

Supplementary Information

Hypoiodite-Catalyzed Oxidative Cyclisation of Michael Adducts of Chalcones with 1,3-Dicarbonyl compounds: A Facile and Versatile Approach to Substituted Furans and Cyclopropanes

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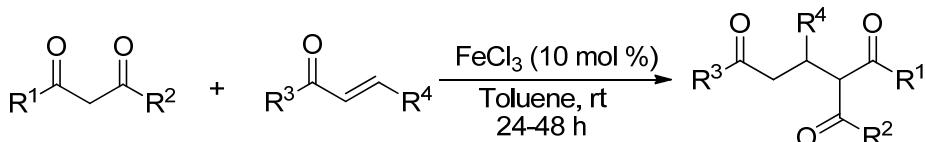
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General Information

¹H NMR spectra were recorded at 600 MHz or 400 MHz and ¹³C NMR spectra were measured at 150MHz or 100 MHz using Bruker AVANCE III NMR spectrometers with CDCl₃ as the solvent. Chemical shifts (δ) were measured in ppm and referenced to the deuterated chloroform (¹H: δ = 7.26 ppm, ¹³C: δ = 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 spectrometry (HRMS) was performed on a micrOTOF-Q II instrument with an ESI source. Melting points were measured with a RD-II type melting point apparatus. Fluorescence excitation and emission spectra were recorded by Cary Eclipse fluorescence spectrophotometer (Varian, America) with C = 1.5 × 10⁻⁵ mol/L. 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).

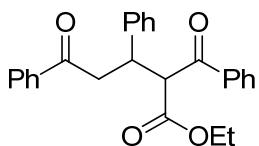
Preparation of Michael Adducts

General procedure:^{S1}



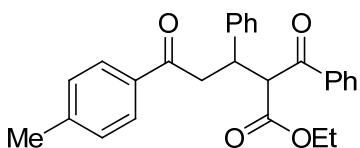
To a solution of chalcone (7.5 mmol) and 1,3-dicarbonyl compounds (5 mmol) in freshly distilled toluene (5 mL) was added FeCl₃ (0.5 mmol). After stirring at room temperature for 24-48 h, the mixture was diluted with H₂O (10 mL) and extracted with EtOAc (3 × 20 mL). The combined organic layers were dried (Na₂SO₄), concentrated in vacuo, and purified by column chromatography to gain Michael adducts **1**or **4**. All products were known compounds and structures have been confirmed by ¹H NMR.

Ethyl 2-benzoyl-5-oxo-3,5-diphenylpentanoate (1a)^{S2}



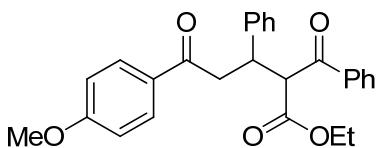
Yield: 55%; white solid; m.p. 139-141 °C; ¹H NMR (CDCl₃, 400 MHz, two isomers ratio 1:1): δ 8.09 (d, 1H, J = 8.0 Hz), 7.90-7.82 (m, 3H), 7.60 (t, 0.5H, J = 7.2 Hz), 7.55-7.37 (m, 5.5H), 7.31 (d, 1H, J = 8.0 Hz), 7.26-7.21 (m, 2H), 7.19-7.11 (m, 1.5H), 7.06 (t, 0.5H, J = 7.2 Hz), 4.92 (dd, 1H, J = 9.6, 4.0 Hz), 4.45-4.35(m, 1H), 4.20-4.10 (m, 1H), 3.87-3.77 (m, 1H), 3.62-3.52 (m, 1H), 3.48 (dd, 0.5 H, J = 16.0, 4.0 Hz), 3.31 (dd, 0.5 H, J = 16.0, 9.6 Hz), 1.16 (t, 1.5H, J = 7.2 Hz), 0.88 (t, 1.5 H, J = 7.2 Hz).

Ethyl 2-benzoyl-5-oxo-3-phenyl-5-(p-tolyl)pentanoate (1b)^{S3}



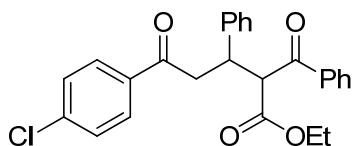
Yield: 60%; white solid; m.p. 87-89 °C; ¹H NMR (CDCl₃, 400 MHz, two isomers ratio 1:1): δ 8.09 (d, 1H, J = 8.0 Hz), 7.85 (d, 1H, J = 8.0 Hz), 7.79 (t, 2H, J = 8.8 Hz), 7.60 (t, 0.5H, J = 7.2 Hz), 7.55-7.45 (m, 1.5H), 7.39 (t, 1H, J = 7.6 Hz), 7.30 (d, 1H, J = 7.2 Hz), 7.26-7.18 (m, 4H), 7.16-7.10 (m, 1.5H), 7.08-7.02 (m, 0.5H), 4.93 (dd, 1H, J = 9.6, 4.0 Hz), 4.45-4.35(m, 1H), 4.20-4.10 (m, 1H), 3.87-3.77 (m, 1H), 3.45-3.40 (m, 1.5 H), 3.27 (dd, 0.5 H, J = 16.0, 9.6 Hz), 2.38 (s, 3H), 1.16 (t, 1.5H, J = 7.2 Hz), 0.87 (t, 1.5 H, J = 7.2 Hz).

Ethyl 2-benzoyl-5-(4-methoxyphenyl)-5-oxo-3-phenylpentanoate (1c)^{S3}



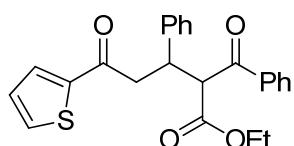
Yield: 60%, white solid; m.p. 102-104 °C; ¹H NMR (CDCl₃, 400 MHz, two isomers ratio 1:3): δ 8.09 (d, 1.4H, J = 7.2 Hz), 7.90-7.80 (m, 2.4H), 7.60 (t, 0.8H, J = 7.2 Hz), 7.55-7.45 (m, 1.7H), 7.39 (t, 0.7H, J = 7.6 Hz), 7.29 (d, 1.6H, J = 7.6 Hz), 7.23 (t, 2H, J = 7.2 Hz), 7.19-7.10 (m, 1.3H), 7.05 (t, 0.3H, J = 7.2 Hz), 6.87 (d, 2H, J = 8.8 Hz), 4.96-4.90 (m, 1H), 4.44-4.32 (m, 0.5H), 3.88-3.78 (m, 4.5H), 3.51-3.40 (m, 1.3H), 3.22 (dd, 0.7H, J = 16.0, 9.6 Hz), 1.16 (t, 0.8H, J = 7.2 Hz), 0.88 (t, 2.4 H, J = 7.2 Hz).

Ethyl 2-benzoyl-5-(4-chlorophenyl)-5-oxo-3-phenylpentanoate (1d)^{S3}



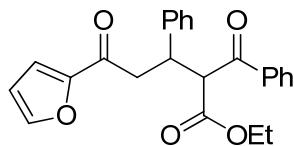
Yield: 44%; white solid; m.p. 94-96 °C; ¹H NMR (CDCl₃, 600 MHz, two isomers ratio 3:2): δ 8.10 (d, 1H, J = 7.2 Hz), 7.87-7.80 (m, 2.8H), 7.59 (t, 0.6H, J = 7.2 Hz), 7.52-7.46 (m, 1.6H), 7.39-7.34 (m, 2.8H), 7.30 (d, 1H, J = 7.2 Hz), 7.26-7.22 (m, 2H), 7.18-7.12 (m, 1.5H), 7.05 (t, 0.5H, J = 7.2 Hz), 4.93 (dd, 1H, J = 12.0, 9.6 Hz), 4.41-4.35 (m, 1H), 4.18-4.11 (m, 0.8H), 3.85-3.77 (m, 1.2H), 3.59 (dd, 0.4H, J = 10.2, 4.2 Hz), 3.53-3.42 (m, 1 H), 3.28 (dd, 0.6H, J=16.2, 10.2 Hz), 1.15 (t, 1.2H, J = 7.2 Hz), 0.86 (t, 1.8H, J=7.2 Hz).

Ethyl 2-benzoyl-5-oxo-3-phenyl-5-(thiophen-2-yl)pentanoate (1e)^{S3}



Yield: 44%; white solid; m.p. 118-120 °C; ¹H NMR (CDCl₃, 400 MHz, two isomers ratio 3:2): δ 8.10 (d, 1H, J = 8.0 Hz), 7.85 (d, 0.8H, J = 8.4 Hz), 7.76-7.71 (m, 1H), 7.63-7.55 (m, 1.7H), 7.53-7.47 (m, 1.6H), 7.39 (t, 1H, J = 7.6 Hz), 7.31 (d, 1.2H, J = 7.6 Hz), 7.23 (d, 1.6H, J = 7.2 Hz), 7.19-7.11 (m, 1.7H), 7.10-7.03 (m, 1.4H), 4.96 (dd, 1H, J = 10.0, 2.8 Hz), 4.42-4.32 (m, 1H), 4.15 (q, 1H, J = 6.8 Hz), 3.88-3.77 (m, 1H), 3.51-3.35 (m, 1.5H), 3.24-3.16 (m, 0.5 H), 1.17 (t, 1.2H, J = 7.2 Hz), 0.87 (t, 1.8 H, J = 7.2 Hz).

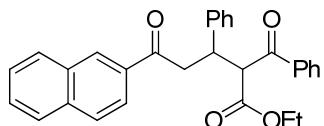
Ethyl 2-benzoyl-5-(furan-2-yl)-5-oxo-3-phenylpentanoate (1f)^{S3}



Yield: 59%; white solid; m.p. 150-152 °C; ¹H NMR (CDCl₃, 400 MHz): δ 8.10 (d, 2H, J = 7.6 Hz), 7.60 (t, 1H, J = 7.6 Hz), 7.49 (t, 3H, J = 7.6 Hz), 7.32 (d, 2H, J = 7.2 Hz), 7.23 (d, 2H, J = 7.6 Hz), 7.19-7.13 (m, 2H), 6.47-6.43 (m, 1H), 4.95 (d, 1H, J = 10.4

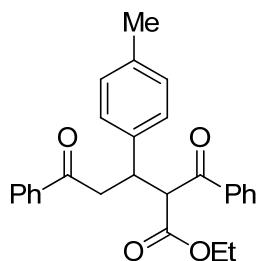
Hz), 4.44-4.32 (m, 1H), 3.87-3.76 (m, 2H), 3.29-3.15 (m, 2H), 0.87 (t, 3H, $J=7.2$ Hz).

Ethyl 2-benzoyl-5-(naphthalen-2-yl)-5-oxo-3-phenylpentanoate (1g)^{S3}



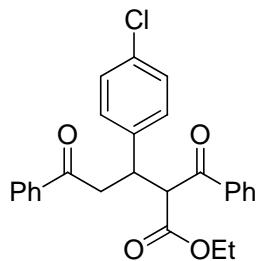
Yield: 59%; white solid; m.p.144-146 °C; ^1H NMR (CDCl_3 , 400 MHz, two isomers ratio 1:1): δ 8.45 (s, 1H), 8.13 (d, 1H, $J = 7.2$ Hz), 7.97-7.81 (m, 4.8 H), 7.64-7.48 (m, 4H), 7.40 (t, 1H, $J = 8.0$ Hz), 7.28 (d, 1H, $J = 8.0$ Hz), 7.23 (d, 1H, $J = 7.6$ Hz), 7.19-7.11 (m, 1.5H), 7.06 (m, 0.5H), 4.97 (dd, 1H, $J = 9.6, 4.80$ Hz), 4.54-4.42 (m, 1H), 4.22-4.14 (m, 1H), 3.88-3.79 (m, 1H), 3.73-3.58 (m, 1.4H), 3.43 (dd, 0.6 H, $J = 15.6, 9.6$ Hz), 1.18 (t, 1.2H, $J = 7.2$ Hz), 0.88 (t, 1.8H, $J=7.2$ Hz).

Ethyl 2-benzoyl-5-oxo-5-phenyl-3-(p-tolyl)pentanoate (1h)^{S3}



Yield: 55%; white solid; m.p.113-115 °C; ^1H NMR (CDCl_3 , 600 MHz, two isomers ratio 1:1): δ 8.09 (d, 1H, $J = 8.4$ Hz), 7.91-7.85 (m, 3H), 7.60 (t, 0.5H, $J = 7.8$ Hz), 7.54-7.47 (m, 2.5H), 7.44-7.38 (m, 3H), 7.18 (d, 1H, $J = 7.8$ Hz), 7.12 (d, 1H, $J = 7.8$ Hz), 7.04 (d, 1H, $J = 7.8$ Hz), 6.94 (d, 1H, $J = 8.4$ Hz), 4.90 (dd, 1H, $J = 12.0, 9.6$ Hz), 4.39-4.31 (m, 1H), 4.19-4.11 (m, 1H), 3.87-3.81 (m, 1H), 3.59-3.43 (m, 1H), 3.27 (dd, 0.5 H, $J = 15.6, 9.6$ Hz), 3.31 (dd, 0.5 H, $J=16.0, 9.6$ Hz), 2.26 (s, 1.5 H), 2.18 (s, 1.5 H), 1.16 (t, 1.5H, $J = 7.2$ Hz), 0.89 (t, 1.5 H, $J=7.2$ Hz).

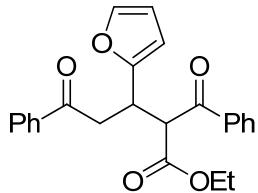
Ethyl 2-benzoyl-3-(4-chlorophenyl)-5-oxo-5-phenylpentanoate (1i)^{S3}



Yield: 45%; white solid; m.p.122-124 °C; ^1H NMR (CDCl_3 , 400 MHz, two isomers

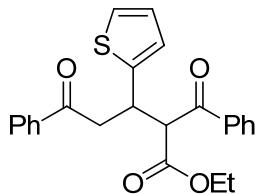
ratio 8:5): δ 8.09 (d, 1.2H, J = 8.0 Hz), 7.90-7.82 (m, 2.7H), 7.62 (t, 0.6H, J = 7.2 Hz), 7.56-7.48 (m, 2.65H), 7.45-7.38 (m, 2.65H), 7.29-7.26 (m, 1H), 7.23-7.17 (m, 2H), 7.11 (d, 0.8H, J = 8.4 Hz), 4.88 (dd, 1H, J = 10.4, 3.2 Hz), 4.43-4.32 (m, 1H), 4.20-4.12 (m, 0.8H), 3.90-3.80 (m, 1.2H), 3.59-3.42 (m, 1.4H), 3.27 (dd, 0.6 H, J = 16.0, 4.0 Hz), 3.31 (dd, 0.5 H, J = 16.0, 9.6 Hz), 1.16 (t, 1.15H, J = 7.2 Hz), 0.91 (t, 1.85 H, J = 7.2 Hz).

Ethyl 2-benzoyl-3-(furan-2-yl)-5-oxo-5-phenylpentanoate (1j)^{S3}



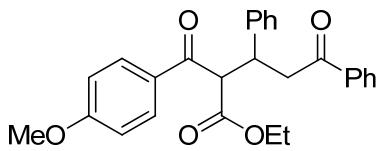
Yield: 35%; white solid; m.p. 58-60 °C; ¹H NMR (CDCl_3 , 600 MHz, two isomers ratio 7:3): δ 8.04 (d, 1.4H, J = 8.4 Hz), 7.96-7.91 (m, 2.6H), 7.59 (t, 0.7H, J = 7.2 Hz), 7.56-7.51 (m, 1.3H), 7.50-7.41 (m, 4H), 7.27 (dd, 0.7H, J = 1.8, 1.2 Hz), 7.13 (dd, 0.3H, J = 1.8, 1.2 Hz), 6.20 (dd, 0.7H, J = 3.6, 1.8 Hz), 6.12 (d, 0.7H, J = 3.0 Hz), 6.09 (dd, 0.3H, J = 3.6, 1.8 Hz), 6.02 (d, 0.3H, J = 3.0 Hz), 5.03 (d, 0.3H, J = 8.4 Hz), 4.98 (d, 0.7H, J = 8.4 Hz), 4.55-4.46 (m, 1H), 4.45-4.35 (m, 1H), 4.18-4.11 (m, 0.6H), 4.04-3.94 (m, 1.4H), 3.63-3.51 (m, 0.6H), 3.49-3.39 (m, 1.4H), 1.16 (t, 0.9H, J = 7.2 Hz), 0.88 (t, 2.1H, J = 7.2 Hz).

Ethyl 2-benzoyl-5-oxo-5-phenyl-3-(thiophen-2-yl)pentanoate (1k)^{S3}



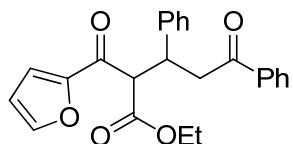
Yield: 36%; brown solid; m.p. 74-76°C; ¹H NMR (CDCl_3 , 400 MHz, two isomers ratio 7:5): δ 8.07 (d, 1.2H, J = 7.6 Hz), 7.95-7.88 (m, 2.8H), 7.62-7.48 (m, 3H), 7.47-7.38 (m, 3H), 7.11 (d, 0.56H, J = 5.2 Hz), 7.00 (d, 0.44H, J = 5.2 Hz), 6.92 (d, 0.58H, J = 3.6 Hz), 6.88-6.82 (m, 1H), 6.77-6.72 (m, 0.42H), 5.01 (dd, 1H, J = 14.4, 8.8 Hz), 4.78-4.68 (m, 1H), 4.19-4.08 (m, 1H), 3.98-3.88 (m, 1H), 3.64-3.52 (m, 1.4H), 3.46-3.37 (m, 0.6 H), 1.15 (t, 1.25H, J = 7.2 Hz), 0.88 (t, 1.75 H, J = 7.2 Hz).

Ethyl 2-(4-methoxybenzoyl)-5-oxo-3,5-diphenylpentanoate (1l)^{S3}



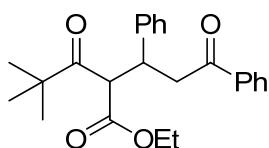
Yield: 54%; white solid; m.p. 105-107°C; ^1H NMR (CDCl_3 , 400 MHz, two isomers ratio 2:1): δ 8.09 (d, 0.65H, J = 8.8 Hz), 7.92-7.84 (m, 3.3H), 7.56-7.47 (m, 1H), 7.45-7.37 (m, 2H), 7.29 (d, 0.7H, J = 7.2 Hz), 7.26-7.20 (m, 2H), 7.19-7.11 (m, 1.65H), 7.05 (t, 0.66H, J = 7.2 Hz), 6.96 (d, 0.67H, J = 8.8 Hz), 6.87 (d, 1.33H, J = 8.8 Hz), 4.92-4.84 (m, 1H), 4.44-4.32 (m, 1H), 4.20-4.10 (m, 1.34H), 3.91-3.78 (m, 3.66H), 3.60-3.43 (m, 1.7H), 3.26 (dd, 0.3H, J = 16.0, 10.0 Hz), 1.17 (t, 2H, J = 7.2 Hz), 0.88 (t, 1H, J = 7.2 Hz).

Ethyl 2-(furan-2-carbonyl)-5-oxo-3,5-diphenylpentanoate (1m)^{S3}



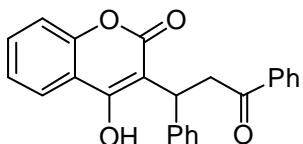
Yield: 59%; white solid; m.p. 119-121°C; ^1H NMR (CDCl_3 , 400 MHz, two isomers ratio 3:1): δ 7.88 (m, 2H), 7.63 (s, 0.7H), 7.55-7.48 (m, 1.4H), 7.45-7.35 (m, 2.8H), 7.31-7.26 (m, 1.6H), 7.26-7.20 (m, 1.8H), 7.18-7.12 (t, 1.4H, J = 7.2 Hz), 7.10-7.05 (m, 0.3H), 6.59-6.54 (m, 0.75H), 6.48-6.45 (m, 0.25), 4.71-4.63 (m, 1H), 4.41-4.31 (m, 1H), 4.21-4.12 (m, 0.6H), 3.93-3.83 (m, 1.4H), 3.62-3.45 (m, 1.3H), 3.38-3.30 (dd, 0.7 H, J = 16.0, 10.0 Hz), 1.18 (t, 0.8H, J = 7.2 Hz), 0.88 (t, 2.2 H, J = 7.2 Hz).

Ethyl 4,4-dimethyl-3-oxo-2-(3-oxo-1,3-diphenylpropyl)pentanoate (1n)^{S3}



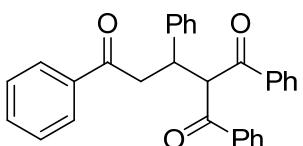
Yield: 47%; colorless oil; ^1H NMR (CDCl_3 , 400 MHz, two isomers ratio 2:1): δ 7.87 (d, 2H, J = 7.2 Hz), 7.52 (t, 1H, J = 7.2 Hz), 7.41 (t, 2H, J = 7.6 Hz), 7.25-7.11 (m, 5.5H), 4.36 (dd, 1H, J = 14.0, 9.6 Hz), 4.28-4.12 (m, 2.5 H), 3.84 (q, 0.6H, J = 7.2 Hz), 1.24 (t, 2H, J = 7.2 Hz), 1.15 (s, 2.5H), 0.93 (t, 1H, J = 7.2 Hz), 0.87 (s, 6.5H).

4-Hydroxy-3-(3-oxo-1,3-diphenylpropyl)-2H-chromen-2-one (1o)^{S4}



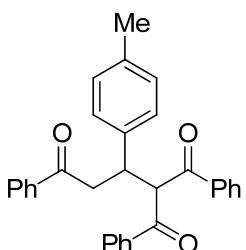
Yield: 56%; white solid; m.p. 162-164 °C; ^1H NMR (CDCl_3 , 600 MHz): δ 9.90 (brs, 1H), 8.09 (dd, 2H, J = 7.6, 1.2 Hz), 7.98 (dd, 1H, J = 7.8, 1.2 Hz), 7.63 (t, 1H, J = 7.2 Hz), 7.52-7.46 (m, 3H), 7.41 (d, 2H, J = 7.8 Hz), 7.33-7.27 (m, 3H), 7.25-7.20 (m, 2H), 4.96 (dd, 1H, J = 10.2, 1.8 Hz), 4.49 (dd, 1H, J = 19.2, 10.2 Hz), 3.80 (dd, 1H, J = 19.2, 2.4 Hz).

2-Benzoyl-1,3,5-triphenylpentane-1,5-dione (1p)^{S1}



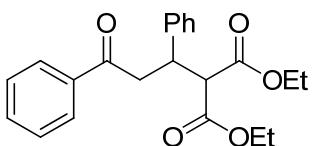
Yield: 65%; white solid; m.p. 154-156 °C; ^1H NMR (CDCl_3 , 400 MHz): δ 7.97 (d, 2H, J = 7.6 Hz), 7.88 (d, 2H, J = 7.6 Hz), 7.80 (d, 2H, J = 7.6 Hz), 7.58-7.40 (m, 7H), 7.34 (t, 2H, J = 7.6 Hz), 7.25 (d, 2H, J = 7.6 Hz), 7.15-7.02 (m, 3H), 5.91 (d, 1H, J = 8.4 Hz), 4.52-4.64 (m, 1H), 3.68-3.54 (m, 2H).

2-Benzoyl-1,5-diphenyl-3-(p-tolyl)pentane-1,5-dione (1q)^{S1}



Yield: 44%; white solid; m.p. 153-155 °C; ^1H NMR (CDCl_3 , 400 MHz): δ 7.98 (d, 2H, J = 8.8 Hz), 7.88 (d, 2H, J = 7.2 Hz), 7.81 (d, 2H, J = 7.6 Hz), 7.57-7.40 (m, 7H), 7.34 (t, 2H, J = 7.6 Hz), 7.13 (d, 2H, J = 8.0 Hz), 6.92 (d, 2H, J = 8.0 Hz), 5.88 (d, 1H, J = 8.4 Hz), 4.58-4.48 (m, 1H), 3.66-3.51 (m, 2H), 2.18 (s, 3H).

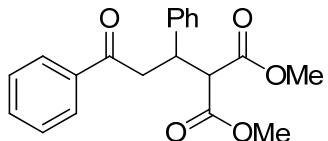
Diethyl 2-(3-oxo-1,3-diphenylpropyl)malonate (4a)^{S1}



Yield: 80%; white solid; m.p. 62-64 °C; ^1H NMR (CDCl_3 , 400 MHz): δ 7.90 (d, 2H, J = 8.0 Hz), 7.53 (t, 1H, J = 7.2 Hz), 7.42 (t, 2H, J = 7.2 Hz), 7.26-7.21 (m, 4H), 7.19-7.14

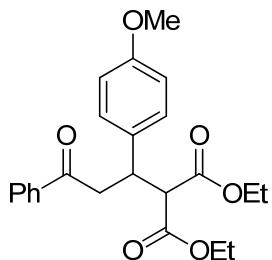
(m, 1H) 4.25-4.14 (m, 3H), 3.95 (q, 2H, $J = 7.2$ Hz), 3.82 (d, 1H, $J = 9.6$ Hz), 3.59-3.41 (m, 2H), 1.24 (t, 3H, $J = 7.2$ Hz), 1.00 (t, 3H, $J = 7.2$ Hz).

Dimethyl 2-(3-oxo-1,3-diphenylpropyl)malonate (4b**)^{S1}**



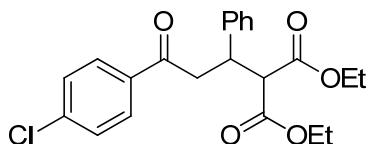
Yield: 75%; white solid; m.p. 103-105 °C; ^1H NMR (CDCl_3 , 400 MHz): δ 7.90 (d, 2H, $J = 8.0$ Hz), 7.53 (t, 1H, $J = 7.2$ Hz), 7.42 (t, 2H, $J = 7.2$ Hz), 7.26-7.21 (m, 4H), 7.19-7.14 (m, 1H) 4.23-4.16 (m, 3H), 3.86 (d, 1H, $J = 9.2$ Hz), 3.73 (s, 3H), 3.58-3.44 (m, 5H).

Diethyl 2-(1-(4-methoxyphenyl)-3-oxo-3-phenylpropyl)malonate (4c**)^{S1}**



Yield: 72%; white solid; m.p. 71-73 °C; ^1H NMR (CDCl_3 , 400 MHz): δ 7.89 (d, 2H, $J = 8.0$ Hz), 7.53 (t, 1H, $J = 7.6$ Hz), 7.42 (t, 2H, $J = 7.2$ Hz), 7.17 (d, 2H, $J = 8.8$ Hz), 6.77 (d, 2H, $J = 8.8$ Hz), 4.25-4.10 (m, 3H), 3.96 (q, 2H, $J = 7.2$ Hz), 3.78 (d, 1H, $J = 9.6$ Hz), 3.73 (s, 3H), 3.51 (d, 1H, $J = 14.4, 4.4$ Hz), 3.40 (d, 1H, $J = 14.4, 9.6$ Hz), 1.24 (t, 3H, $J = 7.2$ Hz), 1.03 (t, 3H, $J = 7.2$ Hz).

Diethyl 2-(3-(4-chlorophenyl)-3-oxo-1-phenylpropyl)malonate (4d**)^{S1}**



Yield: 87%; white solid; m.p. 62-65 °C; ^1H NMR (CDCl_3 , 600 MHz): δ 7.83 (d, 2H, $J = 8.4$ Hz), 7.39 (d, 2H, $J = 8.4$ Hz), 7.25-7.21 (m, 4H), 7.19-7.14 (m, 1H), 4.24-4.12 (m, 3H), 3.95 (q, 2H, $J = 7.2$ Hz), 3.80 (d, 1H, $J = 9.6$ Hz), 3.52 (dd, 1H, $J = 16.8, 4.8$ Hz), 3.40 (d, 1H, $J = 16.8, 9.6$ Hz), 1.24 (t, 3H, $J = 7.2$ Hz), 1.01 (t, 3H, $J = 7.2$ Hz).

General Procedure for the Synthesis of 2, 3 and 5

Synthesis of 2a: A 10 mL oven-dried reaction vessel was charged with Michael adduct of ethyl benzoylacetate with chalcone (**1a**, 40 mg, 0.1 mmol), DBU (31 mg, 0.2 mmol), and NIS (2.3 mg, 0.01 mmol) in 1,4-dioxane (2.0 mL) under air. TBHP (70% in water) (39 mg, 0.3 mmol) was added slowly to the sealed reaction vessel by syringe, and the resulting solution was stirred at 50 °C for 4 h. After the reaction was complete, sat. Na₂S₂O₃ aqueous 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 Na₂SO₄. After the removal of the solvent under vacuo, the residue was purified by flash column chromatography with PE/EtOAc (95 : 5) to give **2a** as white solid; yield: 26.4 mg (66%); m.p. 114-116 °C; TLC, *R*_f = 0.31 (PE:EtOAc = 95:5); ¹H NMR (CDCl₃, 400 MHz): δ 7.93-7.90 (m, 2H), 7.86 (d, 2H, *J* = 7.6 Hz), 7.51-7.45 (m, 4H), 7.40-7.30 (m, 7H), 4.12 (q, 2H, *J* = 7.2 Hz), 0.97 (t, 3H, *J* = 7.2 Hz); ¹³C NMR (CDCl₃, 100 MHz): δ 183.3, 163.8, 156.6, 146.2, 137.2, 135.9, 132.5, 131.2, 130.3, 129.6, 128.7, 128.6, 128.2, 128.1, 127.8, 117.5, 61.2, 13.5; HRMS (ESI) *m/z* calcd. for C₂₆H₂₁O₄ [M+H]⁺: 397.1434, found: 397.1438.

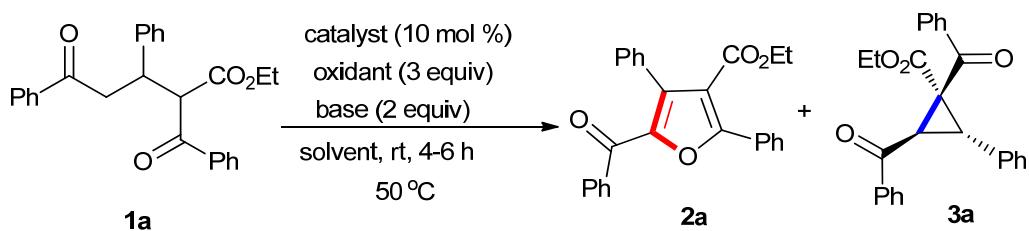
Synthesis of 3a: A 10 mL oven-dried reaction vessel was charged with Michael adduct of ethyl benzoylacetate with chalcone (**1a**, 40 mg, 0.1 mmol), TBAI (3.7 mg, 0.01 mmol) in THF (2.0 mL) under air. TBHP (5-6 M in decane) (39 mg, 0.3 mmol) was added slowly to the sealed reaction vessel by syringe, and the resulting solution was stirred at reflux for 2 h. After the reaction was complete, sat. Na₂S₂O₃ aqueous 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 Na₂SO₄. After the removal of the solvent under vacuo, the residue was purified by flash column chromatography with PE/EtOAc (95 : 5) to give **3a** as white solid; yield: 30.3 mg (76%); m.p. 138-140 °C; TLC, *R*_f = 0.29 (PE:EtOAc = 95:5); ¹H NMR (CDCl₃, 400 MHz): δ 8.05 (d, 2H, *J* = 7.2 Hz), 7.94 (d, 2H, *J* = 7.2 Hz), 7.60 (t, 1H, *J* = 7.2 Hz), 7.49 (t, 3H, *J* = 8.0 Hz), 7.46-7.42 (m, 2H), 7.35 (t, 4H, *J* = 8.0 Hz), 7.32-7.27(m, 1H), 4.56 (d, 1H, *J* =7.2 Hz), 4.02 (d, 1H, *J* = 7.2 Hz), 3.91-3.82 (m, 2H), 0.78 (t, 3H, *J* =7.2 Hz); ¹³C

NMR (CDCl_3 , 100 MHz): δ 194.0, 190.6, 166.8, 136.8, 136.6, 133.69, 133.66, 133.0, 128.8, 128.7, 128.52, 128.48, 128.4, 127.7, 62.0, 50.1, 36.8, 36.5, 13.6; HRMS (ESI) m/z calcd. for $\text{C}_{26}\text{H}_{23}\text{O}_4$ [$\text{M}+\text{H}]^+$: 399.1591, found: 399.1589.

Synthesis of **5a:**⁵⁵ A 10 mL oven-dried reaction vessel was charged with Michael adduct of ethyl malonate with chalcone (**4a**, 36.7 mg, 0.1 mmol), DBU (26 mg, 0.2 mmol), and NIS (2.3 mg, 0.01 mmol) in 1,4-dioxane (2.0 mL) under air. TBHP (70% in water) (26 mg, 0.2 mmol) was added slowly to the sealed reaction vessel by syringe, and the resulting solution was run at 50 °C for 2 h. After the reaction was complete, sat. $\text{Na}_2\text{S}_2\text{O}_3$ aqueous 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 **5a** as colorless oil; yield: 29 mg (94%). colorless oil; TLC, $R_f = 0.38$ (PE:EtOAc = 90:10); ^1H NMR (CDCl_3 , 400 MHz): δ 8.11 (d, 2H, $J = 7.6$ Hz), 7.62 (t, 1H, $J = 7.2$ Hz), 7.51 (t, 2H, $J = 7.2$ Hz), 7.32-7.26 (m, 5H), 4.17-4.10(m, 3H), 4.00 (q, 2H, $J = 6.8$ Hz), 3.80 (d, 1H, $J = 7.6$ Hz), 1.11 (t, 3H, $J = 7.2$ Hz), 0.98 (t, 3H, $J = 7.2$ Hz).

Table S1. Optimization of the reaction conditions for the synthesis of

$$2\mathbf{a}^a$$

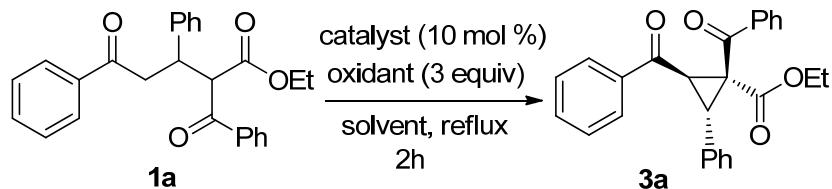


entry	catalyst	base	oxidant	solvent	yield (%) ^b	
					2a	3a
1	NIS	DBU	TBHP	THF	55	42
2	–	DBU	TBHP	THF	0	0
3	NIS	–	TBHP	THF	0	0
4	NIS	DBU	TBHP	EtOAc	47	51
5	NIS	DBU	TBHP	CHCl ₃	52	46
6	NIS	DBU	TBHP	1,4-dioxane	66	30
7	NIS	DBU	TBHP	1,4-dioxane/H ₂ O ^c	40	53
8 ^d	NIS	DBU	TBHP	1,4-dioxane	63	36
9	I ₂	DBU	TBHP	1,4-dioxane	52	41
10	TBAI	DBU	TBHP	1,4-dioxane	46	53
11	DIH	DBU	TBHP	1,4-dioxane	47	52
12	NIS	DBU	H ₂ O ₂	1,4-dioxane	19	17
13	NIS	DBU	CHP ^e	1,4-dioxane	65	33
14	NIS	DABCO	TBHP	1,4-dioxane	trace	19
15	NIS	K ₂ CO ₃	TBHP	1,4-dioxane	0	trace

^a Reaction condition: **1a** (0.1 mmol), catalyst (0.01 mmol), TBHP (0.3 mmol), base (0.2 mmol), solvent (2 mL), stirred at 50 °C for 4-6 h. ^b Isolated yields. ^c

V(1,4-dioxane):V(H₂O) = 3:1. ^d At room temperature, 19 h. ^e CHP = cumene hydrogen peroxide (80%-85% in water).

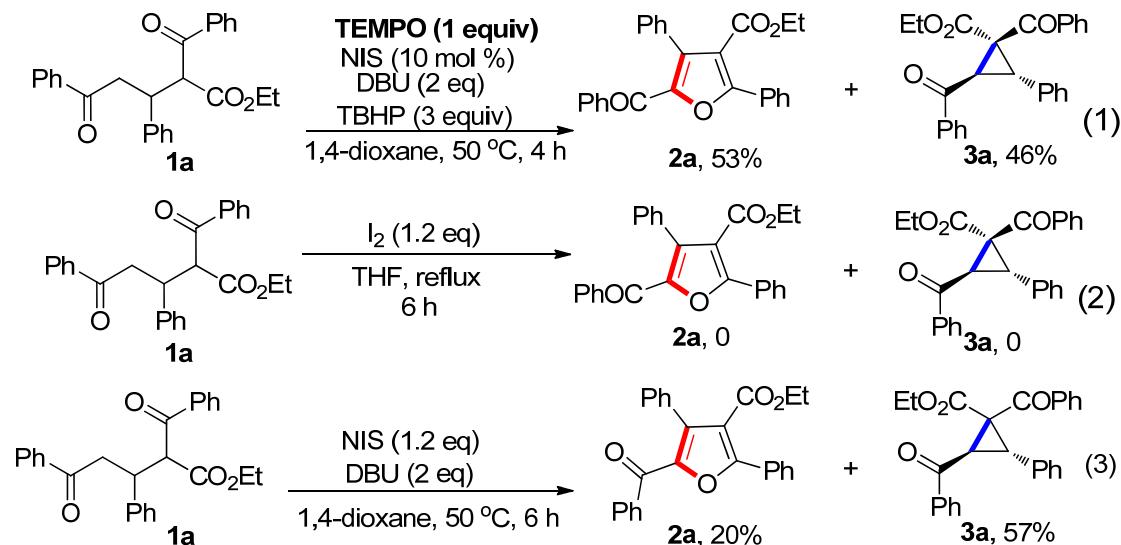
Table S2. Optimization of Reaction Conditions for the Synthesis of 3a^a



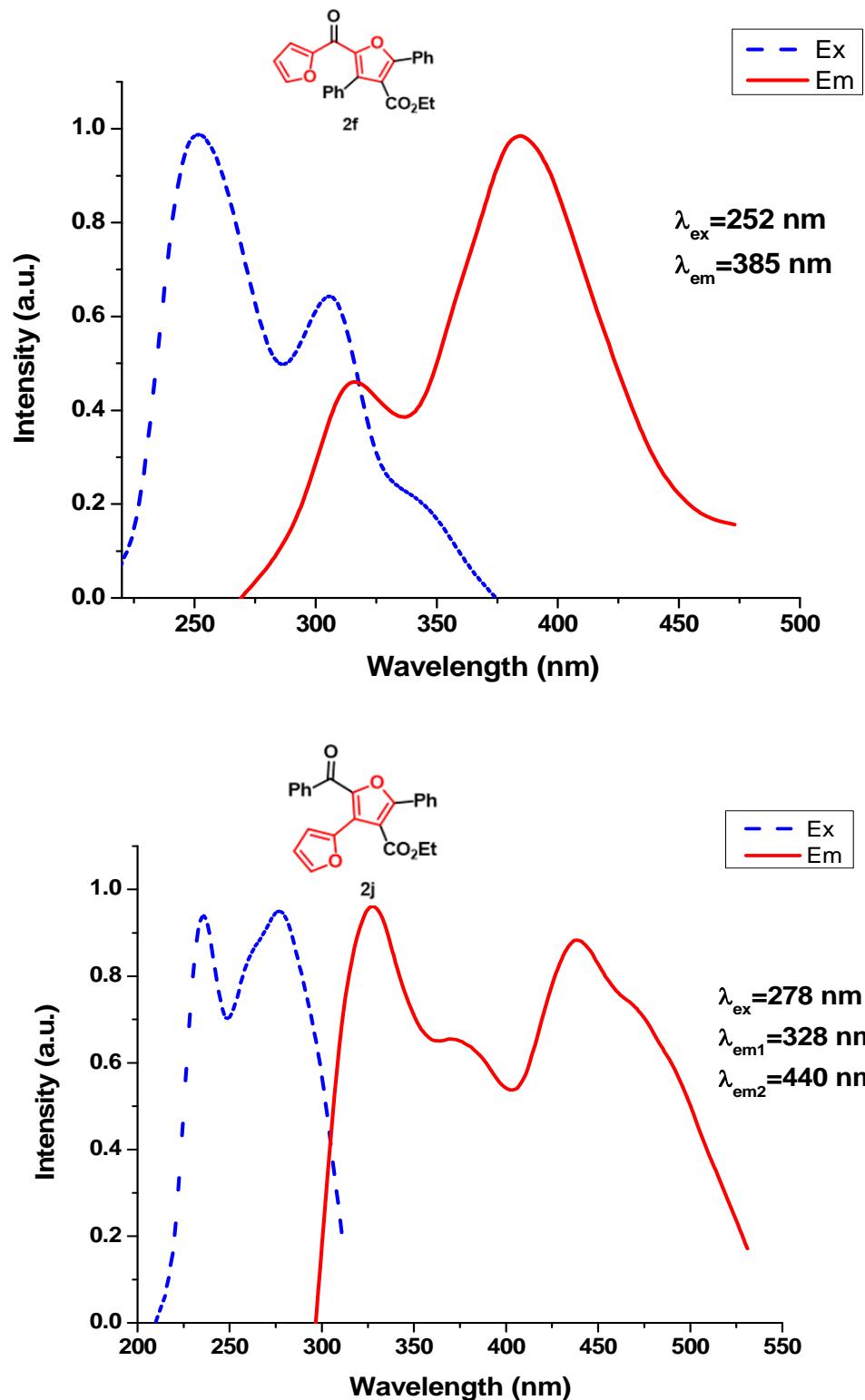
entry	catalyst	oxidant	solvent	yield (%) ^b	dr ^c
1	TBAI	TBHP	1,4-dioxane	45	> 19:1
2	TBAI	TBHP	DME	43	> 19:1
3	TBAI	TBHP	EtOAc	33	> 19:1
4	TBAI	TBHP	CH ₃ CN	29	> 19:1
5	TBAI	TBHP	DCE	28	> 19:1
6	TBAI	TBHP	THF	76	> 19:1
7	TBAI	TBPB	THF	N.R.	N.D.
8	TBAI	H ₂ O ₂	THF	trace	N.D.
9 ^d	TBAI	TBHP	THF	60	> 19:1
10 ^e	TBAI	TBHP	THF	53	> 19:1
11 ^f	TBAI	TBHP	THF	49	> 19:1

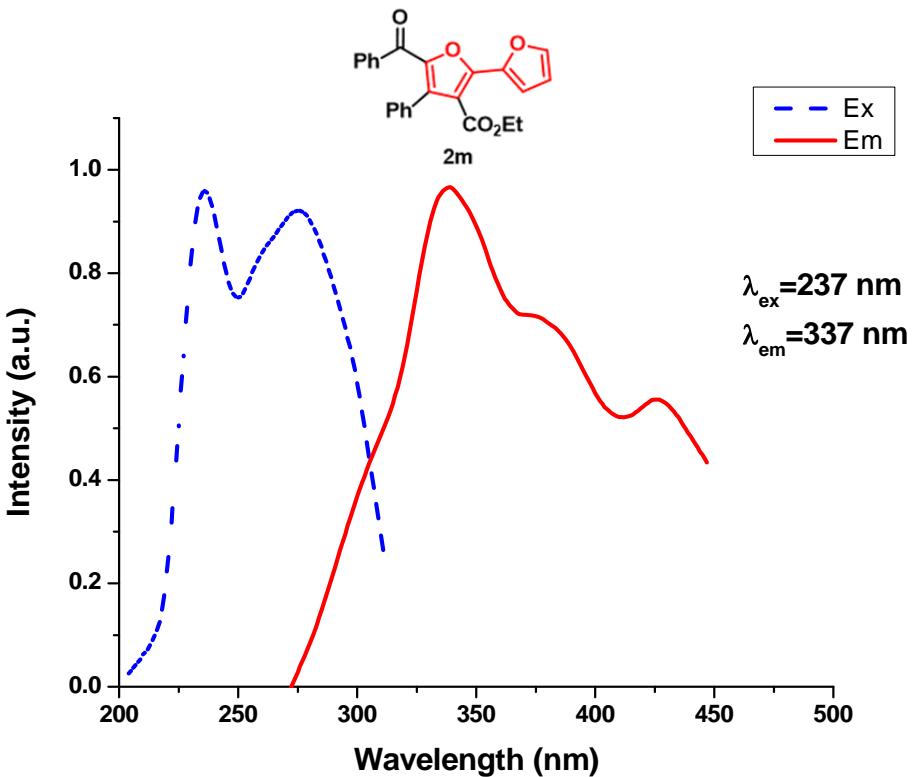
^a Reaction condition: **1a** (0.1 mmol), catalyst (0.01 mmol), TBHP (0.3 mmol) (5-6 M in decane), solvent (1 mL), stirred at reflux for 2 h. ^b Isolated yields. ^c Determined by ¹H NMR. ^d TBHP (70% in water) was used. ^e The reaction was run at 60 °C. ^f The reaction was run at 50 °C.

Experiments on Mechanism Study



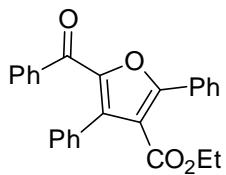
Fluorescence Spectra of 2f, 2j and 2m





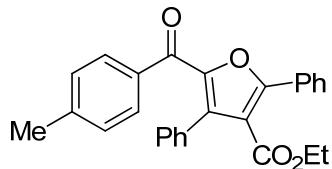
Characterization Data of Products

Ethyl 5-benzoyl-2,4-diphenylfuran-3-carboxylate (2a)



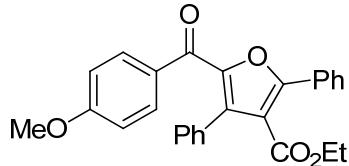
Yield: 26.4 mg (66%); time: 4 h; white solid; m.p. 114-116 °C; TLC, $R_f = 0.31$ (PE:EtOAc = 95:5); ^1H NMR (CDCl_3 , 400 MHz): δ 7.93-7.90 (m, 2H), 7.86 (d, 2H, $J = 7.6$ Hz), 7.51-7.45 (m, 4H), 7.40-7.30 (m, 7H), 4.12 (q, 2H, $J = 7.2$ Hz), 0.97 (t, 3H, $J = 7.2$ Hz); ^{13}C NMR (CDCl_3 , 100 MHz): δ 183.3, 163.8, 156.6, 146.2, 137.2, 135.9, 132.5, 131.2, 130.3, 129.6, 128.7, 128.6, 128.2, 128.1, 127.8, 117.5, 61.2, 13.5; HRMS (ESI) m/z calcd. for $\text{C}_{26}\text{H}_{21}\text{O}_4$ [$\text{M}+\text{H}]^+$: 397.1434, found: 397.1438.

Ethyl 5-(4-methylbenzoyl)-2,4-diphenylfuran-3-carboxylate (2b)



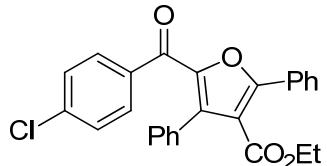
Yield: 25.4 mg (62%); time: 4 h; white solid; m.p. 152-154 °C; TLC, R_f = 0.33 (PE:EtOAc = 95:5); ^1H NMR (CDCl_3 , 400 MHz): δ 7.95-7.90 (m, 2H), 7.82 (d, 2H, J = 8.0 Hz), 7.50-7.45 (m, 3H), 7.40-7.36 (m, 2H), 7.35-7.31 (m, 3H), 7.19 (d, 2H, J = 8.4 Hz), 4.12 (q, 2H, J = 7.2 Hz), 2.39(s, 3H), 0.97 (t, 3H, J = 7.2 Hz); ^{13}C NMR (CDCl_3 , 100 MHz): δ 182.8, 163.9, 156.2, 143.4, 135.5, 134.5, 131.3, 130.2, 129.8, 129.5, 128.85, 128.77, 128.5, 128.01, 127.98, 127.7, 117.4, 61.2, 21.6, 13.5; HRMS (ESI) m/z calcd. for $\text{C}_{27}\text{H}_{23}\text{O}_4[\text{M}+\text{H}]^+$: 411.1591, found: 411.1600.

Ethyl 5-(4-methoxybenzoyl)-2,4-diphenylfuran-3-carboxylate (2c)



Yield: 21.7 mg (51%); time: 4 h; white solid; m.p. 95-97 °C; TLC, R_f = 0.16 (PE:EtOAc = 95:5); ^1H NMR (CDCl_3 , 400 MHz): δ 7.96-7.90 (m, 4H), 7.51-7.46 (m, 3H), 7.40-7.32 (m, 5H), 6.88 (d, 2H, J = 9.2 Hz), 4.12 (q, 2H, J = 7.2 Hz), 3.85(s, 3H), 0.97 (t, 3H, J = 7.2 Hz); ^{13}C NMR (CDCl_3 , 100 MHz): δ 181.7, 163.9, 163.2, 156.1, 146.5, 135.1, 132.1, 131.4, 130.1, 129.8, 129.6, 128.8, 128.5, 128.01, 127.98, 127.8, 117.3, 113.5, 61.1, 55.4, 13.5; HRMS (ESI) m/z calcd. for $\text{C}_{27}\text{H}_{23}\text{O}_5[\text{M}+\text{H}]^+$: 427.1540, found: 427.1539.

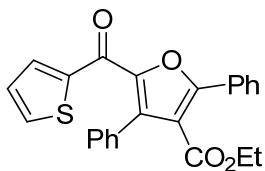
Ethyl 5-(4-chlorobenzoyl)-2,4-diphenylfuran-3-carboxylate (2d)



Yield: 22.8 mg (53%); time: 3 h; white solid; m.p. 79-81 °C; TLC, R_f = 0.36 (PE:EtOAc

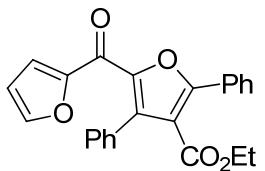
= 95:5); ^1H NMR (CDCl_3 , 600 MHz): δ 7.93-7.89 (m, 2H), 7.82 (d, 2H, J = 8.4 Hz), 7.50-7.46 (m, 3H), 7.37-7.32 (m, 7H), 4.12 (q, 2H, J = 7.2 Hz), 0.97 (t, 3H, J = 7.2 Hz); ^{13}C NMR (CDCl_3 , 150 MHz): δ 181.8, 163.7, 156.7, 145.9, 138.9, 136.3, 135.5, 131.0, 130.9, 130.4, 129.5, 128.8, 128.6, 128.5, 128.4, 128.3, 128.0, 127.8, 117.6, 61.2, 13.5; HRMS (ESI) m/z calcd. for $\text{C}_{26}\text{H}_{20}\text{ClO}_4$ [M+H] $^+$: 431.1045, found: 431.1040.

Ethyl 2,4-diphenyl-5-(thiophene-2-carbonyl)furan-3-carboxylate (2e)



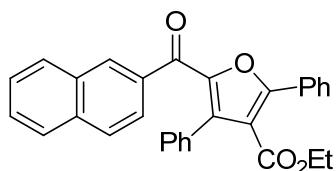
Yield: 25.8 mg (64%); time: 5.5 h; yellow solid; m.p. 101-104 °C; TLC, R_f = 0.23 (PE:EtOAc = 95:5); ^1H NMR (CDCl_3 , 400 MHz): δ 8.16 (d, 1H, J = 4.0 Hz), 8.01-7.96 (m, 2H), 7.71 (dd, 1H, J = 4.8, 0.8 Hz), 7.57-7.49 (m, 3H), 7.48-7.39 (m, 5H), 7.19 (t, 1H, J = 4.8 Hz), 4.11 (q, 2H, J = 7.2 Hz), 0.96 (t, 3H, J = 7.2 Hz); ^{13}C NMR (CDCl_3 , 100 MHz): δ 173.3, 163.7, 156.3, 145.5, 142.6, 136.1, 134.3, 134.1, 131.1, 130.3, 129.4, 128.7, 128.67, 128.2, 128.13, 128.10, 127.8, 118.0, 61.2, 13.5; HRMS (ESI) m/z calcd. for $\text{C}_{24}\text{H}_{19}\text{O}_4\text{S}$ [M+H] $^+$: 403.0999, found: 403.0998.

Ethyl 5-(furan-2-carbonyl)-2,4-diphenylfuran-3-carboxylate (2f)



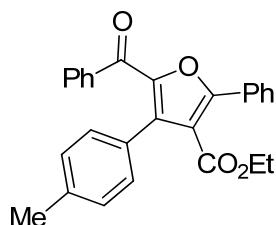
Yield: 23.2 mg (60%); time: 6 h; white solid; m.p. 116-119 °C; TLC, R_f = 0.25 (PE:EtOAc = 95:5); ^1H NMR (CDCl_3 , 400 MHz): δ 7.97-7.93 (m, 2H), 7.64 (d, 1H, J = 1.2 Hz), 7.55-7.48 (m, 4H), 7.47-7.36 (m, 5H), 6.58 (dd, 1H, J = 3.6, 1.6 Hz), 4.11 (q, 2H, J = 7.2 Hz), 0.96 (t, 3H, J = 7.2 Hz); ^{13}C NMR (CDCl_3 , 100 MHz): δ 168.9, 163.7, 156.4, 151.1, 147.2, 145.2, 135.9, 130.9, 130.3, 129.5, 128.8, 128.7, 128.2, 128.0, 127.7, 120.3, 117.8, 112.3, 61.2, 13.5. HRMS (ESI) m/z calcd. for $\text{C}_{24}\text{H}_{19}\text{O}_5$ [M+H] $^+$: 387.1227, found: 387.1224.

Ethyl 5-(2-naphthoyl)-2,4-diphenylfuran-3-carboxylate (2g)



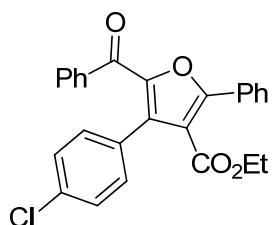
Yield: 24.8 mg (54%); time: 7 h; yellow solid; m.p. 96-99 °C; TLC, R_f = 0.30 (PE:EtOAc = 95:5); ^1H NMR (CDCl_3 , 400 MHz): δ 8.44 (s, 1H), 7.98-7.92 (m, 3H), 7.88-7.81 (m, 3H), 7.58 (t, 1H, J = 7.2 Hz), 7.52 (d, 1H, J = 8.0 Hz), 7.50-7.46 (m, 3H), 7.42-7.38 (m, 2H), 7.32-7.22 (m, 3H), 4.14 (q, 2H, J = 7.2 Hz), 0.99 (t, 3H, J = 7.2 Hz); ^{13}C NMR (CDCl_3 , 100 MHz): δ 183.1, 163.9, 156.5, 146.4, 135.8, 135.2, 134.4, 132.2, 131.7, 131.3, 130.3, 129.53, 129.47, 128.7, 128.6, 128.3, 128.1, 127.99, 127.97, 127.8, 127.7, 126.6, 125.2, 117.5, 61.2, 13.5; HRMS (ESI) m/z calcd. for $\text{C}_{30}\text{H}_{23}\text{O}_4$ [$\text{M}+\text{H}]^+$: 447.1591, found: 447.1597.

Ethyl 5-benzoyl-2-phenyl-4-(p-tolyl)furan-3-carboxylate (2h)



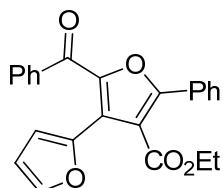
Yield: 21.7 mg (53%); time: 6.5 h; white solid; m.p. 126-128 °C; TLC, R_f = 0.38 (PE:EtOAc = 95:5); ^1H NMR (CDCl_3 , 600 MHz): δ 7.91-7.87 (m, 4H), 7.52-7.45 (m, 4H), 7.38 (t, 2H, J = 7.8 Hz), 7.27 (d, 2H, J = 7.8 Hz), 7.14 (d, 2H, J = 7.8 Hz), 4.15 (q, 2H, J = 7.2 Hz), 2.36 (s, 3H), 1.03 (t, 3H, J = 7.2 Hz); ^{13}C NMR (CDCl_3 , 100 MHz): δ 183.2, 163.9, 156.3, 146.2, 138.0, 137.3, 136.0, 132.4, 130.2, 129.6, 129.4, 128.8, 128.54, 128.49, 128.1, 128.0, 117.5, 61.2, 21.3, 13.6; HRMS (ESI) m/z calcd. for $\text{C}_{27}\text{H}_{23}\text{O}_4$ [$\text{M}+\text{H}]^+$: 411.1591, found: 411.1595.

Ethyl 5-benzoyl-4-(4-chlorophenyl)-2-phenylfuran-3-carboxylate (2i)



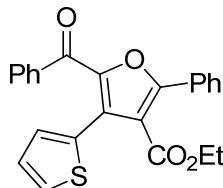
Yield: 29.4 mg (68%); time: 1.5 h; white solid; m.p. 131-133 °C; TLC, R_f = 0.34 (PE:EtOAc = 95:5); ^1H NMR (CDCl_3 , 600 MHz): δ 7.90 (d, 4H, J = 7.2 Hz), 7.54 (t, 1H, J = 7.2 Hz), 7.49-7.47 (m, 3H), 7.41 (t, 2H, J = 7.8 Hz), 7.33 (s, 4H), 4.14 (q, 2H, J = 7.2 Hz), 1.03 (t, 3H, J = 7.2 Hz); ^{13}C NMR (CDCl_3 , 100 MHz): δ 183.0, 163.5, 156.9, 146.3, 137.0, 134.7, 134.2, 132.7, 131.0, 130.4, 129.7, 129.6, 128.6, 128.2, 128.1, 128.0, 117.2, 61.3, 13.6; HRMS (ESI) m/z calcd. for $\text{C}_{26}\text{H}_{20}\text{ClO}_4$ [$\text{M}+\text{H}]^+$: 431.1045, found: 431.1047.

Ethyl 2'-benzoyl-5'-phenyl-[2,3'-bifuran]-4'-carboxylate (2j)



Yield: 20.4 mg (53%); time: 5 h; white solid; m.p. 91-94 °C; TLC, R_f = 0.32 (PE:EtOAc = 95:5); ^1H NMR (CDCl_3 , 400 MHz): δ 7.99-7.93 (m, 2H), 7.84-7.79 (m, 2H), 7.57 (t, 1H, J = 7.2 Hz), 7.51-7.43 (m, 6H), 7.41 (dd, 1H, J = 3.2, 0.4 Hz), 6.49 (dd, 1H, J = 3.1, 1.6 Hz), 4.38 (q, 2H, J = 7.2 Hz), 1.28 (t, 3H, J = 7.2 Hz); ^{13}C NMR (CDCl_3 , 100 MHz): δ 182.9, 164.6, 154.7, 145.2, 144.3, 143.2, 137.6, 132.5, 130.2, 129.5, 128.8, 128.4, 128.3, 127.3, 124.8, 116.1, 114.0, 111.8, 61.8, 14.0; HRMS (ESI) m/z calcd. for $\text{C}_{24}\text{H}_{19}\text{O}_5$ [$\text{M}+\text{H}]^+$: 387.1227, found: 387.1222.

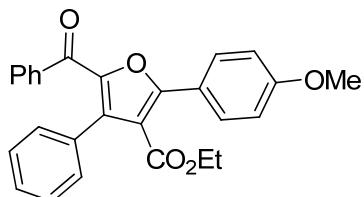
Ethyl 5-benzoyl-2-phenyl-4-(thiophen-2-yl)furan-3-carboxylate (2k)



Yield: 21.5 mg (53%); time: 5 h; yellow solid; m.p. 122-124 °C; TLC, R_f = 0.31 (PE:EtOAc = 95:5); ^1H NMR (CDCl_3 , 400 MHz): δ 7.90 (d, 2H, J = 7.2 Hz), 7.88-7.83 (m, 2H), 7.52 (t, 1H, J = 7.6 Hz), 7.49-7.45 (m, 3H), 7.44-7.36 (m, 3H), 7.22 (dd, 1H, J = 3.6, 1.2 Hz), 4.23 (q, 2H, J = 7.2 Hz), 1.13 (t, 3H, J = 7.2 Hz); ^{13}C NMR (CDCl_3 , 100 MHz): δ 183.1, 163.9, 155.9, 146.6, 137.1, 132.6, 130.7, 130.3, 129.6, 129.5, 128.6,

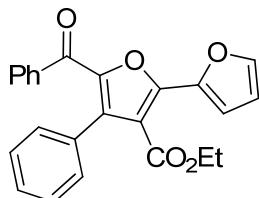
128.5, 128.2, 128.1, 127.8, 127.4, 126.7, 117.7, 61.6, 13.7; HRMS (ESI) m/z calcd. for $C_{24}H_{19}O_4S$ [M+H]⁺: 403.0999, found: 403.0999.

Ethyl 5-benzoyl-2-(4-methoxyphenyl)-4-phenylfuran-3-carboxylate (2l)



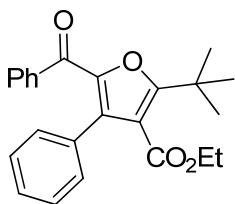
Yield: 23.5 mg (55%); time: 5 h; white solid; m.p. 155-157 °C; TLC, R_f = 0.16 (PE:EtOAc = 95:5); ¹H NMR ($CDCl_3$, 600 MHz): δ 7.91 (d, 2H, J = 9.0 Hz), 7.86-7.82 (m, 2H), 7.47 (t, 1H, J = 7.2 Hz), 7.38-7.30 (m, 7H), 6.99 (d, 2H, J = 9.0 Hz), 4.09 (q, 2H, J = 7.2 Hz), 3.87 (s, 3H), 0.95 (t, 3H, J = 7.2 Hz); ¹³C NMR ($CDCl_3$, 150 MHz): δ 183.2, 164.0, 161.3, 157.1, 145.9, 137.4, 136.1, 132.3, 131.6, 129.8, 129.6, 129.5, 128.04, 127.99, 127.7, 121.4, 116.3, 114.1, 61.0, 55.4, 13.5; HRMS (ESI) m/z calcd. for $C_{27}H_{23}O_5$ [M+H]⁺: 427.1540, found: 427.1534.

Ethyl 5-benzoyl-4-phenyl-[2,2'-bifuran]-3-carboxylate (2m)



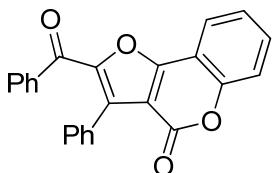
Yield: 20 mg (50%); time: 9 h; yellow oil; TLC, R_f = 0.25 (PE:EtOAc = 95:5); ¹H NMR ($CDCl_3$, 600 MHz): δ 7.89 (d, 2H, J = 7.2 Hz), 7.62 (d, 1H, J = 1.2 Hz), 7.50-7.45 (m, 2H), 7.36 (t, 2H, J = 7.8 Hz), 7.32-7.29 (m, 5H), 6.58 (dd, 1H, J = 3.6, 1.8 Hz), 4.13 (q, 2H, J = 7.2 Hz), 0.98 (t, 3H, J = 7.2 Hz); ¹³C NMR ($CDCl_3$, 150 MHz): δ 182.9, 162.8, 148.8, 146.1, 144.7, 143.8, 137.1, 135.6, 132.5, 131.3, 129.66, 129.64, 128.1, 128.0, 127.6, 115.6, 114.8, 112.2, 60.9, 13.6; HRMS (ESI) m/z calcd. for $C_{24}H_{19}O_5$ [M+H]⁺: 387.1227, found: 387.1223.

Ethyl 5-benzoyl-2-(tert-butyl)-4-phenylfuran-3-carboxylate (2n)



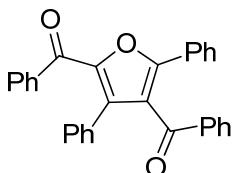
Yield: 15.5 mg (41%); time: 5 h; colorless oil; TLC, $R_f = 0.34$ (PE:EtOAc = 95:5); ^1H NMR (CDCl_3 , 400 MHz): δ 7.84 (d, 2H, $J = 7.2$ Hz), 7.47 (t, 1H, $J = 8.4$ Hz), 7.35 (t, 2H, $J = 7.6$ Hz), 7.33-7.26 (m, 5H), 4.09 (q, 2H, $J = 7.2$ Hz), 1.47 (s, 9H), 1.00 (t, 3H, $J = 7.2$ Hz); ^{13}C NMR (CDCl_3 , 100 MHz): δ 182.9, 165.8, 164.7, 144.5, 137.3, 135.3, 132.3, 131.4, 129.5, 129.3, 128.0, 127.7, 117.1, 61.1, 34.8, 28.6, 13.6; HRMS (ESI) m/z calcd. for $\text{C}_{24}\text{H}_{25}\text{O}_4[\text{M}+\text{H}]^+$: 377.1747, found: 377.1748.

2-Benzoyl-3-phenyl-4H-furo[3,2-c]chromen-4-one (2o)



Yield: 26 mg (71%); time: 1.5 h; white solid; m.p. 147-149 °C; TLC, $R_f = 0.08$ (PE:EtOAc = 95:5); ^1H NMR (CDCl_3 , 400 MHz): δ 8.04 (dd, 1H, $J = 8.0, 1.2$ Hz), 7.75 (d, 2H, $J = 7.6$ Hz), 7.63 (t, 1H, $J = 7.2$ Hz), 7.50-7.39 (m, 5H), 7.34-7.27 (m, 5H); ^{13}C NMR (CDCl_3 , 100 MHz): δ 184.0, 158.6, 156.8, 153.7, 147.9, 136.4, 133.1, 132.9, 132.5, 130.5, 129.6, 129.1, 128.14, 128.11, 127.8, 124.8, 122.0, 117.4, 112.0, 110.1; HRMS (ESI) m/z calcd. for $\text{C}_{24}\text{H}_{15}\text{O}_4 [\text{M}+\text{H}]^+$: 367.0965, found: 367.0969.

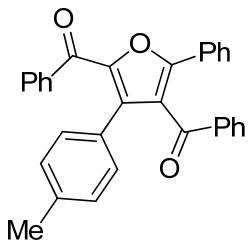
(3,5-Diphenylfuran-2,4-diyl)bis(phenylmethanone) (2p)



Yield: 35 mg (81%); time: 6 h; white solid; m.p. 70-72 °C; TLC, $R_f = 0.41$ (PE:EtOAc = 95:5); ^1H NMR (CDCl_3 , 400 MHz): δ 7.88 (d, 2H, $J = 7.6$ Hz), 7.77 (d, 2H, $J = 8.0$ Hz), 7.69-7.64 (m, 2H), 7.47 (t, 1H, $J = 7.2$ Hz), 7.41 (t, 1H, $J = 7.2$ Hz), 7.38-7.27 (m, 6H), 7.26-7.21 (m, 3H), 7.12-7.07 (m, 3H); ^{13}C NMR (CDCl_3 , 100 MHz): δ 192.7, 183.5,

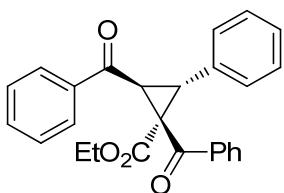
153.9, 145.8, 137.2, 136.8, 136.1, 133.8, 132.5, 130.3, 130.0, 129.71, 129.69, 129.65, 128.8, 128.5, 128.4, 128.3, 128.1, 127.9, 126.9, 123.7; HRMS (ESI) m/z calcd. for C₃₀H₂₁O₃ [M+H]⁺: 429.1485, found: 429.1475.

(5-Phenyl-3-(p-tolyl)furan-2,4-diyl)bis(phenylmethanone) (2q)



Yield: 34 mg (77%); time: 4 h; white solid; m.p. 73-75 °C; TLC, R_f = 0.36 (PE:EtOAc = 95:5); ¹H NMR (CDCl₃, 600 MHz): δ 7.94 (d, 2H, J = 7.2 Hz), 7.82 (t, 2H, J = 7.2 Hz), 7.66 (dd, 2H, J = 6.0, 3.6 Hz), 7.51 (t, 1H, J = 7.2 Hz), 7.45 (t, 1H, J = 7.2 Hz), 7.40 (t, 2H, J = 7.8 Hz), 7.34-7.28 (m, 5H), 7.18 (d, 2H, J = 7.8 Hz), 6.95 (d, 2H, J = 7.8 Hz), 2.21 (s, 3H); ¹³C NMR (CDCl₃, 150 MHz): δ 195.7, 186.3, 156.5, 148.8, 141.0, 140.4, 139.9, 139.1, 136.6, 135.3, 132.7, 132.6, 132.5, 131.7, 131.5, 131.4, 131.0, 130.2, 129.9, 126.8, 24.1; HRMS (ESI) m/z calcd. for C₃₁H₂₃O₃ [M+H]⁺: 443.1642, found: 443.1643.

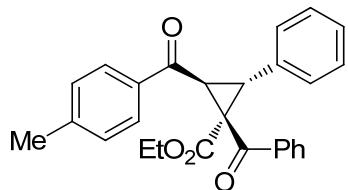
Ethyl 1,2-dibenzoyl-3-phenylcyclopropanecarboxylate (3a)



Yield: 30.3 mg (76%); time: 2 h; white solid; m.p. 138-140 °C; TLC, R_f = 0.29 (PE:EtOAc = 95:5); ¹H NMR (CDCl₃, 400 MHz): δ 8.05 (d, 2H, J = 7.2 Hz), 7.94 (d, 2H, J = 7.2 Hz), 7.60 (t, 1H, J = 7.2 Hz), 7.49 (t, 3H, J = 8.0 Hz), 7.46-7.42 (m, 2H), 7.35 (t, 4H, J = 8.0 Hz), 7.32-7.27(m, 1H), 4.56 (d, 1H, J = 7.2 Hz), 4.02 (d, 1H, J = 7.2 Hz), 3.91-3.82 (m, 2H), 0.78 (t, 3H, J = 7.2 Hz); ¹³C NMR (CDCl₃, 100 MHz): δ 194.0, 190.6, 166.8, 136.8, 136.6, 133.69, 133.66, 133.0, 128.8, 128.7, 128.52, 128.48, 128.4, 127.7, 62.0, 50.1, 36.8, 36.5, 13.6; HRMS (ESI) m/z calcd. for C₂₆H₂₃O₄ [M+H]⁺: 399.1591, found: 399.1589.

Ethyl 1-benzoyl-2-(4-methylbenzoyl)-3-phenylcyclopropanecarboxylate

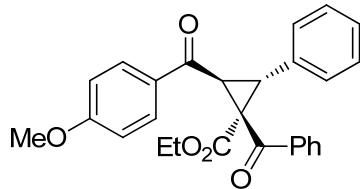
(3b)



Yield: 30.5 mg (74%); time: 2 h; white solid; m.p. 100-102 °C; TLC, $R_f = 0.31$ (PE:EtOAc = 95:5); ^1H NMR (CDCl_3 , 400 MHz): δ 7.94 (t, 4H, $J = 8.0$ Hz), 7.49-7.45 (m, 1H), 7.44-7.41 (m, 2H), 7.35 (t, 4H, $J = 8.0$ Hz), 7.31-7.27 (m, 3H), 4.54 (d, 1H, $J = 7.2$ Hz), 4.01 (d, 1H, $J = 7.2$ Hz), 3.90-3.80 (m, 2H), 2.43 (s, 3H), 0.77 (t, 3H, $J = 7.2$ Hz); ^{13}C NMR (CDCl_3 , 100 MHz): δ 193.5, 190.7, 166.8, 144.6, 136.6, 134.4, 133.8, 133.0, 129.4, 128.7, 128.5, 128.4, 127.6, 62.0, 49.9, 36.7, 36.5, 21.7, 13.6; HRMS (ESI) m/z calcd. for $\text{C}_{27}\text{H}_{25}\text{O}_4$ [M+H]⁺: 413.1747, found: 413.1739.

Ethyl 1-benzoyl-2-(4-methoxybenzoyl)-3-phenylcyclopropanecarboxylate

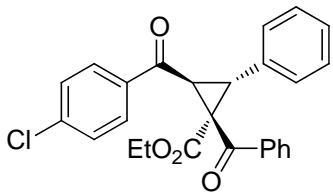
(3c)



Yield: 30 mg (70%); time: 1.5 h; white solid; m.p. 110-112 °C; TLC, $R_f = 0.14$ (PE:EtOAc = 95:5); ^1H NMR (CDCl_3 , 600 MHz): δ 8.04 (d, 2H, $J = 9.0$ Hz), 7.93 (dd, 2H, $J = 7.8, 1.8$ Hz), 7.46 (tt, 1H, $J = 7.2, 1.2$ Hz), 7.42 (d, 2H, $J = 7.2$ Hz), 7.37-7.33 (m, 4H), 7.28 (tt, 1H, $J = 7.2, 1.2$ Hz), 4.51 (d, 1H, $J = 7.8$ Hz), 4.01 (d, 1H, $J = 7.2$ Hz), 3.88 (s, 3H), 3.87-3.84 (m, 2H), 0.77 (t, 3H, $J = 7.2$ Hz); ^{13}C NMR (CDCl_3 , 150 MHz): δ 192.1, 190.7, 166.9, 164.0, 136.8, 134.0, 132.9, 130.9, 130.0, 128.7, 128.41, 128.39, 128.3, 127.6, 114.0, 61.9, 55.5, 49.9, 36.5, 36.4, 13.5. HRMS (ESI) m/z calcd. for $\text{C}_{27}\text{H}_{25}\text{O}_5$ [M+H]⁺: 429.1697, found: 429.1687.

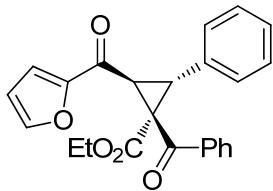
Ethyl 1-benzoyl-2-(4-chlorobenzoyl)-3-phenylcyclopropanecarboxylate

(3d)



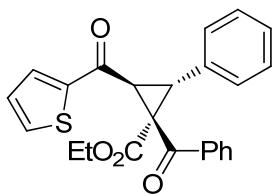
Yield: 30 mg (69%); time: 2 h; white solid; m.p. 151-153 °C; TLC, $R_f = 0.29$ (PE:EtOAc = 95:5); ^1H NMR (CDCl_3 , 600 MHz): δ 7.98 (d, 2H, $J = 9.0$ Hz), 7.92 (d, 2H, $J = 7.2$ Hz), 7.50-7.45 (m, 3H), 7.41 (d, 2H, $J = 7.2$ Hz), 7.38-7.34 (m, 4H), 7.30 (t, 1H, $J = 7.2$ Hz), 4.47 (d, 1H, $J = 7.2$ Hz), 4.01 (d, 1H, $J = 7.8$ Hz), 3.90-3.84 (m, 2H), 0.79 (t, 3H, $J = 7.2$ Hz); ^{13}C NMR (CDCl_3 , 150 MHz): δ 192.8, 190.4, 166.7, 140.3, 136.6, 135.3, 133.6, 133.1, 130.0, 129.1, 128.6, 128.5, 128.45, 128.42, 127.8, 62.1, 50.4, 36.8, 36.4, 13.6. HRMS (ESI) m/z calcd. for $\text{C}_{26}\text{H}_{22}\text{ClO}_4$ [M+H] $^+$: 433.1201, found: 433.1195.

Ethyl 1-benzoyl-2-(furan-2-carbonyl)-3-phenylcyclopropanecarboxylate (3e)



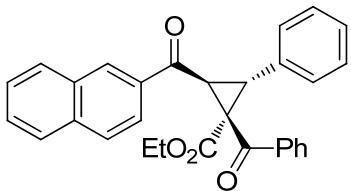
Yield: 23.7 mg (61%); time: 2 h; white solid; m.p. 107-109 °C; TLC, $R_f = 0.23$ (PE:EtOAc = 95:5); ^1H NMR (CDCl_3 , 600 MHz): δ 7.92 (dd, 2H, $J = 8.4, 1.2$ Hz), 7.68 (d, 1H, $J = 0.6$ Hz), 7.47 (t, 1H, $J = 7.2$ Hz), 7.42 (d, 2H, $J = 7.2$ Hz), 7.36 (t, 2H, $J = 7.8$ Hz), 7.34 (t, 2H, $J = 7.8$ Hz), 7.30-7.27 (m, 2H), 6.58 (dd, 1H, $J = 3.6, 1.2$ Hz), 4.47 (d, 1H, $J = 7.8$ Hz), 4.00 (d, 1H, $J = 7.8$ Hz), 3.91-3.83 (m, 2H), 0.79 (t, 3H, $J = 7.2$ Hz); ^{13}C NMR (CDCl_3 , 150 MHz): δ 190.1, 182.4, 166.6, 152.9, 147.1, 136.5, 133.8, 133.0, 128.8, 128.5, 128.4, 128.3, 127.6, 118.0, 112.7, 62.0, 49.8, 36.2, 36.1, 13.6. HRMS (ESI) m/z calcd. for $\text{C}_{24}\text{H}_{21}\text{O}_5$ [M+H] $^+$: 389.1384, found: 389.1375.

Ethyl 1-benzoyl-2-phenyl-3-(thiophene-2-carbonyl)cyclopropane carboxylate (3f)



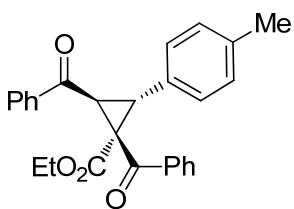
Yield: 23.7 mg (58%); time: 1.5 h; yellow solid; m.p. 86-88 °C; TLC, $R_f = 0.21$ (PE:EtOAc = 95:5); ^1H NMR (CDCl_3 , 400 MHz): δ 8.01 (d, 1H, $J = 4.2$ Hz), 7.93 (d, 2H, $J = 7.8$ Hz), 7.69 (d, 1H, $J = 5.4$ Hz), 7.47 (t, 1H, $J = 7.2$ Hz), 7.42 (d, 2H, $J = 7.2$ Hz), 7.38-7.32 (m, 4H), 7.29 (t, 1H, $J = 7.2$ Hz), 7.20 (t, 1H, $J = 4.2$ Hz), 4.40 (d, 1H, $J = 7.8$ Hz), 4.02 (d, 1H, $J = 7.2$ Hz), 3.90-3.83 (m, 2H), 0.79 (t, 3H, $J = 7.2$ Hz); ^{13}C NMR (CDCl_3 , 150 MHz): δ 190.2, 186.1, 166.7, 144.1, 136.6, 134.8, 133.7, 133.0, 132.9, 128.7, 128.6, 128.5, 128.4, 127.7, 62.0, 49.8, 37.2, 36.5, 13.6. HRMS (ESI) m/z calcd. for $\text{C}_{24}\text{H}_{21}\text{O}_4\text{S} [\text{M}+\text{H}]^+$: 405.1155, found: 405.1142.

Ethyl 2-(2-naphthoyl)-1-benzoyl-3-phenylcyclopropanecarboxylate (3g)



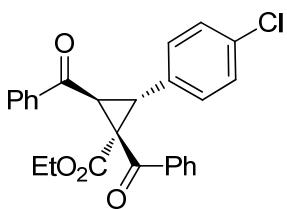
Yield: 27.8 mg (62%); time: 1.5 h; white solid; m.p. 88-90 °C; TLC, $R_f = 0.27$ (PE:EtOAc = 95:5); ^1H NMR (CDCl_3 , 600 MHz): δ 8.67 (s, 1H), 8.03 (d, 1H, $J = 7.8$ Hz), 8.00 (dd, 1H, $J = 9.0, 1.2$ Hz), 7.96 (d, 2H, $J = 7.6$ Hz), 7.89 (d, 2H, $J = 9.0$ Hz), 7.63 (t, 1H, $J = 7.2$ Hz), 7.59 (t, 1H, $J = 7.2$ Hz), 7.48 (d, 2H, $J = 7.8$ Hz), 7.44 (t, 1H, $J = 7.2$ Hz), 7.38 (t, 2H, $J = 7.2$ Hz), 7.35-7.30 (m, 3H), 4.72 (d, 1H, $J = 7.8$ Hz), 4.09 (d, 1H, $J = 7.8$ Hz), 3.94-3.86 (m, 2H), 0.81 (t, 3H, $J = 7.2$ Hz); ^{13}C NMR (CDCl_3 , 150 MHz): δ 193.8, 190.6, 166.9, 136.7, 135.9, 134.4, 133.9, 133.0, 132.5, 130.5, 130.0, 128.8, 128.7, 128.6, 128.5, 128.4, 127.8, 127.7, 127.0, 124.0, 62.0, 50.3, 36.9, 36.6, 13.6. HRMS (ESI) m/z calcd. for $\text{C}_{30}\text{H}_{25}\text{O}_4 [\text{M}+\text{H}]^+$: 449.1747, found: 449.1734.

Ethyl 1,2-dibenzoyl-3-(p-tolyl)cyclopropanecarboxylate (3h)



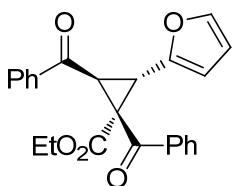
Yield: 32.5 mg (79%); time: 1 h; white solid; m.p. 97-99 °C; TLC, $R_f = 0.36$ (PE:EtOAc = 95:5); ^1H NMR (CDCl_3 , 600 MHz): δ 8.04 (dd, 2H, $J = 8.4, 1.2$ Hz), 7.93 (dd, 2H, $J = 8.4, 1.2$ Hz), 7.60 (t, 1H, $J = 7.2$ Hz), 7.50-7.45 (m, 3H), 7.35 (t, 2H, $J = 7.8$ Hz), 7.31 (d, 2H, $J = 8.4$ Hz), 7.15 (d, 2H, $J = 7.8$ Hz), 4.51 (d, 1H, $J = 7.2$ Hz), 3.98 (d, 1H, $J = 7.2$ Hz), 3.88 (q, 2H, $J = 7.2$ Hz), 2.34 (s, 3H), 0.80 (t, 3H, $J = 7.2$ Hz); ^{13}C NMR (CDCl_3 , 150 MHz): δ 194.0, 190.8, 166.9, 137.4, 137.0, 136.8, 133.6, 132.9, 130.7, 129.1, 128.7, 128.5, 128.4, 61.9, 50.2, 36.8, 36.7, 21.1, 13.6. HRMS (ESI) m/z calcd. for $\text{C}_{27}\text{H}_{25}\text{O}_4$ [M+H] $^+$: 413.1747, found: 417.1730.

Ethyl 1,2-dibenzoyl-3-(4-chlorophenyl)cyclopropanecarboxylate (3i)



Yield: 31.1 mg (72%); time: 1 h; white solid; m.p. 119-121 °C; TLC, $R_f = 0.32$ (PE:EtOAc = 95:5); ^1H NMR (CDCl_3 , 600 MHz): δ 8.02 (dd, 2H, $J = 7.2, 1.2$ Hz), 7.90 (dd, 2H, $J = 7.2, 1.2$ Hz), 7.61 (tt, 1H, $J = 7.2, 1.2$ Hz), 7.51-7.45 (m, 3H), 7.38-7.31 (m, 6H), 4.52 (d, 1H, $J = 7.8$ Hz), 3.98 (d, 1H, $J = 7.2$ Hz), 3.89 (q, 2H, $J = 7.2$ Hz), 0.81 (t, 3H, $J = 7.2$ Hz); ^{13}C NMR (CDCl_3 , 100 MHz): δ 193.6, 190.2, 166.6, 136.7, 136.4, 133.8, 133.6, 133.1, 132.3, 130.0, 128.8, 128.6, 128.5, 128.3, 62.2, 50.1, 36.5, 35.9, 13.6. HRMS (ESI) m/z calcd. for $\text{C}_{26}\text{H}_{22}\text{ClO}_4$ [M+H] $^+$: 433.1201, found: 433.1200.

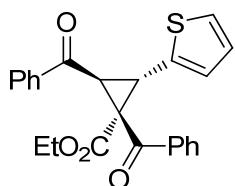
Ethyl 1,2-dibenzoyl-3-(furan-2-yl)cyclopropanecarboxylate (3j)



Yield: 20.2 mg (52%); time: 1 h; yellow oil; TLC, $R_f = 0.30$ (PE:EtOAc = 95:5); ^1H

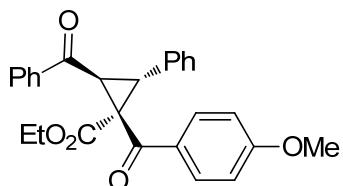
NMR (CDCl_3 , 600 MHz): δ 8.04 (d, 2H, $J = 7.2$ Hz), 7.96 (d, 2H, $J = 7.8$ Hz), 7.60 (t, 1H, $J = 7.2$ Hz), 7.51-7.47 (m, 3H), 7.41-7.36 (m, 3H), 6.38-6.33 (m, 2H), 4.43 (d, 1H, $J = 7.2$ Hz), 4.05-3.97 (m, 2H), 3.88 (d, 1H, $J = 7.2$ Hz), 0.93 (t, 3H, $J = 7.2$ Hz); ^{13}C NMR (CDCl_3 , 100 MHz): δ 193.3, 190.3, 166.6, 148.4, 142.3, 136.8, 136.6, 133.7, 133.1, 128.74, 128.67, 128.6, 128.5, 110.7, 108.5, 62.2, 49.0, 36.6, 29.5, 13.6. HRMS (ESI) m/z calcd. for $\text{C}_{24}\text{H}_{21}\text{O}_5$ [$\text{M}+\text{H}]^+$: 389.1384, found: 389.1381.

Ethyl 1,2-dibenzoyl-3-(thiophen-2-yl)cyclopropanecarboxylate (3k)



Yield: 25.4 mg (63%); time: 2 h; white solid; m.p. 151-153 °C; TLC, $R_f = 0.29$ (PE:EtOAc = 95:5); ^1H NMR (CDCl_3 , 400 MHz): δ 8.04 (d, 2H, $J = 8.0$ Hz), 7.94 (d, 2H, $J = 8.0$ Hz), 7.61 (t, 1H, $J = 7.2$ Hz), 7.49 (t, 3H, $J = 7.2$ Hz), 7.38 (t, 2H, $J = 7.2$ Hz), 7.24 (d, 1H, $J = 5.2$ Hz), 7.10-7.05 (m, 1H), 7.01-6.96 (m, 1H), 4.48 (d, 1H, $J = 7.2$ Hz), 4.05 (d, 1H, $J = 7.2$ Hz), 3.99-3.91 (m, 2H), 0.87 (t, 3H, $J = 7.2$ Hz); ^{13}C NMR (CDCl_3 , 100 MHz): δ 193.4, 190.4, 166.5, 136.6, 136.5, 133.8, 133.1, 128.8, 128.6, 128.5, 128.4, 126.9, 126.8, 125.2, 62.2, 50.4, 38.4, 31.6, 13.6. HRMS (ESI) m/z calcd. for $\text{C}_{24}\text{H}_{21}\text{O}_4\text{S}$ [$\text{M}+\text{H}]^+$: 405.1155, found: 405.1156.

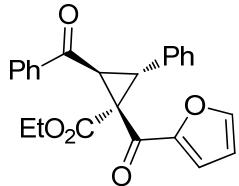
Ethyl 2-benzoyl-1-(4-methoxybenzoyl)-3-phenylcyclopropanecarboxylate (3l)



Yield: 28.2 mg (66%); time: 1.5 h; white solid; m.p. 116-118 °C; TLC, $R_f = 0.14$ (PE:EtOAc = 95:15); ^1H NMR (CDCl_3 , 400 MHz): δ 8.05 (d, 2H, $J = 7.2$ Hz), 7.92 (dt, 2H, $J = 9.2, 2.4$ Hz), 7.60 (tt, 1H, $J = 7.2, 1.2$ Hz), 7.49 (t, 2H, $J = 7.2$ Hz), 7.42 (d, 2H, $J = 6.8$ Hz), 7.34 (t, 2H, $J = 7.2$ Hz), 7.29 (dt, 1H, $J = 7.2, 1.2$ Hz), 6.82 (d, 2H, $J = 8.8$ Hz), 4.53 (d, 1H, $J = 7.2$ Hz), 4.01 (d, 1H, $J = 7.2$ Hz), 3.92-3.85 (m, 2H), 3.80 (s, 3H),

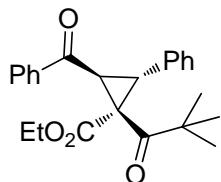
0.81 (t, 3H, $J = 7.2$ Hz); ^{13}C NMR (CDCl_3 , 100 MHz): δ 194.0, 188.9, 167.0, 163.4, 137.0, 133.9, 133.6, 130.7, 129.6, 128.7, 128.66, 128.5, 128.3, 127.6, 113.7, 62.0, 55.4, 50.4, 36.6, 36.0, 13.7. HRMS (ESI) m/z calcd. for $\text{C}_{27}\text{H}_{25}\text{O}_5$ [$\text{M}+\text{H}]^+$: 429.1697, found: 436.1689.

Ethyl 2-benzoyl-1-(furan-2-carbonyl)-3-phenylcyclopropanecarboxylate (3m)



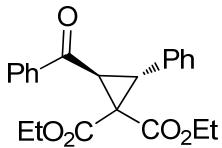
Yield: 19.4 mg (50%); time: 6 h; white solid; m.p. 92-94 °C; TLC, $R_f = 0.23$ (PE:EtOAc = 90:10); ^1H NMR (CDCl_3 , 600 MHz): δ 8.06 (dt, 2H, $J = 7.8, 1.2$ Hz), 7.59 (tt, 1H, $J = 9.0, 1.2$ Hz), 7.48 (t, 2H, $J = 7.8$ Hz), 7.42-7.38 (m, 3H), 7.33 (t, 2H, $J = 7.2$ Hz), 7.28 (t, 1H, $J = 7.2$ Hz), 7.21 (dd, 1H, $J = 3.6, 0.6$ Hz), 6.43 (dd, 1H, $J = 3.6, 1.8$ Hz), 4.44 (d, 1H, $J = 7.2$ Hz), 4.07 (d, 1H, $J = 7.2$ Hz), 3.97-3.92 (m, 2H), 0.88 (t, 3H, $J = 7.2$ Hz); ^{13}C NMR (CDCl_3 , 150 MHz): δ 193.8, 179.2, 166.2, 152.7, 146.0, 137.0, 133.8, 133.5, 128.8, 128.6, 128.5, 128.3, 127.6, 117.2, 112.4, 61.9, 49.6, 36.2, 35.7, 13.8. HRMS (ESI) m/z calcd. for $\text{C}_{24}\text{H}_{21}\text{O}_5$ [$\text{M}+\text{H}]^+$: 389.1384, found: 389.1373.

Ethyl 2-benzoyl-3-phenyl-1-pivaloylcyclopropanecarboxylate (3n)



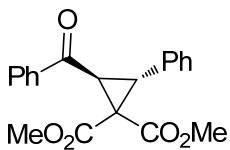
Yield: 20 mg (53%); time: 4 h; colorless oil; TLC, $R_f = 0.32$ (PE:EtOAc = 95:5); ^1H NMR (CDCl_3 , 600 MHz): δ 8.12 (d, 2H, $J = 7.8$ Hz), 7.64 (t, 1H, $J = 7.2$ Hz), 7.54 (t, 2H, $J = 7.8$ Hz), 7.33-7.28 (m, 4H), 7.26-7.23 (m, 1H), 4.33 (d, 1H, $J = 7.2$ Hz), 3.99 (d, 1H, $J = 7.2$ Hz), 3.93-3.88 (m, 2H), 1.18 (s, 9H), 0.97 (t, 3H, $J = 7.2$ Hz); ^{13}C NMR (CDCl_3 , 150 MHz): δ 206.2, 194.5, 166.4, 137.1, 133.9, 133.6, 128.9, 128.7, 128.6, 128.2, 127.5, 61.9, 52.4, 45.1, 36.5, 35.4, 28.1, 13.7. HRMS (ESI) m/z calcd. for $\text{C}_{24}\text{H}_{27}\text{O}_4$ [$\text{M}+\text{H}]^+$: 379.1904 found: 379.1896.

Diethyl 2-benzoyl-3-phenylcyclopropane-1,1-dicarboxylate (5a)^{S5}



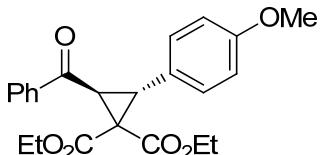
Yield: 34.4 mg (94%); time: 2 h; colorless oil; TLC, $R_f = 0.38$ (PE:EtOAc = 90:10); ^1H NMR (CDCl_3 , 400 MHz): δ 8.11 (d, 2H, $J = 7.6$ Hz), 7.62 (t, 1H, $J = 7.2$ Hz), 7.51 (t, 2H, $J = 7.2$ Hz), 7.32-7.26 (m, 5H), 4.17-4.10(m, 3H), 4.00 (q, 2H, $J = 6.8$ Hz), 3.80 (d, 1H, $J = 7.6$ Hz), 1.11 (t, 3H, $J = 7.2$ Hz), 0.98 (t, 3H, $J = 7.2$ Hz); ^{13}C NMR (CDCl_3 , 100 MHz): δ 193.6, 165.9, 165.7, 136.7, 136.6, 133.6, 133.4, 128.7, 128.58, 128.57, 128.3, 127.7, 62.0, 61.9, 46.0, 35.8, 34.9, 13.83, 13.78.

Dimethyl 2-benzoyl-3-phenylcyclopropane-1,1-dicarboxylate (5b)^{S5}



Yield: 31.4 mg (93%); time: 1.5 h; colorless oil; TLC, $R_f = 0.26$ (PE:EtOAc = 90:10); ^1H NMR (CDCl_3 , 600 MHz): δ 8.10 (d, 2H, $J = 7.2$ Hz), 7.62 (t, 1H, $J = 7.2$ Hz), 7.52 (t, 2H, $J = 7.8$ Hz), 7.34-7.26 (m, 5H), 4.14(d, 1H, $J = 7.8$ Hz), 3.88 (d, 1H, $J = 7.8$ Hz), 3.72 (s, 3H), 3.54 (s, 3H); ^{13}C NMR (CDCl_3 , 150 MHz): δ 193.8, 166.5, 166.1, 136.8, 133.7, 133.4, 128.8, 128.53, 128.49, 128.4, 127.8, 53.0, 52.9, 46.0, 36.6, 35.1;

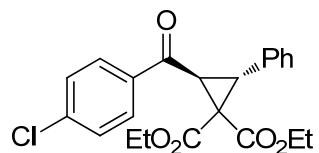
Diethyl 2-benzoyl-3-(4-methoxyphenyl)cyclopropane-1,1-dicarboxylate (5c)^{S5}



Yield: 36.8 mg (93%); time: 2 h; colorless oil; TLC, $R_f = 0.31$ (PE:EtOAc = 90:10); ^1H NMR (CDCl_3 , 400 MHz): δ 8.10 (d, 2H, $J = 7.2$ Hz), 7.61 (t, 1H, $J = 7.2$ Hz), 7.51 (t, 2H, $J = 7.6$ Hz), 7.23 (d, 2H, $J = 8.4$ Hz), 6.83 (d, 2H, $J = 8.4$ Hz), 4.13(q, 2H, $J = 7.2$ Hz), 4.07 (d, 1H, $J = 7.6$ Hz), 4.05-3.98 (m, 2H), 3.82 (d, 1H, $J = 7.6$ Hz), 3.79 (s, 3H), 1.11 (t, 3H, $J = 7.2$ Hz), 1.03 (t, 3H, $J = 7.2$ Hz); ^{13}C NMR (CDCl_3 , 100 MHz): δ 193.7, 165.9, 165.7, 136.7, 136.6, 133.6, 133.4, 128.7, 128.58, 128.57, 128.3, 127.7, 62.0, 61.9, 46.0, 35.8, 34.9, 13.83, 13.78.

166.0, 165.8, 159.1, 136.8, 133.6, 129.7, 128.7, 128.6, 125.4, 113.7, 62.0, 61.8, 55.2, 46.1, 35.4, 35.2, 13.88, 13.84.

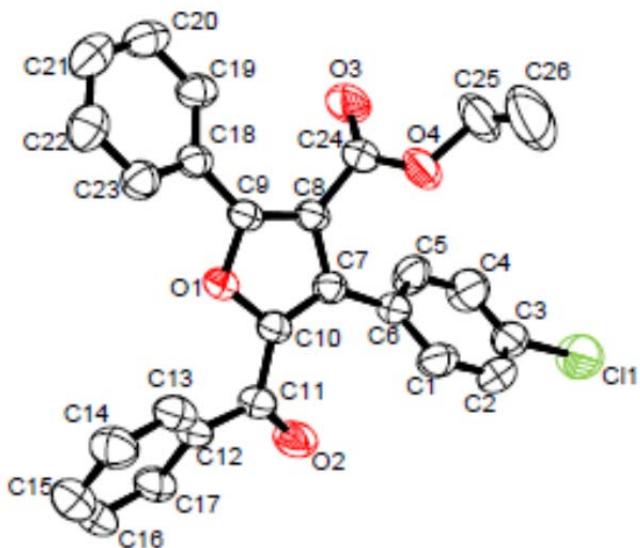
Diethyl 2-(4-chlorobenzoyl)-3-phenylcyclopropane-1,1-dicarboxylate (5d)^{S5}



Yield: 38.4 mg (96%); time: 2.5 h; colorless oil; TLC, $R_f = 0.45$ (PE:EtOAc = 90:10);
 ^1H NMR (CDCl_3 , 400 MHz): δ 8.01 (d, 2H, $J = 8.8$ Hz), 7.45 (d, 2H, $J = 8.8$ Hz), 7.28-7.22 (m, 5H), 4.11 (q, 2H, $J = 7.2$ Hz), 4.01 (d, 1H, $J = 7.6$ Hz), 3.97 (q, 2H, $J = 7.2$ Hz), 3.84 (d, 1H, $J = 7.6$ Hz), 1.08 (t, 3H, $J = 7.2$ Hz), 0.95 (t, 3H, $J = 7.2$ Hz); ^{13}C NMR (CDCl_3 , 100 MHz): δ 192.3, 165.8, 165.6, 140.2, 135.0, 133.2, 129.9, 129.1, 128.5, 128.3, 127.7, 62.1, 61.9, 46.1, 35.7, 34.9, 13.83, 13.75.

X-Ray structure of **2i** and the corresponding data

CCDC 1497561 (**2i**), contains the supplementary crystallographic data for this paper. These data can be obtained free of charge from the Cambridge Crystallographic Data Centre via: www.ccdc.cam.ac.uk/data_request/cif.



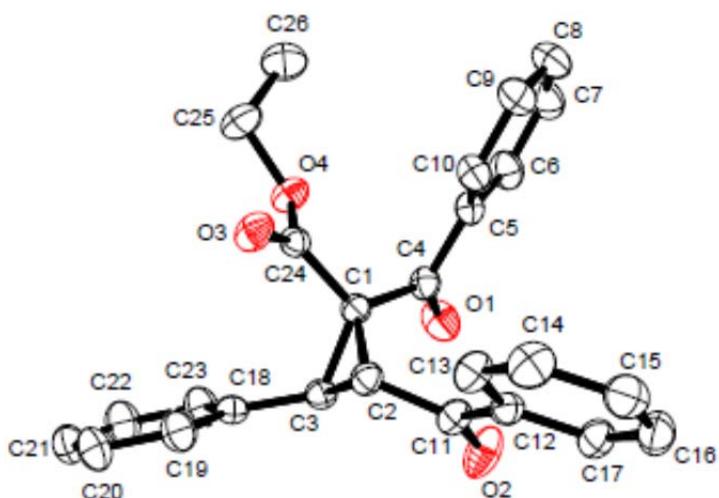
Crystal data	
Chemical formula	C ₂₆ H ₁₉ ClO ₄
M _r	430.86
Crystal system, space group	Triclinic, <i>P</i>
Temperature (K)	293
<i>a</i> , <i>b</i> , <i>c</i> (Å)	8.8081 (4), 9.9881 (5), 12.6194 (6)
α, β, γ (°)	79.9048 (16), 82.4837 (16), 80.2931 (16)
<i>V</i> (Å ³)	1071.49 (9)
<i>Z</i>	2
Radiation type	Mo <i>K</i> α
μ (mm ⁻¹)	0.21
Crystal size (mm)	0.15 × 0.13 × 0.08
Data collection	
Diffractometer	Bruker <i>APEX-II</i> CCD

Absorption correction	Multi-scan <i>SADABS</i>
No. of measured, independent and observed [$I > 2\sigma(I)$] reflections	14454, 3790, 2661
R_{int}	0.027
$(\sin \theta / \lambda)_{\text{max}} (\text{\AA}^{-1})$	0.596
Refinement	
$R[F^2 > 2\sigma(F^2)], wR(F^2), S$	0.051, 0.143, 1.31
No. of reflections	3790
No. of parameters	281
H-atom treatment	H-atom parameters constrained
$\Delta\rho_{\text{max}}, \Delta\rho_{\text{min}} (\text{e \AA}^{-3})$	0.21, -0.29

Computer programs: Bruker *APEX2*, Bruker *SAINT*, *SHELXS97* (Sheldrick 2008), *SHELXL2014/7* (Sheldrick, 2014), Bruker *SHELXTL*.

X-Ray structure of 3a and the corresponding data

CCDC 1497563 (**3a**), contains the supplementary crystallographic data for this paper. These data can be obtained free of charge from the Cambridge Crystallographic Data Centre via: www.ccdc.cam.ac.uk/data_request/cif.



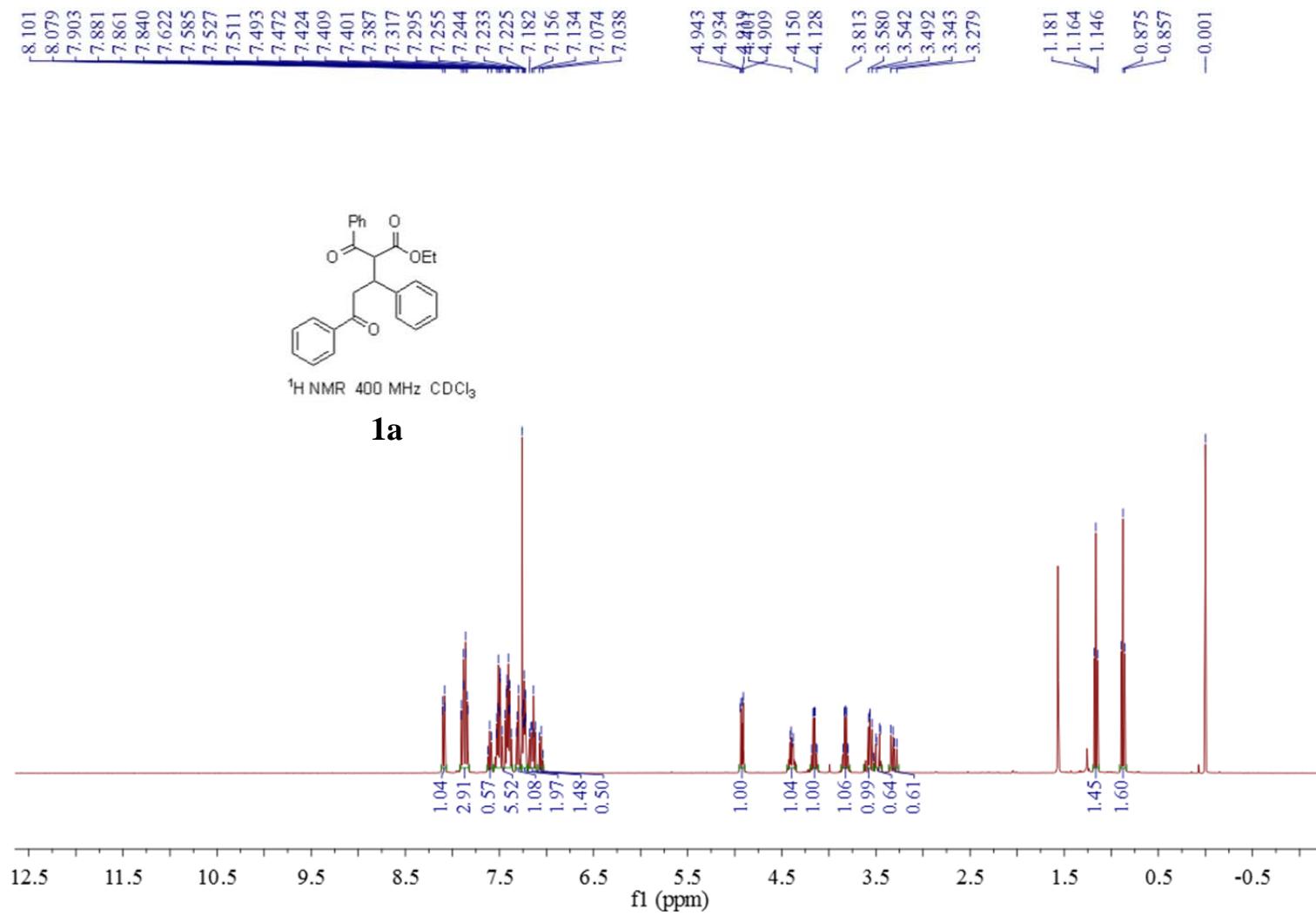
Crystal data	
Chemical formula	C ₂₆ H ₂₂ O ₄
M _r	398.45
Crystal system, space group	Monoclinic, P2 ₁ /n
Temperature (K)	299
<i>a</i> , <i>b</i> , <i>c</i> (Å)	8.5852 (6), 15.9050 (12), 15.6721 (12)
β (°)	95.176 (2)
<i>V</i> (Å ³)	2131.3 (3)
<i>Z</i>	63
Radiation type	Mo Kα
μ (mm ⁻¹)	0.13
Data collection	
Diffractometer	Bruker APEX-II CCD diffractometer
No. of measured, independent and observed [<i>I</i> > 2σ(<i>I</i>)] reflections	15870, 3759, 2729
<i>R</i> _{int}	0.032
(sin θ/λ) _{max} (Å ⁻¹)	0.596
Refinement	
<i>R</i> [<i>F</i> ² > 2σ(<i>F</i> ²)], <i>wR</i> (<i>F</i> ²), <i>S</i>	0.046, 0.126, 1.03
No. of reflections	3759
No. of parameters	272
H-atom treatment	H-atom parameters constrained
Δρ _{max} , Δρ _{min} (e Å ⁻³)	0.16, -0.19

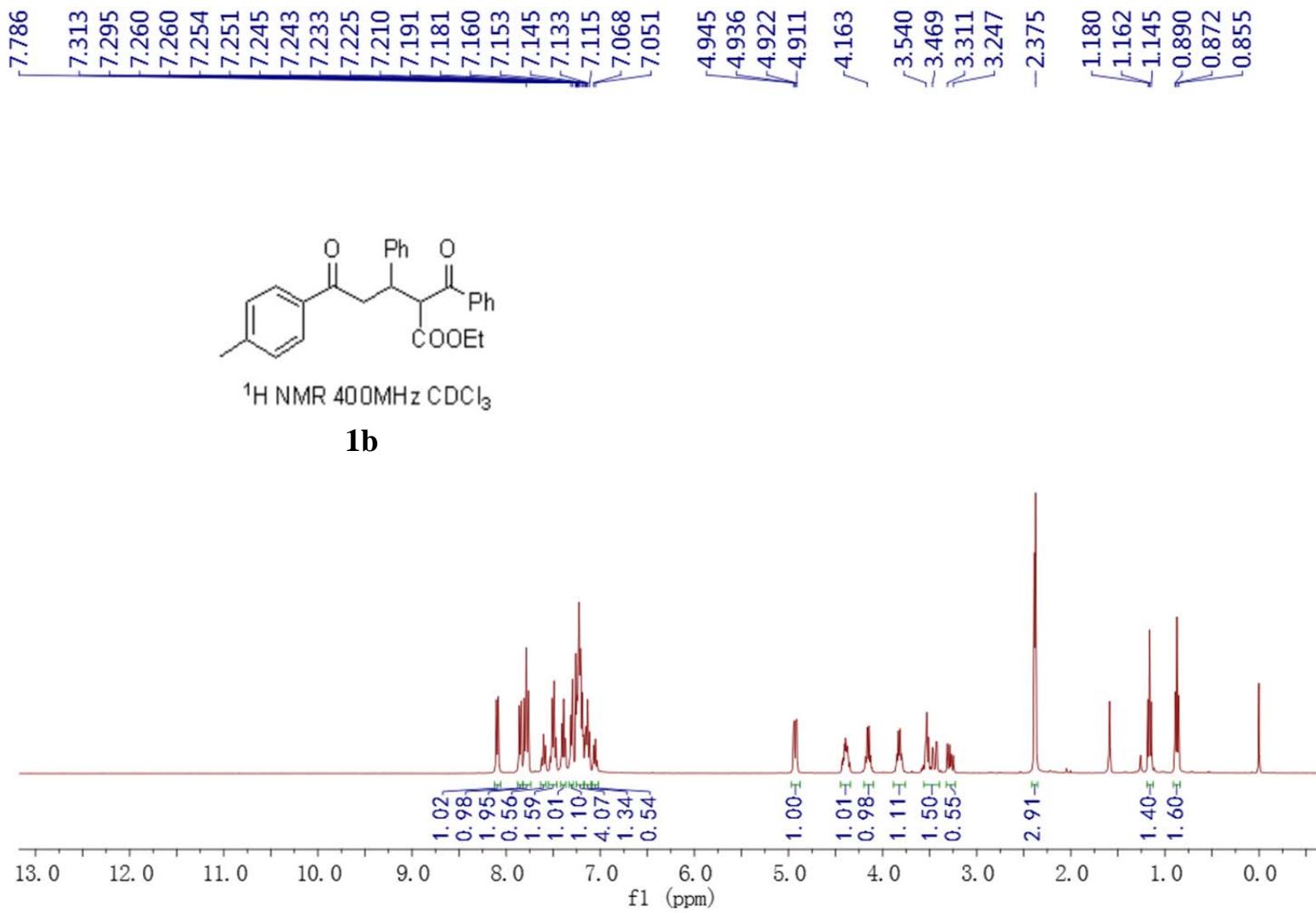
Computer programs: SHELXL2014/7 (Sheldrick, 2014).

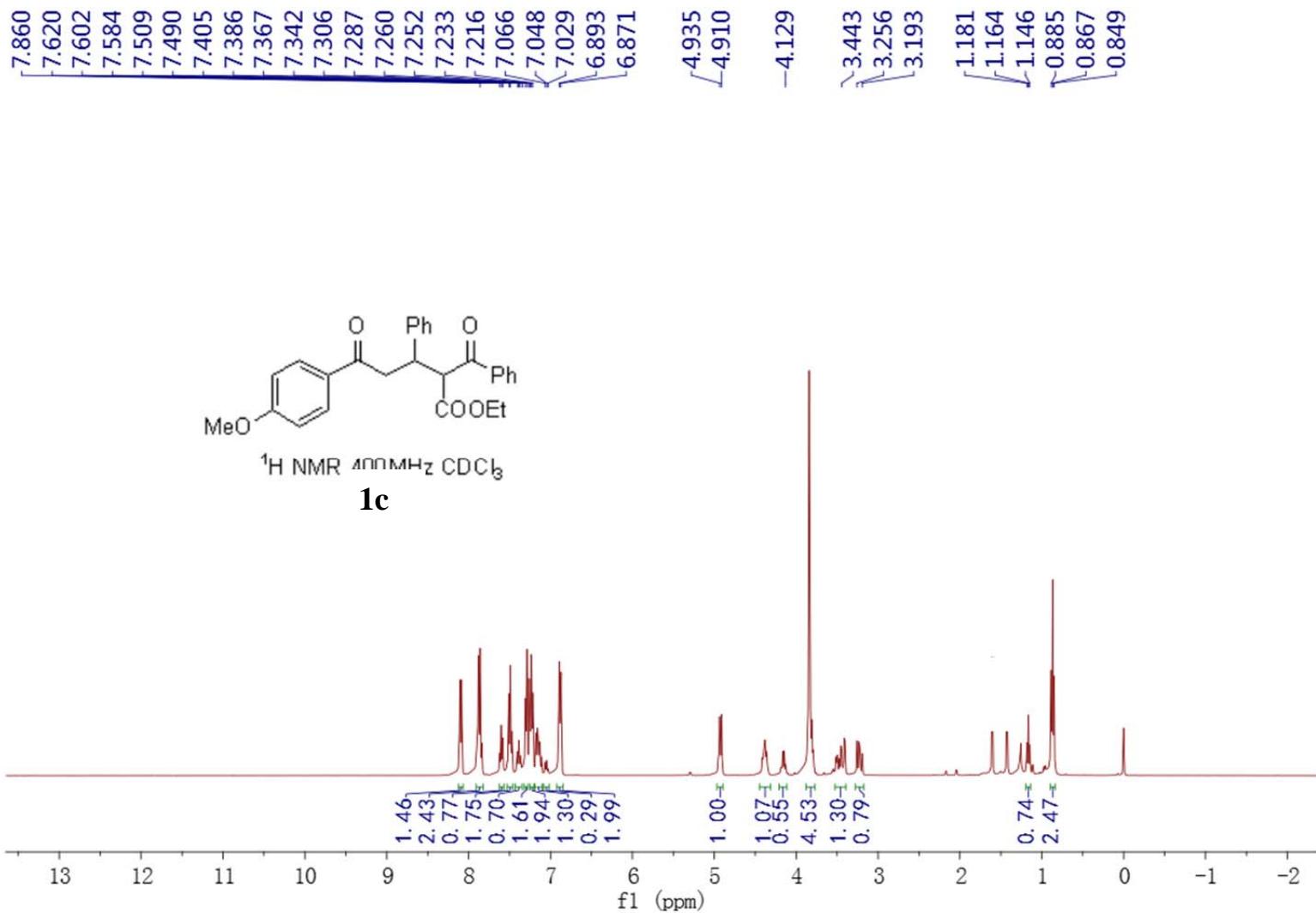
References:

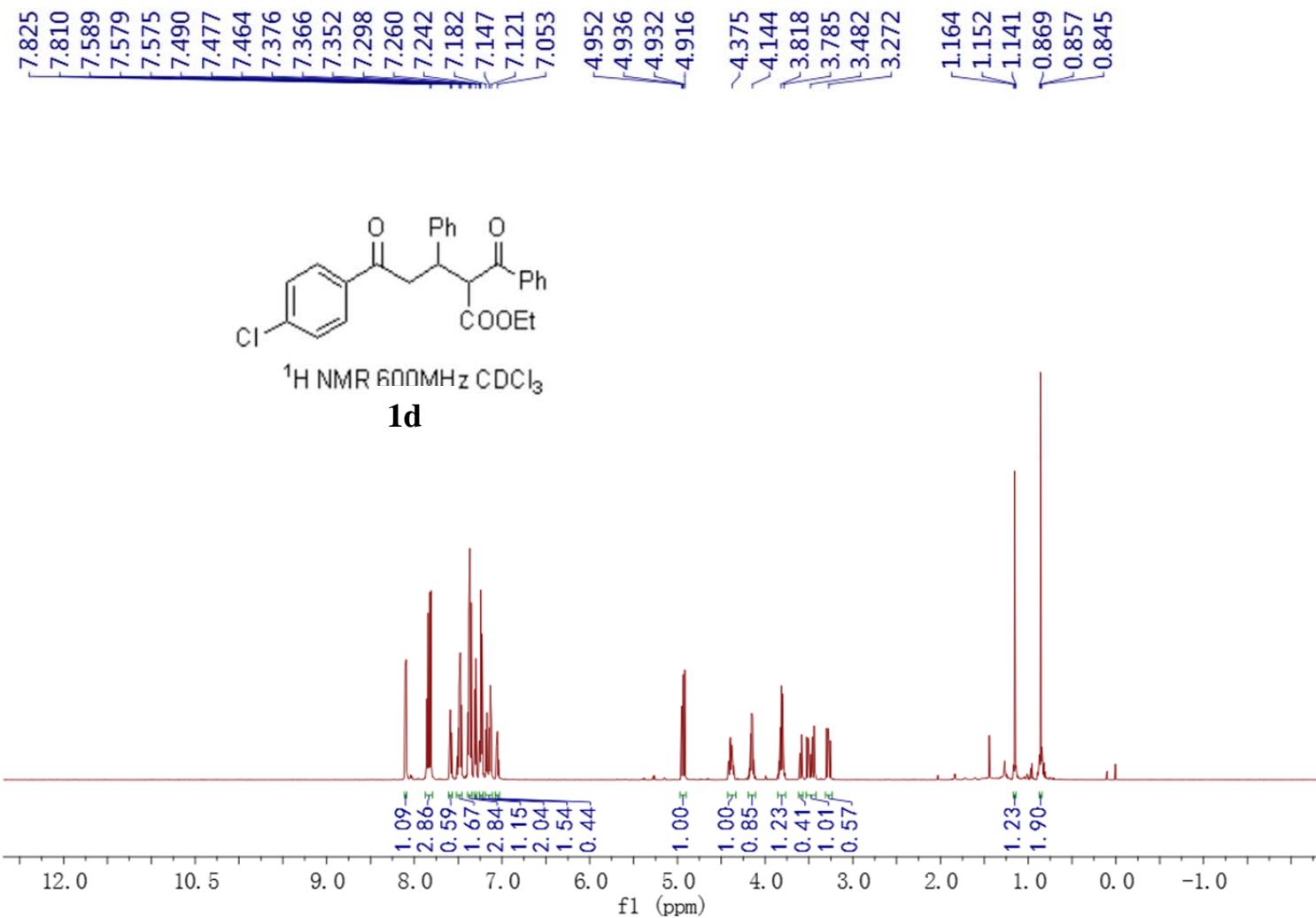
- [S1] Yang, H.-M.; Gao, Y.-H.; Li, L.; Jiang, Z.-Y.; Lai, G.-Q.; Xia, C.-G.; Xu, L.-W. *Tetrahedron Lett.* **2010**, *51*, 3836.
- [S2] Christoffers, J. *Chem. Commun.* **1997**, 943.
- [S3] Sammour, A.; Raouf, A.; Elkasaby, M.; Ibrahim, M. A. *Acta Chimica Academiae Scientiarum Hungaricae* **1973**, *78*, 399.
- [S4] Shkel, A. A.; Mazhukina, O. A.; Fedotova, O. V. *Chem. Heterocycl. Compd.* **2011**, *47*, 656.
- [S5] Ye, Y.; Zheng, C.; Fan, R. *Org. Lett.* **2009**, *11*, 3156.

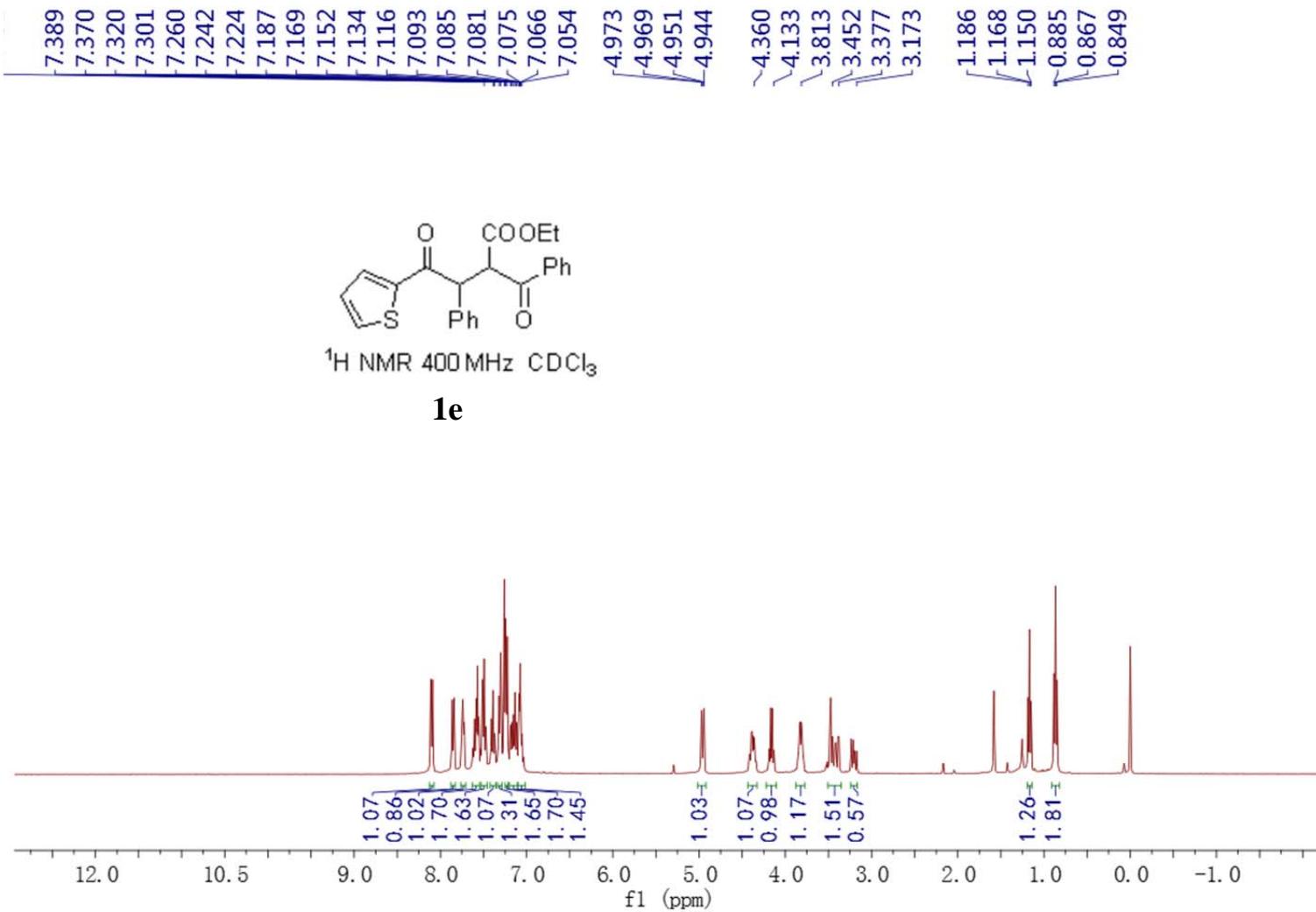
¹H NMR Spectra of Michael Adducts



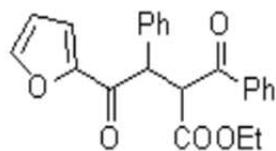






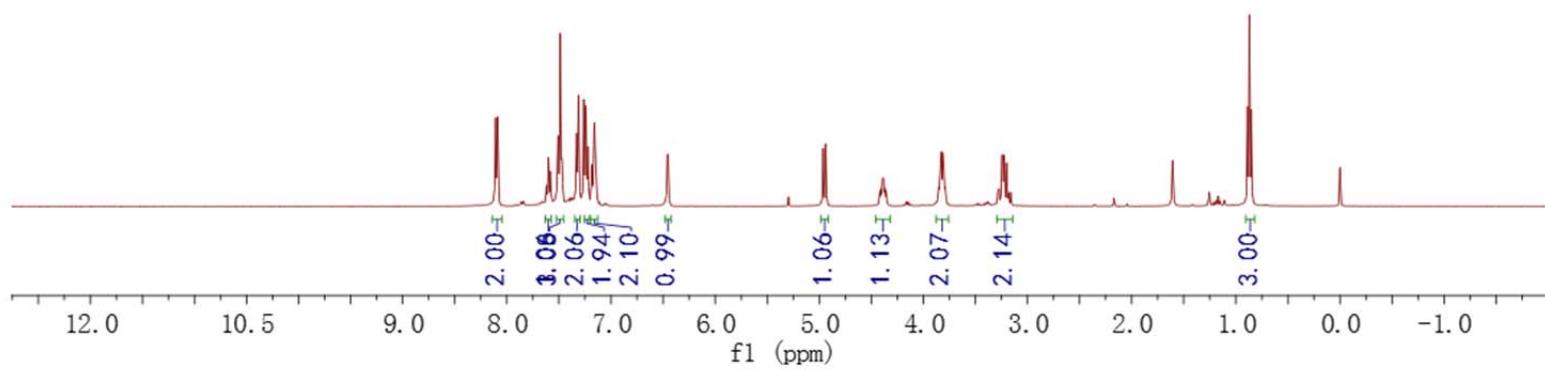


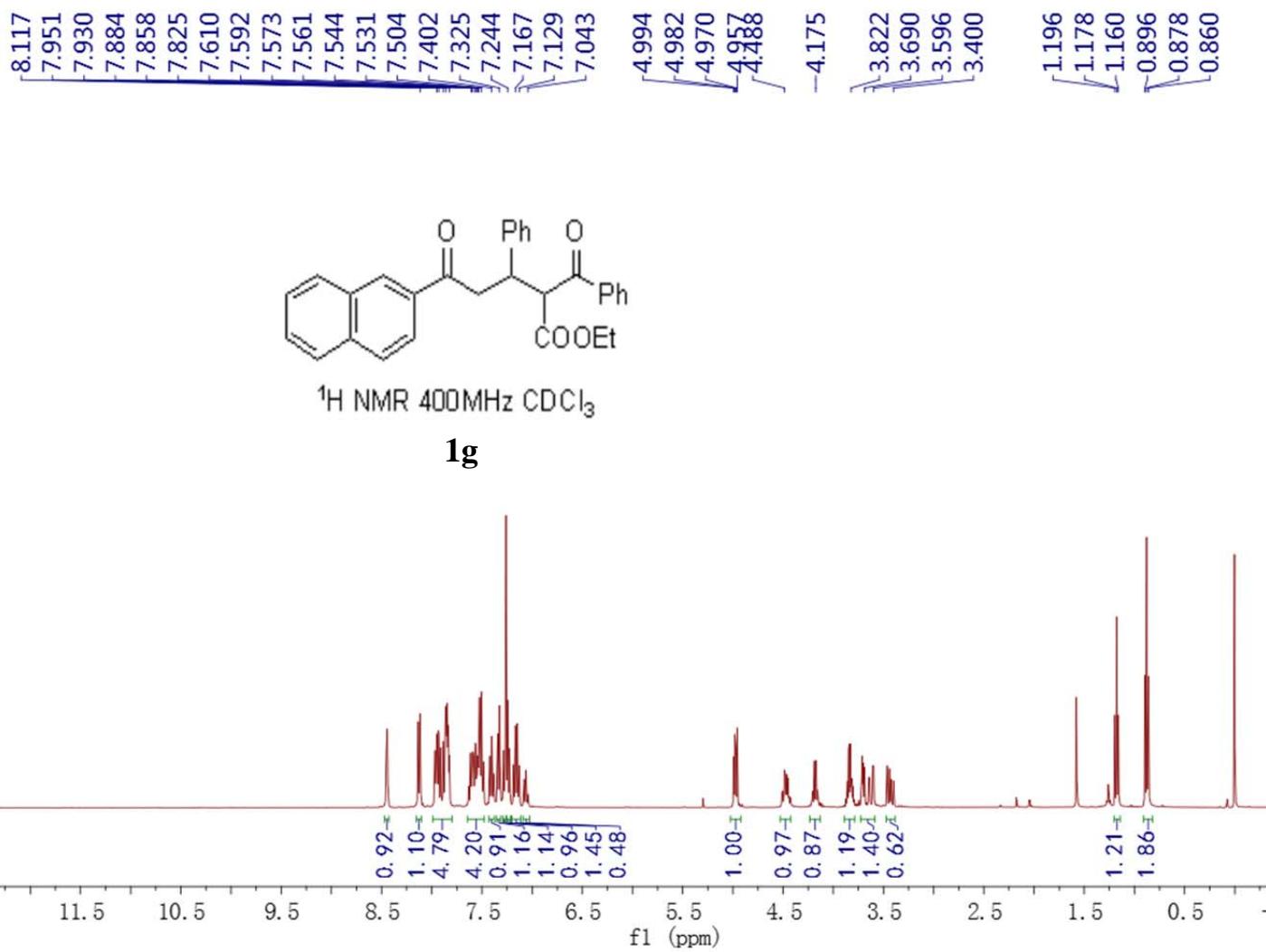
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 6.453

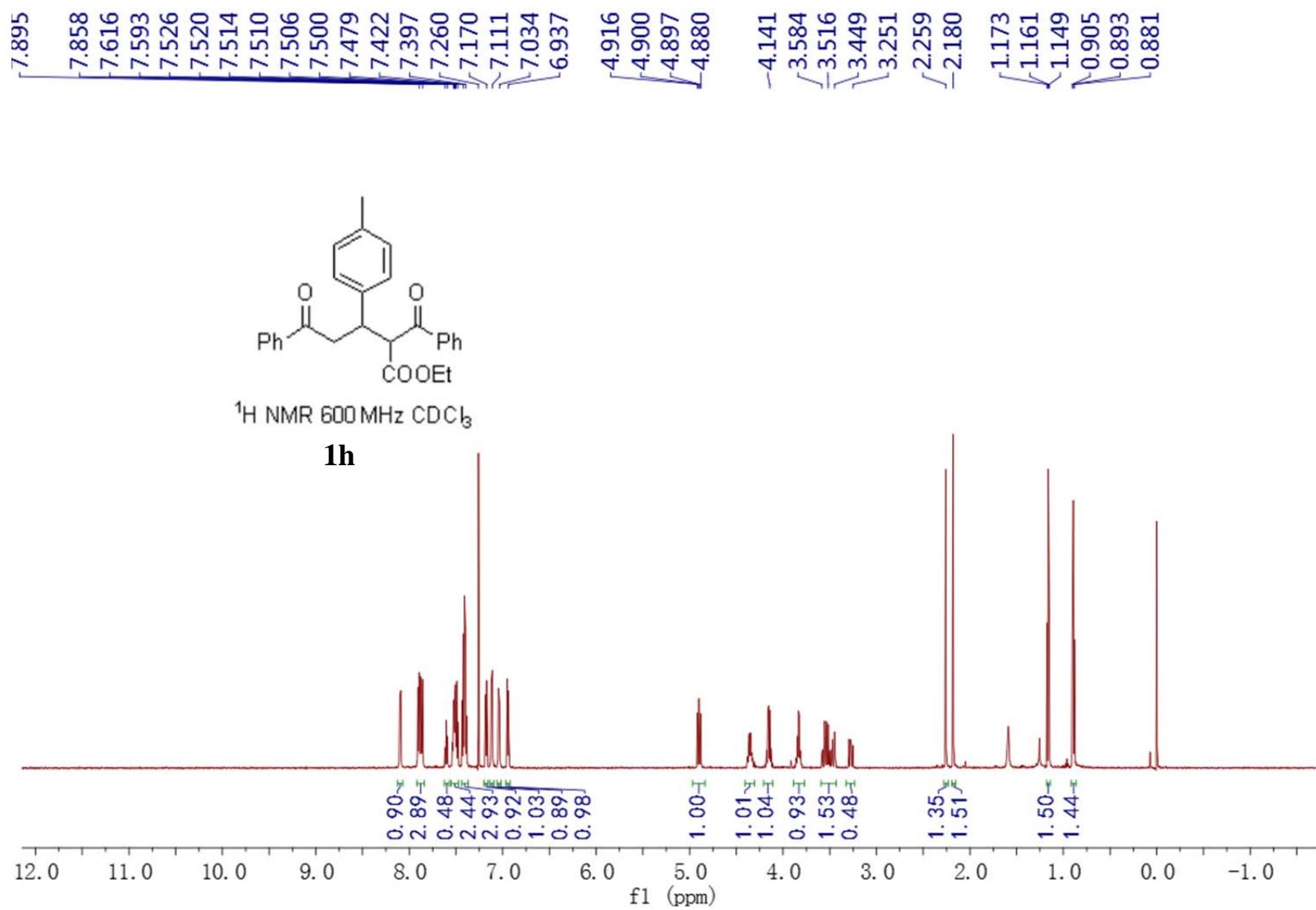


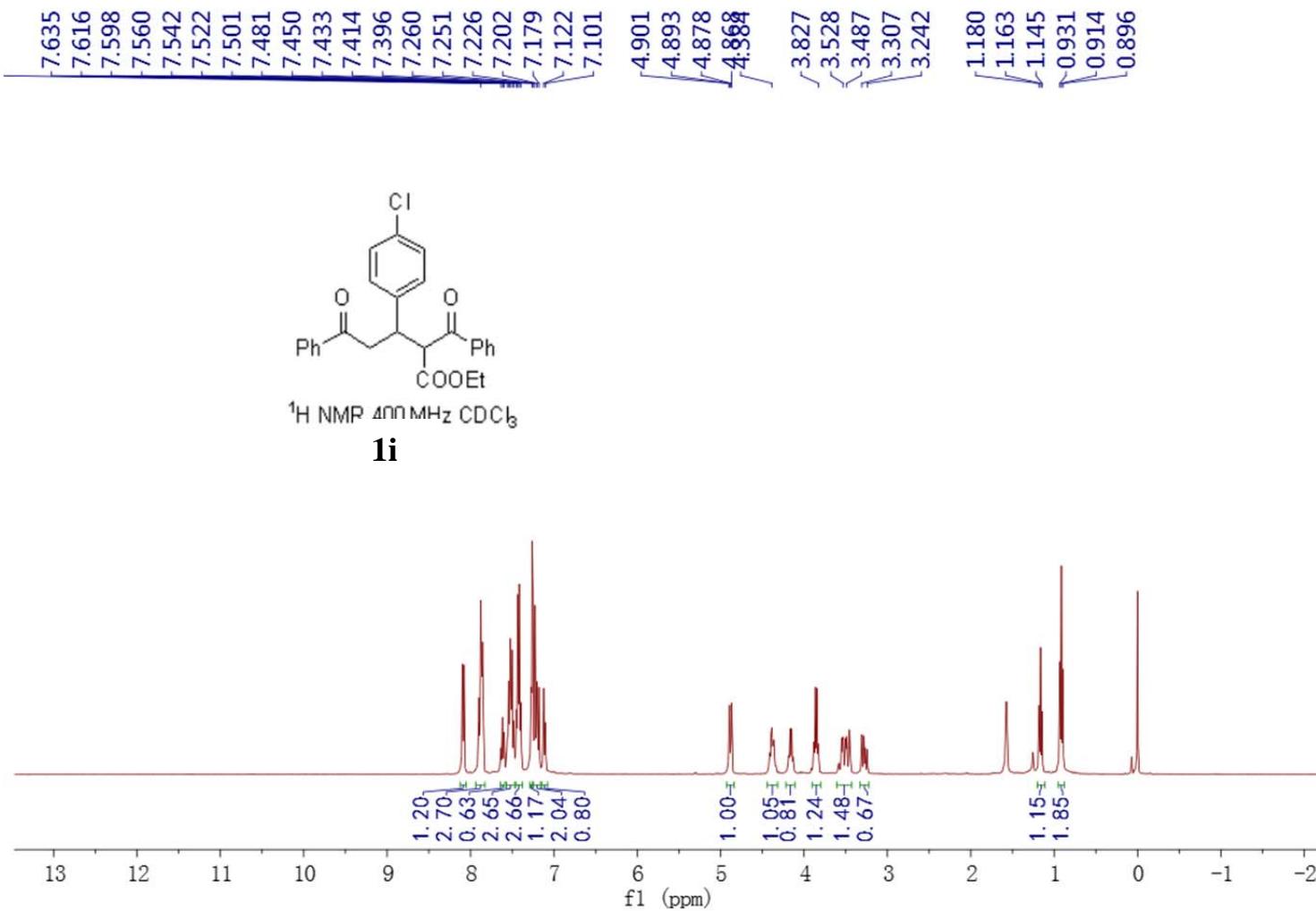
¹H NMR 400MHz CDCl₃

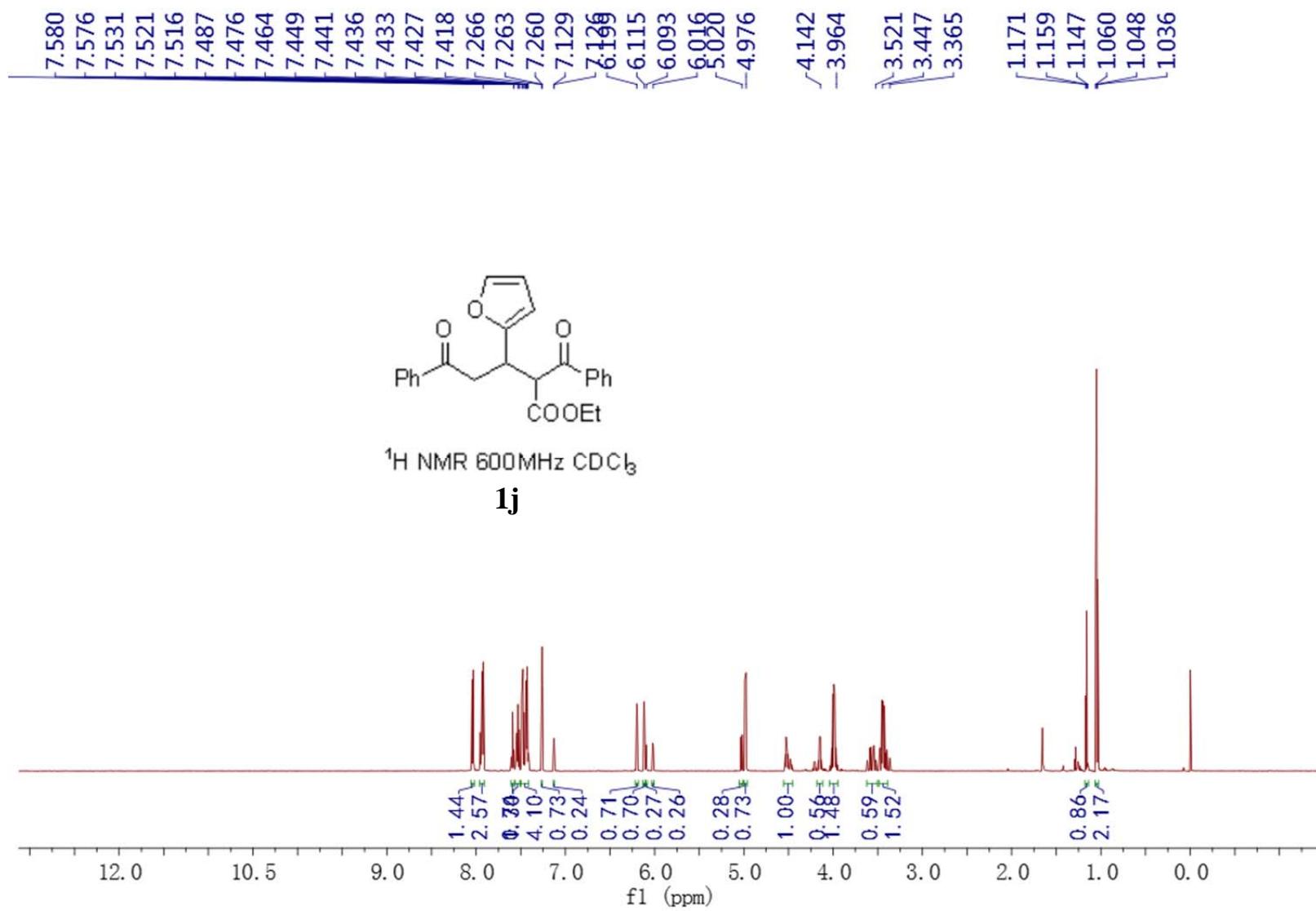
1f



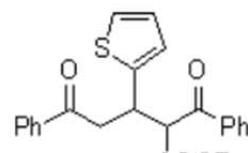






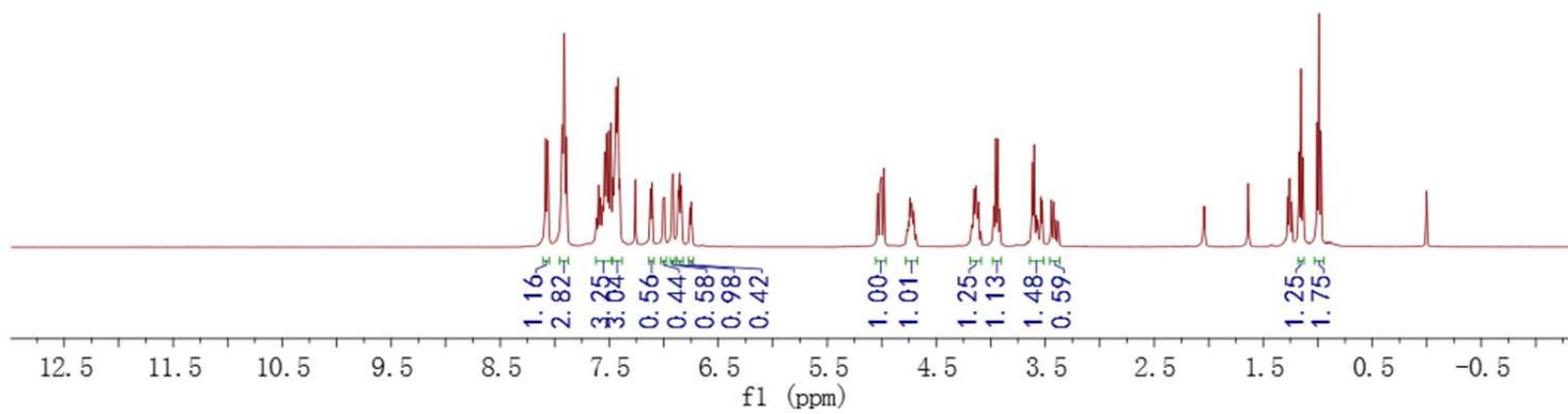


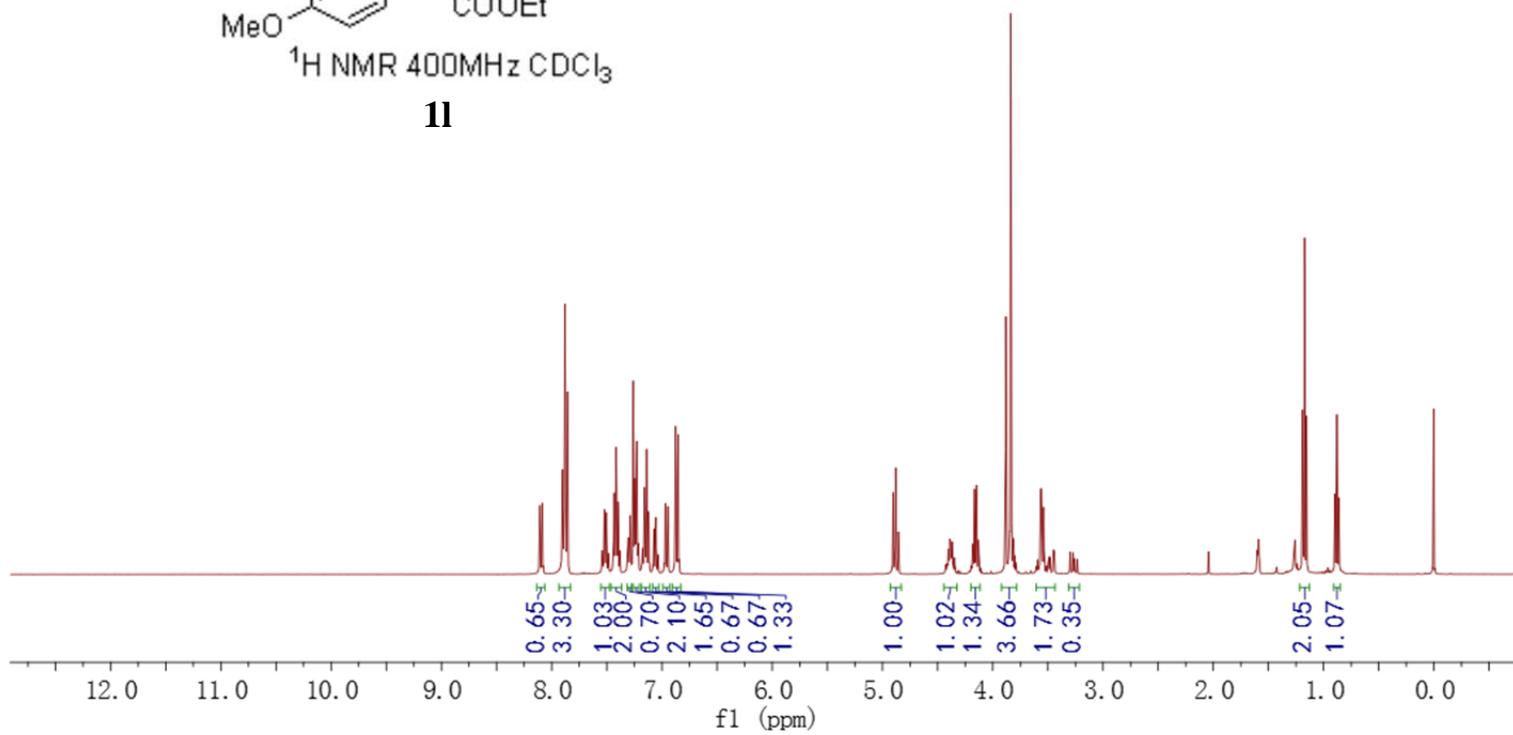
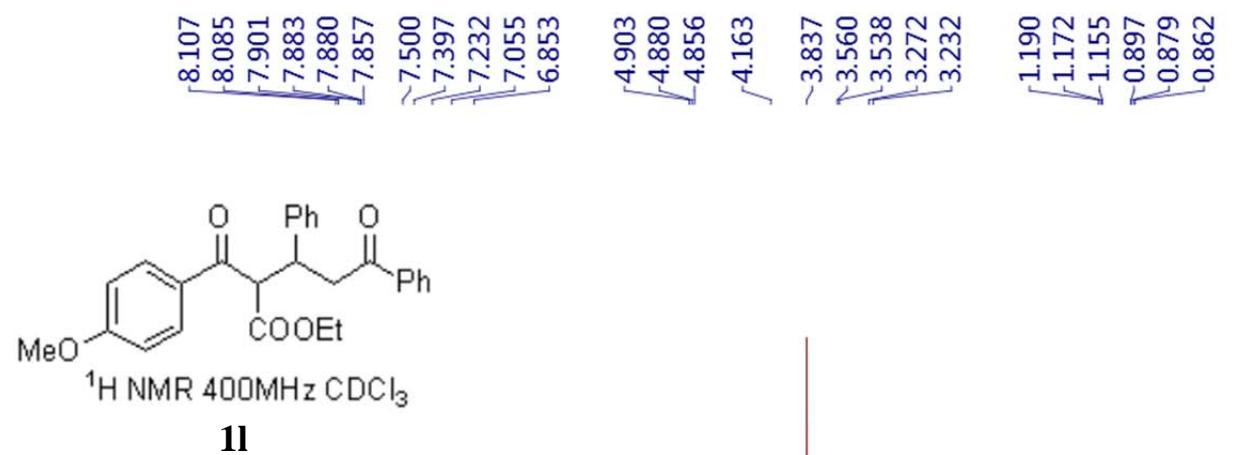
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6.750
6.747
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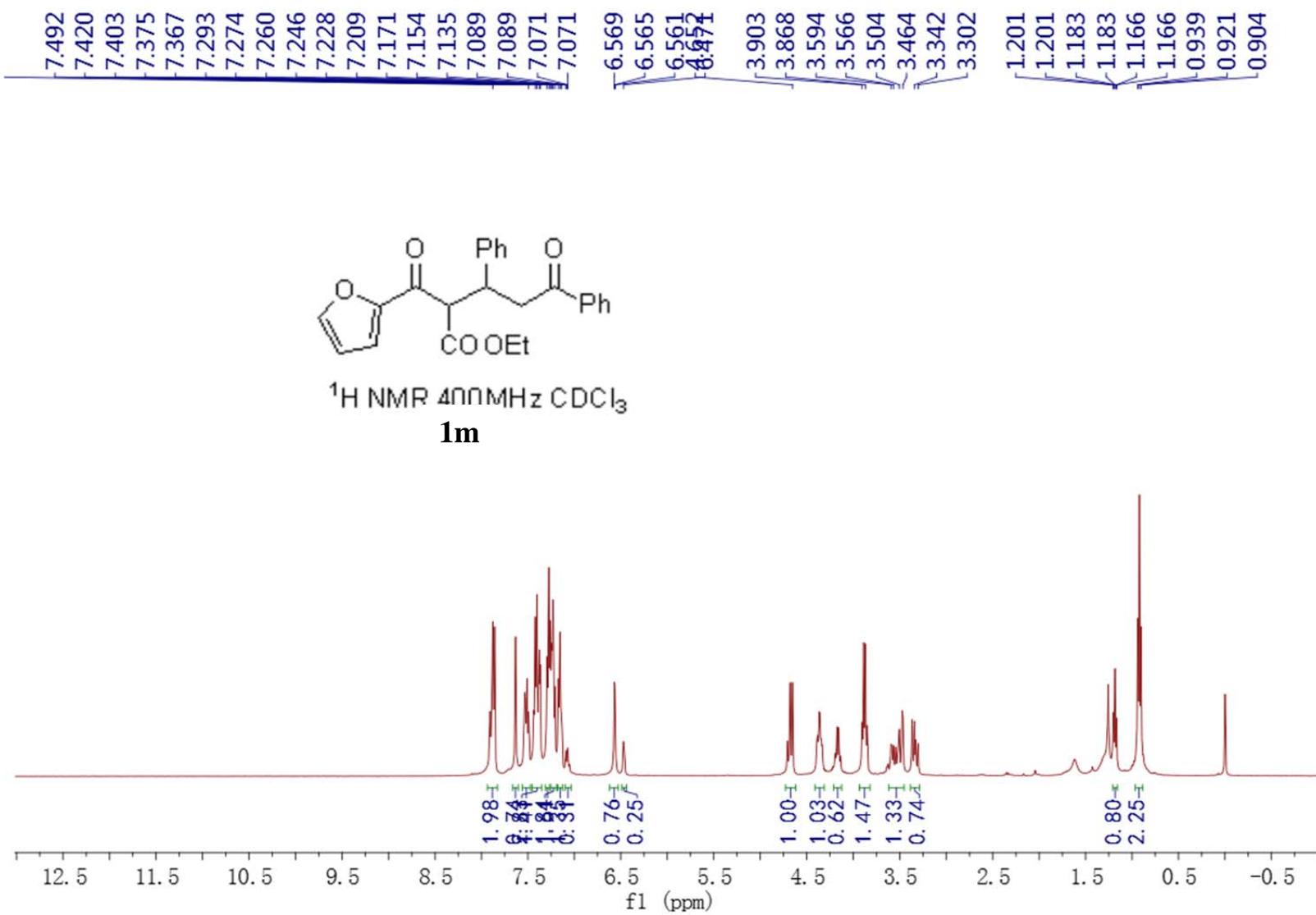


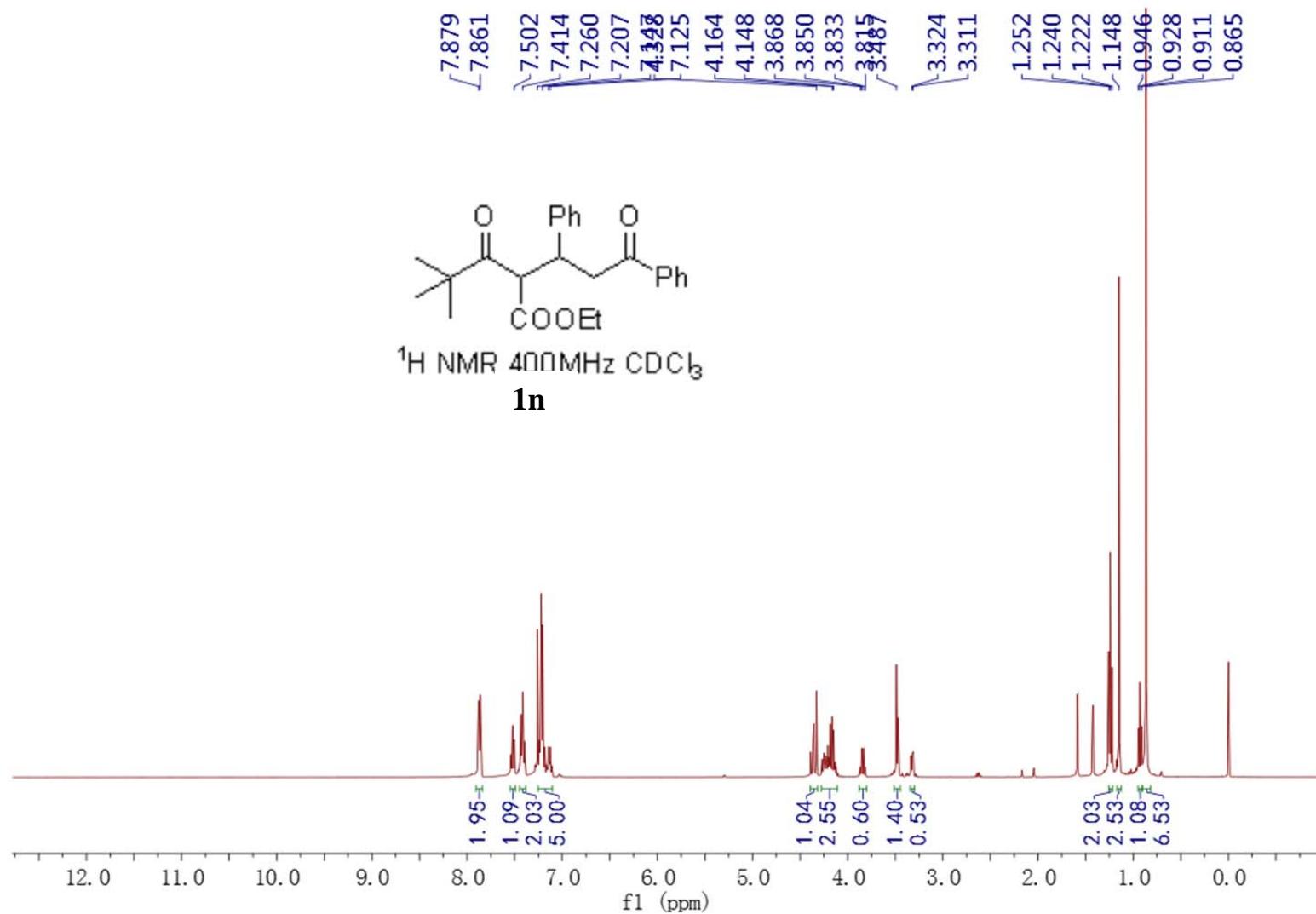
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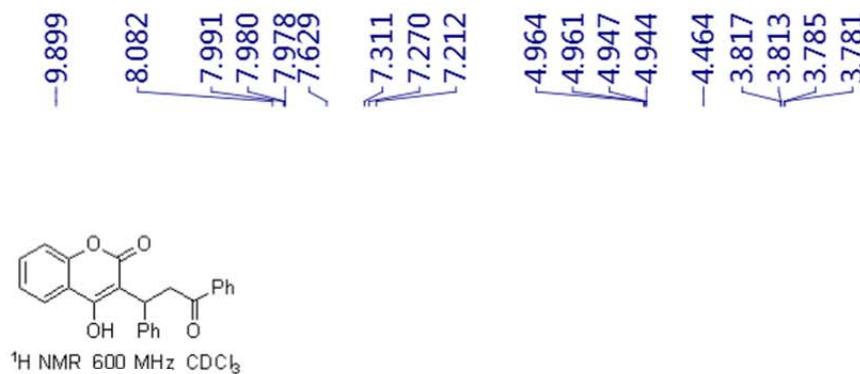
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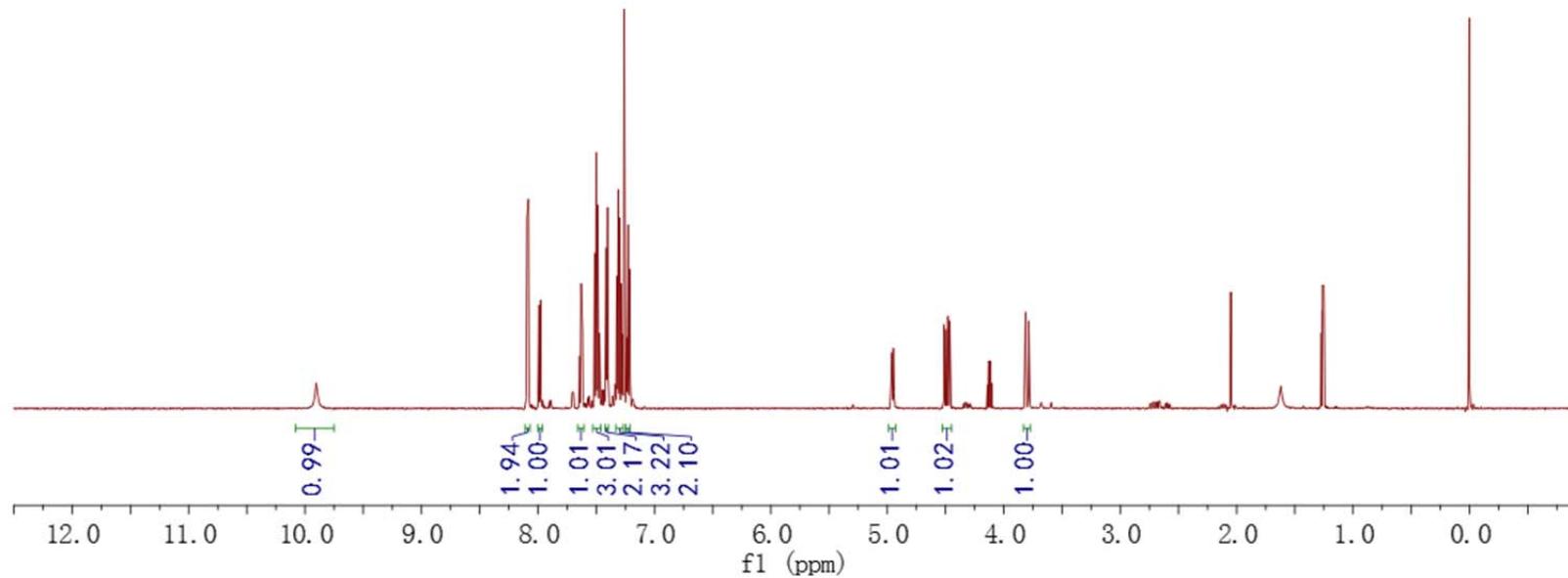


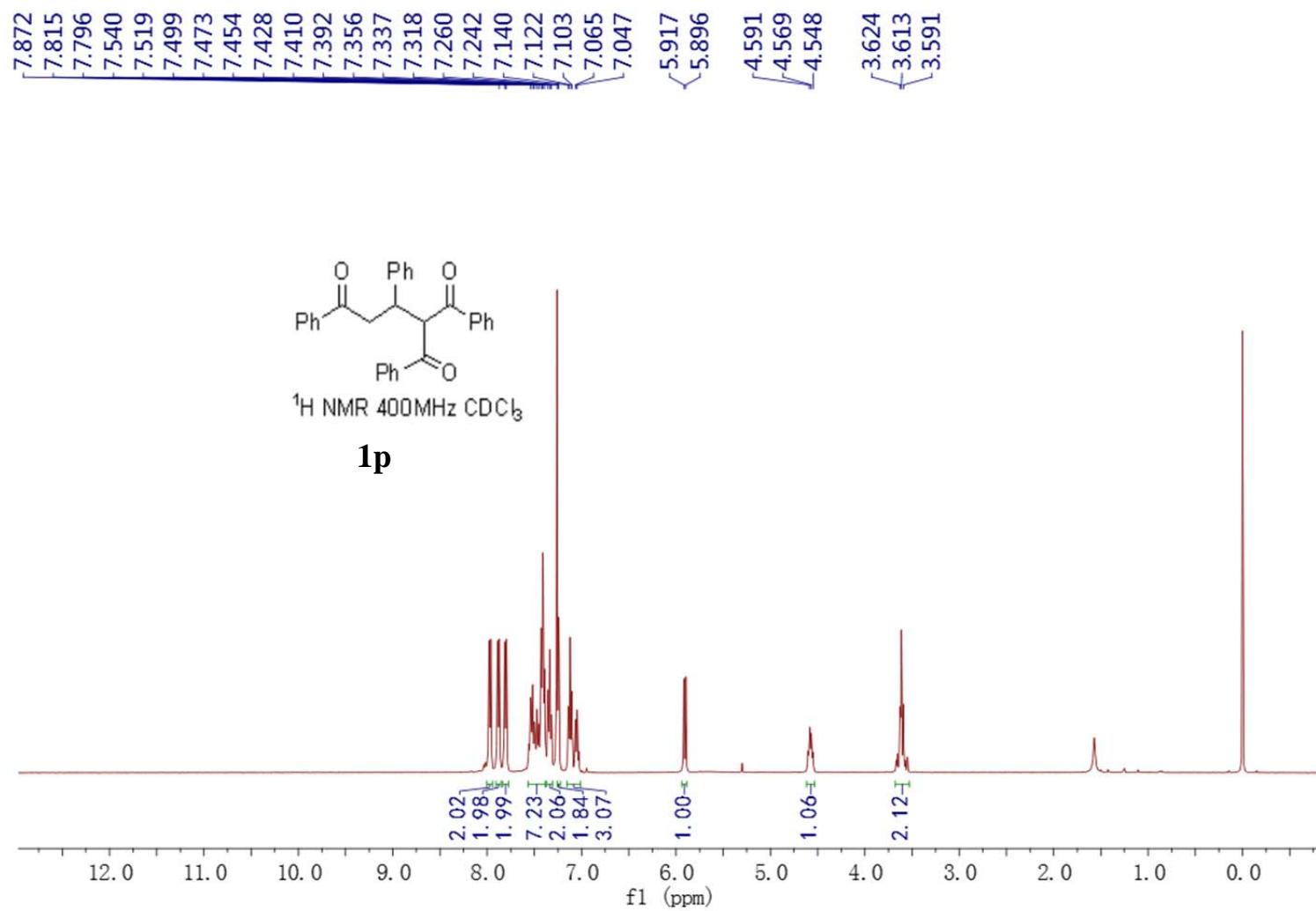


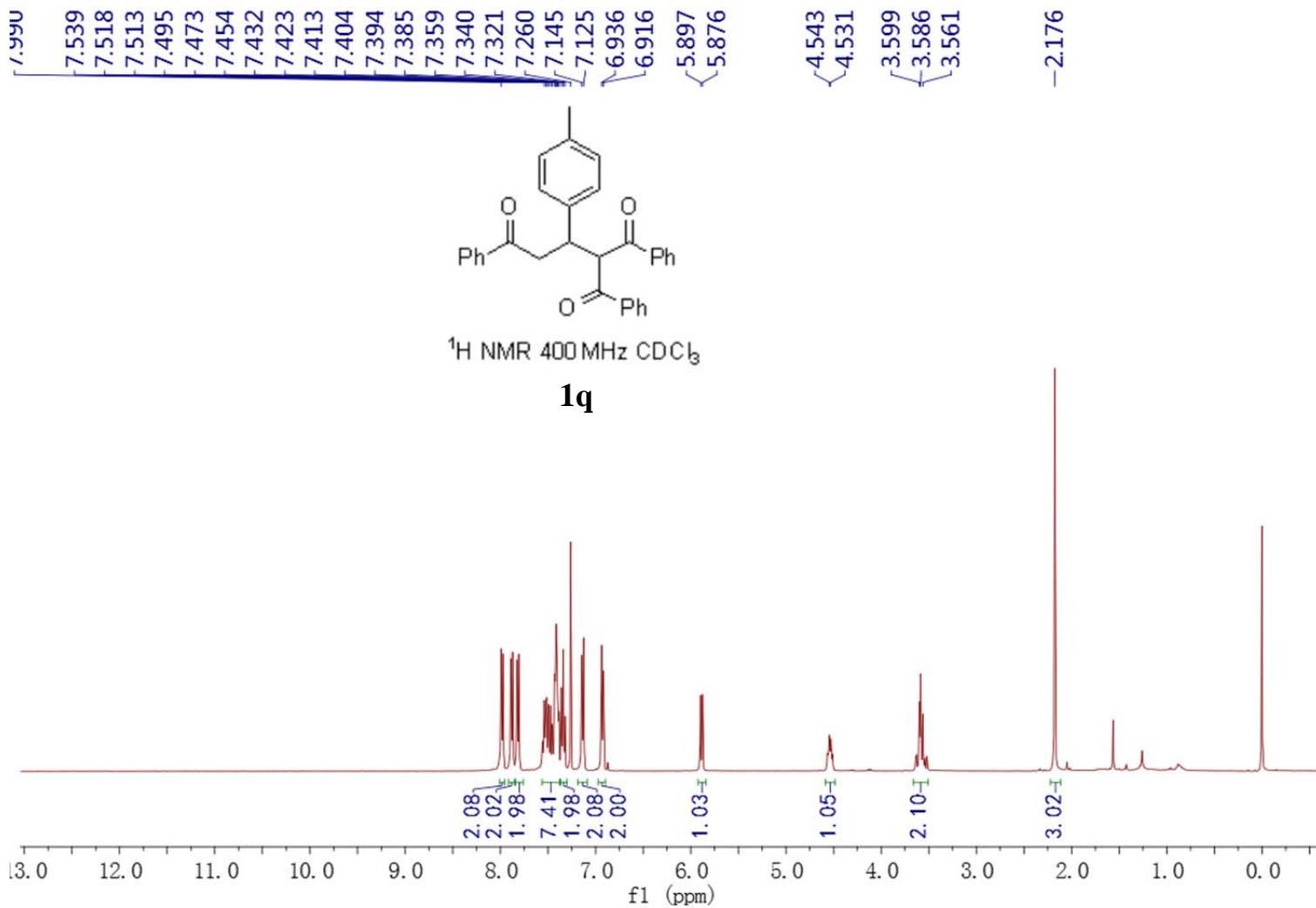


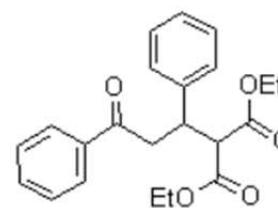


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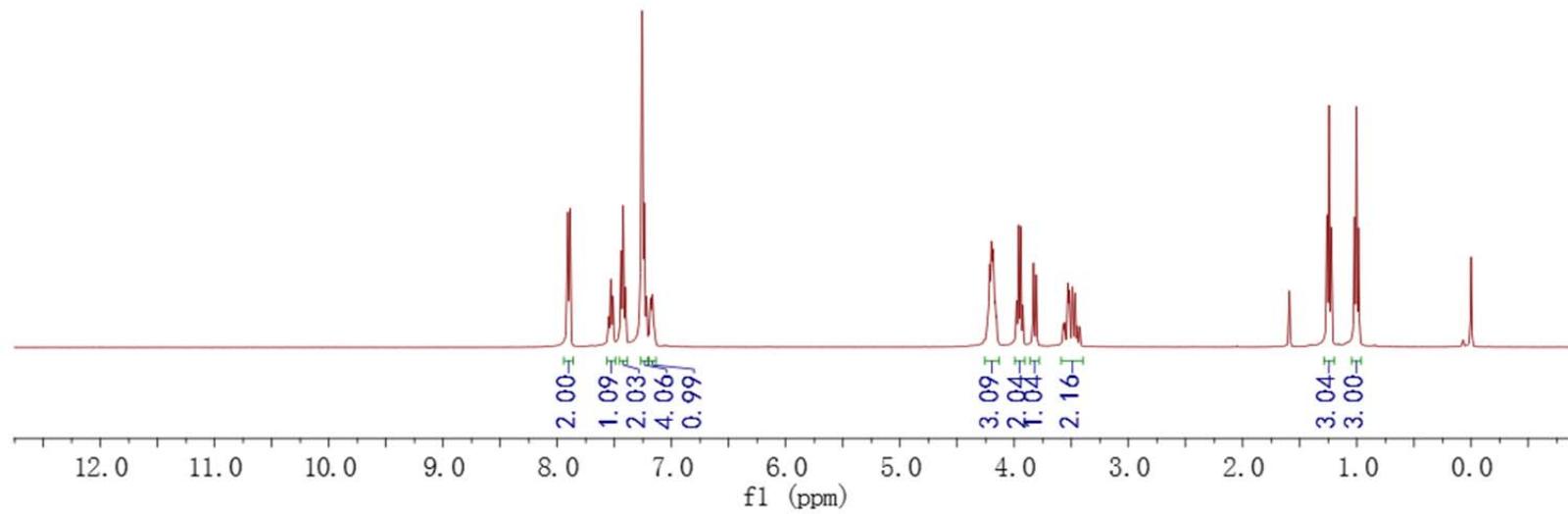


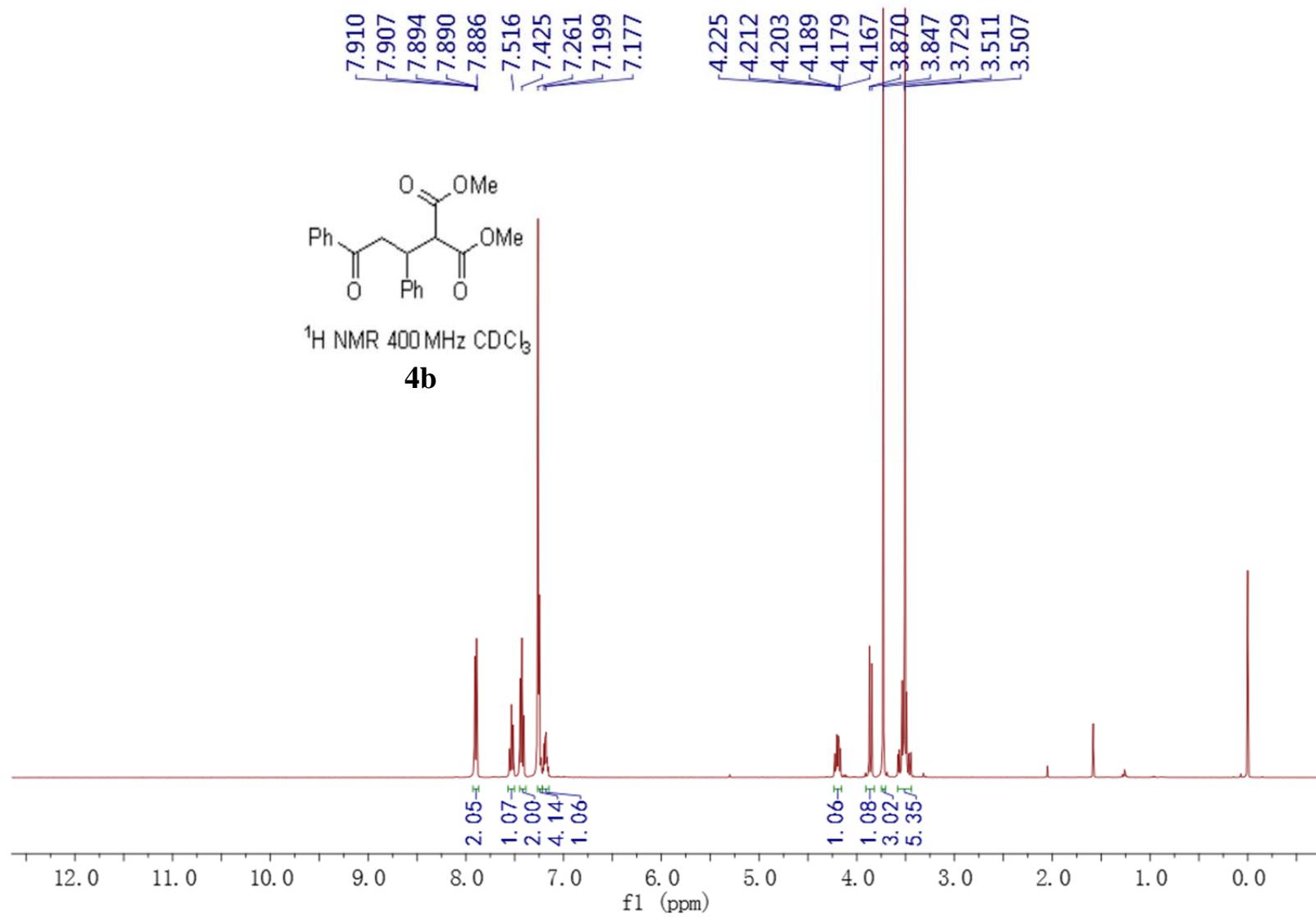


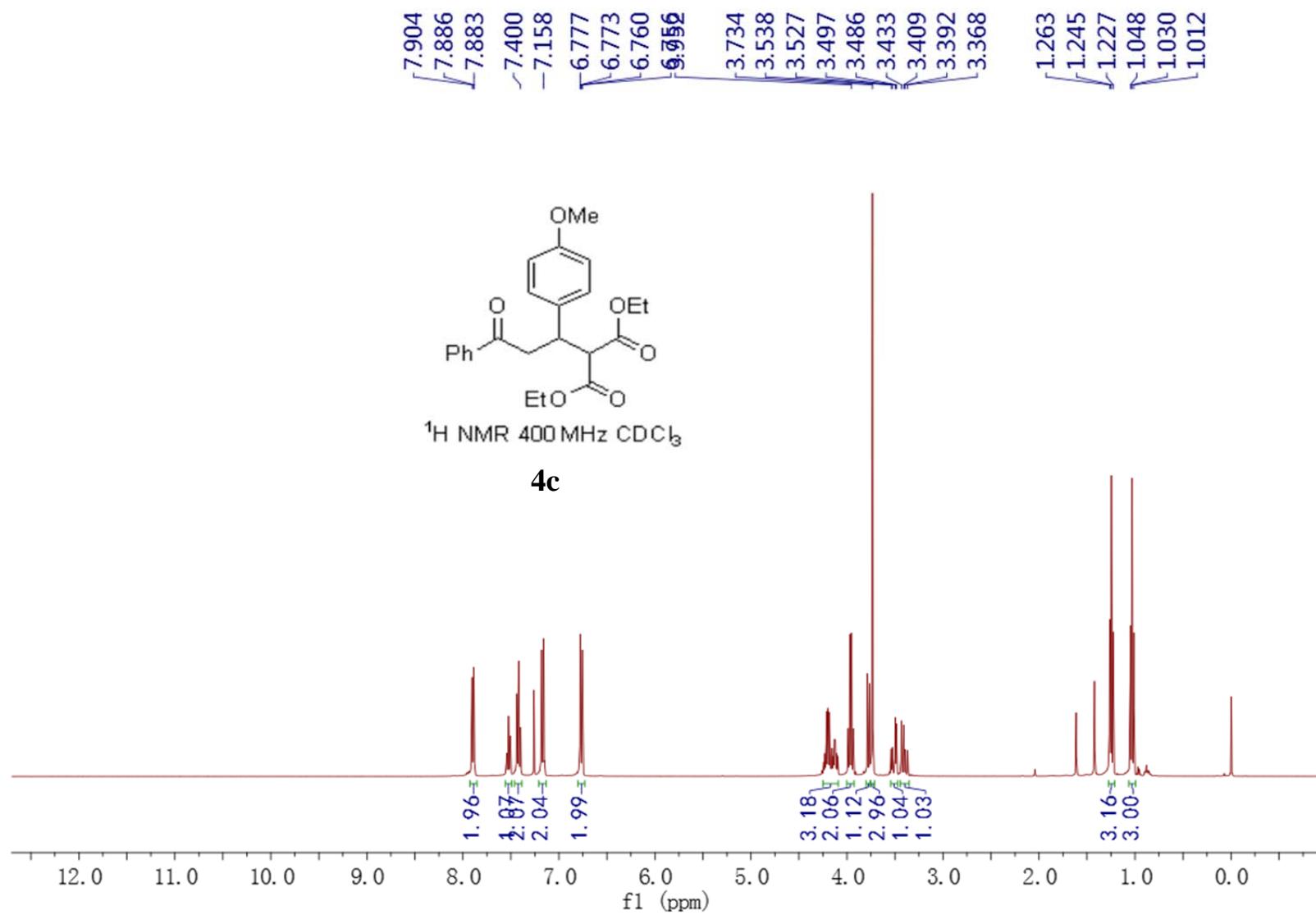


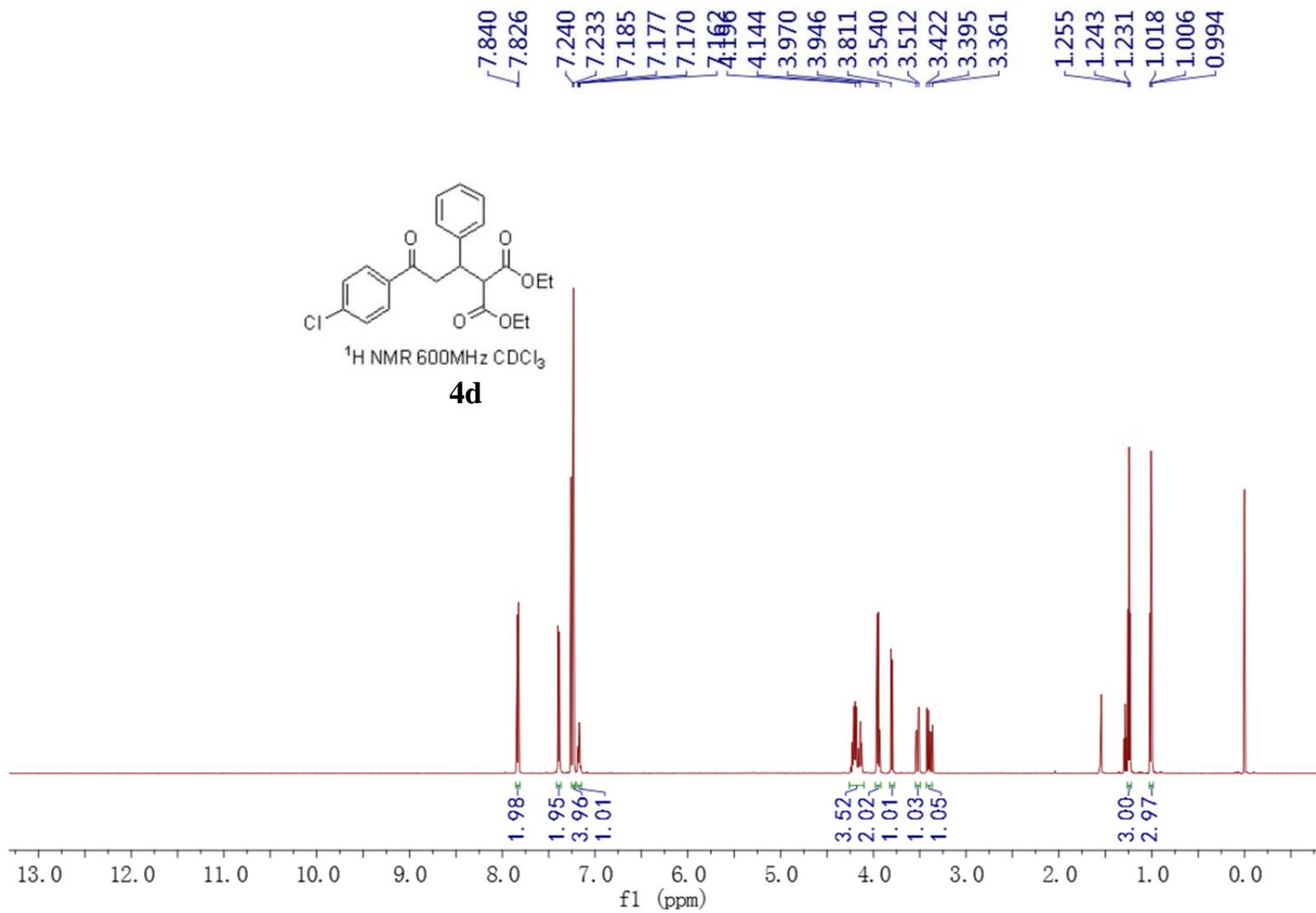
¹H NMR 400MHz CDCl₃

4a

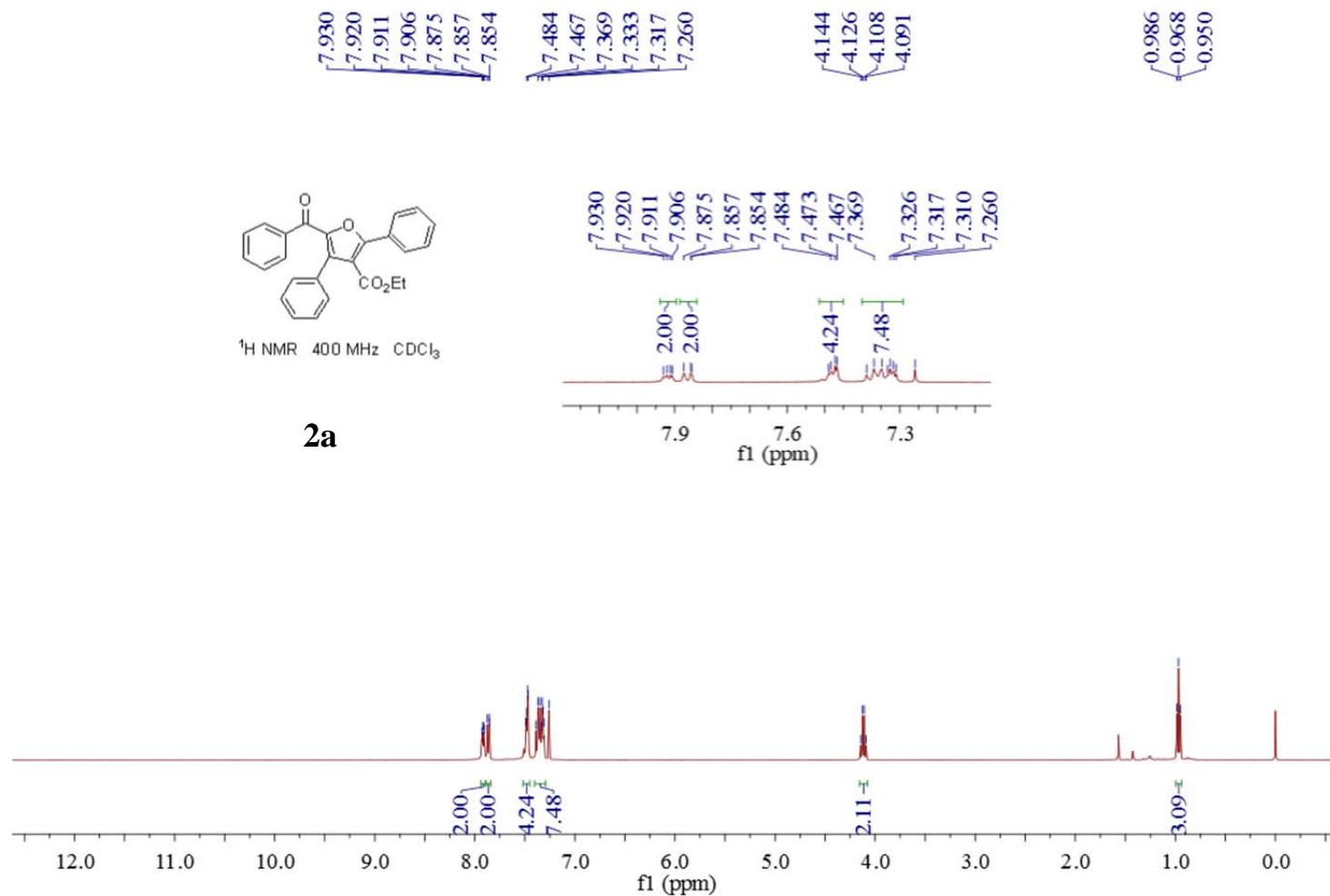


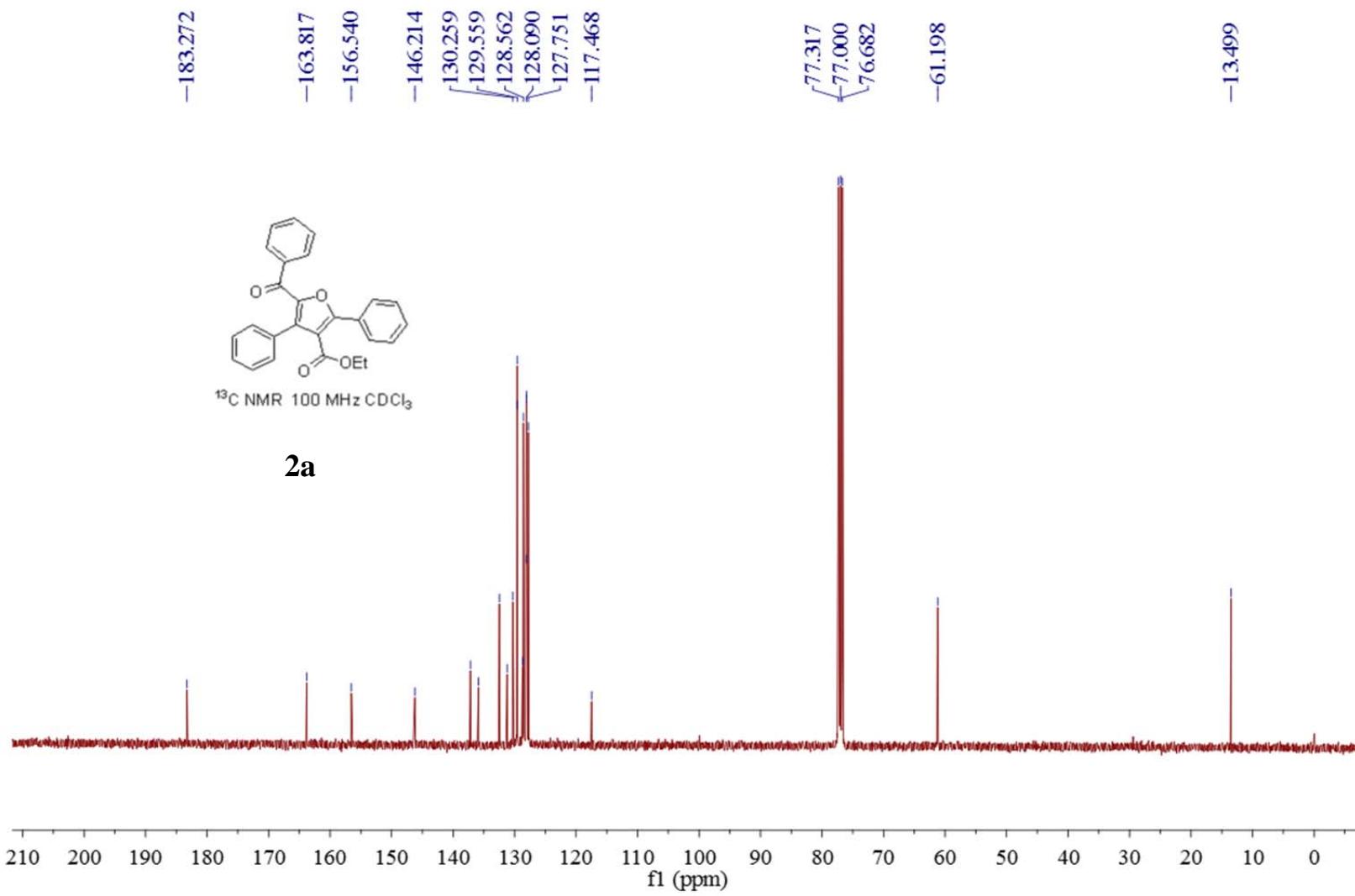


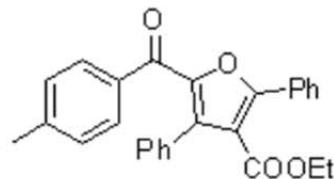




NMR Spectra of Products

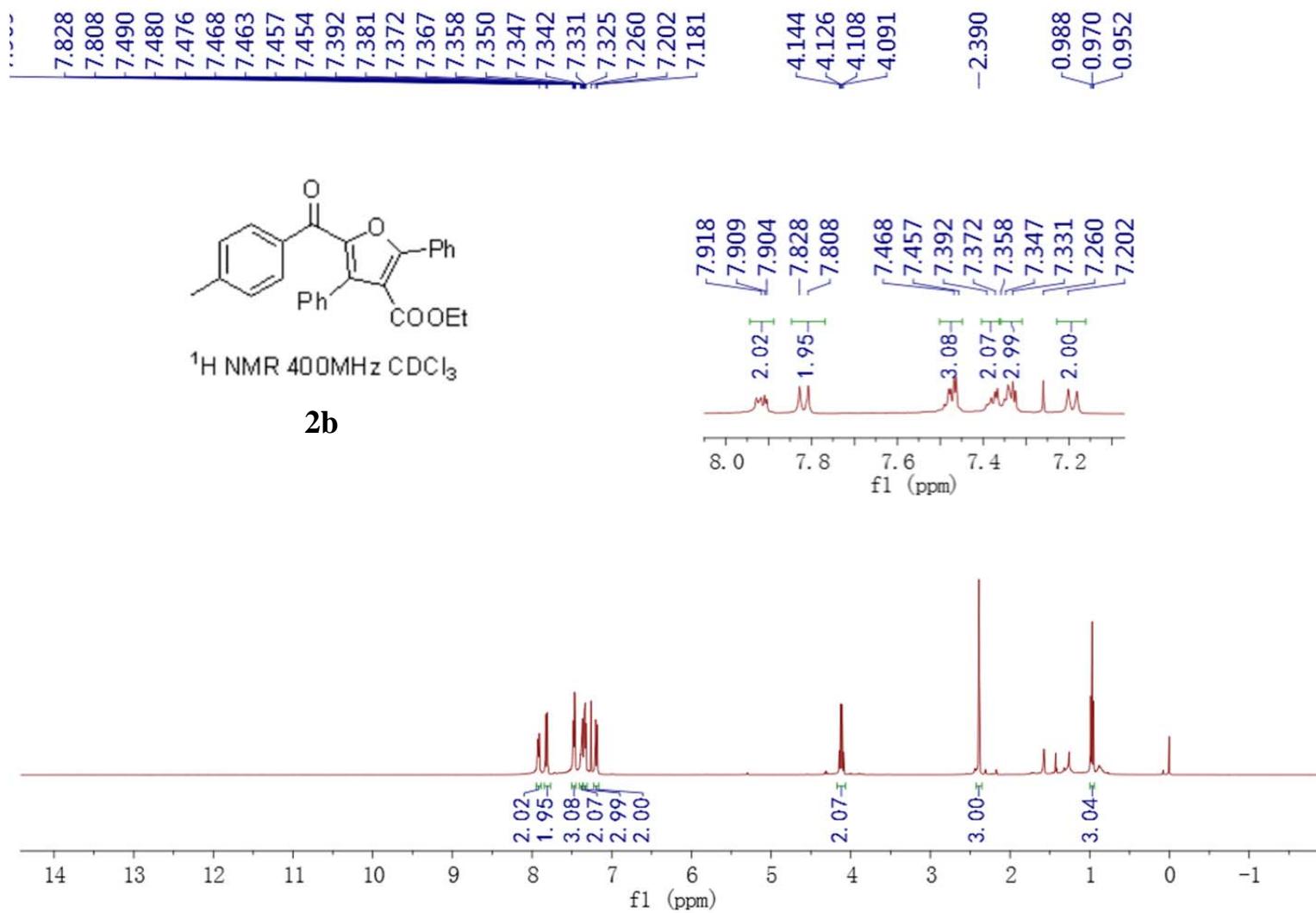


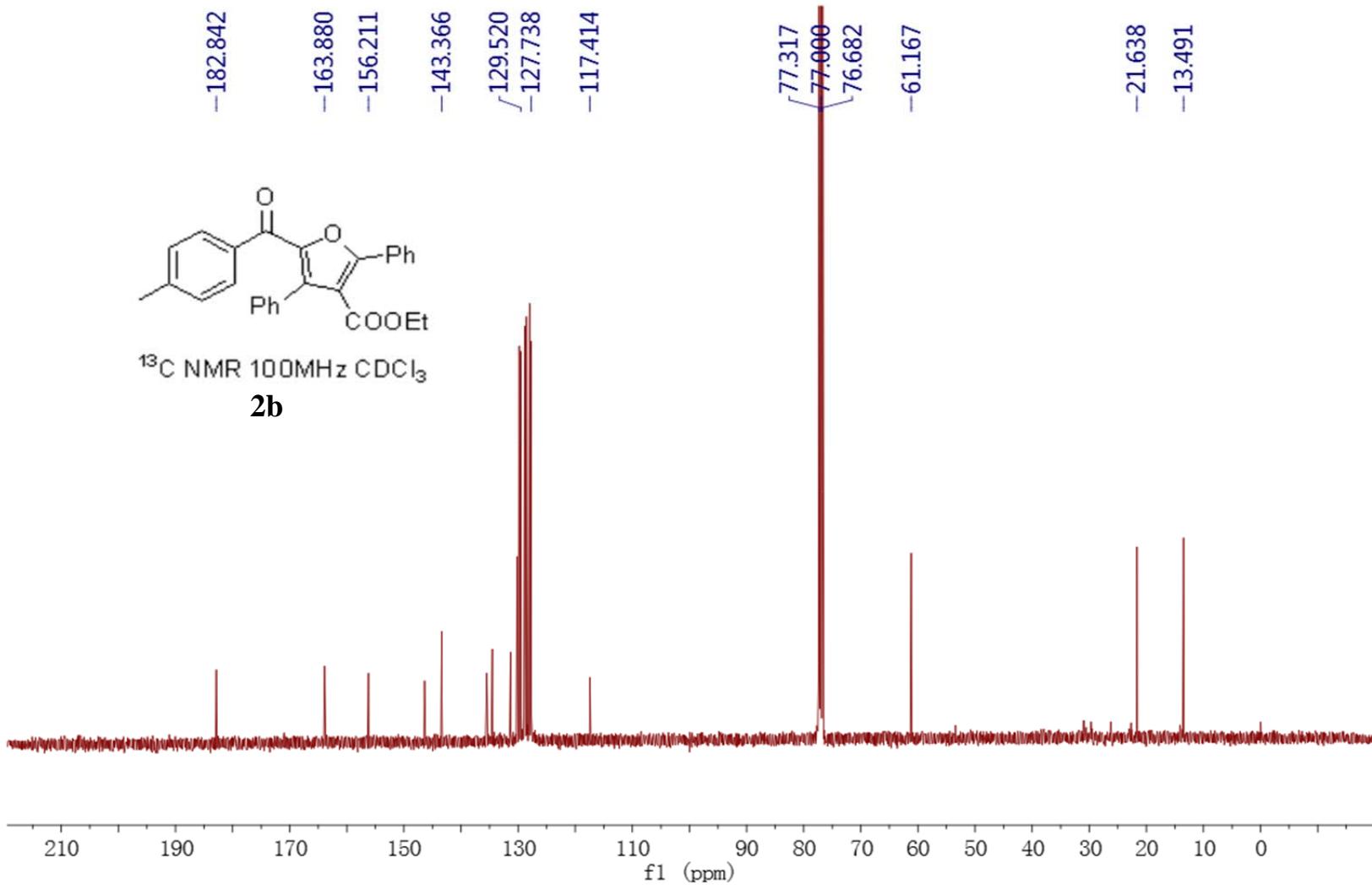


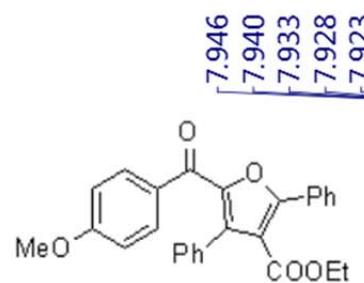


¹H NMR 400MHz CDCl₃

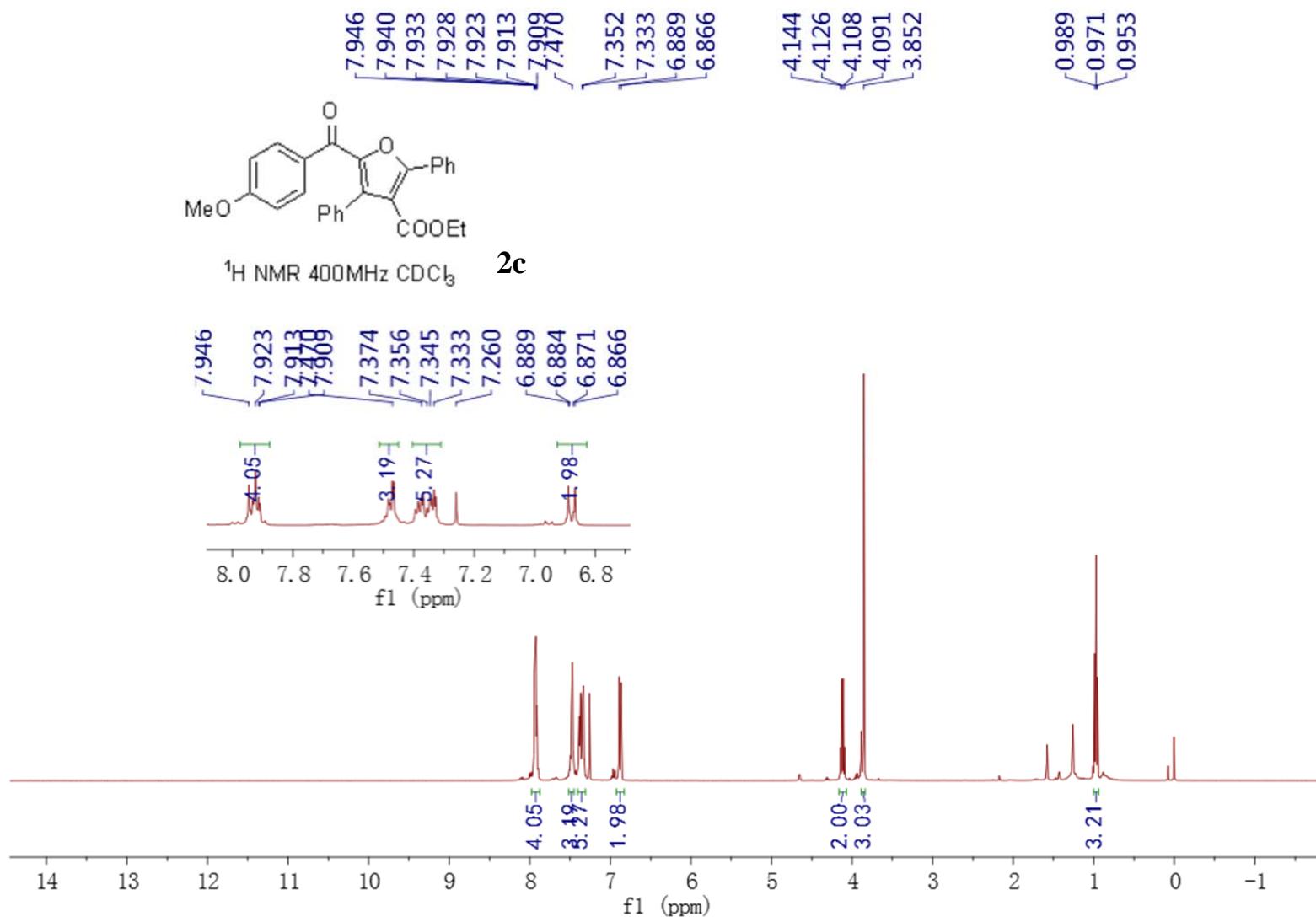
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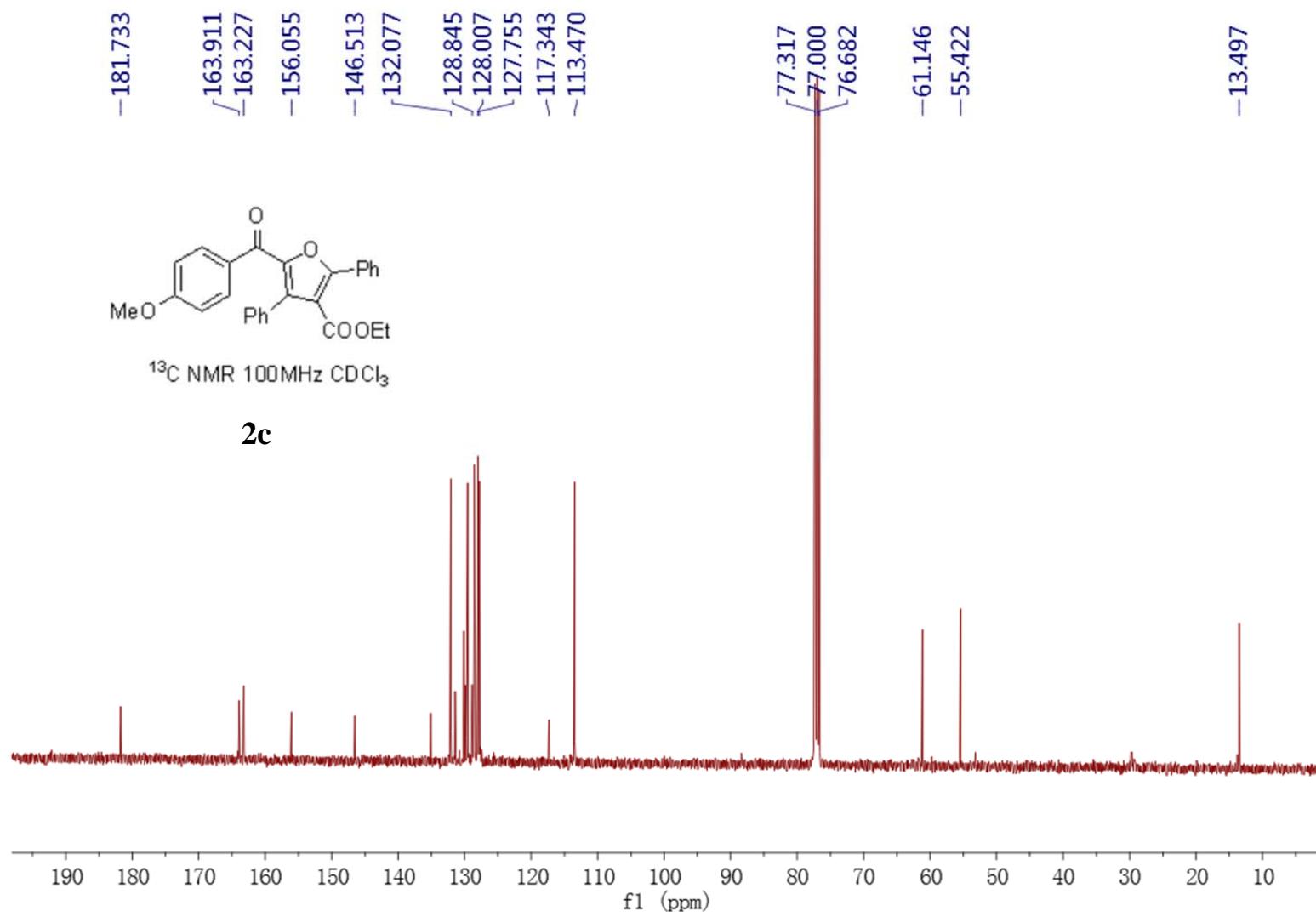


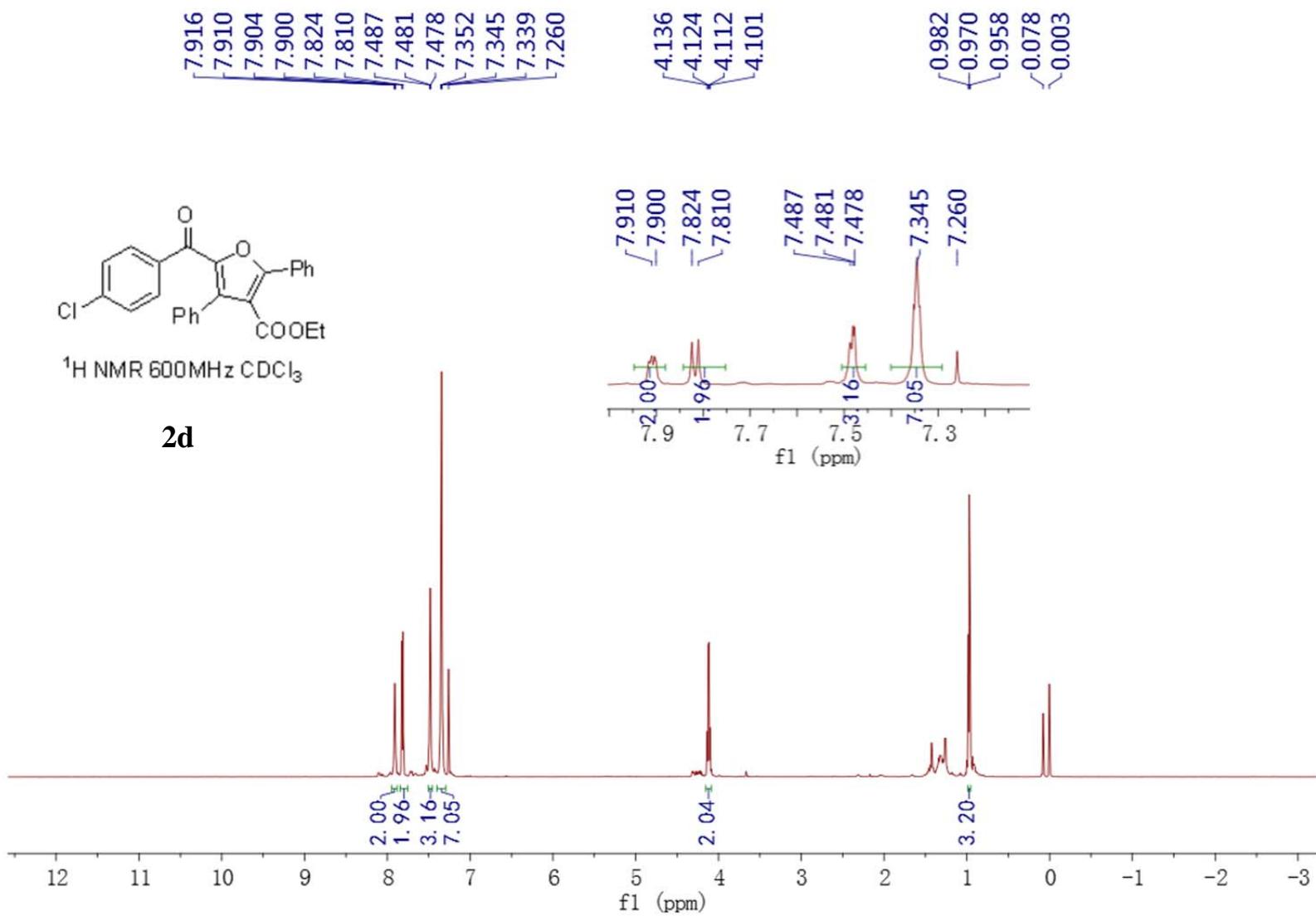


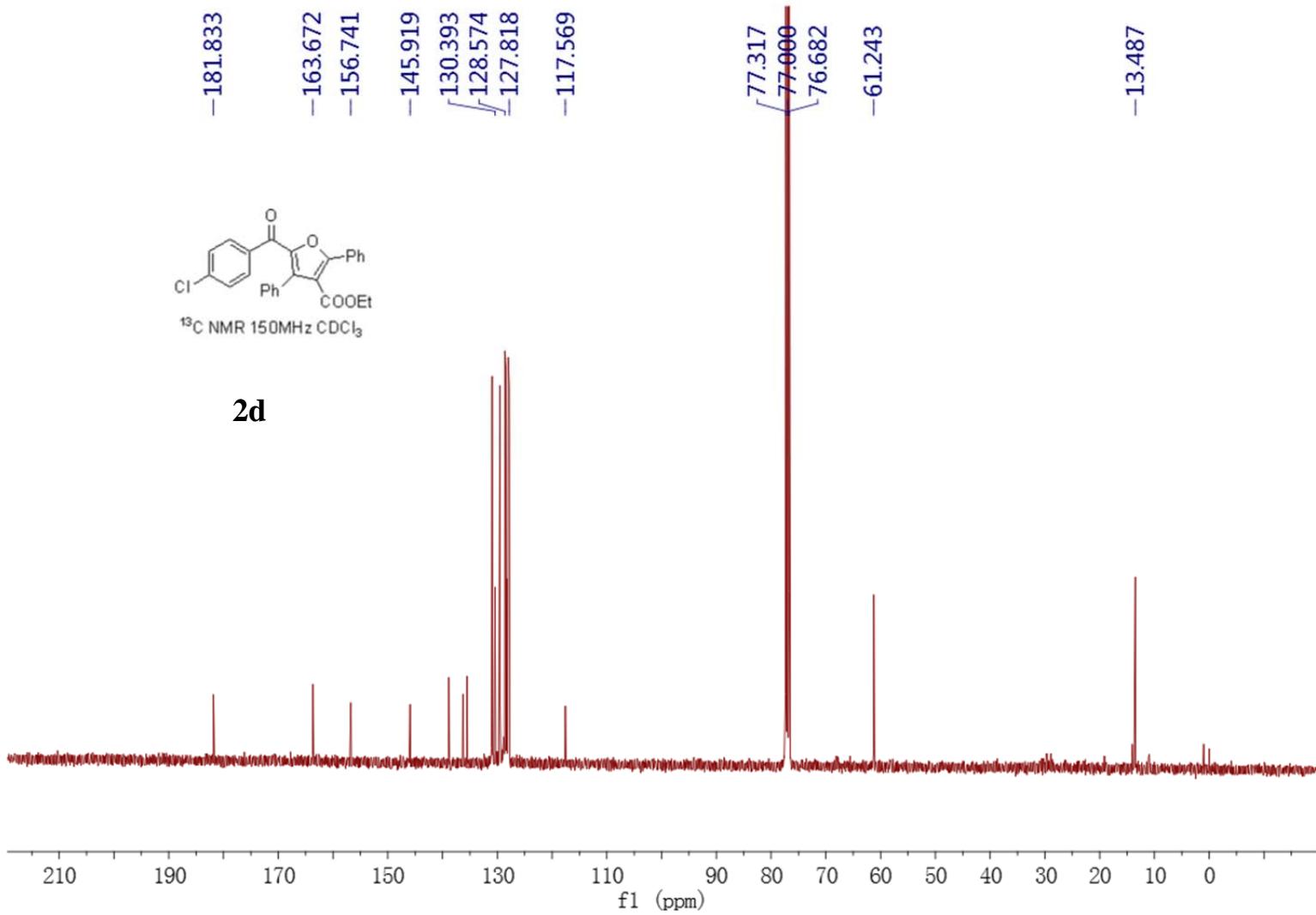


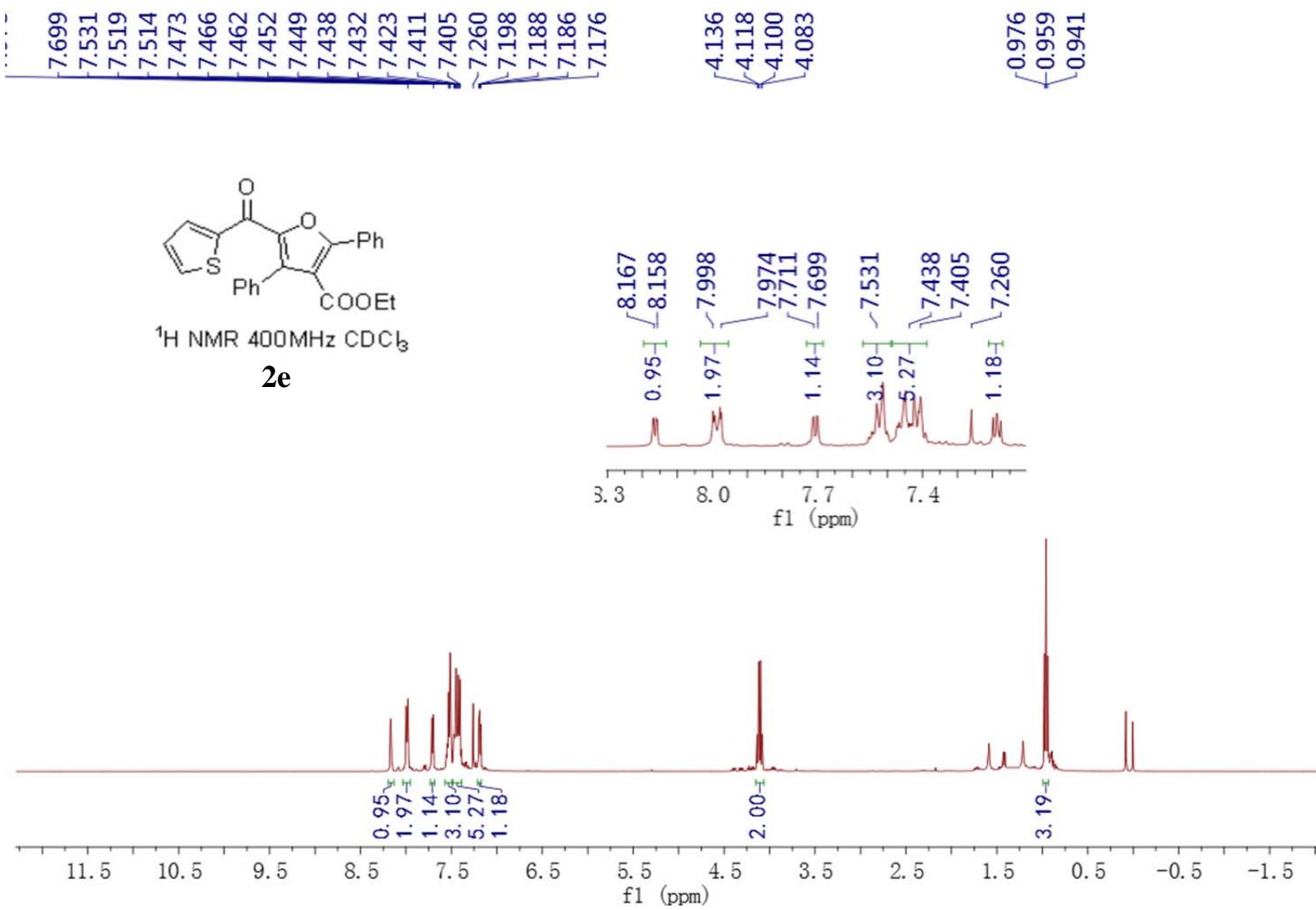
¹H NMR 400MHz CDCl₃ 2c

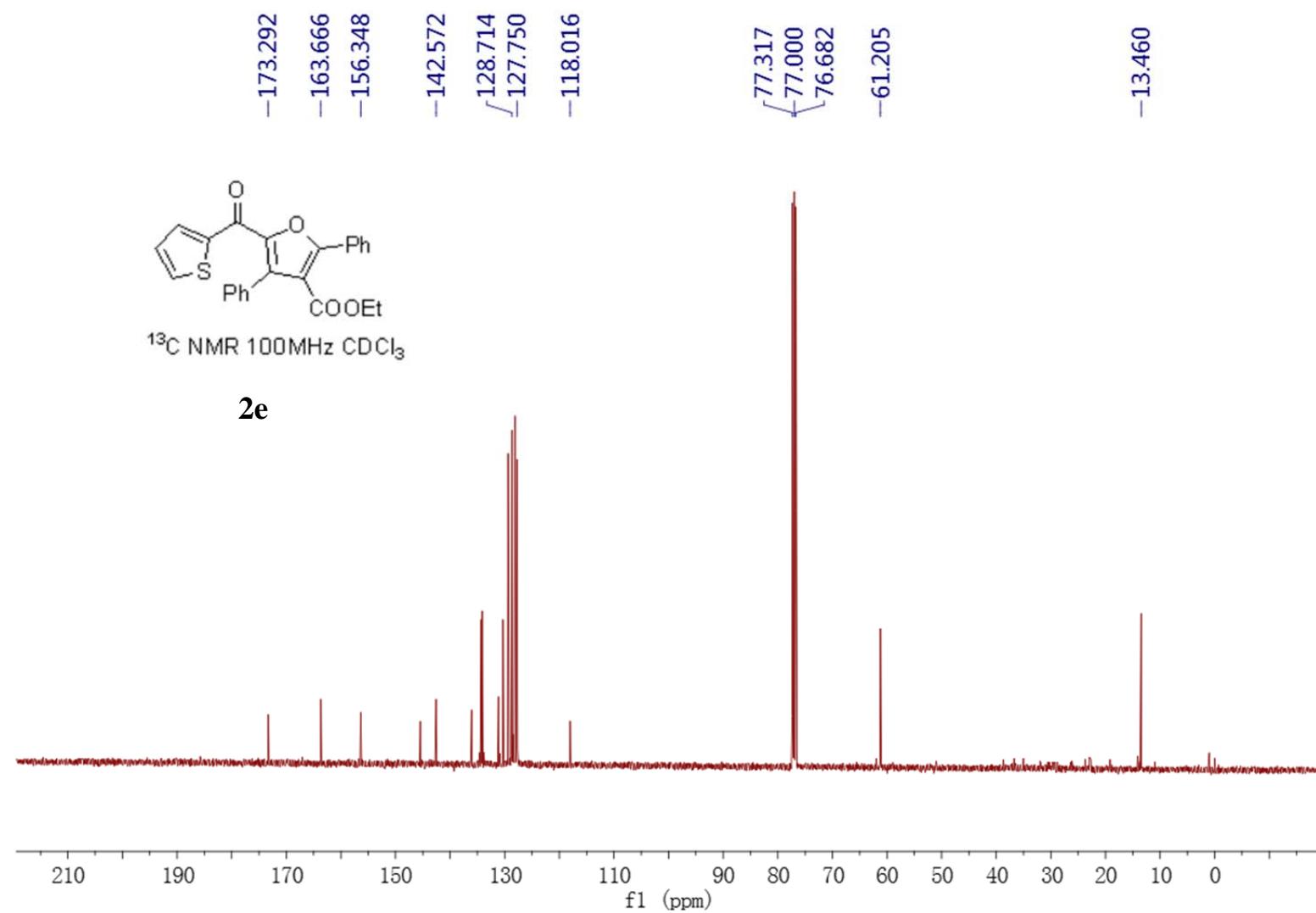


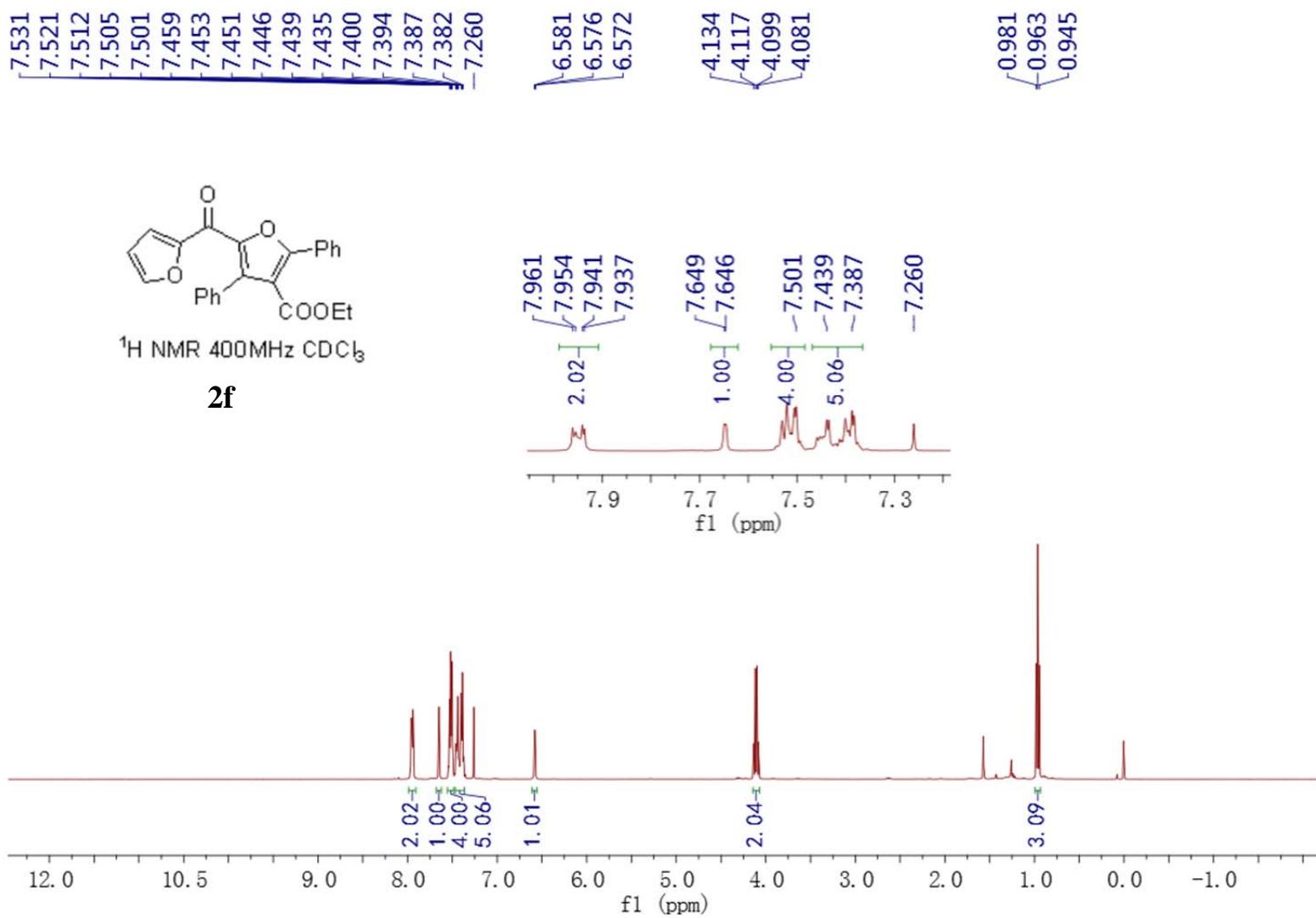


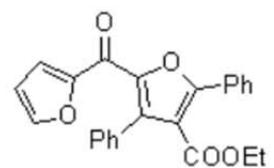






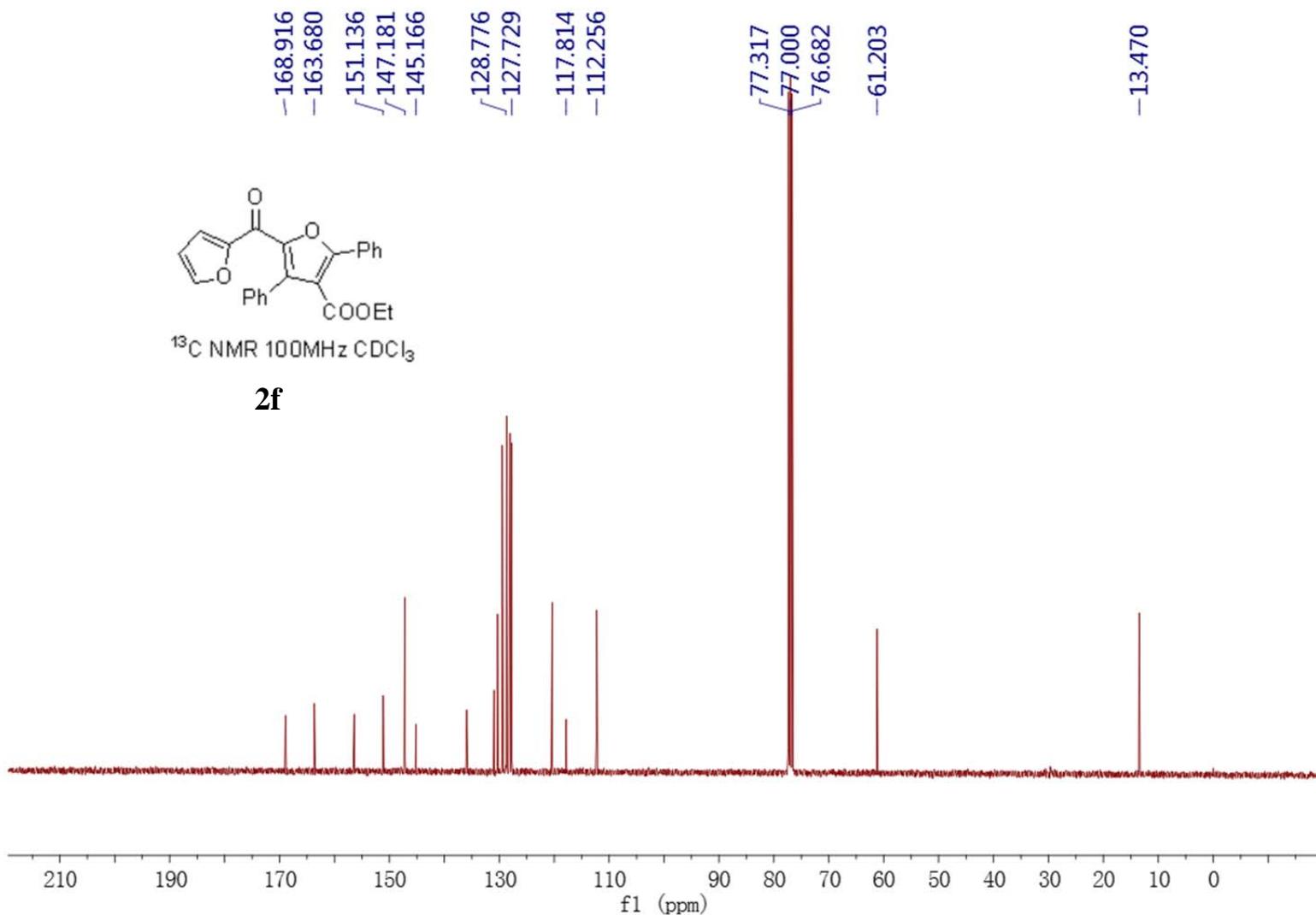


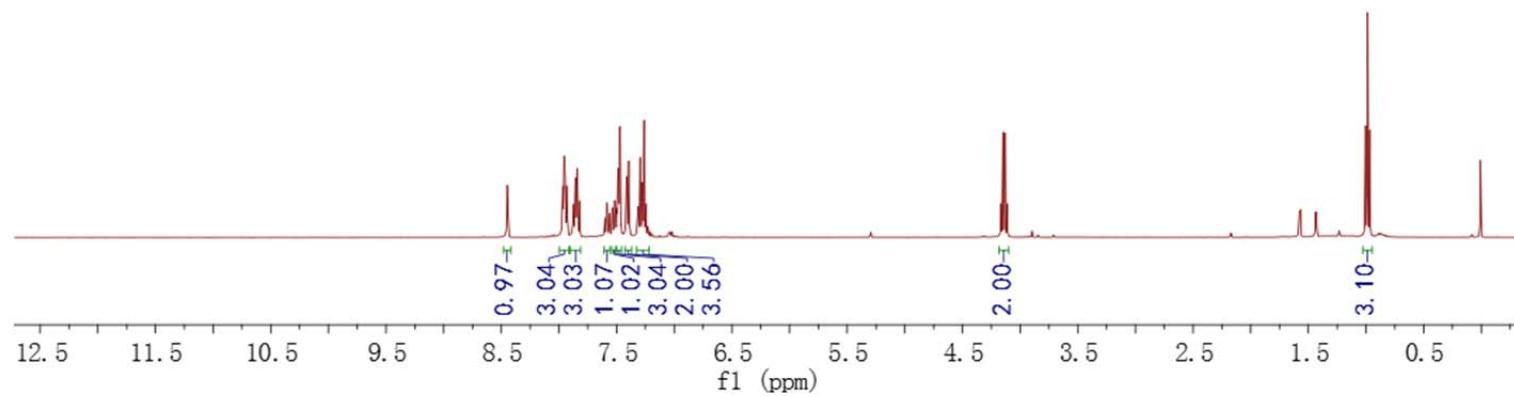
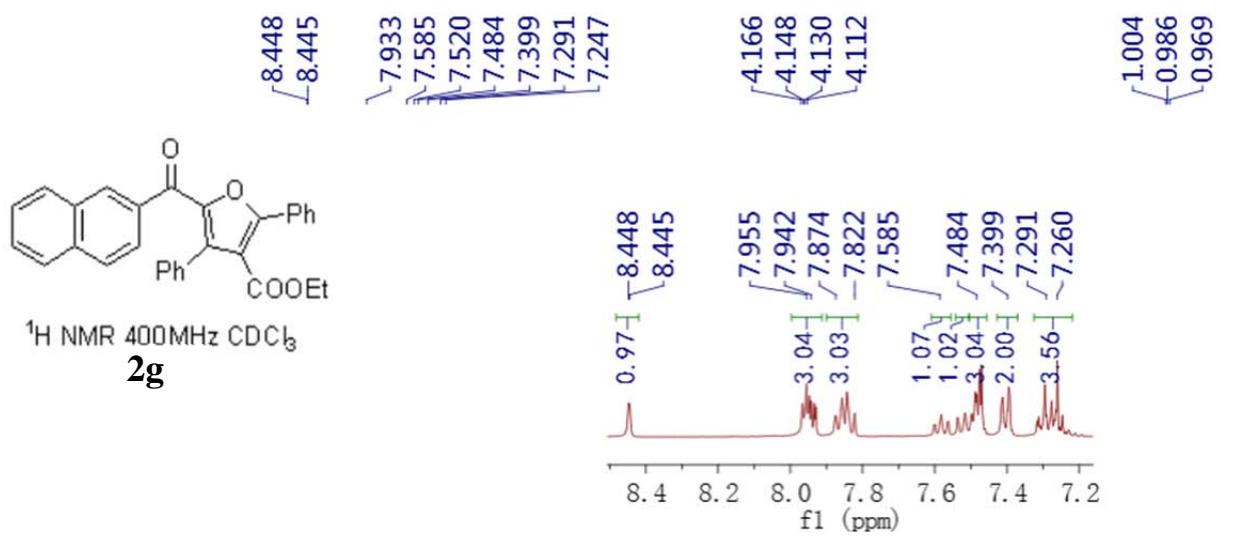


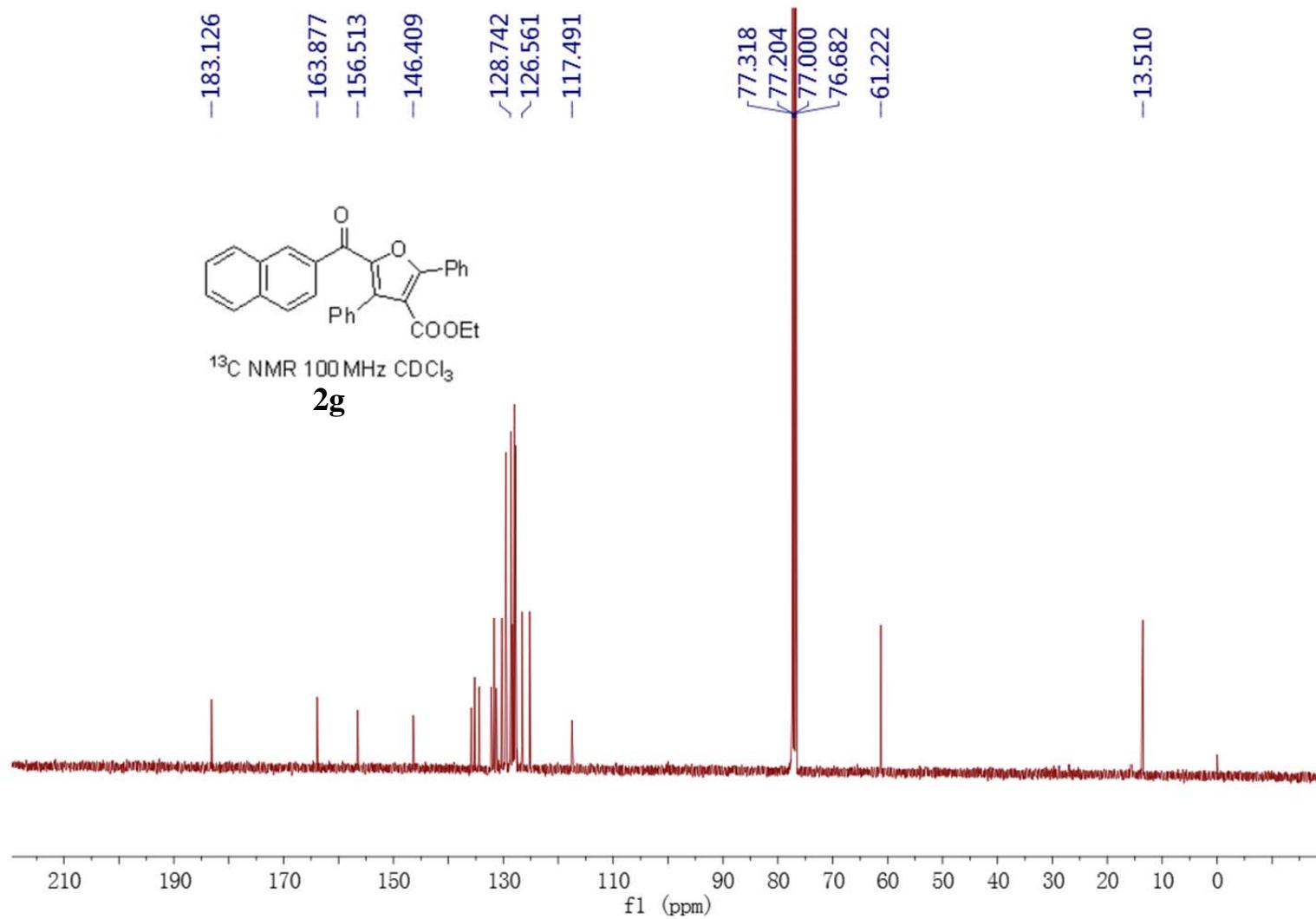


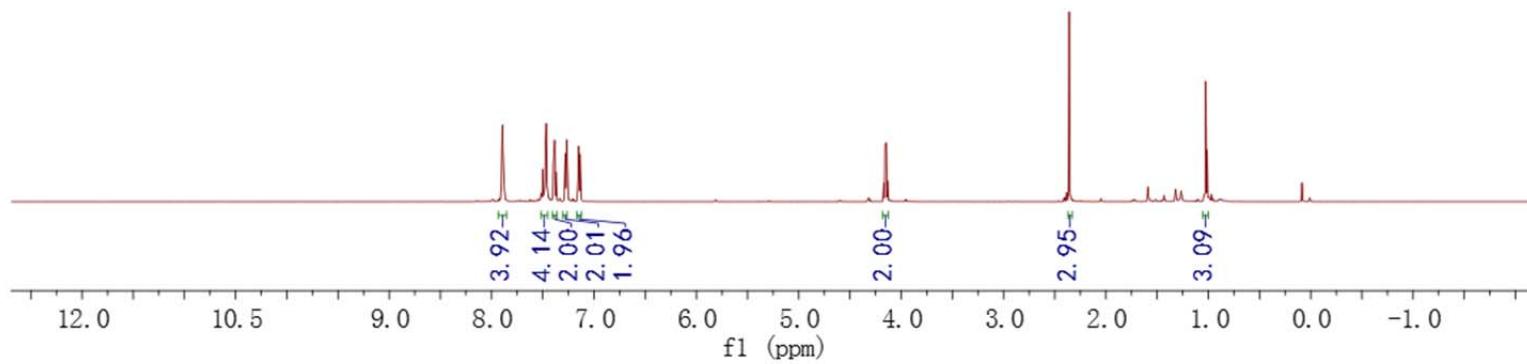
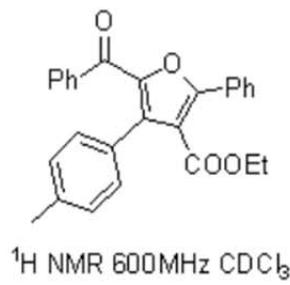
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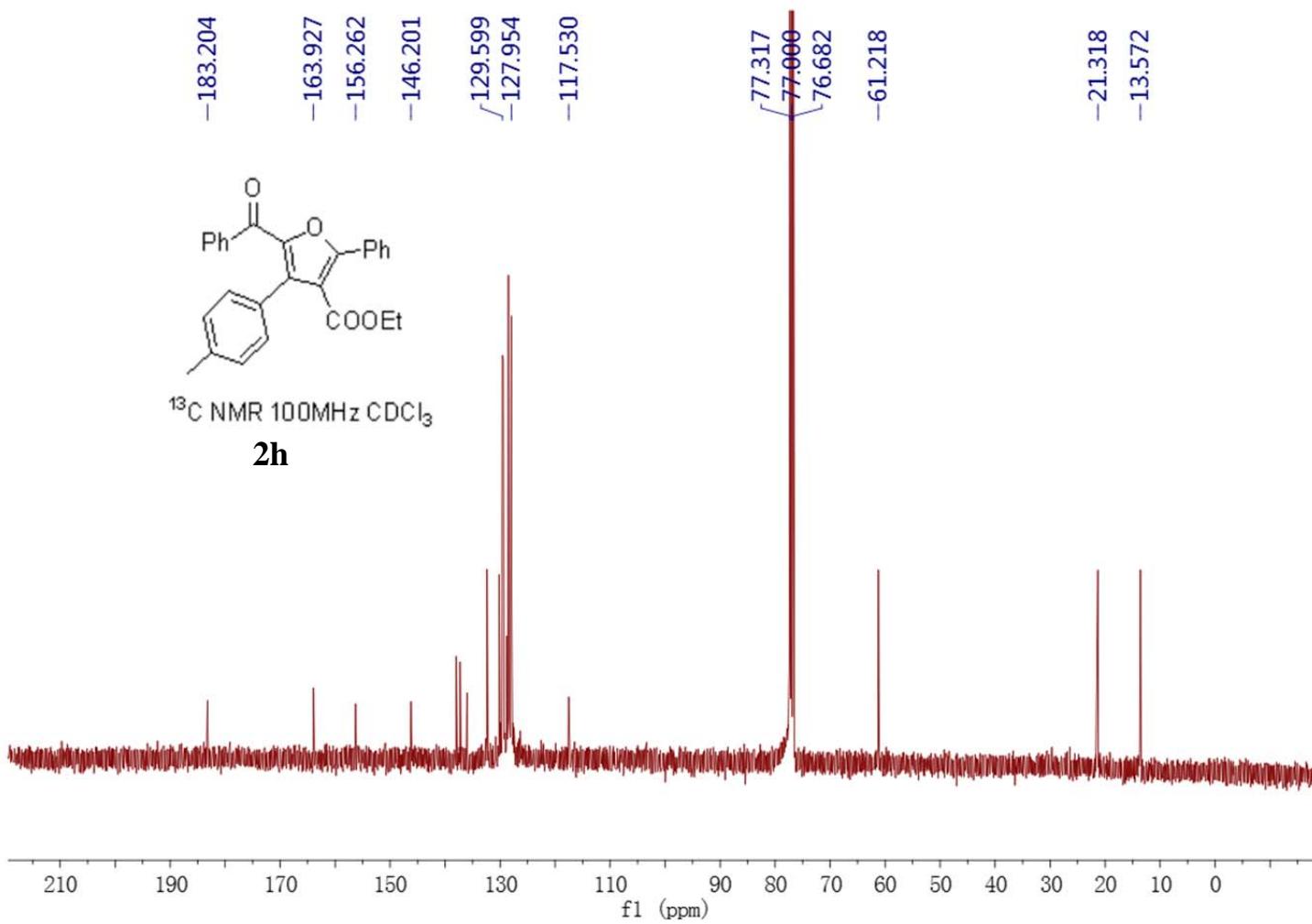
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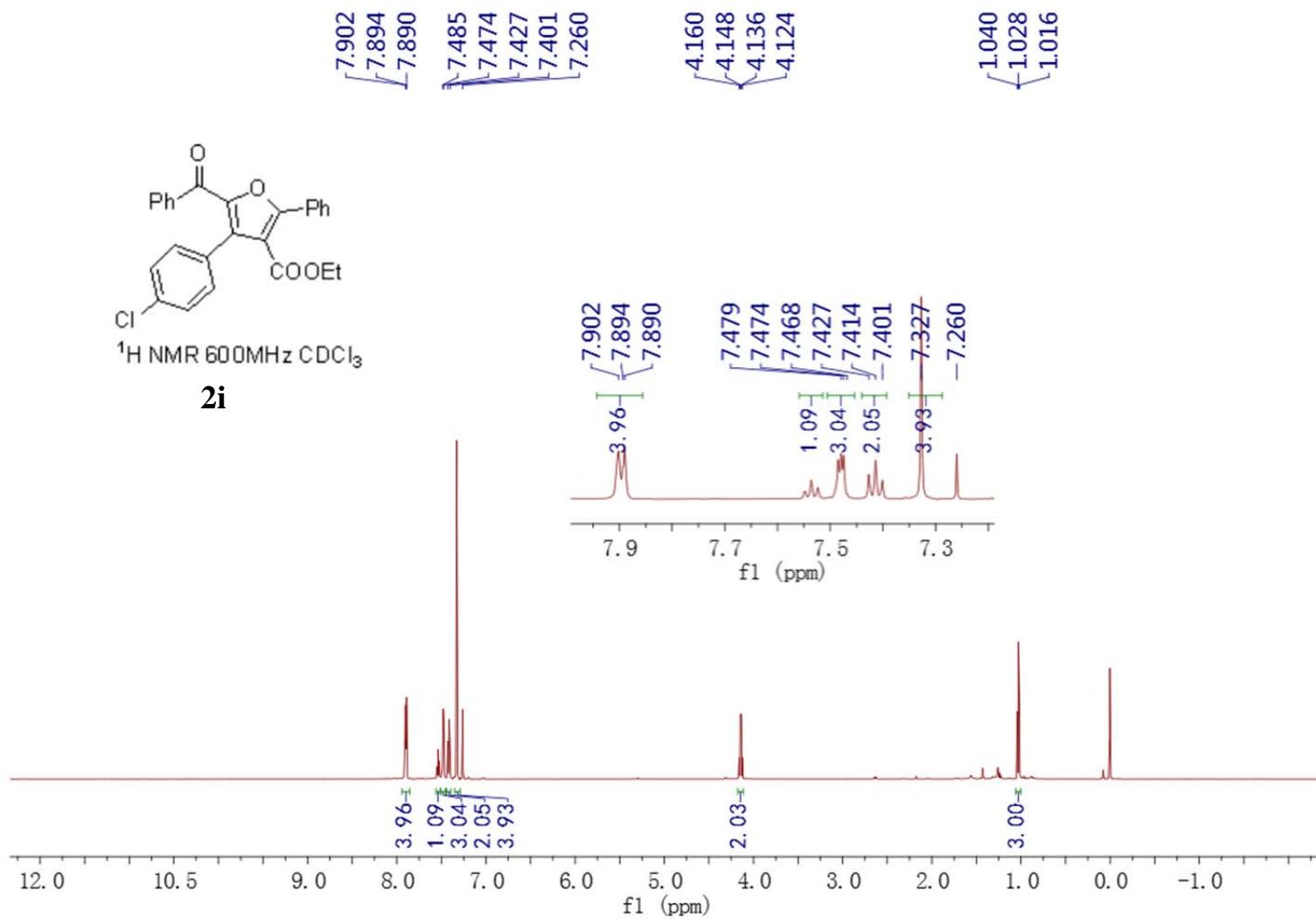


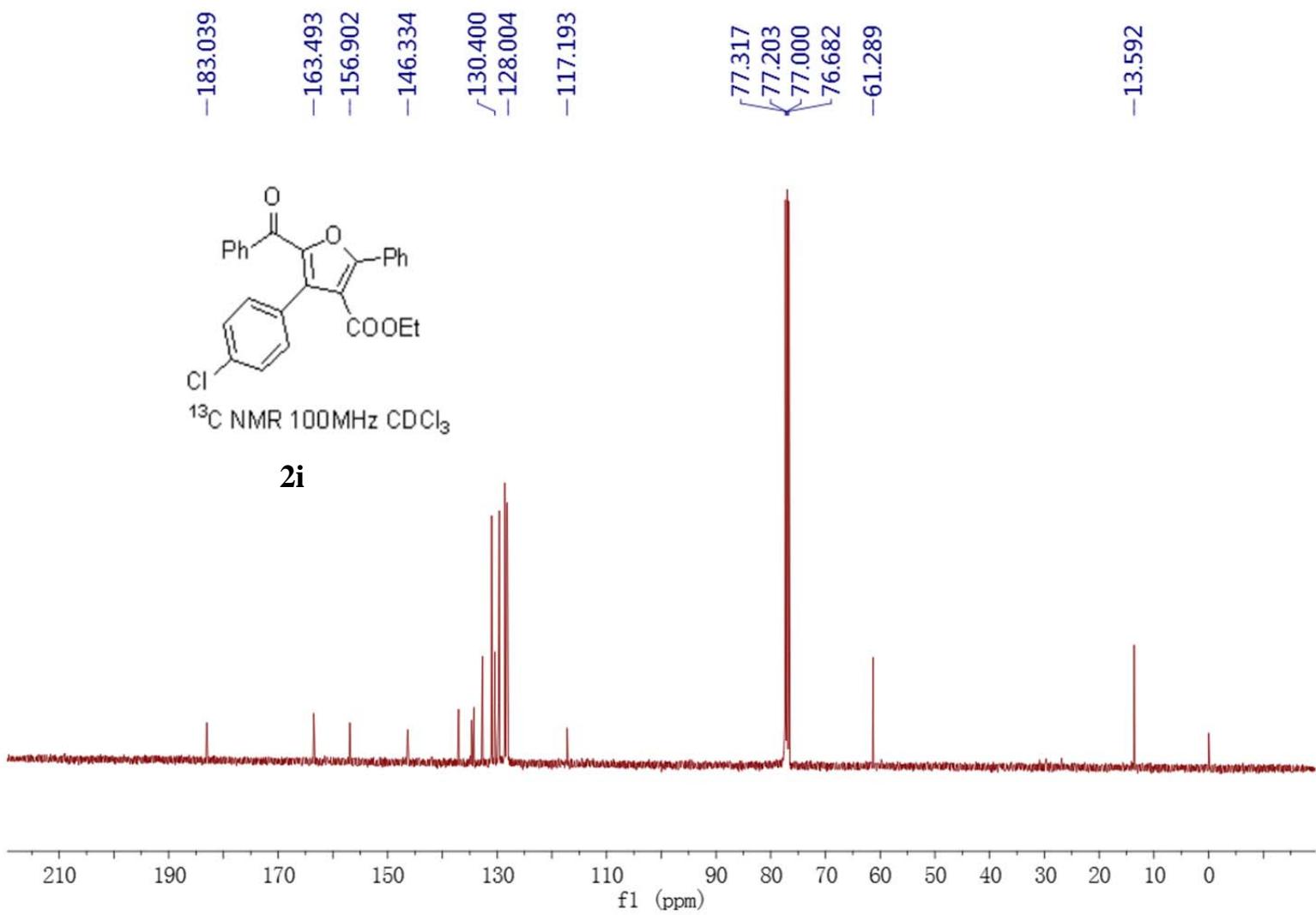


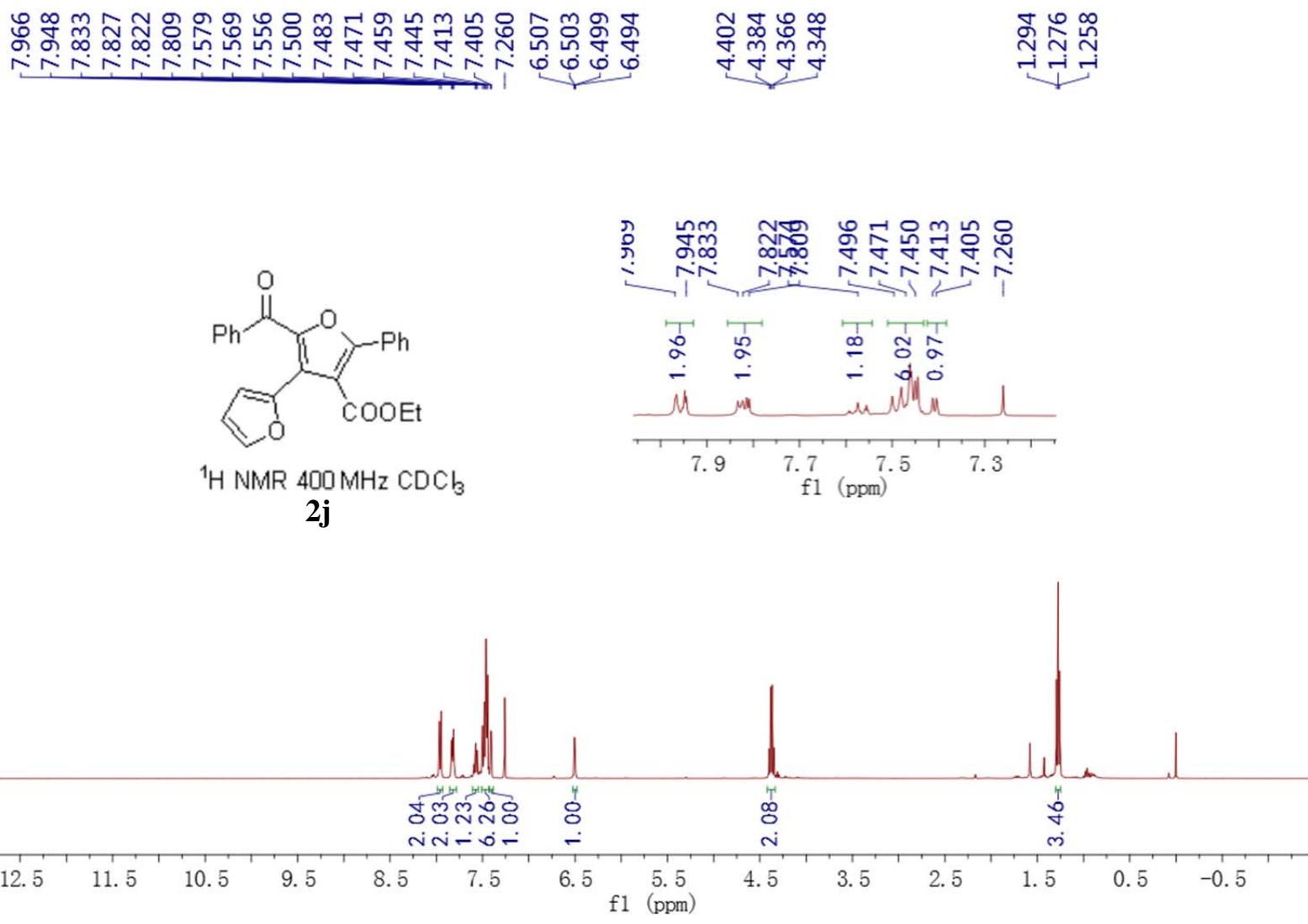


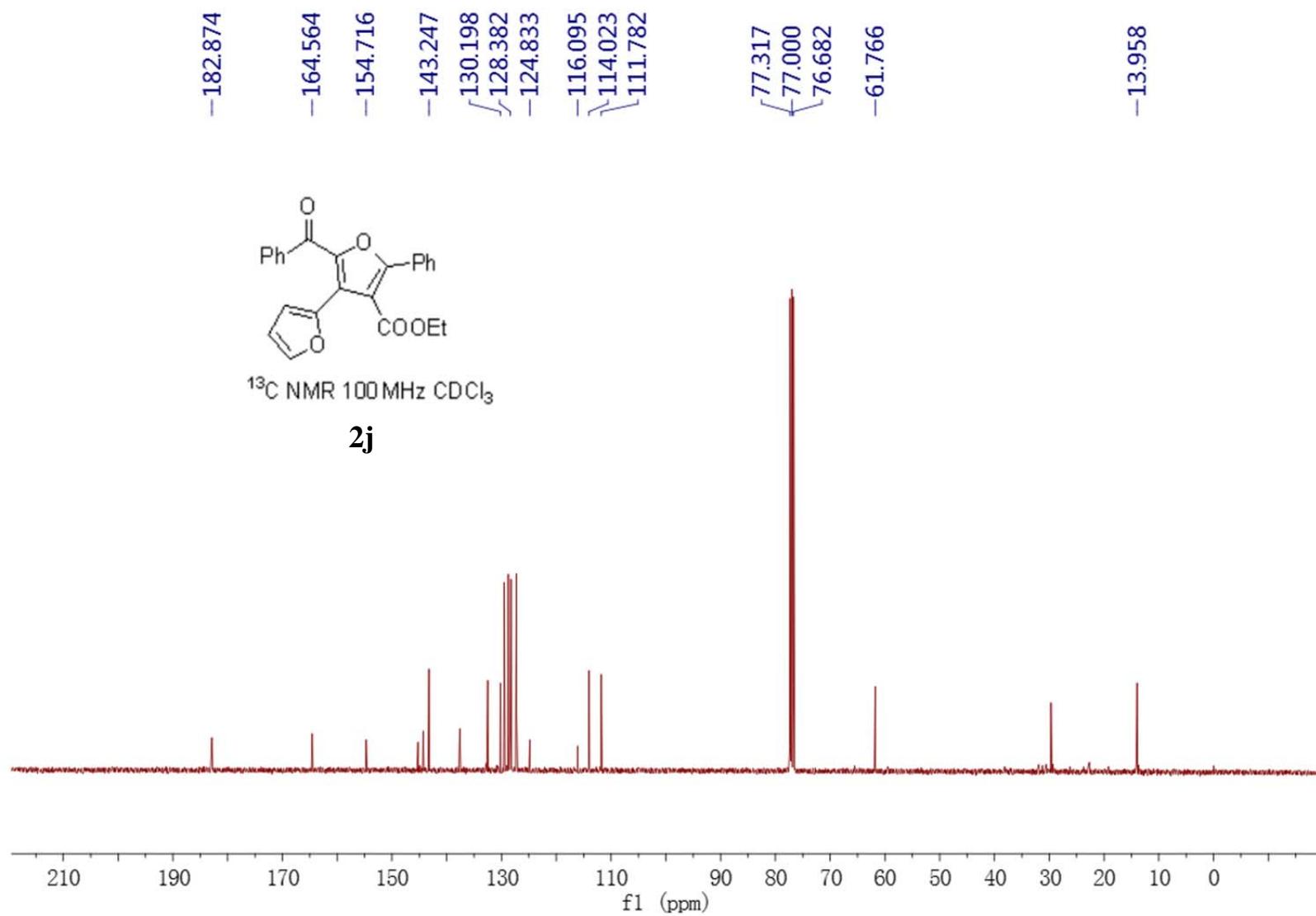


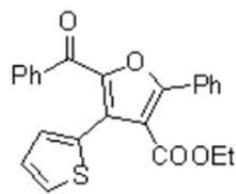






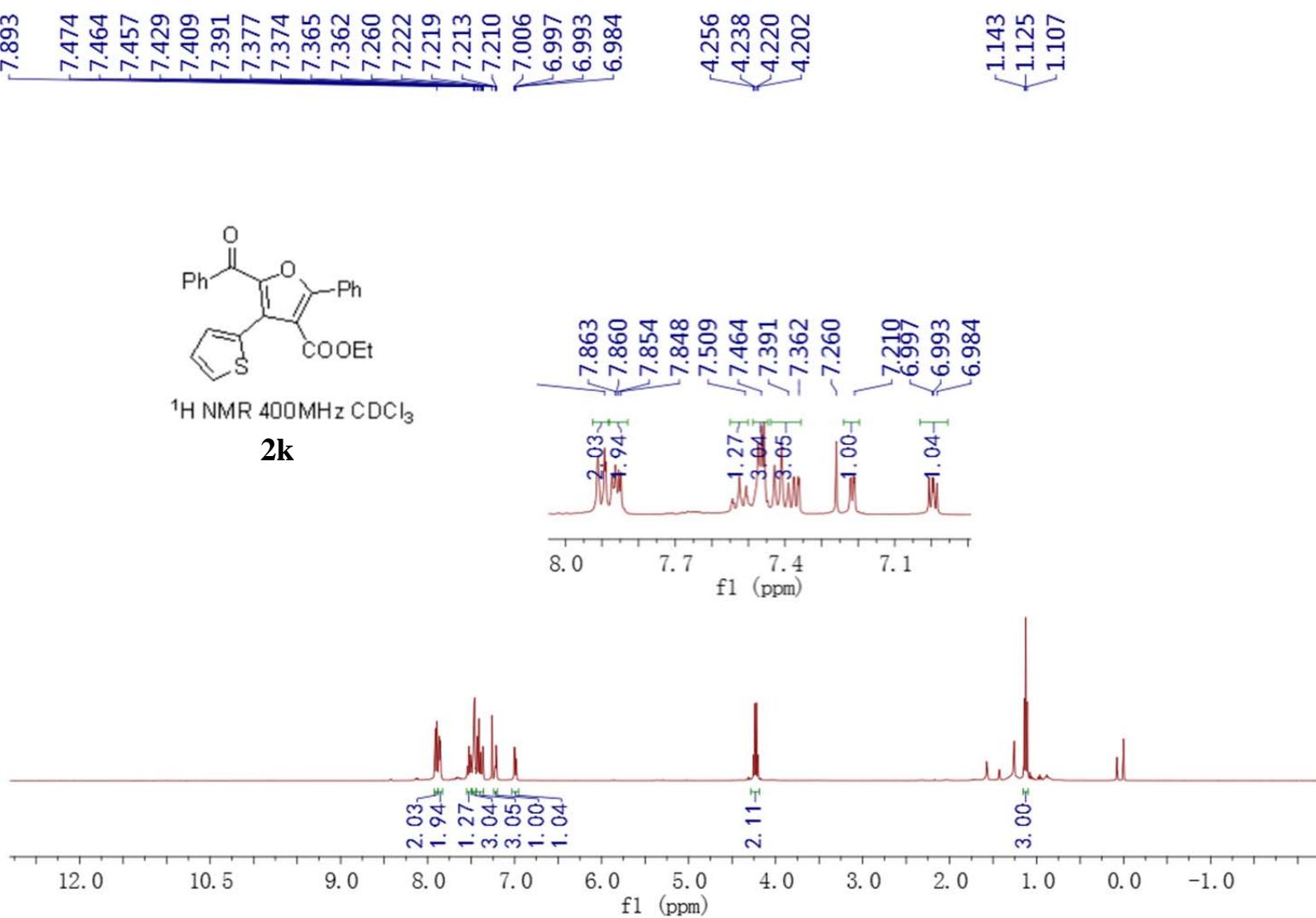


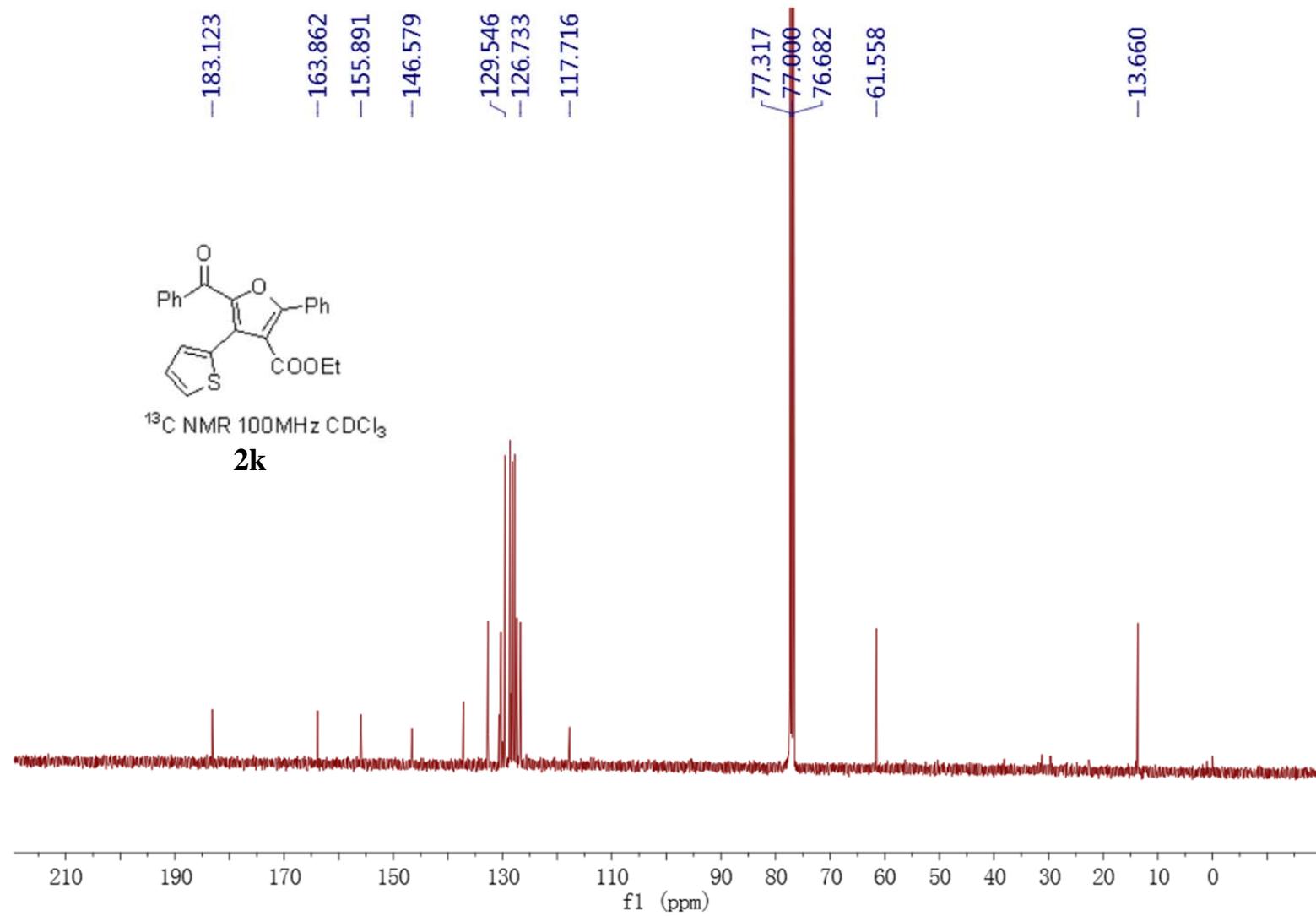


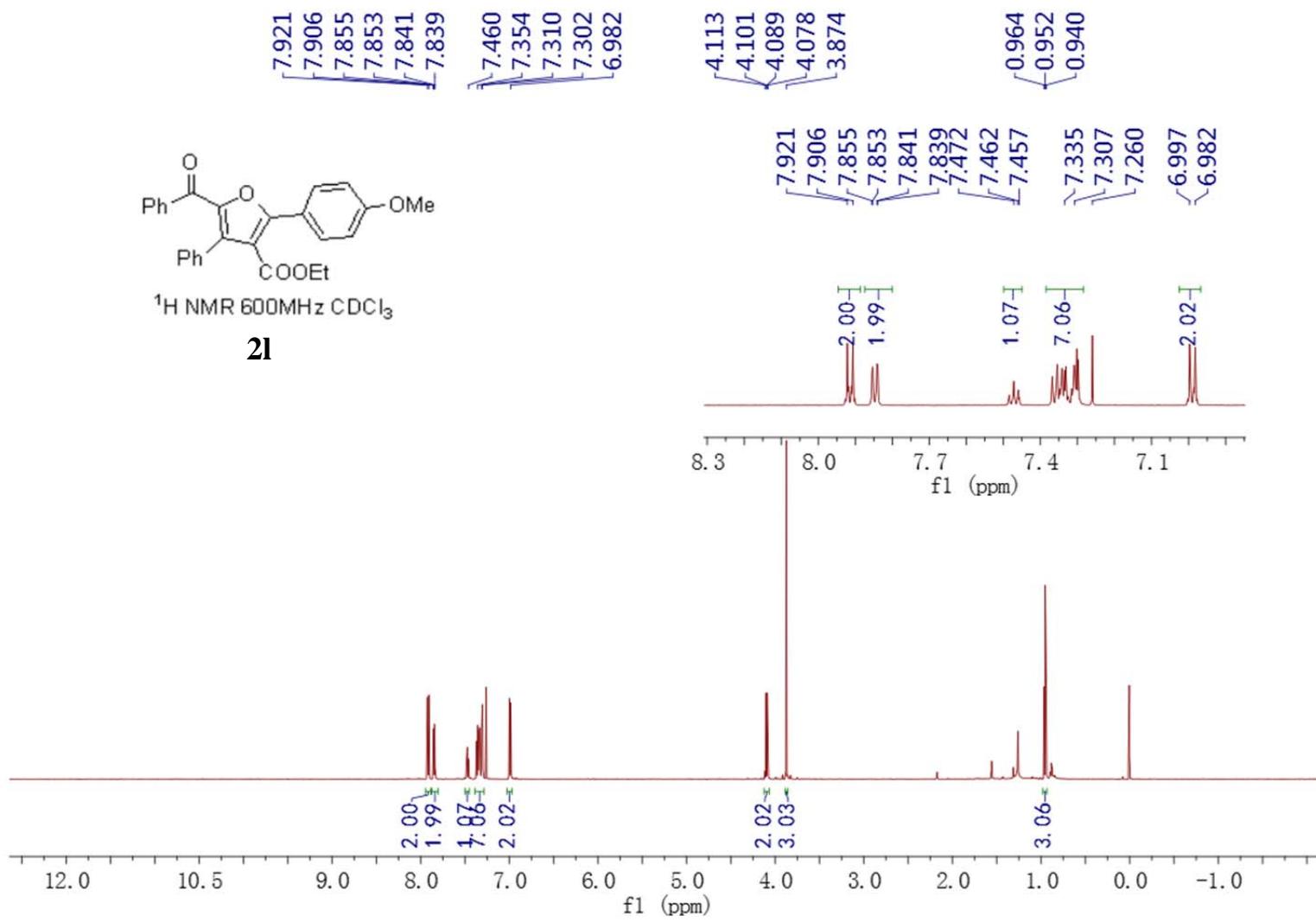


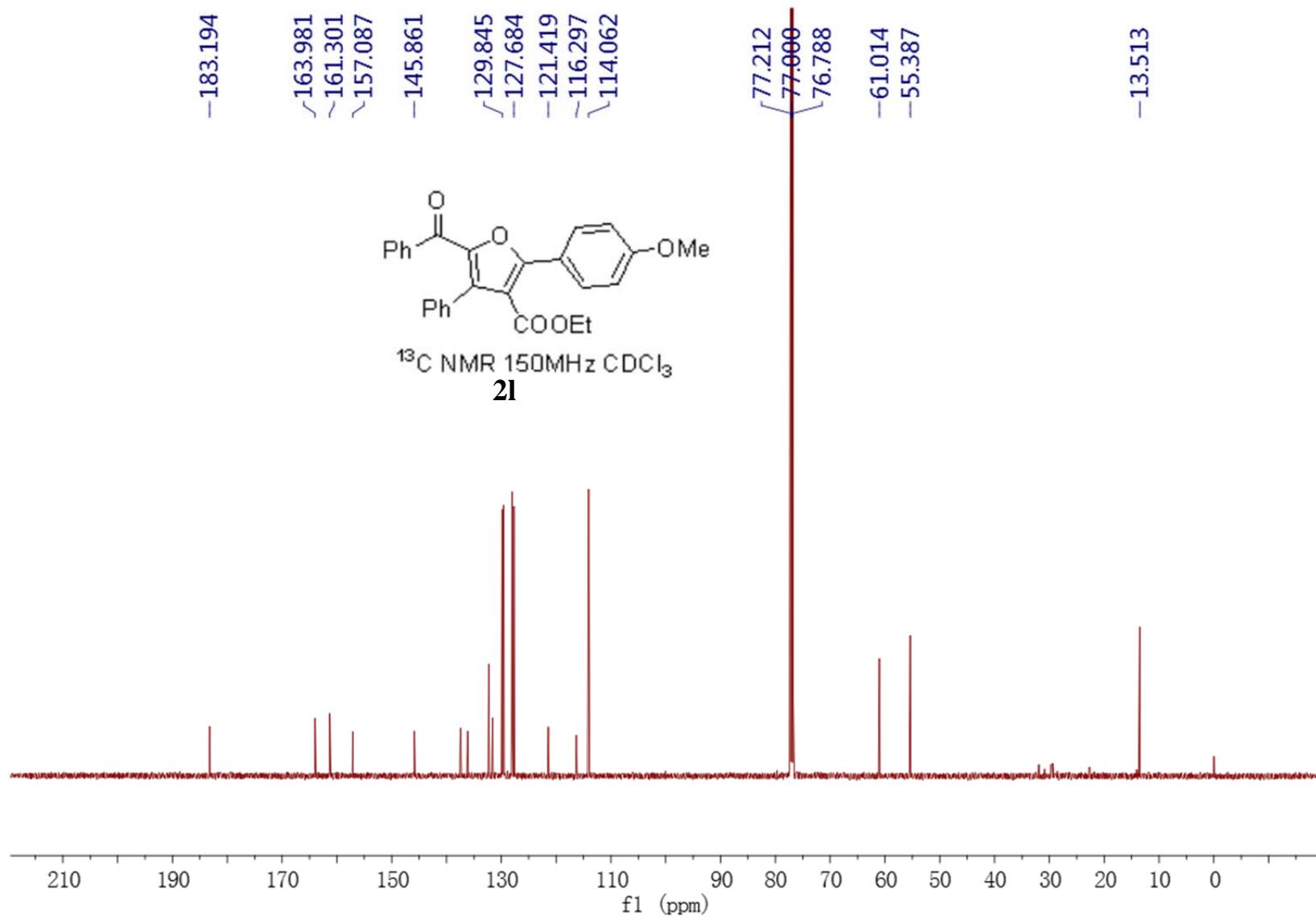
^1H NMR 400MHz CDCl_3

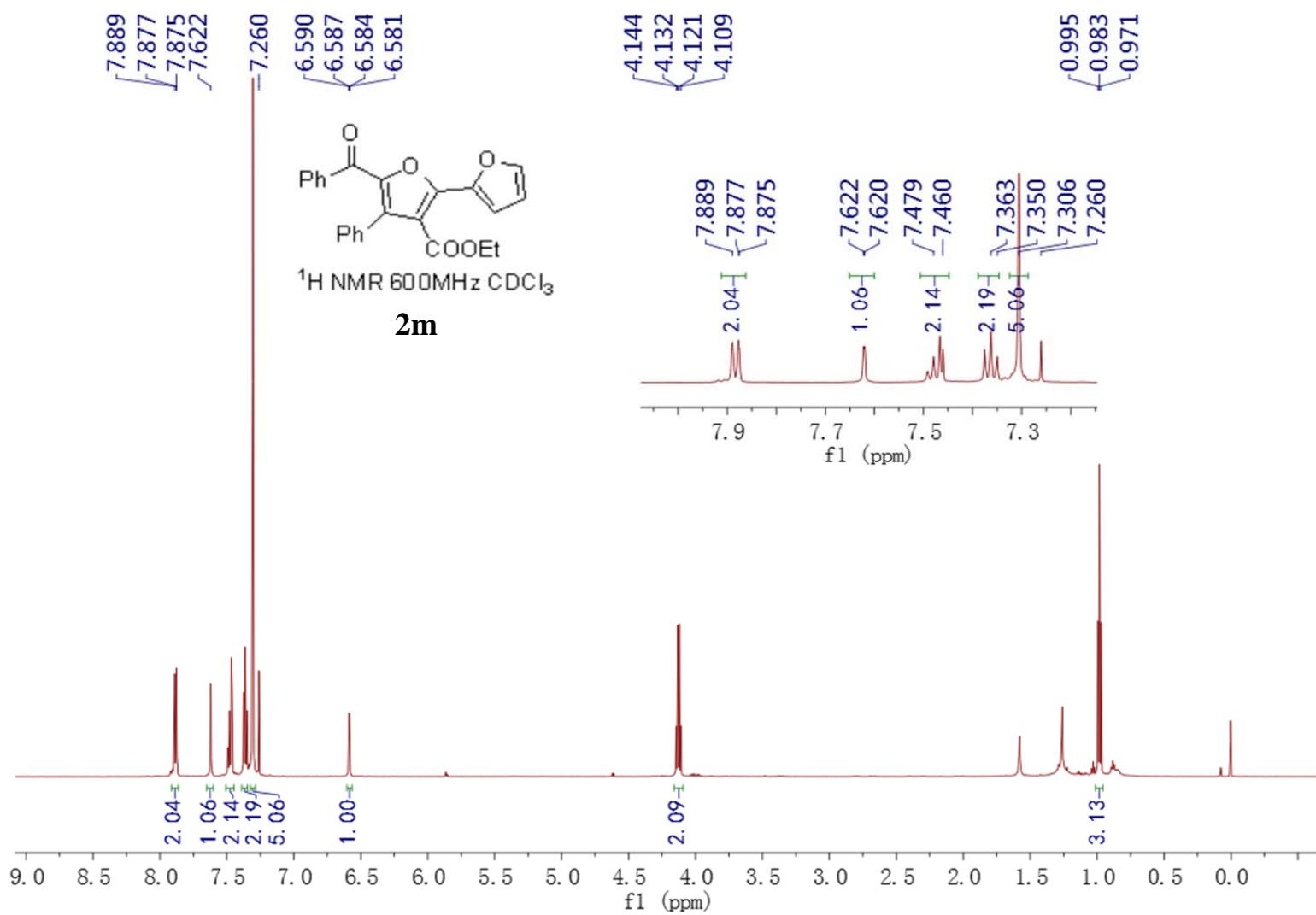
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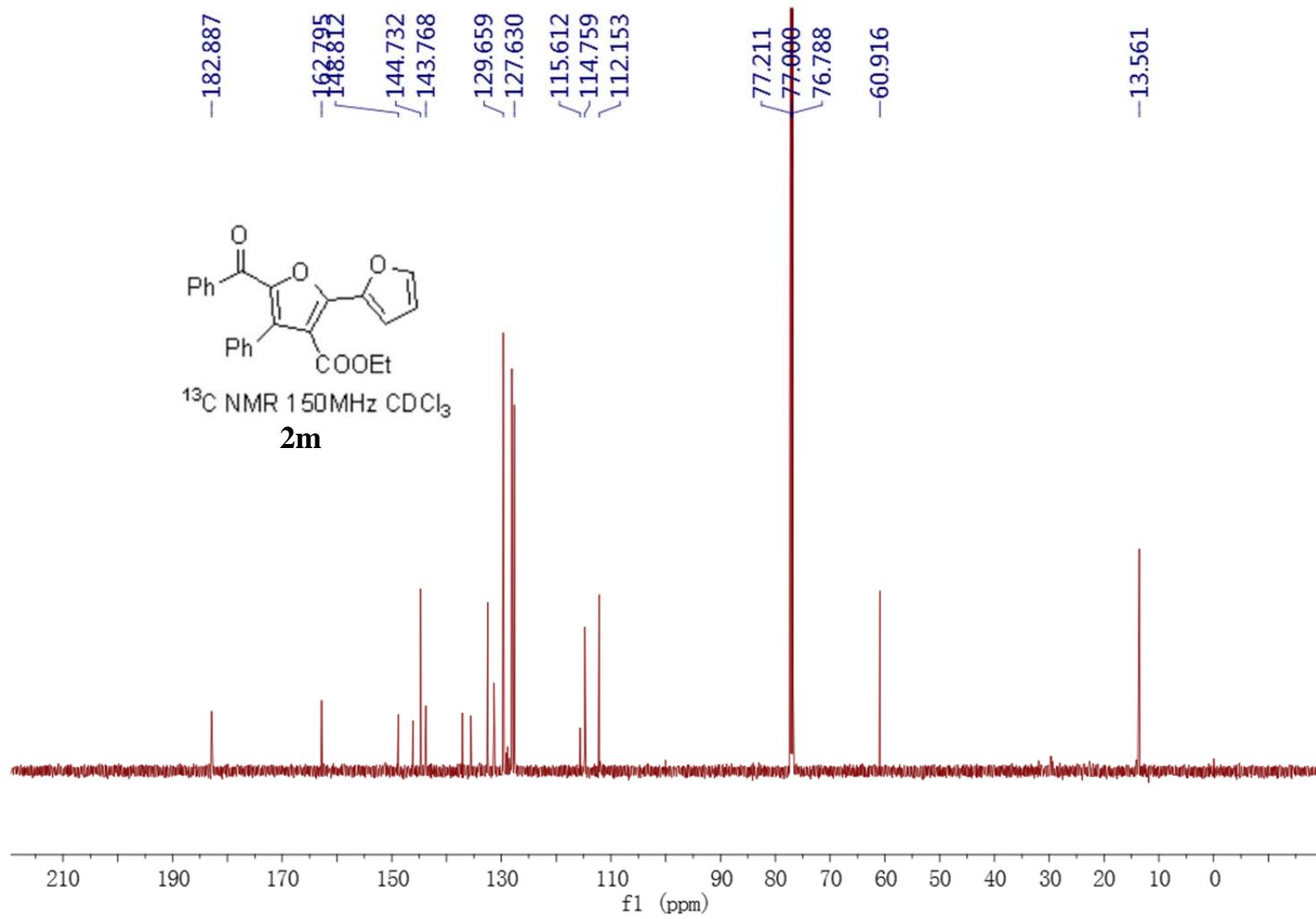


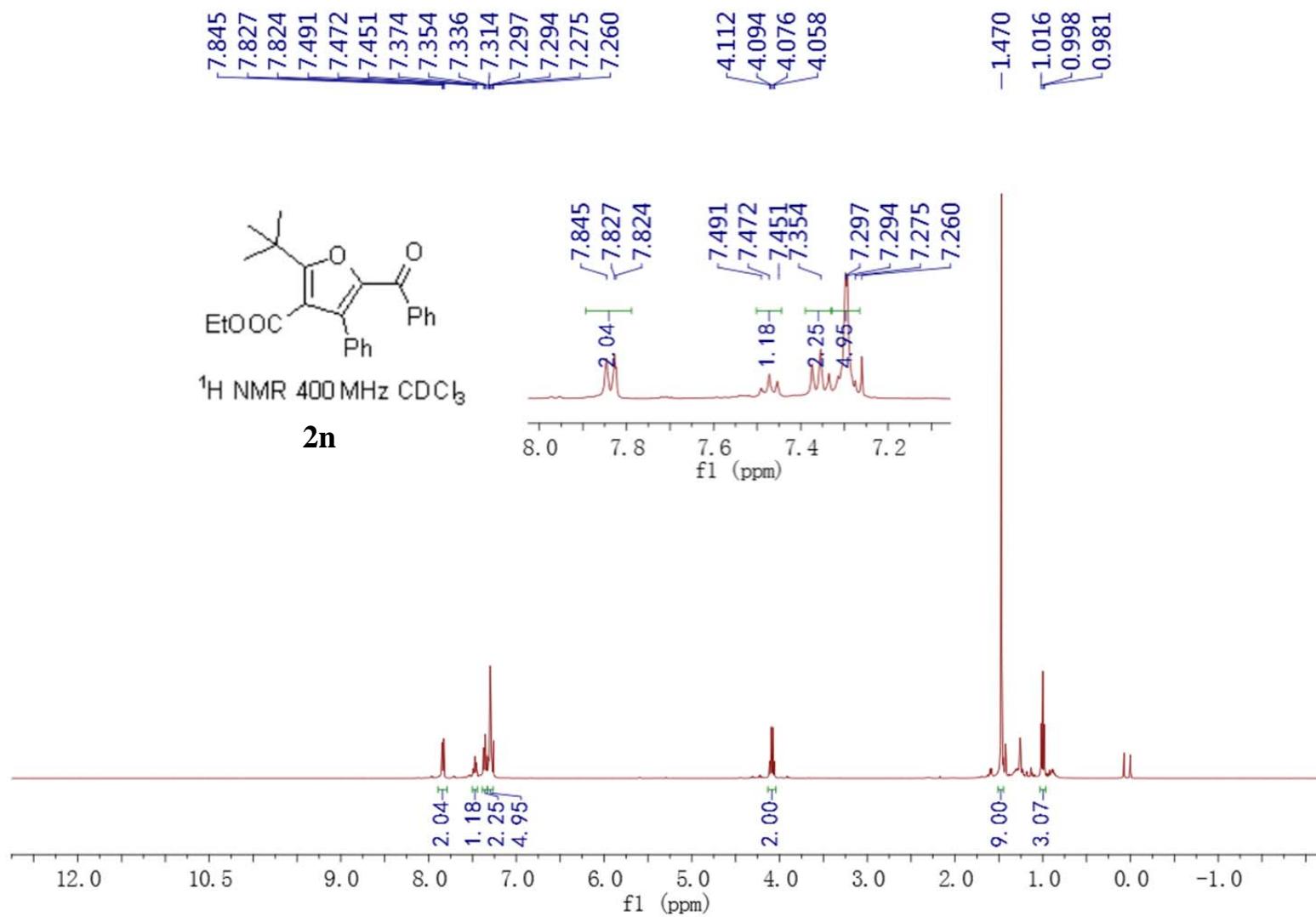


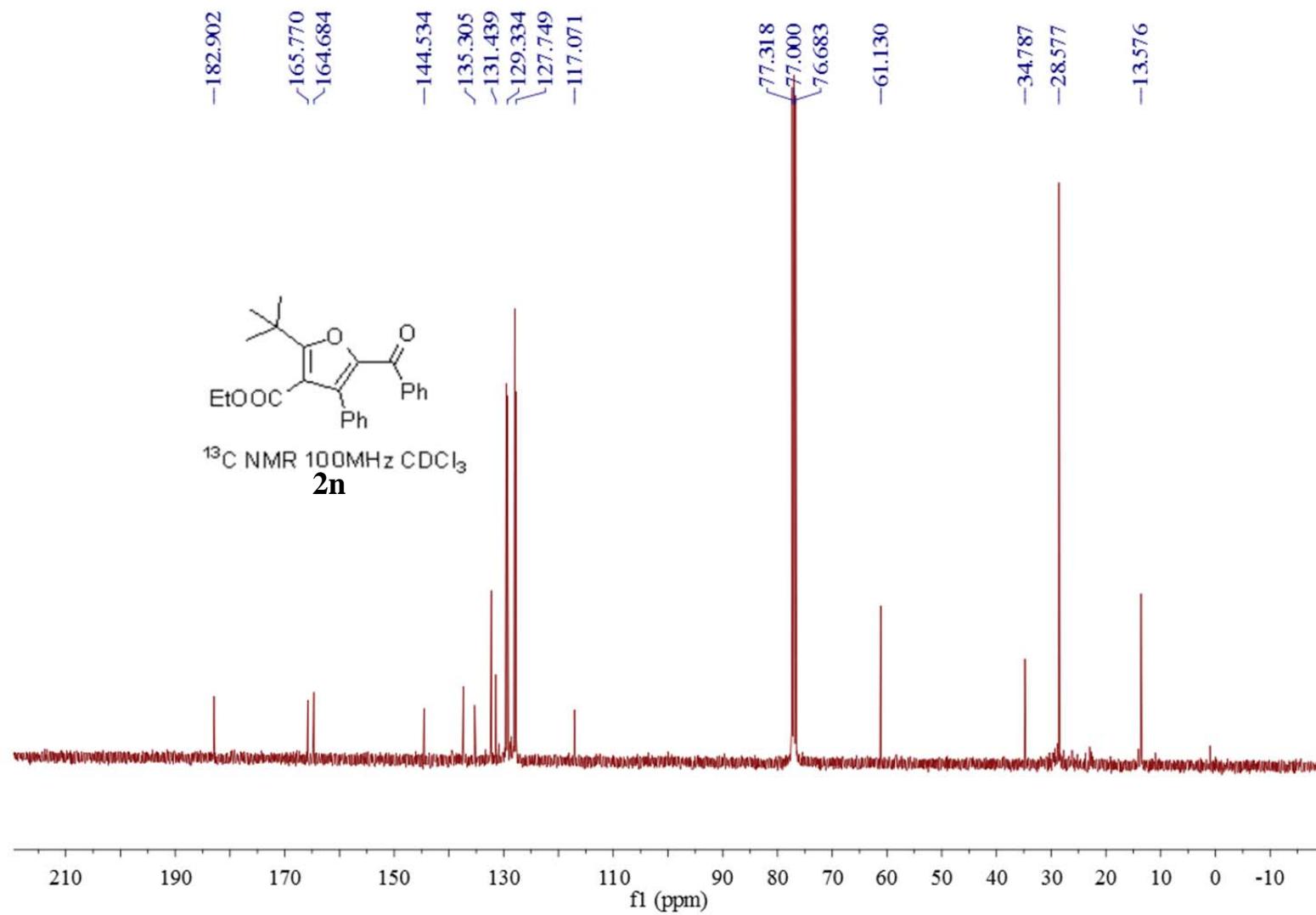


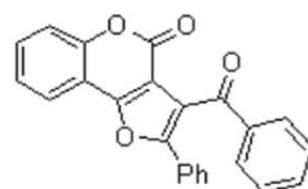






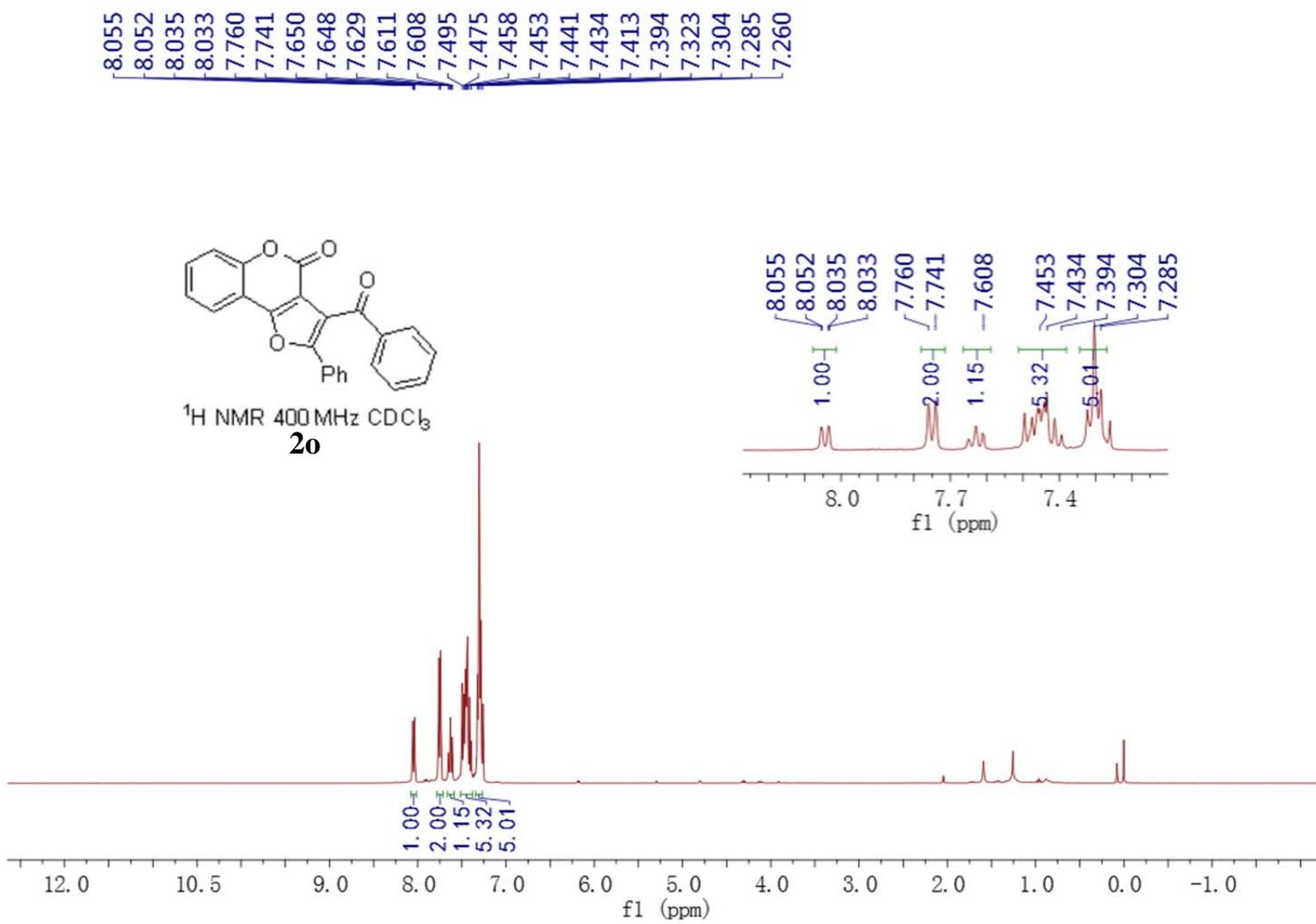


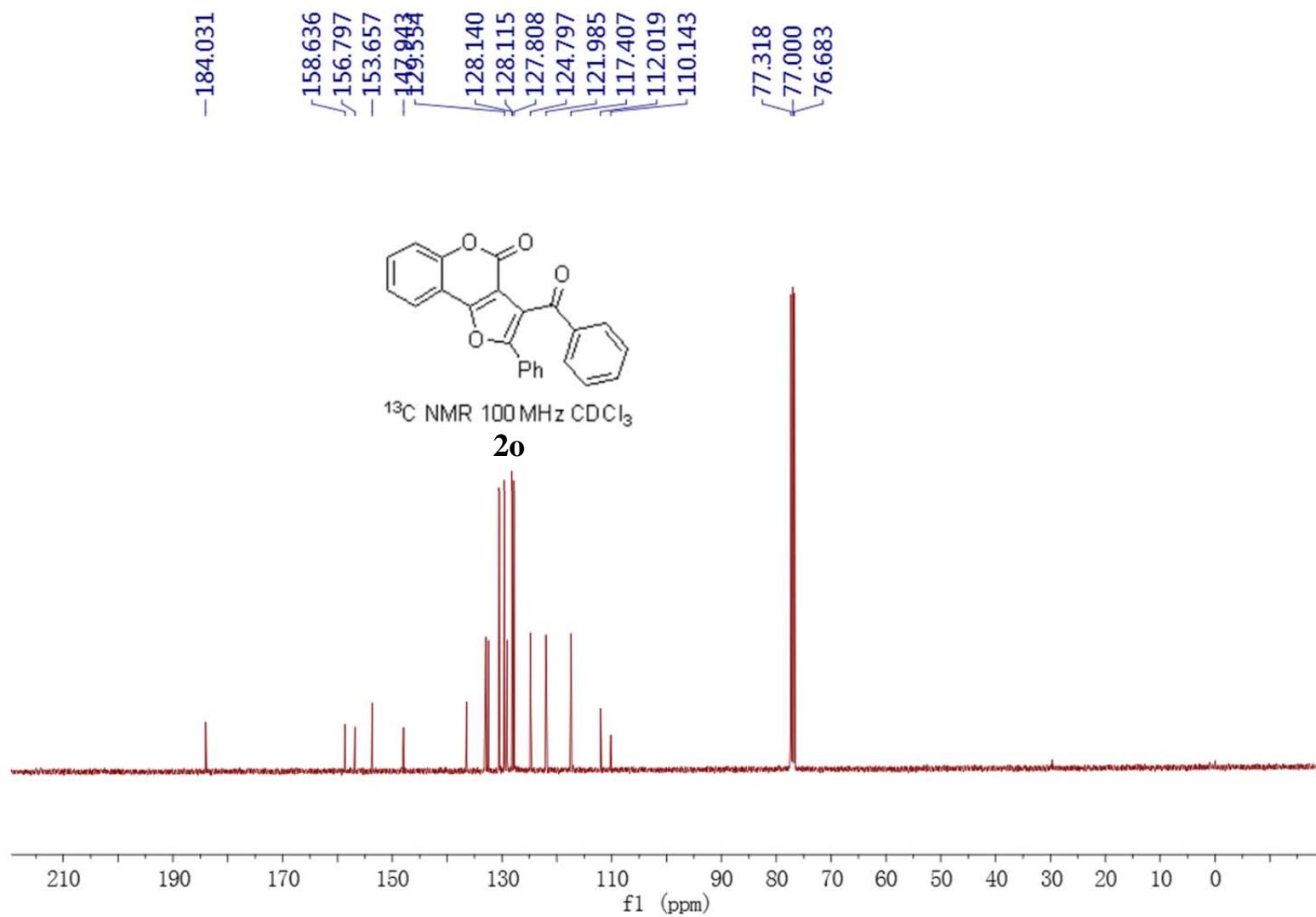


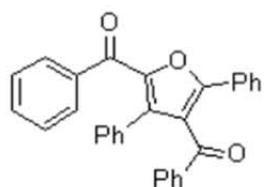


¹H NMR 400 MHz CDCl₃

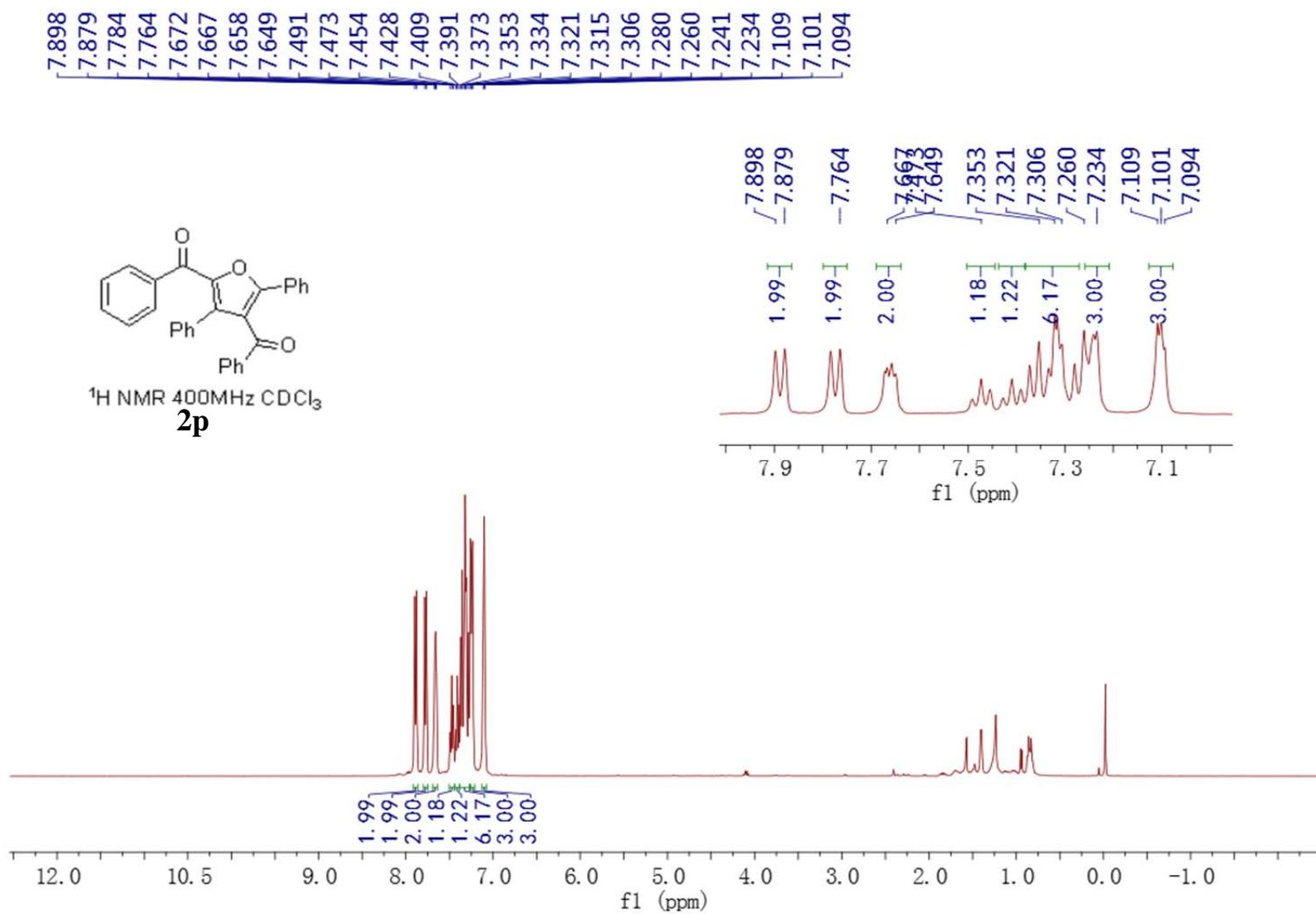
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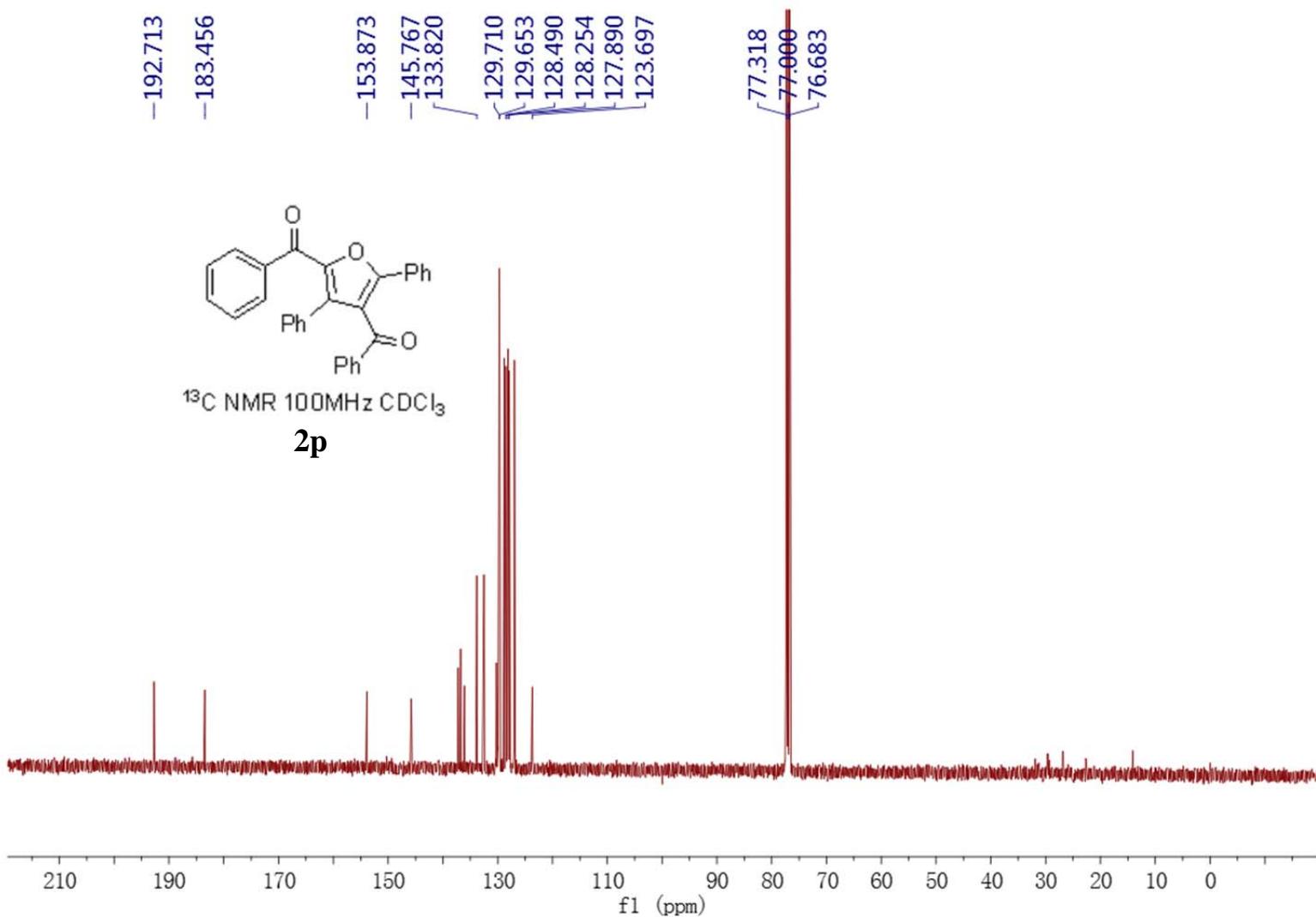


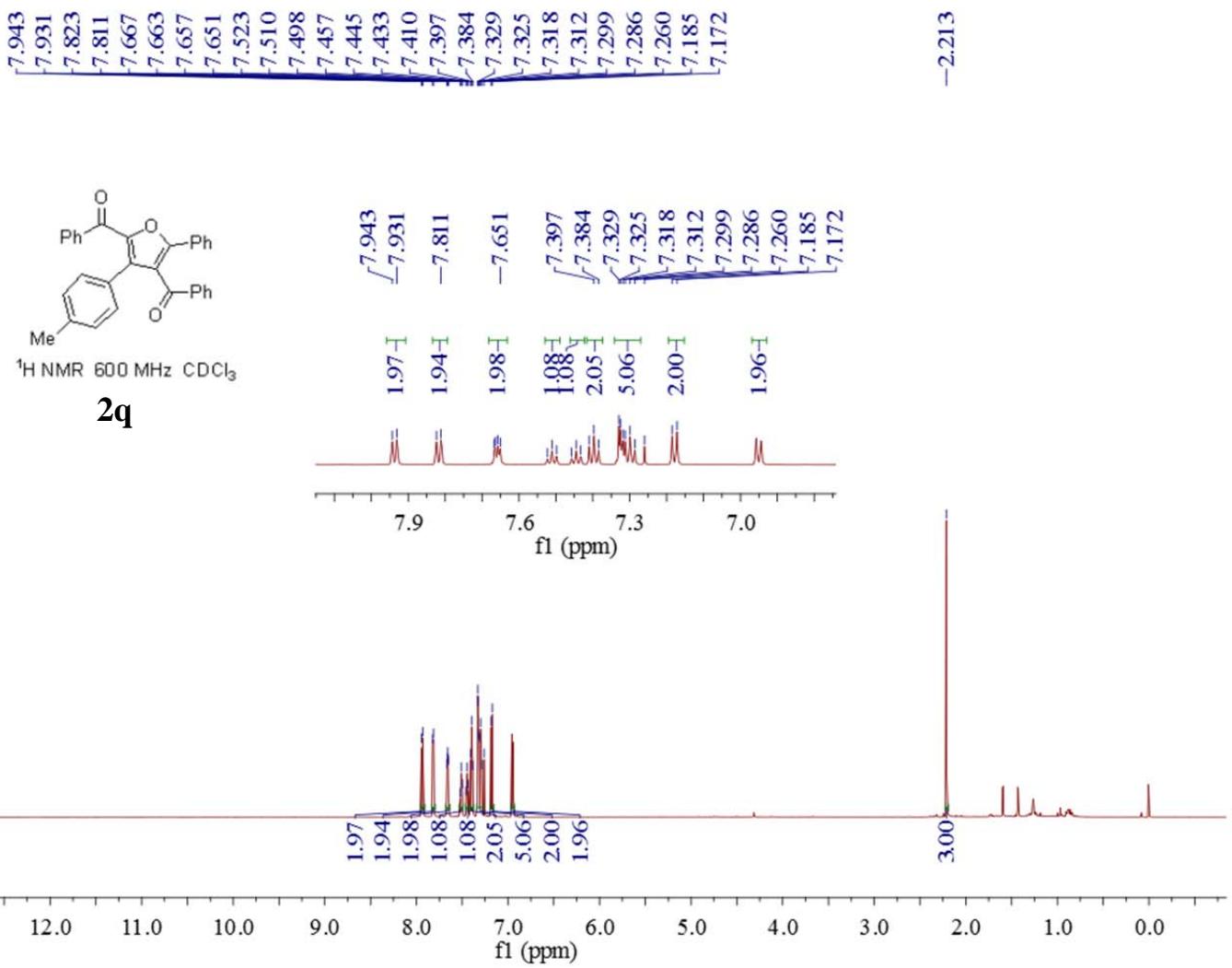


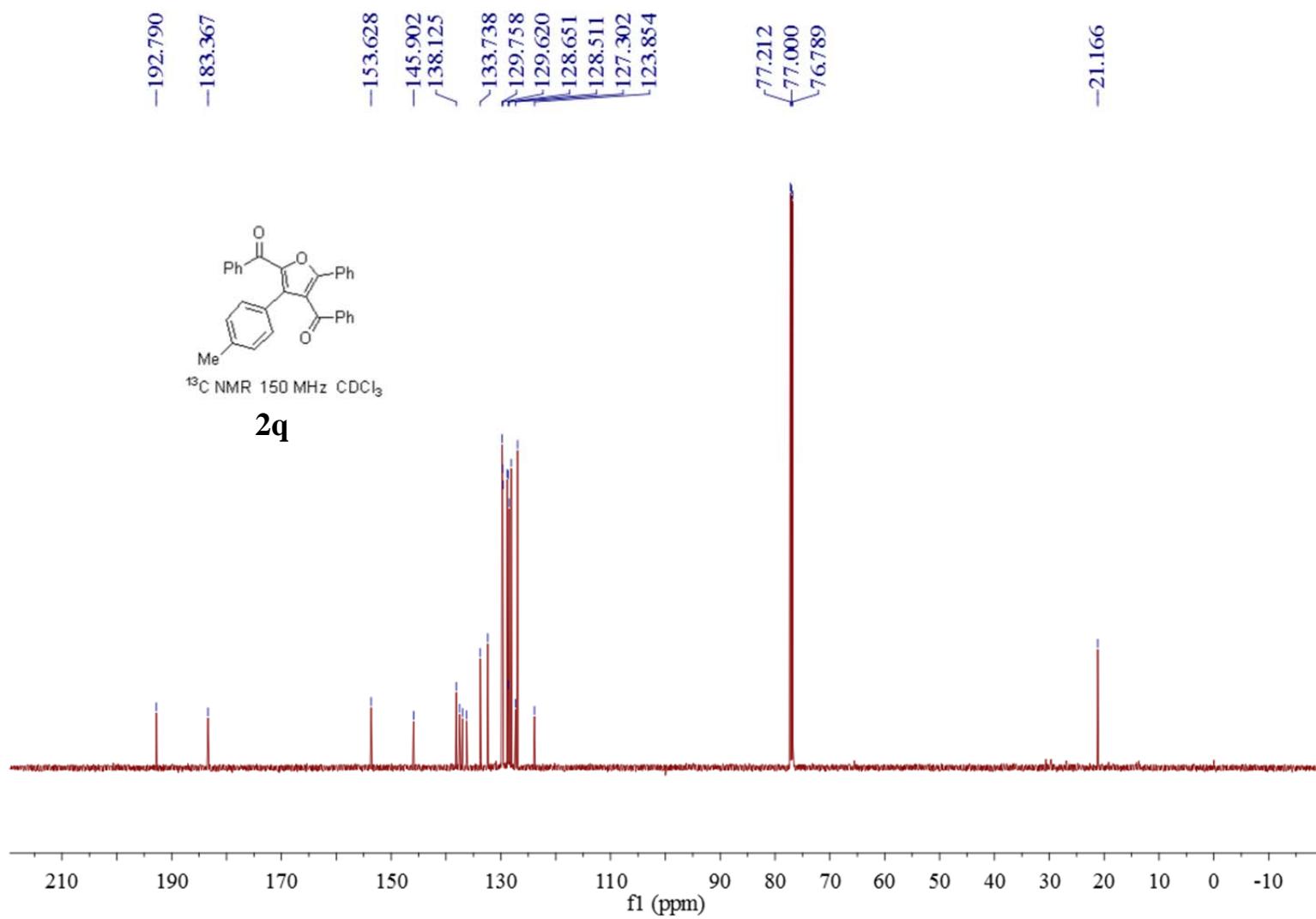


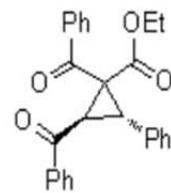
¹H NMR 400MHz CDCl₃





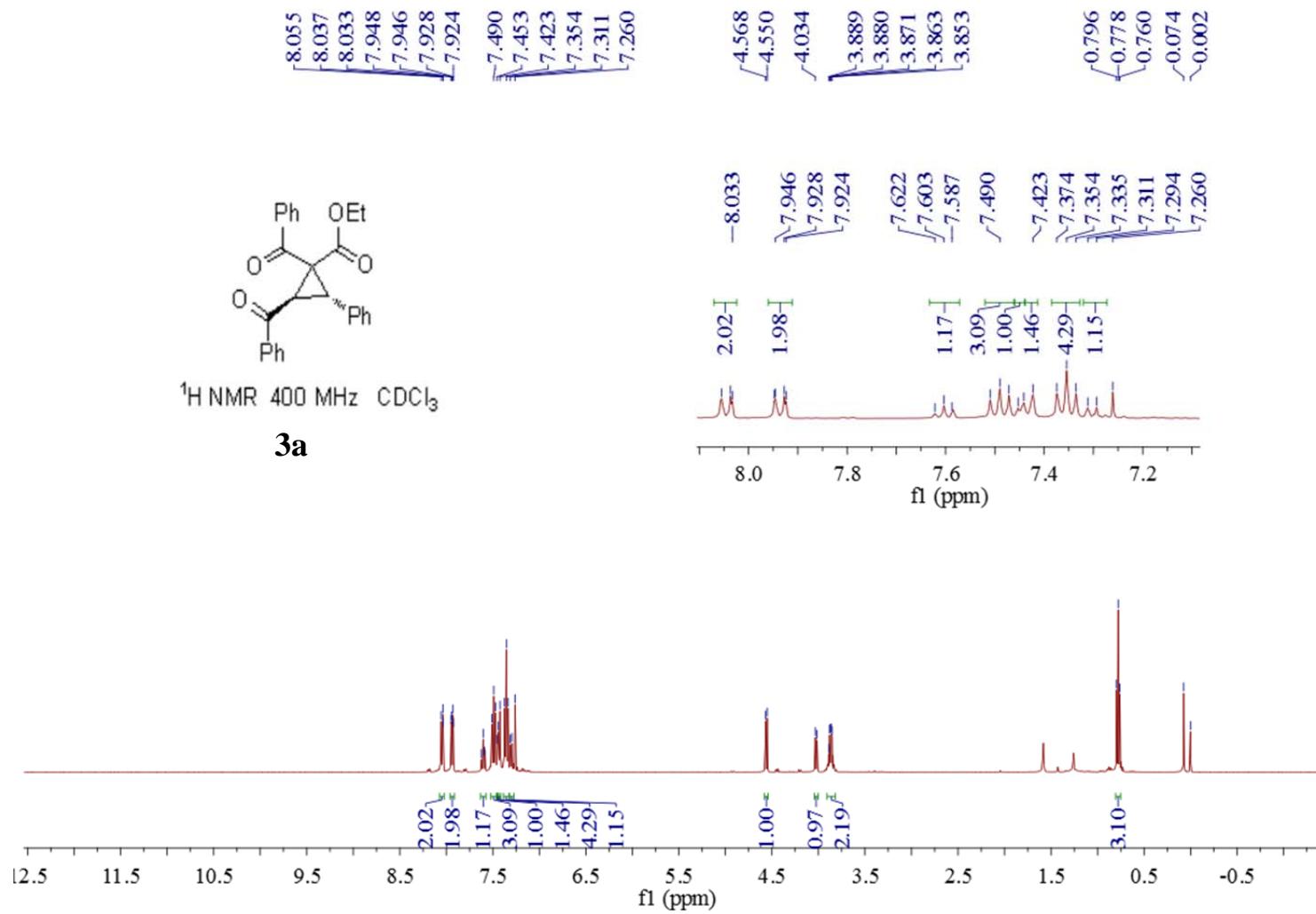


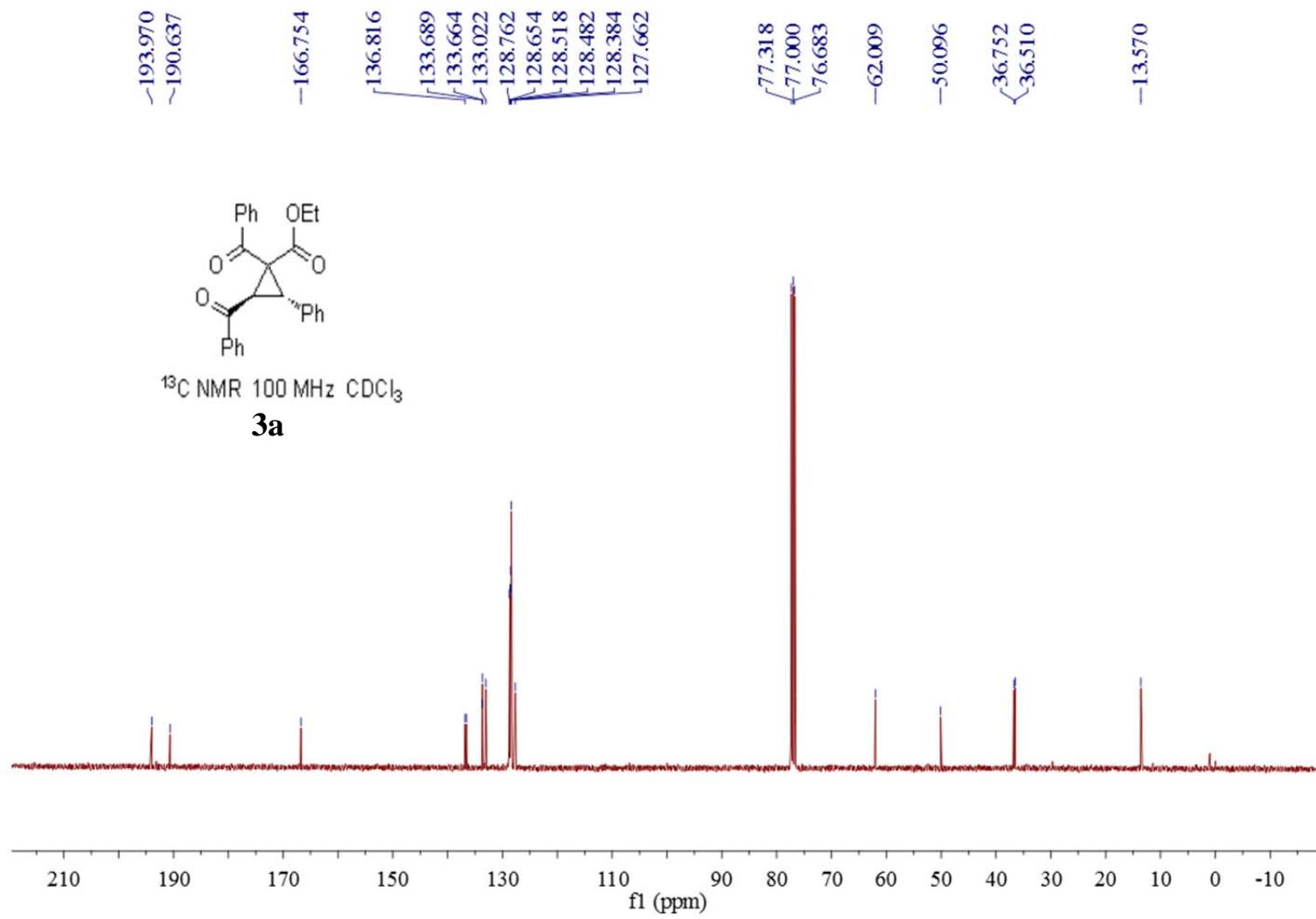


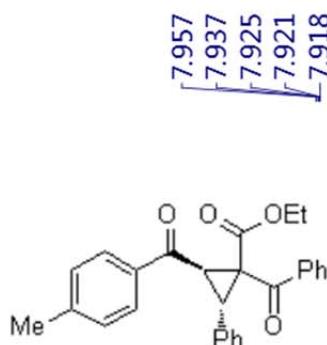


¹H NMR 400 MHz CDCl₃

3a

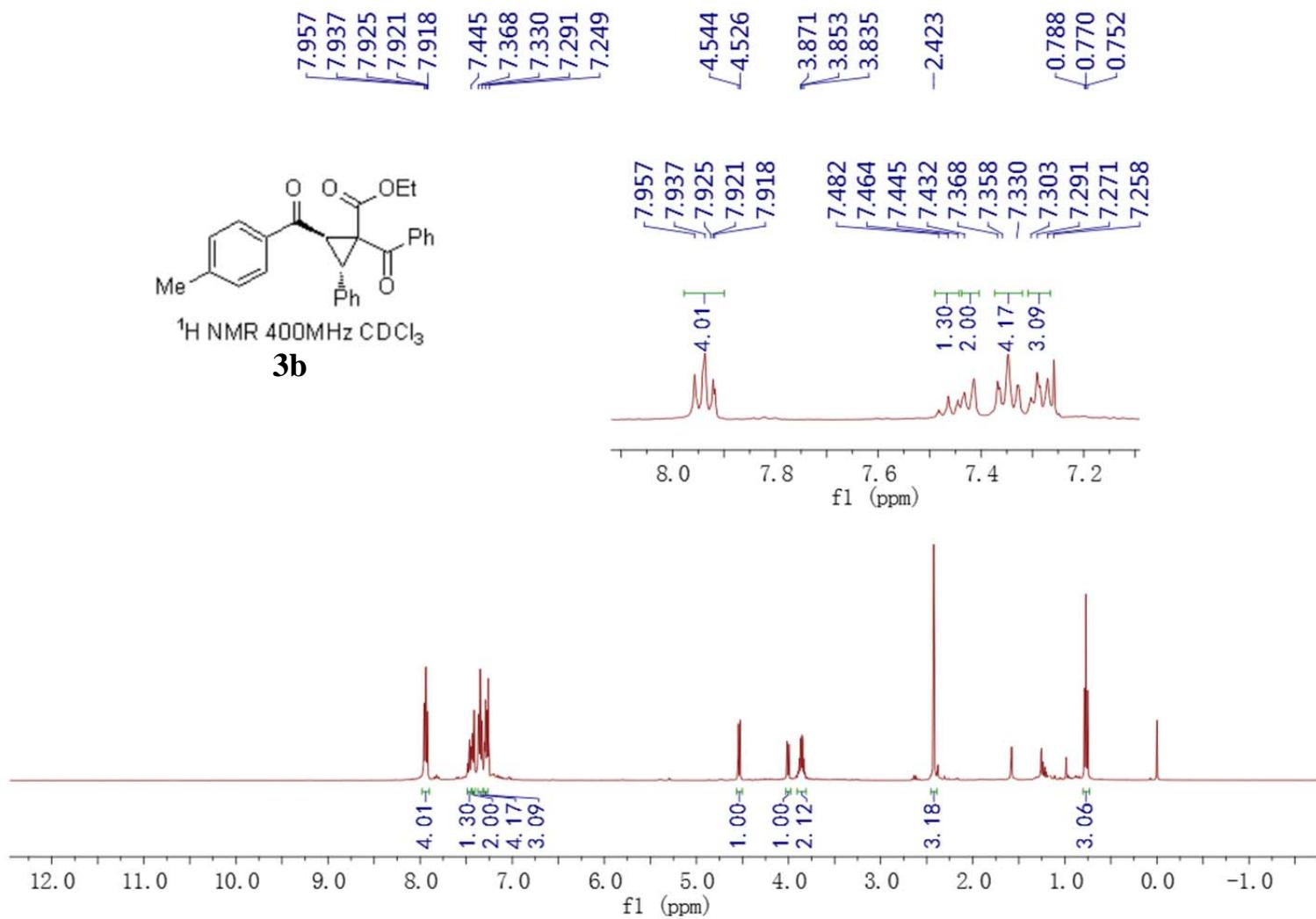


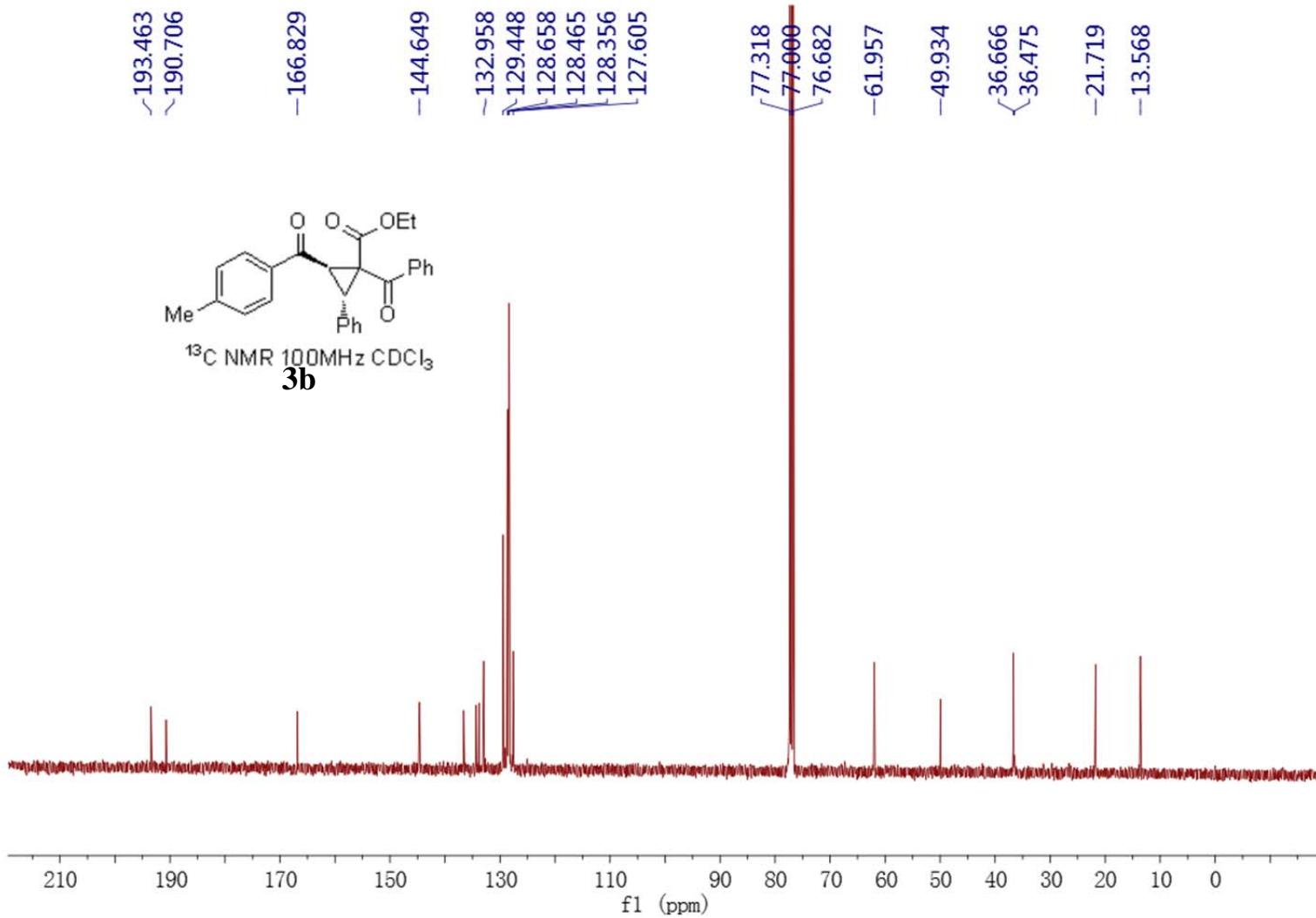


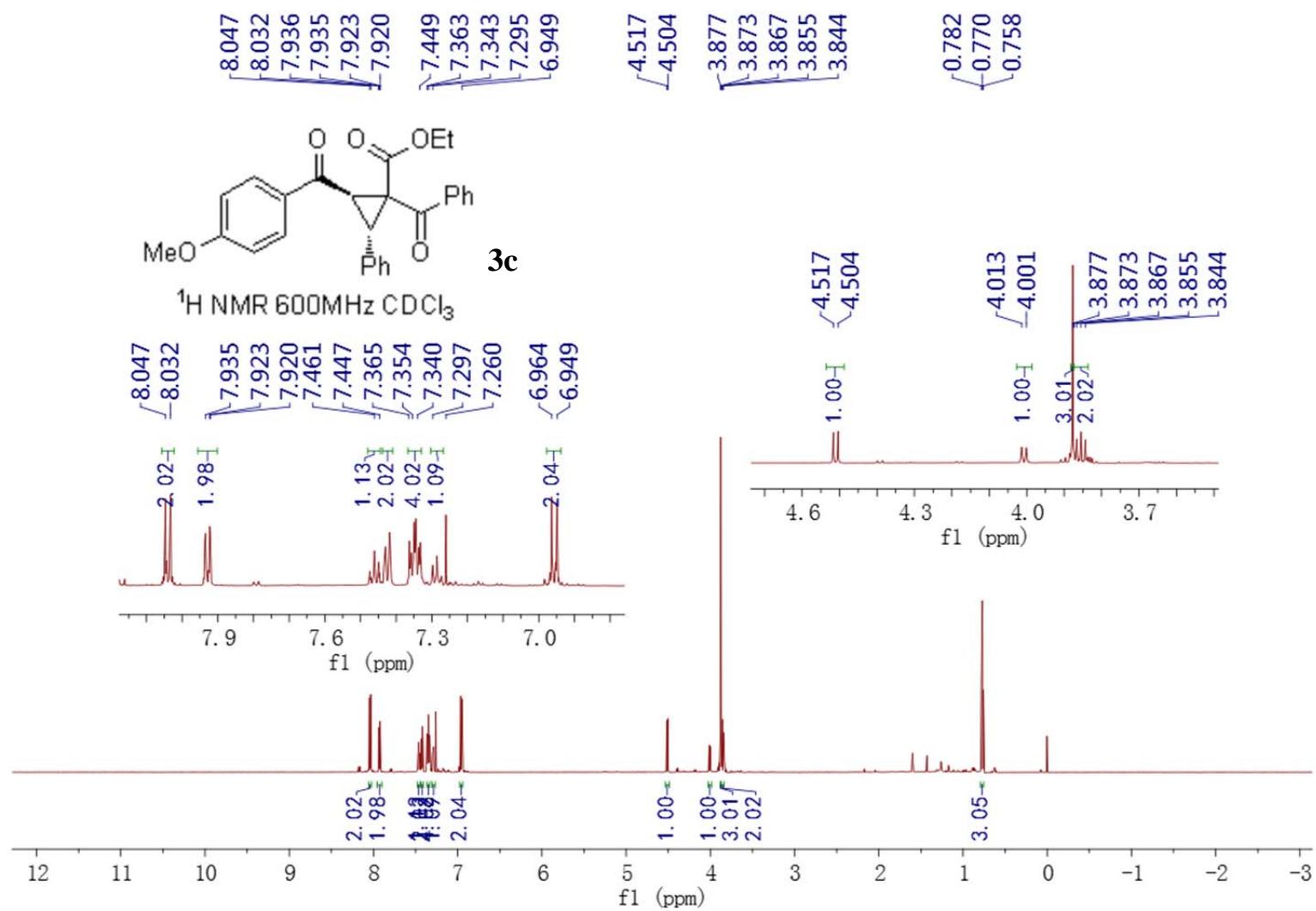


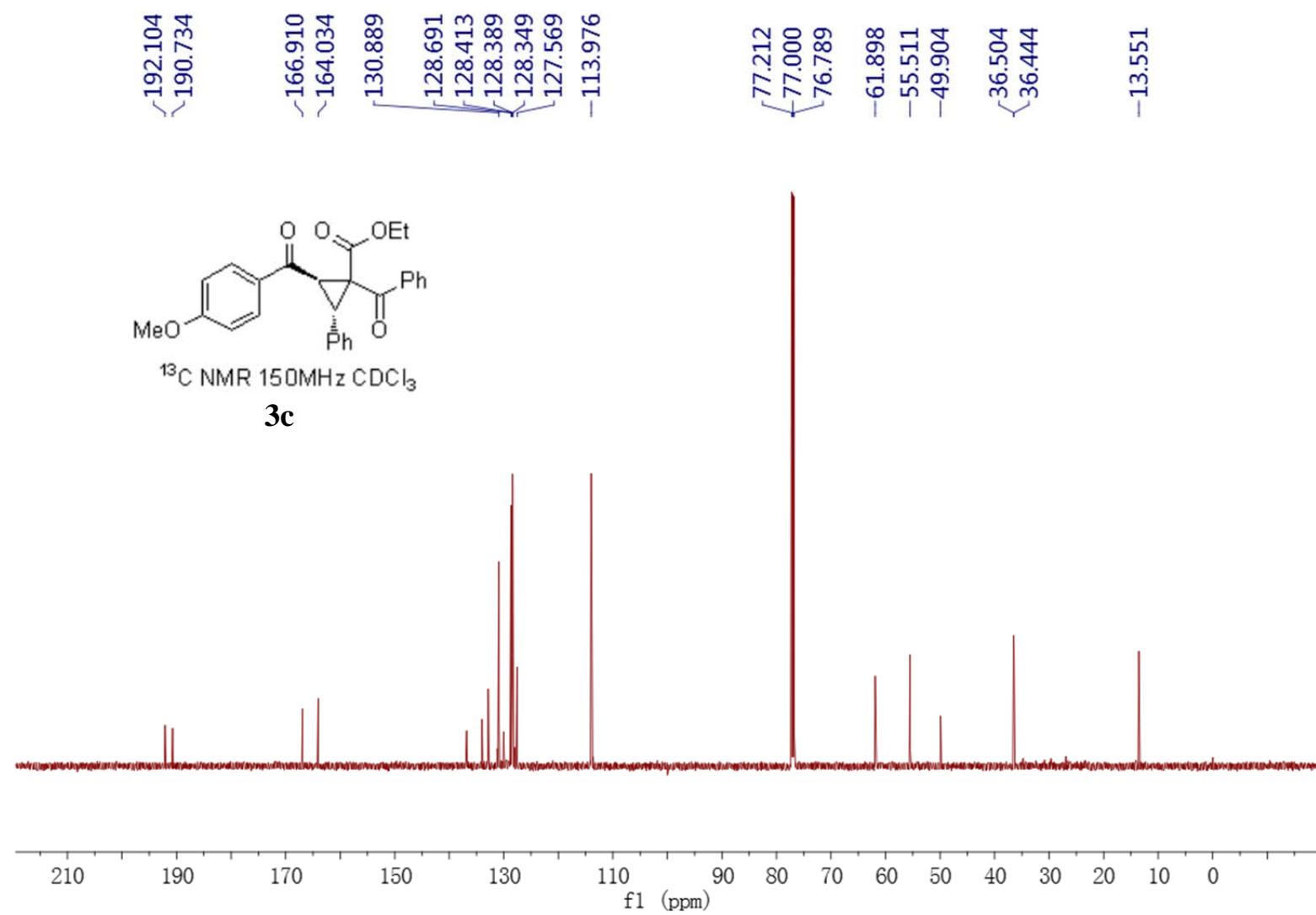
¹H NMR 400MHz CDCl₃

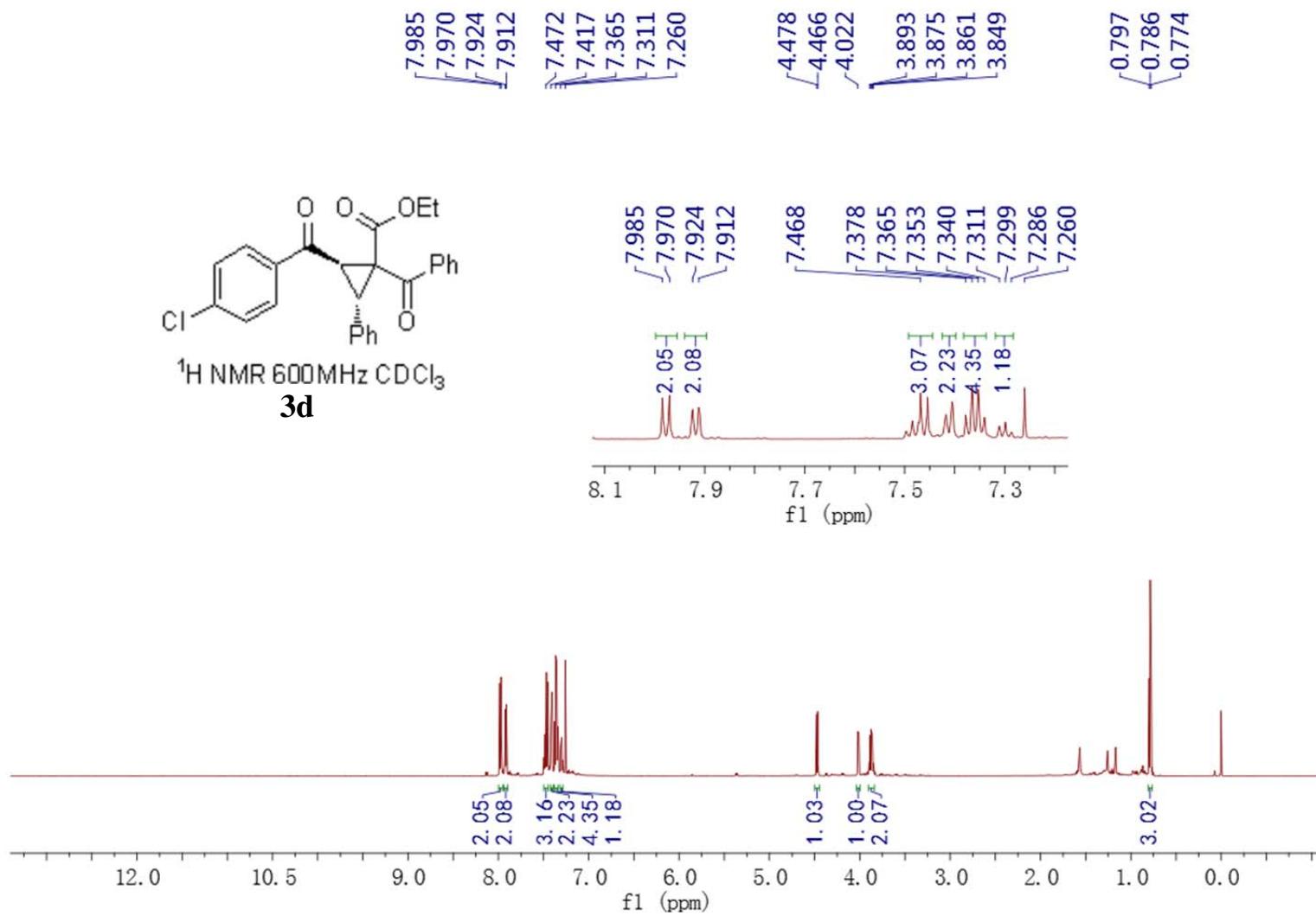
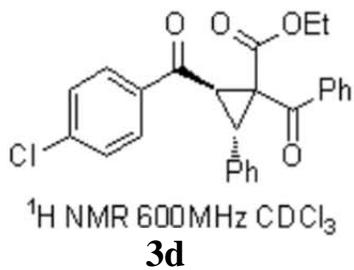
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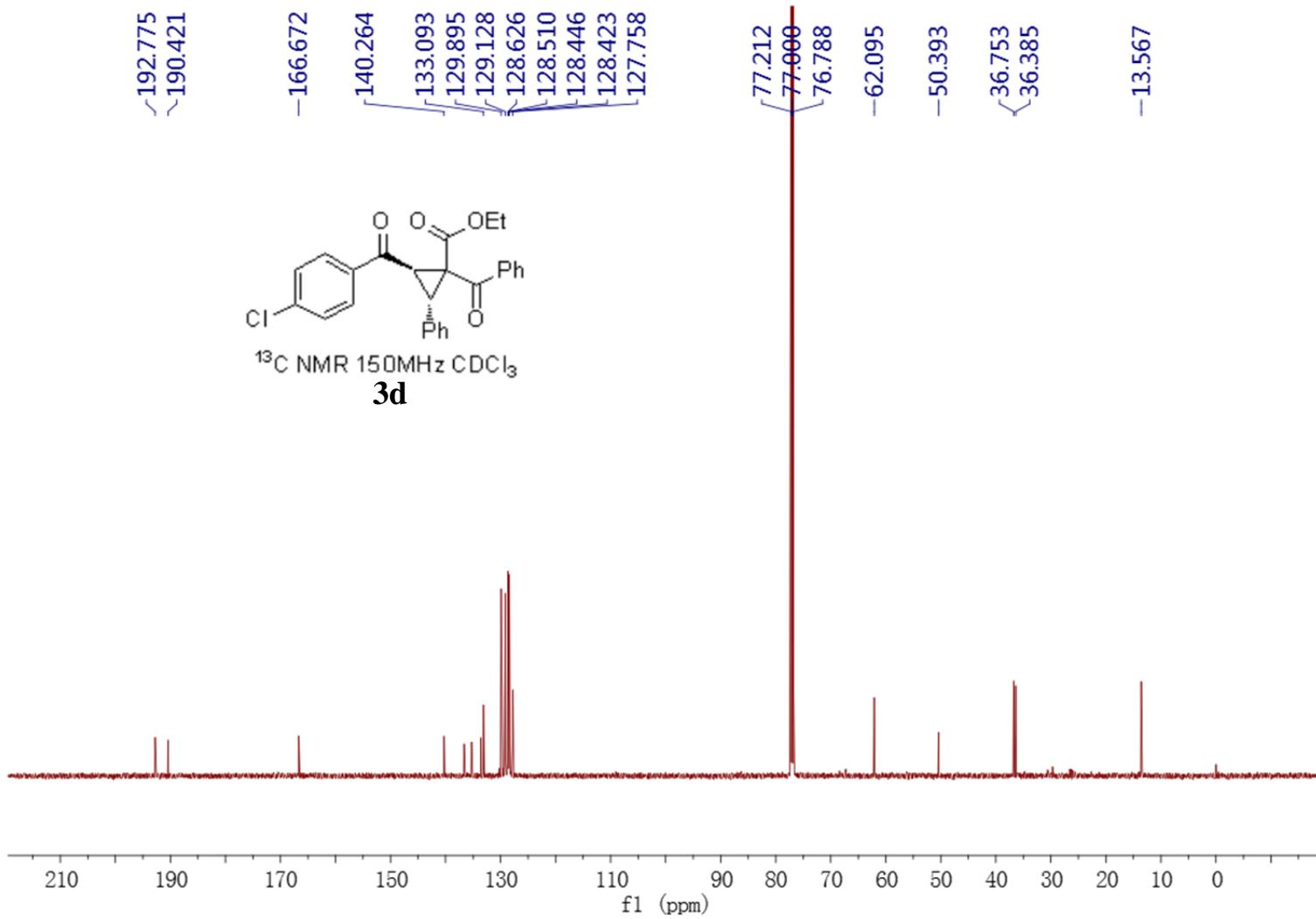


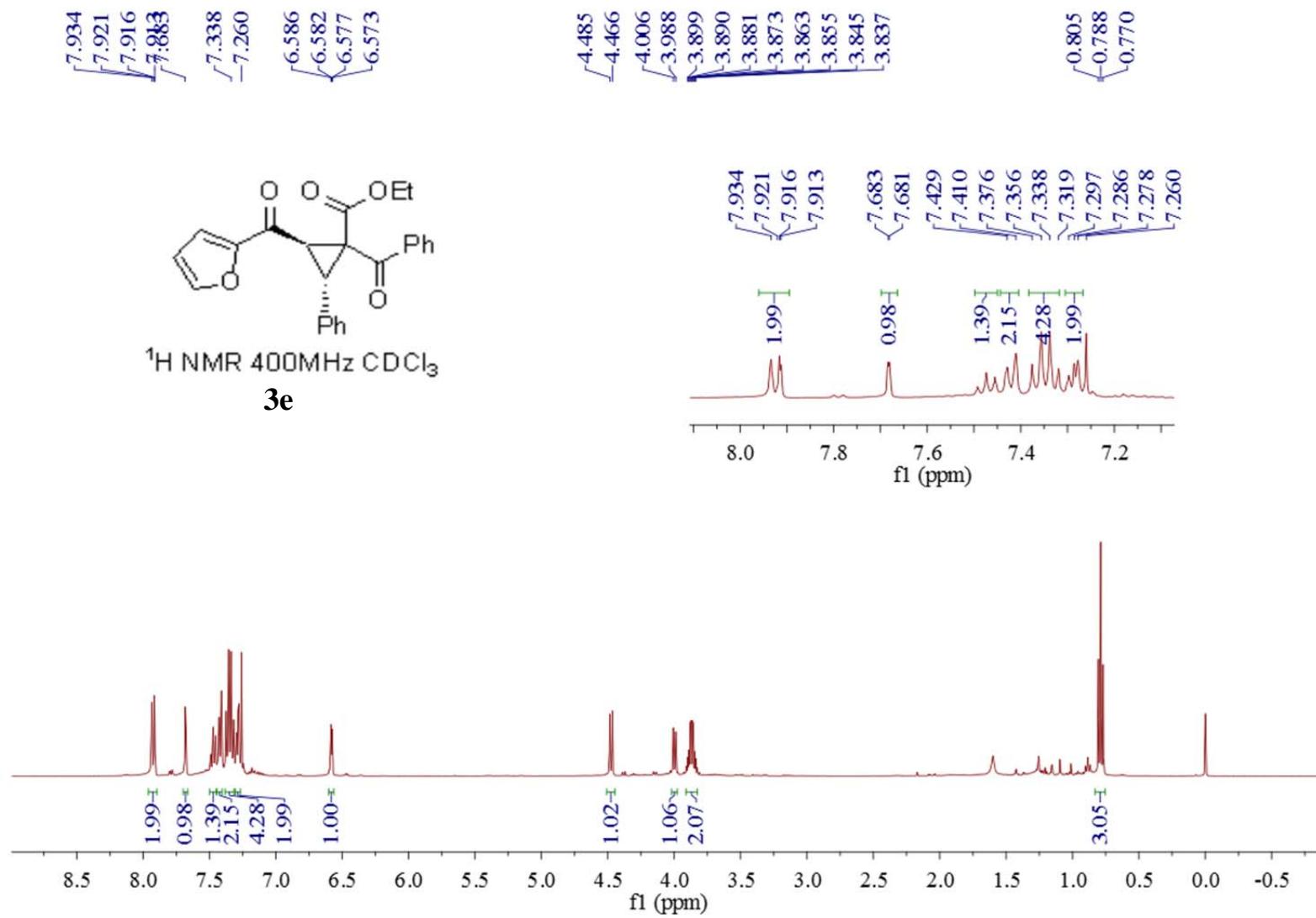


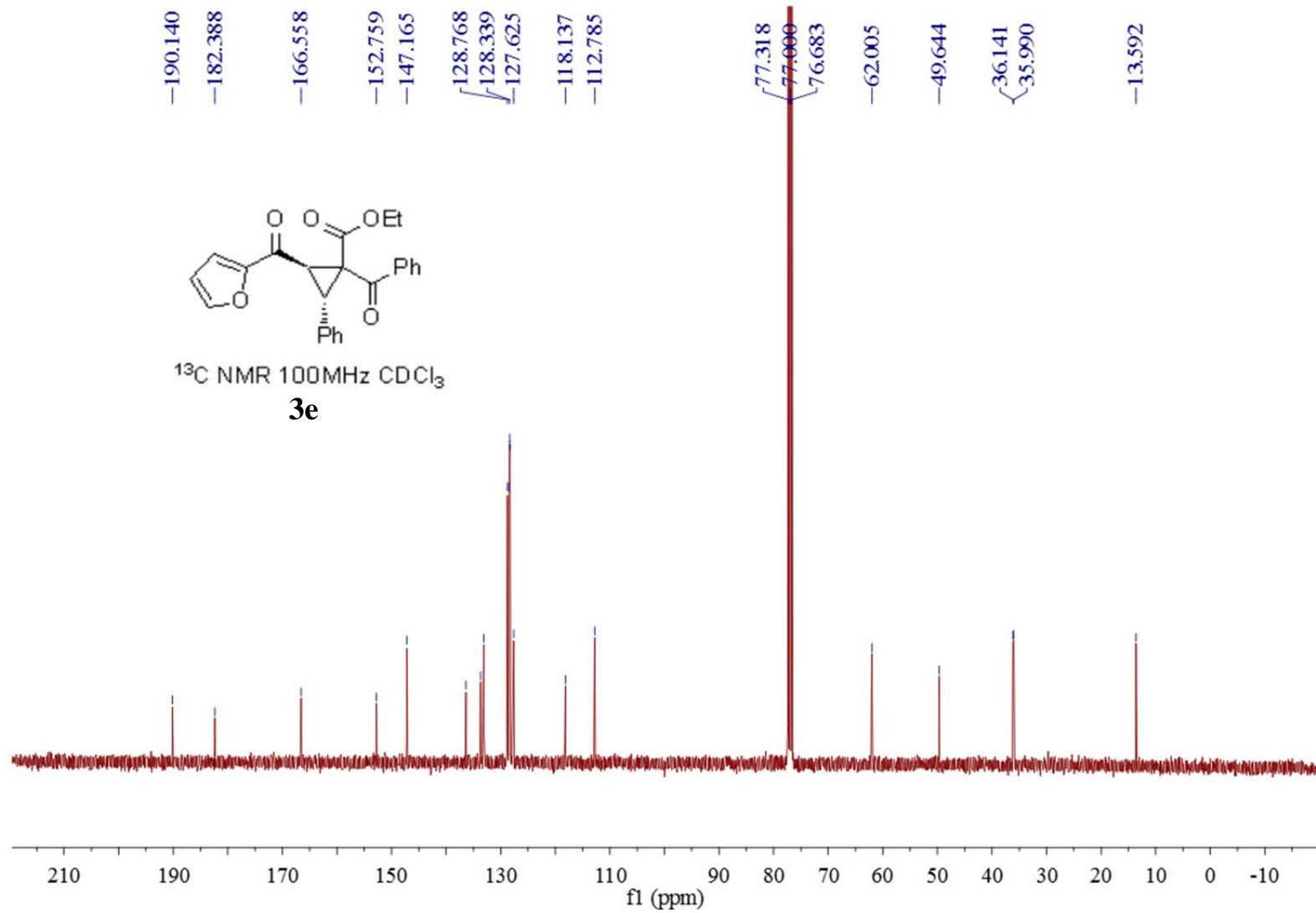


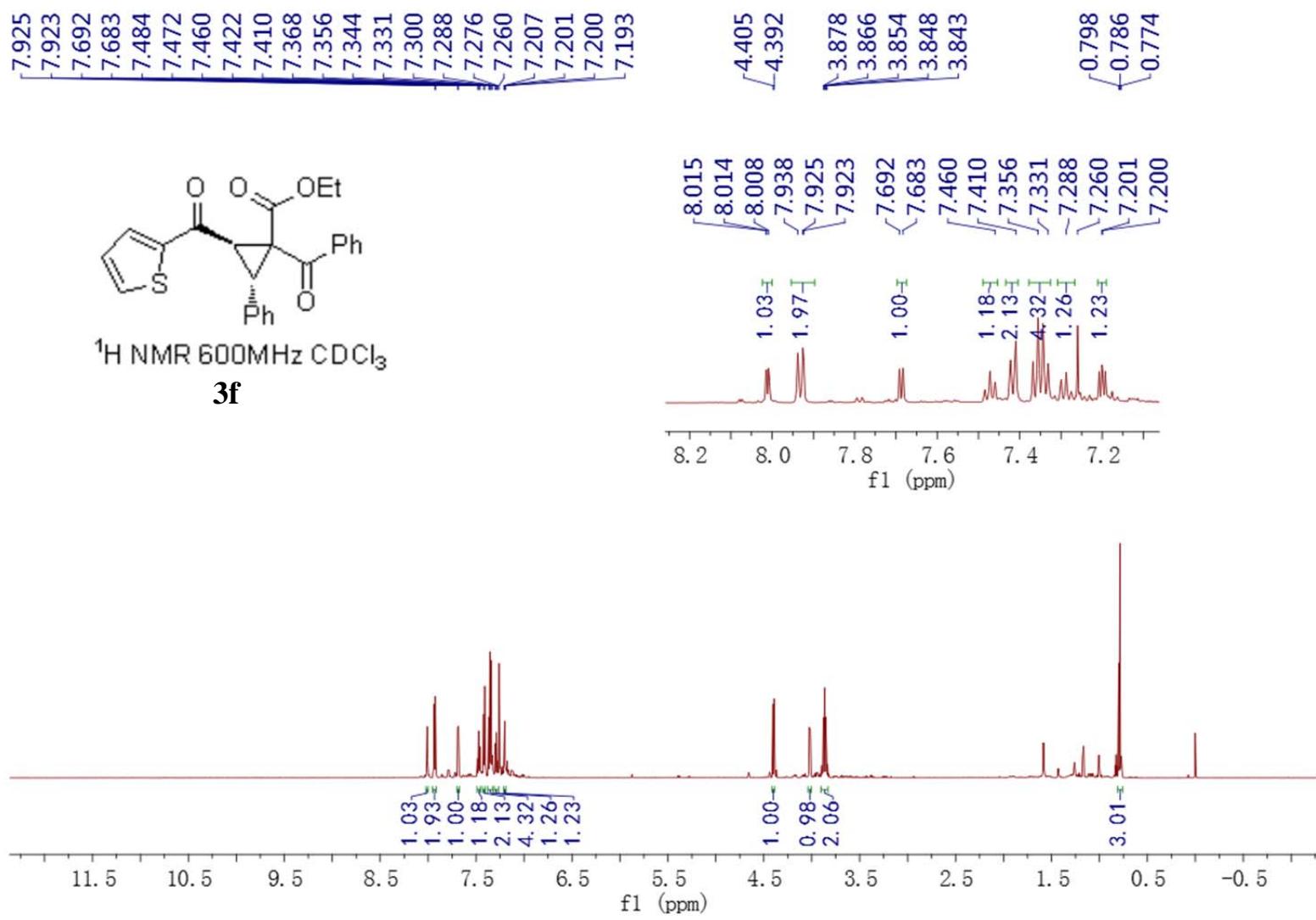


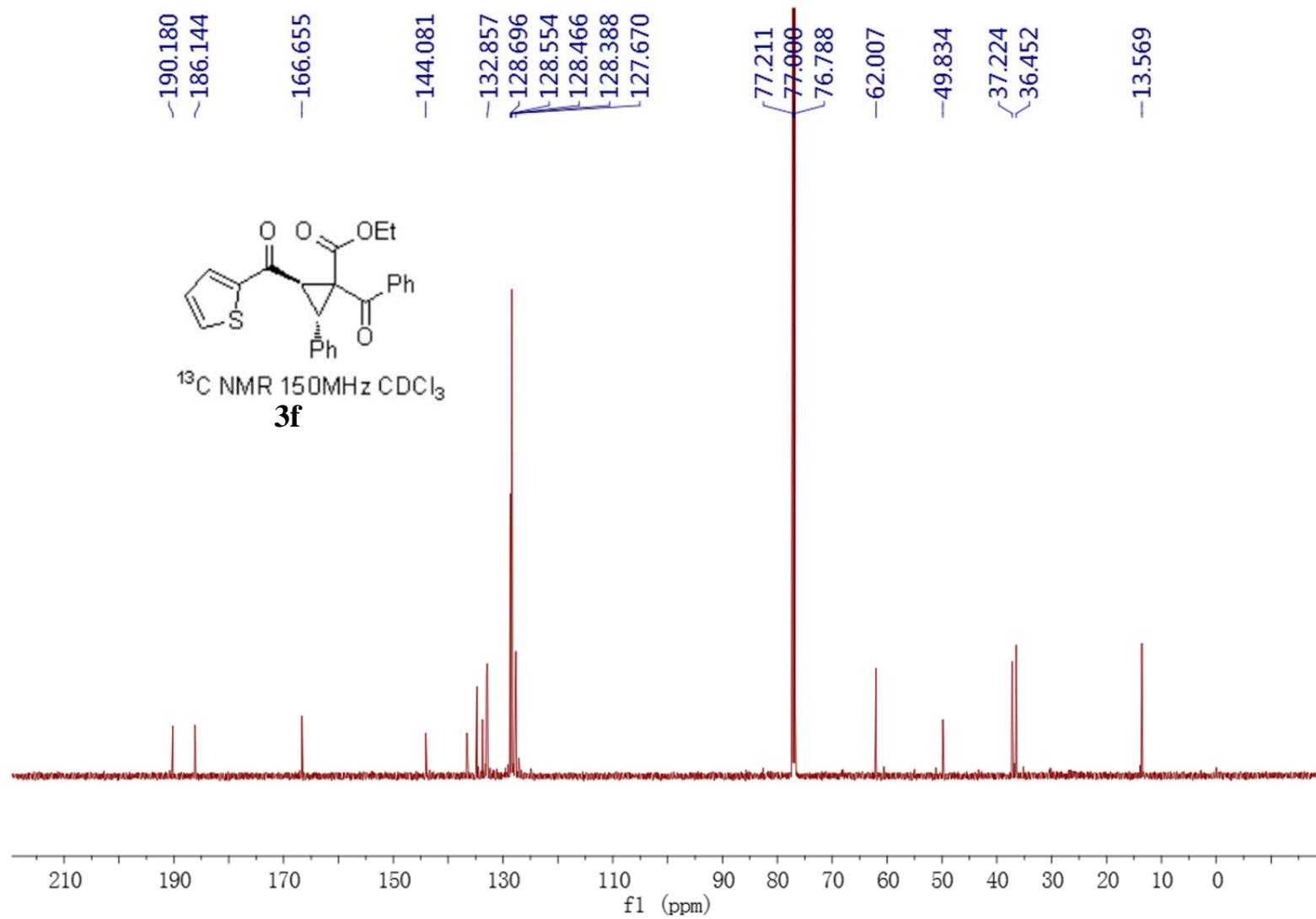


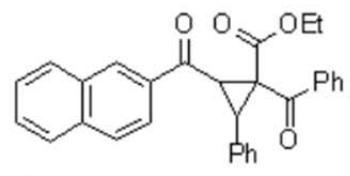






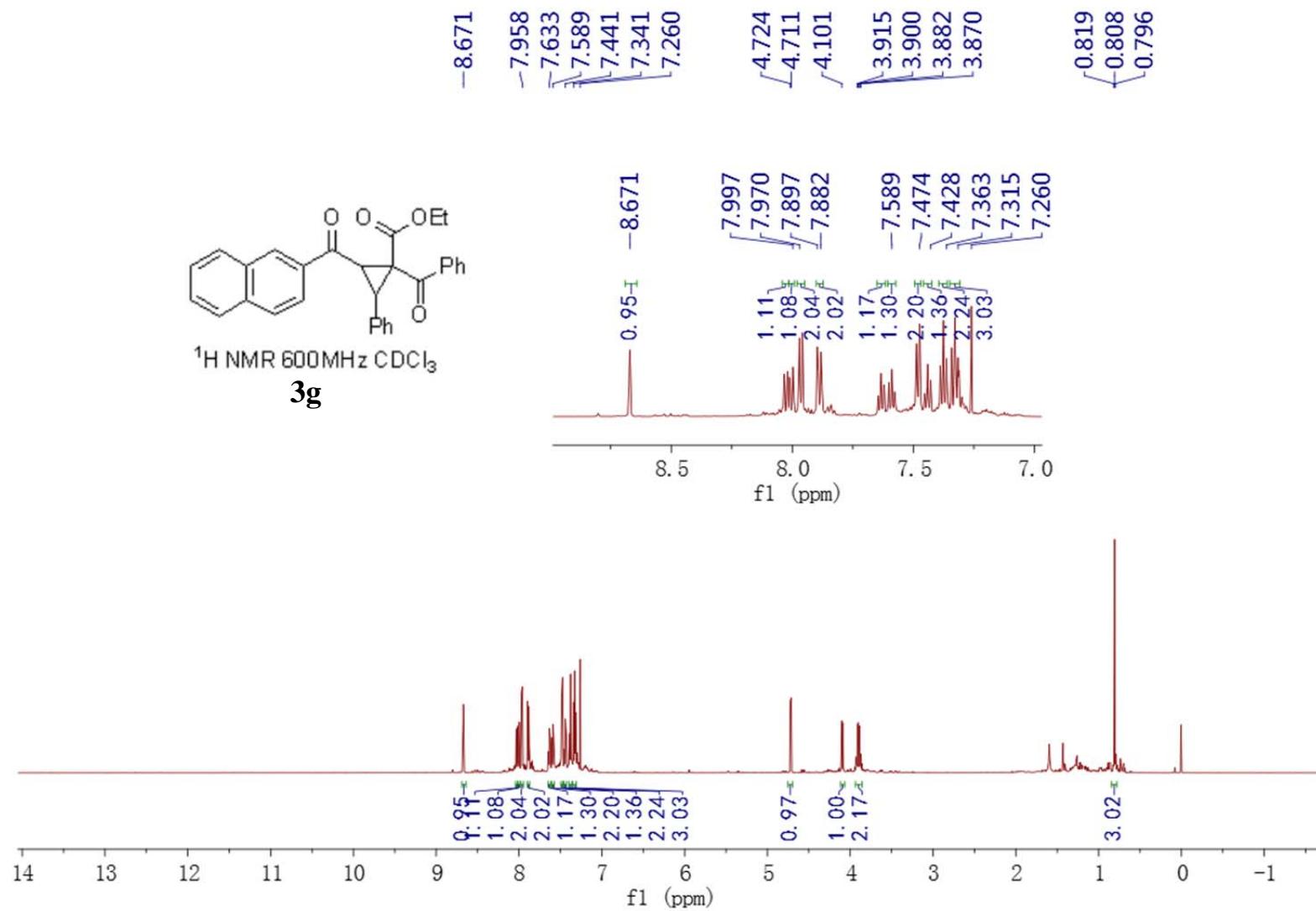


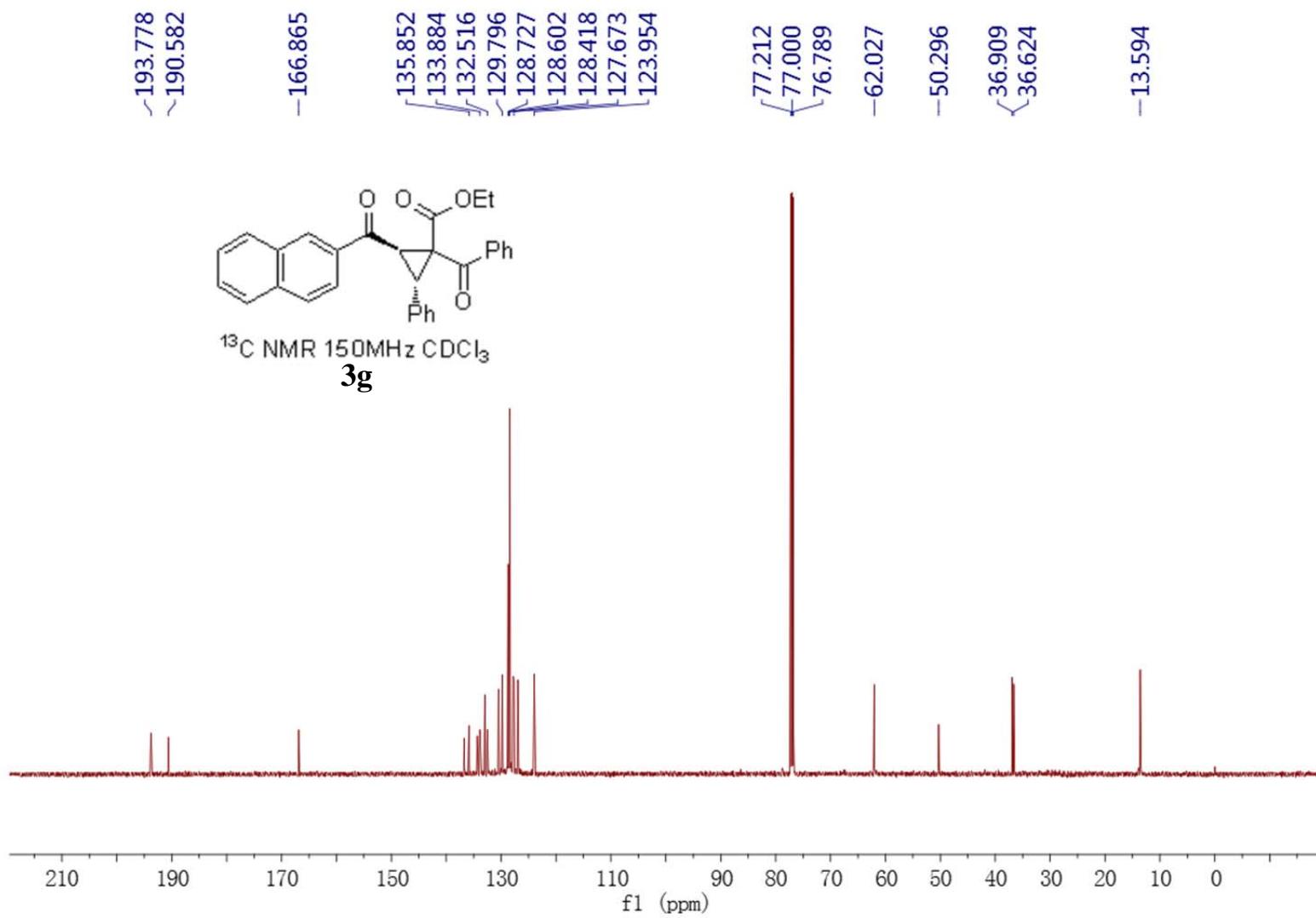


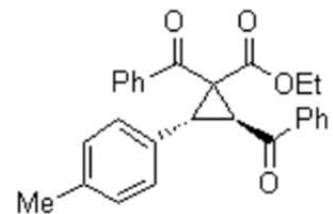


¹H NMR 600MHz CDCl₃

3g

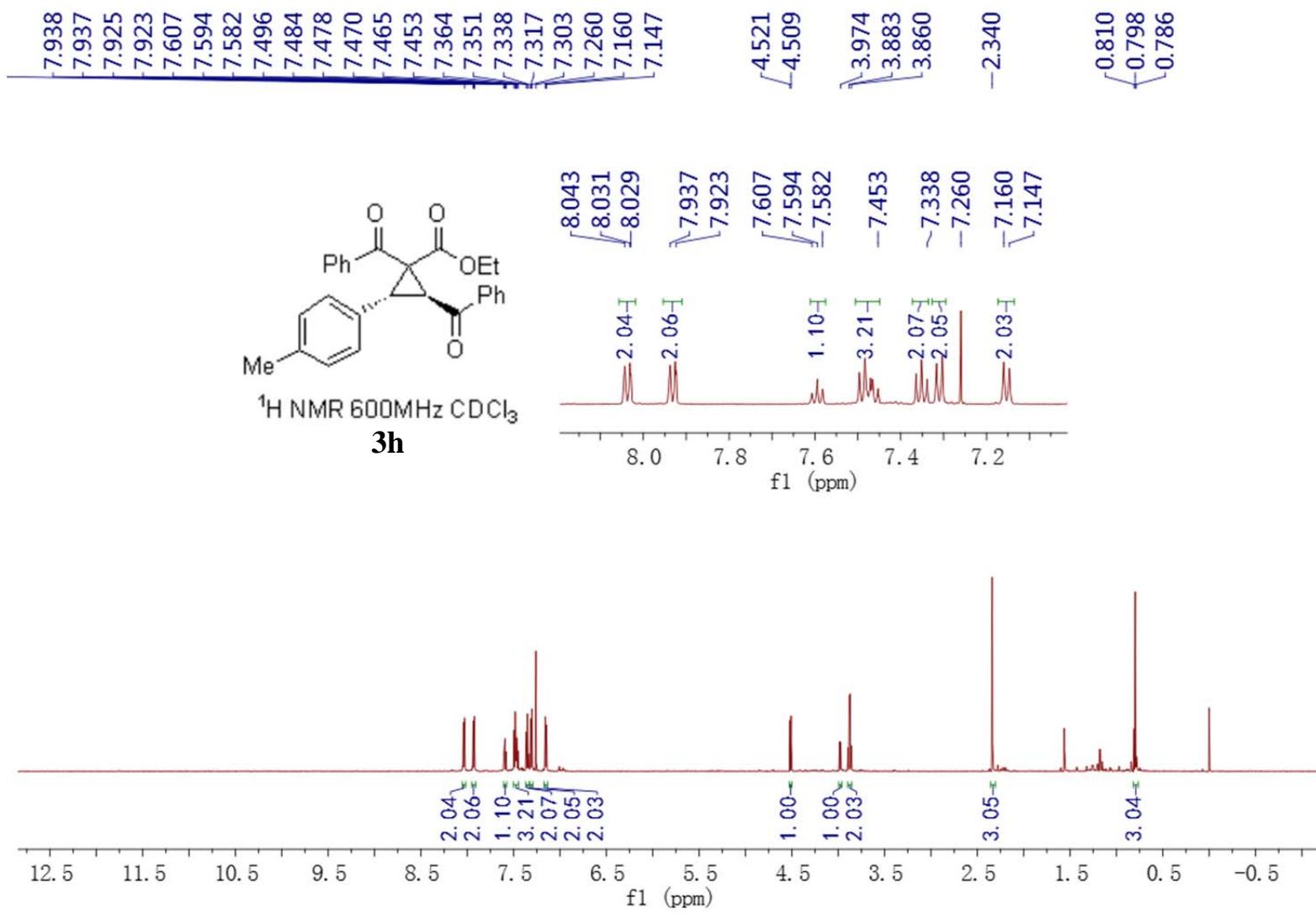


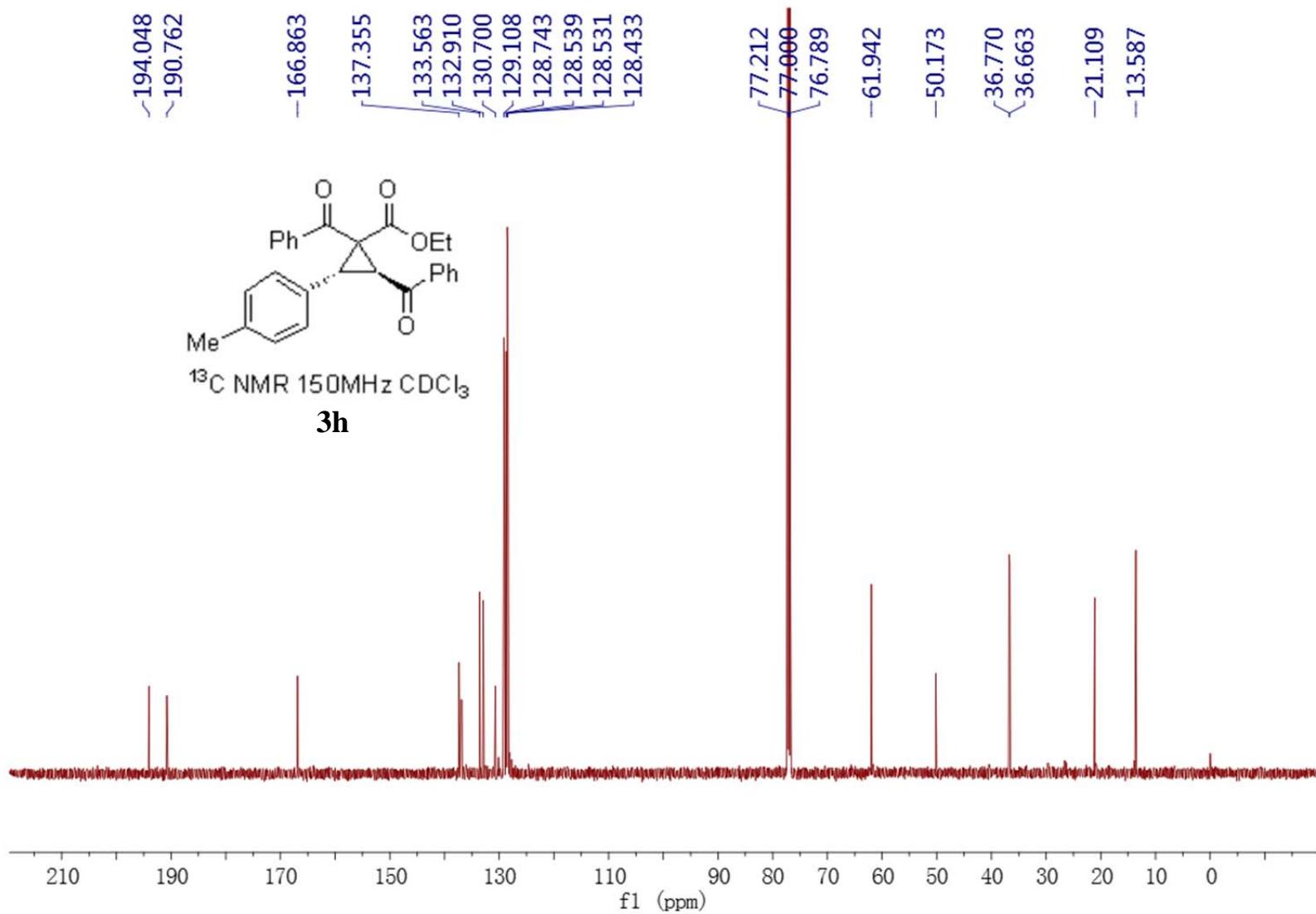


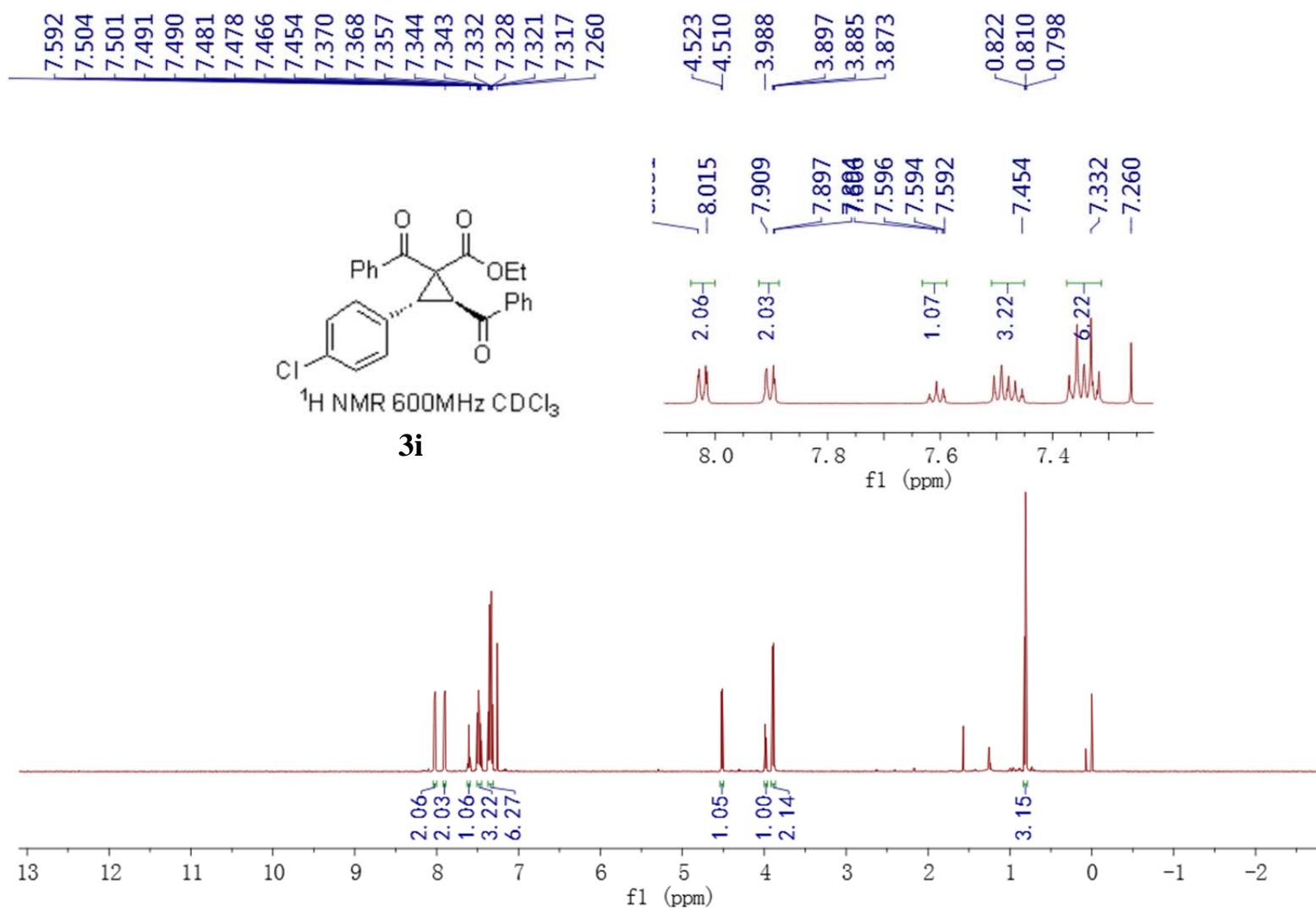


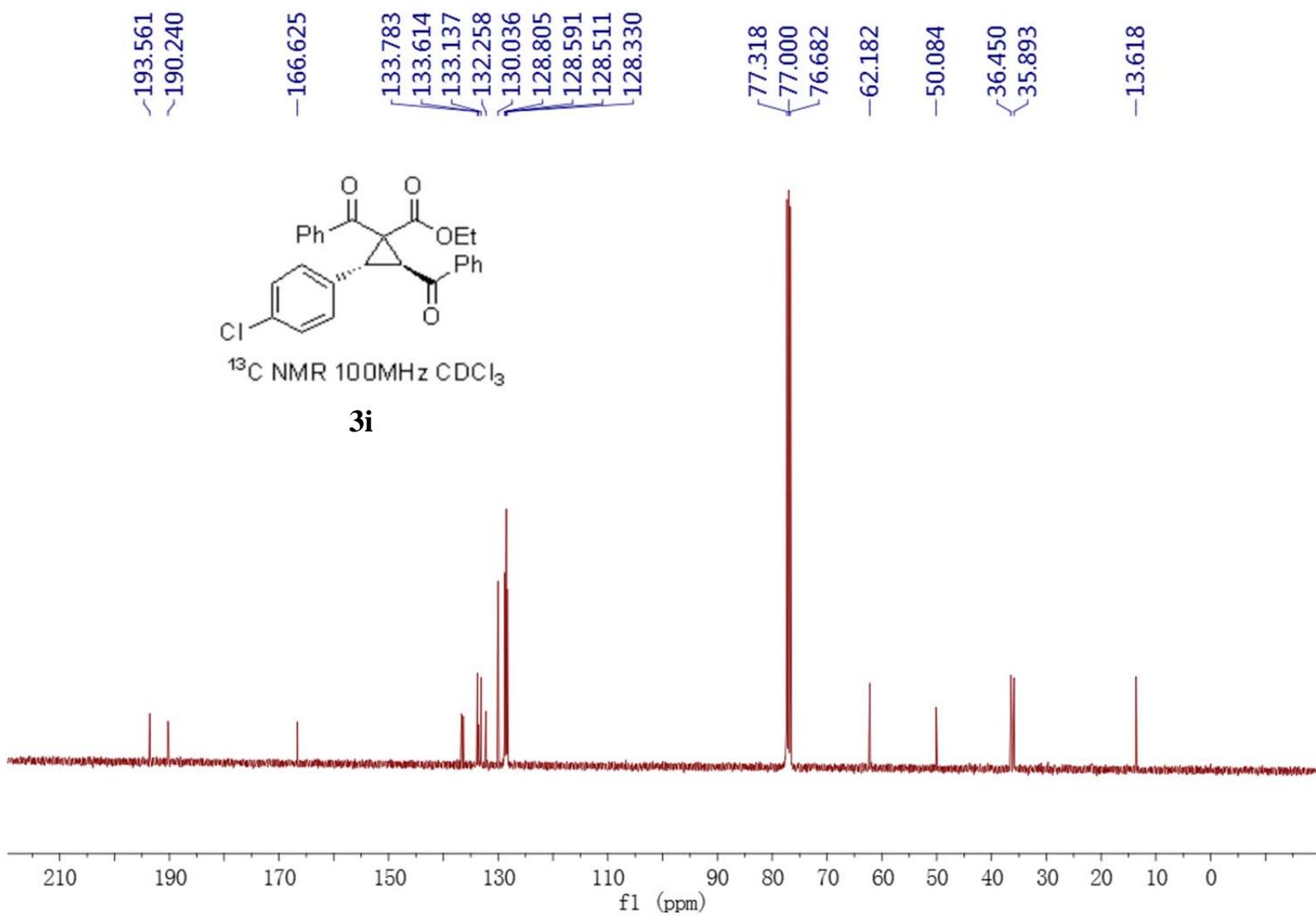
¹H NMR 600MHz CDCl₃

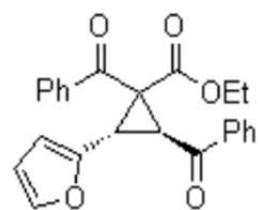
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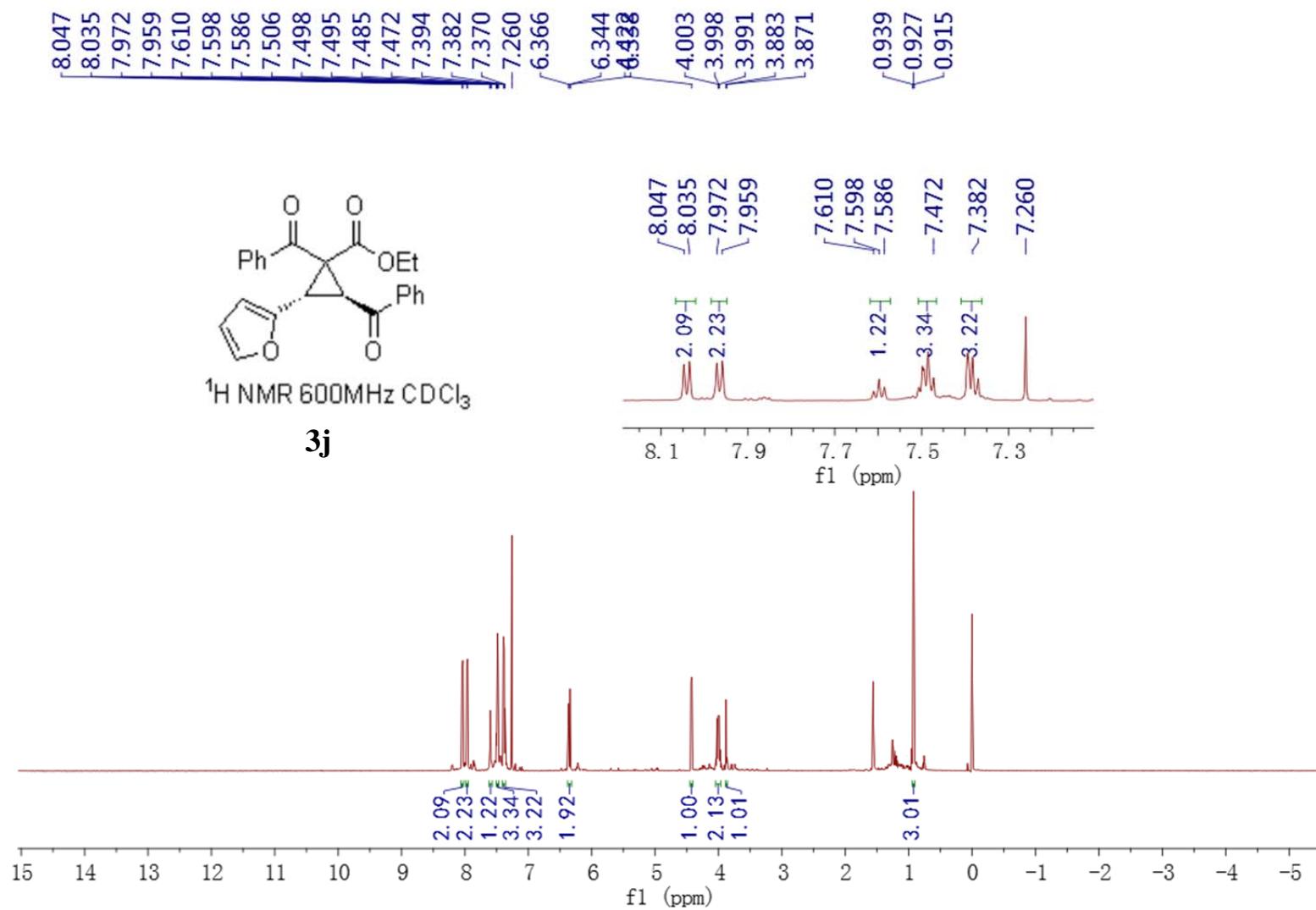


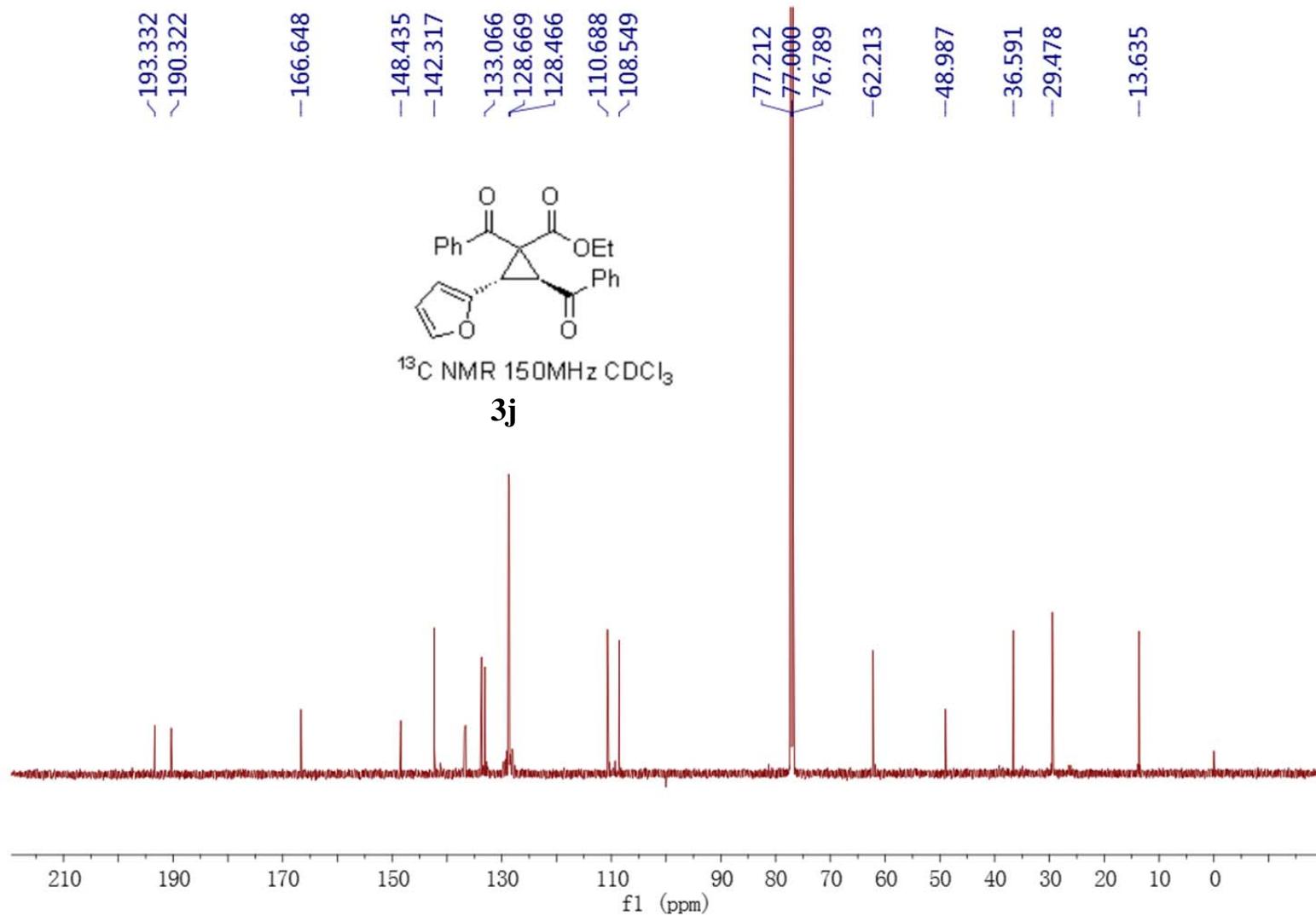




¹H NMR 600MHz CDCl₃

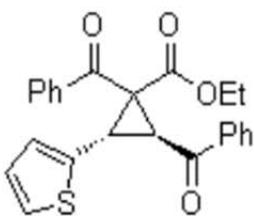
3j





8.048
8.035
8.030
8.026
8.026
7.947
7.930
7.926

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7.472
7.357
7.241
7.083
7.074
6.996
6.974



^1H NMR 400MHz CDCl_3

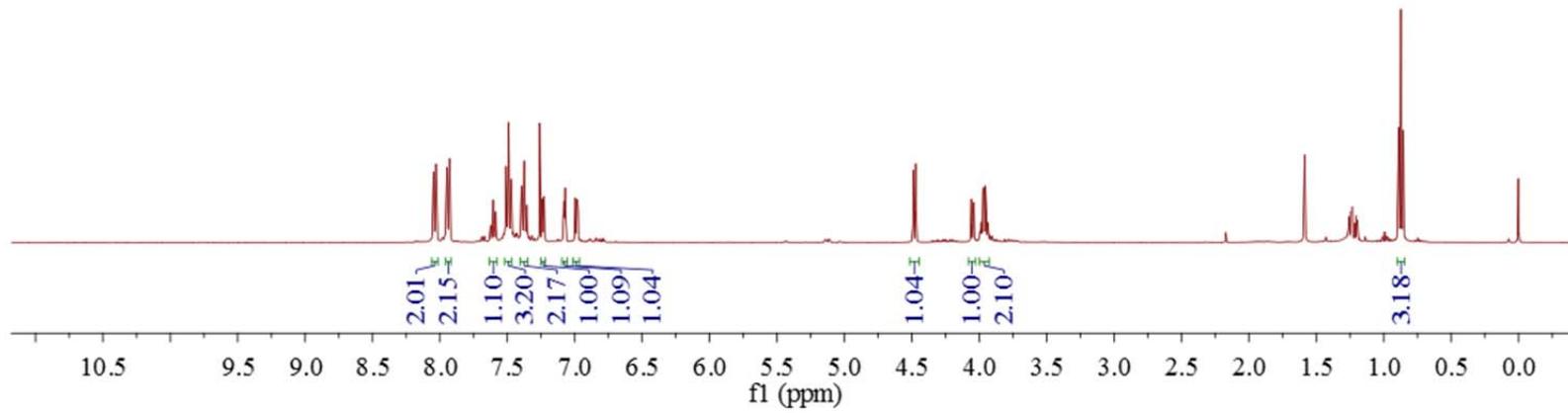
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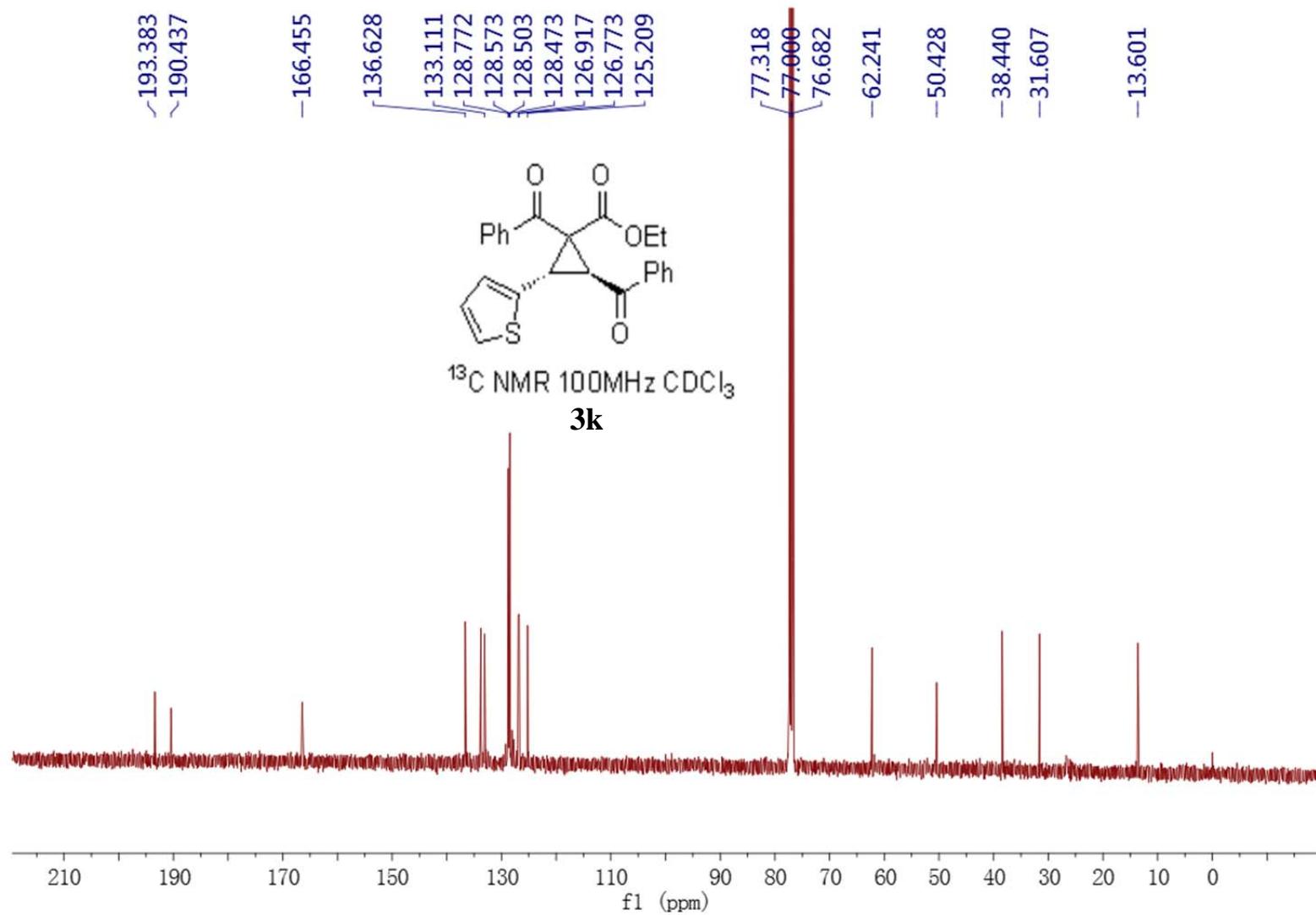
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8.035
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8.026
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4.489
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3.937
3.931

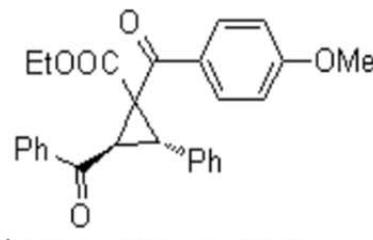
2.01
2.15
2.10
1.10
3.25
2.17
2.17
2.17
1.00
1.09
1.04
7.605
7.587
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6.983

8.2
7.9
7.6
7.3
7.0

f1 (ppm)

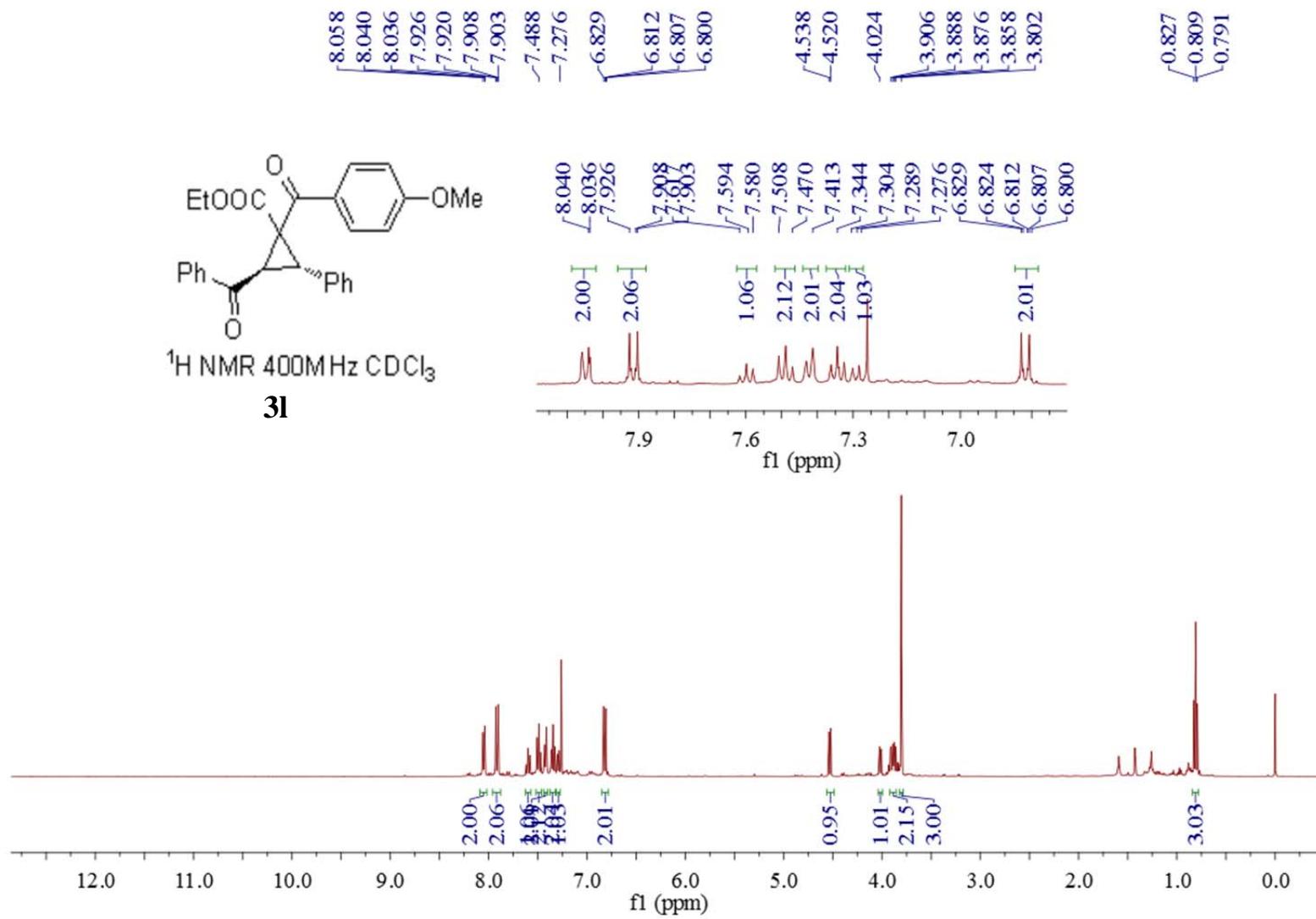


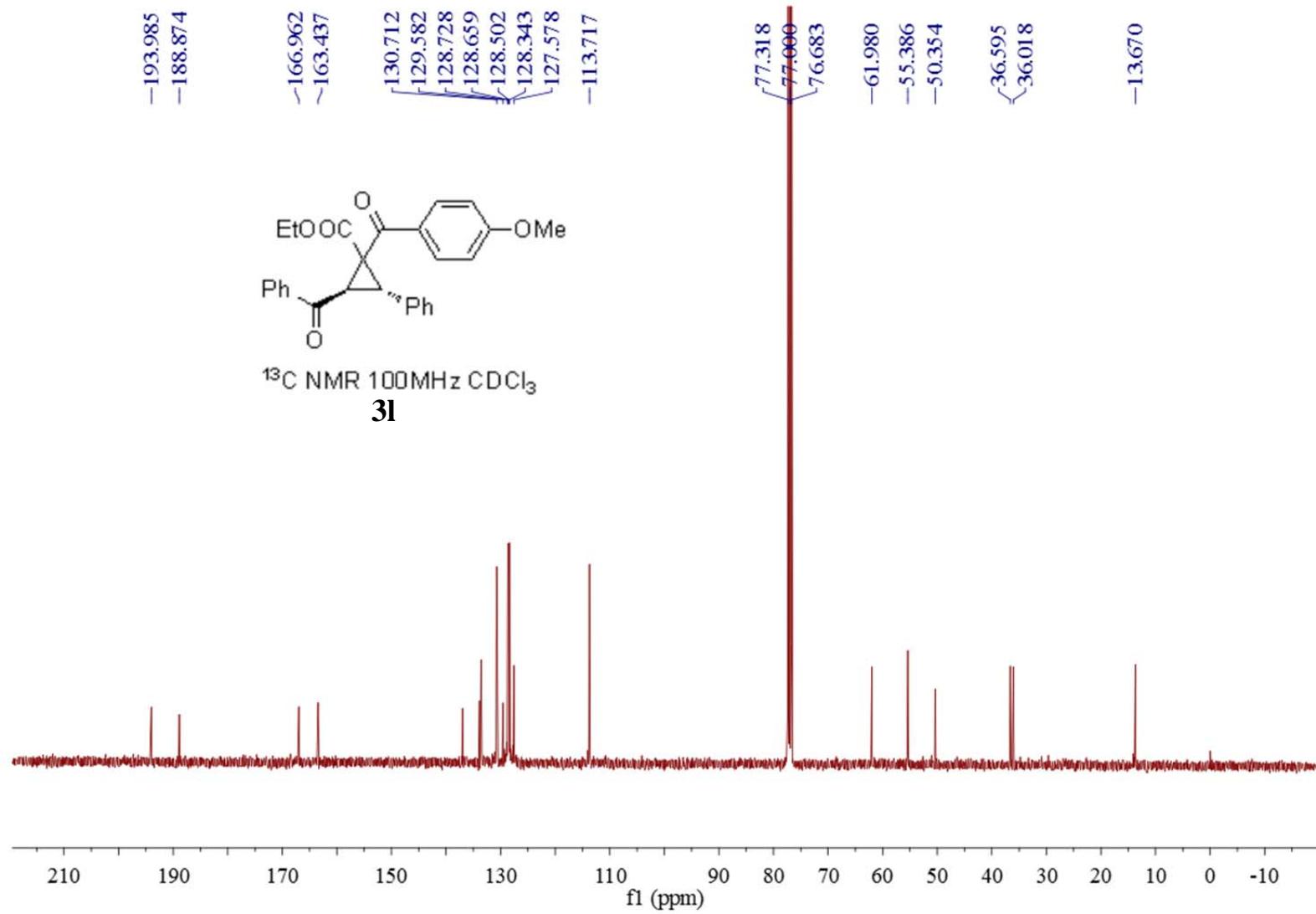


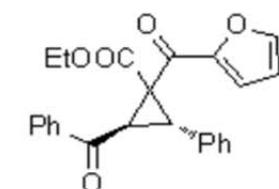


¹H NMR 400MHz CDCl₃

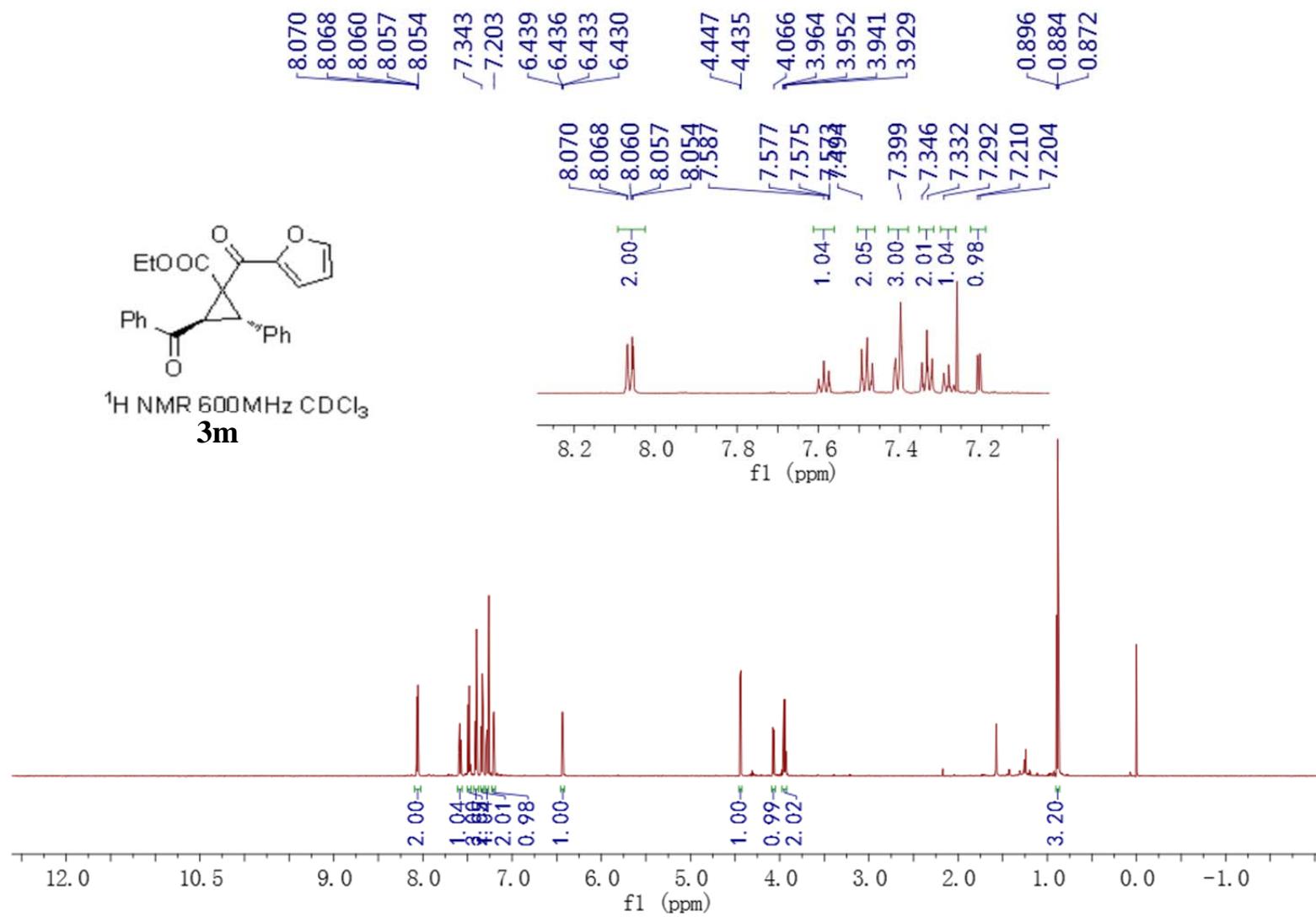
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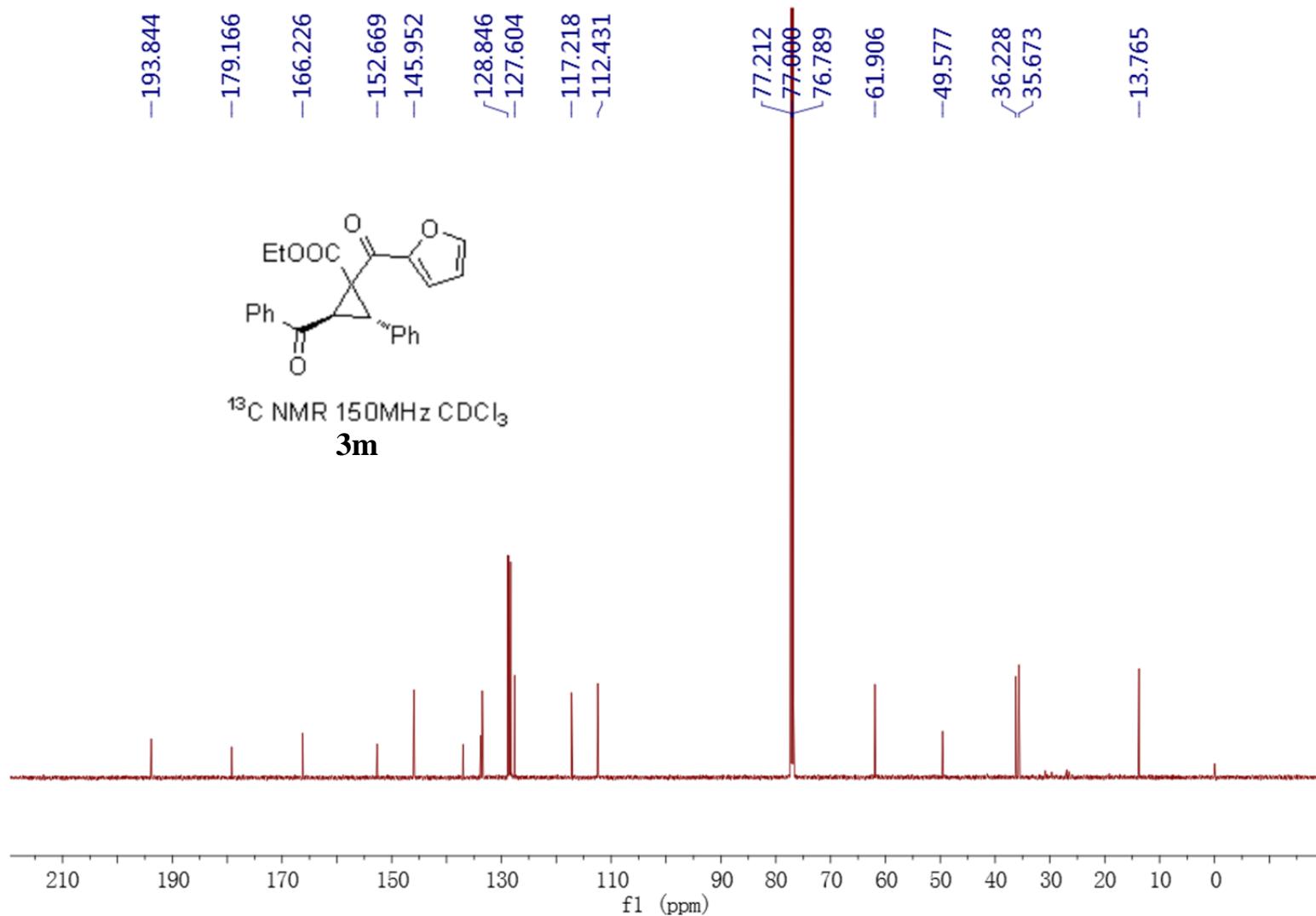


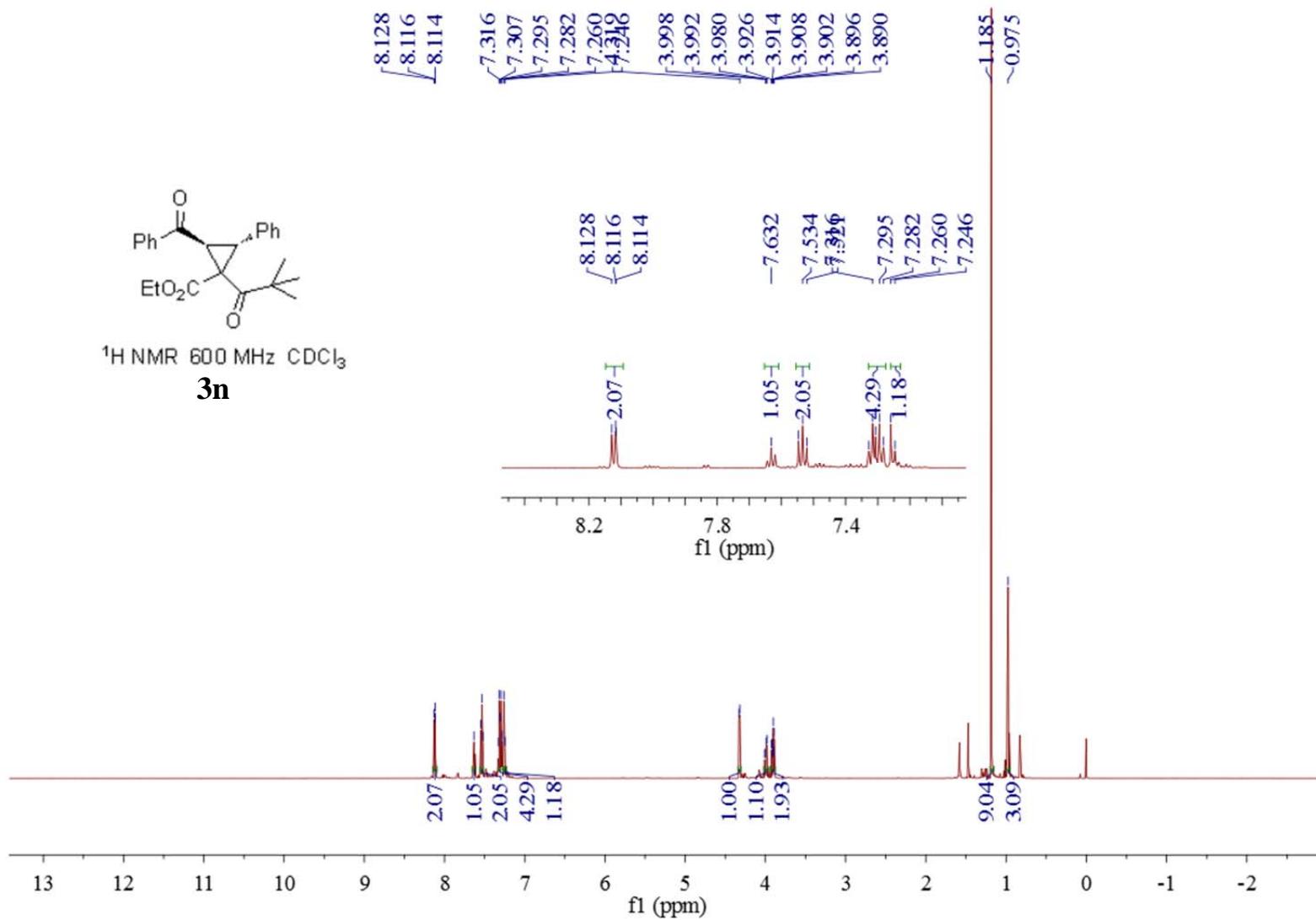


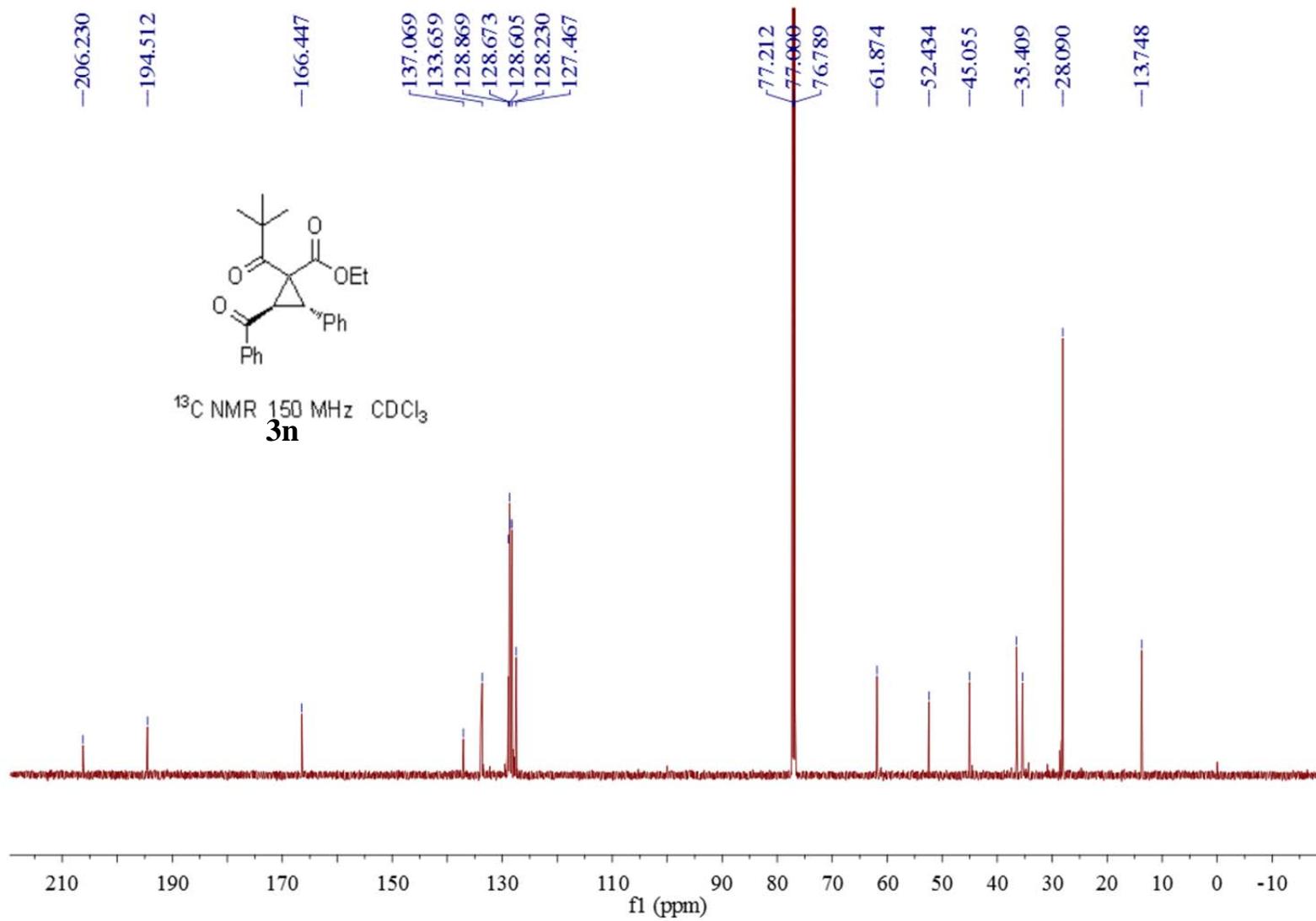


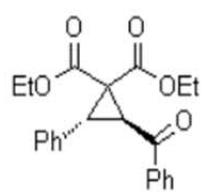
¹H NMR 600 MHz CDCl₃
3m



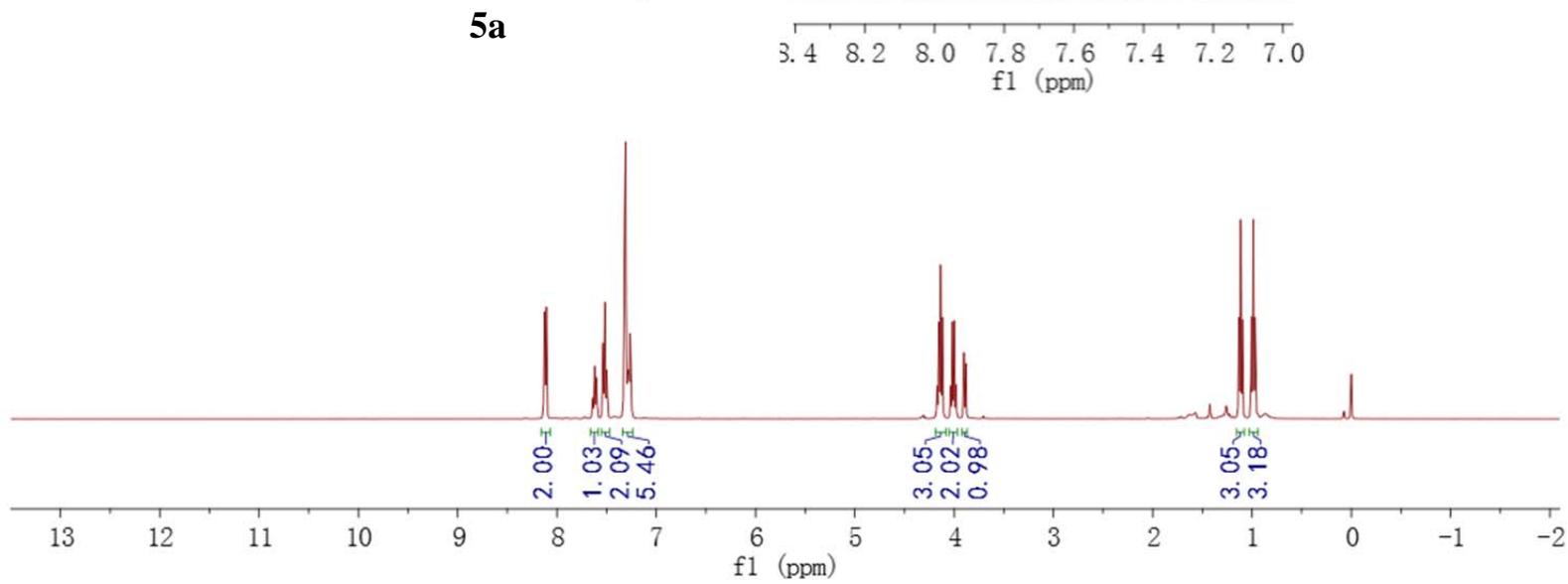


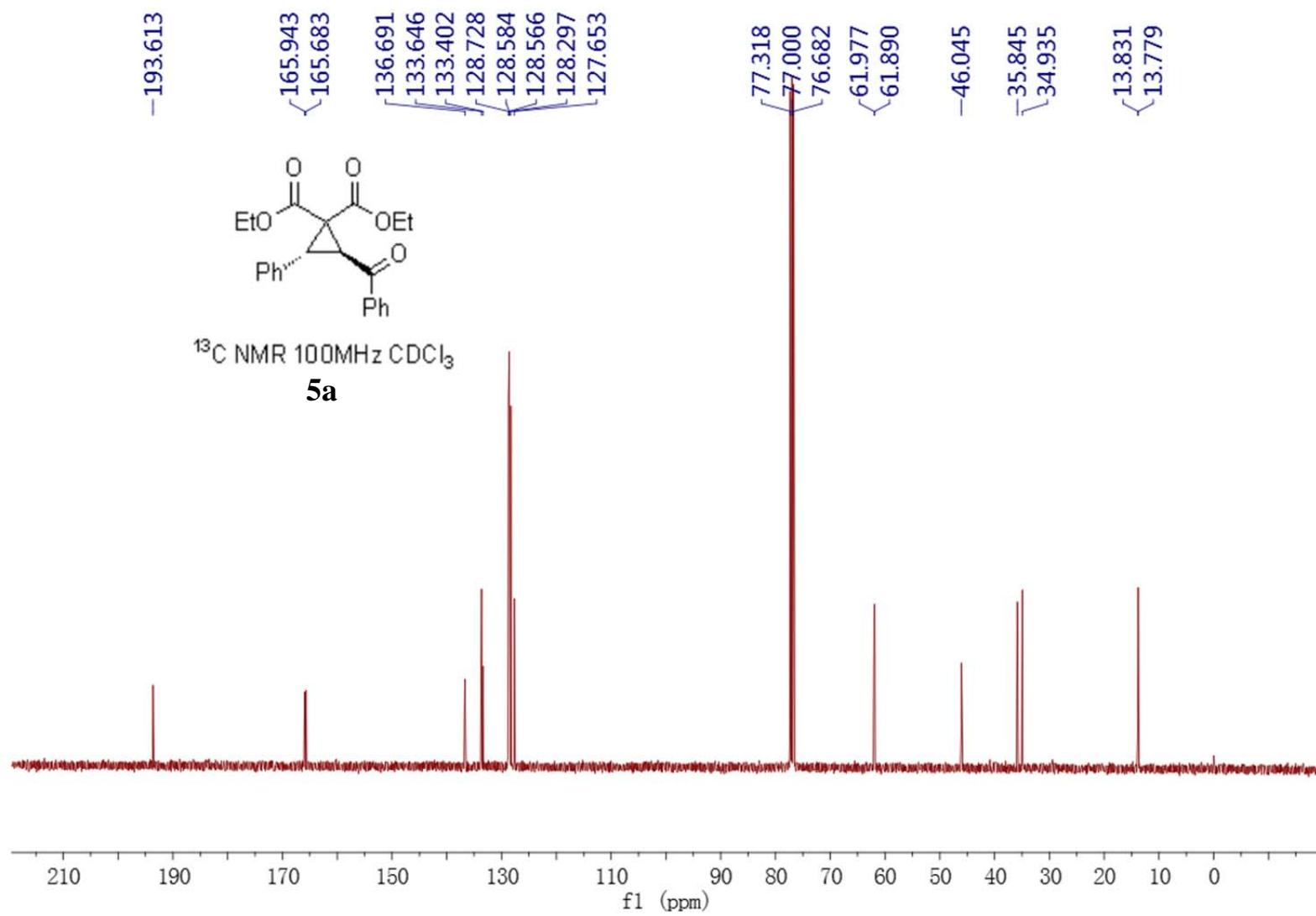


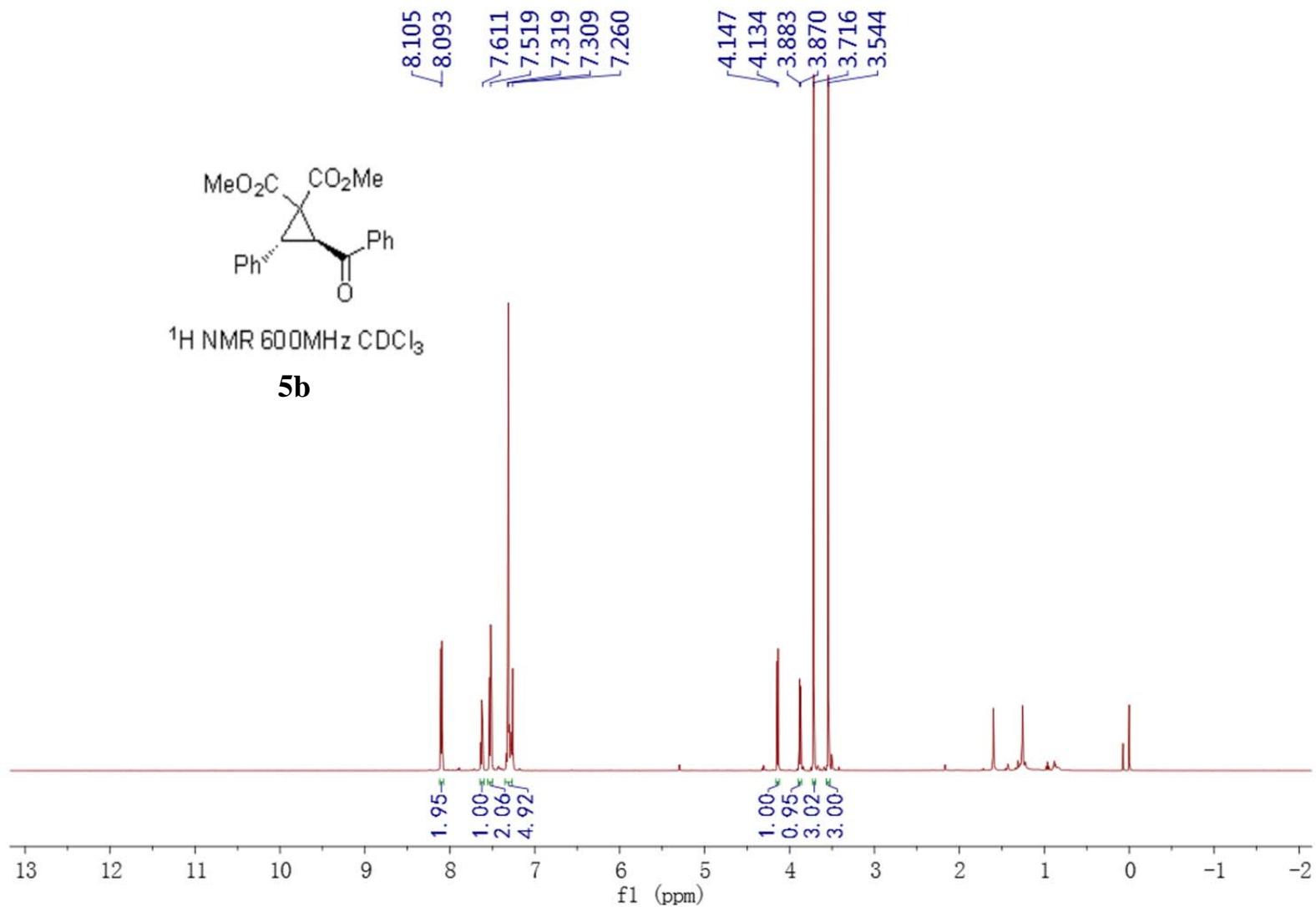


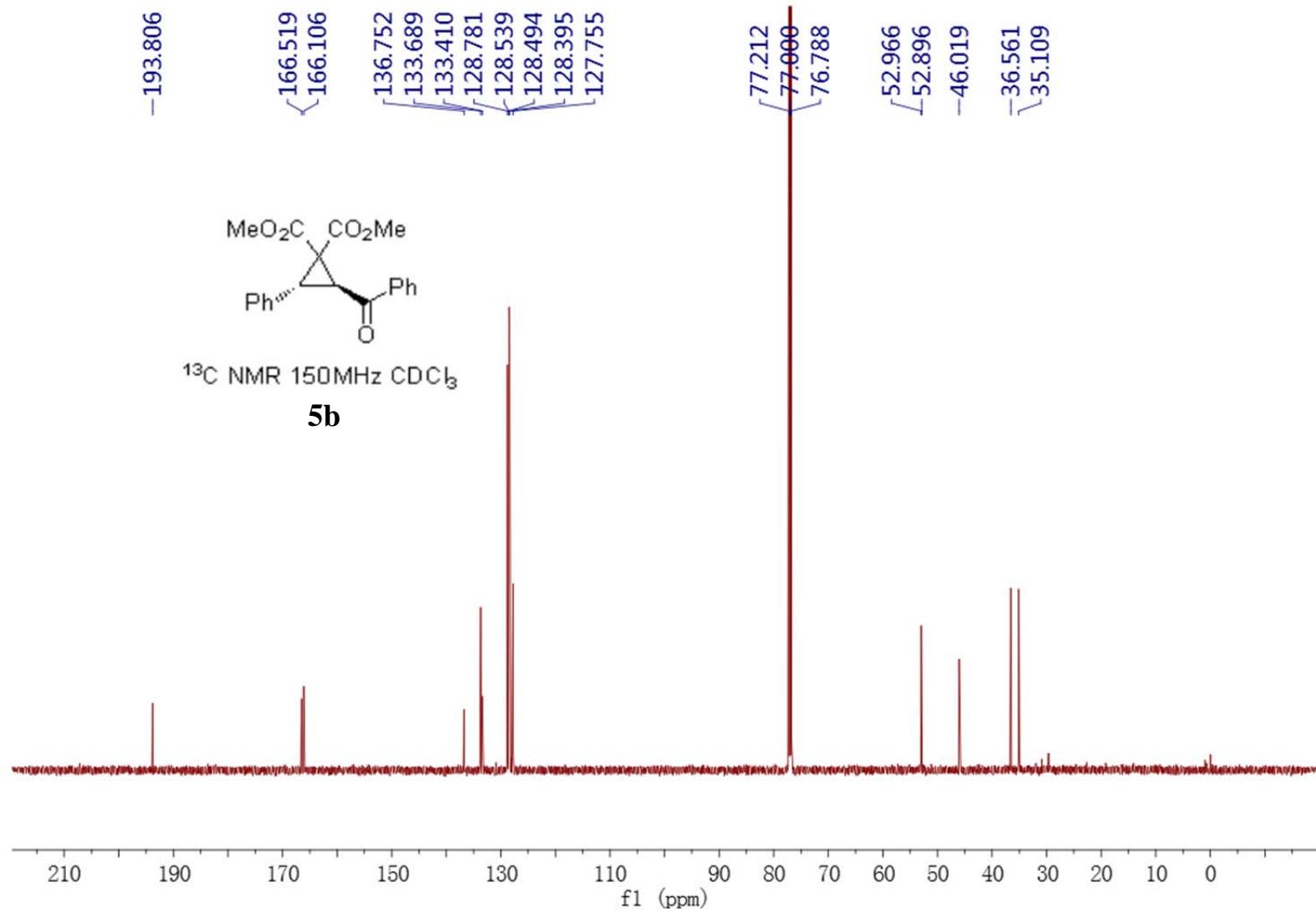


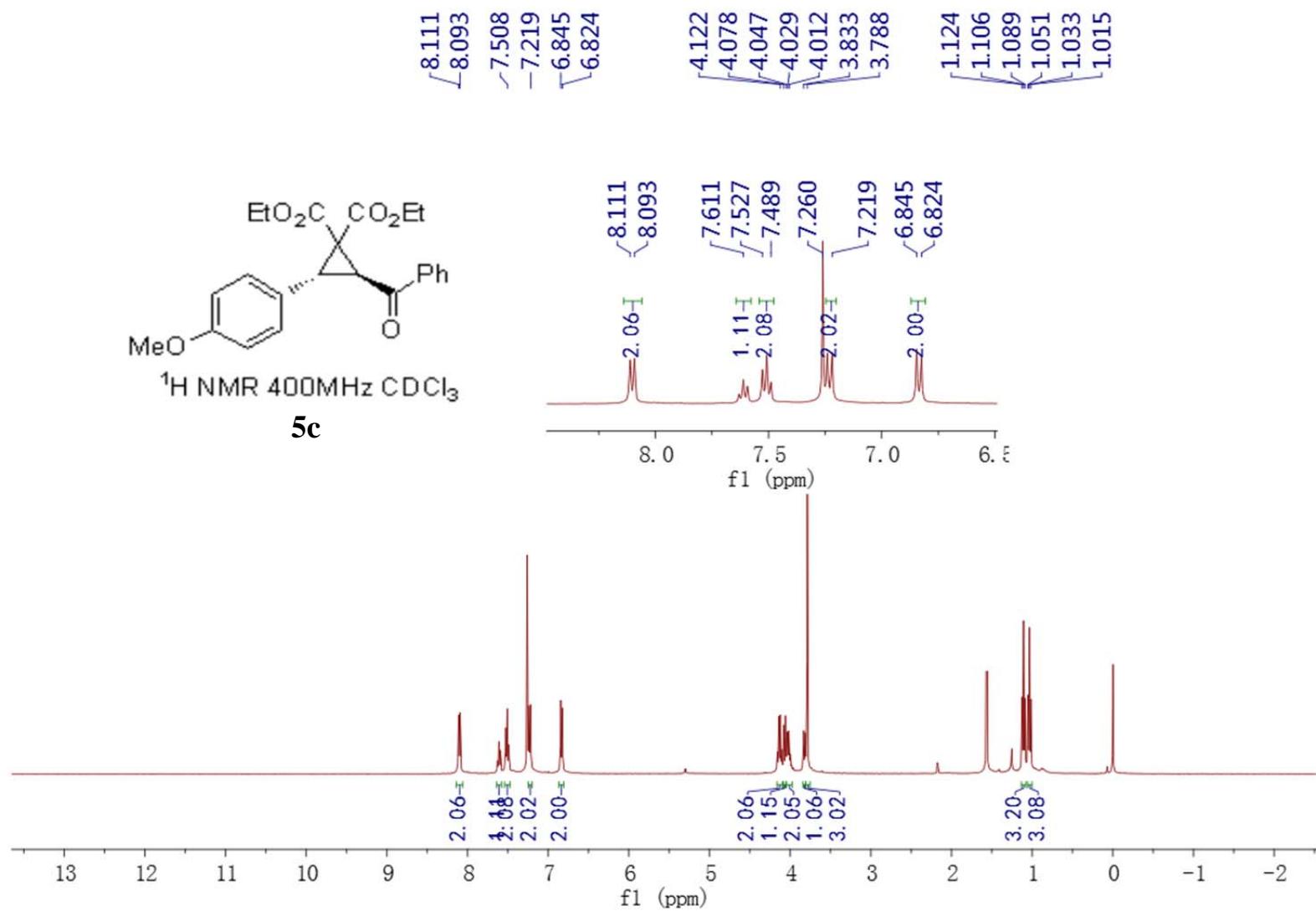
¹H NMR 400MHz CDCl₃

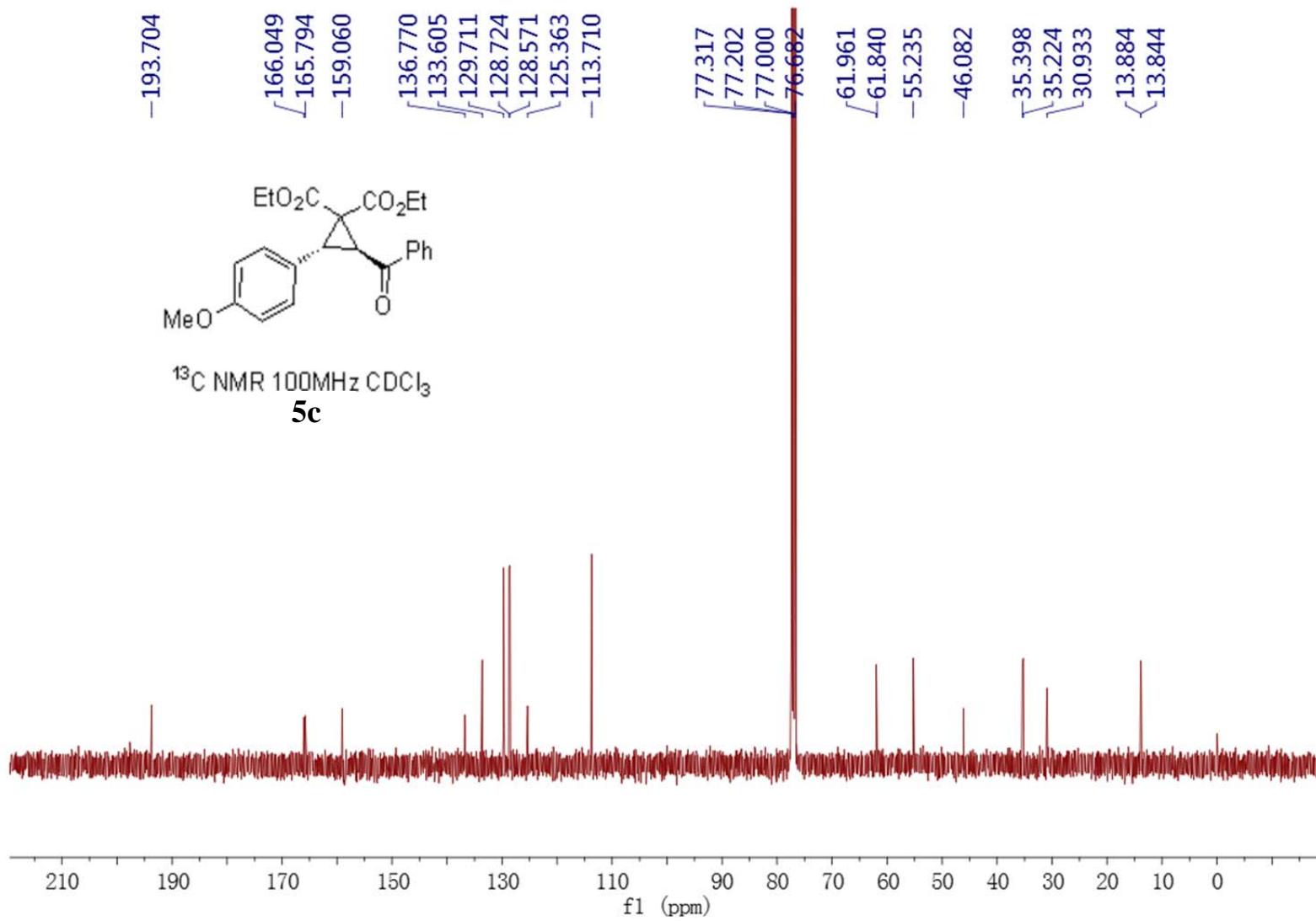


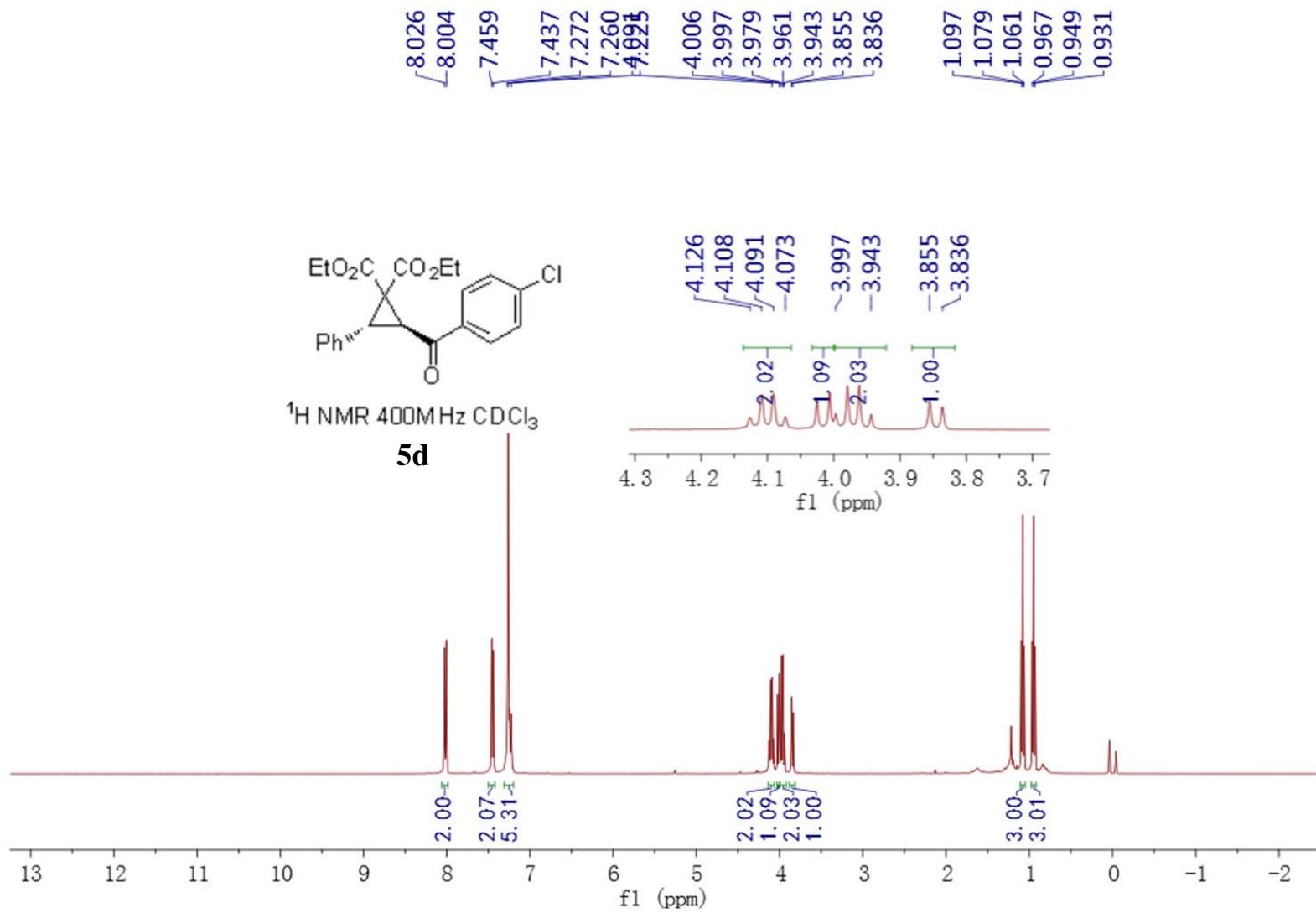


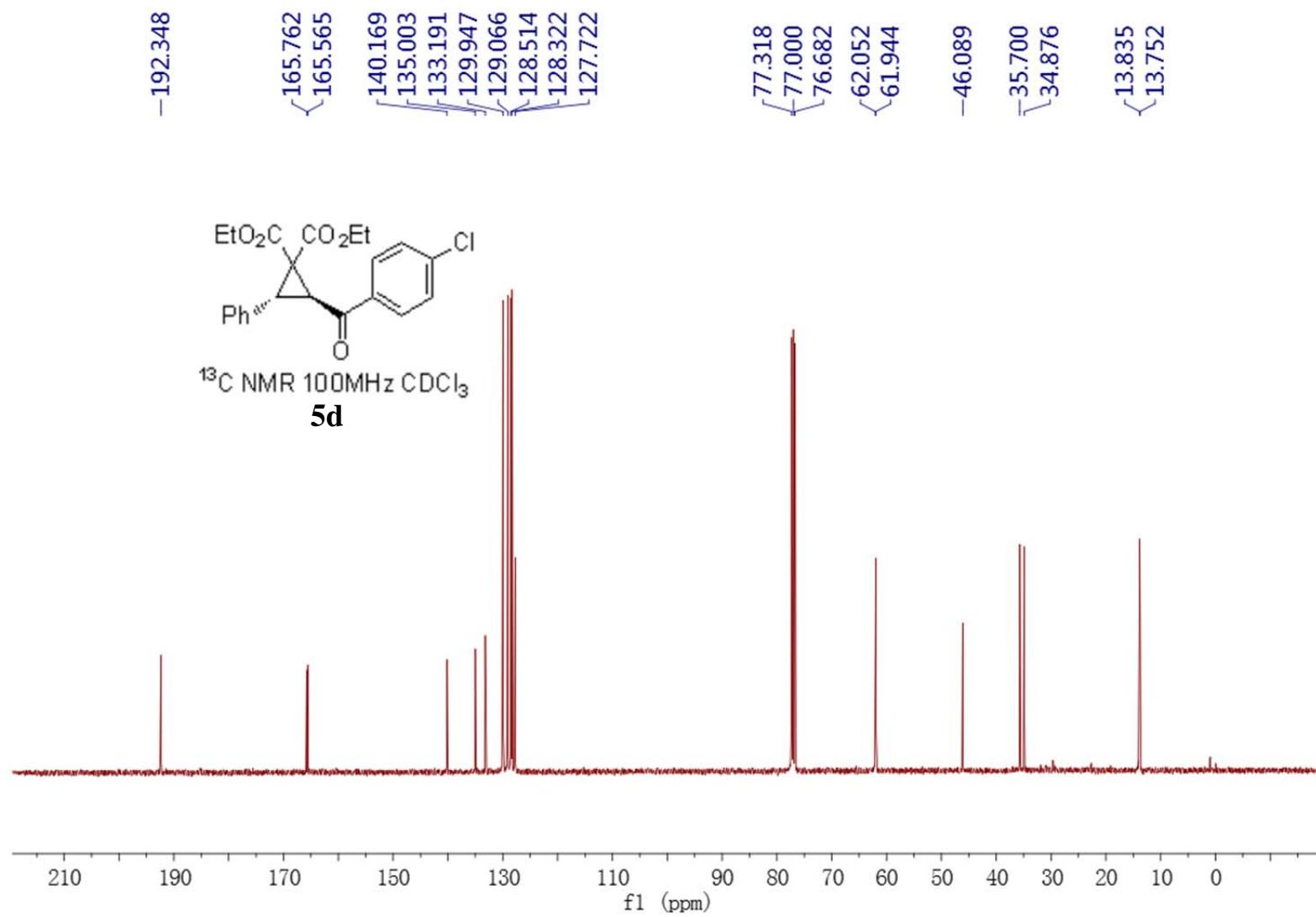


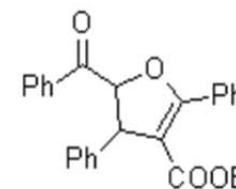
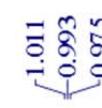
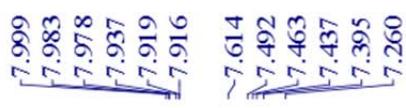












^1H NMR 400MHz CDCl_3

