Supporting Information

Au-Catalyzed Rearrangement/Cyclization Cascade Toward the Synthesis of 2-Phenyl-1,4,5,6-tetrahydrocyclopenta[*b*]pyrrole

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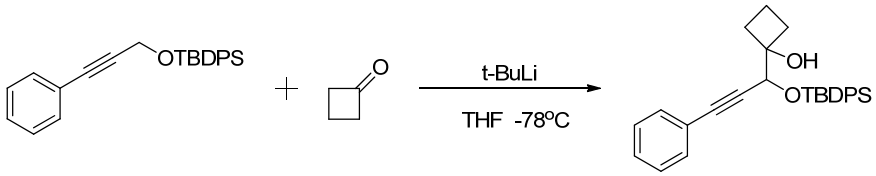
content

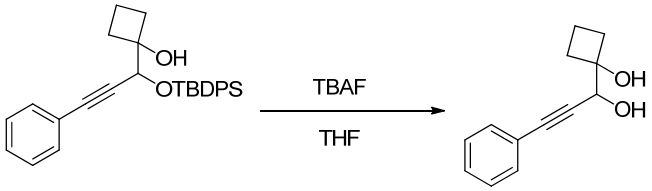
1. General Information:

All reactions under standard conditions were carried out under argon, dry atmosphere and monitored by thin-layer chromatography (TLC) on gel F254 plates. All products were purified through silica gel chromatography (200~300 mesh). Column chromatography was carried out with light petroleum ether (bp. 60~90 ^{o}C), ethyl acetate and dichloromethane as eluent. ^{1}H and ^{13}C spectra were recorded in $CDCl_3$ on 400 (300) MHz instruments and spectral data were reported in ppm. High-resolution mass spectral analysis (HRMS) data were measured on the Apex II by means of the ESI technique.

2. Preparation and characterization of Substrates

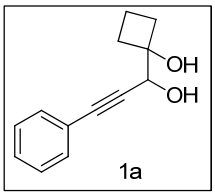


To a solution of tert-butyldiphenyl((3-phenylprop-2-yn-1-yl)oxy)silane (2.0 g, 5.41 mmol) in dry tetrahydrofuran was added tert-butyllithium(t-BuLi) (1.3 M in *n*-hexane) (8.3 mL, 10.81 mmol) at -78 °C under argon atmosphere. After the reaction mixture was stirred for 2 h at the same temperature, cyclobutanone (1.61 mL, 21.63 mmol) was added dropwise. The reaction mixture was stirred for another 2h. Upon completion of the reaction (monitored by TLC), the reaction mixture was quenched by slow addition of aqueous solution of saturated ammonium chloride solution . The aqueous layer was extracted three times with EtOAc and the combined organic layers were washed with brine, dried over sodium sulfate, and evaporated to dryness and purified by column chromatography to afford pure desired product (1.90 g, 80% yield).



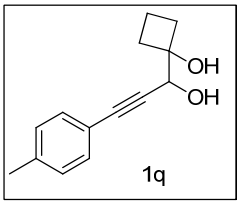
To a stirred solution of 1-(1-((tert-butyldiphenylsilyl)oxy)-3-phenylprop-2-yn-1-yl)cyclobutanol (1.90 g, 4.31 mmol) in dry tetrahydrofuran (30 mL) was added tetrabutylammonium fluoride (2.26 g, 8.64 mmol) at room temperature under argon

atmosphere. The reaction mixture was stirred for 1 h. Upon completion of the reaction (monitored by TLC), the reaction mixture was quenched by slow addition of water. The aqueous layer was extracted three times with EtOAc and the combined organic layers were washed with brine, dried over sodium sulfate, and evaporated to dryness and purified by column chromatography to afford pure desired product **1a** (0.77 g, 88% yield) as a colorless oil.



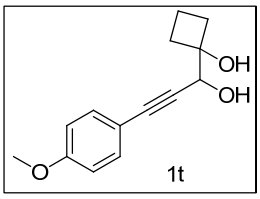
1-(1-hydroxy-3-phenylprop-2-yn-1-yl)cyclobutanol (**1a**)

^{1}H NMR (400 MHz, $CDCl_3$) δ 7.48–7.42 (m, 2H), 7.33–7.27 (m, 3H), 4.57 (s, 1H), 2.81 (s, 2H), 2.40–2.24 (m, 2H), 2.20–2.06 (m, 2H), 1.92–1.80 (m, 1H), 1.68 (m, 1H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 131.80, 128.58, 128.27, 122.20, 86.71, 85.61, 68.09, 32.07, 31.69, 11.98; HRMS (ESI) calcd for $C_{13}H_{14}O_2$ $[M+NH_4]^+$: 220.1332, found 220.1329.



1-(1-hydroxy-3-(p-tolyl)prop-2-yn-1-yl)cyclobutanol (**1q**)

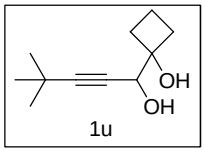
1q was obtained through the similar procedure as **1a** in a total yield of 70% as a colorless oil. ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.34 (d, *J* = 8.1 Hz, 2H), 7.08 (d, *J* = 8.0 Hz, 2H), 4.57 (s, 1H), 3.38 (s, 2H), 2.39–2.24 (m, 5H), 2.20–2.06 (m, 2H), 1.90–1.75 (m, 1H), 1.73–1.54 (m, 1H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 138.73, 131.80, 129.09, 119.28, 86.24, 85.72, 76.78, 68.14, 32.06, 31.85, 21.52, 12.15; HRMS (ESI) calcd for $C_{14}H_{16}O_2$ $[M+NH_4]^+$: 234.1489, found 234.1486.



1-(1-hydroxy-3-(4-methoxyphenyl)prop-2-yn-1-yl)cyclobutanol (**1t**)

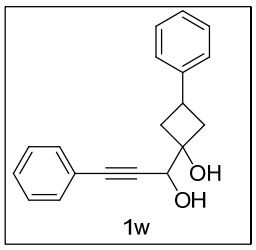
1t was obtained through the similar procedure as **1a** in a total yield of 50% as a colorless oil. ^{1}H NMR (300 MHz, $CDCl_3$) δ 7.50–7.33 (m, 2H), 6.90–6.75 (m, 2H), 4.55 (s, 1H), 3.80 (s, 3H), 2.71 (s, 2H), 2.39–2.24 (m, 2H), 2.21–2.04 (m, 2H), 1.93–1.80 (m,

1H), 1.73–1.61 (m, 1H); ^{13}C NMR (75 MHz, $CDCl_3$) δ 159.77, 133.28, 114.20, 113.90, 85.61, 85.20, 68.16, 55.26, 32.13, 31.61, 11.96; HRMS (ESI) calcd for $C_{14}H_{16}O_3$ $[M+H]^+$: 233.1172, found 233.1169.



1-(1-hydroxy-4,4-dimethylpent-2-yn-1-yl)cyclobutanol (**1u**)

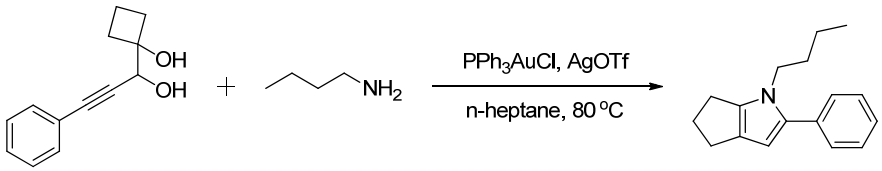
1u was obtained through the similar procedure as **1a** in a total yield of 72% as a colorless oil. ^{1}H NMR (300 MHz, $CDCl_3$) δ 4.26 (s, 1H), 3.17 (s, 2H), 2.33–2.12 (m, 2H), 2.11–1.90 (m, 2H), 1.86–1.69 (m, 1H), 1.67–1.52 (m, 1H), 1.19 (s, 9H); ^{13}C NMR (75 MHz, $CDCl_3$) δ 94.25, 76.47, 76.11, 67.51, 31.56, 31.36, 30.81, 27.27, 11.95; HRMS (ESI) calcd for $C_{11}H_{18}O_2$ $[M+NH_4]^+$: 200.1645, found 200.1642.



1-(1-hydroxy-3-phenylprop-2-yn-1-yl)-3-phenylcyclobutanol (**1w**)

1w was obtained through the similar procedure as **1a** in a total yield of 45% as a colorless oil. ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.49–7.46 (m, 2H), 7.35–7.18 (m, 8H), 4.70 (s, 1H), 3.29–3.18 (m, 1H), 2.86–2.83 (m, 2H), 2.65-2.55 (m, 2H), 2.35–2.26 (m, 2H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 144.92, 131.80, 128.64, 128.31, 126.56, 126.01, 122.08, 86.62, 85.80, 72.74, 68.09, 40.15, 39.65, 29.98; HRMS (ESI) calcd for $C_{19}H_{18}O_2$ $[M+NH_4]^+$: 296.1645, found 296.1642.

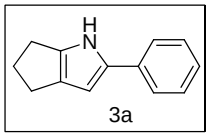
3. General experimental procedure for products 3a-3w



To a stirred solution of 1-(1-hydroxy-3-phenylprop-2-yn-1-yl)cyclobutanol (100 mg, 0.5 mmol) in *n*-heptane (1 mL) was successively added chloro(triphenylphosphine)gold(I) (PPh_3AuCl) (12.4 mg, 0.025 mmol) and silver trifluoromethanesulfonate (AgOTf) (6.1 mg, 0.025 mmol) at 80 °C under argon atmosphere. Butan-1-amine (150 μL, 1.5 mmol) was added after 1h at the same

temperature. The reaction mixture was reflux for 6h. Upon completion of the reaction (monitored by TLC), the reaction mixture was quenched by slow addition of water. The aqueous layer was extracted three times with EtOAc and the combined organic layers were washed with brine, dried over sodium sulfate, and evaporated to dryness and purified by column chromatography to afford pure desired product **3d** (66 mg, 56%) as a pale brown oil.

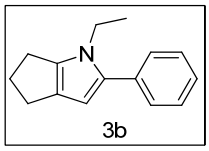
4. Characterization of products 3a-3w



3a

2-phenyl-1,4,5,6-tetrahydrocyclopenta[b]pyrrole (**3a**)[1]

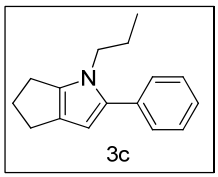
3a was obtained through the general procedure in 74% yield as a colorless oil. ^{1}H NMR (400 MHz, $CDCl_3$) δ 8.10 (s, 1H), 7.44–7.42 (m, 2H), 7.36–7.32 (m, 2H), 7.18–7.15 (m, 1H), 6.32 (d, *J* = 1.8 Hz, 1H), 2.77 (t, *J* = 7.1 Hz, 2H), 2.67 (t, *J* = 6.9 Hz, 2H), 2.49–2.41 (m, 2H); ^{13}C NMR (100 MHz, $CDCl_3$): δ 137.92, 135.17, 133.65, 128.77, 128.54, 125.48, 123.25, 101.64, 30.90, 29.03, 25.42.



3b

1-ethyl-2-phenyl-1,4,5,6-tetrahydrocyclopenta[b]pyrrole (**3b**)

3b was obtained through the general procedure in 51% yield as a colorless oil. ^{1}H NMR (400 MHz, CDCl3): δ 7.44–7.30 (m, 4H), 7.29–7.23 (m, 1H), 5.99 (s, 1H), 3.88 (q, *J* = 7.2 Hz, 2H), 2.77 (t, *J* = 7.1 Hz, 2H), 2.68 (t, *J* = 7.0 Hz, 2H), 2.49–2.39 (m, 2H), 1.27 (t, *J* = 7.2 Hz, 3H); ^{13}C NMR (100 MHz, $CDCl_3$): δ 139.54, 137.00, 134.22, 128.55, 128.32, 126.35, 125.81, 103.51, 40.40, 28.76, 25.44, 25.09, 16.50; HRMS (ESI) calcd for C15H17N $[M+H]^+$:212.1434, found 212.1436.

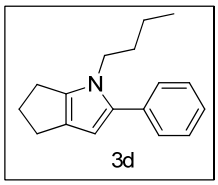


3c

2-phenyl-1-propyl-1,4,5,6-tetrahydrocyclopenta[b]pyrrole (**3c**)[2]

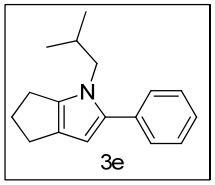
3c was obtained through the general procedure in 64% yield as a colorless oil. ^{1}H NMR (400 MHz, $CDCl_3$): δ 7.44–7.30 (m, 4H), 7.29–7.22 (m, 1H), 5.99 (s, 1H), 3.79 (t, *J* = 4Hz, 2H), 2.75 (t, *J* = 7.0 Hz, 2H), 2.67 (t, *J* = 6.9 Hz, 2H), 2.48–2.38 (m, 2H), 1.69–

1.55 (m, 2H), 0.80 (t, *J* = 7.4 Hz, 3H); ^{13}C NMR (100 MHz, $CDCl_3$): δ 139.89, 137.21, 134.40, 128.60, 128.30, 126.31, 125.56, 103.57, 47.47, 28.74, 25.49, 25.25, 24.44, 11.18;



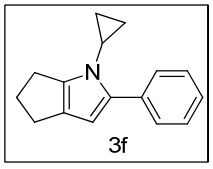
1-butyl-2-phenyl-1,4,5,6-tetrahydrocyclopenta[b]pyrrole (**3d**)

3d was obtained through the general procedure in 56% yield as a pale brown oil. ^{1}H NMR (400 MHz, $CDCl_3$):δ=7.43–7.30 (m, 4H), 7.28–7.24 (m, 1H), 5.98 (s, 1H), 3.83 (t, *J* = 8.0Hz, 2H), 2.75 (t, *J* = 7.0 Hz, 2H), 2.67 (t, *J* = 7.0 Hz, 2H), 2.48–2.38 (m, 2H), 1.61–1.57 (m, 2H), 1.23–1.20 (m, 2H), 0.82 (t, *J* = 7.4 Hz, 3H); ^{13}C NMR (100 MHz, $CDCl_3$): δ 139.82, 137.22, 134.38, 128.62, 128.29, 126.31, 125.60, 103.52, 45.54, 33.27, 28.75, 25.48, 25.26, 19.85, 13.63; HRMS (ESI) calcd for $C_{17}H_{21}N$ $[M+H]^+$:240.1747, found 240.1750.



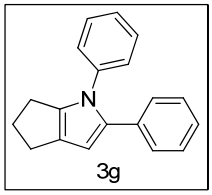
1-isobutyl-2-phenyl-1,4,5,6-tetrahydrocyclopenta[b]pyrrole (**3e**)

3e was obtained through the general procedure in 61% yield as a colorless oil. ^{1}H NMR (400 MHz, CDCl3): δ 7.41–7.28 (m, 4H), 7.28–7.22 (m, 1H), 5.98 (s, 1H), 3.68 (d, *J* = 7.6 Hz, 2H), 2.73 (t, *J* = 7.0 Hz, 2H), 2.67 (t, *J* = 7.0 Hz, 2H), 2.44–2.40 (m, 2H), 1.84–1.72 (m, 1H), 0.69 (d, *J* = 6.7 Hz, 6H); ^{13}C NMR (100 MHz, $CDCl_3$): δ 140.18, 137.38, 134.74, 128.73, 128.24, 126.24, 125.33, 103.87, 53.40, 29.80, 28.75, 25.58, 25.54, 19.94.; HRMS (ESI) calcd for $C_{17}H_{21}N$ $[M+H]^+$: 240.1747, found 240.1749.



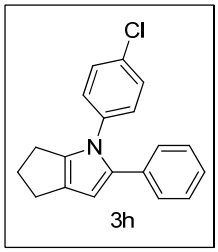
1-cyclopropyl-2-phenyl-1,4,5,6-tetrahydrocyclopenta[b]pyrrole (**3f**)

3f was obtained through the general procedure in 65% yield as a colorless oil. ^{1}H NMR (400 MHz, $CDCl_3$): δ 7.53–7.45 (m, 2H), 7.38–7.29 (m, 2H), 7.26–7.20 (m, 1H), 6.03 (s, 1H), 3.34–3.25 (m, 1H), 2.79 (t, *J* = 8Hz,2H), 2.64 (t, *J* = 7.0 Hz, 2H), 2.46–2.35 (m, 2H), 0.84–0.77 (m, 2H), 0.70–0.62 (m, 2H); ^{13}C NMR (100 MHz, $CDCl_3$): δ 141.22, 138.01, 134.33, 128.04, 127.93, 125.82, 125.39, 103.40, 28.70, 27.88, 25.96, 25.33, 8.31; HRMS (ESI) calcd for $C_{16}H_{17}N$ $[M+H]^+$: 224.1434, found 224.1436.



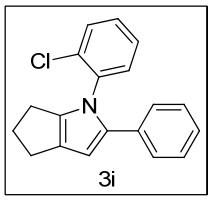
1,2-diphenyl-1,4,5,6-tetrahydrocyclopenta[b]pyrrole (**3g**)

3g was obtained through the general procedure in 58% yield as a colorless oil. ^{1}H NMR (400 MHz, $CDCl_3$): δ 7.31–7.23 (m, 2H), 7.25–7.01 (m, 8H), 6.25 (s, 1H), 2.75–2.70 (m, 4H), 2.48–2.32 (m, 2H); ^{13}C NMR (100 MHz, $CDCl_3$): δ 141.11, 139.88, 136.75, 133.69, 128.86, 127.99, 127.83, 127.02, 126.15, 125.91, 125.66, 105.68, 28.45, 25.99, 25.63; HRMS (ESI) calcd for $C_{19}H_{17}N$ $[M+H]^+$: 260.1434, found 260.1437.



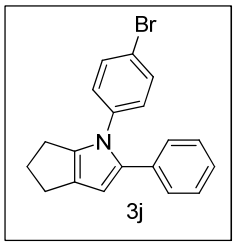
1-(4-chlorophenyl)-2-phenyl-1,4,5,6-tetrahydrocyclopenta[b]pyrrole (**3h**)

3h was obtained through the general procedure in 61% yield as a colorless oil. ^{1}H NMR (400 MHz, $CDCl_3$): δ 7.29–7.00 (m, 9H), 6.24 (s, 1H), 2.81–2.65 (m, 4H), 2.47–2.37 (m, 2H); ^{13}C NMR (100 MHz, $CDCl_3$): δ 140.91, 138.46, 136.79, 133.38, 131.76, 129.07, 128.16, 127.89, 127.44, 127.00, 125.93, 106.10, 28.44, 26.00, 25.60; HRMS (ESI) calcd for $C_{19}H_{16}ClN$ $[M+H]^+$: 294.1044, found 294.1048.



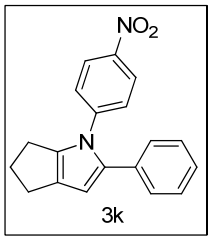
1-(2-chlorophenyl)-2-phenyl-1,4,5,6-tetrahydrocyclopenta[b]pyrrole (**3i**)

3i was obtained through the general procedure in 58% yield as a colorless oil. ^{1}H NMR (400 MHz, $CDCl_3$): δ 7.46–7.44 (m, 1H), 7.28–7.02 (m, 8H), 6.29 (s, 1H), 2.83–2.63 (m, 3H), 2.56–2.35 (m, 3H); ^{13}C NMR (100 MHz, $CDCl_3$): δ 141.91, 137.98, 137.89, 133.55, 132.26, 130.22, 130.06, 128.86, 128.76, 128.04, 127.83, 127.34, 127.10, 126.71, 125.91, 125.73, 104.81, 28.47, 25.71, 25.29; HRMS (ESI) calcd for $C_{19}H_{16}ClN$ $[M+H]^+$:294.1044, found 294.1047.



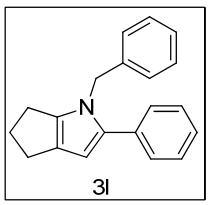
1-(4-bromophenyl)-2-phenyl-1,4,5,6-tetrahydrocyclopenta[b]pyrrole (**3j**)

3j was obtained through the general procedure in 57% yield as a colorless oil. ^{1}H NMR (400 MHz, $CDCl_3$): δ 7.43–7.38 (m, 2H), 7.24–7.16 (m, 2H), 7.14–7.08 (m, 3H), 7.01–6.95 (m, 2H), 6.24 (s, 1H), 2.74–2.60 (m, 4H), 2.49–2.37 (m, 2H); ^{13}C NMR (100 MHz, $CDCl_3$): δ 140.85, 138.95, 136.74, 133.35, 132.03, 128.17, 127.89, 127.51, 127.33, 125.94, 119.64, 106.19, 28.43, 26.02, 25.59; HRMS (ESI) calcd for $C_{19}H_{16}BrN$ $[M+H]^+$: 338.0539, found 338.0541.



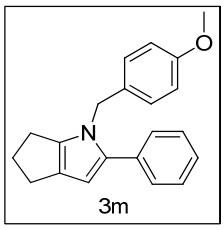
1-(4-nitrophenyl)-2-phenyl-1,4,5,6-tetrahydrocyclopenta[b]pyrrole (**3k**)

3k was obtained through the general procedure in 60% yield as a colorless oil. ^{1}H NMR (400 MHz, $CDCl_3$): δ 8.22–8.08 (m, 2H), 7.26–7.14 (m, 5H), 7.12–7.05 (m, 2H), 6.28 (s, 1H), 2.78 (t, *J* = 7.0 Hz, 2H), 2.73 (t, *J* = 7.0 Hz, 2H), 2.51–2.41 (m, 2H); ^{13}C NMR (100 MHz, CDCl3): δ 145.31, 145.07, 140.61, 137.02, 132.99, 128.93, 128.42, 128.00, 126.47, 125.54, 124.54, 107.92, 28.40, 26.48, 25.49; HRMS (ESI) calcd for $C_{19}H_{16}N_2O_2$ $[M+H]^+$: 305.1285, found 305.1287.



1-benzyl-2-phenyl-1,4,5,6-tetrahydrocyclopenta[b]pyrrole (**3l**)

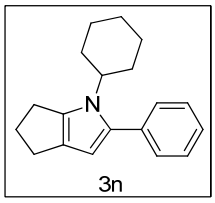
3l was obtained through the general procedure in 62% yield as a colorless oil. ^{1}H NMR (400 MHz, $CDCl_3$) :δ =7.43–7.25 (m, 6H), 7.25–7.13 (m, 2H), 7.00 (d, *J* = 7.1 Hz, 2H), 6.10 (s, 1H), 5.06 (s, 2H), 2.69 (t, *J* = 6.9 Hz, 2H), 2.52 (t, *J* = 6.9 Hz, 2H), 2.40–2.34 (m, 2H); ^{13}C NMR (100 MHz, $CDCl_3$): δ 140.40, 138.84, 137.85, 133.89, 128.60, 128.46, 128.34, 127.04, 126.43, 126.21, 126.13, 103.85, 49.10, 28.68, 25.61, 25.06; HRMS (ESI) calcd for $C_{20}H_{19}N$ $[M+H]^+$: 274.1590, found 274.1593.



3m

1-(4-methoxybenzyl)-2-phenyl-1,4,5,6-tetrahydrocyclopenta[b]pyrrole (**3m**)

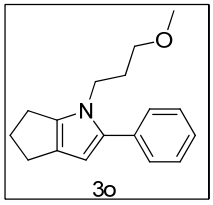
3m was obtained through the general procedure in 54% yield as a colorless oil. ^{1}H NMR (400 MHz, $CDCl_3$): δ 7.36–7.26 (m, 4H), 7.26–7.18 (m, 1H), 6.92 (d, *J* = 8.7 Hz, 2H), 6.86–6.77 (m, 2H), 6.08 (s, 1H), 5.00 (s, 2H), 3.76 (s, 3H), 2.68 (t, *J* = 6.9 Hz, 2H), 2.52 (t, *J* = 7.0 Hz, 2H), 2.42–2.31 (m, 2H); ^{13}C NMR (100 MHz, $CDCl_3$): δ 158.63, 140.33, 137.74, 133.96, 130.85, 128.49, 128.33, 127.50, 126.40, 126.07, 113.95, 103.79, 55.19, 48.59, 28.68, 25.57, 25.12; HRMS (ESI) calcd for $C_{21}H_{21}NO$ $[M+H]^+$: 304.1696, found 304.1697.



3n

1-cyclohexyl-2-phenyl-1,4,5,6-tetrahydrocyclopenta[b]pyrrole (**3n**)

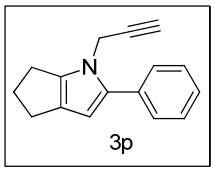
3n was obtained through the general procedure in 22% yield as a colorless oil. ^{1}H NMR (400 MHz, $CDCl_3$): δ 7.45–7.23 (m, 5H), 5.94 (s, 1H), 4.01–3.93 (m, 1H), 2.89 (t, *J* = 7.0 Hz, 2H), 2.63 (t, *J* = 7.0 Hz, 2H), 2.49–2.33 (m, 2H), 1.91 (d, *J* = 12.2 Hz, 2H), 1.85–1.68 (m, 4H), 1.32–1.17 (m, 4H); ^{13}C NMR (100 MHz, $CDCl_3$): δ 137.47, 134.53, 129.16, 128.21, 127.00, 126.38, 103.40, 55.28, 33.39, 29.15, 27.81, 25.94, 25.45, 24.76.; HRMS (ESI) calcd for $C_{19}H_{23}N$ $[M+H]^+$: 266.1903, found 266.1906.



3o

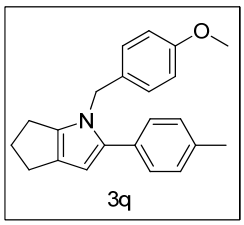
1-(3-methoxypropyl)-2-phenyl-1,4,5,6-tetrahydrocyclopenta[b]pyrrole (**3o**)

3o was obtained through the general procedure in 43% yield as a colorless oil. ^{1}H NMR (400 MHz, $CDCl_3$):δ=7.42–7.32 (m, 4H), 7.30–7.20 (m, 1H), 6.00 (s, 1H), 3.98 (t, *J* = 8Hz, 2H), 3.23–3.13 (m, 5H), 2.75 (t, *J* = 7.1 Hz, 2H), 2.67 (t, *J* = 7.0 Hz, 2H), 2.46–2.40 (m, 2H), 1.86–1.72 (m, 2H); ^{13}C NMR (100 MHz, $CDCl_3$): δ 140.17, 134.30, 128.52, 128.38, 126.39, 125.68, 103.93, 99.98, 69.40, 58.45, 42.80, 31.11, 28.80, 25.56, 25.21; HRMS (ESI) calcd for $C_{17}H_{21}NO$ $[M+H]^+$: 226.1696, found 226.1699.



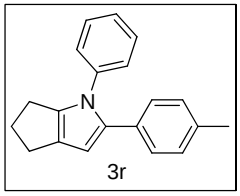
2-phenyl-1-(prop-2-yn-1-yl)-1,4,5,6-tetrahydrocyclopenta[b]pyrrole (**3p**)

3p was obtained through the general procedure in 57% yield as a colorless oil. ^{1}H NMR (400 MHz, $CDCl_3$): δ 7.47–7.43 (m, 2H), 7.41–7.37 (m, 2H), 7.30–7.24 (m, 1H), 6.04 (s, 1H), 4.55 (d, *J* = 2.5 Hz, 2H), 2.83 (t, *J* = 7.1 Hz, 2H), 2.68 (t, *J* = 7.0 Hz, 2H), 2.51–2.39 (m, 2H), 2.36 (t, *J* = 2.5 Hz, 1H); ^{13}C NMR (100 MHz, $CDCl_3$): δ 140.13, 137.23, 133.36, 128.53, 128.41, 126.61, 126.50, 104.06, 79.29, 72.61, 35.23, 28.66, 25.56, 24.91; HRMS (ESI) calcd for $C_{16}H_{15}N$ $[M+H]^+$: 222.1277, found 222.1281.



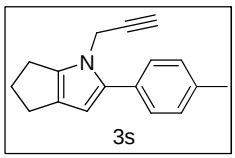
1-(4-methoxybenzyl)-2-(p-tolyl)-1,4,5,6-tetrahydrocyclopenta[b]pyrrole (**3q**)

3q was obtained through the general procedure in 52% yield as a colorless oil. ^{1}H NMR (400 MHz, $CDCl_3$): δ 7.23–7.20 (m, 2H), 7.11 (d, *J* = 7.9 Hz, 2H), 6.92 (d, *J* = 8.8 Hz, 2H), 6.87–6.75 (m, 2H), 6.04 (s, 1H), 4.98 (s, 2H), 3.77 (s, 3H), 2.67 (t, *J* = 6.9 Hz, 2H), 2.51 (t, *J* = 6.9 Hz, 2H), 2.43–2.25 (m, 5H); ^{13}C NMR (100 MHz, $CDCl_3$): δ 137.52, 134.56, 133.85, 133.66, 129.20, 128.76, 128.55, 128.48, 128.25, 127.04, 126.42, 103.44, 55.31, 33.41, 29.18, 27.83, 25.96, 25.48, 24.78; HRMS (ESI) calcd for $C_{22}H_{23}NO$ $[M+H]^+$: 318.1852, found 318.1855.



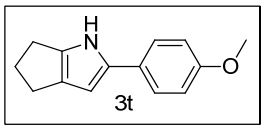
1-phenyl-2-(p-tolyl)-1,4,5,6-tetrahydrocyclopenta[b]pyrrole (**3r**)

3r was obtained through the general procedure in 43% yield as a colorless oil. ^{1}H NMR (400 MHz, $CDCl_3$): δ 7.29 (t, *J* = 7.5 Hz, 2H), 7.25–7.17 (m, 1H), 7.12 (d, *J* = 7.3 Hz, 2H), 7.04–6.89 (m, 4H), 6.21 (s, 1H), 2.75–2.70 (m, 4H), 2.45–2.38 (m, 2H), 2.27 (s, 3H); ^{13}C NMR (100 MHz, $CDCl_3$): δ 140.72, 139.96, 136.85, 135.35, 130.88, 128.83, 128.73, 127.80, 126.90, 126.09, 125.94, 105.23, 28.46, 26.00, 25.65, 21.07.; HRMS (ESI) calcd for $C_{20}H_{19}N$ $[M+H]^+$: 274.1590, found 274.1591.



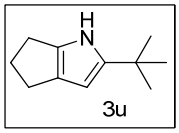
1-(prop-2-yn-1-yl)-2-(p-tolyl)-1,4,5,6-tetrahydrocyclopenta[b]pyrrole (**3s**)

3s was obtained through the general procedure in 49% yield as a colorless oil. ^{1}H NMR (400 MHz, $CDCl_3$): δ 7.34 (d, *J* = 8.1 Hz, 2H), 7.20 (d, *J* = 7.9 Hz, 2H), 6.01 (s, 1H), 4.54 (d, *J* = 2.5 Hz, 2H), 2.83 (t, *J* = 7.1 Hz, 2H), 2.67 (t, *J* = 7.0 Hz, 2H), 2.49–2.41 (m, 2H), 2.37 (s, 3H), 2.35 (t, *J* = 2.5 Hz, 1H); ^{13}C NMR (100 MHz, $CDCl_3$): δ 139.76, 137.31, 136.45, 130.57, 129.26, 128.48, 126.42, 103.74, 79.41,72.53, 35.20, 28.69, 25.62, 24.96, 21.18; HRMS (ESI) calcd for $C_{17}H_{17}N$ $[M+H]^+$: 236.1434, found 234.1437.



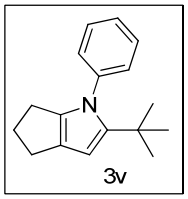
2-(4-methoxyphenyl)-1,4,5,6-tetrahydrocyclopenta[b]pyrrole (**3t**)

3t was obtained through the general procedure in 42% yield as a colorless oil. ^{1}H NMR (400 MHz, $CDCl_3$): δ 7.99 (s, 1H), 7.34 (d, *J* = 8.7 Hz, 2H), 6.88 (d, *J* = 8.8 Hz, 2H), 6.18 (d, *J* = 1.7 Hz, 1H), 3.81 (s, 3H), 2.74 (t, *J* = 7.1 Hz, 2H), 2.65 (t, *J* = 6.9 Hz, 2H), 2.48–2.33 (m, 2H); ^{13}C NMR (100 MHz, $CDCl_3$): δ 157.83, 137.09, 135.27, 128.34, 126.89, 124.78, 114.28, 100.63, 55.30, 29.04, 25.48, 25.43; HRMS (ESI) calcd for $C_{14}H_{15}NO$ $[M+H]^+$: 214.1226, found 214.1229.



2-(tert-butyl)-1,4,5,6-tetrahydrocyclopenta[b]pyrrole (**3u**)

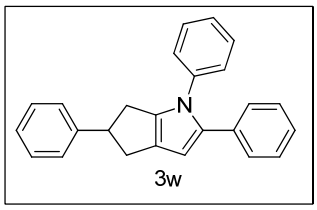
3u was obtained through the general procedure in 68% yield as a colorless oil. ^{1}H NMR (400 MHz, acetone-d_6): δ 9.13 (s, 1H), 5.41 (d, *J* = 1.5 Hz, 1H), 2.43 (t, *J* = 6.9 Hz, 2H), 2.34 (t, *J* = 6.7 Hz, 2H), 2.23–2.08 (m, 2H), 1.12 (s, 9H); ^{13}C NMR (100 MHz, acetone-d_6): δ 145.59, 134.96, 125.35, 98.39, 32.51, 31.15, 29.67, 26.29, 25.97; HRMS (ESI) calcd for $C_{11}H_{17}N$ $[M+H]^+$: 164.1434, found 164.1436.



2-(tert-butyl)-1-phenyl-1,4,5,6-tetrahydrocyclopenta[b]pyrrole (**3v**)

3v was obtained through the general procedure in 59% yield as a colorless oil. ^{1}H

NMR (400 MHz, $CDCl_3$): δ 7.47–7.27 (m, 5H), 5.90 (s, 1H), 2.67 (m, 2H), 2.41–2.26 (m, 4H), 1.15 (s, 9H); ^{13}C NMR (100 MHz, $CDCl_3$): δ 146.72, 142.03, 140.74, 129.22, 128.54, 127.91, 123.19, 100.24, 33.37, 31.55, 28.28, 25.99, 25.21; HRMS (ESI) calcd for $C_{17}H_{21}N$ $[M+H]^+$: 240.1747, found 240.1752.



3w

1,2,5-triphenyl-1,4,5,6-tetrahydrocyclopenta[b]pyrrole (**3w**)

3w was obtained through the general procedure in 35% yield as a colorless oil. ^1H NMR (400 MHz, $CDCl_3$):δ 7.37–7.12 (m, 15H), 6.28 (s, 1H), 4.11–4.00 (m, 1H), 3.24–3.13 (m, 2H), 2.94–2.86 (m, 2H); ^{13}C NMR (100 MHz, $CDCl_3$): δ 146.33, 139.78, 139.26, 136.88, 133.63, 128.95, 128.48, 128.08, 127.90, 127.10, 126.30, 126.17, 125.92, 125.83, 125.72, 105.73, 49.14, 35.21, 34.86; HRMS (ESI) calcd for $C_{25}H_{21}N$ $[M+H]^+$: 336.1747, found 336.1753.

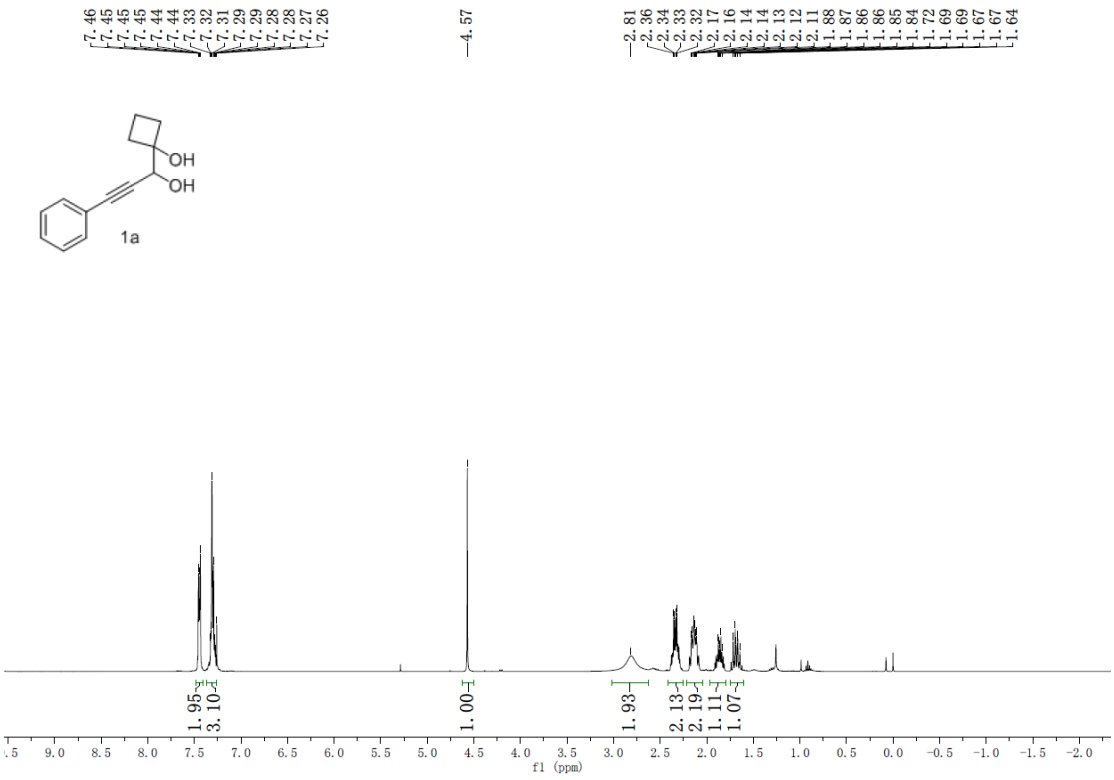
5. References

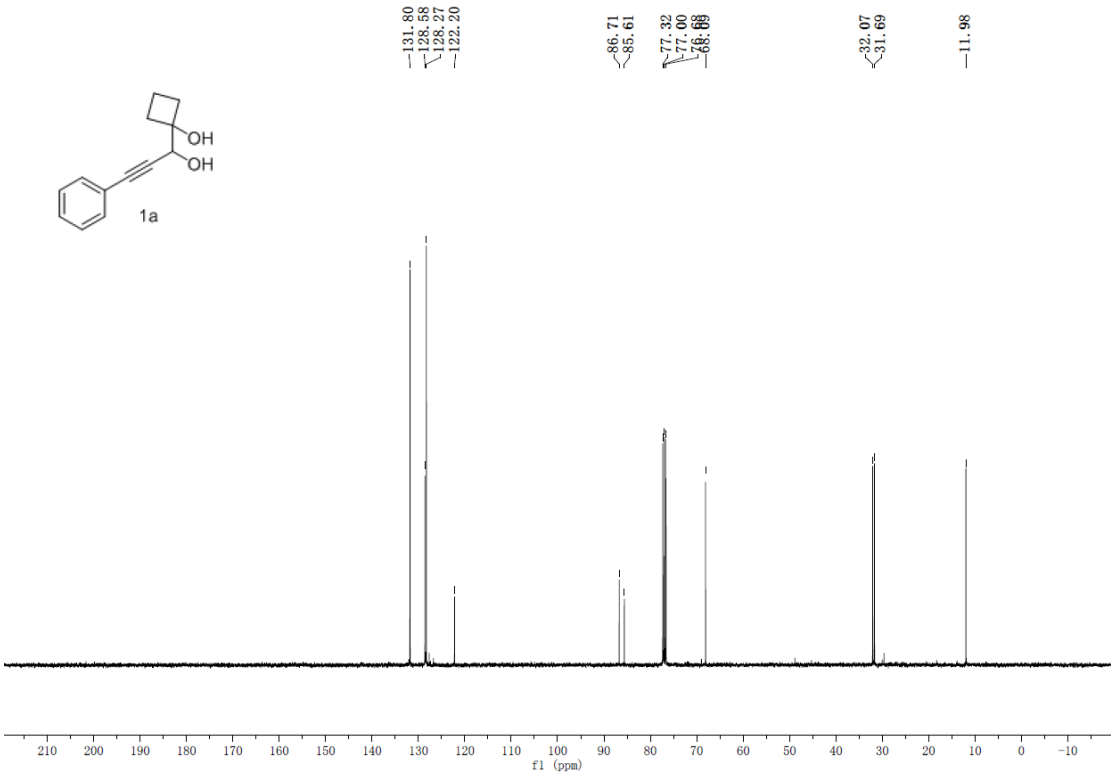
1. Thompson, B. B.; Montgomery, J. *Org. Lett.* 2011, **13**, 3289-3291.

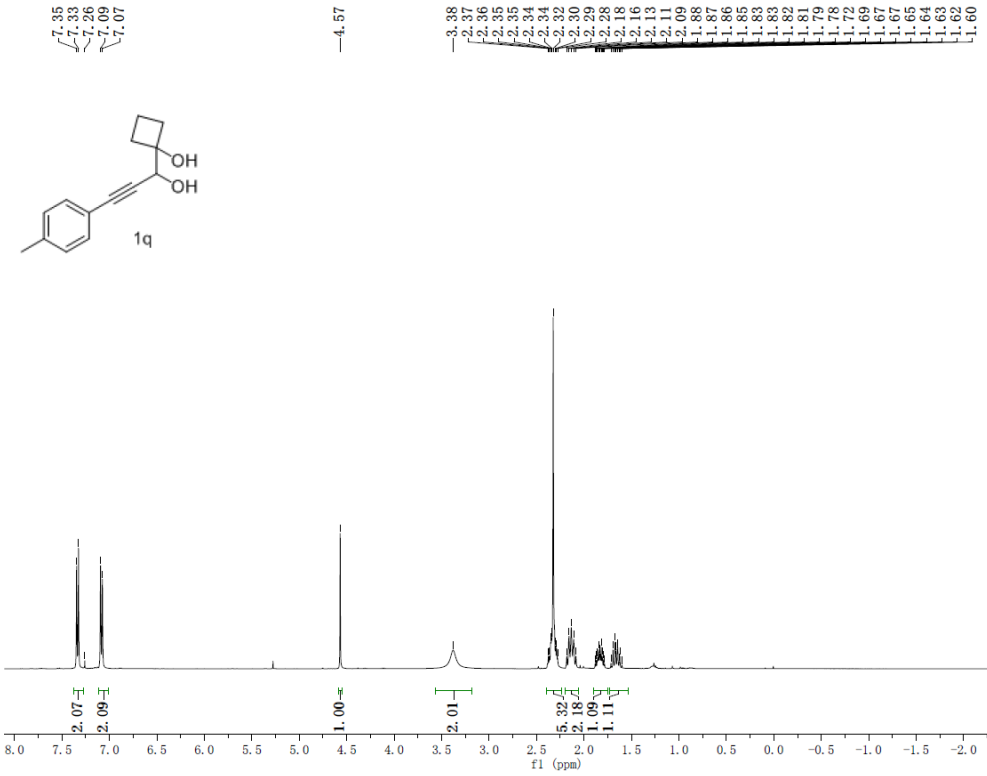
2. Gao, Yuan; Yoshida, Yukio; Sato, Fumie. *Synlett* 1997, 1353-1354.

3. Sun, H.; Yang, C.; Gao, F.; Li, Z.; Xia, W. *Org. Lett.* 2013, **15**, 624-627.

6. ^{1}H NMR and ^{13}C NMR spectra for Substrates and products



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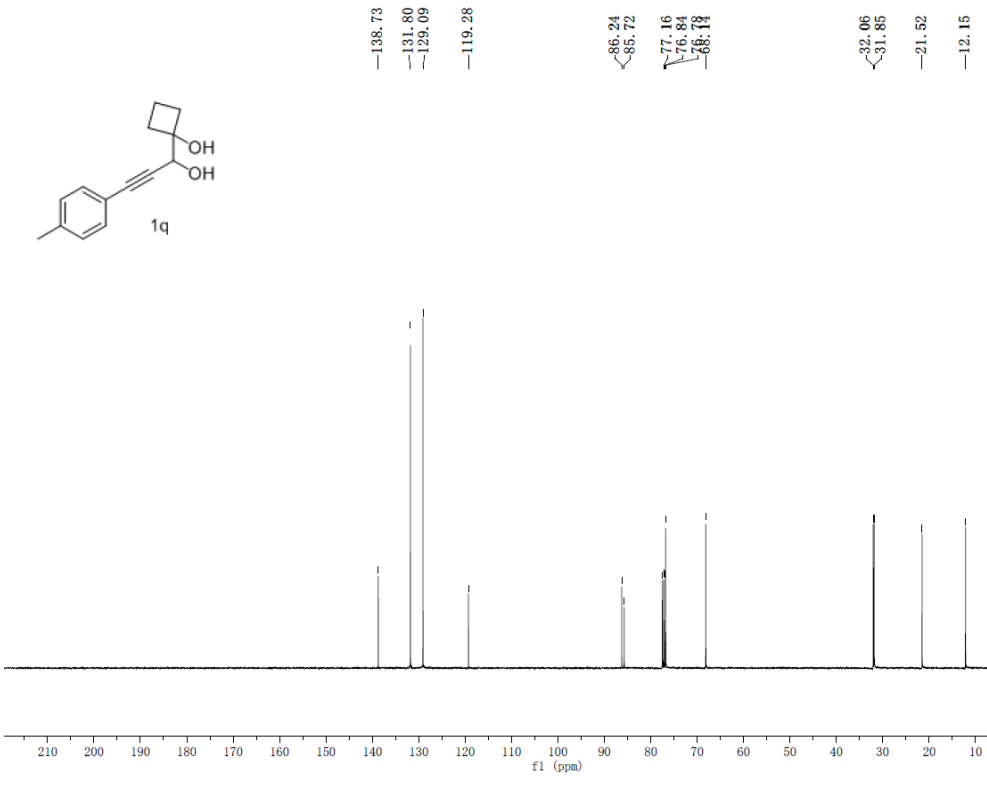
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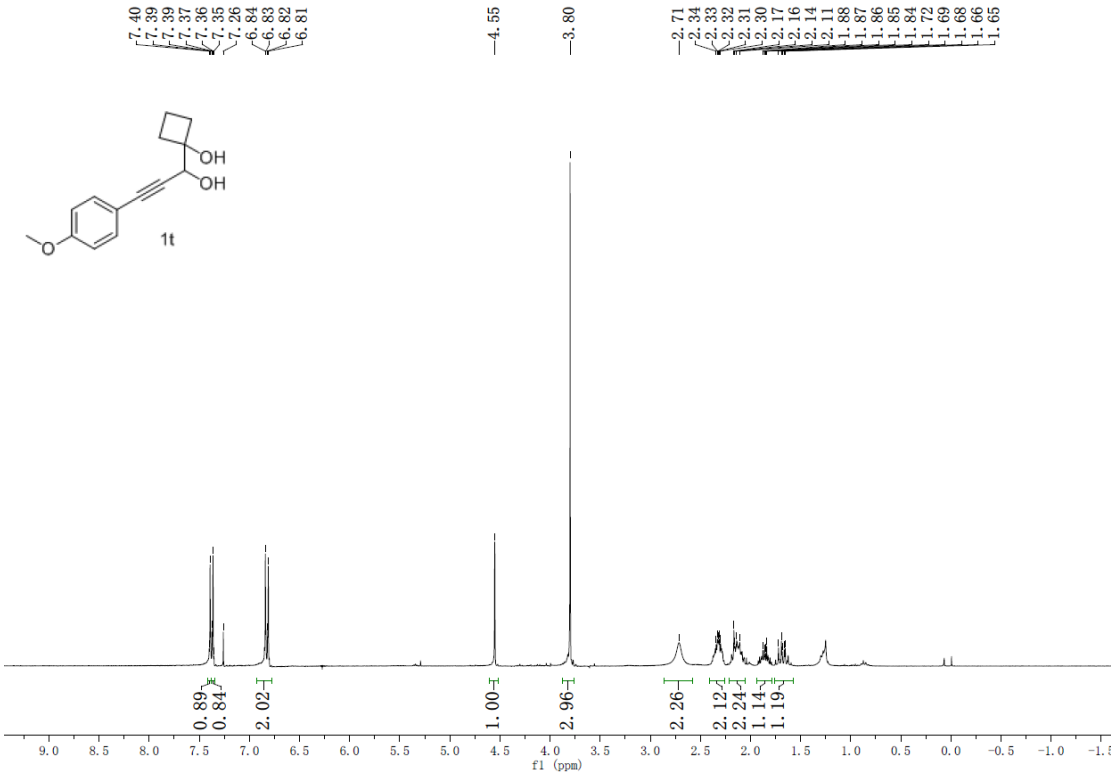
f1 (ppm)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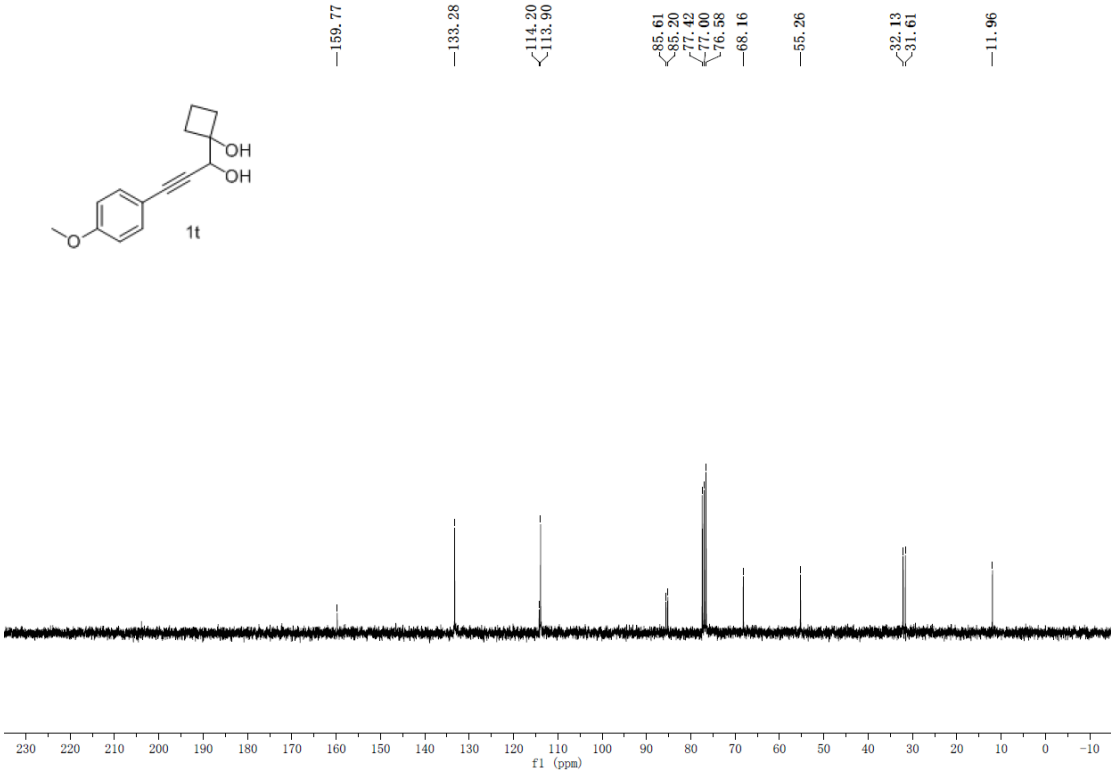
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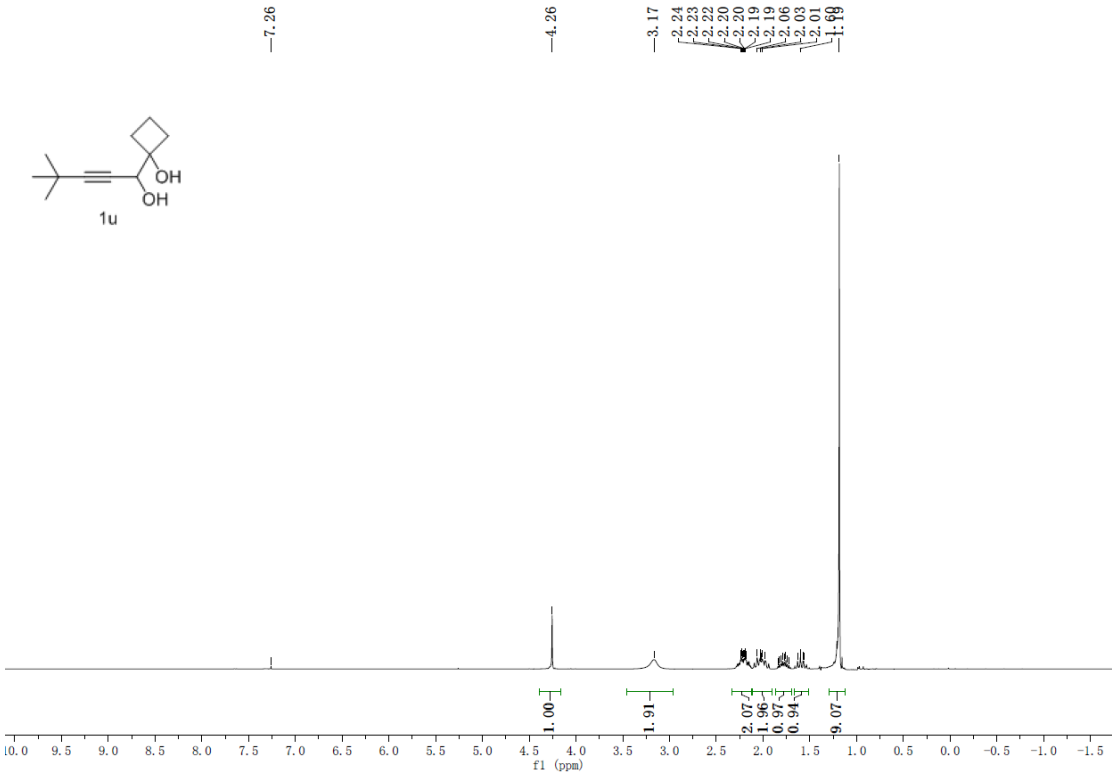
f1 (ppm)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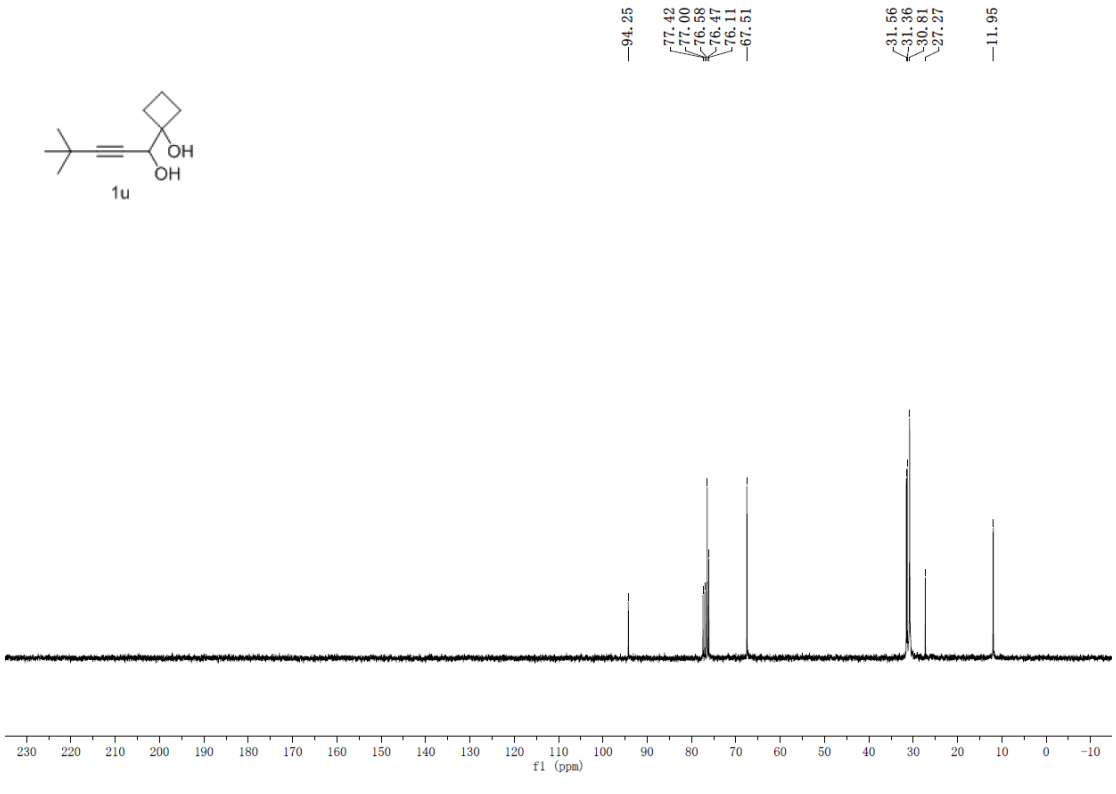
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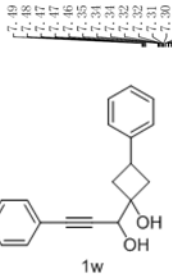

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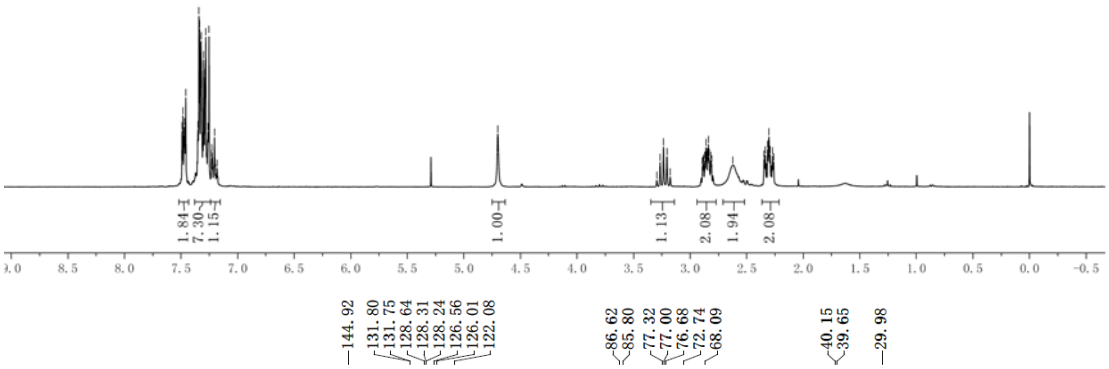

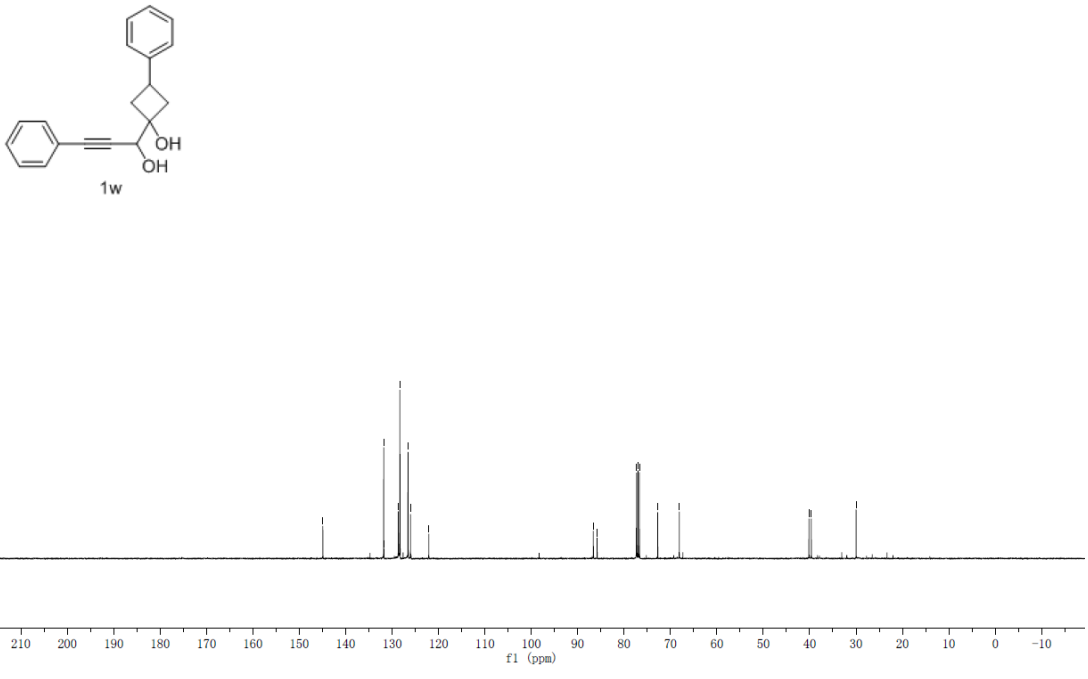

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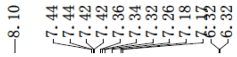
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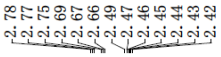
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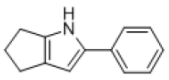
H
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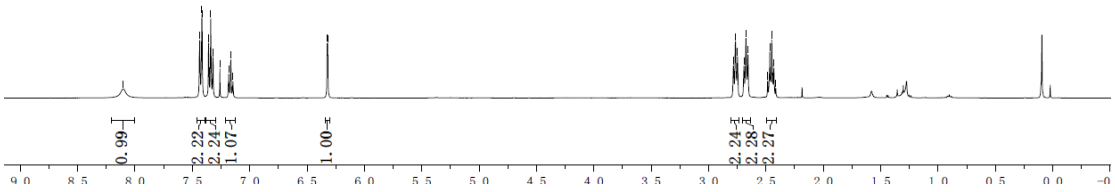
3a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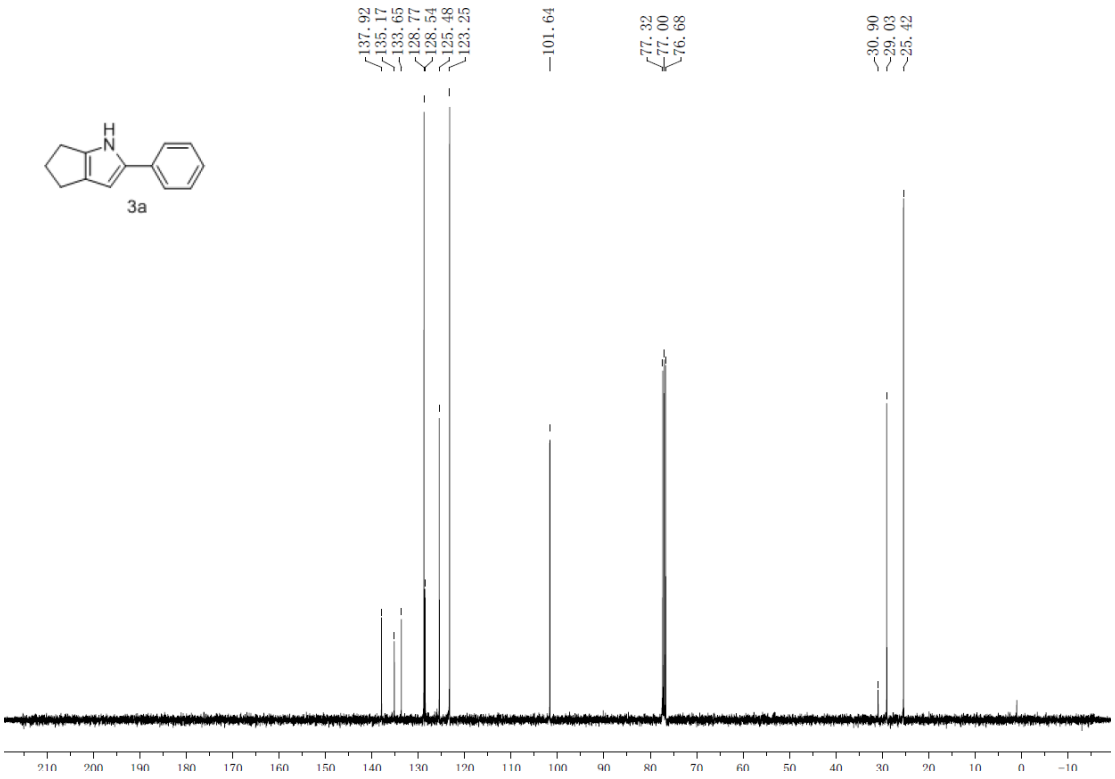
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H
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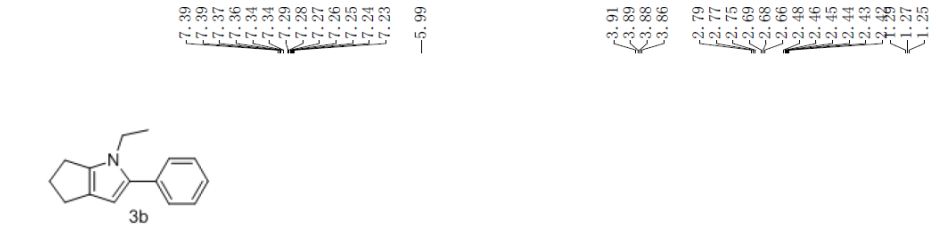
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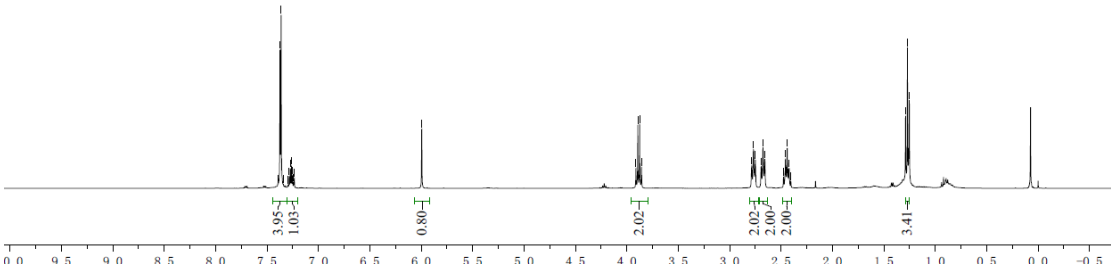
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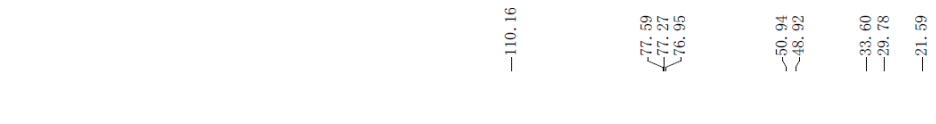
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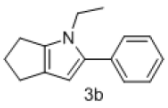
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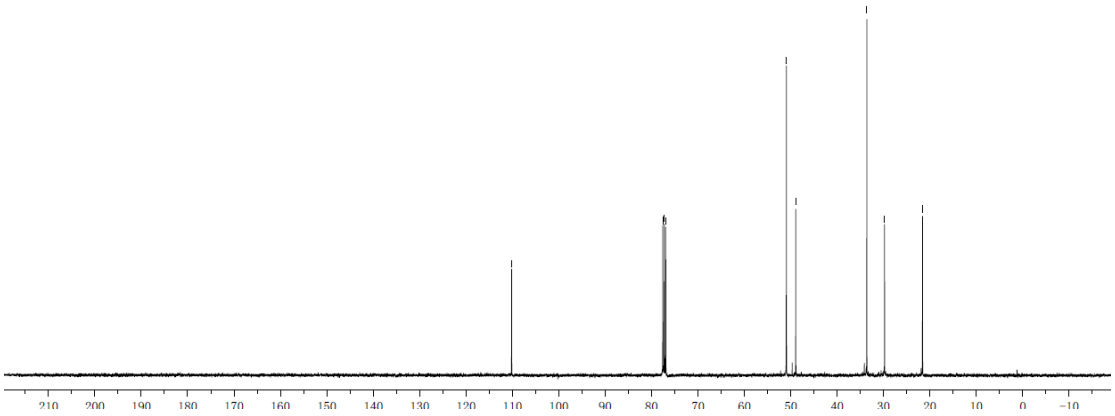


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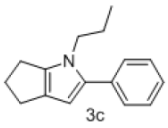
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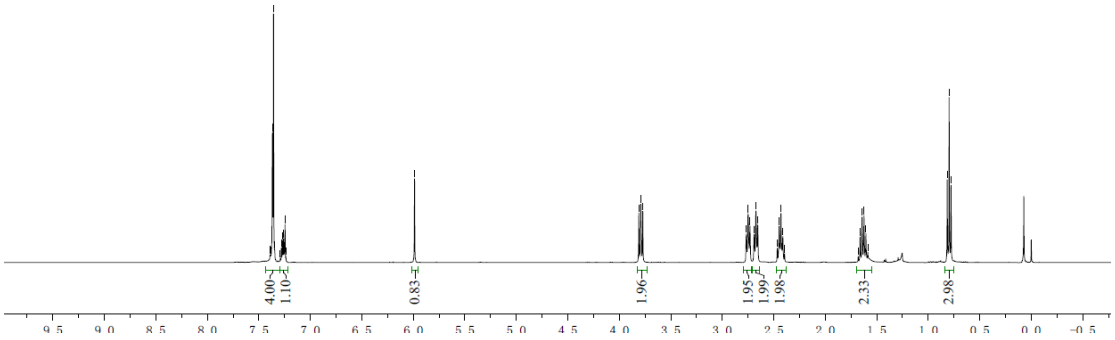
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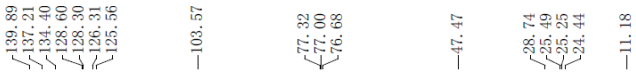
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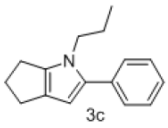
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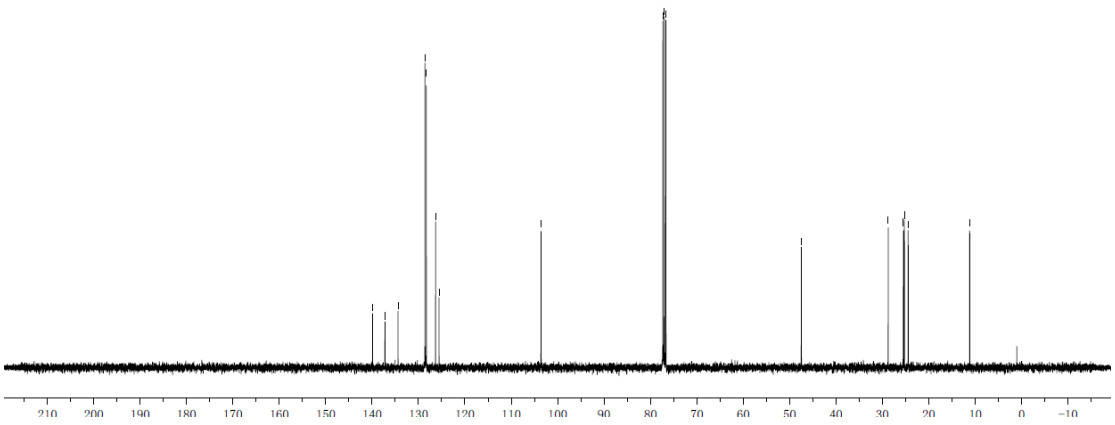


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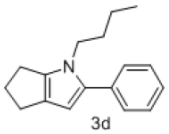
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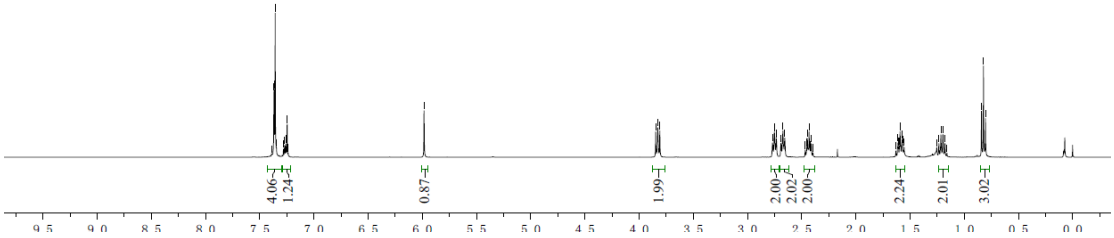
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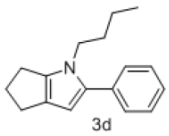
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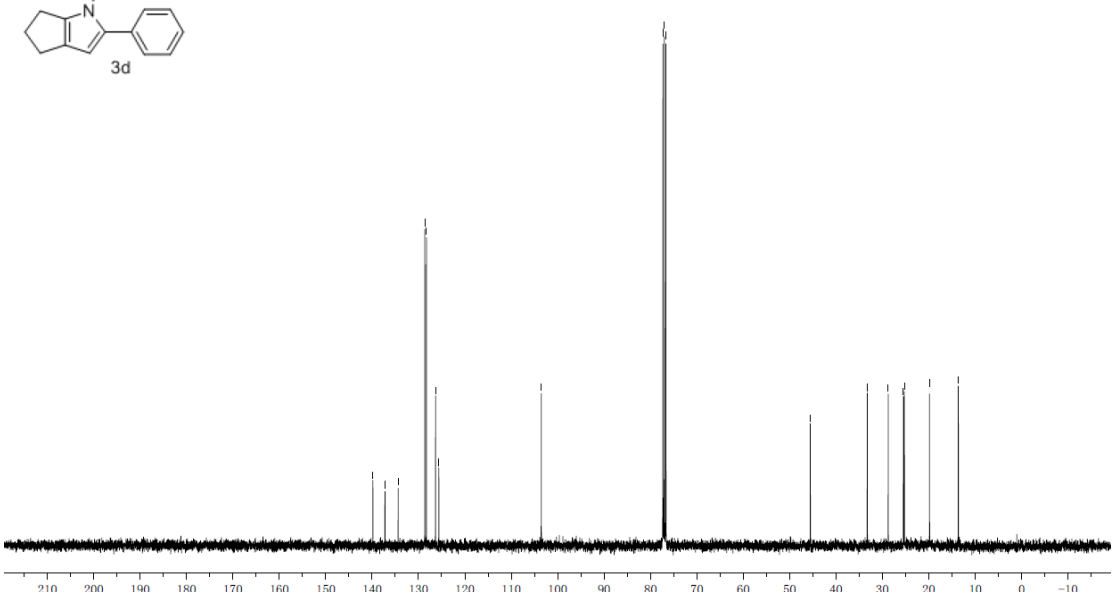

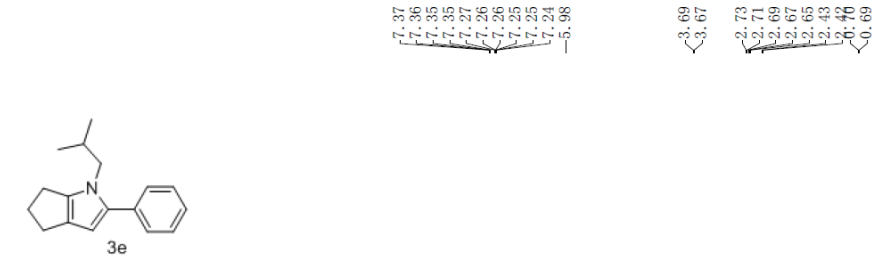
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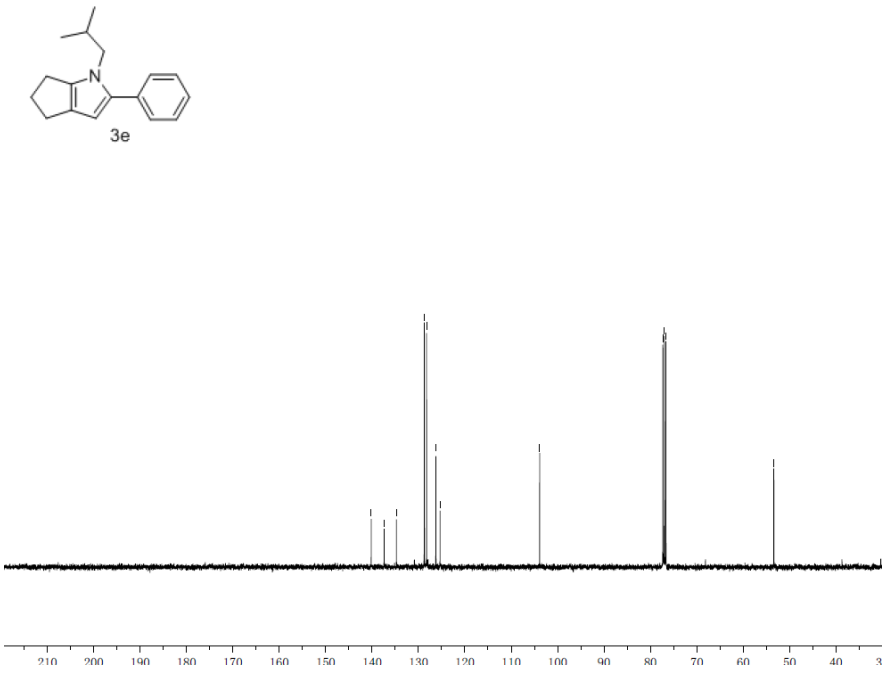
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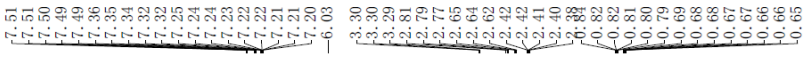
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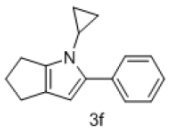
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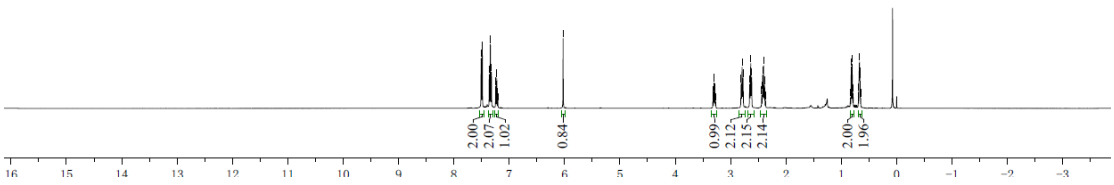
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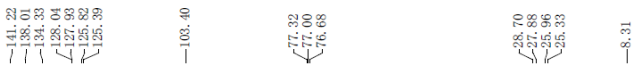
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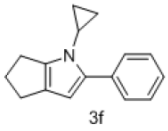
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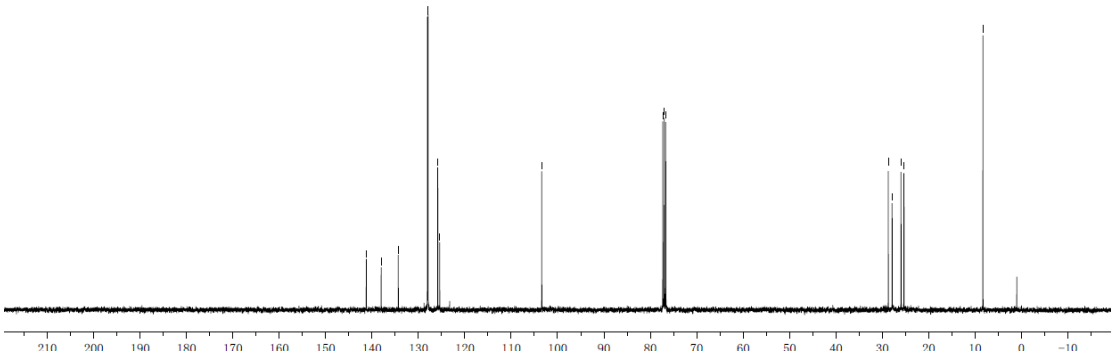
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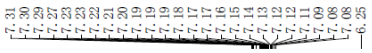
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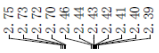
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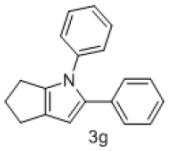
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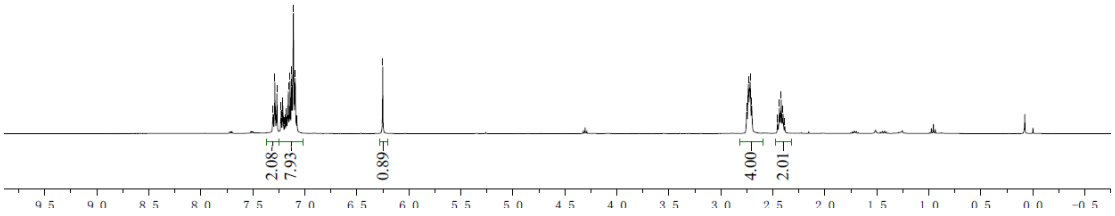
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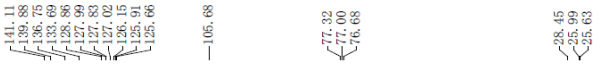
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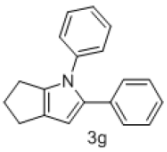
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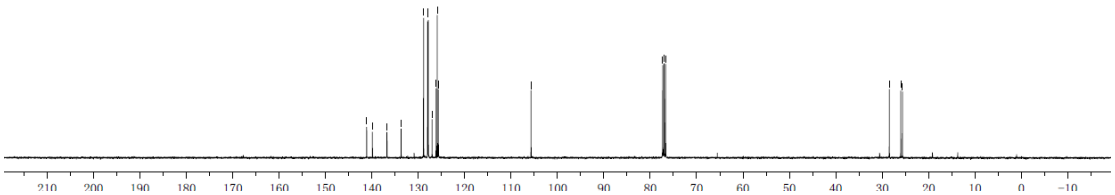
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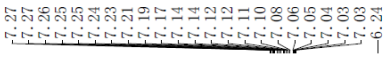
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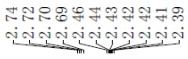
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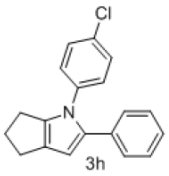
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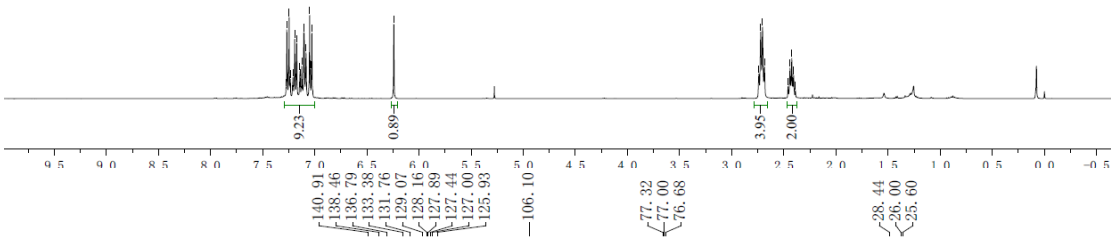
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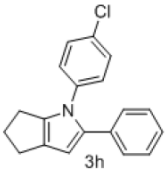
Cl
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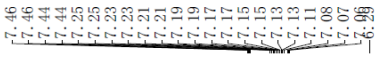

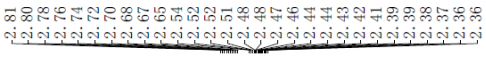

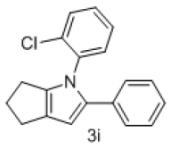

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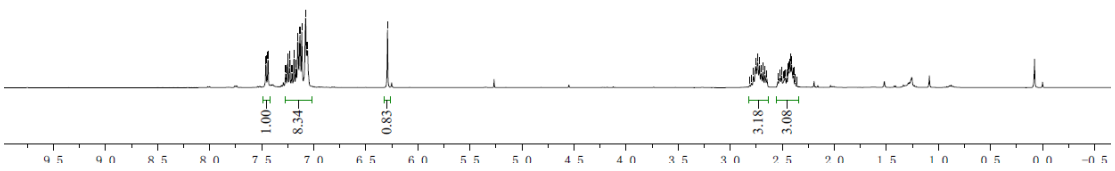

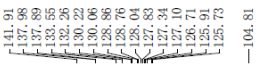

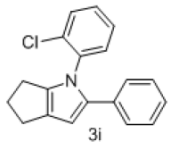

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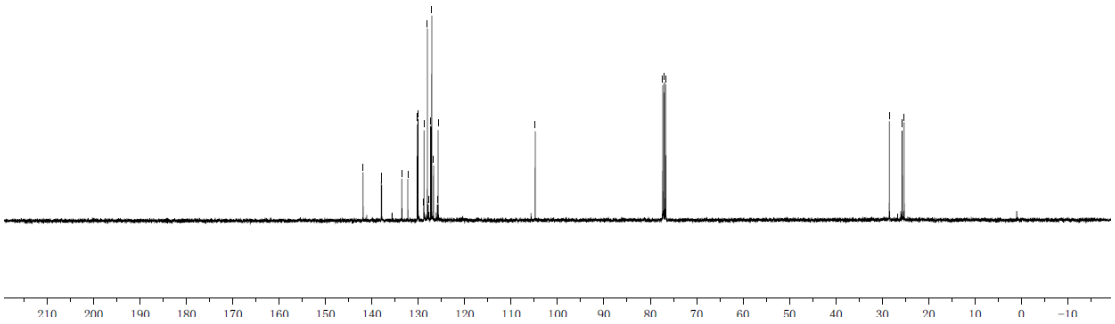

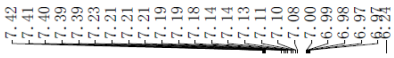
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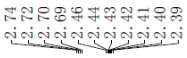
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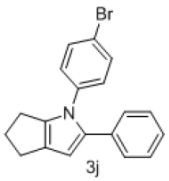
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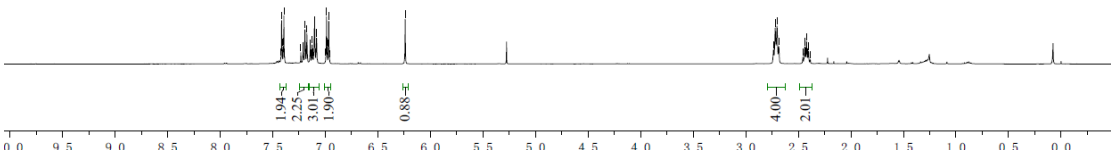
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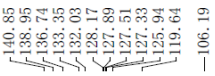
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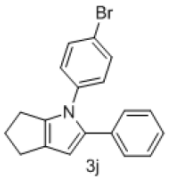
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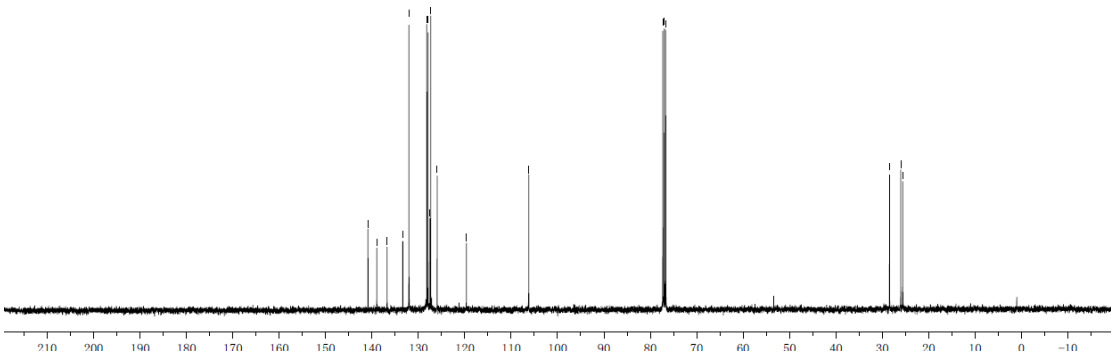

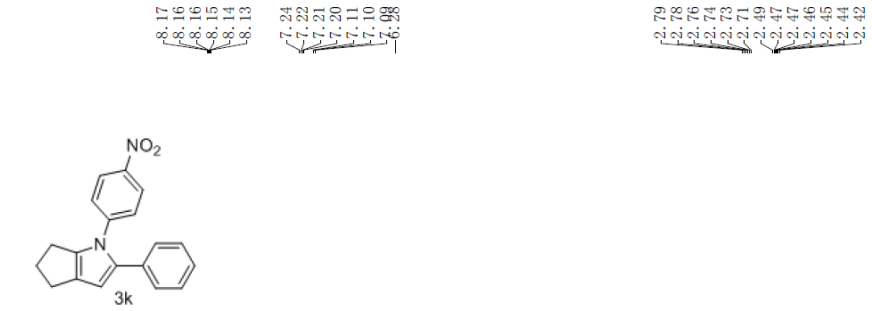
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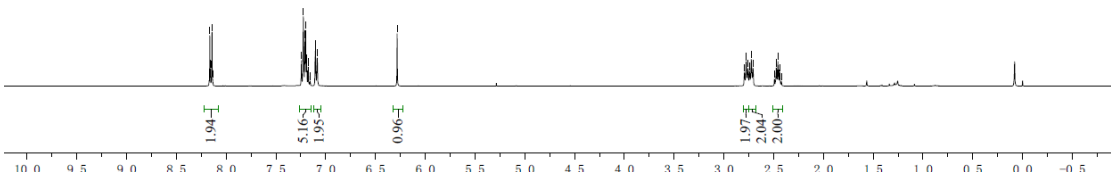
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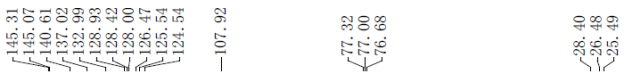
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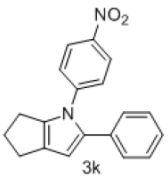
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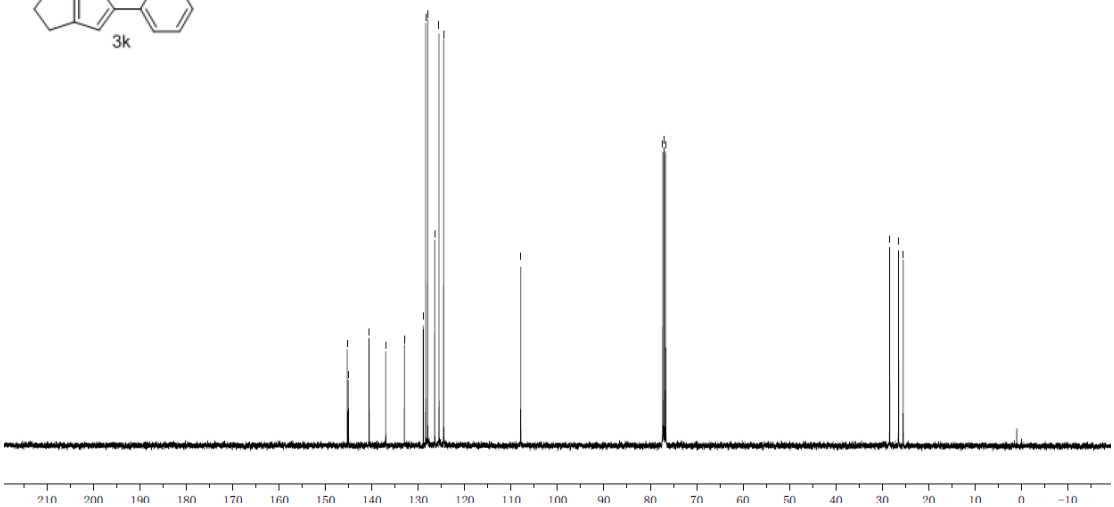

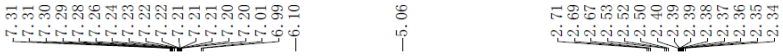
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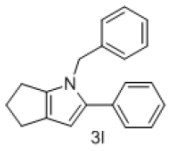
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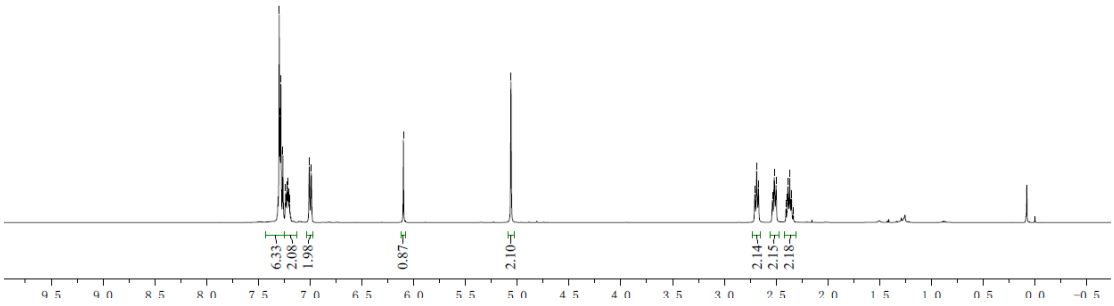
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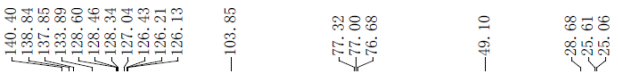
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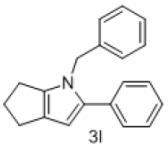
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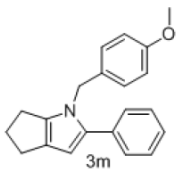
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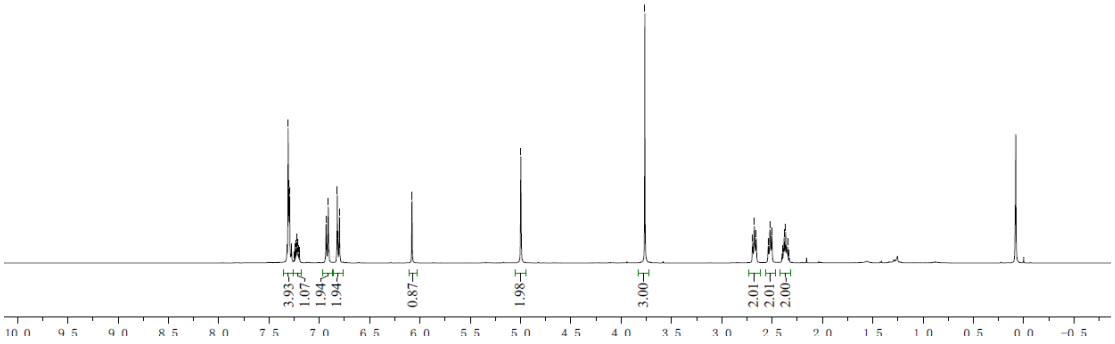
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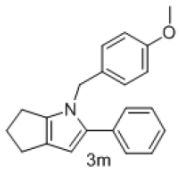
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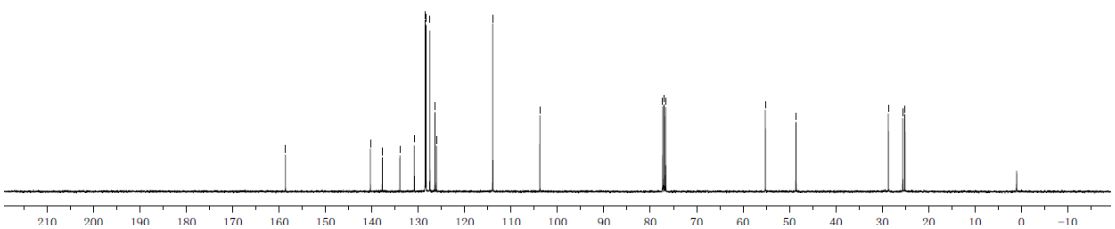

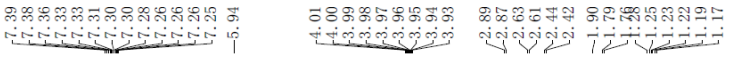
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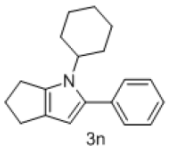
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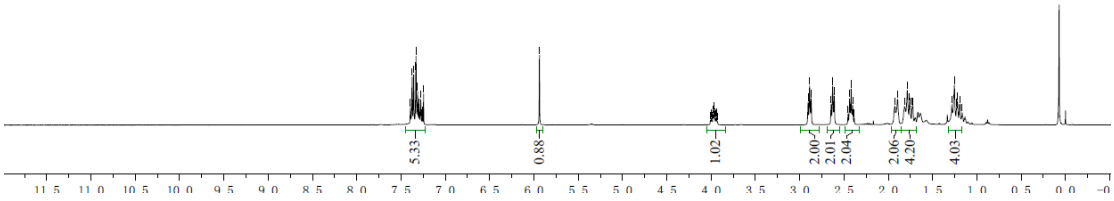
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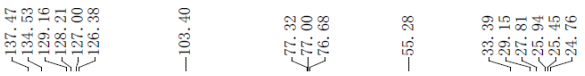
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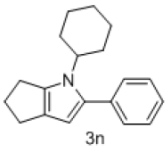
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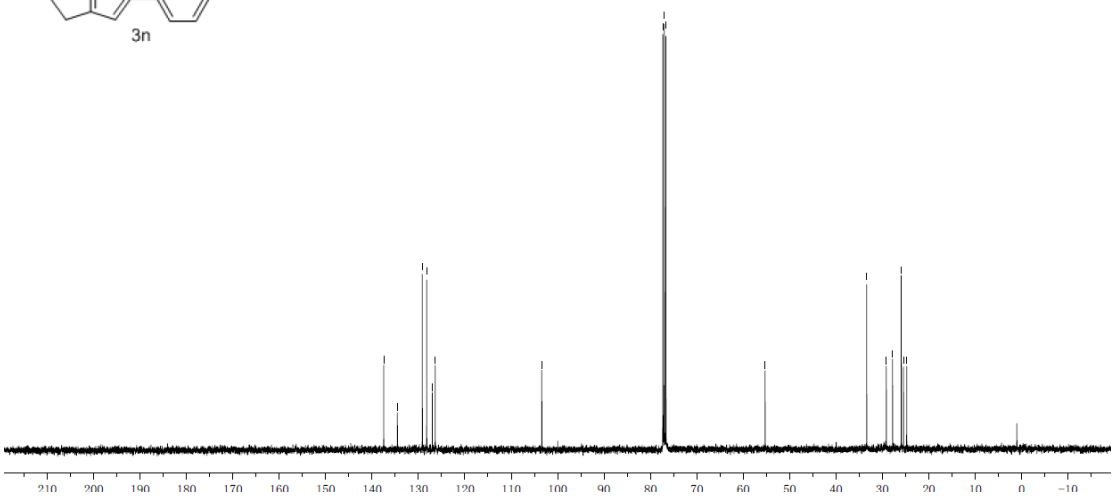

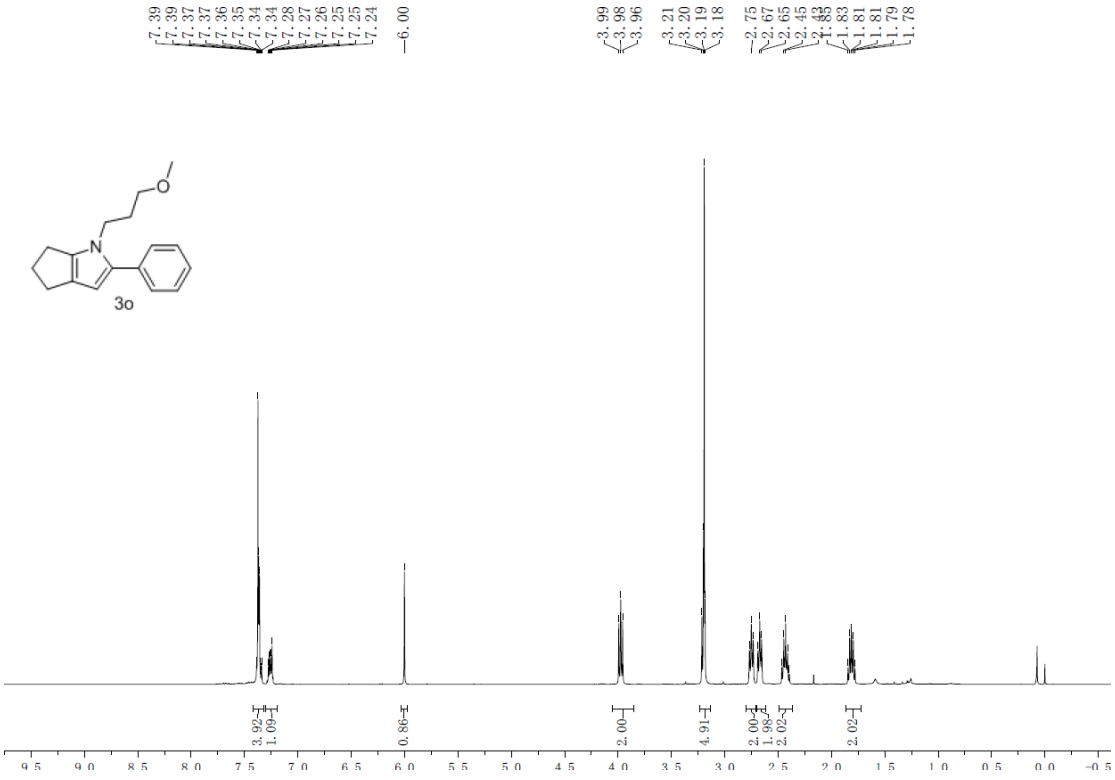
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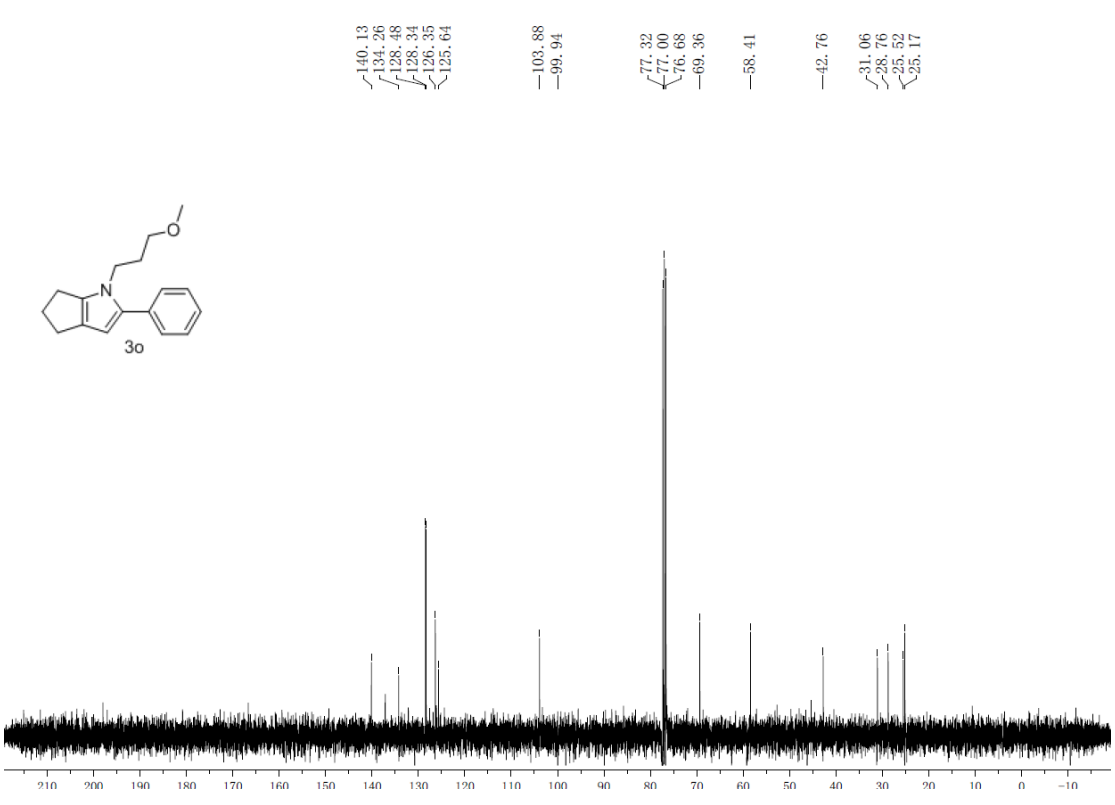
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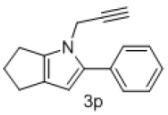
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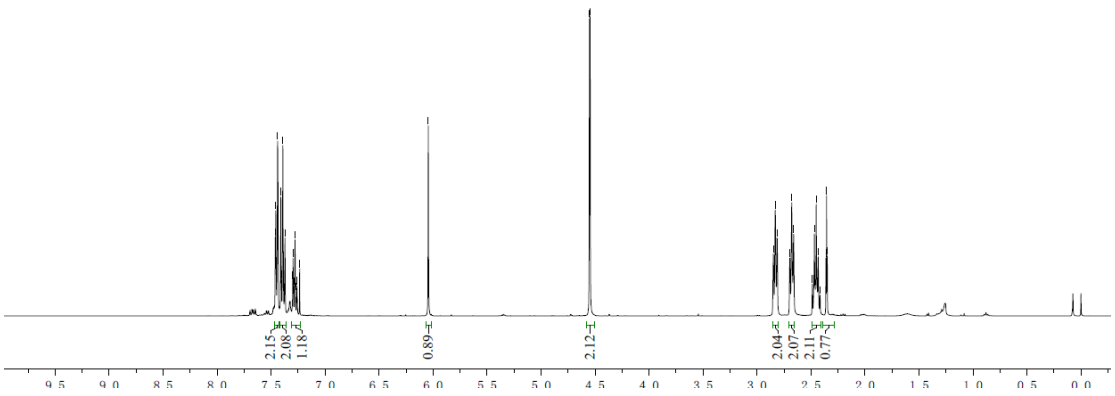
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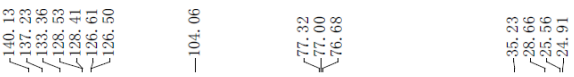
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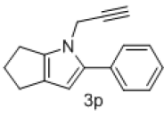
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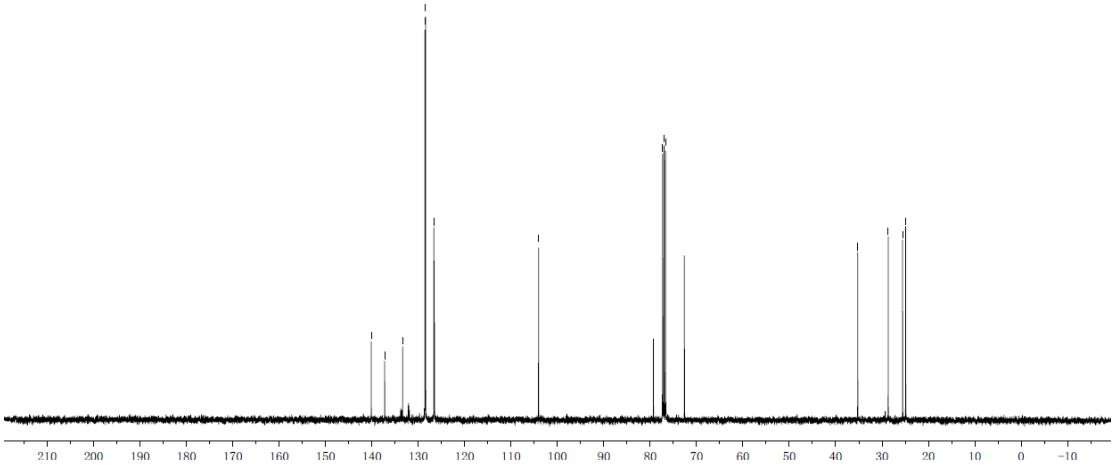

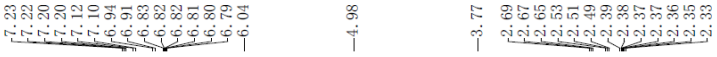
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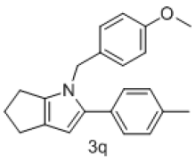
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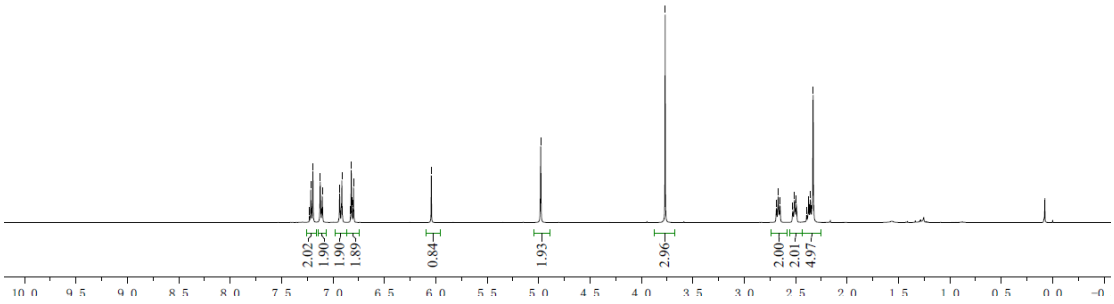
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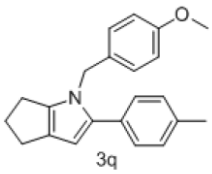
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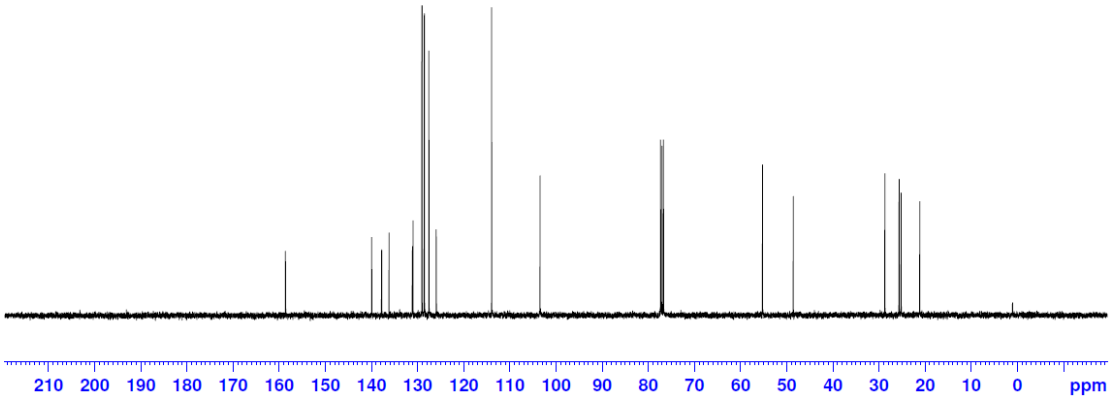
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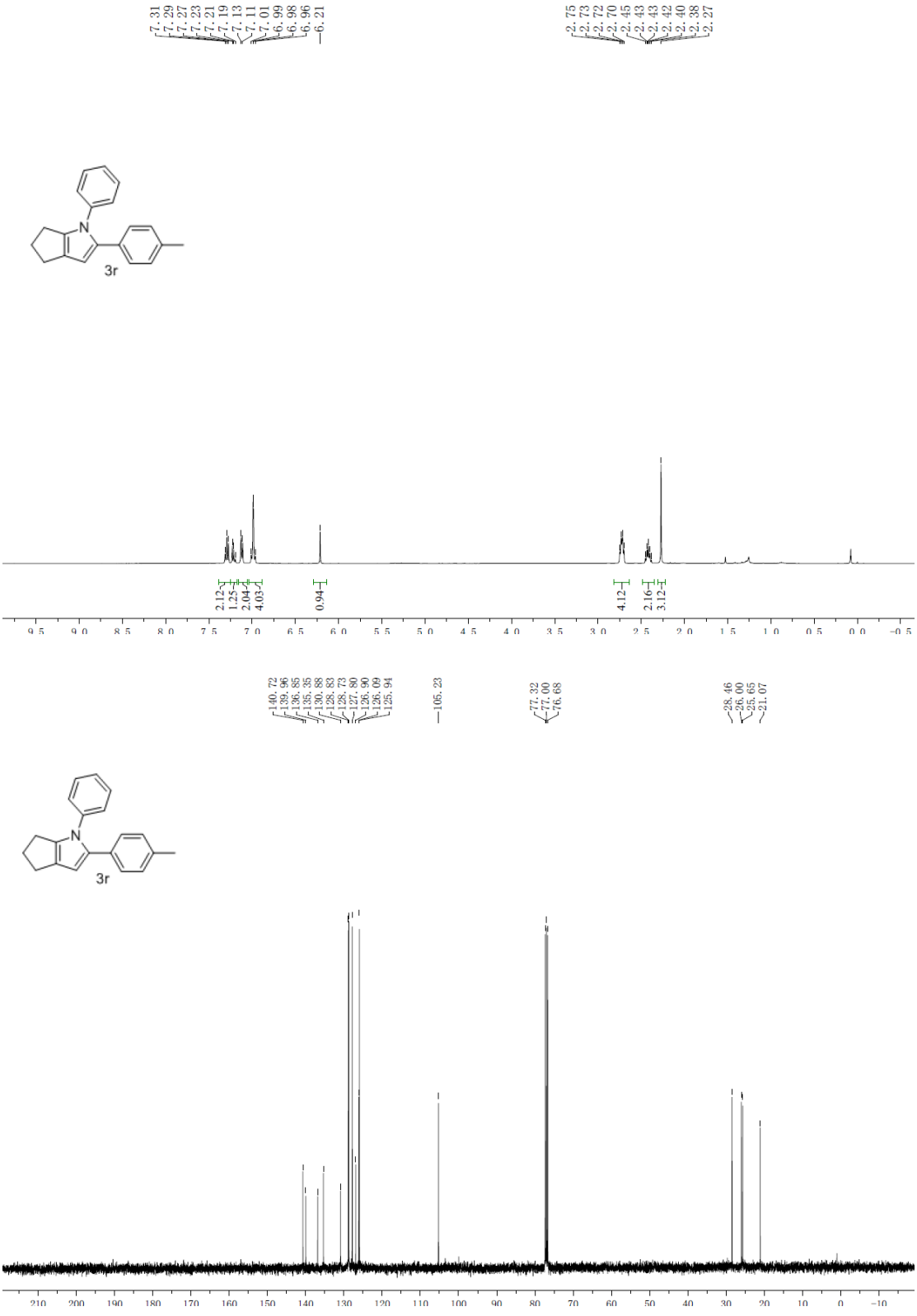
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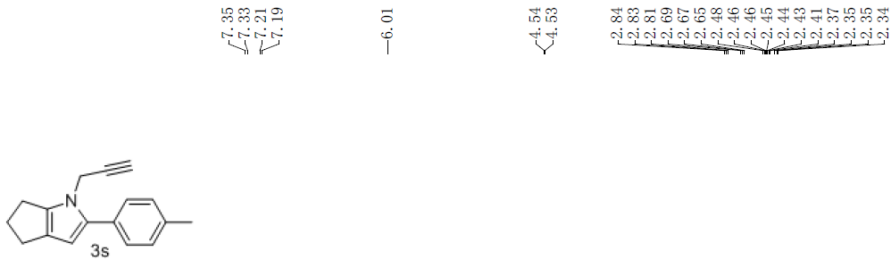
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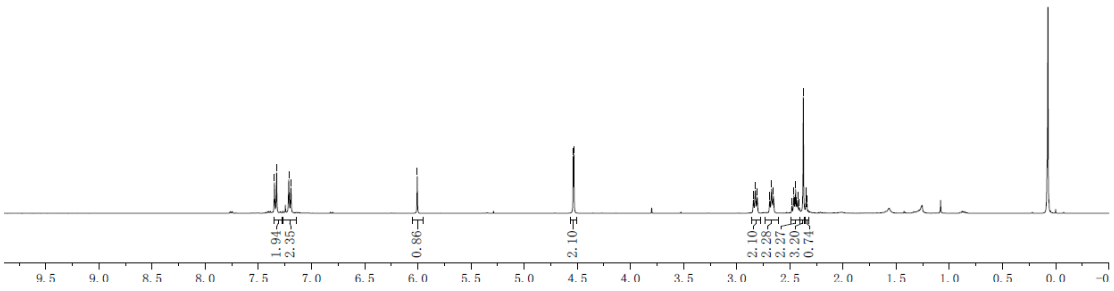
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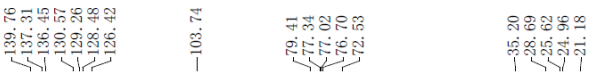
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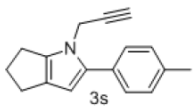
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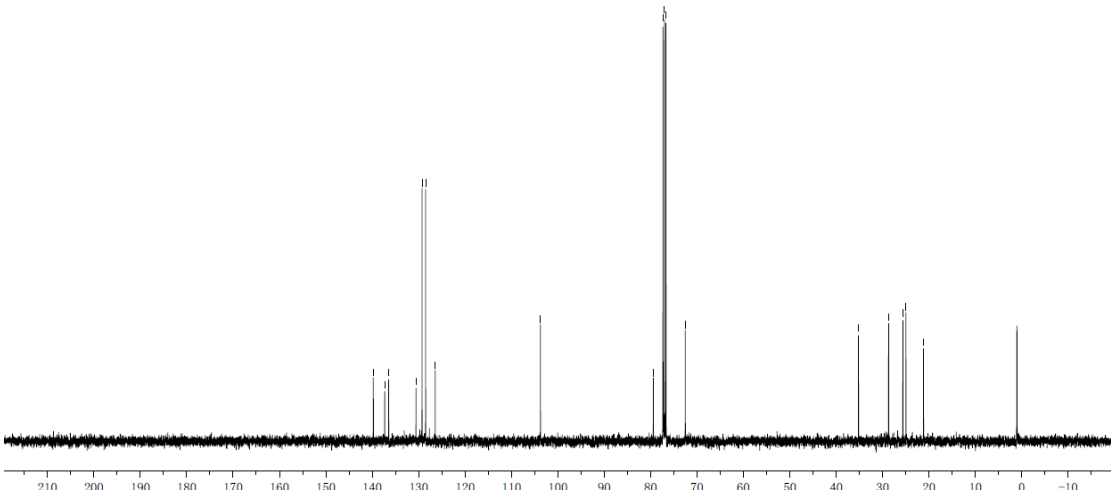
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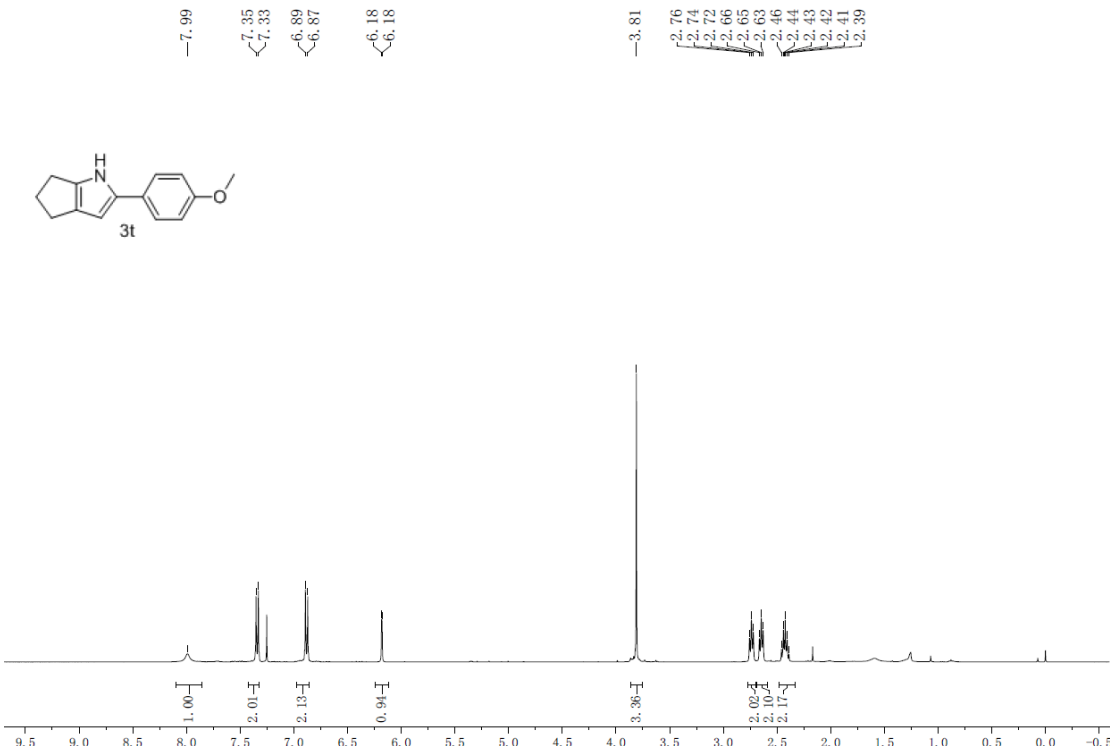
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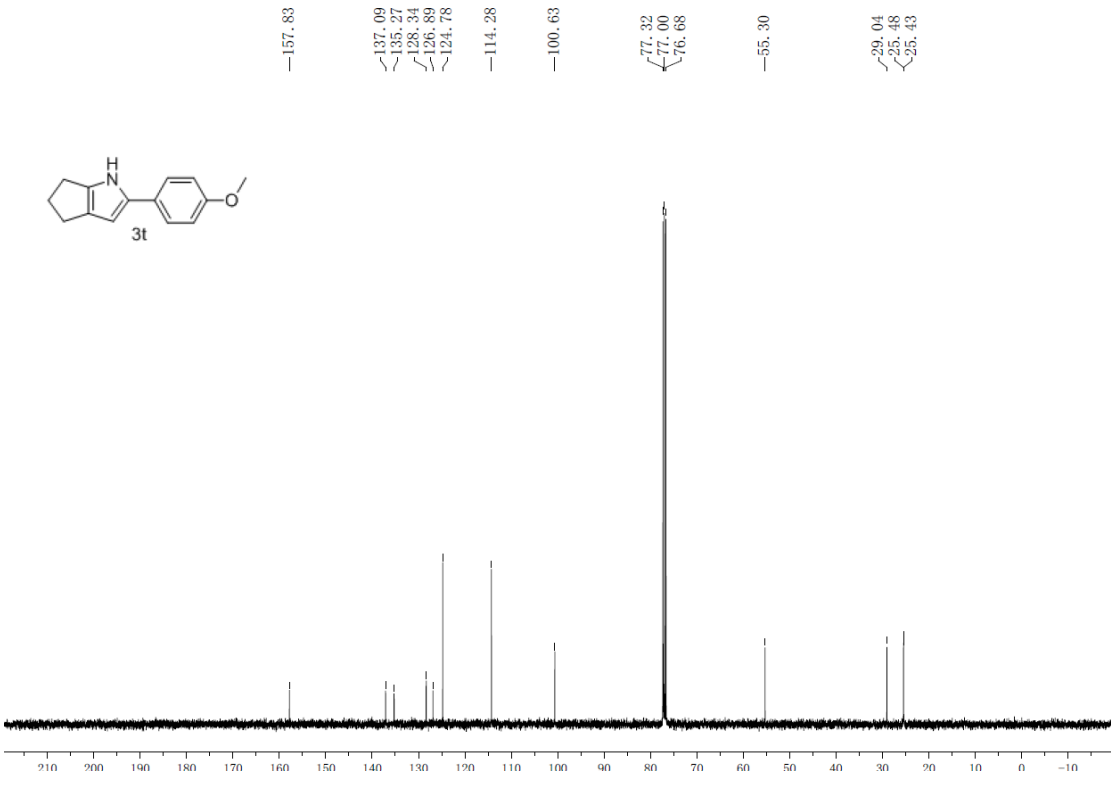
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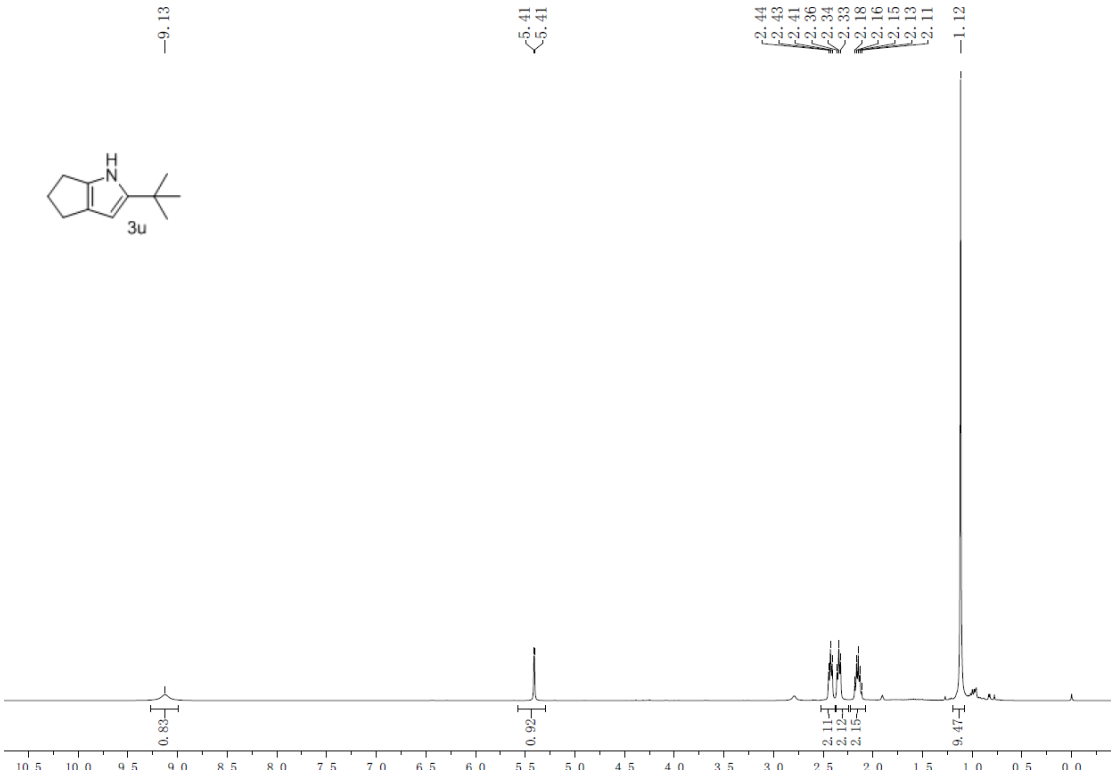
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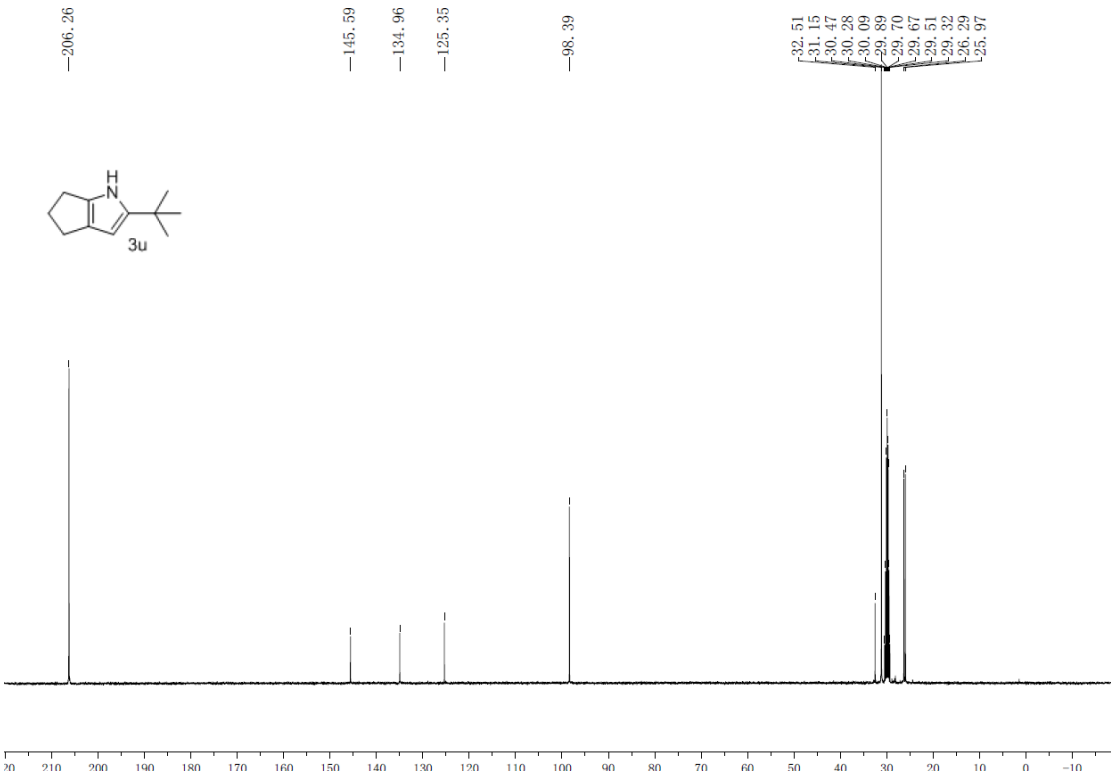
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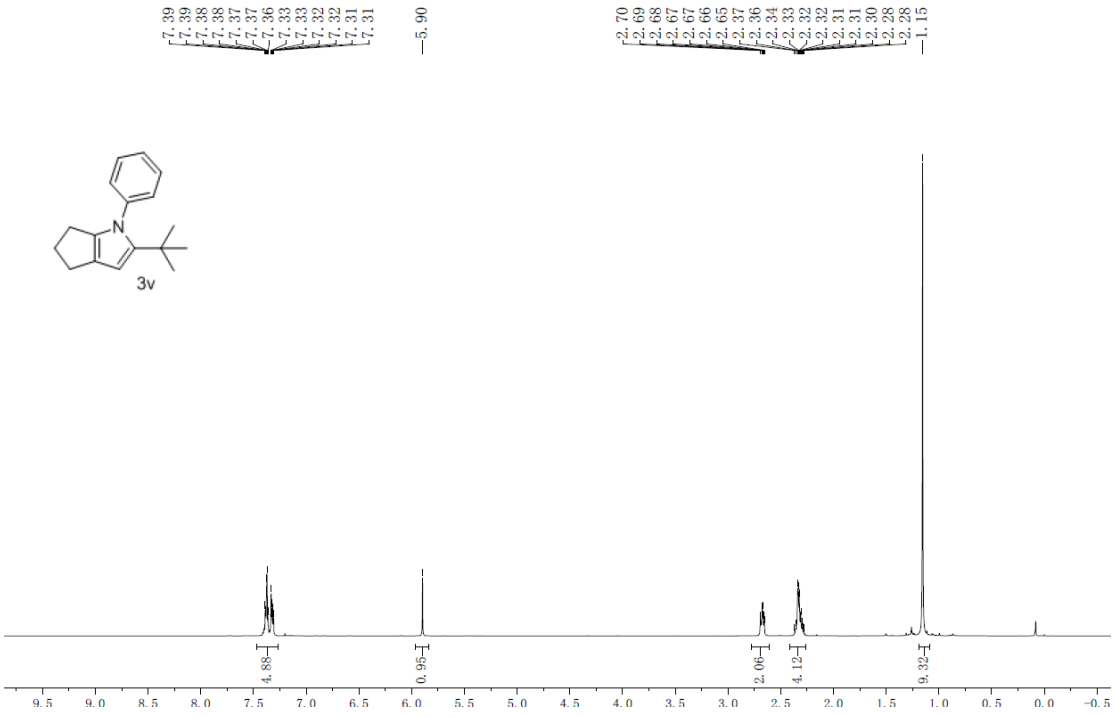
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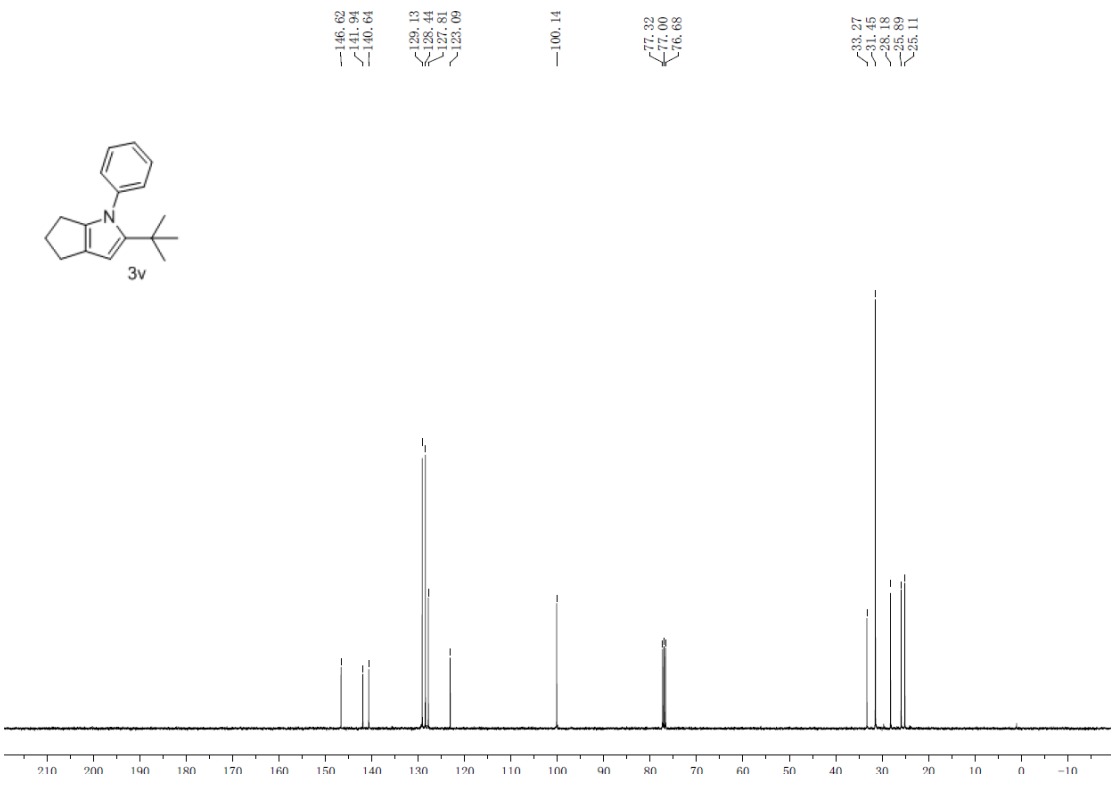
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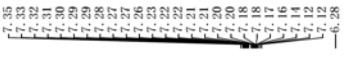
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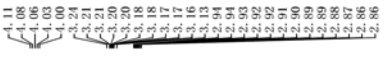
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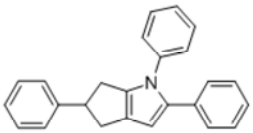
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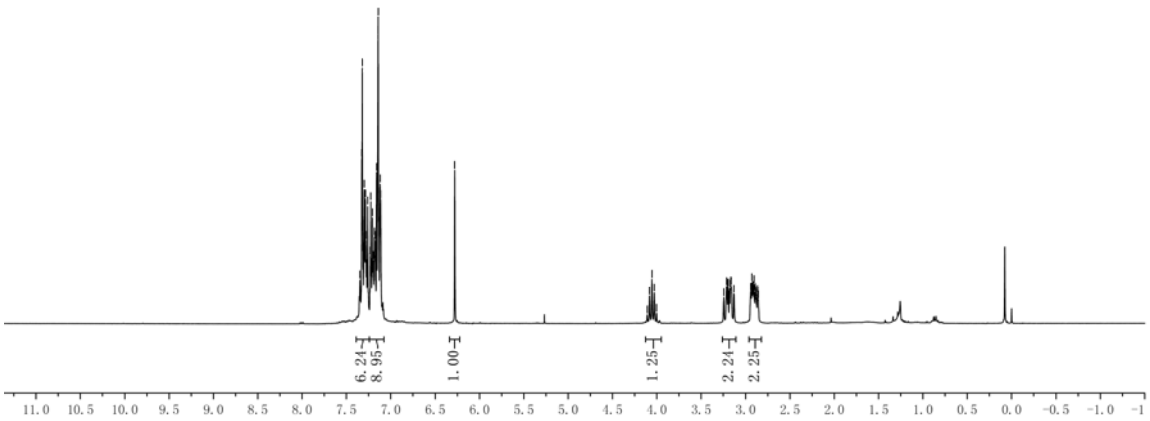
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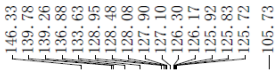
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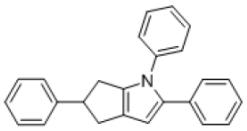
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