

Supporting Information

Oxidative Difunctionalization of Alkynoates through Alkylation and Migration Decarboxylative Arylation

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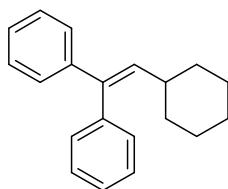
1. General information

All manipulations were carried out under air atmosphere. Commercially available reagents were used as received without purification. Column chromatography was carried out on silica gel (300–400 mesh). Analytical thin-layer chromatography was performed on glass plates of Silica Gel GF-254 with detection by UV. ^1H and ^{13}C NMR spectra were recorded on a Bruker AVANCE 400M 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 point determination was taken on a Melt-Temp apparatus (X-4) from Beijing Fukai Electro-optic Instrument Plant and was uncorrected.

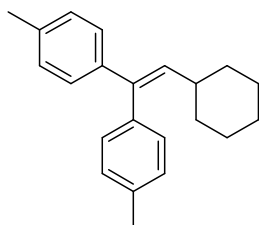
2. General procedure for the cascade radical reaction

A sealable reaction tube equipped with a magnetic stirrer bar was charged with phenyl 3-phenylpropiolate (44.4 mg, 0.2 mmol), DTBP (di-*tert*-butyl peroxide, 0.4 mmol, 74 μL), and cyclohexane (2.0 mL, 10–20 mmol). The rubber septum was then replaced by a Teflon-coated screw cap, and the reaction vessel placed in an oil bath at 120 $^\circ\text{C}$. After stirring the mixture at this temperature for 24 h, it was cooled to room temperature and diluted with ethyl acetate, washed with water, dried over MgSO_4 . After the solvent was removed under reduced pressure, the residue was purified by column chromatography on silica gel (pure hexane) to afford the corresponding product, (2-cyclohexylethene-1,1-diyl)dibenzene (68% yield).

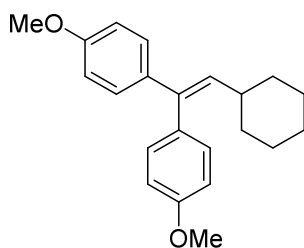
3. Characterization data for 3



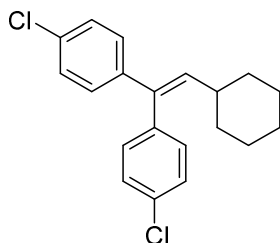
(2-cyclohexylethene-1,1-diyl)dibenzene (3aa): Light yellow oil. Yield: 35.5 mg (68%). ^1H NMR (400 MHz, CDCl_3) δ 7.40–7.27 (m, 3H), 7.27–7.15 (m, 7H), 5.90 (d, J = 10.1 Hz, 1H), 2.21–2.04 (m, 1H), 1.76–1.56 (m, 5H), 1.24–1.05 (m, 5H). ^{13}C NMR (101 MHz, CDCl_3) δ 143.0, 140.6, 139.6, 136.0, 129.8, 128.1, 128.0, 127.2, 126.8, 126.7, 38.3, 33.4, 26.0, 25.6. IR (cm^{-1}): 2923, 2850, 1495, 1446, 762, 700. HRMS (TOF MS EI^+) m/z calcd for $[\text{C}_{20}\text{H}_{22}]$ 262.1722, found 262.1711.



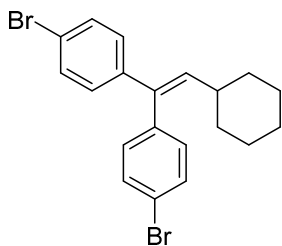
4,4'-(2-cyclohexylethene-1,1-diyl)bis(methylbenzene) (3ba): White solid. mp 45–47 °C. Yield: 26.0 mg (45%). ^1H NMR (400 MHz, CDCl_3) δ 7.20–7.00 (m, 8H), 5.82 (d, J = 10.0 Hz, 1H), 2.38 (s, 3H), 2.31 (s, 3H), 2.20–2.06 (m, 1H), 1.71–1.58 (m, 5H), 1.21–1.12 (m, 5H). ^{13}C NMR (101 MHz, CDCl_3) δ 140.5, 139.3, 137.8, 136.32, 136.2, 135.1, 129.7, 128.8, 128.7, 127.1, 38.2, 33.4, 26.0, 25.6, 21.3, 21.0. IR (cm^{-1}): 2954, 2924, 2854, 1459, 816. HRMS (TOF MS EI^+) m/z calcd for $[\text{C}_{22}\text{H}_{26}]$ 290.2035, found 290.2044.



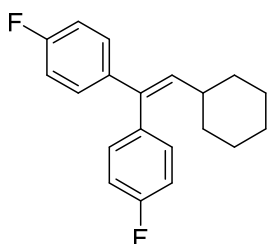
4,4'-(2-cyclohexylethene-1,1-diyl)bis(methoxybenzene) (3ca): Yellow oil. Yield: 32.0 mg (50%). ^1H NMR (400 MHz, CDCl_3) δ 7.09–6.97 (m, 4H), 6.85–6.78 (m, 2H), 6.73–6.66 (m, 2H), 5.68 (d, J = 10.0 Hz, 1H), 3.76 (s, 3H), 3.70 (s, 3H), 2.05 (d, J = 9.1 Hz, 1H), 1.67–1.55 (m, 5H), 1.13–1.03 (m, 5H). ^{13}C NMR (101 MHz, CDCl_3) δ 158.6, 158.4, 138.5, 136.1, 134.3, 133.2, 130.8, 128.3, 113.5, 113.4, 55.3, 55.2, 38.3, 33.5, 26.1, 25.7. IR (cm^{-1}): 2924, 2850, 1607, 1513, 1506, 1463, 1448, 1285, 1244, 1173, 1037, 830. HRMS (TOF MS EI^+) m/z calcd for $[\text{C}_{22}\text{H}_{26}\text{O}_2]$ 322.1933, found 322.1927.



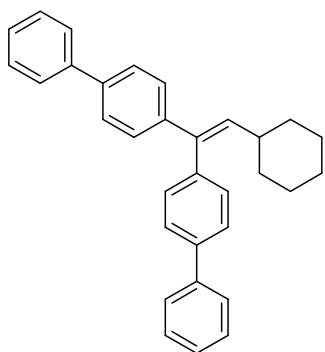
4,4'-(2-cyclohexylethene-1,1-diyl)bis(chlorobenzene) (3da): White solid. mp 84–87 °C. Yield: 38.7 mg (59%). ^1H NMR (400 MHz, CDCl_3) δ 7.38–7.31 (m, 2H), 7.23–7.16 (m, 2H), 7.12–7.05 (m, 4H), 5.88 (d, J = 10.1 Hz, 1H), 2.16–1.99 (m, 1H), 1.78–1.59 (m, 5H), 1.23–1.05 (m, 5H). ^{13}C NMR (101 MHz, CDCl_3) δ 140.9, 138.5, 137.5, 136.9, 133.0, 132.8, 131.1, 128.6, 128.4, 128.2, 38.5, 33.2, 25.9, 25.5. IR (cm^{-1}): 2923, 2849, 1492, 1446, 1401, 1092, 1014, 826. HRMS (TOF MS EI^+) m/z calcd for $[\text{C}_{20}\text{H}_{20}\text{Cl}_2]$ 330.0942, found 330.0936.



4,4'-(2-cyclohexylethene-1,1-diyl)bis(bromobenzene) (3ea): White solid. mp 99–102 °C. Yield: 43.7 mg (52%). ¹H NMR (400 MHz, CDCl₃) δ 7.54–7.45 (m, 2H), 7.41–7.30 (m, 2H), 7.10–6.96 (m, 4H), 5.89 (d, *J* = 10.1 Hz, 1H), 2.15–1.97 (m, 1H), 1.78–1.58 (m, 5H), 1.23–1.06 (m, 5H). ¹³C NMR (101 MHz, CDCl₃) δ 141.3, 138.9, 137.6, 137.0, 131.5, 131.4, 131.2, 128.8, 121.1, 120.9, 38.5, 33.2, 25.9, 25.5. IR (cm⁻¹): 2924, 2845, 1487, 1008, 831, 821, 490. HRMS (TOF MS EI⁺) *m/z* calcd for [C₂₀H₂₀Br₂] 417.9932, found 417.9938.

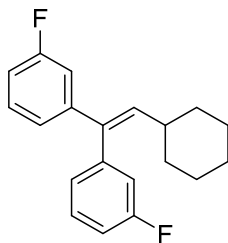


4,4'-(2-cyclohexylethene-1,1-diyl)bis(fluorobenzene) (3fa): Colorless oil. Yield: 32.0 mg (54%). ¹H NMR (400 MHz, CDCl₃) δ 7.19–7.00 (m, 6H), 6.98–6.88 (m, 2H), 5.83 (d, *J* = 10.0 Hz, 1H), 2.16–1.96 (m, 1H), 1.71–1.54 (m, 5H), 1.20–1.07 (m, 5H). ¹³C NMR (101 MHz, CDCl₃) δ 163.2, 163.1, 160.8, 160.7, 138.9, 138.9, 137.7, 136.2, 136.2, 136.2, 131.3, 131.2, 128.7, 128.6, 115.3, 115.1, 115.0, 114.8, 38.4, 33.3, 25.9, 25.6. IR (cm⁻¹): 2929, 1506, 1221, 1158, 842, 833, 569. HRMS (TOF MS EI⁺) *m/z* calcd for [C₂₀H₂₀F₂] 298.1533, found 298.1527.

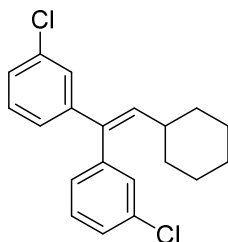


4,4''-(2-cyclohexylethene-1,1-diyl)di-1,1'-biphenyl (3ga): White solid. mp 58–61 °C. Yield: 39.6 mg (48%). ¹H NMR (400 MHz, CDCl₃) δ 7.71–7.54 (m, 6H), 7.53–7.39 (m, 6H), 7.36–7.19 (m, 6H), 5.99 (d, *J* = 10.0 Hz, 1H), 2.31–2.14 (m, 1H), 1.85–1.59 (m, 5H), 1.25–1.09 (m, 5H). ¹³C NMR (101 MHz, CDCl₃) δ 142.0, 140.9, 140.9, 139.6, 139.6, 139.5, 138.9, 136.4, 130.3, 128.8, 128.8, 127.7,

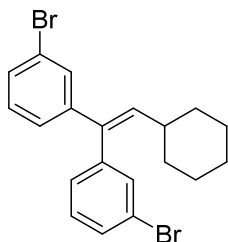
127.3, 127.2, 127.0, 127.0, 126.9, 126.8, 38.4, 33.4, 26.0, 25.6. IR (cm⁻¹): 2928, 2844, 1485, 838, 831, 766, 731. HRMS (TOF MS EI⁺) m/z calcd for [C₃₂H₃₀] 414.2348, found 414.2357.



3,3'-(2-cyclohexylethene-1,1-diyl)bis(fluorobenzene) (3ha): Yellow oil. Yield: 31.9 mg (54%). ¹H NMR (400 MHz, CDCl₃) δ 7.40–7.30 (m, 1H), 7.24–7.15 (m, 1H), 7.08–6.82 (m, 6H), 5.94 (d, *J* = 10.1 Hz, 1H), 2.16–2.00 (m, 1H), 1.77–1.58 (m, 5H), 1.23–1.07 (m, 5H). ¹³C NMR (101 MHz, CDCl₃) δ 164.0, 164.0, 161.6, 161.5, 144.6, 144.5, 142.2, 142.1, 137.6, 137.6, 137.6, 137.4, 129.8, 129.8, 129.5, 129.4, 125.5, 125.5, 122.7, 122.7, 116.7, 116.5, 114.2, 114.0, 114.0, 113.8, 113.8, 113.6, 38.4, 33.1, 25.9, 25.5. IR (cm⁻¹): 2926, 2851, 1609, 1582, 1484, 1446, 783. HRMS (TOF MS EI⁺) m/z calcd for [C₂₀H₂₀F₂] 298.1533, found 298.1530.

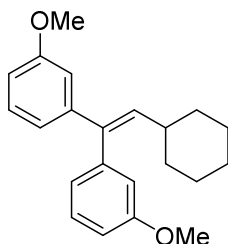


3,3'-(2-cyclohexylethene-1,1-diyl)bis(chlorobenzene) (3ia): Colorless oil. Yield: 35.2 mg (53%). ¹H NMR (400 MHz, CDCl₃) δ 7.36–7.27 (m, 2H), 7.23–7.10 (m, 4H), 7.09–6.98 (m, 2H), 5.92 (d, *J* = 10.1 Hz, 1H), 2.06 (m, 1H), 1.78–1.59 (m, 5H), 1.23–1.09 (m, 5H). ¹³C NMR (101 MHz, CDCl₃) δ 144.1, 141.6, 137.9, 137.4, 134.2, 134.2, 129.6, 129.4, 127.9, 127.4, 127.1, 127.0, 125.4, 38.4, 33.1, 25.9, 25.5. IR (cm⁻¹): 2922, 2848, 1591, 1561, 1470, 890, 789, 696. HRMS (TOF MS EI⁺) m/z calcd for [C₂₀H₂₀Cl₂] 330.0942, found 330.0953.



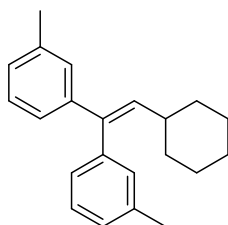
3,3'-(2-cyclohexylethene-1,1-diyl)bis(bromobenzene) (3ja): Yellow oil. Yield: 44.8 mg (54%). ¹H NMR (400 MHz, CDCl₃) δ 7.51–7.41 (m, 1H), 7.37–7.31 (m, 2H), 7.29 (t, *J* = 1.8 Hz, 1H), 7.26–7.22 (m, 1H), 7.15–6.99 (m, 3H), 5.90 (d, *J* = 10.1 Hz, 1H), 2.14–2.00 (m, 1H), 1.77–1.58 (m, 5H), 1.18 (m, 5H). ¹³C NMR (101 MHz, CDCl₃) δ 144.4, 141.9, 138.0, 137.2, 132.5, 130.3, 130.0, 129.9, 129.7,

128.4, 125.9, 122.5, 38.4, 33.1, 25.9, 25.5. IR (cm⁻¹): 2920, 2846, 1556, 890, 799, 788, 694. HRMS (TOF MS EI⁺) m/z calcd for [C₂₀H₂₀Br₂] 417.9932, found 417.9917.



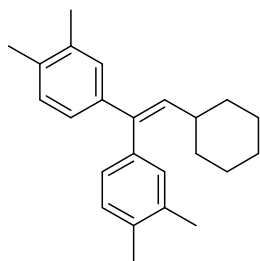
3,3'-(2-cyclohexylethene-1,1-diyl)bis(methoxybenzene) (3ka): Yellow oil. Yield: 29.5 mg (46%).

¹H NMR (400 MHz, CDCl₃) δ 7.22–7.16 (m, 1H), 7.09 (t, *J* = 7.8 Hz, 1H), 6.80–6.62 (m, 6H), 5.82 (d, *J* = 10.1 Hz, 1H), 3.72 (s, 3H), 3.68 (s, 3H), 2.13–1.99 (m, 1H), 1.67–1.54 (m, 5H), 1.09 (m, 5H). ¹³C NMR (101 MHz, CDCl₃) δ 159.4, 159.4, 144.2, 141.8, 139.3, 136.1, 129.1, 128.9, 122.3, 119.8, 115.2, 113.2, 112.4, 111.9, 55.2, 55.2, 38.3, 33.3, 26.0, 25.6. IR (cm⁻¹): 2924, 2849, 1596, 1485, 1285, 1231, 1051. HRMS (TOF MS EI⁺) m/z calcd for [C₂₂H₂₆O₂] 322.1933, found 322.1941.



3,3'-(2-cyclohexylethene-1,1-diyl)bis(methylbenzene) (3la): Light yellow oil. Yield: 34.2 mg

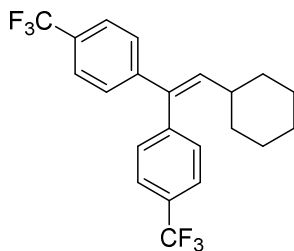
(59%). ¹H NMR (400 MHz, CDCl₃) δ 7.26–7.20 (m, 1H), 7.17–7.07 (m, 2H), 7.06–6.92 (m, 5H), 5.85 (d, *J* = 10.0 Hz, 1H), 2.34 (s, 3H), 2.29 (s, 3H), 2.21–2.03 (m, 1H), 1.76–1.56 (m, 5H), 1.23–1.06 (m, 5H). ¹³C NMR (101 MHz, CDCl₃) δ 143.1, 140.6, 139.8, 137.6, 137.5, 135.8, 130.4, 127.9, 127.9, 127.8, 127.5, 126.9, 124.5, 38.3, 33.4, 26.1, 25.6, 21.5, 21.5. IR (cm⁻¹): 2923, 2844, 1600, 1446, 890, 789, 720, 701. HRMS (TOF MS EI⁺) m/z calcd for [C₂₂H₂₆] 290.2035, found 290.2026.



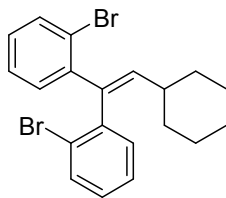
4,4'-(2-cyclohexylethene-1,1-diyl)bis(1,2-dimethylbenzene) (3ma): White solid. mp 116–118 °C.

Yield: 30.4 mg (48%). ¹H NMR (400 MHz, CDCl₃) δ 7.15–7.07 (m, 1H), 7.05–6.86 (m, 5H), 5.78 (d, *J* = 10.0 Hz, 1H), 2.29 (s, 3H), 2.24 (s, 3H), 2.22 (s, 3H), 2.20 (s, 3H), 2.16–2.10 (m, 1H), 1.78–1.56 (m, 5H), 1.23–1.07 (m, 5H). ¹³C NMR (101 MHz, CDCl₃) δ 141.2, 139.5, 138.3, 136.1, 136.0, 135.0, 135.0, 134.8, 130.9, 129.3, 129.2, 128.4, 127.2, 124.9, 38.2, 33.5, 26.1, 25.6, 19.9, 19.9, 19.6, 19.4. IR

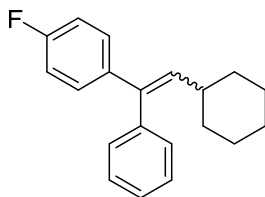
(cm^{-1}): 2933, 2843, 1503, 1444, 889, 827, 814. HRMS (TOF MS EI^+) m/z calcd for $[\text{C}_{24}\text{H}_{30}]$ 318.2348, found 318.2342.



4,4'-(2-cyclohexylethene-1,1-diyl)bis(trifluoromethyl)benzene (3na): Colorless oil. Yield: 35.7 mg (45%). ^1H NMR (400 MHz, CDCl_3) δ 7.65 (d, $J = 8.0$ Hz, 2H), 7.50 (d, $J = 8.3$ Hz, 2H), 7.31–7.23 (m, 4H), 6.04 (d, $J = 10.2$ Hz, 1H), 2.14–2.01 (m, 1H), 1.73–1.63 (m, 5H), 1.22–1.13 (m, 5H). ^{13}C NMR (101 MHz, CDCl_3) δ 145.5, 143.5, 139.0, 137.5, 130.1, 127.3, 125.5, 125.5, 125.4, 125.4, 125.4, 125.2, 125.2, 125.1, 125.1, 122.8, 38.5, 33.0, 25.8, 25.4. IR (cm^{-1}): 2930, 2851, 1615, 1323, 1166, 1123, 1067, 841. HRMS (TOF MS EI^+) m/z calcd for $[\text{C}_{22}\text{H}_{20}\text{F}_6]$ 398.1469, found 398.1454.

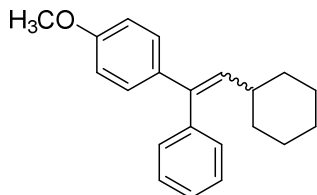


2,2'-(2-cyclohexylethene-1,1-diyl)bis(bromobenzene) (3oa): Yellow oil. Yield: 25.5 mg (31%). ^1H NMR (400 MHz, CDCl_3) δ 7.63–7.53 (m, 2H), 7.38–7.27 (m, 2H), 7.23–7.09 (m, 3H), 7.03 (t, $J = 7.4$ Hz, 1H), 5.87 (d, $J = 10.0$ Hz, 1H), 2.14–1.96 (m, 1H), 1.83–1.56 (m, 5H), 1.26–1.07 (m, 5H). ^{13}C NMR (101 MHz, CDCl_3) δ 142.9, 142.3, 141.0, 135.8, 133.6, 133.1, 132.3, 131.3, 128.5, 128.0, 127.0, 126.9, 123.8, 123.1, 39.0, 32.3, 26.0, 25.5. IR (cm^{-1}): 2924, 2849, 1466, 1447, 1430, 1026, 753, 746. HRMS (TOF MS EI^+) m/z calcd for $[\text{C}_{20}\text{H}_{20}\text{Br}_2]$ 417.9932, found 417.9948.

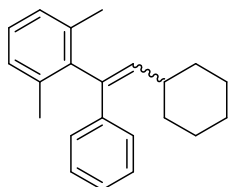


1-(2-cyclohexyl-1-phenylvinyl)-4-fluorobenzene (3pa): Yellow oil. Yield: 31.0 mg (55%). ^1H NMR (400 MHz, CDCl_3) δ 7.39–7.10 (m, 7H), 7.09–7.01 (m, 1H), 6.96–6.88 (m, 1H), 5.90 (d, $J = 10.1$ Hz, 0.47H), 5.83 (d, $J = 10.0$ Hz, 0.51H), 2.21–2.01 (m, 1H), 1.78–1.59 (m, 5H), 1.23–1.03 (m, 5H). ^{13}C NMR (101 MHz, CDCl_3) δ 163.2, 163.1, 160.7, 160.6, 142.7, 140.4, 139.1, 139.1, 138.7,

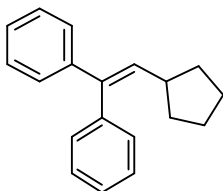
138.6, 136.4, 136.4, 136.3, 135.8, 135.8, 131.4, 131.3, 129.7, 128.8, 128.7, 128.2, 128.1, 127.1, 126.9, 126.9, 115.2, 115.0, 114.9, 114.7, 38.4, 38.3, 33.4, 33.3, 26.0, 25.6, 25.6. IR (cm⁻¹): 2924, 2851, 1508, 1447, 1232, 1223, 832. HRMS (TOF MS EI⁺) m/z calcd for [C₂₀H₂₁F] 280.1627, found 280.1617.



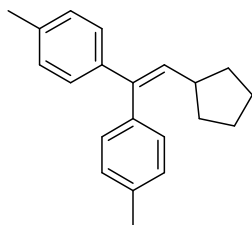
1-(2-cyclohexyl-1-phenylvinyl)-4-methoxybenzene (3qa): Yellow oil. Yield: 38.9 mg (67%). ¹H NMR (400 MHz, CDCl₃) δ 7.39–7.32 (m, 1H), 7.32–7.06 (m, 6H), 6.94–6.86 (m, 1H), 6.78 (d, *J* = 8.1 Hz, 1H), 5.85 (d, *J* = 10.0 Hz, 0.52H), 5.80 (d, *J* = 10.0 Hz, 0.48H), 3.83 (s, 1.58H), 3.77 (s, 1.48H), 2.24–2.02 (m, 1H), 1.76–1.57 (m, 5H), 1.24–1.06 (m, 5H). ¹³C NMR (101 MHz, CDCl₃) δ 158.6, 158.4, 143.4, 140.9, 139.2, 139.0, 135.9, 135.7, 134.4, 132.9, 130.9, 129.8, 128.3, 128.1, 128.0, 127.3, 126.7, 126.7, 113.5, 113.4, 55.3, 55.2, 38.3, 38.3, 33.5, 33.4, 26.0, 25.7. IR (cm⁻¹): 2925, 2852, 1510, 1446, 1246, 1038, 830, 670. HRMS (TOF MS EI⁺) m/z calcd for [C₂₁H₂₄O] 292.1827, found 292.1817.



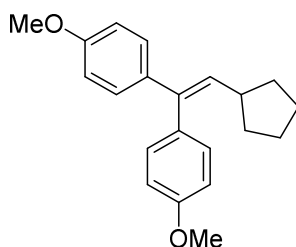
2-(2-cyclohexyl-1-phenylvinyl)-1,3-dimethylbenzene (3ra): Yellow oil. Yield: 26.0 mg (45%). ¹H NMR (400 MHz, CDCl₃) δ 7.32–6.98 (m, 8H), 6.11 (d, *J* = 9.8 Hz, 0.62H), 5.34 (d, *J* = 10.4 Hz, 0.37H), 2.19 (s, 2.17H), 2.11 (s, 3.66H), 1.83–1.53 (m, 6H), 1.25–1.04 (m, 5H). ¹³C NMR (101 MHz, CDCl₃) δ 143.8, 140.4, 139.8, 139.1, 138.3, 136.6, 136.4, 136.4, 136.3, 134.7, 128.9, 128.3, 127.9, 127.4, 127.3, 126.7, 126.7, 126.6, 126.5, 125.7, 38.8, 37.5, 33.3, 32.6, 26.1, 26.0, 25.6, 25.6, 20.6, 20.2. IR (cm⁻¹): 2924, 2851, 1598, 1493, 1463, 1446, 1377, 1030, 969, 897, 762, 695. HRMS (TOF MS EI⁺) m/z calcd for [C₂₂H₂₆] 290.2035, found 290.2027.



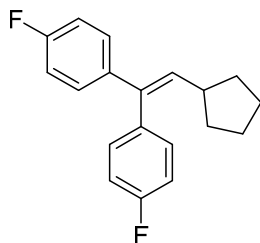
(2-cyclopentylethene-1,1-diyl)dibenzen (3ab): Colorless oil. Yield: 32.1 mg (65%). ^1H NMR (400 MHz, CDCl_3) δ 7.40–7.27 (m, 3H), 7.25–7.15 (m, 7H), 5.97 (d, J = 10.0 Hz, 1H), 2.61–2.42 (m, 1H), 1.89–1.58 (m, 4H), 1.60–1.32 (m, 4H). ^{13}C NMR (101 MHz, CDCl_3) δ 142.9, 140.6, 140.0, 135.5, 130.0, 128.1, 128.1, 127.3, 126.8, 126.7, 40.5, 34.3, 25.6. IR (cm^{-1}): 2952, 2866, 1445, 762, 700. HRMS (TOF MS EI^+) m/z calcd for $[\text{C}_{19}\text{H}_{20}]$ 248.1565, found 248.1562.



4,4'-(2-cyclopentylethene-1,1-diyl)bis(methylbenzene) (3bb): Yellow oil. Yield: 24.5 mg (44%). ^1H NMR (400 MHz, CDCl_3) δ 7.20–7.02 (m, 8H), 5.90 (dd, J = 10.0, 1.8 Hz, 1H), 2.60–2.45 (m, 1H), 2.38 (s, 3H), 2.31 (s, 3H), 1.85–1.60 (m, 4H), 1.53–1.30 (m, 4H). ^{13}C NMR (101 MHz, CDCl_3) δ 140.4, 139.7, 137.8, 136.3, 136.3, 134.5, 129.9, 128.7, 127.2, 40.4, 34.3, 25.6, 21.3, 21.1. IR (cm^{-1}): 2951, 2925, 2854, 1509, 817. HRMS (TOF MS EI^+) m/z calcd for $[\text{C}_{21}\text{H}_{24}]$ 276.1878, found 276.1871.

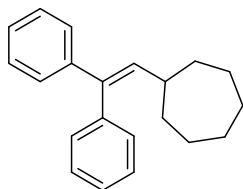


4,4'-(2-cyclopentylethene-1,1-diyl)bis(methoxybenzene) (3cb): Yellow oil. Yield: 31.5 mg (51%). ^1H NMR (400 MHz, CDCl_3) δ 7.10–7.00 (m, 4H), 6.85–6.79 (m, 2H), 6.75–6.68 (m, 2H), 5.75 (d, J = 9.9 Hz, 1H), 3.76 (s, 3H), 3.71 (s, 3H), 2.52–2.37 (m, 1H), 1.78–1.65 (m, 2H), 1.64–1.54 (m, 2H), 1.47–1.37 (m, 2H), 1.36–1.23 (m, 2H). ^{13}C NMR (101 MHz, CDCl_3) δ 157.5, 157.3, 137.9, 135.0, 132.6, 132.1, 130.0, 127.3, 112.4, 54.2, 54.2, 39.4, 33.3, 24.6. IR (cm^{-1}): 2926, 1511, 1245, 1037, 832. HRMS (TOF MS EI^+) m/z calcd for $[\text{C}_{21}\text{H}_{24}\text{O}_2]$ 308.1776, found 308.1770.

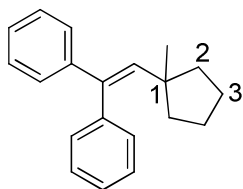


4,4'-(2-cyclopentylethene-1,1-diyl)bis(fluorobenzene) (3fb): Colorless oil. Yield: 26.2 mg (46%).

^1H NMR (400 MHz, CDCl_3) δ 7.18–7.09 (m, 4H), 7.08–7.02 (m, 2H), 6.97–6.90 (m, 2H), 5.89 (d, J = 10.0 Hz, 1H), 2.54–2.39 (m, 1H), 1.82–1.62 (m, 4H), 1.55–1.45 (m, 2H), 1.41–1.30 (m, 2H). ^{13}C NMR (101 MHz, CDCl_3) δ 163.2, 163.1, 160.7, 160.7, 138.9, 138.8, 138.0, 136.2, 136.2, 135.6, 131.5, 131.4, 128.7, 128.7, 115.2, 115.0, 115.0, 114.8, 40.4, 34.2, 25.6. IR (cm^{-1}): 2954, 2867, 1602, 1506, 1224, 1158, 838, 571, 521. HRMS (TOF MS EI^+) m/z calcd for $[\text{C}_{19}\text{H}_{18}\text{F}_2]$ 284.1377, found 284.1365.



(2,2-diphenylvinyl)cycloheptane (3ac): Yellow oil. Yield: 25.0 mg (45%). ^1H NMR (400 MHz, CDCl_3) δ 7.41–7.27 (m, 3H), 7.25–7.13 (m, 7H), 6.01 (d, J = 10.3 Hz, 1H), 2.39–2.25 (m, 1H), 1.79–1.68 (m, 2H), 1.68–1.58 (m, 2H), 1.54–1.35 (m, 8H). ^{13}C NMR (101 MHz, CDCl_3) δ 143.0, 140.6, 138.1, 136.6, 129.8, 128.1, 128.0, 127.2, 126.7, 126.7, 39.6, 35.0, 28.6, 26.2. IR (cm^{-1}): 2924, 2852, 1494, 1458, 1444, 761, 700. HRMS (TOF MS EI^+) m/z calcd for $[\text{C}_{21}\text{H}_{24}]$ 276.1878, found 276.1867.



(2-(1-methylcyclopentyl)ethene-1,1-diyl)dibenzene(3af1):

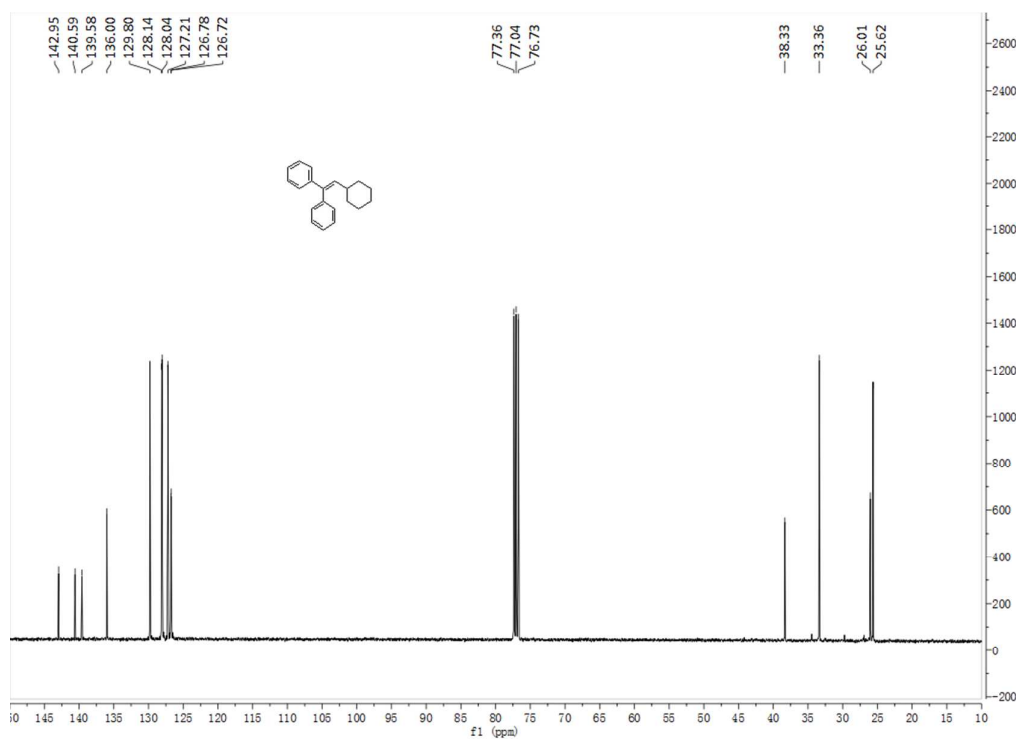
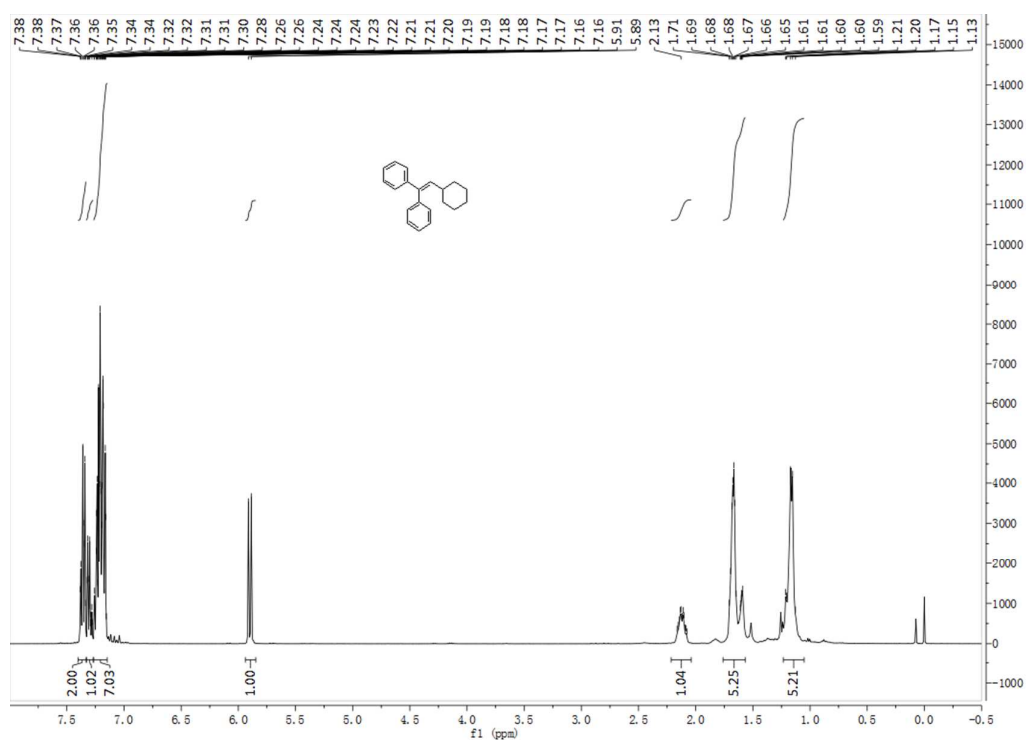
(2-(2-methylcyclopentyl)ethene-1,1-diyl)dibenzene(3af2):

(2-(3-methylcyclopentyl)ethene-1,1-diyl)dibenzene(3af3): Colorless oil. Yield: 35.3 mg (67%).

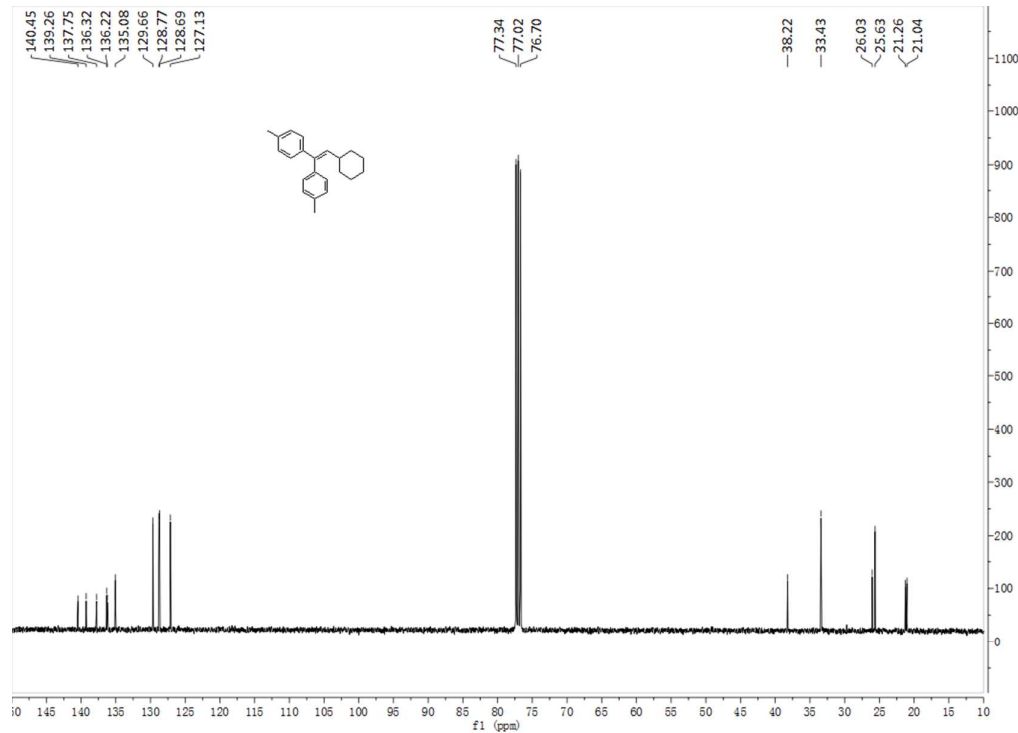
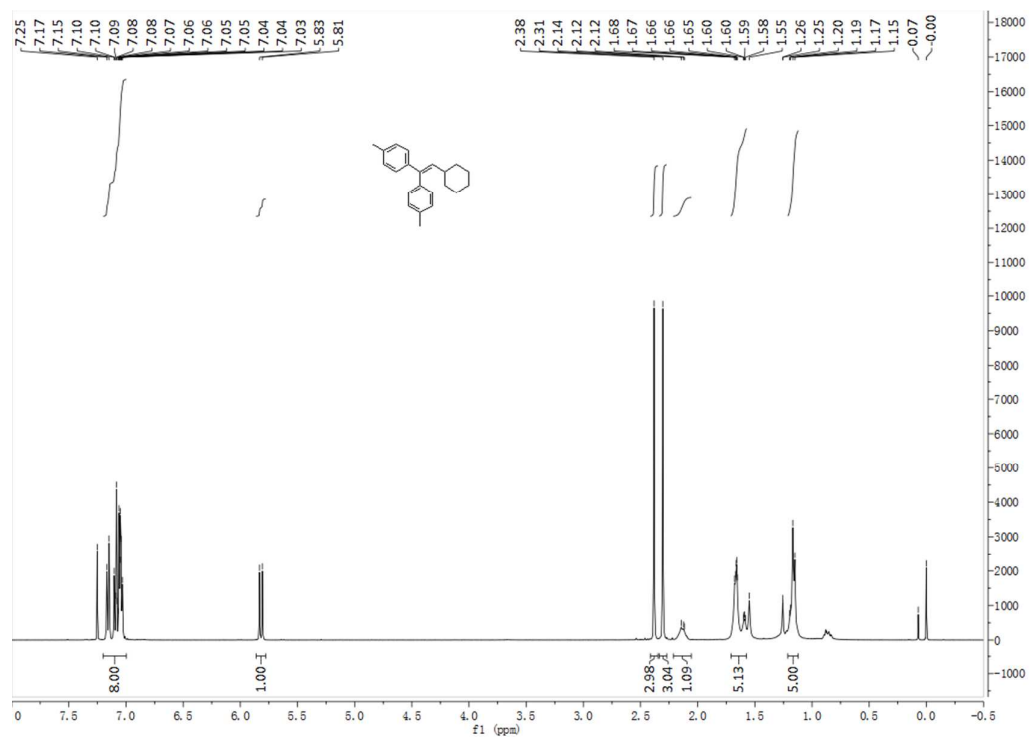
^1H NMR (400 MHz, CDCl_3) δ 7.40 – 7.12 (m, 10H), 6.22 (s, 0.56H), 5.98 (d, J = 10.1 Hz, 0.26H), 5.92 (d, J = 10.1 Hz, 0.14H), 1.97 – 1.69 (m, 1H), 1.72 – 1.45 (m, 4H), 1.34 – 1.18 (m, 2H), 1.16 – 0.79 (m, 4H). ^{13}C NMR (101 MHz, CDCl_3) δ 144.1, 143.0, 142.9, 142.7, 141.4, 141.1, 140.7, 140.5, 139.7, 139.5, 136.0, 135.8, 134.6, 130.3, 130.0, 130.0, 128.1, 128.0, 128.0, 127.8, 127.3, 127.2, 127.1, 127.0, 126.8, 126.7, 126.7, 126.6, 48.4, 45.1, 43.8, 42.5, 42.0, 40.8, 40.7, 39.0, 35.2, 34.9, 34.8, 34.4, 34.0, 33.9, 33.8, 33.3, 26.9, 23.6, 23.5, 21.2, 20.9, 18.5. IR (cm^{-1}): 2952, 2866, 1494, 1445, 762, 701. HRMS (TOF MS EI^+) m/z calcd for $[\text{C}_{20}\text{H}_{22}]$ 262.1722, found 262.1717.

4. ^1H and ^{13}C NMR spectra of **3**

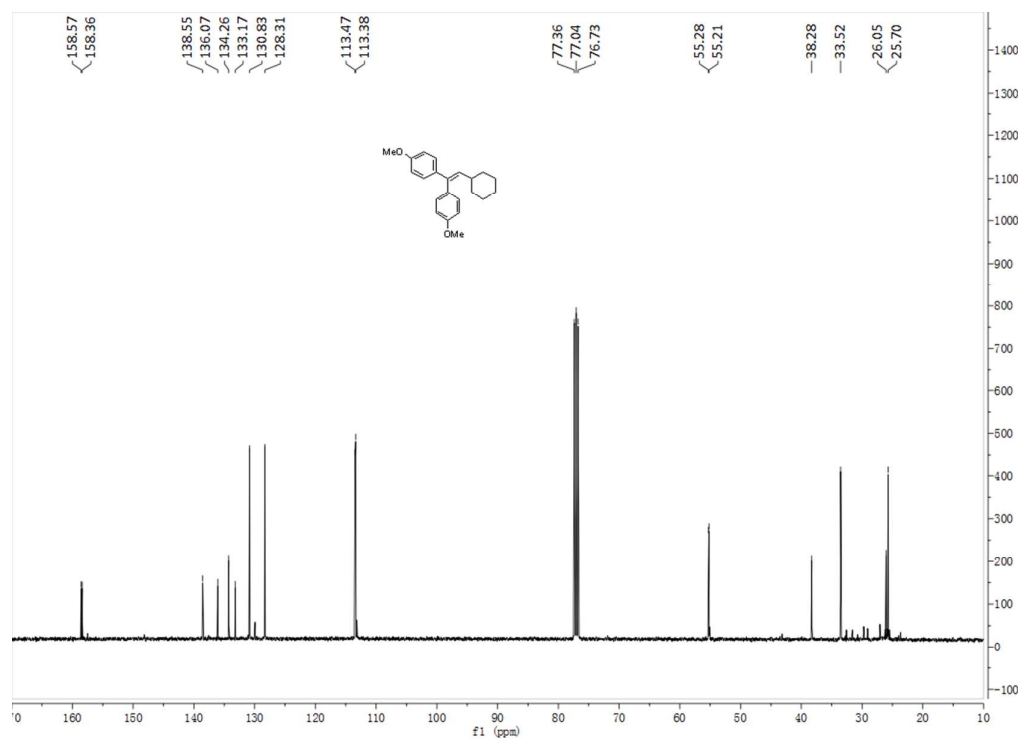
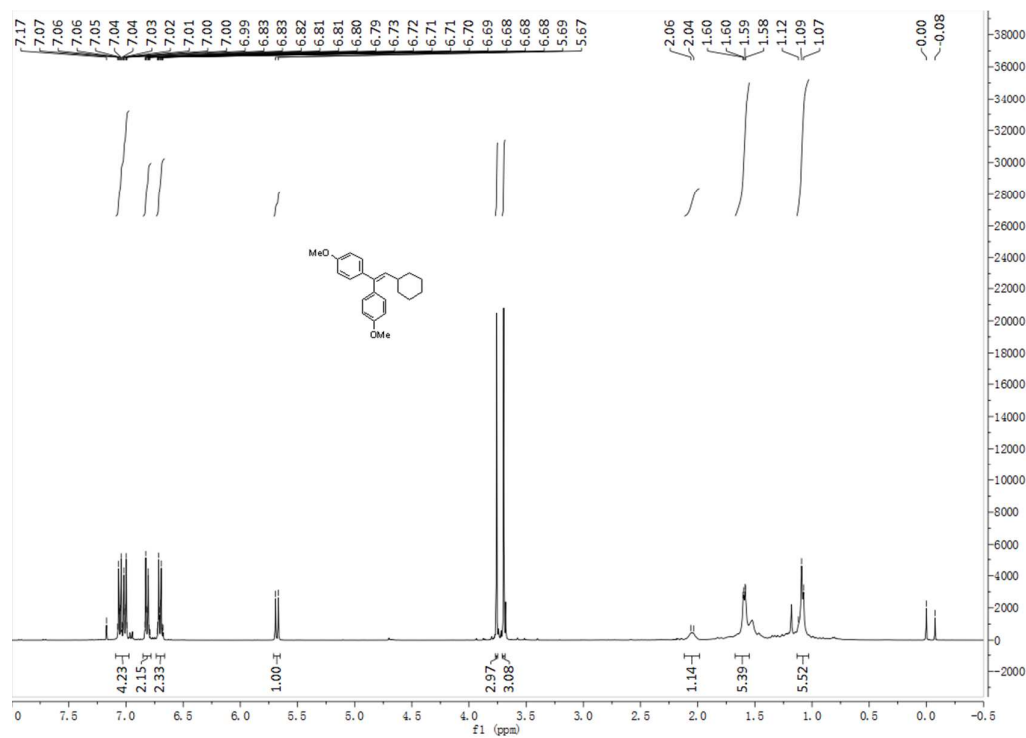
^1H and ^{13}C NMR spectra of **3aa**



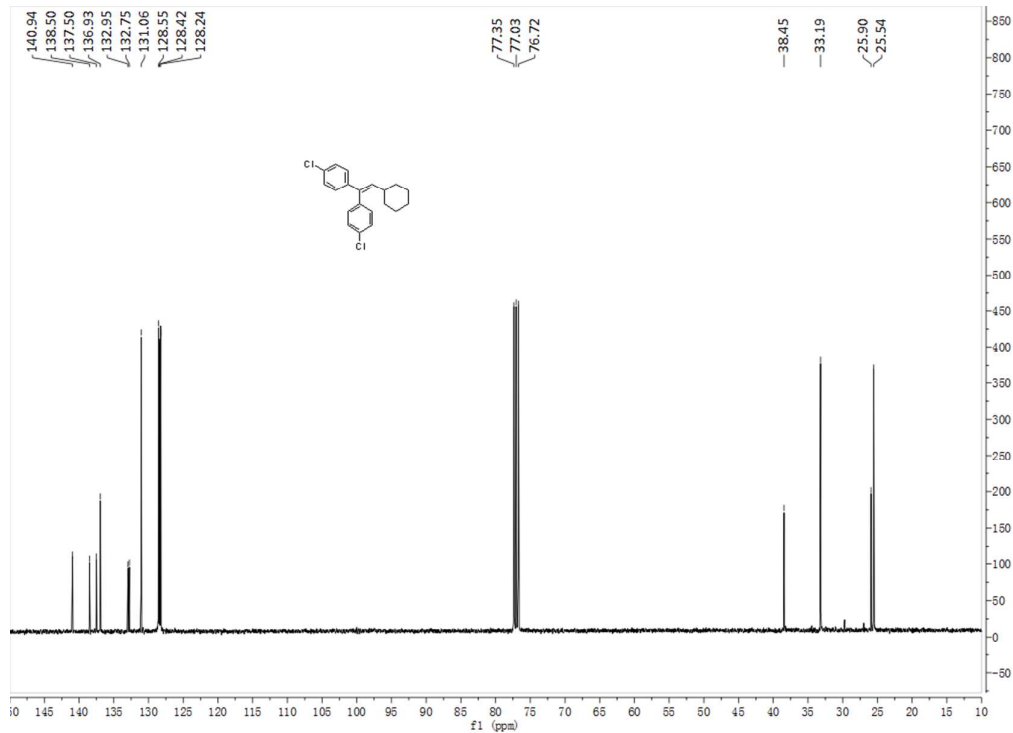
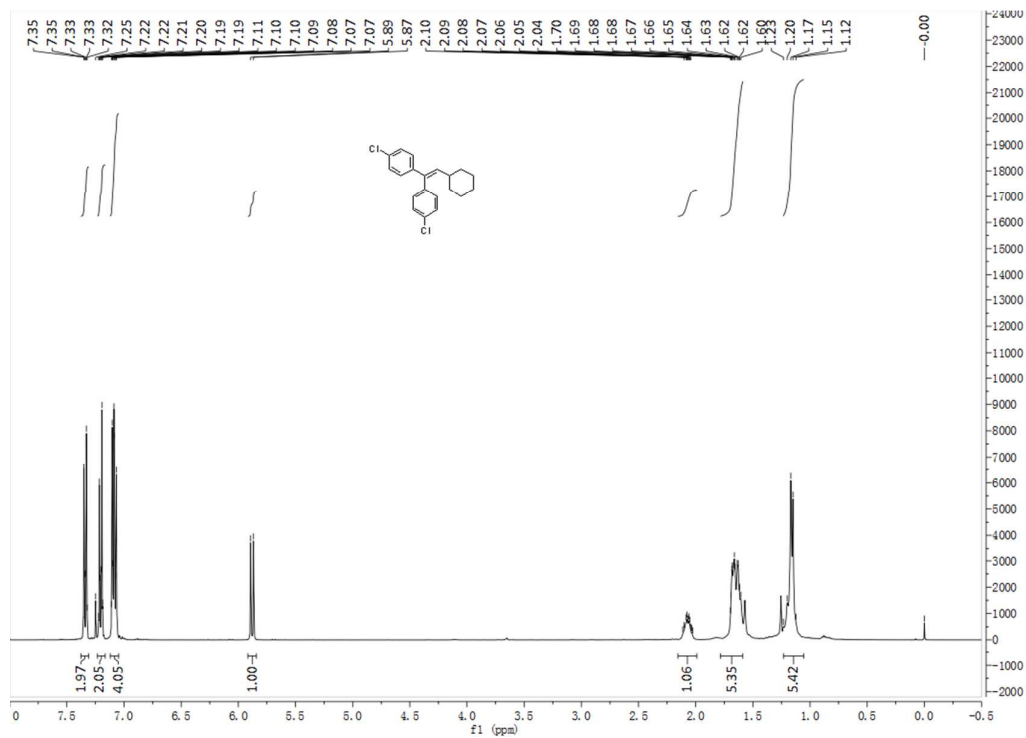
^1H and ^{13}C NMR spectra of **3ba**



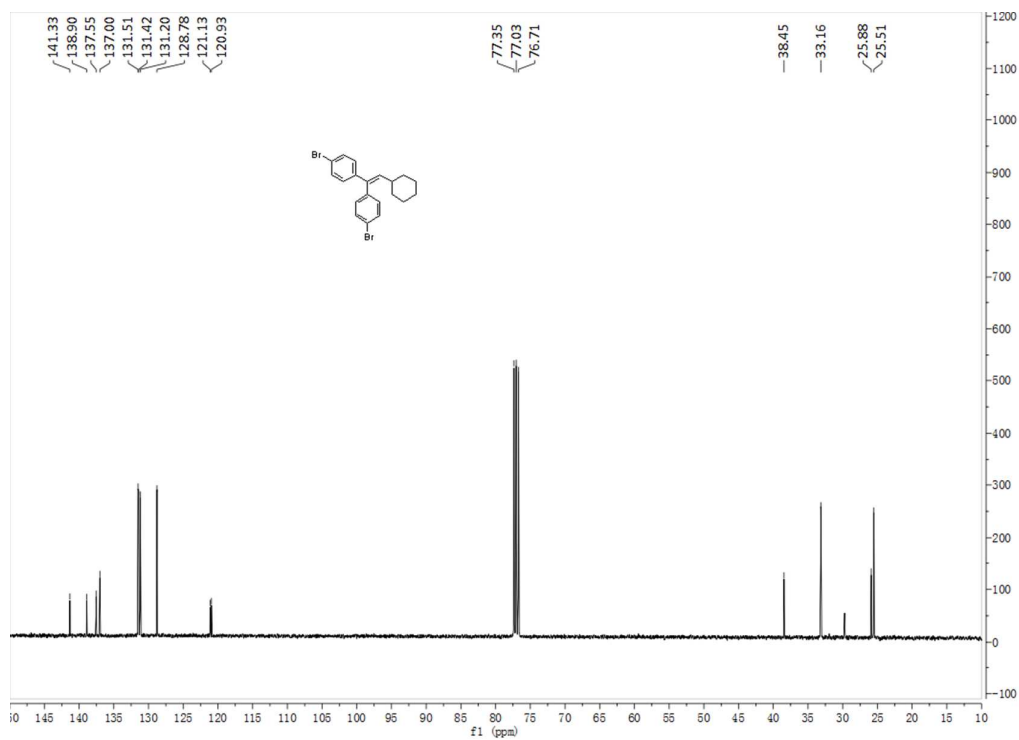
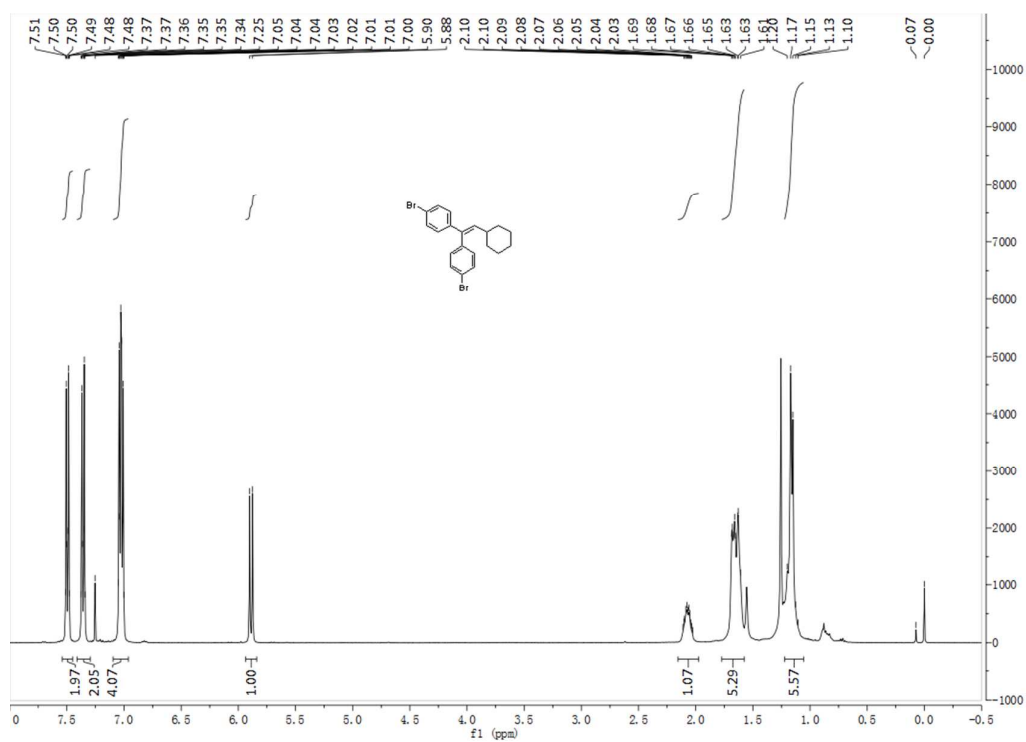
^1H and ^{13}C NMR spectra of **3ca**



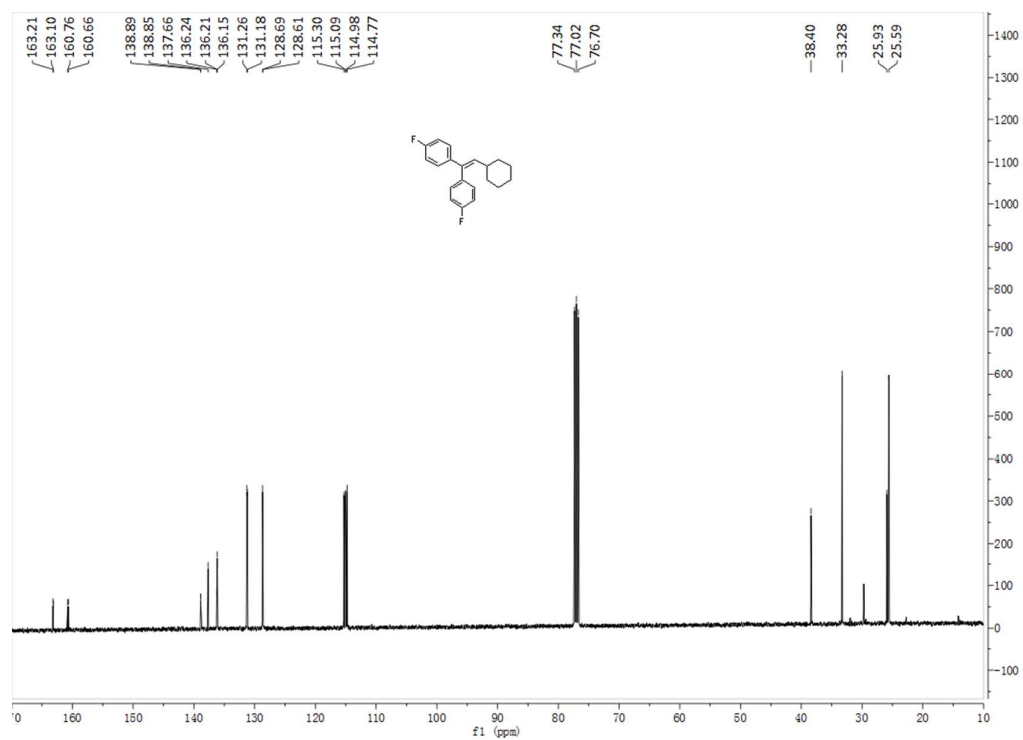
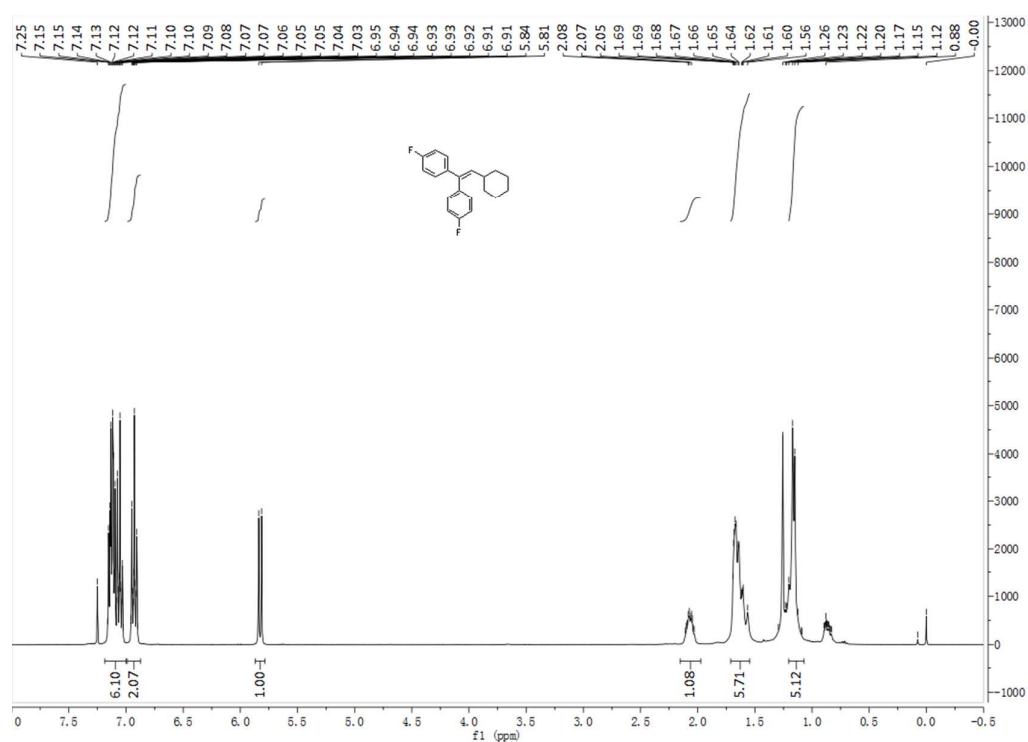
^1H and ^{13}C NMR spectra of **3da**



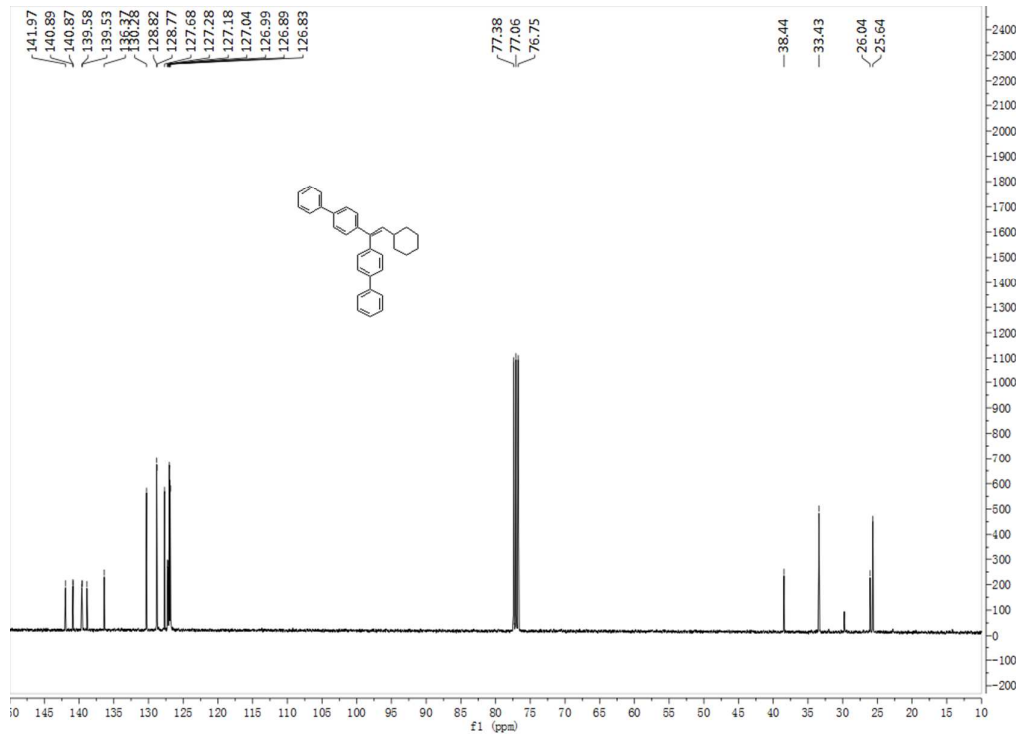
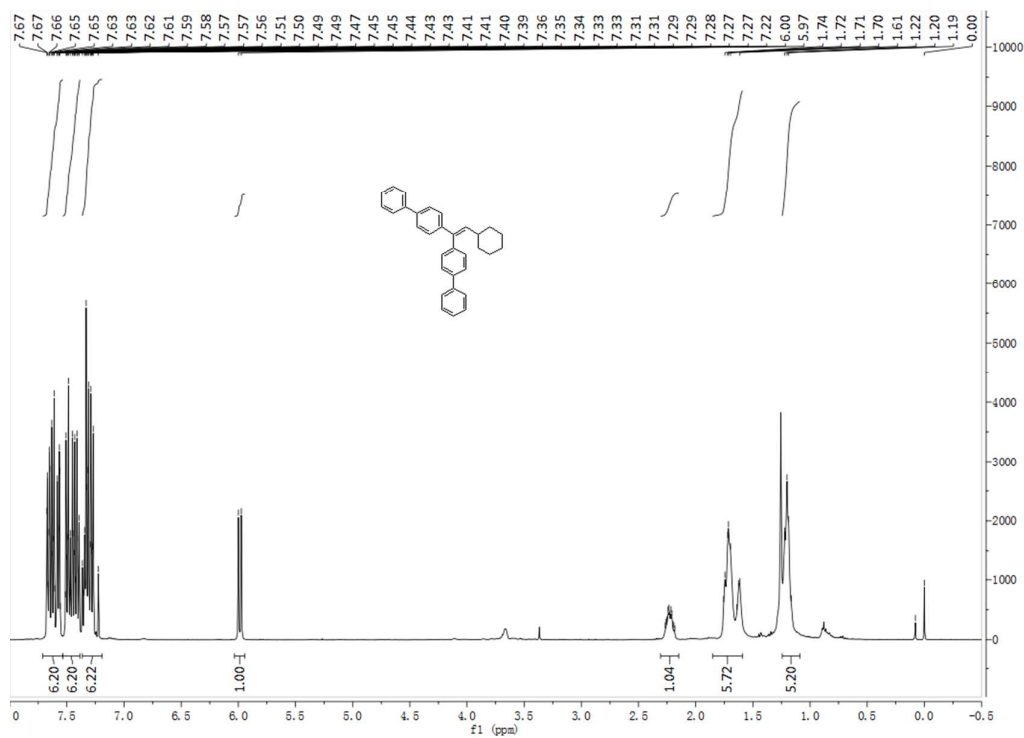
^1H and ^{13}C NMR spectra of **3ea**



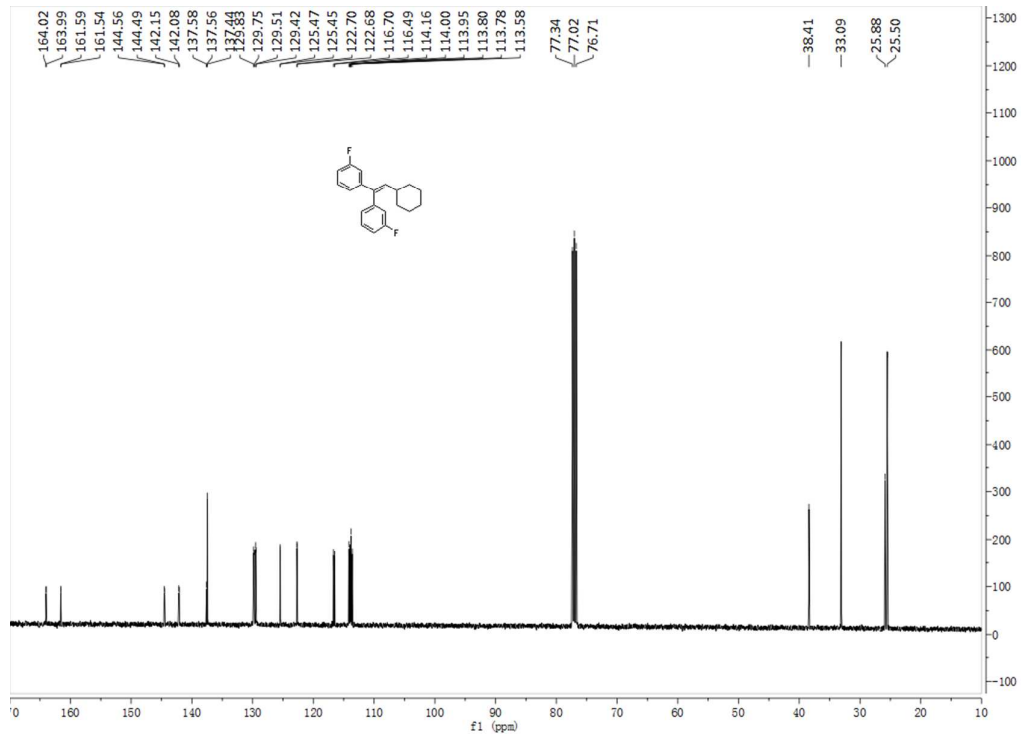
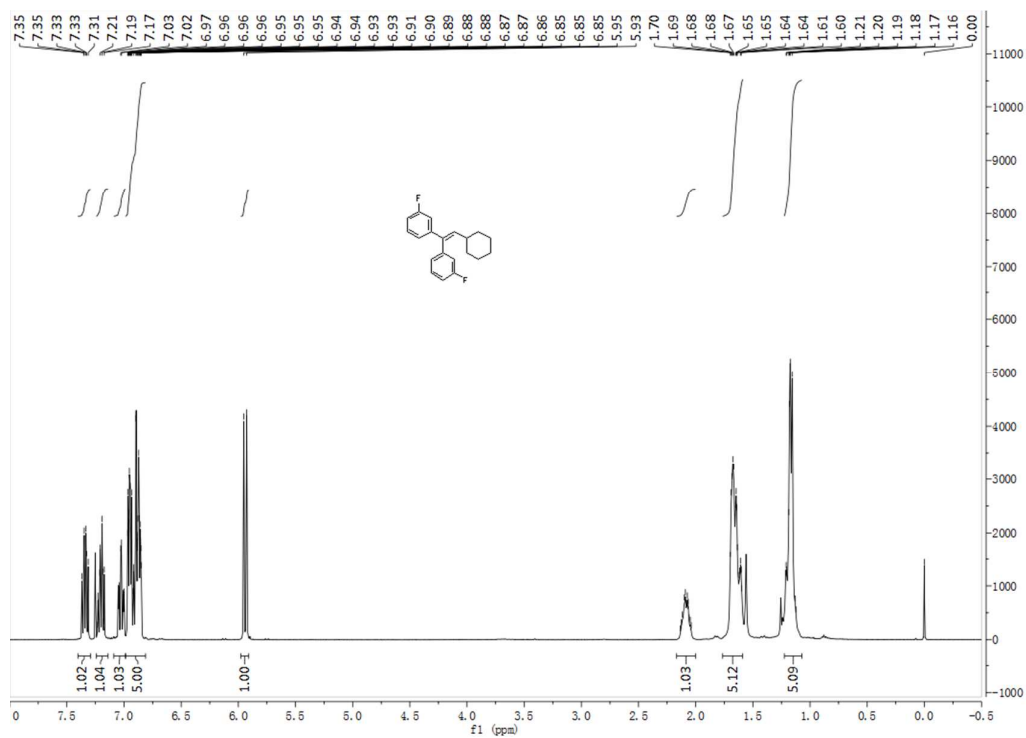
^1H and ^{13}C NMR spectra of **3fa**



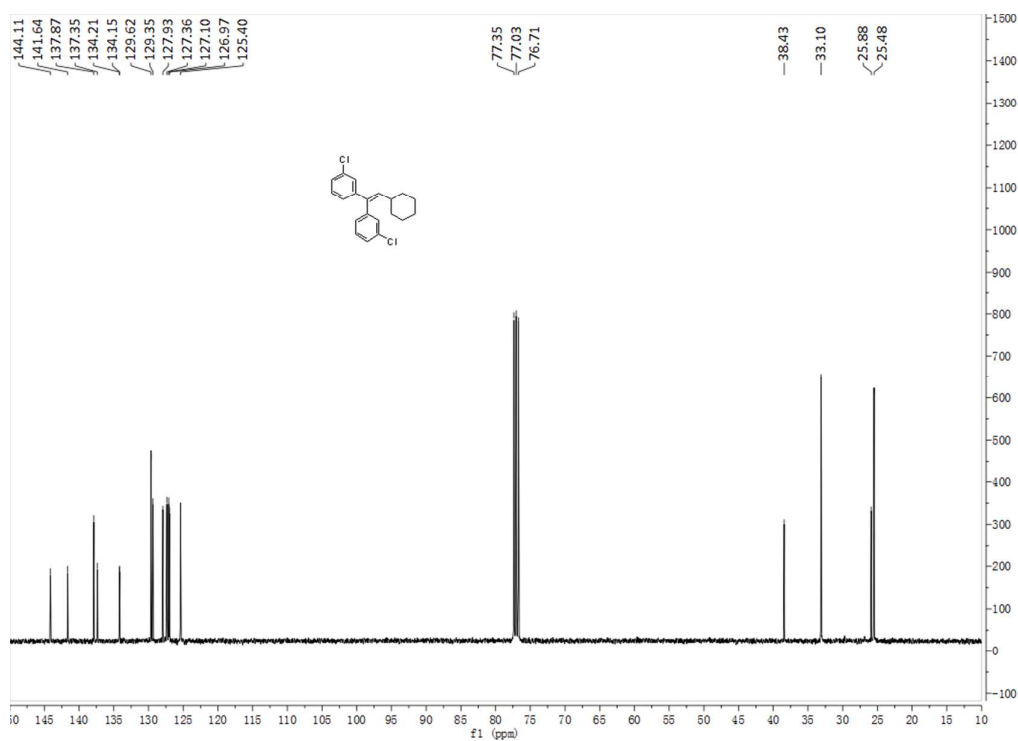
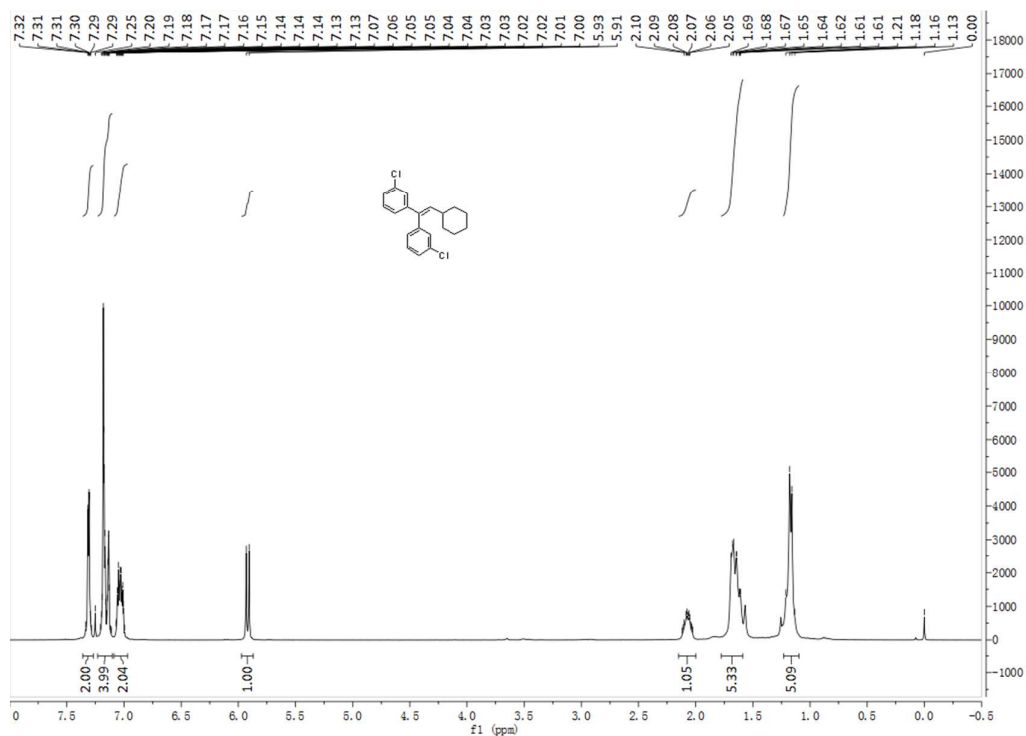
^1H and ^{13}C NMR spectra of **3ga**



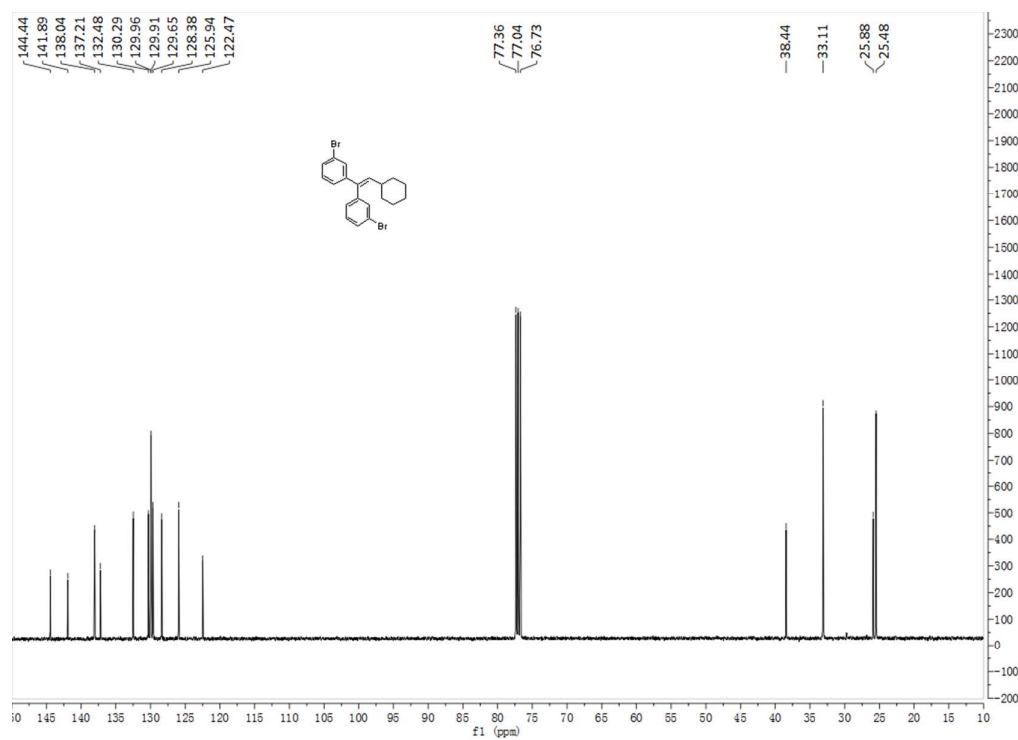
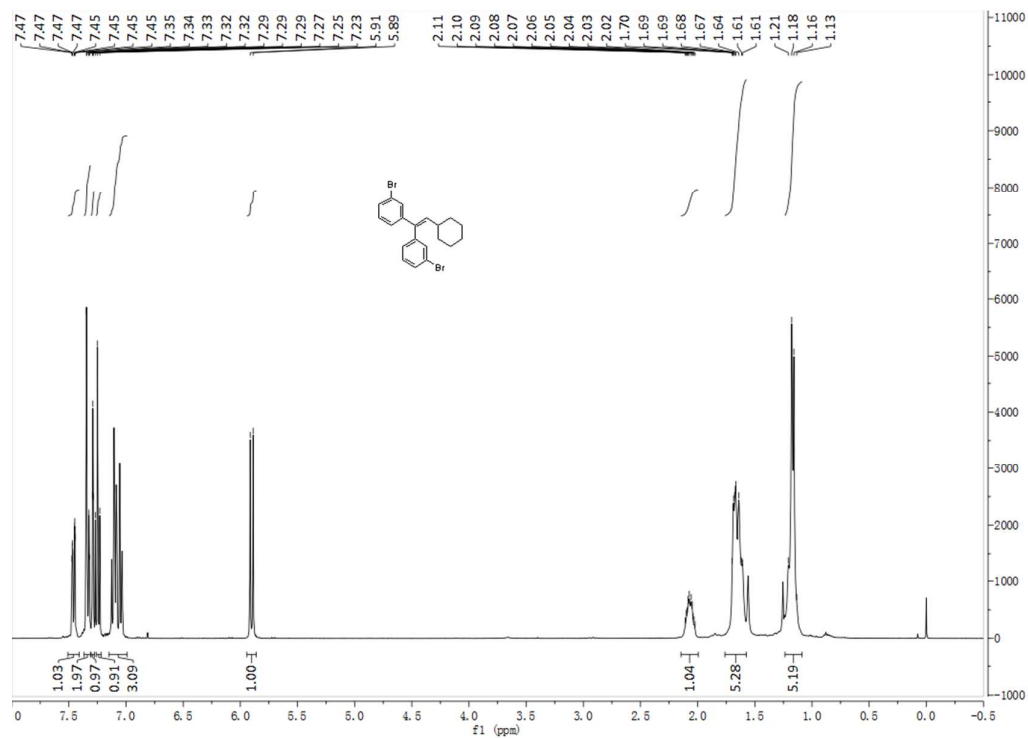
^1H and ^{13}C NMR spectra of **3ha**



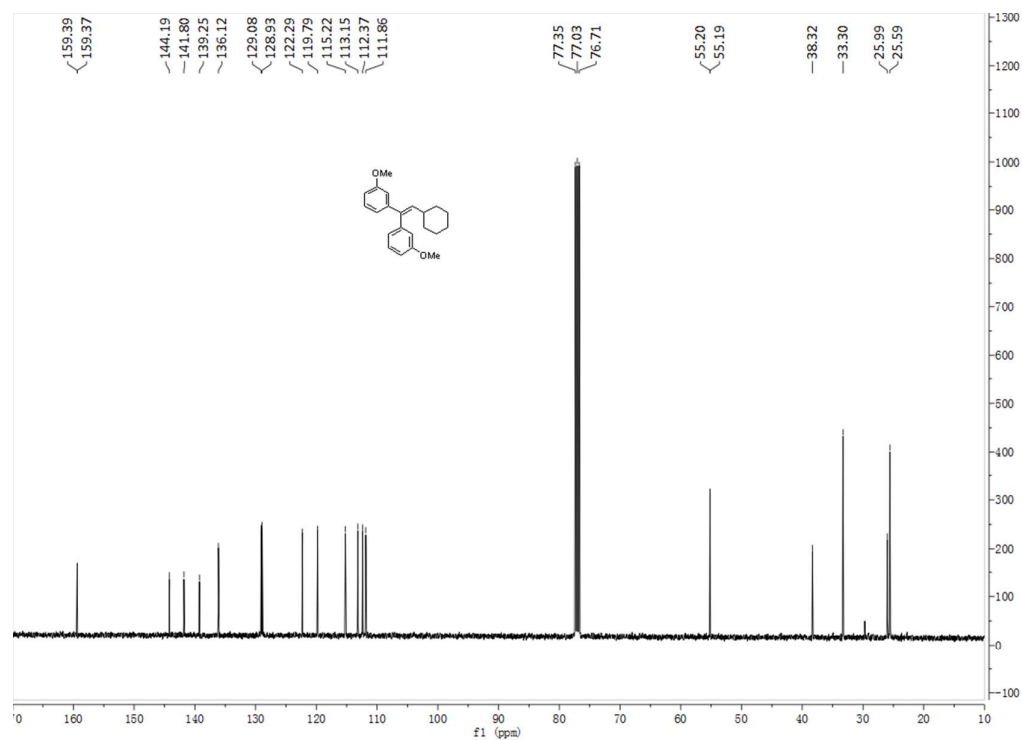
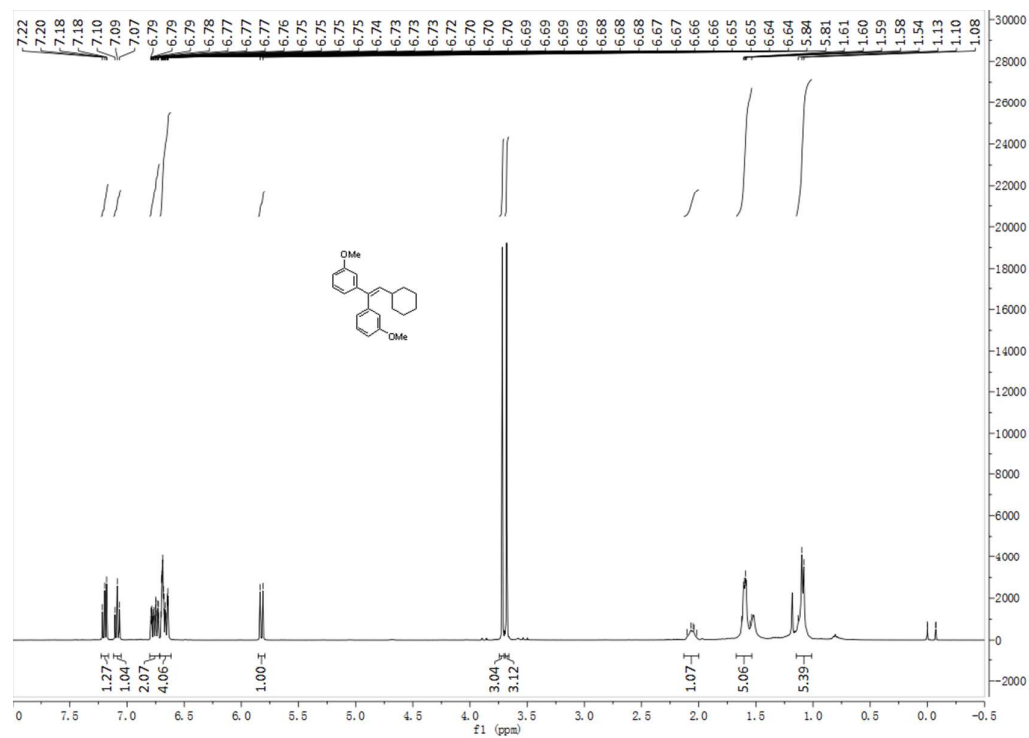
^1H and ^{13}C NMR spectra of **3ia**



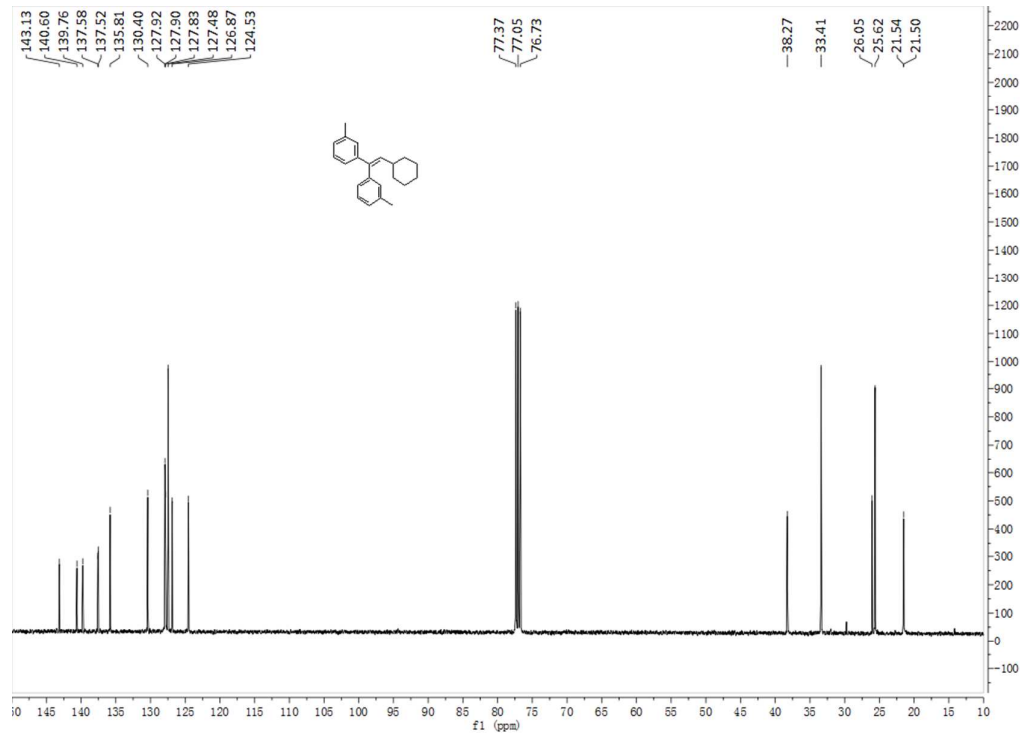
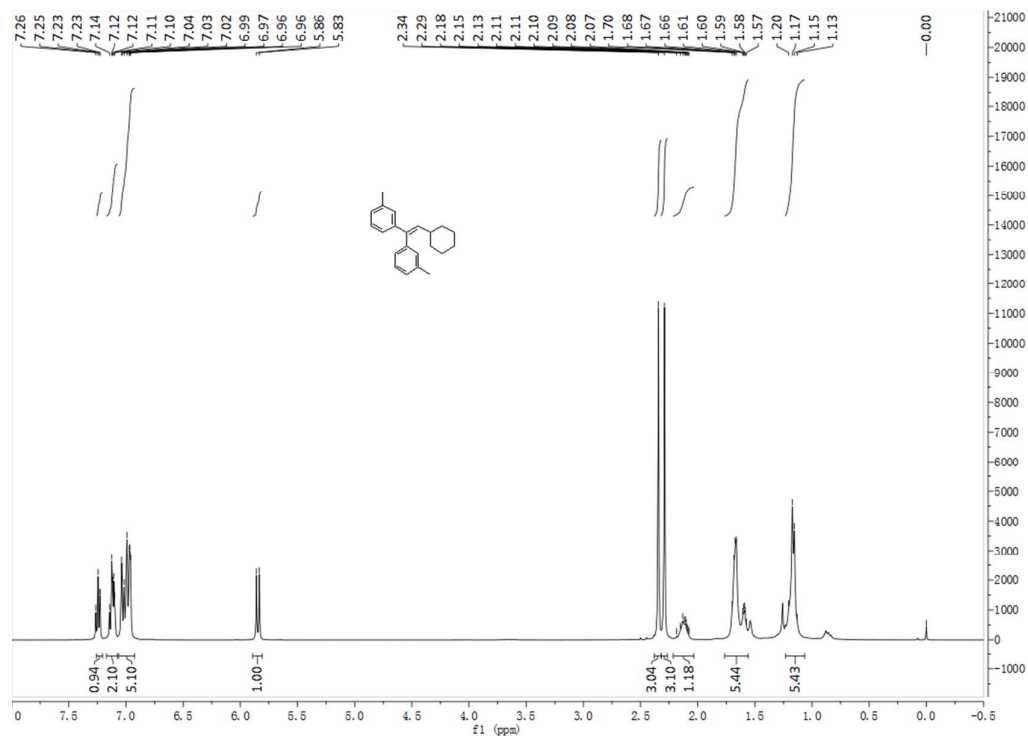
^1H and ^{13}C NMR spectra of **3ja**



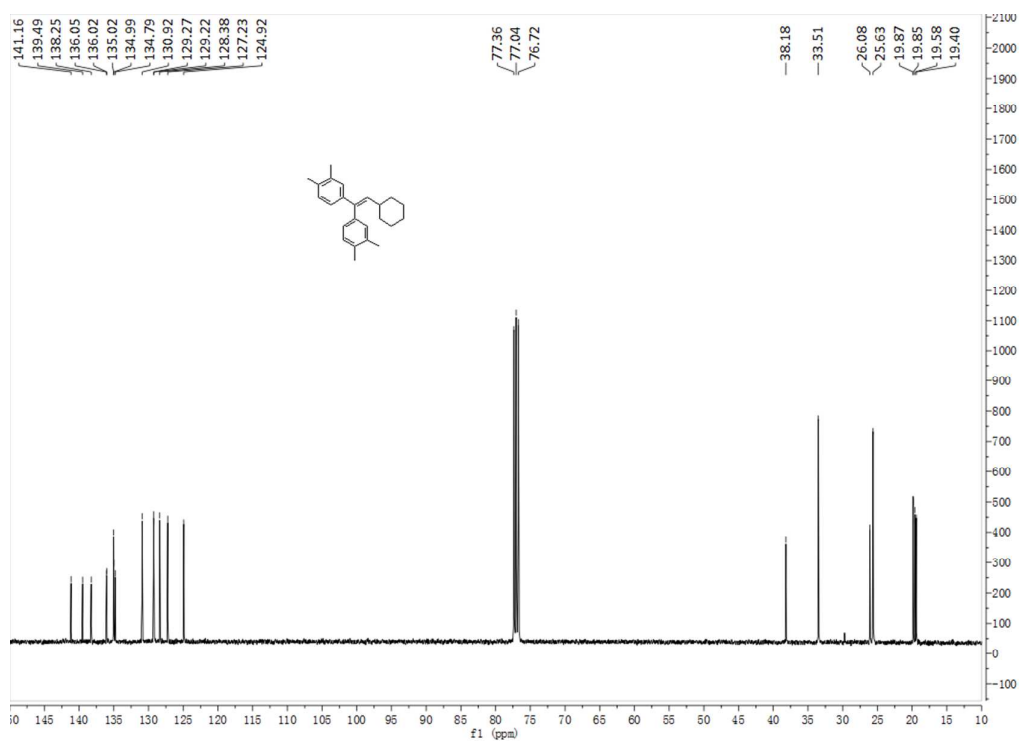
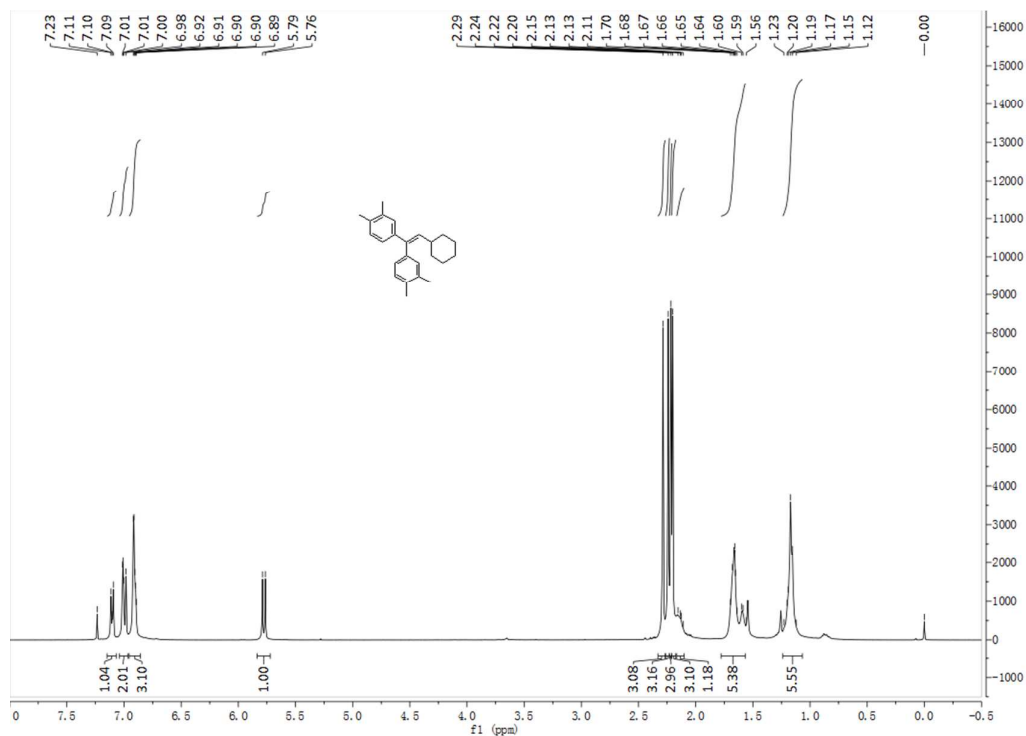
^1H and ^{13}C NMR spectra of **3ka**



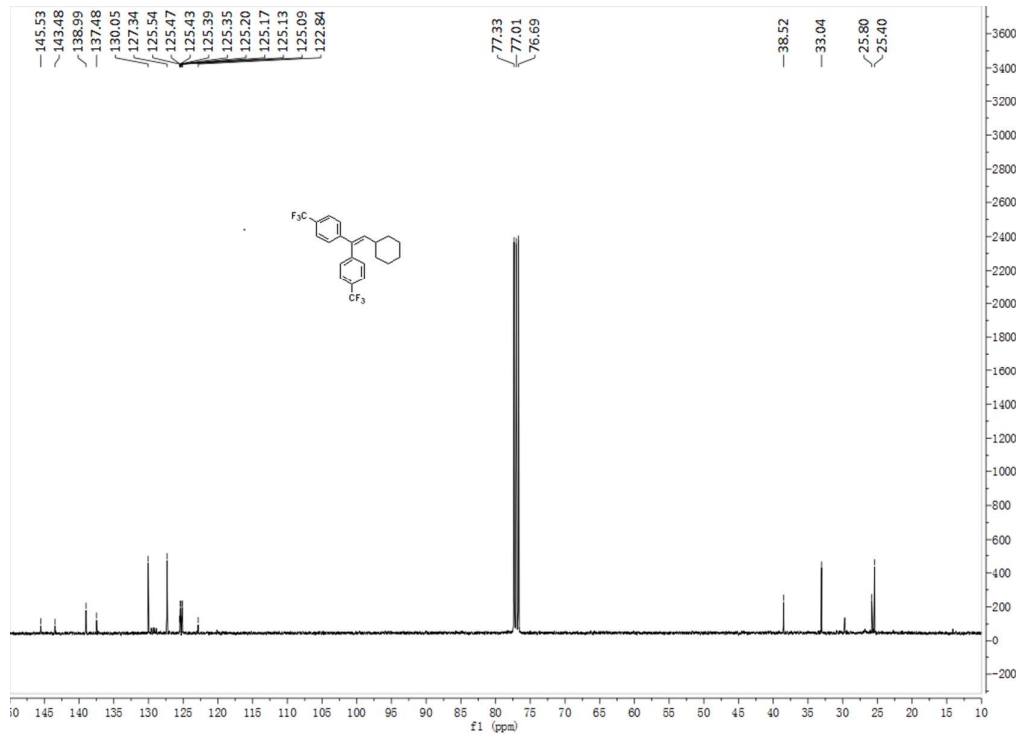
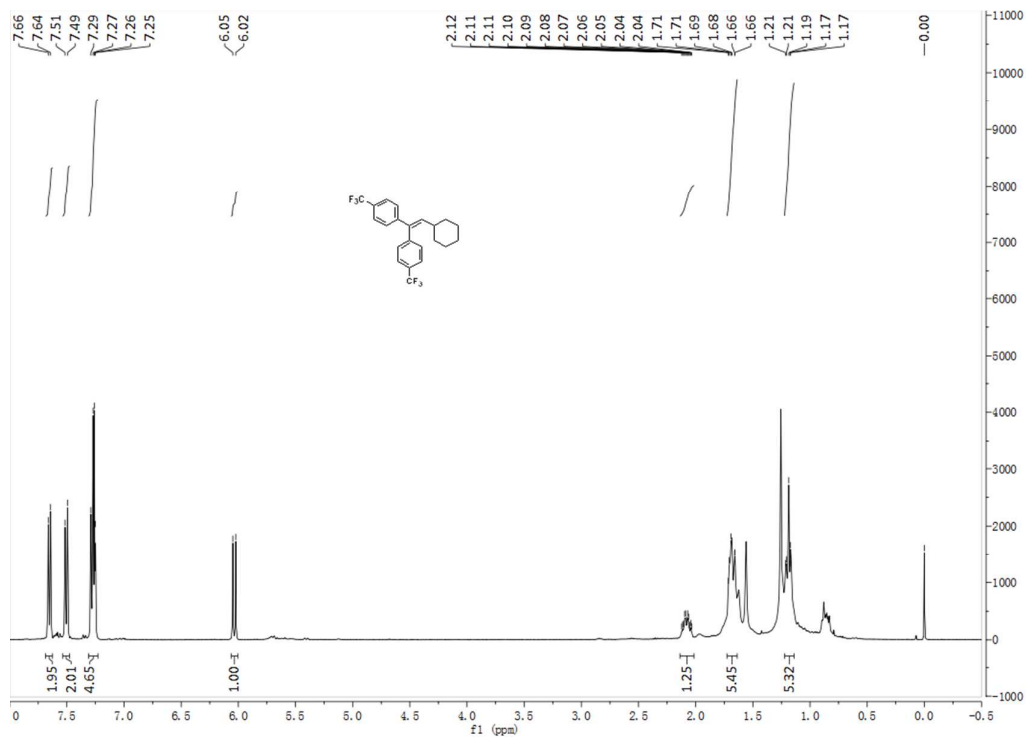
^1H and ^{13}C NMR spectra of **3la**



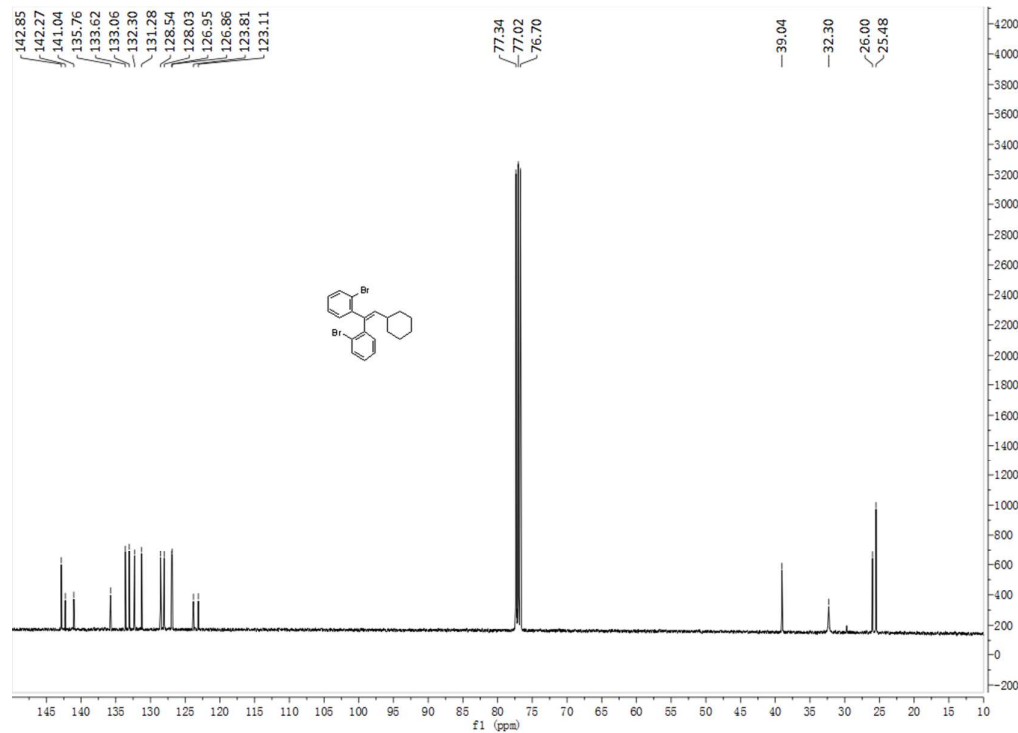
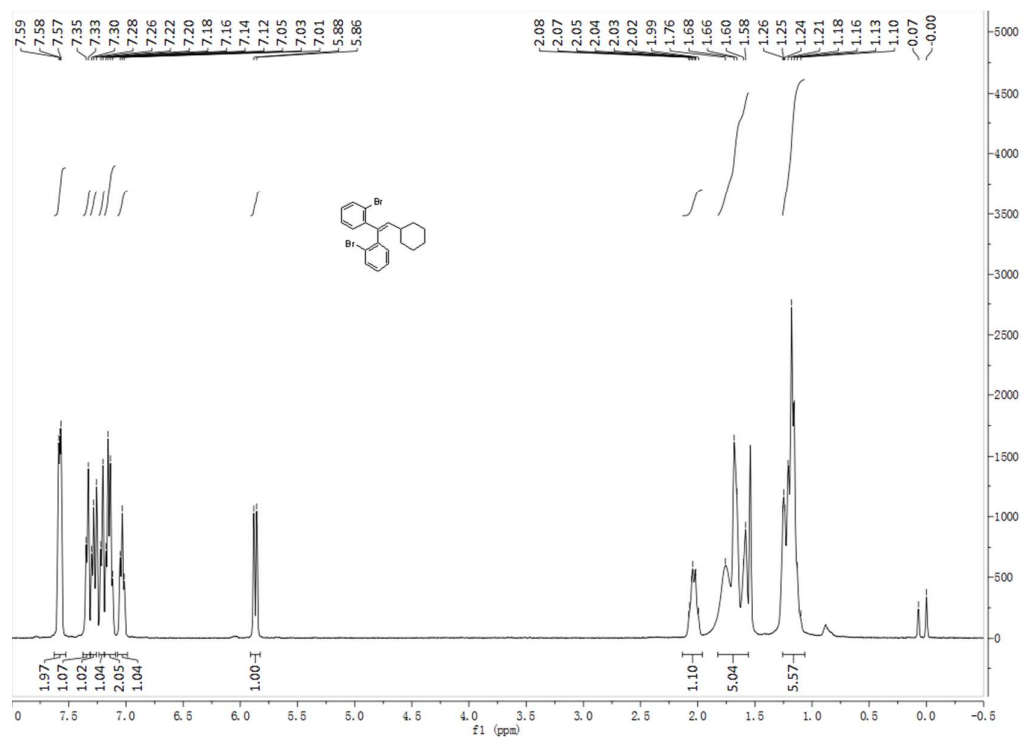
^1H and ^{13}C NMR spectra of **3ma**



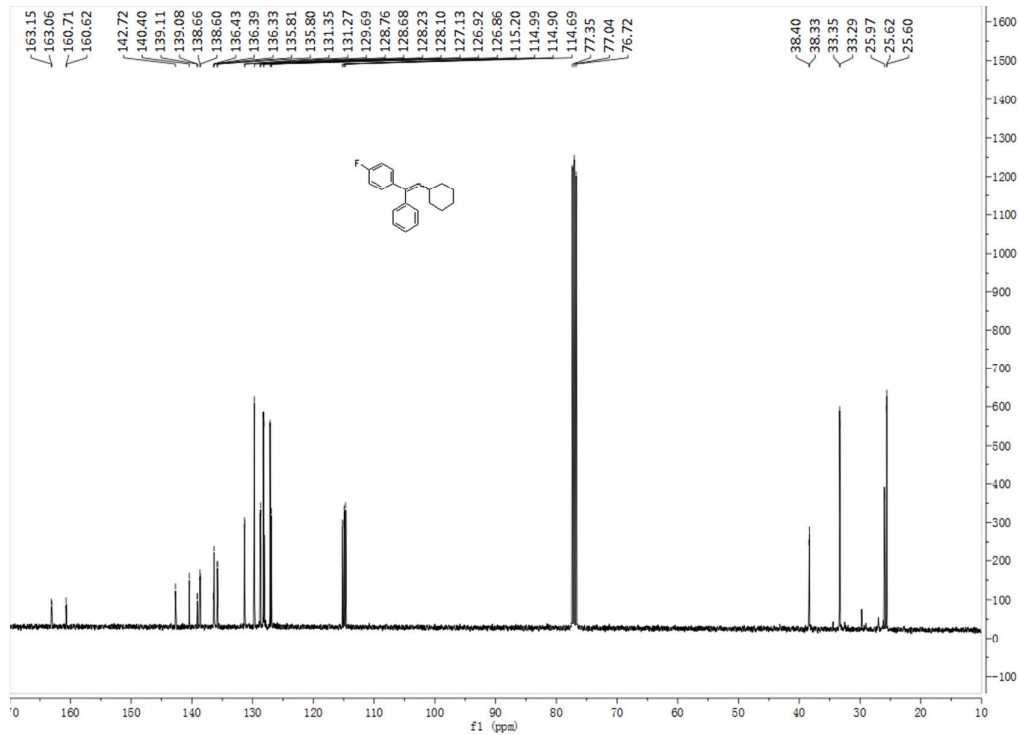
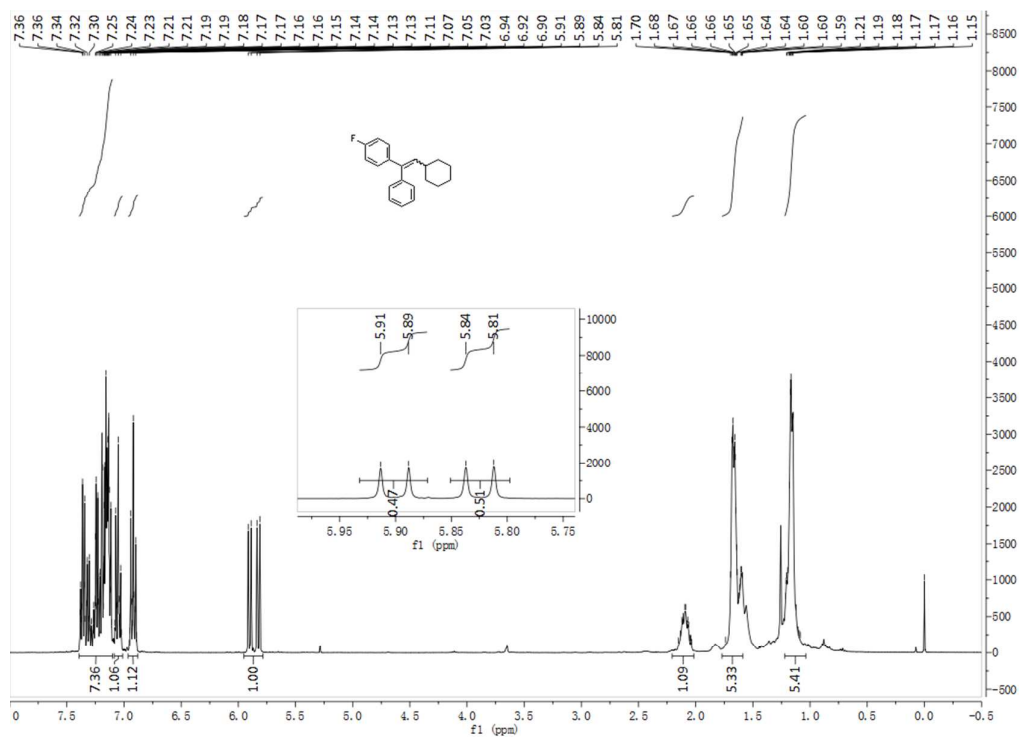
^1H and ^{13}C NMR spectra of **3na**



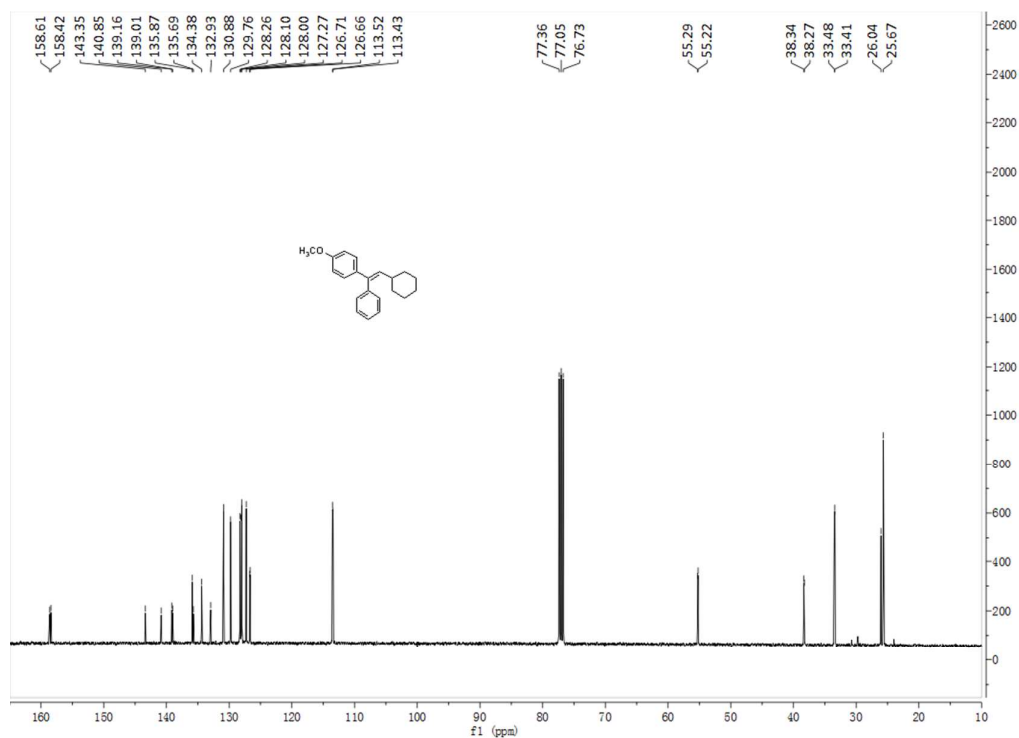
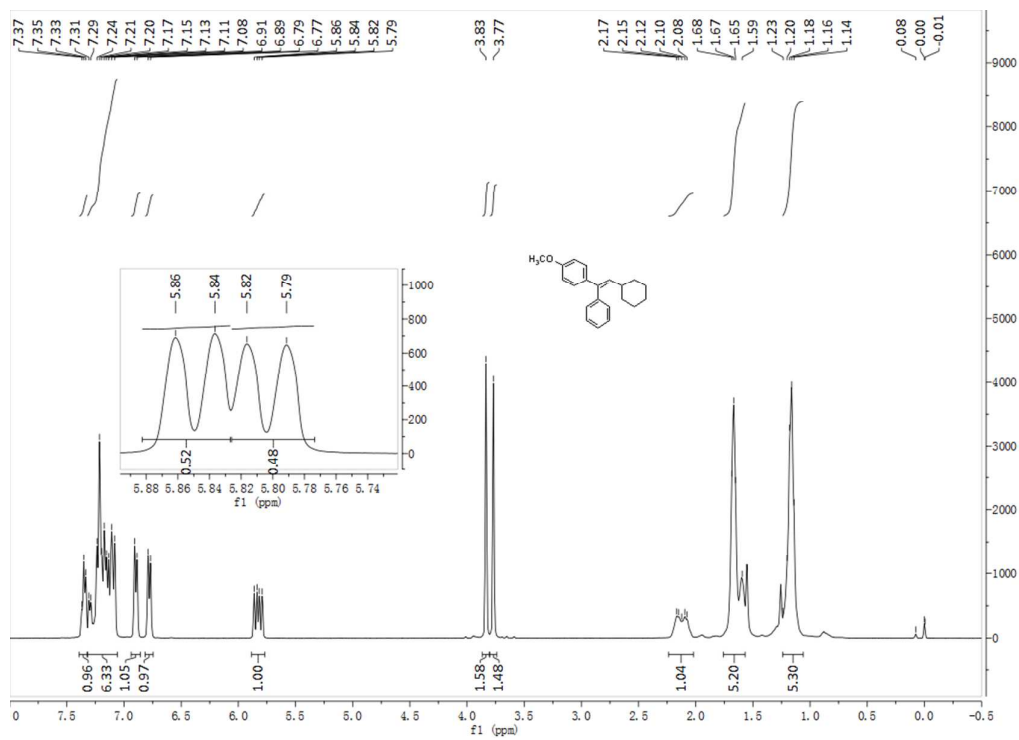
^1H and ^{13}C NMR spectra of **30a**



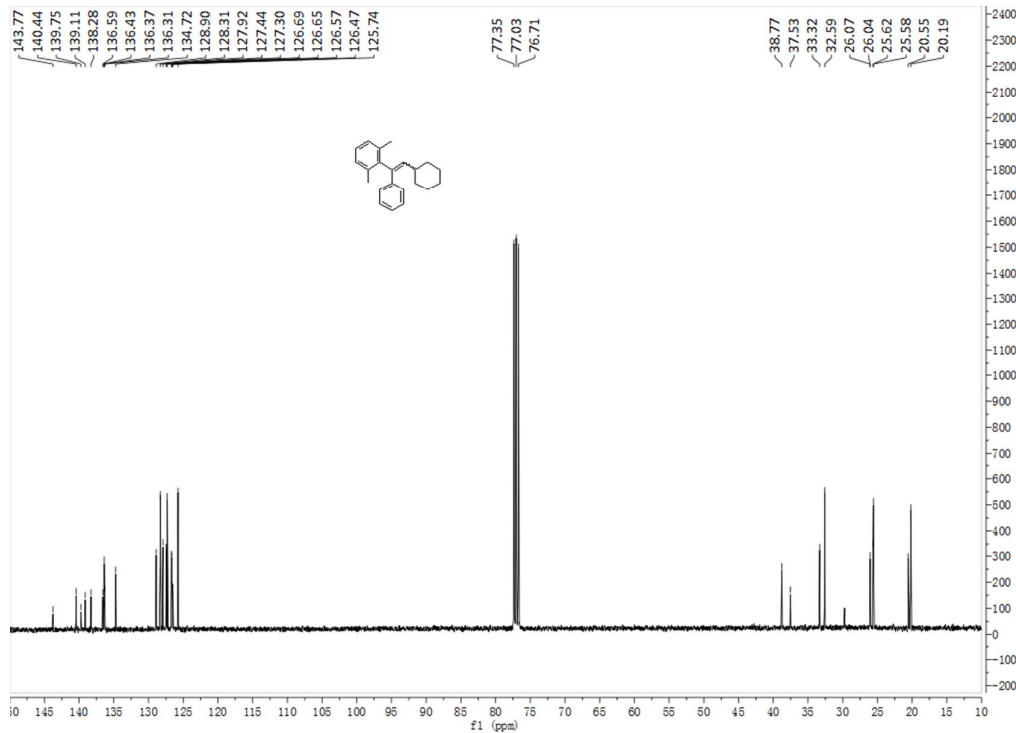
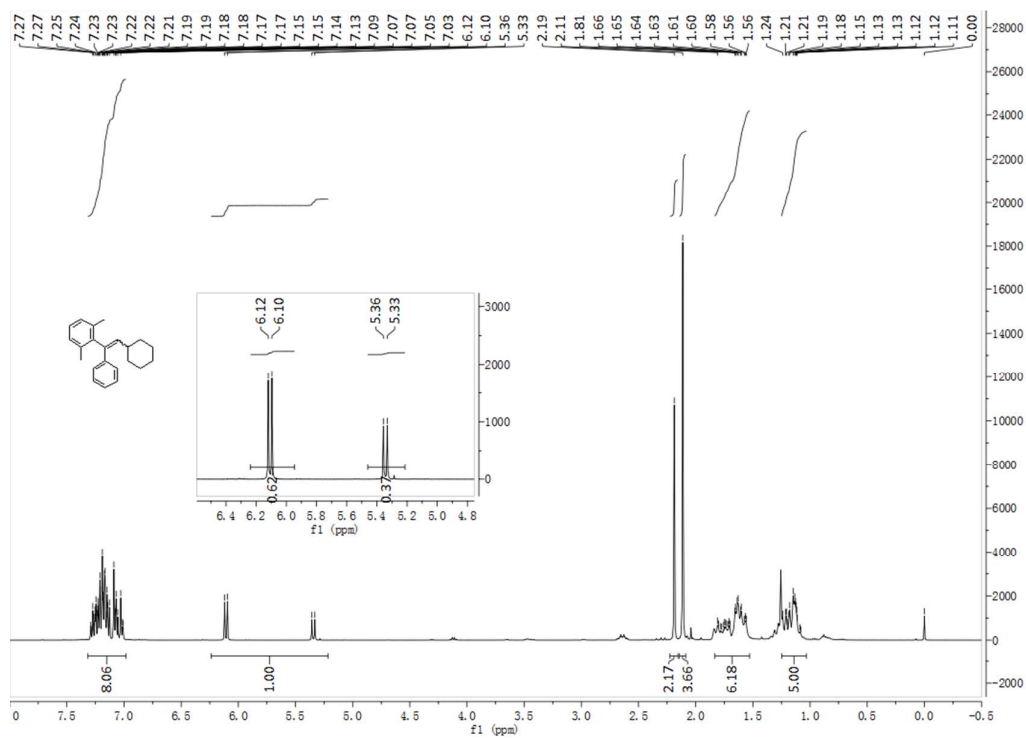
^1H and ^{13}C NMR spectra of **3pa**



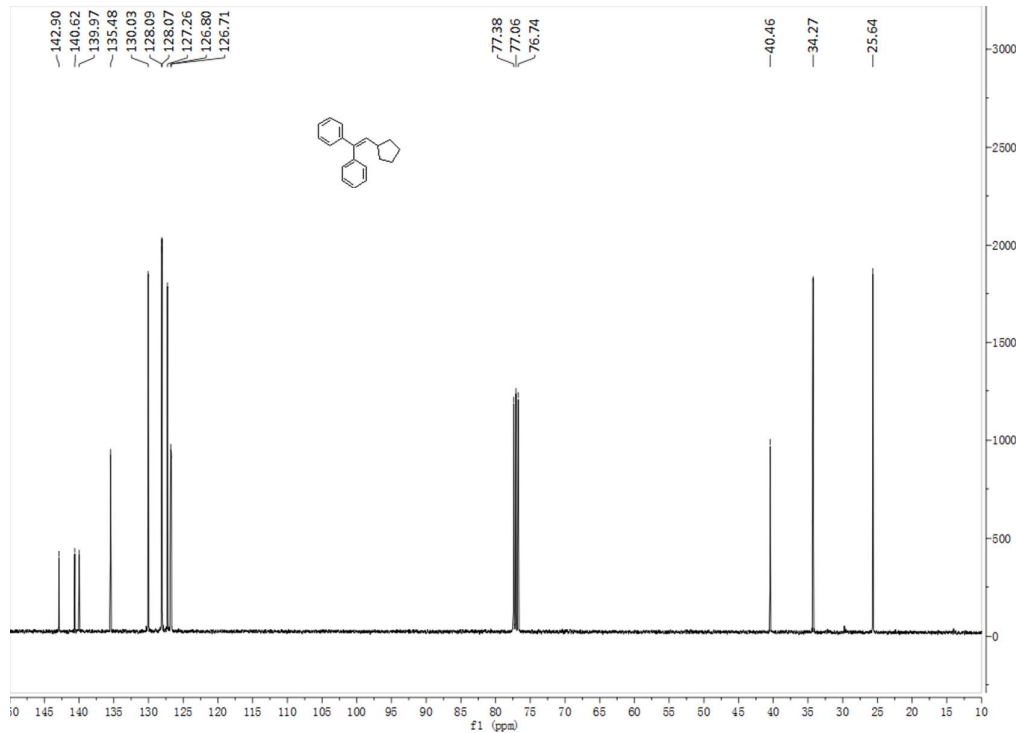
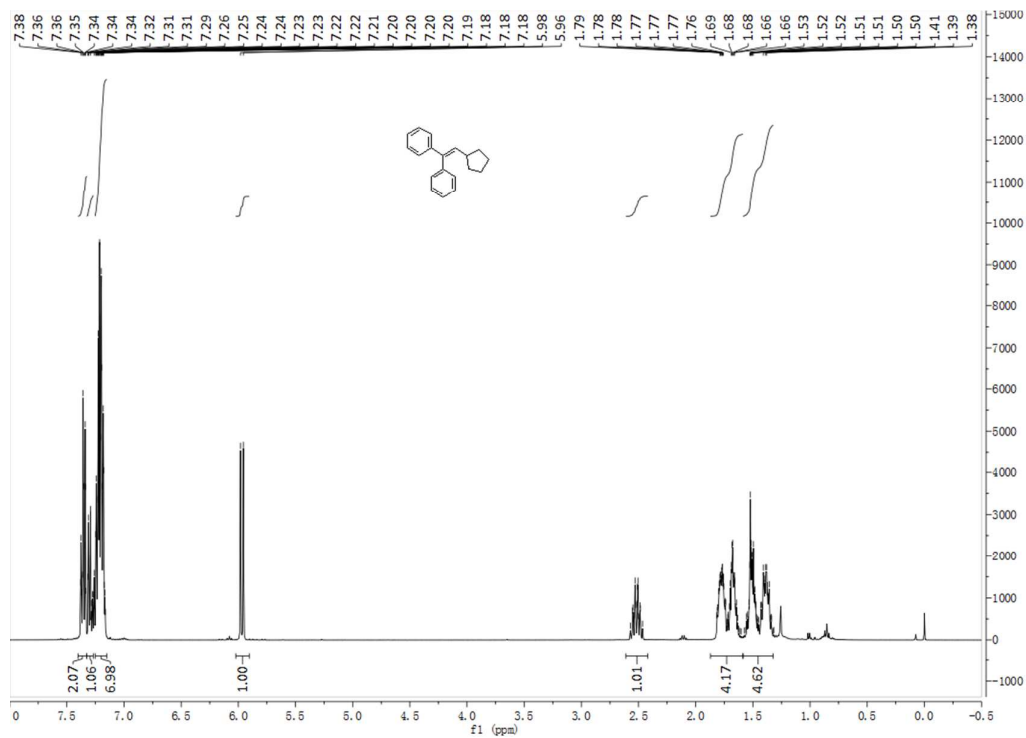
^1H and ^{13}C NMR spectra of **3qa**



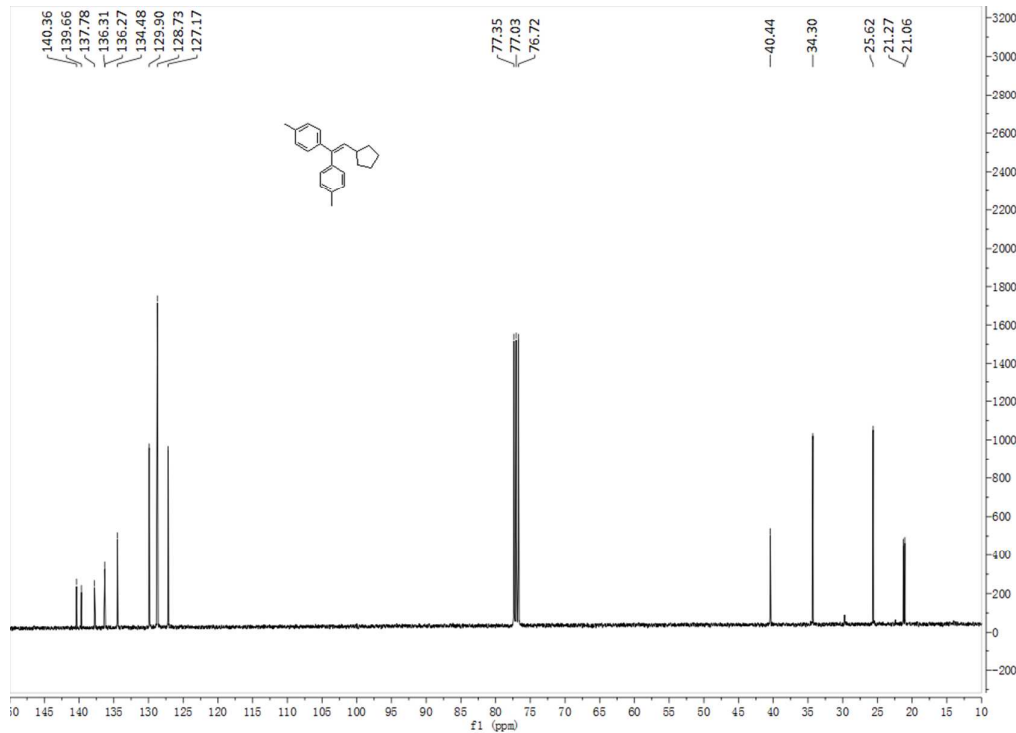
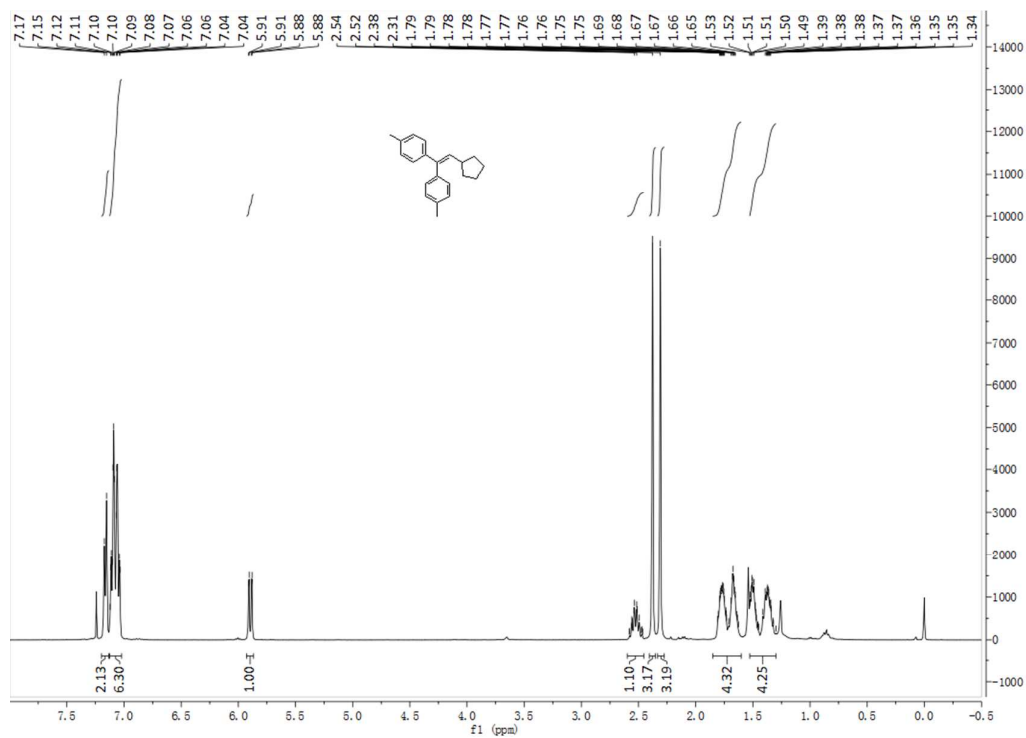
^1H and ^{13}C NMR spectra of **3ra**



^1H and ^{13}C NMR spectra of **3ab**



^1H and ^{13}C NMR spectra of **3bb**



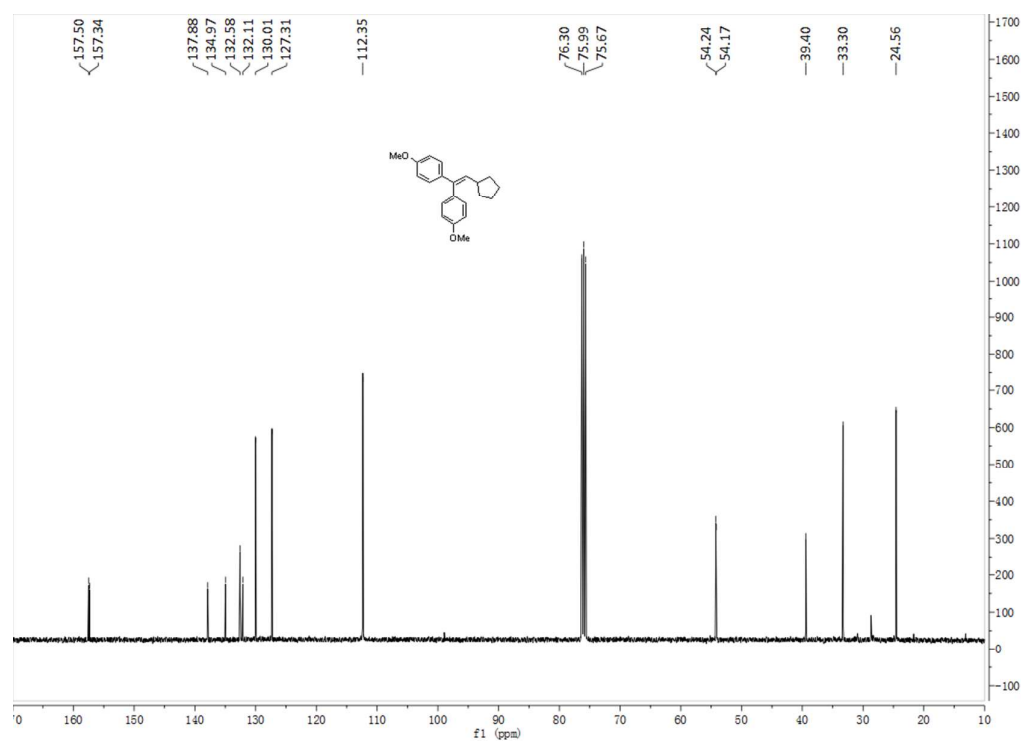
Chemical structure: COc1ccc(cc1)C(Cc2ccc(OC)cc2)C3=CC=CC=C3

¹H NMR spectrum (ppm):

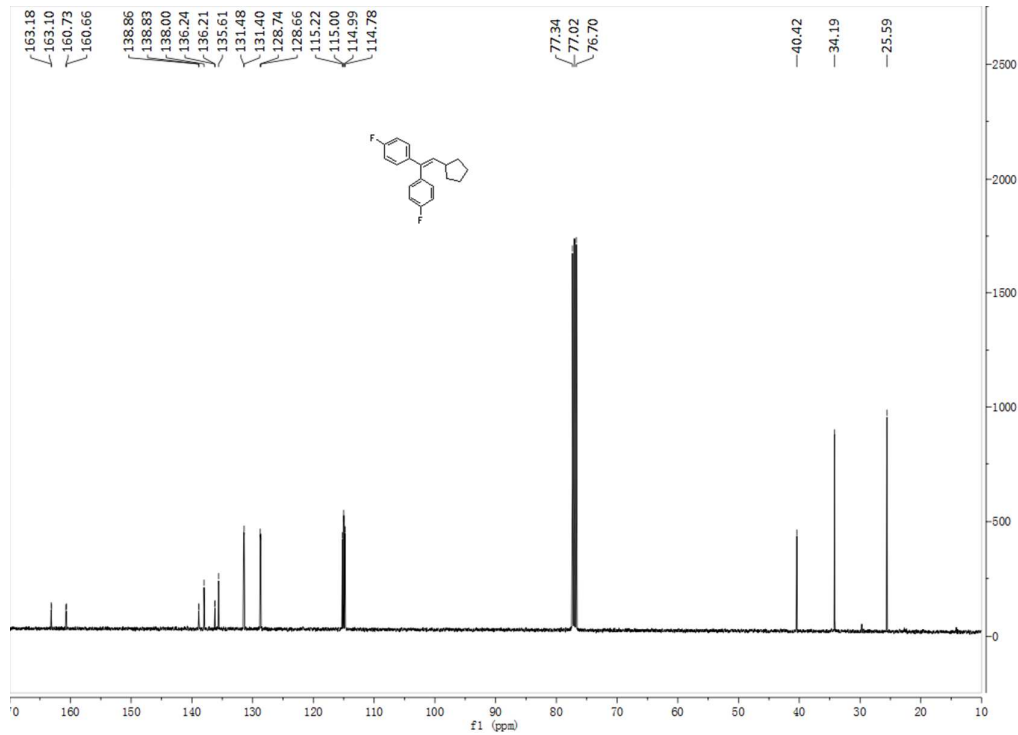
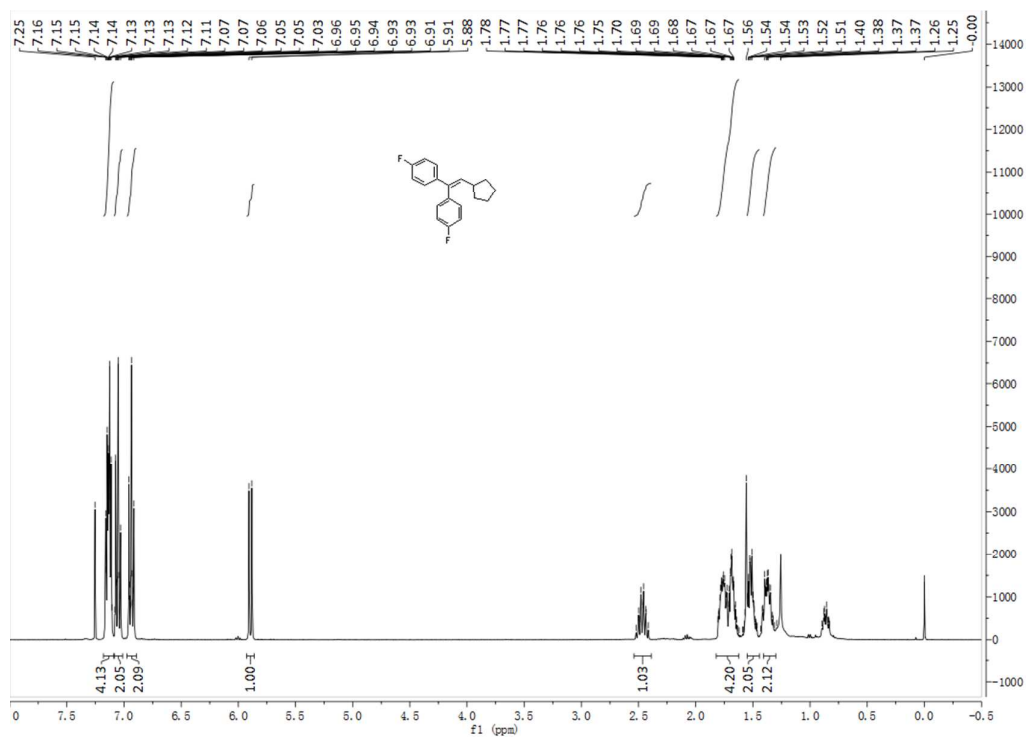
- 7.07, 7.06, 7.05, 7.04, 7.03, 7.02, 6.83, 6.81, 6.81, 6.81, 6.72, 6.71, 6.70, 5.76, 5.74, 3.76, 3.71, 2.46, 2.44, 1.71, 1.71, 1.70, 1.70, 1.69, 1.69, 1.69, 1.68, 1.62, 1.61, 1.61, 1.60, 1.59, 1.59, 1.58, 1.45, 1.44, 1.44, 1.42, 1.42, 1.32, 1.31, 1.30, 1.30, 1.29, 1.28, 1.26, 0.00, 0.00, -0.07

Integration values:

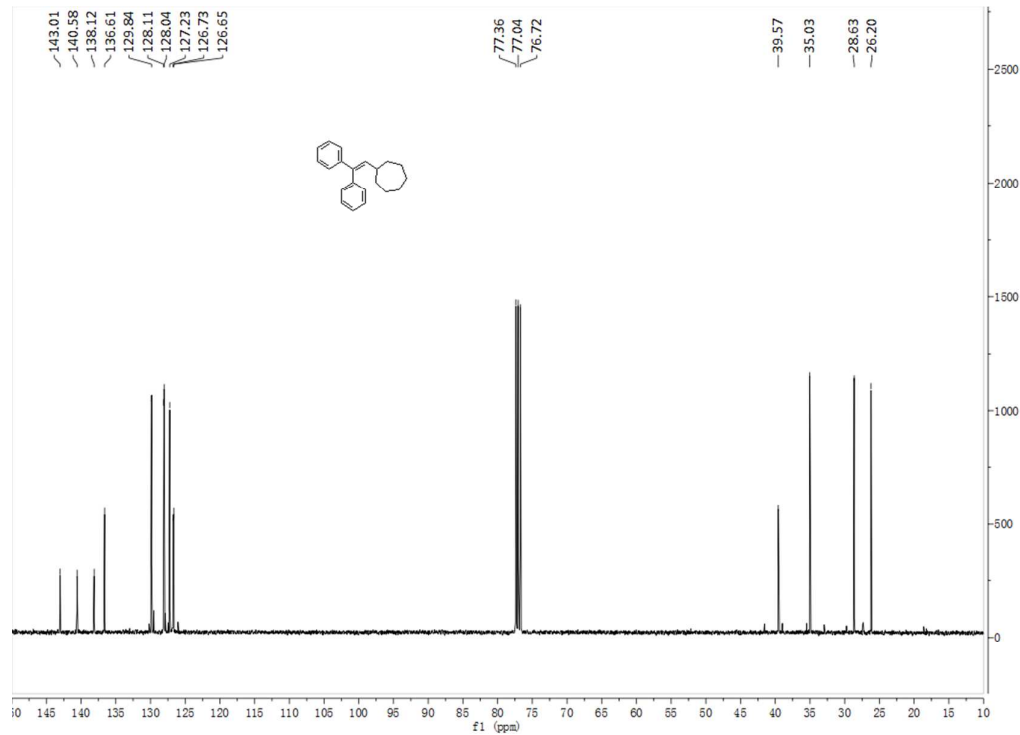
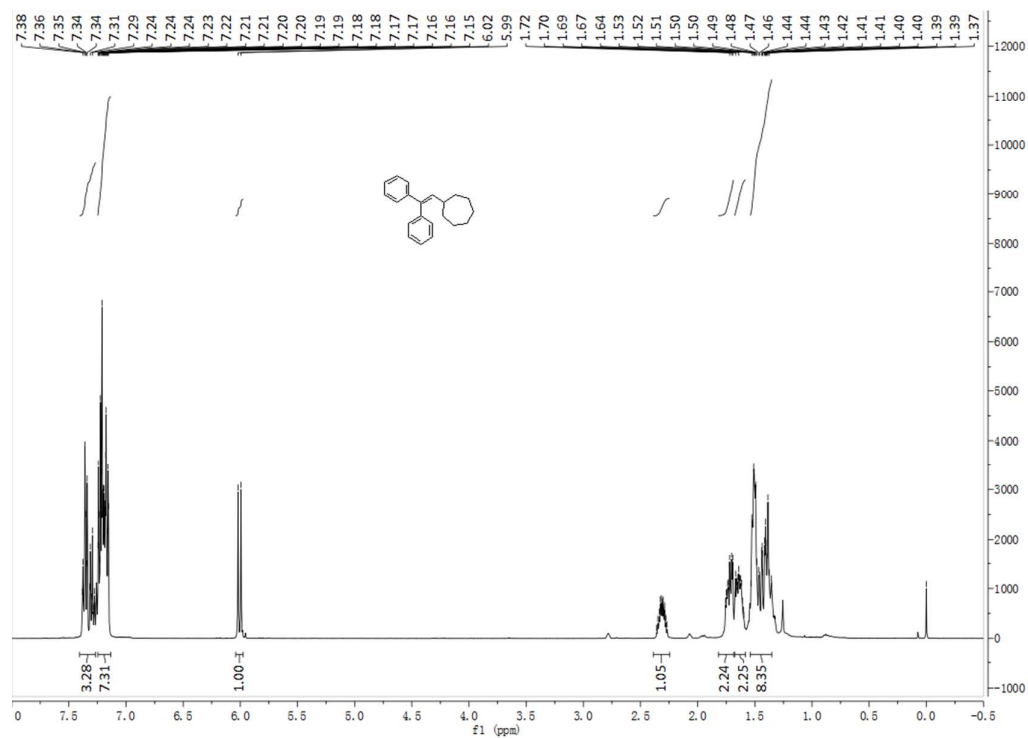
- 4.27, 2.13, 2.15, 1.00, 3.14, 3.22, 1.09, 2.25, 2.31, 2.15, 2.61



^1H and ^{13}C NMR spectra of **3fb**



^1H and ^{13}C NMR spectra of **3ac**



^1H and ^{13}C NMR spectra of **3af**

