EXPERIMENTAL PROCEDURES, MS and NMR DATA, and SPECTRA

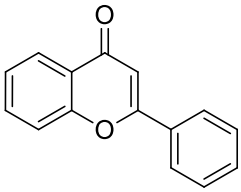
General procedure for the synthesis of the flavone derivatives 2

MS and NMR data

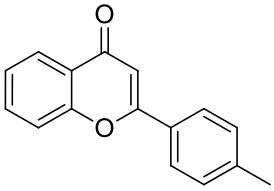
NMR ^{1}H and ^{13}C spectra

General procedure for the synthesis of the flavone derivatives 2:

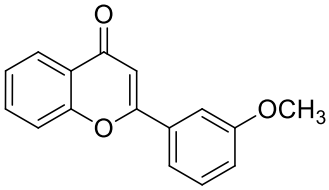
In a reaction tube, under ambient atmosphere (air), was added the NaOH (5 mol%), the appropriated substrate **1** (0,25 mmol) diluted in NMP (1 mL), and the PhSH (1 equiv), at room temperature. Then, the reaction tube was sealed with a rubber septum and the reaction mixture has been stirred out for 30 minutes, at reflux temperature (202 °C). After that, the reaction was quenched with sat. aq. NH_4Cl (5 mL) and the aqueous layer was extracted with EtOAc (3 × 5 mL), and dried ($MgSO_4$). After filtering, the organic solution was concentrated using a rotary evaporator under reduced pressure. The product was purified by flash chromatography in silica gel using hexane/EtOAc as eluent.



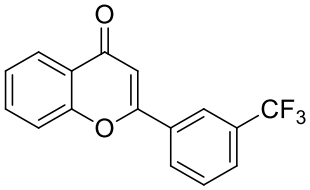
2-phenyl-4*H*-chromen-4-one (2a): Yield: 0.053 g (95%). ^{1}H NMR: $CDCl_3$, 400 MHz, δ (ppm): 8.25-8.22 (m, 1H), 7.90-7.94 (m, 2H), 7.72-7.67 (m, 1H), 7.58-7.46 (m, 4H), 7.44-7.39 (m, 1H), 6.82 (s, 1H). ^{13}C NMR: $CDCl_3$, 100 MHz δ (ppm): 178.4, 163.3, 156.2, 133.7, 131.7, 131.5, 129.0, 126.2, 125.6, 125.2, 123.9, 118.0, 107.5. MS (EI, 70 eV): *m/z* (relative intensity): 64 (21), 82 (16), 92 (62), 97 (16), 120 (79), 165 (16), 194 (52), 221(33), 222 (100).



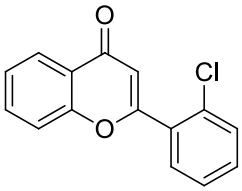
2-(4-tolyl)-4*H*-chromen-4-one (2b): Yield: 0.044 g (75%). ^{1}H NMR: $CDCl_3$, 400 MHz, δ (ppm): 8.10 (dd, *J* = 7.9 Hz, *J* = 1.4 Hz, 1H), 7.79 (d, *J* = 8.3 Hz, 2H), 7.68-7.63 (m, 1H), 7.55-7.49 (m, 1H), 7.40-7.36 (m, 1H), 7.29 (d, *J* = 8.0 Hz, 2H), 6.76 (s, 1H), 2.41 (s, 3H). ^{13}C NMR: $CDCl_3$, 100 MHz δ (ppm): 178.3, 163.6, 156.3, 142.2, 133.5, 129.7, 129.0, 126.2, 125.7, 125.0, 124.0, 117.9, 107.0, 21.4. MS (EI, 70 eV): *m/z* (relative intensity): 89 (14), 92 (29), 115 (39), 116 (23), 120 (50), 208 (33), 221 (32), 236 (100).



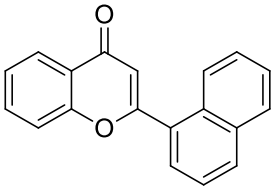
2-(3-methoxyphenyl)-4*H*-chromen-4-one (2c): Yield: 0.055 g (88%). ^{1}H NMR: $CDCl_3$, 400 MHz, δ (ppm): 8.23 (dd, *J* = 7.9 Hz, *J* = 1.7 Hz, 1H), 7.72-7.67 (m, 1H), 7.57-7.55 (m, 1H), 7.52-7.49 (m, 1H), 7.44-7.39 (m, 3H), 7.09-7.06 (m, 1H), 6.81 (s, 1H), 3.89 (s, 3H). ^{13}C NMR: $CDCl_3$, 100 MHz δ (ppm): 178.4, 163.2, 160.0, 156.2, 133.7, 133.1, 130.1, 125.7, 125.2, 124.0, 118.7, 118.1, 117.2, 111.8, 107.8, 55.4. MS (EI, 70 eV): *m/z* (relative intensity): 63 (14), 92 (23), 102 (19), 120 (22), 132 (46), 224 (27), 251 (18), 252 (100).



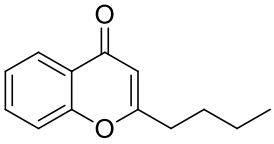
2-(3-(trifluoromethyl)phenyl)-4*H*-chromen-4-one (2d): Yield: 0.040 g (55%). ^{1}H NMR: $CDCl_3$, 400 MHz, δ (ppm): 8.24 (ddd, *J* = 7.9 Hz, *J* = 1.7 Hz, *J* = 0.5 Hz, 1H), 8.20-8.19 (m, 1H), 8.11-8.08 (m, 1H), 7.82-7.79 (m, 1H), 7.76-7.71 (m, 1H), 7.70-7.65 (m, 1H), 7.61 (ddd, *J* = 8.5 Hz, *J* = 1.0 Hz, *J* = 0.5 Hz, 1H), 7.47-7.43 (m, 1H), 6.86 (s, 1H). ^{13}C NMR: $CDCl_3$, 100 MHz δ (ppm): 178.1, 161.6, 156.2, 134.1, 132.8, 131.8 (q, *J* = 32.8 Hz), 129.7, 129.4 (q, *J* = 6.5 Hz), 128.0 (q, *J* = 3.6 Hz), 125.9, 125.8 (q, *J* = 272.1 Hz), 125.5, 123.9, 123.1 (q, *J* = 3.8 Hz), 118.1, 108.4 (q, *J* = 6.8 Hz).



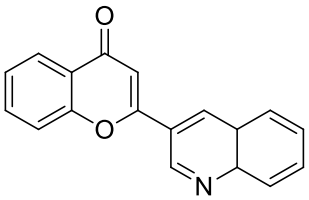
2-(2-chlorophenyl)-4*H*-chromen-4-one (2e): Yield: 0.032 g (50%). ^{1}H NMR: $CDCl_3$, 400 MHz, δ (ppm): 8.27-8.24 (m, 1H), 7,72-7,67 (m, 1H), 7.65-7.62 (m, 1H), 7.55-7.50 (m, 2H), 7.48-7.39 (m, 3H), 6.65 (s, 1H) . ^{13}C NMR: $CDCl_3$, 100 MHz δ (ppm): 178.0, 162.6, 156.6, 133.8, 132.9, 132.0, 131.7, 130.8, 130.6, 127.1, 125.8, 125.3, 123.9, 118.2, 113.0. MS (EI, 70 eV): *m/z* (relative intensity): 63 (11), 82 (17), 92 (57), 120 (100), 165 (12), 221 (56), 228 (44), 230 (15), 256 (88), 258 (27).



2-(naphthalen-1-yl)-4*H*-chromen-4-one (2f): Yield: 0.061g (89%). ^{1}H NMR: $CDCl_3$, 400 MHz, δ (ppm): 8.33-8.30 (m, 1H), 8.15-8.11 (m, 1H), 8.04-7.99 (m, 1H), 7.96-7.91 (m, 1H), 7.76 (dd, *J* = 7.1 Hz, *J* = 1.2 Hz, 1H), 7.73-7.68 (m, 1H), 7.60-7.50 (m, 4H), 7.48-7.43 (m, 1H), 6.68 (s, 1H). ^{13}C NMR: $CDCl_3$, 100 MHz δ (ppm): 178.2, 165.4, 156.7, 133.8, 133.7, 131.5, 130.6, 130.4, 128.7, 127.9, 127.4, 126.5, 125.9, 125.3, 125.0, 124.8, 124.0, 118.2, 113.1. MS (EI, 70 eV): *m/z* (relative intensity): 92 (10), 151 (24), 152 (100), 254 (48), 271 (70), 272 (74), 273 (14).

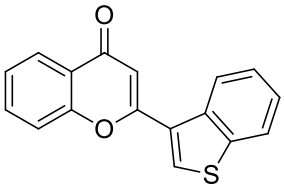


2-butyl-4*H*-chromen-4-one (2g): Yield: 0.032 g (63%). ^{1}H NMR: $CDCl_3$, 400 MHz, δ (ppm): 8.18 (ddd, *J* = 7.9 Hz, *J* = 1.7 Hz, *J* = 0.41 Hz, 1H), 7.65-7.60 (m, 1H), 7.43-7.40 (m, 1H), 7.38-7.34 (m, 1H), 6.17 (s, 1H), 2.62 (t, *J* = 7.5 Hz, 2H), 1,73 (quint, *J* = 7.6 Hz, 2H), 1.43 (sext, *J* = 7.5 Hz, 2H), 0.97 (t, *J* = 7.4 Hz, 3H). ^{13}C NMR: $CDCl_3$, 100 MHz δ (ppm): 178.3, 169.7, 156.5, 133.3, 125.6, 124.8, 123.7, 117.8, 109.7, 33.9, 28.8, 22.0, 13.6. MS (EI, 70 eV): *m/z* (relative intensity): 92 (14), 118 (11), 120 (33), 121 (27), 131 (12), 160 (100), 161 (11), 202 (21).

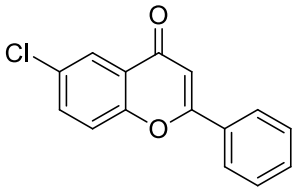


6-chloro-2-(quinolin-3-yl)-4*H*-chromen-4-one (2h): Yield: 0.030 g (44%). ^{1}H NMR: $CDCl_3$, 400 MHz, δ(ppm): 9.4 (brs, 1H), 8.74-8.73 (m, 1H), 8.27 (dd, *J* = 7.8 Hz, *J* = 1.6 Hz, 1H), 8.19 (d, *J* = 8.3 Hz, 1H), 7.99 (dd, *J* = 8.3 Hz, *J* = 1.1 Hz, 1H), 7.87-7.82 (m, 1H), 7.78-7.73 (m, 1H), 7.69-7.64 (m, 2H), 7.49-7.45 (m, 1H), 7.00 (s, 1H). ^{13}C NMR: $CDCl_3$, 100 MHz δ(ppm): 178.0, 161.2, 159.1, 156.4, 149.2, 147.2, 134.3, 134.1, 131.5, 129.6, 128.8, 127.9, 126.6, 125.9,

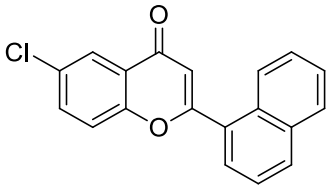
125.6, 124.1, 118.1, 108.6. MS (EI, 70 eV): *m/z* (relative intensity): 64 (31), 73 (100), 93 (35), 120 (34), 123 (30), 157 (39), 245 (27), 273 (25).



2-(benzo[*b*]thiophen-3-yl)-6-chloro-4*H*-chromen-4-one (2i): Yield: 0.027 g (34%). ^{1}H NMR: $CDCl_3$, 400 MHz, δ(ppm): 8.29-8.26 (m, 2H), 8.12 (s, 1H), 7.94 (d, *J* = 8.0 Hz, 1H), 7.74-7.71 (m, 1H), 7.59 (d, *J* = 8.3 Hz, 1H), 7.55-7.51 (m, 1H), 7.49-7.44 (m, 2H), 6.84 (s, 1H). ^{13}C NMR: $CDCl_3$, 100 MHz δ(ppm): 178.3, 160.3, 156.3, 140.7, 135.5, 133.8, 130.4, 128.9, 125.8, 125.5, 125.3, 124.1, 123.3, 123.1, 118.0, 109.4. MS (EI, 70 eV): *m/z* (relative intensity): 63 (8), 92 (9), 114 (21), 125 (8), 158 (99), 261 (12), 277 (29), 278 (100).

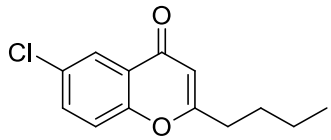


6-chloro-2-phenyl-4*H*-chromen-4-one (2j): Yield: 0.037 g (57%). ^{1}H NMR: $CDCl_3$, 400 MHz, δ (ppm): 8.16 (d, *J* = 2.5 Hz, 1H), 7.91-7.87 (m, 2H), 7.61 (dd, *J* = 8.9 Hz, *J* = 2.6 Hz, 1H), 7.57-7.48 (m, 4H), 6.79 (s, 1H). ^{13}C NMR: $CDCl_3$, 100 MHz δ (ppm): 177.0, 163.3, 154.5, 133.9, 131.8, 131.4, 131.1, 129.0, 126.2, 125.1, 124.9, 119.7, 107.4. MS (EI, 70 eV): *m/z* (relative intensity): 63 (22), 82 (15), 102 (20), 154 (76), 156 (25), 228 (27), 255 (33), 256 (100), 258 (34).

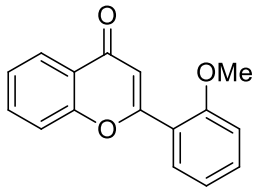


6-chloro-2-(naphthalen-1-yl)-4*H*-chromen-4-one (2k): Yield: 0.064 g (93%). ^{1}H NMR: $CDCl_3$, 400 MHz, δ (ppm): 8.26 (dd, *J* = 2.6 Hz, *J* = 0.3 Hz, 1H), 8.11-8.06 (m, 1H), 8.04-8.00 (m, 1H), 7.97-7.91 (m, 1H), 7.75 (dd, *J* = 7.2 Hz, *J* = 1.3 Hz, 1H), 7.63 (dd, *J* = 8.9 Hz, *J* = 2.6 Hz, 1H), 7.60-7.54 (m, 3H), 7.47 (dd, *J* = 8.9 Hz, *J* = 0.3 Hz, 1H), 6.68 (s, 1H). ^{13}C NMR: $CDCl_3$, 100 MHz δ (ppm): 176.9, 165.6, 155.0, 134.0, 133.7, 131.7, 131.3, 130.3, 130.2, 128.7, 127.9, 127.5,

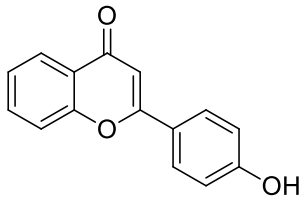
126.6, 125.3, 125.0, 124.9, 124.6, 119.9, 112.9. MS (EI, 70 eV): *m/z* (relative intensity): 106 (7), 151 (18), 152 (100), 289 (23), 305 (29), 306 (36), 307 (17), 308 (13).



2-butyl-6-chloro-4*H*-chromen-4-one (2l): Yield: 0.024 g (41%). NMR ^{1}H: $CDCl_3$, 400 MHz, δ (ppm): 8.13 (dd, *J* = 2.6 Hz, *J* = 0.3 Hz, 1H), 7.56 (dd, *J* = 8.9 Hz, *J* = 2.6 Hz, 1H), 7.36 (dd, *J* = 8.9 Hz, *J* = 0.4 Hz, 1H), 6.16 (s, 1H), 2.62 (t, *J* = 7.5 Hz, 2H), 1.72 (quint, *J* = 7.6 Hz, 2H), 1.43 (sext, *J* = 7.6 Hz, 2H), 0.97 (t, *J* = 7.4 Hz, 3H). ^{13}C NMR: $CDCl_3$, 100 MHz δ (ppm): 176.9, 170.0, 154.8, 133.5, 130.8, 125.1, 124.8, 119.5, 109.7, 33.9, 28.8, 22.0, 13.6. MS (EI, 70 eV): *m/z* (relative intensity): 63 (15), 126 (16), 154 (22), 156 (8), 194 (100), 196 (34), 236 (24), 238 (8).

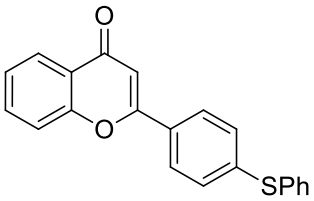


2-(2-methoxyphenyl)-4*H*-chromen-4-one (2m): Yield: 0.025 g (40%). ^{1}H NMR: $CDCl_3$, 400 MHz, δ (ppm): 8.23 (dd, *J* = 7.9 Hz, *J* = 1.5 Hz, 1H), 7.89 (dd, *J* = 7.8 Hz, *J* = 1.6 Hz, 1H), 7.69-7.64 (m, 1H), 7.54-7.45 (m, 2H), 7.42-7.37 (m, 1H), 7.13-7.08 (m, 2H), 7.06-7.03 (d, *J* = 8.4 Hz, 1H), 3.94 (s, 3H). ^{13}C NMR: $CDCl_3$, 100 MHz δ (ppm): 178.8, 165.2, 163.8, 160.9, 158.1, 133.5, 132.3, 129.3, 125.7, 124.9, 121.1, 120.8, 118.0, 112.7, 111.9, 55.7. MS (EI, 70 eV): *m/z* (relative intensity): 63 (17), 89 (14), 92 (20), 121 (100), 120 (10), 131 (49), 132 (28), 252 (48).



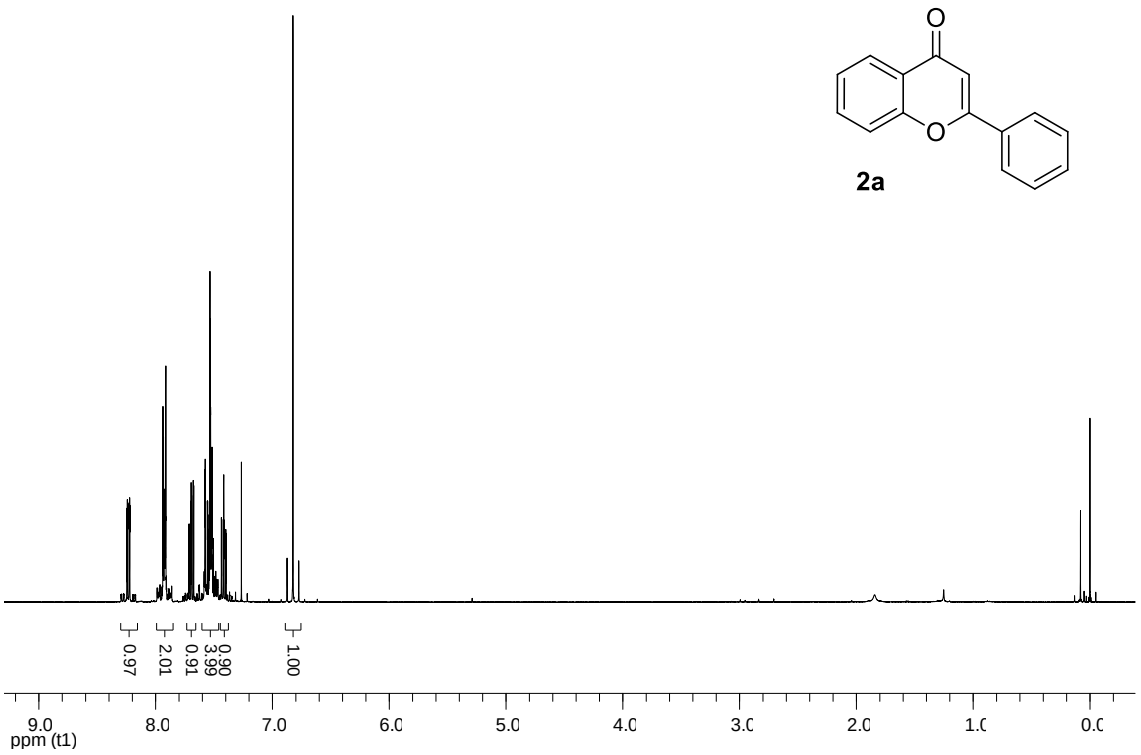
2-(4-hydroxyphenyl)-4*H*-chromen-4-one (2n): Yield: 0.048 g (80%). ^{1}H NMR: DMSO-d_6, 400 MHz, δ (ppm): 10.34 (brs,1H), 8.04-8.01 (m, 1H), 7.98-7.94 (m, 2H), 7.82-7.78 (m, 1H), 7.75-7.72 (m, 1H), 7.49-7.45 (m, 1H), 6.96-6.92 (m, 2H), 6.87 (s, 1H). ^{13}C NMR: DMSO-d_6, 100 MHz δ (ppm): 176.8, 163.0, 160.9,

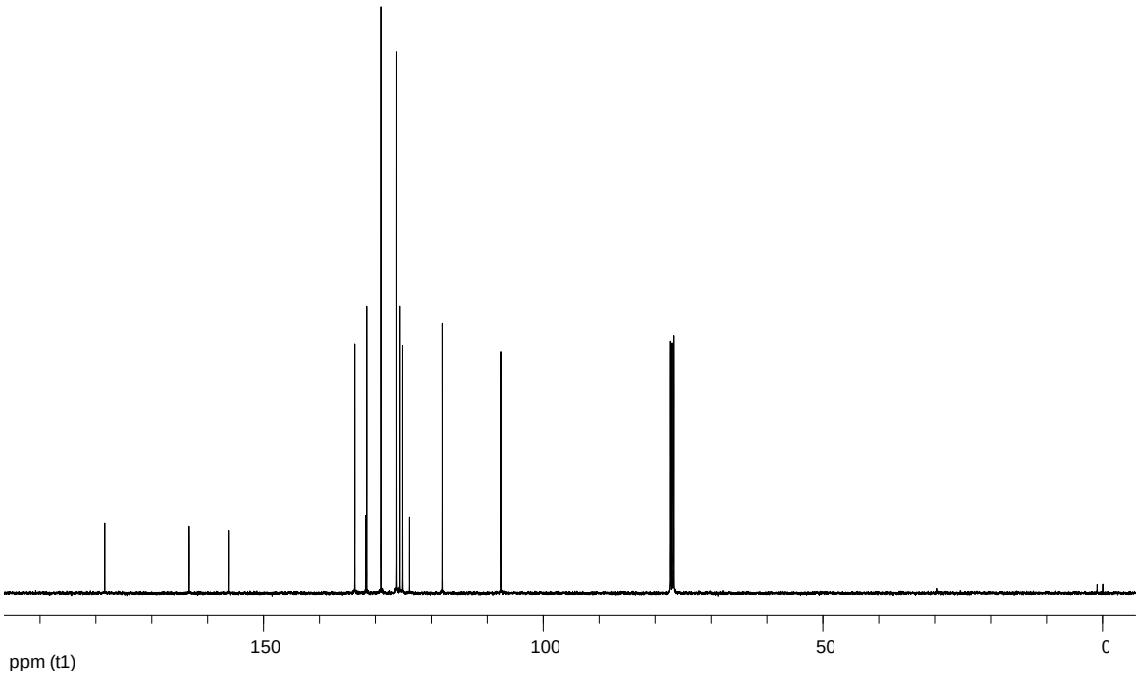
155.5, 134.0, 128.3, 125.3, 124.7, 123.2, 121.5, 118.3, 115.9, 104.7. MS (EI, 70 eV): *m/z* (relative intensity): 63 (21), 64 (18), 89 (16), 92 (29), 118 (38), 121 (72), 210 (27), 238 (100).

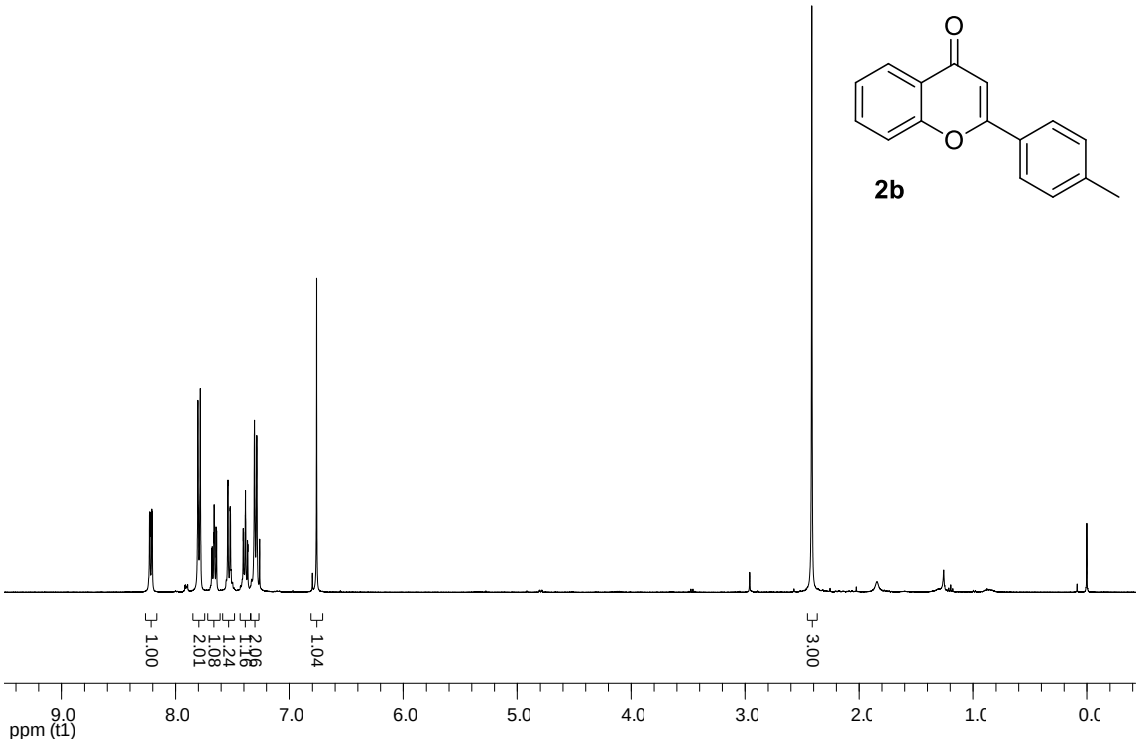


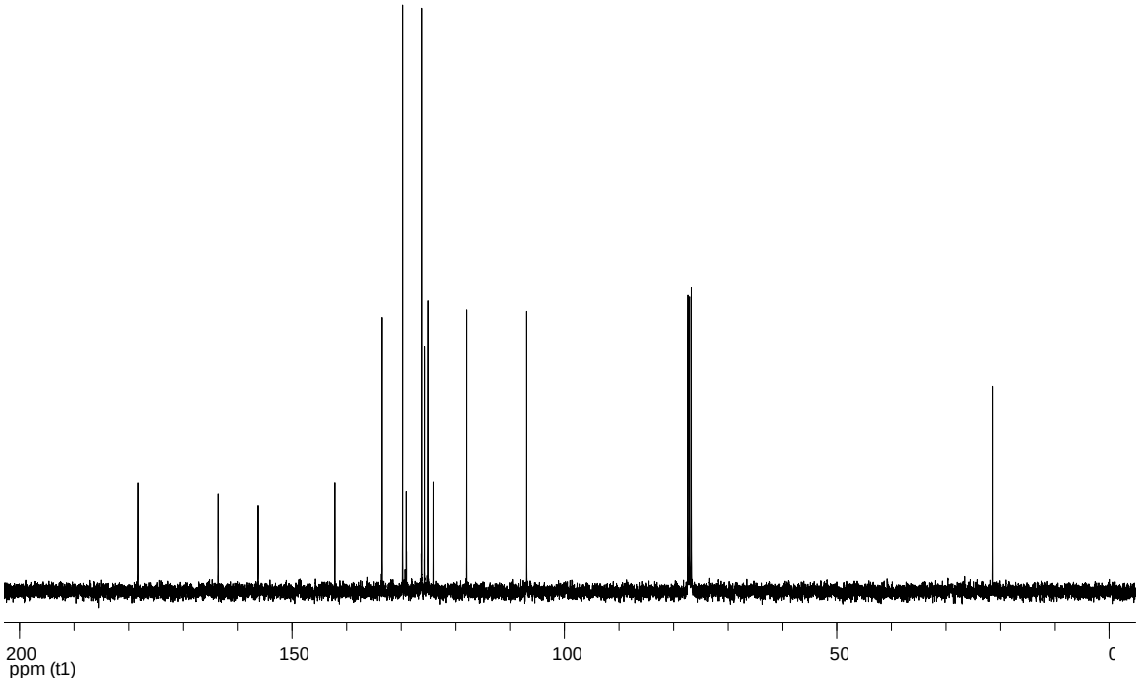
2-(4-(phenylthio)phenyl)-4*H*-chromen-4-one (2o): Yield: 0.019 g (23%). ^{1}H NMR: $CDCl_3$, 400 MHz, δ (ppm): 8.22 (ddd, *J* = 7.9 Hz, *J* = 1.7 Hz, *J* = 0.4 Hz, 1H), 7.81-7.78 (m, 2H), 7.70-7.65 (m, 1H), 7.54-7.48 (m, 3H), 7.43-7.37 (m, 4H), 7.32-7.29 (m, 2H), 6.76 (s, 1H). ^{13}C NMR: $CDCl_3$, 100 MHz δ (ppm): 178.3, 162.8, 156.2, 142.8, 133.7, 133.5, 132.6, 129.7, 129.2, 128.6, 128.5, 126.7, 125.7, 125.2, 124.0, 118.0, 107.1. MS (EI, 70 eV): *m/z* (relative intensity): 92 (7) 165 (10), 208 (19), 209 (16), 210 (28), 221 (16), 329 (11), 221 (16), 330 (100), 332 (7).

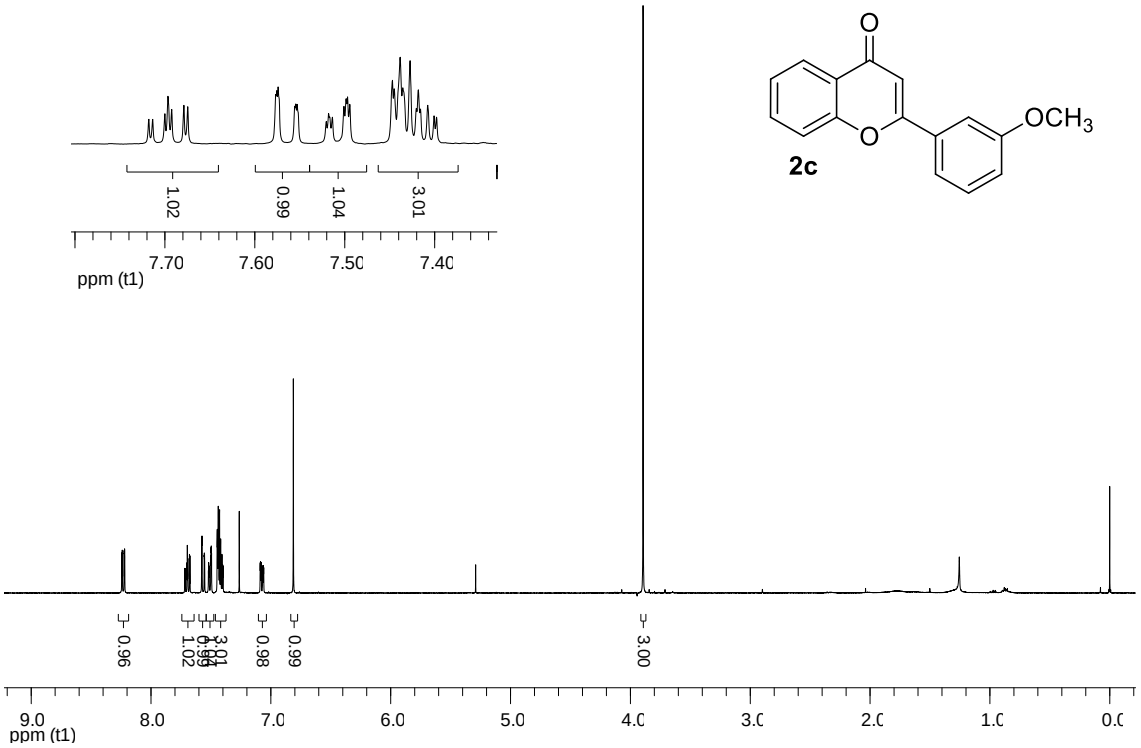
NMR ^{1}H and ^{13}C spectra

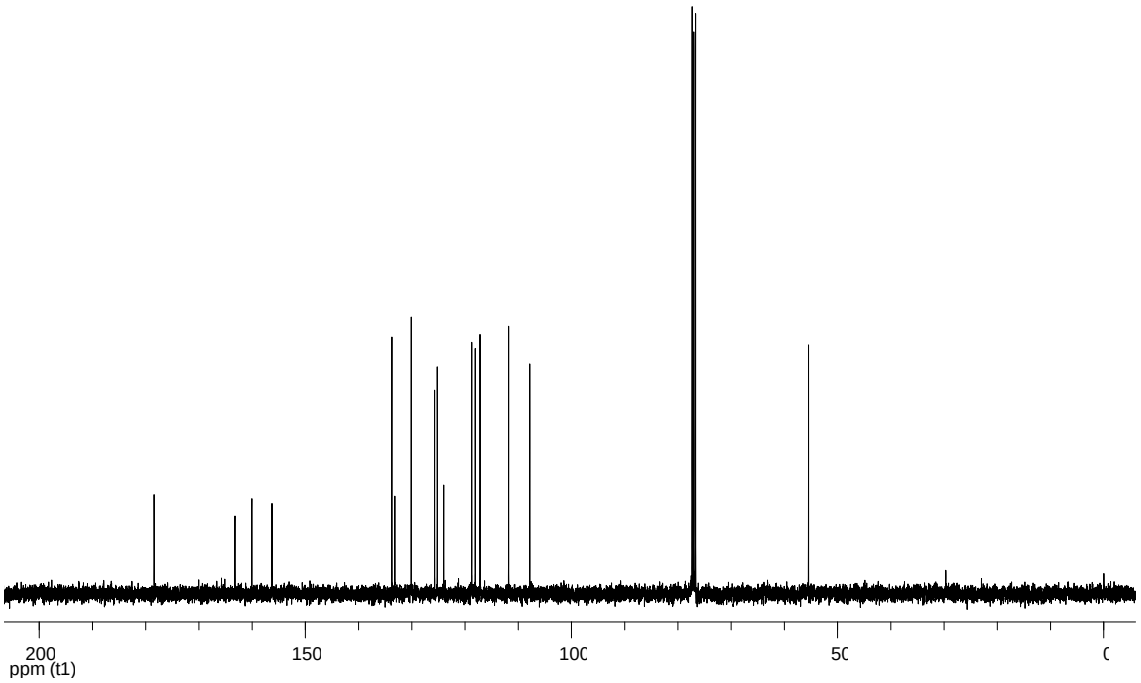


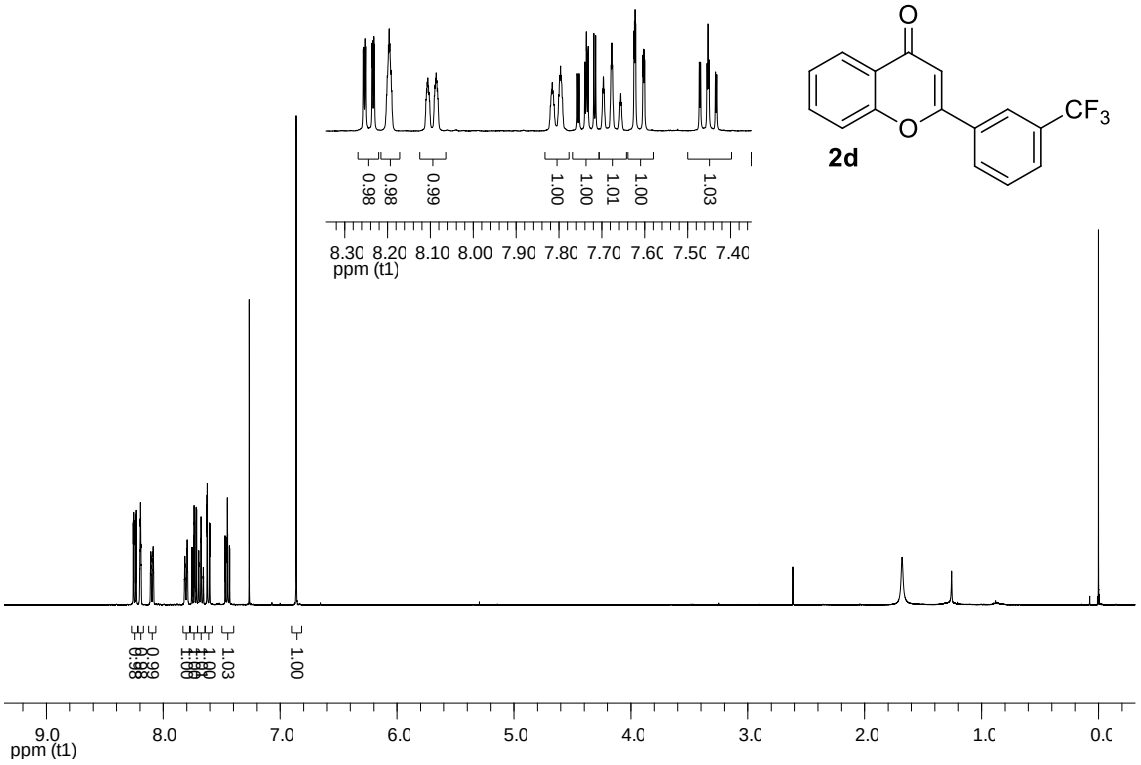


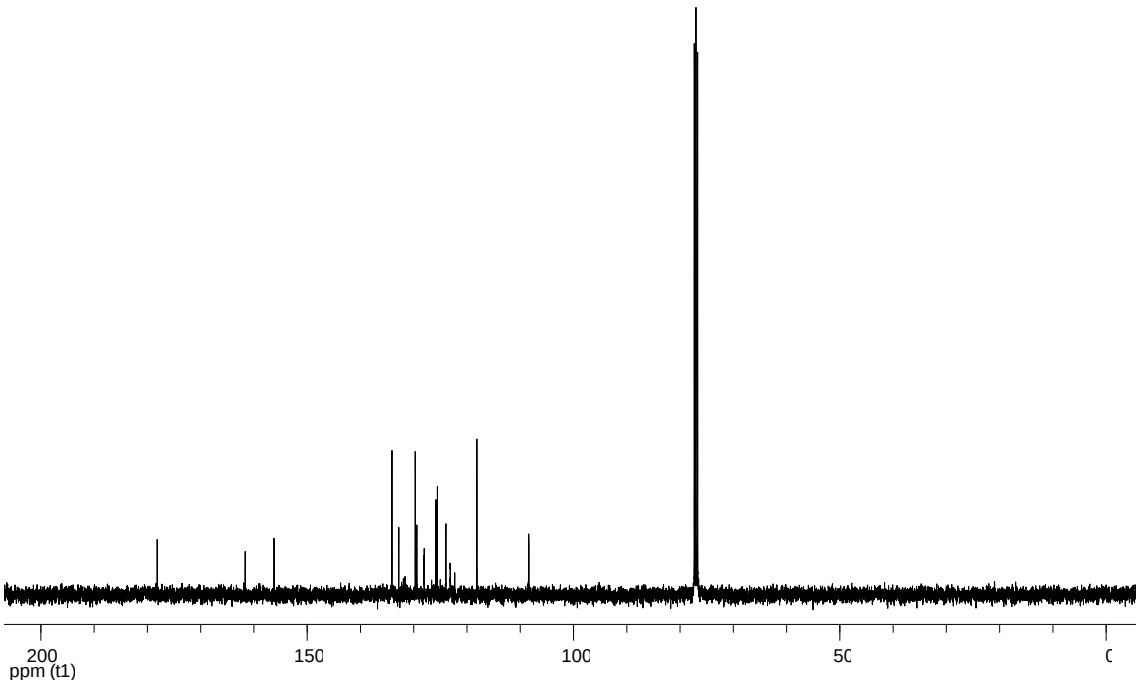

O
O
2b
1.00
2.01
1.08
1.24
1.16
2.06
1.04
3.00
9.0
8.0
7.0
6.0
5.0
4.0
3.0
2.0
1.0
0.0
ppm (t1)


200
150
100
50
0
ppm (t1)


1.02
0.99
1.04
3.01
7.70
7.60
7.50
7.40
ppm (t1)
O
O
OCH3
2c
0.96
1.02
0.98
0.99
3.00
9.0
8.0
7.0
6.0
5.0
4.0
3.0
2.0
1.0
0.0
ppm (t1)

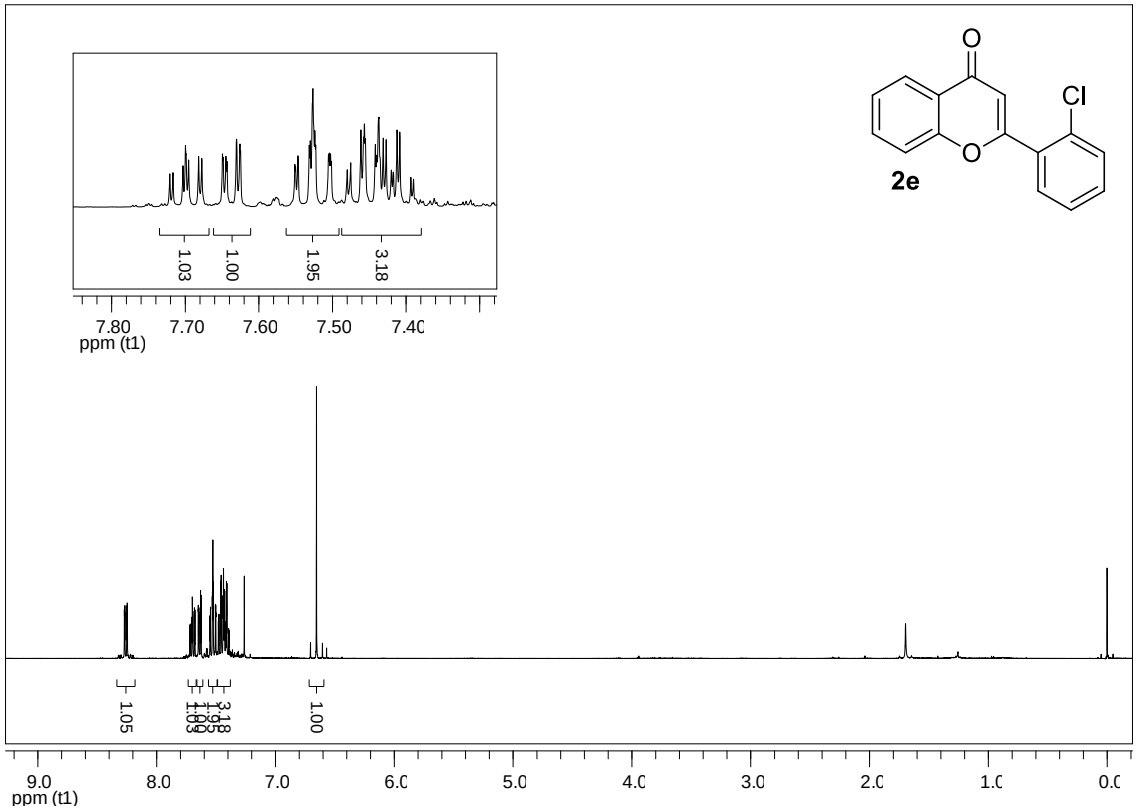

200
150
100
50
0
ppm (t1)

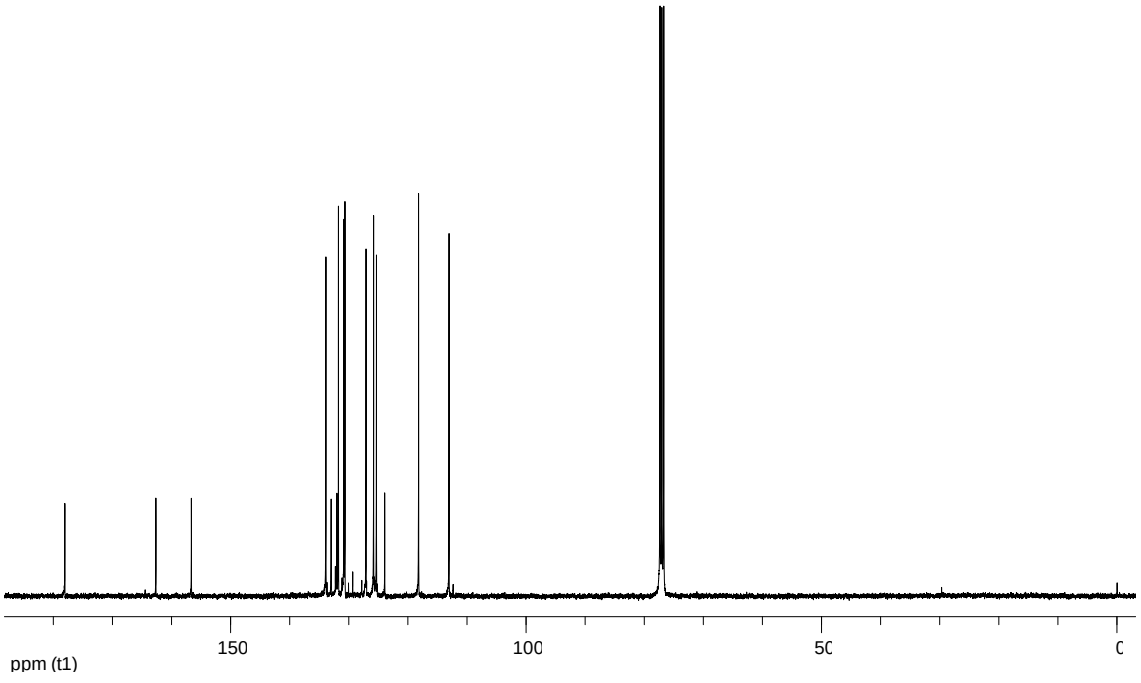

0.98
0.98
0.99
1.00
1.00
1.01
1.00
1.03
8.30 8.20 8.10 8.00 7.90 7.80 7.70 7.60 7.50 7.40
ppm (t1)
1.03
1.00
9.0 8.0 7.0 6.0 5.0 4.0 3.0 2.0 1.0 0.0
ppm (t1)

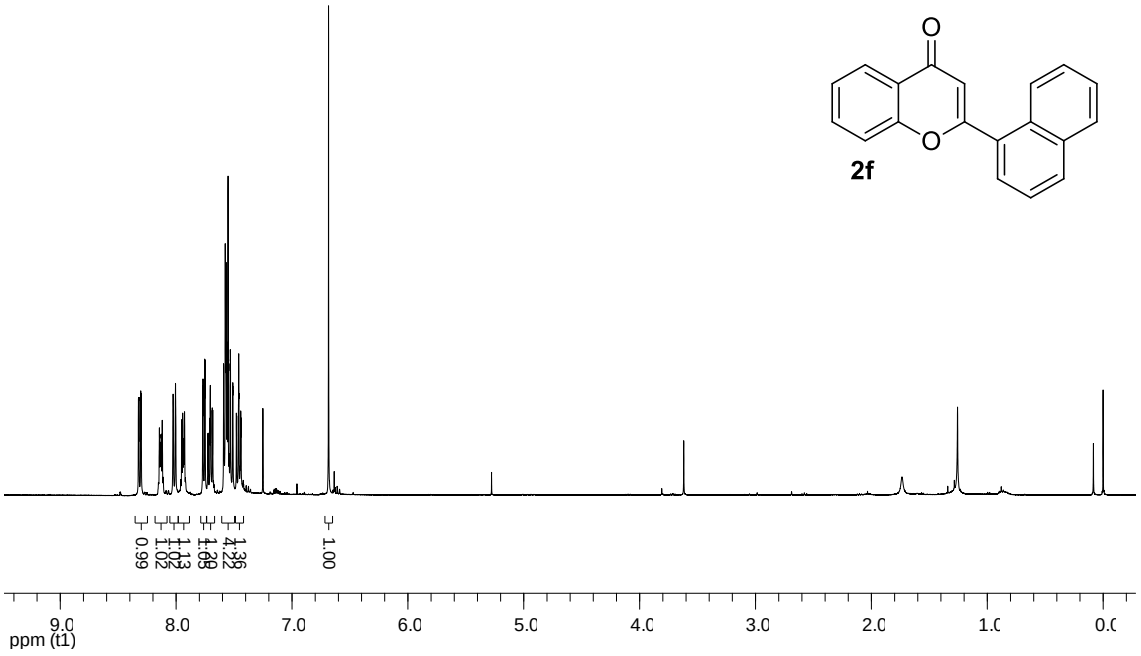

200 150 100 50 0
ppm (t1)

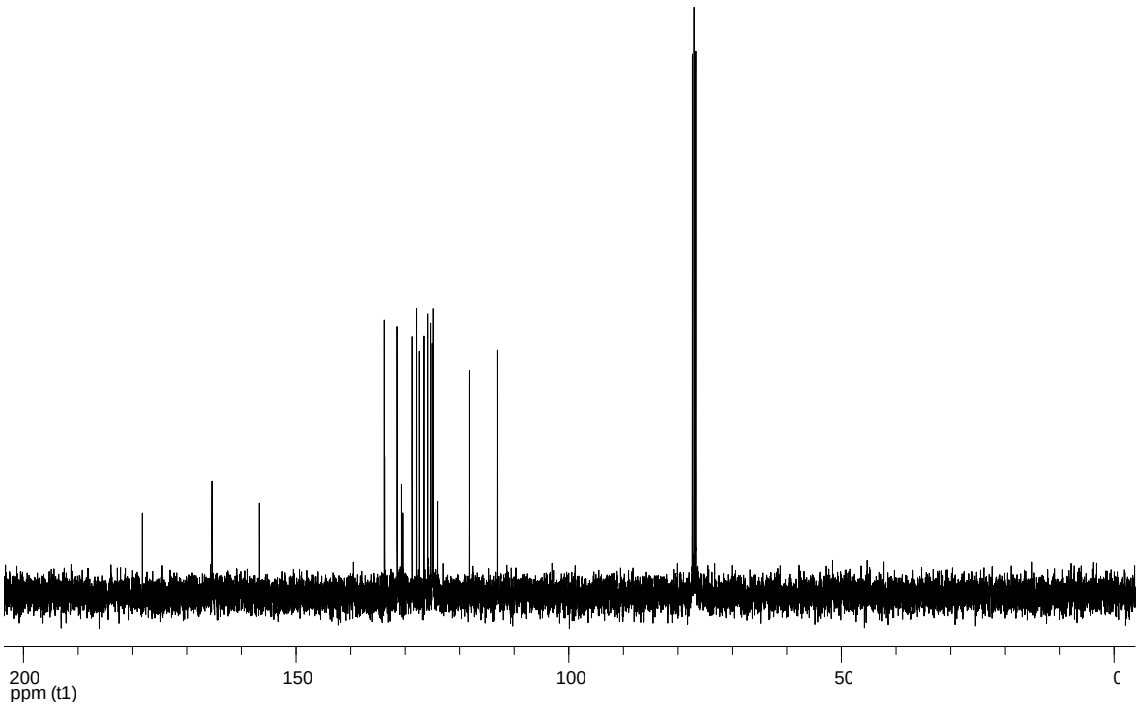
O
O
CF3

2d


O
Cl
O
2e
1.03
1.00
1.95
3.18
7.80
7.70
7.60
7.50
7.40
ppm (t1)
1.05
1.00
9.0
8.0
7.0
6.0
5.0
4.0
3.0
2.0
1.0
0.0
ppm (t1)


150
100
50
0
ppm (t1)

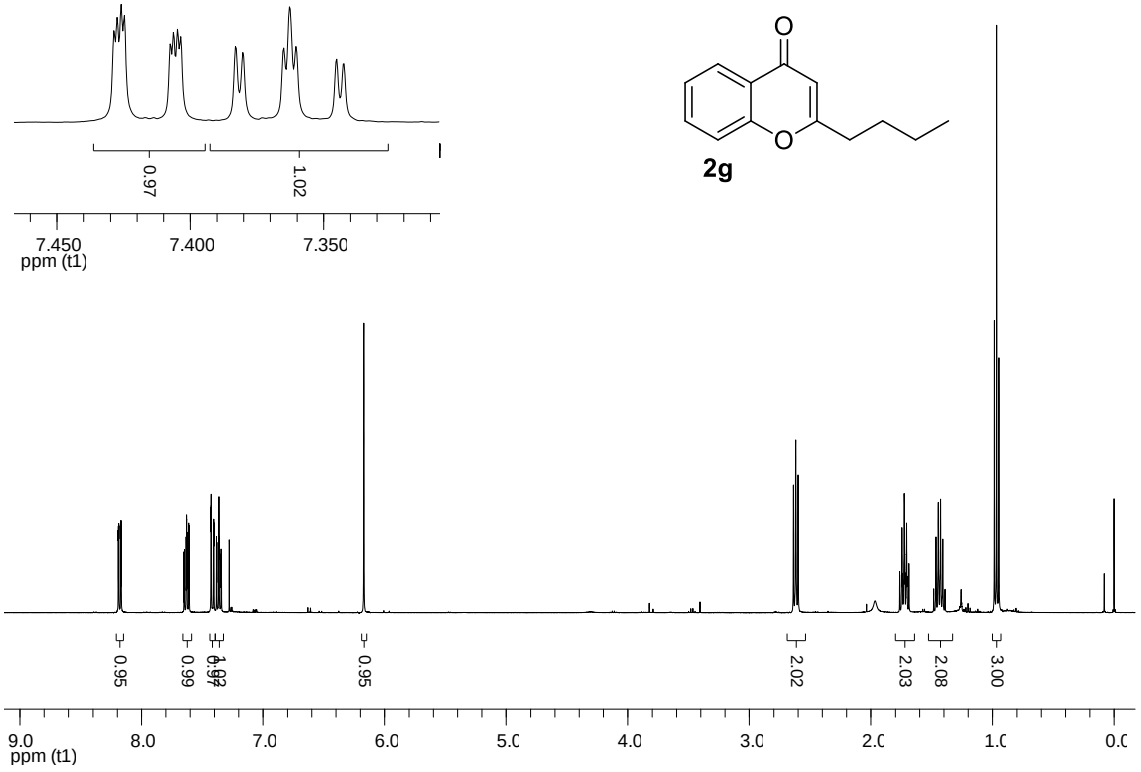

O
O
2f
0.99
1.02
1.13
1.09
1.36
1.00
9.0
8.0
7.0
6.0
5.0
4.0
3.0
2.0
1.0
0.0
ppm (t1)

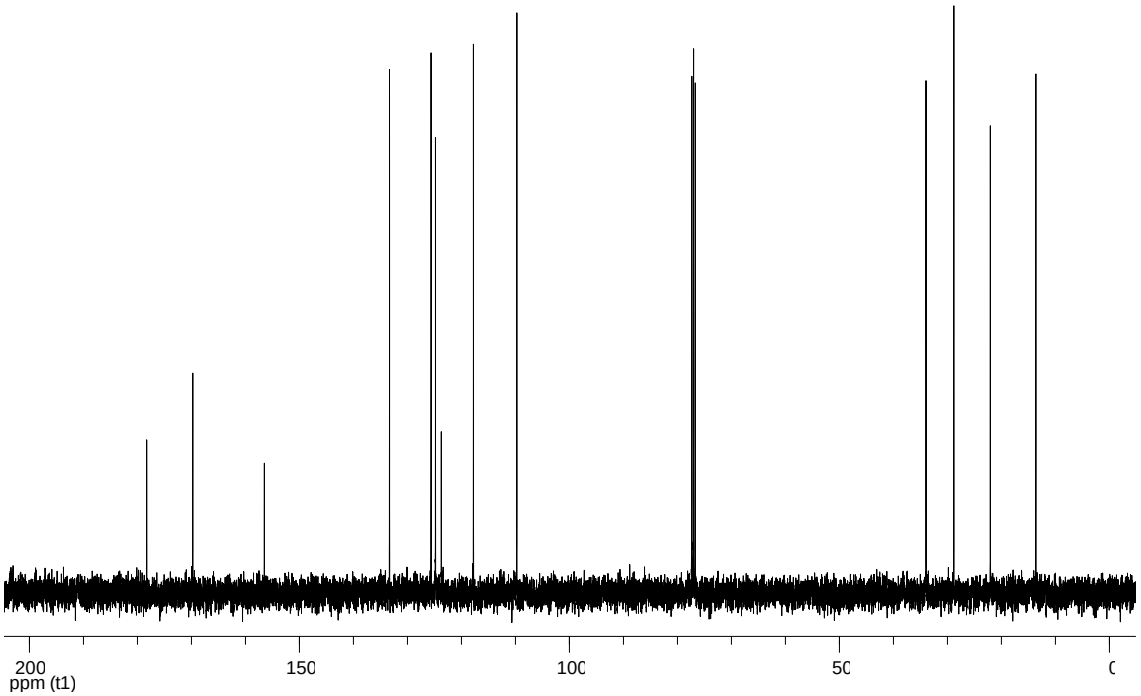

200
150
100
50
0
ppm (t1)

0.97
1.02
7.450
ppm (t1)
7.400
7.350

O
O

2g

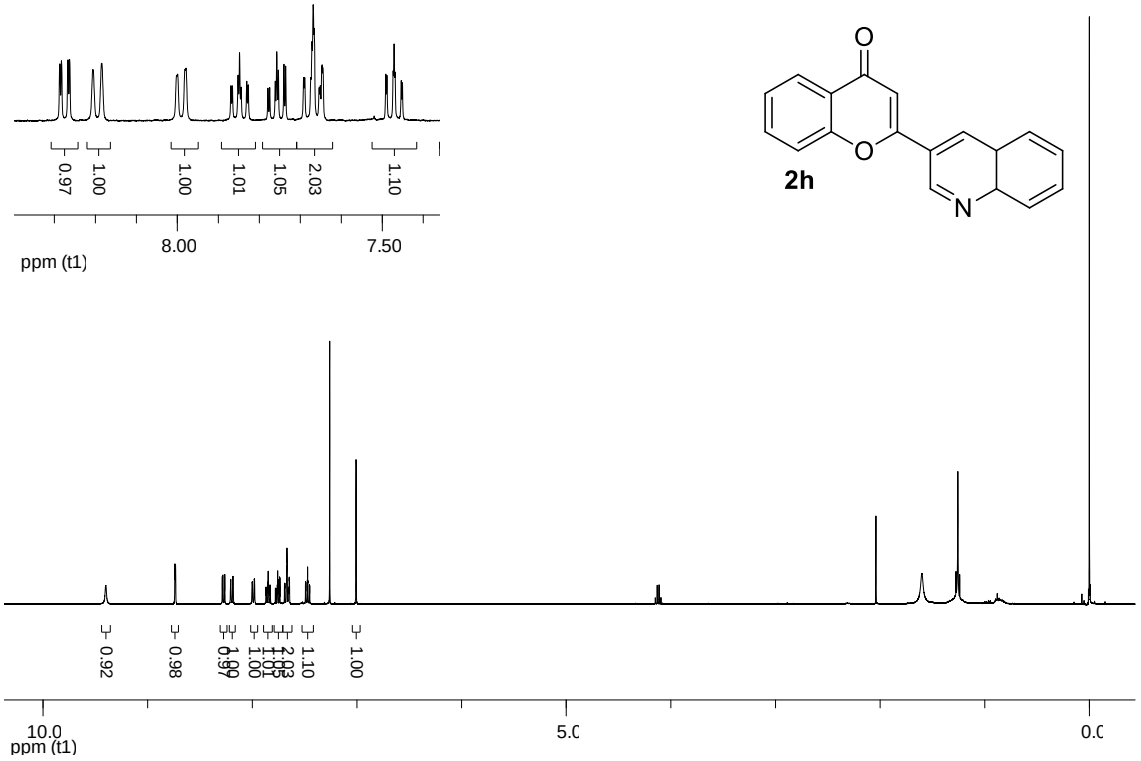

0.95
0.99
0.95
2.02
2.03
2.08
3.00
9.0
ppm (t1)
8.0
7.0
6.0
4.0
3.0
2.0
1.0
0.0

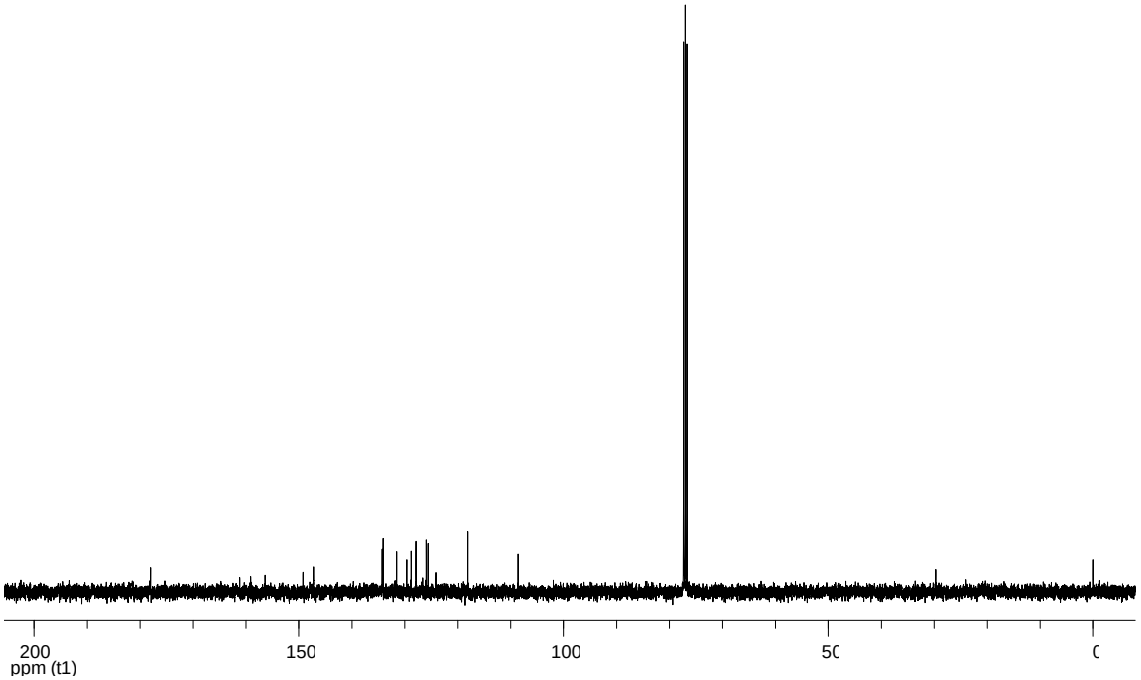

200
ppm (t1)
150
100
50

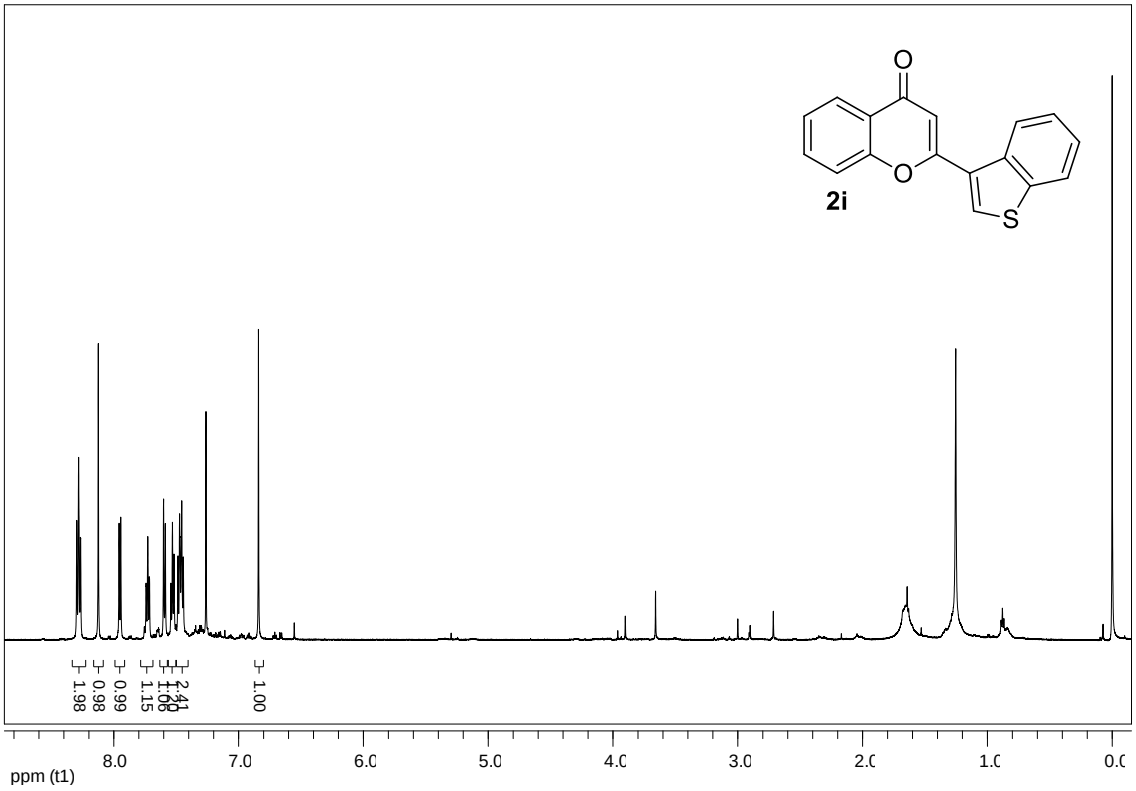
0.97
1.00
1.00
1.01
1.05
2.03
1.10
8.00
7.50
ppm (t1)

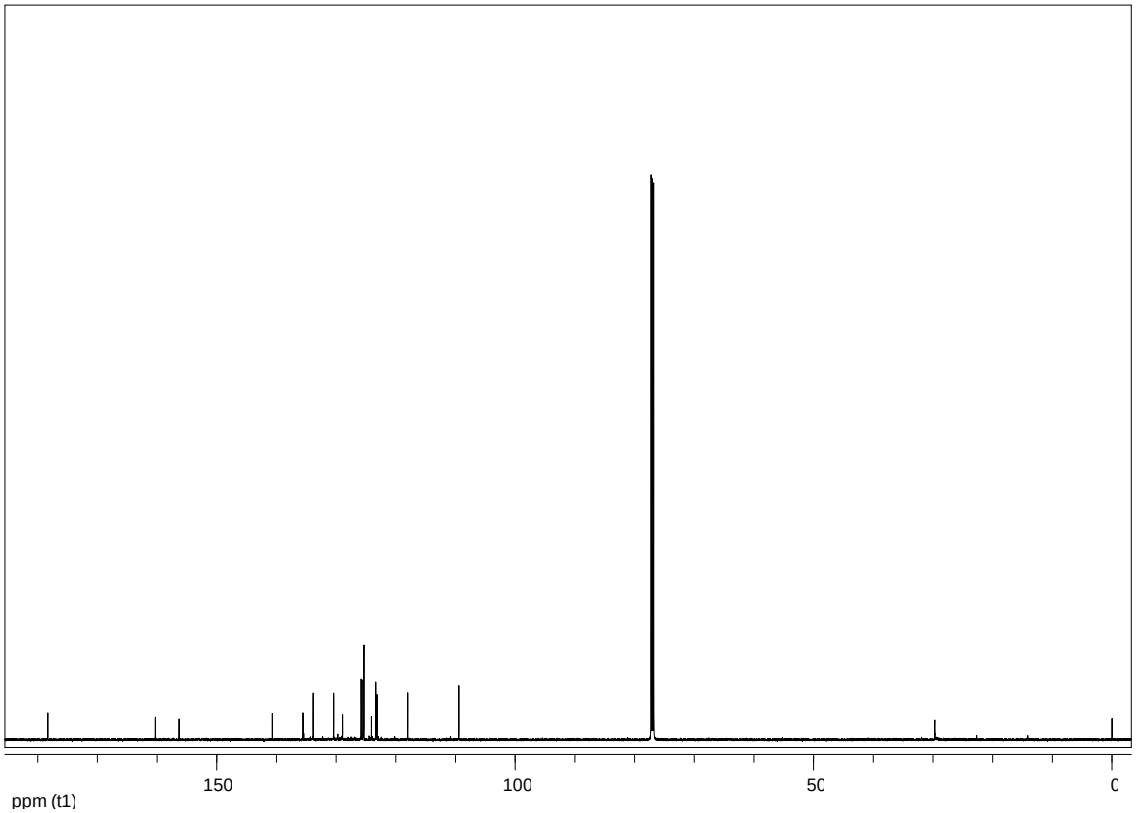
O
O
N

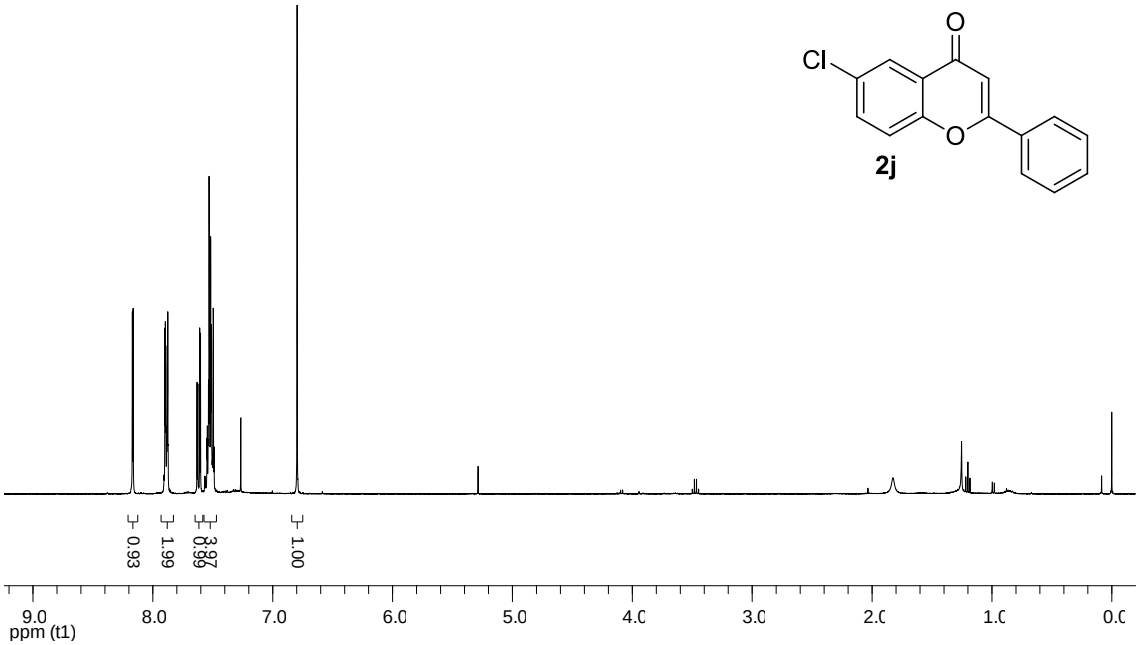
2h

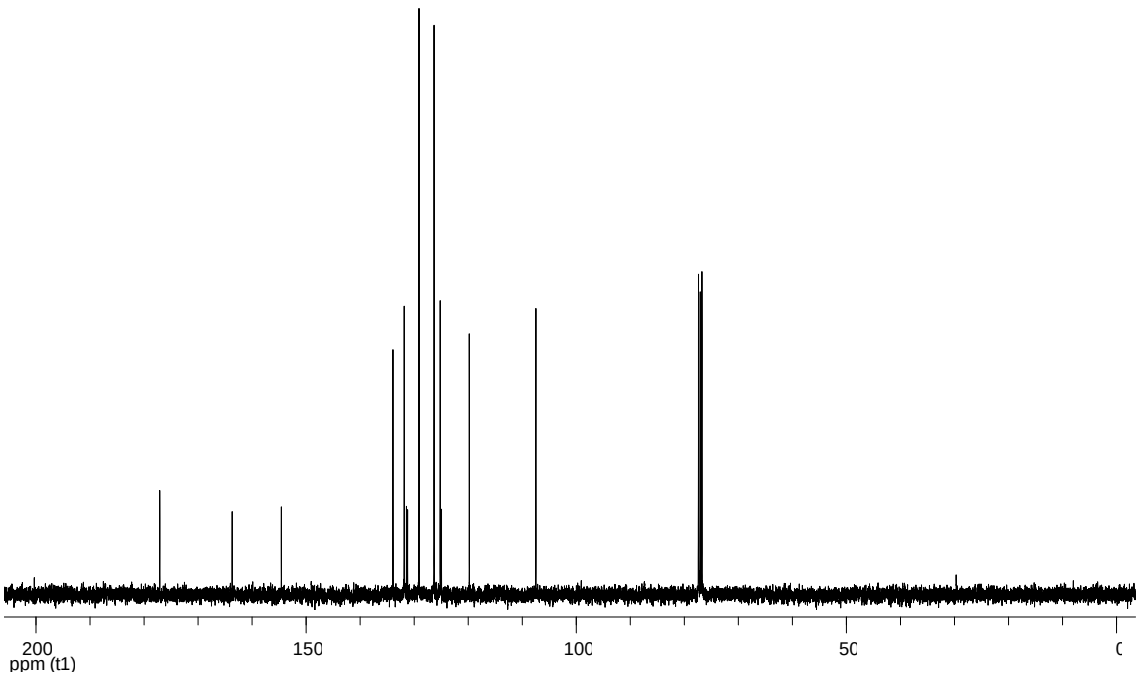

0.92
0.98
1.10
1.00
10.0
ppm (t1)
5.0
0.0

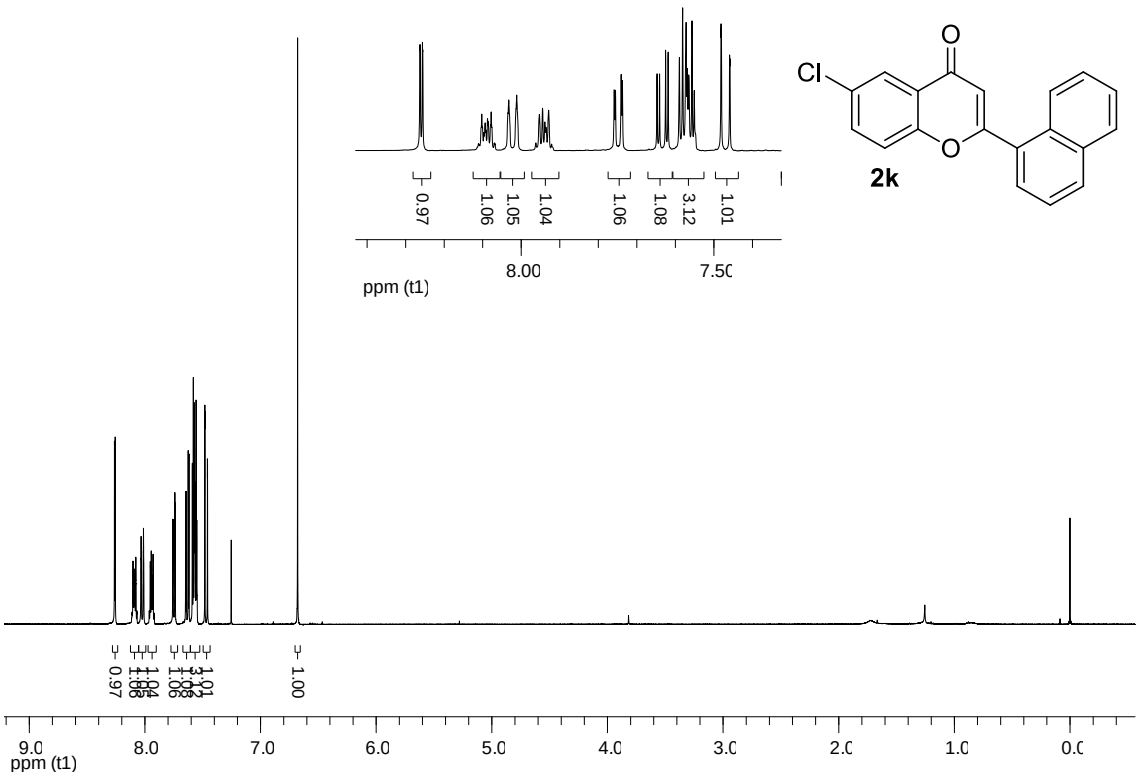

200
ppm (t1)
150
100
50


O
O
S
2i
1.98
0.98
0.99
1.15
1.00
8.0
7.0
6.0
5.0
4.0
3.0
2.0
1.0
0.0
ppm (t1)


150
100
50
0
ppm (t1)

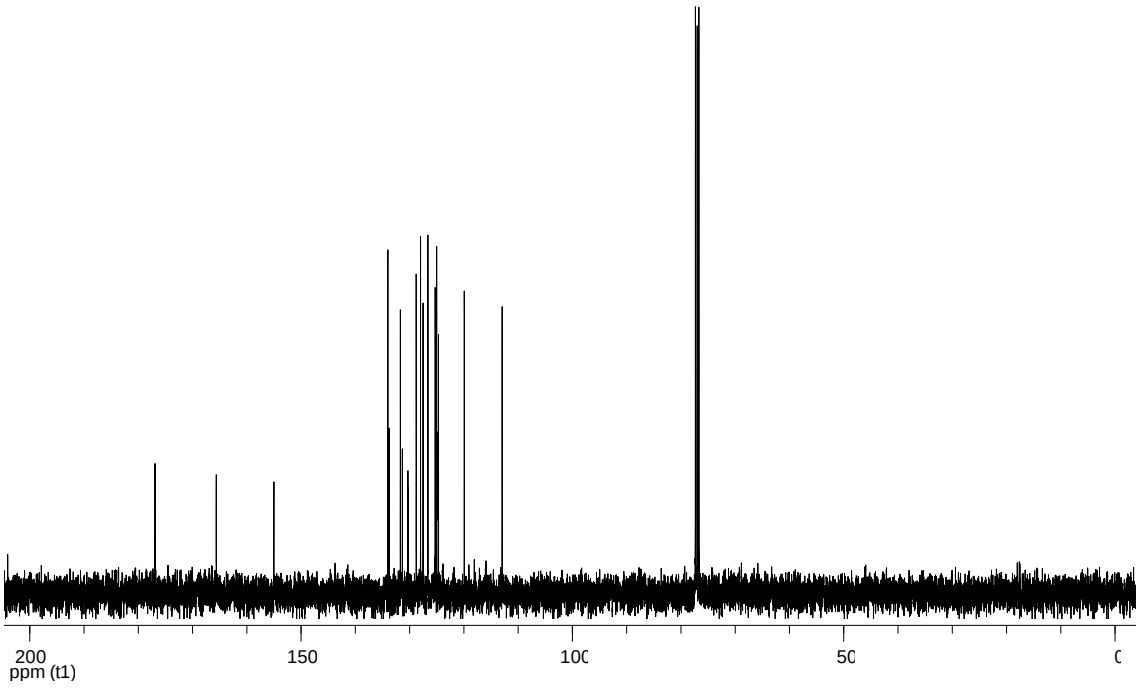

Cl
O
O
2j
0.93
1.99
3.97
1.00
9.0
8.0
7.0
6.0
5.0
4.0
3.0
2.0
1.0
0.0
ppm (t1)

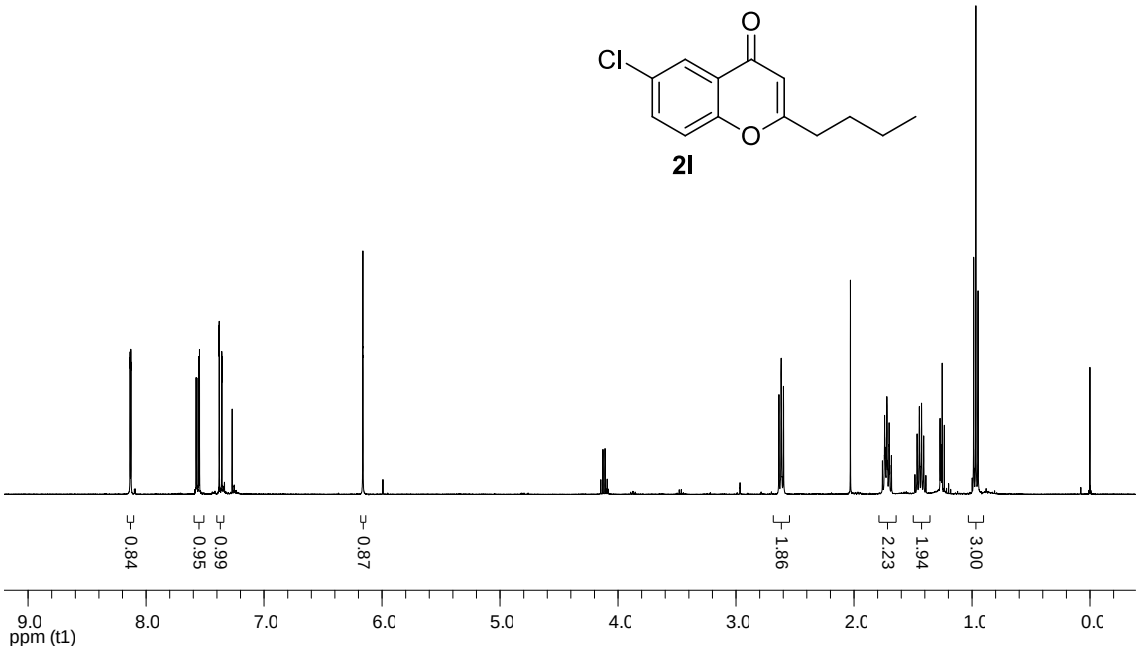

200
150
100
50
0
ppm (t1)

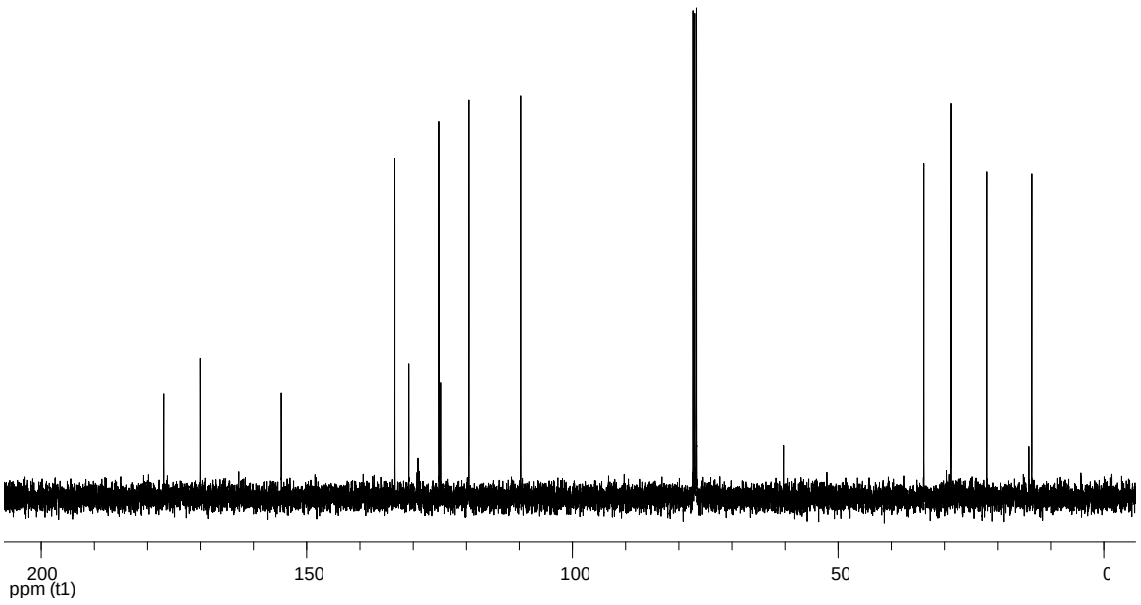

0.97
1.06
1.05
1.04
1.06
1.08
3.12
1.01
8.00
7.50
ppm (t1)
0.97
1.00
9.0
8.0
7.0
6.0
5.0
4.0
3.0
2.0
1.0
0.0
ppm (t1)

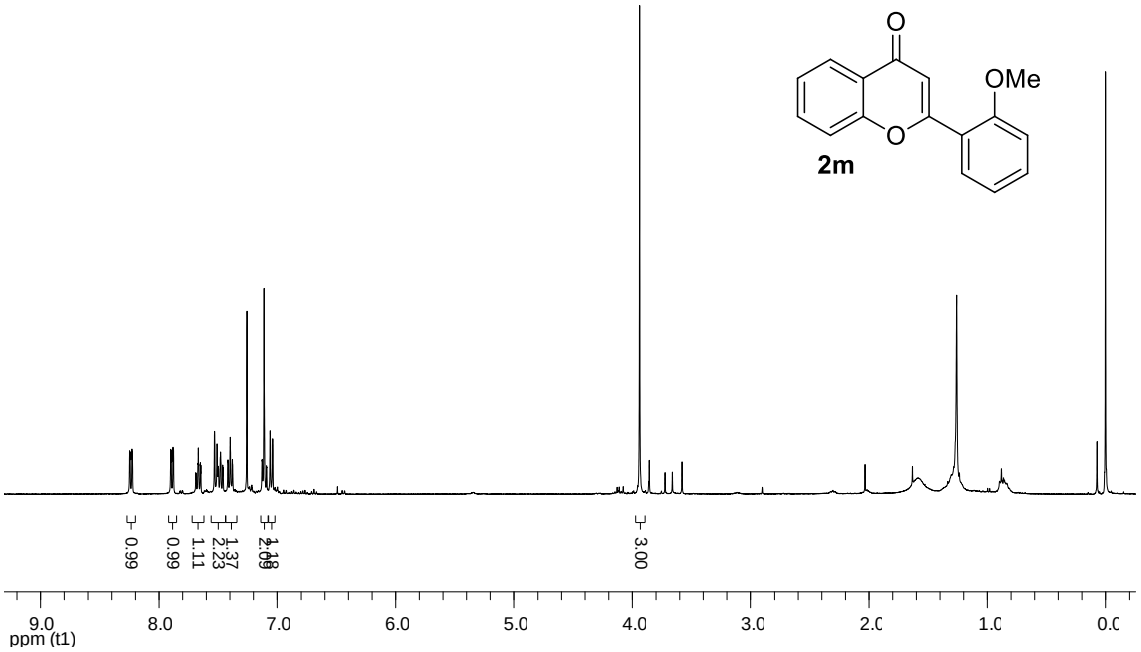
Cl
O
O

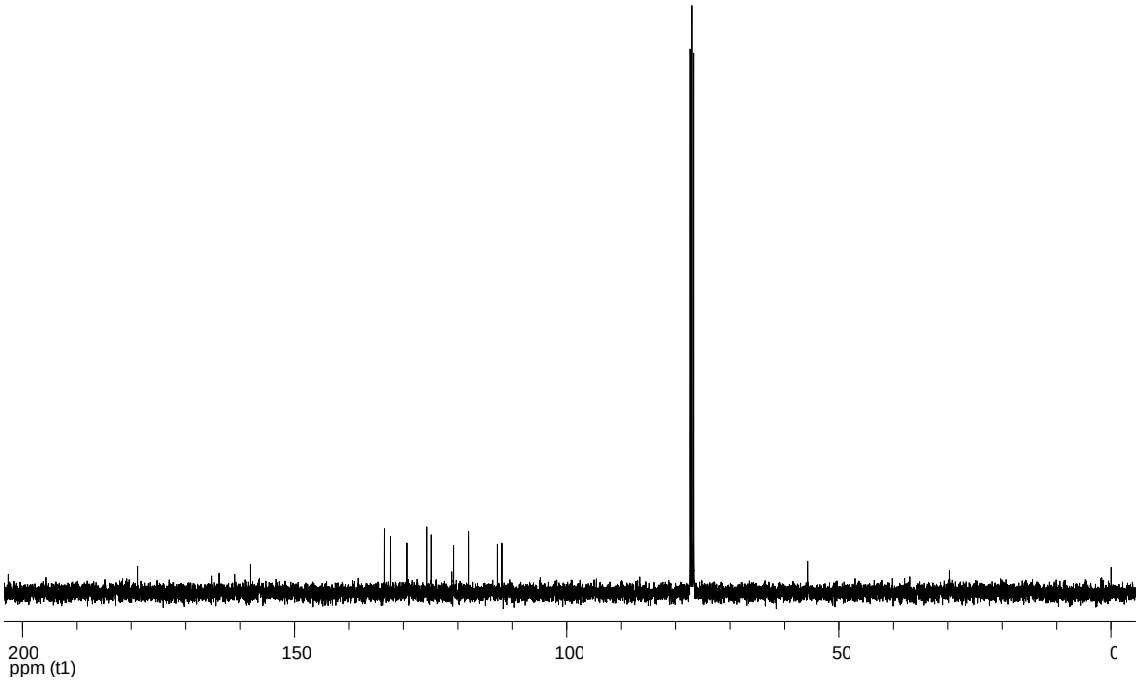
2k

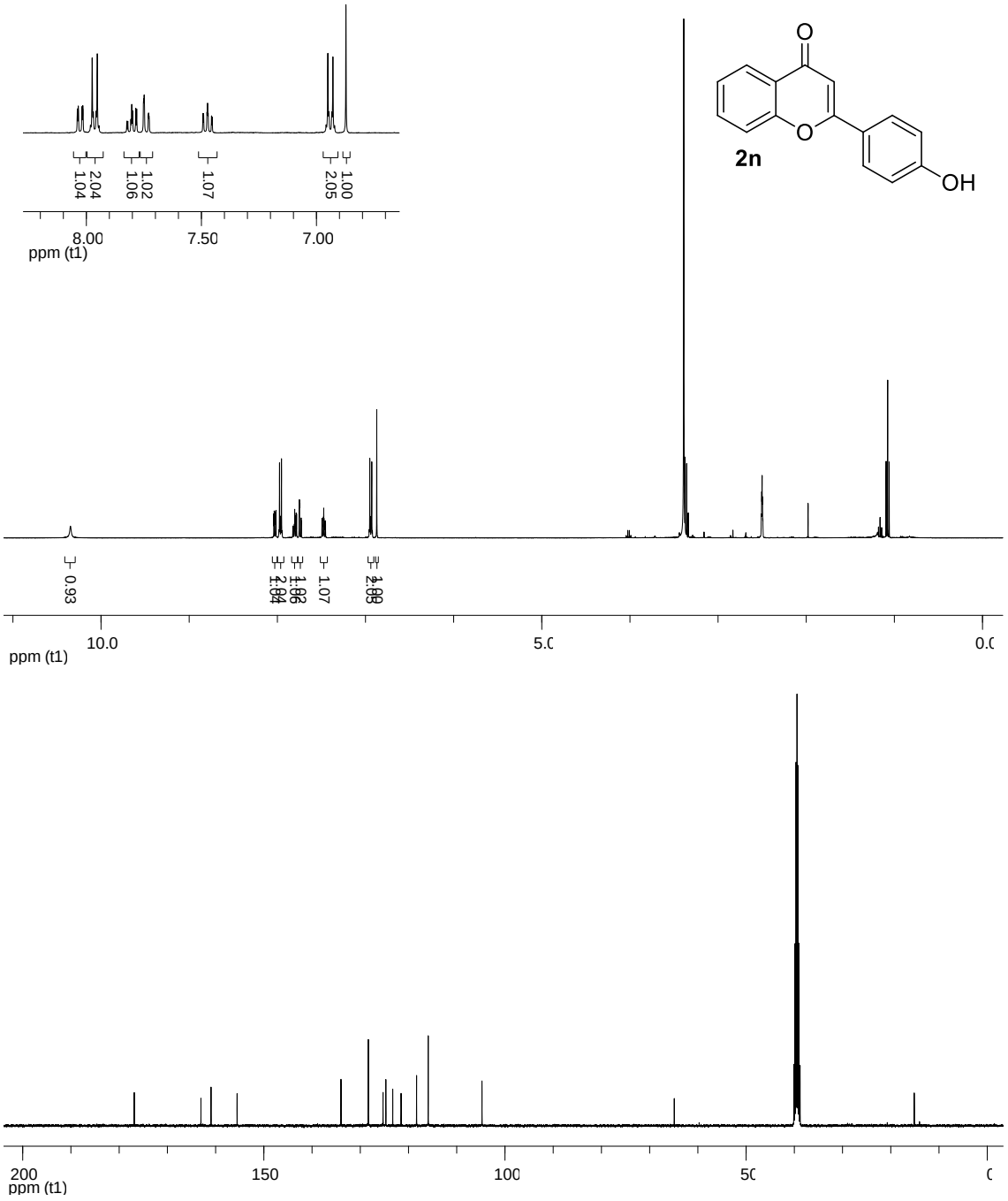

200
150
100
50
0
ppm (t1)

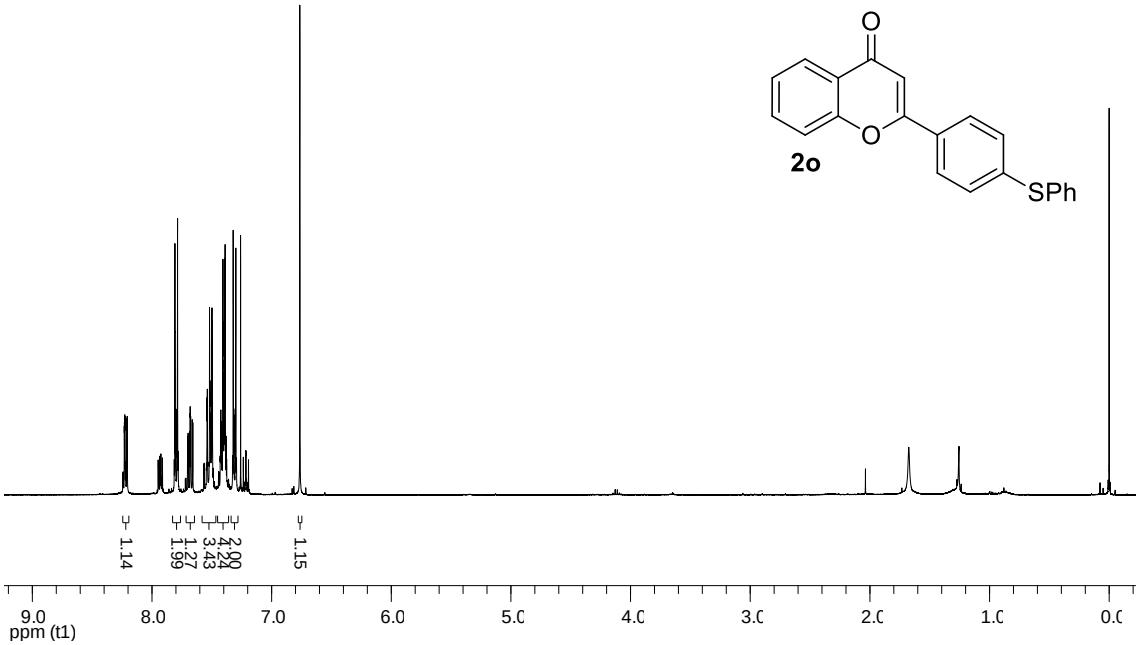

Cl
O
O
2l
0.84
0.95
0.99
0.87
1.86
2.23
1.94
3.00
9.0
8.0
7.0
6.0
5.0
4.0
3.0
2.0
1.0
0.0
ppm (t1)


200
150
100
50
0
ppm (t1)

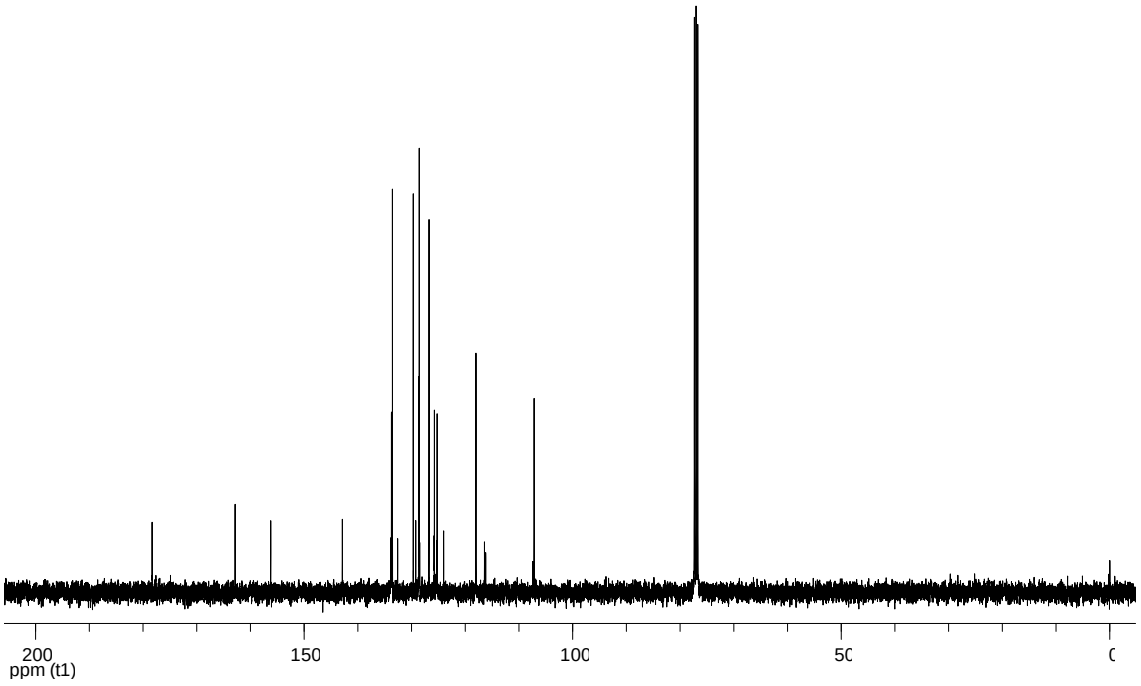

O
OMe
O
2m
0.99
0.99
1.11
2.23
1.37
2.09
3.00
9.0
8.0
7.0
6.0
5.0
4.0
3.0
2.0
1.0
0.0
ppm (t1)


200
150
100
50
0
ppm (t1)


1.04
2.04
1.06
1.02
1.07
2.05
1.00
8.00
7.50
7.00
ppm (t1)
O
O
2n
OH
0.93
1.07
10.0
5.0
ppm (t1)
200
150
100
50
ppm (t1)


O
O
SPh
1.14
1.27
1.99
3.43
3.24
2.00
1.15
9.0
8.0
7.0
6.0
5.0
4.0
3.0
2.0
1.0
0.0
ppm (t1)

2o


200
150
100
50
0
ppm (t1)