

**Iodine-catalysed sp^3 C-H sulfonylation to form β -dicarbonyl sulfones
with sodium sulfinates**

(Supplementary Information)

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Table of contents

General information.....	S2
General procedure for the synthesis of β -dicarbonyl sulfones.....	S2
Characterization data of products.....	S2
Reference.....	S8
NMR Spectra of products.....	S9

General information

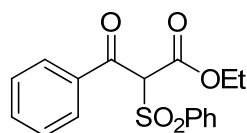
^1H NMR spectra were recorded at 600 MHz and ^{13}C NMR spectra were measured at 150 MHz using Bruker AVANCE III 600 MHz NMR spectrometers with CDCl_3 as the solvent. The chemical shifts (δ) were measured in ppm and referenced to the deuterated chloroform (^1H : $\delta = 7.26$ ppm, ^{13}C : $\delta = 77.00$ ppm). The multiplicities of signals were described using the following abbreviations: s = singlet, d = doublet, t = triplet, q = quartet, m = multiplet, dd = doublet of doublets, tt = triplet of triplets. High-resolution mass spectra (HRMS) were performed on a micrOTOF-Q II instrument with an ESI source. Melting points were measured with a RD-II type melting point apparatus. The known compounds were identified by the comparison of their NMR spectra with reported data in literatures; the new compounds were characterized by NMR, HRMS and melting point for solid samples. Unless otherwise noted, reagents obtained from commercial sources were directly used without further purification; all solvents were obtained from commercial sources and were purified according to standard procedures. Petroleum ether (PE), where used, has the boiling point range 60-90 °C. Column chromatography was performed on silica gel (200-300 mesh). Non-commercial available sodium sulfinates were prepared according to the reported procedure.^{S1}

General procedure for the synthesis of β -dicarbonyl sulfones

A 10 mL oven-dried reaction vessel was charged with ethyl benzoylacetate (**1a**, 96 mg, 0.5 mmol), sodium benzenesulfinate dihydrate (**2a**, 120 mg, 0.6 mmol), and iodine (12.7 mg, 0.05 mmol) in acetonitrile (2.0 mL) under air. TBHP (70% in water) (96 mg, 0.75 mmol) was added slowly to the sealed reaction vessel by syringe, and the resulting solution was stirred at 65 °C for 1 h. After the reaction was complete by TLC analysis, sat. $\text{Na}_2\text{S}_2\text{O}_3$ solution (10 mL) was added to quench the reaction, and the mixture was extracted by ethyl acetate (3×10 mL). The organic layer was separated and dried over anhydrous Na_2SO_4 . After the removal of the solvent under vacuo, the residue was purified by flash column chromatography with PE/EtOAc (9 : 1) to give ethyl 3-oxo-3-phenyl-2-(phenylsulfonyl)propanoate (**3a**) as pale yellow solid; yield: 157 mg (94%); m.p. 70-72 °C.

Characterization data of products

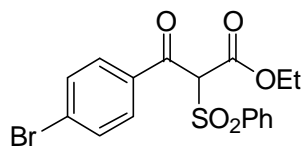
Ethyl 3-oxo-3-phenyl-2-(phenylsulfonyl)propanoate (**3a**)



Yield: 94%; white solid; m.p. 70-72 °C; TLC, $R_f = 0.18$ (PE:EtOAc = 4:1); ^1H NMR (CDCl_3 , 600 MHz): δ 7.97 (t, 4H, $J = 8.4$ Hz), 7.68 (t, 1H, $J = 7.8$ Hz), 7.65 (t, 1H, $J = 7.8$ Hz), 7.55 (t, 2H, $J = 7.8$ Hz), 7.51 (t, 2H, $J = 7.8$ Hz), 6.01 (s, 1H), 4.20 (m, 2H), 1.18 (t, 3H, $J = 7.2$ Hz); ^{13}C NMR (CDCl_3 , 150 MHz): δ 185.8, 161.5, 137.1, 135.5, 134.6, 130.5, 129.3, 128.9, 128.6, 75.4, 63.1, 13.8; HRMS (ESI) m/z calcd. for

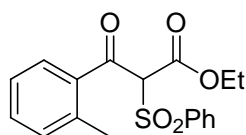
C₁₇H₁₇O₅S [M+H]⁺: 333.0796, found: 333.0791.

Ethyl 3-(4-bromophenyl)-3-oxo-2-(phenylsulfonyl)propanoate (3b)



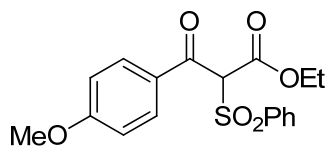
Yield: 71%; pale yellow solid; m.p. 118-120 °C; TLC, *R_f* = 0.30 (PE:EtOAc = 2:1); ¹H NMR (CDCl₃, 600 MHz): δ 7.95 (d, 2H, *J* = 7.8 Hz), 7.86 (d, 2H, *J* = 8.4 Hz), 7.70 (t, 1H, *J* = 7.8 Hz), 7.66 (d, 2H, *J* = 8.4 Hz), 7.56 (t, 2H, *J* = 7.8 Hz), 5.91 (s, 1H), 4.25-4.15 (m, 2H), 1.19 (t, 3H, *J* = 7.2 Hz); ¹³C NMR (CDCl₃, 150 MHz): δ 185.0, 161.3, 137.1, 134.7, 134.3, 132.3, 130.8, 130.4, 129.2, 128.7, 75.6, 63.1, 13.8; HRMS (ESI) *m/z* calcd. for C₁₇H₁₅BrNaO₅S [M+Na]⁺: 434.9695, found: 434.9695.

Ethyl 3-oxo-2-(phenylsulfonyl)-3-(o-tolyl)propanoate (3c)



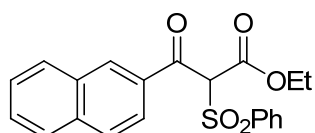
Yield: 77%; white solid; m.p. 84-86 °C; TLC, *R_f* = 0.10 (PE:EtOAc = 4:1); ¹H NMR (CDCl₃, 600 MHz): δ 7.97 (d, 2H, *J* = 7.2 Hz), 7.72 (d, 1H, *J* = 7.8 Hz), 7.68 (t, 1H, *J* = 7.2 Hz), 7.55 (t, 2H, *J* = 7.8 Hz), 7.44 (t, 1H, *J* = 7.8 Hz), 7.30-7.27 (m, 2H), 5.91 (s, 1H), 4.20 (q, 2H, *J* = 7.2 Hz), 2.44 (s, 3H), 1.19 (t, 3H, *J* = 7.2 Hz); ¹³C NMR (CDCl₃, 150 MHz): δ 188.3, 161.6, 139.8, 137.6, 136.1, 134.5, 132.7, 132.2, 130.3, 129.3, 128.7, 125.9, 77.5, 62.9, 20.9, 13.8; HRMS (ESI) *m/z* calcd. for C₁₈H₁₈NaO₅S [M+Na]⁺: 369.0767, found: 369.0767.

Ethyl 3-(4-methoxyphenyl)-3-oxo-2-(phenylsulfonyl)propanoate (3d)



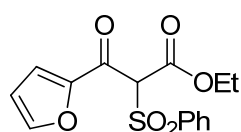
Yield: 87%; white solid; m.p. 116-119 °C; TLC, *R_f* = 0.30 (PE:EtOAc = 4:1); ¹H NMR (CDCl₃, 600 MHz): δ 7.98-7.94 (m, 4H), 7.66 (t, 1H, *J* = 7.8 Hz), 7.53 (t, 2H, *J* = 7.8 Hz), 6.96 (d, 2H, *J* = 9.0 Hz), 5.97 (s, 1H), 4.24-4.14 (m, 2H), 3.88 (s, 3H), 1.75 (t, 3H, *J* = 7.2 Hz); ¹³C NMR (CDCl₃, 150 MHz): δ 183.9, 164.8, 161.7, 137.2, 134.4, 131.9, 130.5, 128.54, 128.48, 114.2, 75.3, 62.9, 55.6, 13.7; HRMS (ESI) *m/z* calcd. for C₁₈H₁₉O₆S [M+H]⁺: 363.0897, found: 363.0896.

Ethyl 3-(naphthalen-2-yl)-3-oxo-2-(phenylsulfonyl)propanoate (3e)



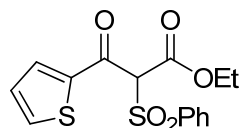
Yield: 93%; white solid; m.p. 94-96 °C; TLC, $R_f = 0.22$ (PE:EtOAc = 7:3); ^1H NMR (CDCl_3 , 600 MHz): δ 8.53 (s, 1H), 8.05-7.95 (m, 4H), 7.92-7.87 (m, 2H), 7.68-7.63 (m, 2H), 7.59 (t, 1H, $J = 7.8$ Hz), 7.54 (t, 2H, $J = 7.8$ Hz), 6.20 (s, 1H), 4.25-4.18 (m, 2H), 1.18 (t, 3H, $J = 7.2$ Hz); ^{13}C NMR (CDCl_3 , 150 MHz): δ 185.7, 161.6, 137.2, 136.1, 134.5, 132.9, 132.3, 132.0, 130.5, 130.0, 129.5, 128.9, 128.6, 127.8, 127.2, 124.0, 75.6, 63.0, 13.8; HRMS (ESI) m/z calcd. for $\text{C}_{21}\text{H}_{18}\text{NaO}_5\text{S}$ $[\text{M}+\text{Na}]^+$: 405.0767, found: 405.0765.

Ethyl 3-(furan-2-yl)-3-oxo-2-(phenylsulfonyl)propanoate (3f)



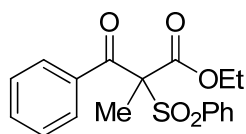
Yield: 71%; pale yellow solid; m.p. 91-93 °C; TLC, $R_f = 0.12$ (PE:EtOAc = 4:1); ^1H NMR (CDCl_3 , 600 MHz): δ 7.96 (d, 2H, $J = 7.2$ Hz), 7.70-7.66 (m, 2H), 7.55 (t, 2H, $J = 7.8$ Hz), 7.43 (d, 1H, $J = 3.6$ Hz), 6.63 (dd, 1H, $J = 3.6, 1.2$ Hz), 5.84 (s, 1H), 4.24-4.17 (m, 2H), 1.21 (t, 3H, $J = 7.2$ Hz); ^{13}C NMR (CDCl_3 , 150 MHz): δ 173.5, 161.1, 151.3, 148.3, 137.2, 134.6, 130.4, 128.7, 120.8, 113.4, 75.3, 63.0, 13.8; HRMS (ESI) m/z calcd. For $\text{C}_{15}\text{H}_{15}\text{O}_6\text{S}$ $[\text{M}+\text{H}]^+$: 323.0584, found: 323.0677.

Ethyl 3-oxo-2-(phenylsulfonyl)-3-(thiophen-2-yl)propanoate (3g)



Yield: 82%; white solid; m.p. 93-96 °C; TLC, $R_f = 0.12$ (PE:EtOAc = 9:1); ^1H NMR (CDCl_3 , 600 MHz): δ 7.96 (d, 2H, $J = 7.2$ Hz), 7.91 (dd, 1H, $J = 3.6, 1.2$ Hz), 7.80 (dd, 1H, $J = 4.8, 1.2$ Hz), 7.69 (t, 1H, $J = 7.8$ Hz), 7.55 (t, 2H, $J = 7.8$ Hz), 7.20 (dd, 1H, $J = 4.8, 4.2$ Hz), 5.77 (s, 1H), 4.25-4.17 (m, 2H), 1.21 (t, 3H, $J = 7.2$ Hz); ^{13}C NMR (CDCl_3 , 150 MHz): δ 177.7, 161.2, 142.6, 137.0, 136.8, 135.3, 134.6, 130.5, 128.8, 128.7, 76.7, 63.1, 13.8; HRMS (ESI) m/z calcd. for $\text{C}_{15}\text{H}_{15}\text{O}_5\text{S}_2$ $[\text{M}+\text{H}]^+$: 339.0355, found: 339.0357.

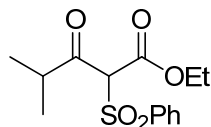
Ethyl 2-methyl-3-oxo-3-phenyl-2-(phenylsulfonyl)propanoate (3h)



Yield: 9%; pale yellow oil; TLC, $R_f = 0.15$ (PE:EtOAc = 9:1); ^1H NMR (CDCl_3 , 600 MHz): δ 8.04 (d, 2H, $J = 7.2$ Hz), 7.83 (d, 2H, $J = 7.2$ Hz), 7.65 (t, 1H, $J = 7.2$ Hz),

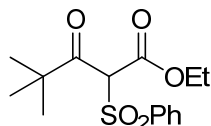
7.55-7.51 (m, 3H), 7.41 (t, 2H, $J = 7.8$ Hz), 4.21-4.12 (m, 2H), 2.01 (s, 3H), 1.03 (t, 3H, $J = 7.2$ Hz); ^{13}C NMR (CDCl_3 , 150 MHz): δ 190.3, 166.5, 137.1, 134.2, 133.4, 131.5, 129.4, 129.0, 128.5, 128.4, 81.6, 62.9, 19.5, 13.4; HRMS (ESI) m/z calcd. for $\text{C}_{18}\text{H}_{19}\text{O}_5\text{S}$ $[\text{M}+\text{H}]^+$: 347.0948, found: 347.0948.

Ethyl 4-methyl-3-oxo-2-(phenylsulfonyl)pentanoate (3i)



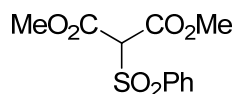
Yield: 66%; colorless oil; TLC, $R_f = 0.18$ (PE:EtOAc = 5:1); ^1H NMR (CDCl_3 , 600 MHz): δ 7.93 (d, 2H, $J = 7.2$ Hz), 7.68 (t, 1H, $J = 7.2$ Hz), 7.56 (t, 2H, $J = 7.8$ Hz), 5.27 (s, 1H), 4.20-4.10 (m, 2H), 3.10-3.05 (m, 1H), 1.20-1.17 (m, 6H), 1.14 (d, 3H, $J = 6.6$ Hz); ^{13}C NMR (CDCl_3 , 150 MHz): δ 199.2, 161.3, 137.3, 134.5, 130.2, 128.7, 77.8, 62.8, 42.1, 18.1, 17.6, 13.7; HRMS (ESI) m/z calcd. for $\text{C}_{14}\text{H}_{19}\text{O}_5\text{S}$ $[\text{M}+\text{H}]^+$: 299.0948, found: 299.0947.

Ethyl 4,4-dimethyl-3-oxo-2-(phenylsulfonyl)pentanoate (3j)



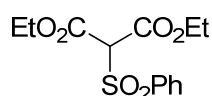
Yield: 51%; colorless oil; TLC, $R_f = 0.20$ (PE:EtOAc = 9:1); ^1H NMR (CDCl_3 , 600 MHz): δ 7.94 (d, 2H, $J = 7.2$ Hz), 7.68 (t, 1H, $J = 7.2$ Hz), 7.56 (t, 2H, $J = 7.8$ Hz), 5.56 (s, 1H), 4.20-4.13 (m, 2H), 1.19 (t, 3H, $J = 7.2$ Hz), 1.18 (s, 9H); ^{13}C NMR (CDCl_3 , 150 MHz): δ 200.1, 160.9, 137.4, 134.4, 130.4, 128.6, 73.9, 63.0, 46.2, 25.7, 13.7; HRMS (ESI) m/z calcd. For $\text{C}_{15}\text{H}_{21}\text{O}_5\text{S}$ $[\text{M}+\text{H}]^+$: 313.1104, found: 313.1104.

Dimethyl 2-(phenylsulfonyl)malonate (3k)



Yield: 66%; colorless oil; TLC, $R_f = 0.25$ (PE:EtOAc = 4:1); ^1H NMR (CDCl_3 , 600 MHz): δ 7.99 (dd, 2H, $J = 8.4, 1.2$ Hz), 7.71 (t, 1H, $J = 7.2$ Hz), 7.59 (t, 2H, $J = 7.2$ Hz), 5.00 (s, 1H), 3.79 (s, 6H); ^{13}C NMR (CDCl_3 , 150 MHz): δ 161.2, 137.3, 134.7, 130.0, 128.9, 74.3, 53.6; HRMS (ESI) m/z calcd. for $\text{C}_{11}\text{H}_{12}\text{NaO}_6\text{S}$ $[\text{M}+\text{Na}]^+$: 295.0247, found: 295.0242.

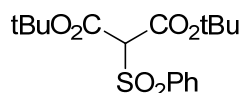
Diethyl 2-(phenylsulfonyl)malonate (3l)



Yield: 54%; colorless oil; TLC, $R_f = 0.30$ (PE: EtOAc = 9:1); ^1H NMR (CDCl_3 , 600 MHz): δ 8.01 (d, 2H, $J = 8.4$ Hz), 7.70 (t, 1H, $J = 7.2$ Hz), 7.58 (t, 2H, $J = 7.2$ Hz),

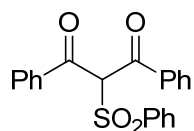
4.97 (s, 1H), 4.26-4.21 (m, 4H), 1.25 (t, 6H, $J = 7.2$ Hz); ^{13}C NMR (CDCl_3 , 150 MHz): δ 160.8, 137.5, 134.6, 130.0, 128.8, 74.6, 63.0, 13.7; HRMS (ESI) m/z calcd. for $\text{C}_{13}\text{H}_{16}\text{NaO}_6\text{S}$ $[\text{M}+\text{Na}]^+$: 323.0560, found: 323.0559.

Di-*tert*-butyl 2-(phenylsulfonyl)malonate (3m)



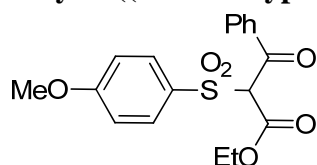
Yield: 48%; colorless oil; TLC, $R_f = 0.20$ (PE: EtOAc = 19:1); ^1H NMR (CDCl_3 , 600 MHz): δ 8.01 (d, 2H, $J = 8.4$ Hz), 7.66 (t, 1H, $J = 7.8$ Hz), 7.55 (t, 2H, $J = 7.8$ Hz), 4.79 (s, 1H), 1.41 (s, 18H); ^{13}C NMR (CDCl_3 , 150 MHz): δ 159.9, 134.2, 130.0, 128.7, 84.5, 76.2, 27.6; HRMS (ESI) m/z calcd. for $\text{C}_{17}\text{H}_{24}\text{NaO}_6\text{S}$ $[\text{M}+\text{Na}]^+$: 379.1186, found: 379.1182.

1,3-Diphenyl-2-(phenylsulfonyl)propane-1,3-dione (3n)



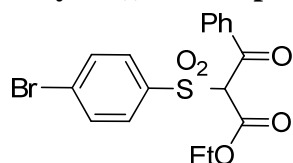
Yield: 81%; white solid; m.p. 150-152 °C; TLC, $R_f = 0.20$ (PE:Et₂O = 2:1); ^1H NMR (CDCl_3 , 600 MHz): δ 7.99-7.95 (m, 6H), 7.68 (t, 1H, $J = 7.2$ Hz), 7.62 (t, 2H, $J = 7.2$ Hz), 7.55 (t, 2H, $J = 7.8$ Hz), 7.49-7.46 (m, 4H), 7.05 (s, 1H); ^{13}C NMR (CDCl_3 , 150 MHz): δ 186.0, 137.2, 135.5, 134.5, 130.8, 129.2, 129.1, 128.5, 127.4, 77.4; HRMS (ESI) m/z calcd. for $\text{C}_{21}\text{H}_{16}\text{NaO}_4\text{S}$ $[\text{M}+\text{Na}]^+$: 387.0662, found: 387.0661.

Ethyl 2-((4-methoxyphenyl)sulfonyl)-3-oxo-3-phenylpropanoate (3o)



Yield: 85%; white solid; m.p. 94-96 °C; TLC, $R_f = 0.15$ (PE:EtOAc = 4:1); ^1H NMR (CDCl_3 , 600 MHz): δ 7.99 (d, 2H, $J = 7.2$ Hz), 7.87 (d, 2H, $J = 9.0$ Hz), 7.65 (t, 1H, $J = 7.2$ Hz), 7.51 (t, 2H, $J = 7.2$ Hz), 6.99 (d, 2H, $J = 9.0$ Hz), 5.99 (s, 1H), 4.25-4.15 (m, 2H), 3.89 (s, 3H), 1.20 (t, 3H, $J = 7.2$ Hz); ^{13}C NMR (CDCl_3 , 150 MHz): δ 186.1, 164.6, 161.7, 134.4, 132.9, 130.8, 129.3, 128.9, 114.4, 113.9, 75.4, 62.9, 55.7, 13.8; HRMS (ESI) m/z calcd. for $\text{C}_{18}\text{H}_{18}\text{NaO}_6\text{S}$ $[\text{M}+\text{Na}]^+$: 385.0716, found: 385.0710.

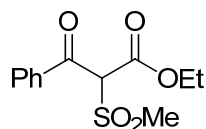
Ethyl 2-((4-bromophenyl)sulfonyl)-3-oxo-3-phenylpropanoate (3p)



Yield: 89%; pale yellow solid; m.p. 91-93 °C; TLC, $R_f = 0.30$ (PE:EtOAc = 4:1); ^1H NMR (CDCl_3 , 600 MHz): δ 7.99 (d, 2H, $J = 8.4$ Hz), 7.82 (d, 2H, $J = 9.0$ Hz), 7.69 (d,

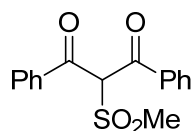
2H, $J = 9.0$ Hz), 7.67 (d, 1H, $J = 7.2$ Hz), 7.53 (t, 2H, $J = 7.8$ Hz), 6.01 (s, 1H), 4.24-4.16 (m, 2H), 1.20 (t, 3H, $J = 7.2$ Hz); ^{13}C NMR (CDCl_3 , 150 MHz): δ 185.9, 161.5, 135.5, 134.7, 132.5, 132.2, 131.9, 130.3, 129.3, 129.0, 75.4, 63.2, 13.8; HRMS (ESI) m/z calcd. for $\text{C}_{17}\text{H}_{16}\text{BrO}_5\text{S}$ $[\text{M}+\text{H}]^+$: 410.9896, found: 410.9896.

Ethyl 2-(methylsulfonyl)-3-oxo-3-phenylpropanoate (3q)



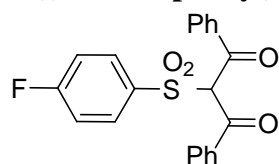
Yield: 43%; colorless oil; TLC, $R_f = 0.30$ (PE:EtOAc = 4:1); ^1H NMR (CDCl_3 , 600 MHz): δ 8.02 (d, 2H, $J = 7.2$ Hz), 7.68 (t, 1H, $J = 7.2$ Hz), 7.54 (d, 2H, $J = 7.8$ Hz), 5.80 (s, 1H), 4.33-4.25 (m, 2H), 3.37 (s, 3H), 1.28 (t, 3H, $J = 7.2$ Hz); ^{13}C NMR (CDCl_3 , 150 MHz): δ 186.6, 161.8, 135.3, 134.9, 129.4, 129.1, 73.8, 63.4, 40.1, 13.8; HRMS (ESI) m/z calcd. for $\text{C}_{12}\text{H}_{14}\text{NaO}_5\text{S}$ $[\text{M}+\text{Na}]^+$: 293.0454, found: 293.0455.

2-(Methylsulfonyl)-1,3-diphenylpropane-1,3-dione(3r)



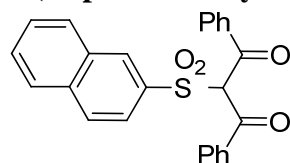
Yield: 57%; white solid; m.p. 166-168 °C; TLC, $R_f = 0.18$ (PE:EtOAc = 4:1); ^1H NMR (CDCl_3 , 600 MHz): δ 8.00-7.97 (m, 4H), 7.65 (tt, 2H, $J = 7.2, 1.2$ Hz), 7.52-7.48 (m, 4H), 6.84 (s, 1H), 3.40 (s, 3H); ^{13}C NMR (CDCl_3 , 150 MHz): δ 186.6, 135.2, 134.8, 129.22, 129.19, 75.3, 40.4; HRMS (ESI) m/z calcd. for $\text{C}_{16}\text{H}_{14}\text{NaO}_4\text{S}$ $[\text{M}+\text{Na}]^+$: 325.0505, found: 325.0508.

2-((4-Fluorophenyl)sulfonyl)-1,3-diphenylpropane-1,3-dione (3s)



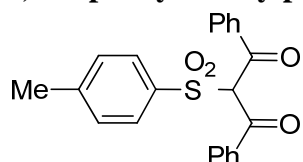
Yield: 82%; white solid; m.p. 153-155 °C; TLC, $R_f = 0.25$ (PE:EtOAc = 4:1); ^1H NMR (CDCl_3 , 600 MHz): δ 8.00-7.95 (m, 6H), 7.64 (t, 2H, $J = 7.2$ Hz), 7.49 (t, 4H, $J = 8.4$ Hz), 7.22 (t, 2H, $J = 8.4$ Hz), 7.07 (s, 1H); ^{13}C NMR (CDCl_3 , 150 MHz): δ 186.1, 166.5 ($J = 256.2$ Hz), 135.4, 134.6, 134.0 ($J = 9.9$ Hz), 130.2, 129.2, 128.5, 115.9 ($J = 22.4$ Hz), 77.3; HRMS (ESI) m/z calcd. for $\text{C}_{21}\text{H}_{15}\text{FNaO}_4\text{S}$ $[\text{M}+\text{Na}]^+$: 405.0567, found: 405.0572.

2-(Naphthalen-2-ylsulfonyl)-1,3-diphenylpropane-1,3-dione (3t)



Yield: 89%; white solid; m.p. 197-199 °C; TLC, $R_f = 0.13$ (PE:EtOAc = 4:1); ^1H NMR (CDCl_3 , 600 MHz): δ 8.53 (s, 1H), 8.01-7.95 (m, 7H), 7.92 (d, 1H, $J = 7.8$ Hz), 7.68 (t, 1H, $J = 7.8$ Hz), 7.63-7.59 (m, 3H), 7.47 (t, 4H, $J = 7.2$ Hz), 7.12 (s, 1H); ^{13}C NMR (CDCl_3 , 150 MHz): δ 186.1, 135.7, 135.5, 134.3, 133.1, 131.8, 129.9, 129.6, 129.3, 129.2, 129.1, 128.5, 127.9, 127.4, 124.9, 77.6; HRMS (ESI) m/z calcd. For $\text{C}_{25}\text{H}_{18}\text{NaO}_4\text{S}$ $[\text{M}+\text{Na}]^+$: 437.0818, found: 437.0819.

1,3-Diphenyl-2-tosylpropane-1,3-dione (3u)



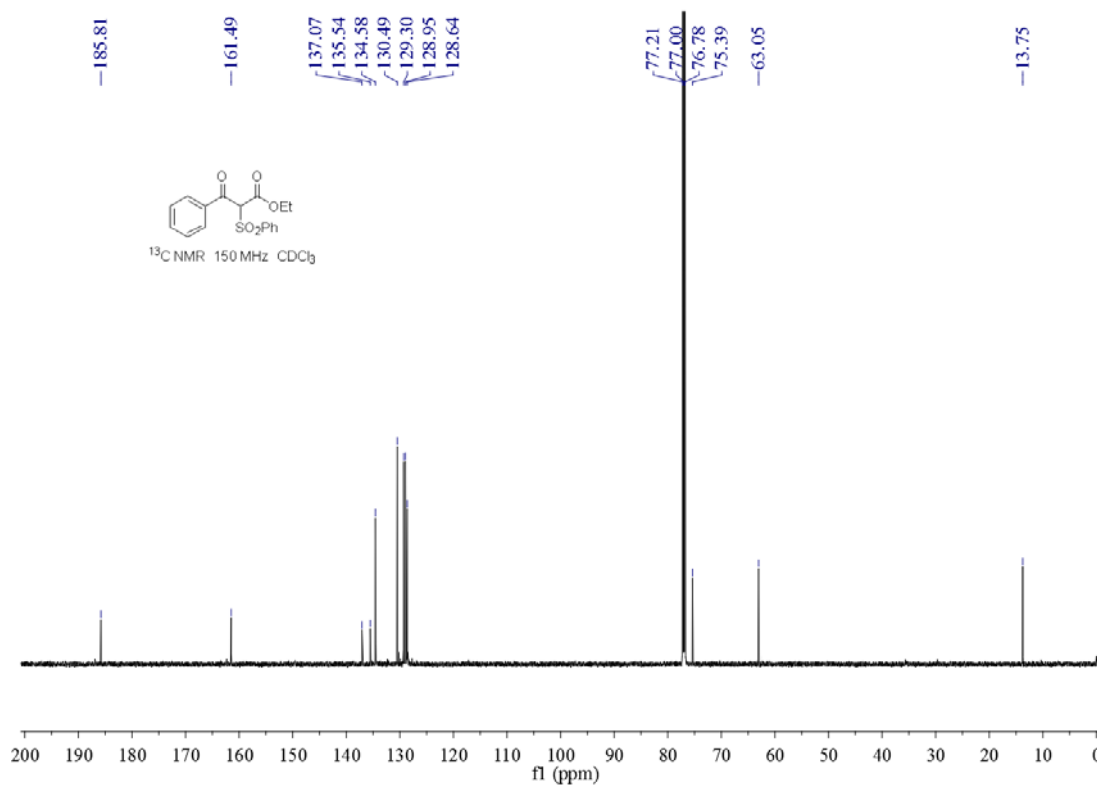
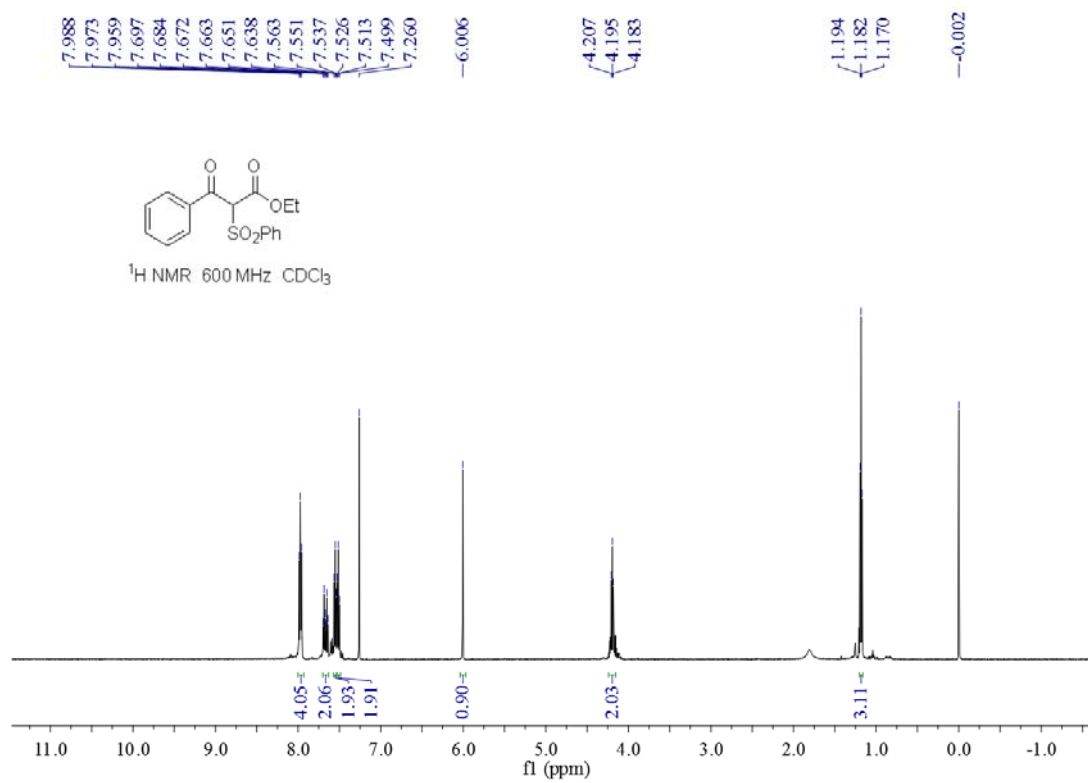
Yield: 95%; white solid; m.p. 164-166 °C; TLC, $R_f = 0.48$ (PE:Acetone = 2:1); ^1H NMR (CDCl_3 , 600 MHz): δ 7.97 (dd, 4H, $J = 8.4, 1.2$ Hz), 7.84 (d, 2H, $J = 7.8$ Hz), 7.62 (tt, 2H, $J = 7.8, 1.2$ Hz), 7.47 (t, 4H, $J = 7.8$ Hz), 7.34 (d, 2H, $J = 7.8$ Hz), 7.04 (s, 1H), 2.45 (s, 3H); ^{13}C NMR (CDCl_3 , 150 MHz): δ 186.1, 145.7, 135.6, 134.4, 130.8, 129.23, 129.18, 129.1, 129.0, 77.4, 21.7; HRMS (ESI) m/z calcd. for $\text{C}_{22}\text{H}_{18}\text{NaO}_4\text{S}$ $[\text{M}+\text{Na}]^+$: 401.0818, found: 401.0820.

Reference

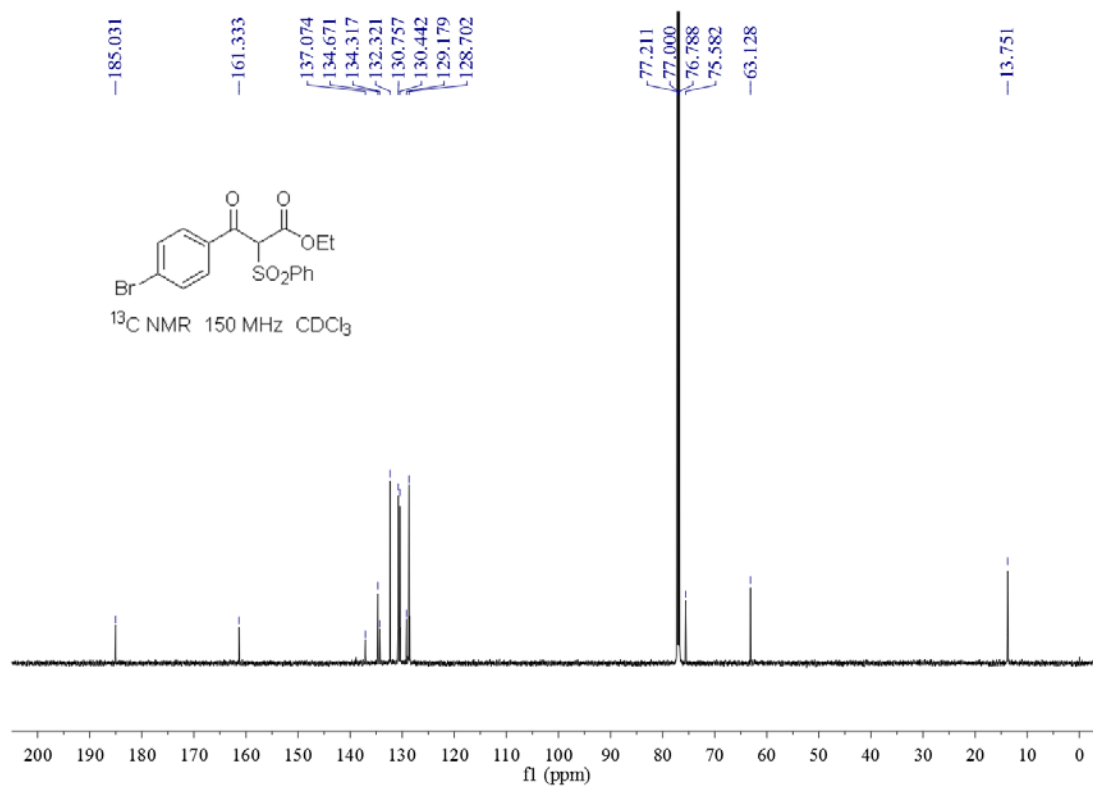
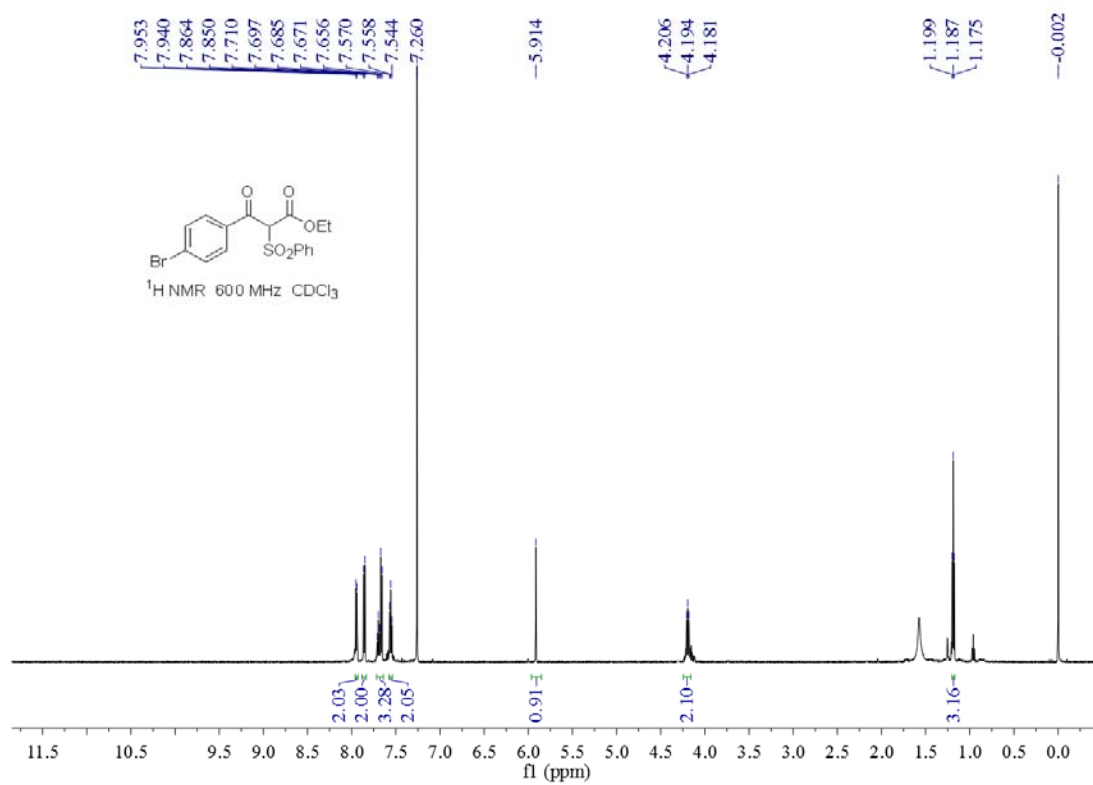
[S1] L. Liu, Y. Chi, K. Jen, *J. Org. Chem.* 1980, **45**, 406-410.

NMR Spectra of products

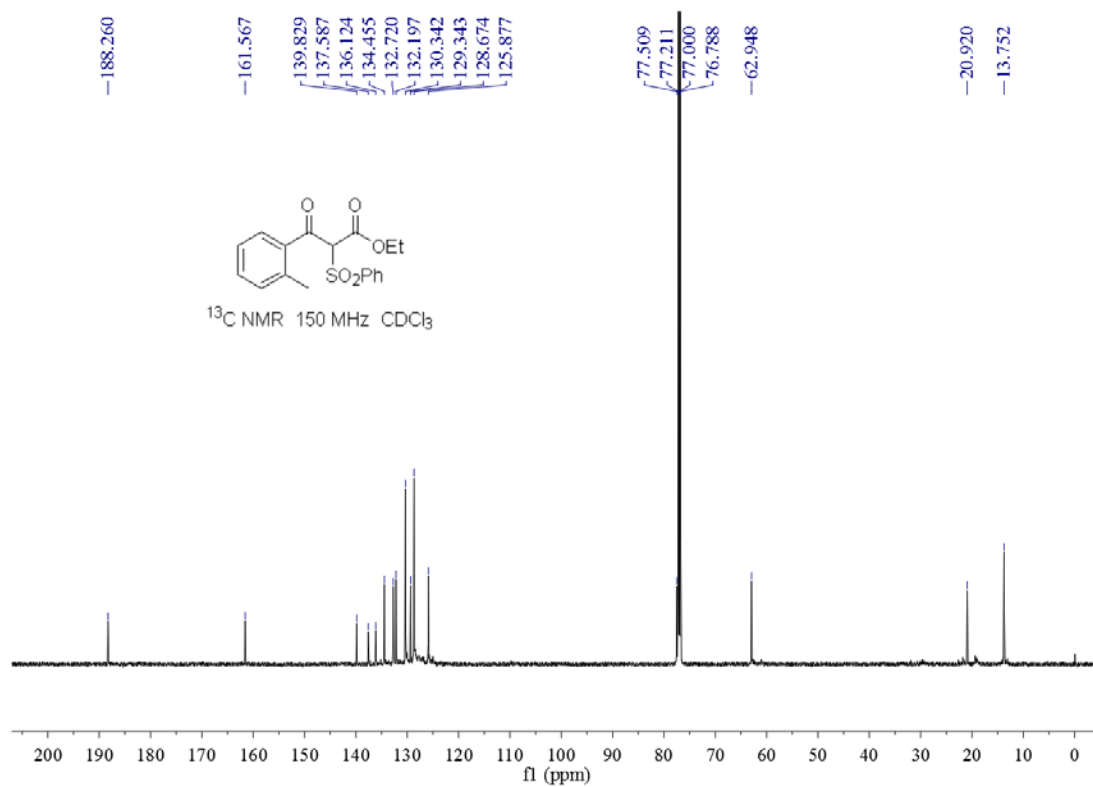
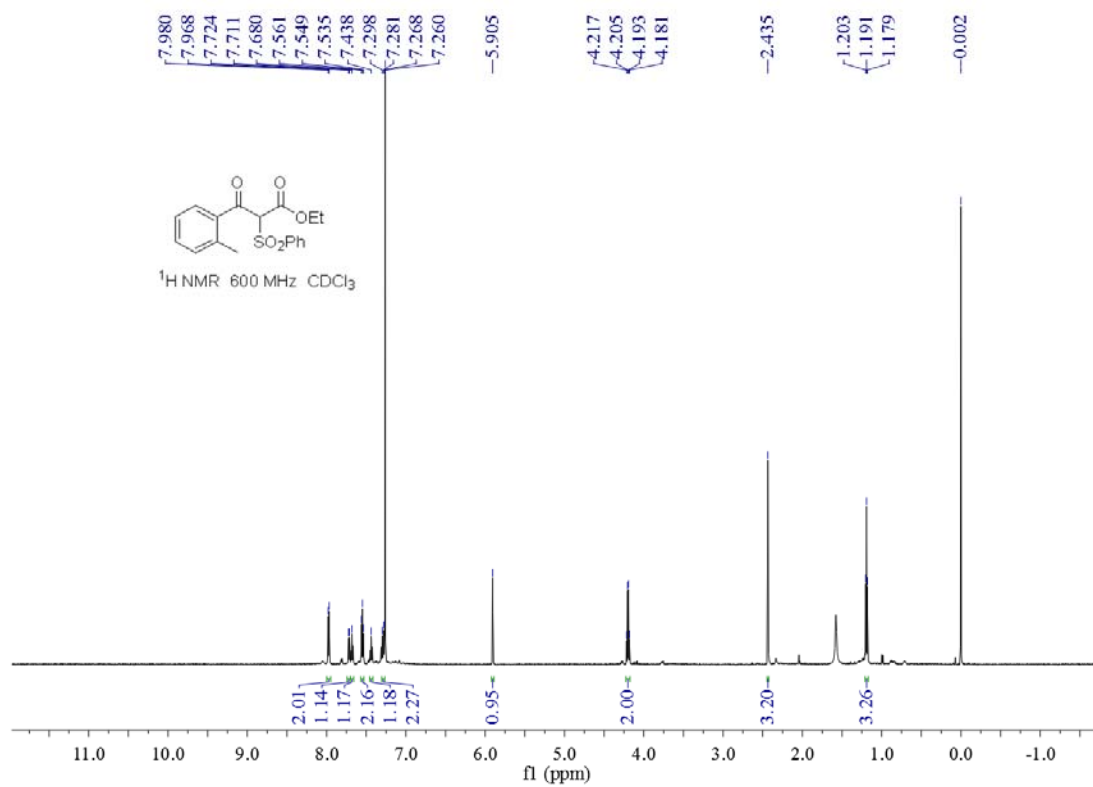
3a



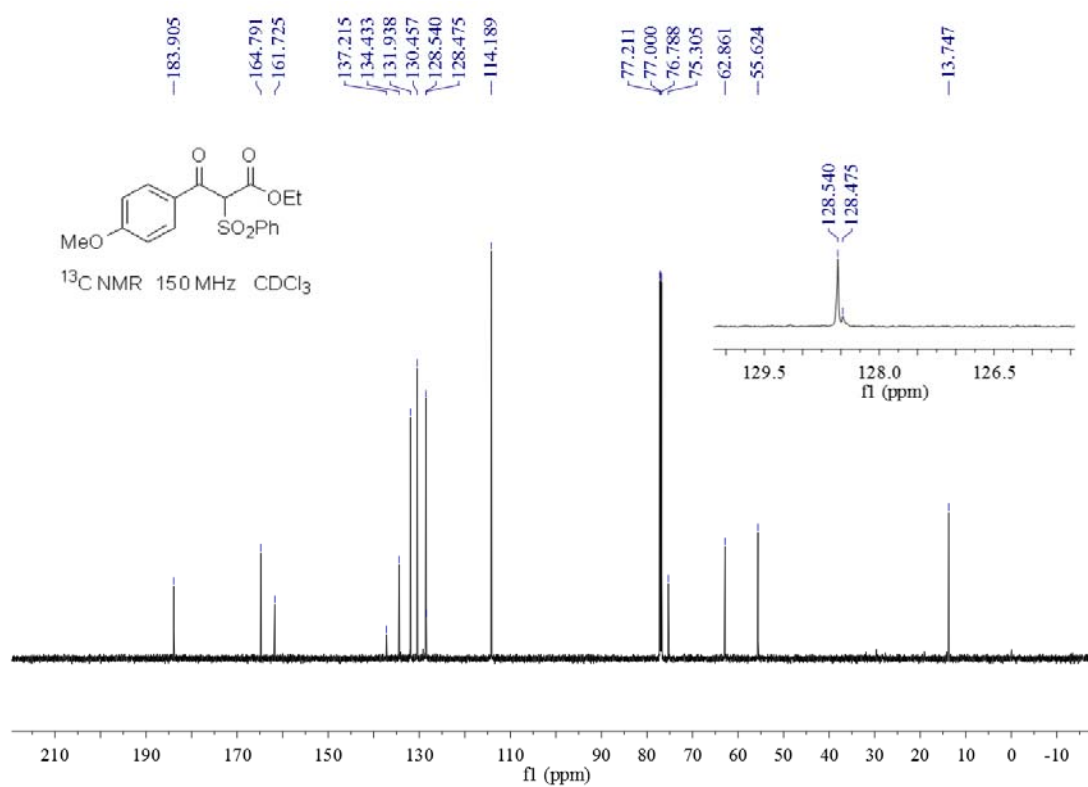
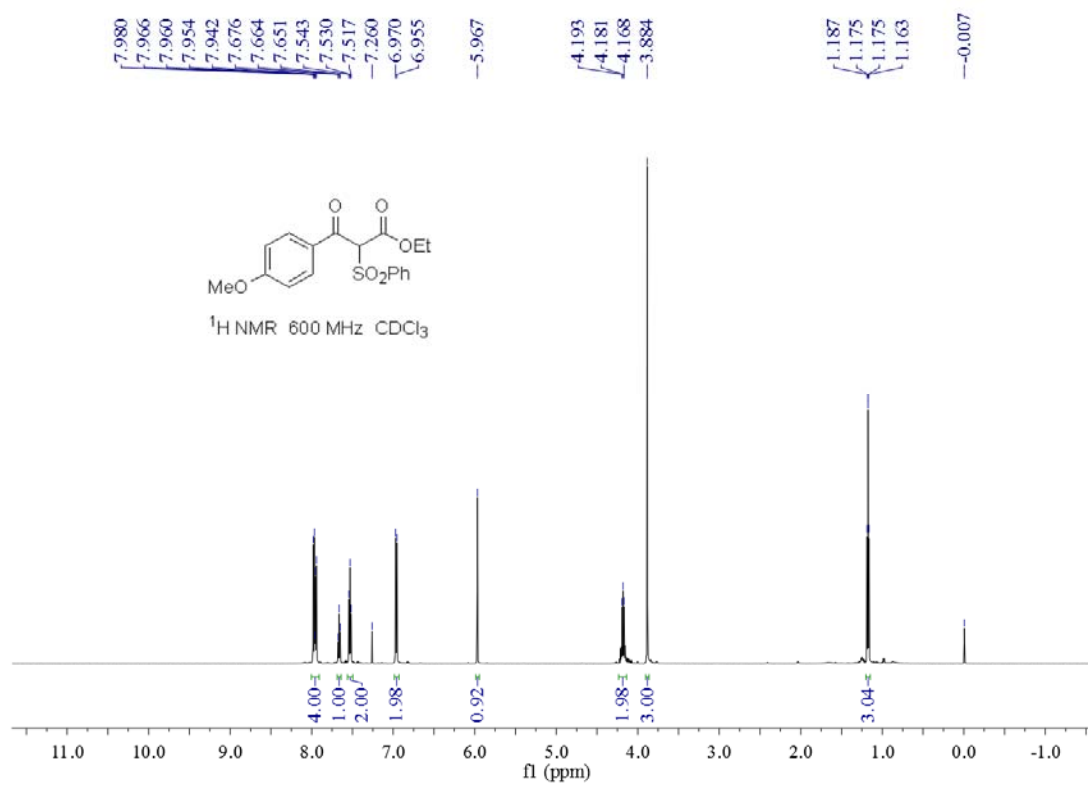
3b



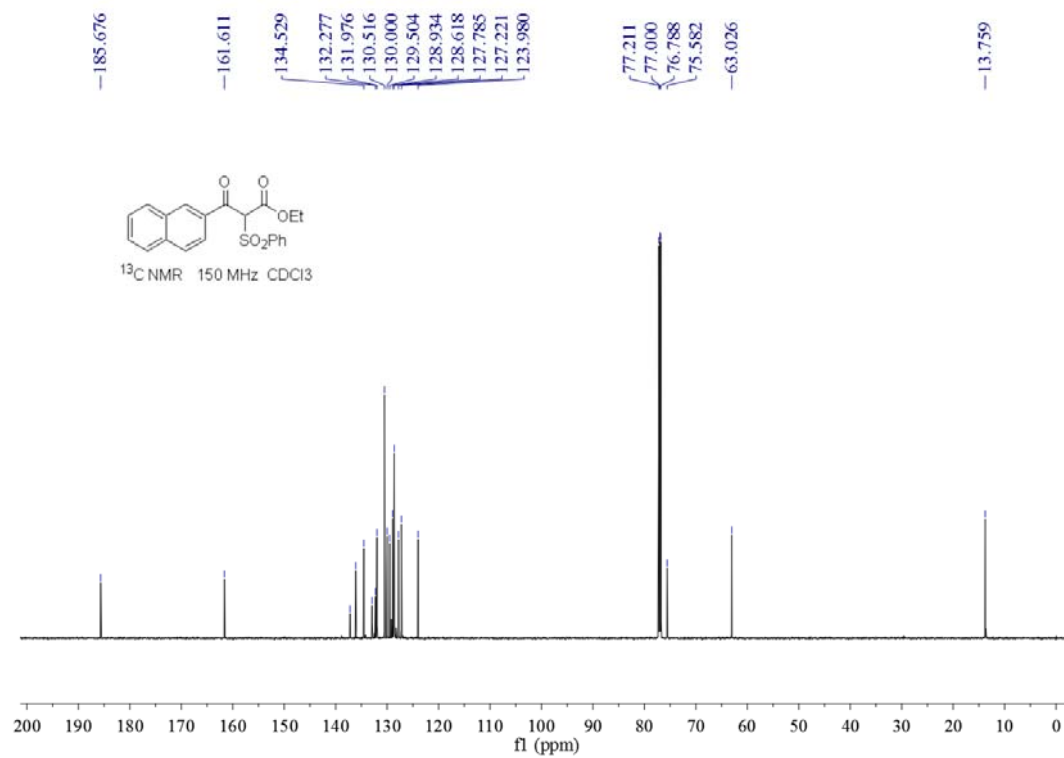
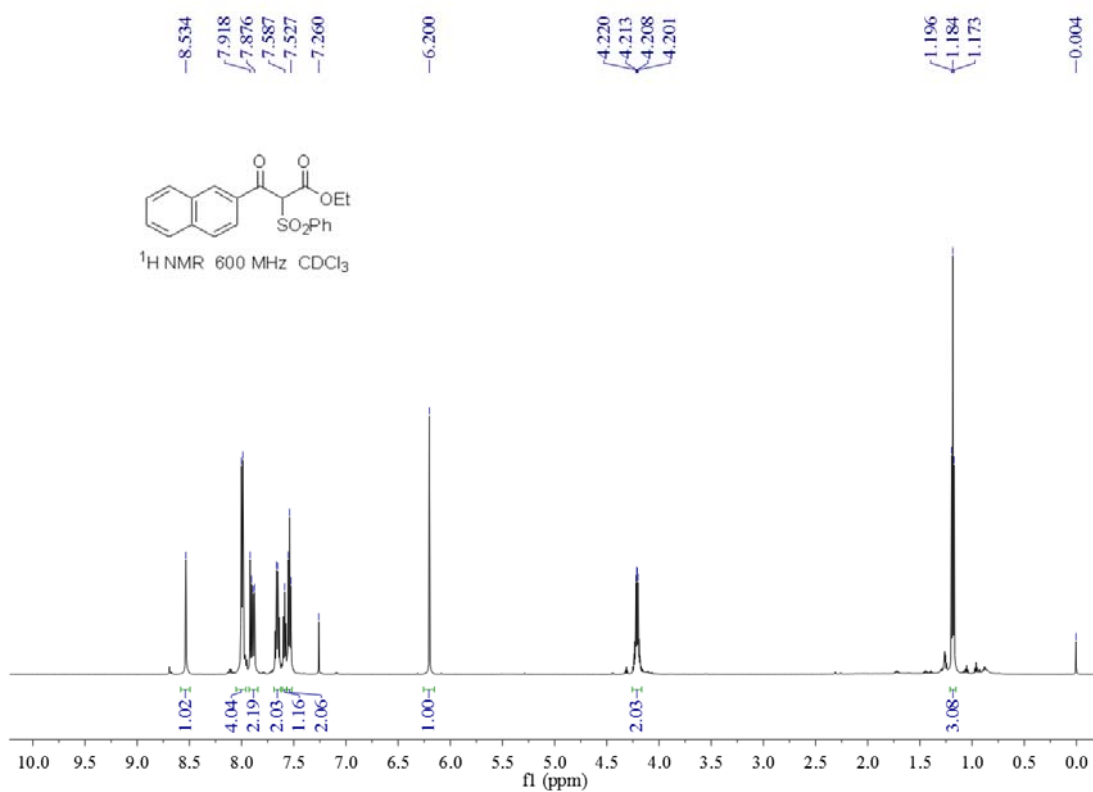
3c



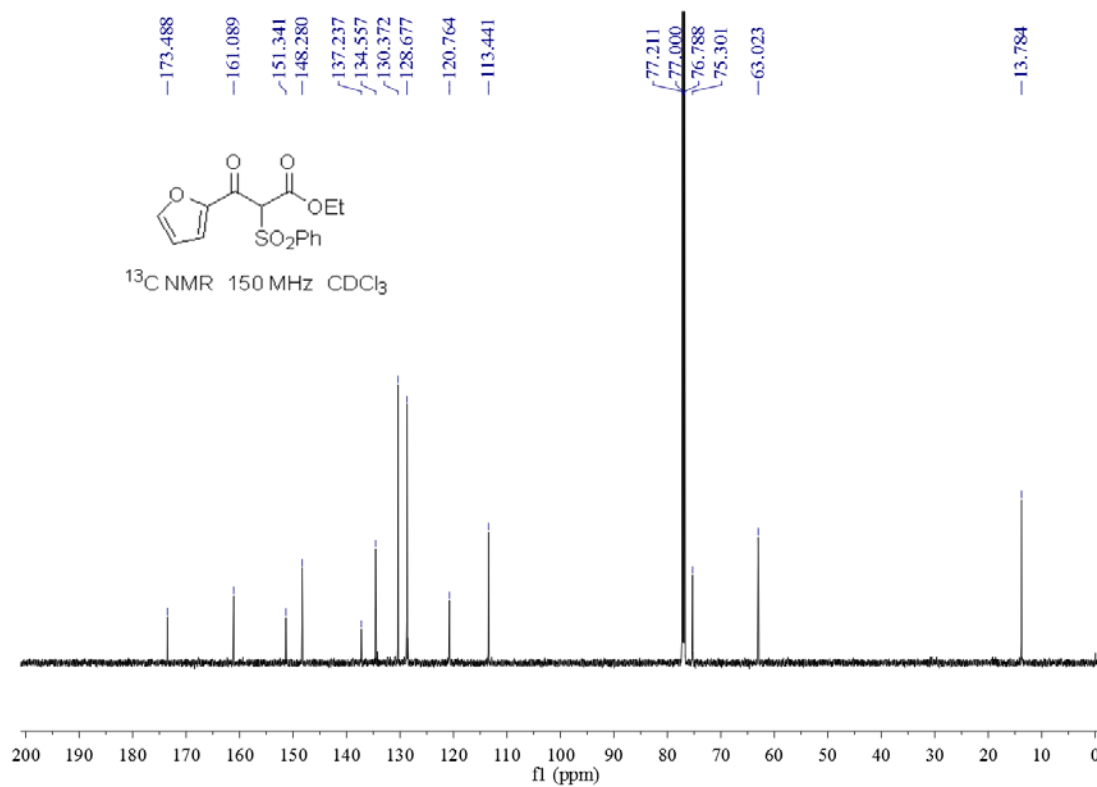
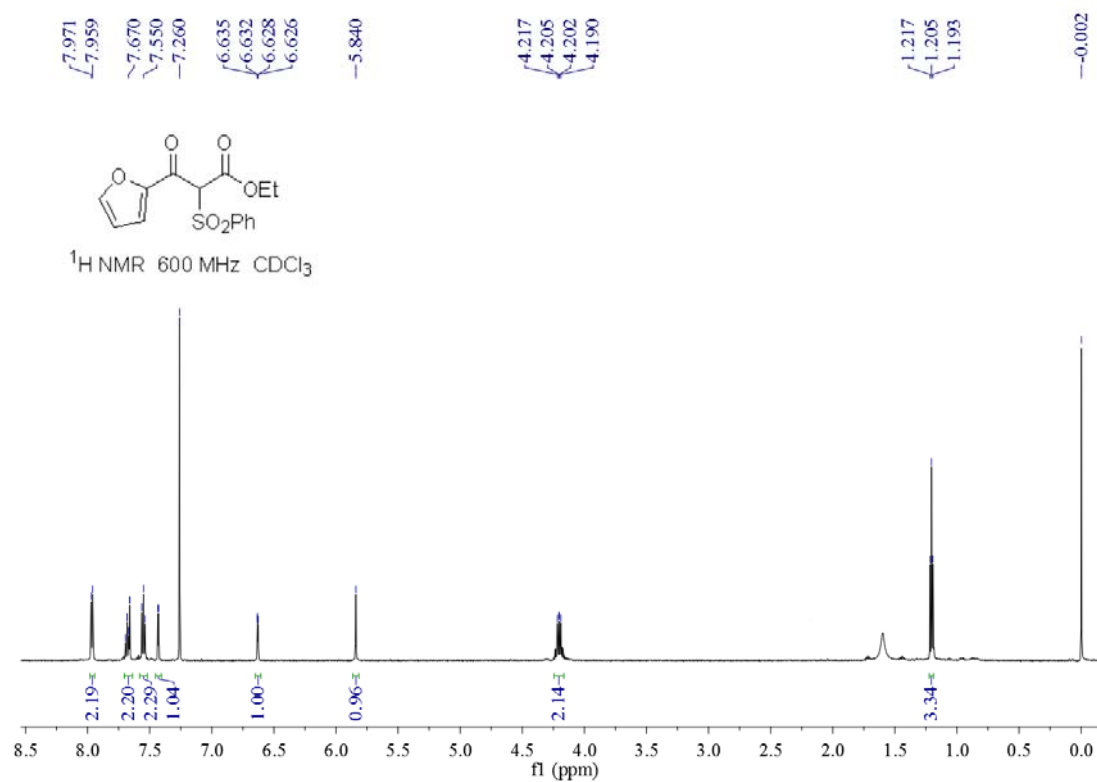
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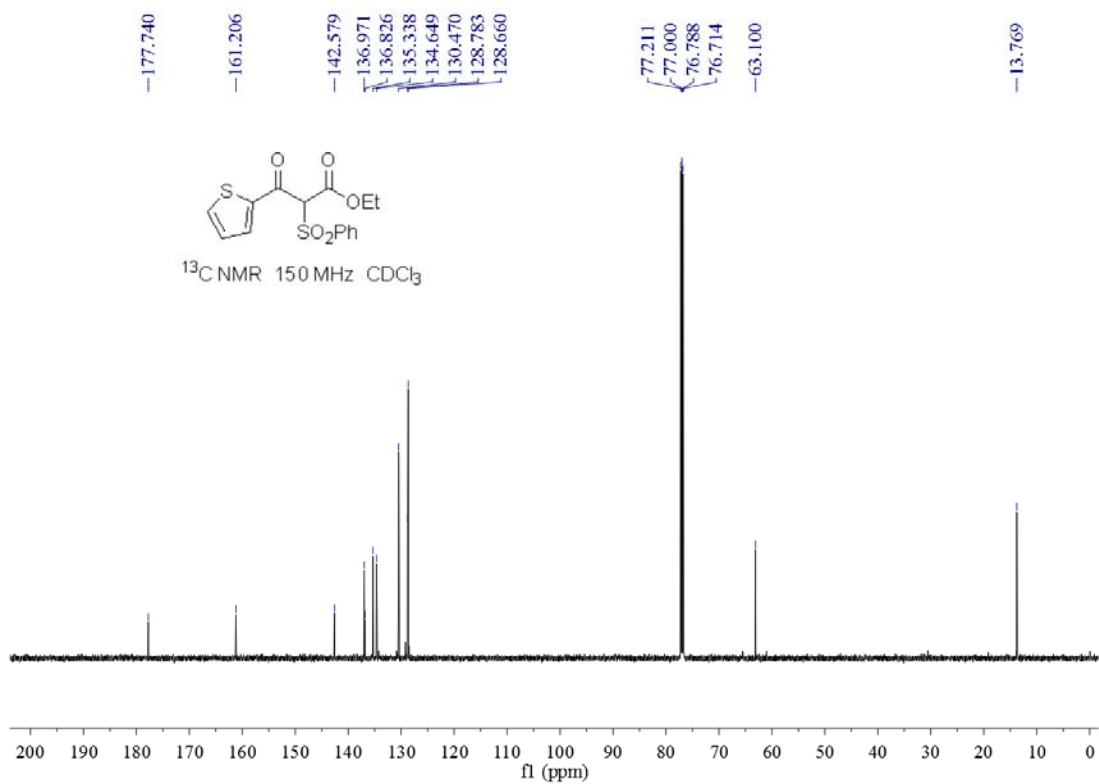
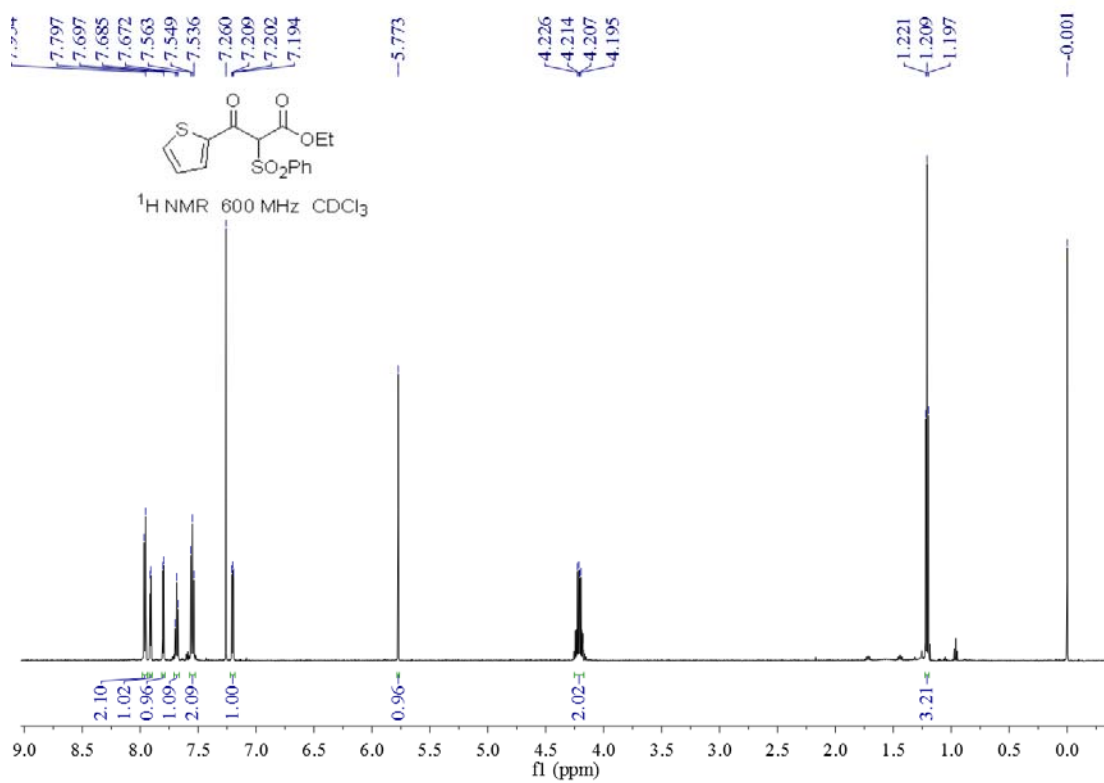
3e



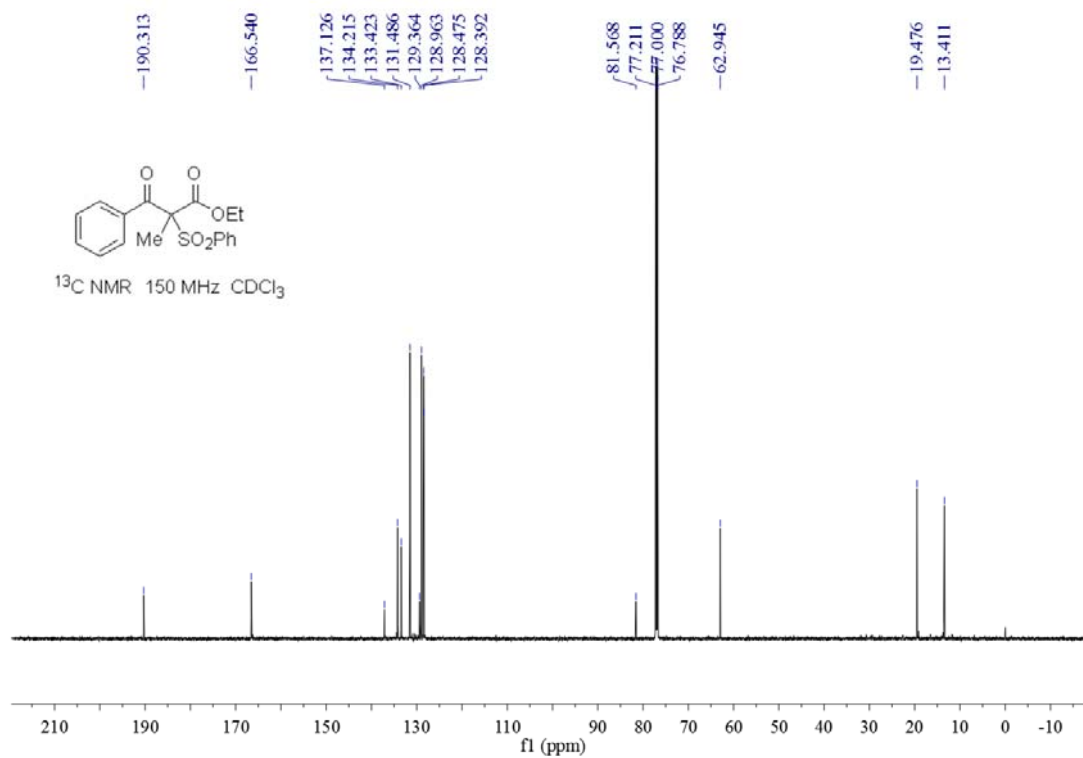
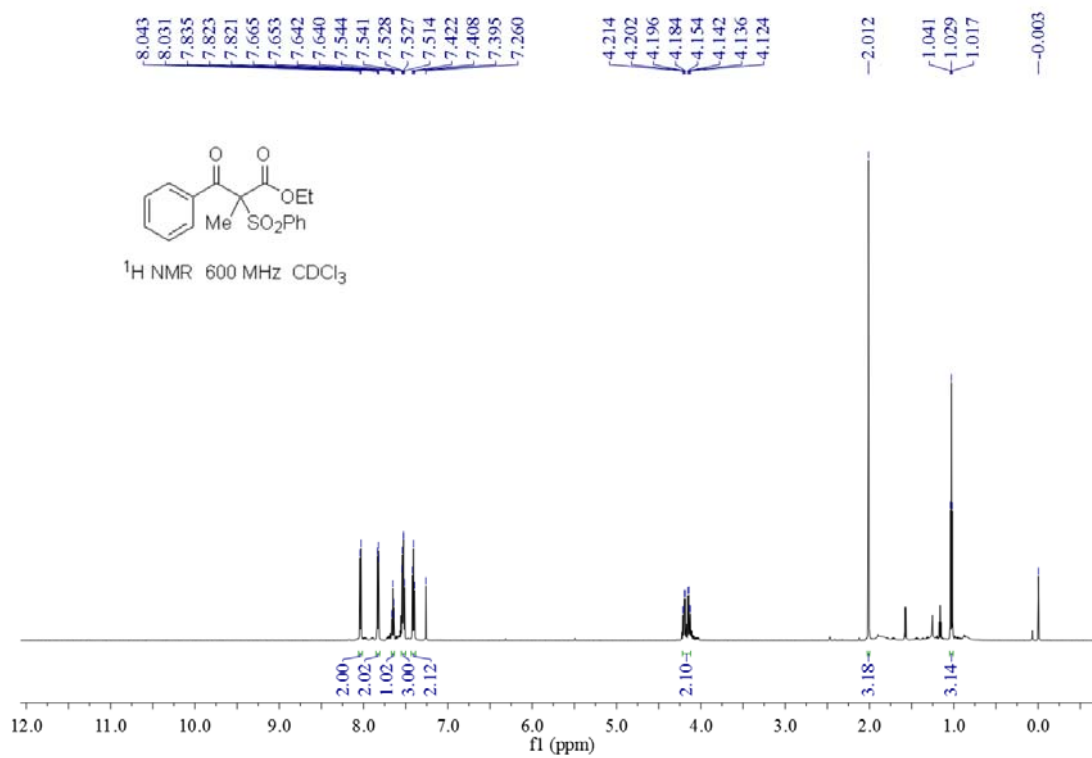
3f



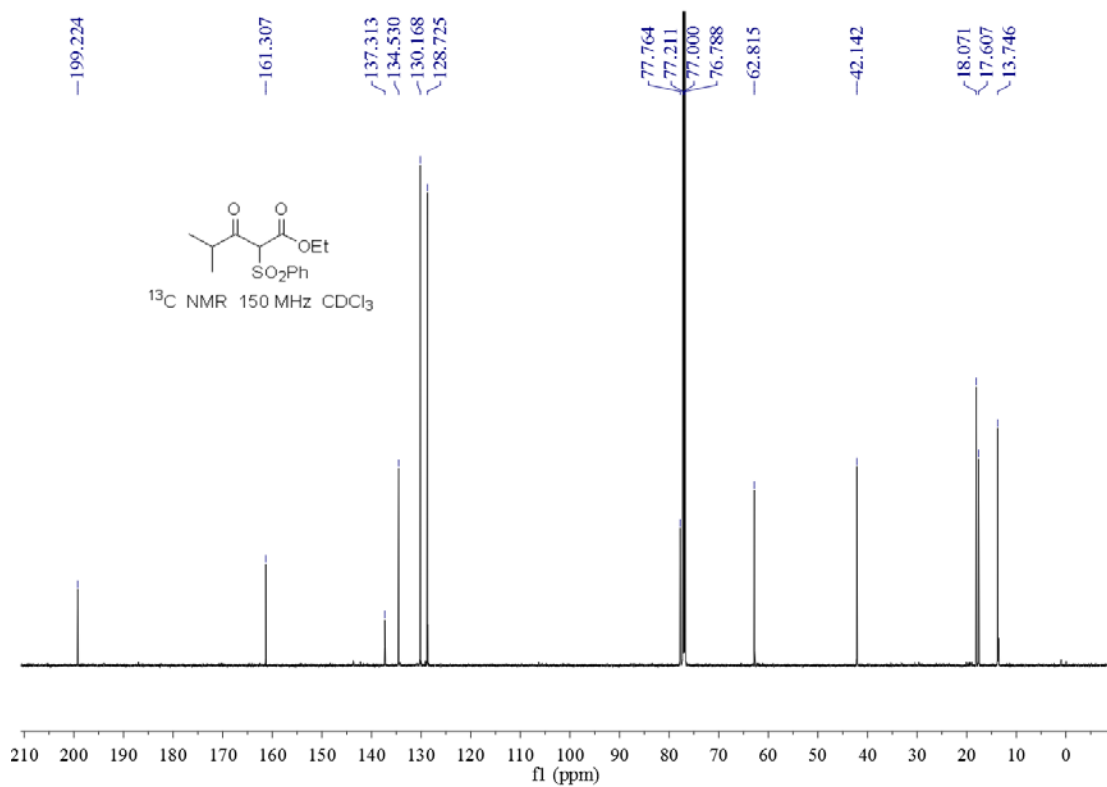
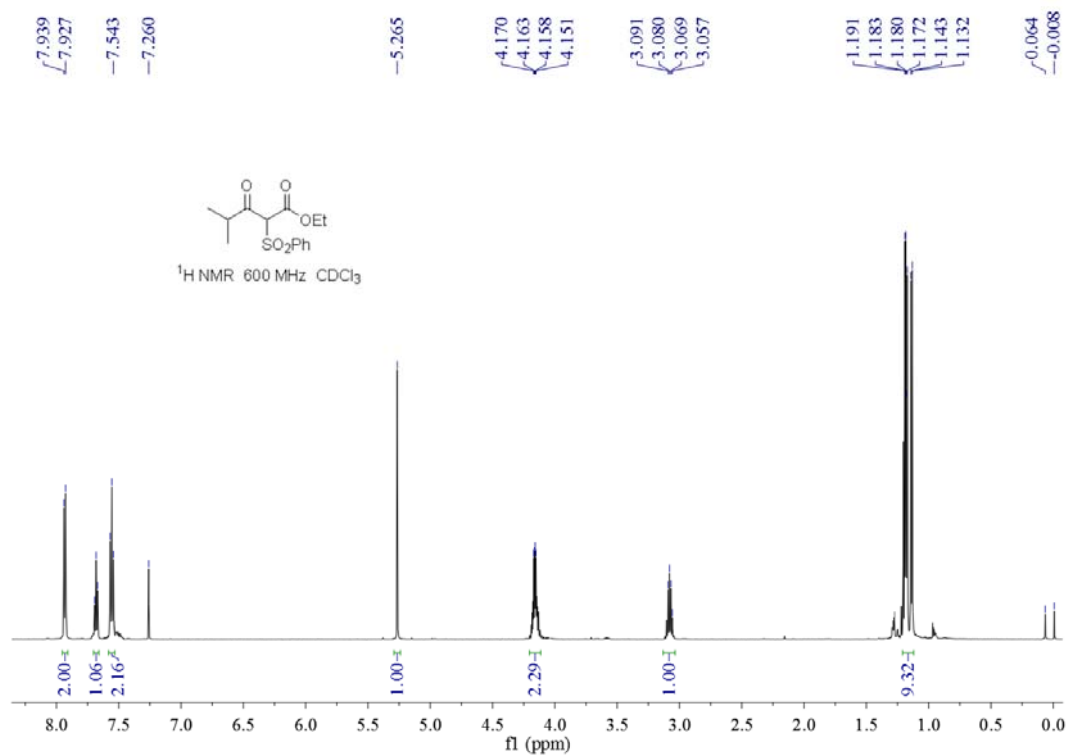
3g



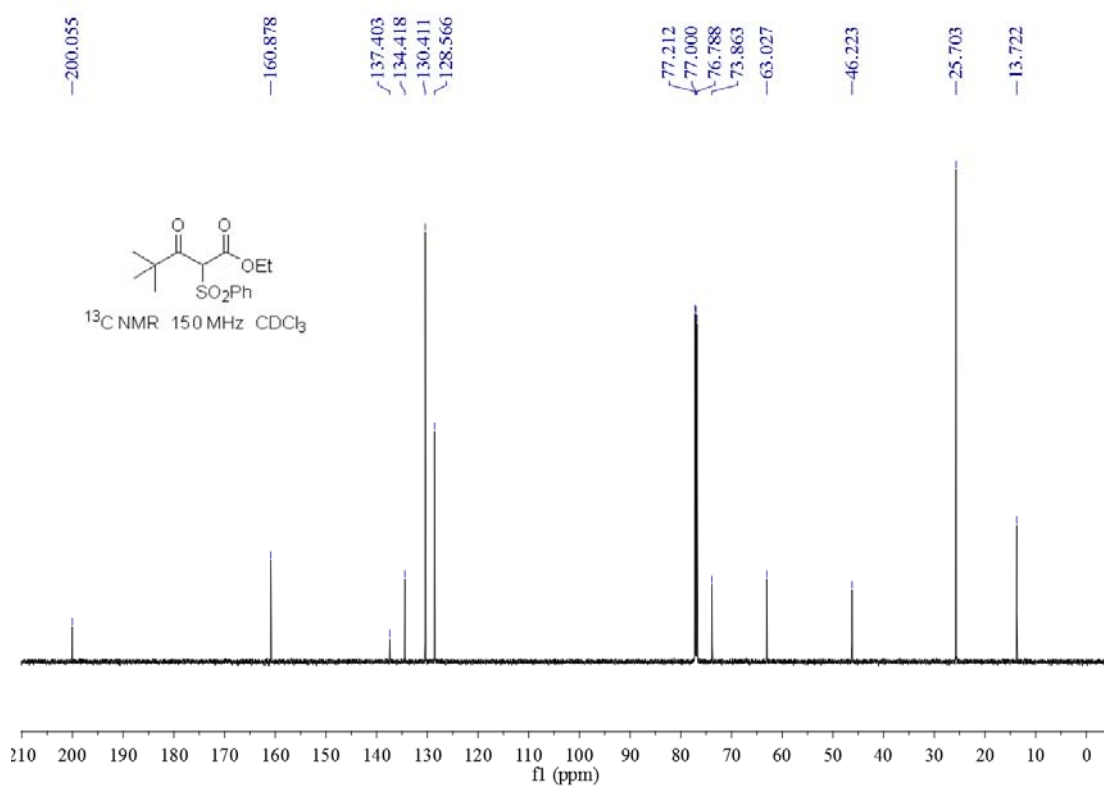
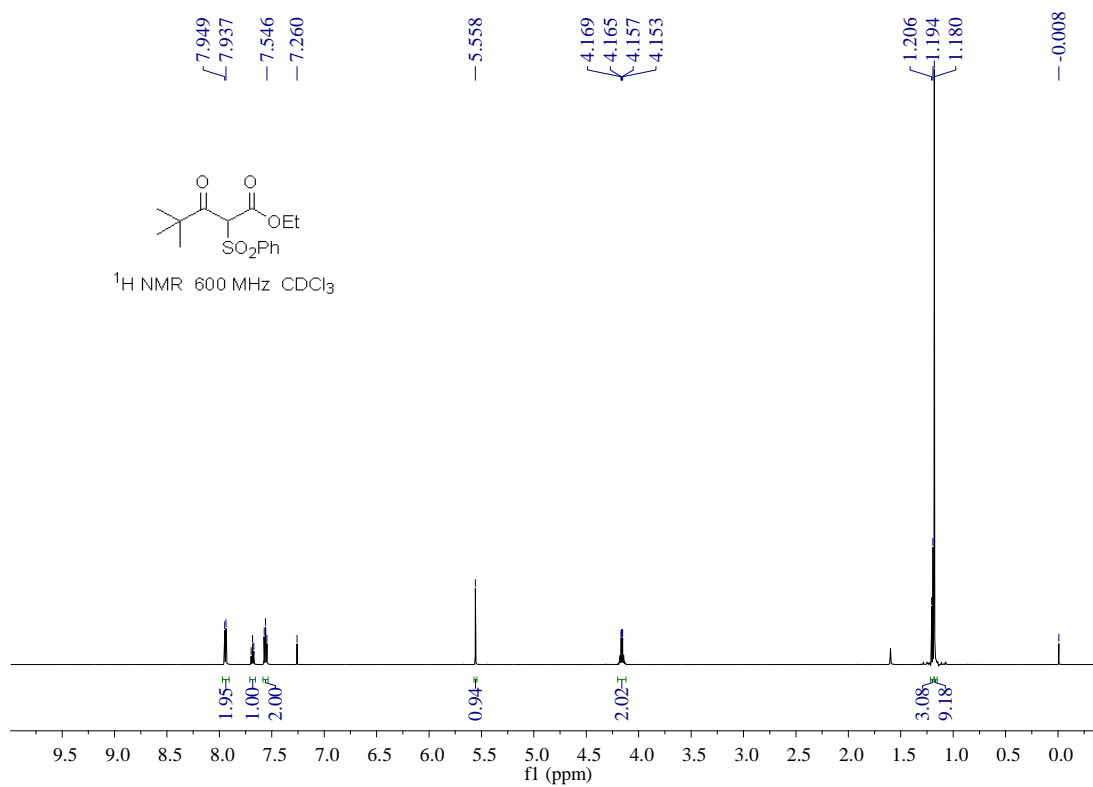
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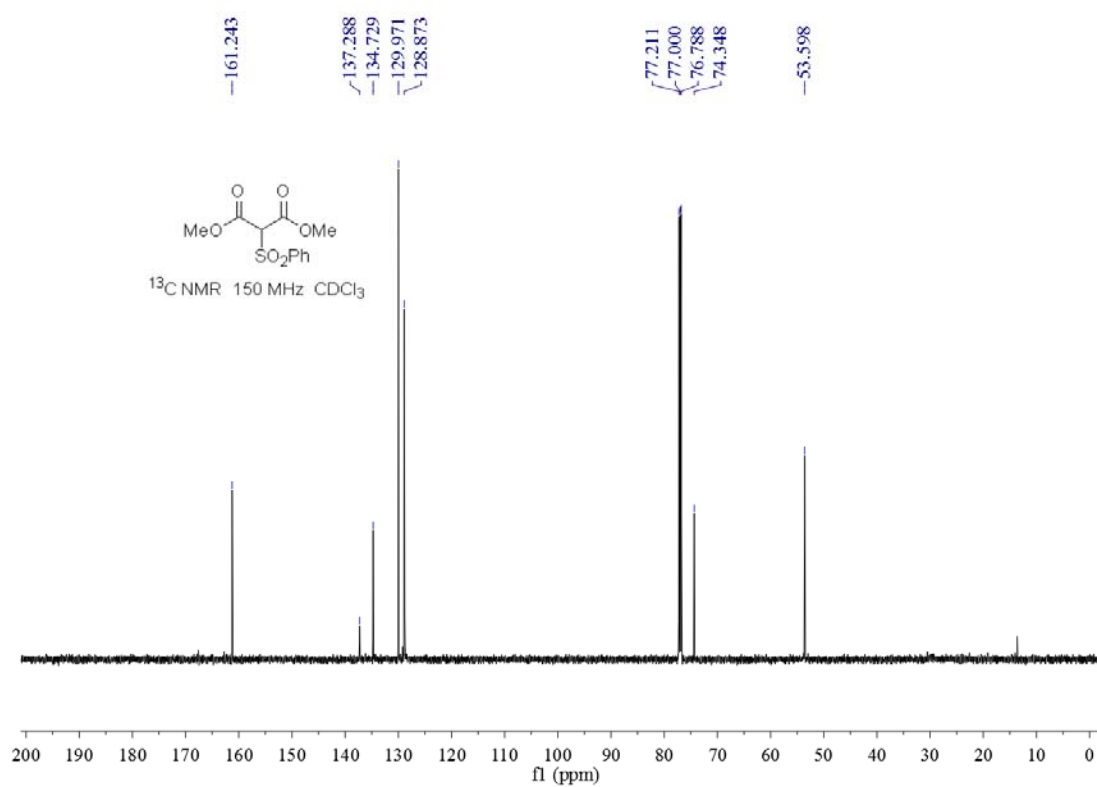
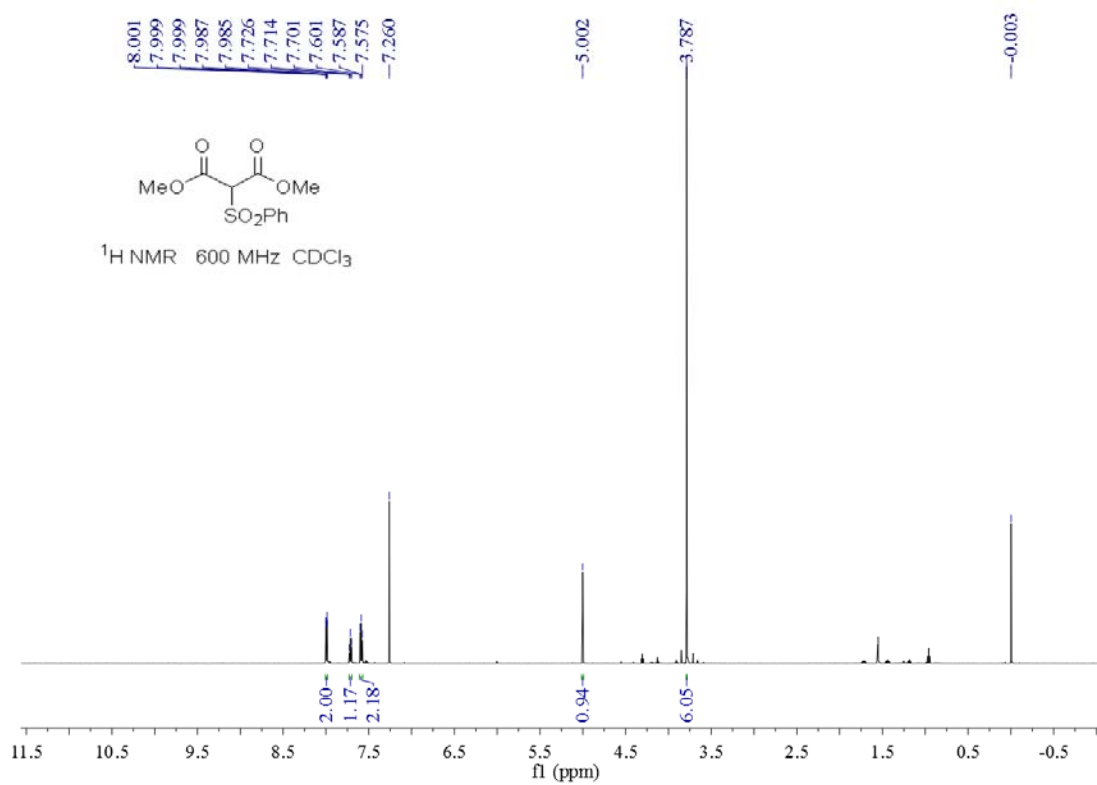
3i



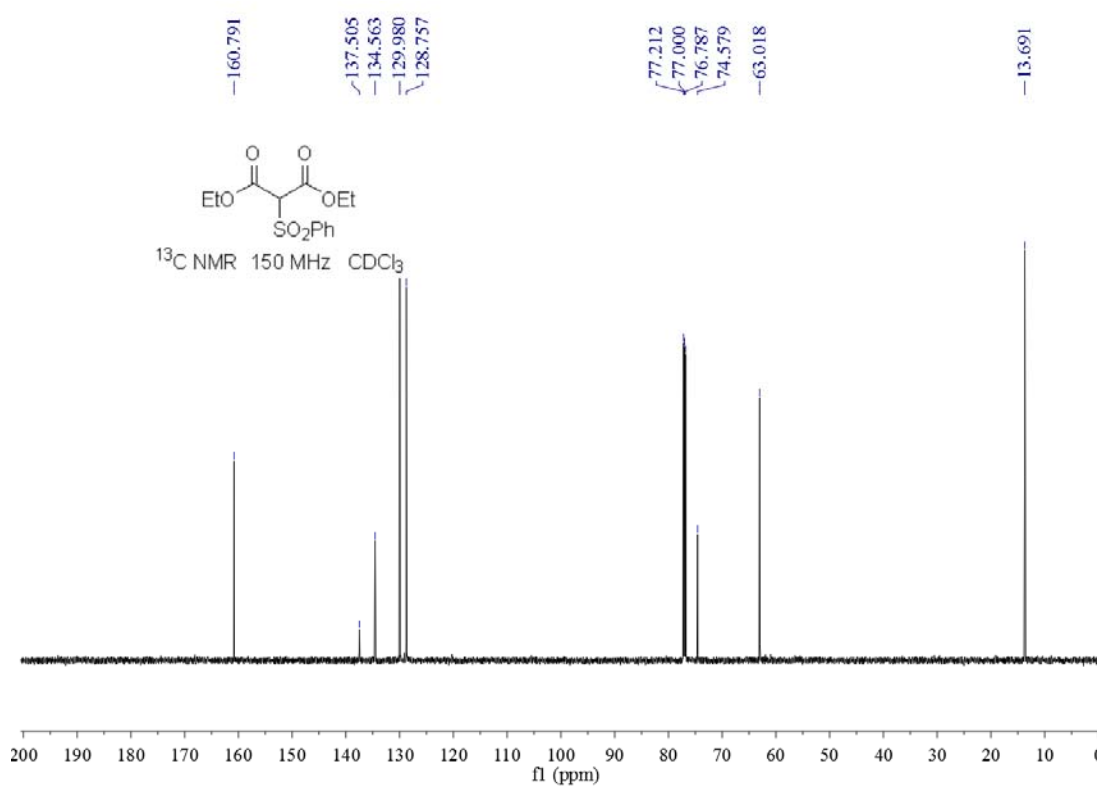
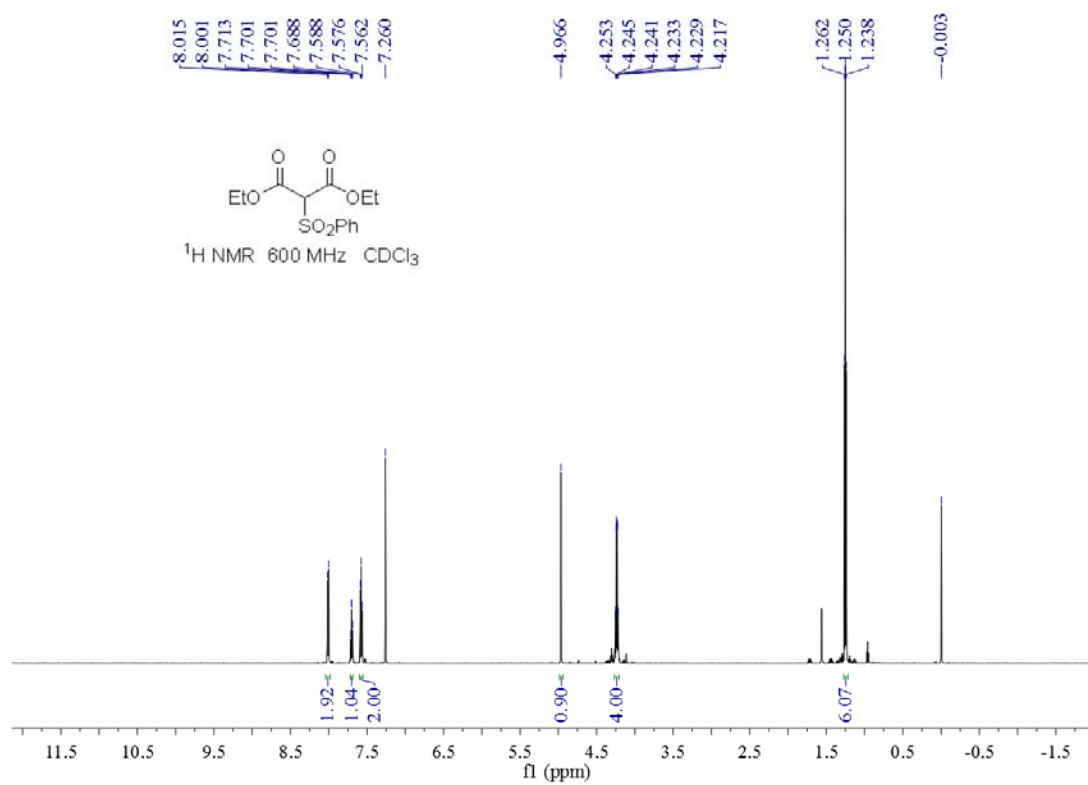
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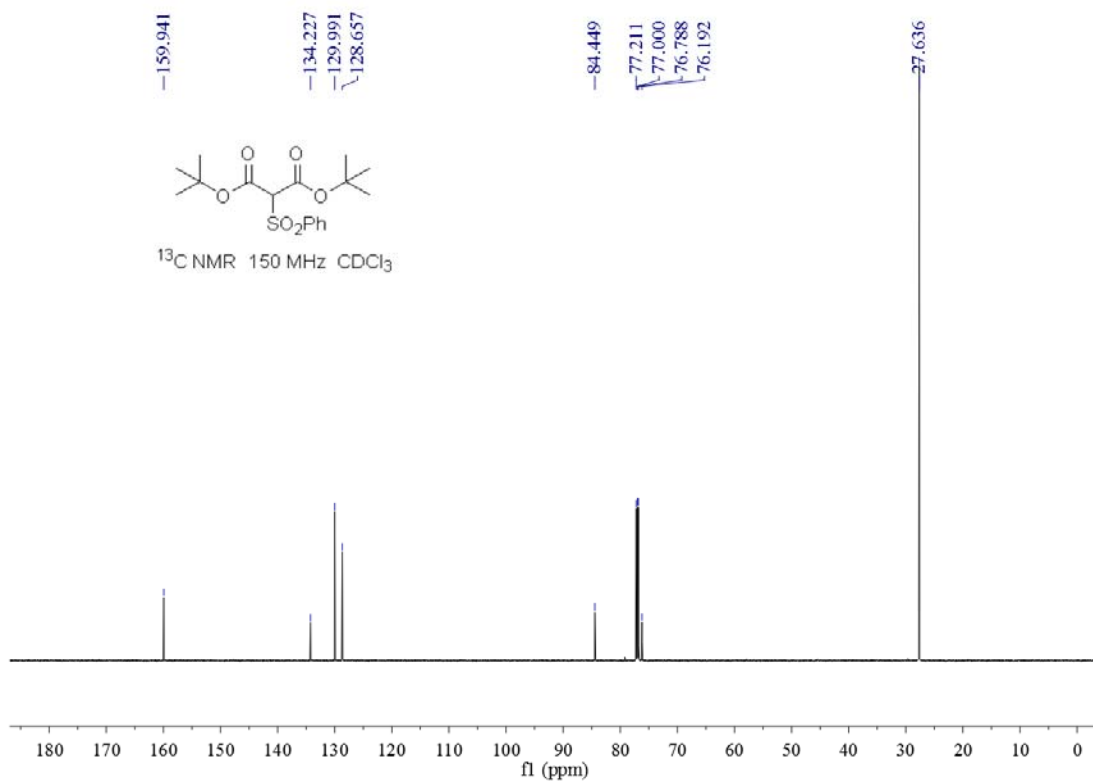
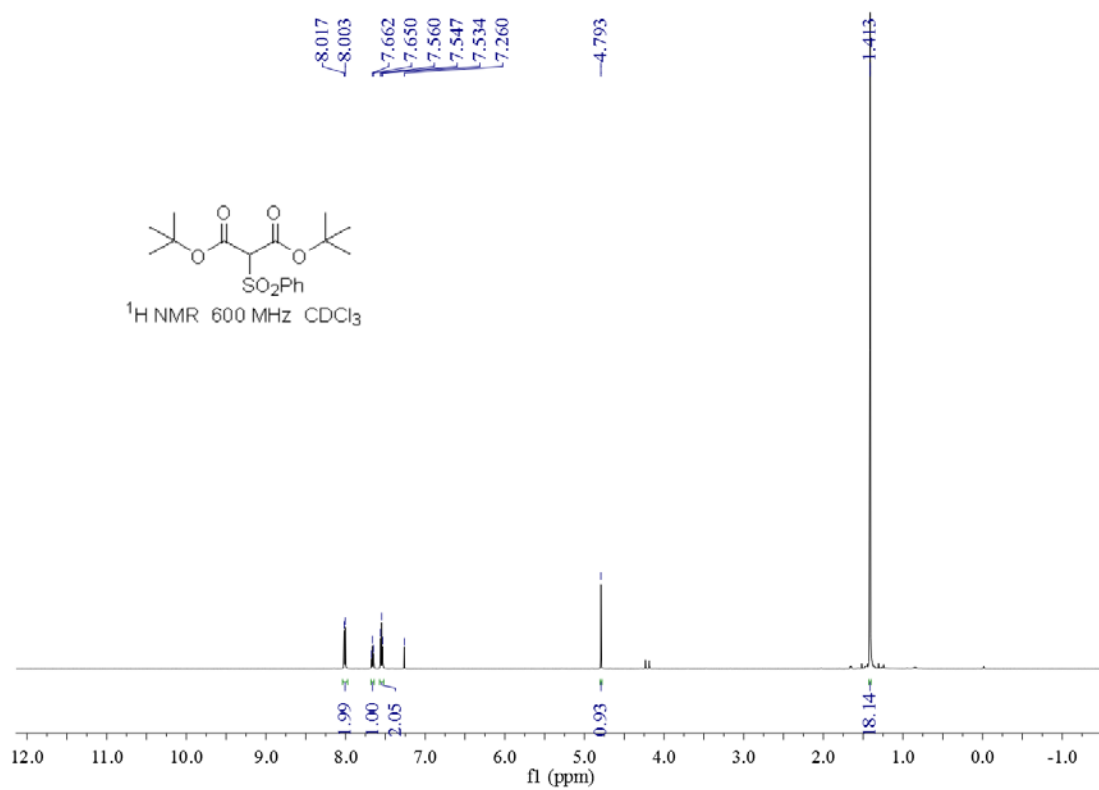
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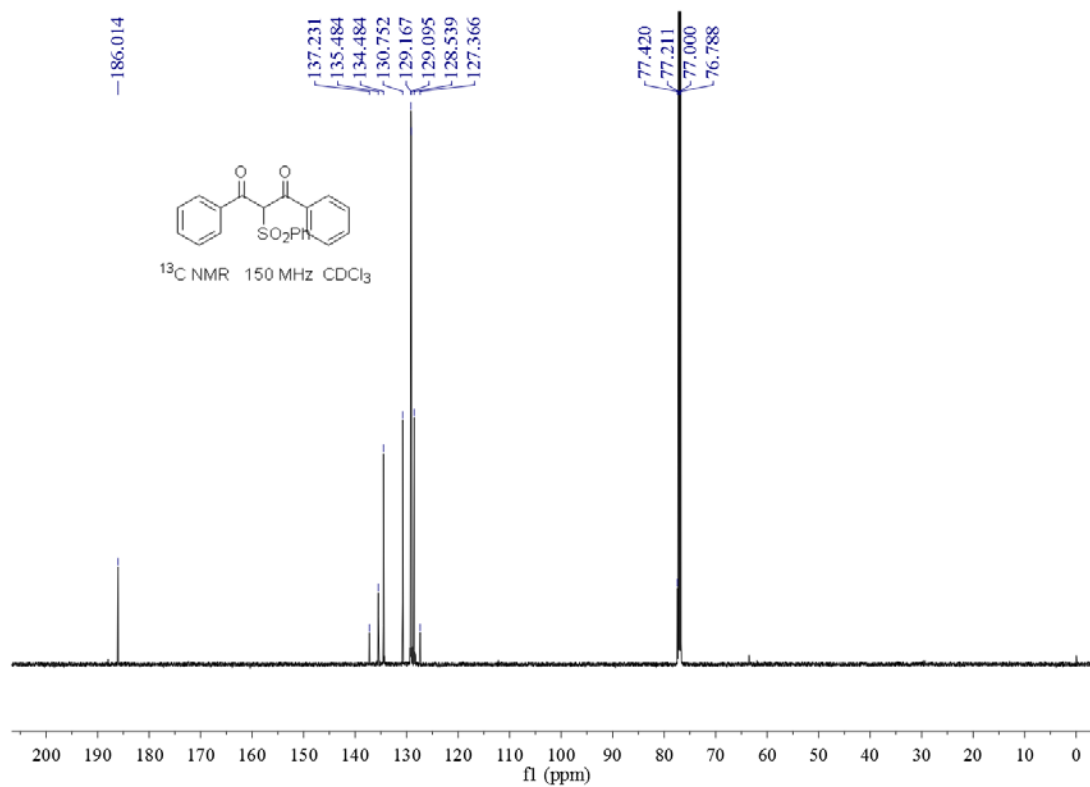
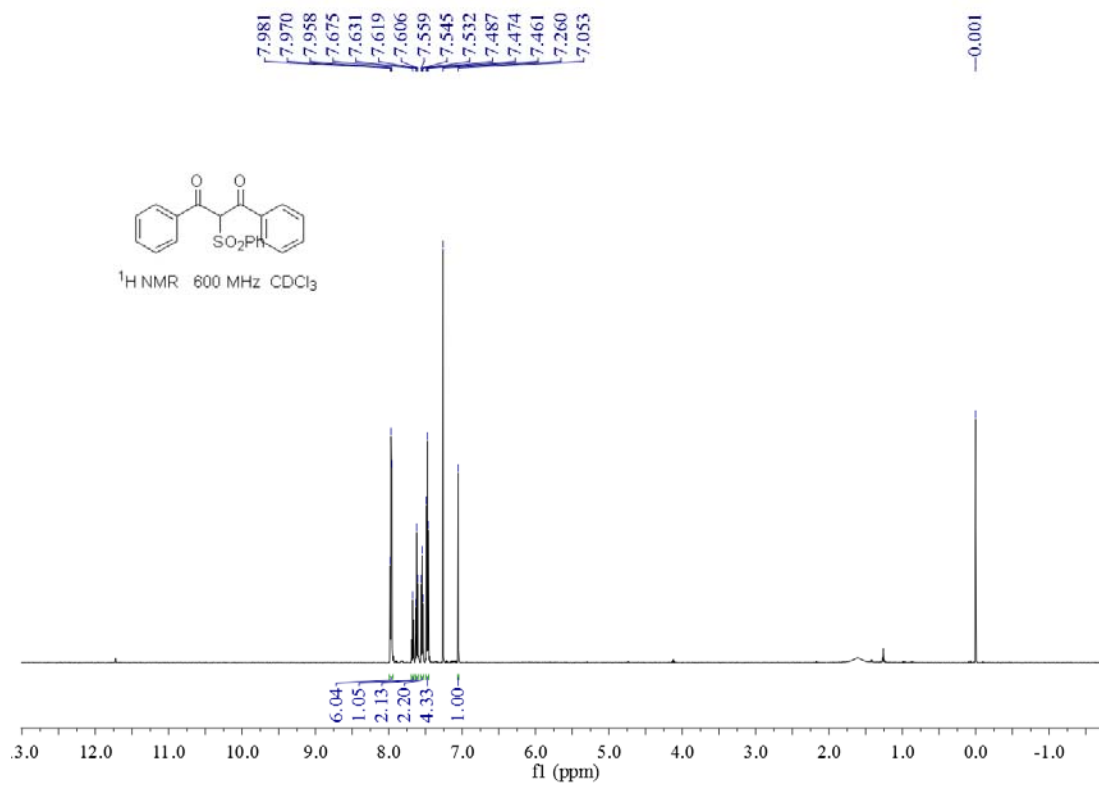
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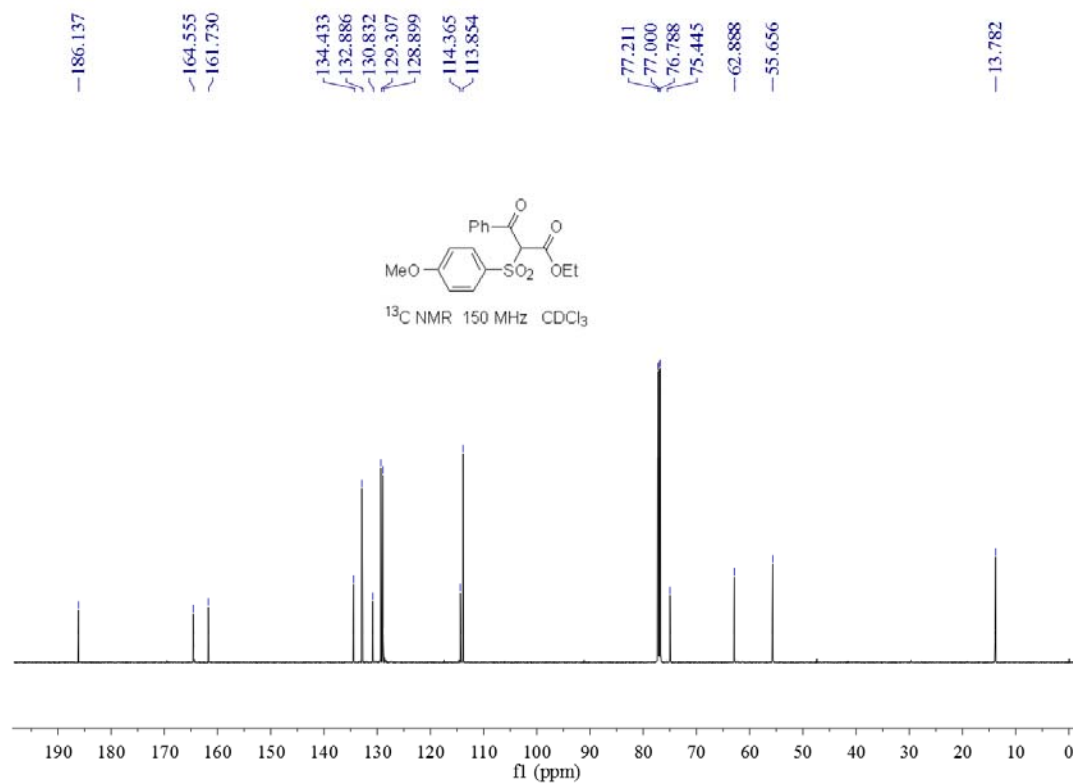
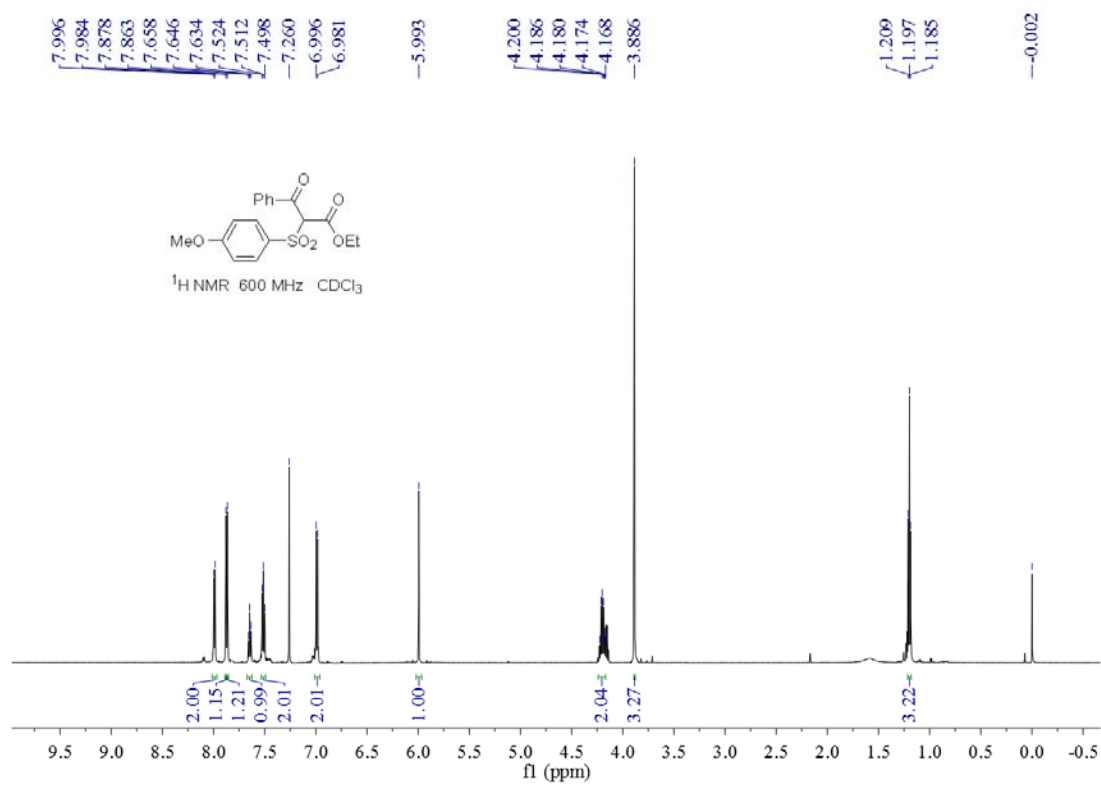
3m



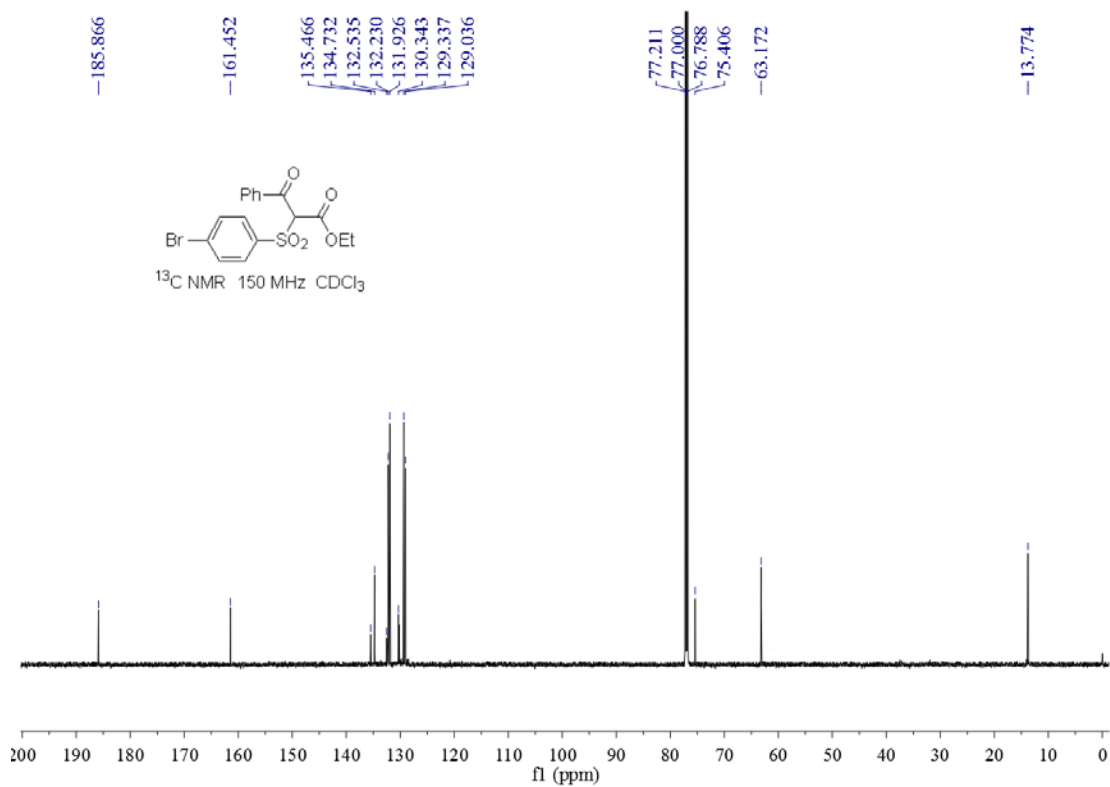
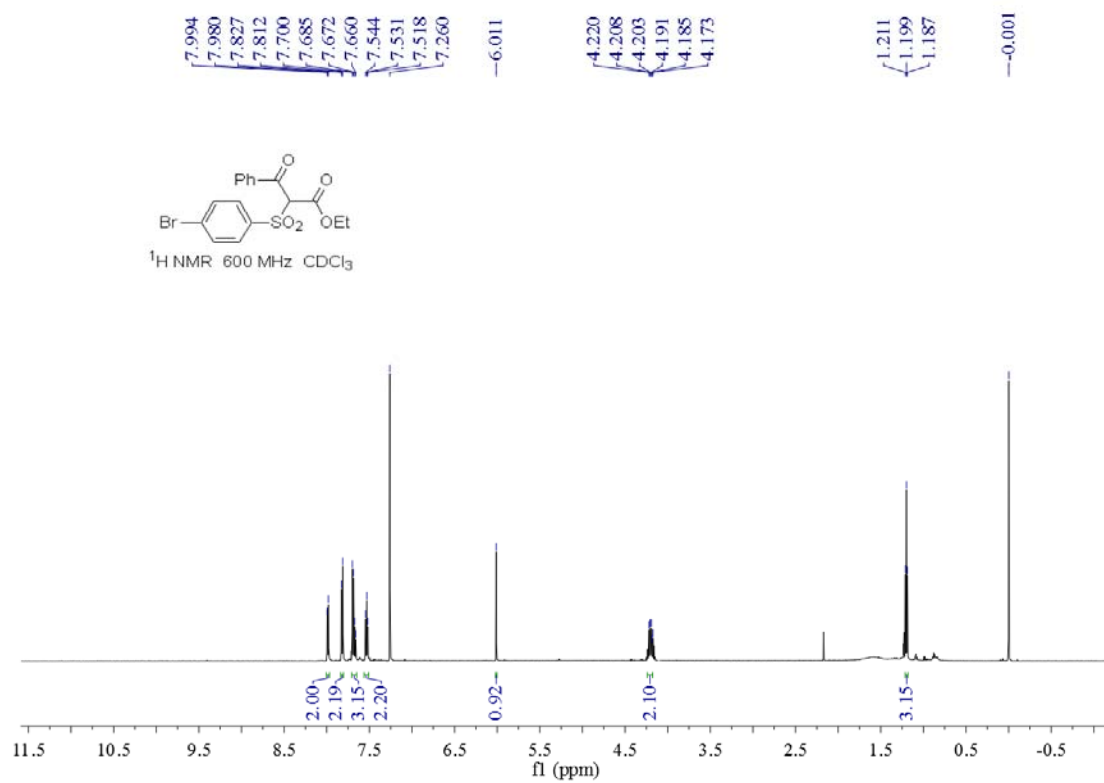
3n



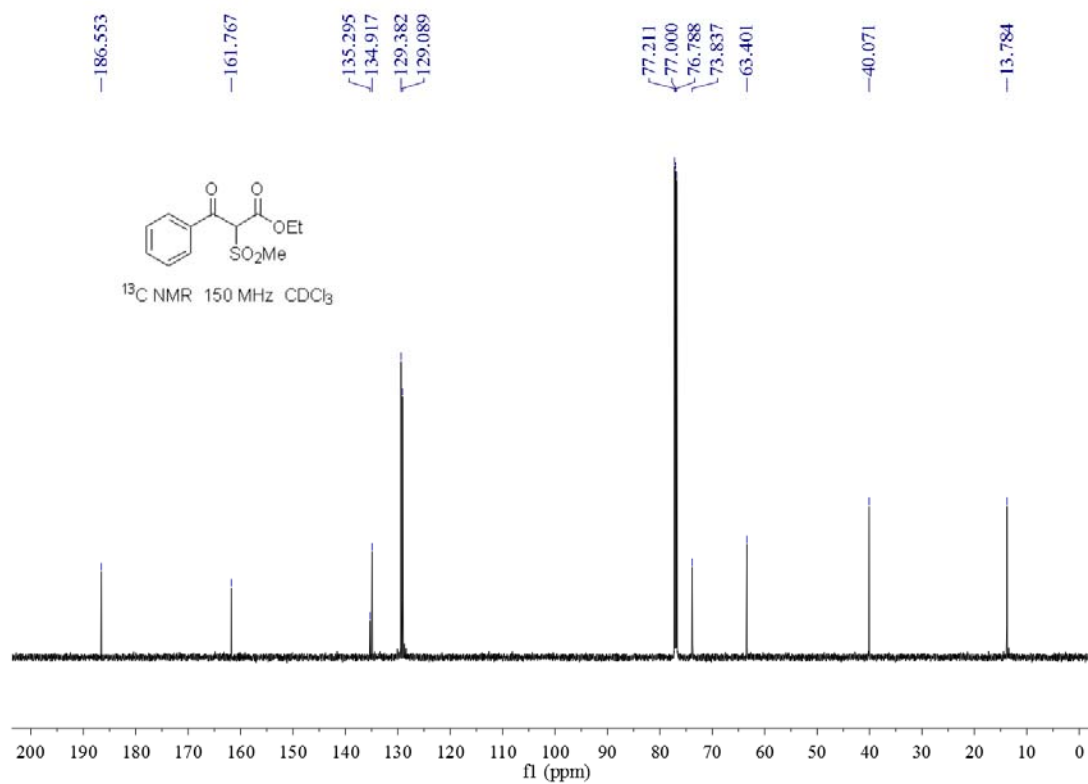
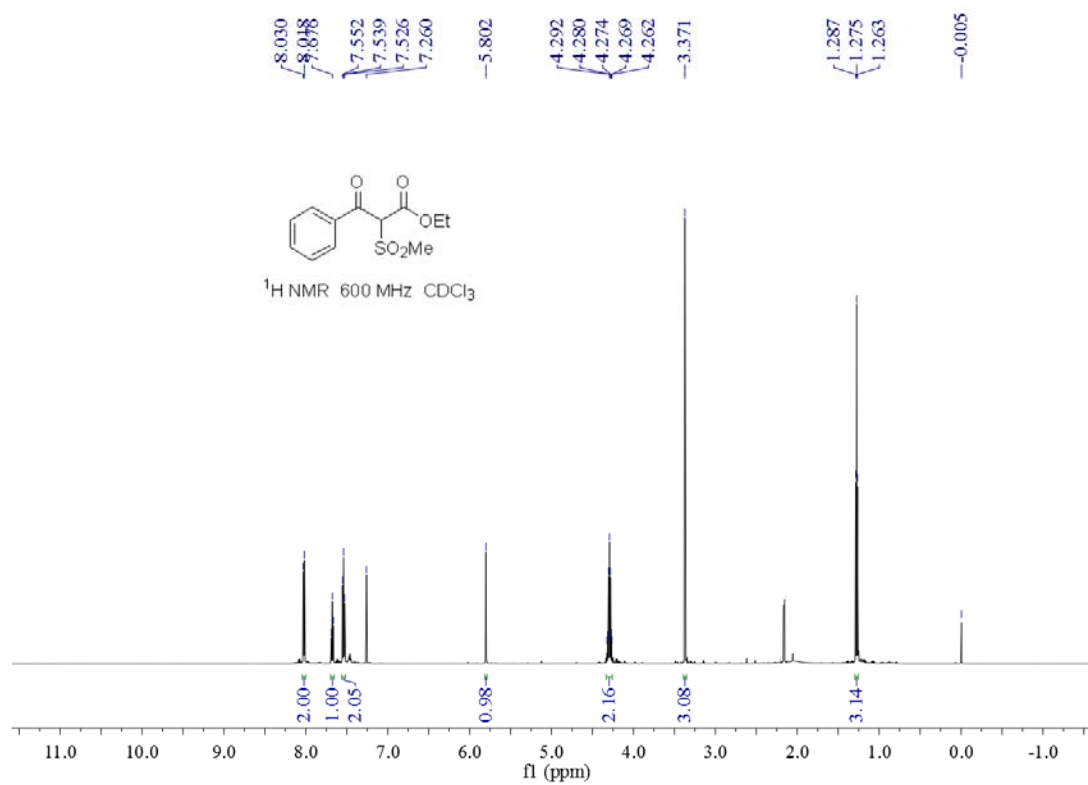
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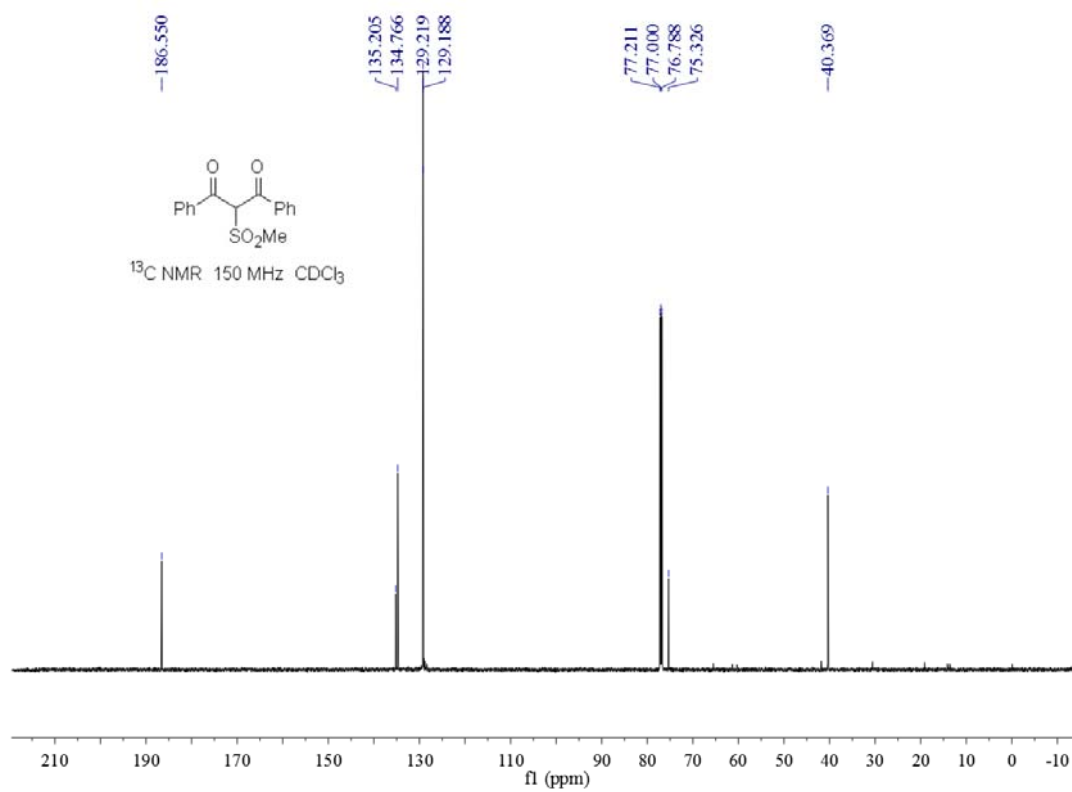
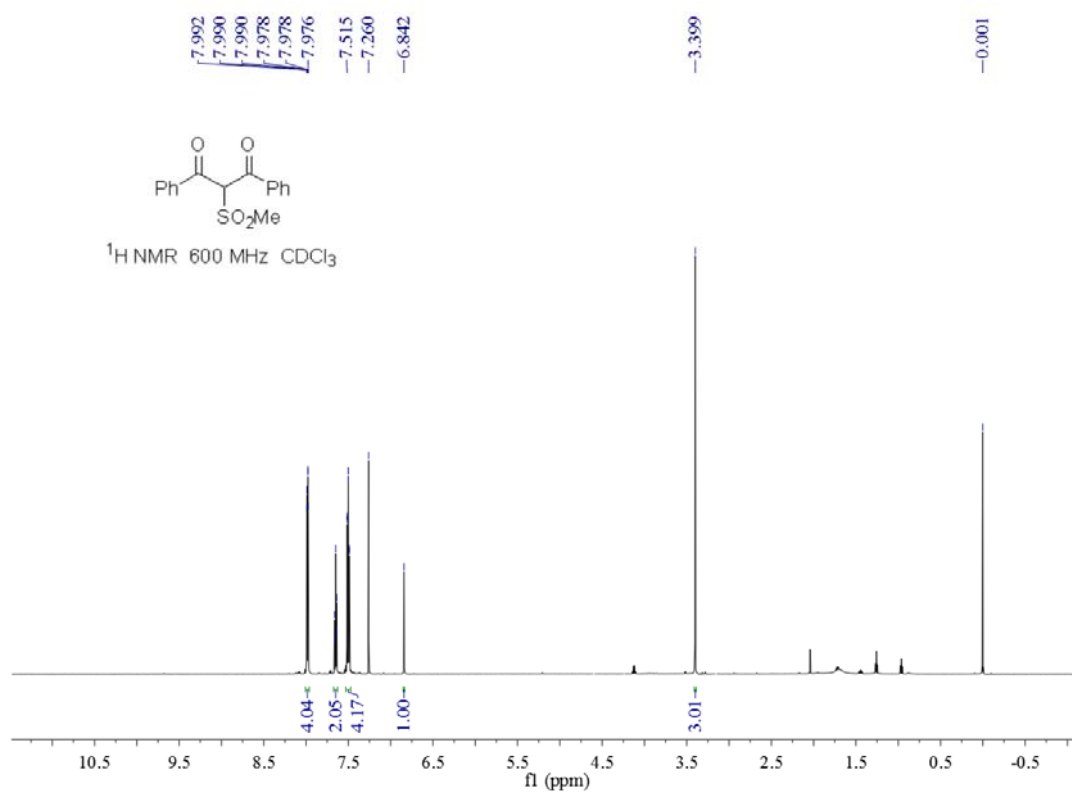
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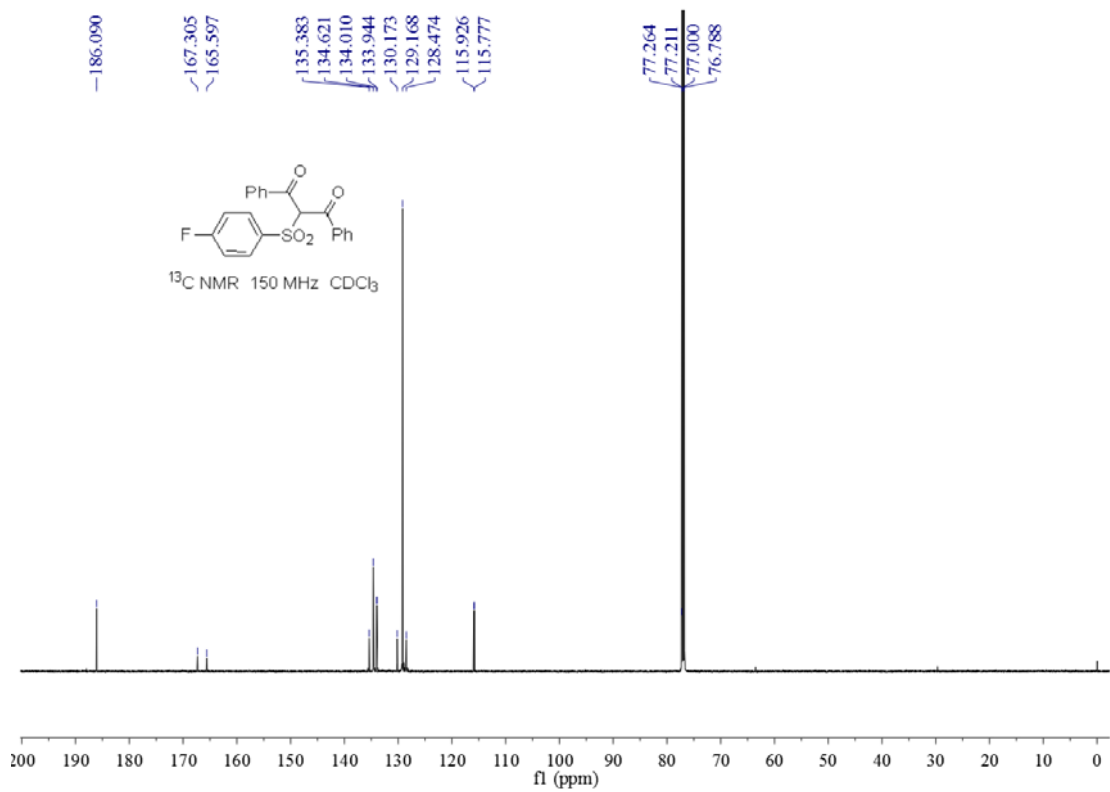
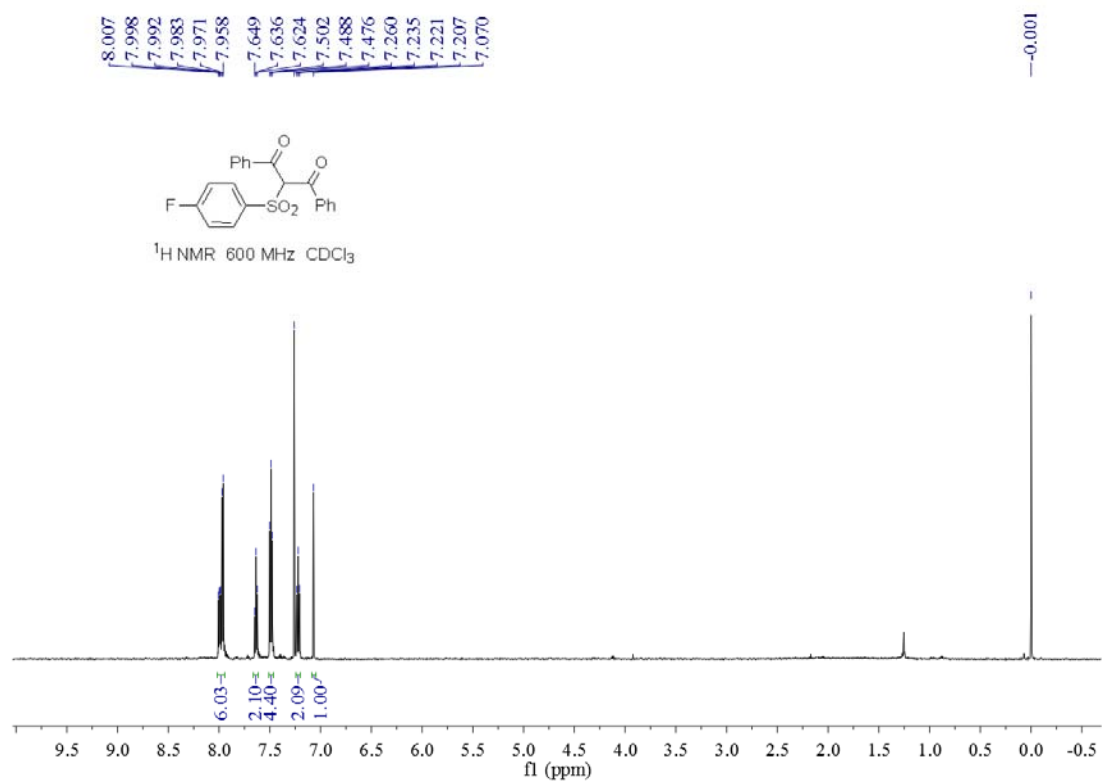
3q



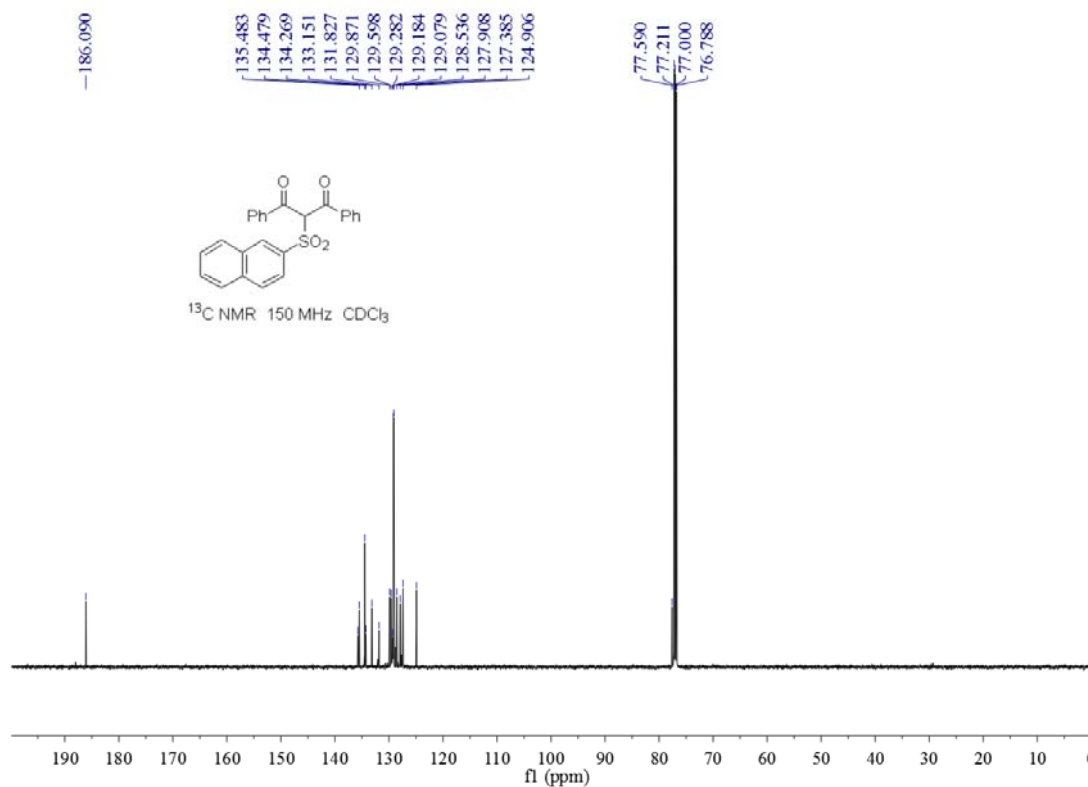
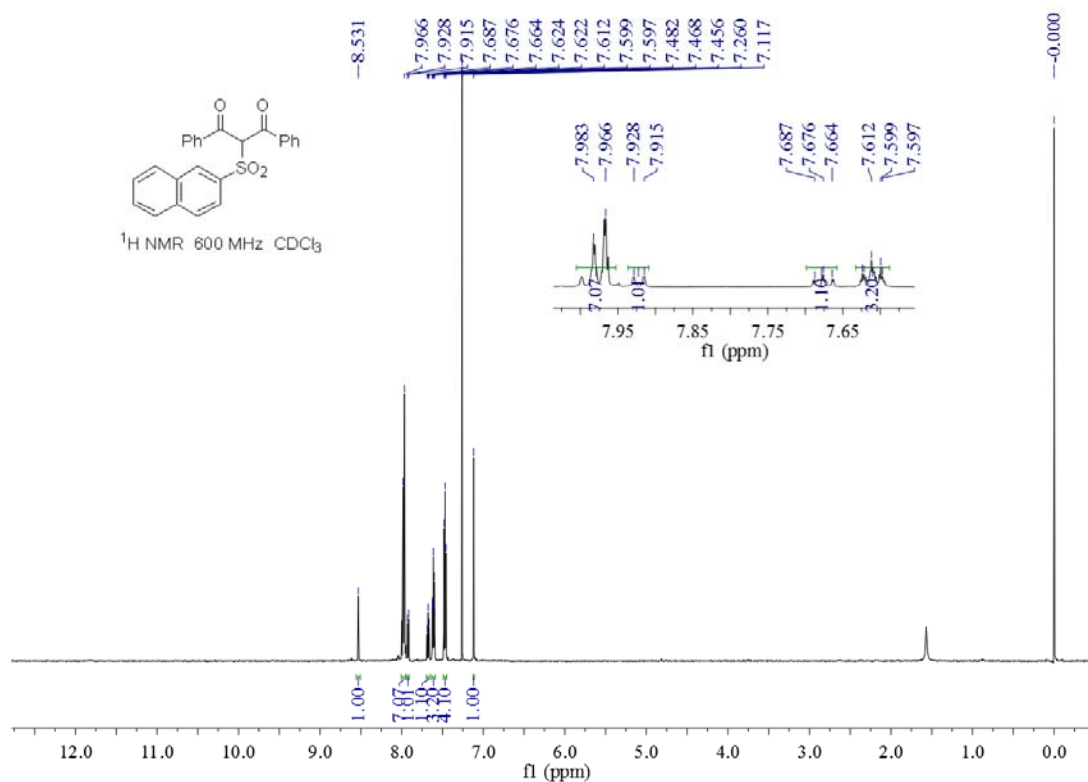
3r



3s



3t



3u

