Supporting Information

Well-Defined (*N*-Heterocyclic Carbene)–Ag(I) Complexes as Catalysts for A^3 Reactions

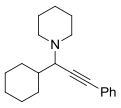
Ming-Tsz Chen, Brant Landers and Oscar Navarro*
Department of Chemistry
University of Hawaii at Manoa
2545 McCarthy Mall
Honolulu, HI 96825-2275
oscarnf@hawaii.edu

Table of Contents

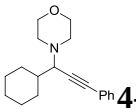
General Information

All aldehydes, amines and phenylacetylene were used as received (Aldrich, Acros, Strem). Solvents were used directly to carry out catalytic reactions. All reactions were carried out under an atmosphere of nitrogen in screw cap vials. Flash chromatography was performed on silica gel 60 (230-400 mesh) (Natland International Corporation) using mixtures hexanes/ethyl acetate (10/1) or petroleum ether/ethyl acetate (10/1), unless otherwise noted. ^{1}H and ^{13}C NMR spectra were recorded on a Varian-300 or Varian-500 MHz spectrometer at ambient temperature in $CDCl_3$ (Cambridge Isotope Laboratories, Inc). Low resolution masses were found using an Aglient 5975E GC-MS (EIMS) instrument. All Infrared spectroscopy were recorded using a Shimadzu IRAffinity-1 spectrometer.

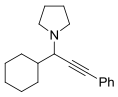
Product Characterization



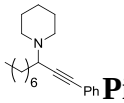
1-(1-Cyclohexyl-3-phenyl-2-propynyl)piperidine (**Scheme 2, 7**).[1] The procedure afforded, after flash **chromatography** (petroleum ether/ethyl acetate 10/1), 259 mg (92%) of the title compound (yellow liquid). ^{1}H NMR ($CDCl_3$, 300 MHz) δ 0.89-1.11 (m, 2H), 1.15-1.35 (m, 3H), 1.45-1.50 (m, 2H), 1.57-1.72 (m, 6H), 1.78-1.82 (m, 2H), 2.05-2.17 (m, 2H), 2.40-2.46 (m, 2H), 2.63-2.70 (m, 2H), 3.13 (d, J = 9.9 Hz, 1H), 7.29-7.35 (m, 3H), 7.45-7.48 (m, 2H). $^{13}C\{^{1}H\}$ NMR ($CDCl_3$, 75 MHz) δ 26.7, 26.1, 26.3, 26.8, 30.4, 31.3, 39.6, 64.3, 86.1, 87.7, 123.8, 127.5, 128.1, 131.7. EIMS m/z 281 (M+).



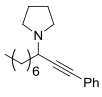
4-(1-cyclohexyl-3-phenylprop-2-yn-1-yl)morpholine (**Scheme 2, 8**). [2] The procedure afforded, after flash chromatography (petroleum ether/ethyl acetate 10/1), 255 mg (90%) of the title compound (yellow liquid). ^{1}H NMR ($CDCl_3$, 300 MHz) δ 0.96-1.32 (m, 5H), 1.56-1.81 (m, 4H), 2.10 (t, *J* = 15.8 Hz, 2H), 2.48-2.55 (m, 2H), 2.68-2.74 (m, 2H), 3.14 (d, *J* = 9.9 Hz, 1H), 3.69-3.81 (m, 4H), 7.28-7.30 (m, Ar*H*, 3H), 7.43-7.46 (m, Ar*H*, 2H).^{13}C{^{1}H} NMR ($CDCl_3$, 75 MHz) δ 25.9, 26.1, 26.6, 30.2, 30.9, 38.9, 49.8, 63.8, 67.1, 86.5 86.7, 123.3, 127.7, 128.1, 131.6. EIMS m/z 283 (M+).



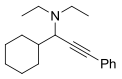
1-[1-Cyclohexyl-3-(4-methylphenyl)-2-propynyl]pyrrodine (**Scheme 2, 9**).[1] The procedure afforded, after flash chromatography (petroleum ether/ethyl acetate 10/1), 254 mg (95%) of the title compound (colorless liquid). ^{1}H NMR ($CDCl_3$, 300 MHz) δ 1.04- 1.37 (m, 5H), 1.54-1.65(m, 1H), 1.66-1.72(m, 1H), 1.73-1.86(m, 4H), 1.96-2.09(m, 2H), 2.10-2.14(m, 2H), 2.61-2.70(m, 2H), 2.72-2.82(m, 2H), 3.36(d, *J* = 8.4Hz, 1H), 7.28-7.32(m, Ar*H*, 3H), 7.43-7.47(m, Ar*H*, 2H). ^{13}C{^{1}H} NMR ($CDCl_3$, 75 MHz) δ 23.5, 26.2, 26.6, 30.3, 30.6, 41.3, 49.9, 61.1, 85.7, 87.9, 123.6, 127.6, 128.1, 131.6. EIMS m/z 267 (M+).



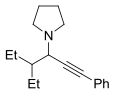
Piperidine, 1-[1-(2-phenylethynyl)octyl] (Scheme 2, 10).[3] The procedure afforded, after flash chromatography (petroleum ether/ethyl acetate 7/3), 268 mg (90%) of the title compound (colorless liquid). ^{1}H NMR ($CDCl_3$, 300 MHz) δ 0.88-0.92 (t, 3H), 1.30-1.32 (m, 10H), 1.46-1.48 (m, 6H), 1.56-1.78 (m, 2H), 2.45-2.50 (m, 2H), 2.69-2.78 (m, 2H), 3.49 (t, *J* = 7.2Hz, 1H), 7.25-7.32 (m, Ar*H*, 3H), 7.43-7.48 (m, Ar*H*, 2H). ^{13}C{^{1}H} NMR ($CDCl_3$, 75 MHz) δ 14.0, 22.5, 24.4, 26.0, 26.8, 29.1, 29.2, 31.7, 33.3, 50.4, 58.4, 85.5, 87.9, 123.4, 127.5, 128.0, 131.5. EIMS m/z 297 (M+).



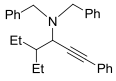
Pyrrolidine, 1-[1-(2-phenylethynyl)octyl]. (**Scheme 2, 11**). The procedure afforded, after flash chromatography (petroleum ether/ethyl acetate 8/2), 269 mg (95%) of the title compound (yellow liquid). ^{1}H NMR ($CDCl_3$, 300 MHz) δ 0.85-0.87 (m, 3H), 1.29 (d, *J* = 7.2Hz, 8H), 1.47 – 1.61 (m, 2H), 1.68–1.79 (m, 6H), 2.68-2.78 (m, 4H), 3.68 (t, *J* = 7.2Hz, 1H), 7.25-7.27 (m, Ar*H*, 3H), 7.40-7.43 (m, Ar*H*, 2H). ^{13}C{^{1}H} NMR ($CDCl_3$, 75 MHz) δ 14.0, 22.5, 23.3, 26.6, 29.1, 29.3, 31.7, 34.9, 85.2, 88.0, 123.3, 127.6, 128.0, 131.5. EIMS m/z 283 (M+).



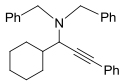
1-cyclohexyl-N,N-diethyl-3-phenylprop-2-yn-1-amine (**Scheme 2, 12**).[4] The procedure afforded, after flash chromatography (petroleum ether/ethyl acetate 8/2), 234 mg (87%) of the title compound (colorless liquid). ^{1}H NMR ($CDCl_3$, 300 MHz) δ 1.09-1.14 (m, 7H), 1.21-1.37 (m, 4H), 1.61-1.64 (m, 1H), 1.68-1.85 (m, 8H), 2.16-2.20 (m, 2H), 2.43-2.54 (m, 2H), 2.66-2.75 (m, 2H), 3.34-3.37 (d, 1H, *J* =9.9Hz), 7.29-7.32 (m, Ar*H*, 3H), 7.45-7.48 (m, Ar*H*, 2H). ^{13}C{^{1}H} NMR ($CDCl_3$, 75 MHz) δ 13.7, 26.0, 26.2, 26.7, 30.6, 31.3, 40.2, 44.7, 59.2, 85.2, 88.3, 123.8, 127.5, 128.1, 131.6. EIMS m/z 269 (M+).



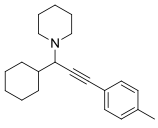
Pyrrolidine, 1-[2-ethyl-1-(2-phenylethynyl)butyl] (**Scheme 2, 13**).[5] The procedure afforded, after flash chromatography (petroleum ether/ethyl acetate 8/2 then ethyl acetate), 243 mg (95%) of the title compound (yellow liquid). ^{1}H NMR ($CDCl_3$, 300 MHz) δ 0.93-1.00 (m, 6H), 1.47-1.70 (m, 4H), 1.81-1.87 (m, 5H), 2.69-2.79 (m, 4H), 3.55 (d, *J* = 6.9 Hz, 1H), 7.29-7.34 (m, Ar*H*, 3H), 7.45-7.48 (m, Ar*H*, 2H). ^{13}C{^{1}H} NMR ($CDCl_3$, 75 MHz) δ 10.7, 10.8, 21.7, 22.2, 23.4, 43.6, 50.1, 58.2, 85.4, 87.8, 123.6, 127.5, 128.0, 131.5. EIMS m/z 255 (M+).



N,N-Dibenzyl-4-ethyl-1-phenyl-1-hexyn-3-amine (**Scheme 2, 14**).[6] The procedure afforded, after flash chromatography (petroleum ether/DCM 1/1), 351 mg (92%) of the title compound (white solid). ^{1}H NMR ($CDCl_3$, 300 MHz) δ 0.80 (t, J = 7.4 Hz, 3H), 1.02 (t, J = 7.2 Hz, 3H), 1.51–161(m, 2 H), 1.65-1.74 (m, 2H), 1.87-1.99 (m, 3H), 3.60 (d, J = 9.9 Hz, 1H), 3.66 (d, J = 13.5 Hz, 2H), 4.08 (d, J =13.8 Hz, 2H), 7.39-7.43 (m, Ar*H*, 2H), 7.47-7.52 (m, Ar*H*, 7H), 7.62 (d, J = 7.5Hz, Ar*H*, 4H), 7.71-7.74 (m, Ar*H*, 2H). ^{13}C{^{1}H} NMR ($CDCl_3$, 75 MHz) δ 8.8, 10.6, 20.0, 22.2, 41.6, 55.2, 86.1, 87.5, 123.7, 126.9, 127.8, 128.2, 128.3, 129.1, 131.8, 139.6. EIMS m/z 381 (M+).

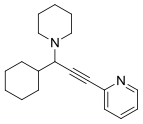


N,N-dibenzyl-1-cyclohexyl-3-phenylprop-2-yn-1-amine (**Scheme 2, 15**).[7] The procedure afforded, after flash chromatography (petroleum ether/DCM 1/1), 362 mg (92%) of the title compound (white solid). ^{1}H NMR ($CDCl_3$, 300 MHz) δ 0.87-1.04(m, 2H), 1.17-1.34(m, 4H), 1.71-1.82(m, 5H), 2.17-2.21(m, 1H), 2.43-2.48 (m, 1H), 3.36-3.91(m, 1H), 3.57-3.62(m, 2H), 3.98-4.02(m, 2H), 7.33-7.35(m, Ar*H*, 2H), 7.40-7.41(m, Ar*H*, 7H), 7.53-7.56(m, Ar*H*,4H), 7.61-7.64(m, Ar*H*, 2H). ^{13}C{^{1}H} NMR ($CDCl_3$, 75 MHz) δ 25.9, 26.1, 26.5, 30.3, 31.3, 39.7, 55.0, 58.3, 77.2, 86.2, 87.1, 109.6, 123.6, 126.8, 127.8, 128.2, 128.8, 131.8, 132.0, 139.7. EIMS m/z 393 (M+).



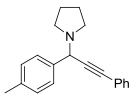
1-[1-Cyclohexyl-3-(4-methylphenyl)-2-propynyl]piperidine. (**Scheme 2, 16**).[1] The procedure afforded, after flash chromatography (petroleum ether/ethyl acetate 10/1), 269 mg (91%) of the title compound (yellow liquid). ^{1}H NMR ($CDCl_3$, 300 MHz) δ 0.91-1.07 (m, 2H), 1.14-1.35 (m, 3H), 1.36-1.46 (m, 2H), 1.47-1.68 (m, 6H), 1.69-1.81 (m, 2H), 1.98-2.12 (m, 2H), 2.34 (s, CH_3, 3H), 2.36-2.43 (m, 2H), 2.54-2.68 (m, 2H), 3.09 (d, J = 9.9Hz, 1H), 7.09 (d, J = 7.8Hz, Ar*H*, 2H), 7.33 (d, J =

8.1Hz, Ar*H*, 2H). $^{13}C\{^{1}H\}$ NMR ($CDCl_3$, 75 MHz) δ 21.4, 24.7, 26.1, 26.3, 26.8, 30.4, 31.3, 39.6, 46.9, 50.7, 64.4, 86.1, 86.9, 120.7, 128.9, 131.5, 137.5. EIMS m/z 295 (M+).



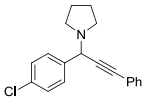
1-[1-Cyclohexyl-3-(2-pyridinyl)-2-propynyl]piperidine. (**Scheme 2, 17**).[8] The procedure afforded, after flash chromatography (petroleum ether/ethyl acetate 8/2), 257 mg (91%) of the title compound (yellow solid). ^{1}H NMR ($CDCl_3$, 300 MHz) δ0.91–1.80 (m, 15H), 2.02–2.15 (m, 2H), 2.42 (br, 2H), 2.65 (br, 2H), 3.15(d, *J* = 9.6 Hz, 1H), 7.20 (t, *J* = 12Hz, 1H), 7.43 (d, *J* = 7.8 Hz, 1H), 7.60 (t, *J* = 7.7 Hz, 1H), 8.57 (d, *J* = 5.1 Hz, 1H). $^{13}C\{^{1}H\}$ NMR ($CDCl_3$, 75 MHz) δ 24.4, 25.4, 26.0, 26.6, 29.0, 30.3, 31.3, 39.3, 50.5, 64.1, 77.2, 122.4, 123.4, 127.3, 127.4, 136.2 149.6, 149.9. EIMS m/z 282 (M+).

N-[1,3-diphenyl-2-propynyl]Pyrrolidine (**Scheme 2, 18**).[1] The procedure afforded, after flash chromatography (petroleum ether/ethyl acetate 3/2), 230 mg (88%) of the title compound (colorless liquid). ^{1}H NMR ($CDCl_3$, 300 MHz) δ 1.81-1.87 (m, 4H), 2.73-2.78 (m, 4H), 4.94 (s, 1H), 7.31-7.37 (m, Ar*H*, 4H), 7.39-7.44 (m, Ar*H*, 2H), 7.52-7.58 (m, Ar*H*, 2H), 7.66-7.69 (m, Ar*H*, 2H). $^{13}C\{^{1}H\}$ NMR ($CDCl_3$, 75 MHz) δ 23.4, 50.2, 59.0, 86.6, 86.8, 123.2, 127.5, 128.0, 128.2, 131.7, 139.5. EIMS m/z 261 (M+).

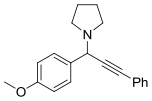


N-[1-(4-tolyl)-3-phenyl-2-propynyl]Pyrrolidine (**Scheme 2, 19**).[9] The procedure afforded, after flash chromatography (petroleum ether/ethyl acetate 7/1), 237 mg (86%) of the title compound (yellow liquid). ^{1}H NMR ($CDCl_3$, 300 MHz) δ 1.83-1.88 (m, 4H), 2.41 (s, CH_3, 3H), 2.73-2.77 (m, 4H), 4.90 (s, 1H), 7.21-7.24 (m, Ar*H*, 2H), 7.33-7.38 (m, Ar*H*, 3H), 7.53-7.57 (m, Ar*H*, 4H). $^{13}C\{^{1}H\}$ NMR ($CDCl_3$, 75 MHz) δ 20.2, 21.6, 23.4, 25.1, 48.4, 50.2, 52.0, 57.9, 59.7, 86.6, 86.9, 87.0, 123.3,

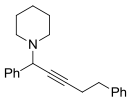
126.8, 127.0, 127.8, 129.0, 129.1, 129.9, 130.6, 132.7, 136.5, 137.1. EIMS m/z 281 (M+). EIMS m/z 275 (M+).



N-[1-(4-Chlorophenyl)-3-phenyl-2-propynyl]Pyrrolidine (**Scheme 2, 20**).[9] The procedure afforded, after flash chromatography (petroleum ether/ethyl acetate 8/2 then ethyl acetate), 243 mg (82%) of the title compound (yellow liquid). ^{1}H NMR ($CDCl_3$, 300 MHz) δ 1.81-1.83 (m, 4H), 2.68 (m, 4H), 4.89 (s, 1H), 7.33-7.38 (m, Ar*H*, 5H), 7.50-7.54 (m, Ar*H*, 2H), 7.57 (d, *J* = 8.4Hz, Ar*H*, 2H). ^{13}C{^{1}H} NMR ($CDCl_3$, 75 MHz) δ 23.8, 50.3, 58.6, 86.2, 87.7, 95.5, 123.2, 128.6, 128.7, 129.9, 132.1, 133.6, 138.2. EIMS m/z 281 (M+). EIMS m/z 295 (M+).



N-[1-(4-methoxyphenyl)-3-phenyl-2-propynyl]Pyrrolidine (**Scheme 2, 21**).[9] The procedure afforded, after flash chromatography (petroleum ether/ethyl acetate 7/1), 256 mg (88%) of the title compound (yellow liquid). ^{1}H NMR ($CDCl_3$, 300 MHz) δ 1.82-1.84 (m, 4H), 2.68-2.73 (m, 4H), 3.82 (s, C*H*$_3$, 3H), 4.89 (s, 1H), 6.91-6.94 (m, Ar*H*, 2H), 7.32-7.35 (m, Ar*H*, 3H), 7.50-7.57 (m, Ar*H*, 4H). ^{13}C{^{1}H} NMR ($CDCl_3$, 75 MHz) δ 21.6, 23.4, 25.1, 48.3, 50.1, 41.9, 52.3, 54.2, 56.1, 57.5, 59.3, 86.6, 87.0, 87.1, 112.4, 114.5, 123.2, 126.9, 127.1, 128.2, 129.0, 129.3, 130.5, 131.7, 132.8, 158.9. EIMS m/z 291 (M+).



Piperidine, 1-(1,5-diphenyl-2-pentyn-1-yl) (**Scheme 2, 22**).[10] The procedure afforded, after flash chromatography (petroleum ether/DCM 1/1 then DCM), 249 mg (82%) of the title compound (yellow liquid). ^{1}H NMR ($CDCl_3$, 300 MHz) δ 1.79 (s, 4H), 2.66 (t, *J* = 7.1Hz, 8H), 2.92 (t, *J* = 6.9Hz, 2H), 4.82 (s, 1H), 7.26-7.35 (m, Ar*H*, 8H), 8.13 (d, *J* = 7.5 Hz, Ar*H*, 1H), 8.68 (br, Ar*H*, 1H). ^{13}C{^{1}H} NMR ($CDCl_3$, 75 MHz) δ 20.8, 23.4, 35.1, 50.0, 58.5, 77.6, 86.9, 126.3, 127.7, 127.9,

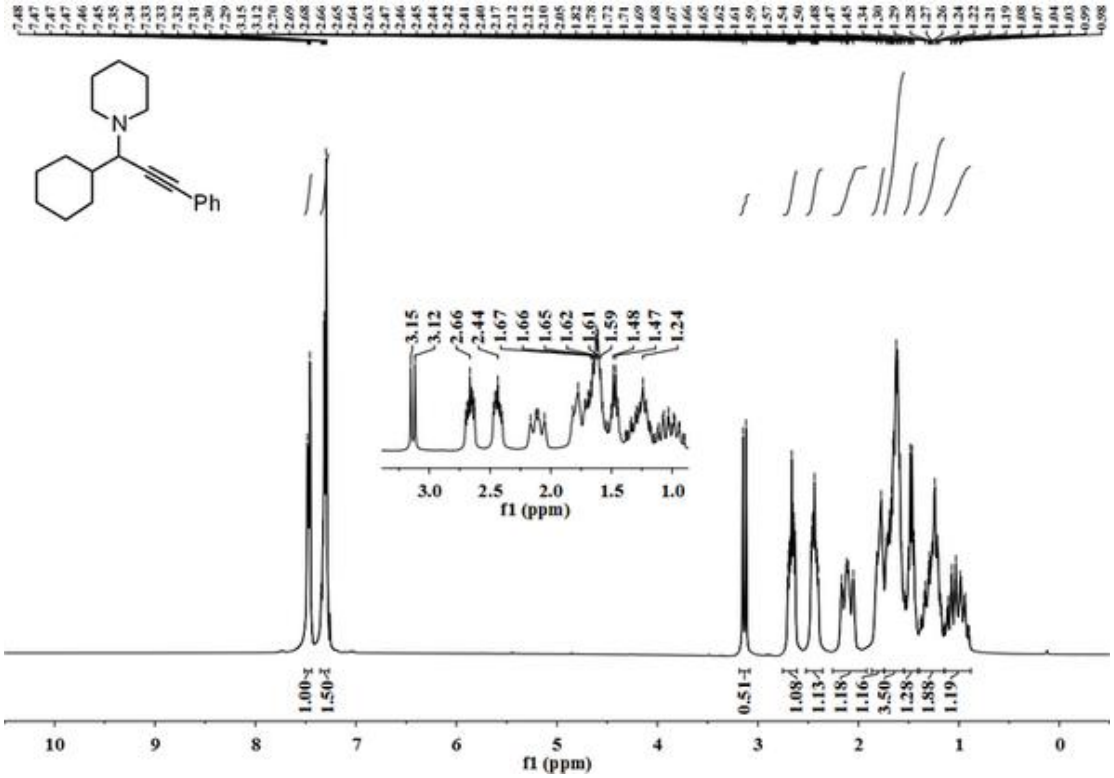
128.2, 128.4, 128.5, 128.6, 129.6, 130.8, 138.7, 140.6. EIMS m/z 303 (M+).

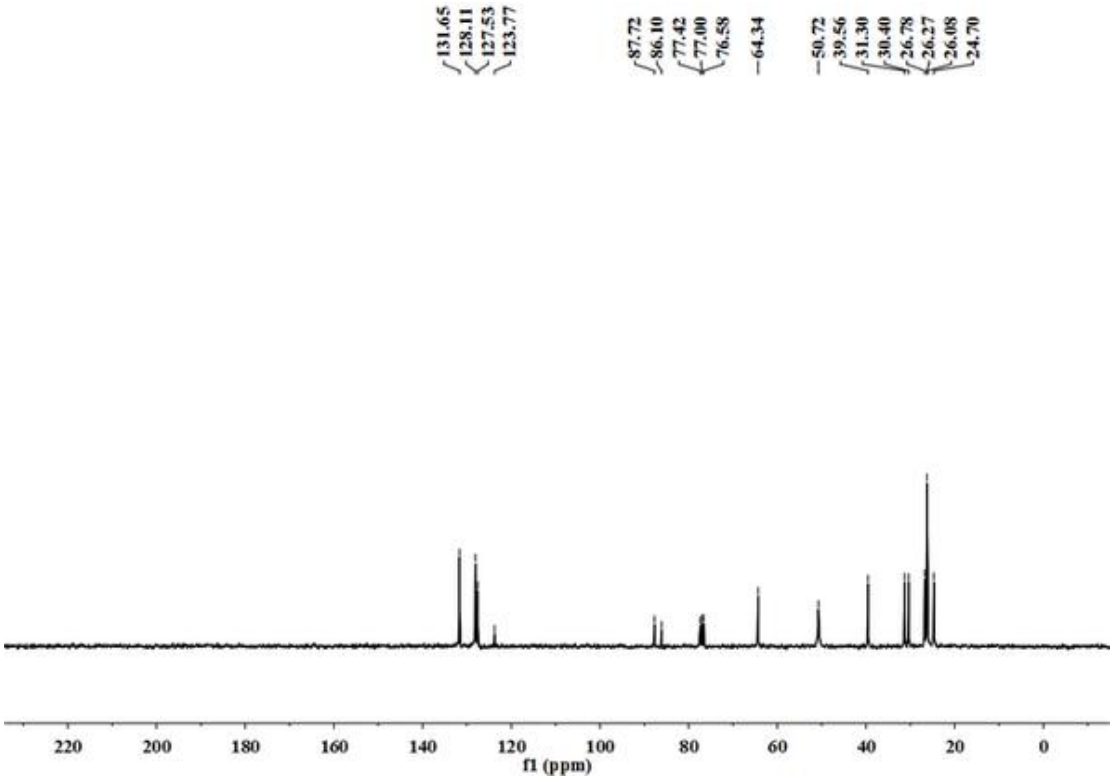
References

1. C. Wei, Z. Li, and C.-J. Li, *Org. Lett.* 2003, **5**, 4473-4475.
2. L. Shi, Y.-Q. Tu, M. Wang, F.-M. Zhang, and C.-A. Fan, *Org. Lett.* 2004, **6**, 1001-1003.
3. X. Zhang and A. Corma, *Angew. Chem. Int. Ed.* 2008, **47**, 4358 –4361.
4. K. Namitharan and K. Pitchumani, *Eur. J. Org. Chem.* 2010, 411–415.
5. V. K.-Y. Lo, C.-Y. Zhou, M.-K. Wong and C.-M. Che, *Chem. Commun.* 2010, **46**, 213–215.
6. N. Gommermann and P. Knochel, *Chem. Eur. J.* 2006, **12**, 4380 – 4392.
7. Z. Li, C. Wei, L. Chen, R. S. Varma and C.-J. Li, *Tetrahedron Lett.* 2004, **45**, 2443-2446.
8. B. Sreedhar, A. S. Kumar and P. S. Reddy, *Tetrahedron Lett*. 2010, **51**, 1891–1895.
9. E. Ramu, R. Varala, N. Sreelatha and S. R. Ada, *Tetrahedron Lett.* 2007, **48**, 7184–7190.
10. M. J. Aliaga, D. J. Ramón and M. Yus, *Org. Biomol. Chem.* 2010, **8**, 43–46.

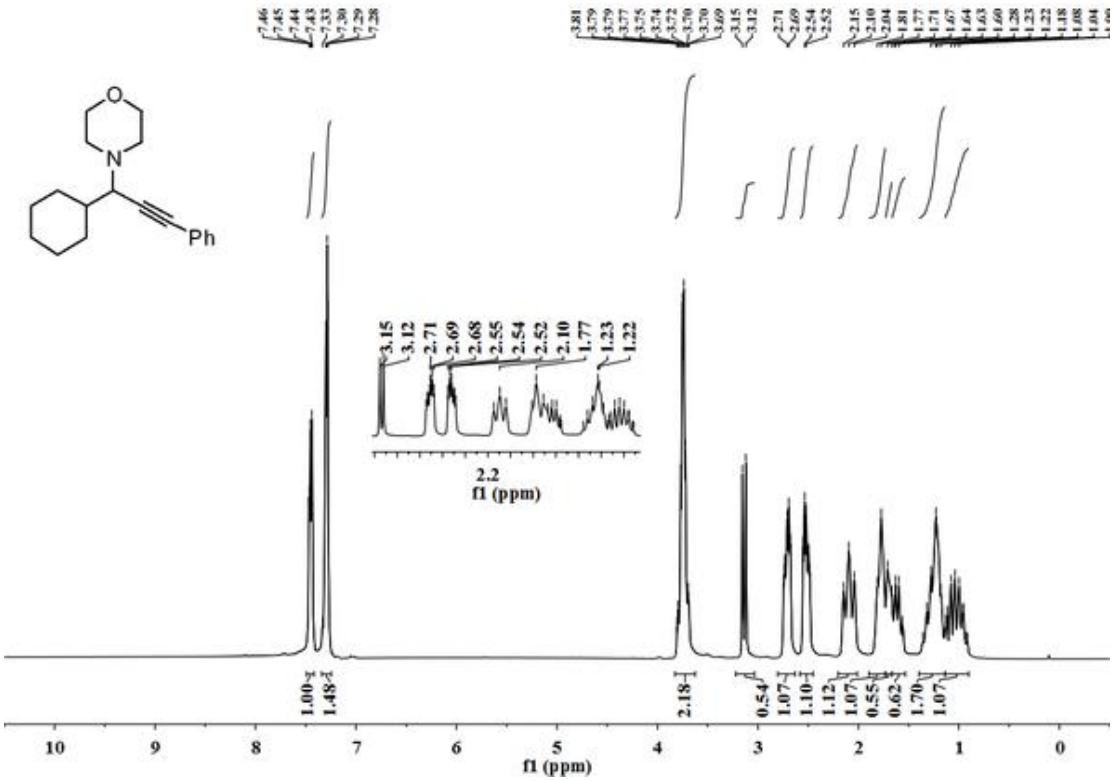
NMR Spectra

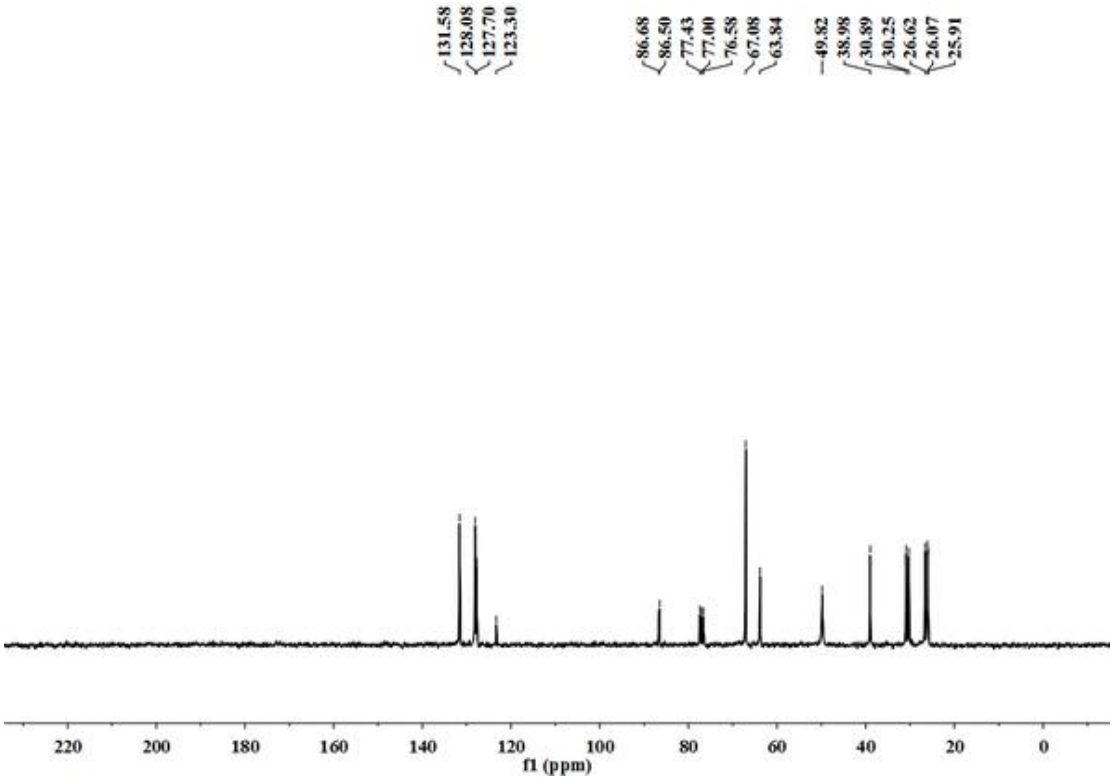
(Scheme 2, **7**)



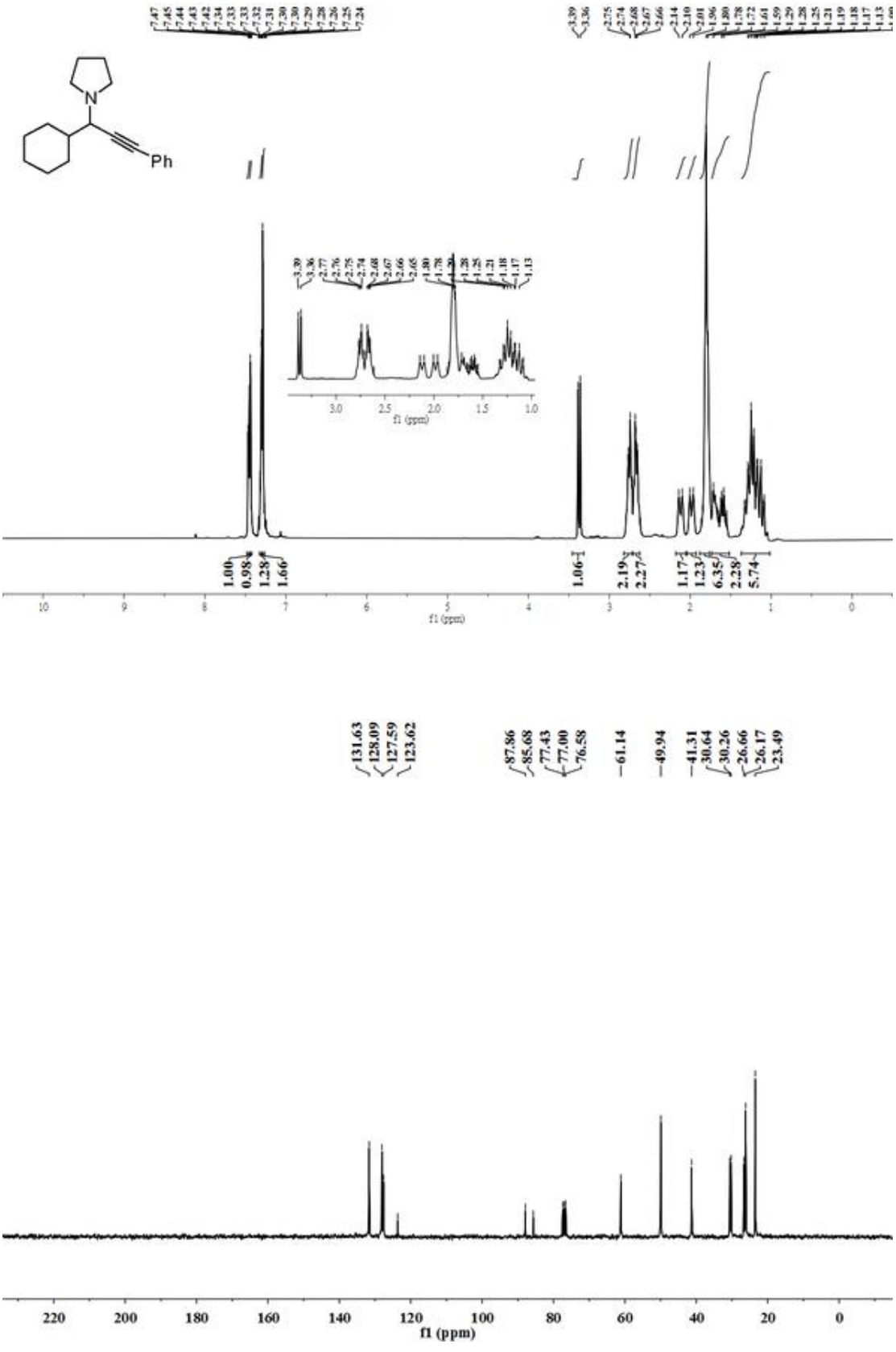


(Scheme 2, **8**)

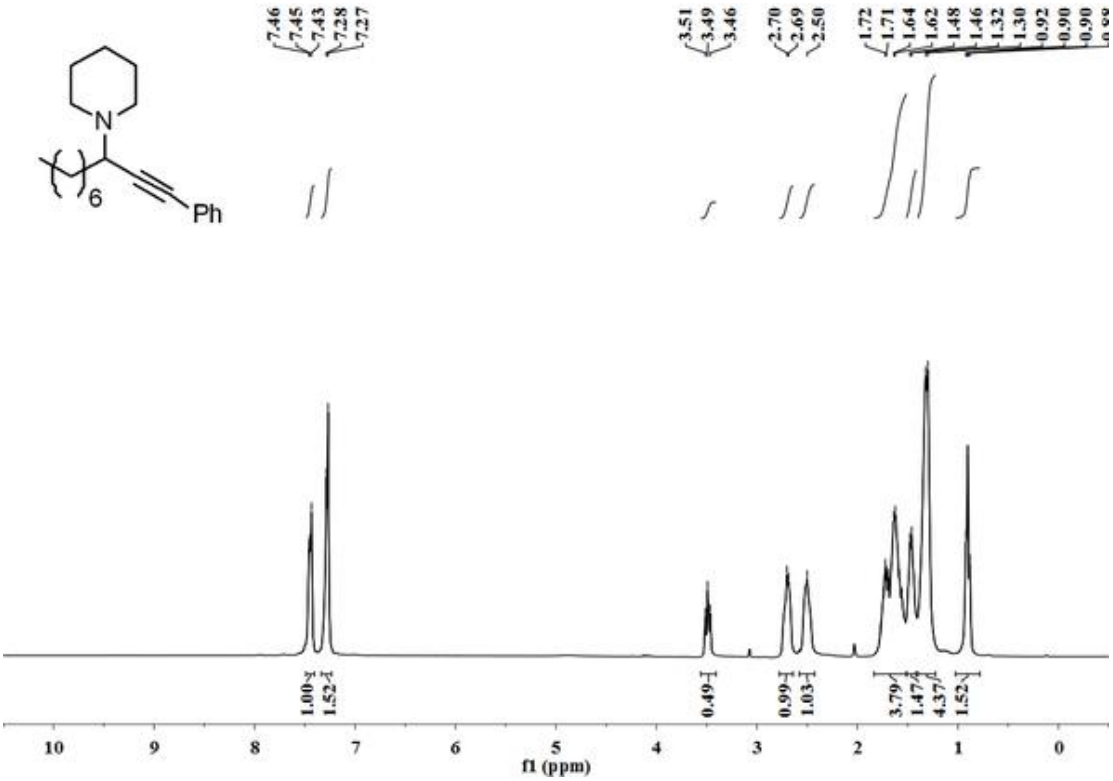

O
N
Ph
3.15
3.12
2.71
2.69
2.68
2.55
2.54
2.52
2.10
1.77
1.23
1.22
2.2
f1 (ppm)
1.00
1.48
2.18
0.54
1.07
1.10
1.12
1.07
0.55
0.62
1.70
1.07
10
9
8
7
6
5
4
3
2
1
0
f1 (ppm)

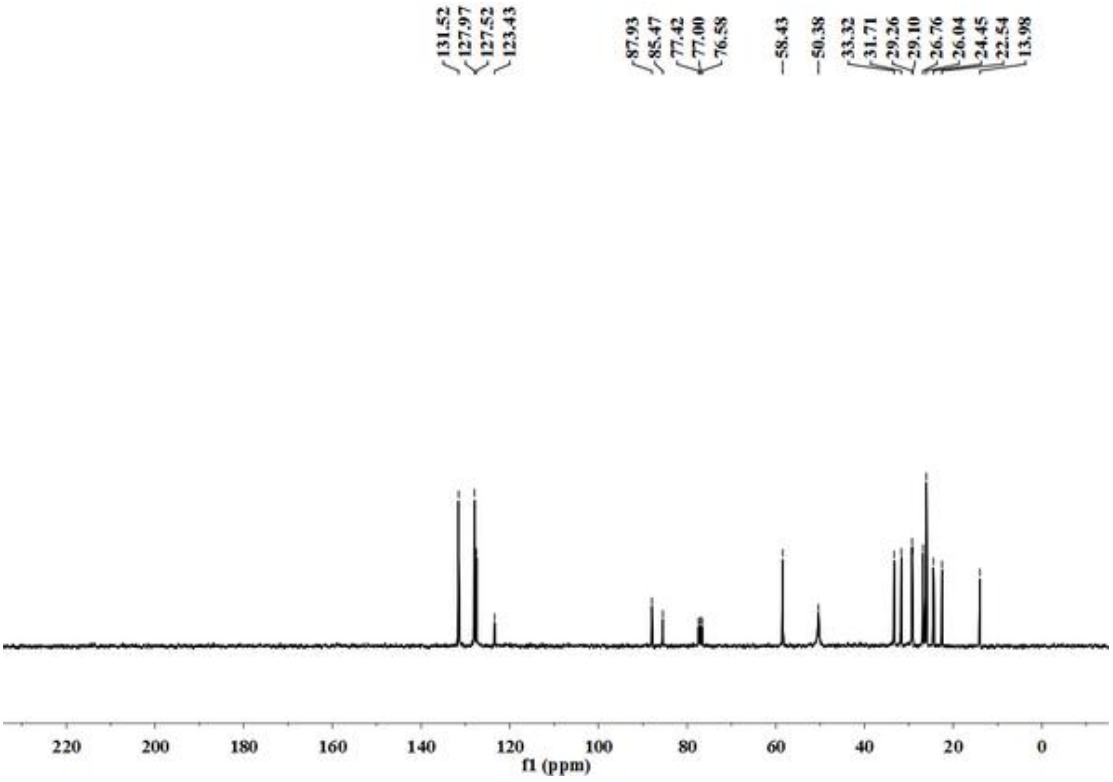

131.58
128.08
127.70
123.30
86.68
86.50
77.43
77.00
76.58
67.08
63.84
49.82
38.98
30.89
30.25
26.62
26.07
25.91
220
200
180
160
140
120
100
80
60
40
20
0
f1 (ppm)

(Scheme 2, **9**)

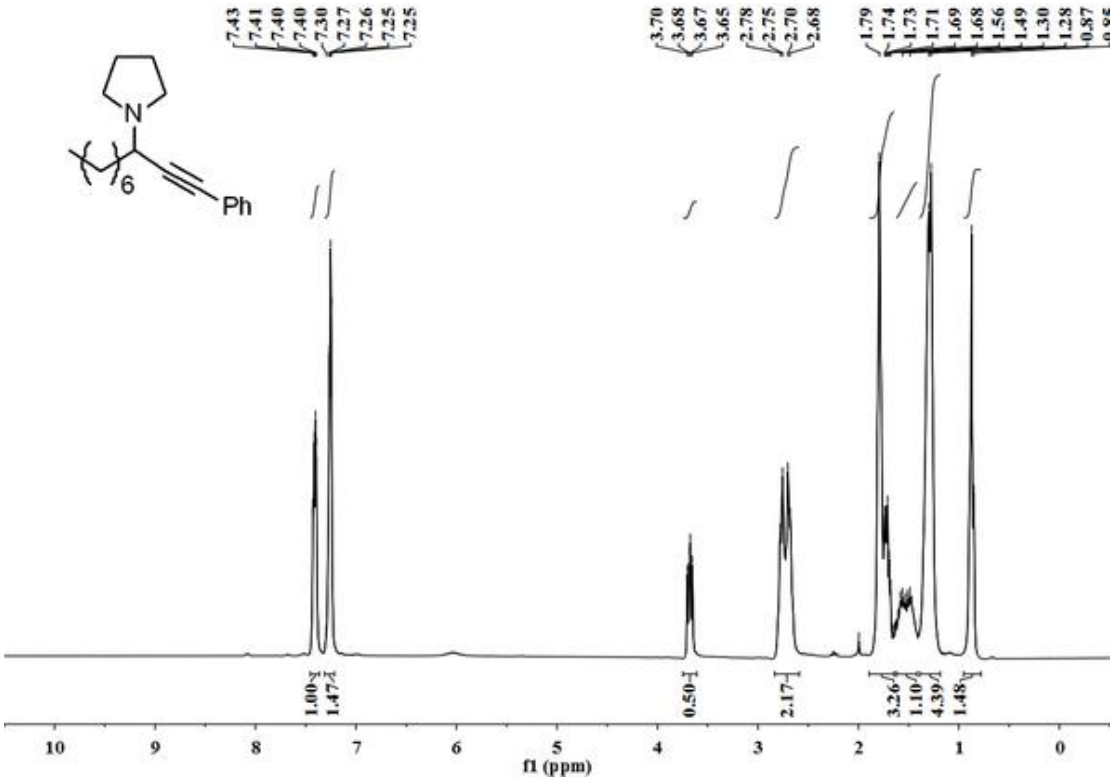

N
Ph
1.00
0.98
1.28
1.66
1.06
2.19
2.27
1.17
1.23
6.35
2.28
5.74
f1 (ppm)
131.63
128.09
127.59
123.62
87.86
85.68
77.43
77.00
76.58
61.14
49.94
41.31
30.64
30.26
26.66
26.17
23.49
220
200
180
160
140
120
100
80
60
40
20
0
f1 (ppm)

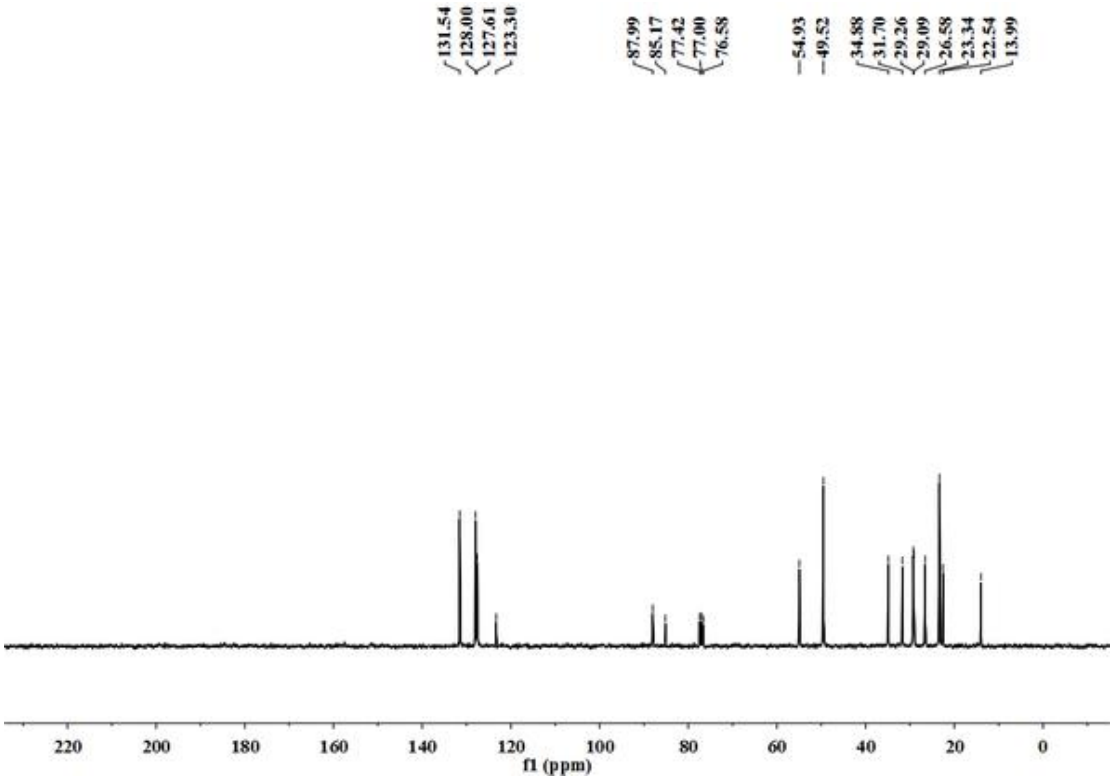
(Scheme 2, **10**)



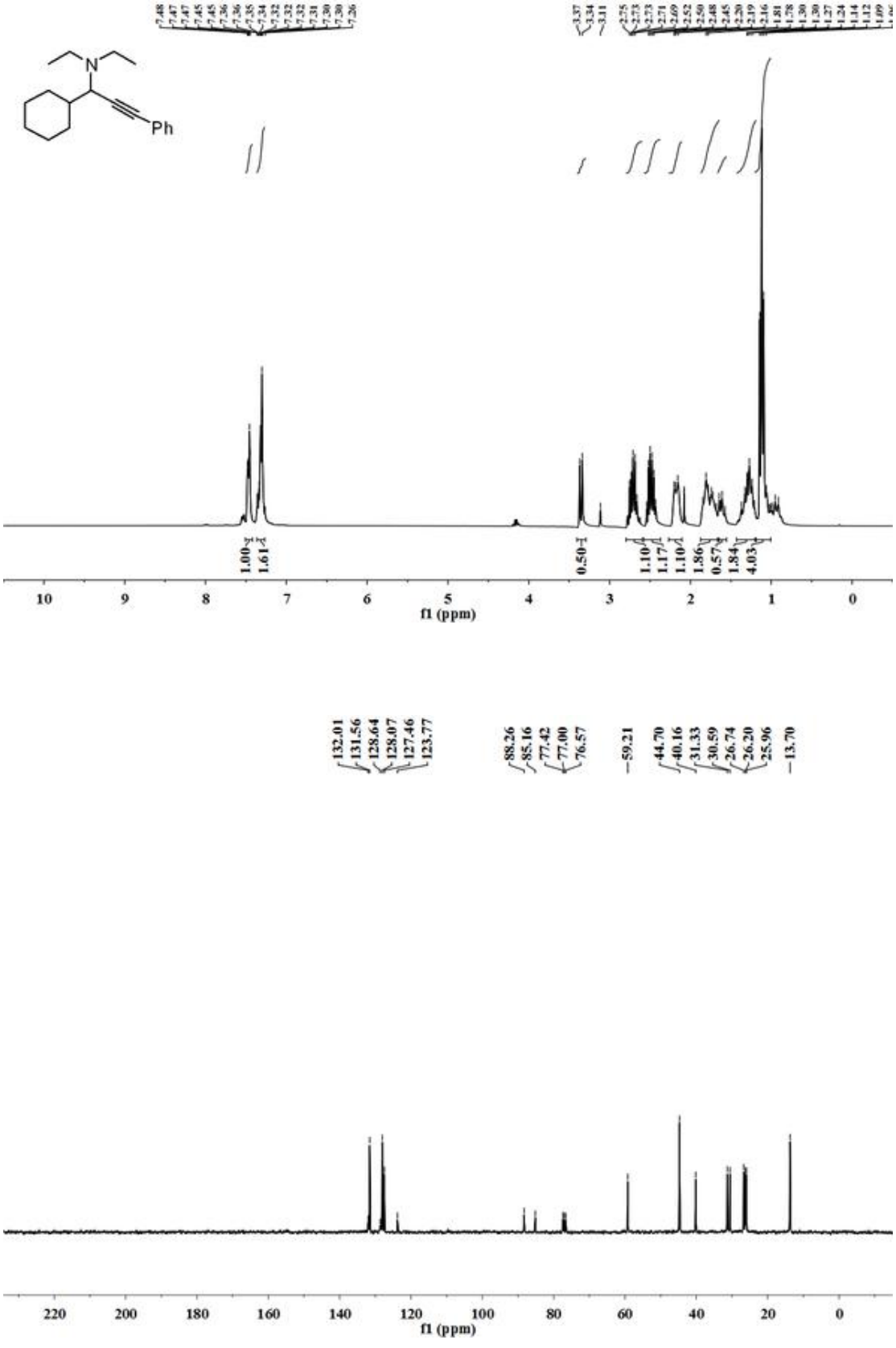


(Scheme 2, **11**)

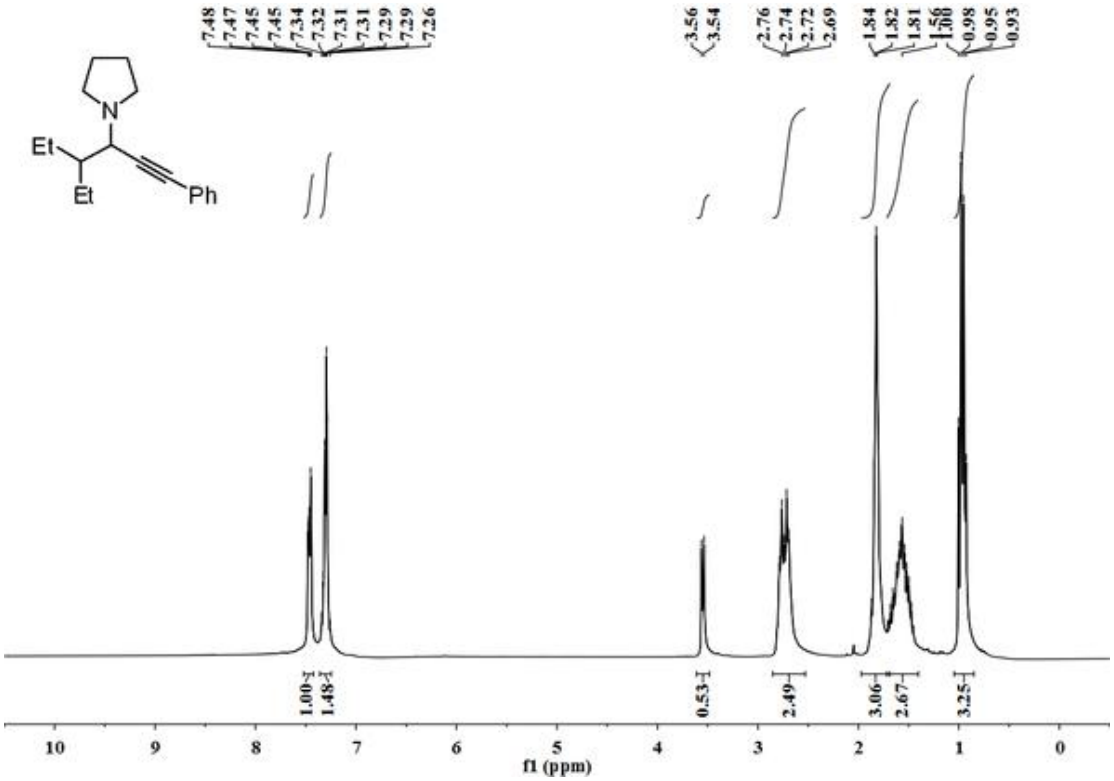


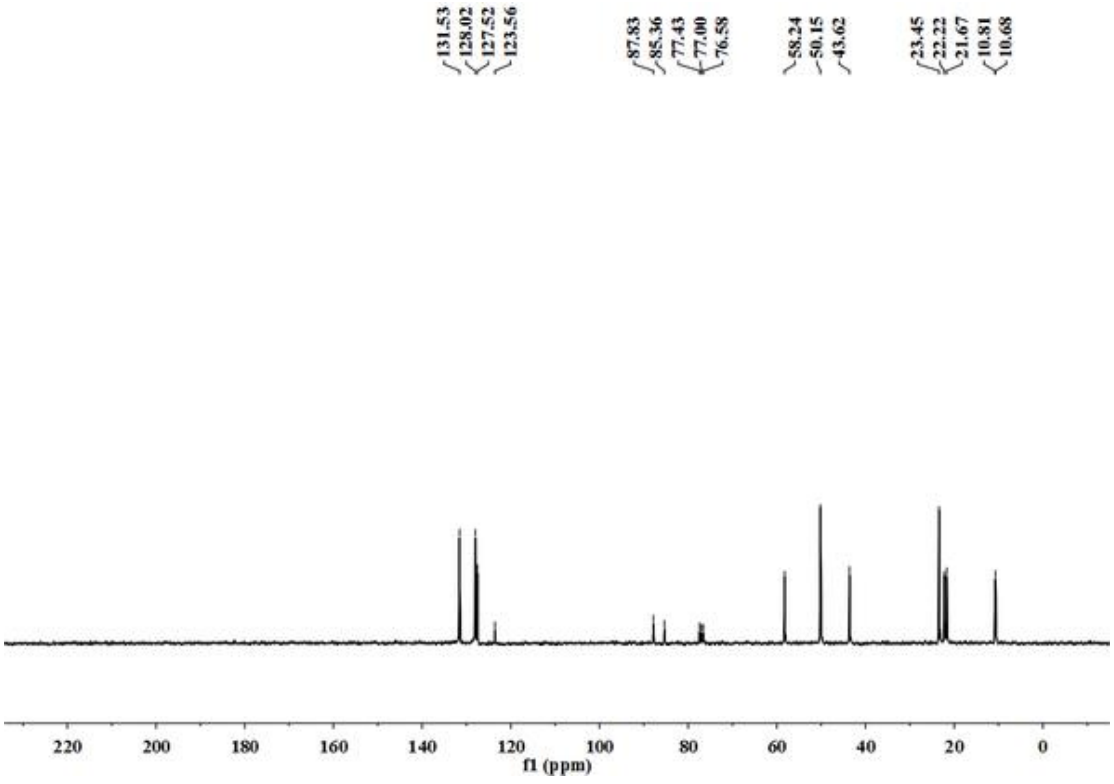


(Scheme 2, **12**)

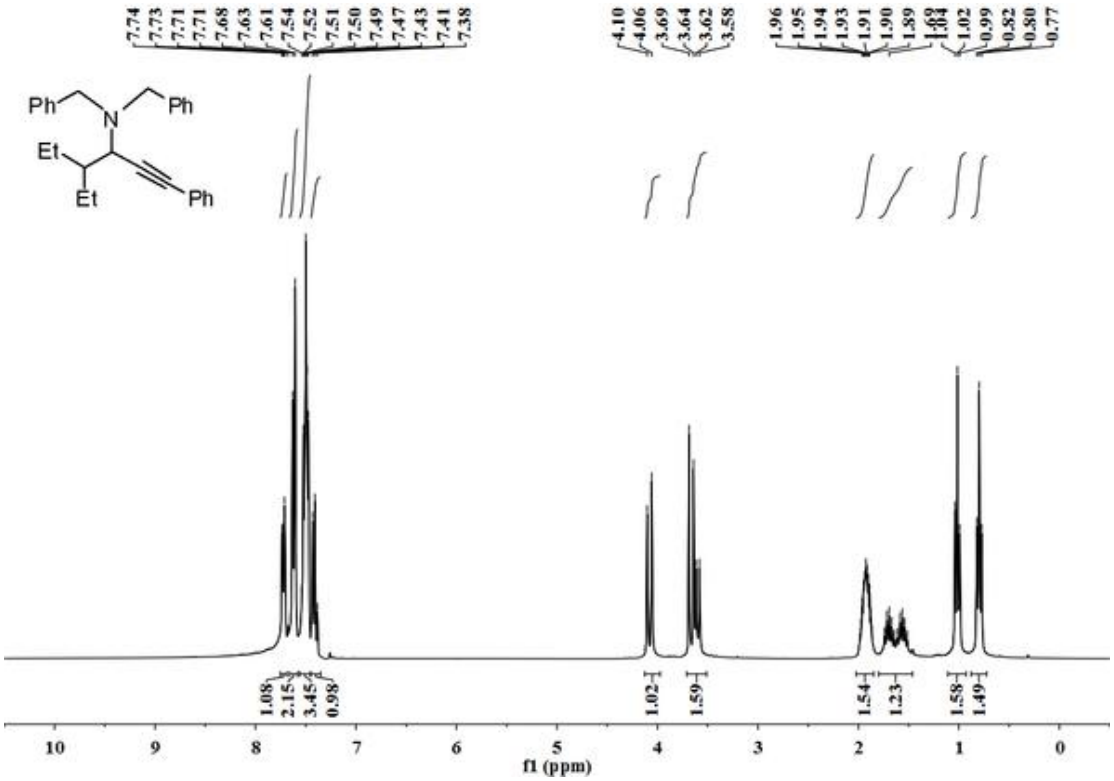

N
Ph
1.00
1.61
0.50
1.10
1.17
1.10
1.86
0.57
1.84
4.03
f1 (ppm)
132.01
131.56
128.64
128.07
127.46
123.77
88.26
85.16
77.42
77.00
76.57
59.21
44.70
40.16
31.33
30.59
26.74
26.20
25.96
13.70
f1 (ppm)

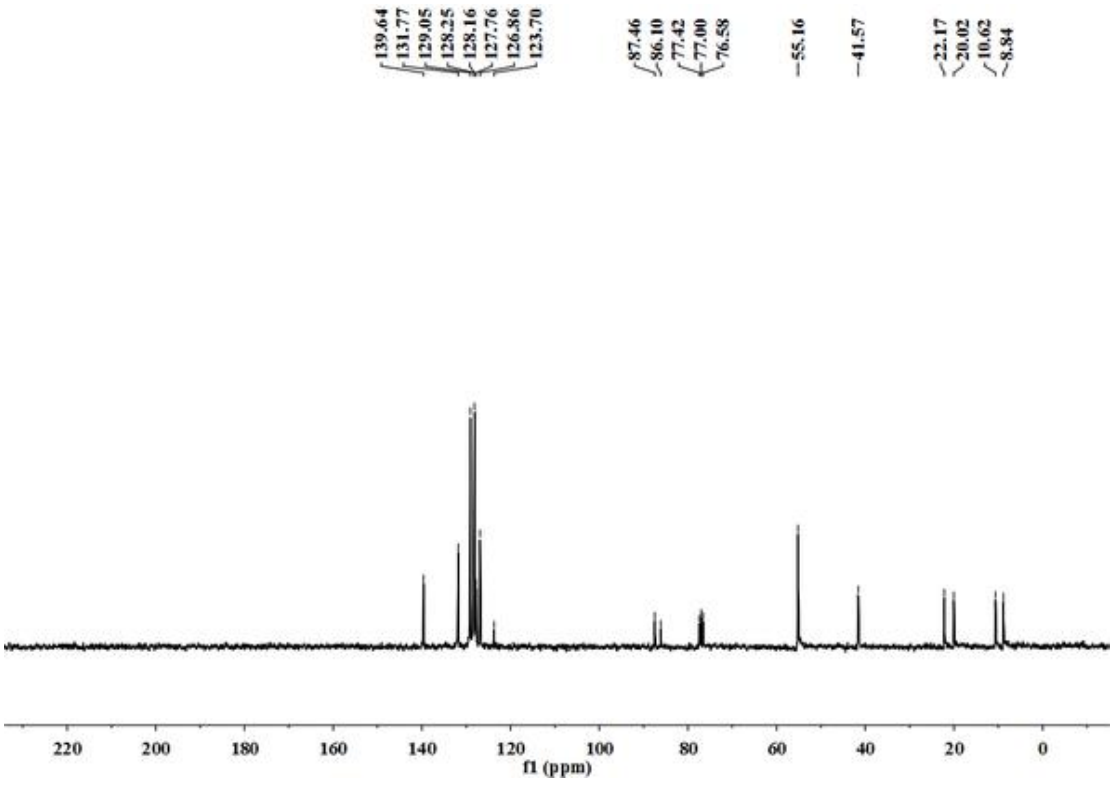
(Scheme 2, **13**)



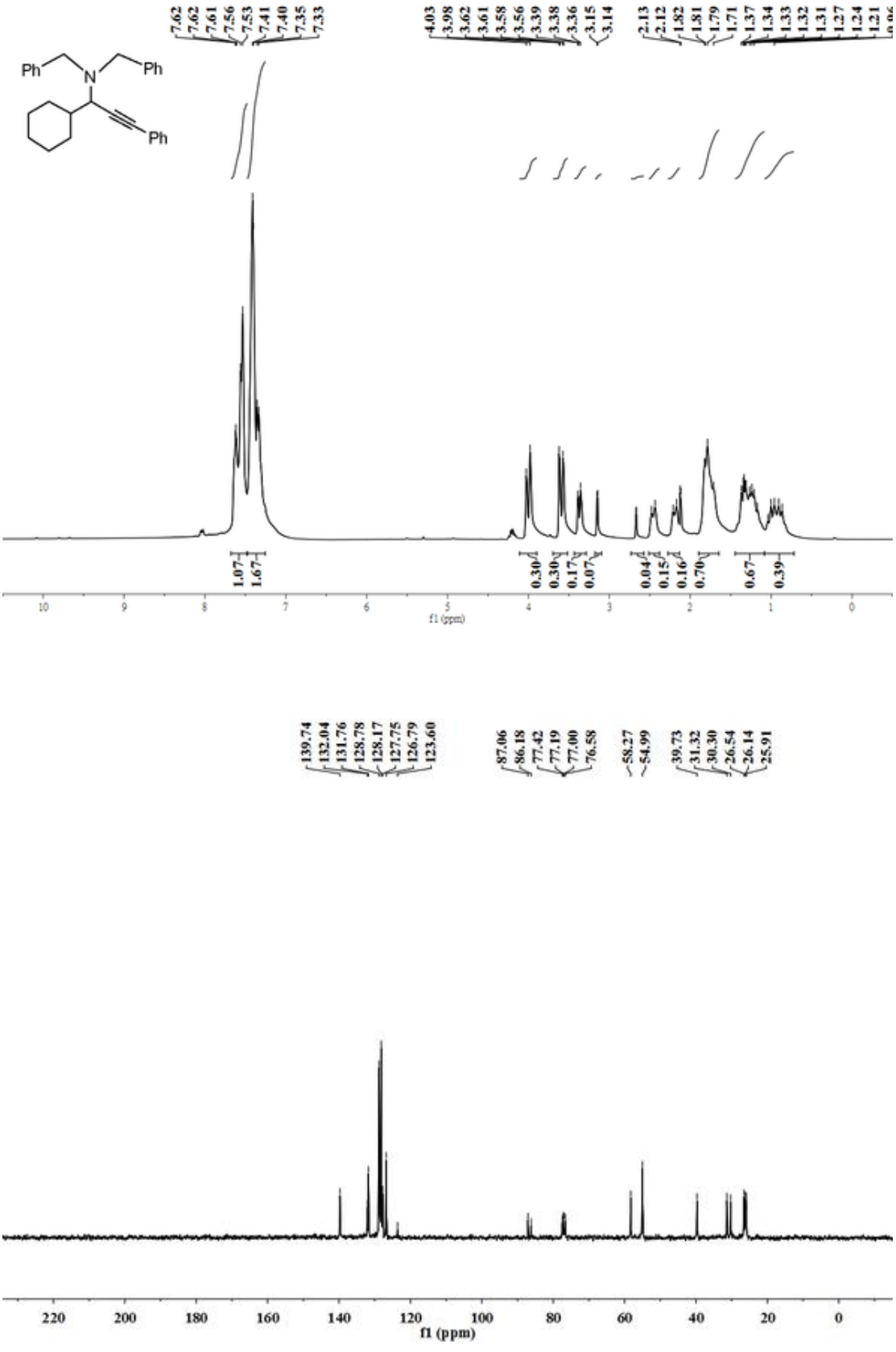


(Scheme 2, **14**)

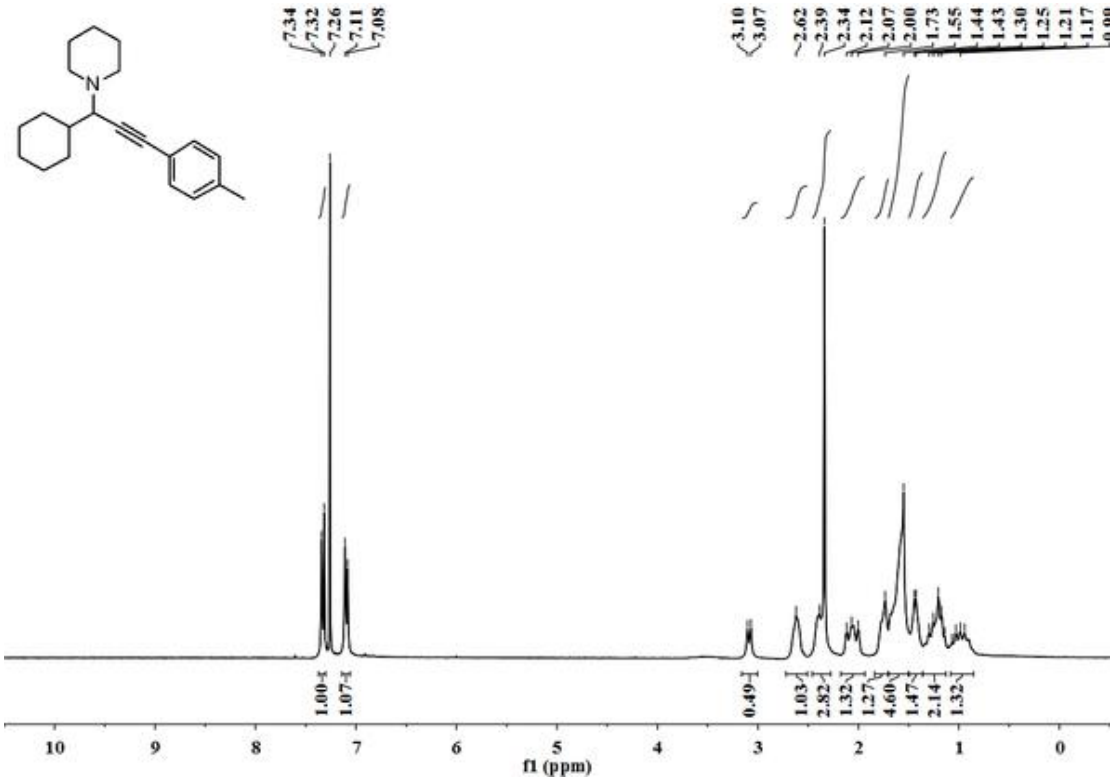

7.74
7.73
7.71
7.71
7.68
7.63
7.61
7.54
7.52
7.51
7.50
7.49
7.47
7.43
7.41
7.38
4.10
4.06
3.69
3.64
3.62
3.58
1.96
1.95
1.94
1.93
1.91
1.90
1.89
1.02
0.99
0.82
0.80
0.77
Ph
N
Ph
Et
Et
Ph
1.08
2.15
3.45
0.98
1.02
1.59
1.54
1.23
1.58
1.49
10
9
8
7
6
5
4
3
2
1
0
f1 (ppm)

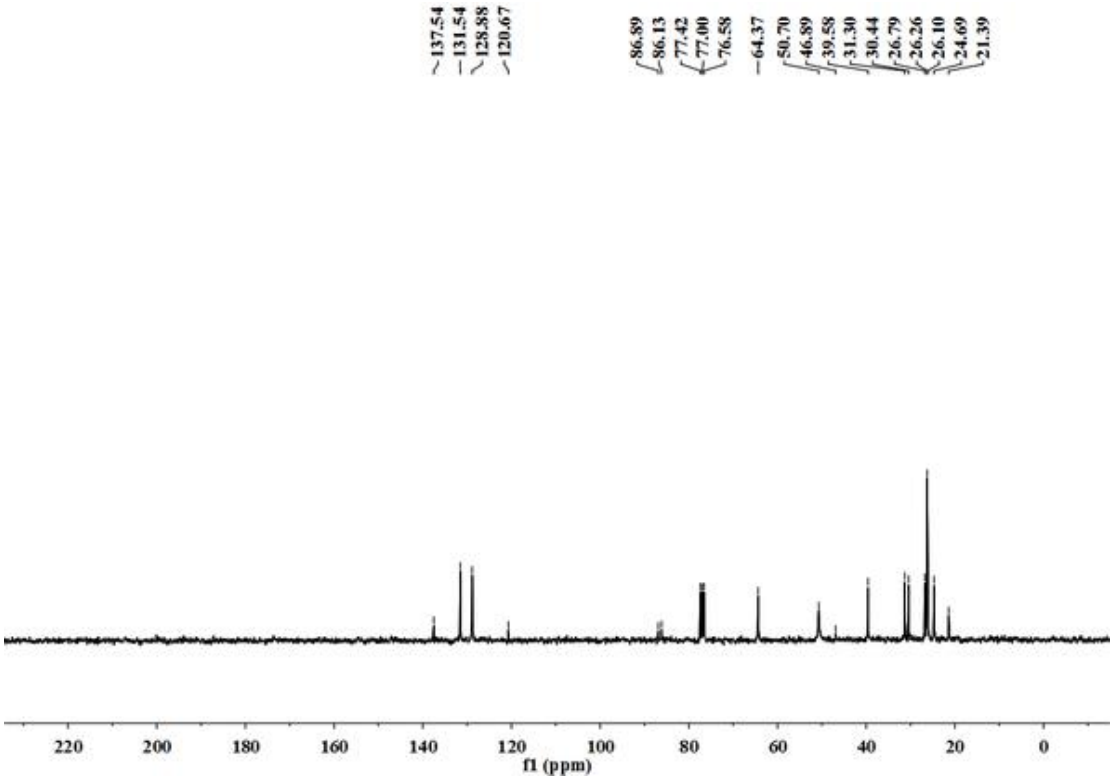

139.64
131.77
129.05
128.25
128.16
127.76
126.86
123.70
87.46
86.10
77.42
77.00
76.58
55.16
41.57
22.17
20.02
10.62
8.84
220
200
180
160
140
120
100
80
60
40
20
0
f1 (ppm)

(Scheme 2, **15**)

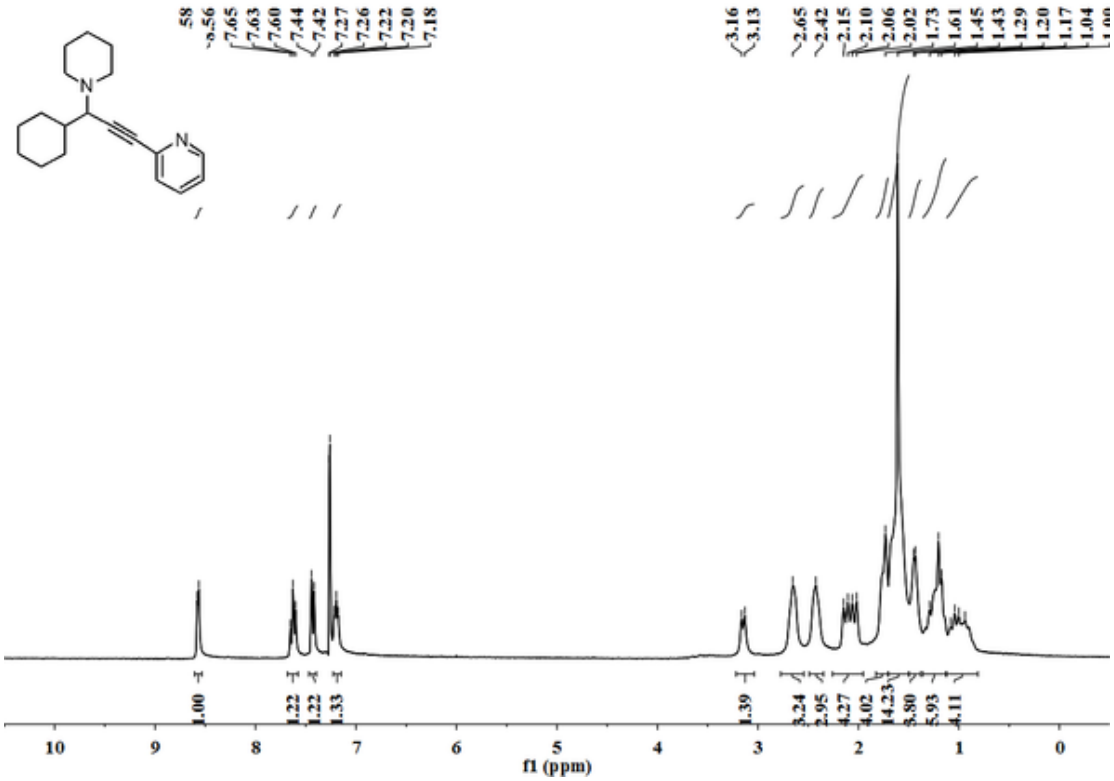

Ph
N
Ph
Ph
7.62
7.62
7.61
7.56
7.53
7.41
7.40
7.35
7.33
4.03
3.98
3.62
3.61
3.58
3.56
3.39
3.38
3.36
3.15
3.14
2.13
2.12
1.82
1.81
1.79
1.71
1.37
1.34
1.33
1.32
1.31
1.27
1.24
1.21
1.07
1.67
0.30
0.30
0.17
0.07
0.04
0.15
0.16
0.70
0.67
0.39
f1 (ppm)
139.74
132.04
131.76
128.78
128.17
127.75
126.79
123.60
87.06
86.18
77.42
77.19
77.00
76.58
58.27
54.99
39.73
31.32
30.30
26.54
26.14
25.91
f1 (ppm)

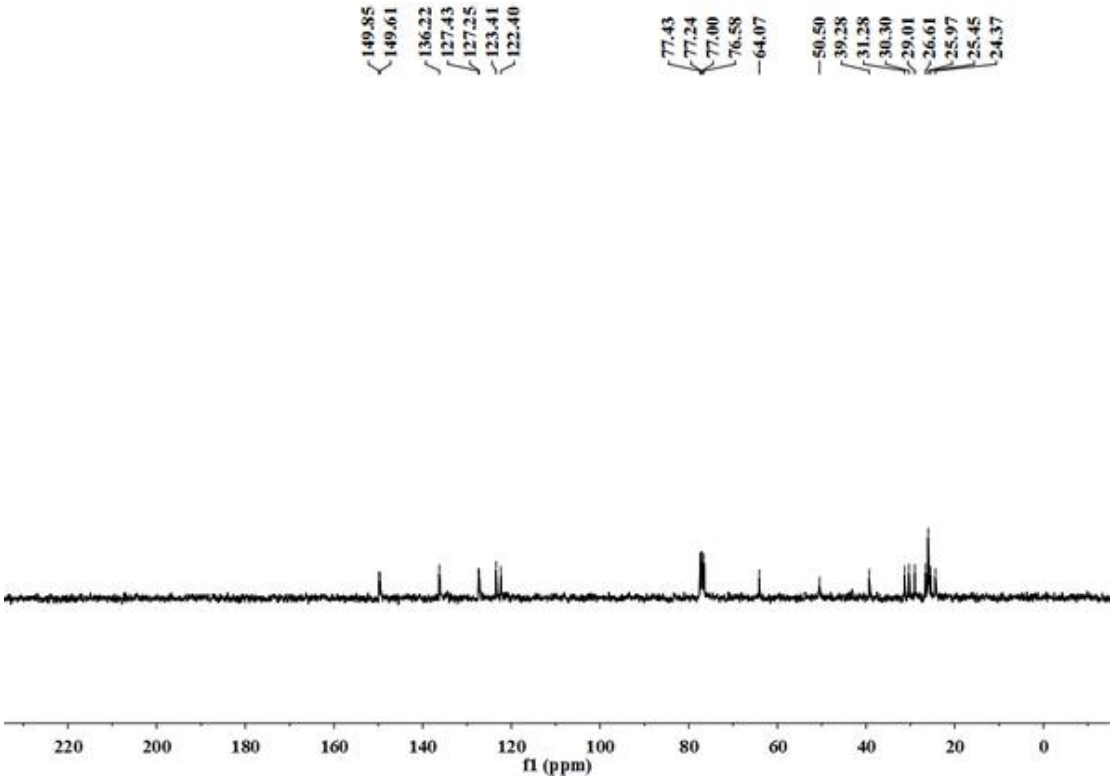
(Scheme 2, **16**)



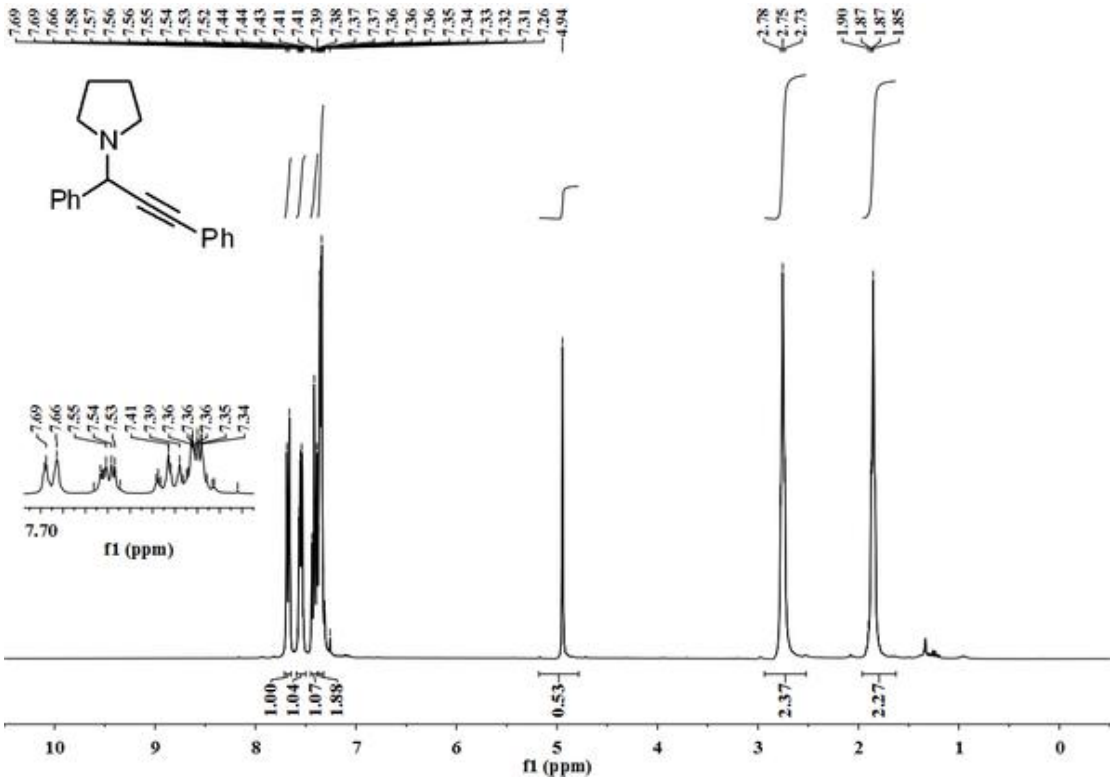


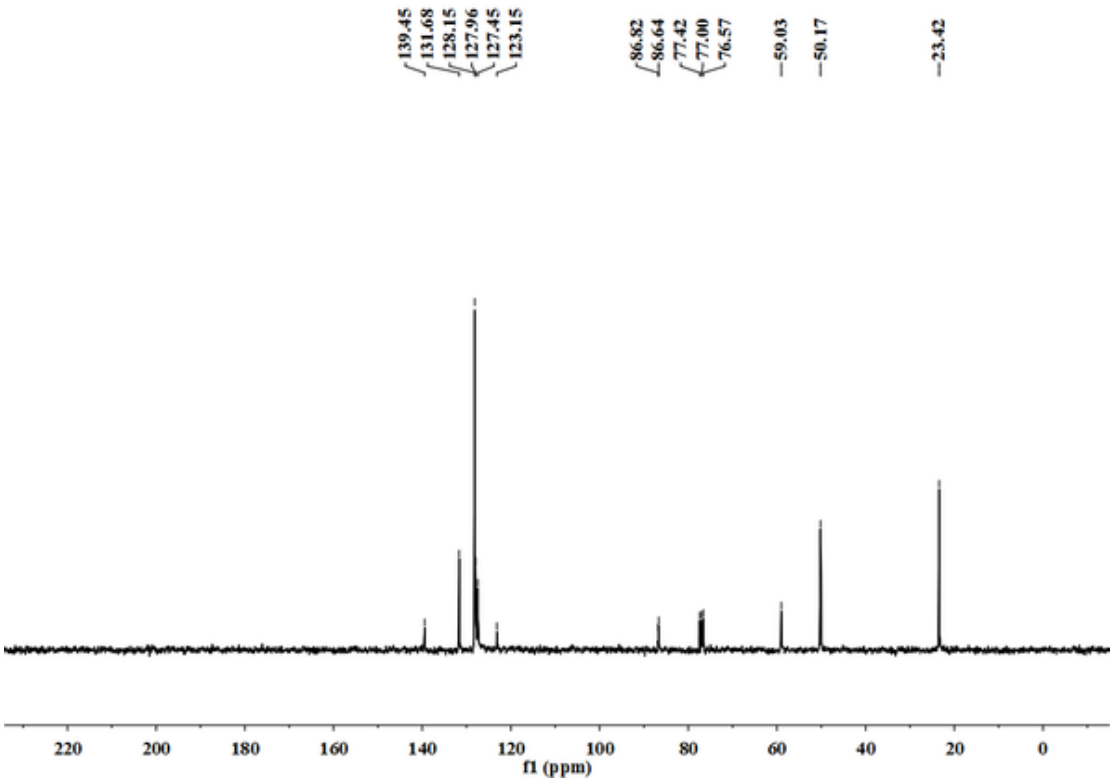
(Scheme 2, **17**)


7.65
7.63
7.60
7.44
7.42
7.27
7.26
7.22
7.20
7.18
3.16
3.13
2.65
2.42
2.15
2.10
2.06
2.02
1.73
1.61
1.45
1.43
1.29
1.20
1.17
1.04
1.00
1.22
1.22
1.33
1.39
3.24
2.95
4.27
4.02
14.23
3.80
5.93
4.11
10
9
8
7
6
5
4
3
2
1
0
f1 (ppm)

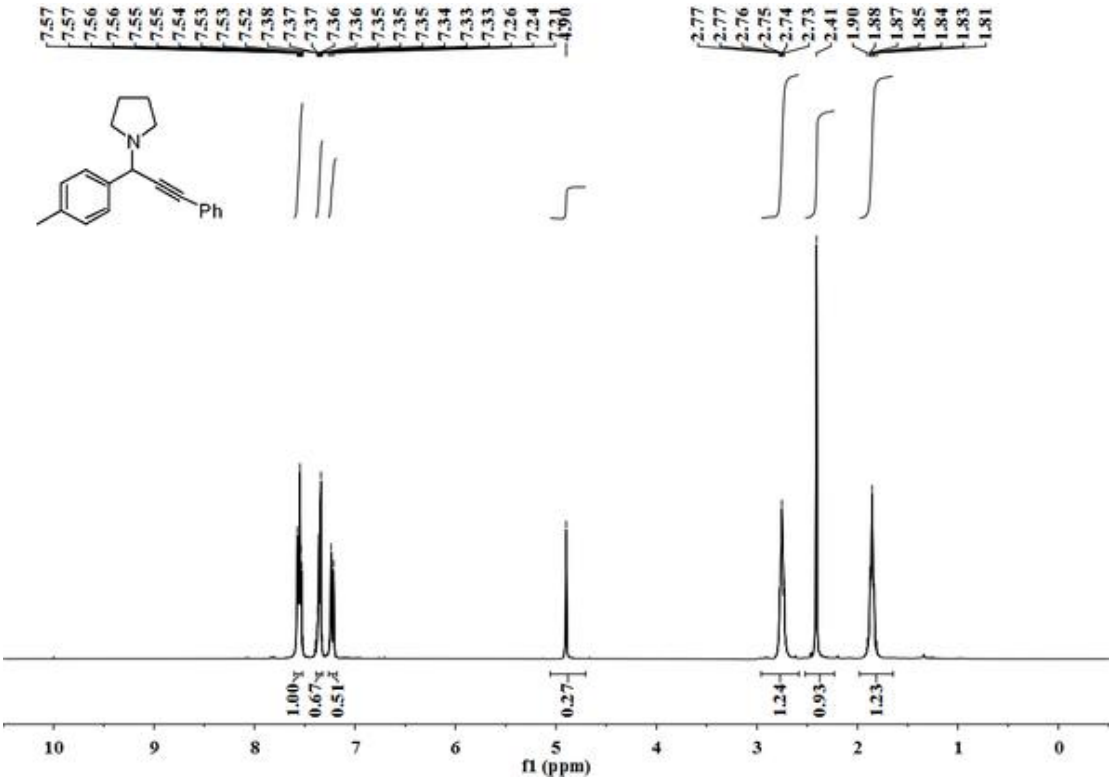

149.85
149.61
136.22
127.43
127.25
123.41
122.40
77.43
77.24
77.00
76.58
64.07
50.50
39.28
31.28
30.30
29.01
26.61
25.97
25.45
24.37
220
200
180
160
140
120
100
80
60
40
20
0
f1 (ppm)

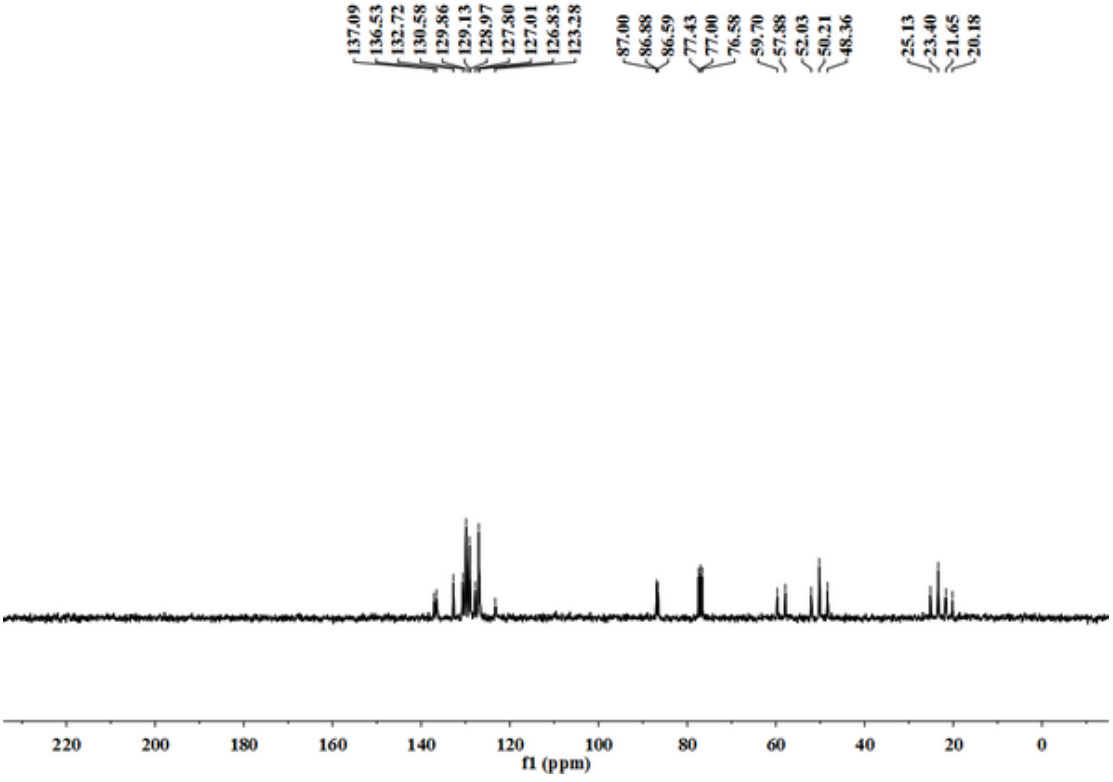
(Scheme 2, **18**)



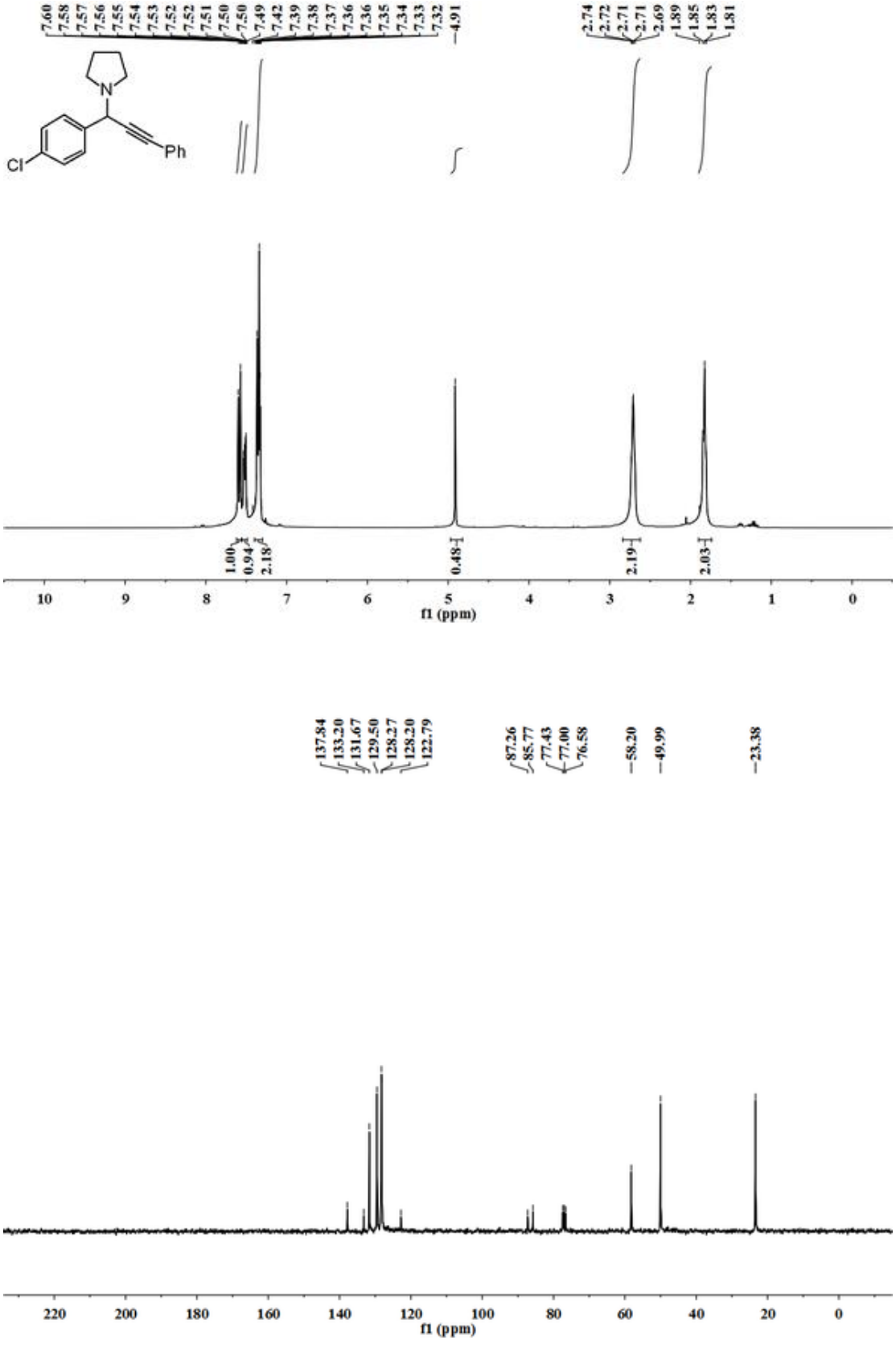


(Scheme 2, **19**)

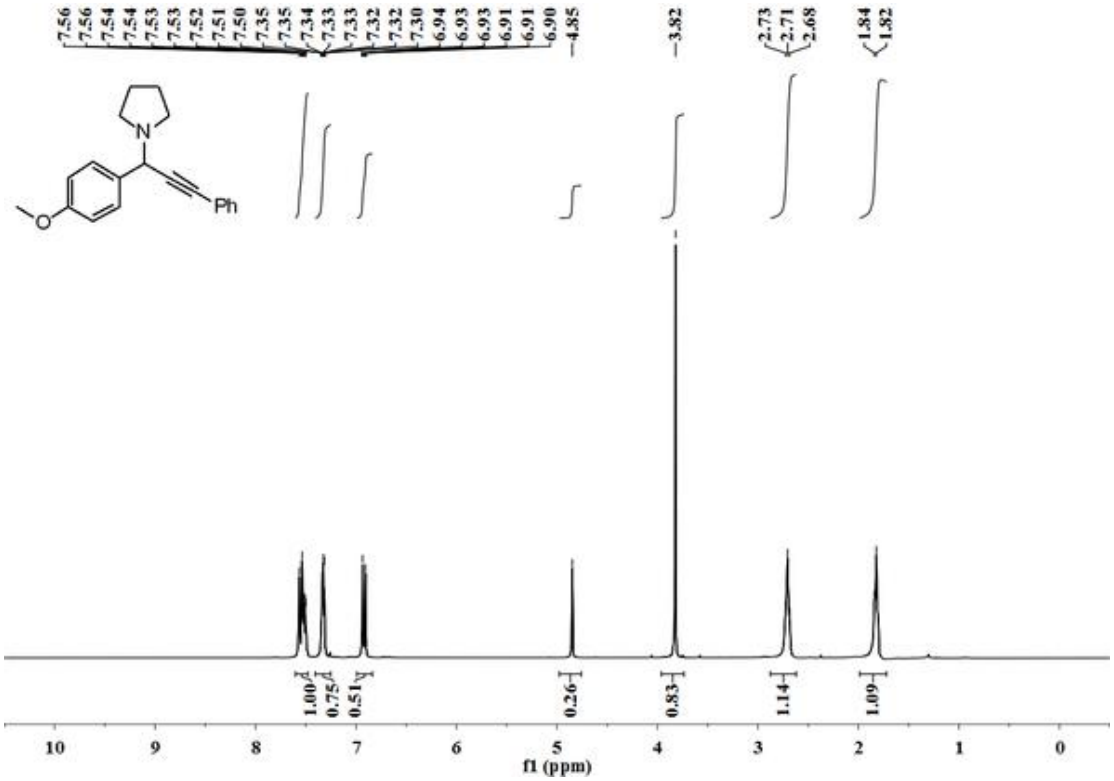

7.57
7.57
7.56
7.56
7.55
7.55
7.54
7.53
7.53
7.52
7.38
7.37
7.37
7.36
7.36
7.35
7.35
7.35
7.34
7.33
7.33
7.26
7.24
7.21
4.90
2.77
2.77
2.76
2.75
2.74
2.73
2.41
1.90
1.88
1.87
1.85
1.84
1.83
1.81
N
Ph
1.00
0.67
0.51
0.27
1.24
0.93
1.23
10
9
8
7
6
5
4
3
2
1
0
f1 (ppm)

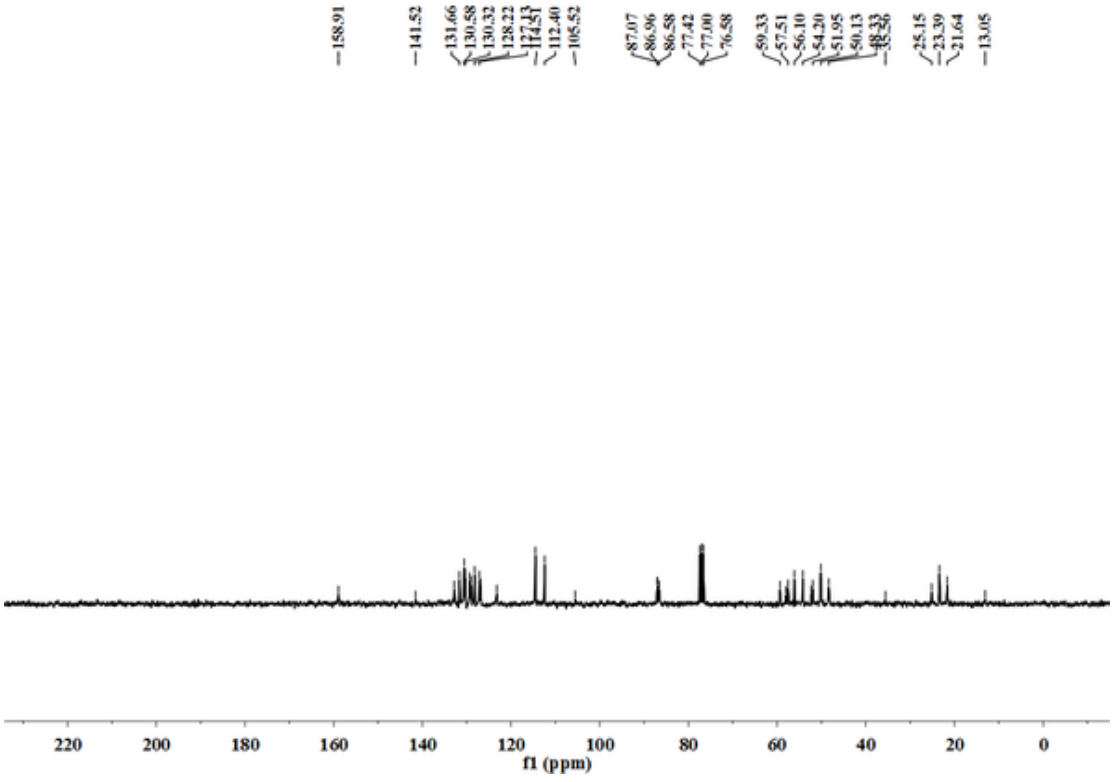

137.09
136.53
132.72
130.58
129.86
129.13
128.97
127.80
127.01
126.83
123.28
87.00
86.88
86.59
77.43
77.00
76.58
59.70
57.88
52.03
50.21
48.36
25.13
23.40
21.65
20.18
220
200
180
160
140
120
100
80
60
40
20
0
f1 (ppm)

(Scheme 2, **20**)

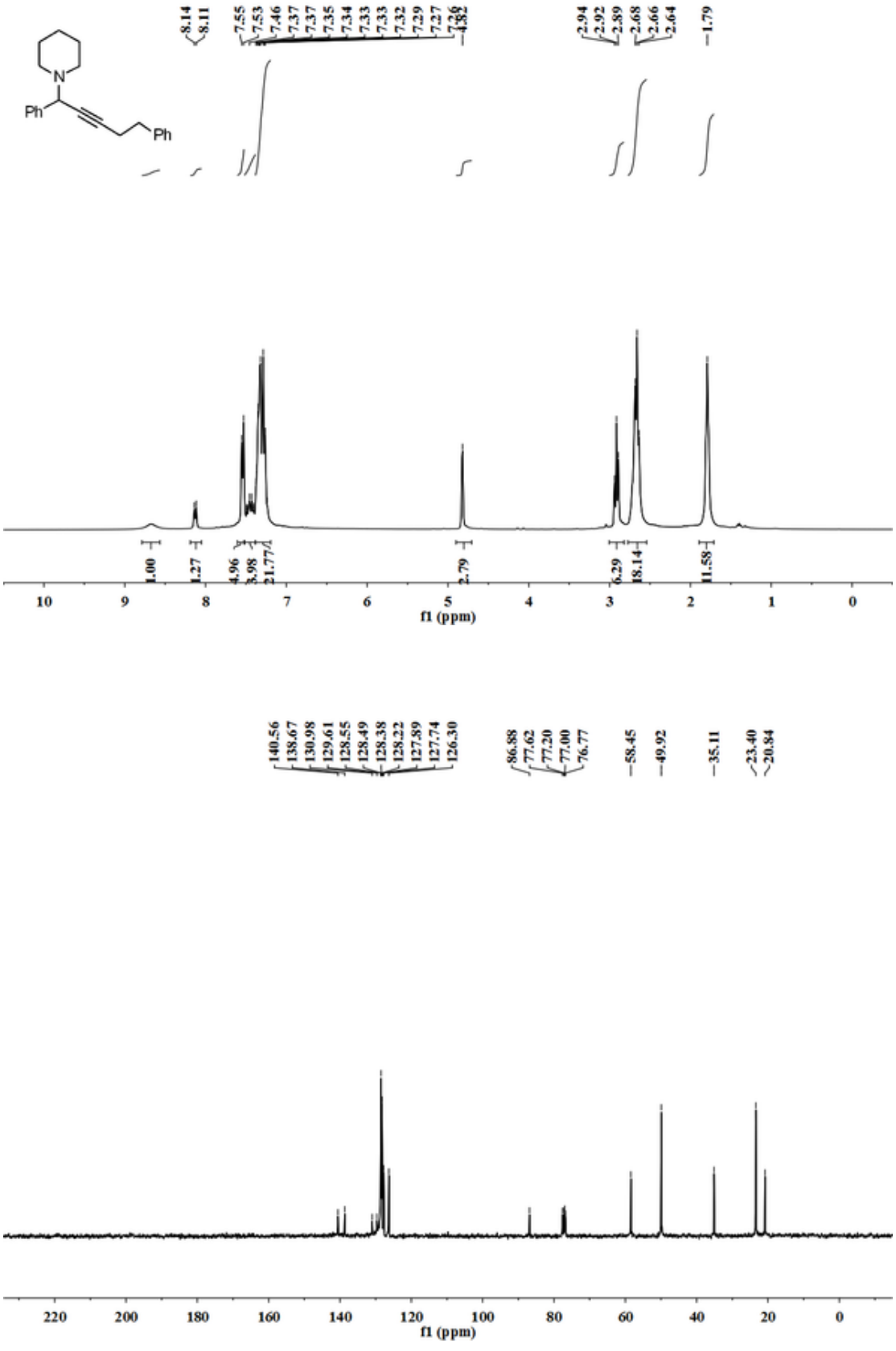

7.60
7.58
7.57
7.56
7.55
7.54
7.53
7.52
7.52
7.51
7.50
7.50
7.49
7.42
7.39
7.38
7.37
7.36
7.36
7.35
7.34
7.33
7.32
4.91
2.74
2.72
2.71
2.71
2.69
1.89
1.85
1.83
1.81
N
Cl
Ph
1.00
0.94
2.18
0.48
2.19
2.03
10
9
8
7
6
5
4
3
2
1
0
f1 (ppm)
137.84
133.20
131.67
129.50
128.27
128.20
122.79
87.26
85.77
77.43
77.00
76.58
58.20
49.99
23.38
220
200
180
160
140
120
100
80
60
40
20
0
f1 (ppm)

(Scheme 2, **21**)

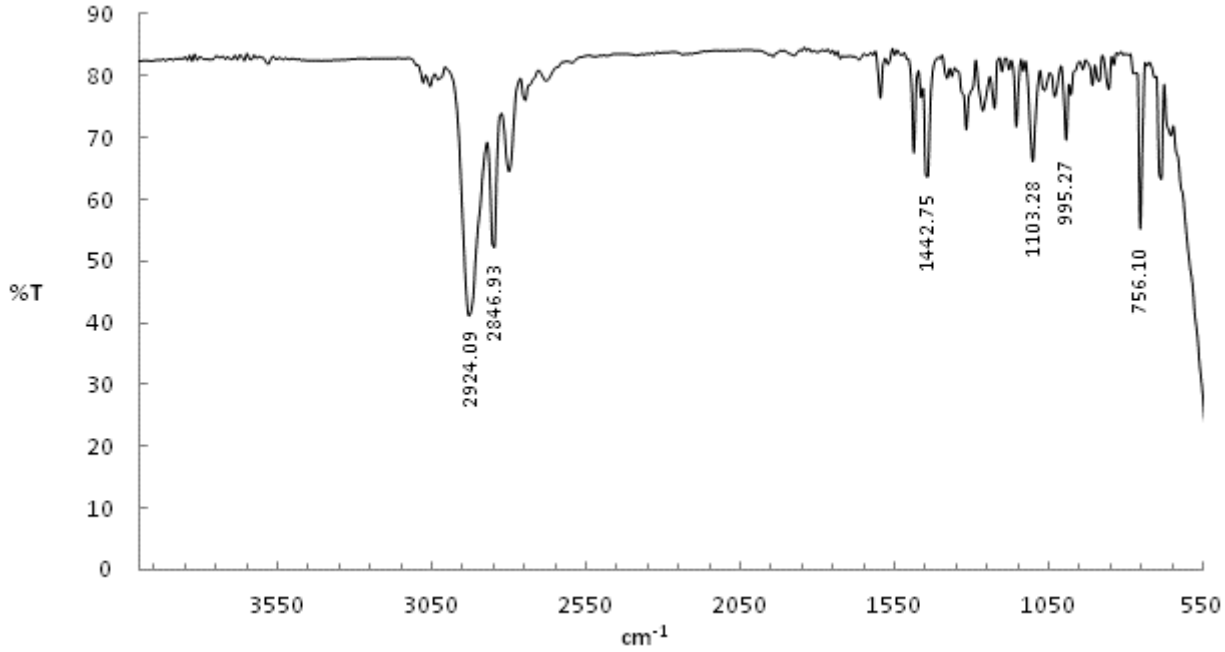




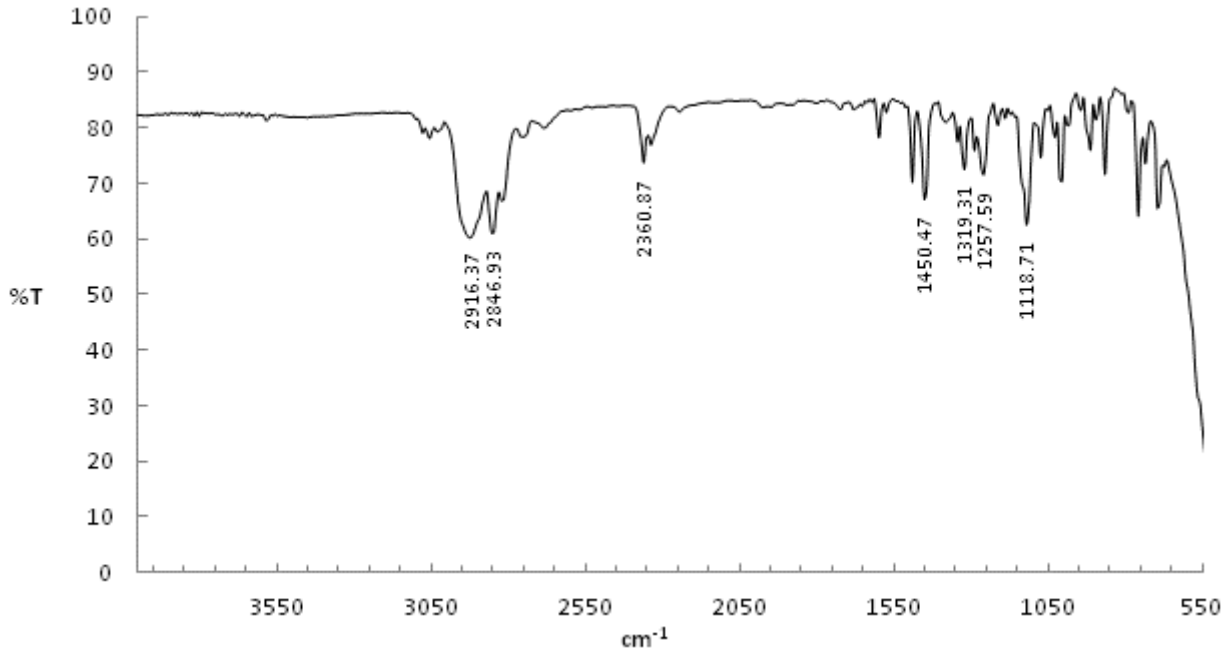
(Scheme 2, **22**)


N
Ph
Ph
8.14
8.11
7.55
7.53
7.46
7.37
7.37
7.35
7.34
7.33
7.33
7.32
7.29
7.27
7.26
2.94
2.92
2.89
2.68
2.66
2.64
1.79
1.00
1.27
4.96
3.98
21.77
2.79
6.29
8.14
11.58
f1 (ppm)
140.56
138.67
130.98
129.61
128.55
128.49
128.38
128.22
127.89
127.74
126.30
86.88
77.62
77.20
77.00
76.77
58.45
49.92
35.11
23.40
20.84
f1 (ppm)

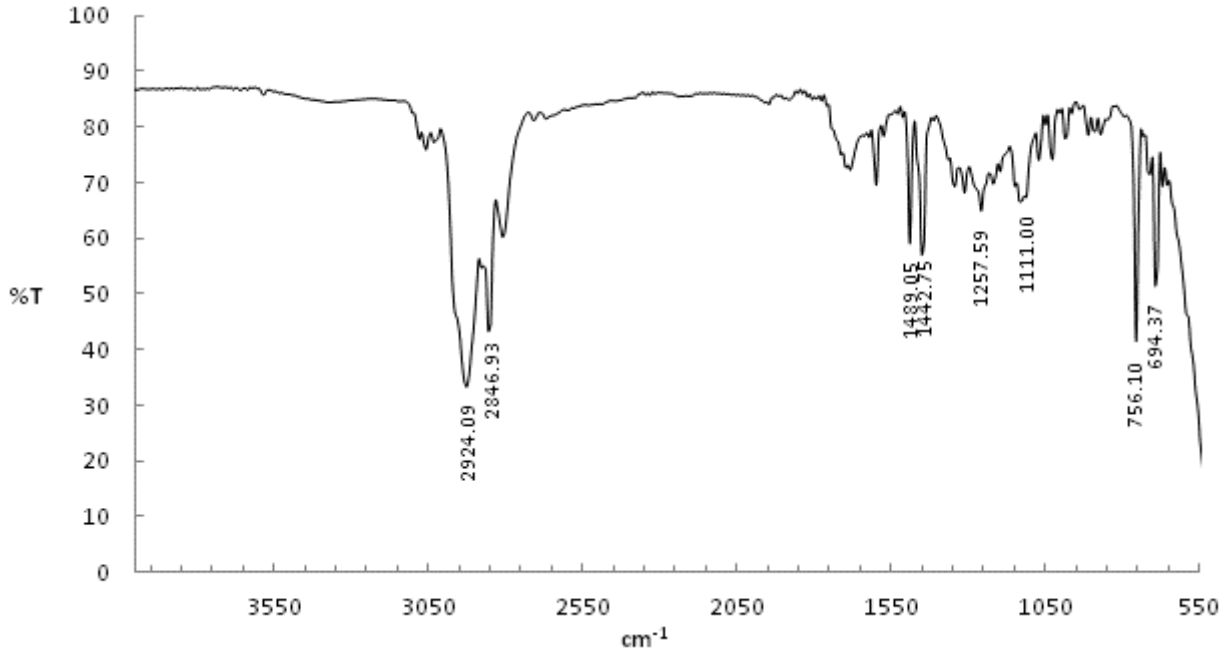
Compound 7: FTIR (NaCl plates, neat)



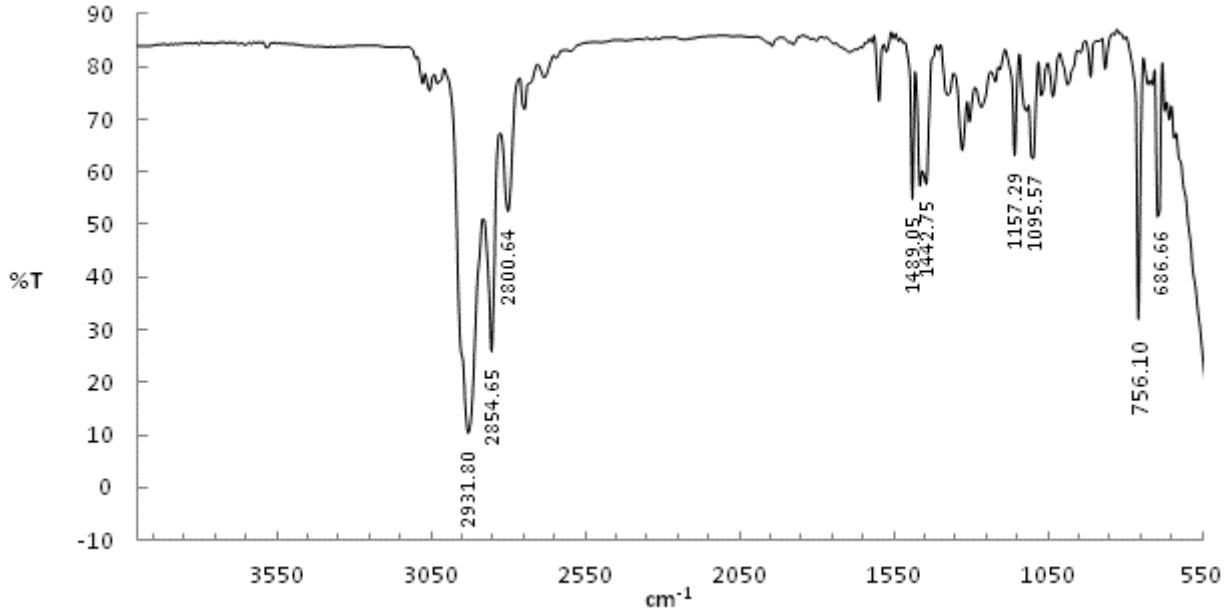
Compound 8: FTIR (NaCl plates, neat)



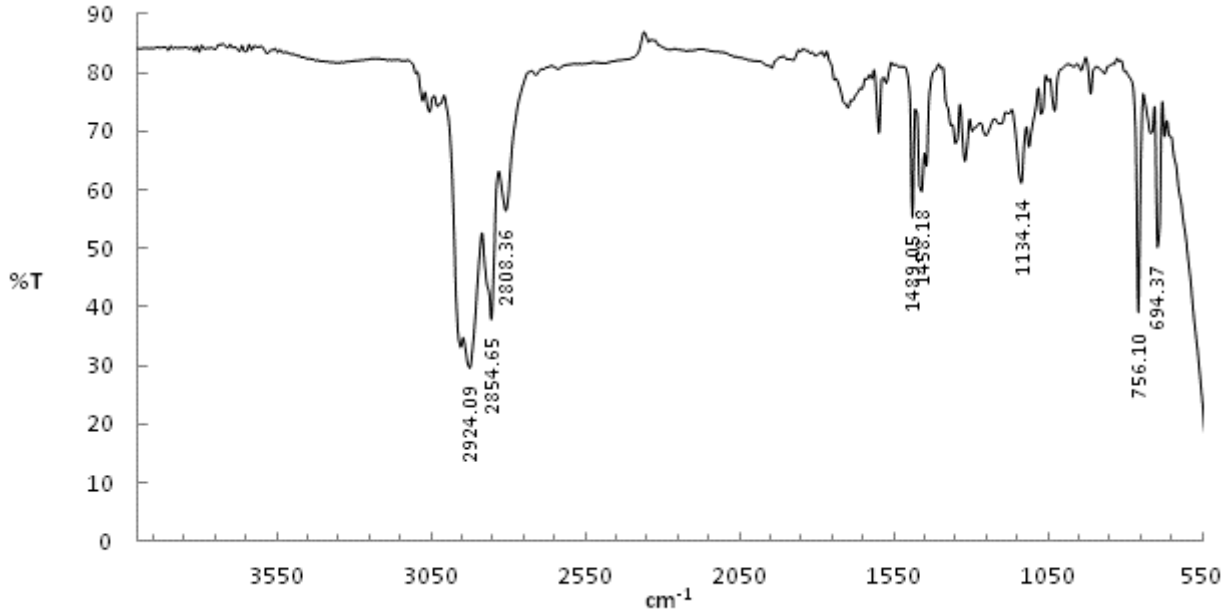
Compound 9: FTIR (NaCl plates, neat)



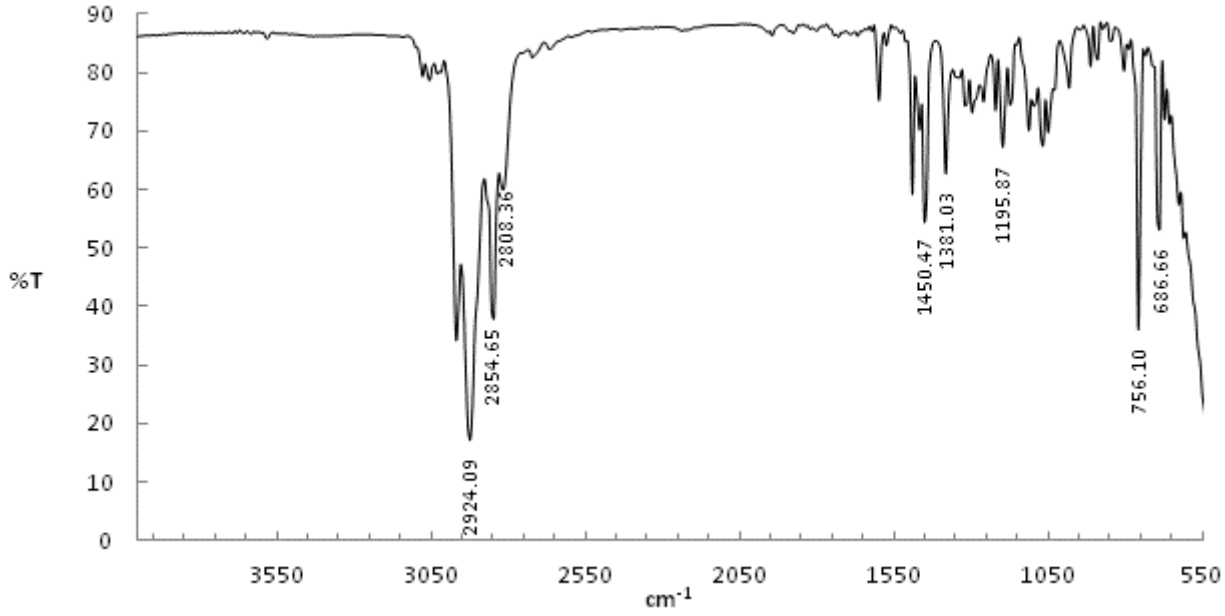
Compound 10: FTIR (NaCl plates, neat)



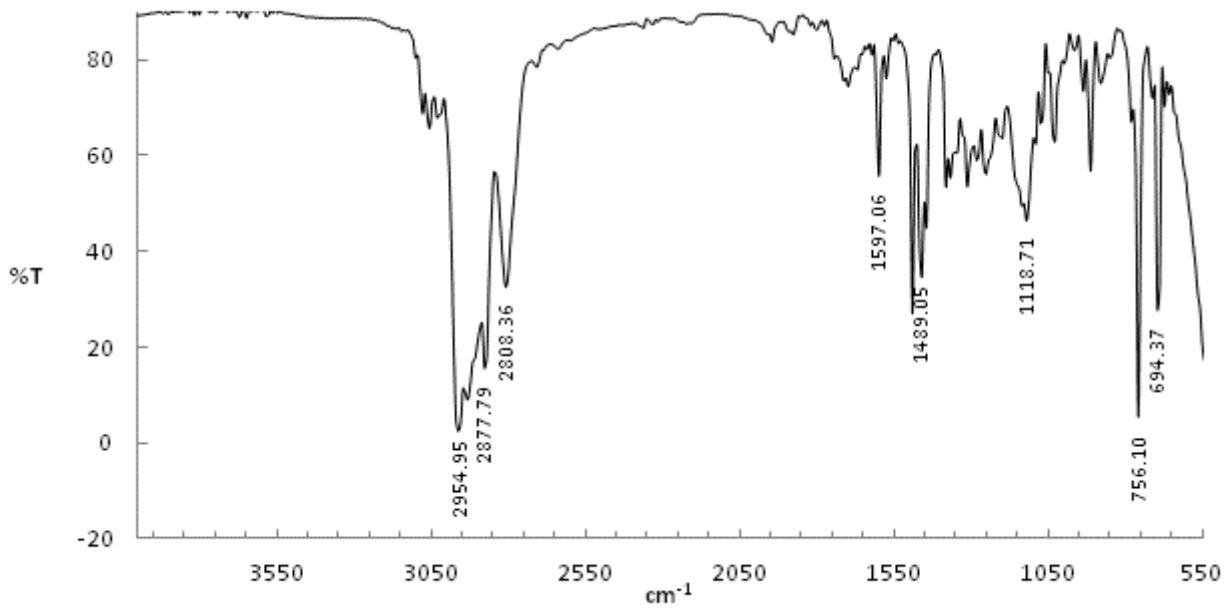
Compound 11: FTIR (NaCl plates, neat)



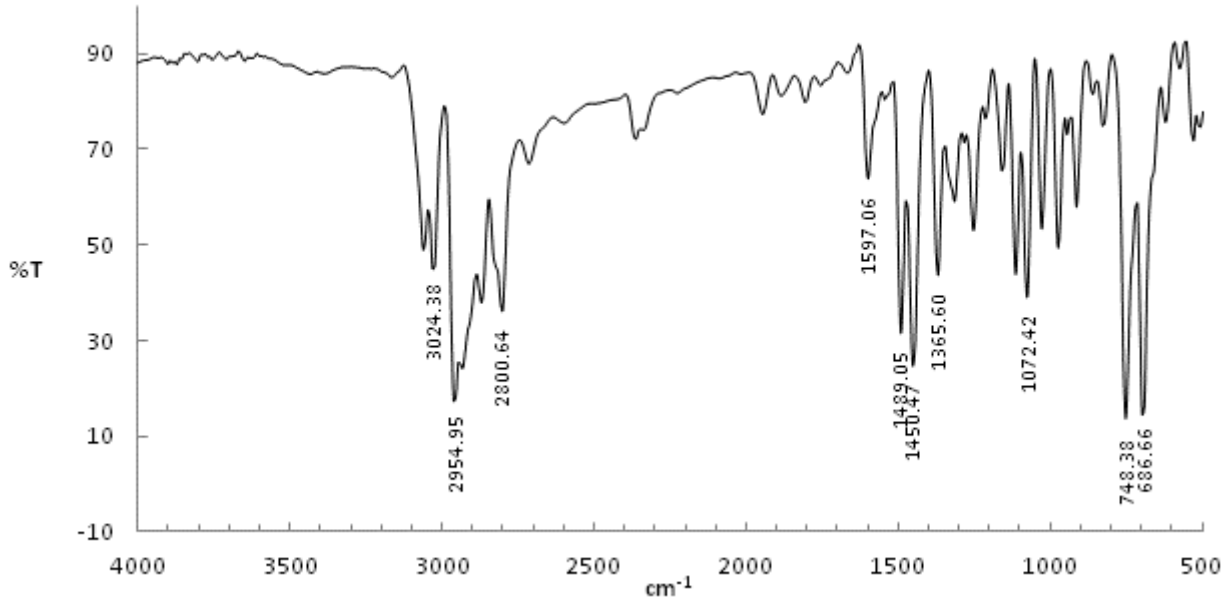
Compound 12: FTIR (NaCl plates, neat)



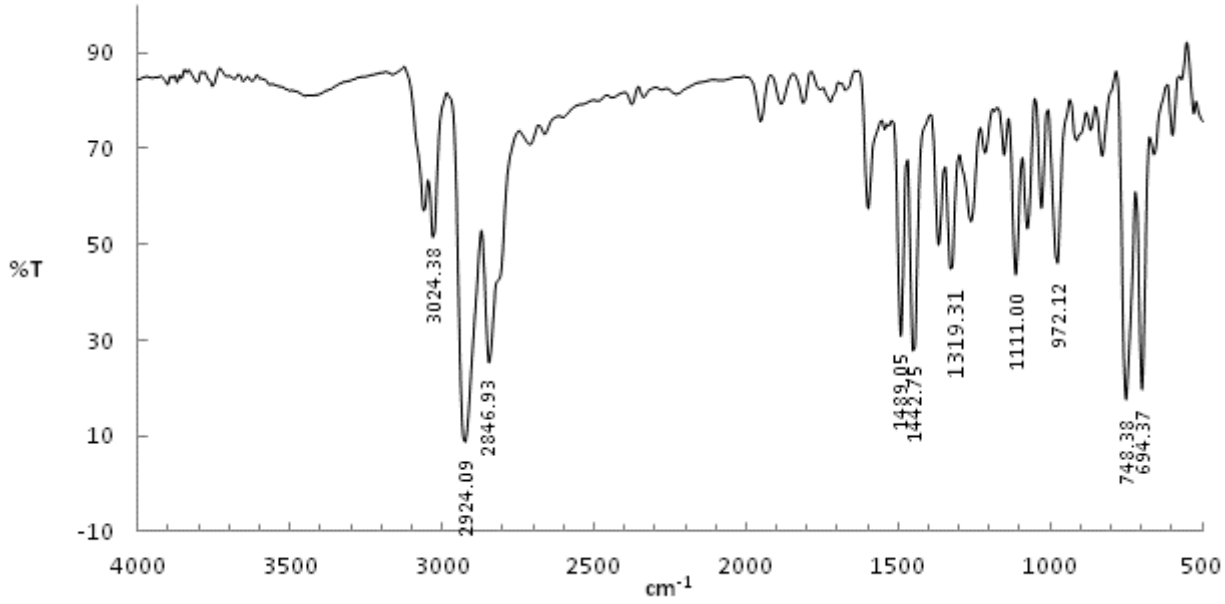
Compound 13: FTIR (NaCl plates, neat)



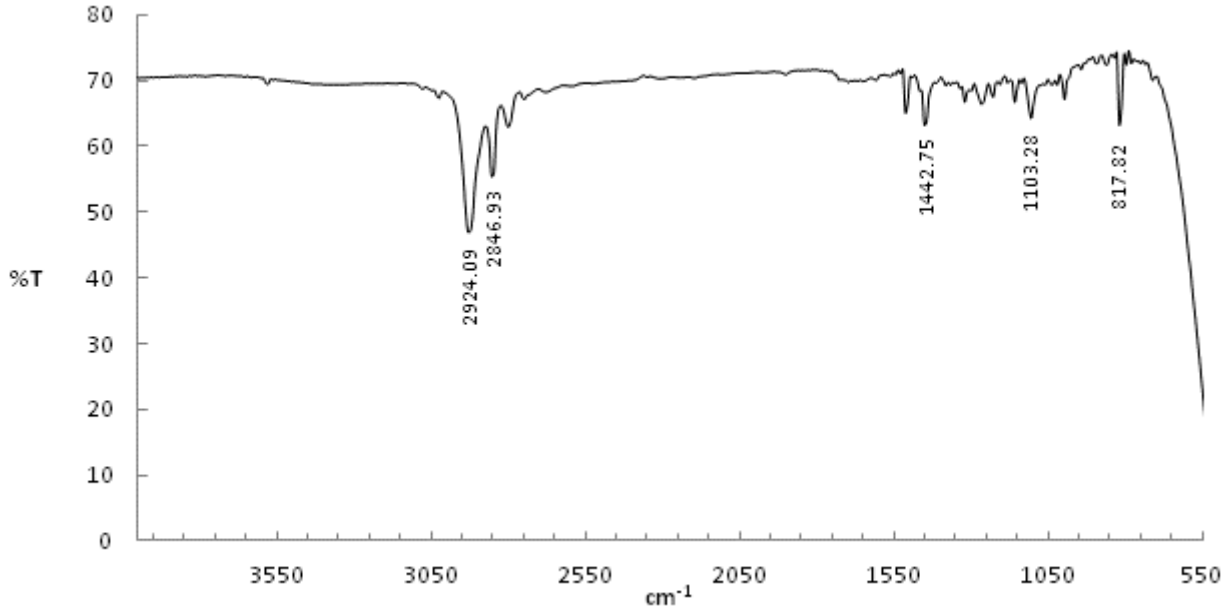
Compound 14: FTIR (KBr pellet)



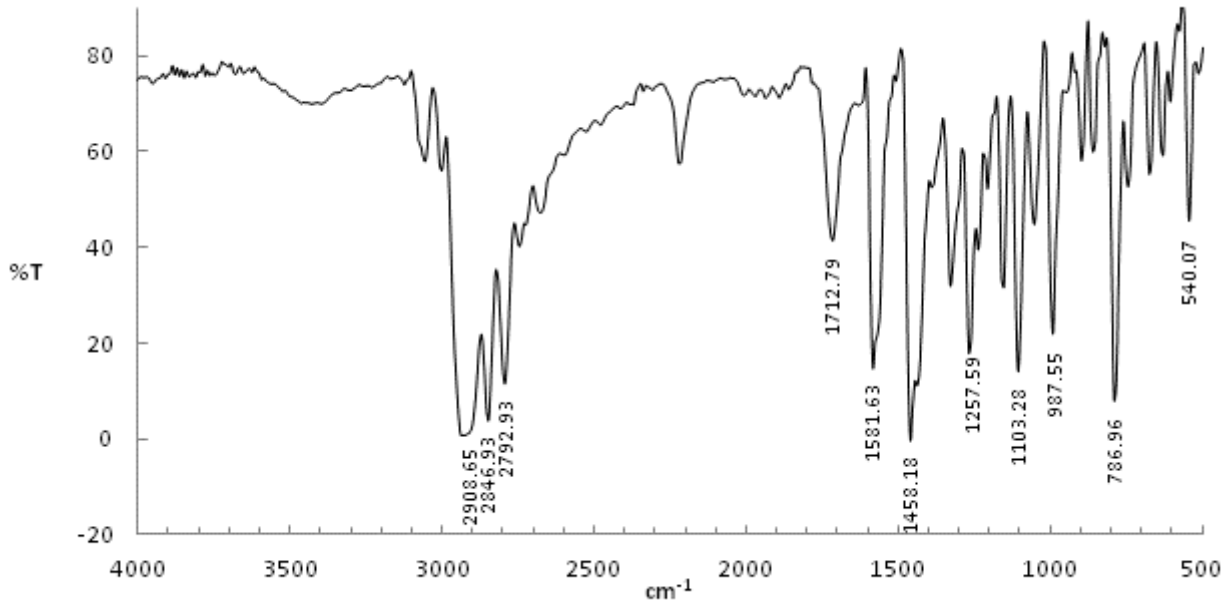
Compound 15: FTIR (KBr pellet)



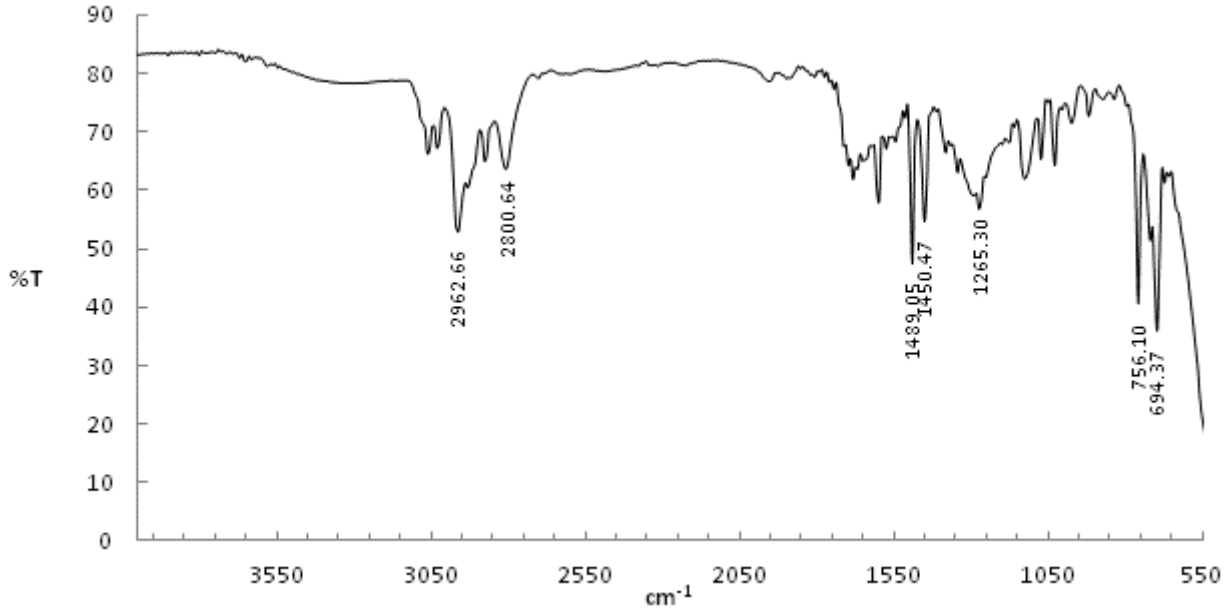
Compound 16: FTIR (NaCl plates, neat)



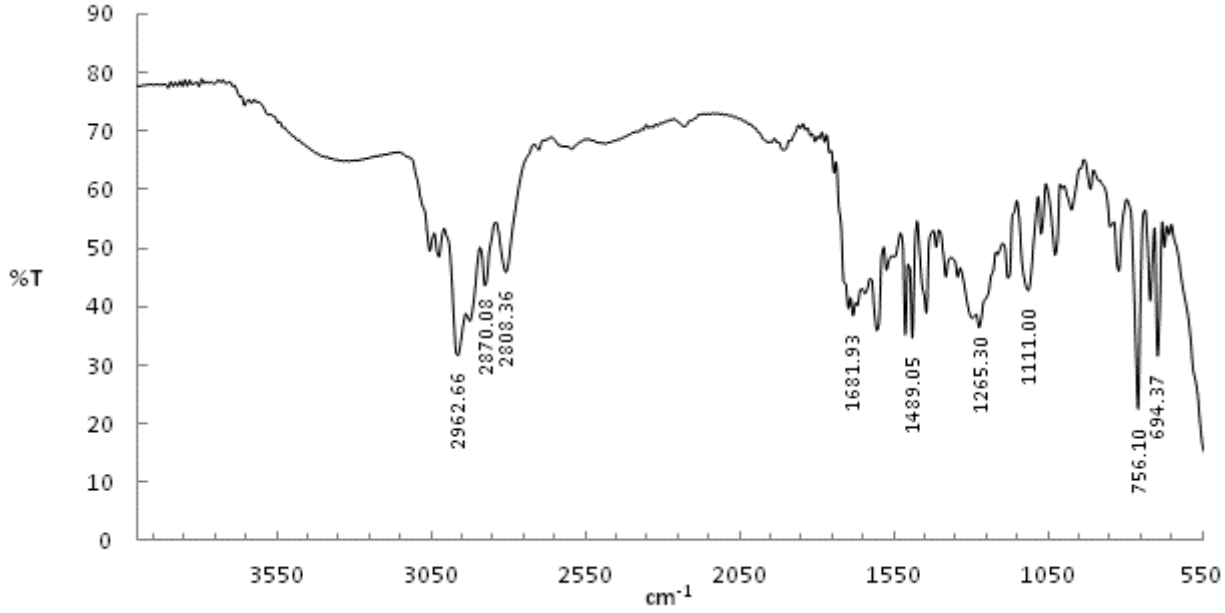
Compound 17: FTIR (KBr pellet)



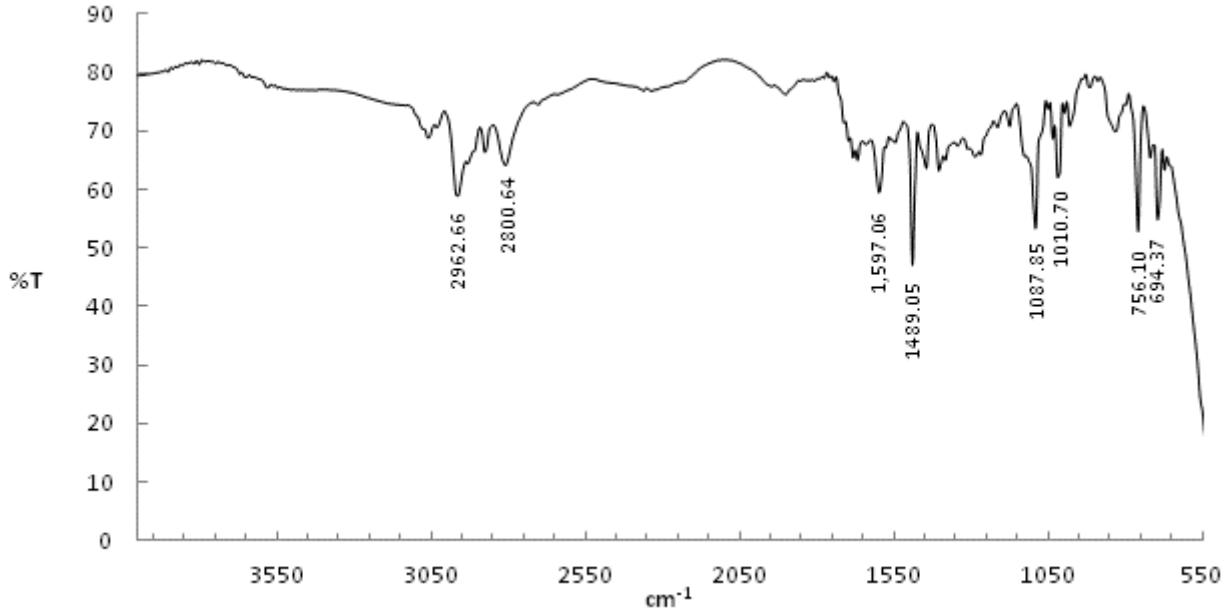
Compound 18: FTIR (NaCl plates, neat)



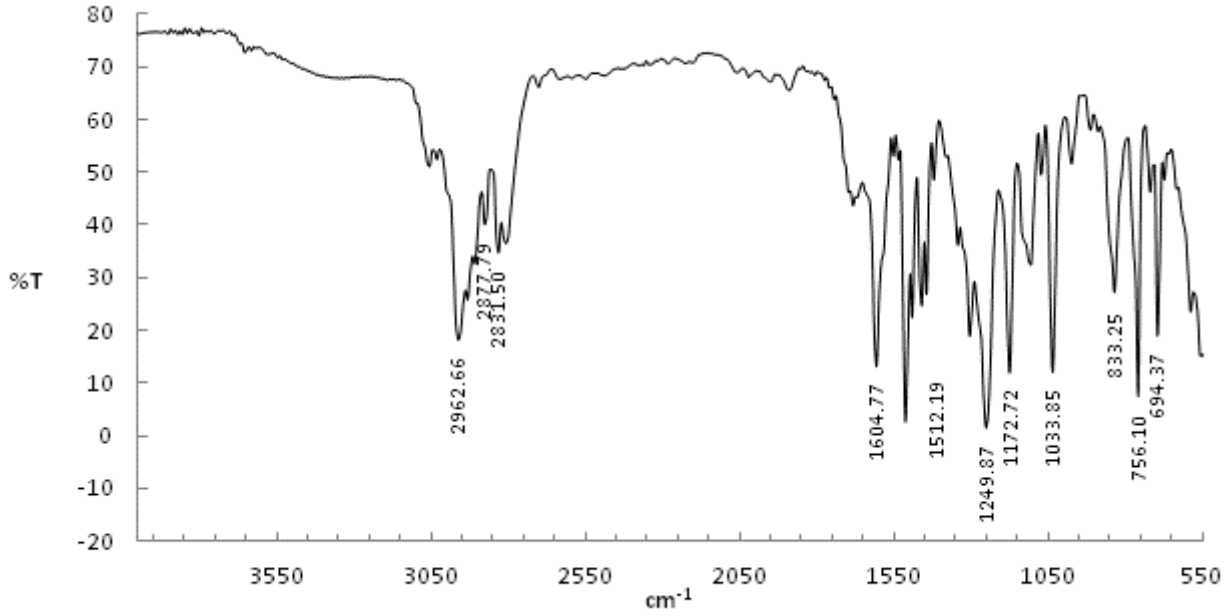
Compound 19: FTIR (NaCl plates, neat)



Compound 20: FTIR (NaCl plates, neat)



Compound 21: FTIR (NaCl plates, neat)



Compound 22: FTIR (NaCl plates, neat)