

Supporting Information

**Copper-catalyzed 1,1-aryalkylation of terminal alkynes with diazo
esters and organoboronic acids**

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1. General Information

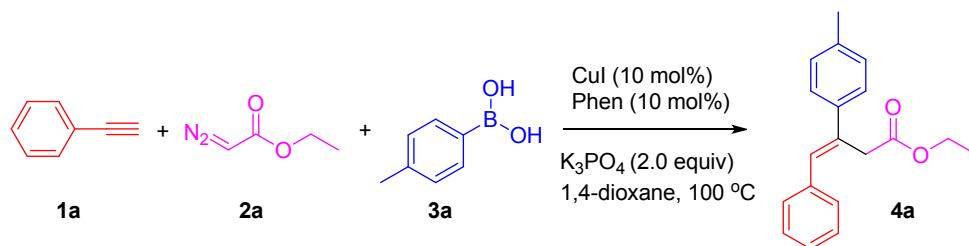
All deuterated solvents were purchased from Cambridge Isotope Laboratories. ^1H NMR and ^{13}C NMR spectra were recorded at 25 °C on a Bruker Advance 400 spectrometer (^1H : 400 MHz and ^{13}C :100 MHz). ^1H NMR chemical shifts were determined relative to internal $(\text{CH}_3)_4\text{Si}$ (TMS) at δ 0.00 ppm or to the signal of the residual protonated solvent: CDCl_3 at δ 7.26 ppm. ^{13}C NMR chemical shifts were determined relative to the signal of the solvent: CDCl_3 at δ 77.00 ppm. Data for ^1H , ^{13}C NMR were recorded as follows: chemical shift (δ , ppm), multiplicity (s = singlet, d = doublet, t = triplet, m = multiplet, q = quartet, dd = doublet of doublets), coupling constants (Hz) and integration. Melting points were obtained with a micro melting point XT4A Beijing Keyi electrooptic apparatus and are uncorrected. High-resolution mass data were recorded on a Waters LCT PremierxeTM (USA). Single-crystal X-ray crystallography was carried out on a Bruker Smart Apex II diffractometer system.

Materials and Methods:

Unless otherwise stated, starting materials were purchased from Aldrich or Energy-Chemical Limited and used as supplied without further purification. Solvents were used directly without further purification. The following starting materials were prepared according to the procedures described previously in the literature: **2b**,^[1] **2c**,^[2] **2d**,^[2] **4a'**,^[3] and **4a''**.^[4] The 1,1-aryllkylation of alkynes were performed under a nitrogen atmosphere in flame dried flasks. All reactions were monitored by thin layer chromatography (TLC) with Taizhou GF254 silica gel coated plates. Flash column chromatography was carried out using 200-300 mesh silica gel at increased pressure.

2. General Procedure for 1,1-Arylalkylation of Terminal Alkynes

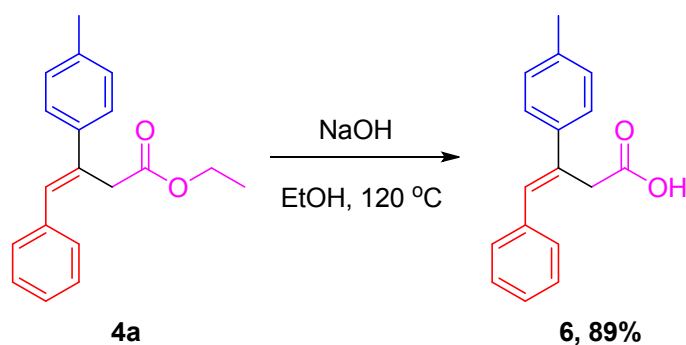
4a as an example



To a solution of the ethyl 2-diazoacetate **2a** (33 μ L, 0.3 mmol) in 1,4-dioxane (1.0 mL) was added the ethynylbenzene **1a** (41 μ L, 0.36 mmol), *p*-tolylboronic acid **3a** (124.9 mg, 0.9 mmol), Phen (5.4 mg, 0.03 mmol), CuI (5.7 mg, 0.03 mmol), and K₃PO₄ (130.0 mg, 0.6 mmol) under a N₂ atmosphere in a Schlenk tube. The reaction mixture was stirred at 100 °C for 1.5 h. After the reaction finished, the reaction mixture was cooled to room temperature and quenched by water. The mixture was extracted with EtOAc (3.0 mL \times 3), the combined organic phases were dried over anhydrous Na₂SO₄ and the solvent was evaporated under vacuum. The residue was purified by column chromatography to give the corresponding products **4a** (71.0 mg, 85%).

3. Synthetic Applications

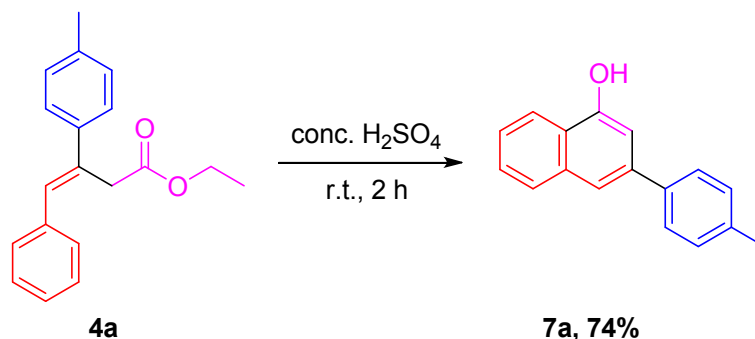
3.1 Hydrolysis of Product 4a



Compound **4a** (56.0 mg, 0.2 mmol) was taken in a Schlenk tube in EtOH (5 mL) with NaOH (0.4 g, 10 mmol). Schlenk tube was tightly closed with teflon cap and heated at 110 °C for 6 h. After completion of the reaction, the mixture was cooled to room temperature. Then 2N HCl was added to the aqueous layer until pH = 2. Next, the aqueous layer was extracted with EtOAc (2 x 20 mL). The organic layer was collected and dried over MgSO₄. After concentration in vacuum, the residue was purified by column chromatography to give the corresponding products **6** (45.0 mg, 89%).

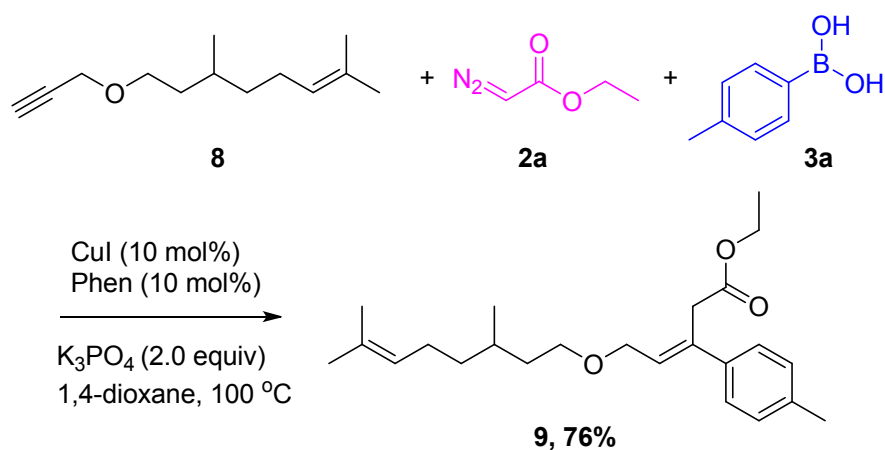
3.2 Synthesis of 7

7a as an example



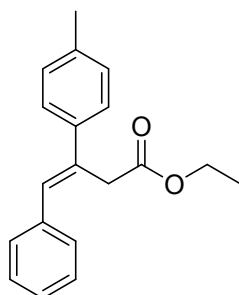
Following the general procedure described previously in the literature,^[5] (*E*)-ethyl 4-phenyl-3-(*p*-tolyl)but-3-enoate **4a** (84 mg, 0.3 mmol) was dissolved in conc. H₂SO₄ (300 mg) and stirred at room temperature for 2 h. The reaction mixture was carefully poured over cold water (2 mL). Next, the aqueous layer was extracted with EtOAc (3 x 5 mL), and the product **7a** (52 mg, 74%) was isolated by flash chromatography.

3.3 Synthesis of 9



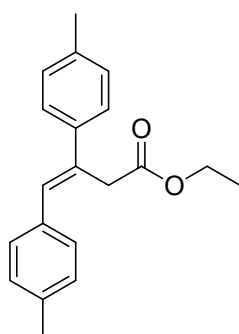
Following the abovementioned general procedure in S2, the reaction with **8** (70 mg, 0.36 mmol), **2a** (33 μ L, 0.3 mmol), **3a** (124.9 mg, 0.9 mmol), Phen (5.4 mg, 0.03 mmol), CuI (5.7 mg, 0.03 mmol), and K₃PO₄ (130.0 mg, 0.6 mmol) under a N₂ atmosphere for 1.5 h at 100 °C afforded **9** as colorless oil (85 mg, 76% yield).

4. Analytical Data of Compounds 4-7 and 9



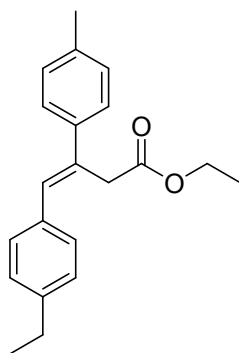
(E)-ethyl 4-phenyl-3-(p-tolyl)but-3-enoate 4a

Colorless oil. $^1\text{H NMR}$ (400 MHz, CDCl_3): δ = 1.18 (t, J = 7.2 Hz, 3H), 2.38 (s, 3H), 3.71 (s, 2H), 4.12 (q, J = 7.2 Hz, 2H), 7.02 (s, 1H), 7.19 (d, J = 7.6 Hz, 2H), 7.28-7.43 (m, 7H); $^{13}\text{C NMR}$ (100 MHz, CDCl_3): δ = 14.1, 21.1, 36.6, 60.7, 126.0, 127.0, 128.4, 128.7, 129.1, 130.4, 134.5, 137.3, 137.5, 138.7, 171.6. HRMS (ESI-TOF). Calcd for $\text{C}_{19}\text{H}_{21}\text{O}_2$, $[\text{M}+\text{H}]^+$ m/z 281.1542, Found 281.1537.



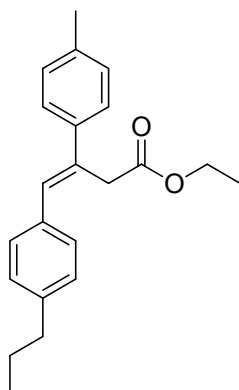
(E)-ethyl 3,4-di-p-tolylbut-3-enoate 4b

Colorless oil. $^1\text{H NMR}$ (400 MHz, CDCl_3): δ = 1.19 (t, J = 7.2 Hz, 3H), 2.37 (s, 3H), 2.38 (s, 3H), 3.71 (s, 2H), 4.12 (q, J = 7.2 Hz, 2H), 6.99 (s, 1H), 7.18 (d, J = 5.2 Hz, 2H), 7.20 (d, J = 5.2 Hz, 2H), 7.29 (d, J = 8.0 Hz, 2H), 7.41 (d, J = 8.0 Hz, 2H); $^{13}\text{C NMR}$ (100 MHz, CDCl_3): δ = 14.1, 21.1, 21.2, 36.6, 60.7, 126.0, 128.6, 129.1, 129.1, 130.4, 133.8, 134.6, 136.8, 137.2, 138.9, 171.7. HRMS (ESI-TOF). Calcd for $\text{C}_{20}\text{H}_{23}\text{O}_2$, $[\text{M}+\text{H}]^+$ m/z 295.1698, Found 295.1688.



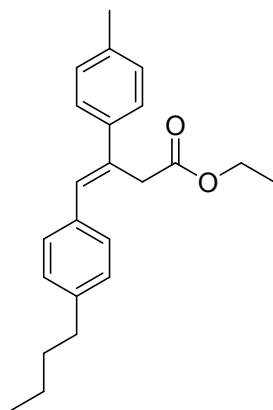
(E)-ethyl 4-(4-ethylphenyl)-3-(p-tolyl)but-3-enoate 4c

Colorless oil. ^1H NMR (400 MHz, CDCl_3): δ = 1.18 (t, J = 7.2 Hz, 3H), 1.26 (t, J = 7.6 Hz, 3H), 2.37 (s, 3H), 2.67 (q, J = 7.6 Hz, 2H), 3.71 (s, 2H), 4.11 (q, J = 7.2 Hz, 2H), 6.98 (s, 1H), 7.17 (d, J = 8.0 Hz, 2H), 7.21 (d, J = 8.0 Hz, 2H), 7.31 (d, J = 8.0 Hz, 2H), 7.40 (d, J = 8.0 Hz, 2H); ^{13}C NMR (100 MHz, CDCl_3): δ = 14.1, 15.5, 21.1, 28.6, 36.7, 60.7, 126.0, 127.9, 128.7, 129.1, 130.4, 133.9, 134.9, 137.2, 139.0, 143.2, 171.7. HRMS (ESI-TOF). Calcd for $\text{C}_{21}\text{H}_{24}\text{NaO}_2$, $[\text{M}+\text{Na}]^+$ m/z 331.1674, Found 331.1660.



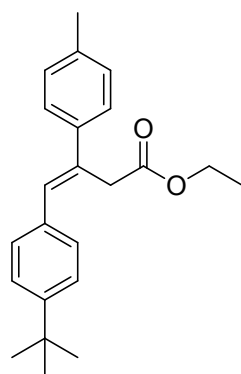
(E)-ethyl 4-(4-propylphenyl)-3-(p-tolyl)but-3-enoate 4d

Colorless oil. ^1H NMR (400 MHz, CDCl_3): δ = 0.98 (t, J = 7.2 Hz, 3H), 1.19 (t, J = 7.2 Hz, 3H), 1.65-1.70 (m, 2H), 2.37 (s, 3H), 2.62 (t, J = 7.6 Hz, 2H), 3.73 (s, 2H), 4.12 (q, J = 7.2 Hz, 2H), 6.99 (s, 1H), 7.18 (d, J = 6.4 Hz, 2H), 7.20 (d, J = 6.4 Hz, 2H), 7.31 (d, J = 8.0 Hz, 2H), 7.41 (d, J = 8.0 Hz, 2H); ^{13}C NMR (100 MHz, CDCl_3): δ = 13.8, 14.1, 21.1, 24.5, 36.6, 37.8, 60.7, 126.0, 128.5, 128.6, 129.1, 130.4, 133.8, 134.9, 137.2, 138.9, 141.6, 171.7. HRMS (ESI-TOF). Calcd for $\text{C}_{22}\text{H}_{27}\text{O}_2$, $[\text{M}+\text{H}]^+$ m/z 323.2011, Found 323.1998.



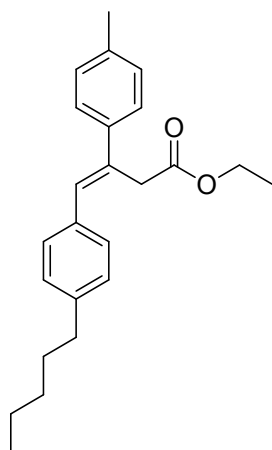
(E)-ethyl 4-(4-butylphenyl)-3-(p-tolyl)but-3-enoate 4e

Colorless oil. ^1H NMR (400 MHz, CDCl_3): δ = 0.95 (t, J = 7.2 Hz, 3H), 1.18 (t, J = 7.2 Hz, 3H), 1.34-1.43 (m, 2H), 1.58-1.66 (m, 2H), 2.37 (s, 3H), 2.63 (t, J = 7.6 Hz, 2H), 3.72 (s, 2H), 4.12 (q, J = 7.2 Hz, 2H), 6.99 (s, 1H), 7.17-7.20 (m, 4H), 7.30 (d, J = 8.0 Hz, 2H), 7.40 (d, J = 8.4 Hz, 2H); ^{13}C NMR (100 MHz, CDCl_3): δ = 14.0, 14.1, 21.1, 22.4, 33.6, 35.4, 36.7, 60.7, 126.0, 128.4, 128.7, 129.1, 130.4, 133.8, 134.8, 137.2, 138.9, 141.9, 171.7. HRMS (ESI-TOF). Calcd for $\text{C}_{23}\text{H}_{29}\text{O}_2$, $[\text{M}+\text{H}]^+$ m/z 337.2168, Found 337.2168.



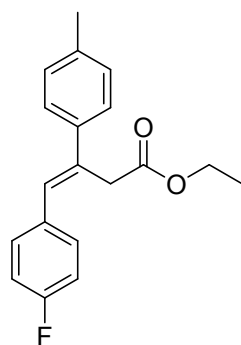
(E)-ethyl 4-(4-tert-butylphenyl)-3-(p-tolyl)but-3-enoate 4f

Colorless oil. ^1H NMR (400 MHz, CDCl_3): δ = 1.18 (t, J = 7.2 Hz, 3H), 1.35 (s, 9H), 2.37 (s, 3H), 3.73 (s, 2H), 4.12 (q, J = 7.2 Hz, 2H), 6.99 (s, 1H), 7.18 (d, J = 8.0 Hz, 2H), 7.34 (d, J = 8.4 Hz, 2H), 7.39-7.42 (m, 4H); ^{13}C NMR (100 MHz, CDCl_3): δ = 14.1, 21.1, 31.3, 34.6, 36.7, 60.7, 125.3, 126.0, 128.5, 129.1, 130.3, 133.9, 134.6, 137.2, 138.9, 150.0, 171.7. HRMS (ESI-TOF). Calcd for $\text{C}_{23}\text{H}_{29}\text{O}_2$, $[\text{M}+\text{H}]^+$ m/z 337.2168, Found 337.2170.



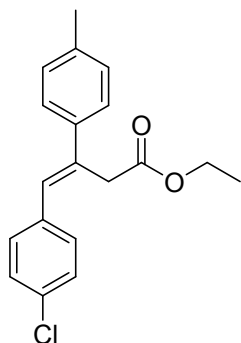
(E)-ethyl 4-(4-pentylphenyl)-3-(p-tolyl)but-3-enoate 4g

Colorless oil. ^1H NMR (400 MHz, CDCl_3): δ = 0.91 (t, J = 6.4 Hz, 3H), 1.18 (t, J = 7.2 Hz, 3H), 1.28-1.36 (m, 4H), 1.59-1.66 (m, 2H), 2.37 (s, 3H), 2.62 (t, J = 7.6 Hz, 2H), 3.72 (s, 2H), 4.12 (q, J = 7.2 Hz, 2H), 6.99 (s, 1H), 7.15-7.20 (m, 4H), 7.30 (d, J = 7.6 Hz, 2H), 7.40 (d, J = 7.6 Hz, 2H); ^{13}C NMR (100 MHz, CDCl_3): δ = 14.0, 14.1, 21.1, 22.5, 31.1, 31.5, 35.7, 36.7, 60.7, 126.0, 128.4, 128.7, 129.1, 130.4, 133.8, 134.8, 137.2, 139.0, 141.9, 171.7. HRMS (ESI-TOF). Calcd for $\text{C}_{24}\text{H}_{30}\text{NaO}_2$, $[\text{M}+\text{Na}]^+$ m/z 373.2143, Found 373.2135.



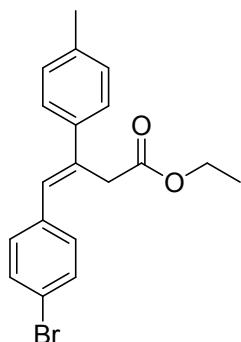
(E)-ethyl 4-(4-fluorophenyl)-3-(p-tolyl)but-3-enoate 4h

Colorless oil. ^1H NMR (400 MHz, CDCl_3): δ = 1.18 (t, J = 7.2 Hz, 3H), 2.37 (s, 3H), 3.65 (s, 2H), 4.11 (q, J = 7.2 Hz, 2H), 6.95 (s, 1H), 7.06 (t, J = 8.8 Hz, 2H), 7.18 (d, J = 8.0 Hz, 2H), 7.34-7.40 (m, 4H); ^{13}C NMR (100 MHz, CDCl_3): δ = 14.1, 21.1, 36.5, 60.8, 115.3 (d, J = 21.0 Hz), 126.0, 129.2, 129.3, 130.4 (d, J = 8.0 Hz), 133.6 (d, J = 4.0 Hz), 134.6, 137.5, 138.5, 161.9 (d, J = 245.0 Hz), 171.5. HRMS (ESI-TOF). Calcd for $\text{C}_{19}\text{H}_{20}\text{FO}_2$, $[\text{M}+\text{H}]^+$ m/z 299.1447, Found 299.1438.



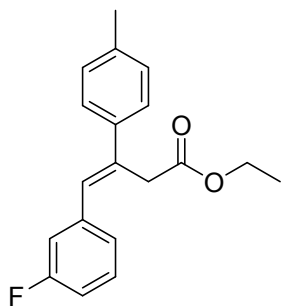
(E)-ethyl 4-(4-chlorophenyl)-3-(p-tolyl)but-3-enoate 4i

Colorless oil. $^1\text{H NMR}$ (400 MHz, CDCl_3): δ = 1.18 (t, J = 7.2 Hz, 3H), 2.37 (s, 3H), 3.65 (s, 2H), 4.11 (q, J = 7.2 Hz, 2H), 6.94 (s, 1H), 7.18 (d, J = 8.0 Hz, 2H), 7.31-7.36 (m, 4H), 7.39 (d, J = 8.0 Hz, 2H); $^{13}\text{C NMR}$ (100 MHz, CDCl_3): δ = 14.1, 21.1, 36.6, 60.9, 126.1, 128.6, 129.1, 129.2, 130.1, 132.9, 135.2, 136.0, 137.6, 138.4, 171.4. HRMS (ESI-TOF). Calcd for $\text{C}_{19}\text{H}_{20}\text{ClO}_2$, $[\text{M}+\text{H}]^+$ m/z 315.1152, Found 315.1148.



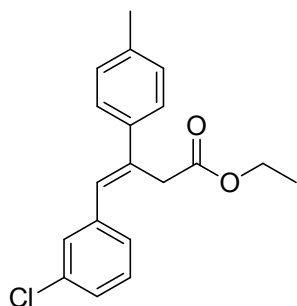
(E)-ethyl 4-(4-bromophenyl)-3-(p-tolyl)but-3-enoate 4j

Colorless oil. $^1\text{H NMR}$ (400 MHz, CDCl_3): δ = 1.16 (t, J = 7.2 Hz, 3H), 2.35 (s, 3H), 3.63 (s, 2H), 4.10 (q, J = 7.2 Hz, 2H), 6.90 (s, 1H), 7.17 (d, J = 8.0 Hz, 2H), 7.25 (d, J = 8.0 Hz, 2H), 7.37 (d, J = 8.0 Hz, 2H), 7.48 (d, J = 8.4 Hz, 2H); $^{13}\text{C NMR}$ (100 MHz, CDCl_3): δ = 14.1, 21.1, 36.5, 60.8, 121.0, 126.0, 129.1, 129.2, 130.4, 131.5, 135.2, 136.4, 137.6, 138.3, 171.3. HRMS (ESI-TOF). Calcd for $\text{C}_{19}\text{H}_{20}\text{BrO}_2$, $[\text{M}+\text{H}]^+$ m/z 359.0647, 361.0626, Found 359.0638, 361.0614.



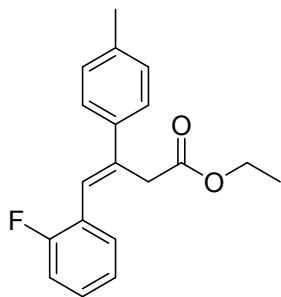
(E)-ethyl 4-(3-fluorophenyl)-3-(p-tolyl)but-3-enoate 4k

Colorless oil. $^1\text{H NMR}$ (400 MHz, CDCl_3): δ = 1.19 (t, J = 7.2 Hz, 3H), 2.38 (s, 3H), 3.69 (s, 2H), 4.13 (q, J = 7.2 Hz, 2H), 6.96 (s, 1H), 6.98-7.01 (m, 1H), 7.11-7.16 (m, 2H), 7.19 (d, J = 8.0 Hz, 2H), 7.31-7.37 (m, 1H), 7.40 (d, J = 8.0 Hz, 2H); $^{13}\text{C NMR}$ (100 MHz, CDCl_3): δ = 14.0, 21.1, 36.6, 60.9, 113.9 (d, J = 21.0 Hz), 115.5 (d, J = 22.0 Hz), 124.5 (d, J = 3.0 Hz), 126.1, 129.1 (d, J = 2.0 Hz), 129.2, 129.8 (d, J = 8.0 Hz), 135.6, 137.7, 138.3, 139.7 (d, J = 7.0 Hz), 162.7 (d, J = 244.0 Hz), 171.3. HRMS (ESI-TOF). Calcd for $\text{C}_{19}\text{H}_{20}\text{FO}_2$, $[\text{M}+\text{H}]^+$ m/z 299.1447, Found 299.1451.



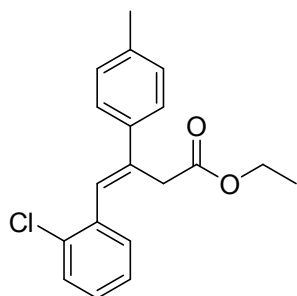
(E)-ethyl 4-(3-chlorophenyl)-3-(p-tolyl)but-3-enoate 4l

Colorless oil. $^1\text{H NMR}$ (400 MHz, CDCl_3): δ = 1.19 (t, J = 7.2 Hz, 3H), 2.36 (s, 3H), 3.65 (s, 2H), 4.11 (q, J = 7.2 Hz, 2H), 6.92 (s, 1H), 7.18 (d, J = 8.0 Hz, 2H), 7.24-7.32 (m, 3H), 7.37-7.39 (m, 3H); $^{13}\text{C NMR}$ (100 MHz, CDCl_3): δ = 14.1, 21.1, 36.7, 60.9, 126.1, 126.9, 127.1, 128.8, 128.9, 129.2, 129.6, 134.3, 135.8, 137.7, 138.3, 139.4, 171.2. HRMS (ESI-TOF). Calcd for $\text{C}_{19}\text{H}_{20}\text{ClO}_2$, $[\text{M}+\text{H}]^+$ m/z 315.1152, Found 315.1141.



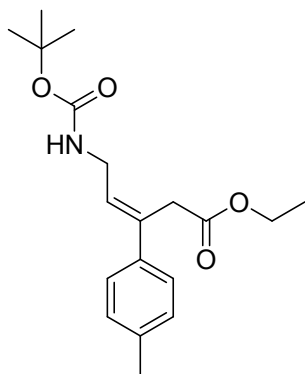
(E)-ethyl 4-(2-fluorophenyl)-3-(p-tolyl)but-3-enoate 4m

Colorless oil. $^1\text{H NMR}$ (400 MHz, CDCl_3): δ = 1.16 (t, J = 7.2 Hz, 3H), 2.37 (s, 3H), 3.65 (s, 2H), 4.09 (q, J = 7.2 Hz, 2H), 6.95 (s, 1H), 7.06-7.15 (m, 2H), 7.18 (d, J = 8.0 Hz, 2H), 7.26-7.30 (m, 1H), 7.41 (d, J = 8.0 Hz, 2H), 7.44-7.47 (m, 1H); $^{13}\text{C NMR}$ (100 MHz, CDCl_3): δ = 14.0, 21.1, 36.8, 60.8, 115.4 (d, J = 22.0 Hz), 123.1 (d, J = 4.0 Hz), 123.9 (d, J = 4.0 Hz), 125.2 (d, J = 15.0 Hz), 126.1, 128.3, 129.0 (d, J = 8.0 Hz), 129.1, 130.3 (d, J = 3.0 Hz), 136.5, 137.9 (d, J = 65.0 Hz), 160.5 (d, J = 246.0 Hz), 171.3. HRMS (ESI-TOF). Calcd for $\text{C}_{19}\text{H}_{20}\text{FO}_2$, $[\text{M}+\text{H}]^+$ m/z 299.1447, Found 299.1438.



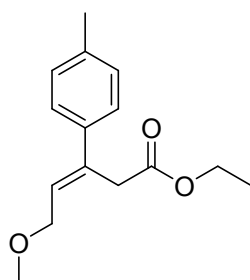
(E)-ethyl 4-(2-chlorophenyl)-3-(p-tolyl)but-3-enoate 4n

Colorless oil. $^1\text{H NMR}$ (400 MHz, CDCl_3): δ = 1.16 (t, J = 7.2 Hz, 3H), 2.37 (s, 3H), 3.60 (s, 2H), 4.09 (q, J = 7.2 Hz, 2H), 7.02 (s, 1H), 7.19 (d, J = 8.0 Hz, 2H), 7.22-7.33 (m, 2H), 7.41-7.44 (m, 3H), 7.51 (dd, J_1 = 1.6 Hz, J_2 = 6.8 Hz, 1H); $^{13}\text{C NMR}$ (100 MHz, CDCl_3): δ = 14.0, 21.1, 36.7, 60.8, 126.2, 126.6, 127.6, 128.6, 129.2, 129.4, 130.3, 134.3, 135.7, 135.9, 137.7, 138.1, 171.4. HRMS (ESI-TOF). Calcd for $\text{C}_{19}\text{H}_{20}\text{ClO}_2$, $[\text{M}+\text{H}]^+$ m/z 315.1152, Found 315.1149.



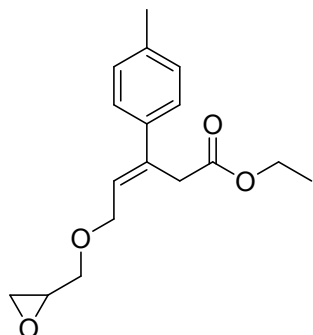
(E)-ethyl 5-((tert-butoxycarbonyl)amino)-3-(p-tolyl)pent-3-enoate 4o

Colorless oil. $^1\text{H NMR}$ (400 MHz, CDCl_3): δ = 1.22 (t, J = 7.2 Hz, 3H), 1.45 (s, 9H), 2.33 (s, 3H), 3.53 (s, 2H), 3.92 (t, J = 5.6 Hz, 2H), 4.12 (q, J = 7.2 Hz, 2H), 4.86 (s, 1H), 5.97 (t, J = 6.8 Hz, 1H), 7.12 (d, J = 8.0 Hz, 2H), 7.26 (d, J = 8.0 Hz, 2H); $^{13}\text{C NMR}$ (100 MHz, CDCl_3): δ = 14.1, 21.0, 28.4, 36.0, 38.9, 61.0, 79.3, 125.8, 127.2, 129.1, 135.3, 137.3, 138.4, 155.8, 171.1. HRMS (ESI-TOF). Calcd for $\text{C}_{19}\text{H}_{28}\text{NO}_4$, $[\text{M}+\text{H}]^+$ m/z 334.2018, Found 334.2032.



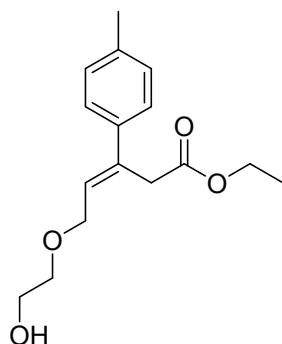
(E)-ethyl 5-methoxy-3-(p-tolyl)pent-3-enoate 4p

Colorless oil. $^1\text{H NMR}$ (400 MHz, CDCl_3): δ = 1.19 (t, J = 7.2 Hz, 3H), 2.33 (s, 3H), 3.39 (s, 3H), 3.52 (s, 2H), 4.10 (q, J = 7.2 Hz, 2H), 4.16 (d, J = 6.4 Hz, 2H), 6.07 (t, J = 6.4 Hz, 1H), 7.13 (d, J = 8.0 Hz, 2H), 7.31 (d, J = 8.0 Hz, 2H); $^{13}\text{C NMR}$ (100 MHz, CDCl_3): δ = 14.1, 21.0, 36.4, 58.2, 60.8, 69.4, 125.9, 127.5, 129.0, 135.4, 137.2, 138.5, 170.9. HRMS (ESI-TOF). Calcd for $\text{C}_{15}\text{H}_{20}\text{NaO}_3$, $[\text{M}+\text{Na}]^+$ m/z 271.1310, Found 271.1303.



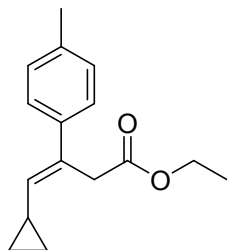
(E)-ethyl 5-(oxiran-2-ylmethoxy)-3-(p-tolyl)pent-3-enoate 4q

Colorless oil. $^1\text{H NMR}$ (400 MHz, CDCl_3): δ = 1.19 (t, J = 7.2 Hz, 3H), 2.33 (s, 3H), 2.63 (dd, J_1 = 2.8 Hz, J_2 = 4.8 Hz, 1H), 2.81 (t, J = 3.6 Hz, 1H), 3.17-3.19 (m, 1H), 3.44 (dd, J_1 = 6.0 Hz, J_2 = 11.2 Hz, 1H), 3.53 (s, 2H), 3.79 (dd, J_1 = 2.8 Hz, J_2 = 11.2 Hz, 1H), 4.10 (q, J = 7.2 Hz, 2H), 4.27-4.30 (m, 2H), 6.08 (t, J = 6.4 Hz, 1H), 7.13 (d, J = 8.0 Hz, 2H), 7.30 (d, J = 8.4 Hz, 2H); $^{13}\text{C NMR}$ (100 MHz, CDCl_3): δ = 14.1, 21.0, 36.4, 44.2, 50.8, 60.8, 68.1, 70.9, 125.9, 127.1, 129.0, 135.7, 137.3, 138.4, 170.8. HRMS (ESI-TOF). Calcd for $\text{C}_{17}\text{H}_{22}\text{NaO}_4$, $[\text{M}+\text{Na}]^+$ m/z 313.1416, Found 313.1418.



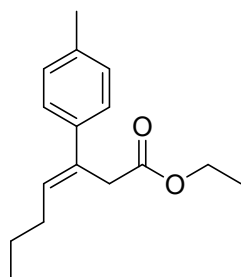
(E)-ethyl 5-(2-hydroxyethoxy)-3-(p-tolyl)pent-3-enoate 4r

Colorless oil. $^1\text{H NMR}$ (400 MHz, CDCl_3): δ = 1.19 (t, J = 7.2 Hz, 3H), 2.27 (s, 1H), 2.34 (s, 3H), 3.53 (s, 2H), 3.62 (t, J = 4.4 Hz, 2H), 3.76 (t, J = 4.4 Hz, 2H), 4.10 (q, J = 7.2 Hz, 2H), 4.26 (d, J = 6.4 Hz, 2H), 6.09 (t, J = 6.4 Hz, 1H), 7.13 (d, J = 8.0 Hz, 2H), 7.30 (d, J = 8.4 Hz, 2H); $^{13}\text{C NMR}$ (100 MHz, CDCl_3): δ = 14.1, 21.1, 36.5, 60.9, 61.9, 68.0, 71.6, 125.9, 127.1, 129.1, 136.0, 137.4, 138.4, 171.0. HRMS (ESI-TOF). Calcd for $\text{C}_{16}\text{H}_{23}\text{O}_4$, $[\text{M}+\text{H}]^+$ m/z 279.1596, Found 279.1589.



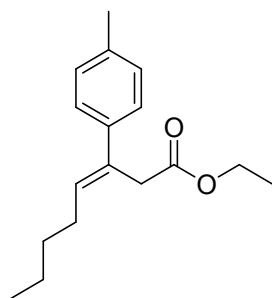
(E)-ethyl 4-cyclopropyl-3-(p-tolyl)but-3-enoate 4s

Colorless oil. $^1\text{H NMR}$ (400 MHz, CDCl_3): δ = 0.48-0.51 (m, 2H), 0.85-0.87 (m, 2H), 1.21 (t, J = 7.2 Hz, 3H), 1.60-1.67 (m, 1H), 2.32 (s, 3H), 3.62 (s, 2H), 4.13 (q, J = 7.2 Hz, 2H), 5.32 (d, J = 9.6 Hz, 1H), 7.10 (d, J = 8.0 Hz, 2H), 7.25 (d, J = 8.4 Hz, 2H); $^{13}\text{C NMR}$ (100 MHz, CDCl_3): δ = 7.5, 11.3, 14.1, 21.0, 36.4, 60.6, 125.4, 128.9, 130.7, 135.7, 136.2, 139.2, 171.7. HRMS (ESI-TOF). Calcd for $\text{C}_{16}\text{H}_{21}\text{O}_2$, $[\text{M}+\text{H}]^+$ m/z 245.1542, Found 245.1538.



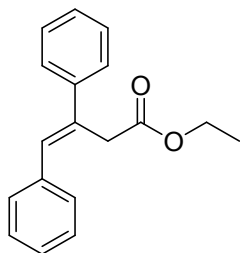
(E)-ethyl 3-(p-tolyl)hept-3-enoate 4t

Colorless oil. $^1\text{H NMR}$ (400 MHz, CDCl_3): δ = 0.96 (t, J = 7.2 Hz, 3H), 1.19 (t, J = 7.2 Hz, 3H), 1.47-1.52 (m, 2H), 2.19 (q, J = 7.2 Hz, 2H), 2.32 (s, 3H), 3.49 (s, 2H), 4.09 (q, J = 7.2 Hz, 2H), 5.91 (t, J = 7.2 Hz, 1H), 7.11 (d, J = 8.0 Hz, 2H), 7.27 (d, J = 8.0 Hz, 2H); $^{13}\text{C NMR}$ (100 MHz, CDCl_3): δ = 13.9, 14.1, 21.0, 22.7, 31.0, 36.1, 60.6, 125.8, 128.9, 131.8, 132.3, 136.4, 139.6, 171.5. HRMS (ESI-TOF). Calcd for $\text{C}_{16}\text{H}_{23}\text{O}_2$, $[\text{M}+\text{H}]^+$ m/z 247.1698, Found 247.1690.



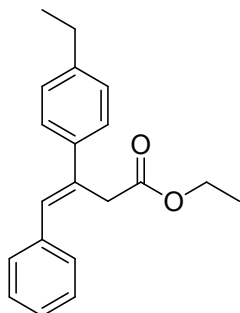
(E)-ethyl 3-(p-tolyl)oct-3-enoate 4u

Colorless oil. ^1H NMR (400 MHz, CDCl_3): δ = 0.92 (t, J = 7.2 Hz, 3H), 1.18 (t, J = 7.2 Hz, 3H), 1.35-1.47 (m, 4H), 2.21 (q, J = 7.2 Hz, 2H), 2.32 (s, 3H), 3.49 (s, 2H), 4.09 (q, J = 7.2 Hz, 2H), 5.91 (t, J = 7.2 Hz, 1H), 7.11 (d, J = 8.0 Hz, 2H), 7.26 (d, J = 8.0 Hz, 2H); ^{13}C NMR (100 MHz, CDCl_3): δ = 14.0, 14.1, 21.0, 22.4, 28.7, 31.6, 36.0, 60.6, 125.8, 128.9, 132.0, 132.0, 136.4, 139.5, 171.6. HRMS (ESI-TOF). Calcd for $\text{C}_{17}\text{H}_{25}\text{O}_2$, $[\text{M}+\text{H}]^+$ m/z 261.1855, Found 261.1846.



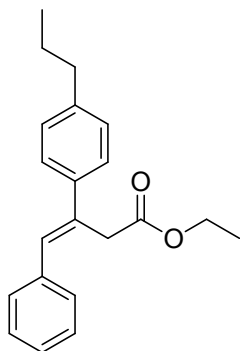
(E)-ethyl 3-(4-ethylphenyl)-4-phenylbut-3-enoate 5a

Colorless oil. ^1H NMR (400 MHz, CDCl_3): δ = 1.17 (t, J = 7.2 Hz, 3H), 3.72 (s, 2H), 4.12 (q, J = 7.2 Hz, 2H), 7.04 (s, 1H), 7.28-7.40 (m, 8H), 7.51-7.53 (m, 2H); ^{13}C NMR (100 MHz, CDCl_3): δ = 14.0, 36.7, 60.7, 126.3, 127.2, 127.5, 128.4, 128.4, 128.7, 131.2, 134.7, 137.4, 141.7, 171.5. HRMS (ESI-TOF). Calcd for $\text{C}_{18}\text{H}_{19}\text{O}_2$, $[\text{M}+\text{H}]^+$ m/z 267.1385, Found 267.1378.



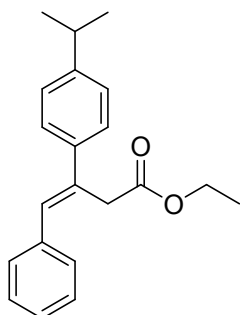
(E)-ethyl 3-(4-ethylphenyl)-4-phenylbut-3-enoate 5b

Colorless oil. ^1H NMR (400 MHz, CDCl_3): δ = 1.17 (t, J = 7.2 Hz, 3H), 1.26 (t, J = 7.6 Hz, 3H), 2.67 (q, J = 7.6 Hz, 2H), 3.70 (s, 2H), 4.11 (q, J = 7.2 Hz, 2H), 7.02 (s, 1H), 7.20 (d, J = 8.4 Hz, 2H), 7.27-7.39 (m, 5H), 7.43 (d, J = 8.4 Hz, 2H); ^{13}C NMR (100 MHz, CDCl_3): δ = 14.1, 15.5, 28.5, 36.7, 60.7, 126.1, 127.1, 127.9, 128.4, 128.7, 130.5, 134.5, 137.6, 139.0, 143.7, 171.6. HRMS (ESI-TOF). Calcd for $\text{C}_{20}\text{H}_{23}\text{O}_2$, $[\text{M}+\text{H}]^+$ m/z 295.1698, Found 295.1701.



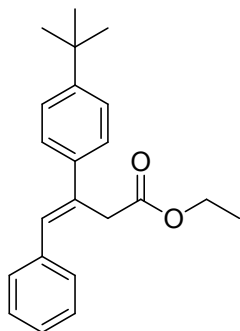
(E)-ethyl 4-phenyl-3-(4-propylphenyl)but-3-enoate 5c

Colorless oil. ^1H NMR (400 MHz, CDCl_3): δ = 0.96 (t, J = 7.2 Hz, 3H), 1.16 (t, J = 7.2 Hz, 3H), 1.63-1.69 (m, 2H), 2.60 (t, J = 7.6 Hz, 2H), 3.70 (s, 2H), 4.11 (q, J = 7.2 Hz, 2H), 7.03 (s, 1H), 7.18 (d, J = 8.0 Hz, 2H), 7.27-7.39 (m, 5H), 7.42 (d, J = 8.4 Hz, 2H); ^{13}C NMR (100 MHz, CDCl_3): δ = 13.8, 14.1, 24.5, 36.6, 37.7, 60.7, 126.0, 127.0, 128.4, 128.5, 128.7, 130.4, 134.5, 137.6, 139.0, 142.2, 171.6. HRMS (ESI-TOF). Calcd for $\text{C}_{21}\text{H}_{25}\text{O}_2$, $[\text{M}+\text{H}]^+$ m/z 309.1855, Found 309.1847.



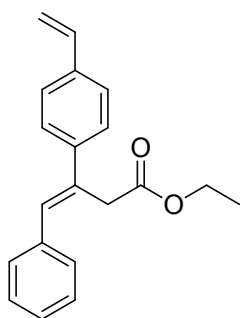
(E)-ethyl 3-(4-isopropylphenyl)-4-phenylbut-3-enoate 5d

Colorless oil. ^1H NMR (400 MHz, CDCl_3): δ = 1.15 (t, J = 7.2 Hz, 3H), 1.26 (d, J = 7.2 Hz, 6H), 2.85-2.95 (m, 1H), 3.69 (s, 2H), 4.10 (q, J = 7.2 Hz, 2H), 7.02 (s, 1H), 7.22 (d, J = 8.4 Hz, 2H), 7.25-7.38 (m, 5H), 7.43 (d, J = 8.0 Hz, 2H); ^{13}C NMR (100 MHz, CDCl_3): δ = 14.0, 23.9, 33.7, 36.6, 60.7, 126.1, 126.5, 127.0, 128.3, 128.7, 130.4, 134.4, 137.5, 139.0, 148.3, 171.6. HRMS (ESI-TOF). Calcd for $\text{C}_{21}\text{H}_{25}\text{O}_2$, $[\text{M}+\text{H}]^+$ m/z 309.1855, Found 309.1846.



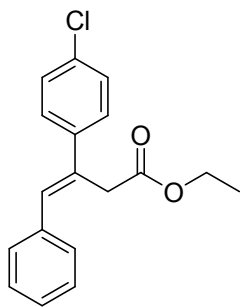
(E)-ethyl 3-(4-(*tert*-butyl)phenyl)-4-phenylbut-3-enoate 5e

Colorless oil. $^1\text{H NMR}$ (400 MHz, CDCl_3): δ = 1.17 (t, J = 7.2 Hz, 3H), 1.34 (s, 9H), 3.70 (s, 2H), 4.12 (q, J = 7.2 Hz, 2H), 7.04 (s, 1H), 7.27-7.40 (m, 7H), 7.45 (d, J = 8.8 Hz, 2H); $^{13}\text{C NMR}$ (100 MHz, CDCl_3): δ = 14.1, 31.3, 34.5, 36.6, 60.7, 125.4, 125.8, 127.1, 128.4, 128.8, 130.5, 134.4, 137.6, 138.7, 150.6, 171.6. HRMS (ESI-TOF). Calcd for $\text{C}_{22}\text{H}_{27}\text{O}_2$, $[\text{M}+\text{H}]^+$ m/z 323.2011, Found 323.1996.



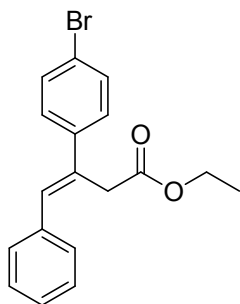
(E)-ethyl 4-phenyl-3-(4-vinylphenyl)but-3-enoate 5f

Colorless oil. $^1\text{H NMR}$ (400 MHz, CDCl_3): δ = 1.18 (t, J = 7.2 Hz, 3H), 3.72 (s, 2H), 4.12 (q, J = 7.2 Hz, 2H), 5.27 (d, J = 10.8 Hz, 1H), 5.78 (d, J = 17.6 Hz, 1H), 6.74 (dd, J_1 = 10.8 Hz, J_2 = 17.6 Hz, 1H), 7.07 (s, 1H), 7.29-7.32 (m, 1H), 7.37-7.43 (m, 6H), 7.48 (d, J = 8.4 Hz, 2H); $^{13}\text{C NMR}$ (100 MHz, CDCl_3): δ = 14.1, 36.5, 60.8, 113.8, 126.3, 127.2, 128.4, 128.8, 131.0, 134.2, 136.4, 136.9, 137.4, 141.0, 171.5. HRMS (ESI-TOF). Calcd for $\text{C}_{20}\text{H}_{21}\text{O}_2$, $[\text{M}+\text{H}]^+$ m/z 293.1542, Found 293.1530.



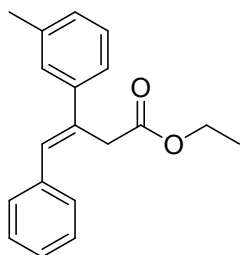
(E)-ethyl 3-(4-chlorophenyl)-4-phenylbut-3-enoate 5g

Colorless oil. ^1H NMR (400 MHz, CDCl_3): δ = 1.17 (t, J = 7.2 Hz, 3H), 3.68 (s, 2H), 4.11 (q, J = 7.2 Hz, 2H), 7.00 (s, 1H), 7.30-7.39 (m, 7H), 7.43 (d, J = 8.4 Hz, 2H); ^{13}C NMR (100 MHz, CDCl_3): δ = 14.1, 36.6, 60.9, 127.4, 127.6, 128.5, 128.6, 128.7, 131.7, 133.4, 133.6, 137.1, 140.2, 171.3. HRMS (ESI-TOF). Calcd for $\text{C}_{18}\text{H}_{18}\text{ClO}_2$, $[\text{M}+\text{H}]^+$ m/z 301.0995, Found 301.0992.



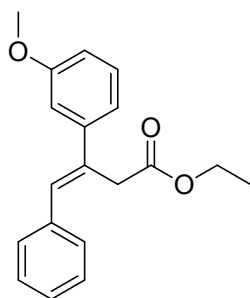
(E)-ethyl 3-(4-bromophenyl)-4-phenylbut-3-enoate 5h

Colorless oil. ^1H NMR (400 MHz, CDCl_3): δ = 1.18 (t, J = 7.2 Hz, 3H), 3.68 (s, 2H), 4.11 (q, J = 7.2 Hz, 2H), 7.01 (s, 1H), 7.29-7.41 (m, 7H), 7.50 (d, J = 8.8 Hz, 2H); ^{13}C NMR (100 MHz, CDCl_3): δ = 14.1, 36.5, 60.9, 121.5, 127.4, 127.9, 128.4, 128.7, 131.5, 131.7, 133.6, 137.0, 140.6, 171.2. HRMS (ESI-TOF). Calcd for $\text{C}_{18}\text{H}_{18}\text{BrO}_2$, $[\text{M}+\text{H}]^+$ m/z 345.0490, 347.0470, Found 345.0486, 347.0462.



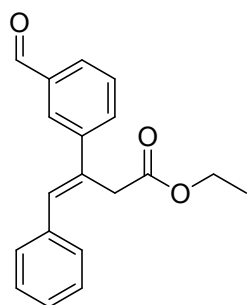
(E)-ethyl 4-phenyl-3-(*m*-tolyl)but-3-enoate 5i

Colorless oil. ^1H NMR (400 MHz, CDCl_3): δ = 1.17 (t, J = 7.2 Hz, 3H), 2.39 (s, 3H), 3.70 (s, 2H), 4.11 (q, J = 7.2 Hz, 2H), 7.02 (s, 1H), 7.12 (d, J = 7.2 Hz, 1H), 7.24-7.32 (m, 4H), 7.38 (d, J = 4.0 Hz, 4H); ^{13}C NMR (100 MHz, CDCl_3): δ = 14.1, 21.5, 36.7, 60.7, 123.3, 127.0, 127.1, 128.3, 128.3, 128.4, 128.7, 131.0, 134.8, 137.5, 138.0, 141.7, 171.6. HRMS (ESI-TOF). Calcd for $\text{C}_{19}\text{H}_{21}\text{O}_2$, $[\text{M}+\text{H}]^+$ m/z 281.1542, Found 281.1540.



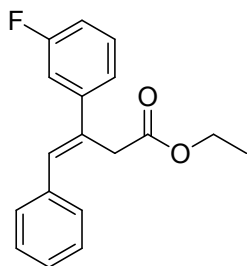
(E)-ethyl 3-(3-methoxyphenyl)-4-phenylbut-3-enoate 5j

Colorless oil. ^1H NMR (400 MHz, CDCl_3): δ = 1.17 (t, J = 7.2 Hz, 3H), 3.69 (s, 2H), 3.84 (s, 3H), 4.11 (q, J = 7.2 Hz, 2H), 6.84-6.87 (m, 1H), 7.04-7.10 (m, 3H), 7.26-7.31 (m, 2H), 7.38 (d, J = 4.4 Hz, 4H); ^{13}C NMR (100 MHz, CDCl_3): δ = 14.1, 36.8, 55.3, 60.8, 112.1, 113.0, 118.7, 127.2, 128.4, 128.7, 129.4, 131.3, 134.6, 137.3, 143.3, 159.7, 171.5. HRMS (ESI-TOF). Calcd for $\text{C}_{19}\text{H}_{21}\text{O}_3$, $[\text{M}+\text{H}]^+$ m/z 297.1491, Found 297.1490.



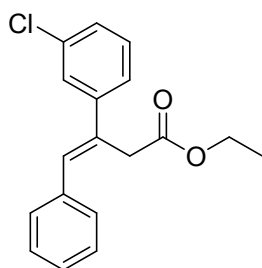
(E)-ethyl 3-(3-formylphenyl)-4-phenylbut-3-enoate 5k

Colorless oil. ^1H NMR (400 MHz, CDCl_3): δ = 1.16 (t, J = 7.2 Hz, 3H), 3.74 (s, 2H), 4.11 (q, J = 7.2 Hz, 2H), 7.09 (s, 1H), 7.30-7.40 (m, 5H), 7.55 (t, J = 7.6 Hz, 1H), 7.77 (d, J = 8.0 Hz, 1H), 7.82 (d, J = 7.2 Hz, 1H), 8.01 (s, 1H), 10.06 (s, 1H); ^{13}C NMR (100 MHz, CDCl_3): δ = 14.1, 36.6, 60.9, 127.5, 127.6, 128.5, 128.7, 128.8, 129.2, 132.3, 132.7, 133.6, 136.6, 136.9, 142.8, 171.1, 192.2. HRMS (ESI-TOF). Calcd for $\text{C}_{19}\text{H}_{19}\text{O}_3$, $[\text{M}+\text{H}]^+$ m/z 295.1334, Found 295.1326.



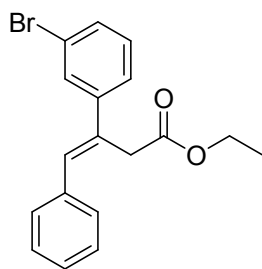
(E)-ethyl 3-(3-fluorophenyl)-4-phenylbut-3-enoate 5l

Colorless oil. ^1H NMR (400 MHz, CDCl_3): δ = 1.17 (t, J = 7.2 Hz, 3H), 3.68 (s, 2H), 4.12 (q, J = 7.2 Hz, 2H), 6.97-7.02 (m, 1H), 7.04 (s, 1H), 7.19-7.22 (m, 1H), 7.28-7.39 (m, 7H); ^{13}C NMR (100 MHz, CDCl_3): δ = 14.1, 36.6, 60.9, 113.3 (d, J = 22.0 Hz), 114.4 (d, J = 21.0 Hz), 121.9 (d, J = 3.0 Hz), 127.5, 128.5, 128.8, 129.9 (d, J = 8.0 Hz), 132.1, 133.7 (d, J = 2.0 Hz), 137.0, 144.1 (d, J = 7.0 Hz), 163.0 (d, J = 244.0 Hz), 171.2. HRMS (ESI-TOF). Calcd for $\text{C}_{18}\text{H}_{18}\text{FO}_2$, $[\text{M}+\text{H}]^+$ m/z 285.1291, Found 285.1295.



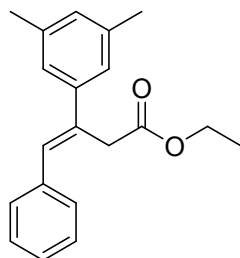
(E)-ethyl 3-(3-chlorophenyl)-4-phenylbut-3-enoate 5m

Colorless oil. ^1H NMR (400 MHz, CDCl_3): δ = 1.18 (t, J = 7.2 Hz, 3H), 3.68 (s, 2H), 4.12 (q, J = 7.2 Hz, 2H), 7.02 (s, 1H), 7.27-7.41 (m, 8H), 7.49 (s, 1H); ^{13}C NMR (100 MHz, CDCl_3): δ = 14.1, 36.6, 60.9, 124.4, 126.5, 127.5, 127.6, 128.5, 128.7, 129.7, 132.3, 133.5, 134.4, 136.9, 143.6, 171.2. HRMS (ESI-TOF). Calcd for $\text{C}_{18}\text{H}_{18}\text{ClO}_2$, $[\text{M}+\text{H}]^+$ m/z 301.0995, Found 301.0992.



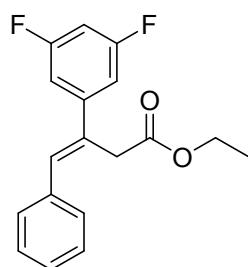
(E)-ethyl 3-(3-bromophenyl)-4-phenylbut-3-enoate 5n

Colorless oil. ^1H NMR (400 MHz, CDCl_3): δ = 1.18 (t, J = 7.2 Hz, 3H), 3.67 (s, 2H), 4.12 (q, J = 7.2 Hz, 2H), 7.01 (s, 1H), 7.22-7.32 (m, 2H), 7.36-7.44 (m, 6H), 7.65 (t, J = 1.6 Hz, 1H); ^{13}C NMR (100 MHz, CDCl_3): δ = 14.1, 36.6, 60.9, 122.6, 124.9, 127.5, 128.5, 128.7, 129.5, 129.9, 130.5, 132.4, 133.5, 136.9, 144.0, 171.1. HRMS (ESI-TOF). Calcd for $\text{C}_{18}\text{H}_{18}\text{BrO}_2$, $[\text{M}+\text{H}]^+$ m/z 345.0490, 347.0470, Found 345.0479, 347.0460.



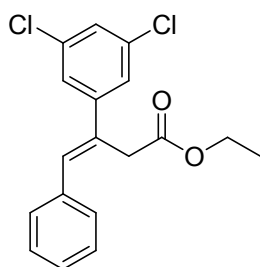
(E)-ethyl 3-(3,5-dimethylphenyl)-4-phenylbut-3-enoate 5o

Colorless oil. ^1H NMR (400 MHz, CDCl_3): δ = 1.19 (t, J = 7.2 Hz, 3H), 2.35 (s, 6H), 3.70 (s, 2H), 4.13 (q, J = 7.2 Hz, 2H), 6.96 (s, 1H), 7.02 (s, 1H), 7.13 (s, 2H), 7.28-7.31 (s, 1H), 7.39 (d, J = 4.4 Hz, 4H); ^{13}C NMR (100 MHz, CDCl_3): δ = 14.1, 21.4, 36.7, 60.7, 124.1, 127.0, 128.4, 128.7, 129.3, 130.8, 134.8, 137.6, 137.8, 141.7, 171.6. HRMS (ESI-TOF). Calcd for $\text{C}_{20}\text{H}_{23}\text{O}_2$, $[\text{M}+\text{H}]^+$ m/z 295.1698, Found 295.1692.



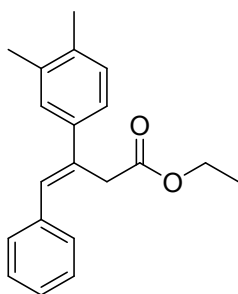
(E)-ethyl 3-(3,5-difluorophenyl)-4-phenylbut-3-enoate 5p

Colorless oil. ^1H NMR (400 MHz, CDCl_3): δ = 1.20 (t, J = 7.2 Hz, 3H), 3.65 (s, 2H), 4.14 (q, J = 7.2 Hz, 2H), 6.73-6.77 (s, 1H), 7.01-7.06 (s, 3H), 7.30-7.42 (s, 5H); ^{13}C NMR (100 MHz, CDCl_3): δ = 14.1, 36.5, 61.0, 102.8 (t, J = 26.0 Hz), 109.2 (dd, J_1 = 8.0 Hz, J_2 = 19.0 Hz), 127.7, 128.5, 128.7, 132.8 (t, J = 3.0 Hz), 133.0, 136.6, 145.3 (t, J = 10.0 Hz), 163.1 (dd, J_1 = 13.0 Hz, J_2 = 246.0 Hz), 171.0. HRMS (ESI-TOF). Calcd for $\text{C}_{18}\text{H}_{17}\text{F}_2\text{O}_2$, $[\text{M}+\text{H}]^+$ m/z 303.1197, Found 303.1191.



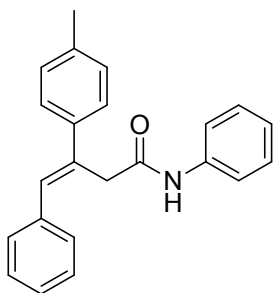
(E)-ethyl 3-(3,5-dichlorophenyl)-4-phenylbut-3-enoate 5q

Colorless oil. ^1H NMR (400 MHz, CDCl_3): δ = 1.20 (t, J = 7.2 Hz, 3H), 3.64 (s, 2H), 4.14 (q, J = 7.2 Hz, 2H), 7.02 (s, 1H), 7.29-7.41 (s, 8H); ^{13}C NMR (100 MHz, CDCl_3): δ = 14.1, 36.5, 61.0, 124.9, 127.4, 127.7, 128.5, 128.7, 132.5, 133.3, 135.0, 136.5, 144.9, 170.9. HRMS (ESI-TOF). Calcd for $\text{C}_{18}\text{H}_{17}\text{Cl}_2\text{O}_2$, $[\text{M}+\text{H}]^+$ m/z 335.0606, Found 335.0601.



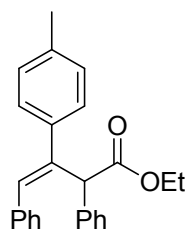
(E)-ethyl 3-(3,4-dimethylphenyl)-4-phenylbut-3-enoate 5r

Colorless oil. ^1H NMR (400 MHz, CDCl_3): δ = 1.19 (t, J = 7.2 Hz, 3H), 2.28 (s, 3H), 2.30 (s, 3H), 3.69 (s, 2H), 4.12 (q, J = 7.2 Hz, 2H), 7.00 (s, 1H), 7.13 (d, J = 8.0 Hz, 1H), 7.22-7.29 (s, 3H), 7.38 (d, J = 4.8 Hz, 4H); ^{13}C NMR (100 MHz, CDCl_3): δ = 14.1, 19.4, 19.9, 36.6, 60.7, 123.6, 127.0, 127.5, 128.4, 128.7, 129.7, 130.3, 134.6, 136.1, 136.5, 137.6, 139.3, 171.6. HRMS (ESI-TOF). Calcd for $\text{C}_{20}\text{H}_{23}\text{O}_2$, $[\text{M}+\text{H}]^+$ m/z 295.1698, Found 295.1692.



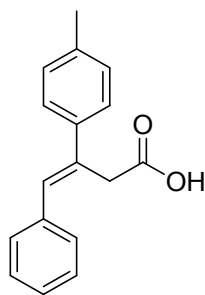
(E)-N,4-diphenyl-3-(p-tolyl)but-3-enamide 5s

White solid. mp: 178-179 °C. ¹H NMR (400 MHz, CDCl₃): δ = 2.38 (s, 3H), 3.83 (s, 2H), 7.07 (t, *J* = 7.2 Hz, 1H), 7.17 (s, 1H), 7.23 (d, *J* = 8.4 Hz, 2H), 7.28-7.42 (m, 10H), 7.49 (d, *J* = 8.0 Hz, 2H); ¹³C NMR (100 MHz, CDCl₃): δ = 21.1, 40.0, 119.8, 124.4, 126.0, 127.6, 128.7, 128.8, 128.9, 129.7, 131.7, 134.6, 136.8, 137.6, 138.2, 138.4, 168.5. HRMS (ESI-TOF). Calcd for C₂₃H₂₂NO, [M+H]⁺ *m/z* 328.1701, Found 328.1697.



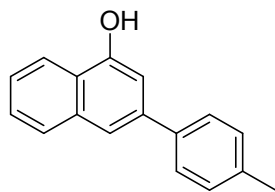
(E)-ethyl 2,4-diphenyl-3-(p-tolyl)but-3-enoate 5t

Colorless oil. ¹H NMR (400 MHz, CDCl₃): δ = 1.06 (t, *J* = 7.2 Hz, 3H), 2.31 (s, 3H), 4.03-4.09 (m, 2H), 5.35 (s, 1H), 7.01-7.06 (s, 3H), 7.20-7.36 (m, 12H); ¹³C NMR (100 MHz, CDCl₃): δ = 13.9, 21.1, 52.5, 60.9, 126.9, 127.2, 128.0, 128.1, 128.4, 128.5, 128.7, 129.3, 132.0, 137.0, 137.1, 137.2, 138.1, 138.7, 172.2. HRMS (ESI-TOF). Calcd for C₂₅H₂₅O₂, [M+H]⁺ *m/z* 357.1855, Found 357.1846.



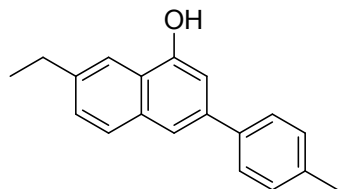
(E)-4-phenyl-3-(p-tolyl)but-3-enoic acid 6

White solid. mp: 154-155 °C. ¹H NMR (400 MHz, CDCl₃): δ = 2.39 (s, 3H), 3.75 (s, 2H), 7.06 (s, 1H), 7.20 (d, *J* = 8.0 Hz, 2H), 7.30-7.43 (m, 7H); ¹³C NMR (100 MHz, CDCl₃): δ = 21.1, 36.1, 126.0, 127.2, 128.5, 128.7, 129.3, 131.0, 133.5, 137.3, 137.6, 138.3, 177.9. HRMS (ESI-TOF). Calcd for C₁₇H₁₇O₂, [M+H]⁺ *m/z* 253.1229, Found 253.1225.



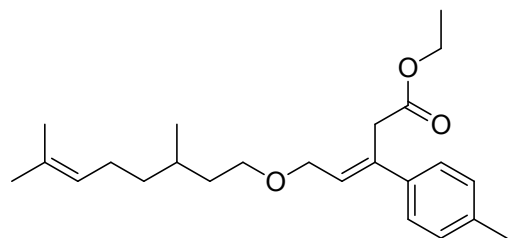
3-(*p*-tolyl)naphthalen-1-ol 7a

White solid. mp: 150-151 °C. ¹H NMR (400 MHz, CDCl₃): δ = 2.44 (s, 3H), 5.54 (s, 1H), 7.07 (s, 1H), 7.29 (d, *J* = 7.6 Hz, 2H), 7.48-7.60 (m, 4H), 7.67 (s, 1H), 7.88 (d, *J* = 7.6 Hz, 1H), 7.21 (d, *J* = 8.0 Hz, 1H); ¹³C NMR (100 MHz, CDCl₃): δ = 21.1, 108.3, 118.4, 121.4, 123.4, 125.2, 126.8, 127.1, 127.9, 129.5, 135.0, 137.2, 137.9, 138.8, 151.6. HRMS (ESI-TOF). Calcd for C₁₇H₁₅O, [M+H]⁺ *m/z* 235.1123, Found 235.1115.



7-ethyl-3-(*p*-tolyl)naphthalen-1-ol 7b

White solid. mp: 132-134 °C. ¹H NMR (400 MHz, CDCl₃): δ = 1.32 (t, *J* = 7.6 Hz, 3H), 2.39 (s, 3H), 2.81 (q, *J* = 7.6 Hz, 2H), 5.42 (s, 1H), 7.00 (s, 1H), 7.23 (d, *J* = 7.6 Hz, 2H), 7.35 (d, *J* = 8.0 Hz, 1H), 7.53 (d, *J* = 8.0 Hz, 2H), 7.58 (s, 1H), 7.75 (d, *J* = 8.4 Hz, 1H), 7.93 (s, 1H); ¹³C NMR (100 MHz, CDCl₃): δ = 15.6, 21.1, 29.2, 108.4, 118.2, 119.0, 123.5, 127.0, 128.0, 128.0, 129.5, 133.5, 137.0, 137.8, 138.1, 141.3, 151.3. HRMS (ESI-TOF). Calcd for C₁₉H₁₉O, [M+H]⁺ *m/z* 263.1436, Found 263.1428.

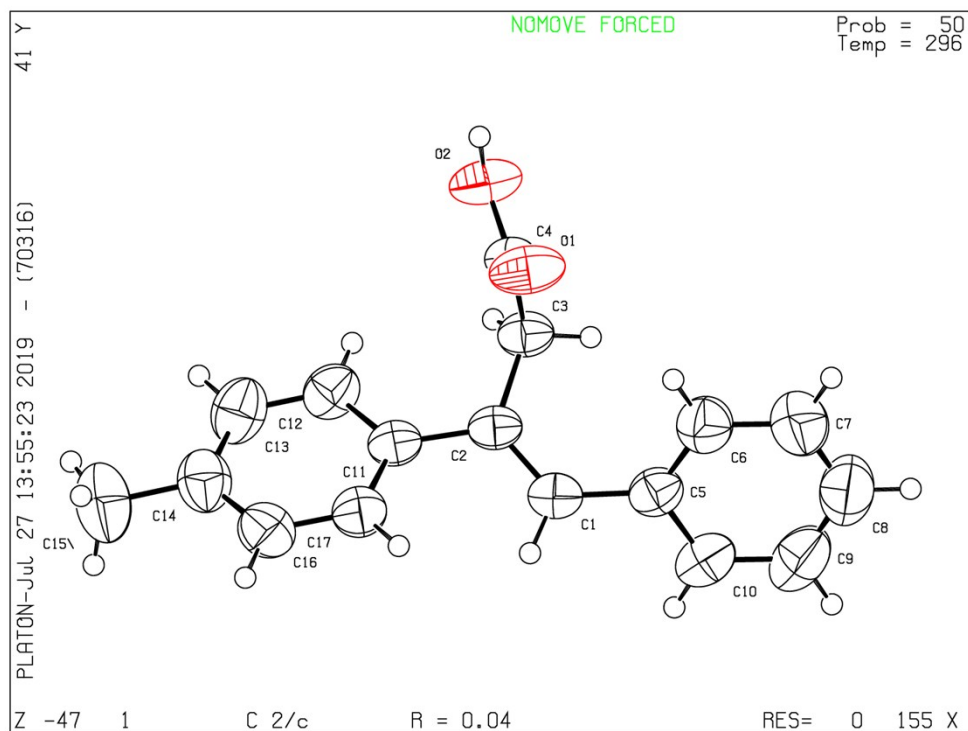


(*E*)-ethyl 5-((3,7-dimethyloct-6-en-1-yl)oxy)-3-(*p*-tolyl)pent-3-enoate 9

Colorless oil. ¹H NMR (400 MHz, CDCl₃): δ = 0.91 (d, *J* = 6.4 Hz, 3H), 1.19 (t, *J* = 7.2 Hz, 3H), 1.35-1.44 (m, 3H), 1.56-1.68 (m, 8H), 1.95-2.03 (m, 2H), 2.33 (s, 3H), 3.49-3.53 (m, 4H), 4.10 (q, *J* = 7.2 Hz, 2H), 4.20 (d, *J* = 6.4 Hz, 2H), 5.10 (t, *J* = 6.4 Hz, 1H), 6.09 (t, *J* = 6.4 Hz, 1H), 7.13 (d,

$J = 8.0$ Hz, 2H), 7.31 (d, $J = 8.0$ Hz, 2H); ^{13}C NMR (100 MHz, CDCl_3): $\delta = 14.1, 17.6, 19.5, 21.0, 25.5, 25.7, 29.6, 36.4, 36.7, 37.2, 60.8, 67.8, 69.0, 124.8, 125.9, 128.0, 129.0, 131.1, 134.9, 137.1, 138.6, 171.0$. HRMS (ESI-TOF). Calcd for $\text{C}_{24}\text{H}_{37}\text{O}_3$, $[\text{M}+\text{H}]^+$ m/z 373.2743, Found 373.2739.

5. Structure Analysis X-Ray Crystallography of 6



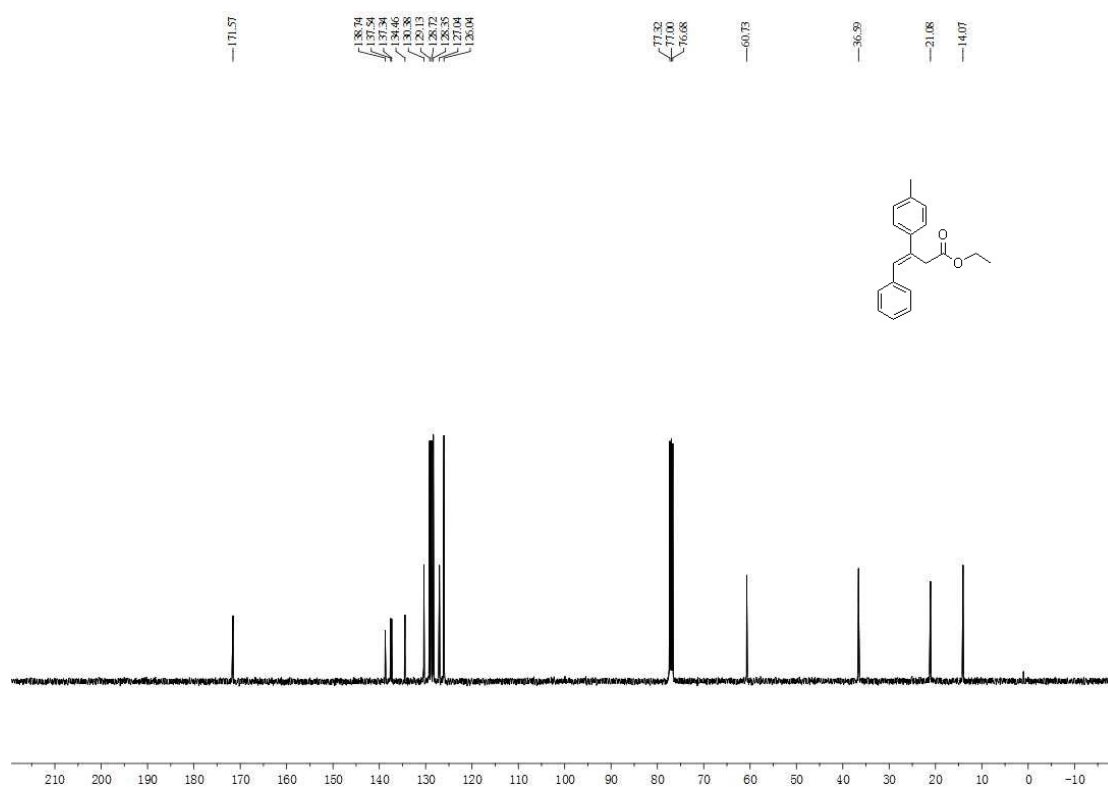
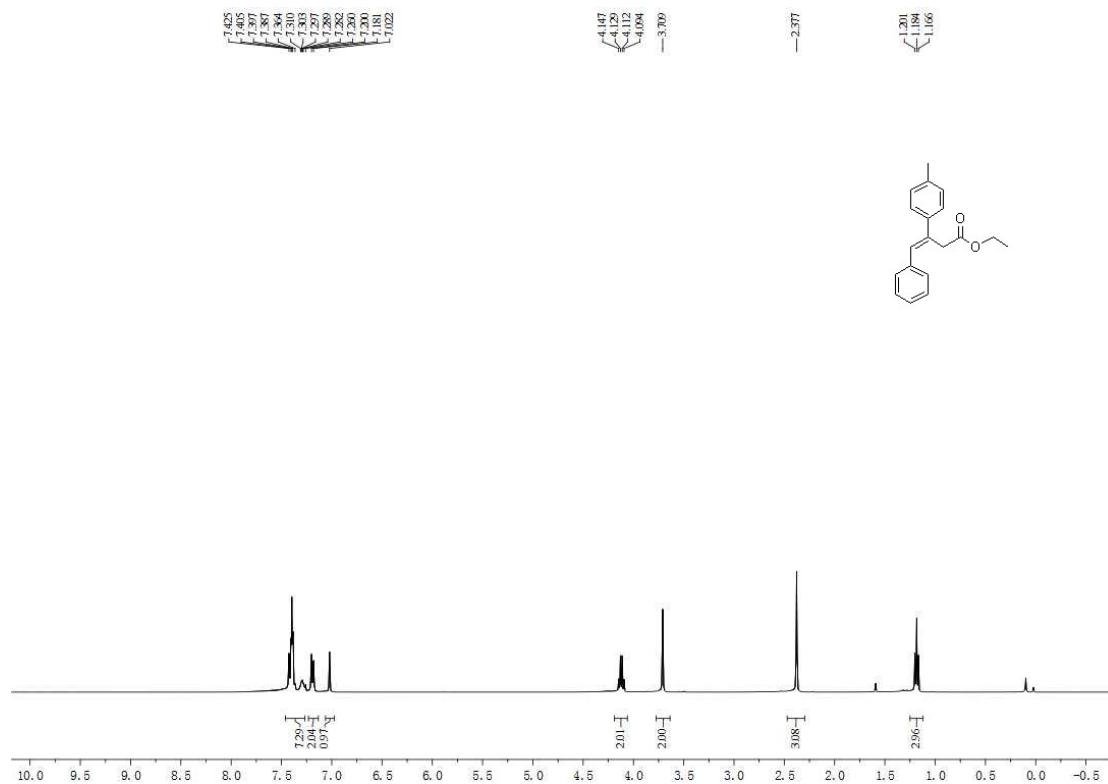
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Empirical formula	C ₁₇ H ₁₆ O ₂
Formula weight	252.30
Crystal system	Monoclinic
Space group	C2/c
<i>a</i> (Å)	18.085(4)
<i>b</i> (Å)	9.7655(19)
<i>c</i> (Å)	17.955(4)
α (°)	90
β (°)	115.852(15)
γ (°)	90
<i>V</i> (Å ³)	2853.7(11)
<i>Z</i>	8
<i>D</i> /g cm ⁻³	1.174
μ /mm ⁻¹	0.076
<i>F</i> (000)	1072.0
Reflns number	14941
<i>R</i> _{int}	0.0364
<i>R</i> _{<i>I</i>}	0.0440
GOF	1.012
<i>wR</i> ² (all data)	0.1346

6. References

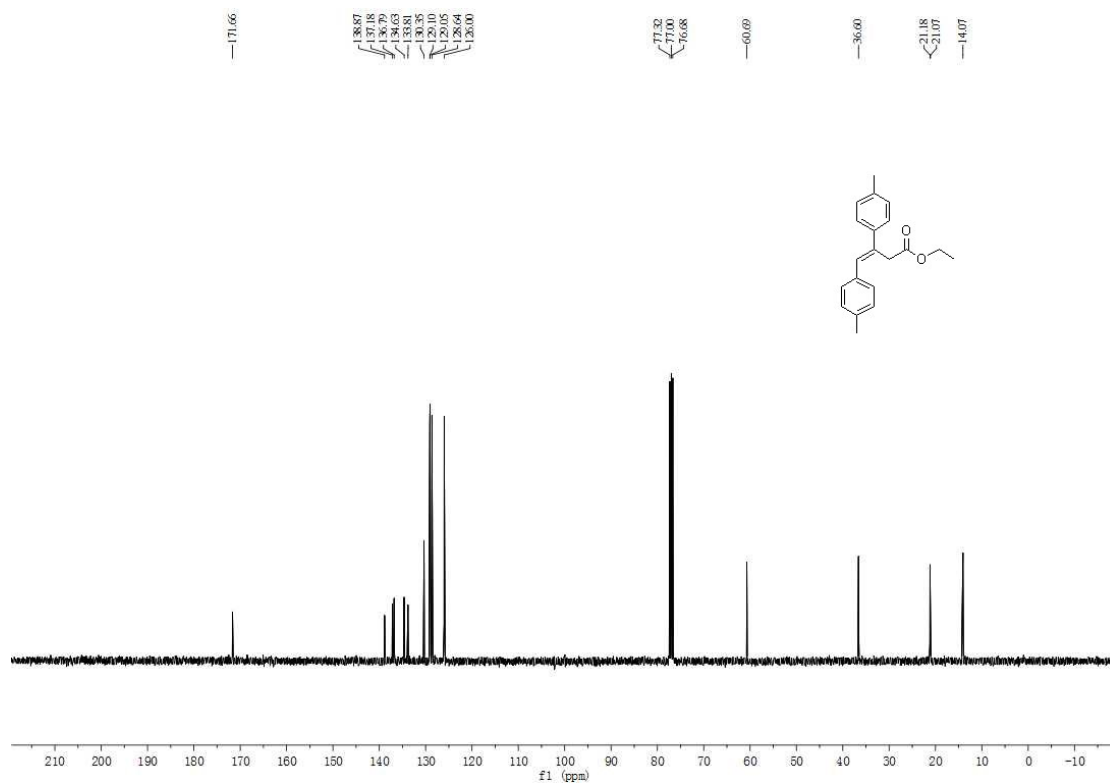
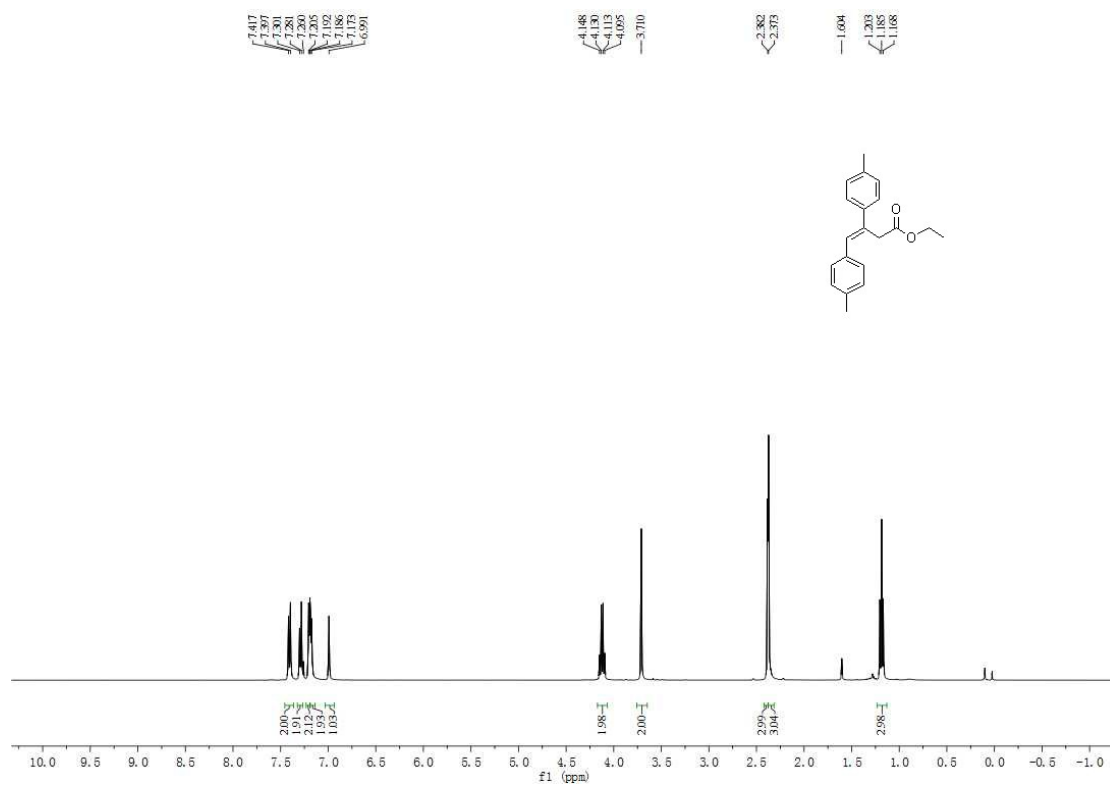
- [1] A. K. Gupta, X. Yin, M. Mukherjee, A. A. Desai, A. Mohammadlou, K. Jurewicz and W. D. Wulff, *Angew. Chem. Int. Ed.*, 2019, **58**, 3361.
- [2] X. Hong, L. Lu and Q. Shen, *Synlett*, 2019, **30**, 1602.
- [3] A. Suárez and G. C. Fu, *Angew. Chem. Int. Ed.*, 2004, **43**, 3580.
- [4] D. Mal and S. Jana, *J. Org. Chem.*, 2016, **81**, 11857.
- [5] J. L. Liang, U. Javed, S. H. Lee, J. G. Park and Y. Jahng, *Arch. Pharm. Res.*, 2014, **37**, 862.

7. ¹H and ¹³C NMR Spectra of Compounds 4-7 and 9

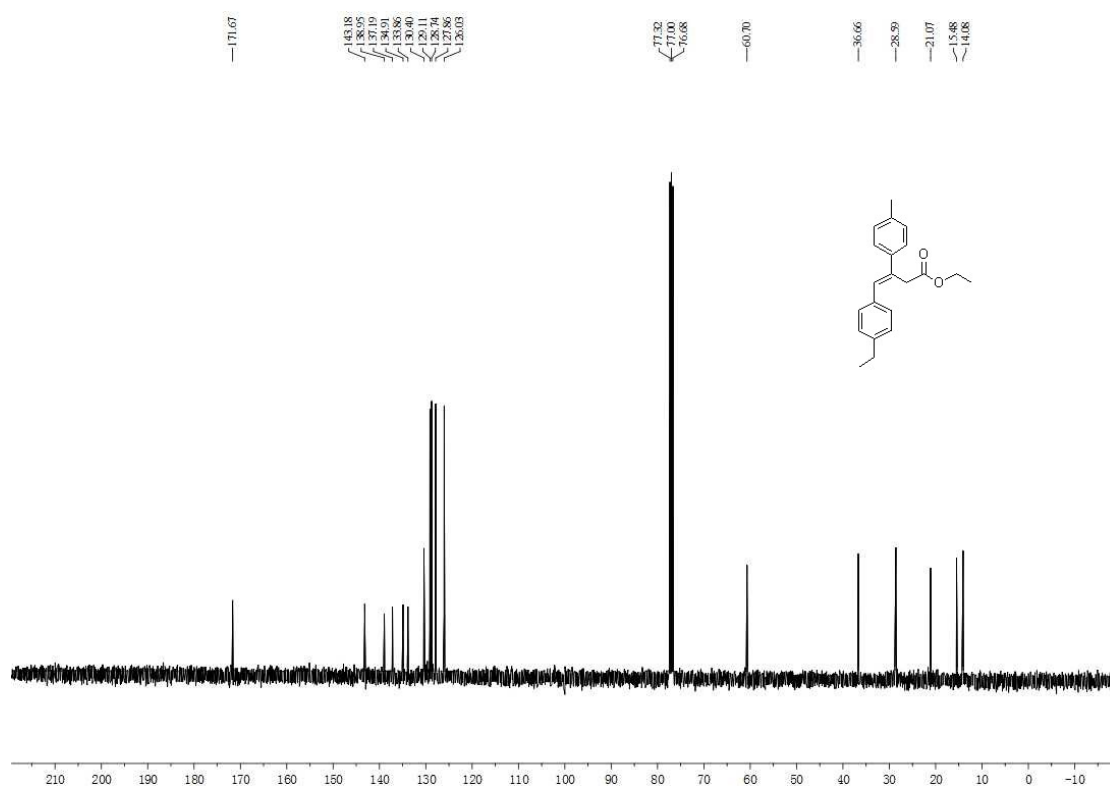
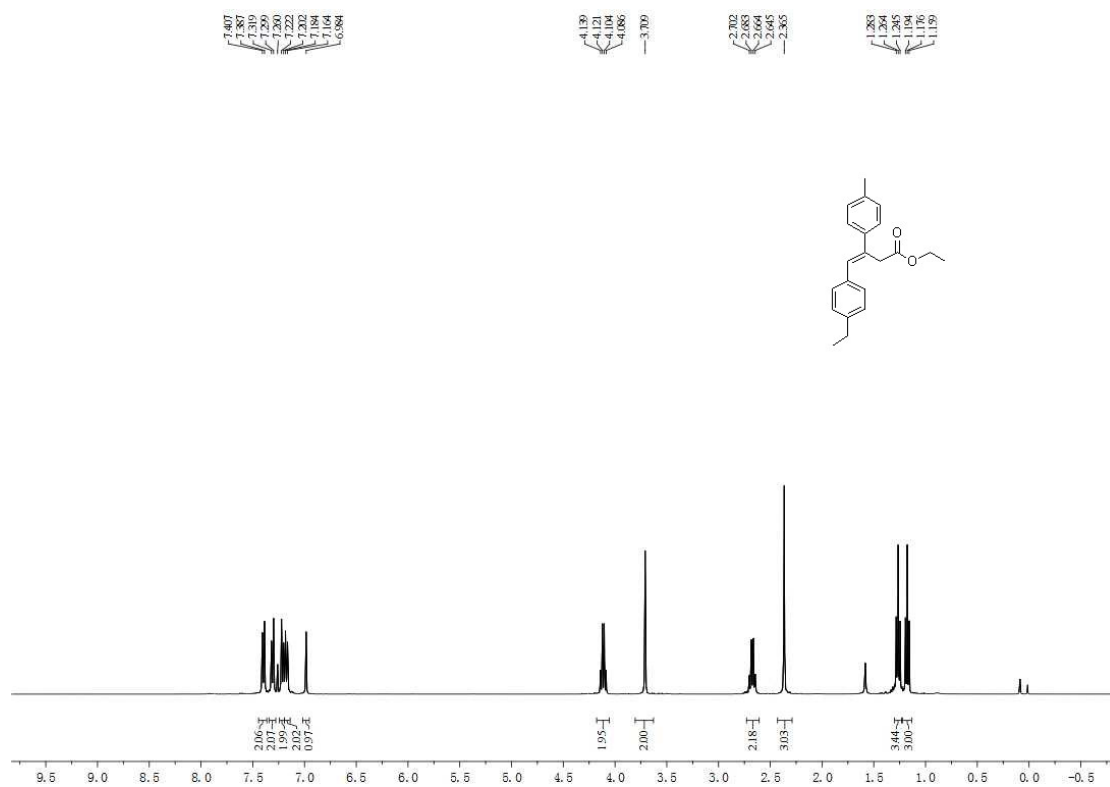
Product 4a



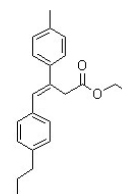
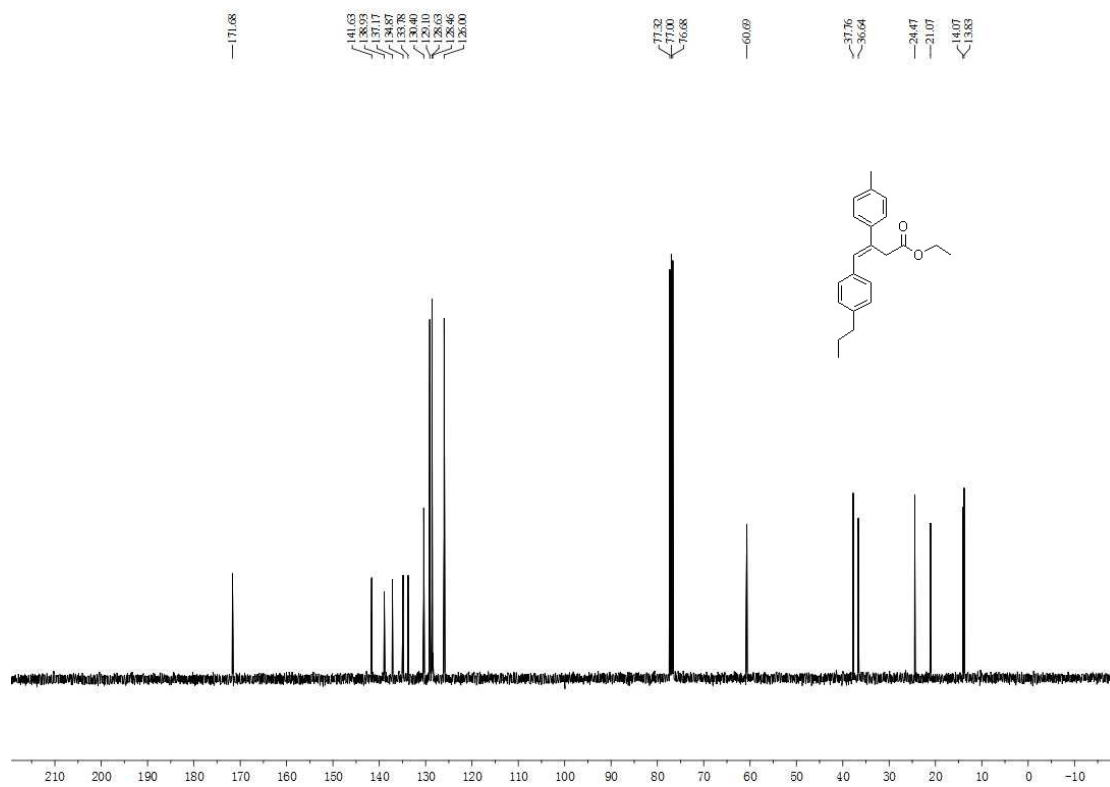
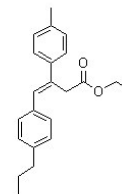
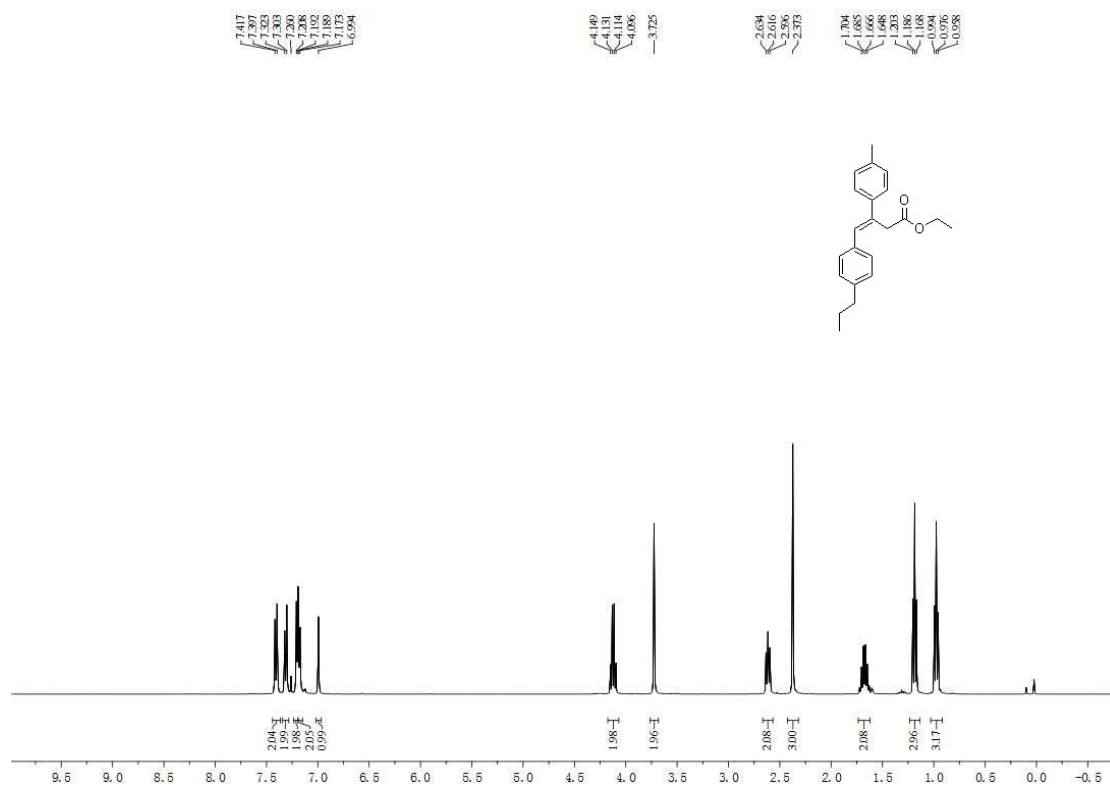
Product 4b



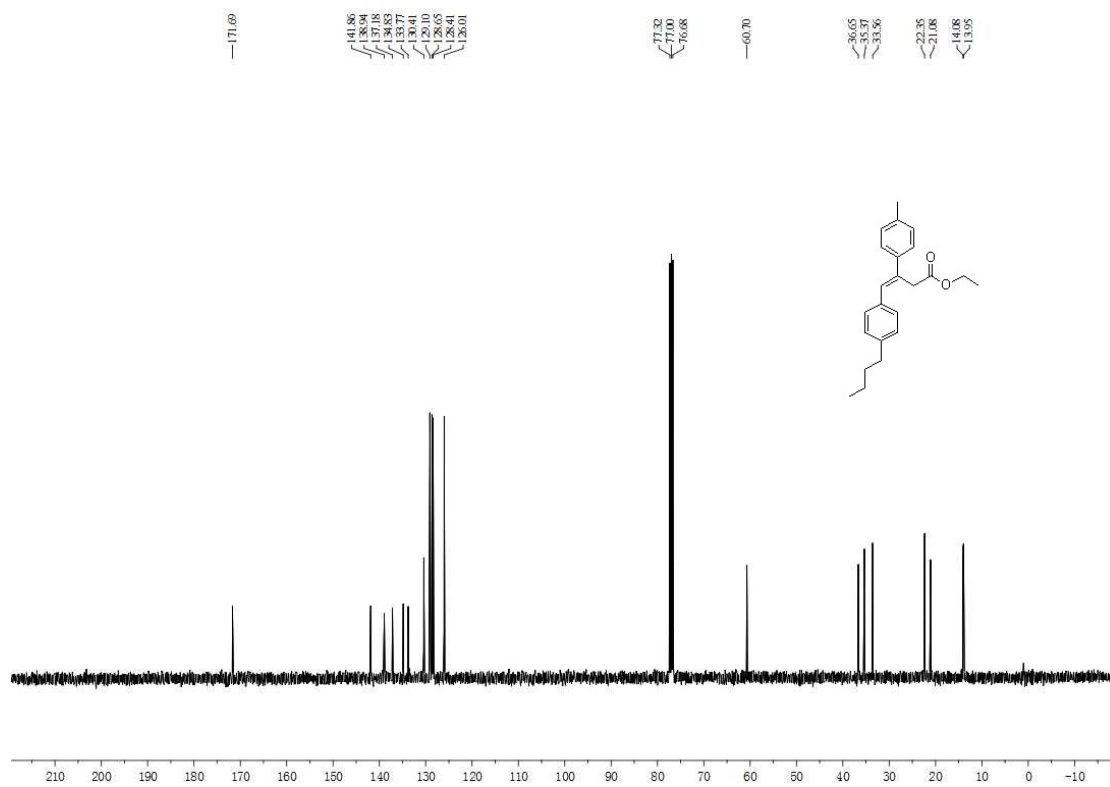
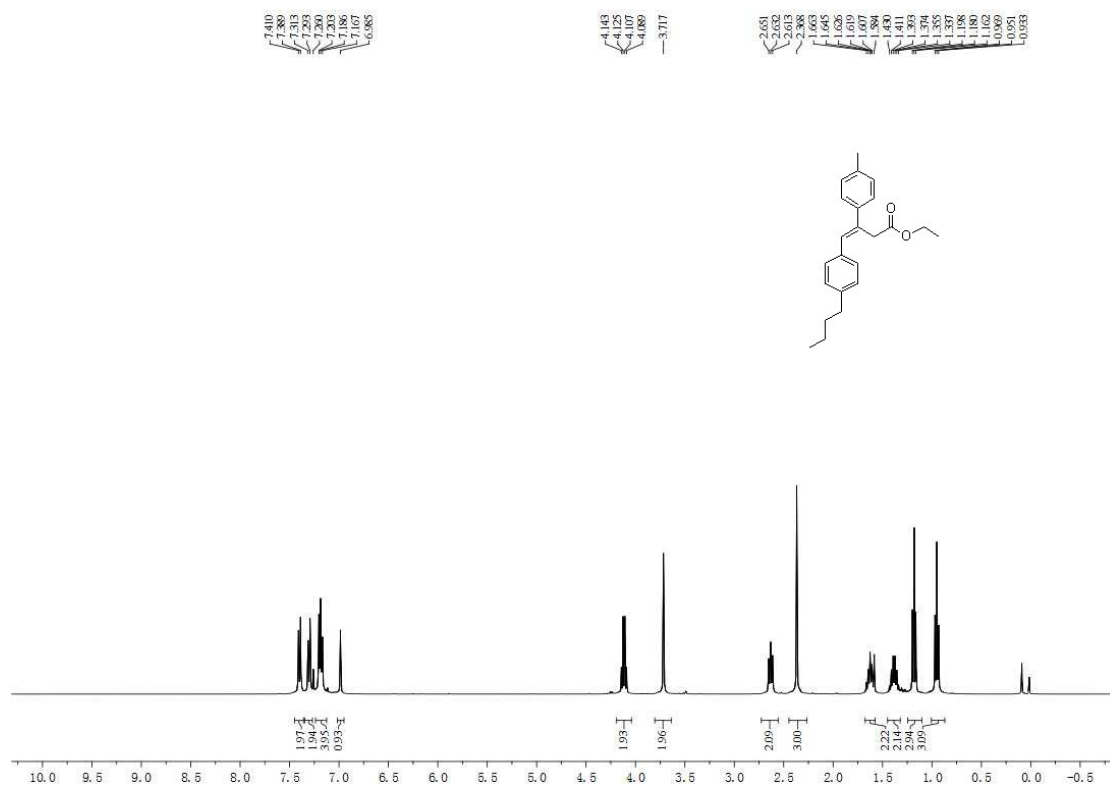
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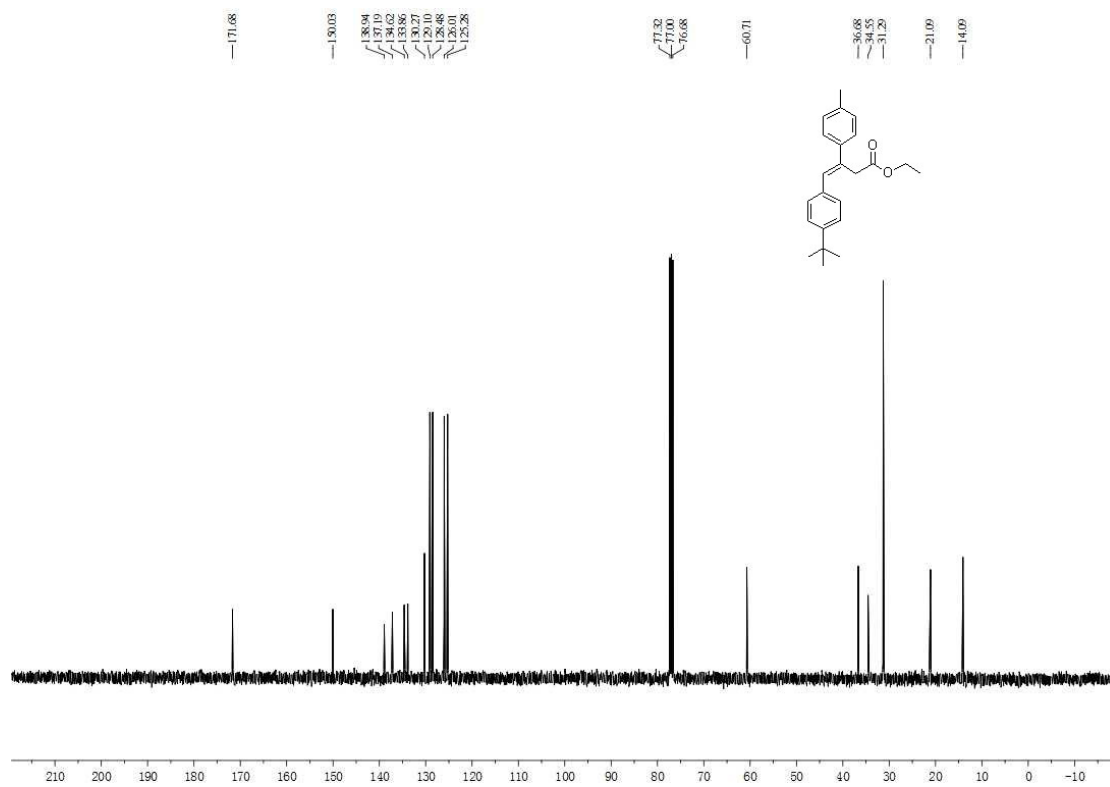
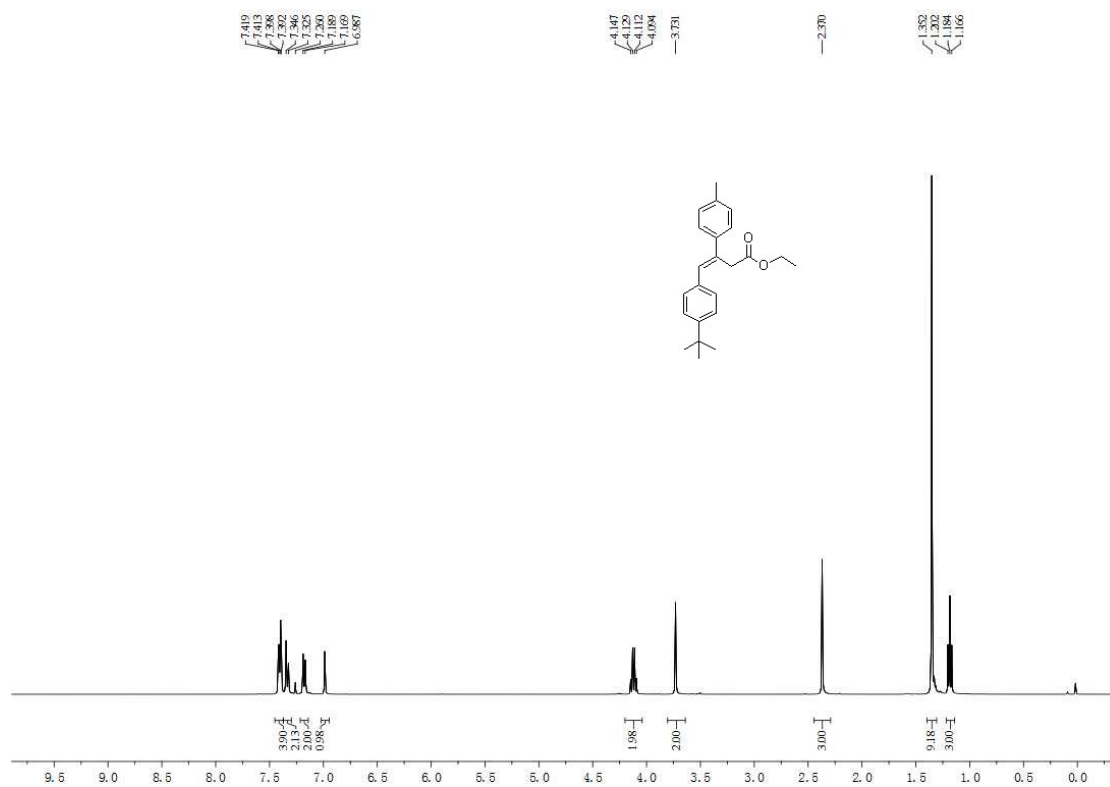
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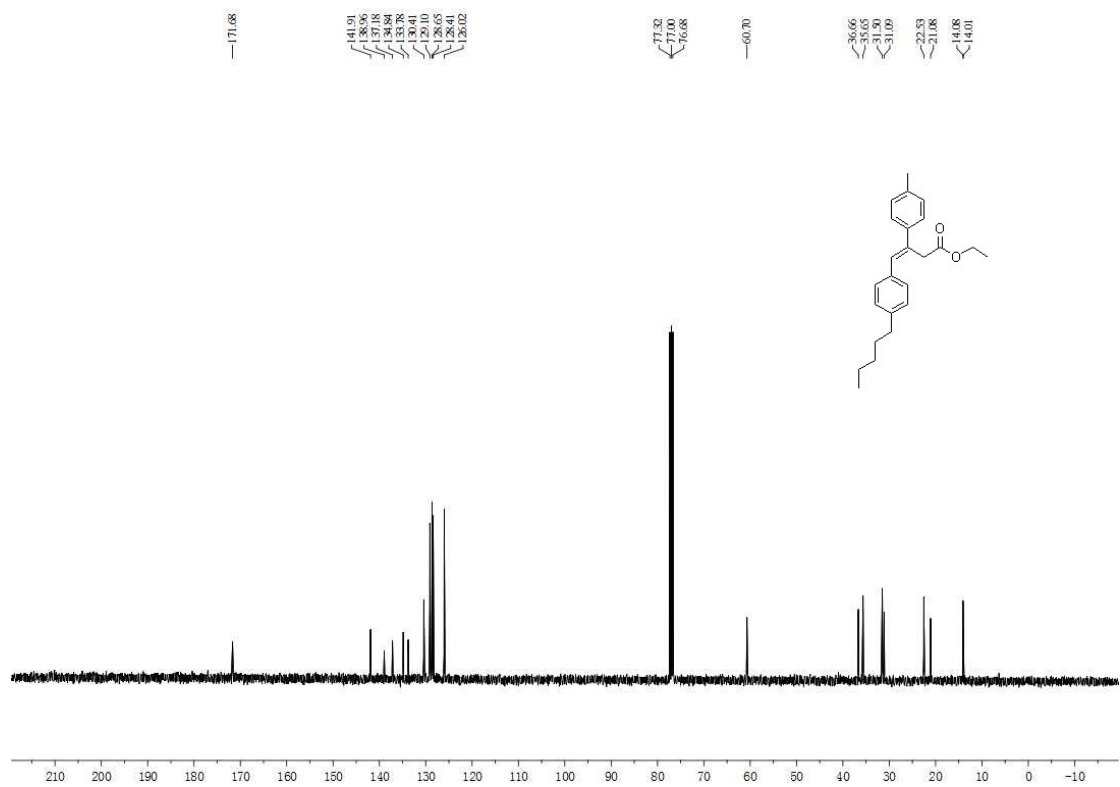
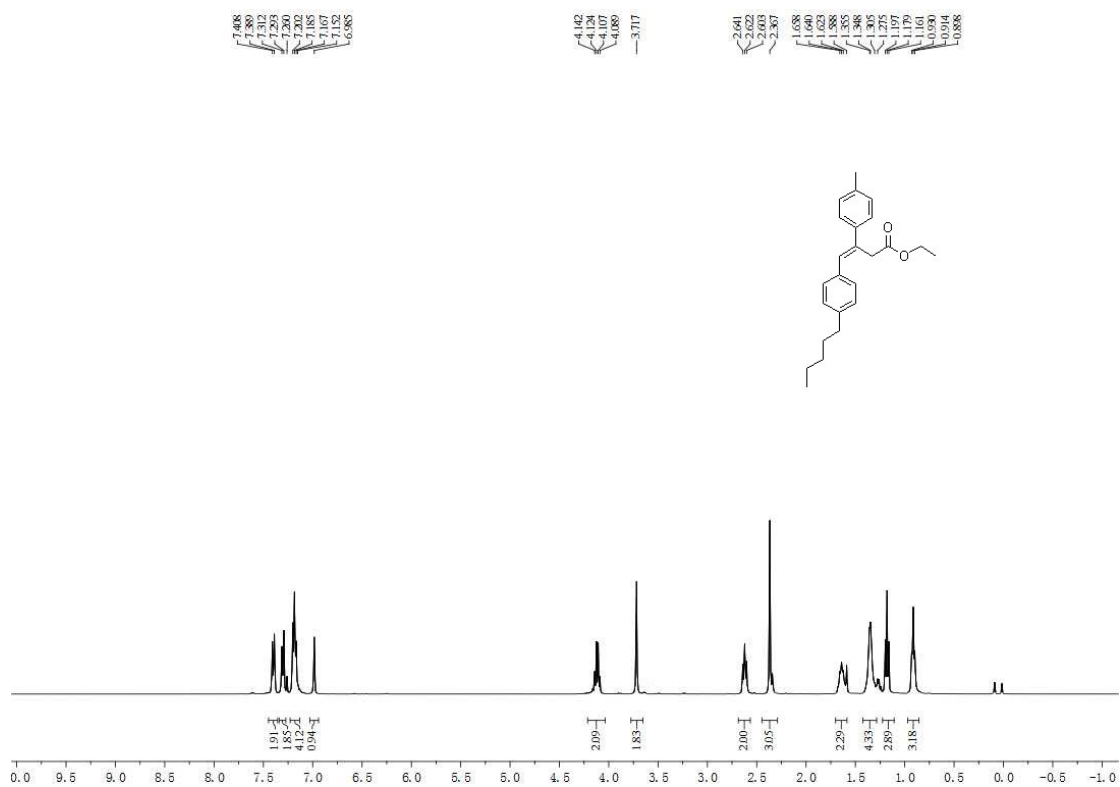
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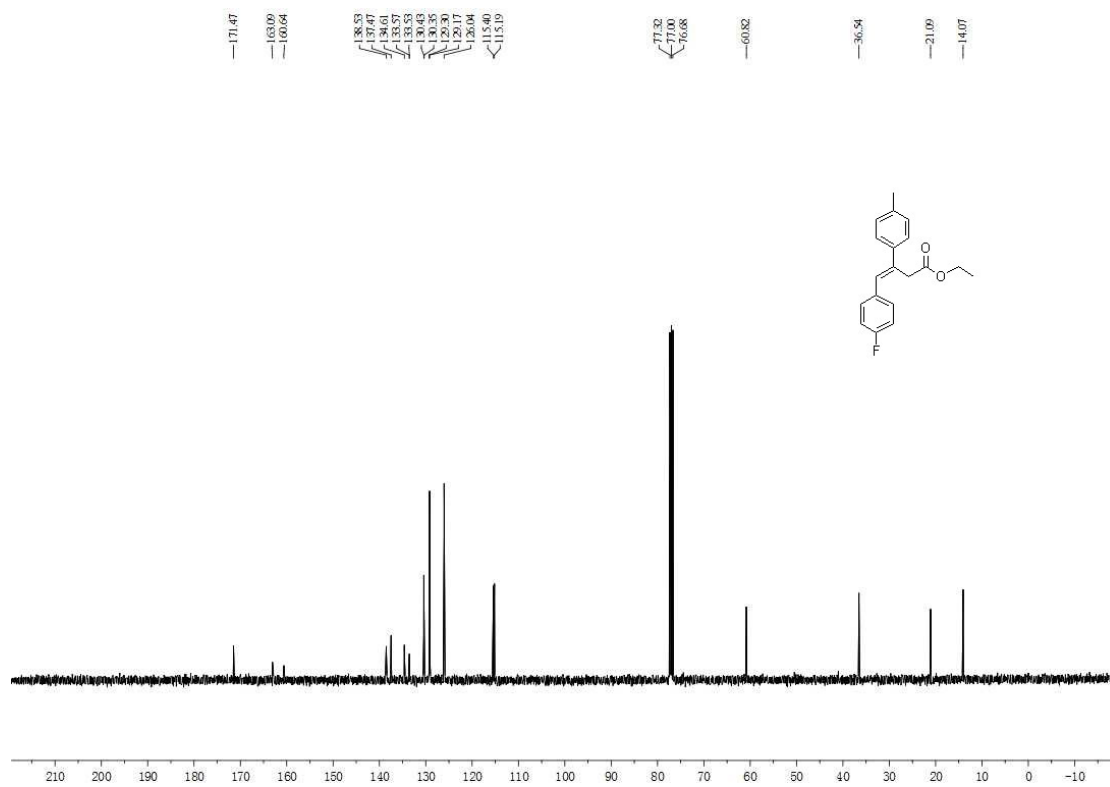
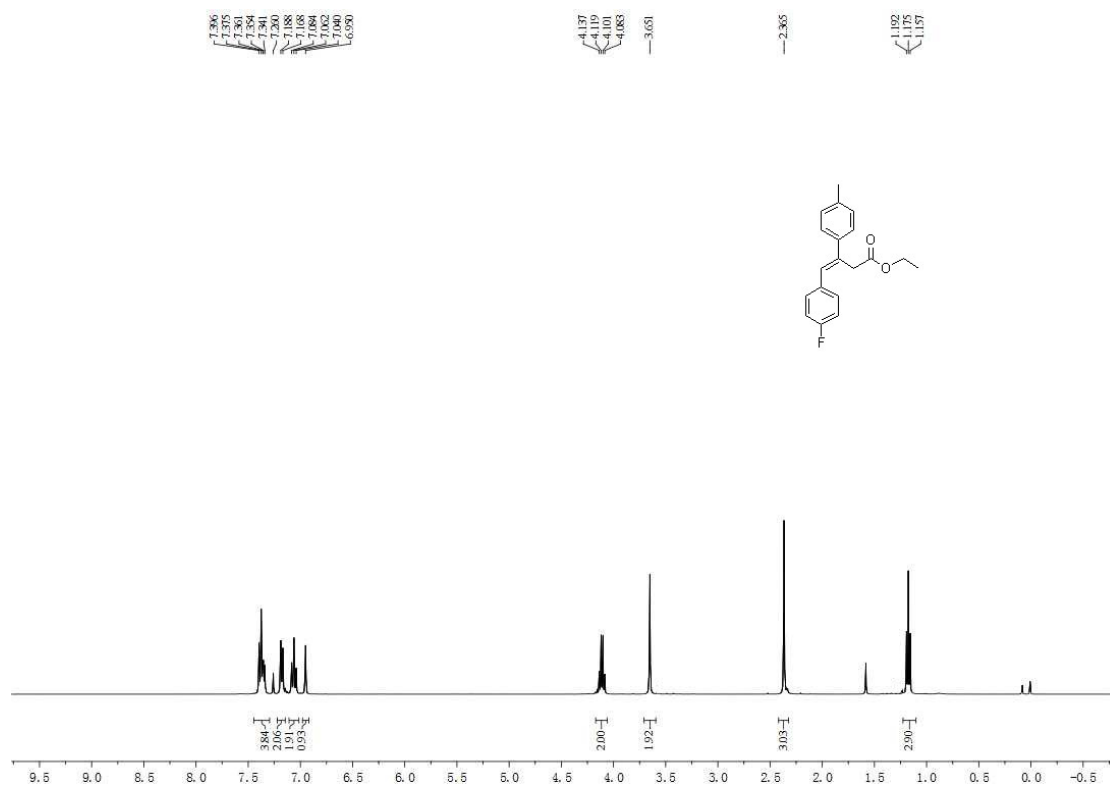
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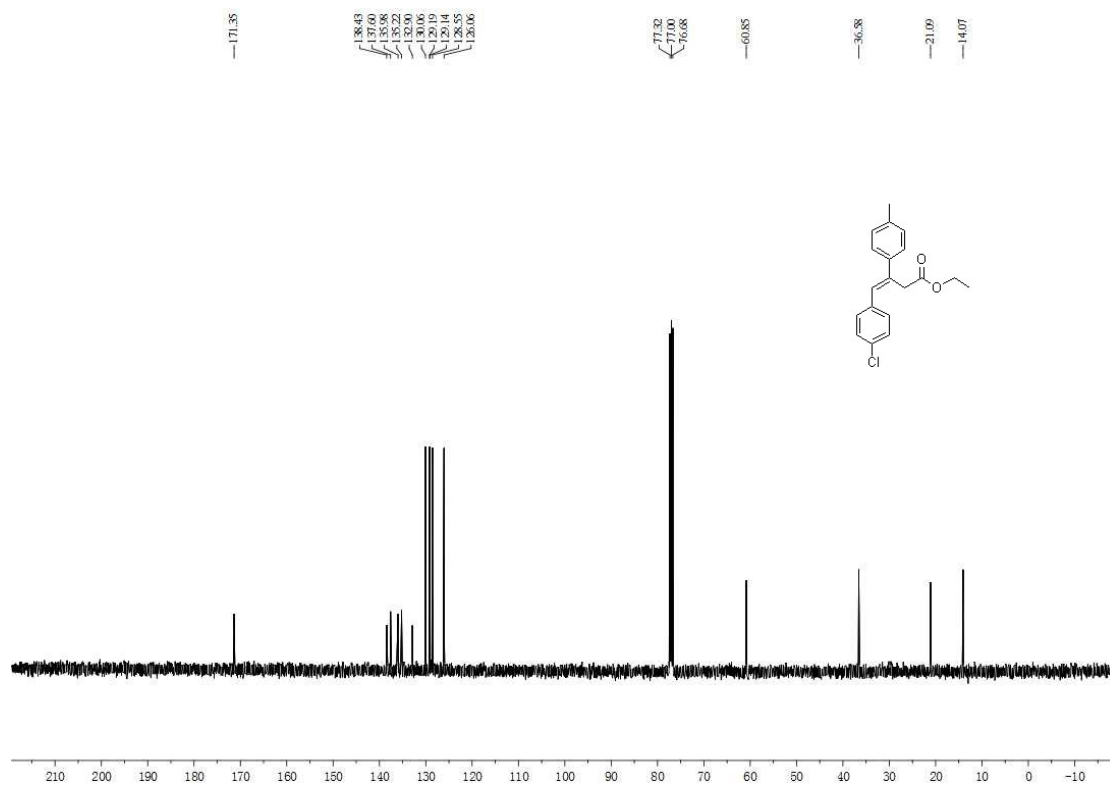
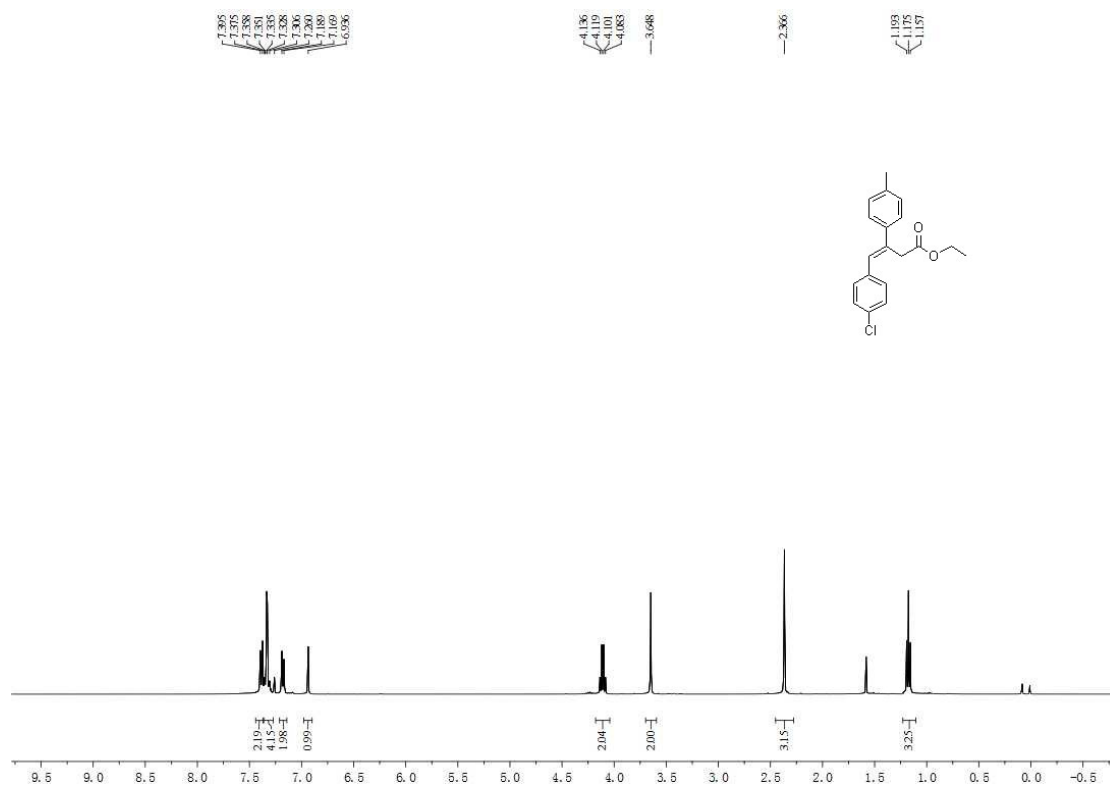
Product 4g



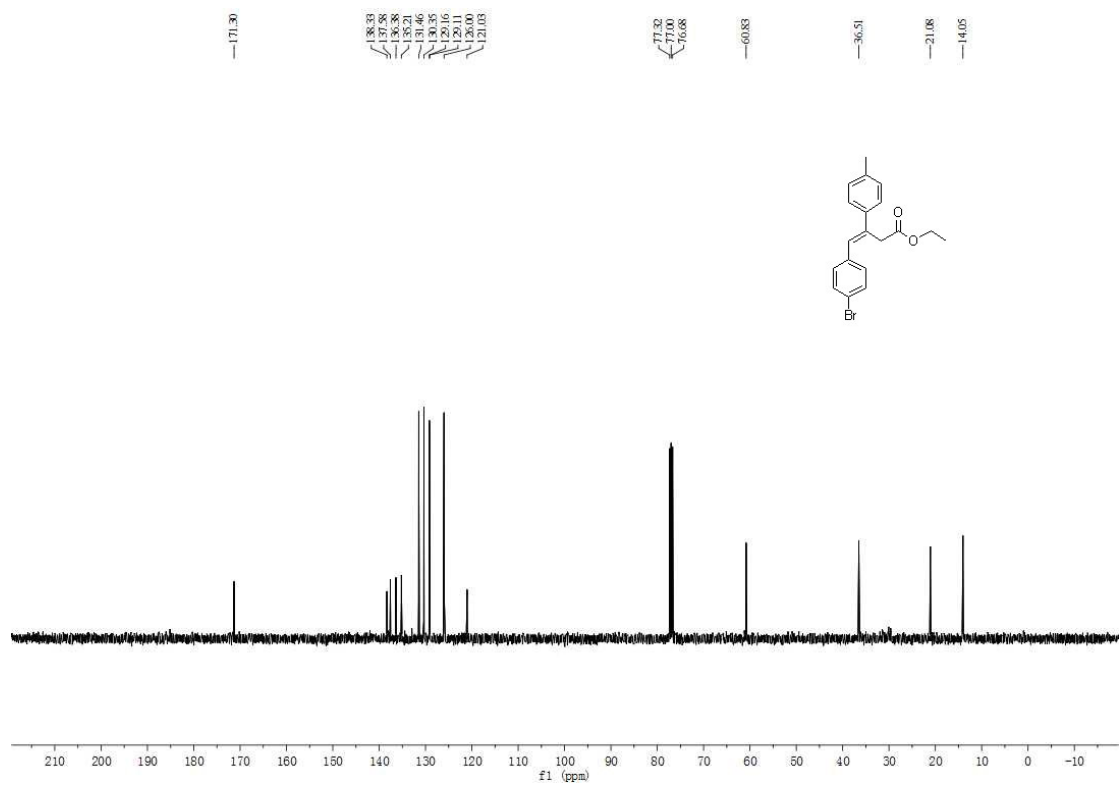
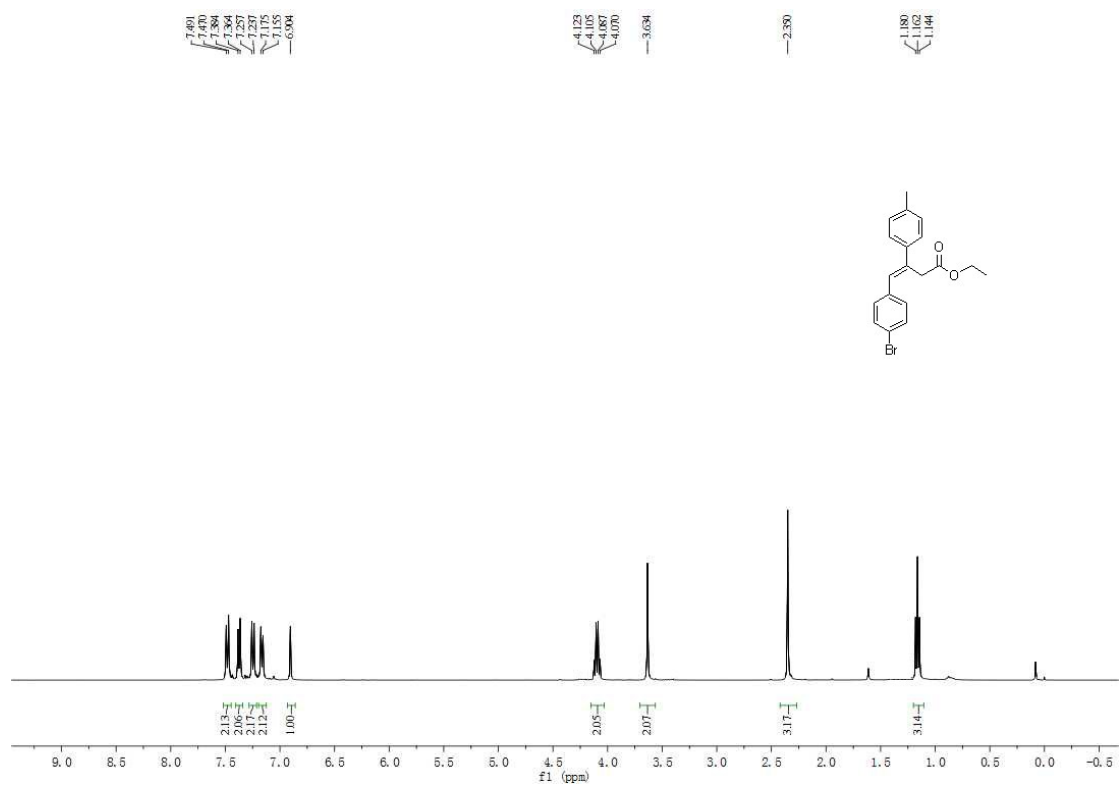
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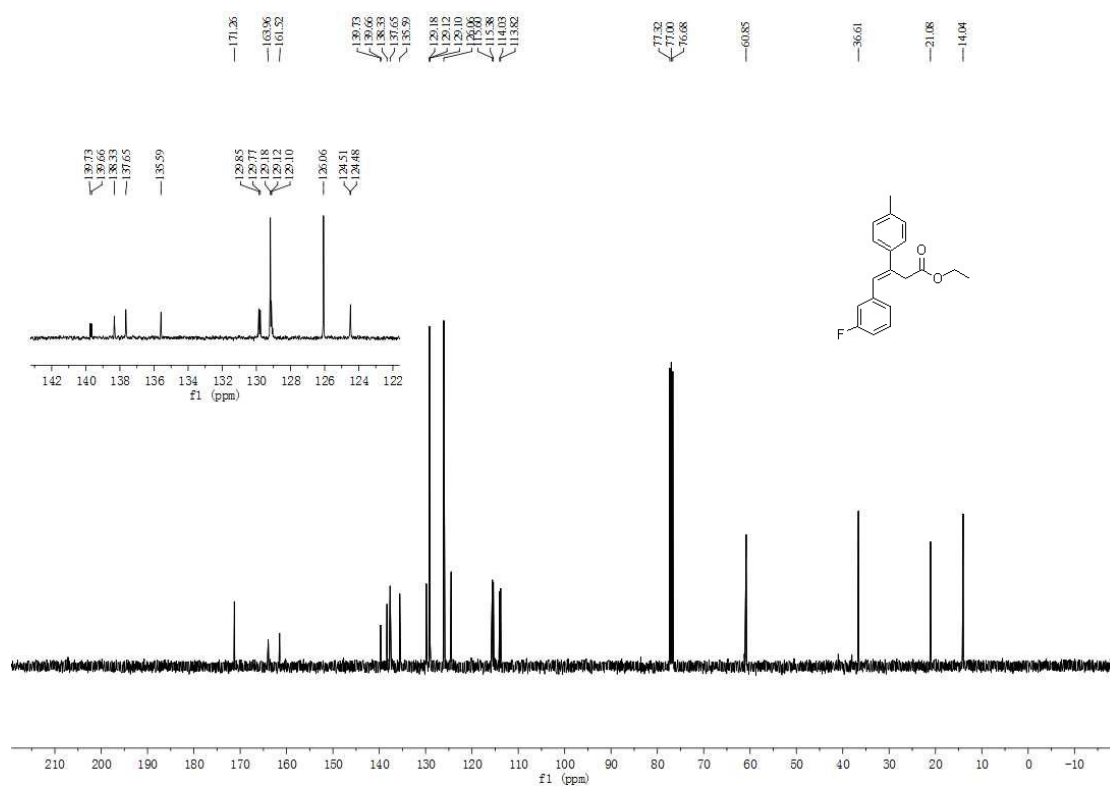
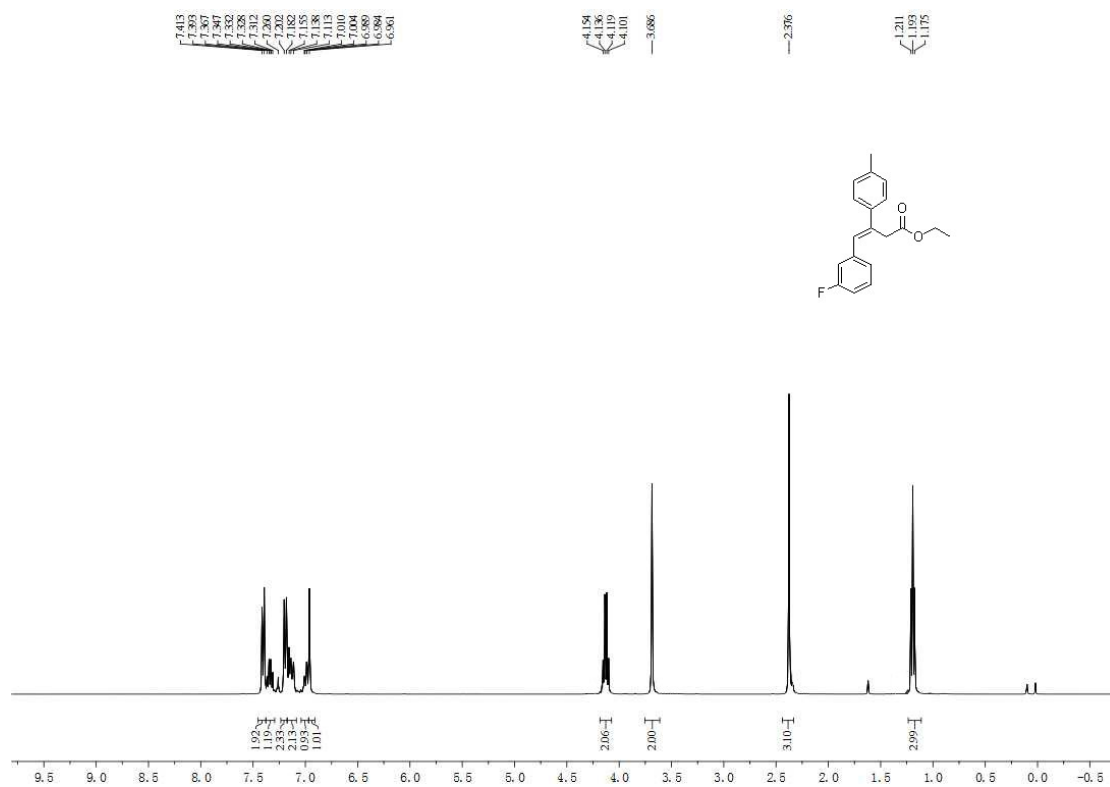
Product 4i



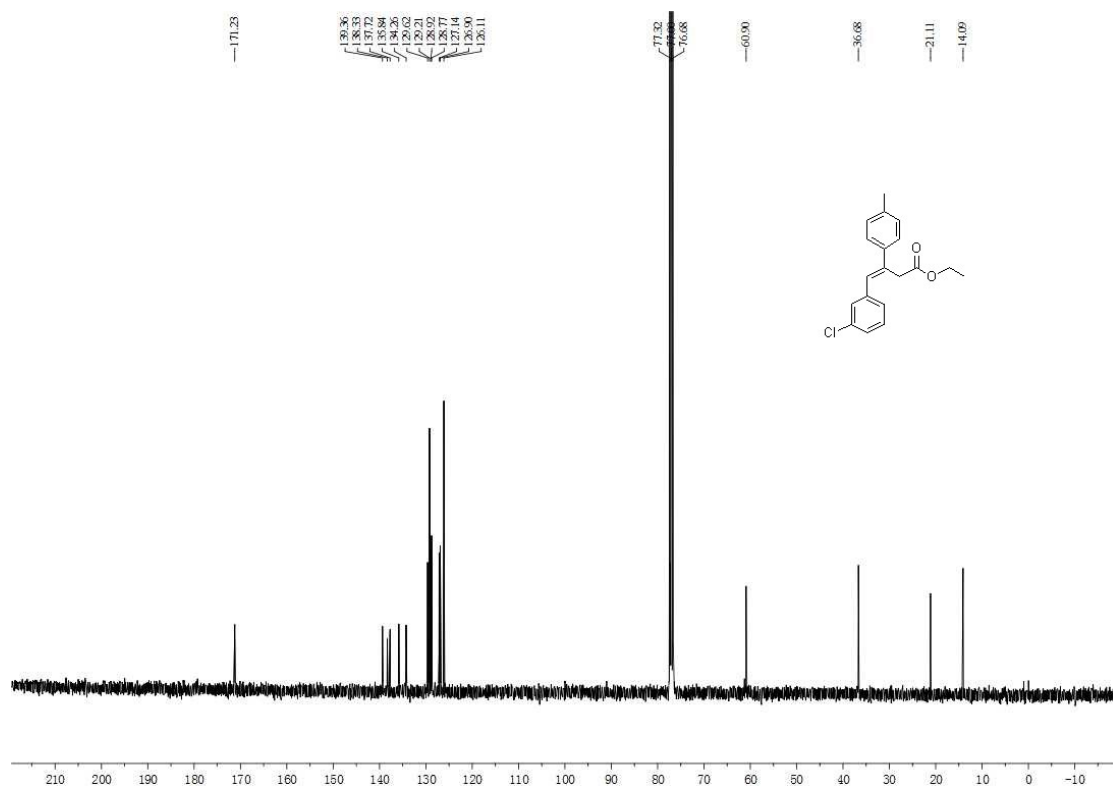
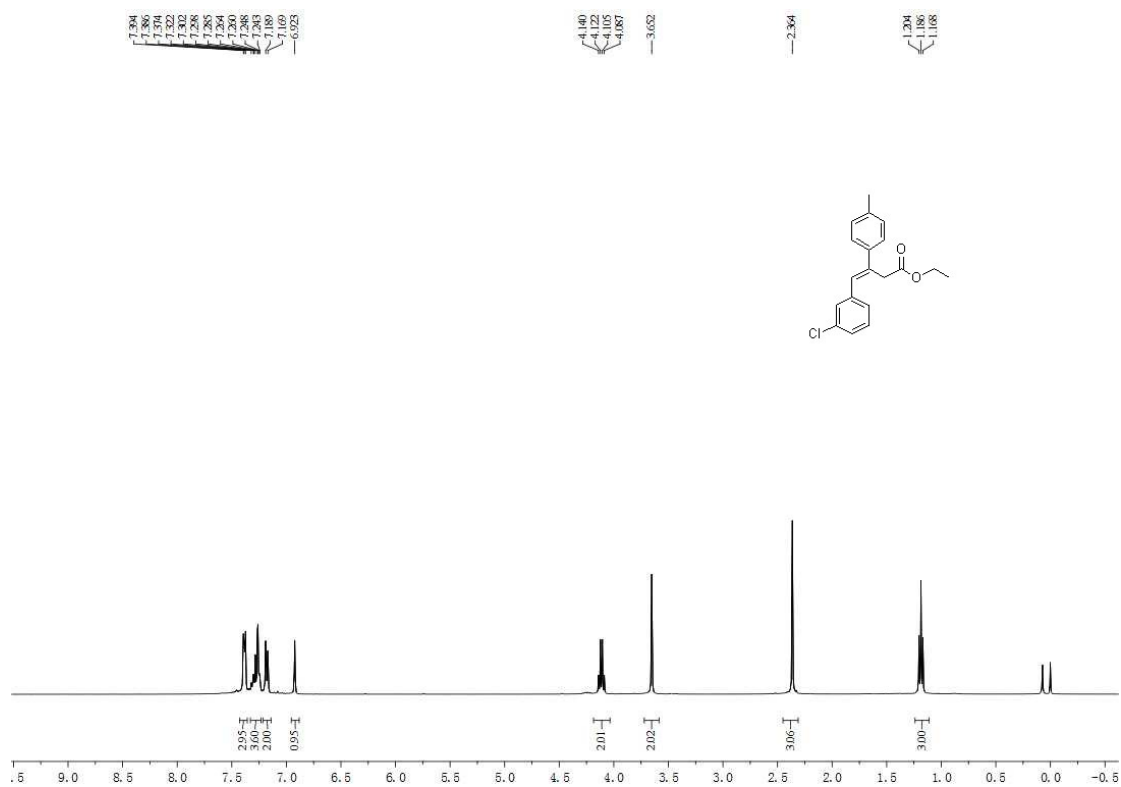
Product 4j



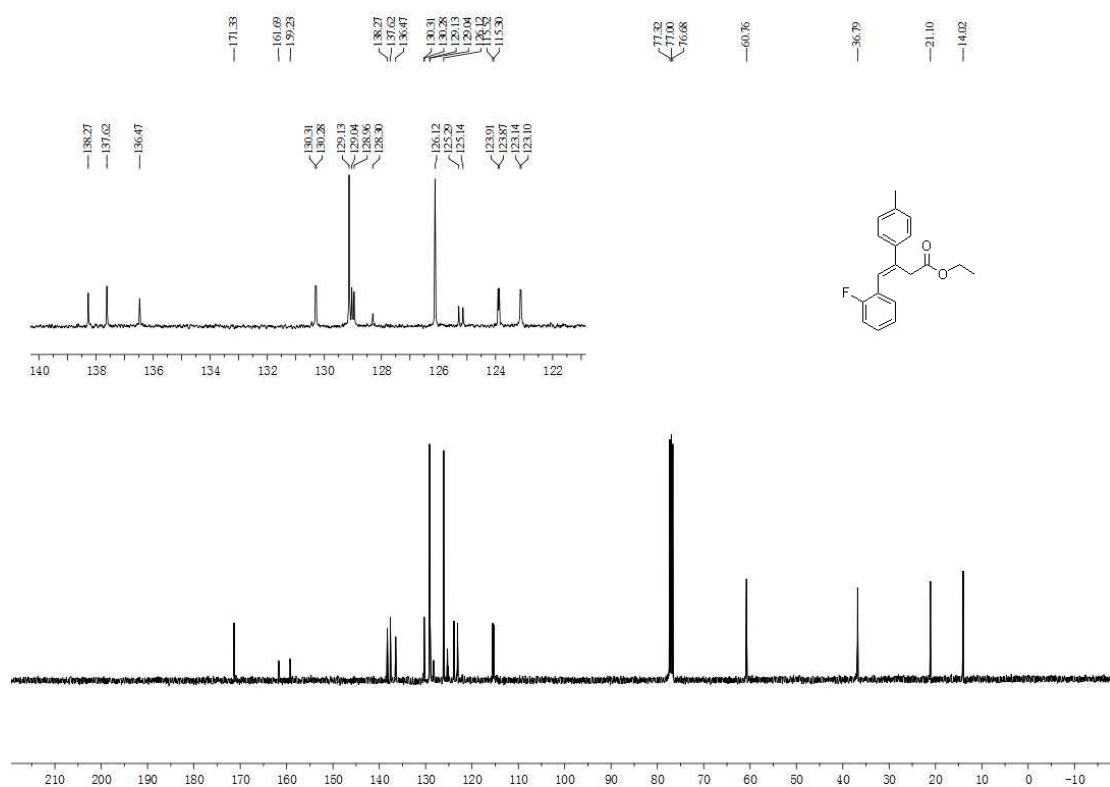
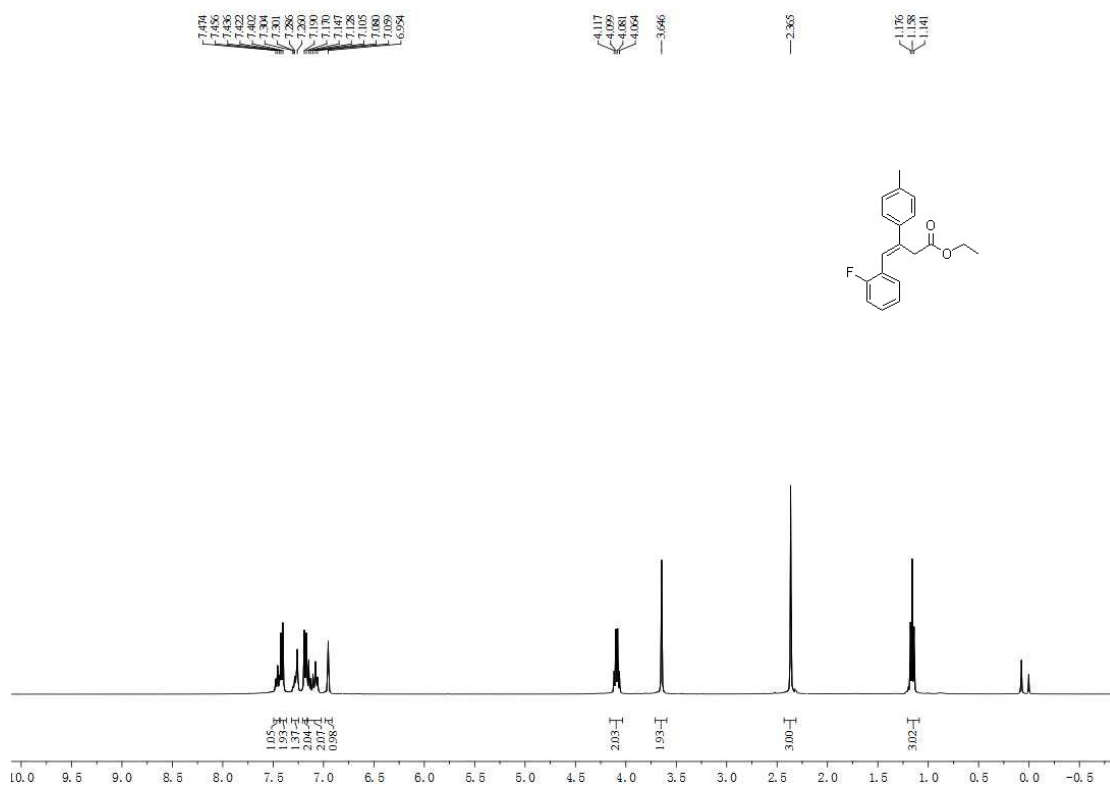
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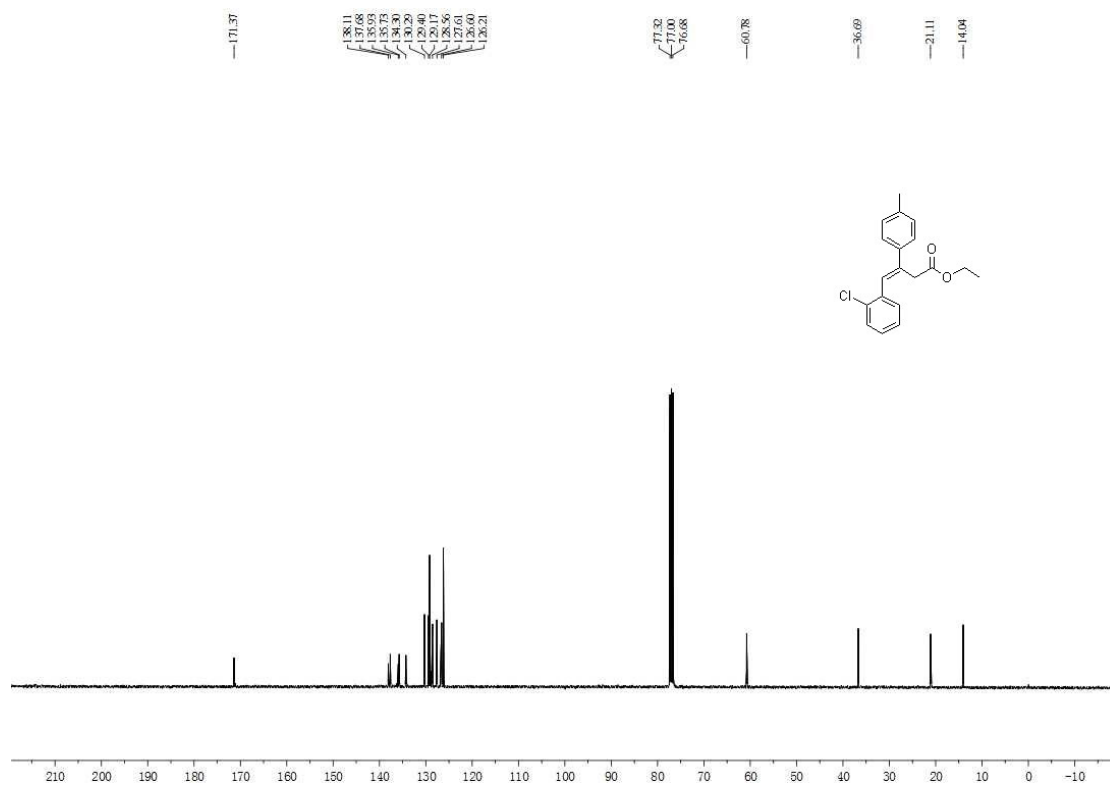
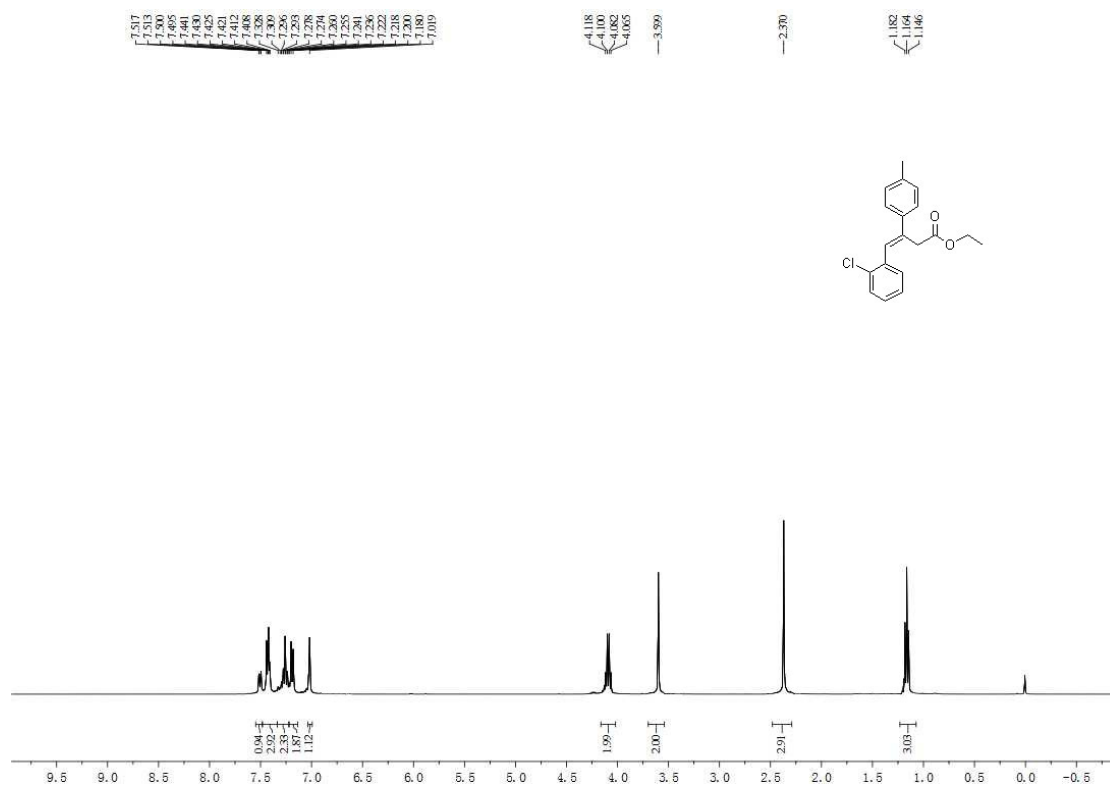
Product 4l



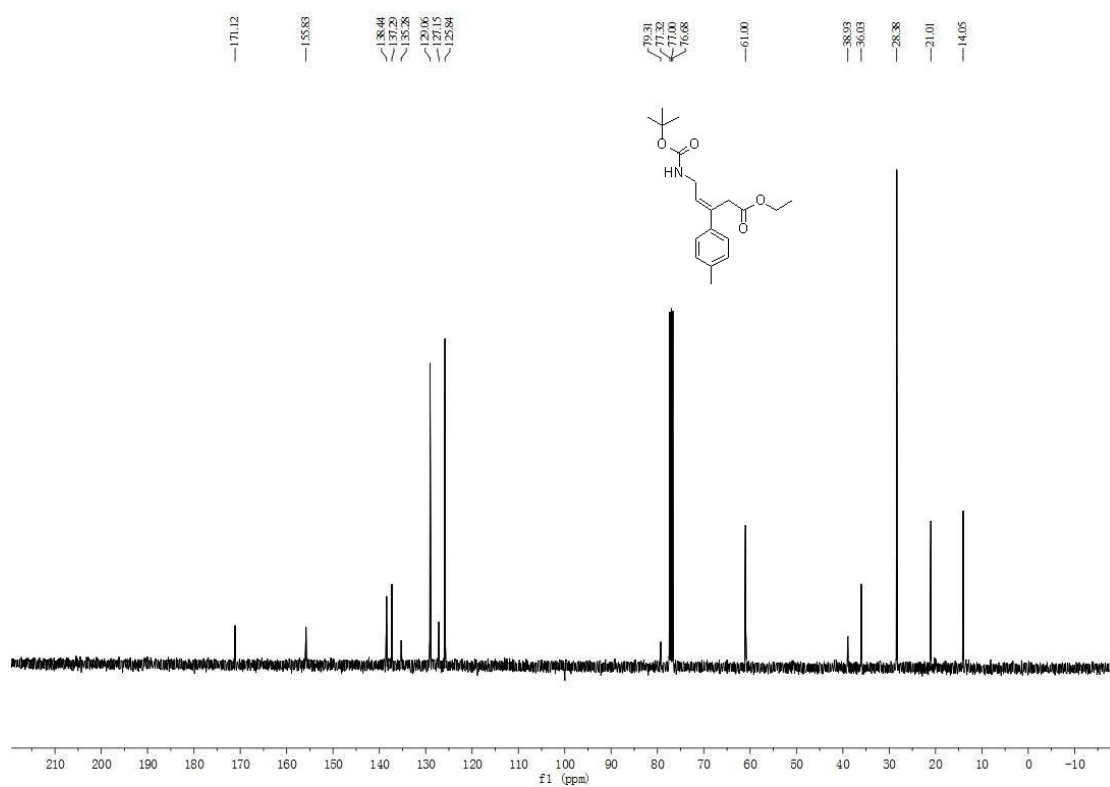
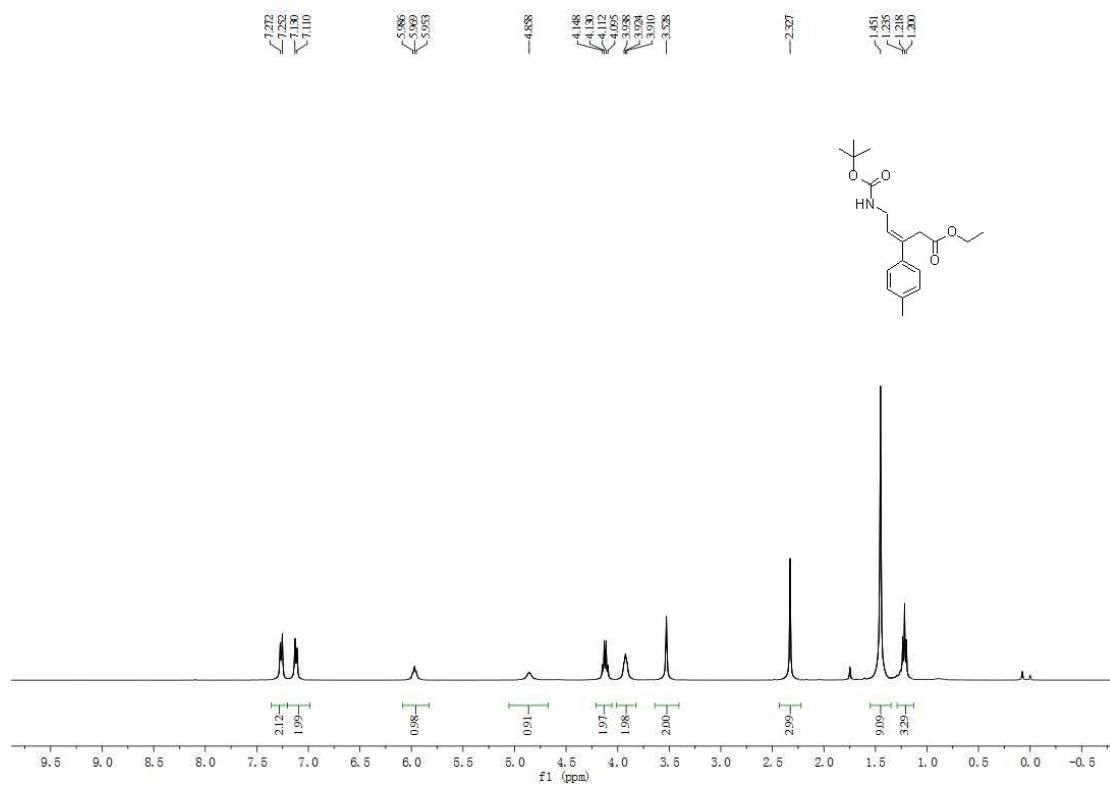
Product 4m



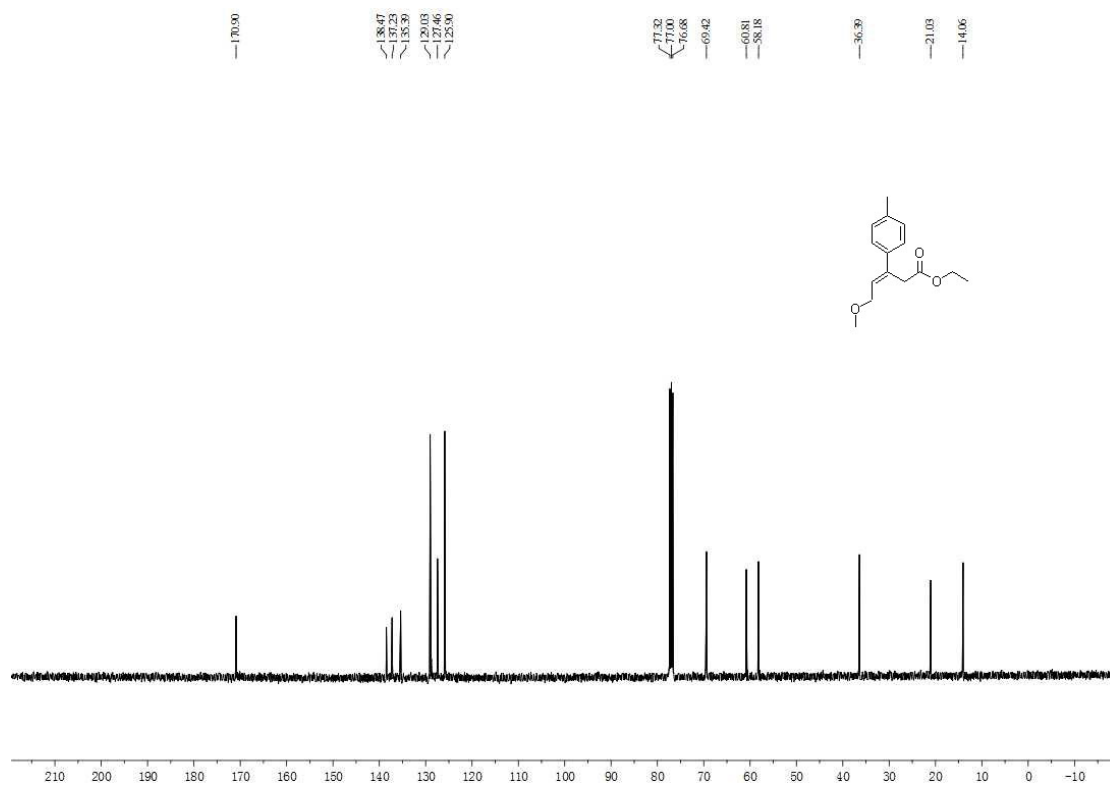
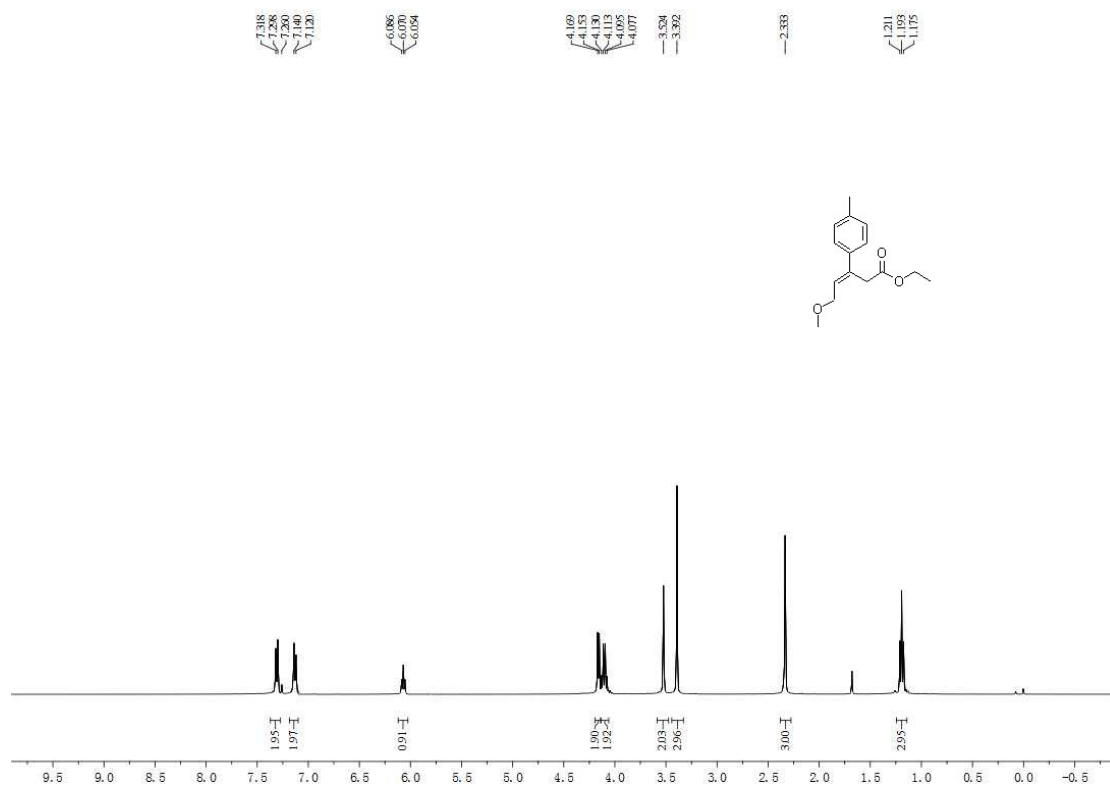
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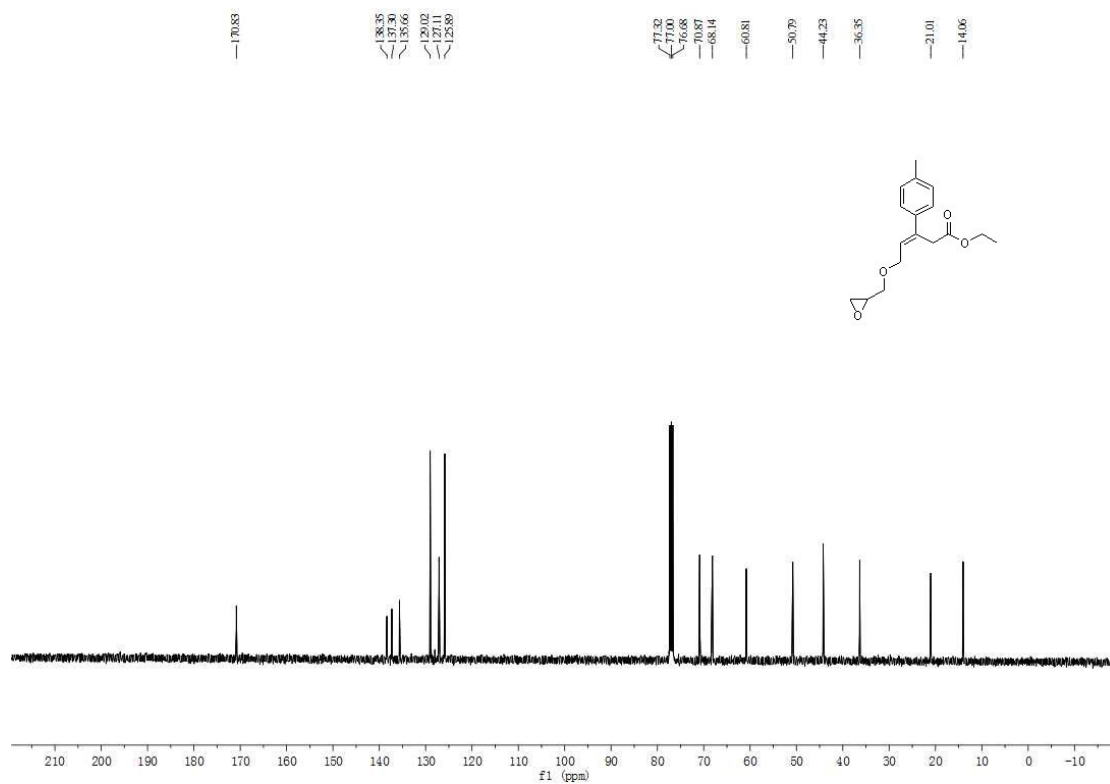
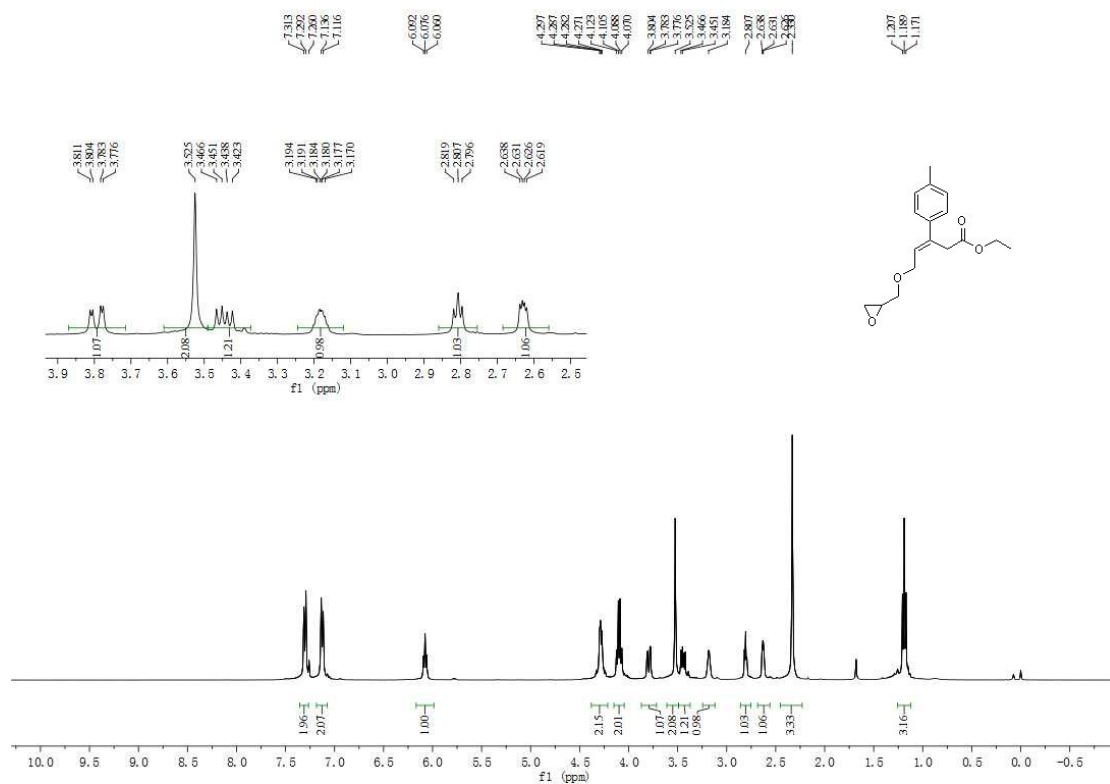
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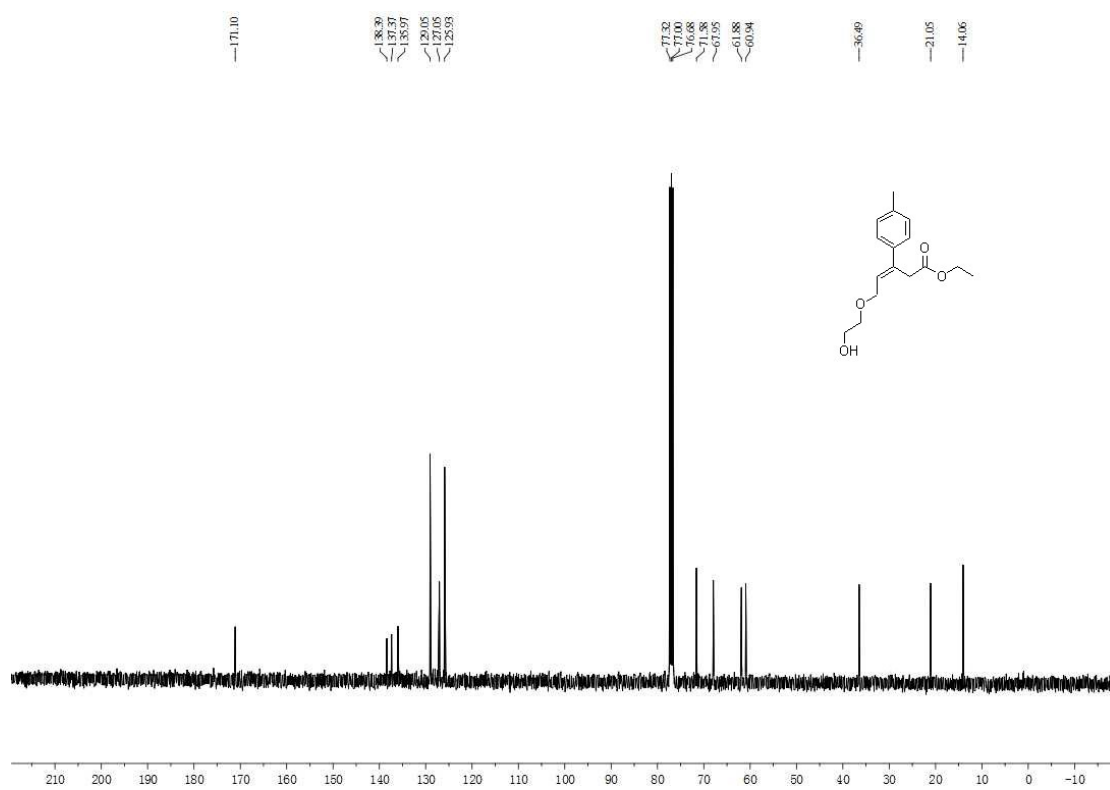
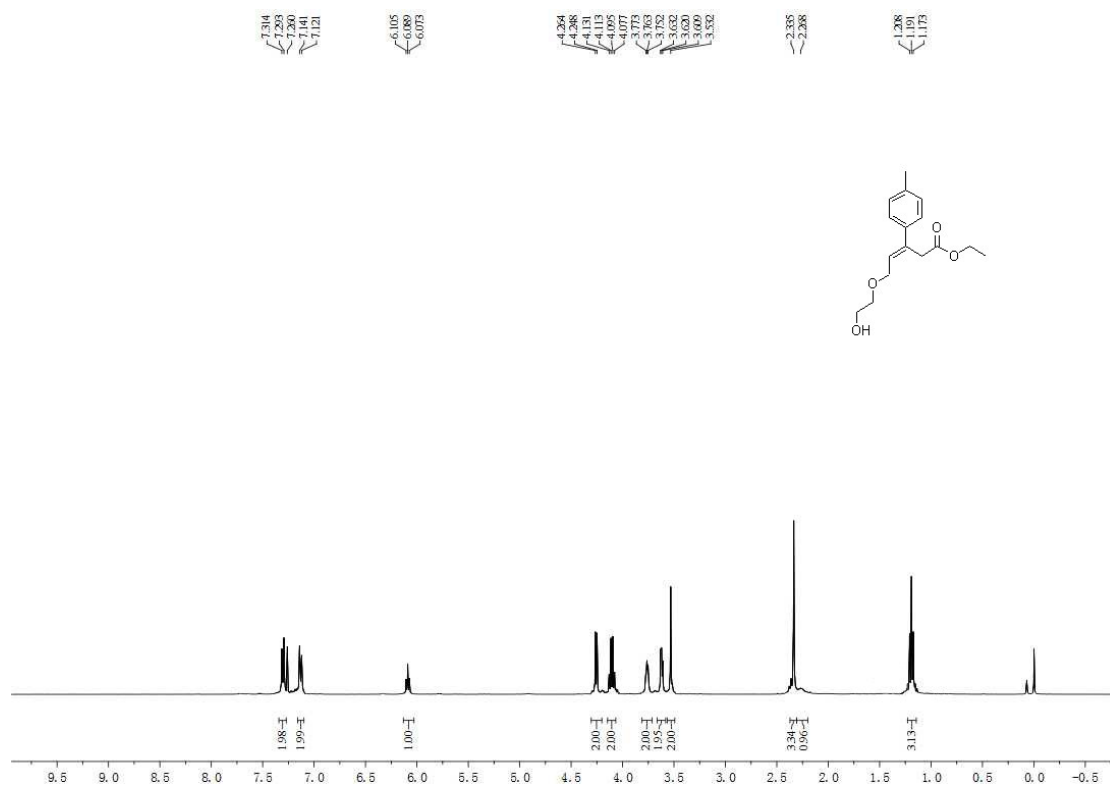
Product 4p



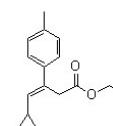
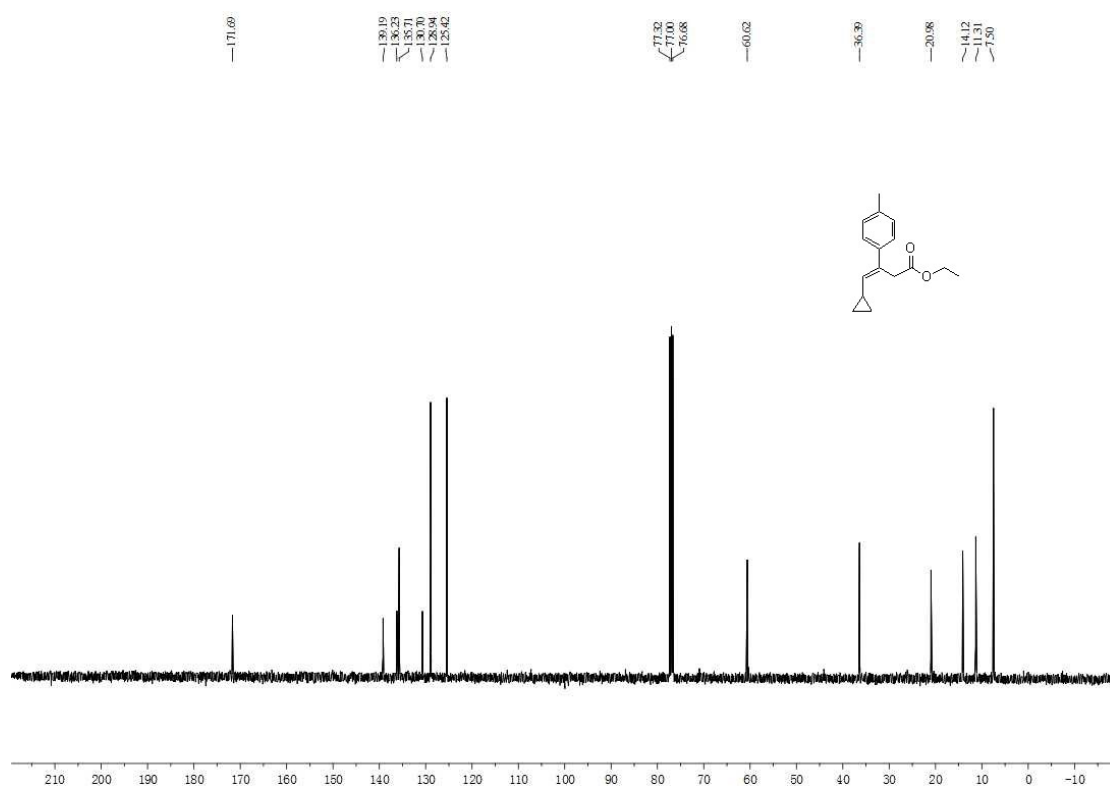
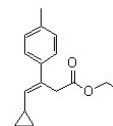
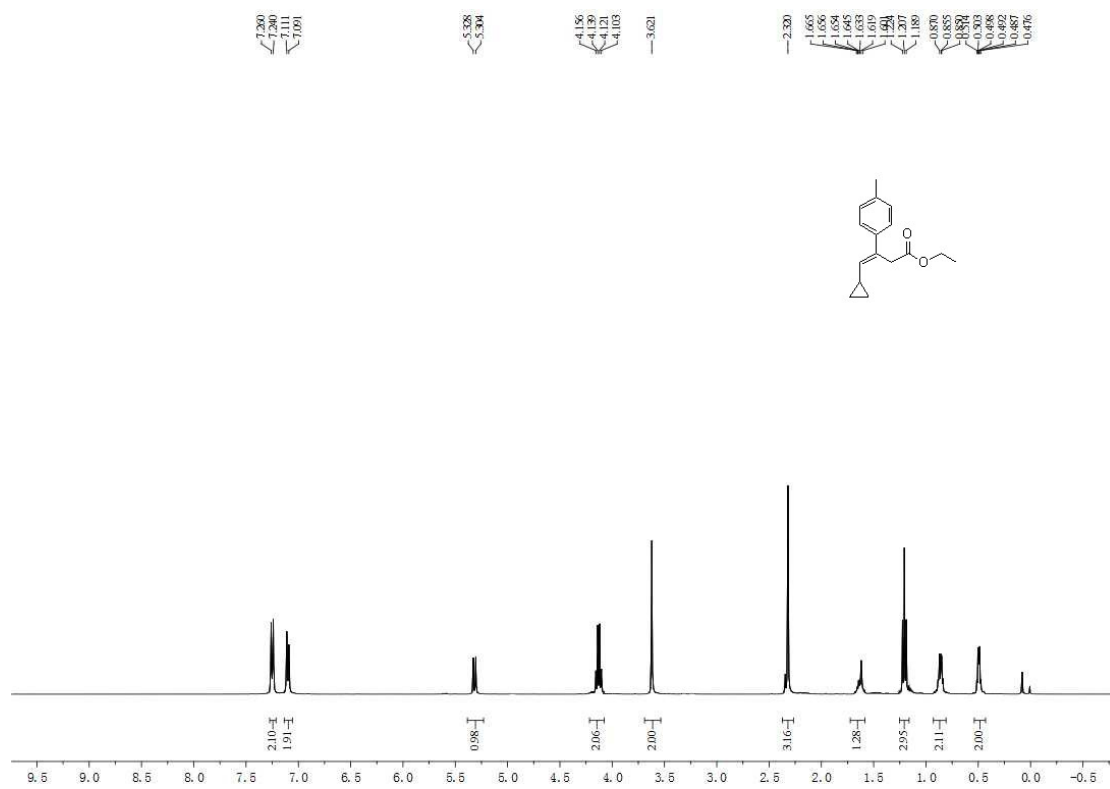
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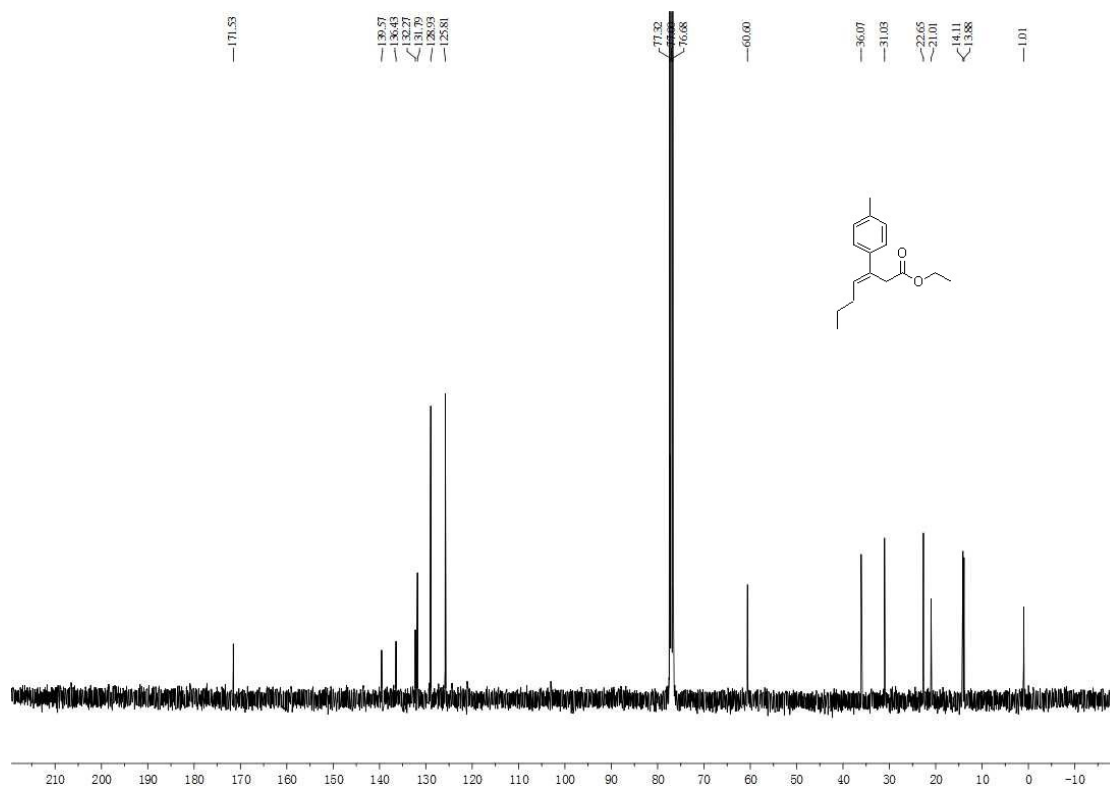
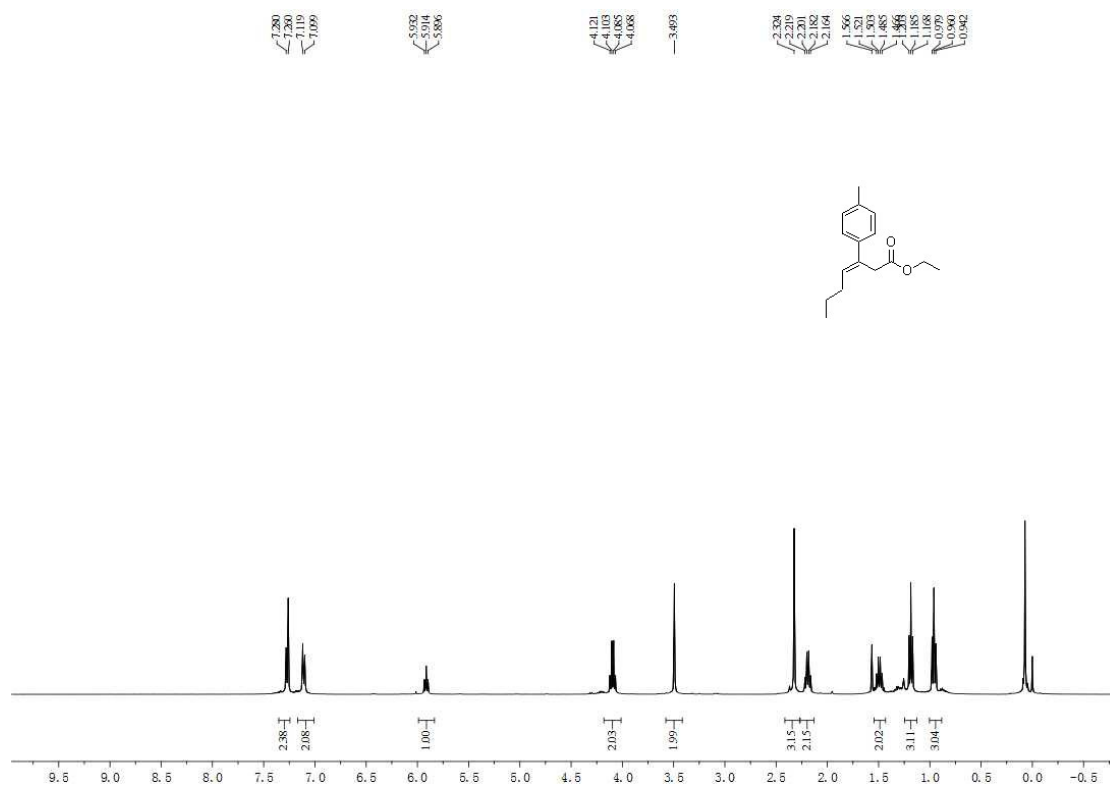
Product 4r



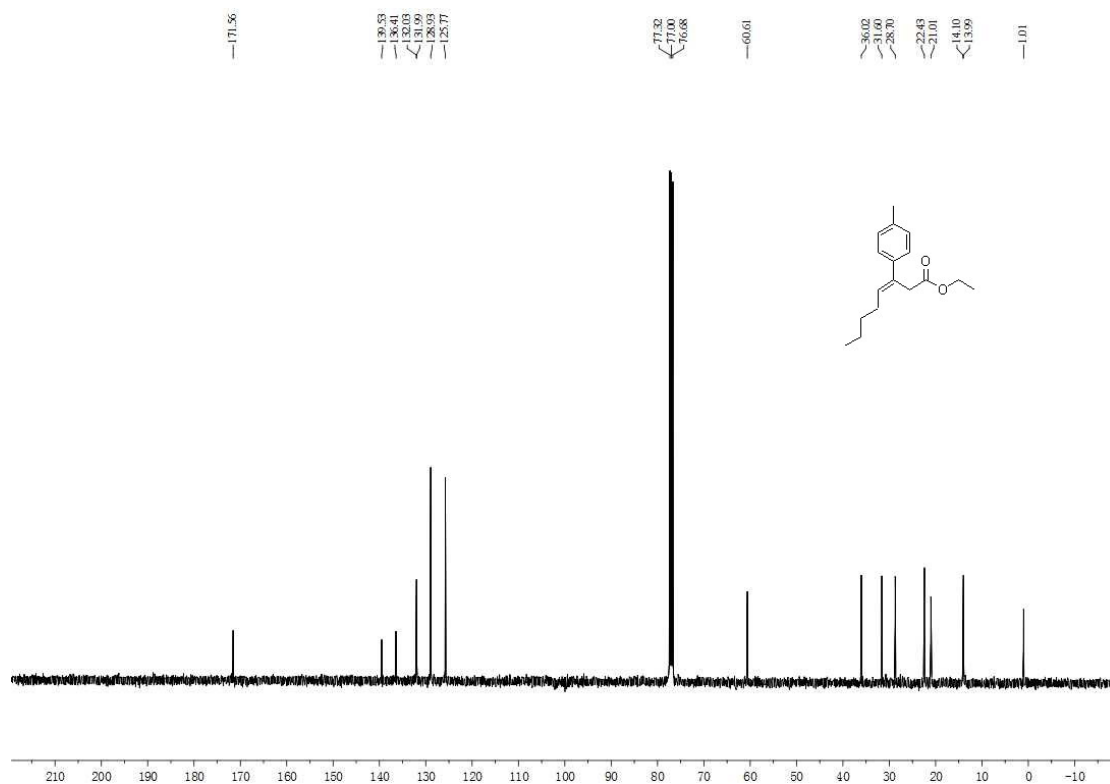
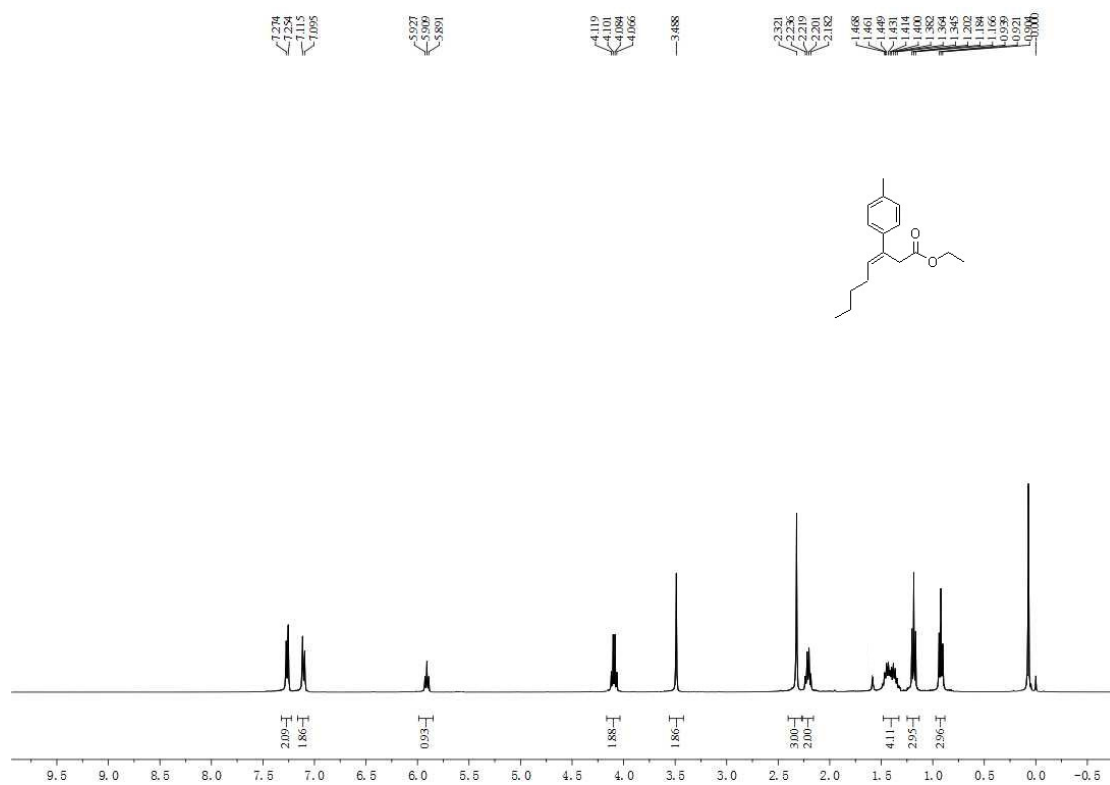
Product 4s



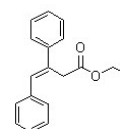
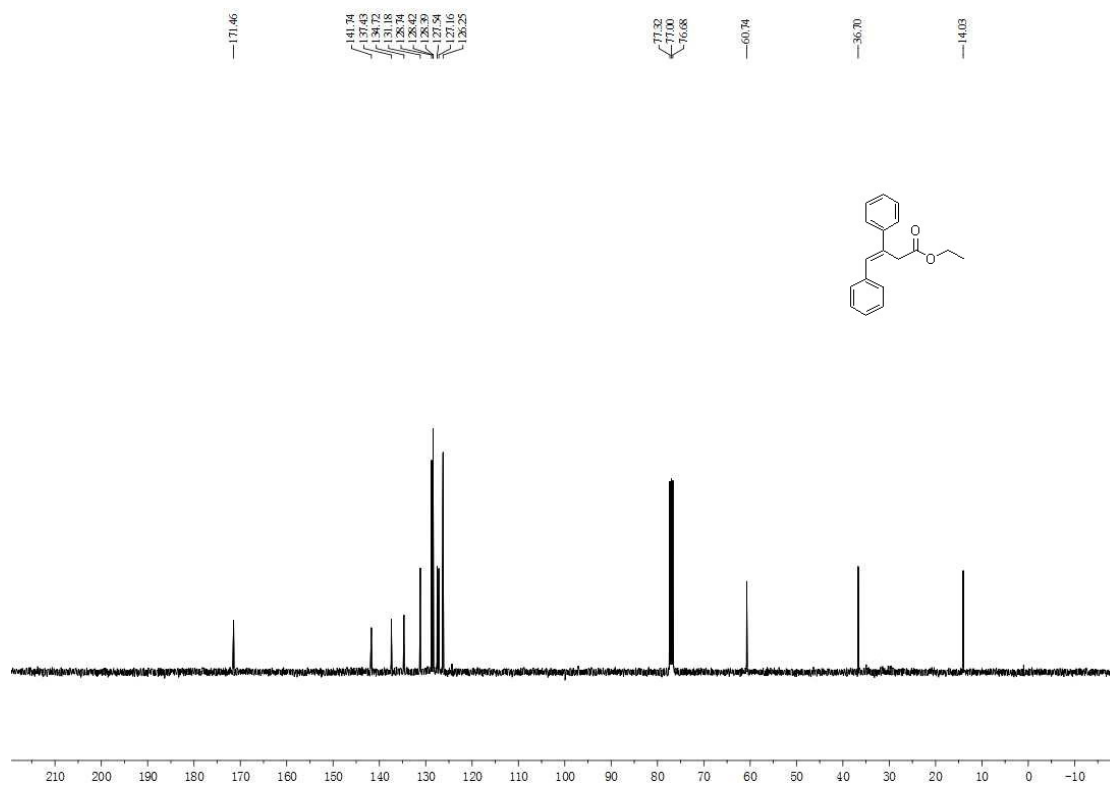
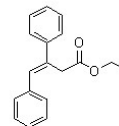
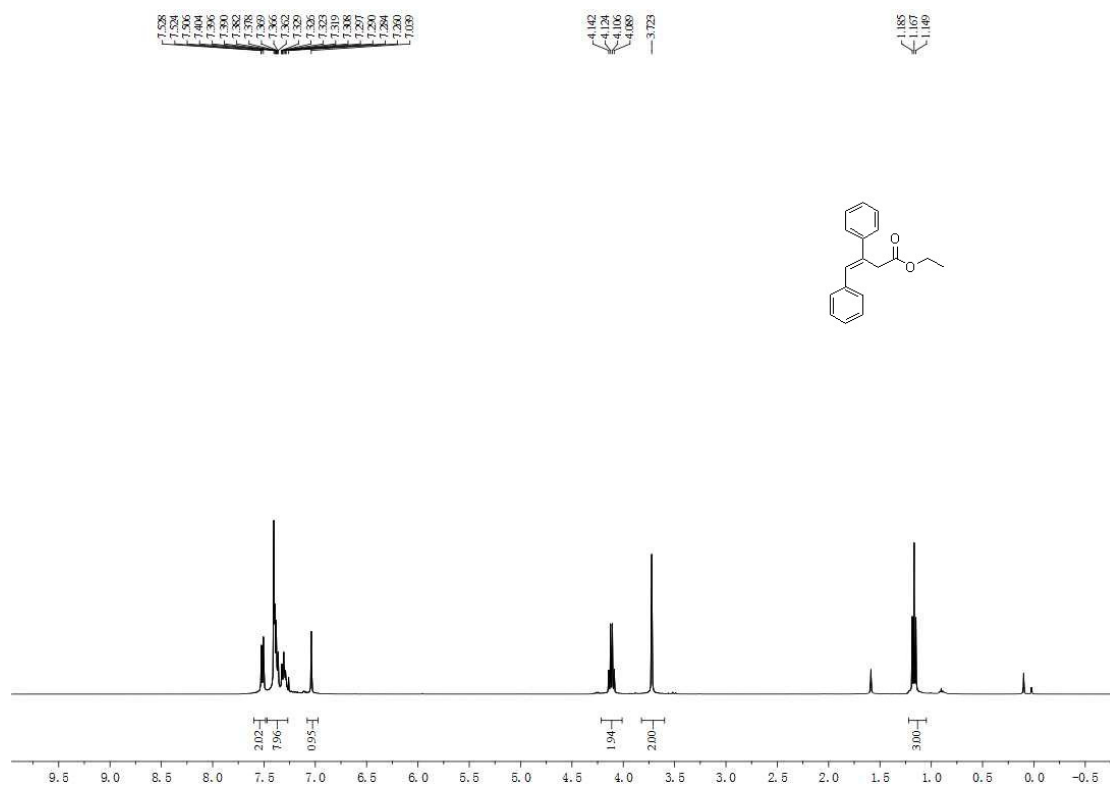
Product 4t



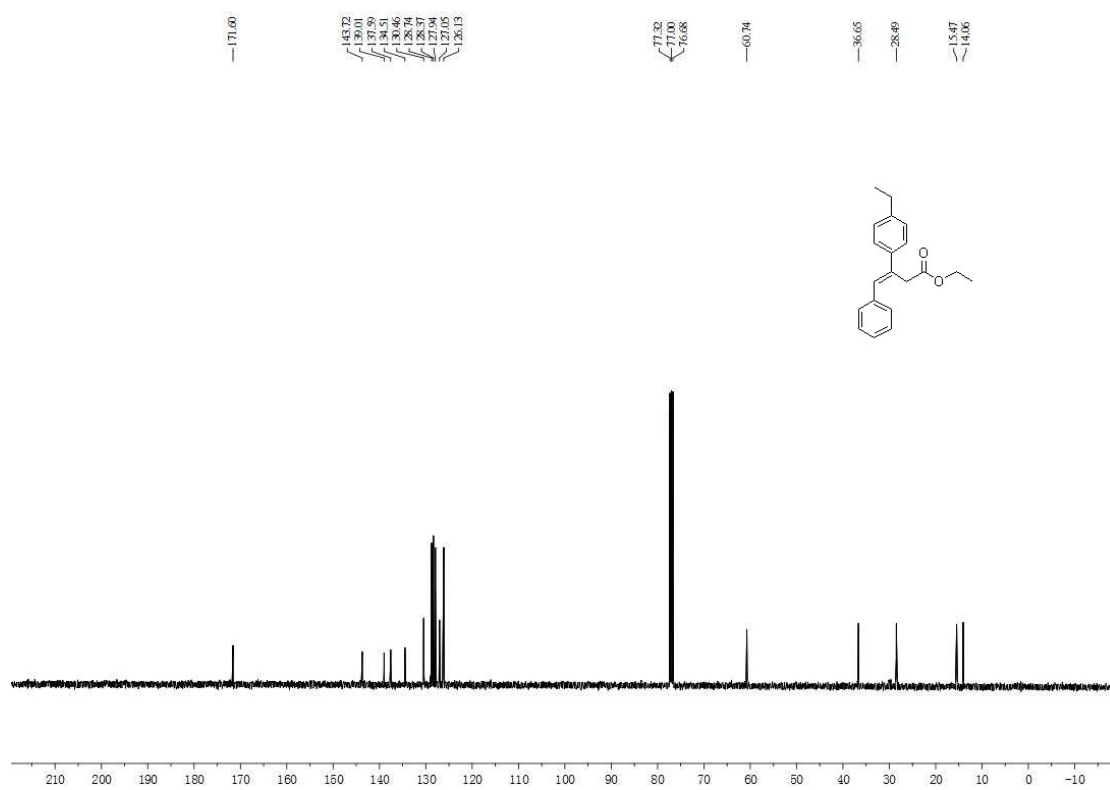
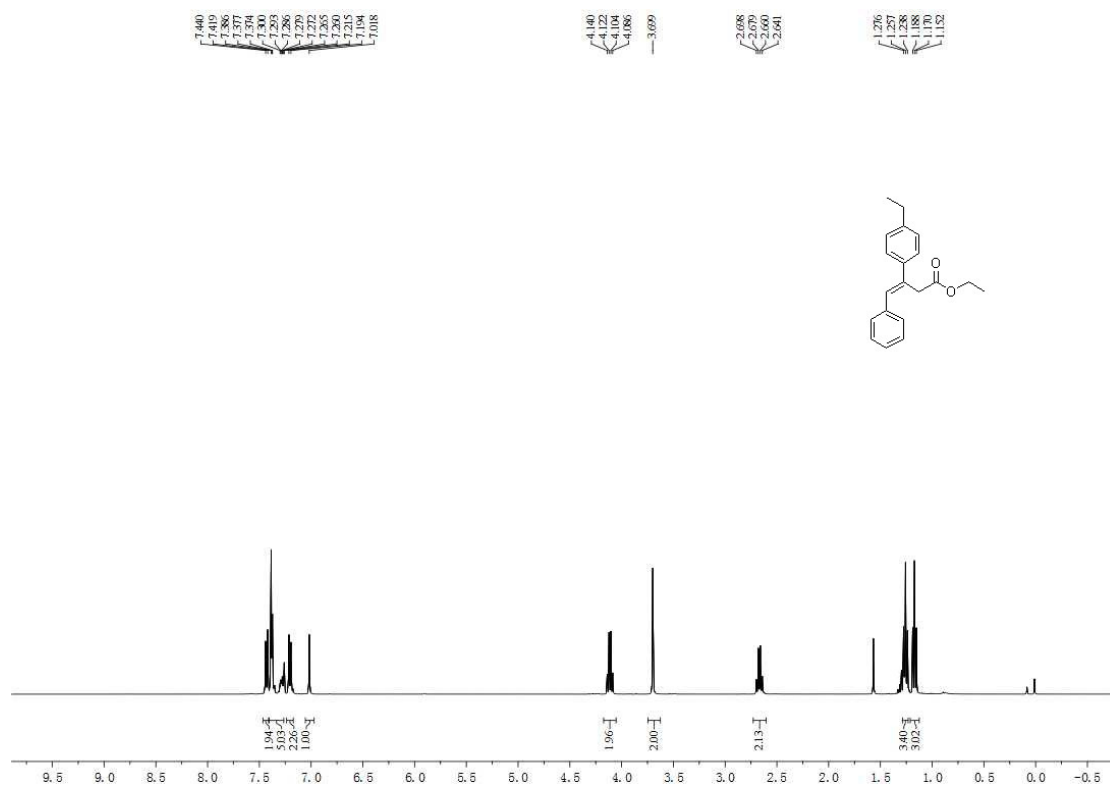
Product 4u



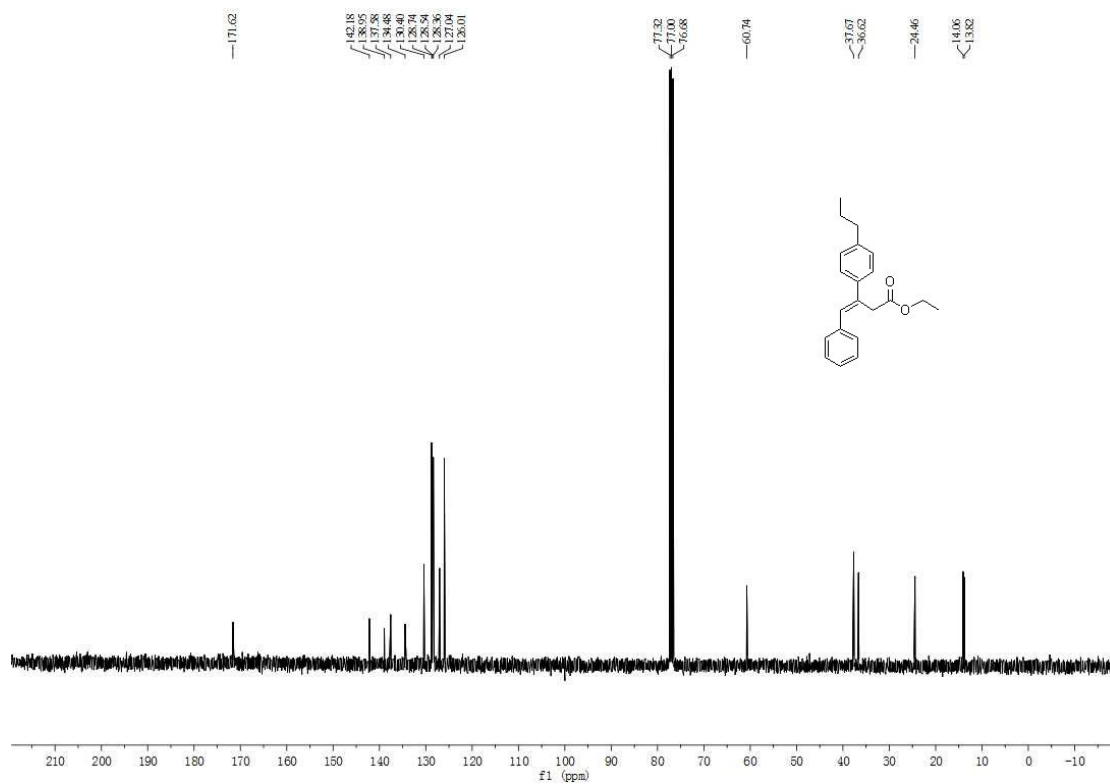
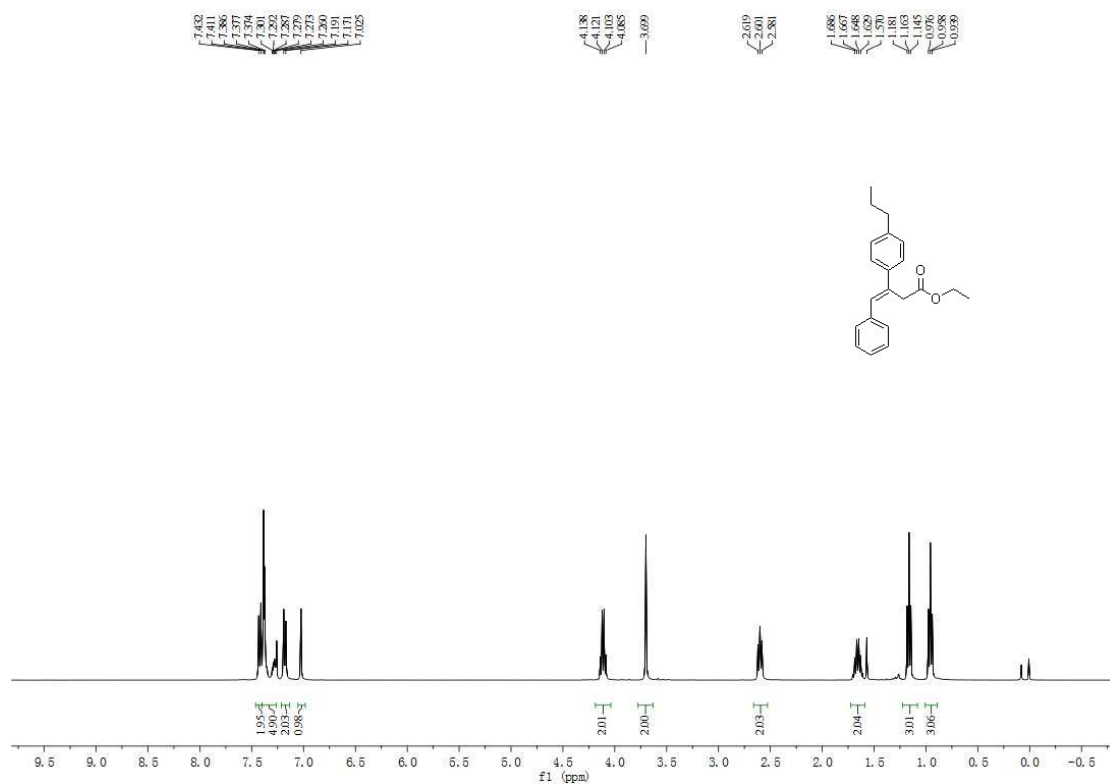
Product 5a



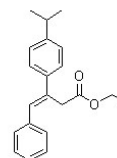
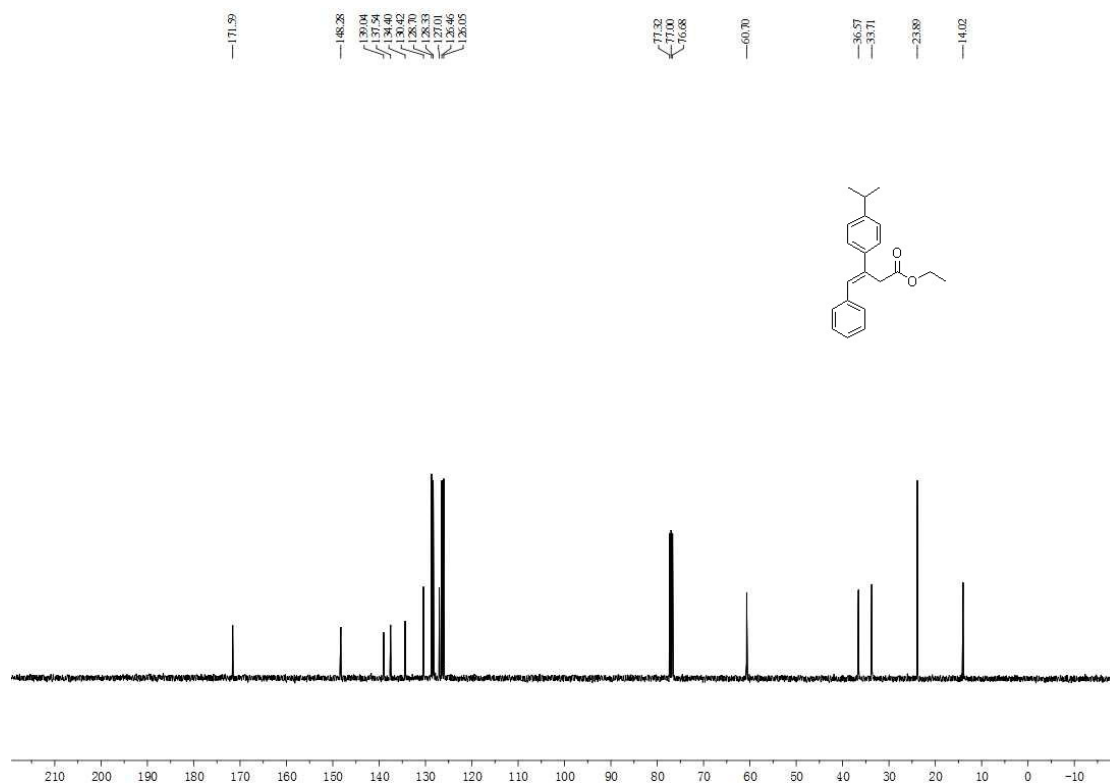
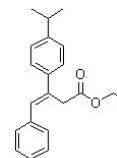
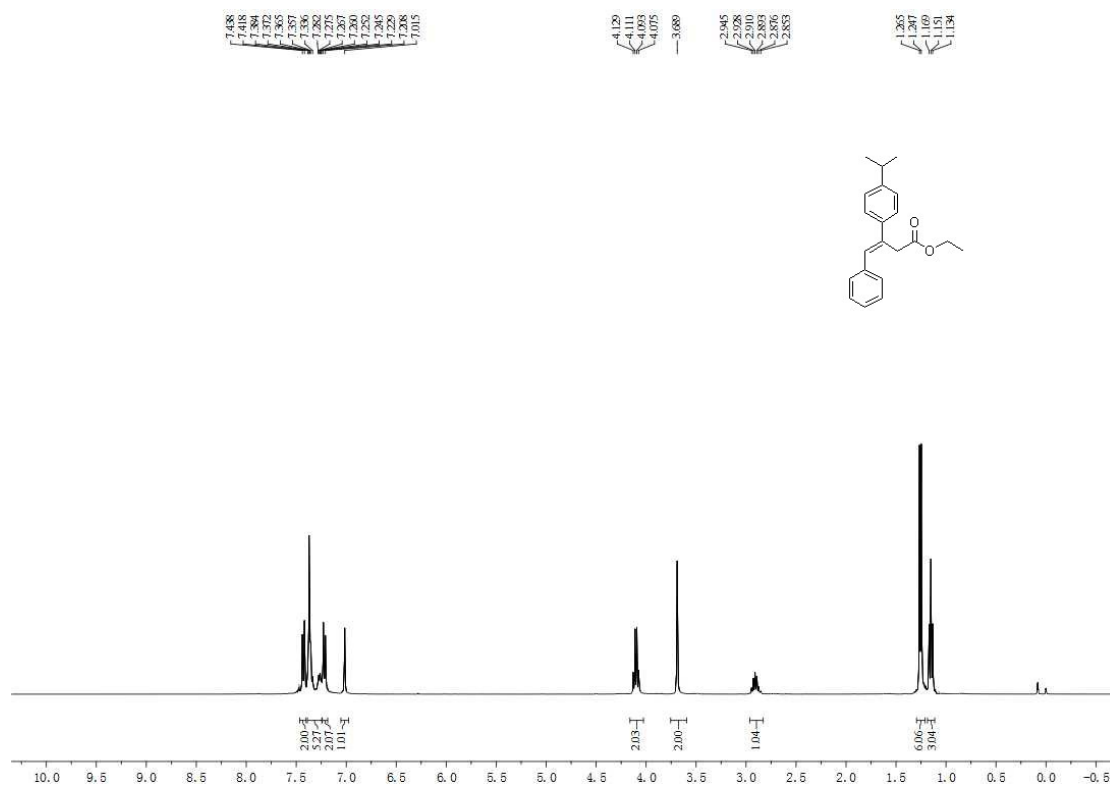
Product 5b



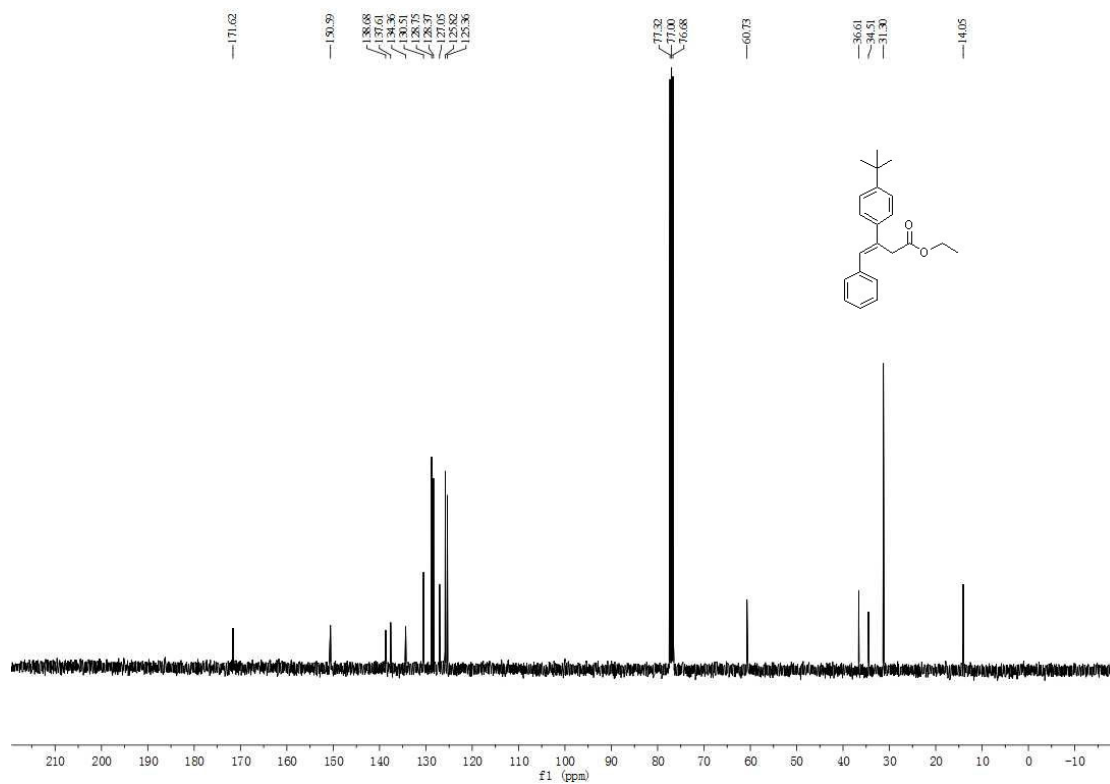
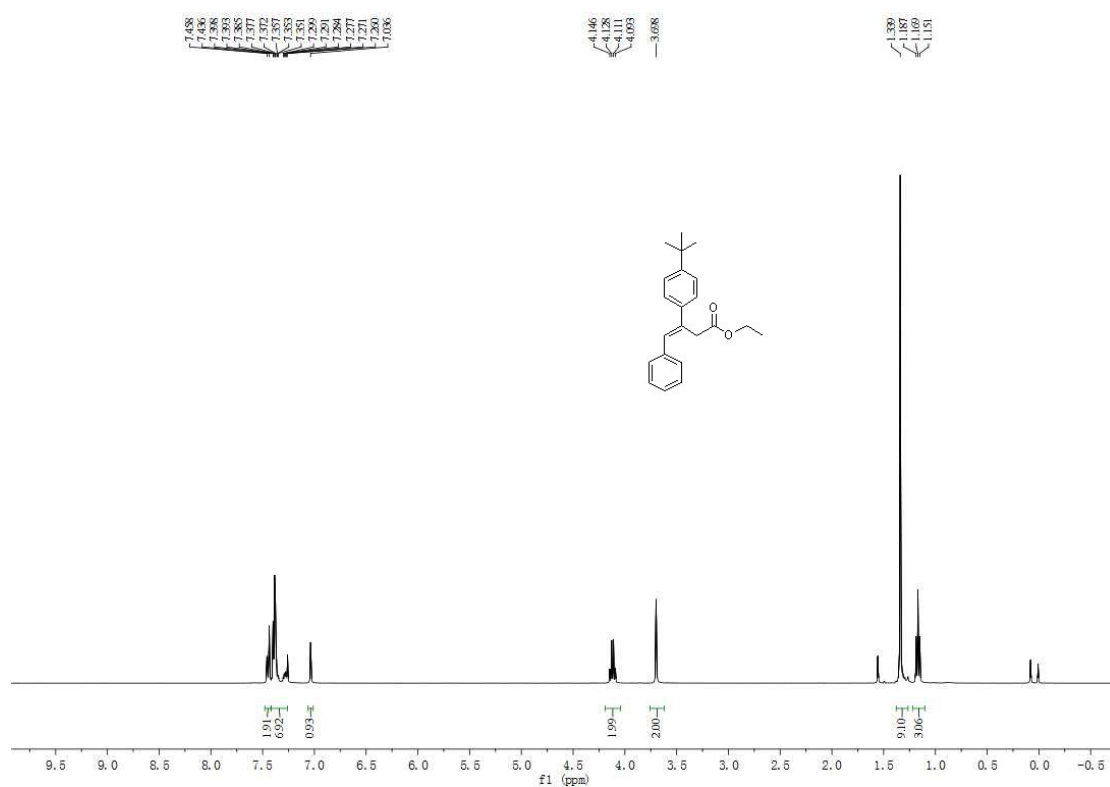
Product 5c



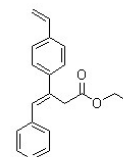
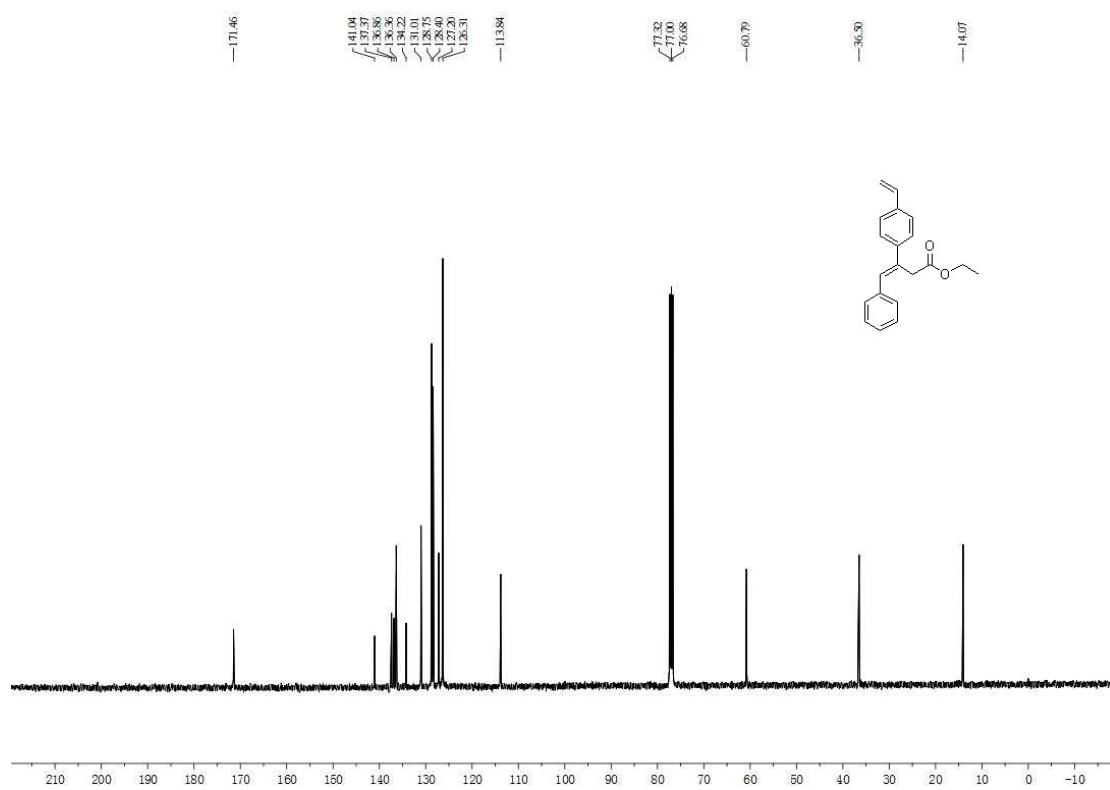
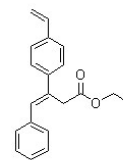
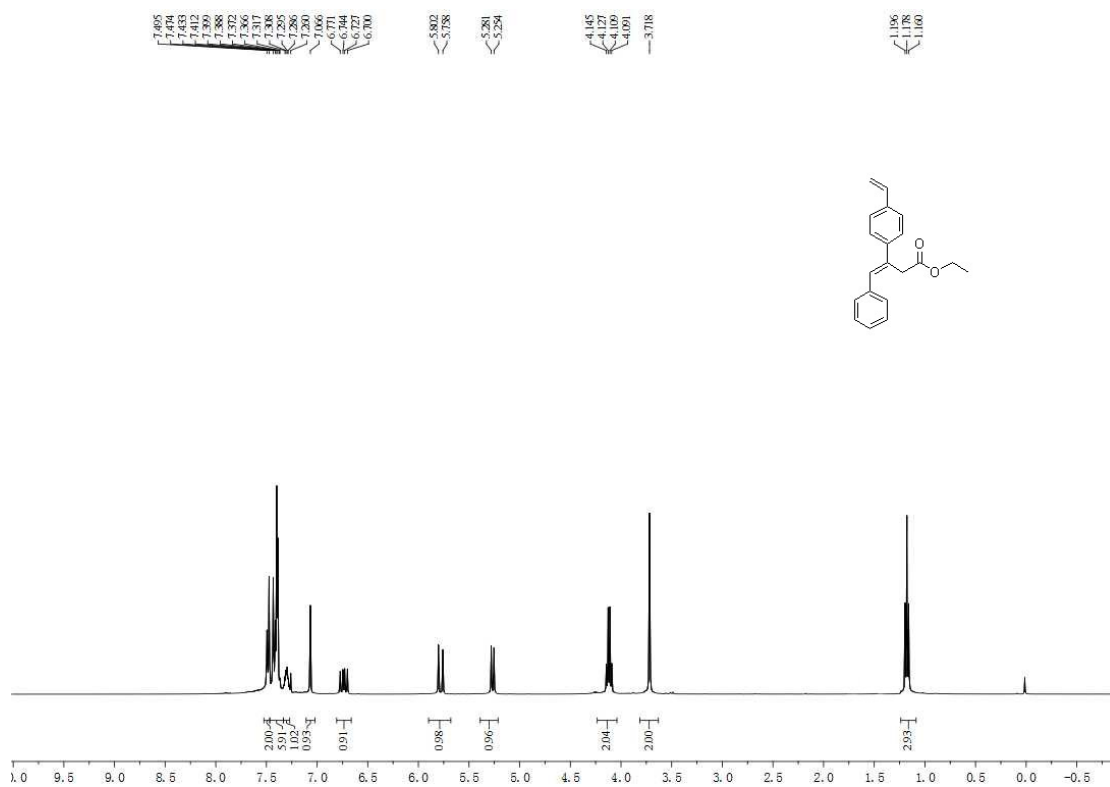
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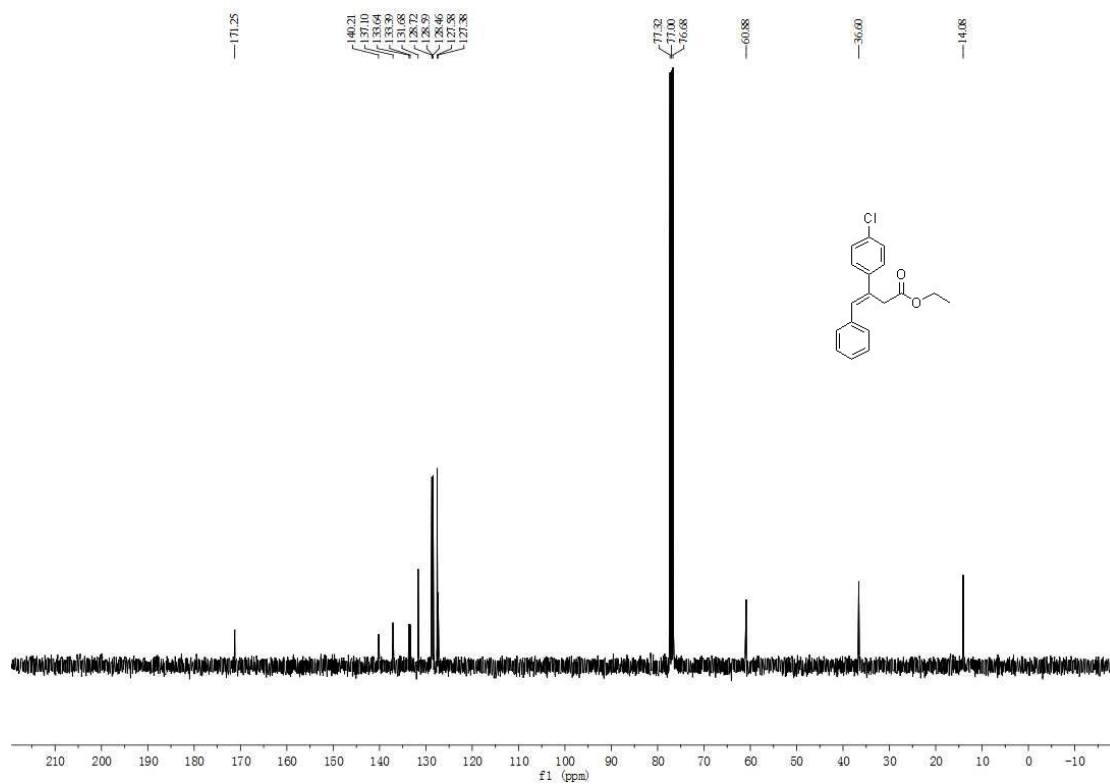
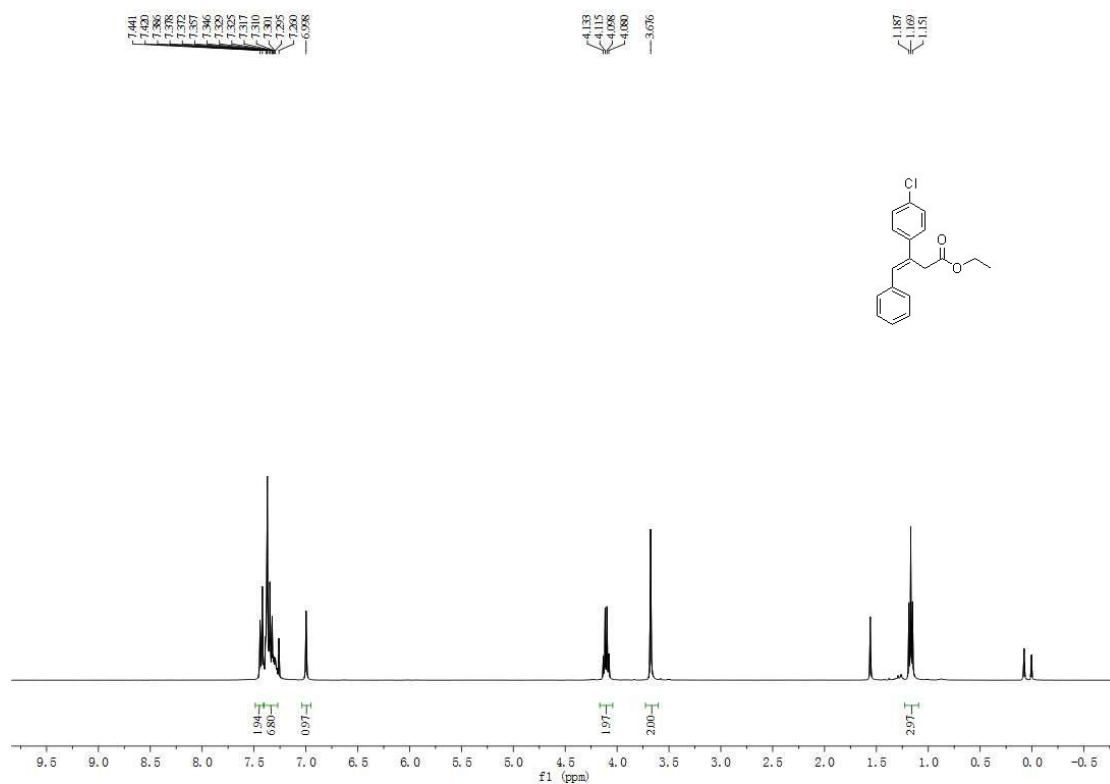
Product 5e



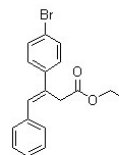
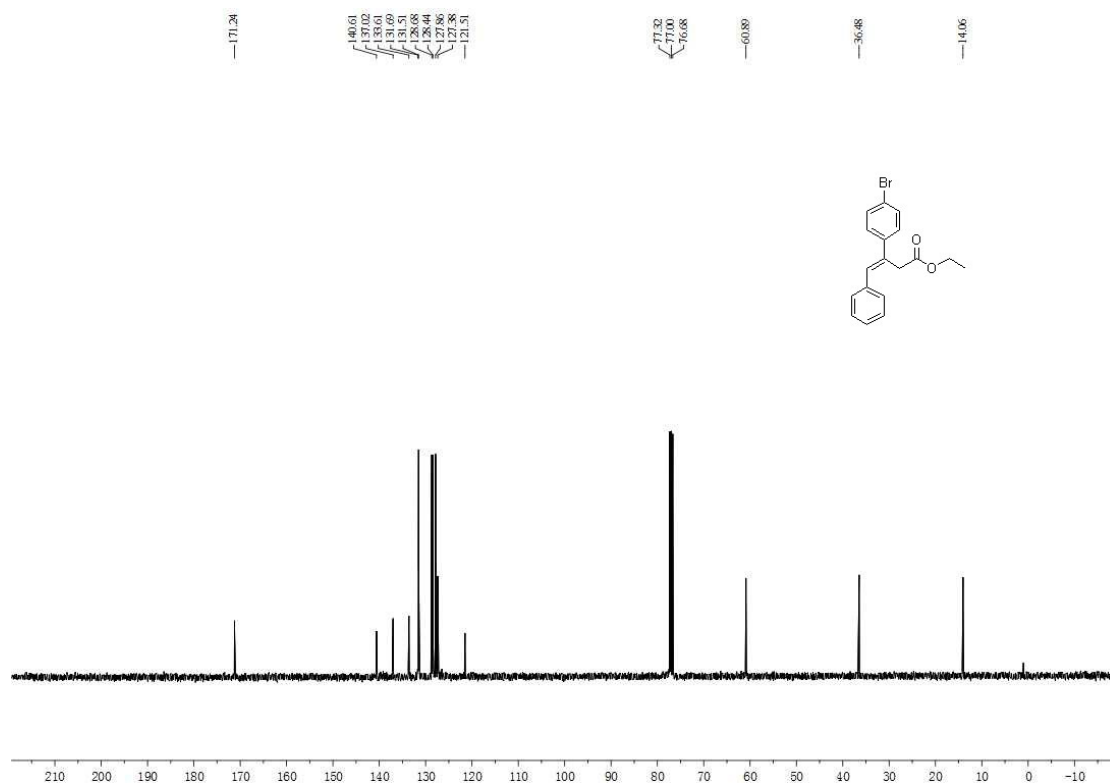
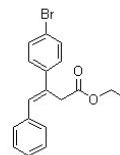
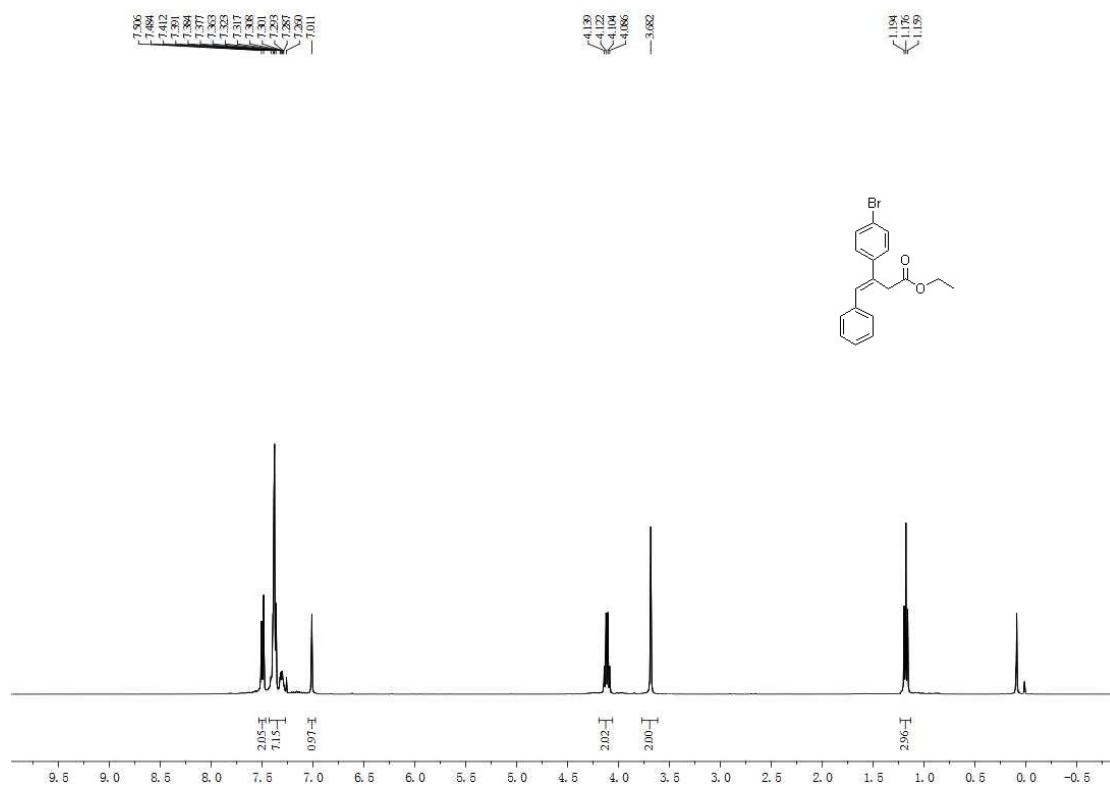
Product 5f



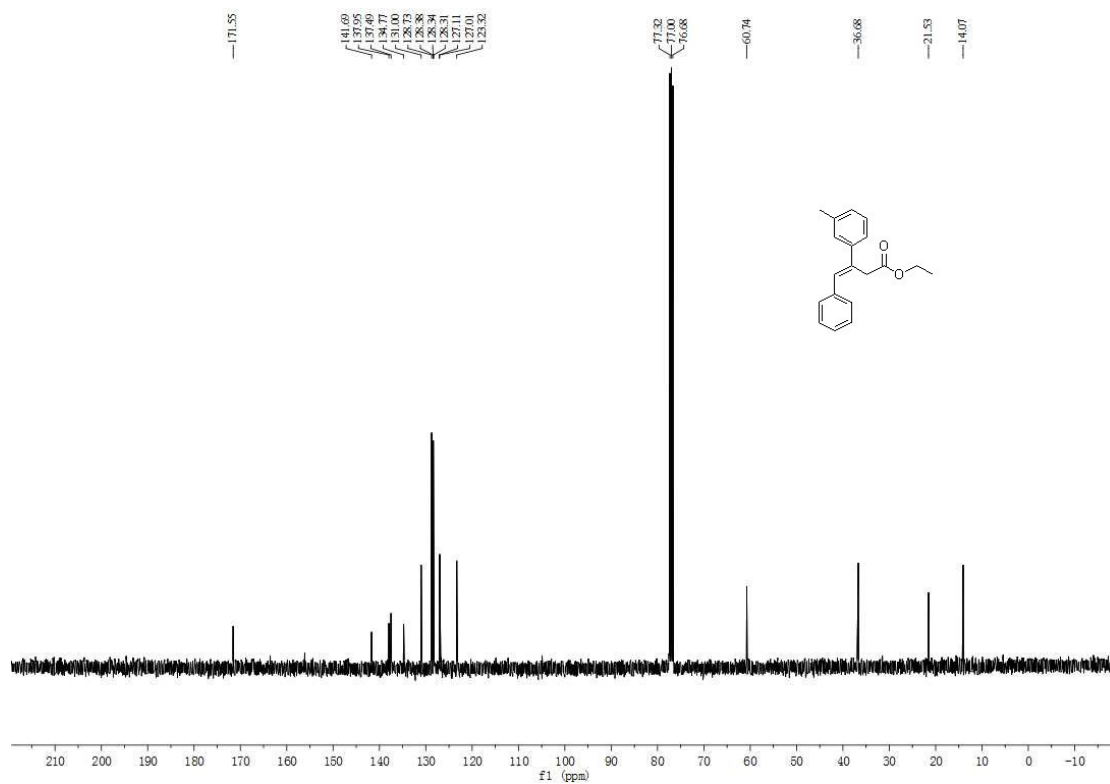
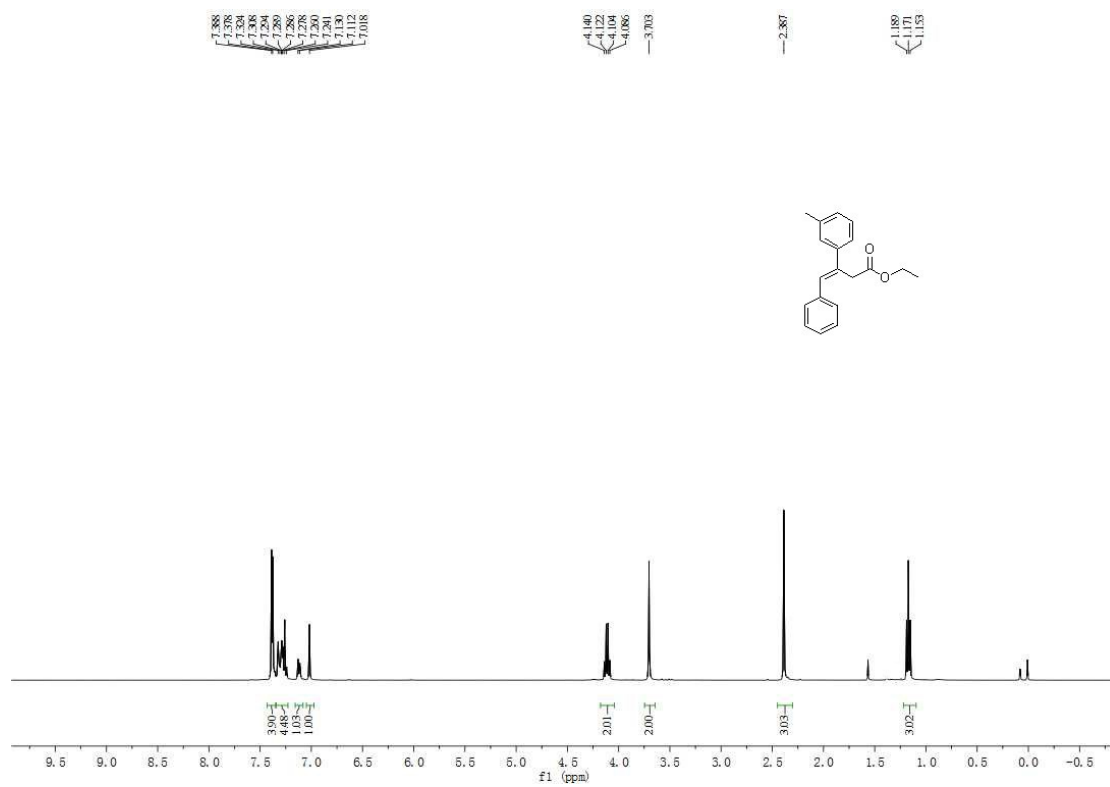
Product 5g



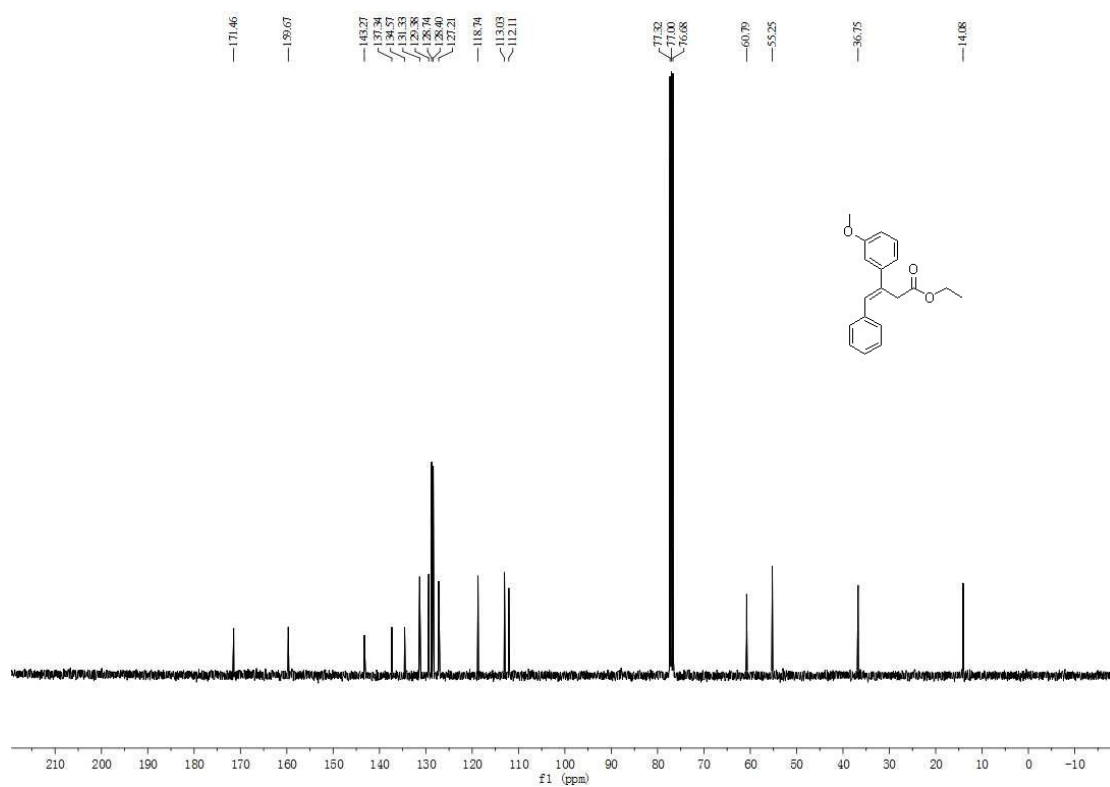
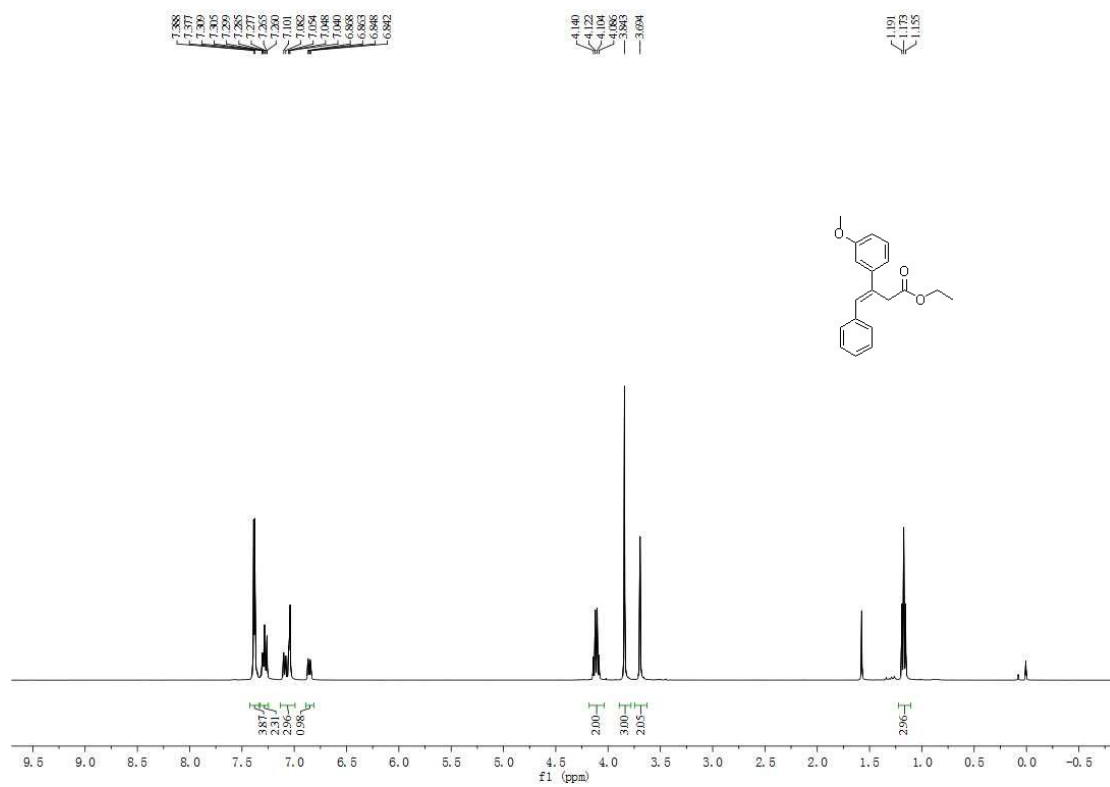
Product 5h



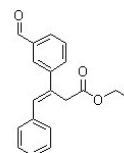
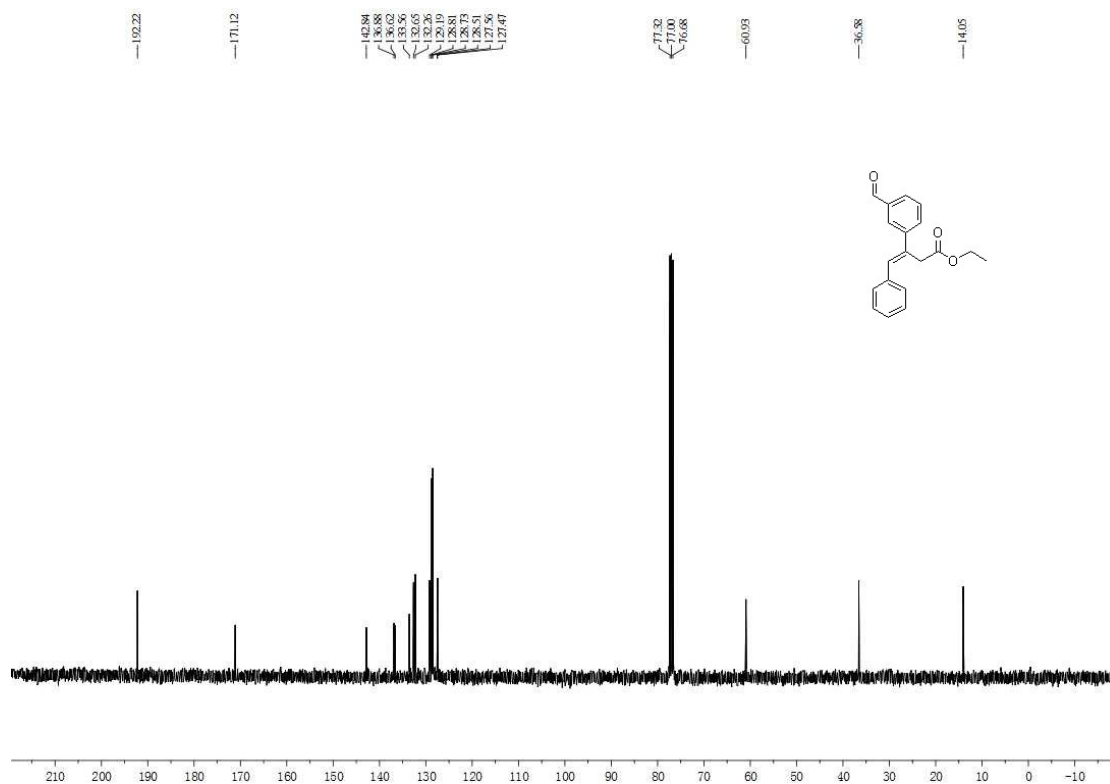
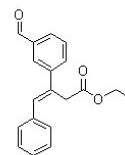
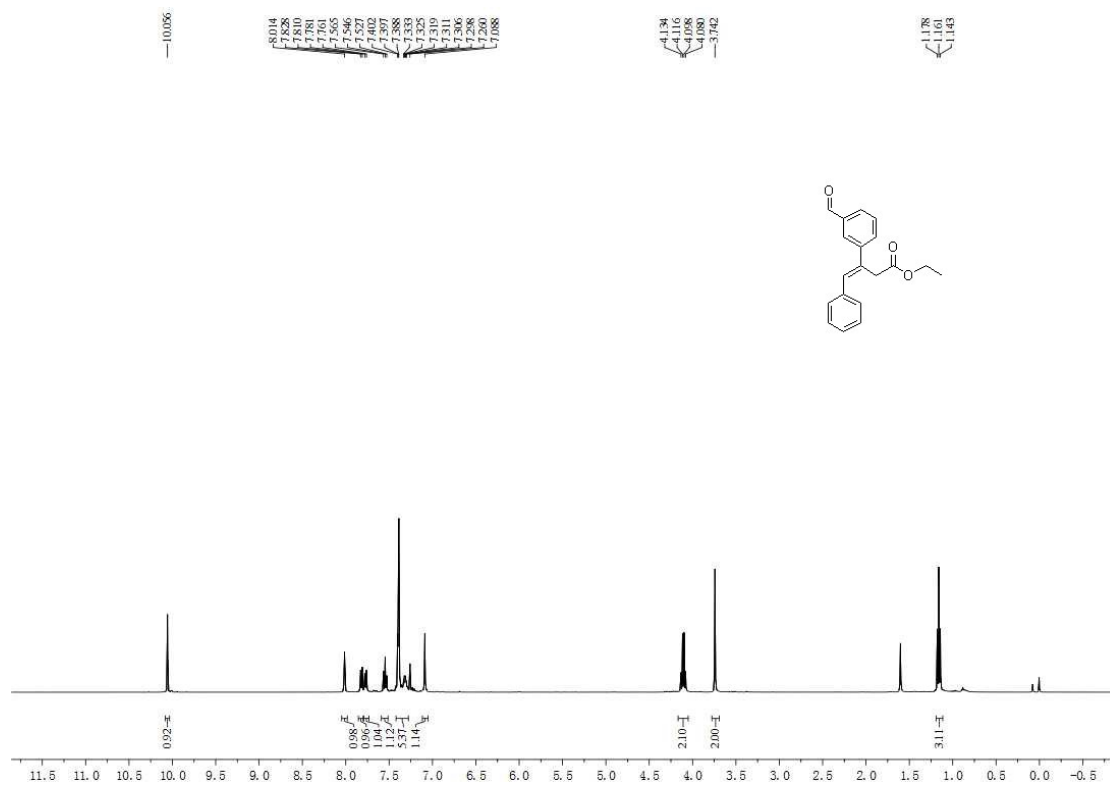
Product 5i



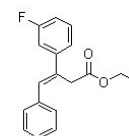
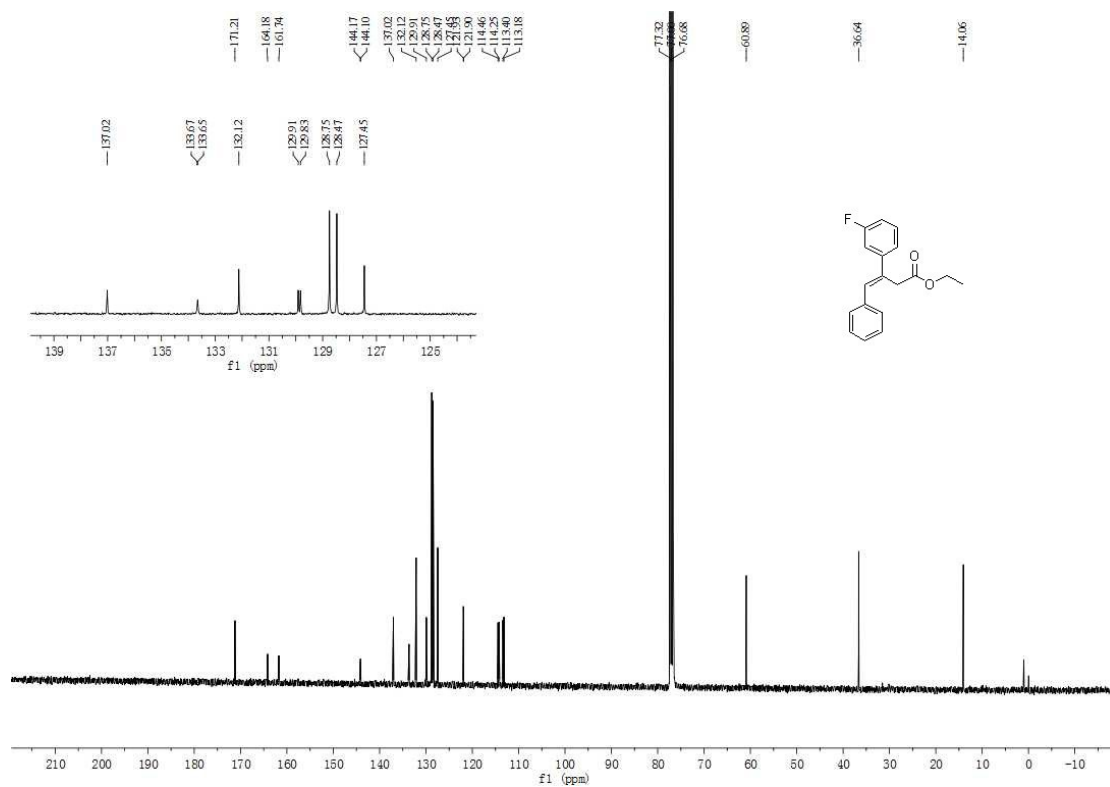
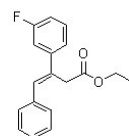
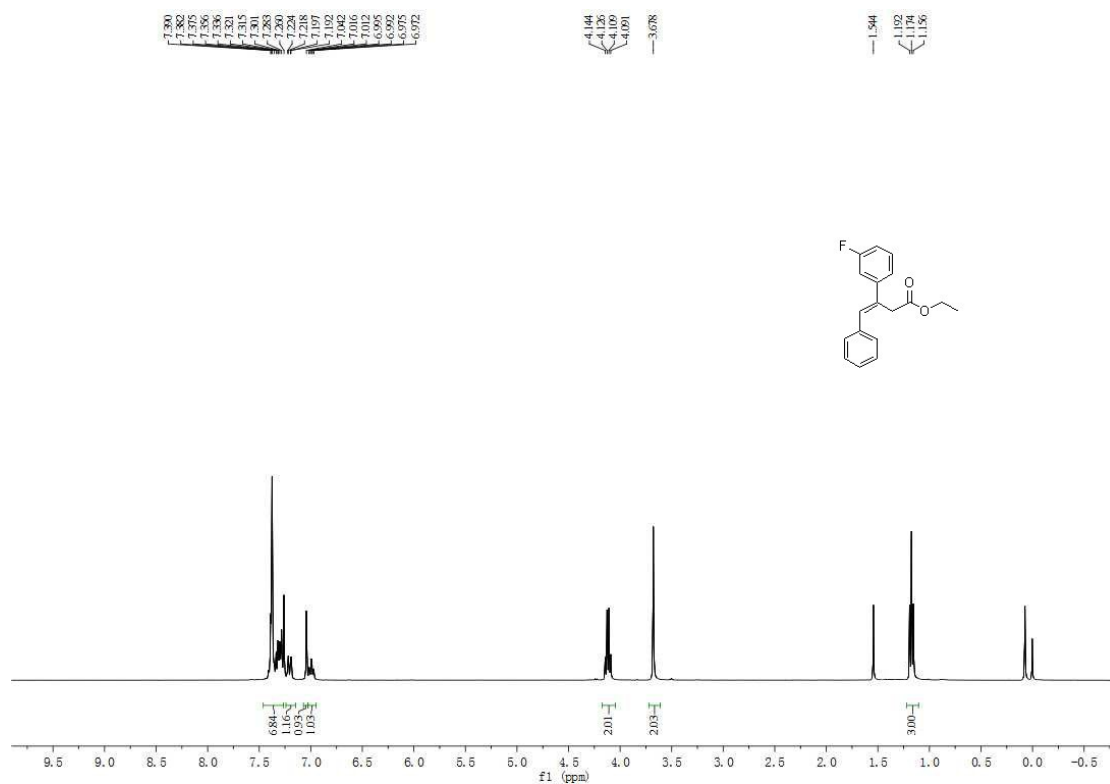
Product 5j



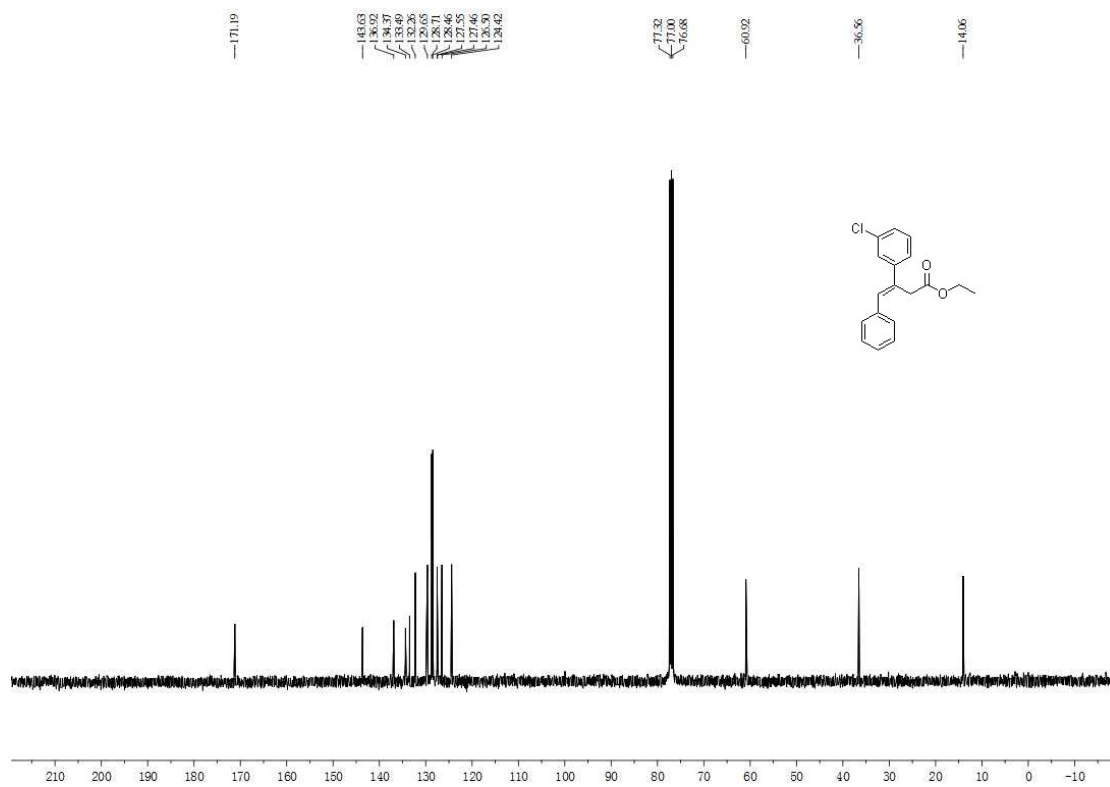
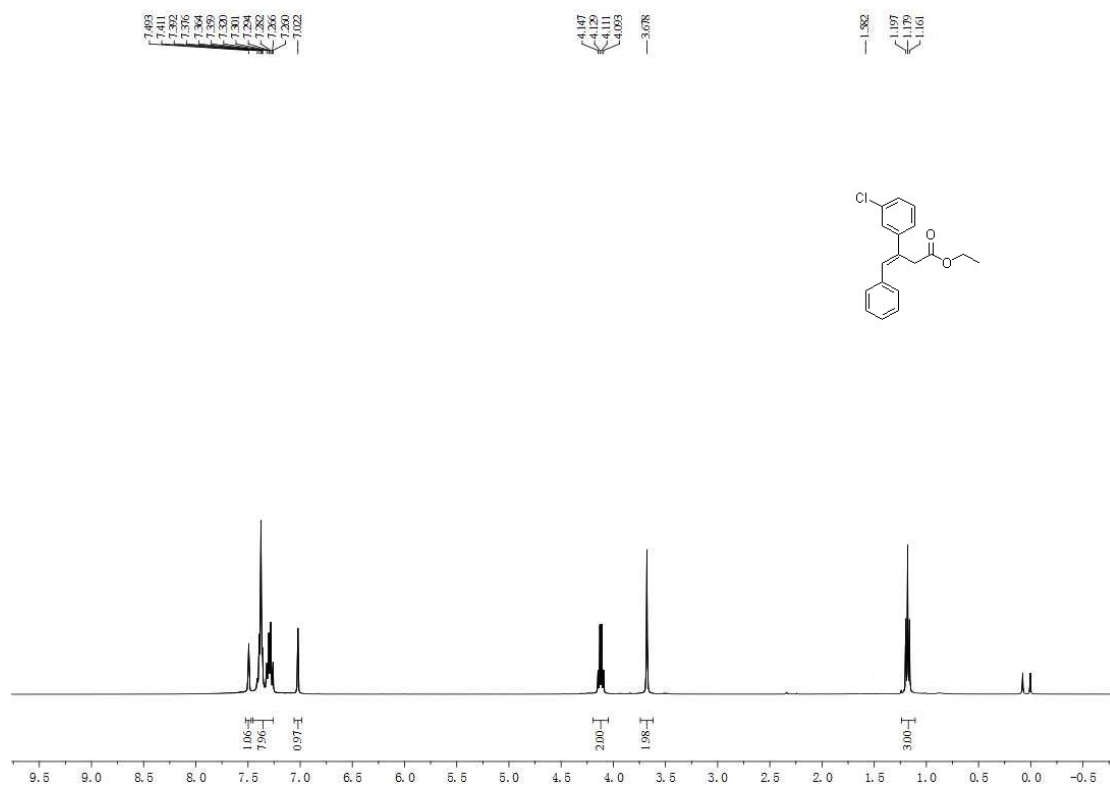
Product 5k



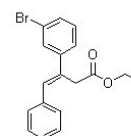
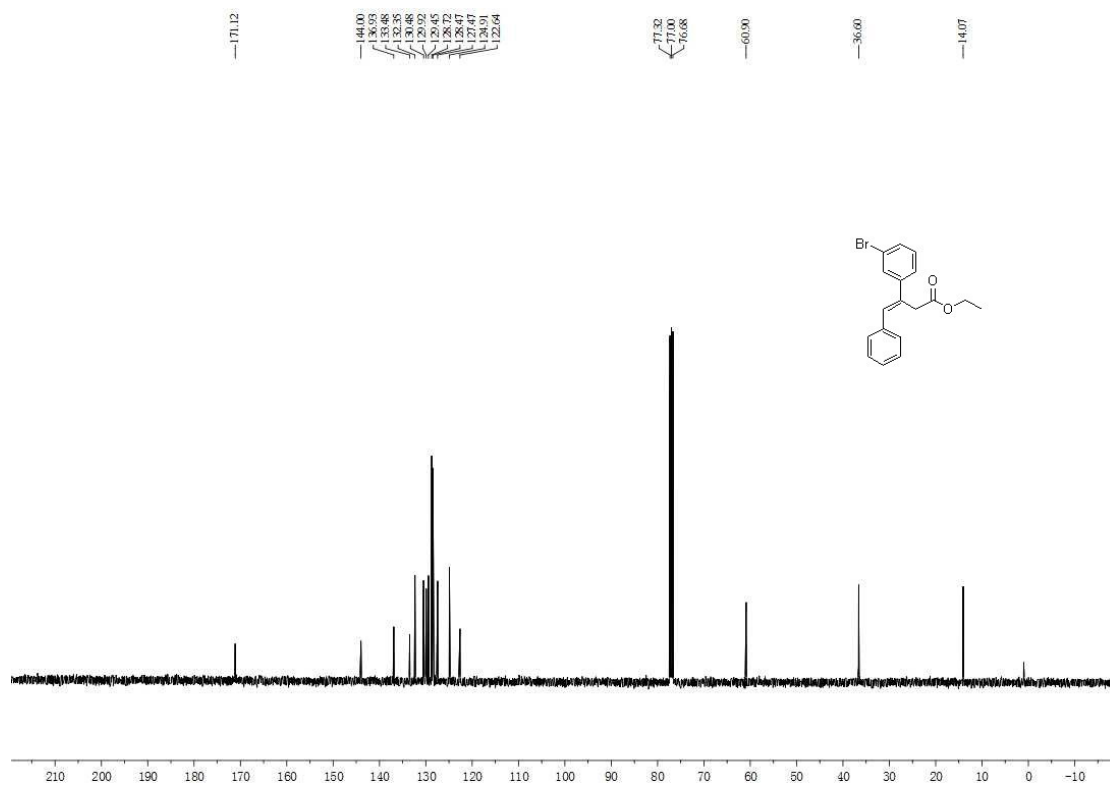
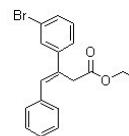
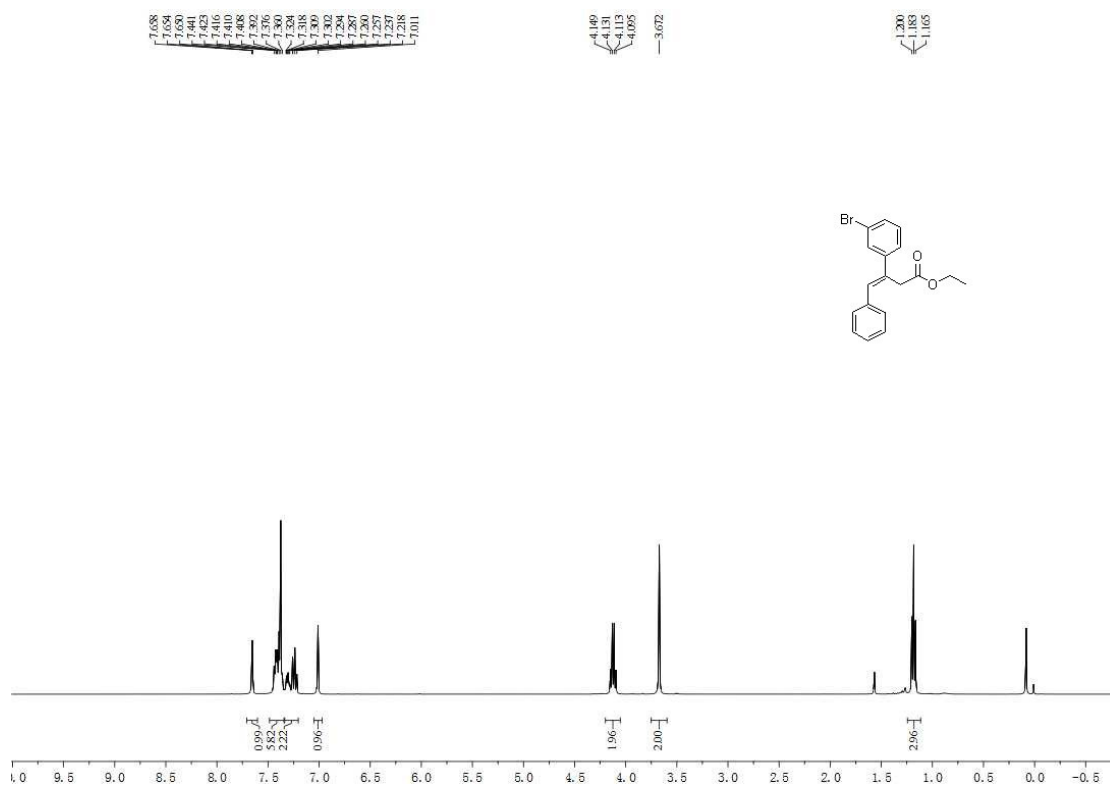
Product 5I



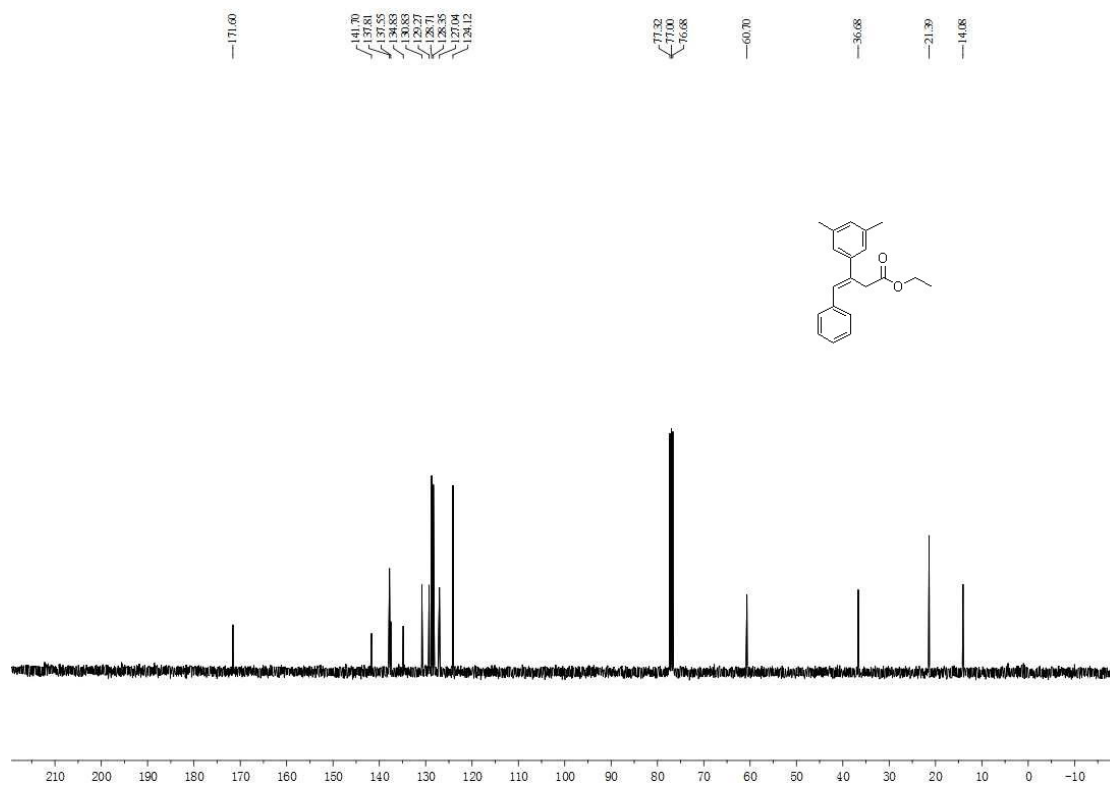
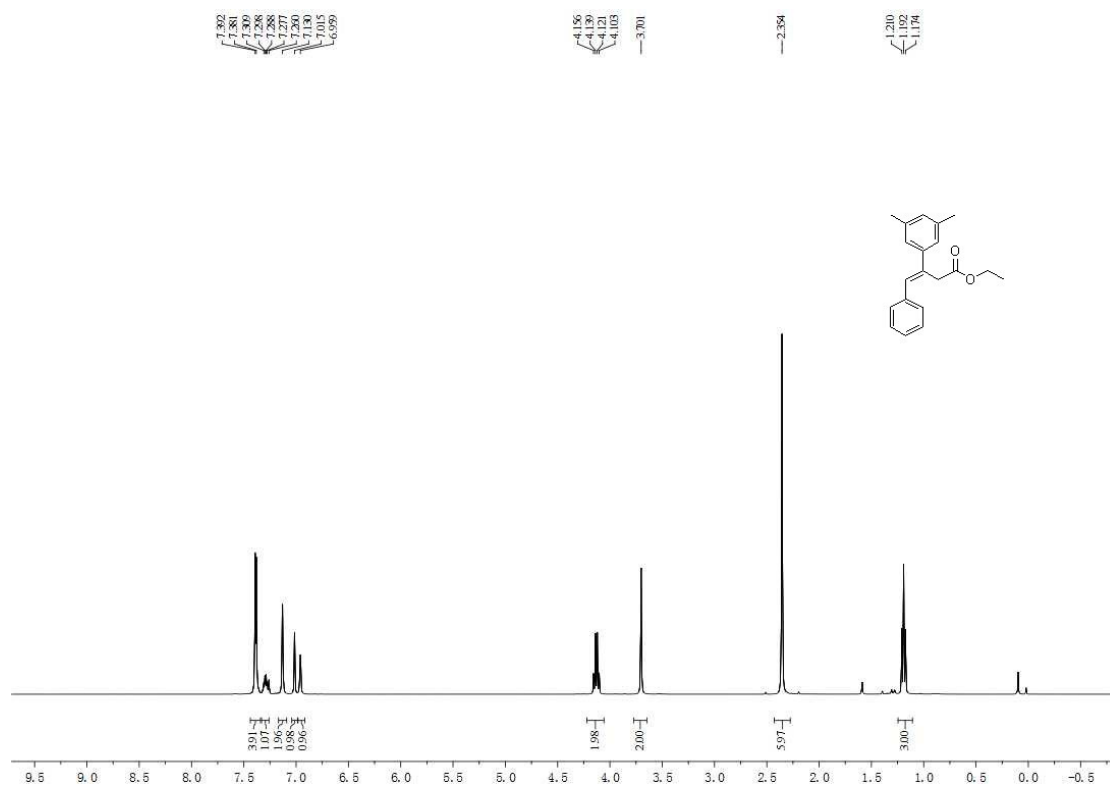
Product 5m



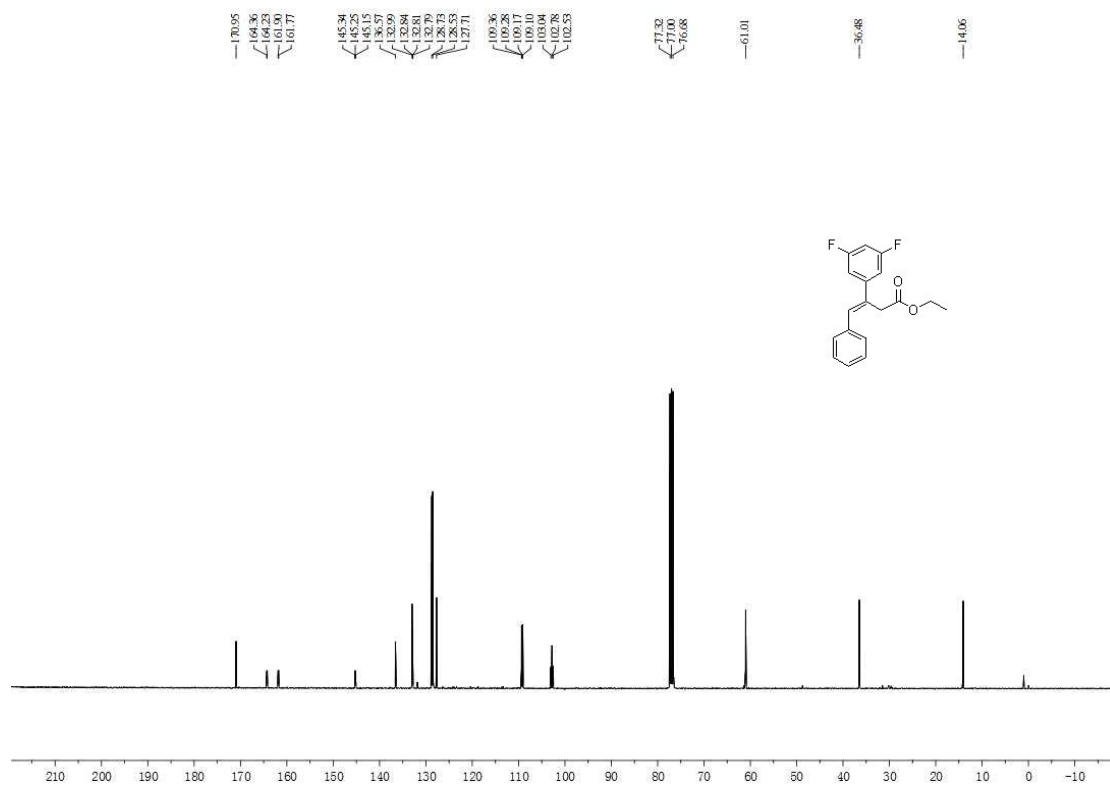
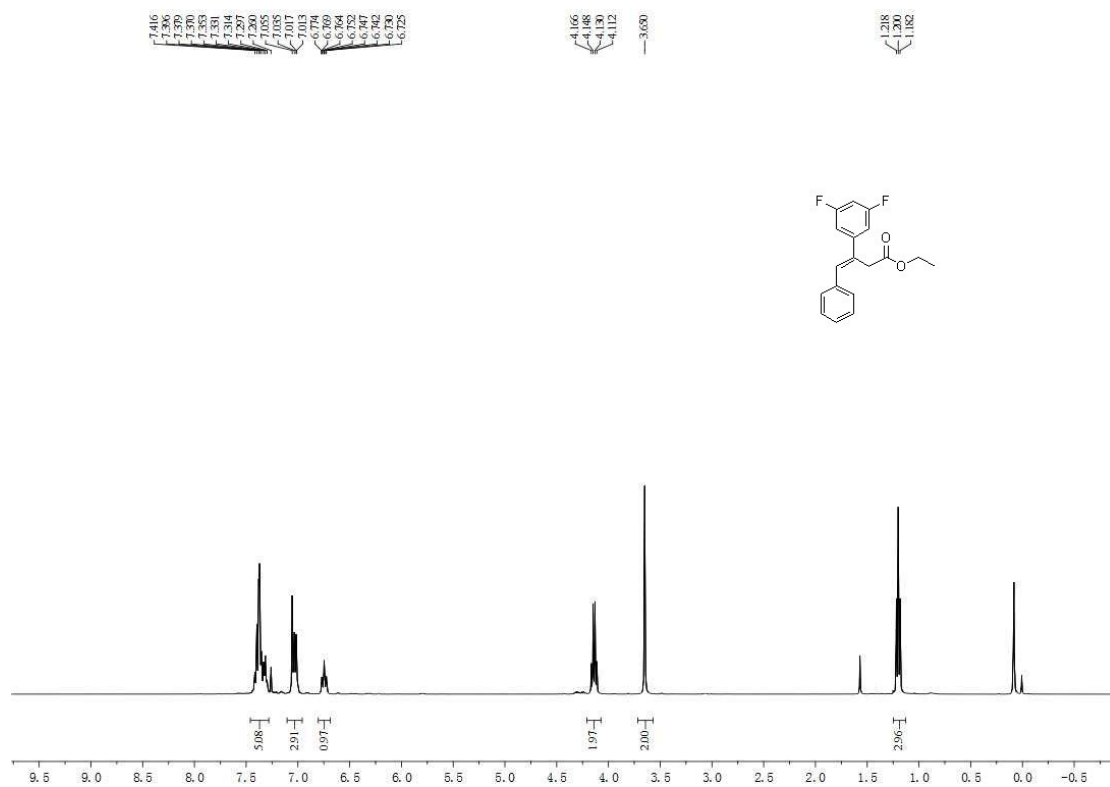
Product 5n



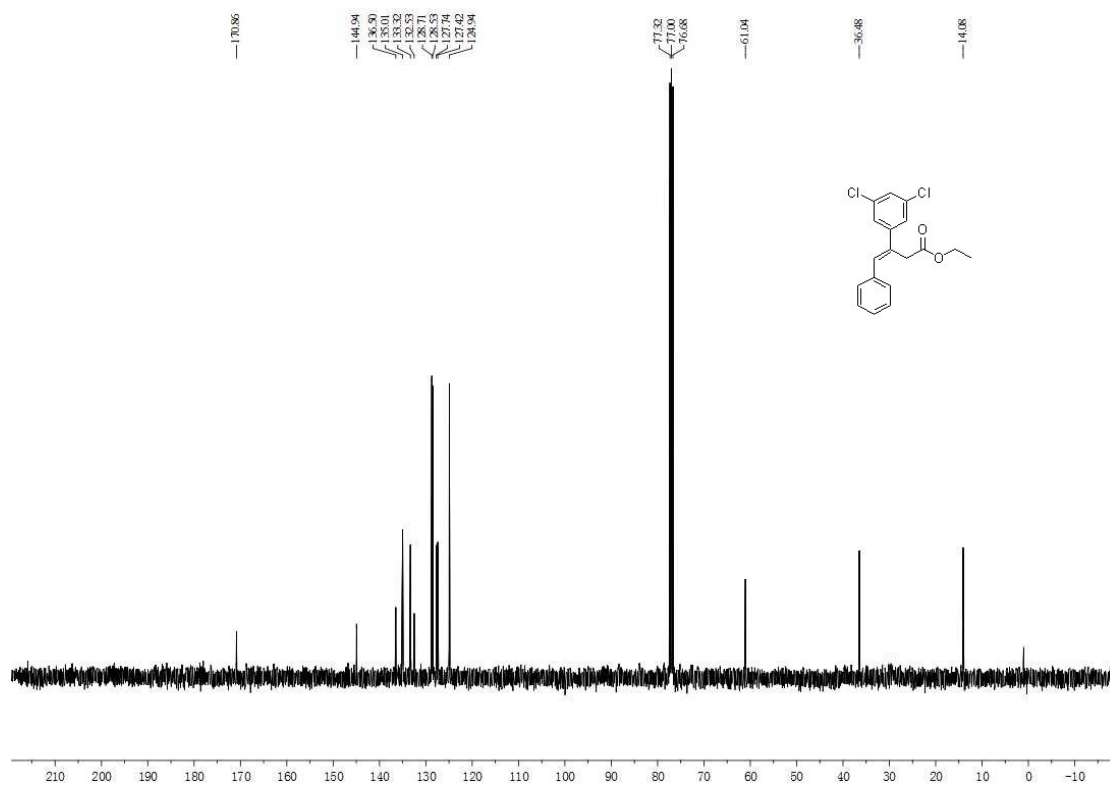
Product 5o



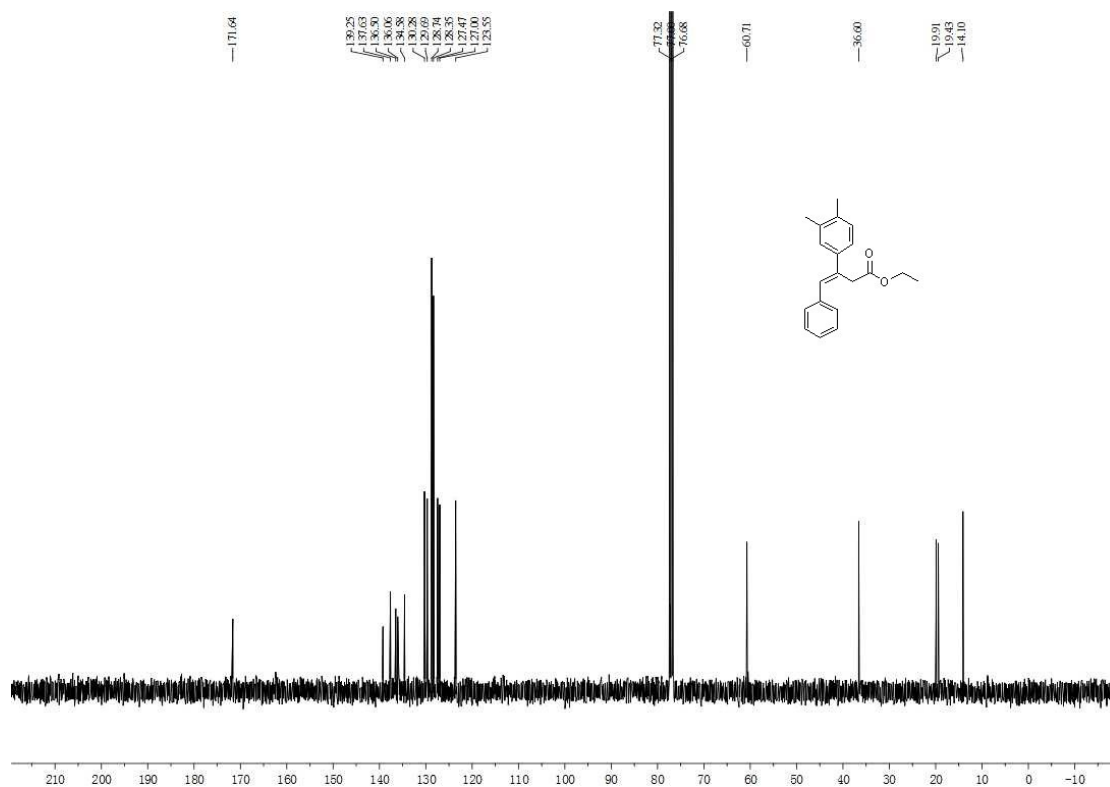
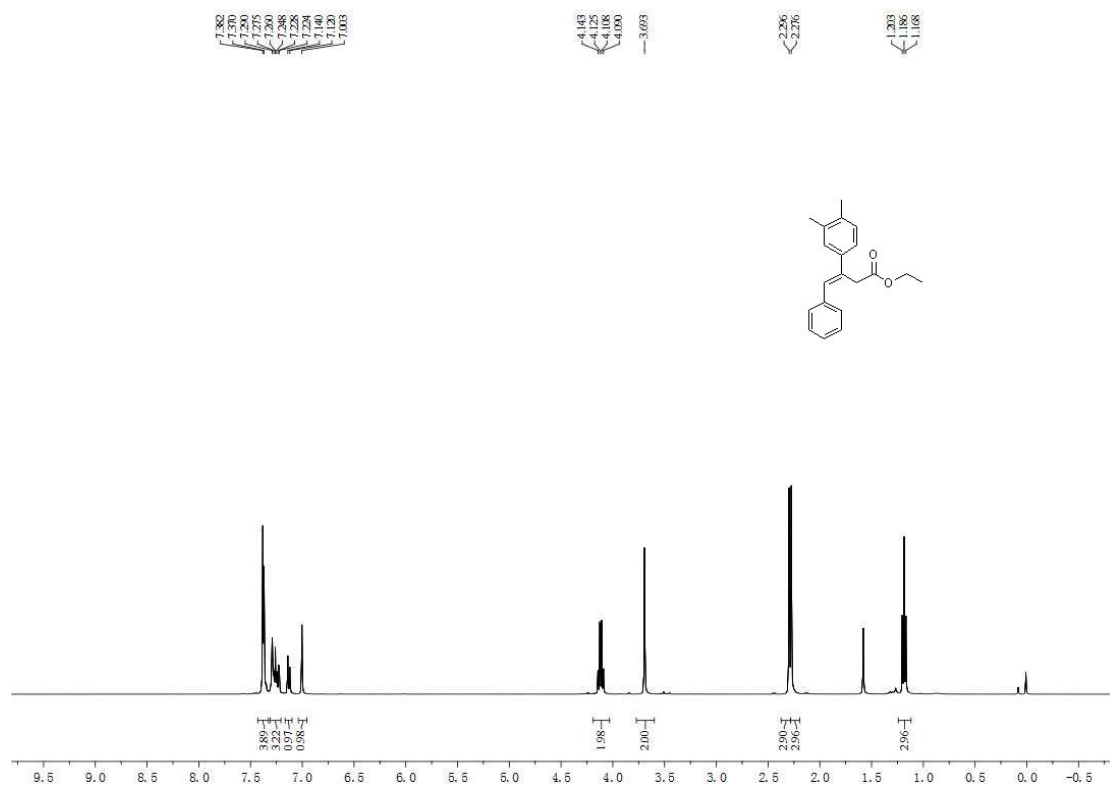
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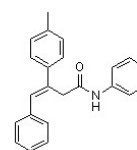
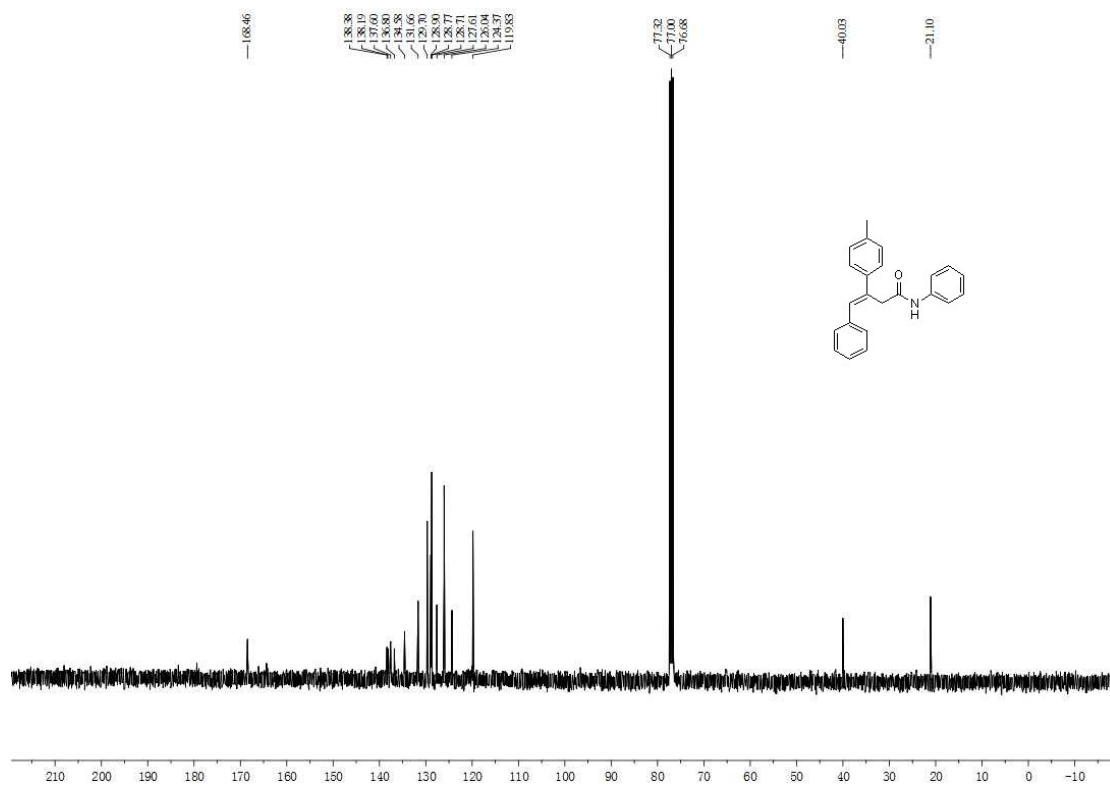
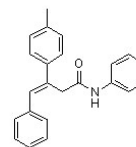
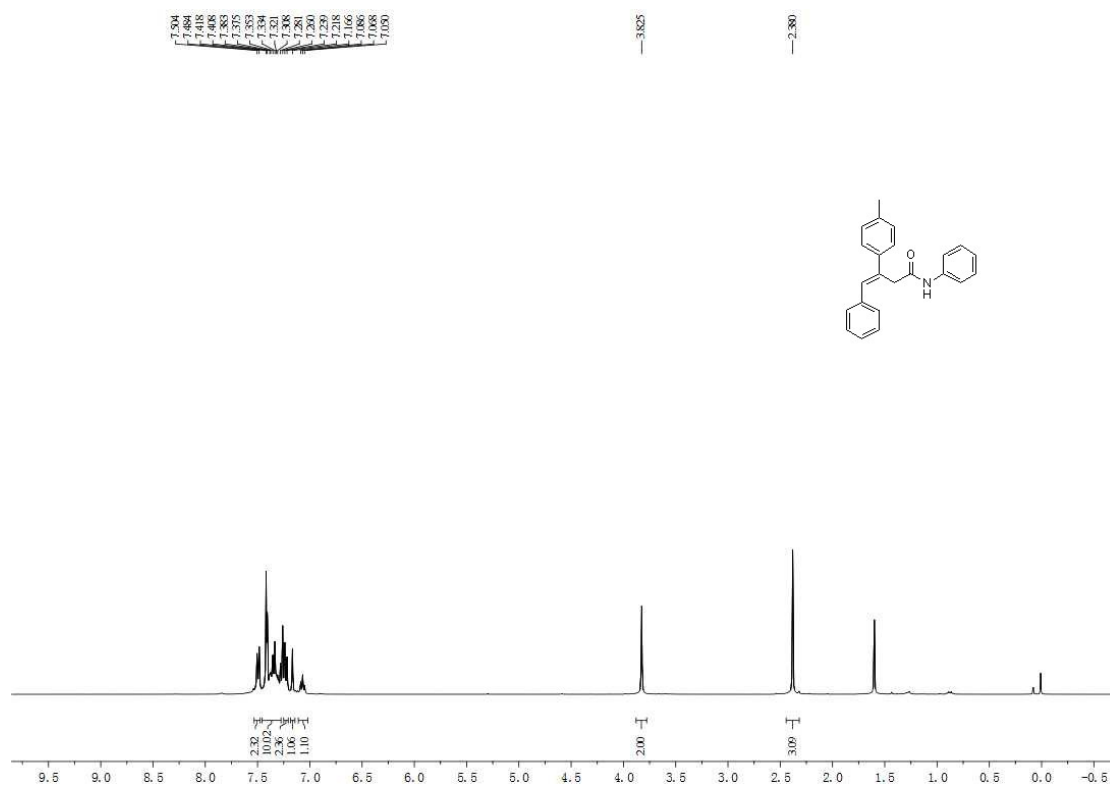
Product 5q



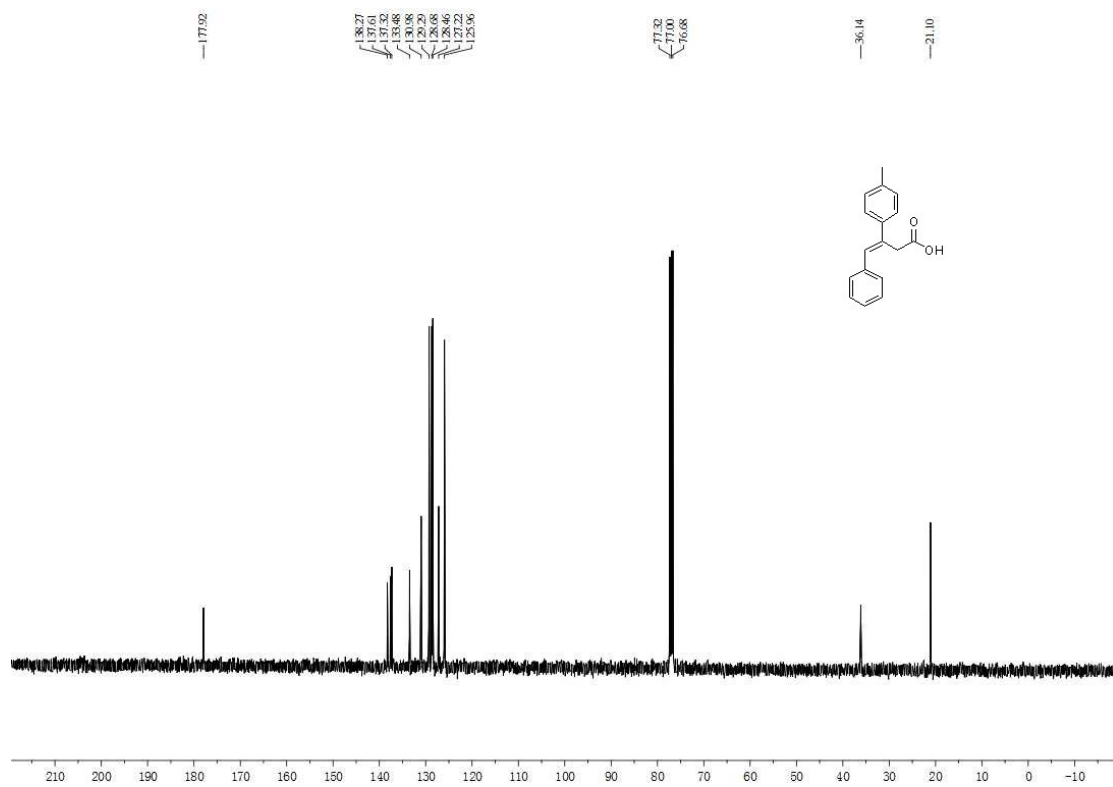
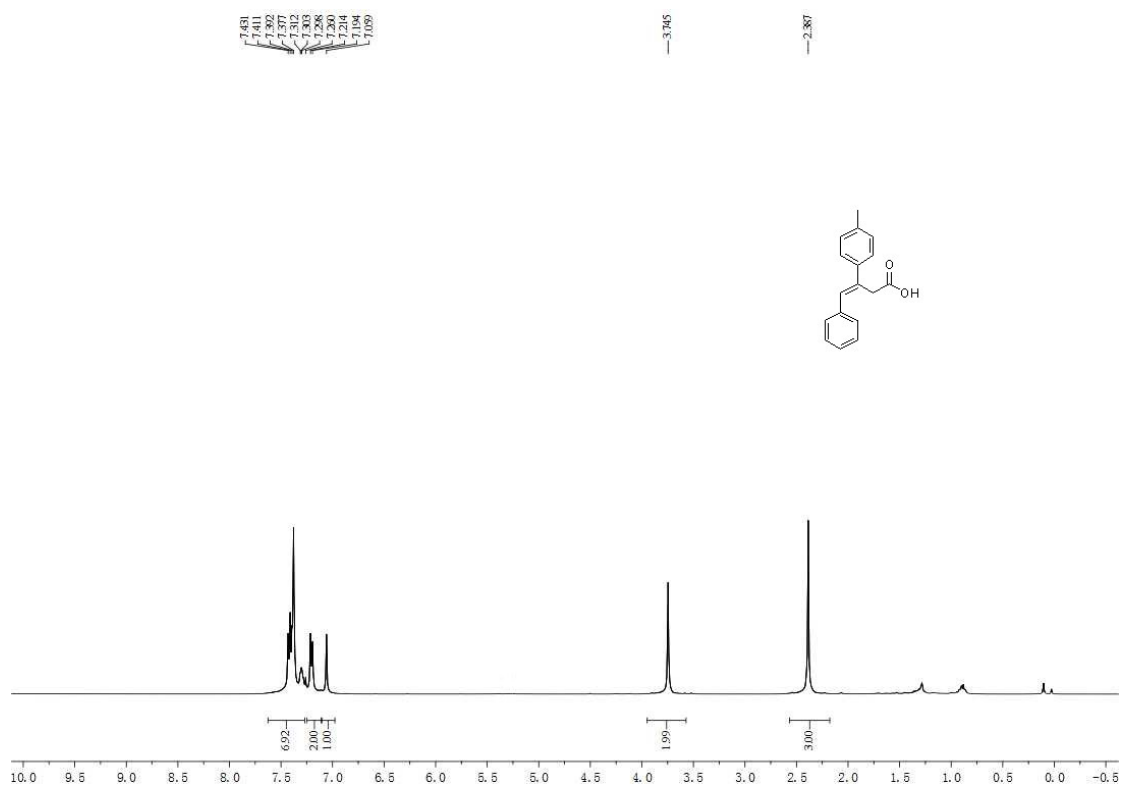
Product 5r



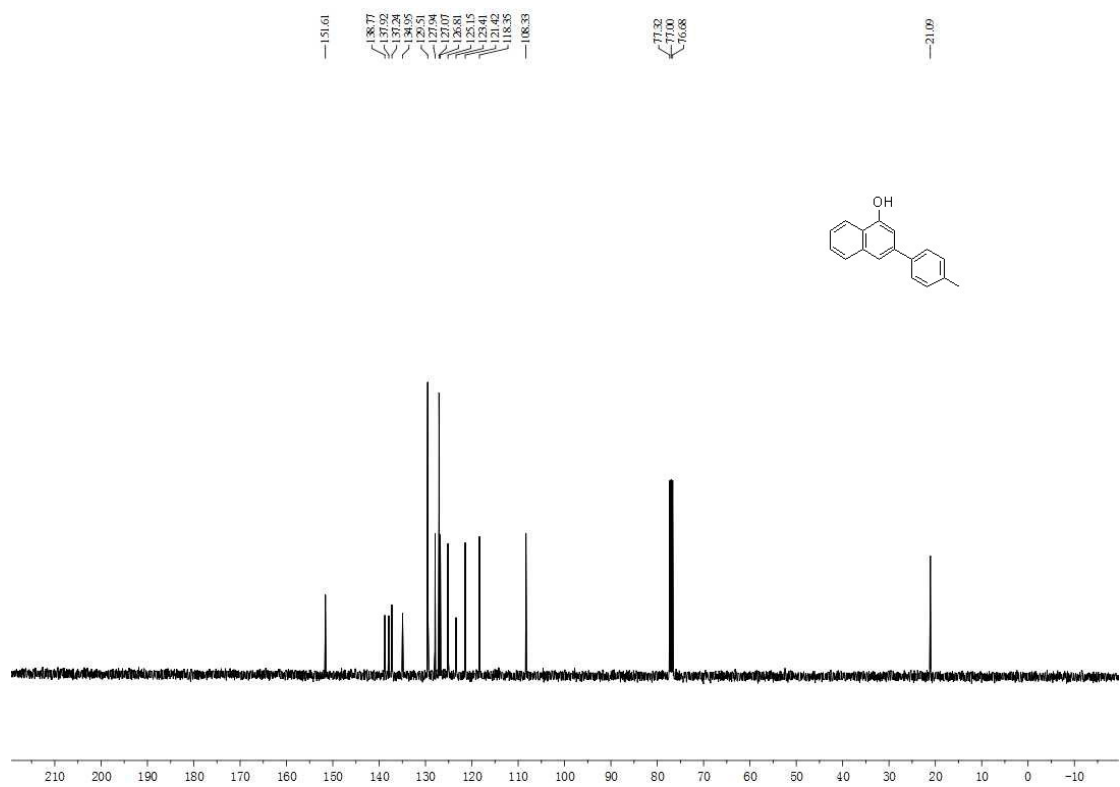
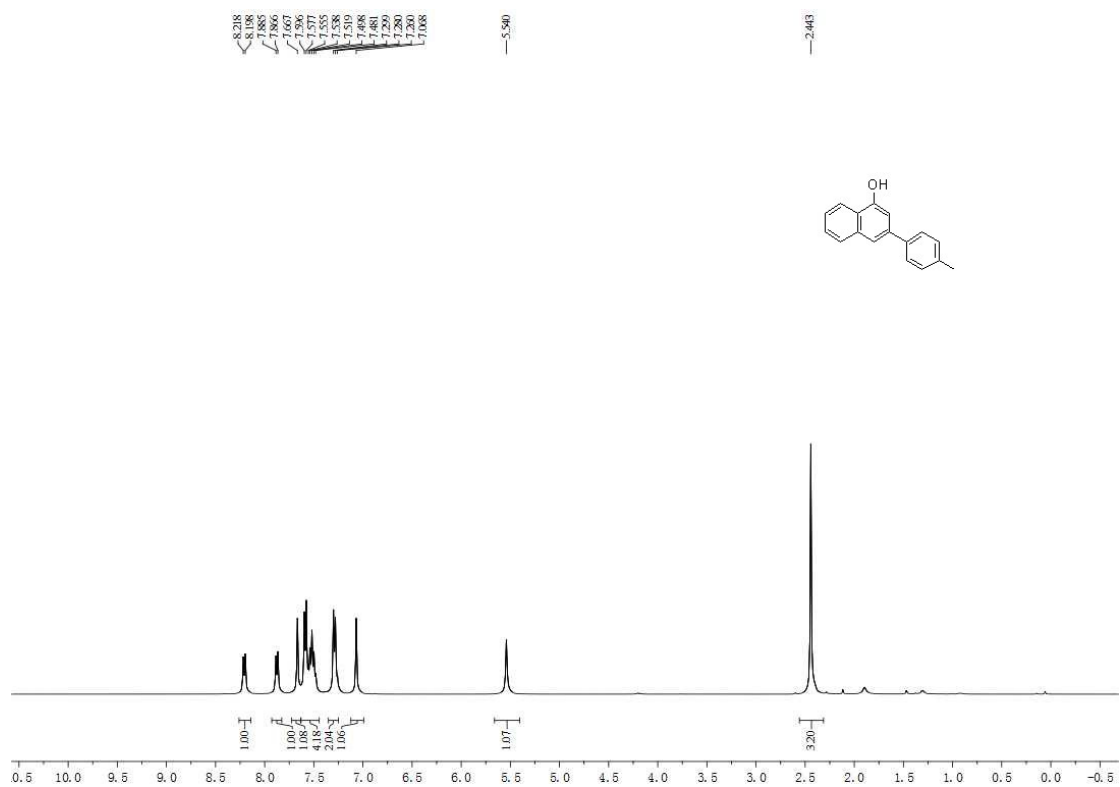
Product 5s



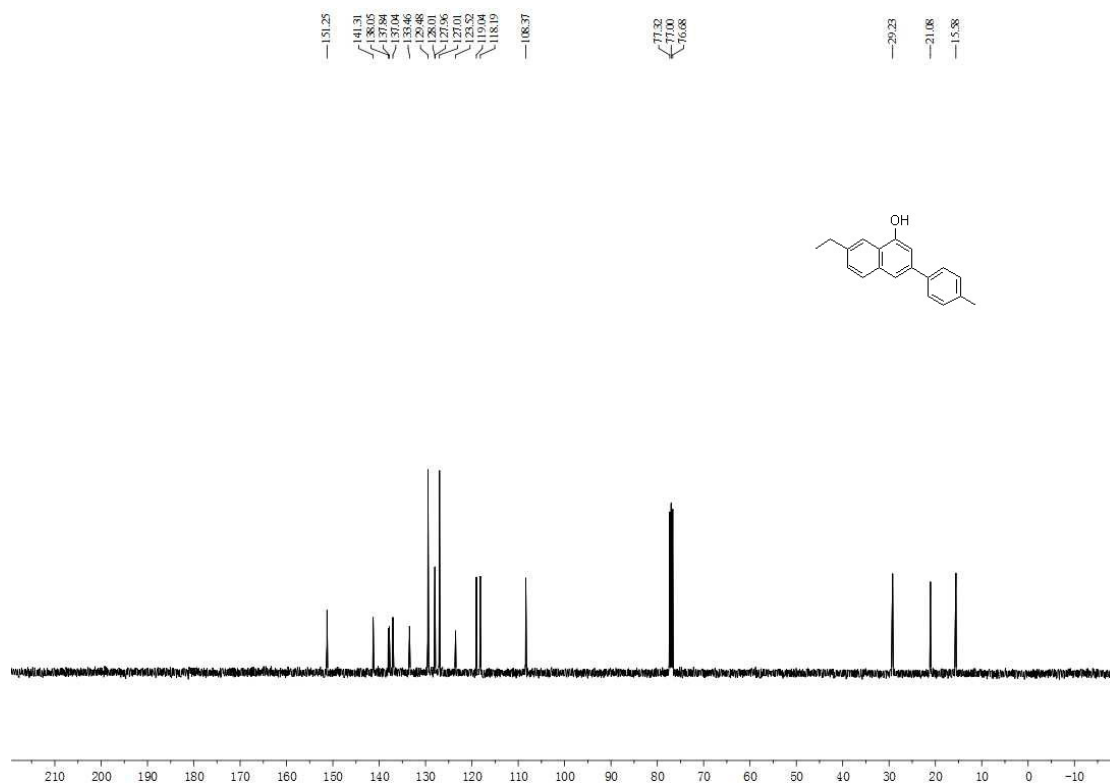
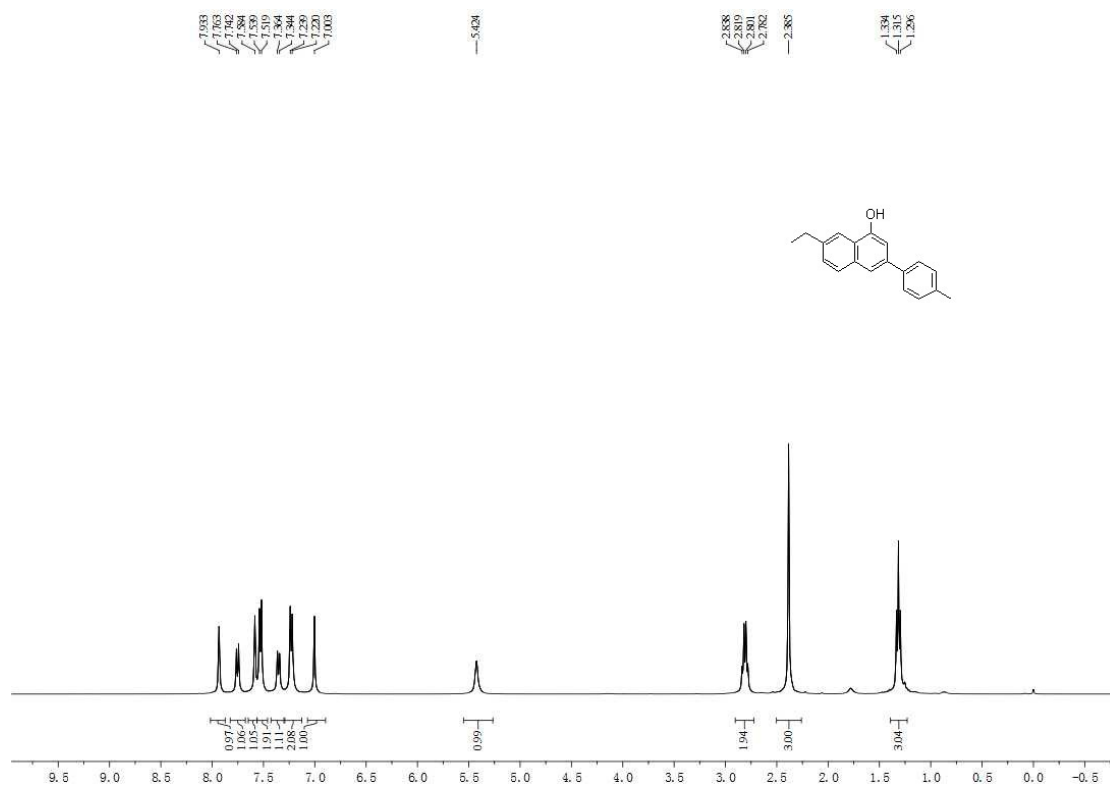
Product 6



Product 7a



Product 7b



Product 9

