

Supporting Information
for
Iron-catalyzed decarboxylative alkenylation of
cycloalkanes with arylvinyl carboxylic acids via a
radical process

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Experimental details and spectral data

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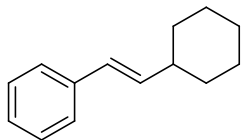
1. Experimental Section

General Information: All reactions were carried out under a nitrogen atmosphere. Column chromatography was carried out on silica gel (300–400 mesh). Analytical thin-layer chromatography (TLC) was performed on glass plates of Silica Gel GF-254 with detection by UV light. Standard techniques for synthesis under inert gas atmosphere by using gasbag and Schlenk glassware equipped with an 8 mm PTFE vacuum stop-cock (Synthware) were employed. All starting materials and reagents were commercially available. ^1H and ^{13}C NMR spectra were recorded on a Bruker AVANCE400M spectrometer. The chemical shift references were as follows: (^1H) CDCl_3 , 7.26 ppm (CHCl_3); (^{13}C) CDCl_3 , 77.00 ppm (CDCl_3). HRMS spectra were carried out at Micromass GCT (TOF MS EI^+). Melting points were determined on a Melt-Temp apparatus (X-4) from Beijing Fukai Electro-optic Instrument Plant and are uncorrected.

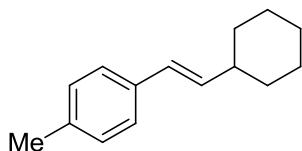
General Procedure: To a Schlenk tube equipped with a magnetic stir bar were added $\text{Fe}(\text{acac})_3$ (21.2 mg, 0.06 mmol) and cinnamic acid (0.3 mmol) under a nitrogen atmosphere. Cycloalkane (2.0 mL, 15–25 mmol) and DTBP (di-*tert*-butyl peroxide, 0.6 mmol, 113 μL) were added under nitrogen and the resulting reaction mixture was kept stirring at 120 $^\circ\text{C}$ for 24 h. After cooling to room temperature and removal of volatiles the products were isolated by flash column chromatography (PE).

Products **3a**, **3e**, **3h**, **3j**, **3k**, **3p**, **4a**, **4c**, **4e**, **4f**, **3b**, **3l**, **3d**, **3n** and **4b**, are known compounds. ^1H and ^{13}C NMR spectra of these compounds have been confirmed to be identical to those of known samples (**3a**, **3e**, **3h**, **3j**, **3k**, **3p**, **4a**, **4c**, **4e** and **4f**) [1], (**3b** and **3l**) [2], **3d** [3], **3n** [4], **4b** [5]).

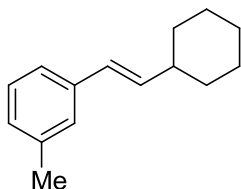
2. Characterization data for 3a–p and 4a–f.



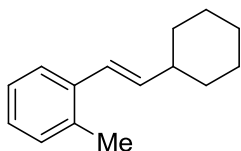
(E)-(2-Cyclohexylvinyl)benzene(3a, known compound): Colorless oil, ^1H NMR (400 MHz, CDCl_3) δ 7.33–7.22 (m, 4H), 7.16–7.12 (m, 1H), 6.32 (d, $J = 16.1$ Hz, 1H), 6.15 (dd, $J = 16.0, 6.9$ Hz, 1H), 2.18–2.00 (m, 1H), 1.86–1.59 (m, 5H), 1.40–1.07 (m, 5H). ^{13}C NMR (100 MHz, CDCl_3) δ 138.02, 136.70, 128.38, 127.26, 126.66, 125.91, 41.12, 32.93, 26.16, 26.03.



(E)-1-(2-Cyclohexylvinyl)-4-methylbenzene(3b, known compound): Colorless oil, ^1H NMR (400 MHz, CDCl_3) δ 7.22 (d, $J = 8.1$ Hz, 2H), 7.06 (d, $J = 8.0$ Hz, 2H), 6.29 (d, $J = 16.0$ Hz, 1H), 6.10 (dd, $J = 16.0, 6.9$ Hz, 1H), 2.29 (s, 3H), 2.13–2.05 (m, 1H), 1.84–1.63 (m, 5H), 1.35–1.12 (m, 5H). ^{13}C NMR (100 MHz, CDCl_3) δ 136.29, 135.73, 135.26, 129.09, 127.06, 125.81, 41.11, 33.01, 26.19, 26.06, 21.06.

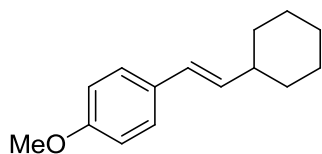


(E)-1-(2-Cyclohexylvinyl)-3-methylbenzene(3c): Colorless oil, ^1H NMR (400 MHz, CDCl_3) δ 7.20–7.06 (m, 3H), 6.97–6.95 (m, 1H), 6.29 (d, $J = 16.0$ Hz, 1H), 6.13 (dd, $J = 16.0, 6.9$ Hz, 1H), 2.29 (s, 3H), 2.18–2.03 (m, 1H), 1.86–1.60 (m, 5H), 1.40–1.08 (m, 5H). ^{13}C NMR (100 MHz, CDCl_3) δ 137.96, 137.79, 136.47, 128.29, 127.46, 127.33, 126.61, 123.11, 41.14, 32.97, 26.17, 26.04, 21.33. HRMS (TOF MS EI^+) m/z calcd for $[\text{C}_{15}\text{H}_{20}]$ 200.1565, found 200.1553.

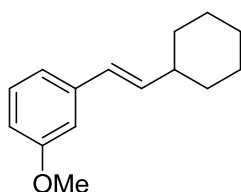


(E)-1-(2-Cyclohexylvinyl)-2-methylbenzene(3d, known compound): Colorless oil, ^1H NMR (400 MHz, CDCl_3) δ 7.63 (d, $J = 7.0$ Hz, 1H), 7.39–7.29 (m, 3H), 6.77 (d, $J = 15.8$ Hz, 1H), 6.26 (dd, $J = 15.8, 7.0$ Hz, 1H), 2.54 (s, 3H), 2.44–2.30 (m, 1H), 2.11–1.85 (m, 5H), 1.65–1.34 (m, 5H). ^{13}C NMR (100 MHz, CDCl_3) δ 138.10, 137.10, 134.81, 130.05, 126.63, 125.92, 125.34, 125.09,

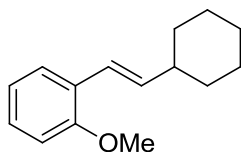
41.43, 33.08, 26.18, 26.05, 19.74.



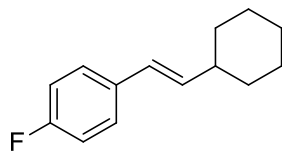
(E)-1-(2-Cyclohexylvinyl)-4-methoxybenzene(3e, known compound): Colorless oil, ^1H NMR (400 MHz, CDCl_3) δ 7.17 (d, $J = 8.7$ Hz, 1H), 6.72 (d, $J = 8.7$ Hz, 1H), 6.19 (d, $J = 16.0$ Hz, 1H), 5.94 (dd, $J = 16.0, 6.9$ Hz, 1H), 3.67 (s, 3H), 2.04-1.96 (m, 1H), 1.76–1.53 (m, 5H), 1.25–1.04 (m, 5H). ^{13}C NMR (100 MHz, CDCl_3) δ 158.57, 134.65, 130.84, 126.94, 126.55, 113.83, 55.15, 41.07, 33.06, 26.17, 26.06.



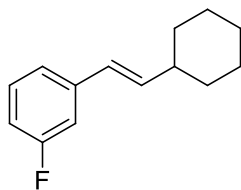
(E)-1-(2-Cyclohexylvinyl)-3-methoxybenzene(3f): Colorless oil, ^1H NMR (400 MHz, CDCl_3) δ 7.18 (t, $J = 7.9$ Hz, 1H), 6.96–6.86 (m, 2H), 6.72 (dd, $J = 8.1, 2.1$ Hz, 1H), 6.30 (d, $J = 16.0$ Hz, 1H), 6.16 (dd, $J = 16.0, 6.8$ Hz, 1H), 3.78 (s, 3H), 2.12-2.06 (m, 1H), 1.85–1.63 (m, 5H), 1.34–1.14 (m, 5H). ^{13}C NMR (100 MHz, CDCl_3) δ 159.76, 139.51, 137.07, 129.33, 127.13, 118.60, 112.35, 111.22, 55.07, 41.08, 32.89, 26.14, 26.00. HRMS (TOF MS EI^+) m/z calcd for $[\text{C}_{15}\text{H}_{20}\text{O}]$ 216.1514, found 216.1510.



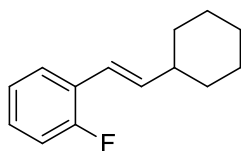
(E)-1-(2-Cyclohexylvinyl)-2-methoxybenzene(3g): Colorless oil, ^1H NMR (400 MHz, CDCl_3) δ 7.41 (dd, $J = 7.6, 1.1$ Hz, 1H), 7.14 (ddd, $J = 8.8, 7.7, 1.1$ Hz, 1H), 6.87 (t, $J = 7.4$ Hz, 1H), 6.80 (d, $J = 8.2$ Hz, 1H), 6.69 (d, $J = 16.1$ Hz, 1H), 6.14 (dd, $J = 16.1, 7.0$ Hz, 1H), 3.78 (s, 3H), 2.23–2.05 (m, 1H), 1.88–1.61 (m, 5H), 1.38–1.11 (m, 5H). ^{13}C NMR (100 MHz, CDCl_3) δ 156.27, 137.27, 127.66, 127.04, 126.12, 121.72, 120.54, 110.70, 55.31, 41.53, 33.04, 26.17, 26.05. HRMS (TOF MS EI^+) m/z calcd for $[\text{C}_{15}\text{H}_{20}\text{O}]$ 216.1514, found 216.1505.



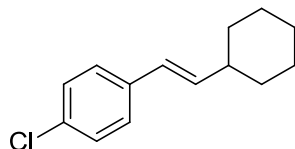
(E)-1-(2-Cyclohexylvinyl)-4-fluorobenzene(3h, known compound): White solid, mp 34–35 °C, ^1H NMR (400 MHz, CDCl_3) δ 7.50 (d, $J = 8.2$ Hz, 2H), 7.38 (d, $J = 8.2$ Hz, 2H), 6.34 (d, $J = 16.1$ Hz, 1H), 6.25 (dd, $J = 16.0, 6.6$ Hz, 1H), 2.17-2.09 (m, 1H), 1.87–1.64 (m, 5H), 1.33-1.16 (m, 5H). ^{13}C NMR (100 MHz, CDCl_3) δ 141.64 (d, $J = 1.2$ Hz), 139.56, 128.61 (d, $J = 32.3$ Hz), 126.18, 126.05, 125.36 (q, $J = 3.9$ Hz), 41.23, 32.76, 26.12, 25.99.



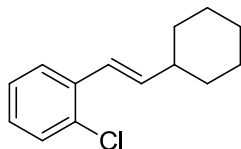
(E)-1-(2-Cyclohexylvinyl)-3-fluorobenzene(3i): Colorless oil, ^1H NMR (400 MHz, CDCl_3) δ 7.20 (td, $J = 7.9, 6.1$ Hz, 1H), 7.04 (ddd, $J = 10.4, 8.1, 4.8$ Hz, 2H), 6.88–6.82 (m, 1H), 6.29 (d, $J = 16.0$ Hz, 1H), 6.16 (dd, $J = 15.9, 6.8$ Hz, 1H), 2.14–2.06 (m, 1H), 1.87–1.62 (m, 5H), 1.35–1.11 (m, 5H). ^{13}C NMR (100 MHz, CDCl_3) δ 163.15 (d, $J = 244.7$ Hz), 140.50 (d, $J = 7.7$ Hz), 138.17, 129.77 (d, $J = 8.5$ Hz), 126.31 (d, $J = 2.5$ Hz), 121.82 (d, $J = 2.6$ Hz), 113.42 (d, $J = 21.4$ Hz), 112.31 (d, $J = 21.7$ Hz), 41.08, 32.81, 26.12, 25.99. HRMS (TOF MS EI^+) m/z calcd for $[\text{C}_{14}\text{H}_{17}\text{F}]$ 204.1314, found 204.1320.



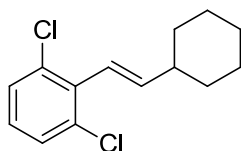
(E)-1-(2-Cyclohexylvinyl)-2-fluorobenzene(3j, known compound): Colorless oil, ^1H NMR (400 MHz, CDCl_3) δ 7.42–7.38 (m, 1H), 7.16–6.93 (m, 3H), 6.50 (d, $J = 16.2$ Hz, 1H), 6.22 (dd, $J = 16.1, 7.0$ Hz, 1H), 2.16–2.09 (m, 1H), 1.87–1.61 (m, 5H), 1.39–1.10 (m, 5H). ^{13}C NMR (100 MHz, CDCl_3) δ 160.00 (d, $J = 248.0$ Hz), 139.26 (d, $J = 4.1$ Hz), 127.83 (d, $J = 8.3$ Hz), 126.85 (d, $J = 4.0$ Hz), 125.76 (d, $J = 12.3$ Hz), 123.88 (d, $J = 3.5$ Hz), 119.61 (d, $J = 3.9$ Hz), 115.50 (d, $J = 22.3$ Hz), 41.52, 32.84, 26.14, 26.00.



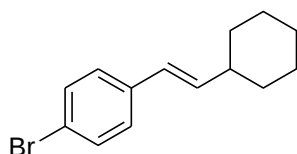
(E)-1-Chloro-4-(2-cyclohexylvinyl)benzene(3k, known compound): Colorless oil, ^1H NMR (400 MHz, CDCl_3) δ 7.25–7.17 (m, 4H), 6.27 (d, $J = 16.0$, 1H), 6.13 (dd, $J = 16.0, 6.8$ Hz, 1H), 2.14–2.06 (m, 1H), 1.80–1.65 (m, 5H), 1.32–1.14 (m, 5H). ^{13}C NMR (100 MHz, CDCl_3) δ 137.46, 136.54, 132.20, 128.50, 127.11, 126.09, 41.10, 32.84, 26.12, 25.99.



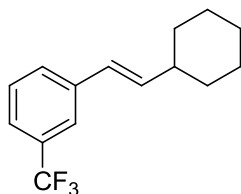
(E)-1-Chloro-2-(2-cyclohexylvinyl)benzene(3l, known compound): Colorless oil, ^1H NMR (400 MHz, CDCl_3) δ 7.47 (dd, $J = 7.7, 1.4$ Hz, 1H), 7.29 (dd, $J = 7.9, 1.1$ Hz, 1H), 7.17–7.04 (m, 2H), 6.72 (d, $J = 15.9$ Hz, 1H), 6.13 (dd, $J = 15.9, 7.0$ Hz, 1H), 2.18–2.13 (m, 1H), 1.87–1.61 (m, 5H), 1.38–1.10 (m, 5H). ^{13}C NMR (100 MHz, CDCl_3) δ 139.56, 136.05, 132.66, 129.51, 127.68, 126.62, 126.46, 123.62, 41.31, 32.83, 26.13, 25.97.



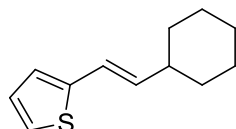
(E)-1,3-Dichloro-2-(2-cyclohexylvinyl)benzene(3m): Colorless oil, ^1H NMR (400 MHz, CDCl_3) δ 7.25 (d, $J = 8.0$ Hz, 2H), 7.00 (t, $J = 8.0$ Hz, 1H), 6.28 (d, $J = 16.4$ Hz, 1H), 6.14 (dd, $J = 16.3$, 6.7 Hz, 1H), 2.24–2.16 (m, 1H), 1.89–1.64 (m, 5H), 1.40–1.17 (m, 5H). ^{13}C NMR (100 MHz, CDCl_3) δ 145.16, 135.49, 134.34, 128.23, 127.38, 120.98, 41.52, 32.51, 26.17, 25.89. HRMS (TOF MS EI^+) m/z calcd for $[\text{C}_{14}\text{H}_{16}\text{Cl}_2]$ 254.0629, found 254.0625.



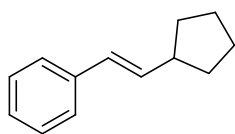
(E)-1-Bromo-4-(2-cyclohexylvinyl)benzene (3n, known compound): White solid, mp 39–40 °C, ^1H NMR (400 MHz, CDCl_3) δ 7.36 (d, $J = 8.5$ Hz, 2H), 7.16 (d, $J = 8.4$ Hz, 2H), 6.25 (d, $J = 16.1$ Hz, 1H), 6.13 (dd, $J = 16.0$, 6.7 Hz, 1H), 2.13–2.05 (m, 1H), 1.87–1.54 (m, 5H), 1.38–1.04 (m, 5H). ^{13}C NMR (100 MHz, CDCl_3) δ 137.54, 136.95, 131.41, 127.44, 126.14, 120.28, 41.08, 32.79, 26.11, 25.98.



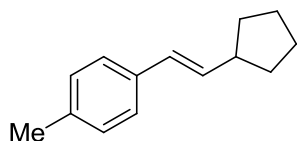
(E)-1-(2-Cyclohexylvinyl)-3-(trifluoromethyl)benzene(3o): Colorless oil, ^1H NMR (400 MHz, CDCl_3) δ 7.57 (s, 1H), 7.46–7.30 (m, 3H), 6.34 (d, $J = 16.1$ Hz, 1H), 6.22 (dd, $J = 16.0$, 6.7 Hz, 1H), 2.18–2.06 (m, 1H), 1.83–1.63 (m, 5H), 1.35–1.11 (m, 5H). ^{13}C NMR (100 MHz, CDCl_3) δ 138.85 (d, $J = 13.6$ Hz), 130.90 (q, $J = 32.0$ Hz), 129.13 (d, $J = 1.1$ Hz), 128.81, 126.12, 125.67, 123.22 (q, $J = 3.8$ Hz), 122.96, 122.57 (q, $J = 3.8$ Hz), 41.19, 32.81, 26.13, 26.00. HRMS (TOF MS EI^+) m/z calcd for $[\text{C}_{15}\text{H}_{17}\text{F}_3]$ 254.1282, found 254.1291.



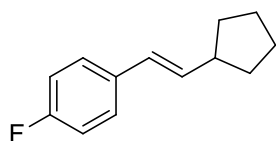
(E)-2-(2-Cyclohexylvinyl)thiophene(3p, known compound): Colorless oil, ^1H NMR (400 MHz, CDCl_3) δ 7.04 (d, $J = 5.1$ Hz, 1H), 6.90 (dd, $J = 5.1$, 3.5 Hz, 1H), 6.84 (d, $J = 3.6$ Hz, 1H), 6.45 (d, $J = 15.8$ Hz, 1H), 6.01 (dd, $J = 15.8$, 6.9 Hz, 1H), 2.12–1.99 (m, 1H), 1.82–1.72 (m, 5H), 1.33–1.11 (m, 5H). ^{13}C NMR (100 MHz, CDCl_3) δ 143.38, 136.69, 127.10, 124.13, 122.89, 120.61, 40.89, 32.77, 26.11, 25.97.



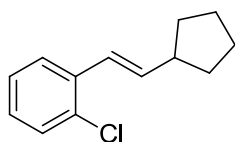
(E)-(2-Cyclopentylvinyl)benzene(4a, known compound): Colorless oil, ^1H NMR (400 MHz, CDCl_3) δ 7.34–7.29 (m, 2H), 7.25 (dd, J = 10.4, 4.9 Hz, 2H), 7.14 (ddd, J = 8.5, 2.5, 1.2 Hz, 1H), 6.35 (d, J = 15.9 Hz, 1H), 6.18 (dd, J = 15.8, 7.7 Hz, 1H), 2.62–2.51 (m, 1H), 1.90–1.78 (m, 2H), 1.74–1.52 (m, 4H), 1.44–1.32 (m, 2H). ^{13}C NMR (100 MHz, CDCl_3) δ 137.90, 135.56, 128.39, 127.88, 126.65, 125.88, 43.78, 33.18, 25.21.



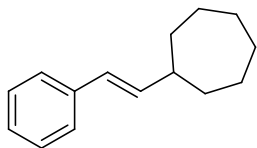
(E)-1-(2-Cyclopentylvinyl)-4-methylbenzene(4b, known compound): Colorless oil, ^1H NMR (400 MHz, CDCl_3) δ 7.48 (d, J = 6.5 Hz, 2H), 7.32 (d, J = 7.5 Hz, 2H), 6.59 (d, J = 15.8 Hz, 1H), 6.39 (ddd, J = 15.8, 7.7, 1.7 Hz, 1H), 2.93–2.73 (m, 1H), 2.55 (s, 3H), 2.21–1.58 (m, 8H). ^{13}C NMR (100 MHz, CDCl_3) δ 136.15, 135.12, 134.37, 129.05, 127.76, 125.78, 43.78, 33.20, 25.19, 21.01.



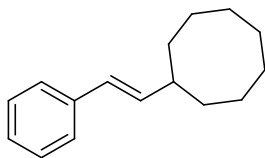
(E)-1-(2-Cyclopentylvinyl)-4-fluorobenzene(4c): Colorless oil, ^1H NMR (400 MHz, CDCl_3) δ 7.31–7.23 (m, 2H), 6.98–6.93 (m, 2H), 6.31 (d, J = 15.8 Hz, 1H), 6.09 (dd, J = 15.8, 7.8 Hz, 1H), 2.71–2.42 (m, 1H), 1.95–1.22 (m, 4H). ^{13}C NMR (100 MHz, CDCl_3) δ 161.82 (d, J = 245.5 Hz), 135.35 (d, J = 2.2 Hz), 134.07 (d, J = 3.2 Hz), 127.27 (d, J = 7.8 Hz), 126.71, 115.23 (d, J = 21.4 Hz), 43.75, 33.19, 25.20. HRMS (TOF MS EI^+) m/z calcd for $[\text{C}_{13}\text{H}_{15}\text{F}]$ 190.1158, found 190.1151.



(E)-1-Chloro-2-(2-cyclopentylvinyl)benzene(4d): Colorless oil, ^1H NMR (400 MHz, CDCl_3) δ 7.31 (s, 1H), 7.24–7.06 (m, 3H), 6.27 (d, J = 15.8 Hz, 1H), 6.18 (dd, J = 15.8, 7.4 Hz, 1H), 2.68–2.41 (m, 1H), 1.92–1.30 (m, 8H). ^{13}C NMR (100 MHz, CDCl_3) δ 139.82, 137.17, 134.35, 129.56, 126.63, 126.55, 125.77, 124.14, 43.71, 33.09, 25.20. HRMS (TOF MS EI^+) m/z calcd for $[\text{C}_{13}\text{H}_{15}\text{Cl}]$ 206.0862, found 206.0861.



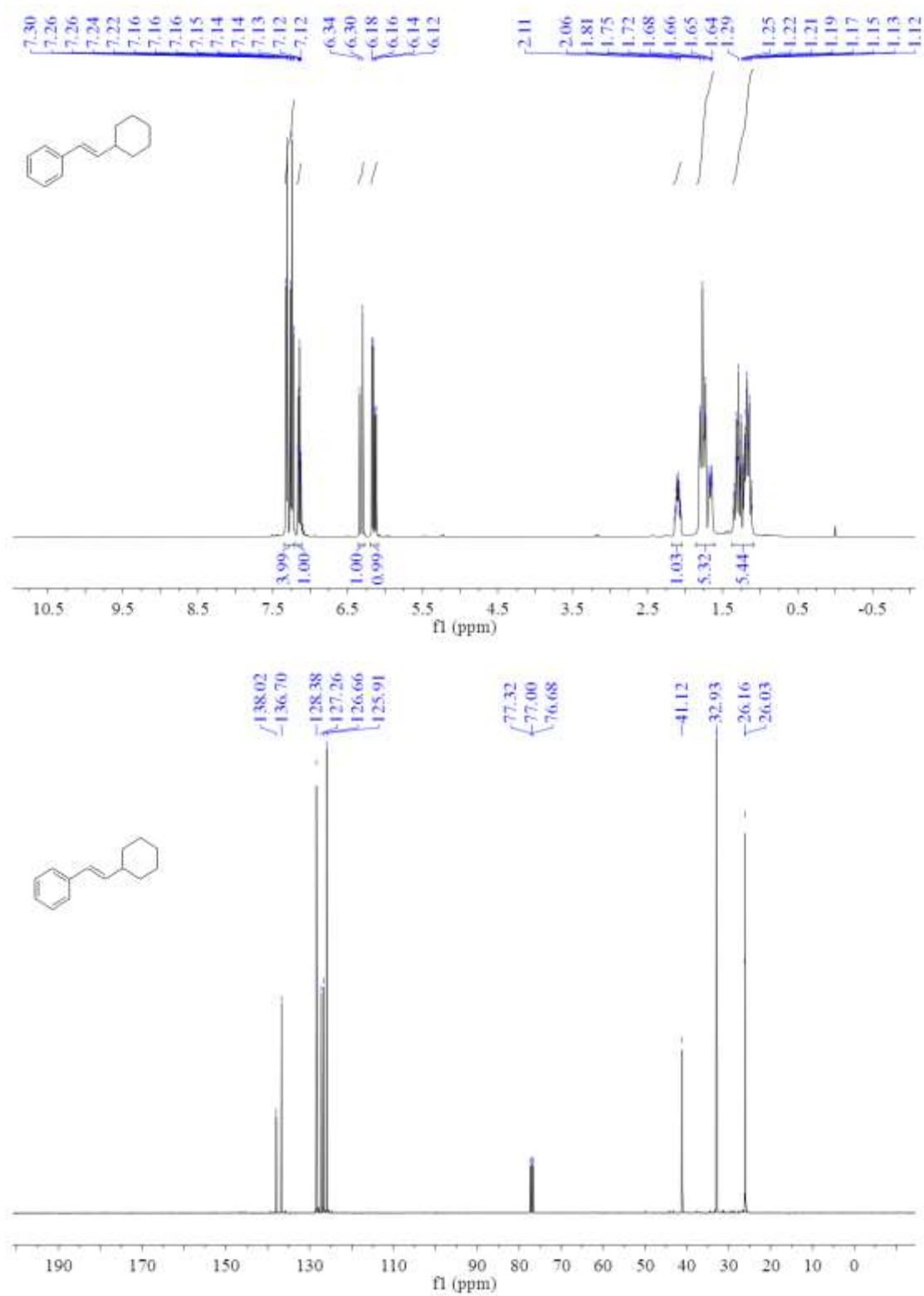
(E)-Styrylcycloheptane(4e, known compound): Colorless oil, ^1H NMR (400 MHz, CDCl_3) δ 7.31 (dd, $J = 8.3, 1.1$ Hz, 2H), 7.25 (dd, $J = 10.4, 4.9$ Hz, 2H), 7.17–7.10 (m, 1H), 6.30 (d, $J = 16.0$ Hz, 1H), 6.19 (dd, $J = 15.9, 7.4$ Hz, 1H), 2.37–2.24 (m, 1H), 1.83–1.78 (m, 2H), 1.68–1.37 (m, 10H). ^{13}C NMR (100 MHz, CDCl_3) δ 138.09, 137.53, 128.38, 126.70, 126.61, 125.90, 43.20, 34.71, 28.38, 26.25.



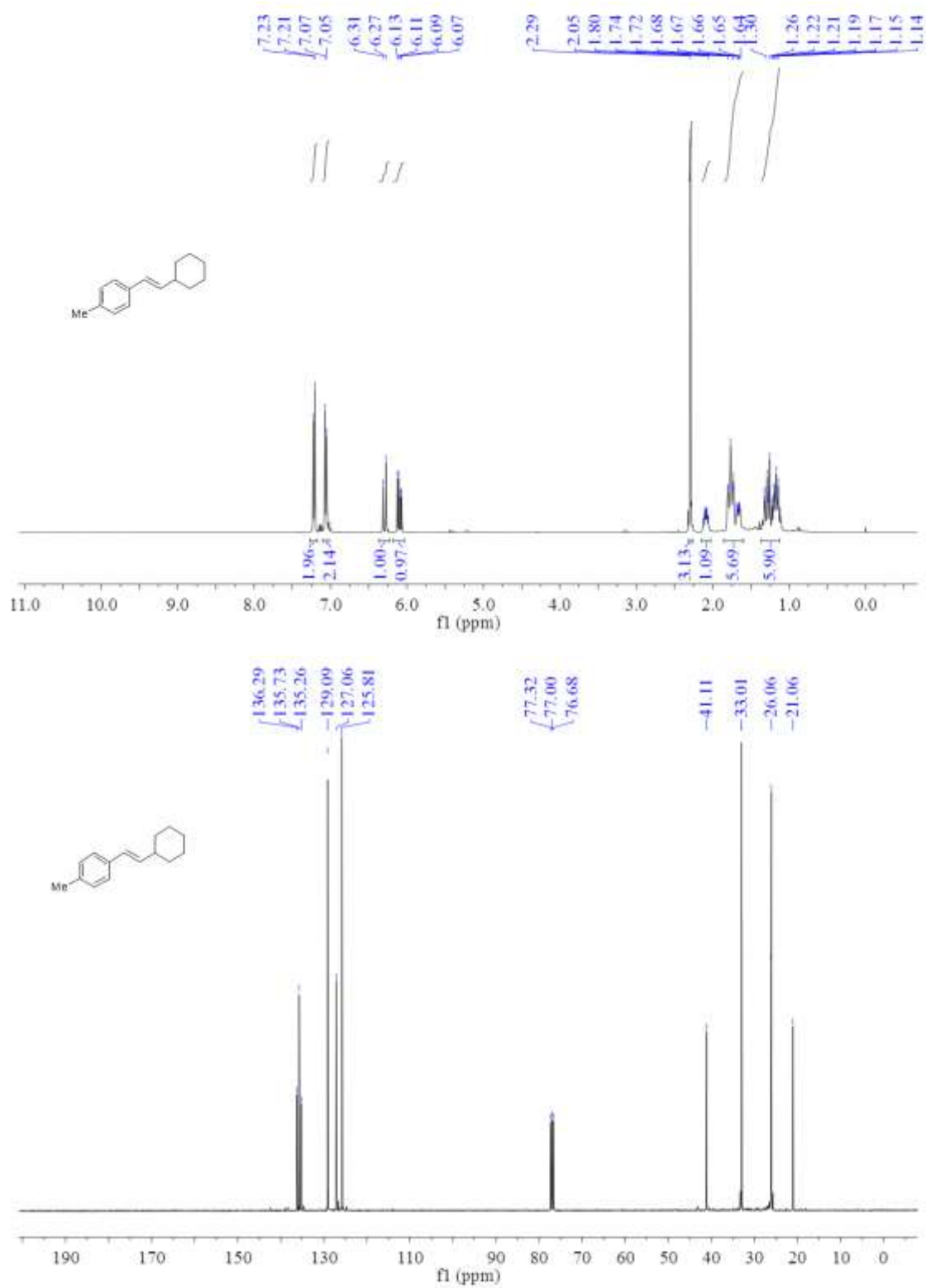
(E)-Styrylcyclooctane(4f, known compound): Colorless oil, ^1H NMR (400 MHz, CDCl_3) δ 7.33–7.28 (m, 2H), 7.24 (dd, $J = 10.4, 4.9$ Hz, 2H), 7.13 (dd, $J = 10.2, 4.3$ Hz, 1H), 6.30 (d, $J = 16.0$ Hz, 1H), 6.18 (dd, $J = 15.9, 7.4$ Hz, 1H), 2.43–2.30 (m, 1H), 1.79–1.44 (m, 14H). ^{13}C NMR (100 MHz, CDCl_3) δ 138.08, 137.61, 128.35, 126.88, 126.58, 125.89, 41.29, 31.83, 27.43, 25.98, 25.06.

3. ^1H and ^{13}C NMR spectra

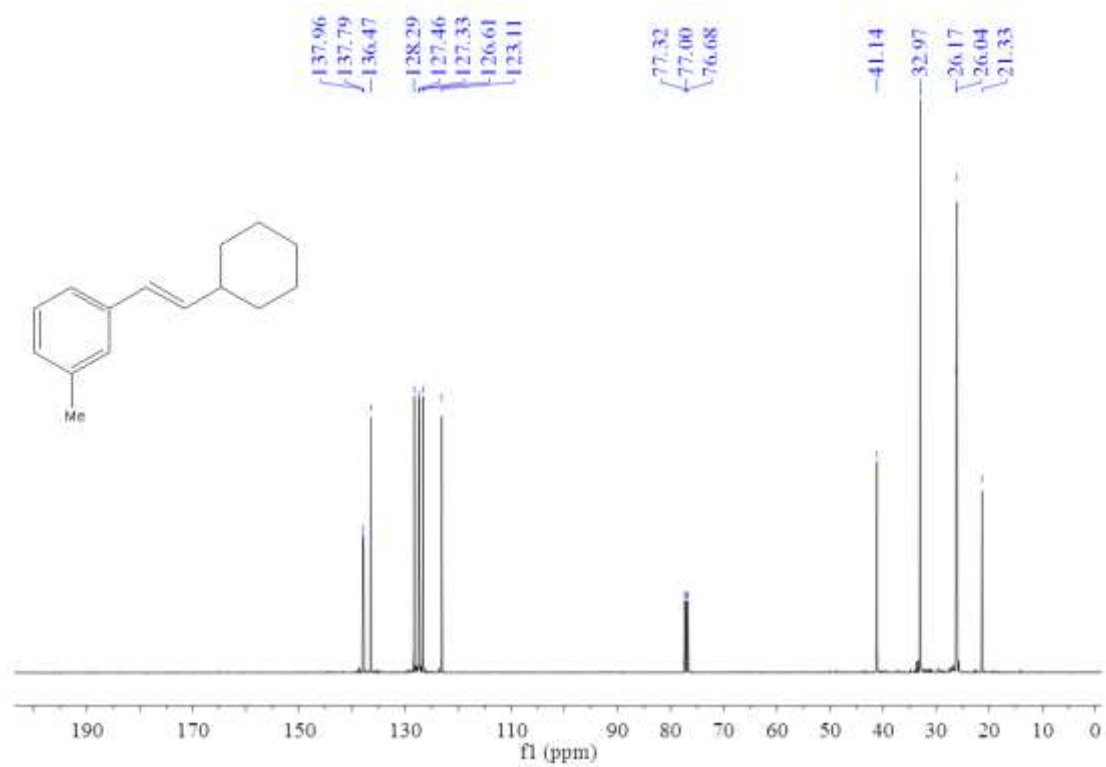
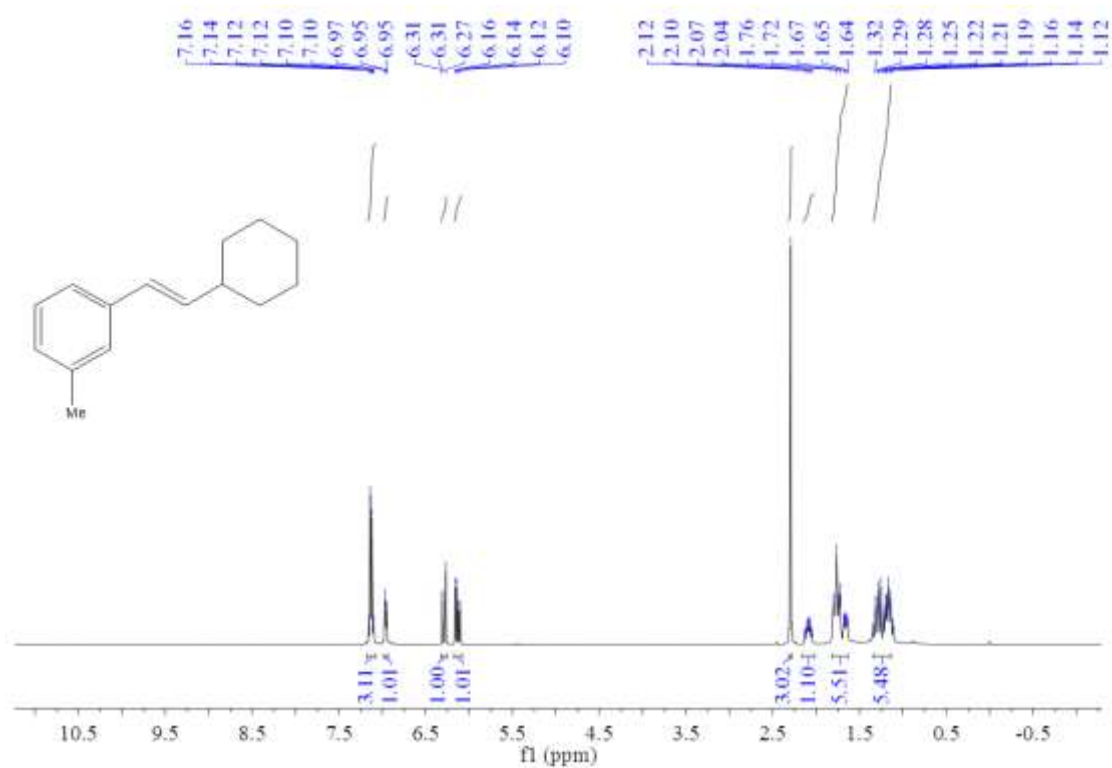
^1H - and ^{13}C NMR of 3a



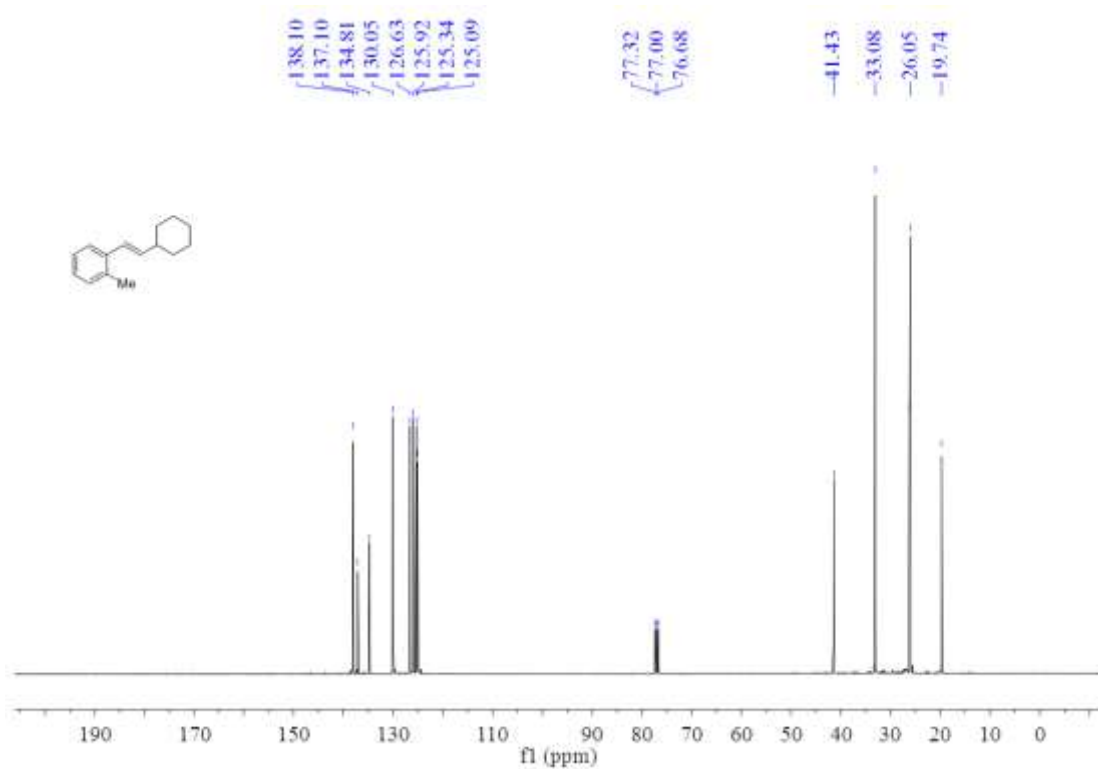
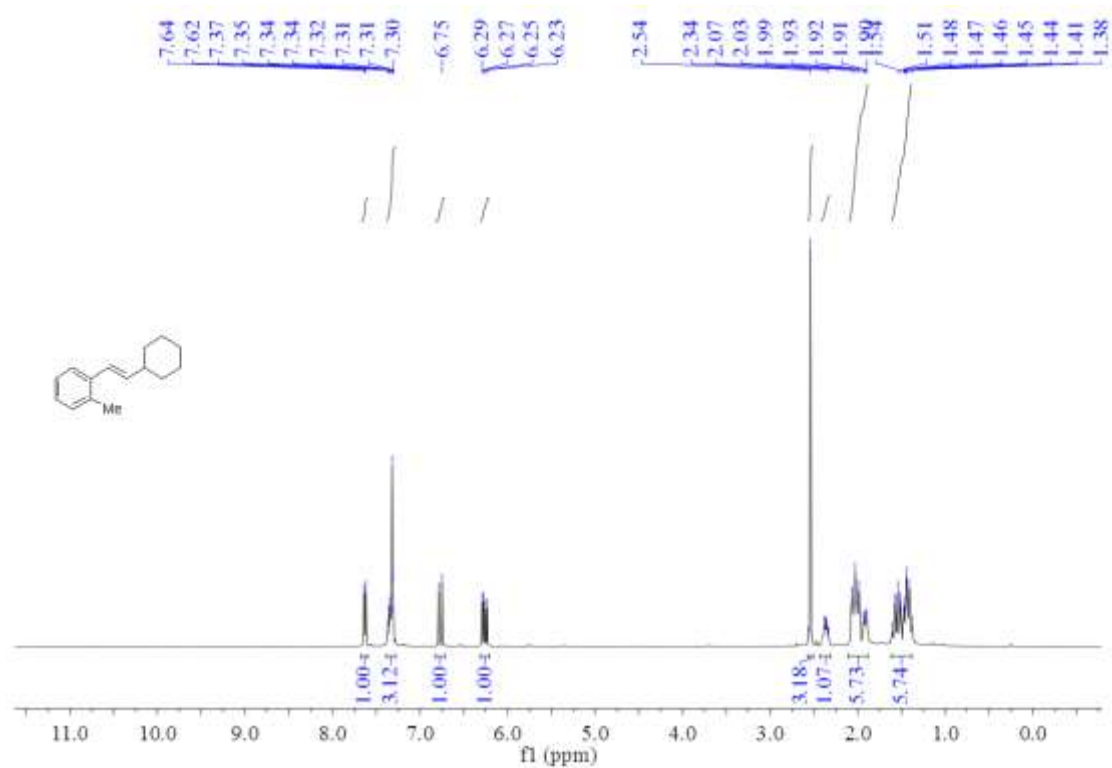
¹H- and ¹³C NMR of 3b



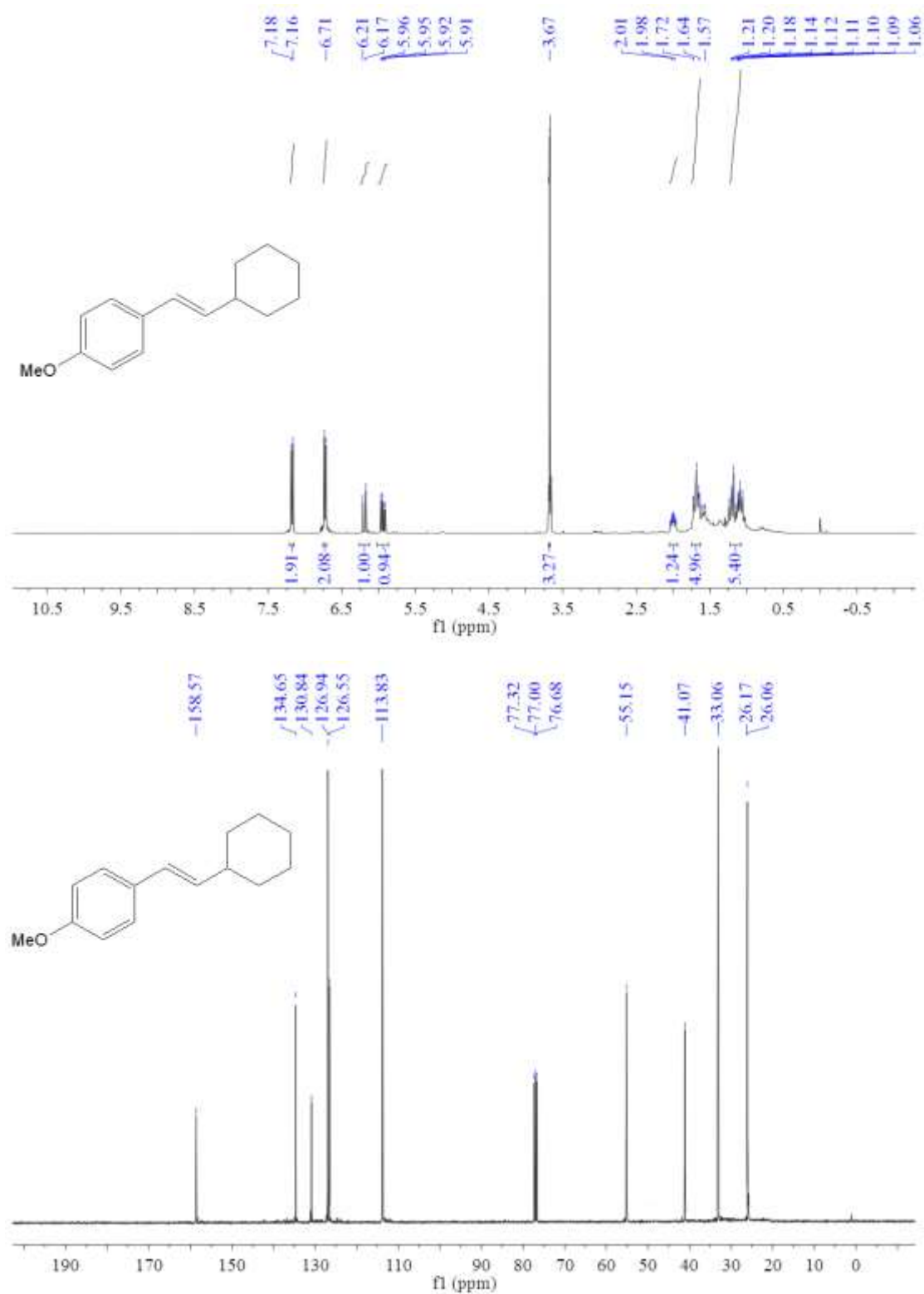
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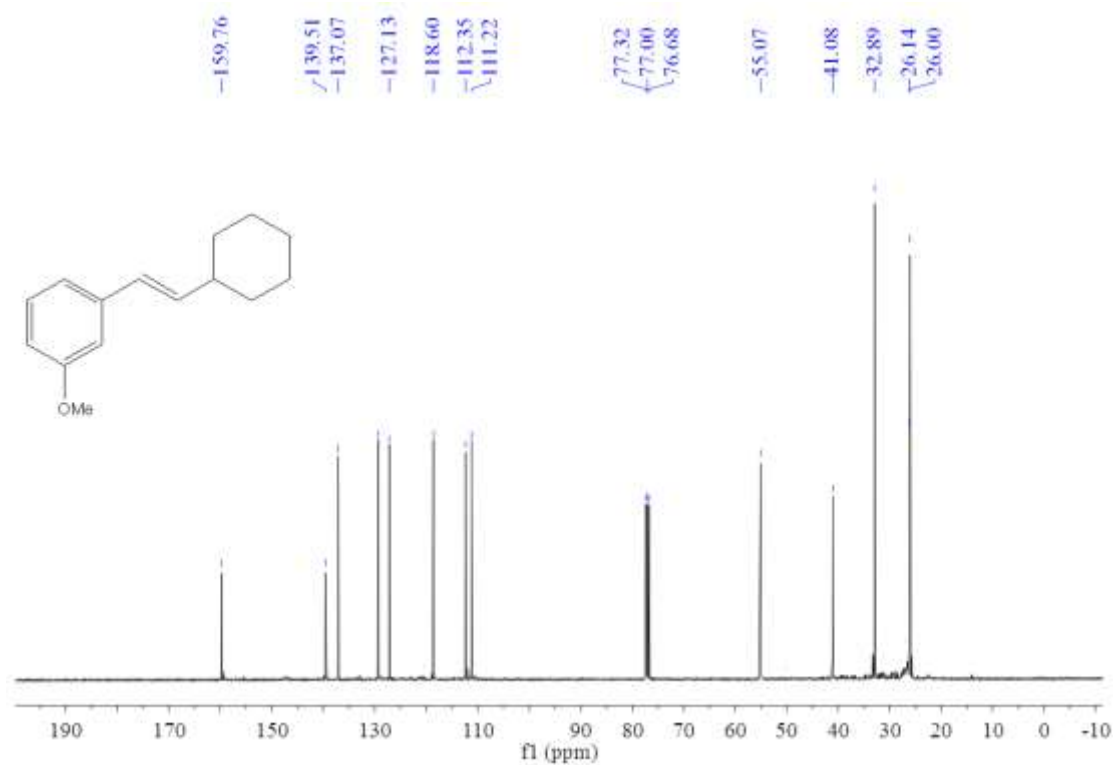
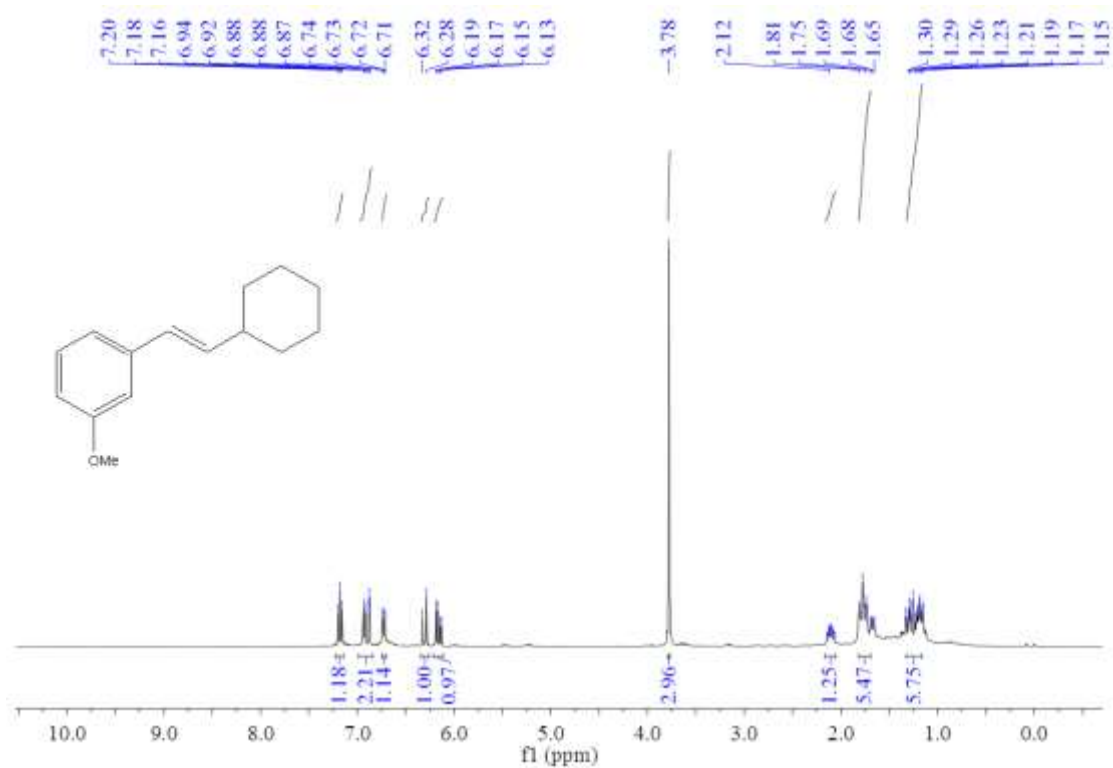
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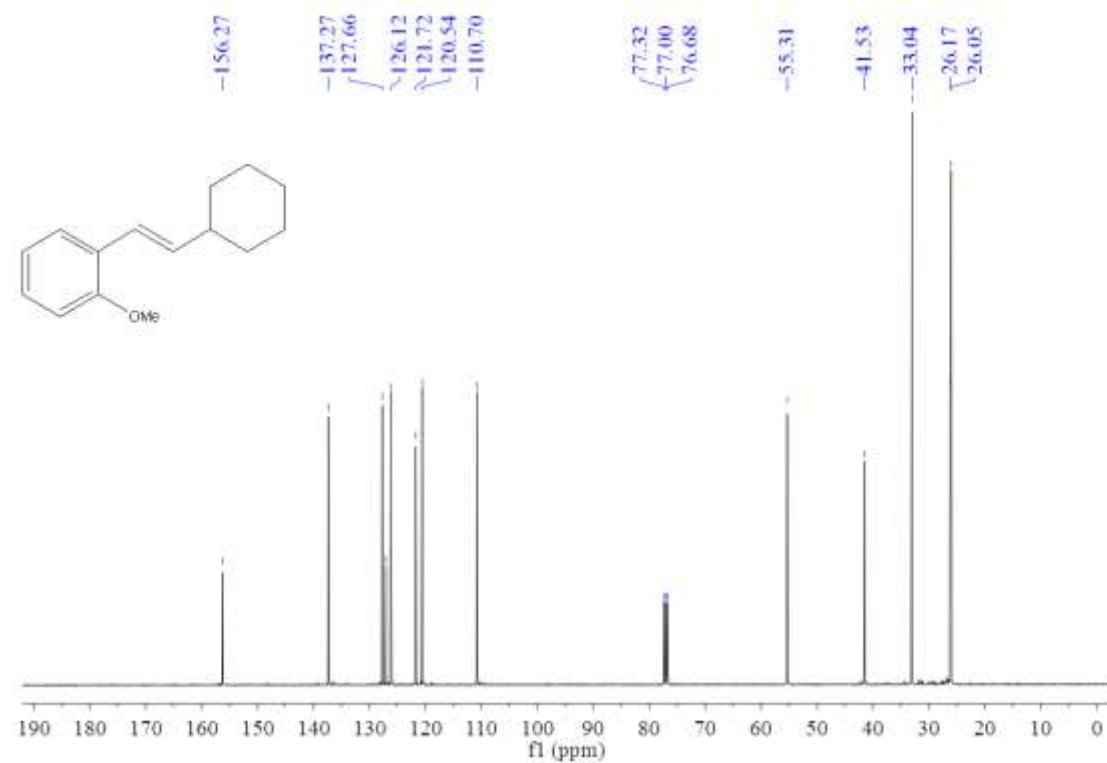
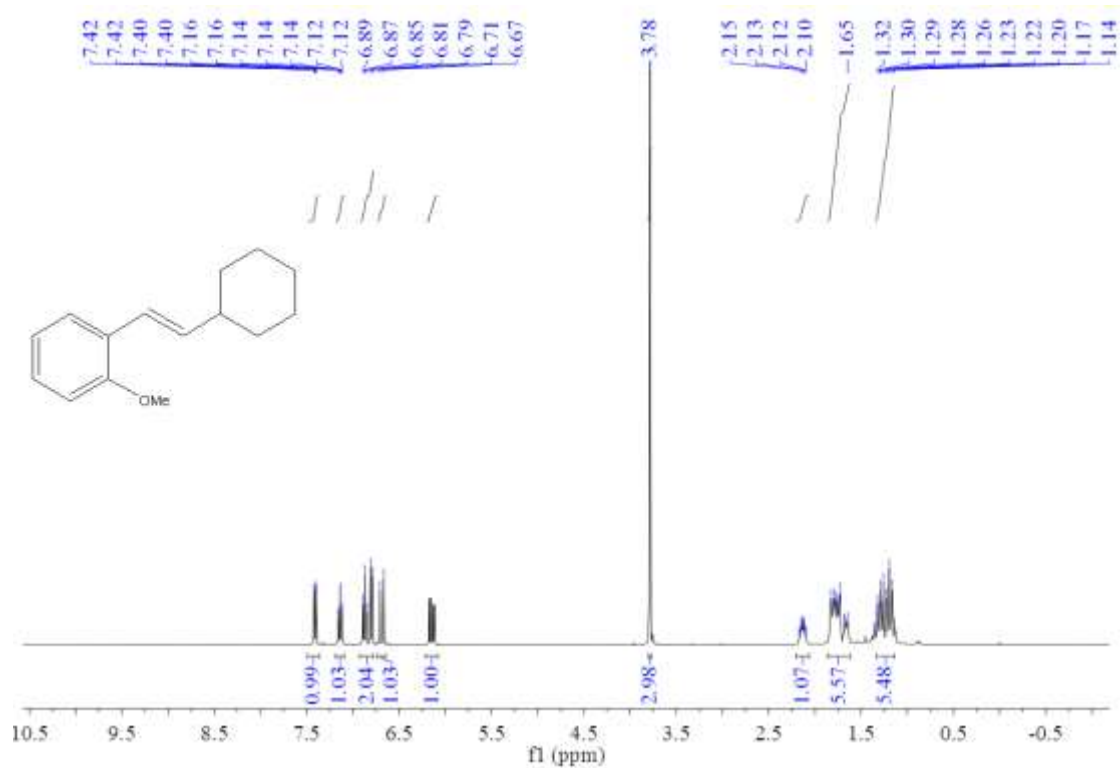
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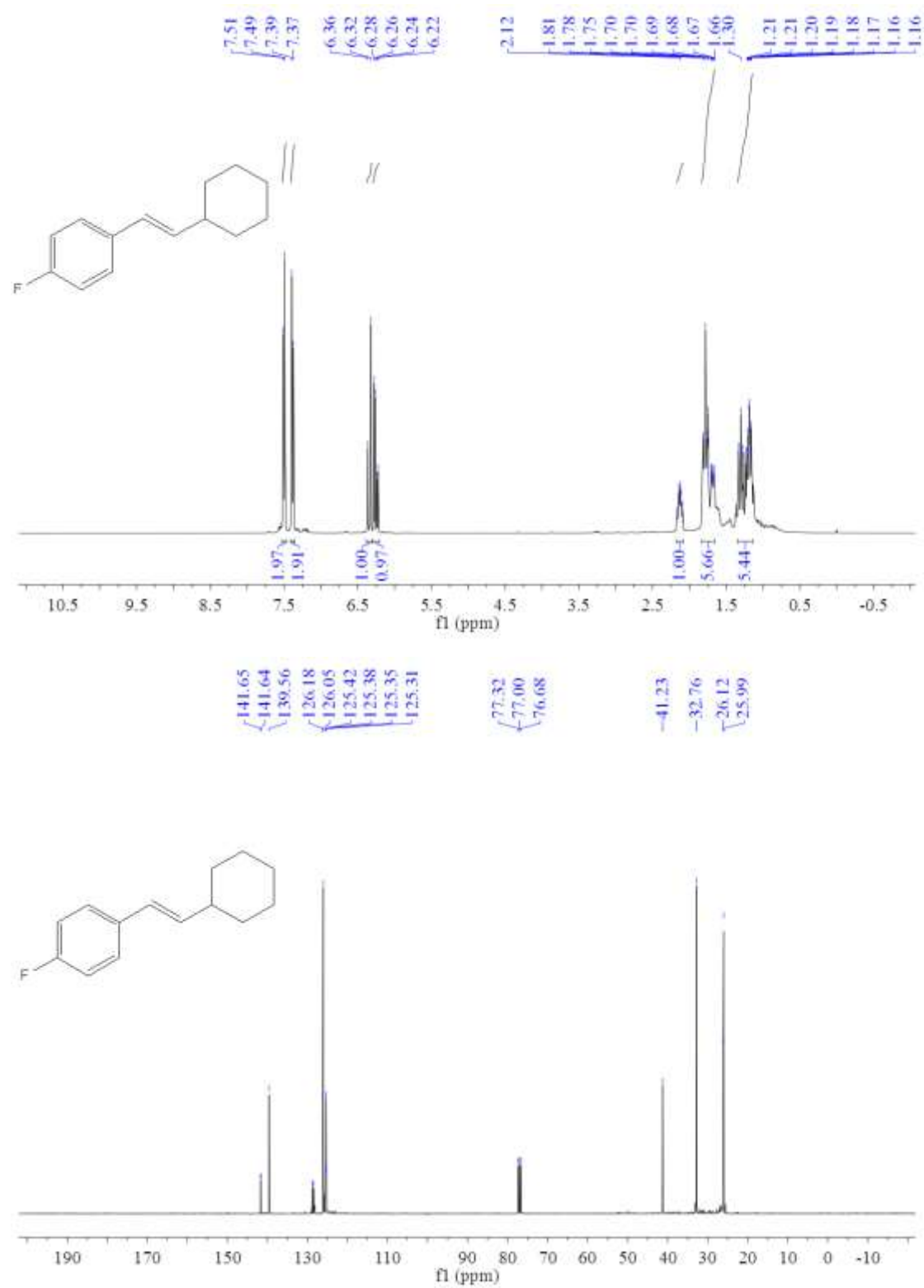
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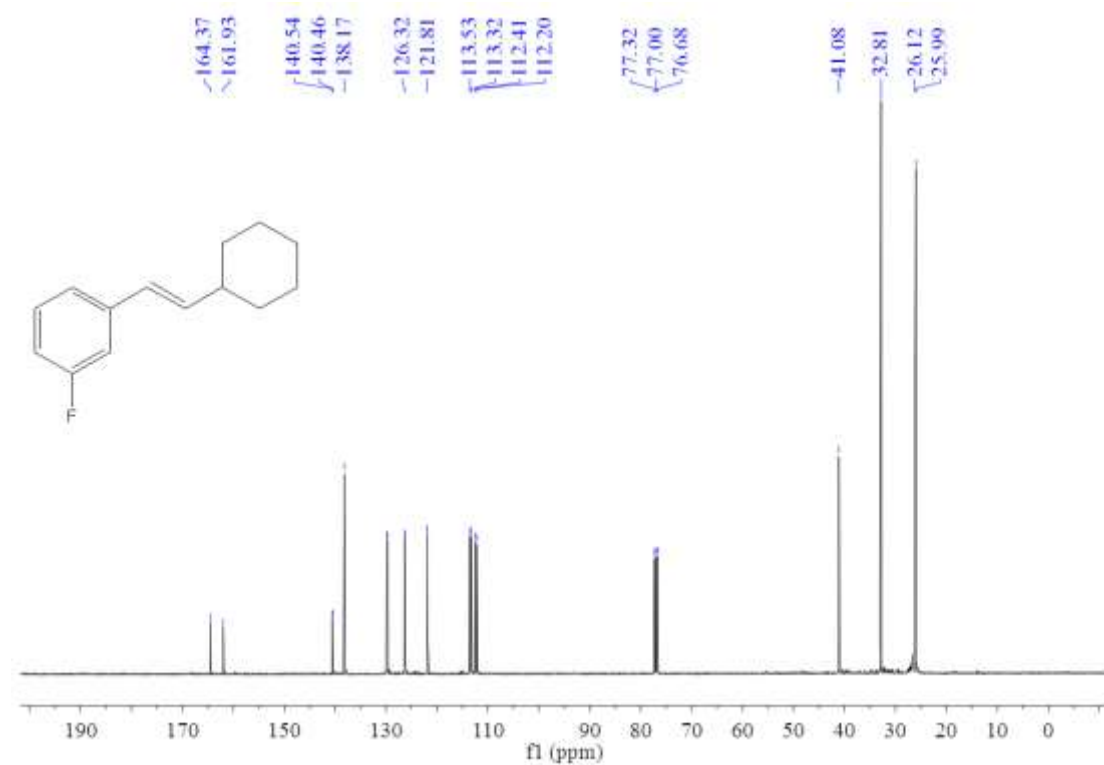
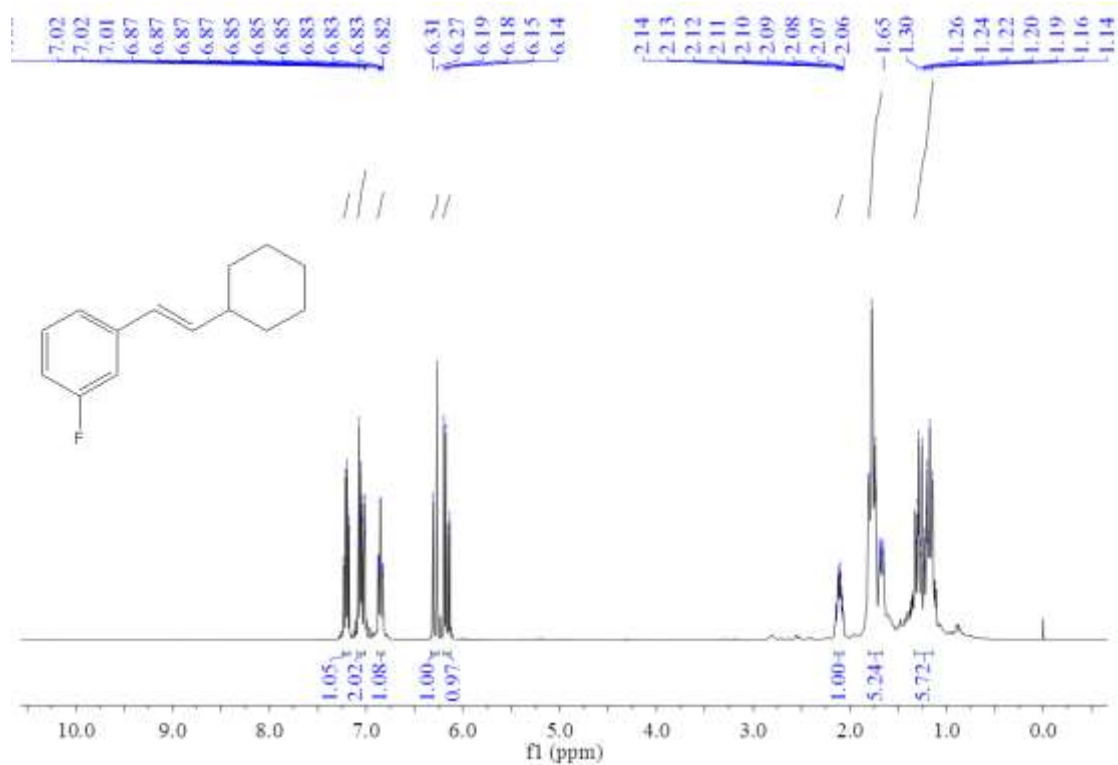
¹H- and ¹³C NMR of 3g



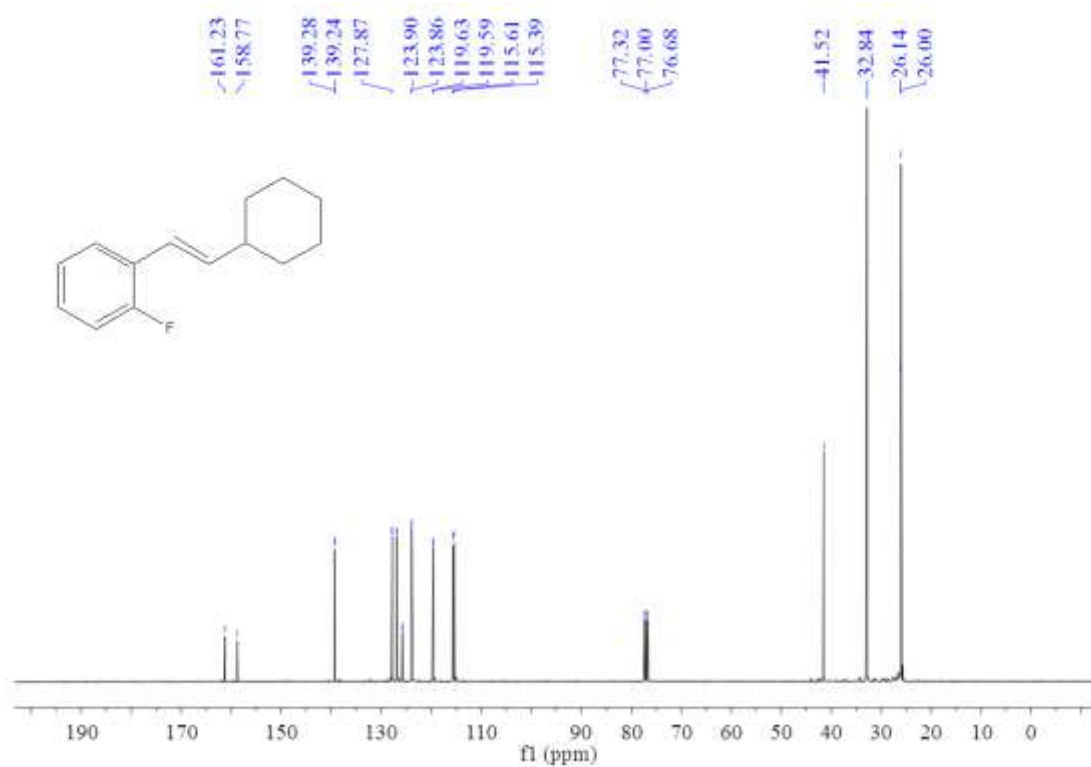
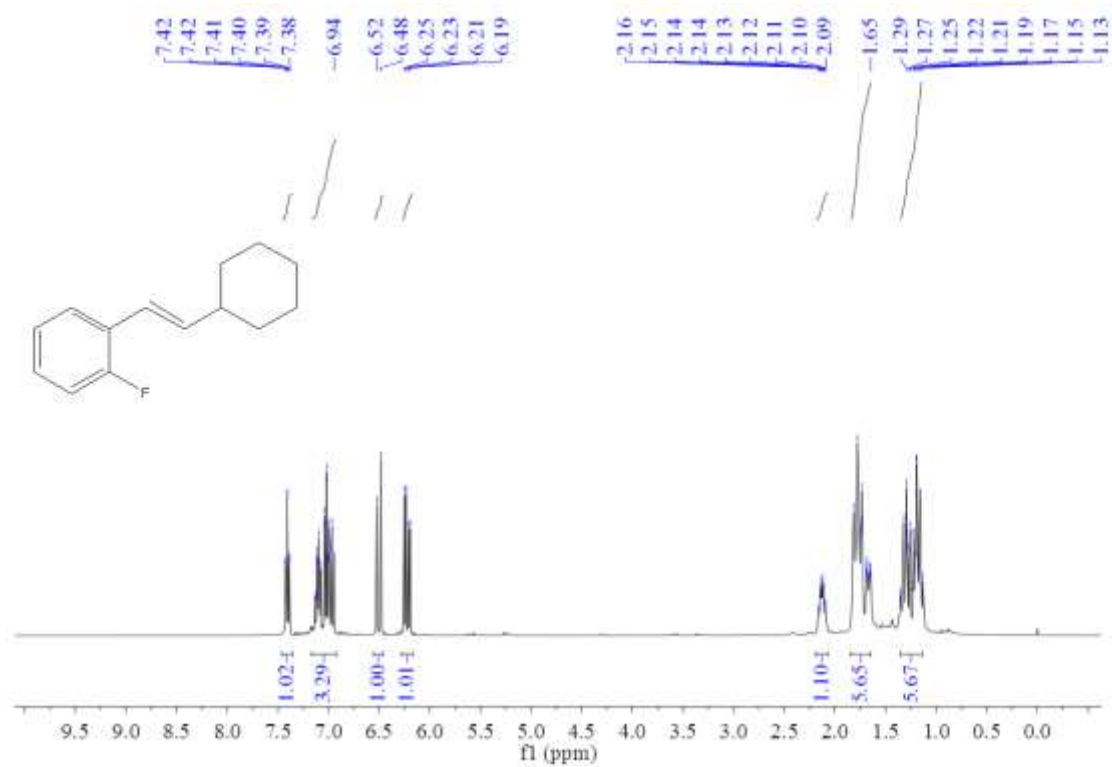
¹H- and ¹³C NMR of 3h



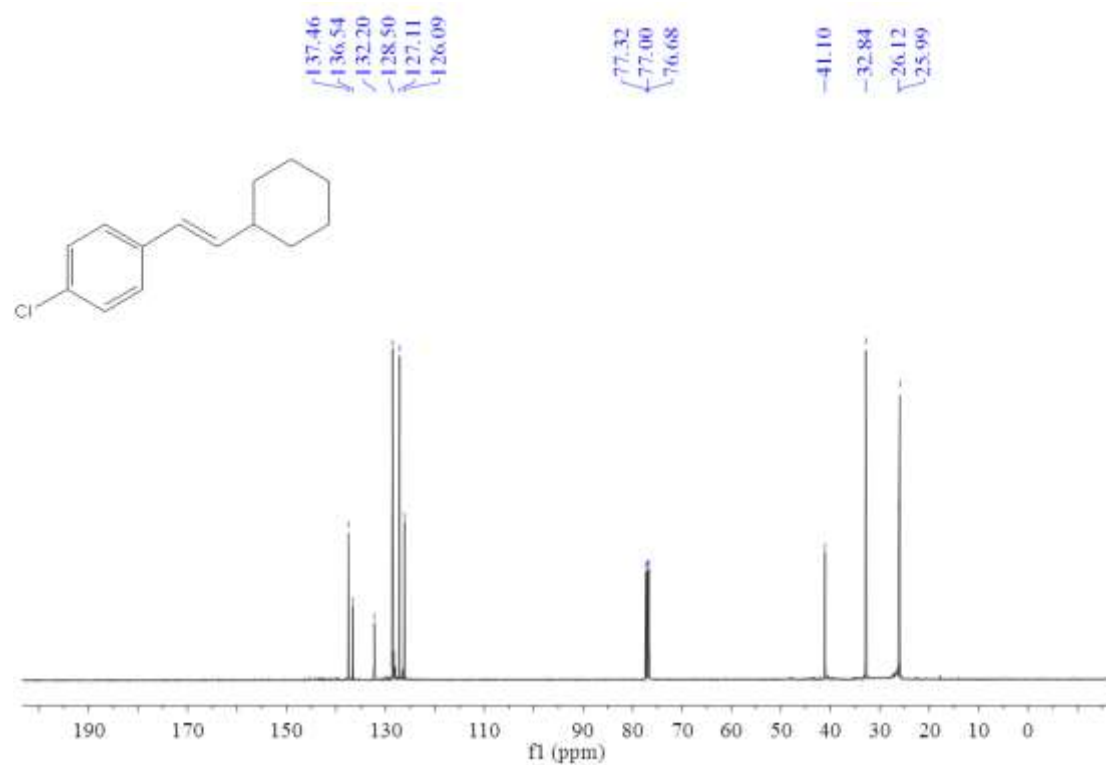
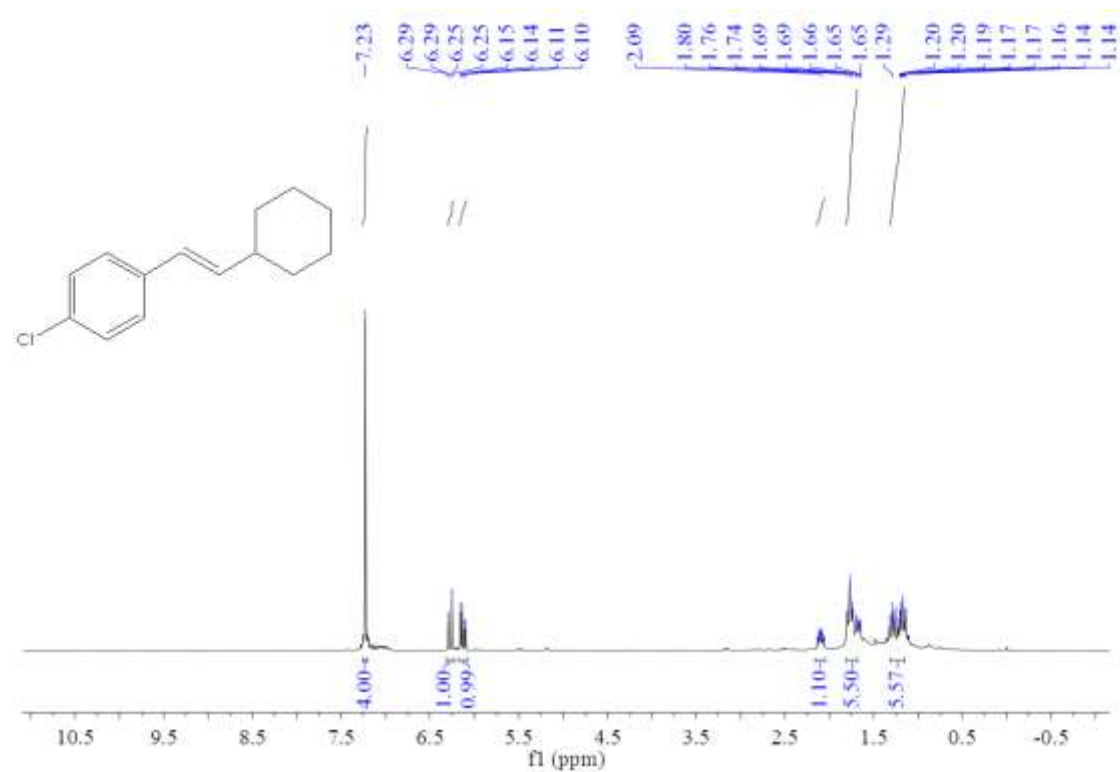
¹H- and ¹³C NMR of 3i



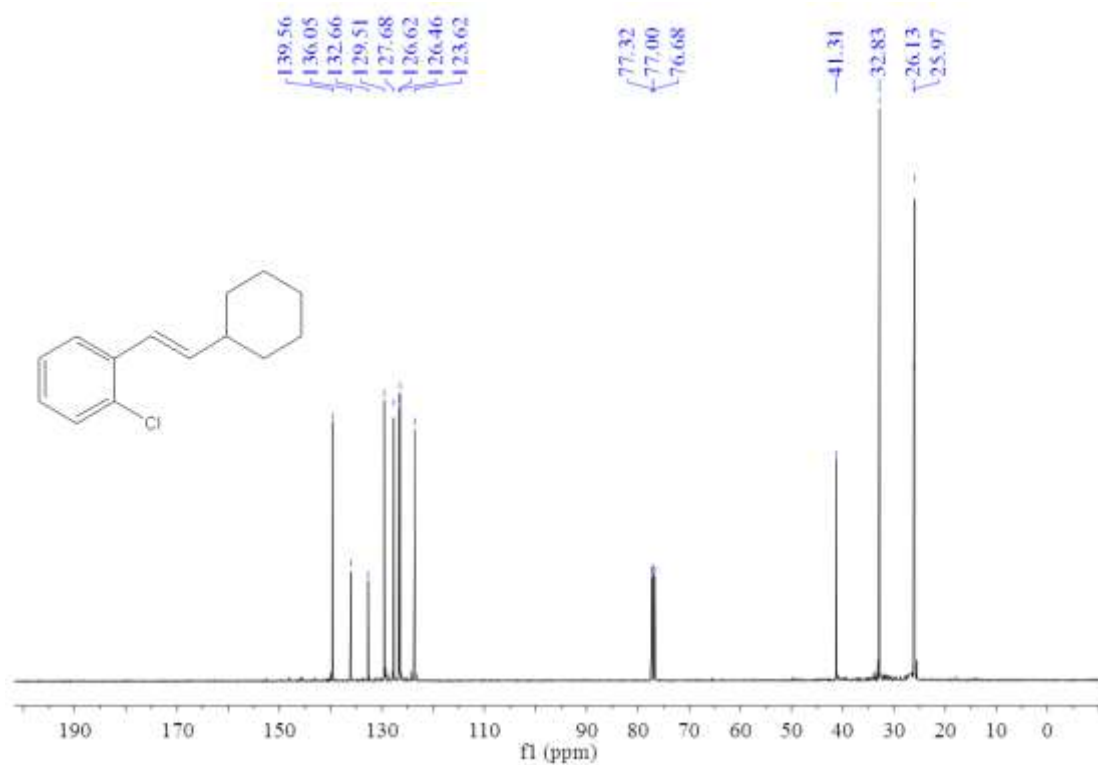
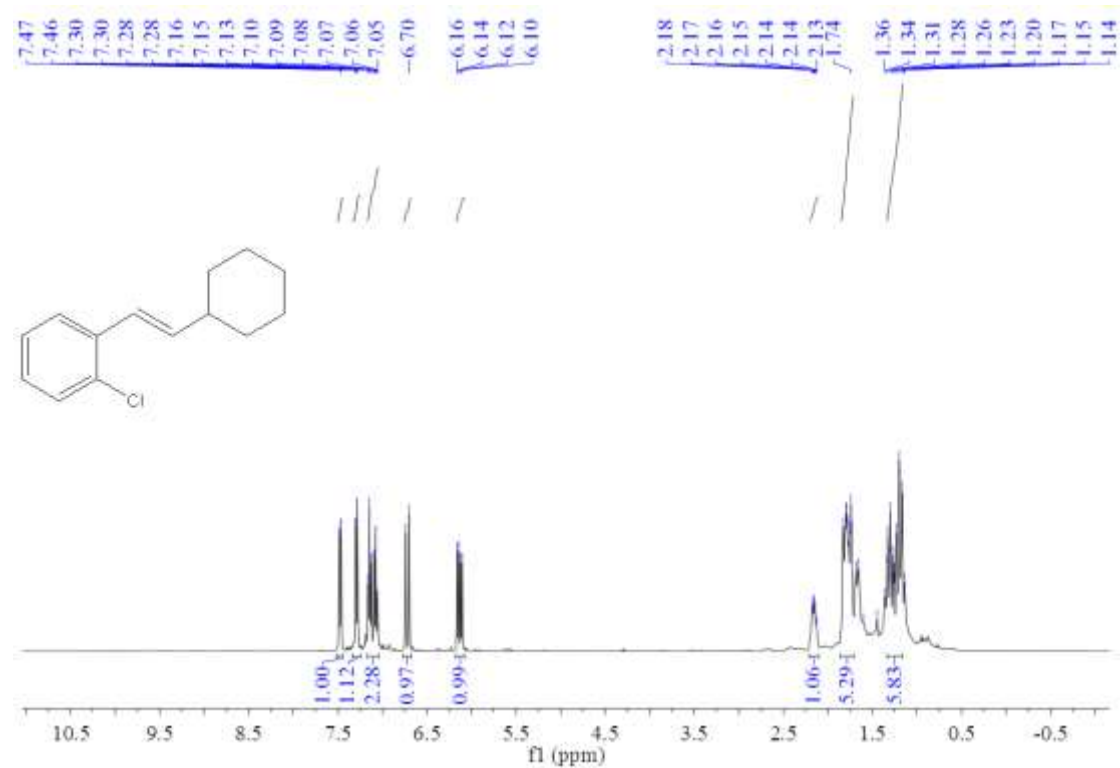
¹H- and ¹³C NMR of 3j



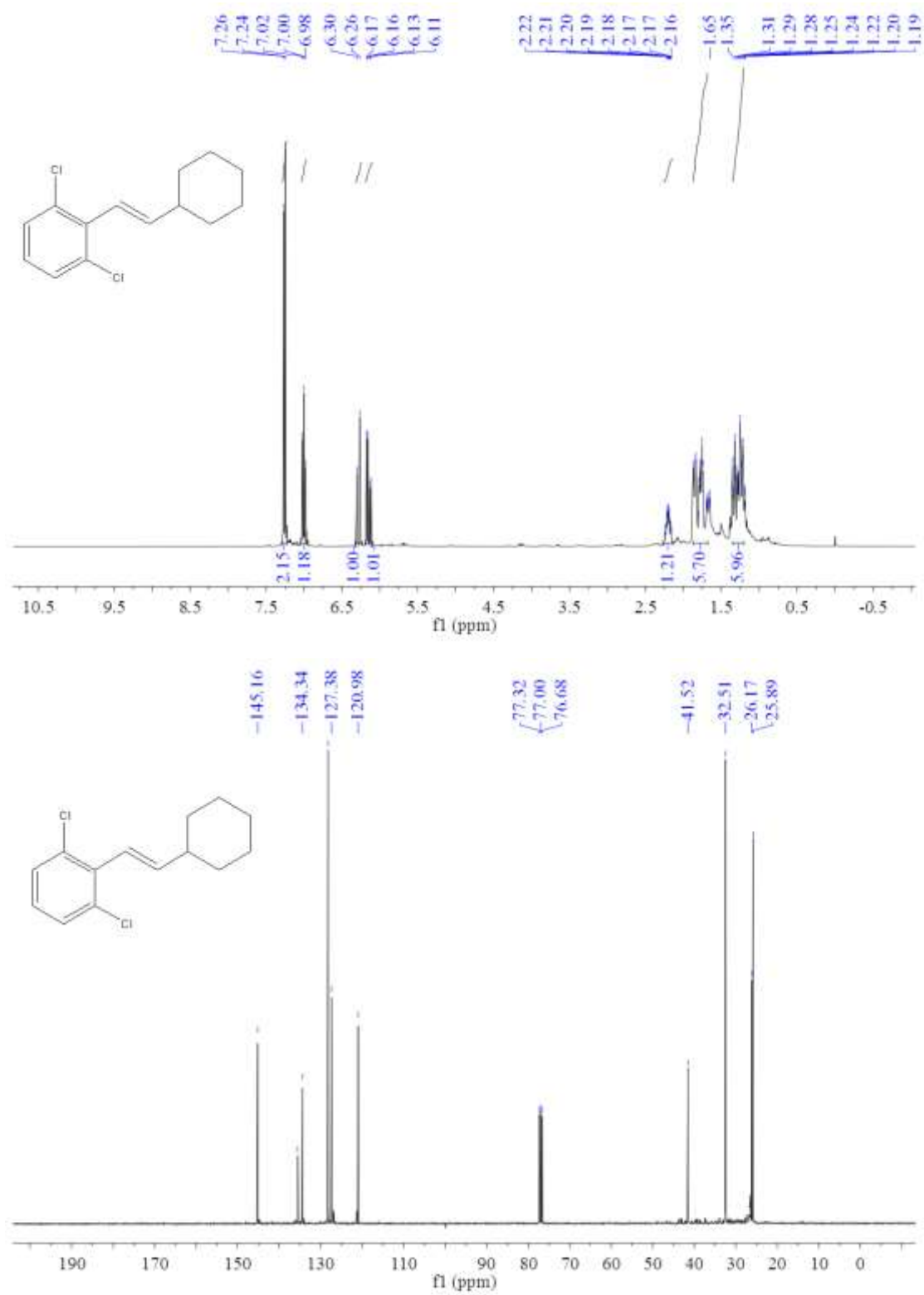
¹H- and ¹³C NMR of 3k



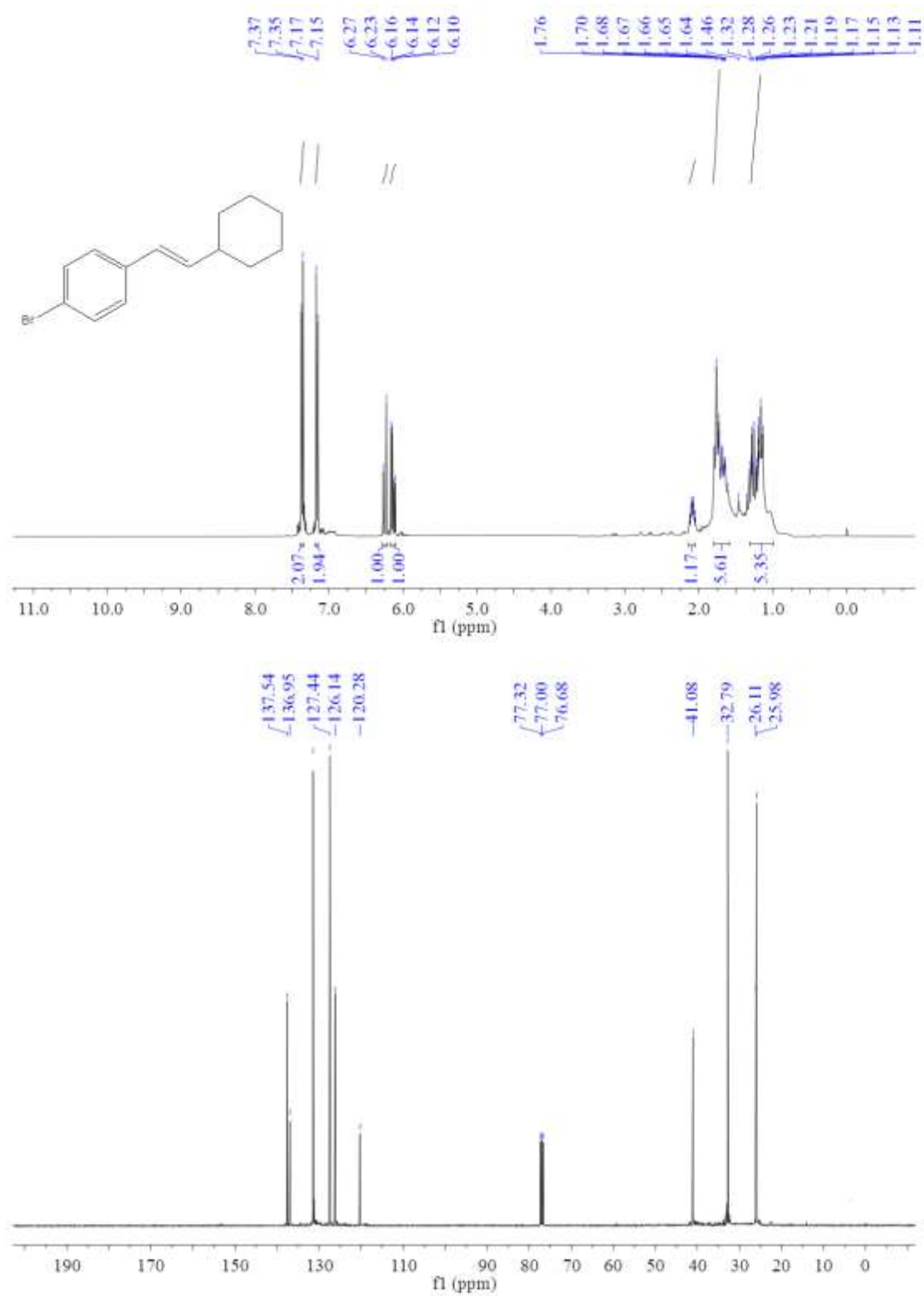
^1H - and ^{13}C NMR of 3l



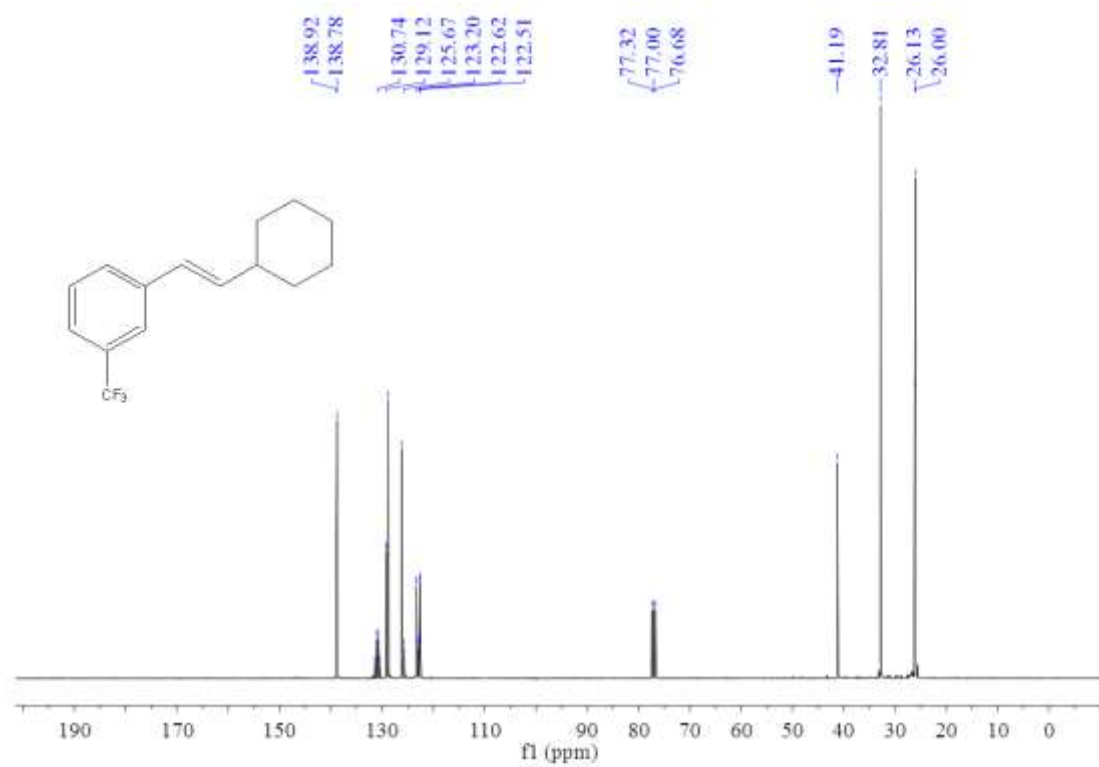
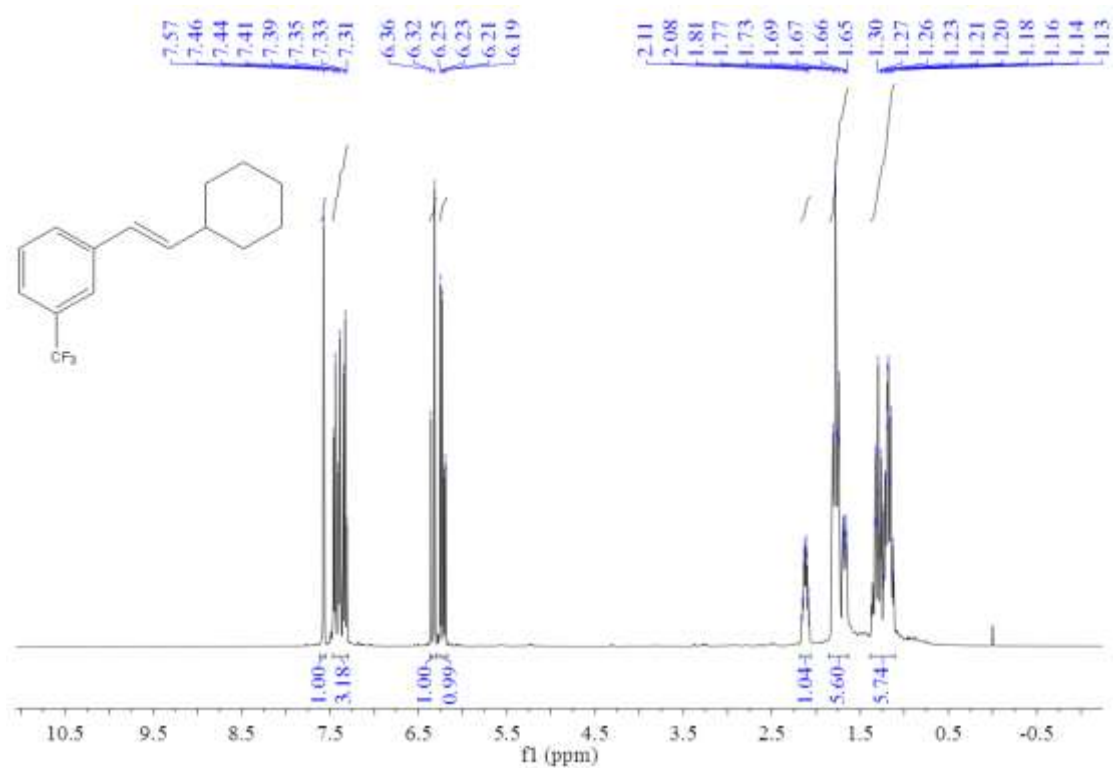
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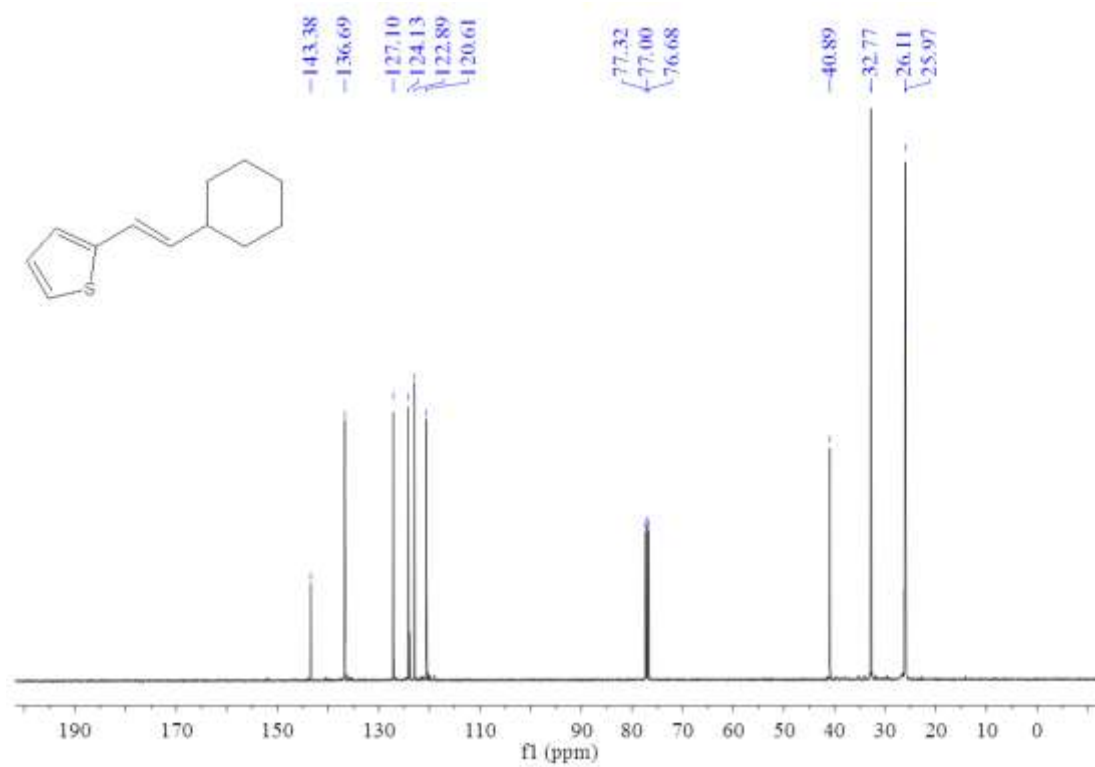
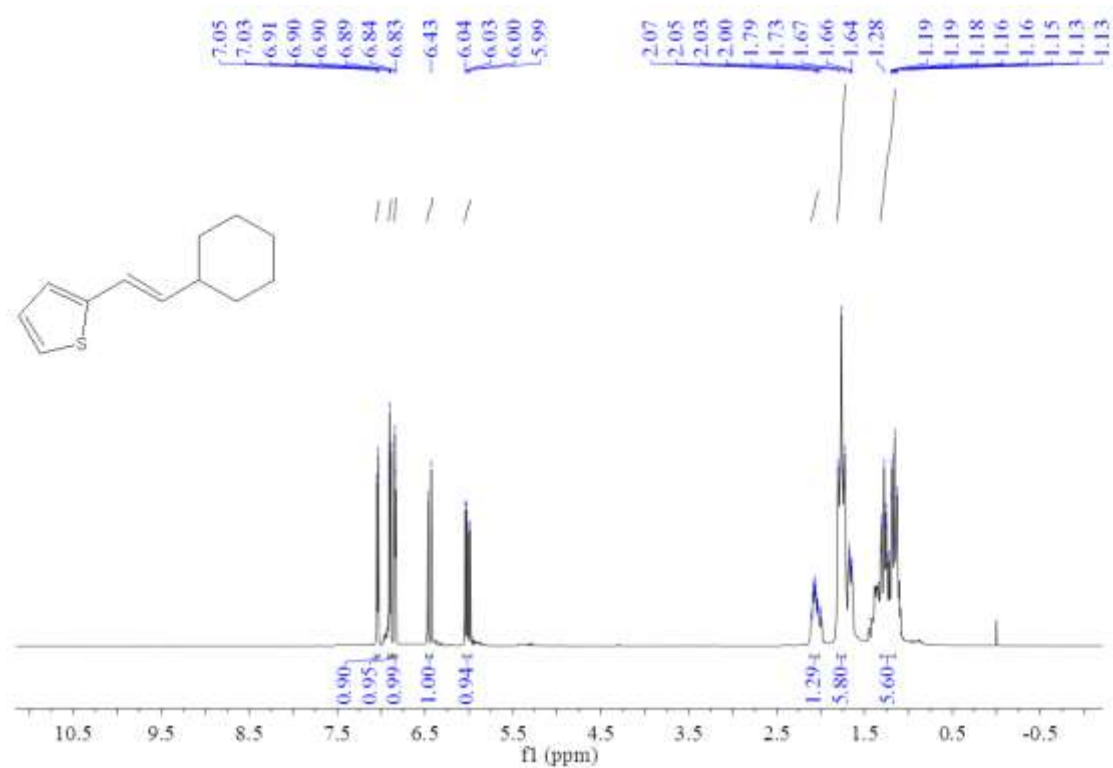
¹H- and ¹³C NMR of 3n



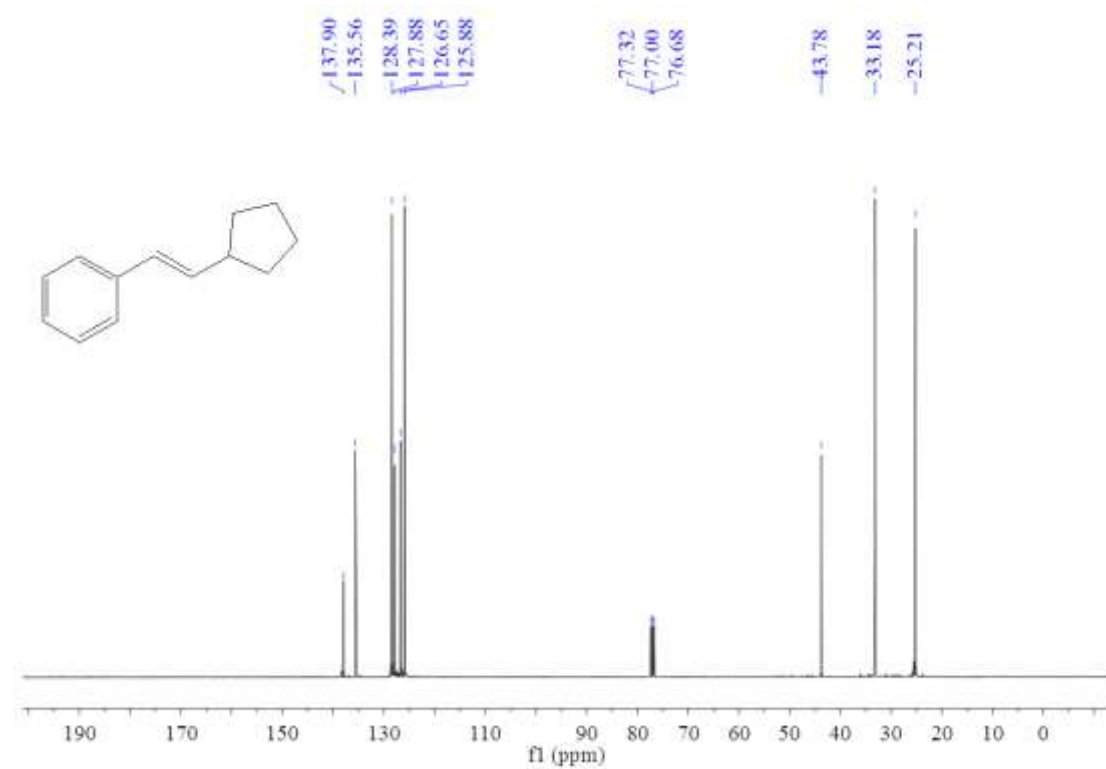
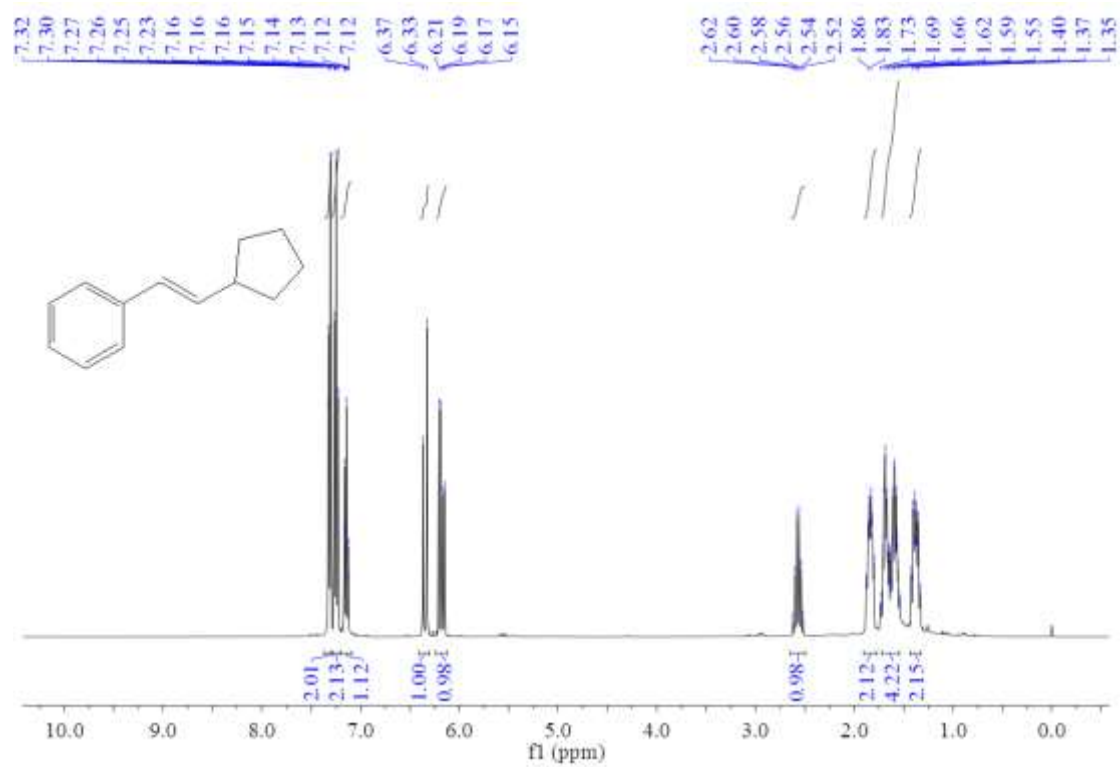
¹H- and ¹³C NMR of 3o



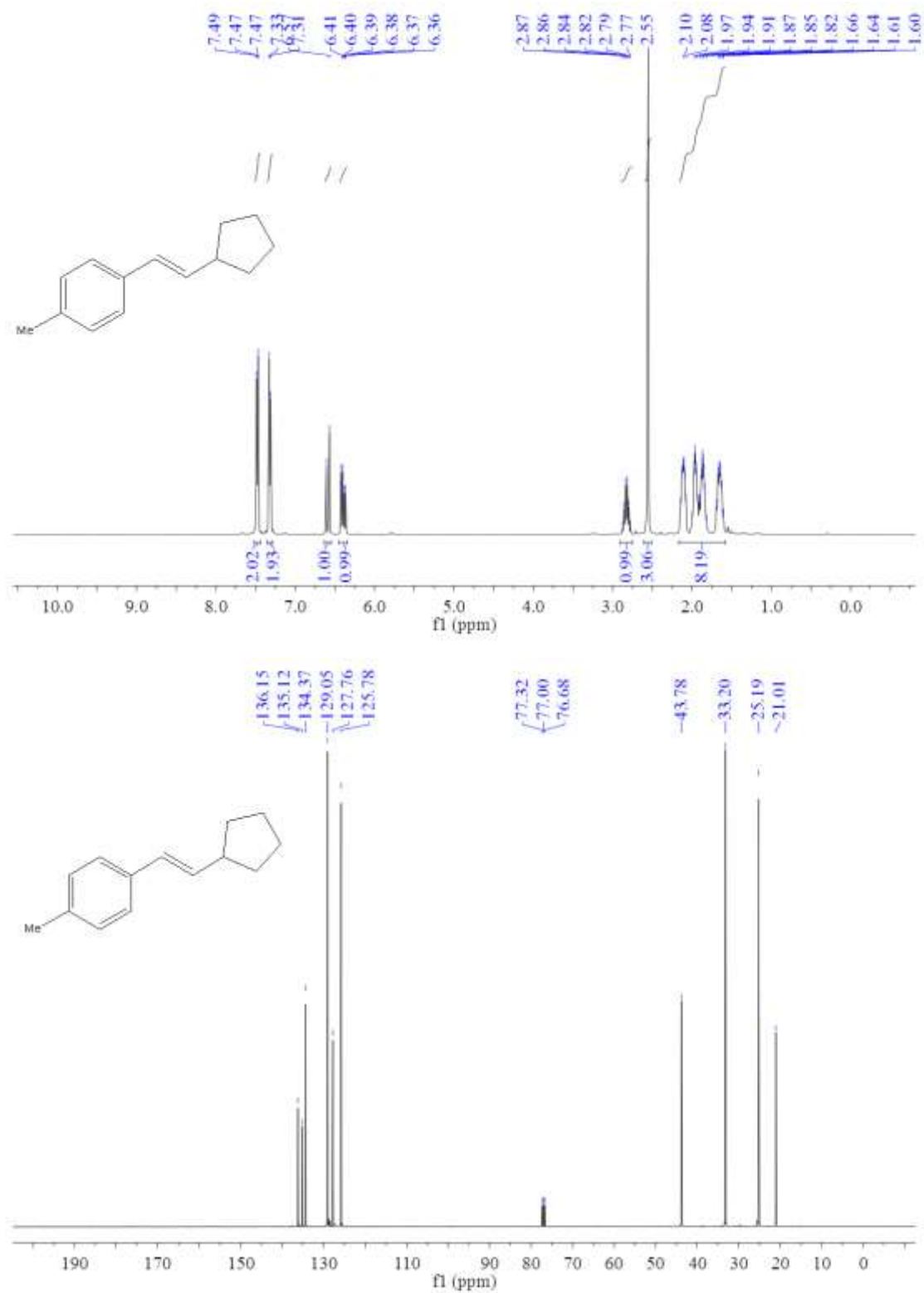
¹H- and ¹³C NMR of 3p



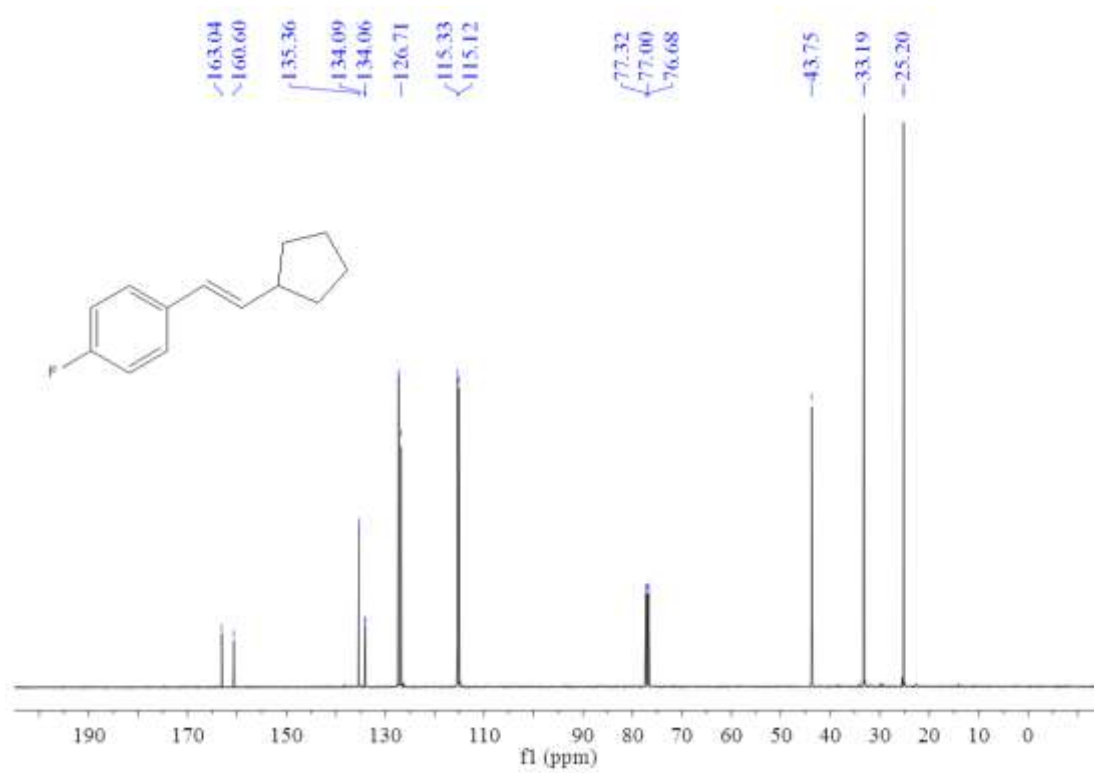
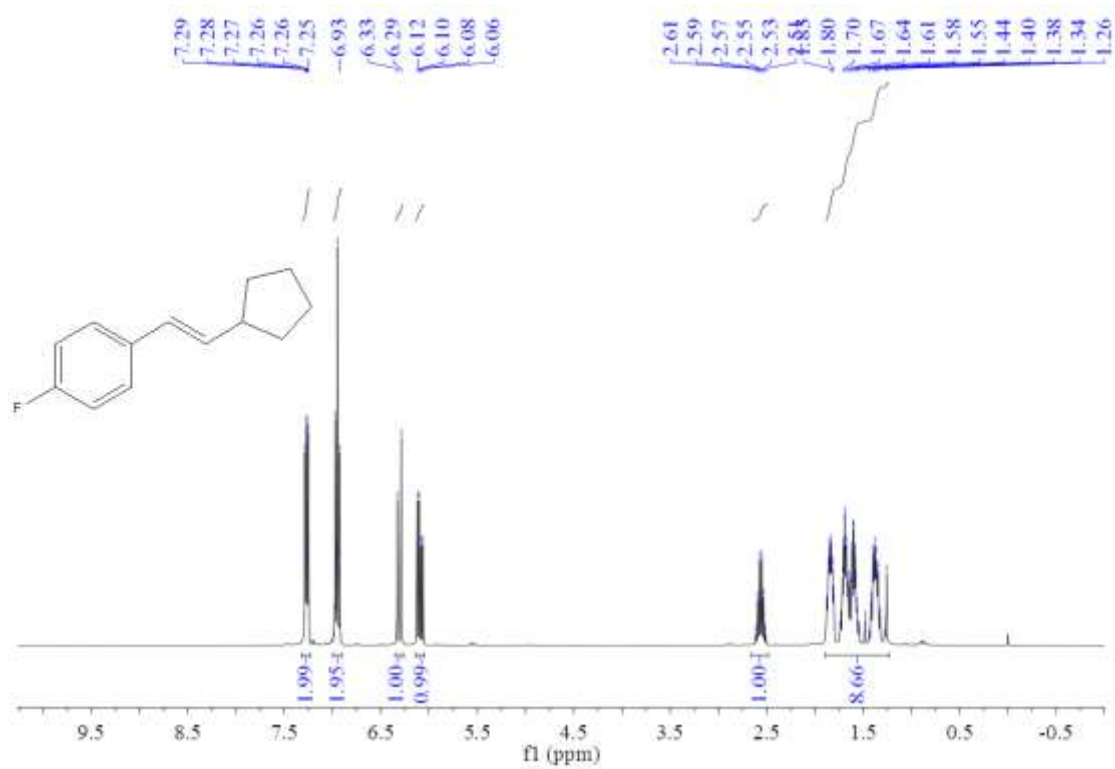
¹H- and ¹³C NMR of 4a



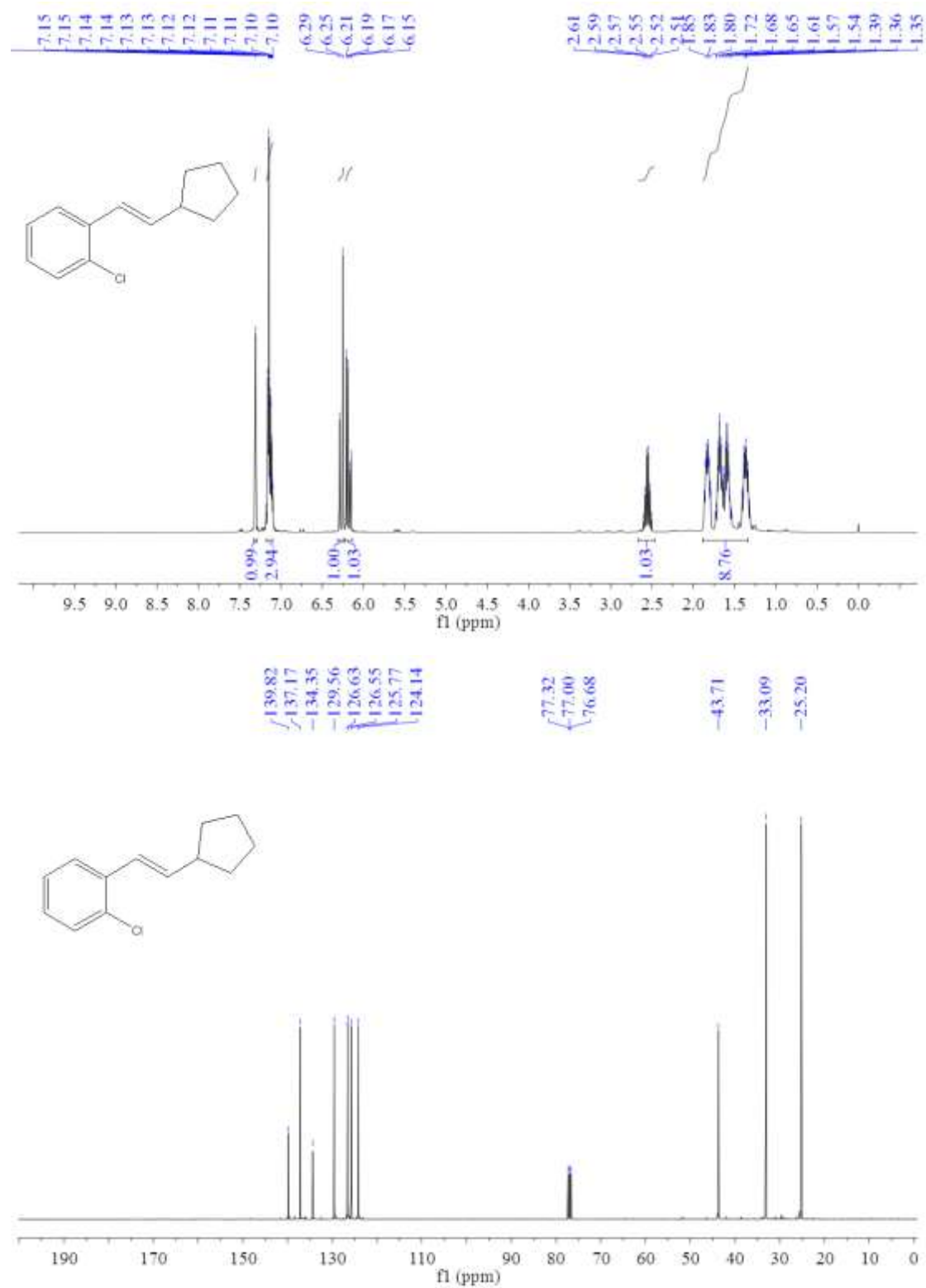
¹H- and ¹³C NMR of 4b



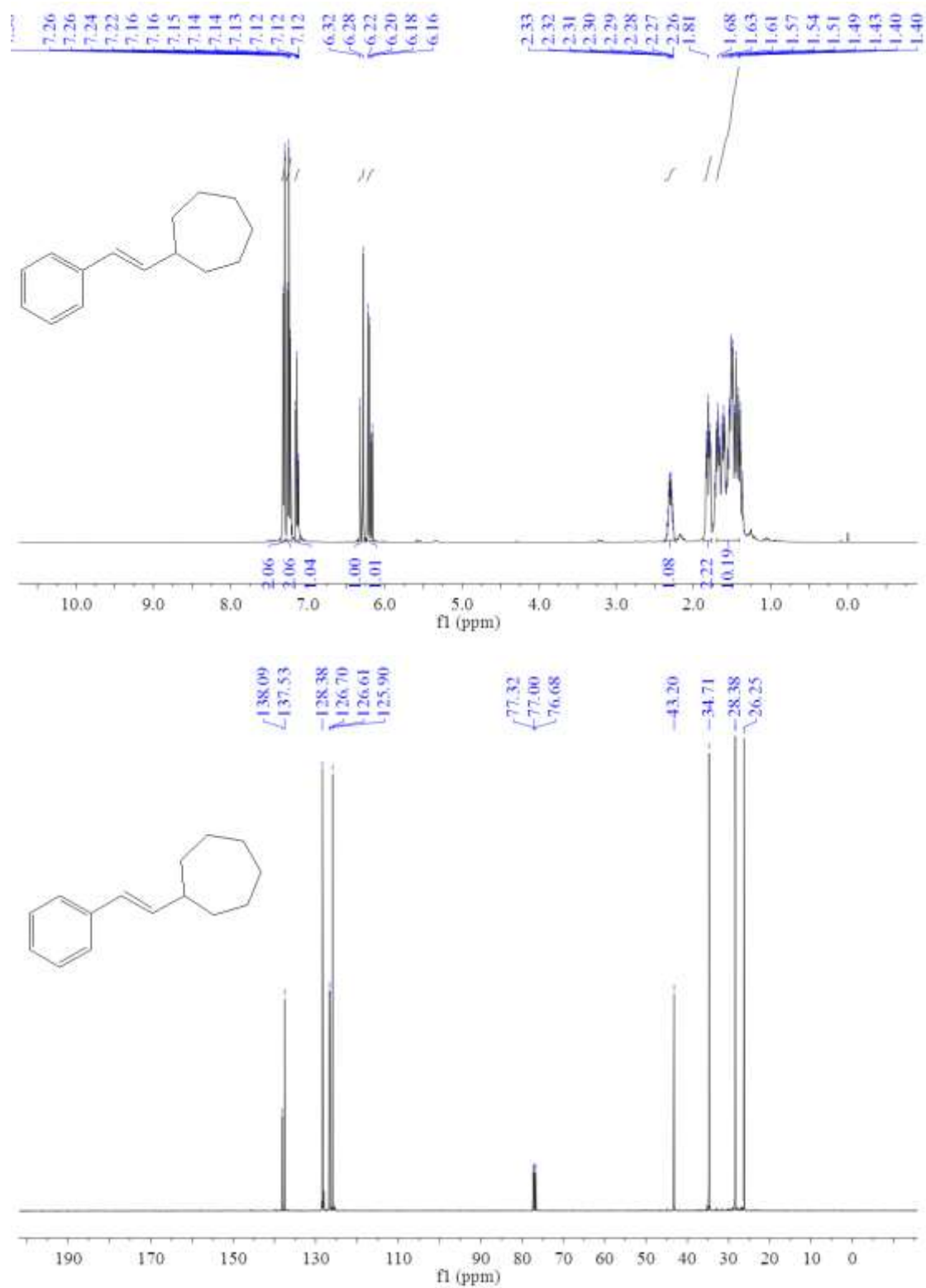
¹H- and ¹³C NMR of 4c



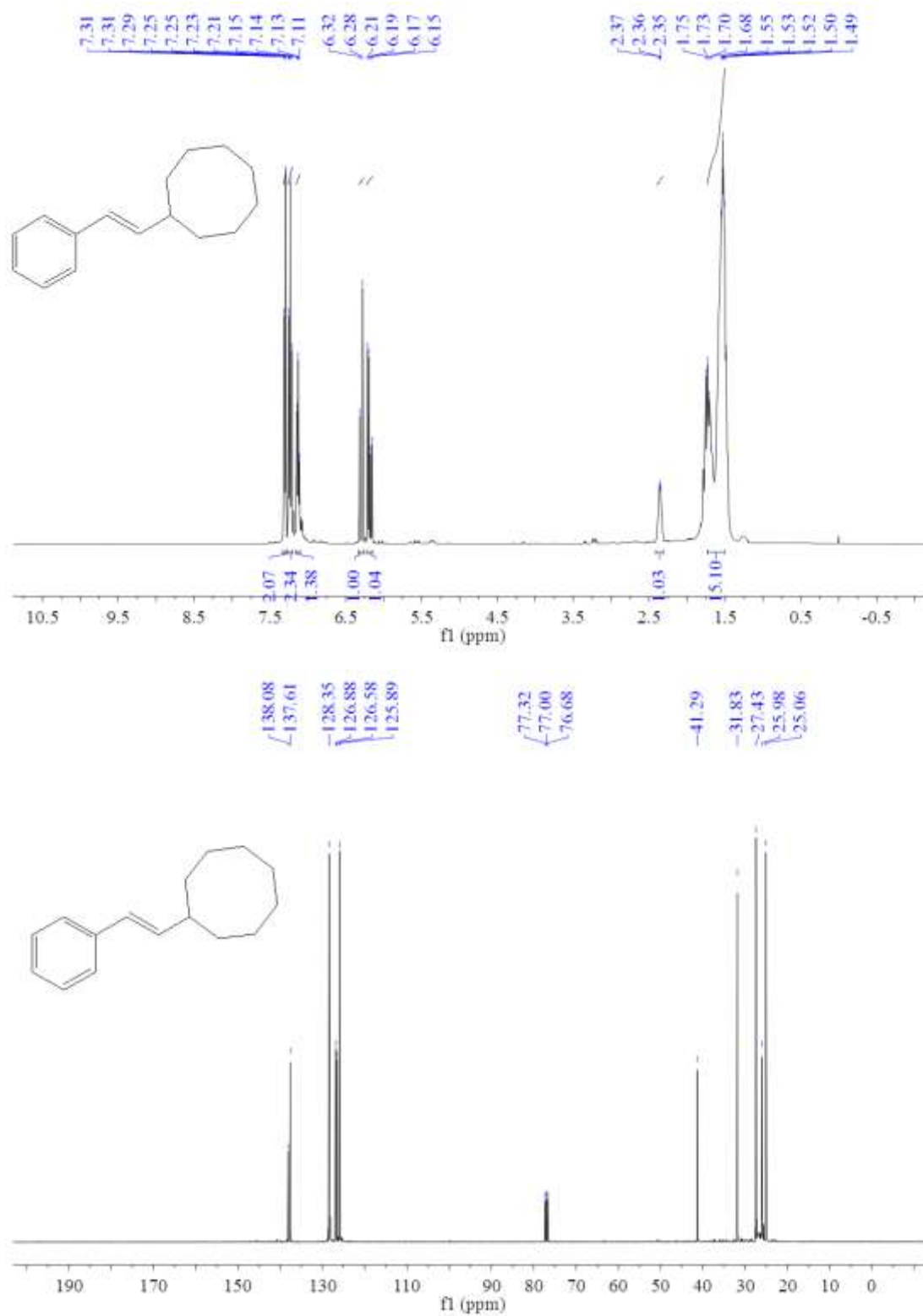
¹H- and ¹³C NMR of 4d



¹H- and ¹³C NMR of 4e



¹H- and ¹³C NMR of 4f



4. References

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