Supporting information

Preparation of Gold Nanoparticles Decorated UiO-66-NH_2 Incorporated Epichlorohydrin and Cyclodextrin as Novel Efficient Catalyst in Cross Coupling and Carbonylative Reactions

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Supporting Information

These materials are available in PDF format.

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Author Contributions

Leila Mohammadi was responsible for synthesizing the UiO-66-NH_2 @ Epichlorohydrin/Cyclodextrin/Au-NPs catalyst, conducting the C-C coupling and Carbonylative Sonogashira reactions, and preparing and assembling the manuscript. Mohammad Reza Vaezi provided valuable contributions to the manuscript's editing.

Conflicts of Interest

The researchers report no conflicts of interest.

Data Availability

All pertinent data for this study are presented within the main article and its supplementary materials.

Acknowledgements

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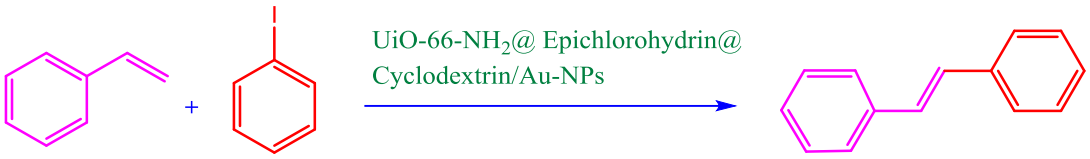
Contents:

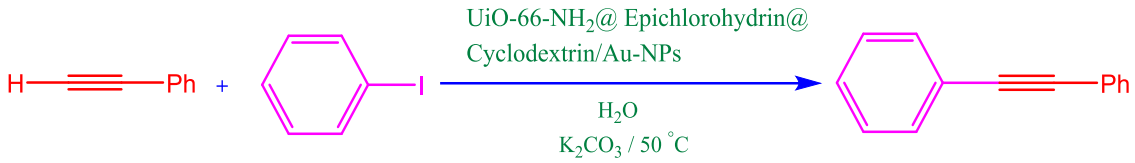
Table S1. The results of the optimization studies on the Heck reaction.



Entry	Catalyst (mg)	Solvent	T (°C)	Base	Time (min)	Yield (%)
1	5	H_2O	25	K_2CO_3	240	-
2	10	H_2O	25	K_2CO_3	240	10
3	20	H_2O	30	K_2CO_3	240	25
4	25	H_2O	45	K_2CO_3	240	35
5	30	H_2O	55	K_2CO_3	200	60
6	30	H_2O	80	K_2CO_3	90	98
7	30	H_2O	90	K_2CO_3	90	94
8	30	H_2O	100	K_2CO_3	90	90
9	30	H_2O	80	K_2CO_3	240	-
10	30	H_2O	90	KOH	240	76
11	30	DMSO	80	K_2CO_3	100	90
12	30	DMAc	80	K_2CO_3	100	90
13	30	NMP	80	K_2CO_3	100	90
14	30	DMF	80	K_2CO_3	100	87
15	30	H_2O	80	Et_3N	100	87
16	30	H_2O	80	Na_2CO_3	100	88
17	30	H_2O	80	NaOH	100	88
18	30	H_2O	80	NaOAc	100	80
19	30	CH_3CN	80	K_2CO_3	100	60
20	30	$C_6H_5CH_3$	80	K_2CO_3	100	65

Reaction condition: iodobenzene (1 mmol), styrene (1.1 mmol), base (2 mmol), solvent (3 ml)

Table S2. The results of the optimization studies on the Sonogashira reaction.



Entry	Catalyst (mg)	Solvent	T (°C)	Base	Time (min)	Yield (%)
1	10	H_2O	50	K_2CO_3	20	70
2	20	H_2O	50	K_2CO_3	20	95
3	25	H_2O	50	K_2CO_3	20	98
4	25	H_2O	25	K_2CO_3	20	35
5	25	H_2O	100	K_2CO_3	20	80
6	20	EtOH	50	K_2CO_3	20	83
7	25	H_2O	50	$NaCO_3$	20	15
8	25	H_2O	50	$CsCO_3$	20	85
9	25	H_2O	50	NaOH	20	70
10	25	H_2O	50	KOH	20	25

Reaction condition: Aryl halide (1 mmol), terminal alkynes (1.2 mmol), base (2 mmol), solvent (3 mL)

Table S3. The results of the optimization studies on the Carbonylative Sonogashira reaction.

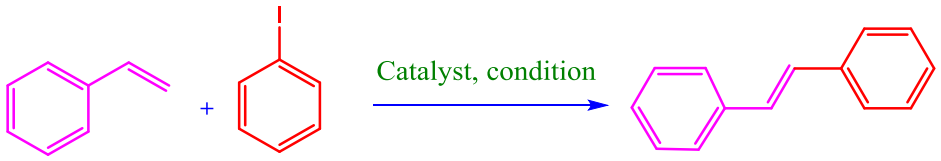
Entry	Base	Ligand	Solvent	Temperature (°C)	Yield (%)
1	Et_3N	DPPF	DMF	60	40
2	Et_3N	DPPF	toluene	60	42

3	Et_3N	DPPF	THF	60	37
4	K_2CO_3	DPPE	toluene	60	41
5	Et_3N	DPPE	DMF	60	41
6	Et_3N	DPPP	toluene	60	42
7	Et_3N	BINAP	DMF	60	40
8	Et_3N	DPPM	DMF	60	40
9	Et_3N	-	toluene	60	20
10	Et_3N	Xantphos	toluene	60	43
11	Et_3N	Pcy_3	DMF	60	38
12	Et_3N	Pcy_3	toluene	60	40
13	Et_3N	PPh_3	toluene	100	96
14	Et_3N	PPh_3	toluene	90	96
15	Et_3N	PPh_3	toluene	80	96
16	Et_3N	PPh_3	toluene	70	96
17	Et_3N	PPh_3	toluene	60	97
18	Et_3N	PPh_3	toluene	55	96
19	Et_3N	PPh_3	toluene	50	96
20	Et_3N	PPh_3	toluene	45	92
21	DBU	PPh_3	toluene	50	90
22	iPr_2NEt	PPh_3	toluene	50	89
23	K_2CO_3	PPh_3	toluene	50	92
24	Et_3N	PPh_3	-	50	75
25	Et_3N	PPh_3	CH_3CN	50	50
26	Et_3N	PPh_3	THF	50	45
27	Et_3N	PPh_3	DMF	50	40
28	Et_3N	-	toluene	50	20
29	-	PPh_3	toluene	50	40

Reaction Condition: iodobenzene (0.2 mmol),phenyl acetylene (0.4 mmol), proposed catalyst UiO-66-NH_2@ Epichlorohydrin@ Cyclodextrin @Au-NPs (30 mg), Ligand (10 mol%), base (3 mmol), HCOOH (2.0 mmol), Acetic anhydride (Ac_2O) (2 mmol), solvent (2.0 ml), 50°C, 8h.

DPPF = 1,1'-Bis (diphenylphosphino) ferrocene; DPPE = 1,2-Bis (diphenylphosphino) ethane; DPEphose = Bis (diphenylphosphino) butane; DPPE = 1,3-Bis (diphenylphosphino) propane; DPPPE = 1,5-Bis (diphenylphosphino) pentane; DPPM = Bis (diphenylphosphino) methane.

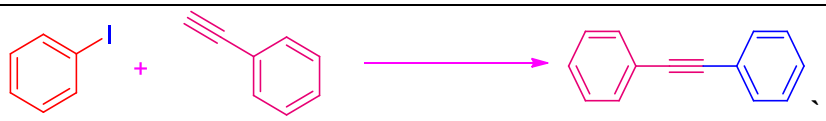
Table S4: Comparison of the catalytic performance of the proposed catalyst for Heck reaction with some related reports in the *literature*



Entry	Catalyst	Reaction condition	Time (h)	Yield %	Reference
1	GA-FSNP@Pd (0.47)	DMF: H_2O (2: 1), Cs_2CO_3, 110°C	0.5	95	[1]

2	Pd-biomagnetic (5)	DMF, TEA, 120°C	3	100	[2]
3	Pd/N-MCNPs	DMAN, TEA, 120°C	3	97.6	[3]
4	SBA-TMG[a] –Pd (0.05)	Solvent-free, 140°C	4	90	[4]
5	Fe_3O_4@PUNP[b]-Pd (0.1)	H_2O, K_2CO_3, reflux	12	trace	[5]
6	Pd/graphene oxide (0.54)	toluene, NEt_3, reflux	5	62	[6]
7	Pd/MCPPY	DMA, TBA, 120°C	3	97	[7]
8	Pd/HCN (0.255)	DMF, K_2CO_3, 120°C	1	100	[8]
9	Pd/Fe_3O_4@C (0.308)	DMF, K_2CO_3, 120°C	2	66	[9]
10	PdNs-PAMAM-g-MWCNTs (0.3)	NMP, K_2CO_3, 100°C	2.5	95	[10]
11	Pd-MNPSS (0.36)	K_2CO_3, H_2O, 100°C	4	90	[11]
12	$PdCl_2$ (1.5), TDTAT (3 W%)	K_2CO_3, H_2O, 80°C	6	96	[12]
13	hydrogel@Pd NPs	K_2CO_3, H_2O, 85°C	0/5	98	[13]
14	*h*-BN@Sal@Pd(OAc)$_2$ (0.05 mol)	H_2O, 90°C	3.5	92	[14]
15	PdL_n@*β*-CD (3)	K_2CO_3, H_2O, reflux	4	94	[15]
16	Pd-MPTA-1 (10 mg)	K_2CO_3, H_2O, 100°C	6	92	[16]
17	Pd@CSP (0.5), TBAB	NEt_3, H_2O, reflux	7	90	[17]
18	UiO-66-NH_2@Epichlorohydrin/CD/Au-NPs	K_2CO_3, H_2O, 80°C, 2h	1.5	98	This research

Table S5. Comparison of the Catalytic Performance of the Synthesized Catalyst for the Sonogashira Reaction with Existing Literature Reports



Entry	Catalyst	Reaction condition	Time (min)	Yield (%)	Ref.
1	Pd-B_3 -Azo_4 (0.08 mol%)	PL-XQ 350 W, Xe lamp with 420 nm UV cut-off, hν	3	94	[18]
2	(Pd^{II}–NHCs)$_n$@$nSiO_2$ (0.54 mol%)	Et_3N, H_2O, 90°C	0.15	92	[19]
3	Pd(II)–acylthiourea complex	KO^{t}Bu, DMAc, 80°C	3	99	[20]
4	Pd@PTC-POP (0.3 mol%)	Et_3N, H_2O, 100°C	2	99	[21]

5	Pd-catalyst CuI/trimethylamine (5 mol%)	Microwave, H_2O, 100°C	2	60	[22]
6	Pd@CS/PEO (7.0 mol%)	DMF, glycol, K_2CO_3, 110°C	5	93	[23]
7	Pd@ONO-IL (0.15 m0l%)	Piperidine , DMF, 70°C	2	94	[24]
8	Pd-NPs/CENFs (0.06 mol%)	$Na_3PO_4.12H_2O$, *i*-PrOH, reflux	3	88	[25]
9	Pd-LHMS-3 (0.03 g)	DMF, Hexamine, N_2 atm, 120°C	14	80	[26]
10	Pd(0)/MCoS-1 (0.02 g)	H_2O, Et_3N, 80°C	6	94	[27]
11	Ru/Al_2O_3 (5 mol%)	CH_3CN, Et_3N, 90°C	24	85	[28]
12	UiO-66-NH_2@Epichlorohydrin/ Cyclodextrin/Au-NPs	K_2CO_3, H_2O, 50°C	20	99	This research

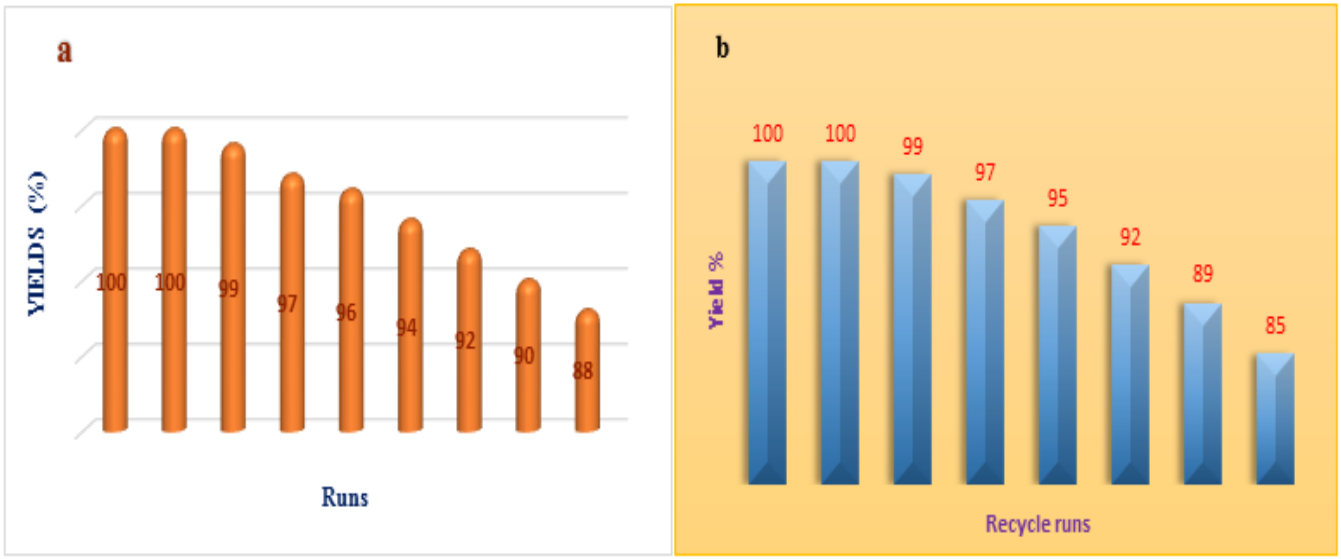
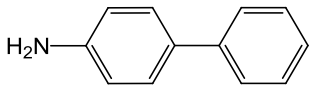


Figure S1. Reusability of catalyst for a) Heck and b) Sonogashira reaction.

Suzuki derivatives

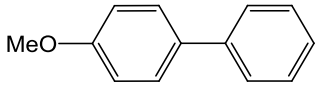
4-amine-[1,1'-biphenyl]



m.p. = 67-68 ºC.

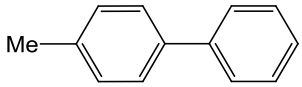
^{1}HNMR (400 MHz, DMSO): δ = 5.23 (2H, NH_2), 6.67 (2H, d, J = 8.3), 7.20 (1H, t, J = 7.2), 7.34-7.38 (4H, m), 7.53 (2H, d, J = 7.6). ^{13}CNMR (100 MHz, DMSO): δ = 114.3, 125.4, 125.7, 127.2 (4 × CH Ar), 127.5 (Ar), 128.8 (CH, Ar), 140.7, 148.4 (Ar). m/z: 170.1

4-Methoxy-1,1'-biphenyl



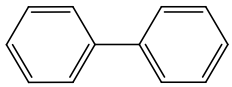
White waxy solid, yield 90%, ^{1}HNMR (500 MHz, $CDCl_3$): 7.57-7.52 (m, 4H), 7.41 (t, J = 8.0 Hz, 2H), 7.30 (t, J = 8.0 Hz, 1H), 6.98 (d, J = 8.5 Hz, 2H), and 3.86 (s, 3H).

4-methyl-1,1'-bipheny



white yellow solid in 92% yield, MP: 46-48 °C; ^{1}HNMR (500 MHz, $CDCl_3$): δ 7.61 (s, 1H), 7.51 (t, J = 7.8 Hz, 1H), 7.39 (m, 3H), 7.34 (m, 1H), 7.24 (t, J = 7.2 Hz, 1H), 7.16 (t, J = 8.1 Hz, 2H), 2.31 (s, 3H); ^{13}CNMR (126 MHz, $CDCl_3$): δ 141.19, 139.80, 138.34, 137.05, 136.72, 129.48, 129.01, 128.74, 128.21, 127.38, 127.01, 126.83, 125.73, 21.11; FTIR (cm^{-1}): 2918, 2318, 1743, 1521, 1325, 1114, 802; GC-MS m/z: 168.

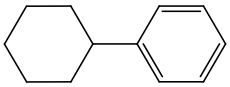
Biphenyl



white solid in 94% yield, MP: 69-71 °C

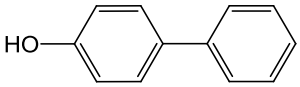
^{1}HNMR (500 MHz, $CDCl_3$): δ 7.59 (d, J =7.7 Hz, 4H), 7.44 (t, J = 7.7 Hz, 4H), 7.34 (t, J = 7.4 Hz, 2H); ^{13}CNMR (126 MHz, $CDCl_3$): δ141.29, 128.82, 127.27; FTIR (cm^{-1}): 2358, 1510, 1029, 580; GC-MS (EI, 70ev) m/z: found: 154 ($C_{12}H_{10}$).

Cyclohexylbenzene



Colorless oil in 73% yield ^{1}HNMR (400 MHz, chloroform-d) δ = 7.31 – 7.25 (m, 2H), 7.23 – 7.13 (m, 3H), 2.49 (m, 1H), 1.92 – 1.82 (m, 4H), 1.74 (m, 1H), 1.51 – 1.34 (m, 4H), 1.25 (m, 1H); ^{13}CNMR (100 MHz, chloroform-d) δ = 148.2, 128.4, 127.0, 125.9, 44.8, 34.6, 27.1, 26.3.

4-hydroxyl-[1,1'-biphenyl]

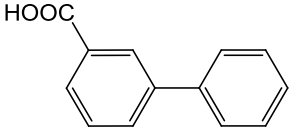


m.p. = 161-162 °C.

^{1}HNMR (400 MHz, DMSO): δ = 6.85 (2H, d, J = 8.5), 7.27 (1H, t, J = 7.4), 7.40 (2H, t, J = 7.4), 7.48

(2H, d, J = 8.5), 7.57 (2H, d, J = 8.5), 9.58 (1H, br s, OH). 13 C NMR (100 MHz, DMSO): δ = 115.8, 126.0, 126.4, 127.8, 128.8 (5 × CH, Ar), 131.0, 140.3, 157.2 (Ar). m/z: 169.1.

3-carboxylic acid-[1,1'-biphenyl]

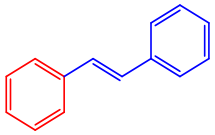


m.p. = 160-161 °C. ^{1}HNMR (400 MHz, DMSO): δ = 7.42 (1H, tt, J = 7.3, 1.1), 7.50 (2H, t, J = 7.3), 7.58 (1H, t, J = 7.7), 7.66 (2H, m), 7.87 (1H, dq, J = 7.7, 1.4), 8.14 (1H, dt, J = 7.7, 1.4), 8.40 (1H, t, J = 1.4).

^{13}CNMR (100 MHz, DMSO): δ = 127.2, 127.8, 128.85, 128.93, 128.96, 128.99 (CH, Ar), 129.8 (Ar), 132.4 (CH Ar), 139.9, 141.6 (2 × CH Ar), 172.3 (C=O). m/z: 197.0

Heck derivatives

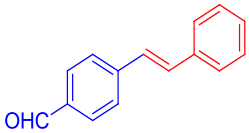
Trans-1, 2-diphenylethene (*E*-Stilbene)



White solid, Mp. 123-124°C (lit. 123.9-124.6°C)

GC–MS (m/z): 180; ^{1}HNMR (400 MHz, $CDCl_3$) δ 7.07-7.08 (s, 2H), 7.21-7.23 (m, 2H), 7.32- 7.34 (m, 4H,) 7.46-7.48 (m, 21H) ppm. ^{13}CNMR (100 MHz, $CDCl_3$) δ 122.1, 123.2, 124.3, 132.9.

(*E*)-4-styrylbenzaldehyde

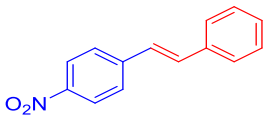


White solid, Mp. 116°C (lit. 116.0-116.8°C)

GC–MS (m/z): 208; ^{1}HNMR (400 MHz, $CDCl_3$): δ = 9.97 (s, 1H), 7.84 (d, J=8.0 Hz, 2H), 7.62 (d, J = 8.0 Hz, 2H), 7.52 (d, J= 7.6 Hz, 2H), 7.36 (t, J = 7.6 Hz, 2H), 7.28 (t, J= 7.6 Hz, 1H), 7.22 (d, J = 5.2 Hz, 1H), 7.11 (d, J=16.4 Hz, 1H).

^{13}CNMR (100 MHz, $CDCl_3$): δ = 191.5, 143.4, 136.5, 135.3, 132.2, 130.2, 128.8, 128.4, 127.3, 126.9. MS (EI) m/z: 208, 179, 165, 152, 102, 89, 76, 63, 51.

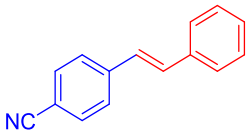
(*E*)-4-Nitrostilbene



Yellow solid, Mp. 154-155°C (lit. 154.2-155.0°C)

GC–MS (m/z): 225; ^{1}HNMR (400 MHz, $CDCl_3$) δ: 8.22 (d. *J* = 8.6 Hz, 2H, Ar-H), 7.63 (d, *J* = 8.7 Hz, 2H, Ar-H), 7.55 (d, *J* = 7.7 Hz, 2H, Ar-H), 7.40 (t, *J* = 7.6 Hz, 2H, Ar-H), 7.36-7.31 (m, 1H, Ar-H), 7.27 (d, *J* = 16.3 Hz, 1H, CH=), 7.14 (d, *J* = 16.3, 1H, Hz, CH=). ^{13}CNMR (100 MHz, $CDCl_3$), δ = 125.2, 127.4, 128.0, 128.2, 130.0, 134.4, 137.3, 145.0, 147.8 ppm.

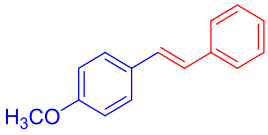
(*E*)-4-Styrylbenzonitrile



White solid, Mp. 114-115°C (lit. 114.9-115.2°C)

GC–MS (m/z): 205; ^{1}HNMR (400 MHz, $CDCl_3$): δ 7.09 (d, 1H, *J* = 16.0 Hz), 7.22 (d, 1H, *J* = 16.0 Hz), 7.31-7.34 (t, 1H, *J* = 4.0 Hz), 7.38-7.41 (t, 2H, *J* = 4.0 Hz), 7.54 (d, 2H, *J* = 7.2 Hz), 7.58 (d, 2H, *J* = 8.4 Hz), 7.64 (d, 2H, *J* = 8.4 Hz) ppm; ^{13}CNMR (100 MHz, $CDCl_3$), δ 111.7, 120.2, 127.8, 128.0, 128.1, 129.8, 130.0, 133.5, 133.6, 137.4, 142.9 ppm.

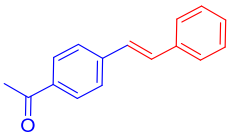
(*E*)-1-methoxy-4-styrylbenzene



White solid, Mp. 135-136°C (lit. 135.3-135.9°C)

GC–MS (m/z): 210; ^{1}H-NMR (400 MHz, $CDC1_3$) δ (ppm): 3.70 (3H, s), 6.78 (1H, d, *J* = 8.75 Hz), 6.90 (1H, d, *J* = 10.75 Hz), 7.00-7.38 (9H, m). ^{13}C-NMR (100 MHz, $CDCl_3$) δ (ppm): 55.3, 114.2, 126.3, 126.6, 127.25, 127.8, 128.2, 128.7, 130.2, 137.7, 159.3.

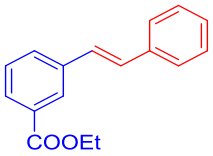
(*E*)-1-(4-styrylphenyl) ethanone



White solid, Mp. 141-142°C (lit. 141.3-142°C)

GC–MS (m/z): 222; ^{1}HNMR (400 MHz, $CDCl_3$): δ 7.97-7.95 (d, 2H), 7.58-7.57 (d, 2H), 7.53-7.52 (d, 2H), 7.40-7.36 (t, 2H), 7.29-7.25 (t, 1H), 7.22-7.20 (d, 1H), 7.12 (d, 1H), 2.64 (s, 3H), ^{13}CNMR (100 MHz, $CDCl_3$): δ 197.6, 142.1, 136.7, 136.1, 131.5, 128.9, 128.8, 128.4, 127.6, 126.8, 126.5, 26.8.

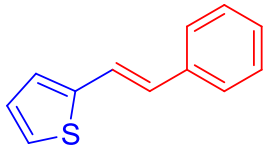
(*E*)-Ethyl-3-styrylbenzoate



Oily liquid

GC–MS (m/z): 252; ^{1}HNMR ($CDCl_3$, 400 MHz): δ (ppm) 8.22 (s, 1H), 7.95 (d, *J* = 7.7 Hz, 1H), 7.70 (d, *J* = 7.7 Hz, 1H), 7.55 (d, *J* = 7.4 Hz, 2H), 7.46–7.27 (m, 4H), 7.21 (d, *J* = 16.0 Hz, 1H), 7.15 (d, *J* = 16.0 Hz, 1H), 4.43 (q, *J* = 7.1 Hz3H), 1.44 (t, *J* = 7.1 Hz); ^{13}CNMR ($CDCl_3$, 100 MHz): δ (ppm) 166.6, 137.7, 137.0, 131.0, 130.7, 129.9, 128.8, 128.7, 128.5, 128.0, 127.6, 127.5, 126.7.

(*E*)-2-styrylthiophene



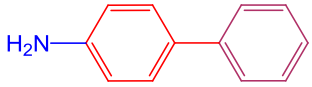
white solid, MP. 111-112°C (lit. 112°C)

GC–MS (m/z): 186; ^{1}H NMR ($CDCl_3$, 400 MHz): δ (ppm) 7.49 (d, *J* = 7.5 Hz, 2H), 7.37 (t, *J* = 7.5 Hz, 2H), 7.28 (d, *J* = 5.1 Hz, 1H), 7.24 (d, *J* = 16 Hz, 1H), 7.21 (d, *J* = 4.9 Hz, 1H), 7.09 (d, *J* = 3.4 Hz, 1H), 7.03 (t, *J* = 4 Hz, 1H), 6.95 (d, *J* = 16.2 Hz, 1H); ^{13}CNMR ($CDCl_3$, 100 MHz): δ (ppm) 142.9, 137.0, 128.7, 128.4, 127.6, 126.3, 126.1, 124.3, 121.8.

Spectrums

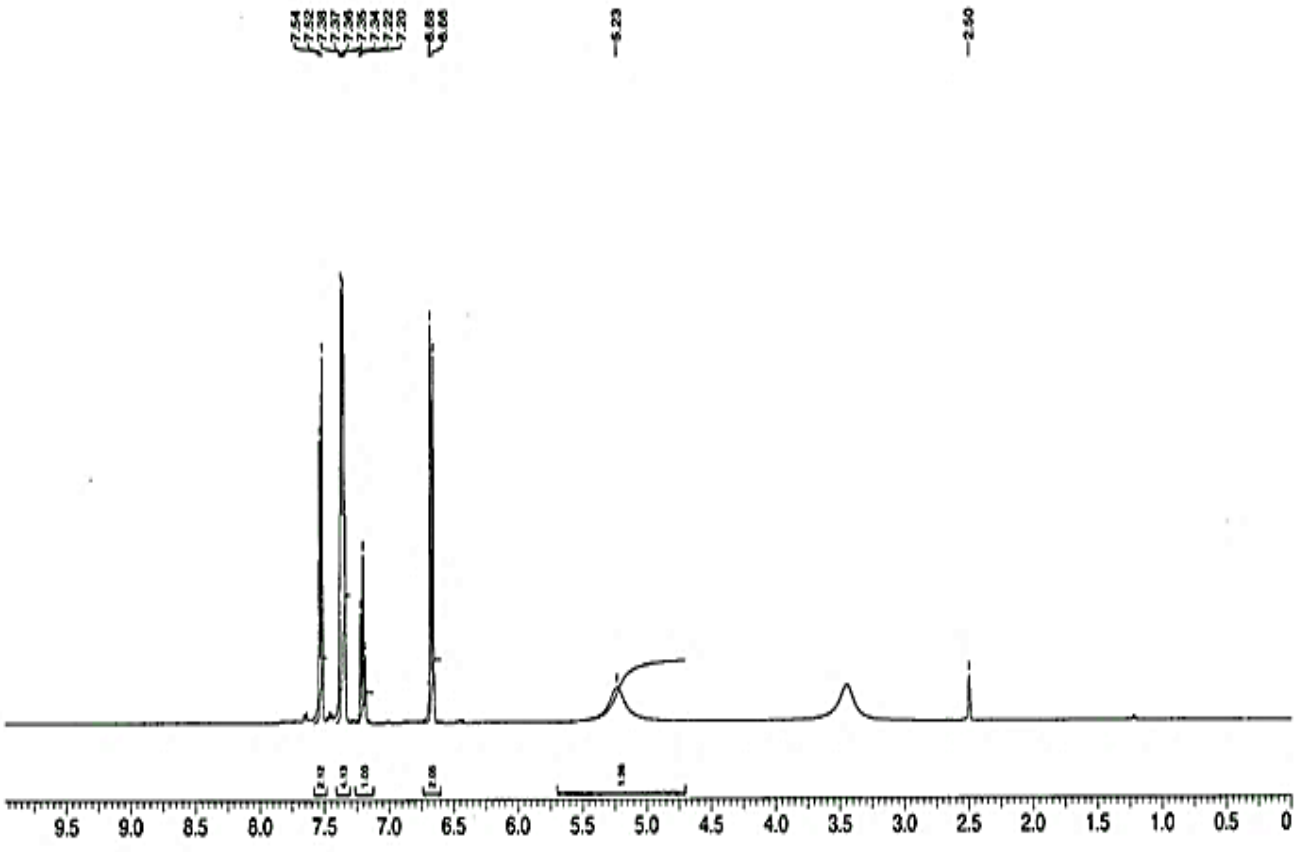
Suzuki derivatives

4-amine-[1,1'-biphenyl]

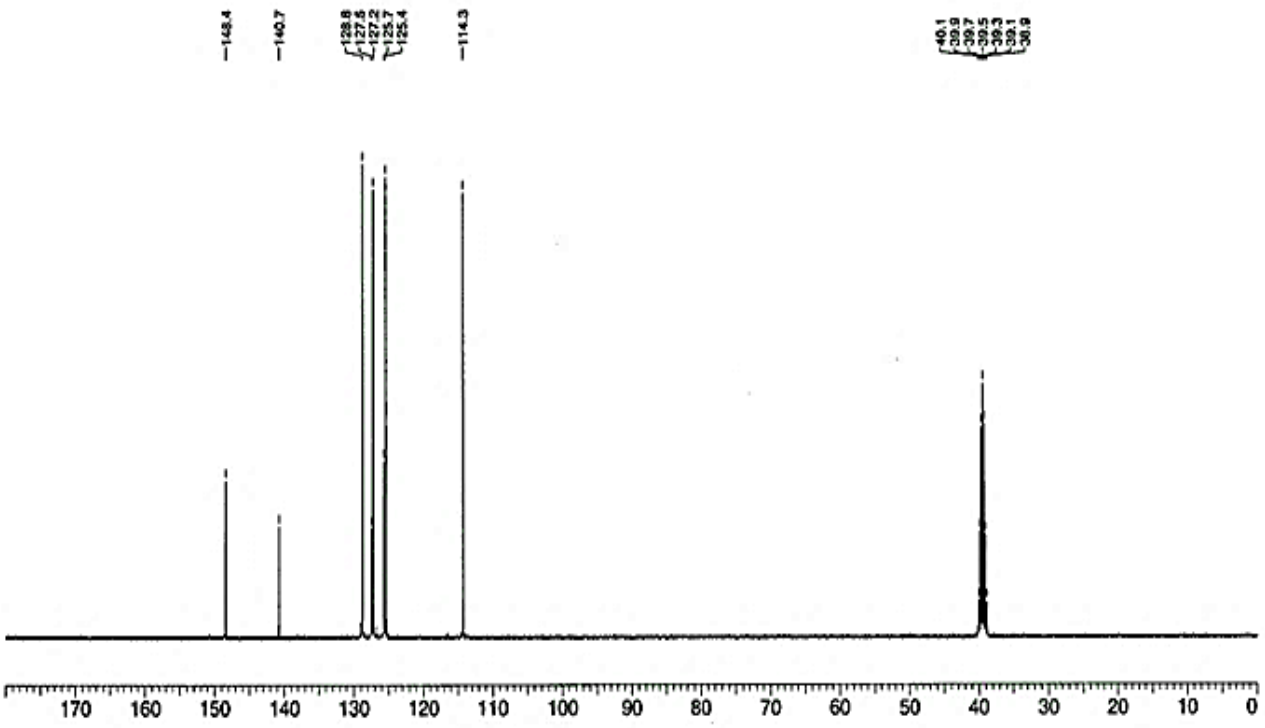


m.p. = 67-68 ºC.

^{1}HNMR (400 MHz, DMSO): δ = 5.23 (2H, NH_2), 6.67 (2H, d, J = 8.3), 7.20 (1H, t, J = 7.2), 7.34-7.38 (4H, m), 7.53 (2H, d, J = 7.6). ^{13}CNMR (100 MHz, DMSO): δ = 114.3, 125.4, 125.7, 127.2 (4 × CH Ar), 127.5 (Ar), 128.8 (CH, Ar), 140.7, 148.4 (Ar). m/z: 170.1

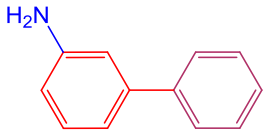


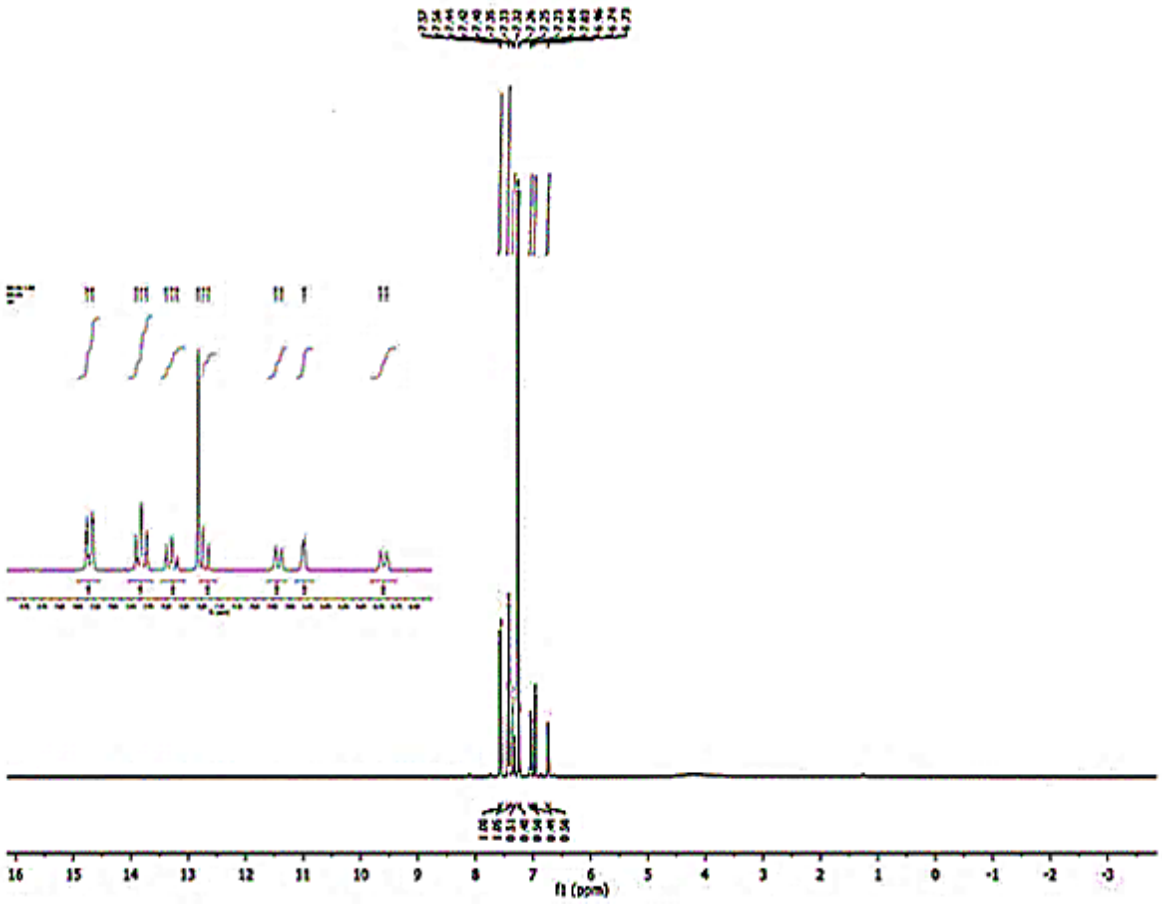
S1. The ^{1}HNMR of 4-amine-[1,1'-biphenyl]



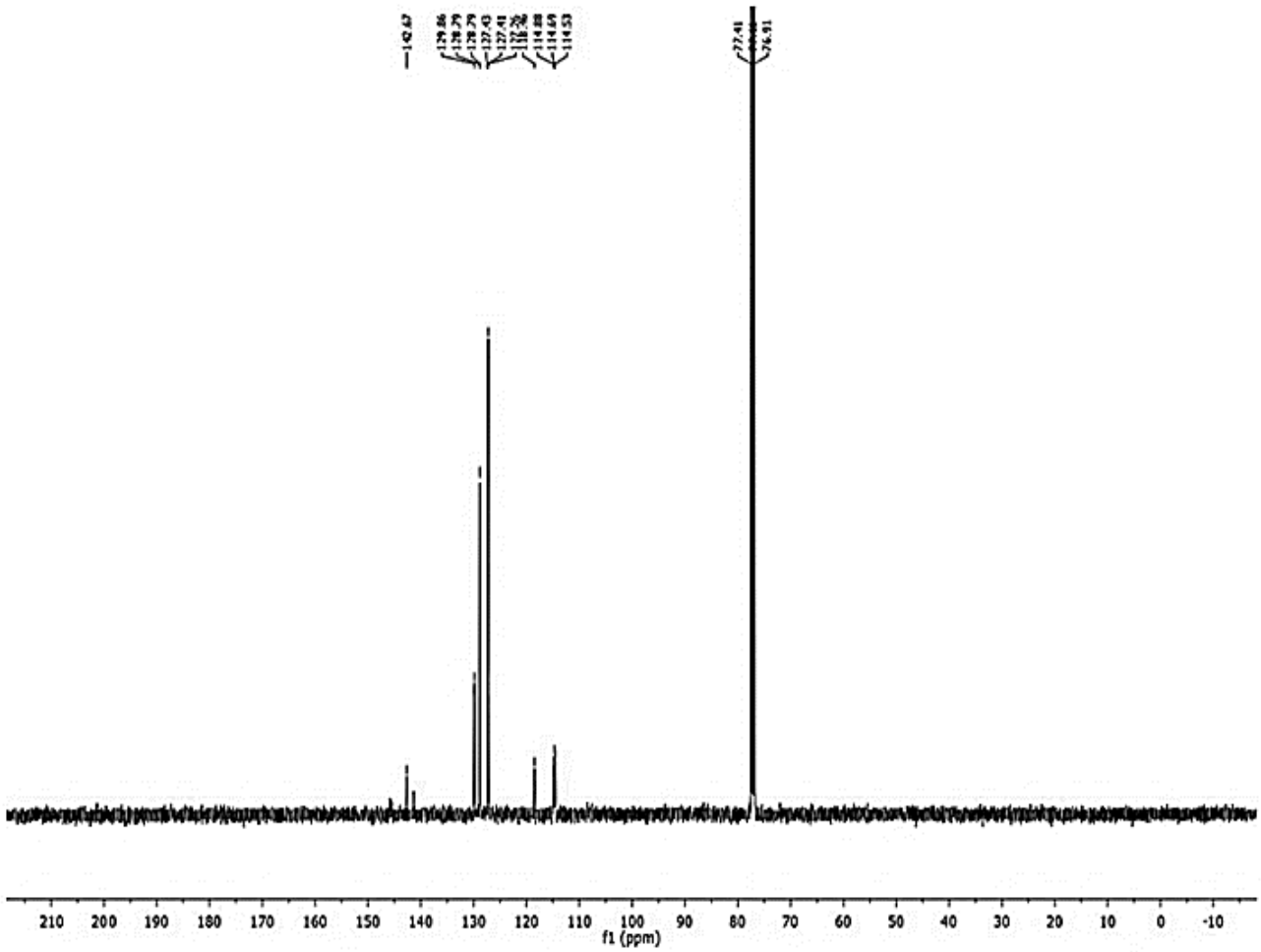
S2. The ^{13}CNMR of 4-amine-[1,1'-biphenyl]

3-amine-[1,1'-biphenyl]



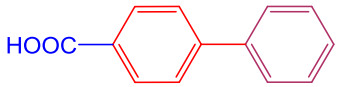


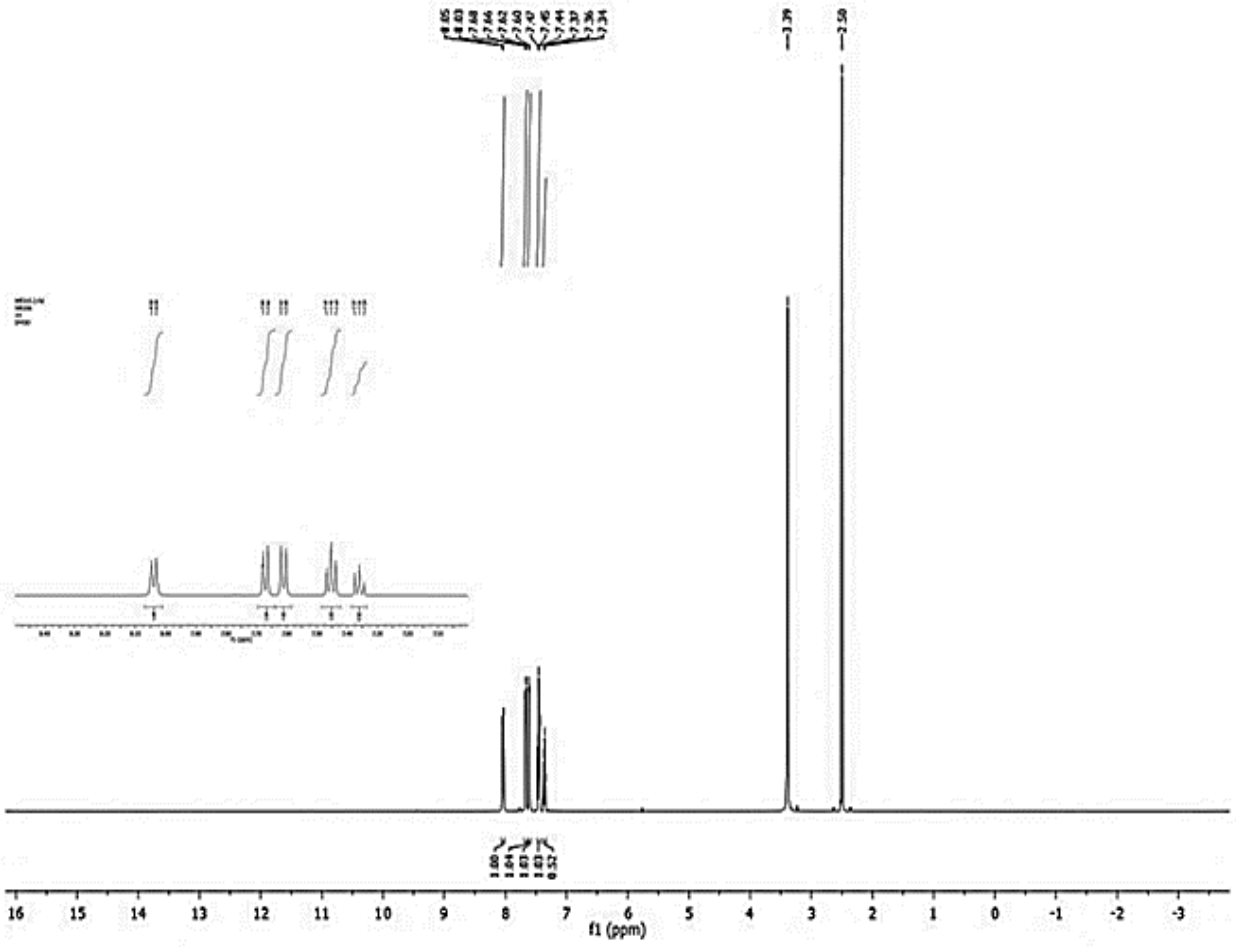
S3. The ^{1}HNMR of 3-amine-[1,1'-biphenyl]



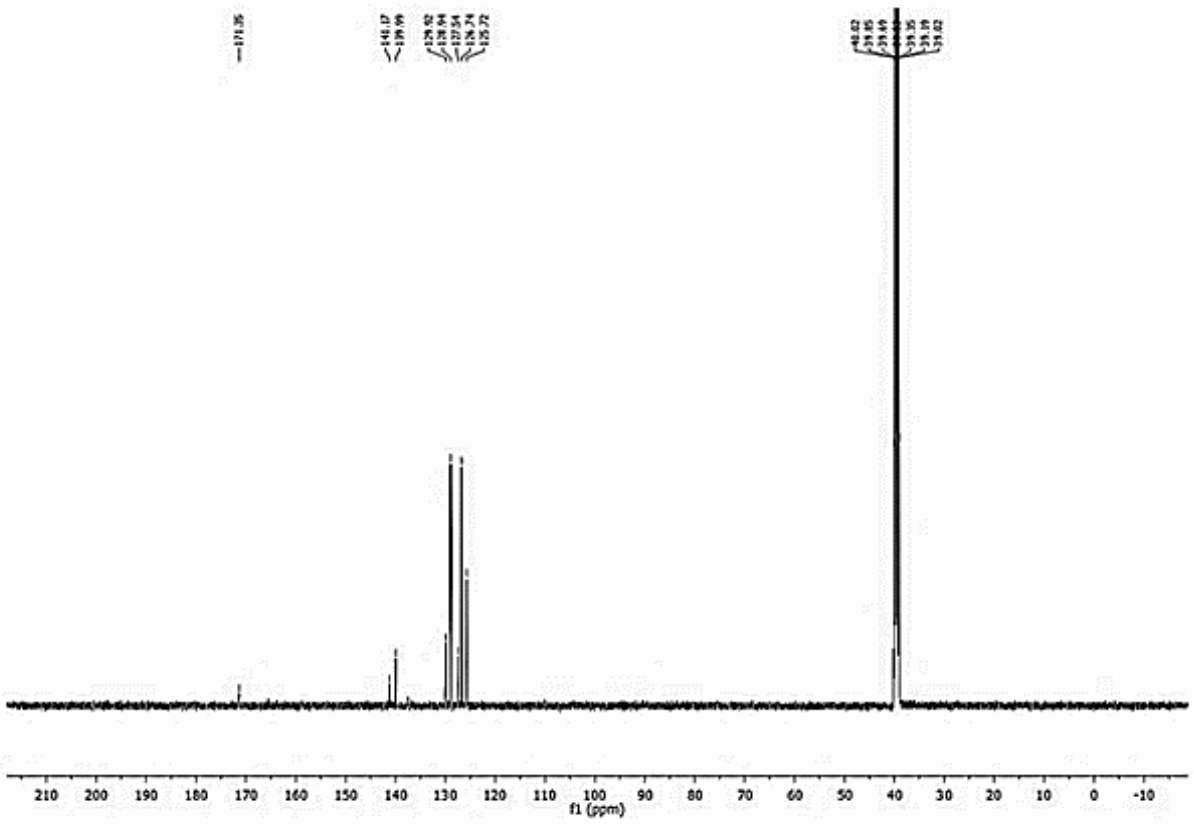
S4. The ^{13}CNMR of 3-amine-[1,1'-biphenyl]

4-carboxylic acid-[1,1'-biphenyl]



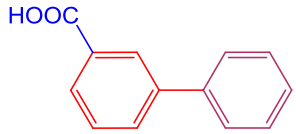


S5. The ^{1}HNMR of 4-carboxylic acid-[1,1'-biphenyl]



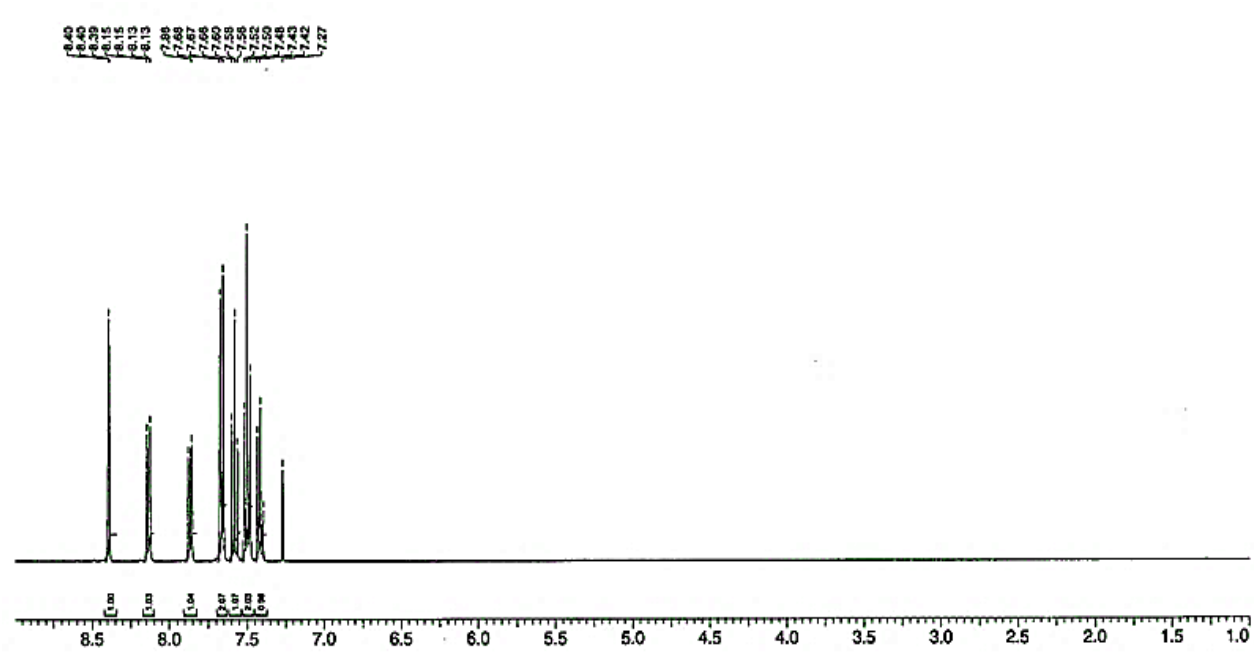
S6. The ^{13}CNMR of 4-carboxylic acid-[1,1'-biphenyl]

3-carboxylic acid-[1,1'-biphenyl]

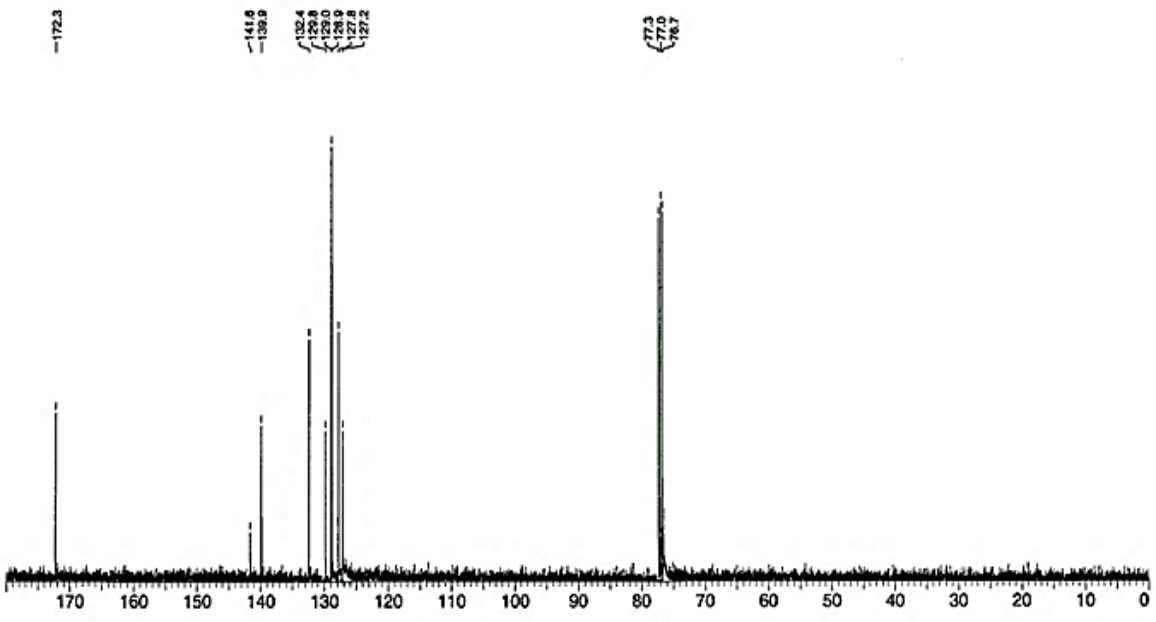


m.p. = 160-161 °C. ^{1}HNMR (400 MHz, DMSO): δ = 7.42 (1H, tt, J = 7.3, 1.1), 7.50 (2H, t, J = 7.3), 7.58 (1H, t, J = 7.7), 7.66 (2H, m), 7.87 (1H, dq, J = 7.7, 1.4), 8.14 (1H, dt, J = 7.7, 1.4), 8.40 (1H, t, J = 1.4).

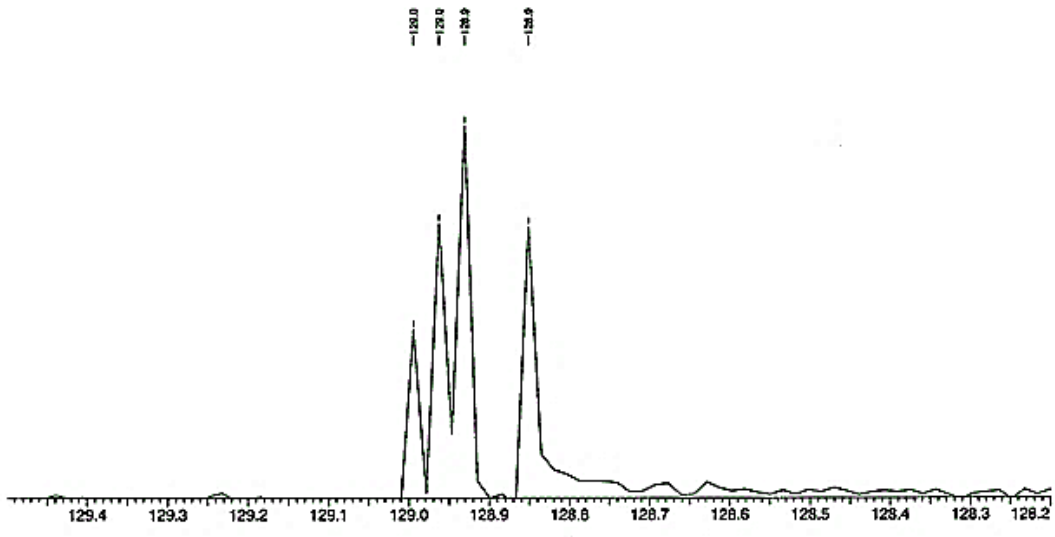
^{13}CNMR (100 MHz, DMSO): δ = 127.2, 127.8, 128.85, 128.93, 128.96, 128.99 (CH, Ar), 129.8 (Ar), 132.4 (CH Ar), 139.9, 141.6 (2 × CH Ar), 172.3 (C=O). m/z: 197.0



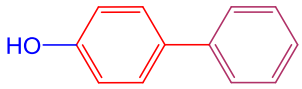
S7. The ^{1}HNMR of 3-carboxylic acid-[1,1'-biphenyl]



S8. The ^{13}CNMR of 3-carboxylic acid-[1,1'-biphenyl]

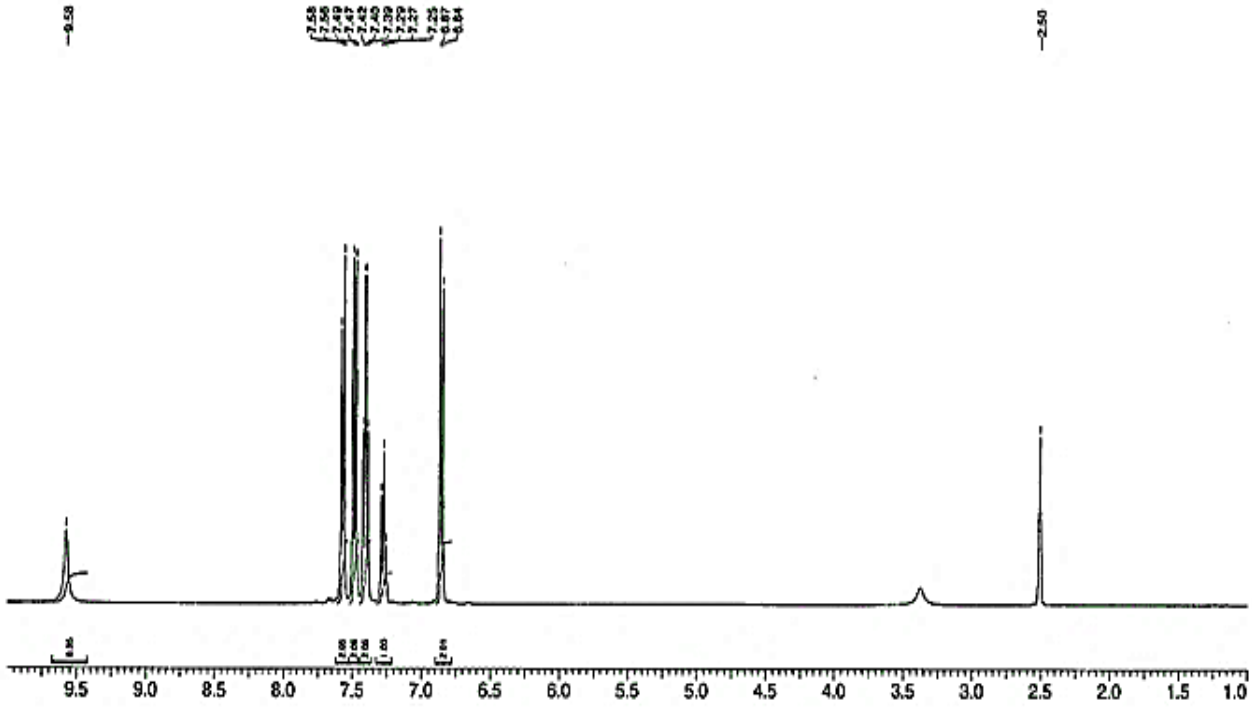


4-hydroxyl-[1,1'-biphenyl]

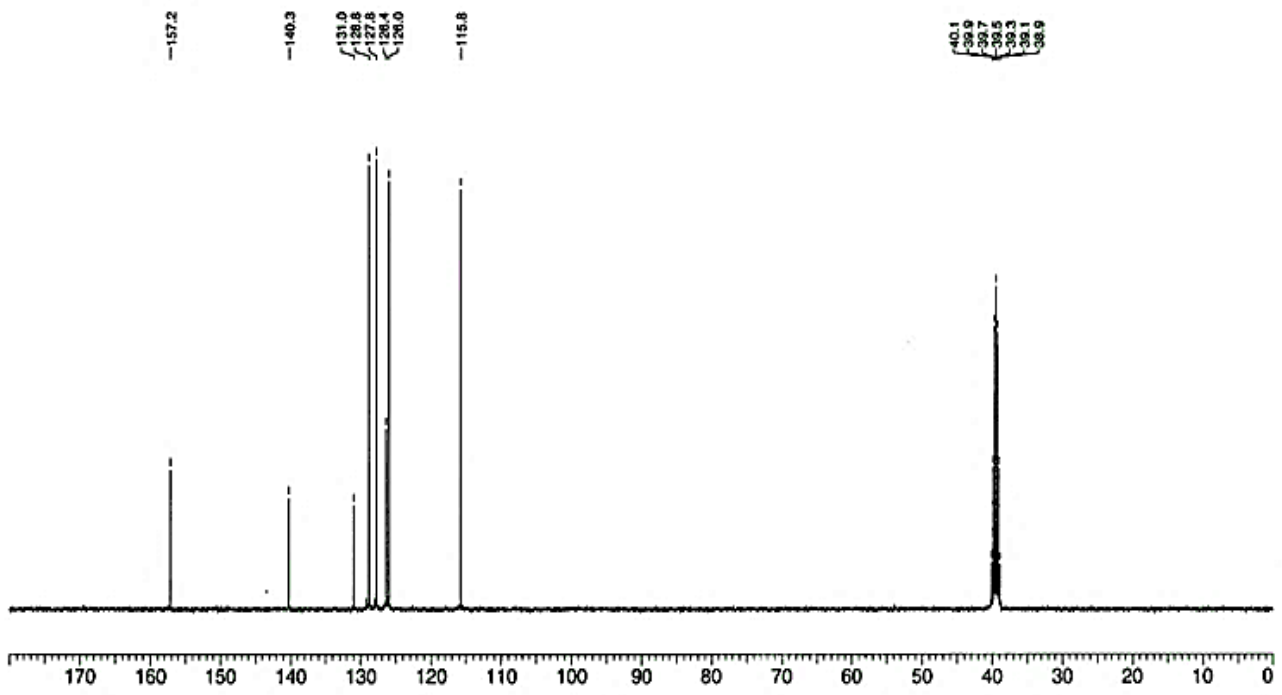


m.p. = 161-162 °C.

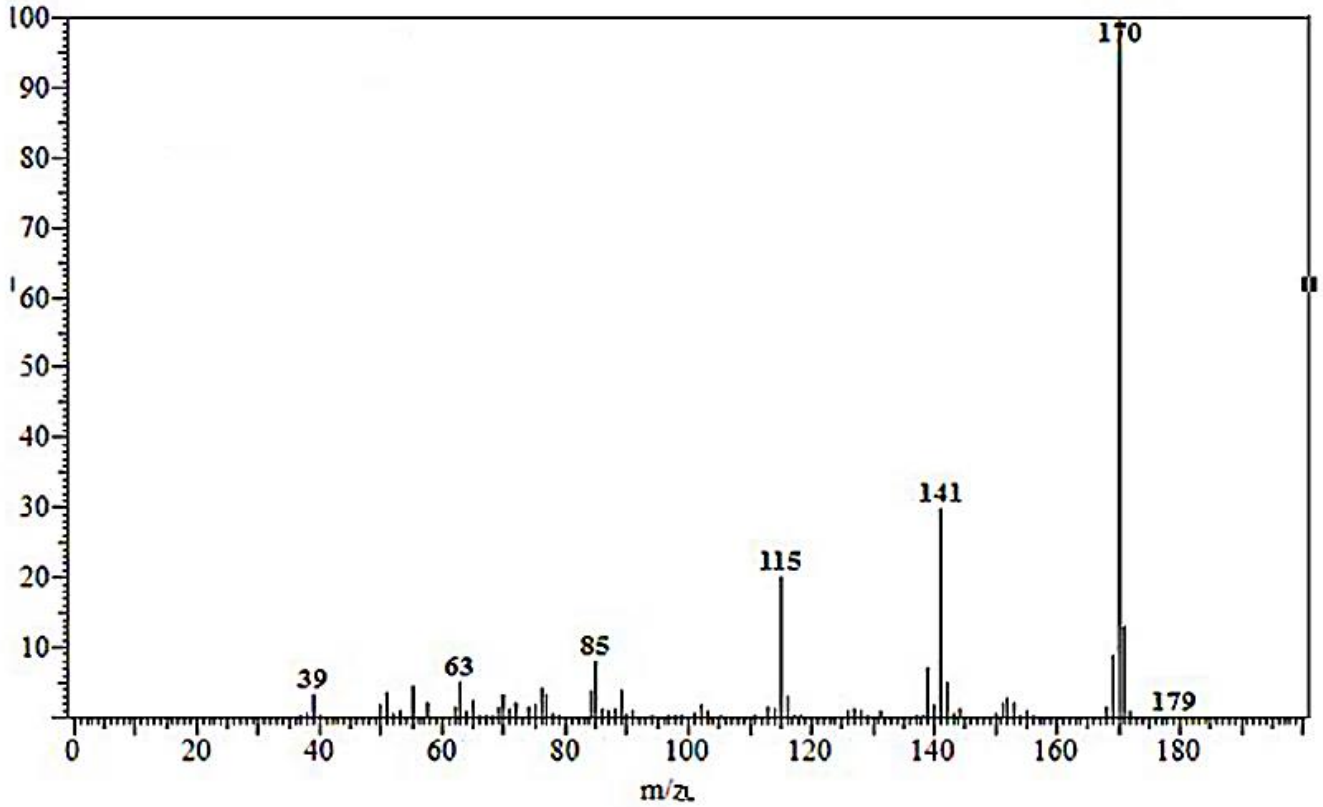
^{1}HNMR (400 MHz, DMSO): δ = 6.85 (2H, d, J = 8.5), 7.27 (1H, t, J = 7.4), 7.40 (2H, t, J = 7.4), 7.48 (2H, d, J = 8.5), 7.57 (2H, d, J = 8.5), 9.58 (1H, br s, OH). 13 C NMR (100 MHz, DMSO): δ = 115.8, 126.0, 126.4, 127.8, 128.8 (5 × CH, Ar), 131.0, 140.3, 157.2 (Ar). m/z: 169.1.



S9. The ^{1}HNMR of 4-hydroxyl-[1,1'-biphenyl]

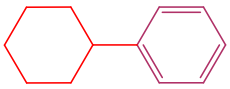


S10. The ^{13}CNMR of 4-hydroxyl-[1,1'-biphenyl]

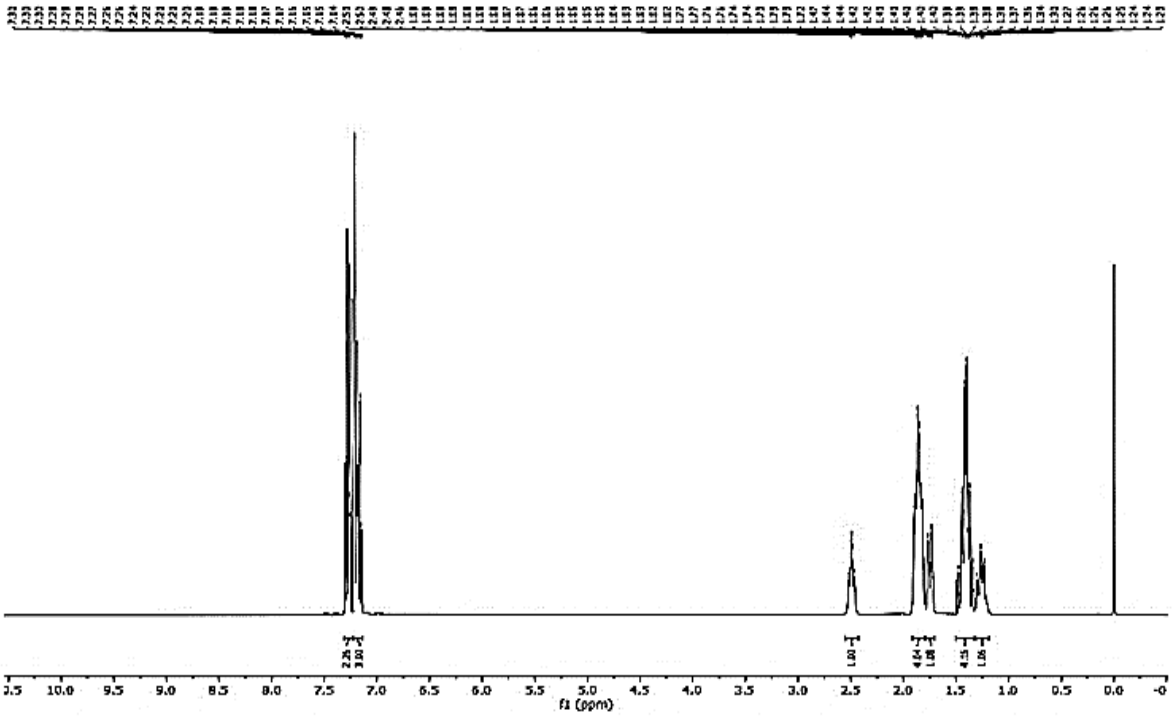


S11. The GC-Mass of 4-hydroxyl-[1,1'-biphenyl]

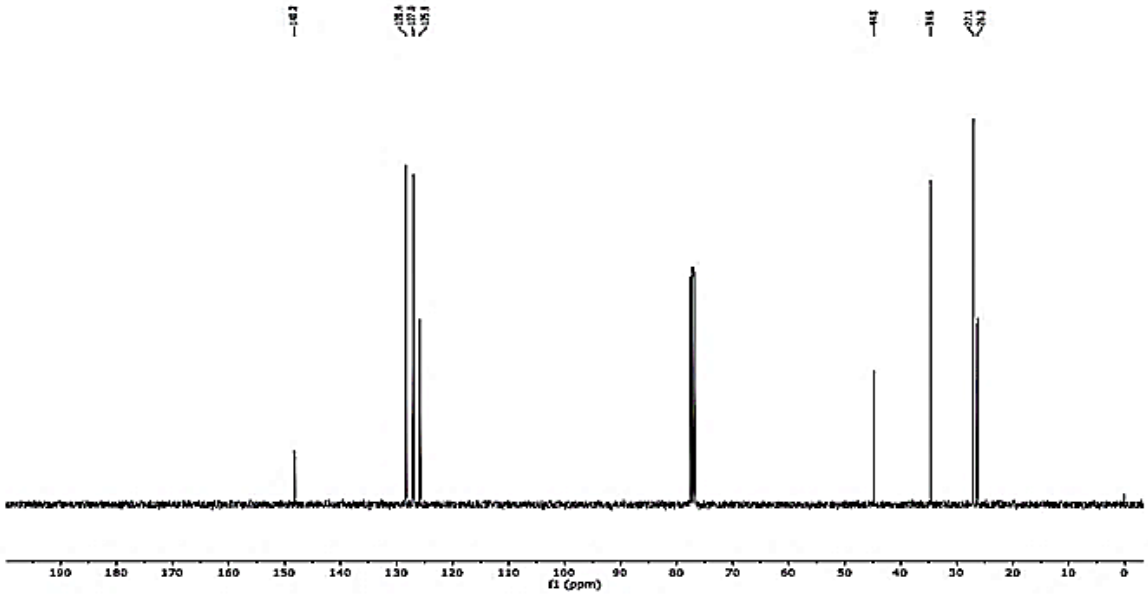
Cyclohexylbenzene



Colorless oil in 73% yield ^{1}HNMR (400 MHz, chloroform-d) δ = 7.31 – 7.25 (m, 2H), 7.23 – 7.13 (m, 3H), 2.49 (m, 1H), 1.92 – 1.82 (m, 4H), 1.74 (m, 1H), 1.51 – 1.34 (m, 4H), 1.25 (m, 1H); ^{13}CNMR (100 MHz, chloroform-d) δ = 148.2, 128.4, 127.0, 125.9, 44.8, 34.6, 27.1, 26.3.



S12. The ^{1}HNMR of Cyclohexylbenzene

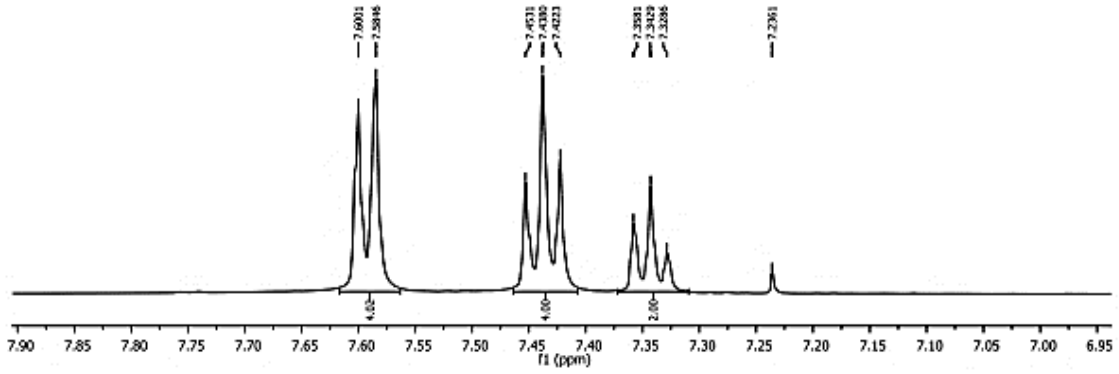


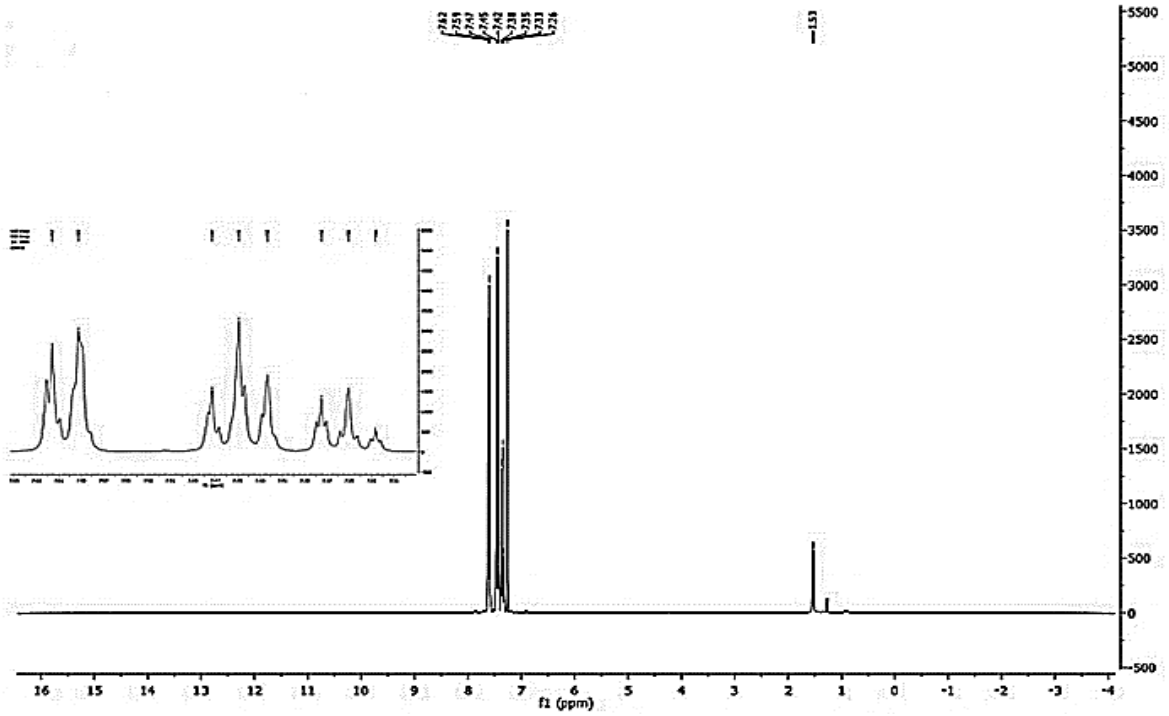
S13. The ^{13}CNMR of Cyclohexylbenzene

Biphenyl

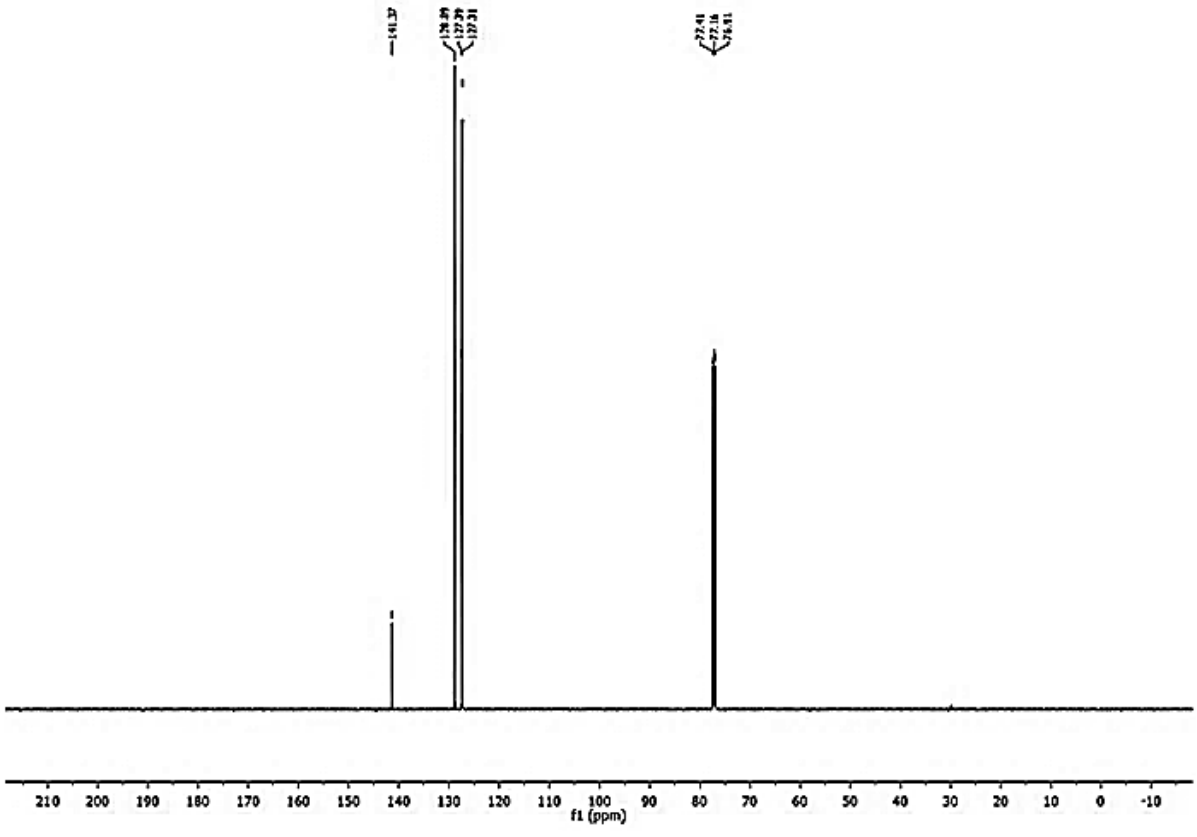
white solid in 94% yield, MP: 69-71 °C

^{1}HNMR (500 MHz, $CDCl_3$): δ 7.59 (d, J =7.7 Hz, 4H), 7.44 (t, J = 7.7 Hz, 4H), 7.34 (t, J = 7.4 Hz, 2H); ^{13}CNMR (126 MHz, $CDCl_3$): δ141.29, 128.82, 127.27; FTIR (cm^{-1}): 2358, 1510, 1029, 580; GC-MS (EI, 70ev) m/z: found: 154 ($C_{12}H_{10}$).



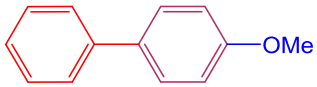


S14. The ^{1}HNMR of Biphenyl

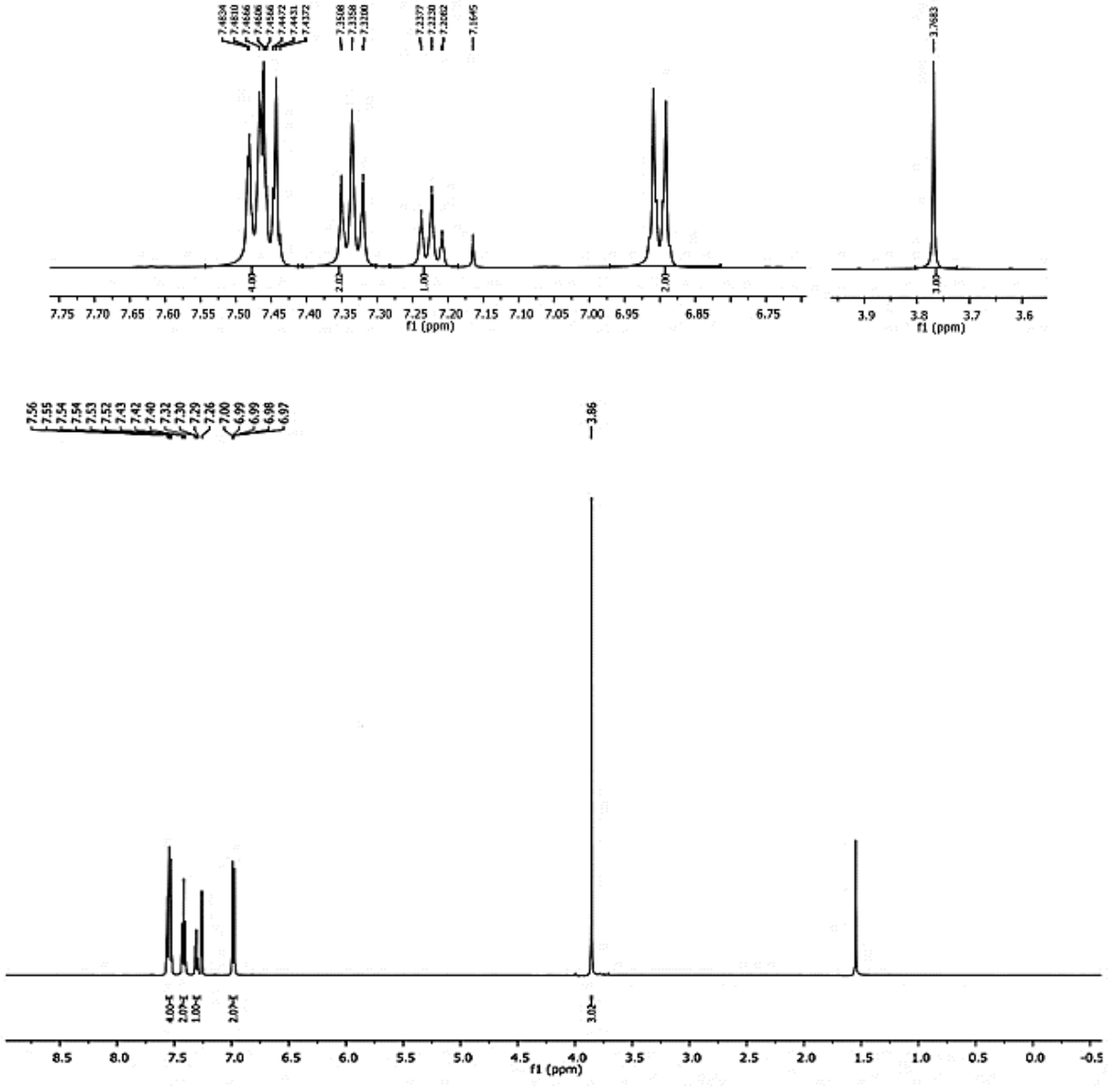


S15. The ^{13}CNMR of Biphenyl

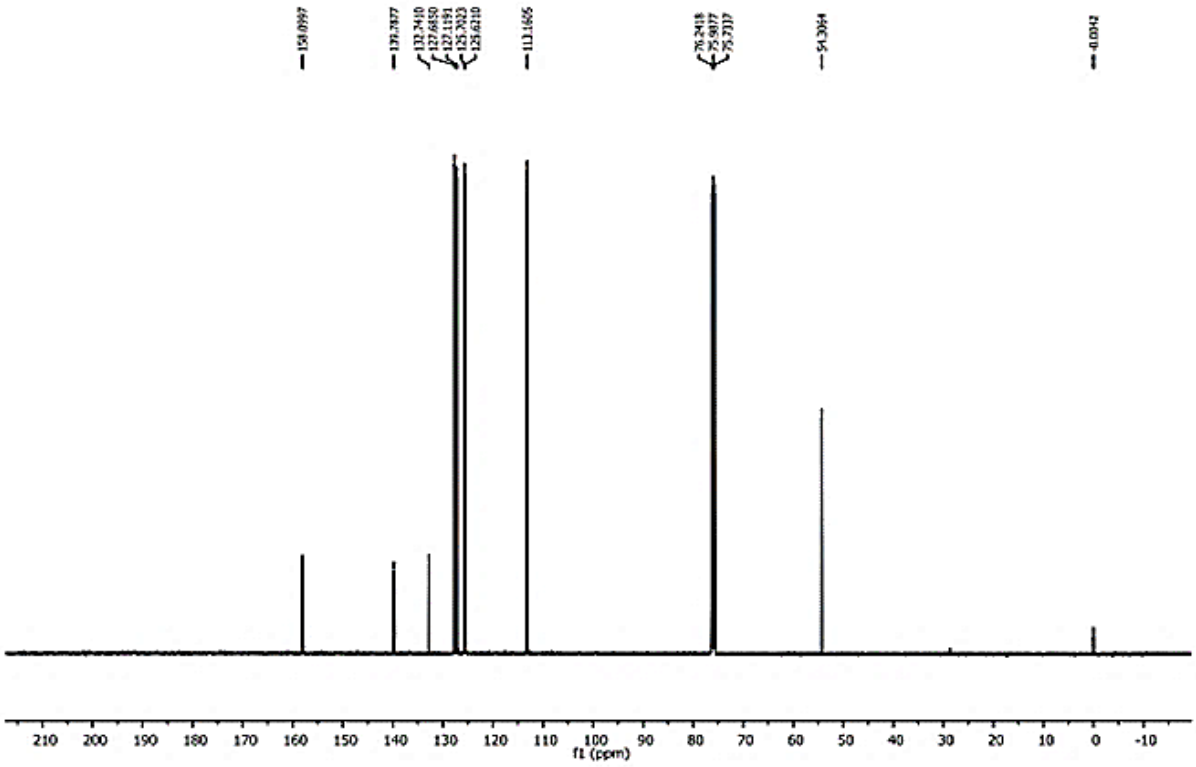
4-Methoxy-1,1'-biphenyl



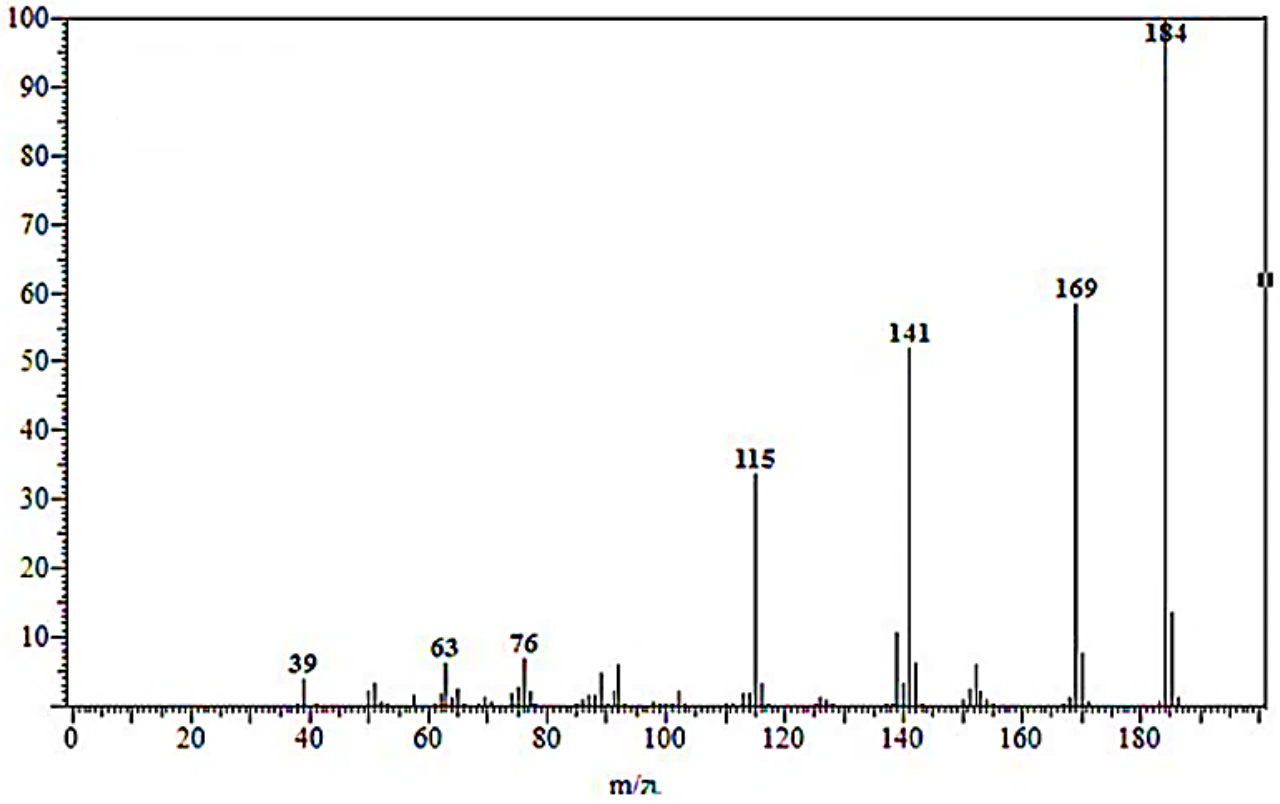
White waxy solid, yield 90%, ^{1}HNMR (500 MHz, $CDCl_3$): 7.57-7.52 (m, 4H), 7.41 (t, J = 8.0 Hz, 2H), 7.30 (t, J = 8.0 Hz, 1H), 6.98 (d, J = 8.5 Hz, 2H), and 3.86 (s, 3H).



S16. The ^{1}HNMR of 4-Methoxy-1,1'-biphenyl

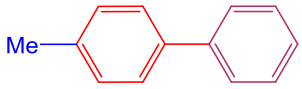


S17. The ^{13}CNMR of 4-Methoxy-1,1'-bipheny

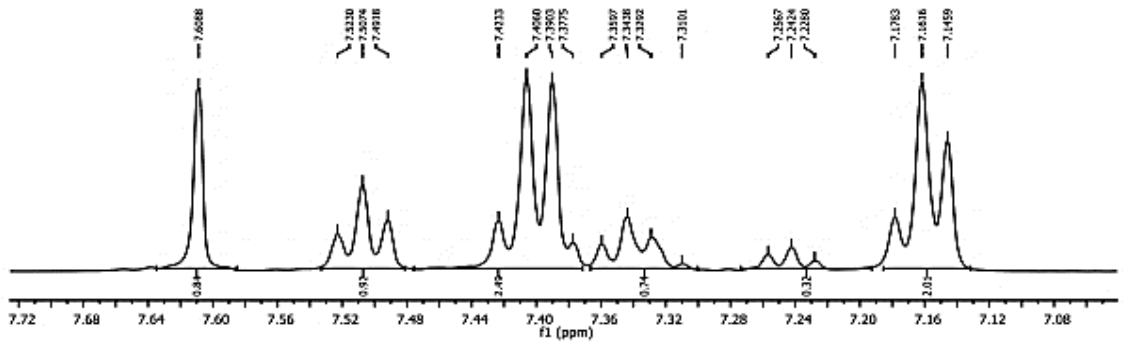


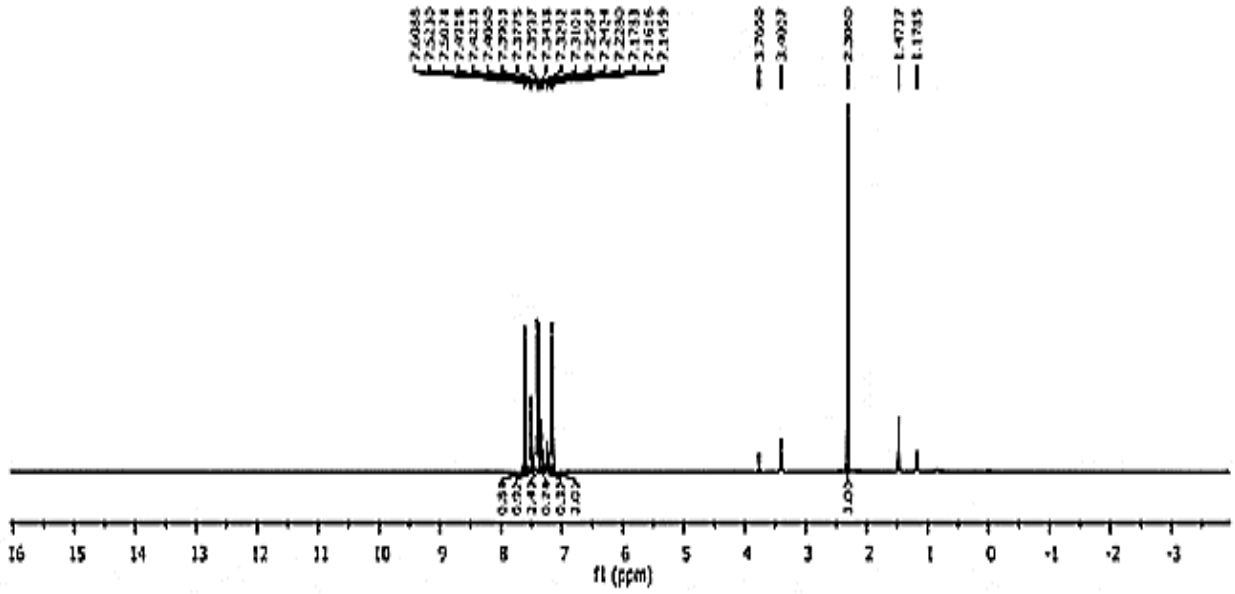
S18. The GC-Mass of 4-Methoxy-1,1'-biphenyl

4-Methyl-1,1'-biphenyl

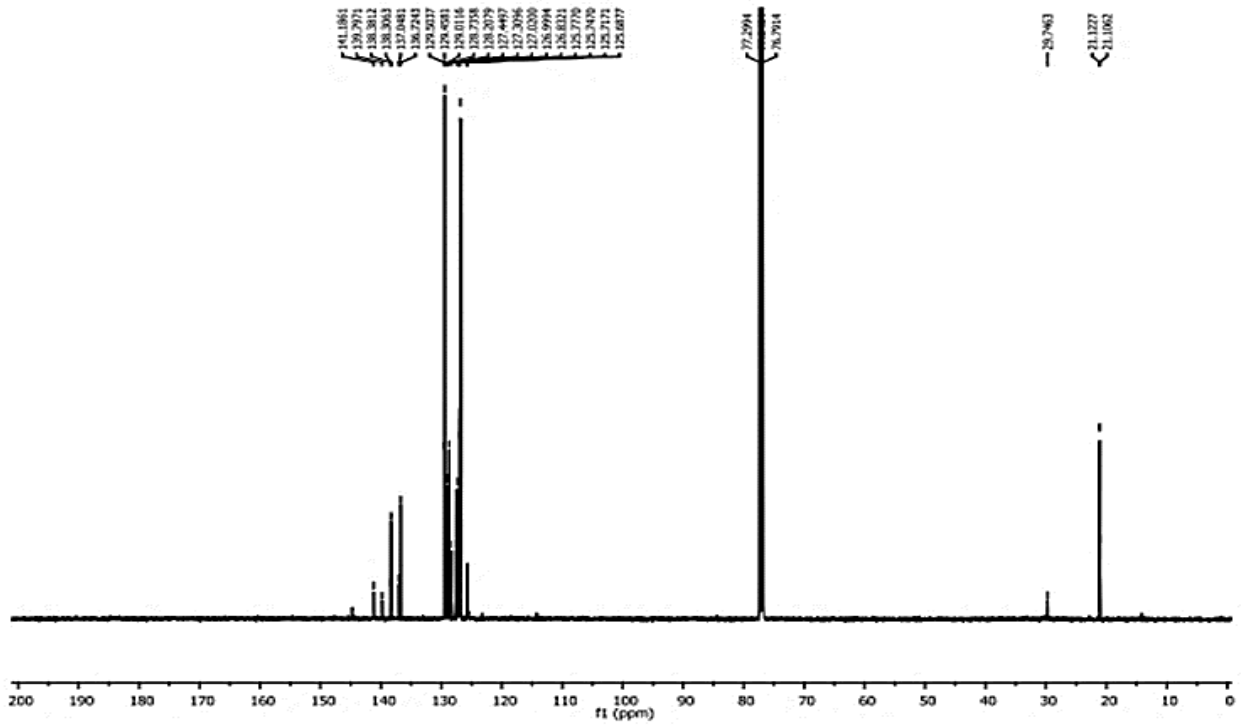


white yellow solid in 92% yield, MP: 46-48 °C; ^{1}HNMR (500 MHz, $CDCl_3$): δ 7.61 (s, 1H), 7.51 (t, J = 7.8 Hz, 1H), 7.39 (m, 3H), 7.34 (m, 1H), 7.24 (t, J = 7.2 Hz, 1H), 7.16 (t, J = 8.1 Hz, 2H), 2.31 (s, 3H); ^{13}CNMR (126 MHz, $CDCl_3$): δ 141.19, 139.80, 138.34, 137.05, 136.72, 129.48, 129.01, 128.74, 128.21, 127.38, 127.01, 126.83, 125.73, 21.11; FTIR (cm^{-1}): 2918, 2318, 1743, 1521, 1325, 1114, 802; GC-MS m/z: 168.

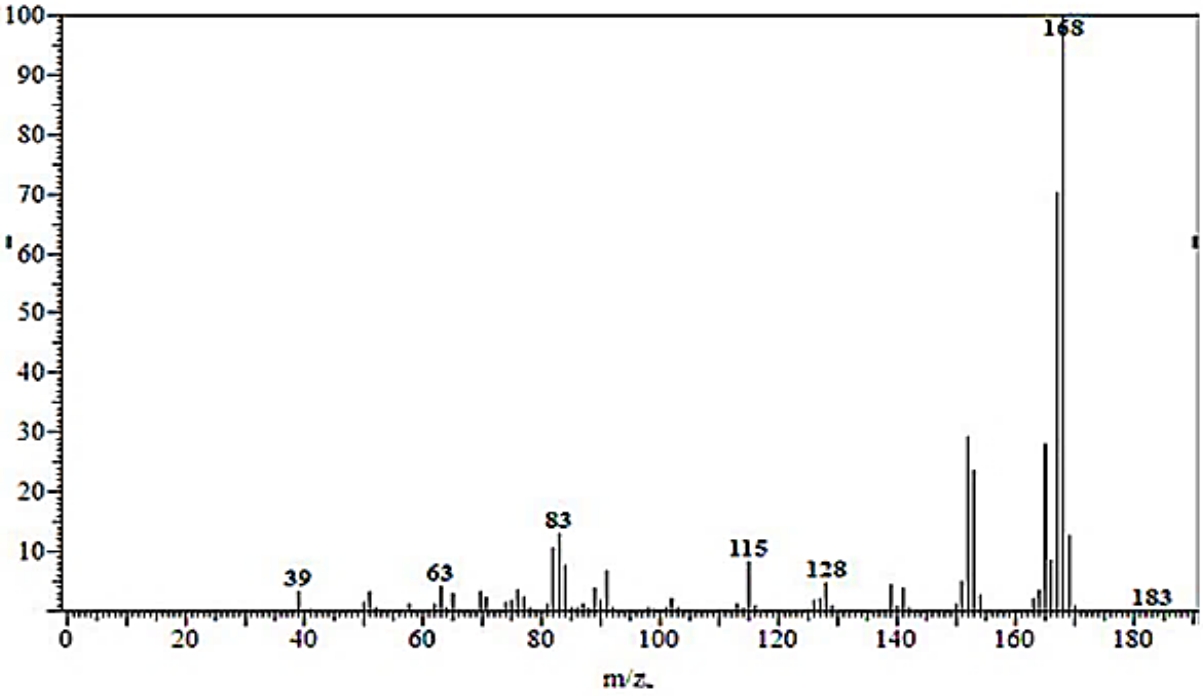




S19. The ^{1}HNMR of 4-Methyl-1,1'-biphenyl

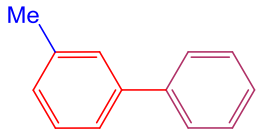


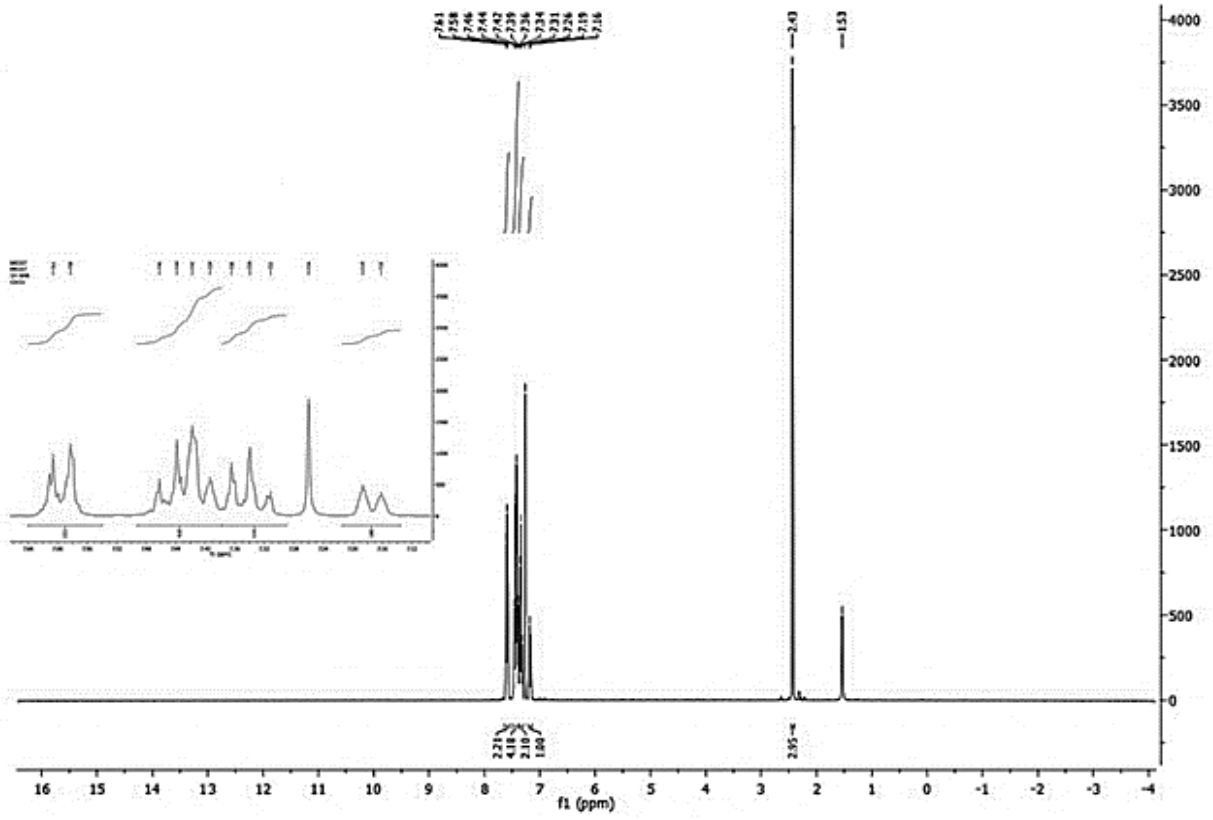
S20. The ^{13}CNMR of 4-Methyl-1,1'-biphenyl



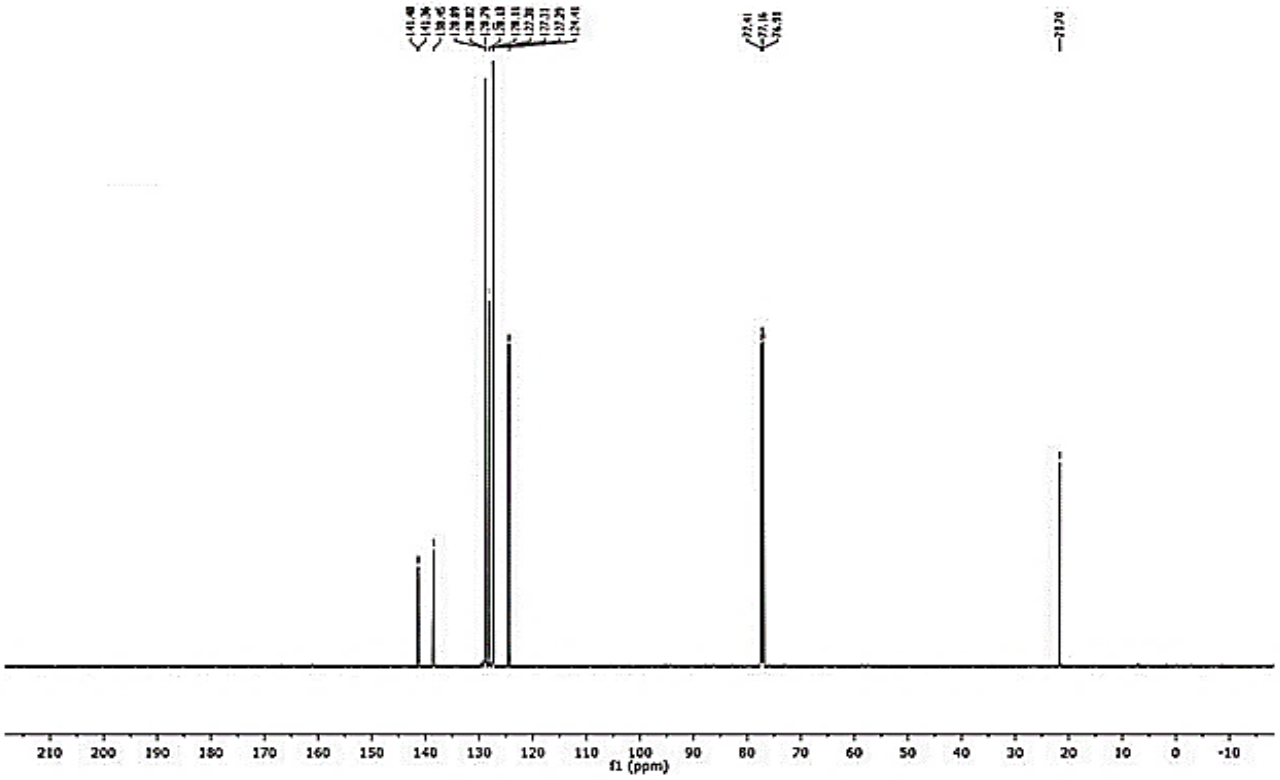
S21. The GC-Mass of 4-Methyl-1,1'-biphenyl

3-methyl-1,1'-bipheny

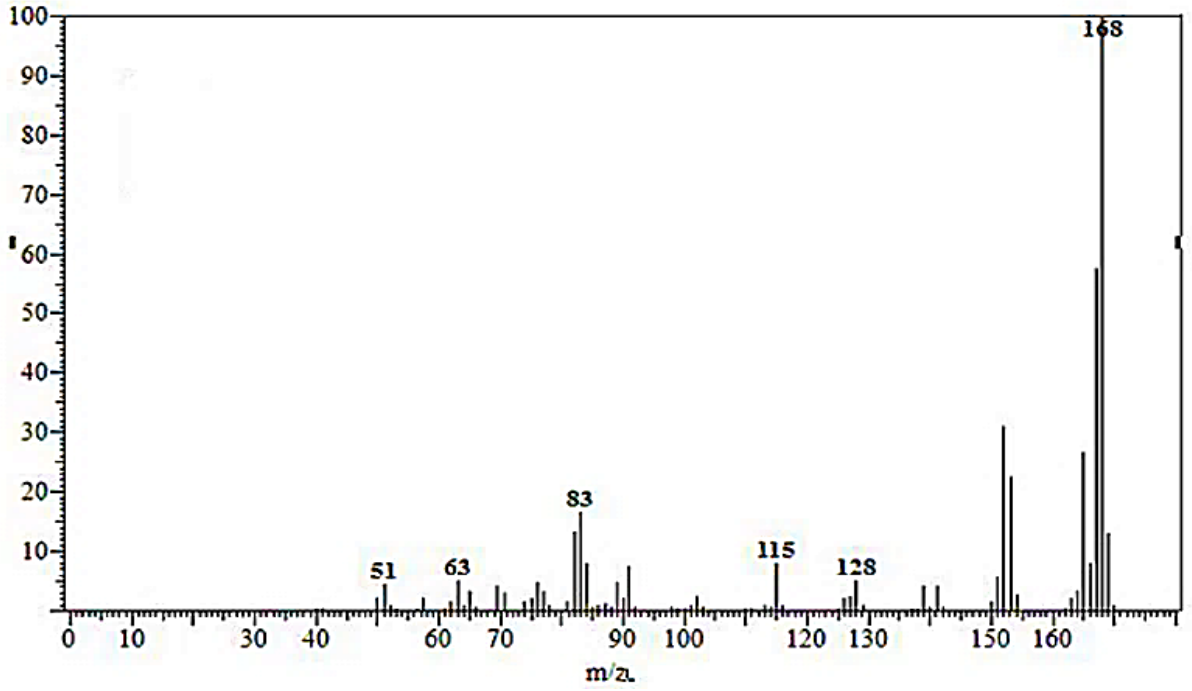




S22. The ^{1}HNMR of 3-methyl-1,1'-bipheny

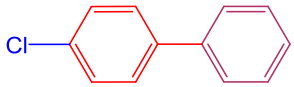


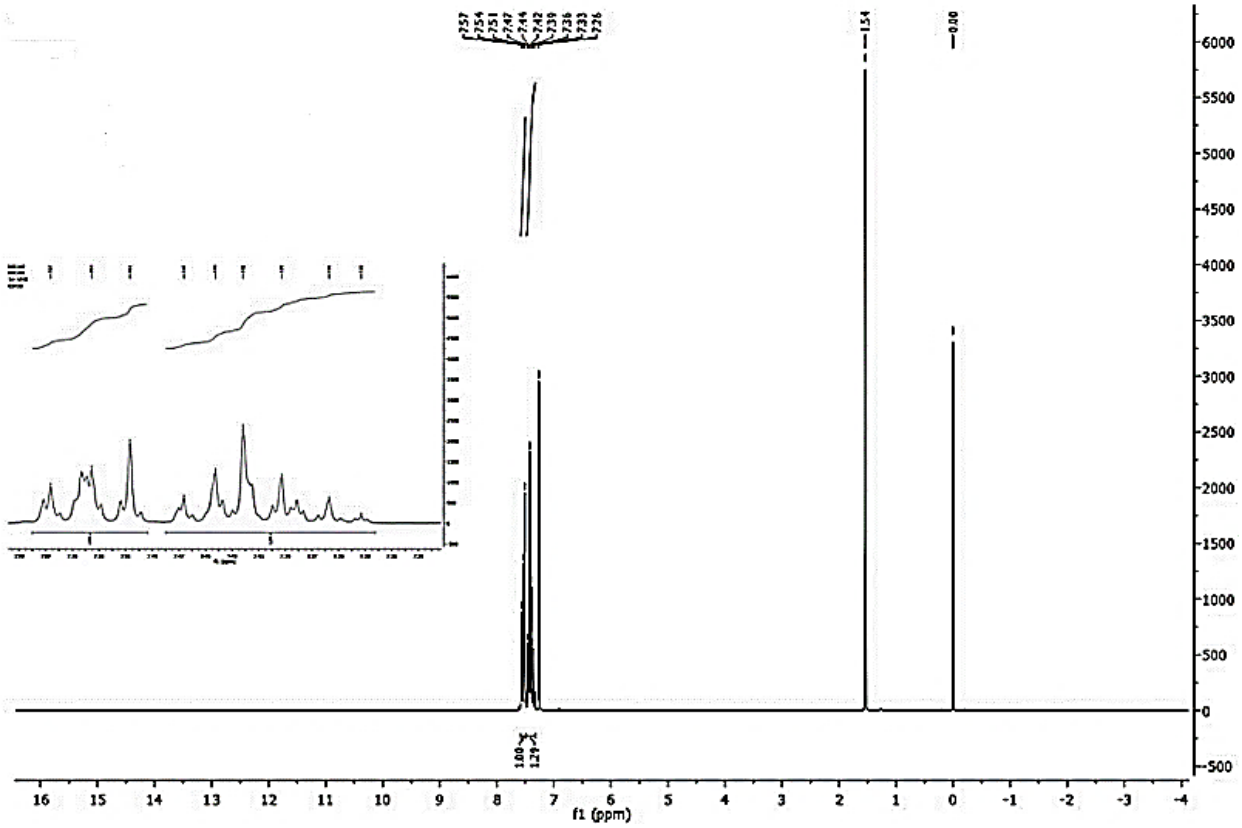
S23. The ^{13}CNMR of 3-methyl-1,1'-bipheny



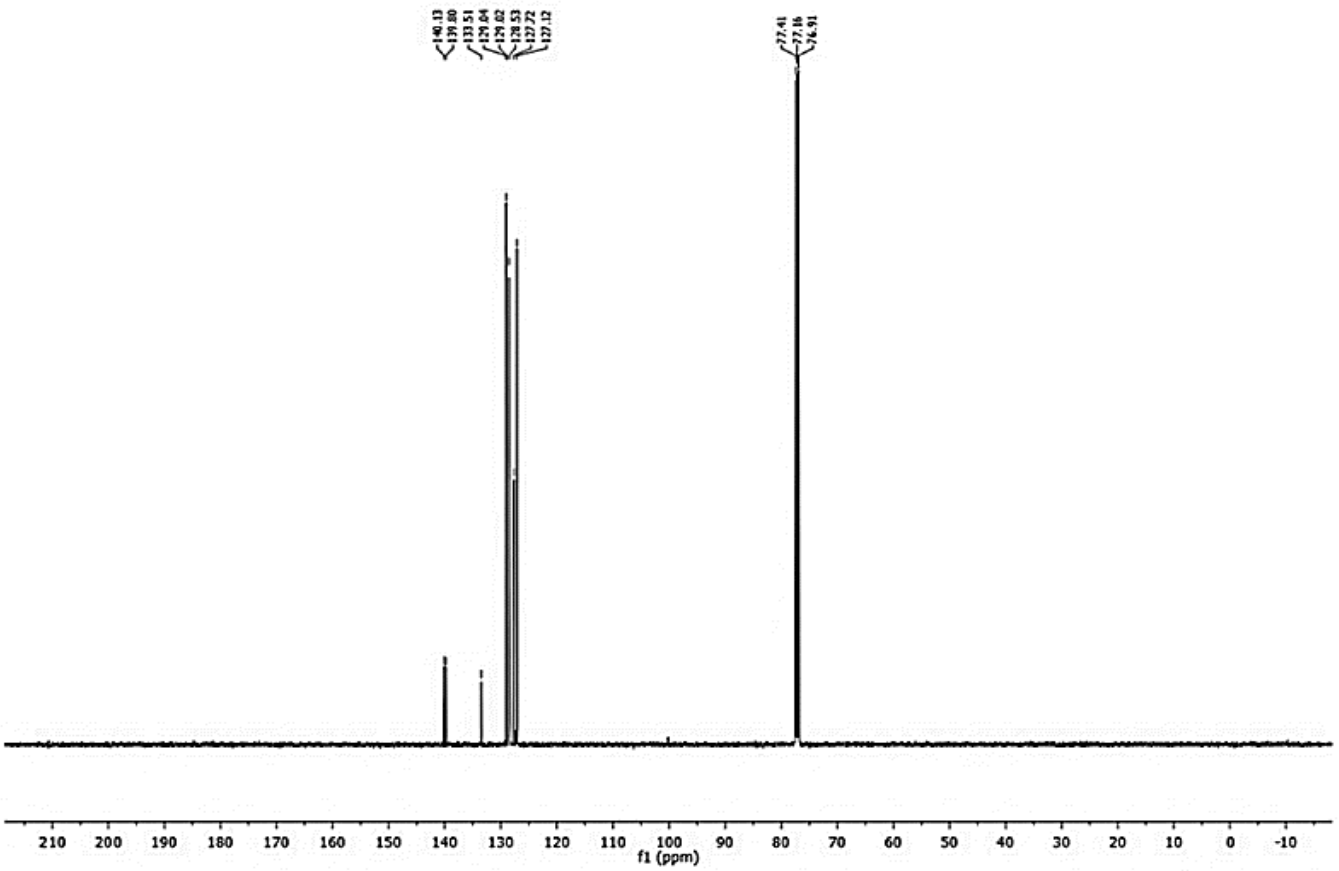
S24. The GC-Mass of 3-methyl-1,1'-bipheny

4-chloro-1,1'-bipheny



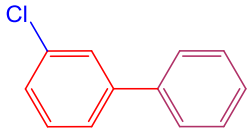


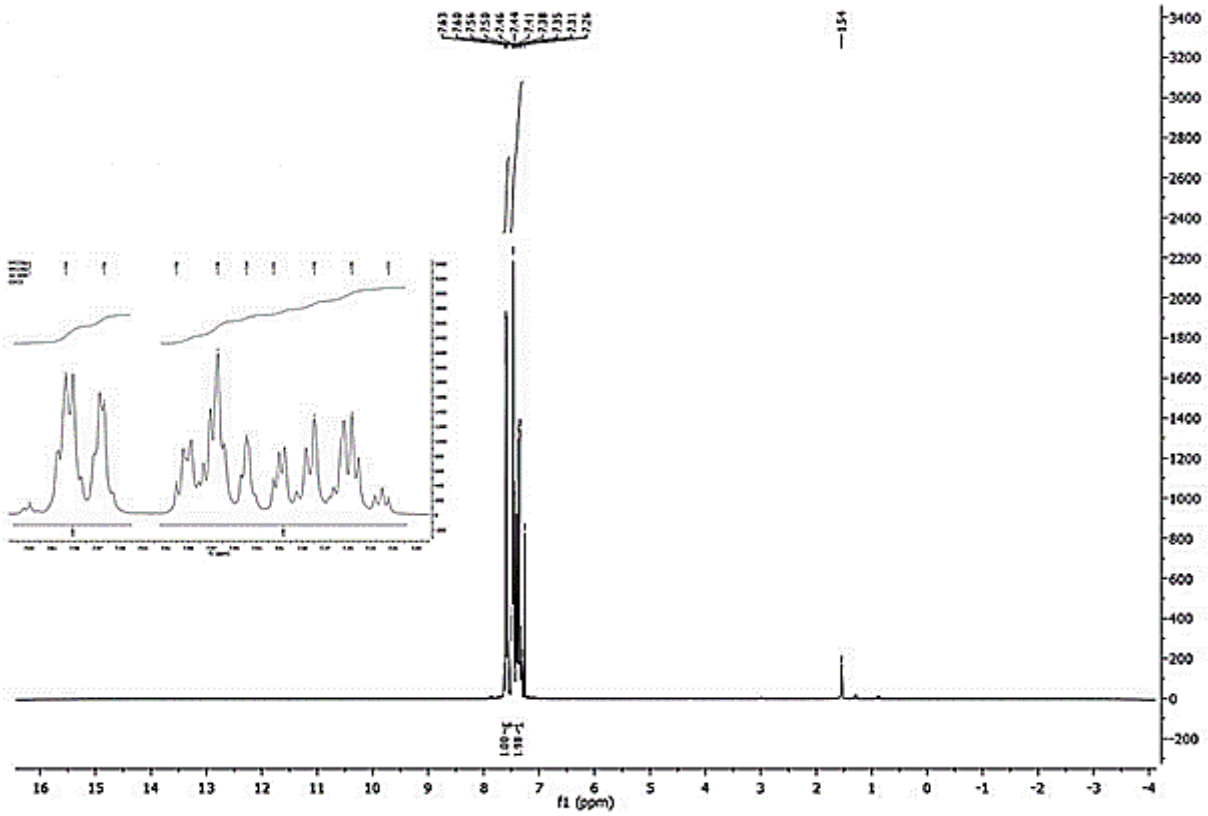
S25. The ^{1}HNMR of 4-chloro-1,1'-bipheny



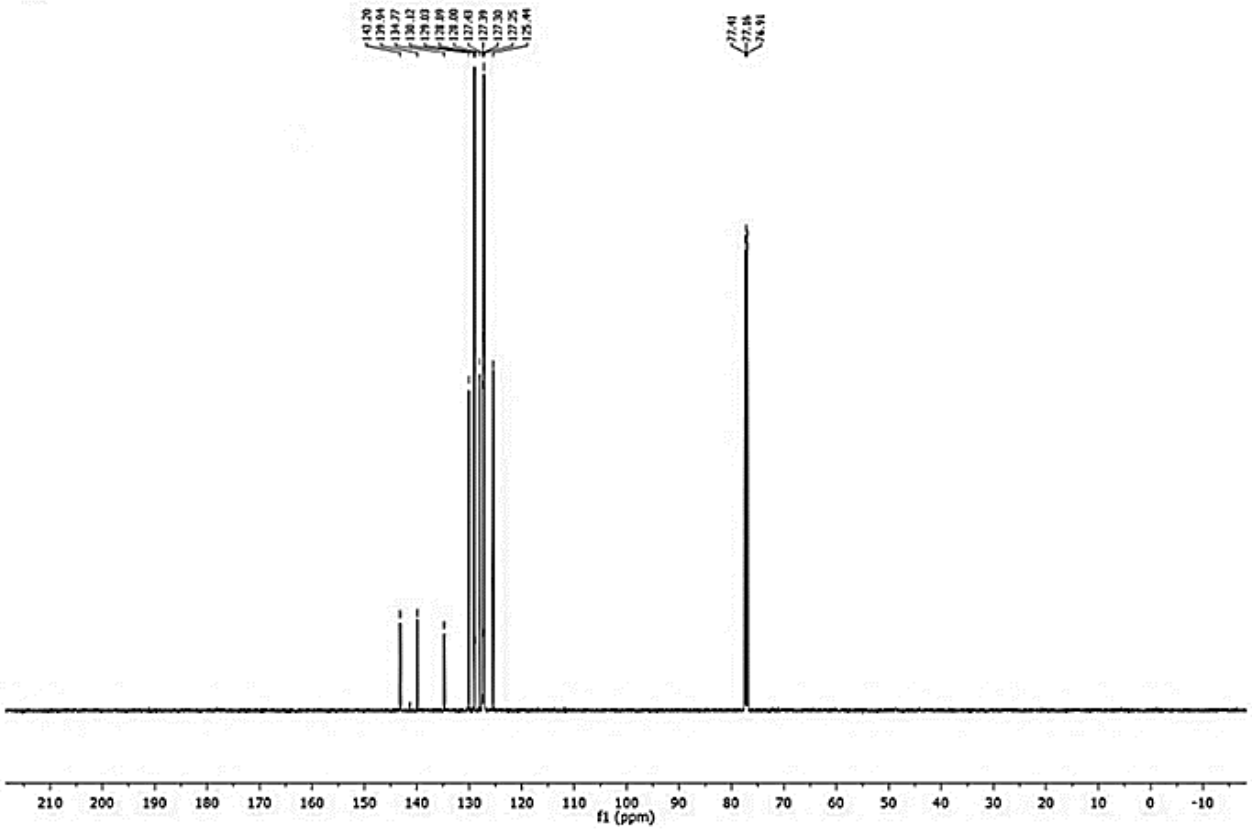
S26. The ^{13}CNMR of 4-chloro-1,1'-bipheny

3-chloro-1,1'-bipheny

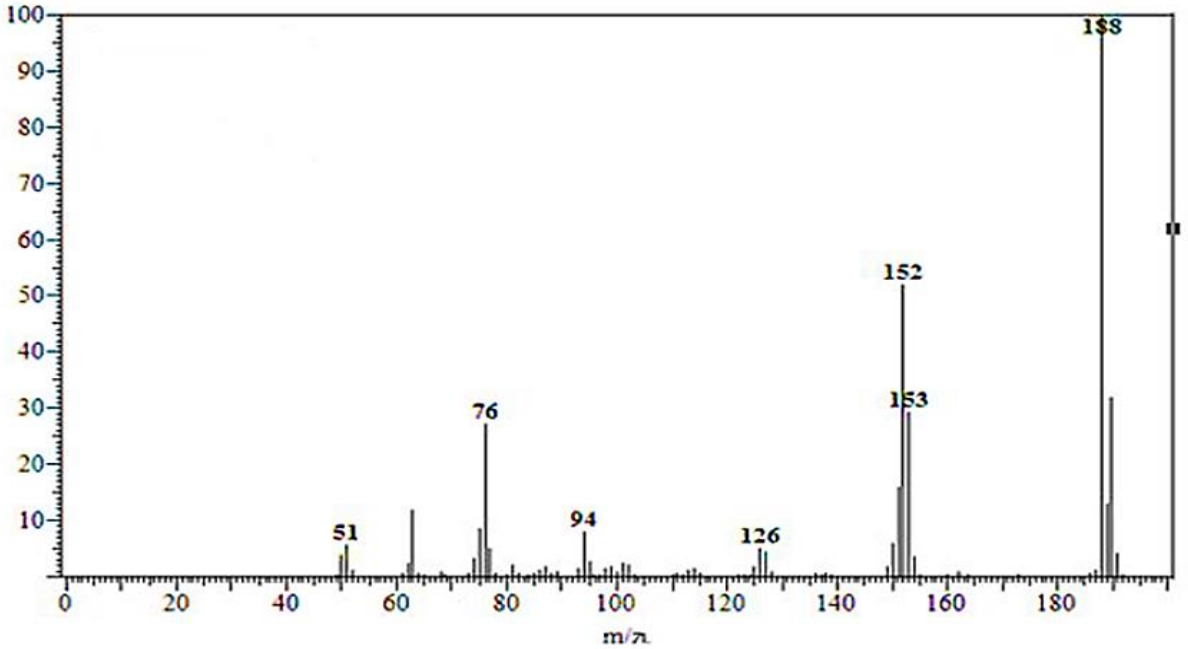




S27. The ^{1}HNMR of 3-chloro-1,1'-bipheny

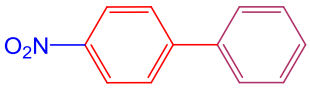


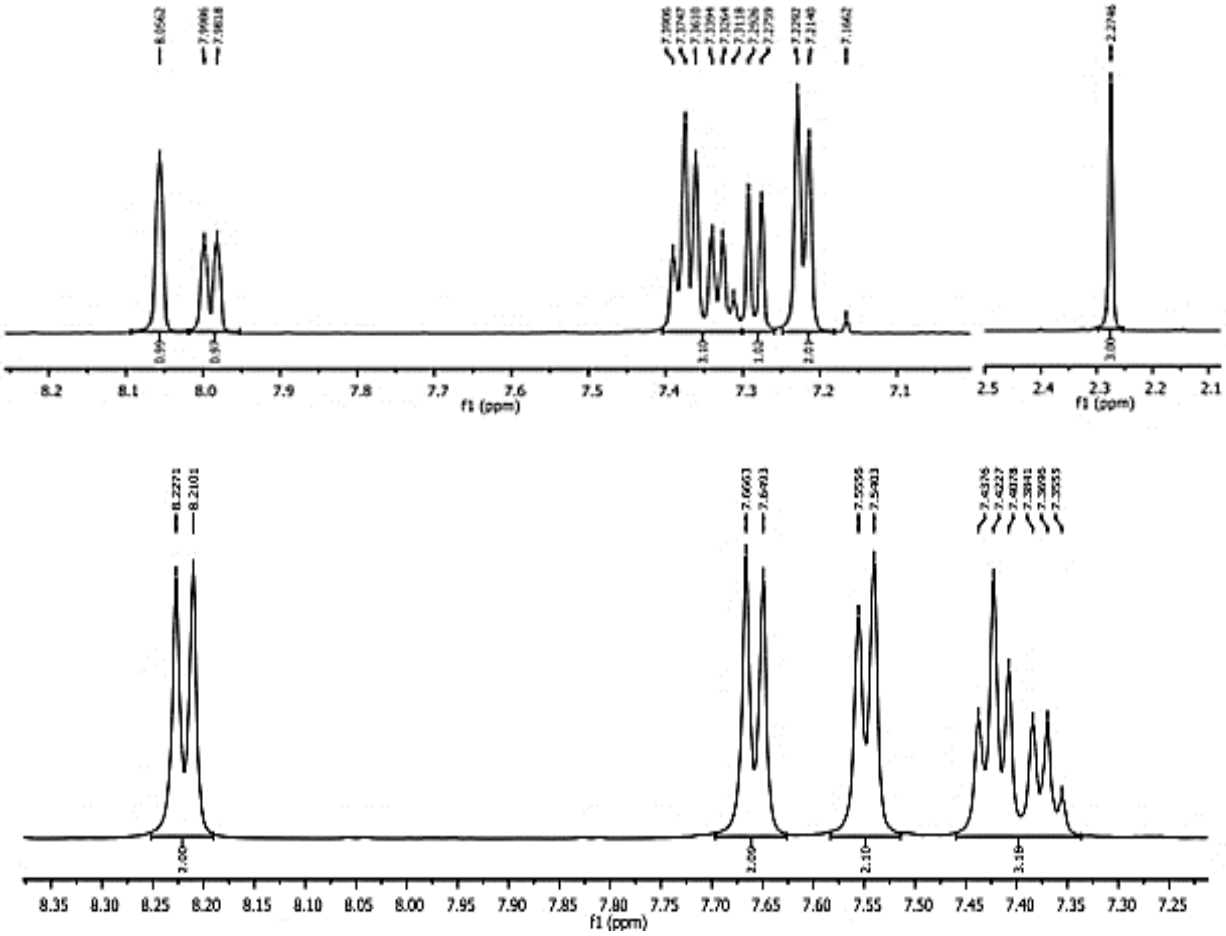
S28. The ^{13}CNMR of 3-chloro-1,1'-bipheny



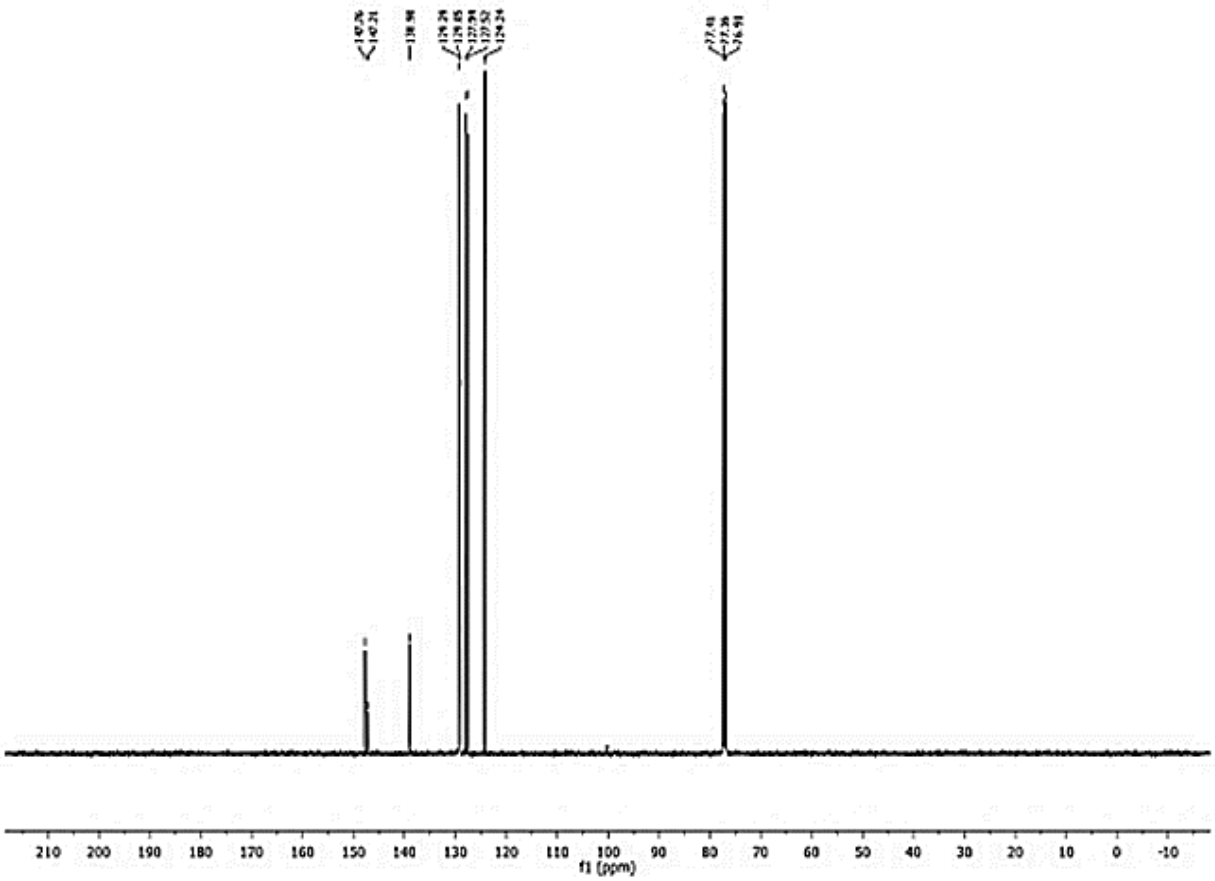
S29. The GC-Mass of 3-chloro-1,1'-bipheny

4-nitro-[1,1'-biphenyl]

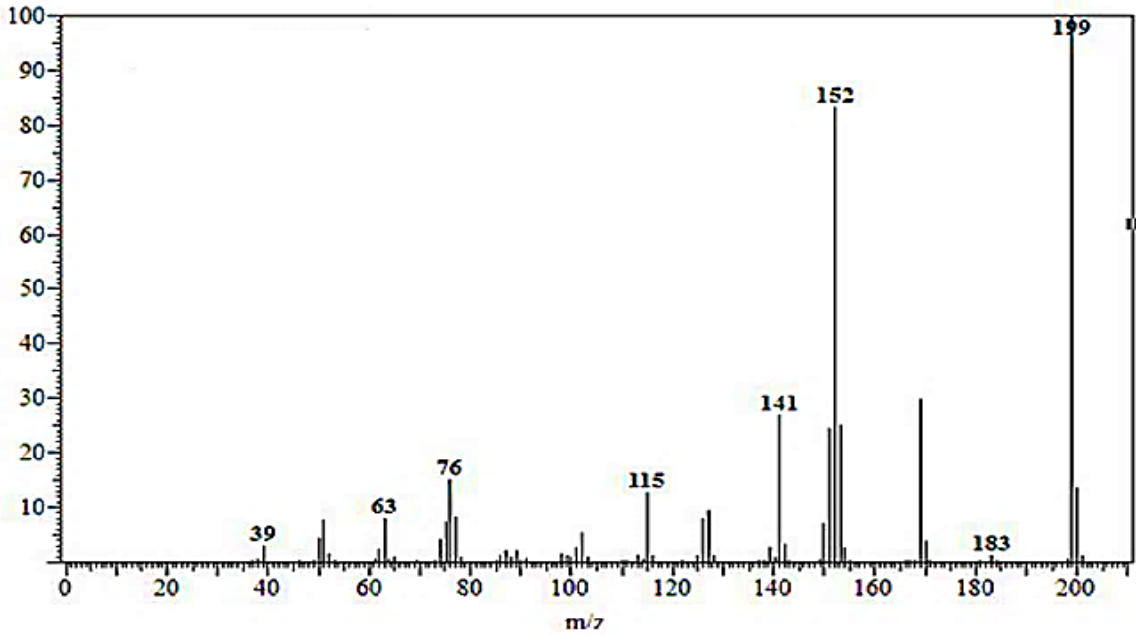




S30. The ^{1}HNMR of 4-nitro-[1,1'-biphenyl]

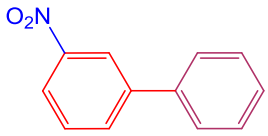


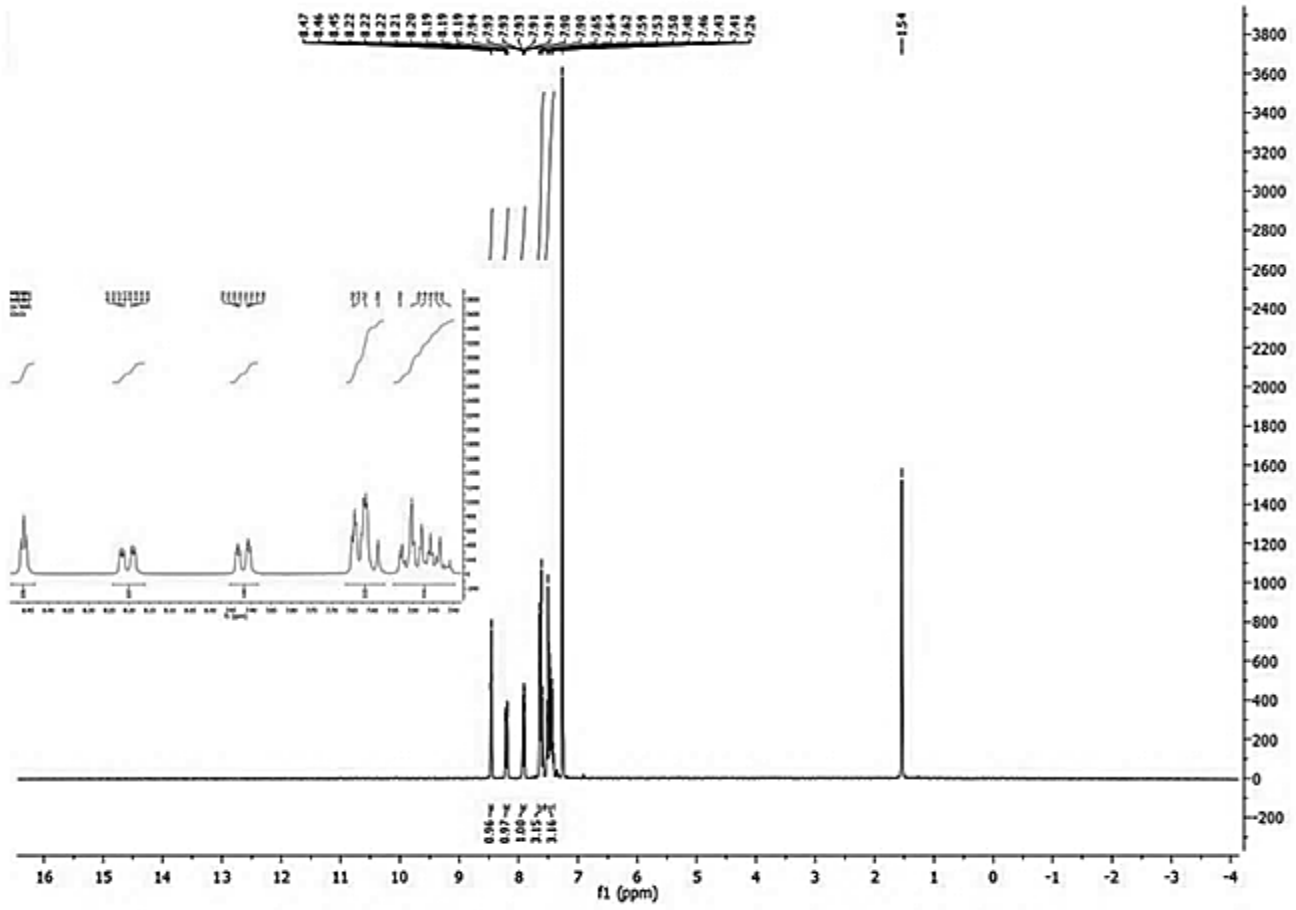
S31. The ^{13}CNMR of 4-nitro-[1,1'-biphenyl]



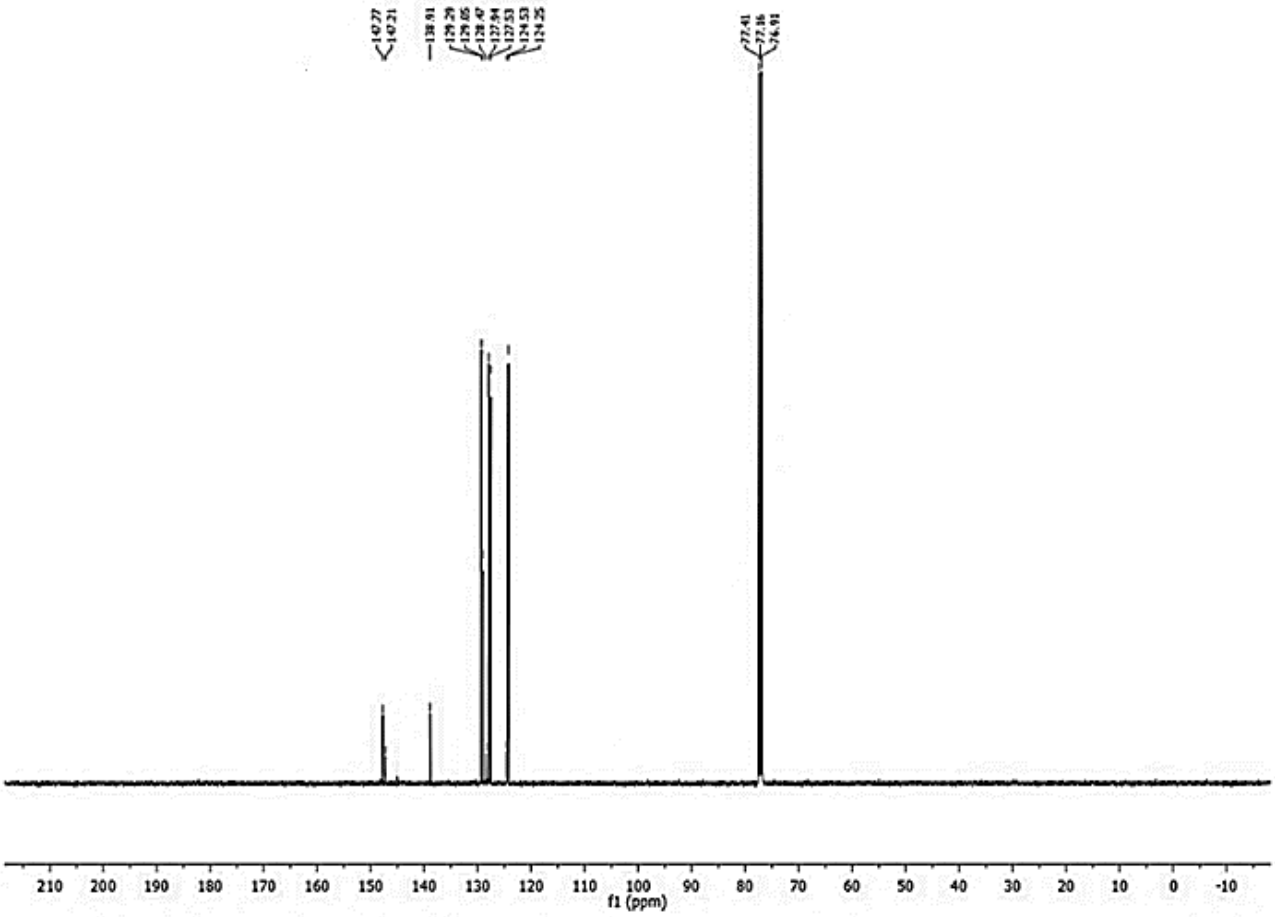
S32. The GC-Mass of 4-nitro-[1,1'-biphenyl]

3-nitro-1,1'-bipheny



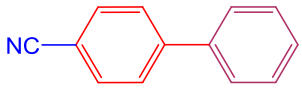


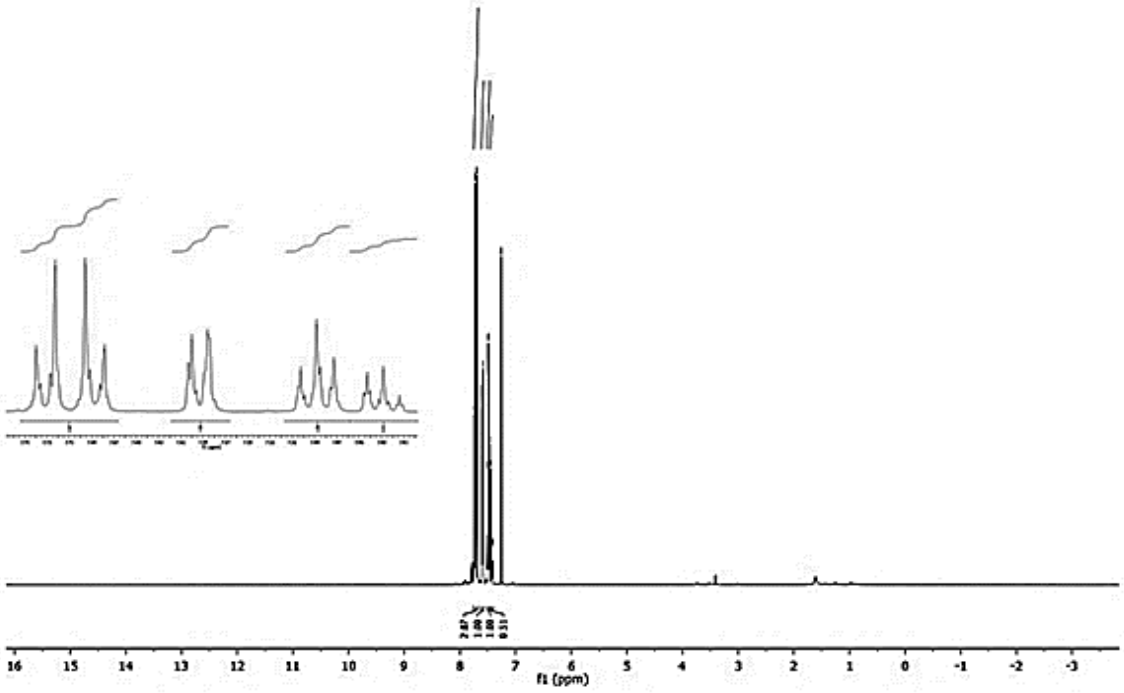
S33. The ^{1}HNMR of 3-nitro-[1,1'-biphenyl]



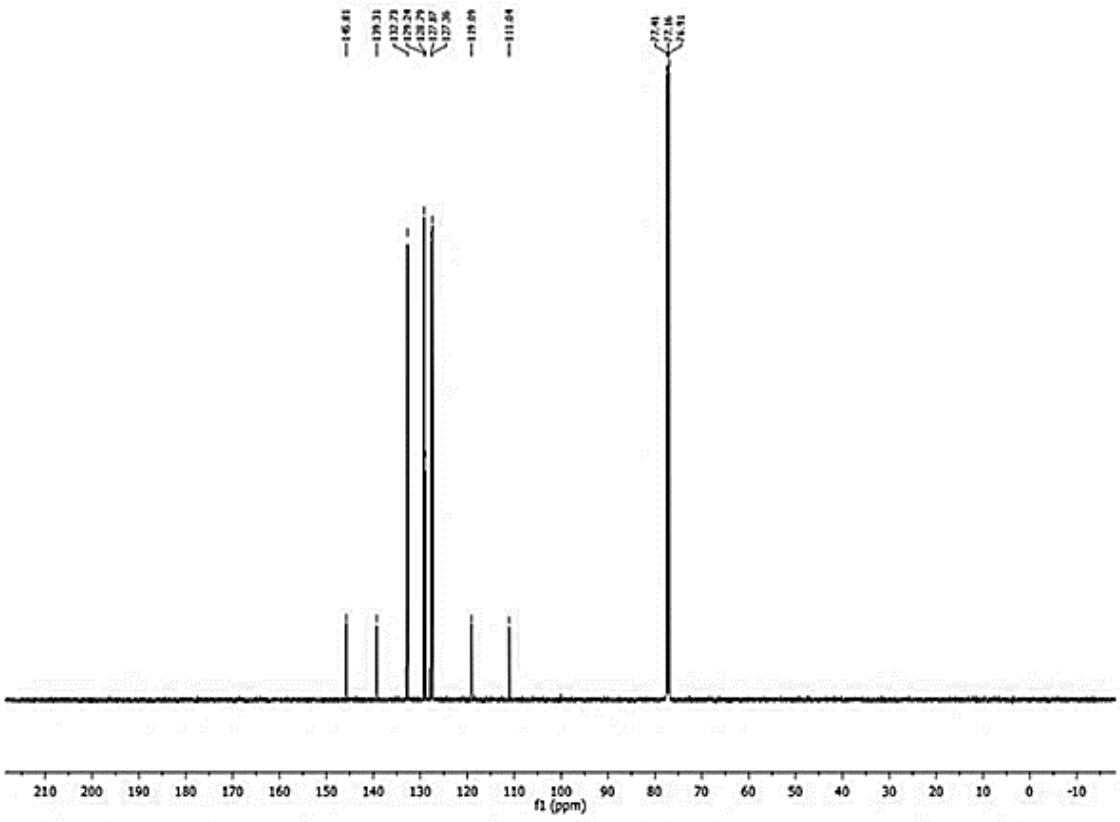
S34. The ^{13}CNMR of 3-nitro-[1,1'-biphenyl]

4-carbonitril-[1,1'-biphenyl]



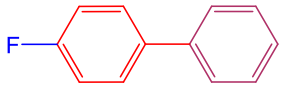


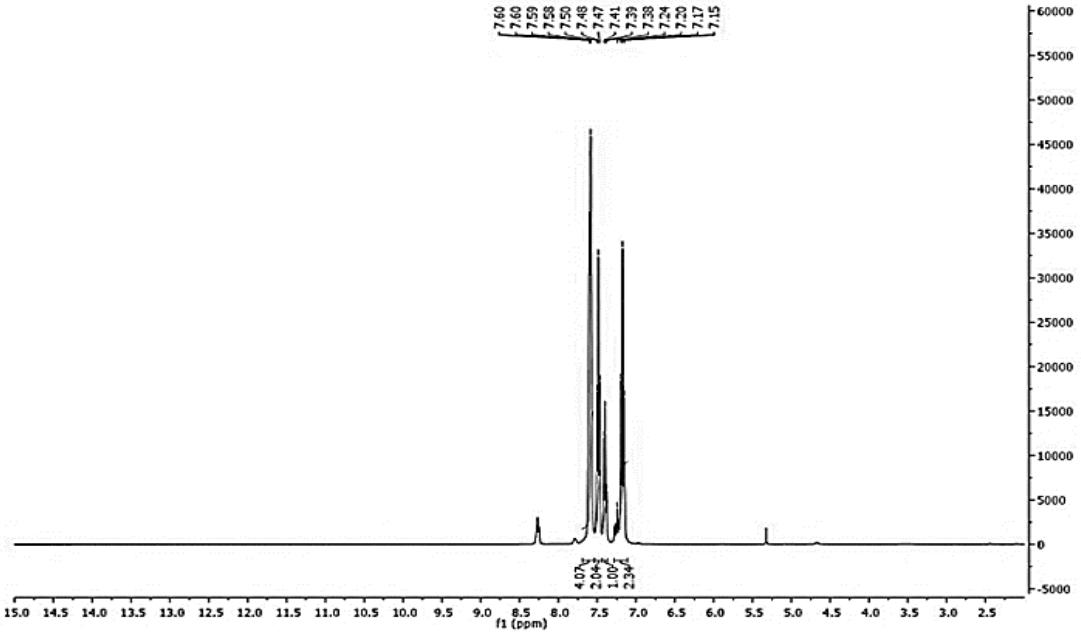
S35. The ^{1}HNMR of 4-carbonitril-[1,1'-biphenyl]



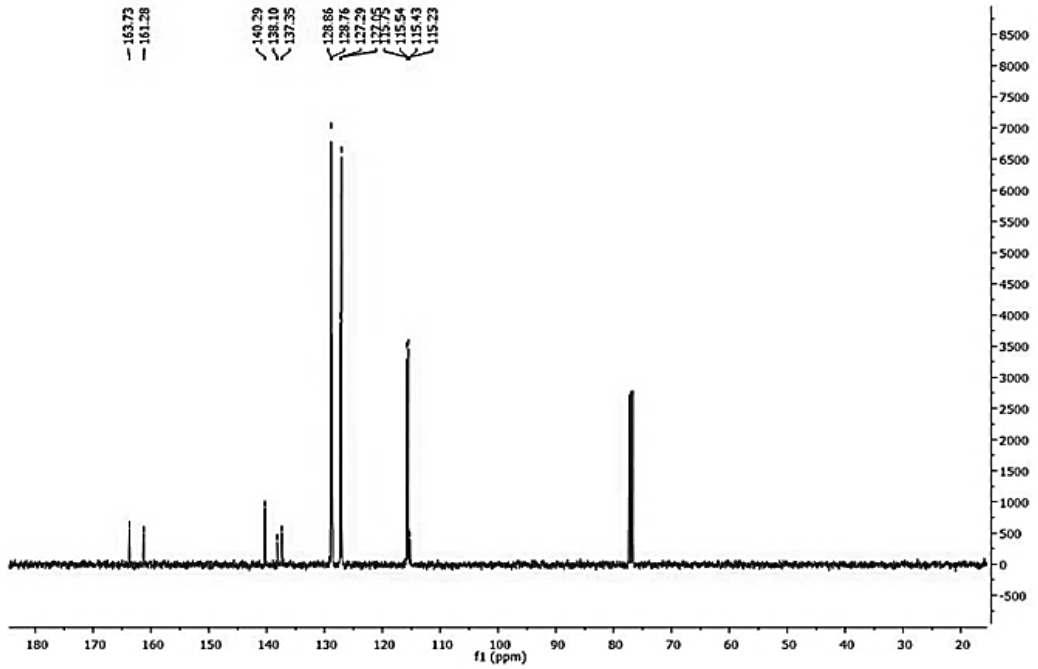
S36. The ^{13}CNMR of 4-carbonitril-[1,1'-biphenyl]

4-fluoro-[1,1'-biphenyl]

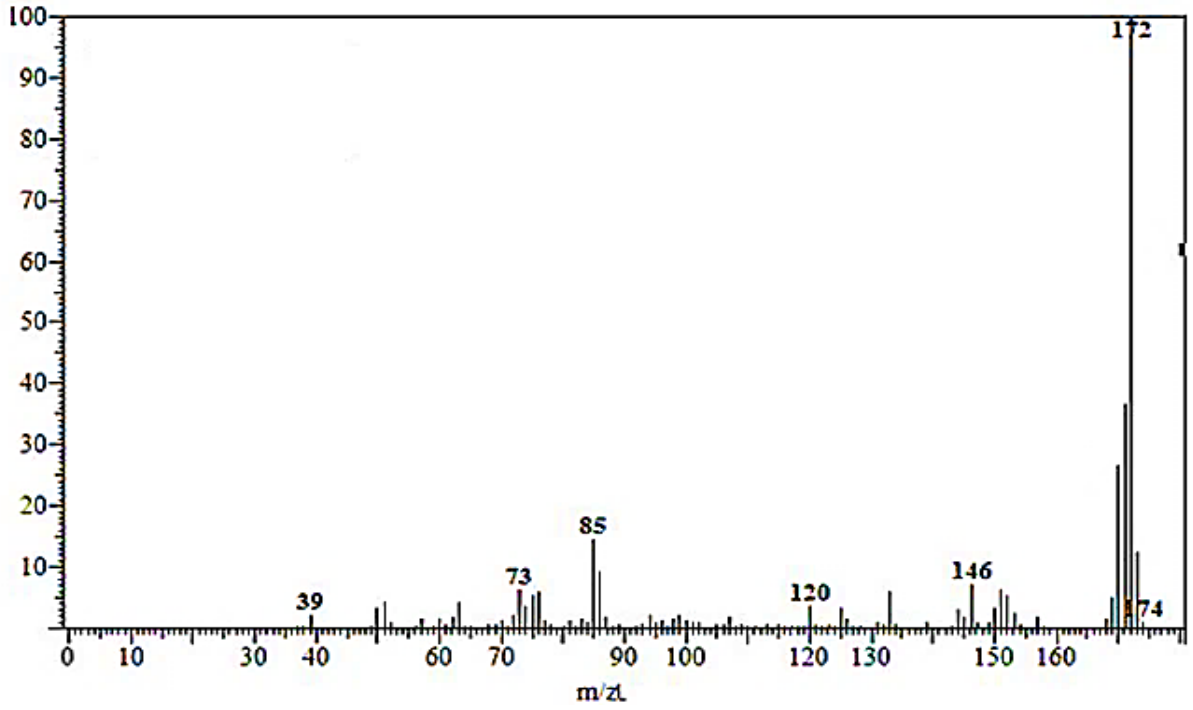




S37. The ^{1}HNMR of 4-fluoro-[1,1'-biphenyl]

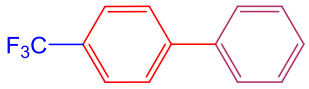


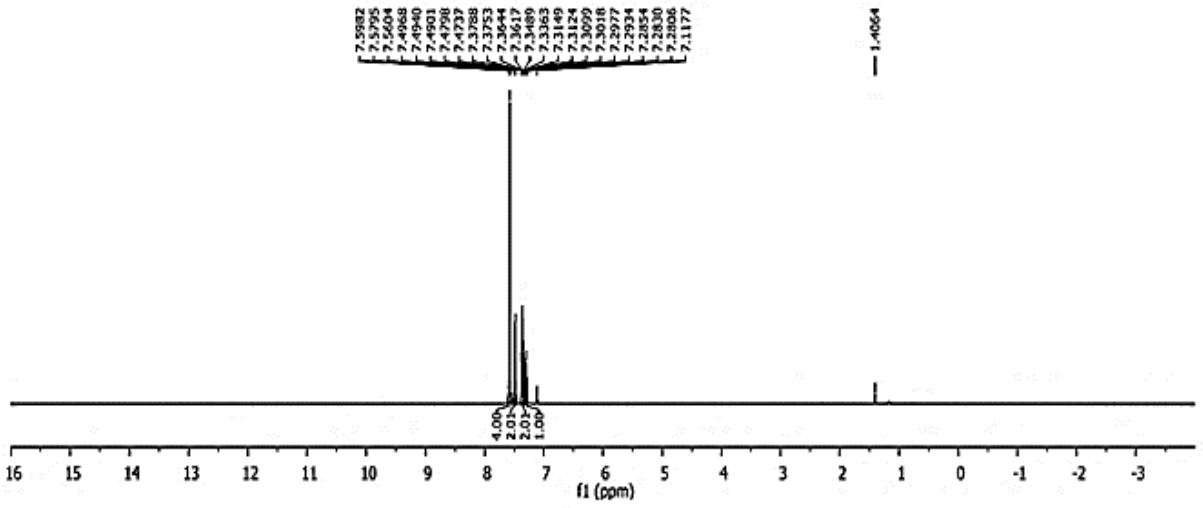
S38. The ^{13}CNMR of 4-fluoro-[1,1'-biphenyl



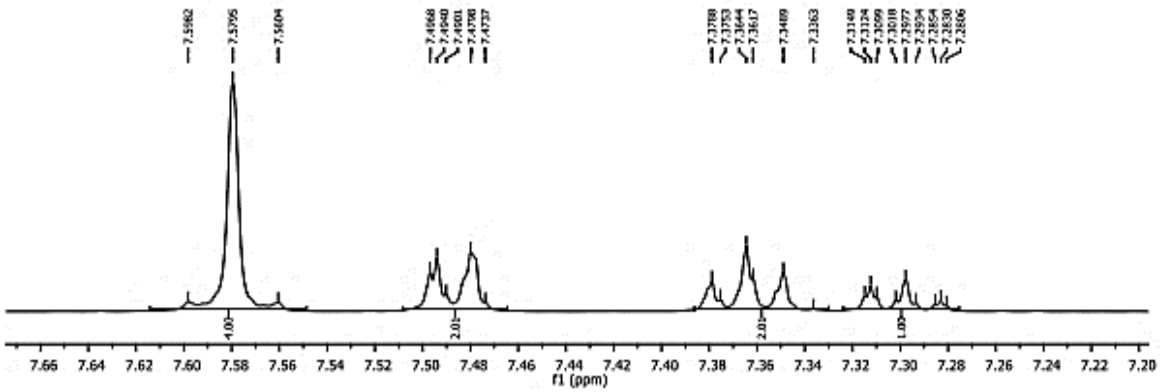
S39. The GC-Mass of 4-fluoro-[1,1'-biphenyl]

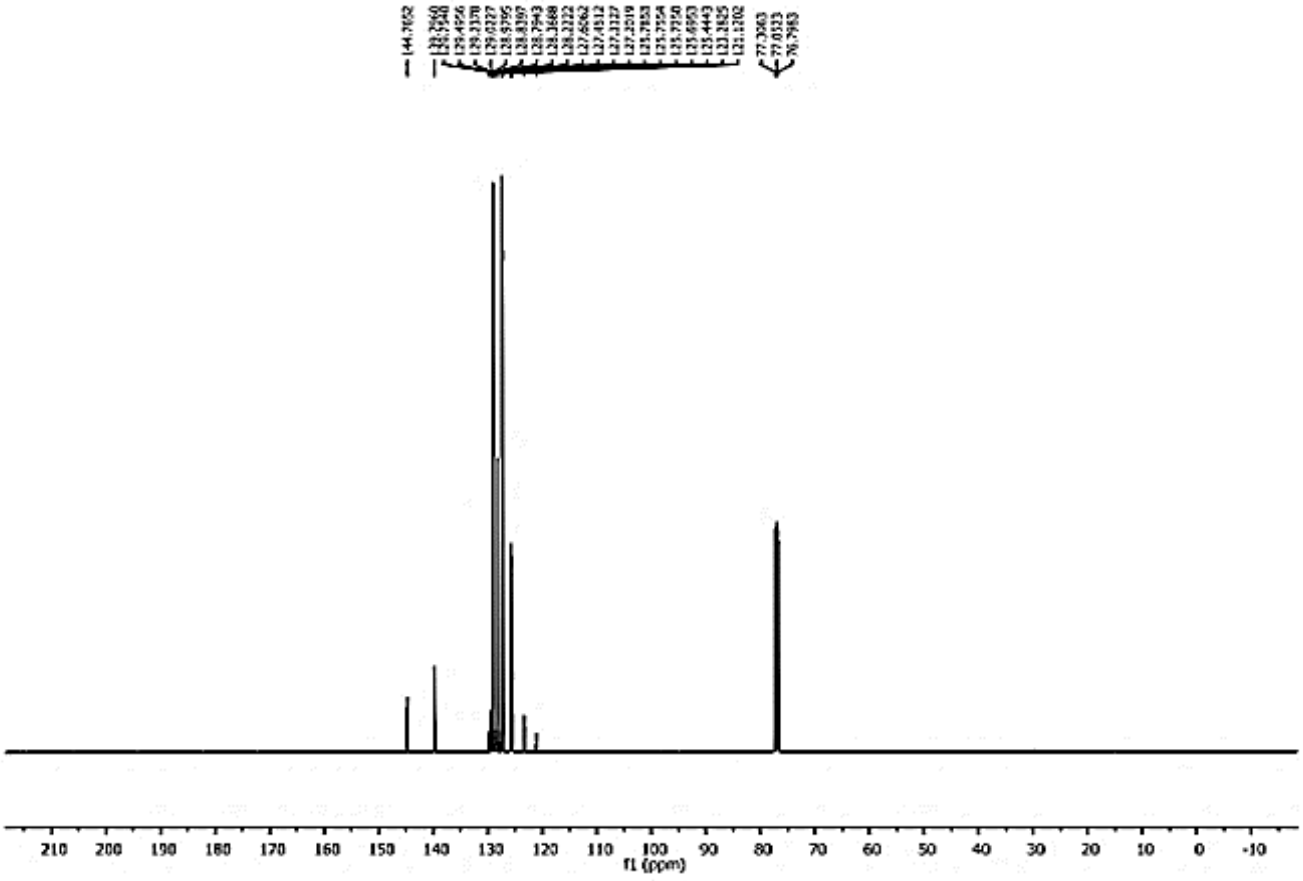
4-(trifluoromethyl)-1,1'-biphenyl





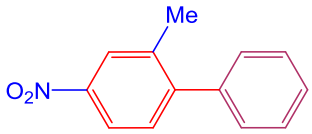
S40. The ^{1}HNMR of 4-(trifluoromethyl)-1,1'-biphenyl

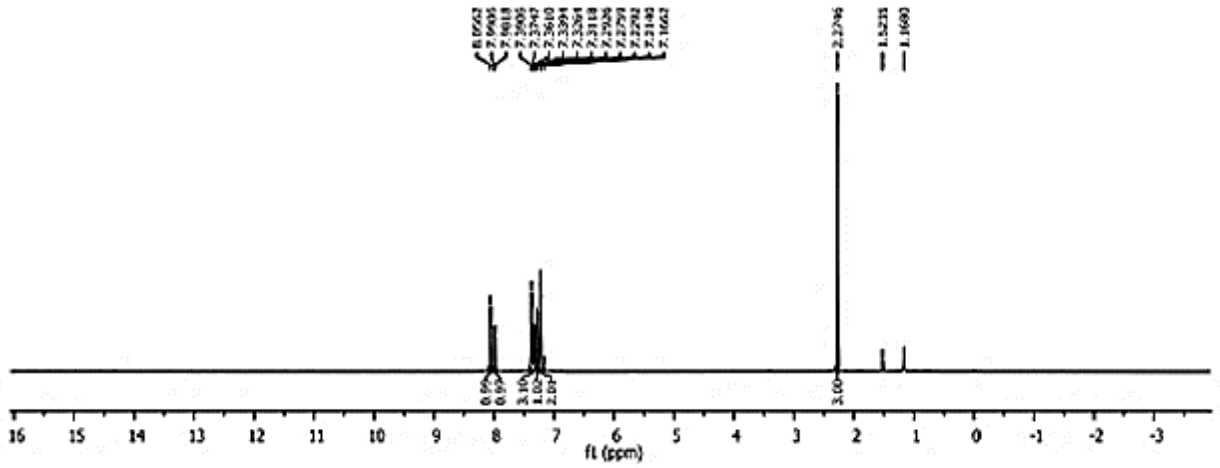




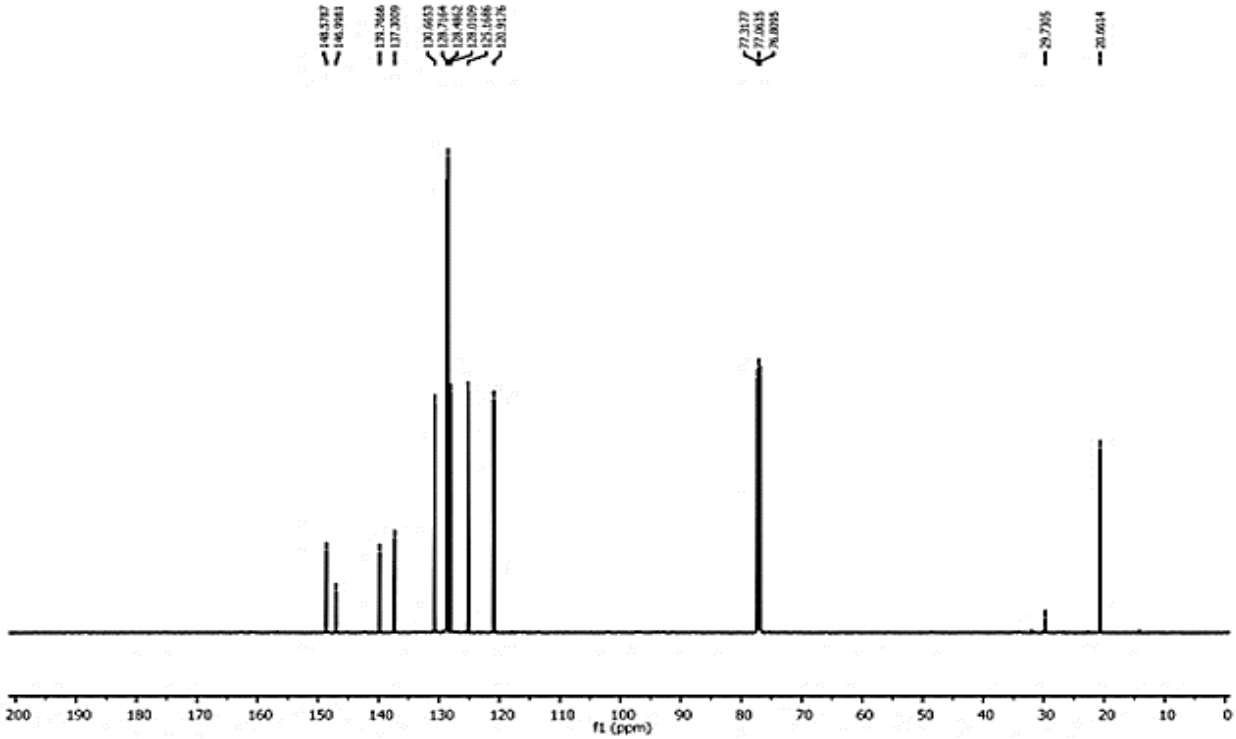
S41. The ^{13}CNMR of 4-(trifluoromethyl)-1,1'-biphenyl

2-methyl-4-nitro-1,1'-biphenyl





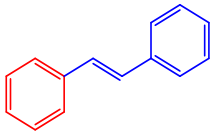
S42. The ^{1}HNMR of 2-methyl-4-nitro-1,1'-biphenyl



S43. The ^{13}CNMR of 2-methyl-4-nitro-1,1'-biphenyl

Heck derivatives

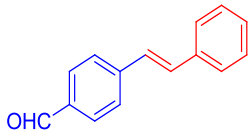
Trans-1, 2-diphenylethene (*E*-Stilbene)



White solid, Mp. 123-124°C (lit. 123.9-124.6°C)

GC–MS (m/z): 180; ^{1}HNMR (400 MHz, $CDCl_3$) δ 7.07-7.08 (s, 2H), 7.21-7.23 (m, 2H), 7.32- 7.34 (m, 4H,) 7.46-7.48 (m, 21H) ppm. ^{13}CNMR (100 MHz, $CDCl_3$) δ 122.1, 123.2, 124.3, 132.9.

(*E*)-4-styrylbenzaldehyde

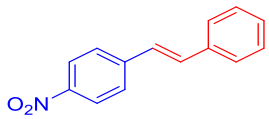


White solid, Mp. 116°C (lit. 116.0-116.8°C)

GC–MS (m/z): 208; ^{1}HNMR (400 MHz, $CDCl_3$): δ = 9.97 (s, 1H), 7.84 (d, J=8.0 Hz, 2H), 7.62 (d, J = 8.0 Hz, 2H), 7.52 (d, J= 7.6 Hz, 2H), 7.36 (t, J = 7.6 Hz, 2H), 7.28 (t, J= 7.6 Hz, 1H), 7.22 (d, J = 5.2 Hz, 1H), 7.11 (d, J=16.4 Hz, 1H).

^{13}CNMR (100 MHz, $CDCl_3$): δ = 191.5, 143.4, 136.5, 135.3, 132.2, 130.2, 128.8, 128.4, 127.3, 126.9. MS (EI) m/z: 208, 179, 165, 152, 102, 89, 76, 63, 51.

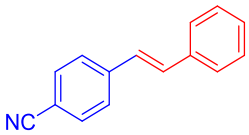
(*E*)-4-Nitrostilbene



Yellow solid, Mp. 154-155°C (lit. 154.2-155.0°C)

GC–MS (m/z): 225; ^{1}HNMR (400 MHz, $CDCl_3$) δ: 8.22 (d. *J* = 8.6 Hz, 2H, Ar-H), 7.63 (d, *J* = 8.7 Hz, 2H, Ar-H), 7.55 (d, *J* = 7.7 Hz, 2H, Ar-H), 7.40 (t, *J* = 7.6 Hz, 2H, Ar-H), 7.36-7.31 (m, 1H, Ar-H), 7.27 (d, *J* = 16.3 Hz, 1H, CH=), 7.14 (d, *J* = 16.3, 1H, Hz, CH=). ^{13}CNMR (100 MHz, $CDCl_3$), δ = 125.2, 127.4, 128.0, 128.2, 130.0, 134.4, 137.3, 145.0, 147.8 ppm.

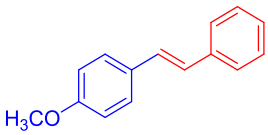
(*E*)-4-Styrylbenzonitrile



White solid, Mp. 114-115°C (lit. 114.9-115.2°C)

GC–MS (m/z): 205; ^{1}HNMR (400 MHz, $CDCl_3$): δ 7.09 (d, 1H, *J* = 16.0 Hz), 7.22 (d, 1H, *J* = 16.0 Hz), 7.31-7.34 (t, 1H, *J* = 4.0 Hz), 7.38-7.41 (t, 2H, *J* = 4.0 Hz), 7.54 (d, 2H, *J* = 7.2 Hz), 7.58 (d, 2H, *J* = 8.4 Hz), 7.64 (d, 2H, *J* = 8.4 Hz) ppm; ^{13}CNMR (100 MHz, $CDCl_3$), δ 111.7, 120.2, 127.8, 128.0, 128.1, 129.8, 130.0, 133.5, 133.6, 137.4, 142.9 ppm.

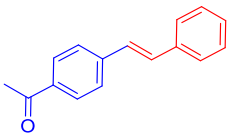
(*E*)-1-methoxy-4-styrylbenzene



White solid, Mp. 135-136°C (lit. 135.3-135.9°C)

GC–MS (m/z): 210; ^{1}H-NMR (400 MHz, $CDC1_3$) δ (ppm): 3.70 (3H, s), 6.78 (1H, d, *J* = 8.75 Hz), 6.90 (1H, d, *J* = 10.75 Hz), 7.00-7.38 (9H, m). ^{13}C-NMR (100 MHz, $CDCl_3$) δ (ppm): 55.3, 114.2, 126.3, 126.6, 127.25, 127.8, 128.2, 128.7, 130.2, 137.7, 159.3.

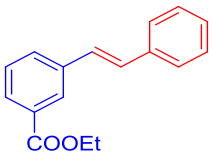
(*E*)-1-(4-styrylphenyl) ethanone



White solid, Mp. 141-142°C (lit. 141.3-142°C)

GC–MS (m/z): 222; ^{1}HNMR (400 MHz, $CDCl_3$): δ 7.97-7.95 (d, 2H), 7.58-7.57 (d, 2H), 7.53-7.52 (d, 2H), 7.40-7.36 (t, 2H), 7.29-7.25 (t, 1H), 7.22-7.20 (d, 1H), 7.12 (d, 1H), 2.64 (s, 3H), ^{13}CNMR (100 MHz, $CDCl_3$): δ 197.6, 142.1, 136.7, 136.1, 131.5, 128.9, 128.8, 128.4, 127.6, 126.8, 126.5, 26.8.

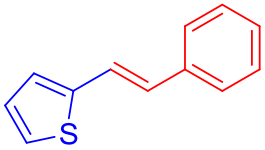
(*E*)-Ethyl-3-styrylbenzoate



Oily liquid

GC–MS (m/z): 252; ^{1}HNMR ($CDCl_3$, 400 MHz): δ (ppm) 8.22 (s, 1H), 7.95 (d, *J* = 7.7 Hz, 1H), 7.70 (d, *J* = 7.7 Hz, 1H), 7.55 (d, *J* = 7.4 Hz, 2H), 7.46–7.27 (m, 4H), 7.21 (d, *J* = 16.0 Hz, 1H), 7.15 (d, *J* = 16.0 Hz, 1H), 4.43 (q, *J* = 7.1 Hz3H), 1.44 (t, *J* = 7.1 Hz); ^{13}CNMR ($CDCl_3$, 100 MHz): δ (ppm) 166.6, 137.7, 137.0, 131.0, 130.7, 129.9, 128.8, 128.7, 128.5, 128.0, 127.6, 127.5, 126.7.

(*E*)-2-styrylthiophene

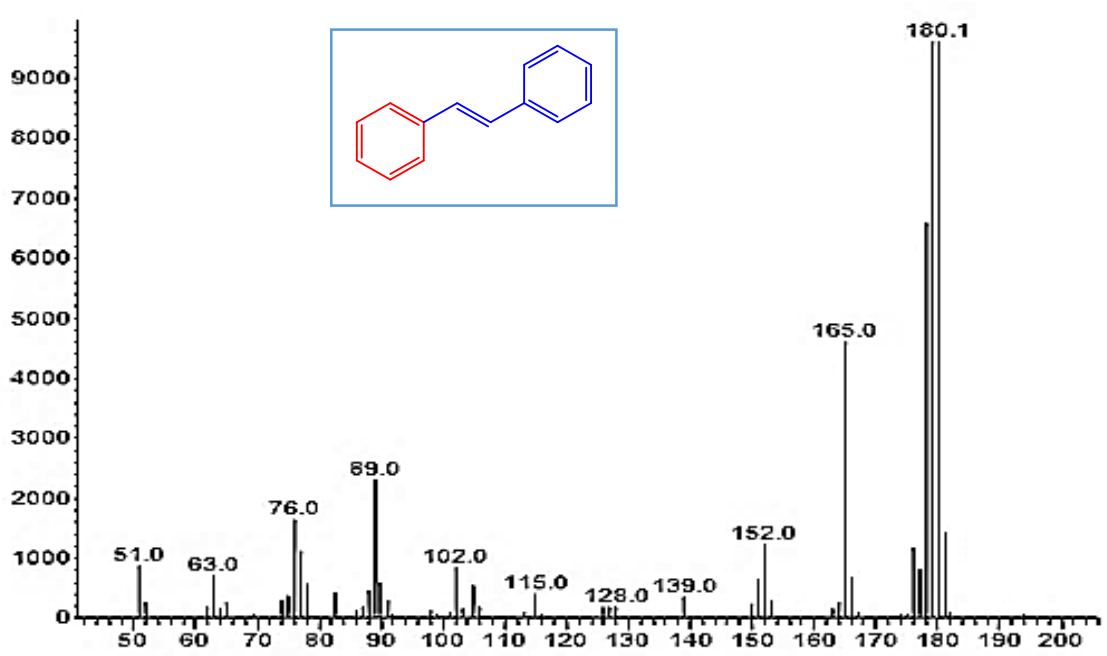


white solid, MP. 111-112°C (lit. 112°C)

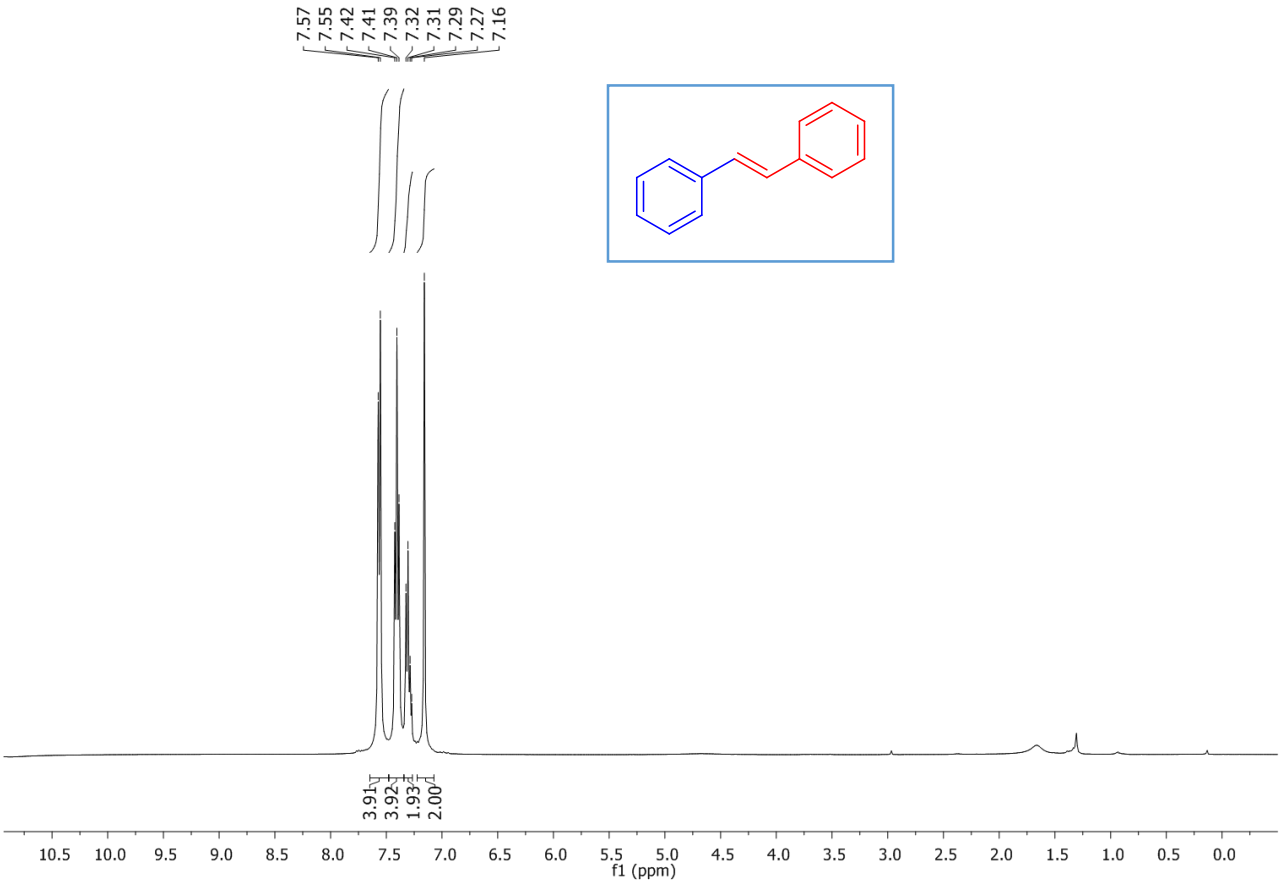
GC–MS (m/z): 186; ^{1}H NMR ($CDCl_3$, 400 MHz): δ (ppm) 7.49 (d, *J* = 7.5 Hz, 2H), 7.37 (t, *J* = 7.5 Hz, 2H), 7.28 (d, *J* = 5.1 Hz, 1H), 7.24 (d, *J* = 16 Hz, 1H), 7.21 (d, *J* = 4.9 Hz, 1H), 7.09 (d, *J* = 3.4 Hz, 1H), 7.03 (t, *J* = 4 Hz, 1H), 6.95 (d, *J* = 16.2 Hz, 1H); ^{13}CNMR ($CDCl_3$, 100 MHz): δ (ppm) 142.9, 137.0, 128.7, 128.4, 127.6, 126.3, 126.1, 124.3, 121.8.

GC-MS and NMR Spectra for Heck Coupling Reaction

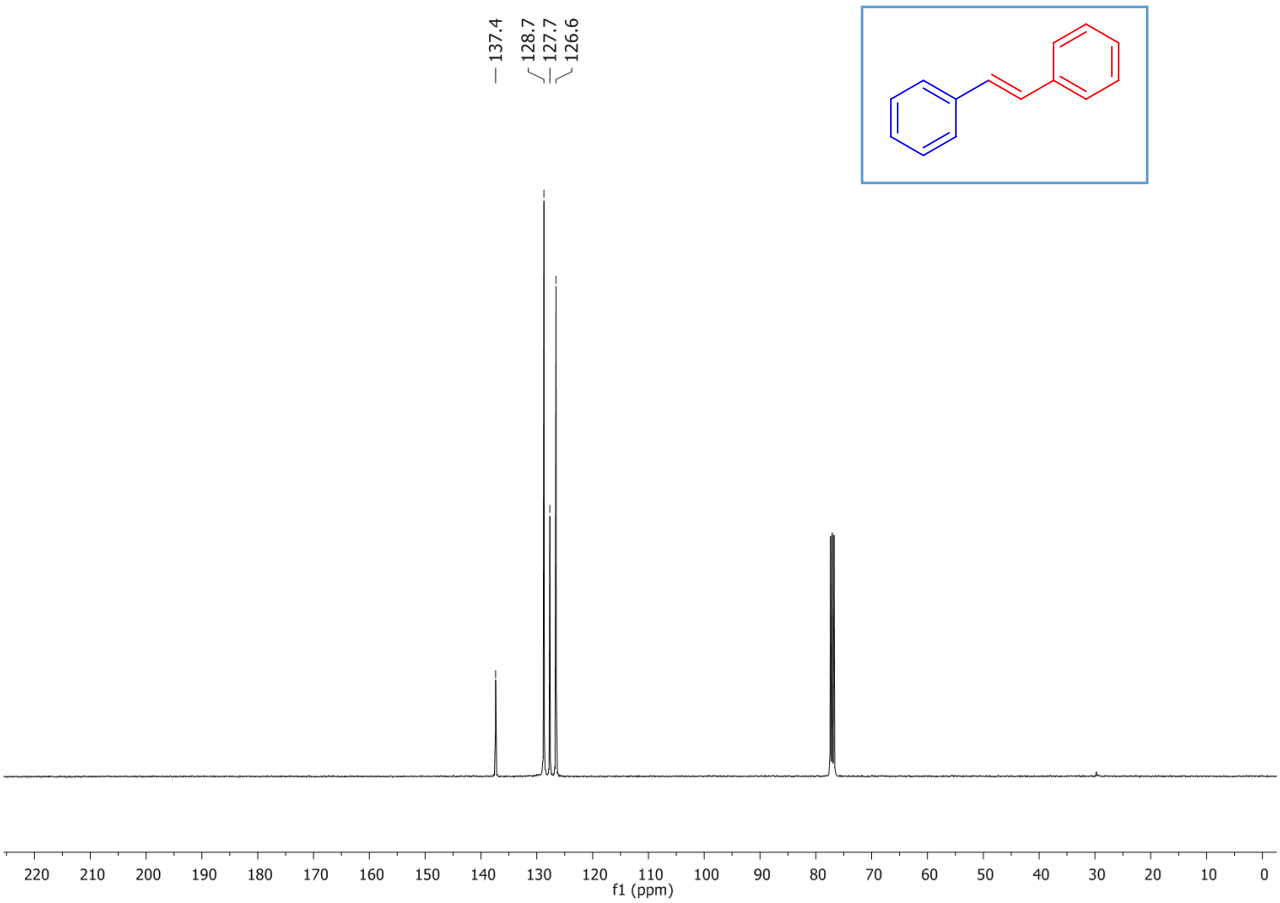
1, 2-diphenylethene



S44. GC-Mass of 1, 2-diphenylethene

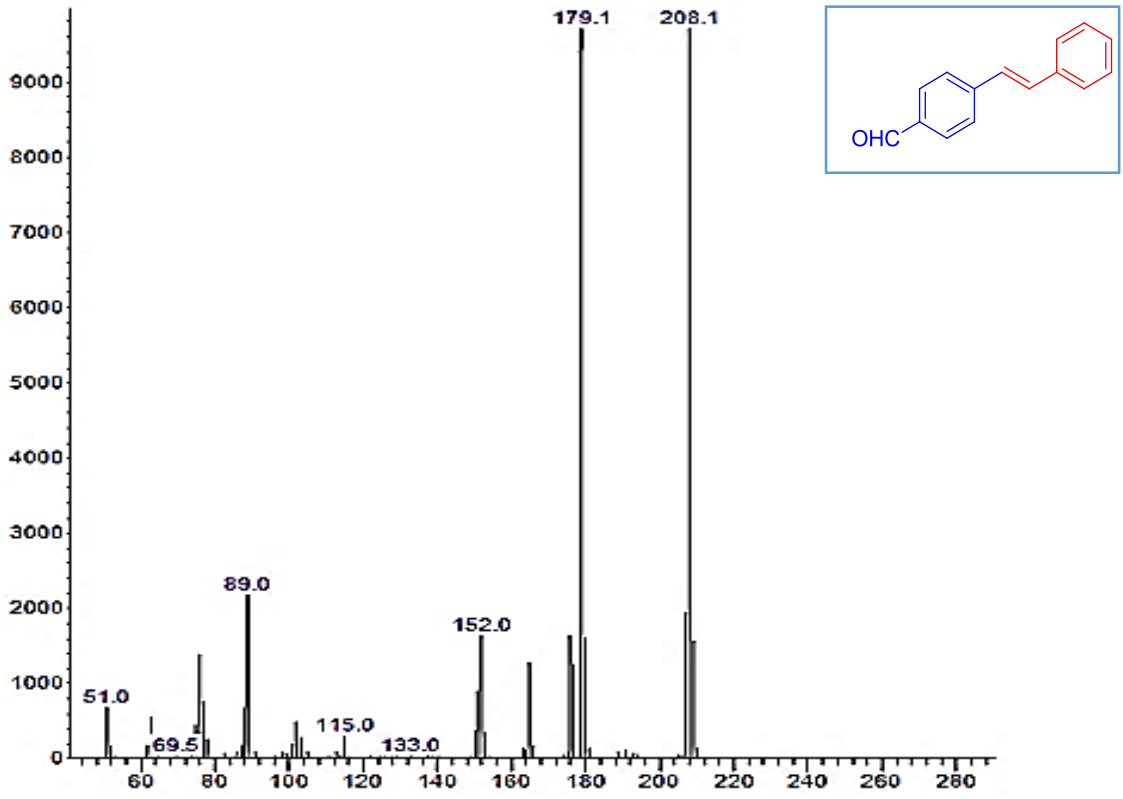


S45. ^{1}HNMR of 1, 2-diphenylethene

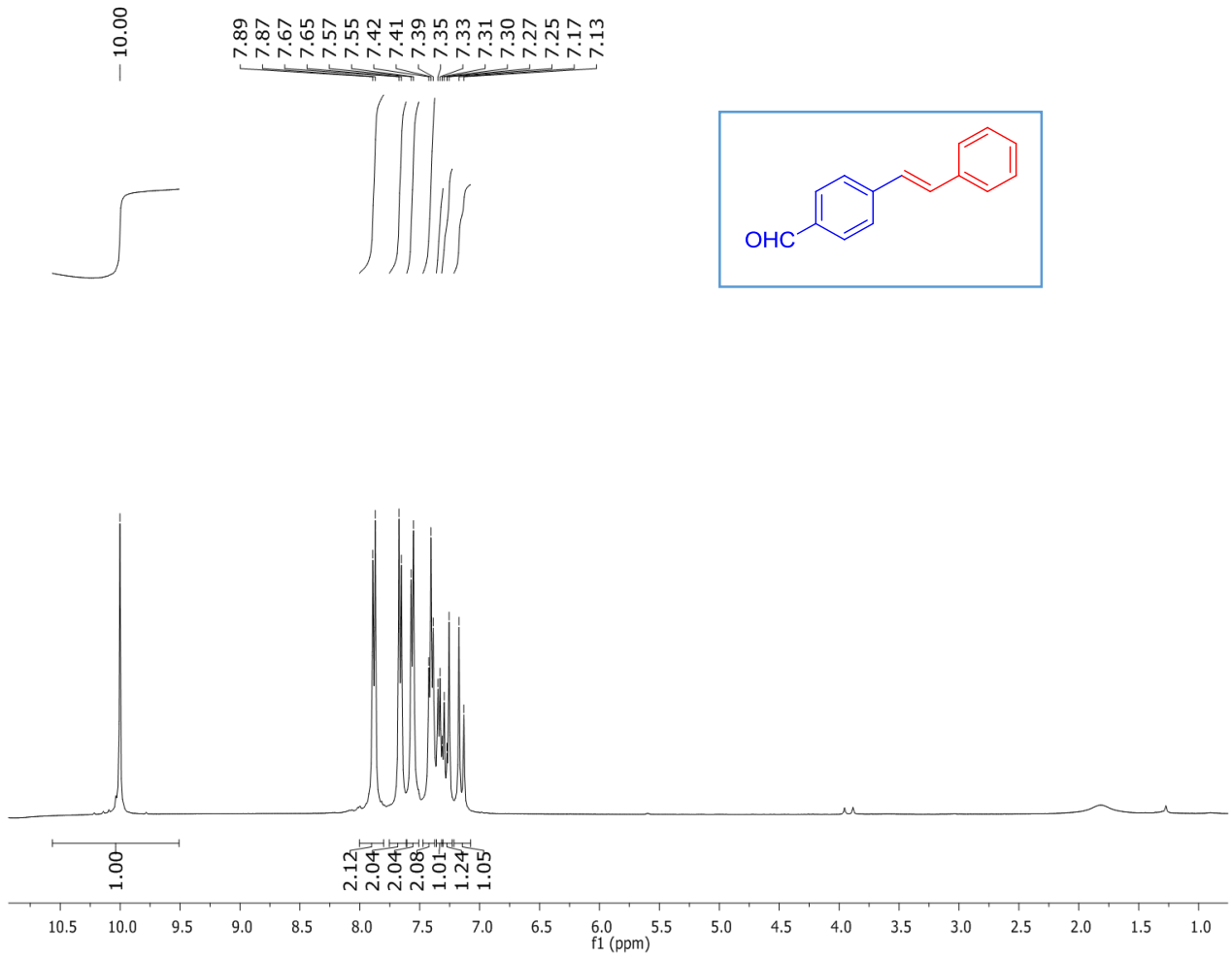


S.46. ^{13}CNMR of 1, 2-diphenylethene

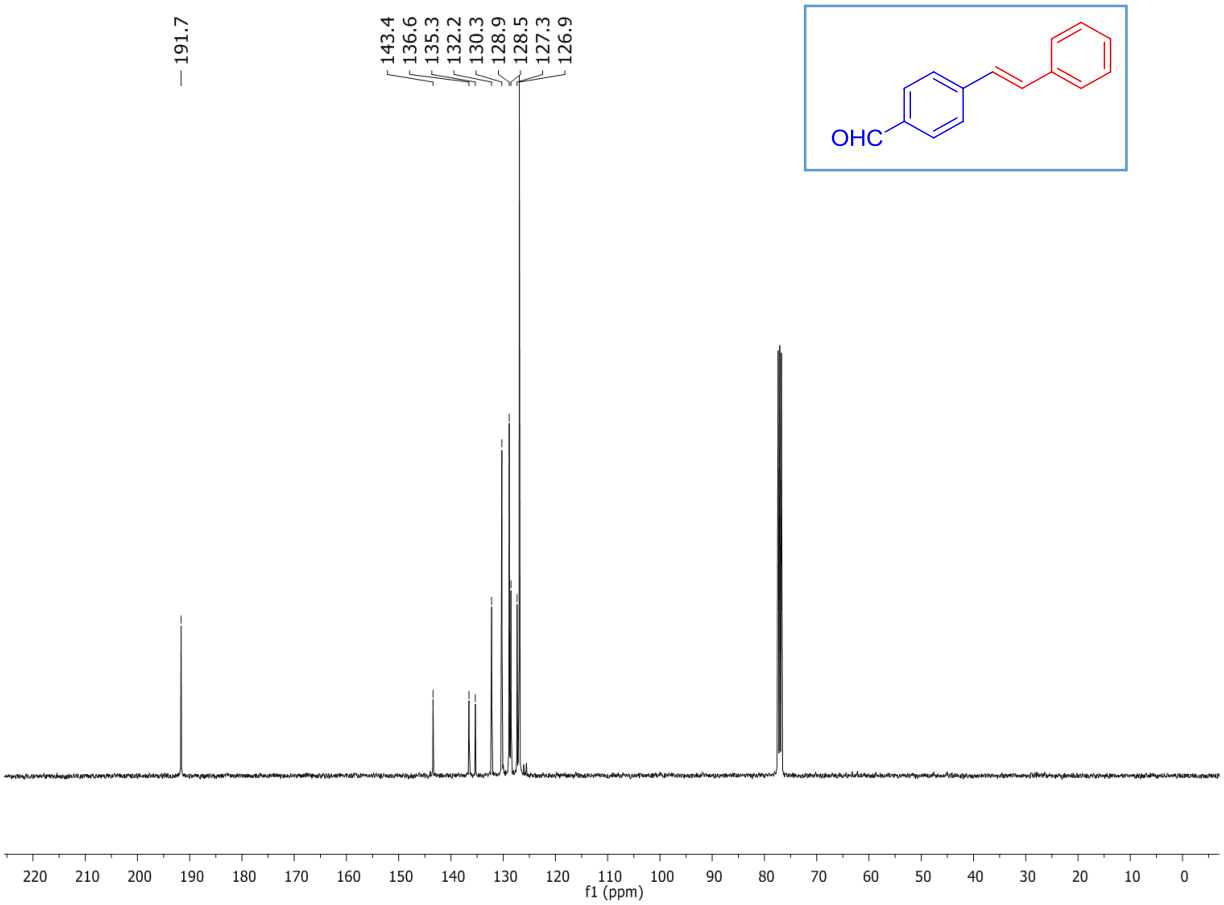
(*E*)-4-styrylbenzaldehyde



S47. GC-Mass of (*E*)-4-styrylbenzaldehyde

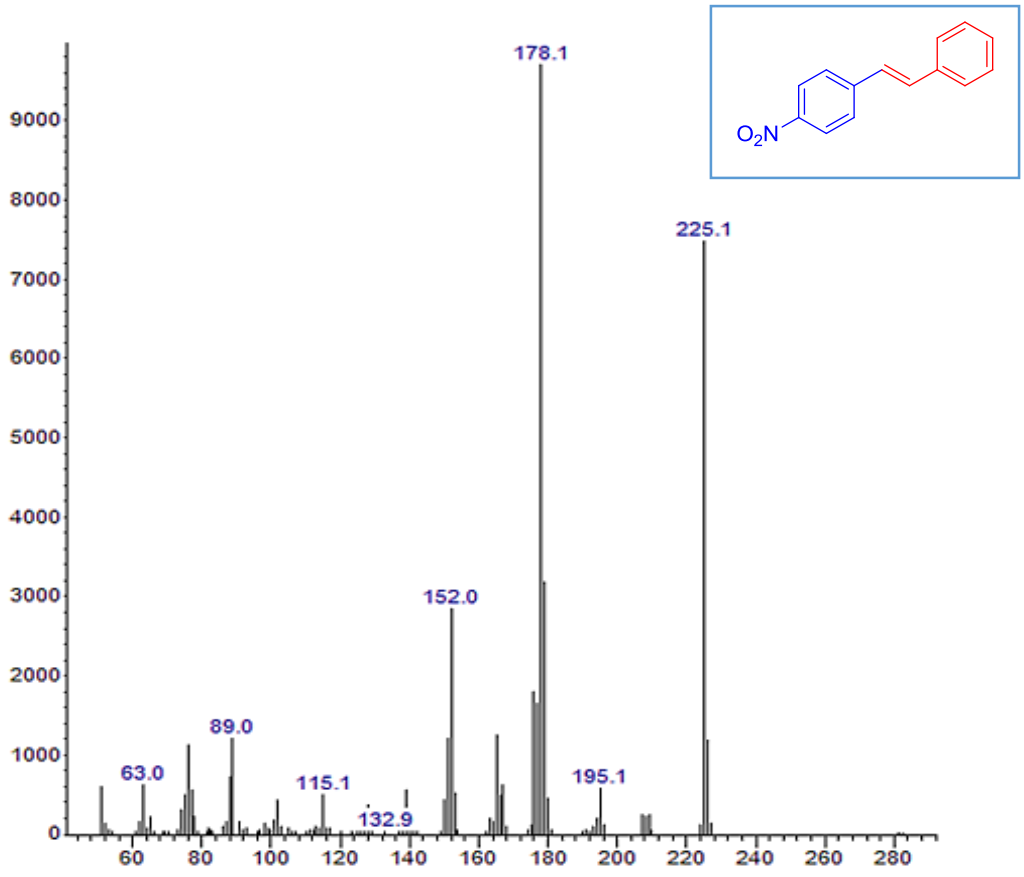


S48. ^{1}HNMR of (*E*)-4-styrylbenzaldehyde

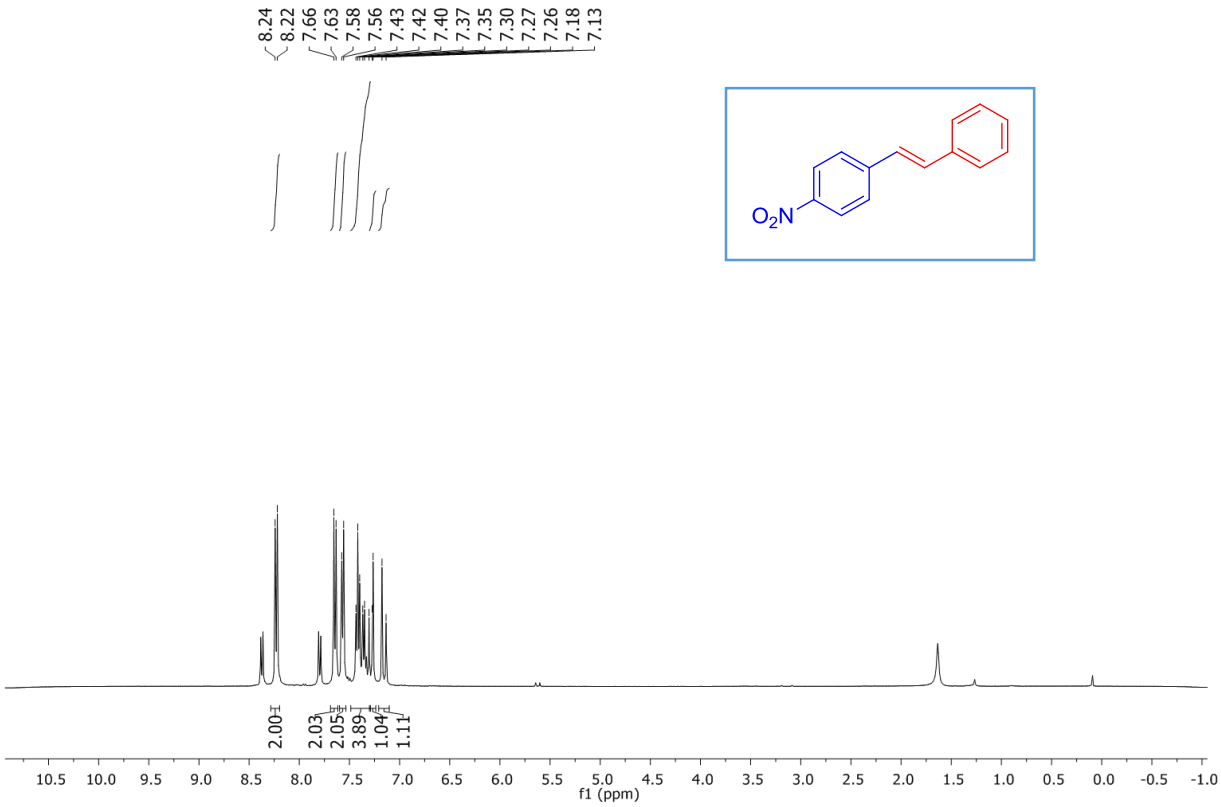


S49. ^{13}CNMR of (*E*)-4-styrylbenzaldehyde

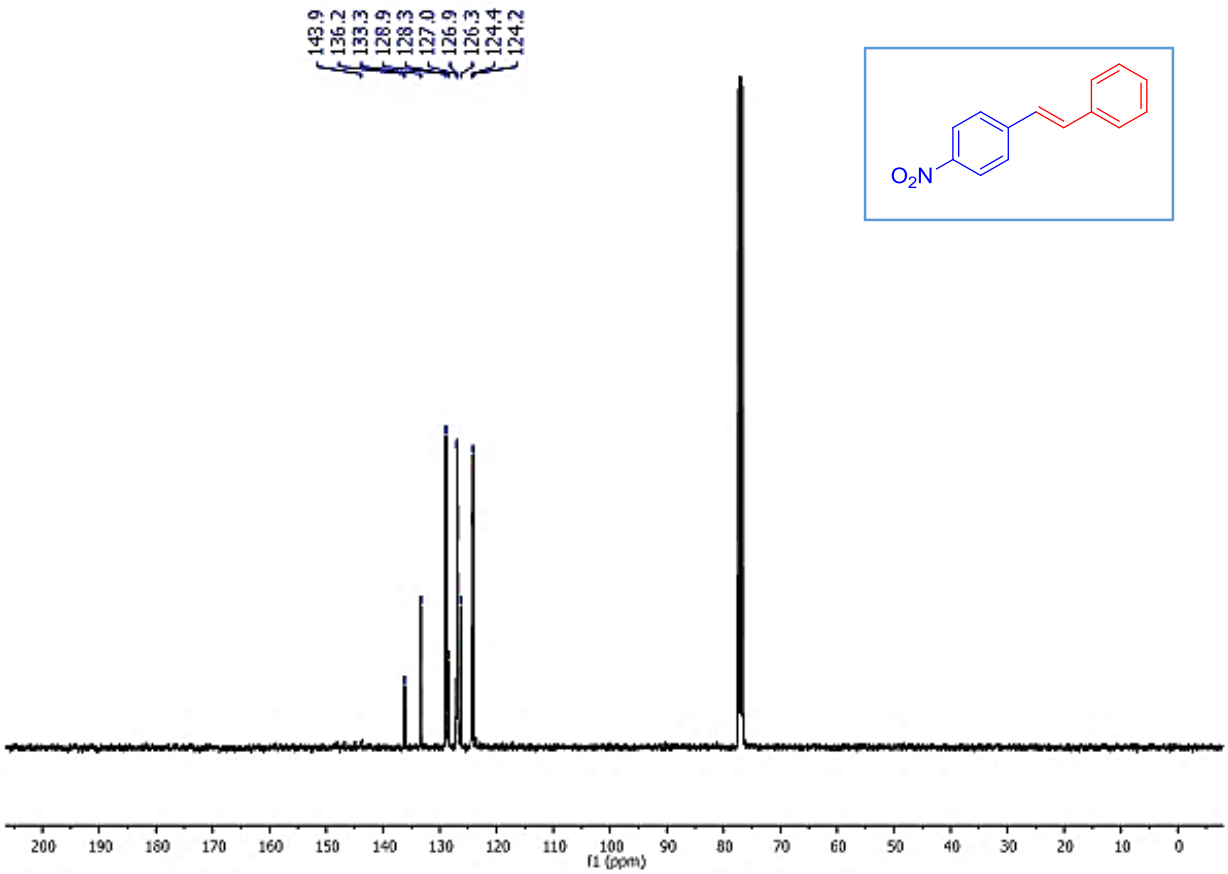
(*E*)-1-nitro-4-styrylbenzene



S50. GC-Mass of (*E*)-1-nitro-4-styrylbenzene

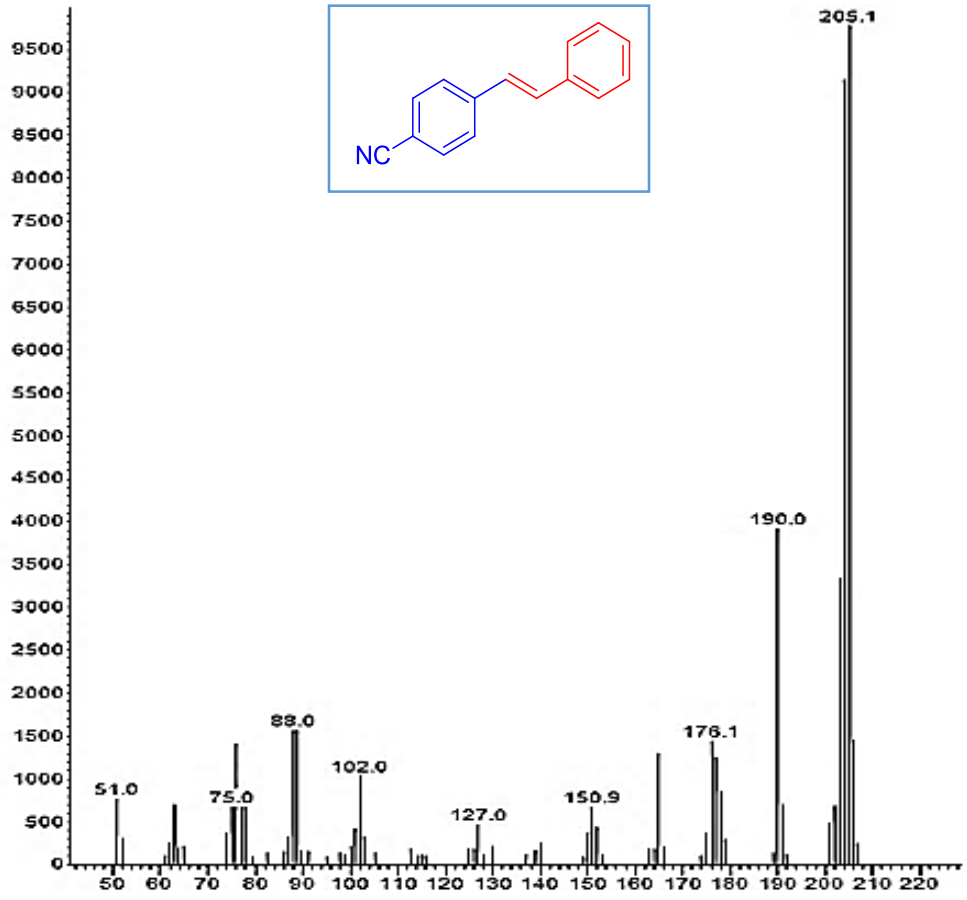


S51. ^{1}HNMR for (*E*)-1-nitro-4-styrylbenzene

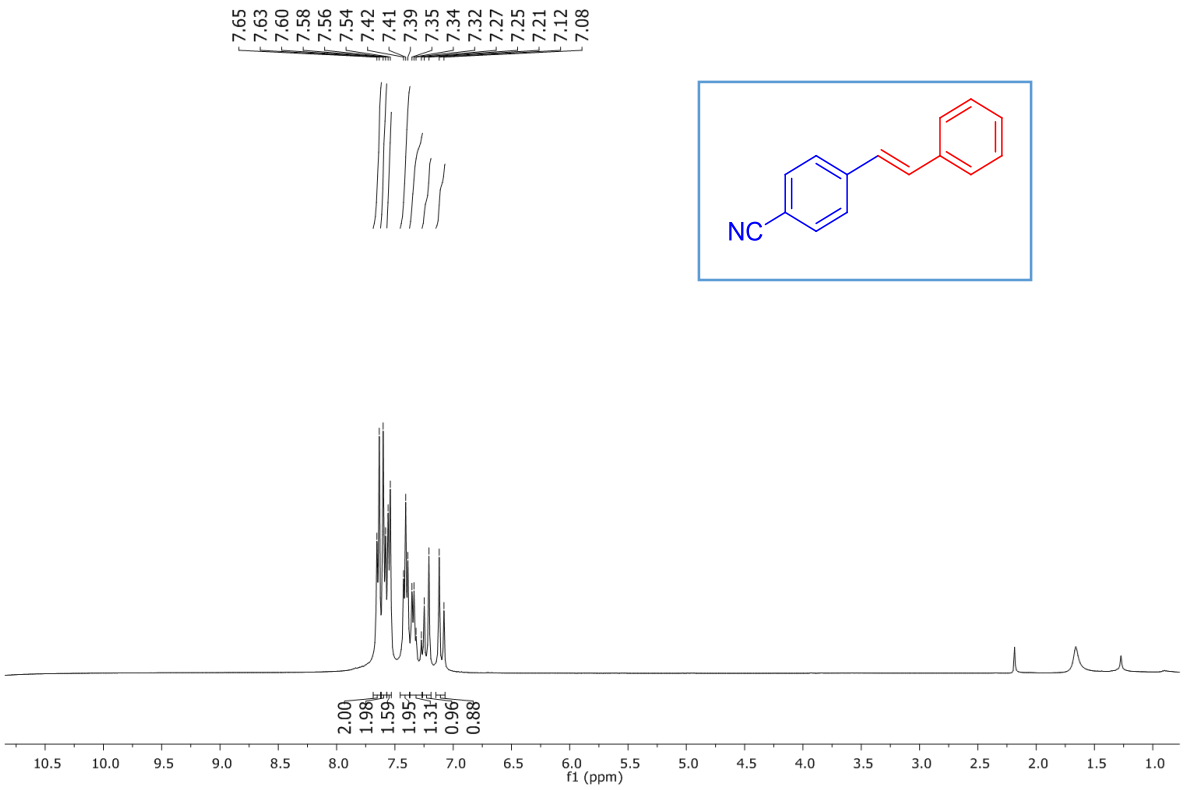


S52. ^{13}CNMR for (*E*)-1-nitro-4-styrylbenzene

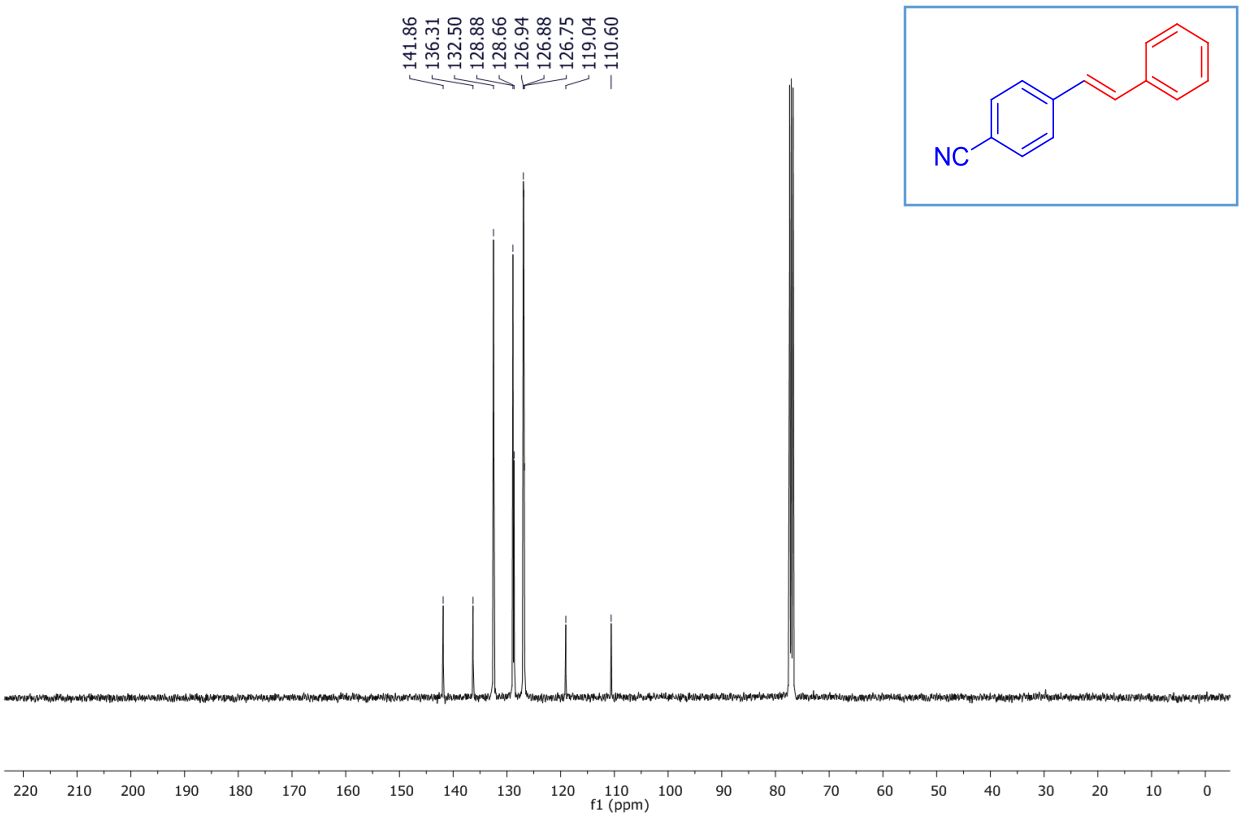
(*E*)-4-styrylbenzonitrile



S53. GC-Mass for (*E*)-4-styrylbenzonitrile

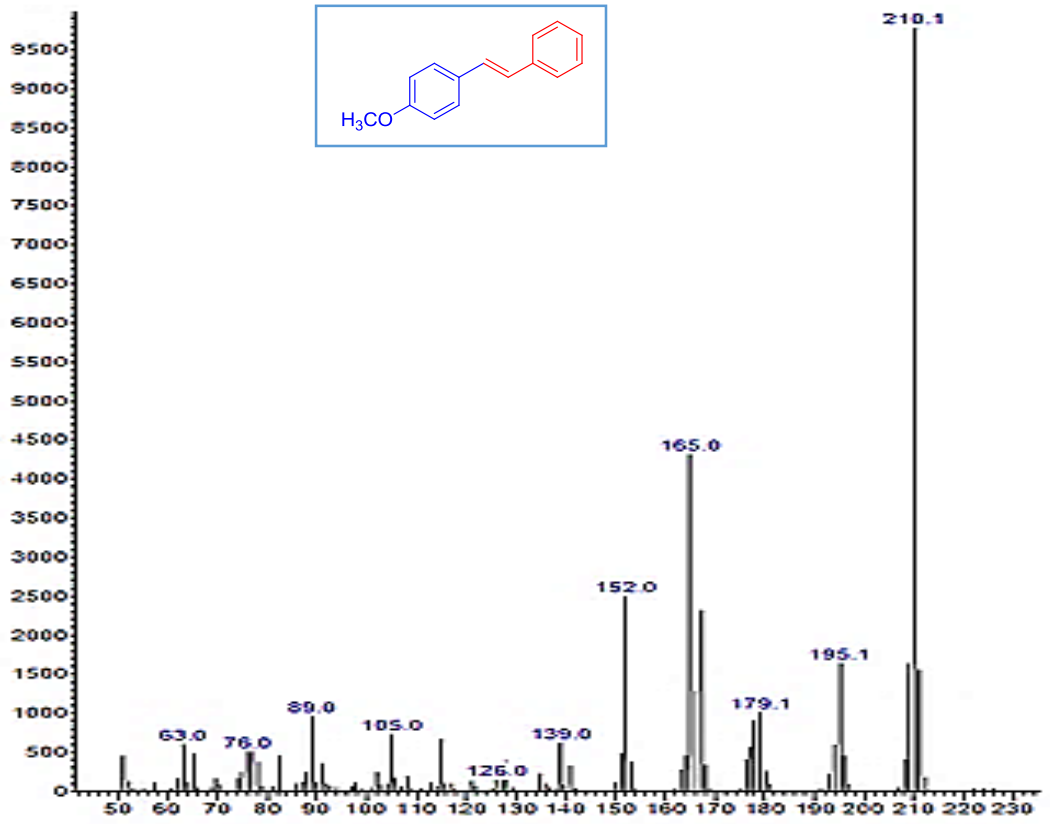


S54. ^{1}HNMR for (*E*)-4-styrylbenzonitrile

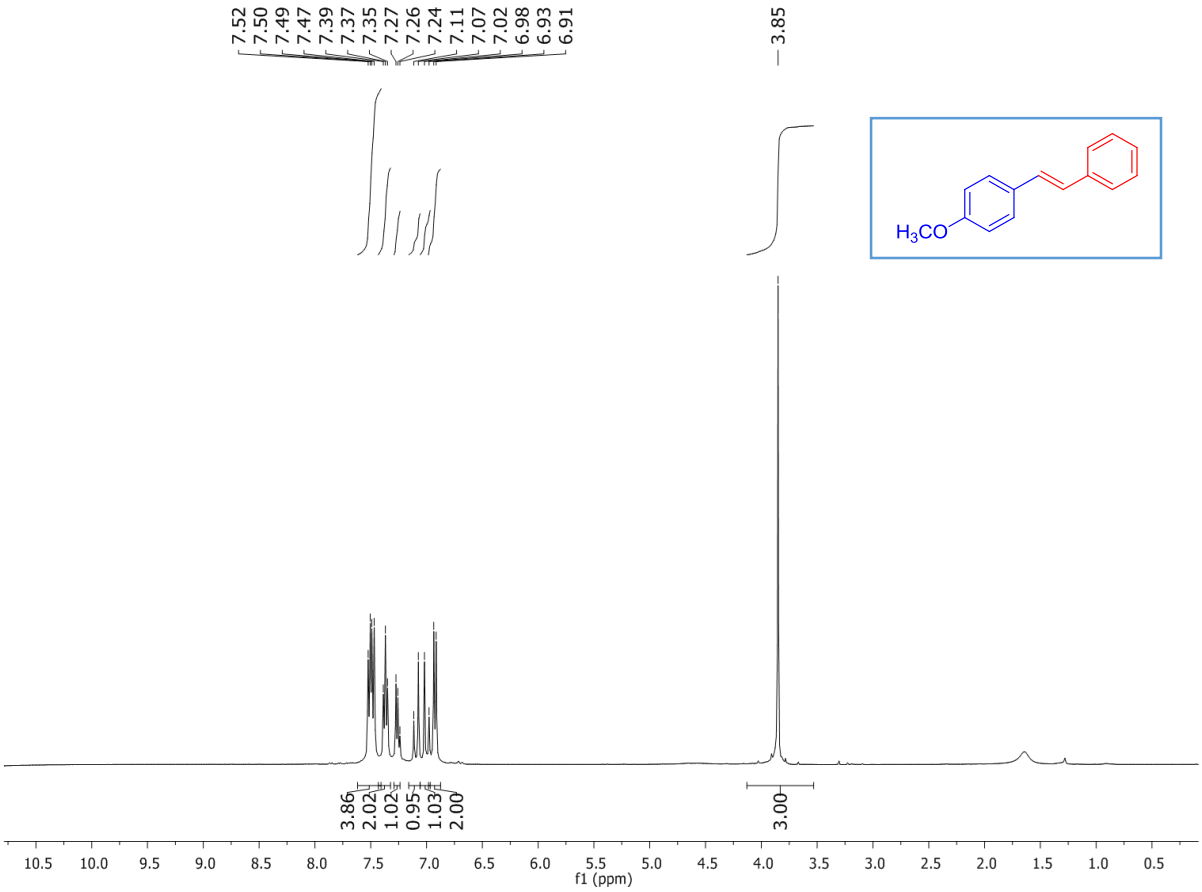


S55. ^{13}CNMR for (*E*)-4-styrylbenzonitrile

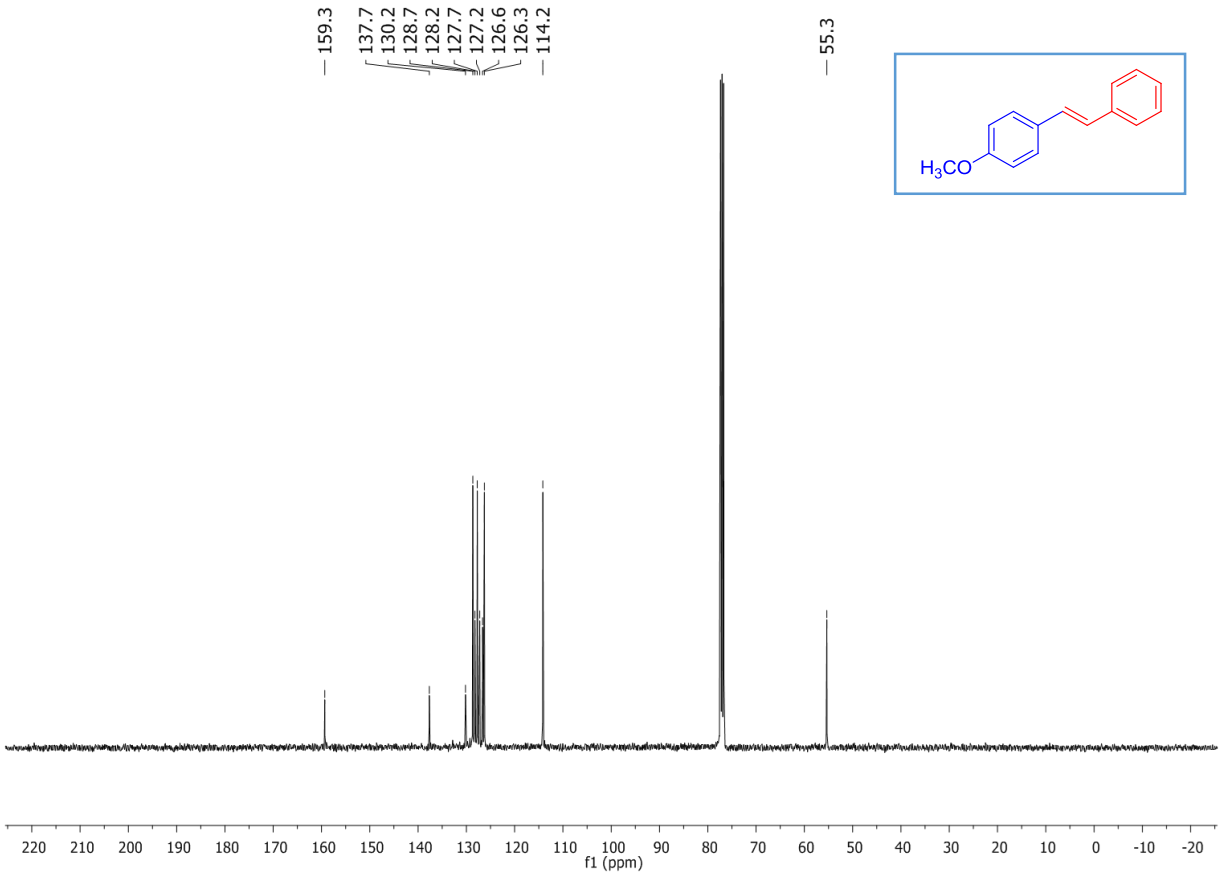
(*E*)-1-methoxy-4-styrylbenzene



S56. GC-Mass for (*E*)-1-methoxy-4-styrylbenzene

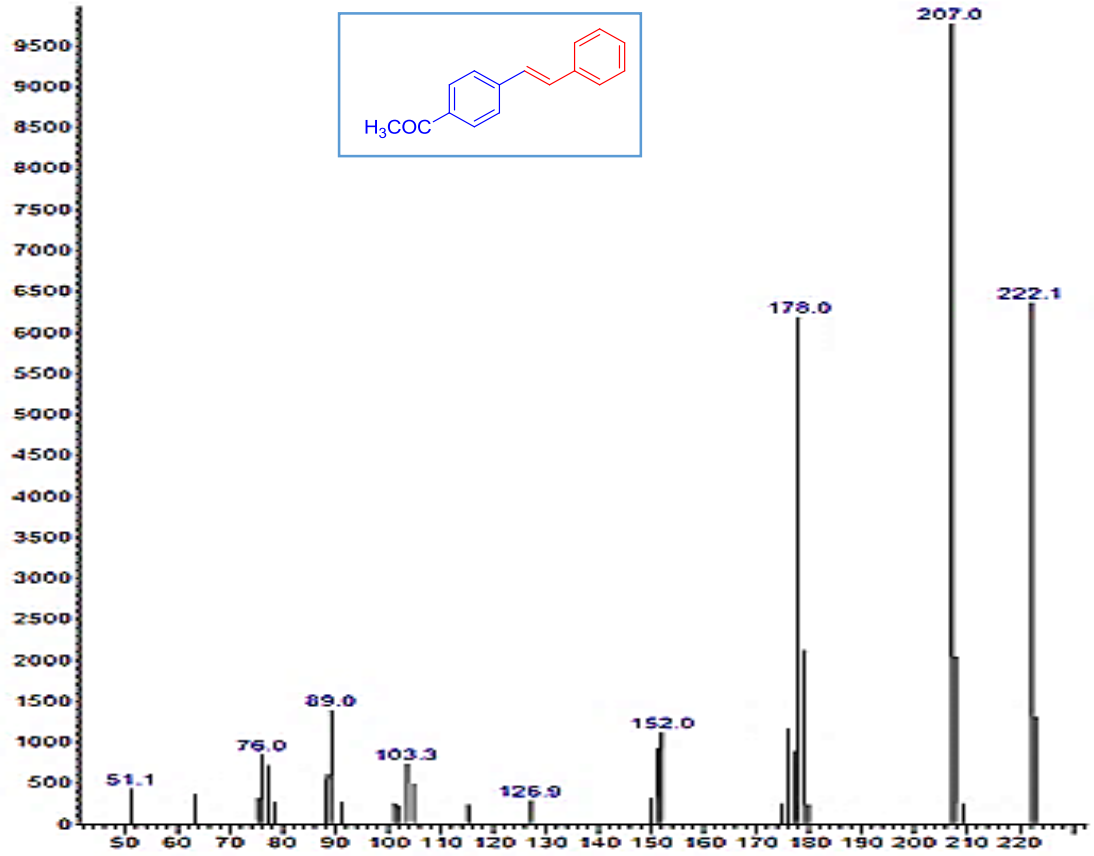


S57. ^{1}HNMR for (*E*)-1-methoxy-4-styrylbenzene

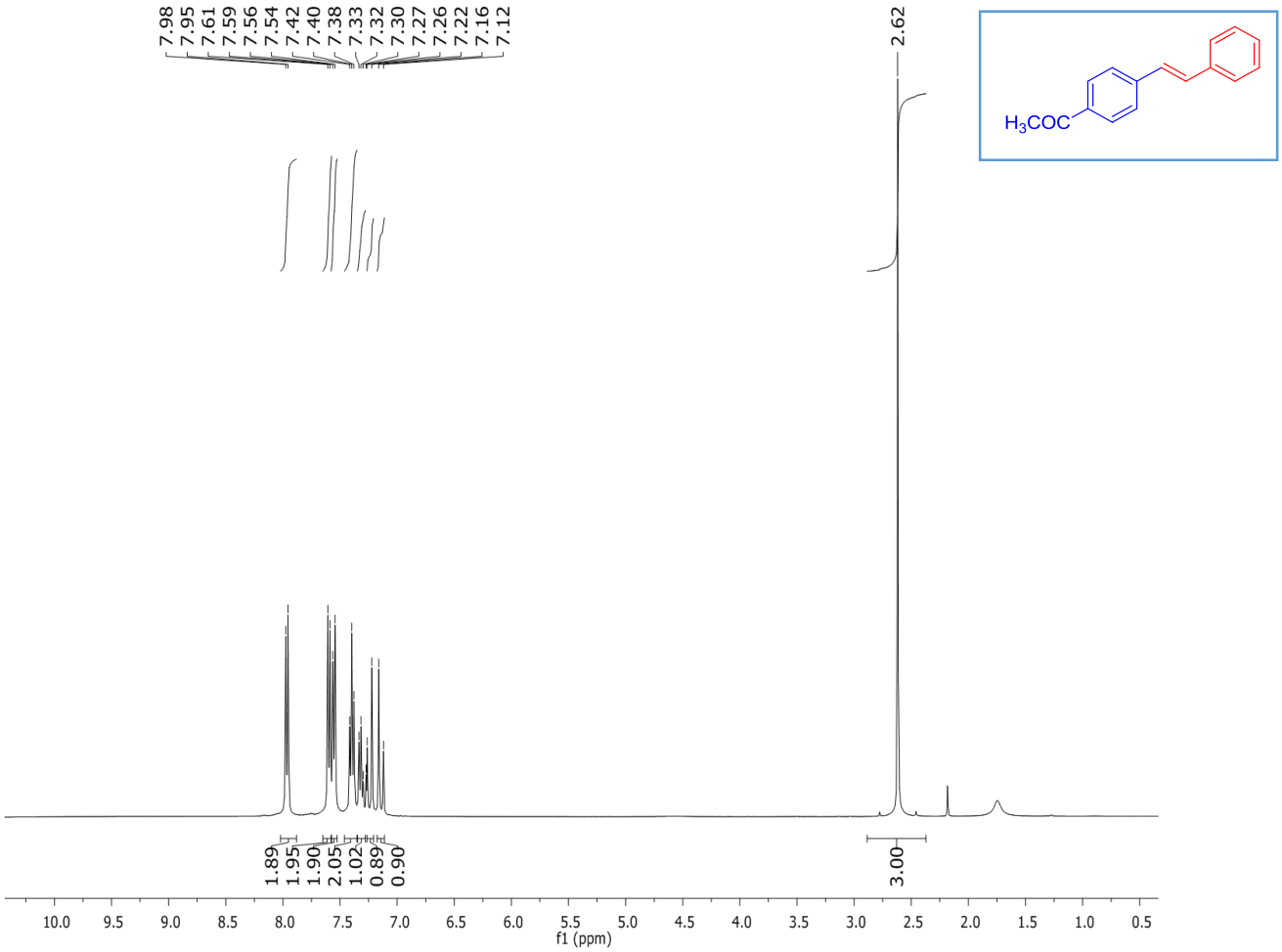


S58. ^{13}CNMR for (*E*)-1-methoxy-4-styrylbenzene

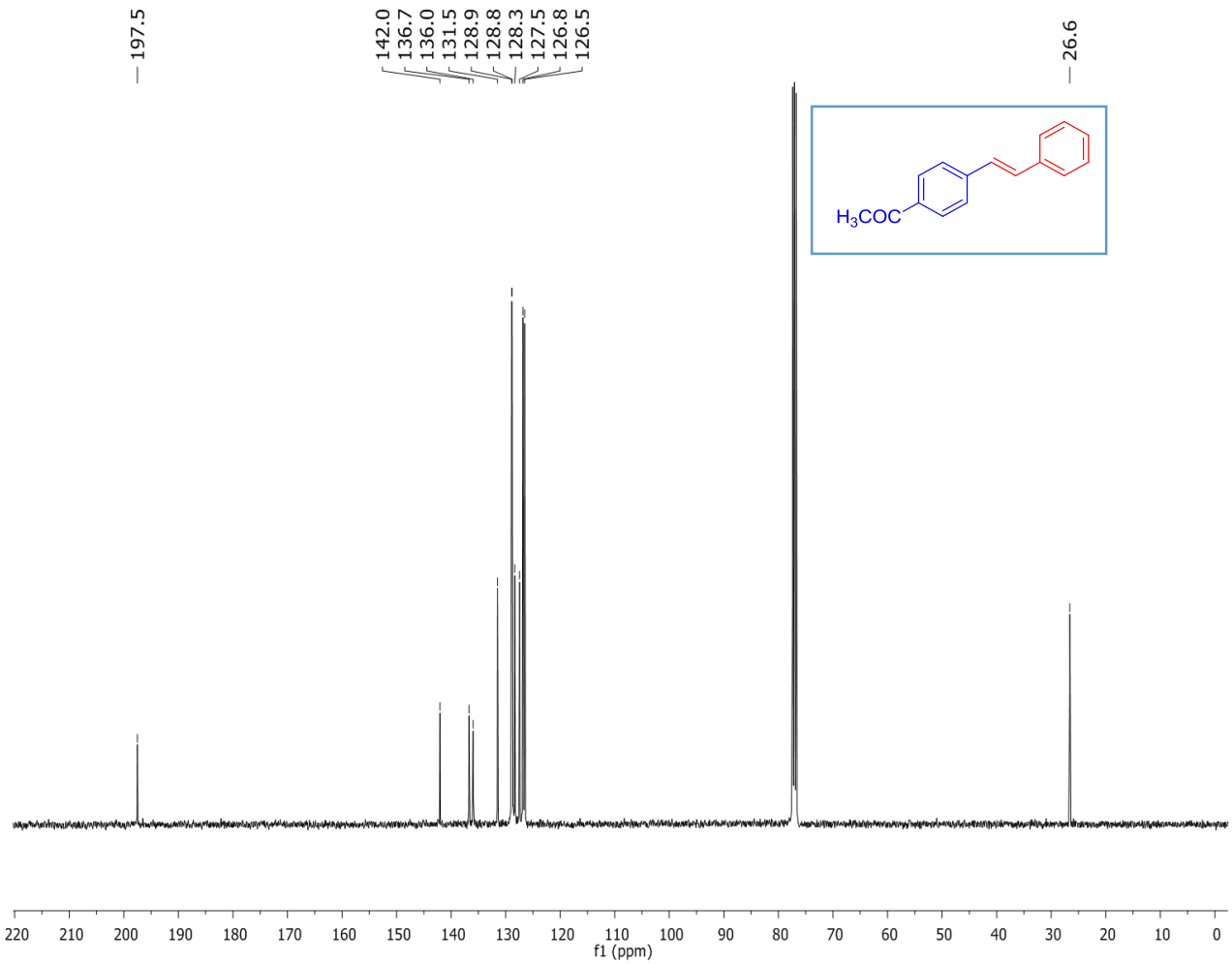
(*E*)-1-(4-styrylphenyl)ethan-1-one



S59. GC-Mass for (*E*)-1-methoxy-4-styrylbenzene

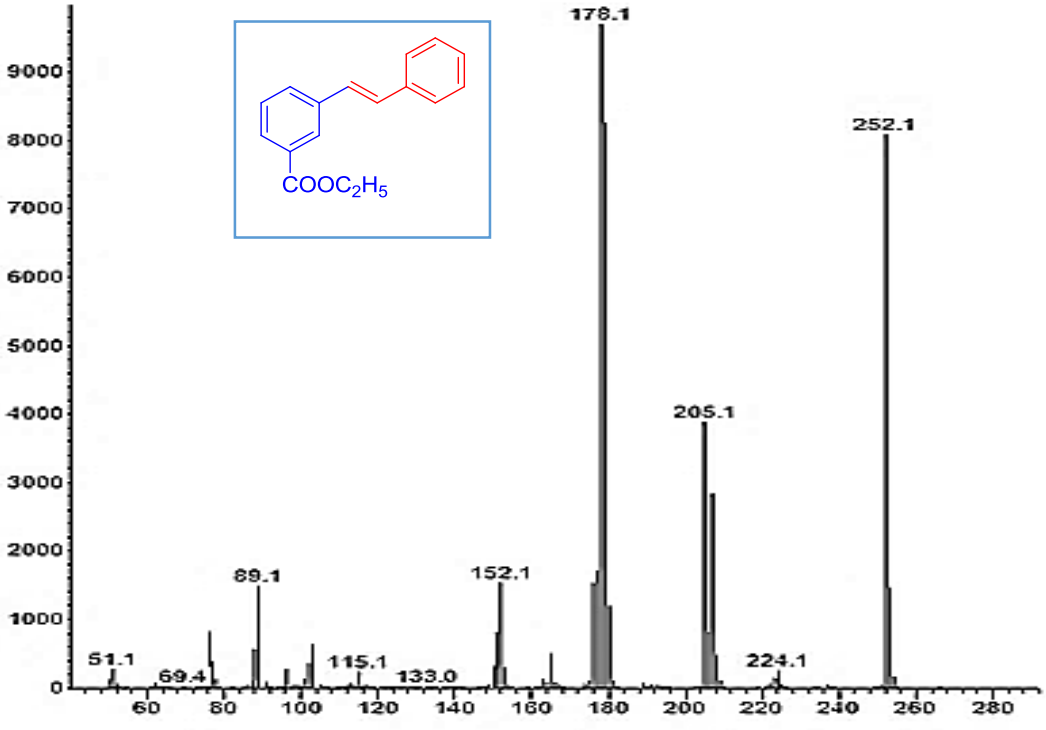


S60. ^{1}HNMR for (*E*)-1-(4-styrylphenyl)ethan-1-one

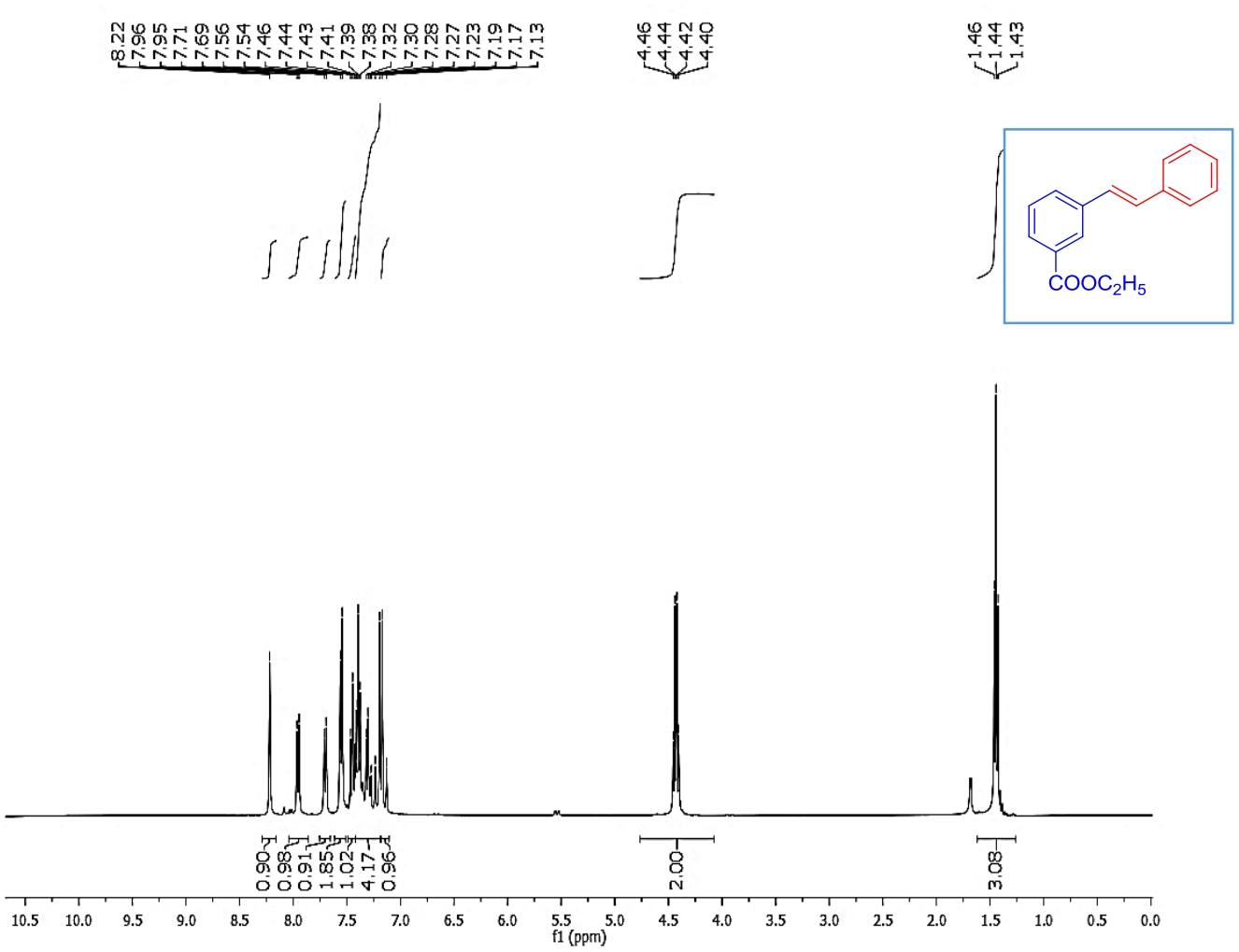


S61. ^{13}CNMR for (*E*)-1-(4-styrylphenyl)ethan-1-one

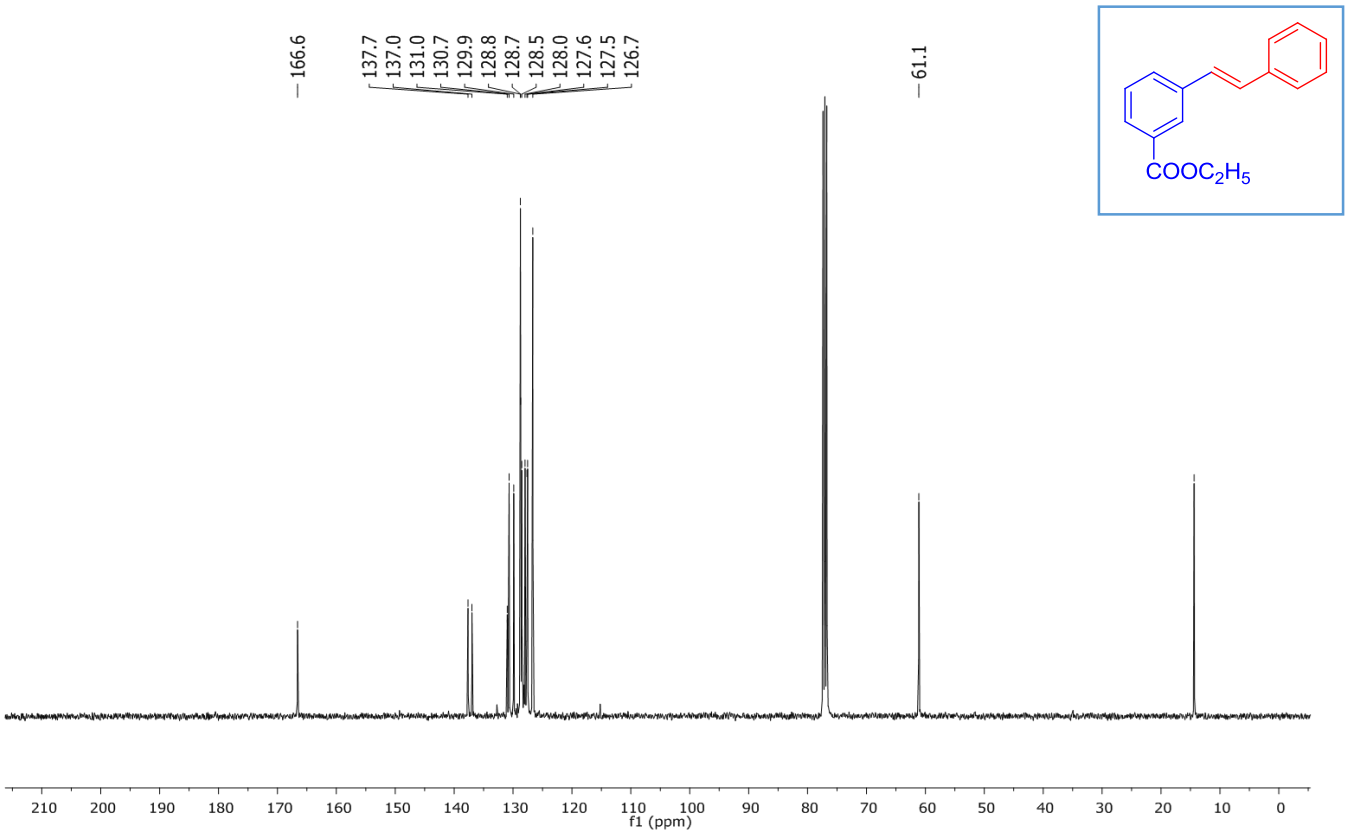
Ethyl (*E*)-3-styrylbenzoate



S62. GC-Mass for Ethyl (*E*)-3-styrylbenzoate

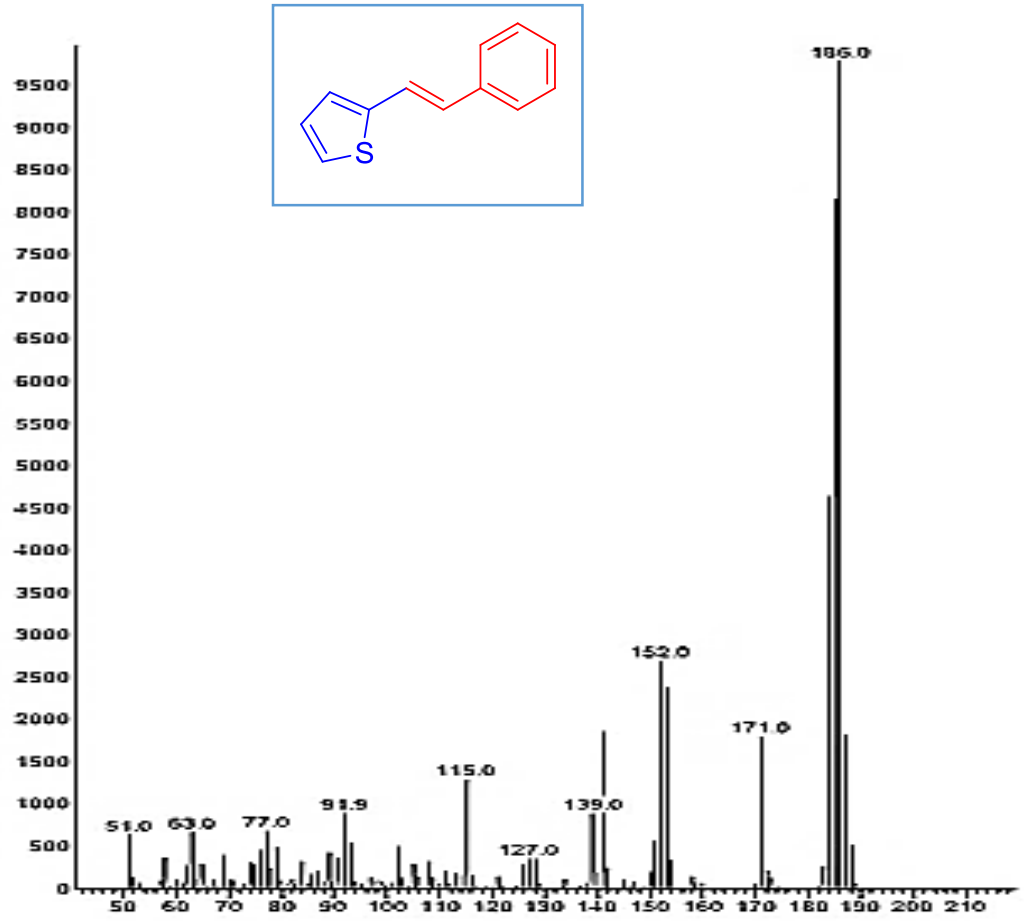


S63. ^{1}HNMR for Ethyl (*E*)-3-styrylbenzoate

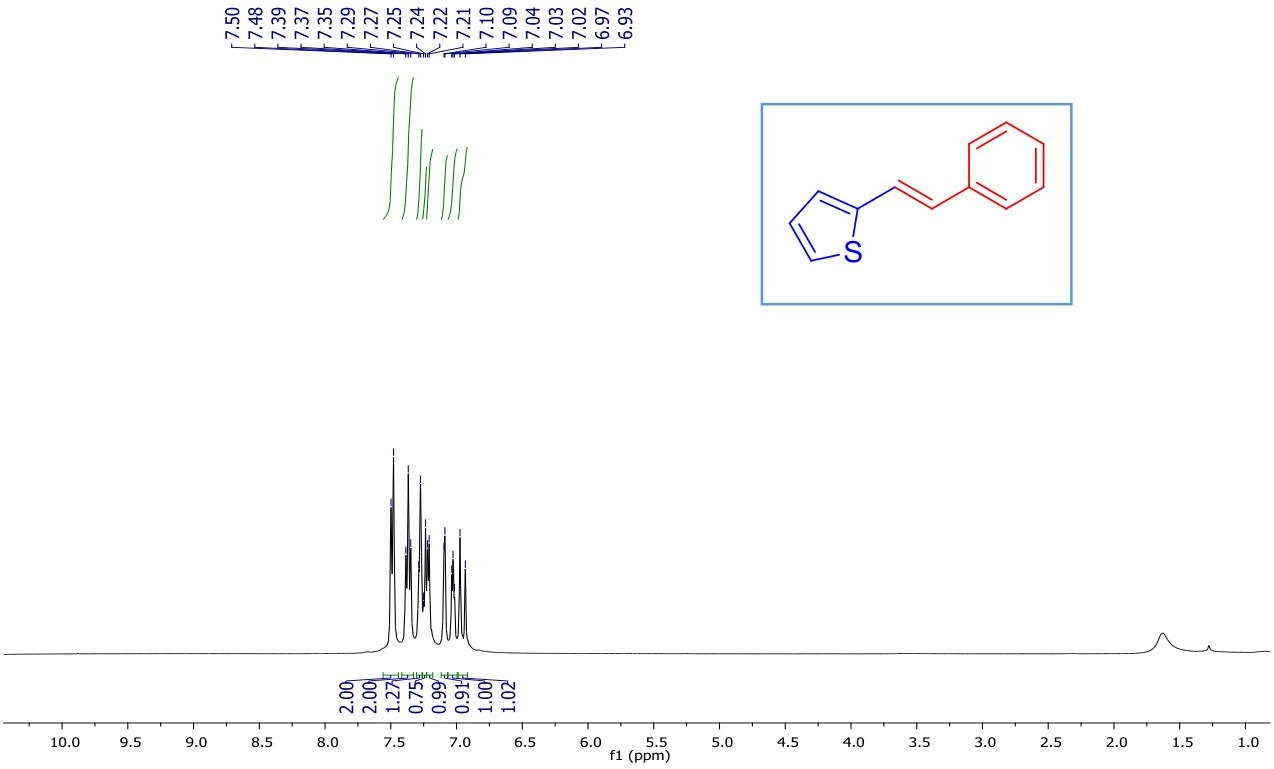


S64. ^{13}CNMR for Ethyl (*E*)-3-styrylbenzoate

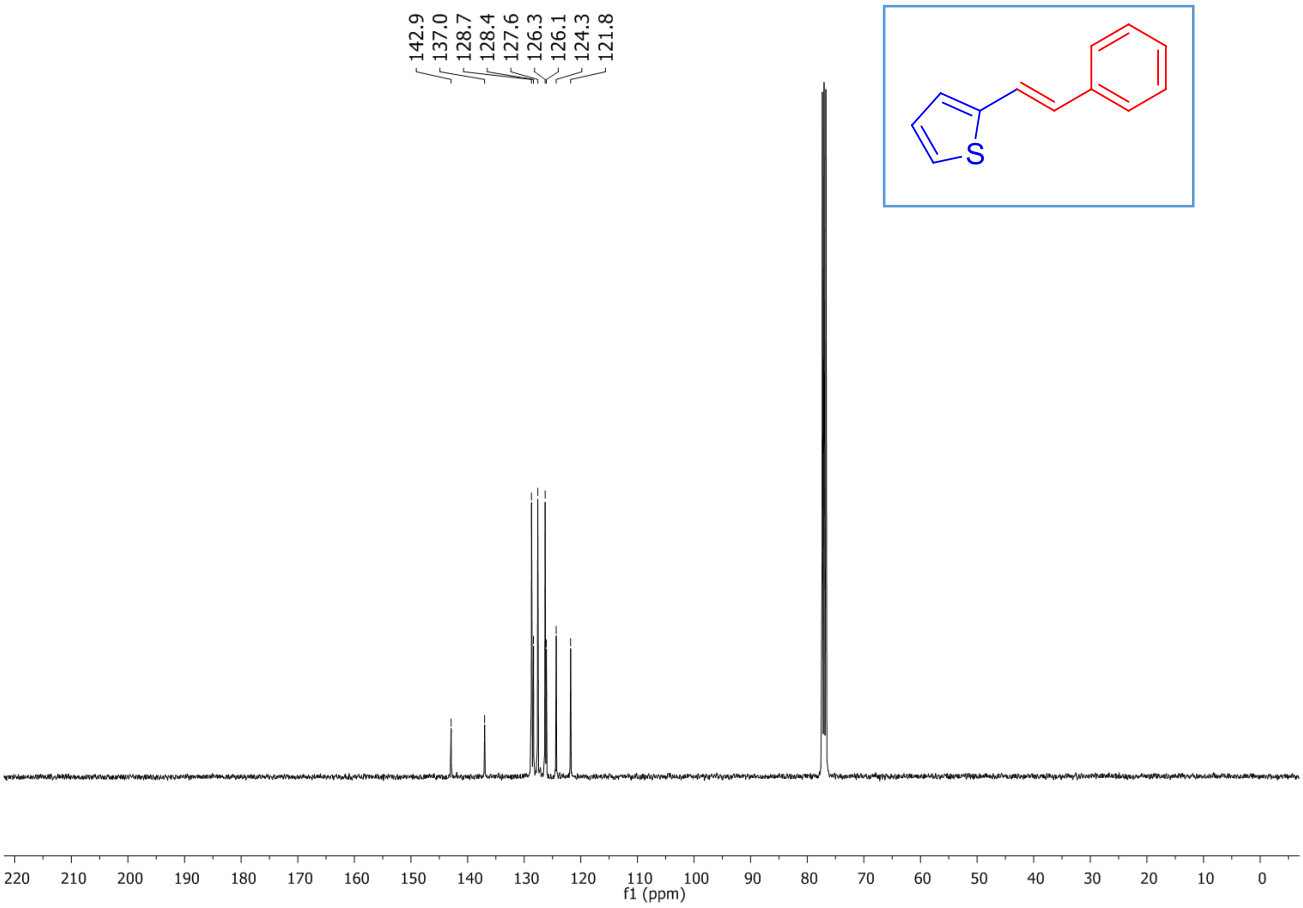
(*E*)-2-styrylthiophene



S65. GC-Mass of (*E*)-2-styrylthiophene



S66. ^{1}HNMR Compound of (*E*)-2-styrylthiophene

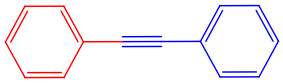


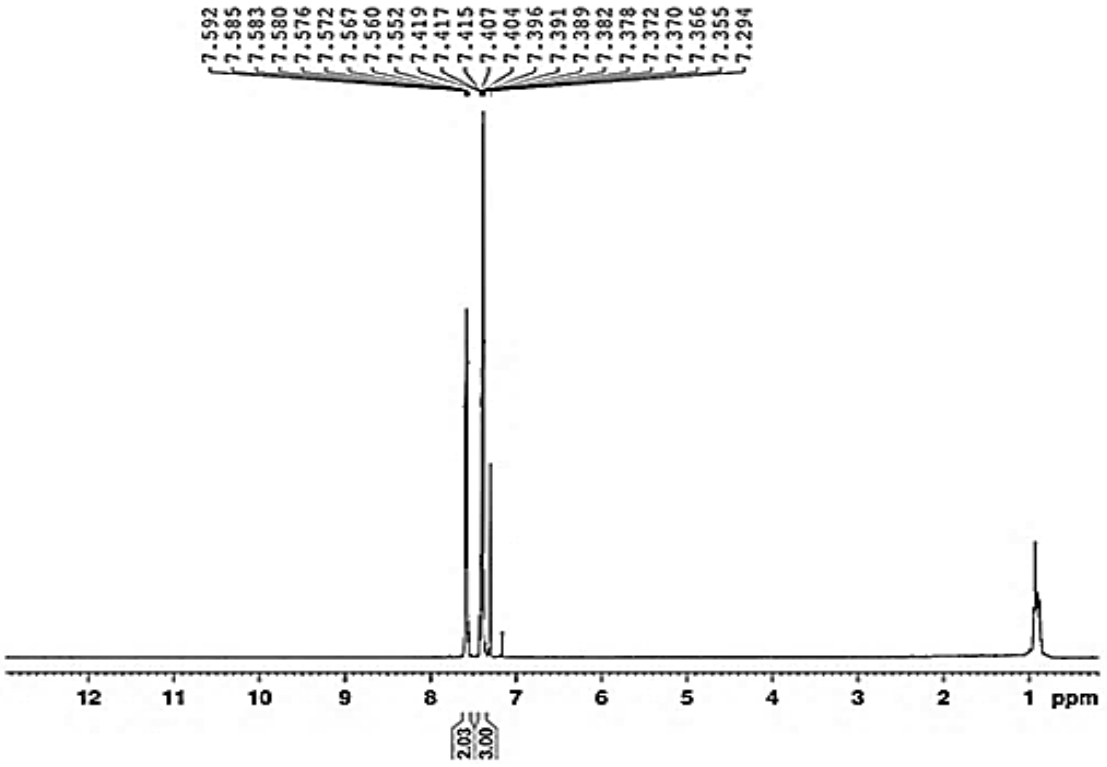
S67. ^{13}CNMR Compound of (*E*)-2-styrylthiophene

Sonogashira derivatives

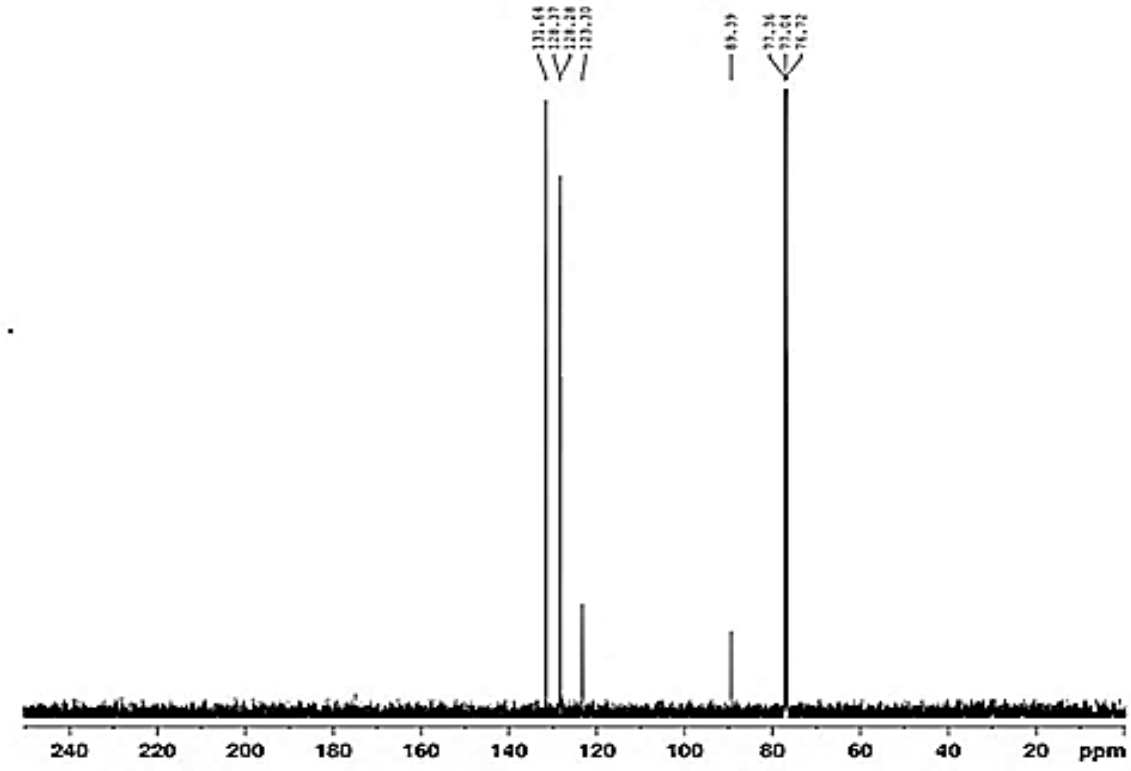
1. 1,2-diphenylethyne

Melting Point (M.P) = 60-62°C





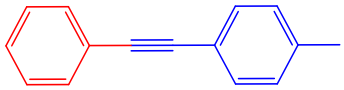
S68. ^{1}HNMR Compound of 1,2-Diphenylethyne

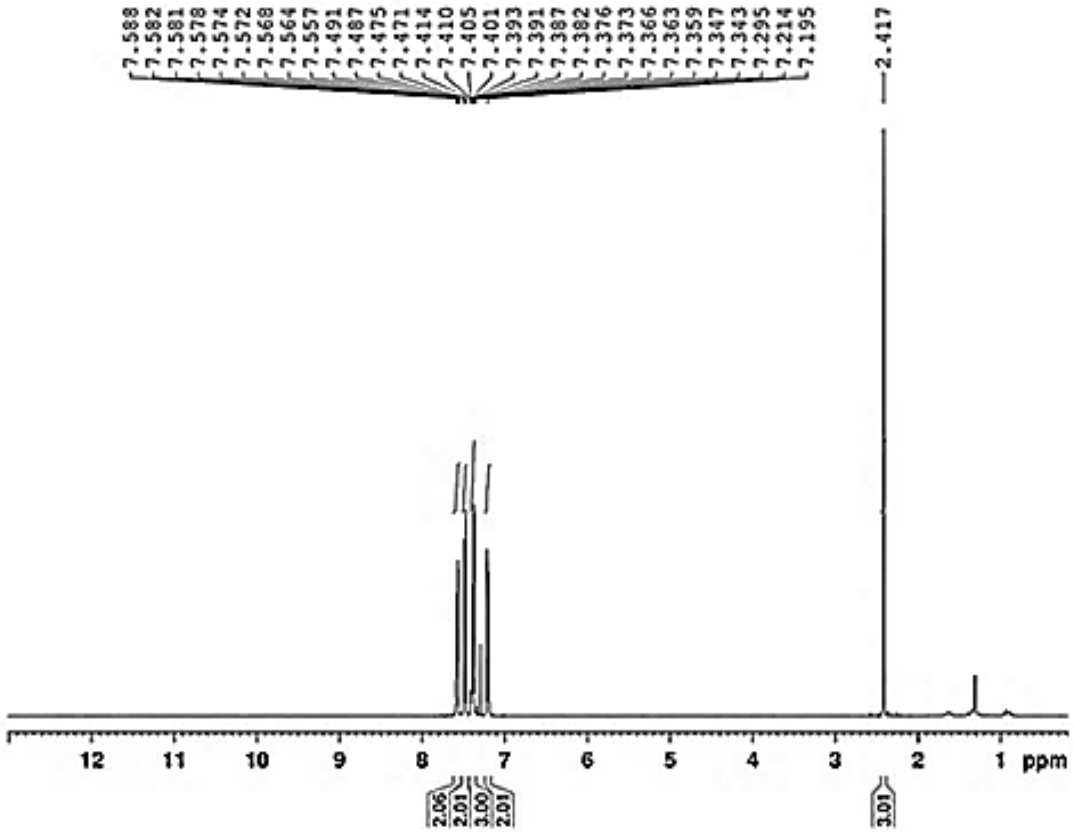


S69.^{13}CNMR Compound of 1,2-Diphenylethyne

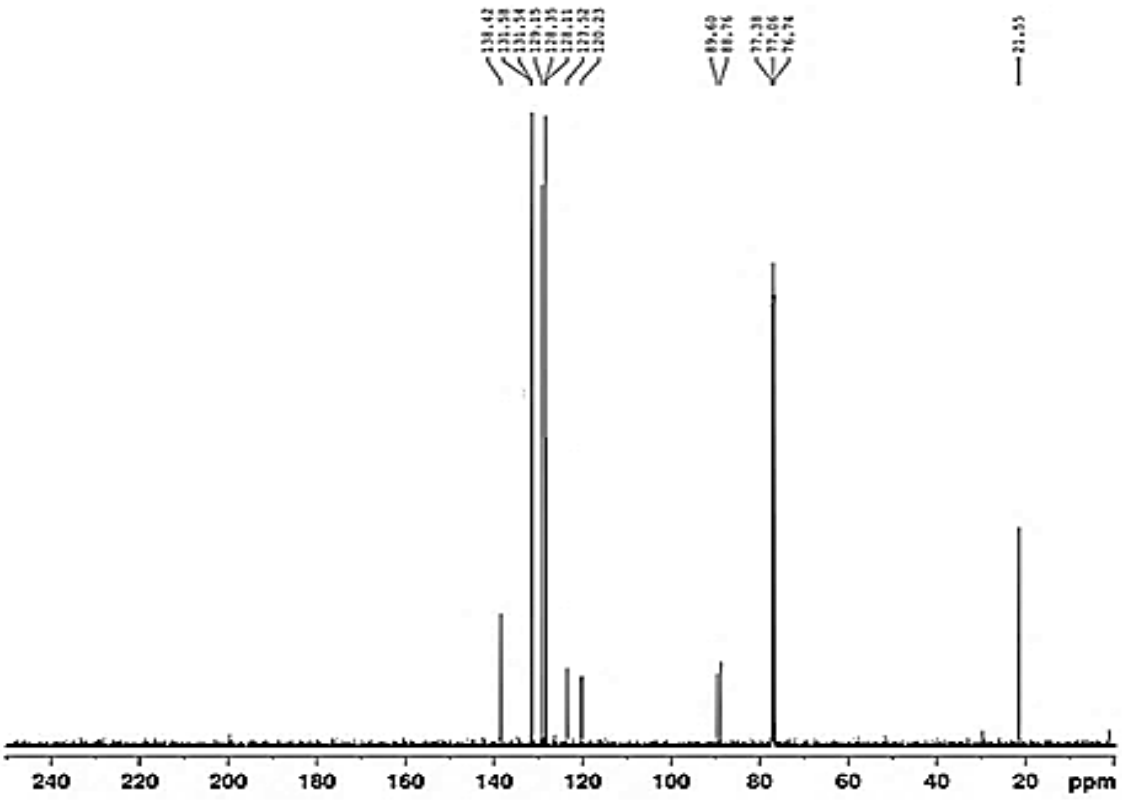
2. 1-methyl-4-(phenylethynyl)benzene

Melting Point (M.P) = 70-71°C





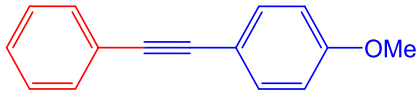
S70. ^{1}HNMR Compound of 1-methyl-4-(phenylethynyl) benzene

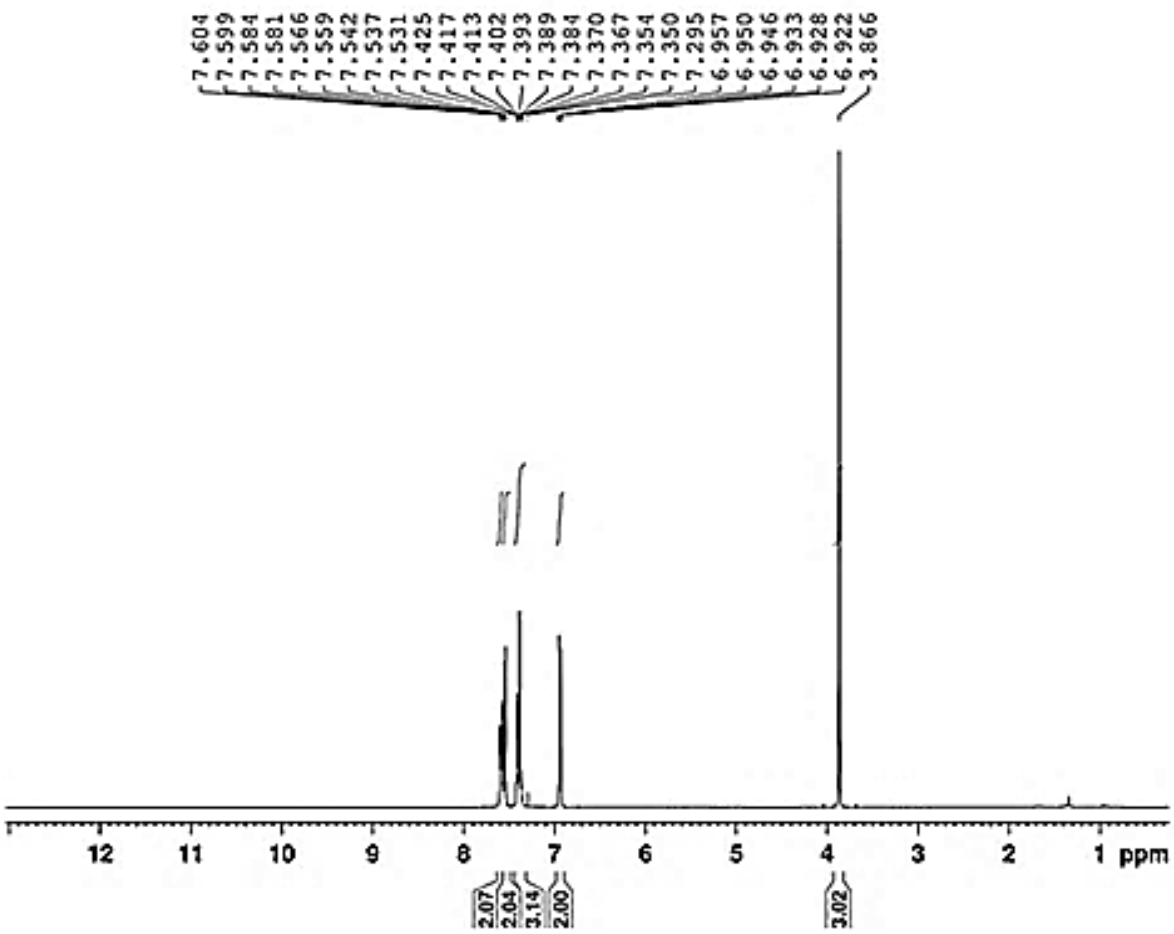


S71. ^{13}CNMR Compound of 1-methyl-4-(phenylethynyl) benzene

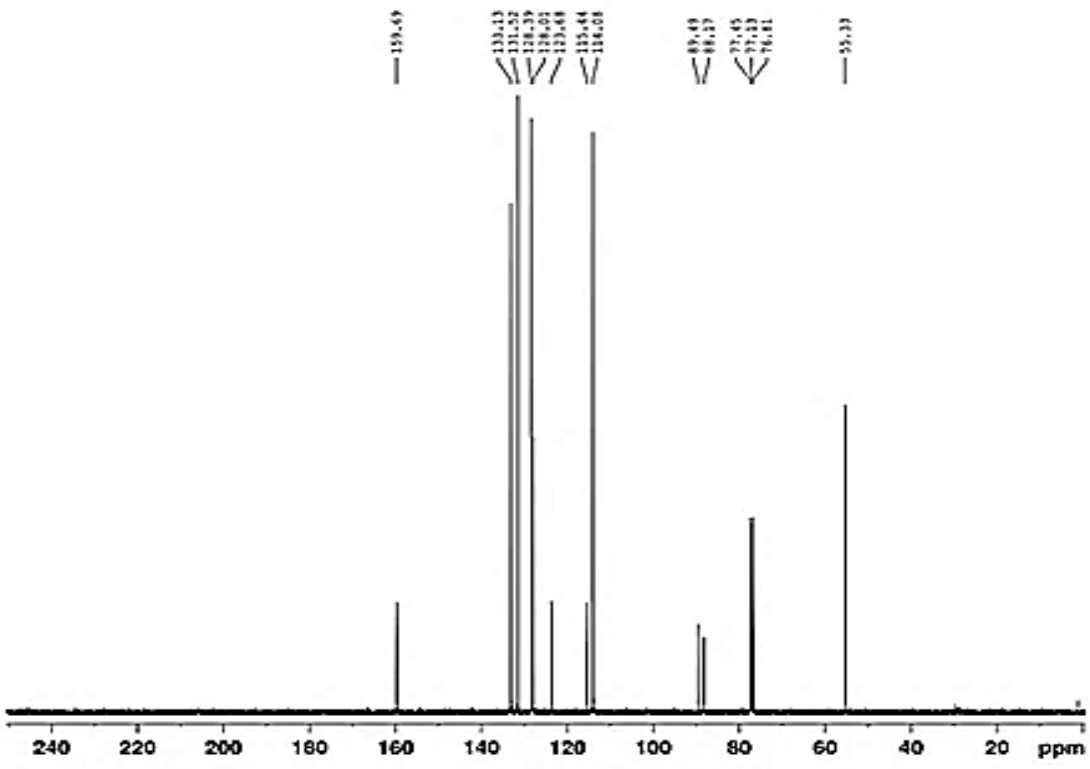
3. 1-methoxyl-4-(phenylethynyl)benzene

Melting Point (M.P) = 61-63°C

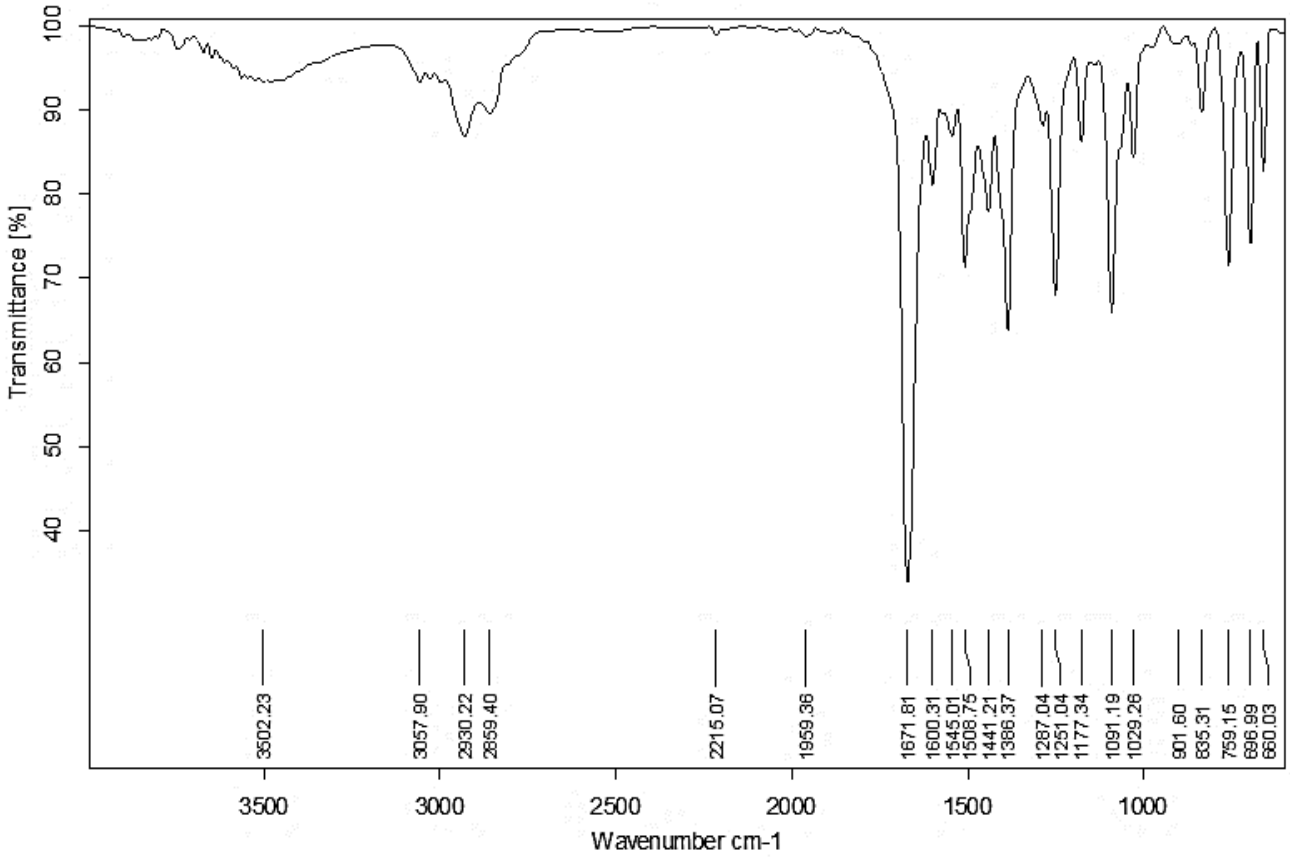




S72. ^{1}HNMR Compound of 1-methoxy-4-(phenylethynyl) benzene



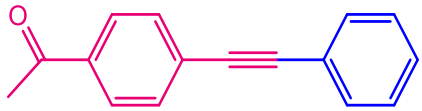
S73. ^{13}CNMR Compound of 1-methoxy-4-(phenylethynyl) benzene

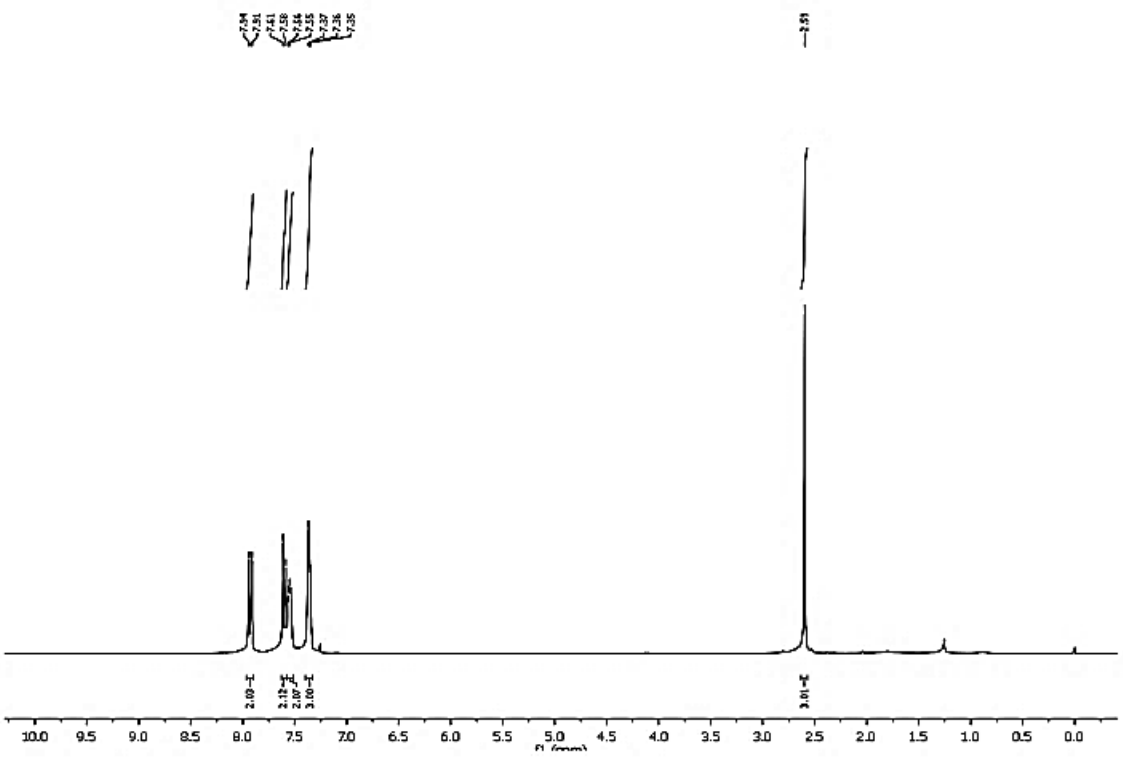


S74. FT-IR Compound of 1-methoxy-4-(phenylethynyl) benzene

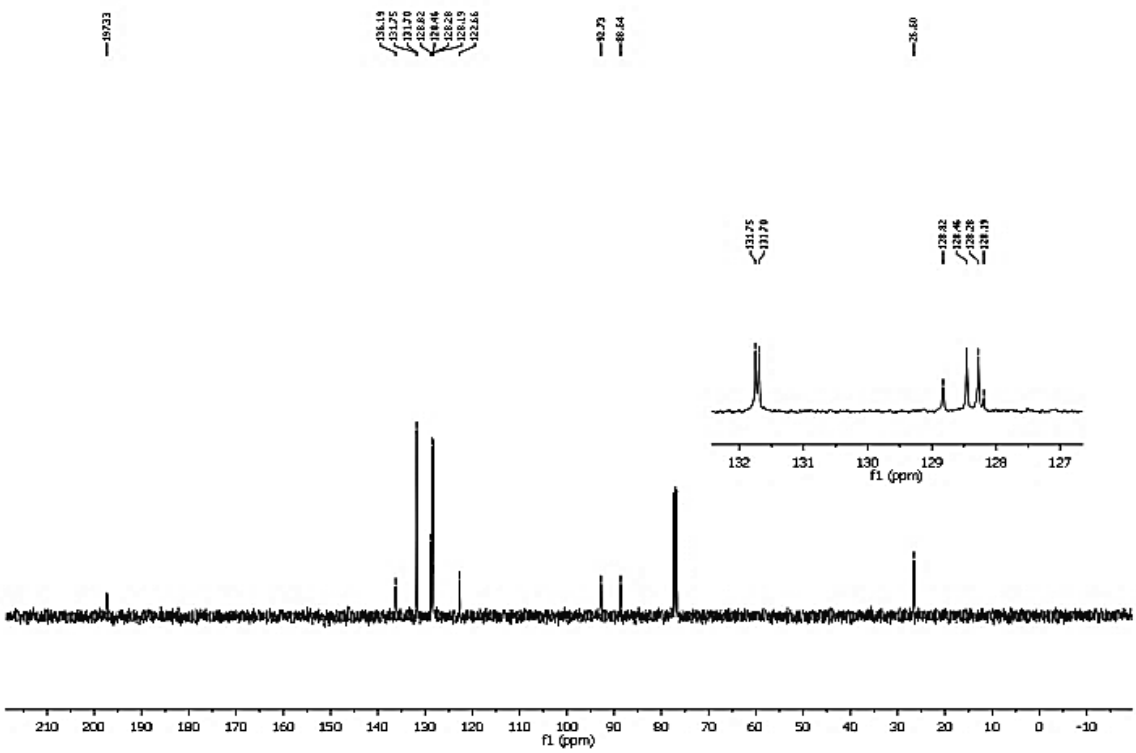
4. 1-(4-(phenylethynyl) phenyl) ethan-1-one

Melting Point (M.P) = 99-101°C





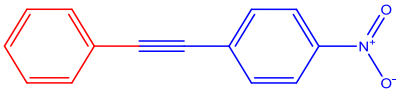
S75. ^{1}HNMR Compound of 1-(4-(phenylethynyl) phenyl) ethan-1-one

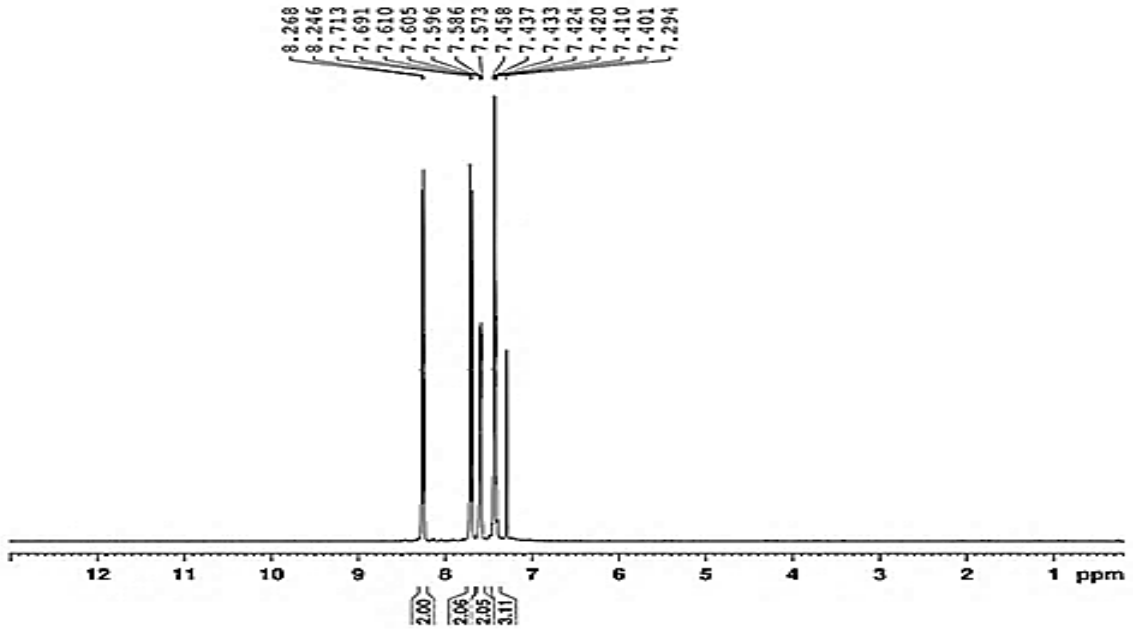


S76. ^{13}CNMR Compound of 1-(4-(phenylethynyl) phenyl) ethan-1-one

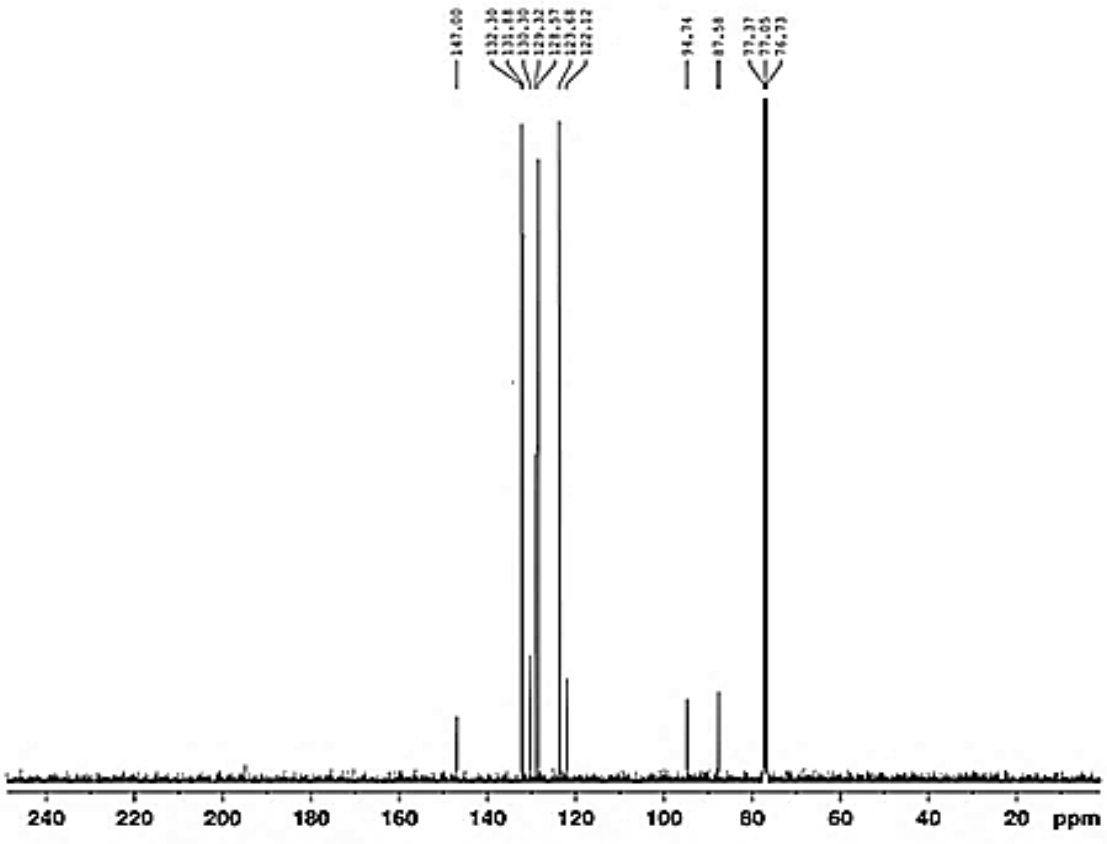
5. 1-nitro-4-(phenylethynyl) benzene

Melting Point (M.P) = 112-114°C

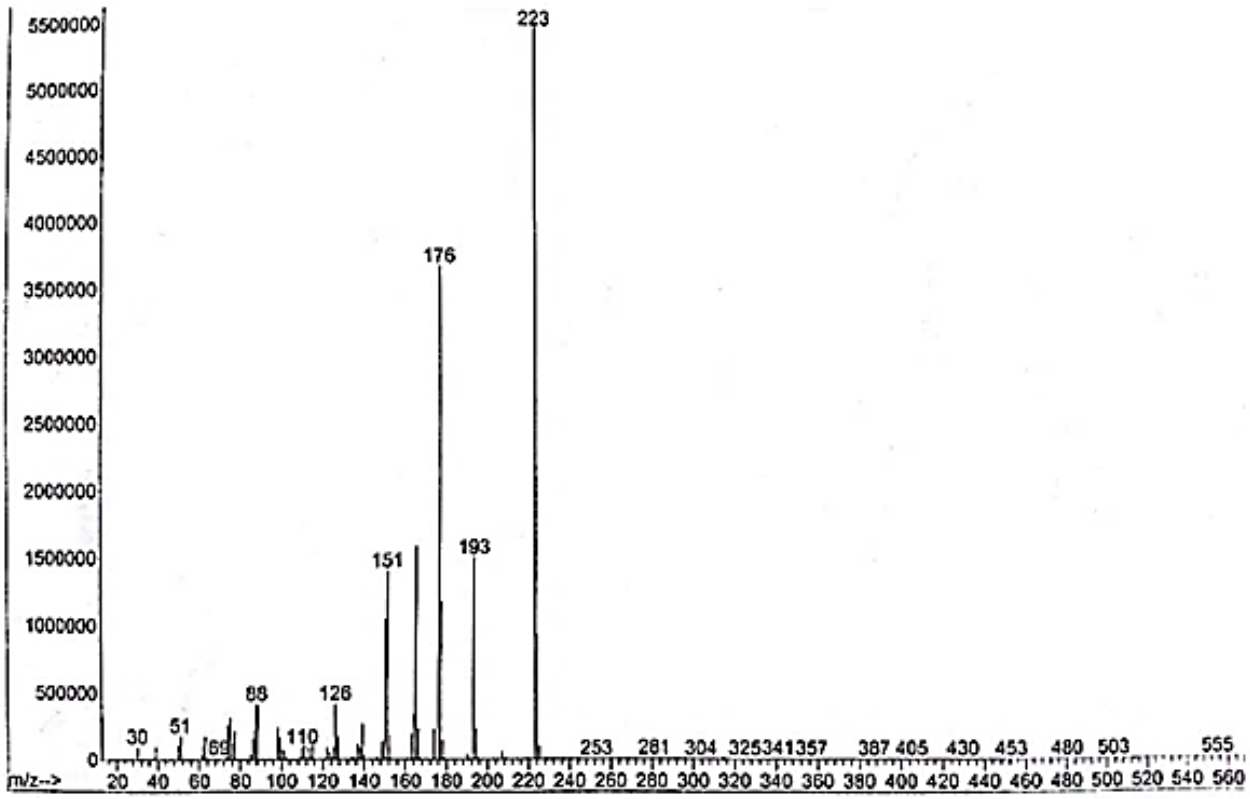




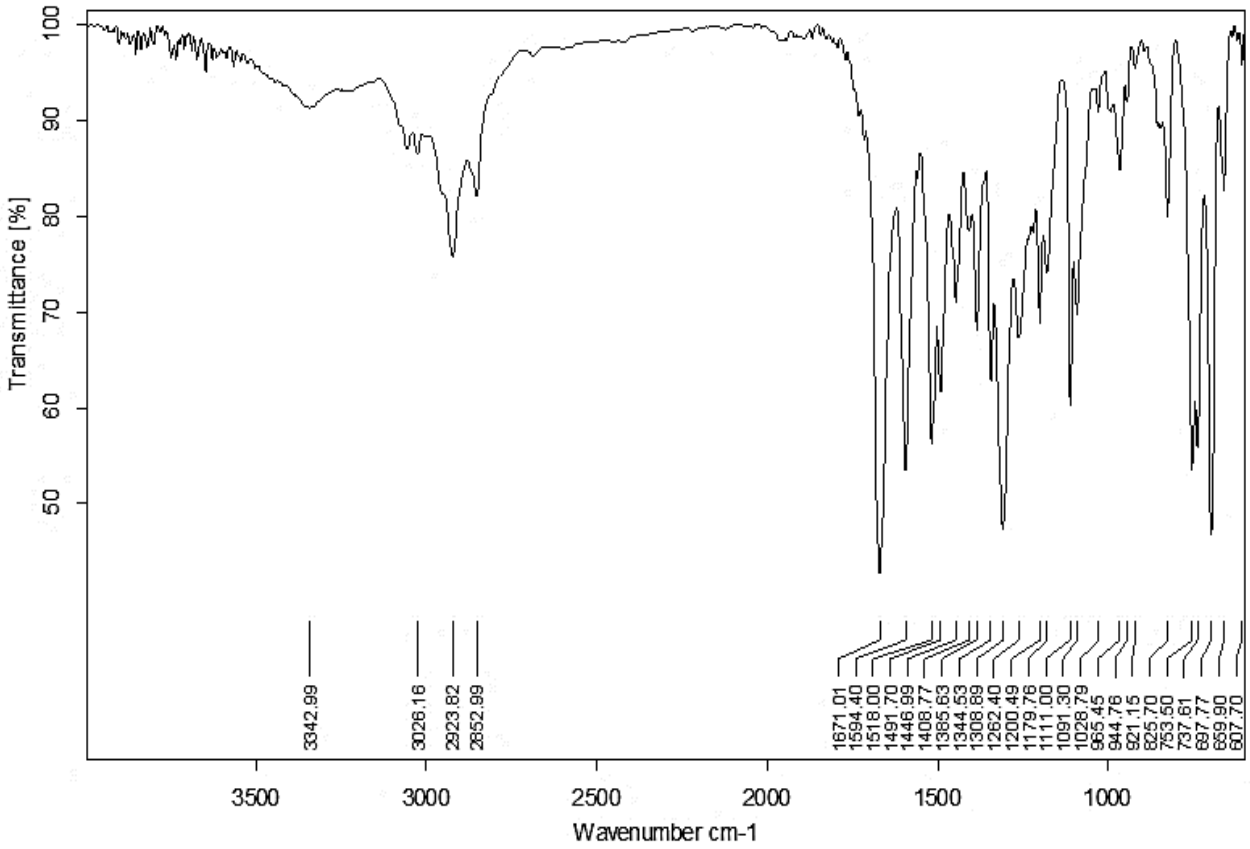
S77. ^{1}HNMR Compound of 1-nitro-4-(phenylethynyl) benzene



S78. ^{13}CNMR Compound of 1-nitro-4-(phenylethynyl) benzene

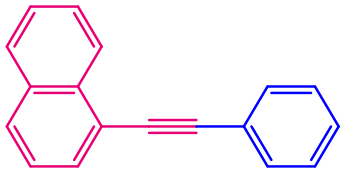


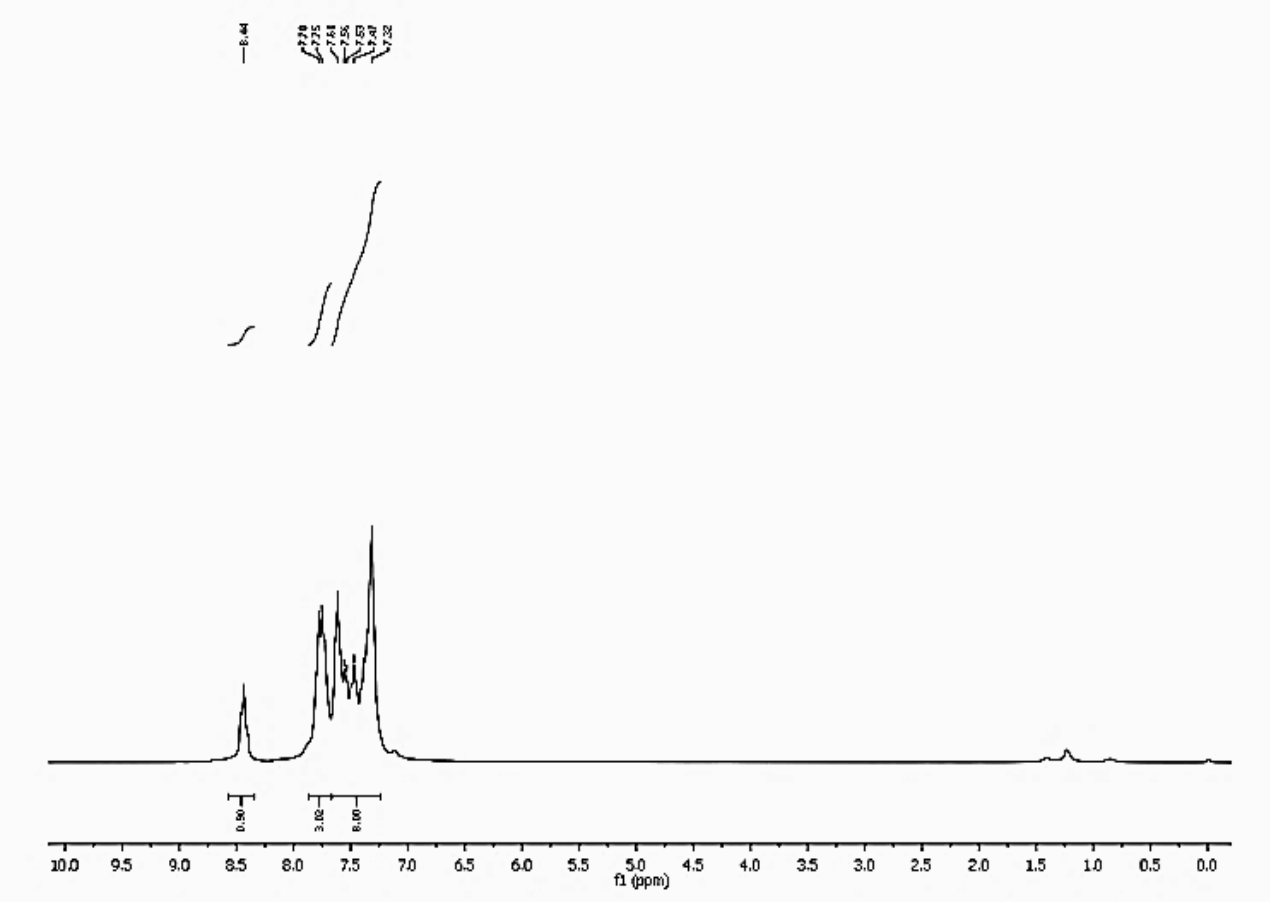
S79. GC-Mass Compound of 1-nitro-4-(phenylethynyl)benzene



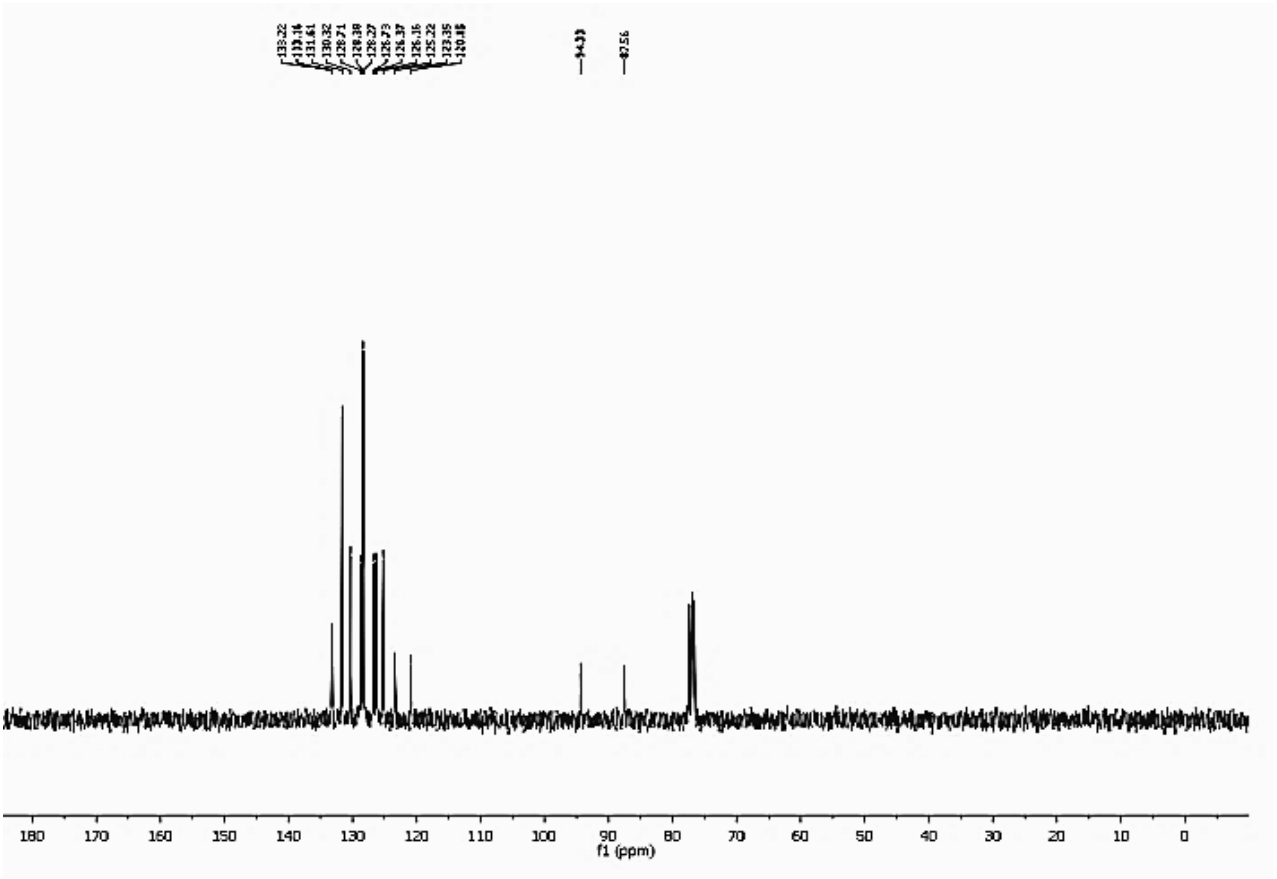
S80. FT-IR Compound of 1-nitro-4-(phenylethynyl)benzene

6. 1-(phenylethynyl)naphthalene





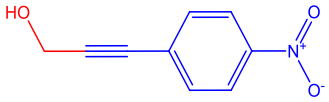
S81. ^{1}HNMR Compound of 1-(phenylethynyl)naphthalene

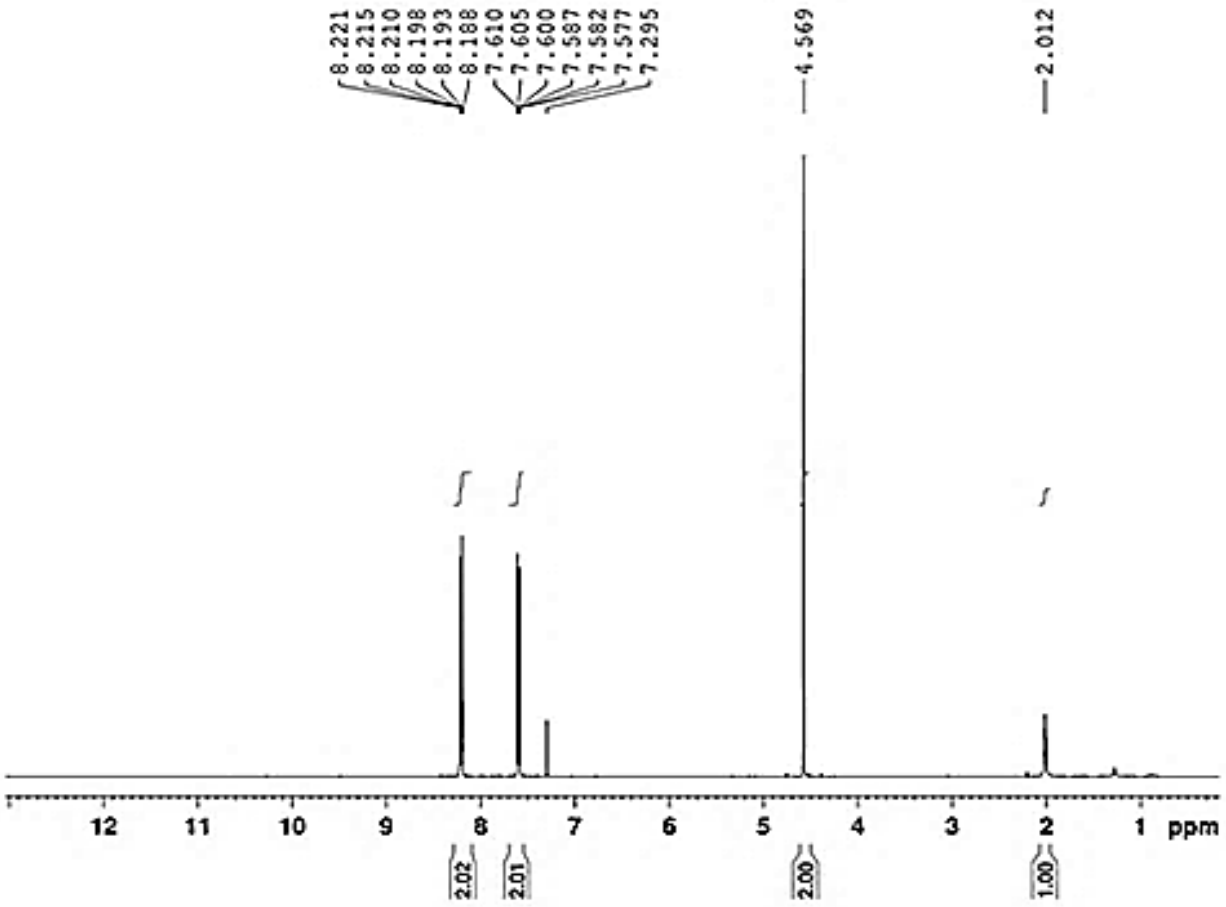


S82. ^{13}CNMR Compound of 1-(phenylethynyl) naphthalene

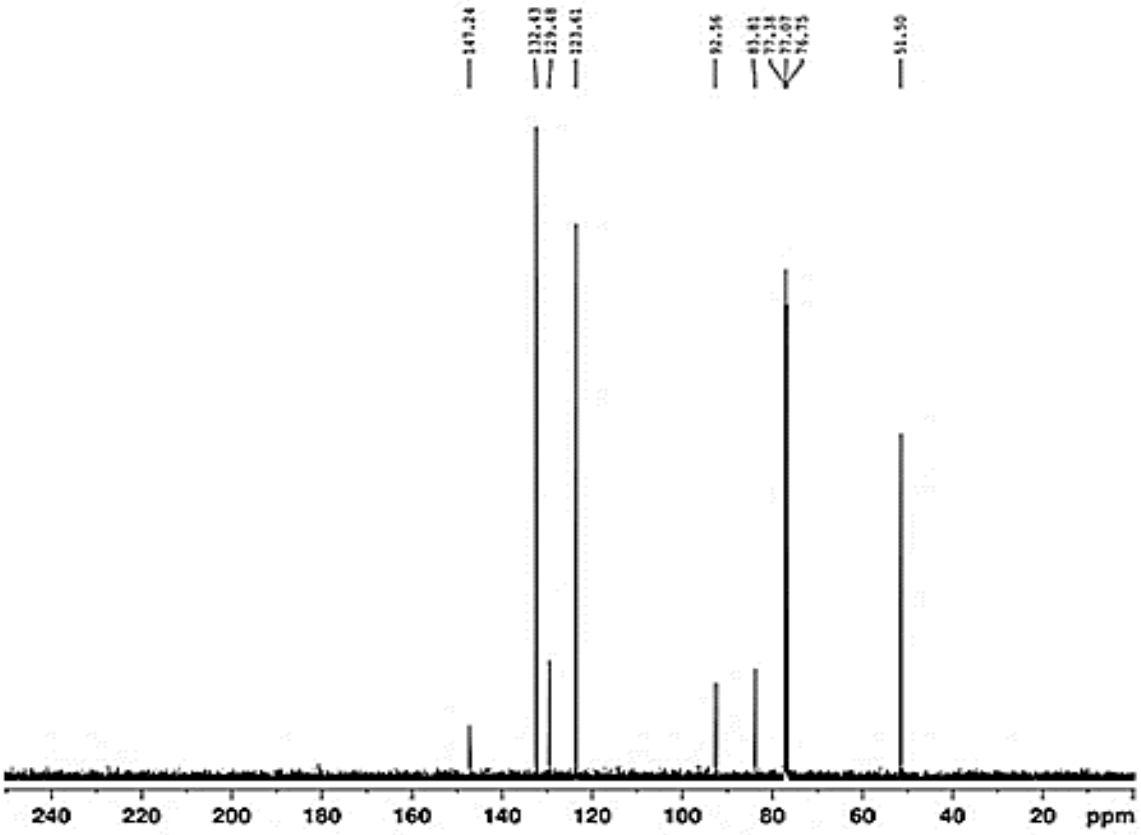
7. 3-(4-nitrophenyl) prop-2-yn-1-ol

Melting Point (M.P) = 94-96°C





S83. ^{1}HNMR Compound of 3-(4-nitrophenyl) prop-2-yn-1-ol



S84. ^{13}CNMR Compound of 3-(4-nitrophenyl) prop-2-yn-1-ol

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