Supporting information

Visible light-induced difunctionalization of electron-enriched styrenes: synthesis of tetrahydrofurans and tetrahydropyrans

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Contents

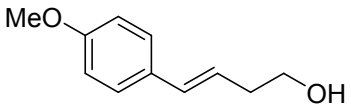
1. General information

All reagents were purchased from commercial sources unless otherwise noted. Solvent was freshly distilled prior to use unless otherwise noted. Reactions were monitored by thin-layer chromatography (TLC) and visualized by UV-light (254 nm) or by treatment with a solution of 10 g phosphomolybdic acid and 100 mL EtOH followed by heating. ^{1}H NMR (400 MHz) and ^{13}C NMR (100 MHz) spectra were obtained on Bruker AV-400 instrument. Chemical shifts for ^{1}H NMR spectra were reported in δ ppm referenced to an internal $SiMe_4$ standard. Chemical shifts for ^{13}C NMR spectra were reported in parts per million relative to the center line signal of the $CDCl_3$ triplet at 77.0 ppm. HR-ESI-MS spectra were recorded on a Bruker Esquire LC mass spectrometer using electrospray ionization.

2. Preparation and characterization of alkenols

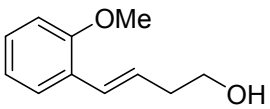
Alkenols **1a-l** were prepared according to reported method[1].

(*E*)-4-(4-methoxyphenyl)but-3-en-1-ol (1a)



^{1}HNMR ($CDCl_3$, 400 MHz): δ 7.27 (d, J = 8.7 Hz, 2H), 6.82 (d, J = 8.7 Hz, 2H), 6.40 (d, J = 15.9 Hz, 1H), 6.03 (dt, J = 15.7, 7.1 Hz, 1H), 3.77 (s, 3H), 3.69 (t, J = 6.4 Hz, 2H), 2.42 (td, J = 7.3, 1.0 Hz, 2H), 2.18 (brs, 1H). Data are consistent with literature values.[1]

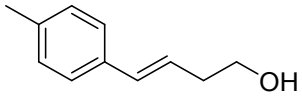
(*E*)-4-(2-methoxyphenyl)but-3-en-1-ol (1b)



^{1}HNMR ($CDCl_3$, 400 MHz): δ 7.43-7.40 (m,1H), 7.26-7.18 (m, 1H), 6.95-6.79 (m, 3H), 6.19 (dt, J = 15.9, 7.2 Hz, 1H), 3.84 (s, 3H), 3.76-3.69 (m, 2H), 2.53-2.47 (m, 2H), 1.68 (brs, 1H). Data are consistent with literature values. [1]

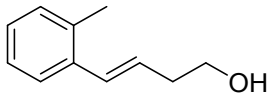
(*E*)-4-*p*-tolylbut-3-en-1-ol (1c)

[1] Zeng, X. H.; Miao, C. X.; Wang, S. F.; Xia, C. G.; Sun, W. *Chem. Commun.* 2013, 49, 2418.



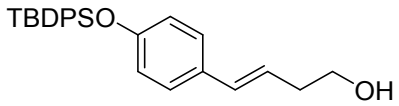
^{1}HNMR ($CDCl_3$, 400 MHz): δ 7.16 (d, J = 8.0 Hz, 2H), 7.01 (d, J = 8.0 Hz, 2H), 6.36 (d, J = 15.9 Hz, 1H), 6.05 (dt, J = 15.8, 7.1 Hz, 1H), 3.64-3.60 (m, 2H), 2.36 (td, J = 7.3, 0.9 Hz, 2H), 2.23 (s, 3H), 2.00 (brs, 1H). Data are consistent with literature values. [1]

(*E*)-4-*o*-tolylbut-3-en-1-ol (1d)



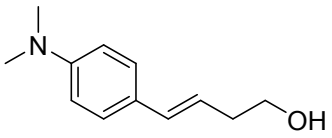
^{1}HNMR ($CDCl_3$, 400 MHz): δ 7.16-7.12 (m, 4H), 6.68 (d, J = 15.7 Hz, 1H), 6.06 (dt, J = 15.5, 7.2 Hz, 1H), 3.73 (t, J = 6.4 Hz, 2H), 2.49 (td, J = 7.6, 1.3 Hz, 2H), 2.33 (s, 3H), 1.81 (brs, 1H). Data are consistent with literature values. [1]

(*E*)-4-(4-((tert-butyldiphenylsilyl)oxy)phenyl)but-3-en-1-ol (1e)



^{1}H NMR (400 MHz, $CDCl_3$): δ 7.71 (d, J = 6.6 Hz, 4H), 7.37 (d, J = 7.4 Hz, 6H), 7.10 (d, J = 8.6 Hz, 2H), 6.70 (d, J = 8.6 Hz, 2H), 6.37 (d, J = 15.9 Hz, 1H), 6.00 – 5.96 (m, 1H), 3.70 (t, J = 5.1 Hz, 2H), 2.42 (q, J = 6.2 Hz, 2H), 1.10 (s, 9H). ^{13}C NMR (100 MHz, $CDCl_3$): δ 155.0, 135.5, 132.8, 129.9, 127.8, 126.9, 119.7, 119.4, 62.0, 36.3, 26.5, 19.4. HRMS-ESI (m/z): $[M+H]^+$ calculated for $C_{26}H_{30}O_2Si$ 403.2093, found 403.2100.

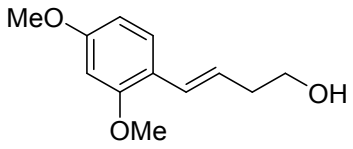
(*E*)-4-(4-(dimethylamino)phenyl)but-3-en-1-ol (1f)



^{1}H NMR (400 MHz, $CDCl_3$): δ 7.24 (d, J = 8.8 Hz, 2H), 6.67 (d, J = 8.8 Hz, 2H), 6.40 (d, J = 15.8 Hz, 1H), 5.96 (dt, J = 15.7, 7.2 Hz, 1H), 3.70 (t, J = 6.3 Hz, 2H), 2.93 (s, 6H), 2.44 (td, J = 7.3, 1.1 Hz, 2H), 1.71 (brs, 1H). ^{13}C NMR (100 MHz, $CDCl_3$): δ

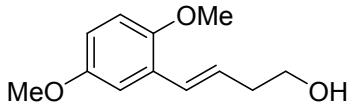
149.9, 132.7, 126.9, 125.9, 121.7, 112.5, 62.1, 40.5, 36.4. HRMS-ESI (*m*/*z*): $[M+H]^+$ calculated for $C_{12}H_{17}NO$ 192.1383, found 192.1387.

(*E*)-4-(2,4-dimethoxyphenyl)but-3-en-1-ol (1g)



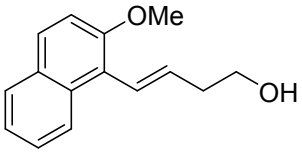
^{1}HNMR ($CDCl_3$, 400 MHz): δ 7.35 (d, J = 8.3 Hz, 1H), 6.74 (d, J = 16.0 Hz, 1H), 6.50-6.45 (m, 2H), 6.10 (dt, J = 15.9, 7.2 Hz, 1H), 3.84 (s, 3H), 3.83 (s, 3H), 3.75 (t, J = 6.3 Hz, 2H), 2.53-2.47 (m, 2H), 1.74 (brs, 1H); ^{13}C NMR ($CDCl_3$, 100 MHz): δ 160.1, 157.4, 127.2, 127.2, 124.7, 119.4, 104.7, 98.38, 62.1, 55.4, 55.3, 36.8. HRMS-ESI (*m*/*z*): $[M+H]^+$ calculated for $C_{12}H_{16}O_3$ 209.1172, found 209.1171.

(*E*)-4-(2,5-dimethoxyphenyl)but-3-en-1-ol (1h)



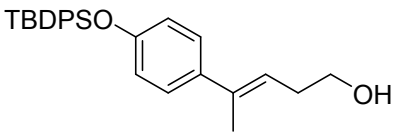
^{1}HNMR ($CDCl_3$, 400 MHz): δ 6.85 (d, J = 2.8 Hz, 1H), 6.78 (dd, J = 8.5, 5.8 Hz, 1H), 6.63 (d, J = 11.6 Hz, 1H), 5.75 (dt, J = 11.6, 7.5 Hz, 1H), 3.78 (s, 3H), 3.77 (s, 3H), 3.71 (t, J = 6.4 Hz, 2H), 2.51 (dd, J = 7.5, 1.5 Hz, 2H), 1.72 (brs, 1H). ^{13}C NMR ($CDCl_3$, 100 MHz): δ 153.7, 150.9, 127.4, 127.3, 113.3, 112.2, 112.0, 62.1, 56.2, 55.8, 36.8. HRMS-ESI (*m*/*z*): $[M+H]^+$ calculated for $C_{12}H_{16}O_3$ 209.1172, found 209.1176.

(*E*)-4-(2-methoxynaphthalen-1-yl)but-3-en-1-ol (1i)



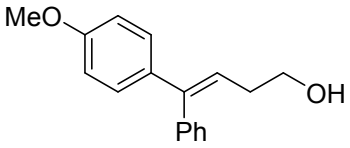
^{1}HNMR ($CDCl_3$, 400 MHz): δ 8.02 (d, J = 8.6 Hz, 1H), 7.68 (t, J = 8.4 Hz, 1H), 7.63 (d, J = 9.0 Hz, 1H), 7.34 (t, J = 7.2 Hz, 1H), 7.24 (dd, J = 12.8, 5.9 Hz, 1H), 7.16 (t, J = 8.6 Hz,1H), 6.73 (d, J = 16.1 Hz, 1H), 6.02 (dt, J = 15.9, 7.0 Hz, 1H), 3.82 (s, 3H), 3.70 (t, J = 6.3 Hz, 2H), 2.52 (q, J = 6.2 Hz, 2H). ^{13}C NMR ($CDCl_3$, 100 MHz): δ 153.0, 131.9, 130.6, 128.2, 127.4, 127.2, 125.3, 124.8, 123.3, 122.4, 119.8, 112.1, 60.8, 55.4, 36.3. HRMS-ESI (*m*/*z*): $[M+H]^+$ calculated for $C_{15}H_{16}O_2$ 229.1229, found 229.1230.

(*E*)-4-(4-((tert-butyldiphenylsilyl)oxy)phenyl)pent-3-en-1-ol (1j)



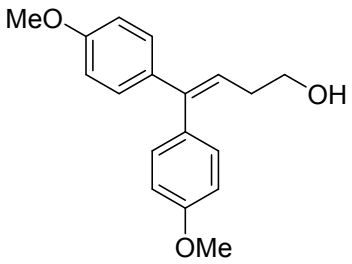
^{1}HNMR ($CDCl_3$, 400 MHz): δ 7.66 (dd, J = 7.9, 1.4 Hz, 4H), 7.34 (dd, J = 5.0, 3.6 Hz, 2H), 7.29 (t, J = 7.1 Hz, 4H), 6.86 (d, J = 8.6 Hz, 2H), 6.65 (d, J = 8.6 Hz, 2H), 5.30 (td, J = 7.3, 1.2 Hz, 1H), 3.50 (t, J = 6.5 Hz, 2H), 2.15 (q, J = 6.5 Hz, 2H), 1.90 (s, 3H), 1.04 (s, 9H); ^{13}C NMR (100 MHz, $CDCl_3$): δ 154.3, 139.0, 135.59, 134.2, 133.0, 129.8, 128.7, 127.7, 122.4, 119.3, 62.7, 35.4, 32.6, 26.5, 19.4. HRMS-ESI (*m*/*z*): $[M+H]^+$ calculated for $C_{27}H_{32}O_2Si$ 417.2250, found 417.2258.

4-(4-methoxyphenyl)-4-phenylbut-3-en-1-ol (1k)



^{1}HNMR ($CDCl_3$, 400 MHz): δ 7.26 (d, J = 7.5 Hz, 2H), 7.15-7.12 (m, 2H), 7.10-7.05 (t, J = 7.5 Hz, 3H), 6.71 (d, J = 8.8 Hz, 2H), 5.93 (t, J = 7.5 Hz, 1H), 3.68 (s, 3H), 3.59 (d, J = 6.7 Hz, 2H), 2.27 (dd, J = 14.1, 6.8 Hz, 2H), 1.54 (brs, 1H).^{13}C NMR ($CDCl_3$, 100 MHz): δ 158.8, 143.6, 131.0, 129.8, 128.3, 128.2, 127.3, 127.0, 123.4, 113.4, 62.6, 55.2, 33.2. HRMS-ESI (*m*/*z*): $[M+H]^+$ calculated for $C_{17}H_{18}O_2$ 255.1385, found 255.1385.

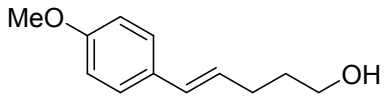
4,4-bis(4-methoxyphenyl)but-3-en-1-ol (1l)



^{1}HNMR ($CDCl_3$, 400 MHz): δ 7.18-7.14 (m, 2H), 7.12-7.09 (m, 2H), 6.92-6.88 (m, 2H), 6.81-6.78 (m, 2H), 5.96 (t, J = 7.5 Hz, 1H), 3.83 (s, 3H), 3.78 (s, 3H), 3.70 (t, J = 6.6 Hz, 2H), 2.40 (dd, J = 14.0, 6.7 Hz, 2H); ^{13}C NMR ($CDCl_3$, 100 MHz): δ 158.8, 158.6, 143.4, 135.5, 132.3, 131.0, 128.4, 123.2, 113.6, 113.4, 62.7, 55.3, 55.2, 33.3. HRMS-ESI (*m*/*z*): $[M+H]^+$ calculated for $C_{18}H_{20}O_3$ 285.1491, found 285.1494.

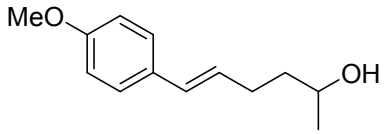
Alkenols **1m-o** were prepared according to reported method.[2]

(*E*)-5-(4-methoxyphenyl)pent-4-en-1-ol (1m)



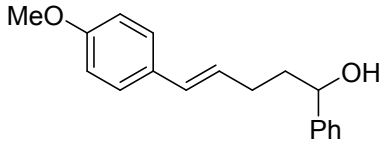
^{1}HNMR ($CDCl_3$, 400 MHz): δ 7.21 (d, J = 8.7 Hz, 2H), 6.86 (d, J = 8.7 Hz, 2H), 6.34 (d, J = 11.0 Hz, 1H), 6.06 (dt, J = 15.8, 6.9 Hz, 1H), 3.79 (s, 3H), 3.64 (t, J = 5.7 Hz, 2H), 3.44 (brs, 1H), 2.43-2.37 (m, 1.8 Hz, 2H), 1.75-1.67 (m, 2H). Data are consistent with literature values.[3]

(*E*)-6-(4-methoxyphenyl)hex-5-en-2-ol (1n)



^{1}HNMR ($CDCl_3$, 400 MHz): δ 7.27 (d, J = 8.1 Hz, 2H), 6.83 (d, J = 8.6 Hz, 2H), 6.36 (d, J = 16.0 Hz, 1H), 6.12-6.04 (m, 1H), 3.88-3.83 (m, 1H), 3.79 (s, 3H), 2.34-2.25 (m, 2H), 1.67-1.57 (m, 2H), 1.47 (brs, 1H), 1.22 (d, J = 6.2 Hz, 3H); ^{13}C NMR ($CDCl_3$, 100 MHz): δ ^{13}C NMR (101 MHz, $CDCl_3$) δ 158.7, 130.5, 129.6, 128.1, 127.0, 113.9, 67.6, 55.3, 38.8, 29.3, 23.5. HRMS-ESI (*m*/*z*): $[M+H]^+$ calculated for $C_{13}H_{18}O_2$ 207.1380, found 207.1387.

(*E*)-5-(4-methoxyphenyl)-1-phenylpent-4-en-1-ol (1o)



^{1}HNMR ($CDCl_3$, 400 MHz): δ 7.37-7.25 (m, 7H), 6.83 (d, J = 8.7 Hz, 2H), 6.35 (d, J = 15.8 Hz, 1H), 6.08 (dt, J = 15.7, 6.9 Hz, 1H), 4.74 (t, J = 6.5 Hz, 1H), 3.79 (s, 3H), 2.35-2.21 (m, 2H), 2.02-1.85 (m, 3H); ^{13}C NMR ($CDCl_3$, 100 MHz): δ 158.7, 144.6, 130.5, 129.8, 128.5, 127.8, 127.6, 127.0, 125.9, 113.9, 74.0, 55.3, 38.6, 29.3. HRMS-ESI (*m*/*z*): $[M+H]^+$ calculated for $C_{18}H_{20}O_2$ 269.1536, found 269.1544.

(*E*)-5-(4-methoxyphenyl)pent-4-enoic acid (1p)

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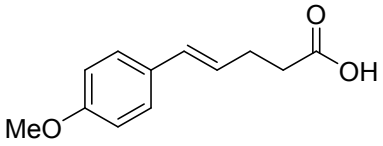
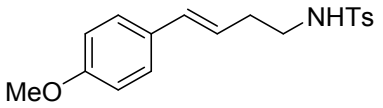
[2] Logan, A.W.J.; Parker, J.S.; Hallside, M.S.; Burton J. W. *Org. Lett.* 2012, 14, 2940.

[3] Belanger, G.; Levesque, F.; Paquet, J.; Barbe, G. *J. Org. Chem.* 2005, 70, 291.



^{1}HNMR ($CDCl_3$, 400 MHz): δ 7.27 (d, J = 8.7 Hz, 2H), 6.83 (d, J = 8.7 Hz, 2H), 6.39 (d, J = 15.8 Hz, 1H), 6.10-6.03 (m, 1H), 3.80 (s, 3H), 2.53-2.52 (m, 4H). Acid **1P** was prepared according to reported method and data are consistent with literature values.[4]

(*E*)-N-(4-(4-methoxyphenyl)but-3-en-1-yl)-4-methylbenzenesulfonamide (1q)



Amide **1q** was prepared according to reported method.[5] ^{1}HNMR ($CDCl_3$, 400 MHz): δ 7.74 (d, J = 8.3 Hz, 2H), 7.28 (d, J = 8.1 Hz, 2H), 7.20 (d, J = 8.7 Hz, 2H), 6.82 (d, J = 8.7 Hz, 2H), 6.29 (d, J = 15.8 Hz, 1H), 5.82 (dt, J = 15.7, 7.1 Hz, 1H), 4.68 (t, J = 6.0 Hz, 1H), 3.80 (s, 3H), 3.07 (q, J = 6.5 Hz, 2H), 2.41 (s, 3H), 2.33 (qd, J = 6.8, 0.9 Hz, 2H); ^{13}C NMR (100 MHz, $CDCl_3$): δ 159.1, 143.3, 136.9, 132.5, 129.7, 129.6, 127.2, 127.1, 123.22, 113.9, 55.2, 42.6, 32.9, 21.5. HRMS-ESI (m/z): $[M+H]^+$ calculated for $C_{18}H_{21}NO_3S$ 332.1320, found 332.1319.

3. General prodedure for photocatalytic cycloetherification of alkenols

To a 10 mL round bottom flask equipped with a magnetic stir bar were added alkenols **1** (0.2 mmol), diethyl 2-bromomalonate (0.24 mmol), K_2HPO_4 (0.4 mmol), [*fac*-$Ir(ppy)_3$] (0.002 mmol) and dry DMF (2 mL). The mixture was irradiated with blue LEDs (1W) at room temperature for 3 hours. Then water was added and the aqueous layer was extracted with ethyl acetate. The combined organic layers were washed with brine, dried over anhydrous Na_2SO_4, concentrated, the residue was purified by flash column chromatography to give the final products **2**.

4. Stereochemical determination of cycloetherification product 2a

[4] R. J. Perkins, H. C. Xu, J. M. Campbell, K. D. Moeller, *Beilstein J. Org. Chem.* 2013**,** *9,* 1630.
[5] S. E. Denmaark, H. M. Chi, *J. Am. Chem. Soc.* 2014, 136, 8915

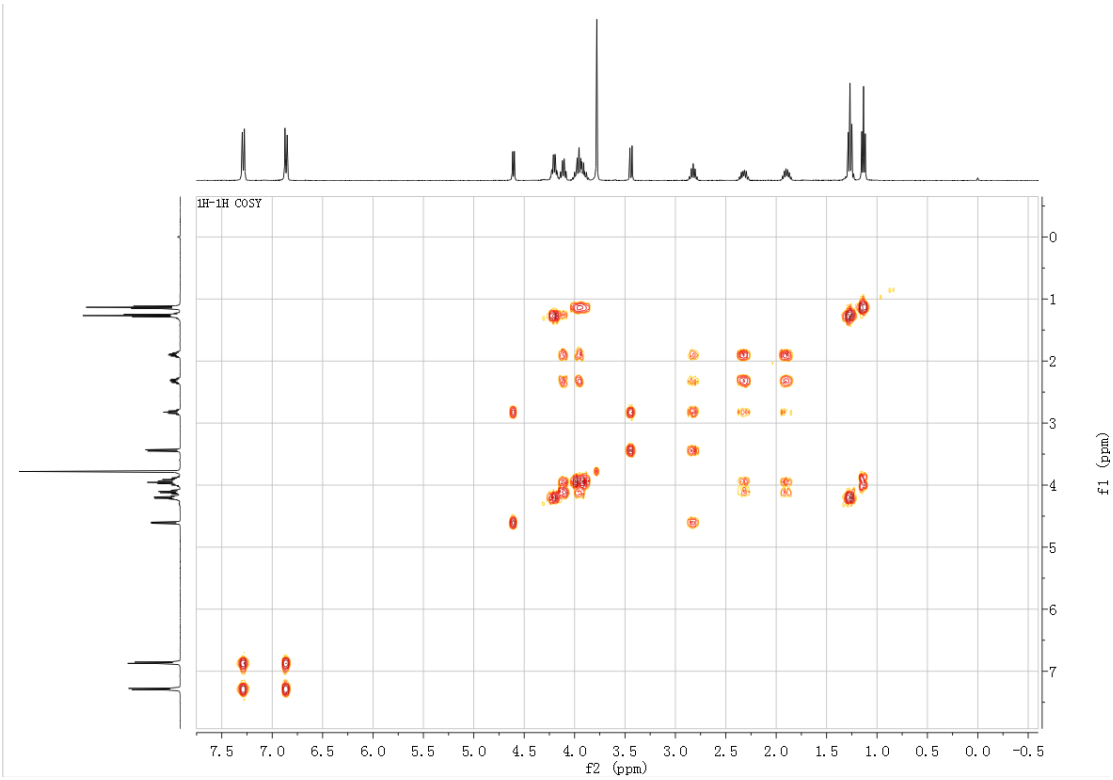


Fig. 1 COSY spectrum of the product **2a**.

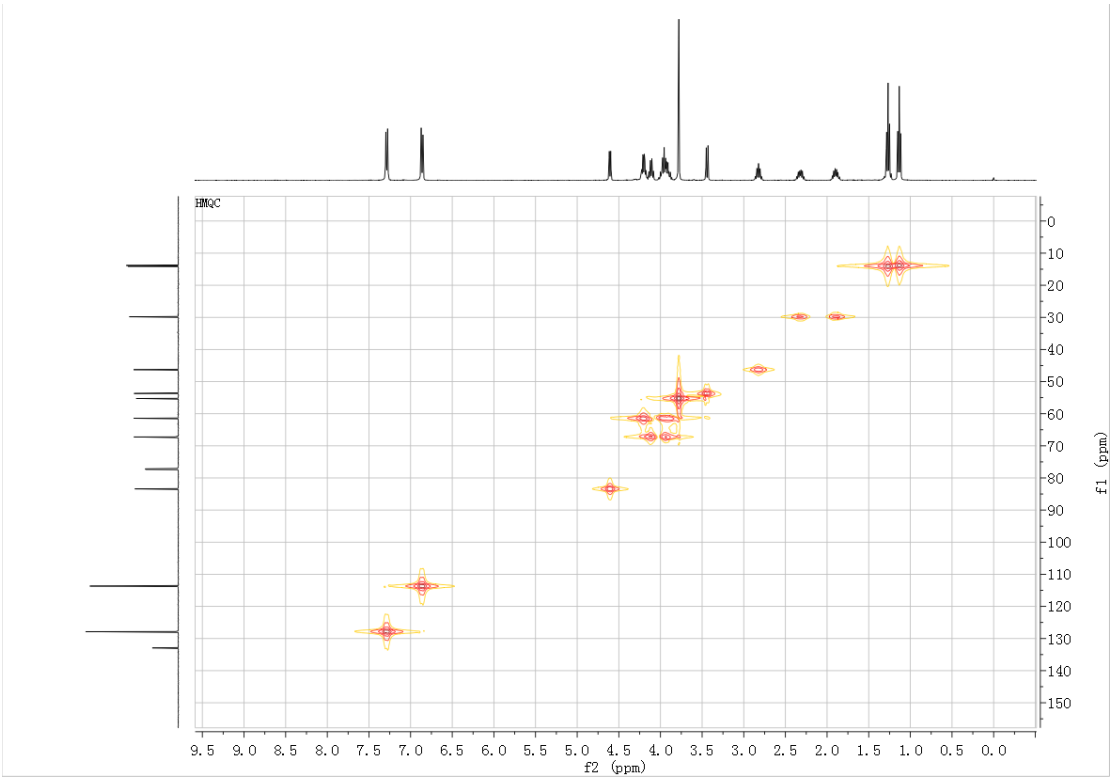


Fig. 2 HMQC spectrum of the product **2a**.

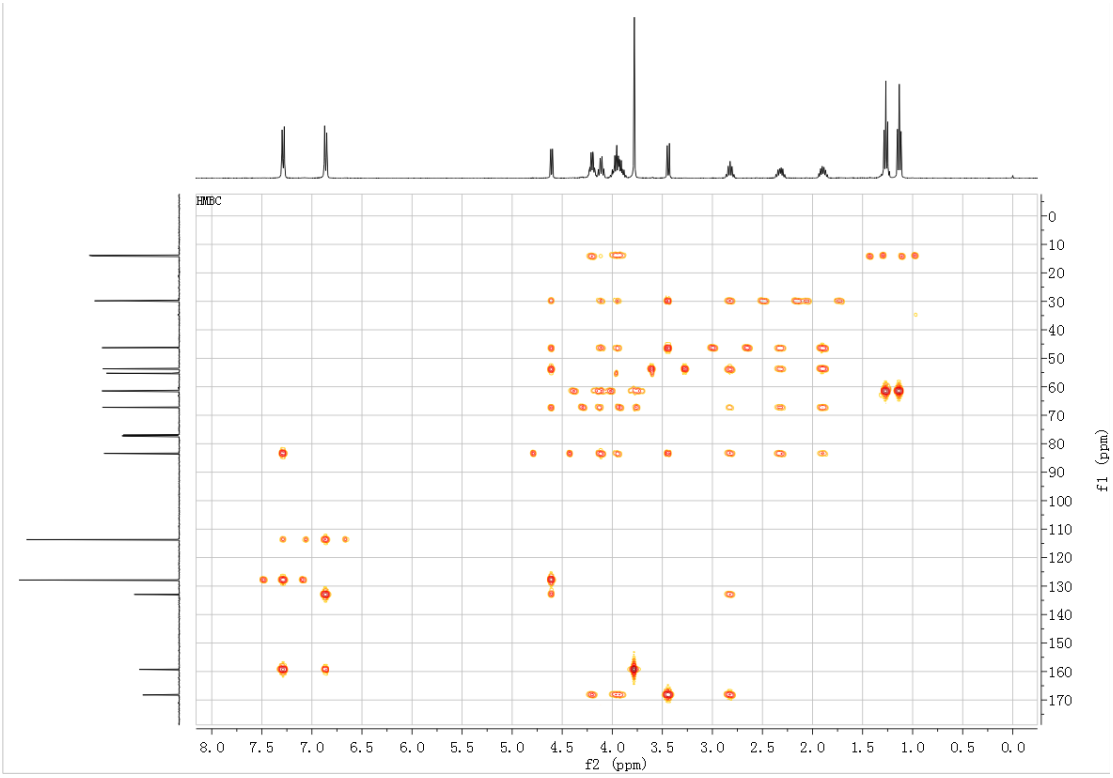
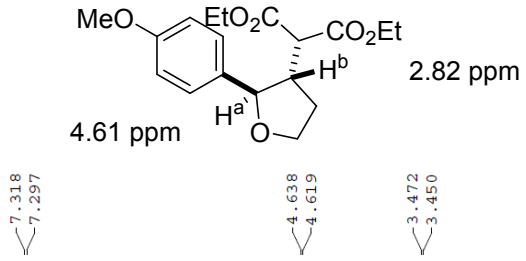


Fig. 3 HMBC spectrum of the product **2a**.



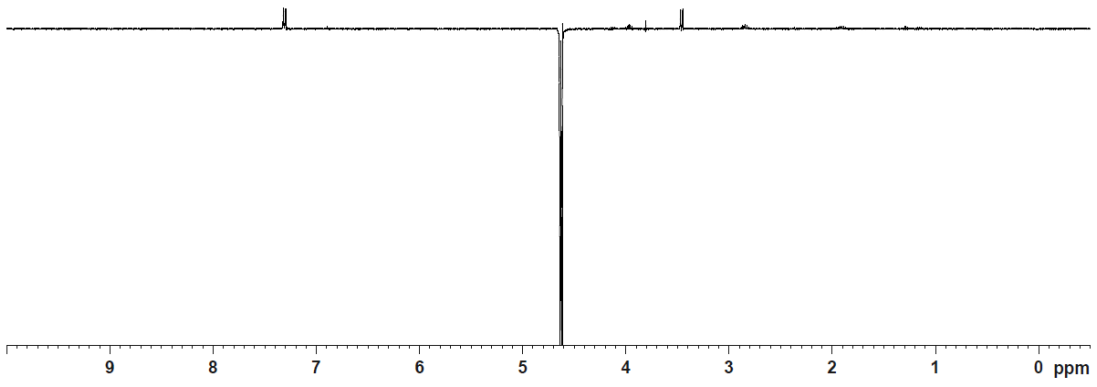
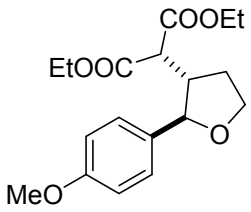


Fig. 4 NOE spectrum of the product **2a**. (There is no NOE between H^a and H^b.)

The NOE data indicate that product **2a** should be *trans* configuration. The stereo-chemistry of all other products was inferred by analogy.

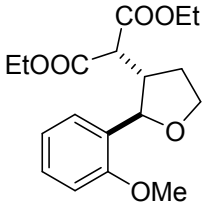
5. Characterization of photocatalytic cycloetherification products

diethyl 2-(2-(4-methoxyphenyl)tetrahydrofuran-3-yl)malonate (2a)



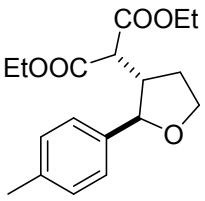
^{1}HNMR ($CDCl_3$, 400 MHz): δ 7.26 (d, J = 8.6 Hz, 2H), 6.83 (d, J = 8.5 Hz, 2H), 4.58 (d, J = 6.9 Hz, 1H), 4.29-4.13 (m, 2H), 4.08 (dd, J = 14.4, 7.4 Hz, 1H), 4.01-3.83 (m, 3H), 3.75 (s, 3H), 3.41 (d, J = 8.7 Hz, 1H), 2.89-2.74 (m, 1H), 2.29 (dd, J = 13.1, 6.7 Hz, 1H), 1.87 (dd, J = 13.3, 6.4 Hz, 1H), 1.24 (t, J = 7.1 Hz, 3H), 1.11 (t, J = 7.1 Hz, 3H); ^{13}C NMR ($CDCl_3$, 100 MHz): δ 168.1, 168.0, 159.1, 132.8, 127.7, 113.5, 83.3, 67.1, 61.4, 61.3, 55.1, 53.5, 46.1, 29.7, 13.9, 13.7. HRMS-ESI (*m*/*z*): $[M+H]^+$ calculated for $C_{18}H_{24}O_6$ 337.1651, found 337.1650.

diethyl 2-(2-(2-methoxyphenyl)tetrahydrofuran-3-yl)malonate (2b)



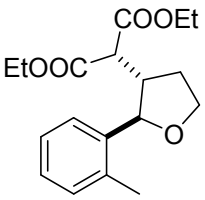
^{1}HNMR ($CDCl_3$, 400 MHz): δ 7.35 (dd, J = 7.5, 1.3 Hz, 1H), 7.27-7.17 (m, 1H), 6.93 (t, J = 7.5 Hz, 1H), 6.83 (d, J = 8.2 Hz, 1H), 5.06 (d, J = 6.9 Hz, 1H), 4.27-4.17 (m, 2H), 4.13 (dd, J = 14.4, 7.9 Hz, 1H), 4.00 (dt, J = 15.9, 6.3 Hz, 2H), 3.92 (dd, J = 10.8, 7.2 Hz, 1H), 3.79 (s, 3H), 3.56 (d, J = 8.1 Hz, 1H), 3.00-2.82 (m, 1H), 2.36-2.17 (m, 1H), 2.02 (td, J = 13.0, 7.1 Hz, 1H), 1.28 (t, J = 7.1 Hz, 3H), 1.13 (t, J = 7.1 Hz, 3H); ^{13}C NMR ($CDCl_3$, 100 MHz): δ 168.4, 168.3, 156.7, 129.5, 128.6, 127.1, 120.5, 110.3, 78.6, 67.5, 61.3, 61.2, 55.1, 53.4, 45.2, 30.0, 14.0, 13.8. HRMS-ESI (*m*/*z*): $[M+H]^+$ calculated for $C_{18}H_{24}O_6$ 337.1651, found 337.1650.

diethyl 2-(2-p-tolyltetrahydrofuran-3-yl)malonate (2c)



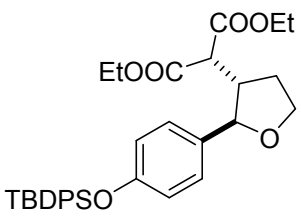
^{1}HNMR ($CDCl_3$, 400 MHz): δ 7.25 (d, J = 7.7 Hz, 2H), 7.14 (d, J = 7.9 Hz, 2H), 4.65 (d, J = 6.6 Hz, 1H), 4.25-4.17 (m, 2H), 4.13 (td, J = 8.2, 6.0 Hz, 1H), 4.04-3.89 (m, 3H), 3.46 (d, J = 8.7 Hz, 1H), 2.92-2.78 (m, 1H), 2.38-2.24 (m, 4H), 1.90 (dt, J = 20.5, 6.4 Hz, 1H), 1.27 (t, J = 7.1 Hz, 3H), 1.15 (t, J = 7.1 Hz, 3H); ^{13}C NMR ($CDCl_3$, 100 MHz): δ 168.2, 168.2, 138.1, 137.4, 129.0, 126.4, 83.5, 67.3, 61.5, 61.4, 53.7, 46.4, 29.6, 21.1, 14.1, 13.8. HRMS-ESI (m/z): $[M+H]^+$ calculated for $C_{18}H_{24}O_5$ 321.1697, found 321.1704.

diethyl 2-(2-o-tolyltetrahydrofuran-3-yl)malonate (2d)



^{1}HNMR ($CDCl_3$, 400 MHz): δ 7.36-7.30 (m, 1H), 7.18 (ddd, J = 9.1, 5.8, 1.6 Hz, 2H), 7.15-7.07 (m, 1H), 4.88 (d, J = 7.8 Hz, 1H), 4.25-4.11 (m, 3H), 3.95 (td, J = 8.4, 6.2 Hz, 1H), 3.91-3.82 (m, 1H), 3.82-3.73 (m, 1H), 3.42 (d, J = 8.3 Hz, 1H), 3.02-2.90 (m, 1H), 2.46-2.39 (m, 1H), 2.37 (d, J = 5.4 Hz, 3H), 1.96 (dt, J = 14.0, 7.9 Hz, 1H), 1.26 (t, J = 7.1 Hz, 3H), 1.09 (t, J = 7.1 Hz, 3H); ^{13}C NMR ($CDCl_3$, 100 MHz): δ 168.2, 168.0, 138.0, 136.4, 130.6, 127.8, 127.1, 126.0, 80.7, 67.3, 61.5,61.4, 53.6, 45.0, 30.4, 19.4, 14.0, 13.7. HRMS-ESI (m/z): $[M+H]^+$ calculated for $C_{18}H_{24}O_5$ 321.1697, found 321.1704.

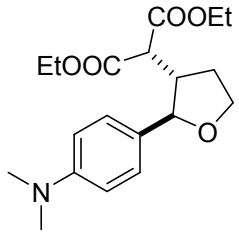
Diethyl 2-(2-(4-((*tert*-butyldiphenylsilyl)oxy)phenyl)tetrahydrofuran-3-yl) malonate (2e)



^{1}HNMR ($CDCl_3$, 400 MHz): δ 7.70 (d, J = 7.0 Hz, 4H), 7.42-7.34 (m, 6H), 7.08 (d, J

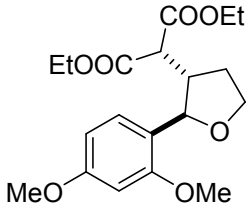
= 8.5 Hz, 2H), 6.72 (d, *J* = 8.5 Hz, 2H), 4.51 (d, *J* = 7.1 Hz, 1H), 4.21-4.17 (m, 2H), 4.06 (dd, *J* = 14.4, 8.0 Hz, 1H), 3.91-3.82 (m, 3H), 3.38 (d, *J* = 8.8 Hz, 1H), 2.81-2.73 (m, 1H), 2.29 (td, *J* = 14.2, 7.8 Hz, 1H), 1.85 (td, *J* = 13.6, 7.3 Hz, 1H), 1.25 (t, *J* = 5.6 Hz, 3H), 1.10-1.06 (m, 12H); ^{13}C NMR ($CDCl_3$, 100 MHz): δ 168.2, 168.0, 155.2, 135.5, 133.0, 132.8, 129.8, 127.7, 127.7, 119.4, 83.5, 67.2, 61.5, 61., 53.7, 46.1, 30.0, 26.5, 19.4, 14.1, 13.8. HRMS-ESI (*m*/*z*): $[M+H]^+$ calculated for $C_{33}H_{40}O_6Si$ 561.2667, found 561.2670.

diethyl 2-(2-(4-(dimethylamino)phenyl)tetrahydrofuran-3-yl)malonate (2f)



^{1}HNMR ($CDCl_3$, 400 MHz): δ 7.22 (d, *J* = 8.7 Hz, 2H), 6.70 (d, *J* = 8.7 Hz, 2H), 4.56 (d, *J* = 7.2 Hz, 1H), 4.21-4.17 (m, 2H), 4.10 (dd, *J* = 8.2, 1.8 Hz, 1H), 4.00-3.86 (m, 3H), 3.43 (d, *J* = 8.5 Hz, 1H), 2.92 (s, 6H), 2.82 (d, *J* = 7.8 Hz, 1H), 2.35-2.29 (m, 1H), 1.90 (dd, *J* = 13.5, 6.0 Hz, 1H), 1.28 (d, *J* = 7.1 Hz, 3H), 1.13 (t, *J* = 7.1 Hz, 3H); ^{13}C NMR ($CDCl_3$, 100 MHz): δ 168.4, 168.2, 150.5, 127.6, 118.3, 112.5, 83.7, 67.1, 61.4, 61.4, 53.7, 46.0, 40.7, 29.9, 14.1, 13.9. HRMS-ESI (*m*/*z*): $[M+H]^+$ calculated for $C_{19}H_{27}NO_5$ 350.1967, found 350.1967.

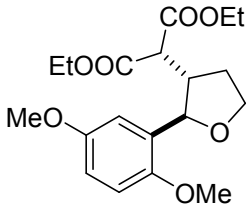
diethyl 2-(2-(2,4-dimethoxyphenyl)tetrahydrofuran-3-yl)malonate (2g)



^{1}HNMR ($CDCl_3$, 400 MHz): δ 7.23 (d, *J* = 8.4 Hz, 1H), 6.50-6.42 (m, 1H), 6.41 (d, *J* = 2.3 Hz, 1H), 4.94 (d, *J* = 7.3 Hz, 1H), 4.27-4.14 (m, 2H), 4.09 (d, *J* = 7.0 Hz, 1H), 4.01-3.85 (m, 3H), 3.77 (d, *J* = 4.3 Hz, 6H), 3.50 (d, *J* = 8.3 Hz, 1H), 2.98-2.81 (m, 1H), 2.27 (dd, *J* = 12.7, 6.0 Hz, 1H), 2.08-1.94 (m, 1H), 1.27 (t, *J* = 7.1 Hz, 1H), 1.12 (t, *J* = 7.1 Hz, 1H); ^{13}C NMR ($CDCl_3$, 100 MHz): δ 168.4, 168.3, 160.4, 158.0, 128.1, 121.5, 104.1, 98.4, 78.6, 67.3, 61.2, 61.2, 55.3, 55.2, 53.5, 44.8, 30.2, 14.0, 13.8.

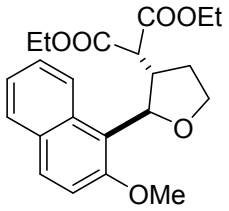
HRMS-ESI (*m*/*z*): $[M+H]^+$ calculated for $C_{19}H_{26}O_7$ 367.1757, found 367.1756.

diethyl 2-(2-(2,5-dimethoxyphenyl)tetrahydrofuran-3-yl)malonate (2h)



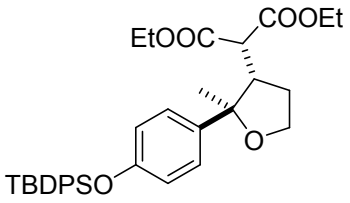
^{1}HNMR ($CDCl_3$, 400 MHz): δ 6.95 (s, 1H), 6.76 (s, 2H), 5.05 (d, J = 6.8 Hz, 1H), 4.25-4.20 (m, 2H), 4.16-4.10 (m, 1H), 4.01 (dd, J = 17.5, 7.2 Hz, 3H), 3.77 (s, 3H), 3.75 (s, 3H), 3.59 (d, J = 7.8 Hz, 1H), 2.83 (t, J = 7.6 Hz, 1H), 2.28-2.20 (m, 1H), 2.09-2.02 (m, 1H), 1.29 (t, J = 7.1 Hz, 3H), 1.16 (t, J = 7.1 Hz, 3H); ^{13}C NMR ($CDCl_3$, 100 MHz): δ 168.5, 168.5, 153.7, 150.9, 130.8, 113.1, 113.0, 111.4, 78.4, 67.6, 61.3, 55.8, 55.7, 53.2, 45.6, 29.8, 14.1, 13.9; HRMS-ESI (*m*/*z*): $[M+H]^+$ calculated for $C_{19}H_{26}O_7$ 367.1757, found 367.1763.

diethyl 2-(2-(2-methoxynaphthalen-1-yl)tetrahydrofuran-3-yl)malonate (2i)



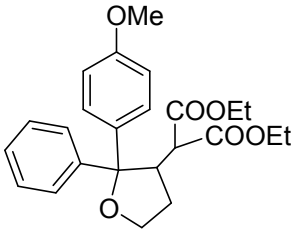
^{1}HNMR ($CDCl_3$, 400 MHz): δ 8.27 (d, J = 8.7 Hz, 1H), 7.77 (d, J = 9.3 Hz, 2H), 7.44 (t, J = 7.7 Hz, 1H), 7.31 (t, J = 7.4 Hz, 1H), 7.23 (d, J = 9.0 Hz, 1H), 5.64 (d, J = 9.0 Hz, 1H), 4.31 (d, J = 8.1 Hz, 2H), 4.18 (dd, J = 7.0, 5.9 Hz, 2H), 4.03 (td, J = 8.4, 6.1 Hz, 1H), 3.92 (s, 3H), 3.65 (dd, J = 10.7, 7.1 Hz, 1H), 3.43 (d, J = 3.4 Hz, 3H), 2.57-2.39 (m, 1H), 2.22-2.14 (m, 1H), 1.25 (t, J = 7.2 Hz, 3H), 0.78 (t, J = 7.1 Hz, 3H); ^{13}C NMR ($CDCl_3$, 100 MHz): δ 168.4, 167.9, 155.7, 132.7, 130.3, 129.5, 128.6, 126.3, 124.2, 123.3, 119.1, 113.1, 77.6, 67.3, 61.3, 61.1, 56.4, 53.9, 43.0, 30.8, 14.0, 13.3. HRMS-ESI (*m*/*z*): $[M+H]^+$ calculated for $C_{22}H_{26}O_6$ 387.1808, found 387.1805.

diethyl 2-(2-(4-(tert-butyldiphenylsilyloxy)phenyl)-2-methyltetrahydrofuran-3-yl)malonate (2j)



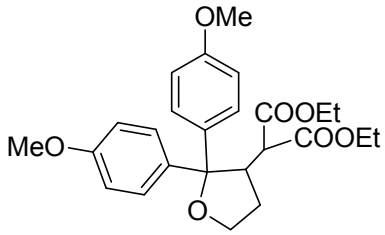
^{1}HNMR ($CDCl_3$, 400 MHz): δ 7.71 (d, J = 6.9 Hz, 4H), 7.38 (ddd, J = 15.0, 7.6, 1.4 Hz, 6H), 7.16 (d, J = 8.7 Hz, 2H), 6.70 (d, J = 8.7 Hz, 2H), 4.20 (dd, J = 13.1, 7.0 Hz, 3H), 3.92 (ddd, J = 12.6, 7.8, 4.6 Hz, 3H), 3.75 (dd, J = 10.8, 7.1 Hz, 1H), 3.44 (d, J = 10.7 Hz, 1H), 3.05 (dt, J = 10.7, 7.0 Hz, 1H), 2.12 (dd, J = 13.2, 6.4 Hz, 1H), 1.73 (dd, J = 13.5, 7.1 Hz, 1H), 1.39-1.19 (m, 9H), 1.18-1.02 (m, 12H); ^{13}C NMR ($CDCl_3$, 100 MHz): δ 168.5, 168.0, 166.6, 154.5, 138.6, 135.5, 133.0, 129.8, 127.7, 126.2, 119.0, 84.8, 65.0, 61.5, 61.4, 53.2, 47.9, 41.6, 29.8, 26.5, 22.3, 19.4, 14.0, 13.7. HRMS-ESI (m/z): $[M+H]^+$ calculated for $C_{34}H_{42}O_6Si$ 575.2829, found 575.2829.

diethyl 2-(2-(4-methoxyphenyl)-2-phenyltetrahydrofuran-3-yl)malonate (2k, cis:trans=1:1)



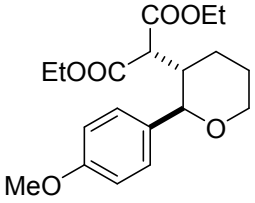
^{1}HNMR ($CDCl_3$, 400 MHz): δ 7.66 (d, J = 7.5 Hz), 7.58 (d, J = 8.8 Hz), 7.40-7.30 (m), 7.20 (ddd, J = 26.9, 15.3, 4.9 Hz), 6.88 (d, J = 8.8 Hz), 6.73 (d, J = 8.9 Hz), 4.32 (q, J = 7.1 Hz), 4.19-4.11 (m), 3.95-3.73 (m), 3.71-3.56 (m), 3.35 (t, J = 9.7 Hz), 2.10 (qd, J = 13.4, 8.3 Hz), 1.97-1.80 (m), 1.23 (t, J = 7.2 Hz), 1.06-0.98 (m); ^{13}C NMR ($CDCl_3$, 100 MHz): δ168.8, 168.8, 168.3, 158.6, 158.4, 145.0, 141.8, 137.0, 133.6, 129.0, 128.3, 127.8, 127.7, 127.5, 127.1, 126.8, 126.5, 113.6, 112.8, 89.5, 89.4, 64.4, 64.4, 62.5, 61., 61.5,61.4, 61.3, 61.3, 55.2, 55.1, 54.3, 54.3, 45.7, 45.7, 30.1, 30.1, 14.0, 13.8, 13.6, 13.5. HRMS-ESI (m/z): $[M+H]^+$ calculated for $C_{24}H_{28}O_6$ 413.1959, found 413.1962.

diethyl 2-(2,2-bis(4-methoxyphenyl)tetrahydrofuran-3-yl)malonate (2l)



^{1}HNMR ($CDCl_3$, 400 MHz): δ 7.57 (d, J = 8.7 Hz, 2H), 7.21 (d, J = 8.7 Hz, 2H), 6.88 (d, J = 8.7 Hz, 2H), 6.72 (d, J = 8.7 Hz, 2H), 4.15 (dd, J = 13.7, 6.6 Hz, 2H), 4.06 (dd, J = 14.2, 8.2 Hz, 1H), 3.91-3.81 (m, 2H), 3.80 (s, 3H), 3.73 (s, 3H), 3.66 (dt, J = 9.9, 6.8 Hz, 2H), 3.33 (d, J = 10.1 Hz, 1H), 2.19-2.05 (m, 1H), 1.87 (dt, J = 15.1, 6.6 Hz, 1H), 1.24 (t, J = 6.9 Hz, 3H), 1.02 (t, J = 7.1 Hz, 3H); ^{13}C NMR ($CDCl_3$, 100 MHz): δ 168.9, 168.3, 158.6, 158.4, 137.2, 134.0, 129.0, 127.8, 113.6, 112.8, 89.3, 64.4, 61.5, 61.3, 55.2, 55.1, 54.5, 45.8, 30.3, 14.1, 13.6. HRMS-ESI (m/z): $[M+H]^+$ calculated for $C_{25}H_{30}O_7$ 443.2070, found 443.2070.

diethyl 2-(2-(4-methoxyphenyl)tetrahydro-2H-pyran-3-yl)malonate (2m)

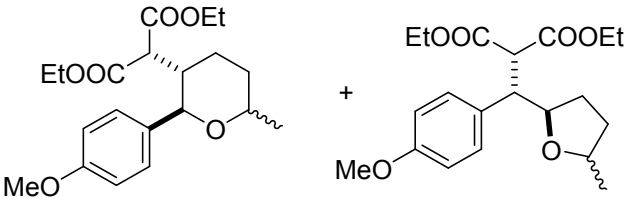


^{1}HNMR ($CDCl_3$, 400 MHz): δ 7.28 (d, J = 8.7 Hz, 2H), 6.86 (d, J = 8.7 Hz, 2H), 4.28 (d, J = 10.1 Hz, 1H), 4.22-4.11 (m, 2H), 4.02 (dddd, J = 21.0, 10.3, 7.0, 3.4 Hz, 3H), 3.79 (s, 3H), 3.55 (td, J = 11.7, 2.0 Hz, 1H), 3.10 (d, J = 4.0 Hz, 1H), 2.48-2.31 (m, 1H), 2.06 (ddd, J = 6.8, 5.0, 2.5 Hz, 1H), 1.87-1.78 (m, 1H), 1.73 (dt, J = 22.1, 7.4 Hz, 1H), 1.28 (t, J = 7.1 Hz, 3H), 1.17 (t, J = 7.1 Hz, 3H); ^{13}C NMR ($CDCl_3$, 100 MHz): δ 168.6, 168.3, 159.5, 132.1, 128.8, 113.8, 82.5, 68.8, 61.2, 61.1, 55.2, 52.5, 42.2, 26.4, 26.2, 14.1, 13.9. HRMS-ESI (m/z): $[M+H]^+$ calculated for $C_{19}H_{26}O_6$ 351.1802, found 351.1805.

diethyl 2-(2-(4-methoxyphenyl)-6-methyltetrahydro-2H-pyran-3-yl)malonate (2n)

and

diethyl 2-((4-methoxyphenyl)(5-methyltetrahydrofuran-2-yl)methyl)malonate (2n') (2n:2n'=1:1.2)



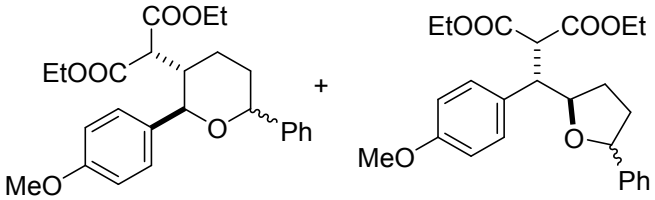
^{1}HNMR ($CDCl_3$, 400 MHz): δ 7.30 (dd, J = 18.0, 8.6 Hz), 6.87 (dd, J = 10.7, 8.7 Hz), 4.71 (d, J = 6.0 Hz), 4.33 (d, J = 10.2 Hz), 4.23-3.96 (m), 3.79 (d, J = 5.3 Hz), 3.60 (d, J = 7.5 Hz), 3.08 (d, J = 3.9 Hz), 2.75-2.61 (m), 2.31 (d, J = 11.9 Hz), 2.05-1.99 (m), 1.84-1.67 (m), 1.58-1.44 (m), 1.29 (dd, J = 13.3, 4.7 Hz), 1.24-1.14 (m); ^{13}C NMR ($CDCl_3$, 100 MHz): δ ^{13}C NMR (101 MHz, $CDCl_3$) δ 168.8, 168.7, 168.7, 168.4, 159.5, 159.0, 132.5, 132.2, 128.9,128.4, 113.9, 113.9, 82.3, 74.4, 74.4, 67.7, 61.4, 61.2, 61.2, 61.0, 55.3, 53.0, 52.9, 52.4, 42.0, 38.3, 33.7, 29.2, 26.4, 22.0, 20.9, 19.2, 14.2, 14.0, 14.0. HRMS-ESI (m/z): $[M+H]^+$ calculated for $C_{20}H_{28}O_6$ 365.1964, found 365.1962.

diethyl 2-(2-(4-methoxyphenyl)-6-phenyltetrahydro-2H-pyran-3-yl)malonate (2o)

and

diethyl 2-((4-methoxyphenyl)(5-phenyltetrahydrofuran-2-yl)methyl)malonate (2o')

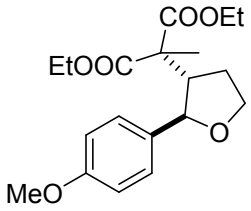
(2o:2o'=1:1.2)



^{1}HNMR ($CDCl_3$, 400 MHz): δ 7.51-7.14 (m, 14H), 6.89 (dd, J = 16.8, 8.6 Hz, 4H), 4.83 (dd, J = 6.9, 3.9 Hz, 1H), 4.73 (d, J = 5.2 Hz, 1H), 4.54 (d, J = 10.3 Hz, 2H), 4.28-4.00 (m, 8H), 3.80 (d, J = 9.4 Hz, 6H), 3.72 (d, J = 8.1 Hz, 1H), 3.16 (d, J = 3.8 Hz, 1H), 2.91 (s, 1H), 2.41 (d, J = 10.4 Hz, 1H), 2.20-2.09 (m, 2H), 2.07-1.86 (m, 4H), 1.85-1.56 (m, 2H), 1.31 (t, J = 7.1 Hz, 4H), 1.23-1.11 (m, 8H); ^{13}C NMR ($CDCl_3$, 100 MHz): δ ^{13}C NMR (101 MHz, $CDCl_3$) δ 168.8, 168.7, 168.6, 168.4, 159.5, 159.1, 142.9, 141.6, 132.4, 131.6, 129.0, 128.5,128.5, 128.2, 127.3, 127.2, 126.4, 125.9, 114.0, 113.8, 82.6, 80.3, 75.5, 72.7, 61.4, 61.3, 61.2, 55.3, 53.0, 52.4, 42.3, 37.2, 34.5, 27.6, 26.7, 21.8, 14.2, 14.0. HRMS-ESI (m/z): $[M+H]^+$ calculated for

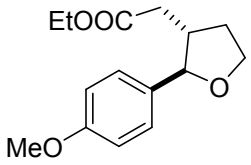
$C_{25}H_{30}O_6$ 427.2121, found 427.2120.

diethyl 2-(2-(4-methoxyphenyl)tetrahydrofuran-3-yl)-2-methylmalonate (3a)



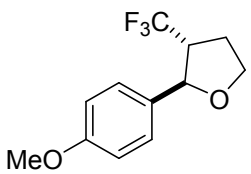
^{1}HNMR ($CDCl_3$, 400 MHz): δ 7.29 (d, J = 8.7 Hz, 2H), 6.85 (d, J = 8.7 Hz, 2H), 4.73 (d, J = 6.4 Hz, 1H), 4.12 (dd, J = 12.0, 7.1 Hz, 2H), 4.05 (d, J = 5.6 Hz, 1H), 3.96 (d, J = 7.1 Hz, 1H), 3.84 (dd, J = 15.4, 8.1 Hz, 1H), 3.79 (s, 3H), 3.72 (dd, J = 10.7, 7.1 Hz, 1H), 3.03 (dt, J = 9.4, 6.1 Hz, 1H), 2.32 (ddd, J = 16.3, 13.2, 8.4 Hz, 1H), 2.06-1.88 (m, 1H), 1.45 (t, J = 7.1 Hz, 3H), 1.21 (t, J = 7.1 Hz, 3H), 1.07 (t, J = 7.1 Hz, 3H); ^{13}C NMR ($CDCl_3$, 100 MHz): δ 171.5, 171.2, 159.2, 134.0, 128.3, 113.6, 81.7, 67.6, 61.3, 61.2, 55.5, 55.3, 50.2, 29.1, 17.4, 14.0, 13.7. HRMS-ESI (m/z): $[M+H]^+$ calculated for $C_{19}H_{26}O_6$ 351.1808, found 351.1810.

ethyl 2-(2-(4-methoxyphenyl)tetrahydrofuran-3-yl)acetate (4a)



^{1}HNMR ($CDCl_3$, 400 MHz): δ 7.26 (d, J = 8.5 Hz, 2H), 6.87 (d, J = 8.5 Hz, 2H), 4.36 (d, J = 7.4 Hz, 1H), 4.18-4.09 (m, 1H), 4.09-3.94 (m, 3H), 3.79 (s, 3H), 2.53-2.40 (m, 2H), 2.40-2.25 (m, 2H), 1.77 (dt, J = 12.3, 7.9 Hz, 1H), 1.20 (t, J = 7.1 Hz, 3H); ^{13}C NMR ($CDCl_3$, 100 MHz): δ 172.2, 159.3, 133.1, 127.6, 113.8, 85.5, 67.6, 60.4, 55.3, 44.1, 36.5, 32.3, 14.1. HRMS-ESI (m/z): $[M+H]^+$ calculated for $C_{15}H_{20}O_4$ 265.1440, found 265.1440.

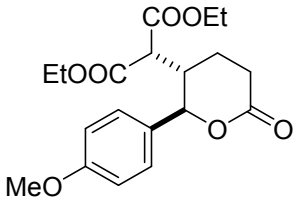
2-(4-methoxyphenyl)-3-(trifluoromethyl)tetrahydrofuran (5a)



^{1}HNMR ($CDCl_3$, 400 MHz): δ 7.35 (d, J = 8.6 Hz, 2H), 6.91 (d, J = 8.7 Hz, 2H), 5.09 (d, J = 6.8 Hz, 1H), 4.24-4.15 (m, 1H), 4.11 (dd, J = 14.9, 8.0 Hz, 1H), 4.01 (dd, J =

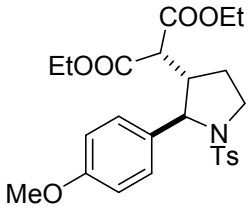
14.1, 6.9 Hz, 1H), 3.83 (s, 1H), 2.63 (td, *J* = 14.1, 7.3 Hz,1H), 2.49-2.33 (m, 1H); ^{13}C NMR ($CDCl_3$, 100 MHz): δ 159.5, 131.4, 127.4, 113.9, 89.5, 67.8, 55.3, 38.3, 27.3. ^{19}F NMR (376MHz, $CDCl_3$): δ -69.3 (S). HRMS-ESI (*m*/*z*): $[M+H]^+$ calculated for $C_{12}H_{13}F_3O_2$ 247.0940, found 247.0935.

diethyl 2-(2-(4-methoxyphenyl)-6-oxotetrahydro-2H-pyran-3-yl)malonate (2p)



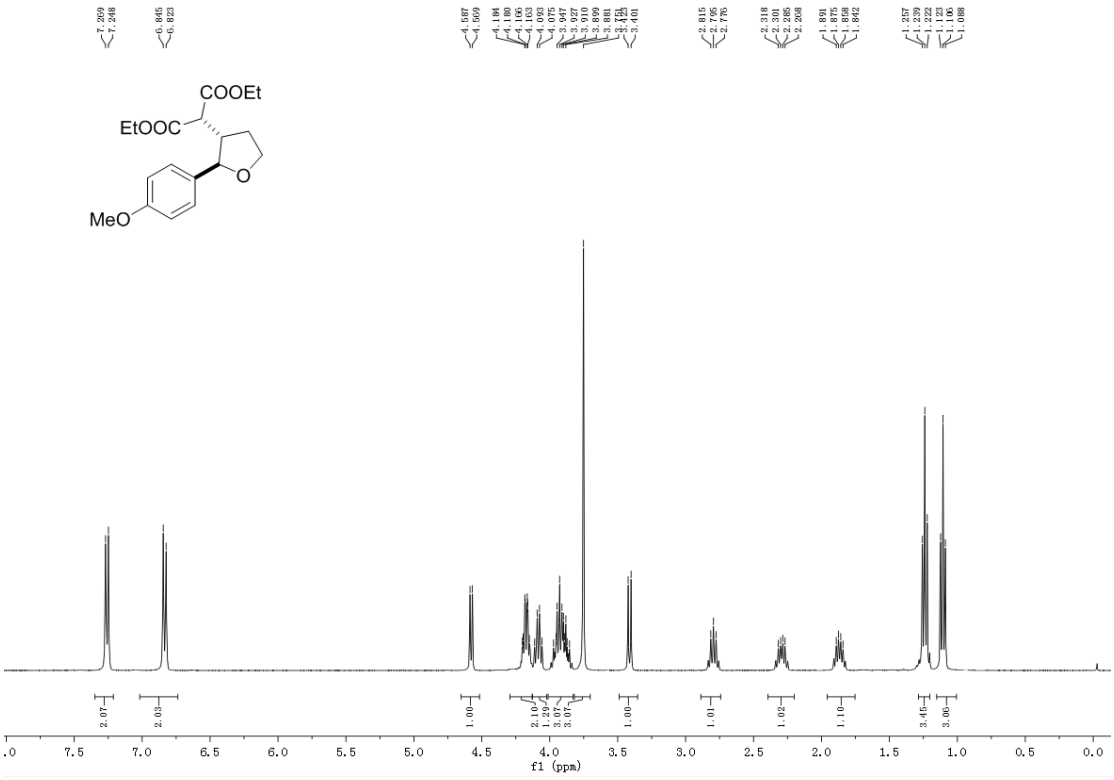
^{1}HNMR ($CDCl_3$, 400 MHz): δ 7.27 (d, *J* = 8.5 Hz, 2H), 6.91 (d, *J* = 8.7 Hz, 2H), 5.34 (d, *J* = 9.8 Hz, 1H), 4.27-4.01 (m, 4H), 3.82 (s, 3H), 3.23 (d, *J* = 4.6 Hz, 1H), 2.80 (dt, *J* = 17.2, 7.7 Hz, 1H), 2.73-2.57 (m, 2H), 2.17-2.04 (m, 2H), 1.29 (t, *J* = 7.1 Hz, 3H), 1.22 (t, *J* = 7.1 Hz, 3H);^{13}C NMR ($CDCl_3$, 100 MHz): δ 171.5, 167.7, 160.0, 129.1, 128.5, 114.1, 82.4, 61.8, 61.6, 55.3, 51.5, 39.3, 28.9, 20.8, 14.0, 13.9. HRMS-ESI (*m*/*z*): $[M+H]^+$ calculated for $C_{19}H_{24}O_7$ 365.1595, found 365.1600.

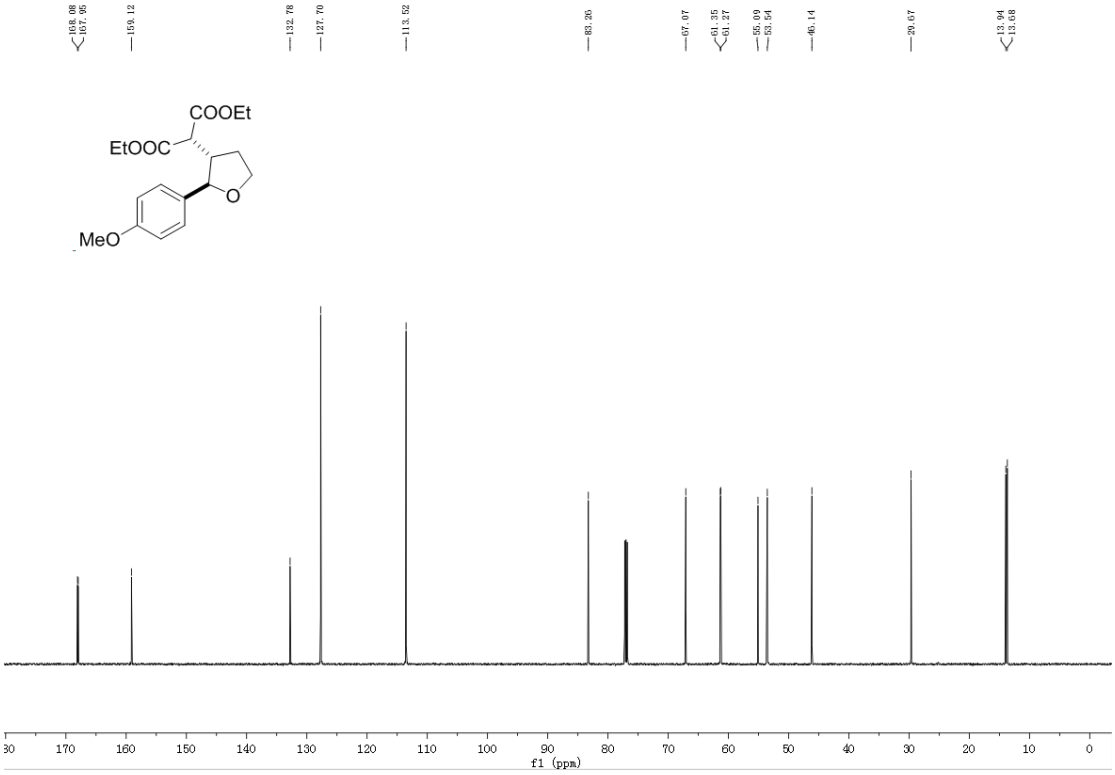
diethyl 2-(2-(4-methoxyphenyl)-1-tosylpyrrolidin-3-yl)malonate (2q)

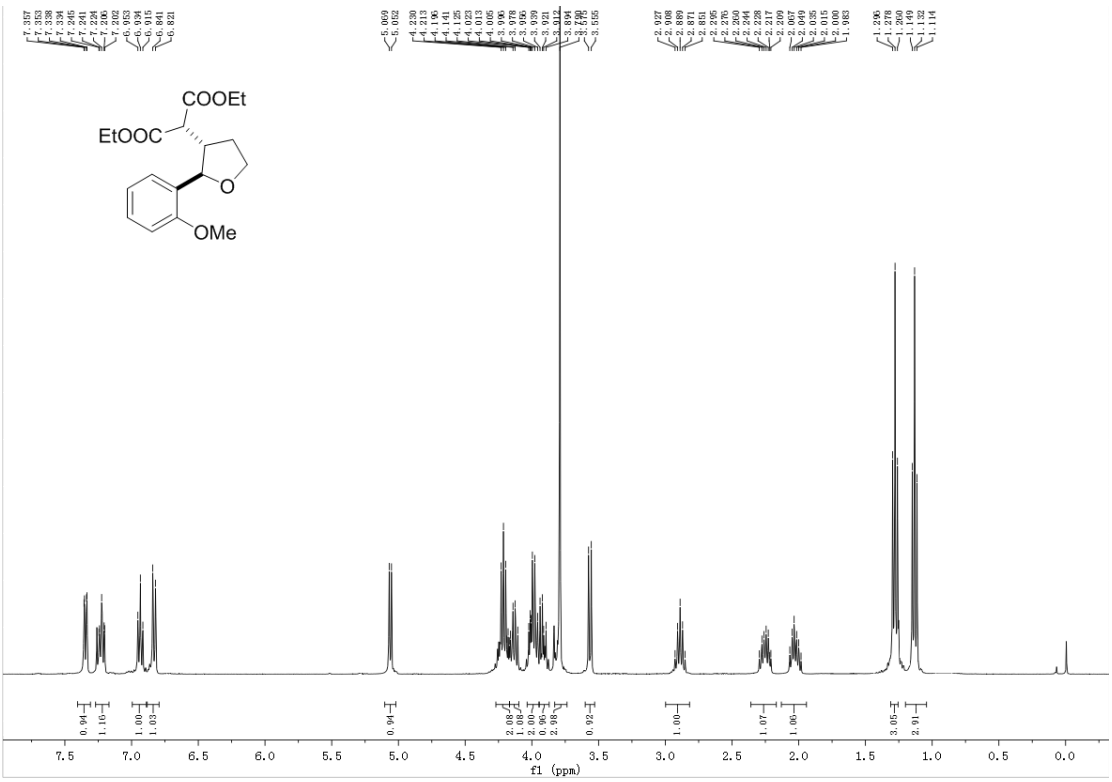


^{1}HNMR ($CDCl_3$, 400 MHz): δ 7.50 (d, *J* = 8.1 Hz, 2H), 7.40 (d, *J* = 8.6 Hz, 2H), 7.28 (d, *J* = 8.0 Hz, 2H), 6.94 (d, *J* = 8.7 Hz, 2H), 4.51 (d, *J* = 7.8 Hz, 1H), 4.19 (q, *J* = 7.1 Hz, 2H), 3.99-3.86 (m, 2H), 3.82 (s, 3H), 3.52-3.38 (m, 2H), 2.93-2.73 (m, 1H), 2.64 (dt, *J* = 10.1, 7.7 Hz, 1H), 2.40 (s, 3H), 2.18 (td, *J* = 12.9, 7.4 Hz, 1H), 1.82 – 1.69 (m, 1H), 1.27 (s, 3H), 1.16 (t, *J* = 7.1 Hz, 3H);^{13}C NMR ($CDCl_3$, 100 MHz): δ 168.0, 168.0, 159.4, 141.0, 140.9, 132.0, 129.5, 129.2, 125.5, 113.9, 67.4, 61.5, 61.4, 55.3, 52.9, 47.1, 39.7, 27.6, 21.3, 14.1, 13.8. HRMS-ESI (*m*/*z*): $[M+H]^+$ calculated for $C_{25}H_{31}NO_7S$ 490.1894, found 490.1894.

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



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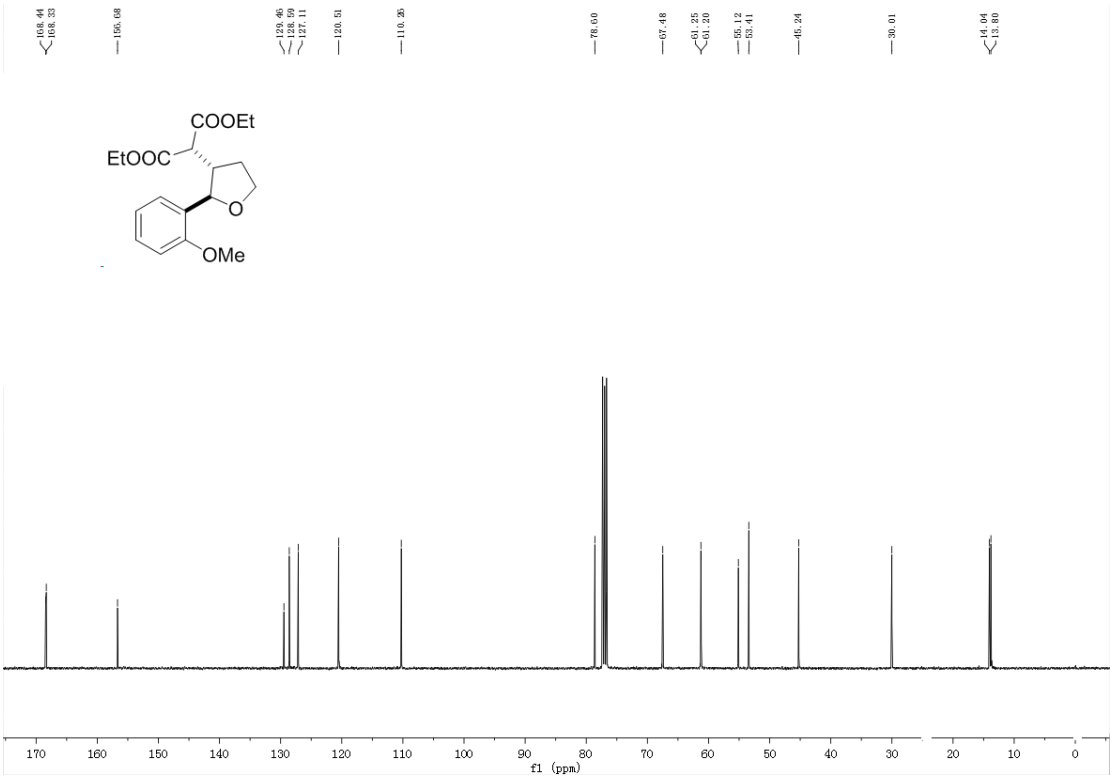
OMe
f1 (ppm)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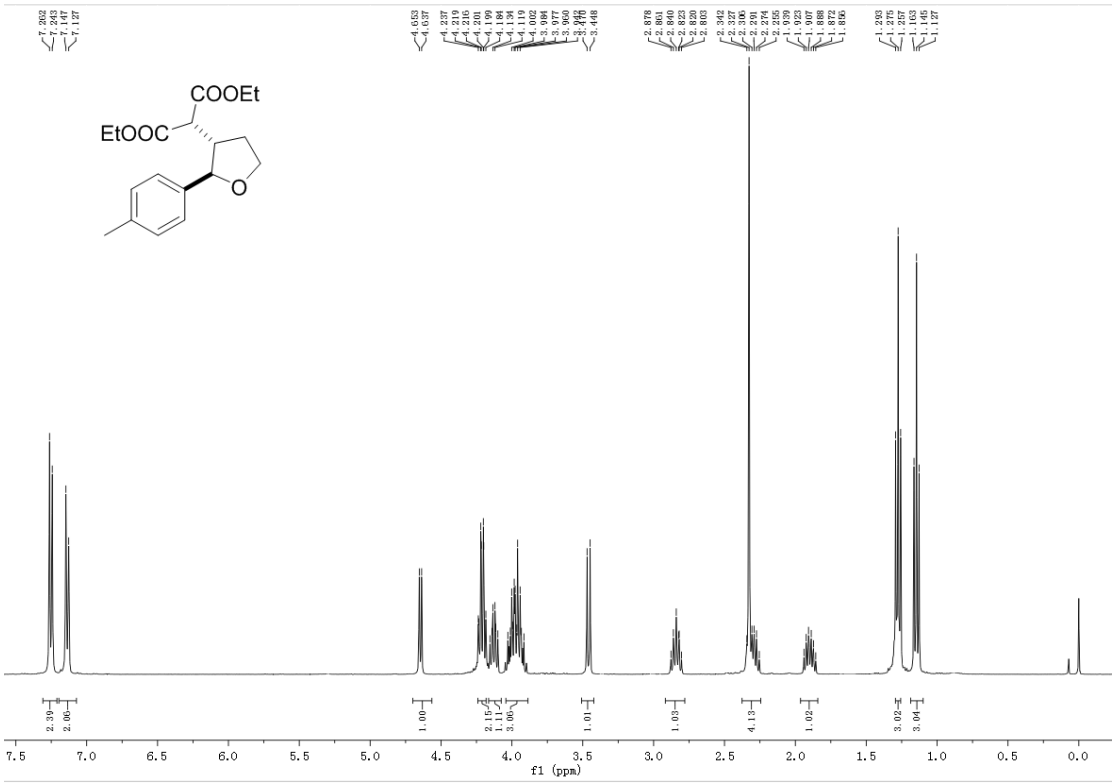
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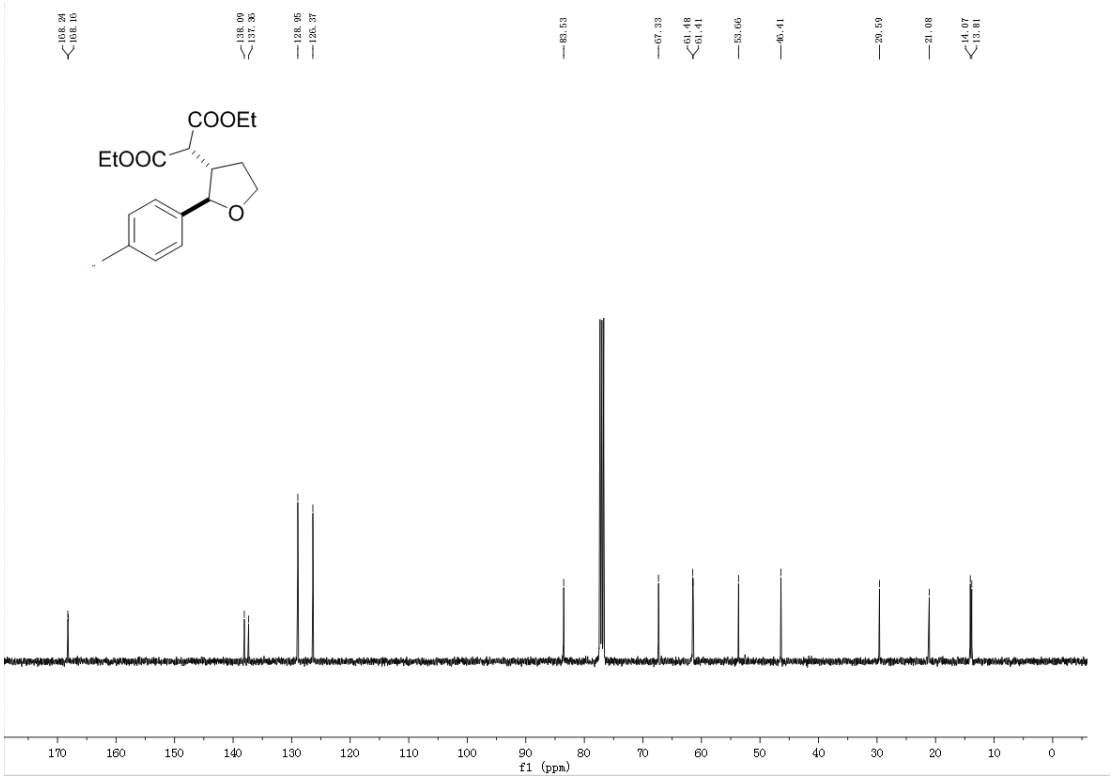
f1 (ppm)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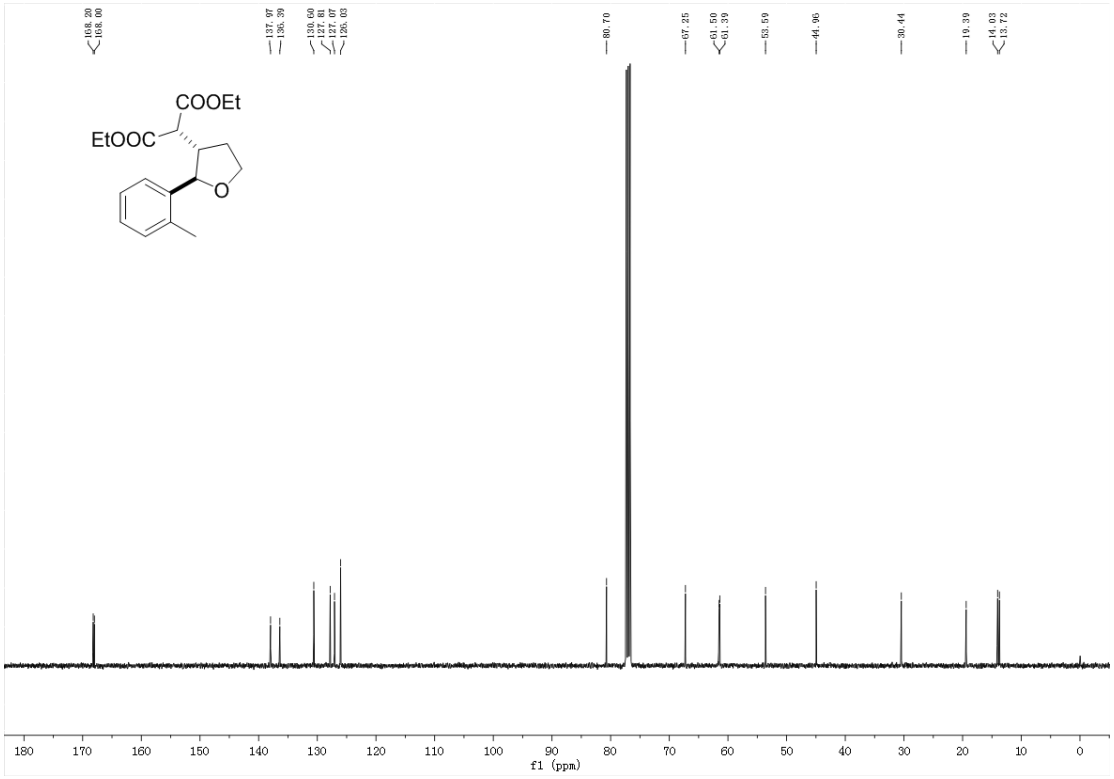
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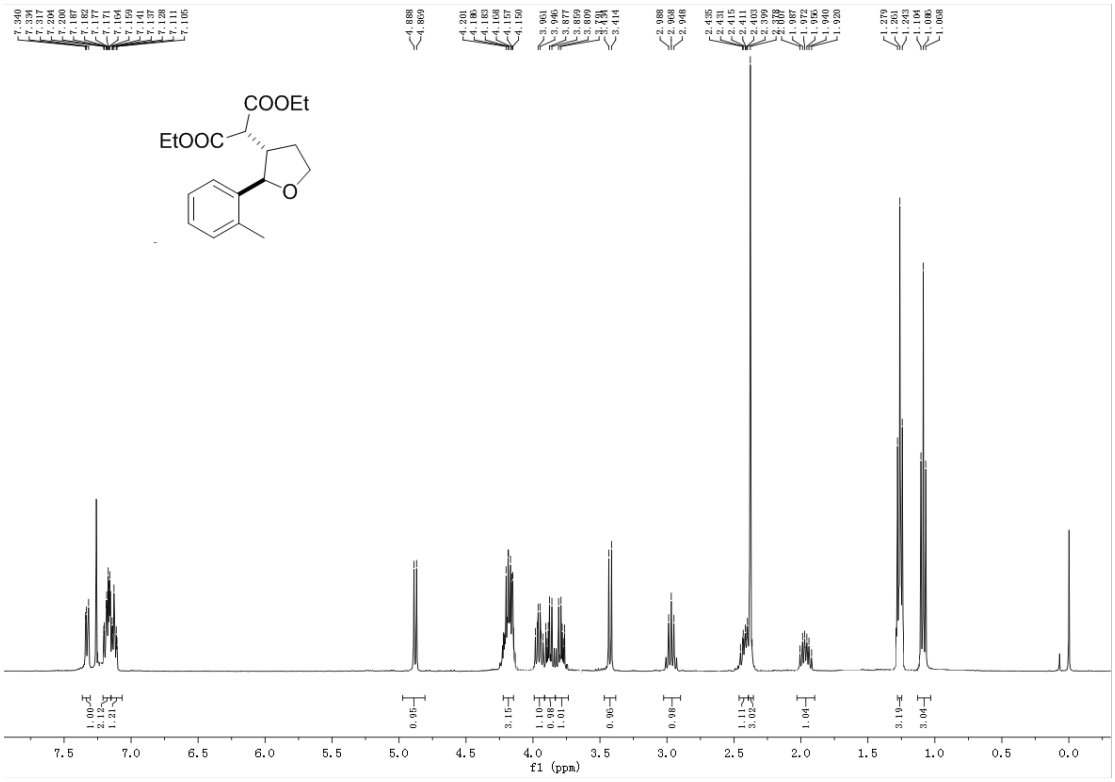
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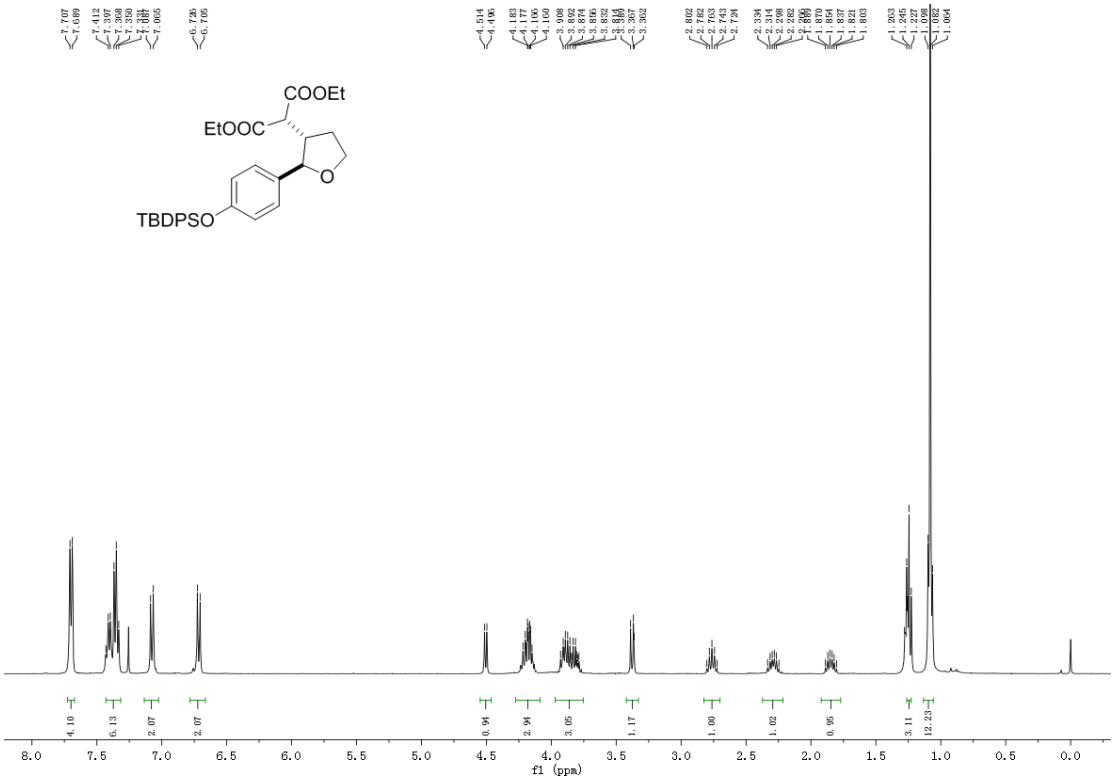
f1 (ppm)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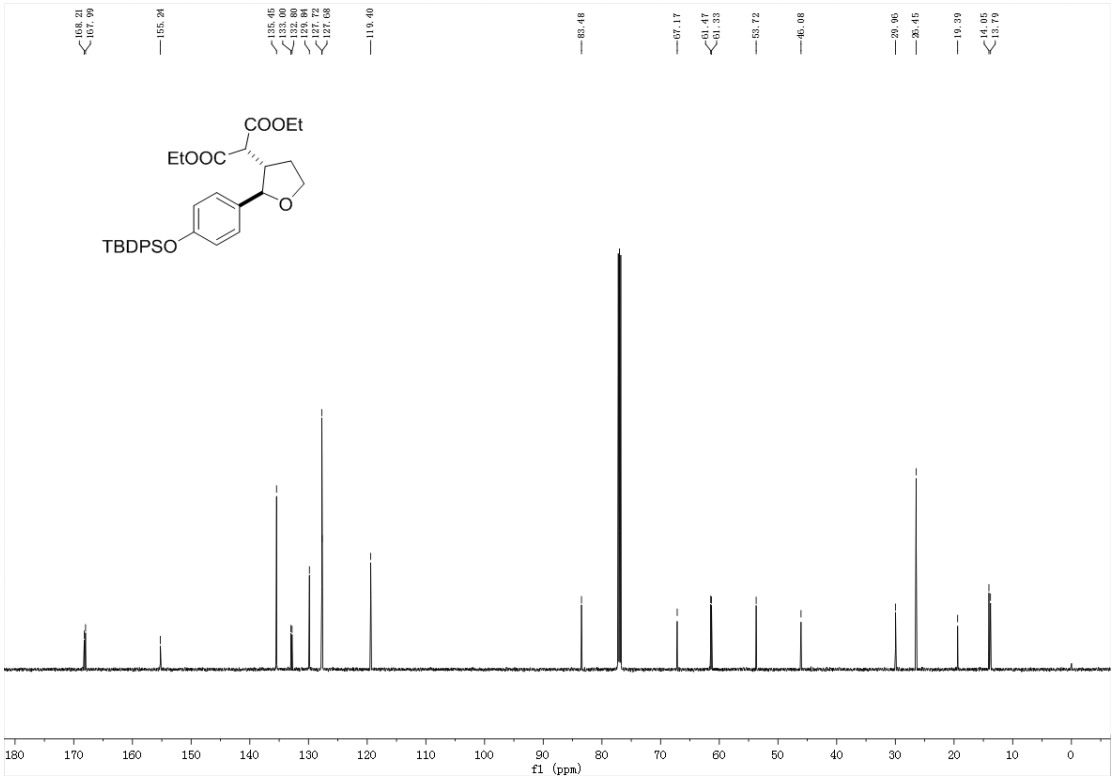
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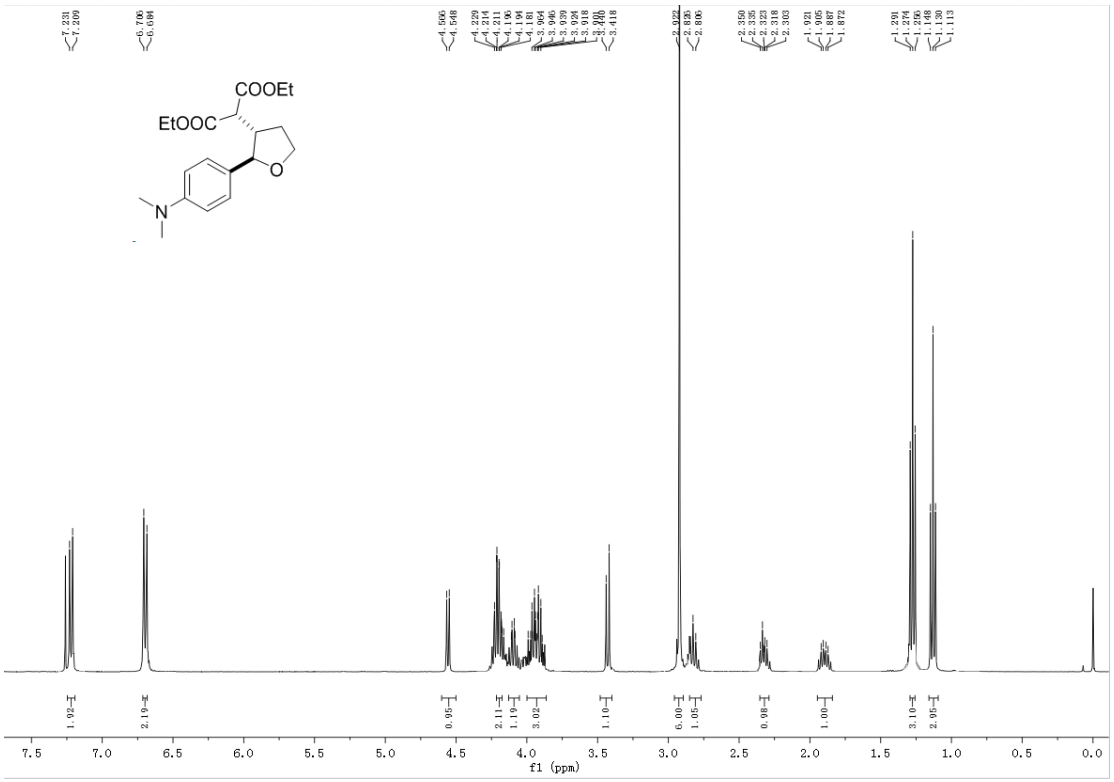
TBDPSO
f1 (ppm)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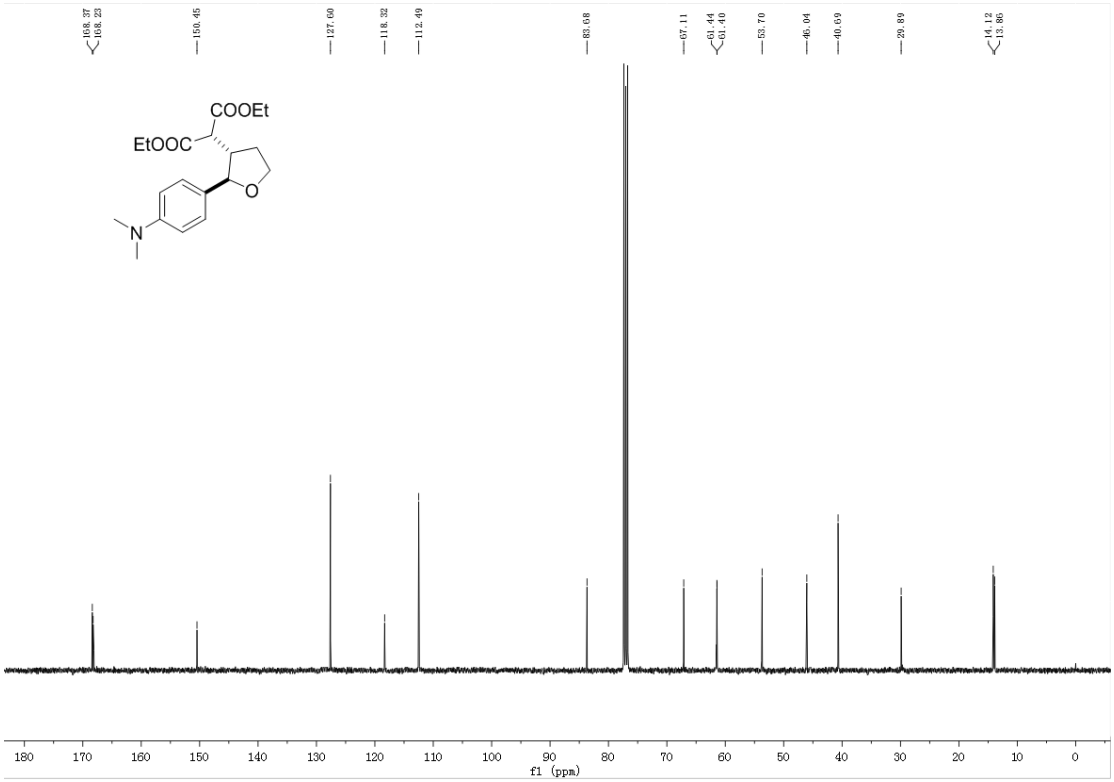
EtOOC
O
N
f1 (ppm)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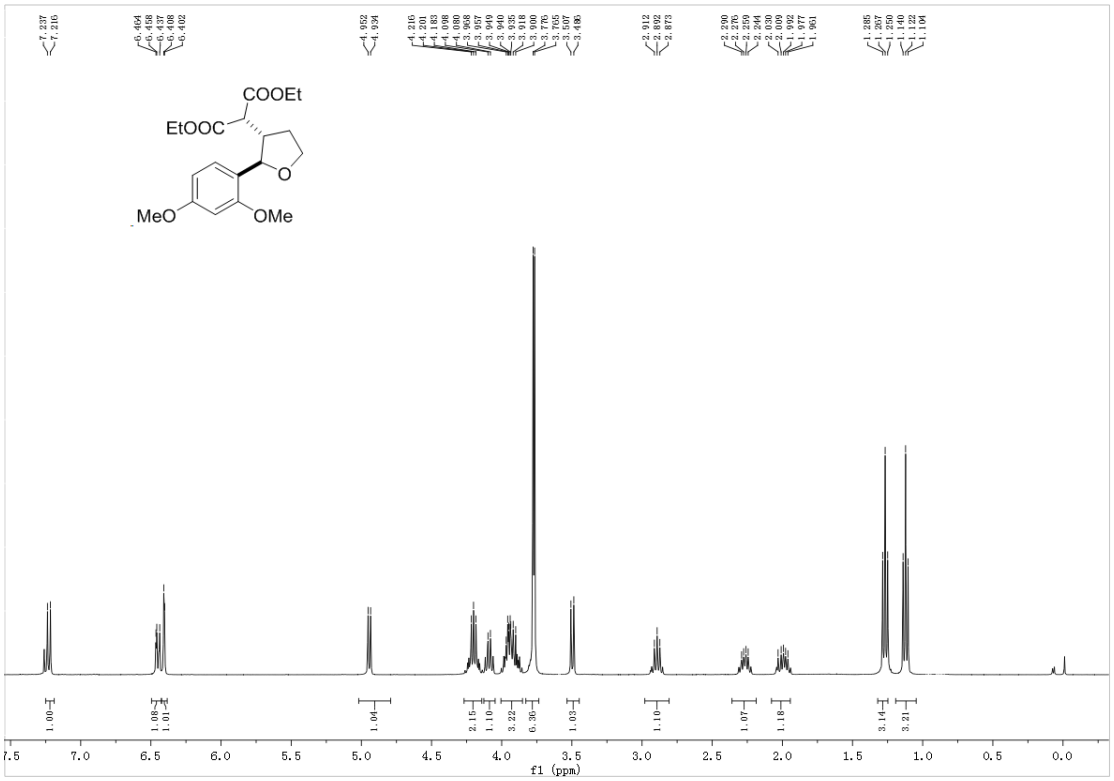
O
N
f1 (ppm)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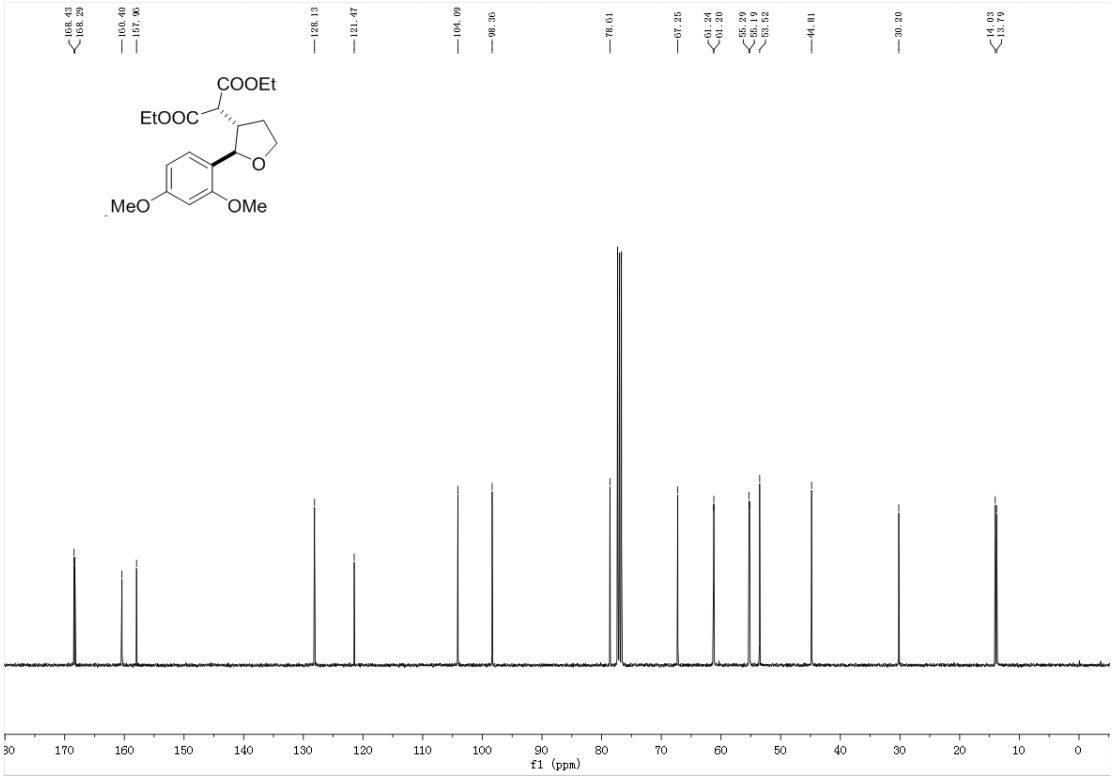
O
MeO
OMe
f1 (ppm)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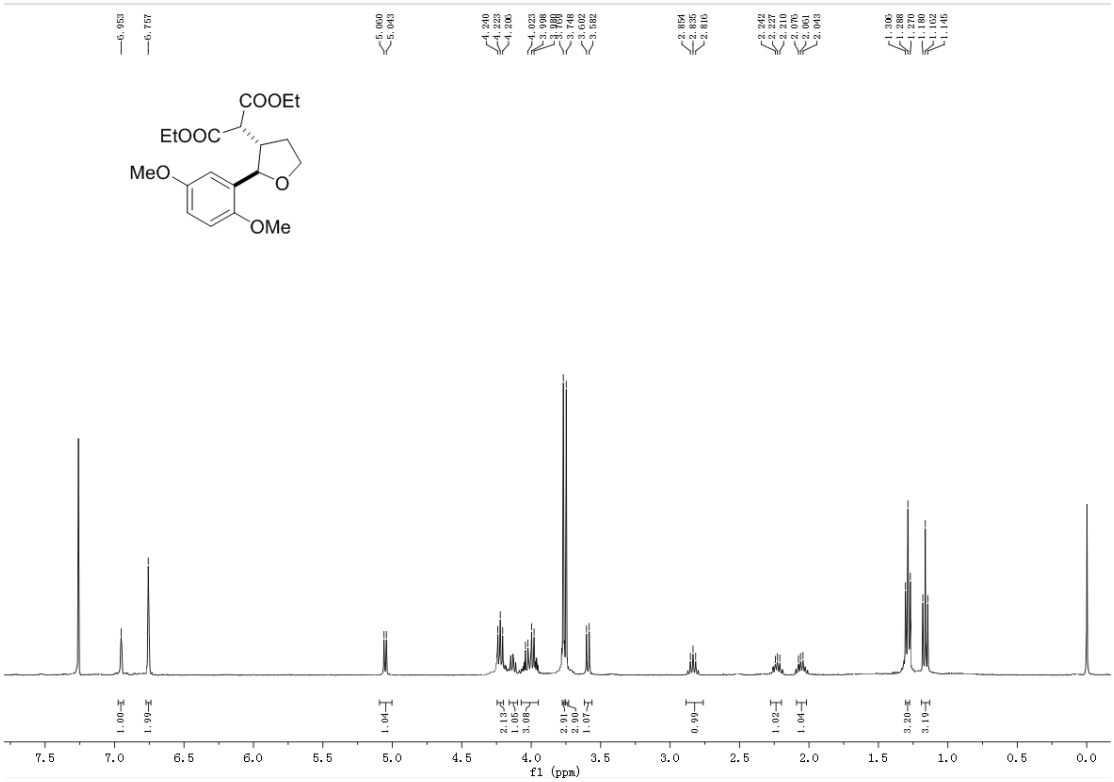
f1 (ppm)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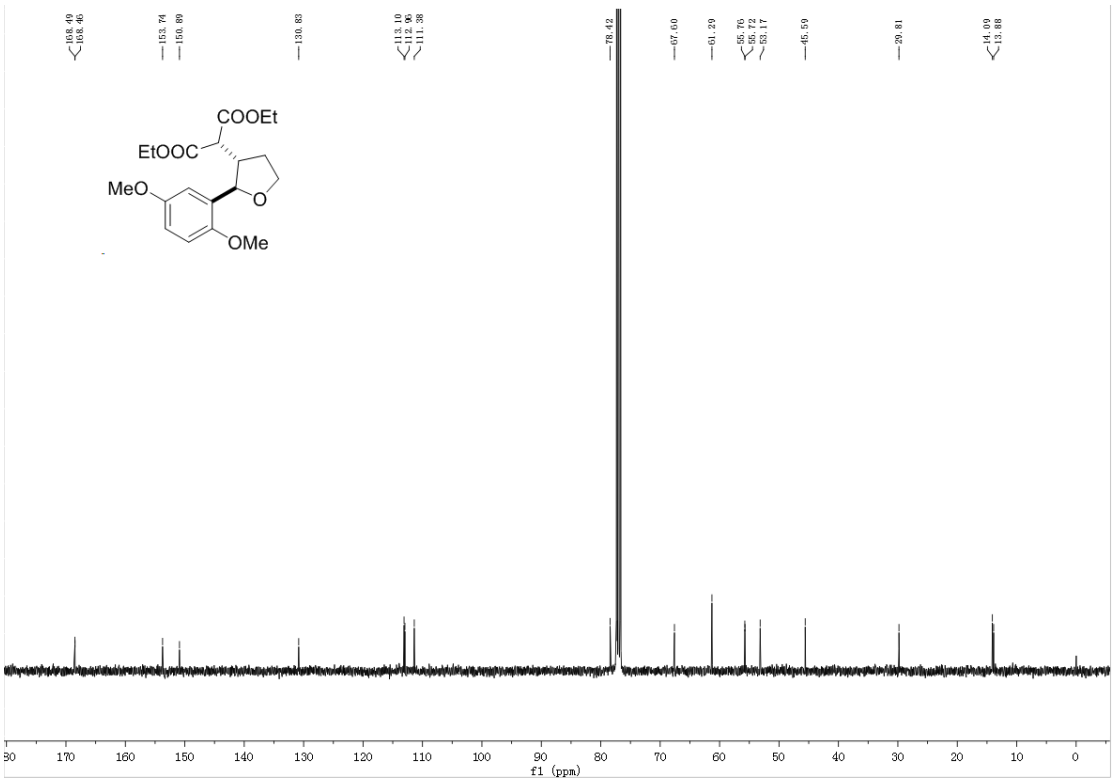
MeO
O
OMe
f1 (ppm)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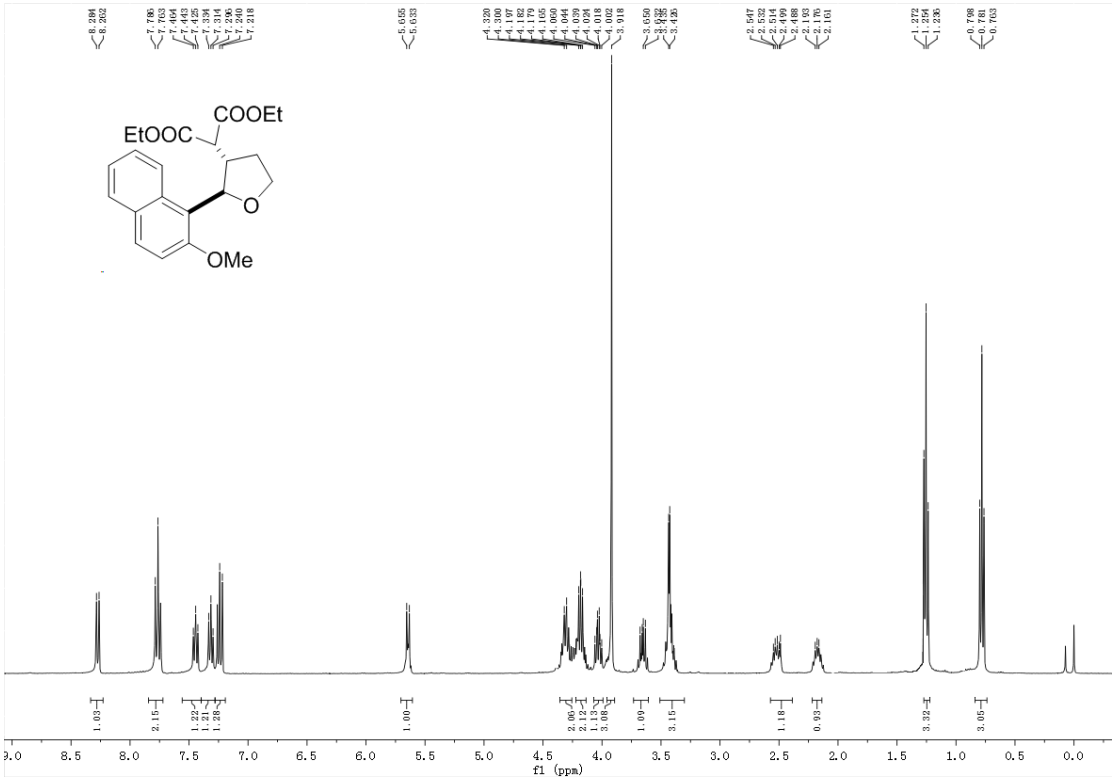
O
OMe
f1 (ppm)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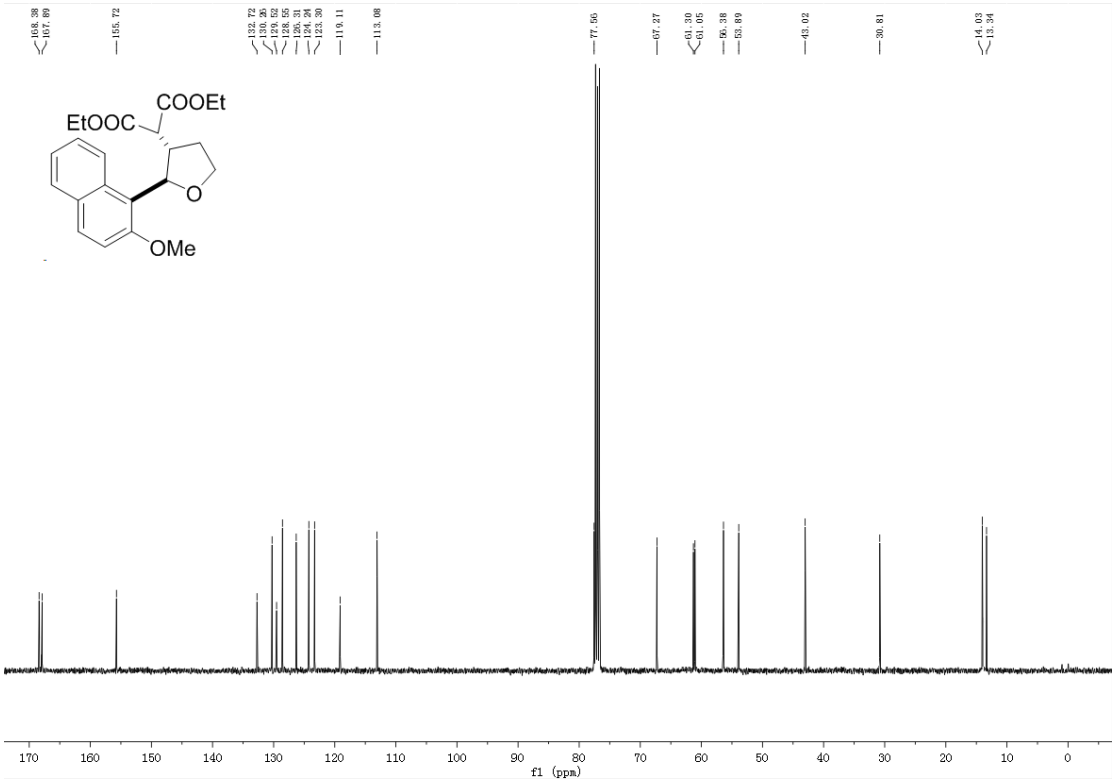
OMe
f1 (ppm)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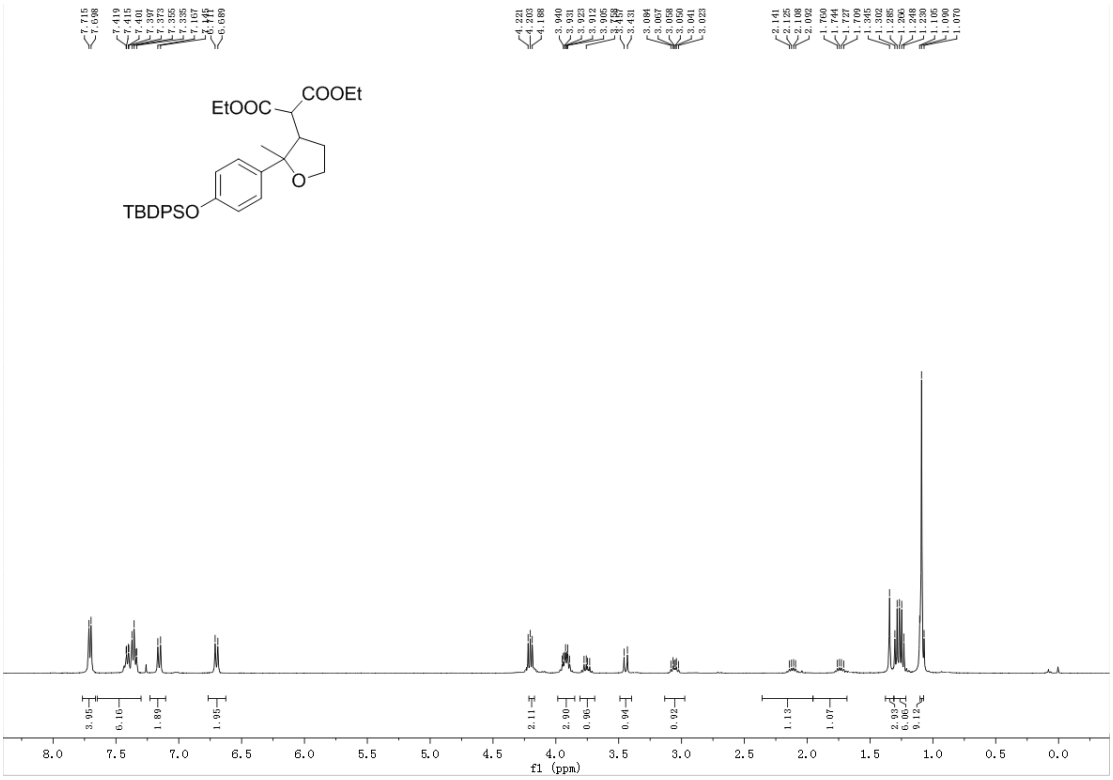
O
OMe
f1 (ppm)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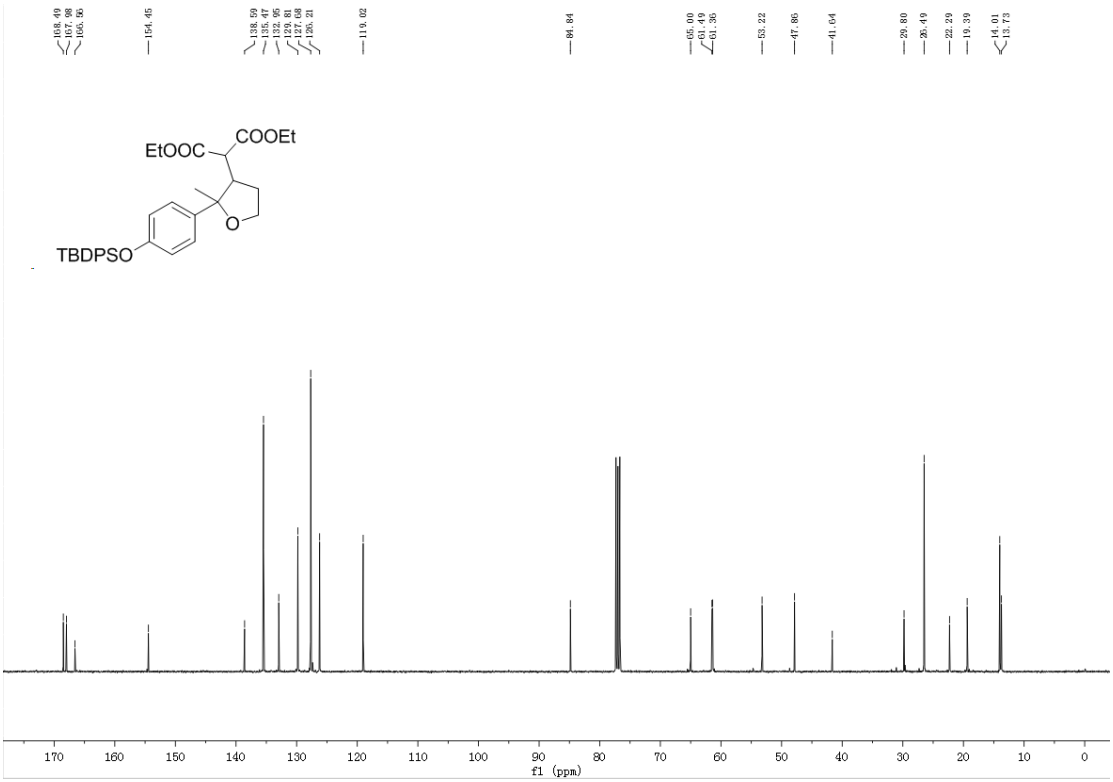
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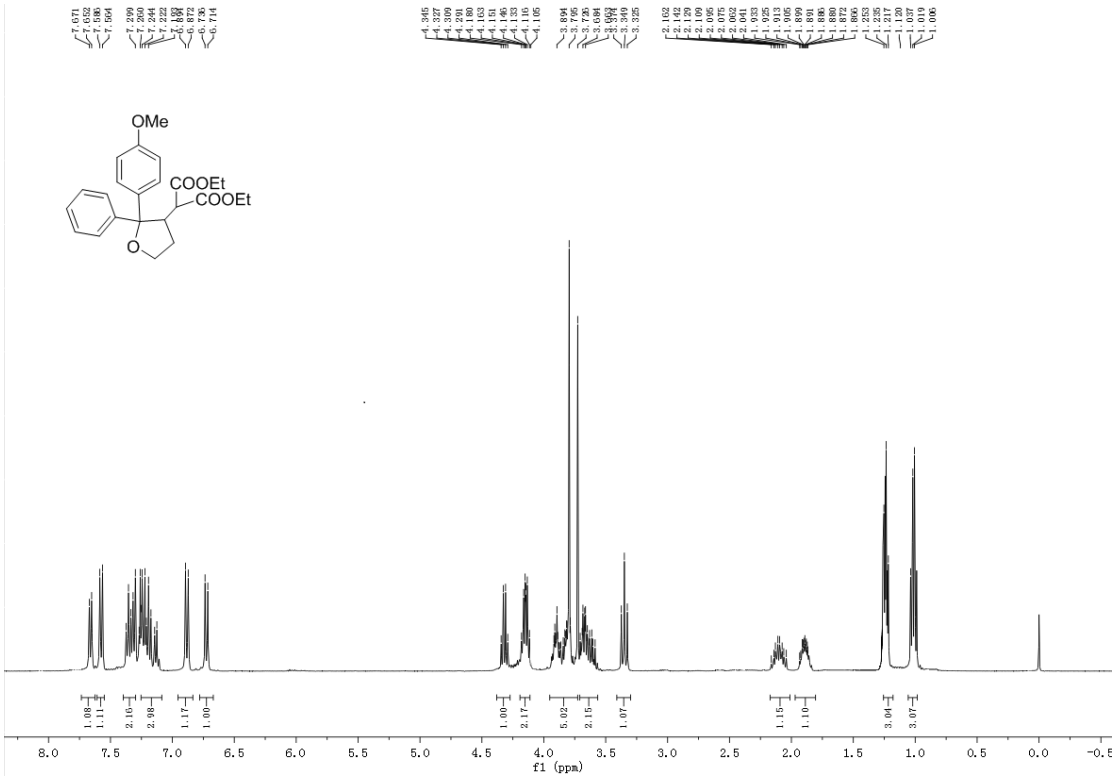
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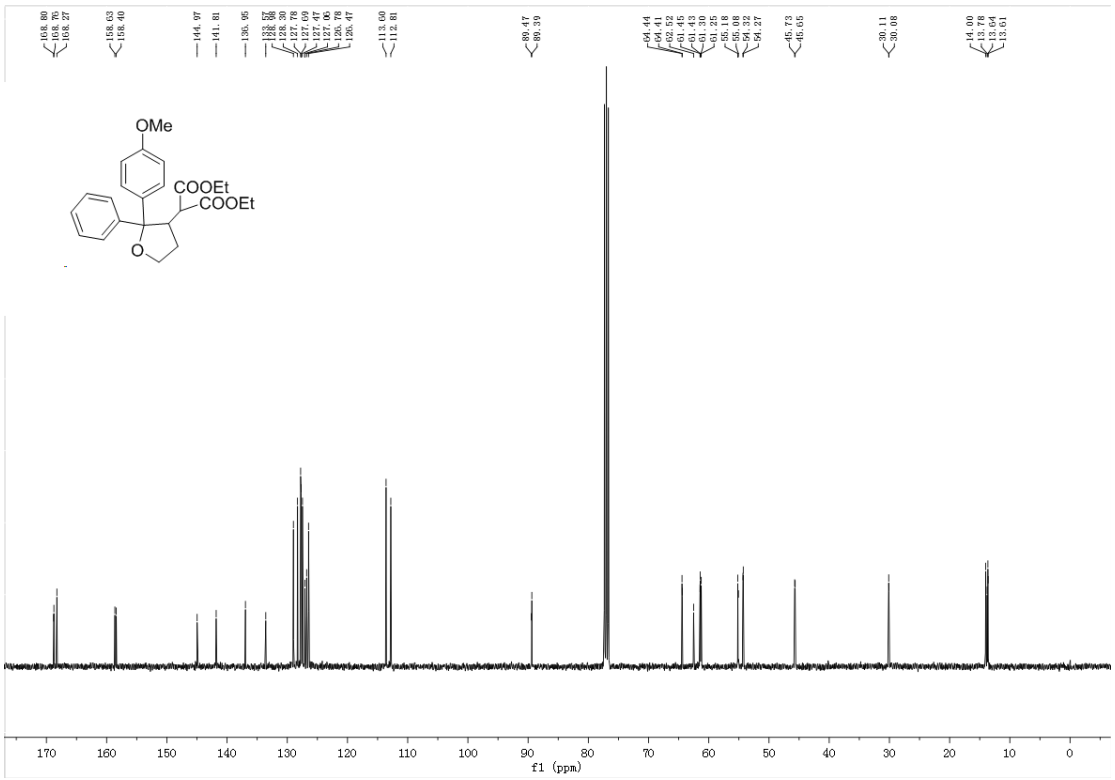
COOEt
O
f1 (ppm)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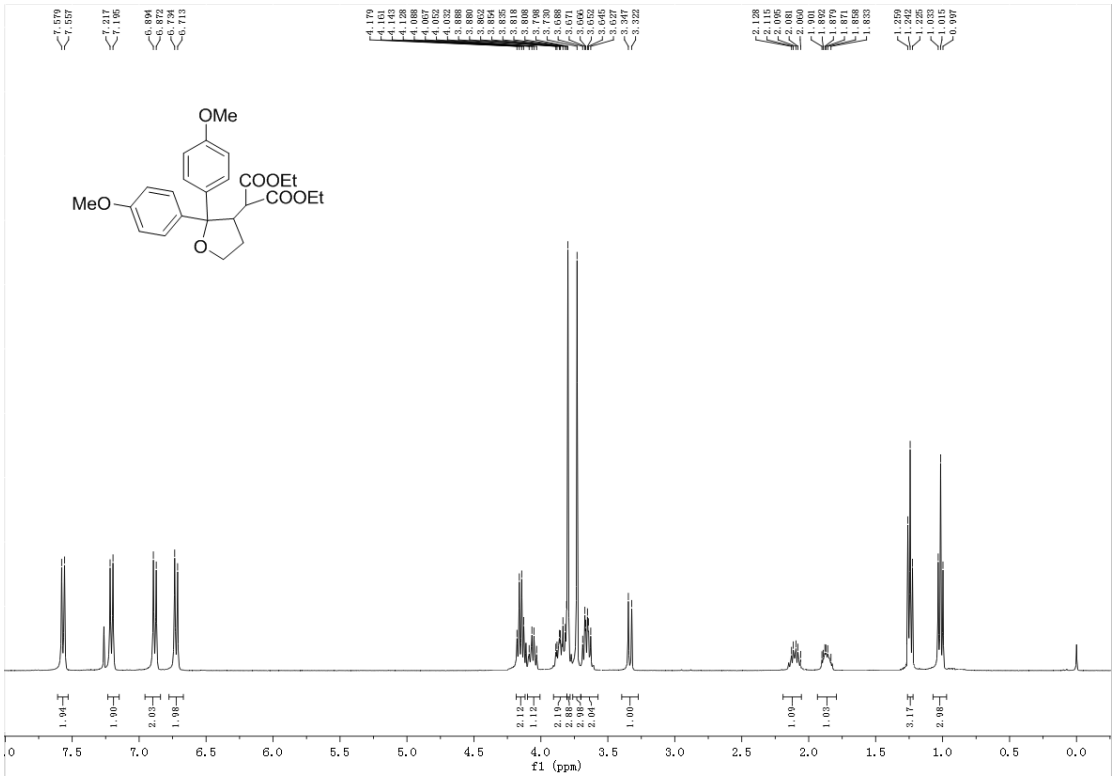
COOEt
O
f1 (ppm)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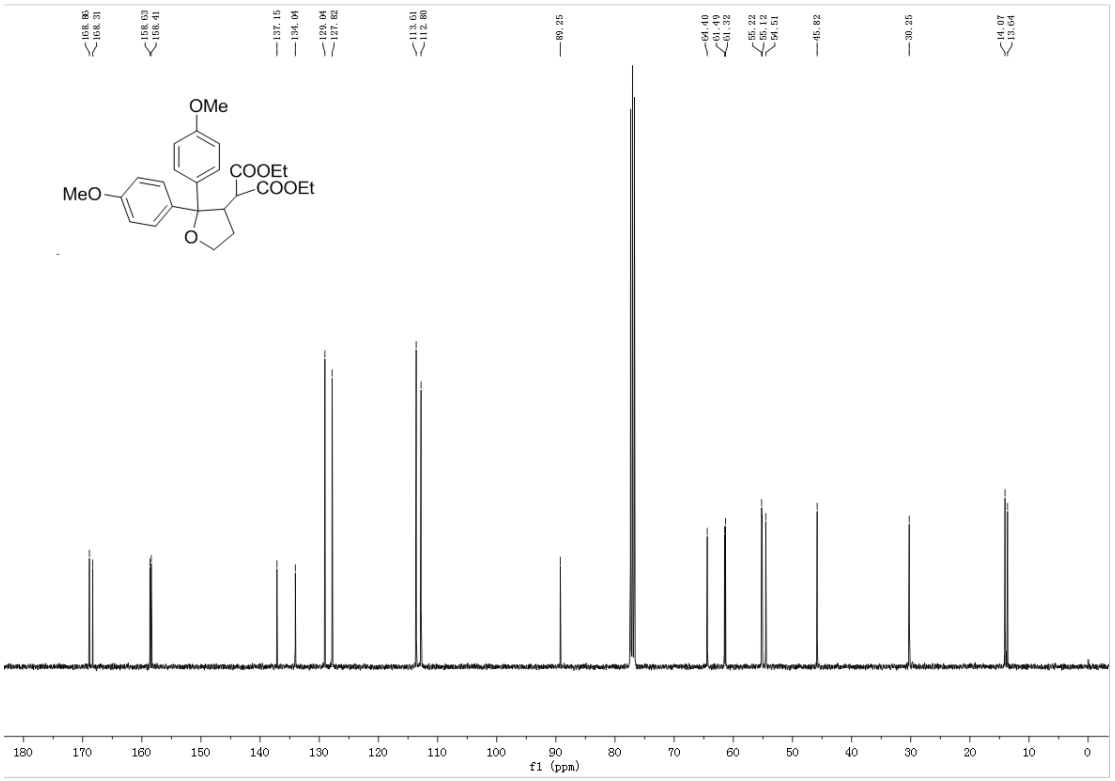
COOEt
MeO
O
f1 (ppm)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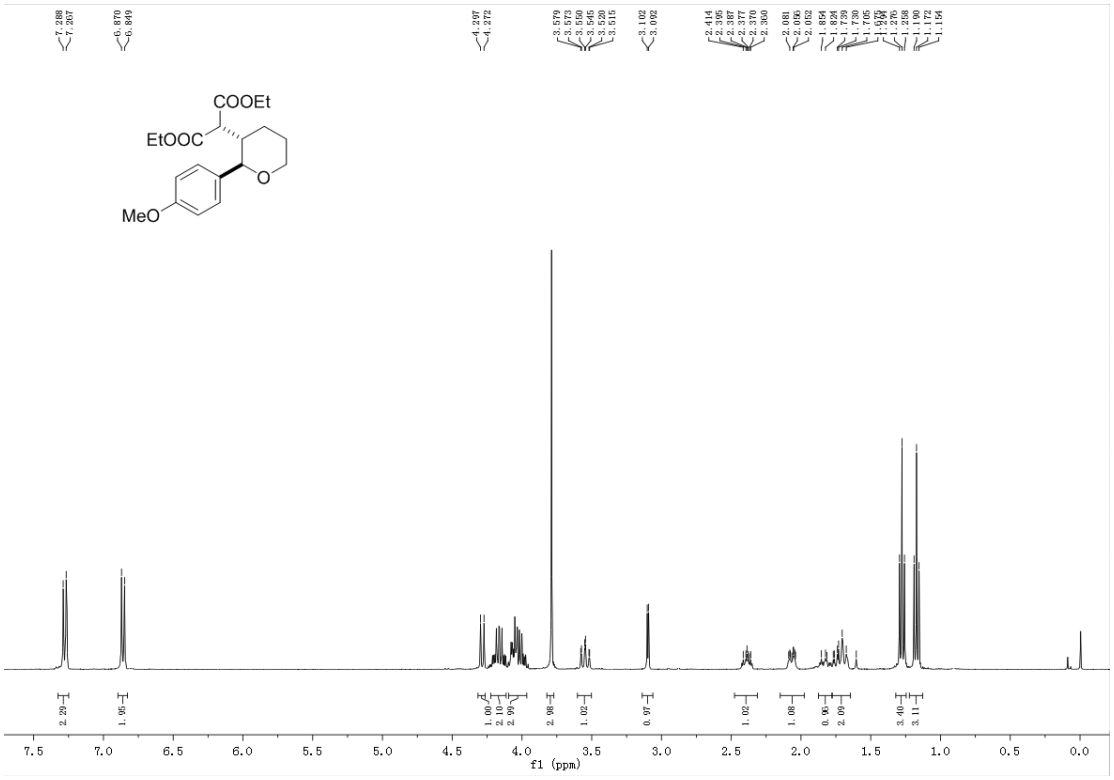
f1 (ppm)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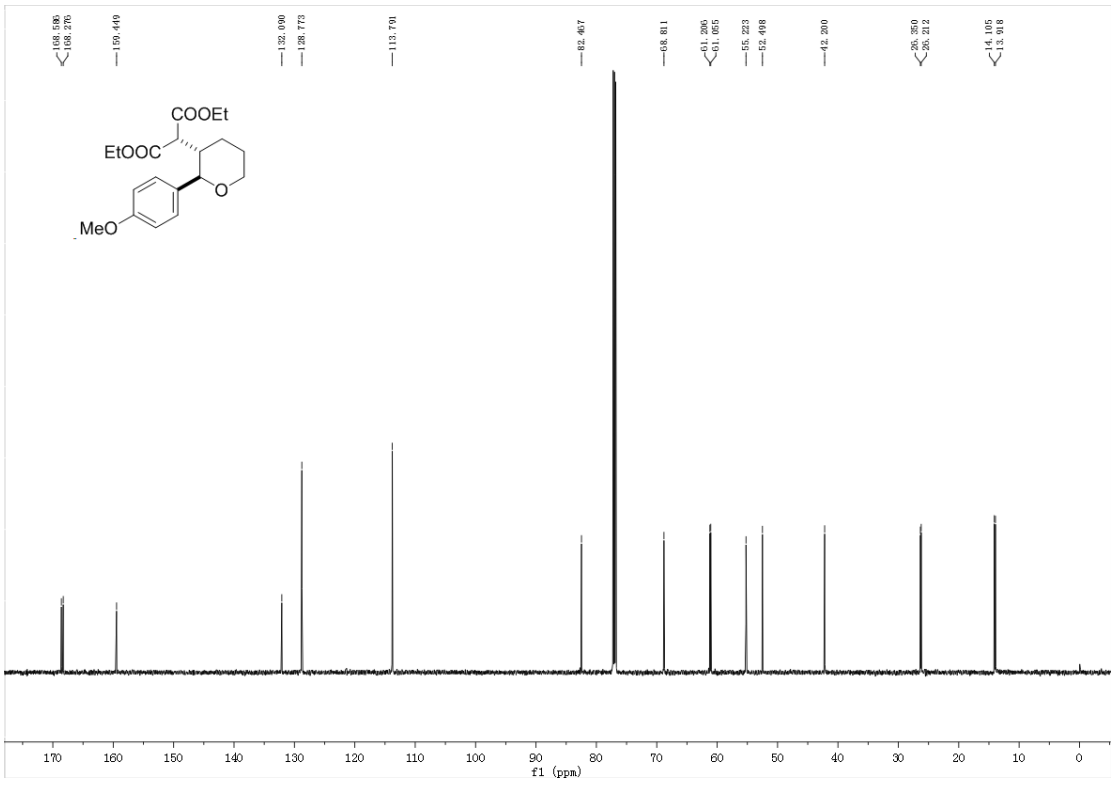
MeO
f1 (ppm)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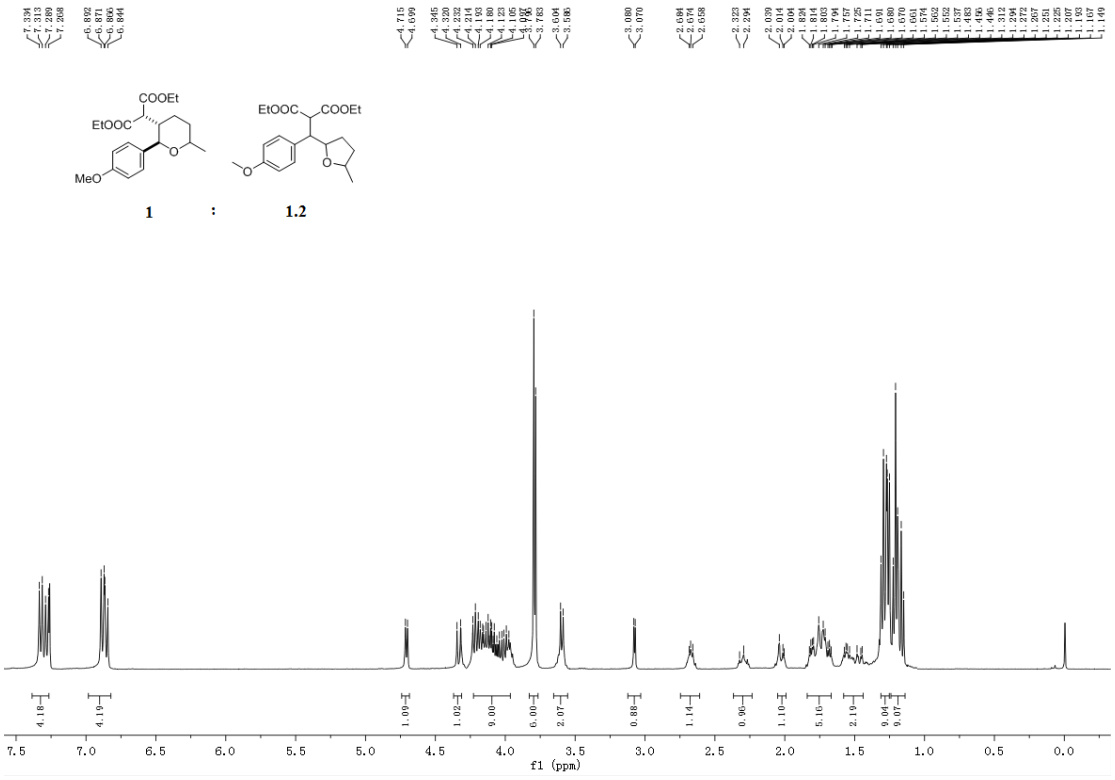
O
MeO
f1 (ppm)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MeO
EtOOC
COOEt
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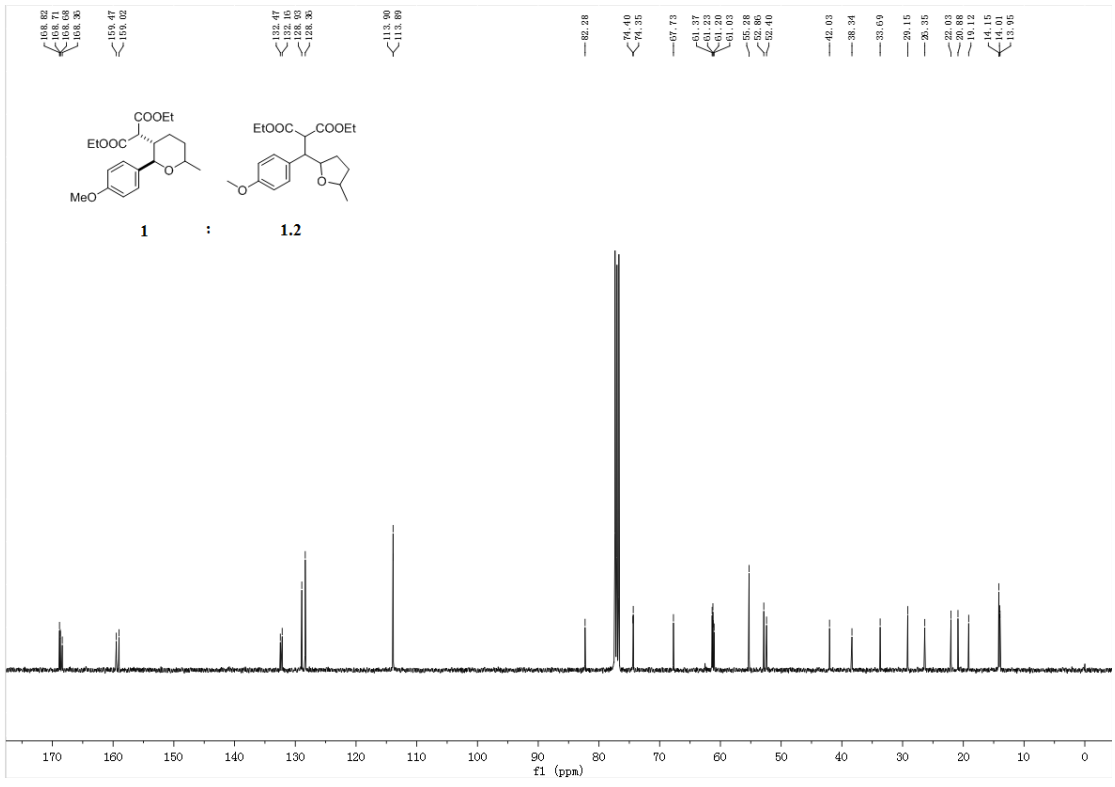
f1 (ppm)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MeO
EtOOC
COOEt
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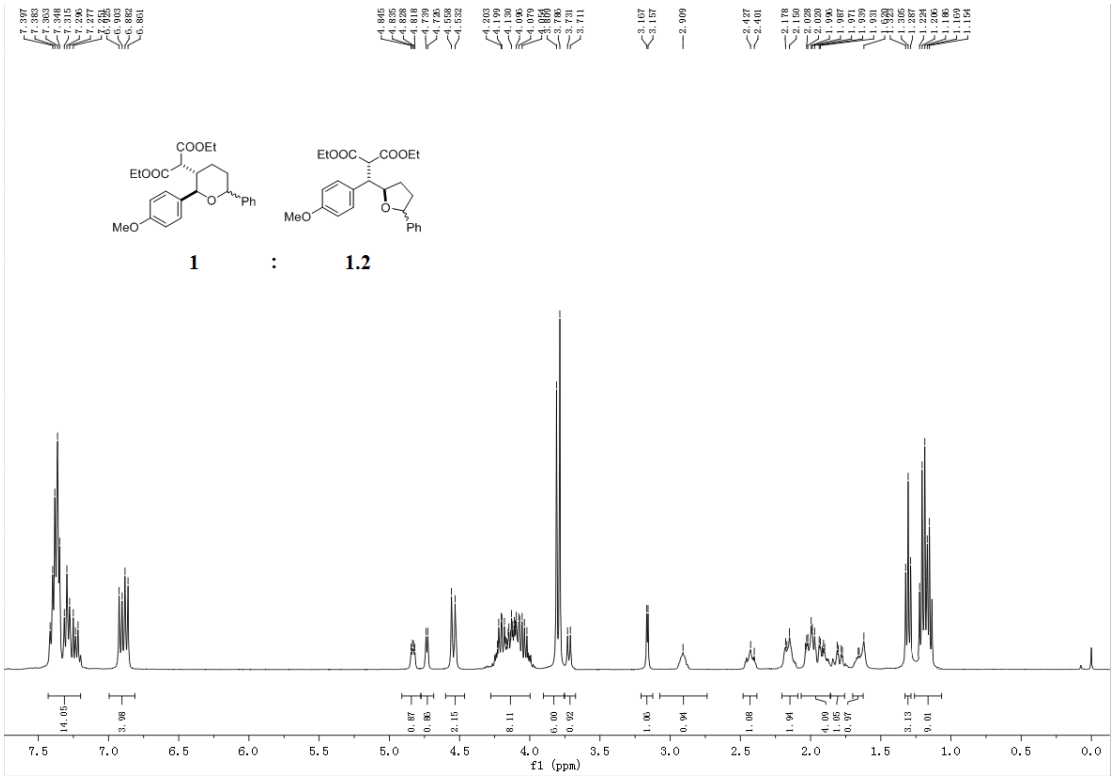
f1 (ppm)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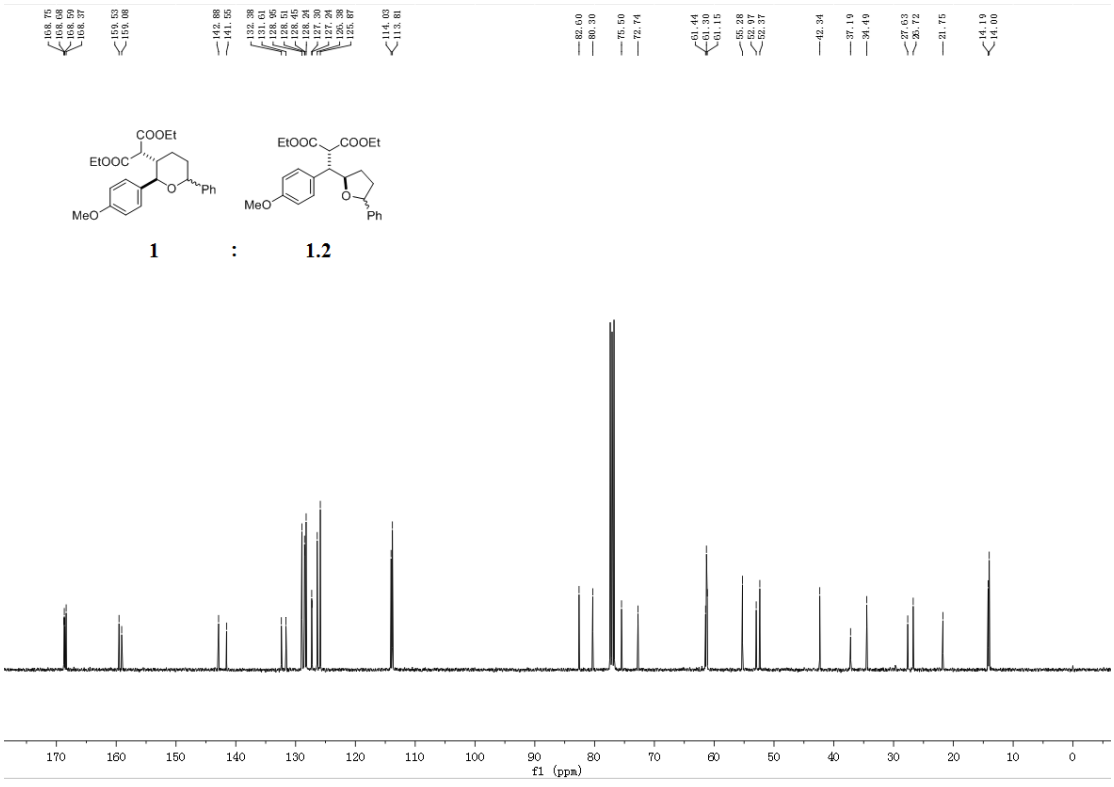
EtOOC
Ph
MeO
EtOOC
COOEt
MeO
Ph
1 : 1.2
f1 (ppm)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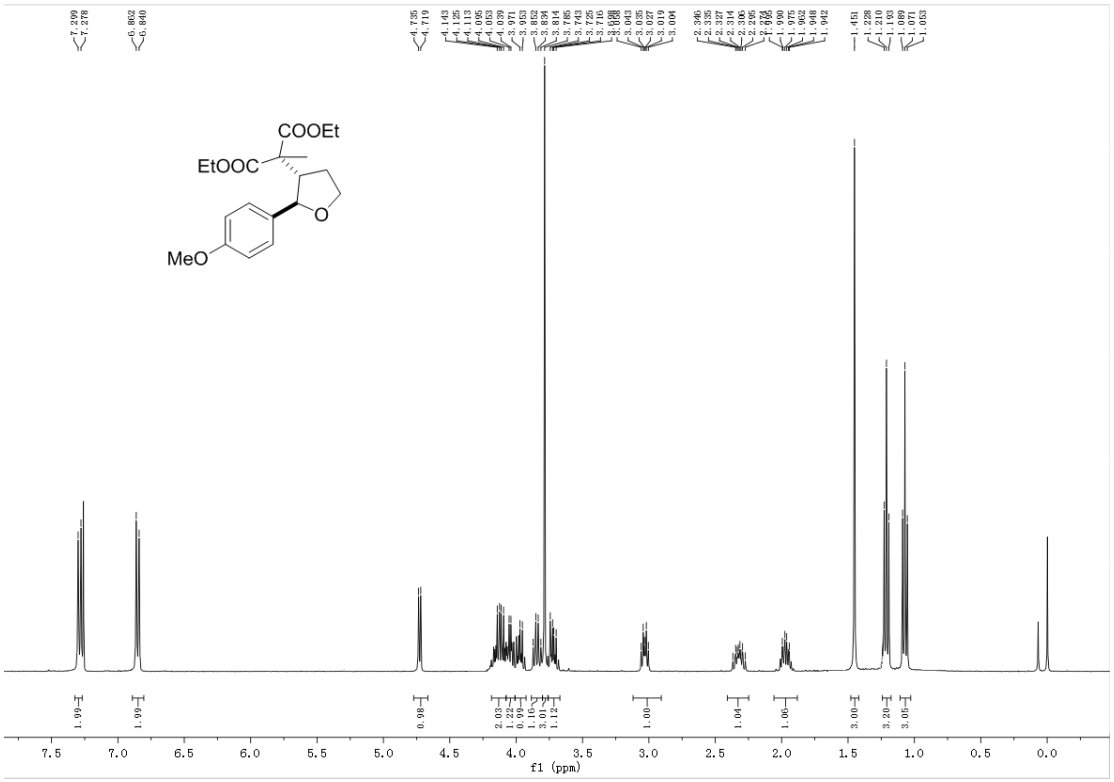
EtOOC
Ph
MeO
EtOOC
COOEt
MeO
Ph
1 : 1.2
f1 (ppm)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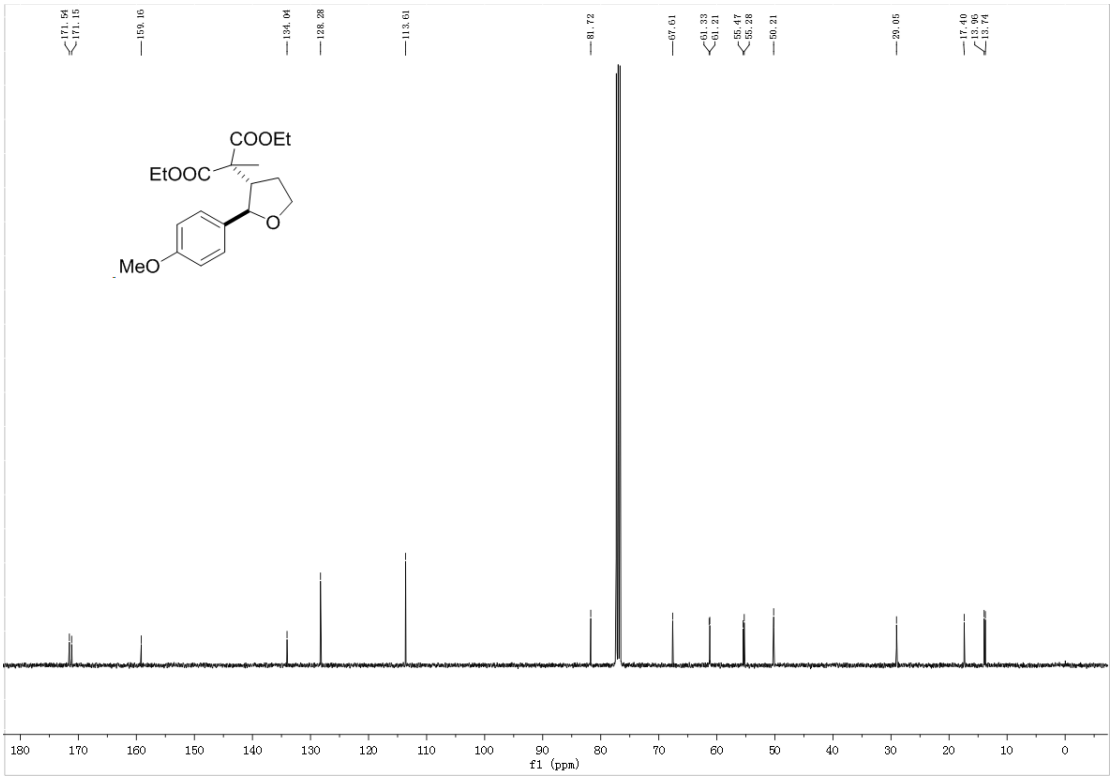
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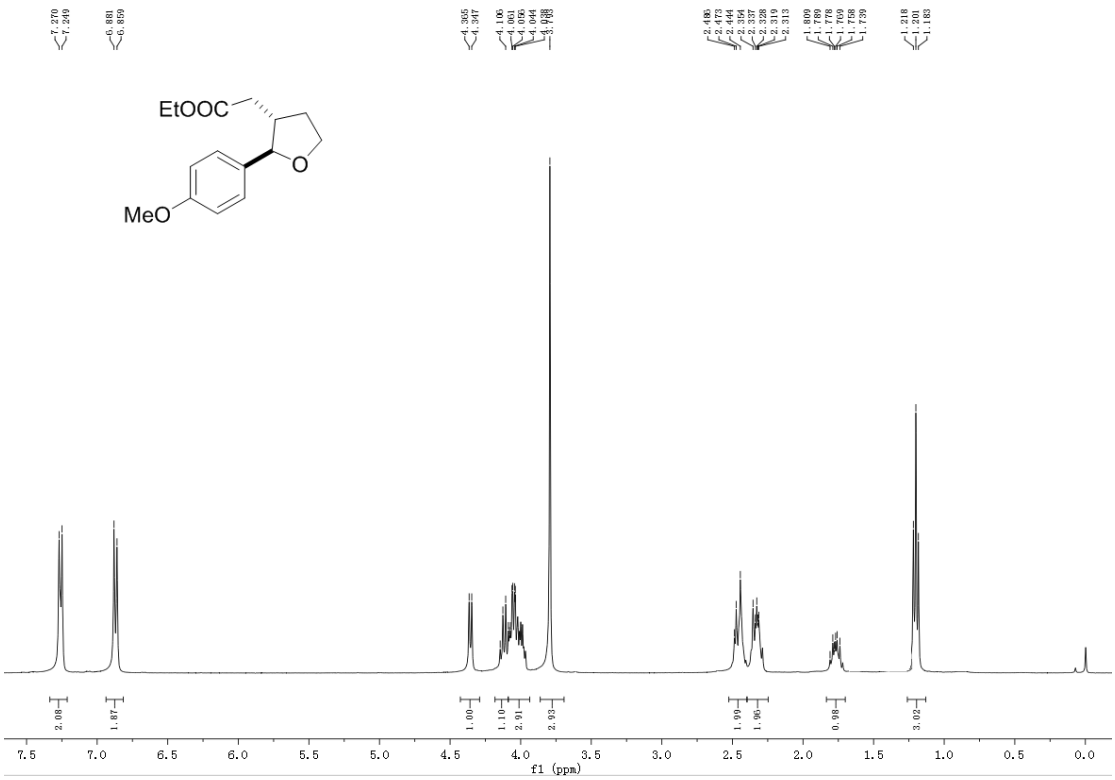
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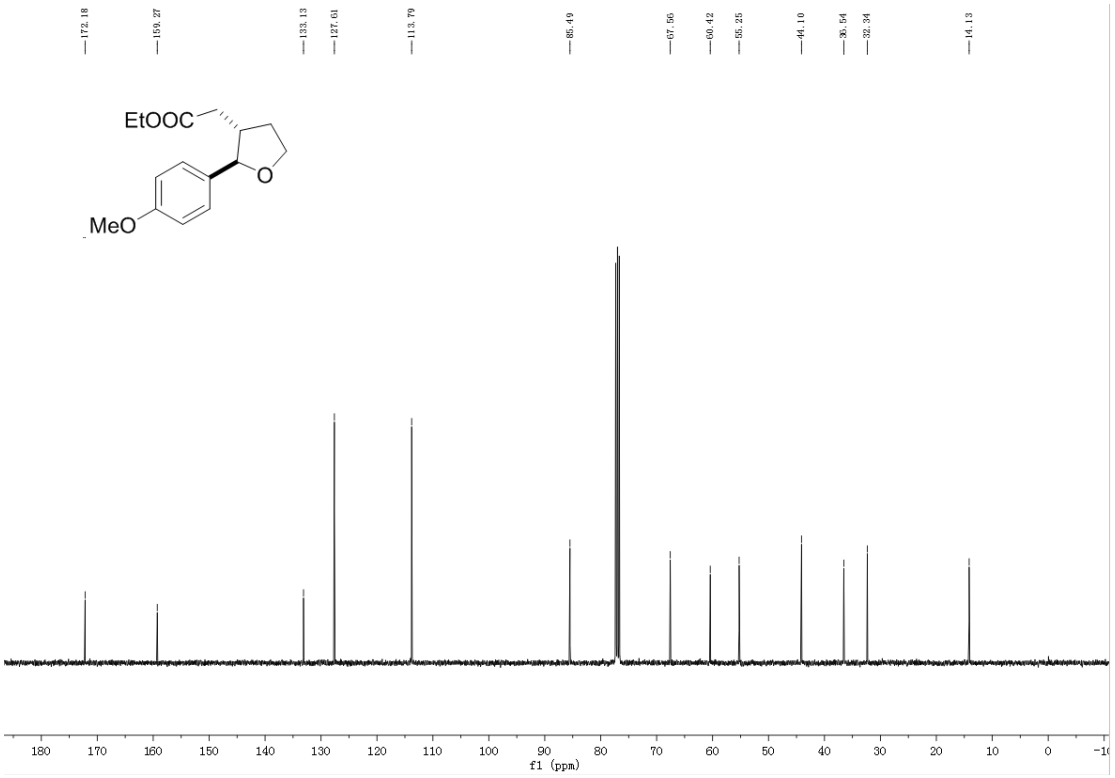
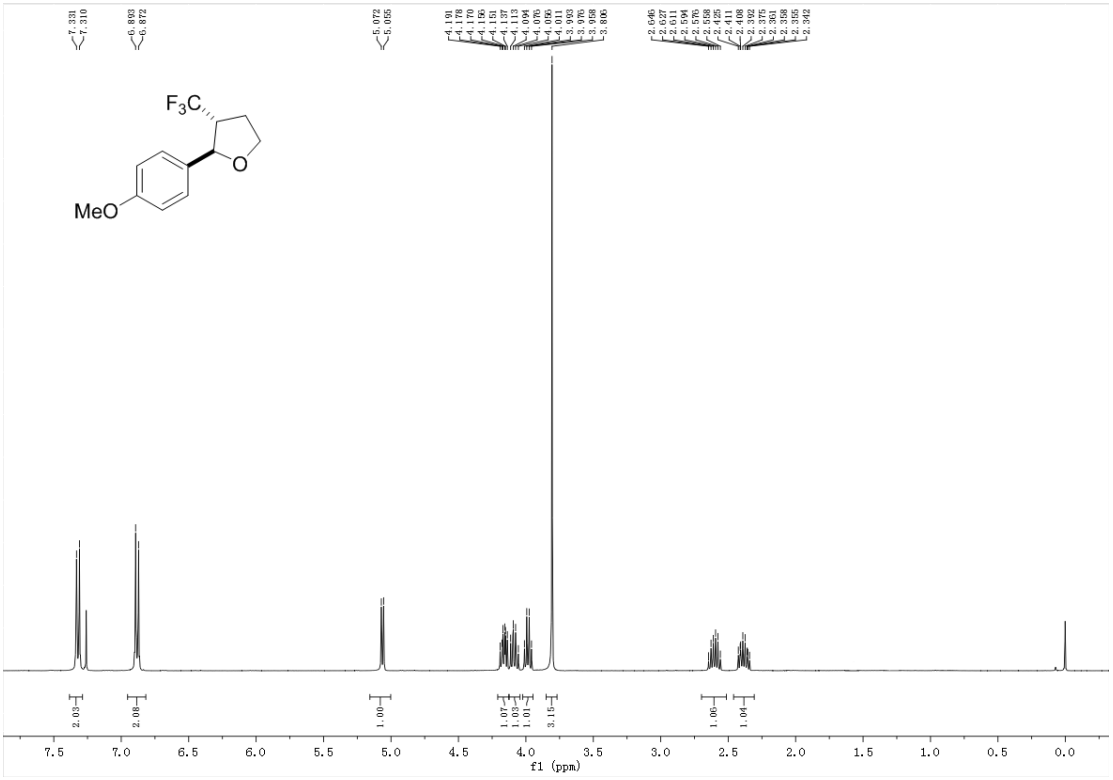
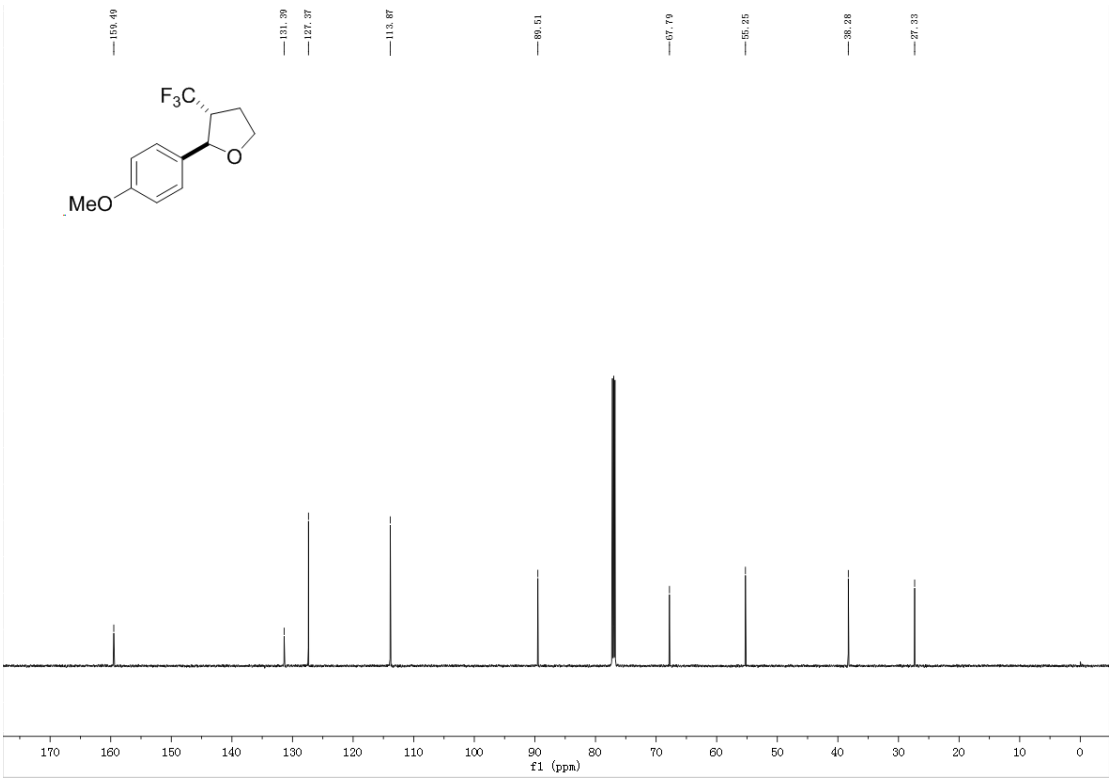
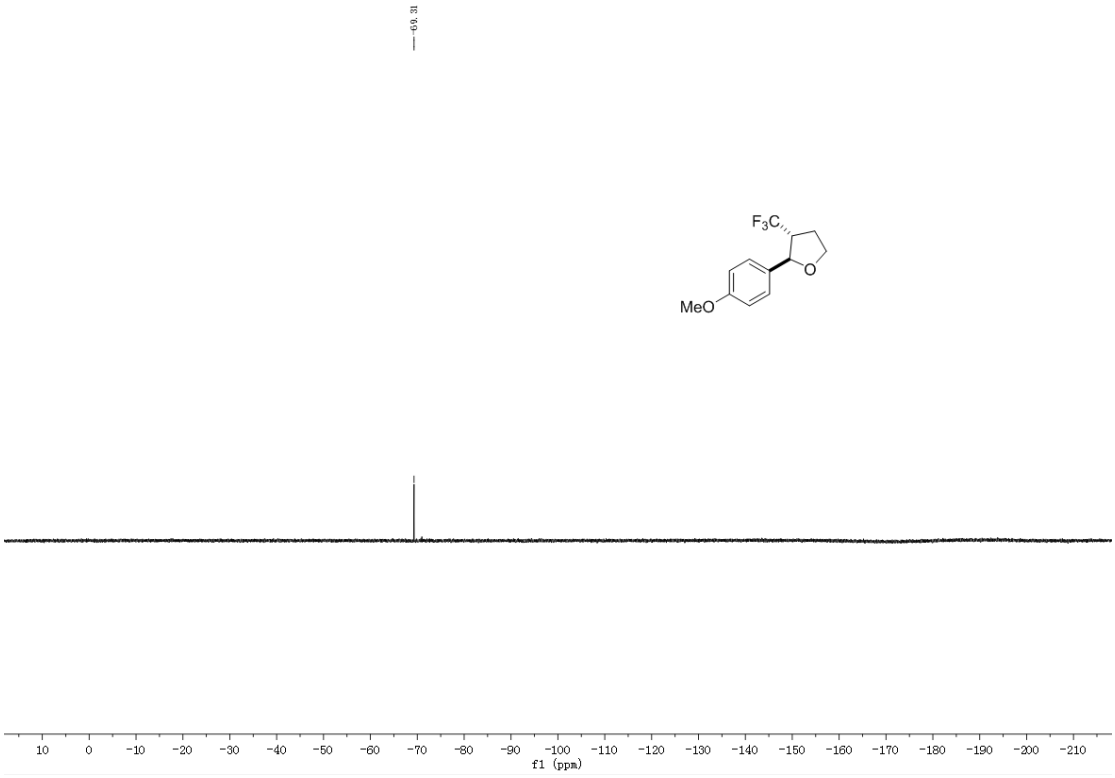
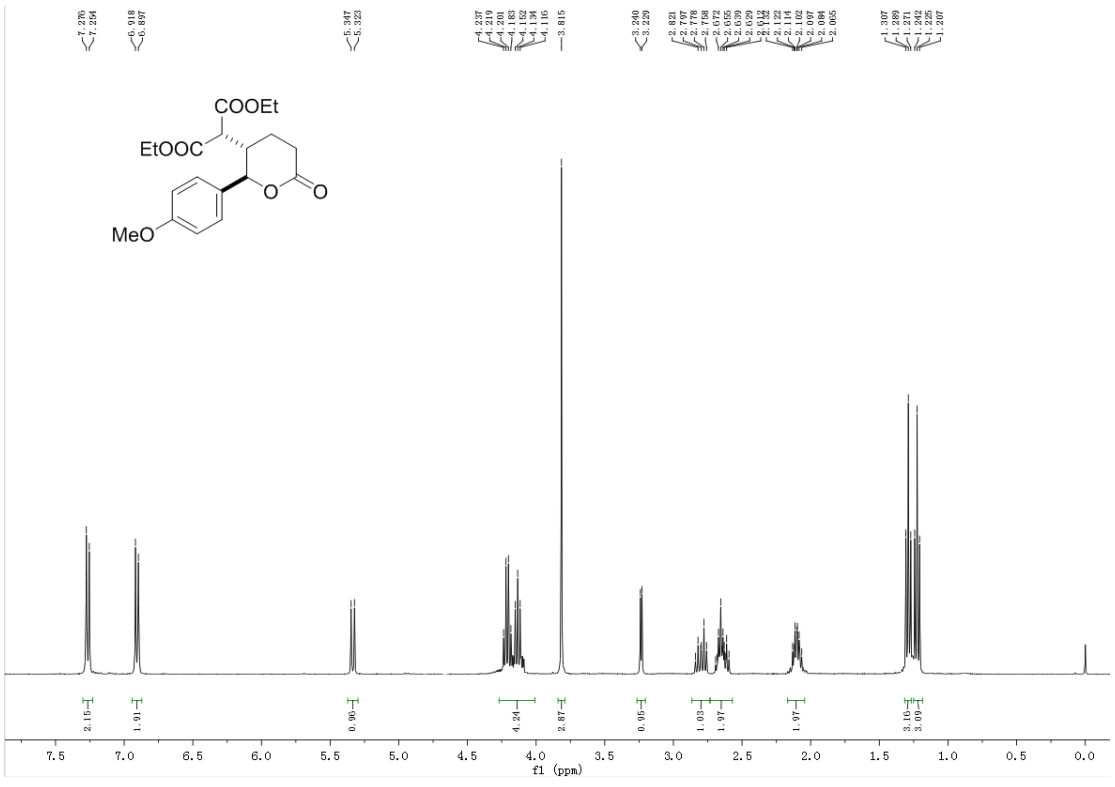
MeO
f1 (ppm)

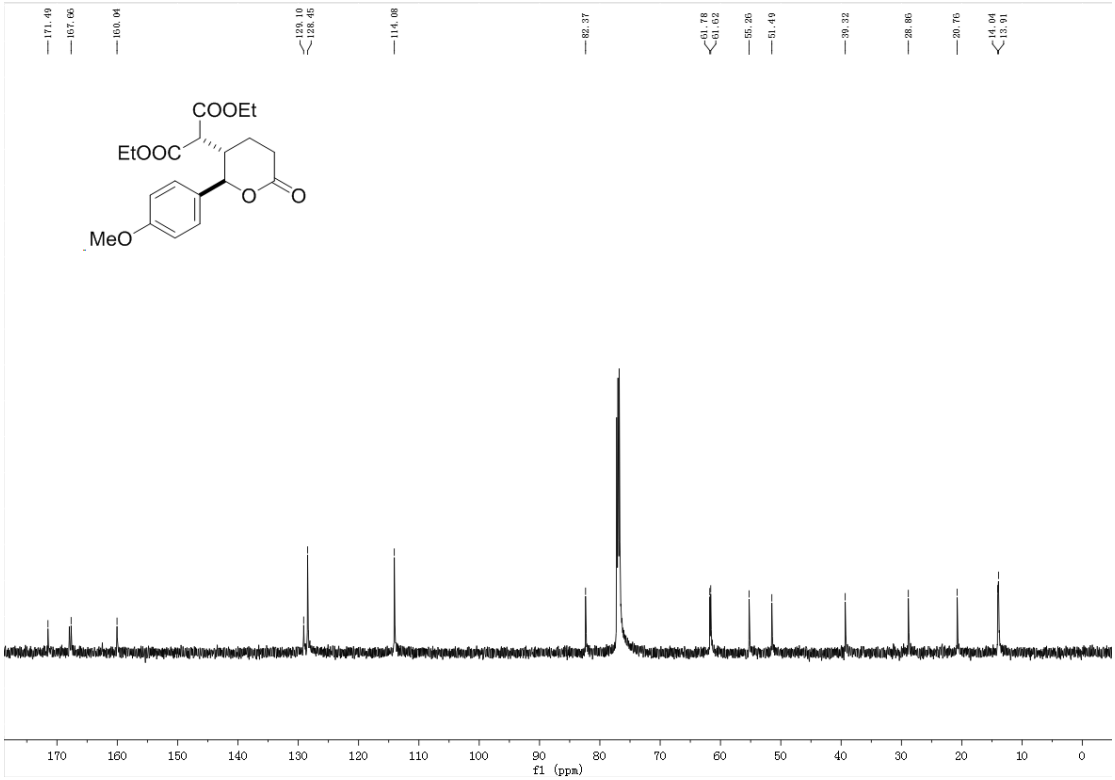

EtOOC
O
MeO
f1 (ppm)

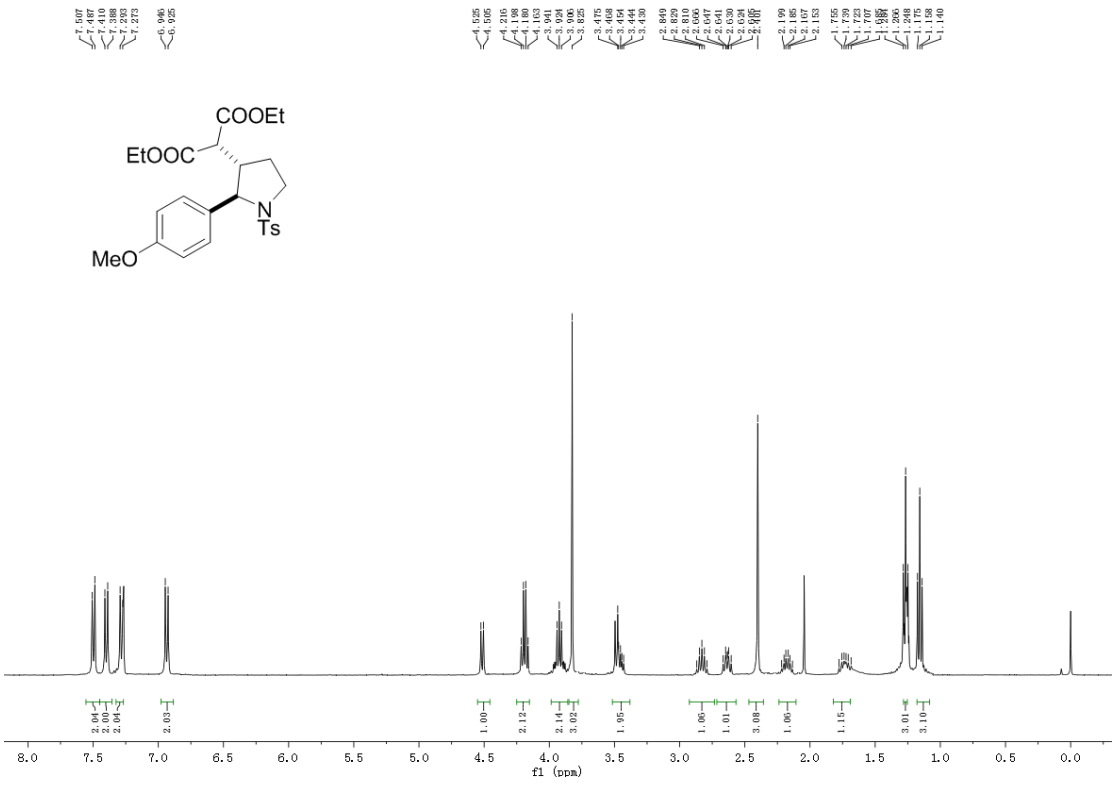

F3C
O
MeO
7.331
7.310
6.893
6.872
5.072
5.055
4.191
4.178
4.170
4.156
4.151
4.137
4.113
4.094
4.076
4.055
4.011
3.993
3.976
3.958
3.806
2.646
2.627
2.611
2.594
2.576
2.558
2.425
2.411
2.408
2.392
2.375
2.361
2.358
2.355
2.342
2.03
2.08
1.00
1.07
1.03
1.01
3.15
1.06
1.04
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
0.5
0.0
f1 (ppm)

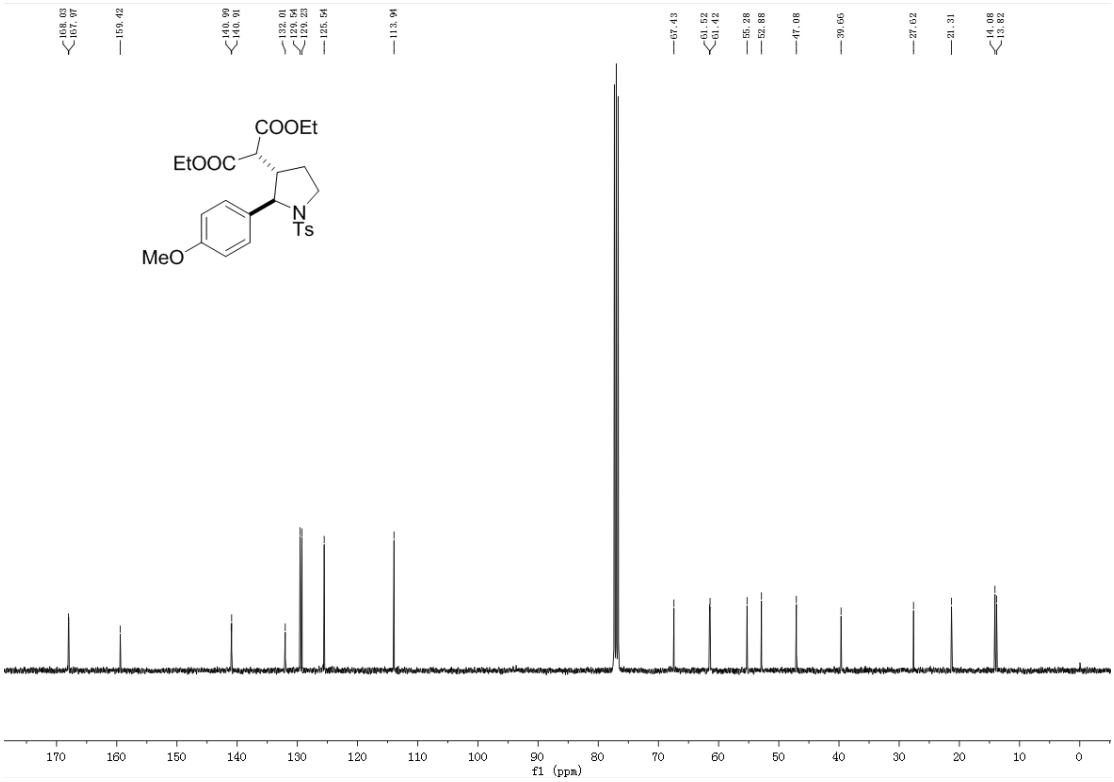

F3C
O
MeO
159.49
131.39
127.37
113.87
89.51
67.79
55.25
38.28
27.33
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
f1 (ppm)


-69.31
F3C
O
MeO
10 0 -10 -20 -30 -40 -50 -60 -70 -80 -90 -100 -110 -120 -130 -140 -150 -160 -170 -180 -190 -200 -210
f1 (ppm)


7.276
7.294
6.918
6.897
5.347
5.323
4.237
4.219
4.201
4.183
4.152
4.134
4.116
3.815
3.240
3.229
2.821
2.797
2.778
2.758
2.672
2.655
2.639
2.529
2.122
2.114
2.102
2.097
2.084
2.065
1.307
1.289
1.271
1.242
1.225
1.207
COOEt
EtOOC
O
O
MeO
2.15
1.91
0.96
4.24
2.87
0.95
1.03
1.97
1.97
3.16
3.09
7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 0.5 0.0
f1 (ppm)


COOEt
EtOOC
O
O
MeO
f1 (ppm)


COOEt
EtOOC
N
Ts
MeO
f1 (ppm)


168.03
167.97
159.42
140.99
140.91
130.01
129.54
129.23
125.54
113.94
67.43
61.52
61.42
55.28
52.88
47.08
39.66
27.62
21.31
14.08
13.82
COOEt
EtOOC
N
Ts
MeO
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
f1 (ppm)