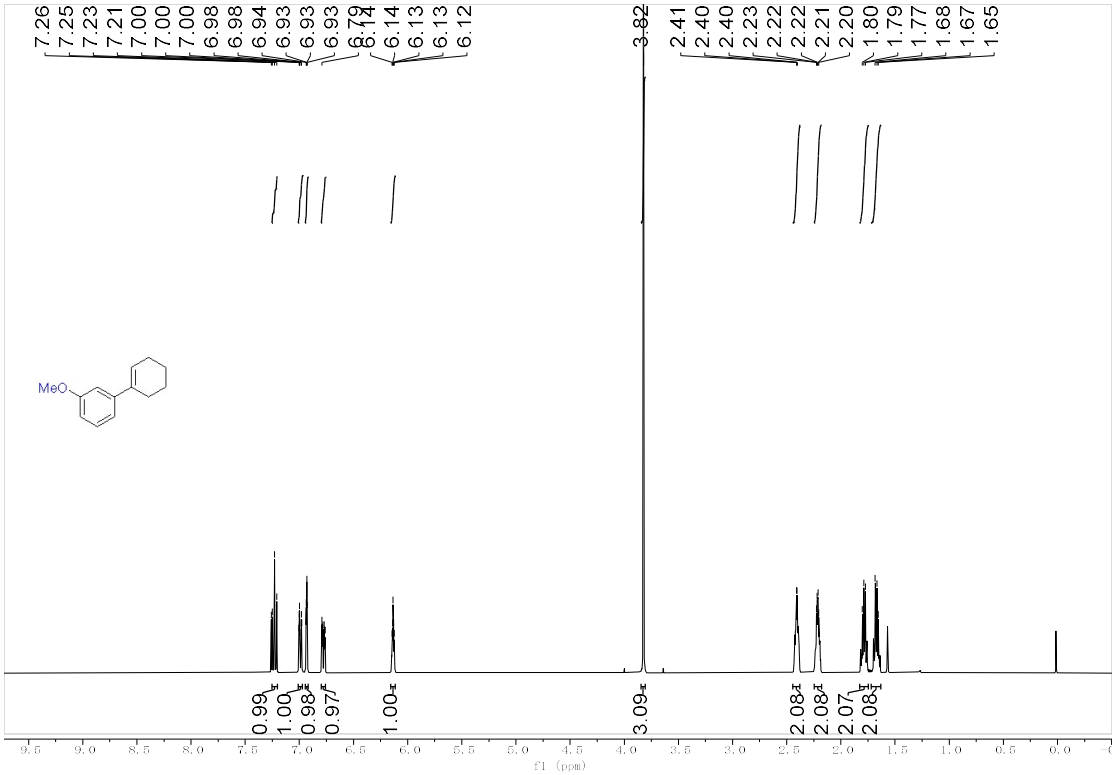
^{1}H NMR of 1h (400 MHz, $CDCl_3$)



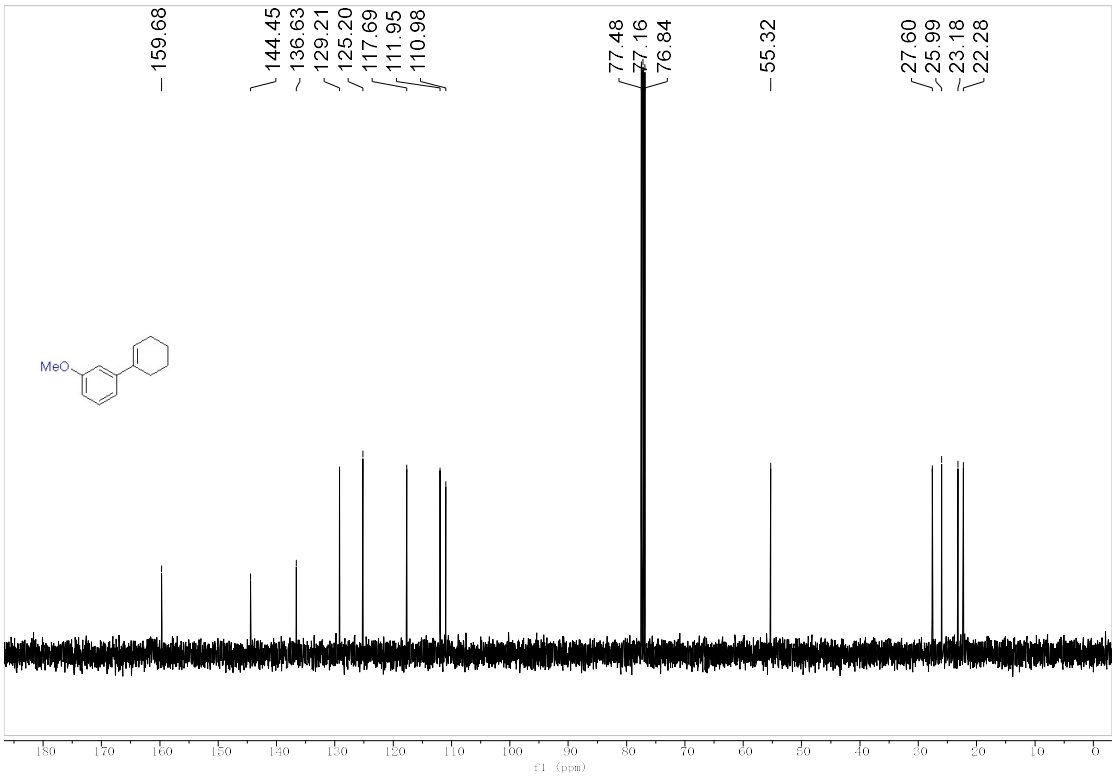
^{13}C NMR of 1h (100 MHz, $CDCl_3$)



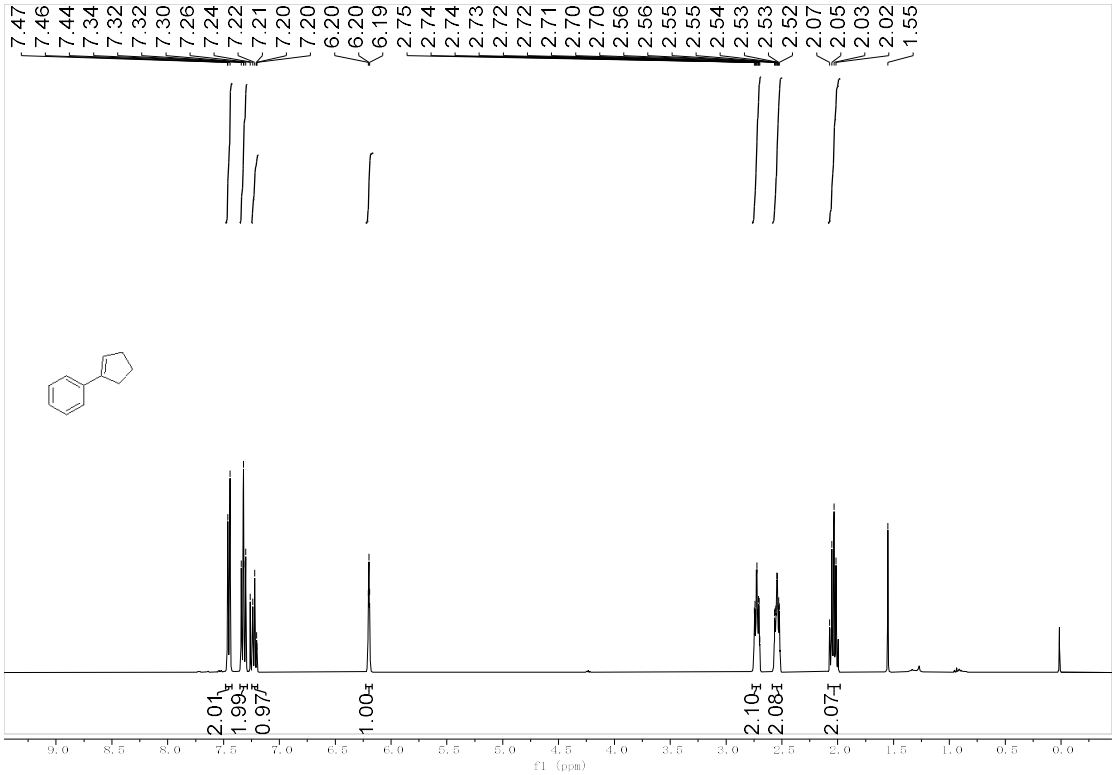
^{1}H NMR of 1i (400 MHz, $CDCl_3$)



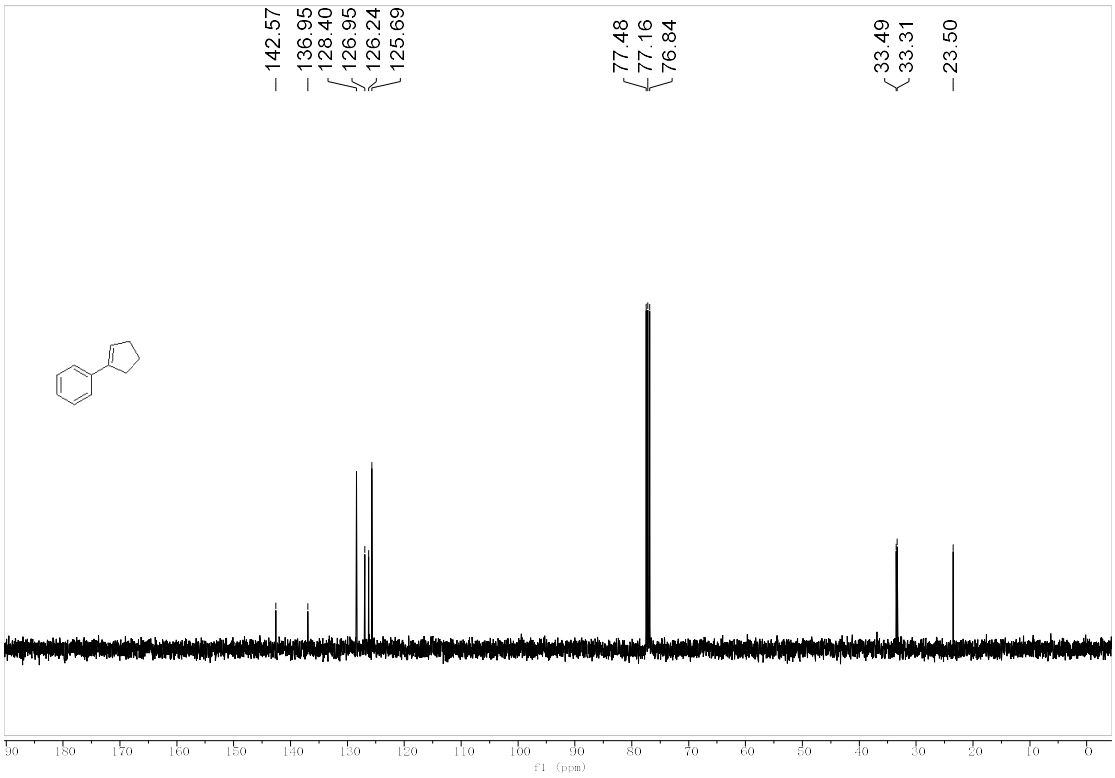
^{13}C NMR of 1i (100 MHz, $CDCl_3$)



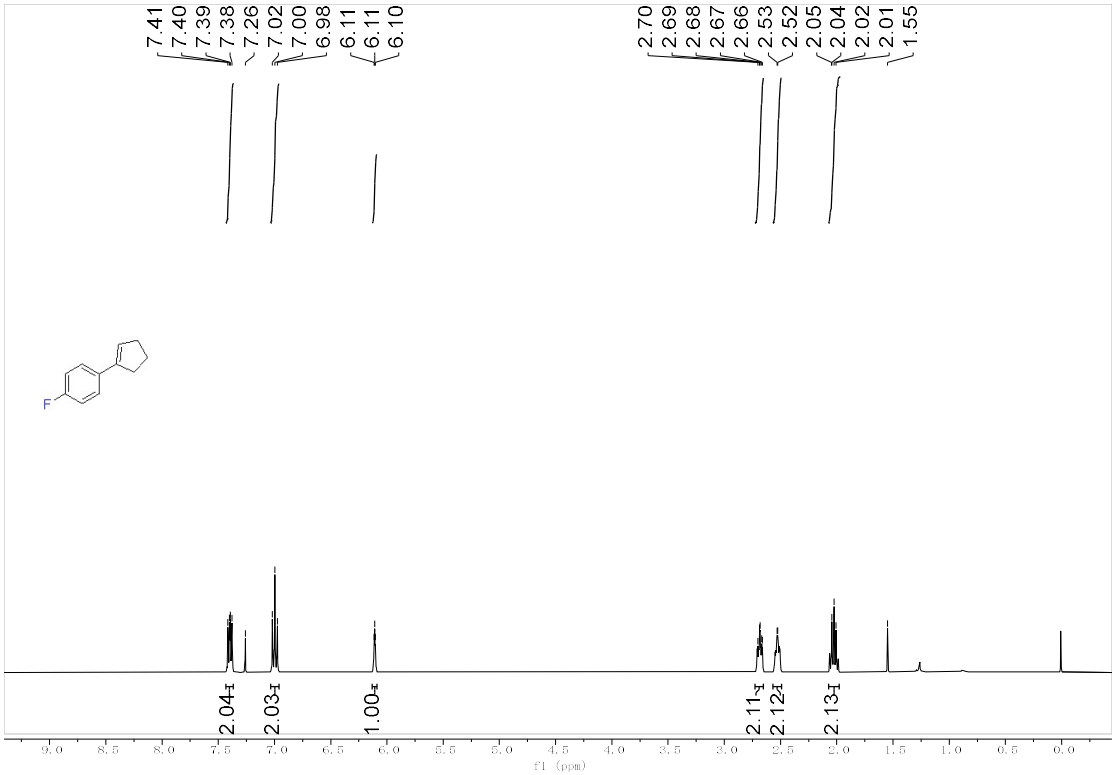
^{1}H NMR of 1j (400 MHz, $CDCl_3$)



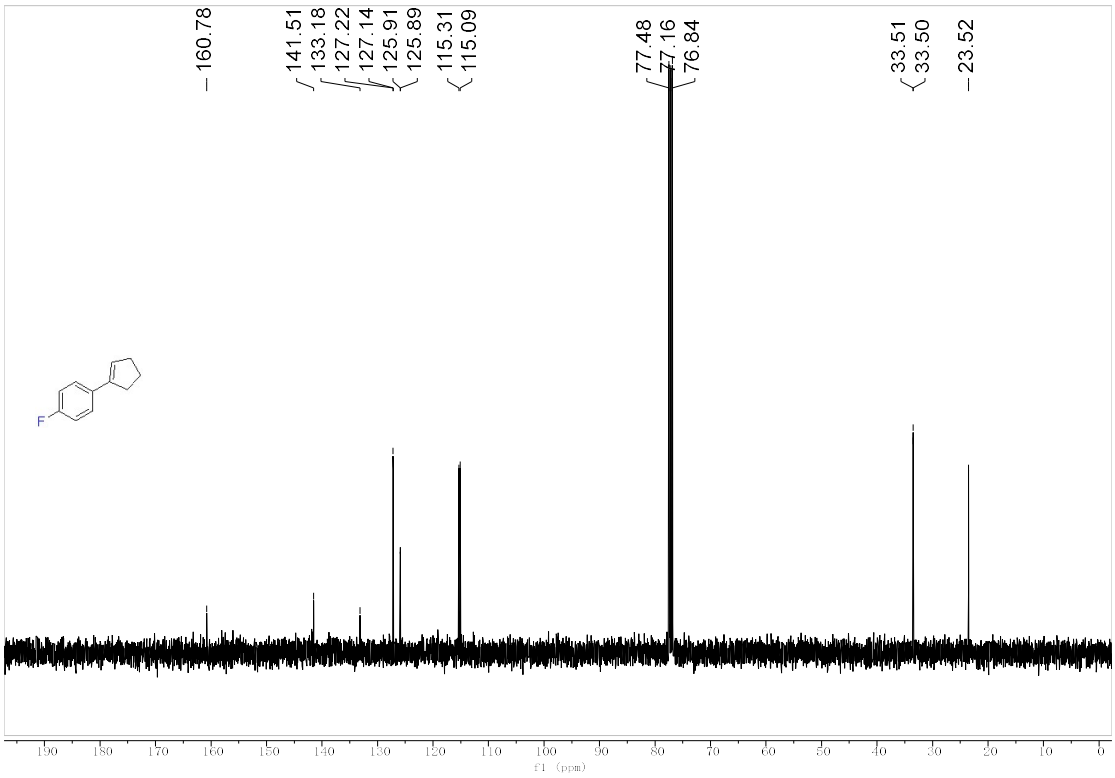
^{13}C NMR of 1j (100 MHz, $CDCl_3$)



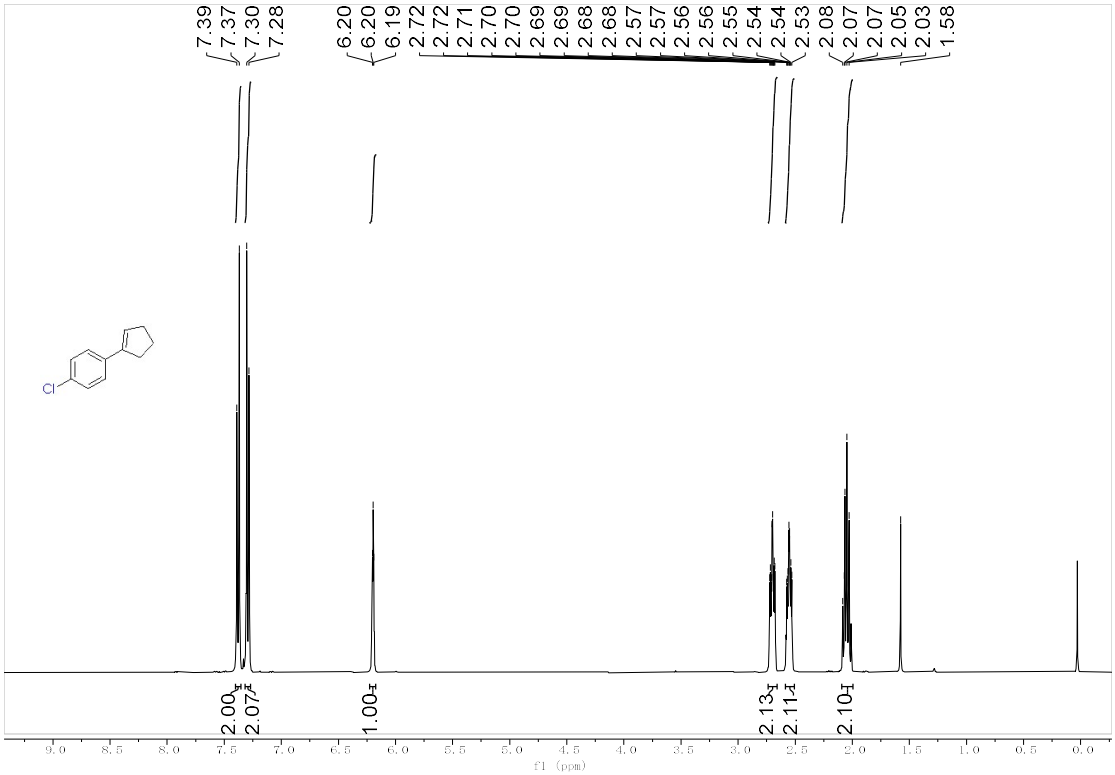
^{1}H NMR of 1k (400 MHz, $CDCl_3$)



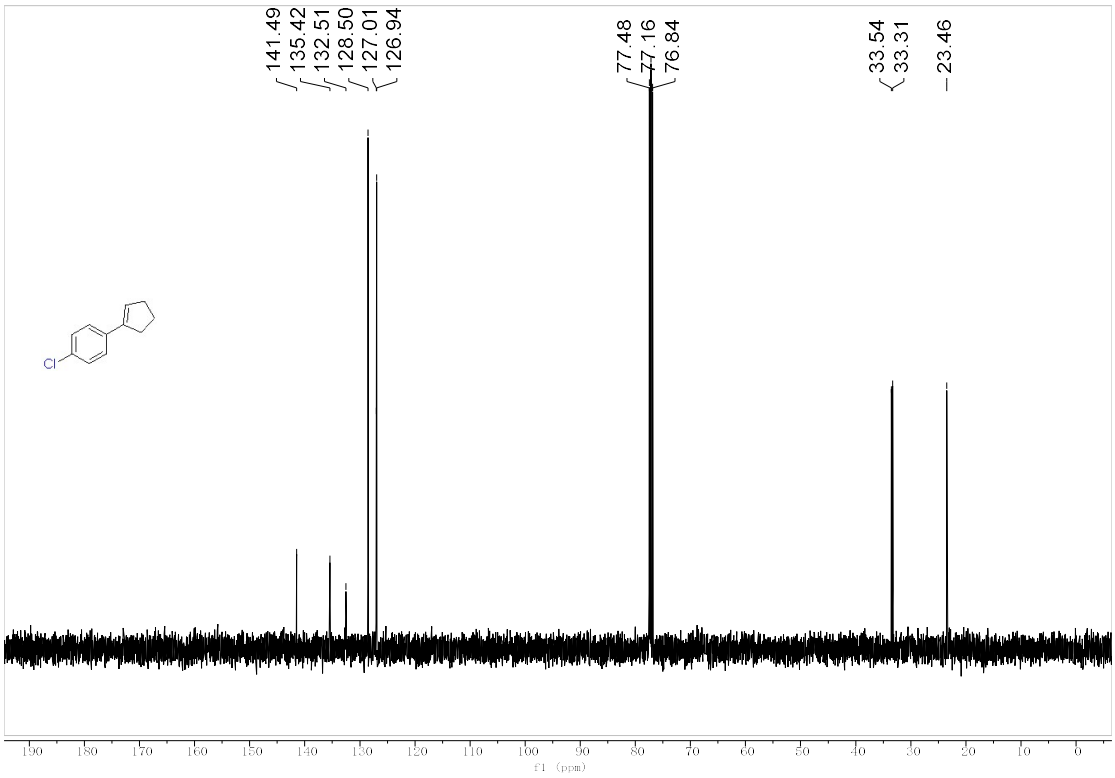
^{13}C NMR of 1k (100 MHz, $CDCl_3$)



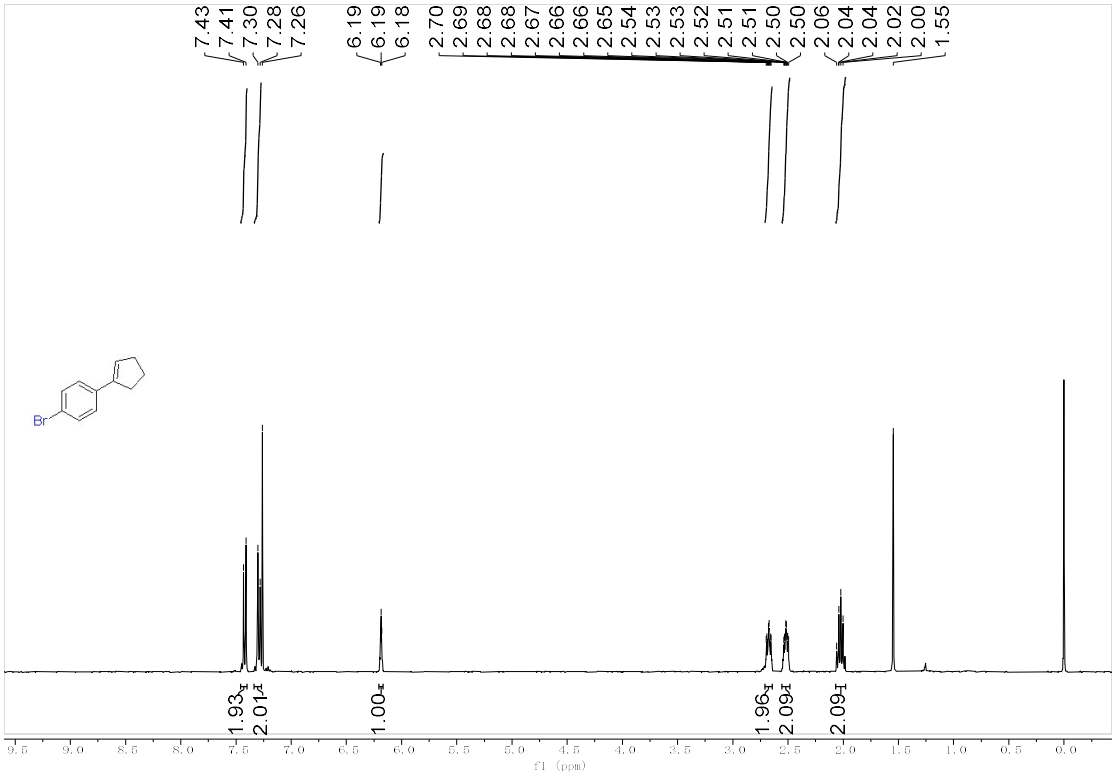
^{1}H NMR of 1l (400 MHz, $CDCl_3$)



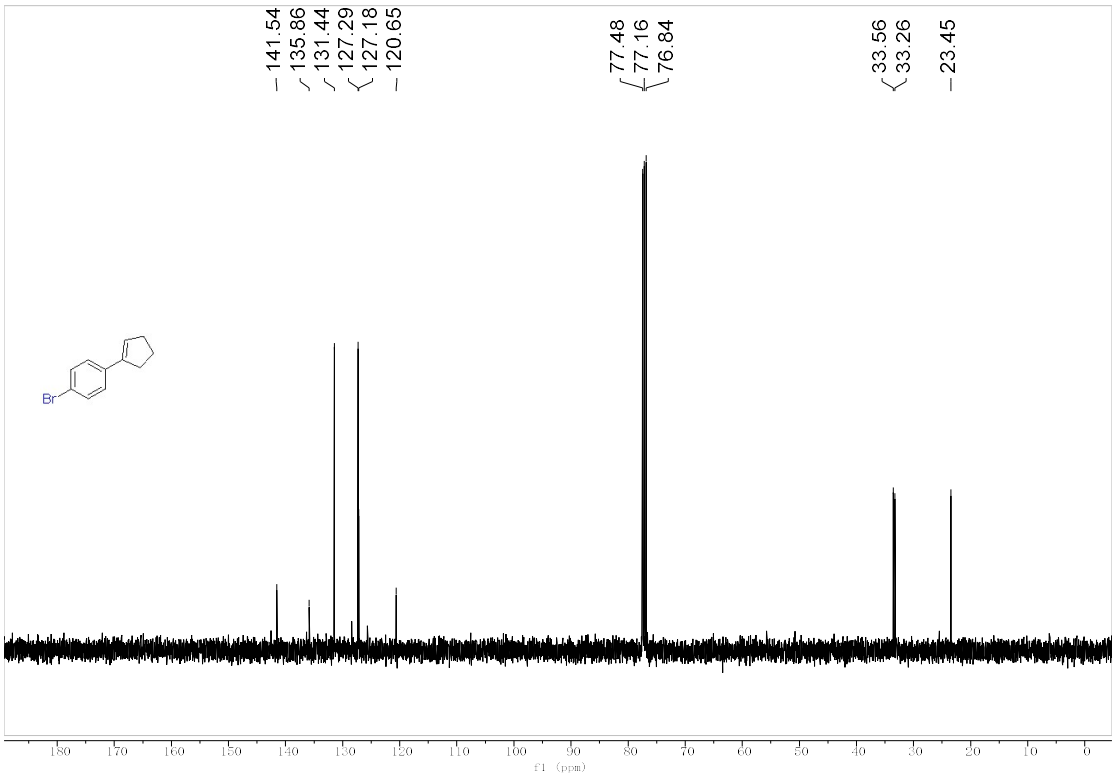
^{13}C NMR of 1l (100 MHz, $CDCl_3$)



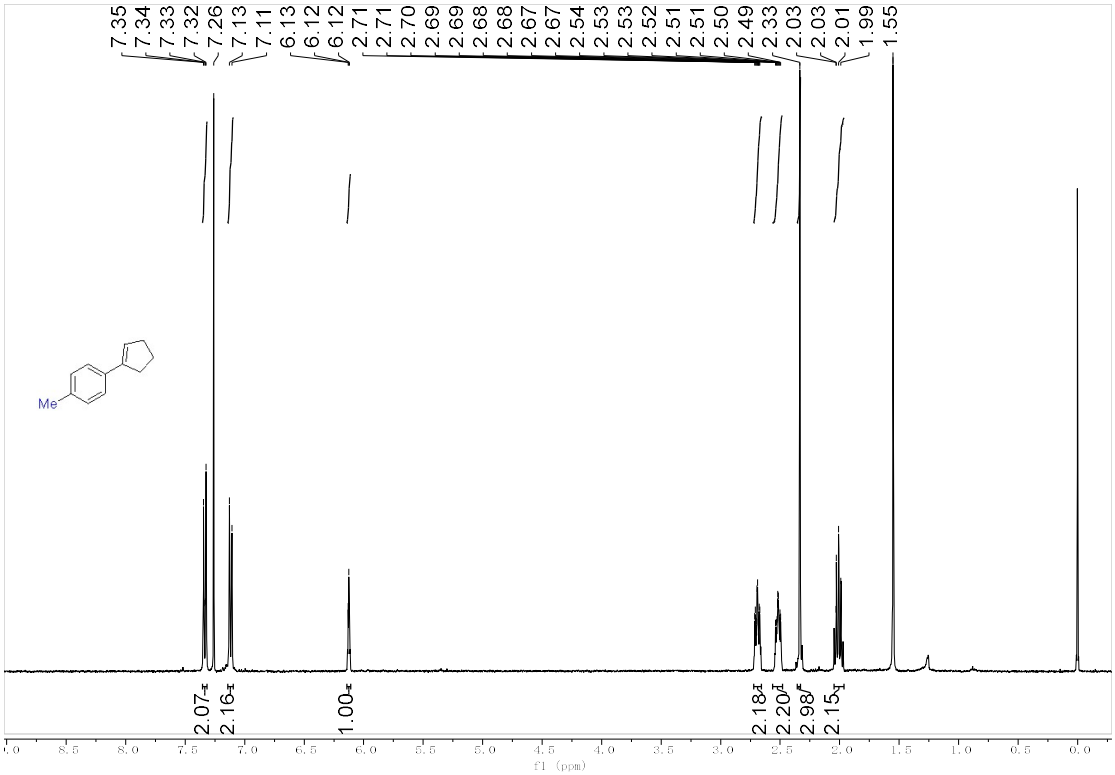
^{1}H NMR of 1m (400 MHz, $CDCl_3$)



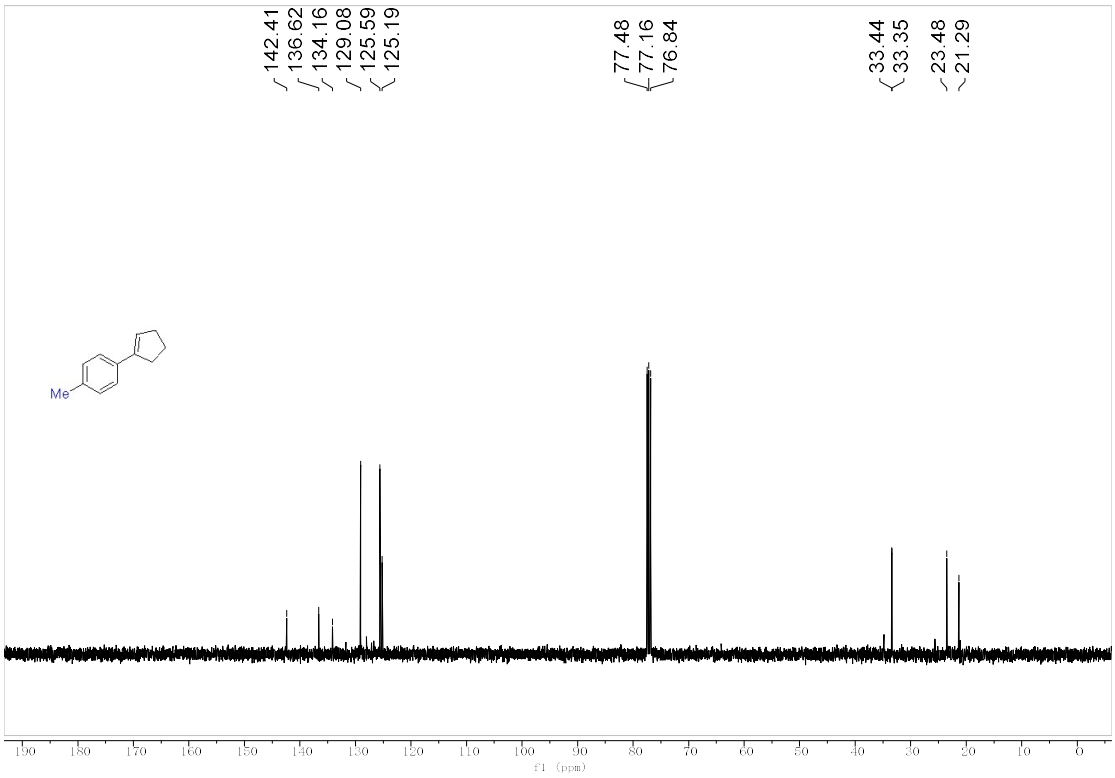
^{13}C NMR of 1m (100 MHz, $CDCl_3$)



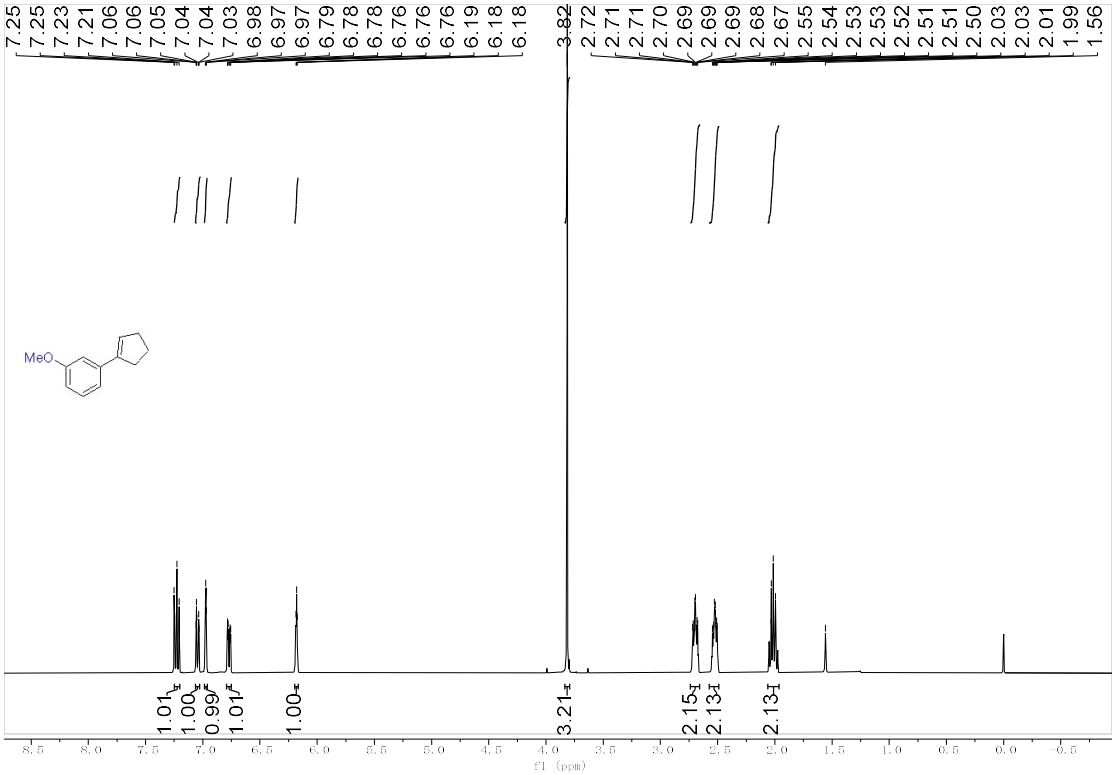
^{1}H NMR of 1n (400 MHz, $CDCl_3$)



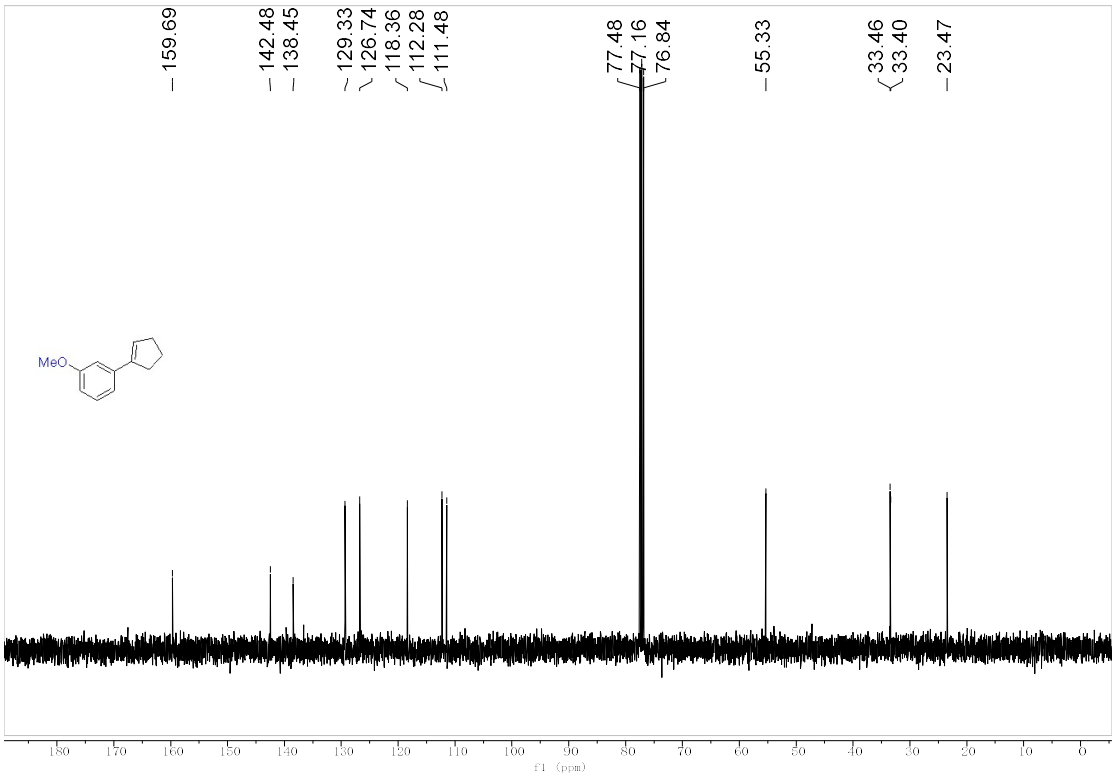
^{13}C NMR of 1n (100 MHz, $CDCl_3$)



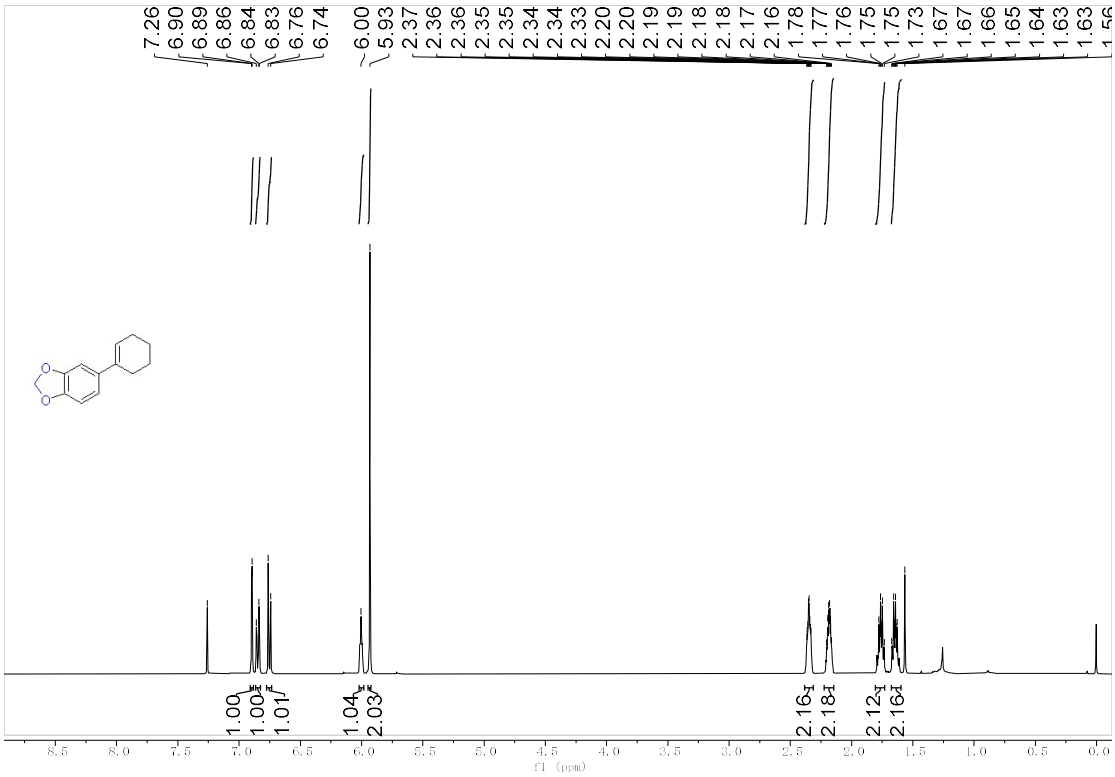
^{1}H NMR of 1o (400 MHz, $CDCl_3$)



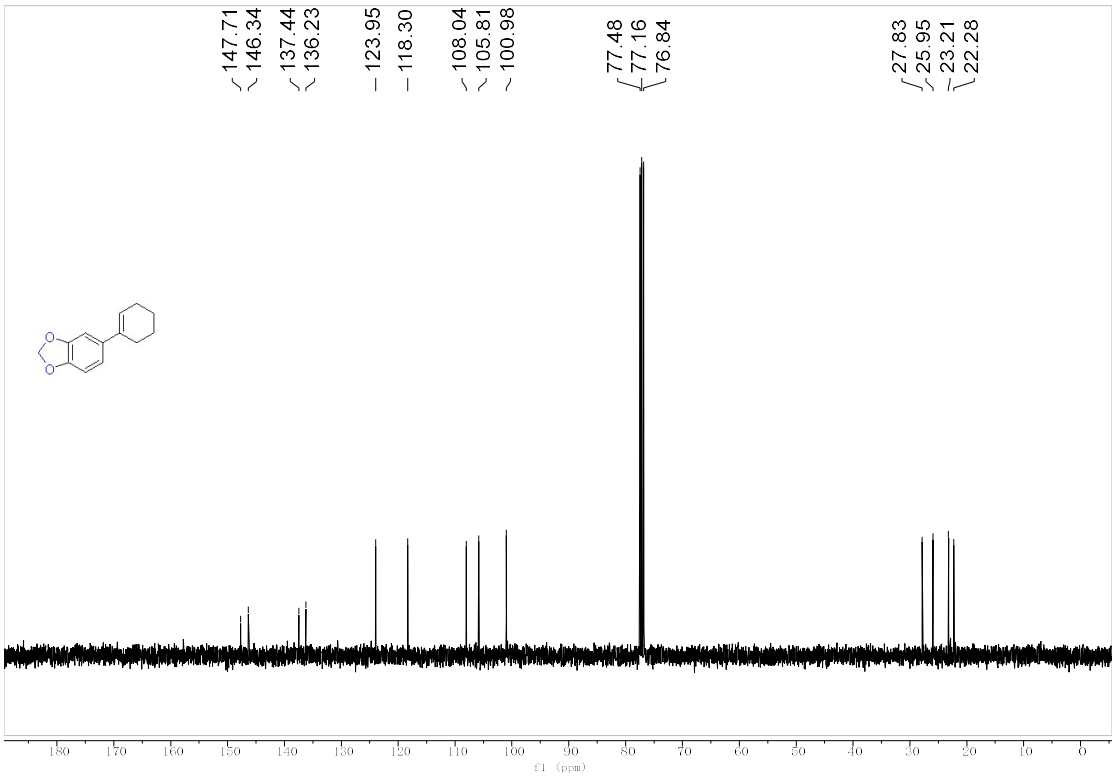
^{13}C NMR of 1o (100 MHz, $CDCl_3$)



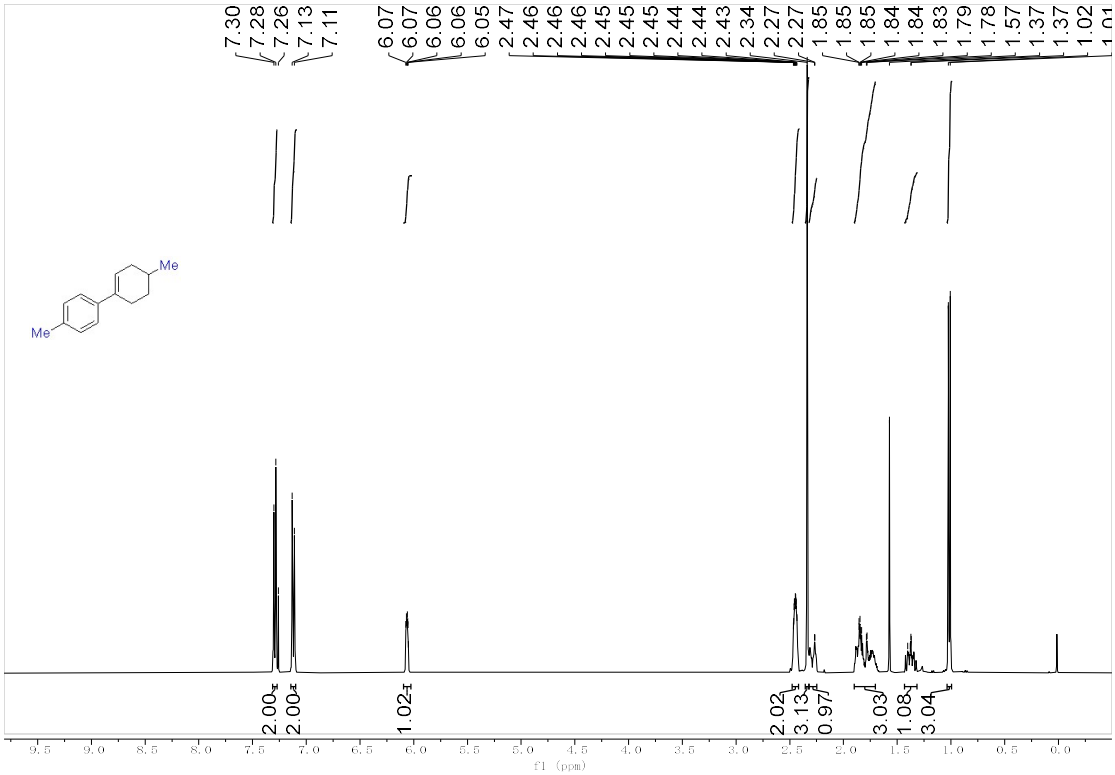
^{1}H NMR of 1p (400 MHz, $CDCl_3$)



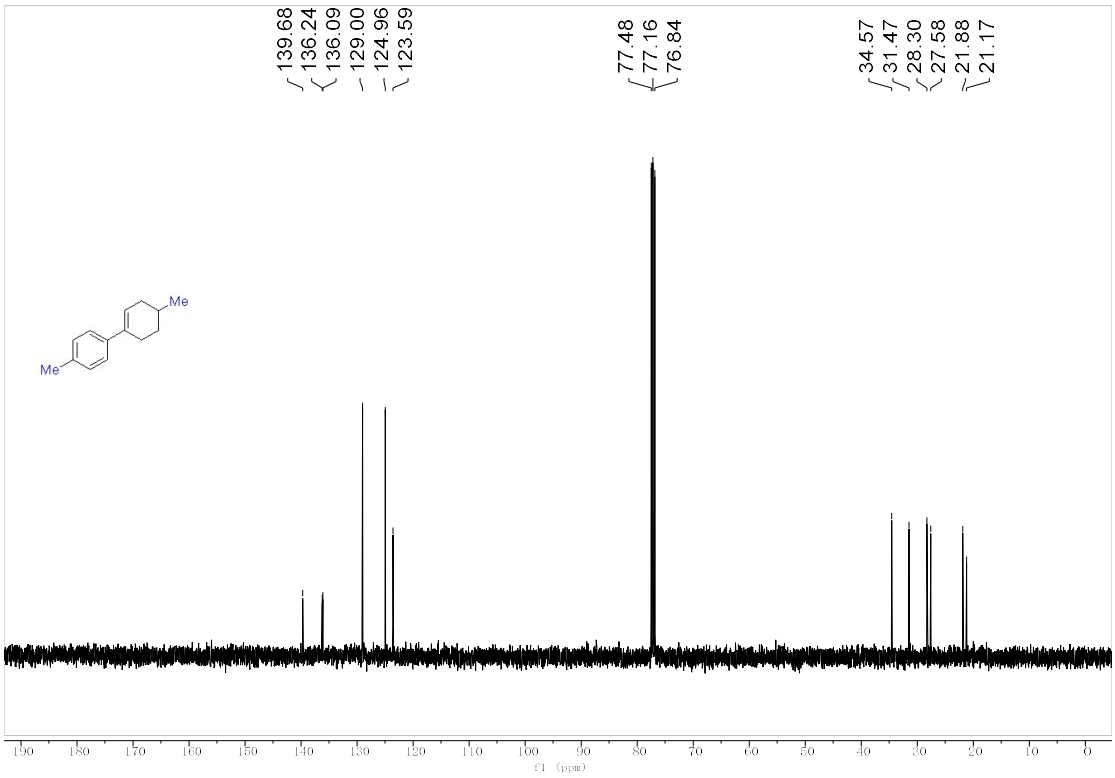
^{13}C NMR of 1p (100 MHz, $CDCl_3$)



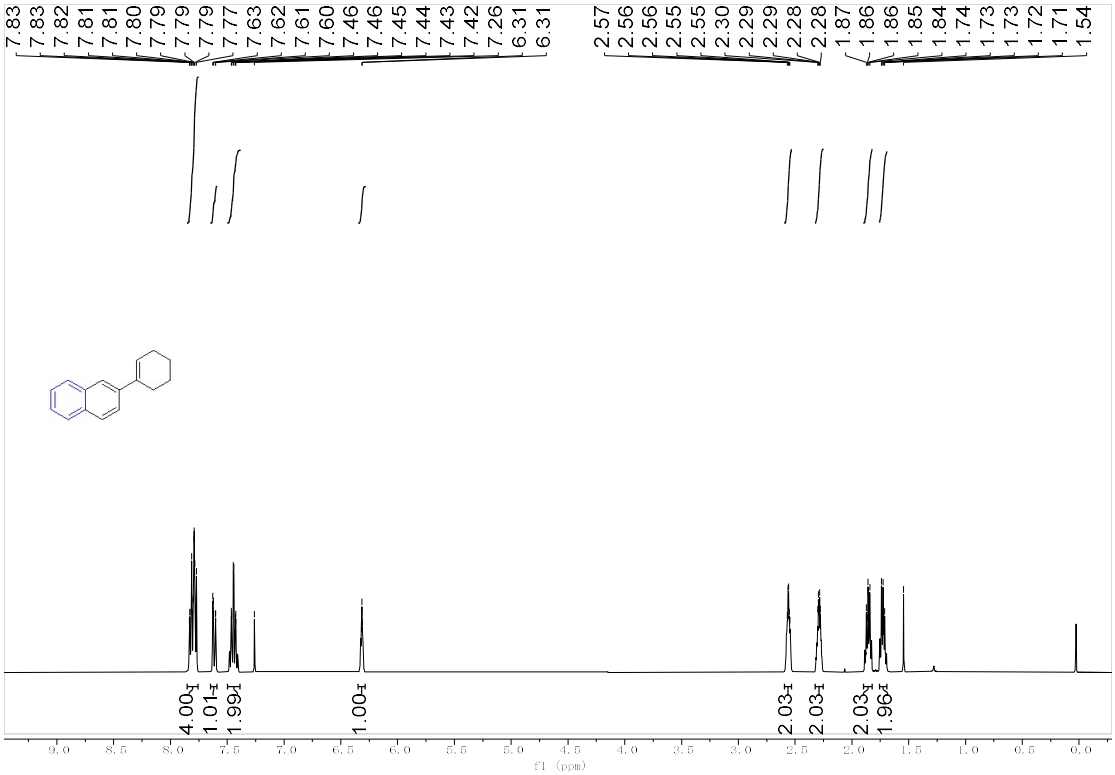
^{1}H NMR of 1q (400 MHz, $CDCl_3$)



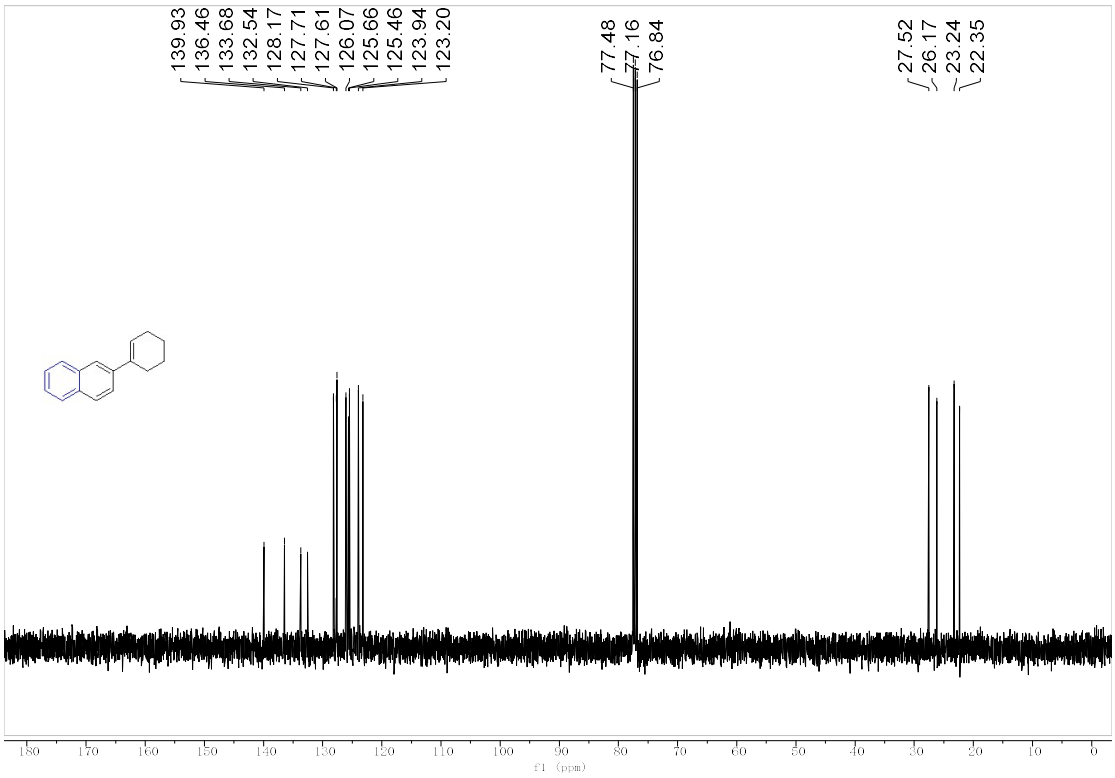
^{13}C NMR of 1q (100 MHz, $CDCl_3$)



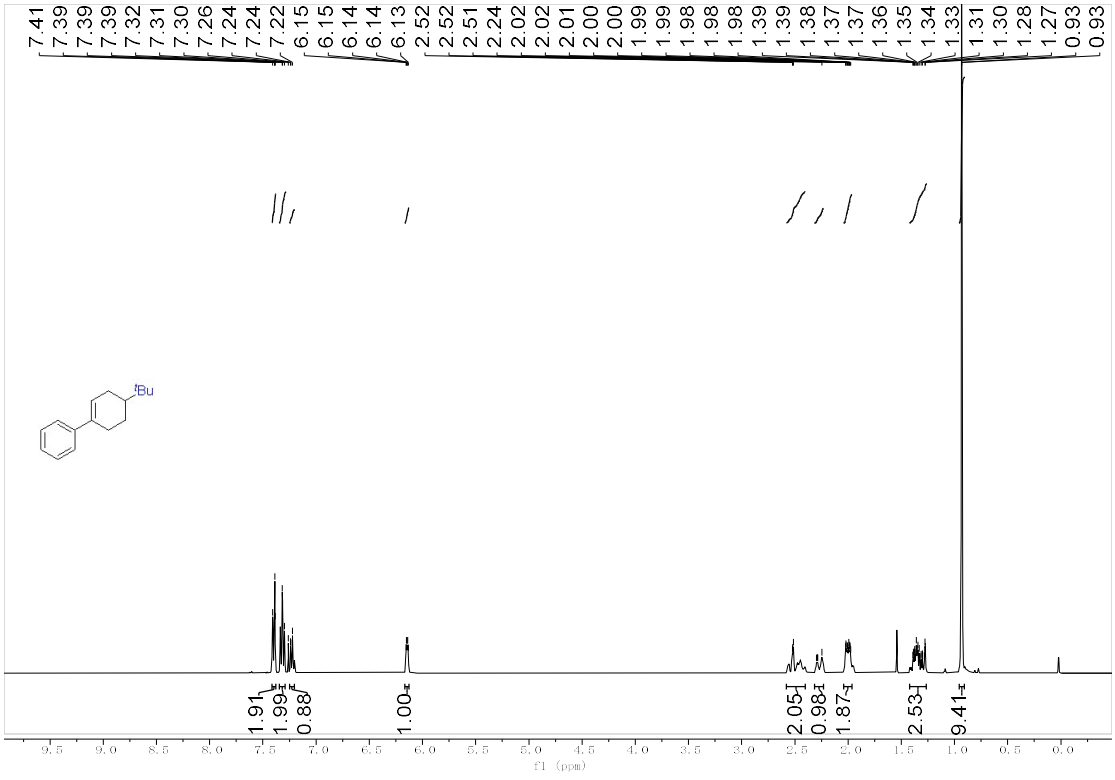
^{1}H NMR of 1r (400 MHz, $CDCl_3$)



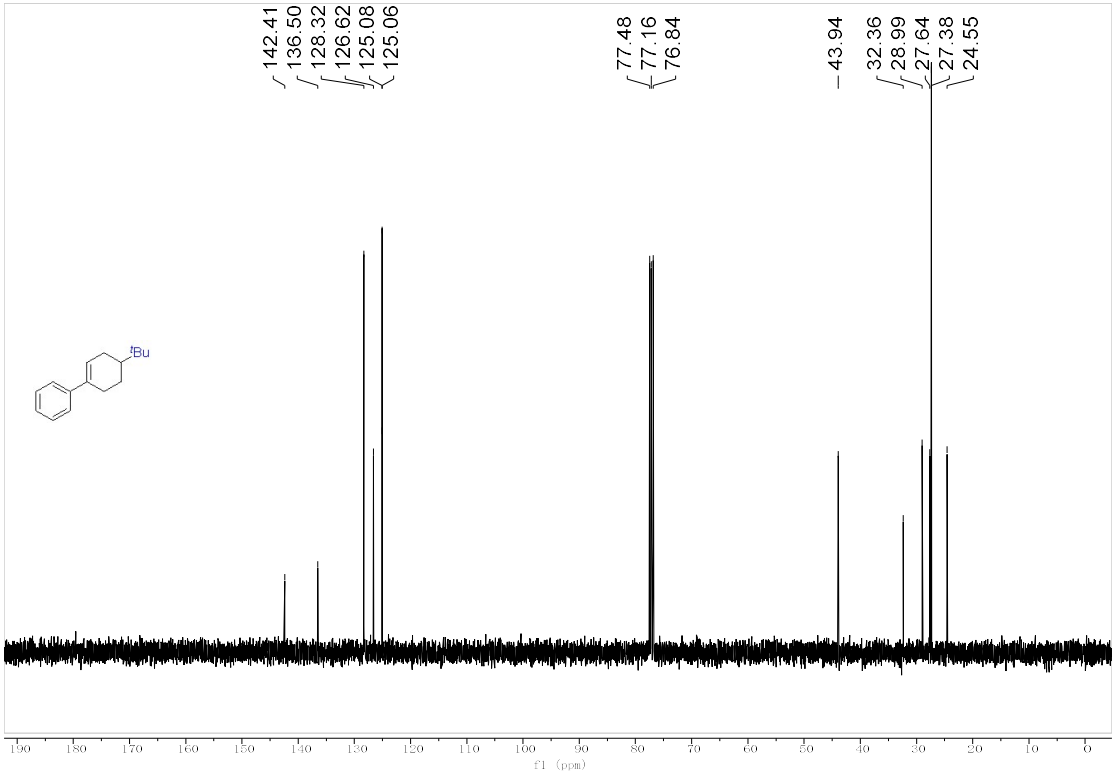
^{13}C NMR of 1r (100 MHz, $CDCl_3$)



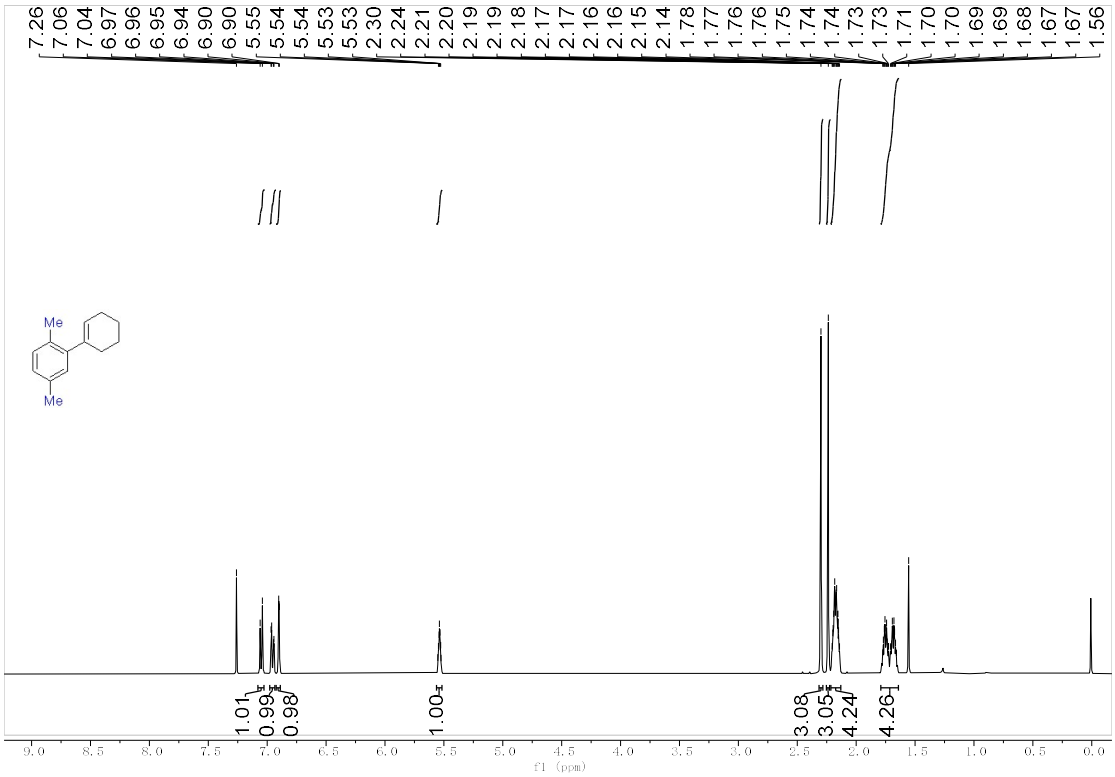
^{1}H NMR of 1s (400 MHz, $CDCl_3$)



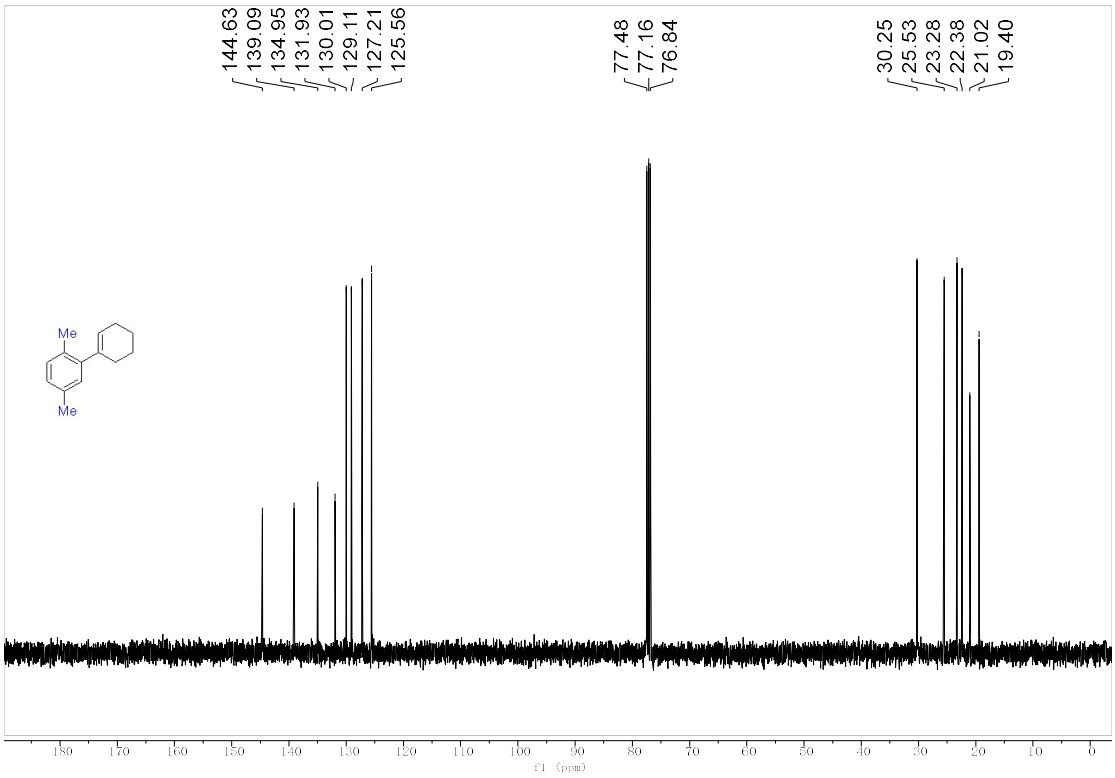
^{13}C NMR of 1s (100 MHz, $CDCl_3$)



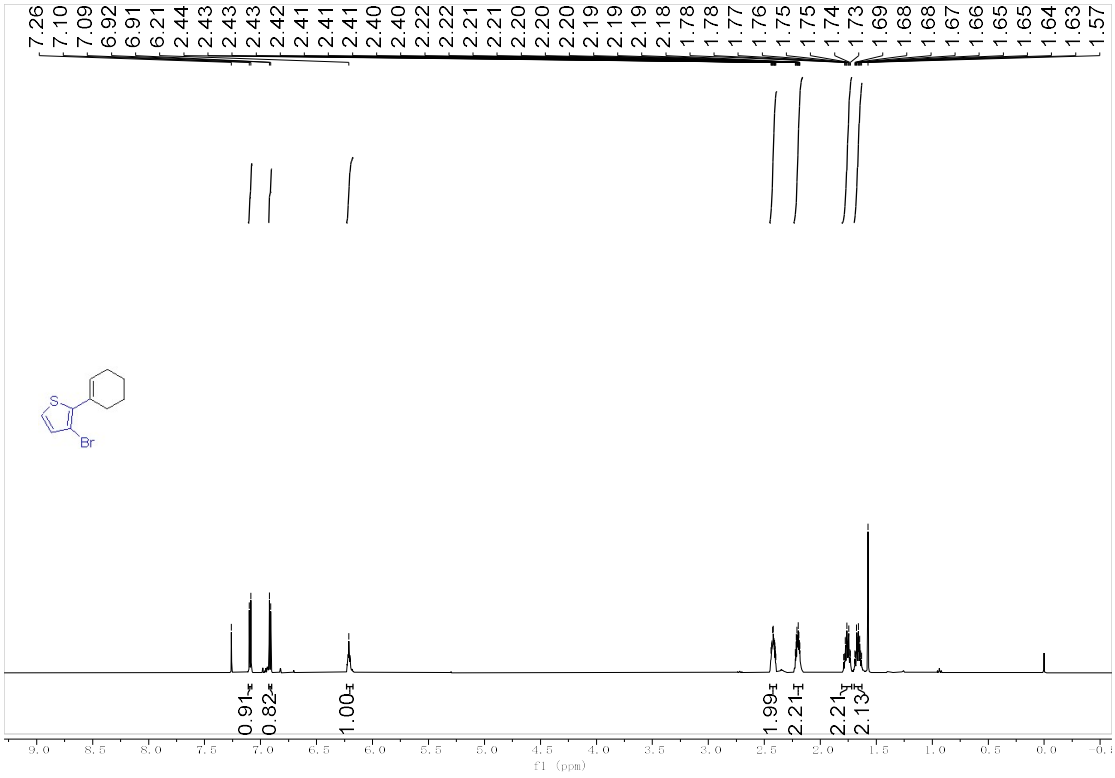
^{1}H NMR of 1t (400 MHz, $CDCl_3$)



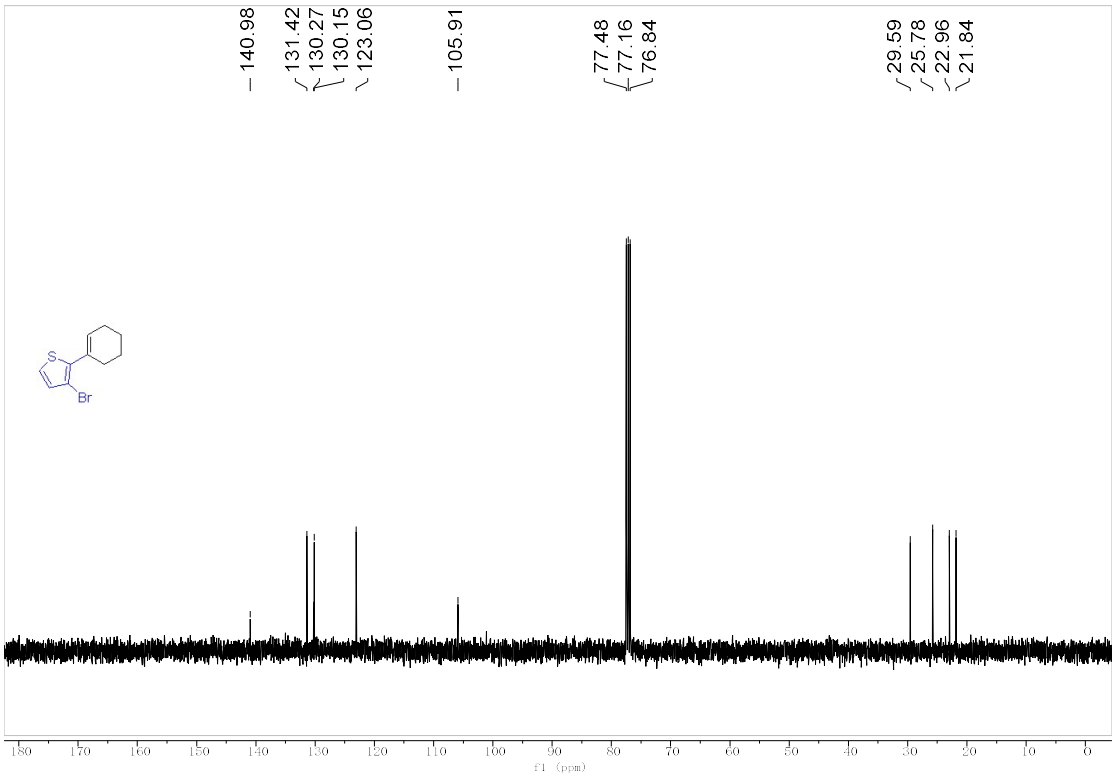
^{13}C NMR of 1t (100 MHz, $CDCl_3$)



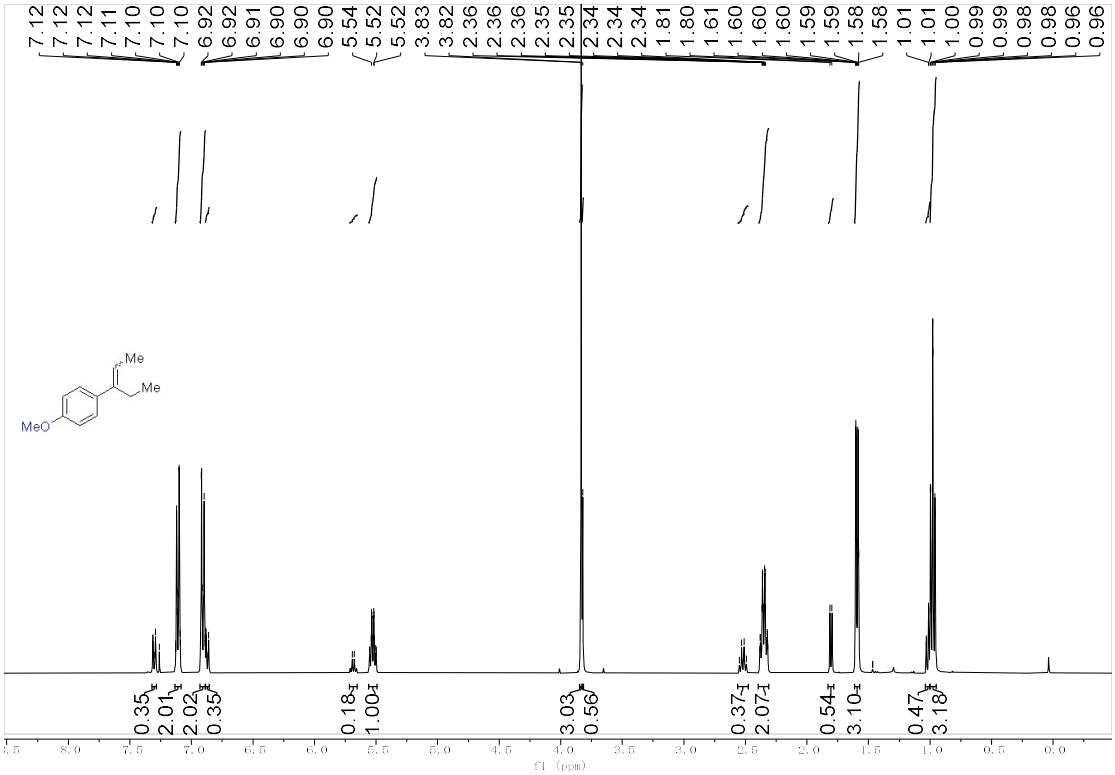
^{1}H NMR of 1u (400 MHz, $CDCl_3$)



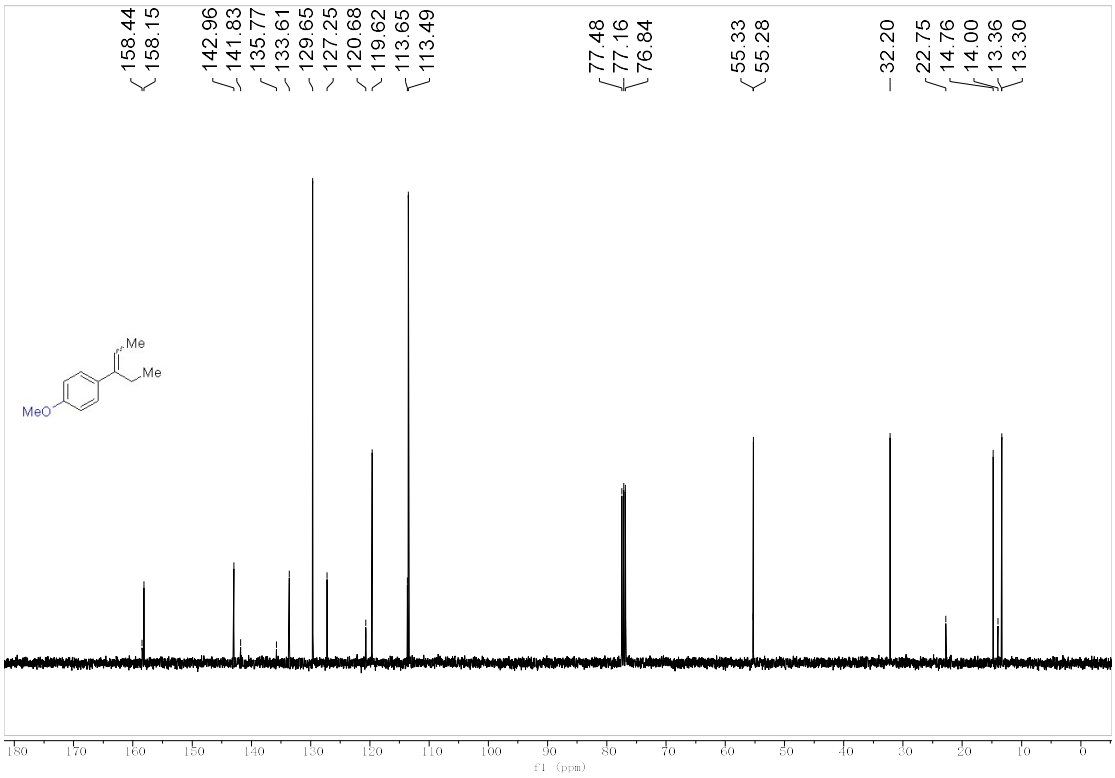
^{13}C NMR of 1u (100 MHz, $CDCl_3$)



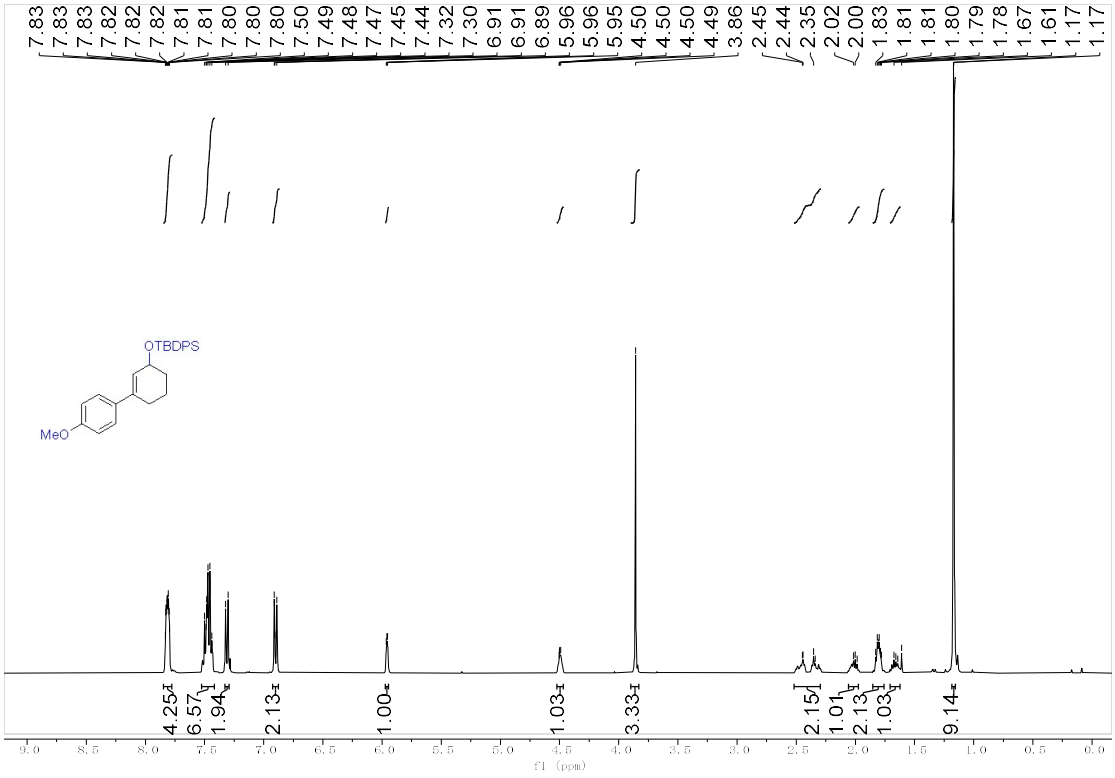
^{1}H NMR of 1v (400 MHz, $CDCl_3$)



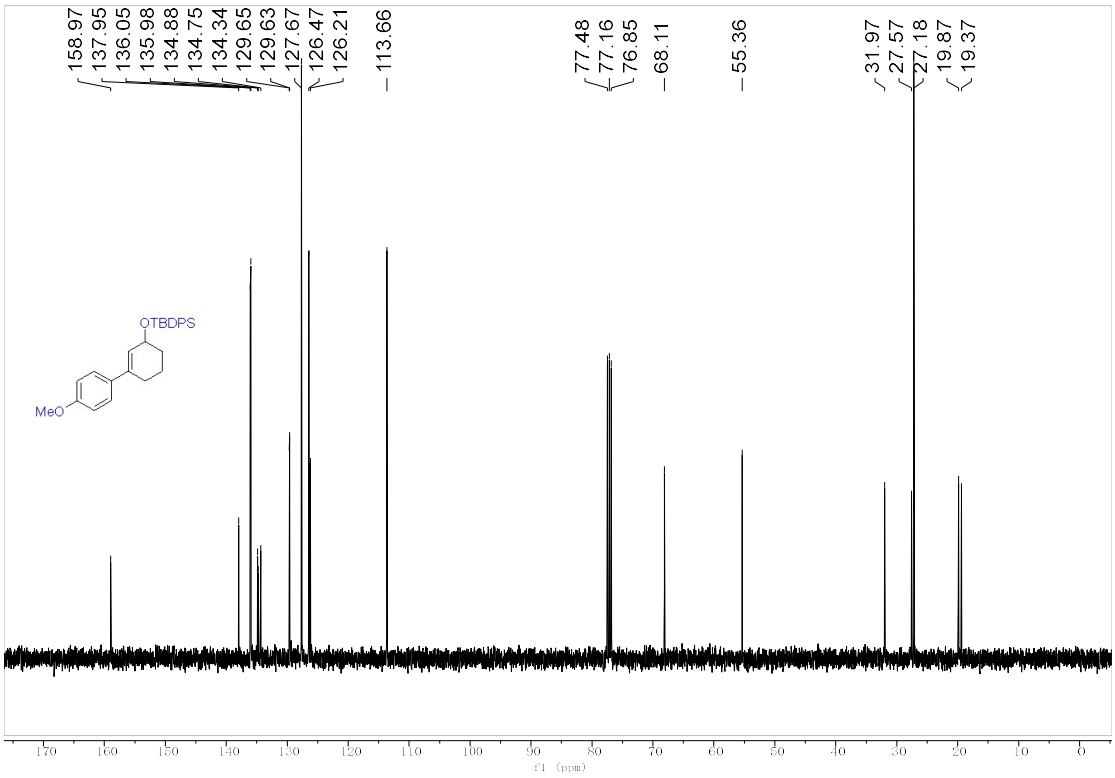
^{13}C NMR of 1v (100 MHz, $CDCl_3$)



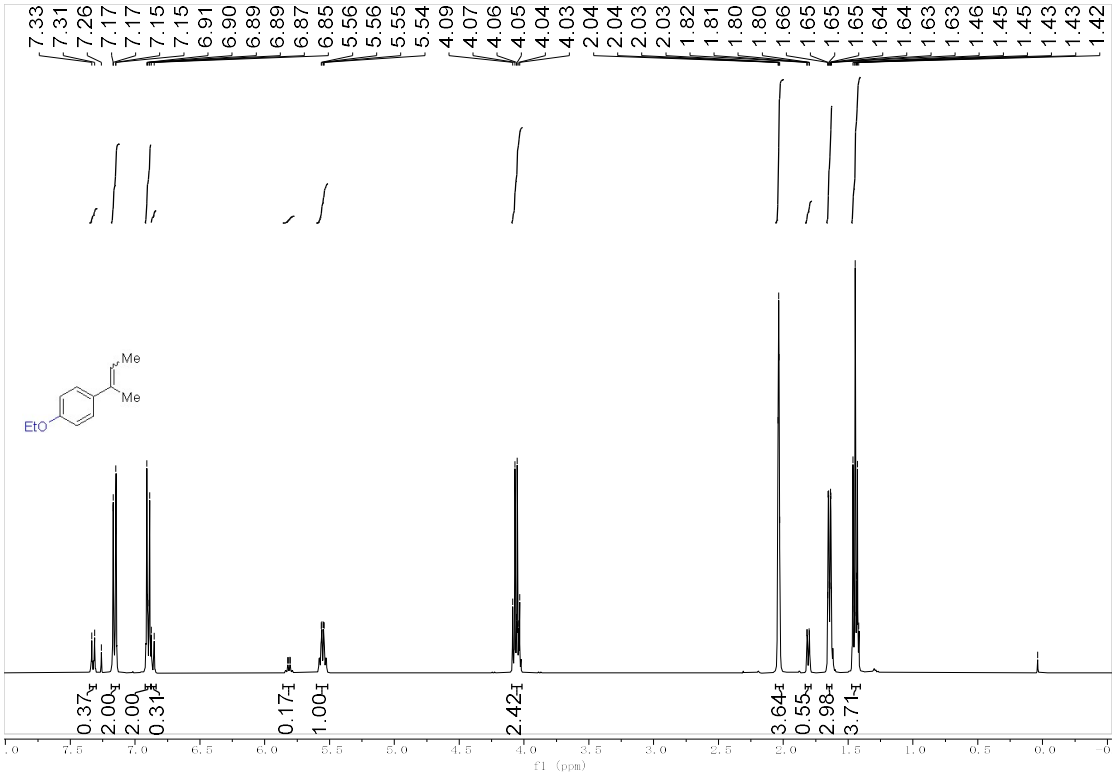
^{1}H NMR of 1w (400 MHz, $CDCl_3$)



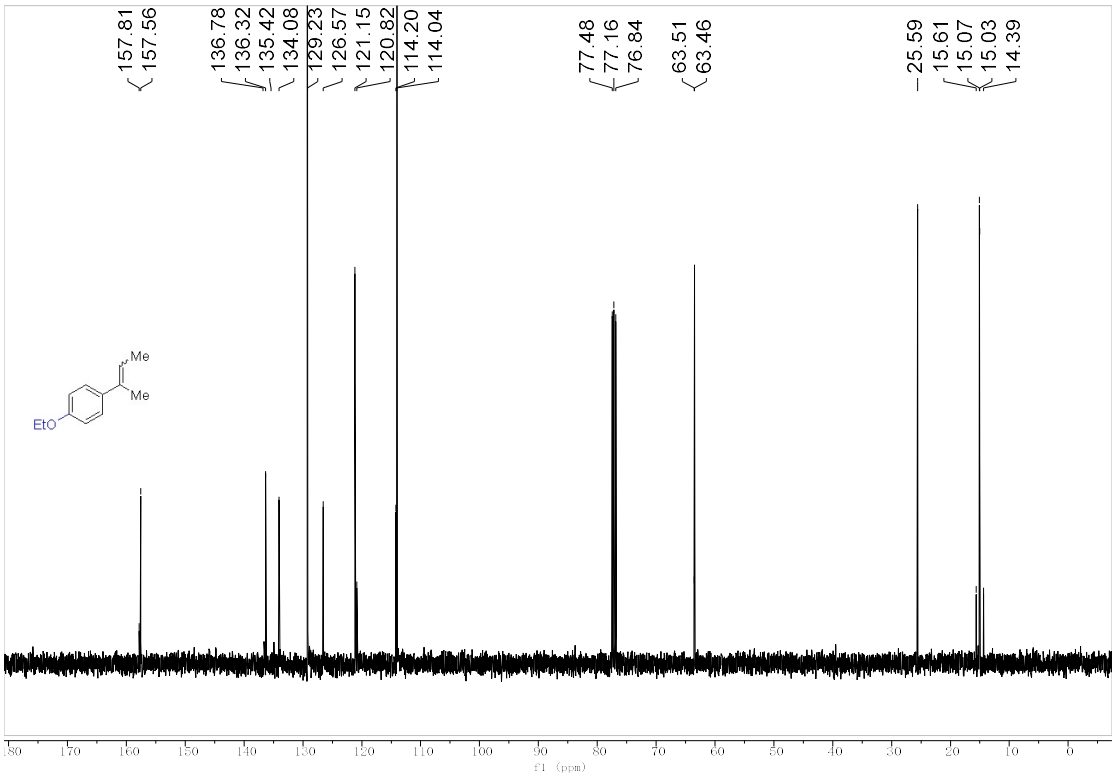
^{13}C NMR of 1w (100 MHz, $CDCl_3$)



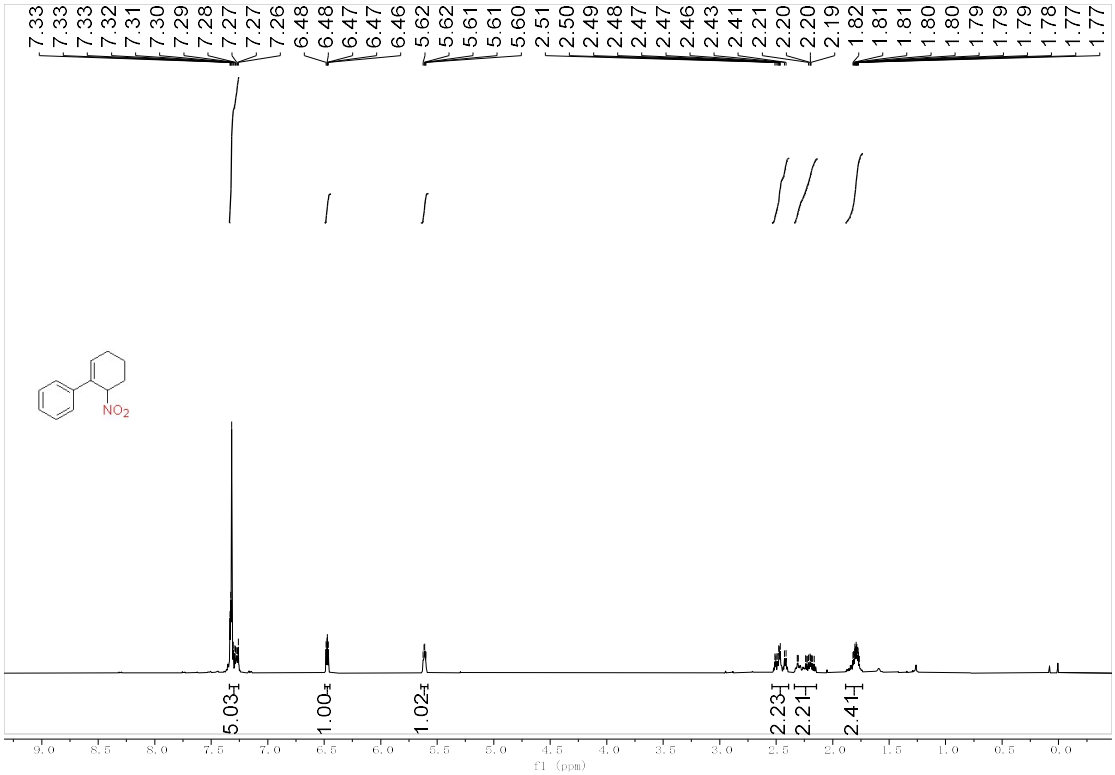
^{1}H NMR of 1x (400 MHz, $CDCl_3$)



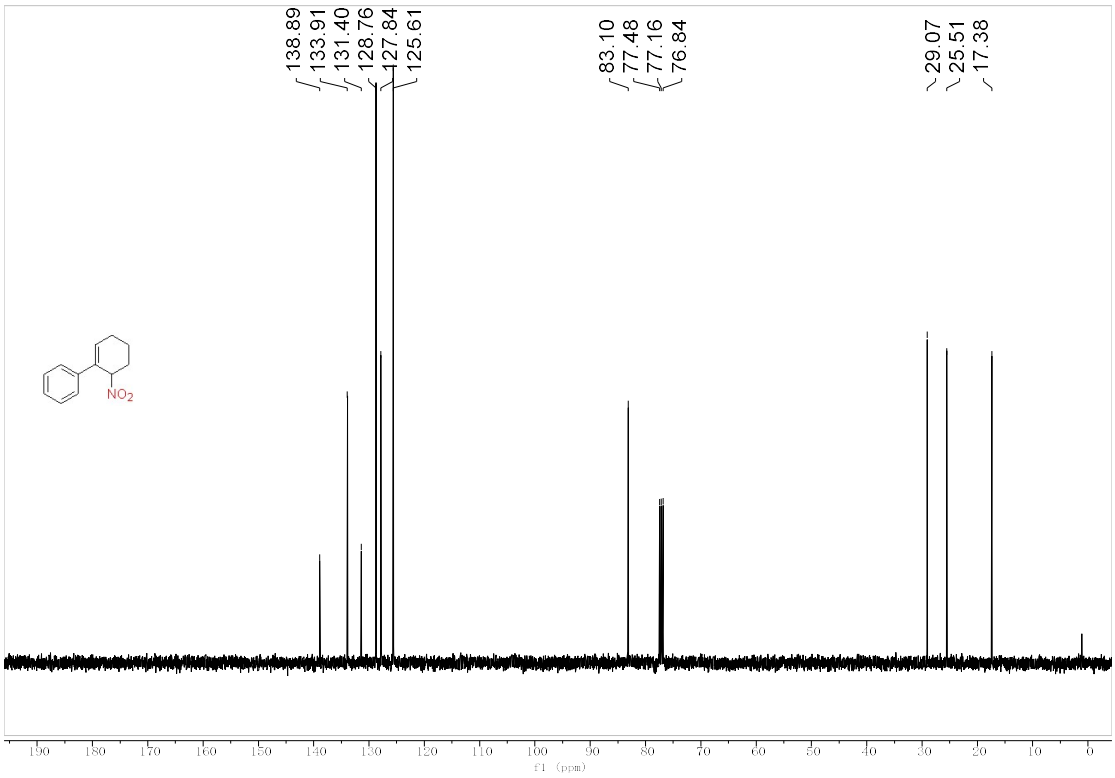
^{13}C NMR of 1x (100 MHz, $CDCl_3$)



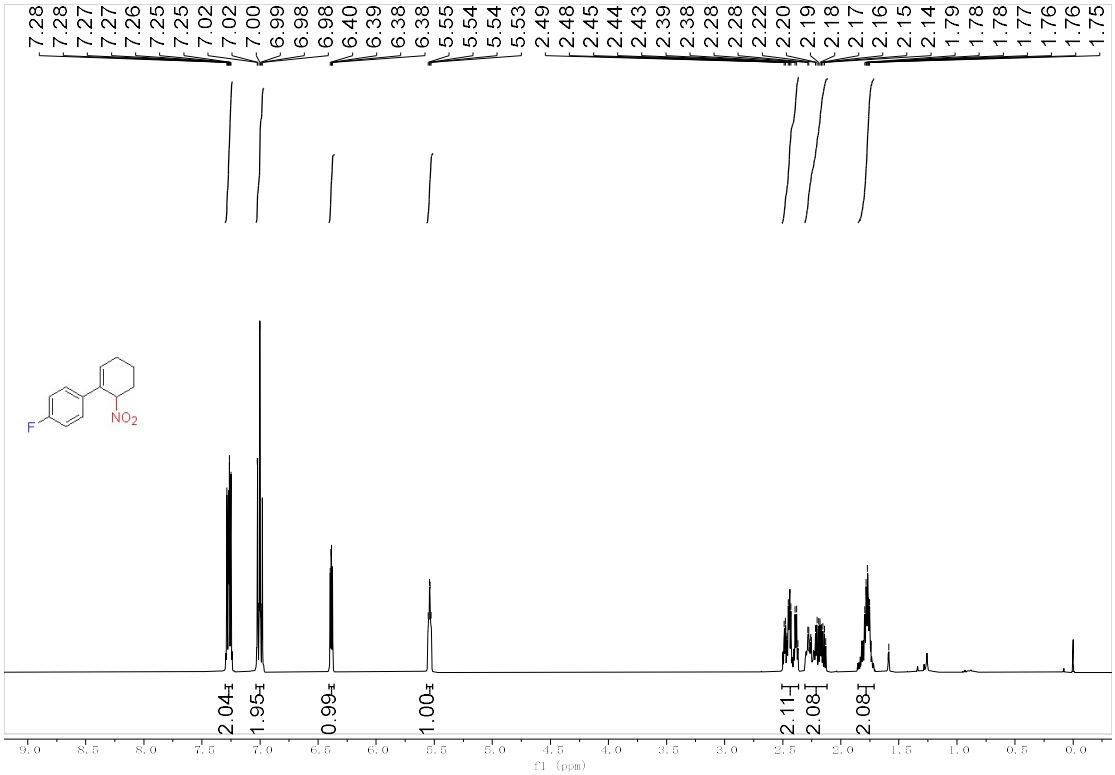
^{1}H NMR of 3a (400 MHz, $CDCl_3$)



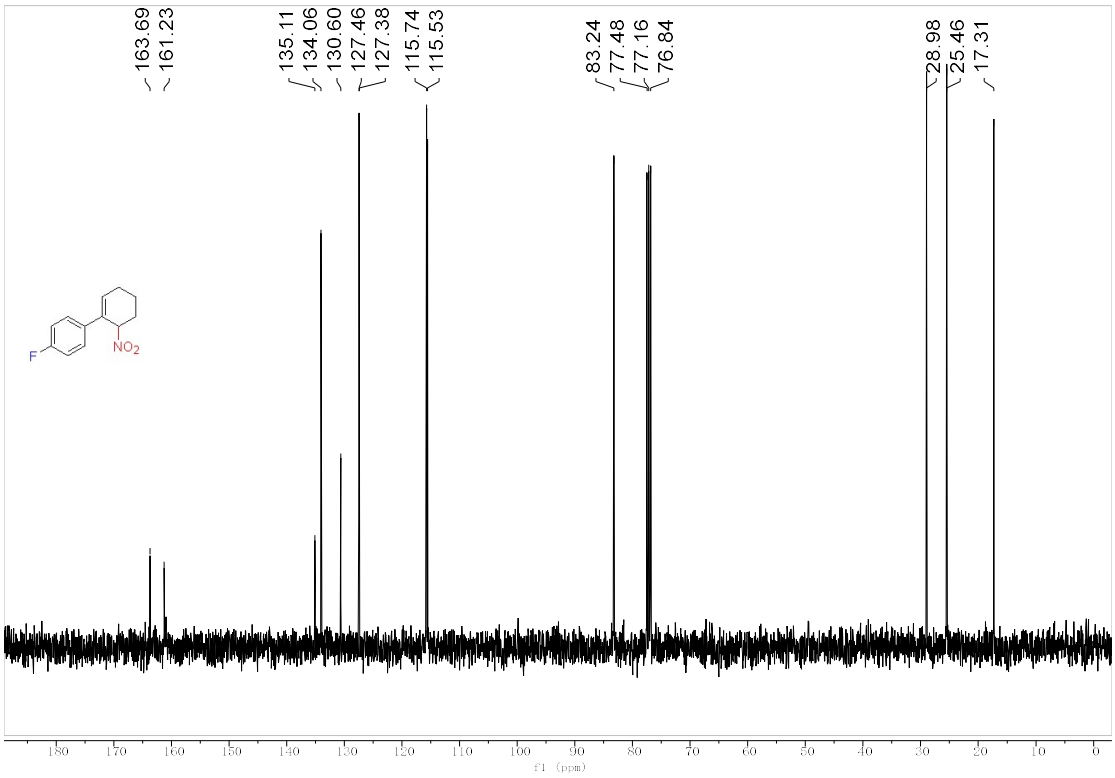
^{13}C NMR of 3a (100 MHz, $CDCl_3$)



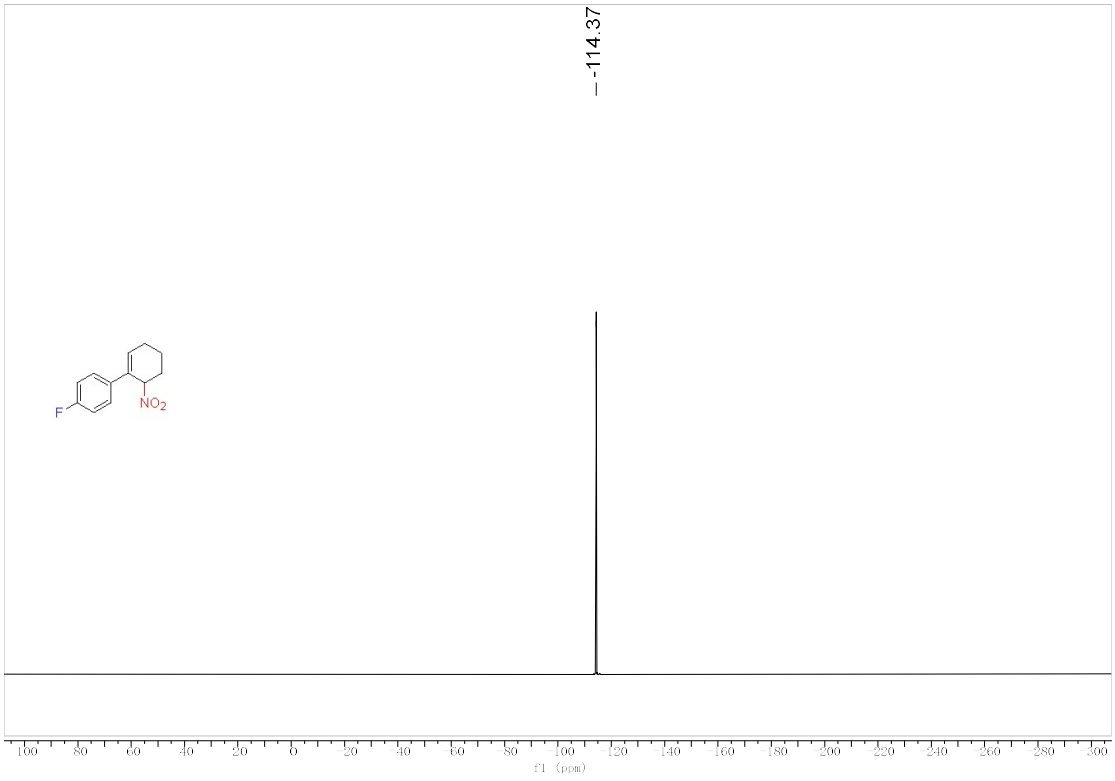
^{1}H NMR of 3b (400 MHz, $CDCl_3$)



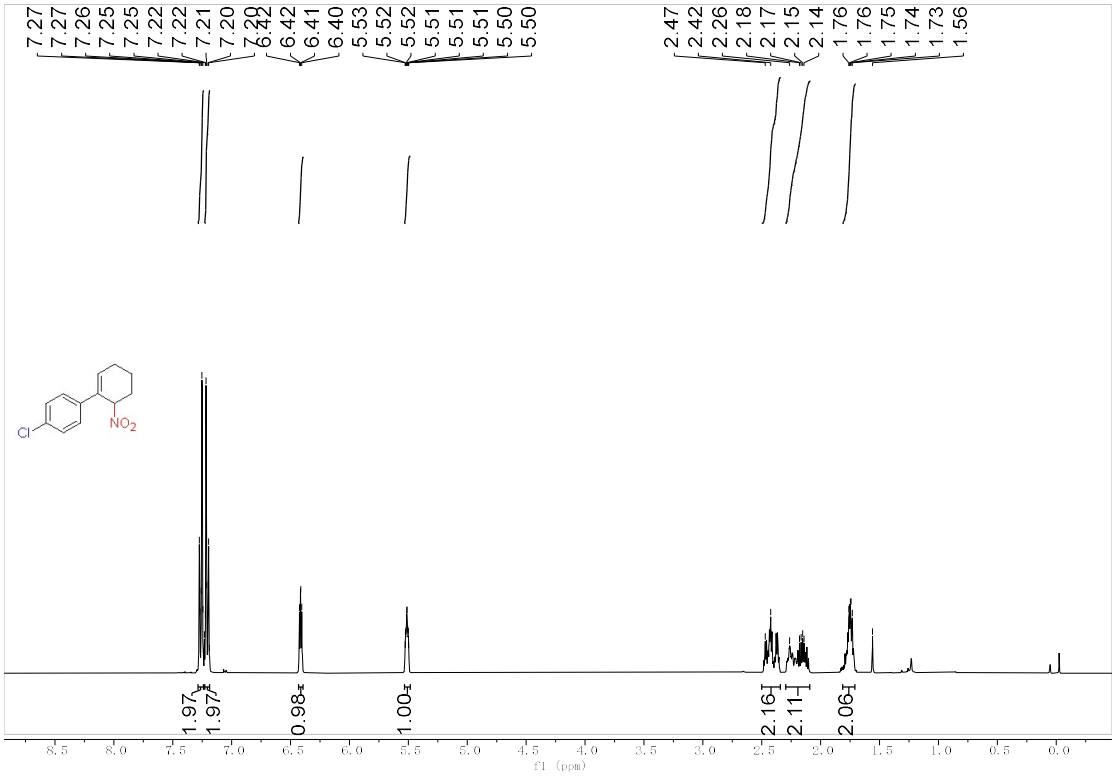
^{13}C NMR of 3b (100 MHz, $CDCl_3$)



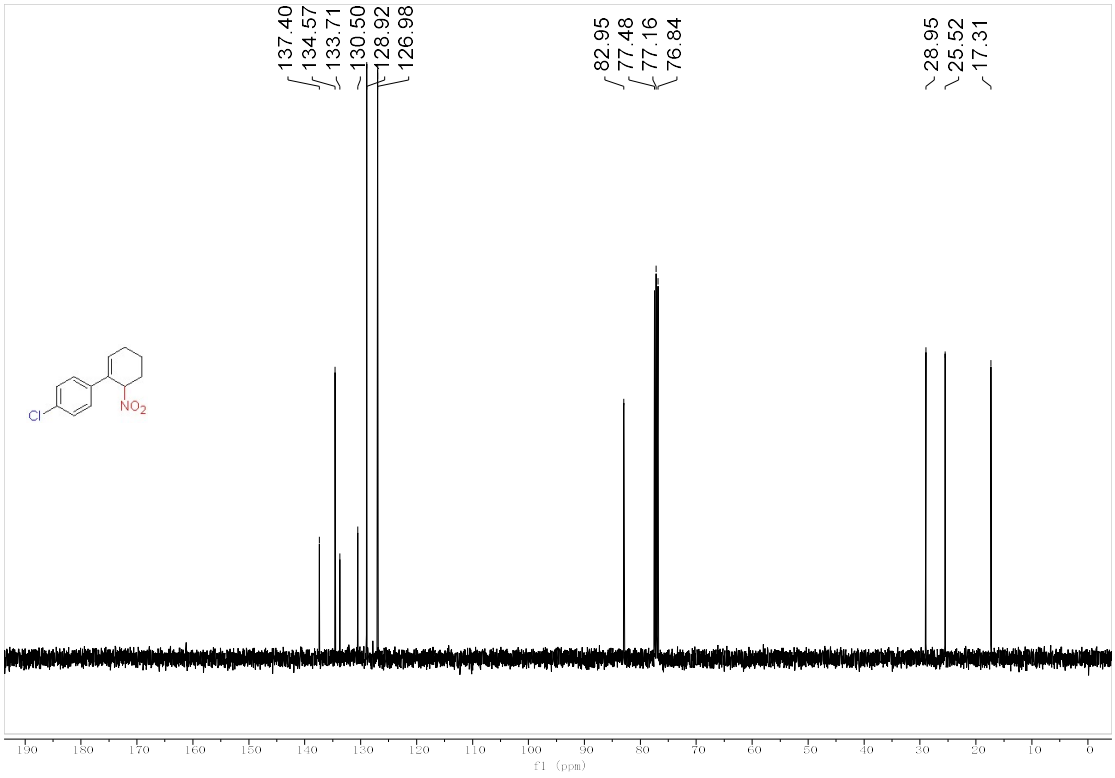
^{19}F NMR of 3b (376 MHz, $CDCl_3$)



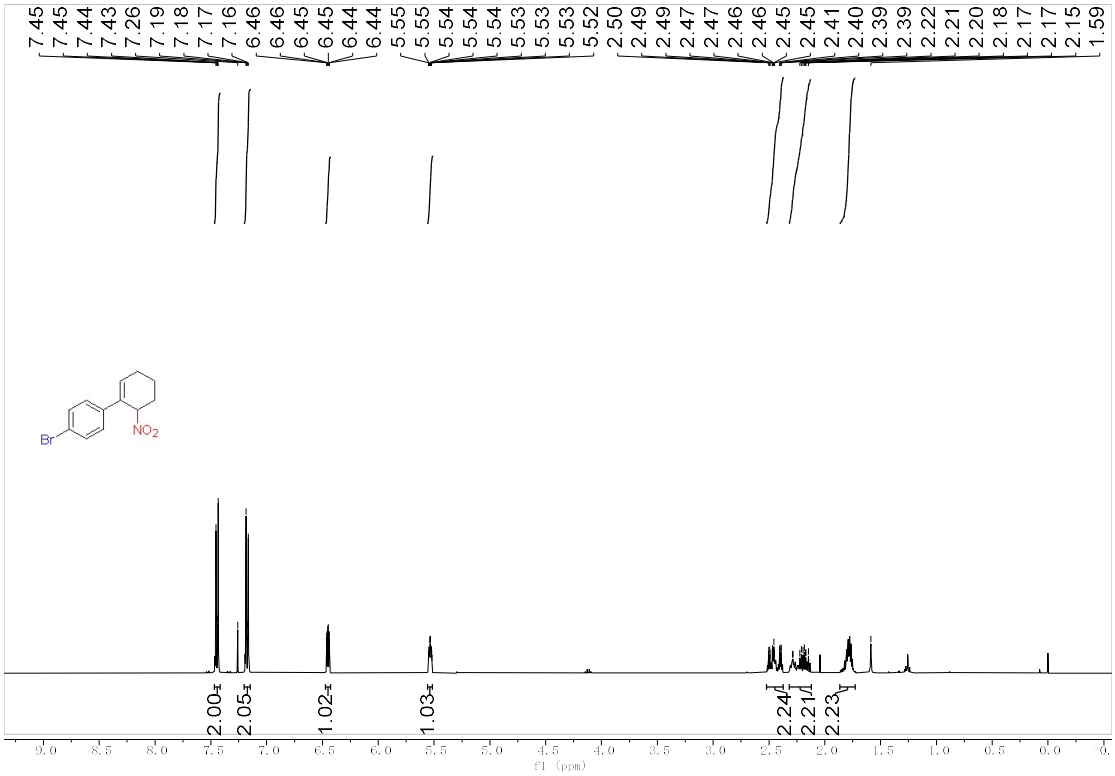
^{1}H NMR of 3c (400 MHz, $CDCl_3$)



^{13}C NMR of 3c (100 MHz, $CDCl_3$)



^{1}H NMR of 3d (400 MHz, $CDCl_3$)



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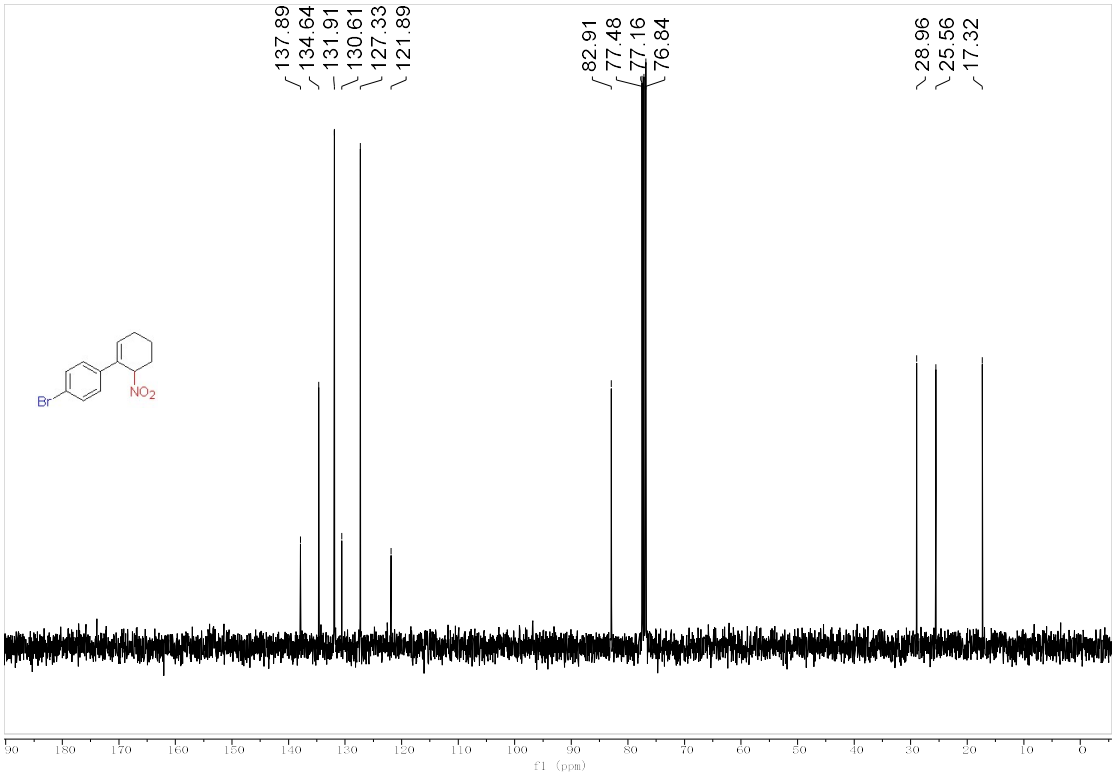
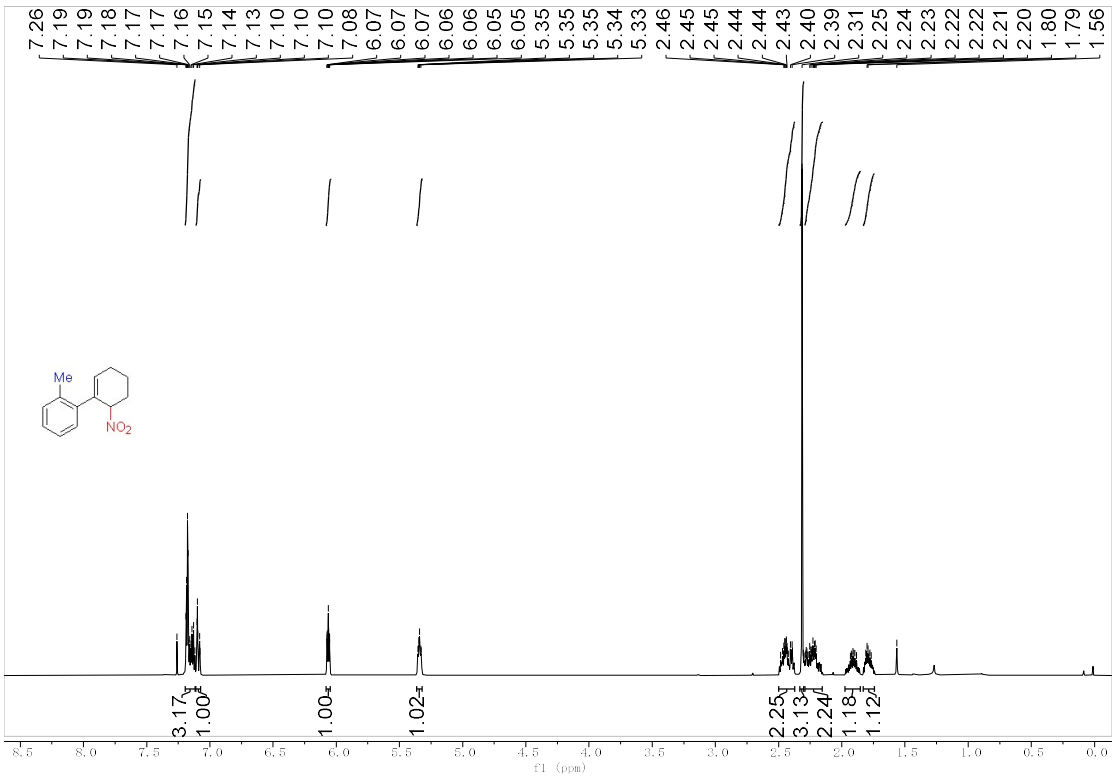
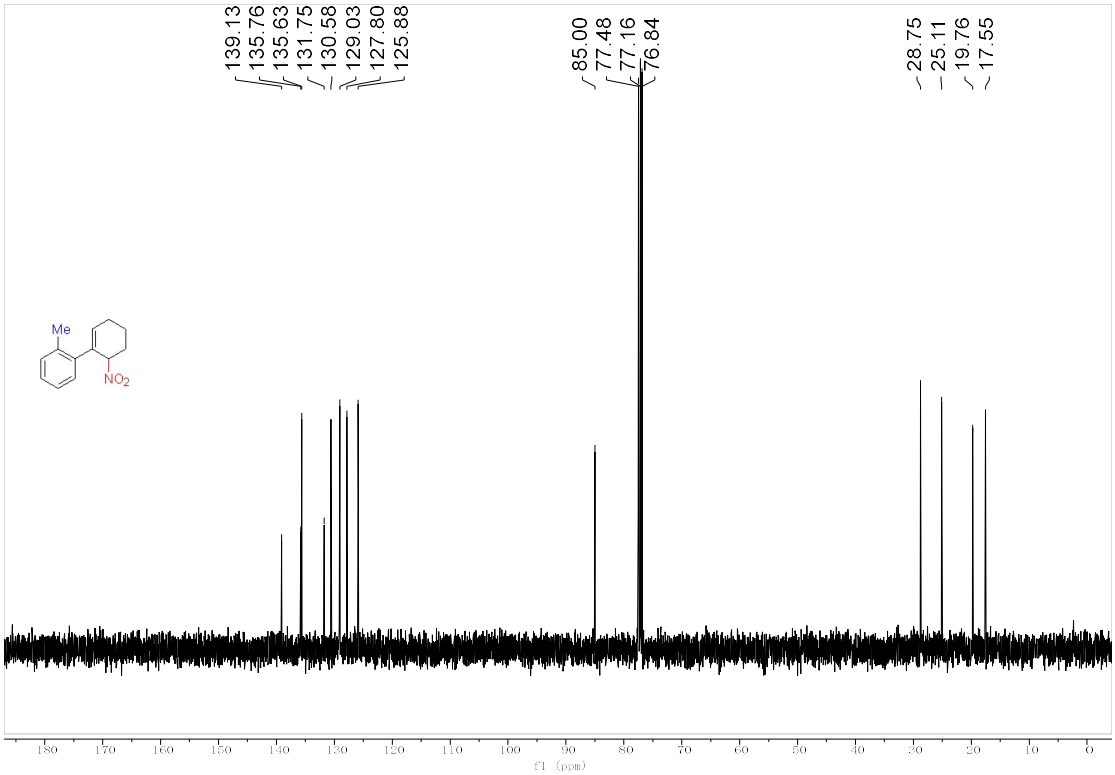


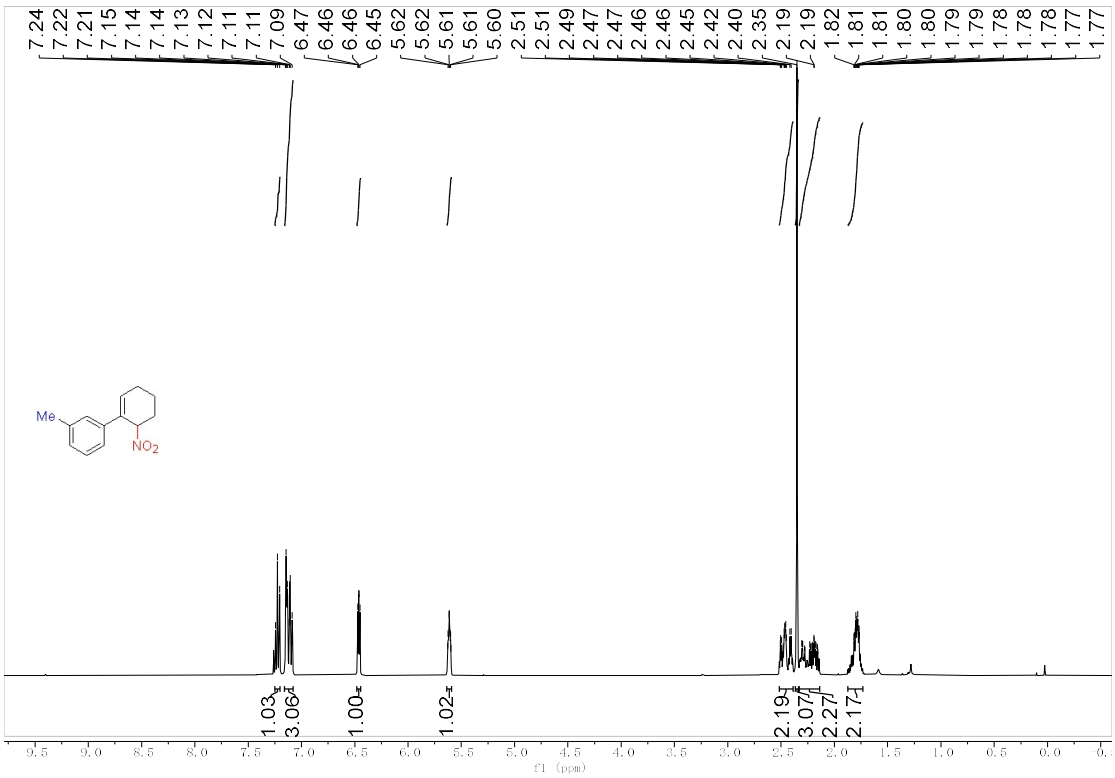
^{1}H NMR of 3e (400 MHz, $CDCl_3$)



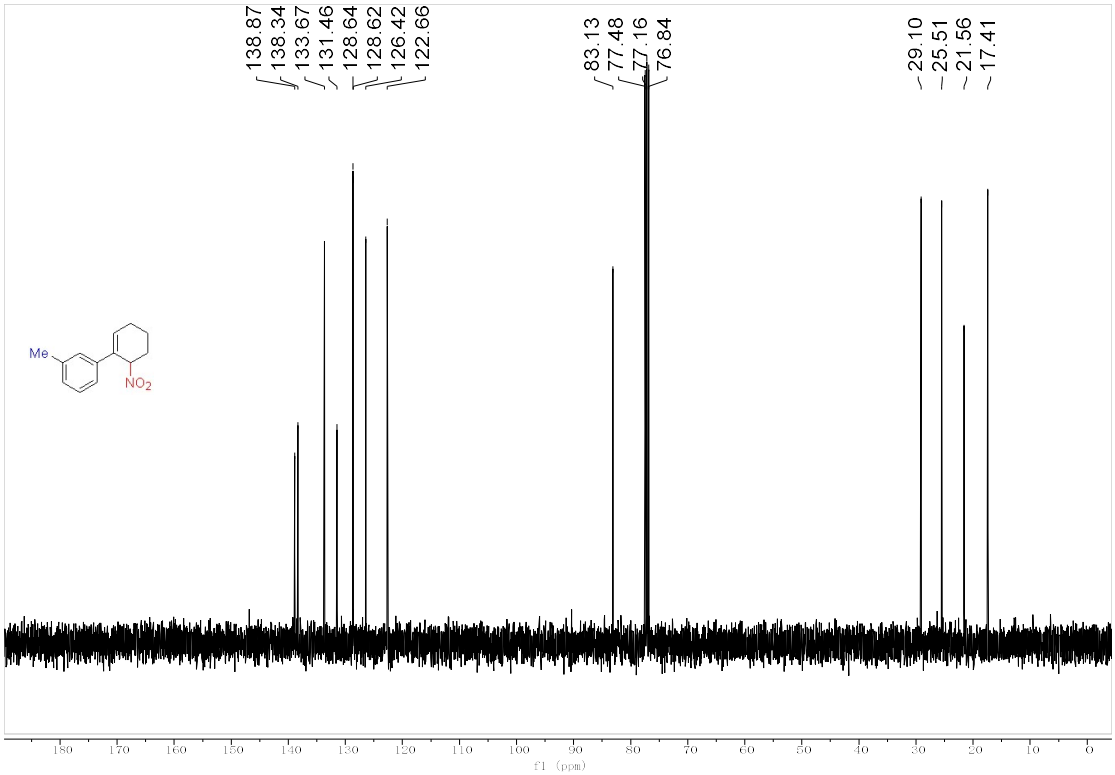
^{13}C NMR of 3e (100 MHz, $CDCl_3$)



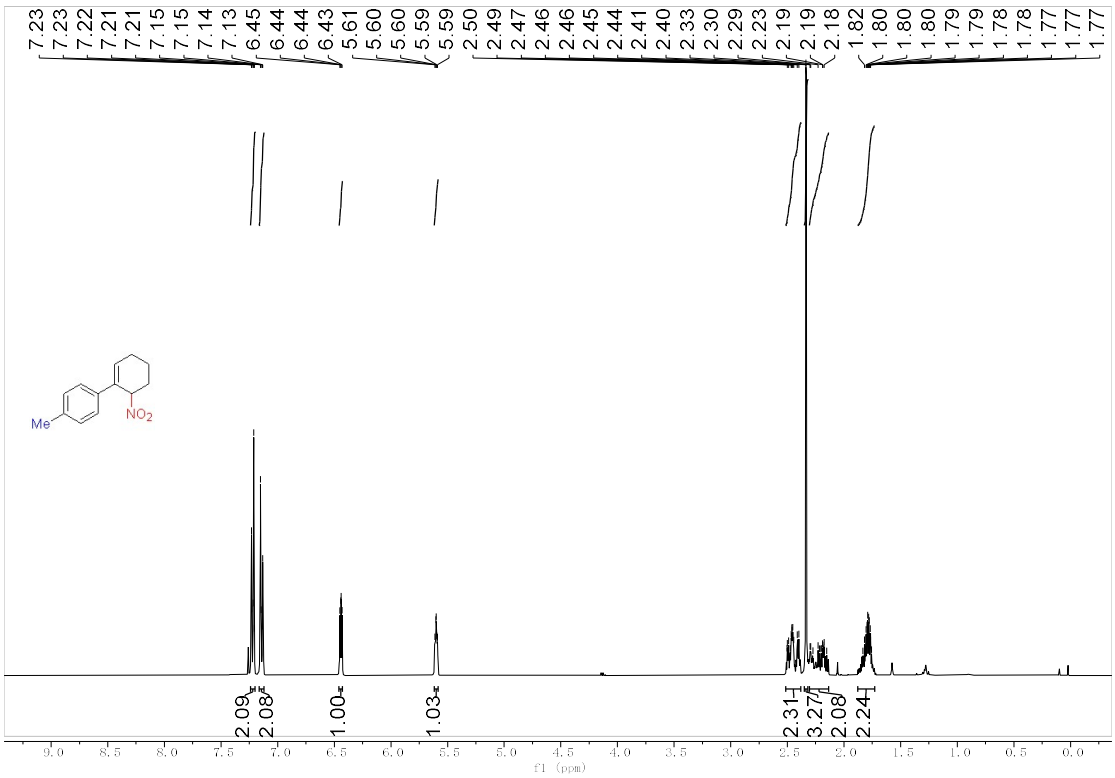
^{1}H NMR of 3f (400 MHz, $CDCl_3$)



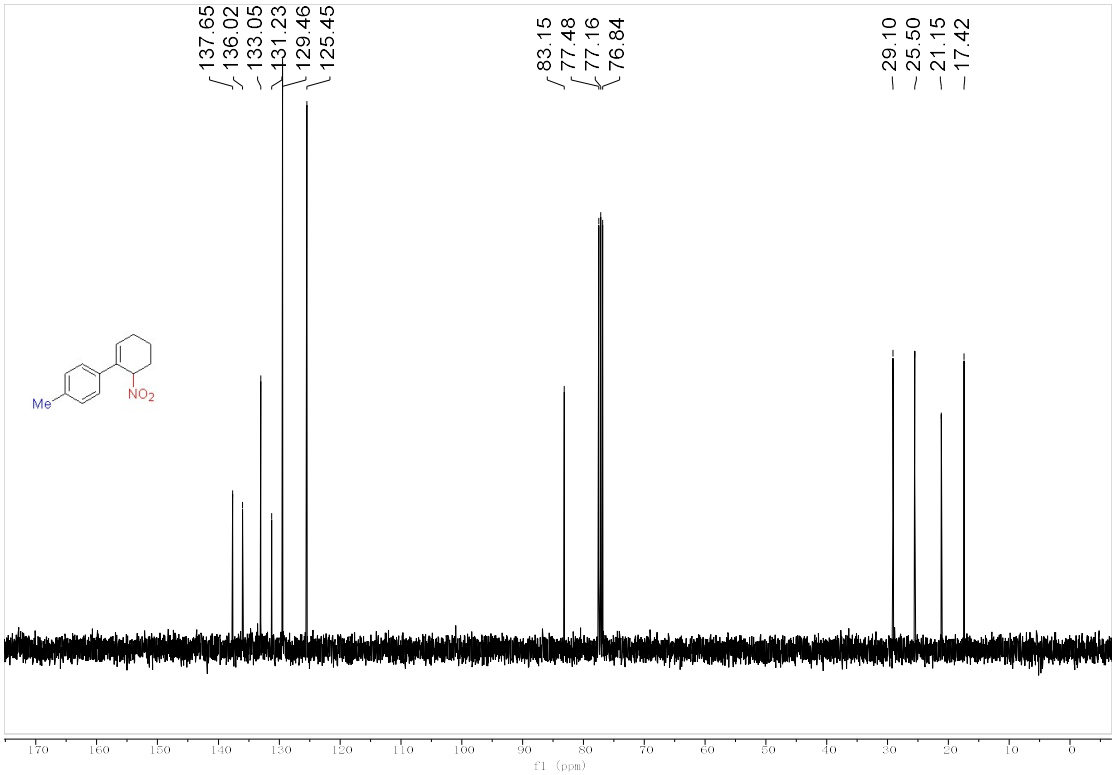
^{13}C NMR of 3f (100 MHz, $CDCl_3$)



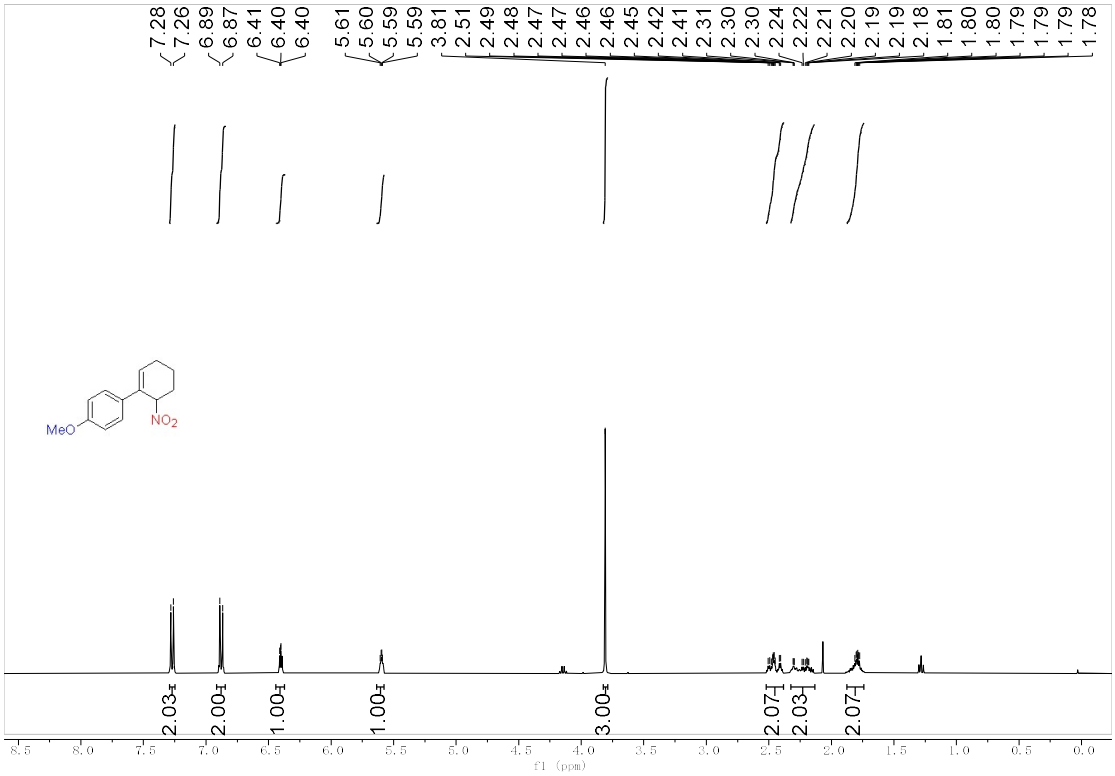
^{1}H NMR of 3g (400 MHz, $CDCl_3$)



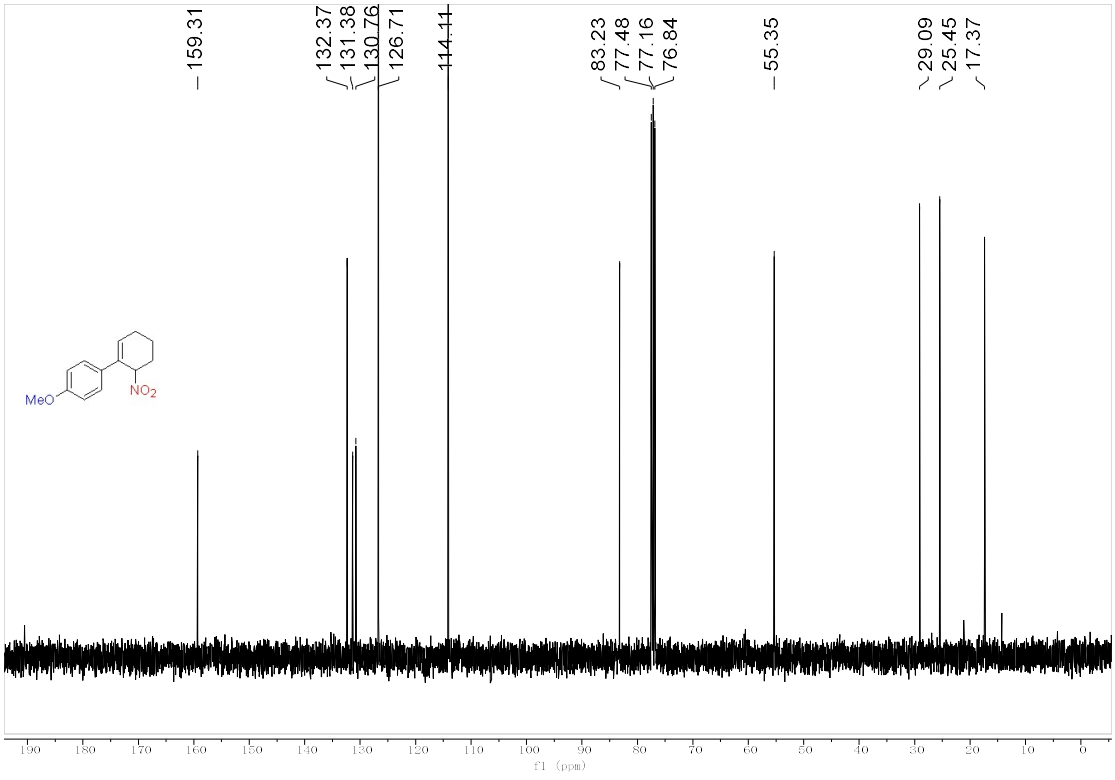
^{13}C NMR of 3g (100 MHz, $CDCl_3$)



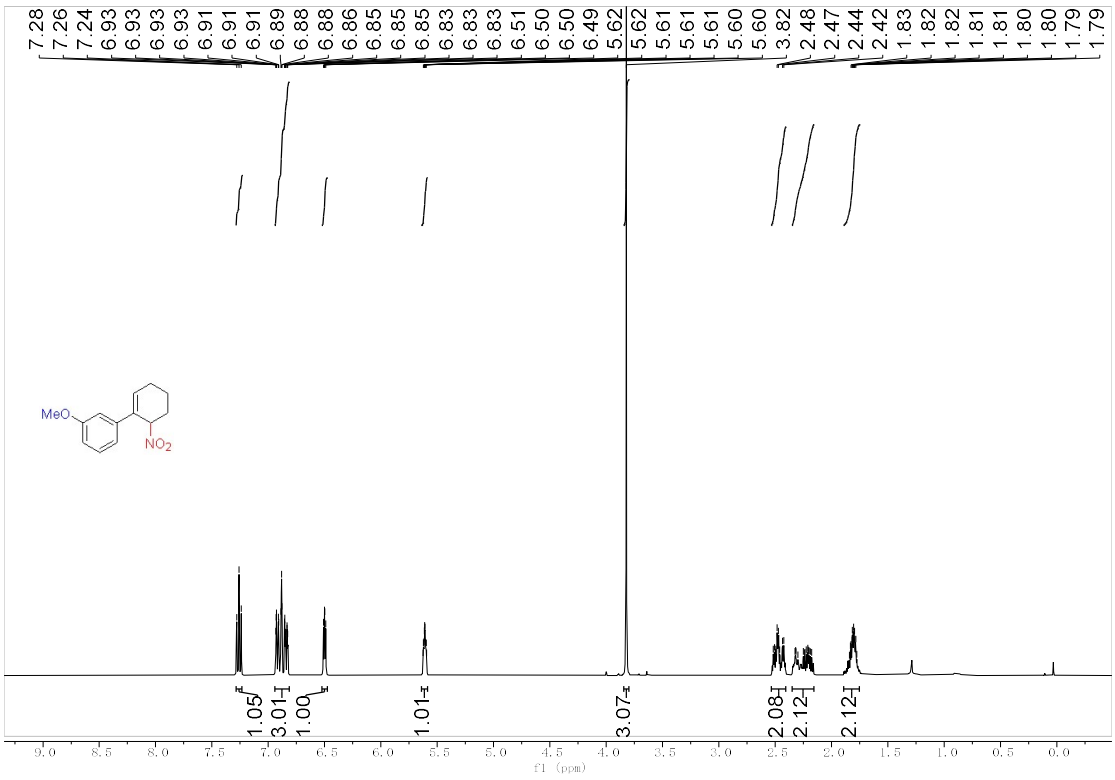
^{1}H NMR of 3h (400 MHz, $CDCl_3$)



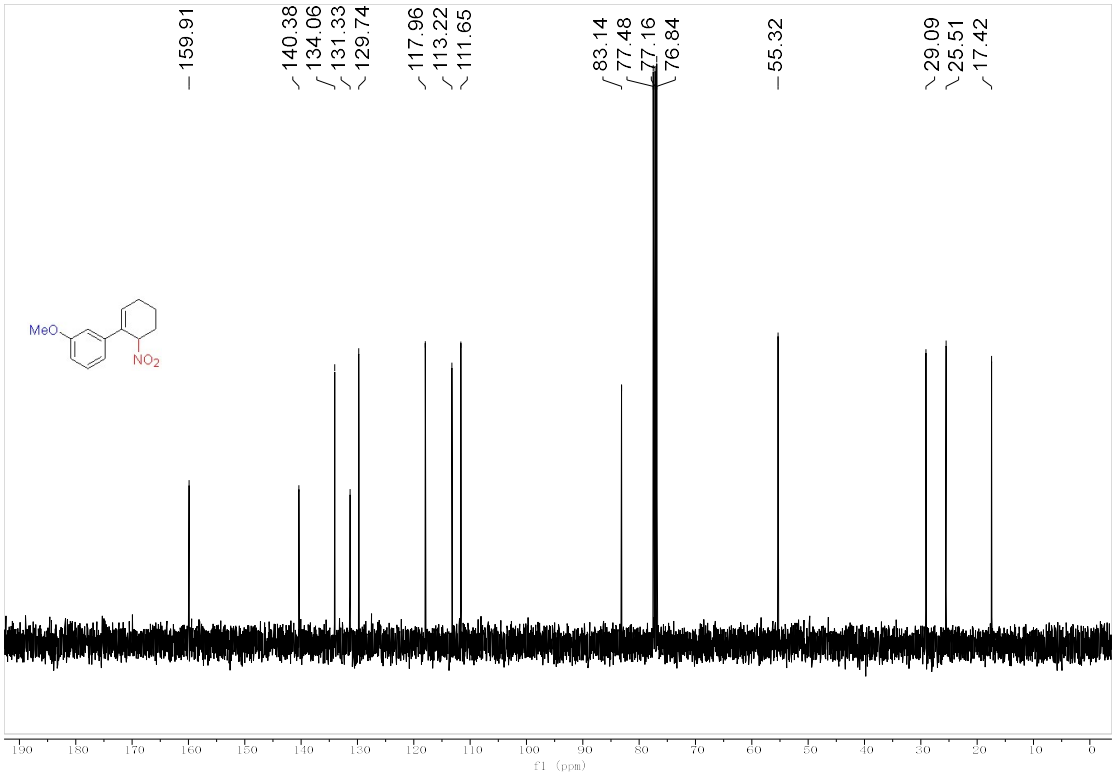
^{13}C NMR of 3h (100 MHz, $CDCl_3$)



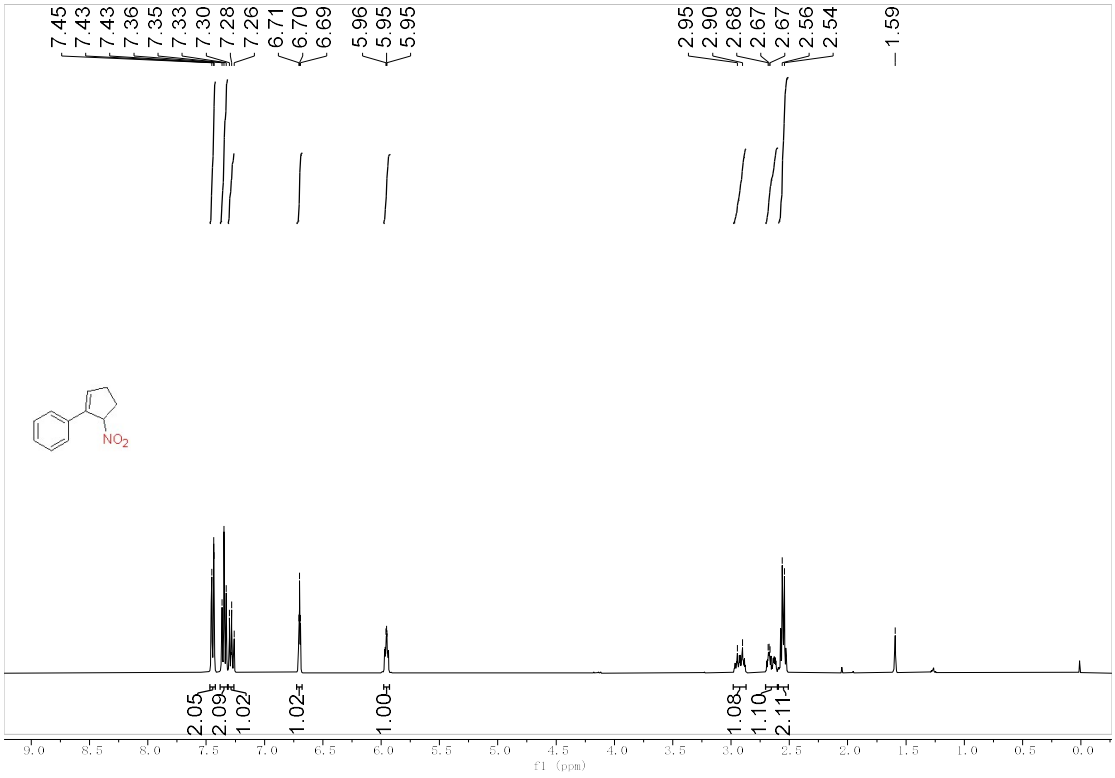
^{1}H NMR of 3i (400 MHz, $CDCl_3$)



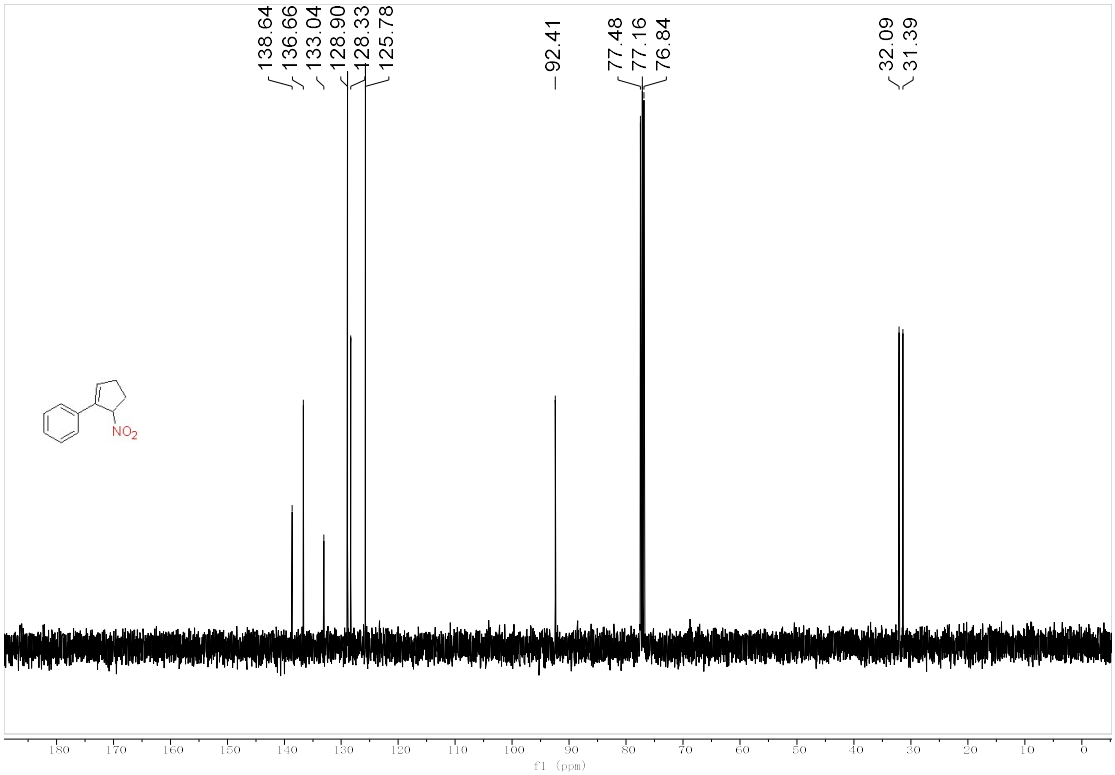
^{13}C NMR of 3i (100 MHz, $CDCl_3$)



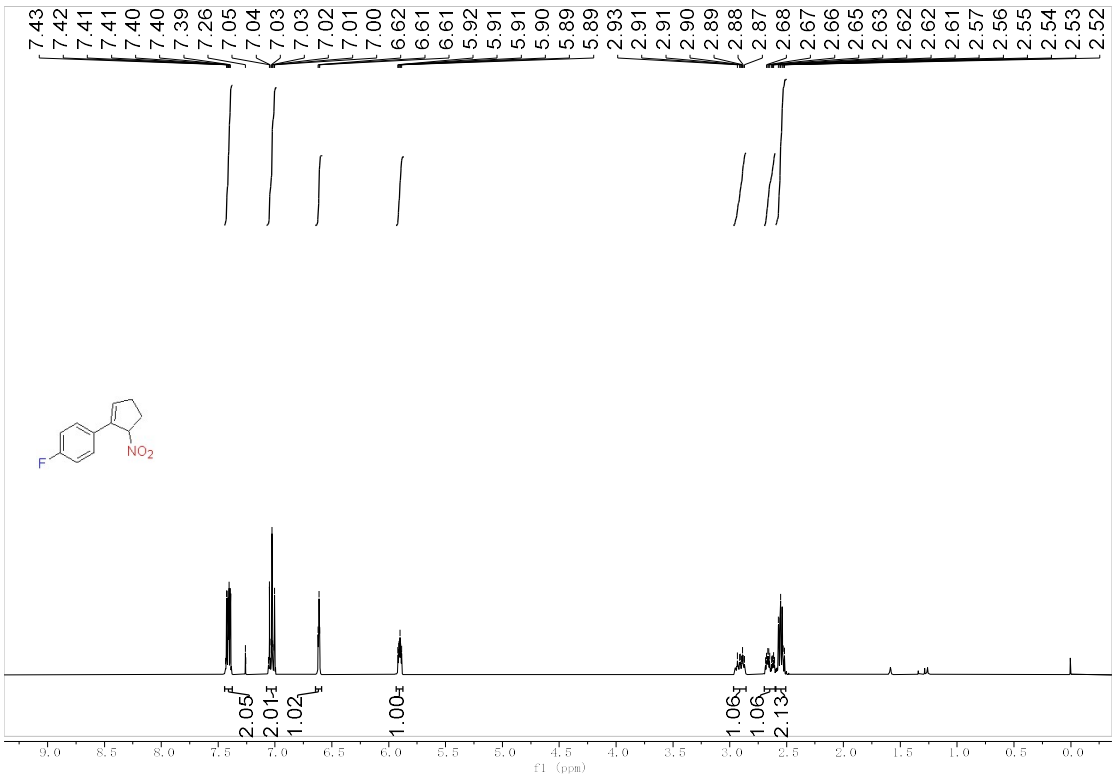
^{1}H NMR of 3j (400 MHz, $CDCl_3$)



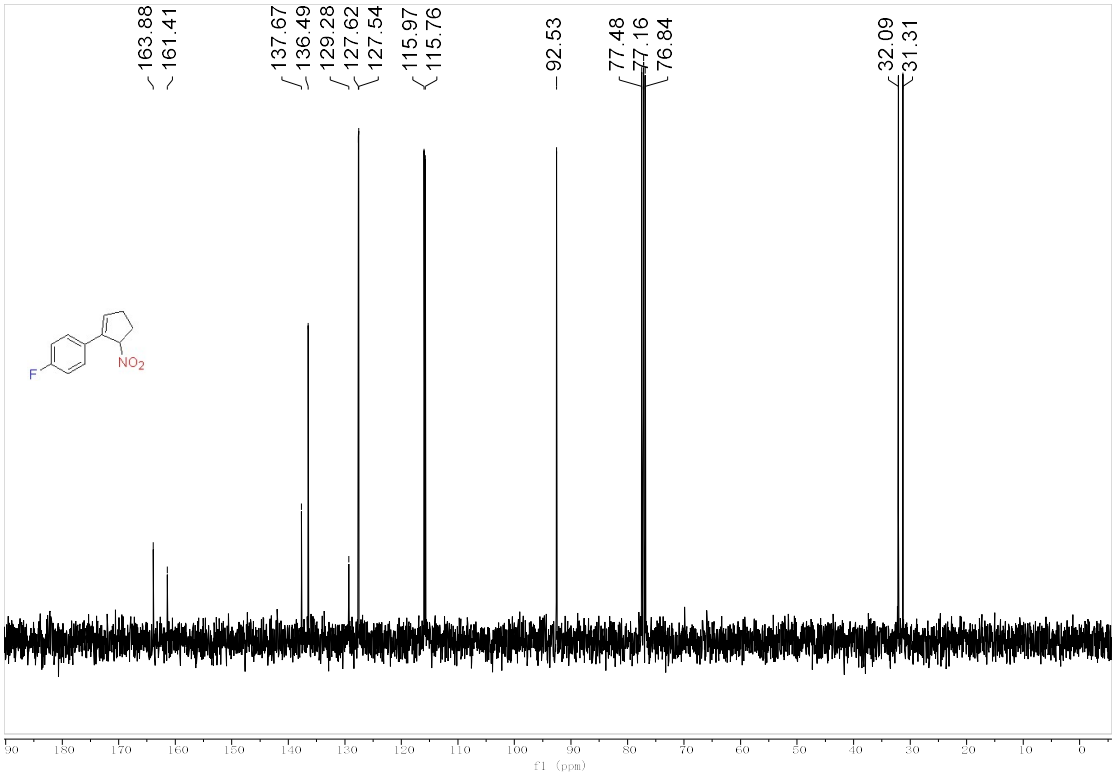
^{13}C NMR of 3j (100 MHz, $CDCl_3$)



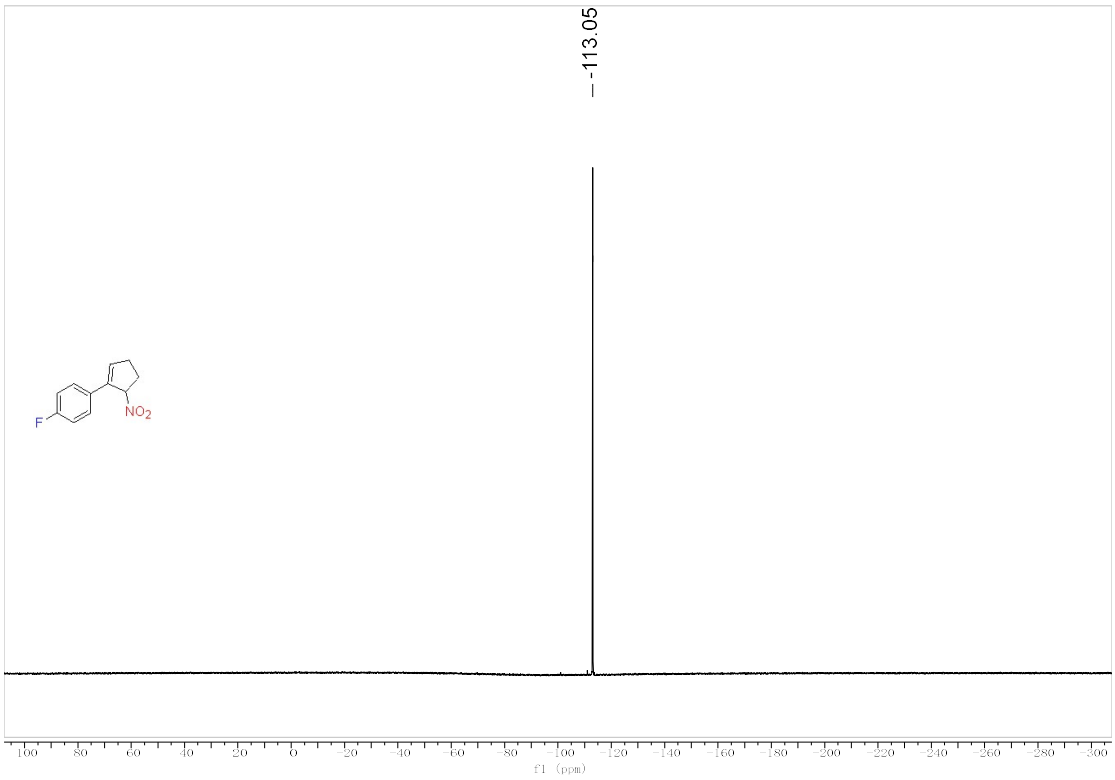
^{1}H NMR of 3k (400 MHz, $CDCl_3$)



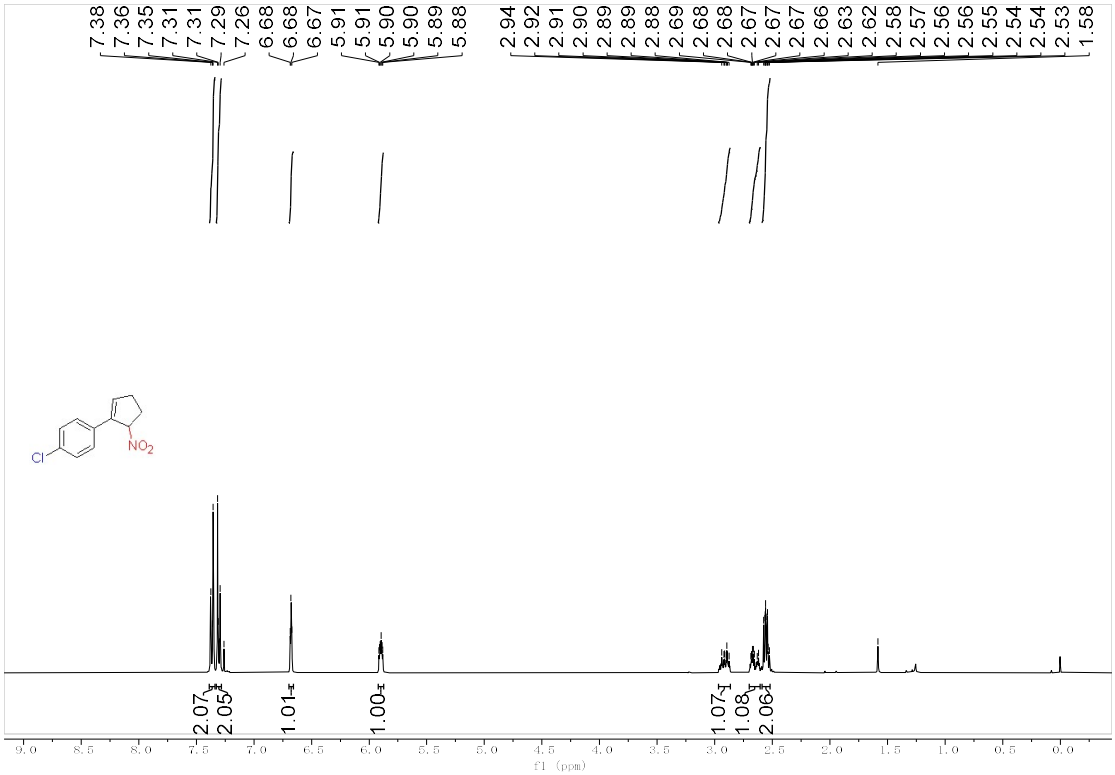
^{13}C NMR of 3k (100 MHz, $CDCl_3$)



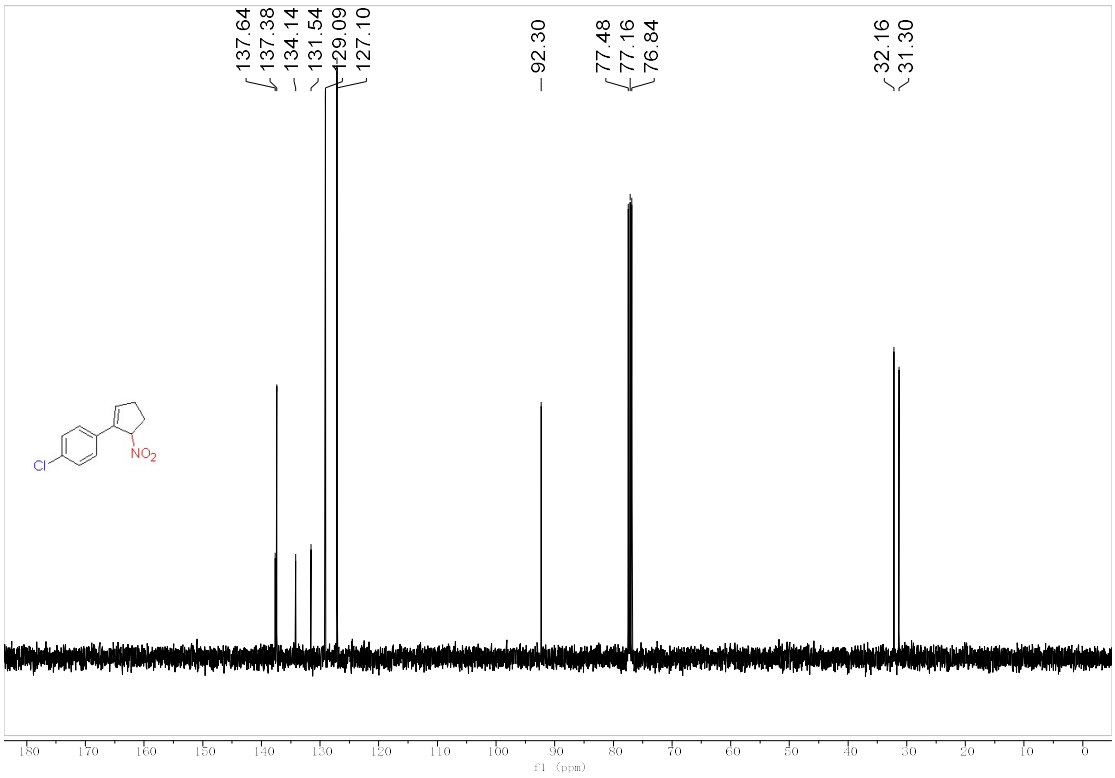
^{19}F NMR of 3k (376 MHz, $CDCl_3$)



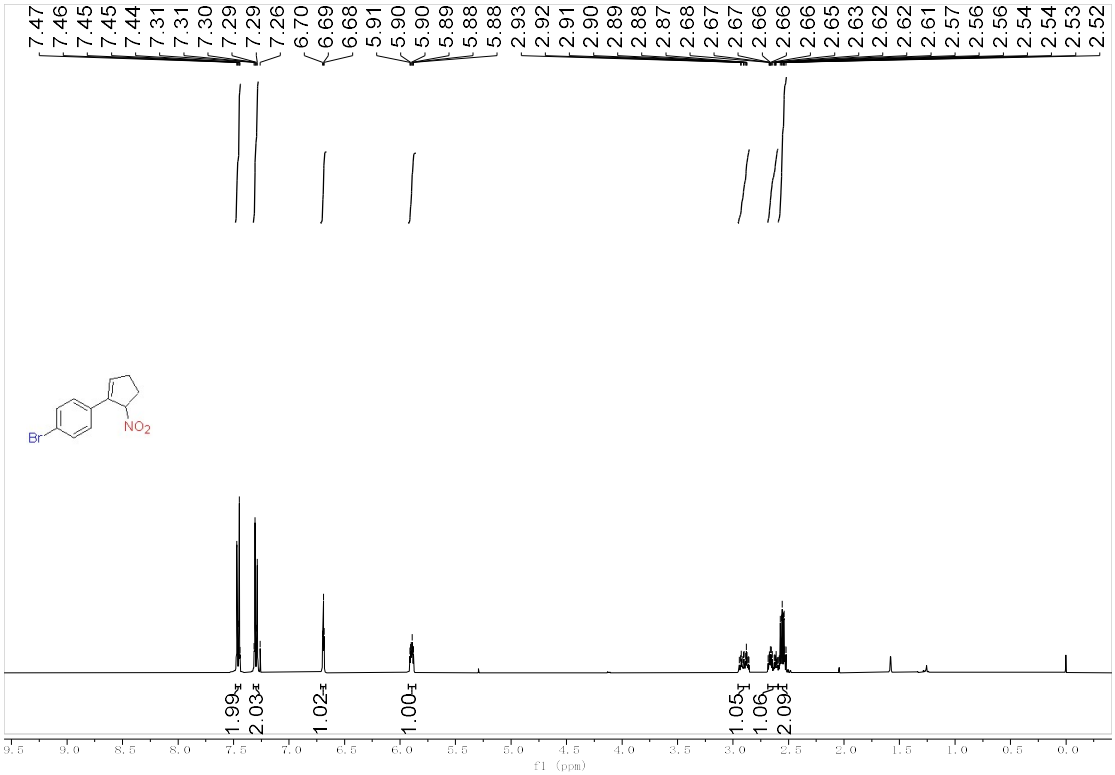
^{1}H NMR of 3l (400 MHz, $CDCl_3$)



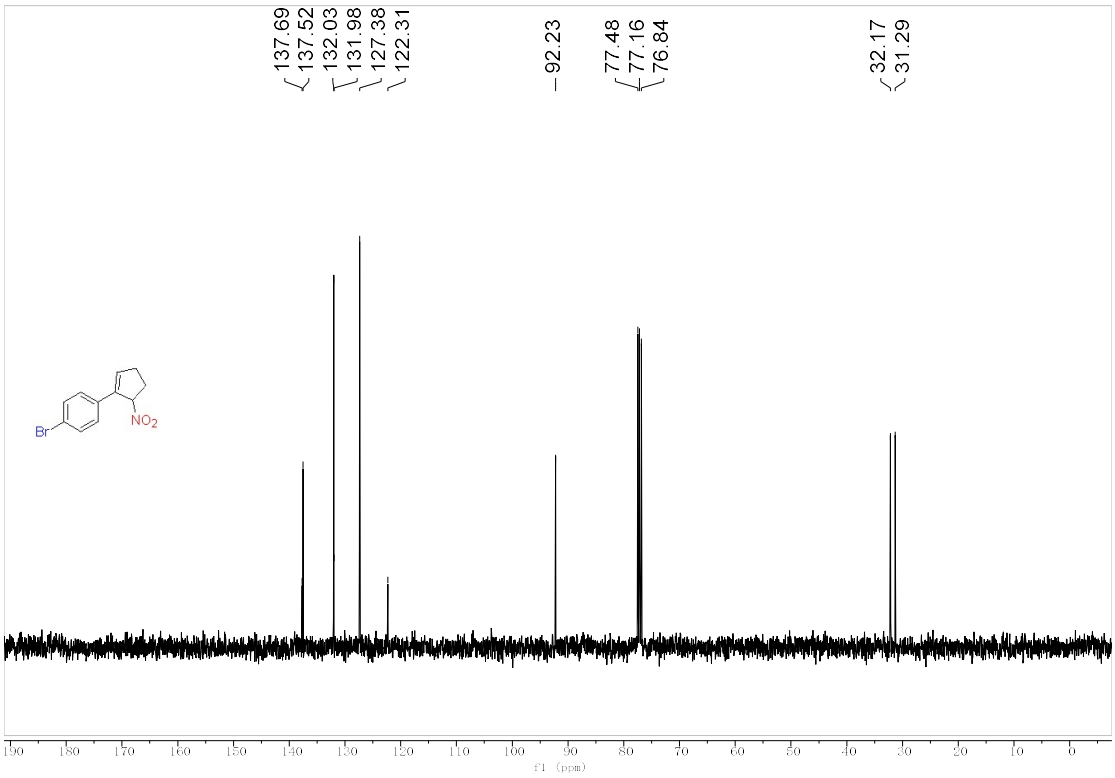
^{13}C NMR of 3l (100 MHz, $CDCl_3$)



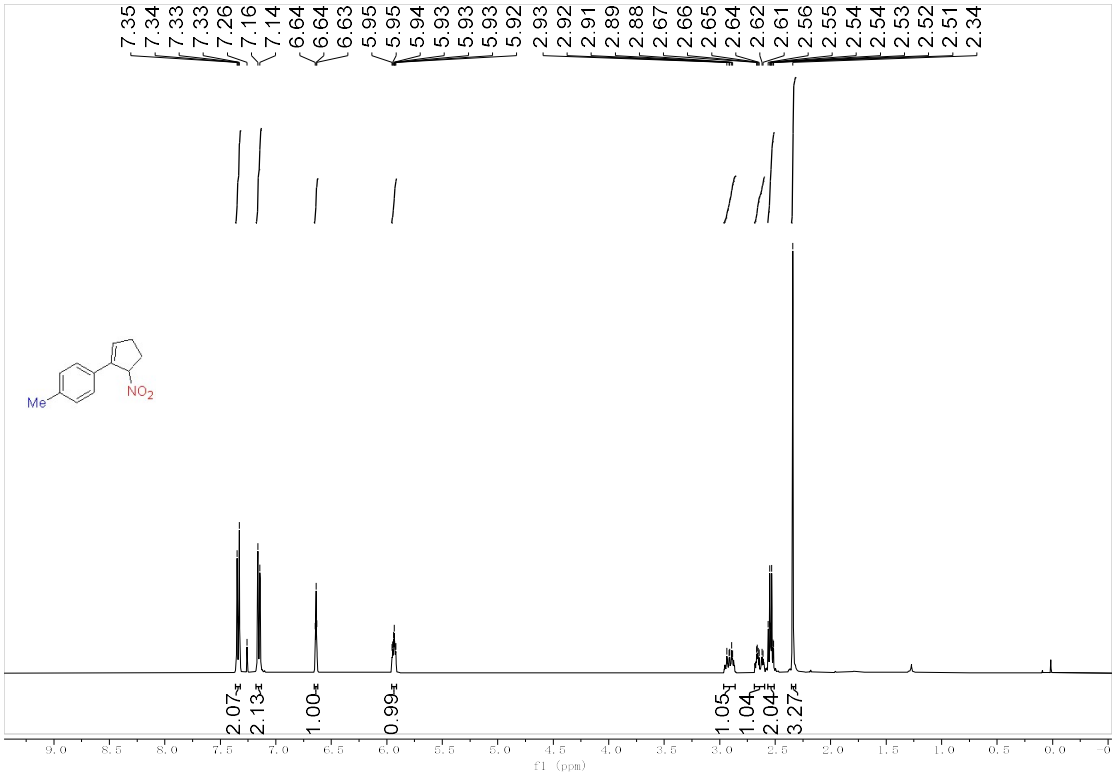
^{1}H NMR of 3m (400 MHz, $CDCl_3$)



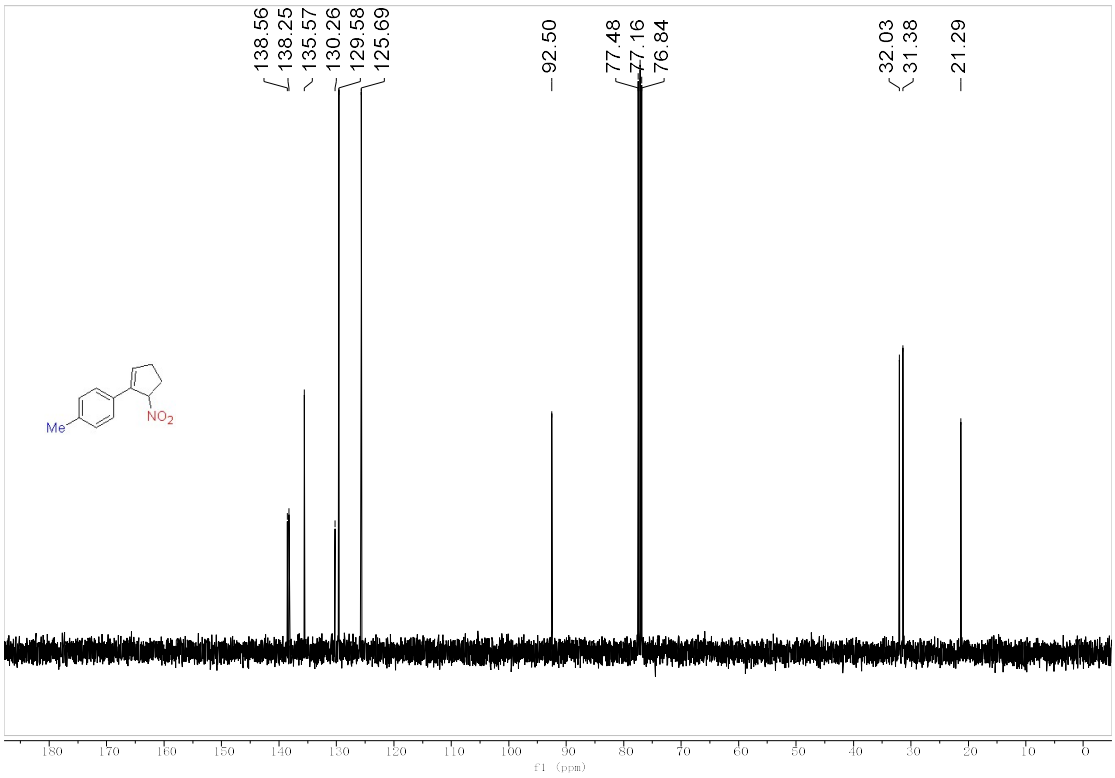
^{13}C NMR of 3m (100 MHz, $CDCl_3$)



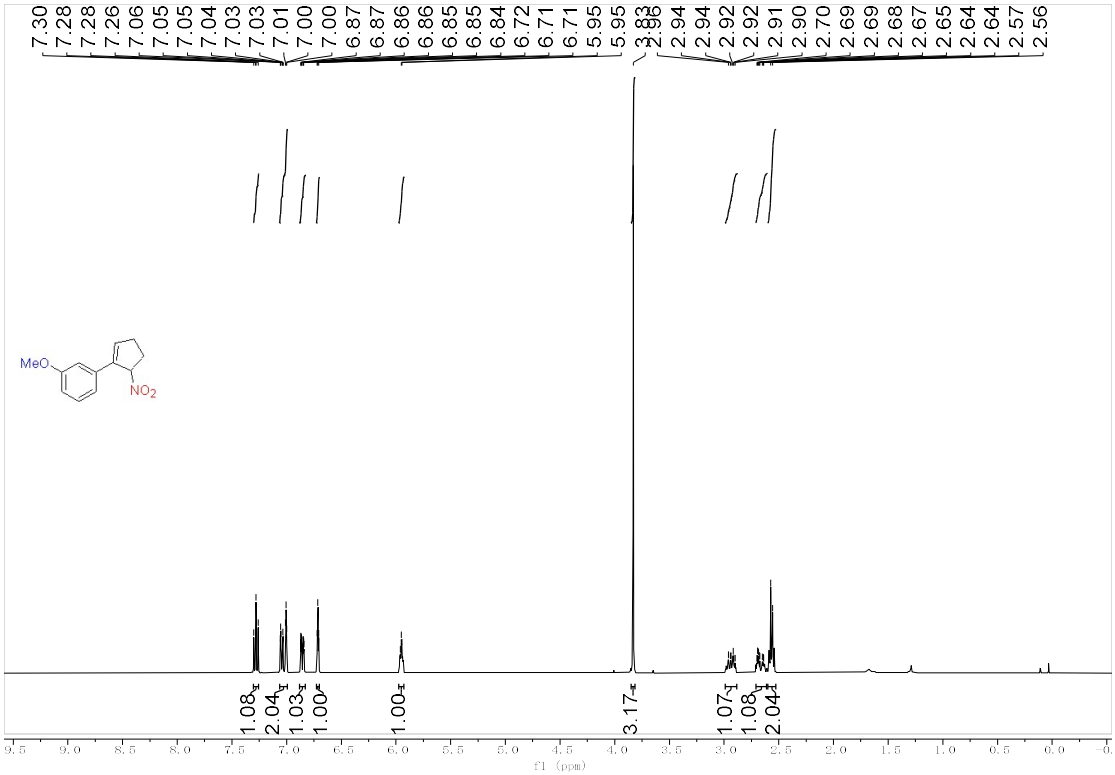
^{1}H NMR of 3n (400 MHz, $CDCl_3$)



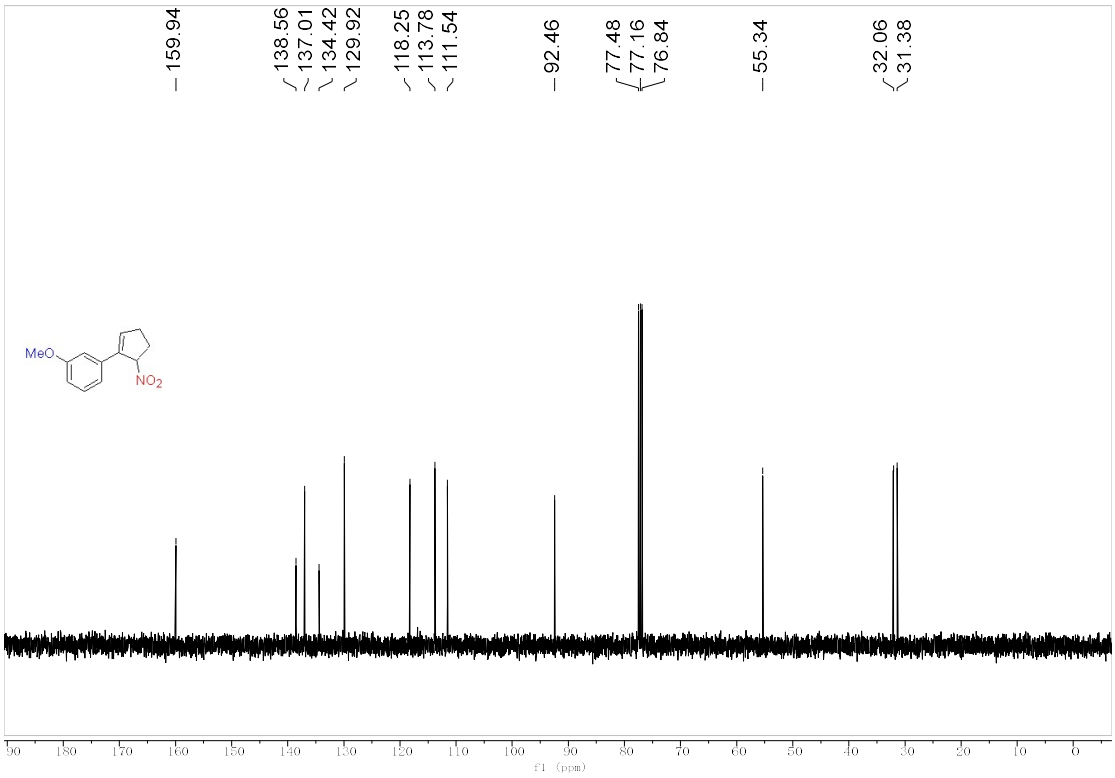
^{13}C NMR of 3n (100 MHz, $CDCl_3$)



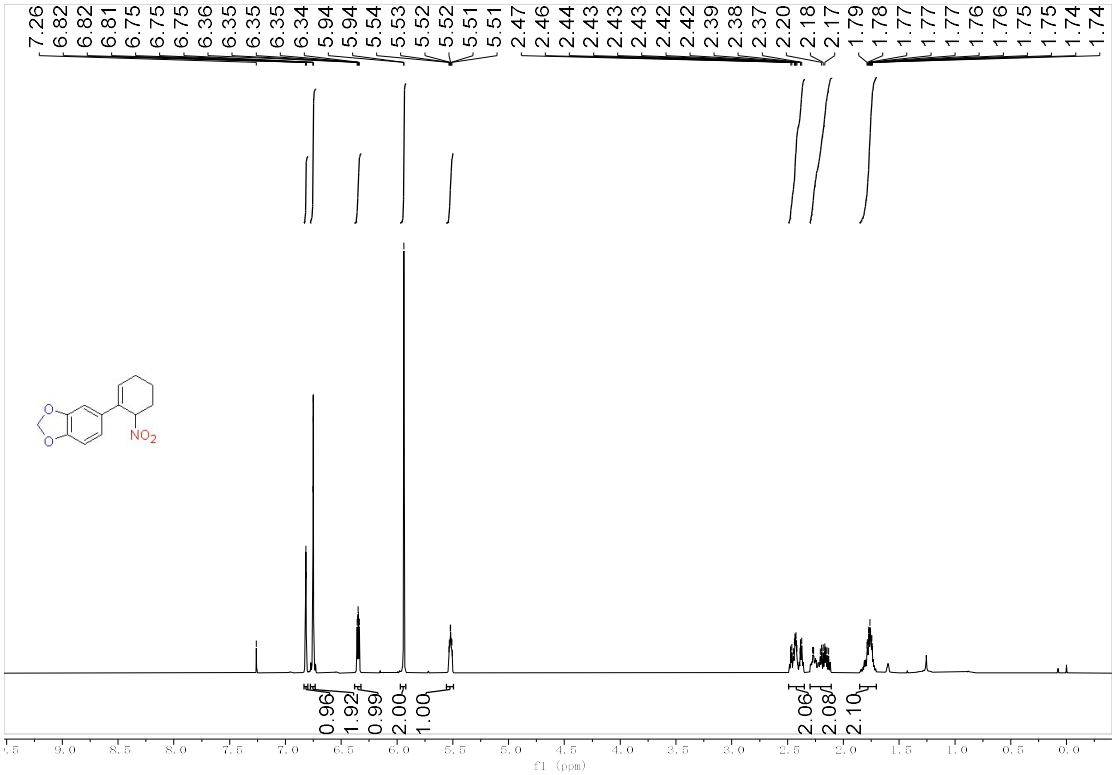
^{1}H NMR of 3o (400 MHz, $CDCl_3$)



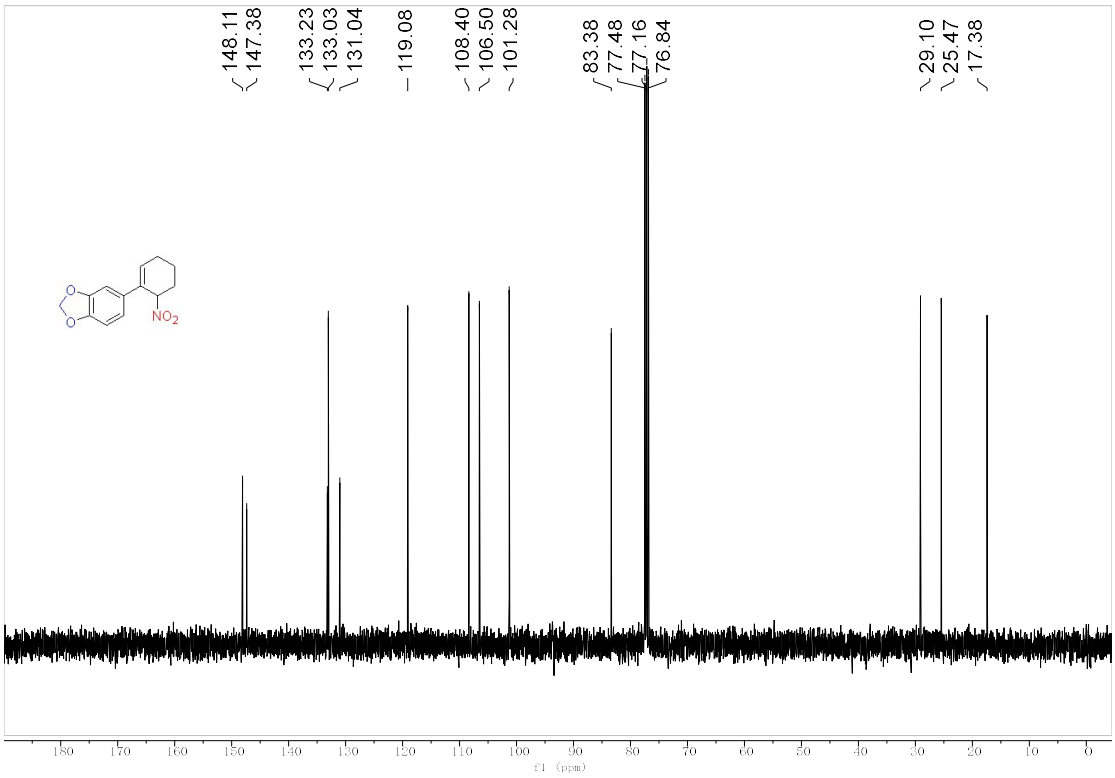
^{13}C NMR of 3o (100 MHz, $CDCl_3$)



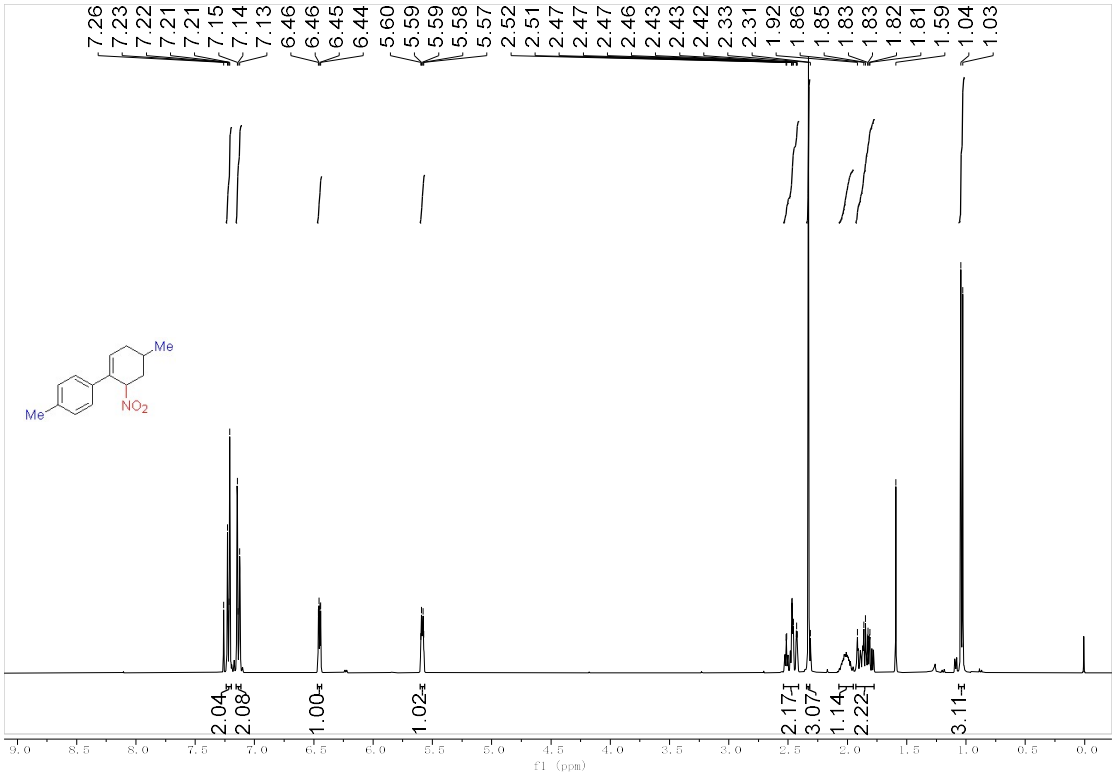
^{1}H NMR of 3p (400 MHz, $CDCl_3$)



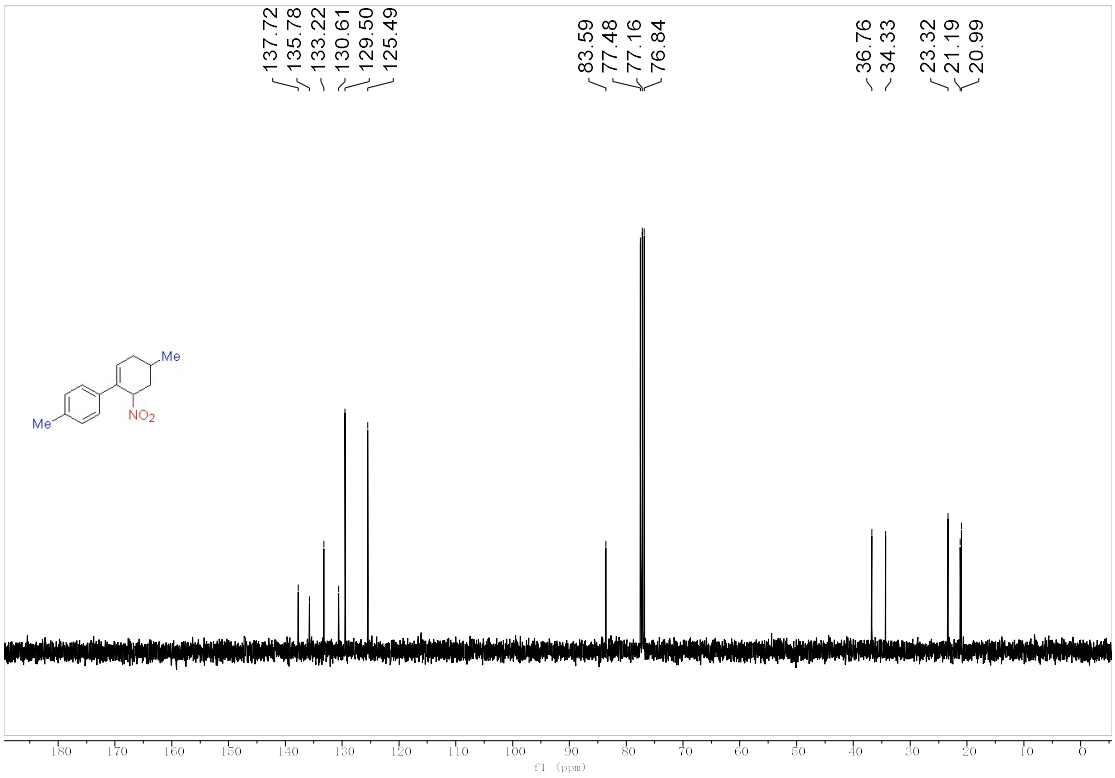
^{13}C NMR of 3p (100 MHz, $CDCl_3$)



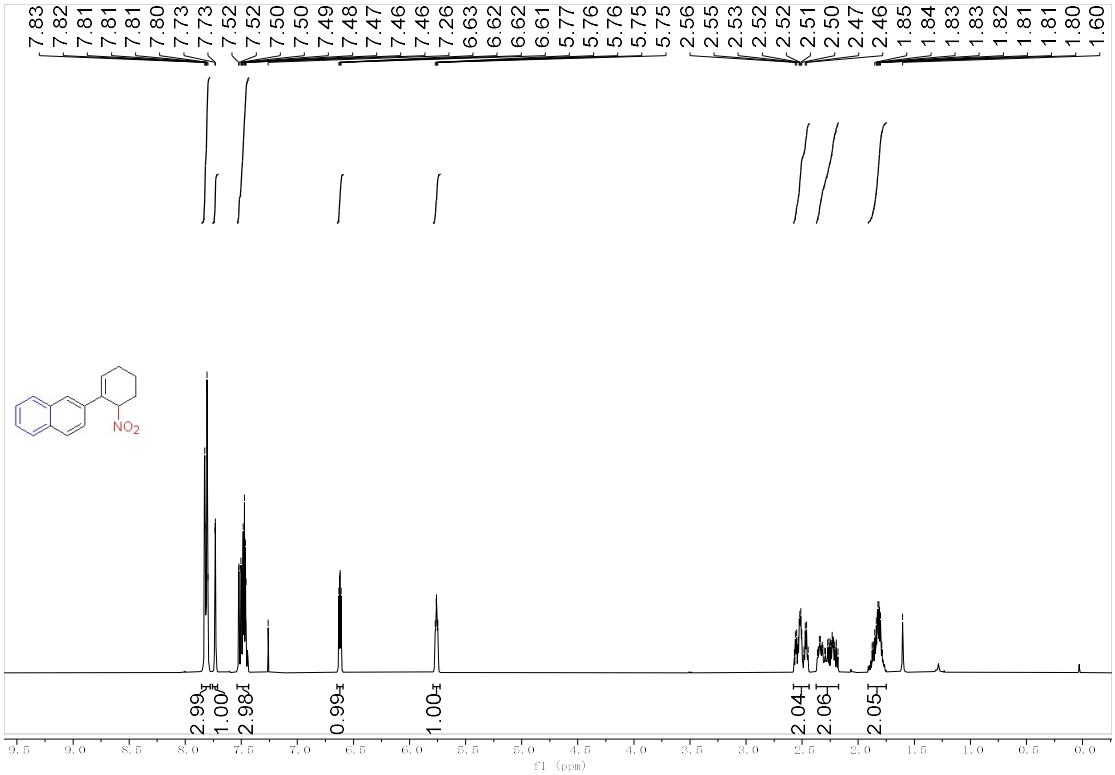
^{1}H NMR of 3q (400 MHz, $CDCl_3$)



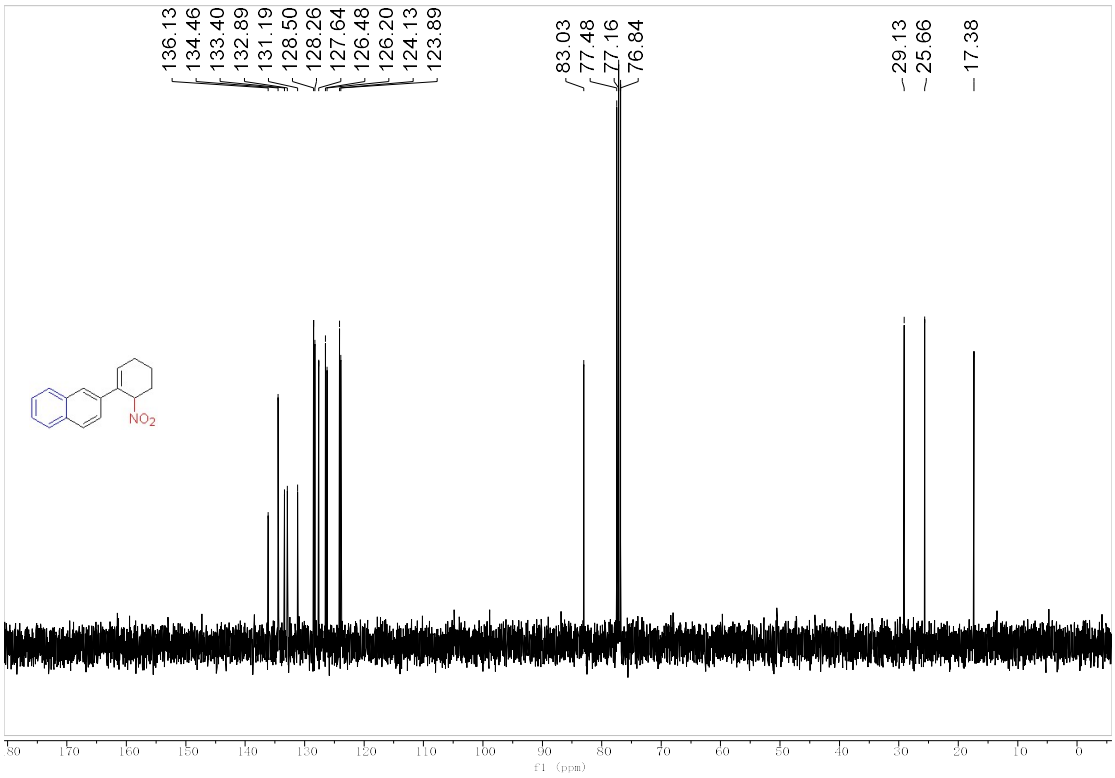
^{13}C NMR of 3q (100 MHz, $CDCl_3$)



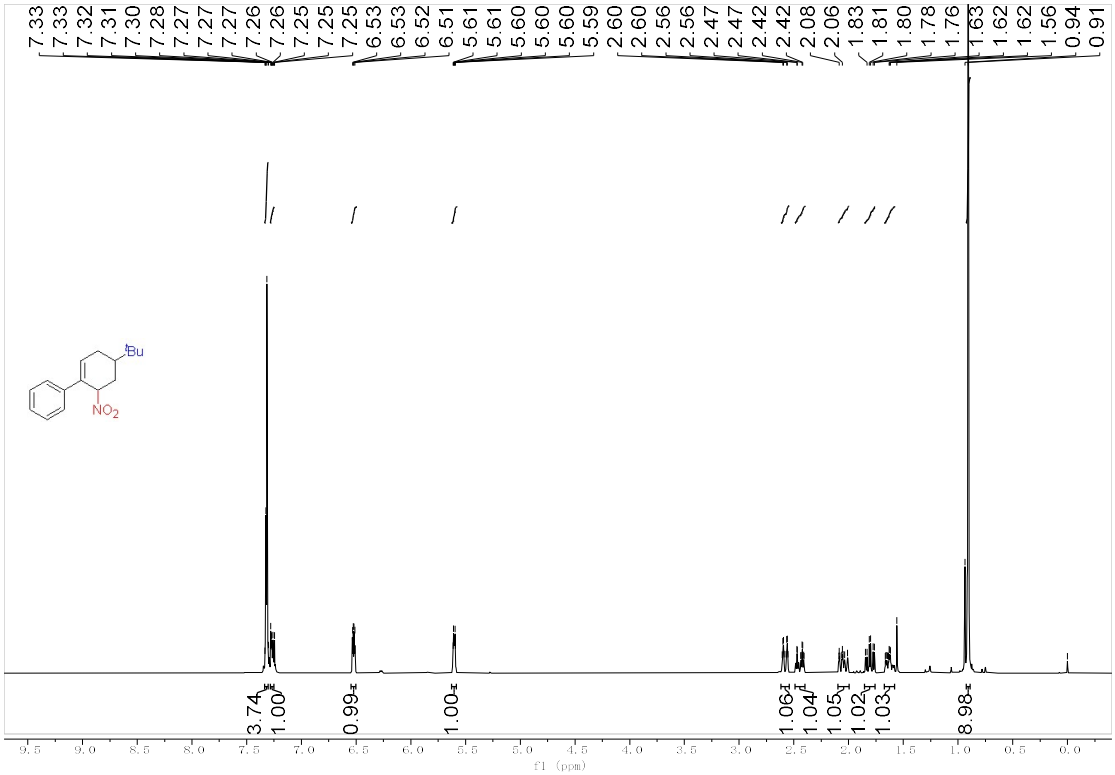
^{1}H NMR of 3r (400 MHz, $CDCl_3$)



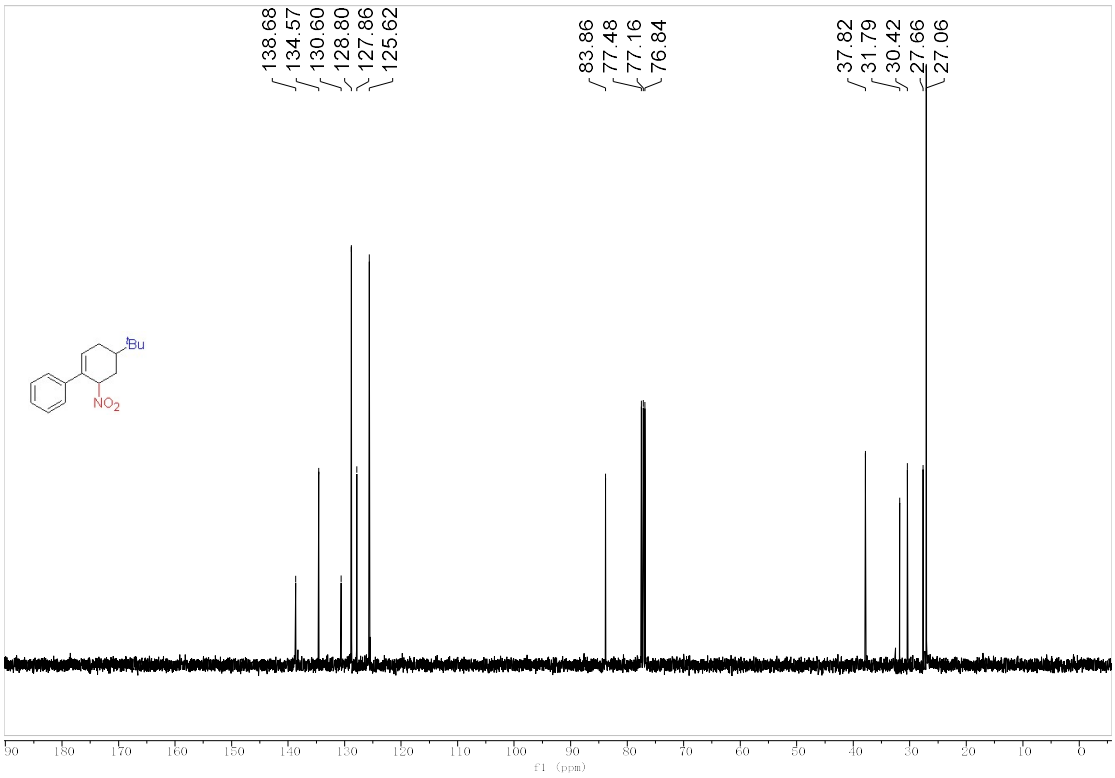
^{13}C NMR of 3r (100 MHz, $CDCl_3$)



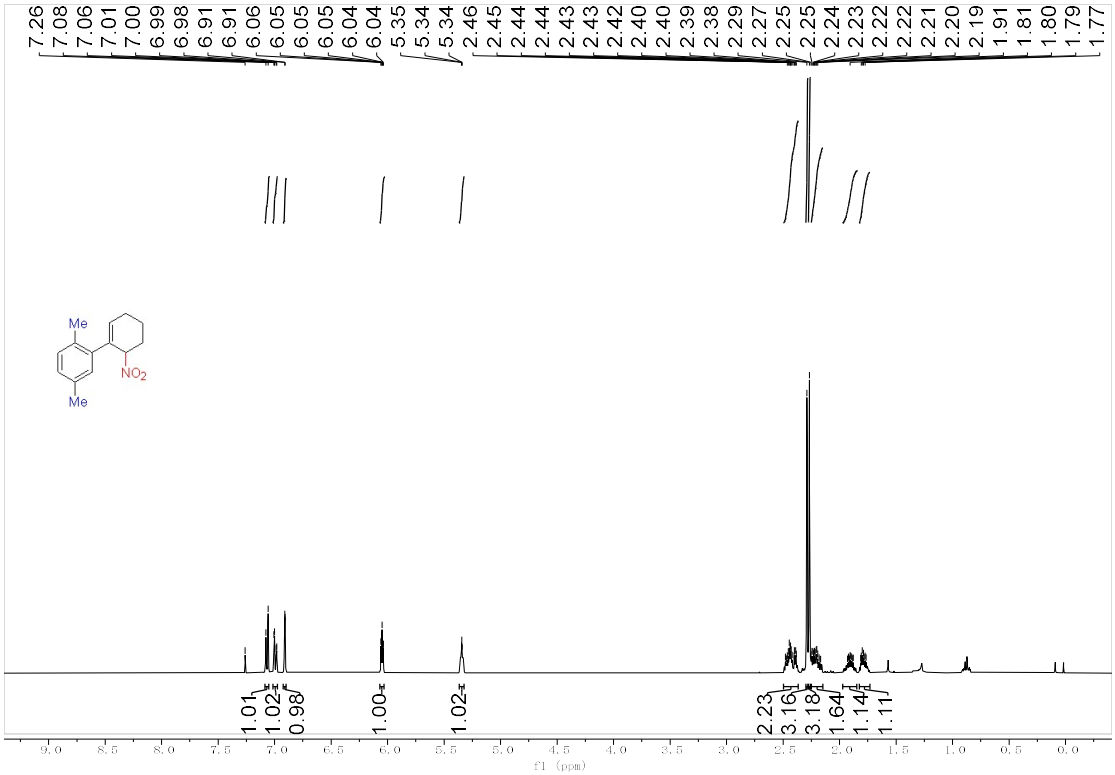
^{1}H NMR of 3s (400 MHz, $CDCl_3$)



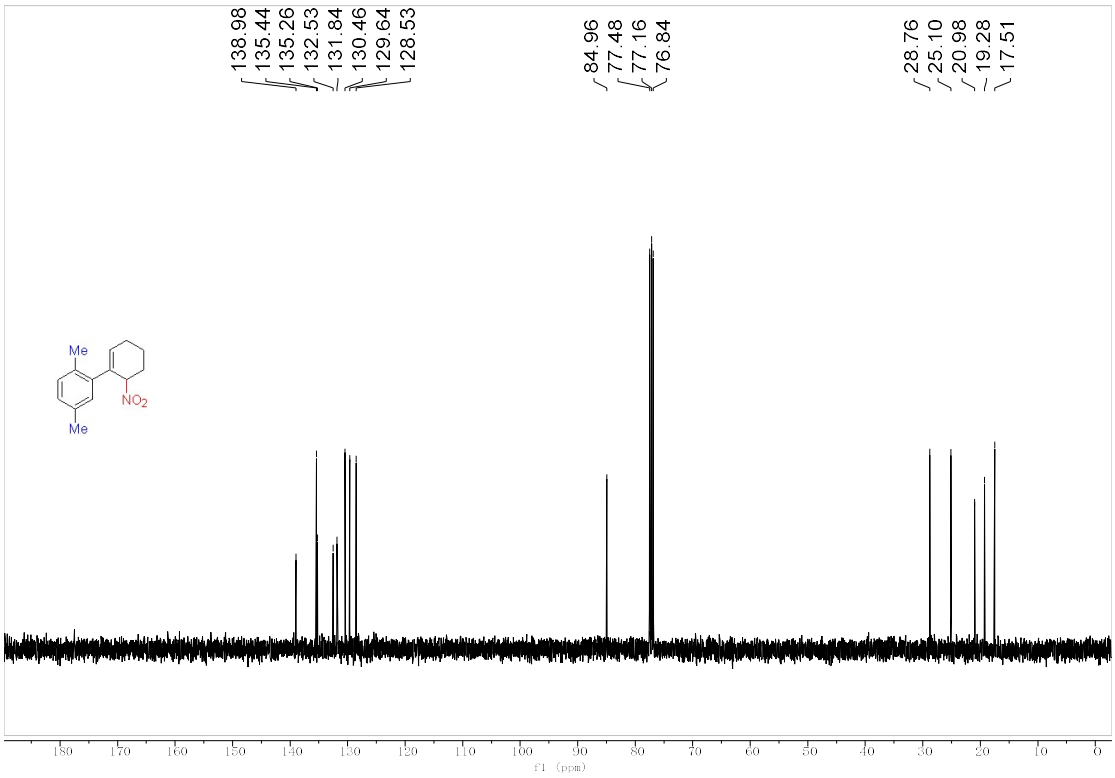
^{13}C NMR of 3s (100 MHz, $CDCl_3$)



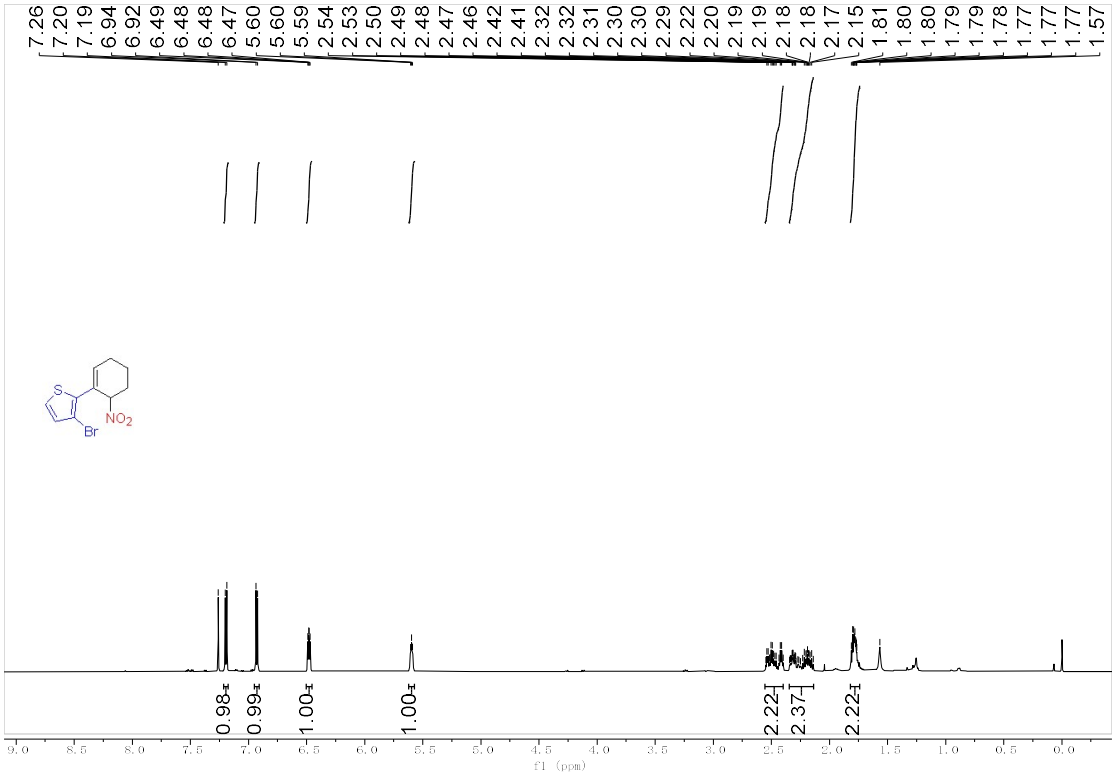
^{1}H NMR of 3t (400 MHz, $CDCl_3$)



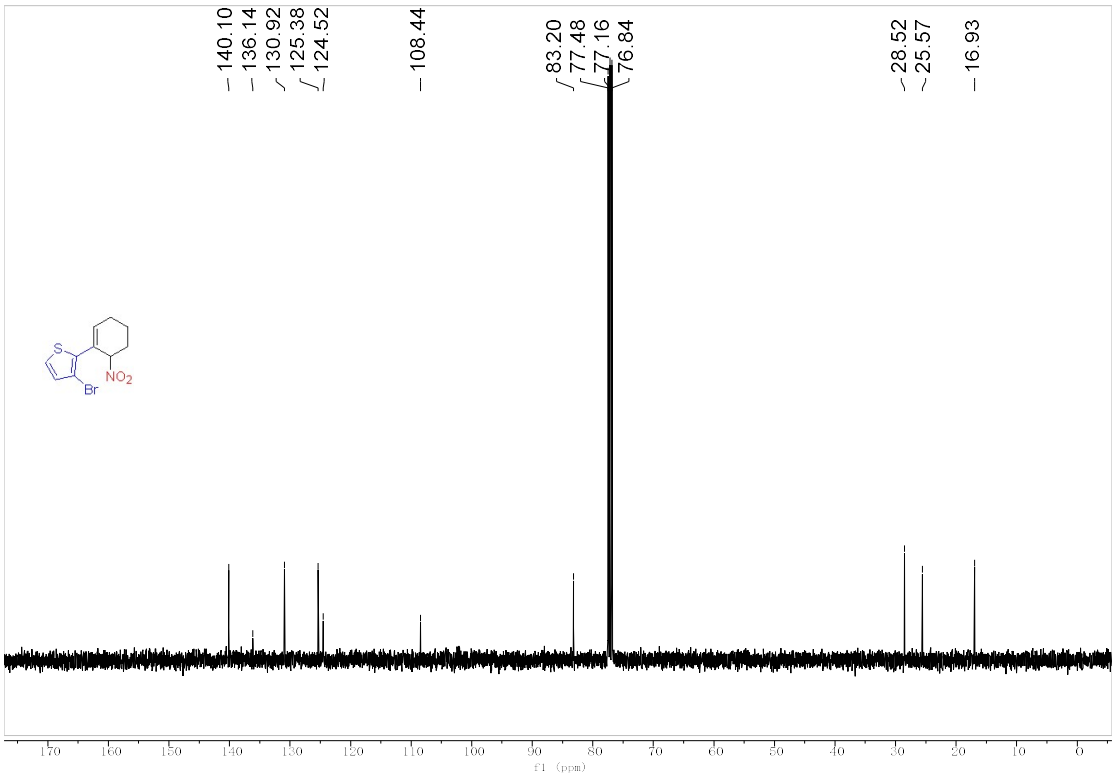
^{13}C NMR of 3t (100 MHz, $CDCl_3$)



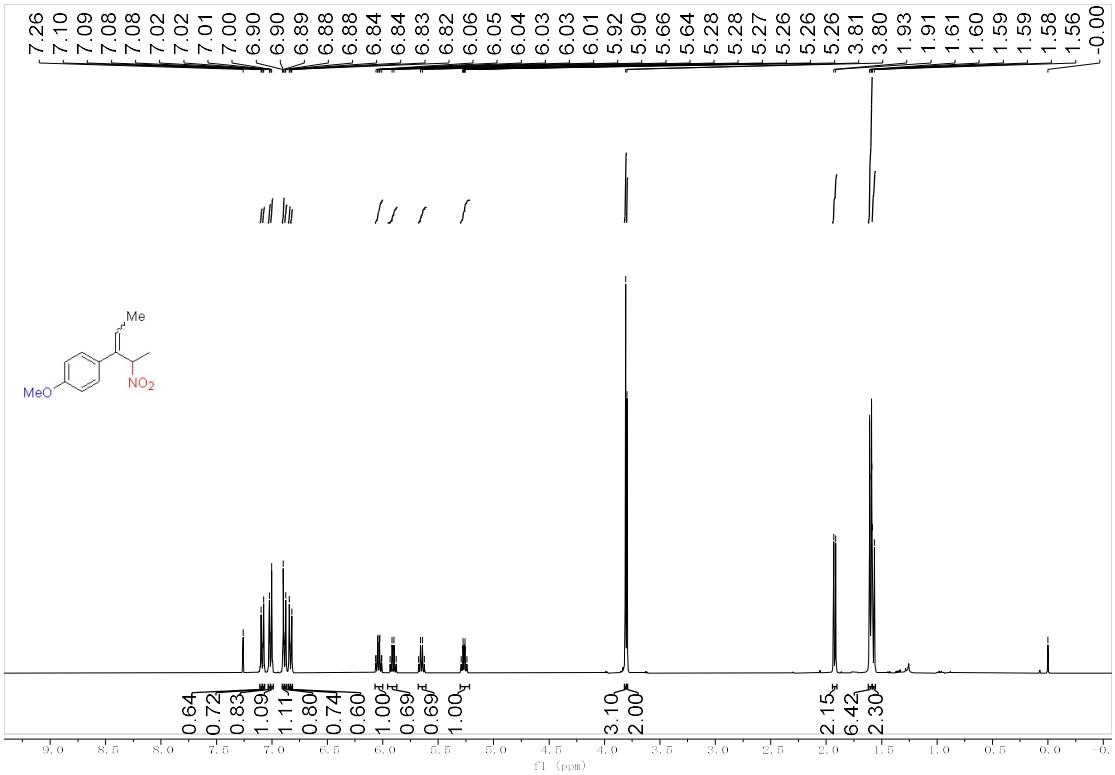
^{1}H NMR of 3u (400 MHz, $CDCl_3$)



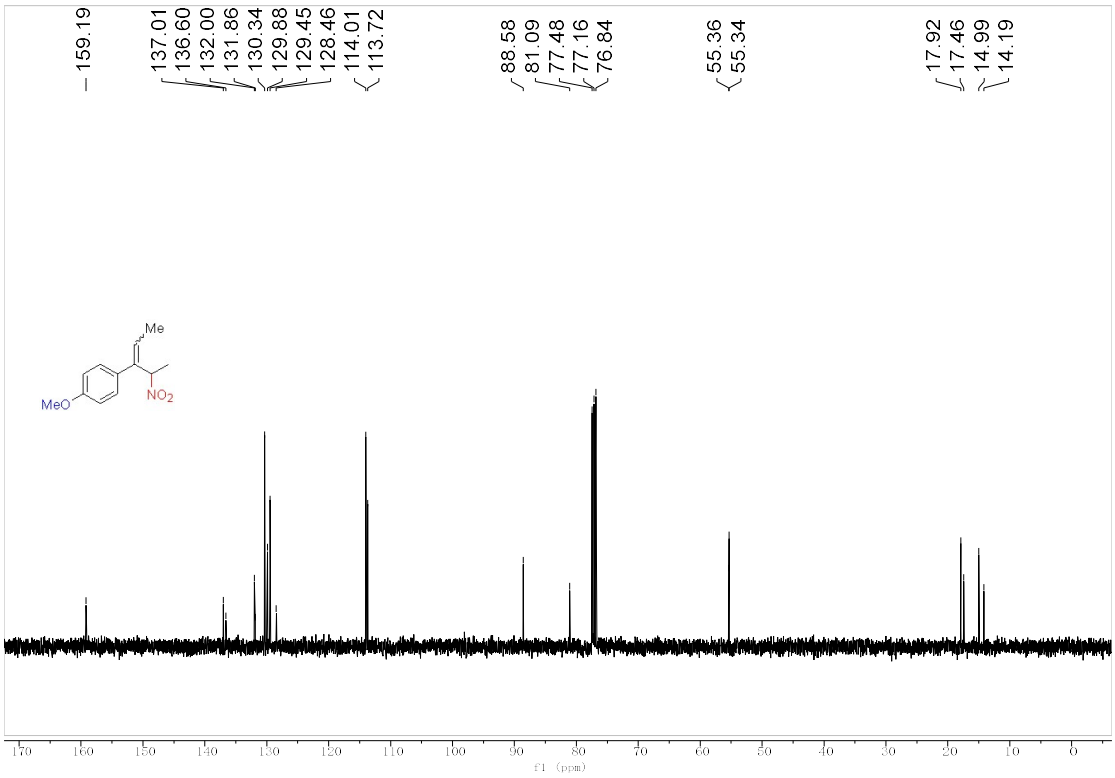
^{13}C NMR of 3u (100 MHz, $CDCl_3$)



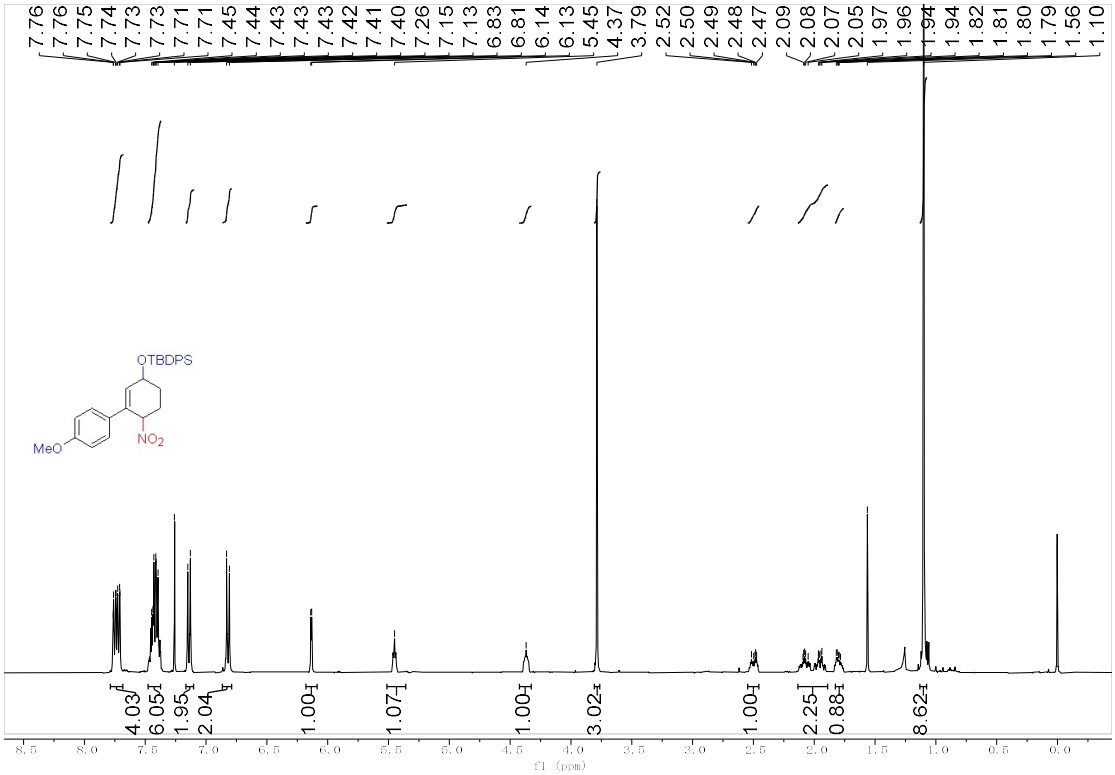
^{1}H NMR of 3v (400 MHz, $CDCl_3$)



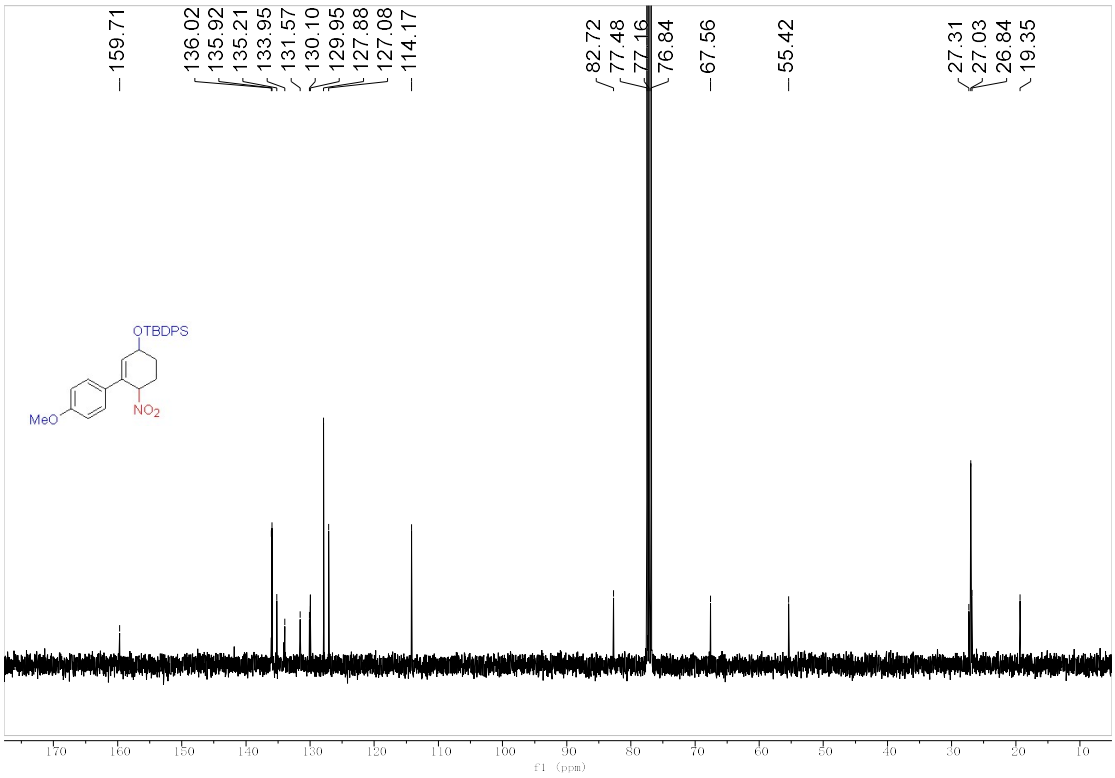
^{13}C NMR of 3v (100 MHz, $CDCl_3$)



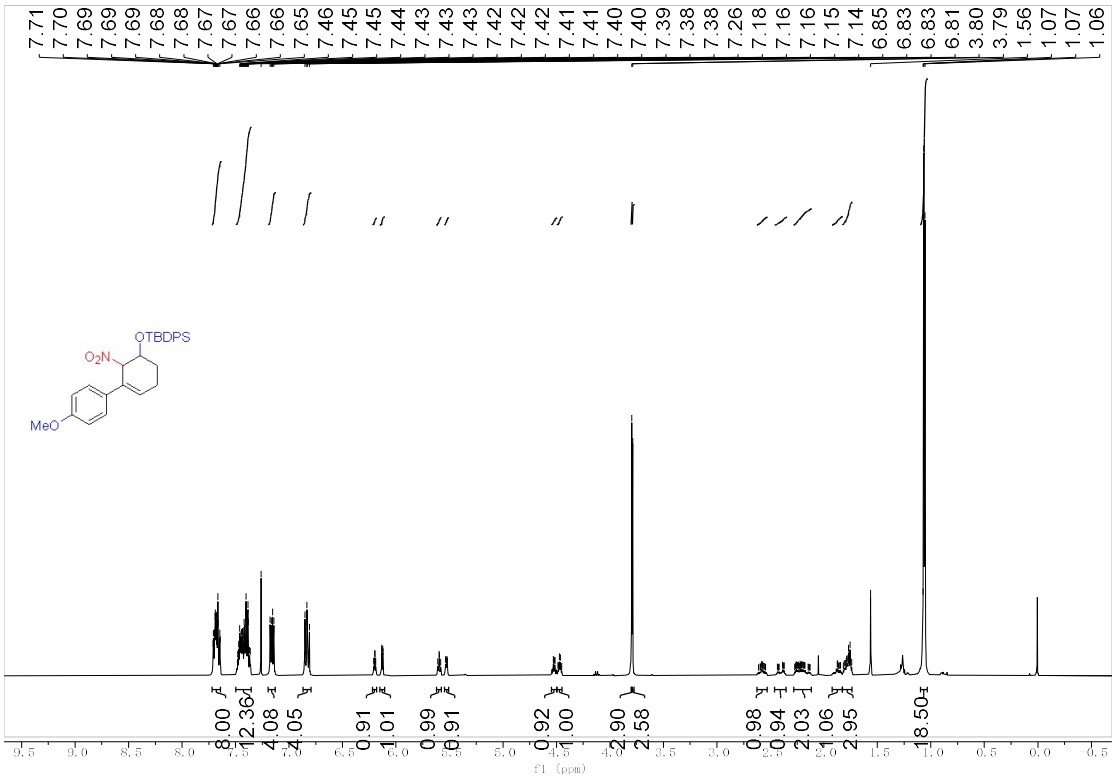
^{1}H NMR of 3w (400 MHz, $CDCl_3$)



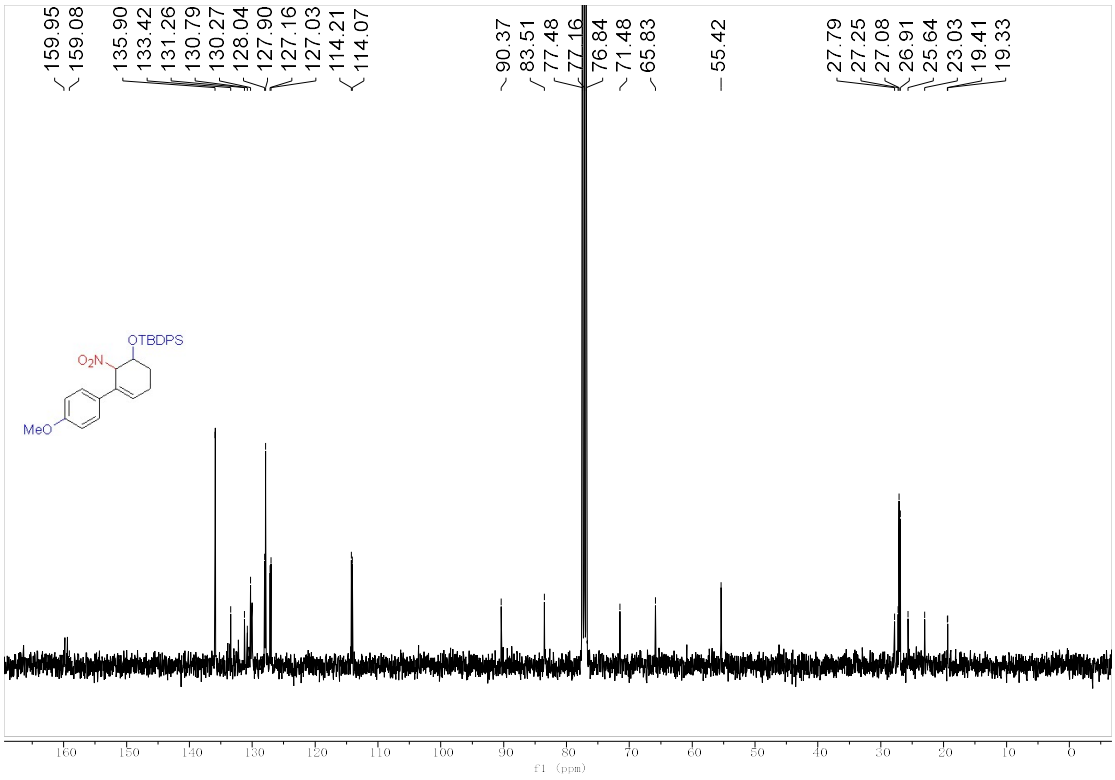
^{13}C NMR of 3w (100 MHz, $CDCl_3$)



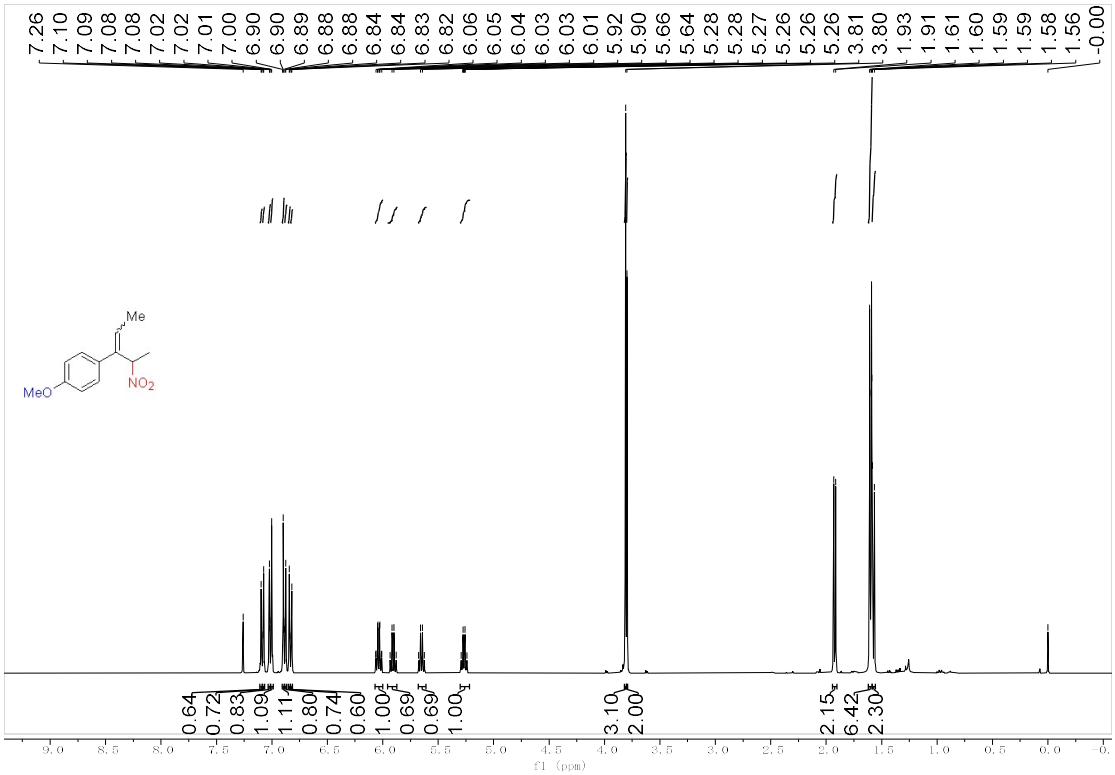
^{1}H NMR of 3w' (400 MHz, $CDCl_3$)



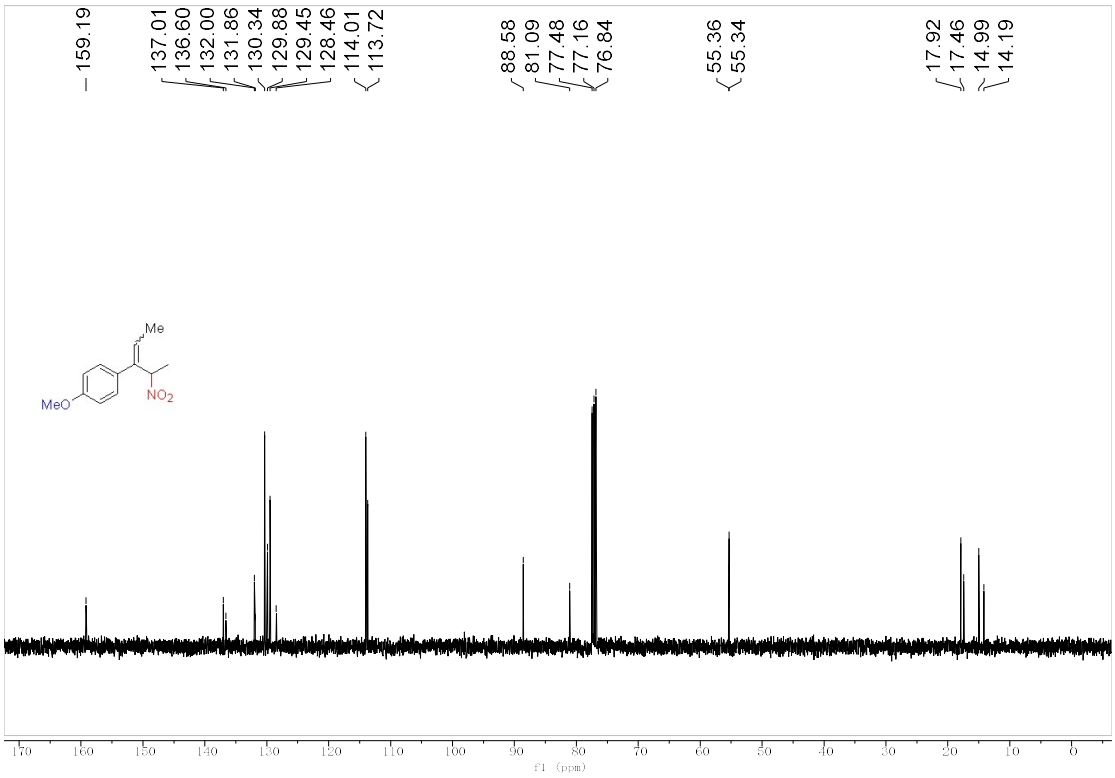
^{13}C NMR of 3w' (100 MHz, $CDCl_3$)



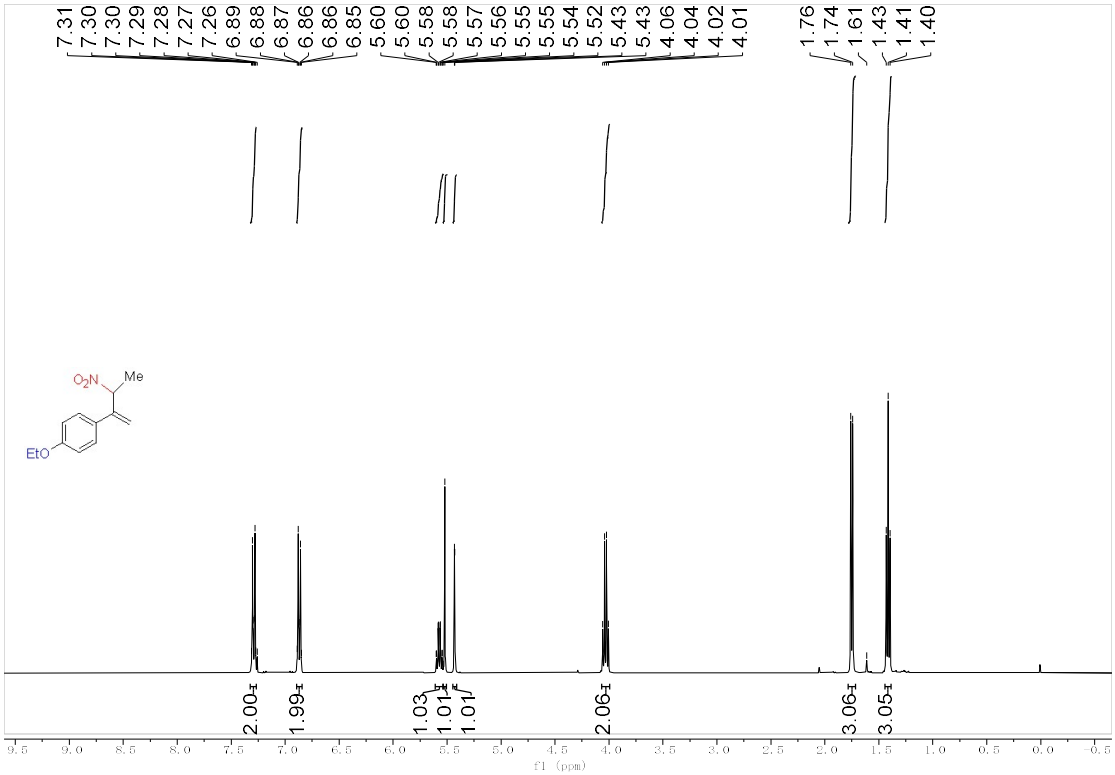
^{1}H NMR of 3x (400 MHz, $CDCl_3$)



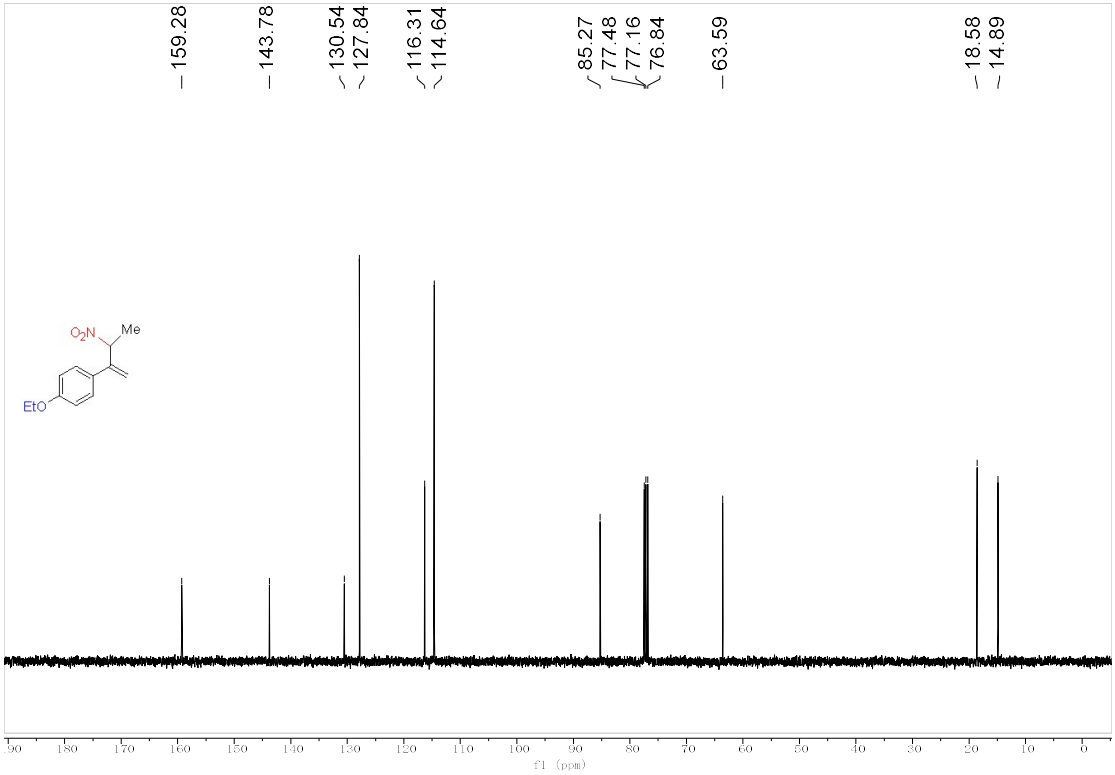
^{13}C NMR of 3x (100 MHz, $CDCl_3$)



^{1}H NMR of 3x' (400 MHz, $CDCl_3$)



^{13}C NMR of 3x' (100 MHz, $CDCl_3$)



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