

Supporting Information for:

Metal Catalyzed C(sp³)-H Bond Amination of 2-Alkyl Azaarenes with Diethyl Azodicarboxylate

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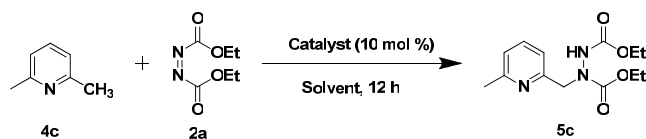
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General information:

Melting points were recorded with a micro melting point apparatus and uncorrected. NMR spectra were recorded with a 400 MHz spectrometer for ¹H NMR, 100 MHz for ¹³C NMR. Chemical shifts δ are given in ppm relative to tetramethylsilane as internal standard, residual CHCl₃ for ¹H NMR or CDCl₃ in ¹³C NMR spectroscopy. Multiplicities are reported as follows: singlet (s), doublet (d), doublet of doublets (dd), triplet (t), quartet (q), multiplet (m). High resolution mass spectra were taken with a 3000 mass spectrometer, using Waters Q-ToFMS/MS system. For column chromatography silica gel (200-300 mesh) was used as the stationary phase. All reactions were monitored by thin layer chromatography (TLC). DMSO and tetrahydrofuran used in reactions were reagent grade and distilled from calcium hydride and sodium. All reagents and solvents were purchased from commercial sources and purified commonly before used. All known quinolones were prepared according to literature procedures.^[1] All pyridines and azodicarboxylate are commercially available compounds.

Table S1. The optimization of Cu(OTf)₂ catalyzed C(sp³)-H amination of 2,6-dimethylpyridine



Entry	Catalyst	Ligand	Solvent	T/°C	Yield/ % ^b
1	Pd(OAc) ₂	1,10-phenanthroline	DMSO	90	trace
2	Pd(OAc) ₂	1,10-phenanthroline	DMSO	110	27
3	Pd(OAc) ₂	1,10-phenanthroline	DMSO	120	25
4	Cu(OTf) ₂	1,10-phenanthroline	DMSO	110	35
5	Cu(OTf)₂	1,10-phenanthroline	THF	110	58
6	Cu(OAc) ₂	1,10-phenanthroline	THF	110	41
7	CuI	1,10-phenanthroline	THF	110	38
8	Cu(OTf) ₂	1,10-phenanthroline	toluene	110	0
9	Cu(OTf) ₂	1,10-phenanthroline	<i>i</i> -PrOH	110	0
10	Cu(OTf) ₂	1,10-phenanthroline	dioxane	110	28
11	Cu(OTf) ₂	none	THF	110	7

^a Unless otherwise stated, all reactions were carried out with **4c** (0.75 mmol), **2a** (0.3 mmol), catalyst (10 mol %), ligand (10 mol %), solvent (1.5 mL), 12 h. ^b Isolated yield.

General procedure

Palladium-catalyzed direct C(sp³)-H amination of 2-alkylquinolines

Under nitrogen, Pd(OAc)₂ (3.4 mg, 5 mol %), 1,10-phenanthroline (2.7 mg, 5 mol %), 2-methylquinoline **1a** (0.75 mmol), diethyl azodicarboxylate **2a** (0.3 mmol), and dry DMSO (1.0 mL) were added to a Schlenk tube. The mixture was stirred at 90 °C and monitored by TLC. After completion of the reaction, the reaction was extracted with ethyl acetate, then the organic phase was evaporated under reduced pressure and the residue was purified by column chromatography on silica gel (eluent: petroleum ether/ethyl acetate) to give the desired product.

Palladium-catalyzed direct C(sp³)-H diamination of 2-alkylquinolines

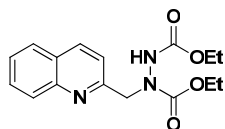
Under nitrogen, Pd(OAc)₂ (6.8 mg, 10 mol %), 1,10-phenanthroline (5.4 mg, 10 mol %), 2-methylquinoline **1a** (0.3 mmol), diethyl azodicarboxylate **2a** (0.9 mmol), dry DMSO (1.0 mL) were added to a Schlenk tube. The mixture was kept stirring at 110 °C for 24 h and monitored by TLC. After completion of the reaction, the reaction was extracted with ethyl acetate, then, organic phase was evaporated under reduced pressure and the residue purified by column chromatography on silica gel (eluent: petroleum ether/ethyl acetate) to give the desired product.

Copper-catalyzed direct C(sp³)-H amination of 2-alkylpyridines

Cu(OTf)₂ (10.9 mg, 10 mol %), 1,10-phenanthroline (5.4 mg, 10 mol %), 2,6-dimethylpyridine **4c** (88 μL, 0.75 mmol) and diethyl azodicarboxylate **2a** (48 μL, 0.3 mmol) were mixed in the screw cap vial and then dry THF (0.8 mL) was added. The mixture was stirred at 110 °C under nitrogen in a closed reaction vessel containing a stir bar. The reaction was monitored by TLC. After completion of the reaction, the solvent was evaporated under reduced pressure and the residue was purified by column chromatography on silica gel (eluent: petroleum ether/ethyl acetate) to give the desired product.

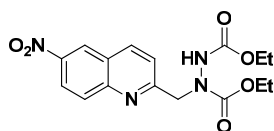
Characterization of compounds

Diethyl 1-(quinolin-2-ylmethyl)hydrazine-1,2-dicarboxylate (3a):



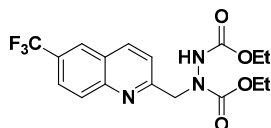
White solid; m.p. 77-79 °C; ^1H NMR (400 MHz, CDCl_3): δ 8.11 (d, J = 8.4 Hz, 1H), 8.02 (d, J = 8.4 Hz, 1H), 7.79 (d, J = 8.4 Hz, 1H), 7.70 (t, J = 7.2 Hz, 1H), 7.52 (t, J = 7.2 Hz, 1H), 7.35-7.33 (m, 1H), 5.02 (s, 2H), 4.23-4.16 (m, 4H), 1.31-1.19 (m, 6H); ^{13}C NMR (400 MHz, CDCl_3): δ 157.3, 156.8, 145.8, 135.8, 132.1, 130.7, 130.5, 127.9, 126.2, 62.8, 62.1, 61.9, 14.4; HRMS: calcd for $\text{C}_{16}\text{H}_{20}\text{N}_3\text{O}_4$ $[\text{M}+\text{H}]^+$ 318.1448, found 318.1452.

Diethyl 1-((6-nitroquinolin-2-yl)methyl)hydrazine-1,2-dicarboxylate (3b):



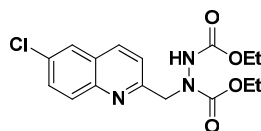
Weak yellow solid; m.p. 123-125 °C; ^1H NMR (400 MHz, CDCl_3): δ 8.76 (s, 1H), 8.45 (d, J = 8.8 Hz, 1H), 8.31 (d, J = 7.6 Hz, 1H), 8.14 (d, J = 9.2 Hz, 1H), 7.56 (s, 1H), 5.06 (s, 2H), 4.25-4.16 (m, 4H), 1.31-1.16 (m, 6H); ^{13}C NMR (400 MHz, CDCl_3): δ 156.7, 149.6, 145.4, 138.4, 130.9, 126.1, 124.3, 123.1, 63.0, 62.1, 14.4; HRMS: calcd for $\text{C}_{16}\text{H}_{18}\text{N}_4\text{NaO}_6$ $[\text{M}+\text{Na}]^+$ 385.1119, found 385.1124.

Diethyl 1-((6-(trifluoromethyl)quinolin-2-yl)methyl)hydrazine-1,2-dicarboxylate (3c):



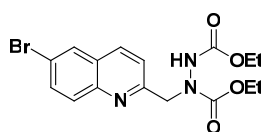
Colorless oil; ^1H NMR (400 MHz, CDCl_3): δ 8.18 (s, 1H), 8.09 (s, 2H), 7.84 (d, J = 7.6 Hz, 1H), 7.47 (d, J = 0.8 Hz, 1H), 5.04 (s, 2H), 4.24-4.16 (m, 4H), 1.30-1.16 (m, 6H); ^{13}C NMR (400 MHz, CDCl_3): δ 156.9, 136.7, 136.7, 129.6, 129.5, 129.0, 128.8, 127.5, 127.2, 126.4, 62.7, 61.8, 14.4; HRMS: calcd for $\text{C}_{17}\text{H}_{18}\text{F}_3\text{N}_3\text{NaO}_4$ $[\text{M}+\text{Na}]^+$ 408.1142, found 408.1138.

Diethyl 1-((6-chloroquinolin-2-yl)methyl)hydrazine-1,2-dicarboxylate (3d):



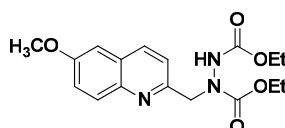
White solid; m.p. 72-74 °C; ^1H NMR (400 MHz, CDCl_3): δ 7.96 (t, J = 16.0 Hz, 2H); 7.74 (s, 1H), 7.59 (s, 1H), 7.37 (s, 1H), 4.98 (s, 2H), 4.22-4.15(m, 4H), 1.29-1.16 (m, 6H); ^{13}C NMR (400 MHz, CDCl_3): δ 156.9, 147.4, 136.7, 129.5, 127.5, 127.2, 126.3, 62.7, 62.0, 61.8, 14.5, 14.4; HRMS: calcd for $\text{C}_{16}\text{H}_{19}\text{ClN}_3\text{O}_4[\text{M}+\text{H}]^+$ 352.1059, found 352.1057.

Diethyl 1-((6-bromoquinolin-2-yl)methyl)hydrazine-1,2-dicarboxylate (3e):



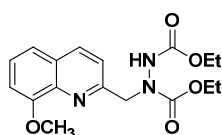
Colorless oil; ^1H NMR (400 MHz, CDCl_3): δ 7.98-7.84 (m, 3H), 7.73 (s, 1H), 7.36 (d, J = 4.8Hz, 1H), 4.98 (s, 2H), 4.23-4.16 (m, 4H), 1.23 (t, J = 7.2 Hz, 6H); ^{13}C NMR (400 MHz, CDCl_3): δ 157.3, 157.3, 156.8, 146.1, 135.7, 133.0, 129.6, 128.4, 120.2, 62.8, 61.9, 14.5, 14.4; HRMS: calcd for $\text{C}_{16}\text{H}_{18}\text{BrN}_3\text{NaO}_4 [\text{M}+\text{Na}]^+$ 418.0373, found 418.0381.

Diethyl 1-((6-methoxyquinolin-2-yl)methyl)hydrazine-1,2-dicarboxylate (3f):



Colorless oil; ^1H NMR (400 MHz, CDCl_3): δ 7.98 (s, 1H), 7.89 (d, J = 8.0Hz, 1H), 7.32 (d, J = 9.6Hz, 2H), 7.03 (s, 1H), 4.96 (s, 2H), 4.22-4.16 (m, 4H), 3.91 (s, 3H), 1.30-1.19 (m, 6H); ^{13}C NMR (400 MHz, CDCl_3): δ 157.7, 156.9, 143.6, 135.6, 130.5, 128.3, 122.2, 105.1, 62.7, 61.9, 55.5, 14.5, 14.4; HRMS: calcd for $\text{C}_{17}\text{H}_{21}\text{N}_3\text{NaO}_5 [\text{M}+\text{Na}]^+$ 370.1373, found 370.1370.

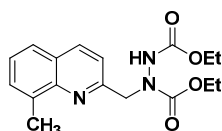
Diethyl 1-((8-methoxyquinolin-2-yl)methyl)hydrazine-1,2-dicarboxylate (3g):



White solid; m.p. 128-130 °C; ^1H NMR (400 MHz, CDCl_3): δ 8.05 (d, J = 1.2Hz, 1H), 7.43 (t, J =

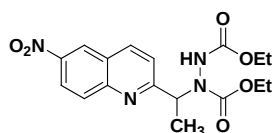
8.0 Hz, 2H), 7.34 (d, $J = 8.0$ Hz, 1H), 7.03 (d, $J = 6.8$ Hz, 1H), 5.04 (s, 2H), 4.25-4.14 (m, 4H), 4.05 (s, 3H), 1.23 (t, $J = 7.2$ Hz, 6H); ^{13}C NMR (400 MHz, CDCl_3): δ 156.7, 156.1, 155.0, 139.4, 136.7, 128.4, 126.5, 119.5, 108.1, 62.8, 61.9, 56.1, 14.5, 14.4; HRMS: calcd for $\text{C}_{17}\text{H}_{21}\text{N}_3\text{NaO}_5$ $[\text{M}+\text{Na}]^+$ 370.1373, found 370.1378.

Diethyl 1-((8-methylquinolin-2-yl)methyl)hydrazine-1,2-dicarboxylate (3h):



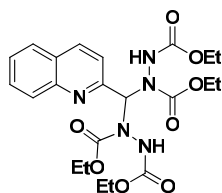
Colorless oil; ^1H NMR (400 MHz, CDCl_3): δ 8.10 (d, $J = 8.4$ Hz, 1H), 7.65 (d, $J = 8.4$ Hz, 1H), 7.56 (d, $J = 6.8$ Hz, 1H), 7.41 (t, $J = 7.6$ Hz, 1H), 7.28 (d, $J = 7.6$ Hz, 1H), 5.02 (s, 2H), 4.26-4.17 (m, 4H), 2.75 (s, 3H), 1.34-1.14 (m, 6H); ^{13}C NMR (400 MHz, CDCl_3): δ 157.5, 157.3, 156.1, 146.5, 137.2, 129.9, 127.2, 126.1, 125.6, 62.7, 61.8, 17.9, 14.4; HRMS: calcd for $\text{C}_{17}\text{H}_{21}\text{N}_3\text{NaO}_4$ $[\text{M}+\text{Na}]^+$ 354.1424, found 354.1418.

Diethyl 1-(1-(6-nitroquinolin-2-yl)ethyl)hydrazine-1,2-dicarboxylate (3i):



Weak yellow solid; m.p. 124-126 °C; ^1H NMR (400 MHz, CDCl_3): δ 8.74 (d, $J = 2.4$ Hz, 1H), 8.43-8.40 (m, 1H), 7.29 (d, $J = 8.8$ Hz, 1H), 8.12 (d, $J = 9.2$ Hz, 1H), 7.56-7.48 (m, 1H), 5.68 (t, $J = 2.4$ Hz, 1H), 4.19-4.15 (m, 4H), 1.67 (d, $J = 6.4$ Hz, 3H), 1.27-1.21 (m, 6H); ^{13}C NMR (400 MHz, CDCl_3): δ 165.7, 156.7, 149.4, 145.4, 138.4, 126.0, 124.2, 123.0, 62.7, 61.9, 17.2, 14.5, 14.4; HRMS: calcd for $\text{C}_{17}\text{H}_{20}\text{N}_4\text{NaO}_6$ $[\text{M}+\text{Na}]^+$ 399.1275, found 399.1283.

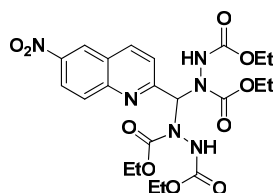
Tetraethyl 1,1'-(quinolin-2-ylmethylene)bis(hydrazine-1,2-dicarboxylate) (3aa):



White solid; m.p. 122-124 °C; ^1H NMR (400 MHz, CDCl_3): δ 8.16 (d, $J = 8.4$ Hz, 1H), 8.01 (d, $J =$

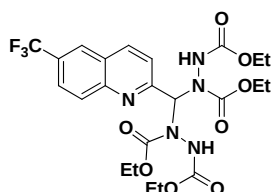
8.0Hz, 1H), 7.81 (d, J = 8.0Hz, 2H), 7.71-7.68 (m, 1H), 7.56-7.52 (m, 1H), 6.89-6.87 (m, 1H), 4.27-4.11 (m, 8H), 1.27-1.16 (m, 12H); ^{13}C NMR (400 MHz, CDCl_3): δ 161.2, 156.8, 149.6, 145.3, 138.5, 130.9, 126.1, 124.4, 123.1, 123.1, 63.0, 62.0, 14.4; HRMS: calcd for $\text{C}_{22}\text{H}_{30}\text{N}_5\text{O}_8$ $[\text{M}+\text{H}]^+$ 492.2089, found 492.90.

Tetraethyl 1,1'-((6-nitroquinolin-2-yl)methylene)bis(hydrazine-1,2-dicarboxylate) (3bb):



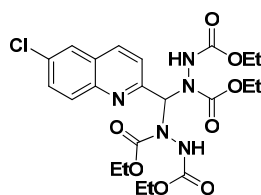
White solid; m.p. 136-138 °C; ^1H NMR (400 MHz, CDCl_3): δ 8.07 (d, J = 7.6Hz, 1H), 7.95 (d, J = 8.8Hz, 1H), 7.79 (s, 1H), 7.65 (d, J = 9.2Hz, 2H), 6.88-6.83 (m, 1H), 4.30-4.06 (m, 8H), 1.27 (s, 12H); ^{13}C NMR (400 MHz, CDCl_3): δ 156.4, 155.7, 145.2, 135.9, 131.0, 130.4, 128.2, 126.3, 62.9, 62.1, 14.4; HRMS: calcd for $\text{C}_{22}\text{H}_{29}\text{N}_6\text{O}_{10}$ $[\text{M}+\text{H}]^+$ 537.1940, found 537.1944.

Tetraethyl 1,1'-((6-(trifluoromethyl)quinolin-2-yl)methylene)bis(hydrazine-1,2-dicarboxylate) (3cc):



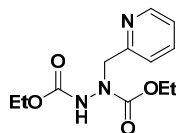
White solid; m.p. 103-105 °C; ^1H NMR (400 MHz, CDCl_3): δ 8.24 (d, J = 8.0 Hz, 1H), 8.13-8.00 (m, 2H), 7.87 (s, 1H), 7.69-7.68 (m, 1H), 6.91-6.83 (m, 1H), 4.30-4.20 (m, 8H), 1.32-1.25 (m, 12H); ^{13}C NMR (400 MHz, CDCl_3): δ 156.8, 156.4, 155.7, 147.9, 137.5, 130.6, 126.6, 125.5, 125.5, 125.3, 122.6, 121.6, 121.6, 121.5, 63.0, 63.0, 62.2, 14.4; HRMS: calcd for $\text{C}_{23}\text{H}_{29}\text{F}_3\text{N}_5\text{O}_8$ $[\text{M}+\text{H}]^+$ 560.1963, found 560.1970.

Tetraethyl 1,1'-((6-chloroquinolin-2-yl)methylene)bis(hydrazine-1,2-dicarboxylate) (3dd):



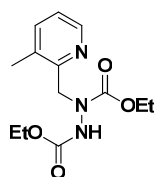
White solid; m.p. 140-142 °C; ^1H NMR (400 MHz, CDCl_3): δ 8.07 (d, J = 8.4Hz, 1H), 7.95 (d, J = 8.8Hz, 1H), 7.79 (s, 1H), 7.62 (d, J = 8.0Hz, 2H), 6.89-6.84 (m, 1H), 4.32-4.19 (m, 8H), 1.35-1.25 (m, 12H); ^{13}C NMR (400 MHz, CDCl_3): δ 156.4, 155.7, 145.2, 135.9, 131.0, 130.3, 128.2, 126.3, 62.9, 62.1, 14.4; HRMS: calcd for $\text{C}_{22}\text{H}_{28}\text{ClN}_5\text{NaO}_8$ $[\text{M}+\text{Na}]^+$ 548.1519, found 548.1525.

Diethyl 1-(pyridin-2-ylmethyl) hydrazine-1,2-dicarboxylate (5a):



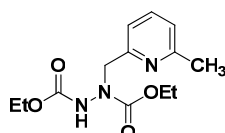
White solid; m.p. 128-129 °C; ^1H NMR (400 MHz, CDCl_3): δ 8.52 (d, J = 4.0Hz, 1H), 7.65 (t, J = 7.6Hz, 1H), 7.18 (t, J = 6.8Hz, 2H), 4.83 (s, 2H), 4.22-4.15 (m, 4H), 1.25 (t, J = 7.6 Hz, 6H); ^{13}C NMR (400 MHz, CDCl_3): δ 156.7, 156.6, 156.5, 149.3, 122.4, 62.7, 61.8, 58.2, 14.4; HRMS: calcd for $\text{C}_{12}\text{H}_{17}\text{N}_3\text{NaO}_4$ $[\text{M}+\text{Na}]^+$ 290.1111, found 290.1111.

Diethyl 1-((3-methylpyridin-2-yl) methyl) hydrazine-1,2-dicarboxylate (5b):



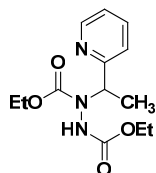
Colorless oil; ^1H NMR (400 MHz, CDCl_3): δ 8.34 (d, J = 4.4Hz, 1H), 7.42 (d, J = 7.6Hz, 1H), 7.11-7.08-4.15 (m, 1H), 4.78 (s, 2H), 4.21-4.16 (m, 4H), 2.26 (s, 3H), 1.28-1.23 (m, 6H); ^{13}C NMR (400 MHz, CDCl_3): δ 158.0, 158.0, 156.7, 136.8, 121.8, 62.5, 61.7, 24.3, 14.4; HRMS: calcd for $\text{C}_{13}\text{H}_{19}\text{N}_3\text{NaO}_4$ $[\text{M}+\text{Na}]^+$ 304.1268, found 304.1262.

Diethyl 1-((6-methylpyridin-2-yl)methyl)hydrazine-1,2-dicarboxylate (5c):



White solid; m.p. 75-77 °C; ¹H NMR (400 MHz, CDCl₃): δ 7.51 (t, *J* = 7.6Hz, 1H), 7.01 (d, *J* = 7.6Hz, 2H), 4.77 (s, 2H), 4.17 (t, *J* = 6.8Hz, 4H), 2.47 (s, 3H), 1.23 (t, *J* = 7.6Hz, 6H); ¹³C NMR (400 MHz, CDCl₃): δ 158.0, 158.0, 156.7, 136.8, 121.8, 62.5, 61.7, 24.3, 14.4; HRMS: calcd for C₁₃H₁₉N₃NaO₄ [M+Na]⁺ 304.1268, found 304.1269.

Diethyl 1-(1-(pyridin-2-yl)ethyl)hydrazine-1,2-dicarboxylate (5d):



Colorless oil; ¹H NMR (400 MHz, CDCl₃): δ 8.46 (d, *J* = 4.0Hz, 1H), 7.62 (t, *J* = 7.6Hz, 1H), 7.18 (t, *J* = 6.8Hz, 2H), 4.83 (s, 2H), 4.22-4.15 (m, 4H), 1.25 (t, *J* = 7.6 Hz, 6H); ¹³C NMR (400 MHz, CDCl₃): δ 161.3, 156.8, 156.4, 149.0, 136.6, 122.2, 62.4, 61.7, 17.6, 14.5, 14.4; HRMS: calcd for C₁₃H₁₉N₃NaO₄ [M+Na]⁺ 304.1268, found 304.1261.

Diethyl 1-(1-(pyridin-2-yl)propyl)hydrazine-1,2-dicarboxylate (5e):



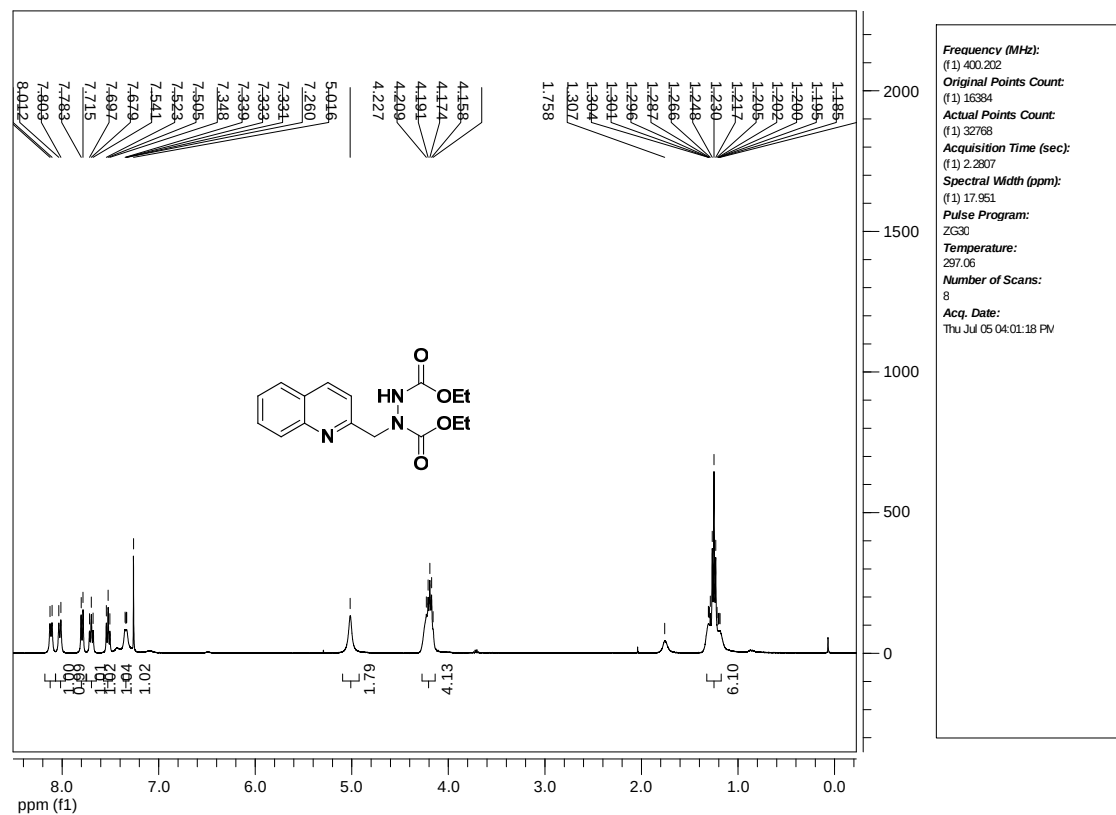
Colorless oil; ¹H NMR (400 MHz, CDCl₃): δ 8.48 (d, *J* = 3.6Hz, 1H), 7.62 (t, *J* = 7.6Hz, 1H), 7.16 (t, *J* = 10.0Hz, 2H), 5.20-5.04 (m, 1H), 4.20-4.13 (m, 4H), 1.99-1.89 (m, 2H), 1.24 (t, *J* = 12.8Hz, 6H), 1.02 (t, *J* = 7.2Hz, 3H); ¹³C NMR (400 MHz, CDCl₃): δ 160.5, 156.8, 157.1, 149.1, 136.4, 122.3, 62.4, 61.6, 24.9, 14.5, 14.4, 11.2; HRMS: calcd for C₁₃H₁₉N₃NaO₄ [M+H]⁺ 296.1605, found 296.1611.

References:

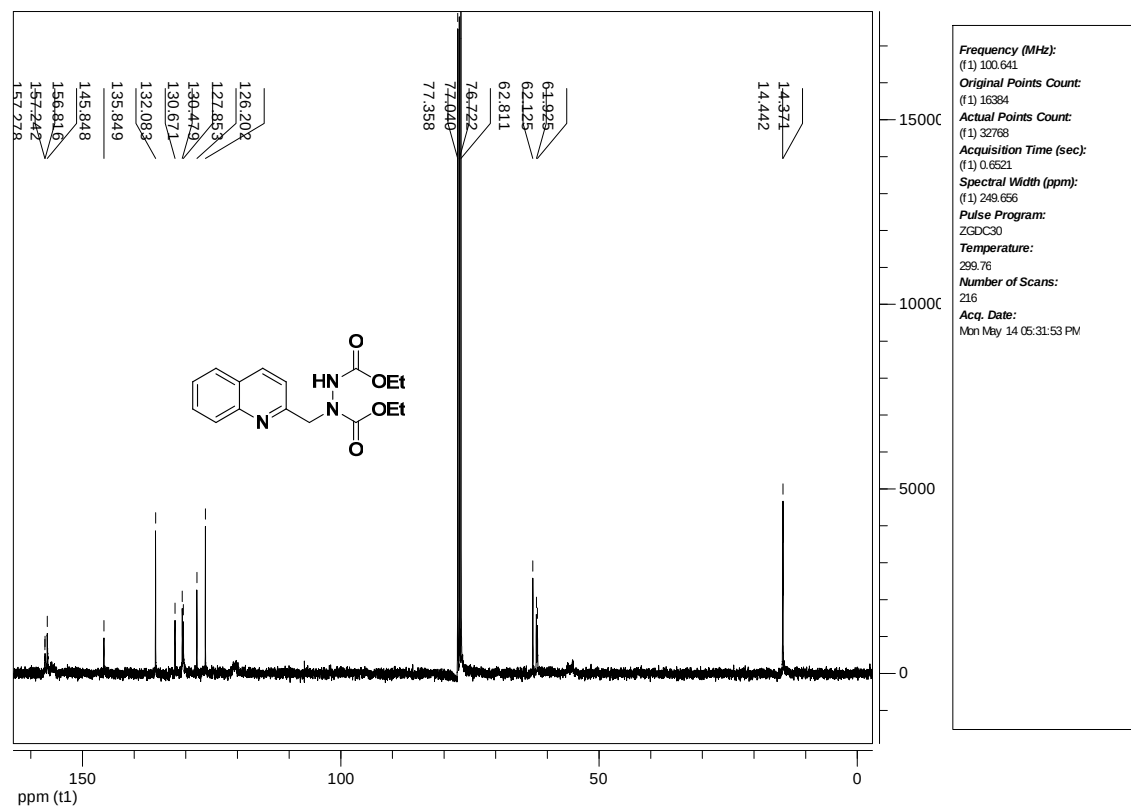
1. M. Matsugi, F. Tabusa and J. Minamikawa, *Tetrahedron Lett.*, 2000, **41**, 8523–8525.

Copies of ^1H NMR and ^{13}C NMR spectra

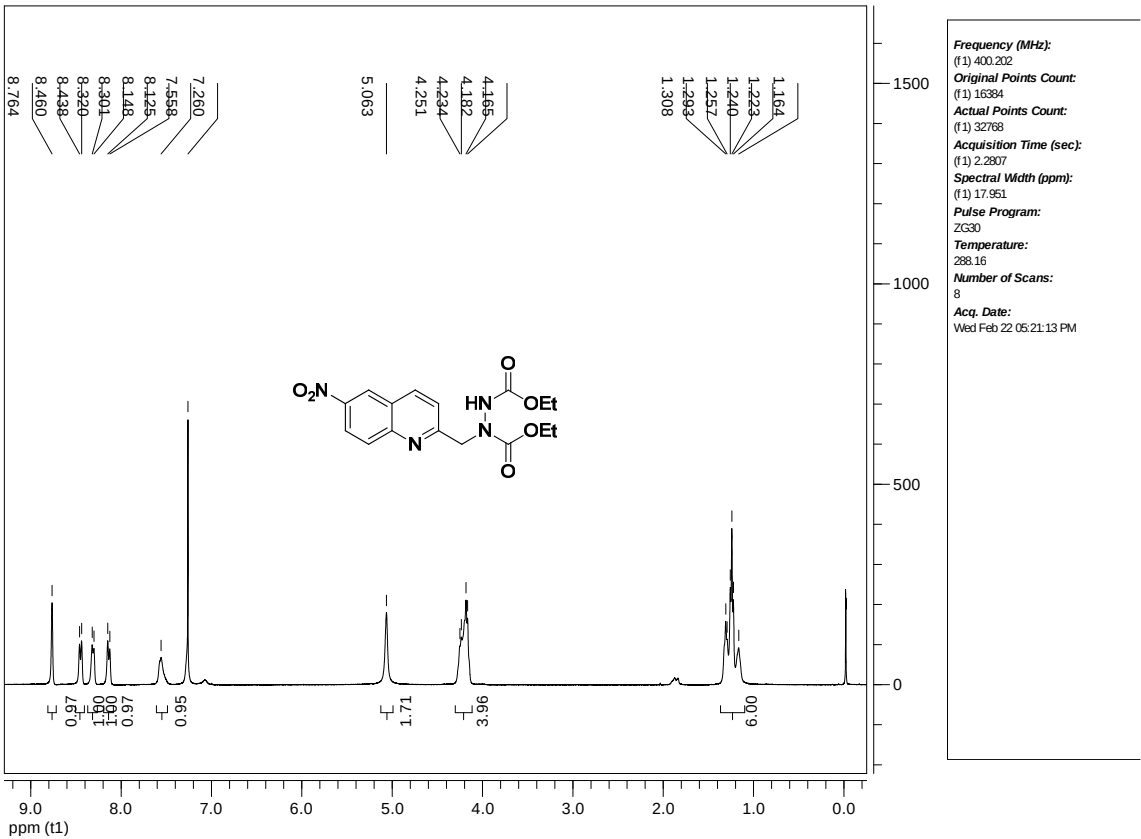
^1H NMR Spectrum for 3a



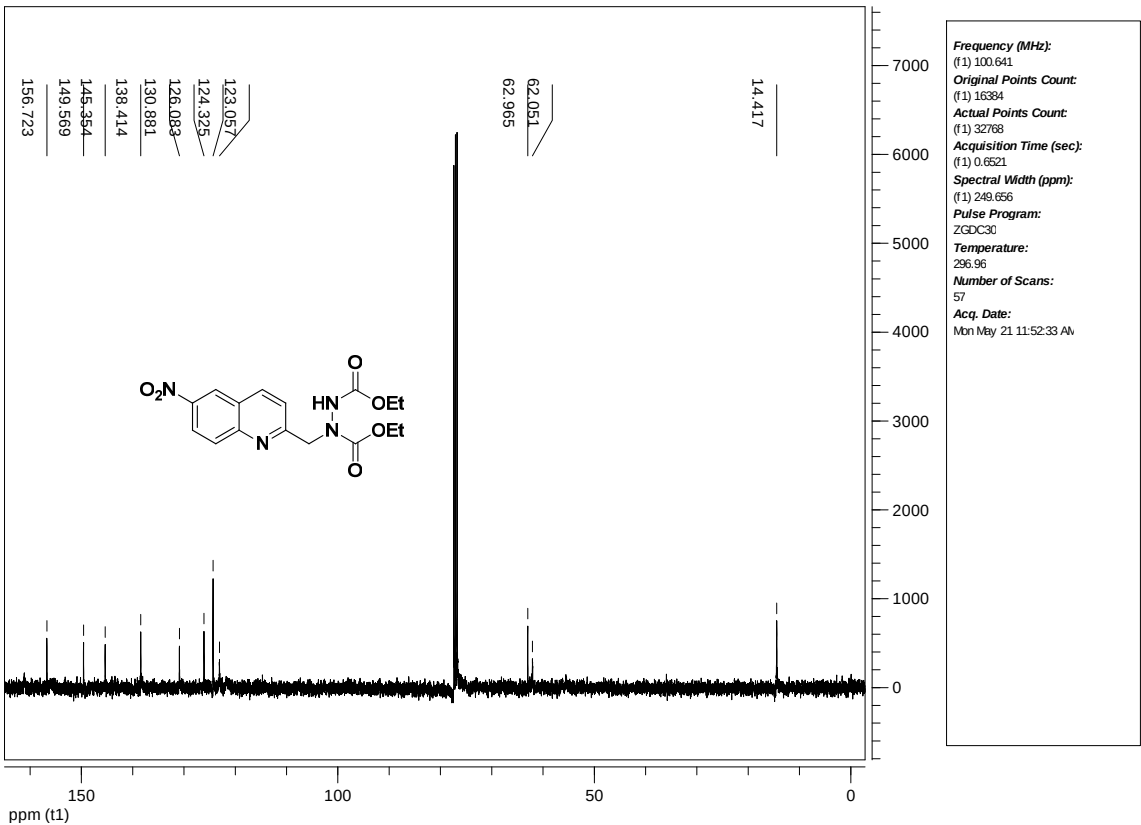
^{13}C NMR Spectrum for 3a



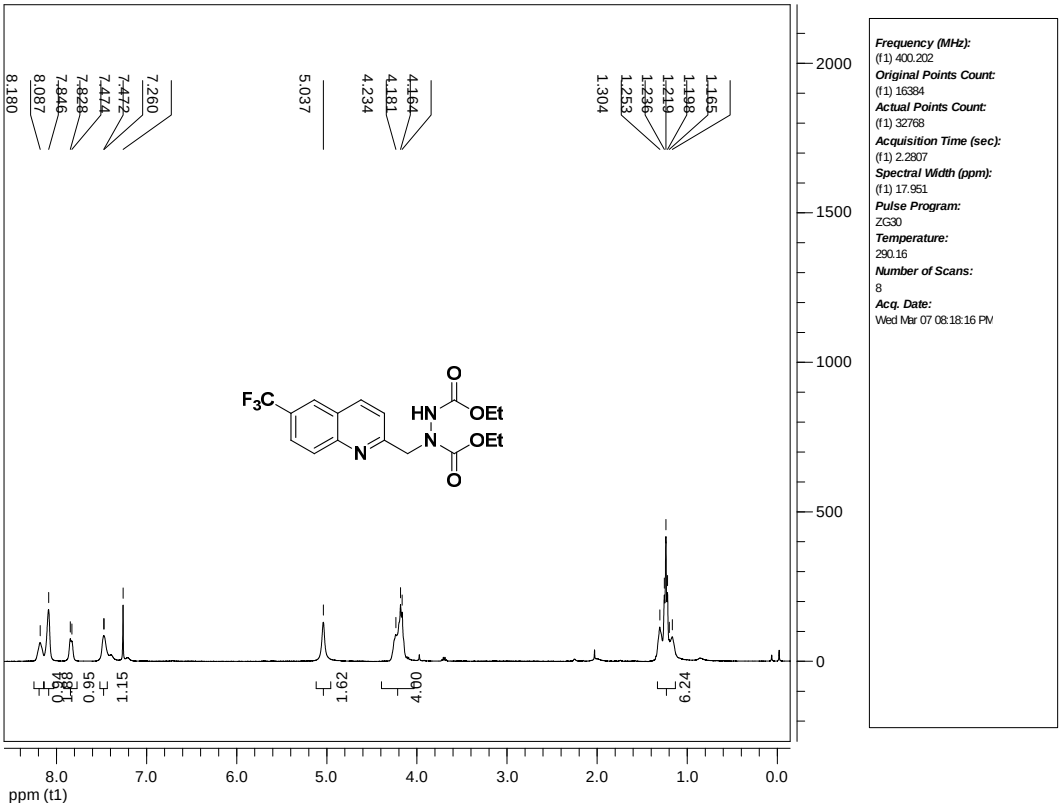
¹H NMR Spectrum for 3b



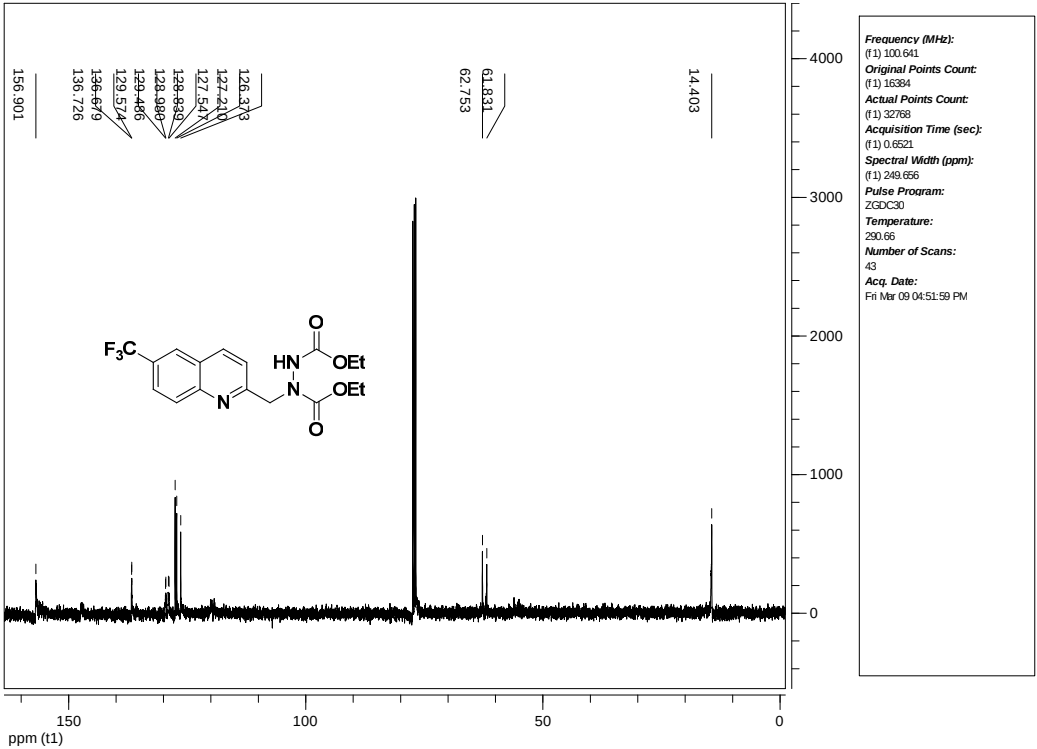
¹³C NMR Spectrum for 3b



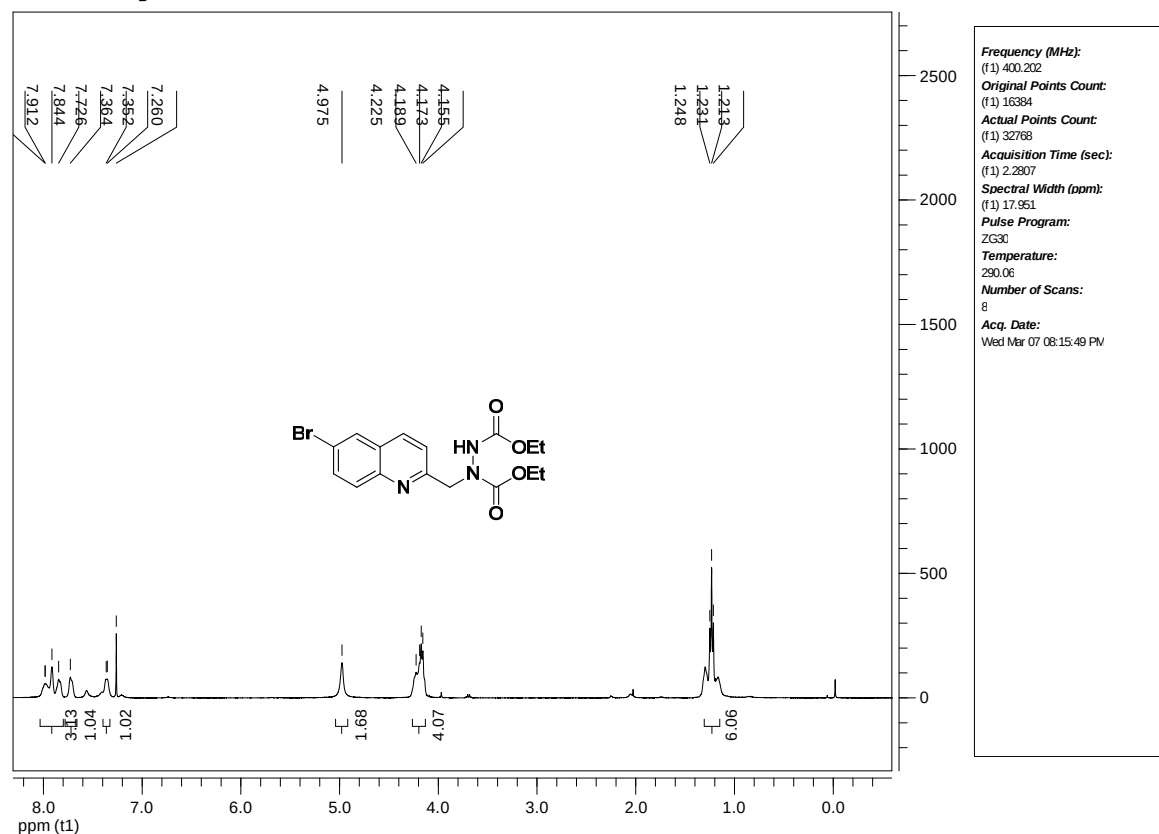
¹H NMR Spectrum for 3c



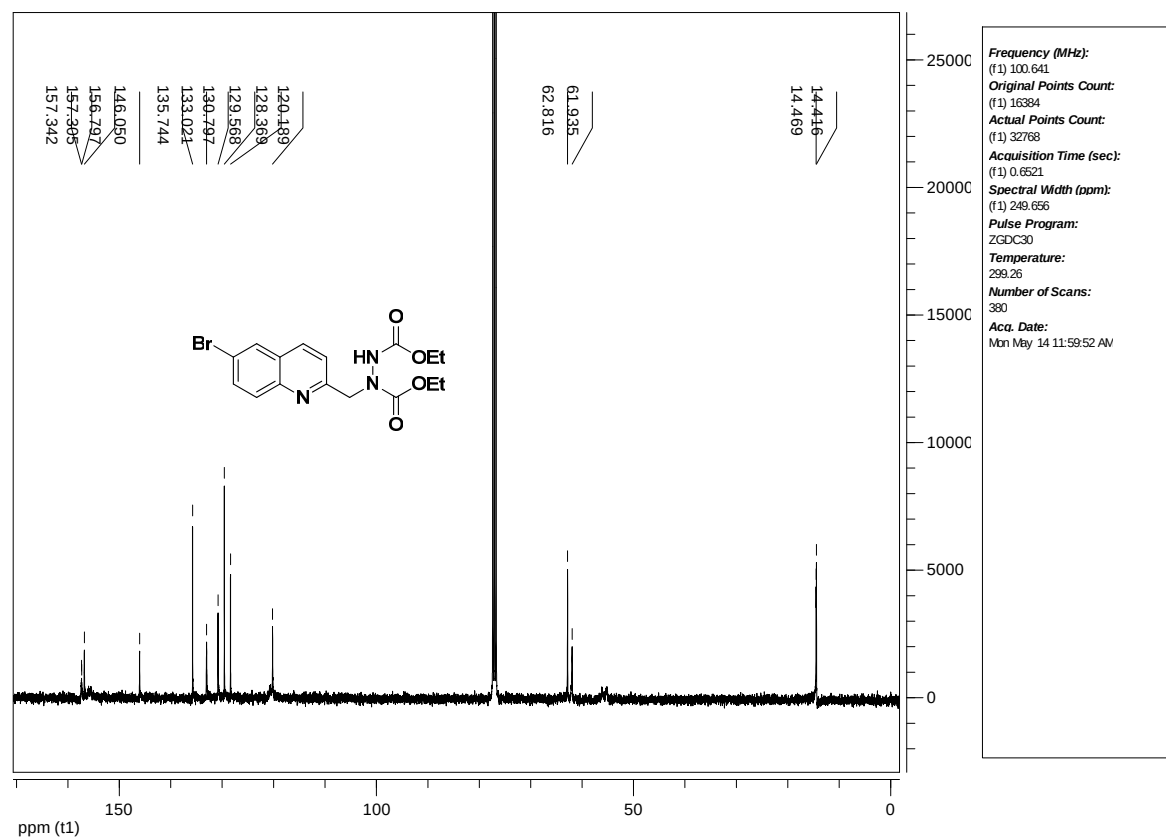
¹³C NMR Spectrum for 3c



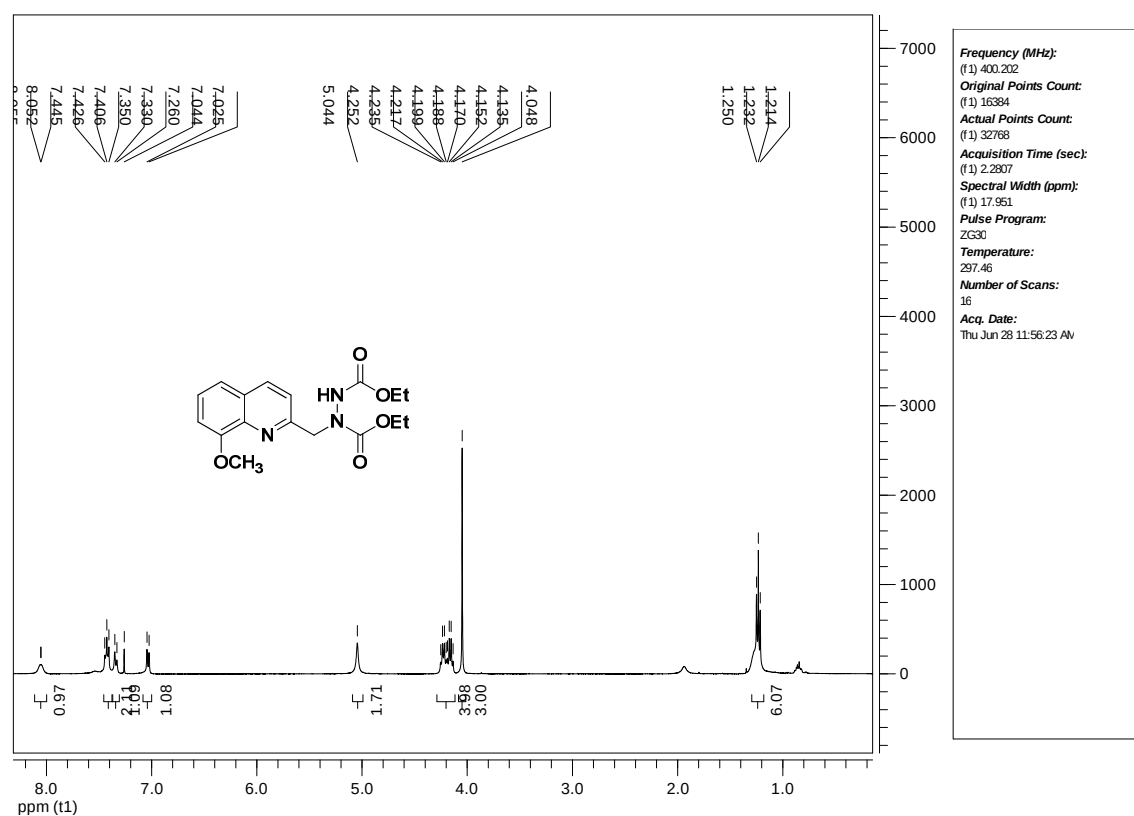
¹H NMR Spectrum for 3e



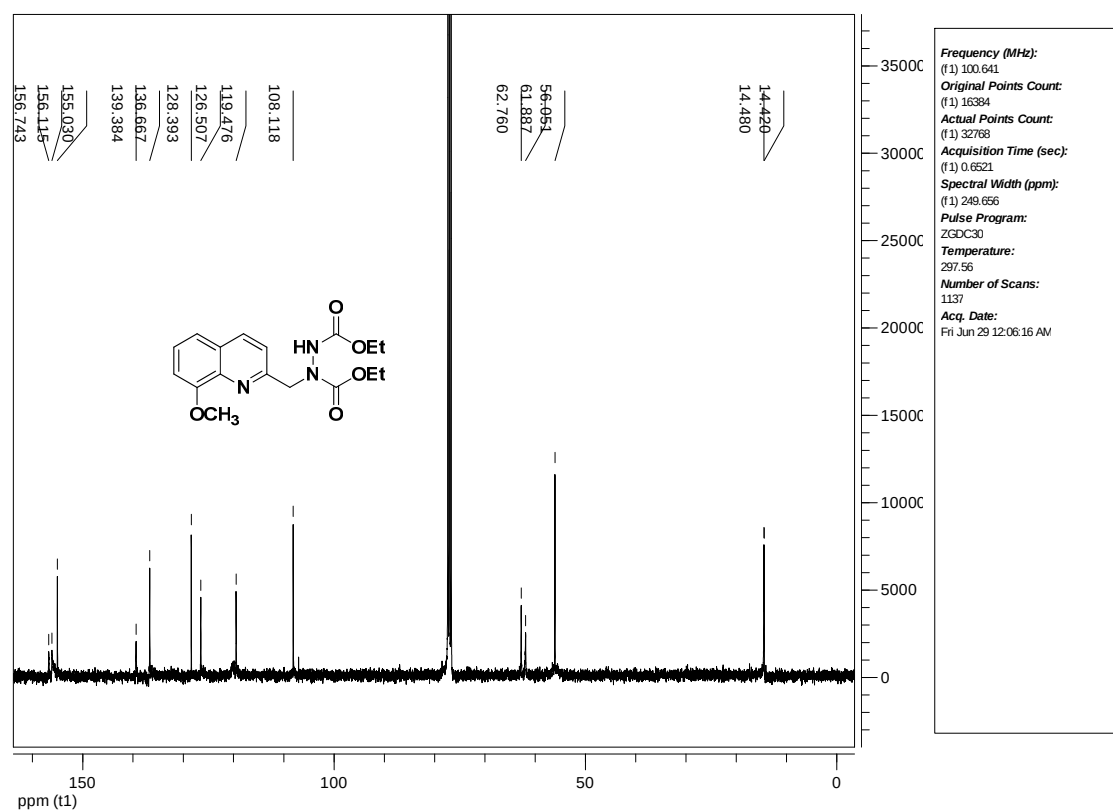
¹³C NMR Spectrum for 3e



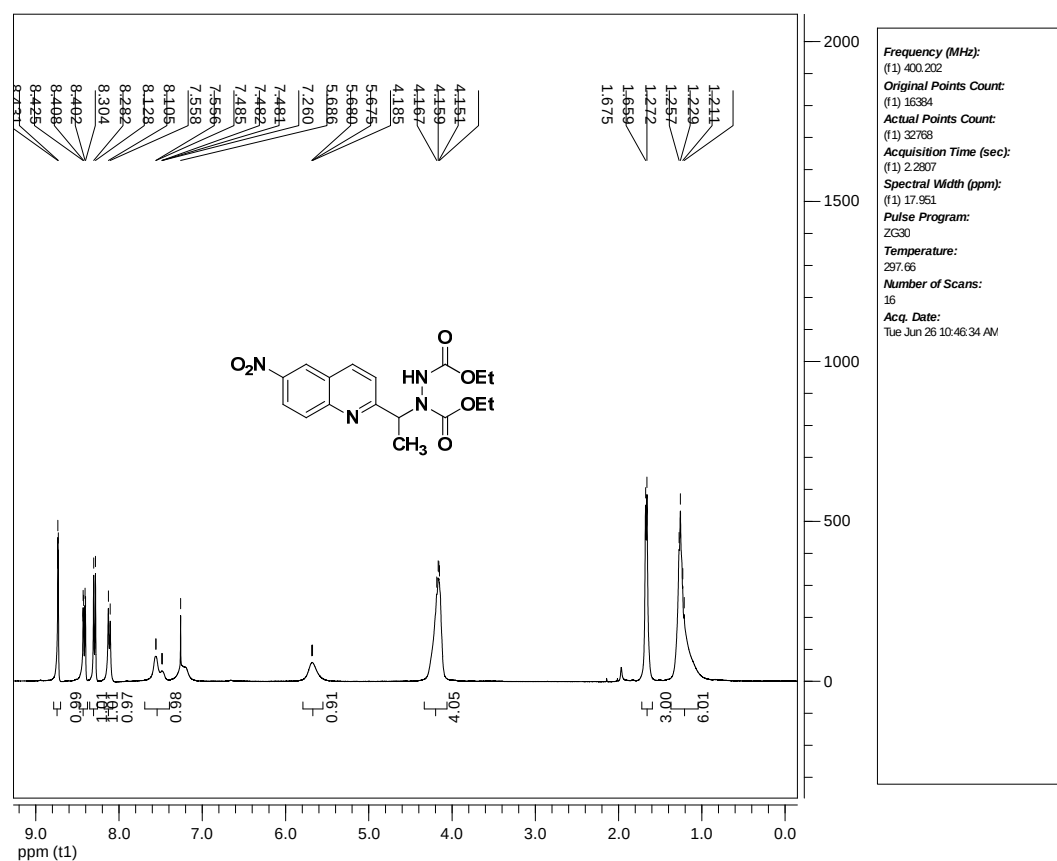
¹H NMR Spectrum for 3g



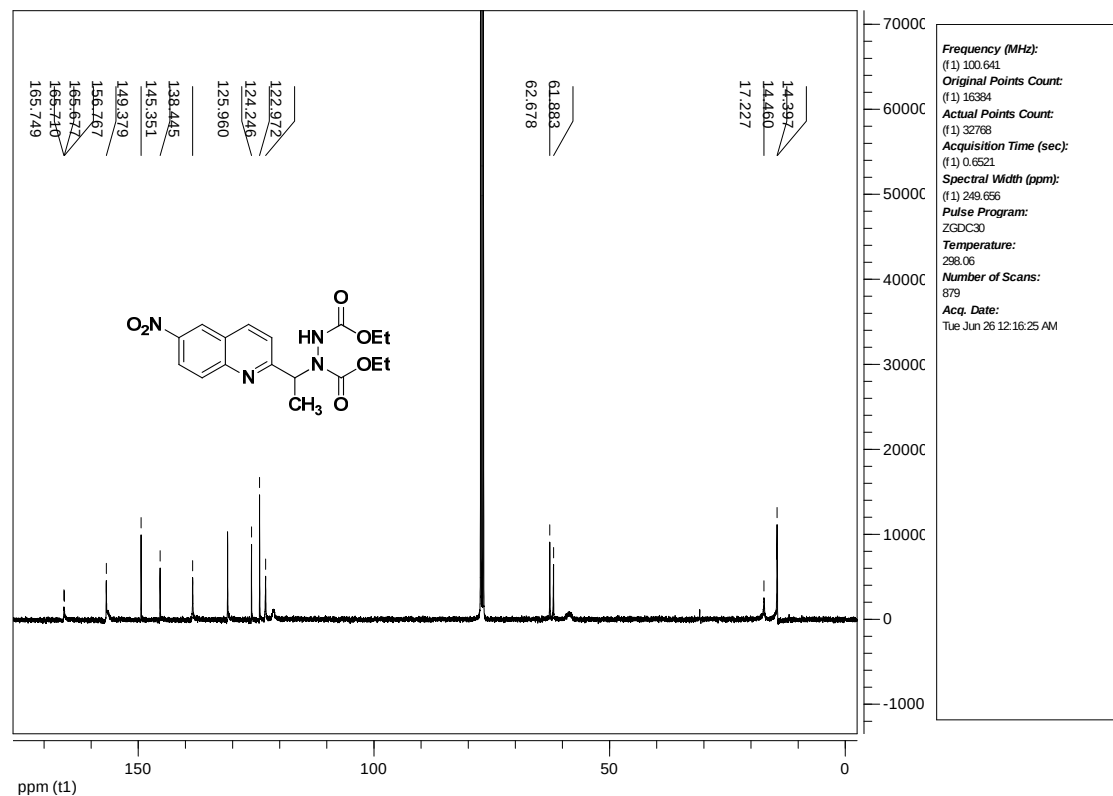
¹³C NMR Spectrum for 3g



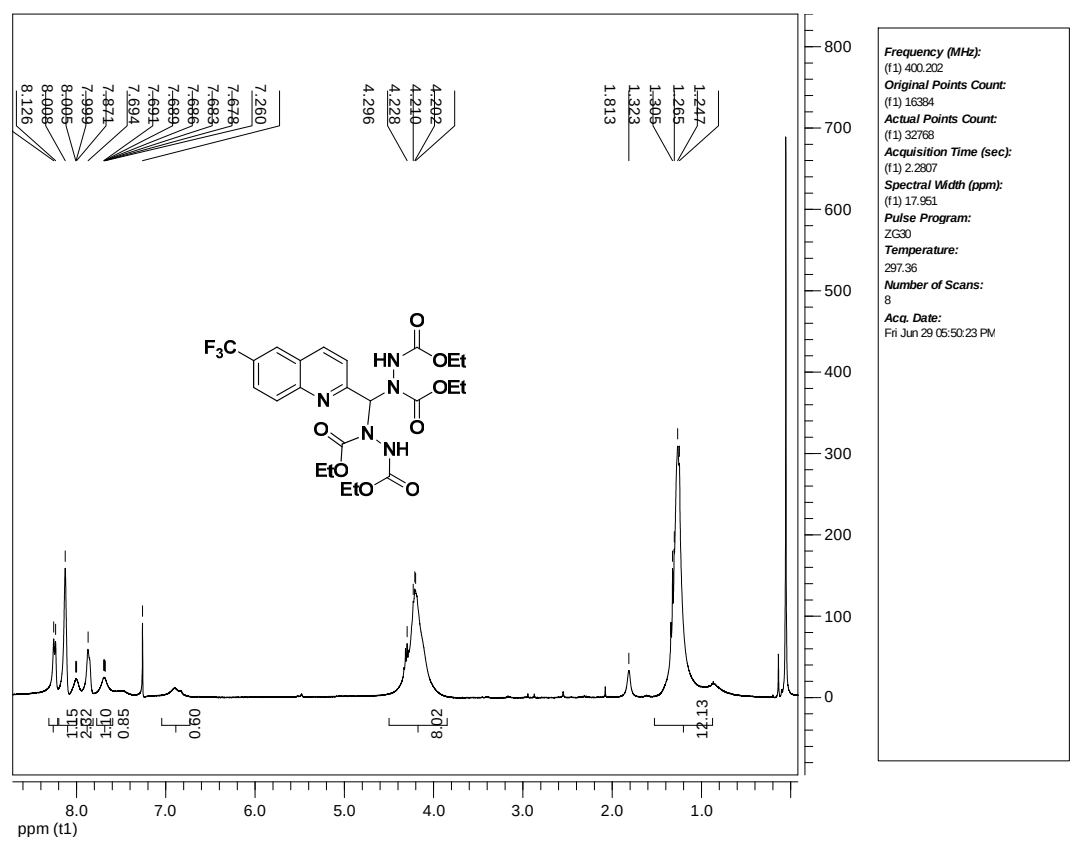
¹H NMR Spectrum for 3i



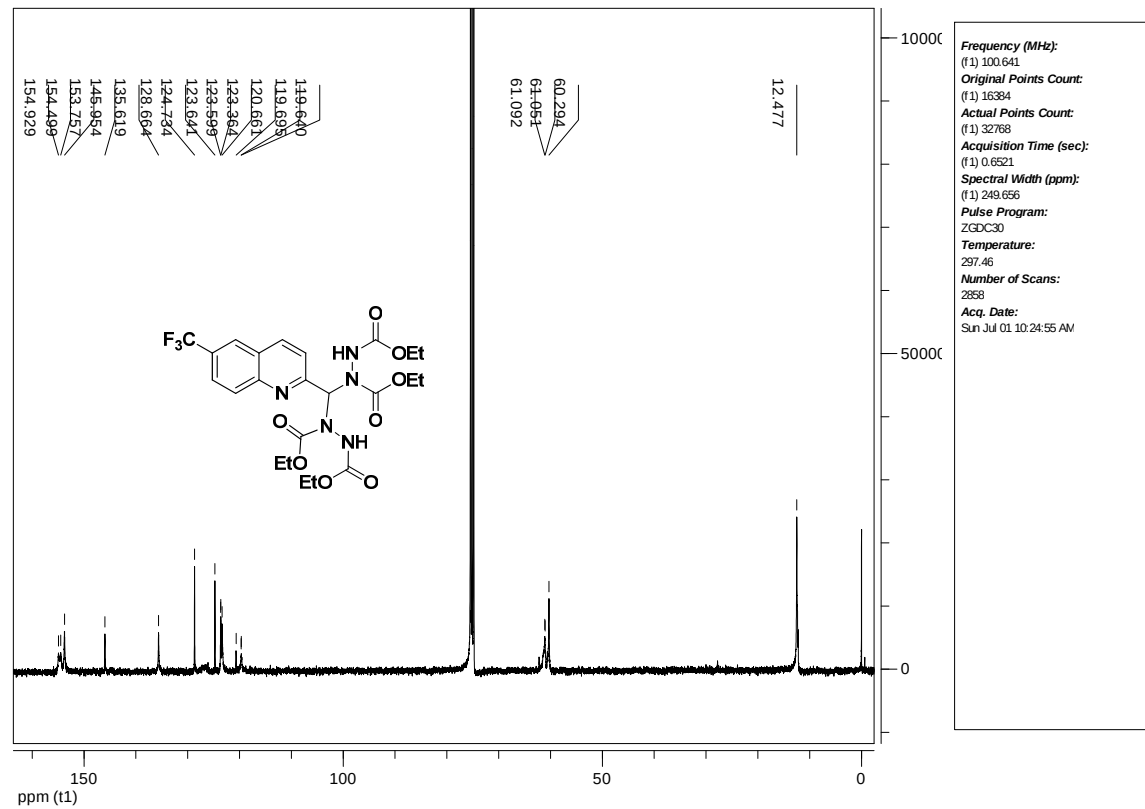
¹³C NMR Spectrum for 3i



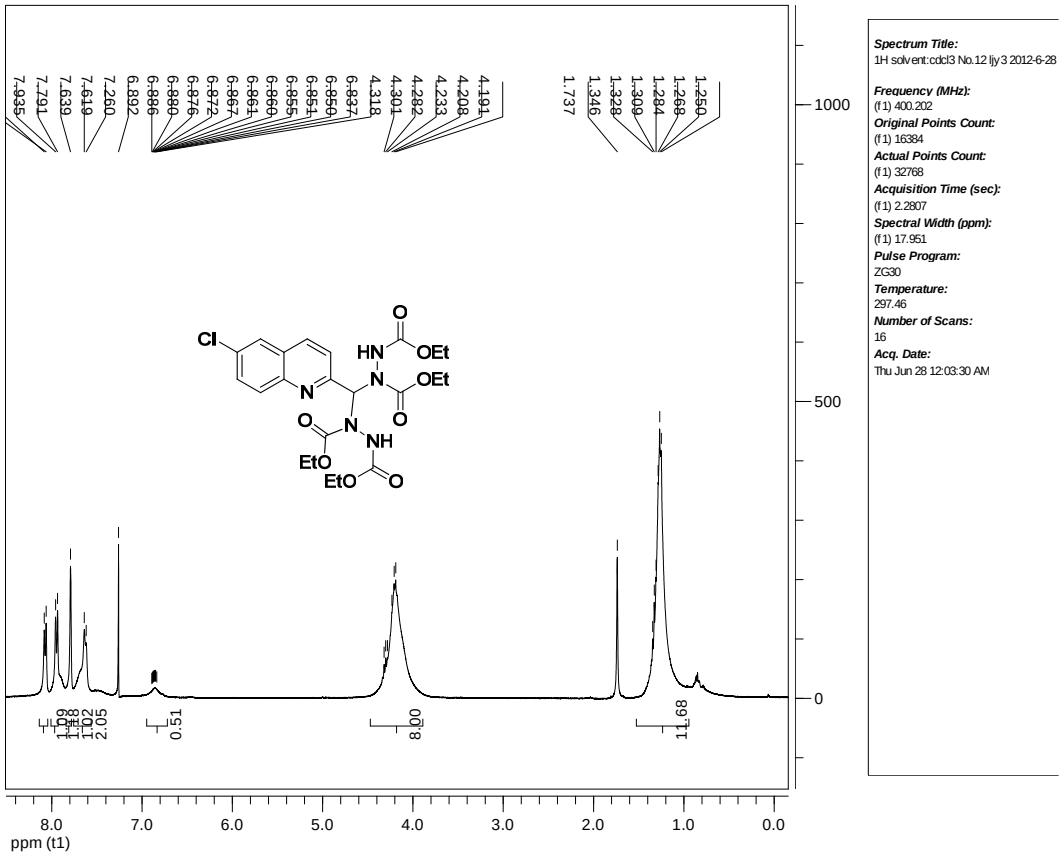
¹H NMR Spectrum for 3cc



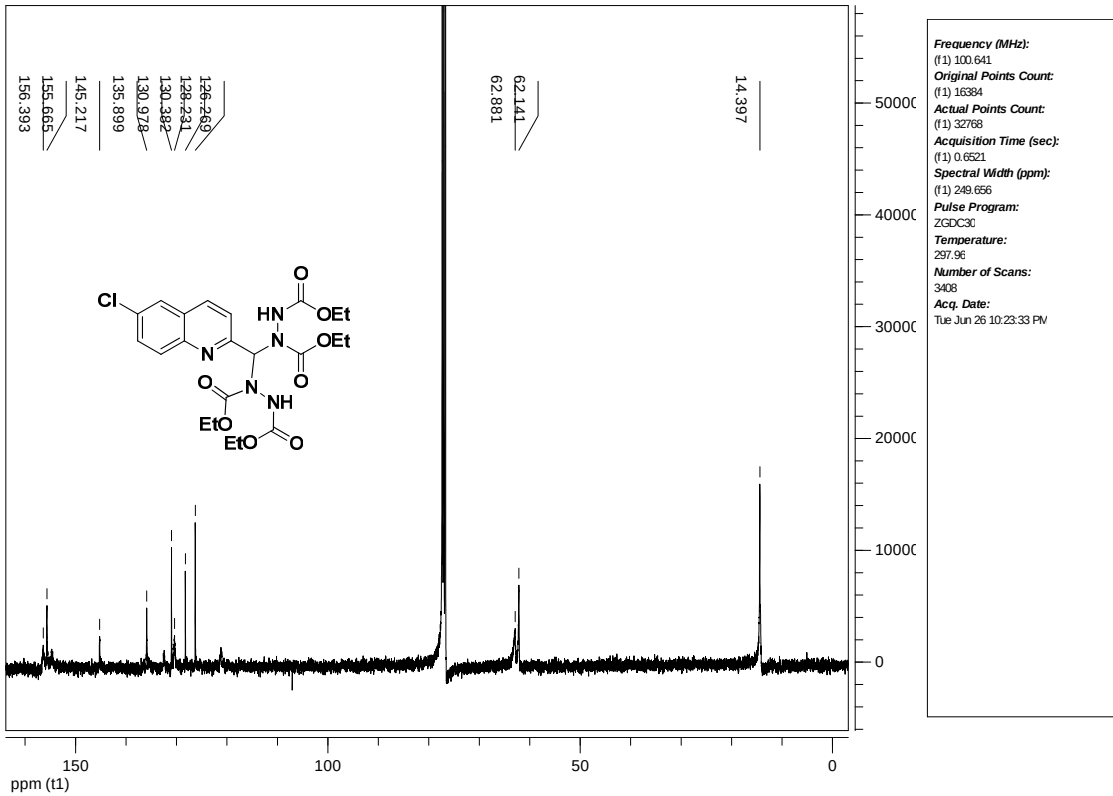
¹³C NMR Spectrum for 3cc



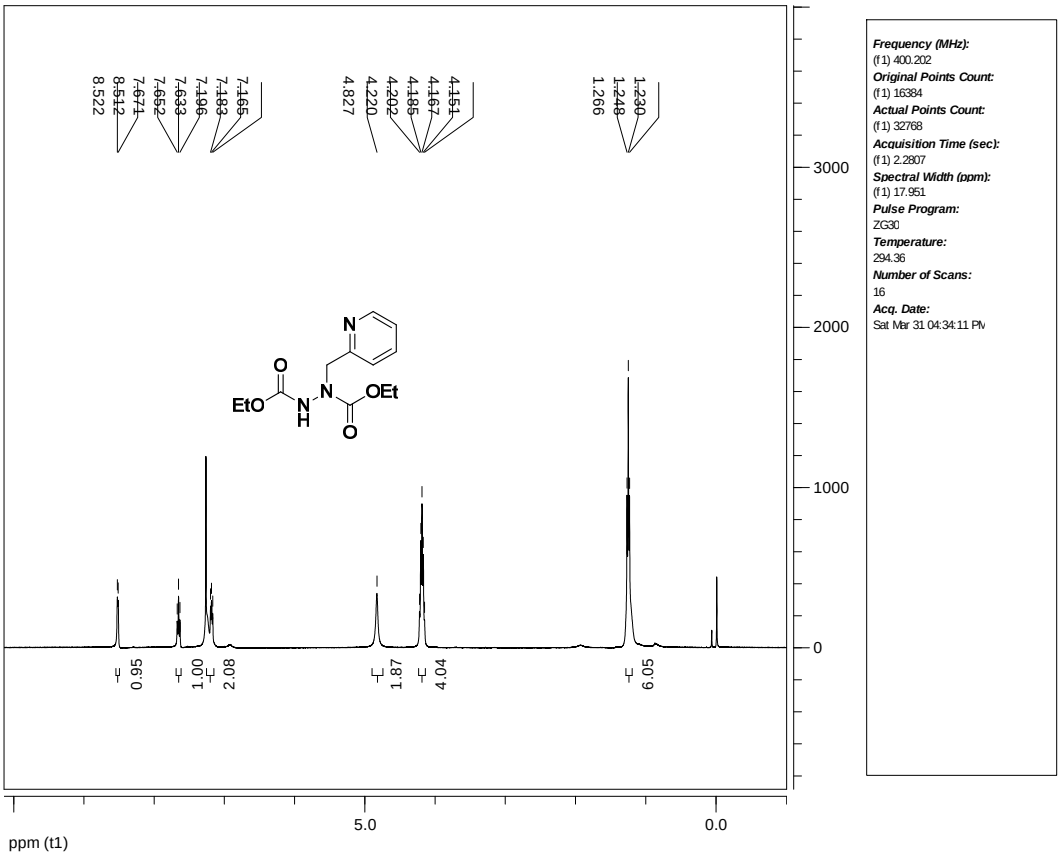
¹H NMR Spectrum for 3dd



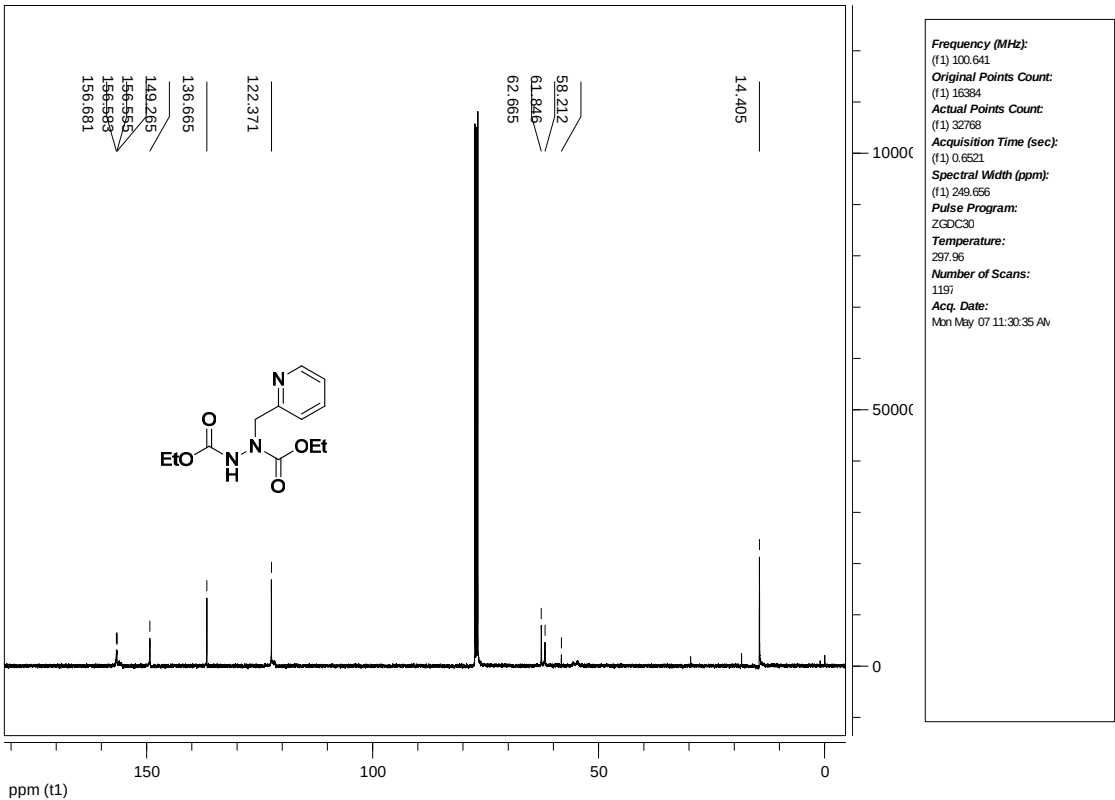
¹³C NMR Spectrum for 3dd



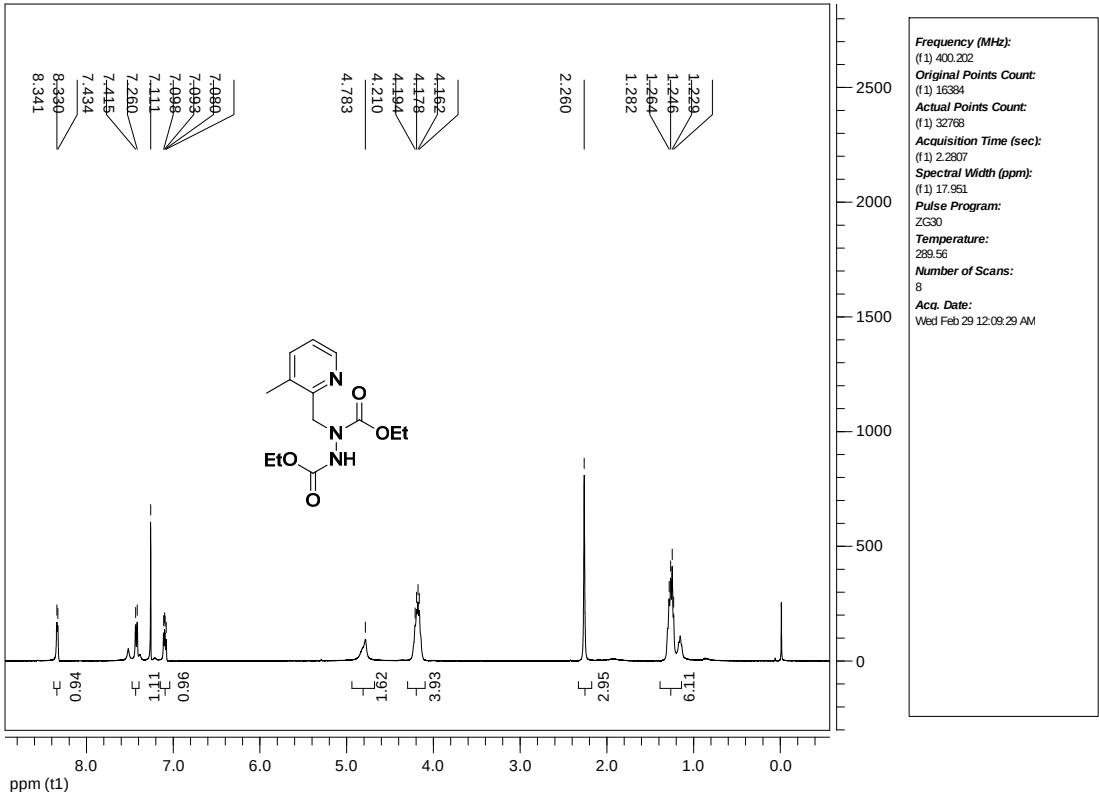
¹H NMR Spectrum for 5a



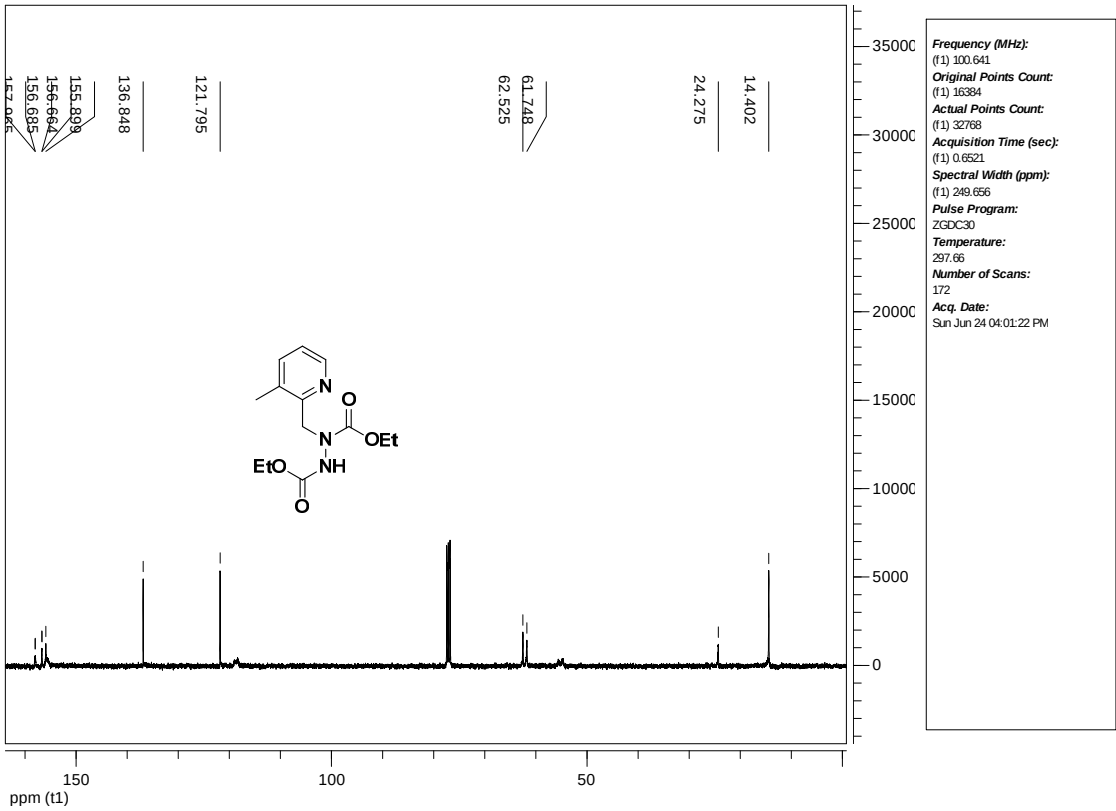
¹³C NMR Spectrum for 5a



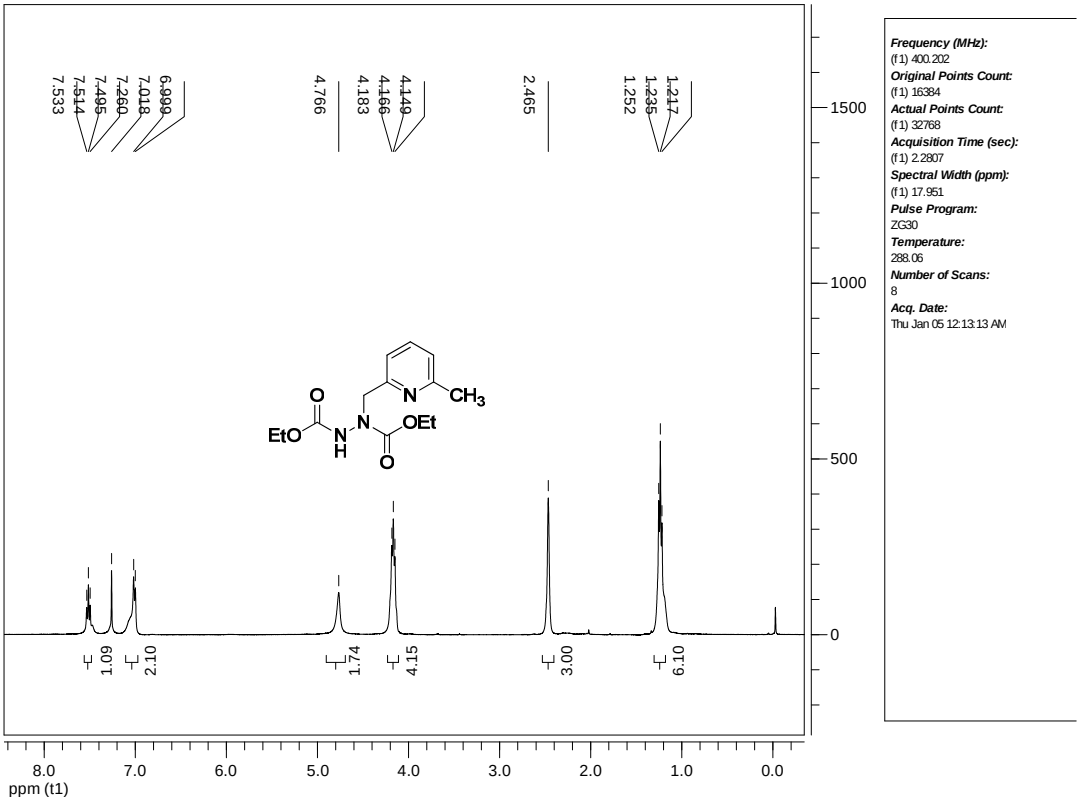
¹H NMR Spectrum for 5b



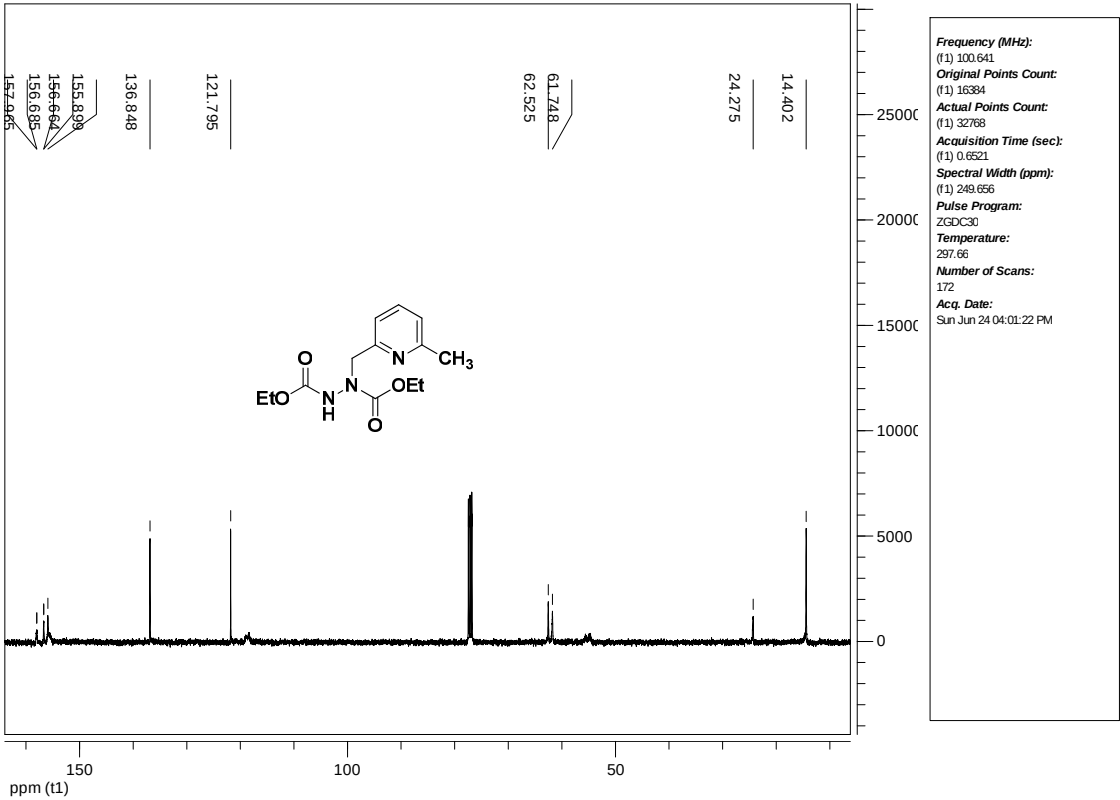
¹³C NMR Spectrum for 5b



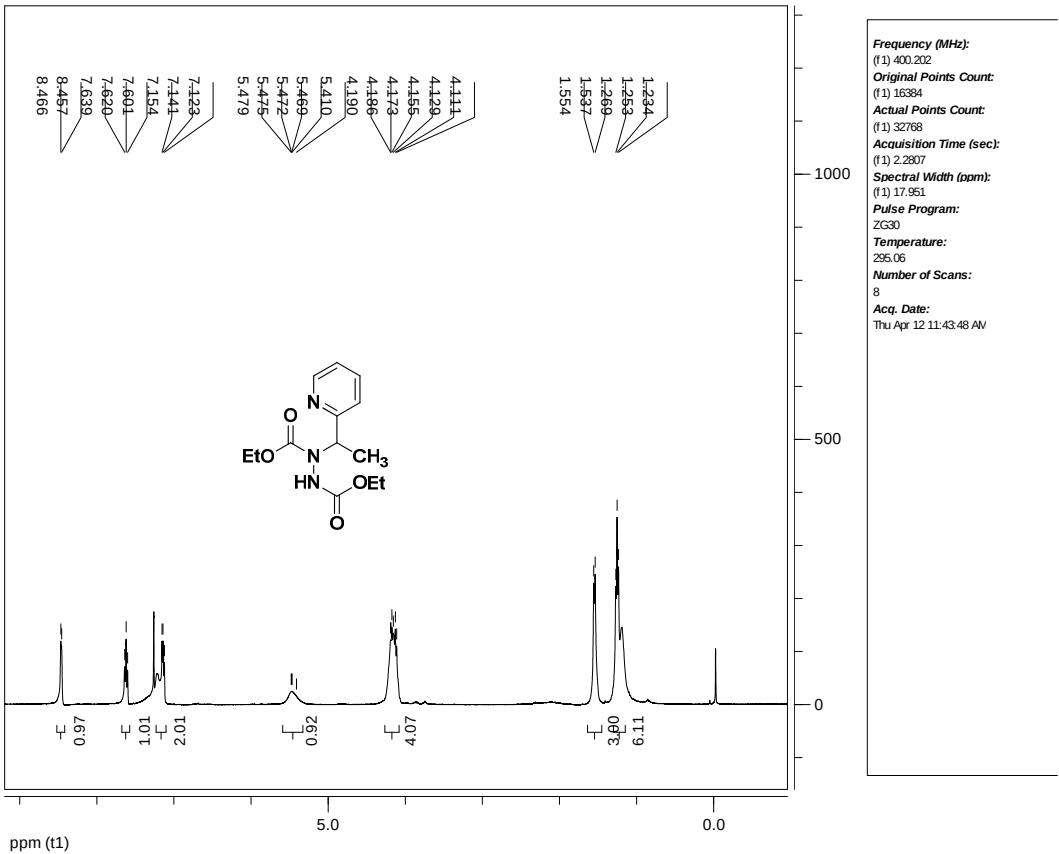
¹H NMR Spectrum for 5c



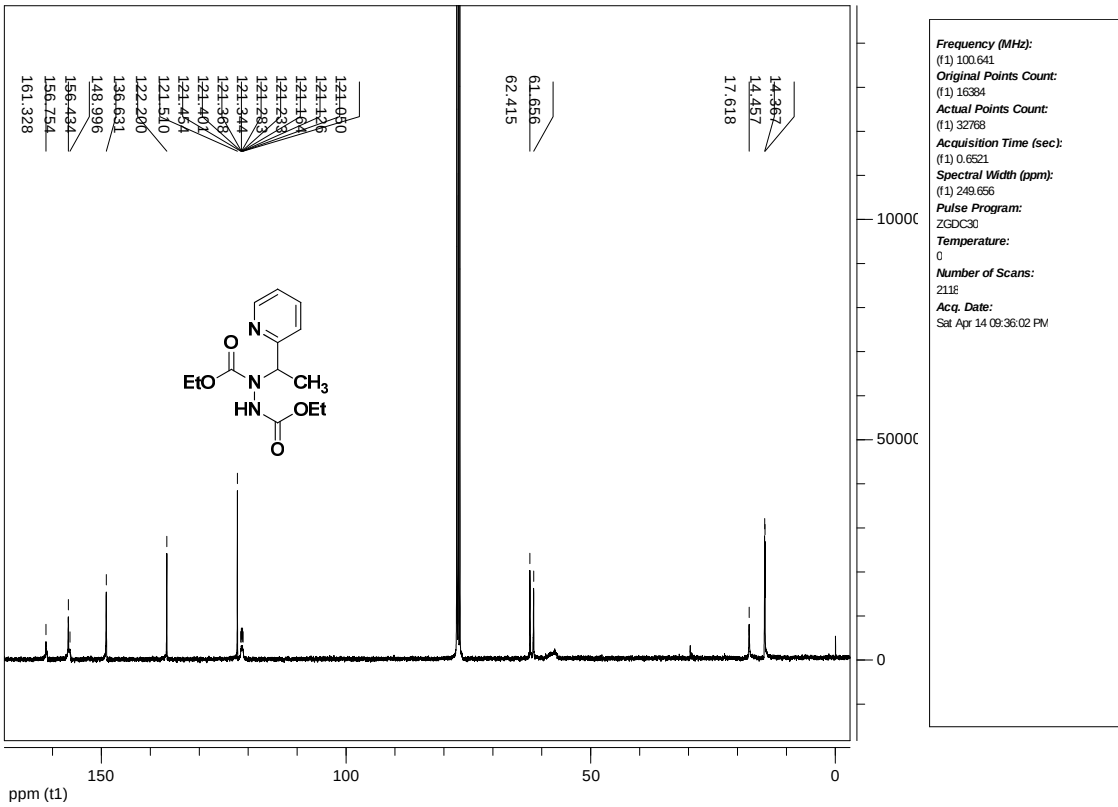
¹³C NMR Spectrum for 5c



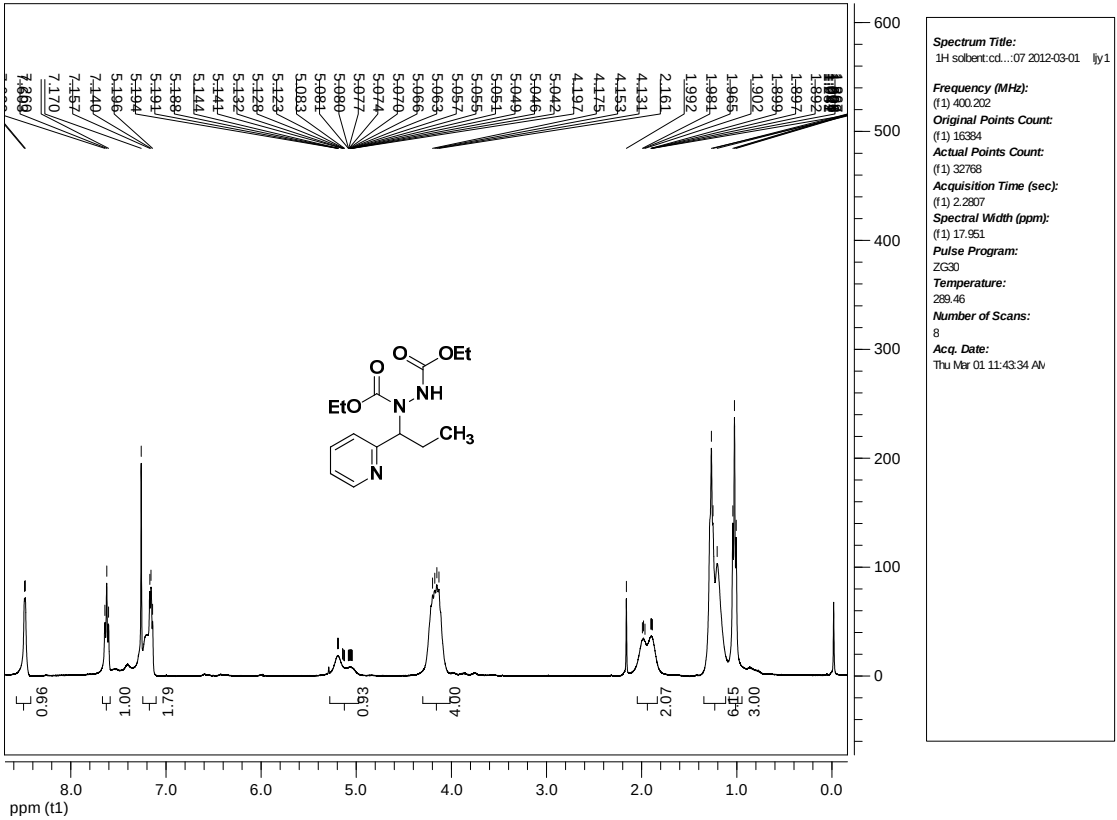
¹H NMR Spectrum for 5d



¹³C NMR Spectrum for 5d



¹H NMR Spectrum for 5e



¹³C NMR Spectrum for 5e

