

Electronic supplementary information

Functionalized 2,3-dihydrofurans via palladium-catalyzed oxyarylation of α -allyl- β -ketoesters

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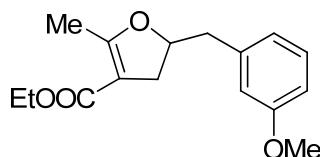
1. General information.

Reagents and methods. All the aryl halides, catalysts, bases, and solvents are commercially available and were used as purchased, without further purification. The α -allyl- β -chetoesters were prepared according to literature.¹

2. compounds 3a – 3zg.

General procedure: an oven-dried Schlenk tube equipped with a magnetic stirring bar was charged under argon with Pd₂dba₃ (11.4 mg, 0.0125 mmol), Ruphos (11.7 mg, 0.025 mmol) and anhydrous acetonitrile (1.0 ml). The resultant solution was stirred under N₂ at room temperature for 10 minutes before adding cesium carbonate (195.5 mg, 0.6 mmol), the aryl halide (93.0 mg, 0.5 mmol), the 2-allyl- β -ketoester solved in anhydrous acetonitrile (1.0 ml). The reaction mixture was warmed at 100°C and stirred for the indicated time. After cooling, the reaction mixture was diluted with AcOEt and washed twice with H₂O, and with a saturated NaCl solution. The organic layer was dried over Na₂SO₄, filtered and concentrated under reduced pressure. The residue was purified by chromatography on silica gel, eluting with a *n*-hexane/AcOEt mixture.

3 a.



2.5 h, pale yellow liquid, yield: 79%;

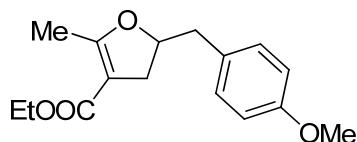
¹**H NMR** (400 MHz) (CDCl₃) δ 7.26-7.22 (m, 1 H), 6.84-6.79 (m, 3 H), 4.90-4.82 (m, 1 H), 4.17 (q, *J* = 7.2 Hz, 2 H), 3.81 (s, 3 H), 3.05 (dd, *J*₁ = 14 Hz, *J*₂ = 6.8 Hz, 1 H), 2.94 (dd, *J*₁ = 13.6 Hz, *J*₂ = 6.4 Hz, 1 H), 2.80 (m, 1 H), 2.68-2.62 (m, 1 H), 2.21 (s, 3 H), 1.28 (t, *J* = 7.2 Hz);

¹³**C NMR** (100.6 MHz) (CDCl₃) δ 167.5, 166.2, 159.7, 138.6, 129.4, 121.7, 115.2, 111.8, 101.7, 82.5, 59.4, 55.1, 42.0, 34.8, 14.5, 14.1;

IR (neat, cm⁻¹) 2935, 2836, 1695, 1646, 1513, 1384, 1247, 1228, 1081, 1035, 975 cm⁻¹;

MS m/z (relative intensity) 109 (9.8%), 155 (15.1%), 276 (M⁺ 22.5%), 83 (24.1%), 122 (100%); Anal. Calcd. For C₁₆H₂₀O₄; C, 69.54; H, 7.30; Found C, 69.65; H, 7.32.

3 b.



4.5 h, pale yellow liquid, yield: 64%;

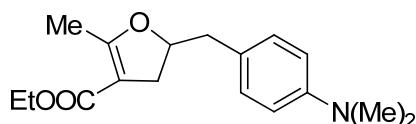
¹**H NMR** (400 MHz) (CDCl₃) δ 7.17-7.15 (m, 2 H), 6.88-6.86 (m, 2 H), 4.86-4.78 (m, 1 H), 4.20-4.15 (m, 2 H), 3.81 (s, 3 H), 3.00 (dd, *J*₁ = 14 Hz, *J*₂ = 6.8 Hz, 1 H), 2.95-2.91 (m, 1 H), 2.89-2.79 (m, 1 H), 2.66-2.60 (m, 1 H), 2.20 (s, 3 H), 1.28 (t, *J* = 7.2 Hz);

¹³**C NMR** (100.6 MHz) (CDCl₃) δ 167.6, 166.3, 158.4, 130.3, 129.0, 113.9, 101.6, 82.8, 59.4, 55.2, 41.1, 34.7, 14.5, 14.2;

IR (neat, cm⁻¹) 2935, 2836, 1695, 1646, 1612, 1583, 1513, 1442, 1384, 1247, 1228, 1081, 1035, 975, 873, 763;

MS m/z (relative intensity) 147 (4.6%), 231 (8.1%), 65 (8.5%), 109 (9.8%), 155 (15.1%), 134 (16.2%), 91 (19.8%), 55 (22.4%), 276 (M⁺ 22.5%), 83 (24.1%), 122 (100%); Anal. Calcd. For C₁₆H₂₀O₄; C, 69.54; H, 7.30; Found C, 69.64; H, 7.32.

3 c.



5 h, Yellow liquid, yield: 33%;

¹H NMR (400 MHz) (CDCl_3) δ 7.12 (d, J = 8.8 Hz, 2 H), 6.73 (d, J = 8.8 Hz, 2 H), 4.86-4.78 (m, 1 H), 4.21-4.15 (m, 2 H), 3.02-2.88 (m, 8 H), 2.78 (dd, J_1 = 14.0 Hz, J_2 = 6.8 Hz, 1 H), 2.68-2.62 (m, 1 H), 2.22 (s, 3 H), 1.29 (t, J = 7.2 Hz 3 H);

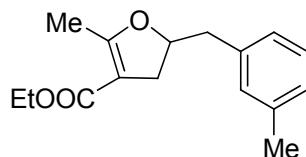
¹³C NMR (100.6 MHz) (CDCl_3) δ 167.7, 166.4, 149.4, 130.0, 125.0, 112.9, 101.6, 83.2, 59.4, 41.0, 40.8, 34.7, 14.5, 14.2;

IR (neat, cm^{-1}) 2923, 1695, 1644, 1521, 1444, 1384, 1226, 1081, 973, 763;

MS m/z (relative intensity) 65 (2.4%), 55 (4.5%), 91 (7.3%), 289 (M^+ 9.2%), 118 (12.9%), 134 (100%)

Anal. Calcd. For $\text{C}_{17}\text{H}_{23}\text{NO}_3$ C, 70.56; H, 8.01; N, 4.84; Found C, 70.65; H, 8.00; N, 4.85.

3 d.



5 h, yellow liquid, yield: 79%;

¹H NMR (400 MHz) (CDCl_3) δ 7.25-7.21 (m, 1 H), 7.09-7.04 (m, 3 H), 4.91-4.83 (m, 1 H), 4.22-4.16 (m, 2 H), 3.08-3.03 (m, 1 H), 2.97-2.91 (m, 1 H), 2.91-2.83 (m, 1 H), 2.82-2.63 (m, 1 H), 2.43 (s, 3 H), 2.22 (t, J = 1.6 Hz, 3 H), 1.32-1.28 (t, J = 7.2 Hz, 3 H);

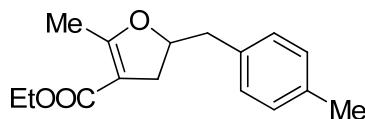
¹³C NMR (100.6 MHz) (CDCl_3) δ 167.6, 166.3, 138.0, 136.9, 130.1, 128.4, 127.4, 126.3, 101.7, 82.7, 59.4, 42.0, 34.8, 21.4, 14.5, 14.2;

IR (neat, cm^{-1}) 2925, 1697, 1648, 1444, 1384, 1226, 1081, 975

MS m/z (relative intensity) 260 (M^+ 13.5%), 127 (20.5 %), 91 (34.9%), 155 (37.7%), 83 (29.9%), 106 (100%);

Anal. Calcd. For $\text{C}_{16}\text{H}_{20}\text{O}_3$ C, 73.82; H 7.74; Found C, 73.75; H, 7.75.

3 e.



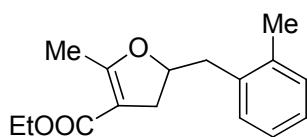
4.5 h, yellow liquid, yield: 73%;

¹H NMR (400 MHz) (CDCl_3) δ 7.14-7.12 (m, 4 H), 4.89-4.81 (m, 1 H), 4.22-4.16 (m, 2 H), 3.07-3.02 (m, 1 H), 2.97-2.90 (m, 1 H), 2.87-2.62 (m, 1 H), 2.36 (s, 3 H), 2.22 (t, J = 1.6 Hz, 3 H), 1.31-1.28 (t, J = 7.2 Hz, 3 H);

¹³C NMR (100.6 MHz) (CDCl_3) δ 167.6, 166.3, 136.1, 133.9, 129.2, 129.1, 101.6, 82.8, 59.4, 41.6, 34.8, 21.0, 14.5, 14.2;

IR (neat, cm^{-1}) 2925, 1697, 1648, 1446, 1384, 1226, 1081, 975 cm^{-1} ;
MS m/z (relative intensity) 127 (24.4%), 260 (M^+ 25.9%), 91 (32.9%), 155 (35.0%), 83 (38.7%), 106 (100%);
Anal. Calcd. For $C_{16}H_{20}O_3$ C, 73.82; H 7.74; Found C, 73.70; H, 7.75.

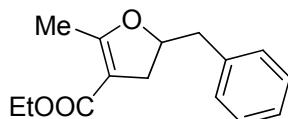
3 f.



2 h, yellow liquid, yield: 70%;

$^1\text{H NMR}$ (400 MHz) (CDCl_3) δ 7.22-7.17 (m, 4 H), 4.93-4.86 (m, 1 H), 4.20 (q, $J = 7.2 \text{ Hz}$, 2 H), 3.11 (dd, $J_1 = 14 \text{ Hz}$, $J_2 = 7.2 \text{ Hz}$, 1 H), 3.01-2.95 (m, 1 H), 2.86 (dd, $J_1 = 14.4 \text{ Hz}$, $J_2 = 6.4 \text{ Hz}$, 1 H), 2.71-2.60 (m, 1 H), 2.36 (s, 3 H), 2.22 (s, 3 H), 1.31 (t, $J = 7.2$, 3 H);
 $^{13}\text{C NMR}$ (100.6 MHz) (CDCl_3) 167.6, 166.3, 136.5, 135.5, 130.4, 129.8, 126.8, 126.0, 101.7, 82.0, 59.4, 39.2, 35.1, 19.7, 14.5, 14.2.;
IR (neat, cm^{-1}) 2977, 1697, 1648, 1461, 1384, 1326, 1261, 1226, 1081, 975, 873, 763, 744;
MS m/z (relative intensity) 171 (5.1%), 143 (6.9%), 65 (12.3%), 215 (15.2%), 117 (22.8%), 260 (M^+ 31.2%), 127 (49.1%), 91 (55.5%), 55 (59.0%), 155 (66.2%), 83 (87.6%), 106 (100%);
Anal. Calcd. For $C_{16}H_{20}O_3$ C, 73.82; H, 7.74; Found C, 73.90; H, 7.72.

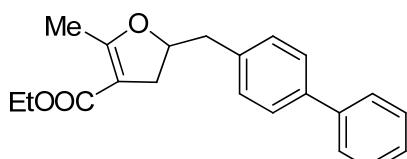
3 g.



2 h, yellow liquid, yield 95%;

$^1\text{H NMR}$ (400 MHz) (CDCl_3) δ 7.35-7.24 (m, 5 H), 4.91-4.83 (m, 1 H), 4.24-4.16 (m, 2 H), 3.08 (dd, $J_1 = 14 \text{ Hz}$, $J_2 = 6.8 \text{ Hz}$, 1 H), 2.97-2.86 (m, 2 H), 2.66 (dd, $J_1 = 14.8 \text{ Hz}$, $J_2 = 7.2 \text{ Hz}$, 1 H), 2.22 (s, 3 H), 1,13 (t, $J = 6.8 \text{ Hz}$);
 $^{13}\text{C NMR}$ (100.6 MHz) (CDCl_3) δ 167.6, 166.3, 137.0, 129.4, 128.5, 126.7, 101.7, 82.6, 59.4, 42.0, 34.8, 14.5, 14.2.;
IR (neat, cm^{-1}) 2931, 1695, 1644, 1454, 1384, 1228, 1083, 873, 752, 700;
MS m/z (relative intensity) 93 (10.3%), 97 (14.6%), 115 (15.2%), 201 (15.3%), 104 (22.4%), 246 (M^+ 26.0%), 65 (33.6%), 127 (46.9%), 55 (58.1%), 155 (67.6%), 83 (78.6%), 91 (100%);
Anal. Calcd. For $C_{15}H_{18}O_3$ C, 73.15; H, 7.37; Found C, 73.32; H, 7.30.

3 h.



1.5 h, yellow solid, mp: 47-50°C, yield 82%;

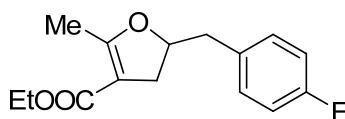
¹H NMR (400 MHz) (CDCl_3) δ 7.59 (dd, $J_1 = 15.8$ Hz, $J_2 = 7.2$ Hz, 4 H), 7.46 (t, $J = 7.2$ Hz, 2 H), 7.35 (dd, $J_1 = 18.8$ Hz, $J_2 = 7.2$ Hz, 3 H), 4.95-4.87 (m, 1 H), 4.25-4.16 (m, 2 H), 3.12 (dd, $J_1 = 14.0$ Hz, $J_2 = 6.8$ Hz, 1 H), 3.02-2.90 (m, 2 H), 2.72-2.59 (m, 1 H), 2.23 (s, 3 H), 1.30 (t, $J = 6.8$ Hz, 3 H);
¹³C NMR(100.6 MHz) (CDCl_3) 167.6, 166.3, 140.9, 139.6, 136.2, 129.8, 128.8, 127.2, 127.0, 117.5, 101.7, 82.6, 59.5, 41.7, 34.9, 14.5, 14.2;

IR (KBr, cm^{-1}) 3029, 2989, 2925, 2876, 1695, 1648, 1484, 1448, 1384, 1367, 1321, 1261, 1228, 1143, 1079, 975, 819, 761, 698;

MS m/z (relative intensity) 63 (5.4%), 91 (12.1%), 109 (12.2%), 191 (13.5%), 253 (13.5%), 322 (M^+ 21.7%), 281 (25.2%), 152 (26.0%), 127 (27.8%), 55 (41.7%), 83 (51.4%), 167 (52.9%), 207 (66.1%), 168 (100%);

Anal. Calcd. For $\text{C}_{21}\text{H}_{22}\text{O}_3$ C, 78.23; H, 6.88; Found C, 78.32; H, 6.86.

3 i.



5 h, white solid, mp: 52-54 °C, yield: 70%;

¹H NMR (400 MHz) (CDCl_3) δ 7.21-7.18 (m, 2 H), 7.03-6.99 (m, 2 H), 4.86-4.78 (m, 1 H), 4.20-4.14 (m, 2 H), 3.04-2.83 (m, 3 H), 2.65-2.59 (m, 1 H), 2.19 (t, $J = 1.6$ Hz, 3 H), 1.28 (t, $J = 6.8$ Hz, 3 H);

¹³C NMR (100.6 MHz) (CDCl_3) δ 167.5, 166.2, 161.8, (d, $J = 245$ Hz), 132.7 (d, $J = 3$ Hz), 130.8 (d, $J = 8$ Hz), 115.2, (d, $J = 21$ Hz), 101.7, 82.4, 59.5, 41.1, 34.7, 14.5, 14.1;

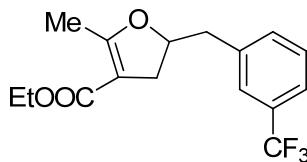
¹⁹F (376.5 MHz) (CDCl_3) δ -105.94 (s)

IR (KBr, cm^{-1}) 2923, 1693, 1654, 1508, 1380, 1324, 1261, 1218, 1147, 1091, 979, 759;

MS m/z (relative intensity) 219 (13.7%), 122(17.8%), 97 (18.6%), 264 (M^+ 31.2%), 127 (37.4%), 155 (48.0%), 55 (51.7%), 83 (85.0%), 109 (100%);

Anal. Calcd. For $\text{C}_{15}\text{H}_{17}\text{FO}_3$ C, 68.17; H, 6.48; Found C, 68.25; H, 6.46.

3 j.



2.5 h, pale yellow liquid, yield: 92%;

¹H NMR (400 MHz) (CDCl_3) δ 7.50-7.42 (m, 4 H), 4.90-4.82 (m, 1 H), 4.16 (q, $J = 6.8$ Hz, 2 H), 3.06 (dd, $J_1 = 14$ Hz, $J_2 = 7.2$ Hz, 1 H), 3.01-2.92 (m, 2 H), 2.65-2.60 (m, 1 H), 2.18 (s, 3 H) 1.27 (t, $J = 6.8$ Hz, 3 H);

¹³C NMR (100.6 MHz) (CDCl_3) δ 167.3, 165.9, 138.0, 132.8, 130.7(q, $J = 32$ Hz), 128.8, 126.0 (q, $J = 4$ Hz), 124.1 (q, $J = 272$ Hz), 123.5 (q, $J = 4$ Hz), 101.7, 81.8, 59.4, 41.7, 34.8, 14.4, 14.0;

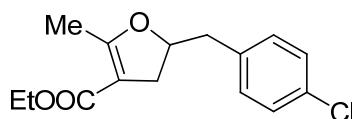
¹⁹F NMR (376.5 MHz) (CDCl_3) δ -62.59 (s)

IR (neat, cm^{-1}) 3397, 2358, 1695, 1650, 1450, 1384, 1330, 1124, 1076, 703;

MS m/z (relative intensity) 97 (20.9%), 269 (22.7%), 314 (M^+ 32.8%), 109 (48.0%), 127 (54.4%), 55 (61.1%), 155 (96.6%), 83 (100%);

Anal. Calcd. For $\text{C}_{16}\text{H}_{17}\text{F}_3\text{O}_3$; C, 61.14; H, 5.45; Found C, 61.21; H, 5.43.

3 k.



3 h, yellow liquid, yield 66%;

¹H NMR (400 MHz) (CDCl_3) δ 7.29 (d, $J = 4.4$ Hz, 2 H), 7.20 (d, $J = 8$ Hz, 2 H), 4.86-4.78 (m, 1 H), 4.22-4.14 (m, 2 H), 3.03-2.83 (m, 3 H), 2.61 (dd, $J_1 = 13.4$ Hz, $J_2 = 6.8$, 1 H), 2.19 (s, 3 H), 1.28 (t, $J = 6.8$ Hz);

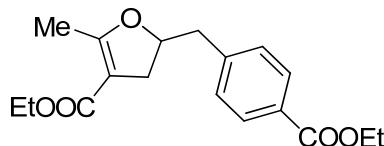
¹³C NMR(100.6 MHz) (CDCl_3) δ 167.4, 166.1, 135.5, 132.5, 130.7, 128.6, 101.7, 82.2, 59.4, 41.3, 34.8, 14.4, 14.1);

IR (neat, cm^{-1}) 2981, 2360, 2341, 1695, 1644, 1492, 1444, 1384, 1263, 1228, 1085, 873, 763, 669;

MS m/z (relative intensity) 63 (7.3%), 235 (16.3%), 109 (19.3%), 91 (24.1%), 280 (M⁺ 29.9%), 125 (52.8%), 55 (57.4%), 127 (74.3%), 155 (93.1%), 83 (100%).

Anal. Calcd. For C₁₅H₁₇ClO₃ C, 64.17; H, 6.10; Found C, 64.19; H, 6.09.

3 l.



1.25 h, yellow liquid, yield 91%;

¹H NMR (400 MHz) (CDCl_3) δ 7.99 (d, $J = 8$ Hz 2 H), 7.29 (d, $J = 8$ Hz, 2 H), 4.89-4.82 (m, 1 H), 4.37 (q, $J = 6.8$ Hz, 2 H) 4.15 (q, $J = 6.4$ Hz, 2 H), 3.07 (dd, $J_1 = 14$ Hz, $J_2 = 7.2$ Hz, 1 H) 2.97-2.90 (m, 2 H), 2.64-2.59 (m, 1 H), 2.17 (s, 3 H), 1.39 (t, $J = 7.2$ Hz, 3 H), 1.26 (t, $J = 7.2$ Hz, 3 H);

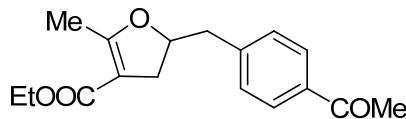
¹³C NMR(100.6 MHz) (CDCl_3) δ 167.4, 166.4, 166.1, 142.3, 129.7, 129.3, 129.0, 101.7, 82.0, 60.8, 59.4, 41.9, 34.8, 14.4, 14.3, 14.1;

IR (neat, cm^{-1}) 2981, 1714, 1648, 1612, 1446, 1384, 1369, 1276, 1228, 1178, 1105, 1022, 979, 873, 761;

MS m/z (relative intensity) 65 (7.1%), 281 (7.6%), 227 (7.6%), 118 (16.2%), 207 (16.9%), 318 (M⁺ 18.4%), 109 (18.5%), 273 (19.7%), 55 (43.2%), 91 (51.5%), 83 (56.4%), 155 (57.9%), 136 (65.1%), 164 (100%);

Anal. Calcd. For C₁₈H₂₂O₅ C, 67.91; H, 6.97; Found C, 67.99; H, 6.95.

3 m.



1 h, Yellow wax, mp: 51-53 °C, yield: 91%;

¹H NMR (400 MHz) (CDCl_3) δ 7.89 (d, $J = 8$ Hz, 2 H), 7.31 (d, $J = 8$ Hz, 2 H), 4.88-4.81 (m, 1 H), 4.13 (q, $J = 7.2$ Hz, 2 H), 3.06 (dd, $J_1 = 14$ Hz, $J_2 = 7.2$ Hz, 1 H), 2.96-2.87 (m, 2 H), 2.63-2.57 (m, 4 H), 2.15 (s, 3 H), 1.24(t, $J = 7.0$ Hz, 3 H);

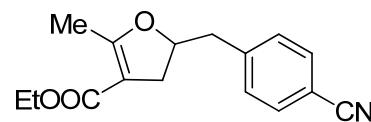
¹³C NMR (100.6 MHz) (CDCl_3) δ 197.7, 167.3, 166.0, 142.7, 135.7, 129.6, 128.5, 101.7, 81.9, 59.4, 41.9, 34.8, 26.5, 14.4, 14.1;

IR (KBr, cm^{-1}) 2958, 2902, 2867, 1697, 1679, 1648, 1604, 1267, 1224, 1145, 1087, 962, 761 cm^{-1} ;

MS m/z (relative intensity) 115 (5.4%), 243 (8.1%), 105 (11.1%), 288 (M^+ 11.2%), 155 (13.3%), 90 (14.7%), 55 (27.0%), 83 (28.1%), 134 (100 %);

Anal. Calcd. For $\text{C}_{17}\text{H}_{20}\text{O}_4$ C, 70.81; H 6.99; Found C, 70.72; H, 7.01.

3 n.



1.25 h, Yellow solid, mp: 115-117 °C, yield: 86%;

¹H NMR (400 MHz) (CDCl_3) δ 7.61(d, J = 8.2 Hz, 2 H), 7.35 (d, J = 8.0 Hz, 2 H), 4.89-4.81(m, 1 H), 4.16 (q, J = 7.2 Hz, 2 H), 3.10-2.93 (m, 3 H), 2.64-2.58 (m, 1 H), 2.17 (s, 3H), 1.28 (t, J = 7.2 Hz, 3 H);

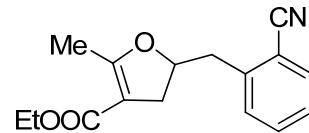
¹³C NMR (100.6 MHz) (CDCl_3) δ 167.2, 165.9, 142.7, 132.2, 130.1, 118.8, 110.7, 101.8, 81.5, 59.5, 42.0, 34.9, 14.4, 14.1;

IR (KBr, cm^{-1}) 3855, 2227, 1697, 1654, 1457, 1384, 1228, 1149, 1085, 977;

MS m/z (relative intensity) 271 (M^+ 7.0%), 109 (10.3%), 116 (14.1%), 55 (14.4%), 127 (20.5%), 83 (21.5%), 40 (22.9%), 155 (27.7%), 44 (100%).

Anal. Calcd. For $\text{C}_{16}\text{H}_{17}\text{NO}_3$ C, 70.83; H, 6.32; N, 5.16; Found C, 70.91; H, 6.30; N, 5.15.

3 o.



2.5 h, yellow solid, mp: 61-65°C, yield: 92%;

¹H NMR (400 MHz) (CDCl_3) δ 7.66 (d, J = 7.6 Hz, 1 H), 7.588 (t, J = 1.2 Hz, 1 H), 7.416-7.354 (m, 2 H), 4.946-4.870 (m, 1 H), 4.184 (q, J = 7.2 Hz, 2 H), 3.248 (dd, J_1 = 14 Hz, J_2 = 8.4 Hz, 1 H), 3.124-3.029 (m, 2 H), 2.717-2.660 (m, 1 H), 2.205(t, J = 1.2 Hz, 3 H), 1.294 (t, J = 7.2 Hz, 3 H);

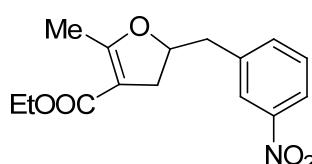
¹³C NMR (100.6 MHz) (CDCl_3) δ 167.2, 166.0, 141.1, 132.9, 132.8, 130.5, 127.3, 118.0, 113.3, 101.8, 81.3, 59.5, 40.6, 35.1, 14.4, 14.1;

IR (KBr, cm^{-1}) 2989, 2954, 2225, 2689, 2648, 1263, 1224, 1128, 1089, 979, 761;

MS m/z (relative intensity) 271 (M^+ 21.6%), 226 (22.4%), 89 (42.8%), 109 (48.5%), 55 (60.5%), 116 (67.1%), 83 (81.6%), 127 (88.0%), 155 (100%);

Anal. Calcd. For $\text{C}_{16}\text{H}_{17}\text{NO}_3$ C, 70.83; H, 6.32; N, 5.16; Found C, 70.85; H, 6.31 ; N, 5.17.

3 p.



4.5 h, Yellow solid, mp: 58-60 °C, yield: 81%;

¹H NMR (400 MHz) (CDCl_3) δ 8.14-8.12 (m, 2 H), 7.58 (d, J = 7.6 Hz, 1 H), 7.52-7.48 (m, 1 H), 4.93-4.46 (m, 1 H), 4.17 (q, J = 6.8 Hz, 2 H), 3.12 (dd, J_1 = 14.2 Hz, J_2 = 7.6 Hz, 1 H), 3.05-2.99 (m, 2 H), 2.67-2.61 (m, 1 H), 2.19 (t, J = 1.2 Hz, 3 H), 1.28 (t, J = 7.2 Hz, 3 H);

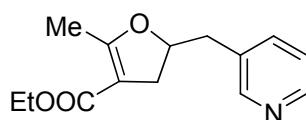
¹³C NMR (100.6 MHz) (CDCl_3) δ 167.2, 166.0, 148.3, 139.1, 135.6, 129.3, 124.2, 121.8, 101.8, 81.5, 59.5, 41.5, 34.9, 14.4, 14.0;

IR (KBr, cm^{-1}) 2925, 1698, 1654, 1429, 1382, 1351, 1230, 1089, 690;

MS m/z (relative intensity) 63 (9.5%), 115 (11.9%), 246 (16.7%), 291 (M^+ 20.8%), 90 (29.4%), 109 (35.7%), 55 (57.2%), 127 (60.8%), 83 (79.2%), 155 (100%);

Anal. Calcd. For $\text{C}_{15}\text{H}_{17}\text{NO}_5\text{C}$, C, 61.85; H, 5.88; N, 4.81; Found C, 61.93; H, 5.86; N, 4.80.

3 q.



2.4 h, Brown liquid, yield: 57%;

¹H NMR (400 MHz) (CDCl_3) δ 8.48 (br s, 2 H), 7.54 (d, J = 7.7 Hz, 1 H), 7.24 (dd, J_1 = 7.7 Hz, J_2 = 4.9 Hz, 1 H); 4.85-4.77 (m, 1 H), 4.12 (q, J = 6.8 Hz, 2 H), 3.00-2.84 (m, 3 H), 2.62-2.56 (m, 1 H), 2.14 (s, 3 H), 1.24 (t, J = 6.8 Hz, 3 H);

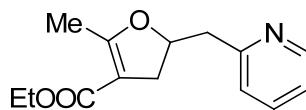
¹³C NMR (100.6 MHz) (CDCl_3) δ 167.5, 166.2, 157.4, 149.4, 136.3, 123.9, 121.7, 101.7, 81.4, 59.4, 44.3, 34.9, 14.4, 14.1;

IR (neat, cm^{-1}) 2925, 1695, 1648, 1425, 1384, 1326, 1263, 1226, 1083, 1027, 979, 763, 715, 759;

MS m/z (relative intensity) 109 (10.1%), 202 (12.0%), 130 (12.0%), 83 (17.3%), 93 (100%), 247 (M^+ 18.4%);

Anal. Calcd. For $\text{C}_{14}\text{H}_{17}\text{NO}_3\text{C}$, C, 68.0; H, 6.44; N, 6.93; Found C, 68.8; H, 6.45; N, 6.92.

3 r.

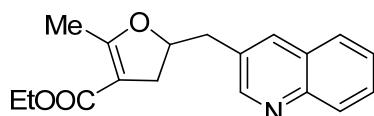


0.75 h, Brown liquid, yield: 74%;

¹H NMR (400 MHz) (CDCl_3) δ 8.535 (t, J = 0.8 Hz, 1 H), 7.625-7.582 (m, 1 H), 7.189-7.127 (m, 2 H), 5.132-5.055 (m, 1 H), 4.141 (q, J = 6.8 Hz, 2 H), 3.188 (dd, J_1 = 14 Hz, J_2 = 7.6 Hz, 1 H), 3.60-2.950 (m, 2 H), 2.692-2.634 (m, 1 H), 2.159 (t, J = 1.6 Hz, 3 H), 1.251 (t, J = 7.2 Hz, 3 H)

¹³C NMR (100.6 MHz) (CDCl_3) δ 167.5, 166.2, 157.4, 149.4, 136.3, 123.9, 121.7, 101.7, 81.4, 59.4, 44.3, 34.9, 14.4, 14.1;
IR (neat, cm^{-1}) 2927, 1957, 1695, 1644, 1589, 1436, 1384, 1228, 1081, 975, 761 cm^{-1} ;
MS m/z (relative intensity) 247 (M^+ 0.1%), 202 (2.6%), 65 (3.4%), 130 (7.2%), 93 (100%);
Anal. Calcd. For $\text{C}_{14}\text{H}_{17}\text{NO}_3$ C, 68.00; H 6.93; Found C, 68.15; H, 6.91.

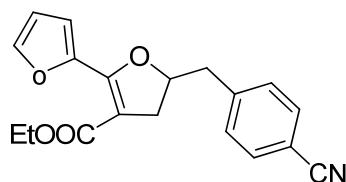
3 s.



2.75 h, Yellow liquid, yield: 75%;

¹H NMR (400 MHz) (CDCl_3) δ 8.79 (d, J = 2.0 Hz, 1 H), 8.08 (d, J = 8.8 Hz, 1 H), 7.97 (s, 1 H), 7.76 (d, J = 8.4 Hz, 1 H), 7.68-7.64 (m, 1 H), 7.51 (t, J = 7.6 Hz, 1 H), 4.94-4.87 (m, 1 H), 4.13 (q, J = 7.2 Hz, 2 H), 3.15 (dd, J_1 = 14.4 Hz, J_2 = 7.2 Hz, 1 H), 3.07-2.95 (m, 2 H), 2.69-2.63 (m, 1 H), 2.17 (s, 3 H), 1.23 (t, J = 7.2 Hz, 3 H);
¹³C NMR (100.6 MHz) (CDCl_3) δ 167.3, 166.0, 152.1, 147.1, 135.7, 129.9, 129.2, 129.1, 127.9, 127.4, 126.7, 101.8, 81.8, 59.5, 39.2, 34.9, 14.4, 14.1;
IR (neat, cm^{-1}) 2927, 1695, 1650, 1494, 1444, 1382, 1326, 1261, 1224, 1083, 977, 755;
MS m/z (relative intensity) 252 (6.0%), 155 (6.3%), 207 (6.8%), 127 (8.9%), 180 (11.2%), 297 (M^+ 15.0%), 83 (17.5%), 55 (18.5%), 115 (23.2%) 143 (100%);
Anal. Calcd. For $\text{C}_{18}\text{H}_{19}\text{NO}_3$ C, 72.71; H, 6.44; N, 4.71; Found C, 72.65; H, 6.45; N, 4.70.

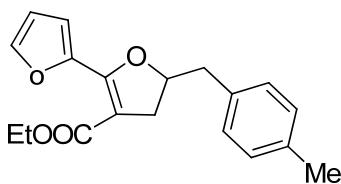
3 t.



0.75 h, pale yellow solid, mp: 59-62 °C, yield 84%;

¹H NMR (400 MHz) (CDCl_3) δ 8.43 (s, 1 H), 7.61 (d, J = 8.0 Hz, 2 H), 7.41-7.35 (m, 3 H), 6.91 (s, 1 H), 4.95-4.87 (m, 1 H), 4.20 (q, J = 7.2 Hz, 2 H), 3.20-3.11 (m, 2 H), 2.99 (dd, J_1 = 14.0 Hz, J_2 = 5.2 Hz, 1 H), 2.79 (dd, J_1 = 15.2 Hz, J_2 = 7.2 Hz, 1 H), 1.31-1.26 (m, 3 H);
¹³C NMR (100.6 MHz) (CDCl_3) δ 165.2, 157.7, 146.8, 142.8, 142.5, 132.2, 130.2, 118.8, 116.3, 110.7, 110.0, 101.1, 80.9, 59.8, 42.0, 36.4, 14.5;
IR (KBr, cm^{-1}) 3160, 2979, 2931, 2227, 1695, 1633, 1504, 1446, 1369, 1332, 1249, 1157, 1103, 1074, 908, 873, 817, 763, 601;
MS m/z (relative intensity) 63 (10.0%), 278 (10.8%), 105 (22.1%), 135 (23.0%), 73 (29.2%), 323 (M^+ 44.6%), 116 (47.0%), 207 (49.1%), 161 (69.4%), 95 (100%);
Anal. Calcd. For $\text{C}_{19}\text{H}_{17}\text{NO}_3$ C, 74.25; H, 5.58; N, 4.56; Found C, 74.34; H, 5.56; N, 4.55.

3 u.



2.5 h, Yellow liquid, yield: 74%;

¹H NMR (400 MHz) (CDCl_3) δ 8.49 (d, $J = 0.8$ Hz, 1 H), 7.45-7.44 (m, 1 H), 7.17 (s, 4 H), 7.02 (dd, $J_1 = 2.0$ Hz, $J_2 = 0.7$ Hz, 1 H), 4.96-4.88 (m, 1 H), 4.30-4.18 (m, 2 H), 3.16-3.10 (m, 2 H), 2.92-2.81 (m, 2 H), 2.38 (s, 3 H), 1.33 (t, $J = 7.2$ Hz 3 H);

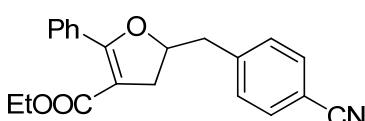
¹³C NMR (100.6 MHz) (CDCl_3) δ 165.5, 158.1, 146.8, 142.3, 136.2, 134.0, 129.3, 129.2, 116.6, 110.2, 101.1, 82.1, 59.7, 41.6, 36.3, 21.1, 14.5;

IR (neat, cm^{-1}) 3160, 2979, 2360, 1698, 1629, 1504, 1446, 1367, 1332, 1247, 1157, 1103, 1074, 873, 806, 763, 599;

MS m/z (relative intensity) 134 (19.8%), 51 (22.7%), 117 (23.1%), 207 (38.2%), 77 (48.2%), 312 (M^+ 51.5%), 95 (87.0%), 105 (100%);

Anal. Calcd. For $C_{18}H_{20}O_2$: C, 77.0%; H, 6.80%. Found: C, 76.8%; H, 6.81.

3 v



2.5 h .Brown solid. mp:108-110 °C, yield: 84%;

¹H NMR (400 MHz) (CDCl₃) δ 7.754-7.733 (m, 2 H), 7.632 (d, J = 8.0 Hz, 2 H), 7.457-7.281 (m, 5 H), 5.040-4.964 (m, 1 H), 4.144 (q, J = 6.8 Hz, 2 H), 3.277-3.179 (m, 2 H), 3.062 (dd, J₁ = 14.2 Hz, J₂ = 5.6 Hz, 1 H), 2.874 (dd, J₁ = 15.2 Hz, J₂ = 7.6 Hz, 1 H), 1.216 (t, J = 7.2 Hz, 3 H);

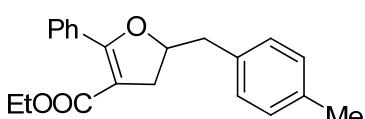
¹³C NMR (100.6 MHz) (CDCl_3) δ 165.1, 164.3, 142.7, 132.3, 130.4, 130.2, 129.8, 129.2, 127.7, 118.8, 110.7, 102.2, 81.1, 59.9, 42.0, 36.8, 14.2.

IR (KBr, cm⁻¹) 2925, 2225, 1702, 1637, 1598, 1247, 1091, 1025, 755, 698.

MS m/z (relative intensity) 63 (11.2%), 127 (17.3%), 253 (17.7%), 51 (23.4%), 281 (23.6%), 145 (28.4%), 217 (35.7%), 333 (M^+ 35.9%), 89 (36.5%), 207 (58.0%), 116 (74.2%), 77 (82.9%), 171 (85.1%), 105 (100%);

Anal. Calcd. For $C_{21}H_{19}NO_3$: C, 75.66; H, 5.74; N, 4.20; Found: C, 75.56; H, 5.73; N, 4.22.

3 w.



4 h, orange liquid, yield: 66%;

¹H NMR (400 MHz) (CDCl_3) δ 7.822-7.802 (m, 2 H), 7.444-7.388 (m, 3 H), 7.213-7.160 (m, 4 H), 5.036-4.959 (m, 1 H), 4.215-4.135 (m, 2 H), 3.215-3.153 (m, 2 H), 2.895-2.886 (m, 2 H), 2.376 (s, 3 H), 1.251-1.192 (m, 3 H);

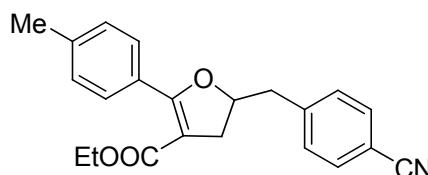
¹³C NMR (100.6 MHz) (CDCl_3) δ 165.4, 164.7, 136.2, 133.9, 130.2, 129.4, 129.3, 129.2, 128.7, 127.6, 102.2, 82.3, 59.7, 41.4, 36.5, 21.1, 14.3;

IR (neat, cm^{-1}) 2979, 2925, 1737, 1687, 1625, 1598, 1446, 1384, 1243, 1085, 873, 757, 692;

MS m/z (relative intensity) 308 (M^+ 0.6%), 188 (2.3%), 322 (3.4%), 65 (9.4%), 128 (11.1%), 51 (15.1%), 144 (16.7%), 91 (31.6%), 171 (36.6%), 115 (55.0%), 77 (67.5%), 105 (100%);

Anal. Calcd. For $\text{C}_{21}\text{H}_{22}\text{O}_3\text{C}$, 78.23; H, 6.88; Found C, 78.16; H, 6.86.

3 x.



1.25 h, yellow solid, mp: 84 - 90 °C, yield: 69%;

¹H NMR (400 MHz) (CDCl_3) δ 7.67 (d, J = 8.0 Hz, 2 H), 7.62 (d, J = 8.0 Hz, 2 H), 7.40 (d, J = 8.0 Hz, 2 H), 7.20 (d, J = 8.0 Hz, 2 H), 5.13-4.94 (m, 1 H), 4.15 (q, J = 7.2 Hz, 2 H), 3.26-3.16 (m, 2 H), 3.05 (dd, J_1 = 14.0 Hz, J_2 = 5.2 Hz, 1 H), 2.86 (dd, J_1 = 15.2 Hz, J_2 = 7.6 Hz, 1 H), 2.40 (s, 3 H), 1.24 (t, J = 6.8 Hz, 3 H);

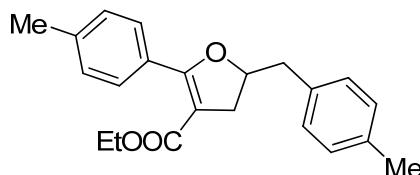
¹³C NMR (100.6 MHz) (CDCl_3) δ 165.2, 164.5, 142.8, 140.8, 132.3, 130.2, 129.2, 128.4, 126.9, 118.9, 110.7, 101.6, 80.9, 59.8, 42.0, 36.8, 21.5, 14.3;

IR (KBr, cm^{-1}) 2927, 2225, 1697, 1621, 1509, 1243, 1083, 829, 755;

MS m/z (relative intensity) 103 (12.6%), 281 (14.6%), 347 (26.9%), 158 (30.3%), 297 (34.5%), 65 (33.1%), 73 (48.3%), 129 (46.0%), 185 (51.6%), 91 (87.4%), 116 (100%);

Anal. Calcd. For $\text{C}_{22}\text{H}_{21}\text{NO}_3$, 76.06; H, 6.09; N, 4.03; Found C, 76.21; H, 6.07; N, 4.04.

3 y.



2 h, yellow liquid, yield: 70%;

¹H NMR (400 MHz) (CDCl_3) δ 7.7450 (d, J = 8 Hz, 2 H), 7.235-7.178 (m, 6 H), 5.014-4.937 (m, 1 H), 4.199-4.146 (m, 2 H), 3.203-3.142 (m, 2 H), 2.974-2.876 (m, 2 H), 2.416 (s, 3 H), 2.377 (s, 3 H), 1.271-1.201 (m, 3 H);

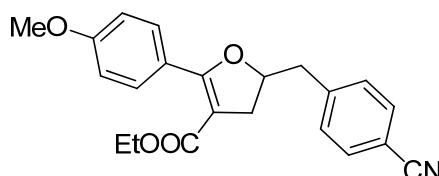
¹³C NMR (100.6 MHz) (CDCl_3) δ 165.5, 164.9, 140.5, 136.1, 134.0, 129.4, 129.3, 129.2, 128.8, 128.3, 117.3, 101.5, 82.1, 59.6, 36.6, 21.7, 21.5, 14.3;

IR (KBr, cm^{-1}) 2979, 1737, 1683, 1608, 1511, 1446, 1384, 1243, 1184, 1081, 821, 761;

MS m/z (relative intensity) 291 (9.0%), 141 (11.1%), 207 (14.4%), 65 (30.7%), 336 (M^+ 32.9%), 77 (34.6%), 158 (35.7%), 230 (47.6%), 129 (52.9%), 185 (62.9%), 105 (83.2%), 119 (86.6%), 91 (100%);

Anal. Calcd. For C₂₂H₂₄O₃ C, 78.54; H, 7.19; Found C, 78.44; H, 7.18.

3 z.



1 h, yellow liquid, yield: 51%;

¹H NMR (400 MHz) (CDCl₃) δ 7.80 (d, *J* = 8.8 Hz, 2 H), 7.62 (d, *J* = 8 Hz, 2 H) 7.39 (d, *J* = 8 Hz, 2 H), 6.91 (d, *J* = 8.8 Hz, 2 H), 4.99-4.91 (m, 1 H), 4.16 (q, *J* = 6.8 Hz, 2 H), 3.85 (s, 3 H), 3.25-3.16 (m, 2 H), 3.04 (dd, *J*₁ = 14.4 Hz, *J*₂ = 5.6 Hz, 2 H) 2.85 (dd, *J*₁ = 14.8 Hz, *J*₂ = 7.6 Hz, 2 H), 1.25 (t, *J* = 7.2 Hz, 3 H);

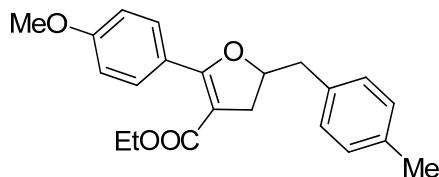
¹³C NMR (100.6 MHz) (CDCl₃) δ 165.3, 164.2, 161.3, 142.9, 132.3, 131.1, 130.2, 122.0, 118.8, 113.0, 110.7, 100.8, 80.7, 59.7, 55.3, 42.0, 36.9, 14.3;

IR (Neat, cm⁻¹) 2933, 2227, 1695, 1606, 1509, 1461, 1384, 1247, 1178, 1079, 1027, 873, 838, 763;

MS m/z (relative intensity) 267 (10.1%), 118 (10.6%), 63 (11.4%), 191 (17.1%), 103 (19.6%), 253 (22.1%), 89 (22.3%), 147 (23.5%), 246 (24.4%), 363 (M⁺ 24.8%), 174 (25.4%), 116 (29.7%), 281 (38.2%), 73 (88.3%), 135 (88.3%), 207 (100%);

Anal. Calcd. For C₂₂H₂₁NO₃ C, 72.71; H, 5.82; N, 3.85; Found C, 72.83; H, 5.83, N, 3.86.

3 za.



2.5 h, yellow solid, yield 52%;

¹H NMR (400 MHz) (CDCl₃) δ 7.86 (d, *J* = 8.8 Hz, 2 H), 7.21-7.16 (m, 4 H), 6.93 (d, *J* = 8.8 Hz, 2 H), 4.99-4.91 (m, 1 H), 4.18 (q, *J* = 6.8 Hz, 2 H), 3.87 (s, 3 H), 3.17 (dd, *J*₁ = 14 Hz, *J*₂ = 8.8 Hz, 2 H), 2.96-2.86 (m, 2 H), 2.38 (s, 3 H), 1.26 (t, *J* = 6.8 Hz, 3 H);

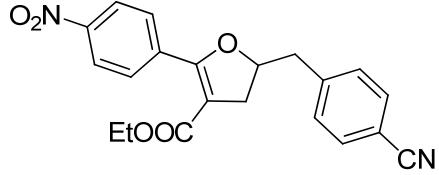
¹³C NMR (100.6 MHz) (CDCl₃) δ 165.7, 164.6, 161.2, 136.1, 134.0, 131.1, 129.3, 129.2, 122.5, 113.0, 100.7, 81.9, 59.6, 55.3, 41.5, 36.7, 21.1, 14.4;

IR (KBr, cm⁻¹) 2933, 2227, 1695, 1606, 1509, 1461, 1384, 1303, 1247, 1178, 1079, 1027, 873, 763;

MS m/z (relative intensity) 267 (10.1%), 218 (10.6%), 63 (11.4%), 191 (17.1%), 103 (19.6%), 253 (22.1%), 89 (22.3%), 147 (23.5%), 246 (24.4%), 363 (M⁺ 24.8%) 174 (25.4%), 116 (29.7%), 281 (38.2%), 73 (88.3%), 135 (88.3%), 207 (100%);

Anal. Calcd. For C₂₂H₂₄O₄ C, 74.98; H, 6.86; Found C, 75.06; H, 6.85.

3 zb.



24 h, yellow liquid, yield: 23%;

¹H NMR (400 MHz) (CDCl_3) δ 8.253 (d, $J = 9.2$ Hz, 2 H), 7.962 (d, $J = 8.8$ Hz, 2 H), 7.657 (d, $J = 8.4$ Hz, 2 H), 7.399 (d, $J = 8.4$ Hz, 2 H), 5.106-5.029 (m, 1 H), 4.198-4.135 (m, 2 H), 3.321-3.203 (m, 2 H), 3.099 (dd, $J_1 = 14.4$ Hz, $J_2 = 5.6$ Hz, 1 H), 2.922 (dd, $J_1 = 15.6$ Hz, $J_2 = 8$ Hz, 1 H), 1.246 (t, $J = 7.2$ Hz, 3 H);

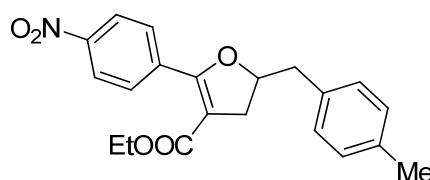
¹³C NMR (100.6 MHz) (CDCl_3) δ 164.5, 161.4, 148.6, 142.2, 135.8, 132.4, 130.3, 130.1, 123.6, 122.8, 111.0, 105.2, 81.6, 60.3, 41.9, 36.9, 14.2;

IR (Neat, cm^{-1}) 2925, 2227, 1695, 1592, 1519, 1454, 1384, 1346, 1245, 1085, 873, 754;

MS m/z (relative intensity) 63 (17.3%), 55 (33.0%), 378 (M^+ 41.5%), 155 (100%);

Anal. Calcd. For $\text{C}_{21}\text{H}_{18}\text{N}_2\text{O}_5\text{C}$, 66.66; H, 4.79; N, 7.40; Found C, 66.75; H, 4.80; N, 7.38.

3 zc.



24 h, yellow liquid, yield: 11%

¹H NMR (400 MHz) (CDCl_3) δ 8.24 (dd, $J_1 = 5.6$ Hz, $J_2 = 2$ Hz, 2 H), 8.00-7.98 (m, 2 H), 7.17 (m, 4H), 5.08-5.00 (m, 1 H), 4.16 (q, $J = 7.2$ Hz, 2 H), 3.24-3.13 (m, 2 H), 3.00-2.90 (m, 2 H), 2.37 (s, 3 H), 1.24 (t, $J = 7.2$ Hz, 3 H);

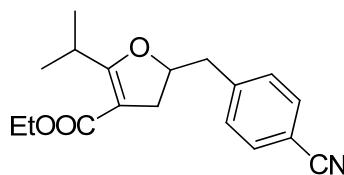
¹³C NMR (100.6 MHz) (CDCl_3) δ 164.2, 161.3, 142.9, 132.3, 131.1, 130.2, 122.0, 118.8, 113.0, 110.7, 100.7, 80.7, 59.7, 42.0, 36.9, 21.7, 14.4;

IR (Neat, cm^{-1}) 2925, 1695, 1592, 1519, 1461, 1374, 1301, 1247, 1079, 1085, 873, 763;

MS m/z (relative intensity), 55 (13.4%), 63 (15.4%) 105 (21.7%), 367 (M^+ 33.5%), 155 (100%);

Anal. Calcd. For $\text{C}_{21}\text{H}_{21}\text{NO}_5\text{C}$, 68.65; H, 5.76 N, 3.81; Found C, 68.70; H, 5.75 N, 3.83

3 zd.



0.75 h, yellow liquid, yield: 90%;

¹H NMR (400 MHz) (CDCl_3) δ 7.60 (d, $J = 8.0$ Hz, 2 H), 7.34 (d, $J = 8.4$ Hz, 2 H), 4.85-4.77 (m, 1 H), 4.15 (q, $J = 6.8$ Hz, 2 H), 3.60 (ept, $J = 6.8$ Hz, 1 H), 3.06-2.89 (m, 3 H), 2.58 (dd, $J_1 = 14.4$ Hz, $J_2 = 7.2$ Hz, 1 H), 1.26 (t, $J = 7.2$ Hz, 3 H), 1.10 (d, $J = 6.8$ Hz, 3H), 1.08 (d, $J = 6.8$ Hz, 3H);

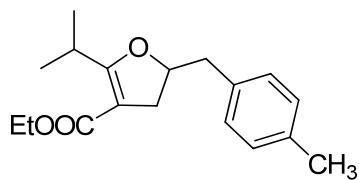
¹³C NMR (100.6 MHz) (CDCl_3) 174.9, 165.8, 142.8, 132.2, 130.3, 118.8, 110.6, 99.4, 81.3, 59.4, 42.0, 34.9, 26.7, 19.6, 19.5, 14.4;

IR (neat, cm^{-1}) 2973, 2873, 2229, 1695, 1633, 1469, 1371, 1342, 1236, 1116, 1045, 815, 765, 557;

MS m/z (relative intensity) 141 (23.2%), 254 (24.9%), 111 (25.2%), 69 (28.3%), 55 (37.0%), 299 (M^+ 44.2%), 137 (44.9%), 155 (65.9%), 116 (77.3%), 183 (100%);

Anal. Calcd. For $\text{C}_{18}\text{H}_{21}\text{NO}_3\text{C}$, 72.22; H, 7.07; N, 4.68; Found C, 72.35; H, 7.05, N, 4.65.

3 ze.



2.5 h, brown liquid, yield 83%;

¹H NMR (400 MHz) (CDCl_3) δ 7.14 (s, 4 H), 4.85-4.78 (m, 1 H), 4.21-4.15 (m, 2 H), 3.65 (ept, J = 6.9 Hz, 1 H), 3.09 (dd, J_1 = 14 Hz, J_2 = 6.4 Hz, 1 H), 2.92 (dd, J_1 = 14.4 Hz, J_2 = 10 Hz, 1 H), 2.81 (dd, J_1 = 14 Hz, J_2 = 6.4 Hz, 1 H), 2.63 (dd, J_1 = 14.8 Hz, J_2 = 7.2 Hz, 1 H), 2.36 (s, 3 H), 1.29 (t, J = 7.2 Hz, 4 H), 1.17 (d, J = 6.9 Hz, 3H), 1.14 (d, J = 6.9 Hz, 3H);

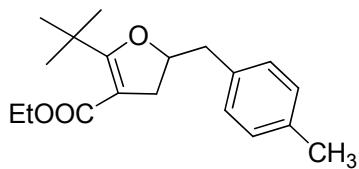
¹³C NMR (100.6 MHz) (CDCl_3) δ 175.4, 166.2, 136.1, 134.0, 129.4, 129.1, 99.2, 82.5, 59.3, 41.5, 34.7, 26.8, 21.0, 19.7, 19.6, 14.4;

IR (neat, cm^{-1}) 2923, 1959, 1633, 1450, 1384, 1047, 873, 713, 406;

MS m/z (relative intensity) 243 (10.5%), 141 (12.4%), 69 (17.6%), 55 (18.6%), 155 (20.0%), 113 (21.7%), 79 (30.6%), 288 (M^+ 30.6%), 153 (30.7%), 91 (35.3%), 105 (69.7%), 106 (100%);

Anal. Calcd. For $\text{C}_{18}\text{H}_{24}\text{O}_3$ C, 74.97; H, 8.39; Found C, 74.86; H, 8.37.

3 zf.



22 h, yellow liquid, yield 10%;

¹H NMR (400 MHz) (CDCl_3) δ 7.16 (s, 4 H), 4.77-4.69 (m, 1 H), 4.18-4.12 (m, 2 H), 3.03-2.95 (m, 2 H), 2.79 (dd, J_1 = 14 Hz, J_2 = 6.4 Hz, 1 H), 2.69 (dd, J_1 = 14.4 Hz, J_2 = 7.2 Hz, 1 H), 2.35 (s, 3 H), 1.31 (m, 12 H);

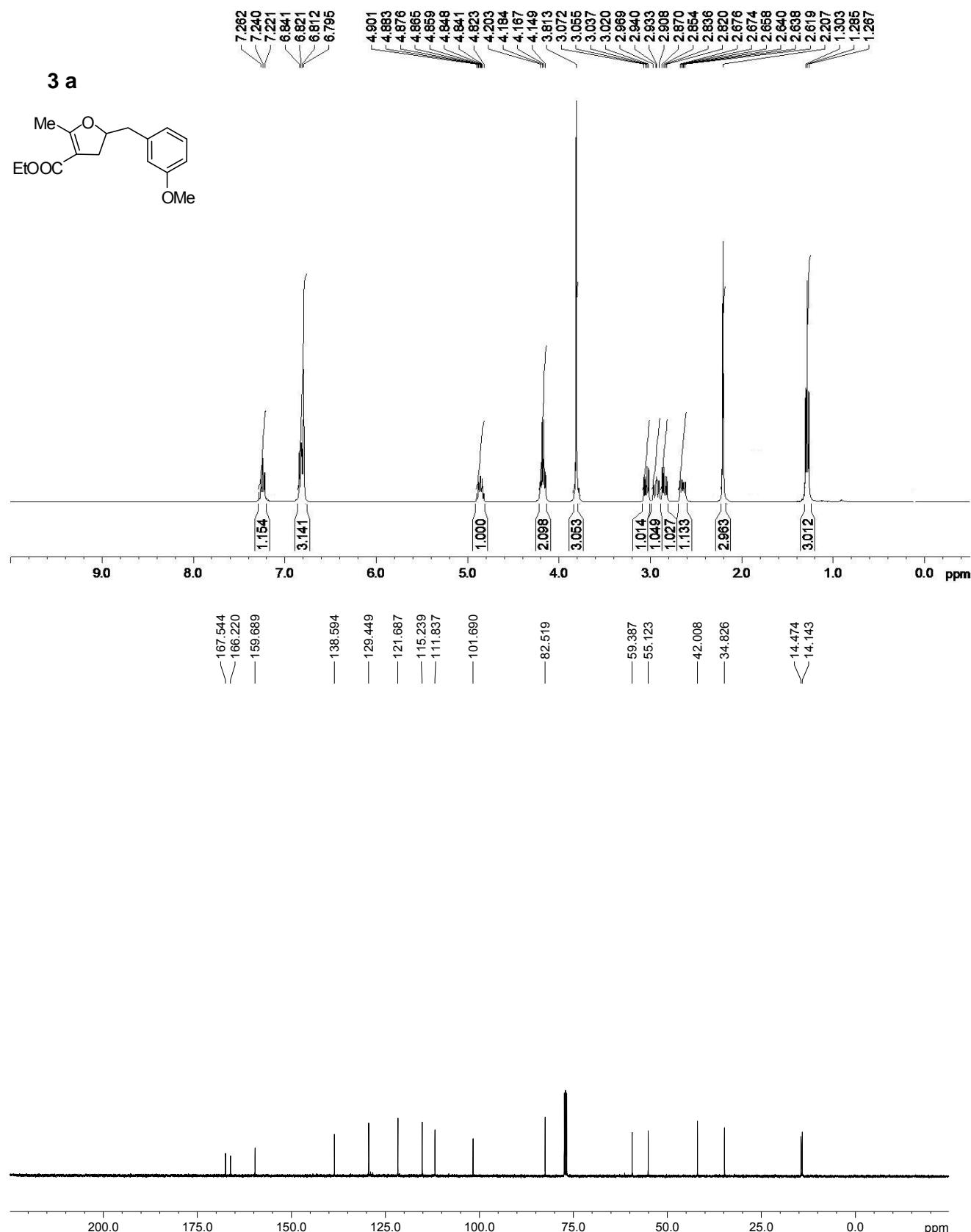
¹³C NMR (100.6 MHz) (CDCl_3) δ 167.6, 162.8, 136.2, 133.2, 129.2, 129.1, 101.6, 82.8, 59.4, 41.6, 34.7, 32.8, 21.0, 14.2;

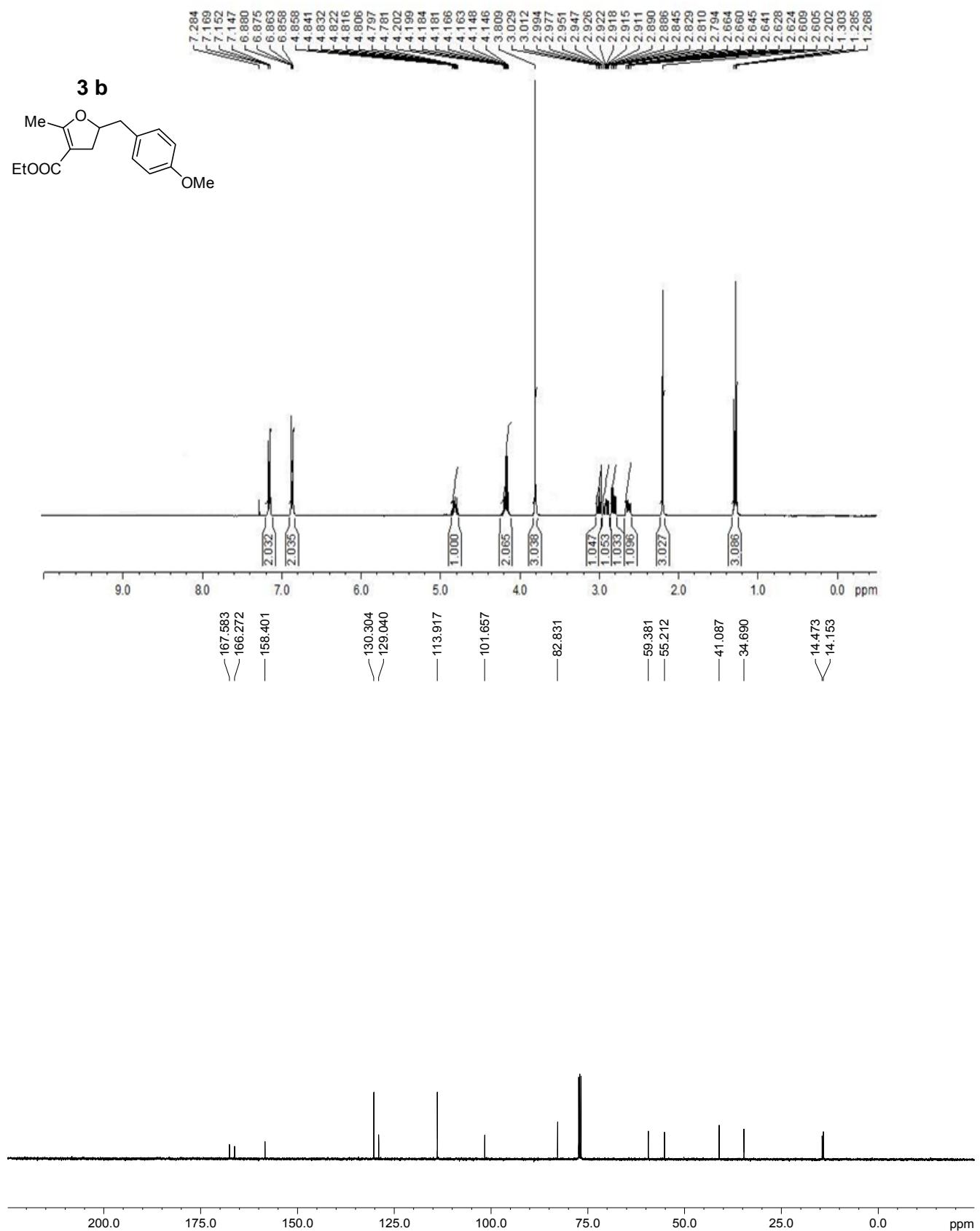
IR (neat, cm^{-1}) 2923, 1959, 1633, 1450, 1397, 1370, 1047, 873, 713, 406

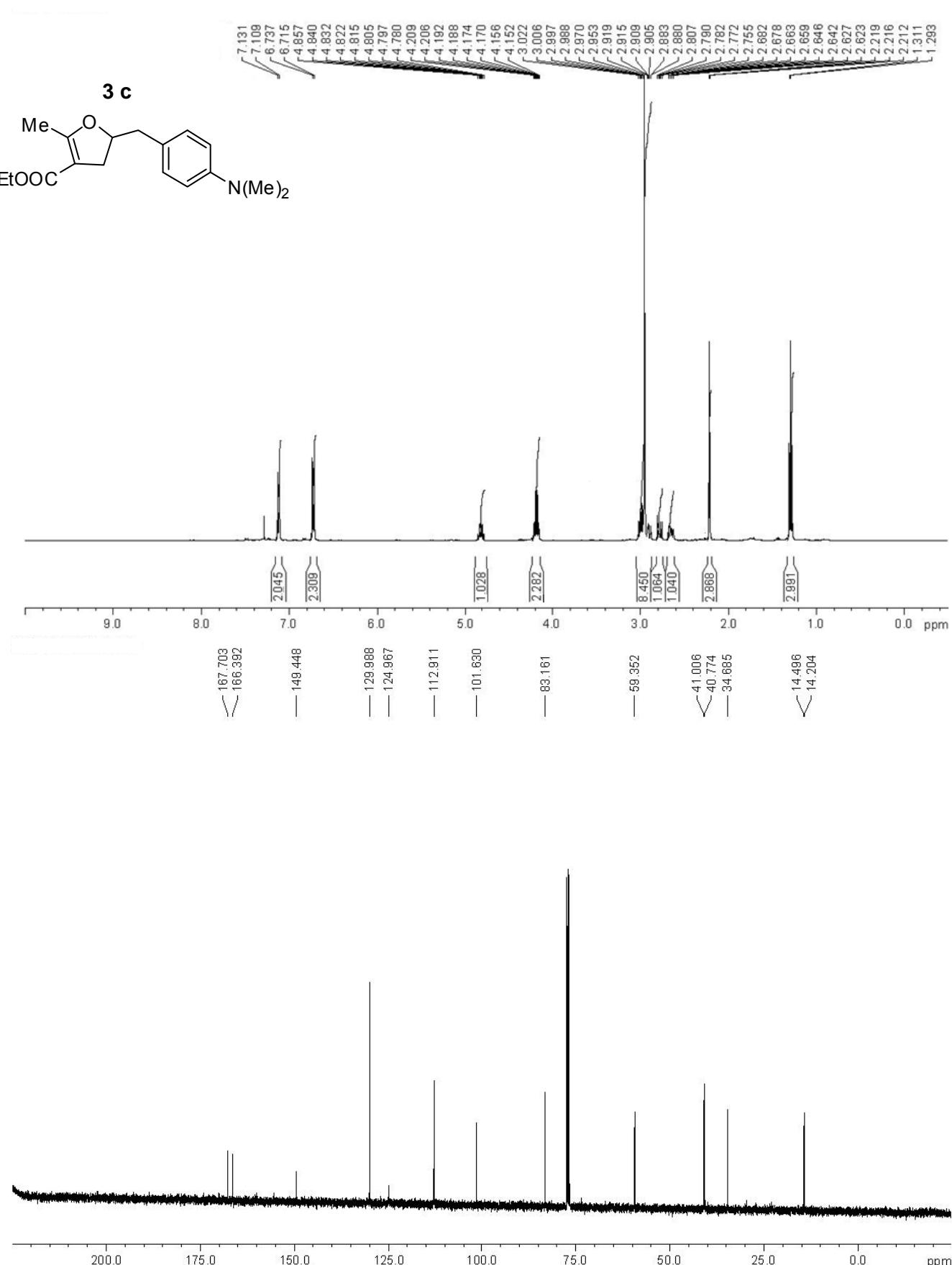
MS m/z (relative intensity) 105 (11.3%), 302 (M^+ 27.3%), 63 (55.1%), 155 (100%);

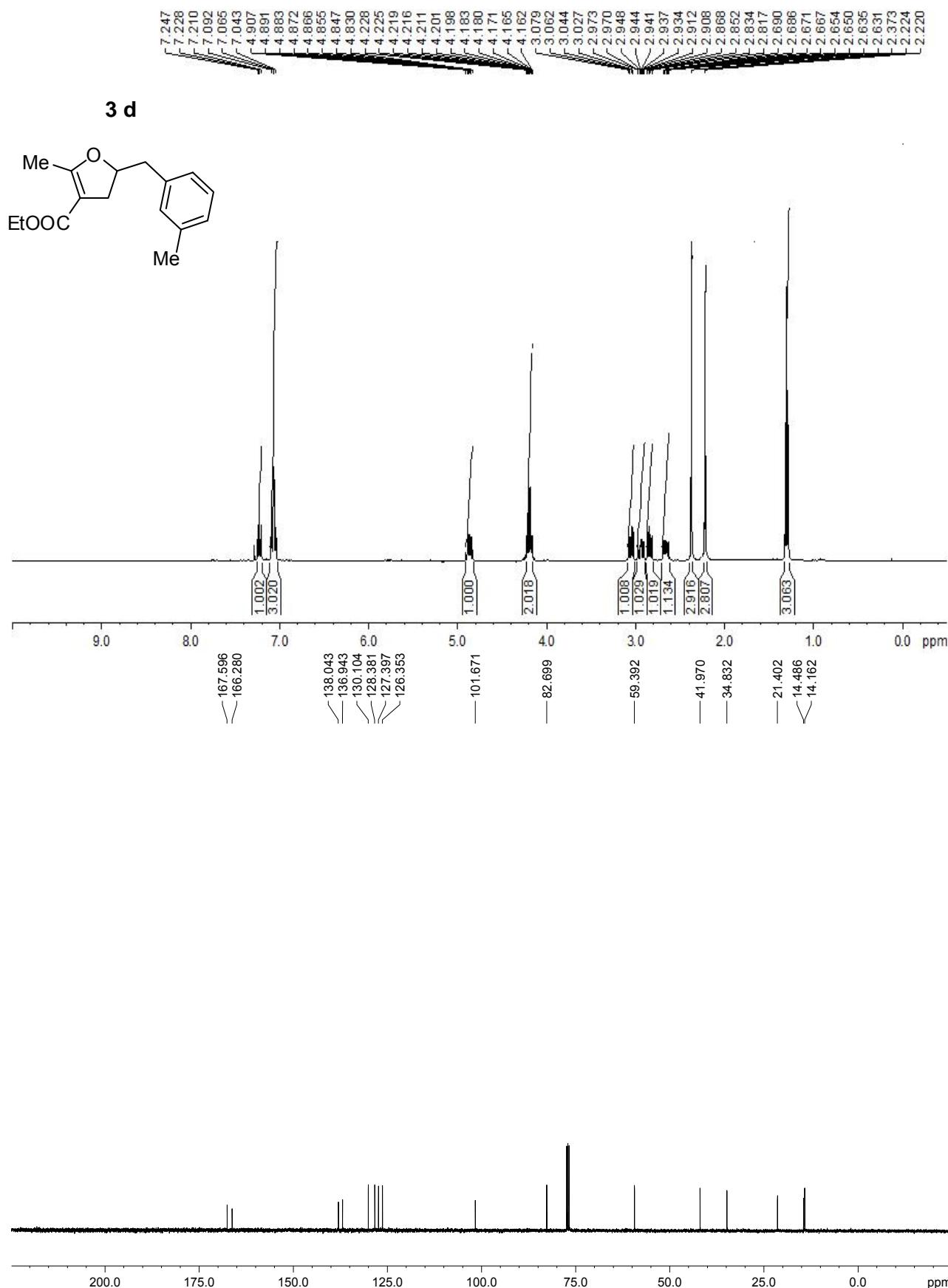
Anal. Calcd. For $\text{C}_{19}\text{H}_{26}\text{O}_3$ C, 75.46; H, 8.67; Found C, 75.66; H, 8.65

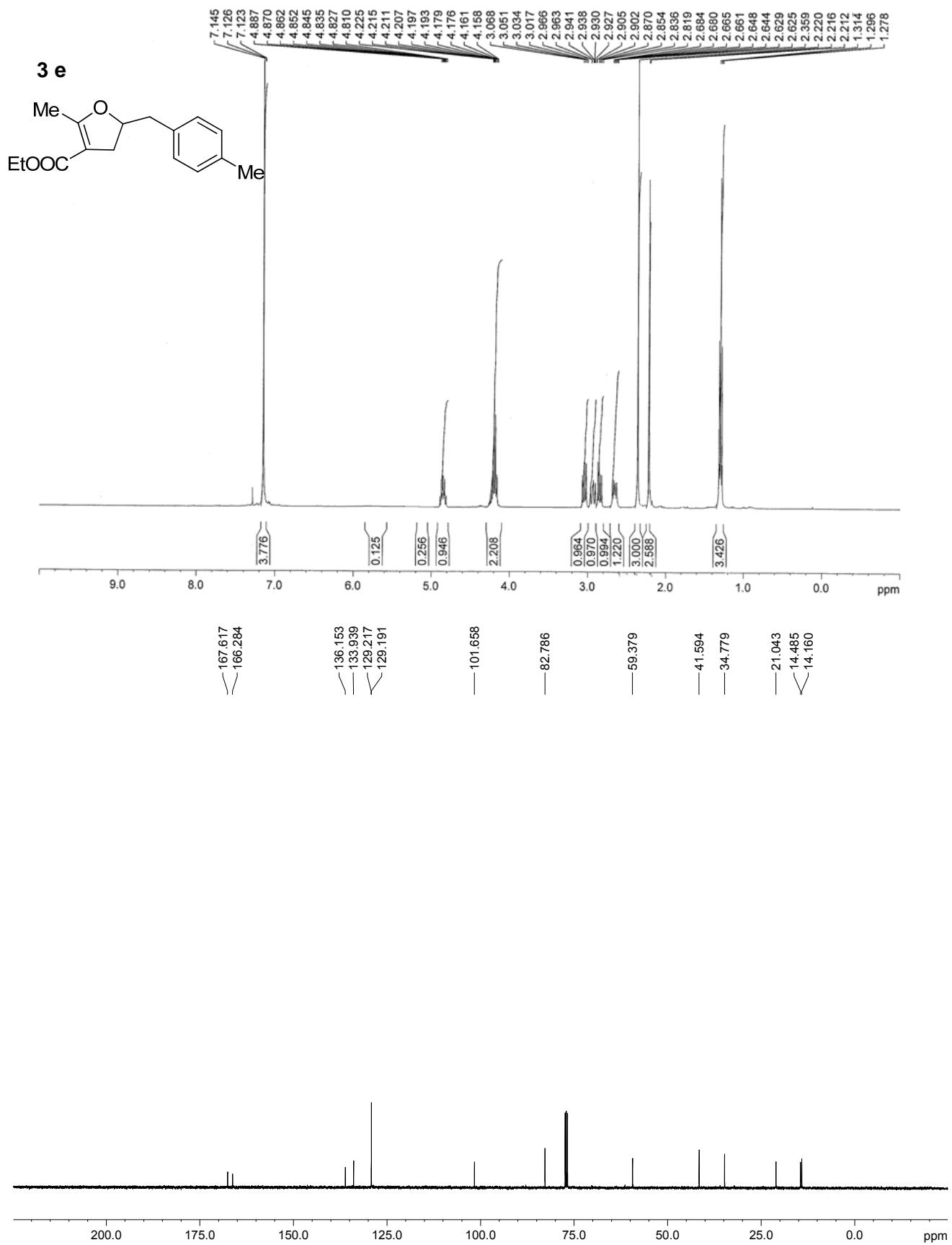
9. Copy of ^1H and ^{13}C spectra.

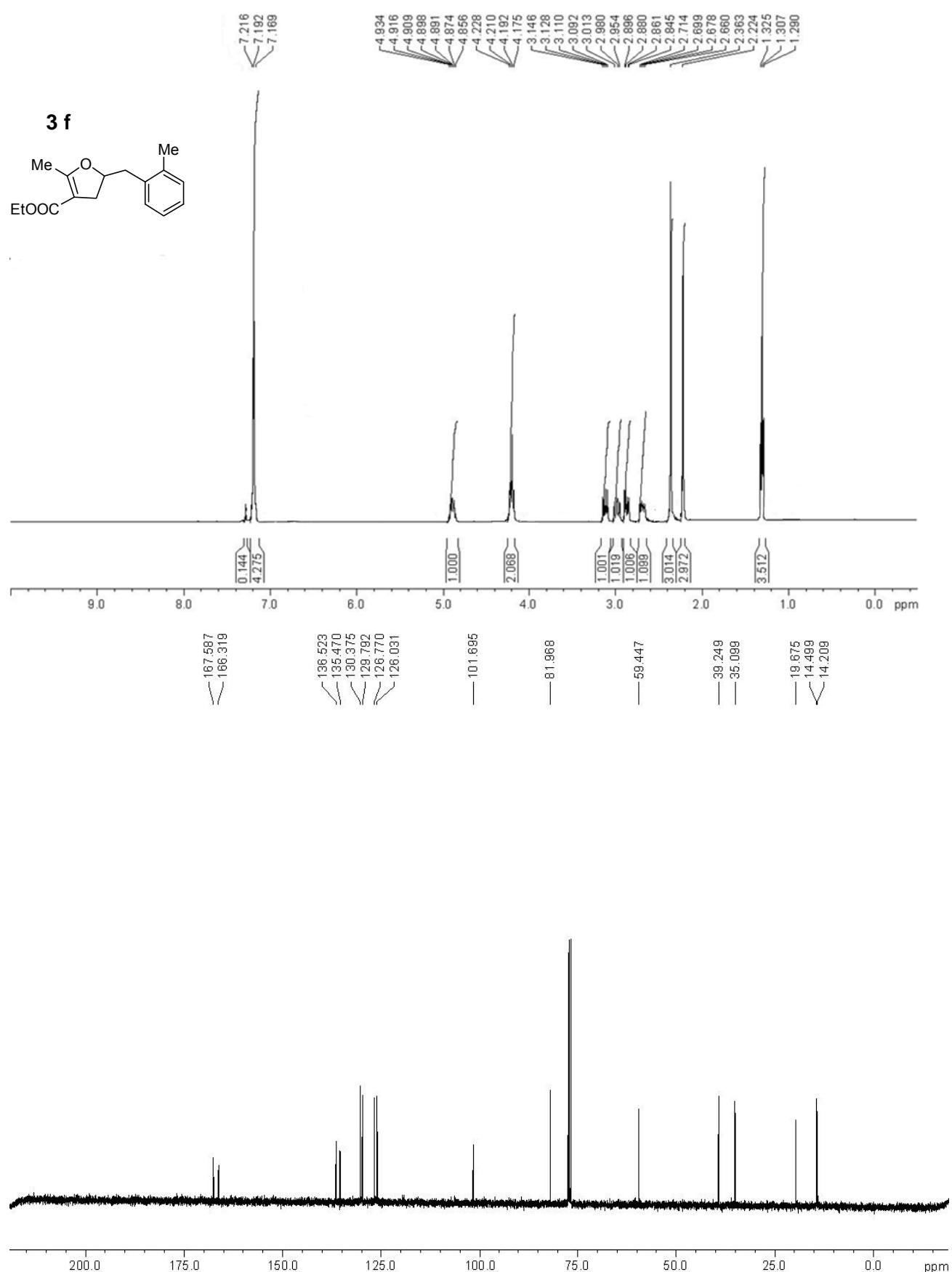


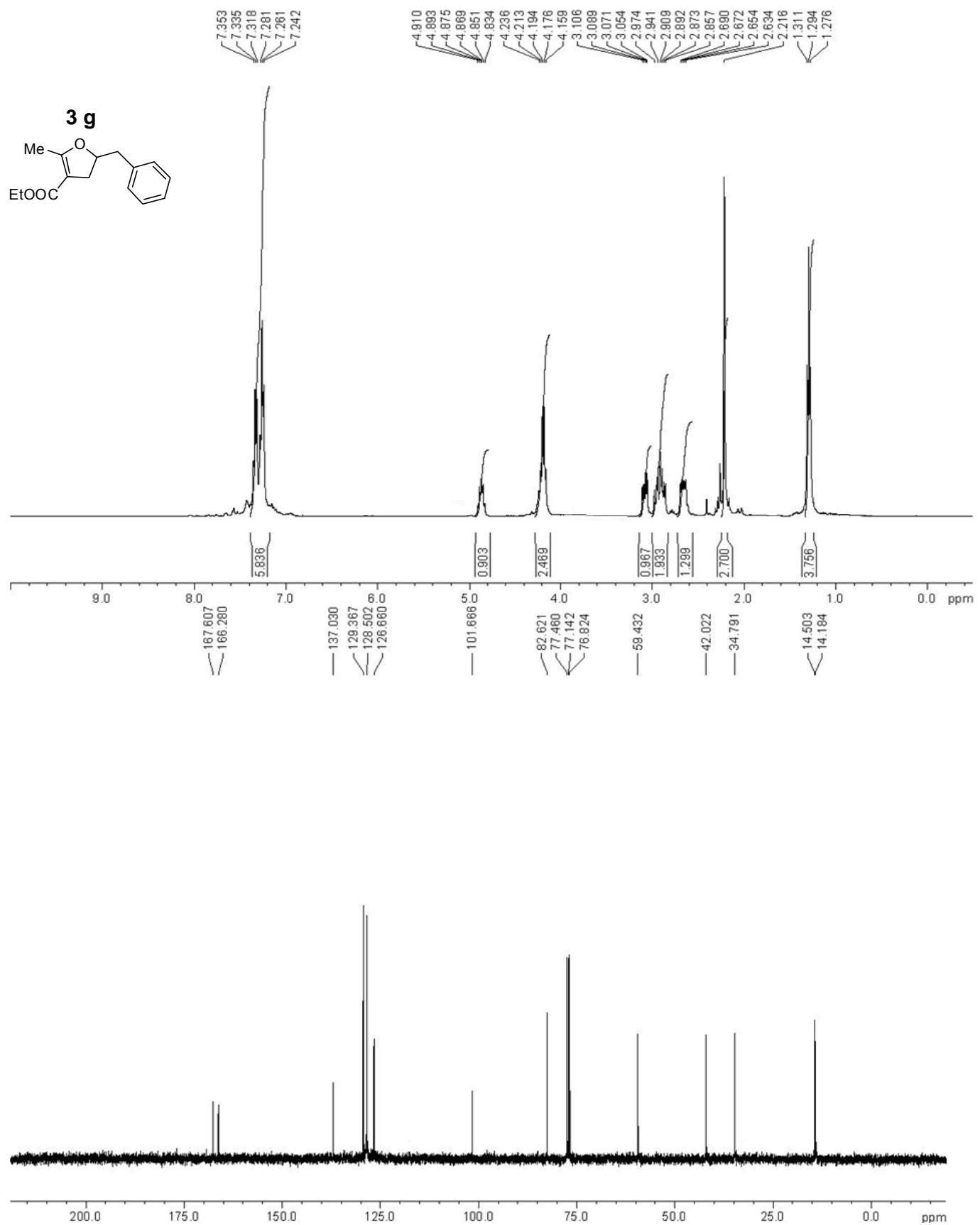


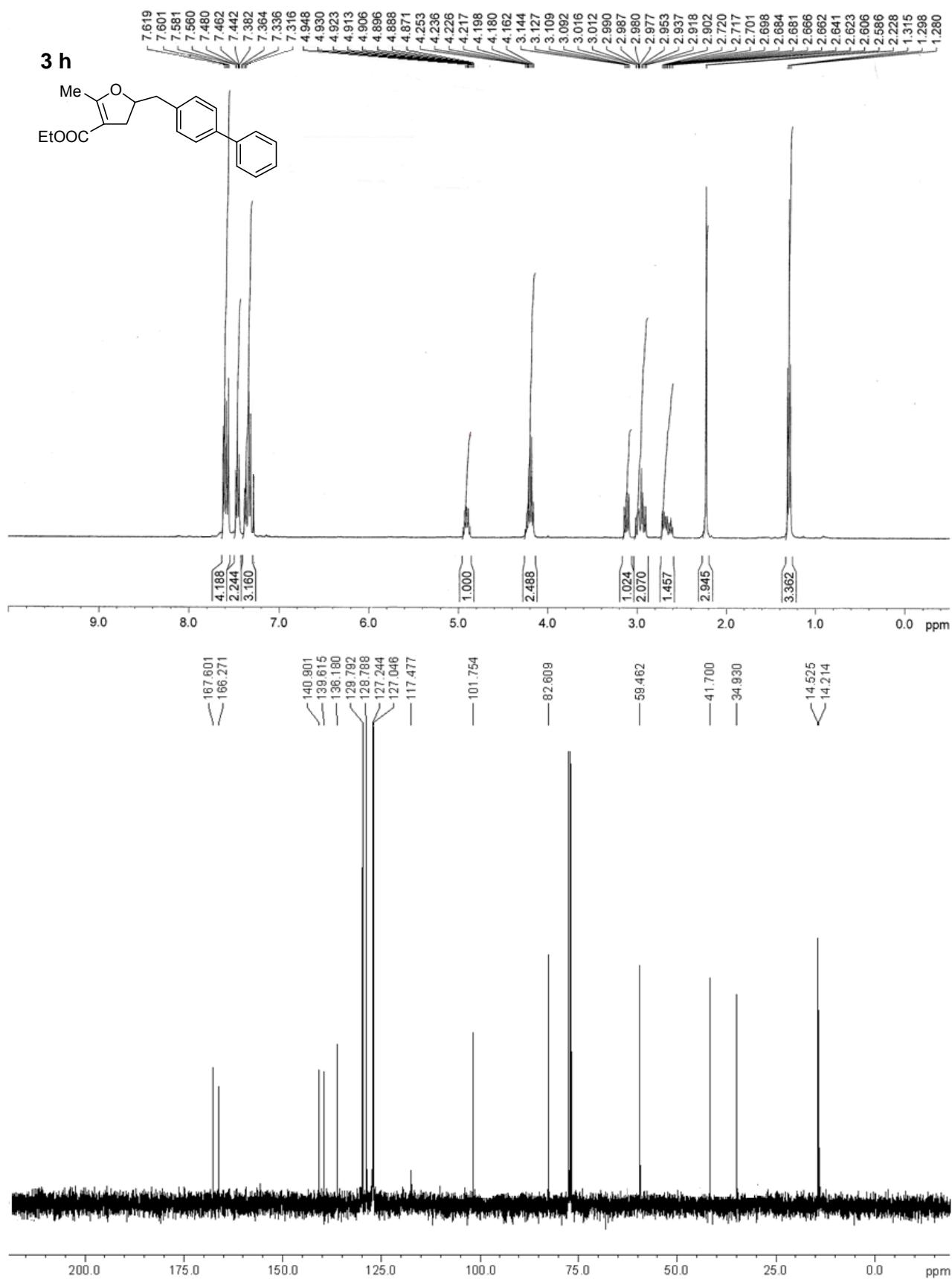


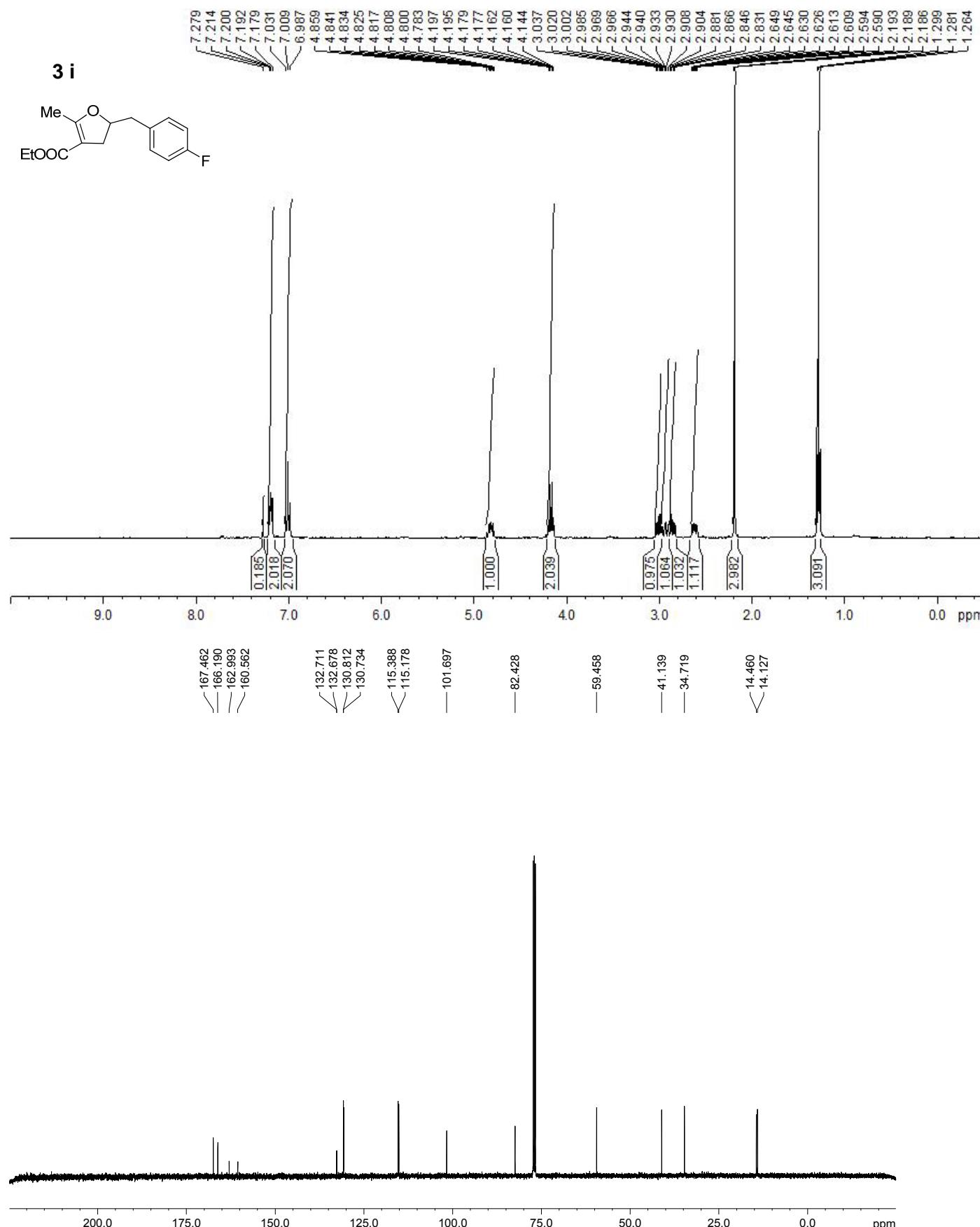


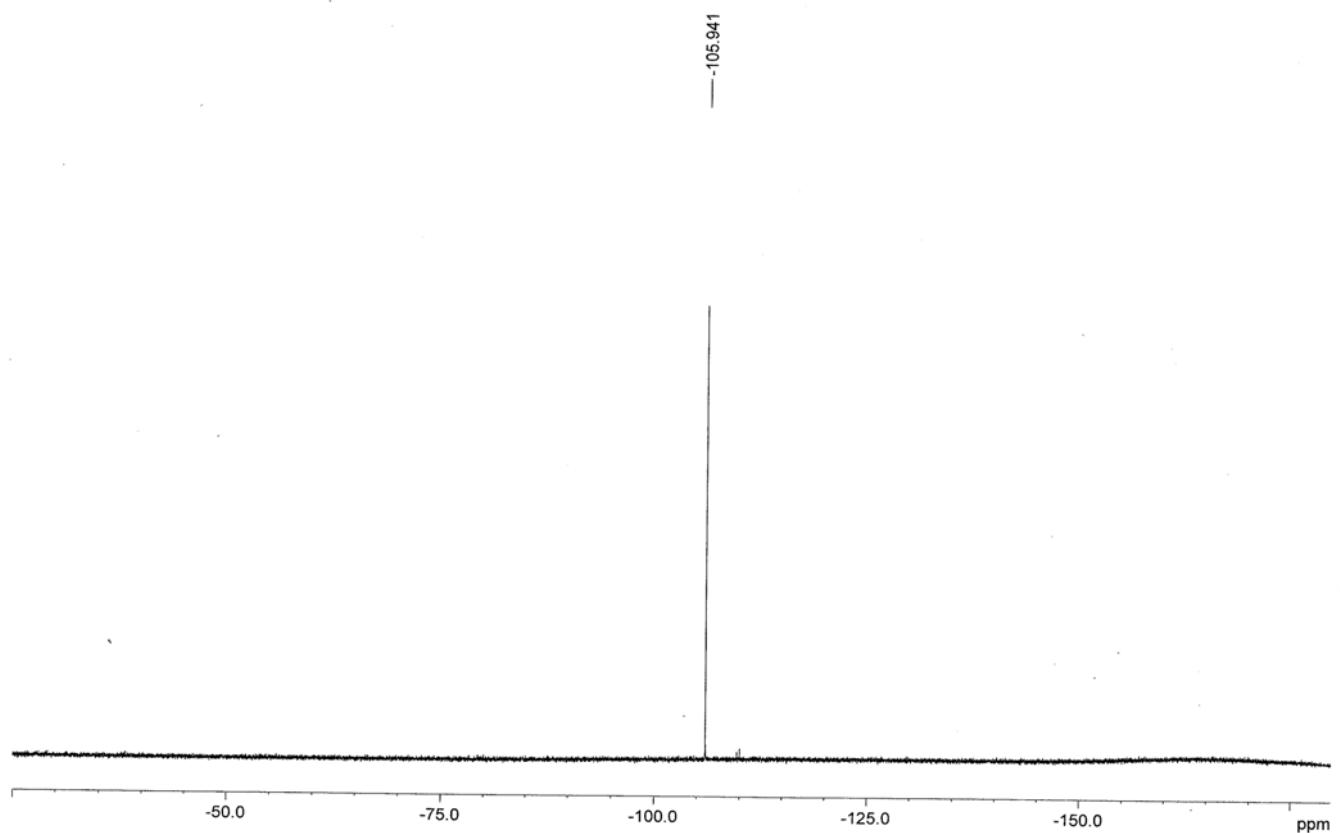


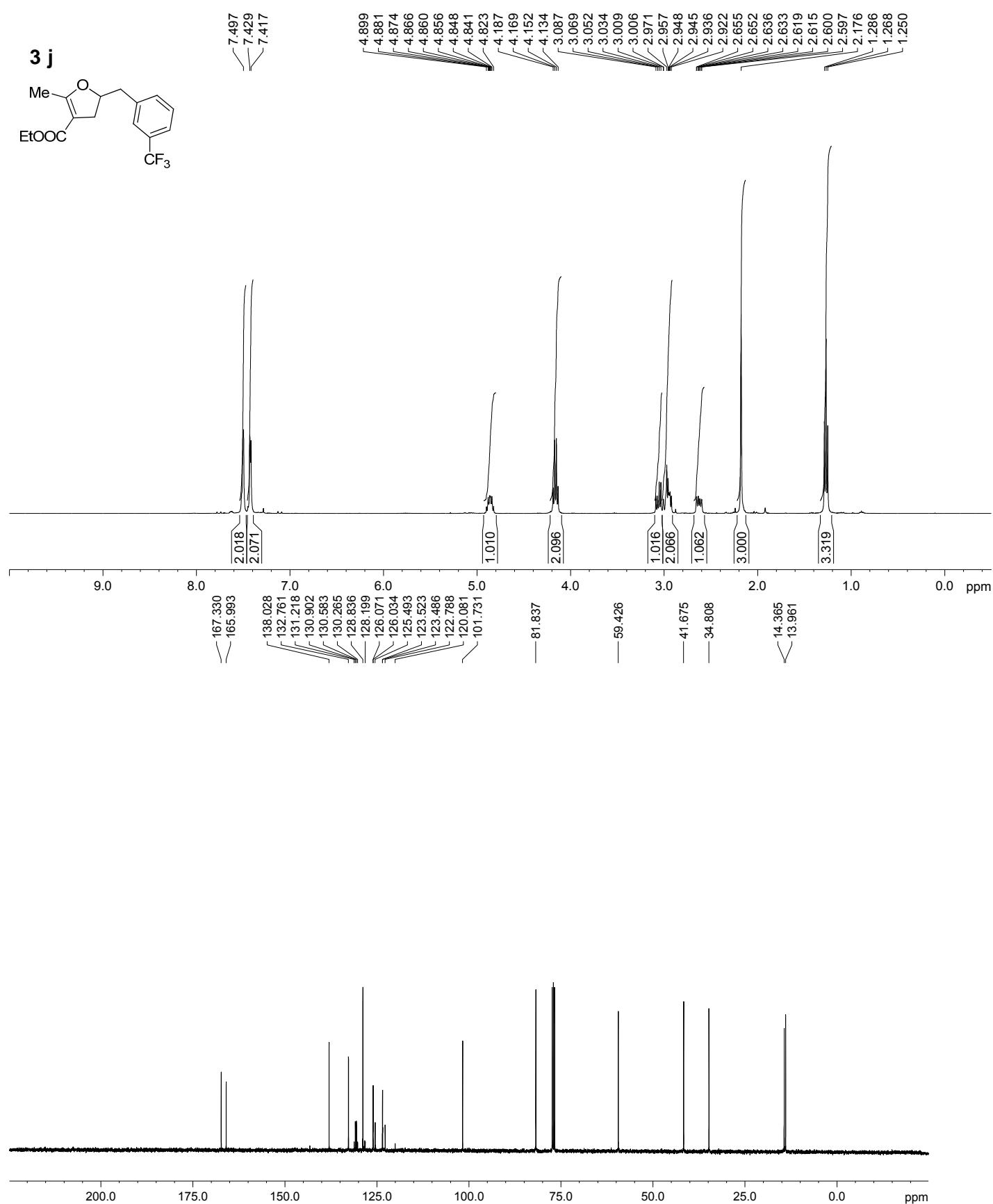


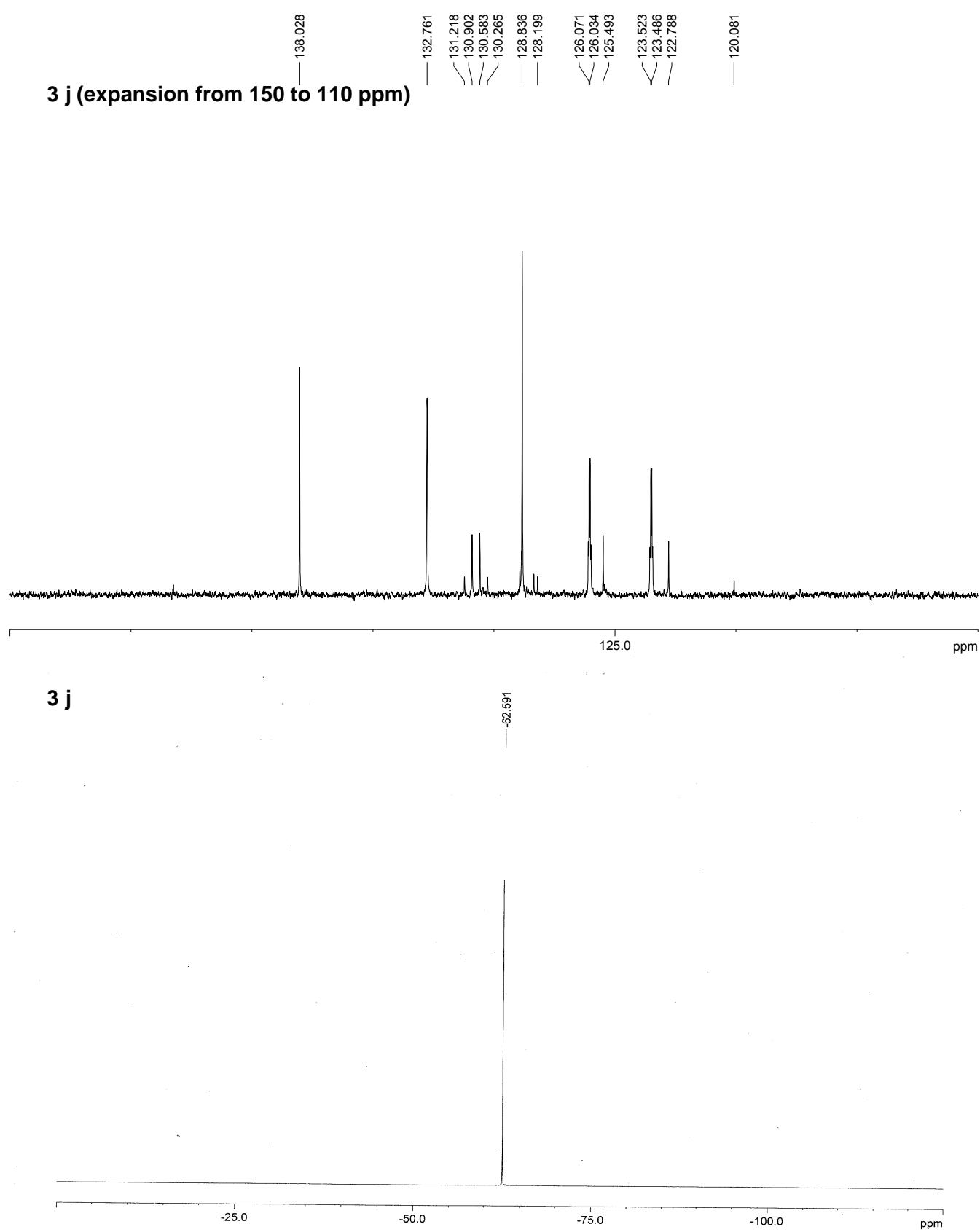


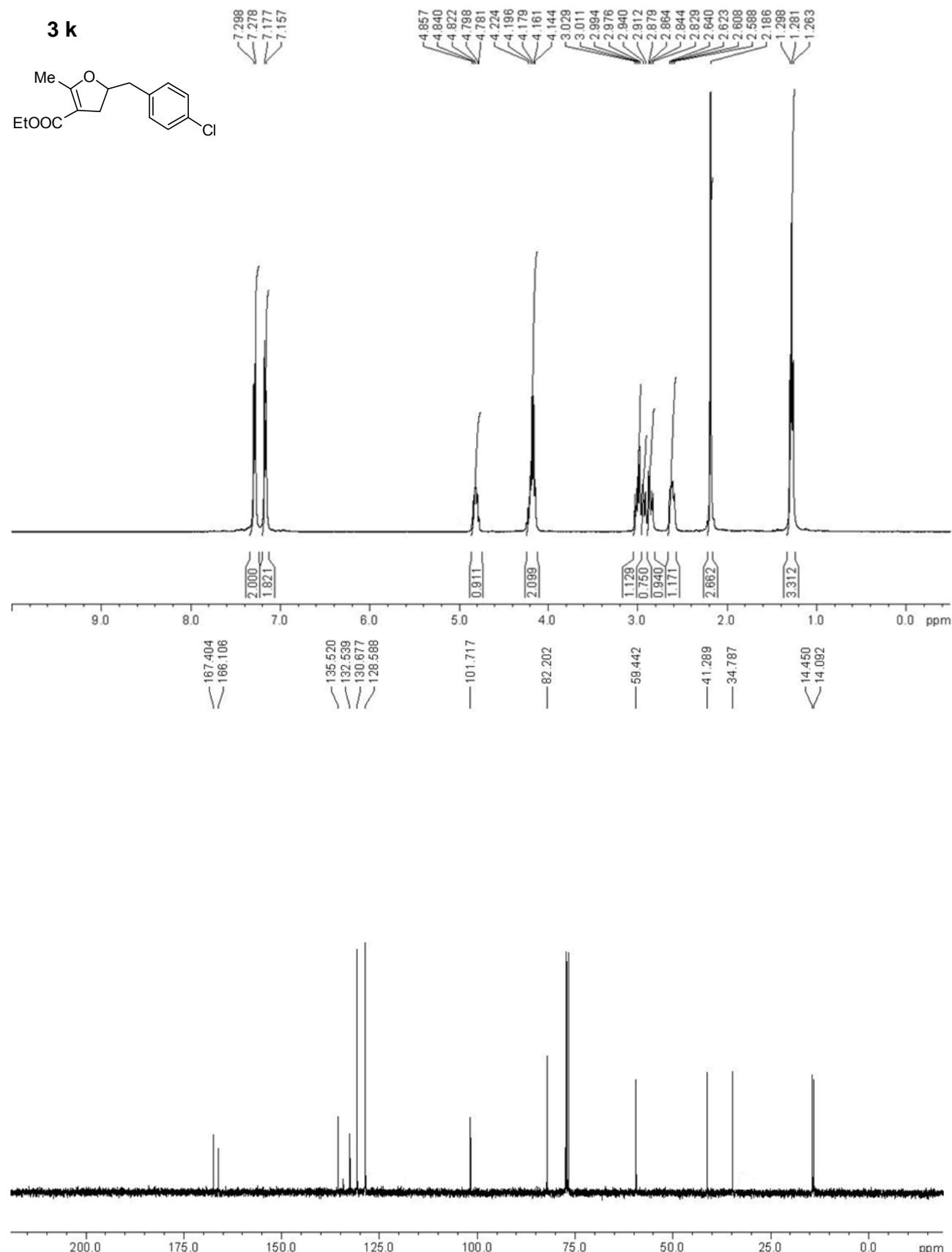


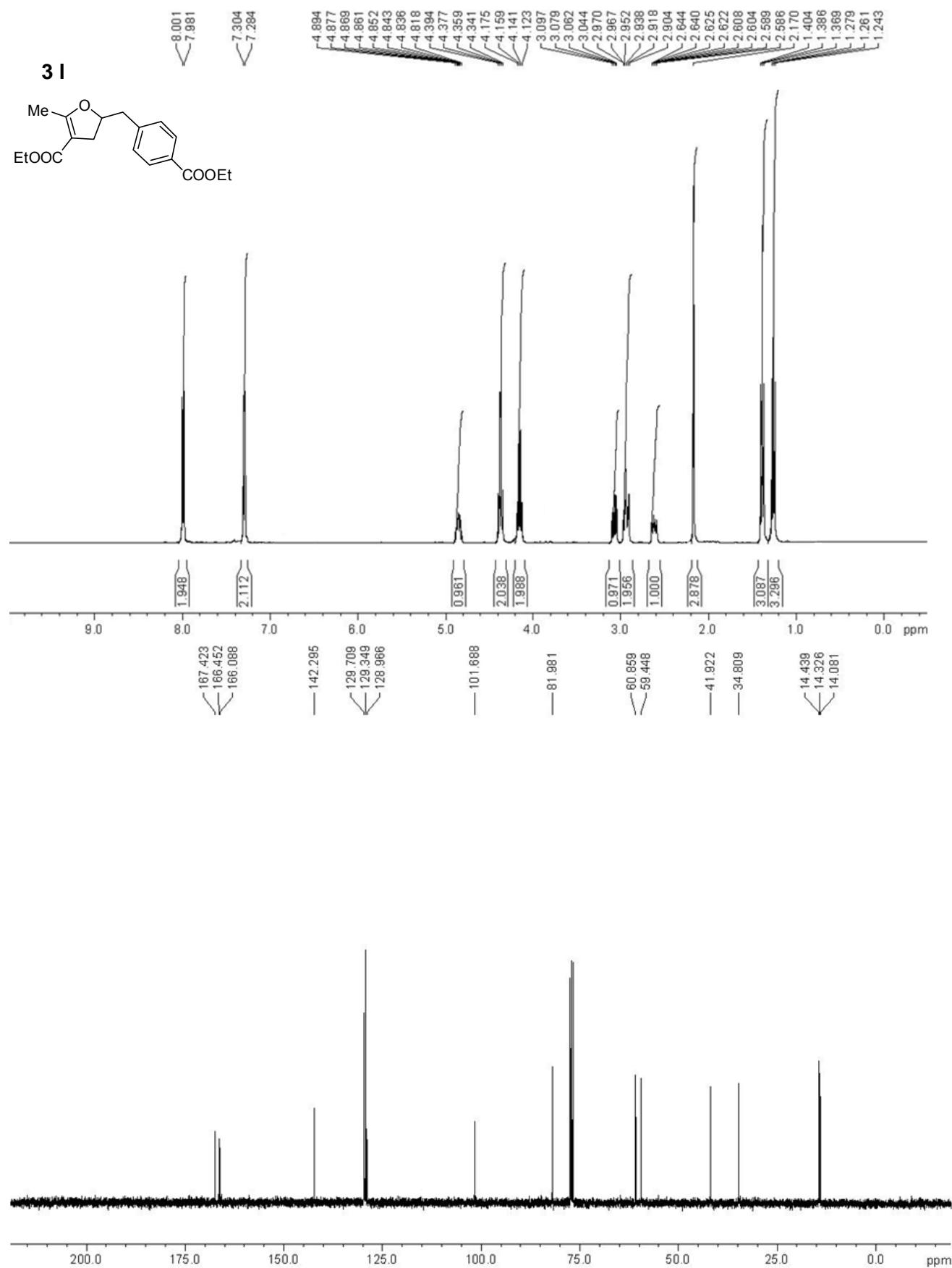


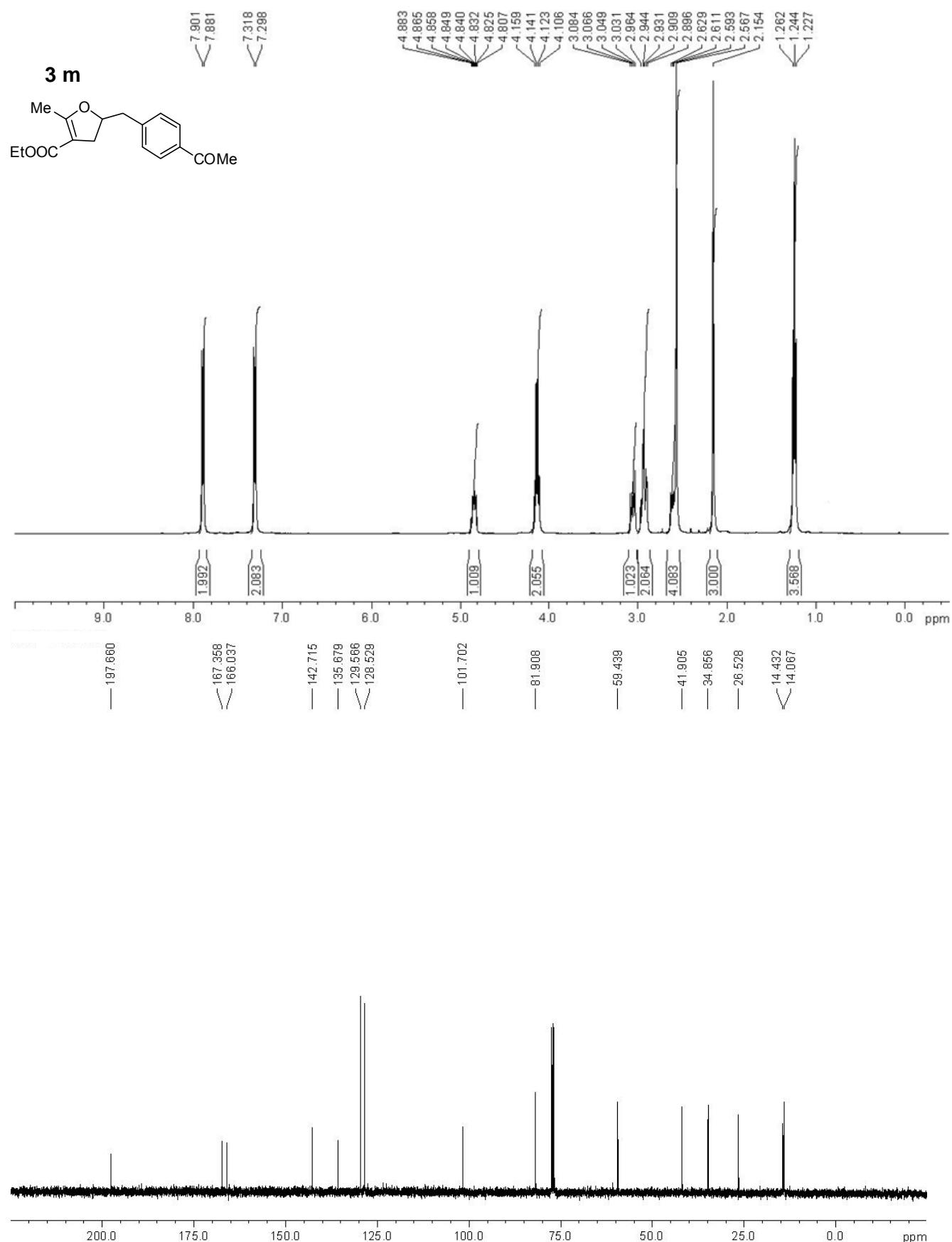


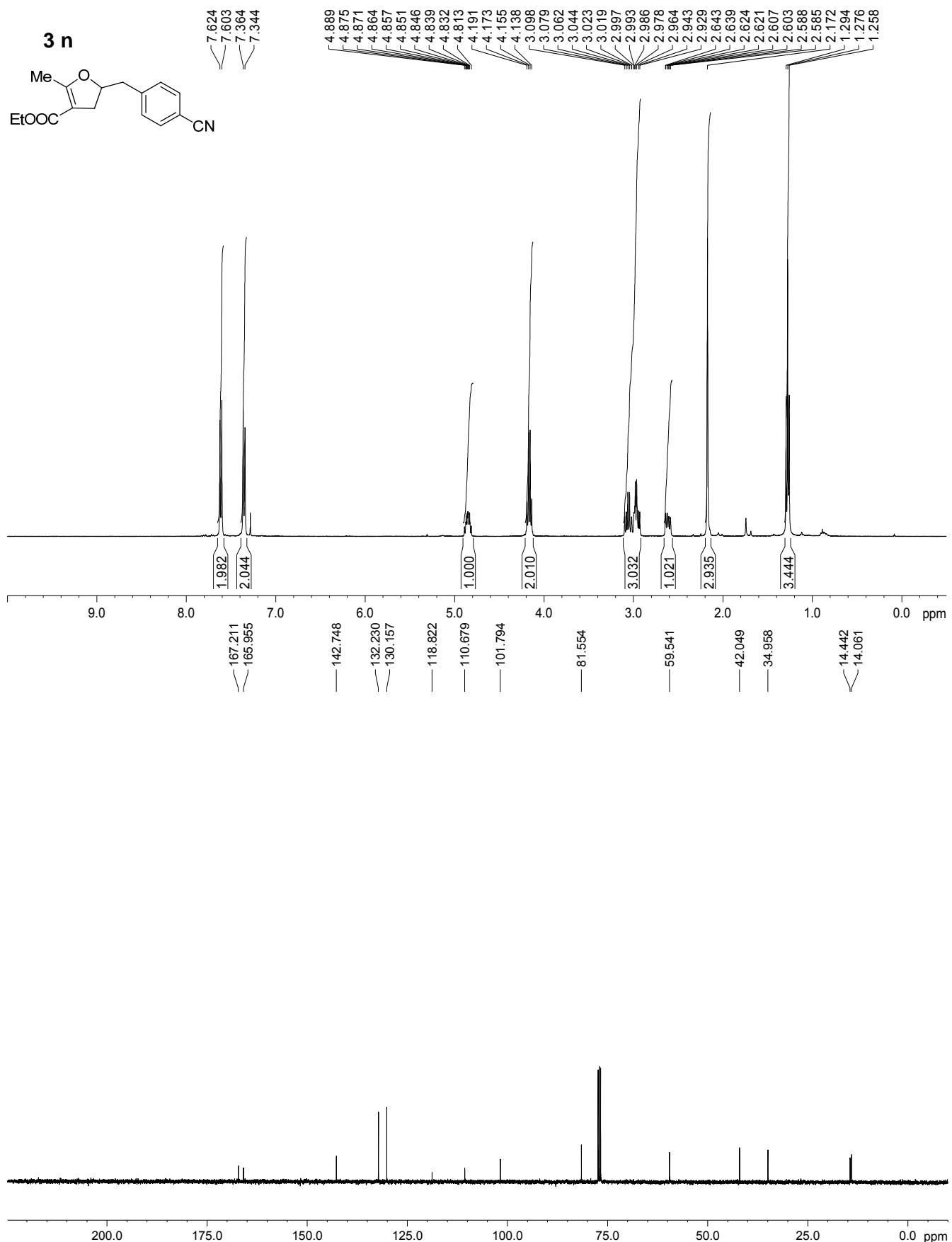


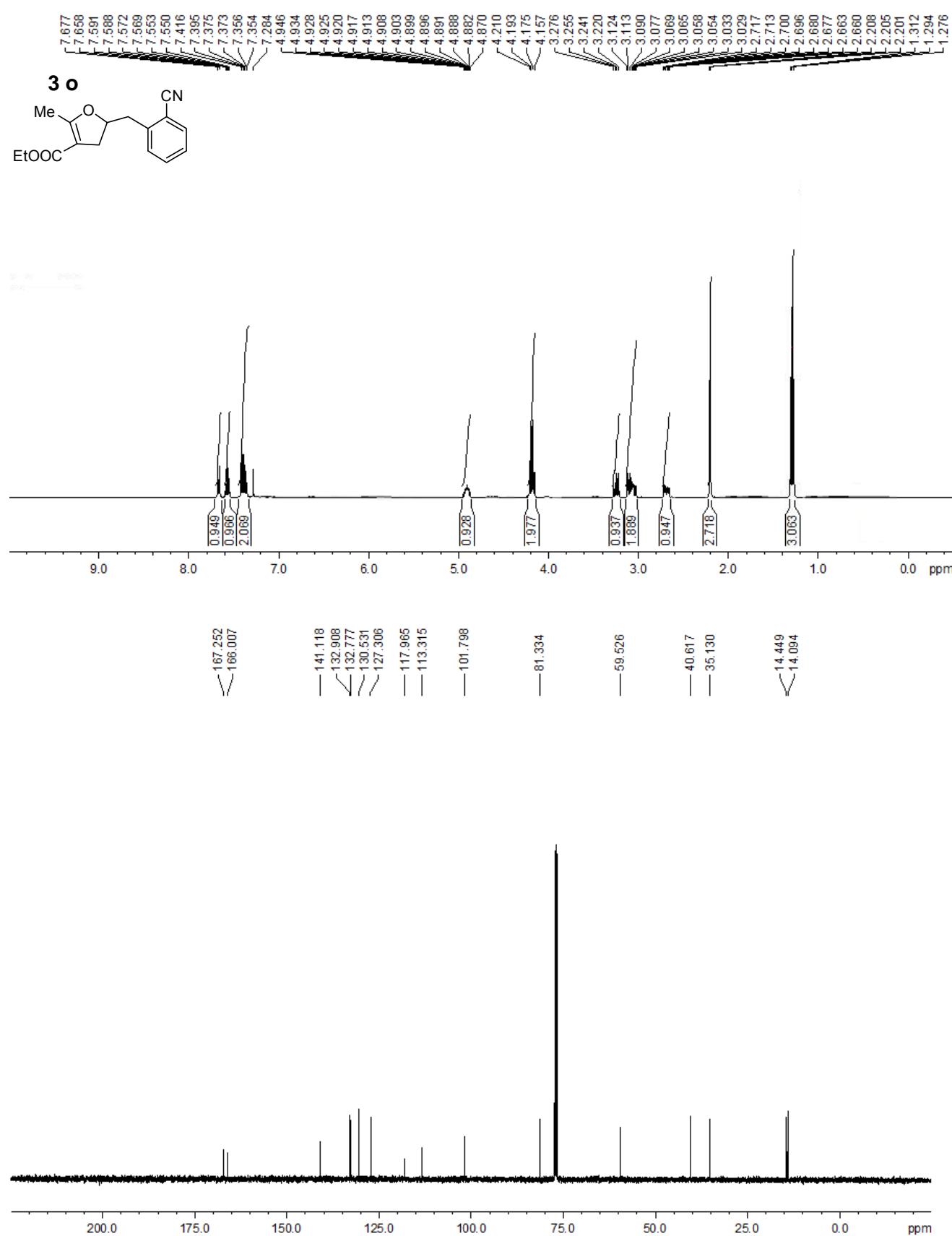


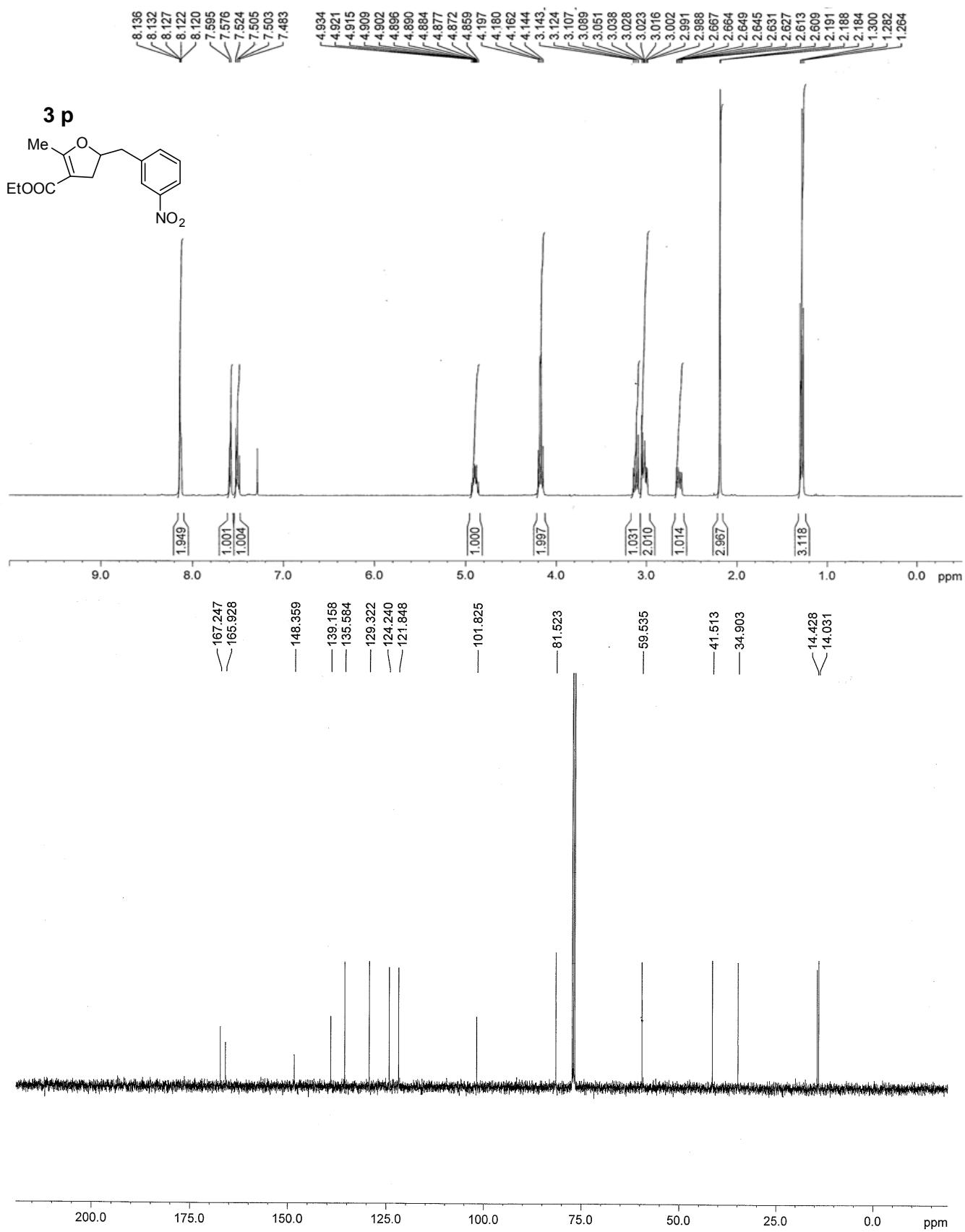


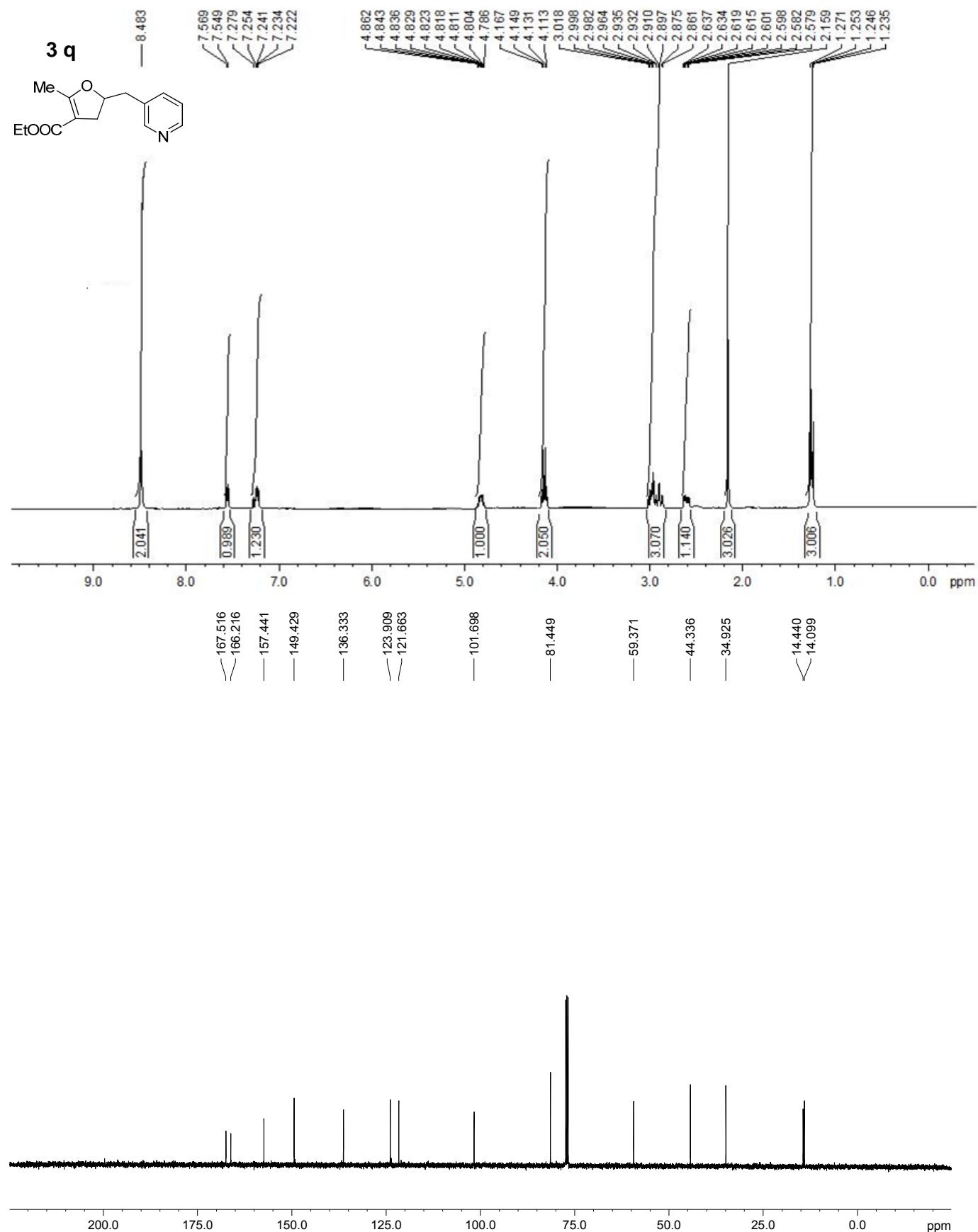


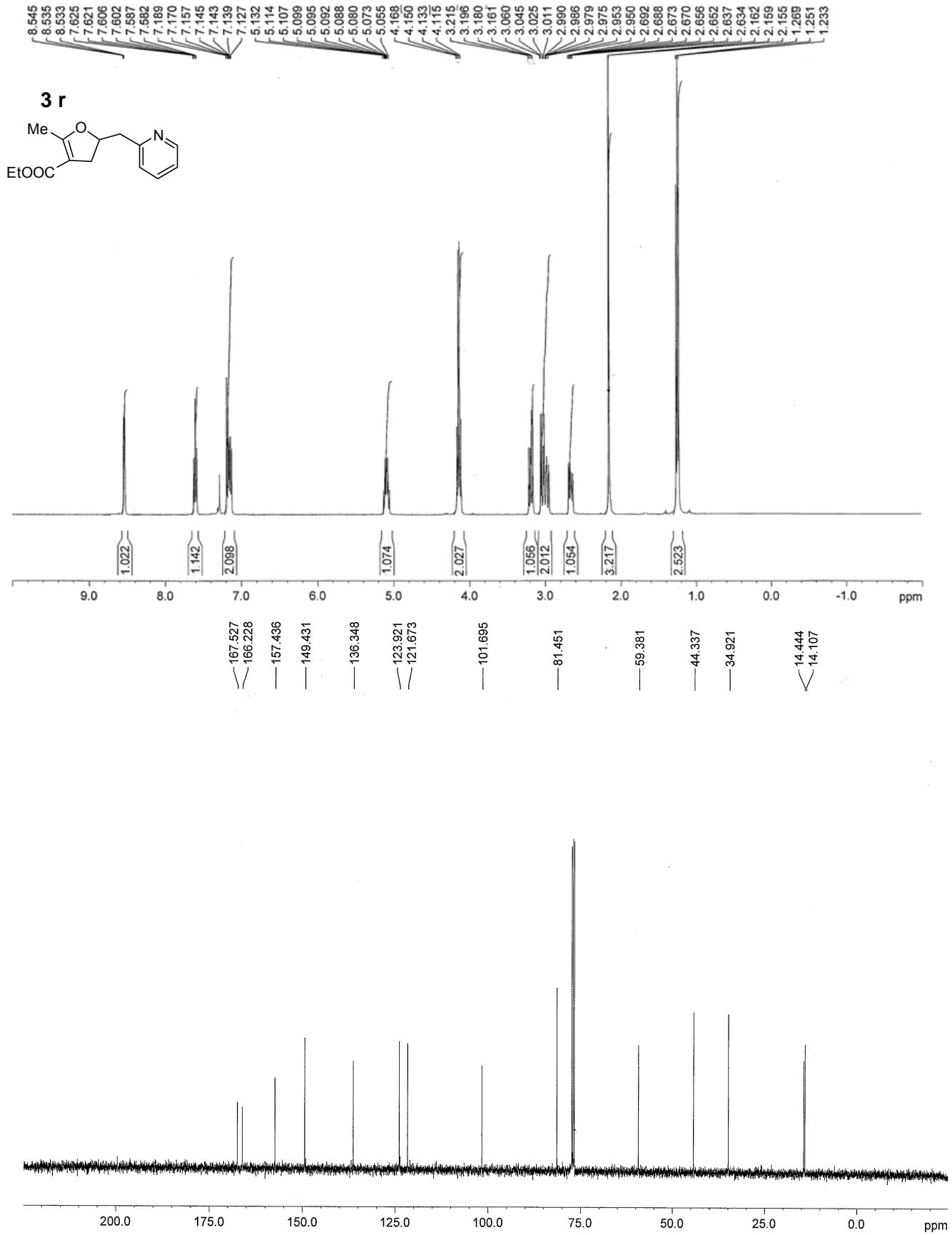


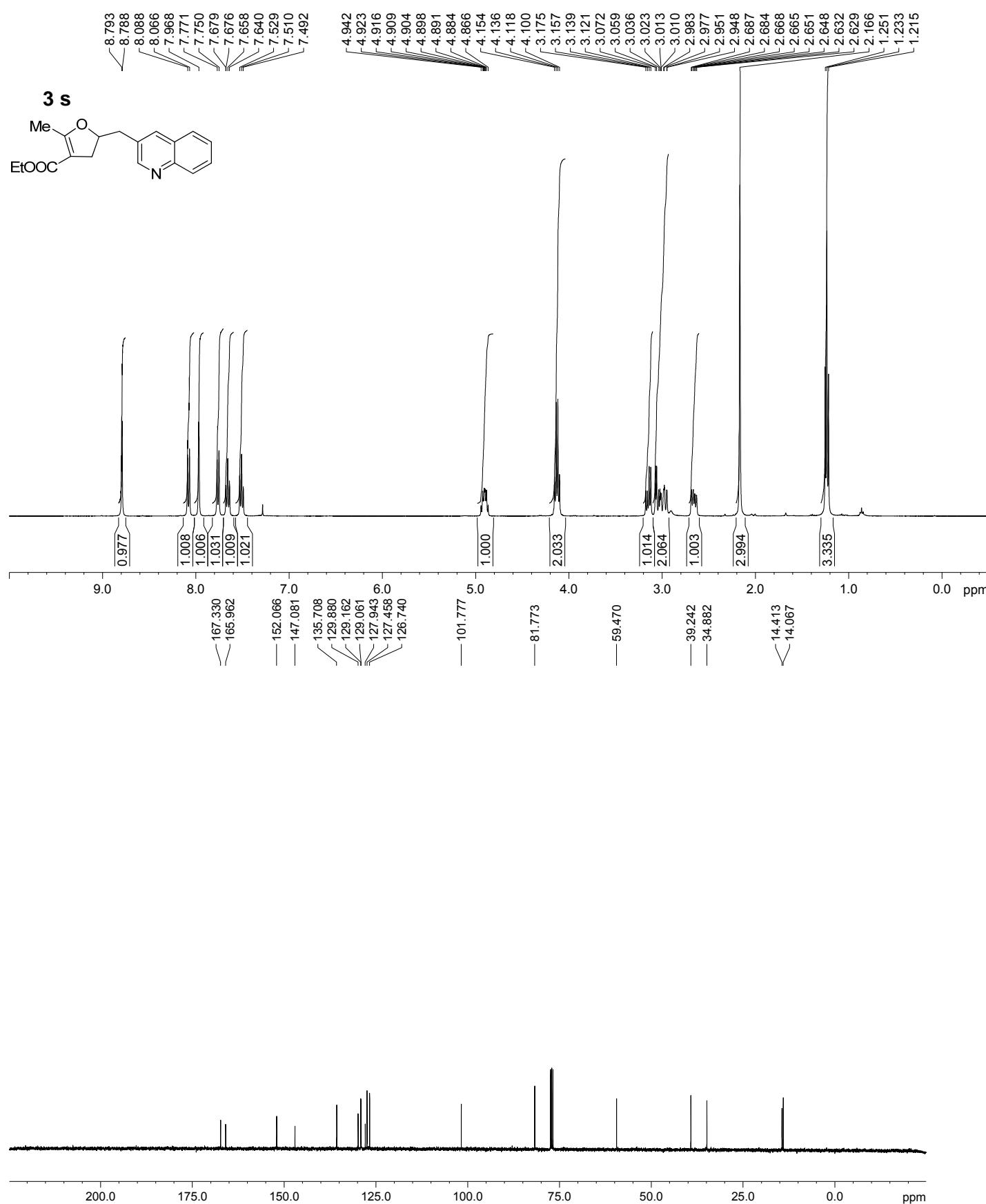


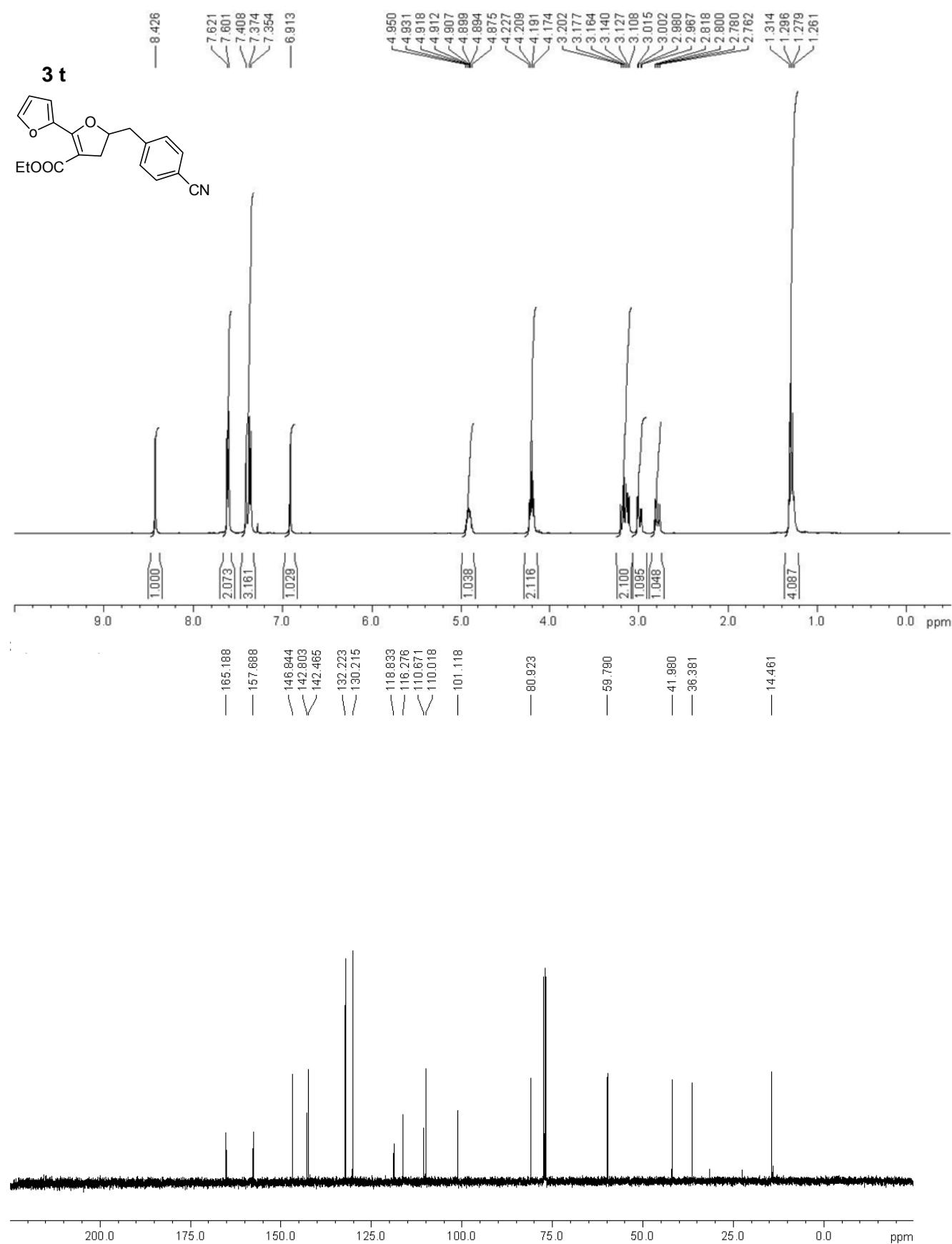


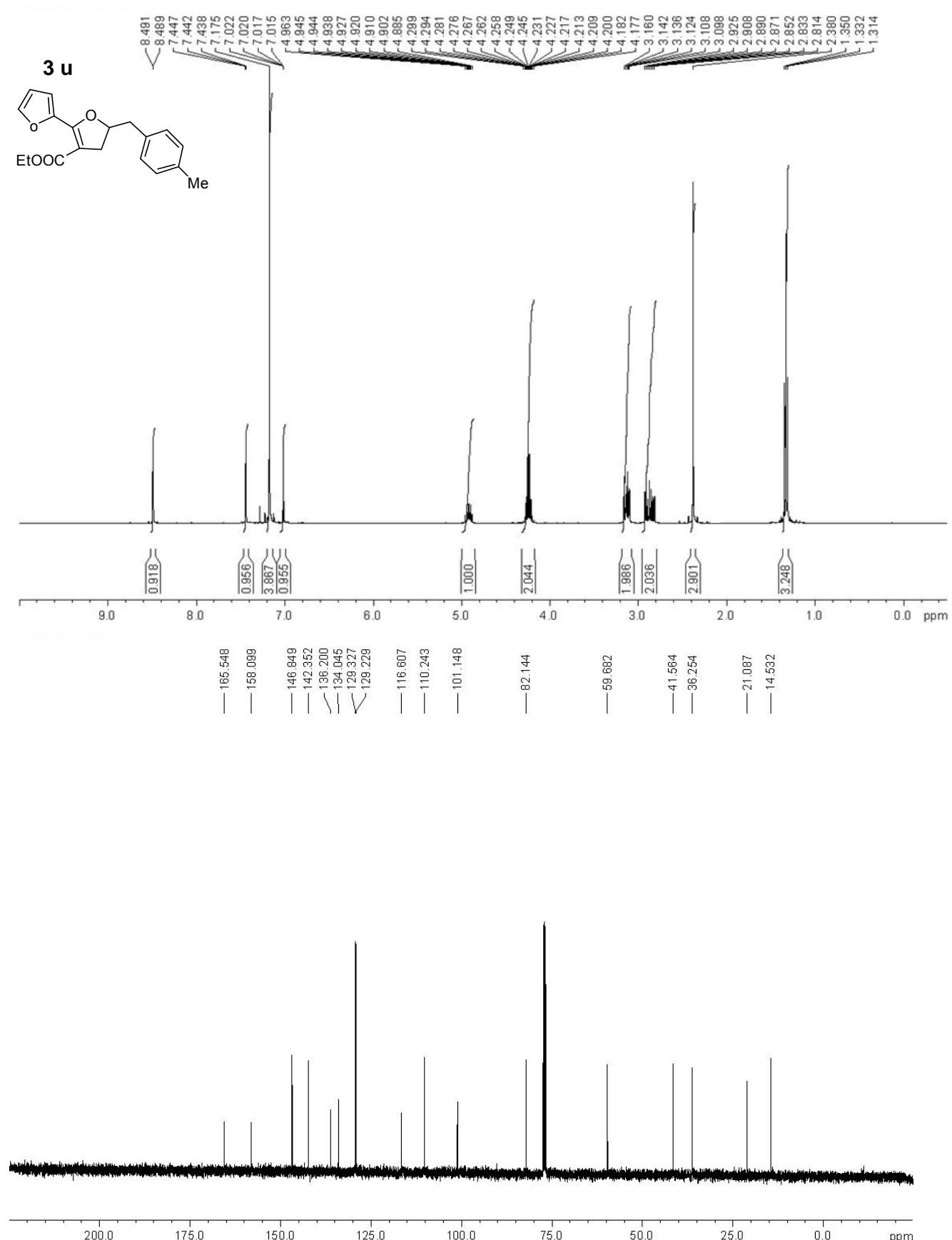


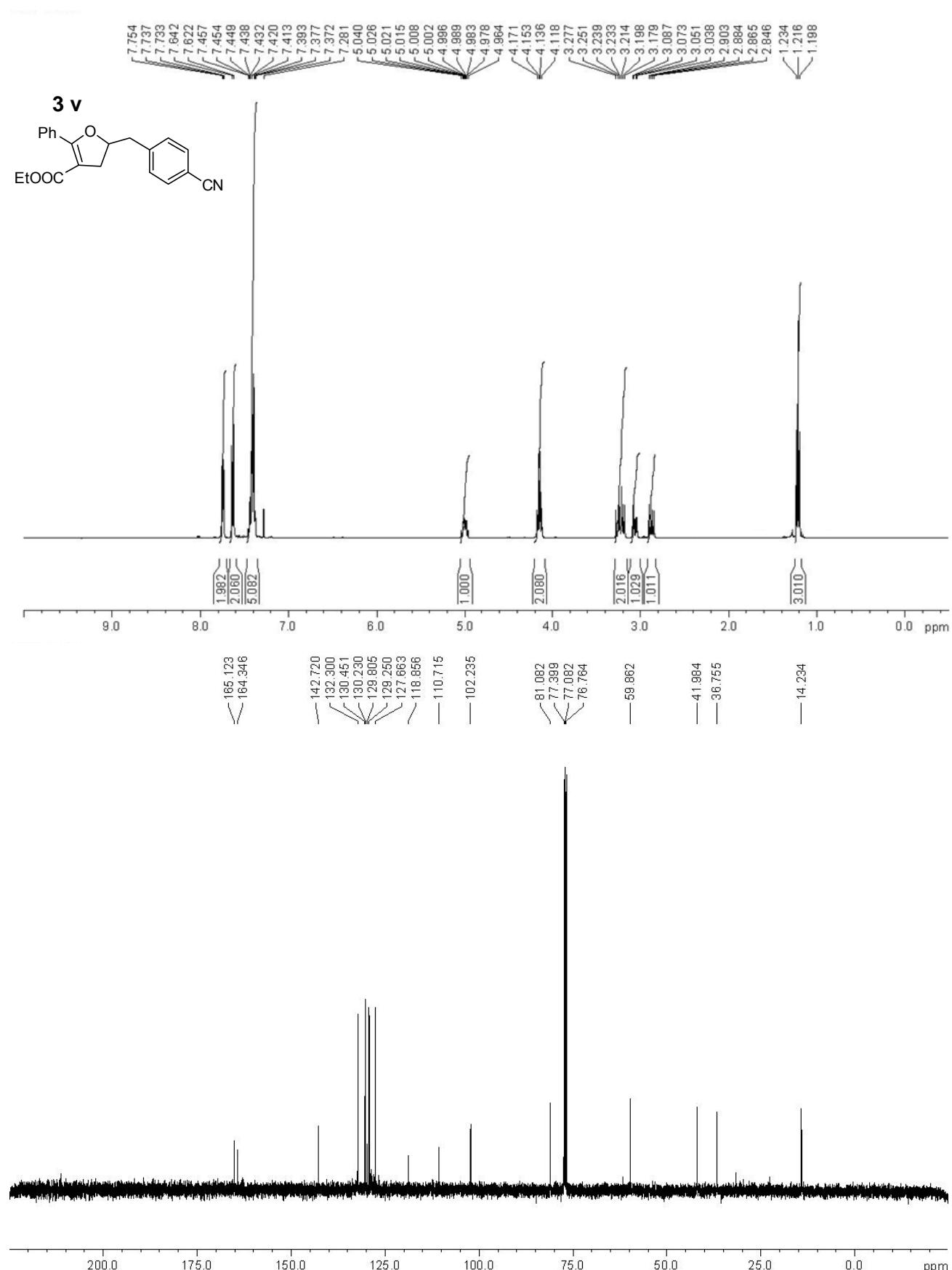


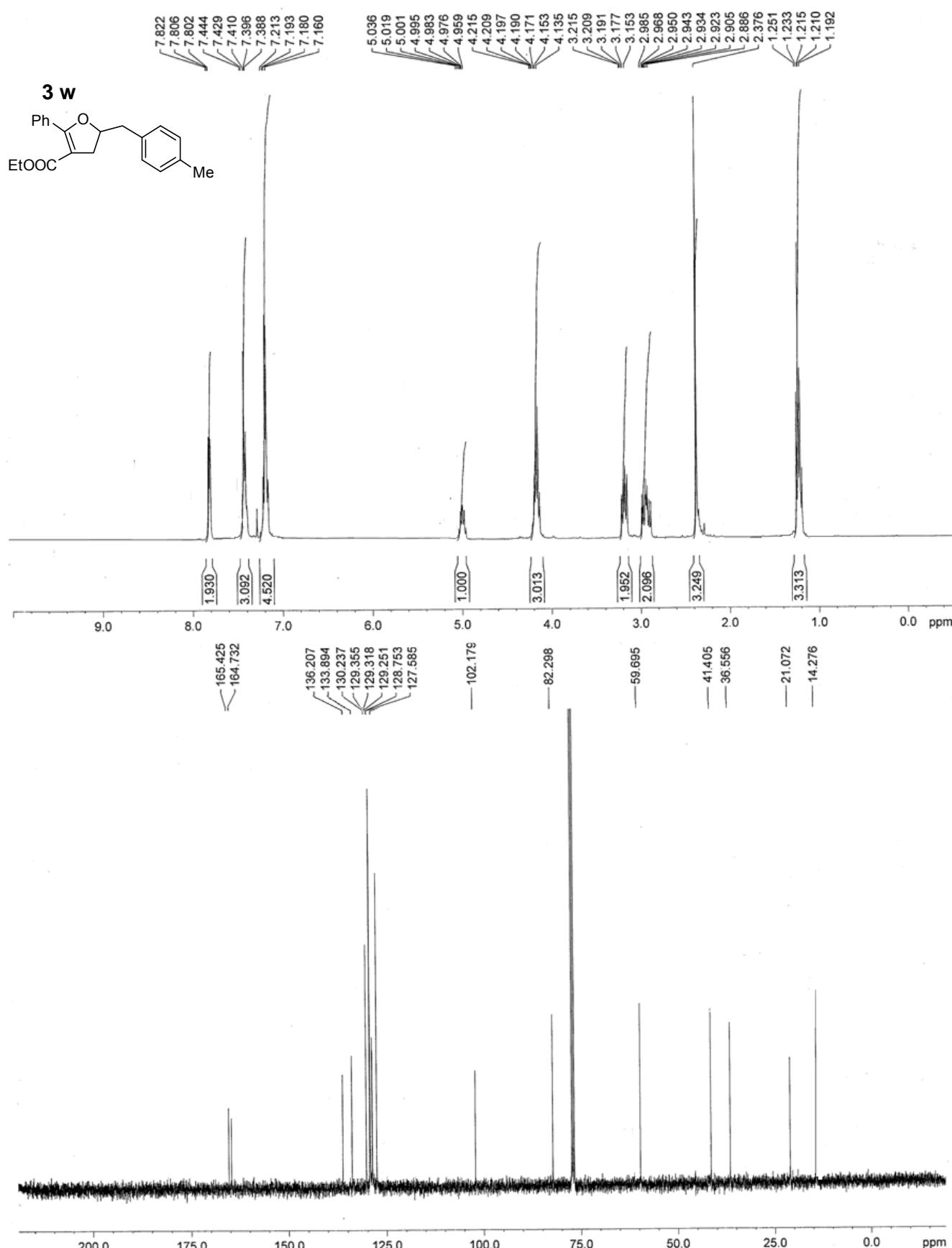


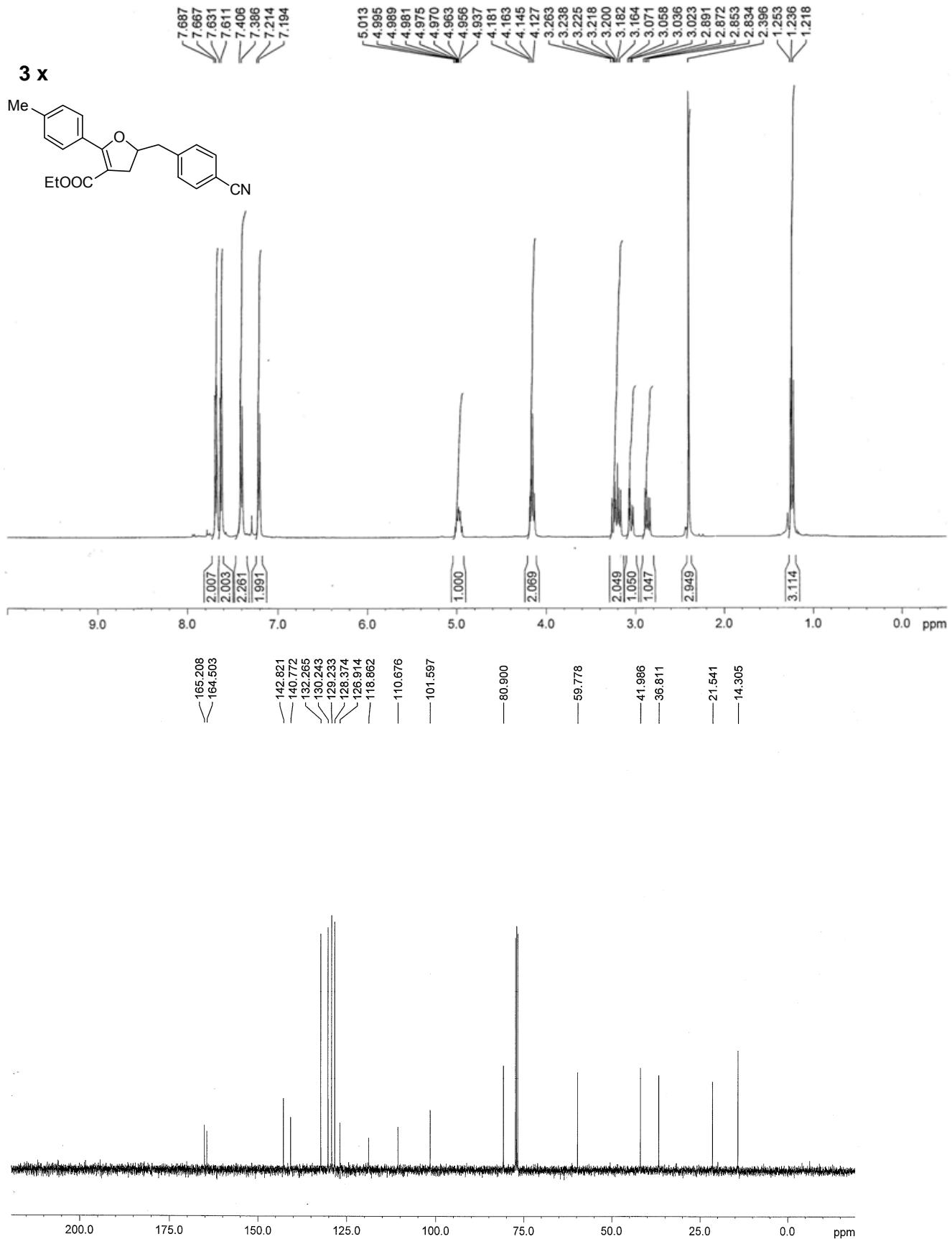


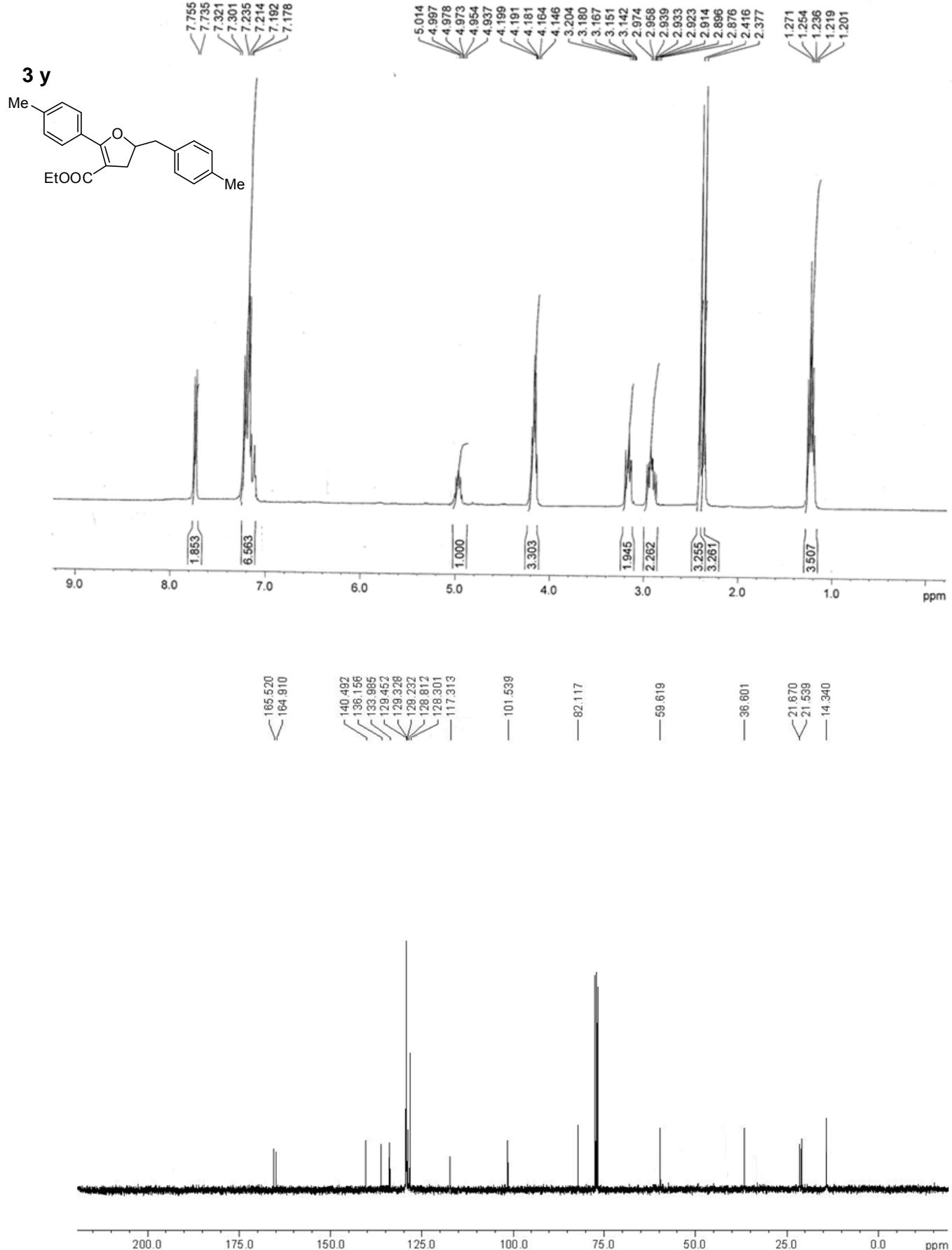


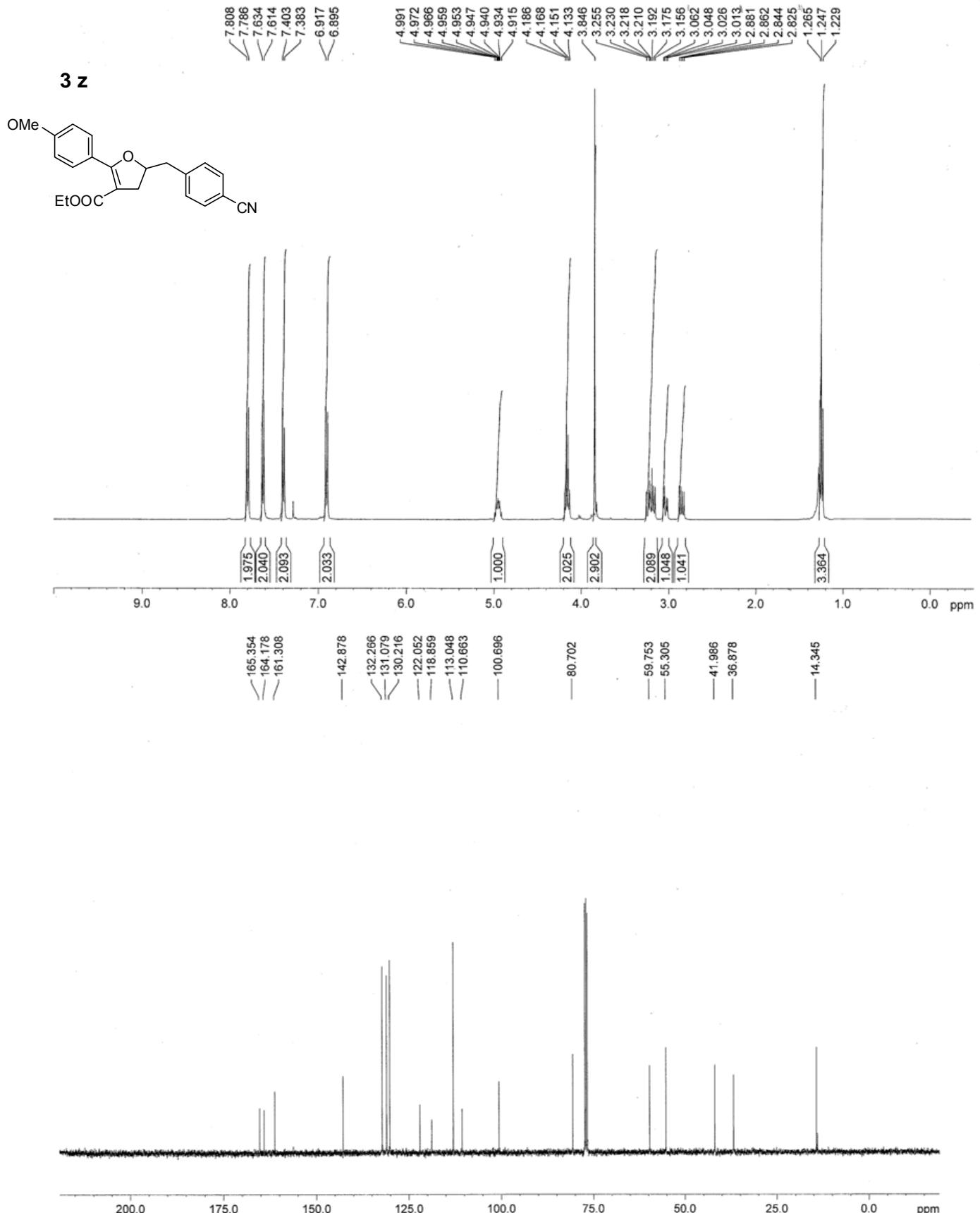




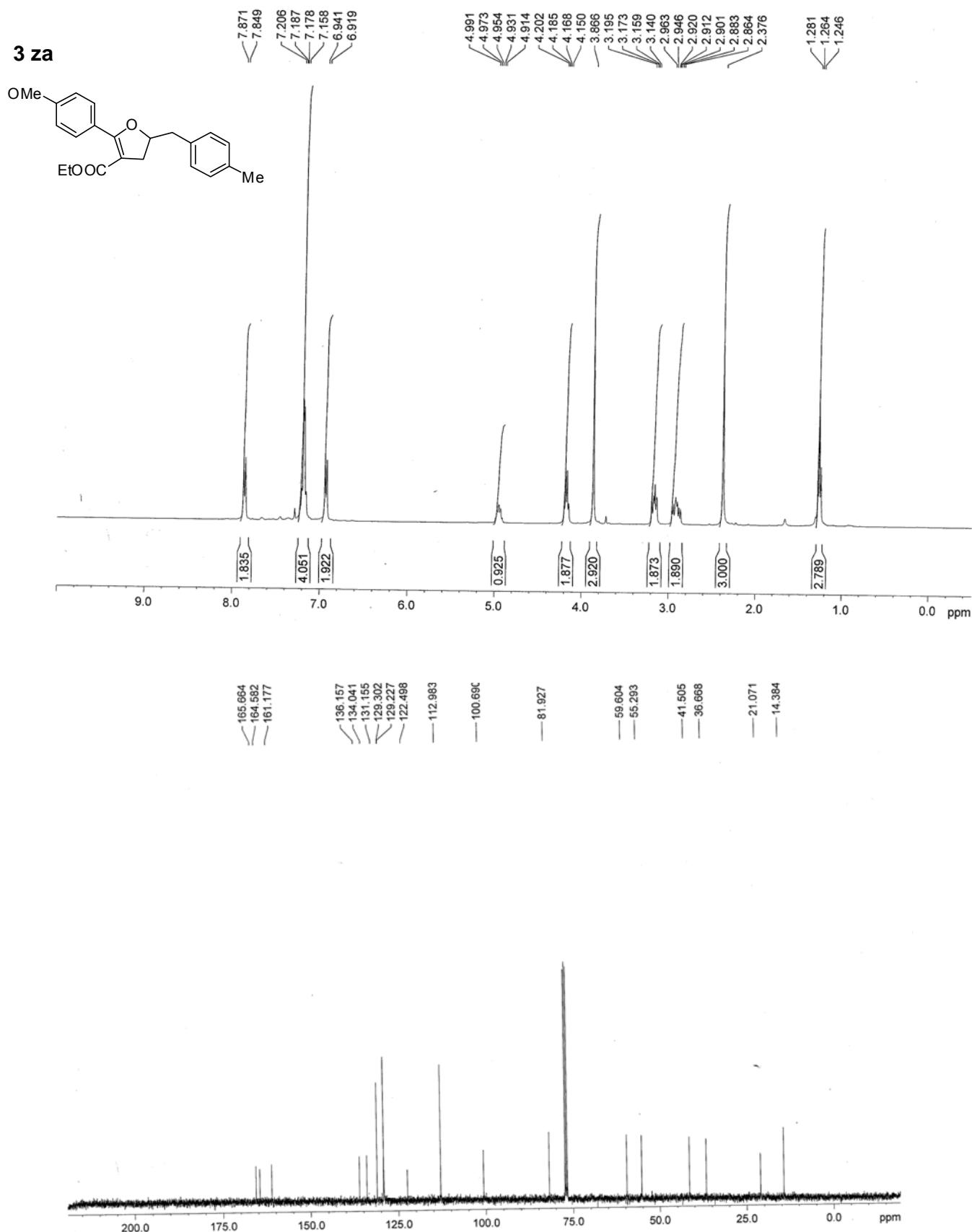


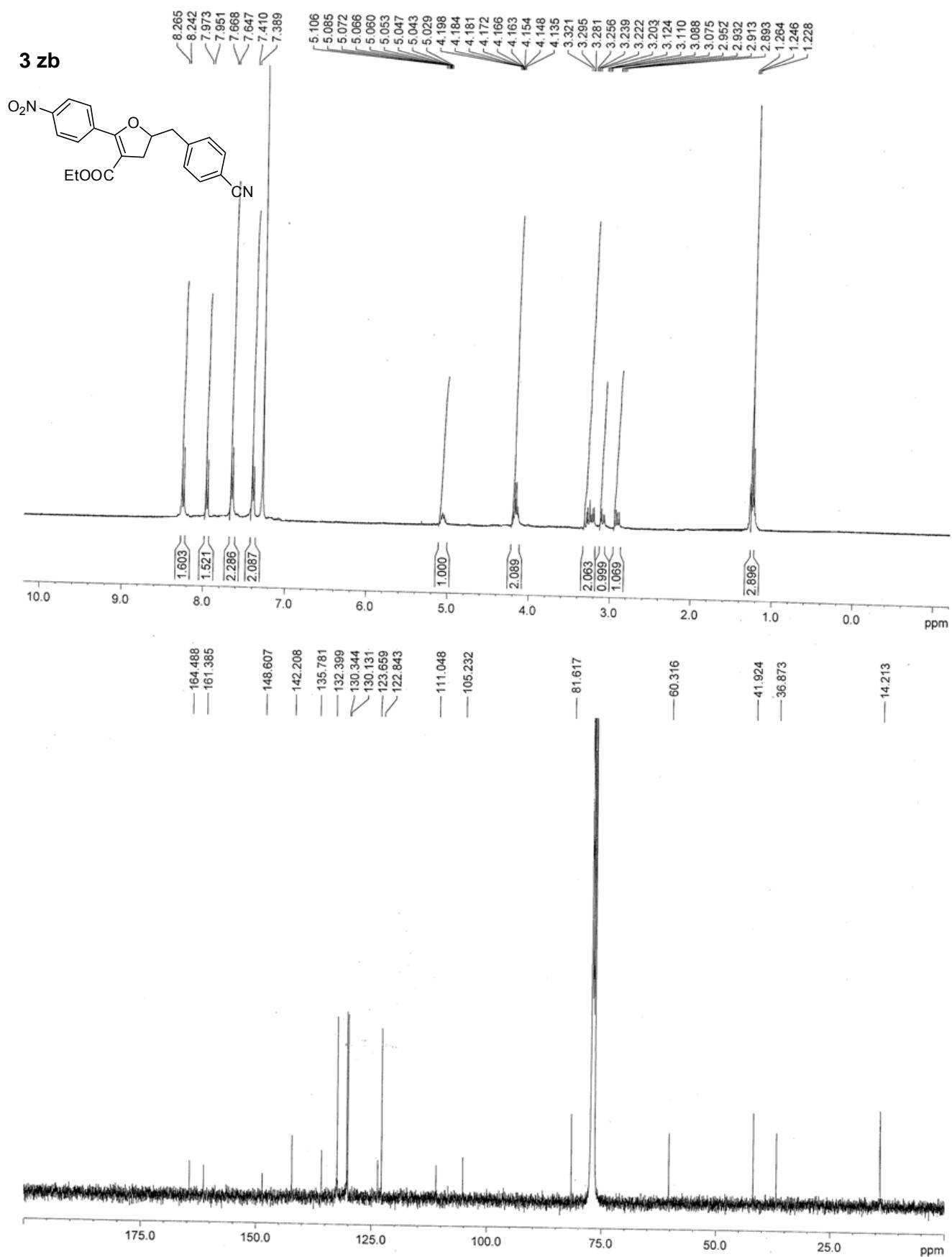


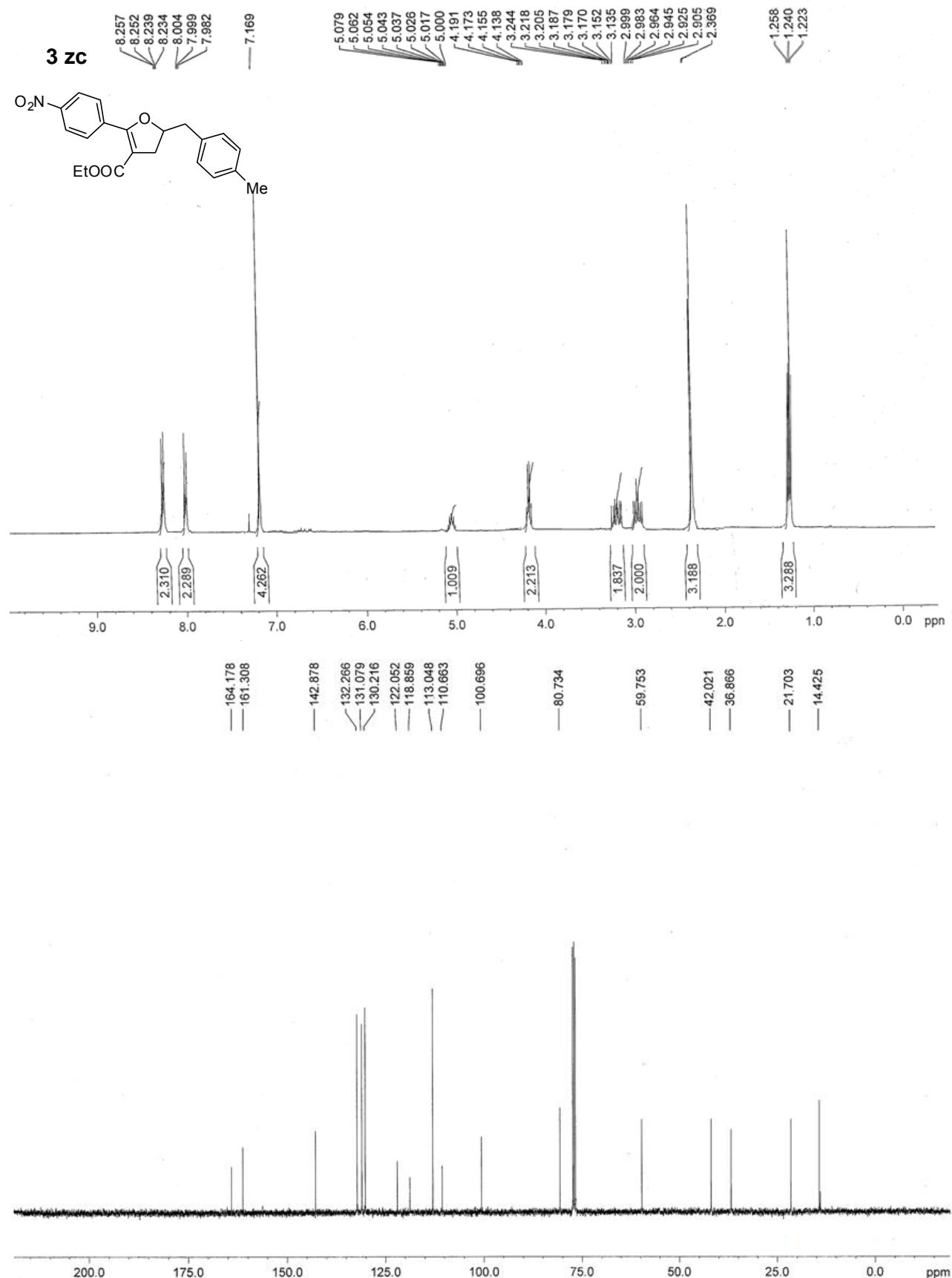


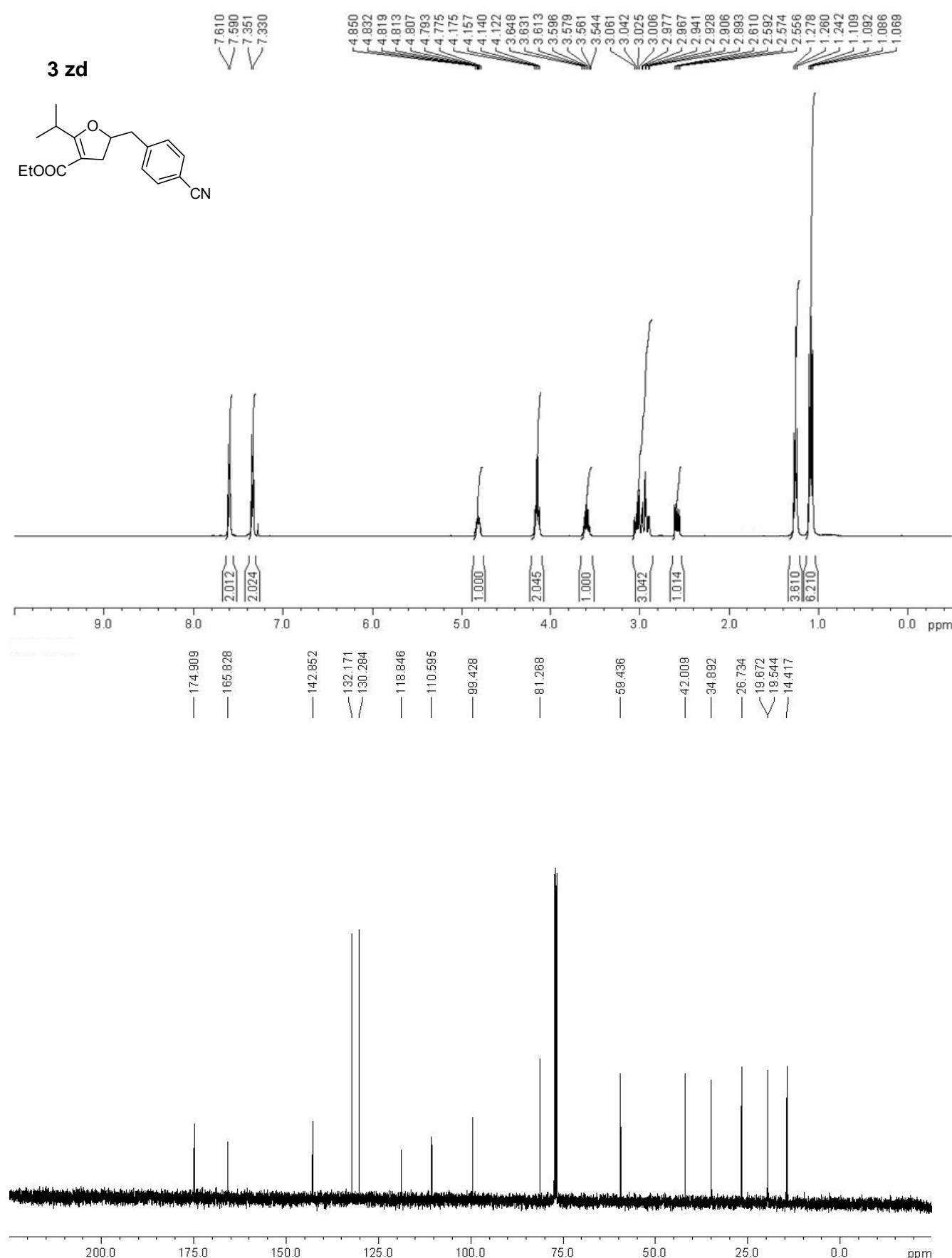


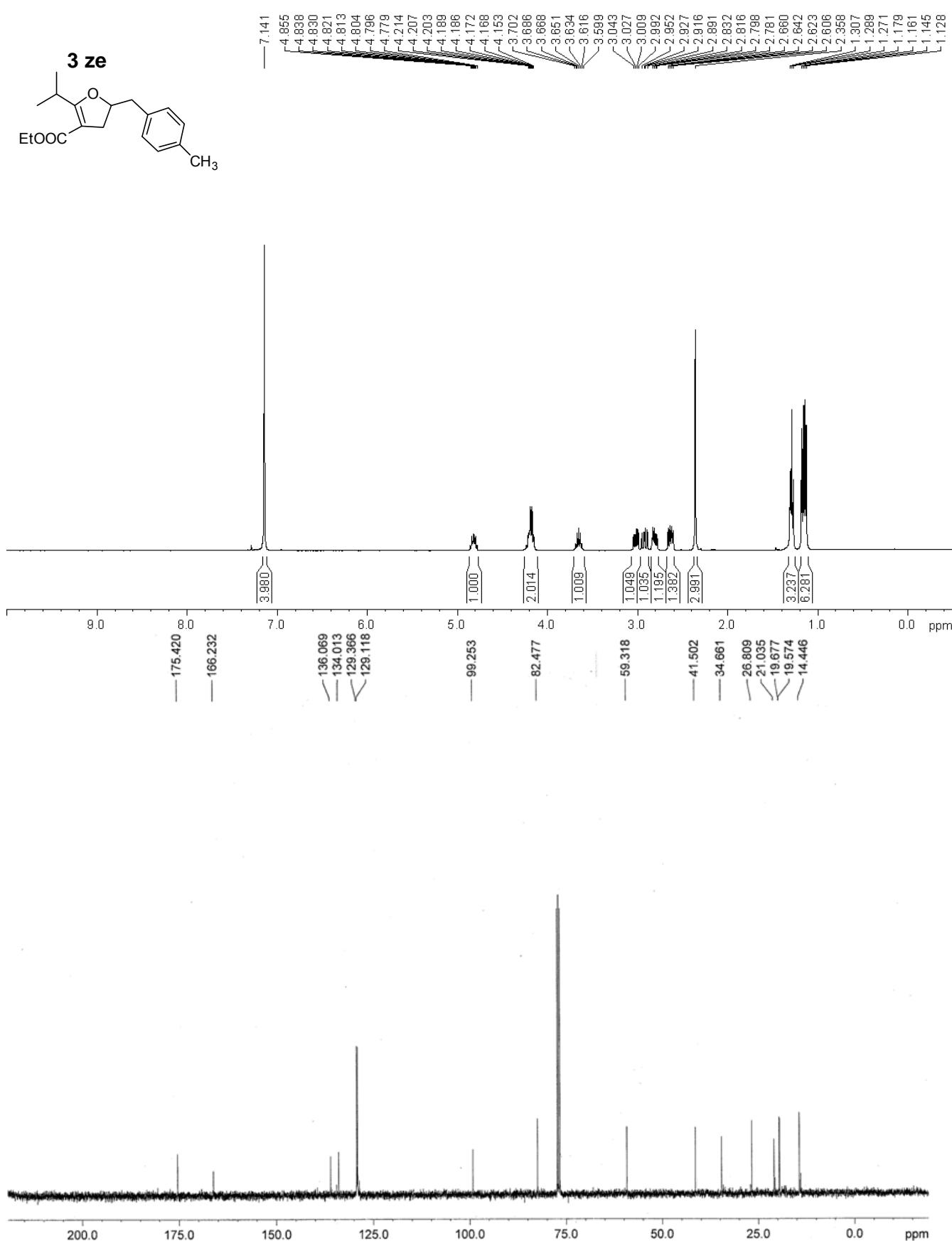
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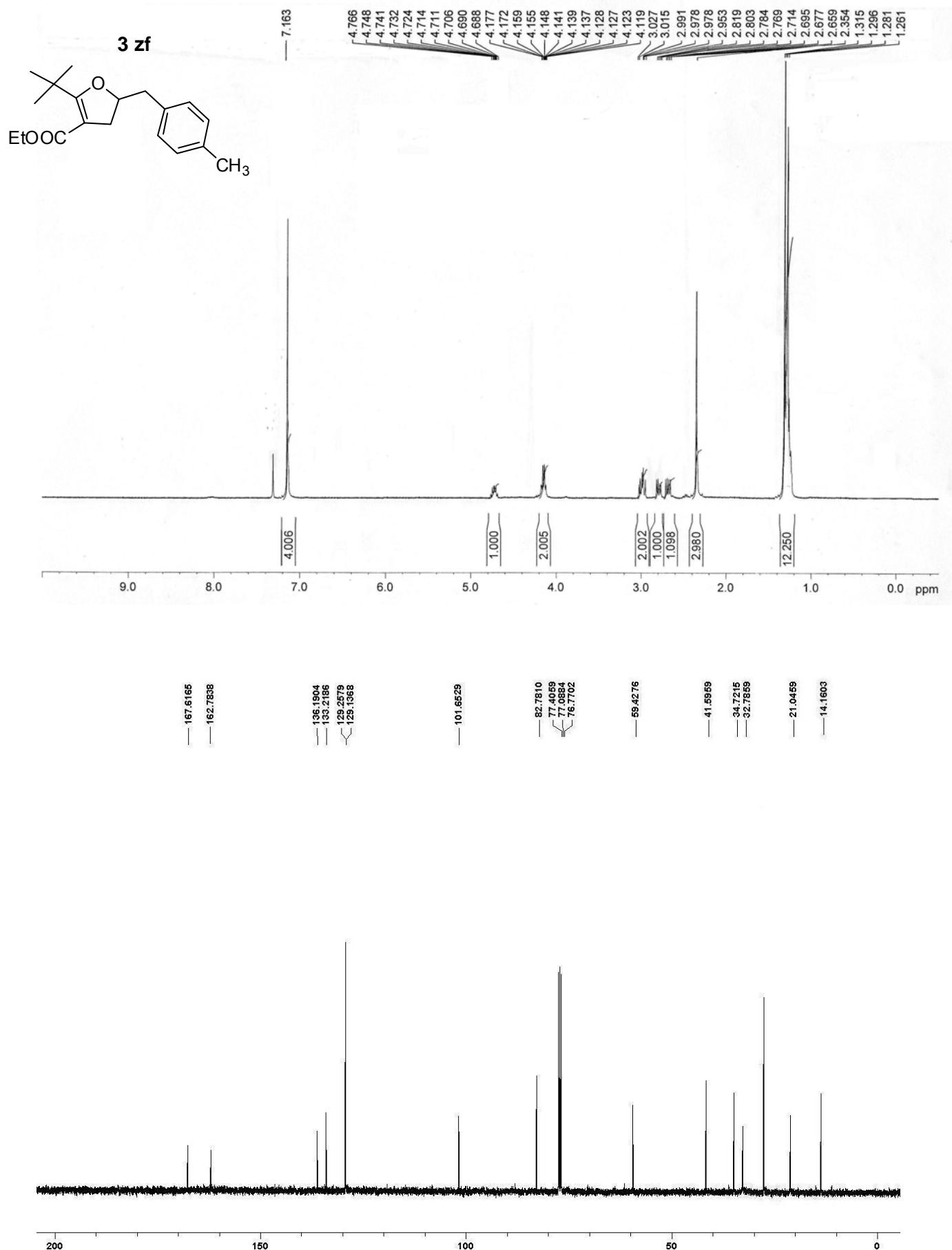












ⁱ Antonioletti, Roberto et al. *Gazzetta Chimica Italiana*, **1992** 122(6) 237-8.