

Supporting Information

for

**Functionalization of *N*-arylglycine esters:
electrocatalytic access to C–C bonds mediated by
n-Bu₄NI**

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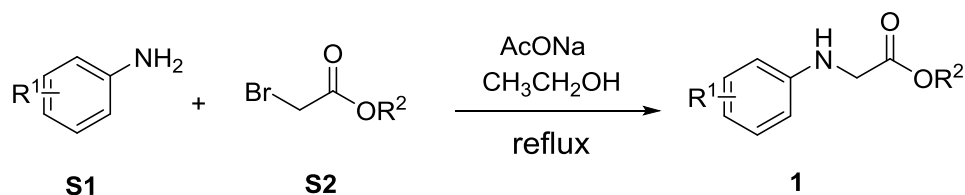
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General procedure for the synthesis of *N*-ethyl acetate-substituted anilines.¹



In an ordinary vial equipped with a magnetic stirring bar, aniline **S1** (10 mmol, 1.0 equiv), ethyl bromoacetate **S2** (10 mmol, 1.0 equiv), sodium acetate (10 mmol, 1.0 equiv), and ethanol (6.6 M) were added. After the reaction mixture was stirred at 85–90 °C overnight, the solvent was removed in vacuo. The residue was dissolved in DCM and washed with saturated NaCl (aqueous), the aqueous layer was washed two times with DCM, and the combined organic layers were dried over MgSO_4 . Finally, the residue was purified by column chromatography.

Compounds characterization

Ethyl 2-(4-tolylamino)-2-(2,4,6-trimethoxyphenyl)acetate (**3aa**) [2] White solid; mp: 99-101 °C; ^1H NMR(400 MHz, CDCl_3), δ (ppm): 1.19 (t, $J = 7.2$ Hz, 3H), 2.22 (s, 3H), 3.81 (s, 3H), 3.85 (s, 6H), 4.11- 4.25 (m, 2H), 4.79 (br, 1H), 5.67 (s, 1H), 6.13 (s, 2H), 6.68 (d, $J = 8.4$ Hz, 2H), 6.96 (d, $J = 8.0$ Hz, 2H); ^{13}C NMR(100 MHz, CDCl_3), δ (ppm): 14.2, 20.4, 51.4, 55.3, 55.9, 61.0, 91.0, 108.9, 114.1, 126.8, 129.5, 145.2, 158.8, 160.9, 173.1.

Ethyl 2-((4-methoxyphenyl)amino)-2-(2,4,6-trimethoxyphenyl)acetate (**3ba**) White solid; mp: 76-77 °C; ^1H NMR (400 MHz, CDCl_3), δ (ppm): 1.16 (t, $J = 7.2$ Hz, 3H), 3.71 (s, 3H), 3.78 (s, 3H), 3.82 (s, 6H), 4.08-4.26 (m, 2H), 5.58 (s, 1H), 6.11 (s, 2H), 6.64 (s, 4H); ^{13}C NMR (100 MHz, CDCl_3), δ (ppm): 14.2, 52.3, 55.3, 55.7, 55.9, 61.0, 91.0, 108.8, 114.6, 115.6, 141.7, 152.4, 158.8, 160.9, 173.2; HRMS (ESI) m/z calculated for $\text{C}_{20}\text{H}_{25}\text{NO}_6$ ($\text{M}+\text{Na}$)⁺ 398.1574, Found 398.1578.

Ethyl 2-(4-bromophenylamino)-2-(2,4,6-trimethoxyphenyl)acetate (**3ca**) [2] White solid; mp:

113-115 °C; ¹H NMR(300 MHz, CDCl₃), δ (ppm): 1.18 (t, *J* = 7.2 Hz, 3H), 3.80 (s, 3H), 3.84 (s, 6H), 4.11-4.24 (m, 2H), 4.93 (br, 1H), 5.61 (s, 1H), 6.12 (s, 2H), 6.61 (d, *J* = 9.0 Hz, 2H), 7.20 (d, *J* = 8.7 Hz, 2H); ¹³C NMR(100 MHz, CDCl₃), δ (ppm): 14.2, 50.9, 55.3, 55.9, 61.2, 91.0, 108.0, 109.2, 115.4, 131.7, 146.4, 158.7, 161.1, 172.6.

Ethyl 2-(4-chlorophenylamino)-2-(2,4,6-trimethoxyphenyl)acetate (**3da**) [2] White solid; mp: 106-107 °C; ¹H NMR (400 MHz, CDCl₃), δ (ppm): 1.17 (t, *J* = 7.2 Hz, 3H), 3.79 (s, 3H), 3.83 (s, 6H), 4.09-4.23 (m, 2H), 5.60 (s, 1H), 6.11 (s, 2H), 6.64 (d, *J* = 8.8 Hz, 2H), 7.05 (d, *J* = 8.8 Hz, 2H); ¹³C NMR (100 MHz, CDCl₃), δ (ppm): 14.2, 51.0, 55.3, 55.9, 61.2, 91.0, 108.1, 114.9, 122.1, 128.8, 146.0, 158.7, 161.1, 172.7;

Ethyl 2-((4-(trifluoromethyl)phenyl)amino)-2-(2,4,6-trimethoxyphenyl)acetate (**3ea**) White solid; mp: 99-100°C; ¹H NMR (400 MHz, CDCl₃), δ (ppm): 1.20 (t, *J* = 7.2 Hz, 3H), 3.82 (s, 3H), 3.87 (s, 6H), 4.12-4.27 (m, 2H), 5.23 (d, *J* = 7.6 Hz, 1H), 5.70 (d, *J* = 7.6 Hz, 1H), 6.15 (s, 2H), 6.73 (d, *J* = 7.6 Hz, 2H), 7.36 (d, *J* = 8.4 Hz, 2H); ¹³C NMR (100 MHz, CDCl₃), δ (ppm): 14.2, 50.3, 55.3, 55.9, 61.3, 91.0, 107.8, 112.5, 118.8 (q, *J*_{CF} = 32.5 Hz), 125.0 (q, *J*_{CF} = 268.7 Hz), 126.4 (q, *J*_{CF} = 3.8 Hz), 149.7, 158.7, 161.2, 172.3; HRMS (ESI) *m/z* calculated for C₂₀H₂₂F₃NO₅ (M+Na)⁺ 436.1342, Found 436.1338.

Ethyl 2-(phenylamino)-2-(2,4,6-trimethoxyphenyl)acetate (**3fa**) [2] White solid; mp: 106-108 °C; ¹H NMR(300 MHz, CDCl₃), δ (ppm): 1.18 (t, *J* = 7.2 Hz, 3H), 3.80 (s, 3H), 3.85 (s, 6H), 4.11-4.24 (m, 2H), 5.69 (s, 1H), 6.13 (s, 2H), 6.67 (t, *J* = 7.2 Hz, 1H), 6.74 (d, *J* = 7.8 Hz, 2H), 7.14 (t, *J* = 7.5 Hz, 2H); ¹³C NMR(100 MHz, CDCl₃), δ (ppm): 14.2, 51.0, 55.3, 55.9, 61.1, 91.0, 108.7, 113.8, 117.6, 129.0, 147.4, 158.8, 160.9, 173.0.

Ethyl 2-(2-tolylamino)-2-(2,4,6-trimethoxyphenyl)acetate (**3ga**) [2] White solid; mp: 104-106 °C; ¹H NMR(400 MHz, CDCl₃), δ (ppm): 1.17 (t, *J* = 7.2 Hz, 3H), 2.20 (s, 3H), 3.79 (s, 3H), 3.85 (s, 6H), 4.12-4.22 (m, 2H), 5.74 (s, 1H), 6.12 (s, 2H), 6.58-6.61 (m, 1H), 6.77 (d, *J* = 8.0 Hz, 1H), 6.99-7.05 (m, 2H); ¹³C NMR (100 MHz, CDCl₃), δ (ppm): 14.2, 17.5, 51.0, 55.3, 55.9, 61.1, 91.0, 108.7, 111.0, 117.1, 122.6, 126.8, 130.0, 145.4, 158.8, 160.9, 173.0.

Ethyl 2-(3-tolylamino)-2-(2,4,6-trimethoxyphenyl)acetate (**3ha**) [2] White solid; mp: 100-102 °C; ¹H NMR (400 MHz, CDCl₃), δ (ppm): 1.19 (t, *J* = 7.2 Hz, 3H), 2.26 (s, 3H), 3.81 (s, 3H), 3.86 (s, 6H), 4.12-4.24 (m, 2H), 4.93 (br, 1H), 5.68 (s, 1H), 6.13 (s, 2H), 6.50 (d, *J* = 7.2 Hz, 1H), 6.55-6.58 (m, 2H), 7.02 (t, *J* = 8.0 Hz, 1H); ¹³C NMR (100 MHz, CDCl₃), δ (ppm): 14.2,

21.6, 50.9, 55.3, 55.8, 61.0, 91.0, 108.8, 110.8, 114.5, 118.5, 128.8, 138.6, 147.3, 158.7, 160.9, 173.0.

Methyl 2-(4-tolylamino)-2-(2,4,6-trimethoxyphenyl)acetate (**3ia**) [2] White solid; mp: 162-163 °C; ¹HNMR(300 MHz, CDCl₃), δ (ppm): 2.21 (s, 3H), 3.69 (s, 3H), 3.80 (s, 3H), 3.85 (s, 6H), 5.69 (s, 1H), 6.13 (s, 2H), 6.67 (d, *J* = 8.4 Hz, 2H), 6.95 (d, *J* = 8.1 Hz, 2H); ¹³C NMR (100 MHz, CDCl₃), δ (ppm): 20.4, 51.2, 52.3, 55.3, 55.9, 91.0, 108.5, 114.1, 126.9, 129.6, 145.0, 158.8, 161.0, 173.6;

Benzyl 2-(*p*-tolylamino)-2-(2,4,6-trimethoxyphenyl)acetate (**3ja**) White solid; mp: 128-129 °C; ¹HNMR(300 MHz, CDCl₃), δ (ppm): 2.22 (s, 3H), 3.72 (s, 6H), 3.80 (s, 3H), 4.80 (br, 1H), 5.07 (d, *J* = 12.6 Hz, 1H), 5.25 (d, *J* = 12.3 Hz, 1H), 5.76 (s, 1H), 6.10 (s, 2H), 6.68 (d, *J* = 8.1 Hz, 2H), 6.96 (d, *J* = 8.1 Hz, 2H), 7.19-7.21 (m, 2H), 7.28-7.29 (m, 3H); ¹³CNMR (75 MHz, CDCl₃), δ (ppm): 20.4, 51.4, 55.3, 55.7, 66.3, 90.8, 108.4, 114.1, 126.9, 127.8, 127.9, 128.3, 129.6, 136.4, 145.1, 158.7, 161.0, 173.0; HRMS (ESI) *m/z* calculated for C₂₅H₂₇NO₅ (M+Na)⁺ 444.1781, Found 444.1777.

Diethyl 2-benzoyl-3-(*p*-tolylamino)succinate (**3ab**) [3] Yellow liquid; ¹H NMR (400 MHz, CDCl₃), δ (ppm): 1.12-1.23 (m, 6H), 2.25 (s, 3H), 4.12-4.20 (m, 4H), 4.33-4.60 (br, 1H), 4.87-5.07 (m, 2H), 6.63 (d, *J* = 8.0 Hz, 1H), 6.69 (d, *J* = 8.0 Hz, 1H), 6.98-7.01 (m, 2H), 7.46-7.52 (m, 2H), 7.58-7.61 (m, 1H), 7.95 (d, *J* = 7.6 Hz, 2H); ¹³CNMR (100 MHz, CDCl₃), δ (ppm): 13.87, 13.90, 14.0, 20.41, 20.44, 55.8, 56.8, 57.3, 58.0, 61.63, 61.69, 61.87, 61.92, 114.3, 114.8, 115.3, 128.28, 128.34, 128.42, 128.55, 128.75, 129.7, 129.8, 133.6, 133.7, 136.1, 136.7, 143.9, 144.7, 167.8, 167.9, 171.6, 171.8, 193.0, 194.5.

2-Ethyl 1,1-dimethyl 2-(*p*-tolylamino)ethane-1,1,2-tricarboxylate (**3ac**) [4] Yellow liquid; ¹HNMR (400 MHz, CDCl₃), δ (ppm): 1.25 (t, *J* = 7.2 Hz, 3H), 2.26 (s, 3H), 3.74 (s, 3H), 3.81 (s, 3H), 4.10 (d, *J* = 5.6 Hz, 1H), 4.15-4.27 (m, 2H), 4.53 (d, *J* = 8.0 Hz, 1H), 4.72 (t, *J* = 7.2 Hz, 1H), 6.65 (d, *J* = 8.0 Hz, 2H), 7.02 (d, *J* = 8.0 Hz, 2H); ¹³CNMR (100 MHz, CDCl₃), δ (ppm): 14.0, 20.4, 52.8, 53.9, 57.1, 61.9, 114.2, 128.4, 129.8, 143.8, 167.6, 168.0, 171.0.

Triethyl 2-(*p*-tolylamino)ethane-1,1,2-tricarboxylate (**3ad**) [4] Yellow liquid; ¹HNMR (400 MHz, CDCl₃), δ (ppm): 1.22-1.33 (m, 9H), 2.25 (s, 3H), 4.07 (d, *J* = 5.2 Hz, 1H), 4.18-4.27 (m, 6H), 4.55 (d, *J* = 9.2 Hz, 1H), 4.70-4.73 (m, 1H), 6.66 (d, *J* = 8.4 Hz, 2H), 7.01 (d, *J* = 8.4 Hz, 2H); ¹³C NMR (100 MHz, CDCl₃), δ (ppm): 14.0, 20.4, 54.3, 57.0, 61.75, 61.8, 61.9, 114.2,

128.3, 129.8, 144.0, 167.1, 167.6, 171.1.

Ethyl 2-(*p*-tolylamino)-2-(2-hydroxynaphthalen-1-yl)acetate (**3ae**) [2] Brown oil; ^1H NMR (400 MHz, CDCl_3), δ (ppm): 1.12 (t, $J = 7.2$ Hz, 3H), 2.22 (s, 3H), 4.07-4.15 (m, 1H), 4.24-4.32 (m, 1H), 5.10 (br, 1H), 5.86 (s, 1H), 6.65 (d, $J = 8.4$ Hz, 2H), 6.94 (d, $J = 8.4$ Hz, 2H), 7.09 (d, $J = 8.8$ Hz, 1H), 7.41 (t, $J = 7.4$ Hz, 1H), 7.58 (t, $J = 8.0$ Hz, 1H), 7.76 (d, $J = 8.8$ Hz, 1H), 7.84 (d, $J = 8.0$ Hz, 1H), 8.14 (d, $J = 8.8$ Hz, 1H), 10.32 (br, 1H); ^{13}C NMR (100 MHz, CDCl_3), δ (ppm): 13.9, 20.5, 57.5, 62.5, 111.4, 116.2, 119.8, 121.9, 123.0, 126.9, 129.0, 129.2, 129.9, 130.6, 130.8, 132.4, 142.9, 156.0, 171.3;

Ethyl 2-(2,7-dihydroxynaphthalen-1-yl)-2-(*p*-tolylamino)acetate (**3af**) Brown oil; ^1H NMR (400 MHz, CDCl_3), δ (ppm): 1.13 (t, $J = 6.8$ Hz, 3H), 2.21 (s, 3H), 4.10-4.15 (m, 1H), 4.24-4.28 (m, 1H), 5.04 (br, 1H), 5.28 (br, 1H), 5.65 (s, 1H), 6.63 (d, $J = 8.0$ Hz, 2H), 6.89-7.00 (m, 4H), 7.44 (d, $J = 1.6$ Hz, 1H), 7.66 (d, $J = 9.2$ Hz, 1H), 7.71 (t, $J = 8.8$ Hz, 1H), 10.26 (br, 1H); ^{13}C NMR (100 MHz, CDCl_3), δ (ppm): 13.9, 20.5, 57.6, 62.5, 104.9, 110.3, 114.6, 116.2, 117.2, 124.5, 129.9, 130.5, 130.8, 130.9, 134.0, 142.8, 154.8, 156.4, 171.4; HRMS (ESI) m/z calculated for $\text{C}_{21}\text{H}_{21}\text{NO}_4(\text{M}+\text{Na})^+$ 374.1363, Found 374.1367.

Ethyl 2-(2,6-dihydroxynaphthalen-1-yl)-2-(*p*-tolylamino)acetate (**3ag**) Brown oil; ^1H NMR (400 MHz, CDCl_3), δ (ppm): 1.36 (t, $J = 7.2$ Hz, 3H), 2.24 (s, 3H), 4.09 (d, $J = 18.3$ Hz, 1H), 4.30-4.43 (m, 2H), 4.74 (d, $J = 18.3$ Hz, 1H), 5.27 (br, 1H), 6.34 (d, $J = 8.4$ Hz, 2H), 6.98-7.03 (m, 3H), 7.18 (d, $J = 2.0$ Hz, 1H), 7.29 (d, $J = 9.3$ Hz, 1H), 7.38 (d, $J = 9.0$ Hz, 1H), 7.63 (d, $J = 9.0$ Hz, 1H), 8.72 (s, 1H); ^{13}C NMR (100 MHz, CDCl_3), δ (ppm): 14.2, 20.3, 53.7, 62.6, 110.8, 112.0, 118.8, 119.8, 123.4, 123.8, 127.3, 127.7, 127.9, 130.0, 130.9, 144.2, 151.3, 151.9, 174.5; HRMS (ESI) m/z calculated for $\text{C}_{21}\text{H}_{21}\text{NO}_4(\text{M}+\text{H})^+$ 352.1543, Found 352.1544.

Ethyl 6-methyl-4-phenylquinoline-2-carboxylate (**3ah**) [5] White solid; mp: 172.2-172.3°C; ^1H NMR (400 MHz, CDCl_3), δ (ppm): 1.50 (t, $J = 7.2$ Hz, 3H), 2.51 (s, 3H), 4.58 (q, $J = 7.2$ Hz, 2H), 7.53-7.57 (m, 5H), 7.63 (dd, $J = 8.4, 1.6$ Hz, 1H), 7.72 (s, 1H), 8.11 (s, 1H), 8.29 (d, $J = 8.8$ Hz, 2H); ^{13}C NMR (100 MHz, CDCl_3), δ (ppm): 14.4, 22.0, 62.7, 121.4, 124.4, 127.8, 128.6, 128.7, 129.5, 130.9, 132.3, 137.8, 138.9, 146.8, 146.9, 148.9, 165.6.

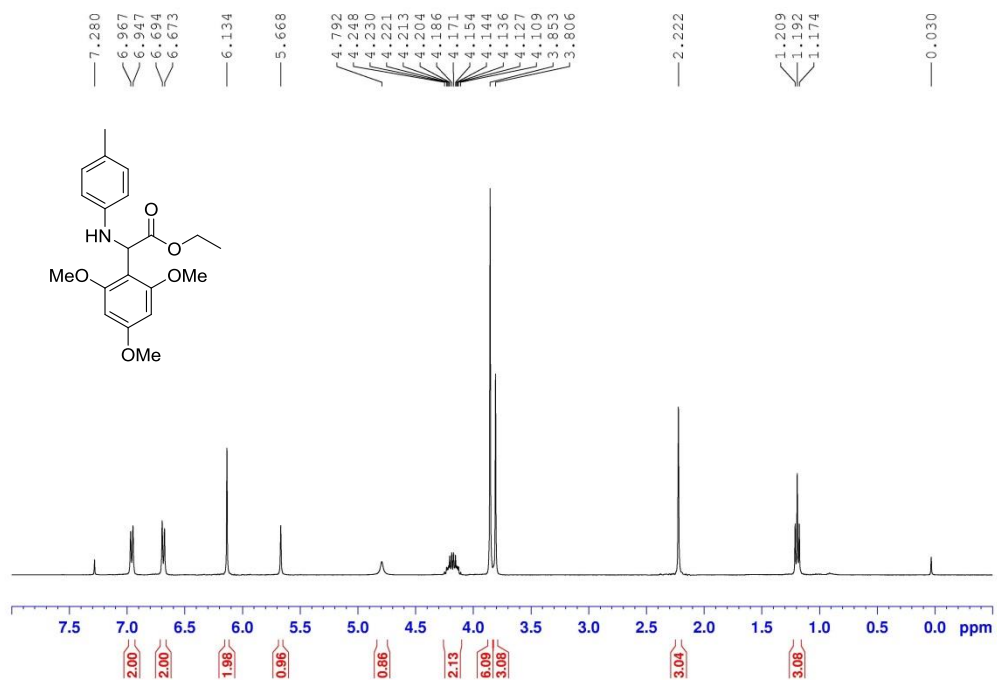
ethyl 6-methyl-4-(*p*-tolyl)quinoline-2-carboxylate (**3ai**) White solid; mp: 150-151°C; ^1H NMR (400 MHz, CDCl_3), δ (ppm): 1.50 (t, $J = 7.2$ Hz, 3H), 2.50 (s, 3H), 2.51 (s, 3H), 4.58 (q, $J = 7.2$ Hz, 2H), 7.38 (d, $J = 8.0$ Hz, 5H), 7.44 (d, $J = 8.0$ Hz, 5H), 7.63 (dd, $J = 8.4, 1.2$ Hz, 1H), 7.75 (s,

1H), 8.10 (s, 1H), 8.28 (d, $J = 8.4$ Hz, 1H); ^{13}C NMR (100 MHz, CDCl_3), δ (ppm):14.4, 21.3, 22.0, 62.1, 121.4, 124.5, 127.9, 129.4, 129.5, 130.9, 132.2, 134.9, 138.6, 138.8, 146.8, 146.9, 149.0, 165.7.

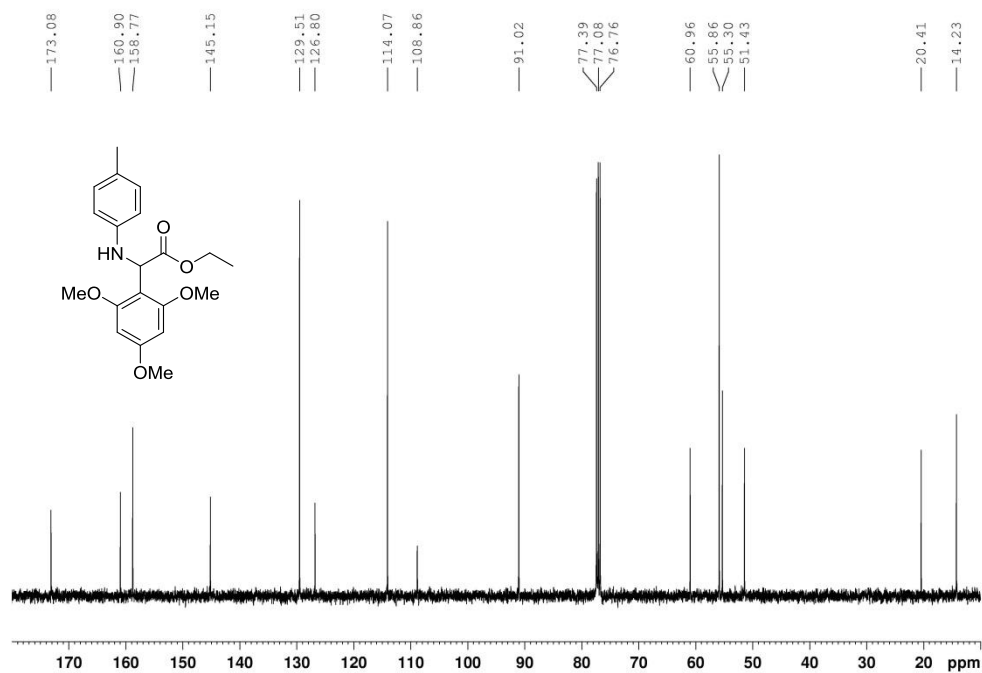
ethyl 4-(4-fluorophenyl)-6-methylquinoline-2-carboxylate (**3aj**) White solid; mp: 174-175°C; ^1H NMR (400 MHz, CDCl_3), δ (ppm):1.51 (t, $J = 7.2$ Hz, 3H), 2.52 (s, 3H), 4.58 (q, $J = 7.2$ Hz, 2H), 7.38 (d, $J = 8.0$ Hz, 5H), 7.44 (d, $J = 8.0$ Hz, 5H), 7.63 (dd, $J = 8.4, 1.2$ Hz, 1H), 7.75 (s, 1H), 8.10 (s, 1H), 8.28 (d, $J = 8.4$ Hz, 1H); ^{13}C NMR (100 MHz, CDCl_3), δ (ppm):14.4, 22.0, 62.2, 115.8 (d, $J_{\text{CF}} = 21.5$ Hz), 121.4, 124.1, 127.8, 131.0, 131.2, 131.3, 132.4, 133.7 (d, $J_{\text{CF}} = 3.4$ Hz), 139.2, 146.9 (d, $J_{\text{CF}} = 11.0$ Hz), 147.8, 163.0 (d, $J_{\text{CF}} = 247.0$ Hz), 165.6.

Spectra of prepared compounds

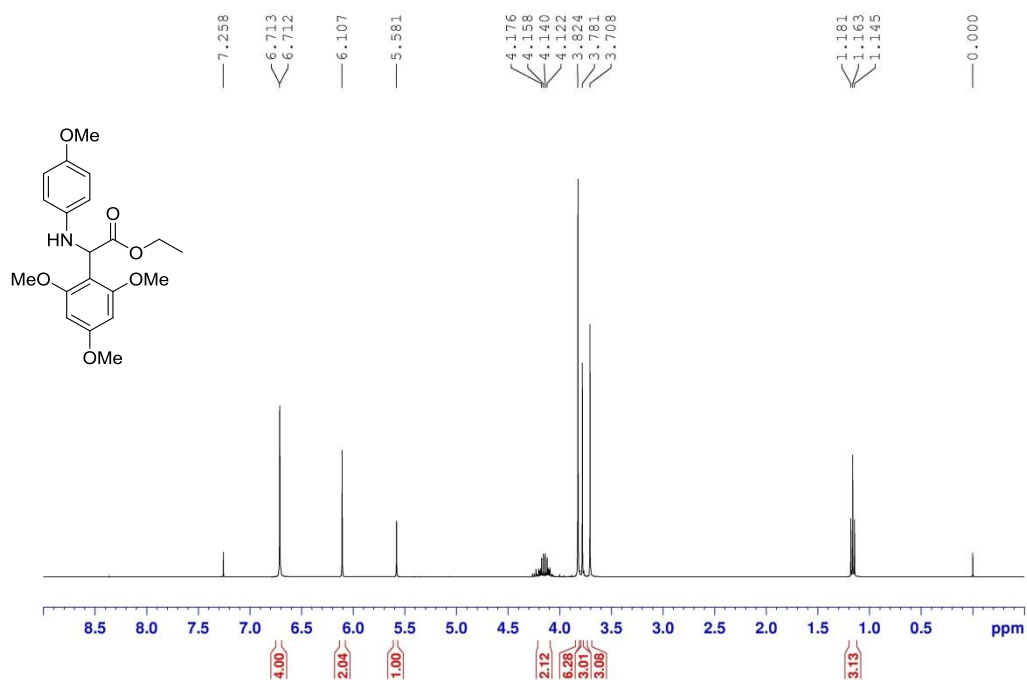
¹H NMR spectra of ethyl 2-(4-tolylamino)-2-(2,4,6-trimethoxyphenyl)acetate (3aa) [2]



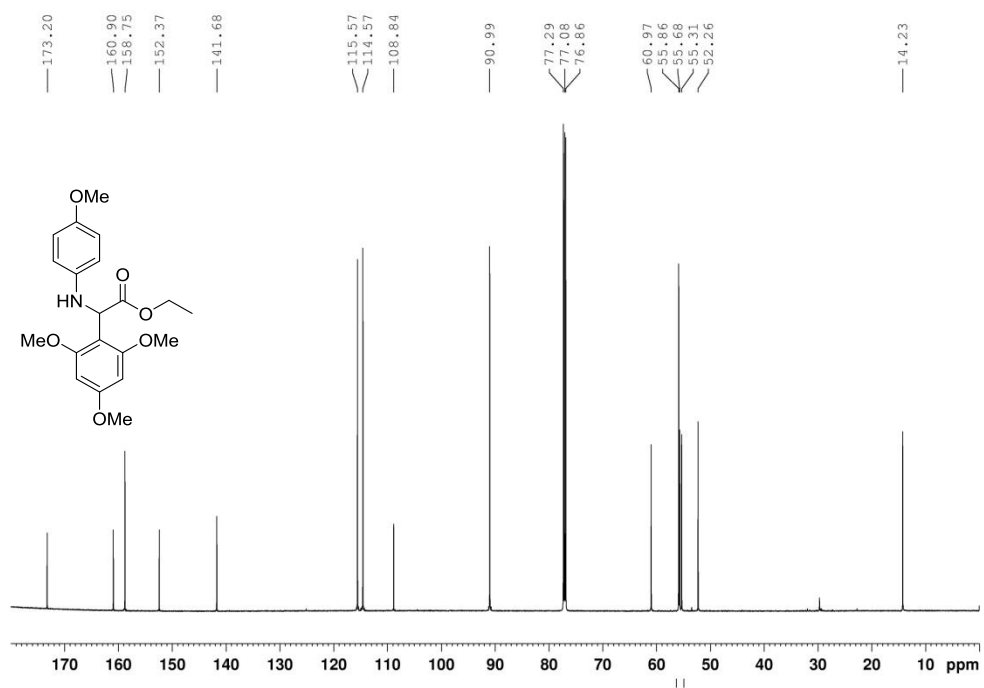
¹³C NMR spectra of ethyl 2-(4-tolylamino)-2-(2,4,6-trimethoxyphenyl)acetate (3aa) [2]



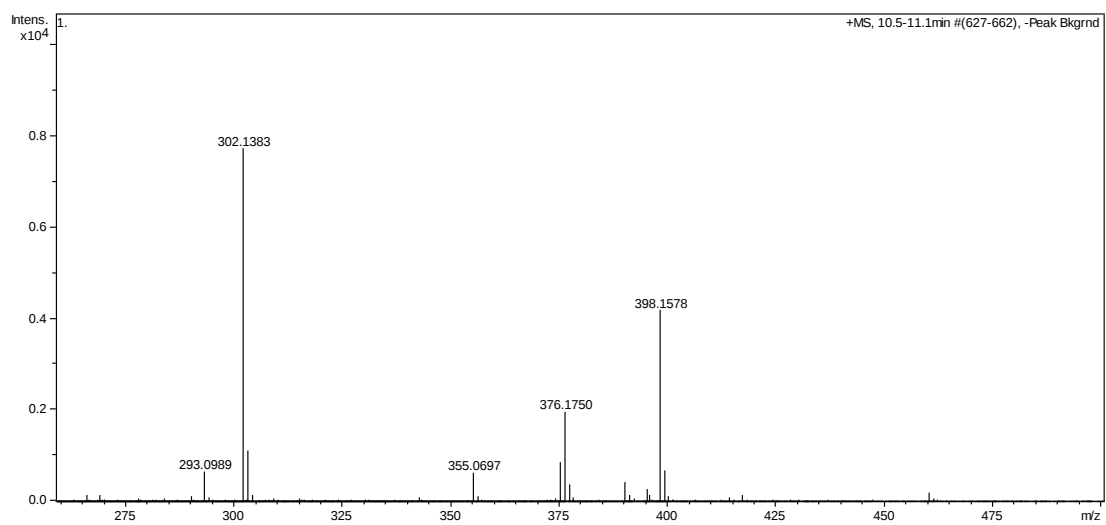
¹H NMR spectra of ethyl 2-((4-methoxyphenyl) amino)-2-(2,4,6-trimethoxyphenyl)acetate (3ba)



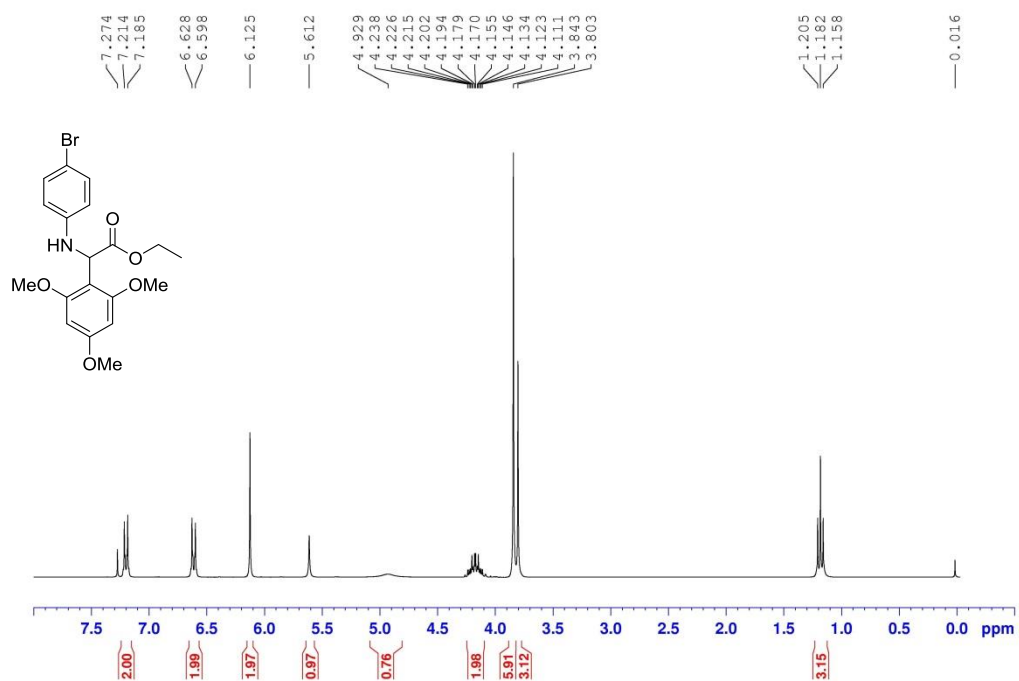
¹³C NMR spectra of ethyl 2-((4-methoxyphenyl) amino)-2-(2,4,6-trimethoxyphenyl)acetate (3ba)



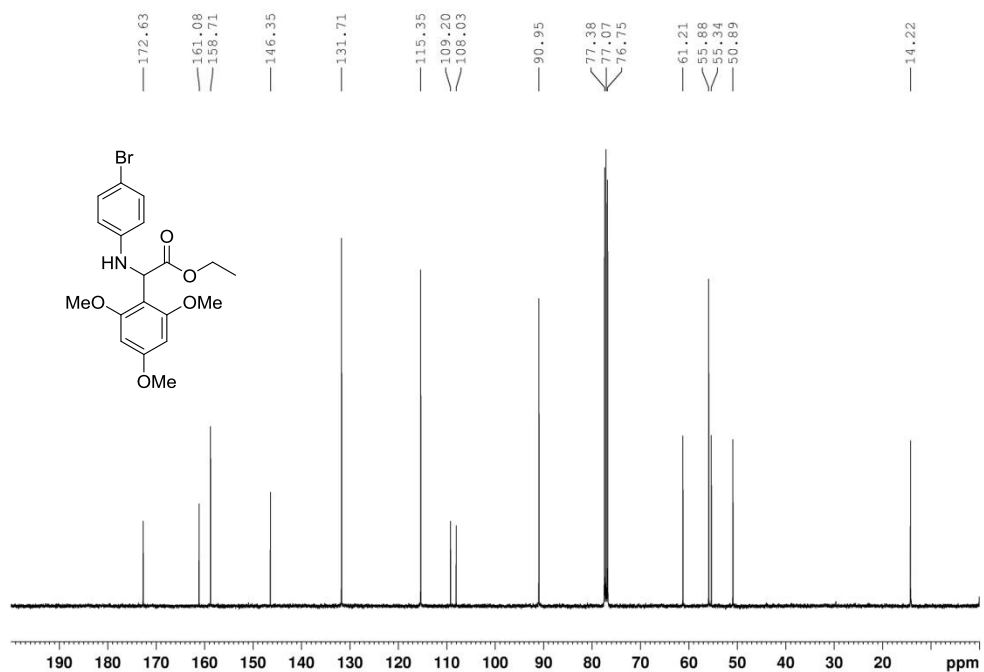
HRMS spectra of ethyl 2-((4-methoxyphenyl) amino)-2-(2,4,6-trimethoxyphenyl)acetate (3ba)



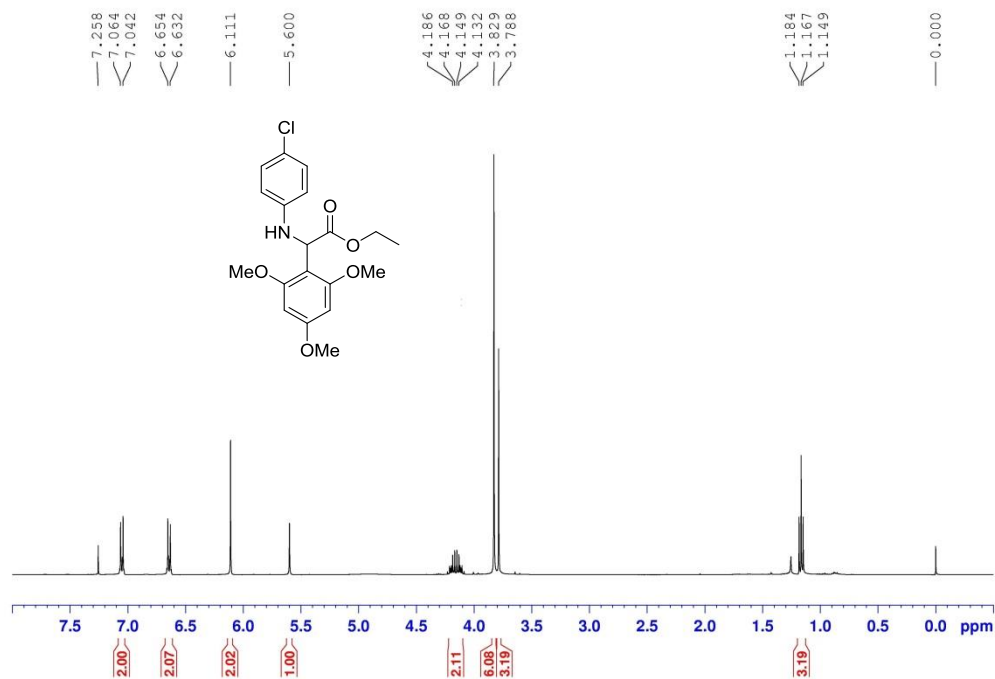
¹H NMR spectra of ethyl-2-(4-bromophenylamino)-2-(2,4,6-trimethoxyphenyl)acetate (3ca) [2]



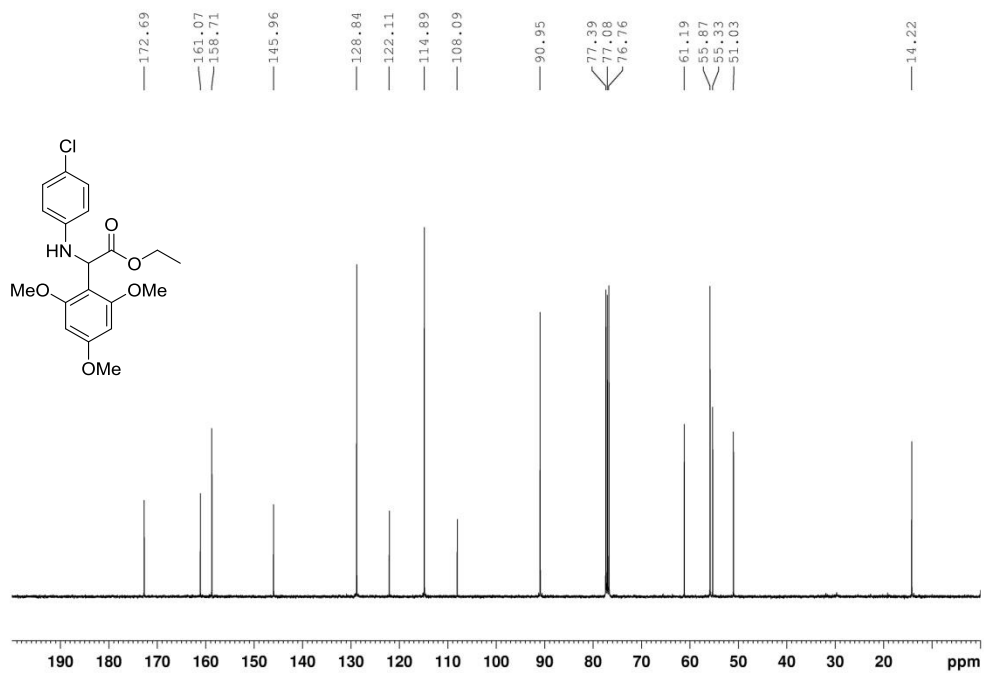
¹³C NMR spectra of ethyl-2-(4-bromophenylamino)-2-(2,4,6-trimethoxyphenyl)acetate (3ca) [2]



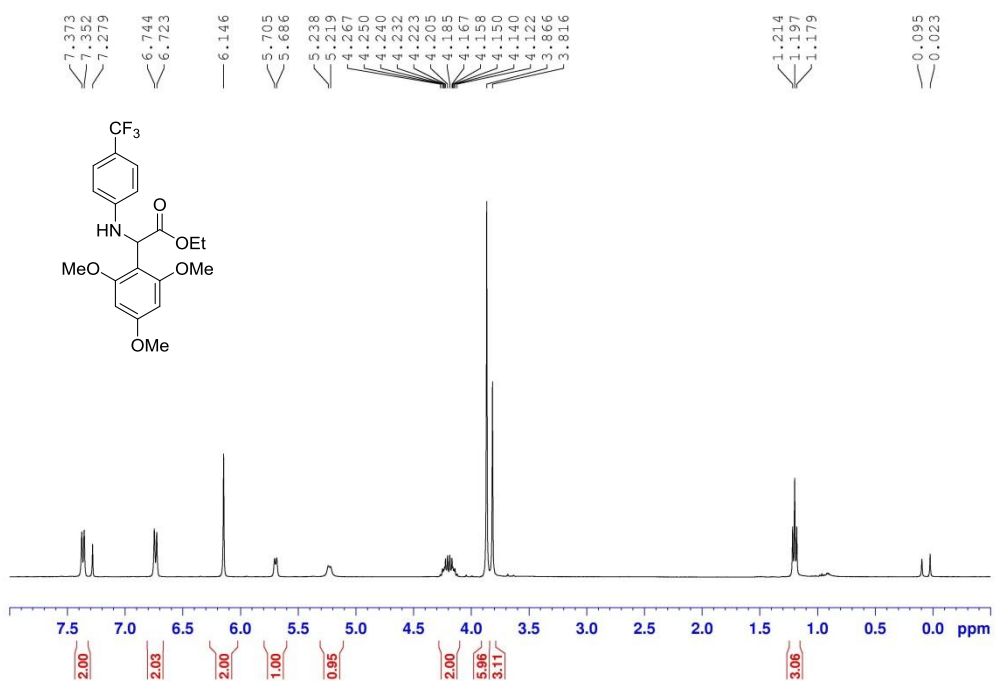
¹H NMR spectra of ethyl 2-(4-chlorophenylamino)-2-(2,4,6-trimethoxyphenyl)acetate (3da) [2]



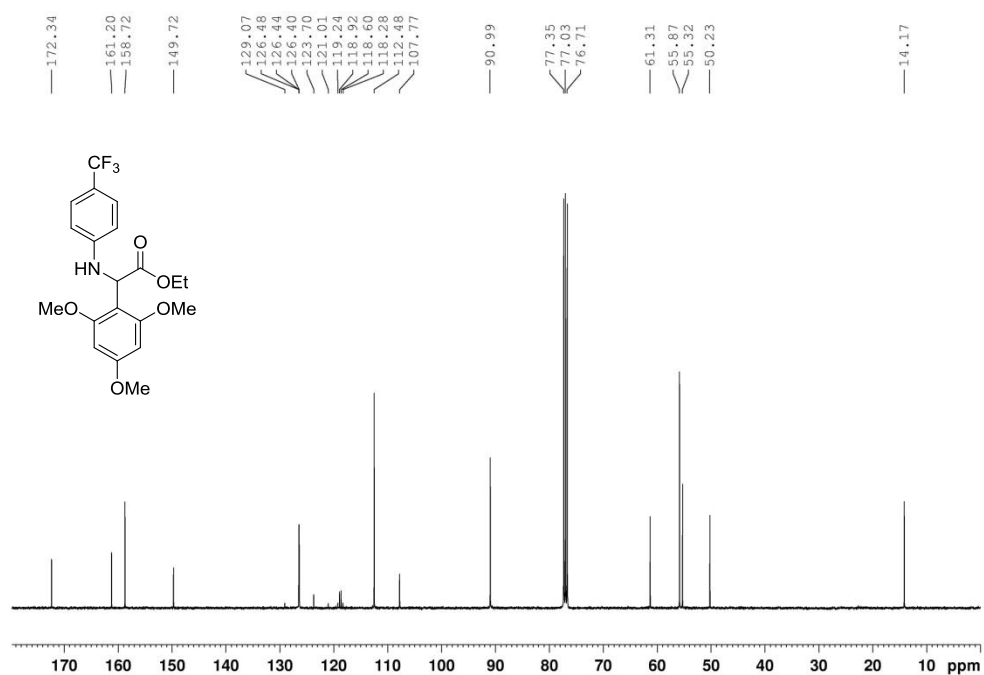
¹³C NMR spectra of ethyl 2-(4-chlorophenylamino)-2-(2,4,6-trimethoxyphenyl)acetate (3da) [2]



¹H NMR spectra of ethyl 2-((4-(trifluoromethyl)phenyl)amino)-2-(2,4,6-trimethoxyphenyl)acetate (3ea)

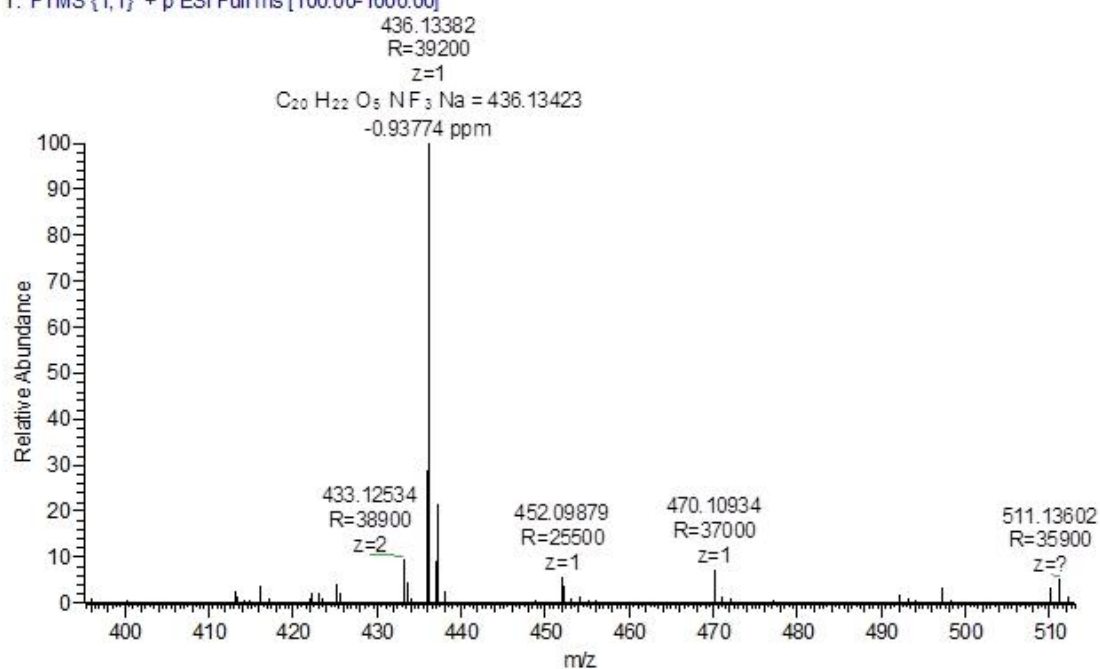


¹³C NMR spectra of ethyl 2-((4-(trifluoromethyl)phenyl)amino)-2-(2,4,6-trimethoxyphenyl)acetate (3ea)

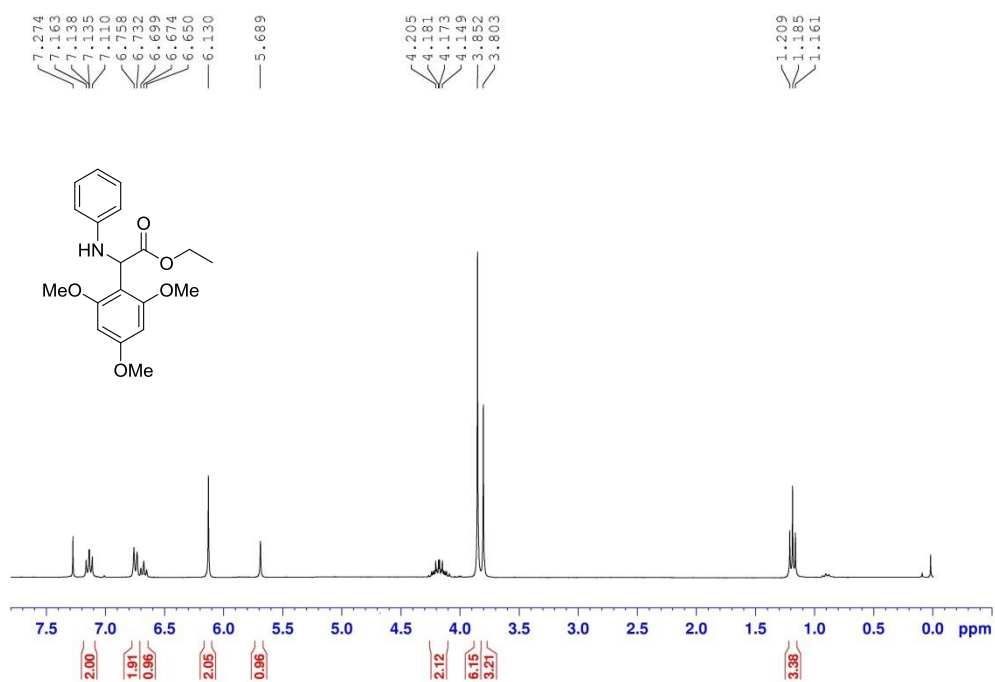


HRMS spectra of ethyl 2-((4-(trifluoromethyl)phenyl)amino)-2-(2,4,6-trimethoxyphenyl)acetate (3ea)

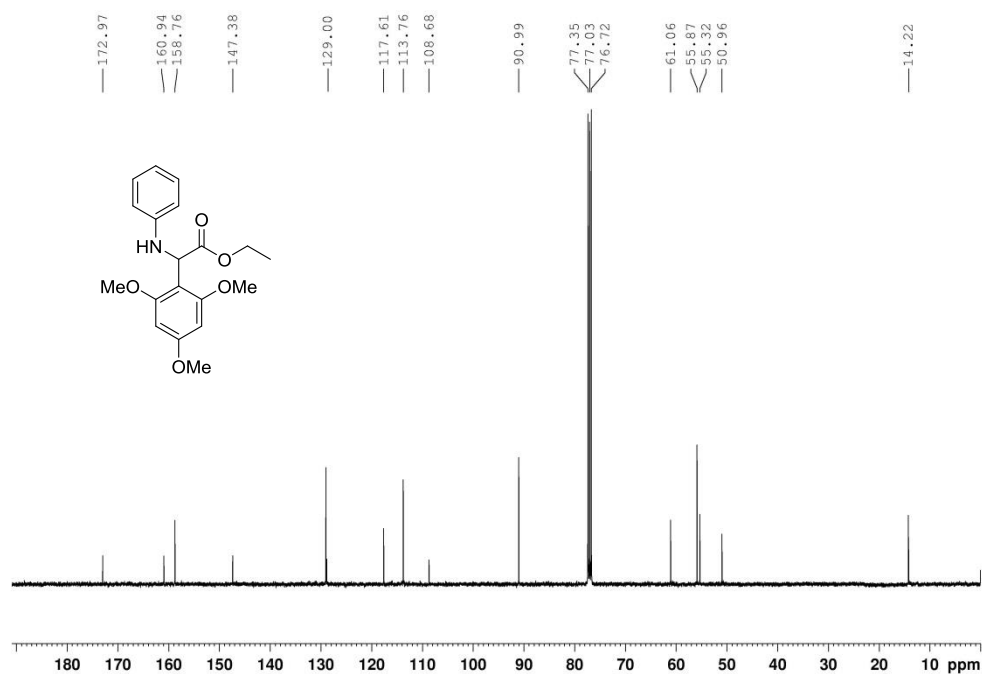
LLJ-925-1#42 RT: 0.40 AV: 1 NL: 2.33E5
T: FTMS {1,1} + p ESI Full ms [100.00-1000.00]



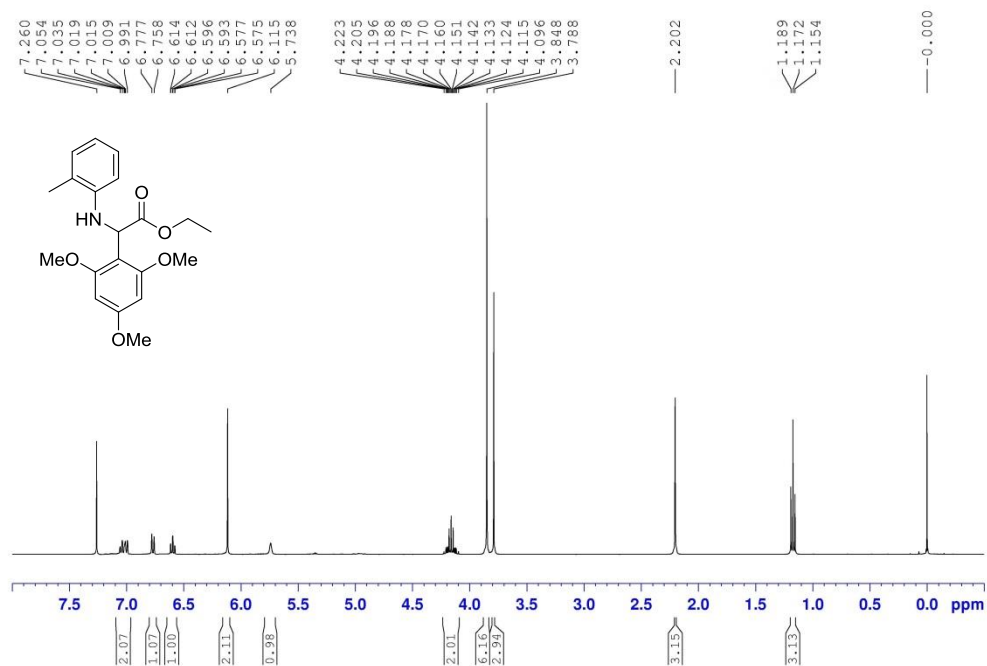
¹H NMR spectra of ethyl 2-(phenylamino)-2-(2,4,6-trimethoxyphenyl)acetate (3fa) [2]



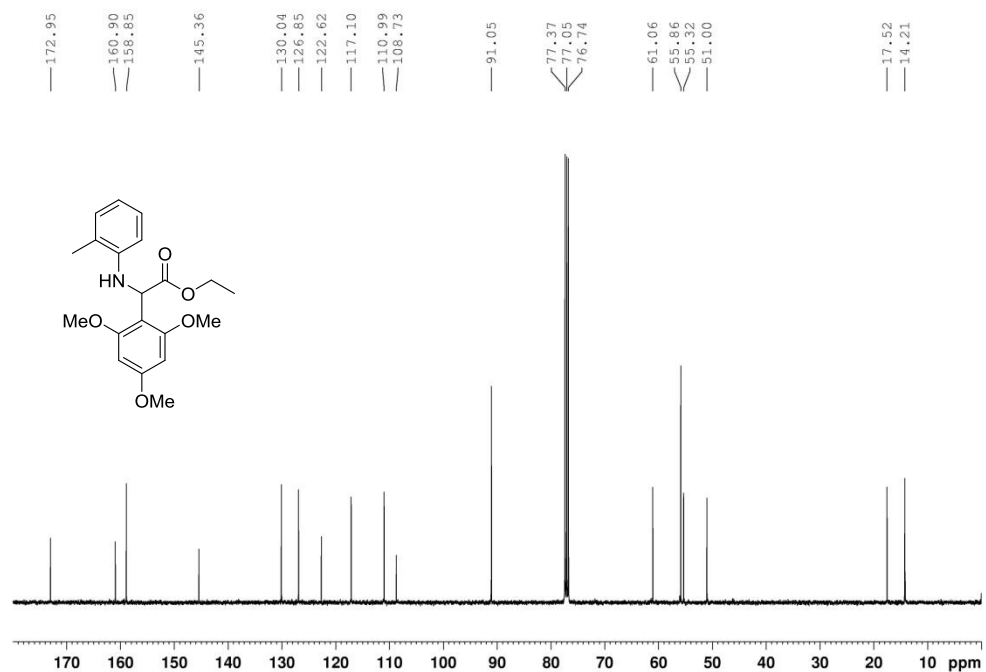
¹³C NMR spectra of ethyl 2-(phenylamino)-2-(2,4,6-trimethoxyphenyl)acetate (3fa) [2]



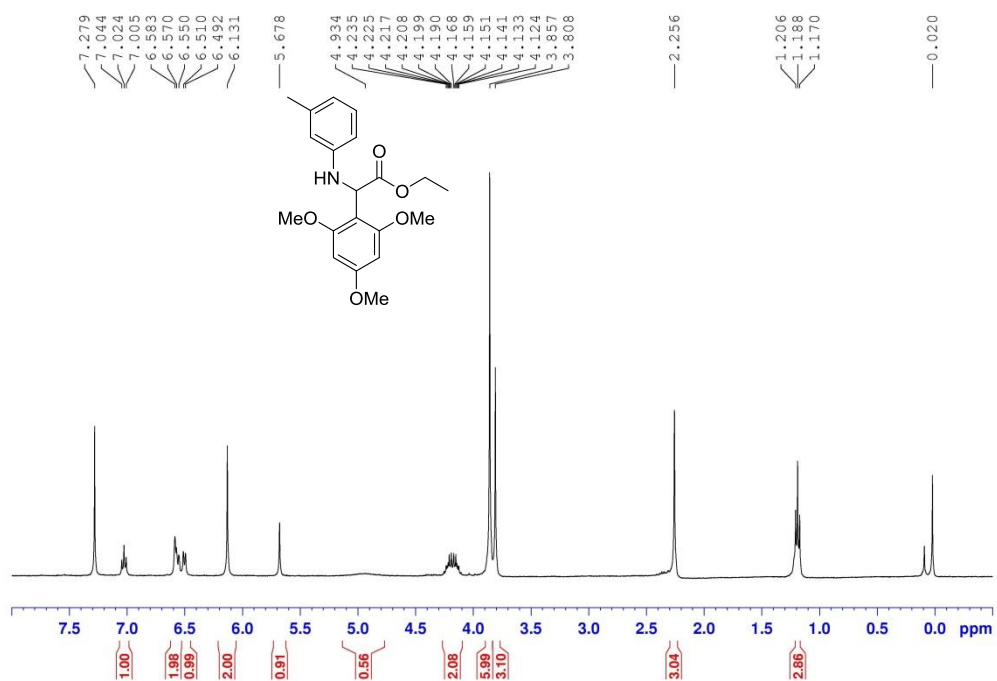
¹H NMR spectra of ethyl 2-(2-tolylamino)-2-(2,4,6-trimethoxyphenyl)acetate (3ga) [2]



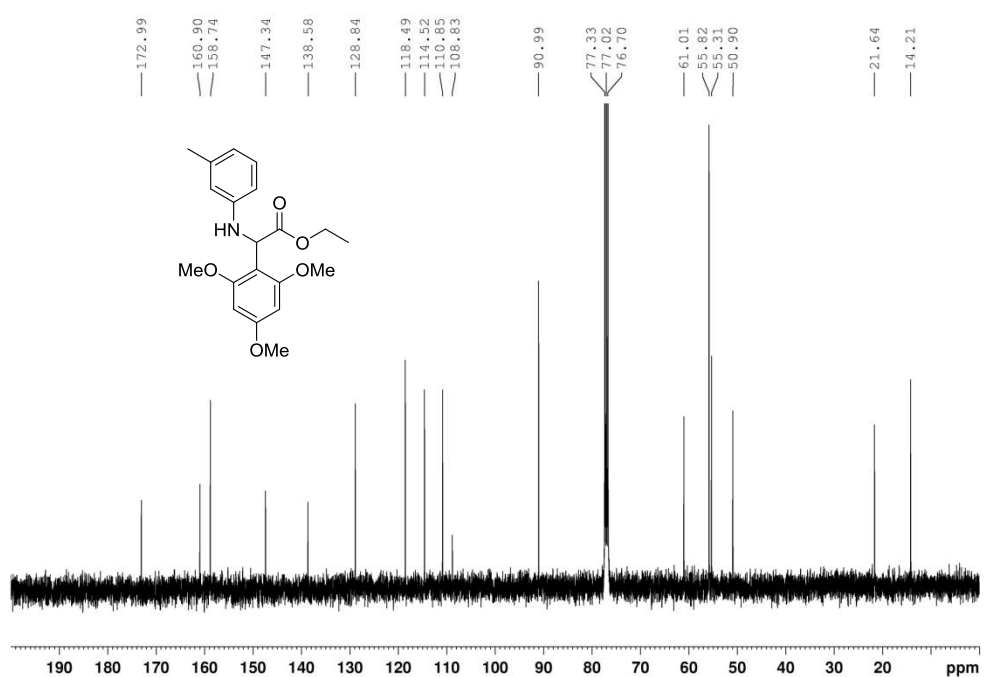
¹³C NMR spectra of ethyl 2-(2-tolylamino)-2-(2,4,6-trimethoxyphenyl)acetate (3ga) [2]



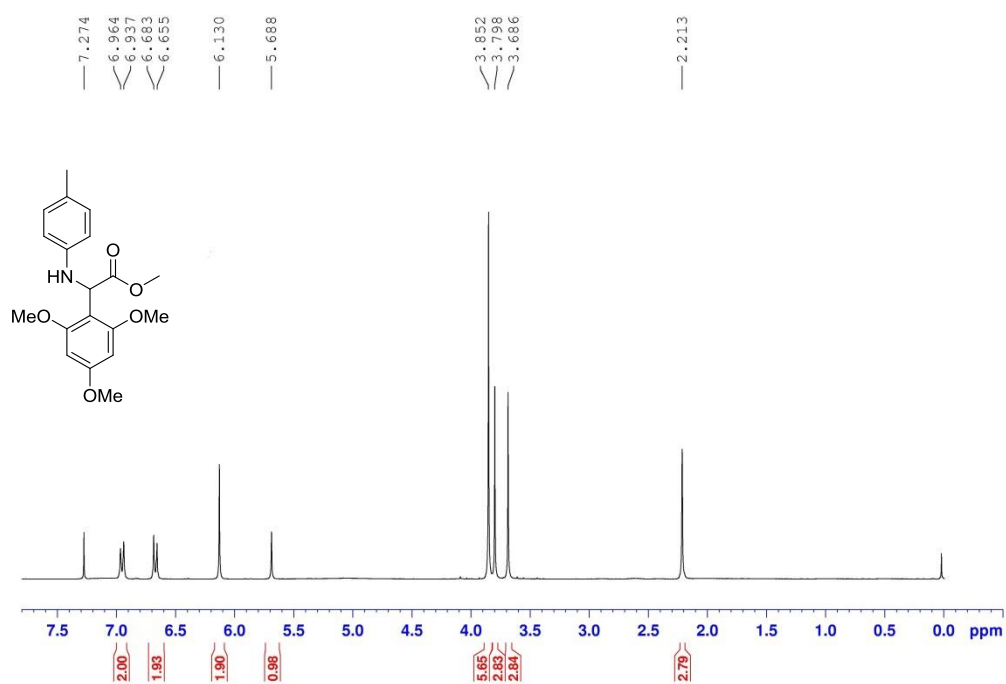
¹H NMR spectra of ethyl 2-(3-tolylamino)-2-(2,4,6-trimethoxyphenyl)acetate (3ha) [2]



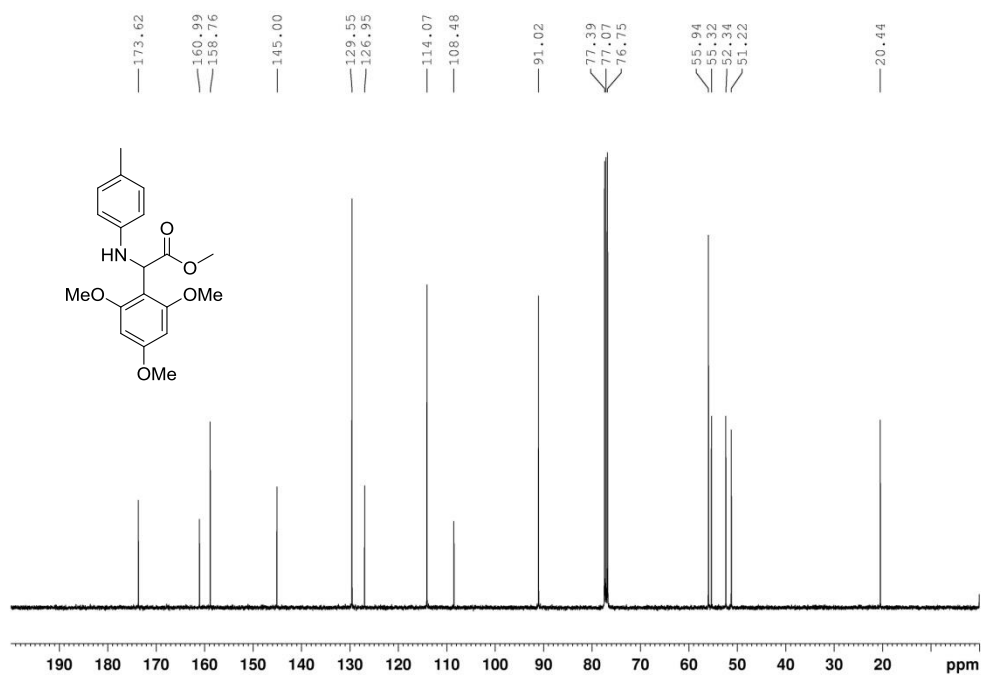
¹³C NMR spectra of ethyl 2-(3-tolylamino)-2-(2,4,6-trimethoxyphenyl)acetate (3ha) [2]



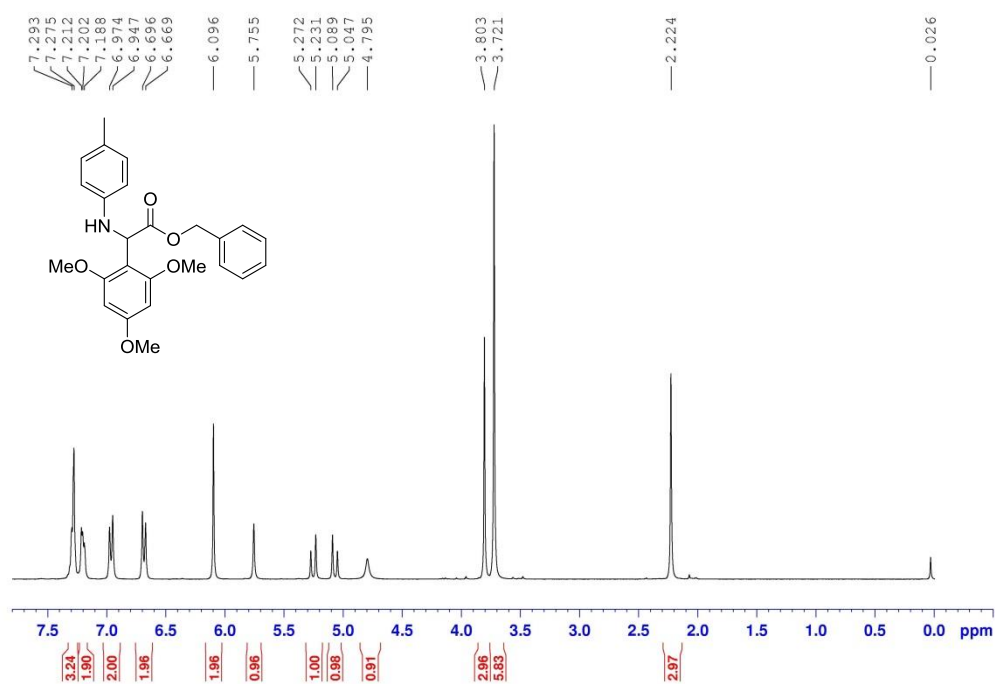
¹H NMR spectra of methyl 2-(4-tolylamino)-2-(2,4,6-trimethoxyphenyl)acetate (3ia) [2]



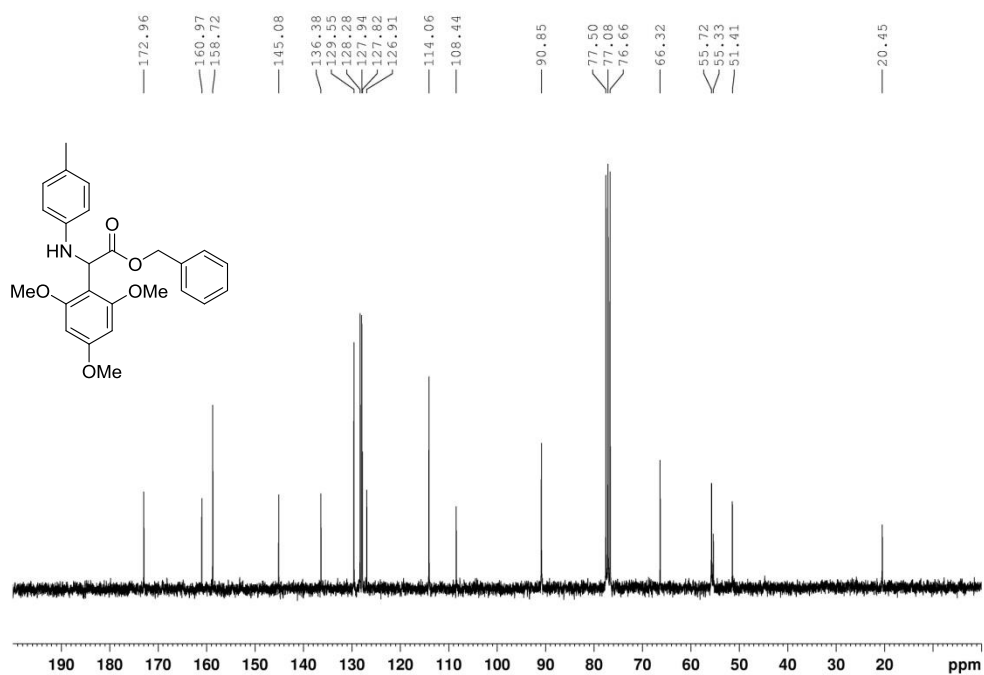
¹³C NMR spectra of methyl 2-(4-tolylamino)-2-(2,4,6-trimethoxyphenyl)acetate (3ia) [2]



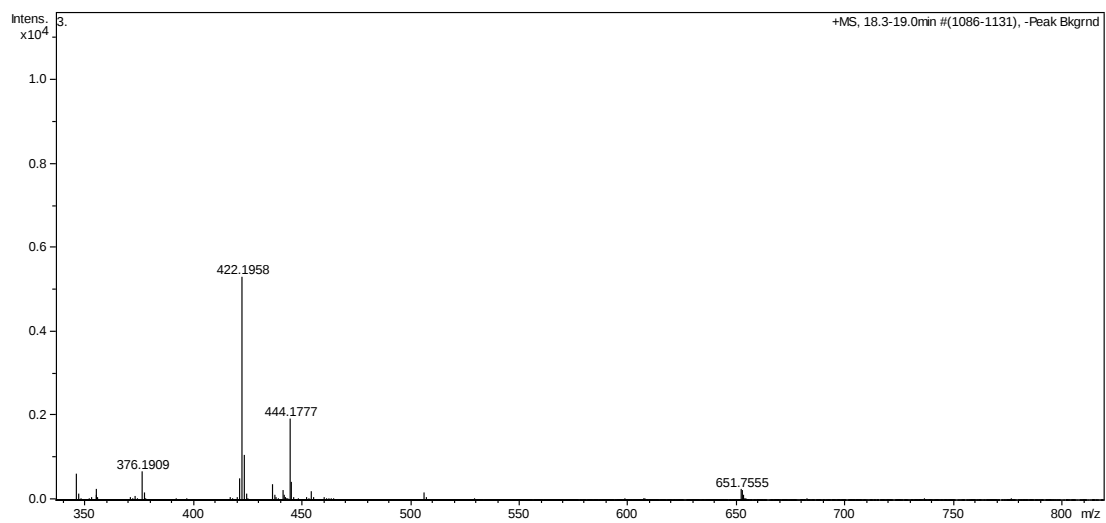
¹H NMR spectra of benzyl 2-(*p*-tolylamino)-2-(2,4,6-trimethoxyphenyl)acetate (3ja)



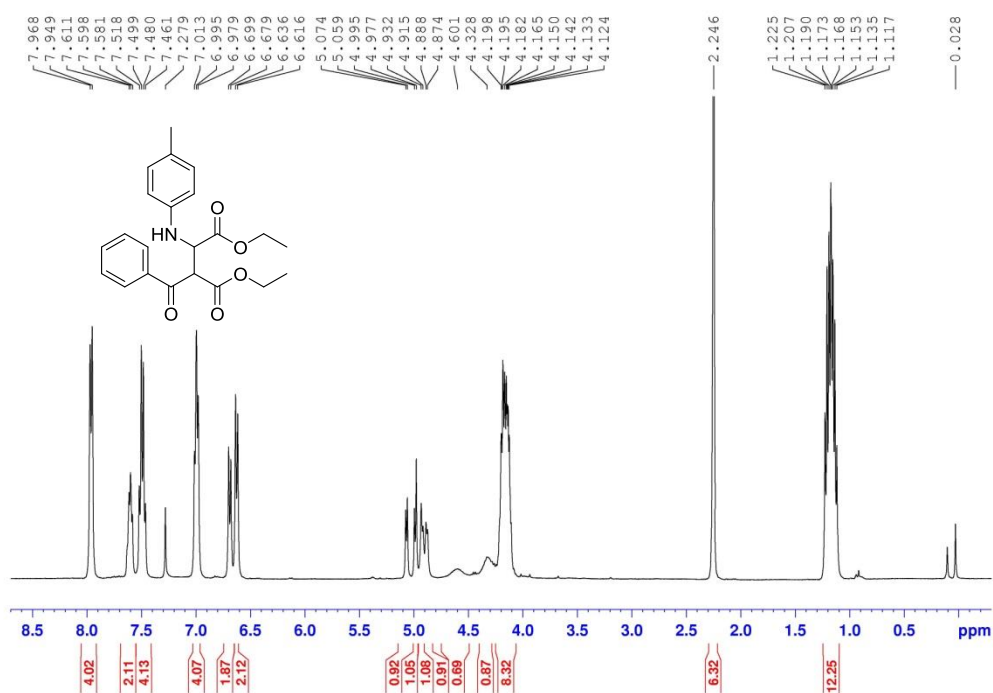
¹³C NMR spectra of benzyl 2-(*p*-tolylamino)-2-(2,4,6-trimethoxyphenyl)acetate (3ja)



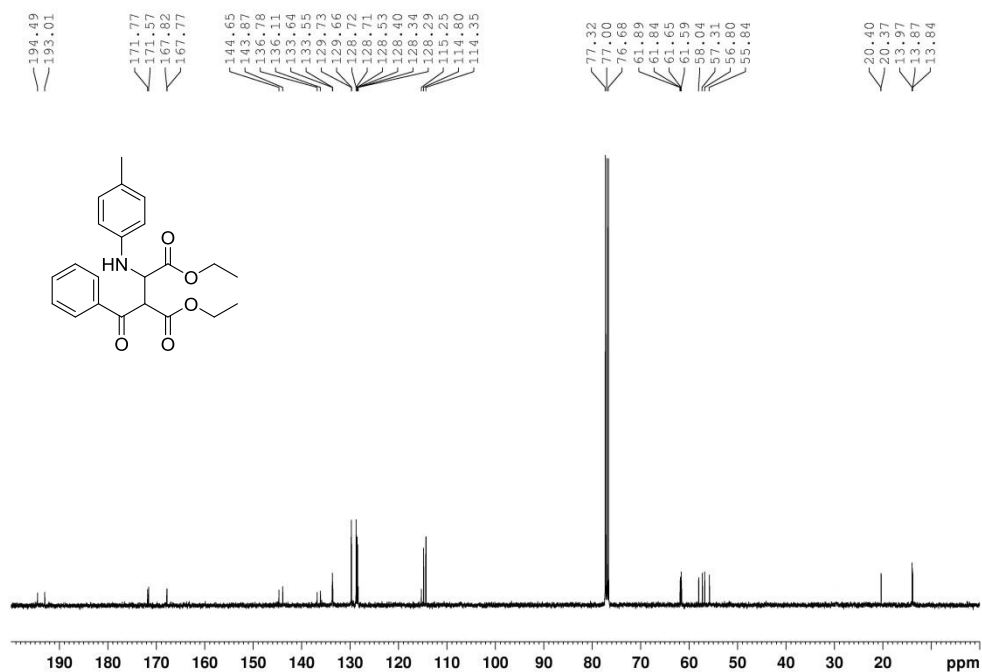
HRMS spectra of benzyl 2-(*p*-tolylamino)-2-(2,4,6-trimethoxyphenyl)acetate (3ja)



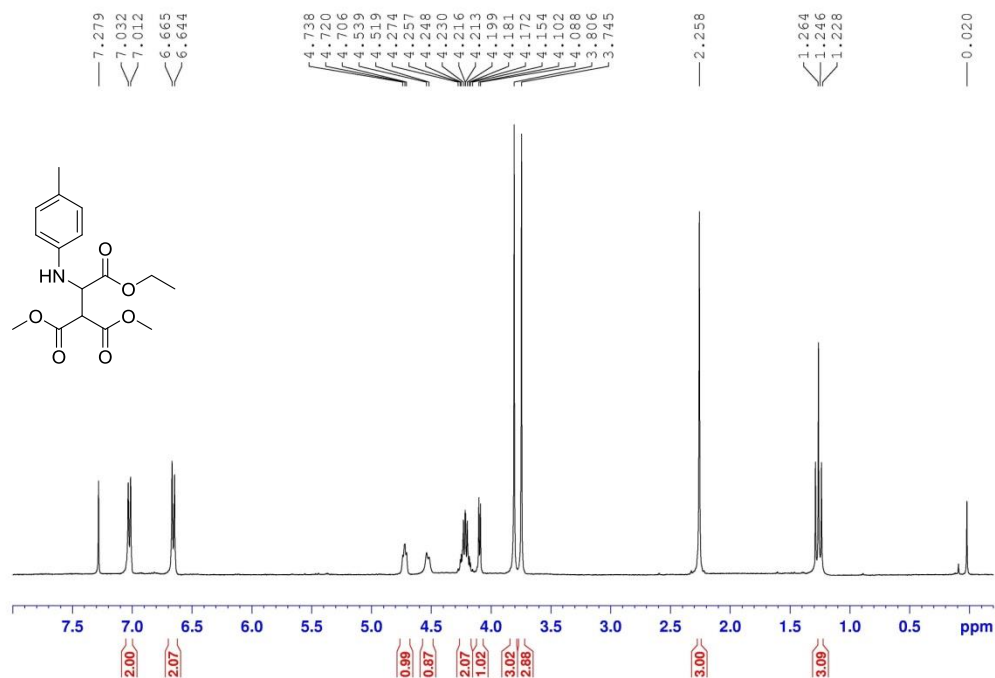
¹H NMR spectra of diethyl 2-benzoyl-3-(*p*-tolylamino)succinate (3ab) [3]



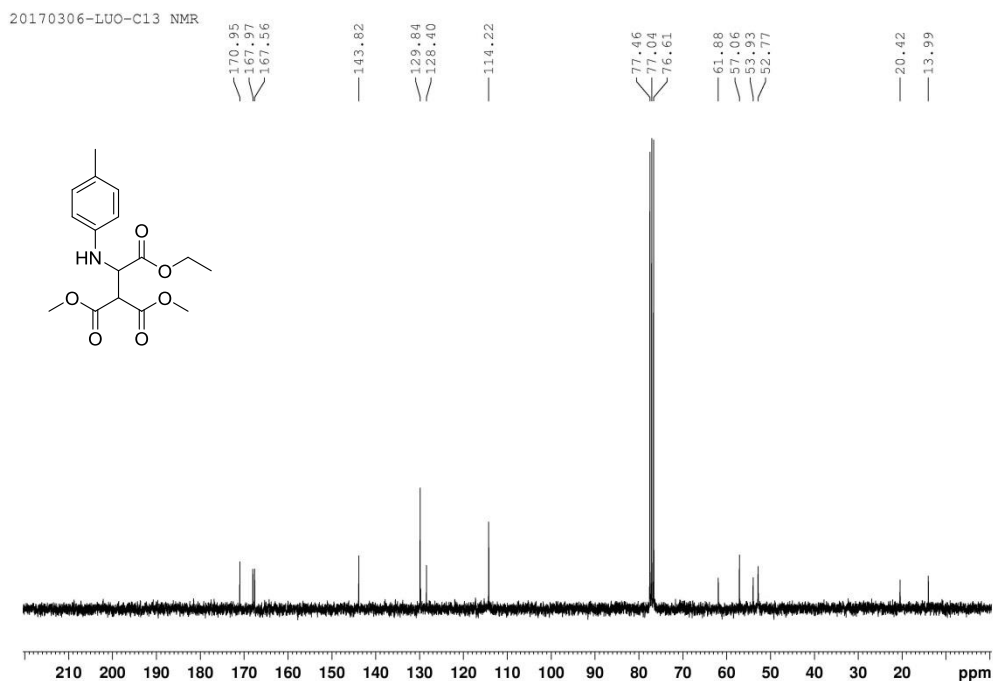
¹³C NMR spectra of diethyl 2-benzoyl-3-(*p*-tolylamino)succinate (3ab) [3]



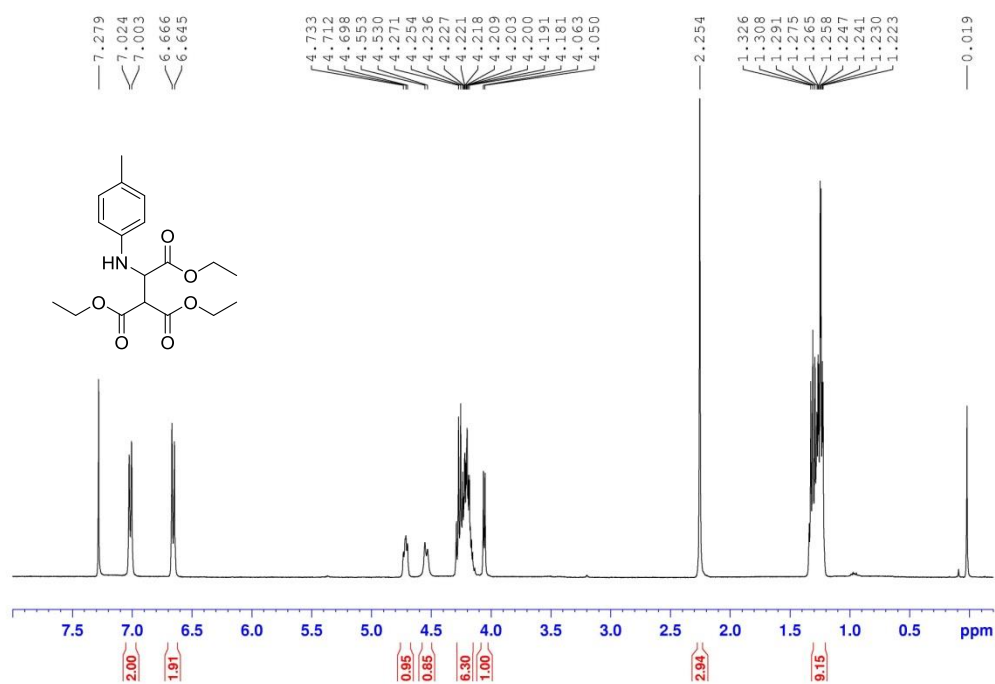
¹H NMR spectra of 2-ethyl 1,1-dimethyl 2-(*p*-tolylamino)ethane-1,1,2-tricarboxylate (3ac)



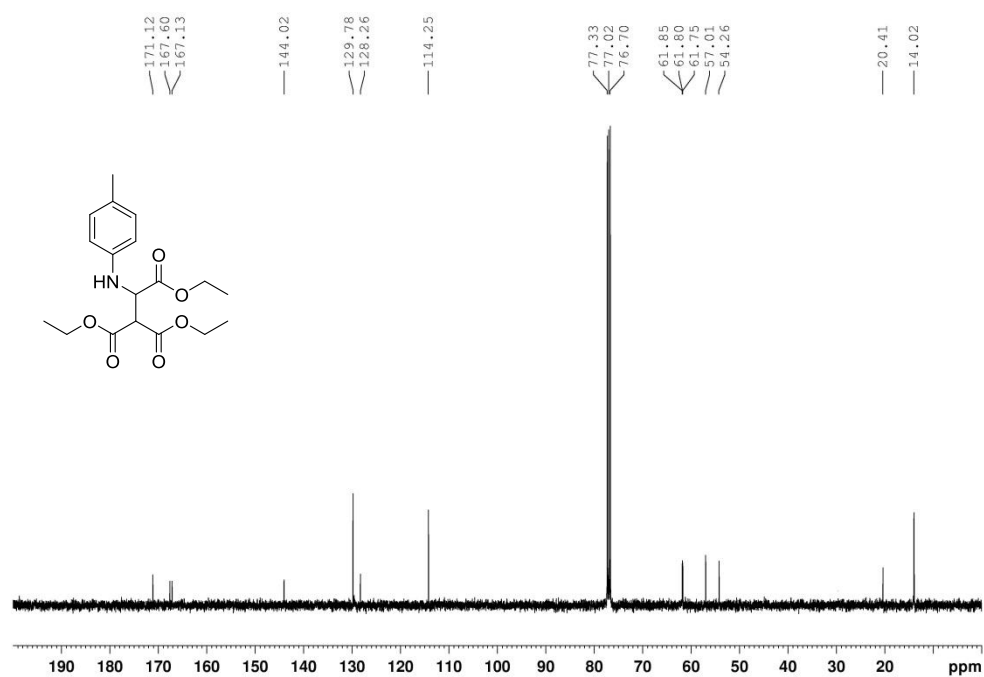
¹³C NMR spectra of 2-ethyl 1,1-dimethyl 2-(*p*-tolylamino)ethane-1,1,2-tricarboxylate (3ac)



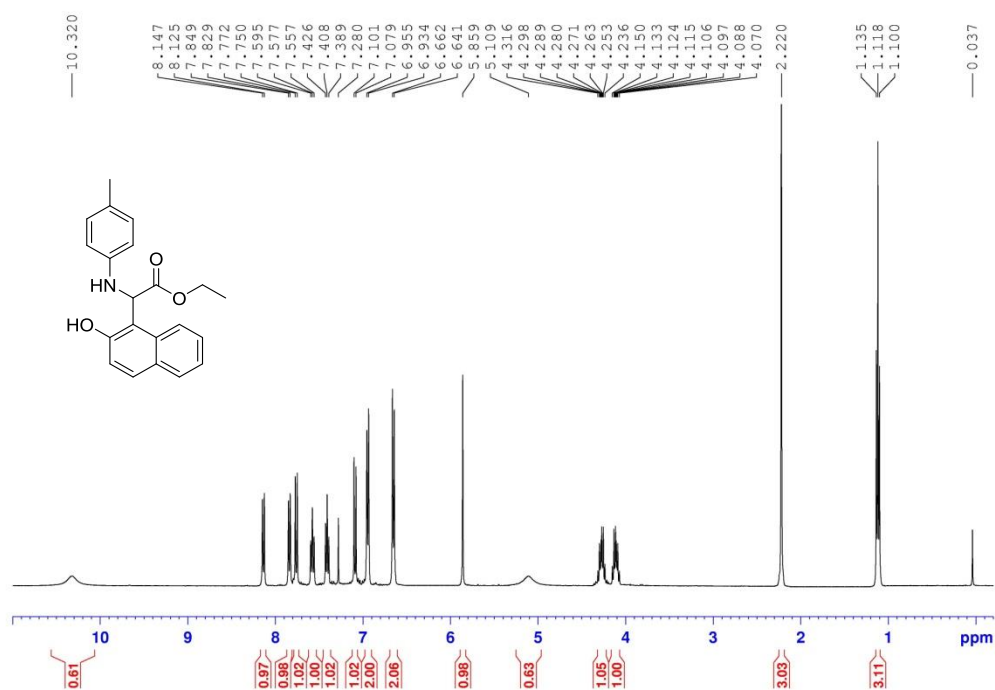
¹H NMR spectra of triethyl 2-(*p*-tolylamino)ethane-1,1,2-tricarboxylate (3ad) [4]



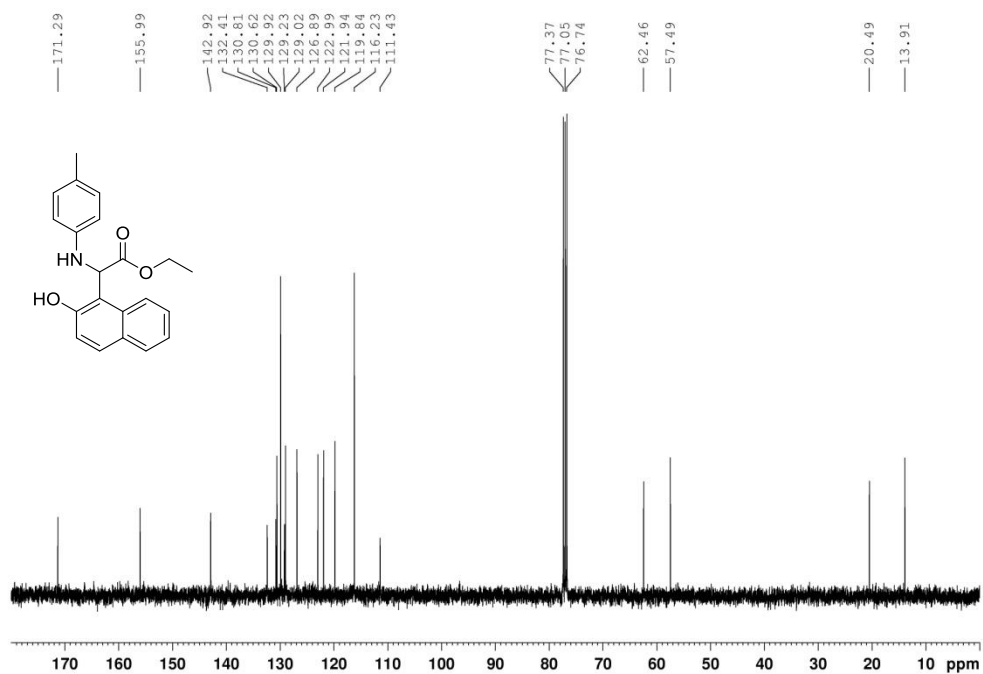
¹³C NMR spectra of triethyl 2-(*p*-tolylamino)ethane-1,1,2-tricarboxylate (3ad) [4]



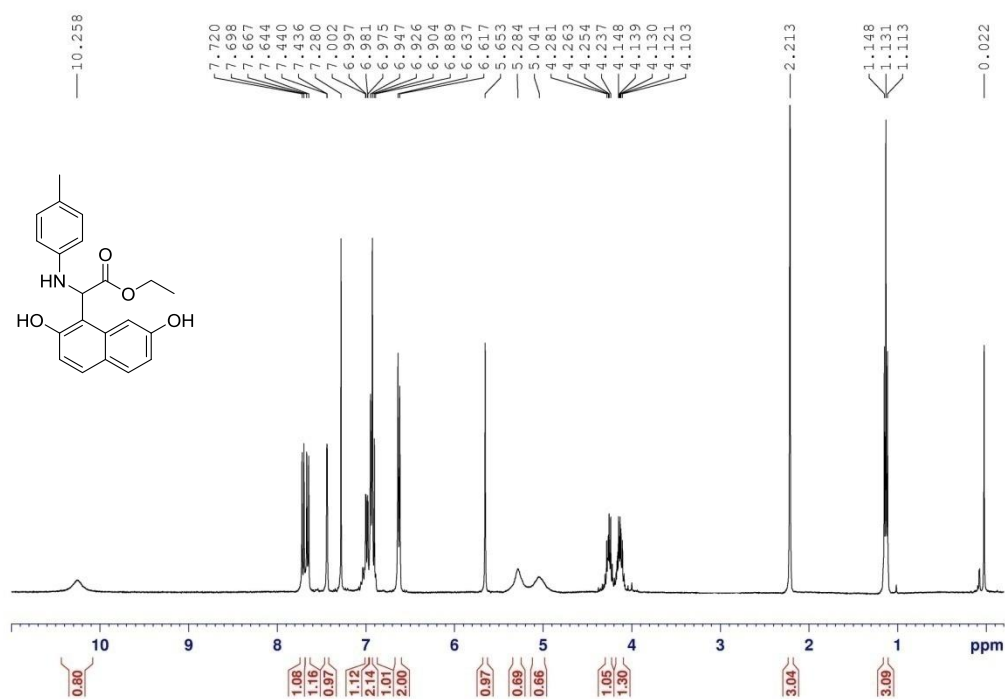
¹H NMR spectra of ethyl 2-(4-tolylamino)-2-(2-hydroxynaphthalen-1-yl)acetate (3ae) [2]



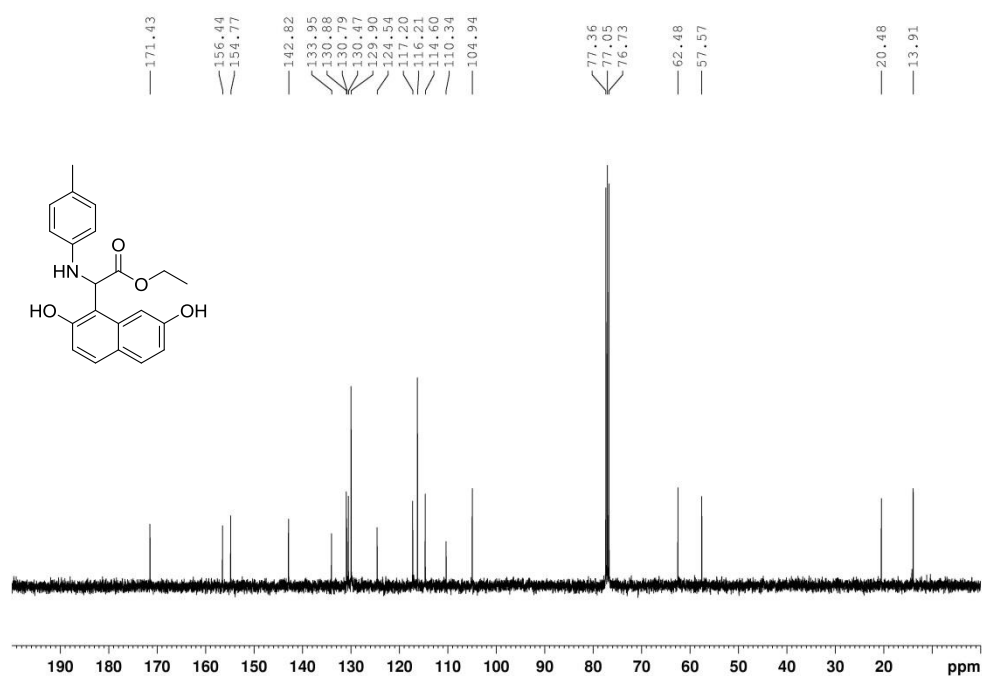
¹³C NMR spectra of ethyl 2-(4-tolylamino)-2-(2-hydroxynaphthalen-1-yl)acetate (3ae) [2]



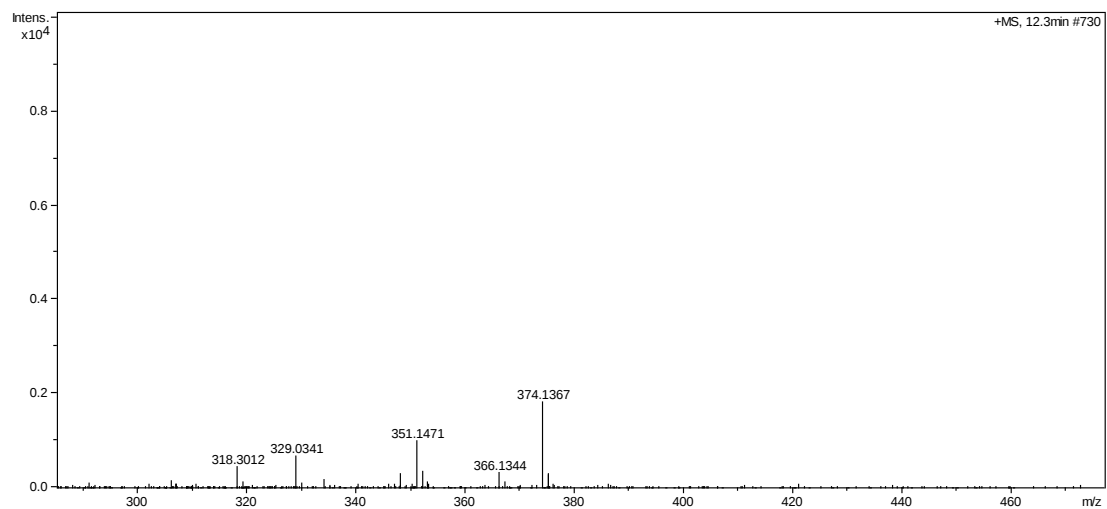
¹H NMR spectra of ethyl 2-(2,7-dihydroxynaphthalen-1-yl)-2-(*p*-tolylamino)acetate (3af)



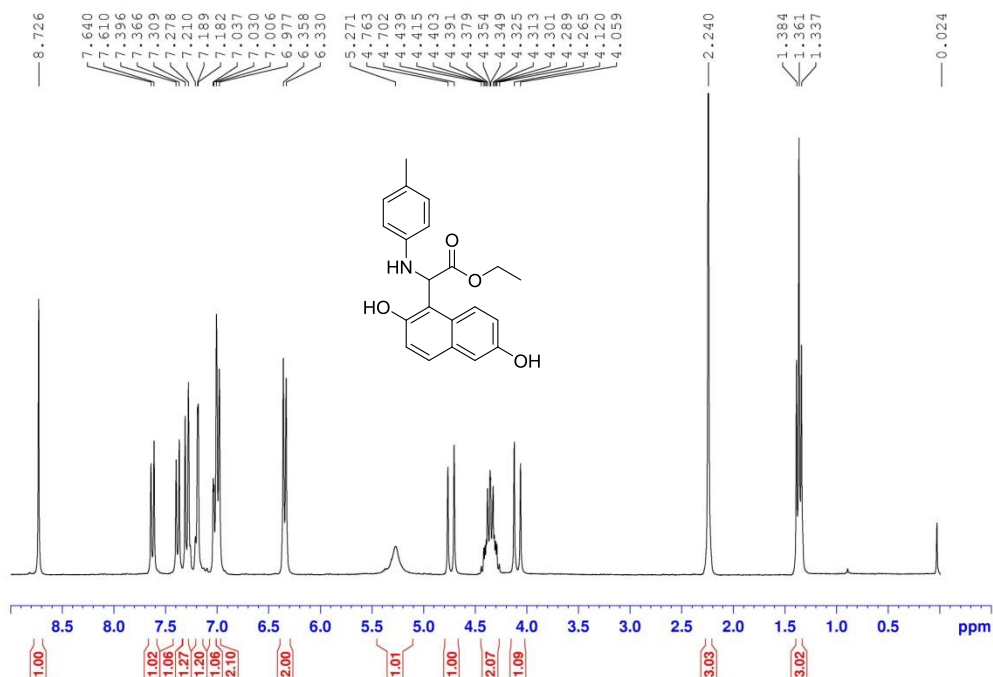
¹³C NMR spectra of ethyl 2-(2,7-dihydroxynaphthalen-1-yl)-2-(*p*-tolylamino)acetate (3af)



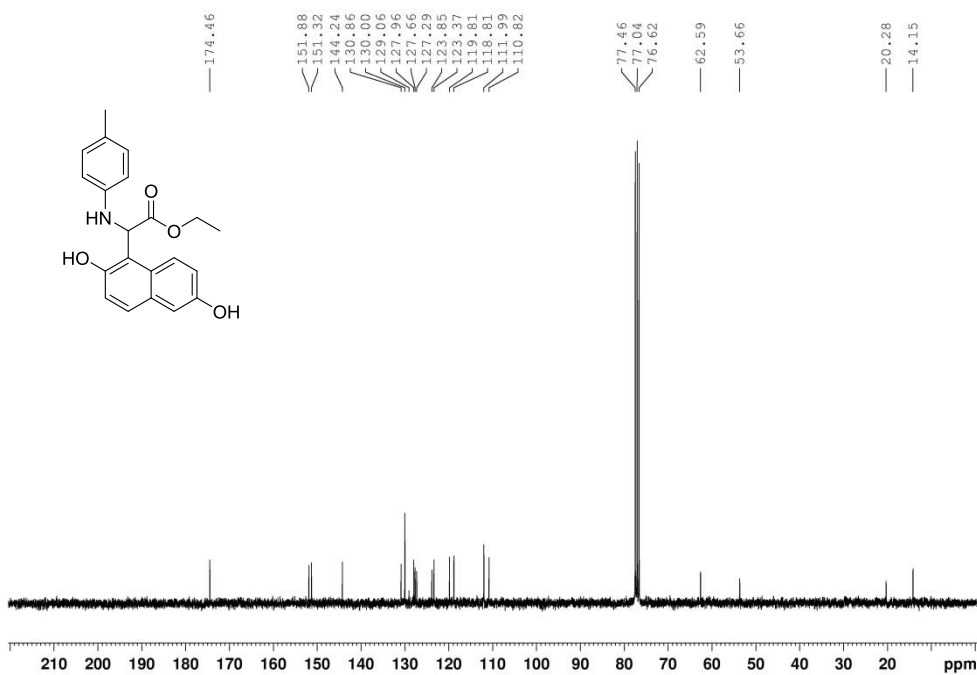
HRMS spectra of ethyl 2-(2,7-dihydroxynaphthalen-1-yl)-2-(*p*-tolylamino)acetate (3af)



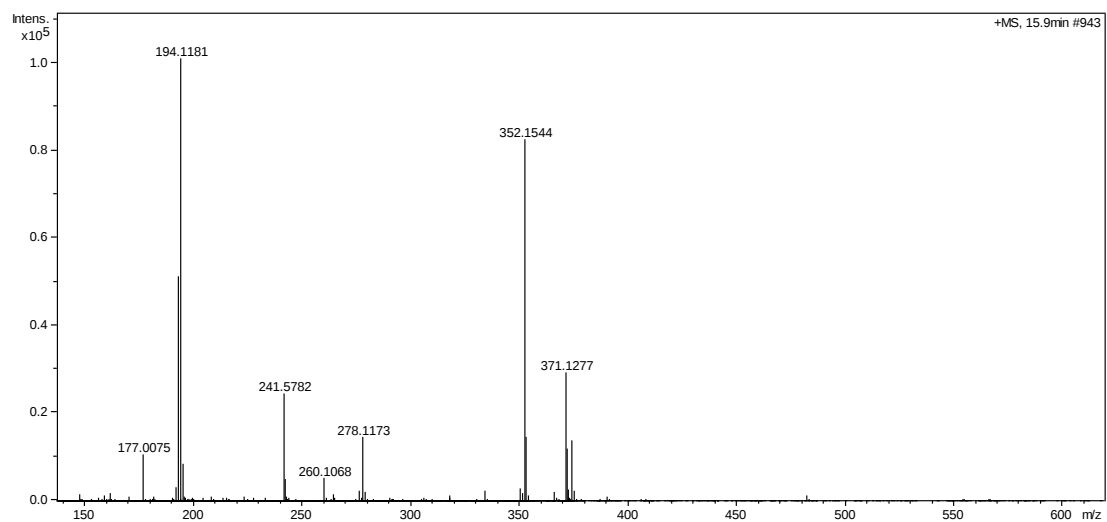
¹H NMR spectra of ethyl 2-(2,6-dihydroxynaphthalen-1-yl)-2-(*p*-tolylamino)acetate (3ag)



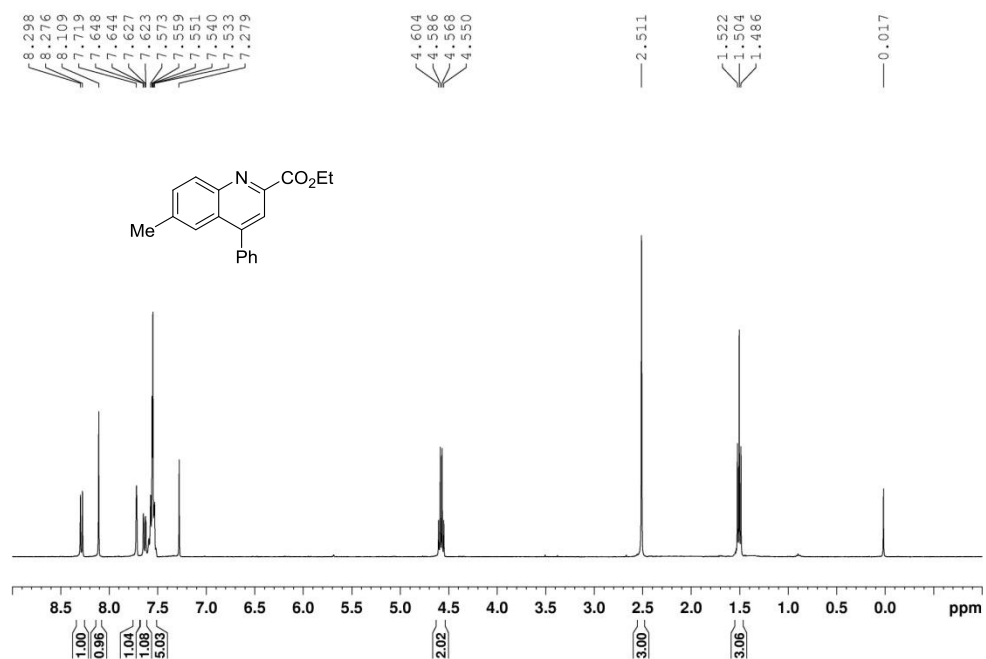
¹³C NMR spectra of ethyl 2-(2,6-dihydroxynaphthalen-1-yl)-2-(*p*-tolylamino)acetate (3ag)



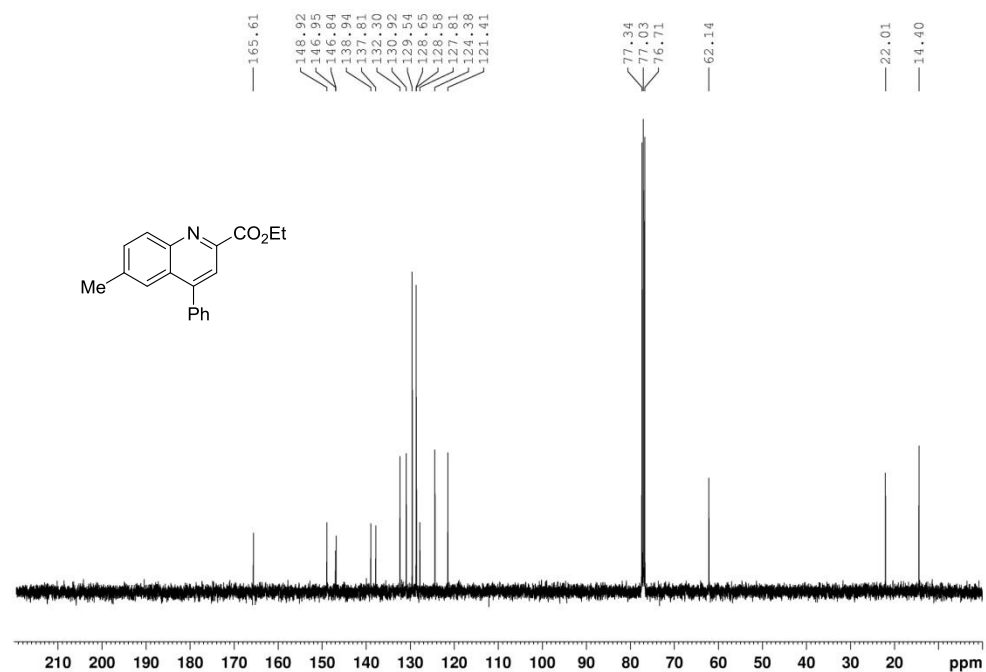
HRMS spectra of ethyl 2-(2,6-dihydroxynaphthalen-1-yl)-2-(*p*-tolylamino)acetate (3ag)



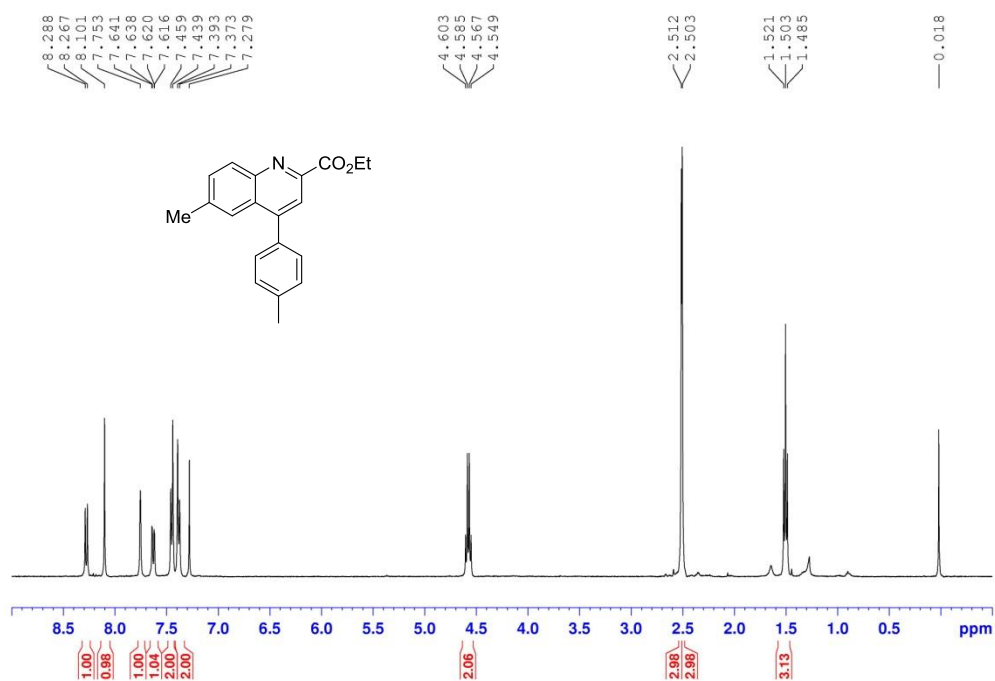
¹H NMR spectra of ethyl 6-methyl-4-phenylquinoline-2-carboxylate (3ah) [5]



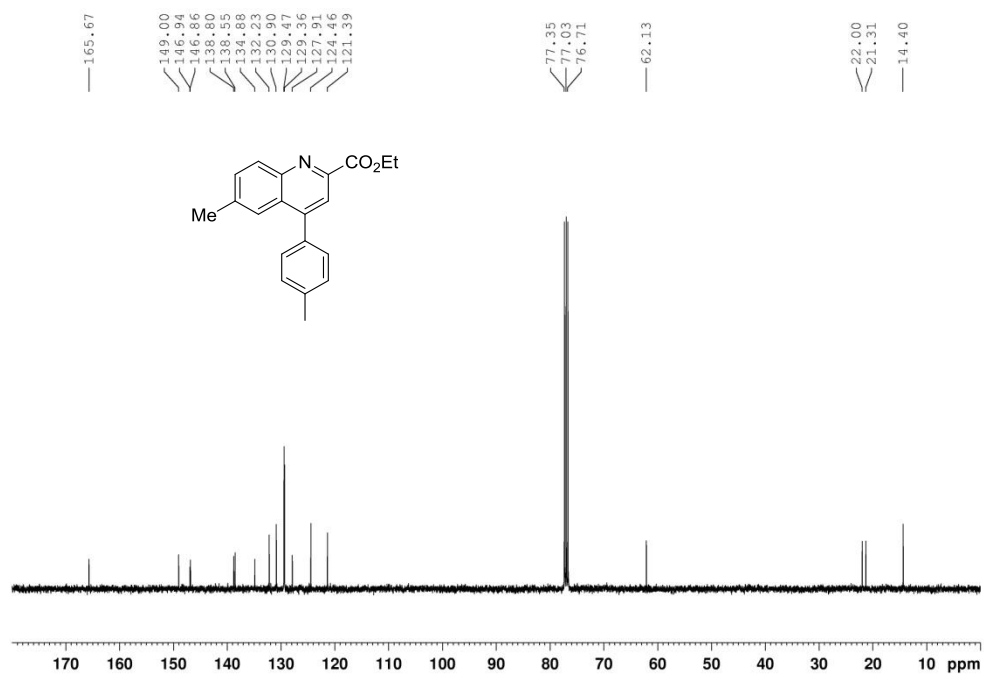
¹³C NMR spectra of ethyl 6-methyl-4-phenylquinoline-2-carboxylate (3ah) [5]



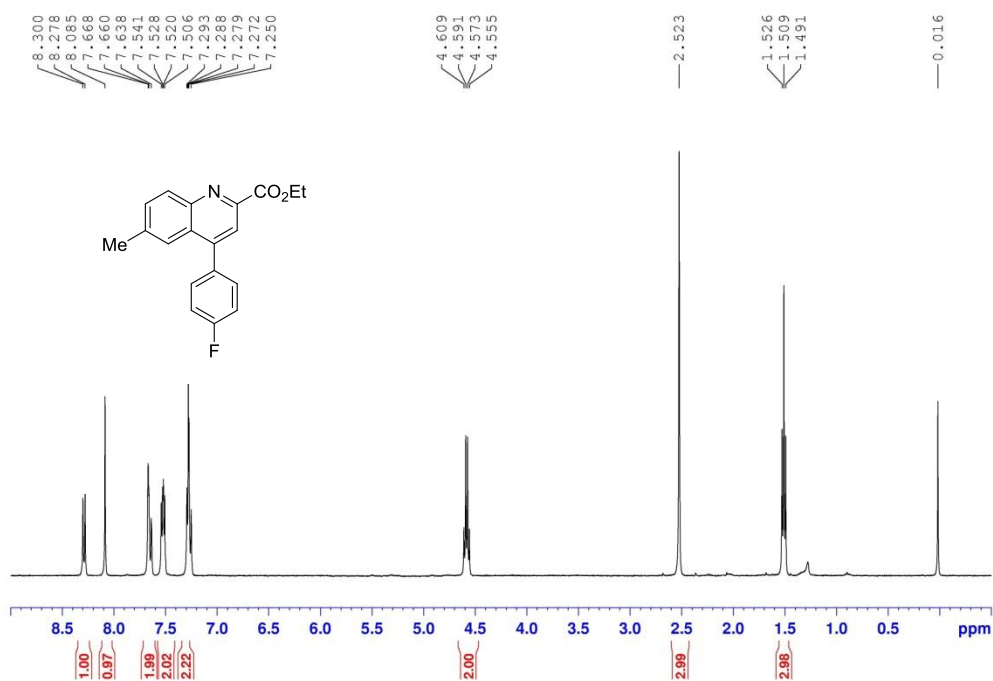
¹H NMR spectra of ethyl 6-methyl-4-(*p*-tolyl)quinoline-2-carboxylate (3ai) [6]



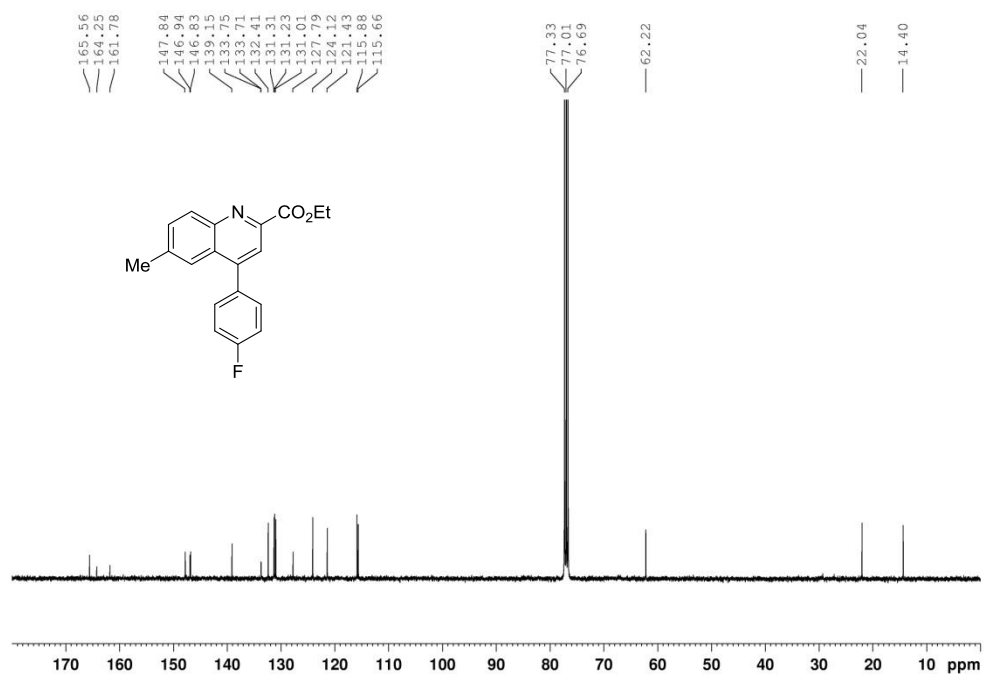
¹³C NMR spectra of ethyl 6-methyl-4-(*p*-tolyl)quinoline-2-carboxylate (3ai) [6]



¹H NMR spectra of ethyl 4-(4-fluorophenyl)-6-methylquinoline-2-carboxylate (3aj) [6]



¹³C NMR spectra of ethyl 4-(4-fluorophenyl)-6-methylquinoline-2-carboxylate (3aj) [6]



References

- 1 Rohlmann, R.; Stopka, T.; Richter, H.; Mancheno, O. G. *J. Org. Chem.***2013**, *78*, 6050.
- 2 Salman, M.; Zhu, Z.-Q.; Huang, Z.-Z. *Org. Lett.***2016**, *18*, 1526.
- 3 Wu, L.-Z.; Gao, X.-W.; Meng, Q.-Y.; Lei, T.; Zhong, J.-J.; Xiang, M.; Tong, Z.-H. Synthesis method using visible-light catalysis for secondary amine α -alkylation CN 104230734-A.
- 4 Chen, X.-M.; Li, X.-S.; Chan, A. S. C. *Chin. Chem. Lett.***2009**, *20*, 407.
- 5 Ni, M.-J.; Zhang, Y.; Gong, T.-T.; Feng, B.-N. *Adv. Synth. Catal.***2017**, *359*, 824.
- 6 Jia, X.; Peng, F.; Qing, C.; Huo, C.; Wang, X. *Org. Lett.* **2012**, *14*, 4030