

Supplementary Information
For

**Visible Light-Mediated Arylalkylation of Allylic Alcohols through
Concomitant 1,2-Aryl Migration**

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1. General information

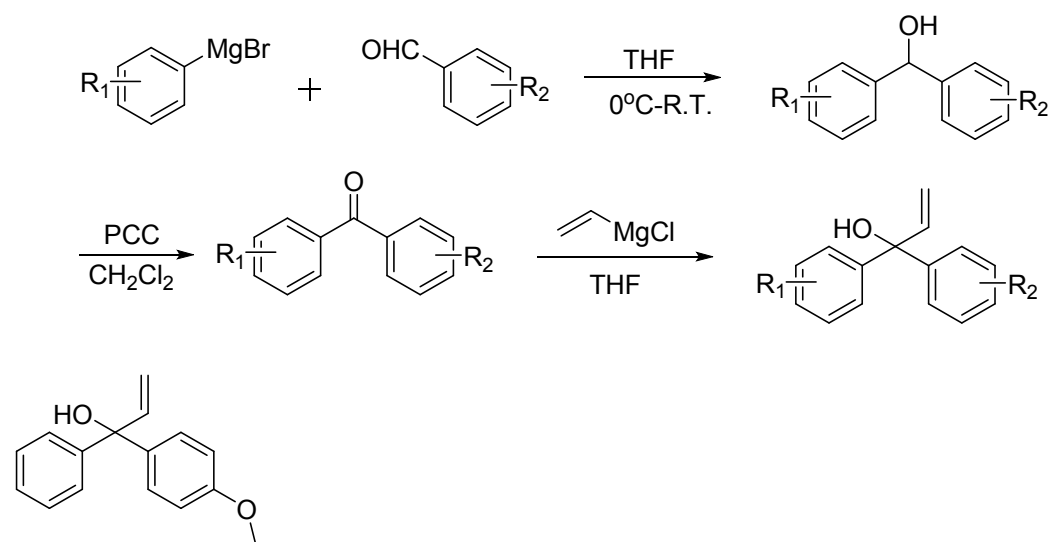
Unless otherwise noted, all reactions were carried out in round bottom flask under a nitrogen atmosphere with dry solvents, and were monitored by TLC and visualized by UV lamp (254 nm) /or by treatment with a solution of 10 g phosphomolybdic acid and 100 mL EtOH followed by heating. Silica gel (200-300 mesh) and silica gel GF₂₅₄ (10-40 μ m) were used for Column chromatography (CC) and prepare thin layer chromatography (PTLC), respectively. All reagents were used as received from commercial sources without further purification. Solvents were dried and purified according to the procedure from "Purification of Laboratory Chemicals book". ¹H (400 MHz) and ¹³C NMR (100 MHz) spectra were conducted on Bruker AV-400 instrument internally referenced to SiMe₄ or chloroform signals. The following abbreviations (or combinations thereof) were used to explain multiplicities: s = singlet, d = doublet, t = triplet, q = quartet, and m = multiplet. HRMS(ESI) spectra were recorded on a Bruker Esquire LC mass spectrometer using electrospray ionization. GC-MS analysis was performed on 7890A-5975C/Agilent.

2. Preparation and Characterization of Substrates

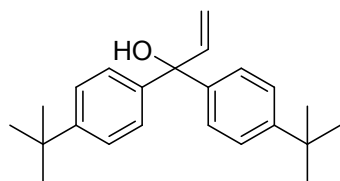
1) Synthesis of **1a**, **1d**, **1g**, **1h**, **1j-1r** and **1t**

Diaryl allylic alcohols **1a**, **1d**, **1g**, **1h**, **1j-1r** and **1t** were synthesized according to ref.

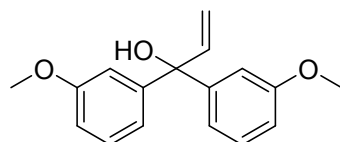
Angew. Chem., Int. Ed., 2013, **52**, 6962.¹



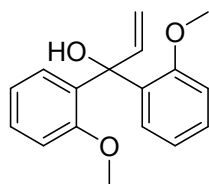
1-(4-Methoxyphenyl)-1-phenylprop-2-en-1-ol (1a)¹: Pale yellow oil. ¹H NMR (400 MHz, CDCl₃): δ_H 7.41 (d, J = 7.2 Hz, 2H), 7.34 (t, J = 8.0 Hz, 2H), 7.33-7.28 (m, 3H), 6.87 (d, J = 8.0 Hz, 2H), 6.50 (dd, J = 17.6, 10.0 Hz, 1H), 5.33 (dd, J = 17.6, 1.2 Hz, 1H), 5.32 (dd, J = 10.0, 1.2 Hz, 1H), 3.81 (s, OCH₃, 3H), 2.30 (s, 1H, -OH).



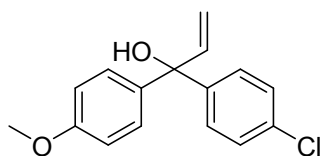
1,1-Bis(4-tert-butylphenyl)prop-2-en-1-ol (1d): Colorless oil. ^1H NMR (400 MHz, CDCl_3): δ_H 7.36 (d, $J = 8.8$ Hz, 4H), 7.32 (d, $J = 8.8$ Hz, 4H), 6.52 (dd, $J = 17.2, 10.8$ Hz, 1H), 5.36 (dd, $J = 17.2, 1.2$ Hz, 1H), 5.30 (dd, $J = 10.8, 1.2$ Hz, 1H), 2.26 (s, 1H, -OH), 1.33 (s, $6 \times \text{CH}_3$, 18H); ^{13}C NMR (100 MHz, CDCl_3): δ_c 150.0, 143.8, 142.8, 126.5, 125.0, 113.3, 79.0, 34.4, 31.3; GC-MS (EI): 322.2, 307.2, 265.1, 161.1, 131.1; HRMS(ESI): $[\text{M} - \text{H}_2\text{O} + \text{H}]^+$ calcd for $\text{C}_{20}\text{H}_{29}^+$: 305.2269, found: 305.2286.



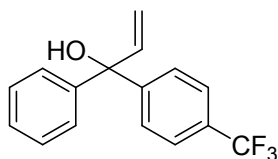
1-(3-Methoxyphenyl)-1-phenylprop-2-en-1-ol (1g): Pale yellow oil. ^1H NMR (400 MHz, CDCl_3): δ_H 7.38 (d, $J = 7.6$ Hz, 2H), 7.29 (t, $J = 7.6$ Hz, 2H), 7.23 (d, $J = 7.6$ Hz, 1H), 7.19 (d, $J = 7.6$ Hz, 1H), 6.99 (d, $J = 1.6$ Hz, 1H), 6.93 (dd, $J = 7.6, 0.8$ Hz, 1H), 6.77 (dd, $J = 7.6, 1.6$ Hz, 1H), 6.48 (dd, $J = 17.2, 10.8$ Hz, 1H), 5.29 (dd, $J = 17.2, 1.2$ Hz, 1H), 5.27 (dd, $J = 10.8, 1.2$ Hz, 1H), 3.75 (s, OCH_3 , 3H), 2.43 (s, 1H, -OH); ^{13}C NMR (100 MHz, CDCl_3): δ_c 159.0, 147.6, 145.8, 143.4, 128.7, 127.7, 126.8, 126.7, 119.3, 113.6, 112.6, 112.0, 78.7, 54.9; GC-MS (EI): 270.1, 135.1, 107.1, 77.1; HRMS(ESI): $[\text{M} - \text{H}_2\text{O} + \text{H}]^+$ calcd for $\text{C}_{17}\text{H}_{17}\text{O}_2^+$: 253.1228, found: 253.1231.



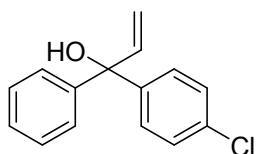
1,1-Bis(2-methoxyphenyl)prop-2-en-1-ol (1h): Colorless oil. ^1H NMR (400 MHz, CDCl_3): δ_H 7.48 (dd, $J = 8.0, 1.6$ Hz, 2H), 7.22 (t, $J = 8.0$ Hz, 2H), 6.95 (t, $J = 8.0$ Hz, 2H), 6.83 (d, $J = 8.0$ Hz, 2H), 6.66 (dd, $J = 17.2, 10.6$ Hz, 1H), 5.36 (dd, $J = 17.2, 1.4$ Hz, 1H), 5.23 (dd, $J = 10.6, 1.4$ Hz, 1H), 4.75 (s, 1H, -OH), 3.53 (s, $2 \times \text{OCH}_3$, 6H).



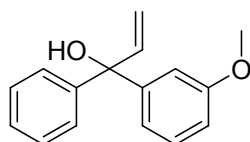
1-(4-Chlorophenyl)-1-(4-methoxyphenyl)prop-2-en-1-ol (1j)²: Colorless oil. ¹H NMR (400 MHz, CDCl₃): δ_H 7.31-7.28 (m, 4H), 7.25 (d, J = 8.6 Hz, 2H), 6.84 (d, J = 8.6 Hz, 2H), 6.42 (dd, J = 17.2, 10.4 Hz, 1H), 5.29 (d, J = 10.4 Hz, 1H), 5.28 (d, J = 17.2 Hz, 1H), 3.78 (s, OCH₃, 3H), 2.30 (br s, 1H, -OH).



1-Phenyl-1-(4-(trifluoromethyl)phenyl)prop-2-en-1-ol (1k)¹: Colorless oil. ¹H NMR (400 MHz, CDCl₃): δ_H 7.55 (d, J = 7.6 Hz, 2H), 7.48 (d, J = 8.4 Hz, 2H), 7.37-7.24 (m, 5H), 6.46 (dd, J = 17.2, 10.8 Hz, 1H), 5.33 (dd, J = 10.8, 0.8 Hz, 1H), 5.30 (dd, J = 17.2, 0.8 Hz, 1H), 2.44 (s, 1H, -OH).

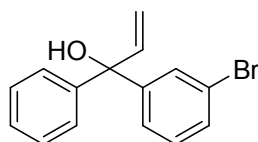


1-(4-Chlorophenyl)-1-phenylprop-2-en-1-ol (1l): Colorless oil. ¹H NMR (400 MHz, CDCl₃): δ_H 7.40-7.36 (m, 4H), 7.33 (d, J = 7.6 Hz, 2H), 7.32 (d, J = 7.6 Hz, 2H), 7.30-7.28 (m, 1H), 6.49 (dd, J = 17.2, 10.4 Hz, 1H), 5.35 (dd, J = 10.4, 1.2 Hz, 1H), 5.33 (dd, J = 17.2, 1.2 Hz, 1H), 2.37 (s, 1H, -OH); ¹³C NMR (100 MHz, CDCl₃): δ_C 145.3, 144.1, 143.0, 133.1, 128.3, 128.2, 128.2, 127.5, 126.8, 114.5, 79.0; GC-MS (EI): 244.1, 209.1, 139.0, 105.1, 77.1; HRMS(ESI): [M - H₂O + H]⁺ calcd for C₁₅H₁₂Cl⁺: 227.0627, found: 227.0627.

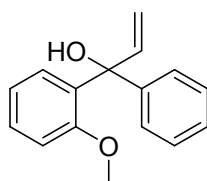


1-(3-Methoxyphenyl)-1-phenylprop-2-en-1-ol (1m): Pale yellow oil. ¹H NMR (400 MHz, CDCl₃): δ_H 7.38 (d, J = 7.6 Hz, 2H), 7.29 (t, J = 7.6 Hz, 2H), 7.23 (d, J = 7.6 Hz, 1H), 7.19 (d, J = 7.6 Hz, 1H), 6.99 (d, J = 1.6 Hz, 1H), 6.93 (dd, J = 7.6, 0.8 Hz, 1H), 6.77 (dd, J = 7.6, 1.6 Hz, 1H), 6.48 (dd, J = 17.2, 10.8 Hz, 1H), 5.29 (dd, J =

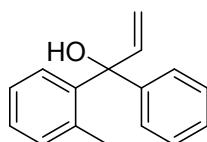
17.2, 1.2 Hz, 1H), 5.27 (dd, $J = 10.8, 1.2$ Hz, 1H), 3.75 (s, OCH₃, 3H), 2.43 (s, 1H, -OH); ¹³C NMR (100 MHz, CDCl₃): δ_c 159.0, 147.6, 145.8, 143.4, 128.7, 127.7, 126.8, 126.7, 119.3, 113.6, 112.6, 112.0, 78.7, 54.9; GC-MS (EI): 240.1, 135.1, 105.1, 77.1; HRMS(ESI): [M - H₂O + H]⁺ calcd for C₁₆H₁₅O⁺: 223.1123, found: 223.1119.



1-(3-Bromophenyl)-1-phenylprop-2-en-1-ol (1n): Colorless oil. ¹H NMR (400 MHz, CDCl₃): δ_H 7.63 (s, 1H), 7.44-7.29 (m, 7H), 7.21 (t, $J = 8.0$ Hz, 1H), 6.48 (dd, $J = 16.8, 10.4$ Hz, 1H), 5.37 (d, $J = 10.4$ Hz, 1H), 5.35 (d, $J = 16.8$ Hz, 1H), 2.41 (s, 1H, -OH); ¹³C NMR (100 MHz, CDCl₃): δ_c 148.0, 145.1, 142.8, 130.2, 129.8, 129.6, 128.3, 127.5, 126.8, 125.6, 122.4, 114.7, 79.0; GC-MS (EI): 290.0, 288.0, 209.1, 184.9, 182.9, 105.0, 77.1; HRMS(ESI): [M - H₂O + H]⁺ calcd for C₁₅H₁₂Br⁺: 271.0122, found: 271.0120.

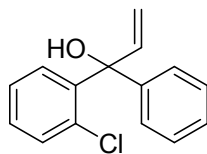


1-(2-Methoxyphenyl)-1-phenylprop-2-en-1-ol (1o)¹: Colorless oil. ¹H NMR (400 MHz, CDCl₃): δ_H 7.32 (d, $J = 7.6$ Hz, 2H), 7.28 (t, $J = 7.6$ Hz, 3H), 7.22 (t, $J = 7.6$ Hz, 2H), 6.95 (t, $J = 7.6$ Hz, 1H), 6.91 (d, $J = 7.6$ Hz, 1H), 6.40 (dd, $J = 17.2, 10.8$ Hz, 1H), 5.28 (dd, $J = 10.8, 1.2$ Hz, 1H), 5.06 (dd, $J = 17.2, 1.2$ Hz, 1H), 4.76 (s, 1H, OH), 3.64 (s, OCH₃, 3H).

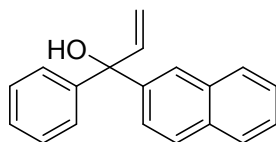


1-Phenyl-1-o-tolylprop-2-en-1-ol (1p): Colorless oil. ¹H NMR (400 MHz, CDCl₃): δ_H 7.57-7.55 (m, 1H), 7.29 (d, $J = 8.0$ Hz, 4H), 7.25-7.19 (m, 3H), 7.13-7.11 (m, 1H), 6.52 (dd, $J = 17.2, 10.8$ Hz, 1H), 5.26 (d, $J = 10.8$ Hz, 1H), 5.24 (d, $J = 17.2$ Hz, 1H), 2.72 (s, 1H, -OH), 2.03 (s, CH₃, 3H); ¹³C NMR (100 MHz, CDCl₃): δ_c 145.4, 143.7, 143.2, 137.3, 132.3, 128.0, 127.6, 127.4, 126.8, 126.2, 125.2, 113.3, 79.8, 21.4; GC-

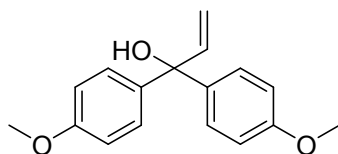
MS (EI): 224.1, 209.1, 119.0, 105.0, 91.1, 77.1; HRMS(ESI): $[M - H_2O + H]^+$ calcd for $C_{16}H_{15}^+$: 207.1173, found: 207.1163.



1-(2-Chlorophenyl)-1-phenylprop-2-en-1-ol (1q): Colorless oil. 1H NMR (400 MHz, $CDCl_3$): δ_H 7.72 (dd, $J = 7.6, 2.0$ Hz, 1H), 7.36-7.29 (m, 8H), 6.62 (dd, $J = 17.2, 10.8$ Hz, 1H), 5.36 (dd, $J = 10.8, 1.2$ Hz, 1H), 5.35 (dd, $J = 17.2, 1.2$ Hz, 1H), 3.24 (s, 1H, -OH); ^{13}C NMR (100 MHz, $CDCl_3$): δ_C 144.9, 142.3, 141.3, 132.5, 131.3, 129.2, 129.0, 128.2, 127.4, 126.7, 126.5, 115.0, 79.6; GC-MS (EI): 244.1, 209.1, 139.1, 105.1, 77.1; HRMS(ESI): $[M - H_2O + H]^+$ calcd for $C_{15}H_{12}Cl^+$: 227.0627, found: 227.0623.

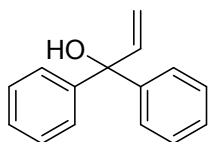


1-(Naphthalen-2-yl)-1-phenylprop-2-en-1-ol (1r): Pale yellow oil. 1H NMR (400 MHz, $CDCl_3$): δ_H 7.94 (d, $J = 8.4$ Hz, 1H), 7.78 (d, $J = 8.4$ Hz, 2H), 7.72 (dd, $J = 7.6, 1.2$ Hz, 1H), 7.41 (t, $J = 7.6$ Hz, 1H), 7.34-7.31 (m, 3H), 7.25-7.17 (m, 4H), 6.61 (dd, $J = 17.2, 10.4$ Hz, 1H), 5.28 (dd, $J = 17.2, 0.8$ Hz, 1H), 5.27 (dd, $J = 10.4, 0.8$ Hz, 1H), 2.56 (s, 1H, -OH); ^{13}C NMR (100 MHz, $CDCl_3$): δ_C 145.7, 143.9, 140.6, 134.8, 130.6, 129.2, 128.7, 128.2, 127.4, 127.0, 126.3, 125.6, 125.3, 125.2, 124.6, 113.8, 80.2; GC-MS (EI): 260.1, 155.1, 127.1, 105.1, 77.1; HRMS(ESI): $[M - H_2O + H]^+$ calcd for $C_{19}H_{15}^+$: 243.1173, found: 243.1178.

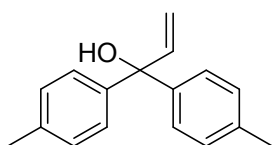


1,1-Bis(4-methoxyphenyl)prop-2-en-1-ol (1t)³: Pale yellow oil. 1H NMR (400 MHz, $CDCl_3$): δ_H 7.30 (d, $J = 8.8$ Hz, 4H), 6.86 (d, $J = 8.8$ Hz, 4H), 6.47 (dd, $J = 17.2, 10.4$ Hz, 1H), 5.30 (d, $J = 17.2$ Hz, 1H), 5.28 (d, $J = 10.4$ Hz, 1H), 3.81 (s, $2 \times OCH_3$, 6H).

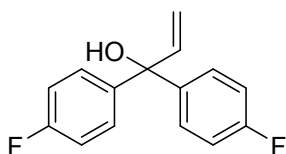
2) **1b**, **1c**, **1e**, **1f**, and **1s** were prepared from vinylmagnesium chloride and the corresponding benzophenone according to ref. *Org. Biomol. Chem.*, 2013, **11**, 2498.⁴



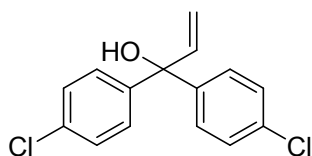
1,1-Diphenylprop-2-en-1-ol (1b)⁴: Colorless oil. ¹H NMR (400 MHz, CDCl₃): δ_H 7.36 (d, $J = 7.6$ Hz, 4H), 7.30 (t, $J = 7.6$ Hz, 4H), 7.24 (t, $J = 7.6$ Hz, 2H), 6.48 (dd, $J = 16.8, 10.8$ Hz, 1H), 5.30 (d, $J = 16.8$ Hz, 1H), 5.29 (d, $J = 10.8$ Hz, 1H), 2.34 (s, 1H, -OH).



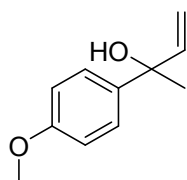
1,1-Dip-tolylprop-2-en-1-ol (1c)¹: Colorless oil. ¹H NMR (400 MHz, CDCl₃): δ_H 7.23 (d, $J = 7.8$ Hz, 4H), 7.09 (d, $J = 7.8$ Hz, 4H), 6.44 (dd, $J = 17.2, 10.6$ Hz, 1H), 5.27 (d, $J = 17.2$ Hz, 1H), 5.24 (d, $J = 10.6$ Hz, 1H), 2.30 (s, 6H, 2×CH₃).



1,1-Bis(4-fluorophenyl)prop-2-en-1-ol (1e)⁵: Colorless oil. ¹H NMR (400 MHz, CDCl₃): δ_H 7.35 (dd, $J = 9.0$ Hz, 4H), 7.02 (t, $J = 9.0$ Hz, 4H), 6.46 (dd, $J = 17.2, 10.6$ Hz, 1H), 5.34 (d, $J = 10.6$ Hz, 1H), 5.29 (d, $J = 17.2$ Hz, 1H), 2.36 (br s, 1H, -OH).



1,1-Bis(4-chlorophenyl)prop-2-en-1-ol (1f)¹: Colorless oil. ¹H NMR (400 MHz, CDCl₃): δ_H 7.27 (s, 8H), 6.40 (dd, $J = 17.2, 10.4$ Hz, 1H), 5.32 (d, $J = 10.4$ Hz, 1H), 5.26 (d, $J = 17.2$ Hz, 1H), 2.34 (s, 1H, -OH).

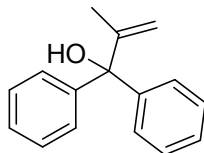


2-(4-Methoxyphenyl)but-3-en-2-ol (1s)⁶: Pale yellow oil. ¹H NMR (400 MHz, CDCl₃): δ_H 7.40 (d, $J = 9.0$ Hz, 2H), 6.88 (d, 2H, $J = 9.0$ Hz), 6.16 (dd, $J = 17.2, 10.8$

Hz, 1H), 5.29 (dd, $J = 17.2, 0.8$ Hz, 1H), 5.13 (dd, $J = 10.8, 0.8$ Hz, 1H), 3.79 (s, OCH₃, 3H), 1.64 (s, CH₃, 3H).

3) Synthesis of **1i**

1i was prepared from isopropenylmagnesium bromide and benzophenone according to ref. *Org. Lett.*, **2011**, *13*, 3648.⁷

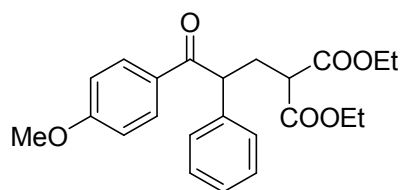


2-Methyl-1,1-diphenylprop-2-en-1-ol (1i)⁷: Pale yellow oil. ¹H NMR (400 MHz, CDCl₃): δ_H 7.41 (d, $J = 6.8$ Hz, 4H), 7.36 (t, $J = 6.8$ Hz, 4H), 7.31 (t, $J = 6.8$ Hz, 2H), 5.18 (s, 1H), 4.78 (s, 1H), 2.54 (s, 1H, -OH), 1.84 (s, CH₃, 3H).

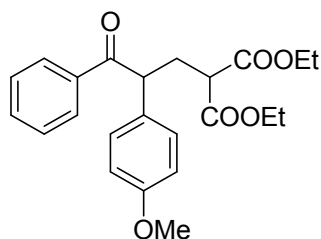
3. General Procedure for Photocatalytic Arylalkylation Reaction

A 10 mL round bottom flask was equipped with magnetic stir bar and was charged with 1-(4-methoxyphenyl)-1-phenylprop-2-en-1-ol **1a** (24.0 mg, 0.1 mmol), **2** (47.4 mg, 0.2 mmol), 2,6-dimethyl pyridine (21.4 mg, 0.2 mmol), DMSO (1 mL) and *fac*-Ir(ppy)₃ (3.3 mg, 0.005 mmol). The round bottom flask was evacuated and backfilled with N₂ three times under -78°C. The mixture was irradiated with blue LEDs for 143 h (monitored by TLC). After the reaction was completed, the reaction mixture was quenched with water (3 mL) and was extracted with EtOAc (5 mL $\times$ 4). The organic layer was combined, dried (MgSO₄), filtered, and concentrated in *vacuo*. The resulting residue was purified by PTLC (petroleum ether/EtOAc) to afford the desired product. The isomer ratio was detected by ¹H NMR analysis of the crude product.

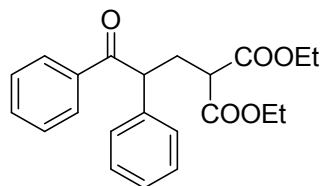
4. Characterization of products



Diethyl 2-(3-(4-methoxyphenyl)-3-oxo-2-phenylpropyl)malonate (3a1): Colorless oil. ^1H NMR (400 MHz, CDCl_3) δ_{H} 7.86 (d, $J = 8.8$ Hz, 2H), 7.24-7.19 (m, 4H), 7.16-7.11 (m, 1H), 6.77 (d, $J = 8.8$ Hz, 2H), 4.59 (t, $J = 7.6$ Hz, 1H), 4.14 (q, $J = 7.2$ Hz, 2H), 4.08-4.00 (m, 2H), 3.73 (s, OCH_3 , 3H), 3.22 (t, $J = 7.6$ Hz, 1H), 2.67-2.60 (m, 1H), 2.38-2.31 (m, 1H), 1.19 (t, $J = 7.2$ Hz, 3H), 1.11 (t, $J = 7.2$ Hz, 3H); ^{13}C NMR (100 MHz, CDCl_3) δ_{C} 197.1, 169.3, 169.2, 163.4, 138.6, 131.0, 129.3, 129.1, 128.3, 127.3, 113.7, 61.4, 55.4, 50.4, 49.7, 32.4, 14.1, 13.9; GC-MS (EI): 398.2, 135.0, 107.0, 92.0, 77.0; HRMS(ESI): $[\text{M} + \text{H}]^+$ calcd for $\text{C}_{23}\text{H}_{27}\text{O}_6^+$: 399.1808, found: 399.1808.

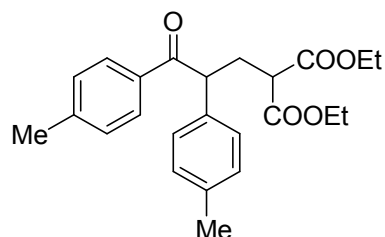


Diethyl 2-(2-(4-methoxyphenyl)-3-oxo-3-phenylpropyl)malonate (3a2): Colorless oil. ^1H NMR (400 MHz, CDCl_3) δ_{H} 7.93 (d, $J = 7.6$ Hz, 2H), 7.47 (t, $J = 7.6$ Hz, 1H), 7.37 (t, $J = 7.6$ Hz, 2H), 7.19 (d, $J = 8.4$ Hz, 2H), 6.82 (d, $J = 8.4$ Hz, 2H), 4.66 (t, $J = 7.2$ Hz, 1H), 4.22 (q, $J = 7.2$ Hz, 2H), 4.16-4.08 (m, 2H), 3.75 (s, OCH_3 , 3H), 3.28 (t, $J = 7.2$ Hz, 1H), 2.71-2.64 (m, 1H), 2.44-2.37 (m, 1H), 1.27 (t, $J = 7.2$ Hz, 3H), 1.18 (t, $J = 7.2$ Hz, 3H); ^{13}C NMR (100 MHz, CDCl_3) δ_{C} 198.8, 169.4, 169.2, 158.9, 136.3, 132.9, 130.0, 129.4, 128.7, 128.5, 114.5, 61.4, 55.2, 49.8, 49.6, 32.3, 14.1, 13.9; GC-MS (EI): 398.2, 293.1, 219.1, 145.0, 105.1, 77.0; HRMS(ESI): $[\text{M} + \text{H}]^+$ calcd for $\text{C}_{23}\text{H}_{27}\text{O}_6^+$: 399.1808, found: 399.1806.

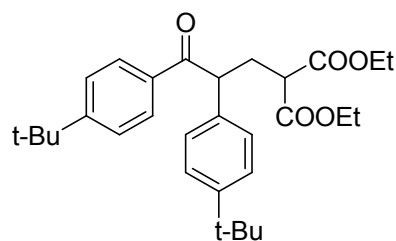


Diethyl 2-(3-oxo-2,3-diphenylpropyl)malonate (3b)⁸: Colorless oil. ^1H NMR (400 MHz, CDCl_3) δ_{H} 7.94 (dd, $J = 7.2$, 1.6 Hz, 2H), 7.47 (t, $J = 7.2$ Hz, 1H), 7.37 (t, $J =$

7.2 Hz, 2H), 7.30-7.28 (m, 4H), 7.23-7.19 (m, 1H), 4.72 (t, $J = 7.2$ Hz, 1H), 4.22 (q, $J = 7.2$ Hz, 2H), 4.15-4.07 (m, 2H), 3.30 (t, $J = 7.2$ Hz, 1H), 2.76-2.69 (m, 1H), 2.47-2.40 (m, 1H), 1.27 (t, $J = 7.2$ Hz, 3H), 1.18 (t, $J = 7.2$ Hz, 3H); ^{13}C NMR (100 MHz, CDCl_3) δ_c 198.6, 169.3, 169.1, 138.2, 136.3, 132.9, 129.1, 128.7, 128.5, 128.3, 127.4, 61.4, 50.7, 32.3, 14.0, 13.9; HRMS(ESI): $[\text{M} + \text{H}]^+$ calcd for $\text{C}_{22}\text{H}_{24}\text{O}_5^+$: 369.1702, found: 369.1697.

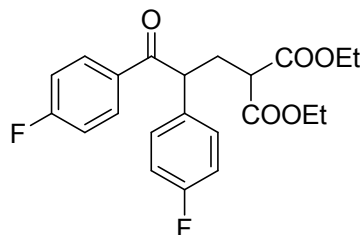


Diethyl 2-(3-oxo-2,3-dip-tolylpropyl)malonate (3c): Colorless oil. ^1H NMR (400 MHz, CDCl_3) δ_H 7.83 (d, $J = 8.0$ Hz, 2H), 7.16 (d, $J = 8.0$ Hz, 2H), 7.15 (d, $J = 8.0$ Hz, 2H), 7.09 (d, $J = 8.0$ Hz, 2H), 4.64 (t, $J = 7.2$ Hz, 1H), 4.21 (q, $J = 7.2$ Hz, 2H), 4.15-4.07 (m, 2H), 3.28 (t, $J = 7.6$ Hz, 1H), 2.72-2.64 (m, 1H), 2.44-2.37 (m, 1H), 2.34 (s, 3H), 2.27 (s, 3H), 1.27 (t, $J = 7.2$ Hz, 3H), 1.18 (t, $J = 7.2$ Hz, 3H); ^{13}C NMR (100 MHz, CDCl_3) δ_c 198.3, 169.4, 169.2, 143.7, 137.1, 135.3, 133.9, 129.8, 129.2, 128.9, 128.2, 61.4, 50.2, 49.7, 32.3, 21.6, 21.0, 14.1, 13.9; GC-MS (EI): 396.2, 305.1, 277.1, 185.0, 119.0, 91.0; HRMS(ESI): $[\text{M} + \text{Na}]^+$ calcd for $\text{C}_{24}\text{H}_{28}\text{O}_5\text{Na}^+$: 419.1834, found: 419.1831.

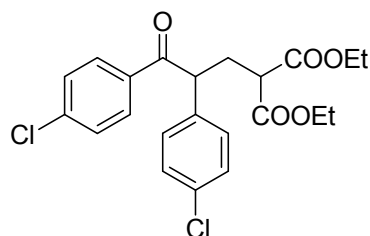


Diethyl 2-(2,3-bis(4-tert-butylphenyl)-3-oxopropyl)malonate (3d): Colorless oil. ^1H NMR (400 MHz, CDCl_3) δ_H 7.91 (d, $J = 8.4$ Hz, 2H), 7.40 (d, $J = 8.4$ Hz, 2H), 7.30 (d, $J = 8.4$ Hz, 2H), 7.21 (d, $J = 8.4$ Hz, 2H), 4.67 (t, $J = 7.2$ Hz, 1H), 4.20 (q, $J = 7.2$ Hz, 2H), 4.15-4.06 (m, 2H), 3.29 (t, $J = 7.2$ Hz, 1H), 2.72-2.65 (m, 1H), 2.45-2.38 (m, 1H), 1.29-1.25 (m, 21H), 1.17 (t, $J = 7.2$ Hz, 3H); ^{13}C NMR (100 MHz, CDCl_3) δ_c 198.3, 169.4, 169.2, 156.6, 150.2, 135.1, 133.8, 128.7, 127.9, 126.0, 125.5, 61.4, 50.0,

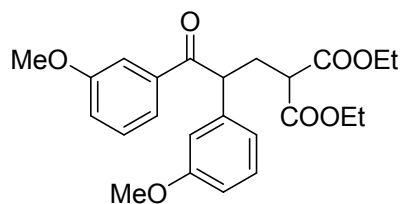
49.7, 35.0, 34.4, 32.5, 31.2, 31.0, 14.1, 13.9; GC-MS (EI): 480.3, 319.2, 161.1, 133.1; HRMS(ESI): $[M + Na]^+$ calcd for $C_{30}H_{40}O_5Na^+$: 503.2773, found: 503.2782.



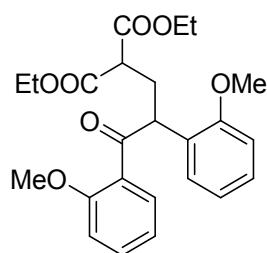
Diethyl 2-(2,3-bis(4-fluorophenyl)-3-oxopropyl)malonate (3e): Colorless oil. 1H NMR (400 MHz, $CDCl_3$) δ_H 7.96 (d, $J = 8.8$ Hz, 1H), 7.95 (d, $J = 8.8$ Hz, 1H), 7.25 (d, $J = 8.8$ Hz, 1H), 7.24 (d, $J = 8.8$ Hz, 1H), 7.06 (t, $J = 8.8$ Hz, 2H), 7.00 (t, $J = 8.8$ Hz, 2H), 4.69 (t, $J = 7.2$ Hz, 1H), 4.26-4.18 (m, 2H), 4.16-4.08 (m, 2H), 3.27 (t, 1H, $J = 7.2$ Hz), 2.73-2.66 (m, 1H), 2.42-2.34 (m, 1H), 1.27 (t, $J = 7.2$ Hz, 3H), 1.19 (t, $J = 7.2$ Hz, 3H); ^{13}C NMR (150 MHz, $CDCl_3$) δ_C 197.0, 169.2, 169.1, 165.6 (d, $J = 254.0$ Hz), 162.1 (d, $J = 245.4$ Hz), 133.7 (d, $J = 3.2$ Hz), 132.5 (d, $J = 3.0$ Hz), 131.4 (d, $J = 8.7$ Hz), 129.9 (d, $J = 7.5$ Hz), 116.2 (d, $J = 21.3$ Hz), 115.7 (dd, $J = 21.9, 3.3$ Hz), 61.5, 49.8, 49.4, 32.3, 14.1, 13.9; GC-MS (EI): 404.2, 281.1, 123.1, 95.1; HRMS(ESI): $[M + Na]^+$ calcd for $C_{22}H_{22}F_2O_5Na^+$: 427.1333, found: 427.1327.



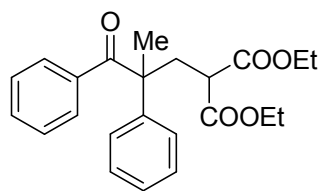
Diethyl 2-(2,3-bis(4-chlorophenyl)-3-oxopropyl)malonate (3f): Colorless oil. 1H NMR (400 MHz, $CDCl_3$) δ_H 7.85 (d, $J = 8.4$ Hz, 2H), 7.36 (d, $J = 8.4$ Hz, 2H), 7.28 (d, $J = 8.4$ Hz, 2H), 7.20 (d, $J = 8.4$ Hz, 2H), 4.68 (t, $J = 7.2$ Hz, 1H), 4.26-4.18 (m, 2H), 4.17-4.08 (m, 2H), 3.26 (t, $J = 7.2$ Hz, 1H), 2.73-2.66 (m, 1H), 2.41-2.34 (m, 1H), 1.27 (t, $J = 7.2$ Hz, 3H), 1.20 (t, $J = 7.2$ Hz, 3H); ^{13}C NMR (100 MHz, $CDCl_3$) δ_C 197.1, 169.1, 169.0, 139.7, 136.3, 134.4, 133.6, 133.0, 129.6, 129.4, 128.9, 61.5, 50.0, 49.4, 32.1, 14.0, 13.9; GC-MS (EI): 436.1, 297.0, 139.0, 111.0; HRMS(ESI): $[M + H]^+$ calcd for $C_{22}H_{23}Cl_2O_5^+$: 437.0923, found: 437.0923.



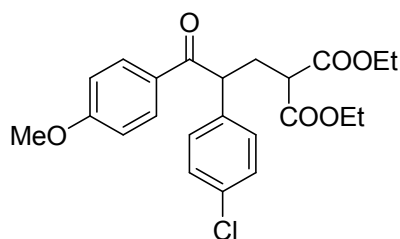
Diethyl 2-(2,3-bis(3-methoxyphenyl)-3-oxopropyl)malonate (3g): Colorless oil. ^1H NMR (400 MHz, CDCl_3) δ_H 7.52 (d, $J = 8.0$ Hz, 1H), 7.48 (t, $J = 2.0$ Hz, 1H), 7.27 (t, $J = 8.0$ Hz, 1H), 7.21 (t, $J = 8.0$ Hz, 1H), 7.02 (dd, $J = 8.0, 2.0$ Hz, 1H), 6.87 (d, $J = 8.0$ Hz, 1H), 6.81 (t, $J = 2.0$ Hz, 1H), 6.76 (dd, $J = 8.0, 2.0$ Hz, 1H), 4.66 (t, $J = 7.2$ Hz, 1H), 4.22 (q, $J = 7.2$ Hz, 2H), 4.17-4.09 (m, 2H), 3.80 (s, OCH_3 , 3H), 3.76 (s, OCH_3 , 3H), 3.30 (t, $J = 7.2$ Hz, 1H), 2.74-2.66 (m, 1H), 2.47-2.39 (m, 1H), 1.27 (t, $J = 7.2$ Hz, 3H), 1.19 (t, $J = 7.2$ Hz, 3H); ^{13}C NMR (100 MHz, CDCl_3) δ_c 198.2, 169.3, 169.1, 160.0, 159.6, 139.6, 137.6, 130.1, 129.4, 121.3, 120.7, 119.5, 113.8, 112.9, 112.8, 61.4, 55.3, 55.1, 50.9, 49.5, 32.2, 14.0, 13.9; GC-MS (EI): 428.2, 337.1, 135.1, 107.1, 77.1; HRMS(ESI): $[\text{M} + \text{Na}]^+$ calcd for $\text{C}_{24}\text{H}_{28}\text{O}_7\text{Na}^+$: 451.1733, found: 451.1733.



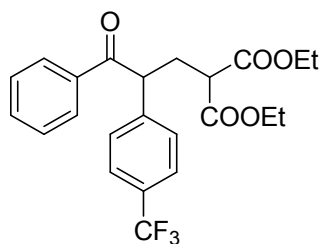
Diethyl 2-(2,3-bis(2-methoxyphenyl)-3-oxopropyl)malonate (3h): Colorless oil. ^1H NMR (400 MHz, CDCl_3) δ_H 7.46 (d, $J = 7.6$ Hz, 1H), 7.26 (t, $J = 7.6$ Hz, 1H), 7.13-7.07 (m, 2H), 6.85-6.79 (m, 2H), 6.75-6.70 (m, 2H), 4.96 (t, $J = 7.2$ Hz, 1H), 4.27-4.15 (m, 2H), 4.09 (q, $J = 7.2$ Hz, 2H), 3.71 (s, OCH_3 , 3H), 3.68 (s, OCH_3 , 3H), 3.34 (t, $J = 7.2$ Hz, 1H), 2.84-2.77 (m, 1H), 2.34-2.27 (m, 1H), 1.26 (t, $J = 7.2$ Hz, 3H), 1.20 (t, $J = 7.2$ Hz, 3H); ^{13}C NMR (100 MHz, CDCl_3) δ_c 202.2, 169.5, 169.2, 157.3, 156.9, 132.3, 130.0, 129.5, 128.6, 128.2, 126.8, 120.4, 120.0, 110.9, 110.3, 61.0, 61.0, 55.0, 54.9, 49.9, 49.0, 30.1, 13.9, 13.8; GC-MS (EI): 428.2, 293.1, 135.1, 77.1; HRMS(ESI): $[\text{M} + \text{Na}]^+$ calcd for $\text{C}_{24}\text{H}_{28}\text{O}_7\text{Na}^+$: 451.1733, found: 451.1738.



Diethyl 2-(2-methyl-3-oxo-2,3-diphenylpropyl)malonate (3i): Colorless oil. ^1H NMR (400 MHz, CDCl_3) δ_{H} 7.42 (dd, $J = 7.6, 1.6$ Hz, 2H), 7.36 (t, $J = 7.6$ Hz, 3H), 7.32-7.27 (m, 3H), 7.21 (t, $J = 7.6$ Hz, 2H), 4.11-3.97 (m, 4H), 3.29 (t, $J = 6.4$ Hz, 1H), 2.78 (dd, $J = 6.4, 2.0$ Hz, 1H), 2.69 (dd, $J = 6.4, 2.0$ Hz, 1H), 1.63 (s, CH_3 , 3H), 1.19 (t, $J = 7.2$ Hz, 3H), 1.18 (t, $J = 7.2$ Hz, 3H); ^{13}C NMR (100 MHz, CDCl_3) δ_{C} 202.6, 169.6, 169.6, 142.0, 136.2, 131.8, 129.6, 129.0, 127.9, 127.3, 126.7, 61.4, 61.4, 54.1, 48.6, 39.0, 22.9, 13.9, 13.9; GC-MS (EI): 382.2, 277.1, 185.0, 129.0, 105.0; HRMS(ESI): $[\text{M} + \text{Na}]^+$ calcd for $\text{C}_{23}\text{H}_{26}\text{O}_5\text{Na}^+$: 405.1678, found: 405.1679.

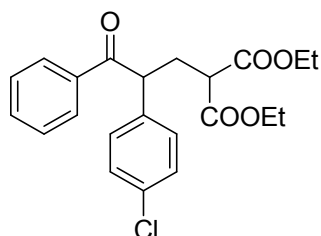


Diethyl 2-(2-(4-chlorophenyl)-3-(4-methoxyphenyl)-3-oxopropyl)malonate (3j): Colorless oil. ^1H NMR (400 MHz, CDCl_3) δ_{H} 7.91 (d, $J = 8.8$ Hz, 2H), 7.27 (d, $J = 8.8$ Hz, 2H), 7.23 (d, $J = 8.8$ Hz, 2H), 6.86 (d, $J = 8.8$ Hz, 2H), 4.67 (t, $J = 7.2$ Hz, 1H), 4.24-4.18 (m, 2H), 4.16-4.08 (m, 2H), 3.82 (s, OCH_3 , 3H), 3.27 (t, $J = 7.2$ Hz, 1H), 2.72-2.65 (m, 1H), 2.41-2.34 (m, 1H), 1.27 (t, $J = 7.2$ Hz, 3H), 1.19 (t, $J = 7.2$ Hz, 3H); ^{13}C NMR (100 MHz, CDCl_3) δ_{C} 196.7, 169.2, 169.1, 163.6, 137.1, 133.3, 131.0, 129.6, 129.2, 129.1, 113.8, 61.5, 55.4, 49.6, 49.6, 32.3, 14.0, 13.9; GC-MS (EI): 432.2, 341.1, 135.1, 77.1; HRMS(ESI): $[\text{M} + \text{Na}]^+$ calcd for $\text{C}_{23}\text{H}_{25}\text{ClO}_6\text{Na}^+$: 455.1237, found: 455.1237.



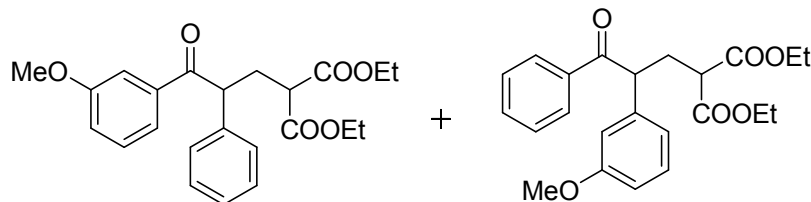
Diethyl 2-(3-oxo-3-phenyl-2-(4-(trifluoromethyl)phenyl)propyl)malonate (3k):

Colorless oil. ^1H NMR (400 MHz, CDCl_3) δ_{H} 7.93 (d, $J = 8.0$ Hz, 2H), 7.57 (d, $J = 8.0$ Hz, 2H), 7.51 (t, $J = 7.2$ Hz, 1H), 7.39-7.44 (m, 4H), 4.84 (t, $J = 7.2$ Hz, 1H), 4.26-4.18 (m, 2H), 4.16-4.08 (m, 2H), 3.28 (t, $J = 7.2$ Hz, 1H), 2.79-2.72 (m, 1H), 2.46-2.39 (m, 1H), 1.27 (t, $J = 7.2$ Hz, 3H), 1.18 (t, $J = 7.2$ Hz, 3H); ^{13}C NMR (100 MHz, CDCl_3) δ_{C} 198.0, 169.0, 168.9, 142.3, 136.0, 133.4, 129.8 (q, $J = 32.5$ Hz), 128.8, 128.7, 128.7, 126.1 (q, $J = 3.8$ Hz), 124.0 (q, $J = 270.7$ Hz), 61.6, 61.6, 50.4, 49.5, 32.3, 14.0, 13.9; GC-MS (EI): 436.2, 145.1, 105.1, 77.1; HRMS(ESI): $[\text{M} + \text{Na}]^+$ calcd for $\text{C}_{23}\text{H}_{23}\text{F}_3\text{O}_5\text{Na}^+$: 459.1395, found: 459.1397.



Diethyl 2-(2-(4-chlorophenyl)-3-oxo-3-phenylpropyl)malonate (3l): Colorless oil.

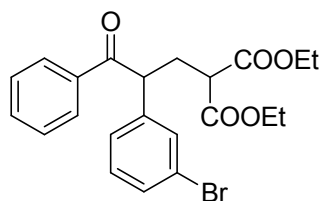
^1H NMR (400 MHz, CDCl_3) δ_{H} 7.92 (d, $J = 7.2$ Hz, 2H), 7.50 (t, $J = 7.2$ Hz, 1H), 7.39 (t, $J = 7.2$ Hz, 2H), 7.27 (d, $J = 8.4$ Hz, 2H), 7.23 (d, $J = 8.4$ Hz, 2H), 4.72 (t, $J = 7.2$ Hz, 1H), 4.25-4.18 (m, 2H), 4.16-4.08 (m, 2H), 3.27 (t, $J = 7.2$ Hz, 1H), 2.74-2.67 (m, 1H), 2.43-2.36 (m, 1H), 1.27 (t, $J = 7.2$ Hz, 3H), 1.18 (t, $J = 7.2$ Hz, 3H); ^{13}C NMR (100 MHz, CDCl_3) δ_{C} 198.3, 169.1, 169.0, 136.6, 136.0, 133.4, 133.2, 129.7, 129.3, 128.6, 128.6, 61.5, 49.9, 49.5, 32.2, 14.0, 13.9; GC-MS (EI): 402.2, 297.1, 105.1, 77.1; HRMS(ESI): $[\text{M} + \text{Na}]^+$ calcd for $\text{C}_{22}\text{H}_{23}\text{ClO}_5\text{Na}^+$: 425.1132, found: 425.1142.



Diethyl 2-(2-(3-methoxyphenyl)-3-oxo-3-phenylpropyl)malonate or diethyl 2-(3-(3-methoxyphenyl)-3-oxo-2-phenylpropyl)malonate (4:1) (3m): Colorless oil.

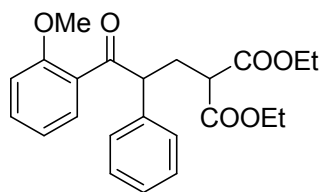
^1H NMR (400 MHz, CDCl_3) δ_{H} 7.94 (d, $J = 7.6$ Hz, 2H), 7.37 (t, $J = 7.6$ Hz, 2H), 7.30-7.25 (m, 1H), 7.21 (t, $J = 8.0$ Hz, 1H), 6.87 (d, $J = 7.6$ Hz, 1H), 6.81 (t, $J = 2.0$ Hz, 1H), 6.75 (dd, $J = 8.0, 2.0$ Hz, 1H), 4.70-4.66 (m, 1H), 4.22 (q, $J = 7.2$ Hz, 2H), 4.16-

4.08 (m, 2H), 3.76 (s, OCH₃, 3H), 3.30 (t, $J = 7.2$ Hz, 1H), 2.75-2.66 (m, 1H), 2.47-2.39 (m, 1H), 1.27 (t, $J = 7.2$ Hz, 3H), 1.18 (t, $J = 7.2$ Hz, 3H); ¹³C NMR (100 MHz, CDCl₃) δ_c 198.4, 169.3, 169.2, 160.0, 139.6, 136.3, 133.0, 130.1, 128.7, 128.5, 120.7, 113.8, 112.9, 61.4, 55.2, 50.7, 49.5, 32.2, 14.1, 13.9; GC-MS (EI): 398.2, 307.1, 135.1, 105.1, 77.1; HRMS(ESI): [M + Na]⁺ calcd for C₂₃H₂₆O₆Na⁺: 421.1627, found: 421.1636.



Diethyl 2-(2-(3-bromophenyl)-3-oxo-3-phenylpropyl)malonate (3n): Colorless oil.

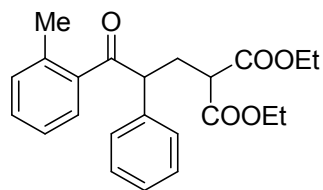
¹H NMR (400 MHz, CDCl₃) δ_H 7.93 (d, $J = 7.6$ Hz, 2H), 7.50 (t, $J = 7.6$ Hz, 1H), 7.45 (s, 1H), 7.40 (t, $J = 7.6$ Hz, 2H), 7.36 (d, $J = 7.6$ Hz, 1H), 7.23 (d, $J = 7.6$ Hz, 1H), 7.17 (t, $J = 7.6$ Hz, 1H), 4.72 (t, $J = 7.2$ Hz, 1H), 4.22 (q, $J = 7.2$ Hz, 2H), 4.16-4.08 (m, 2H), 3.29 (t, $J = 7.2$ Hz, 1H), 2.75-2.68 (m, 1H), 2.44-2.37 (m, 1H), 1.28 (t, $J = 7.2$ Hz, 3H), 1.19 (t, $J = 7.2$ Hz, 3H); ¹³C NMR (100 MHz, CDCl₃) δ_c 198.0, 169.1, 168.9, 140.4, 136.1, 133.3, 131.3, 130.7, 130.6, 128.7, 128.6, 126.9, 123.1, 61.5, 61.5, 50.2, 49.5, 32.3, 14.0, 13.9; GC-MS (EI): 448.1, 446.1, 355.0, 105.1, 77.1; HRMS(ESI): [M + Na]⁺ calcd for C₂₂H₂₃BrO₅Na⁺: 469.0627, found: 469.0632.



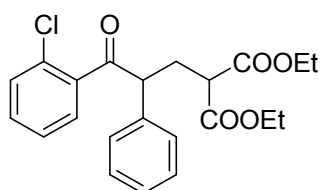
Diethyl 2-(3-(2-methoxyphenyl)-3-oxo-2-phenylpropyl)malonate (3o): Colorless

oil. ¹H NMR (400 MHz, CDCl₃) δ_H 7.45 (dd, $J = 7.6, 1.6$ Hz, 1H), 7.34 (dt, $J = 7.6, 1.6$ Hz, 1H), 7.26-7.15 (m, 5H), 6.90-6.84 (m, 2H), 4.76 (t, $J = 7.6$ Hz, 1H), 4.21 (q, $J = 7.2$ Hz, 2H), 4.17-4.07 (m, 2H), 3.82 (s, OCH₃, 3H), 3.31 (t, $J = 7.6$ Hz, 1H), 2.78-2.71 (m, 1H), 2.43-2.36 (m, 1H), 1.27 (t, $J = 7.2$ Hz, 3H), 1.21 (t, $J = 7.2$ Hz, 3H); ¹³C NMR (100 MHz, CDCl₃) δ_c 202.0, 169.3, 169.2, 157.7, 138.1, 133.0, 130.5, 128.7, 128.5, 128.5, 127.1, 120.6, 111.4, 61.3, 55.3, 54.9, 49.9, 32.0, 14.0, 13.9; GC-MS (EI):

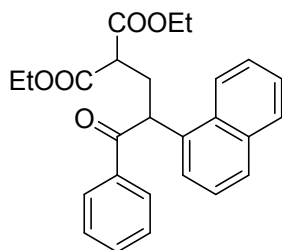
398.2, 293.1, 135.1, 77.1; HRMS(ESI): $[M + Na]^+$ calcd for $C_{23}H_{26}O_6Na^+$: 421.1627, found: 421.1636.



Diethyl 2-(3-oxo-2-phenyl-3-oxo-2-phenylpropyl)malonate (3p): Colorless oil. 1H NMR (400 MHz, $CDCl_3$) δ_H 7.54 (d, $J = 7.6$ Hz, 2H), 7.29-7.25 (m, 3H), 7.22-7.19 (m, 3H), 7.15 (t, $J = 7.6$ Hz, 1H), 7.14 (t, $J = 7.6$ Hz, 1H), 4.55 (t, $J = 7.2$ Hz, 1H), 4.25-4.18 (m, 2H), 4.17-4.10 (m, 2H), 3.30 (t, $J = 7.2$ Hz, 1H), 2.79-2.72 (m, 1H), 2.47-2.40 (m, 1H), 2.31 (s, CH_3 , 3H), 1.26 (t, $J = 7.2$ Hz, 3H), 1.21 (t, $J = 7.2$ Hz, 3H); ^{13}C NMR (100 MHz, $CDCl_3$) δ_c 202.6, 169.4, 169.1, 138.2, 138.2, 137.4, 131.6, 131.0, 129.0, 128.5, 128.0, 127.5, 125.4, 61.5, 53.8, 49.7, 31.7, 20.7, 14.1, 14.0; GC-MS (EI): 382.2, 291.1, 119.1, 77.1; HRMS(ESI): $[M + Na]^+$ calcd for $C_{23}H_{26}O_5Na^+$: 405.1678, found: 405.1680.

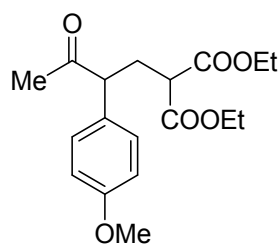


Diethyl 2-(3-(2-chlorophenyl)-3-oxo-2-phenylpropyl)malonate (3q): Colorless oil. 1H NMR (400 MHz, $CDCl_3$) δ_H 7.31 (dd, $J = 7.6, 1.2$ Hz, 1H), 7.29-7.22 (m, 4H), 7.17 (dd, $J = 7.6, 1.2$ Hz, 2H), 7.14 (dt, $J = 7.6, 1.2$ Hz, 1H), 7.09 (dd, $J = 7.6, 1.2$ Hz, 1H), 4.56 (t, $J = 7.2$ Hz, 1H), 4.21 (q, $J = 7.2$ Hz, 2H), 4.17-4.10 (m, 2H), 3.31 (t, $J = 7.2$ Hz, 1H), 2.83-2.76 (m, 1H), 2.51-2.44 (m, 1H), 1.27 (t, $J = 7.2$ Hz, 3H), 1.23 (t, $J = 7.2$ Hz, 3H); ^{13}C NMR (100 MHz, $CDCl_3$) δ_c 201.8, 169.2, 169.0, 139.1, 136.2, 131.2, 130.5, 130.2, 129.0, 128.9, 128.8, 127.8, 126.5, 61.4, 55.1, 49.5, 30.9, 14.0, 14.0; GC-MS (EI): 402.2, 263.1, 139.0, 77.1; HRMS(ESI): $[M + Na]^+$ calcd for $C_{22}H_{23}ClO_5Na^+$: 425.1132, found: 425.1137.

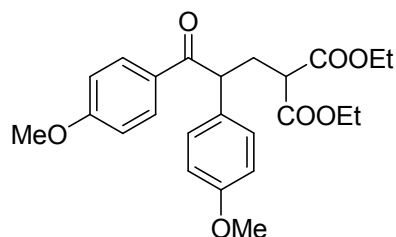


Diethyl 2-(2-(naphthalen-1-yl)-3-oxo-3-phenylpropyl)malonate (3r): Colorless oil.

^1H NMR (400 MHz, CDCl_3) δ_H 8.39 (d, $J = 8.0$ Hz, 1H), 7.88 (d, $J = 8.0$ Hz, 1H), 7.85 (d, $J = 8.0$ Hz, 2H), 7.73 (d, $J = 8.0$ Hz, 1H), 7.66 (t, $J = 7.6$ Hz, 1H), 7.54 (t, $J = 8.0$ Hz, 1H), 7.39 (t, $J = 7.6$ Hz, 1H), 7.32 (t, $J = 7.6$ Hz, 1H), 7.25 (t, $J = 7.6$ Hz, 3H), 5.55 (dd, $J = 9.0, 6.0$ Hz, 1H), 4.23-4.11 (m, 4H), 3.50 (dd, $J = 9.0, 6.0$ Hz, 1H), 2.86-2.79 (m, 1H), 2.52-2.45 (m, 1H), 1.22 (t, $J = 7.2$ Hz, 3H), 1.19 (t, $J = 7.2$ Hz, 3H); ^{13}C NMR (100 MHz, CDCl_3) δ_c 199.0, 169.6, 169.2, 136.4, 135.0, 134.4, 132.9, 130.9, 129.2, 128.5, 128.1, 127.0, 125.9, 125.7, 122.6, 61.4, 61.4, 49.8, 32.2, 14.0, 13.9; GC-MS (EI): 418.2, 313.1, 165.1, 105.1, 77.1; HRMS(ESI): $[\text{M} + \text{Na}]^+$ calcd for $\text{C}_{26}\text{H}_{26}\text{O}_5\text{Na}^+$: 441.1678, found: 441.1674.



Diethyl 2-(2-(4-methoxyphenyl)-3-oxobutyl)malonate (3s): Colorless oil. ^1H NMR (400 MHz, CDCl_3) δ_H 7.09 (d, $J = 8.8$ Hz, 2H), 6.87 (d, $J = 8.8$ Hz, 2H), 4.19 (q, $J = 7.2$ Hz, 2H), 4.13 (qd, $J = 7.2, 1.2$ Hz, 2H), 3.80 (s, OCH_3 , 3H), 3.69 (dd, $J = 8.4, 6.4$ Hz, 1H), 3.17 (dd, $J = 8.4, 6.4$ Hz, 1H), 2.59-2.52 (m, 1H), 2.26-2.19 (m, 1H), 2.04 (s, CH_3 , 3H), 1.26 (t, $J = 7.2$ Hz, 3H), 1.23 (t, $J = 7.2$ Hz, 3H); ^{13}C NMR (100 MHz, CDCl_3) δ_c 207.2, 169.3, 169.1, 159.2, 129.4, 129.4, 114.6, 61.4, 61.4, 55.7, 55.2, 49.5, 30.5, 28.9, 14.0, 14.0; GC-MS (EI): 336.2, 293.1, 219.1, 145.1, 43.1; HRMS(ESI): $[\text{M} + \text{H}]^+$ calcd for $\text{C}_{18}\text{H}_{25}\text{O}_6^+$: 337.1651, found: 337.1651.



Diethyl 2-(2,3-bis(4-methoxyphenyl)-3-oxopropyl)malonate (3t)⁹: Colorless oil. ¹H NMR (400 MHz, CDCl₃) δ_H 7.92 (d, J = 8.8 Hz, 2H), 7.19 (d, J = 8.8 Hz, 2H), 6.85 (d, J = 8.8 Hz, 2H), 6.82 (d, J = 8.8 Hz, 2H), 4.61 (t, J = 7.2 Hz, 1H), 4.21 (q, J = 7.2 Hz, 2H), 4.16-4.08 (m, 2H), 3.80 (s, OCH₃, 3H), 3.74 (s, OCH₃, 3H), 3.29 (t, J = 7.2 Hz, 1H), 2.70-2.63 (m, 1H), 2.43-2.36 (m, 1H), 1.27 (t, J = 7.2 Hz, 3H), 1.19 (t, 3H, J = 7.2 Hz, 3H); ¹³C NMR (100 MHz, CDCl₃) δ_c 197.3, 169.4, 169.2, 163.3, 158.8, 131.0, 130.5, 129.3, 129.3, 114.4, 113.6, 61.3, 55.3, 55.1, 49.6, 49.5, 32.4, 14.0, 13.9 . HRMS(ESI): [M + H]⁺calcd for C₂₄H₂₉O₇⁺: 429.1913, found: 429.1917.

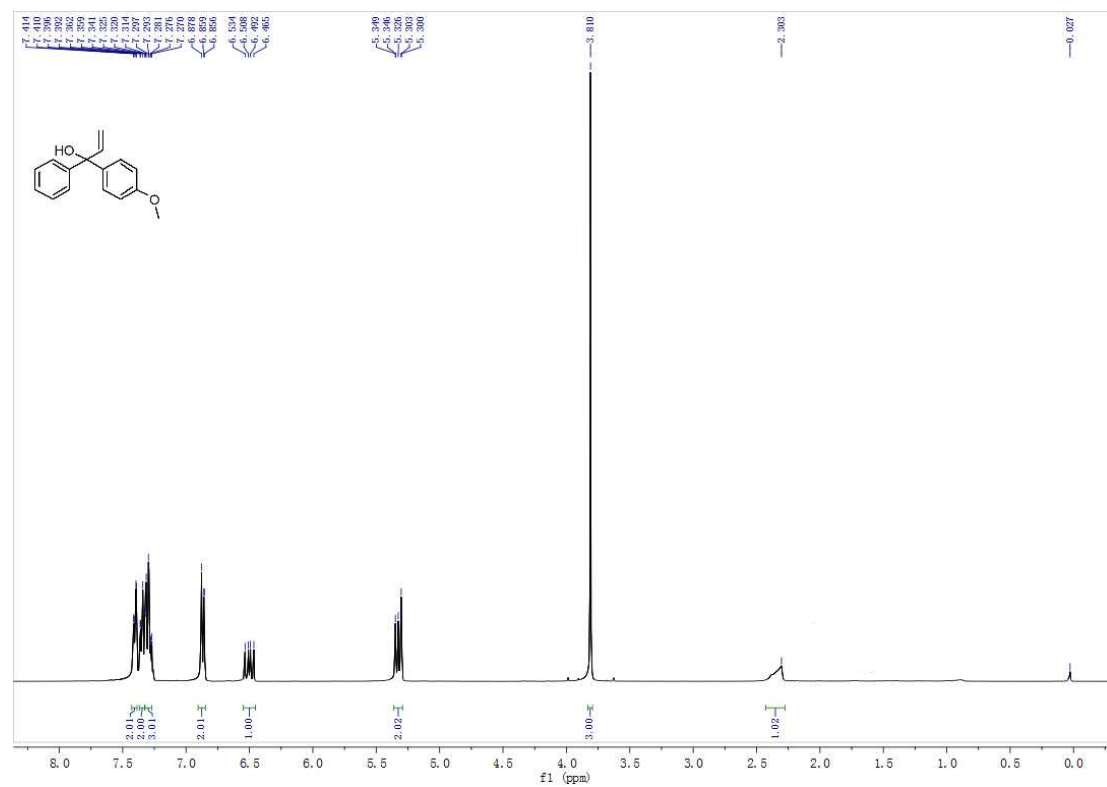
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- (1) X. W. Liu, F. Xiong, X. P. Huang, L. Xu, P. F. Li, X. X. Wu. *Angew. Chem., Int. Ed.*, 2013, **52**, 6962.
- (2) H. Egami, R. Shimizu, Y. Usuiac, M. Sodeoka. *Chem. Commun.*, 2013, **49**, 7346.
- (3) S. Hayashi, H. Yorimitsu, K. Oshima. *J. Am. Chem. Soc.*, 2009, **131**, 2052-2053.
- (4) J. J. Zhang, C. S. Yan, Y. Peng, Z. B. Luo, X. B. Xu, Y. W. Wang. *Org. Biomol. Chem.*, 2013, **11**, 2498.
- (5) C. Botteghi, M. Marchetti, S. Paganellia, F. Persi-Paoli. *Tetrahedron* 2001, **57**, 1631.
- (6) S. Prevost, N. Dupre, M. Leutzsch, Q. G. Wang, V. Wakchaure, B. List, *Angew. Chem., Int. Ed.*, 2014, **53**, 8770.
- (7) D. Rosa, A. Orellana. *Org. Lett.*, 2011, **13**, 3648.
- (8) B. Giese, J. N. He, W. Mehl. *Chem. Ber.*, 1988, **121**, 2063.
- (9) M. P. Reddy, G. S. K. Rao. *J. Chem. Soc., Perkin Trans., 1*, 1981, 2662.

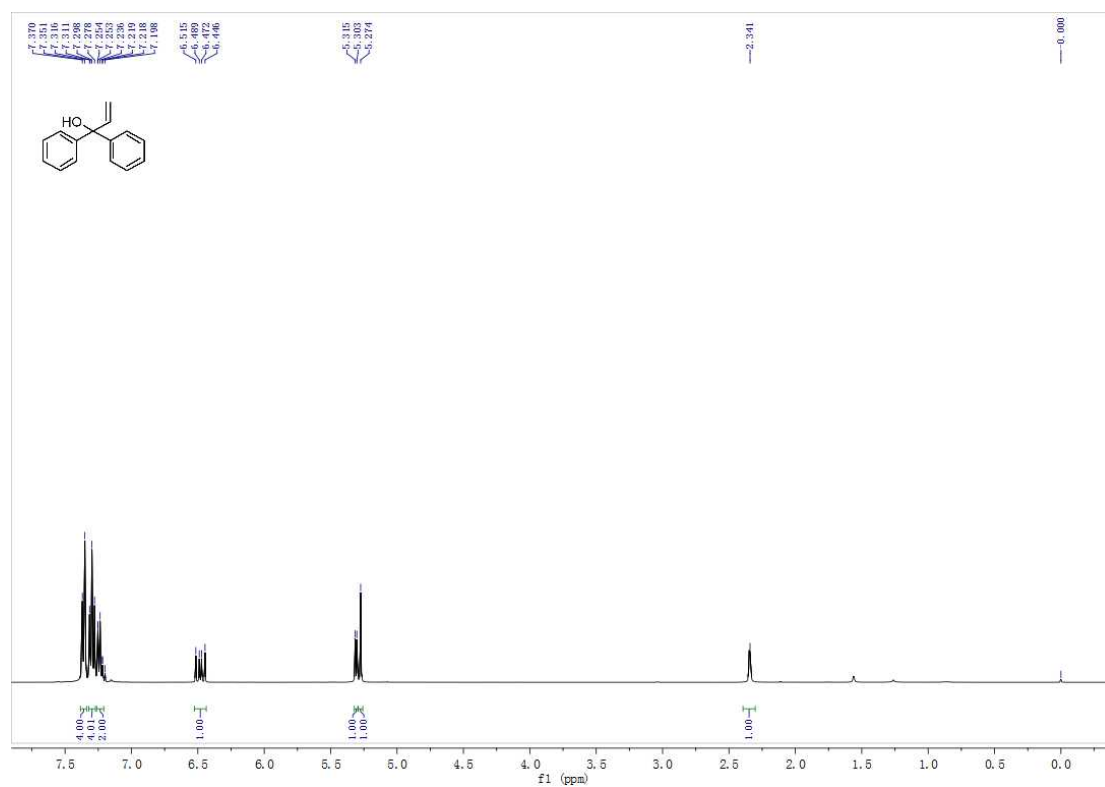
5. NMR Spectra

Substrates NMR spectra:

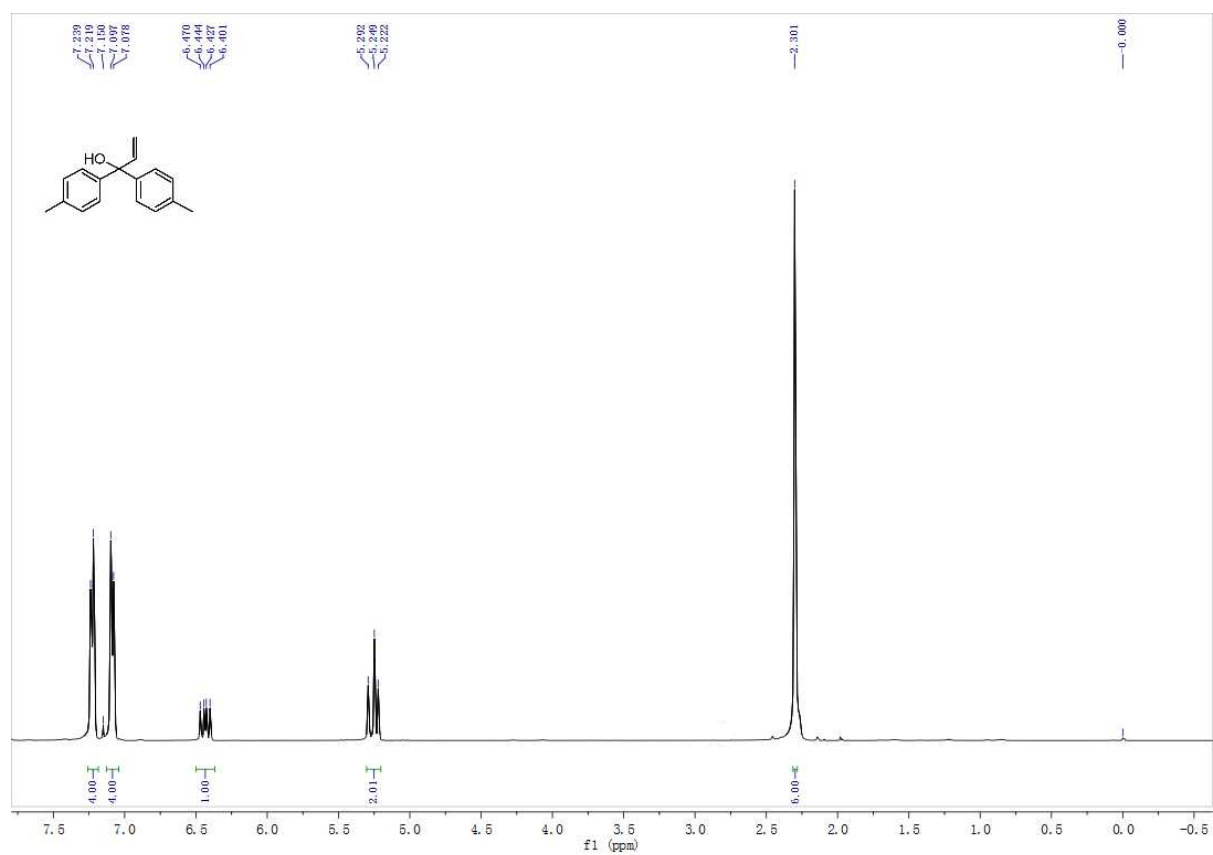
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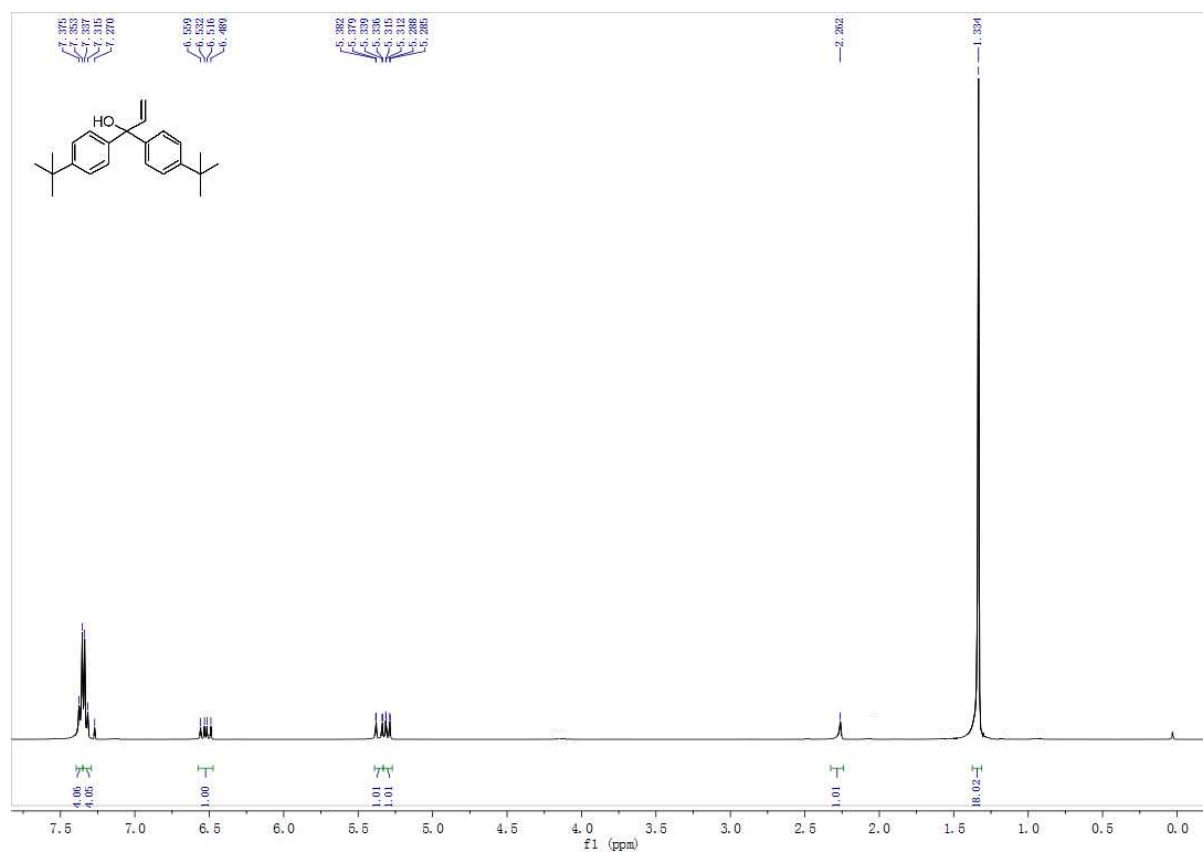
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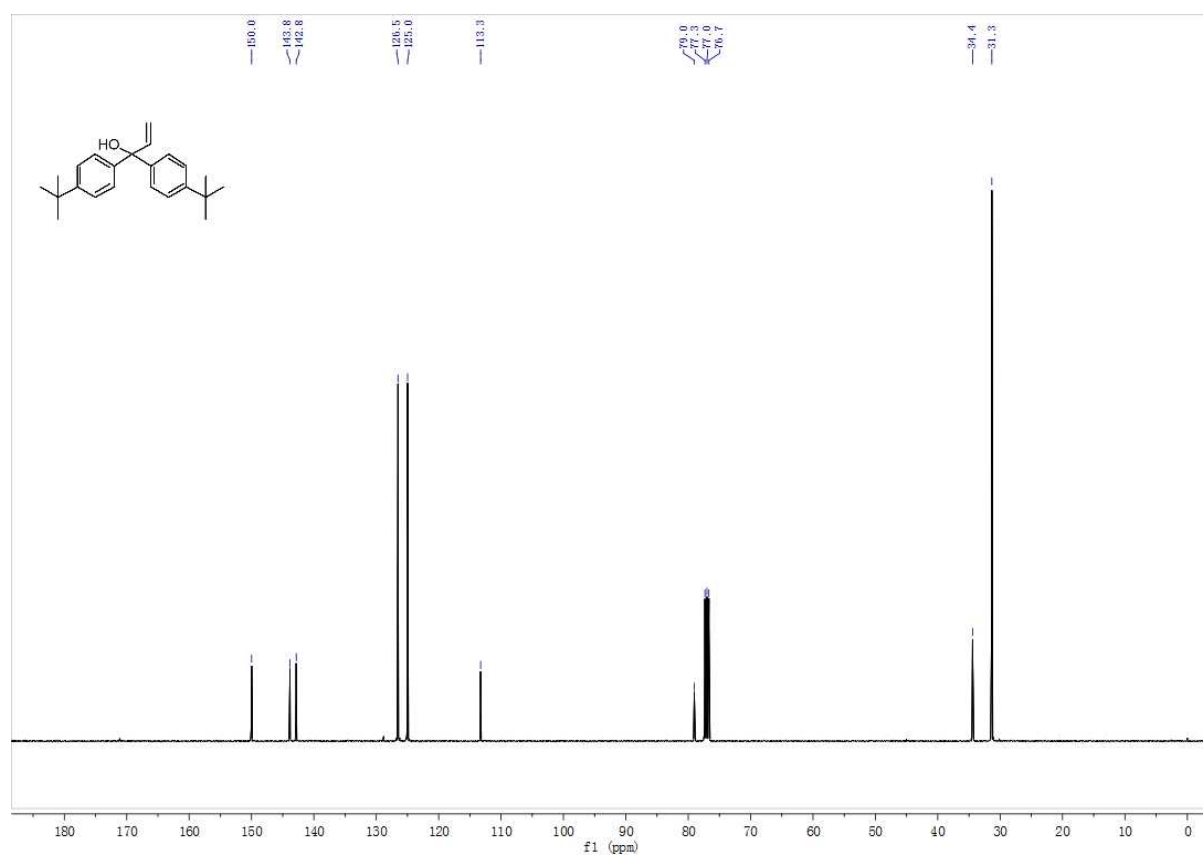
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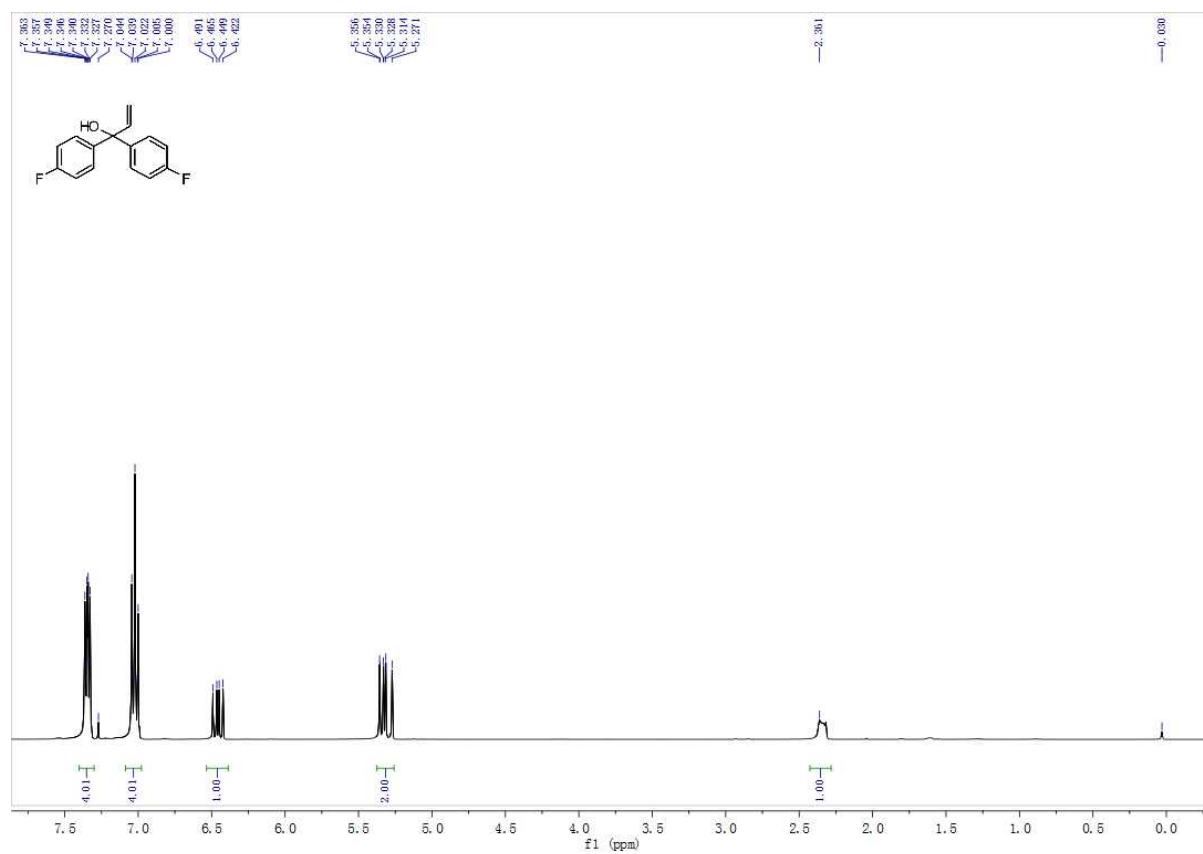
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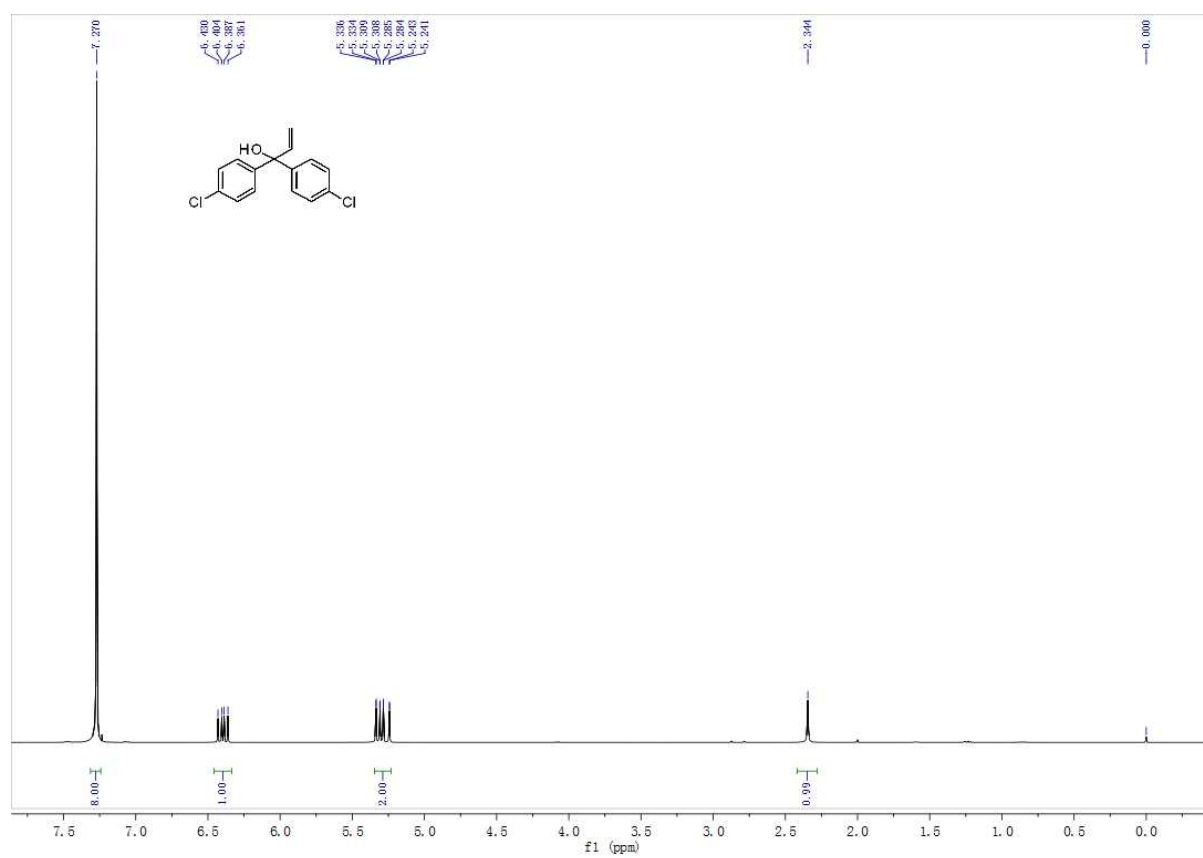
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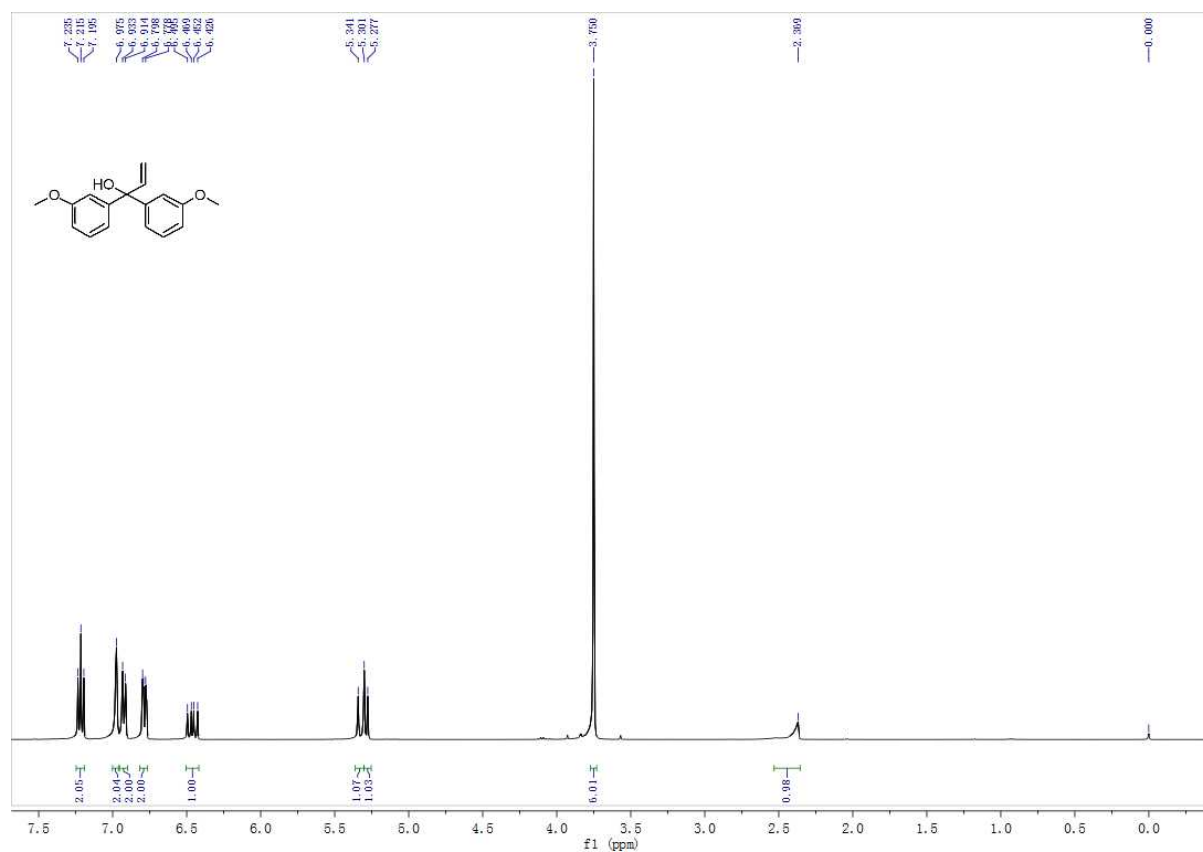
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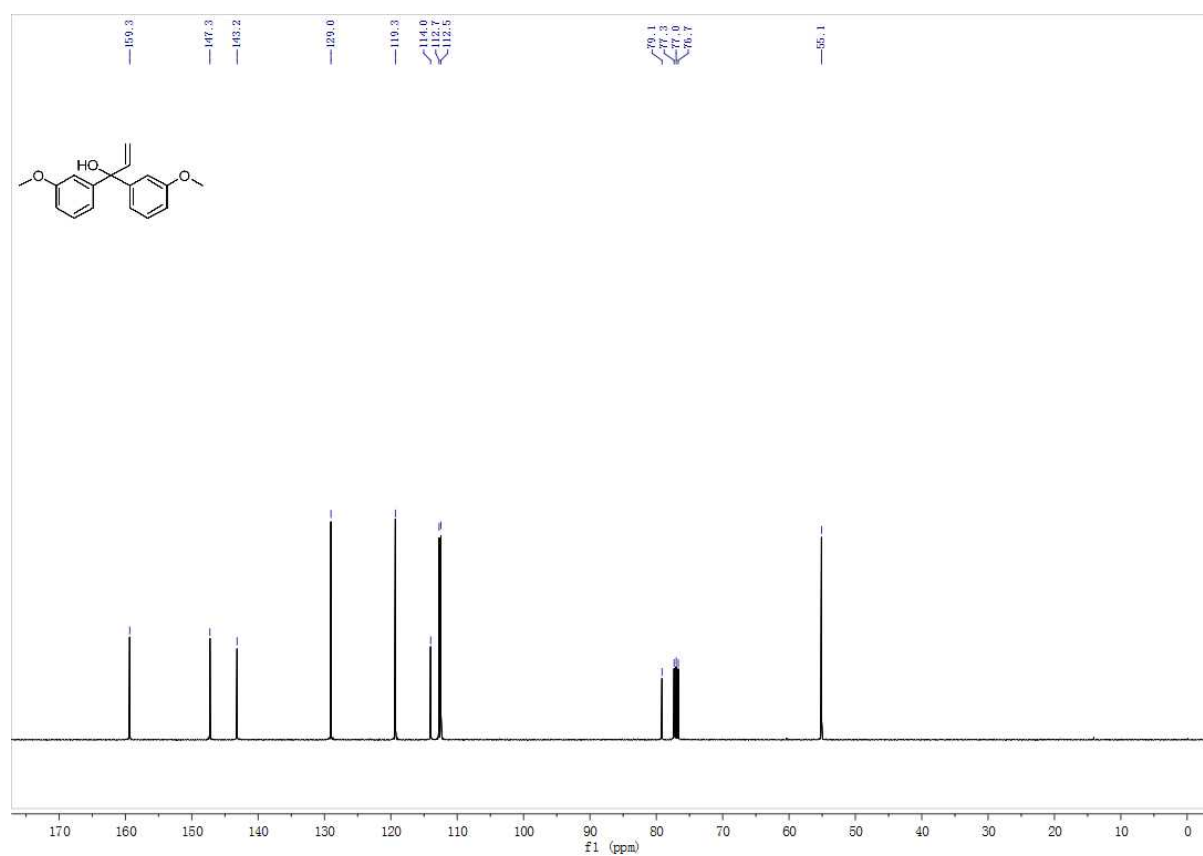
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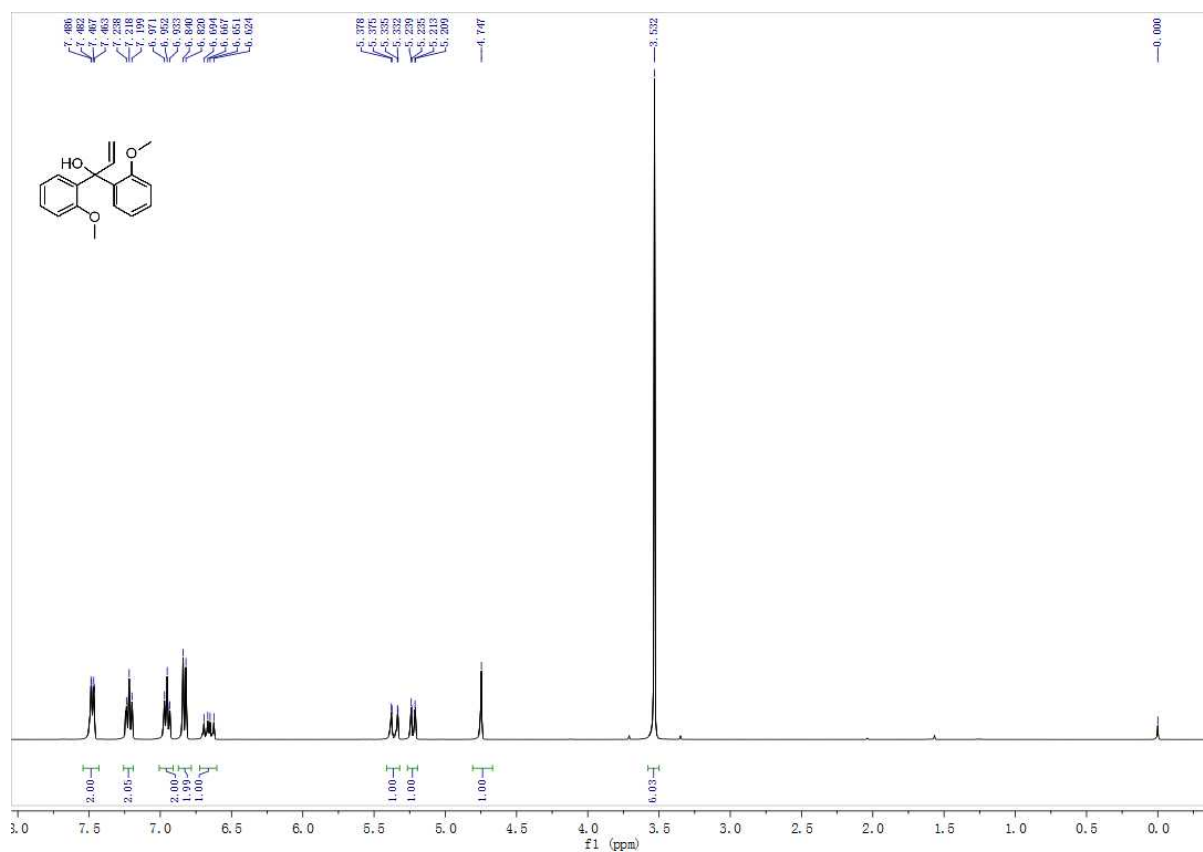
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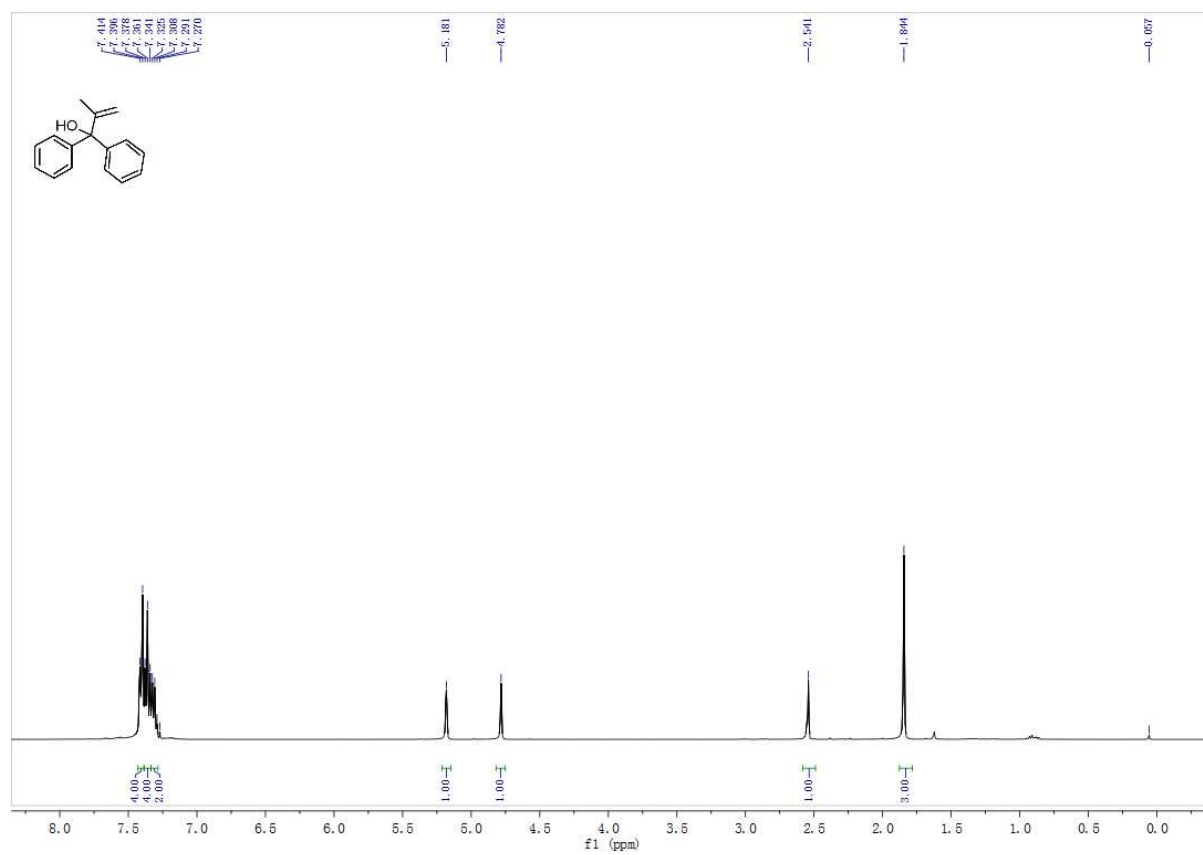
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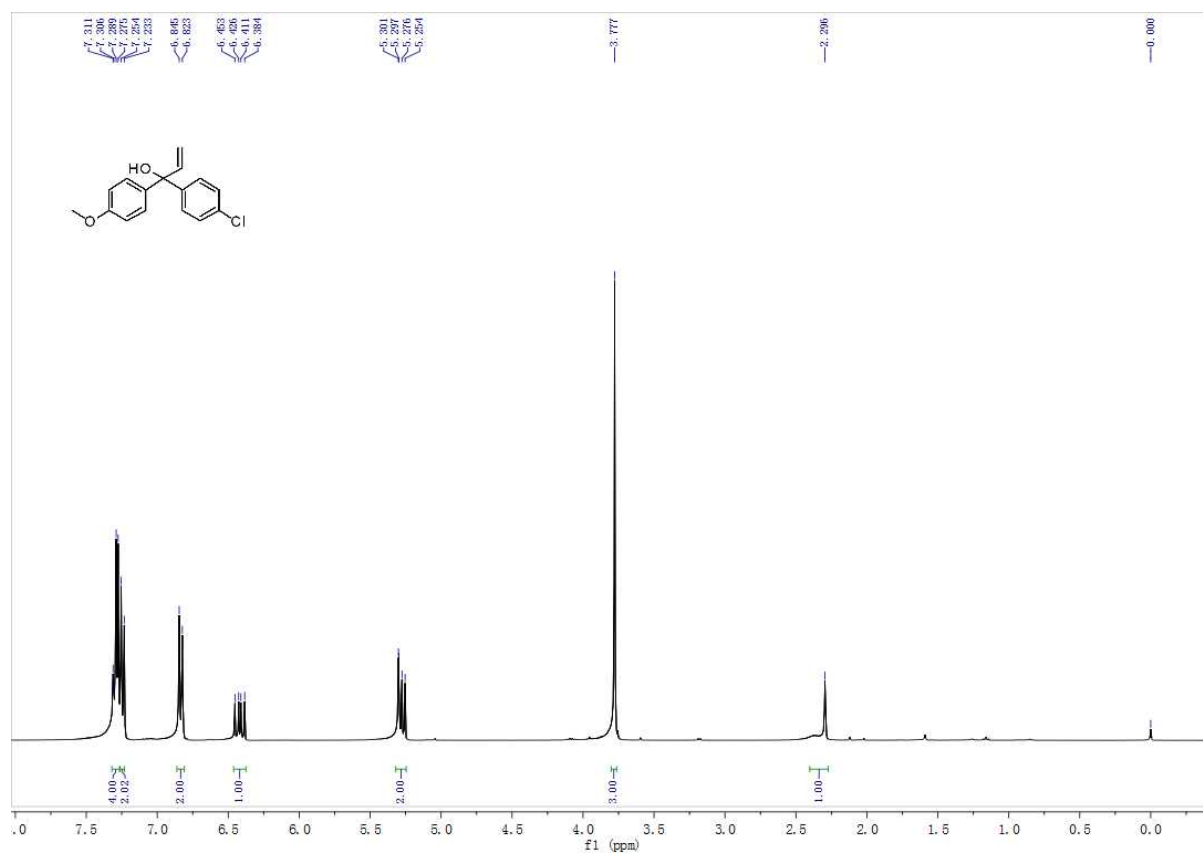
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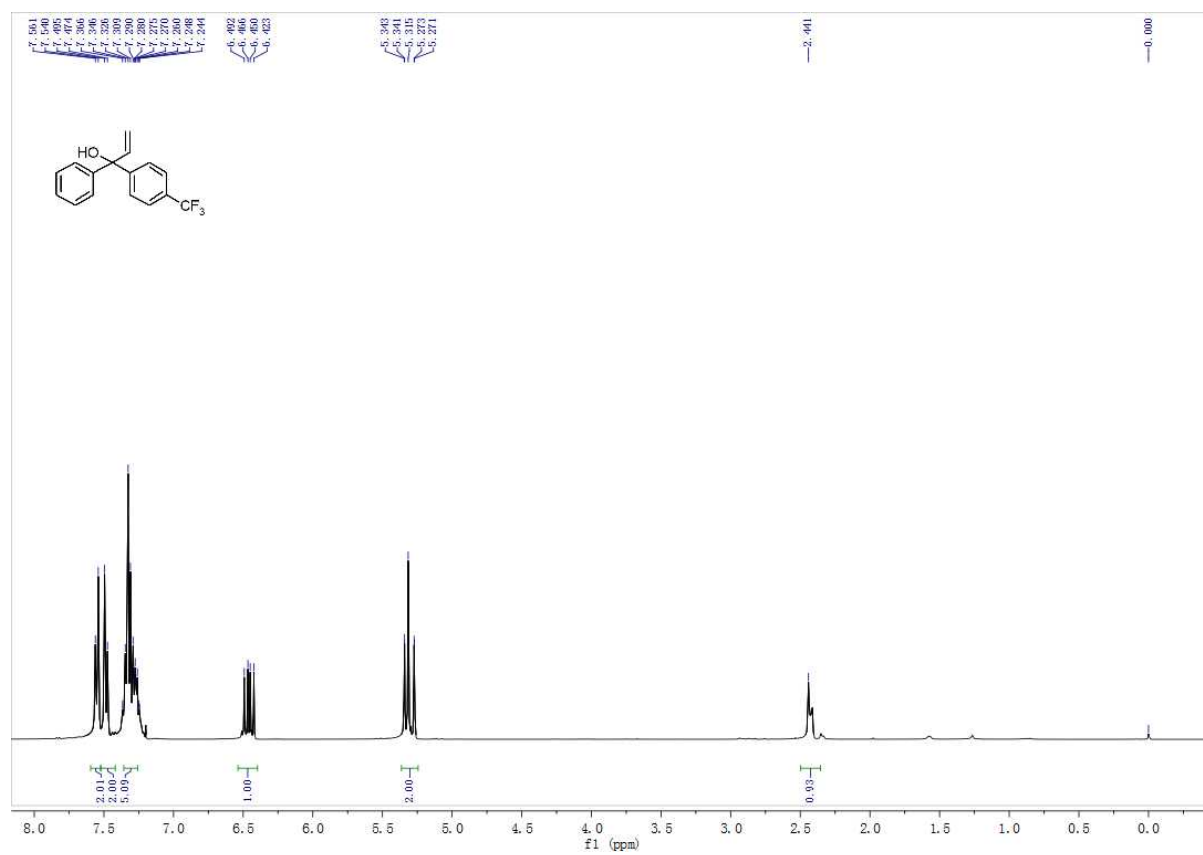
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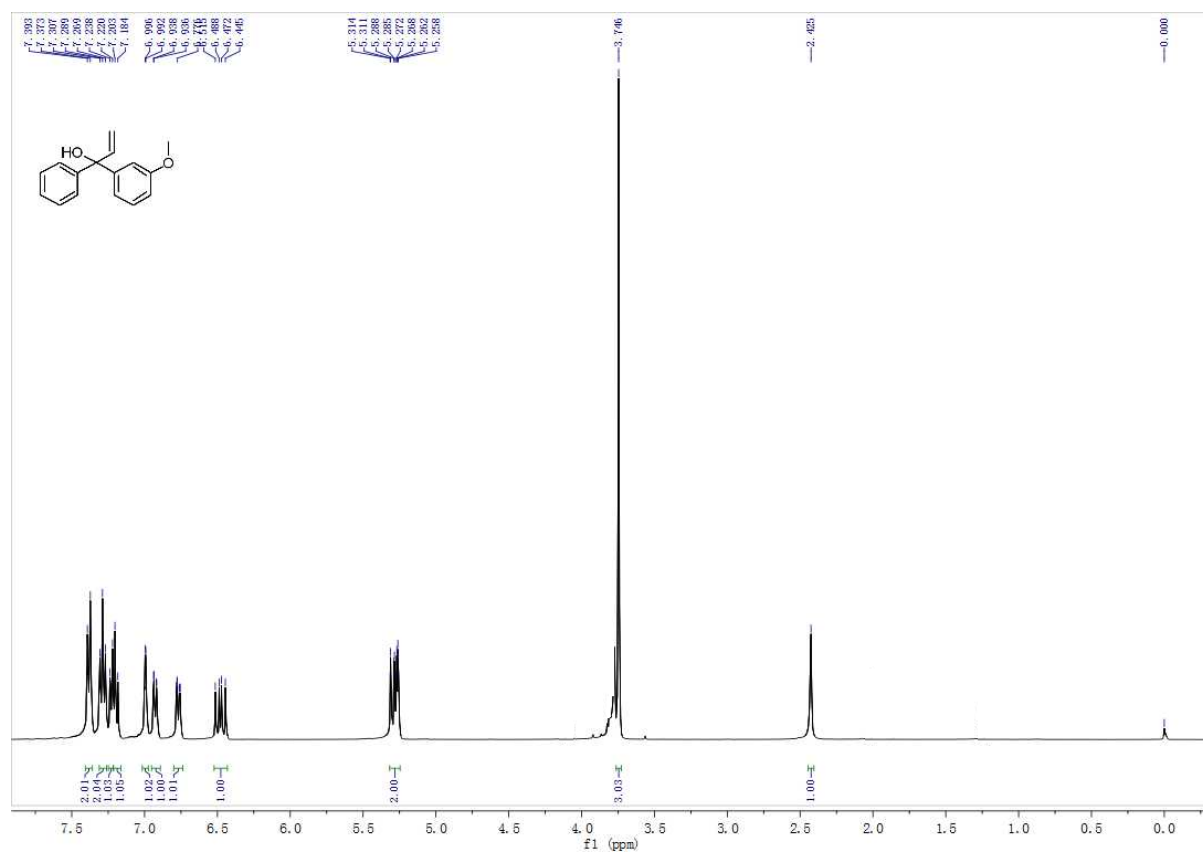
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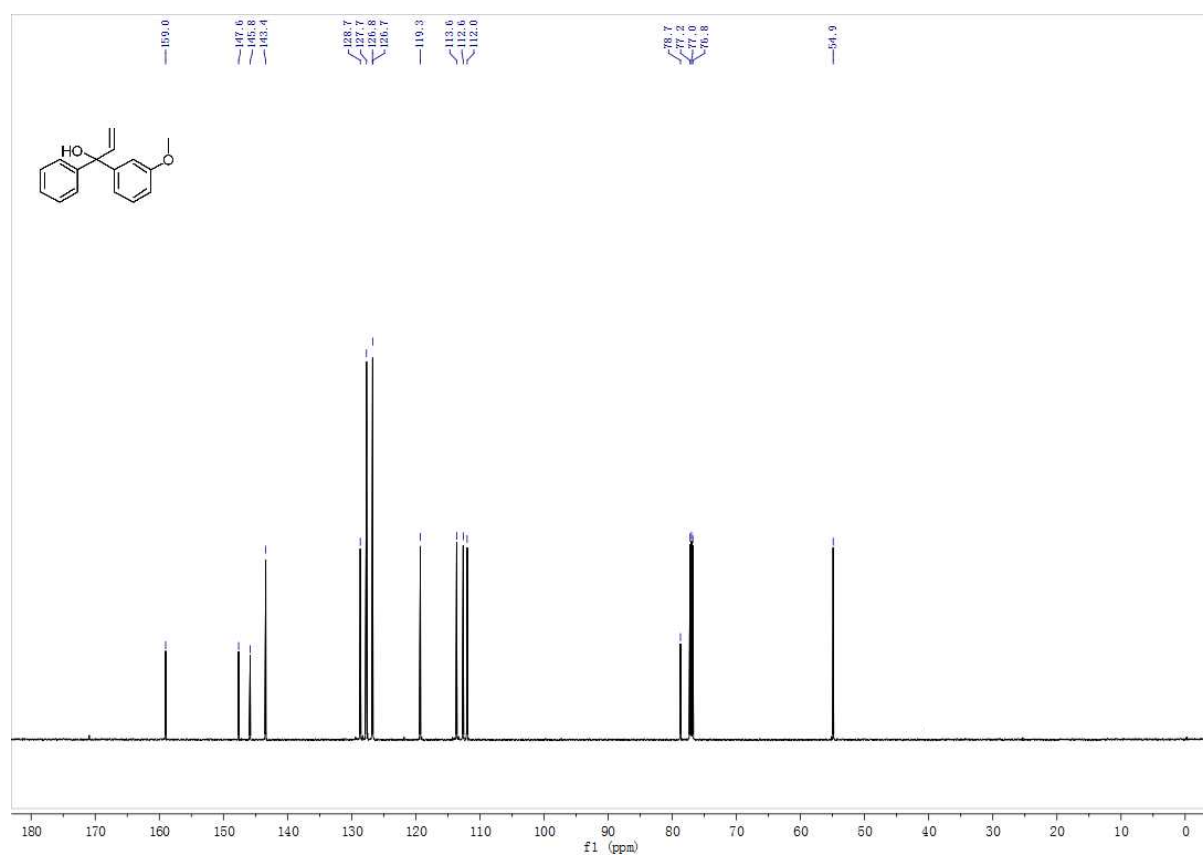
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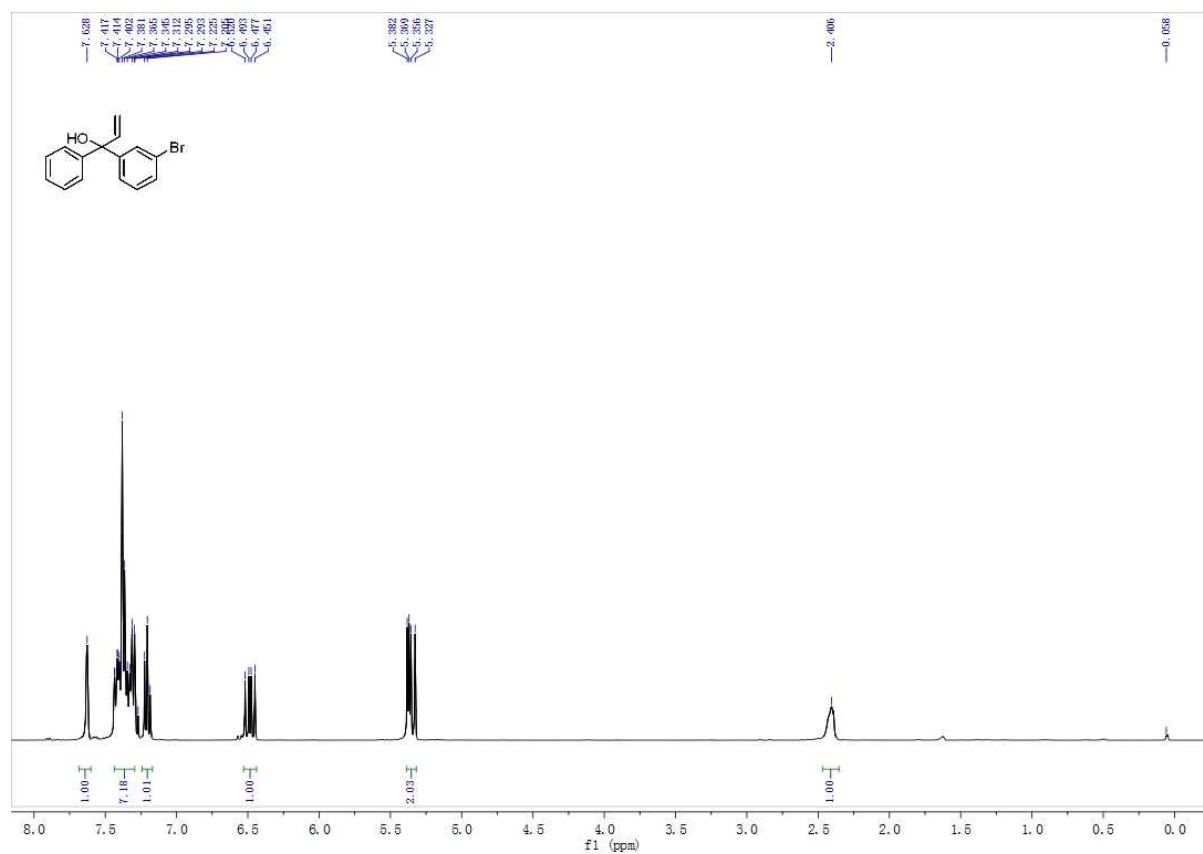
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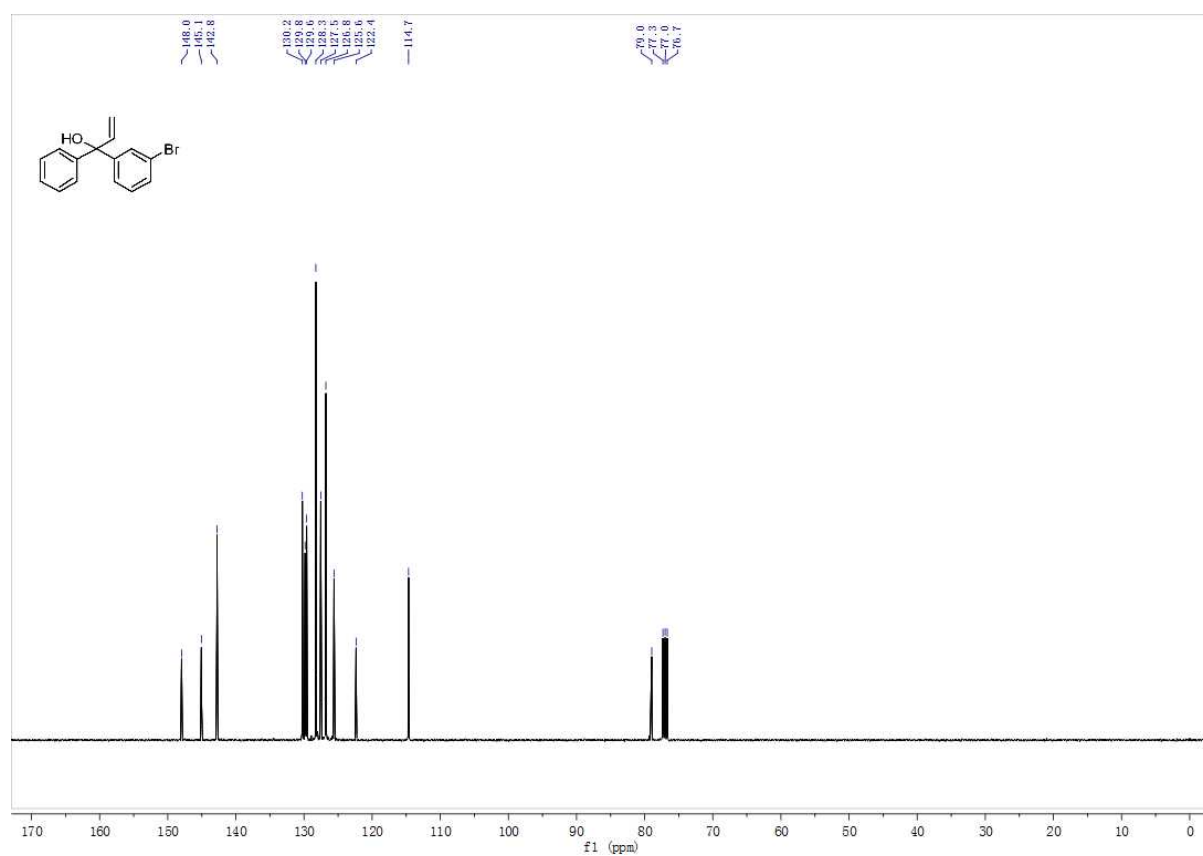
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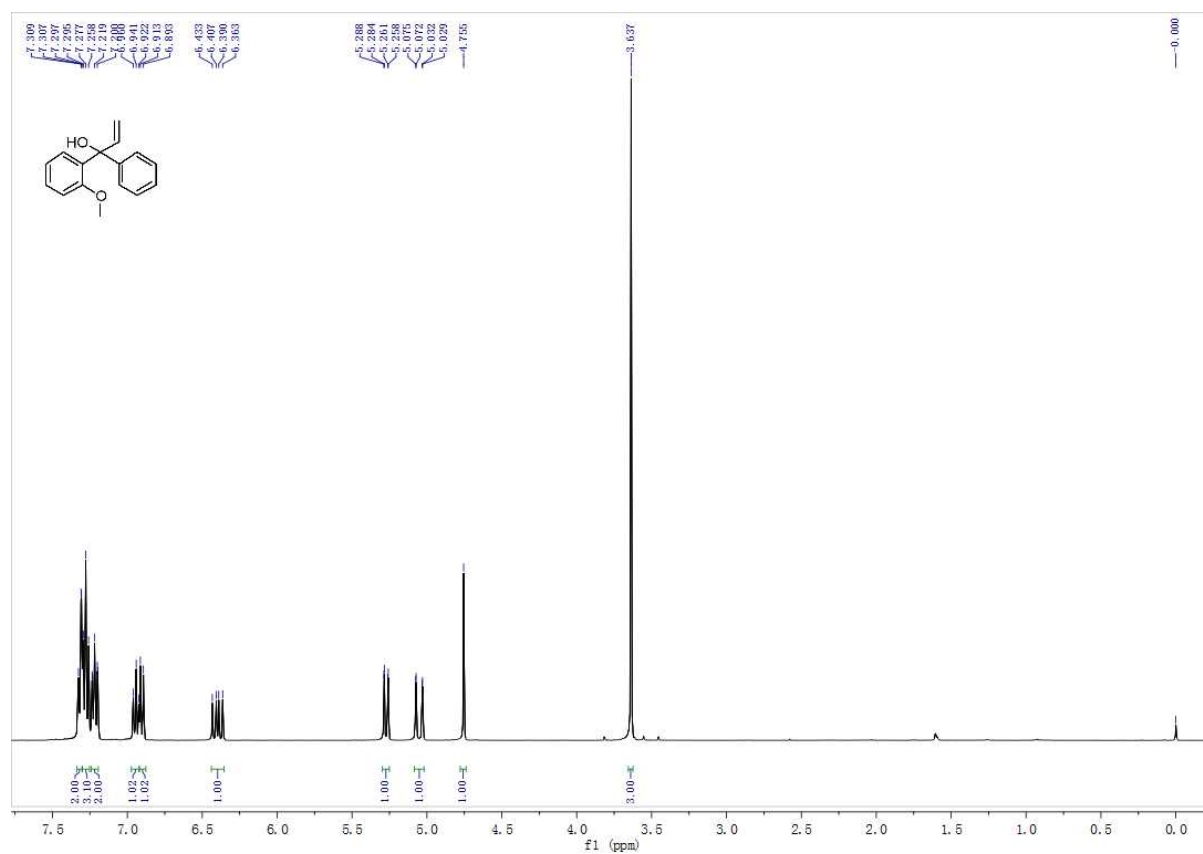
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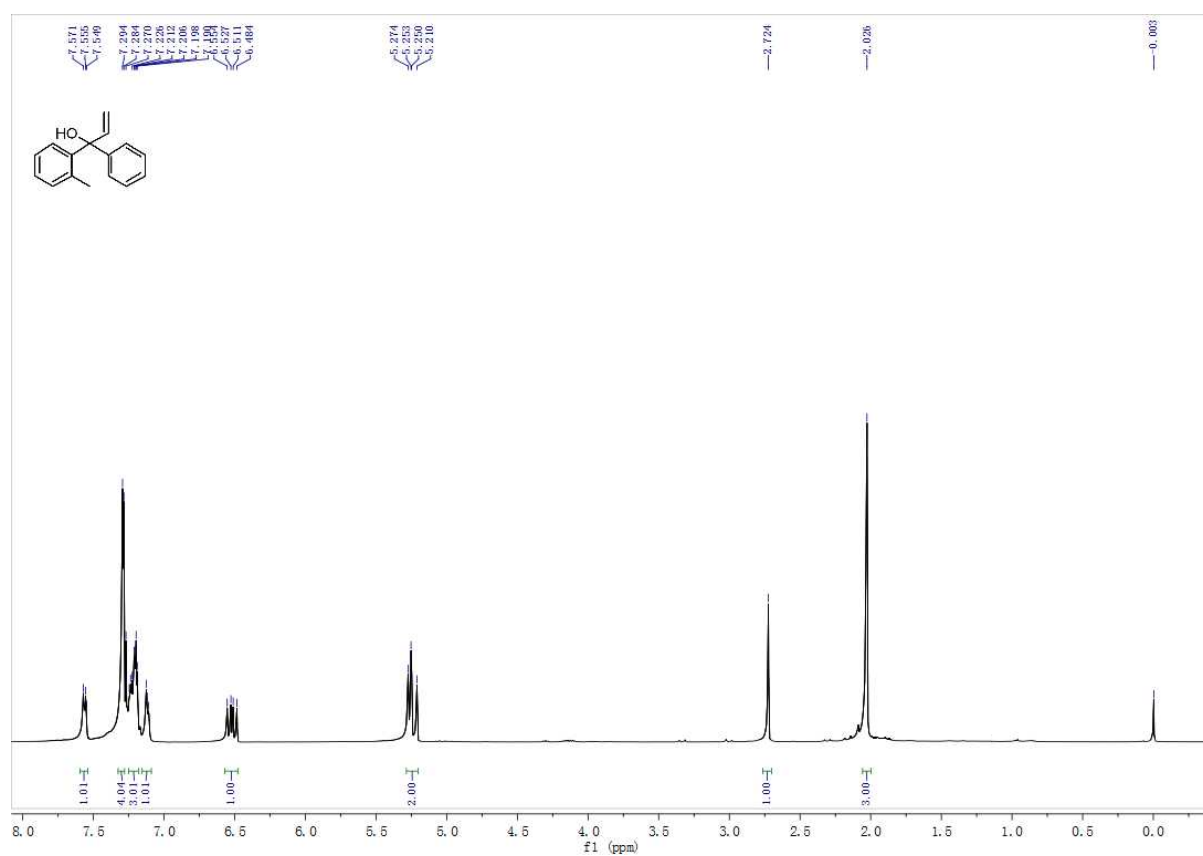
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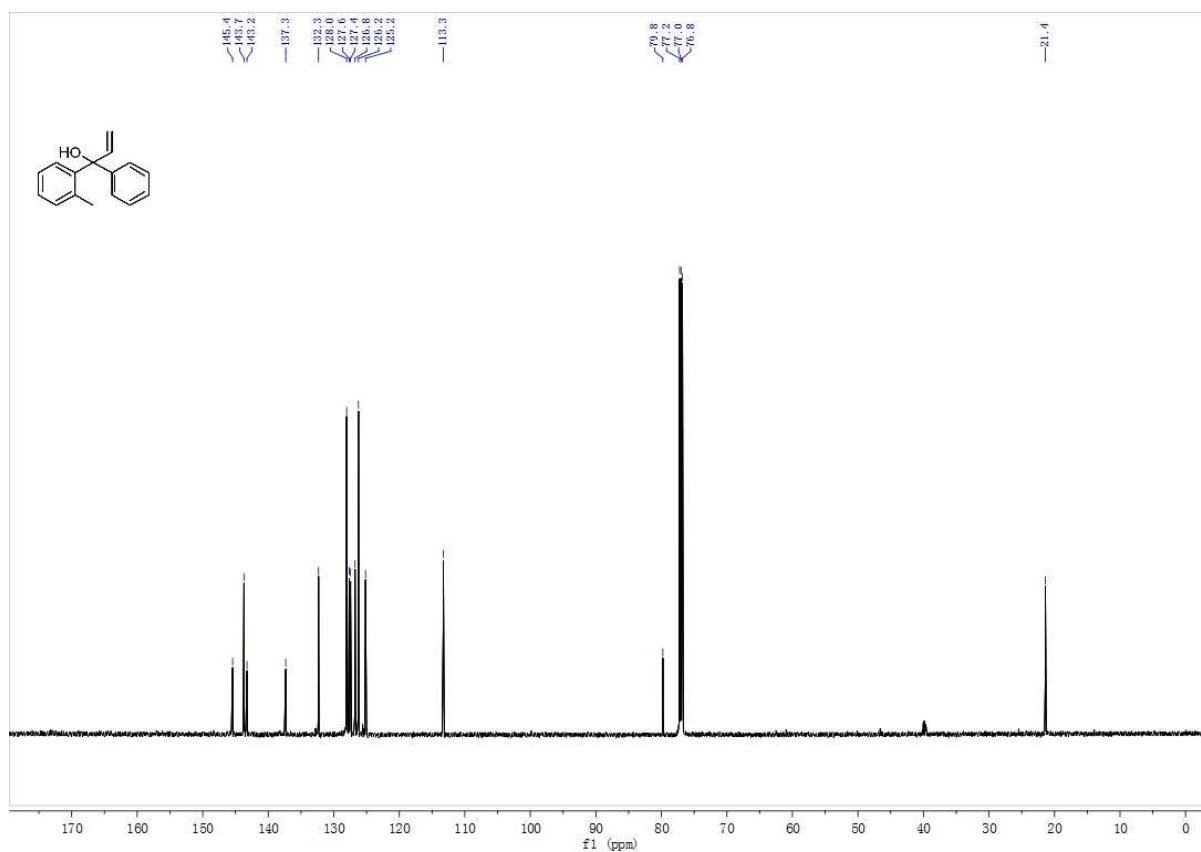
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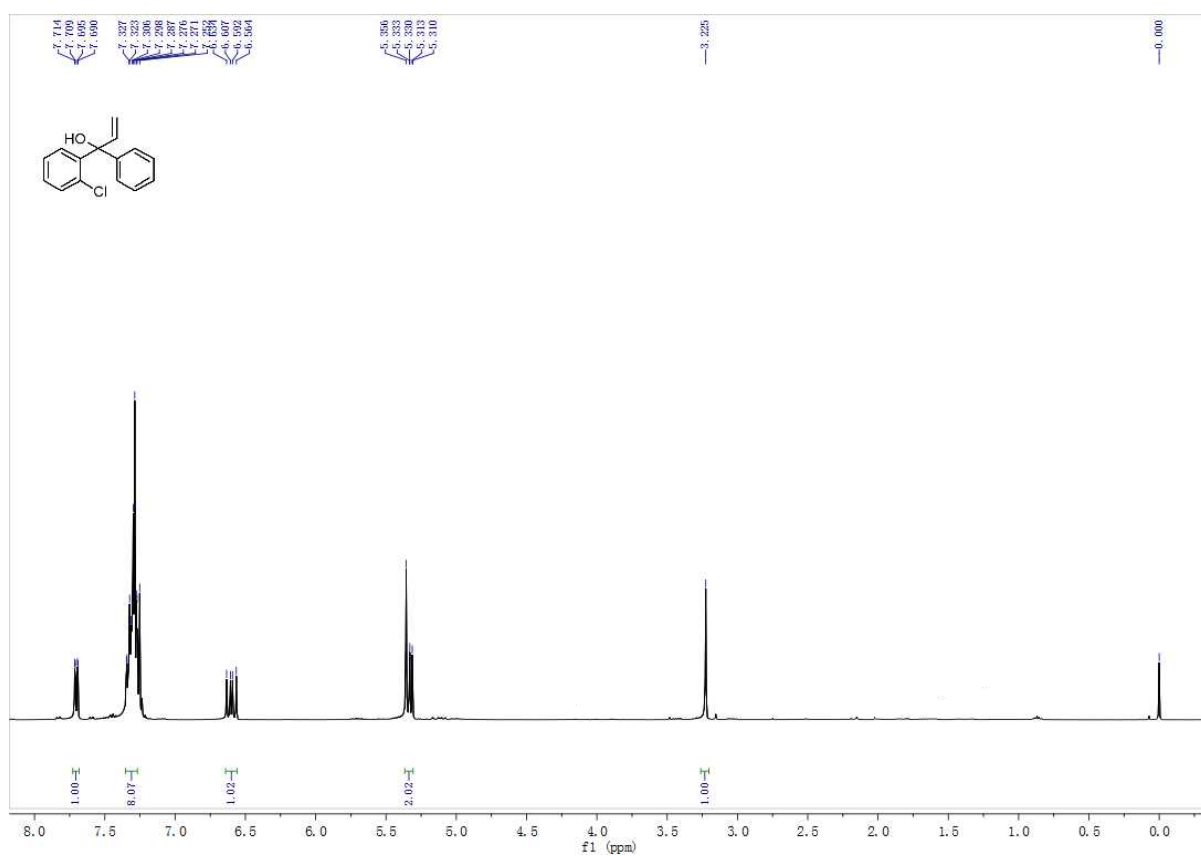
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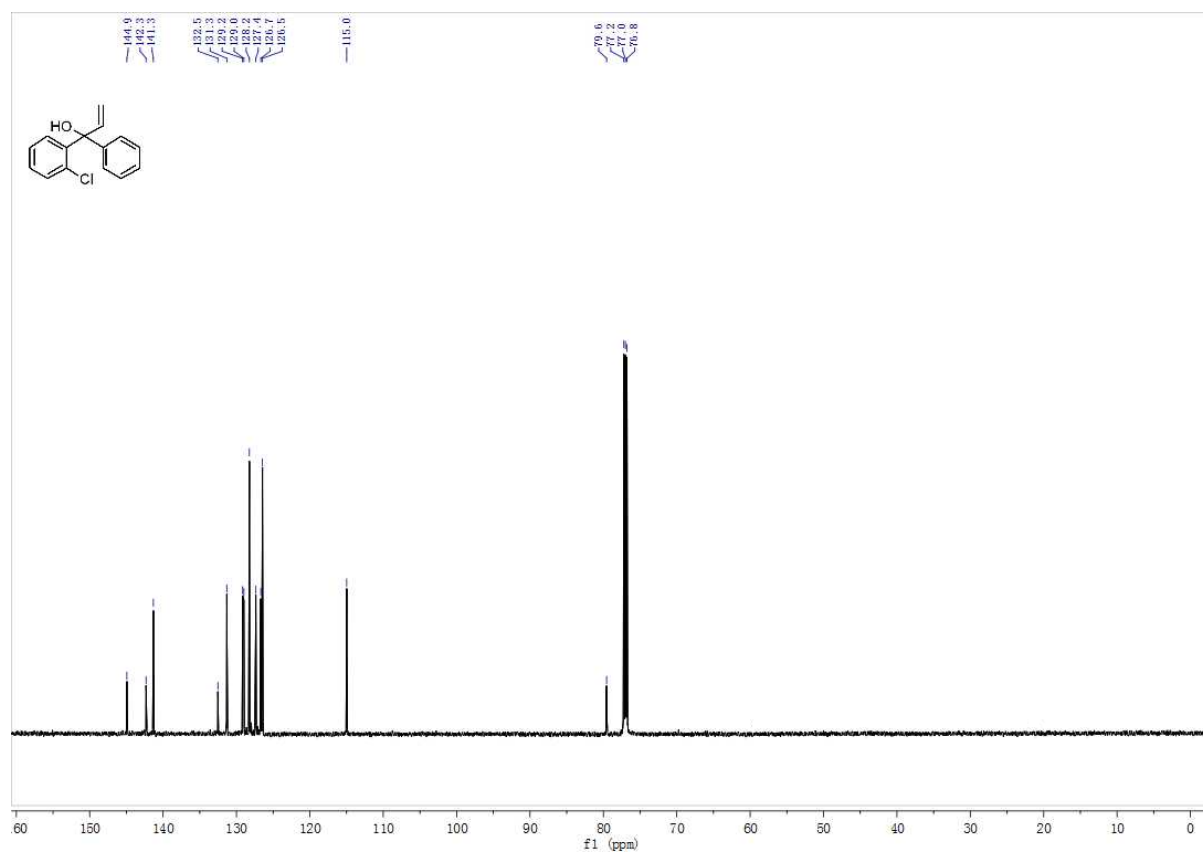
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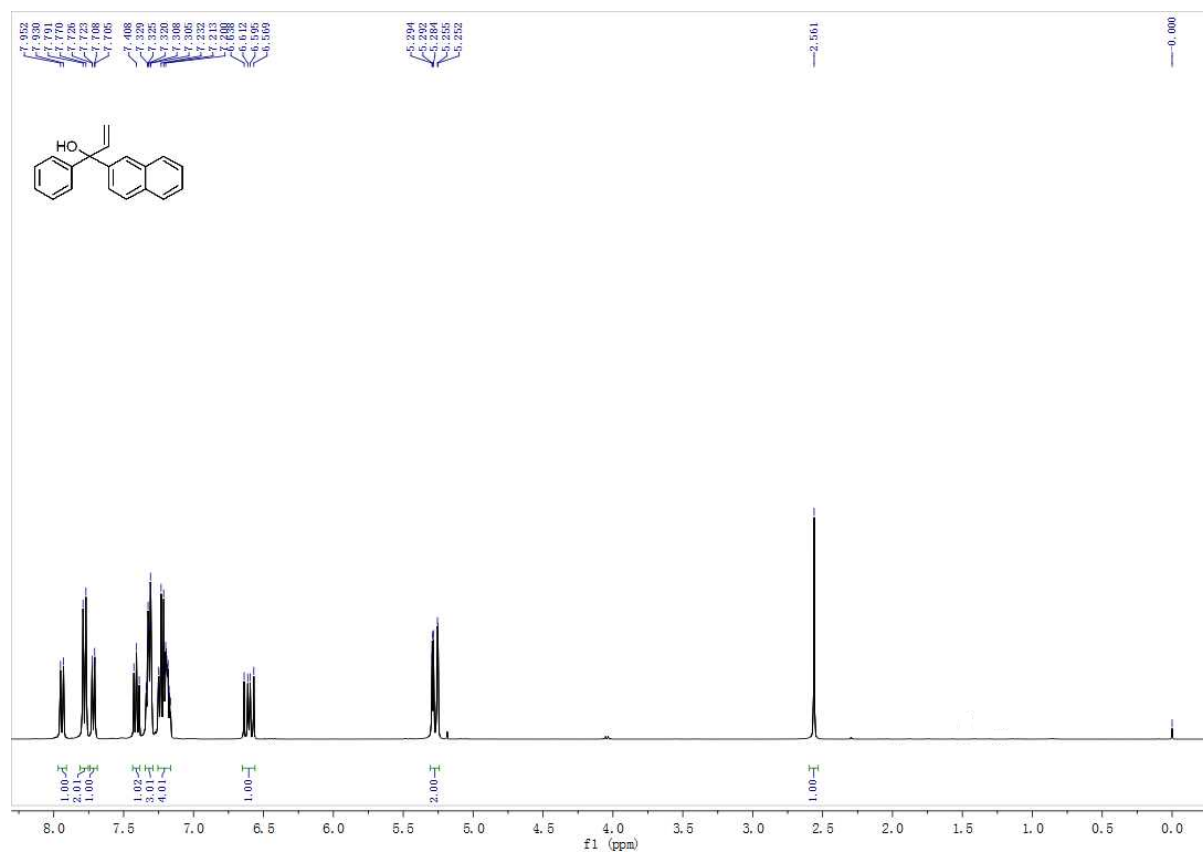
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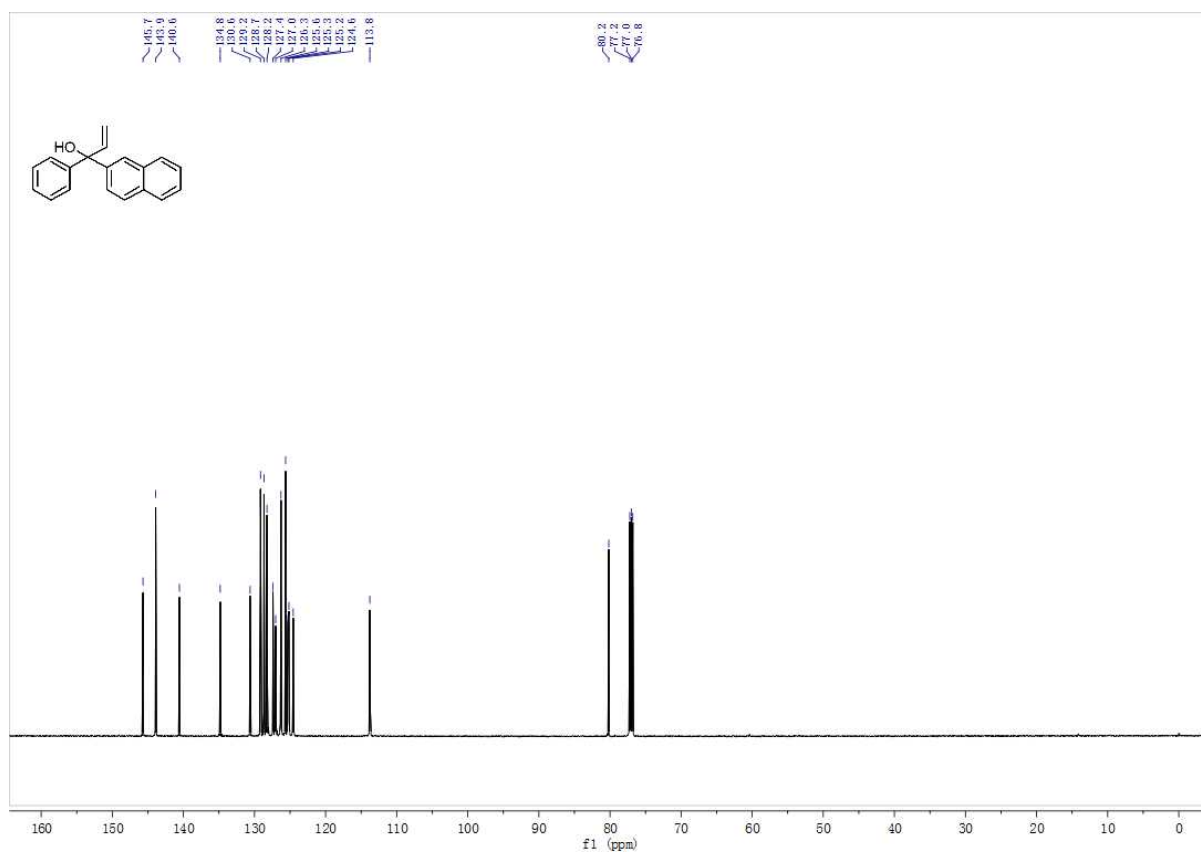
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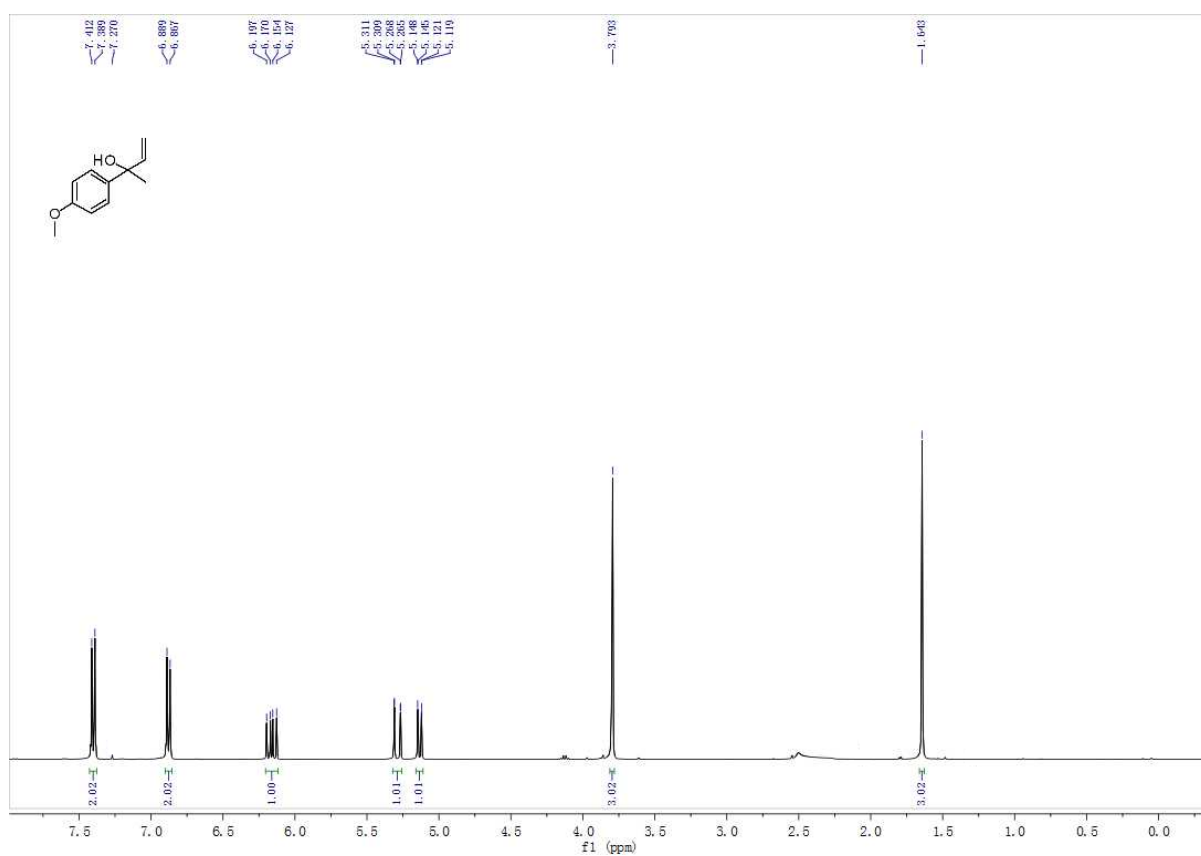
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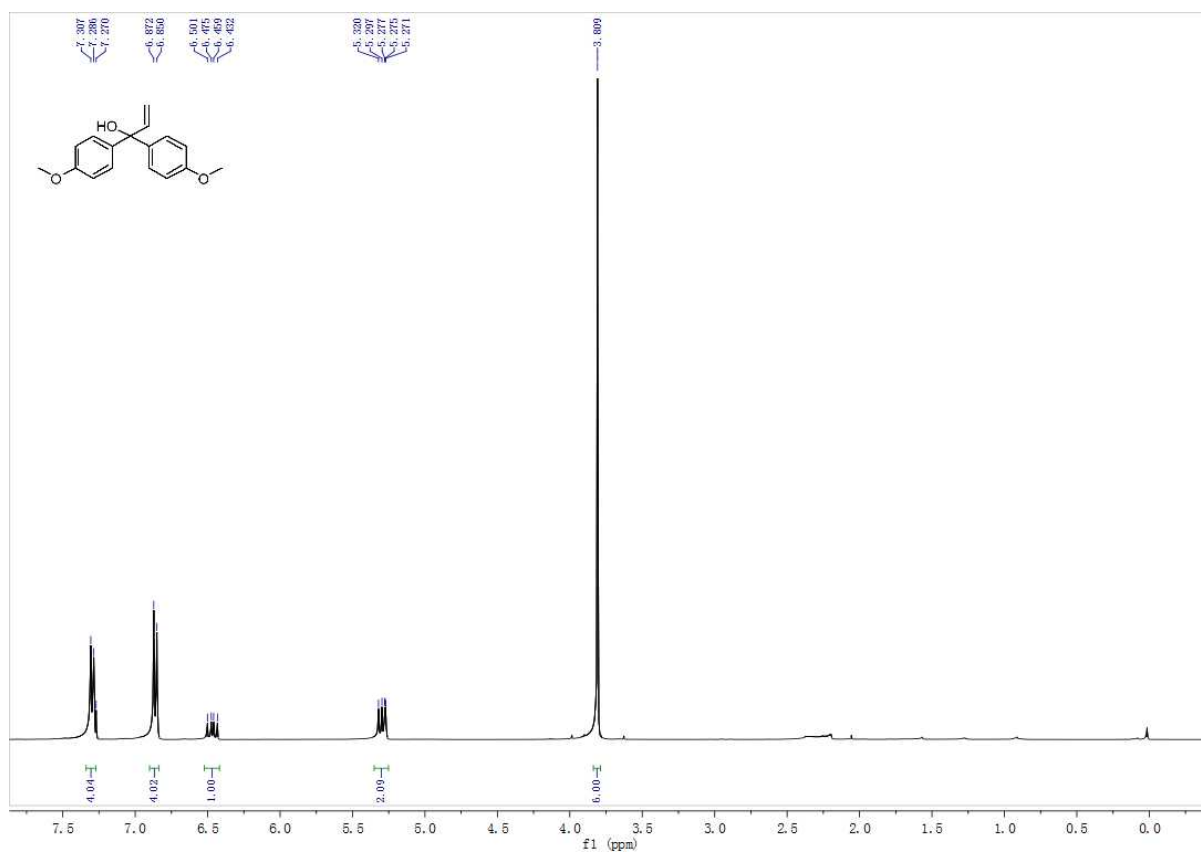
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1s ¹H NMR

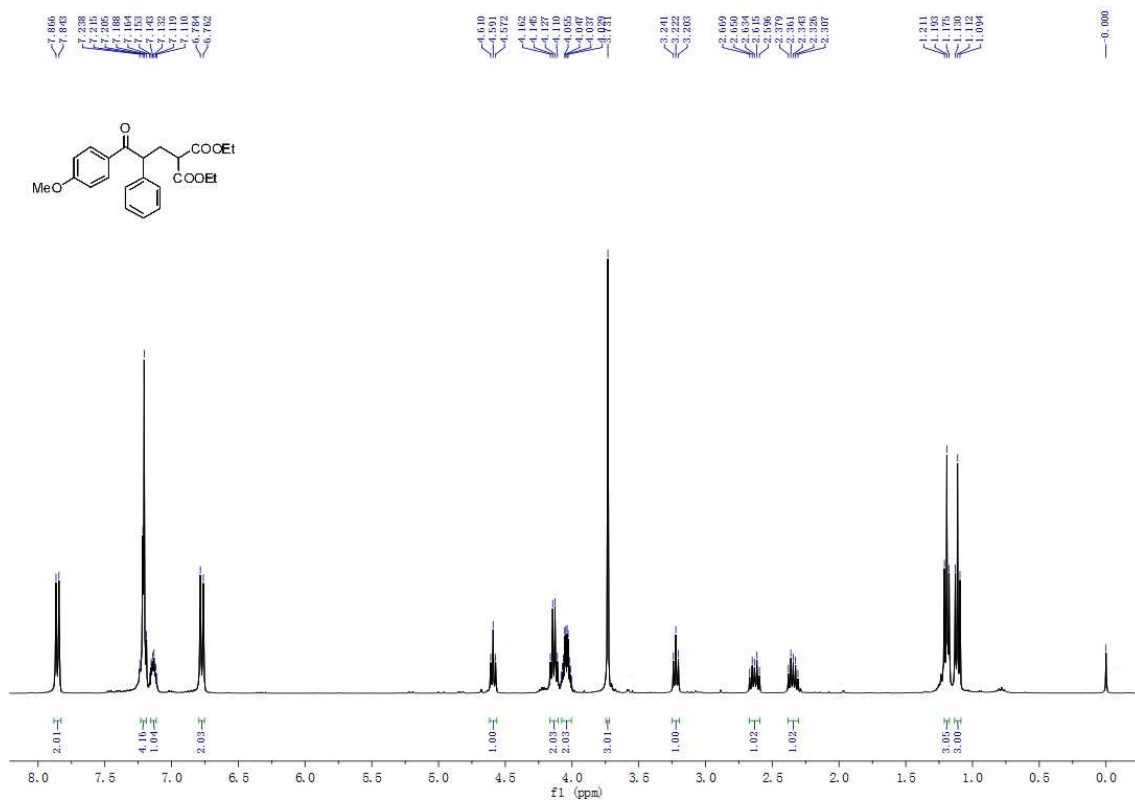


1t ¹H NMR

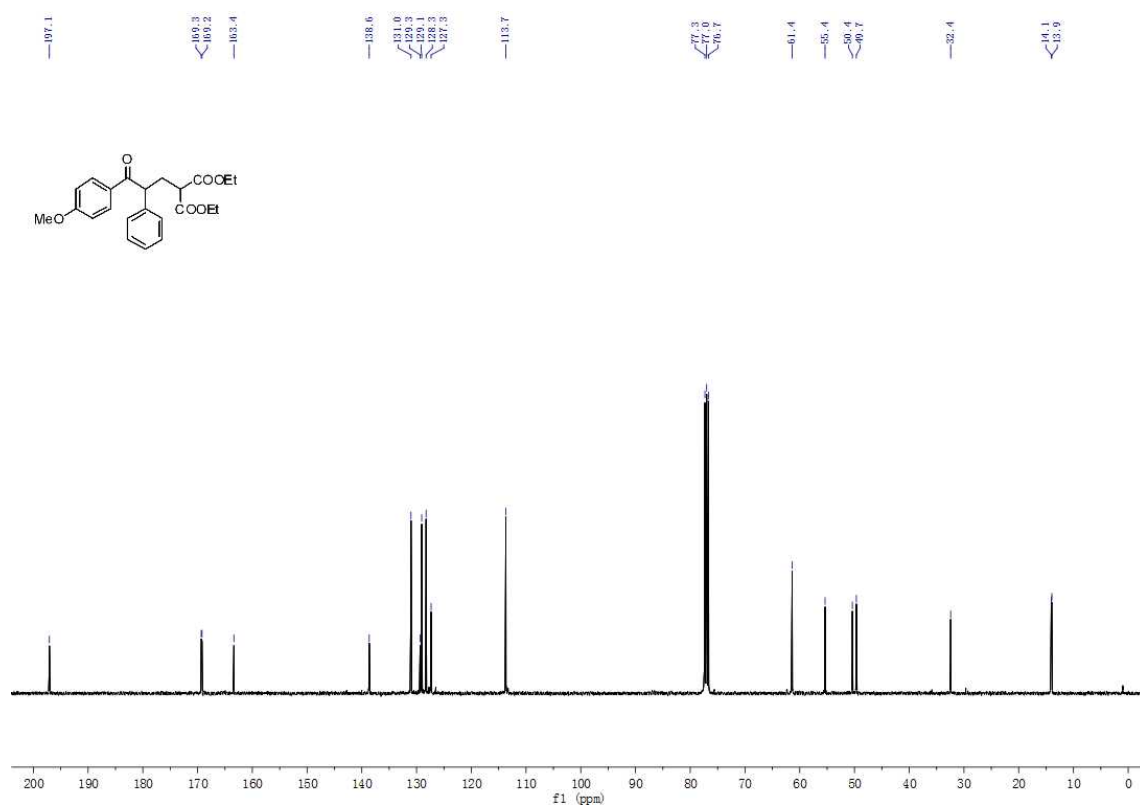


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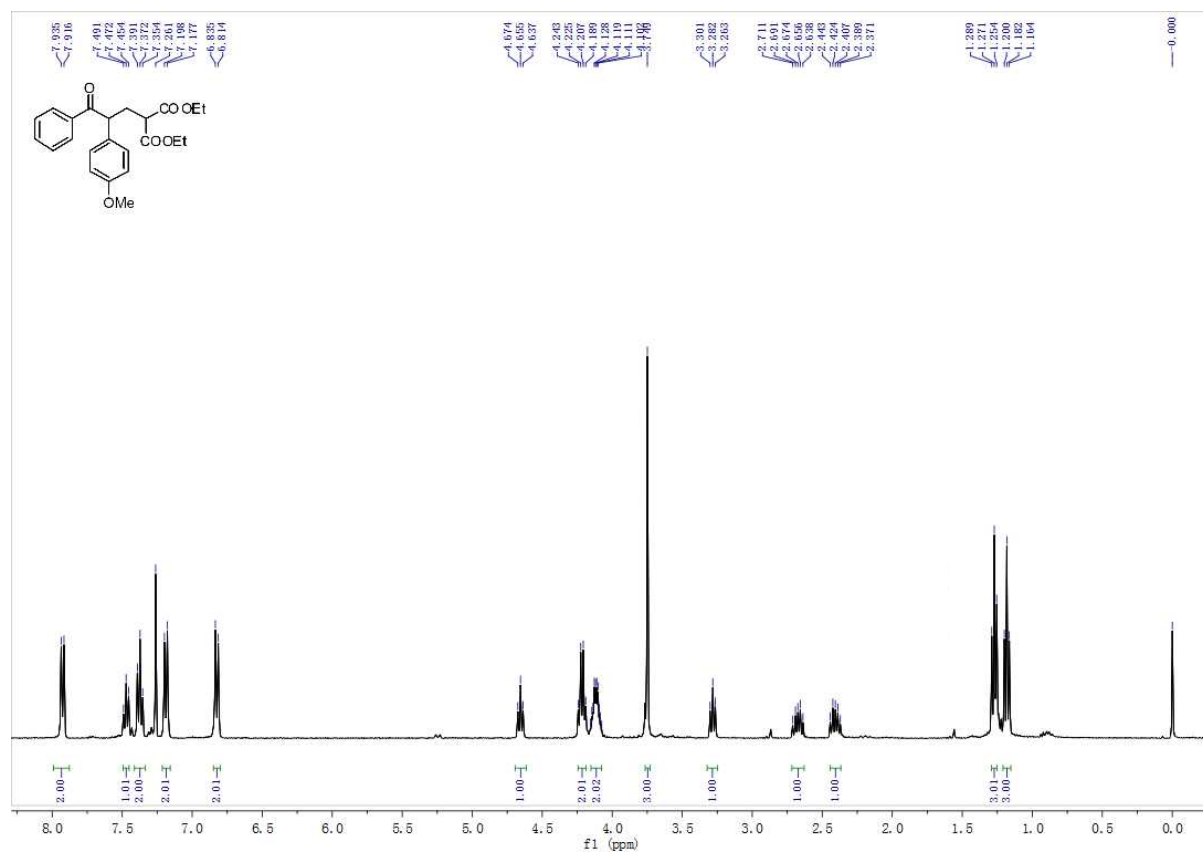
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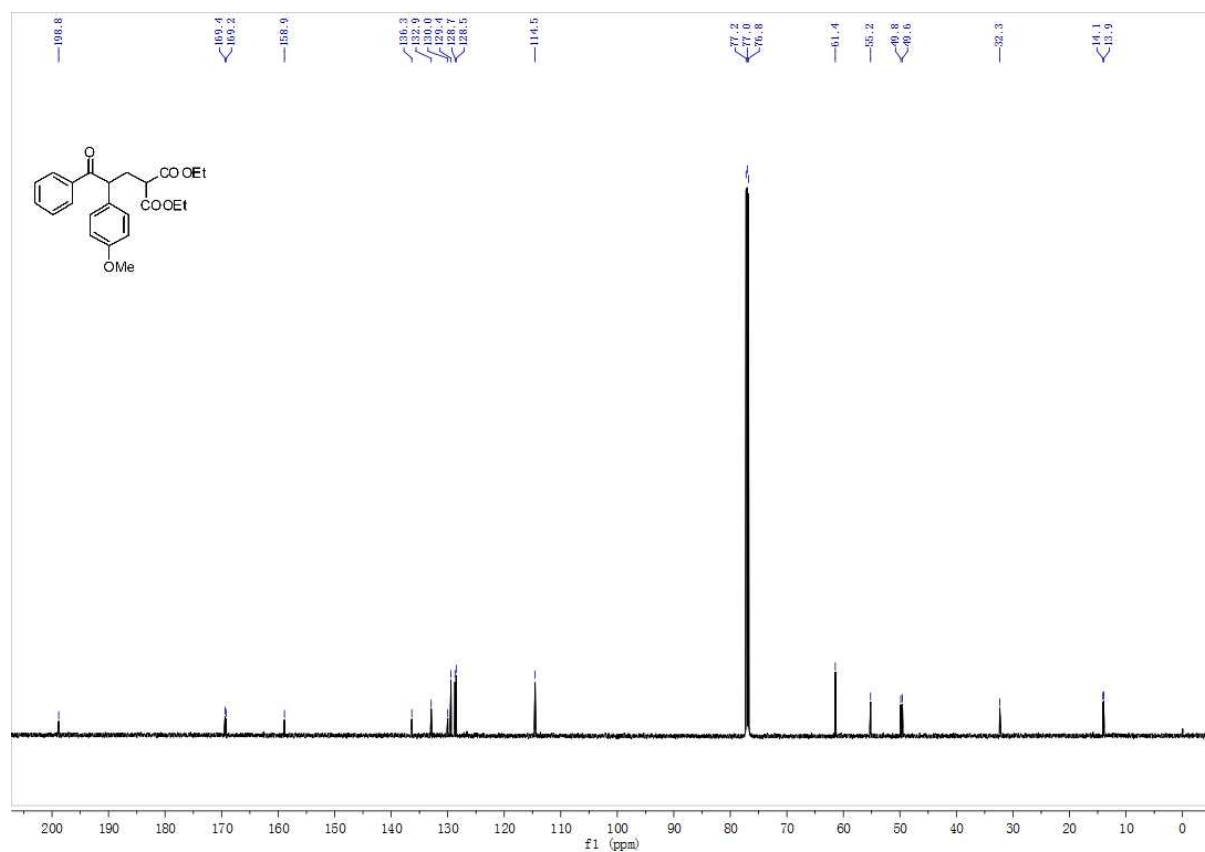
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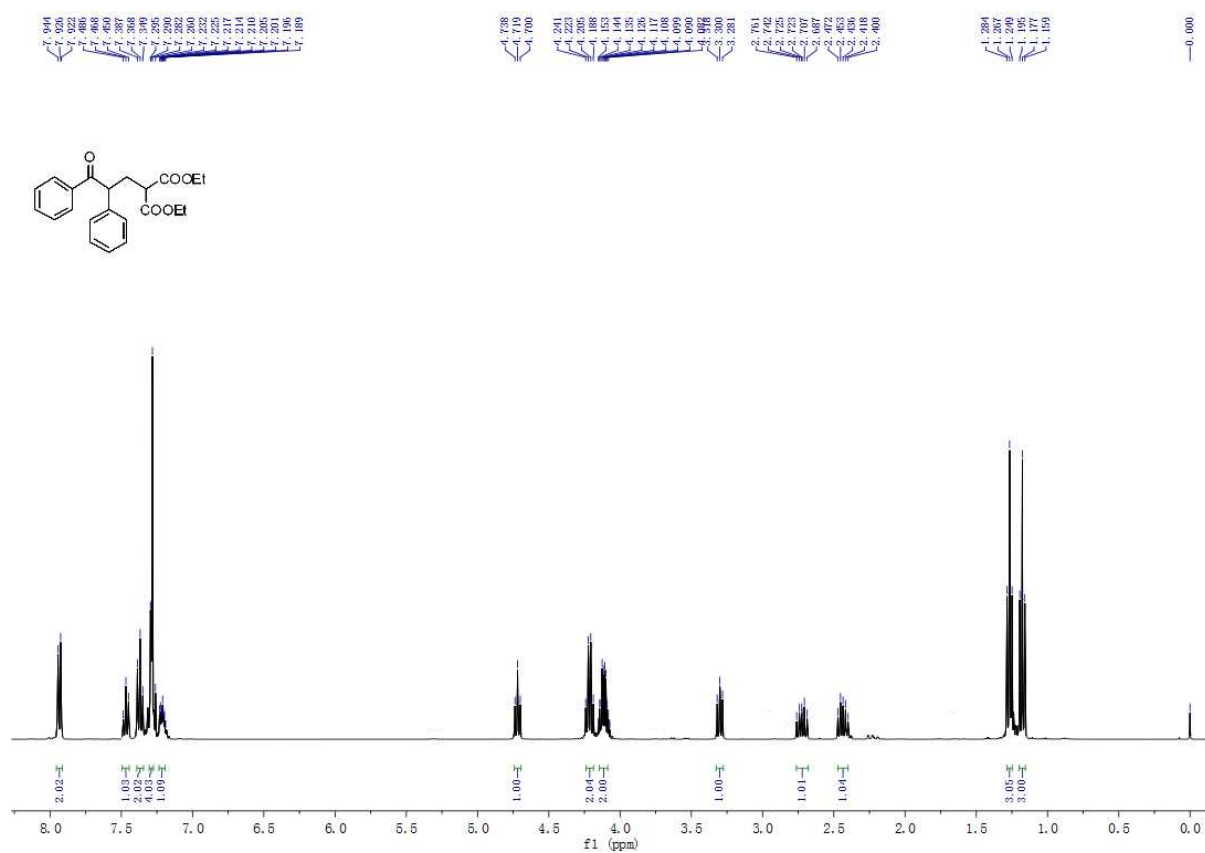
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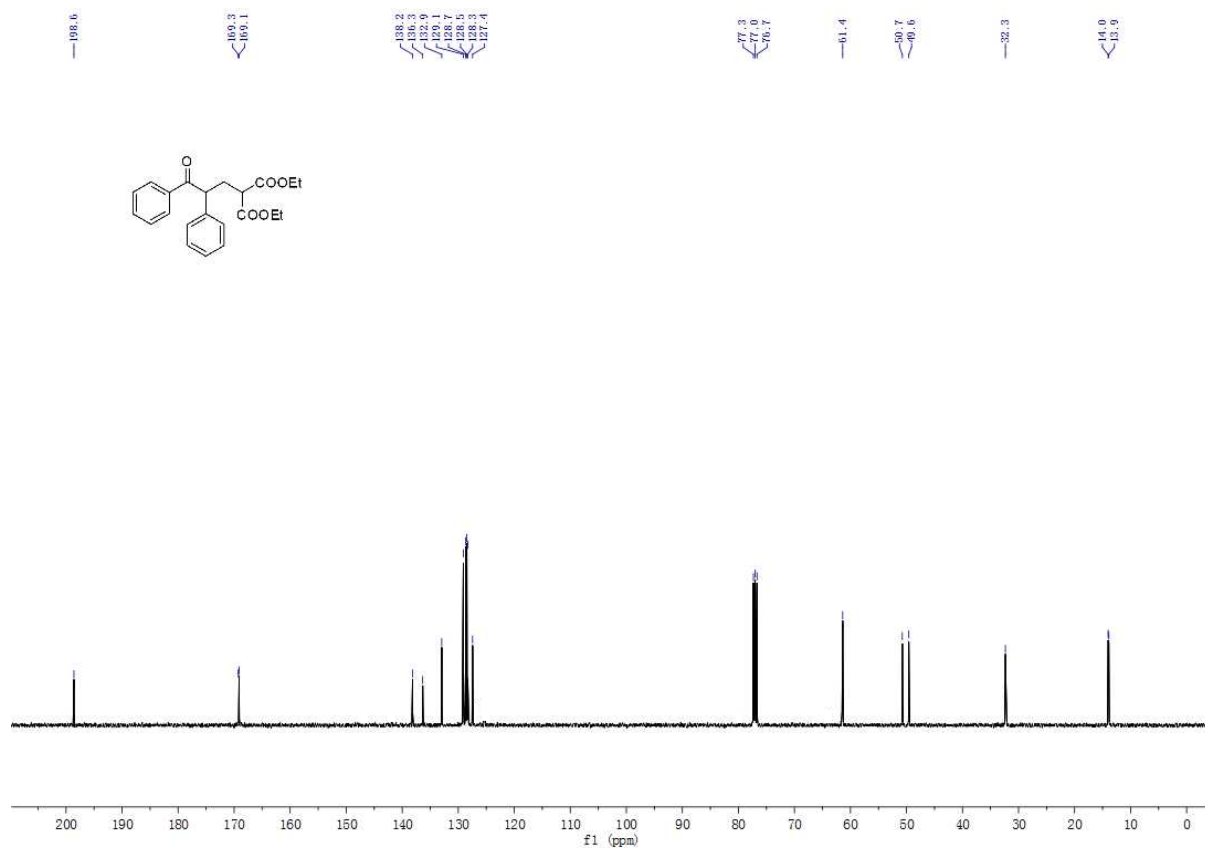
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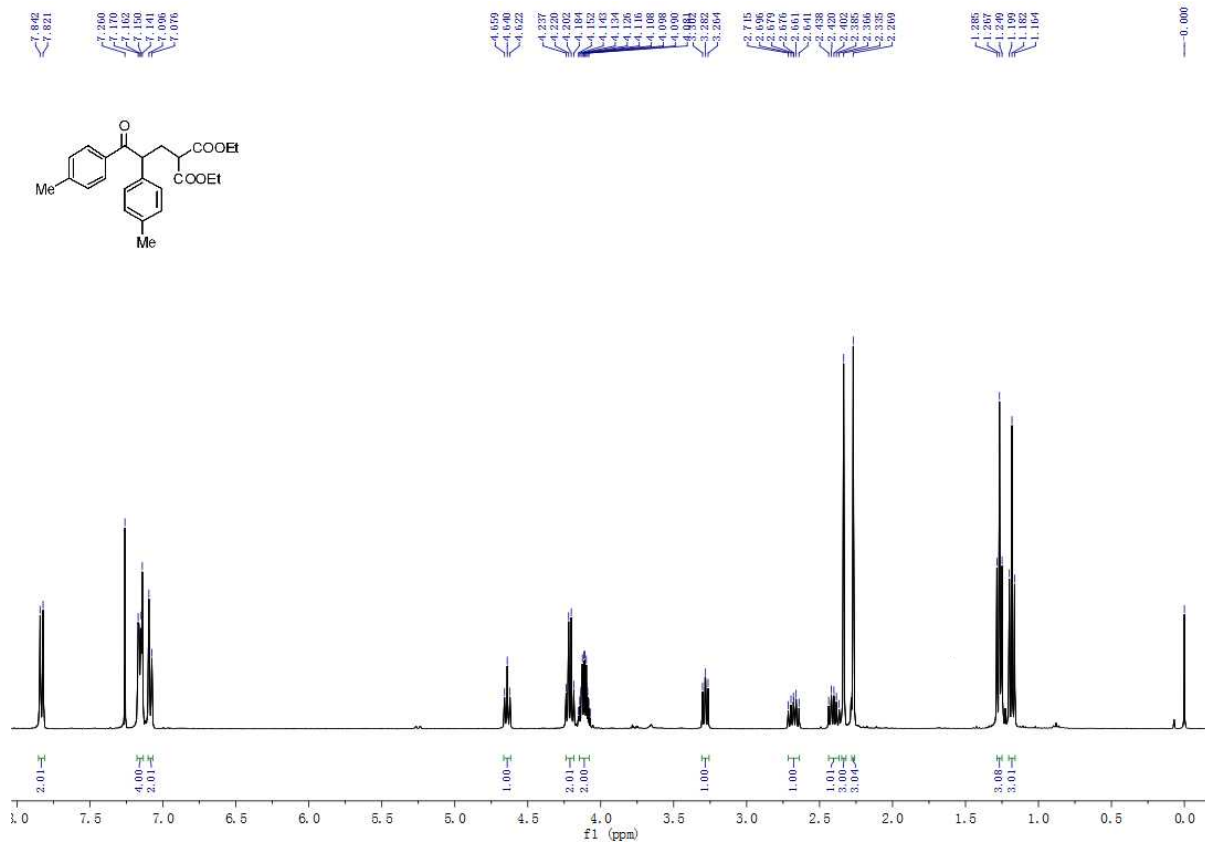
3b ¹H NMR



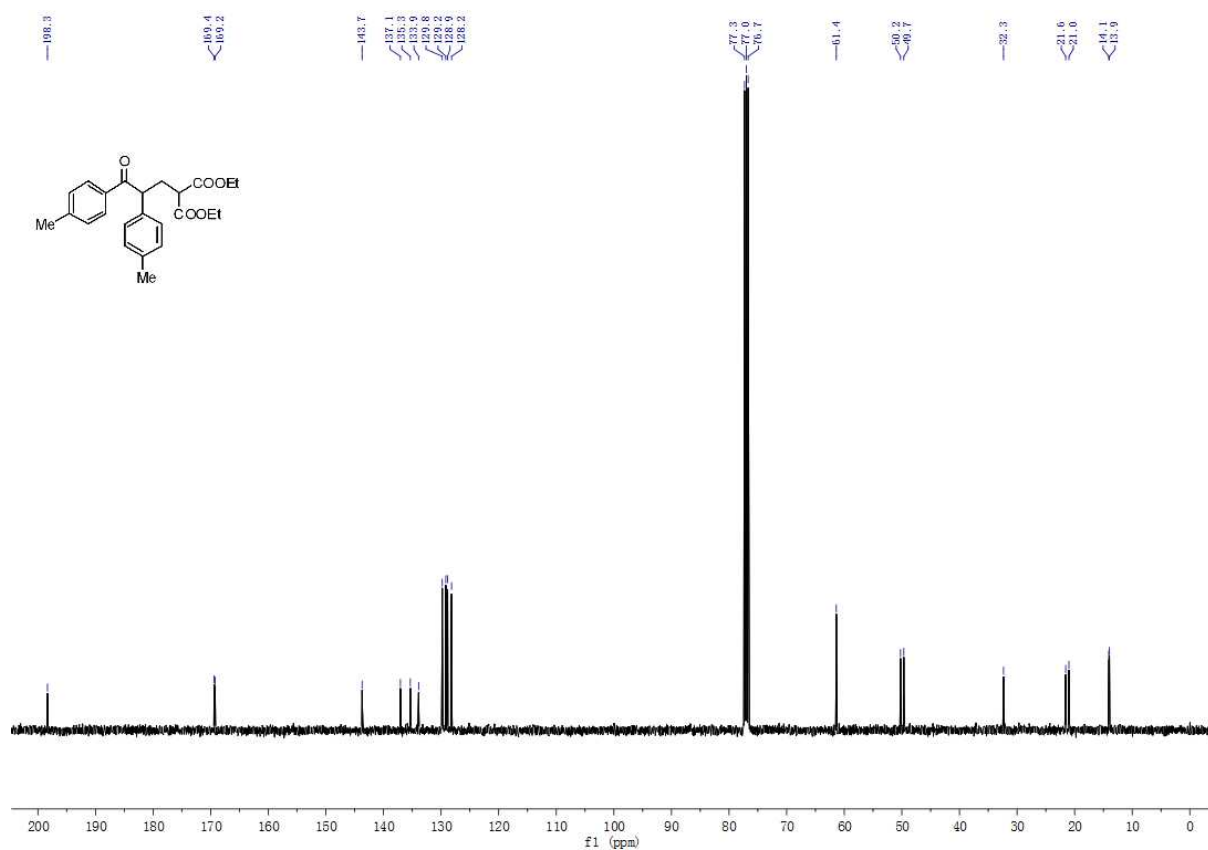
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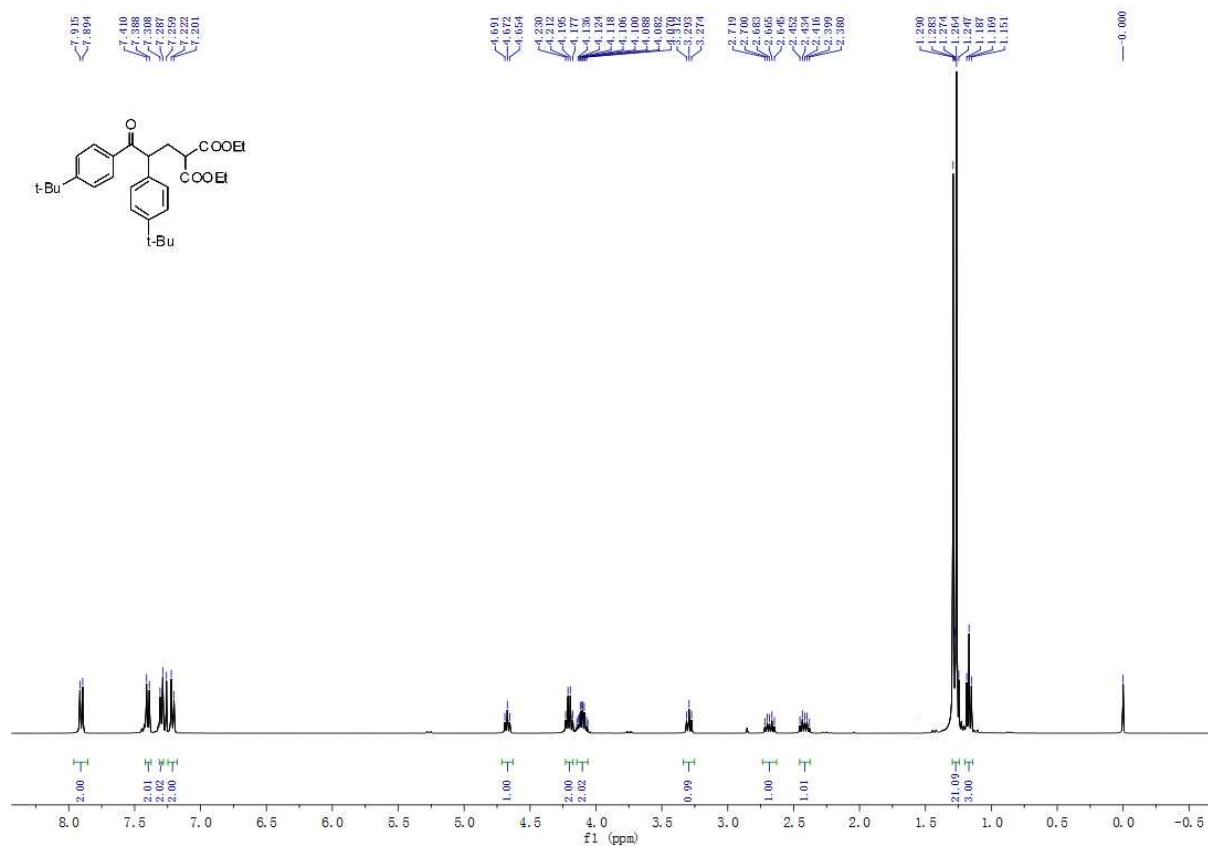
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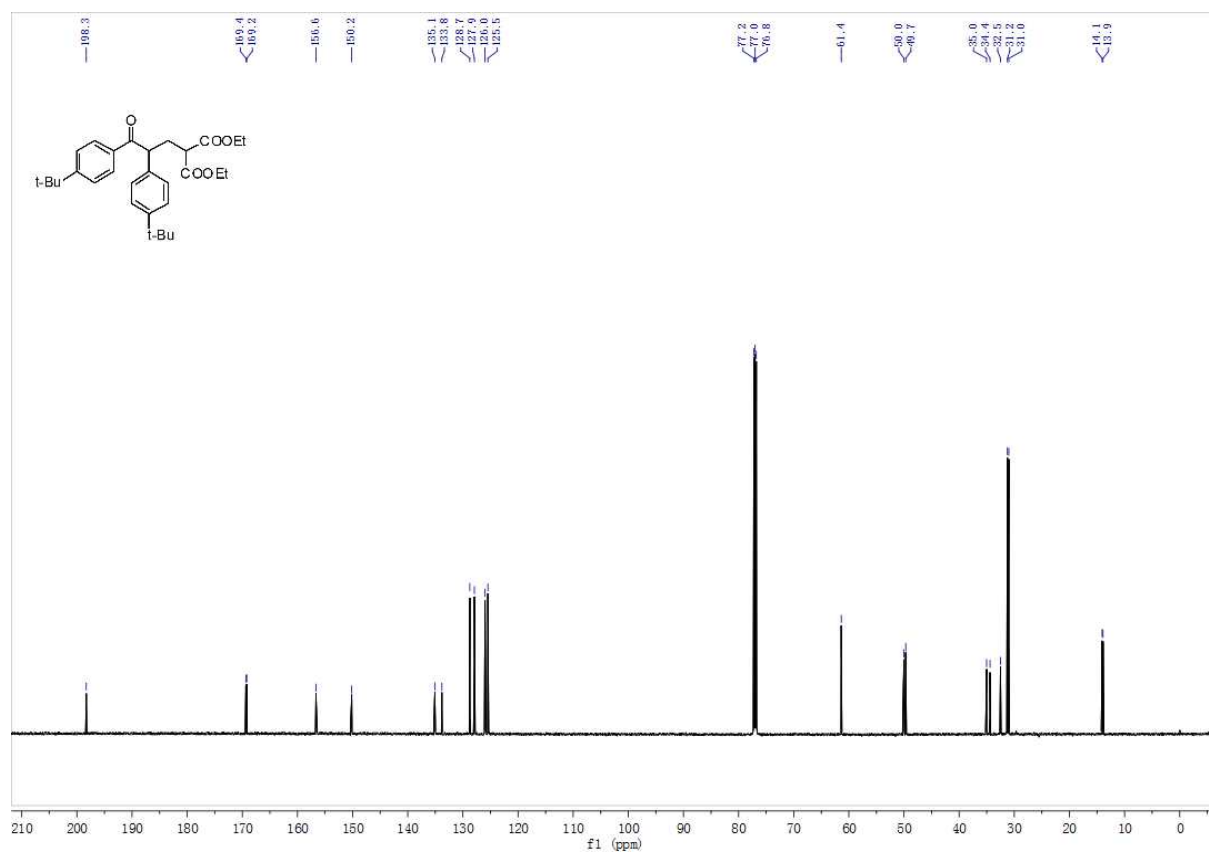
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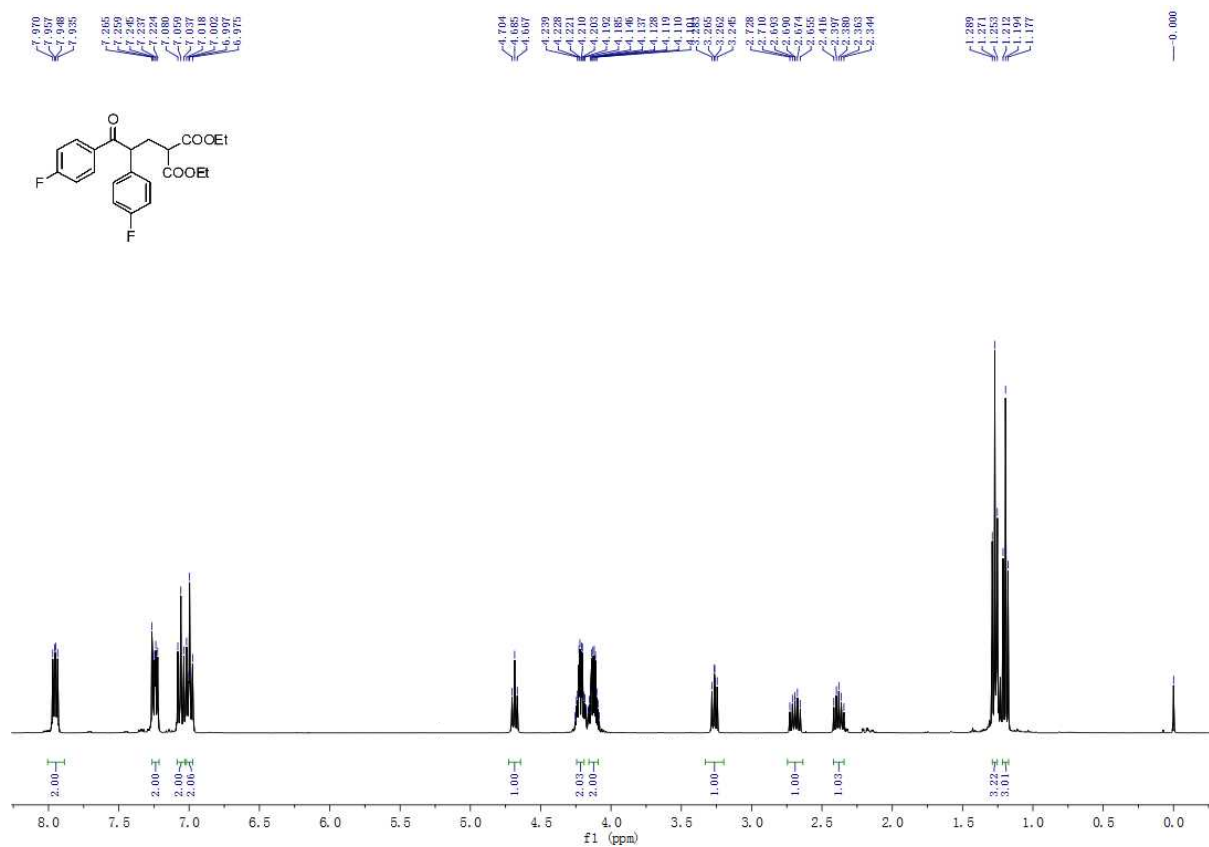
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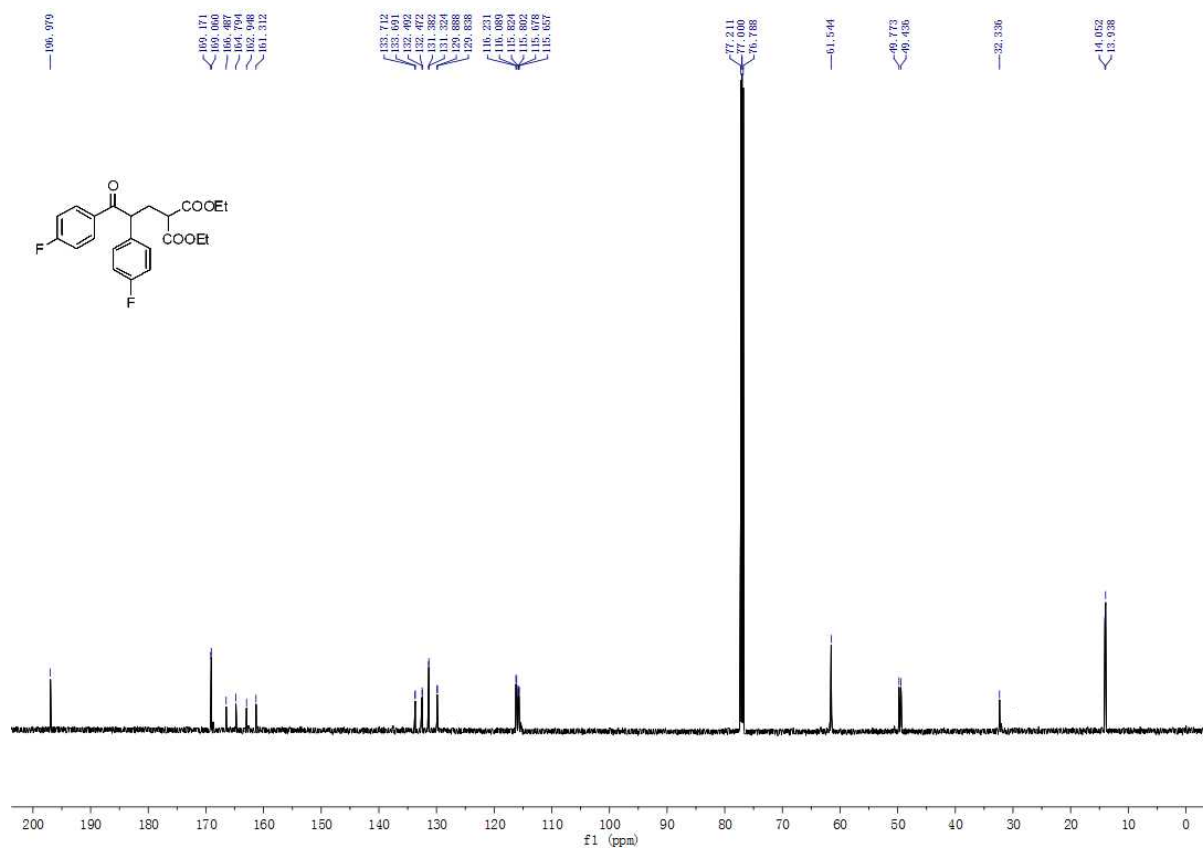
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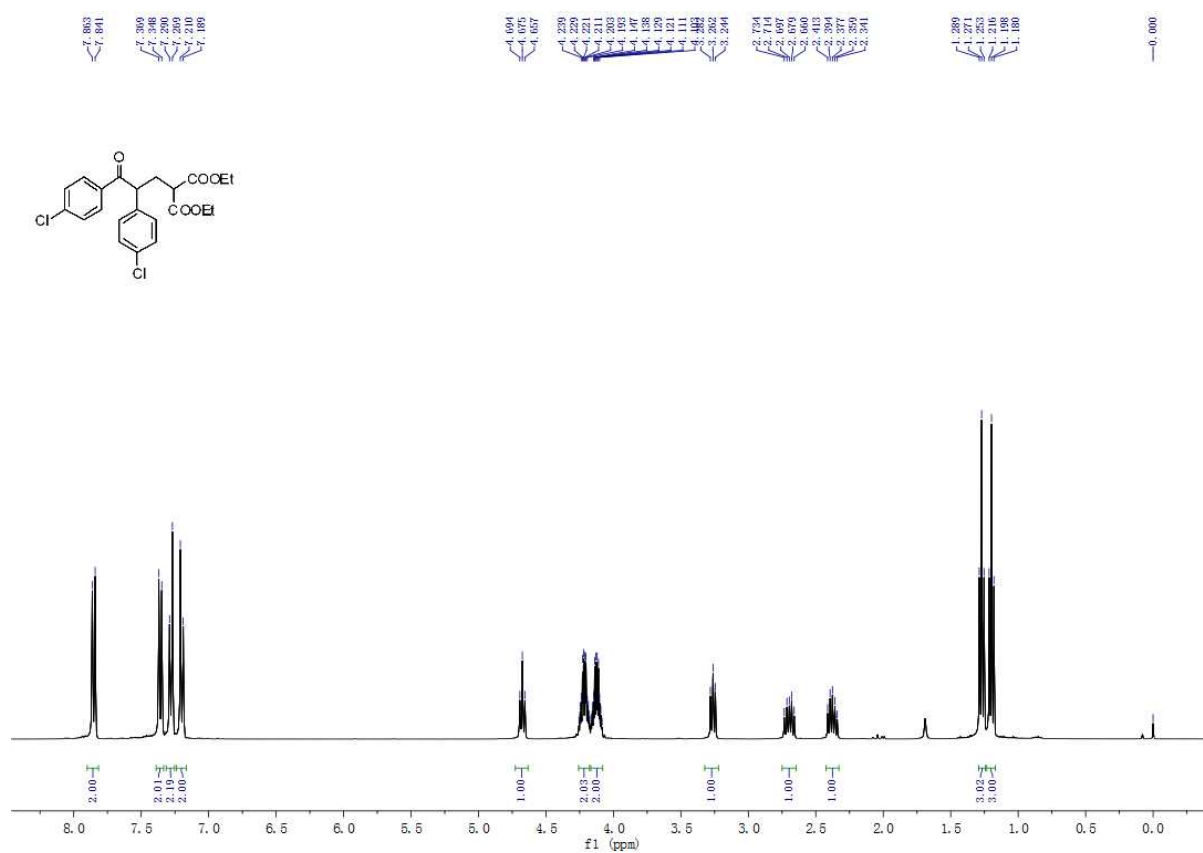
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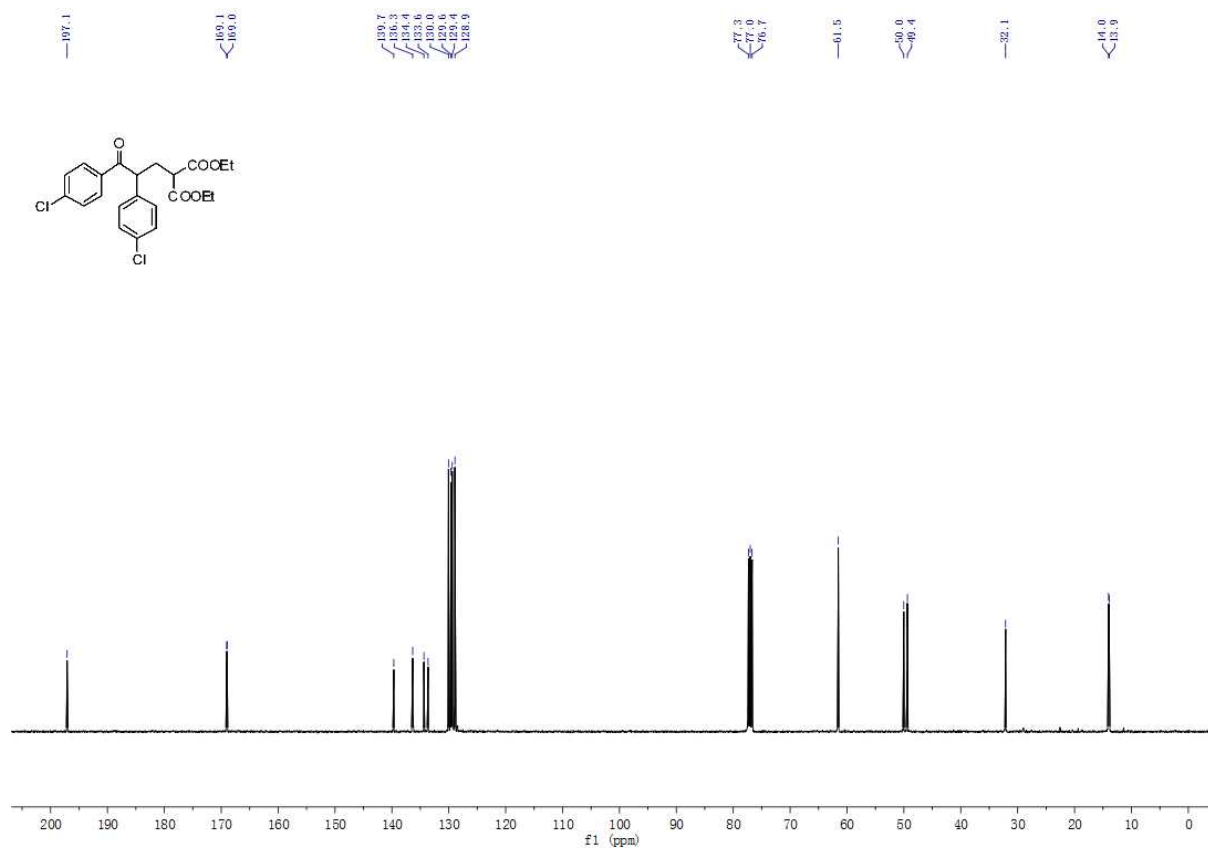
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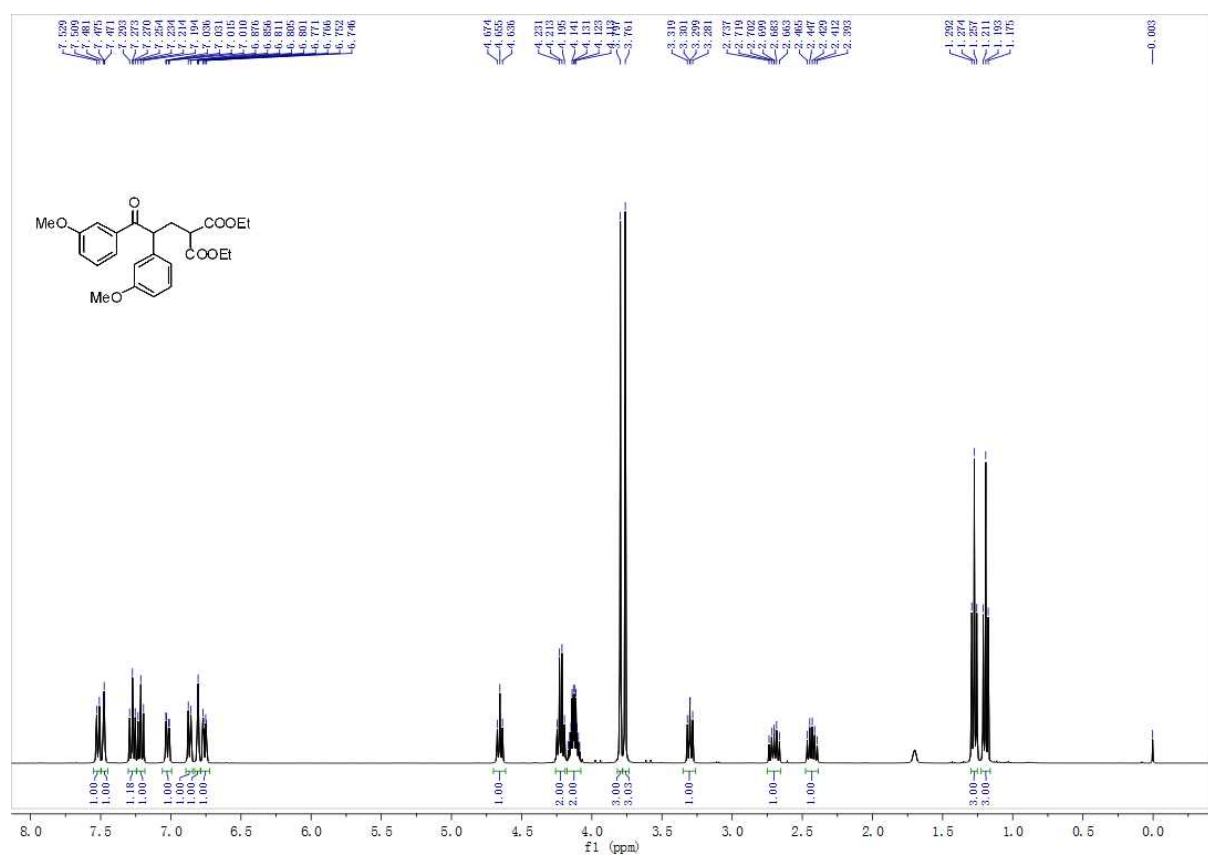
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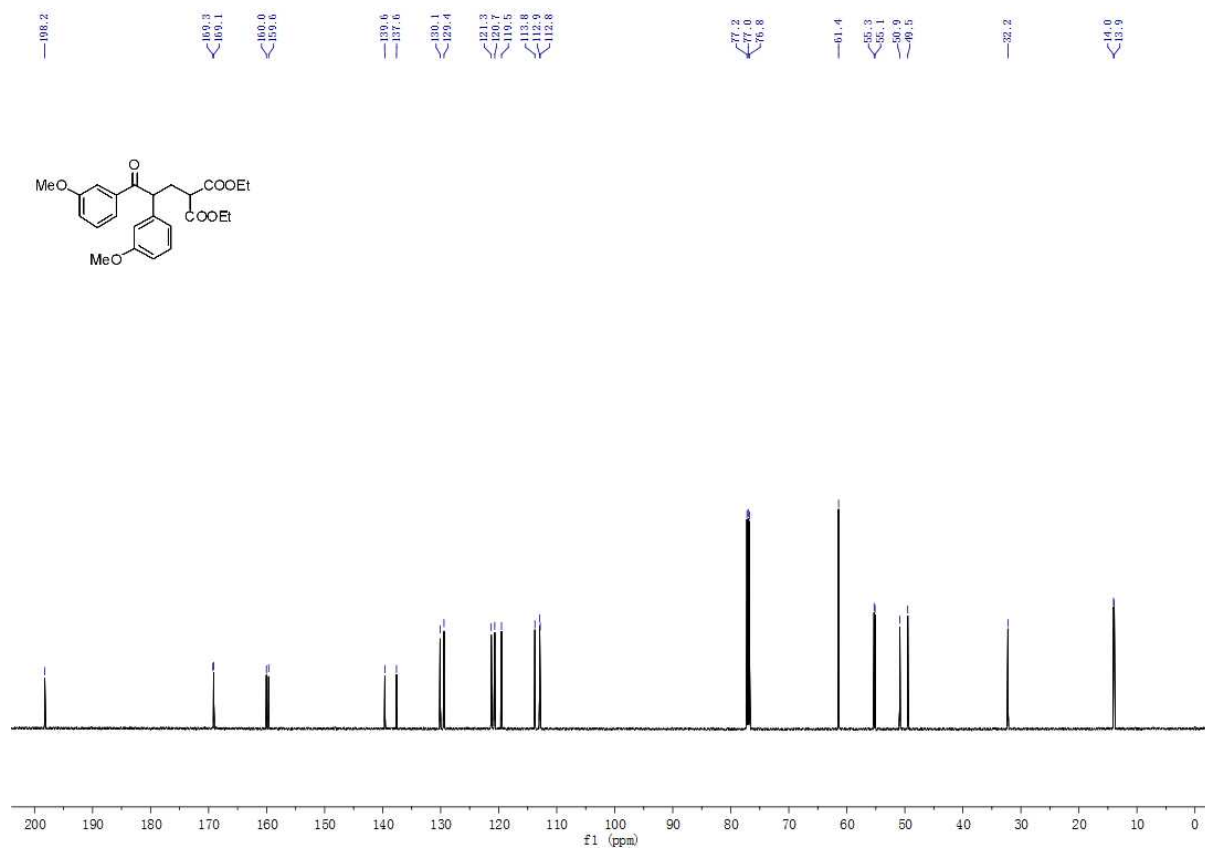
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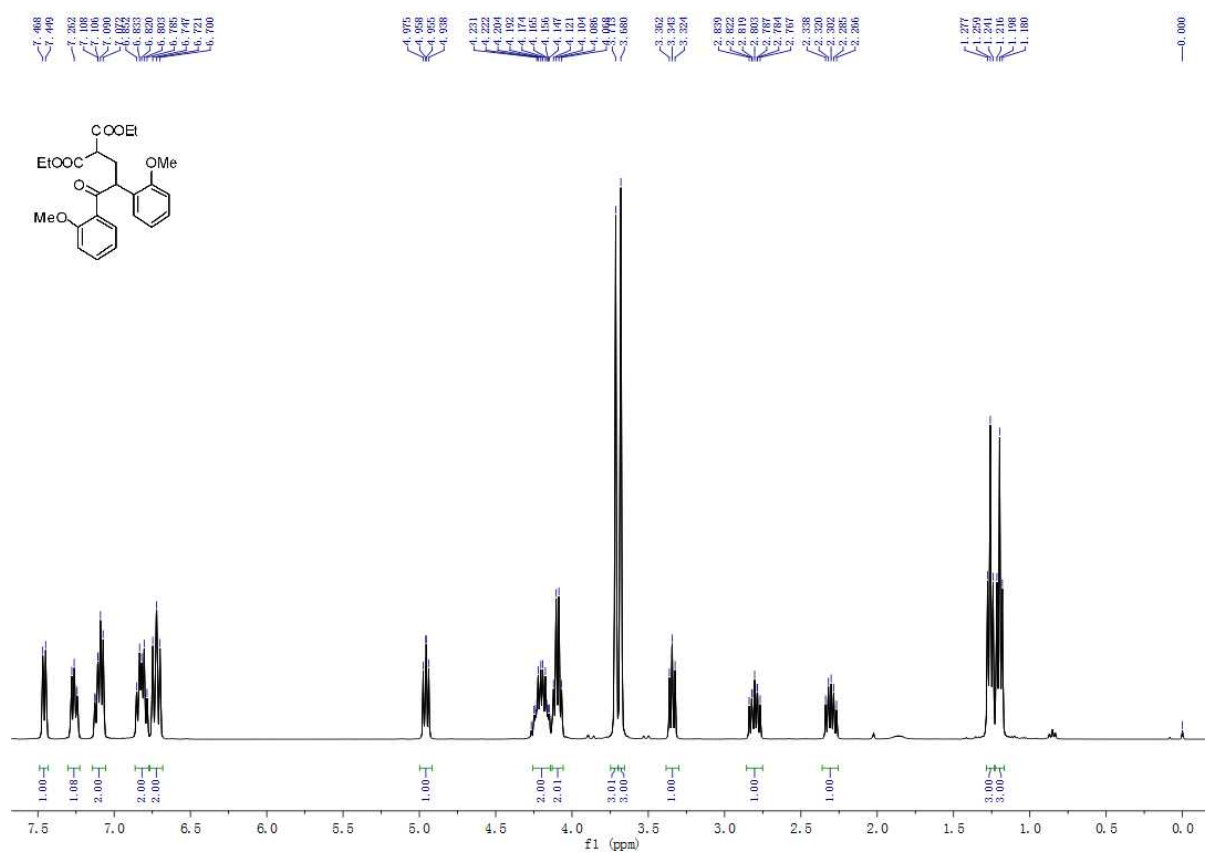
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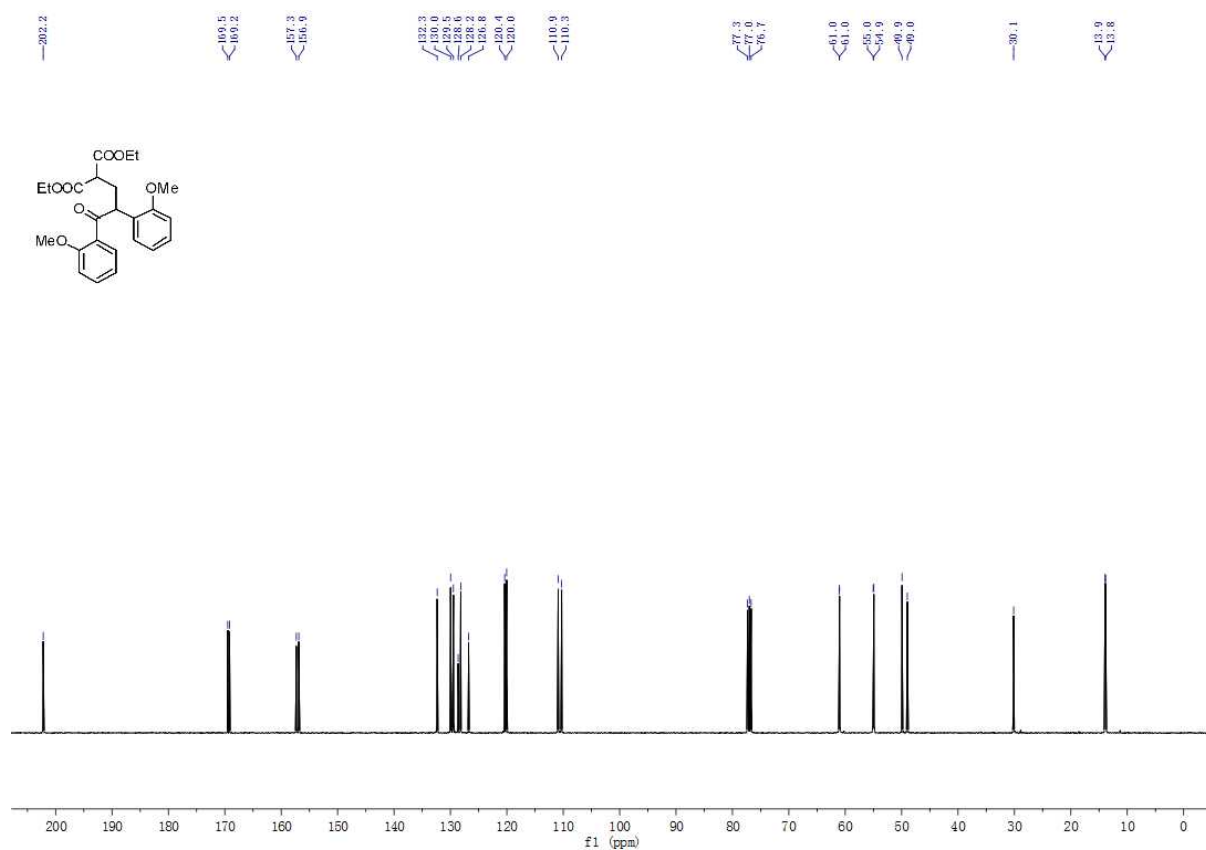
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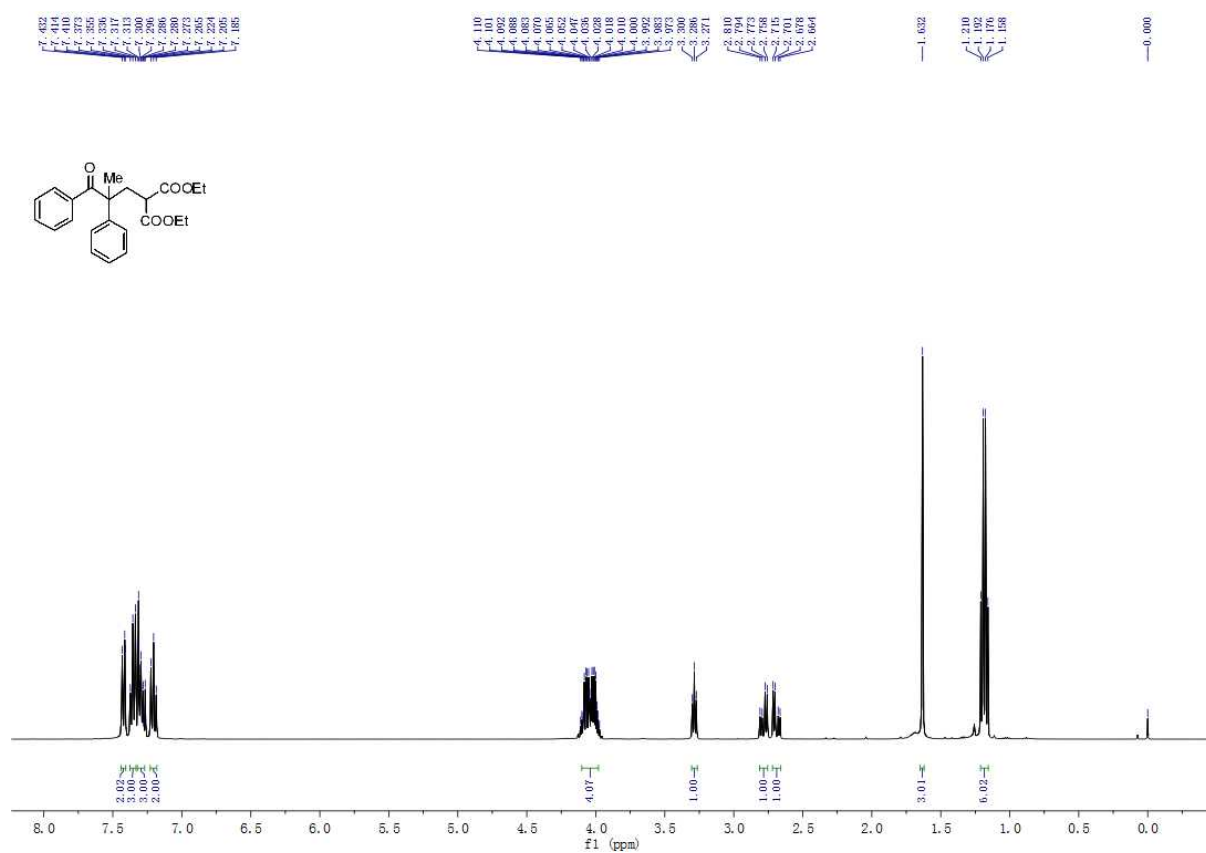
3h ¹H NMR



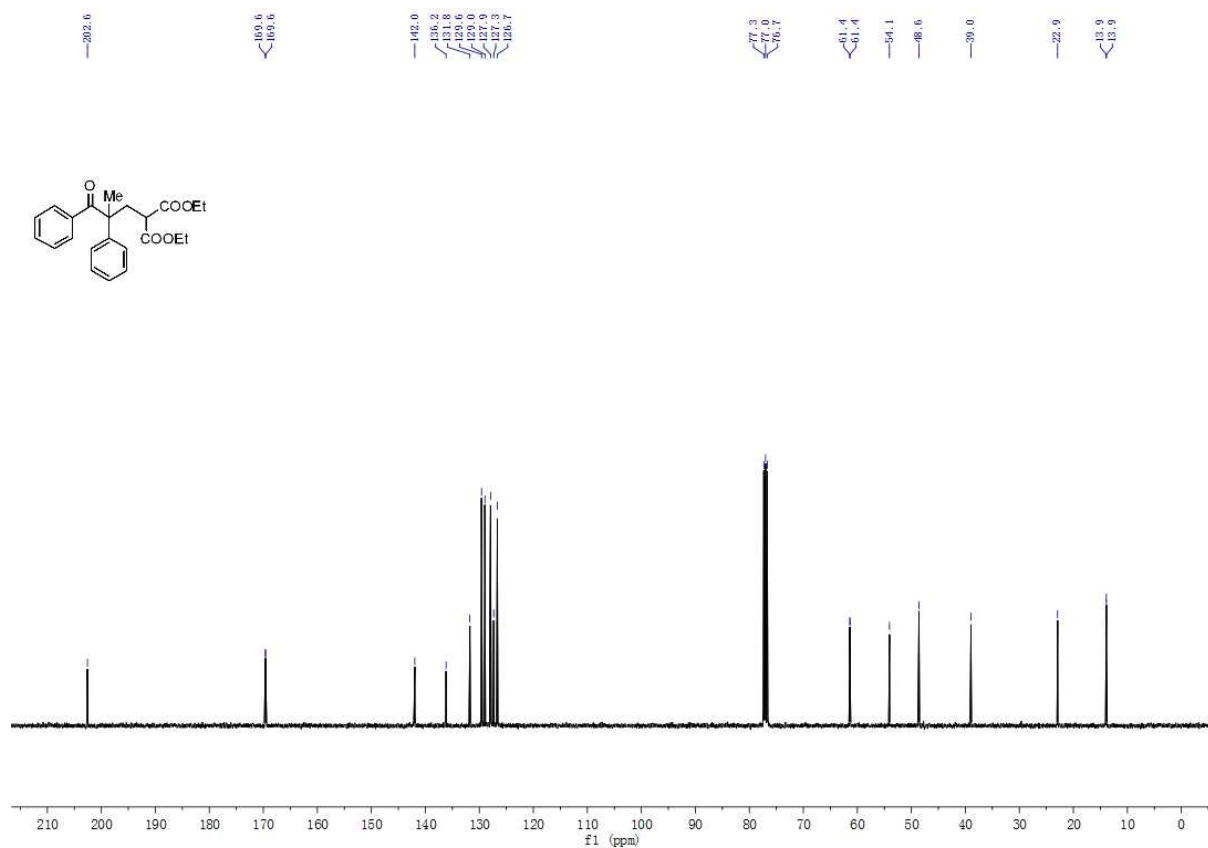
3h ¹³C NMR



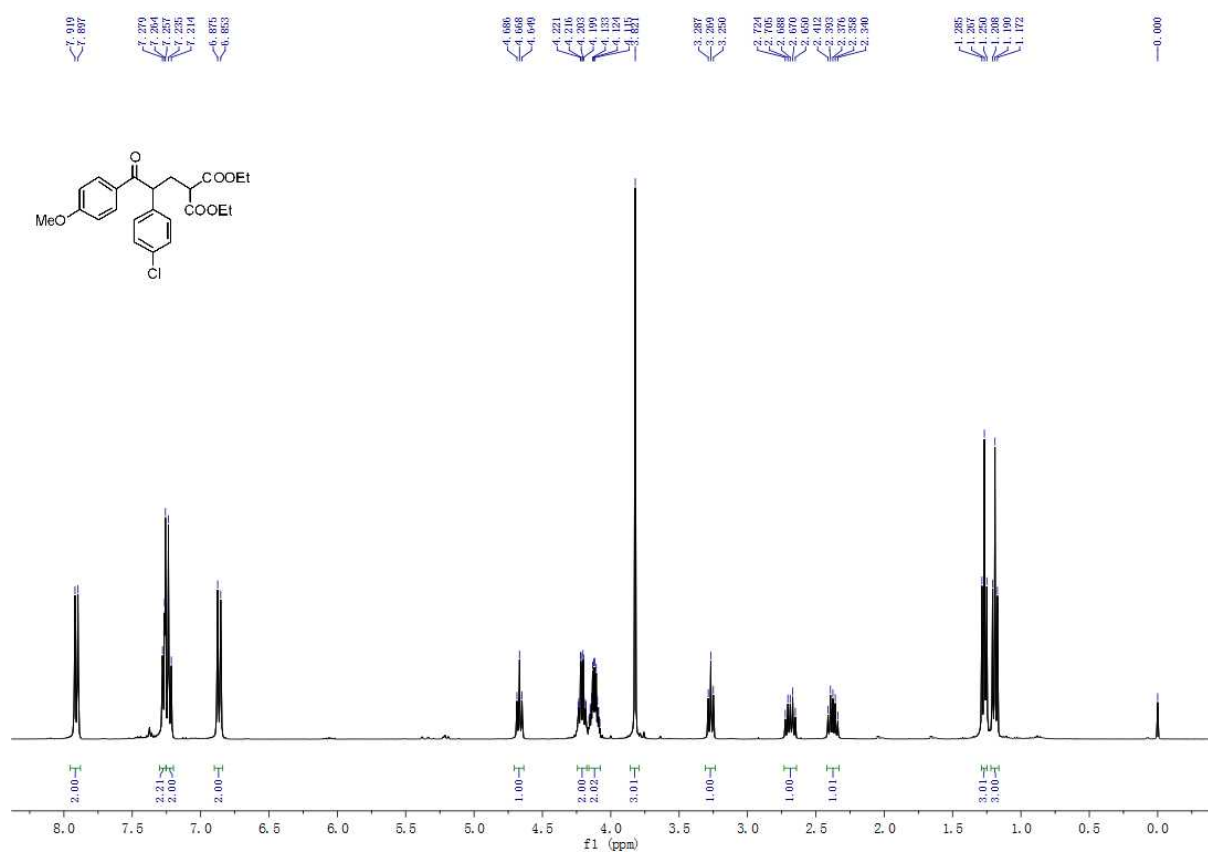
3i ¹H NMR



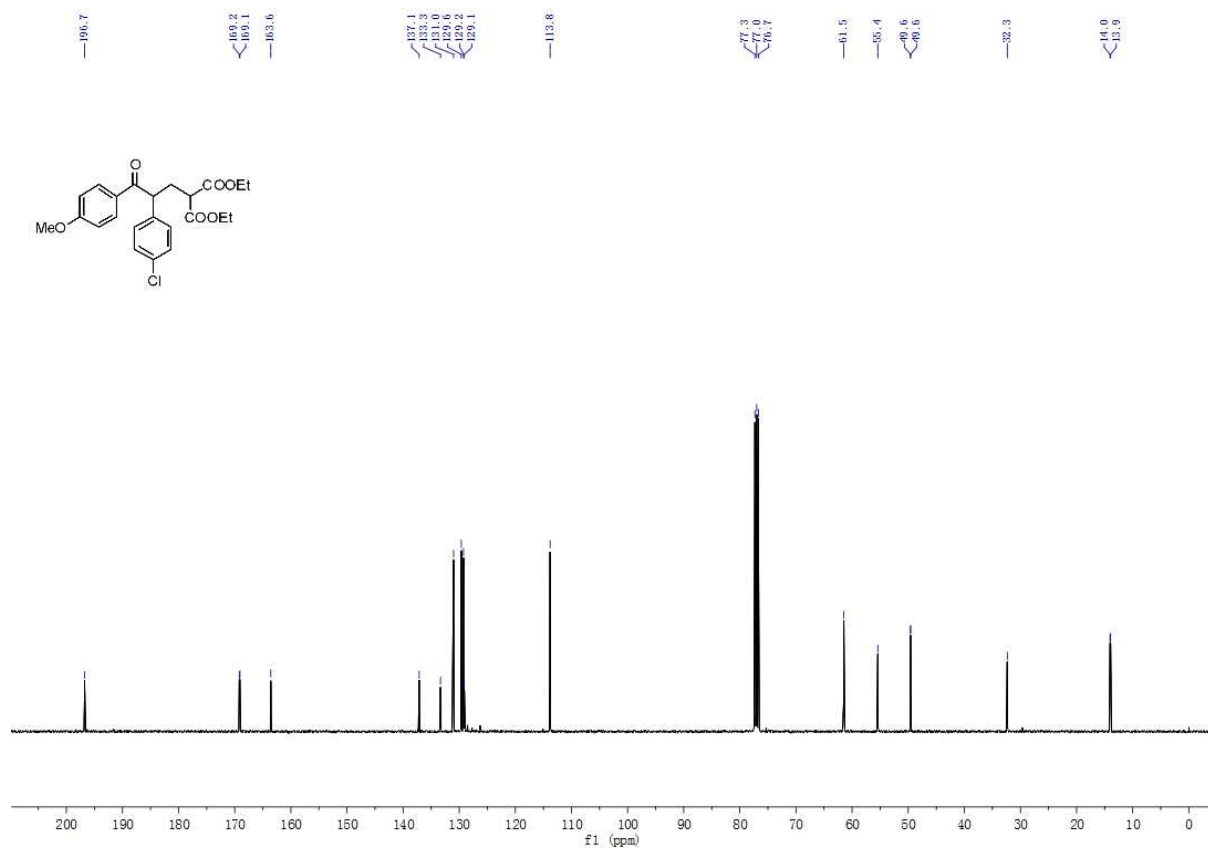
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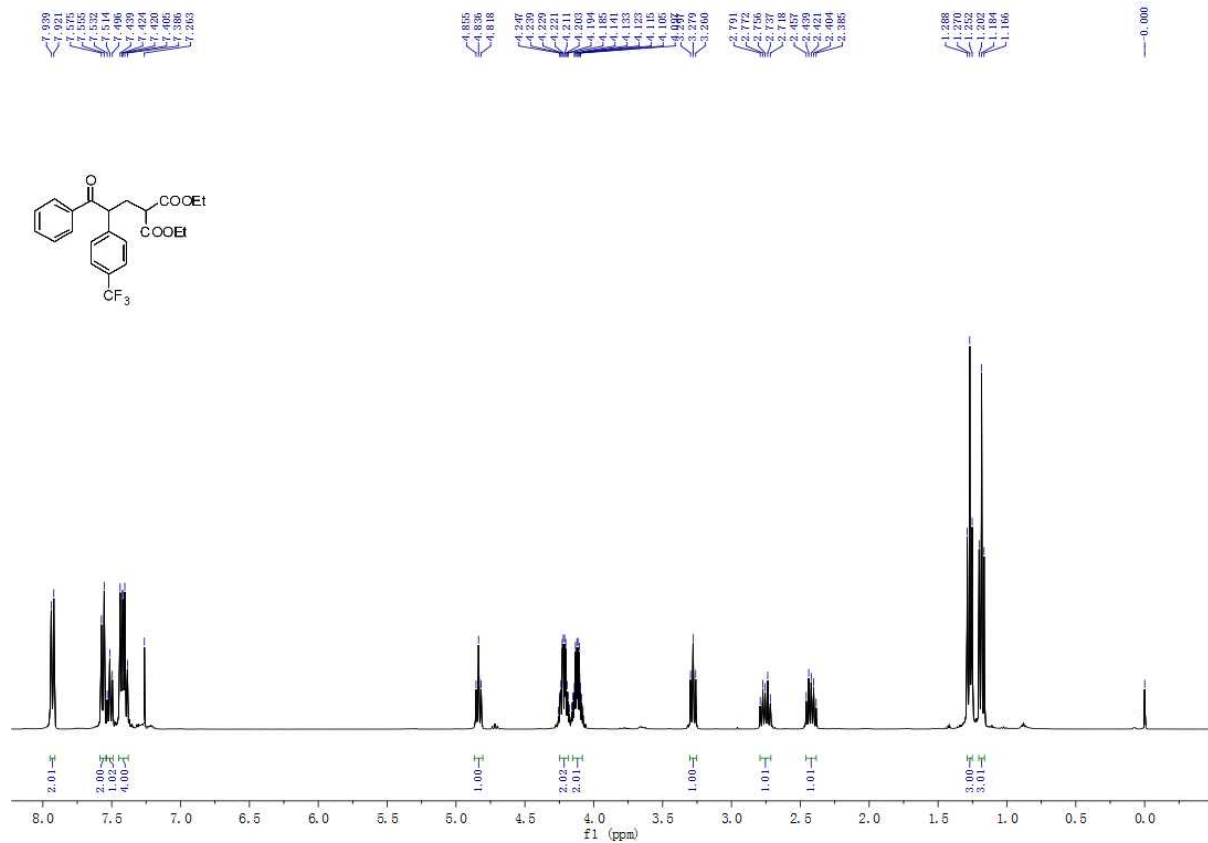
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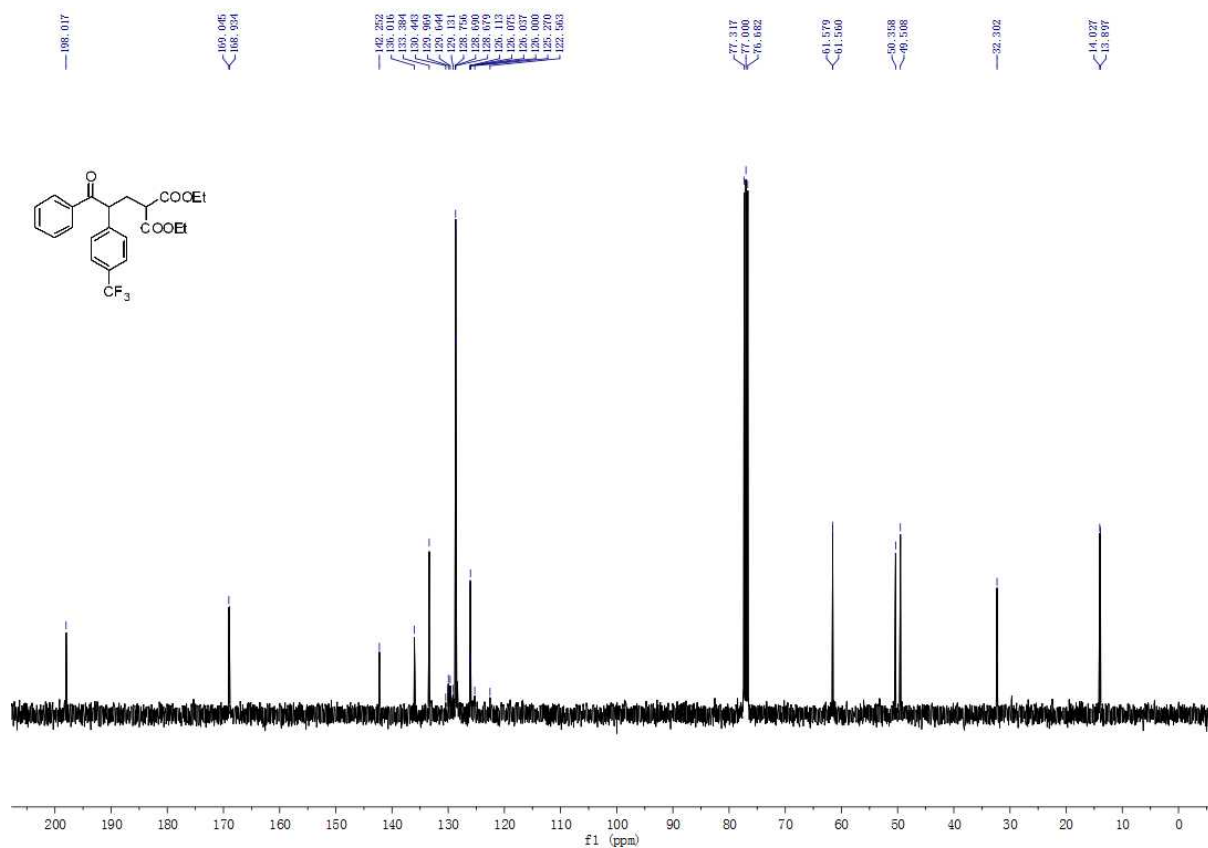
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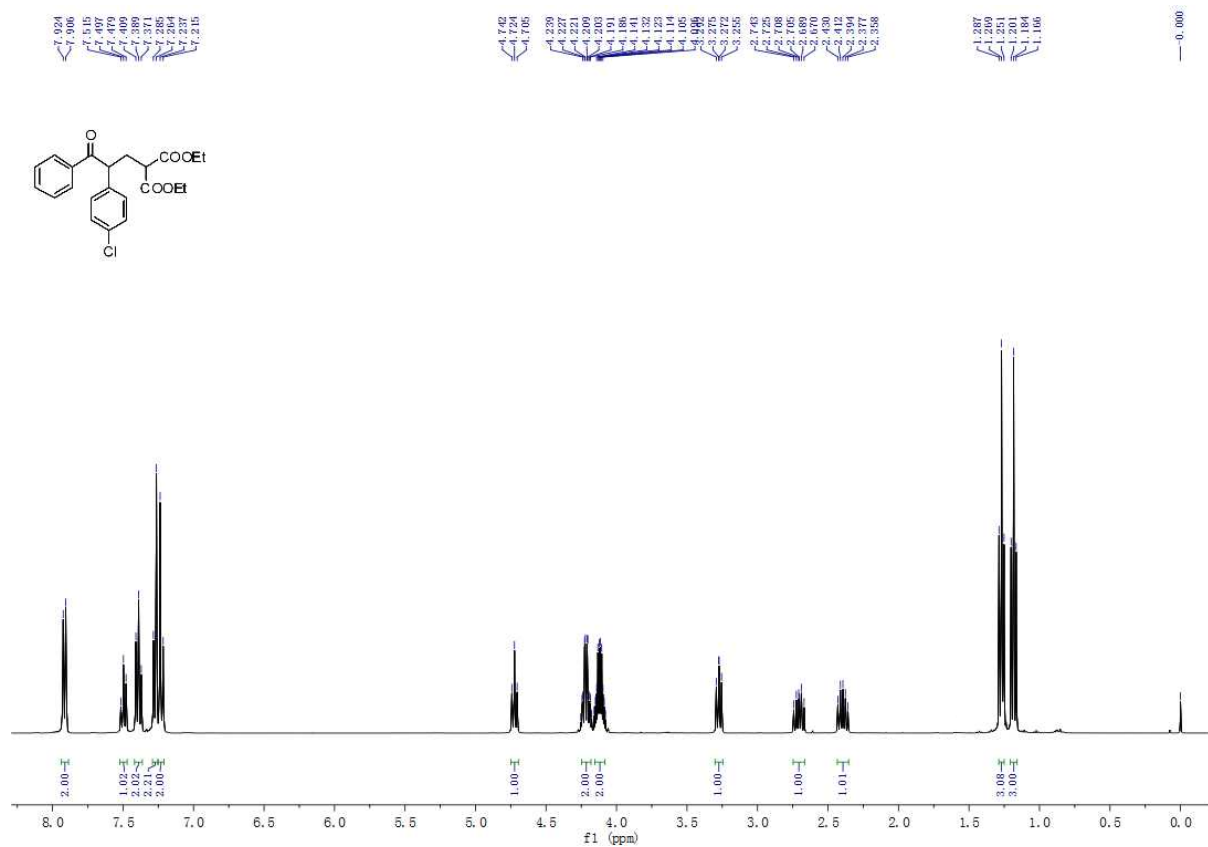
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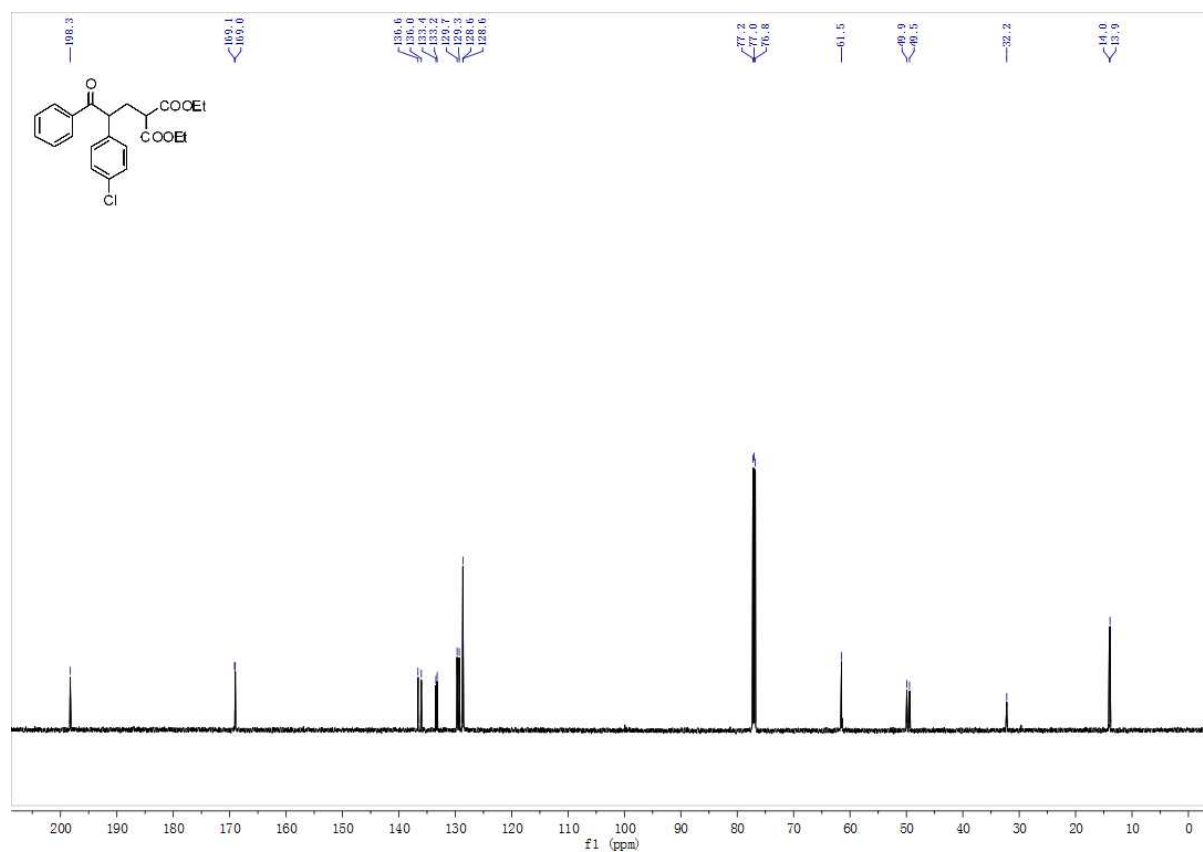
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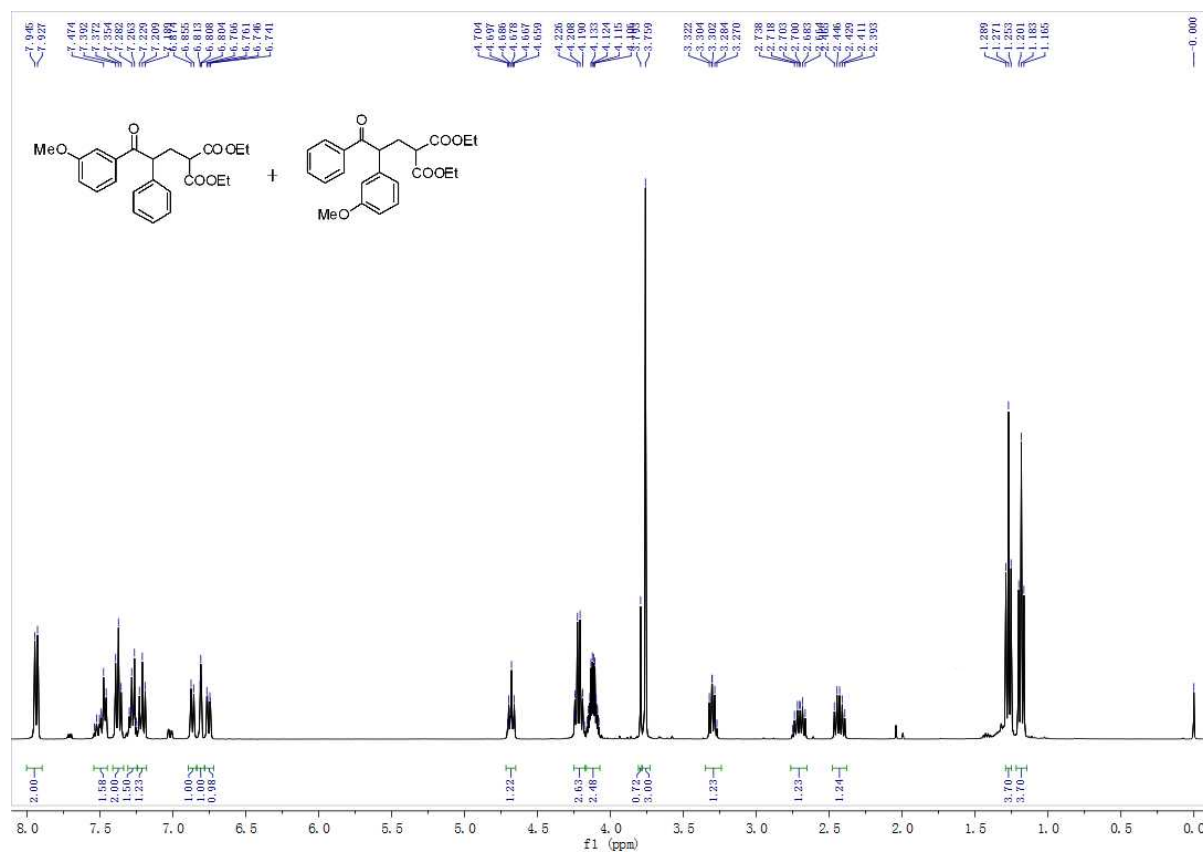
31 ¹H NMR



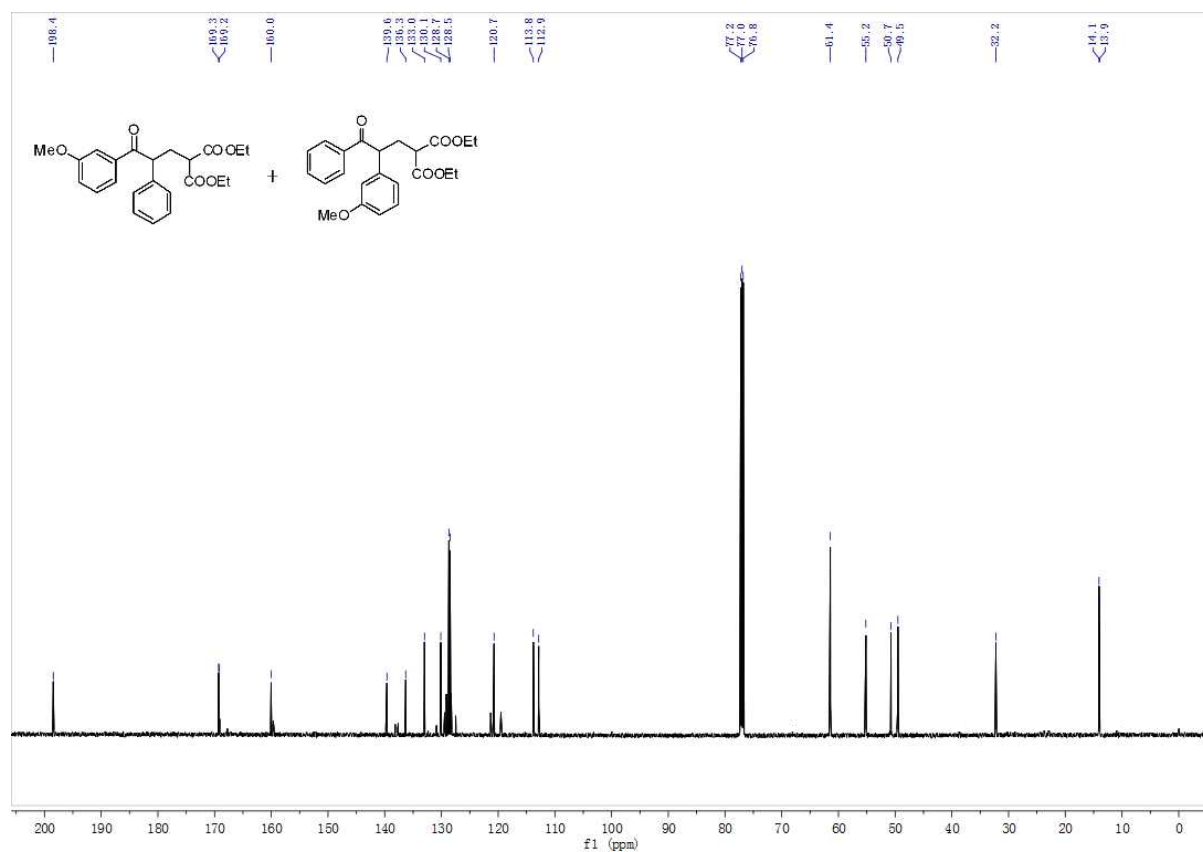
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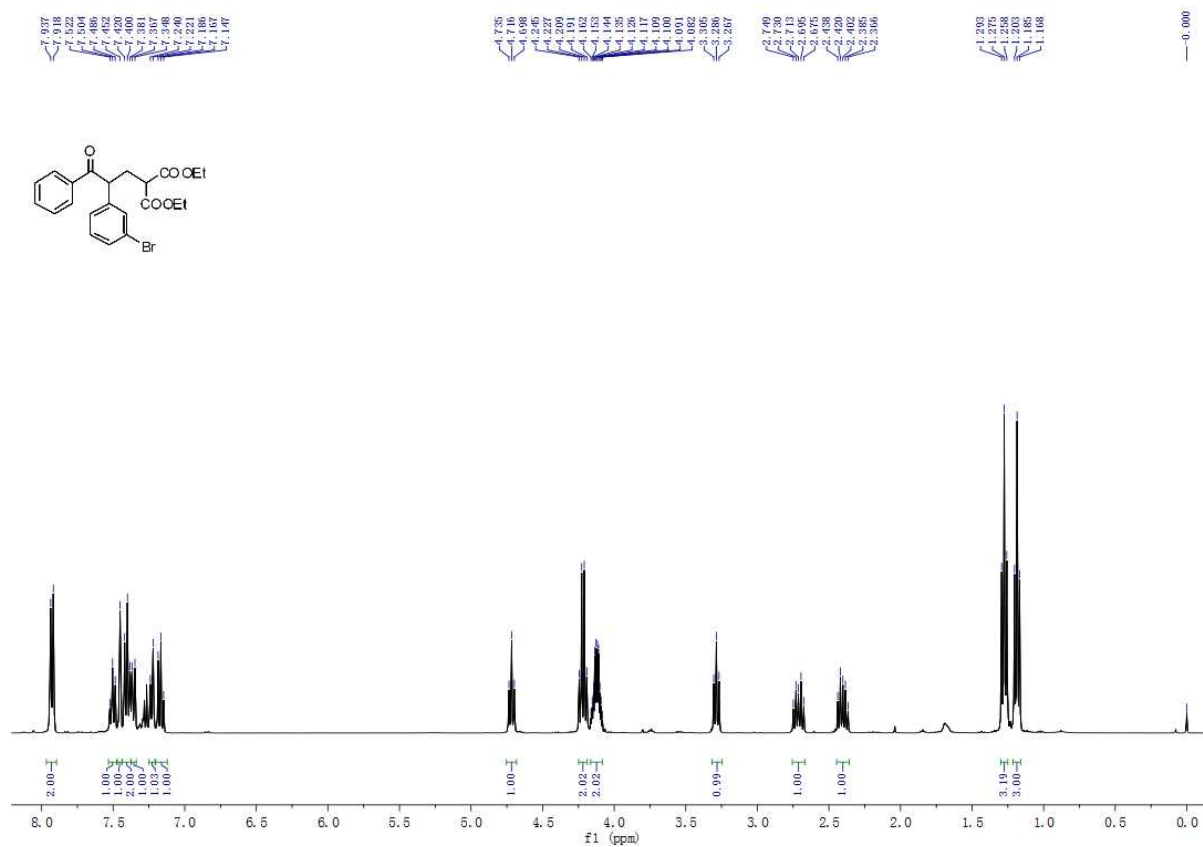
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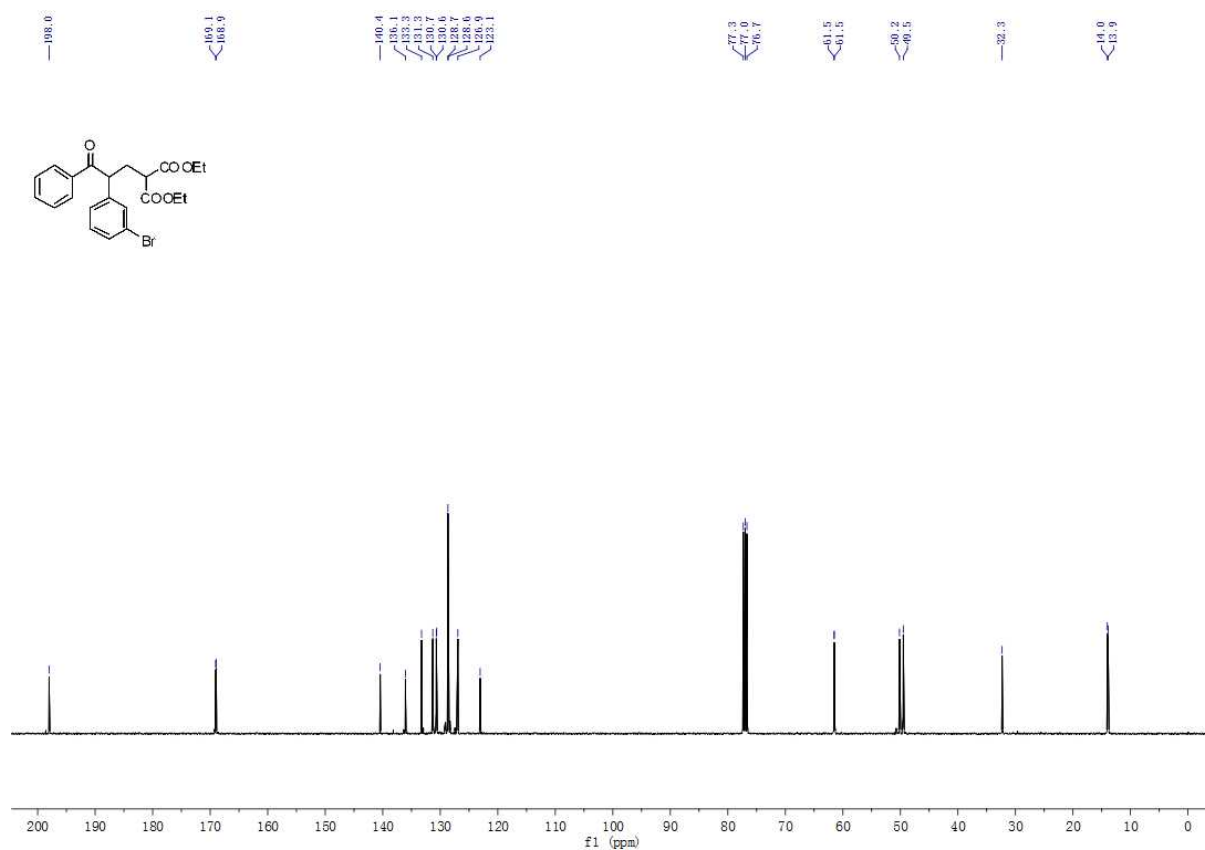
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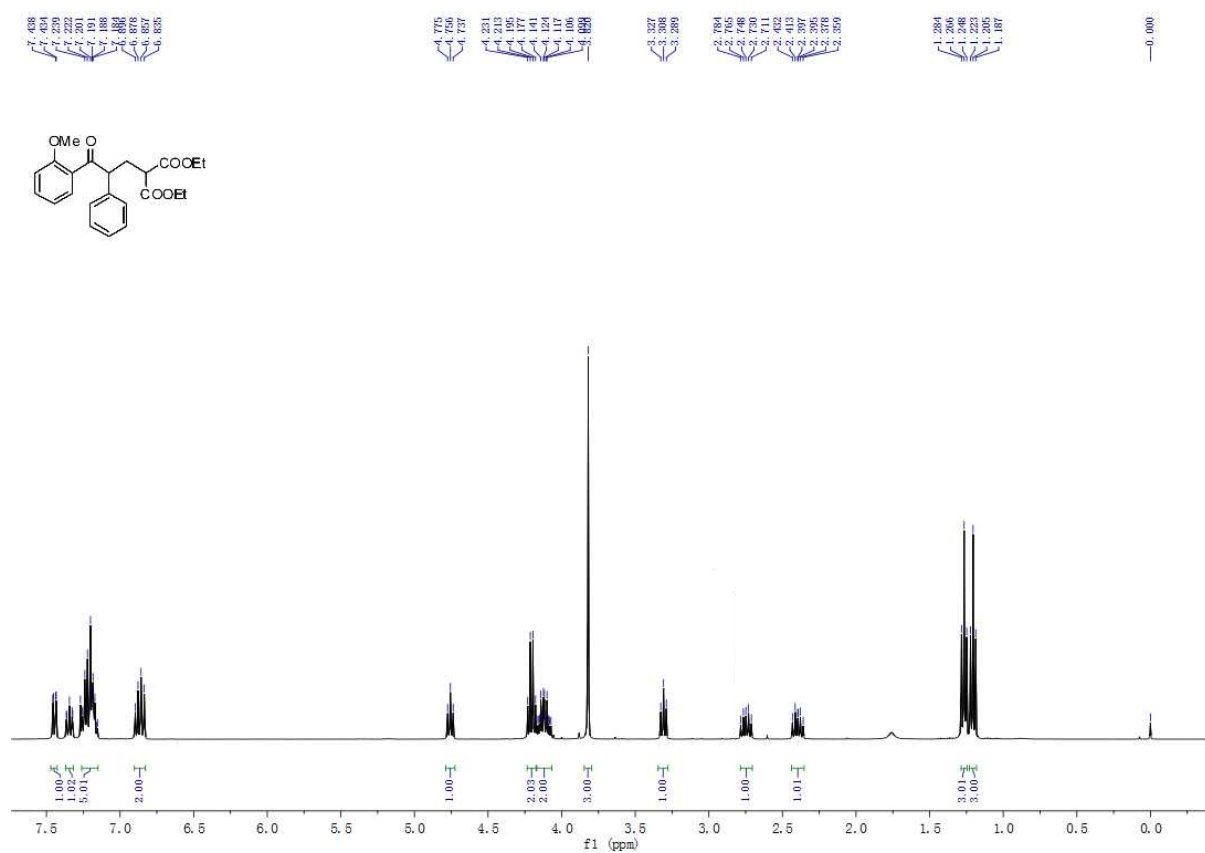
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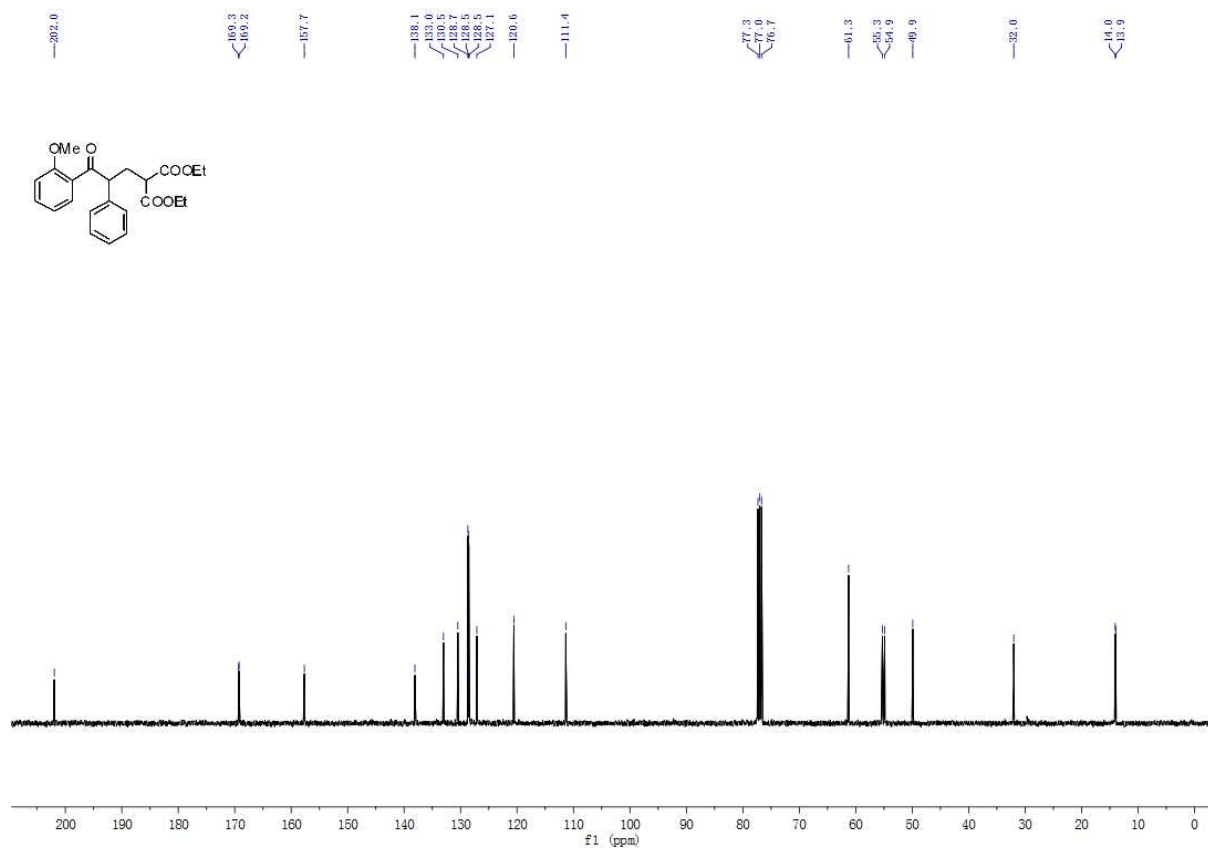
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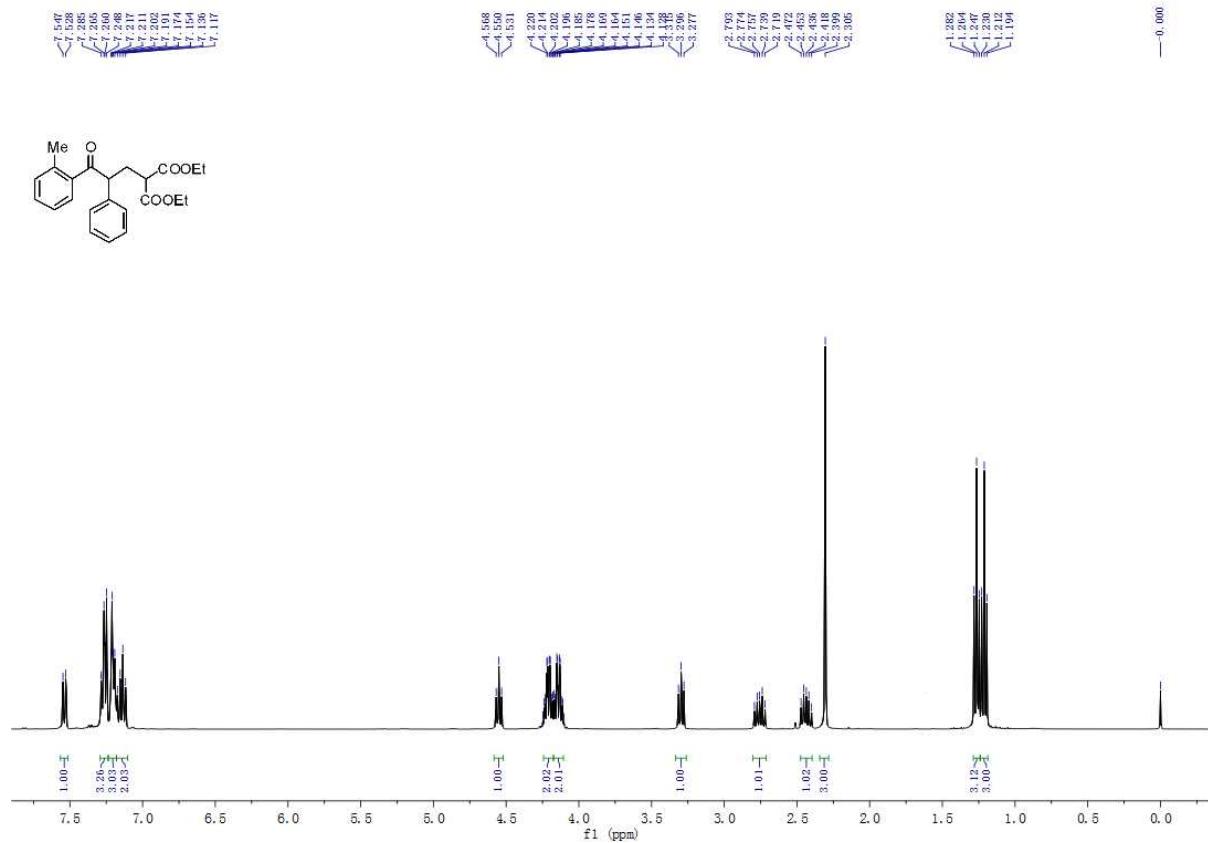
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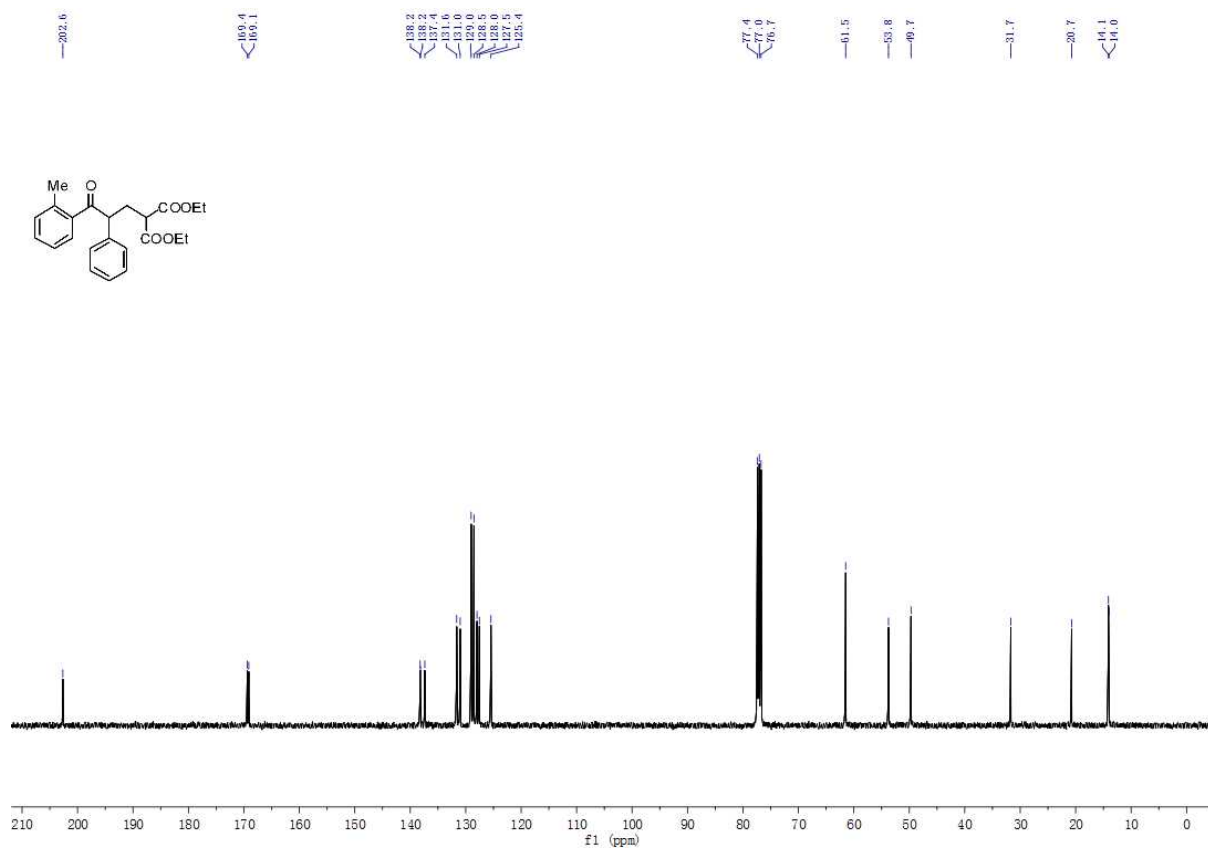
30 ¹³C NMR



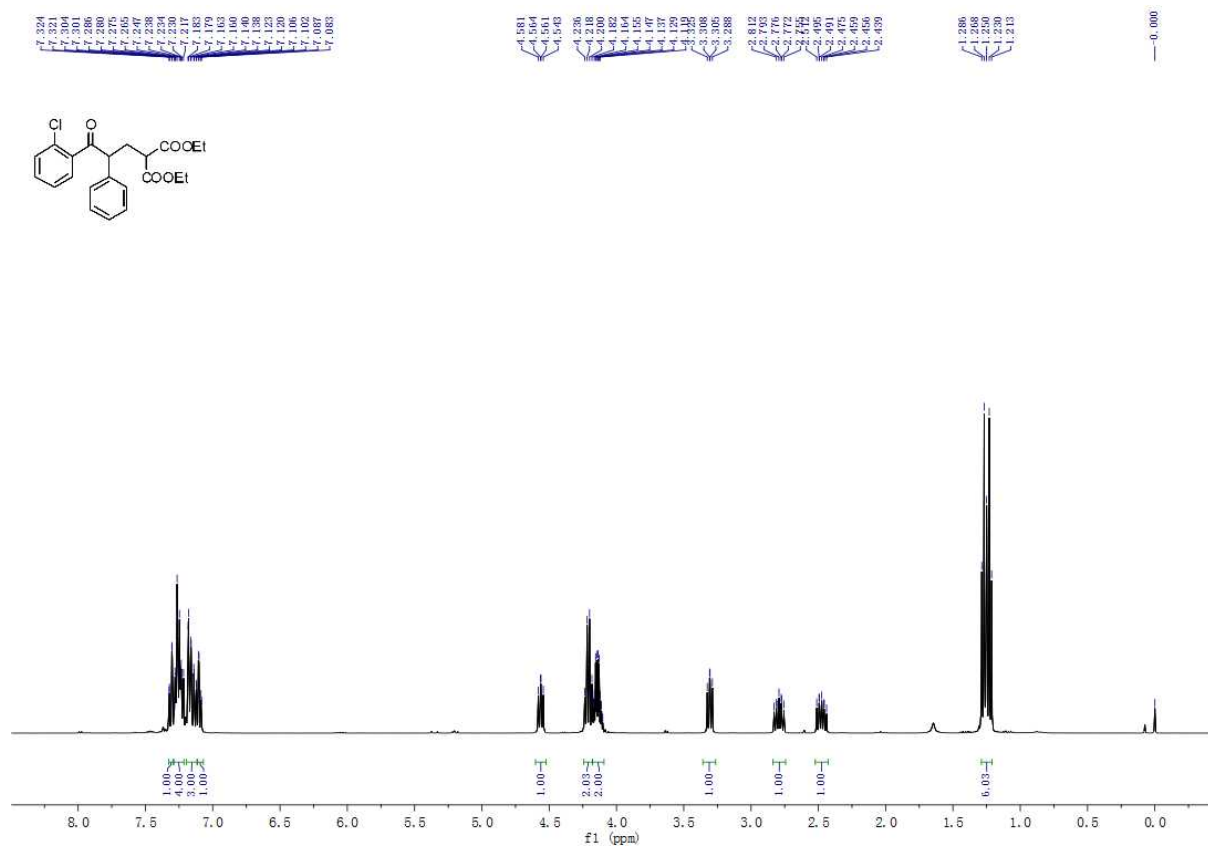
3p ¹H NMR



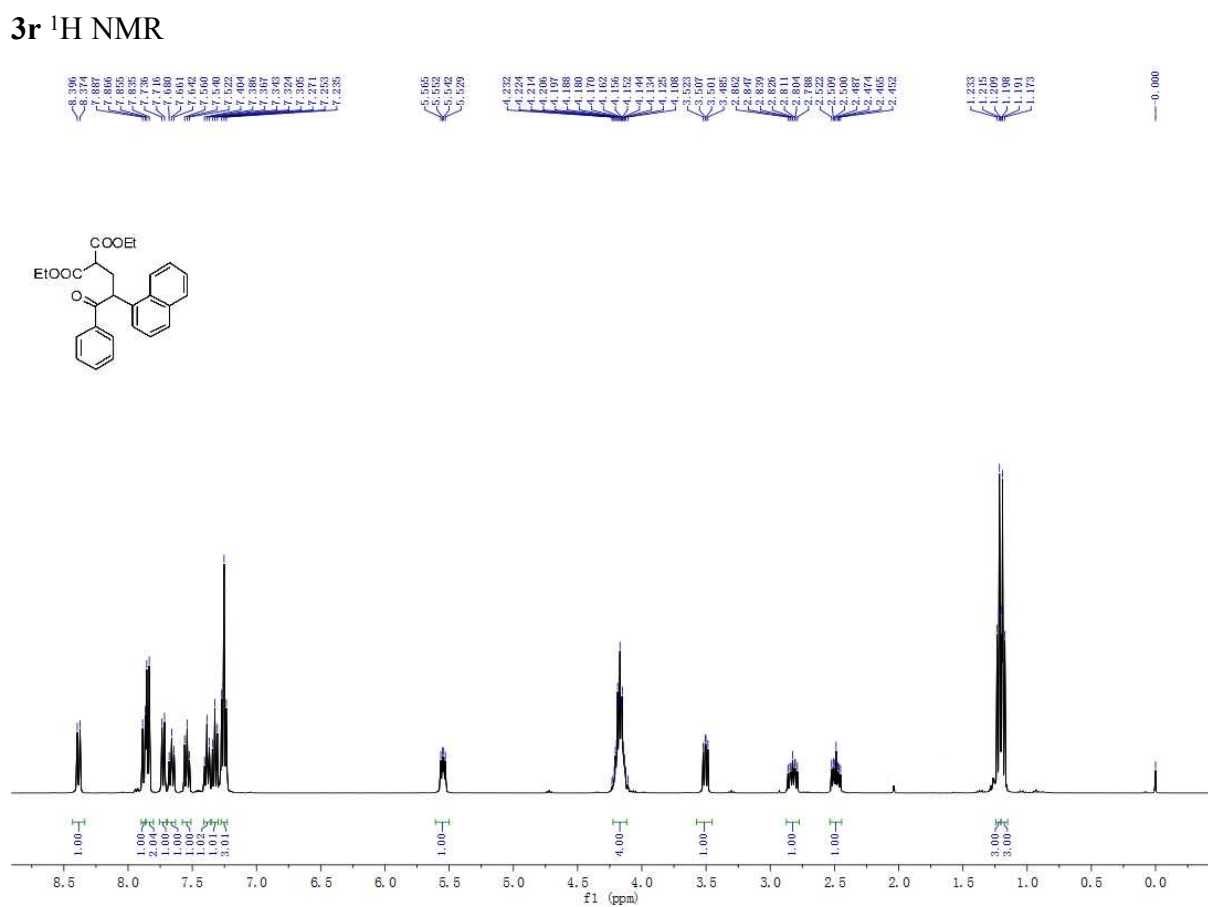
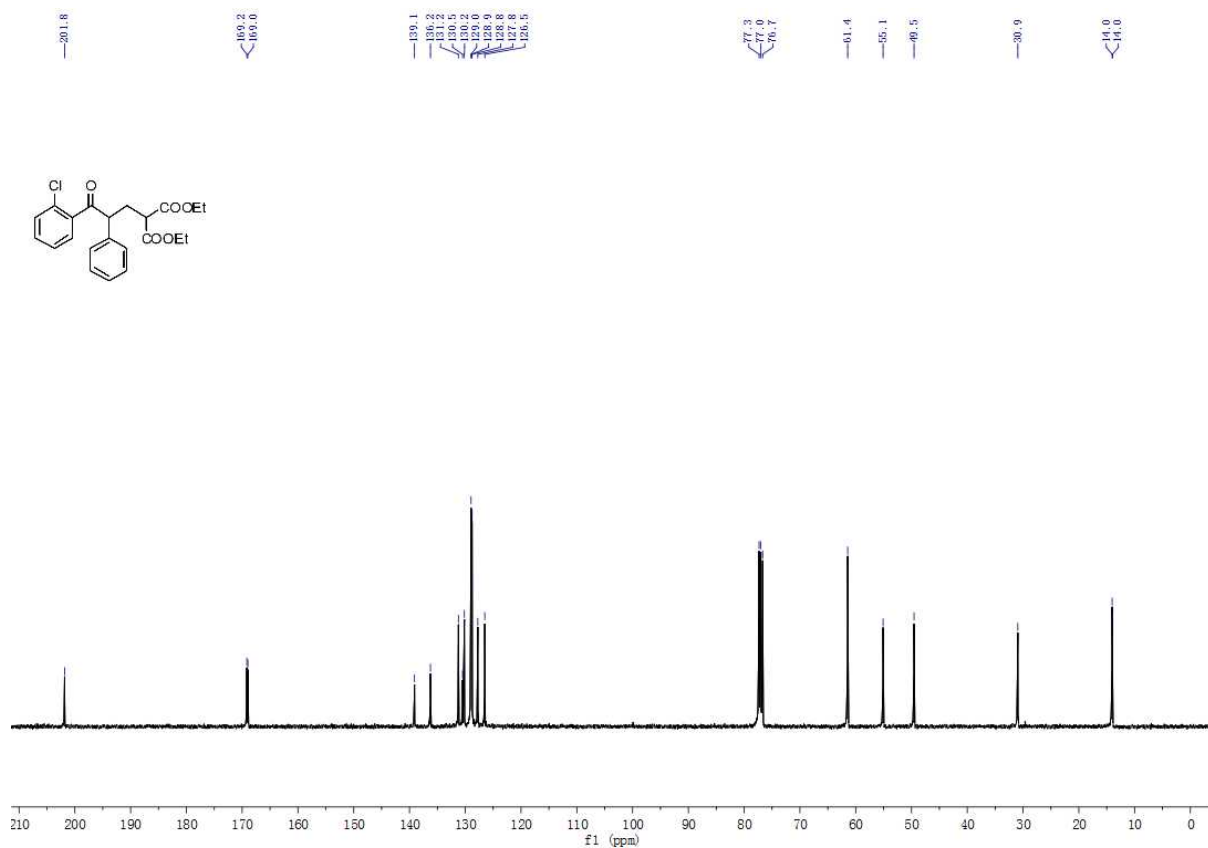
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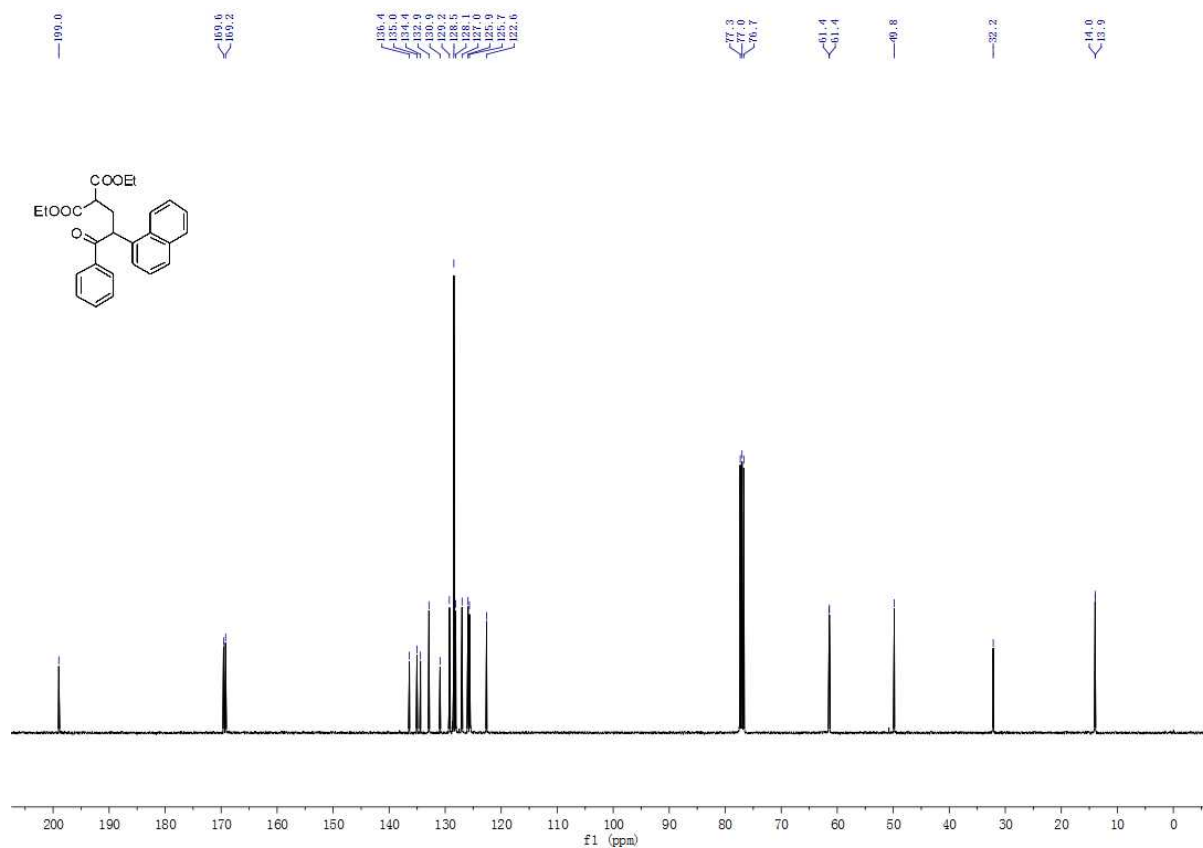
3q ¹H NMR



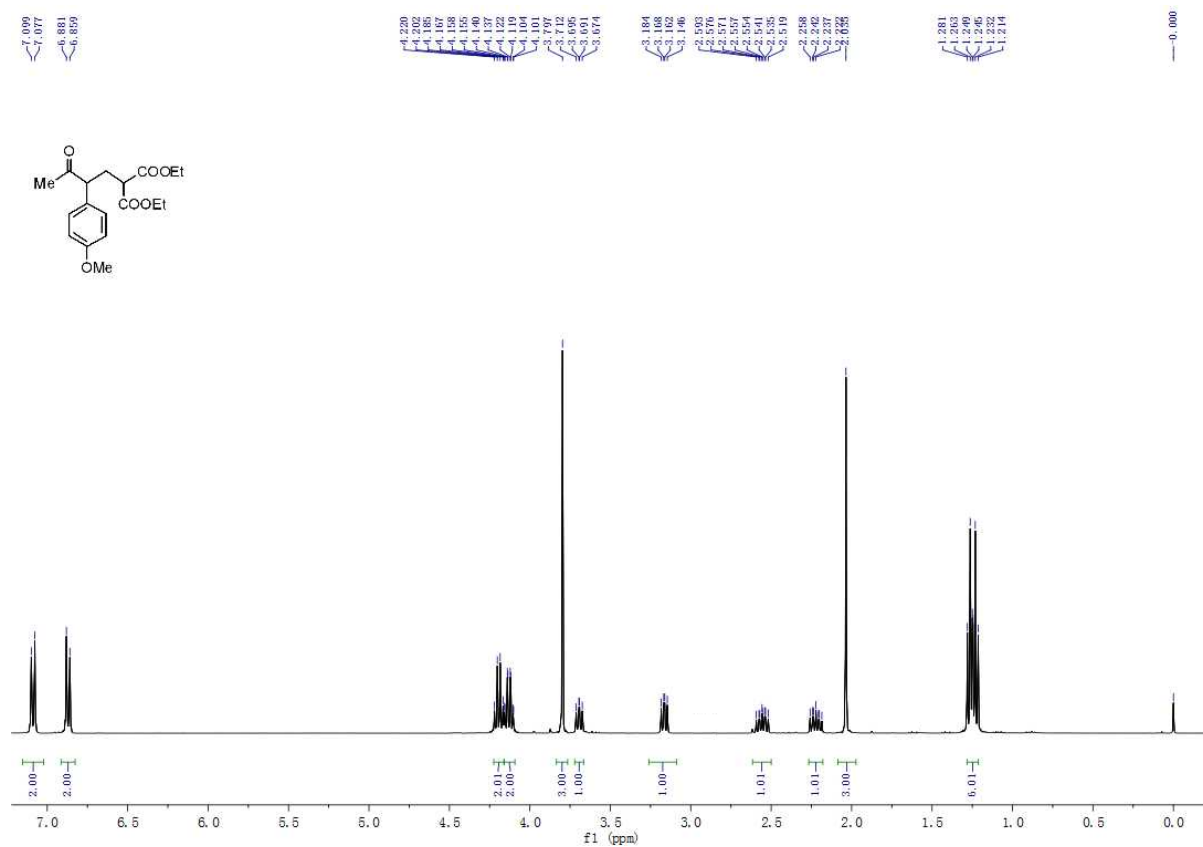
3q ¹³C NMR



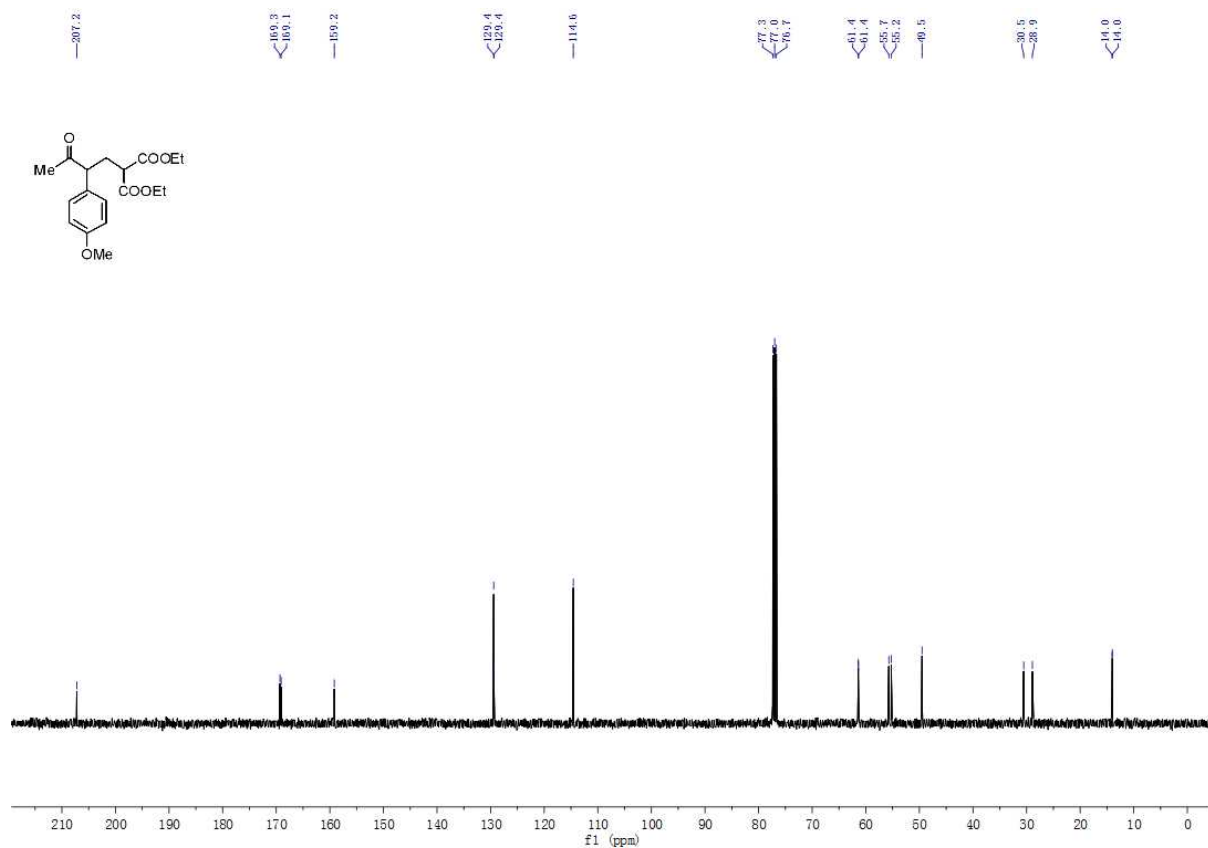
3r ¹³C NMR



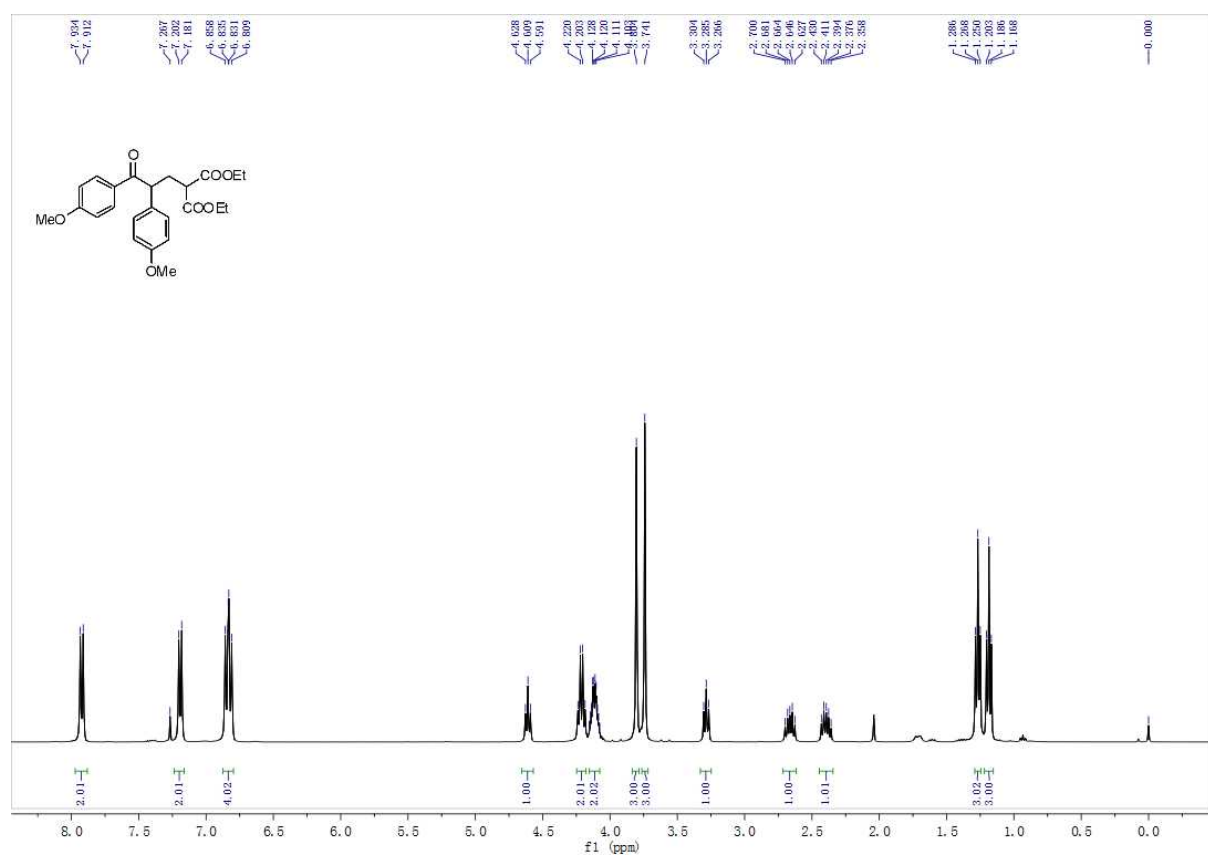
3s ¹H NMR



3s ¹³C NMR



3t ¹H NMR



3t ¹³C NMR

