

Supporting Information for

Copper-Catalysed Addition of α -Alkyl Azaarenes to Ethyl Glyoxylate via Direct C(sp³)-H Activation

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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{¹H} NMR. Chemical shifts δ are given in ppm relative to tetramethylsilane as internal standard, residual CHCl₃ for ¹H or CDCl₃ in ¹³C{¹H} 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). Tetrahydrofuran used in reactions was reagent grade and distilled from sodium. All reagents and solvents were purchased from commercial sources and purified commonly before used. All known quinolines (**1a-1l**) were prepared according to literature procedures.¹ All pyridines and ethyl glyoxylate are commercially available compounds.

General procedure for the copper-catalysed direct C(sp³)-H Activation of quinolines.

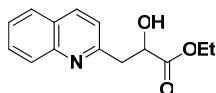
Cu(OTf)₂ (7.2 mg, 10 mol%), 1,10-phenanthroline (1.8 mg, 5 mol%), 2-methyl quinoline **1a** (82 µL, 0.6 mmol) and ethyl glyoxylate **2** (40 µL 0.2 mmol) were mixed in a Schlenk tube and then dry THF (0.8 mL) was added. The mixture was stirred at 60 °C in a closed reaction vessel. The reaction was monitored by TLC. After completion of the reaction, the solvent 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.

General procedure for the copper-catalysed direct C(sp³)-H Activation of pyridines.

Cu(OTf)₂ (7.2 mg, 10 mol%), 1,10-phenanthroline (1.8 mg, 5 mol%), 2-methyl pyridine **4a** (60 µL , 0.6 mmol) and ethyl glyoxylate **2** (40 µL, 0.2 mmol) were mixed in a Schlenk tube and then dry THF (0.8 mL) was added. The mixture was stirred at 110 °C in a closed reaction vessel. The reaction was monitored by TLC. After completion of the reaction, the solvent 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.

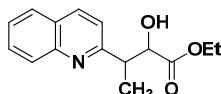
Characterisation of Compounds

Ethyl 2-hydroxy-3-(quinolin-2-yl)propanoate (3a):



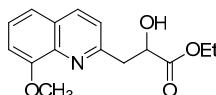
White solid. M.p. 107-109 °C. ^1H NMR (400 MHz, CDCl_3): δ 8.14 (d, $J = 8.4$ Hz, 1H), 8.07-8.05 (m, 1H), 7.81 (d, $J = 8.4$ Hz, 1H), 7.72 (t, $J = 7.2$ Hz, 1H), 7.54 (t, $J = 7.6$ Hz, 1H), 7.34 (d, $J = 8.4$ Hz, 1H), 4.77 (q, $J = 3.6$ Hz, 1H), 4.22 (q, $J = 7.2$ Hz, 2H), 3.56-3.41 (m, 2H), 1.24 (t, $J = 7.6$ Hz, 3H); ^{13}C NMR (100 MHz, CDCl_3): δ 173.6, 158.7, 147.1, 136.7, 129.7, 128.6, 127.5, 126.8, 126.2, 121.9, 70.4, 61.3, 40.9, 14.1; HRMS: calcd for $\text{C}_{14}\text{H}_{15}\text{NNaO}_3$ $[\text{M}+\text{Na}]^+$ 268.0950, found 268.0952.

Ethyl 2-hydroxy-3-(quinolin-2-yl)butanoate (3b):



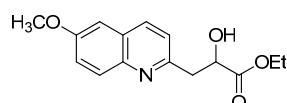
Yellow liquid. ^1H NMR (400 MHz, CDCl_3): δ 8.10 (d, $J = 8.4$ Hz, 1H), 7.99 (d, $J = 8.8$ Hz, 1H), 7.76 (d, $J = 8.0$ Hz, 1H), 7.70-7.66 (m, 1H), 7.52-7.48 (m, 1H), 7.29-7.27 (m, 1H), 4.45 (d, $J = 2.8$ Hz, 1H), 4.08-4.02 (m, 2H), 3.68-3.61 (m, 1H), 1.57 (d, $J = 7.2$ Hz, 3H), 1.04 (t, $J = 7.2$ Hz, 3H); ^{13}C NMR (100 MHz, CDCl_3): δ 172.9, 164.0, 146.9, 137.0, 129.7, 128.8, 127.5, 127.0, 126.3, 120.7, 73.6, 61.3, 43.8, 14.7, 14.2; HRMS: calcd for $\text{C}_{15}\text{H}_{17}\text{NNaO}_3$ $[\text{M}+\text{Na}]^+$ 282.1106, found 282.1109.

Ethyl 2-hydroxy-3-(8-methoxyquinolin-2-yl)propanoate (3c):



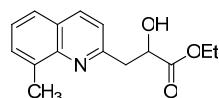
Orange liquid. ^1H NMR (400 MHz, CDCl_3): δ 8.07 (d, $J = 8.4$ Hz, 1H), 7.43 (t, $J = 7.8$ Hz, 1H), 7.36-7.33 (m, 2H), 7.03 (d, $J = 7.6$ Hz, 1H), 4.77 (q, $J = 3.6$ Hz, 1H), 4.22 (q, $J = 7.2$ Hz, 2H), 4.03 (s, 3H), 3.54-3.35 (m, 2H), 1.24 (t, $J = 7.2$ Hz, 3H); ^{13}C NMR (100 MHz, CDCl_3): δ 173.5, 157.5, 154.8, 138.9, 136.6, 127.9, 126.4, 122.3, 119.2, 108.0, 70.6, 61.2, 55.9, 40.7, 14.1; HRMS: calcd for $\text{C}_{15}\text{H}_{17}\text{NNaO}_4$ $[\text{M}+\text{Na}]^+$ 298.1055, found 298.1057.

Ethyl 2-hydroxy-3-(6-methoxyquinolin-2-yl)propanoate (3d):



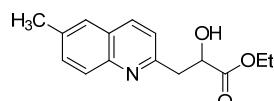
Light yellow solid, M.p. 95-97 °C. ^1H NMR (400 MHz, CDCl_3): δ 8.00 (d, $J = 8.4$ Hz, 1H), 7.89 (d, $J = 9.2$ Hz, 1H), 7.36-7.33 (m, 1H), 7.24 (s, 1H), 7.05 (d, $J = 2.4$ Hz, 1H), 4.74 (q, $J = 3.6$ Hz, 1H), 4.21 (q, $J = 7.2$ Hz, 2H), 3.92 (s, 3H), 3.48-3.33 (m, 2H), 1.23 (t, $J = 7.2$ Hz, 3H); ^{13}C NMR (100 MHz, CDCl_3): δ 173.6, 157.5, 156.0, 143.2, 135.5, 130.1, 127.8, 122.3, 122.2, 105.1, 70.5, 61.2, 55.5, 40.6, 14.1; HRMS: calcd for $\text{C}_{15}\text{H}_{18}\text{NO}_4$ $[\text{M}+\text{H}]^+$ 276.1236, found 276.1240.

Ethyl 2-hydroxy-3-(8-methylquinolin-2-yl)propanoate (3e):



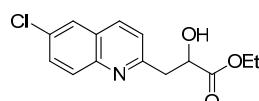
Yellow liquid. ^1H NMR (400 MHz, CDCl_3): δ 8.08 (d, $J = 8.4$ Hz, 1H), 7.63 (d, $J = 8.0$ Hz, 1H), 7.55 (d, $J = 6.8$ Hz, 1H), 7.41 (t, $J = 7.6$ Hz, 1H), 7.29 (s, 1H), 4.74 (q, $J = 3.6$ Hz, 1H), 4.20 (q, $J = 7.2$ Hz, 2H), 3.54-3.43 (m, 2H), 2.76 (s, 3H), 1.22 (t, $J = 7.2$ Hz, 3H); ^{13}C NMR (100 MHz, CDCl_3): δ 173.4, 157.8, 145.9, 137.2, 136.1, 130.1, 126.8, 126.1, 125.6, 121.5, 70.5, 61.1, 40.0, 18.1, 14.1; HRMS: calcd for $\text{C}_{15}\text{H}_{17}\text{NNaO}_3$ $[\text{M}+\text{Na}]^+$ 282.1106, found 282.1111.

Ethyl 2-hydroxy-3-(6-methylquinolin-2-yl)propanoate (3f):



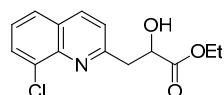
Yellow solid, M.p. 66-68 °C. ^1H NMR (400 MHz, CDCl_3): δ 8.00 (d, $J = 8.4$ Hz, 1H), 7.87 (d, $J = 8.4$ Hz, 1H), 7.53-7.50 (m, 2H), 7.26-7.24 (m, 1H), 4.75 (q, $J = 3.6$ Hz, 1H), 4.20 (q, $J = 7.2$ Hz, 2H), 3.49-3.40 (m, 2H), 2.51 (s, 3H), 1.22 (t, $J = 7.2$ Hz, 3H); ^{13}C NMR (100 MHz, CDCl_3): δ 173.6, 157.7, 145.7, 136.1, 131.9, 128.3, 126.9, 126.4, 121.9, 107.0, 70.5, 61.2, 40.8, 21.5, 14.1; HRMS: calcd for $\text{C}_{15}\text{H}_{18}\text{NO}_3$ $[\text{M}+\text{H}]^+$ 260.1287, found 260.1283.

Ethyl 3-(6-chloroquinolin-2-yl)-2-hydroxypropanoate (3g):



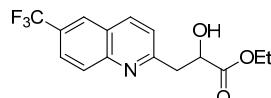
Yellow liquid. ^1H NMR (400 MHz, CDCl_3): δ 8.01 (d, $J = 8.4$ Hz, 1H), 7.93 (d, $J = 9.2$ Hz, 1H), 7.77 (d, $J = 2.0$ Hz, 1H), 7.63-7.61 (m, 1H), 7.33 (d, $J = 8.4$ Hz, 1H), 4.74 (q, $J = 3.2$ Hz, 1H), 4.22 (q, $J = 7.2$ Hz, 2H), 3.51-3.36 (m, 2H), 1.23 (t, $J = 7.2$ Hz, 3H); ^{13}C NMR (100 MHz, CDCl_3): δ 173.2, 159.9, 143.1, 137.1, 132.9, 129.7, 128.0, 126.5, 126.2, 122.7, 70.4, 61.3, 40.2, 14.1; HRMS: calcd for $\text{C}_{14}\text{H}_{14}\text{ClNNaO}_3$ $[\text{M}+\text{Na}]^+$ 302.0560, found 302.0556.

Ethyl 3-(8-chloroquinolin-2-yl)-2-hydroxypropanoate (3h):



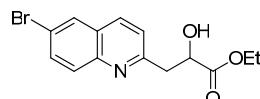
Yellow liquid. ^1H NMR (400 MHz, CDCl_3): δ 8.12 (d, $J = 8.4$ Hz, 1H), 7.80 (d, $J = 7.6$ Hz, 1H), 7.71 (d, $J = 8.4$ Hz, 1H), 7.42 (t, $J = 8.0$ Hz, 1H), 7.36 (d, $J = 8.4$ Hz, 1H), 5.93 (d, $J = 6.0$ Hz, 1H), 4.81 (d, $J = 4.0$ Hz, 1H), 4.21 (q, $J = 7.2$ Hz, 2H), 3.57-3.45 (m, 2H), 1.23 (t, $J = 7.2$ Hz, 3H); ^{13}C NMR (100 MHz, CDCl_3): δ 173.6, 159.0, 145.6, 135.7, 131.9, 130.6, 130.3, 127.5, 126.2, 122.9, 70.2, 61.4, 41.2, 14.1; HRMS: calcd for $\text{C}_{14}\text{H}_{14}\text{ClNNaO}_3$ $[\text{M}+\text{Na}]^+$ 302.0560, found 302.0565.

Ethyl 2-hydroxy-3-(6-(trifluoromethyl)quinolin-2-yl)propanoate(3i):



Yellow solid, M.p. 79-82 °C. ^1H NMR (400 MHz, CDCl_3): δ 8.19 (d, $J = 8.4$ Hz, 1H), 8.11 (d, $J = 5.2$ Hz, 2H), 7.86 (dd, $J_1 = 1.6$ Hz, $J_2 = 1.6$ Hz, 1H), 7.42 (d, $J = 8.4$ Hz, 1H), 4.77 (q, $J = 3.6$ Hz, 1H), 4.23 (q, $J = 7.2$ Hz, 2H), 3.56-3.41 (m, 2H), 1.24 (t, $J = 7.2$ Hz, 3H); ^{13}C NMR (100 MHz, CDCl_3): δ 173.6, 161.1, 148.2, 137.3, 130.0, 128.3, 127.9, 125.8, 125.5, 125.5, 125.3, 125.3, 123.3, 122.6, 70.1, 61.5, 41.5, 14.1; HRMS: calcd for $\text{C}_{15}\text{H}_{15}\text{F}_3\text{NO}_3$ $[\text{M}+\text{H}]^+$ 314.1004, found 314.1000.

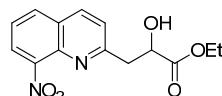
Ethyl 3-(6-bromoquinolin-2-yl)-2-hydroxypropanoate (3j):



Orange liquid. ^1H NMR (400 MHz, CDCl_3): δ 8.00 (d, $J = 8.4$ Hz, 1H), 7.94 (d, $J = 1.6$ Hz, 2H), 7.86 (d, $J = 8.8$ Hz, 1H), 7.75 (dd, $J_1 = 2.0$ Hz, $J_2 = 2.0$ Hz, 1H), 7.32 (d, $J = 8.4$ Hz, 1H), 4.77 (q, $J = 3.6$ Hz, 1H), 4.23 (q, $J = 7.2$ Hz, 2H), 3.56-3.41 (m, 2H), 1.24 (t, $J = 7.2$ Hz, 3H); ^{13}C NMR (100 MHz, CDCl_3): δ 173.5,

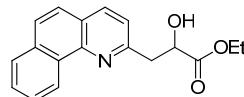
159.1, 135.6, 133.0, 130.0, 130.4, 129.5, 127.9, 122.9, 119.9, 70.1, 61.4, 41.2, 14.1; HRMS: calcd for $C_{14}H_{14}BrNNaO_3 [M+Na]^+$ 346.0055, found 346.0059.

Ethyl 2-hydroxy-3-(8-nitroquinolin-2-yl)propanoate (3k):



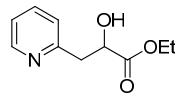
Red solid, M.p. 98-101 °C. 1H NMR (400 MHz, $CDCl_3$): δ 8.20 (d, $J = 8.4$ Hz, 1H), 8.10 (dd, $J_1 = 0.8$ Hz, $J_2 = 1.2$ Hz, 1H), 8.02 (dd, $J_1 = 0.8$ Hz, $J_2 = 0.8$ Hz, 1H), 7.59 (t, $J = 8.0$ Hz, 1H), 7.46 (d, $J = 8.4$ Hz, 1H), 4.76 (q, $J = 5.2$ Hz, 1H), 4.65 (d, $J = 6.8$ Hz, 1H), 4.22 (q, $J = 7.2$ Hz, 2H), 3.58-3.48 (m, 2H), 1.27 (t, $J = 7.2$ Hz, 3H); ^{13}C NMR (100 MHz, $CDCl_3$): δ 173.4, 161.3, 147.2, 138.5, 136.6, 132.2, 127.7, 124.9, 124.6, 123.9, 69.8, 61.6, 41.2, 14.1; HRMS: calcd for $C_{14}H_{14}N_2NaO_5 [M+Na]^+$ 313.0800, found 313.0802.

Ethyl 3-(benzo[h]quinolin-2-yl)-2-hydroxypropanoate (3l):



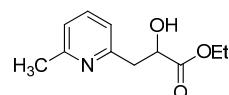
Red-brown liquid. 1H NMR (400 MHz, $CDCl_3$): δ 9.16 (d, $J = 7.6$ Hz, 1H), 8.14 (d, $J = 8.4$ Hz, 1H), 7.91 (t, $J = 7.2$ Hz, 1H), 7.81-7.66 (m, 4H), 7.42 (d, $J = 8.0$ Hz, 1H), 4.84 (q, $J = 3.2$ Hz, 1H), 4.23 (q, $J = 7.2$ Hz, 2H), 3.64-3.54 (m, 2H), 1.23 (t, $J = 7.2$ Hz, 3H); ^{13}C NMR (100 MHz, $CDCl_3$): δ 173.8, 157.1, 145.5, 136.7, 133.8, 130.8, 128.3, 127.9, 127.5, 127.1, 125.0, 124.9, 124.3, 122.4, 70.3, 61.3, 40.7, 14.1; HRMS: calcd for $C_{18}H_{17}NNaO_5 [M+Na]^+$ 318.1106, found 318.1104.

Ethyl 2-hydroxy-3-(pyridin-2-yl)propanoate (5a):



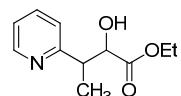
Light yellow liquid. 1H NMR (400 MHz, $CDCl_3$): δ 8.51 (d, $J = 4.4$ Hz, 1H), 7.70-7.65 (m, 1H), 7.24-7.20 (m, 2H), 4.65 (q, $J = 3.6$ Hz, 1H), 4.21 (q, $J = 7.2$ Hz, 2H), 3.38-3.19 (m, 2H), 1.24 (t, $J = 7.2$ Hz, 3H); ^{13}C NMR (100 MHz, $CDCl_3$): δ 173.7, 158.0, 148.7, 136.8, 123.9, 121.9, 70.5, 61.3, 40.6, 14.1; HRMS: calcd for $C_{10}H_{14}NO_3 [M+H]^+$ 196.0974, found 196.0976.

Ethyl 2-hydroxy-3-(6-methylpyridin-2-yl)propanoate (5b):



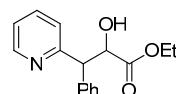
Light yellow liquid. ^1H NMR (400 MHz, CDCl_3): δ 7.51 (t, $J = 8.0$ Hz, 1H), 7.03-6.96 (m, 2H), 4.62 (q, $J = 3.6$ Hz, 1H), 4.19 (q, $J = 7.2$ Hz, 2H), 3.28-3.11 (m, 2H), 2.51 (s, 3H), 1.23 (t, $J = 7.2$ Hz, 3H); ^{13}C NMR (100 MHz, CDCl_3): δ 173.9, 162.8, 148.6, 137.1, 123.0, 122.1, 75.5, 61.0, 43.2, 18.7, 14.2; HRMS: calcd for $\text{C}_{11}\text{H}_{16}\text{NO}_3$ $[\text{M}+\text{H}]^+$ 210.1130, found 210.1134.

Ethyl 2-hydroxy-3-(pyridin-2-yl)butanoate (5c):



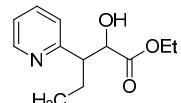
Light yellow liquid. ^1H NMR (400 MHz, CDCl_3): δ 8.51 (d, $J = 4.0$ Hz, 1H), 7.69-7.64 (m, 1H), 7.24-7.16 (m, 2H), 4.65 (d, $J = 2.8$ Hz, 1H), 4.25 (q, $J = 7.2$ Hz, 2H), 3.46-3.39 (m, 1H), 1.33-1.26 (m, 6H); ^{13}C NMR (100 MHz, CDCl_3): δ 173.9, 162.8, 148.6, 137.1, 123.0, 122.1, 75.5, 61.0, 43.2, 18.7, 14.2; HRMS: calcd for $\text{C}_{11}\text{H}_{16}\text{NO}_3$ $[\text{M}+\text{H}]^+$ 210.1130, found 210.1133.

Ethyl 2-hydroxy-3-phenyl-3-(pyridin-2-yl)propanoate (5d):



Yellow liquid. ^1H NMR (400 MHz, CDCl_3): δ 8.53 (d, $J = 4.4$ Hz, 1H), 7.62-7.58 (m, 1H), 7.34-7.28 (m, 4H), 7.24-7.17 (m, 2H), 7.10 (d, $J = 7.6$ Hz, 1H), 4.74 (d, $J = 4.0$ Hz, 1H), 4.62 (q, $J = 4.4$ Hz, 1H), 4.12-4.03 (m, 2H), 1.07 (t, $J = 7.2$ Hz, 3H); ^{13}C NMR (100 MHz, CDCl_3): δ 173.2, 160.8, 148.0, 140.1, 137.1, 128.9, 128.5, 127.1, 124.6, 122.1, 75.6, 61.9, 53.7, 14.0; HRMS: calcd for $\text{C}_{16}\text{H}_{18}\text{NO}_3$ $[\text{M}+\text{H}]^+$ 272.1287, found 272.1289.

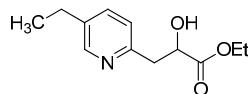
Ethyl 2-hydroxy-3-(pyridin-2-yl)pentanoate (5e):



Light yellow liquid. ^1H NMR (400 MHz, CDCl_3): δ 8.47 (d, $J = 4.0$ Hz, 1H), 7.63-7.58 (m, 1H), 7.18-7.14 (m, 1H), 7.08 (d, $J = 7.6$ Hz, 1H), 4.48 (s, 1H), 4.06-3.98 (m, 2H), 3.11-3.07 (m, 1H), 1.97-1.91 (m, 2H),

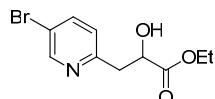
1.05 (t, $J = 7.2$ Hz, 3H), 0.93 (t, $J = 7.2$ Hz, 3H); ^{13}C NMR (100 MHz, CDCl_3): δ 174.0, 161.7, 148.6, 136.6, 123.8, 122.0, 73.6, 60.7, 50.1, 26.0, 14.0, 12.0; HRMS: calcd for $\text{C}_{12}\text{H}_{18}\text{NO}_3$ $[\text{M}+\text{H}]^+$ 224.1287, found 224.1283.

Ethyl 3-(5-ethylpyridin-2-yl)-2-hydroxypropanoate (5f):



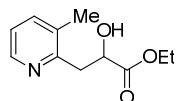
Orange liquid. ^1H NMR (400 MHz, CDCl_3): δ 8.33 (s, 1H), 7.45 (d, $J = 8.0$ Hz, 1H), 7.09 (d, $J = 8.0$ Hz, 1H), 4.61 (q, $J = 3.6$ Hz, 1H), 4.19 (q, $J = 7.2$ Hz, 2H), 3.29-3.10 (m, 2H), 2.61 (q, $J = 7.6$ Hz, 2H), 1.25-1.21 (m, 6H); ^{13}C NMR (100 MHz, CDCl_3): δ 173.7, 155.2, 148.3, 137.4, 136.2, 123.4, 70.7, 61.2, 40.1, 25.7, 15.2, 14.1; HRMS: calcd for $\text{C}_{12}\text{H}_{18}\text{NO}_3$ $[\text{M}+\text{H}]^+$ 224.1287, found 224.1289.

Ethyl 3-(5-bromopyridin-2-yl)-2-hydroxypropanoate (5g):



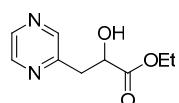
Orange liquid. ^1H NMR (400 MHz, CDCl_3): δ 8.57 (d, $J = 1.6$ Hz, 1H), 7.75 (dd, $J_1 = 2.4$ Hz, $J_2 = 2.0$ Hz, 1H), 7.11 (d, $J = 8.4$ Hz, 1H), 4.61 (d, $J = 3.6$ Hz, 1H), 4.22 (q, $J = 7.2$ Hz, 2H), 4.10 (d, $J = 5.6$ Hz, 1H), 3.29-3.10 (m, 2H), 1.28 (t, $J = 7.2$ Hz, 3H); ^{13}C NMR (100 MHz, CDCl_3): δ 173.6, 156.2, 150.0, 139.1, 125.3, 118.8, 70.1, 61.6, 40.6, 14.1; HRMS: calcd for $\text{C}_{10}\text{H}_{12}\text{BrNNaO}_3$ $[\text{M}+\text{Na}]^+$ 295.9898, found 295.9895.

Ethyl 2-hydroxy-3-(3-methylpyridin-2-yl)propanoate (5h):



Orange liquid. ^1H NMR (400 MHz, CDCl_3): δ 8.30 (d, $J = 4.4$ Hz, 1H), 7.44 (d, $J = 7.2$ Hz, 1H), 7.09-7.06 (m, 1H), 4.70 (t, $J = 5.2$ Hz, 1H), 4.22-4.16 (m, 2H), 3.22 (d, $J = 5.2$ Hz, 2H), 2.29 (s, 3H), 1.22 (t, $J = 7.2$ Hz, 3H); ^{13}C NMR (100 MHz, CDCl_3): δ 173.8, 156.7, 145.7, 137.9, 131.8, 121.7, 70.2, 61.1, 36.7, 18.6, 14.1; HRMS: calcd for $\text{C}_{11}\text{H}_{16}\text{NO}_3$ $[\text{M}+\text{H}]^+$ 210.1130, found 210.1135.

Ethyl 2-hydroxy-3-(pyrazin-2-yl)propanoate (5i**):**



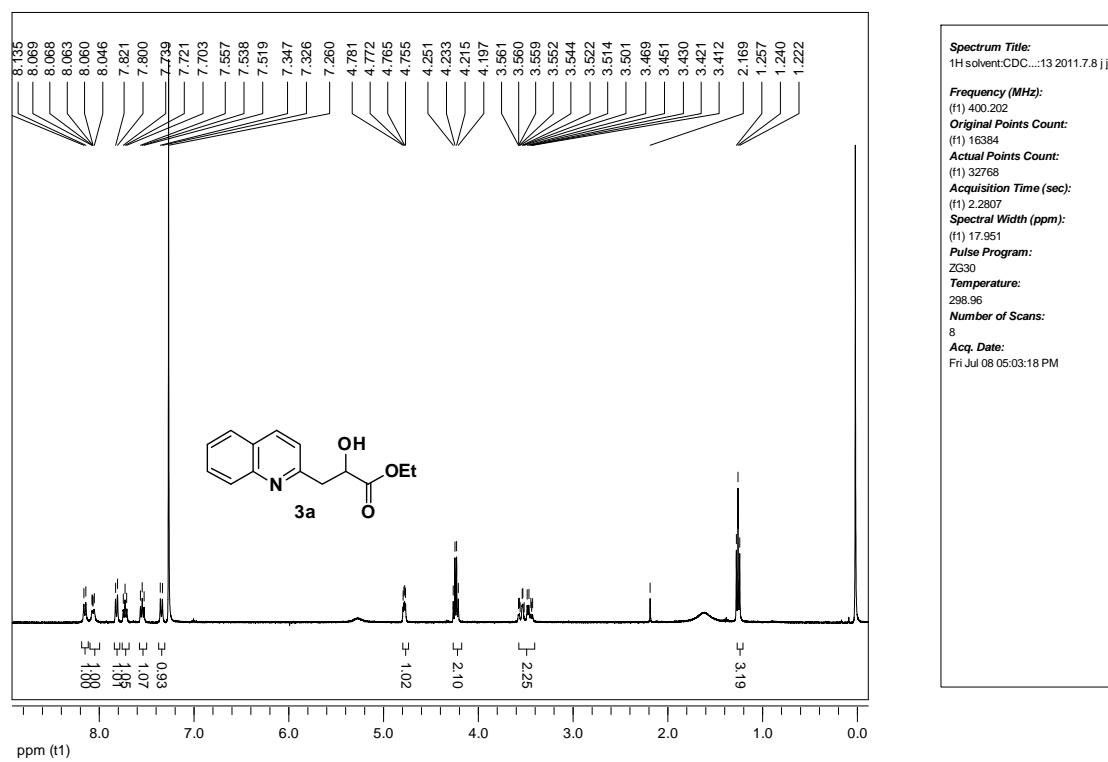
Yellow liquid. ^1H NMR (400 MHz, CDCl_3): δ 8.64-8.44 (m, 3H), 4.65 (s, 1H), 4.24 (q, $J = 7.2$ Hz, 2H), 3.68 (s, 1H), 3.36-3.18 (m, 2H), 1.26 (t, $J = 7.2$ Hz, 3H); ^{13}C NMR (100 MHz, CDCl_3): δ 173.6, 69.7, 61.7, 39.0, 14.0; HRMS: calcd for $\text{C}_9\text{H}_{12}\text{N}_2\text{NaO}_3$ $[\text{M}+\text{Na}]^+$ 219.0746, found 219.0750.

References:

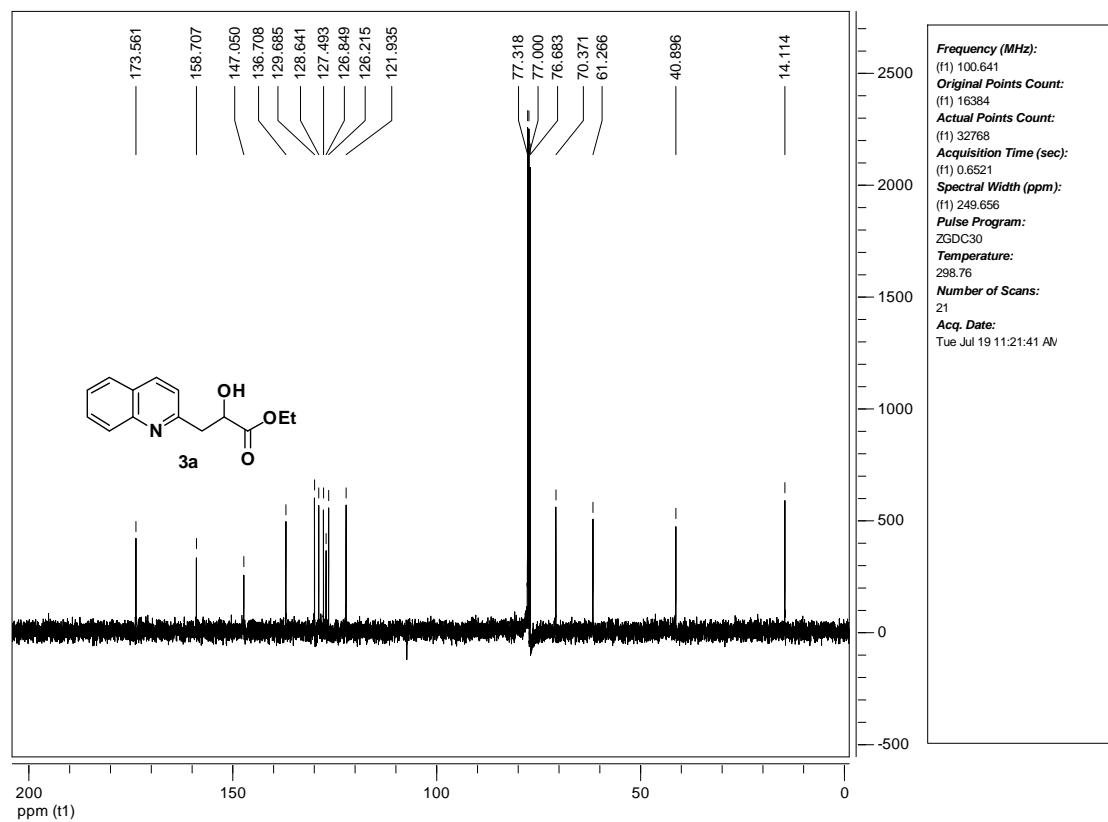
1. M. Matsugi, F. Tabusa and J. Minamikawa, *Tetrahedron Lett.*, 2000, **41**, 8523–8525.

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

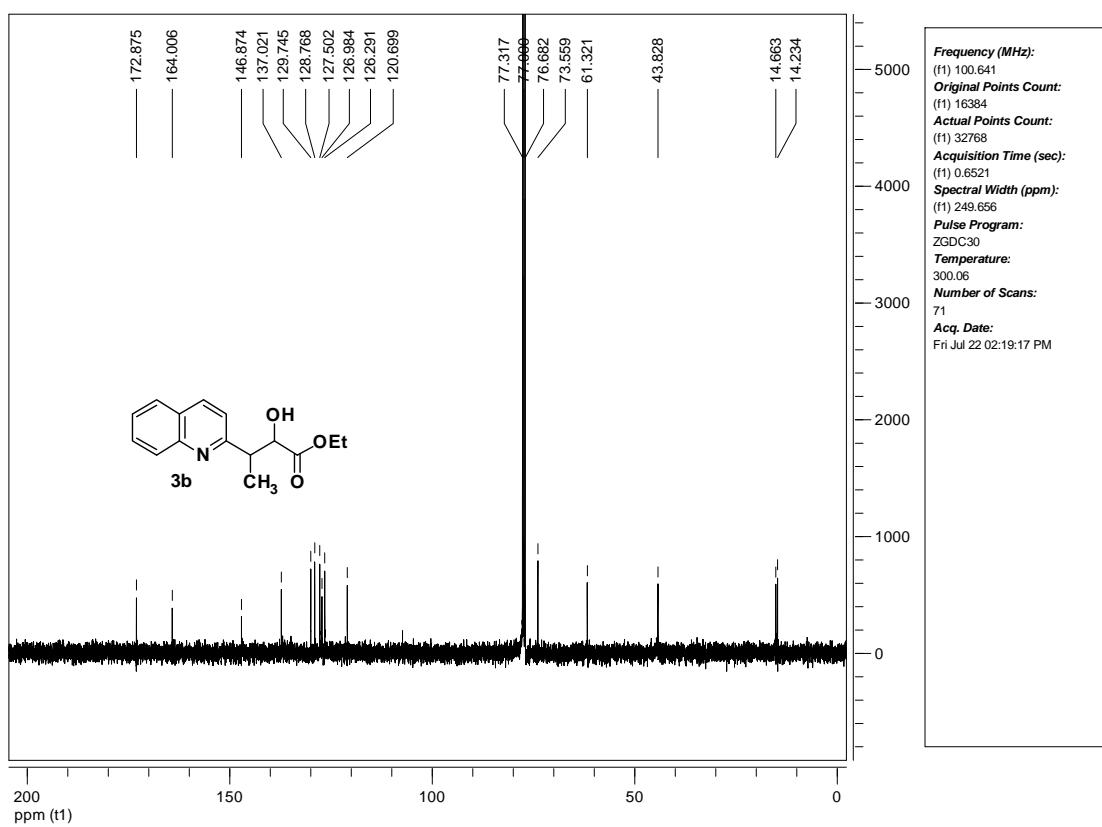
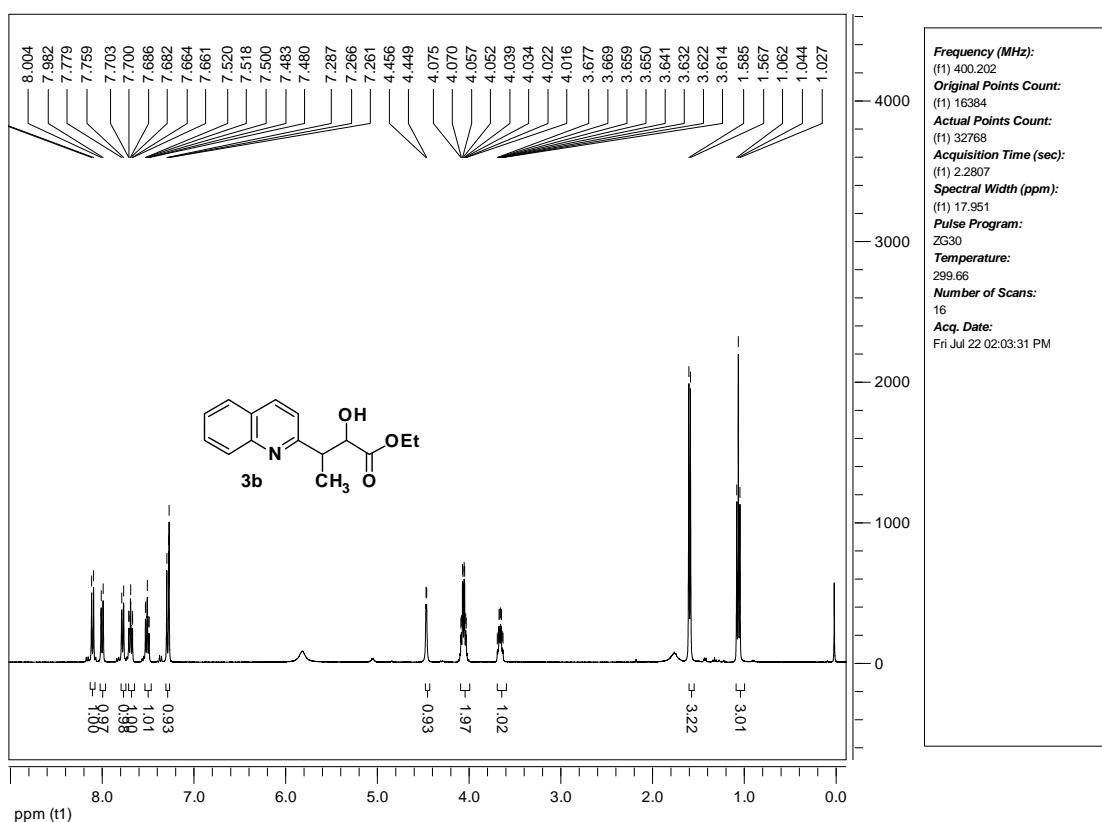
^1H NMR Spectrum for 3a



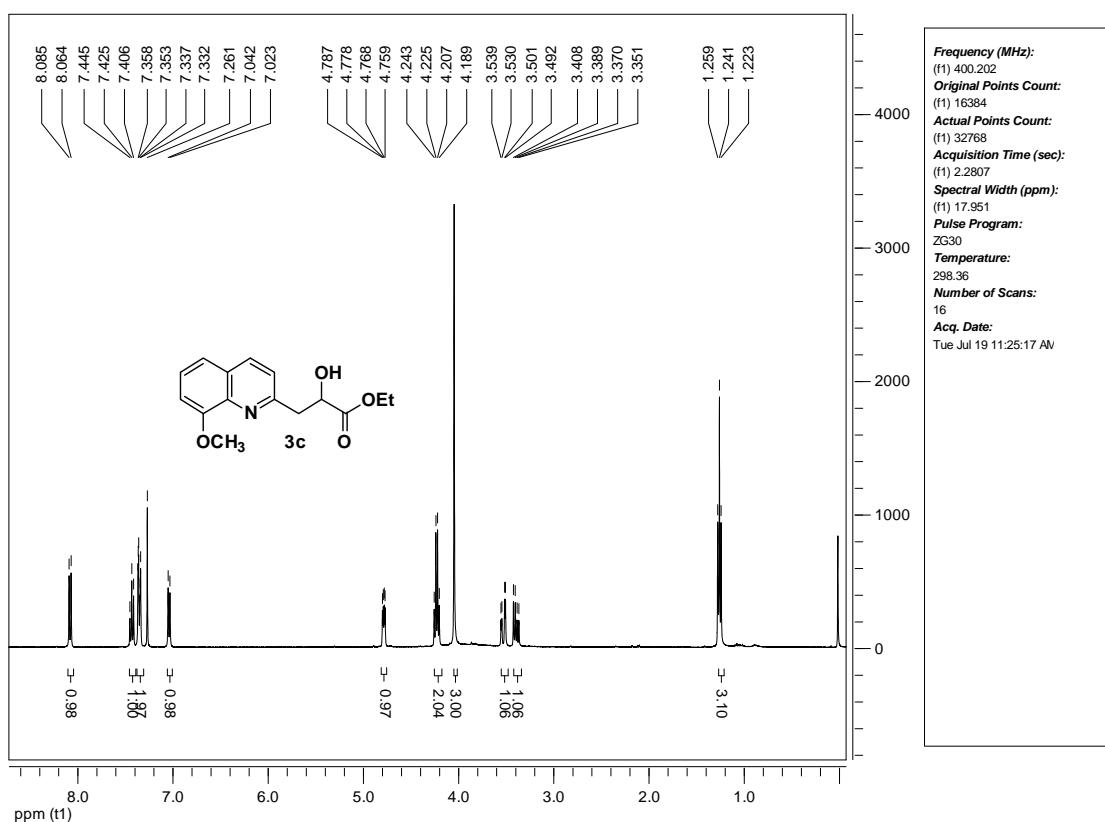
^{13}C NMR Spectrum for 3a



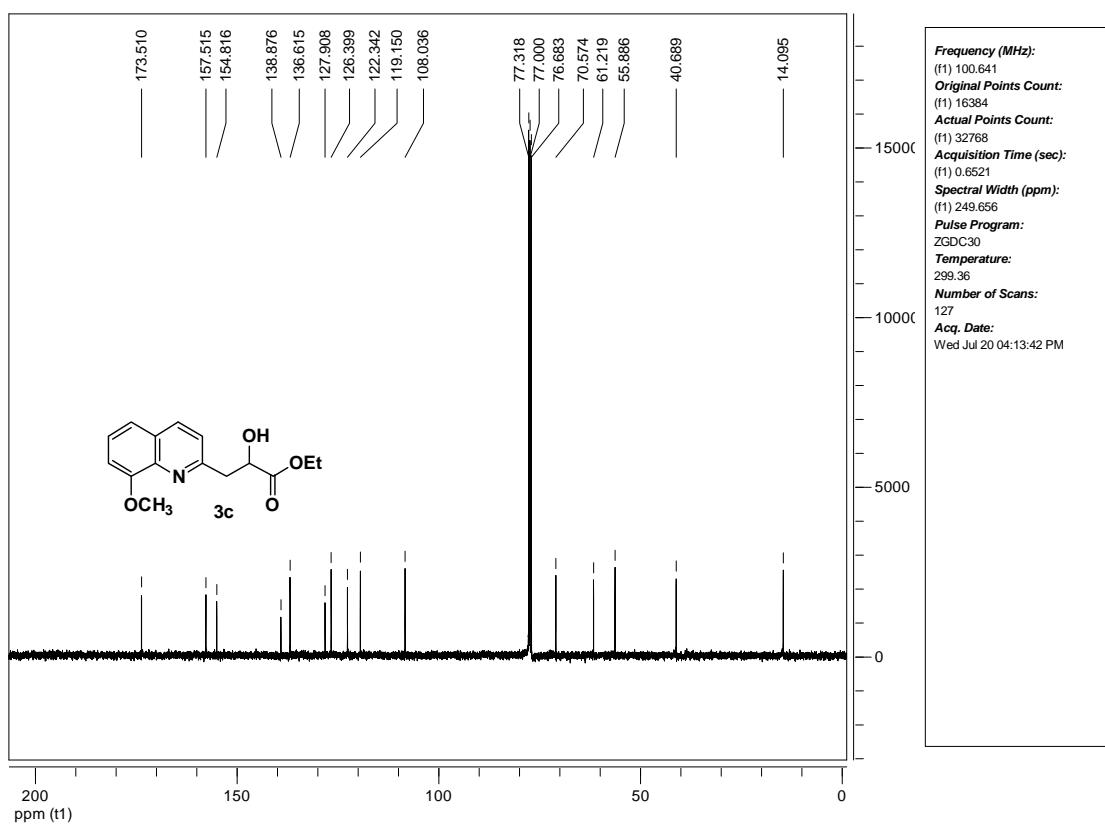
¹H NMR Spectrum for 3b



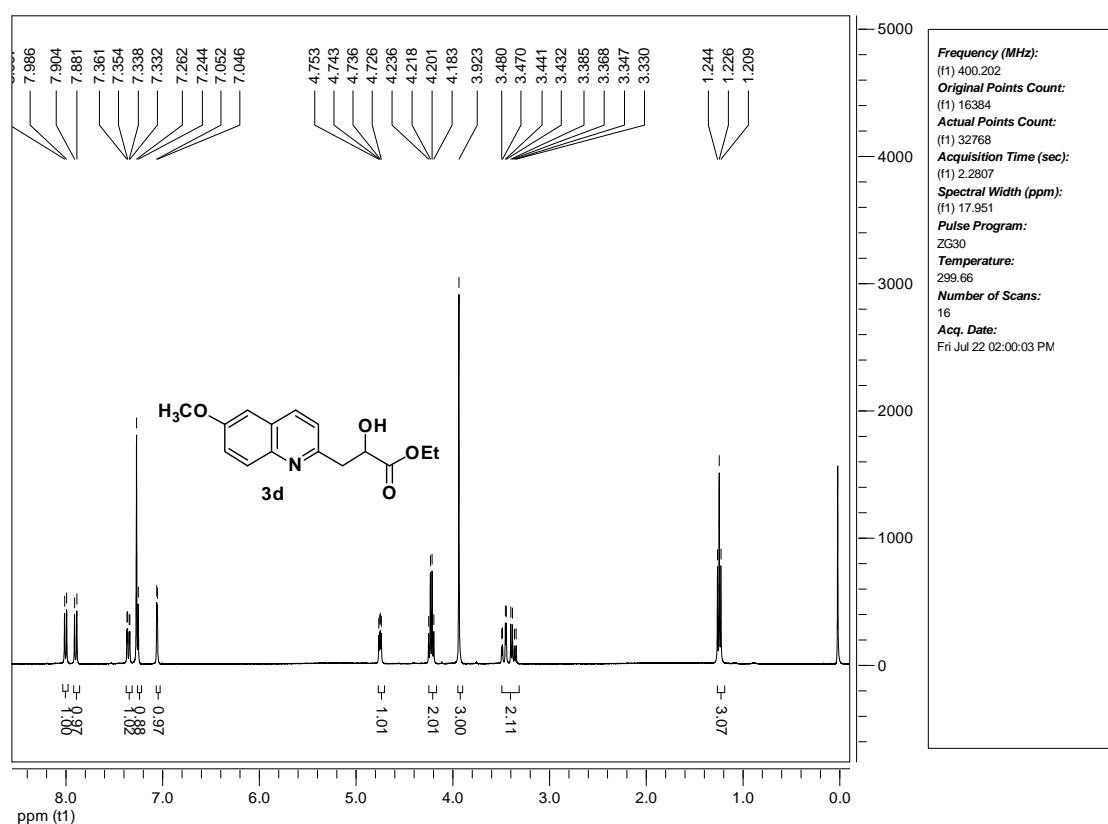
¹H NMR Spectrum for 3c



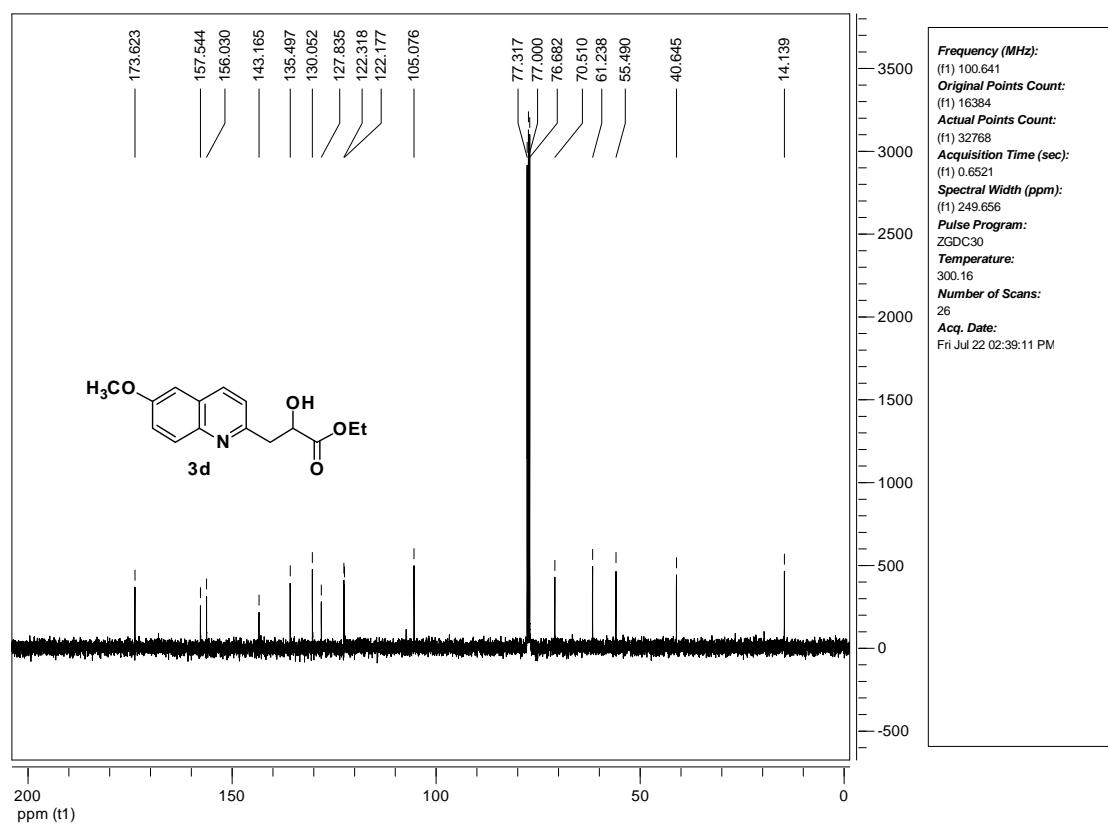
¹³C NMR Spectrum for 3c



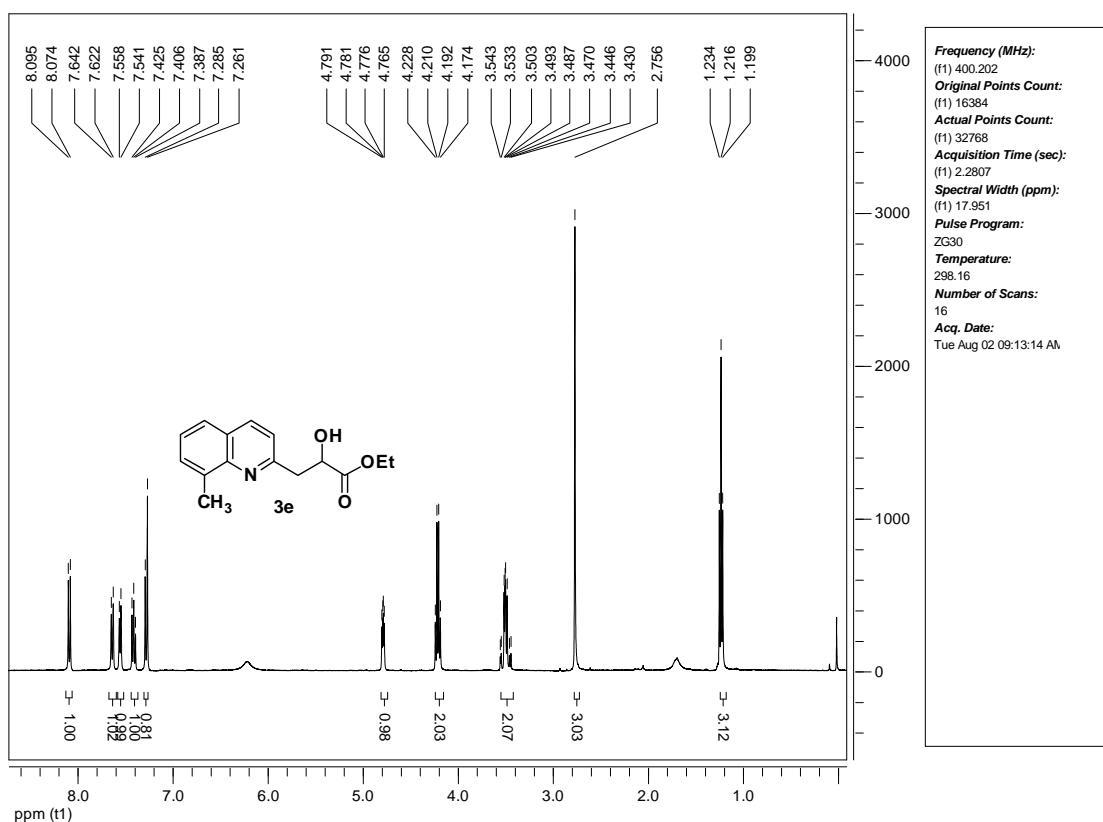
¹H NMR Spectrum for 3d



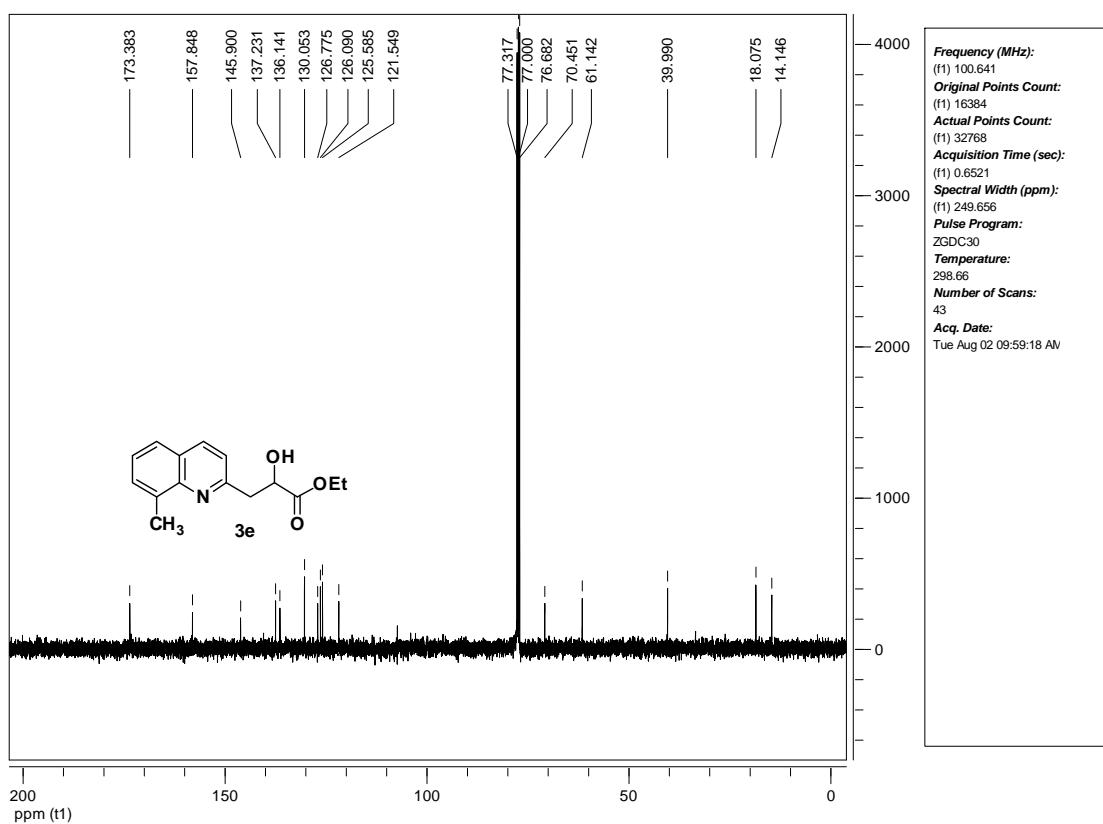
¹³C NMR Spectrum for 3d



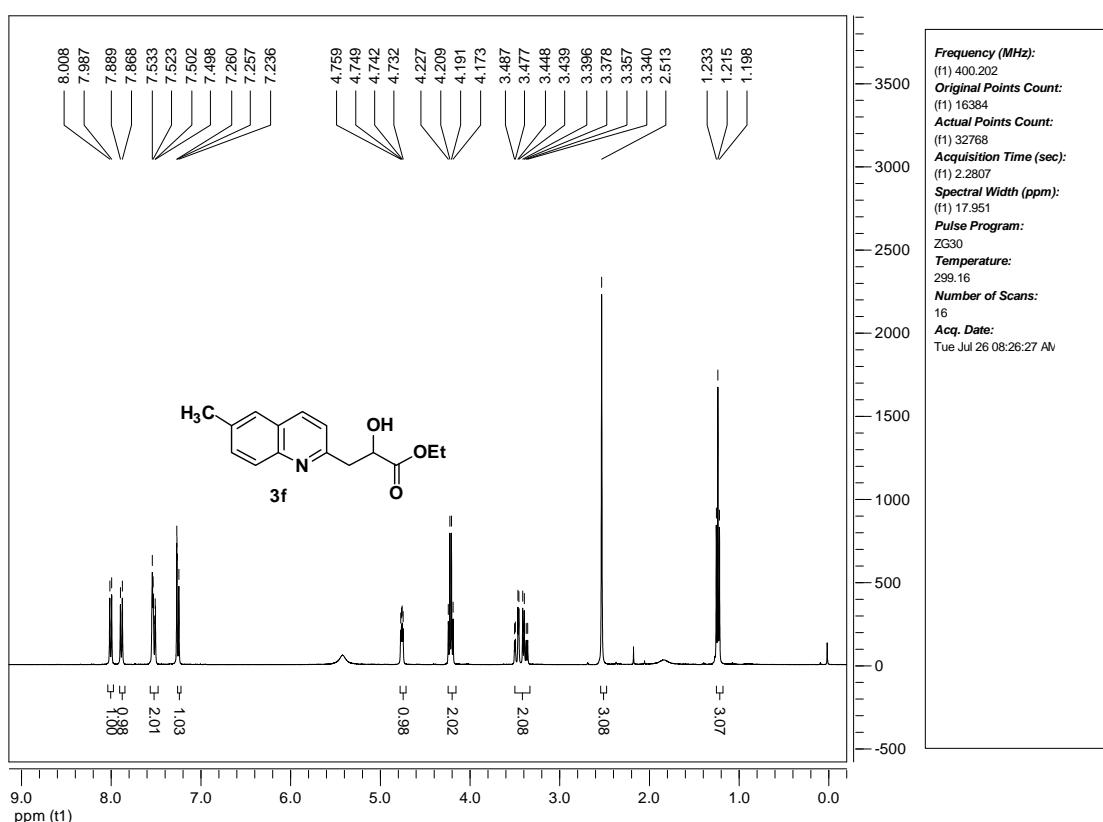
¹H NMR Spectrum for 3e



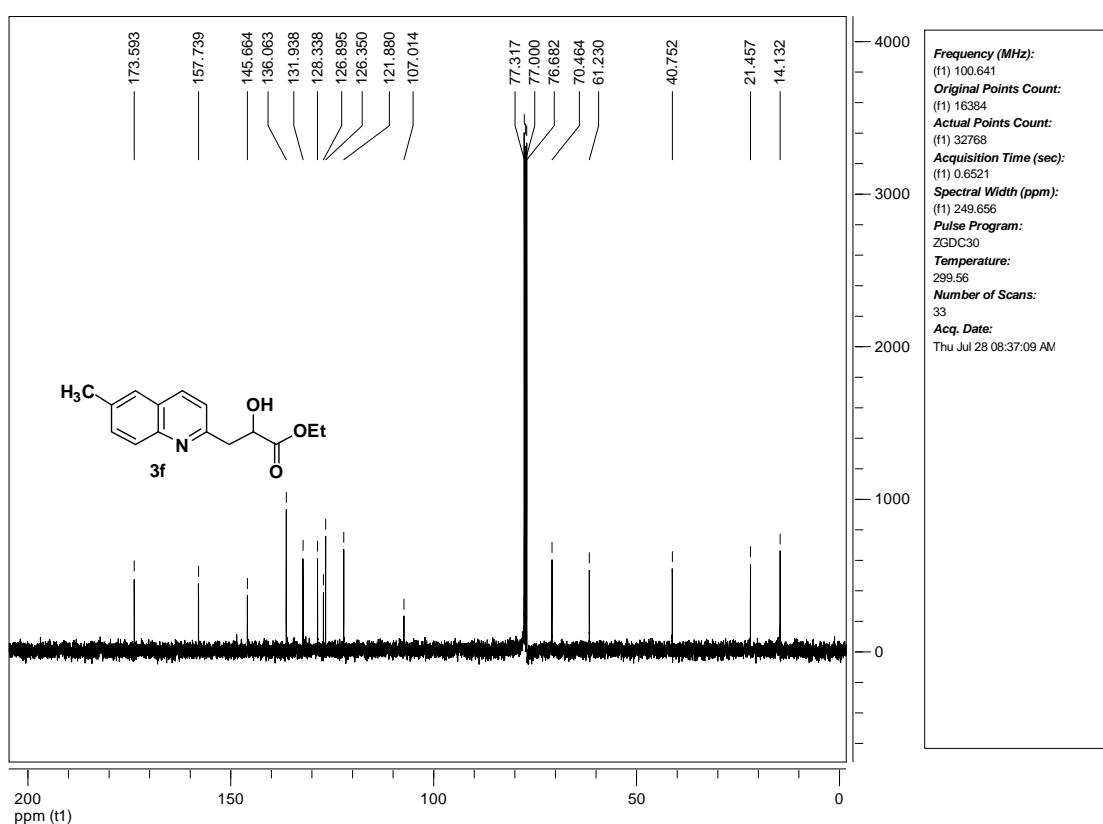
¹³C NMR Spectrum for 3e



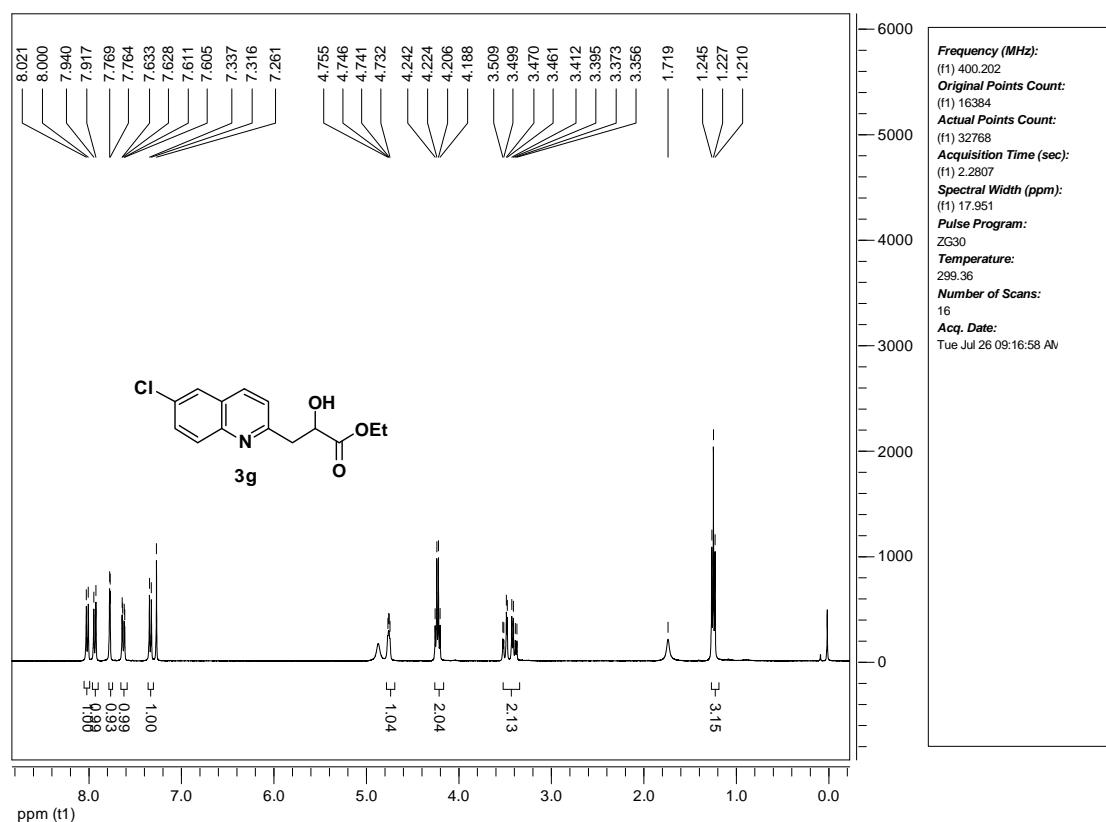
¹H NMR Spectrum for 3f



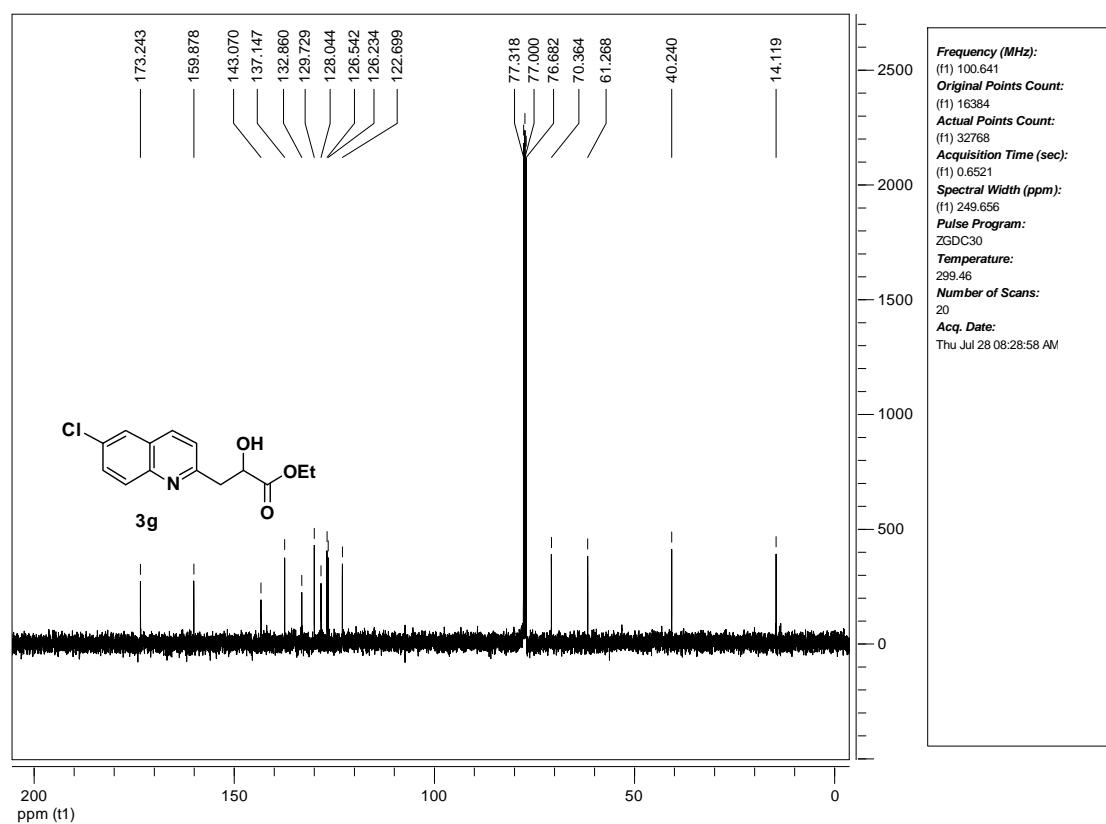
¹³C NMR Spectrum for 3f



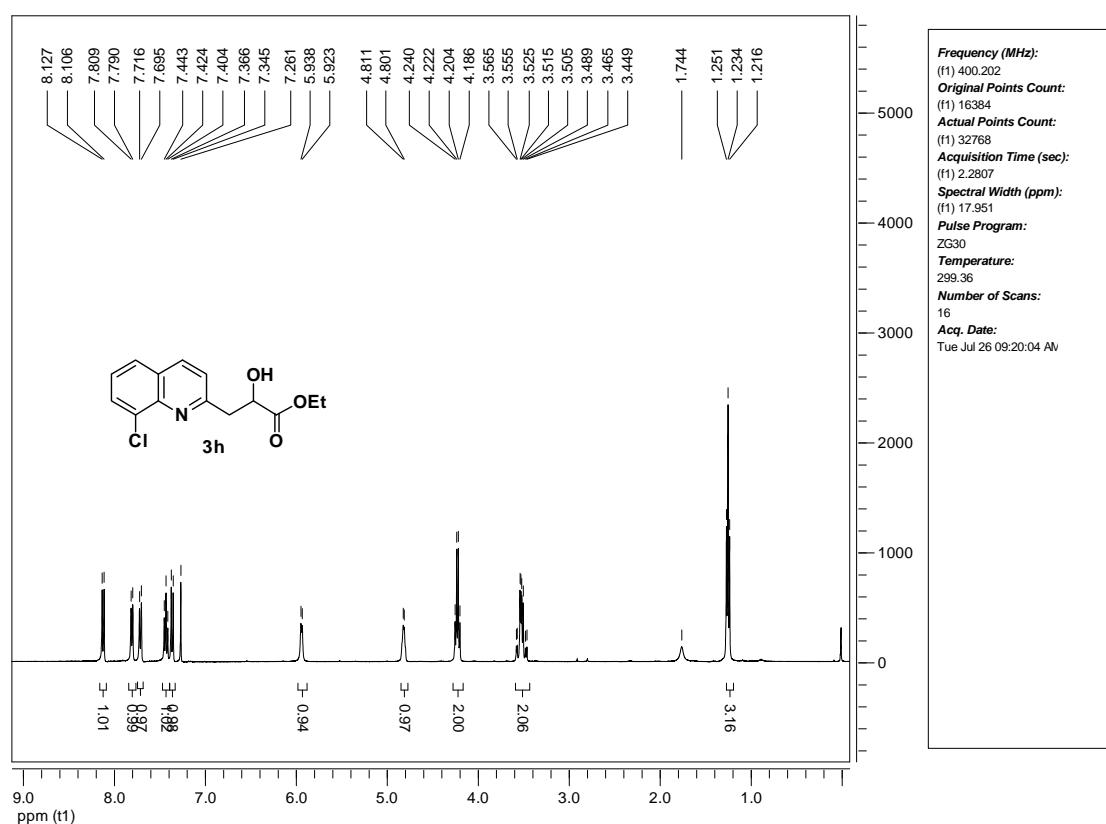
¹H NMR Spectrum for 3g



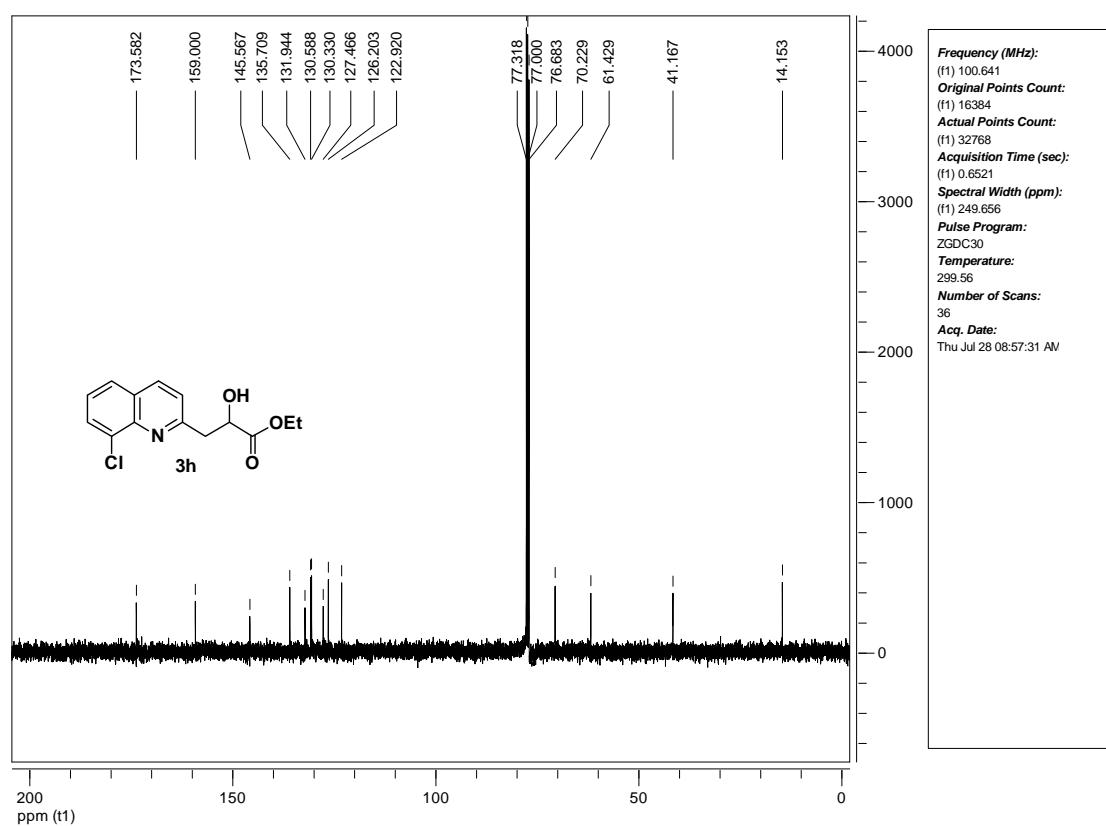
¹³C NMR Spectrum for 3g



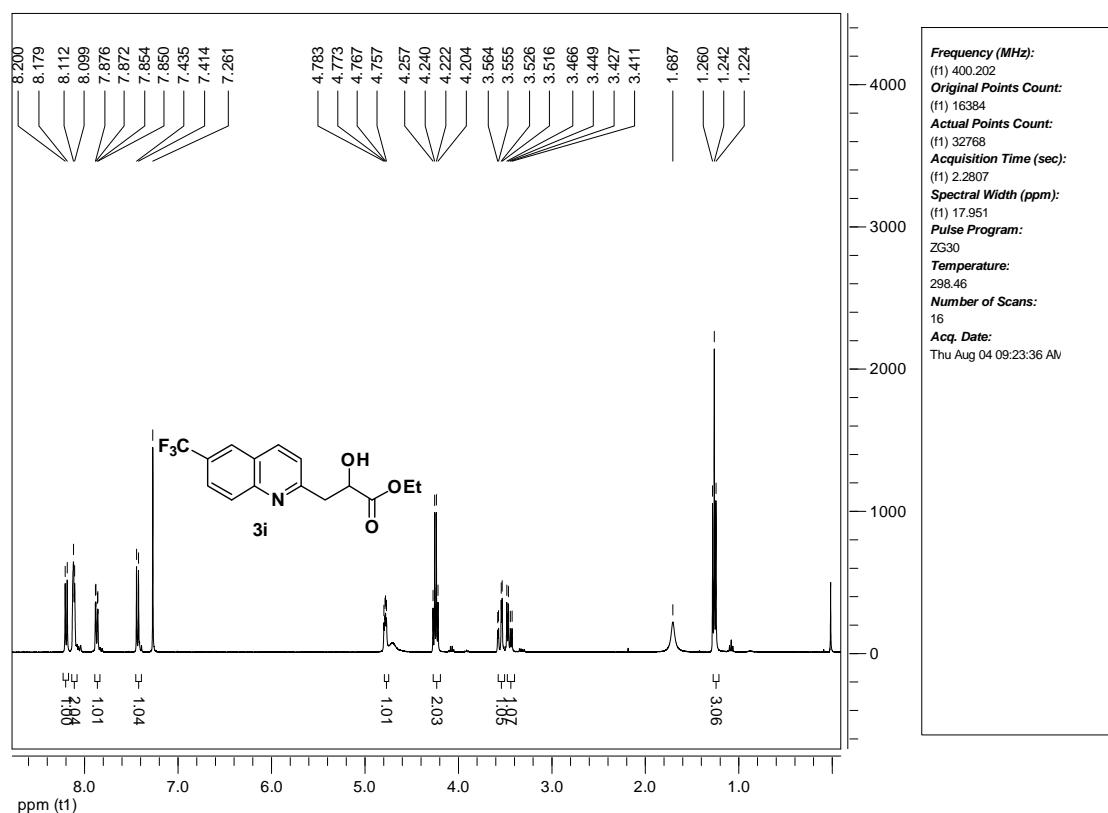
¹H NMR Spectrum for 3h



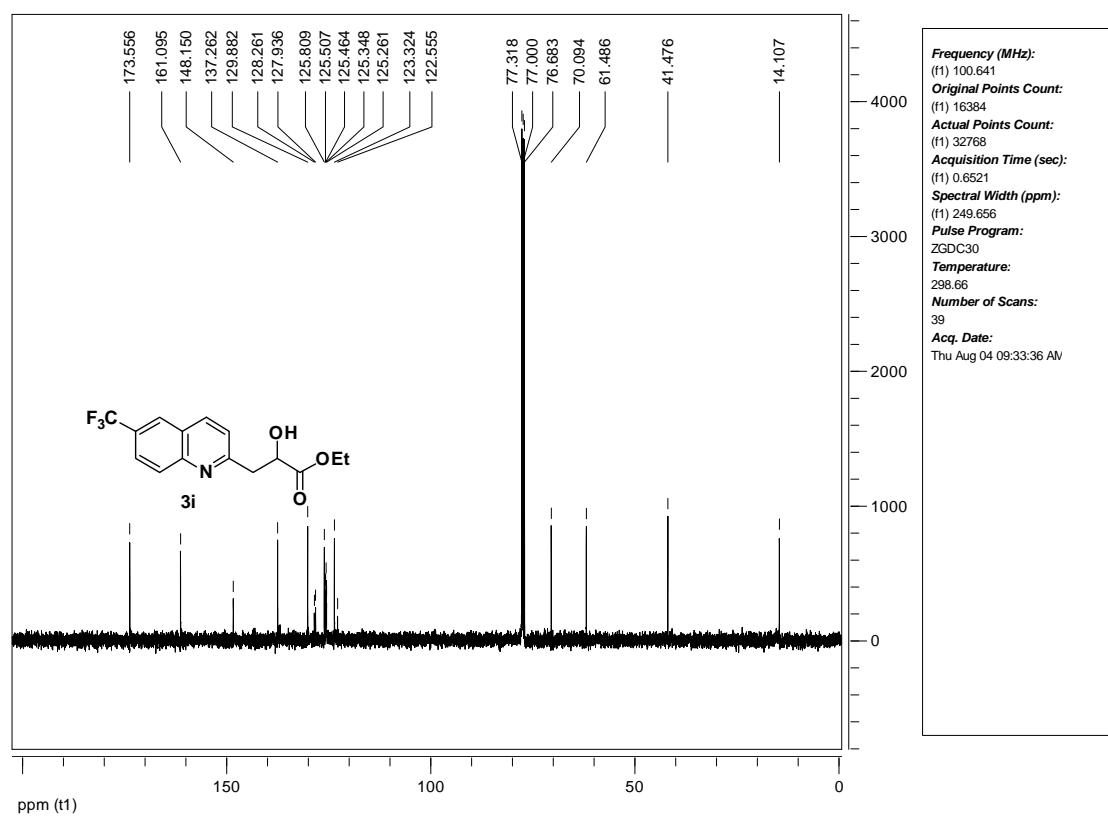
¹³C NMR Spectrum for 3h



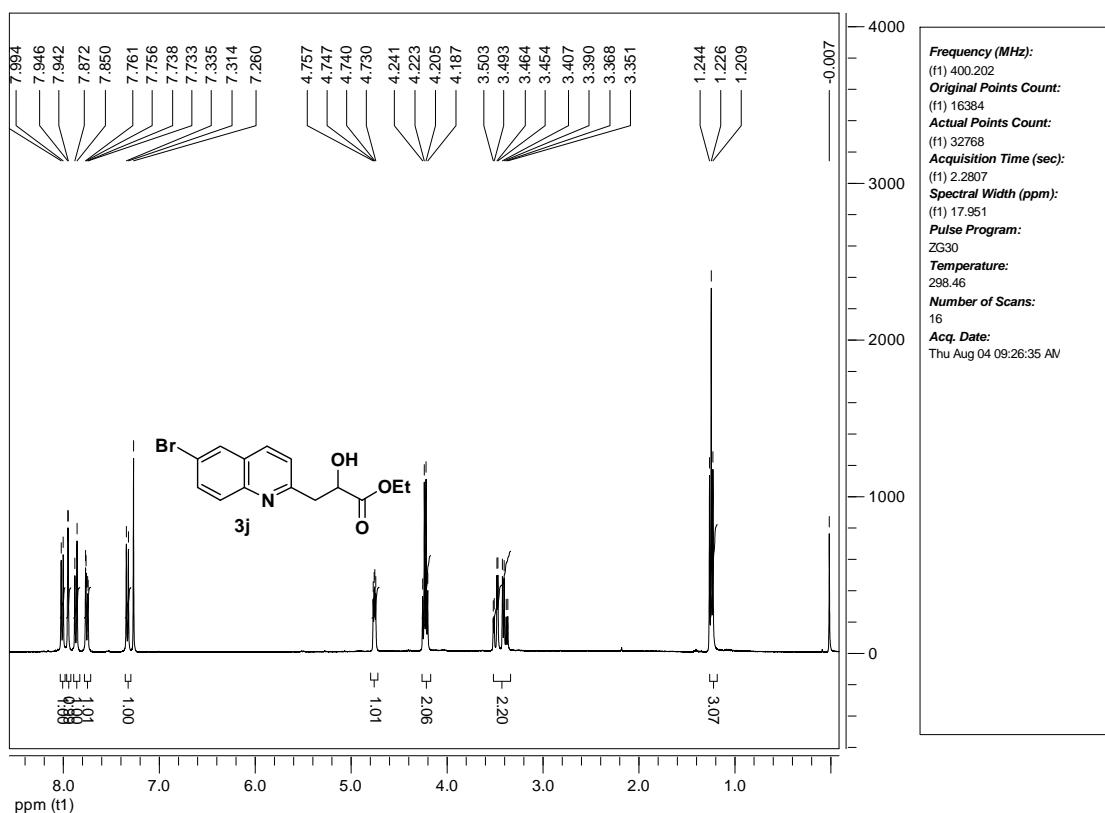
¹H NMR Spectrum for 3i



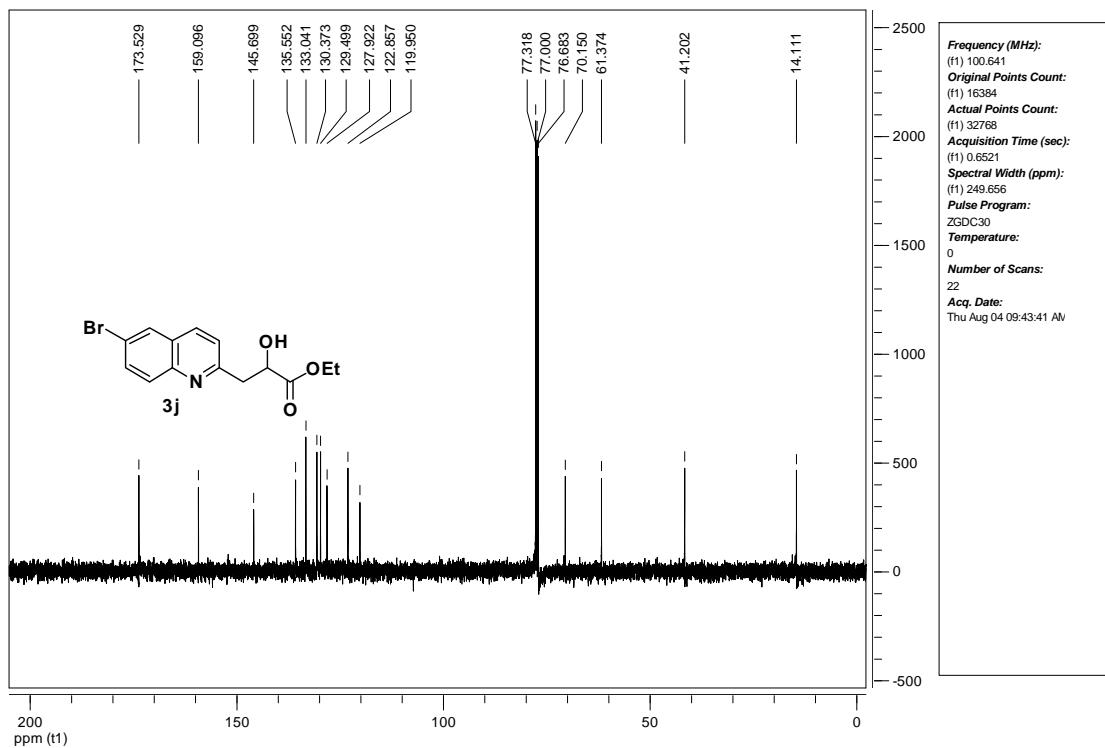
¹³C NMR Spectrum for 3i



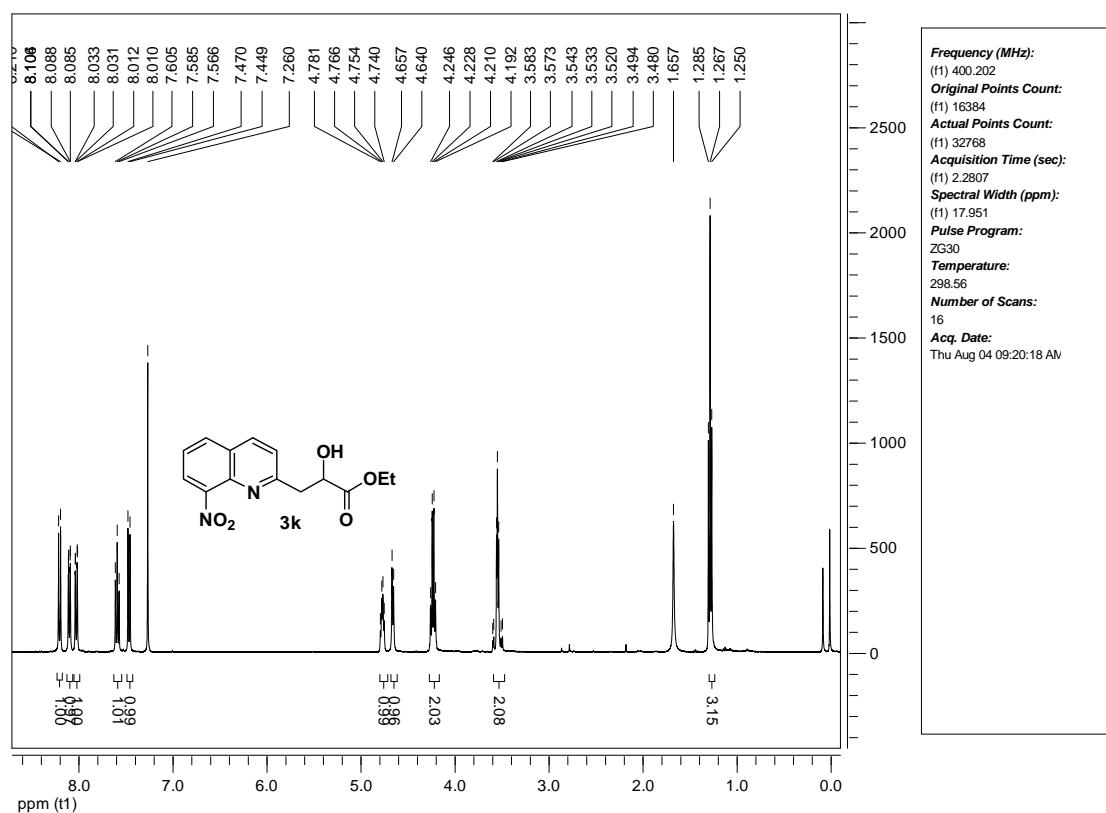
¹H NMR Spectrum for 3j



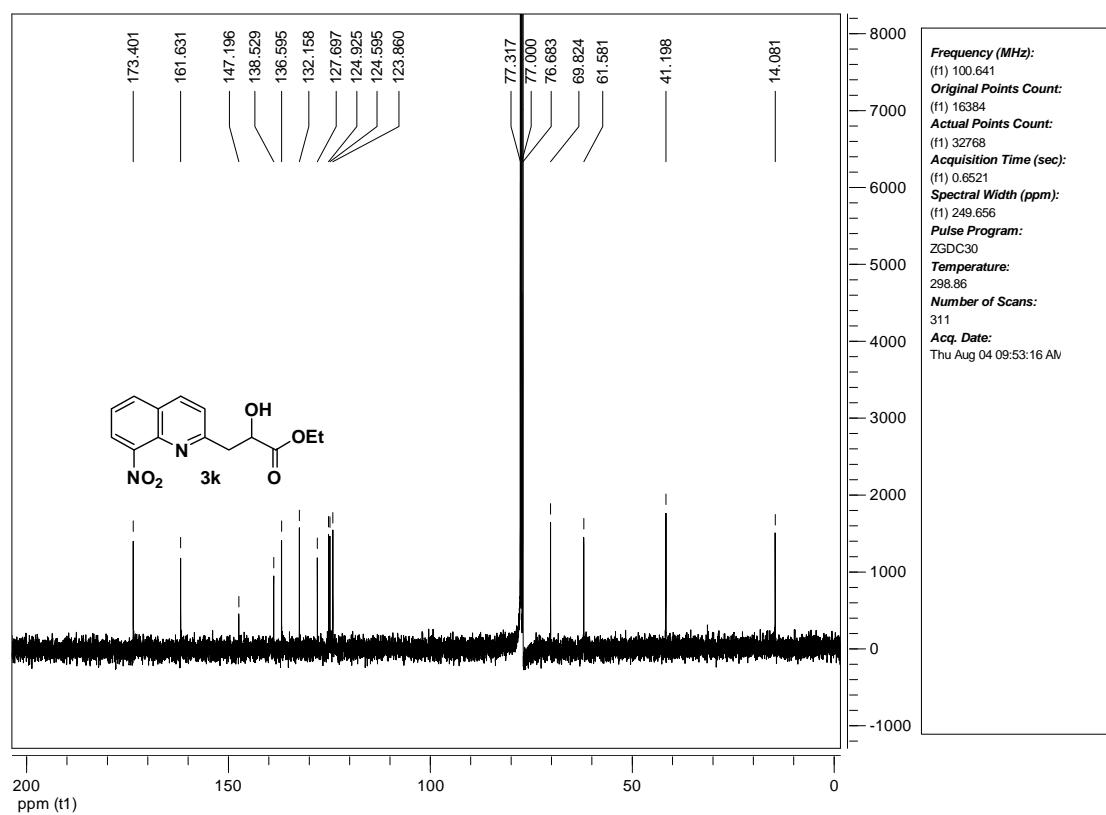
¹³C NMR Spectrum for 3j



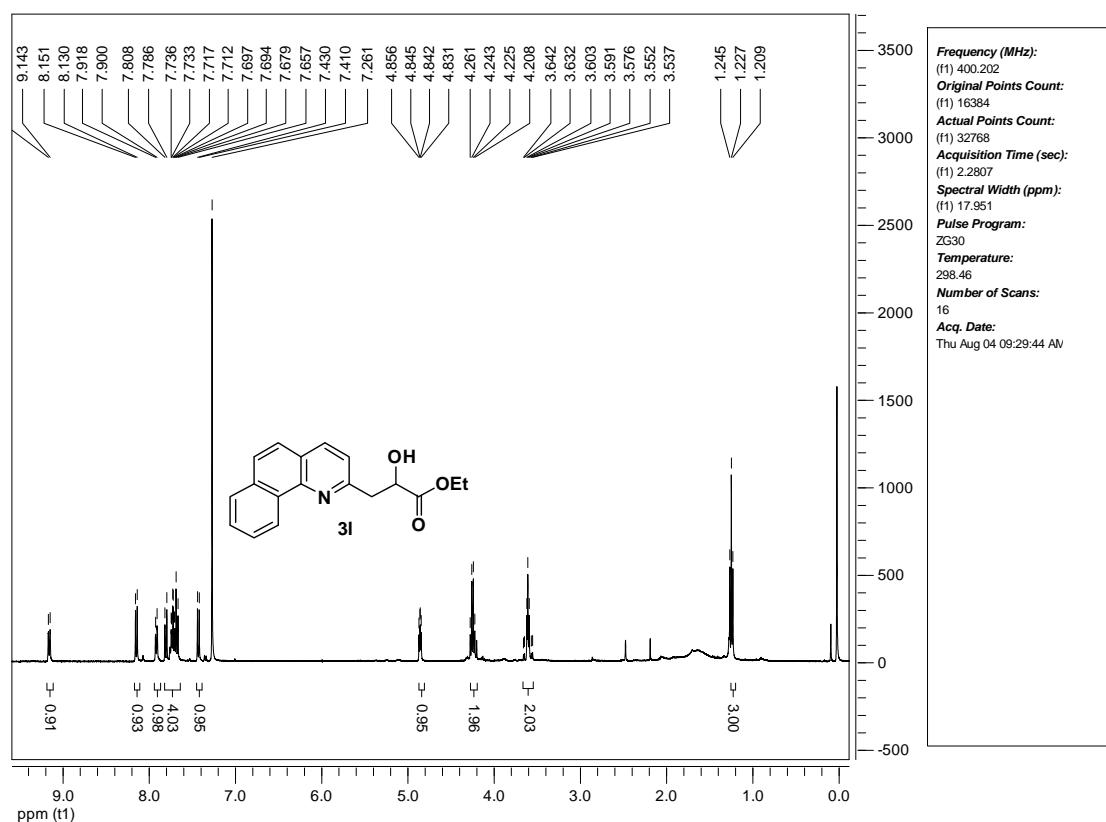
¹H NMR Spectrum for 3k



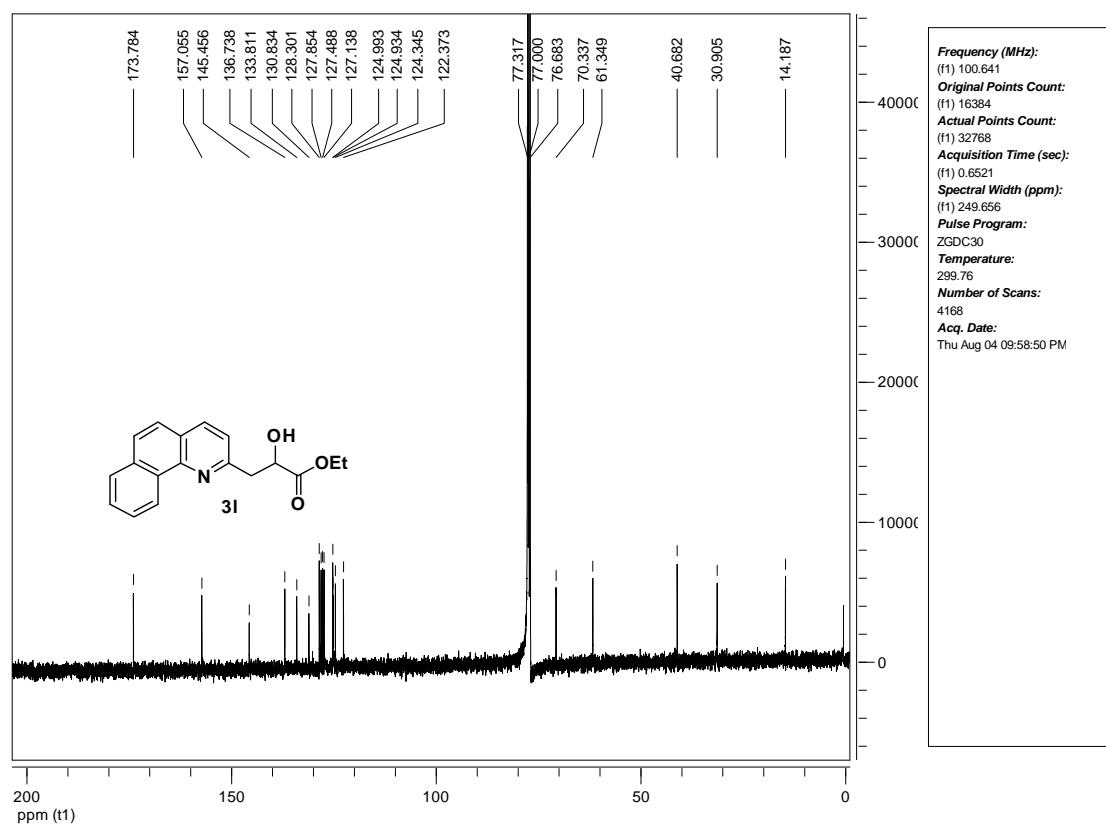
¹³C NMR Spectrum for 3k



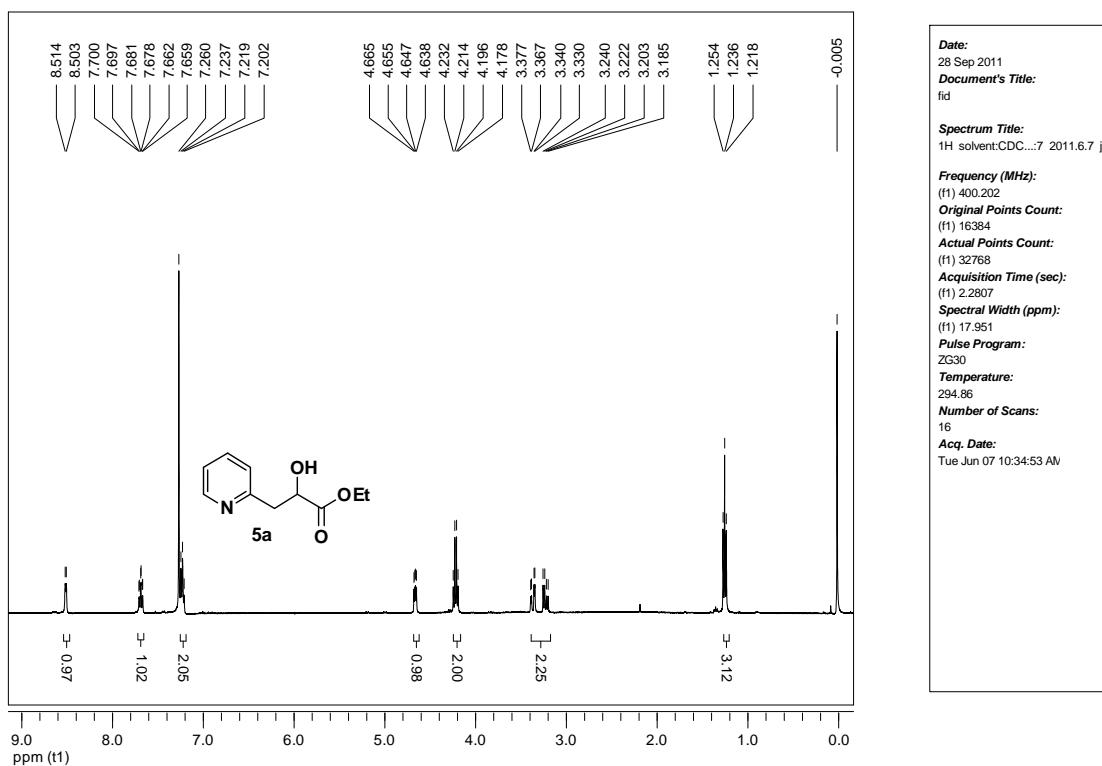
¹H NMR Spectrum for 3l



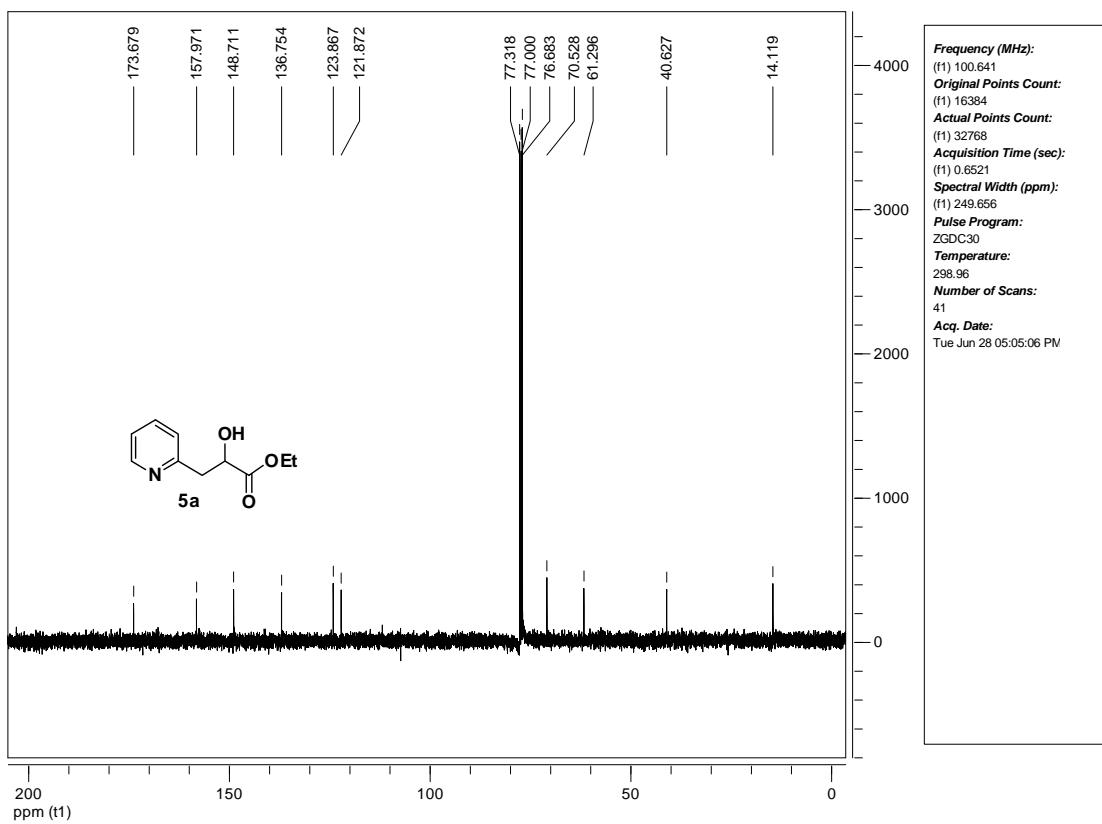
¹³C NMR Spectrum for 3l



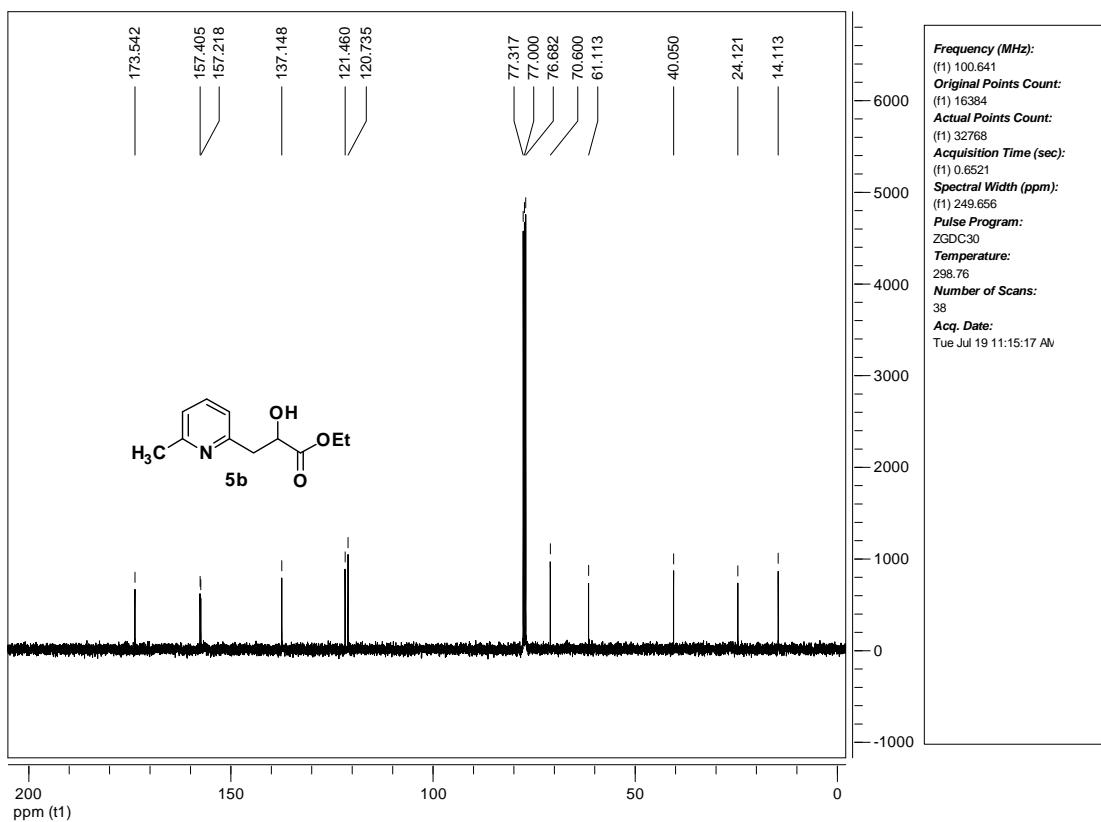
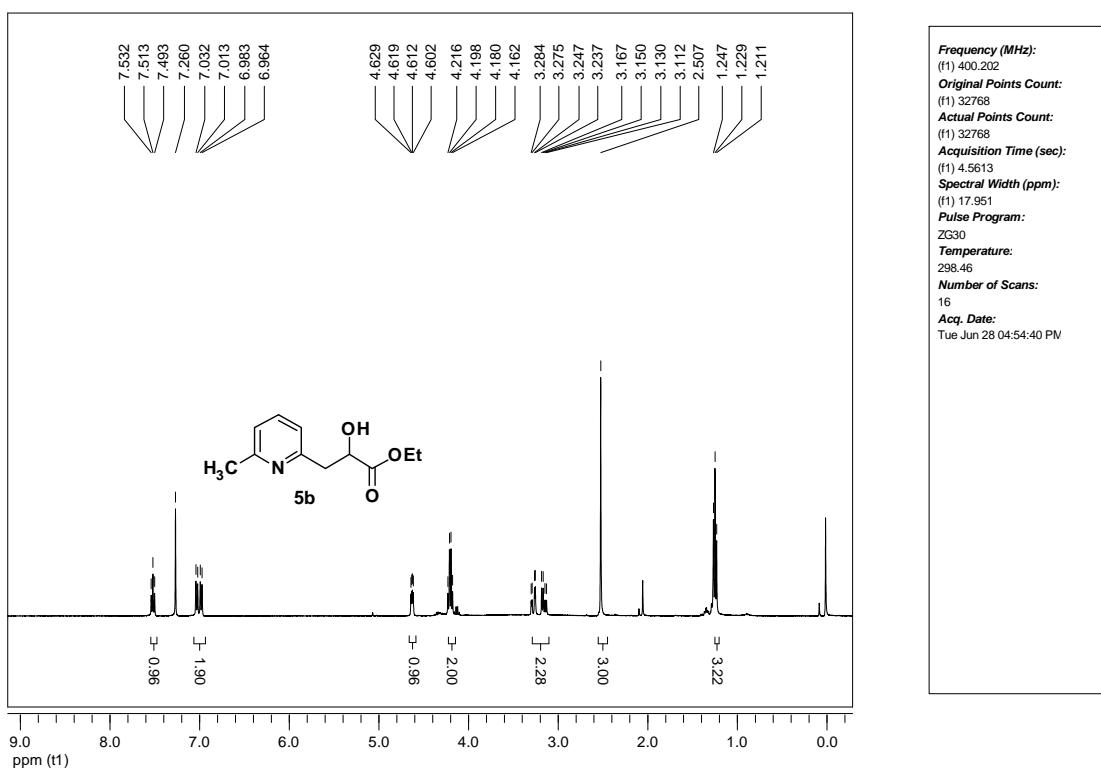
¹H NMR Spectrum for 5a



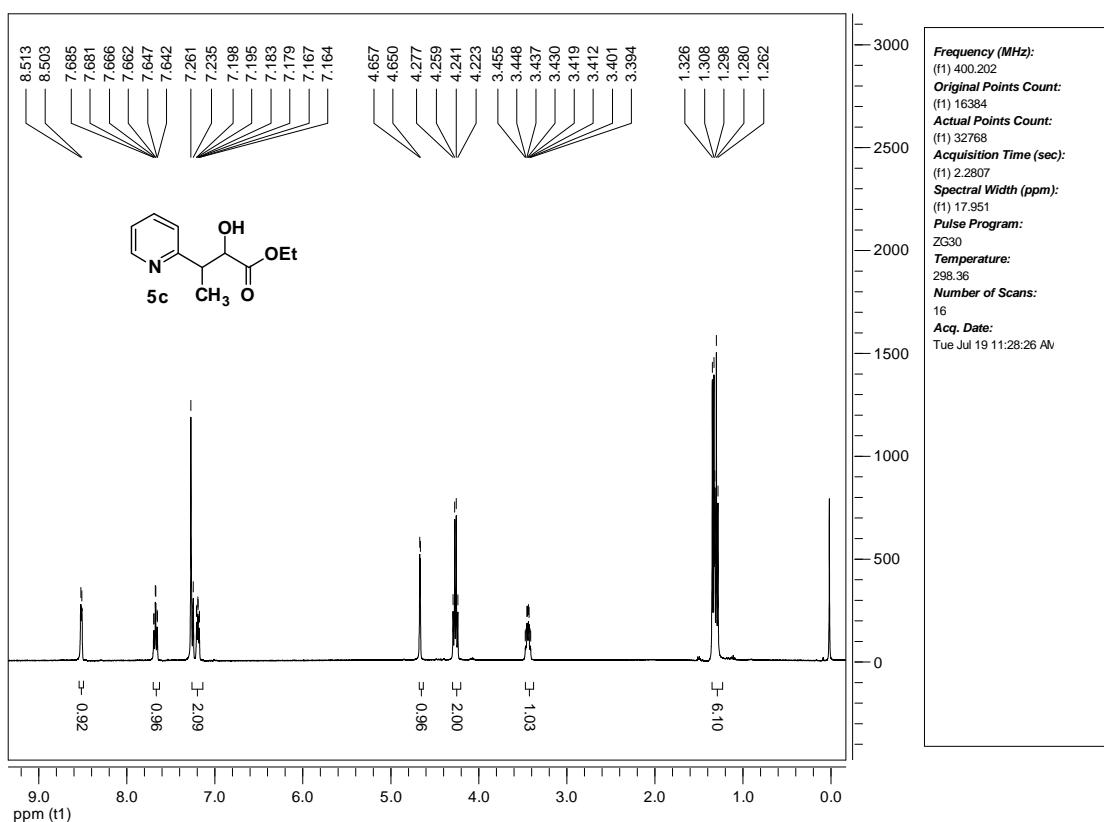
¹³C NMR Spectrum for 5a



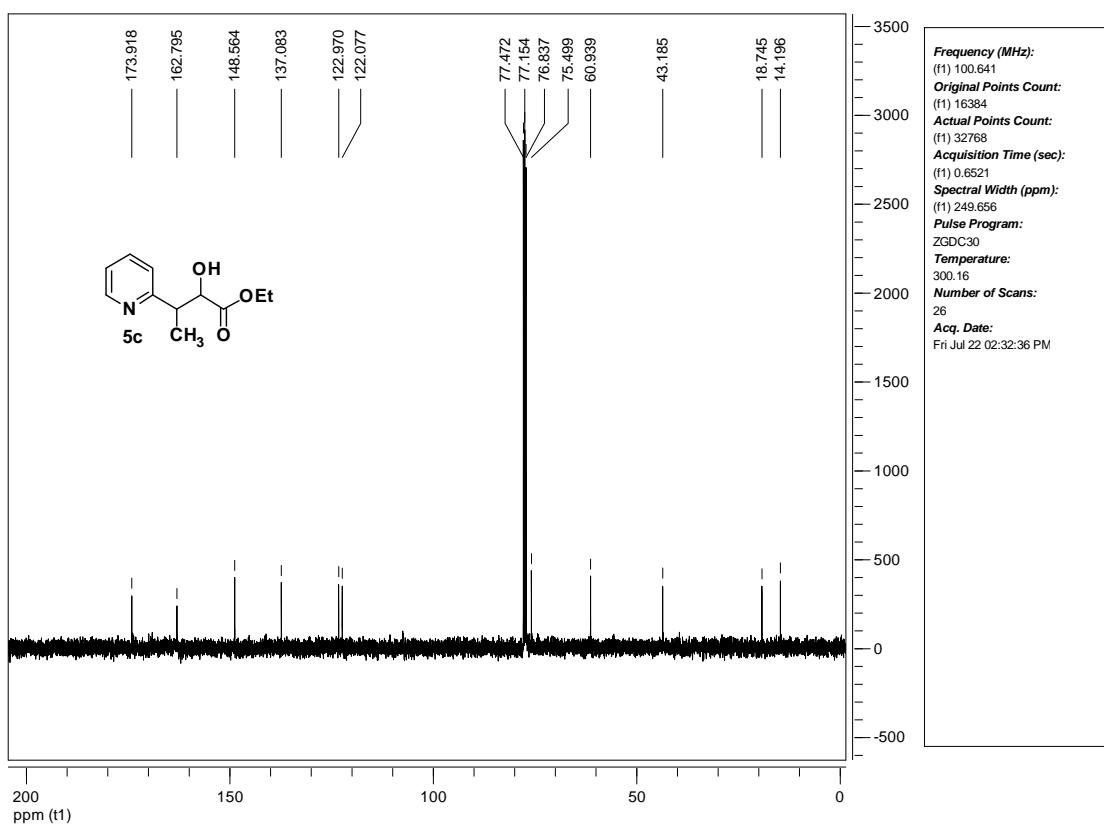
¹H NMR Spectrum for 5b



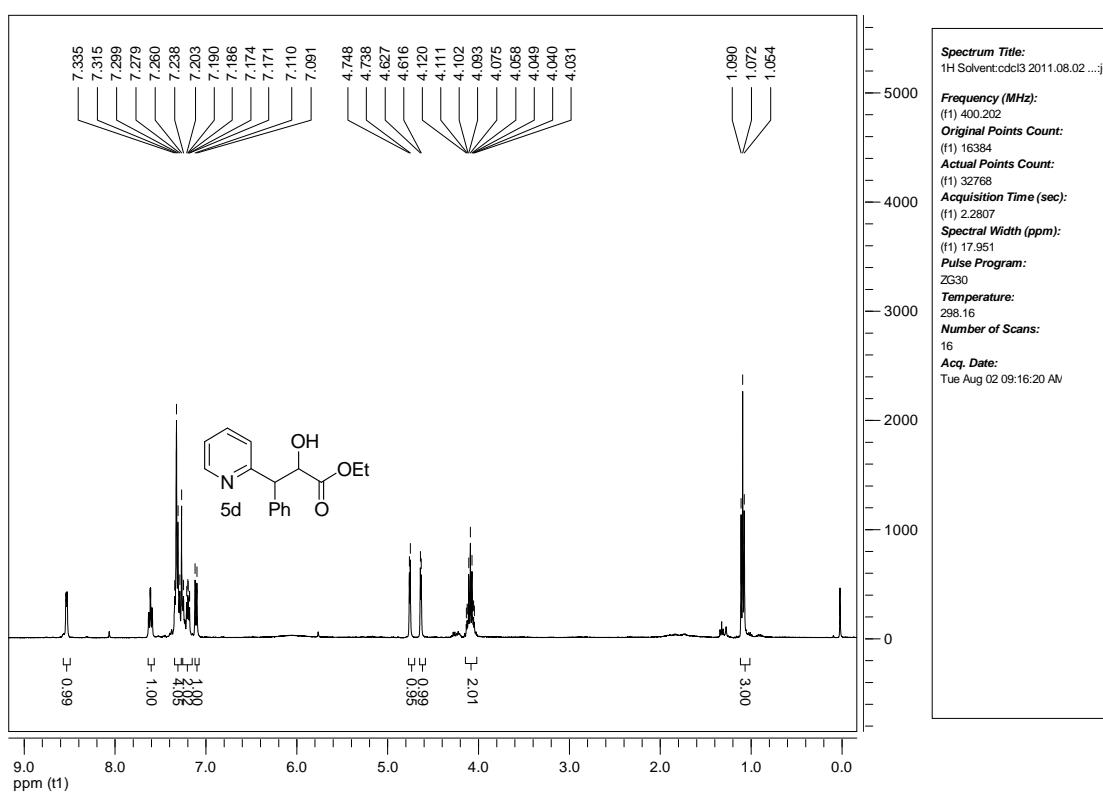
¹H NMR Spectrum for 5c



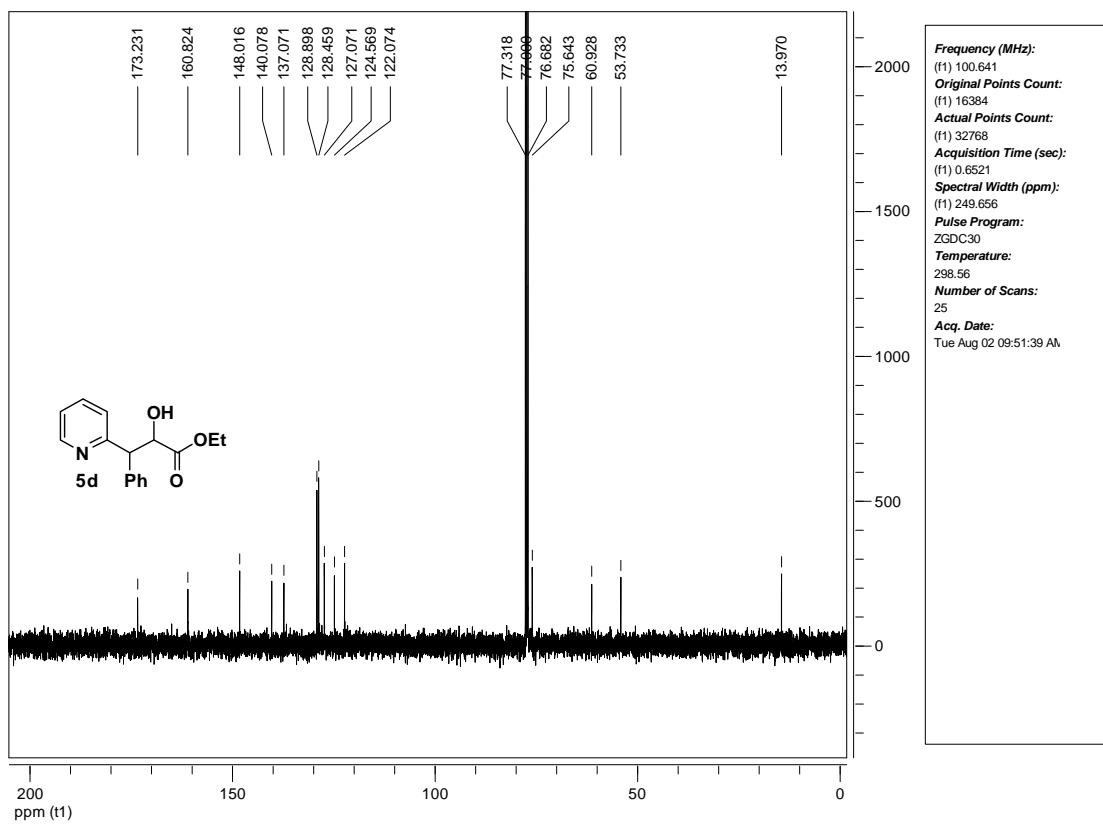
¹³C NMR Spectrum for 5c



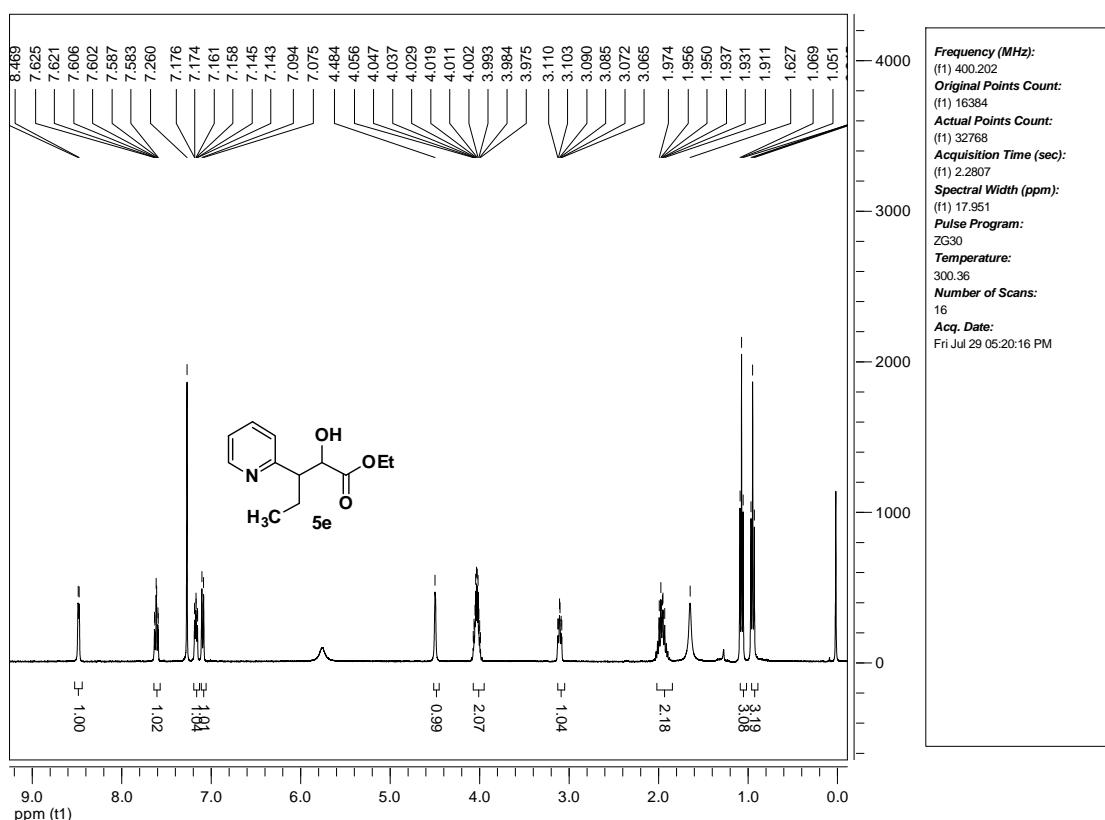
¹H NMR Spectrum for 5d



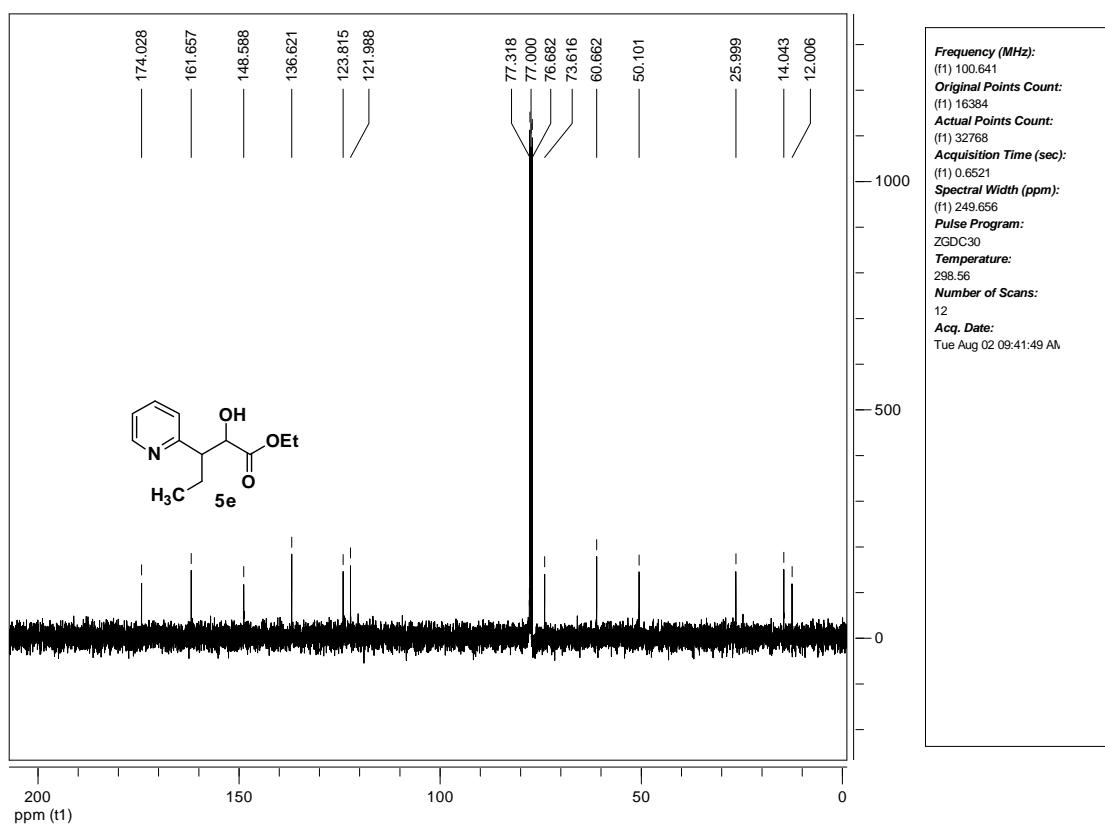
¹³C NMR Spectrum for 5d



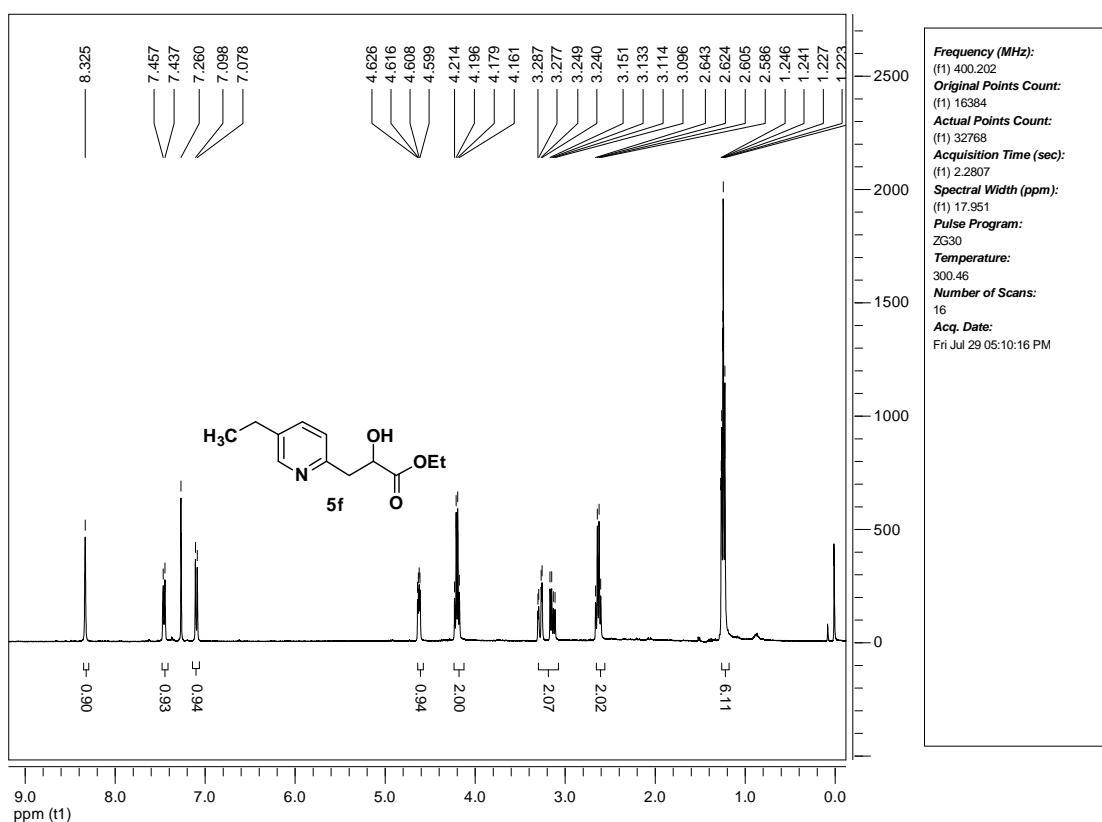
¹H NMR Spectrum for 5e



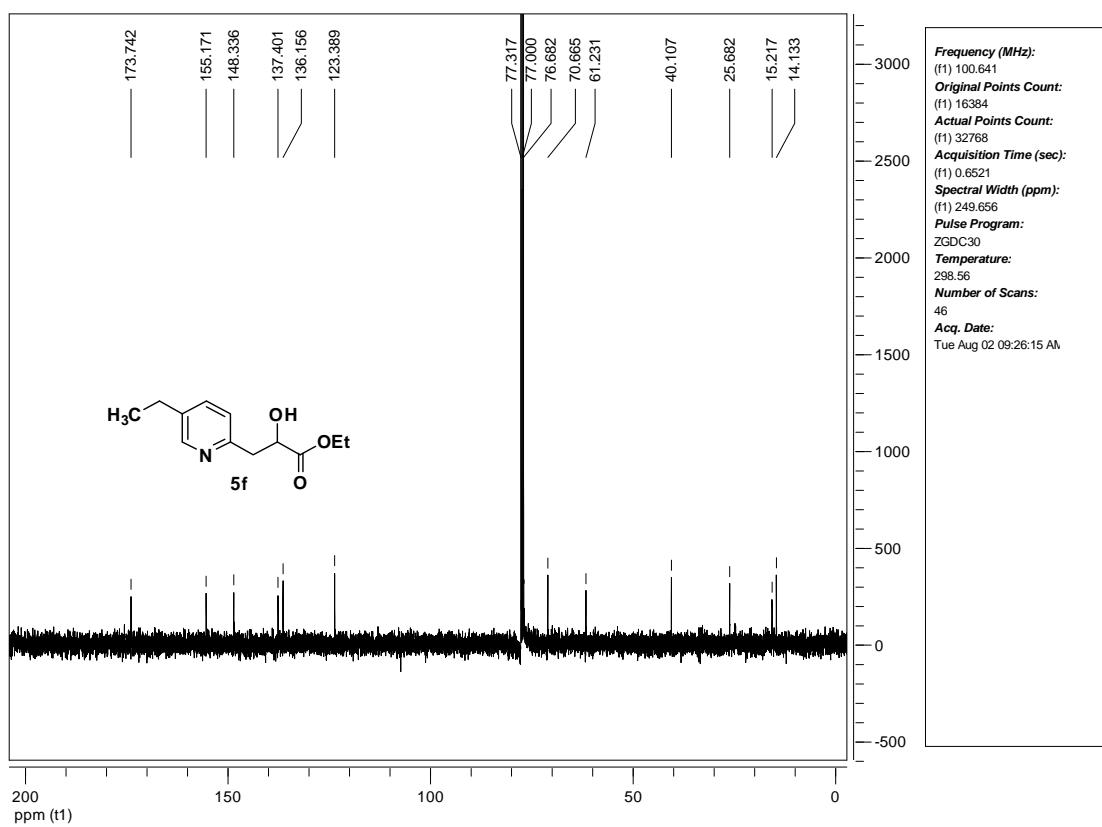
¹³C NMR Spectrum for 5e



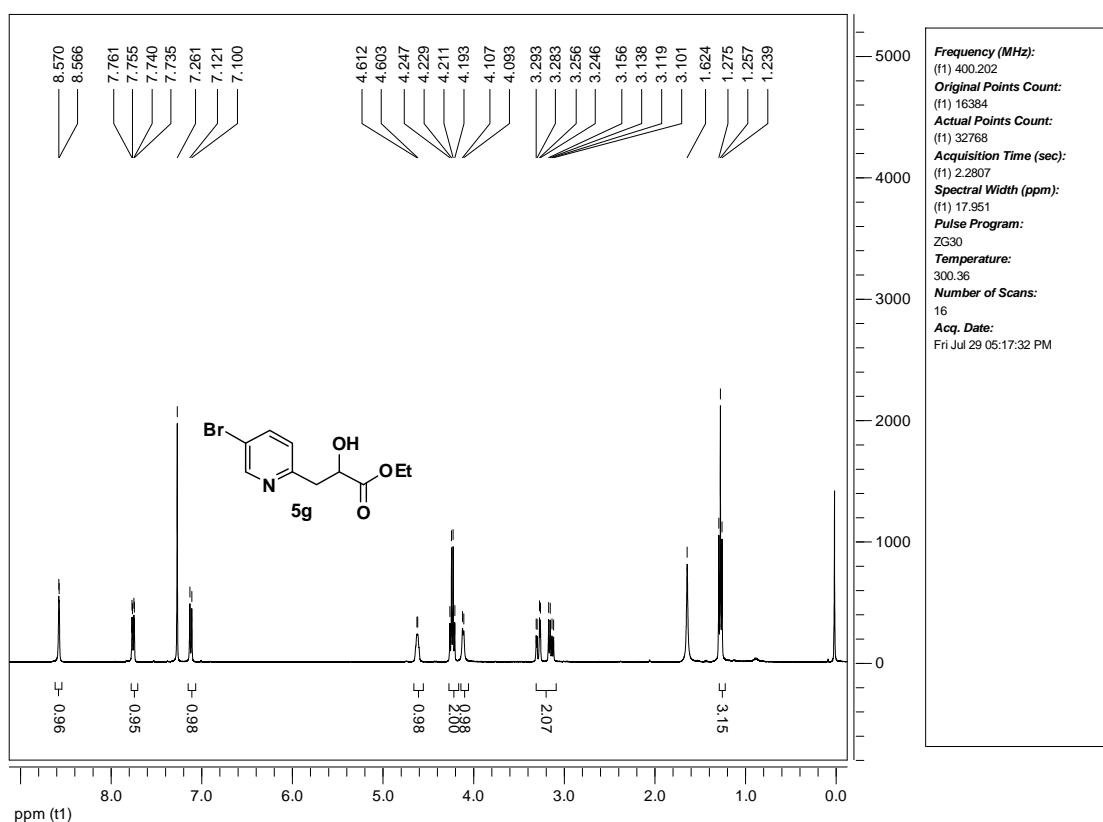
¹H NMR Spectrum for 5f



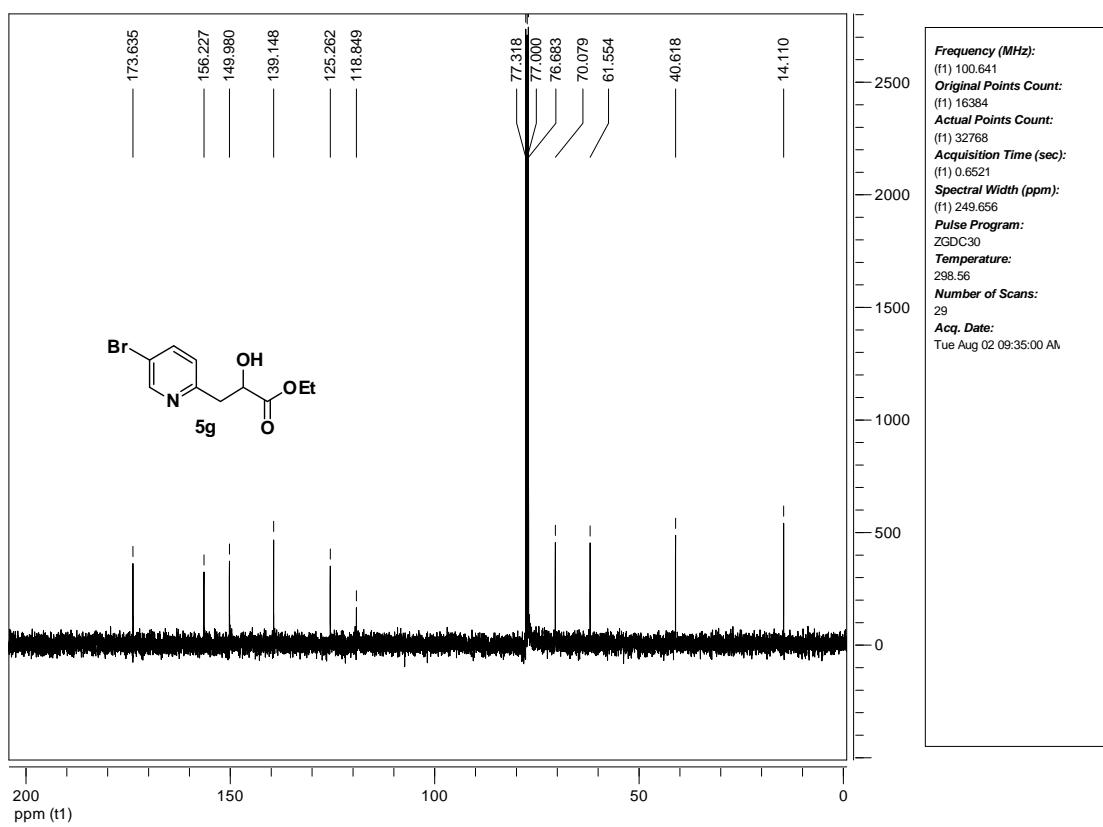
¹³C NMR Spectrum for 5f



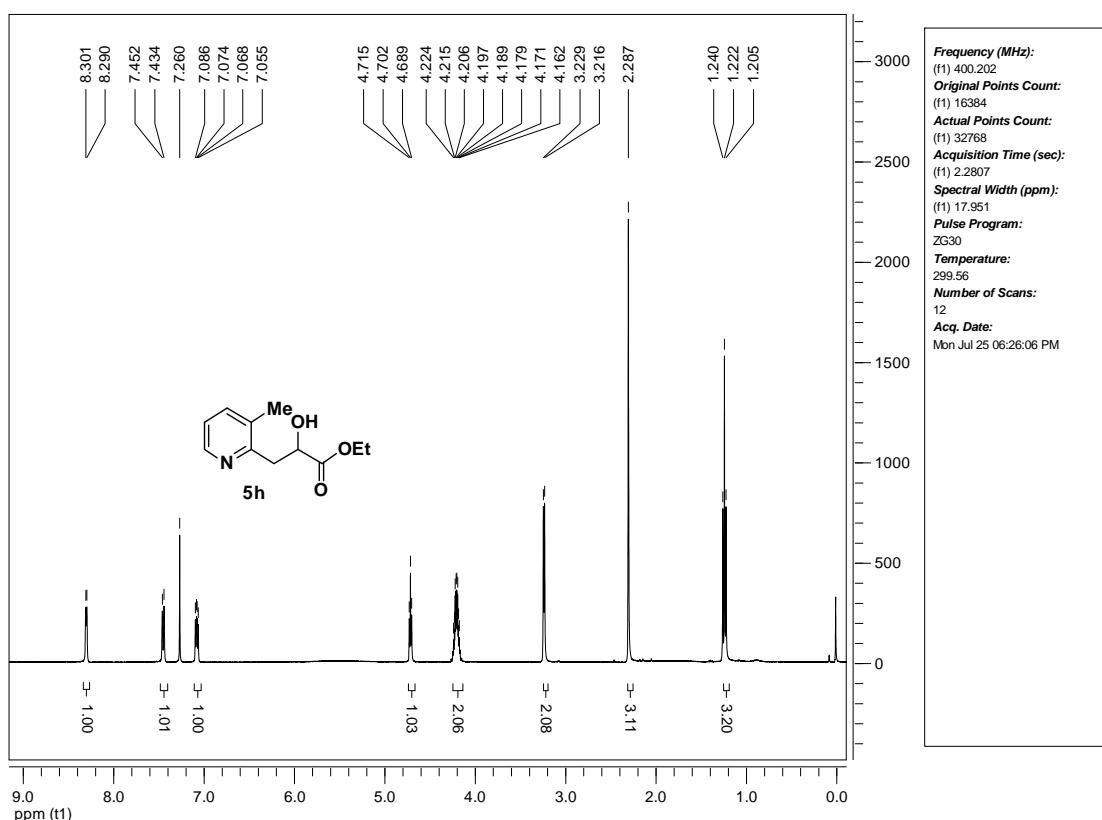
¹H NMR Spectrum for 5g



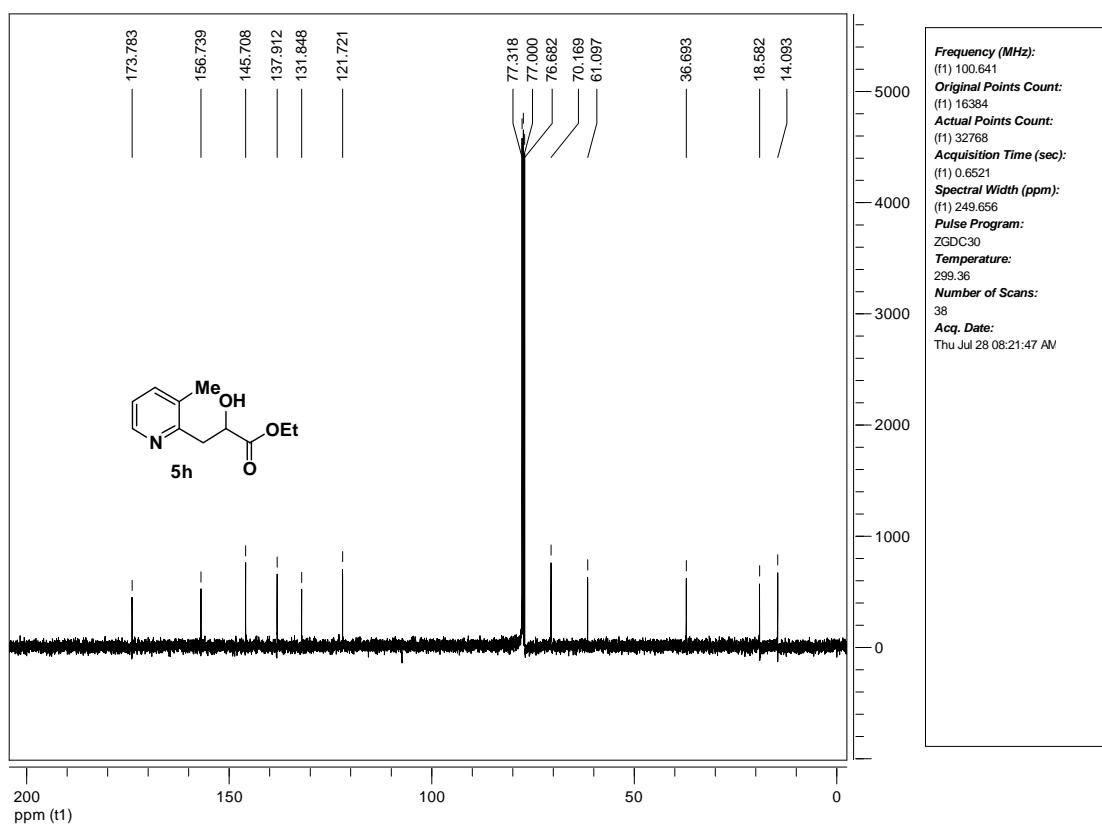
¹³C NMR Spectrum for 5g



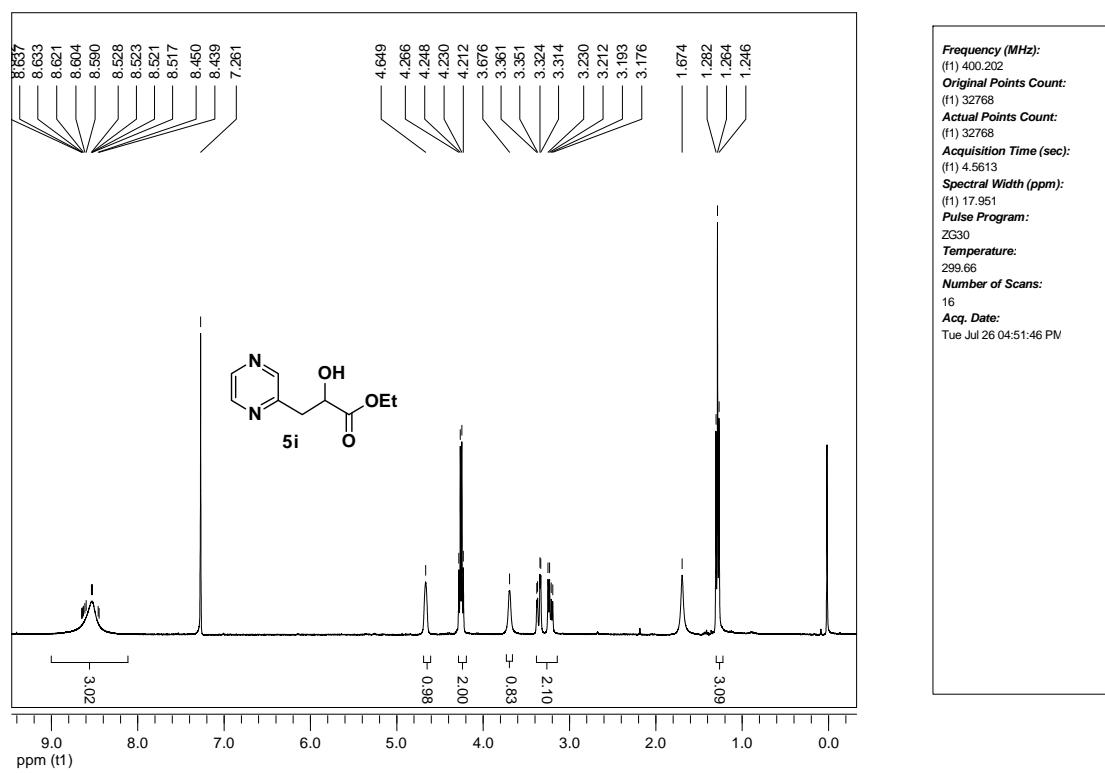
¹H NMR Spectrum for 5h



¹³C NMR Spectrum for 5h



¹H NMR Spectrum for 5i



¹³C NMR Spectrum for 5i

