

Electronic supplementary information

Visible light-mediated Photocatalytic Oxidative Cleavage of Activated Alkynes via Hydroamination: A Direct Approach to Oxamates

Narenderreddy Katta, Mamata Ojha, Arumugavel Murugan, Sagar Arepally, and Duddu S. Sharada*

Department of Chemistry, Indian Institute of Technology Hyderabad, Kandi – 502 285, Sangareddy, Telangana, India, Phone:

(040) 2301 7058; Fax: (040) 2301 6032, E-mail: sharada@iith.ac.in

Table of Contents

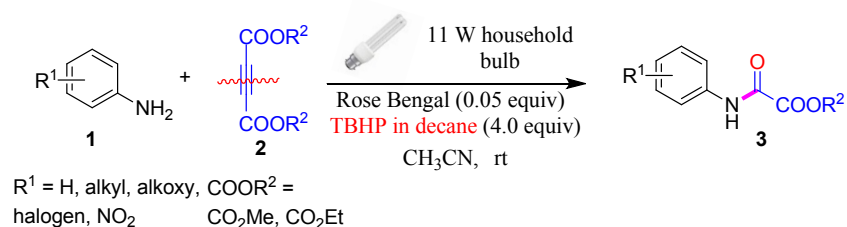
General considerations	(S2)
General procedure (GP-I) for the synthesis of Oxamates ((3aa to 3nb)).....	(S3)
General procedure (GP-II) for the synthesis of 2ac and 2ad	(S4)
General procedure (GP-III) for the synthesis of 2ae	(S5)
Spectral data of all compounds (3aa-3nb).....	(S8-S18)
Copies of ¹ H, ¹³ C NMR spectra of all compounds (3aa-3nb).....	(S18-S49)
References.....	

General Considerations

IR spectra were recorded on an FTIR spectrophotometer. ^1H NMR spectra recorded on 400 MHz spectrometer at 295 K in CDCl_3 ; chemical shifts (δ ppm) and coupling constants (Hz) are reported in standard fashion with reference to either internal standard tetramethylsilane (TMS) ($\delta_{\text{H}} = 0.00$ ppm) or CHCl_3 ($\delta_{\text{H}} = 7.25$ ppm). ^{13}C NMR spectra were recorded on 100 MHz spectrometer at 25 °C in CDCl_3 ; chemical shifts (δ ppm) are reported relative to CHCl_3 [$\delta_{\text{C}} = 77.00$ ppm (central line of triplet)]. In the ^1H NMR, the following abbreviations were used throughout: s = singlet, d = doublet, t = triplet, q = quartet, qui = quintet, dd = doublet of doublets, m = multiplet and br s. = broad singlet. The assignment of signals was confirmed by ^1H , ^{13}C CPD, and DEPT spectra. High-resolution mass spectra (HR-MS) were recorded using Q-TOF multimode source. Melting points were determined on an electrothermal melting point apparatus and are uncorrected. Aqueous tert-butyl hydroperoxide (aqTBHP), tert-butyl hydroperoxide in decane (TBHP in decane) and all metal and metal-free photo catalysts purchased from Sigma Aldrich. Other reagents were purchased as reagent grade and used without further purification. All dry solvents were used, CH_3CN and DCE were dried over CaH_2 , DCM, DMF, and DMSO were dried over P_2O_5 and distilled before use.

All small scale dry reactions were carried out using the standard syringe-septum technique. Reactions were monitored by TLC on silica gel using a combination of petroleum ether and ethyl acetate as eluents. All the reactions are performed in Borosilicate Glass Tubes and Fischer Disposable Borosilicate Glass Tubes (25mL, 16*100mm) with Threaded End (Fischer Scientific Order No. **1495935A**). Solvents were distilled prior to use; petroleum ether with a boiling range of 40 to 60 °C is used. Organic solutions were concentrated by rotary evaporation under vacuum. Acme's silica gel (60-120 mesh) was used for column chromatography (approximately 20 g per one gram of crude material).

1. General procedure (GP-I) for the synthesis of Oxamate derivatives (3aa to 3nb):



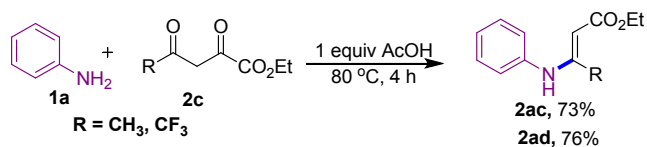
A solution of aniline **1** (1.0 mmol) and alkynes **2** (1.0 mmol) were added slowly in CH₃CN (2 mL) in a 25mL Borosilicate Glass Tube, and the reaction mixture was stirred (if required) at room temperature for 10 min to 3 h. The formation of hydroamination product was monitored by TLC. Then Rose Bengal (0.05 equiv) followed by addition of TBHP in decane (4.0 equiv), and this resulting mixture shifted to room temperature stirred for 48 h under visible light (11 W CFL household bulbs as shown below). Monitored the reaction until hydroaminated product was consumed as indicated by TLC. After completion of the reaction, the reaction mixture was concentrated in vacuo, and purified of the crude product with column chromatography (Silica gel; petroleum ether: ethyl acetate = 9.5:0.5) gave **3aa** to **3na**. All the compounds (**3aa** to **3na**) were confirmed by ¹H NMR and ¹³C NMR spectral analyses.



2. General procedure (GP-II) for the synthesis of 2ac & 2ad:

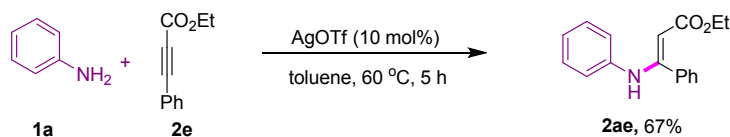
Compound **2ac** & **2ad** was prepared according to the literature method.¹ A mixture of alkyl acetoacetate **2** (1 mmol) and aniline **1a** (0.9 mmol) was refluxed in acetic acid (1 mmol) at 80 °C for 4 h. The progress of the reaction was monitored by TLC. The reaction mixture was quenched by addition of saturated solution

of NaHCO₃ and dried over anhydrous MgSO₄ and concentrated in vacuo. The residue was purified through a silica gel column using petroleum ether/ethyl acetate (9.8:0.2 to 9.9:0.1) as eluent to give the pure product **2ac** & **2ad**.

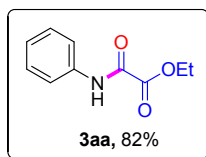


3. General procedure (GP-III) for the synthesis of **2ae**:

Compound **2ae** was prepared according to the literature method.² Aniline **1a** (0.02g, 1 mmol) was added to a Schlenk flask, charged with AgOTf (0.0603g, 0.1 mmol) and alkyne **2e** (0.336g, 1.0 mmol). The mixture was stirred at 60 °C for 5 h, then cooled down to room temperature, diluted with 10 mL dichloromethane and washed with 10 mL H₂O. The aqueous layer was extracted twice with dichloromethane (10 mL), and the organic layer was dried over anhydrous MgSO₄ and concentrated in vacuo. The residue was purified through a silica gel column using petroleum ether/ethyl acetate (9.98/0.02) as eluent to give the pure product **2ae**.

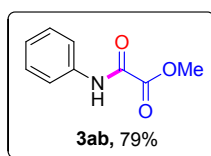


Spectral Data and Copies of ^1H NMR and ^{13}C NMR Spectra:



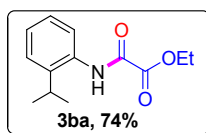
Ethyl 2-oxo-2-(phenylamino)acetate (3aa)

White solid (85mg, 82%); IR (MIR-ATR, 4000–600 cm^{-1}): ν_{max} = 3307.71, 2936.48, 1685.95, 1599.04, 1536.15, 1495.87, 1444.08, 1277.46, 1175.39, 1158.45, 1014.09, 752.00, 691.09; ^1H NMR (CDCl_3 , 400 MHz): δ_{H} = 9.07 (br. s., 1H), 7.64 (d, J = 7.82 Hz, 2H), 7.32 (t, J = 7.58 Hz, 2H), 7.11 - 7.17 (m, 1H), 4.30 - 4.38 (q, J = 6.85 Hz, 2H), 1.32 - 1.37 (t, J = 7.09 Hz, 3H); ^{13}C NMR (CDCl_3 , 100 MHz): 161.0, 154.1, 136.5, 129.2, 125.5, 120.0, 63.7, 14.0. HR-MS (ESI+) m/z calculated for $[\text{C}_{10}\text{H}_{10}\text{NaN}_2\text{O}_5] = [\text{M}+\text{Na}]^+$: 261.0482; found: 261.0482.



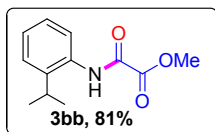
Methyl 2-oxo-2-(phenylamino)acetate (3ab)

White solid (76mg, 79%); IR (MIR-ATR, 4000–600 cm^{-1}): ν_{max} = 3330.70, 2930.40, 1670.55, 1580.01, 1546.20, 1495.87, 1270.46, 1170.12, 1020.10, 740.00, 698.20; ^1H NMR (CDCl_3 , 400 MHz): δ_{H} = 8.91 (br. s., 1H), 7.65 (d, J = 7.83 Hz, 2H), 7.38 (t, J = 7.82 Hz, 2H), 7.17 - 7.22 (m, 1H), 3.96 (s, 3H); ^{13}C NMR (CDCl_3 , 100 MHz): 161.5, 153.6, 136.3, 129.3, 125.6, 119.9, 54.1; HR-MS (ESI+) m/z value calculated for $[\text{C}_{12}\text{H}_{15}\text{NO}_3]^+ = [\text{M}+\text{K}]^+$: 260.0684; found: 260.0684.



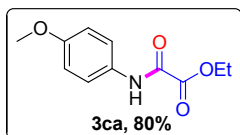
Ethyl 2-((2-isopropylphenyl)amino)-2-oxoacetate (3ba)

White solid (64mg, 74%); ^1H NMR (CDCl_3 , 400 MHz): δ_{H} = 8.96 (br. s., 1H), 7.92 - 7.96 (m, 1H), 7.32 (dd, J = 6.85, 1.96 Hz, 1H), 7.21 - 7.27 (m, 2H), 4.40 - 4.46 (q, J = 6.35, 2H), 3.02 - 3.10 (m, 1H), 1.45 (t, J = 7.09 Hz, 3H), 1.29 (d, J = 6.85 Hz, 6H); ^{13}C NMR (CDCl_3 , 100 MHz): 161.3, 154.2, 139.4, 132.8, 126.7, 126.5, 125.8, 122.8, 63.8, 28.1, 22.9, 14.0. HR-MS (ESI+) m/z value calculated for $[\text{C}_{13}\text{H}_{17}\text{NO}_3]^+ = [\text{M}+\text{H}]^+$: 236.1281; found: 236.1280.



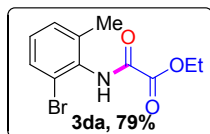
Methyl 2-((2-isopropylphenyl)amino)-2-oxoacetate (**3bb**)

White solid (61mg, 75%); ^1H NMR (CDCl_3 , 400 MHz): $\delta_{\text{H}} = 8.93$ (br. s., 1H), 7.90 - 7.94 (m, 1H), 7.31 - 7.34 (m, 1H), 7.22 - 7.28 (m, 3H), 3.98 (s, 4H), 3.06 (dt, $J = 13.69, 6.85$ Hz, 1H), 1.29 (d, $J = 6.85$ Hz, 6H); ^{13}C NMR (CDCl_3 , 100 MHz): 161.8, 154.0, 139.6, 132.7, 126.7, 126.7, 125.9, 123.0, 54.1, 28.1, 22.9.



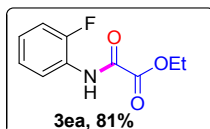
Ethyl 2-((4-methoxyphenyl)amino)-2-oxoacetate (**3ca**)

White solid (68mg, 80%); ^1H NMR (CDCl_3 , 400 MHz): $\delta_{\text{H}} = 8.81$ (br. s., 1H), 7.61-7.52 (m, 2H), 6.94-6.86 (m, $J = 9.3$ Hz, 2H), 4.41 (q, $J = 6.8$ Hz, 2H), 3.81 (s, 3H), 1.43 (t, $J = 7.1$ Hz, 3H); ^{13}C NMR (CDCl_3 , 100 MHz): 161.2, 157.2, 153.7, 129.5, 121.4, 114.4, 63.7, 55.5, 14.0;



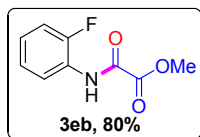
Ethyl 2-((2-bromo-6-methylphenyl)amino)-2-oxoacetate (**3da**)

White solid (61mg, 79%); ^1H NMR (CDCl_3 , 400 MHz): $\delta_{\text{H}} = 9.42$ (br. s., 1H), 8.27 (d, $J = 8.31$ Hz, 1H), 7.33-7.43 (m, 1H), 7.09-7.18 (m, 1H), 4.43 (q, $J = 6.85$ Hz, 2H), 2.31 (s, 3H), 1.42-1.49 (m, 2H); ^{13}C NMR (CDCl_3 , 100 MHz): 160.5, 153.7, 136.6, 132.8, 132.6, 131.9, 129.2, 129.0, 121.2, 115.8, 113.6, 63.8, 20.7, 20.1, 14.0; HR-MS (ESI+) m/z calculated for $[\text{C}_{11}\text{H}_{12}\text{BrNO}_3] = [\text{M}+\text{H}]^+$: 286.0073; found: 286.0072.



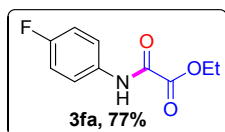
Ethyl 2-((2-fluorophenyl)amino)-2-oxoacetate (**3ea**)

Brown solid (77mg, 81%); ^1H NMR (CDCl_3 , 400 MHz): $\delta_{\text{H}} = 9.13$ (br. s., 1H), 8.37 (t, $J = 7.83$ Hz, 1H), 7.11 - 7.21 (m, 3H), 4.44 (q, $J = 7.17$ Hz, 2H), 1.44 (t, $J = 7.09$ Hz, 3H); ^{13}C NMR (CDCl_3 , 100 MHz): 160.4, 153.9, 151.4, 125.7, 125.0, 124.8, 121.4, 115.1, 63.9, 14.0; HR-MS (ESI+) m/z value calculated for $[\text{C}_{10}\text{H}_{10}\text{FNO}_3]^+ = [\text{M}+\text{Na}]^+$: 235.0571; found: 235.0769.



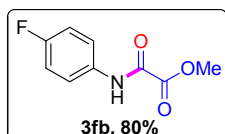
Methyl 2-((2-fluorophenyl)amino)-2-oxoacetate (3eb)

Brown solid (71mg, 80%); ^1H NMR (CDCl_3 , 400 MHz): $\delta_{\text{H}} = 9.10$ (br. s., 1H), 8.38 (t, $J = 7.58$ Hz, 1H), 7.13 - 7.21 (m, 3H), 3.99 (s, 3H); ^{13}C NMR (CDCl_3 , 100 MHz): 160.9, 153.7, 151.4, 125.9, 124.8, 121.5, 115.1, 54.2; HR-MS (ESI+) m/z value calculated for $[\text{C}_{10}\text{H}_{10}\text{FNO}_3]^+ = [\text{M}+\text{Na}]^+$: 235.0571; found: 235.0769.



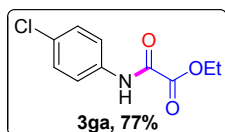
Ethyl 2-((2-fluorophenyl)amino)-2-oxoacetate (3fa)

Brown solid (73mg, 77%); ^1H NMR (CDCl_3 , 400 MHz): $\delta_{\text{H}} = 8.87$ (br. s., 1H), 7.58 - 7.68 (m, 2H), 7.03 - 7.13 (m, 2H), 4.43 (q, $J = 7.01$ Hz, 2H), 1.44 (t, $J = 7.09$ Hz, 3H); ^{13}C NMR (CDCl_3 , 100 MHz): 161.0, 158.8, 153.8, 132.4, 121.5, 116.0, 63.8, 14.0; HR-MS (ESI+) m/z value calculated for $[\text{C}_{10}\text{H}_{10}\text{FNO}_3]^+ = [\text{M}+\text{H}]^+$: 212.0716; found: 212.0717.



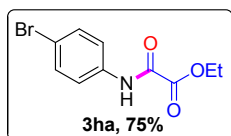
Methyl 2-(((4-fluorophenyl)amino)-2-oxoacetate (3fb)

White solid (71mg, 80%); ^1H NMR (CDCl_3 , 400 MHz): $\delta_{\text{H}} = 8.91$ (br. s., 1H), 7.59 - 7.66 (m, 2H), 7.03 - 7.12 (m, 2H), 3.97 (s, 3H); ^{13}C NMR (CDCl_3 , 100 MHz): 161.3, 158.8, 153.6, 132.3, 121.7, 116.1, 54.1; HR-MS (ESI+) m/z value calculated for $[\text{C}_9\text{H}_8\text{FNO}_3]^+ = [\text{M}+\text{K}]^+$: 236.0117.9476; found: 236.0120.



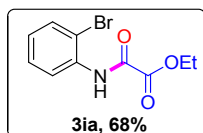
Ethyl 2-((4-chlorophenyl)amino)-2-oxoacetate (3ga)

White solid (64mg, 77%); ^1H NMR (CDCl_3 , 400 MHz): $\delta_{\text{H}} = 8.97$ (br. s., 1H), 7.62 (d, $J = 8.31$ Hz, 2H), 7.34 (d, $J = 8.31$ Hz, 2H), 4.41 (q, $J = 6.85$ Hz, 2H), 1.42 (t, $J = 6.85$ Hz, 3H); ^{13}C NMR (CDCl_3 , 100 MHz): 160.8, 153.9, 134.9, 130.6, 129.3, 121.1, 63.9, 14.0; HR-MS (ESI+) m/z value calculated for $[\text{C}_{10}\text{H}_{10}\text{ClNO}_3]^+ = [\text{M}+\text{Na}]^+ : 250.0241$; found: 250.0241.



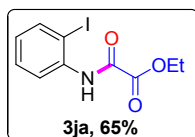
Ethyl 2-((4-bromophenyl)amino)-2-oxoacetate (3ha)

Yellow solid (56mg, 75%); ^1H NMR (CDCl_3 , 400 MHz): $\delta_{\text{H}} = 8.95$ (br. s., 1H), 7.56 (m, $J = 8.31$ Hz, 2H), 7.49 (m, $J = 8.80$ Hz, 2H), 4.41 (q, $J = 6.85$ Hz, 2H), 1.42 (t, $J = 7.09$ Hz, 3H); ^{13}C NMR (CDCl_3 , 100 MHz): 160.8, 153.9, 135.4, 132.3, 121.4, 118.3, 63.9, 14.0; HR-MS (ESI+) m/z value calculated for $[\text{C}_{10}\text{H}_{10}\text{BrNO}_3]^+ = [\text{M}+\text{Na}]^+ : 293.9739$; found: 293.9736.



Ethyl 2-((2-bromophenyl)amino)-2-oxoacetate (3ia)

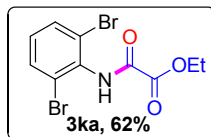
Yellow solid (51mg, 68%); ^1H NMR (CDCl_3 , 400 MHz): $\delta_{\text{H}} = 9.51$ (br. s., 1H), 8.43 (dd, $J = 8.31, 0.98$ Hz, 1H), 7.59 (dd, $J = 8.07, 1.22$ Hz, 1H), 7.34 - 7.39 (m, 1H), 7.04 - 7.09 (m, 1H), 4.41 - 4.47 (m, 2H), 1.41 - 1.47 (m, 3H); ^{13}C NMR (CDCl_3 , 100 MHz): 160.5, 153.9, 134.4, 132.5, 128.6, 126.4, 121.4, 113.8, 63.9, 14.0. HR-MS (ESI+) m/z value calculated for $[\text{C}_{10}\text{H}_{10}\text{BrNO}_3]^+ = [\text{M}+\text{K}]^+ : 309.9476$; found: 309.9475.



Ethyl 2-((2-iodophenyl)amino)-2-oxoacetate (3ja)

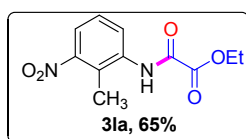
Brown solid (47mg, 65%); ^1H NMR (CDCl_3 , 400 MHz): $\delta_{\text{H}} = 9.37$ (br. s., 1H), 8.35 (dd, $J = 8.31, 1.47$ Hz, 1H), 7.83 (dd, $J = 7.83, 1.47$ Hz, 1H), 7.37 - 7.42 (m, 1H), 6.92 (td, $J = 7.58, 1.47$ Hz, 1H), 4.43 - 4.48 (m, 2H), 1.43 - 1.47 (m, 3H); ^{13}C NMR (CDCl_3 , 100 MHz): 160.5, 154.0, 139.1, 137.0, 129.5, 127.0, 121.3,

89.6, 63.9, 14.0. HR-MS (ESI+) m/z value calculated for $[C_{10}H_{10}INO_3]^+ = [M+NH_4]^+$: 337.0042; found: 337.0044.



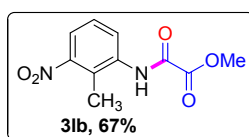
Ethyl 2-((2,6-dibromophenyl)amino)-2-oxoacetate (3ka)

Yellow solid (39mg, 62%); 1H NMR ($CDCl_3$, 400 MHz): $\delta_H = 9.48$ (br. s., 1 H), 8.63 (d, $J = 2.4$ Hz, 1 H), 7.43 (d, $J = 8.8$ Hz, 1 H), 7.24 - 7.14 (m, 1 H), 4.45 (q, $J = 7.3$ Hz, 2 H), 1.45 (t, $J = 7.1$ Hz, 3 H); ^{13}C NMR ($CDCl_3$, 100 MHz): 208.3, 160.2, 153.9, 135.5, 133.4, 132.6, 129.3, 128.3, 124.1, 122.2, 119.4, 115.7, 112.2, 64.1, 14.0; HR-MS (ESI+) m/z calculated for $[C_{10}H_9Br_2NO_3] = [M+NH_4]^+$: 368.9267; found: 368.9266.



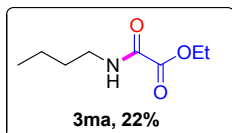
Ethyl 2-((2-methyl-3-nitrophenyl)amino)-2-oxoacetate (3la)

Yellow solid (54mg, 65%); 1H NMR ($CDCl_3$, 400 MHz): $\delta_H = 8.98$ (br. s., 1H), 8.22 (d, $J = 8.31$ Hz, 1H), 7.69 (d, $J = 8.31$ Hz, 1H), 7.41 (t, $J = 8.07$ Hz, 1H), 4.43 - 4.49 (q, $J = 6.31$ Hz, 2H), 2.45 (s, 3H), 1.43 - 1.47 (t, $J = 7.27$ Hz, 3H); ^{13}C NMR ($CDCl_3$, 100 MHz): 160.7, 154.3, 151.1, 136.0, 127.2, 126.4, 123.9, 121.5, 64.2, 14.0, 13.3. HR-MS (ESI+) m/z value calculated for $[C_{11}H_{12}BrN_2O_5]^+ = [M+NH_4]^+$: 270.1083; found: 270.1084.



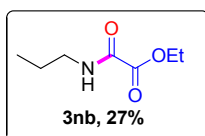
Methyl 2-((2-methyl-3-nitrophenyl)amino)-2-oxoacetate (3lb)

Yellow solid (62mg, 75%); 1H NMR ($CDCl_3$, 400 MHz): $\delta_H = 8.95$ (br. s., 1H), 8.21 (d, $J = 8.31$ Hz, 1H), 7.70 (d, $J = 8.31$ Hz, 1H), 7.41 (t, $J = 8.07$ Hz, 1H), 4.02 (s, 3H), 2.45 (s, 3H); ^{13}C NMR ($CDCl_3$, 100 MHz): 161.1, 154.0, 151.2, 135.8, 127.3, 126.5, 124.0, 121.7, 54.4, 13.3.



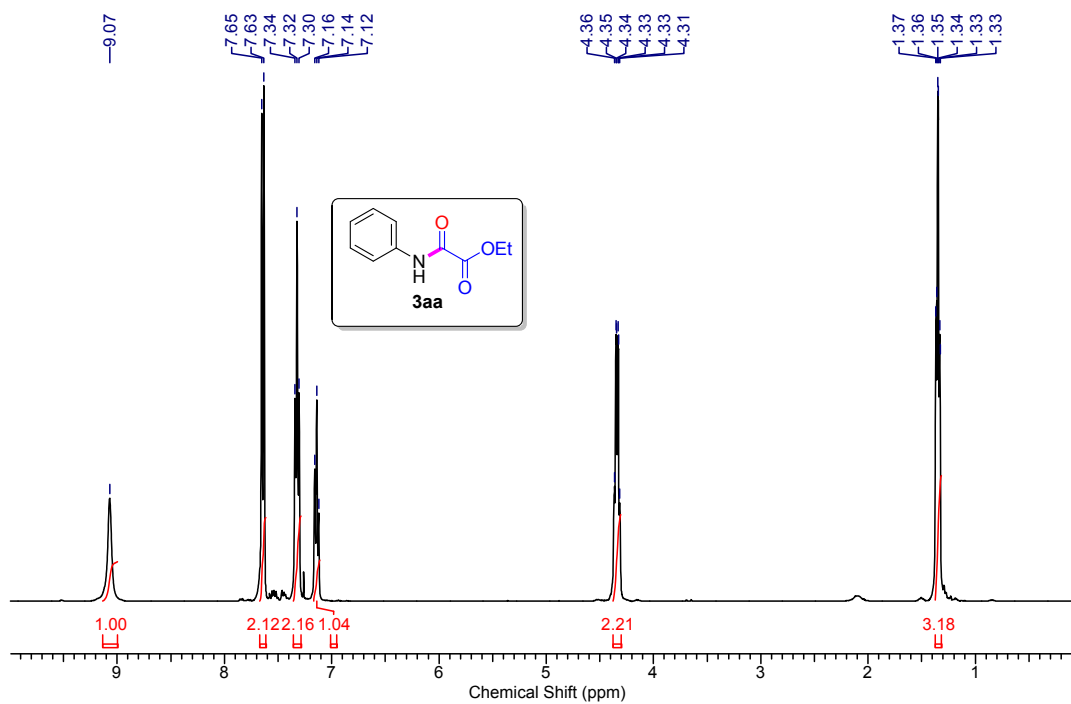
Ethyl 2-(butylamino)-2-oxoacetate (3ma)

Brown liquid (26mg, 22%); ^1H NMR (CDCl_3 , 400 MHz): $\delta_{\text{H}} = 7.28$ (br. s., 1H), 4.35 (q, $J = 7.17$ Hz, 2H), 3.35 (q, $J = 6.52$ Hz, 2H), 1.52 - 1.60 (m, 2H), 1.34 - 1.43 (m, 5H), 0.94 (t, $J = 7.34$ Hz, 3H); ^{13}C NMR (CDCl_3 , 100 MHz): 160.8, 156.6, 63.1, 39.6, 31.1, 19.9, 13.9, 13.6; HR-MS (ESI+) m/z value calculated for $[\text{C}_8\text{H}_{15}\text{NO}_3]^+ = [\text{M}+\text{Na}]^+$: 197.0976; found: 197.0971.

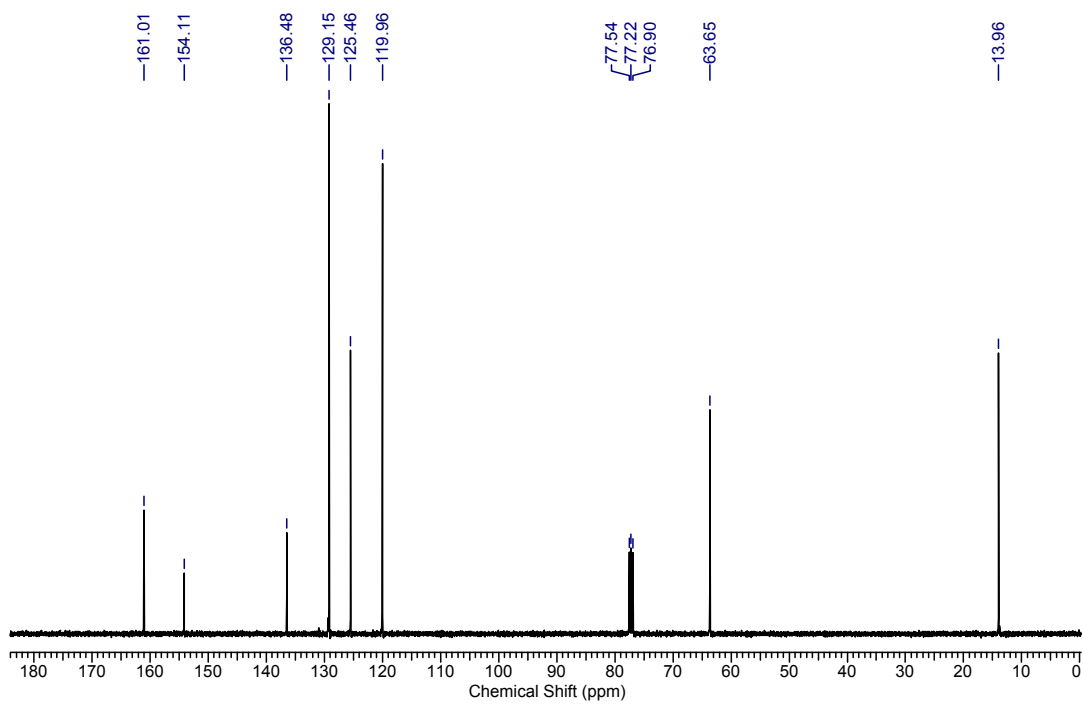


Ethyl 2-(propylamino)-2-oxoacetate (3nb)

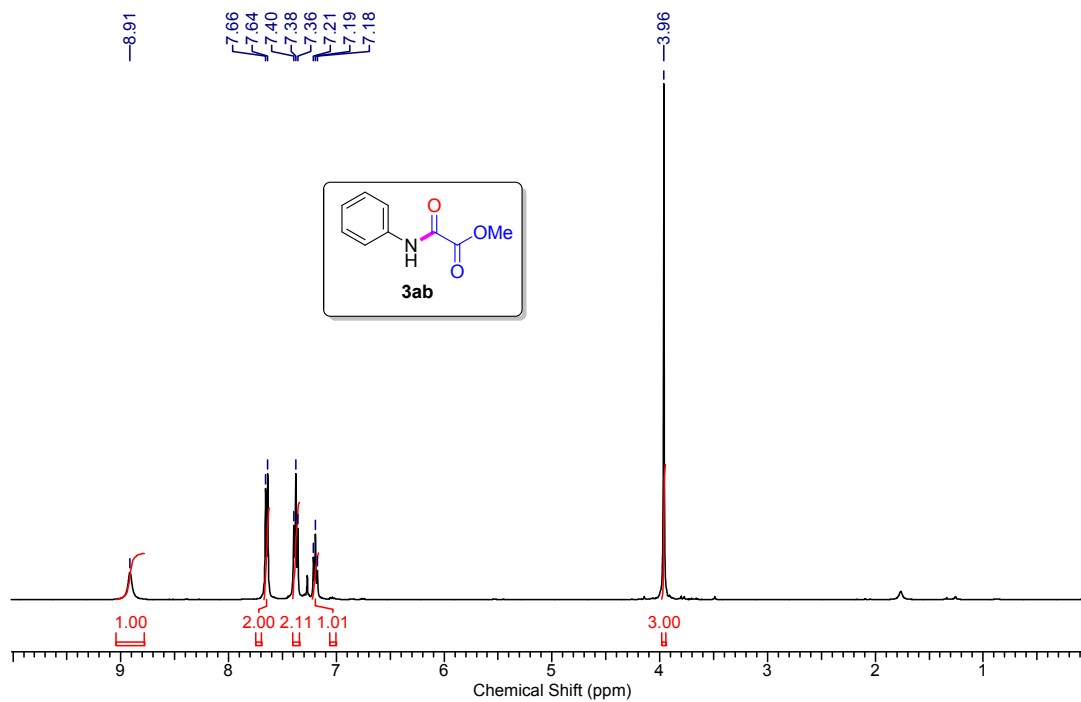
Brown liquid (36mg, 27%); ^1H NMR (CDCl_3 , 400 MHz): $\delta_{\text{H}} = 7.14$ (br. s., 1H), 4.33 (q, $J = 6.8$ Hz, 2H), 3.34-3.25 (m, 2H), 1.64-1.54 (m, 2H), 1.38-1.33 (m, 2H), 0.94 (t, $J = 7.3$ Hz, 3H); ^{13}C NMR (CDCl_3 , 100 MHz): 160.9, 156.6, 63.2, 41.6, 22.4, 14.0, 11.3; HR-MS (ESI+) m/z value calculated for HR-MS (ESI+) m/z calculated for $[\text{C}_7\text{H}_{13}\text{NO}_3] = [\text{M}+\text{H}]^+$: 169.0974; found: 169.0973.



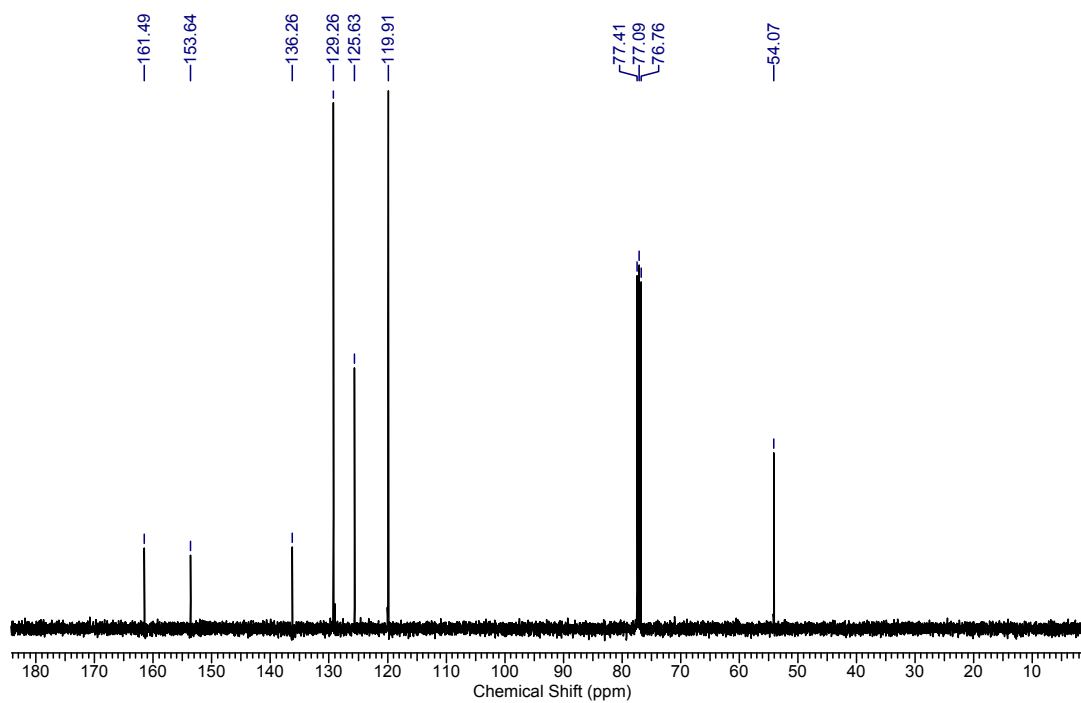
¹H NMR (400 MHz) spectrum of compound 3aa in CDCl₃



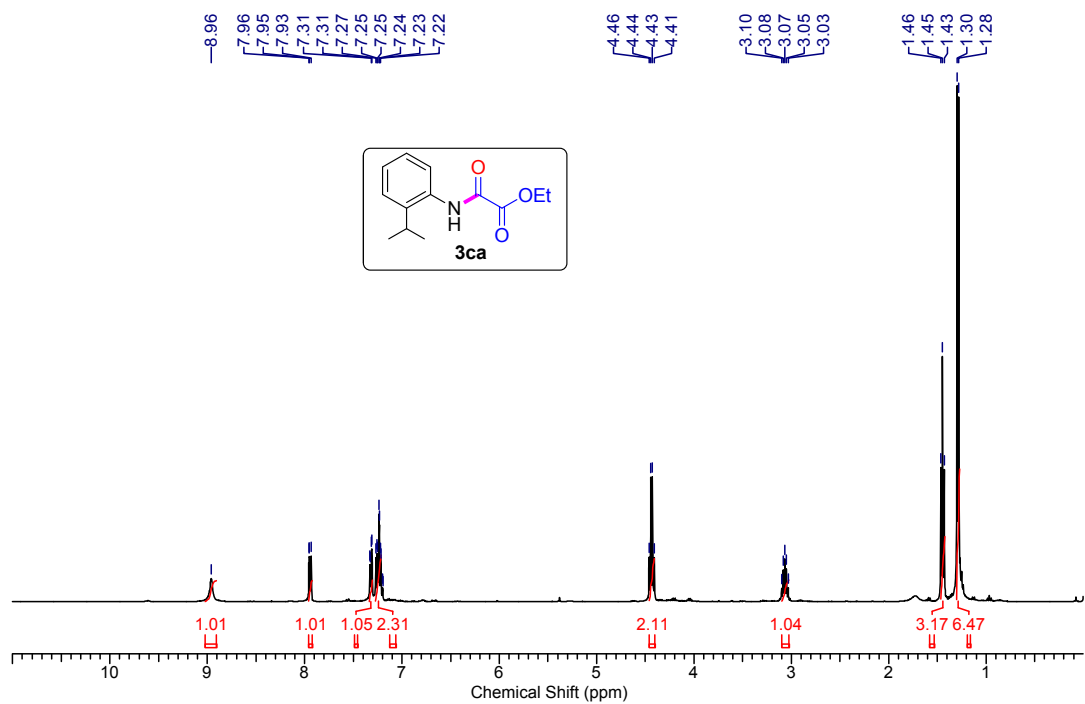
¹³C NMR (100 MHz) spectrum of compound 3aa in CDCl₃



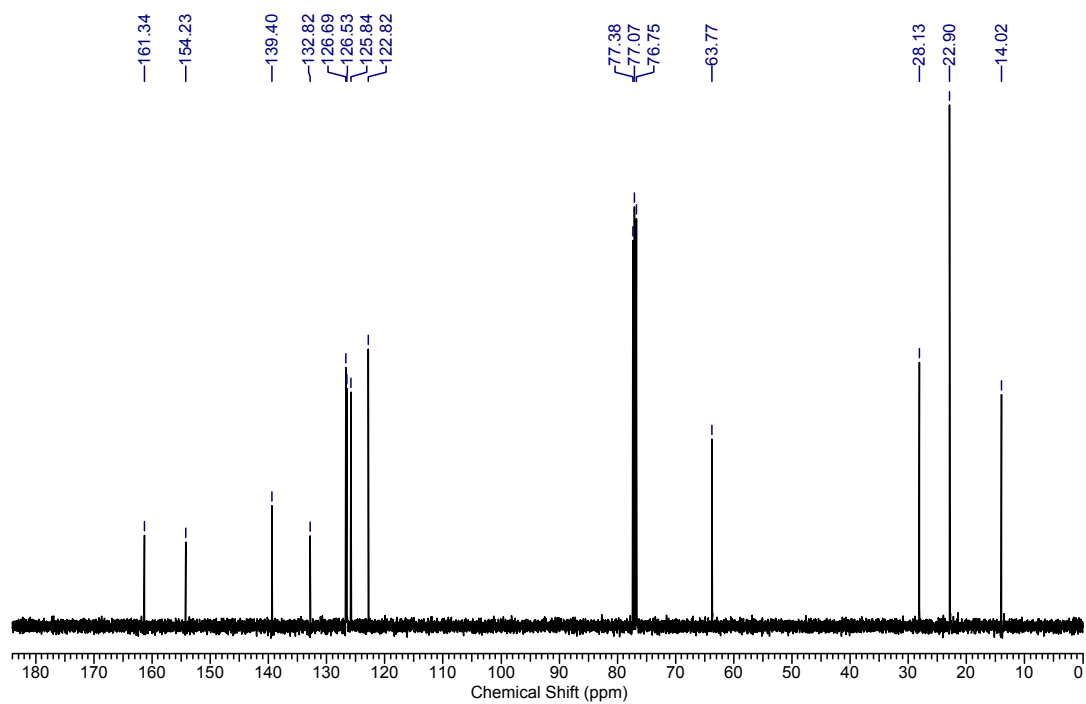
¹H NMR (400 MHz) spectrum of compound 3ab in CDCl₃



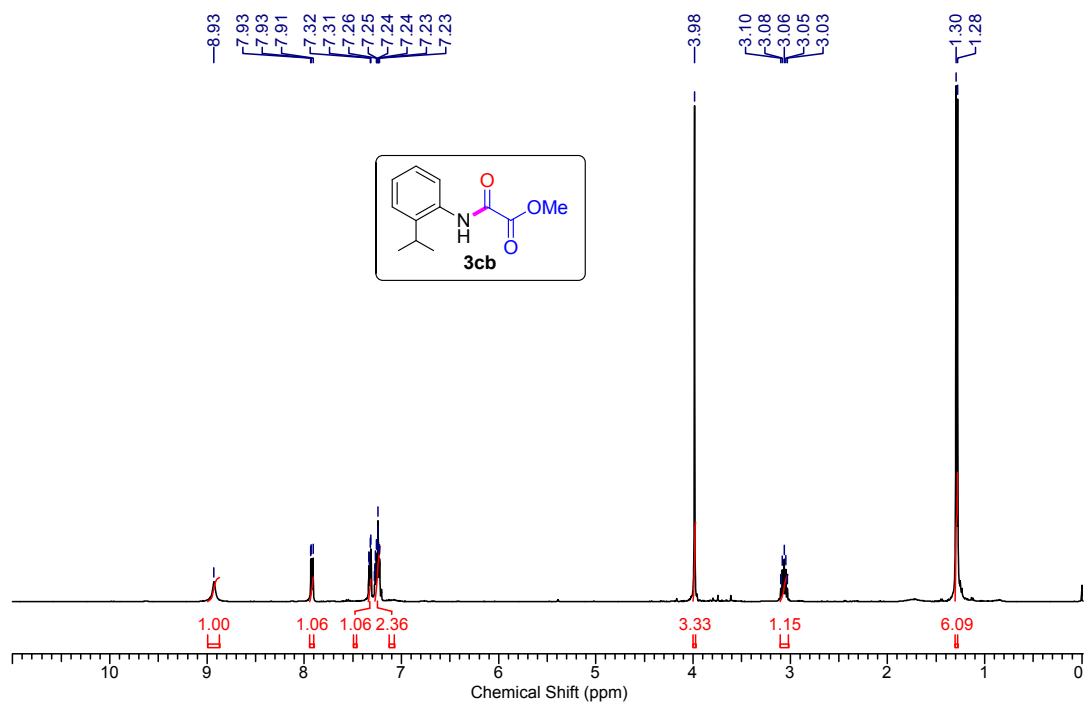
¹³C NMR (100 MHz) spectrum of compound 3ab in CDCl₃



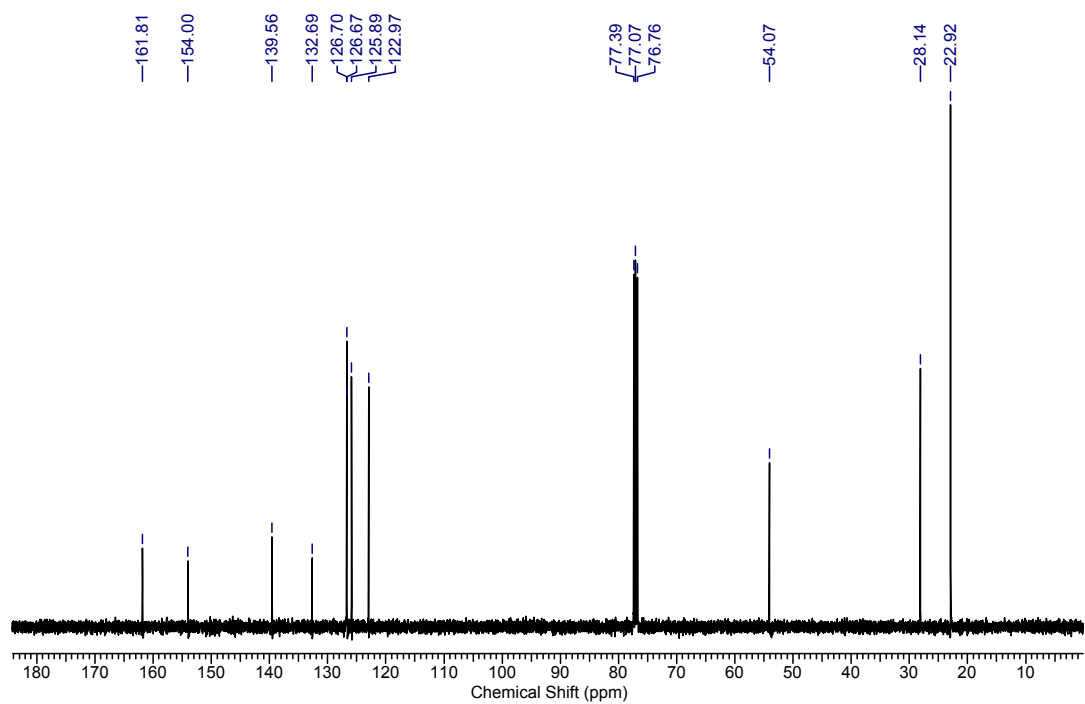
¹H NMR (400 MHz) spectrum of compound 3ca in CDCl₃



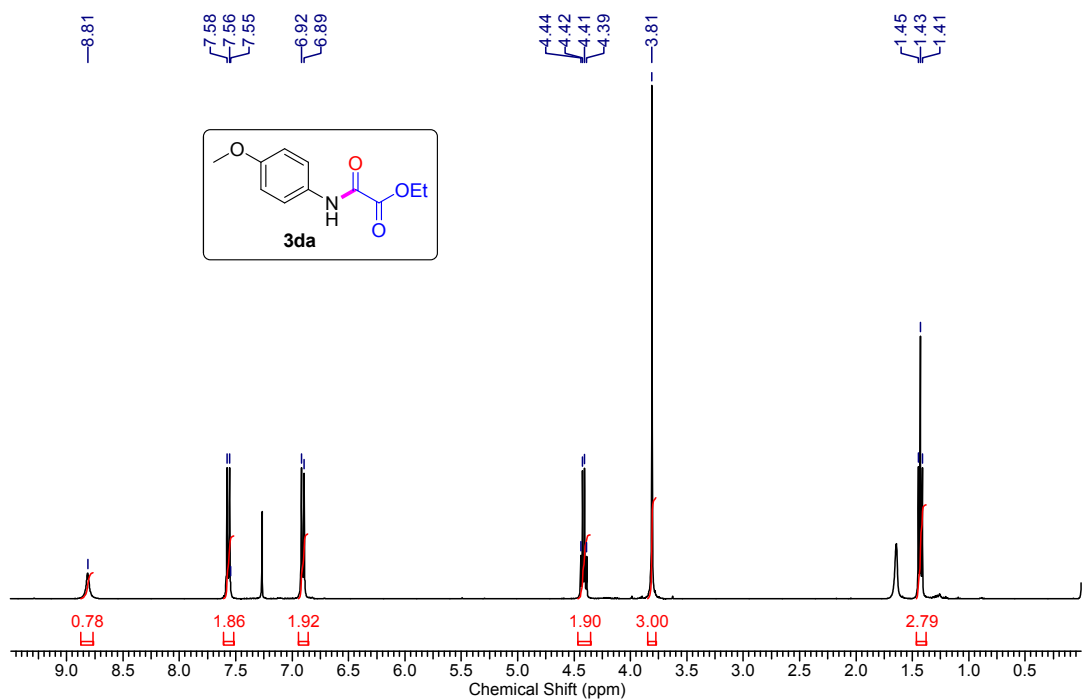
¹³C NMR (100 MHz) spectrum of compound 3ca in CDCl₃



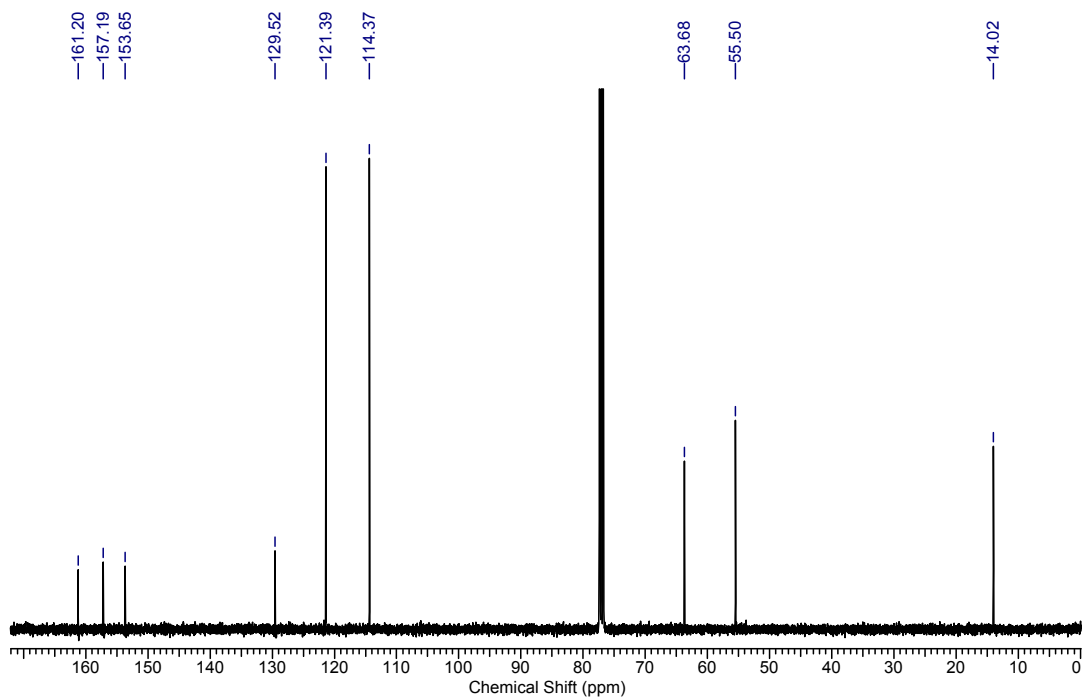
¹H NMR (400 MHz) spectrum of compound 3cb in CDCl₃



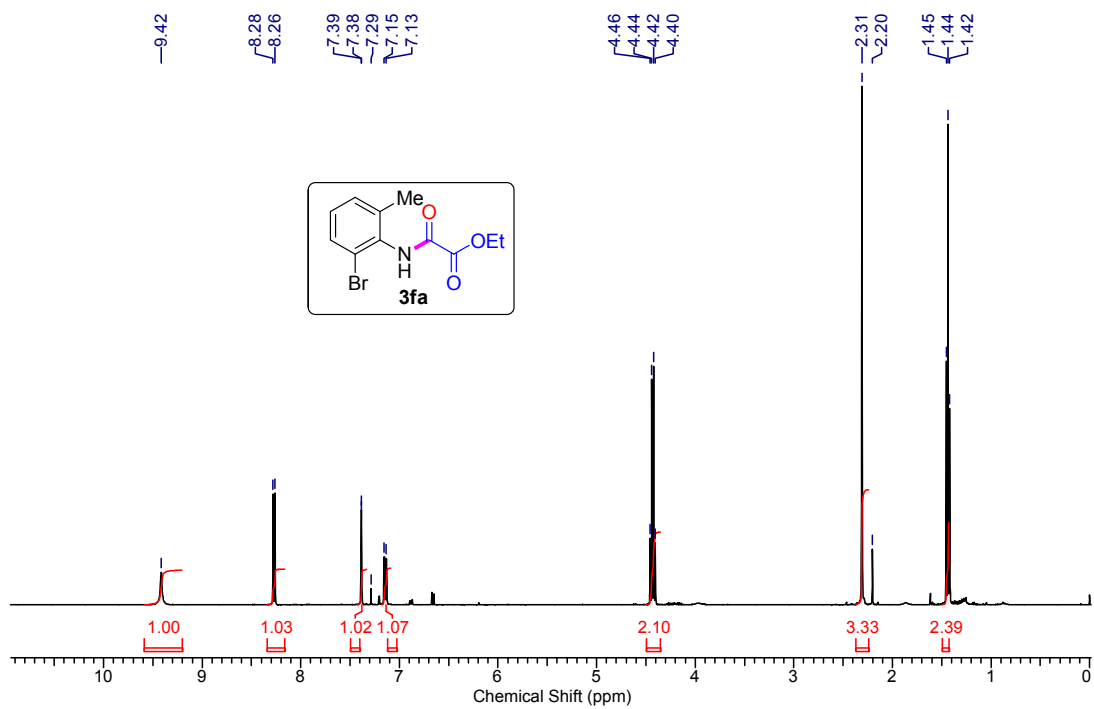
¹³C NMR (100 MHz) spectrum of compound 3cb in CDCl₃



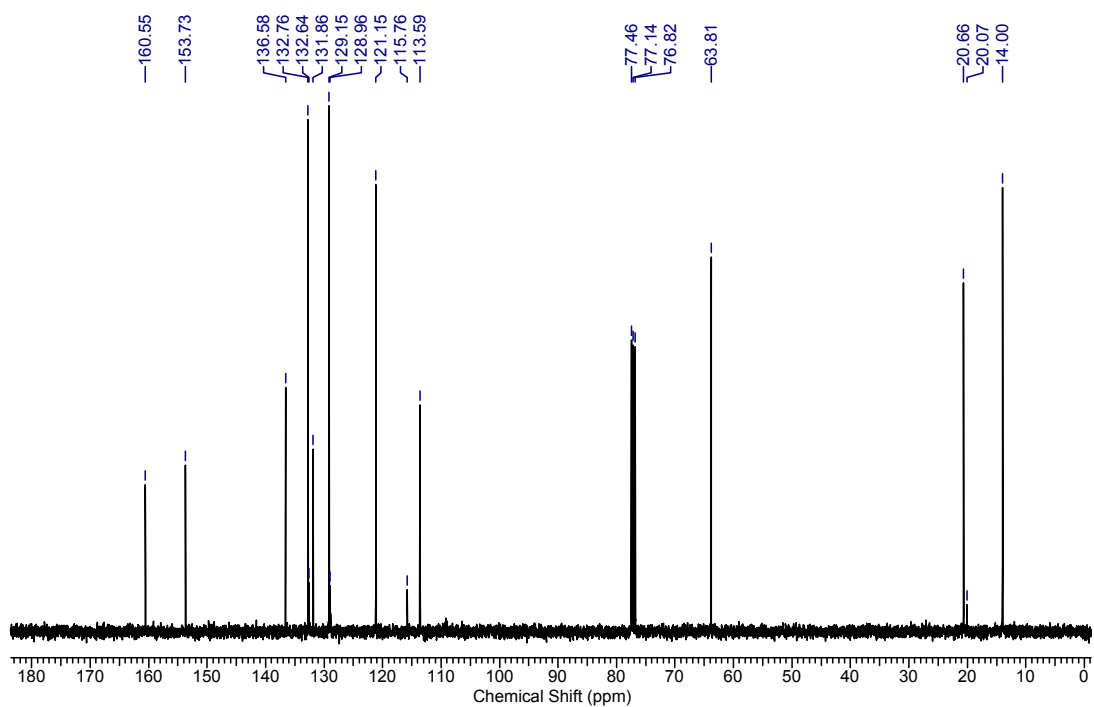
¹H NMR (400 MHz) spectrum of compound 3da in CDCl₃



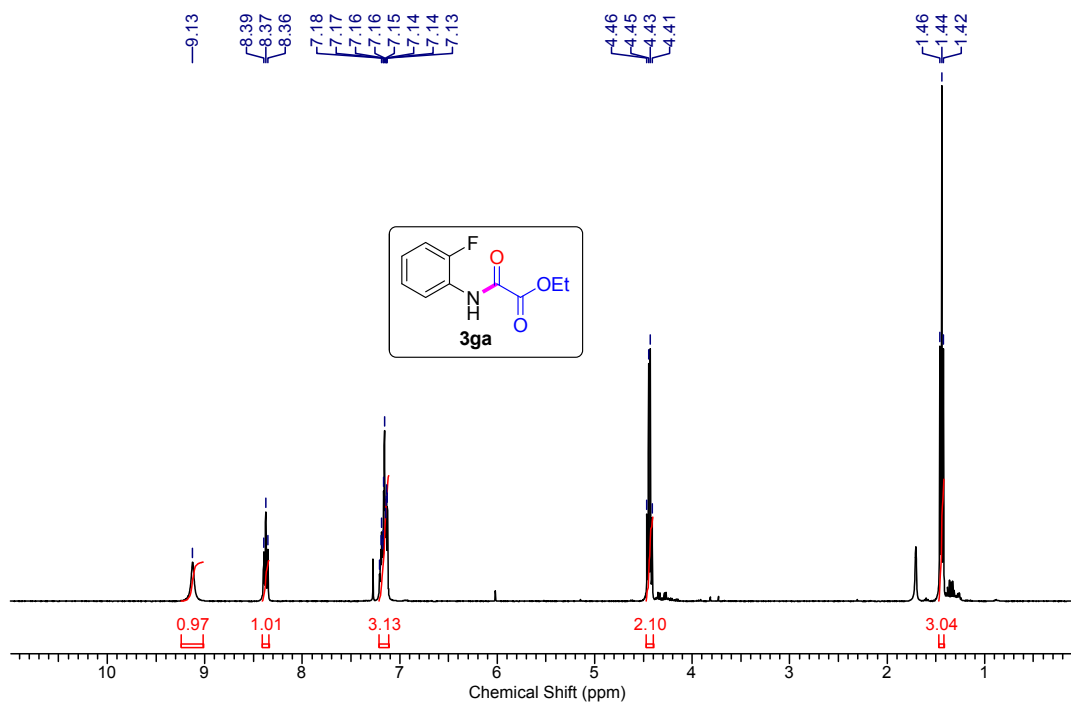
¹³C NMR (100 MHz) spectrum of compound 3da in CDCl₃



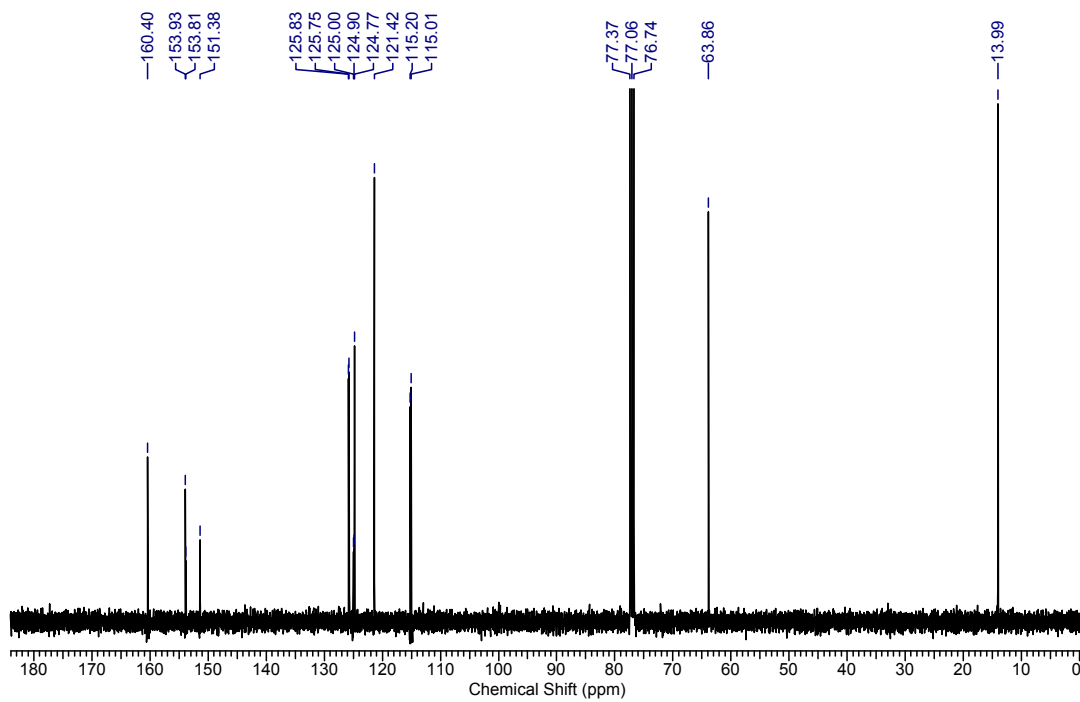
¹H NMR (400 MHz) spectrum of compound 3fa in CDCl₃



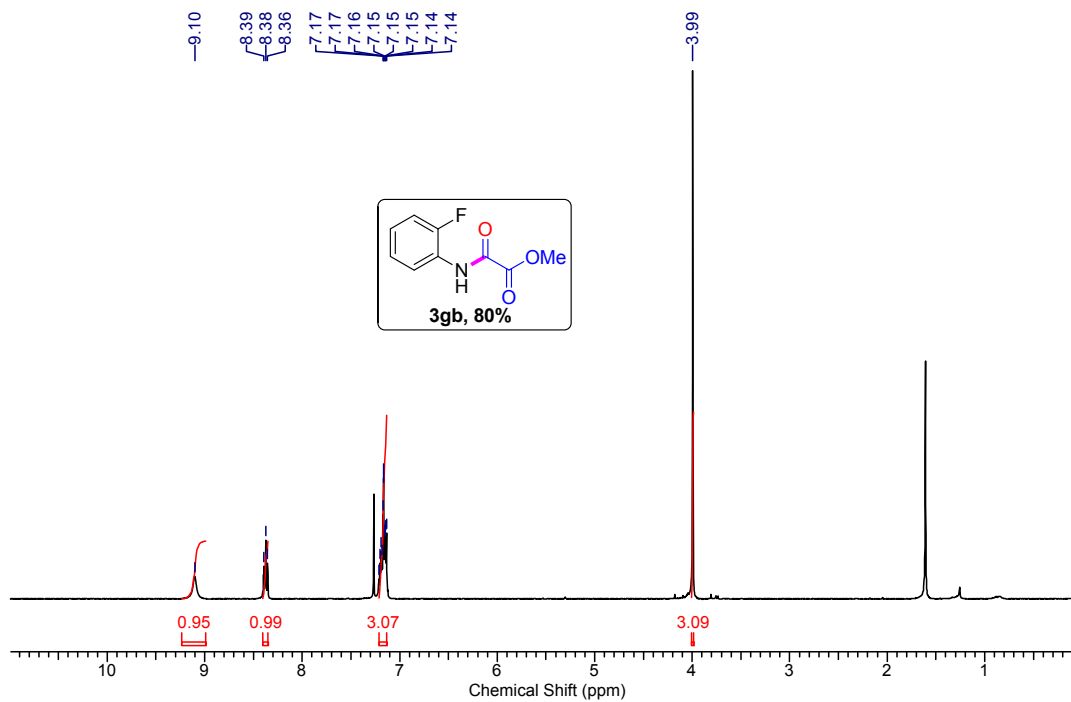
¹³C NMR (100 MHz) spectrum of compound 3fa in CDCl₃



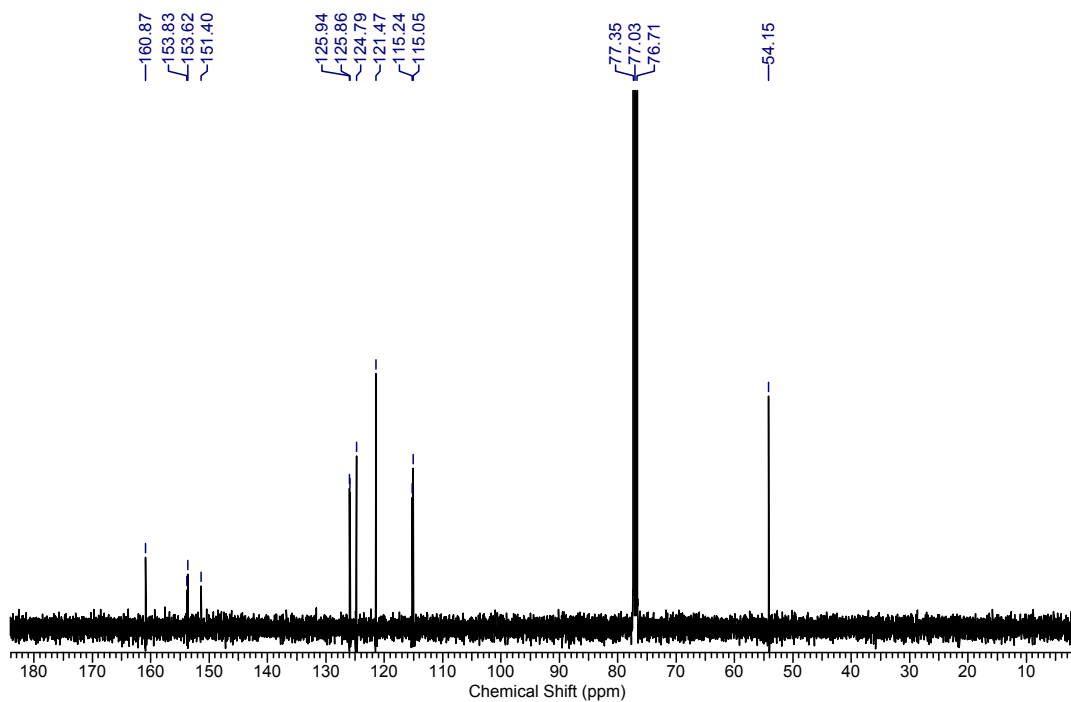
¹H NMR (400 MHz) spectrum of compound 3ga in CDCl₃



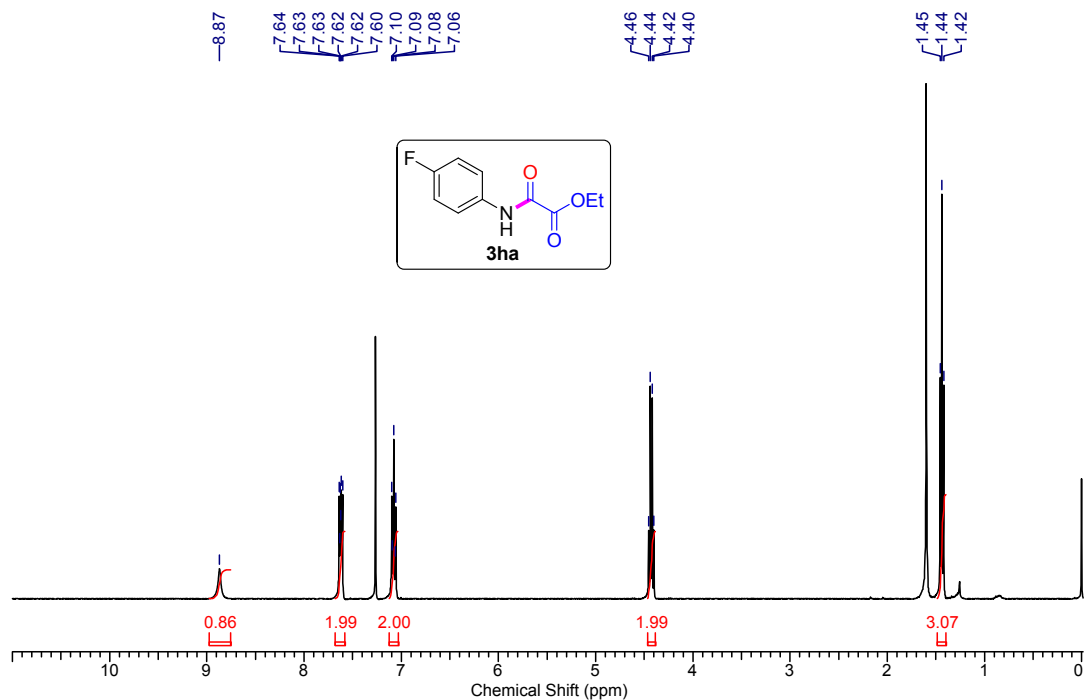
¹³C NMR (100 MHz) spectrum of compound 3ga in CDCl₃



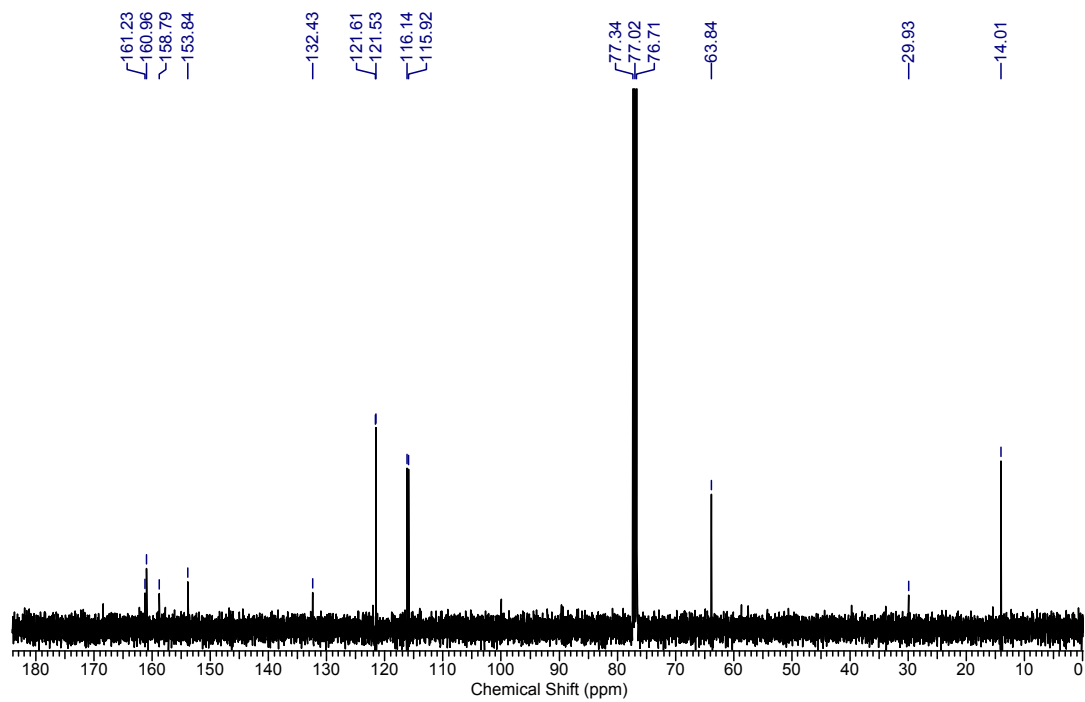
¹H NMR (400 MHz) spectrum of compound 3gb in CDCl₃



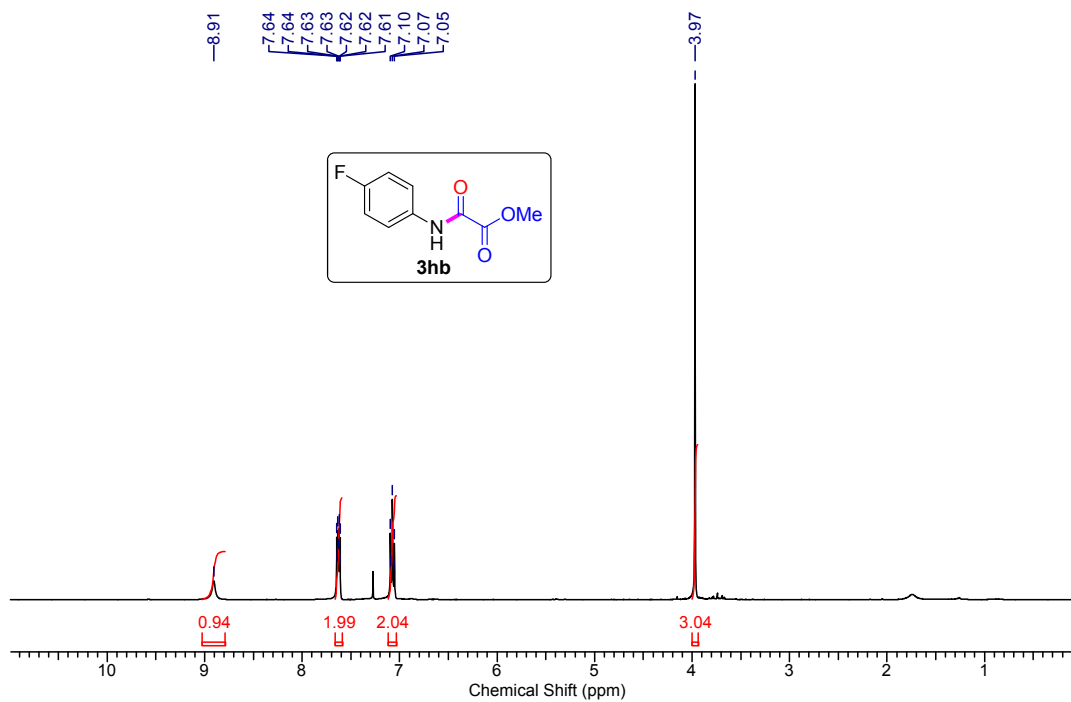
¹³C NMR (100 MHz) spectrum of compound 3gb in CDCl₃



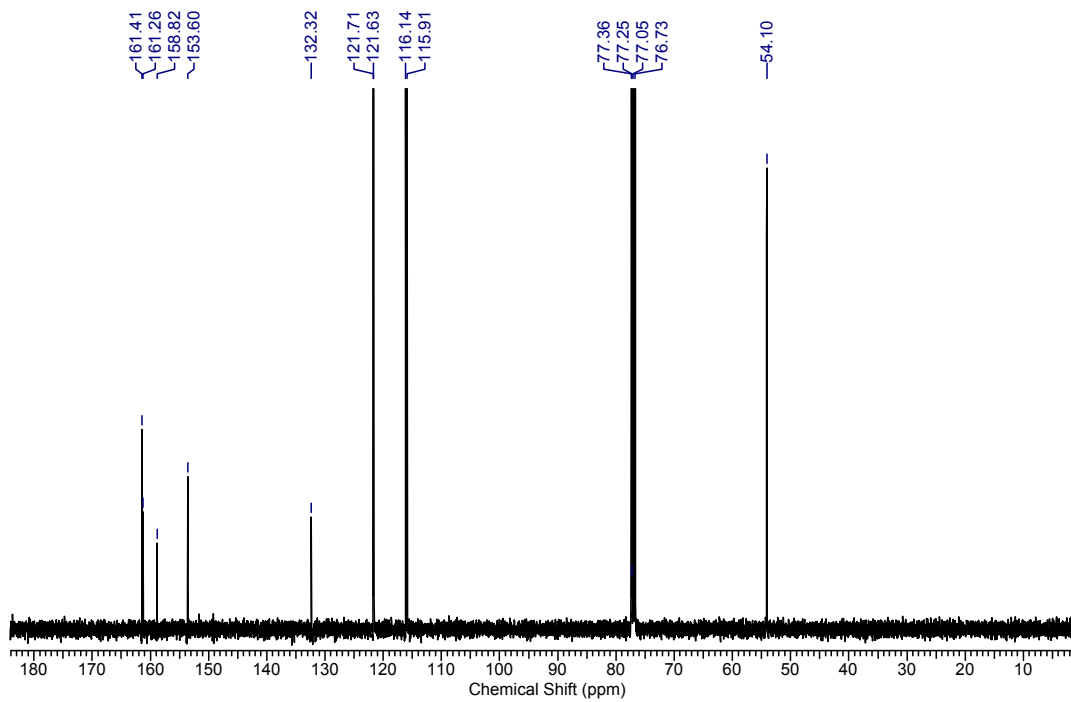
¹H NMR (400 MHz) spectrum of compound 3ha in CDCl₃



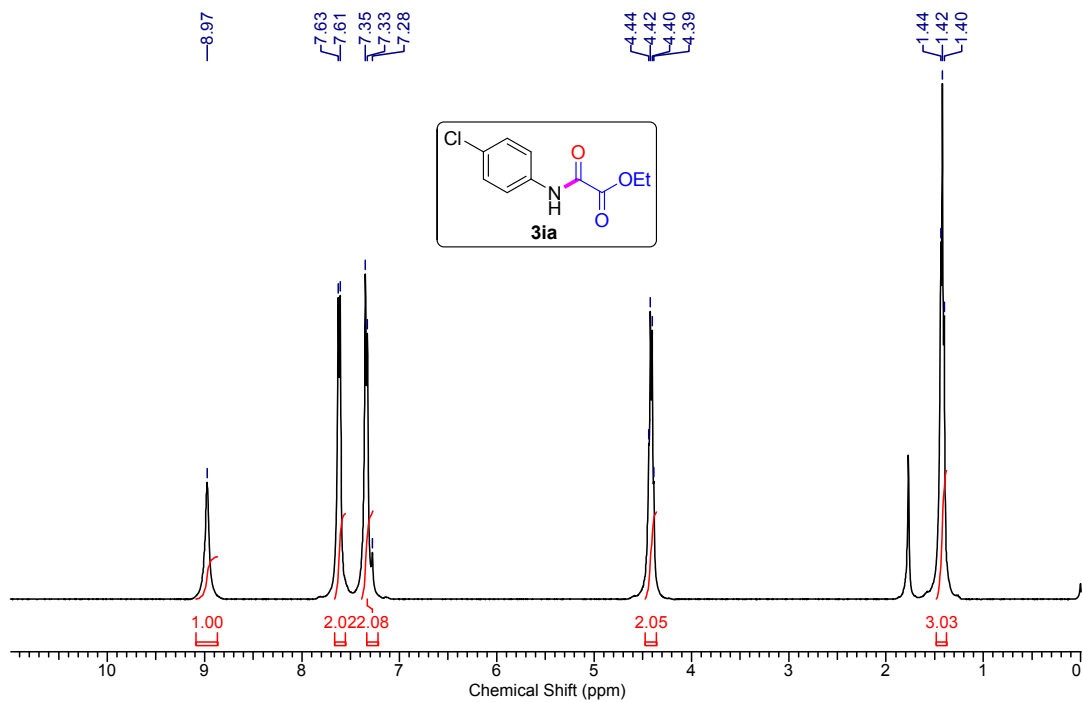
¹³C NMR (100 MHz) spectrum of compound 3ha in CDCl₃



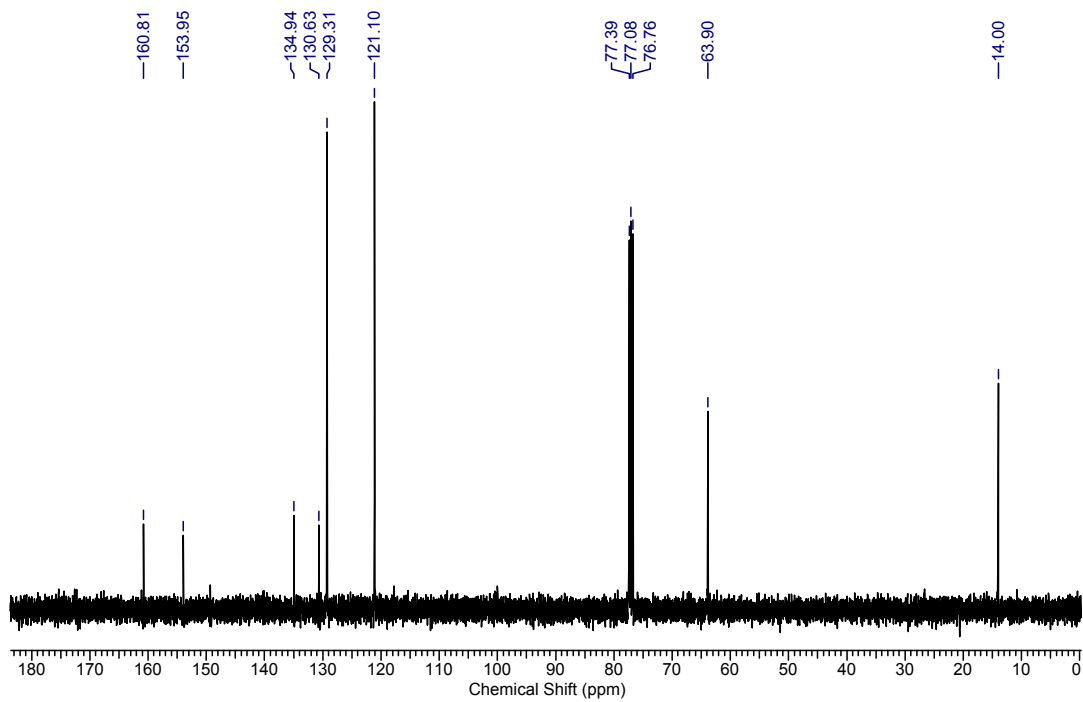
¹H NMR (400 MHz) spectrum of compound 3hb in CDCl₃



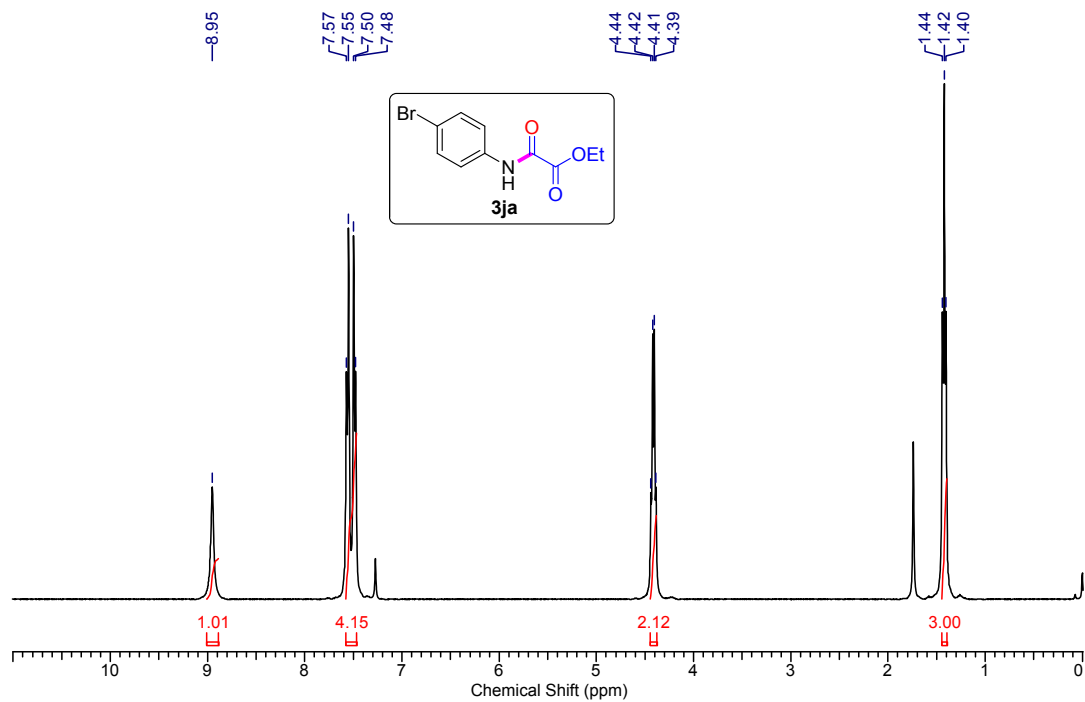
¹³C NMR (100 MHz) spectrum of compound 3hb in CDCl₃



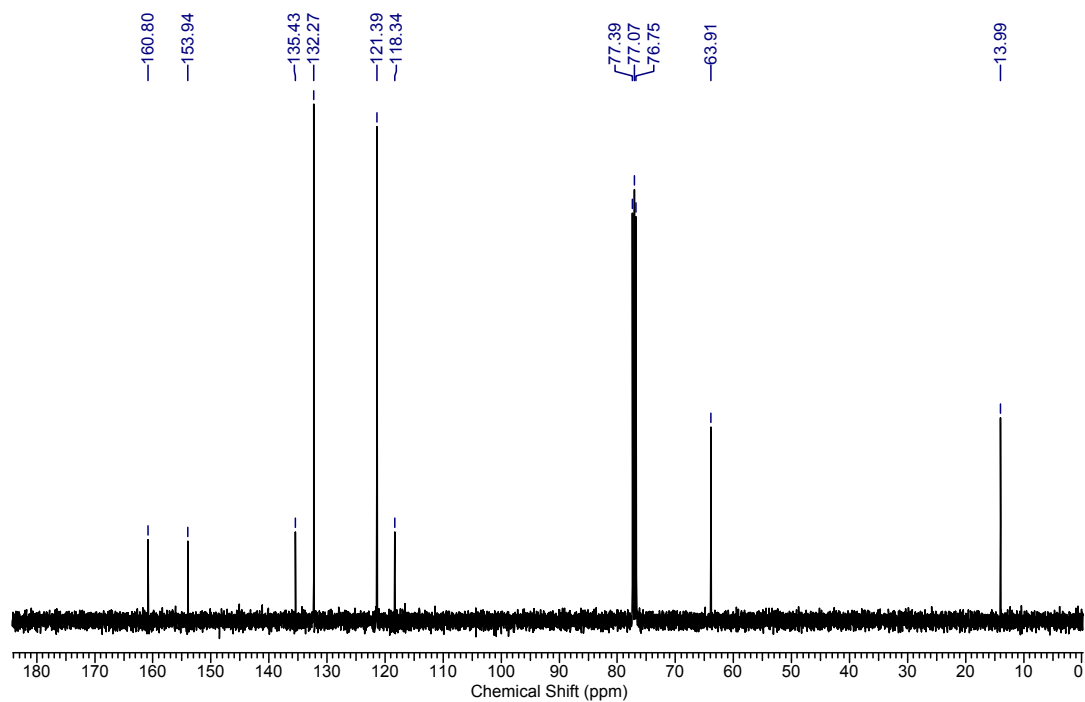
¹H NMR (400 MHz) spectrum of compound 3ia in CDCl₃



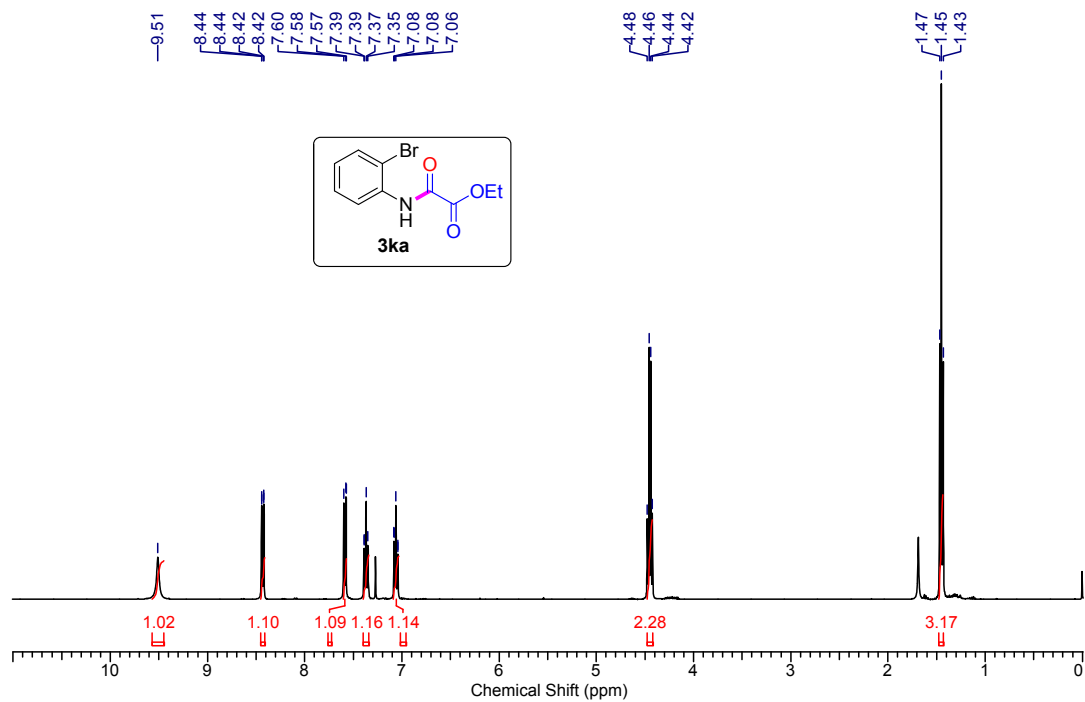
¹³C NMR (100 MHz) spectrum of compound 3ia in CDCl₃



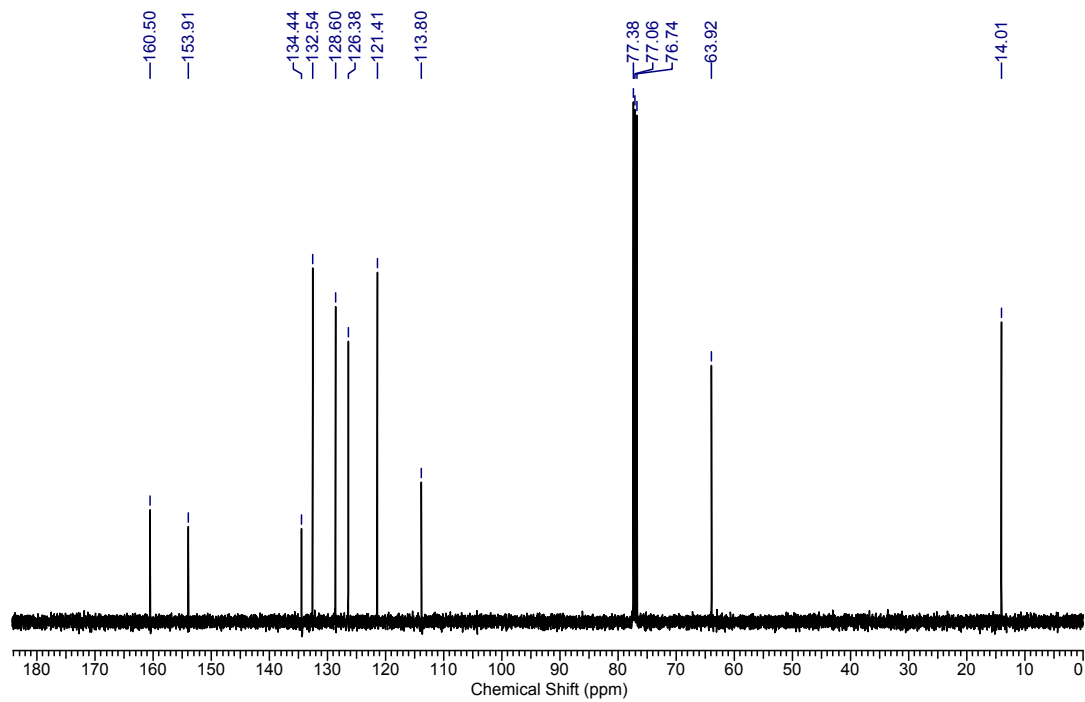
¹H NMR (400 MHz) spectrum of compound 3ja in CDCl₃



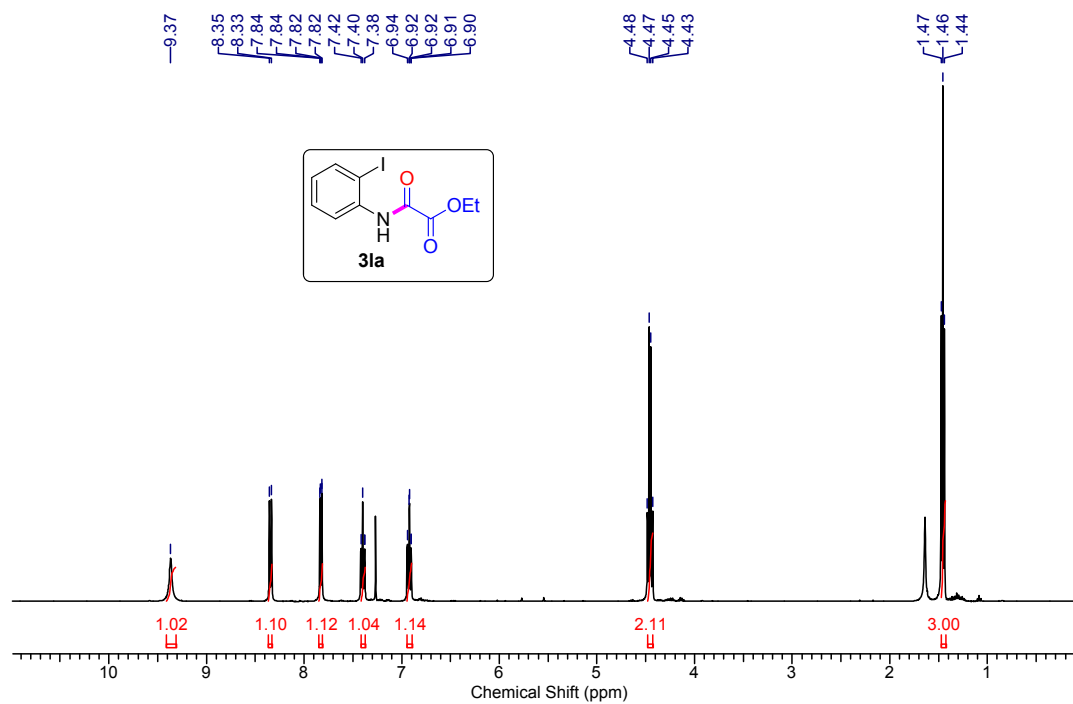
¹³C NMR (100 MHz) spectrum of compound 3ja in CDCl₃



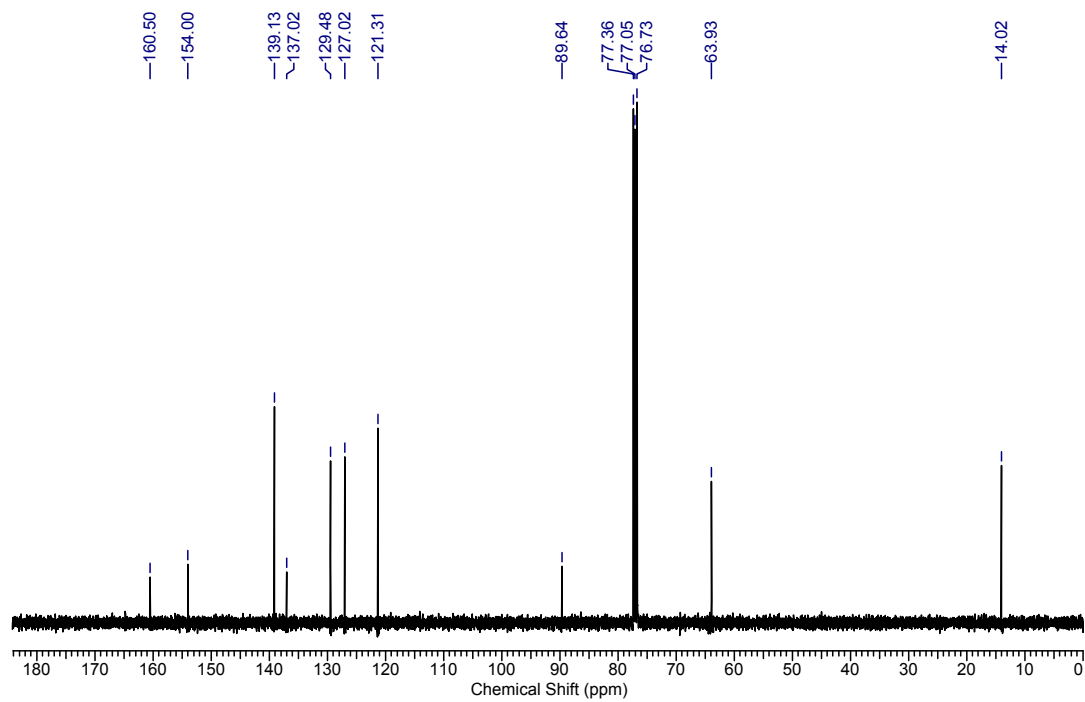
¹H NMR (400 MHz) spectrum of compound 3ka in CDCl₃



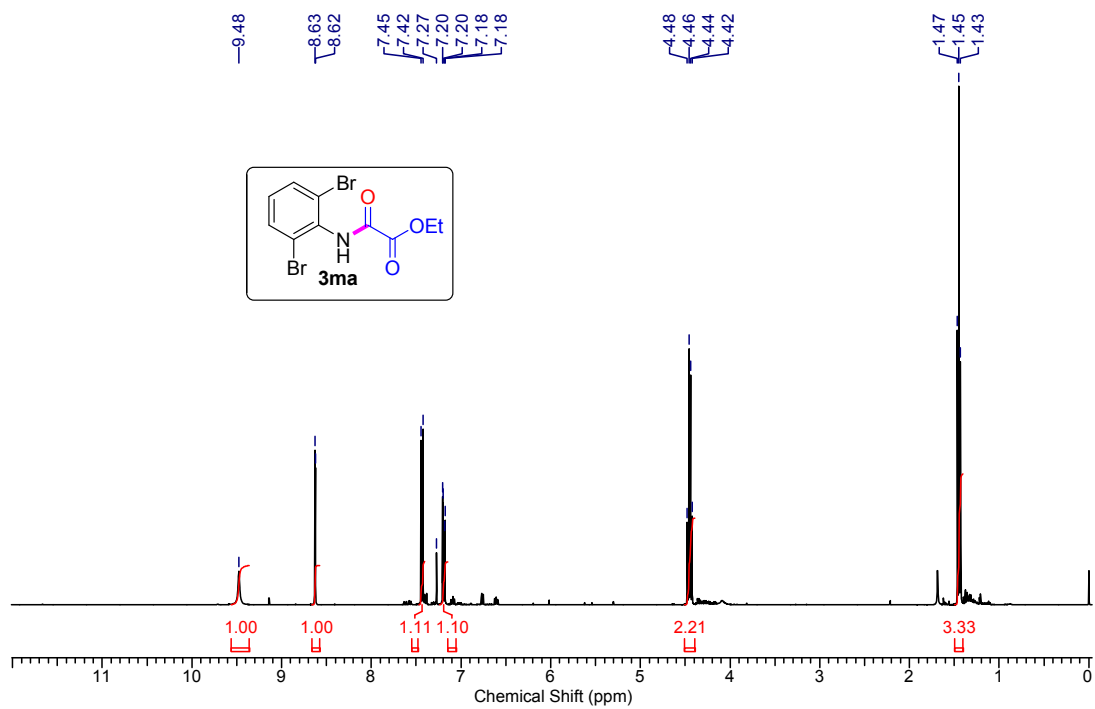
¹³C NMR (100 MHz) spectrum of compound 3ka in CDCl₃



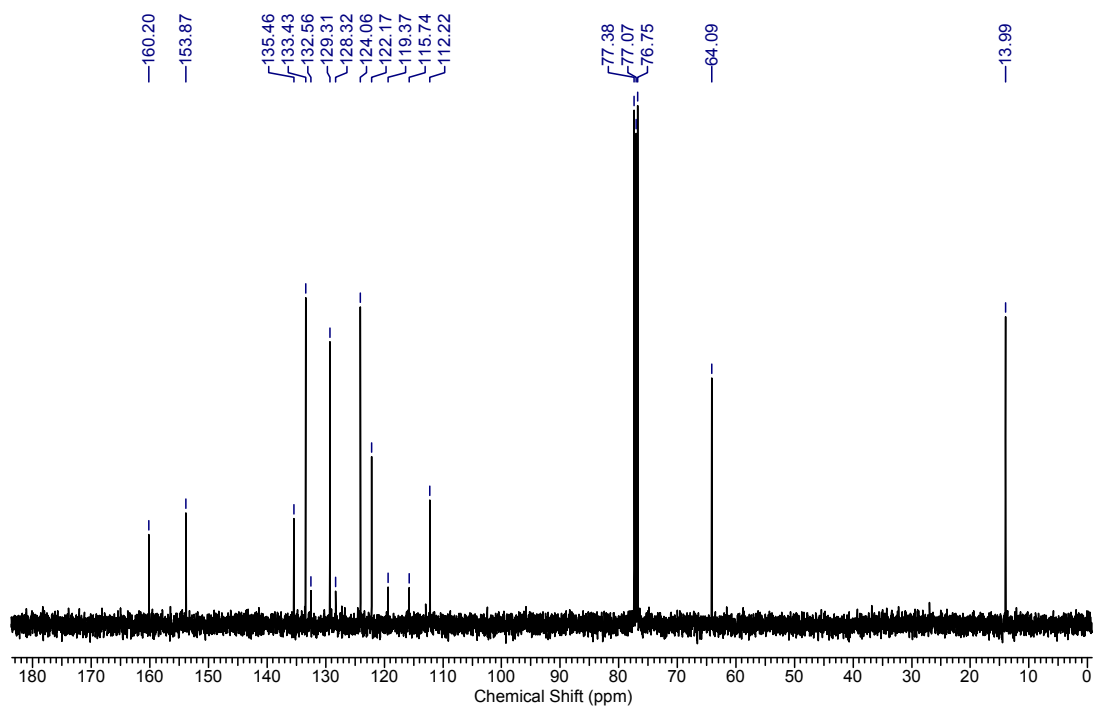
¹H NMR (400 MHz) spectrum of compound 3la in CDCl₃



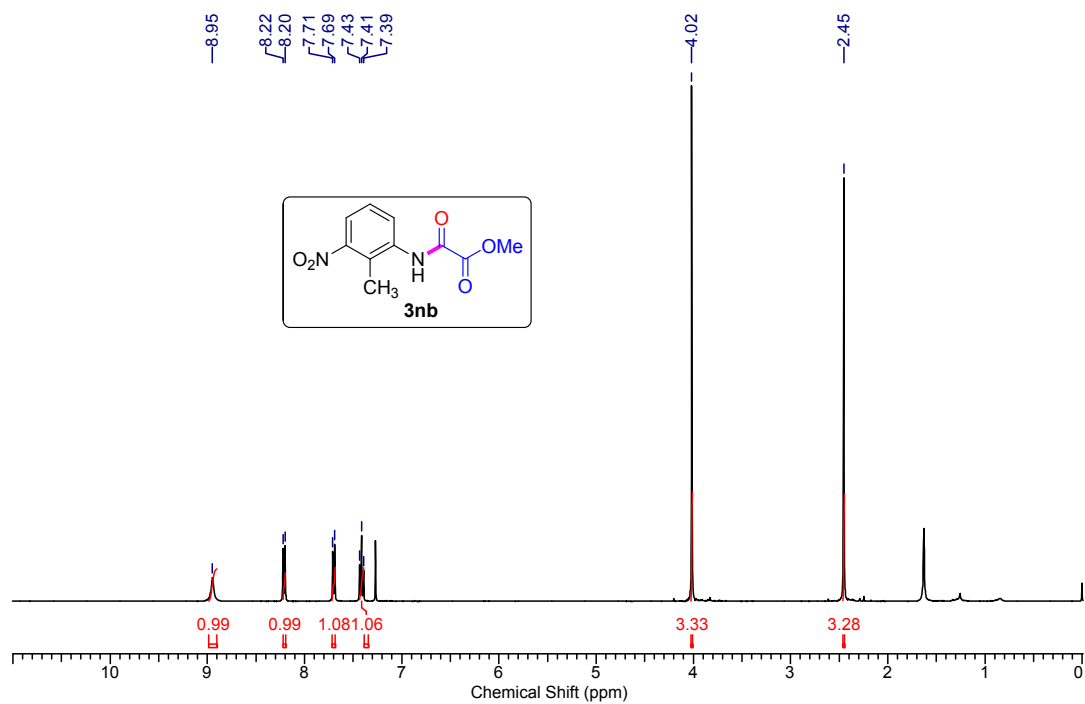
¹³C NMR (100 MHz) spectrum of compound 3la in CDCl₃



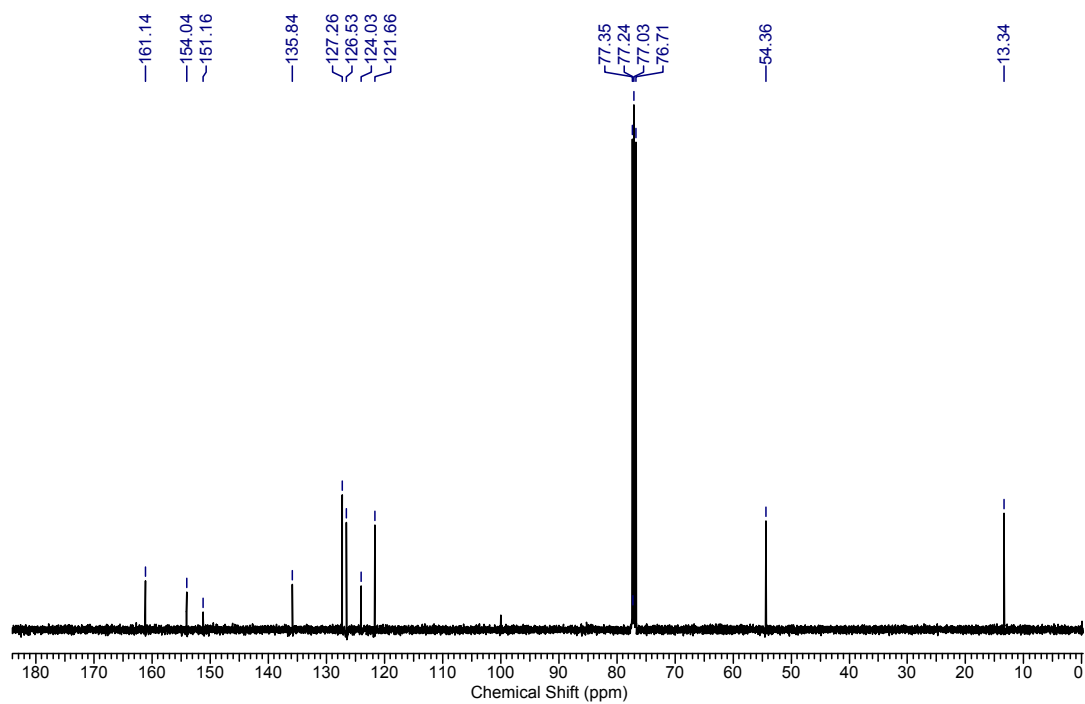
¹H NMR (400 MHz) spectrum of compound 3ma in CDCl₃



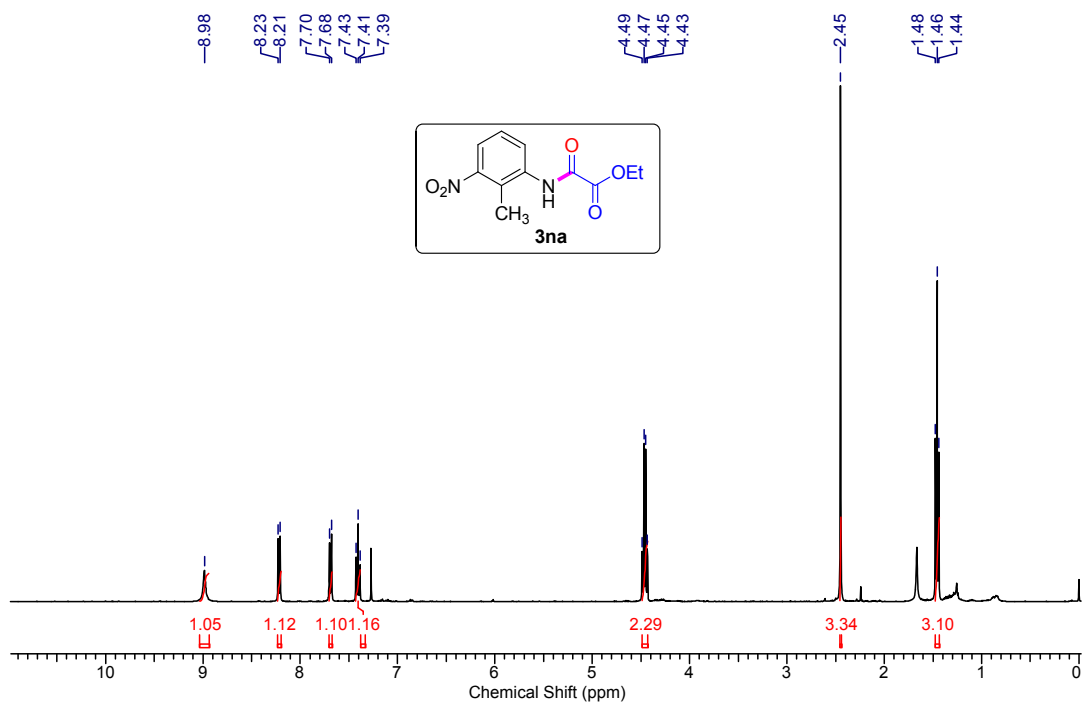
¹³C NMR (100 MHz) spectrum of compound 3ma in CDCl₃



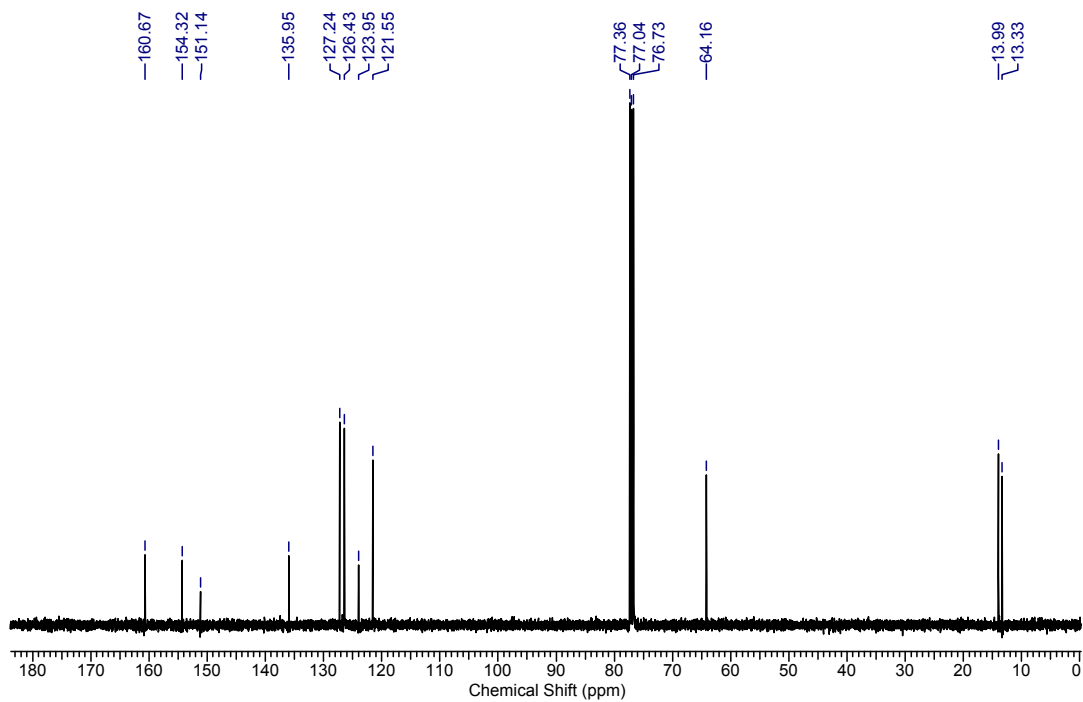
¹H NMR (400 MHz) spectrum of compound 3nb in CDCl₃



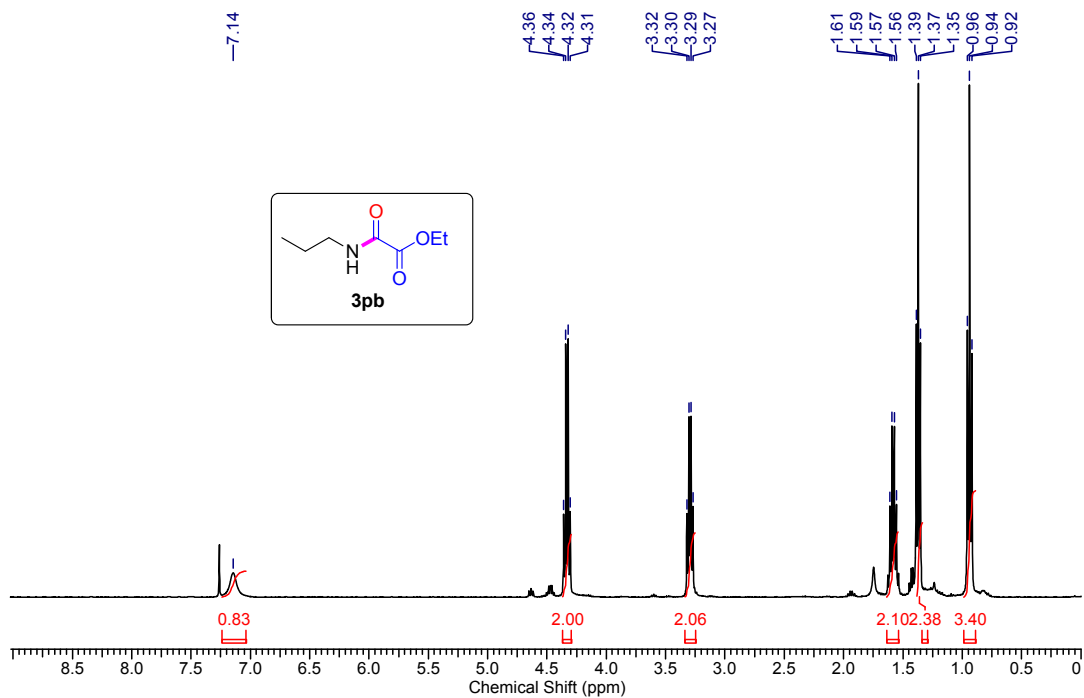
¹³C NMR (100 MHz) spectrum of compound 3nb in CDCl₃



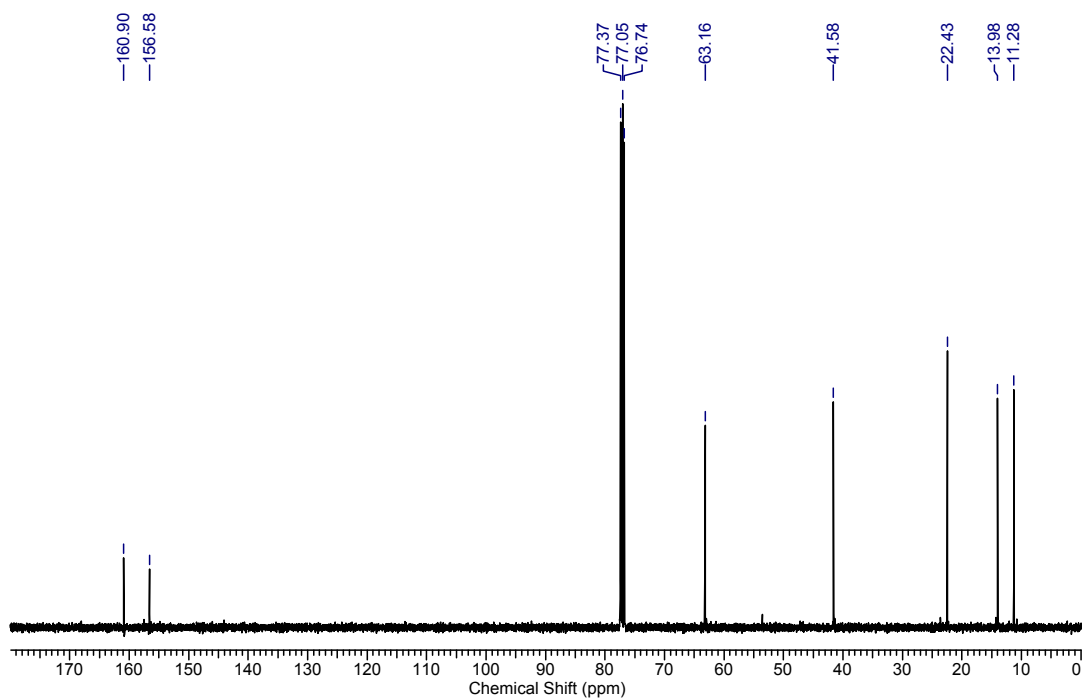
¹H NMR (400 MHz) spectrum of compound 3na in CDCl₃



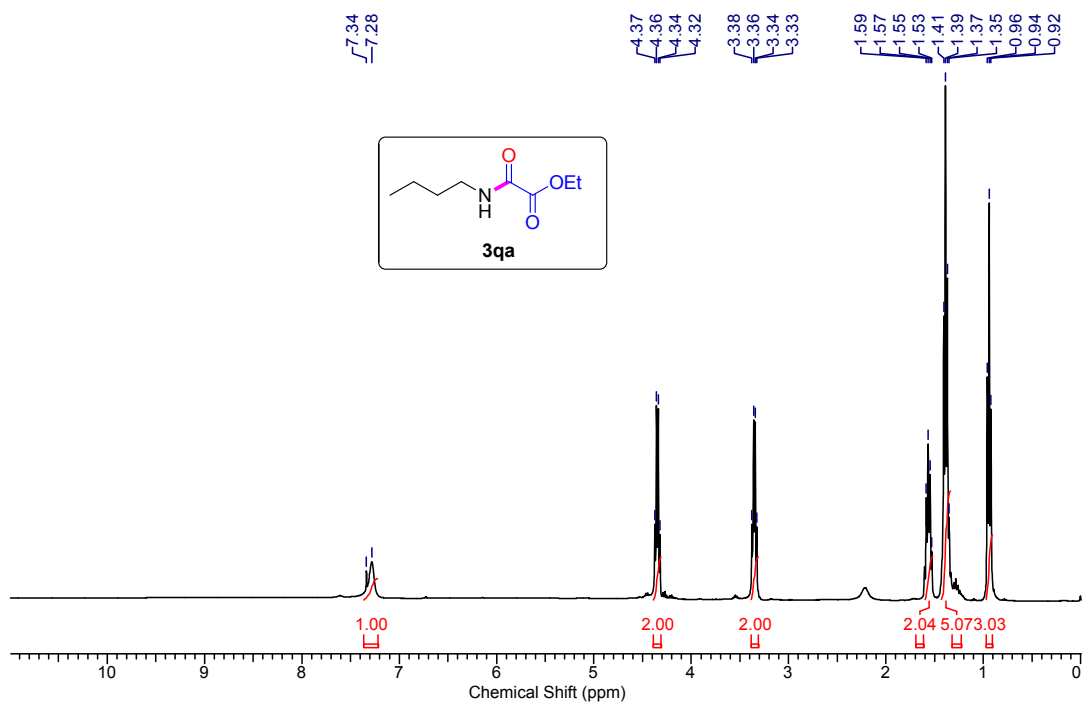
¹³C NMR (100 MHz) spectrum of compound 3na in CDCl₃



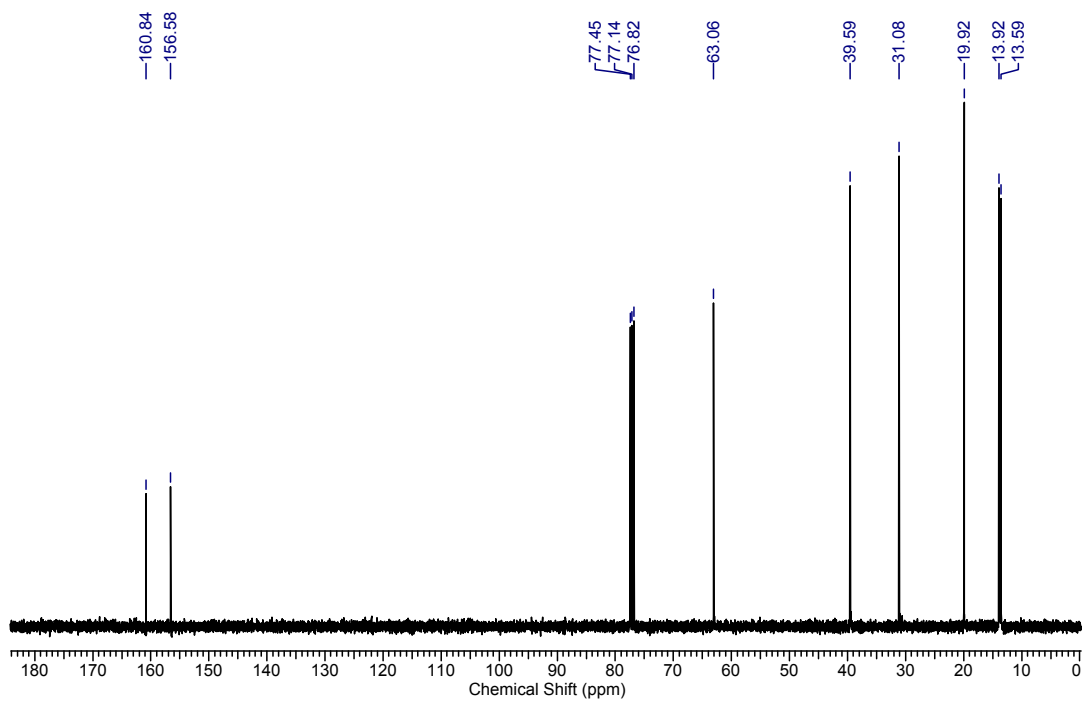
¹H NMR (400 MHz) spectrum of compound 3na in CDCl₃



¹³C NMR (100 MHz) spectrum of compound 3qa in CDCl₃



¹H NMR (400 MHz) spectrum of compound 3qa in CDCl₃



¹³C NMR (100 MHz) spectrum of compound 3qa in CDCl₃

References:

1. Dai, Q.; Yang, W.; Zhang, X. *Org. Lett.* **2005**, *7*, 5343.
2. Zhang, X.; Yang, B.; Li, G.; Shu, X.; Mungra, D.; C.; Zhu, J. *Synlett* **2012**, *23*, 622.