

Supporting Information
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Direct C-3 Alkylation of Coumarins *via* Decarboxylative Coupling with Carboxylic Acids

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General remarks: The following includes general experimental procedures, specific details for representative reactions, isolation and spectroscopic information for the compounds. All reagents were commercially available and used as received. Coumarins coumarin-3-carboxylic acid derivatives were synthesized following the procedures described in the literature.¹ The reactions were carried out in an oil bath using Microwave Vials (2-5 ml). Column chromatography was carried out on silica gel (230–400 mesh). TLC was conducted on silica gel 250 micron, F254 plates. ¹H NMR spectra were recorded at room temperature on Bruker and Varian 300, 400 and 500 MHz spectrometers, using CDCl₃ as solvent. Chemical shifts are reported in ppm with TMS as an internal standard (TMS: δ 0.0 ppm). ¹³C NMR spectra are referenced from the solvent central peak (77.23 ppm). Chemical shifts are given in ppm. Mass spectra were recorded on Agilent 5975C Mass Spectrometer.

Typical Experimental Procedure for alkylation of coumarins. A vial equipped with a stir bar was charged with coumarin (0.2 mmol, 1equiv), carboxylic acid (0.4 mmol, 2 equiv), K₂S₂O₈ (0.4 mmol, 2 equiv), K₂CO₃ (0.4 mmol, 2 equiv), ACN/H₂O (0.1/0.5 mL). Then the vial was capped and the resulting mixture was heated in an oil bath at 120 °C for 2 h. Upon completion of the reaction, the resulting mixture was extracted with ethyl acetate. The combined organic layers was dried over anhydrous Na₂SO₄. After the removal of the solvent under reduced pressure, the crude product was purified by column chromatography on silica gel (EtOAc/hexane gradient, 5%) to provide the desired products.

3-Tert-butyl-2H-chromen-2-one (3a). Colorless solid (30 mg, 74%), mp 71-72 °C (Ref.² 82-83 °C); ¹H NMR (400 MHz, CDCl₃): δ 7.54 (s, 1H), 7.46-7.42 (m, 2H), 7.29-7.27 (m, 1H), 7.25-7.21 (m, 1H), 1.39 (s, 9H); ¹³C NMR (100 MHz, CDCl₃): δ 159.9, 153.2, 137.1, 136.5, 130.5, 127.5, 123.9, 119.4, 116.0, 35.0, 28.6; IR: 2926, 1712, 1611, 1476, 1273, 962, 776 cm⁻¹; MS analysis m/z 202 (M⁺, 34%), 187 (63), 160 (45), 147 (45), 83 (45), 57 (100); Anal. Calcd for C₁₃H₁₄O₂: C, 77.20; H, 6.98. Found: C, 77.56; H, 7.12.

3-Isopropyl-2H-chromen-2-one (3b). Colorless solid (23 mg, 62%), mp 60-61 °C (Ref.³, 47-48 °C); ¹H NMR (500 MHz, CDCl₃): δ 7.49 (s, 1H), 7.47-7.45 (m, 2H), 7.32 (m, 1H), 7.27 (m, 1H), 3.13 (t, J = 7.0 Hz, 1H), 1.28 (d, J = 7.0 Hz, 6H); ¹³C NMR (125 MHz, CDCl₃): δ 161.4, 152.8, 135.9, 135.6, 130.4, 127.3, 124.1, 119.5, 116.3, 28.7, 21.4; Anal. Calcd for C₁₂H₁₂O₂: C, 76.57; H, 6.43. Found: C, 76.89; H, 6.54.

¹ (a) Sánchez-Recillas, A.; Navarrete-Vázquez, G.; Hidalgo-Figueroa, S.; Rios, M. Y.; Ibarra-Barajas, M.; Estrada-Soto, S. *Eur. J. Med. Chem.*, **2014**, *77*, 400; (b) Plisson, F.; Huang, X.-C.; Zhang, H.; Khalil, Z.; Capon, R. J. *Chem. An Asian J.*, **2012**, *7*, 1616.

² Russell, G. A.; Shi, B. Z.; Jiang, W.; Hu, S.; Kim, B. H.; Baik, W. Electron transfer processes. 57. *J. Am. Chem. Soc.*, **1995**, *117*, 3952.

³ Yuan, J.-W.; Yang, L.-R.; Yin, Q.-Y.; Mao, P.; Qu, L.-B. *RSC Adv.*, **2016**, *6*, 35936.

3-(Heptan-3-yl)-2H-chromen-2-one (3c). Colorless oil (28 mg, 57%); ¹H NMR (500 MHz, CDCl₃): δ 7.47-7.44 (m, 2H), 7.43 (s, 1H), 7.31 (d, *J* = 8.5 Hz, 1H), 7.25 (t, *J* = 7.5 Hz, 1H), 2.81 (qui, *J* = 7.1 Hz, 1H), 1.71-1.62 (m, 4H), 1.34-1.19 (m, 4H), 0.87 (t, *J* = 7.5 Hz, 3H), 0.86 (t, *J* = 7.1 Hz, 3H); ¹³C NMR (125 MHz, CDCl₃): δ 161.5, 152.9, 137.8, 132.9, 130.4, 127.2, 124.1, 119.5, 116.2, 41.6, 33.2, 29.5, 26.7, 22.7, 13.9, 11.6. IR: 2929, 2864, 1710, 1610, 1455, 1225, 1173, 755 cm⁻¹; MS analysis *m/z* 244 (M⁺, 57%), 215 (36), 188 (78), 159 (100), 115 (39); Anal. Calcd for C₁₆H₂₀O₂: C, 78.65; H, 8.25. Found: C, 78.29; H, 8.10.

3-Cyclopropyl-2H-chromen-2-one (3d). Yellow solid (25 mg, 67%), mp 70-72 °C (Ref.⁴ 83-84 °C); ¹H NMR (500 MHz, CDCl₃): δ 7.45-7.40 (m, 1H), 7.39 (d, *J* = 7.8 Hz, 1H), 7.27 (d, *J* = 8.1 Hz, 1H), 7.25-7.19 (m, 2H), 2.08-2.01 (m, 1H), 1.05-0.97 (m, 2H), 0.77-0.74 (m, 2H); ¹³C NMR (125 MHz, CDCl₃): δ 161.9, 152.5, 134.4, 131.4, 130.3, 127.0, 124.3, 119.4, 116.3, 11.3, 7.7; IR: 3012, 1691, 1602, 1453, 1402, 1175, 1018, 746 cm⁻¹; MS analysis *m/z* 186 (M⁺, 100%), 171 (50), 157 (55), 131 (50), 115 (33). Anal; Calcd for C₁₂H₁₀O₂: C, 77.40; H, 5.41. Found: C, 77.69; H, 5.53.

3-Cyclobutyl-2H-chromen-2-one (3e). Yellow solid (20 mg, 51%), mp 72-74 °C; ¹H NMR (500 MHz, CDCl₃): δ 7.48 (s, 1H), 7.47-7.45 (m, 2H), 7.32 (d, *J* = 8.5 Hz, 1H), 7.28 (m, 1H), 3.60 (q, *J* = 8.7 Hz, 1H), 2.43-2.38 (m, 2H), 2.15-2.05 (m, 3H), 1.90-1.84 (m, 1H); ¹³C NMR (125 MHz, CDCl₃): δ 161.1, 152.8, 136.1, 132.9, 130.4, 127.2, 124.2, 119.5, 116.3, 36.0, 27.7, 18.2; IR: 2954, 2861, 1706, 1602, 1451, 1251, 1182, 1083, 969, 745 cm⁻¹; MS analysis *m/z* 200 (M⁺, 17%), 172 (100), 144 (37), 115 (33); Anal. Calcd for C₁₃H₁₂O₂: C, 77.98; H, 6.04. Found: C, 78.22; H, 6.14.

3-Cyclopentyl-2H-chromen-2-one (3f).⁵ Colorless solid (29 mg, 67%), mp 68-69°C; ¹H NMR (500 MHz, CDCl₃): δ 7.50 (s, 1H), 7.48-7.44 (m, 2H), 7.32 (d, *J* = 8.1 Hz, 1H), 7.27-7.24 (m, 1H), 3.16 (qui, *J* = 8.5 Hz, 1H), 2.14-2.08 (m, 2H), 1.85-1.70 (m, 4H), 1.63-1.56 (m, 2H); ¹³C NMR (125 MHz, CDCl₃): δ 161.7, 152.8, 135.9, 133.4, 130.3, 127.2, 124.1, 119.5, 116.3, 40.7, 31.7, 25.0; IR: 2958, 2867, 1764, 1689, 1453, 1178, 1076, 818, 778 cm⁻¹; MS analysis *m/z* 214 (M⁺, 100%), 171 (24), 157(50), 128 (15), 115 (33); Anal. Calcd for C₁₄H₁₄O₂: C, 78.48; H, 6.59. Found: C, 78.73; H, 6.72.

3-Cyclohexyl-2H-chromen-2-one (3g). Colorless solid (30 mg, 65%), mp 86-87 °C (Ref.⁴ 90-92 °C); ¹H NMR (500 MHz, CDCl₃): δ 7.47-7.44 (m, 3H), 7.30 (d, *J* = 8.5 Hz, 1H), 7.27-7.24 (m, 1H), 2.78 (tt, *J* = 11.8, 3.3 Hz, 1H), 2.00-1.97 (m, 2H), 1.87-1.85 (m, 2H), 1.79-1.77 (m, 1H), 1.50-1.41 (m, 2H), 1.35-1.24 (m, 3H); ¹³C NMR (125 MHz, CDCl₃): δ 161.5, 152.7, 136.3, 134.8, 130.4, 127.3, 124.1, 119.6, 116.2, 38.2, 32.1, 26.5, 26.1; Anal. Calcd for C₁₅H₁₆O₂: C, 78.92; H, 7.06. Found: C, 78.63; H, 6.95.

⁴ Hou, J.; Ee, A.; Feng, W.; Xu, J.-H.; Zhao, Y.; Wu, J. *J. Am. Chem. Soc.*, **2018**, *140*, 5257.

⁵ Banerjee, A.; Santra, S. K.; Khatun, N.; Ali, W.; Patel, B. K. *Chem. Commun.*, **2015**, *51*, 15422.

3-Propyl-2H-chromen-2-one (3h). Colorless oil (22 mg, 59%); ¹H NMR (500 MHz, CDCl₃): δ 7.47 (s, 1H), 7.46-7.42 (m, 2H), 7.29 (d, *J* = 8.3 Hz, 1H), 7.25-7.22 (m, 1H), 2.54 (t, *J* = 7.5 Hz, 2H), 1.71-1.64 (m, 2H), 0.99 (t, *J* = 7.3 Hz, 3H); ¹³C NMR (125 MHz, CDCl₃): δ 161.7, 153.1, 138.4, 130.4, 129.8, 127.0, 124.1, 119.5, 116.3, 32.8, 21.2, 13.7; IR: 3054, 2960, 1716, 1609, 1455, 1176, 1054, 753 cm⁻¹; MS analysis *m/z* 189 (M⁺, 100%), 173 (15), 159 (15), 147 (30); Anal. Calcd for C₁₂H₁₂O₂: C, 76.57; H, 6.43. Found: C, 76.92; H, 6.55.

3-Methyl-2H-chromen-2-one (3i). Colorless solid (15 mg, 47%), mp 90-91 °C (Ref.⁴ 86-87 °C); ¹H NMR (400 MHz, CDCl₃): δ 7.44 (s, 1H), 7.40-7.32 (m, 2H), 7.23 (d, *J* = 8.2 Hz, 1H), 7.19-7.15 (m, 1H), 2.14 (d, *J* = 1.4 Hz 3H); ¹³C NMR (100 MHz, CDCl₃): δ 162.3, 153.3, 139.3, 130.5, 127.0, 125.9, 124.3, 119.6, 116.5, 17.2; Anal. Calcd for C₁₀H₈O₂: C, 74.99; H, 5.03. Found: C, 74.75; H, 4.03.

3-Tert-butyl-7-methoxy-2H-chromen-2-one (3k). Colorless solid (38 mg, 82%), mp 82-83 °C; ¹H NMR (300 MHz, CDCl₃): δ 7.48 (s, 1H), 7.33 (d, *J* = 8.4 Hz, 1H), 6.81-6.75 (m, 2H), 3.84 (s, 3H), 1.36 (s, 9H); ¹³C NMR (75 MHz, CDCl₃): δ 161.8, 160.2, 154.7, 136.7, 133.3, 128.5, 113.0, 112.1, 99.9, 55.6, 34.7, 28.6; IR: 2958, 1711, 1611, 1576, 1476, 1275, 1099, 962, 776 cm⁻¹; MS analysis *m/z* 232 (M⁺, 81%), 217 (100), 189 (93), 174 (15), 149 (15), 129 (12); Anal. Calcd for C₁₄H₁₆O₃: C, 72.39; H, 6.94. Found: C, 72.60; H, 7.05.

3-Tert-butyl-6-bromo-2H-chromen-2-one (3l). Colorless solid (34 mg, 60%), mp 128-130 °C; ¹H NMR (500 MHz, CDCl₃): δ 7.61 (d, *J* = 2.5 Hz, 1H), 7.54 (dd, *J* = 8.5, 2.5 Hz, 1H), 7.47 (s, 1H), 7.18 (d, *J* = 8.5 Hz, 1H), 1.40 (s, 9H); ¹³C NMR (125 MHz, CDCl₃): δ 159.1, 152.0, 138.4, 135.3, 133.2, 129.9, 121.0, 117.7, 116.4, 35.2, 28.5; IR: 2956, 1712, 1602, 1562, 1478, 1360, 1250, 1214, 1139, 979, 812 cm⁻¹; MS analysis *m/z* 280 (M⁺, 70%), 265 (100), 238 (94), 158 (41), 128 (23), 115 (29); Anal. Calcd for C₁₃H₁₃BrO₂: C, 55.54; H, 4.66. Found: C, 55.80; H, 4.80.

6-Bromo-3-cyclohexyl-2H-chromen-2-one (3m). Colorless solid (33 mg, 54%), mp 125-128 °C; ¹H NMR (500 MHz, CDCl₃): δ 7.60 (d, *J* = 2.0 Hz, 1H), 7.55 (dd, *J* = 8.5, 2.0 Hz, 1H), 7.37 (s, 1H), 7.20 (d, *J* = 8.5 Hz, 1H), 2.79 (m, 1H), 2.04-1.92 (m, 2H), 1.93-1.80 (m, 2H), 1.79 (m, 1H), 1.53-1.37 (m, 2H), 1.35-1.17 (m, 3H); ¹³C NMR (125 MHz, CDCl₃): δ 160.8, 151.5, 136.2, 134.9, 133.1, 129.6, 121.2, 118.0, 116.6, 38.3, 32.0, 26.4, 26.1; IR: 2918, 2853, 1707, 1475, 1242, 1065, 812 cm⁻¹; MS analysis *m/z* 306 (M⁺, 100%), 250 (90), 238 (72), 224 (31), 128 (32), 115 (72); Anal. Calcd for C₁₅H₁₅BrO₂: C, 58.65; H, 4.92. Found: C, 58.99; H, 5.07.

3-Tert-butyl-8-methoxy-2H-chromen-2-one (3n). Colorless solid (35 mg, 75%), mp 80-81 °C; ¹H NMR (400 MHz, CDCl₃): δ 7.51 (s, 1H), 7.18-7.14 (m, 1H), 7.03-6.98 (m, 2H), 3.95 (s, 3H), 1.38 (s, 9H); ¹³C NMR (100 MHz, CDCl₃): δ 159.3, 146.9, 143.0,

137.5, 136.7, 122.7, 123.9, 120.2, 119.2, 112.5, 56.3, 35.2, 28.7; IR: 2956, 1711, 1611, 1576, 1473, 1273, 1099, 962, 776 cm^{-1} ; MS analysis m/z 232 (M^+ , 90%), 217 (100), 190 (72), 174 (31), 161 (9), 146 (9); Anal. Calcd for $\text{C}_{14}\text{H}_{16}\text{O}_3$: C, 72.39; H, 6.94. Found: C, 72.10; H, 6.83.

3-(Heptan-3-yl)-8-methoxy-2H-chromen-2-one (3o). Yellow oil (39 mg, 72%); ^1H NMR (500 MHz, CDCl_3): δ 7.40 (s, 1H), 7.19 (m, 1H), 7.04 (m, 2H), 3.97 (s, 3H), 2.82 (qui, $J = 7.0$ Hz, 1H), 1.71-1.63 (m, 4H), 1.34-1.21 (m, 4H), 0.87 (t, $J = 7.5$ Hz, 3H), 0.86 (t, $J = 7.1$ Hz, 3H); ^{13}C NMR (125 MHz, CDCl_3): δ 160.9, 147.0, 142.6, 137.9, 133.2, 123.9, 120.1, 118.7, 112.4, 56.2, 41.6, 33.2, 29.4, 26.7, 22.7, 14.0, 11.6; IR: 2928, 2862, 1711, 1610, 1473, 1271, 1101, 773, 730 cm^{-1} ; MS analysis m/z 274 (M^+ , 63%), 217 (78), 203 (42), 189 (100), 161 (23), 115 (15); Anal. Calcd for $\text{C}_{17}\text{H}_{22}\text{O}_3$: C, 74.42; H, 8.08. Found: C, 74.72; H, 8.20.

3-Isopropyl-7-methoxy-4-methyl-2H-chromen-2-one (3p). Colorless solid (26 mg, 55%), mp 82-84 $^\circ\text{C}$; ^1H NMR (500 MHz, CDCl_3): δ 7.49 (d, $J = 9.0$ Hz, 1H), 6.81 (dd, $J = 9.0, 2.5$ Hz, 1H), 6.74 (d, $J = 2.5$ Hz, 1H), 3.83 (s, 3H), 3.27 (t, $J = 7.0$ Hz, 1H), 2.39 (s, 3H), 1.34 (d, $J = 7.0$ Hz, 6H); ^{13}C NMR (125 MHz, CDCl_3): δ 161.5, 160.2, 153.8, 145.2, 127.9, 125.7, 114.3, 111.8, 100.3, 55.7, 28.6, 20.0, 14.7; IR: 2918, 2849, 1691, 1608, 1462, 1290, 1150, 1063, 929, 847 cm^{-1} ; MS analysis m/z 232 (M^+ , 70%), 217 (100), 203 (32), 189 (29), 174 (8), 115 (8); Anal. Calcd for $\text{C}_{14}\text{H}_{16}\text{O}_3$: C, 72.39; H, 6.94. Found: C, 72.68; H, 7.07.

3-Tert-butyl-6-nitro-2H-chromen-2-one (3q). Colorless solid (10 mg, 20%), mp 112-115 $^\circ\text{C}$; ^1H NMR (400 MHz, CDCl_3): δ 8.41 (d, $J = 2.6$ Hz, 1H), 8.32 (dd, $J = 9.0, 2.6$ Hz, 1H), 7.61 (s, 1H), 7.40 (d, $J = 9.0$ Hz, 1H), 1.41 (s, 9H); ^{13}C NMR (100 MHz, CDCl_3): δ 157.2, 155.7, 142.8, 138.6, 134.4, 124.4, 122.4, 118.4, 116.1, 34.4, 27.4; IR: 2937, 2851, 1698, 1608, 1542, 1361, 1150, 1063, 929, 847 cm^{-1} ; MS analysis m/z 247 (M^+ , 54%), 232 (95), 205 (100), 186 (28), 158 (50), 115 (31); Anal. Calcd for $\text{C}_{13}\text{H}_{13}\text{NO}_4$: C, 63.15; H, 5.30; N, 5.67. Found: C, 63.48; H, 5.45; N, 5.89.

3-Tert-butyl-7-methyl-2H-chromen-2-one (3r). Colorless solid (32 mg, 75%), mp 70-73 $^\circ\text{C}$; ^1H NMR (400 MHz, CDCl_3): δ 7.49 (s, 1H), 7.32 (d, $J = 7.8$ Hz, 1H), 7.07 (s, 1H), 7.03 (d, $J = 7.8$ Hz, 1H), 2.41 (s, 3H), 1.37 (s, 9H); ^{13}C NMR (100 MHz, CDCl_3): δ 160.2, 153.4, 141.6, 136.7, 135.9, 127.3, 125.2, 117.1, 116.2, 35.0, 28.7, 21.8; IR: 2957, 2863, 1765, 1697, 1450, 1178, 1088, 812, 776 cm^{-1} ; MS analysis m/z 216 (M^+ , 10%), 201 (20), 167 (40), 149 (100), 113 (12), 57 (40); Anal. Calcd for $\text{C}_{14}\text{H}_{16}\text{O}_2$: C, 77.75; H, 7.46. Found: C, 77.49; H, 7.37.

3-Cyclopentyl-6-methyl-2H-chromen-2-one (3s). Colorless solid (32 mg, 70%), mp 60-61 $^\circ\text{C}$; ^1H NMR (500 MHz, CDCl_3): δ 7.44 (s, 1H), 7.27-7.23 (m, 2H), 7.20 (m, 1H), 7.05 (d, $J = 7.8$ Hz, 1H), 3.15 (qui, $J = 8.2$ Hz, 1H), 2.40 (s, 3H), 2.12-2.06 (m, 2H),

1.83-1.69 (m, 4H), 1.62-1.55 (m, 2H); ^{13}C NMR (125 MHz, CDCl_3): δ 161.9, 152.9, 141.4, 135.9, 132.1, 126.8, 125.3, 117.1, 116.4, 40.6, 31.7, 25.0, 21.6; IR: 2947, 2867, 1766, 1697, 1450, 1178, 1088, 812, 778 cm^{-1} ; MS analysis m/z 228 (M^+ , 100%), 200 (12), 187 (40), 171 (24), 157(10), 128 (15); Anal. Calcd for $\text{C}_{15}\text{H}_{16}\text{O}_2$: C, 78.92; H, 7.06. Found: C, 79.16; H, 7.16.

3-Cyclohexyl-7-methoxy-2H-chromen-2-one (3t). Colorless solid (41 mg, 80%), mp 107-109 °C (Ref.⁶ 91.9-92.5 °C); ^1H NMR (400 MHz, CDCl_3): δ 7.36 (s, 1H), 7.31 (d, J = 8.4 Hz, 1H), 6.80 (dd, J = 8.4, 2.4 Hz, 1H), 6.77 (d, J = 2.4 Hz, 1H), 3.83 (s, 3H), 2.74-2.67 (m, 1H), 1.96-1.91 (m, 2H), 1.84-1.79 (m, 2H), 1.76-1.72 (m, 1H), 1.47-1.36 (m, 2H), 1.31-1.19 (m, 3H); ^{13}C NMR (100 MHz, CDCl_3): δ 161.8, 161.7, 154.3, 136.3, 131.2, 128.1, 113.2, 112.1, 100.3, 55.6, 37.9, 32.1, 26.5, 26.1; IR: 2925, 2872, 1715, 1630, 1473, 1271, 1101, 775, 730 cm^{-1} ; MS analysis m/z 258 (M^+ , 100%), 243 (11), 215 (26), 202 (35), 189 (77), 159 (24); Anal. Calcd for $\text{C}_{16}\text{H}_{18}\text{O}_3$: C, 74.40; H, 7.02. Found: C, 74.75; H, 7.18.

2-Tert-butyl-3H-benzo[f]chromen-3-one (3u). Colorless solid (29 mg, 57%), mp 130-132 °C; ^1H NMR (500 MHz, CDCl_3): δ 8.35 (s, 1H), 8.27 (d, J = 8.0 Hz, 1H), 7.91 (d, J = 9.0, 1H), 7.90 (d, J = 8.0 Hz, 1H), 7.70-7.66 (m, 1H), 7.57-7.54 (m, 1H), 7.44 (d, J = 9.0 Hz, 1H), 1.50 (s, 9H); ^{13}C NMR (125 MHz, CDCl_3): δ 159.9, 152.5, 136.1, 132.1, 131.7, 130.2, 129.1, 127.8, 125.6, 121.2, 116.5, 113.1, 35.3, 28.6; IR: 3061, 2946, 1695, 1573, 1356, 1251, 983, 808, 736 cm^{-1} ; MS analysis m/z 252 (M^+ , 61%), 237 (100), 209 (60), 165 (30); Anal. Calcd for $\text{C}_{17}\text{H}_{16}\text{O}_2$: C, 80.93; H, 6.39. Found: C, 81.32; H, 6.56.

6-Chloro-3-isopropyl-2H-chromen-2-one (3v). Colorless solid (32 mg, 73%), mp 102-104 °C; ^1H NMR (500 MHz, CDCl_3): δ 7.43 (d, J = 2.5 Hz, 1H), 7.39 (dd, J = 8.8, 2.5 Hz, 1H), 7.38 (s, 1H), 7.24 (d, J = 8.8 Hz, 1H), 3.10 (hpt, J = 7.0 Hz, 1H), 1.25 (d, J = 7.0 Hz, 6H); ^{13}C NMR (125 MHz, CDCl_3): δ 160.6, 151.2, 137.0, 134.6, 130.3, 129.3, 126.5, 120.6, 117.7, 28.8, 21.3; IR: 3054, 2961, 1715, 1468, 1250, 1150, 1004, 816 cm^{-1} ; MS analysis m/z 222 (M^+ , 63%), 207 (100), 194 (54), 179 (36), 115 (50); Anal. Calcd for $\text{C}_{12}\text{H}_{11}\text{ClO}_2$: C, 64.73; H, 4.98. Found: C, 64.98; H, 5.10.

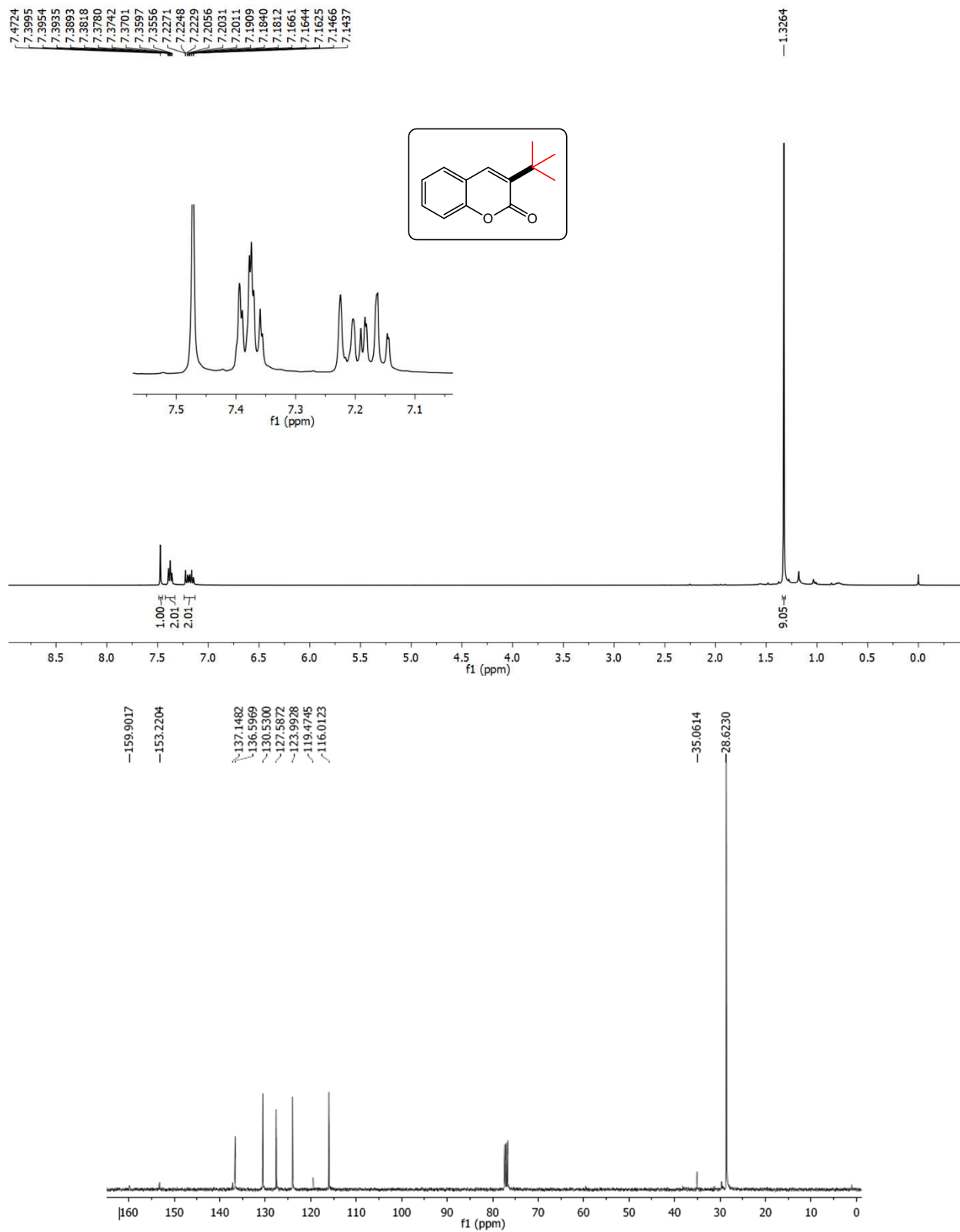
3-Phenyl-2H-chromen-2-one (4a). Colorless solid, isolated yield 40% (18 mg, 40%), mp 134-136 °C (Ref.⁷ 136-137 °C); ^1H NMR (500 MHz, CDCl_3): δ 7.81 (s, 1H), 7.71-7.69 (m, 2H), 7.55-7.50 (m, 2H), 7.46-7.39 (m, 3H), 7.36 (d, J = 8.2 Hz, 1H), 7.29 (tt, J = 7.5, 1.2 Hz, 1H); ^{13}C NMR (125 MHz, CDCl_3): δ 160.6, 153.6, 139.9, 134.8, 131.5, 128.9, 128.6, 128.5, 128.4, 128.0, 124.6, 119.7, 116.5; Anal. Calcd for $\text{C}_{15}\text{H}_{10}\text{O}_2$: C, 81.07; H, 4.54. Found: C, 81.36; H, 4.65.

⁶ Wang, C.; Mi, X.; Li, Q.; Li, Y.; Huang, M.; Zhang, J.; Wu, Y. *Tetrahedron*, **2015**, *71*, 6689.

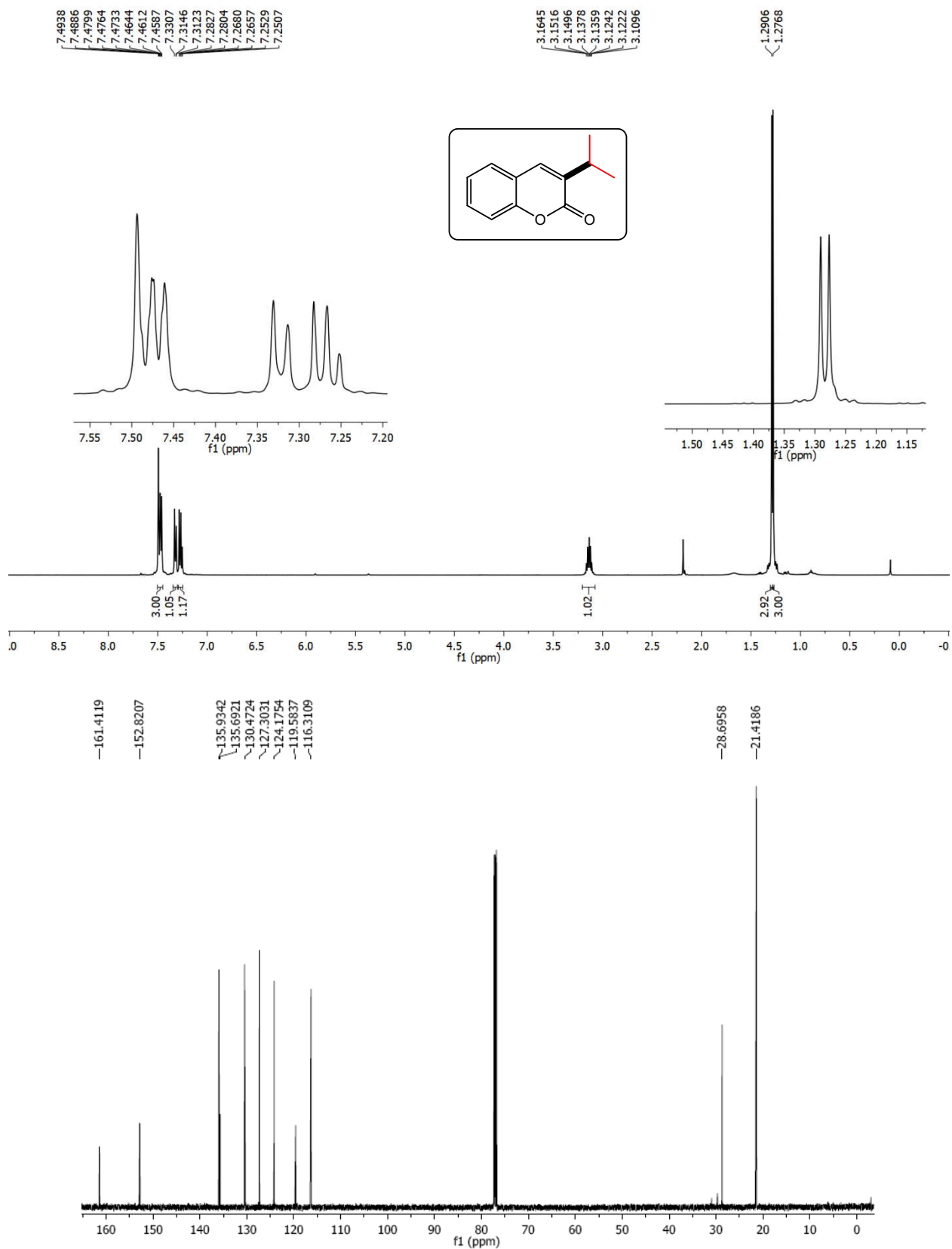
⁷ Zeng, H. Y.; Li, C. *J. Angew. Chem. Int. Ed.*, **2014**, *53*, 13862.

3-p-Tolyl-2H-chromen-2-one (4b). Colorless solid (25 mg, 53%), mp 144-146 °C (Ref.⁷ 158-159 °C); ¹H NMR (500 MHz, CDCl₃): δ 7.79 (s, 1H), 7.62-7.60 (m, 1H), 7.61 (d, *J* = 8.2, Hz, 1H), 7.54-7.50 (m, 2H), 7.36 (d, *J* = 8.2 Hz, 1H), 7.29 (td, *J* = 7.5, 1.1 Hz, 1H), 7.27 (d, *J* = 7.5 Hz, 2H), 2.40 (s, 3H); ¹³C NMR (125 MHz, CDCl₃): δ 160.8, 153.5, 139.3, 139.0, 131.9, 131.3, 129.3, 128.5, 128.5, 127.9, 124.5, 119.9, 116.5, 21.4; Anal. Calcd for C₁₆H₁₂O₂: C, 81.34; H, 5.12. Found: C, 81.68; H, 5.26.

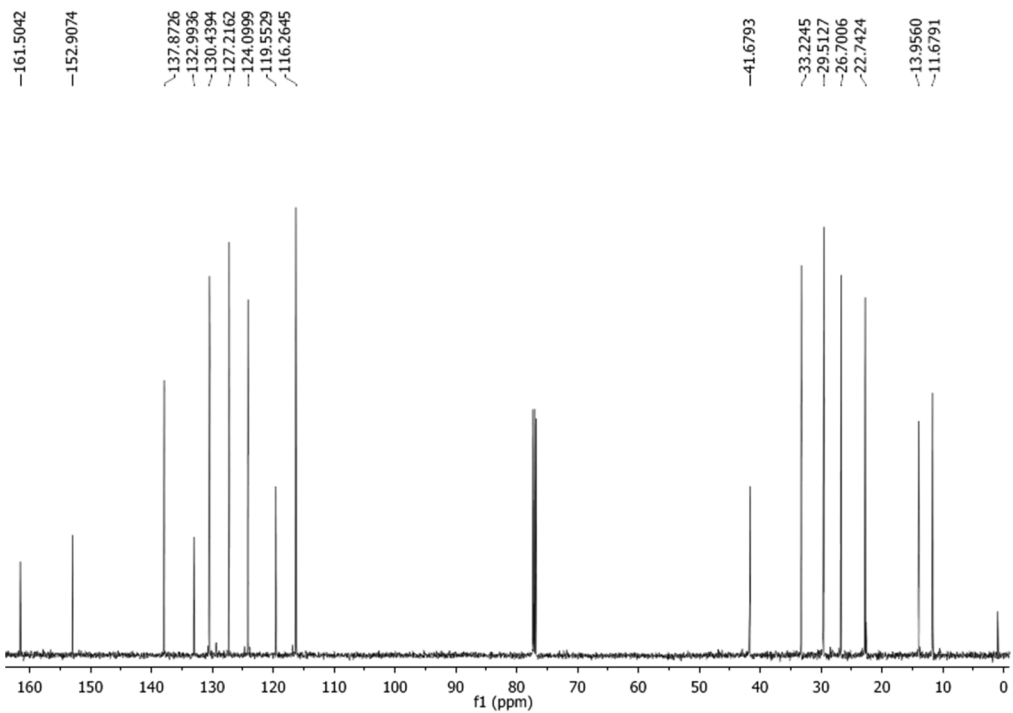
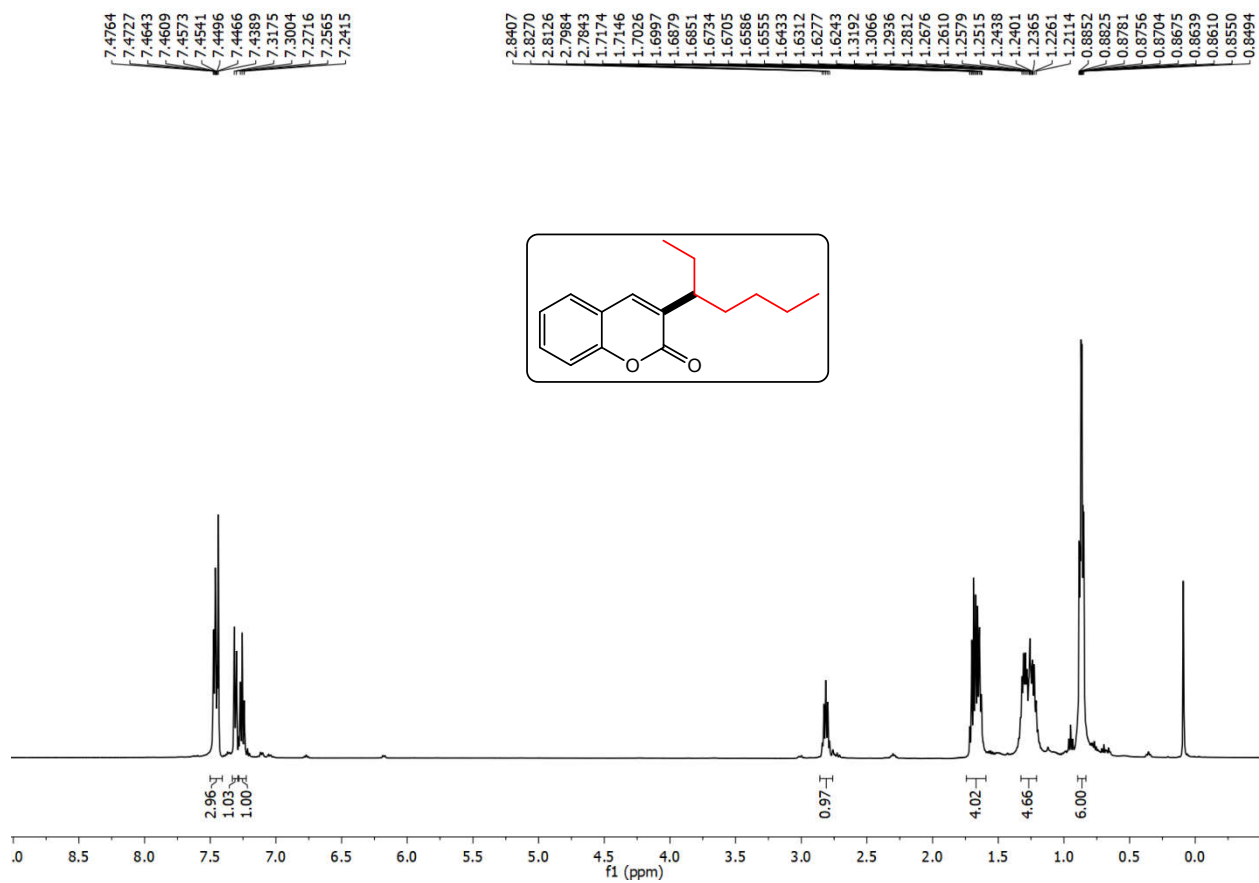
Compound 3a



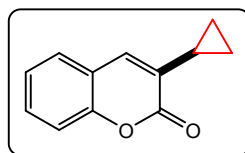
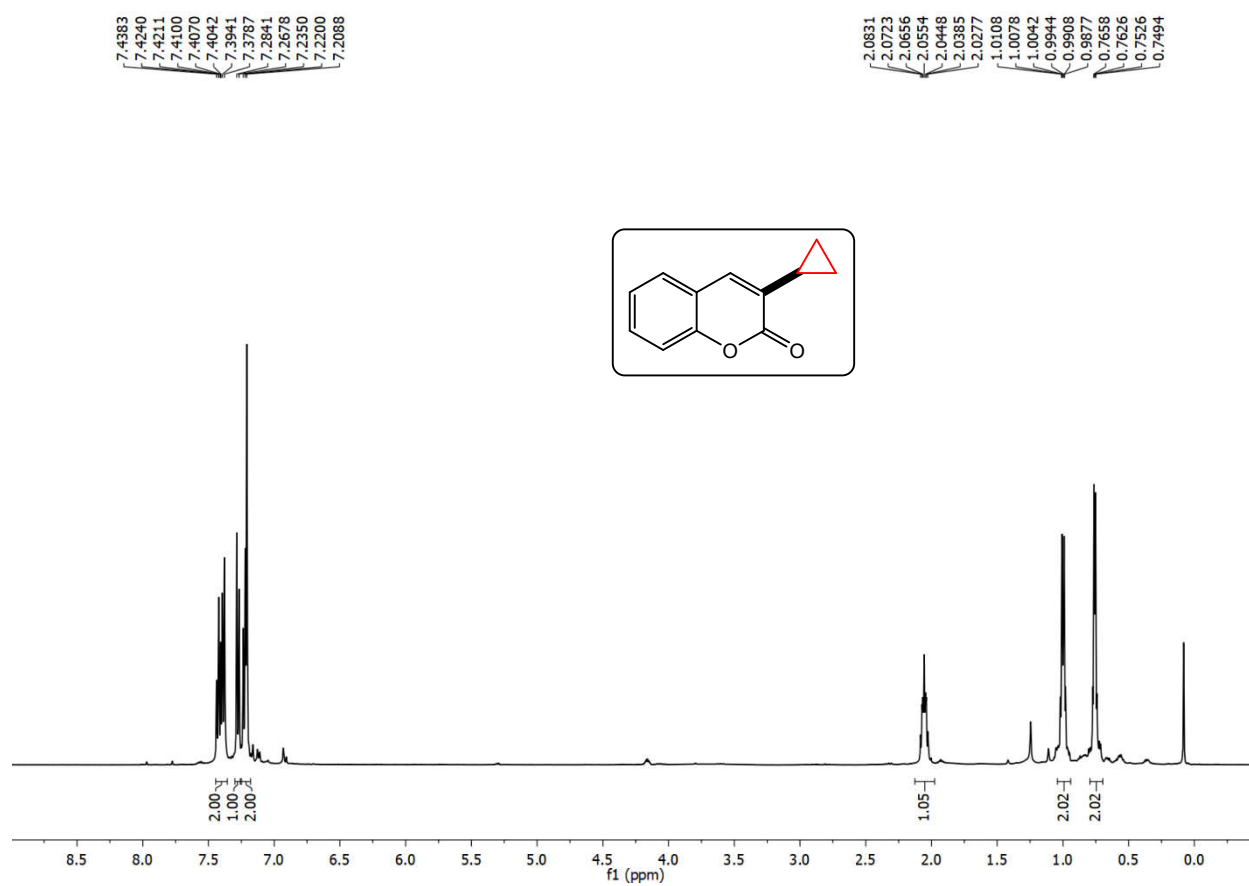
Compound 3b



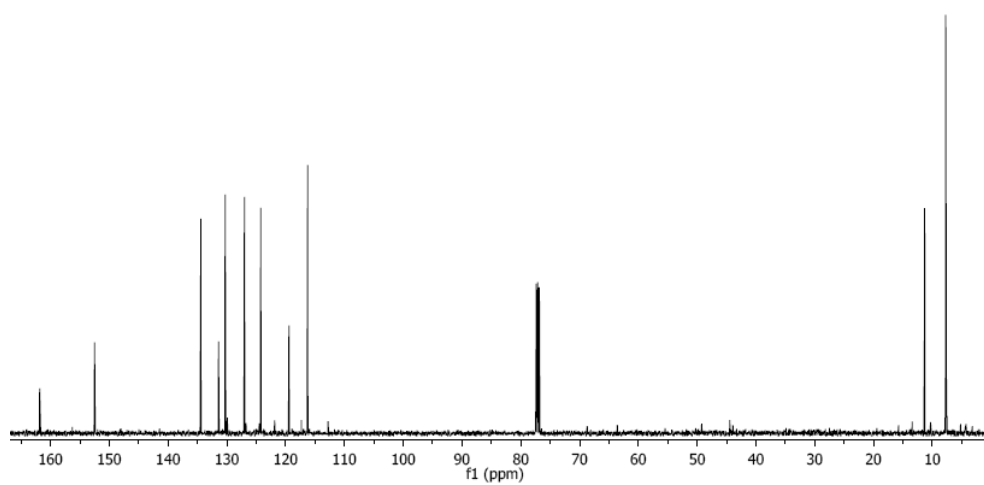
Compound 3c



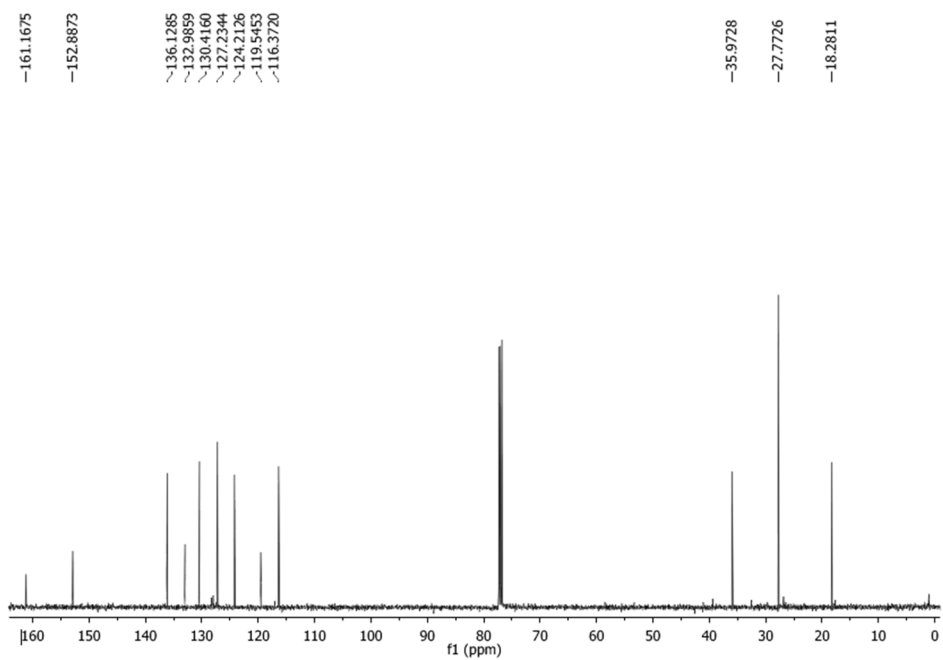
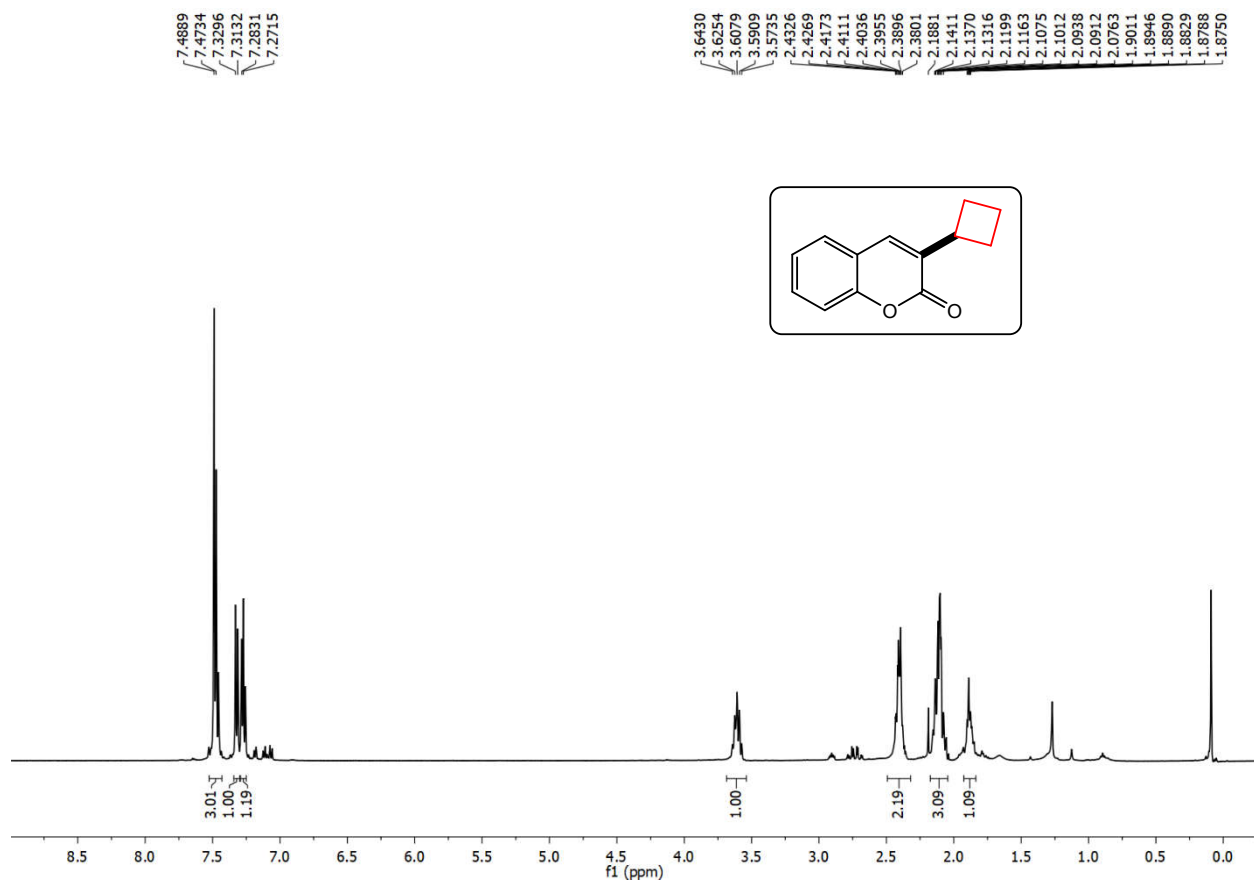
Compound 3d



Chemical shift values (ppm): -161.8403, -152.4989, -134.4099, -131.4193, -130.2827, -127.0111, -124.2413, -119.4349, -116.2534, -11.2968, -7.7121.

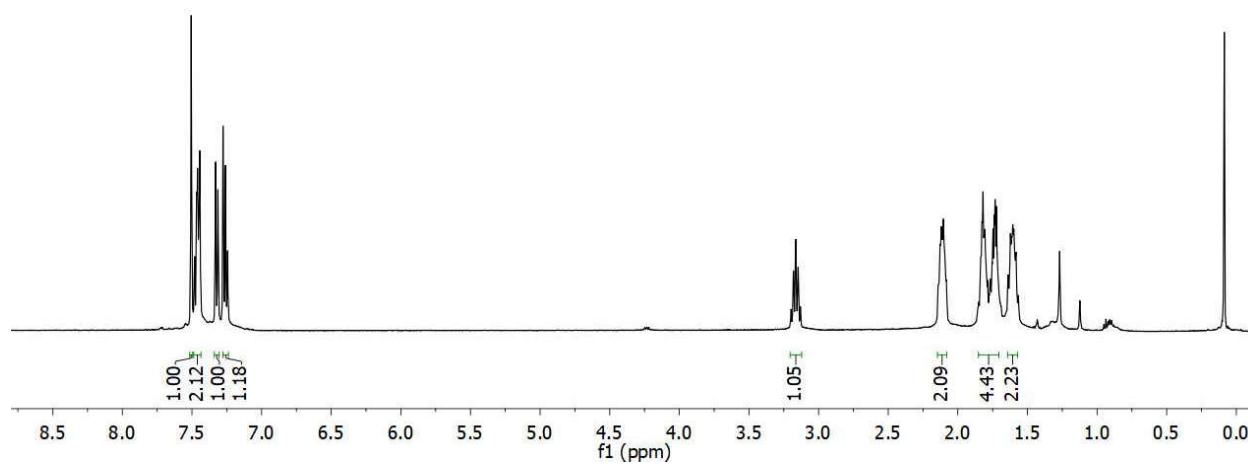
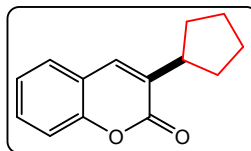


Compound **3e**

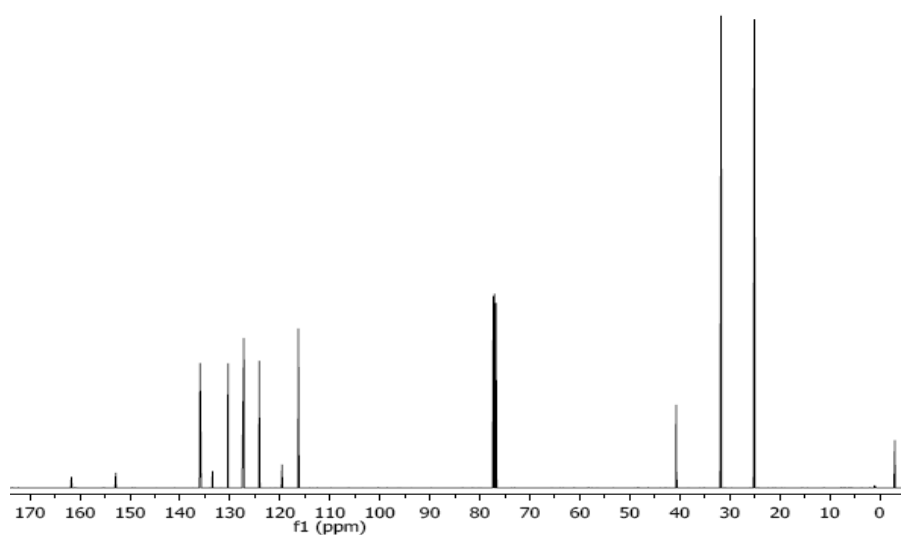


Compound 3f

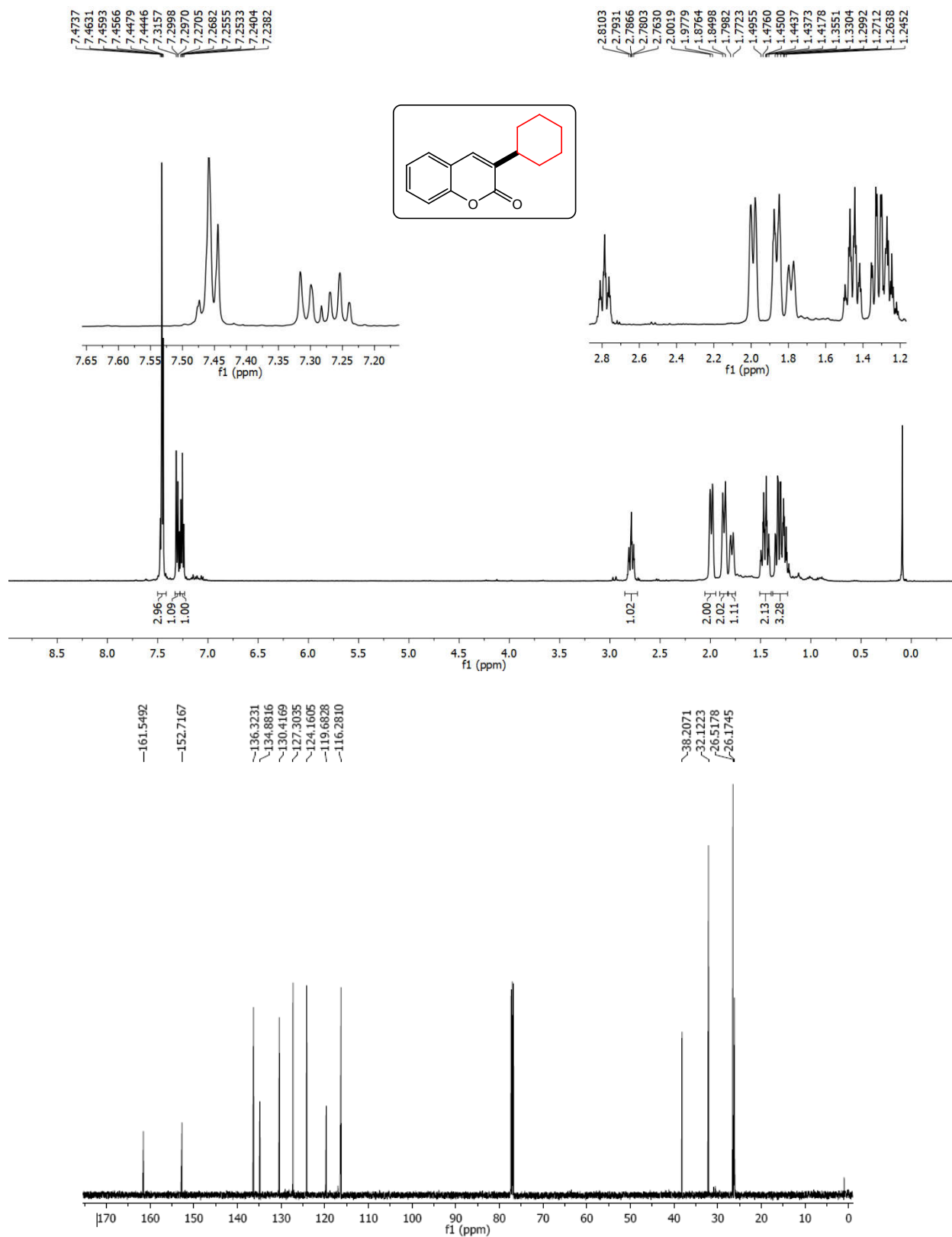
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7.4806
7.4656
7.4590
7.4496
7.4435
7.3300
7.3137
7.2737
7.2594
7.2442
3.1807
3.1639
3.1471
2.1409
2.1292
2.1261
2.1189
2.1151
2.1042
2.0941
2.0869
2.0813
1.8414
1.8357
1.8277
1.8193
1.8110
1.8050
1.7984
1.7932
1.7860
1.7757
1.7695
1.7661
1.7544
1.7475
1.7387
1.7324
1.7228
1.7148
1.7080
1.6389
1.6228
1.6164
1.6117
1.6057
1.5981
1.5933
1.5886
1.5815
1.5737
1.5655



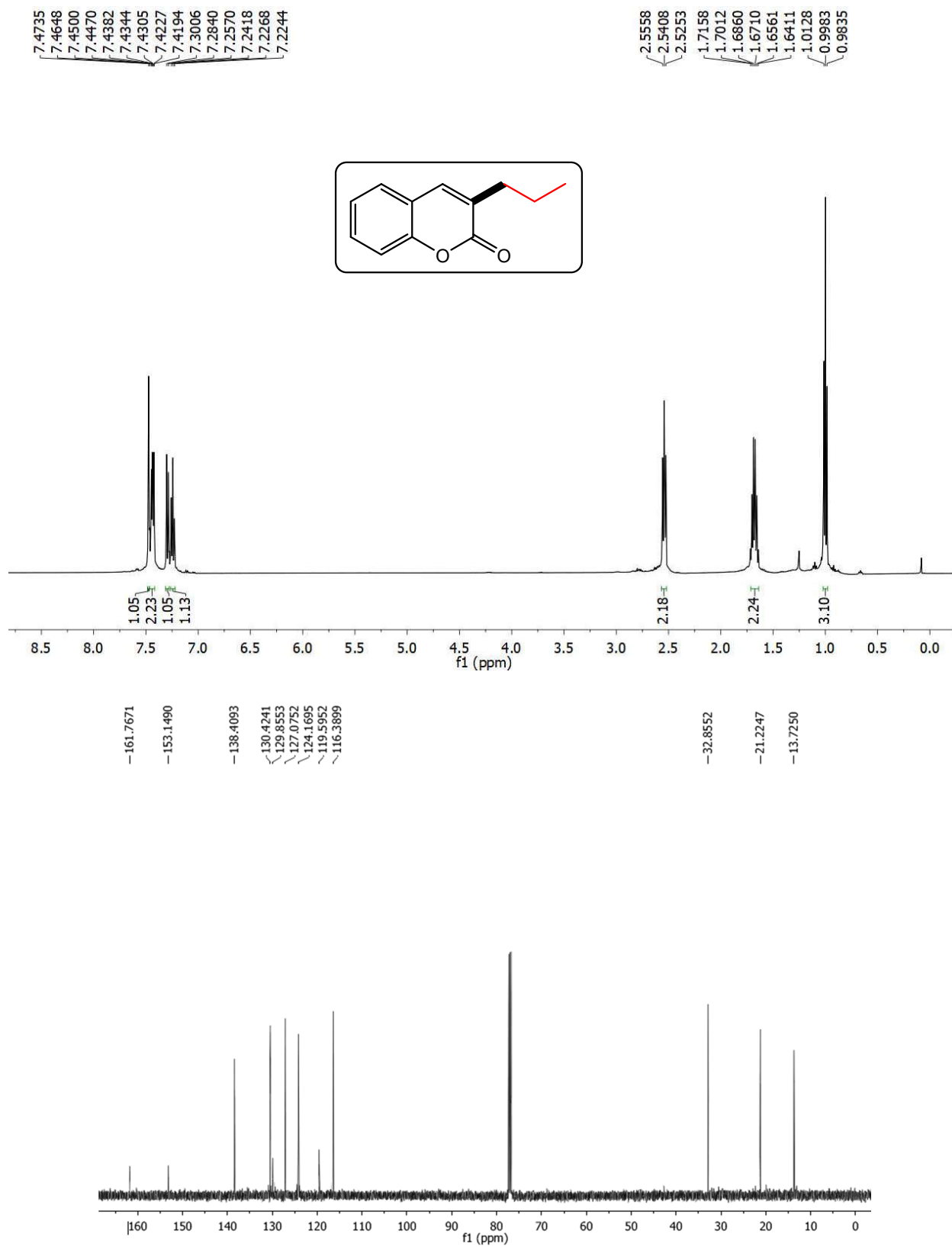
-161.7097
-152.8342
135.9877
133.4707
130.3849
127.2164
124.1604
119.5798
116.3074
40.7137
31.7165
25.0693



Compound 3g



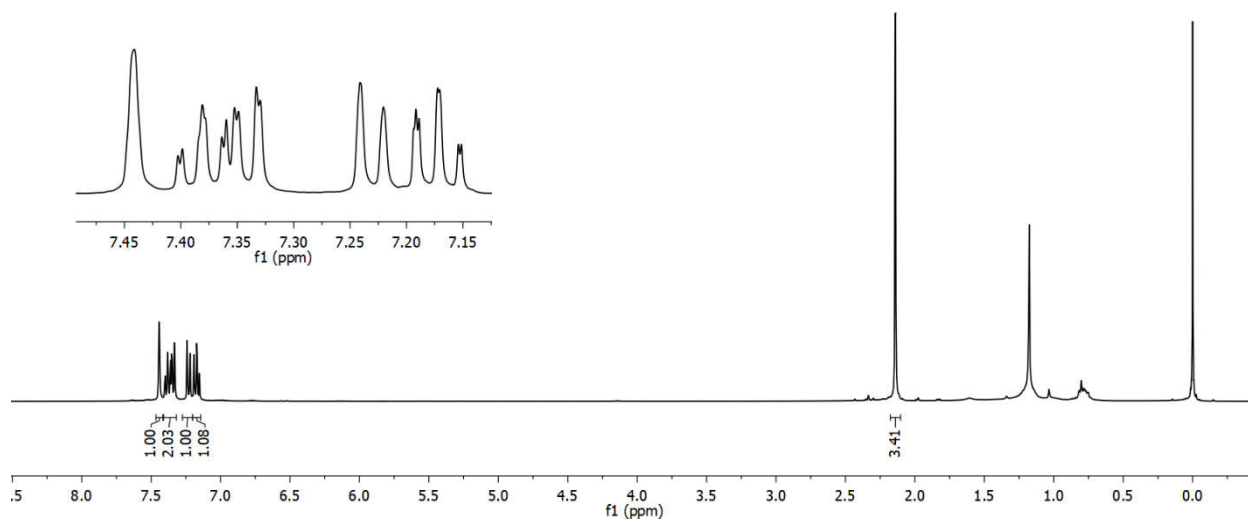
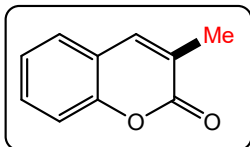
Compound 3h



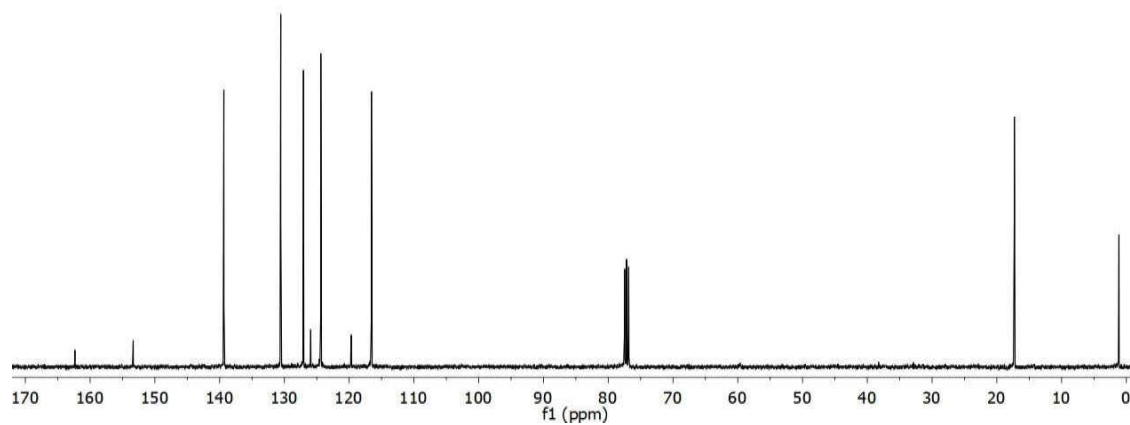
Compound 3i

7.4478
7.4442
7.4403
7.4364
7.4027
7.3986
7.3846
7.3812
7.3778
7.3638
7.3597
7.3528
7.3488
7.3332
7.3293
7.2408
7.2212
7.2192
7.1940
7.1918
7.1888
7.1730
7.1701
7.1542
7.1513

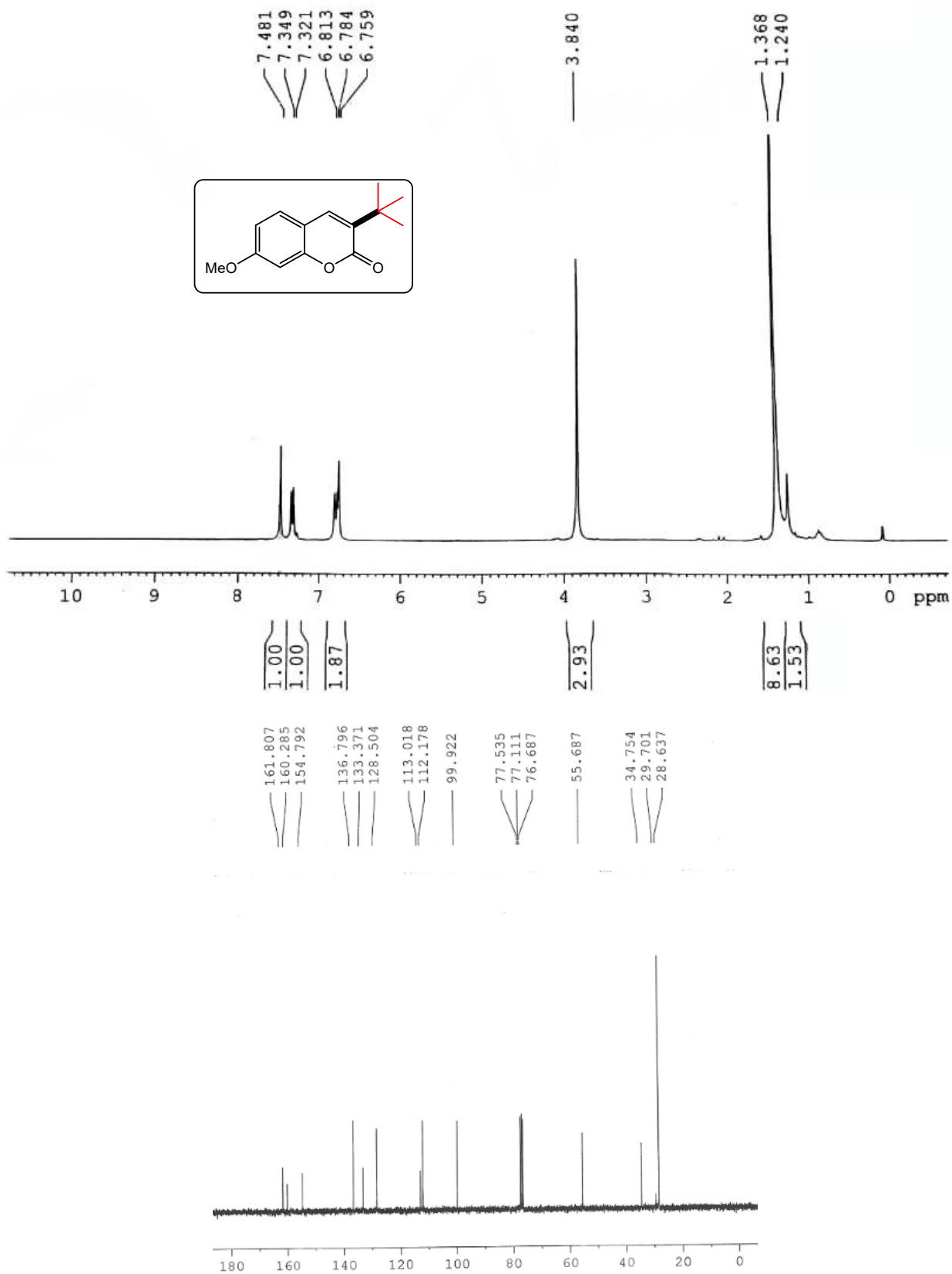
2.1428
2.1393



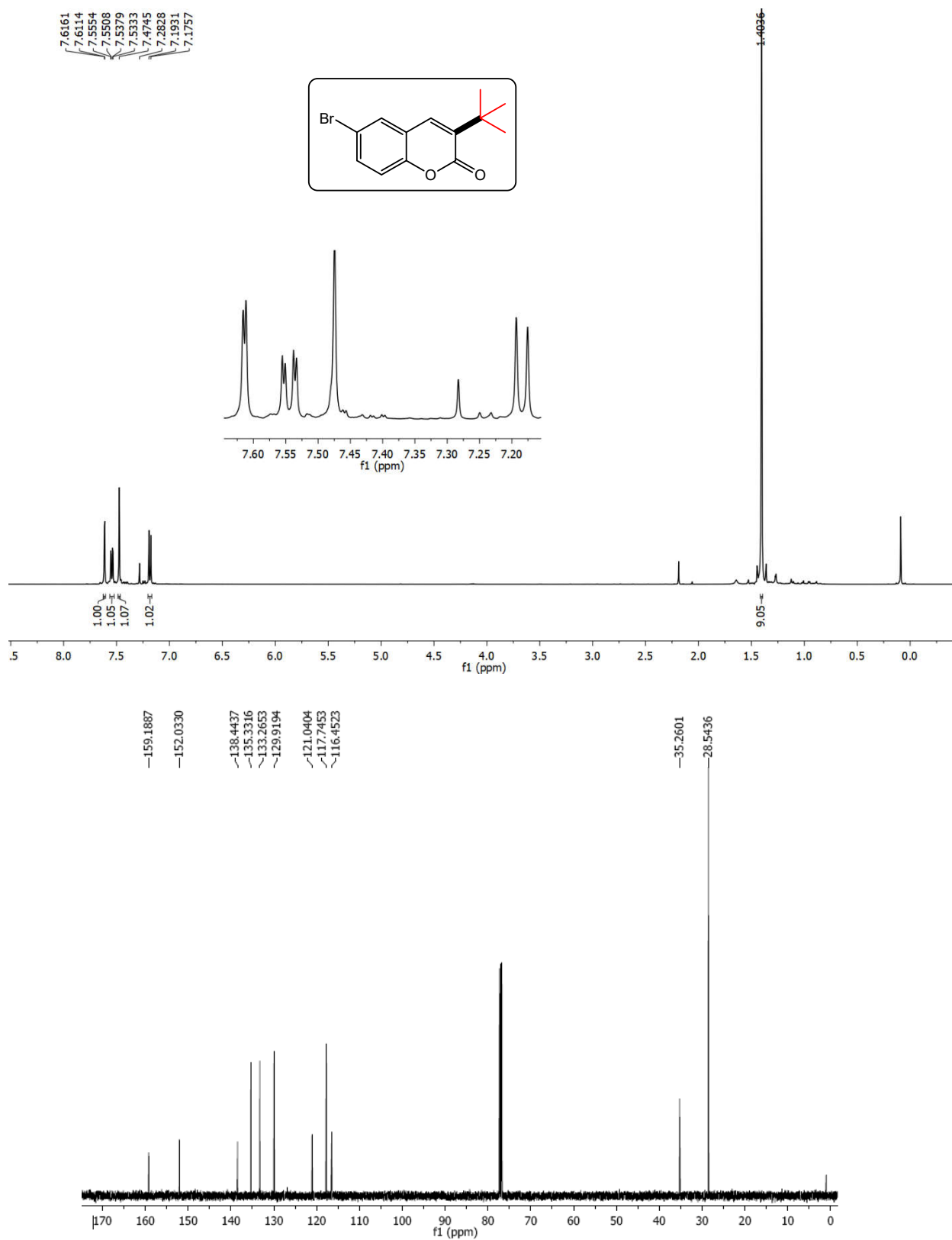
-162.3429
-153.3428
-139.3488
-130.5633
-127.0554
-125.9368
-124.3769
-119.6941
-116.5615
-17.2954



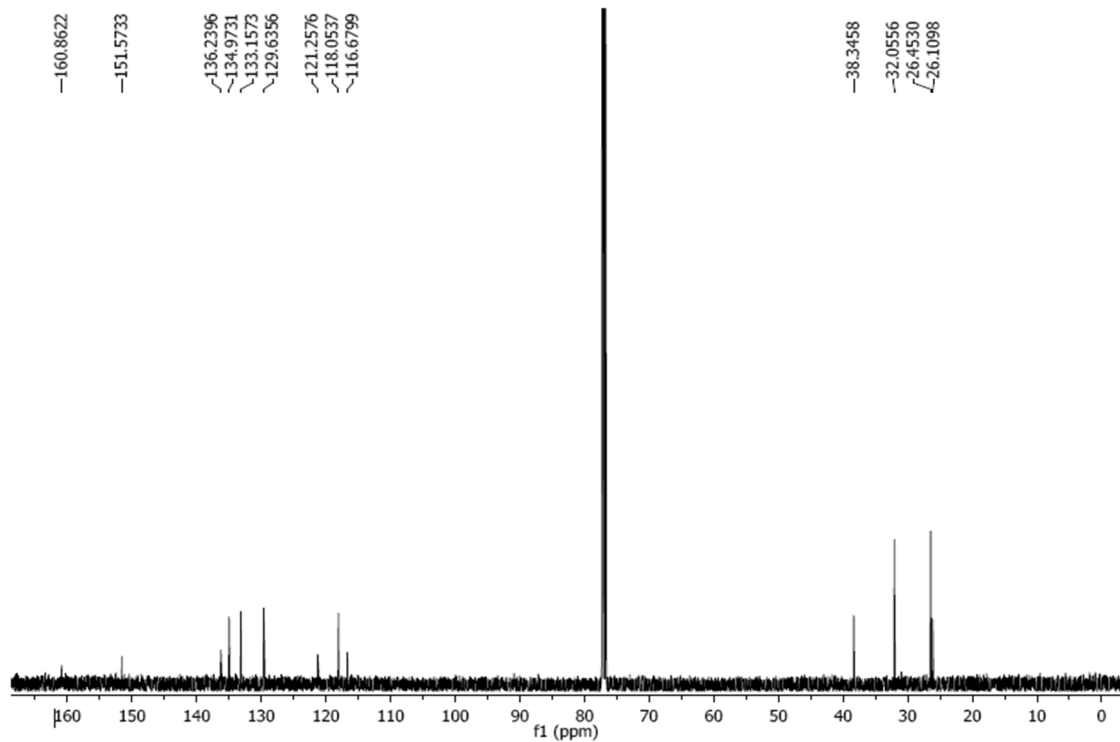
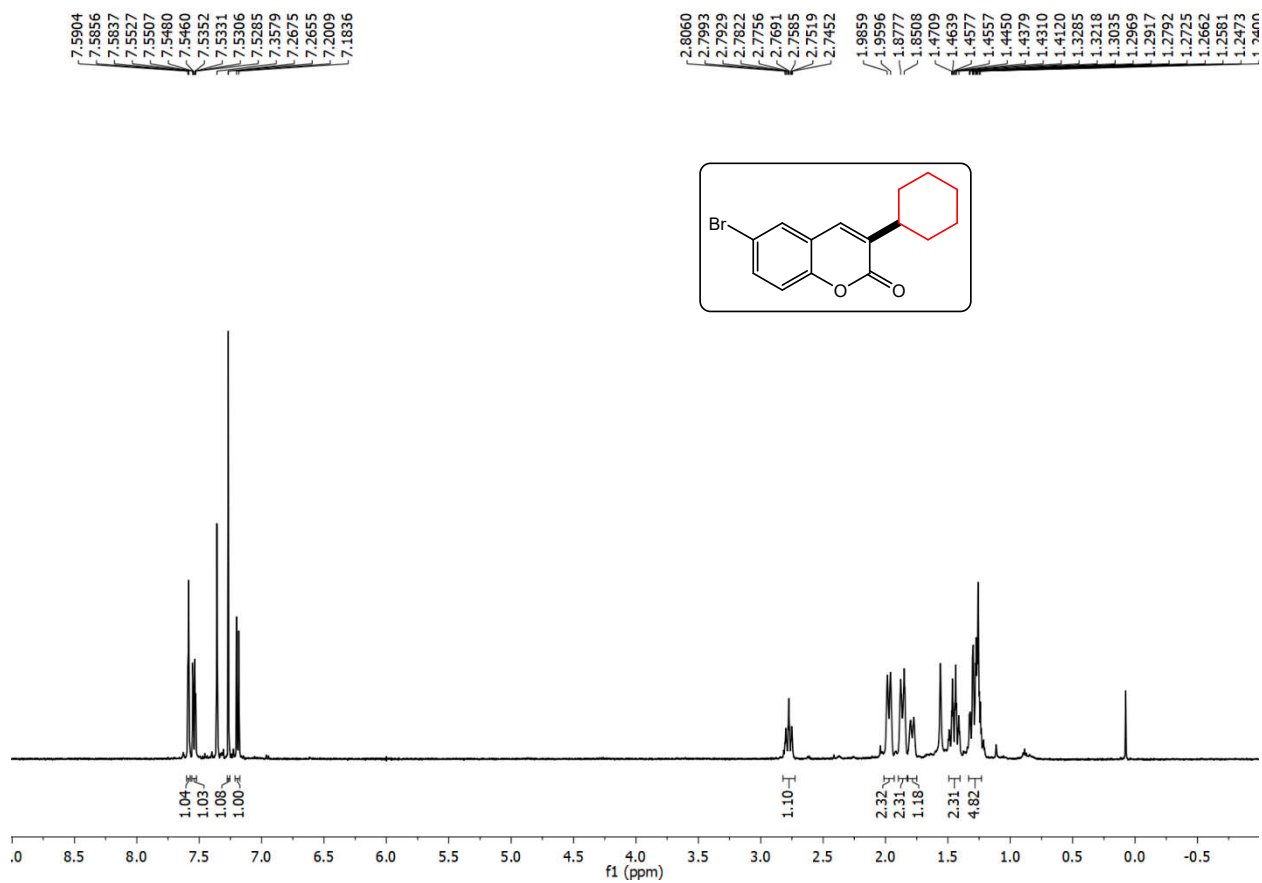
Compound **3k**



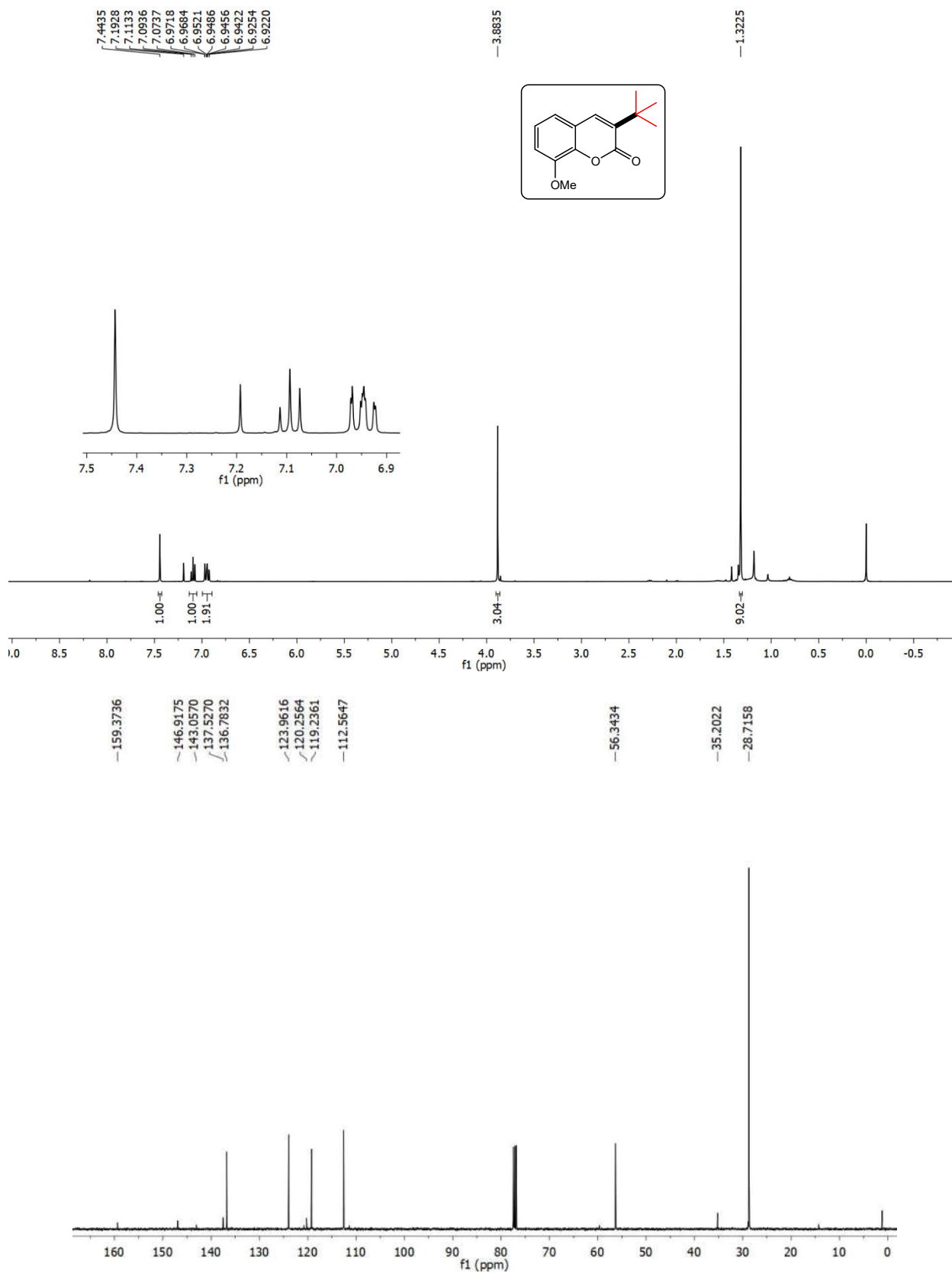
Compound 31



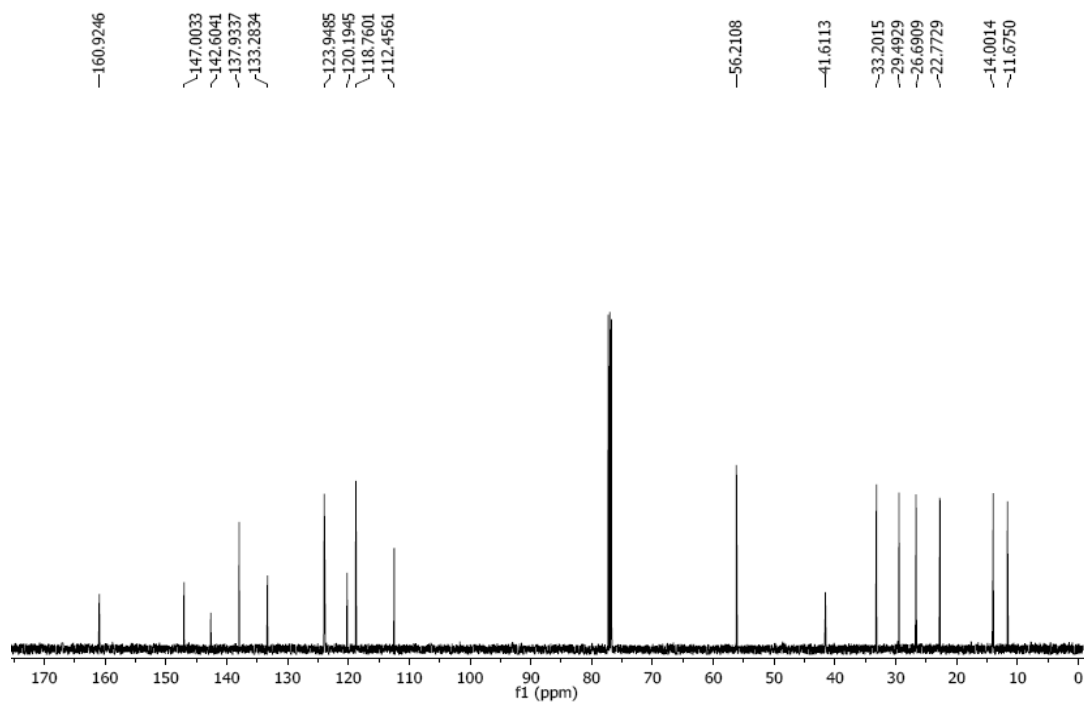
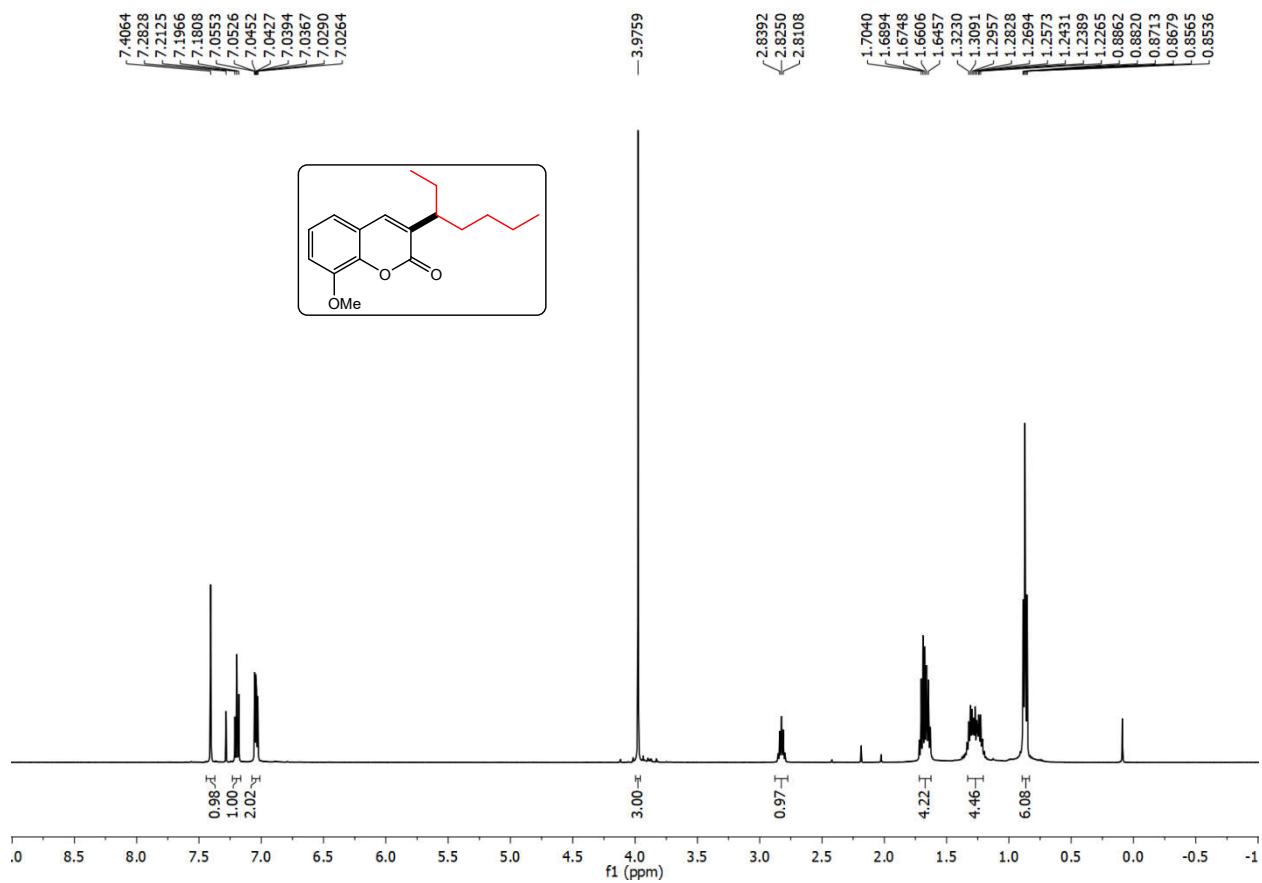
Compound 3m



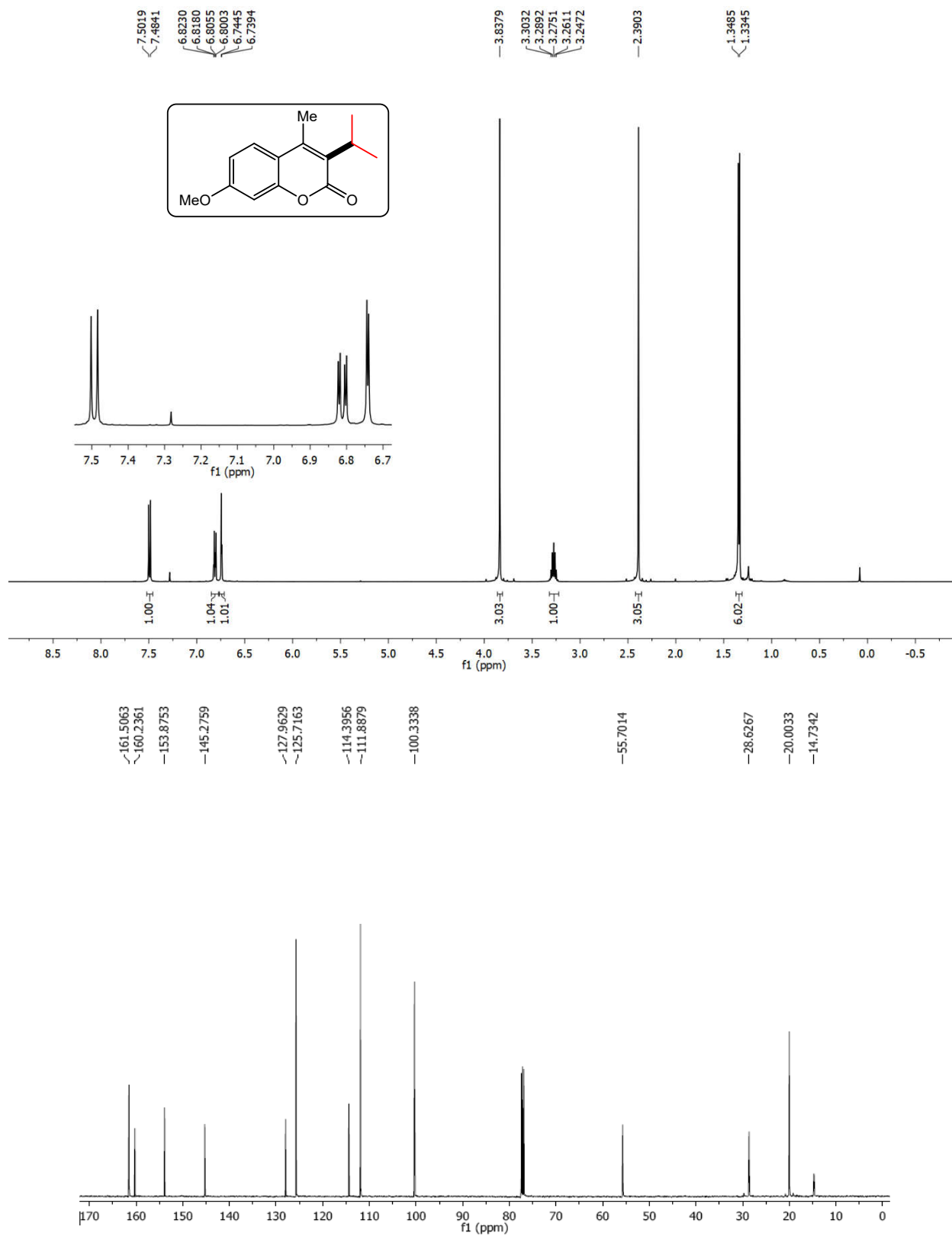
Compound 3n



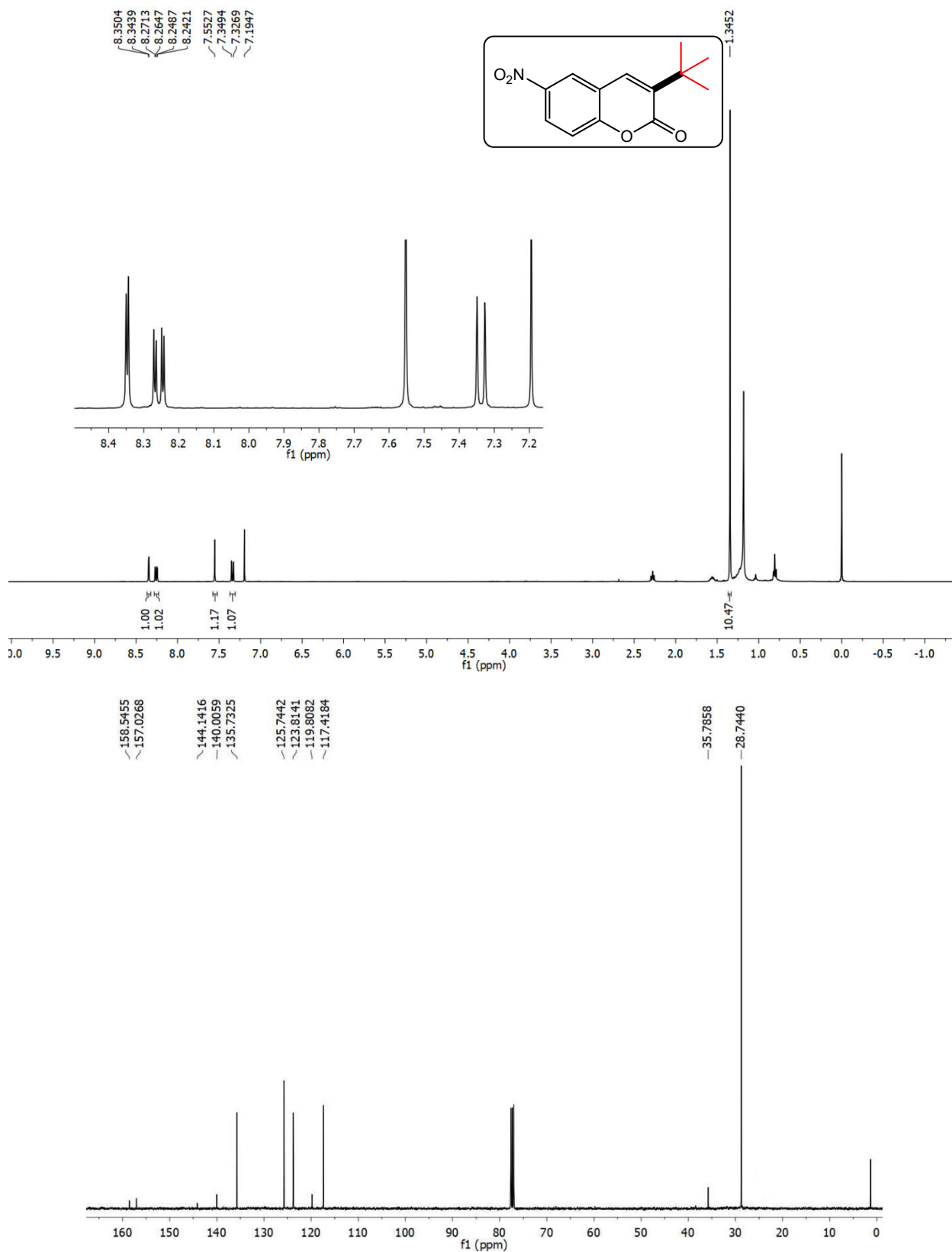
Compound 30



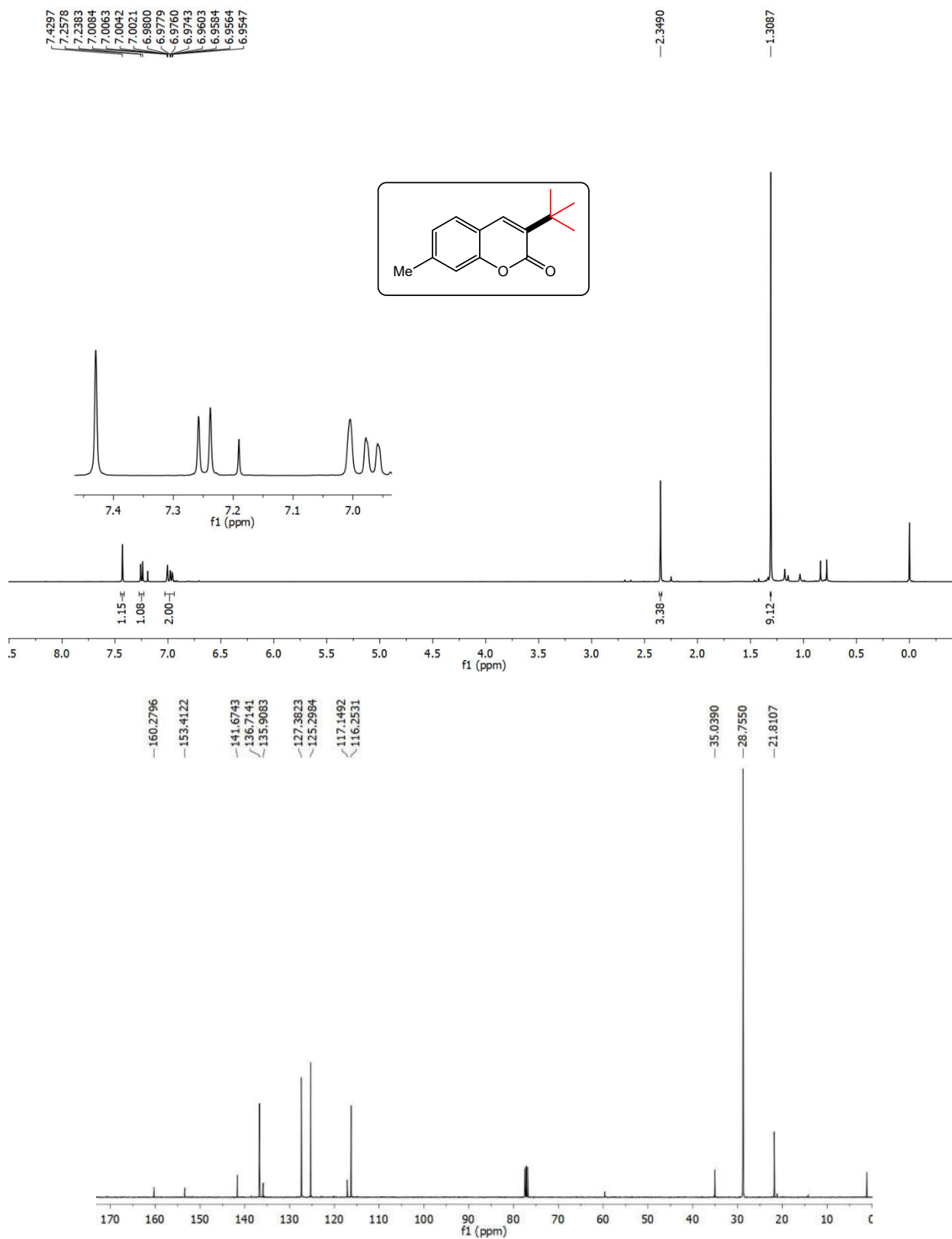
Compound **3p**



Compound 3q

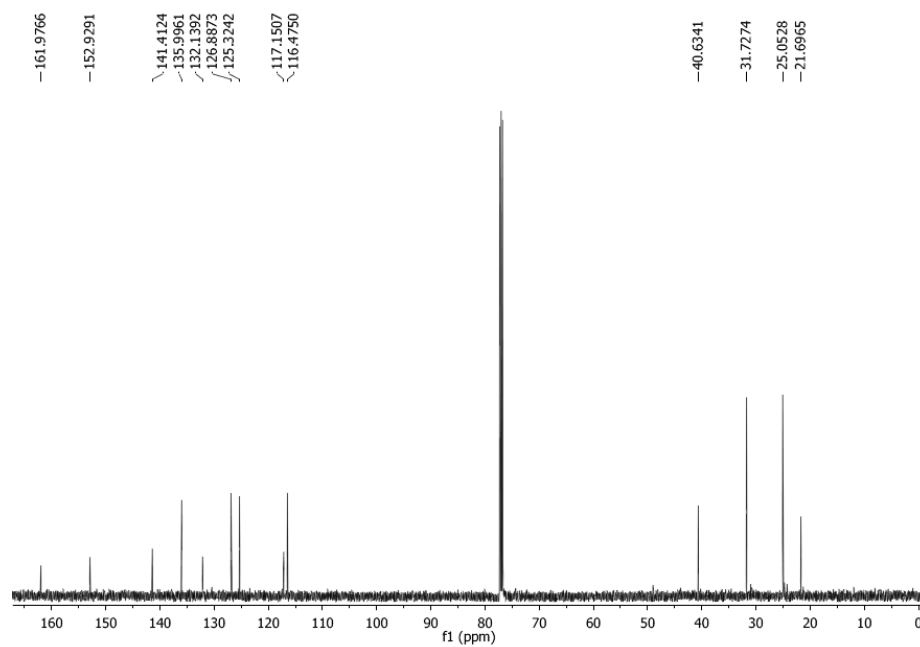
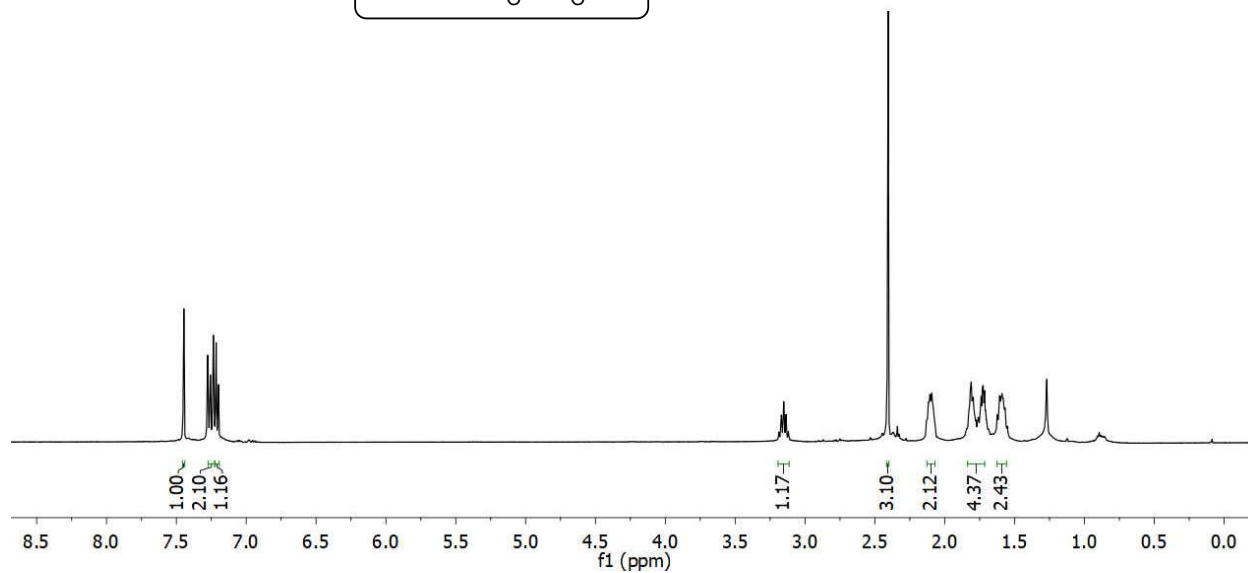
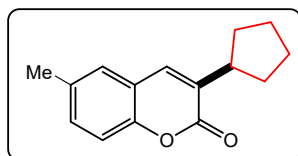


Compound 3r

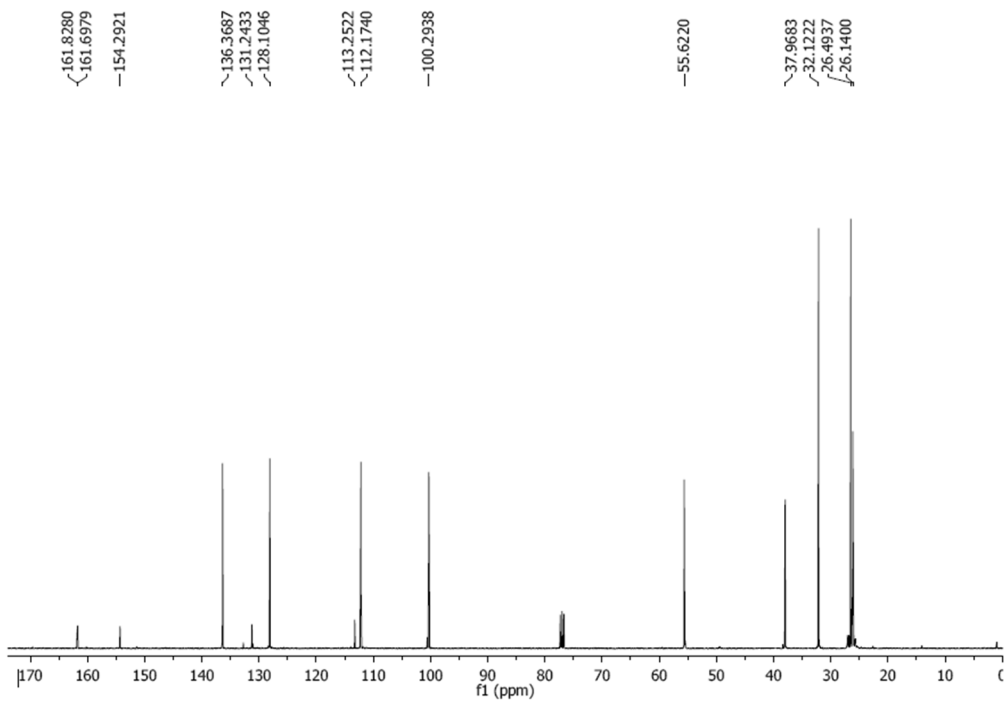
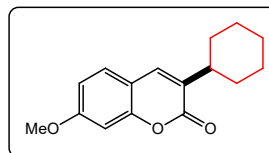
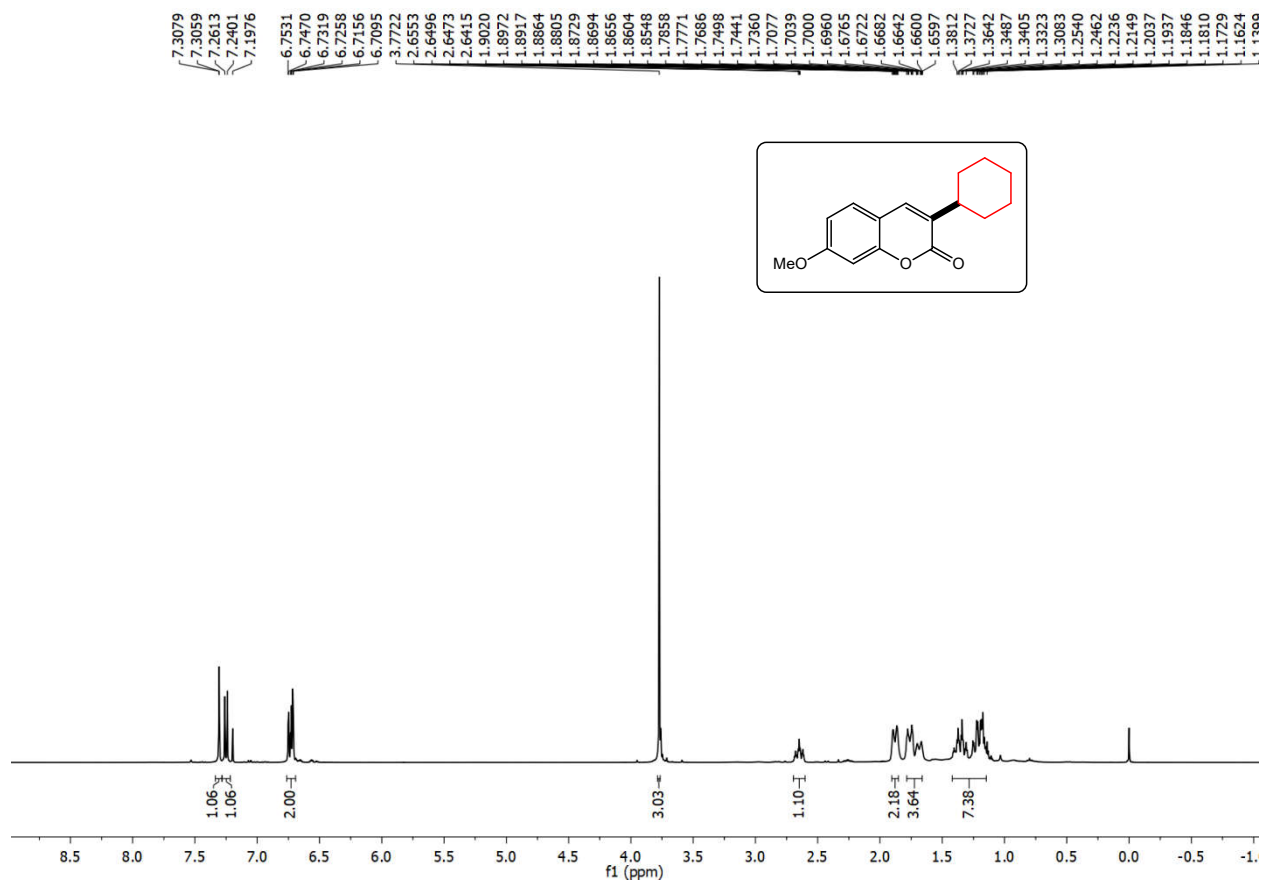


Compound 3s

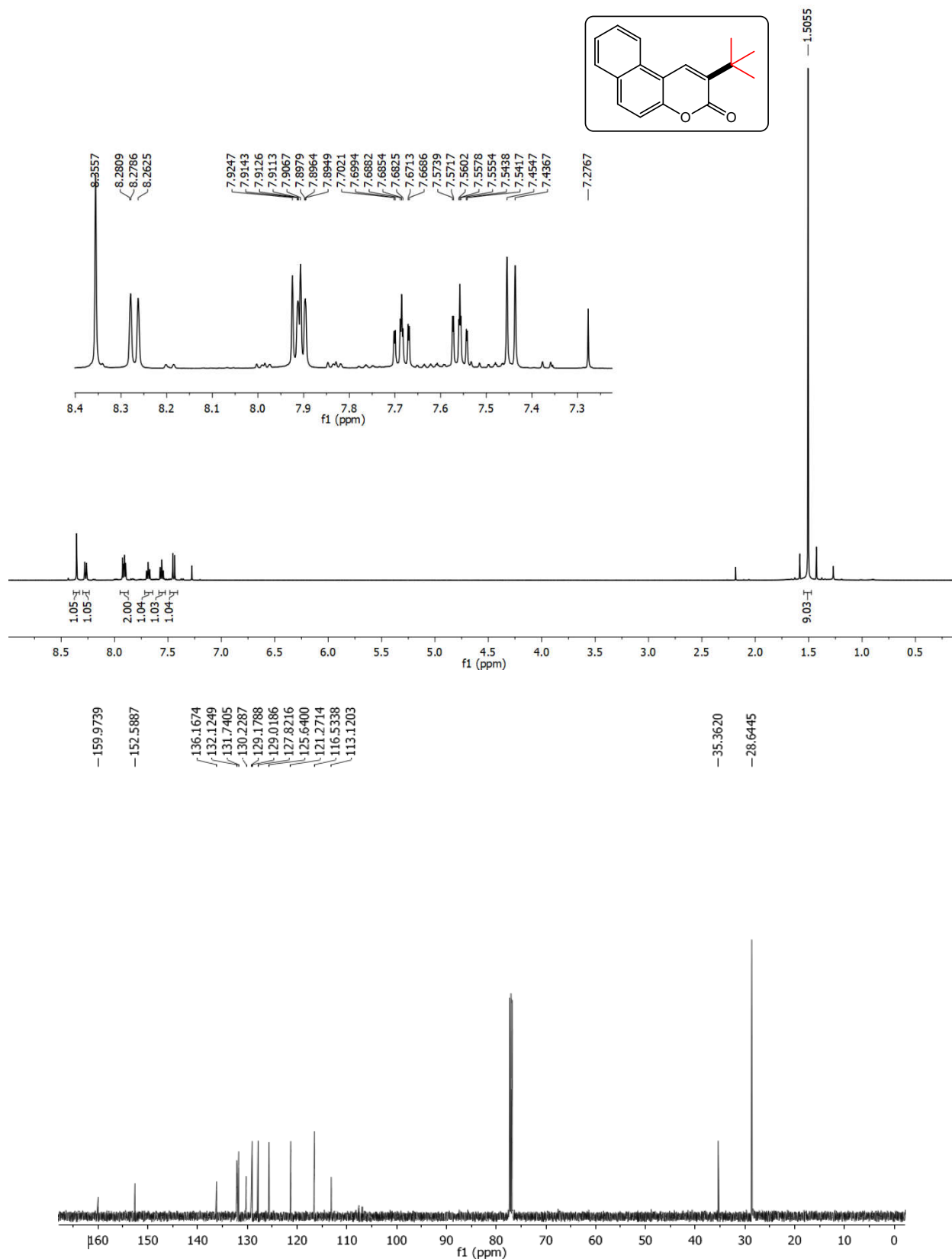
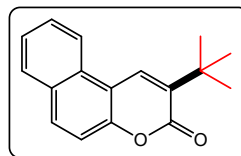
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7.2583
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7.2353
7.2155
7.1989
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3.1695
3.1526
3.1358
3.1189
2.4047
2.1296
2.1150
2.1059
2.1020
2.0946
2.0901
2.0819
2.0741
2.0693
1.8309
1.8257
1.8177
1.8112
1.8081
1.8011
1.7951
1.7865
1.7818
1.7760
1.7643
1.7599
1.7568
1.7495
1.7460
1.7393
1.7303
1.7242
1.7147
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1.6236
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1.5901
1.5826
1.5771
1.5736
1.5664
1.5515



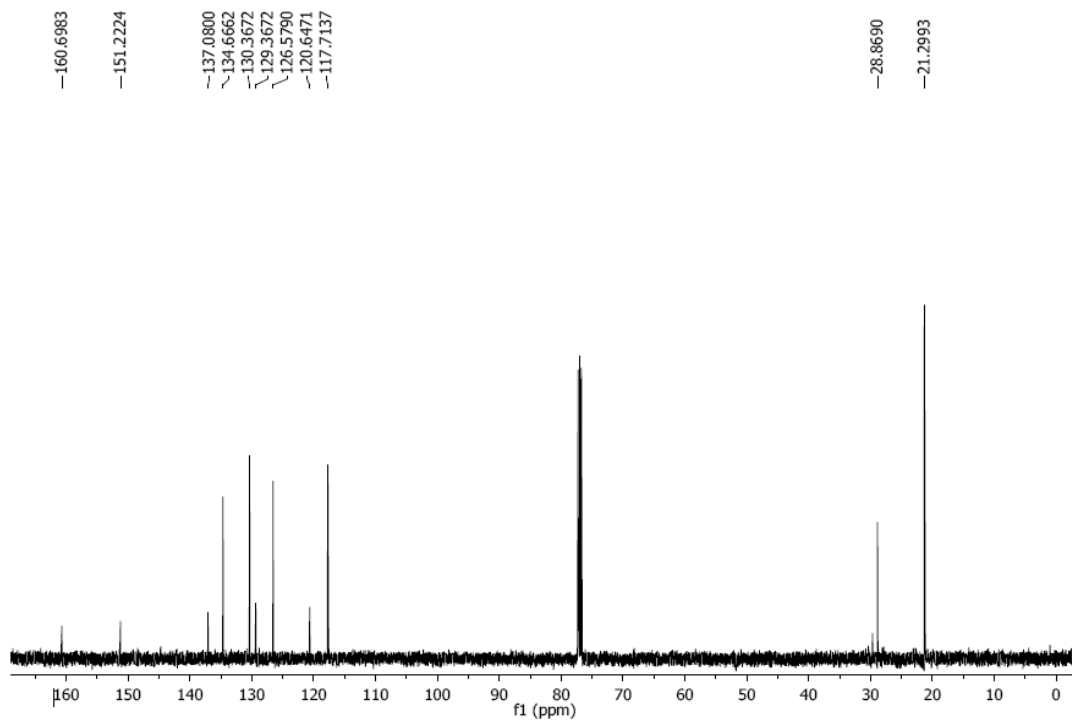
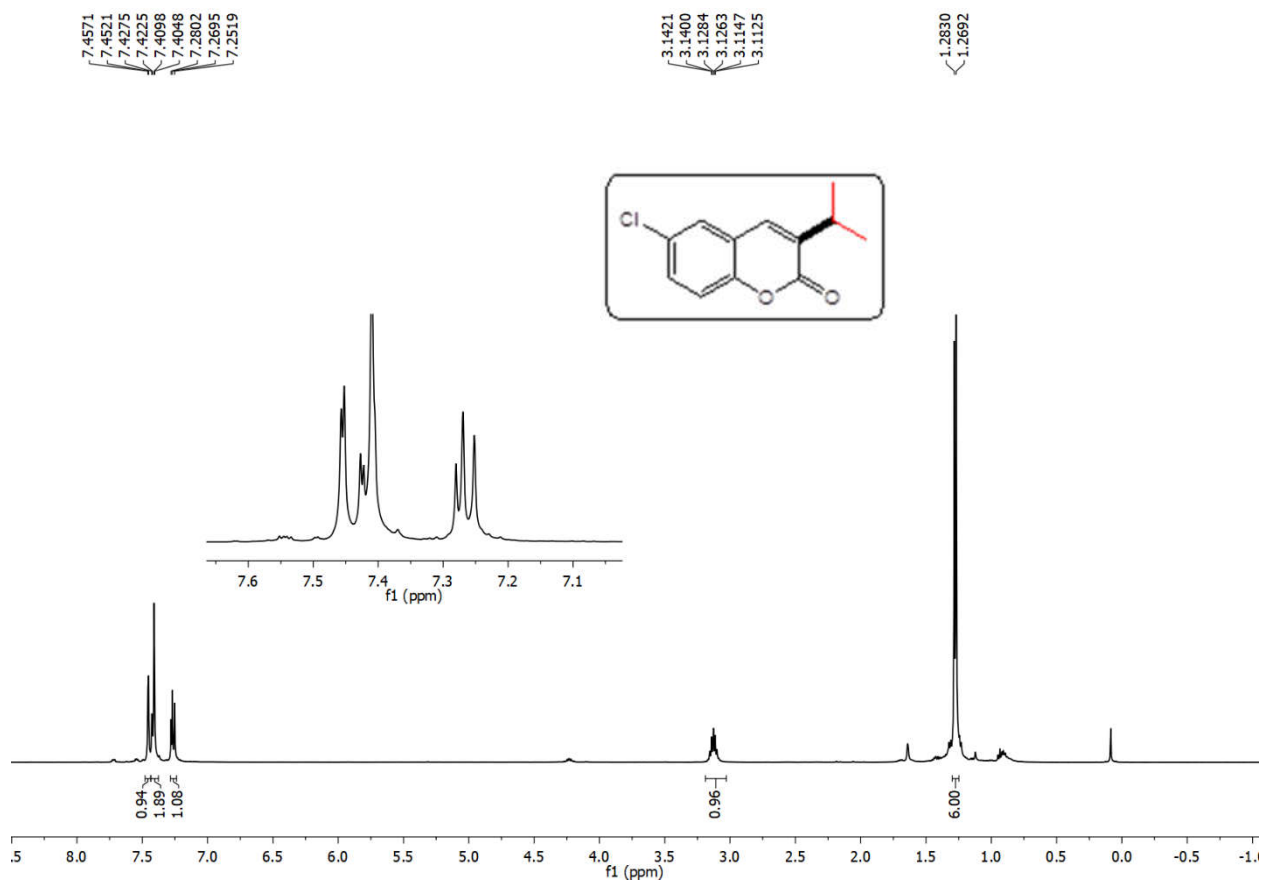
Compound 3t



Compound **3u**

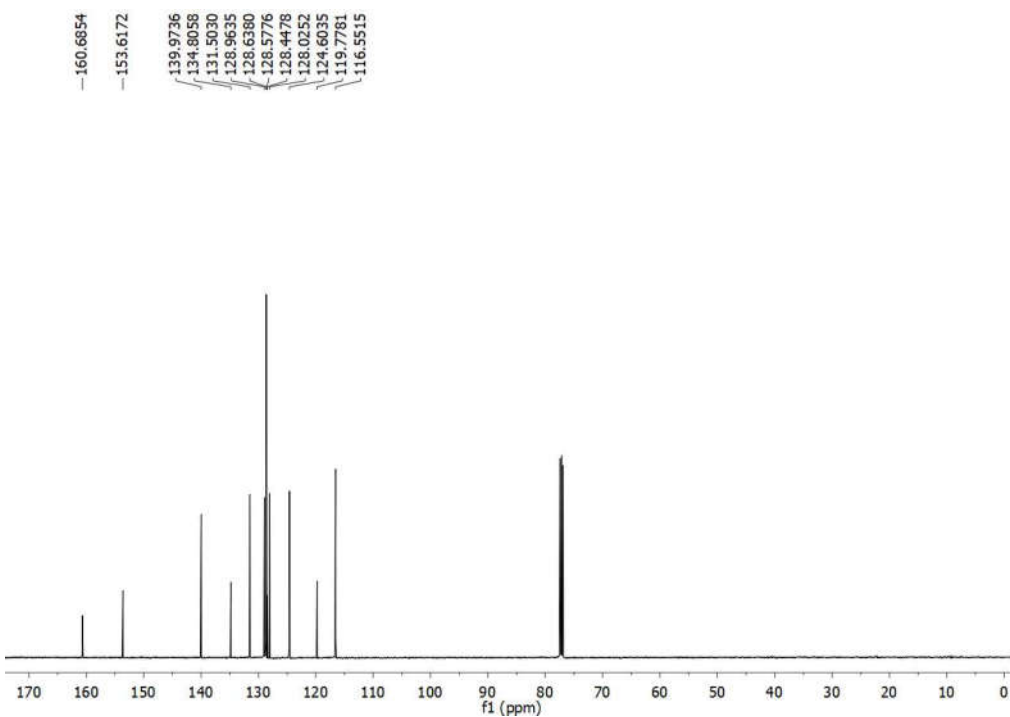
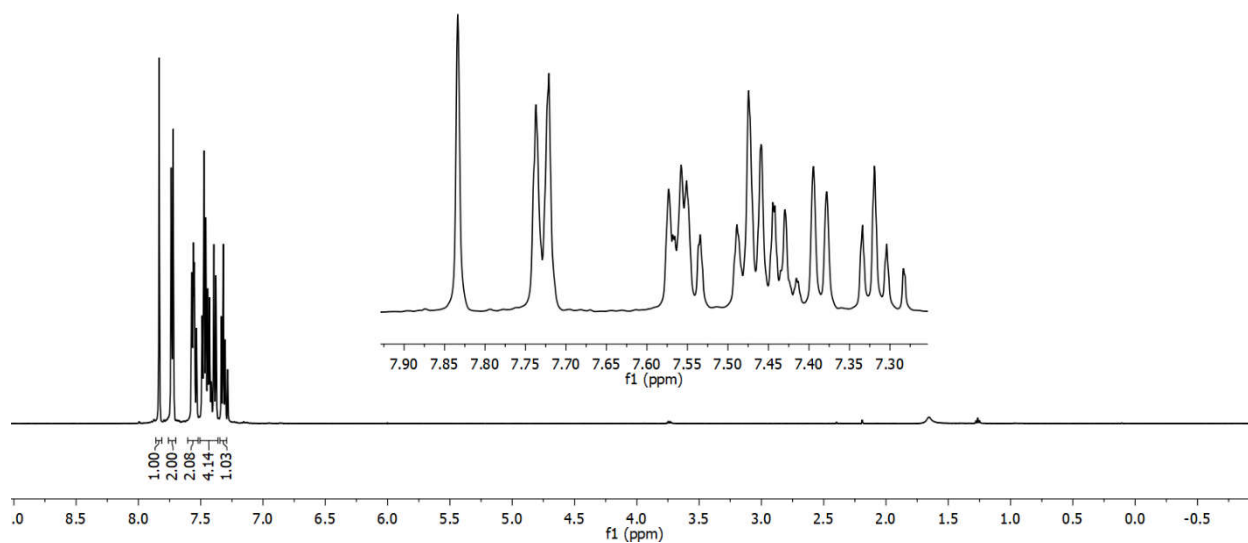
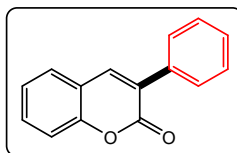


Compound 3v



Compound 4a

7.8336
7.7403
7.7374
7.7350
7.7236
7.7210
7.7185
7.5731
7.5578
7.5512
7.5485
7.5368
7.5341
7.5314
7.4922
7.4892
7.4865
7.4835
7.4748
7.4722
7.4696
7.4631
7.4602
7.4580
7.4476
7.4447
7.4420
7.4392
7.4350
7.4301
7.4281
7.3945
7.3781
7.3363
7.3340
7.3315
7.3212
7.3190
7.3166
7.3062
7.3039
7.2835



Compound 4b

