Total Synthesis of (−)-Stemoamide

Staffan Torssell, Emil Wanngren and Peter Somfai*

KTH Chemical Science and Engineering, Organic Chemistry
Royal Institute of Technology
S-100 44 Stockholm (Sweden).

somfai@kth.se

Supporting Information

Table of Content **Page**

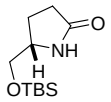
General Experimental Procedure S-2

^{1}H and ^{13}C NMR spectra for compound **1**-**3**, **5**-**7**, **10**, **12**, **13** S-3 – S-12

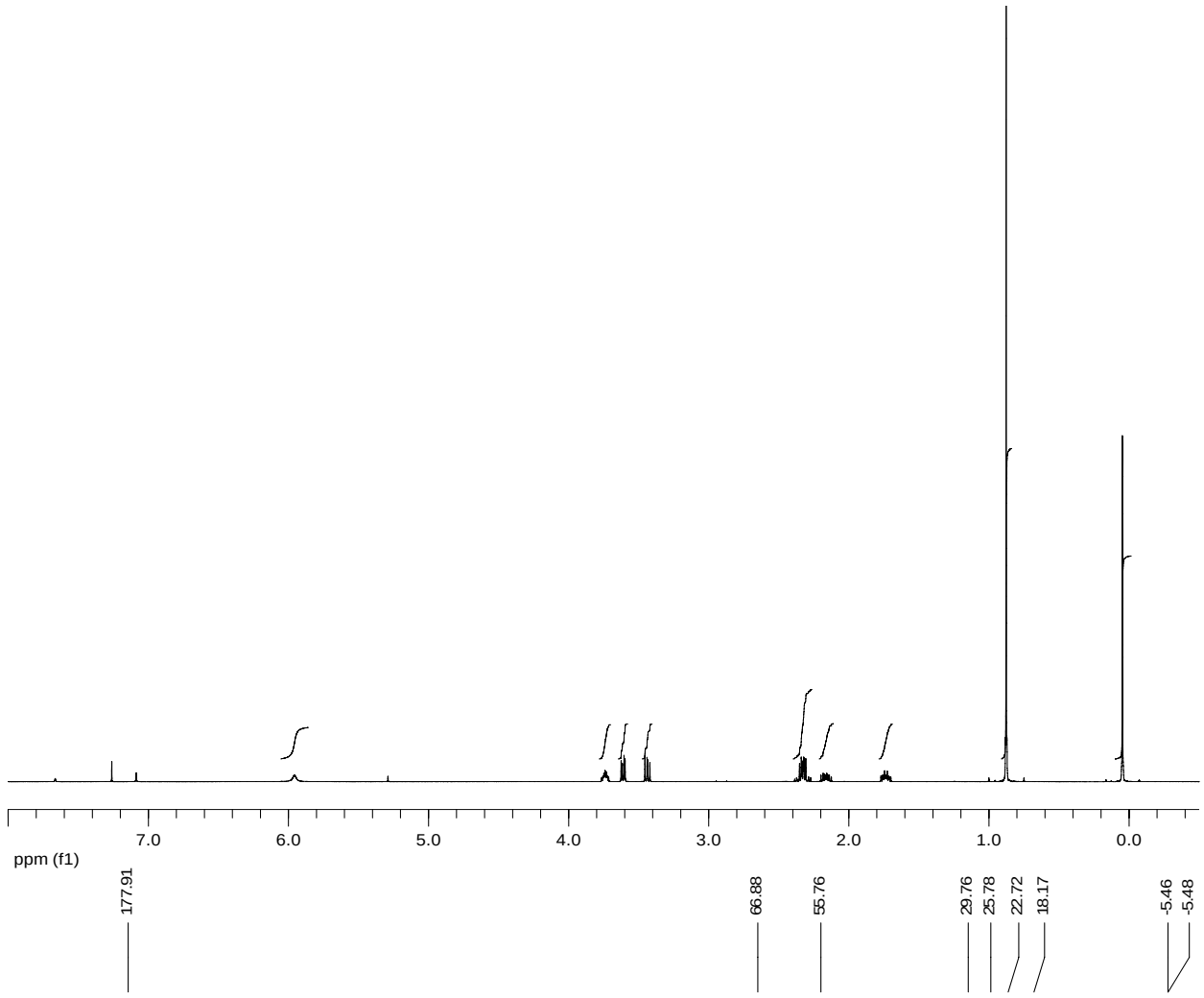
* Corresponding author. Phone: +46 8 7906960. Fax: +46 8 7912333

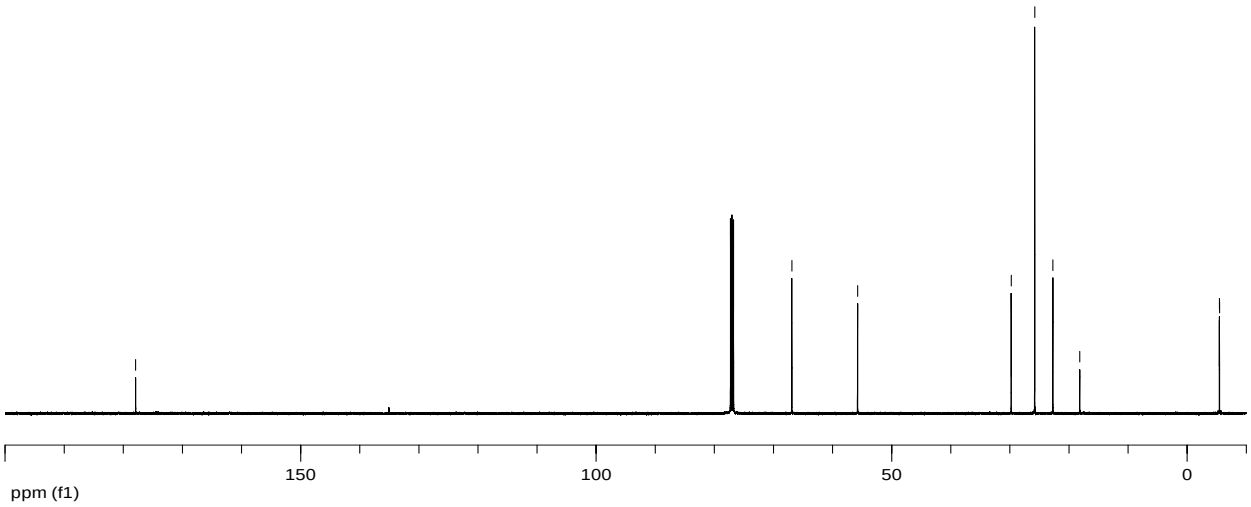
General Experimental Procedure.

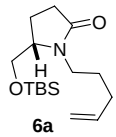
^{1}H and ^{13}C NMR spectra were recorded at 500 MHz (125 MHz) in $CDCl_3$ using the residual peak of $CHCl_3$ (^{1}H NMR δ = 7.26 ppm) and $CDCl_3$ (^{13}C NMR δ = 77.0 ppm) as internal standard. Chemical shifts are reported in the δ-scale with multiplicity (br = broad, s = singlet, d = doublet, t = triplet, q = quartet and m = multiplet), integration and coupling constant (Hz). IR-spectra were recorded on a FTIR instrument and only the strongest/structurally most important peaks (ν_{max}, cm^{-1}) are listed. Optical rotations were determined at the sodium D line (589 nm) at ambient temperature. Melting points are uncorrected. Dichloromethane (CH_2Cl_2), dimethylformamide (DMF), hexane and tetrahydrofuran (THF) were dried by passing through a solvent column composed of activated alumina. DMPU and ethyl α-bromoacetate were distilled from CaH_2 under a N_2 atmosphere before use. Air- and moisture sensitive reactions were carried out in flame-dried, septum-capped flasks under an atmospheric pressure of nitrogen. All liquid reagents were transferred via oven-dried syringes. Commercially available compounds were used without further purification unless otherwise indicated. TLC analyses were visualized using UV and a solution of phosphomolybdic acid in ethanol (5 wt%). Flash chromatography was performed using silica gel 60 (35-63 μm).


O
NH
H
OTBS

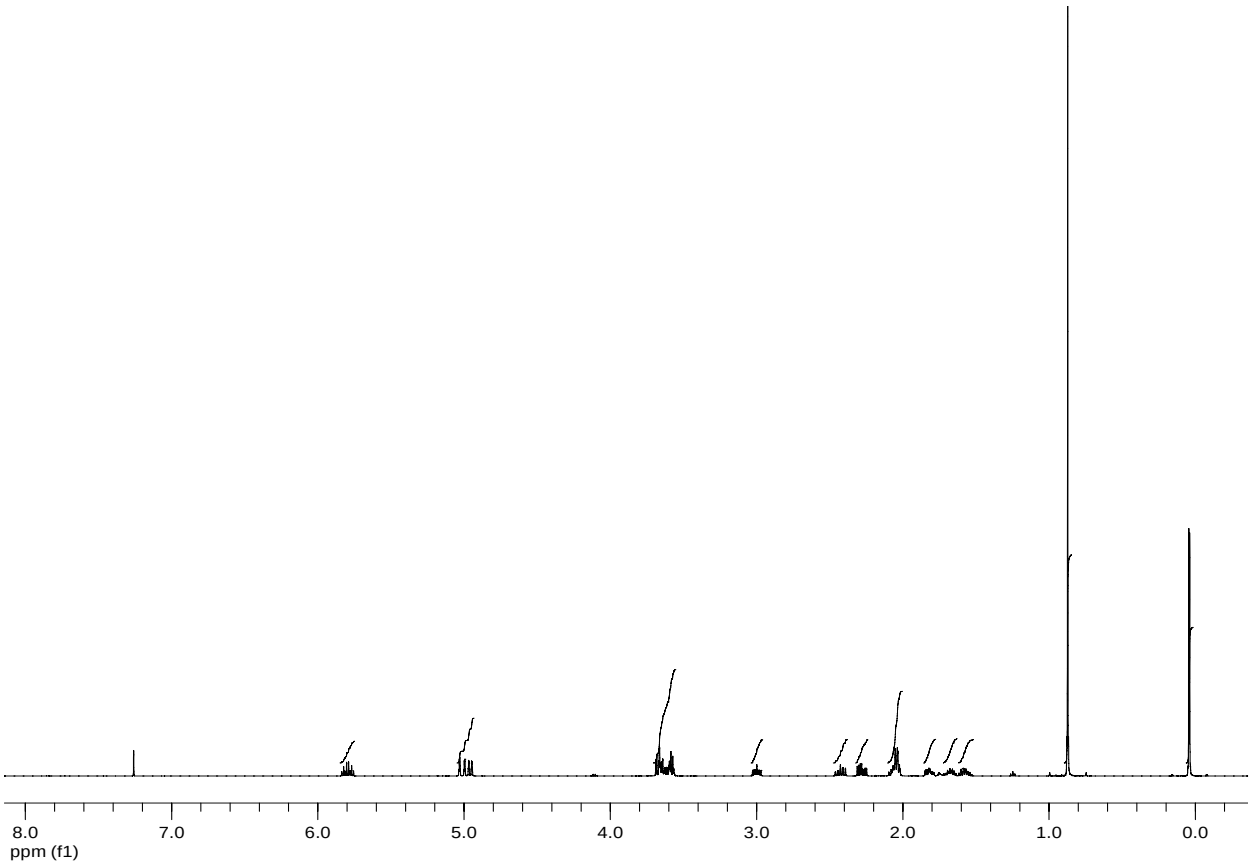
6

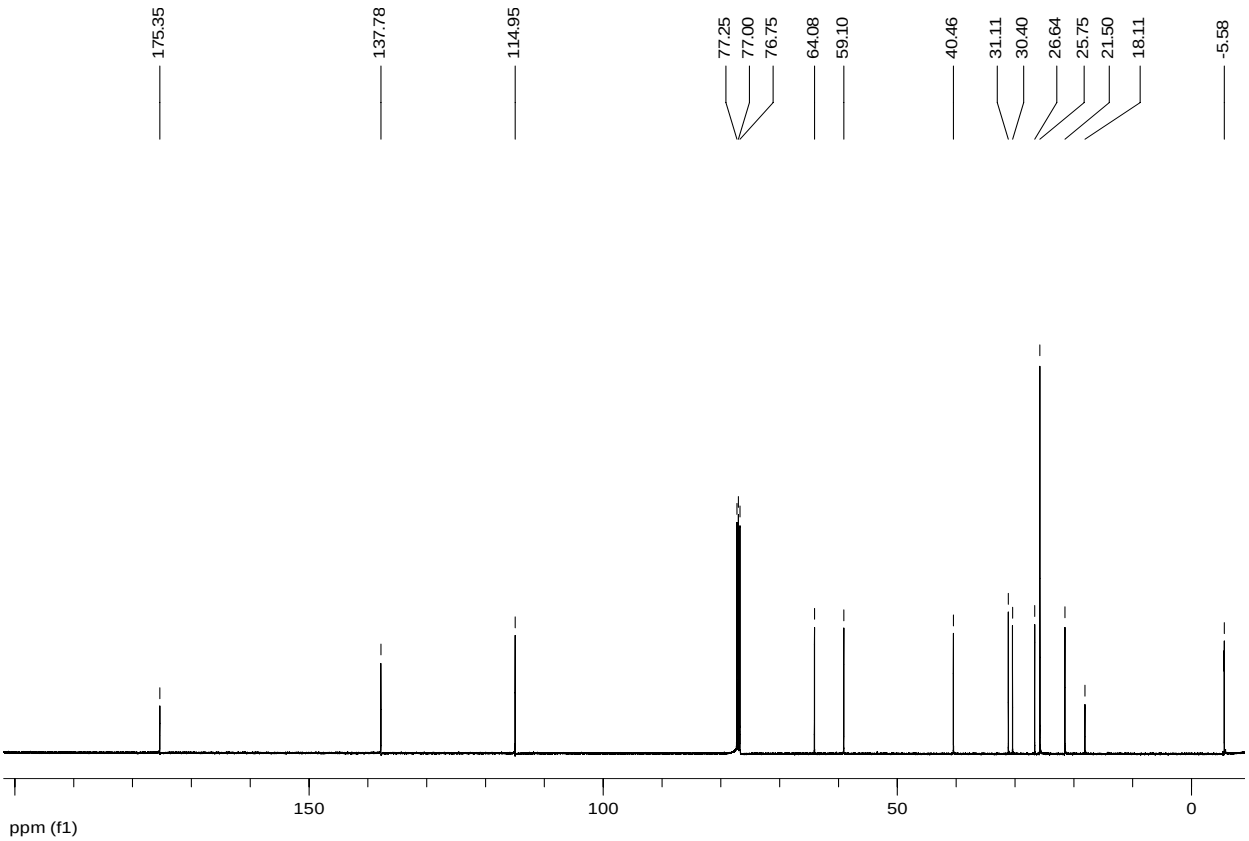

7.0
6.0
5.0
4.0
3.0
2.0
1.0
0.0
ppm (f1)
177.91
66.88
55.76
29.76
25.78
22.72
18.17
-5.46
-5.48

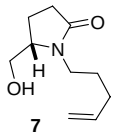

150
100
50
0
ppm (f1)


O
N
H
OTBS

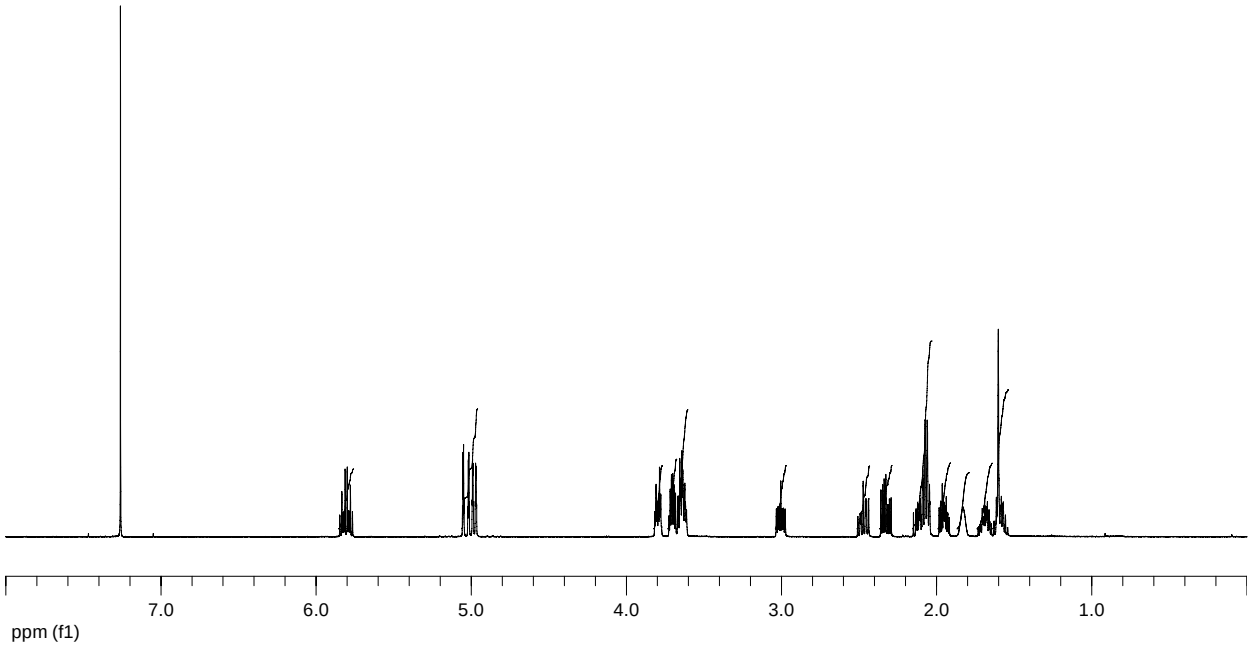
6a

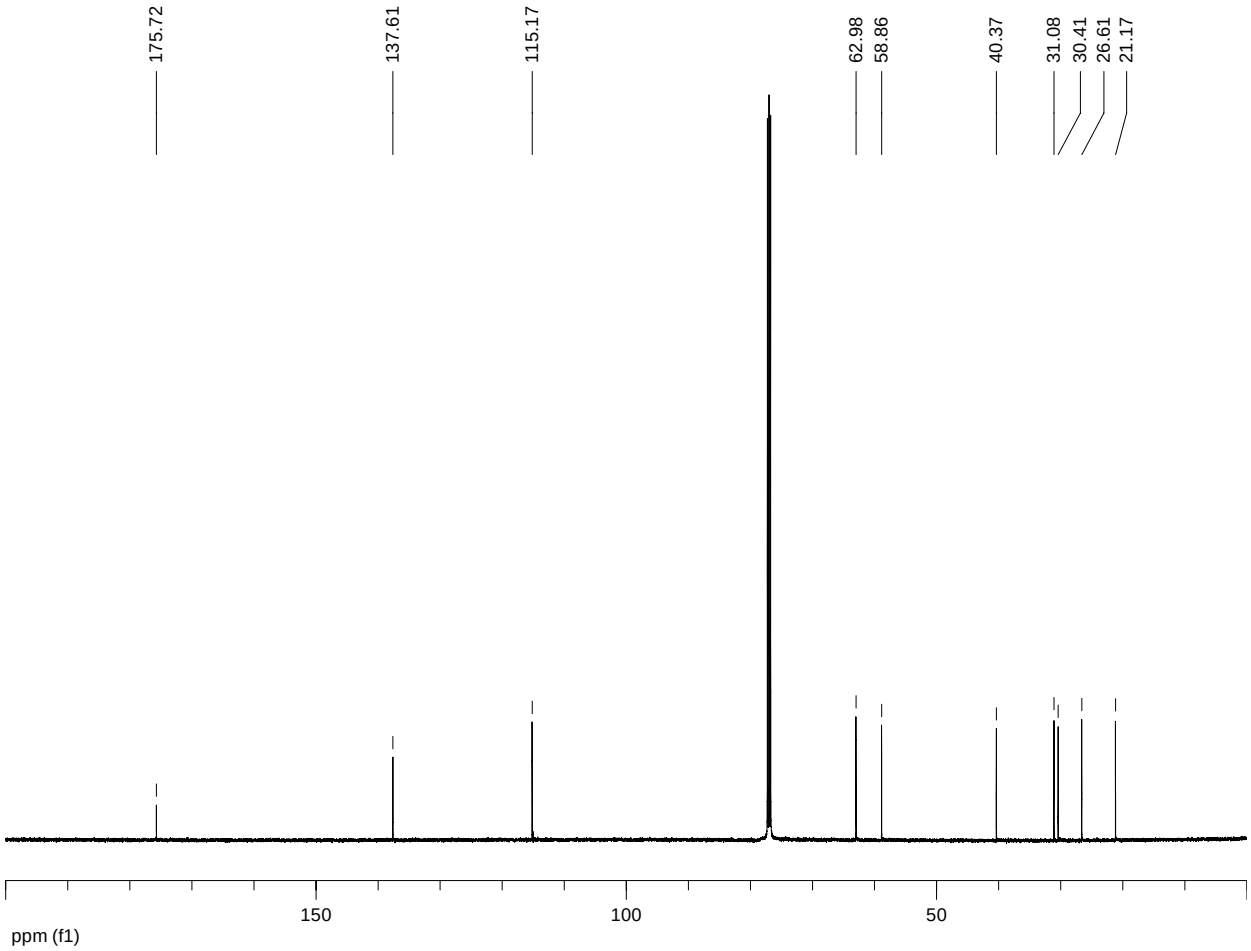

8.0
7.0
6.0
5.0
4.0
3.0
2.0
1.0
0.0
ppm (f1)

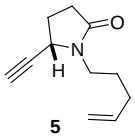

175.35
137.78
114.95
77.25
77.00
76.75
64.08
59.10
40.46
31.11
30.40
26.64
25.75
21.50
18.11
-5.58
150
100
50
0
ppm (f1)


O
N
H
OH

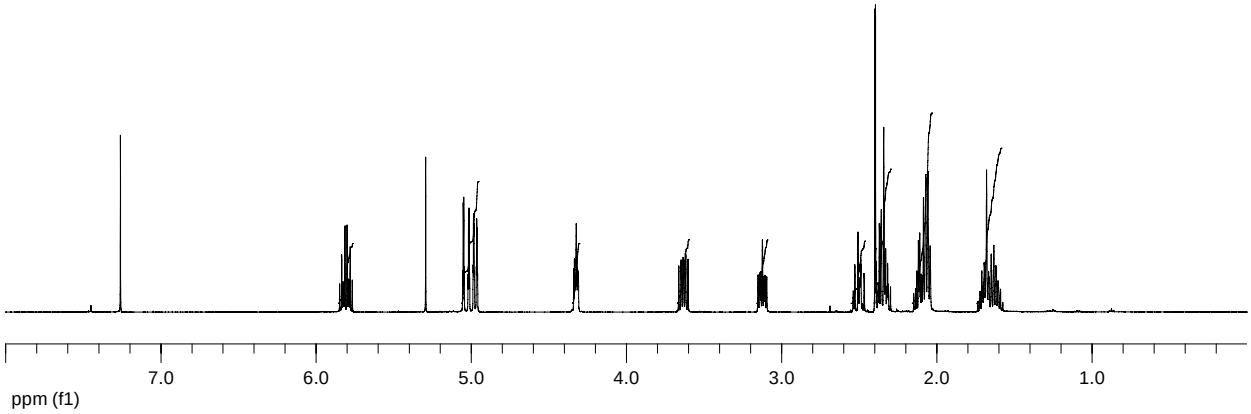
7

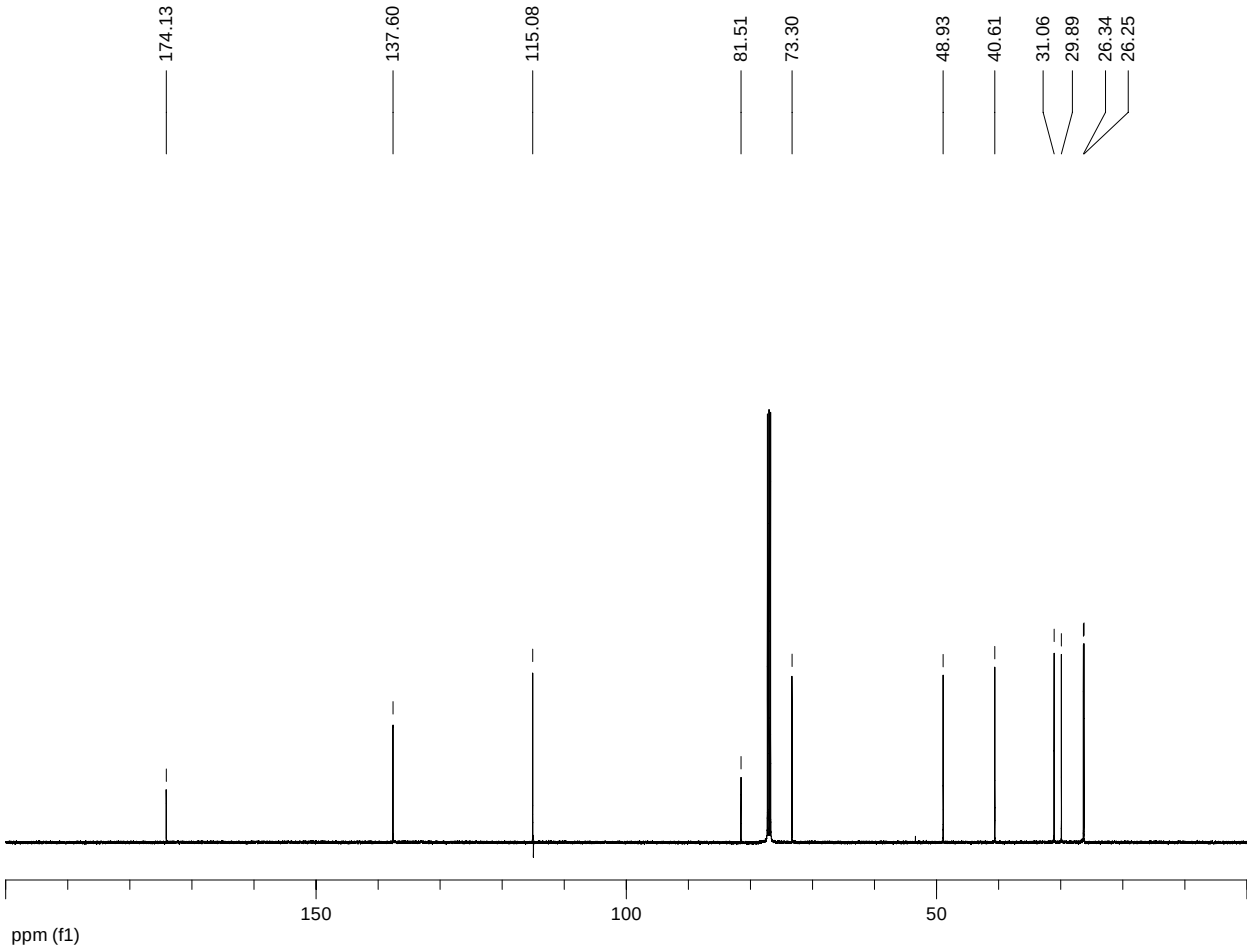

7.0
6.0
5.0
4.0
3.0
2.0
1.0
ppm (f1)

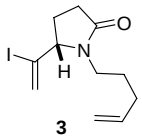

175.72
137.61
115.17
62.98
58.86
40.37
31.08
30.41
26.61
21.17
150
100
50
ppm (f1)


O
N
H

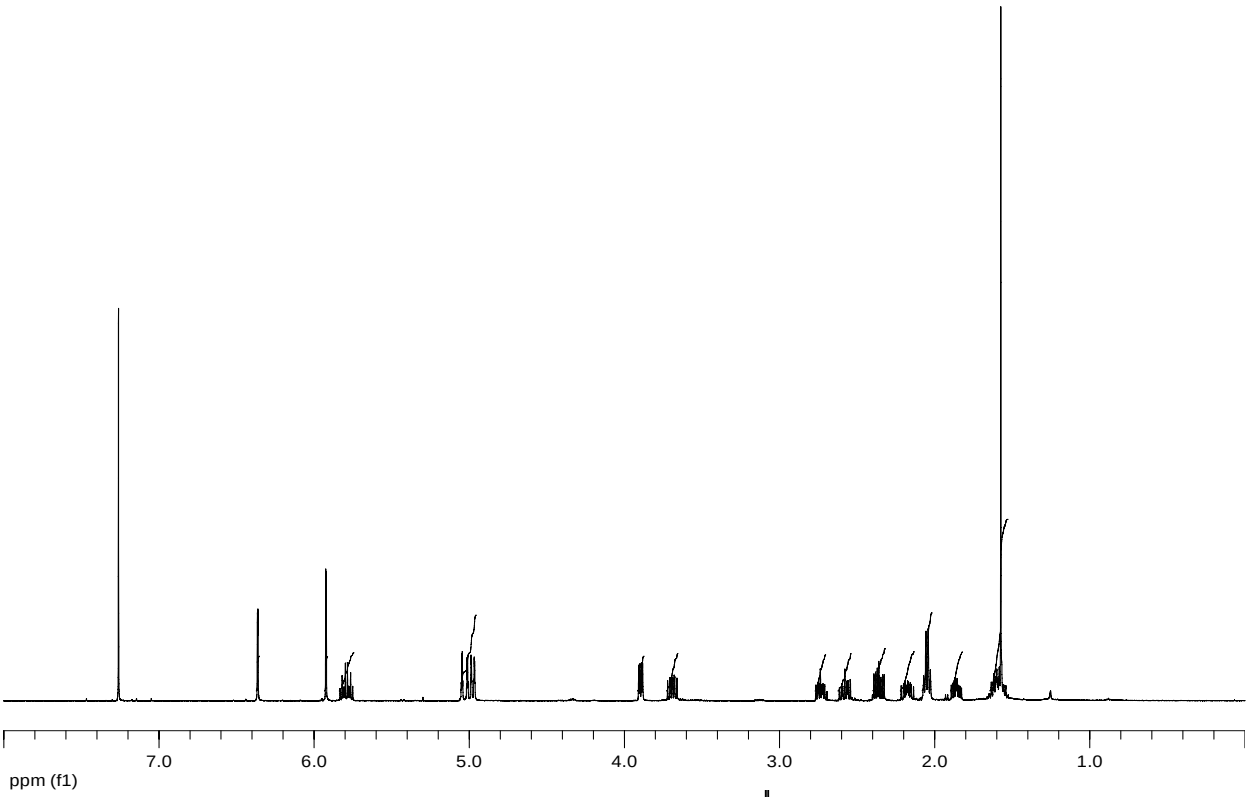
5

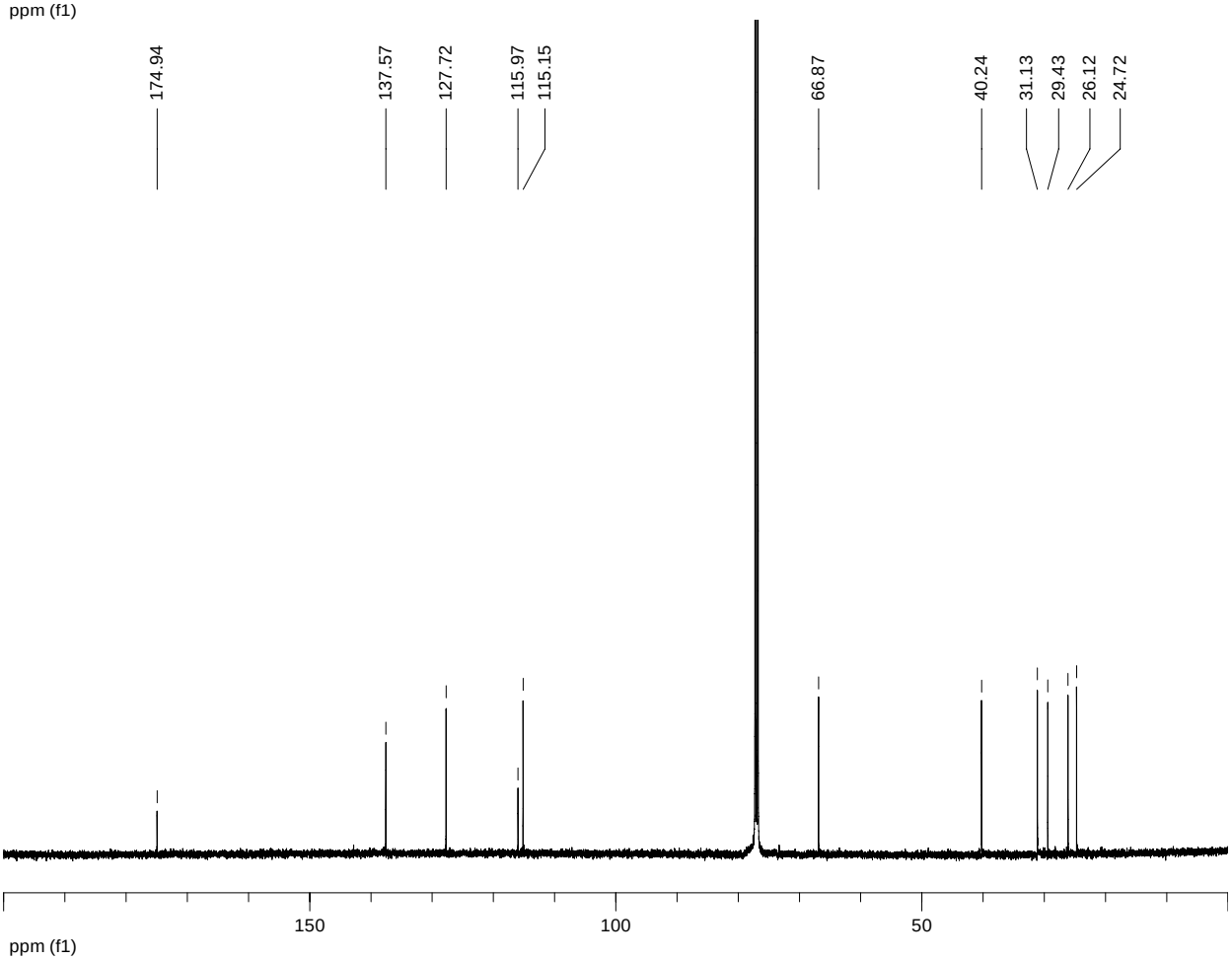

7.0
6.0
5.0
4.0
3.0
2.0
1.0
ppm (f1)

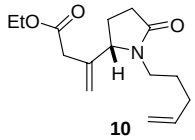

174.13
137.60
115.08
81.51
73.30
48.93
40.61
31.06
29.89
26.34
26.25
150
100
50
ppm (f1)


O
I
N
H

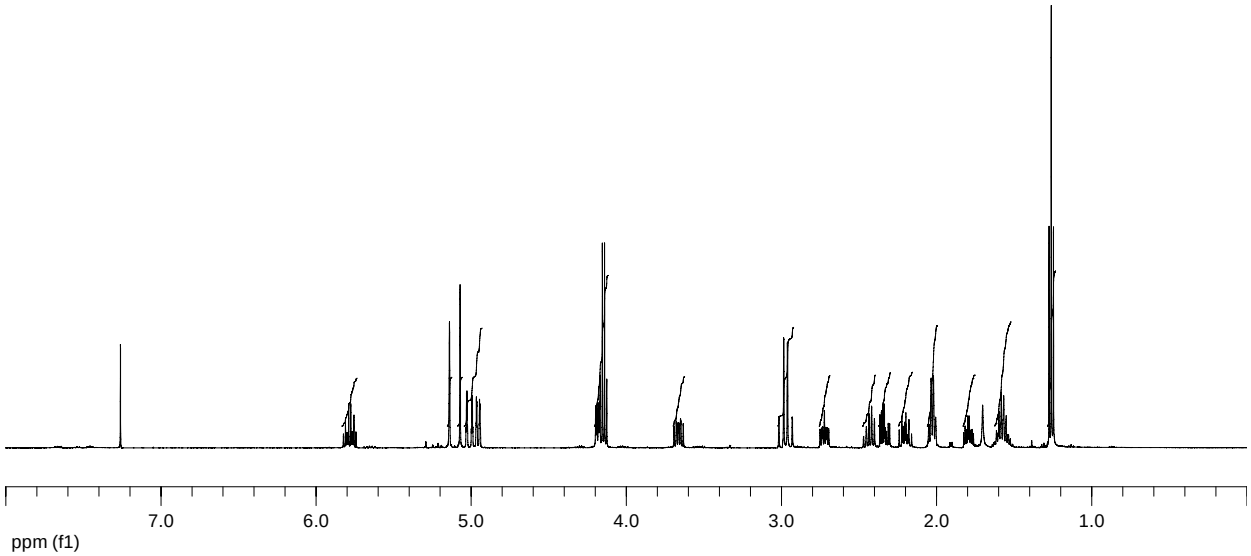
3

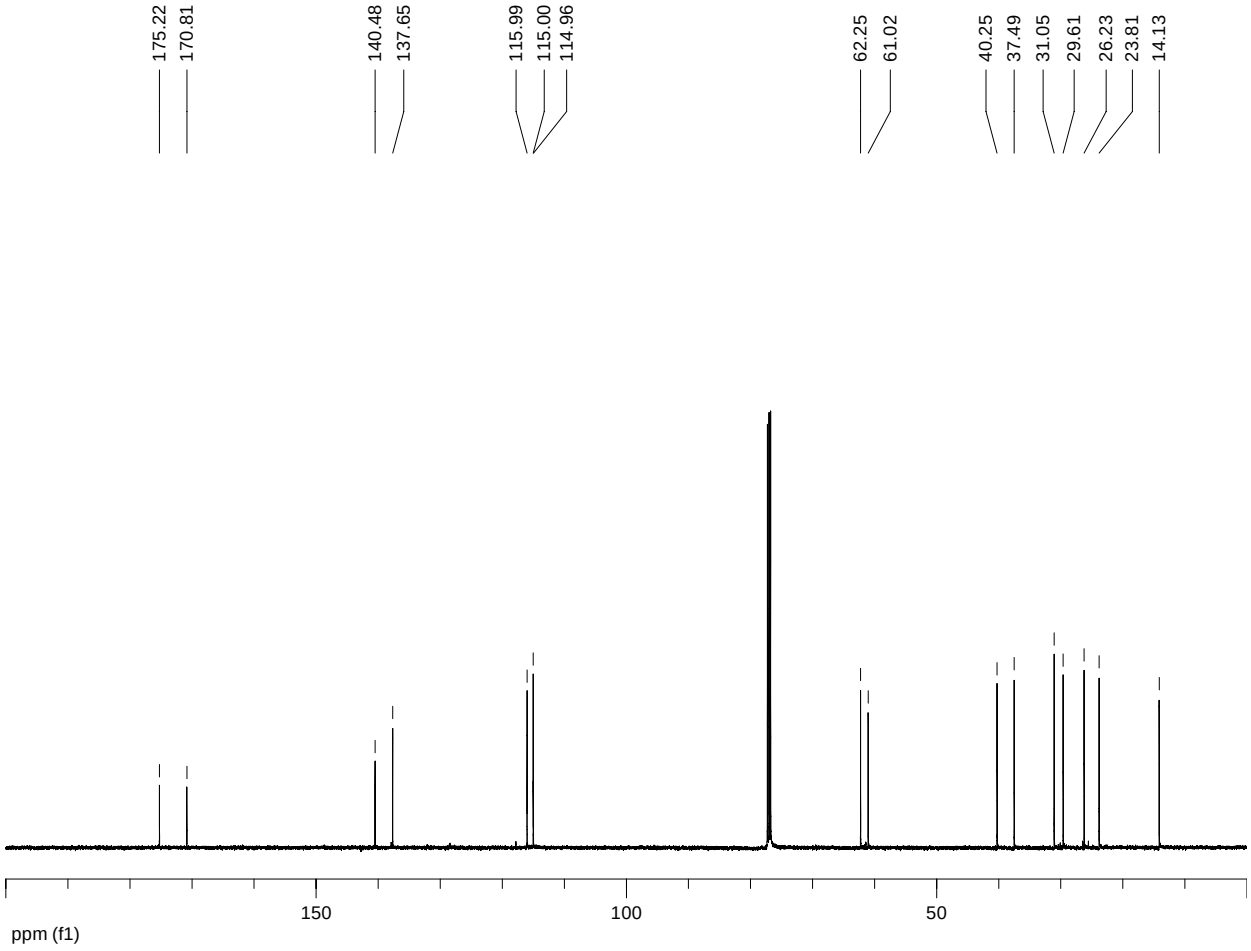

7.0
6.0
5.0
4.0
3.0
2.0
1.0
ppm (f1)

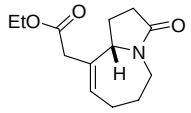

174.94
137.57
127.72
115.97
115.15
66.87
40.24
31.13
29.43
26.12
24.72
150
100
50
ppm (f1)


EtO
O
O
N
H

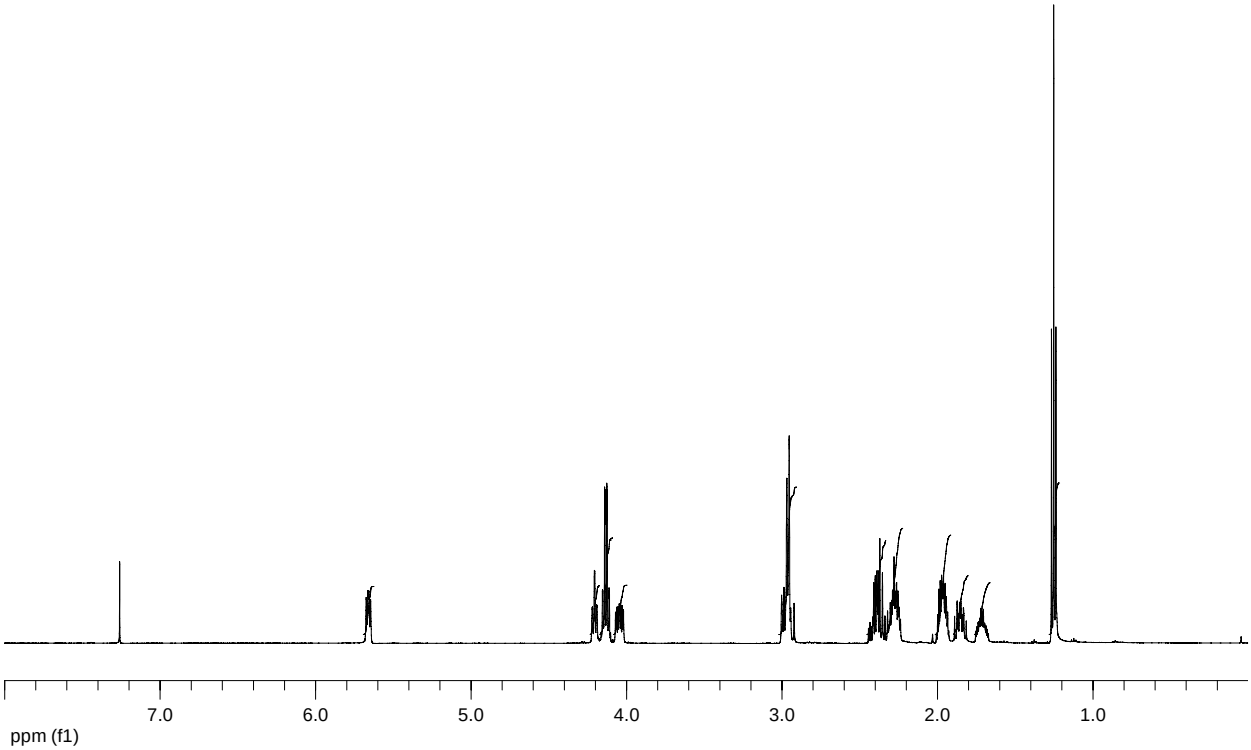
10

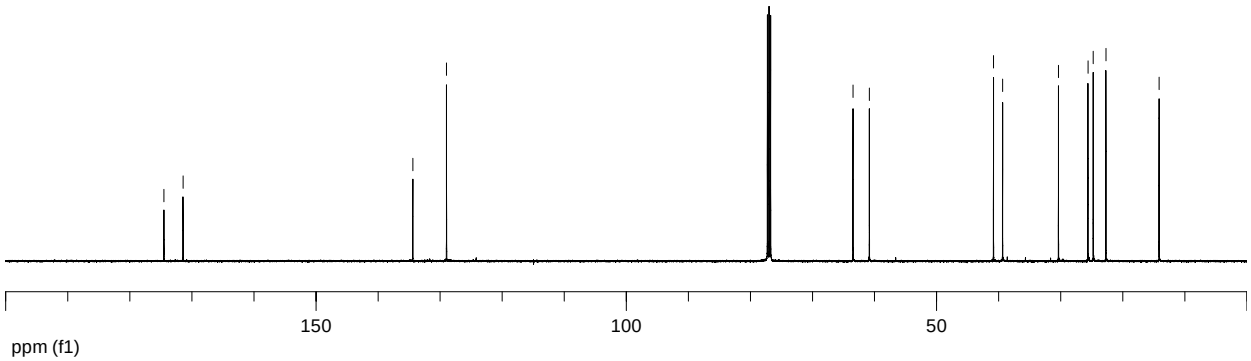

7.0
6.0
5.0
4.0
3.0
2.0
1.0
ppm (f1)

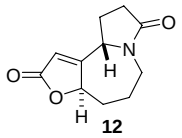

175.22
170.81
140.48
137.65
115.99
115.00
114.96
62.25
61.02
40.25
37.49
31.05
29.61
26.23
23.81
14.13
150
100
50
ppm (f1)


EtO
O
O
N
H

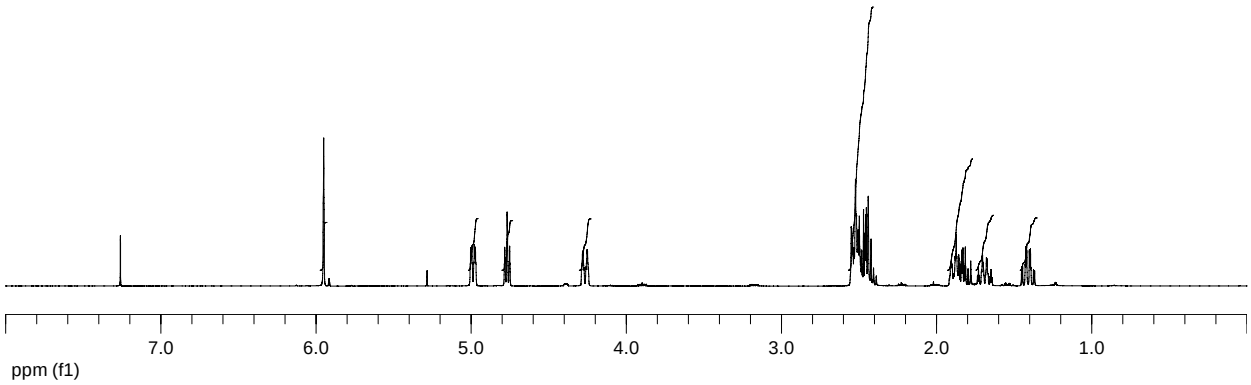
2

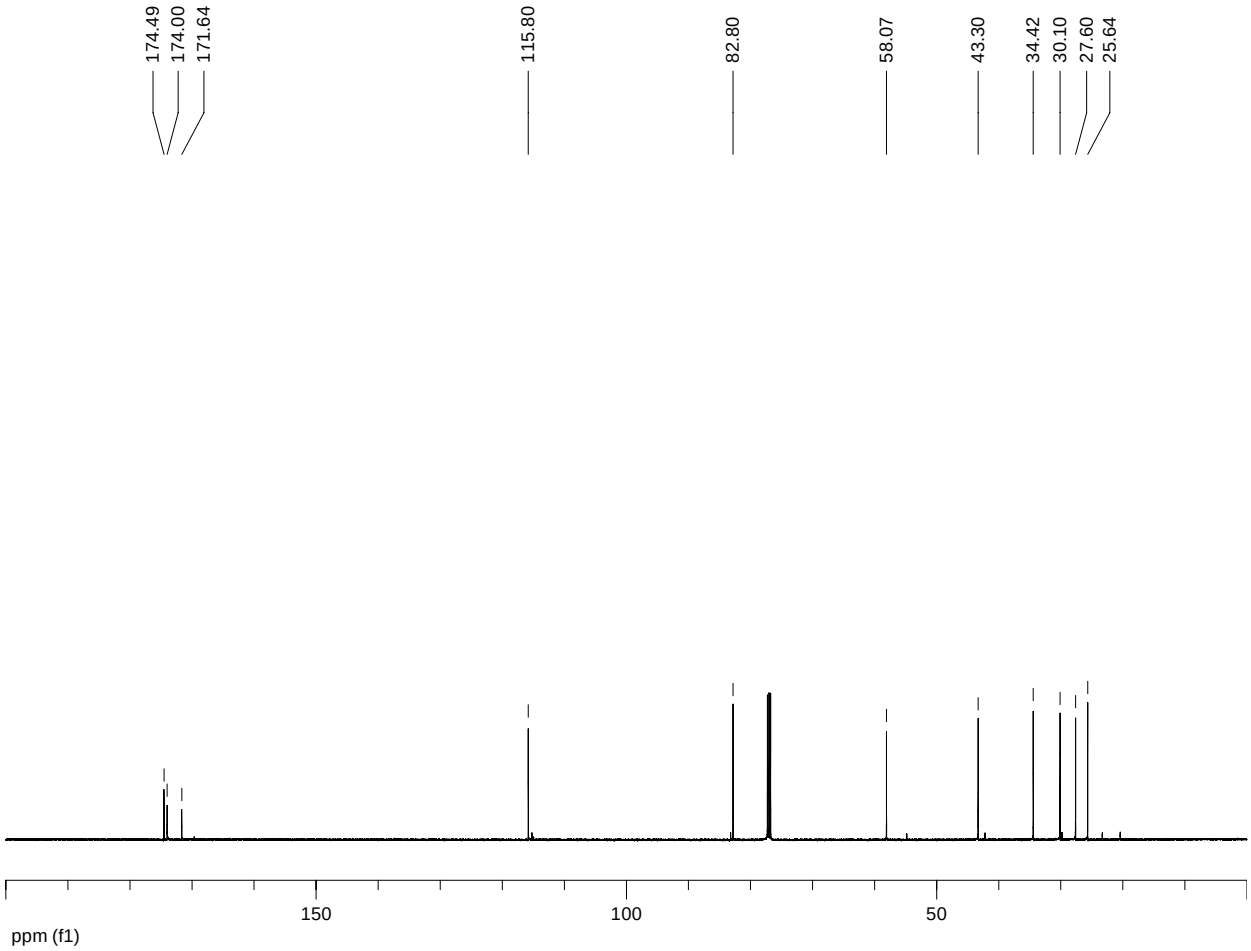

7.0
6.0
5.0
4.0
3.0
2.0
1.0
ppm (f1)
174.50
171.42
134.39
128.96
63.47
60.83
40.82
39.36
30.35
25.61
24.76
22.71
14.15

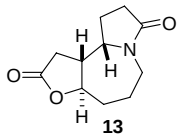

150
100
50
ppm (f1)


O
N
H
O
O
H

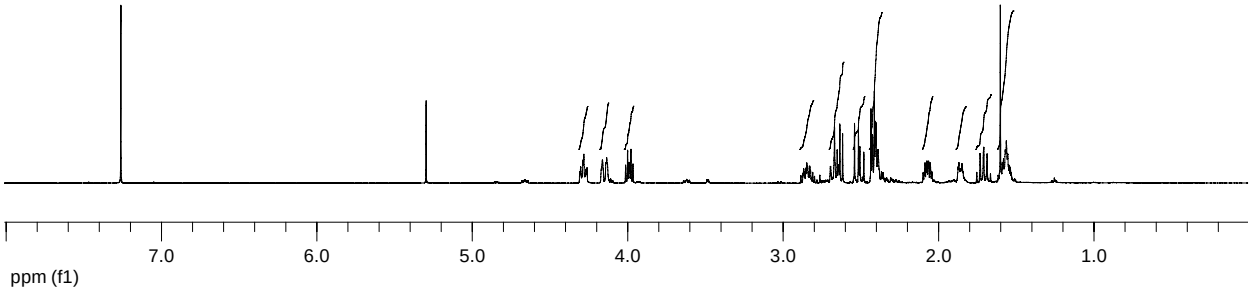
12

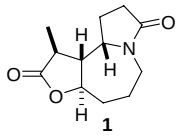

7.0
6.0
5.0
4.0
3.0
2.0
1.0
ppm (f1)


174.49
174.00
171.64
115.80
82.80
58.07
43.30
34.42
30.10
27.60
25.64
150
100
50
ppm (f1)

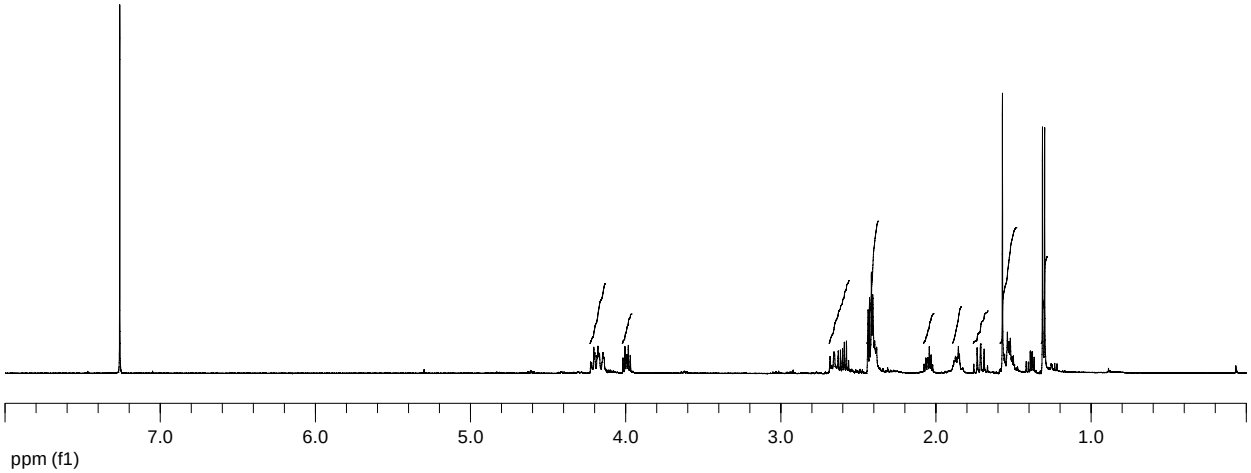


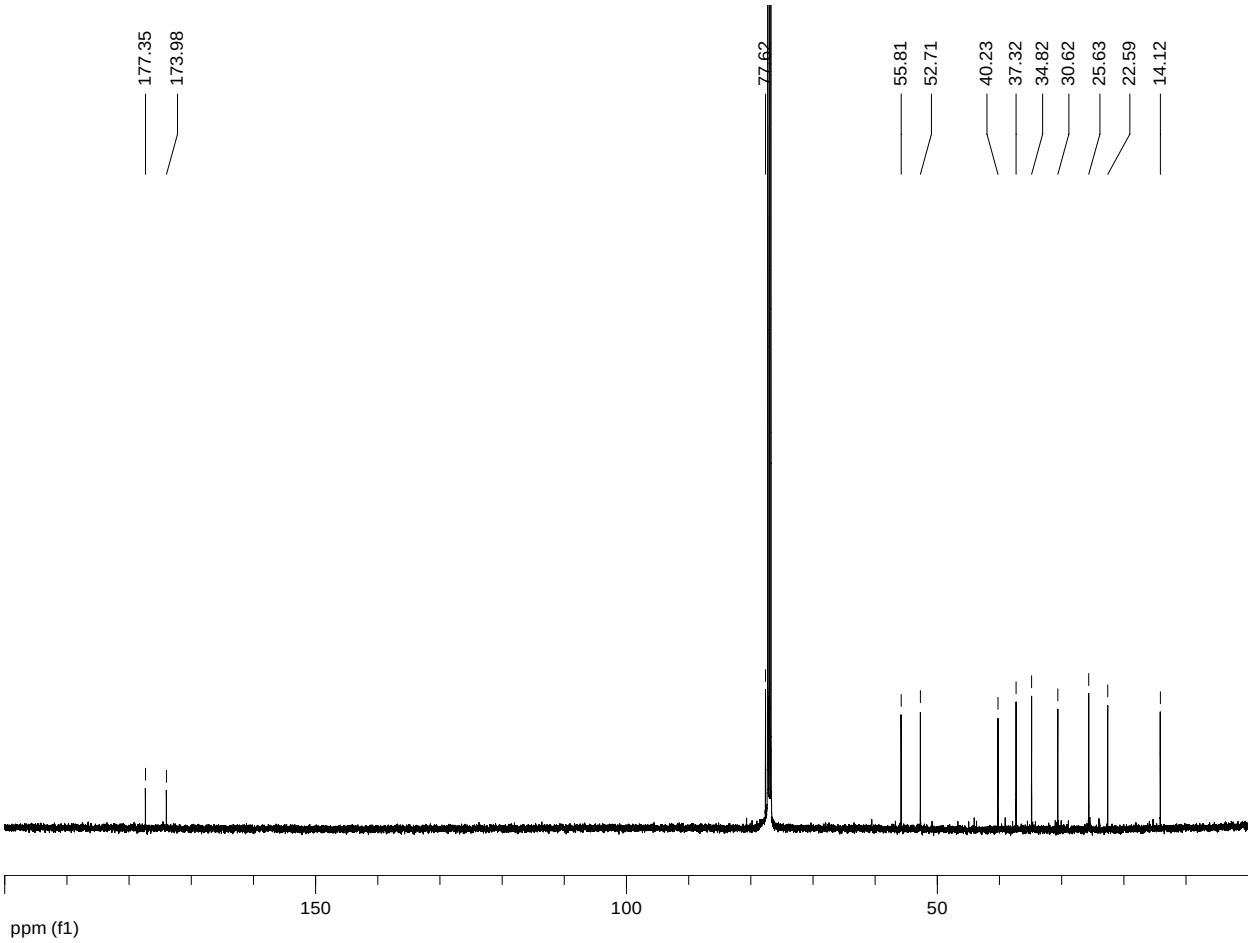
13




H
N
H
O
O
O
H

1


7.0
6.0
5.0
4.0
3.0
2.0
1.0
ppm (f1)


177.35
173.98
77.62
55.81
52.71
40.23
37.32
34.82
30.62
25.63
22.59
14.12
150
100
50
ppm (f1)