SUPPORTING INFORMATION FOR:

Mild Method for the Selective Esterification of Carboxylic Acids Based on the Garegg-Samuelsson Reaction

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Contents

General Details. All the solvents used in these reactions were freshly purified as is described.[1] Products were purified by flash chromatography on Merck silica gel 50. Yields refer to analytically pure samples. NMR spectra were recorded in a NMR 400 MHz spectrometer. ^{1}H NMR: $CDCl_3$ (δ = 7.26 ppm) in the indicated solvent as internal standard in the same solvent. ^{13}C NMR: $CDCl_3$ (δ =77.16 ppm) as internal standard in the same solvent; coupling constants measured in Hz and always given as $J_{H,H}$ coupling constants. ^{31}P NMR spectra: in $CDCl_3$ and CH_2Cl_2 (when is indicated) with 85% H_3PO_4 as external standard. The following known compounds were isolated as pure samples and showed NMR spectra matching those of the reported compounds: **2**,[2] **3**,[3] **4**,[4] **18**,[5] **20**,[6] **26**,[7] **27**,[8] **29**,[9] **30**,[10] **31**,[11] **32**,[12] **33**,[13] **34**,[14] **40**.[15]

Characterization of new compounds

5: (188 mg, 82%), colorless oil; ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.12 (d, *J* = 8.5 Hz, 2H), 6.83 (d, *J* = 8.5 Hz, 2H), 5.89 (m, 1H) 5.25 (m, 2H), 4.57 (d, *J* = 5.7 Hz, 2H), 3.78 (s, 3H), 2.91 (t, *J* = 7.8 Hz, 2H), 2.63 (d, *J* = 8.0 Hz, 2H); ^{13}C NMR (100 MHz, $CDCl_3$; DEPT) δ 172.5 (C), 158.1 (C), 132.5 (CH), 132.2 (C), 129.3 (CH), 118.1 (CH_2), 113.9 (CH), 65.1 (CH_2), 55.2 (CH_3), 36.1 (CH_2), 30.1 (CH_2). HRMS (EI) *m/z* calcd for $C_{13}H_{16}O_3$ [M^+] 220.1099, found 220.1103.

6: (268 mg, 37%), yellow oil; ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.12 (d, *J* = 8.2 Hz, 2H), 6.82 (d, *J* = 8.3 Hz, 2H), 5.05-4.94 (m, 1H), 3.77 (s, 3H), 2.88 (t, *J* = 7.7 Hz, 2H), 2.56 (t, *J* = 7.8 Hz, 2H), 1.20 (d, *J*= 6.3 Hz, 6H); ^{13}C NMR (100 MHz, $CDCl_3$; DEPT) δ 172.7 (C), 158.3 (C), 132.9 (C), 129.5 (CH), 114.1 (CH), 67.8 (CH), 55.4 (CH_3), 36.8 (CH_2), 30.4 (CH_2), 22.0 (CH_3), 22.0 (CH_3); HRMS (EI) *m/z* calcd for $C_{13}H_{18}O_3$ [M^+] 222.1256, found 222.1257.

7: (92 mg, 28%), colorless oil; ^{1}H NMR (400 MHz, $CDCl_3$) δ 8.06 (d, *J* = 7.7 Hz, 1H), 7.87 (d, *J* = 7.5 Hz, 1H), 7.80 (d, *J* = 8.3 Hz, 1H), 7.55-7.48 (m, 3H), 7.44 (t, *J* = 7.5 Hz, 1H), 7.10 (d, *J* = 7.9 Hz, 2H), 6.79 (t, *J* = 6.4 Hz, 2H), 3.78 (s, 3H), 2.92 (t, *J* = 7.7 Hz, 2H), 2.68 (t, *J* = 8.0 Hz, 2H), 1.68 (d, *J* = 6.6 Hz, 3H); ^{13}C NMR (100 MHz, $CDCl_3$; DEPT) δ 172.5 (C), 158.3 (C), 137.7 (C), 134.1 (C), 132.8 (C), 130.5 (C), 129.5 (C), 129.2 (C), 128.7 (C), 126.5 (C), 125.9 (C), 125.6 (C), 123.5 (C),114.1 (C), 69.8 (CH), 55.5 (CH_3), 36.7 (CH_2), 30.4 (CH_2), 22.0 (CH_3); HRMS (FAB) *m/z* calcd for $C_{22}H_{22}O_3Na$ [M^++Na] 357.1467, found 357.1461.

9: (289 mg, 99%), colorless oil; ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.12 (d, *J* = 8.3 Hz, 2H), 6.83 (d, *J* = 6.3 Hz, 2H), 3.78 (s, 3H), 3.20 (dd, *J* = 12.8, 6.2 Hz, 2H), 2.91 (t, *J* = 7.6 Hz, 2H), 2.43 (t, *J* = 7.5 Hz, 2H), 1.42-1.16 (m, 12H), 0.89 (t, *J* = 6.6 Hz, 3H); ^{13}C NMR (100 MHz, $CDCl_3$; DEPT) δ 173.0 (C), 158.2 (C), 133.3 (C), 129.6 (CH), 114.2 (CH), 55.6 (CH_3), 39.9 (CH_2), 39.2 (CH_2), 32.14 (CH_2), 31.3 (CH_2), 29.9

(CH_2), 29.5 (CH_2), 27.2 (CH_2), 22.9 (CH_3); HRMS (EI) *m/z* calcd for $C_{18}H_{29}O_2N$ [M^+] 291.2198, found 291.2199.

10: (230 mg, 60%), colorless oil; ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.59 (d, *J* = 8.6 Hz, 2H), 7.21 (d, *J* = 8.6 Hz, 2H), 7.15 (d, *J* = 8.2 Hz, 2H), 6.84 (d, *J* = 8.2 Hz, 2H), 3.79 (s, 3H), 2.91 (t, *J* = 6.9 Hz, 1H), 2.61 (t, *J* = 7.8 Hz, 2H); ^{13}C NMR (100 MHz, $CDCl_3$; DEPT) δ 170.7 (C), 158.2 (C), 137.9 (CH), 137.5 (C), 132.4 (C), 129.3 (CH), 121.6 (CH), 114.1 (CH), 55.3 (CH_3), 39.8 (CH_2), 30.6 (CH_2); HRMS (EI) *m/z* calcd for $C_{16}H_{16}O_2NI$ [M^+] 381.0226, found 381.0227.

11: (90 mg, 32%), colorless oil; ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.13 (d, *J* = 7.8 Hz, 2H), 6.83 (d, *J* = 7.8 Hz, 2H), 3.78 (s, 3H), 3.30 (t, *J* = 7.3 Hz, 2H), 3.13 (dd, *J* = 8.5, 7.3 Hz, 2H), 2.92 (t, *J* = 6.4 Hz, 2H), 2.55 (t, *J* = 6.5 Hz, 2H), 1.65 (sa, 1H), 1.27 (dt, *J* = 15.9, 7.9 Hz, 4H), 0.91 (t, *J* = 7 Hz, 3H), 0.90 (t, *J*= 7 Hz, 3H); ^{13}C NMR (100 MHz, $CDCl_3$; DEPT) δ 170.3 (C), 158.2 (C), 133.9 (C), 114.1 (C), 55.4 (CH_3), 46.0 (CH_2), 35.6 (CH_2), 31.5 (CH_2), 31.1 (CH_2), 30.2 (CH_2), 20.5 (CH_2), 20.3 (CH_2), 14.1(CH_3), 14.0 (CH_3); HRMS (EI) *m/z* calcd for $C_{18}H_{29}O_2N$ [M^+] 291.2198, found 291.2188.

12: (267 mg, 94%), colorless oil; ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.33-7.27 (m, 1H), 7.26 (s, 2H), 7.18 (d, *J* = 7.2 Hz, 2H), 7.09 (d, *J* = 8.0 Hz, 2H), 6.80 (d, *J* = 9.2 Hz, 2H), 5.09 (t, *J* = 7.0 Hz, 1H), 3.78 (s, 3H), 2.90 (t, *J* = 8.0 Hz, 2H), 2.44 (t, *J* = 8.3 Hz, 2H), 1.41 (d, *J* = 7.0 Hz, 2H); ^{13}C NMR (100 MHz, $CDCl_3$; DEPT) δ 171.4 (C), 158.3 (C), 143.3 (C), 133.03 (C), 129.5 (CH), 128.8 (CH), 127.4 (CH), 126.3 (CH), 114.1 (CH), 55.4 (CH_3), 48.8 (CH), 39.0 (CH_2), 31.1 (CH_2), 21.8 (CH_3); HRMS (EI) *m/z* calcd for $C_{18}H_{21}O_2N$ [M^+] 283.1572, found 283.1581.

14: (109 mg, 99%), yellow oil; ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.12 (d, *J* = 8.6 Hz, 2H), 6.83 (t, *J* = 5.7 Hz, 2H), 3.79 (s, 3H), 2.89 (t, *J* = 7.7 Hz, 2H), 2.59 (t, *J* = 7.7 Hz, 2H), 1.79 – 1.51 (m, 2H), 1.51 – 1.40 (m, 4H), 1.35 (d, *J* = 8.5 Hz, 1H), 1.26 (s, 4H); ^{13}C NMR (100 MHz, $CDCl_3$; DEPT) δ 173.5 (C), 158.6 (C), 133.1 (C), 129.7 (CH), 114.4 (CH), 68.4 (CH), 64.8 (CH_2), 55.7 (CH_3), 39.3 (CH_2), 36.7 (CH_2), 30.6 (CH_2), 29.1 (CH_2), 24.0 (CH_3), 22.5 (CH_2); HRMS (EI) *m/z* calcd for $C_{16}H_{24}O_4$ [M^+] 280.1675, found 280.1687.

28: (162 mg, 89%), colorless oil; ^{1}H NMR (400 MHz, $CDCl_3$) δ 3.64 (s, 3H), 2.40 (t, *J* = 7.4 Hz, 2H), 2.18 (t, *J* = 6.7 Hz, 2H), 2.10 (t, *J* = 5.9 Hz, 2H), 1.84–1.69 (m, 2H), 1.48–1.29 (m, 4H), 0.87 (t, *J* = 7.0 Hz, 3H); ^{13}C NMR (100 MHz, $CDCl_3$; DEPT) δ 173.7 (C), 81.2 (C), 76.7 (C), 55.4 (CH_3), 51.5 (CH_2), 32.8 (CH_2), 31.1 (CH_2), 24.3 (CH_2), 21.9 (CH_2), 18.4 (CH_3), 13.6 (CH_2). HRMS (EI) *m/z* calcd for $C_{11}H_{19}O_2$ [M^++H] 183.1385, found 183.1379.

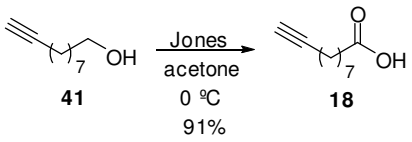
37: (252 mg, 89%), colorless oil; ^{1}H NMR (400 MHz, $CDCl_3$) δ 5.88-5.71 (m, 1H), 5.44-5.28 (m, 2H), 3.24 (dd, *J* = 13.5, 6.1 Hz, 2H), 2.15 (t, *J* = 7.6 Hz, 1H), 2.08–1.99 (m, 2H), 1.67–1.56 (m, 1H), 1.53–1.43 (m, 1H), 1.41–1.34 (m, 1H), 0.88 (t, *J* = 6.4 Hz, 3H); ^{13}C NMR (100 MHz, $CDCl_3$; DEPT) δ 173.3 (C), 139.3 (CH), 114.4 (CH_2), 39.8 (CH_2), 37.1 (CH_2), 34.0 (CH_2), 32.1 (CH_2), 30.0 (CH_2), 29.6 (CH_2), 29.5

(CH_2), 29.5 (CH_2), 29.2 (CH_2), 29.1 (CH_2), 27.2 (CH_2), 26.1 (CH_2), 22.9 (CH_2), 14.3 (CH_3); HRMS (EI) *m/z* calcd for $C_{18}H_{35}ON$ [M^+] 281.2719, found 281.2726.

38: (270 mg, 85%), colorless oil; ^{1}H NMR (400 MHz, $CDCl_3$) δ 3.53 (t, *J* = 6.5 Hz, 2H), 3.24 (dd, *J* = 12.5, 6.1 Hz, 2H), 2.15 (t, *J* = 7.3 Hz, 2H), 1.83–1.71 (m, 2H), 1.60 (dd, *J* = 13.8, 1.9 Hz, 3H), 1.54–1.45 (m, 2H), 1.45–1.38 (m, 2H), 1.30 (s, 16H), 0.88 (t, *J* = 6.1 Hz, 3H); ^{13}C NMR (100 MHz, $CDCl_3$; DEPT) δ 173.3 (C) 45.5 (CH_2), 39.8 (CH_2), 37.2 (CH_2), 32.9 (CH_2), 32.1 (CH_2), 30.0 (CH_2), 29.6 (CH_2), 29.6 (CH_2), 29.58 (CH_2), 29.57 (CH_2), 29.5 (CH_2), 29.1 (CH_2), 27.3 (CH_2), 27.1 (CH_2), 26.1 (CH_2), 22.96 (CH_2), 14.4 (CH_3); HRMS (EI) *m/z* calcd for $C_{18}H_{36}ONCl$ [M^+] 317.2485, found 317.2481.

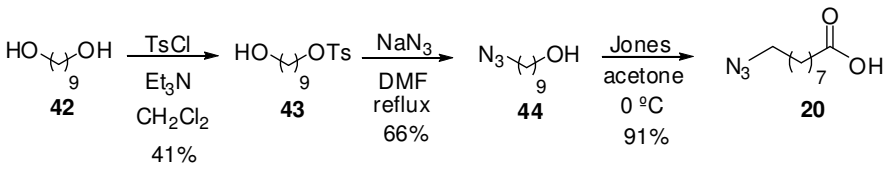
39: (139 mg, 88%), colorless oil; ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.60 (d, *J* = 15.5 Hz, 1H), 7.46-7.39 (m, 2H), 7.29-7.23 (C), 6.52 (d, *J* = 15.5 Hz, 2H), 3.35 (dd, *J* = 12.5, 6.1 Hz, 2H), 1.69-1.44 (m, 2H), 1.41-1.1 (m, 12H), 0.83 (t, *J* = 6.0 Hz, 3H); ^{13}C NMR (100 MHz, $CDCl_3$; DEPT) δ 166.1 (C), 140.7 (CH), 135.2 (C), 129.7 (CH), 129.0 (CH), 128.0 (CH), 121.5 (CH), 40.1 (CH_2), 32.1 (CH_2), 30.0 (CH_2), 29.6 (CH_2), 29.5 (CH_2), 27.3 (CH_2), 22.9 (CH_2), 14.3 (CH_3); HRMS (FAB) *m/z* calcd for $C_{17}H_{25}ON$ [M^+] 259.1936, found 259.1938.

Synthesis of acid 18



Synthesis of acid 18: To a solution of alcohol **41** (300 mg, 1.75 mmol) in acetone (15 mL) at 0 ºC, Jones reagent (1 mL) was added dropwise and the new solution was stirred for 2 h at 0º C. Then, the solvent was removed, the residue was dissolved in Et_2O (50 mL) and washed with brine. The organic layer was dried (anhyd Na_2SO_4) and the solvent removed. The residue was submitted to flash chromatography (EtOAc/Hexane, 4/6) to give alcohol **18** (294 mg, 91%) as a colorless oil.

Synthesis of acid 20



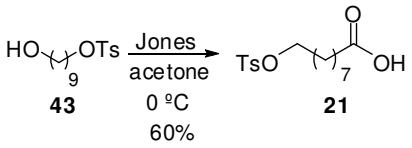
Synthesis of tosylate 43: To a solution of diol **42** (500 mg, 3.1 mmol) in CH_2Cl_2 (30 mL), TsCl (475 mg, 2.5 mmol) and Et_3N (315 mg, 3.1 mmol) were added, and the mixture was stirred at room temperature for 6 h. Then, the solvent was removed. The residue was submitted to flash chromatography (EtOAc/Hexane, 35/65) to give alcohol **43** (397 mg, 41%) as a colorless oil. Its ^{1}H and ^{13}C NMR spectra matched with previously described.[16]

Synthesis of azide 44: To a solution of tosylate **43** (197 mg, 0.63 mmol) in DMF (25 mL), NaN_3 (49 mg, 0.75 mmol) was added and the mixture was heated at reflux for 2 h. Then, AcOEt (40 mL) was added and

the organic layer was washed with brine, dried (anhyd Na_2SO_4) and the solvent removed. The residue was submitted to flash chromatography (EtOAc/Hexane, 1/1) to give azide **44** (83 mg, 66%) as a colorless oil. Its ^1H and ^{13}C NMR spectra matched with previously described.[17]

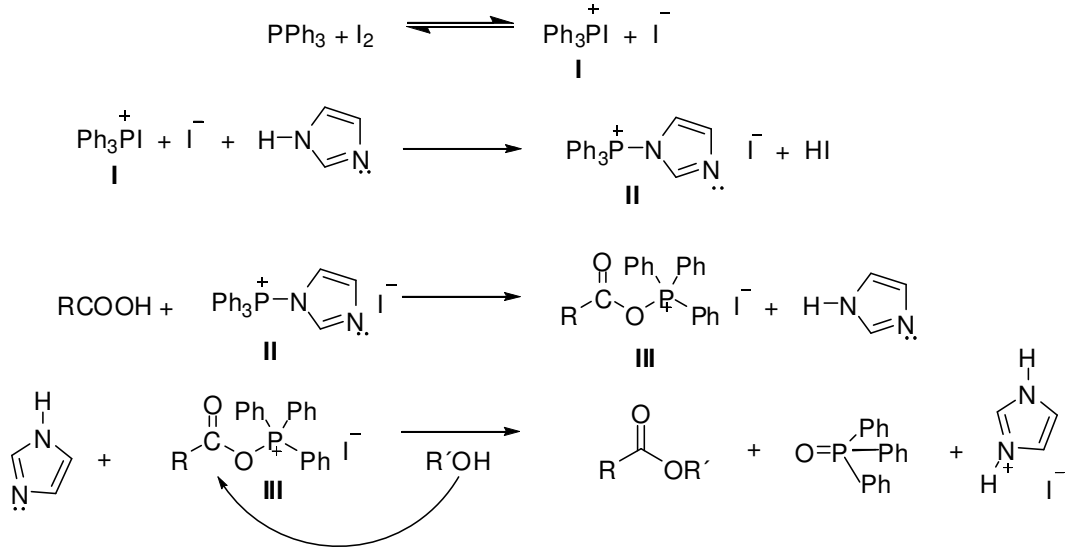
Synthesis of acid 20: To a solution of azide **44** (83 mg, 0.41 mmol) in acetone (15 mL) at 0 ºC, Jones reagent (0.4 mL) was added dropwise and the new solution was stirred for 2 h at 0º C. Then, the solvent was removed, the residue was dissolved in Et_2O (50 mL) and washed with brine. The organic layer was dried (anhyd Na_2SO_4) and the solvent removed. The residue was submitted to flash chromatography (EtOAc/Hexane, 4/6) to give alcohol **20** (75 mg, 77%) as a colorless oil.

Synthesis of acid 21

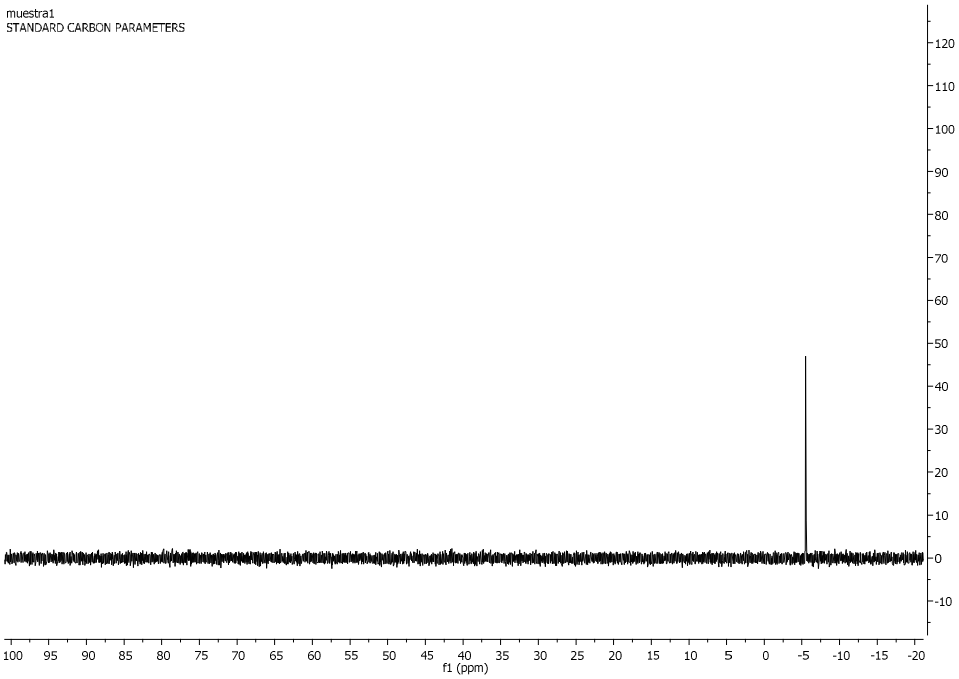


Synthesis of acid 21: To a solution of tosylate **43** (180 mg, 0.57 mmol) in acetone (15 mL) at 0 ºC, Jones reagent (1 mL) was added dropwise and the new solution was stirred for 2 h at 0º C. Then, the solvent was removed, the residue was dissolved in Et_2O (50 mL) and washed with brine. The organic layer was dried (anhyd Na_2SO_4) and the solvent removed. The residue was submitted to flash chromatography (EtOAc/Hexane, 4/6) to give alcohol **21** (116 mg, 60%) as a colorless oil; ^1H NMR (400 MHz, $CDCl_3$) δ 7.77 (d, *J* = 8.0 Hz, 2H), 7.32 (d, *J* = 8.0 Hz, 2H), 4.00 (t, *J* = 6.5 Hz, 2H), 2.43 (s, 3H), 2.31 (t, *J* = 7.5 Hz, 2H), 1.70–1.44 (m, 4H), 1.44–1.05 (m, 10H); ^{13}C NMR (100 MHz, $CDCl_3$; DEPT) δ 179.2 (C), 144.6 (C), 133.4 (C), 129.8 (CH), 127.9 (CH), 70.6 (CH_2), 53.4 (CH_2), 33.9 (CH_2), 29.0 (CH_2), 25.3 (CH_2), 24.6 (CH_2), 21.6 (CH_3); HRMS (FAB) *m/z* calcd for $C_{17}H_{26}O_5SNa$ [M^++Na] 365.1399, found 365.1410.

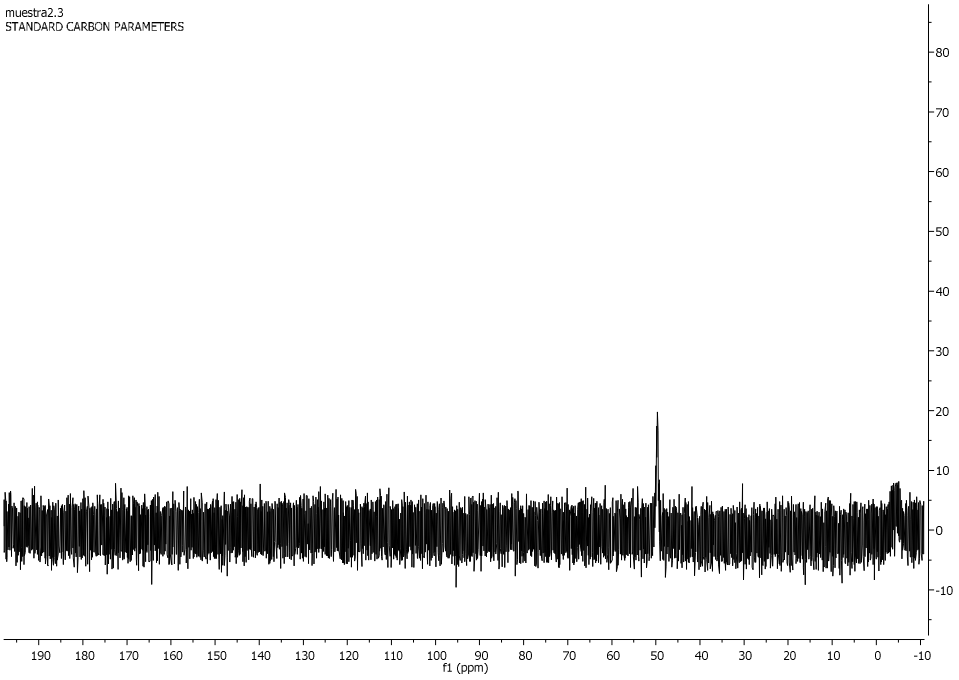
^{31}P NMR of the proposed intermediates

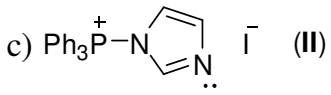


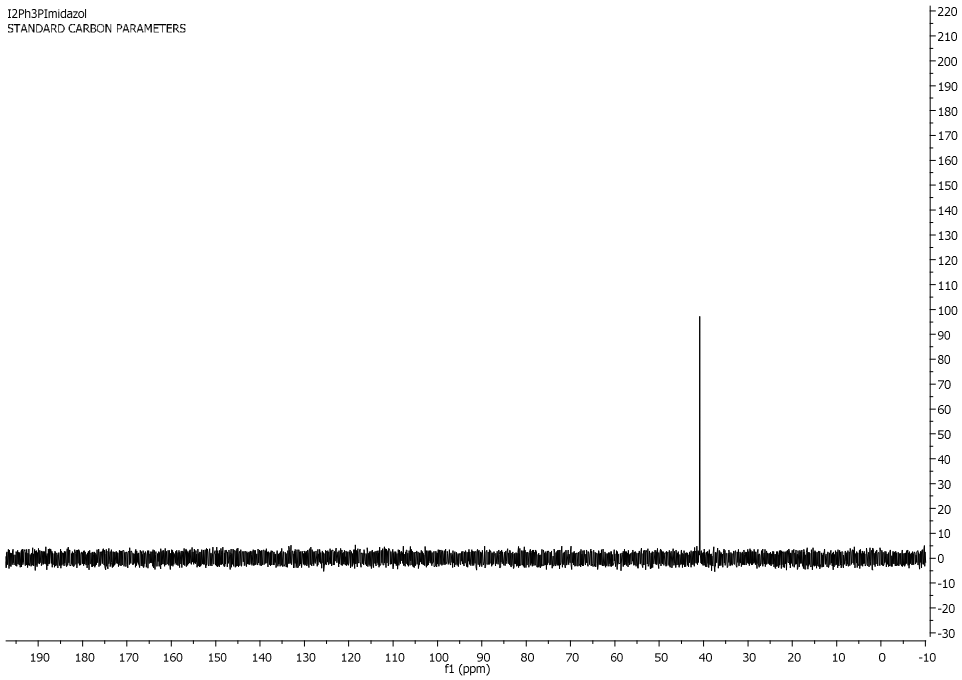
a) Ph_3P



b) $Ph_3P^+I\ I^-$ (**I**)



c)  (**II**)



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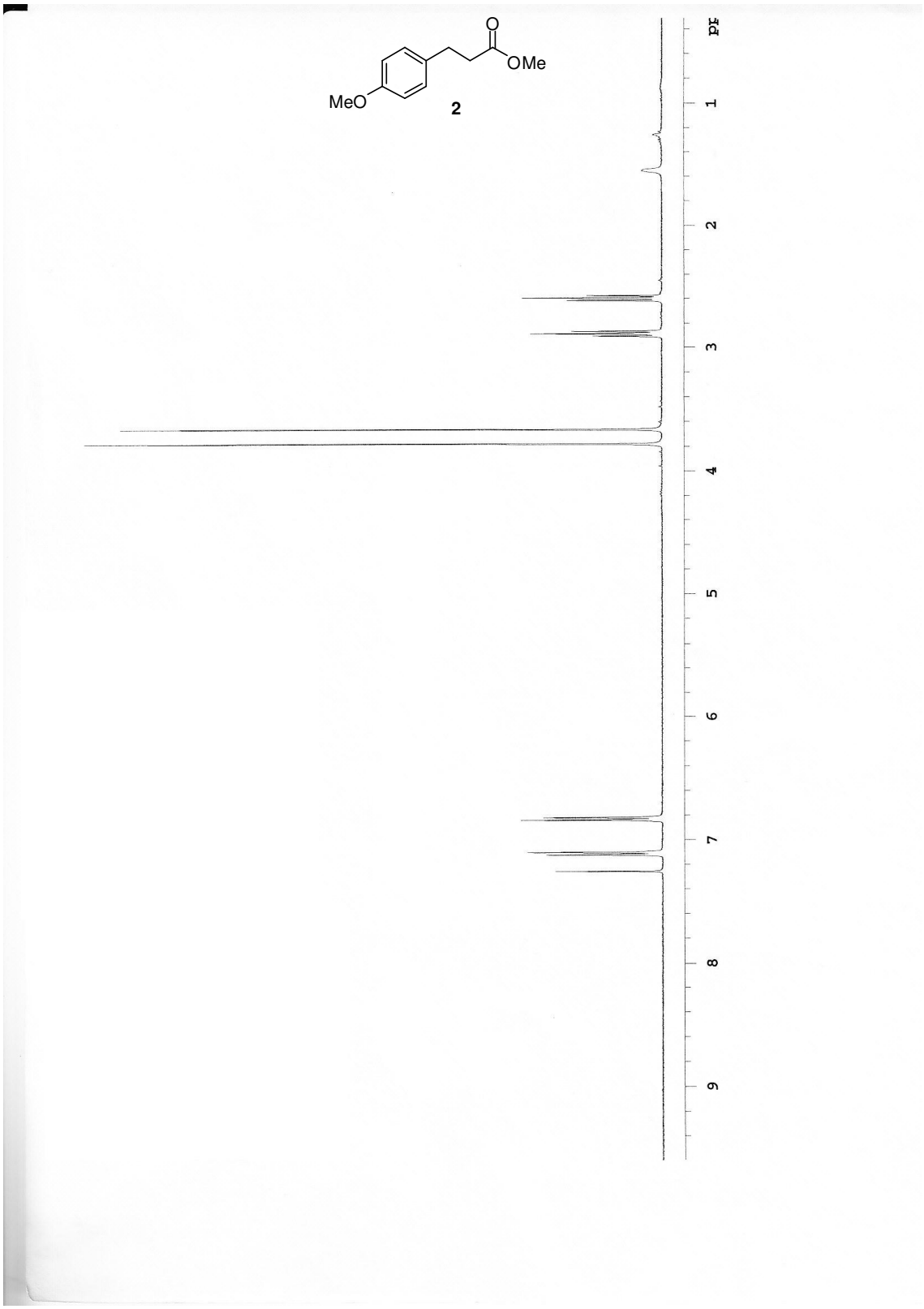
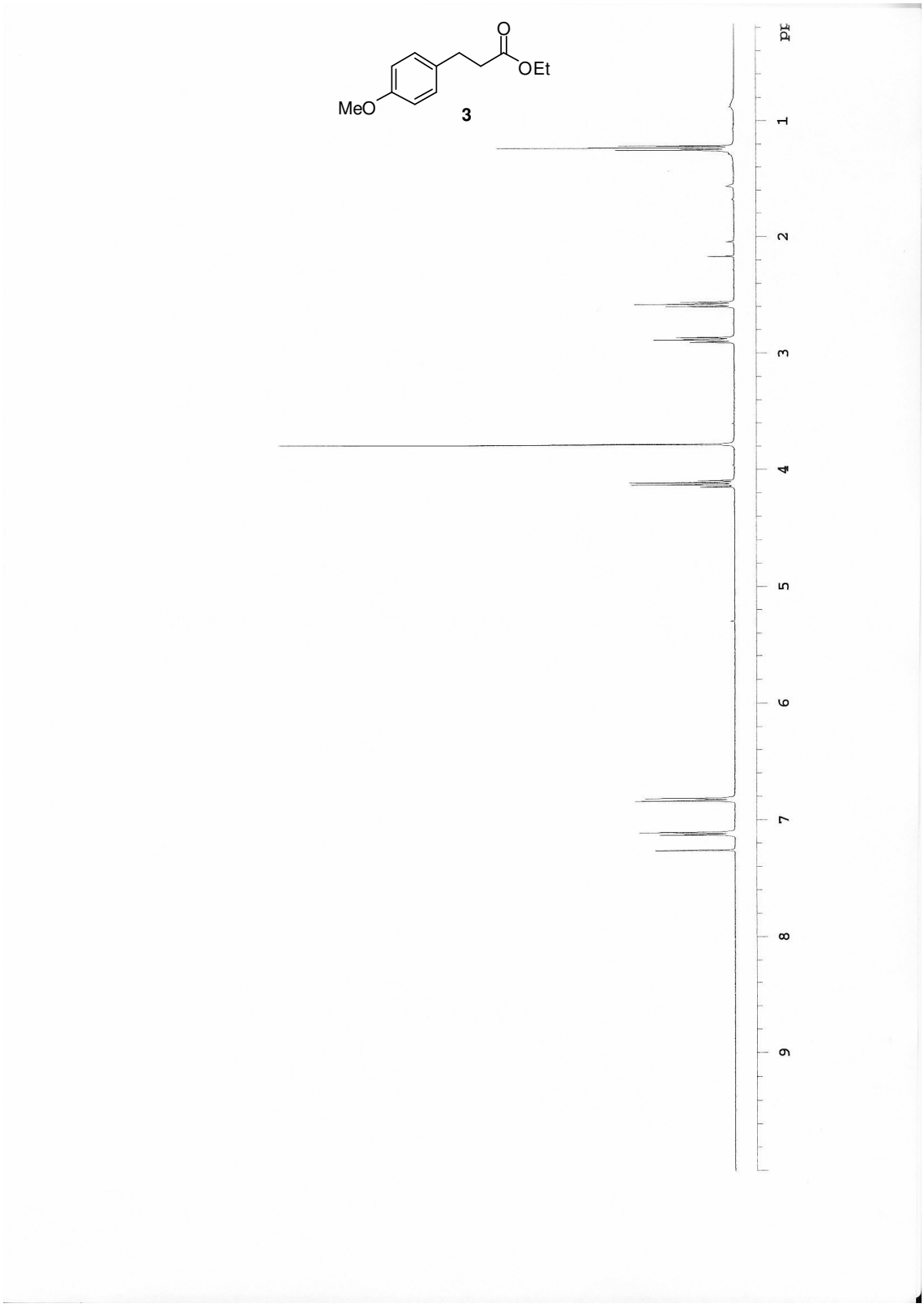
^{1}H and ^{13}C NMR SPECTRA FOR KNOWN AND NEW COMPOUNDS

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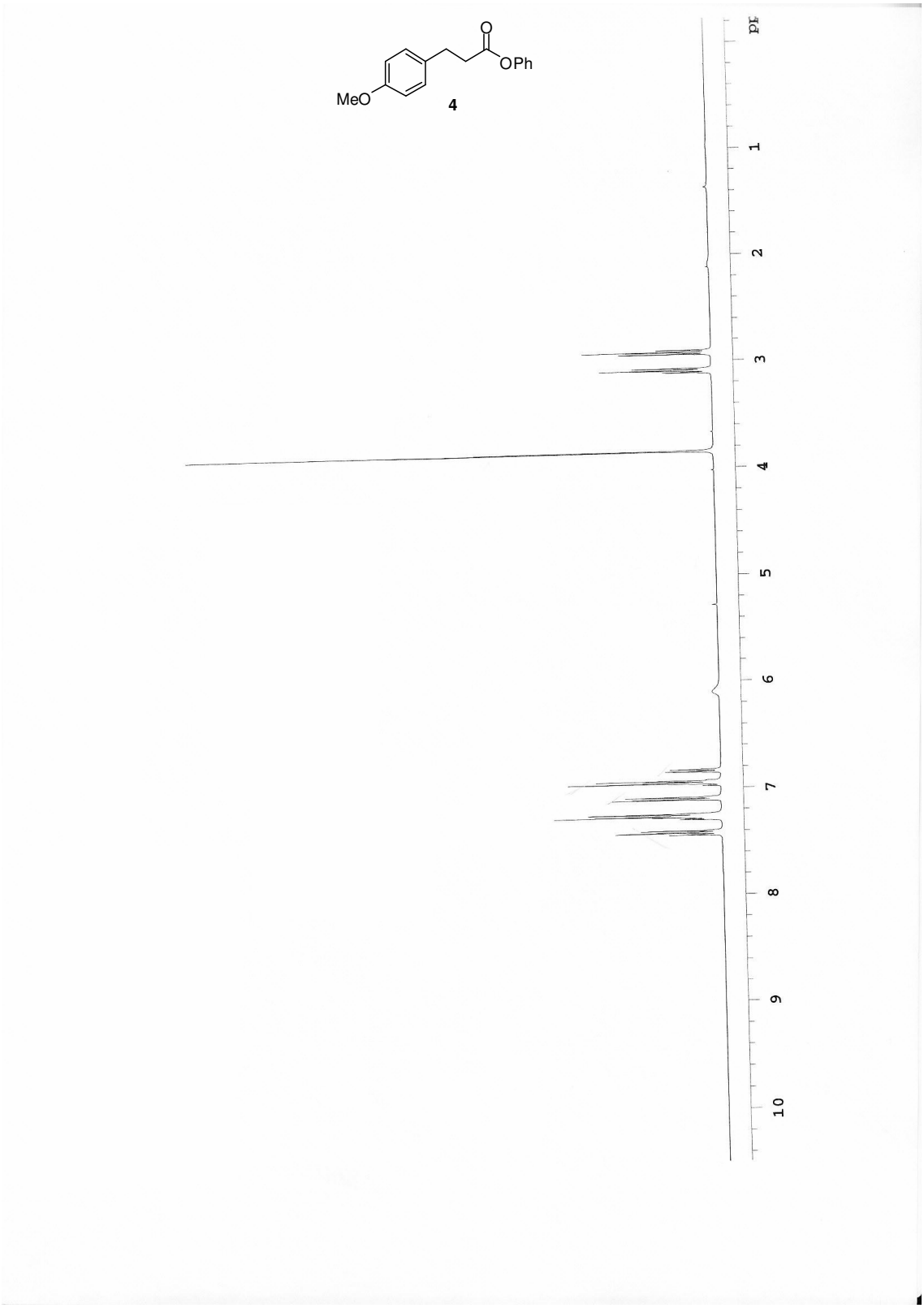
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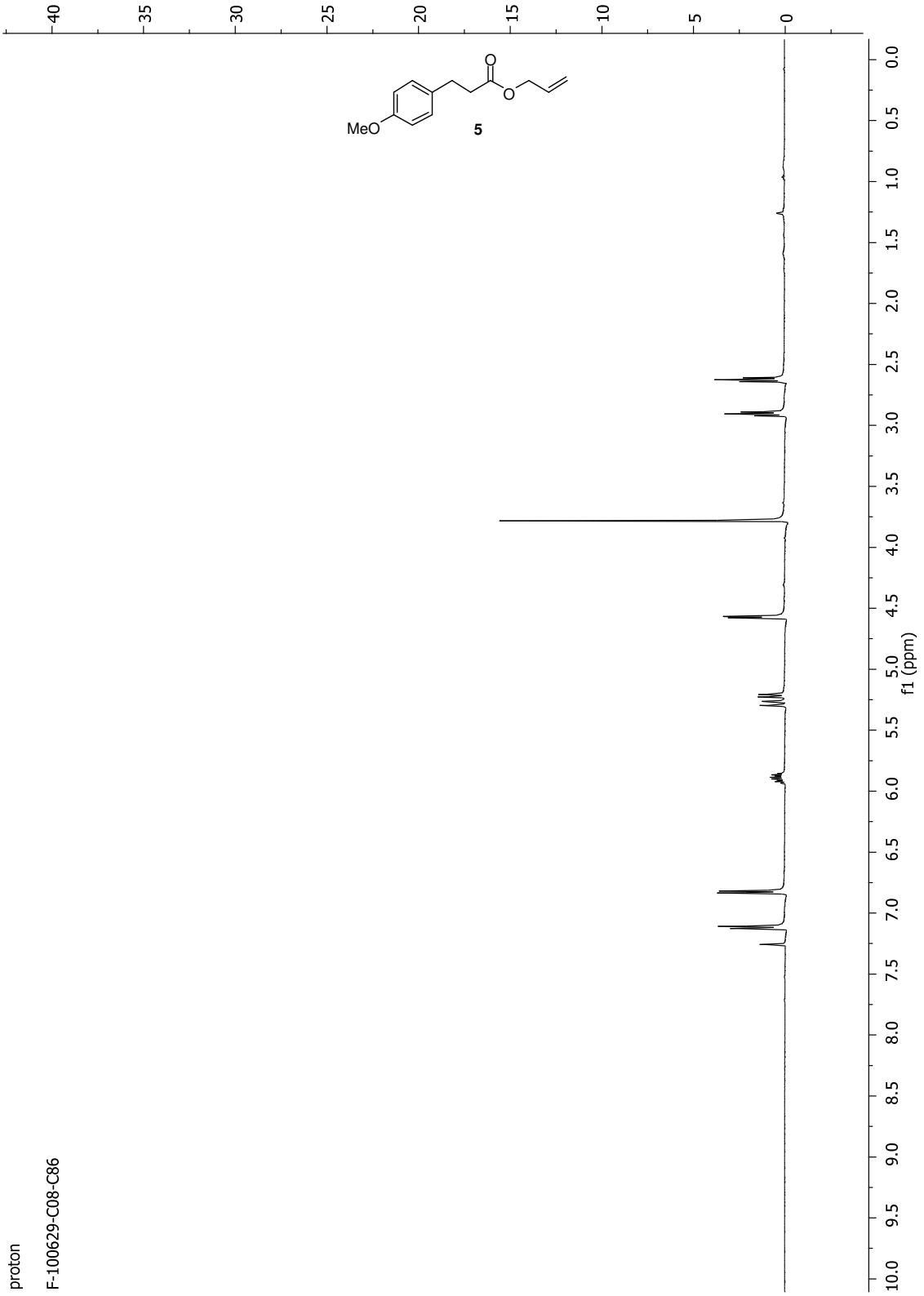
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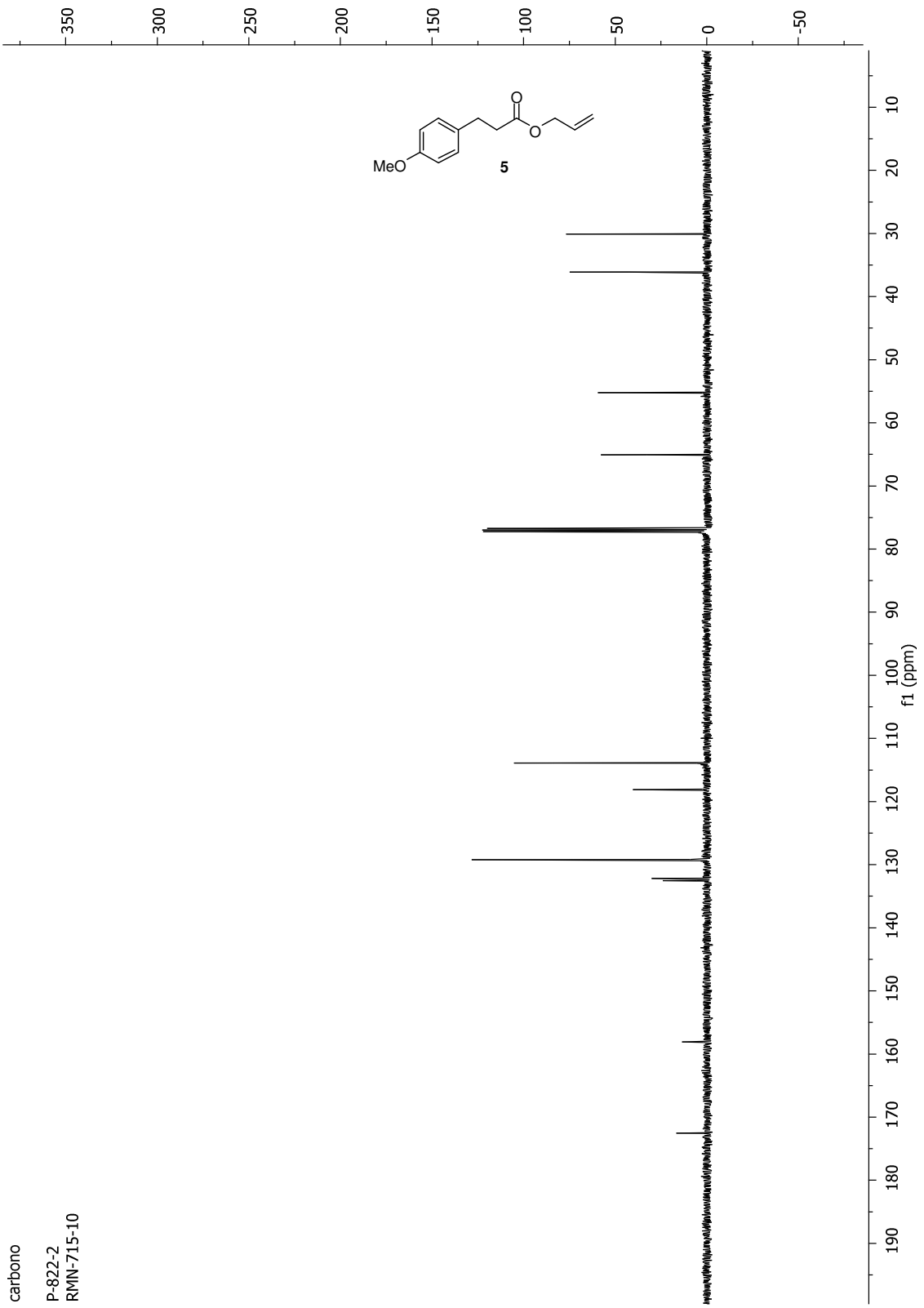
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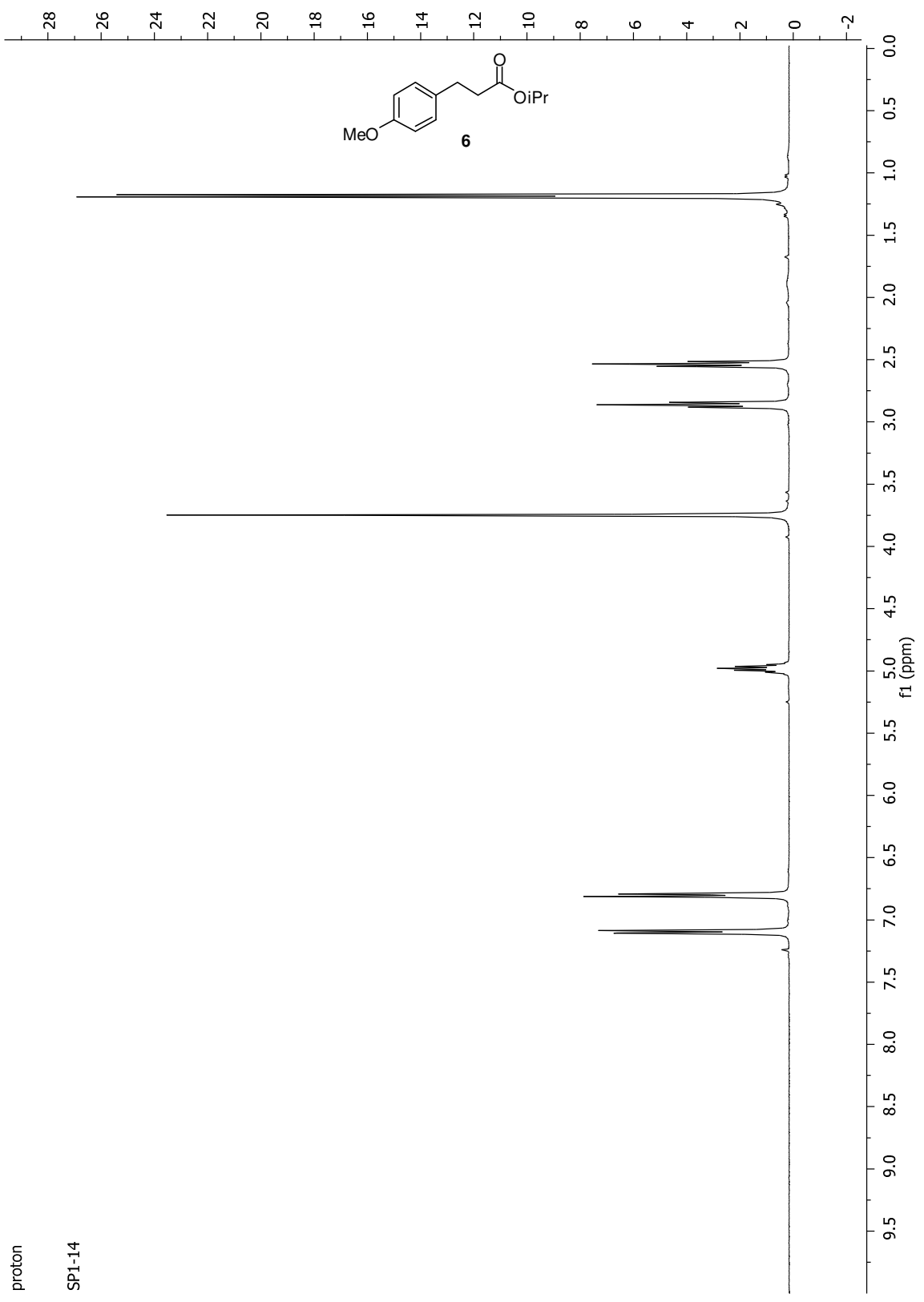
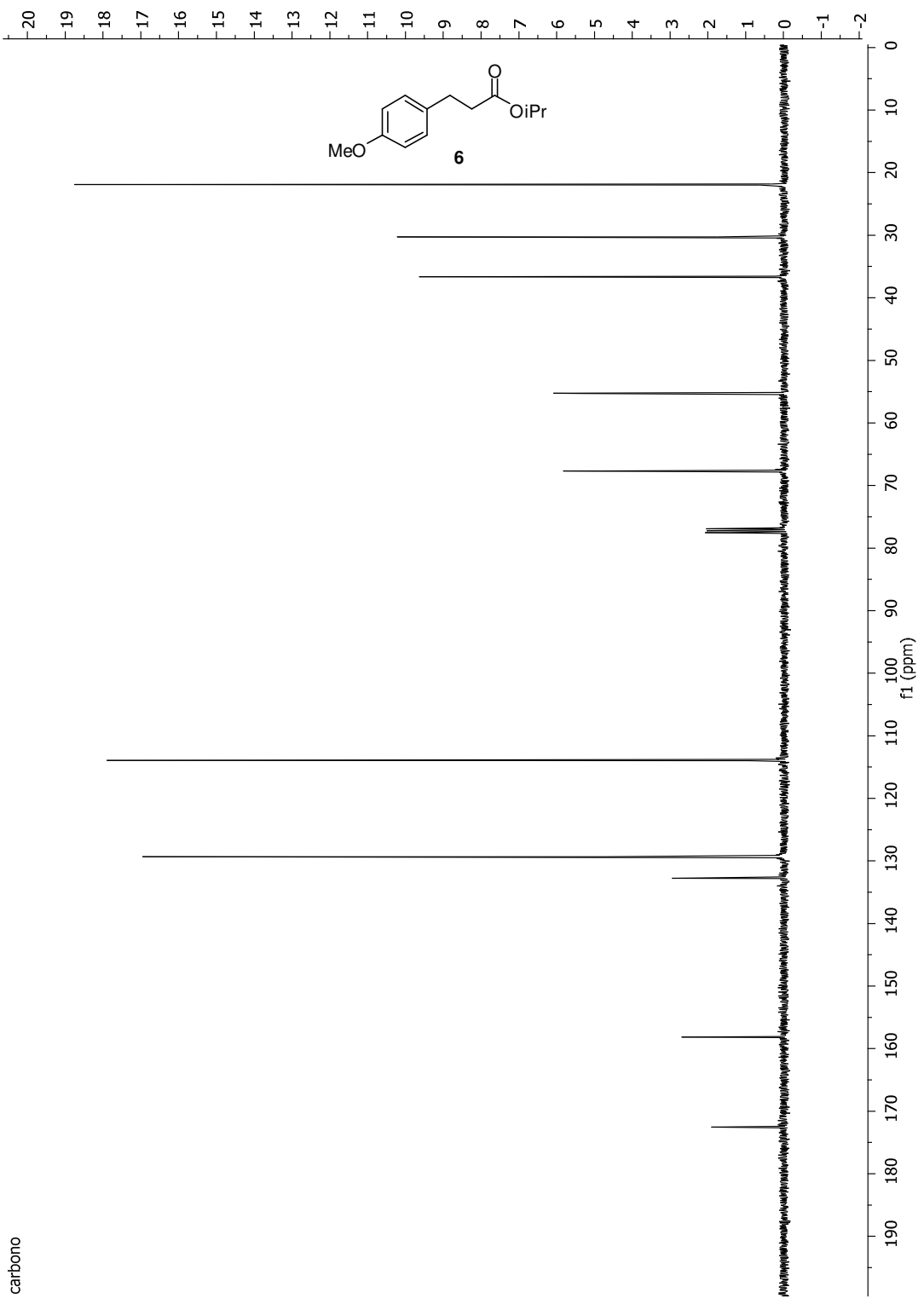
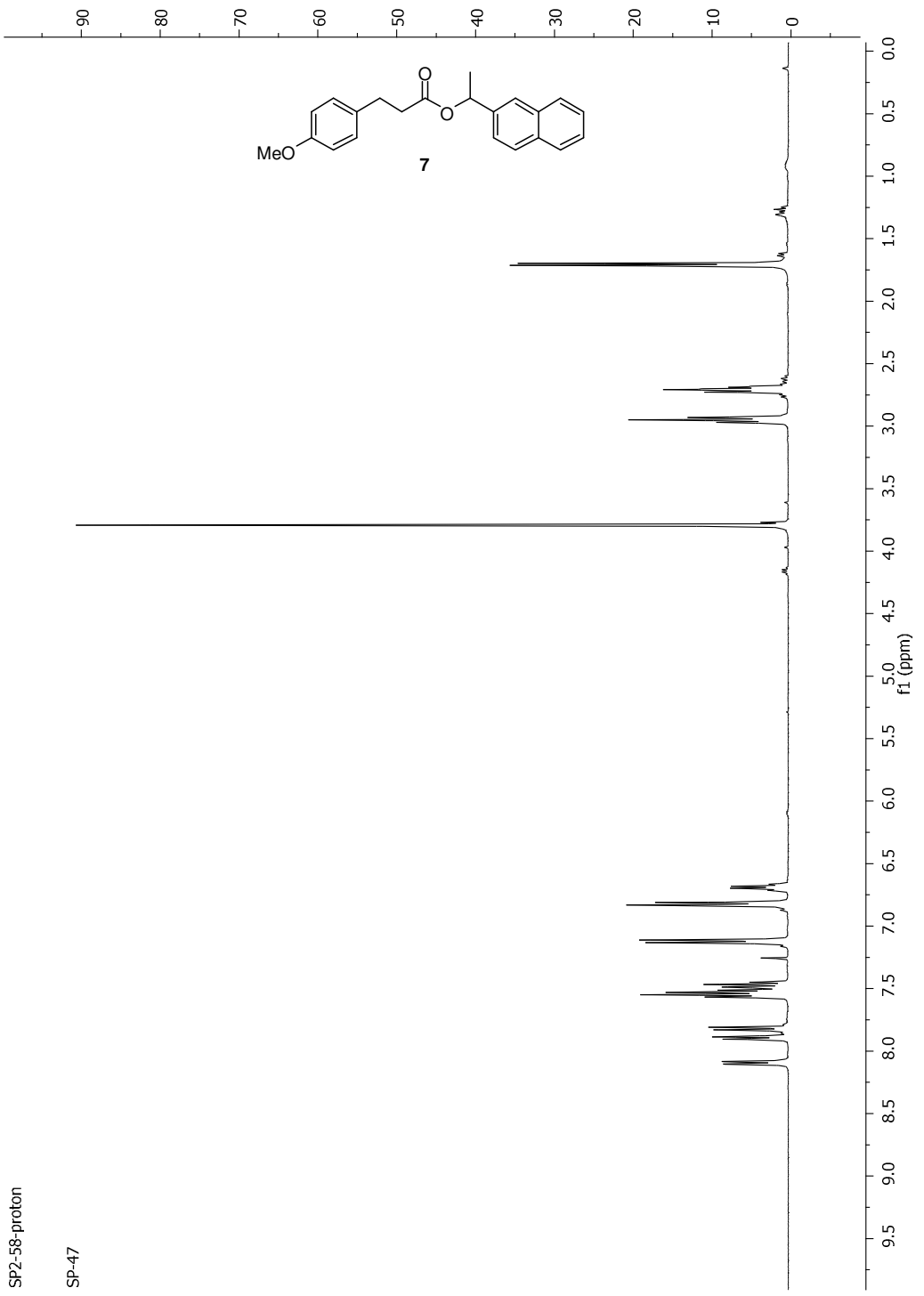
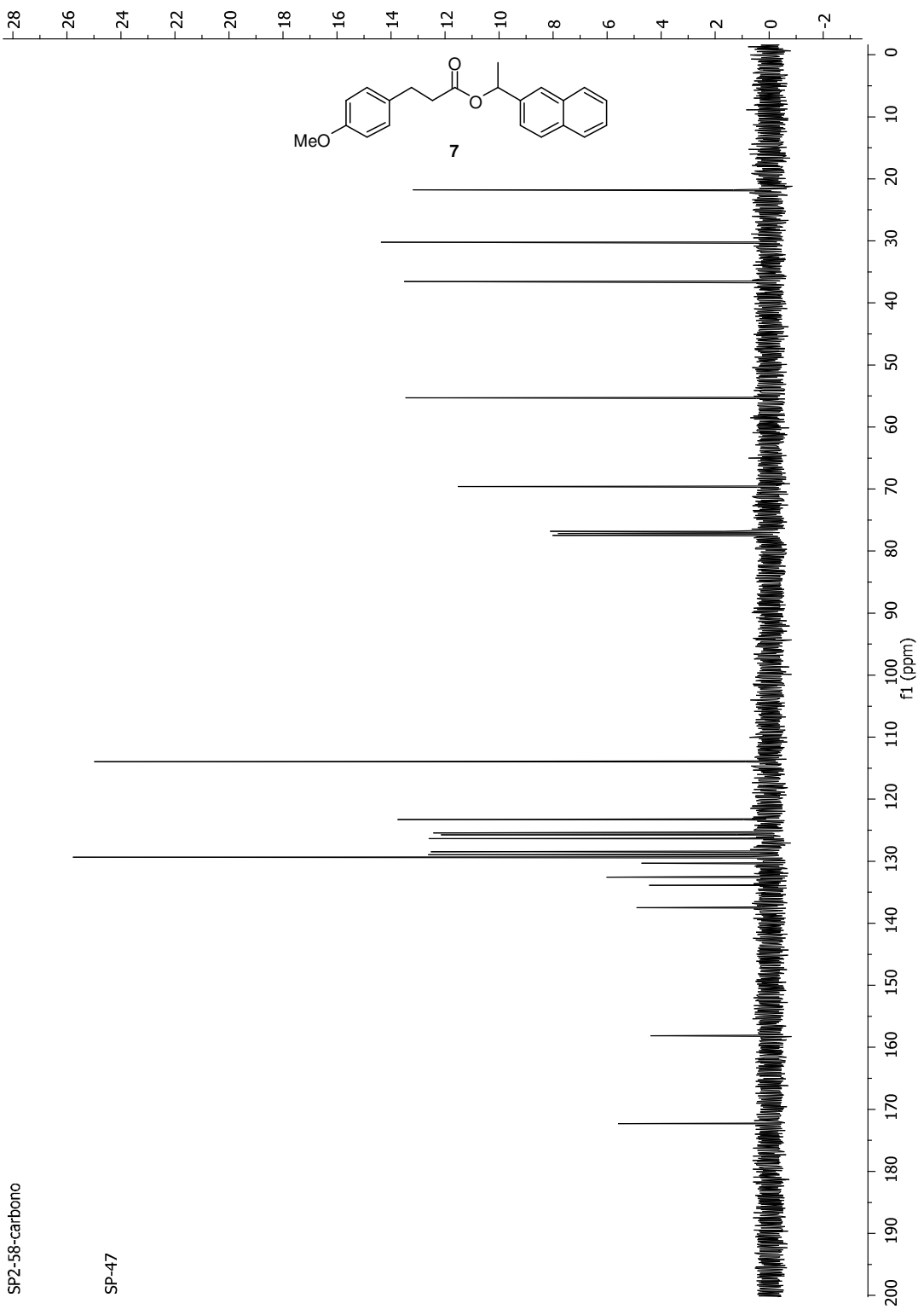
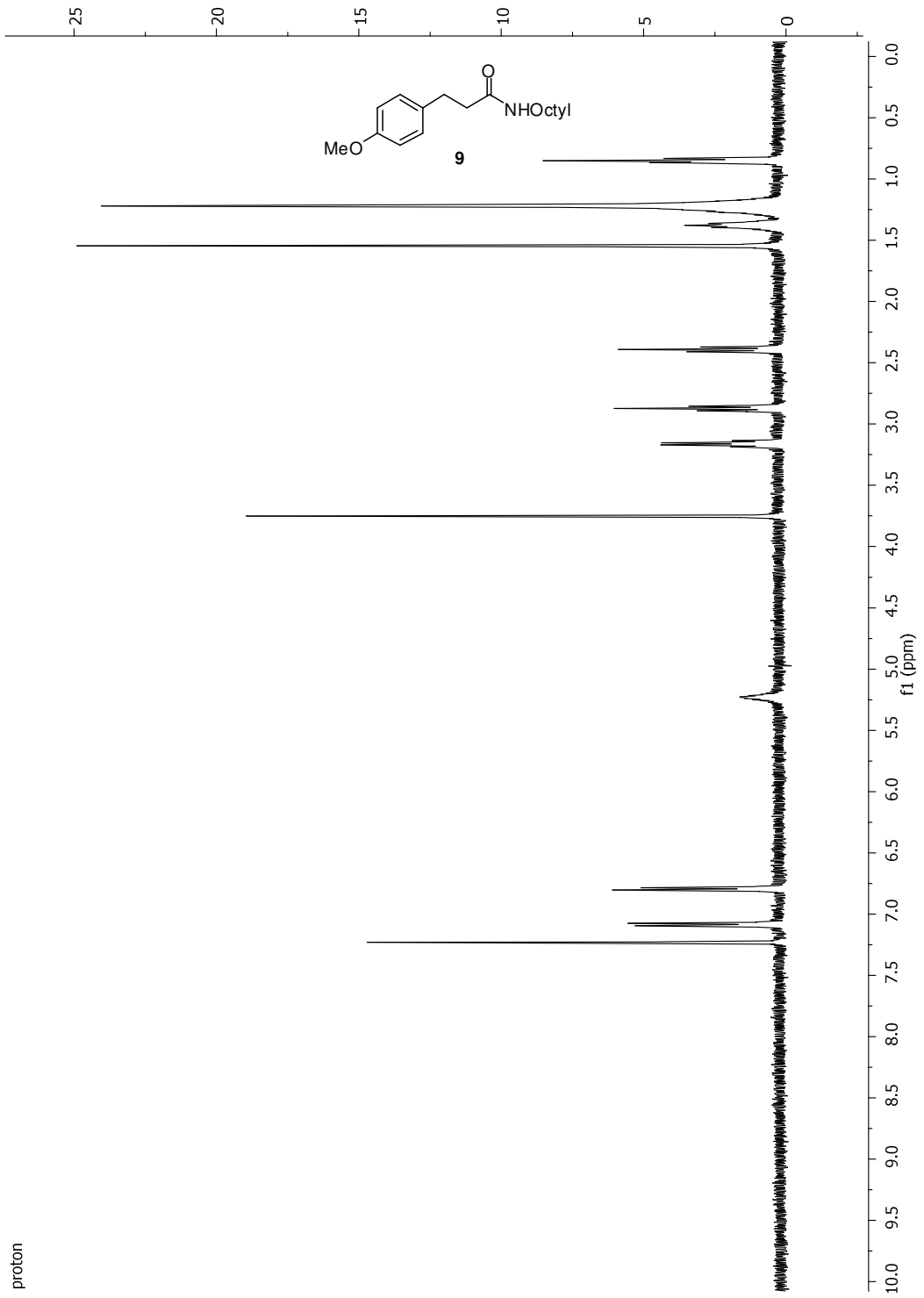
5
f1 (ppm)

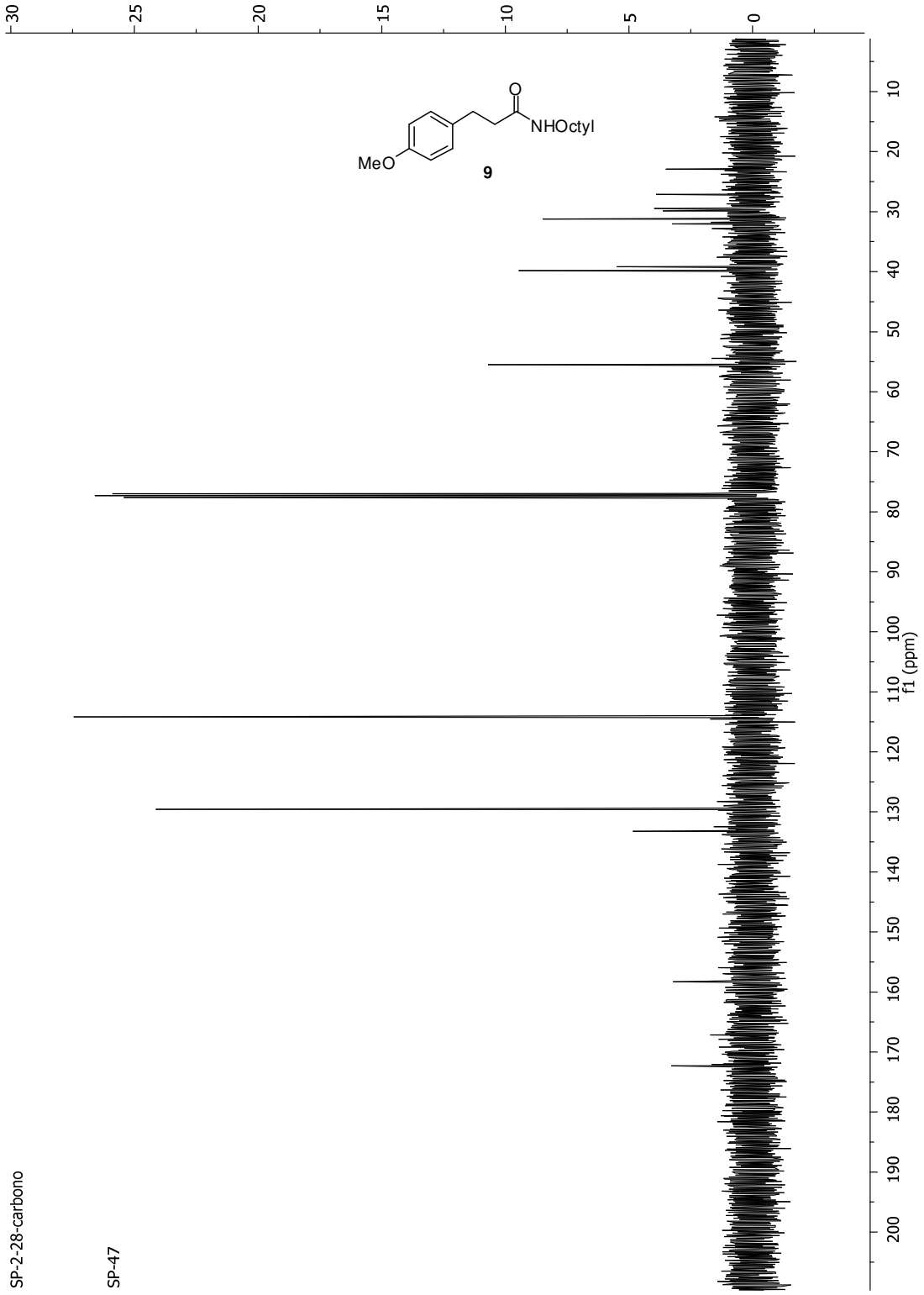

proton
SP1-14
MeO
OiPr
6
f1 (ppm)

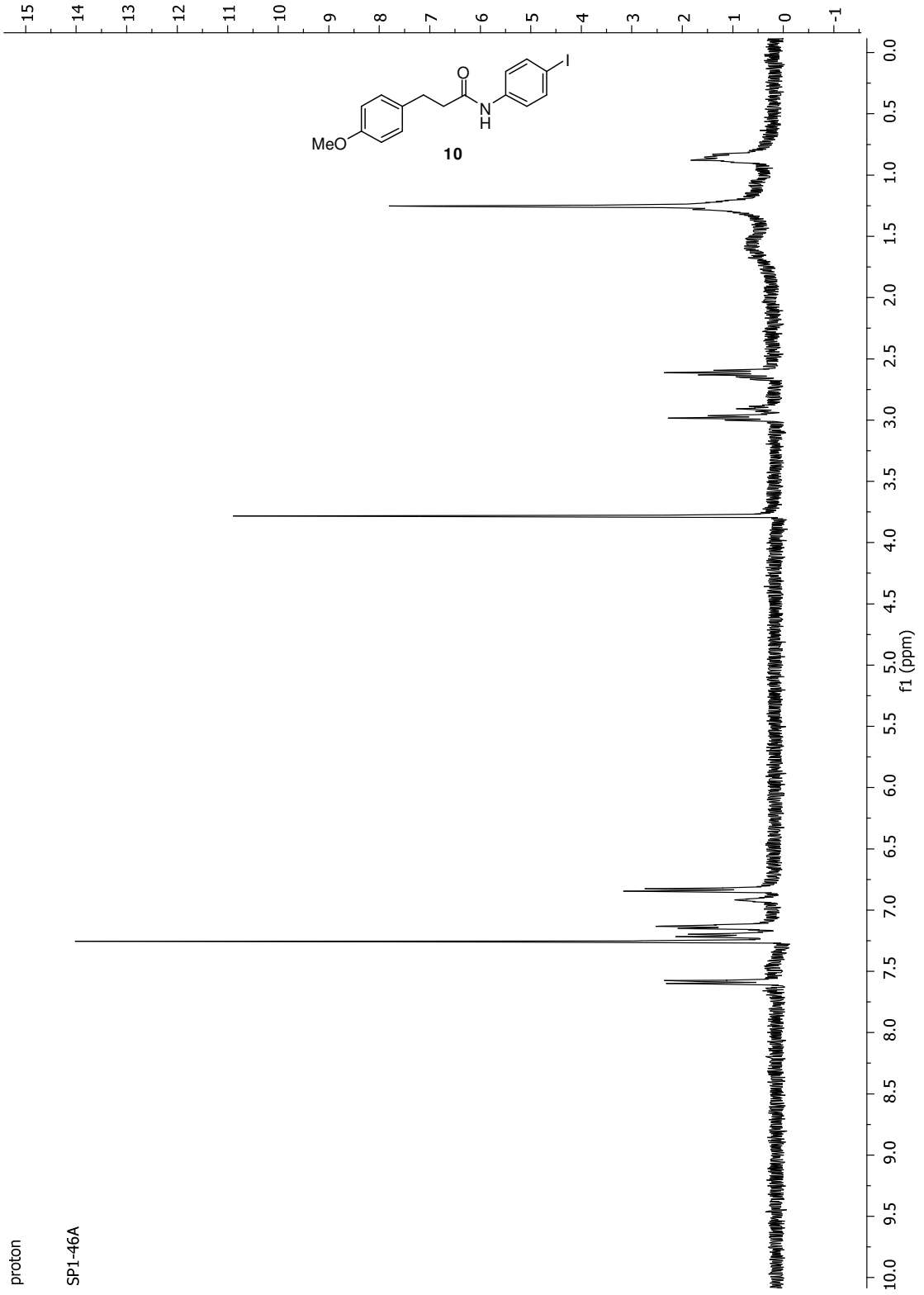

carbono
f1 (ppm)
MeO
OiPr
O
6

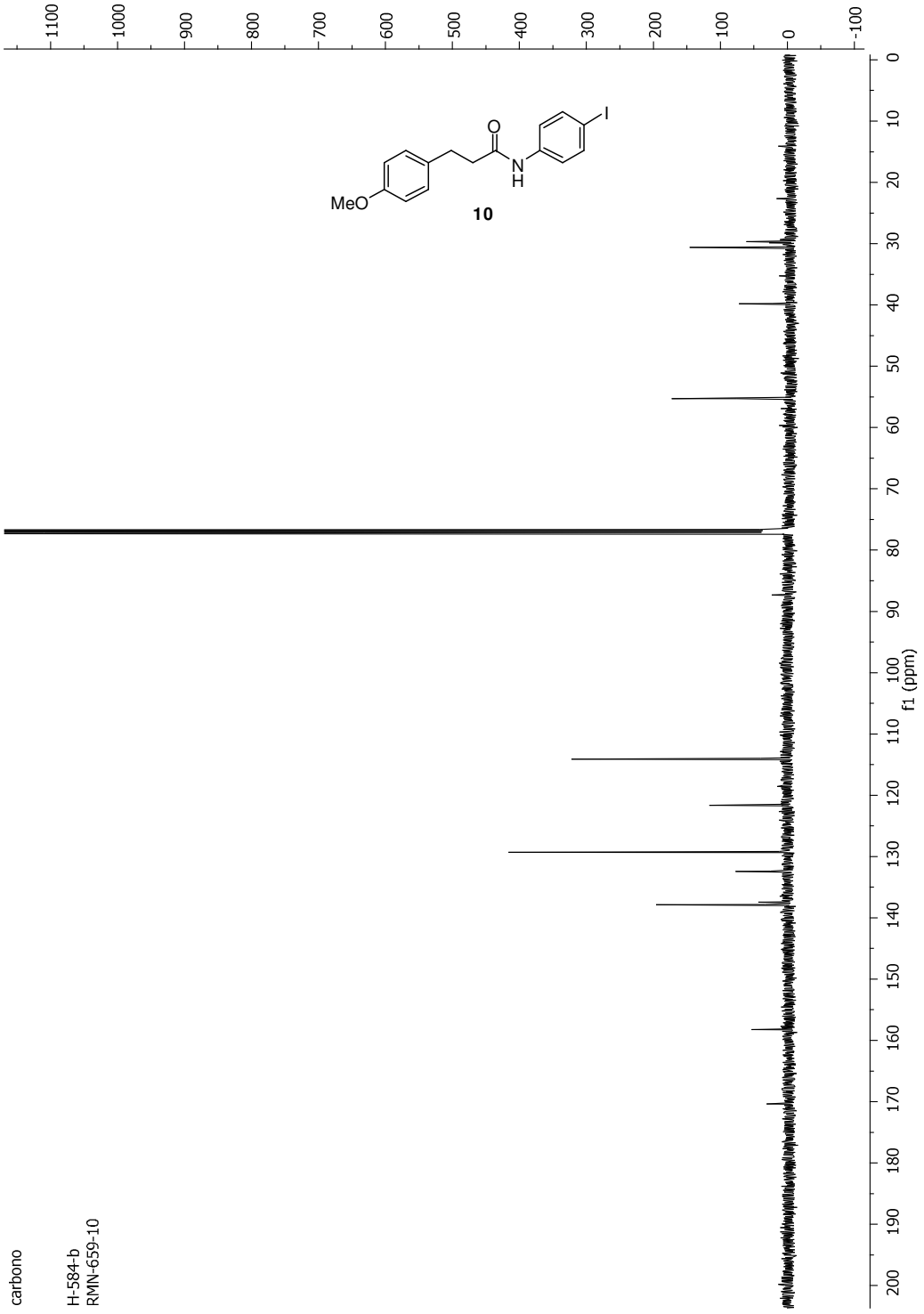

MeO
O
O
7
SP2-58-proton
SP-47
f1 (ppm)

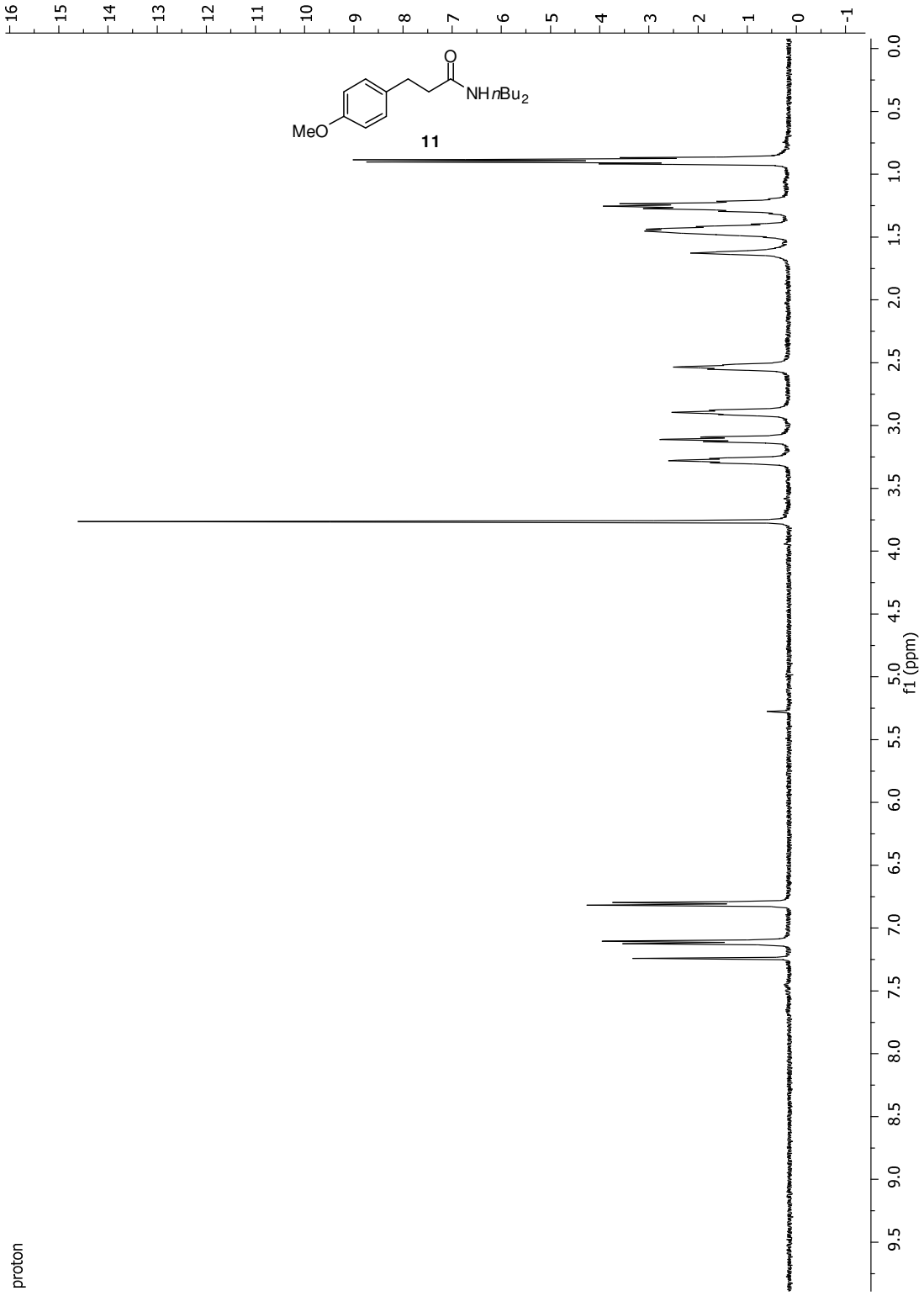

SP2-58-carbono
SP-47
MeO
O
O
7
f1 (ppm)

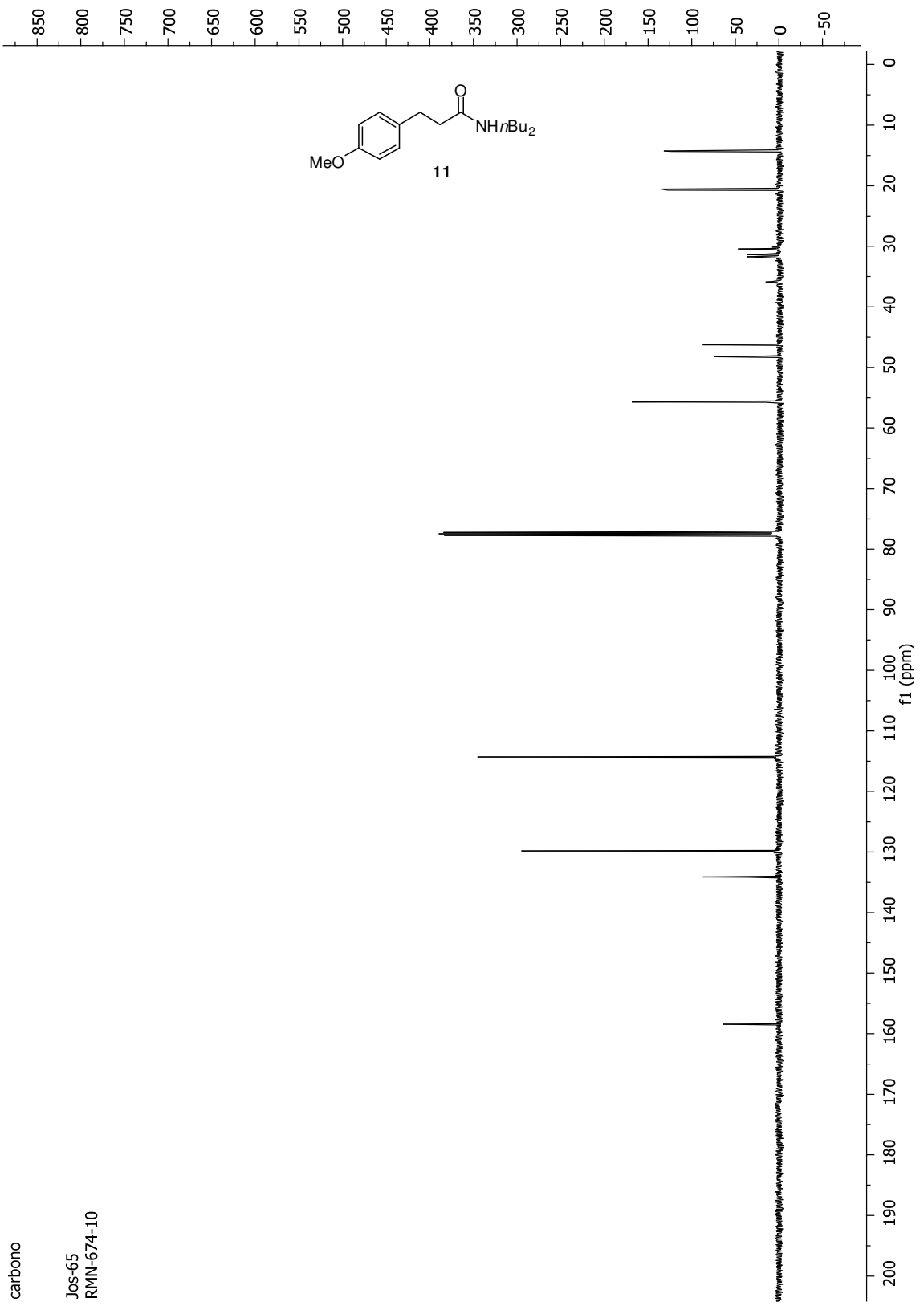

O
NHOctyl
MeO
9
proton
f1 (ppm)

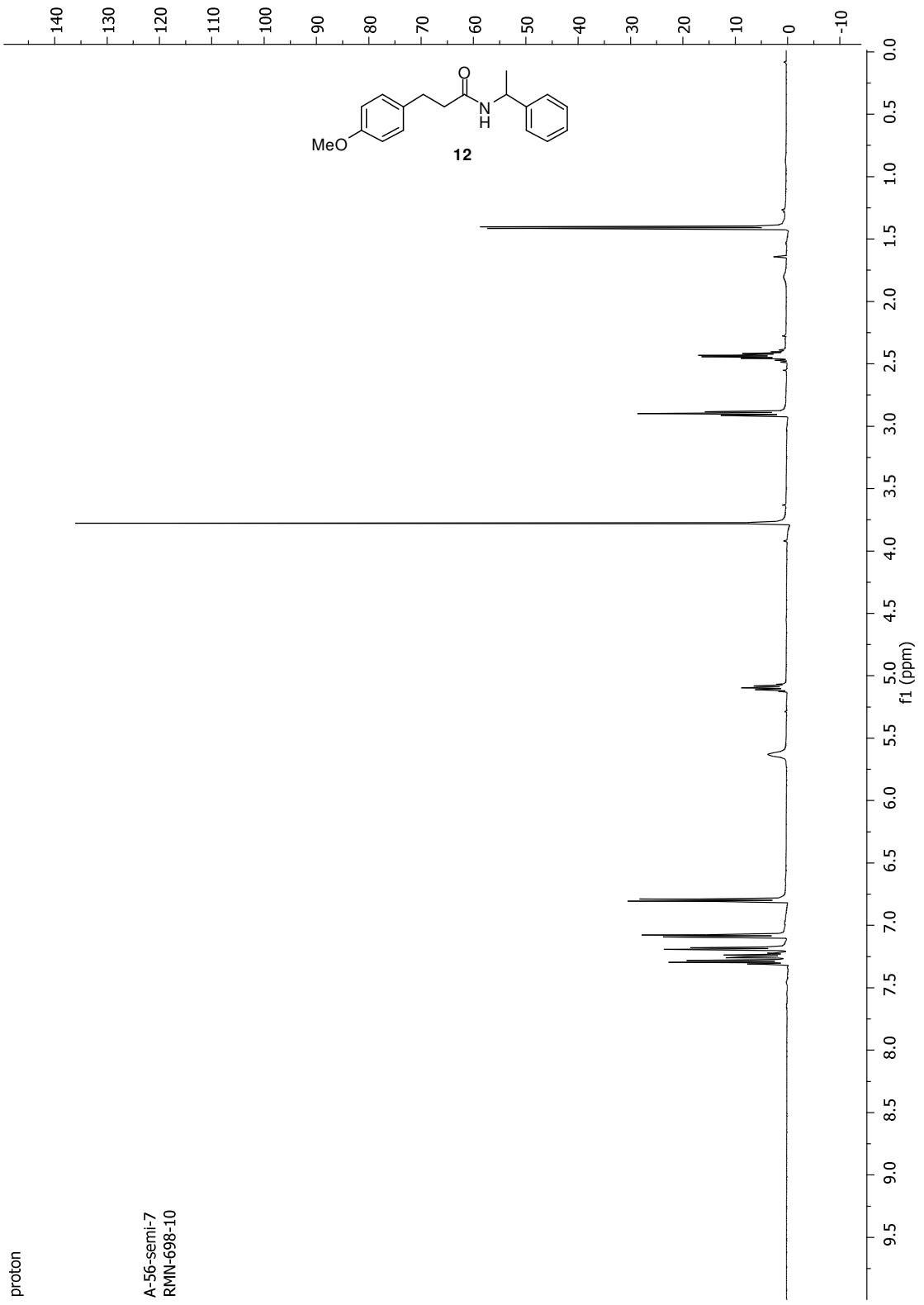

SP-2-28-carbono
SP-47
MeO
NHOctyl
O
9
f1 (ppm)

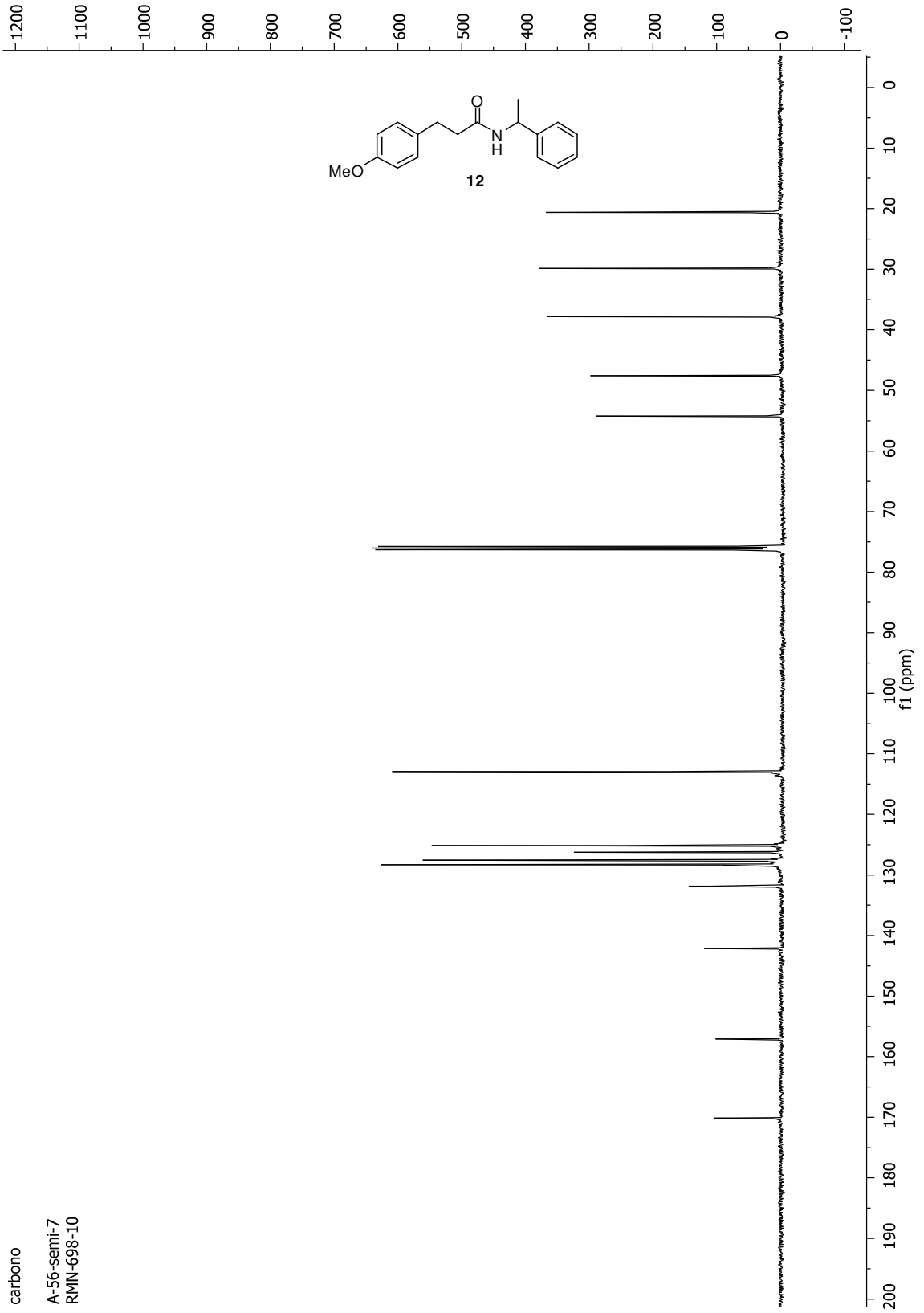

proton
SP1-46A
MeO
O
N
H
I
10
f1 (ppm)

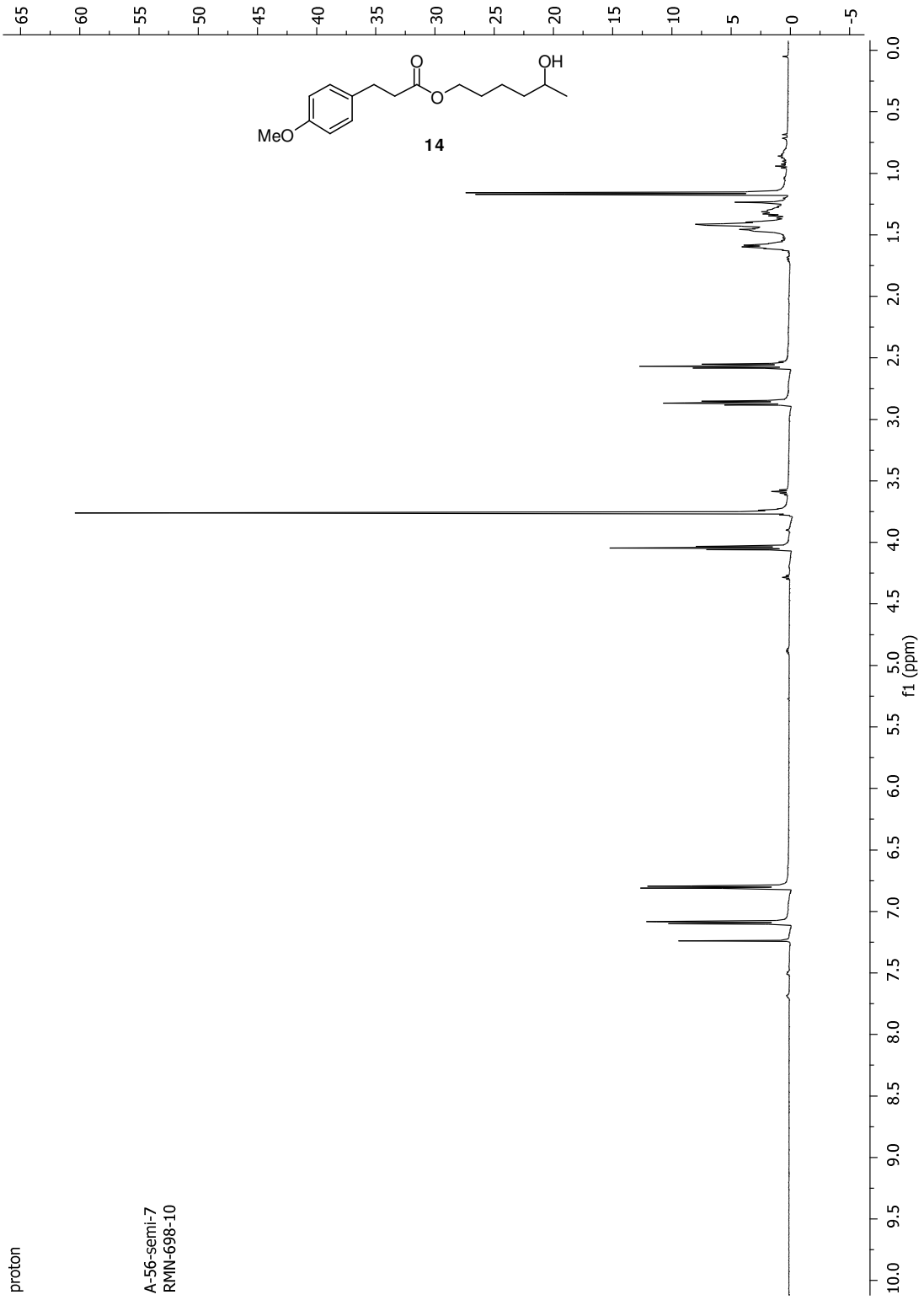

carbono
H-584-b
RMN-659-10
MeO
O
N
H
I
10
f1 (ppm)

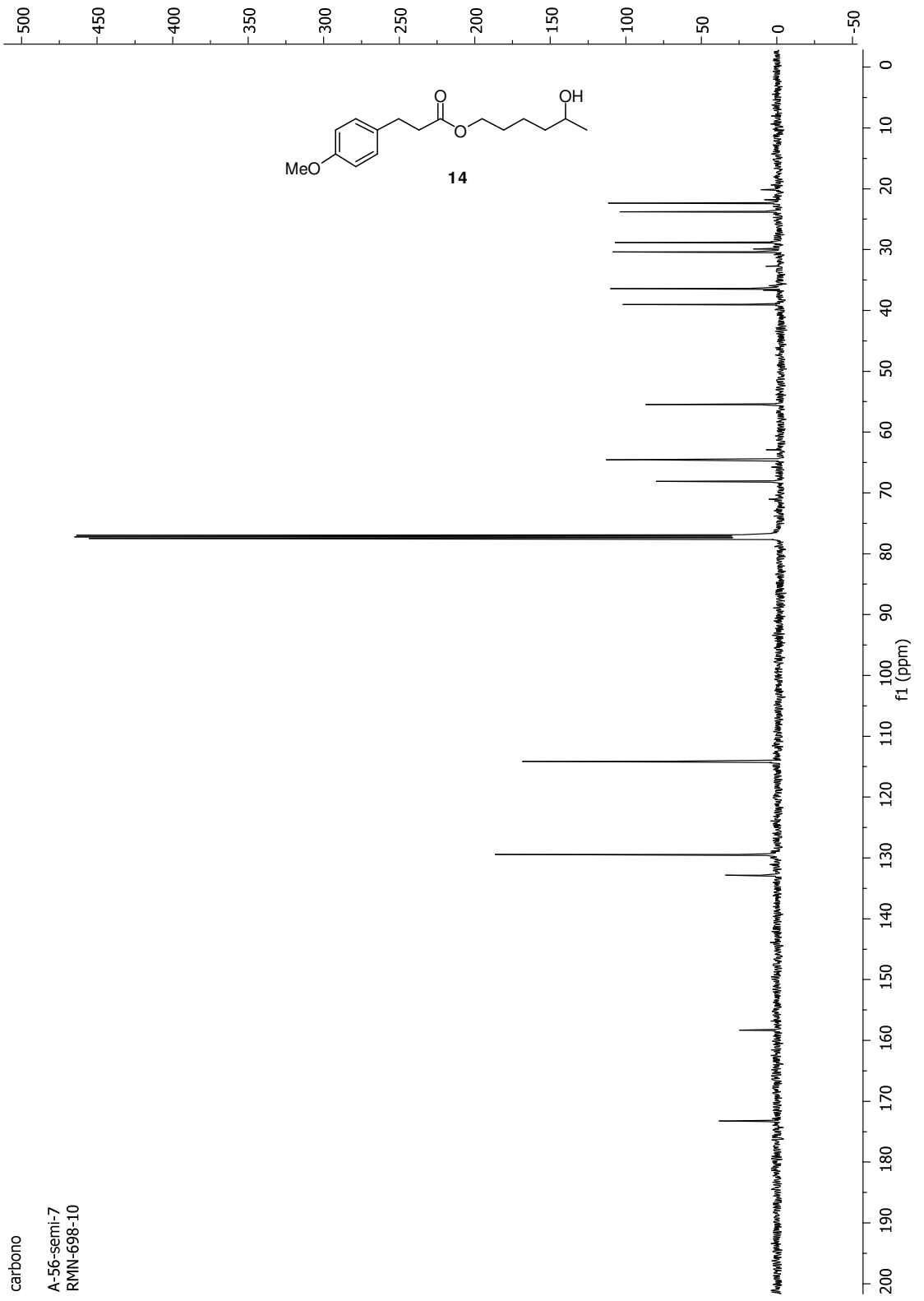

proton
f1 (ppm)
MeO
NHnBu$_2$
O
11

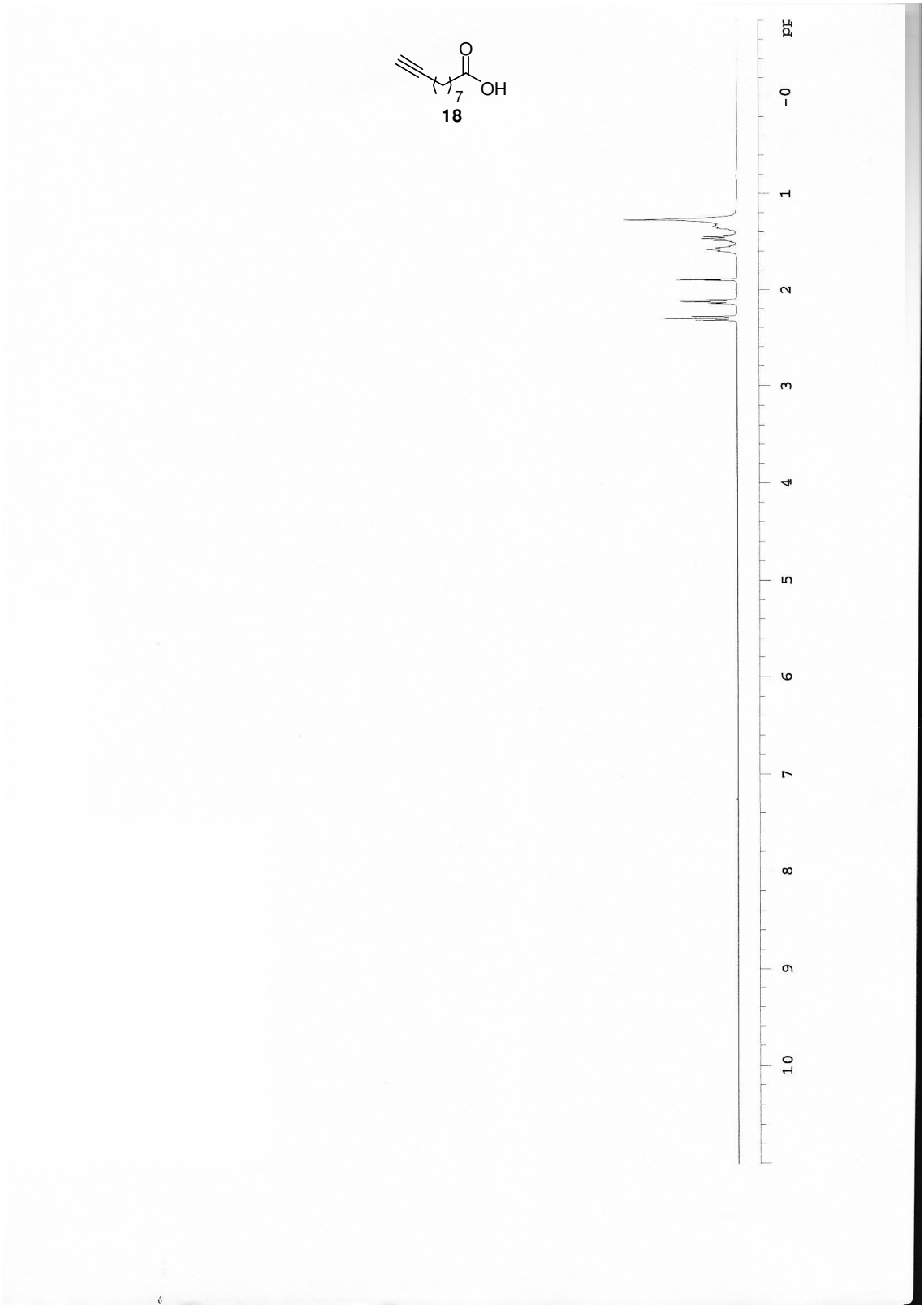

carbono
Jos-65
RMN-674-10
MeO
NHnBu$_2$
O
11
f1 (ppm)

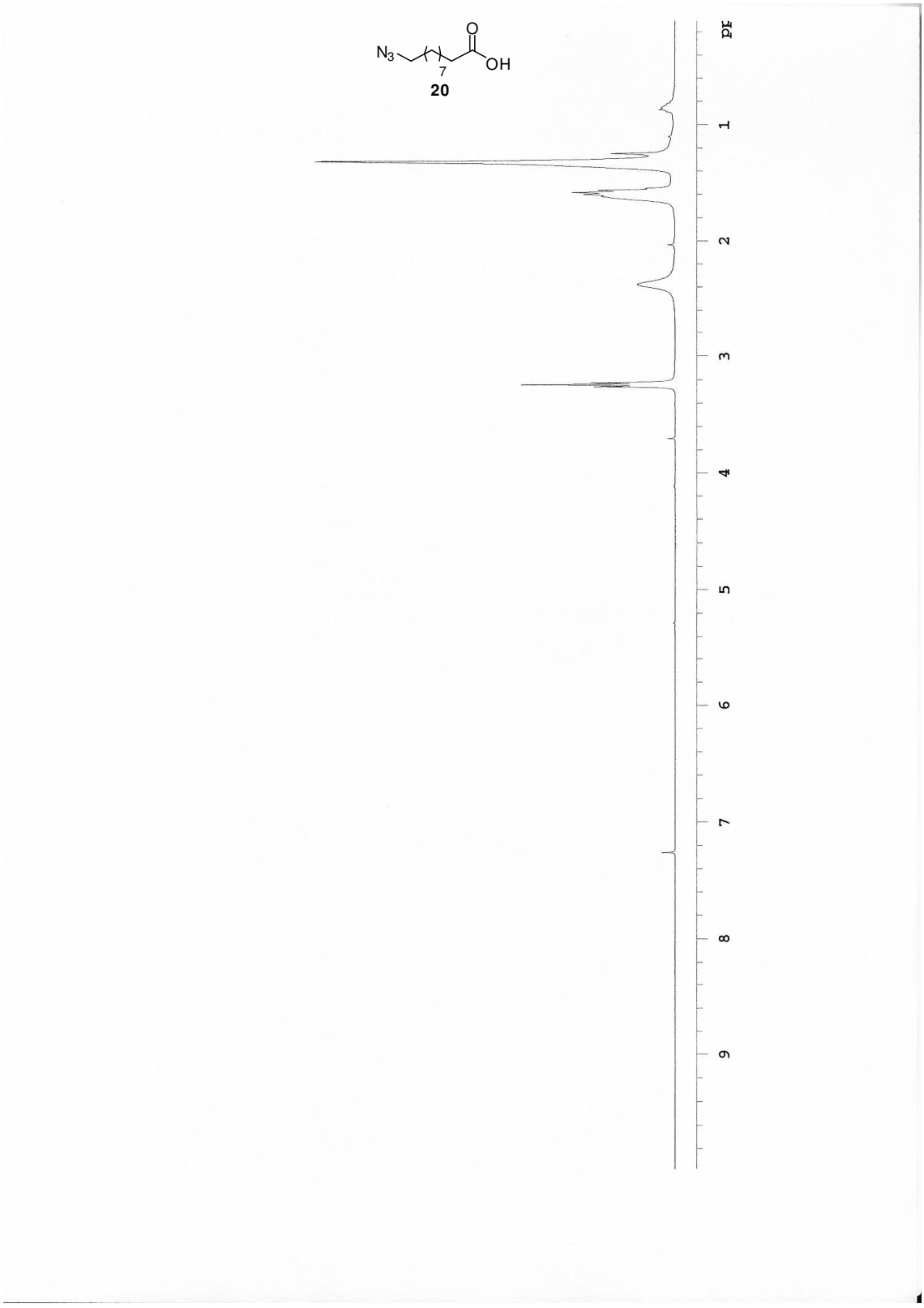

proton
A-56-semi-7
RMN-698-10
MeO
12
f1 (ppm)

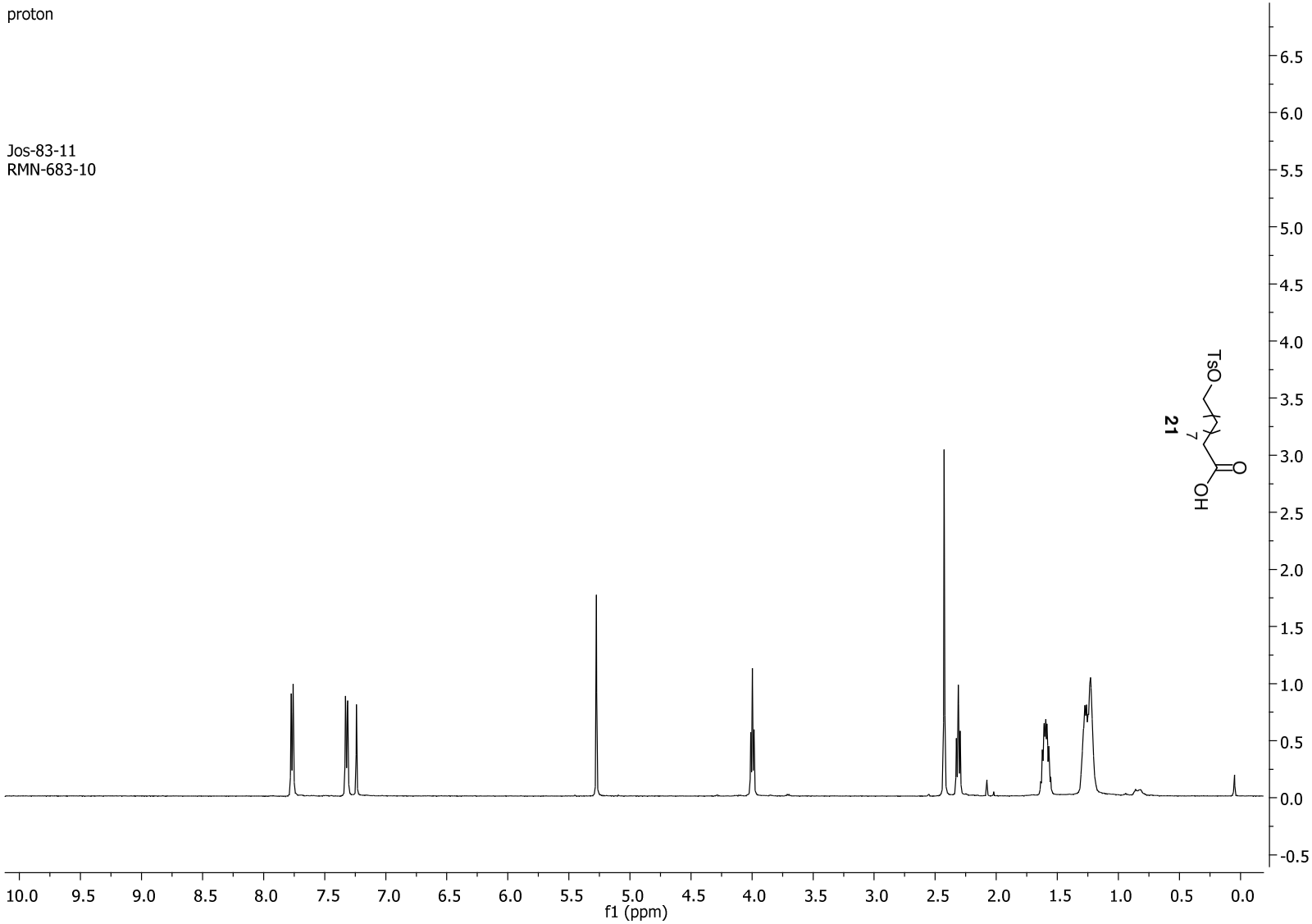

carbono
A-56-semi-7
RMN-698-10
MeO
12
f1 (ppm)

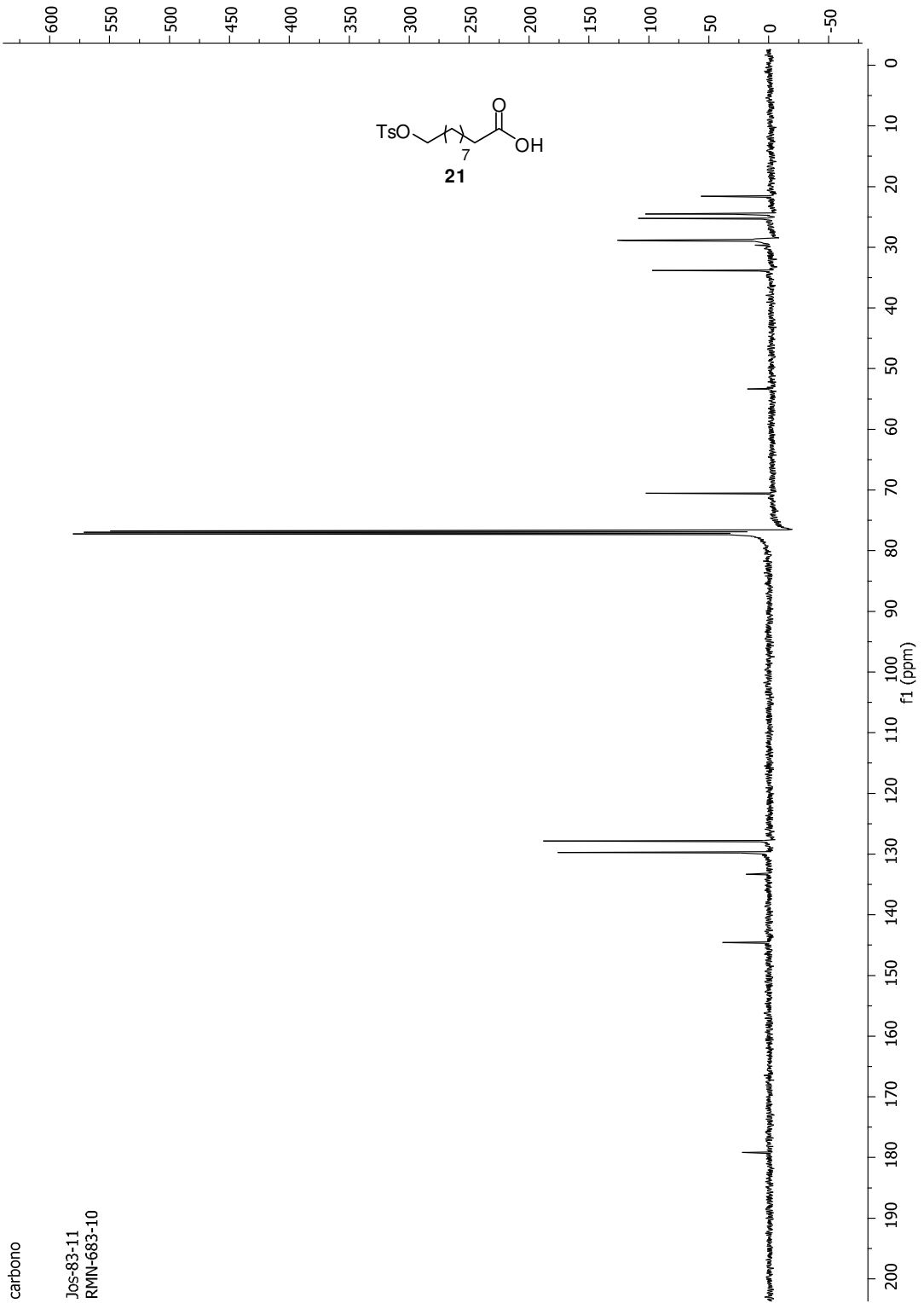

proton
A-56-semi-7
RMN-698-10
MeO
O
O
OH
14
f1 (ppm)

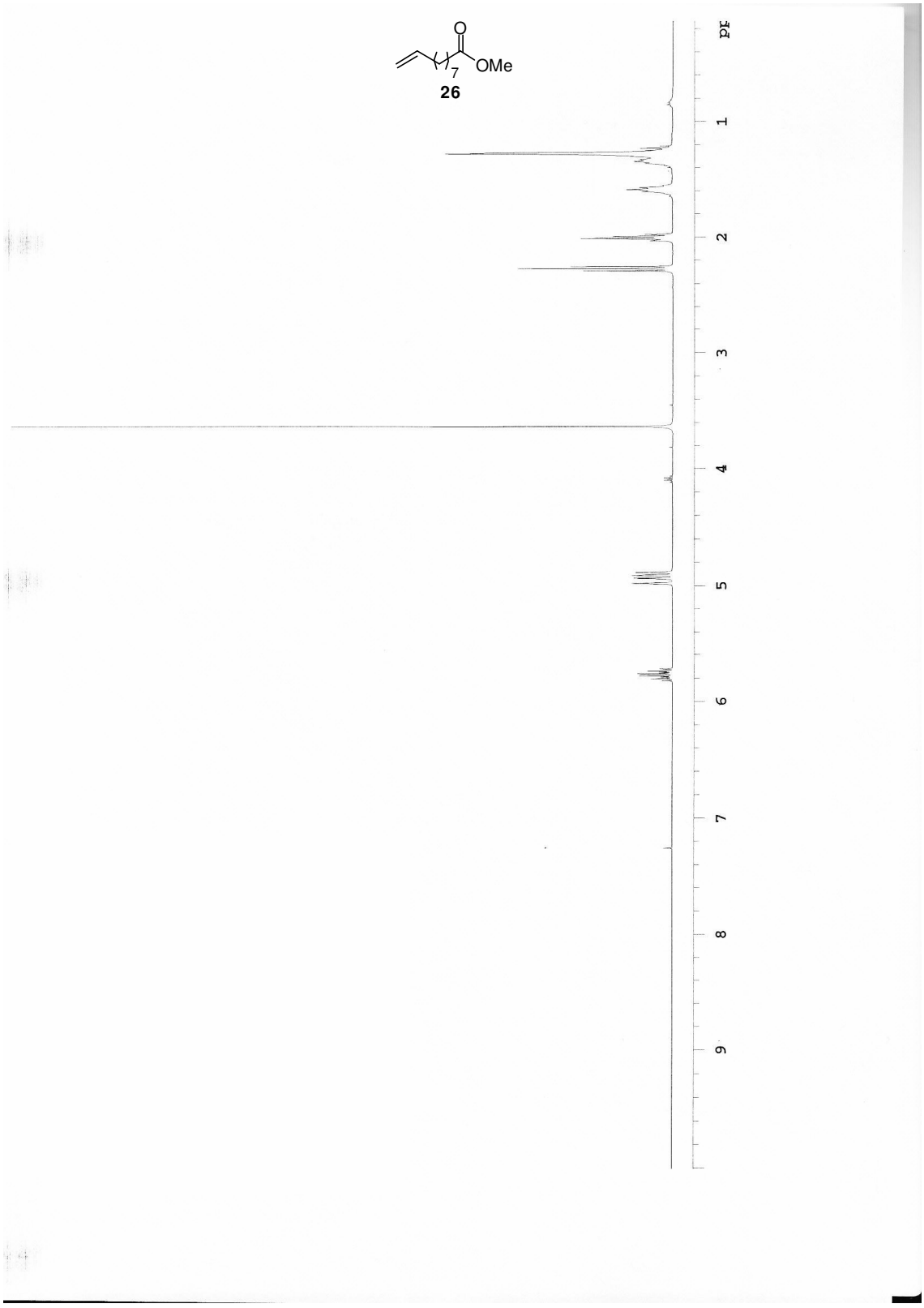

carbono
A-56-semi-7
RMN-698-10
MeO
O
O
OH
14
f1 (ppm)

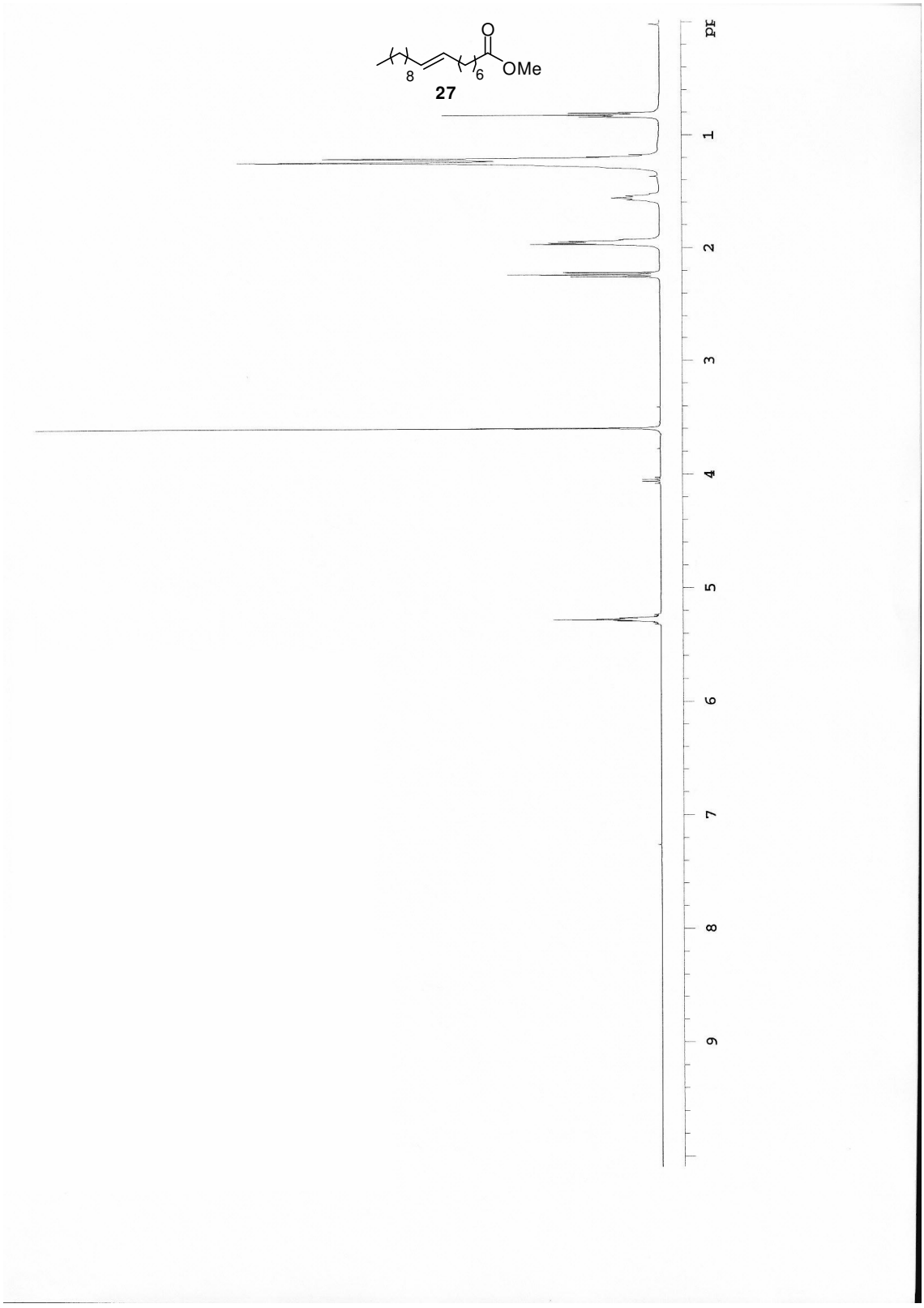

O
OH
7
18
PPM
-0
1
2
3
4
5
6
7
8
9
10

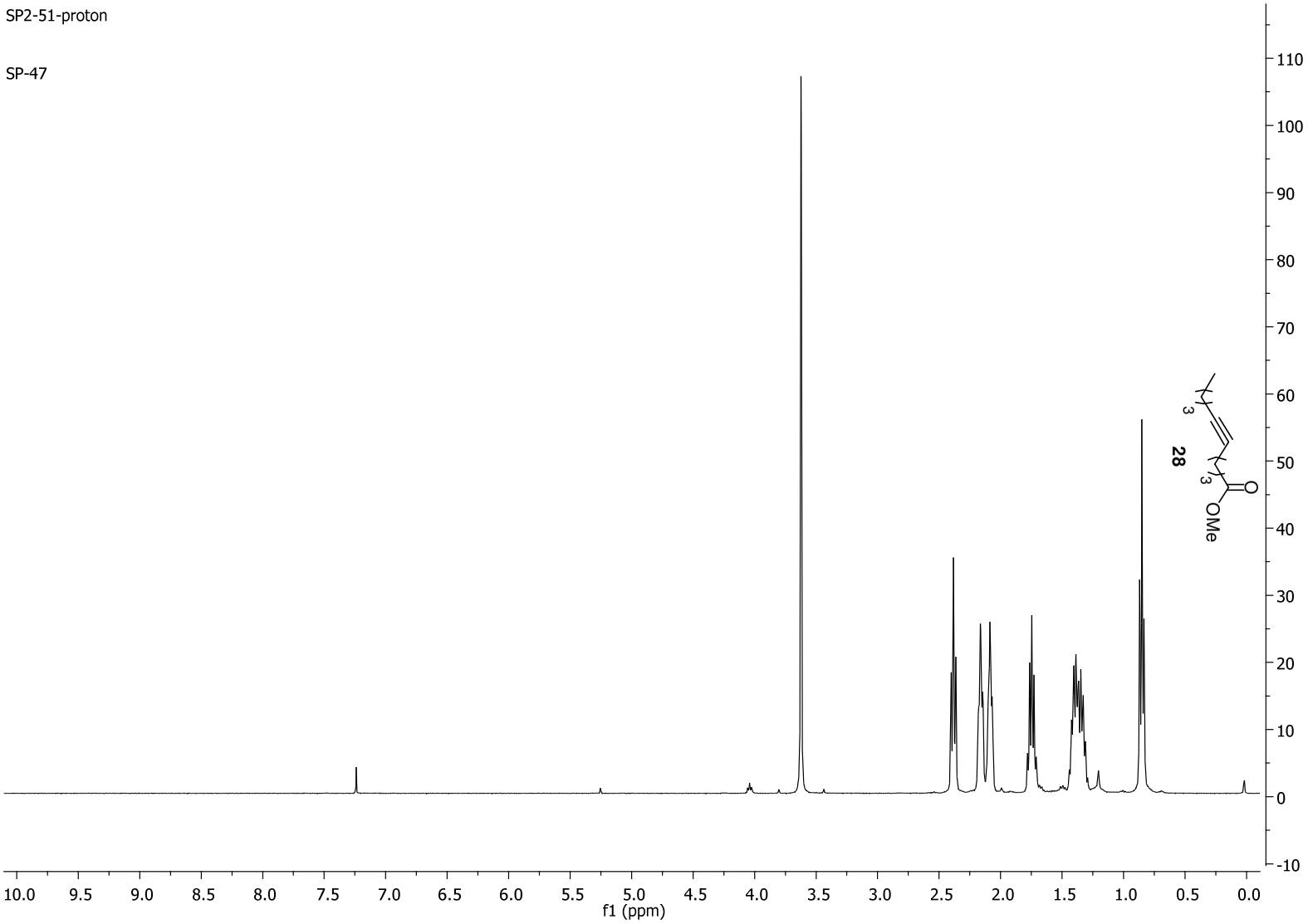

N3
7
OH
O
20
pp
1
2
3
4
5
6
7
8
9

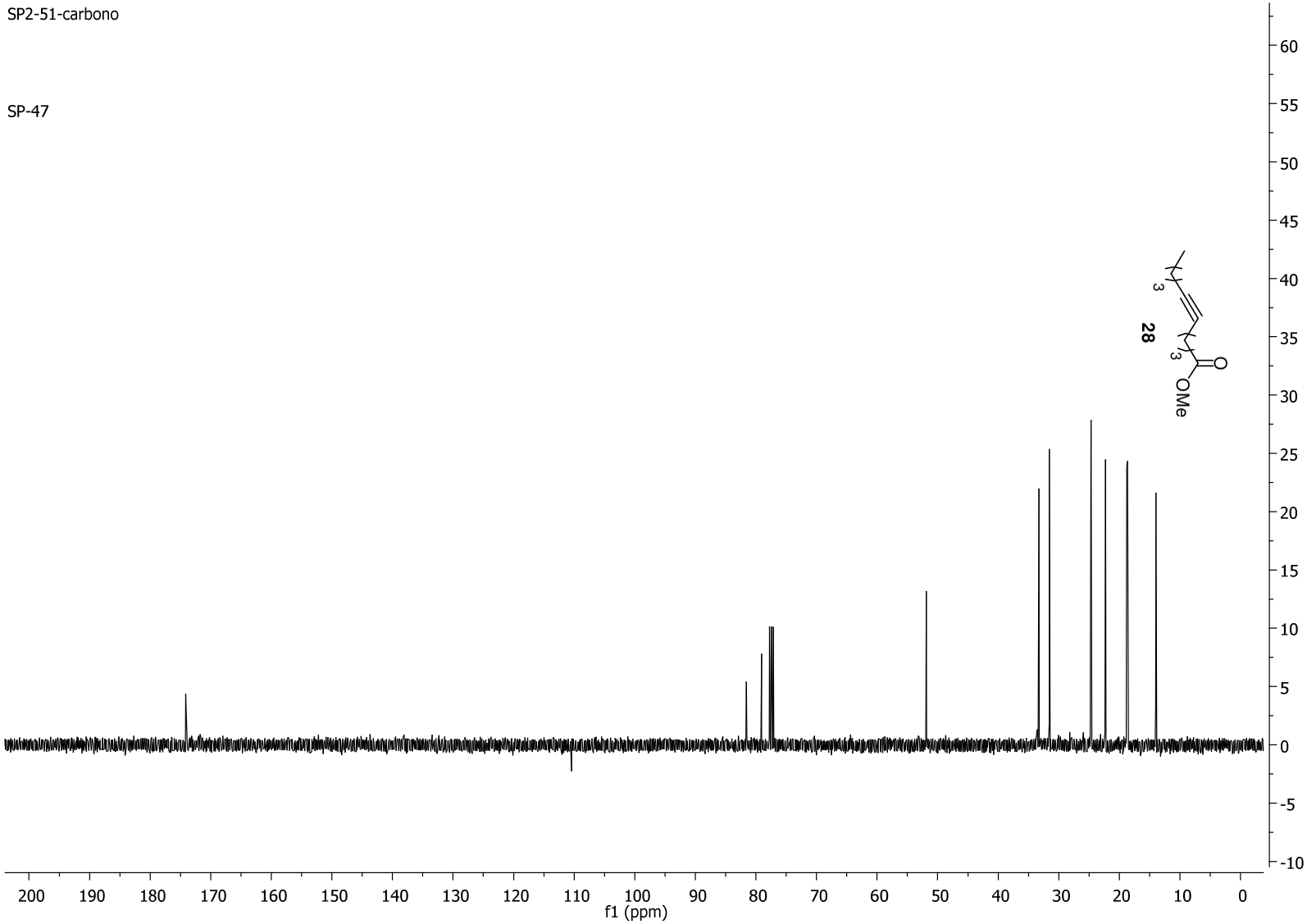

proton
Jos-83-11
RMN-683-10
TsO
7
21
OH
O
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
0.5
0.0
-0.5
10.0
9.5
9.0
8.5
8.0
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
0.5
0.0
f1 (ppm)

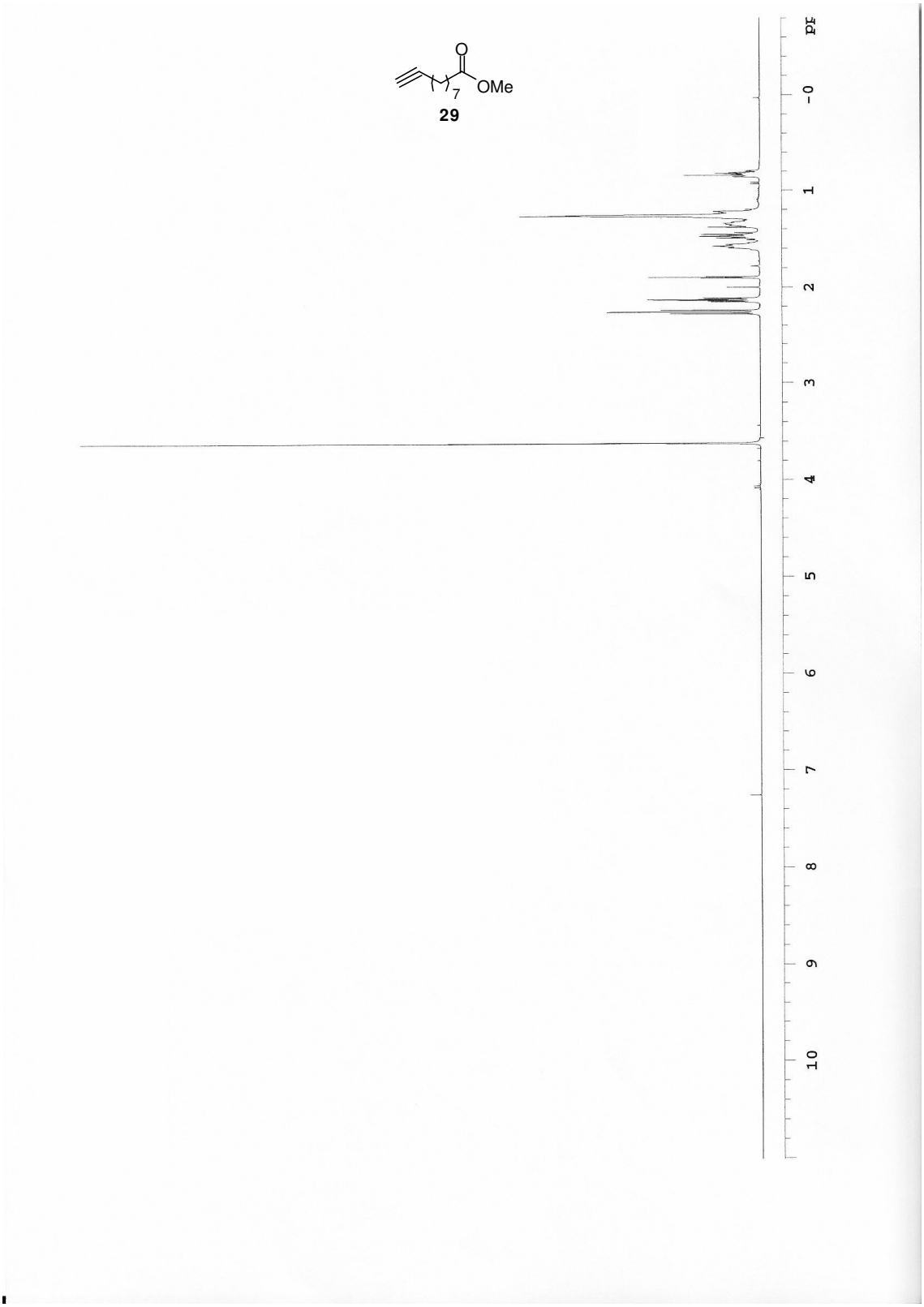

carbono
Jos-83-11
RMN-683-10
TsO
7
O
OH
21
f1 (ppm)

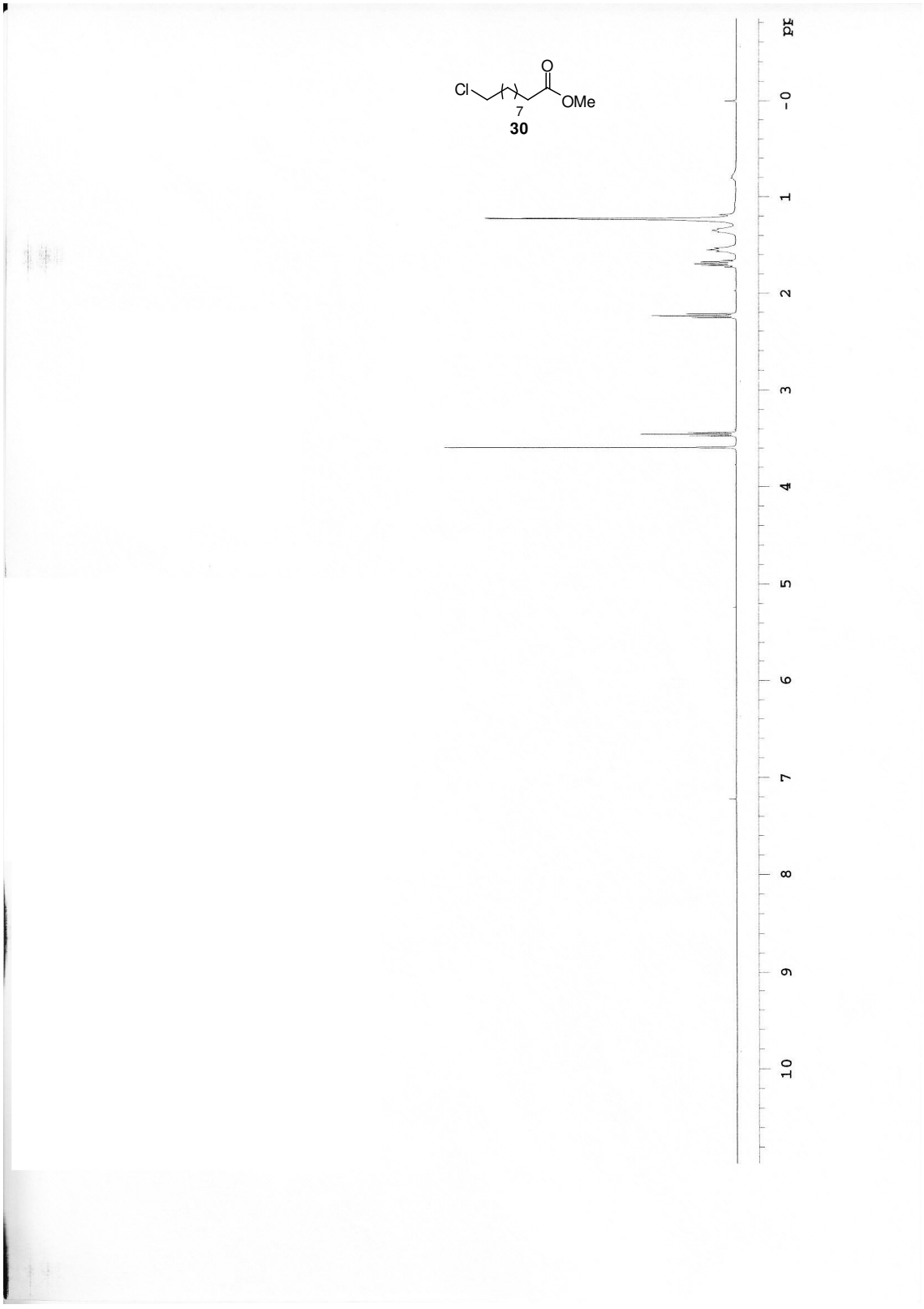

O
OMe
7
26
ppm
1
2
3
4
5
6
7
8
9

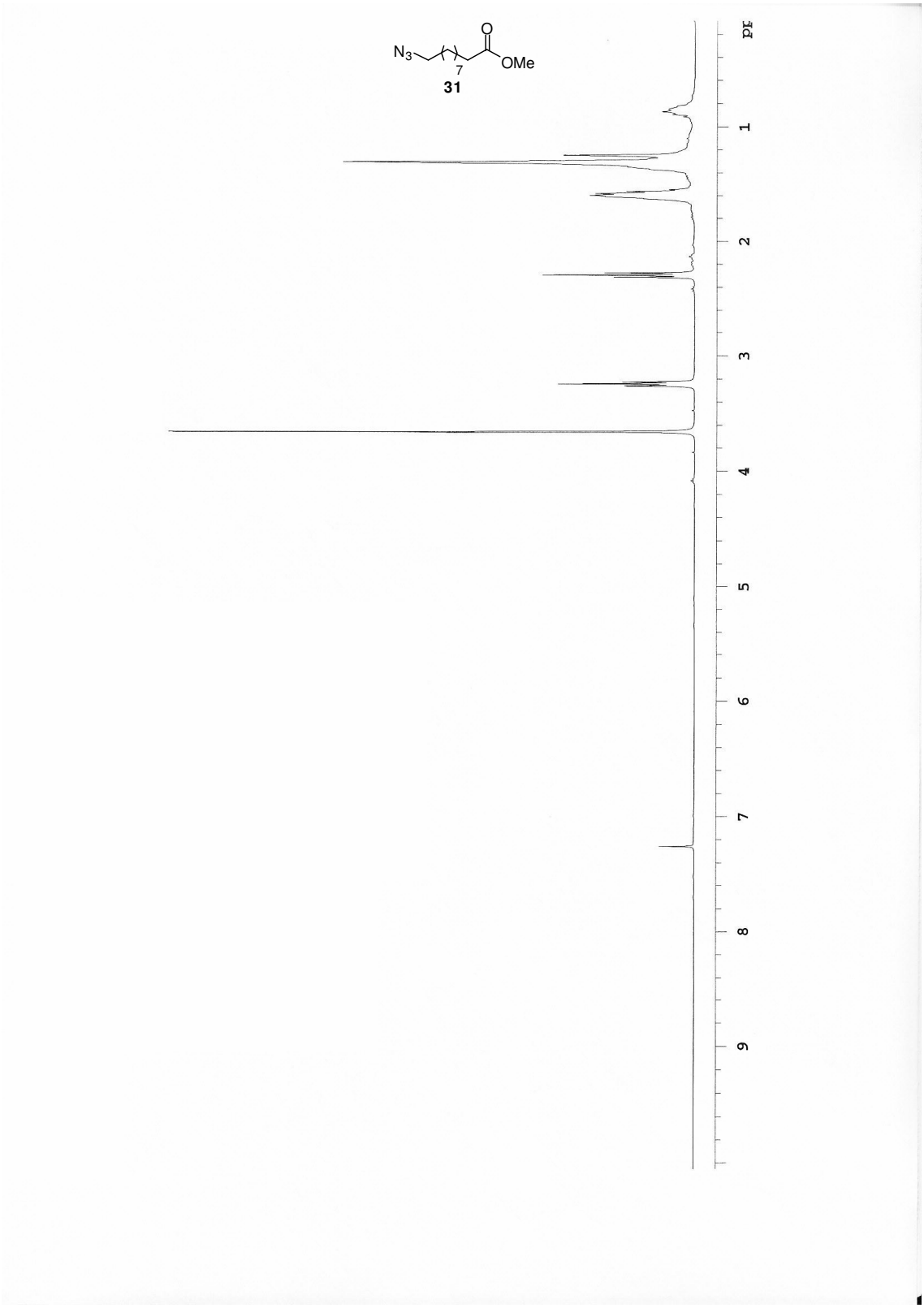

O
8
6
OMe
27
PPM
1
2
3
4
5
6
7
8
9

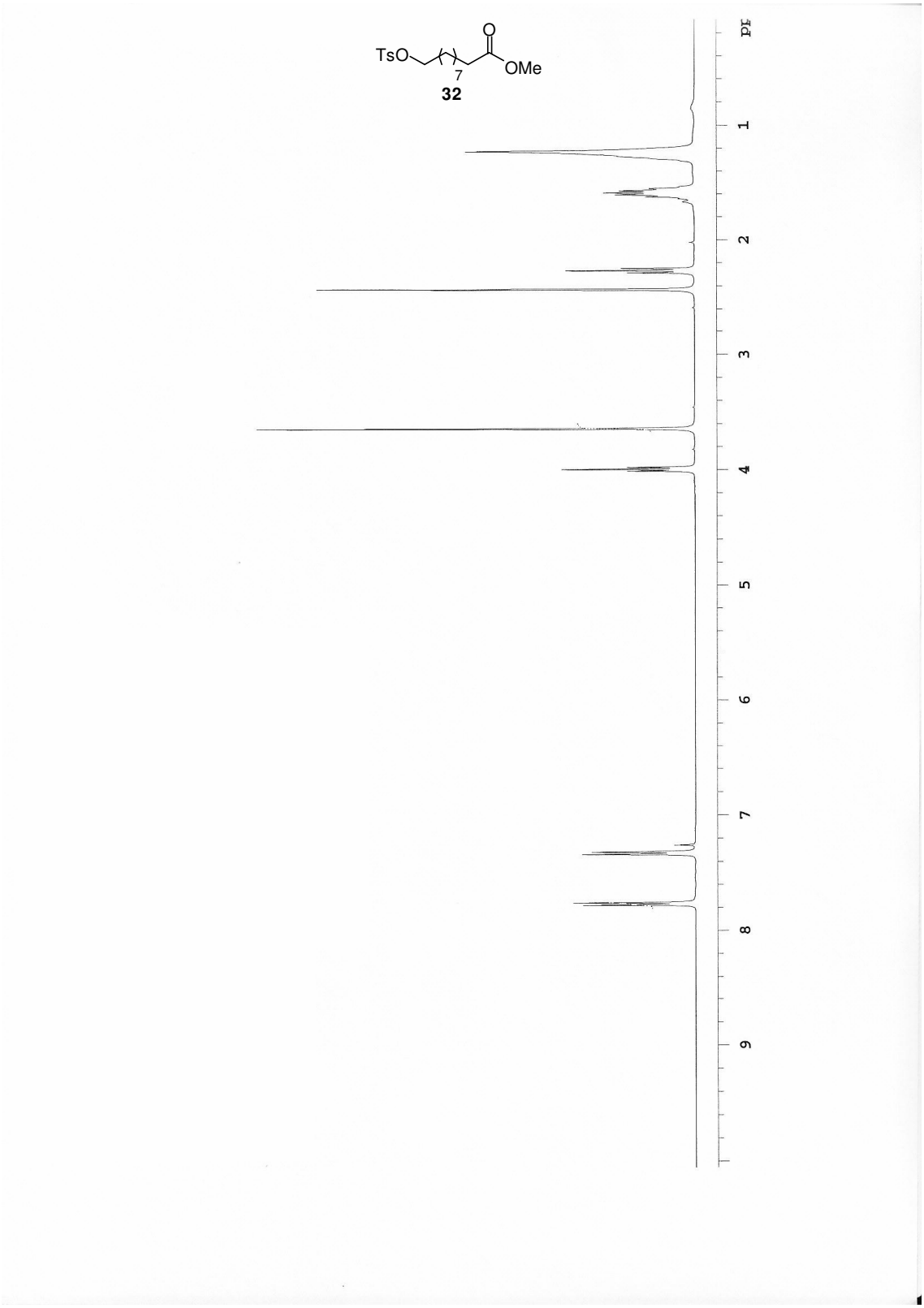

SP2-51-proton
SP-47
28
OMe
O
3
3
110
100
90
80
70
60
50
40
30
20
10
0
-10
10.0
9.5
9.0
8.5
8.0
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
0.5
0.0
f1 (ppm)

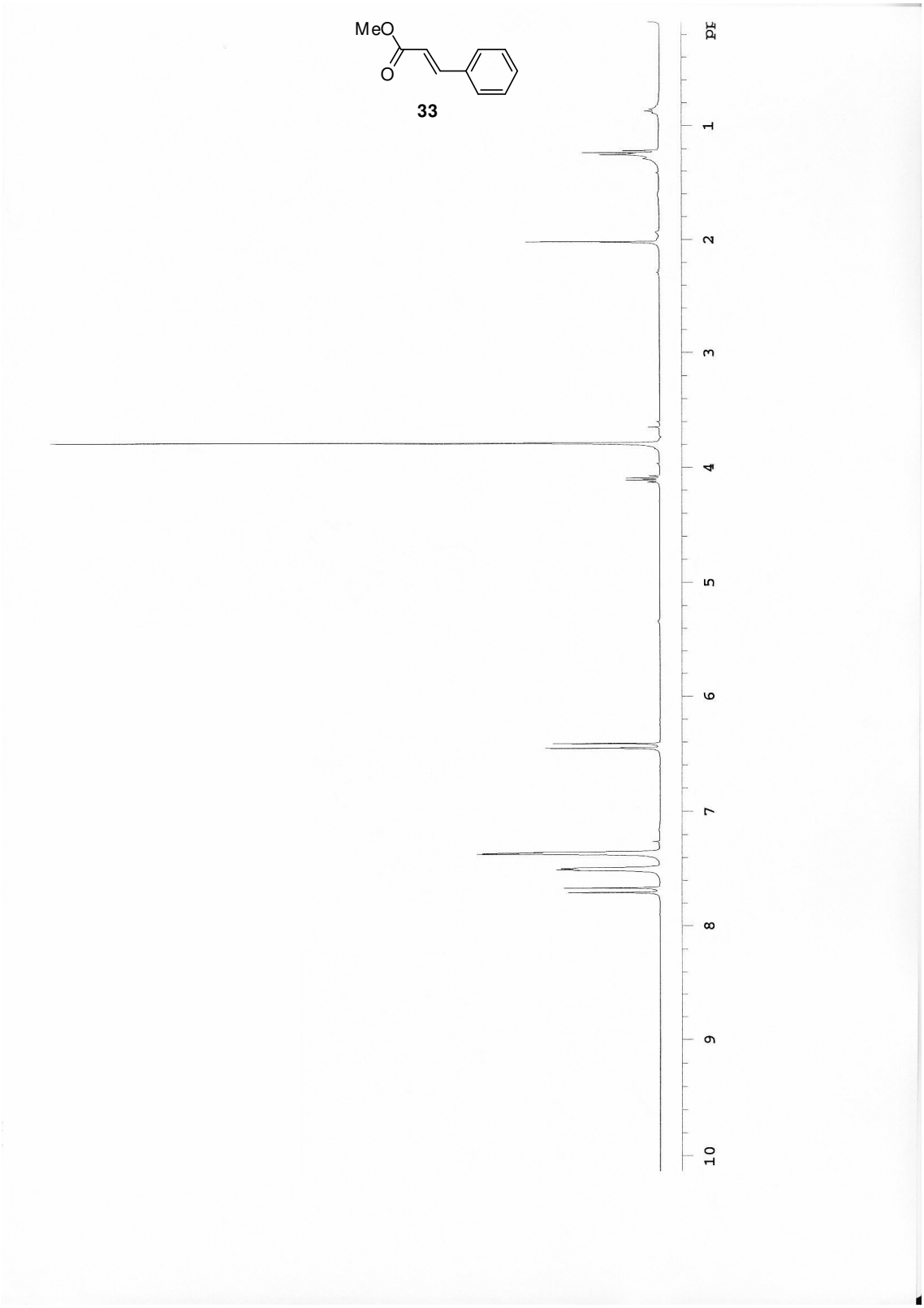

SP2-51-carbono
SP-47
28
OMe
f1 (ppm)

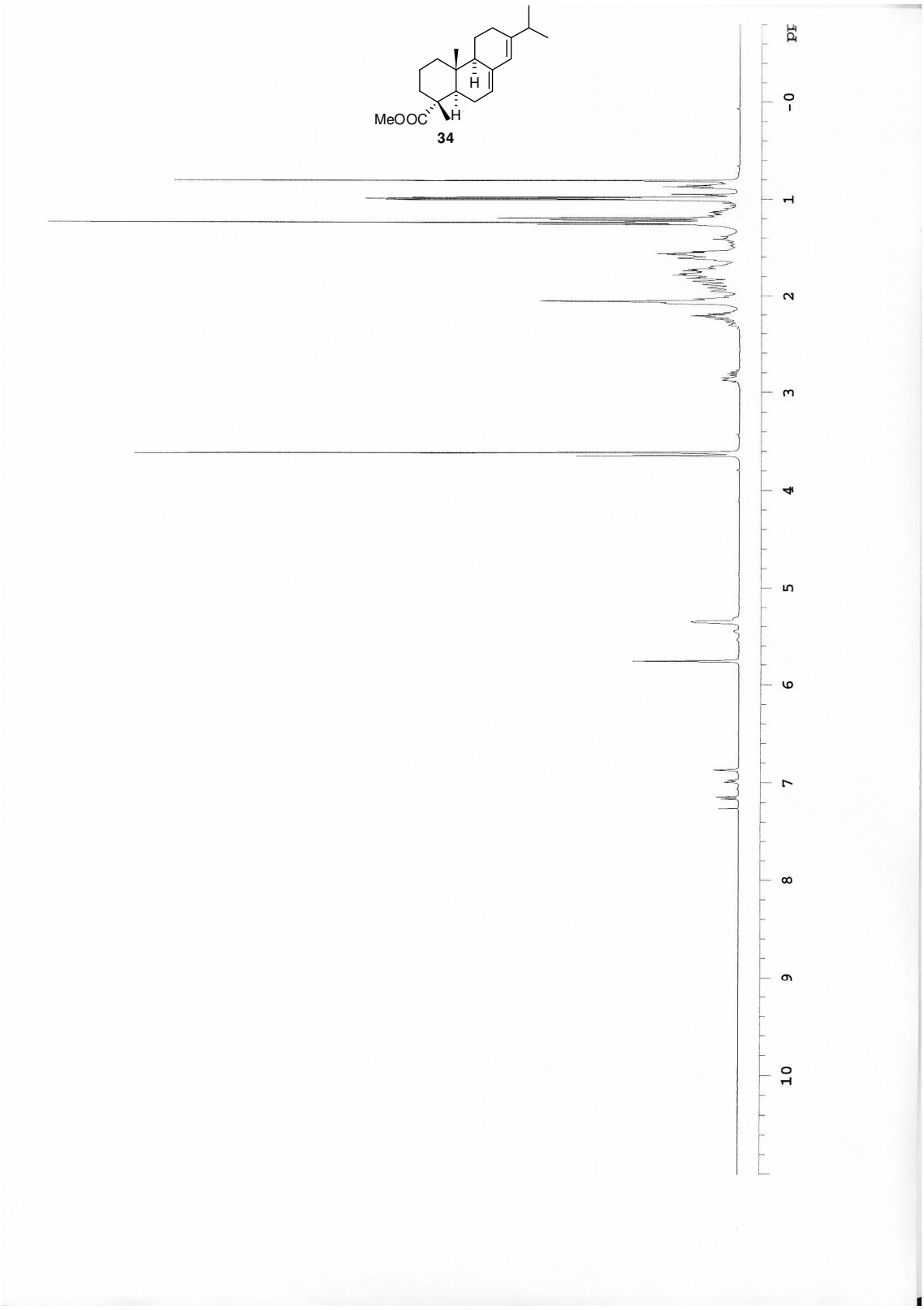

O
OMe
7
29
PPM
-0
1
2
3
4
5
6
7
8
9
10

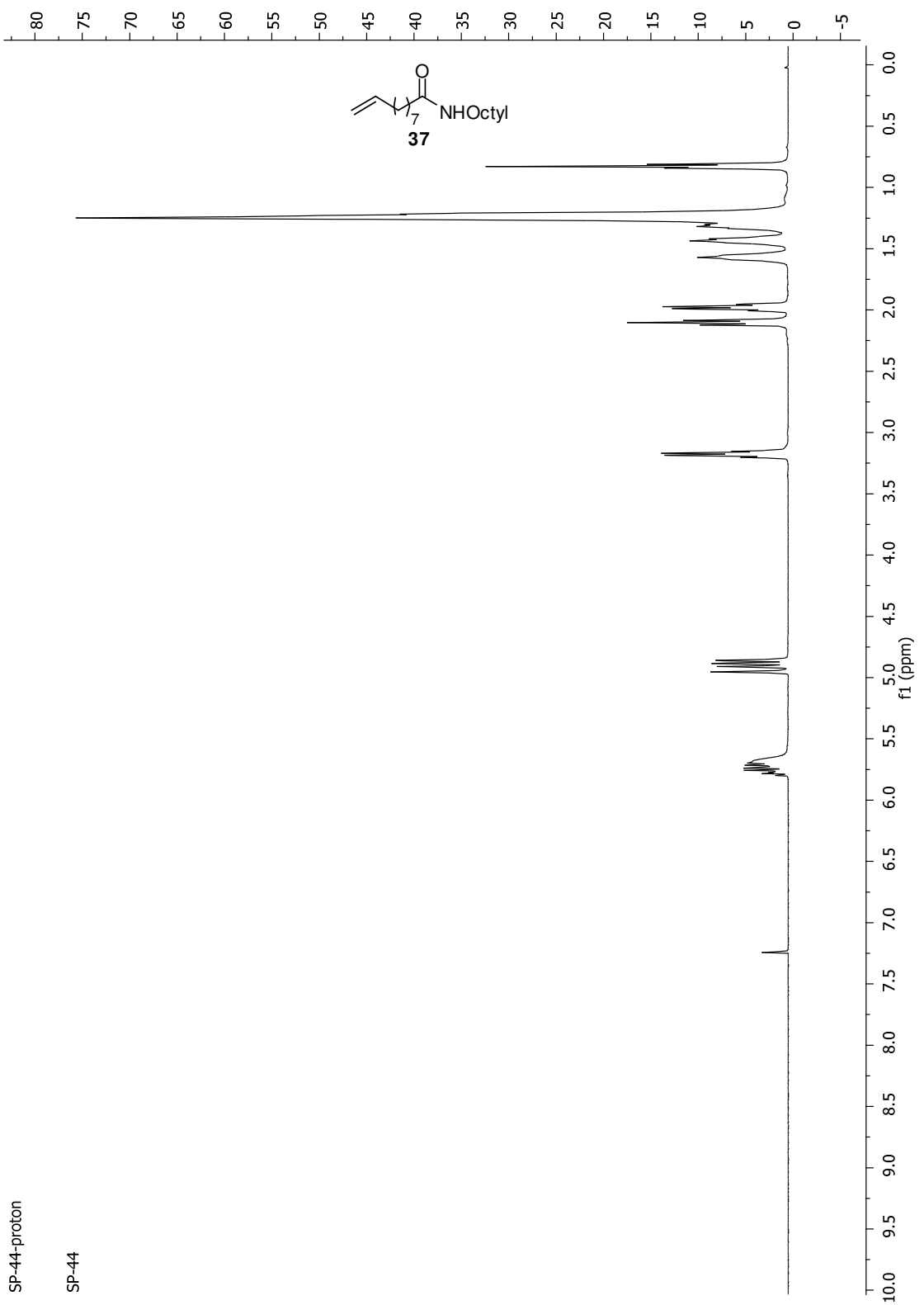

Cl
O
OMe
7
30
PP
-0
1
2
3
4
5
6
7
8
9
10

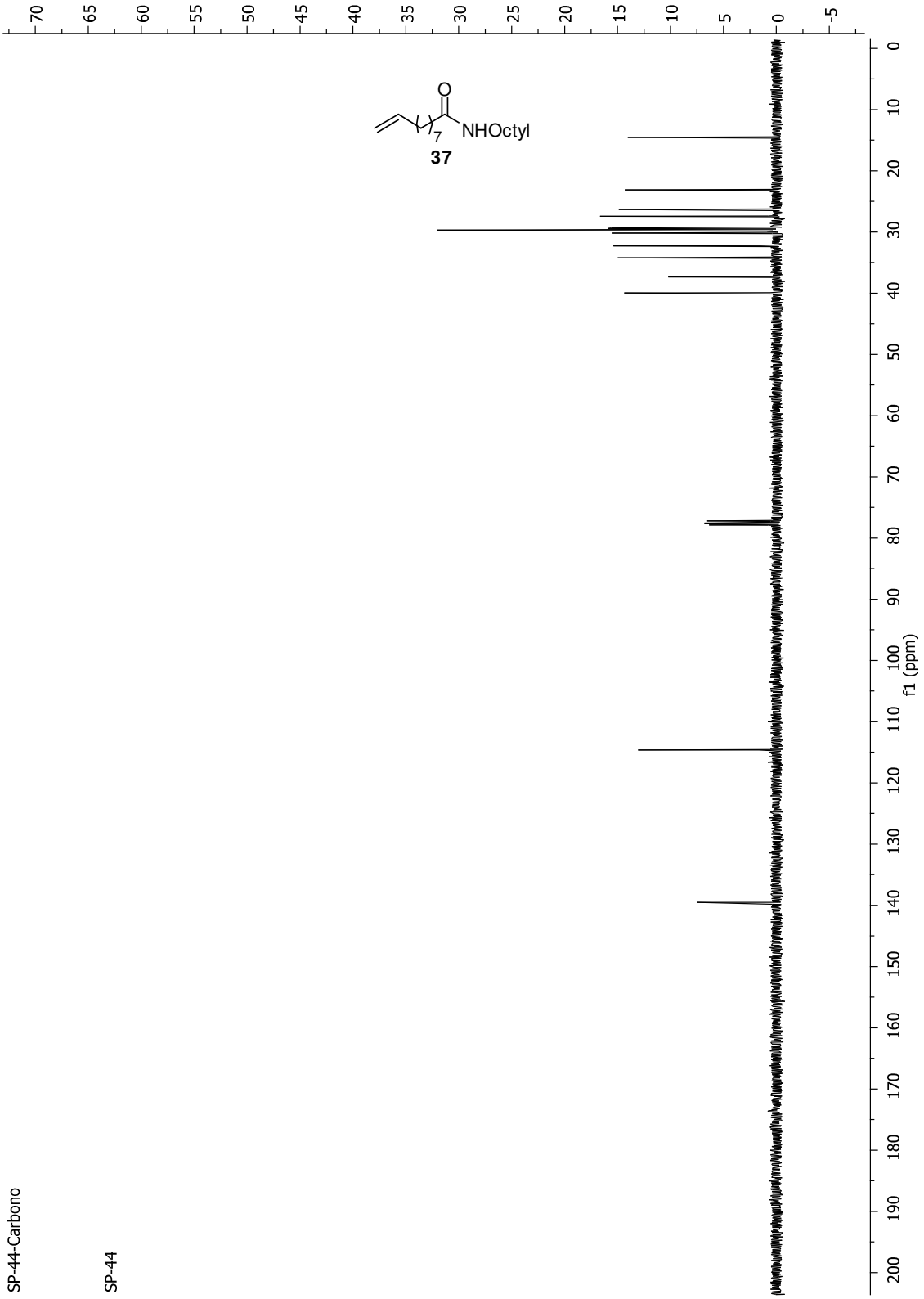

O
N3
7
OMe
31
ppm
1
2
3
4
5
6
7
8
9

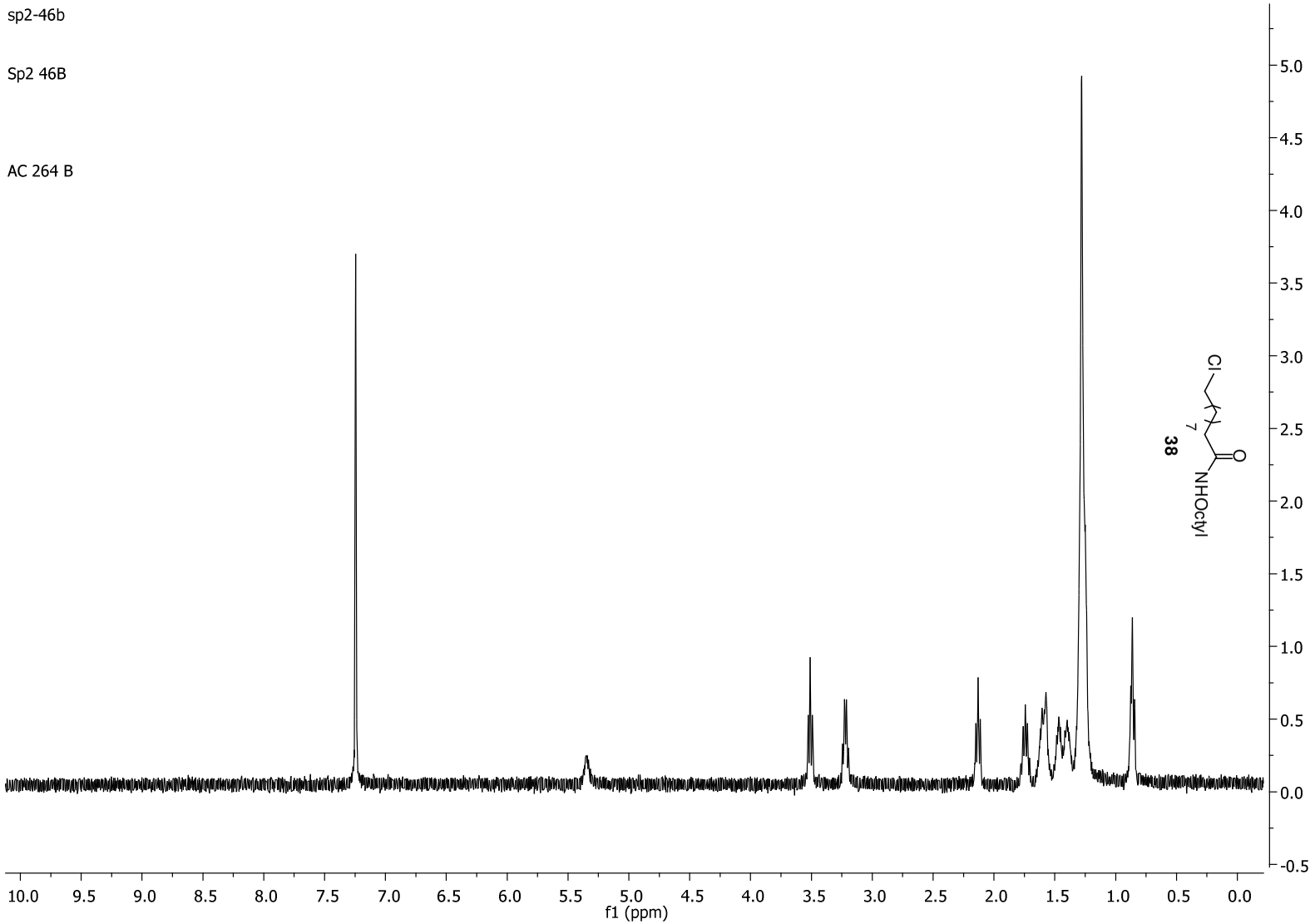

TsO
7
O
OMe
32
ppm
1
2
3
4
5
6
7
8
9

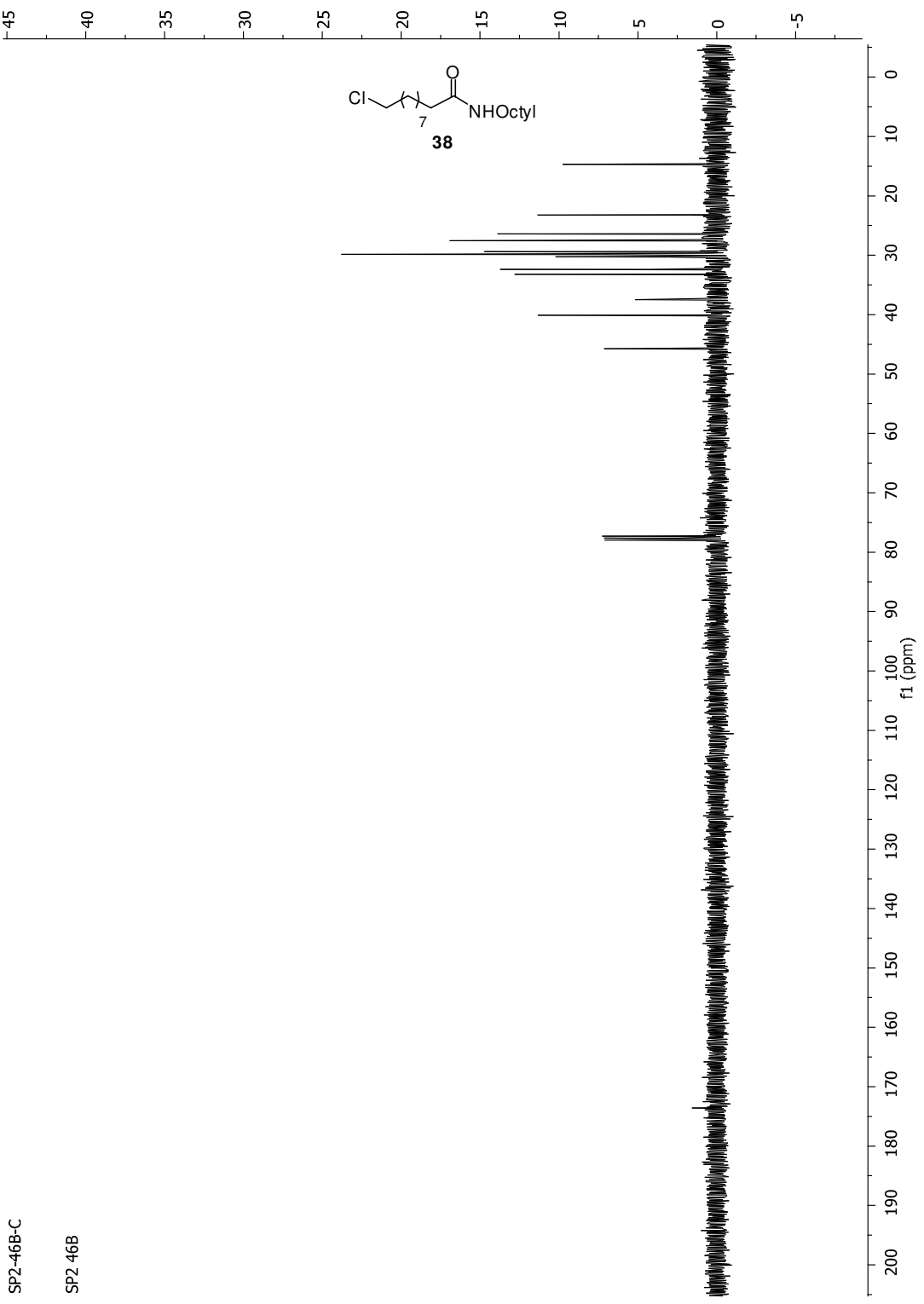

MeO
O
33
ppm
1
2
3
4
5
6
7
8
9
10

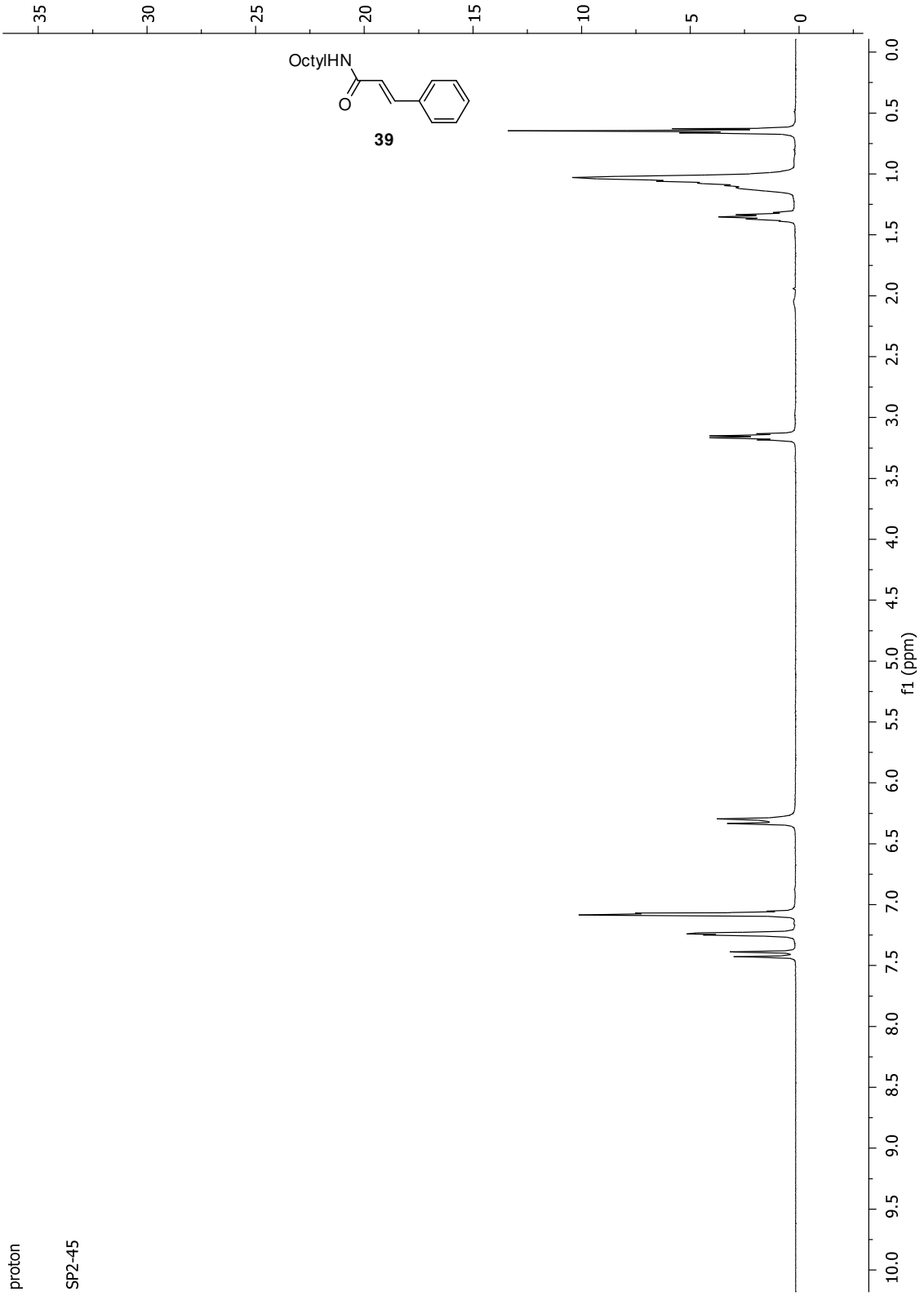

MeOOC
H
H
34
ppm
-0
1
2
3
4
5
6
7
8
9
10

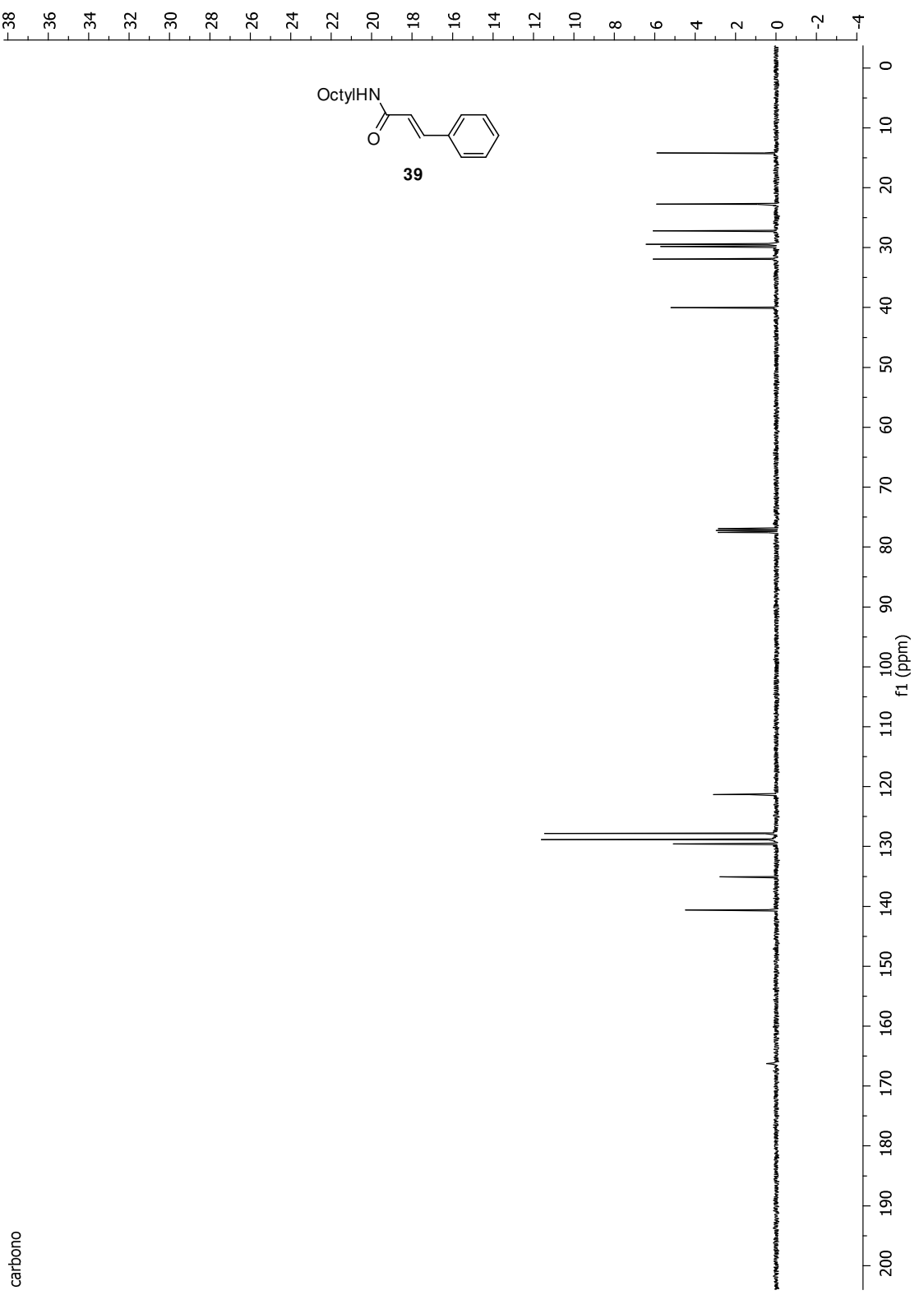

SP-44-proton
SP-44
NHOctyl
37
f1 (ppm)

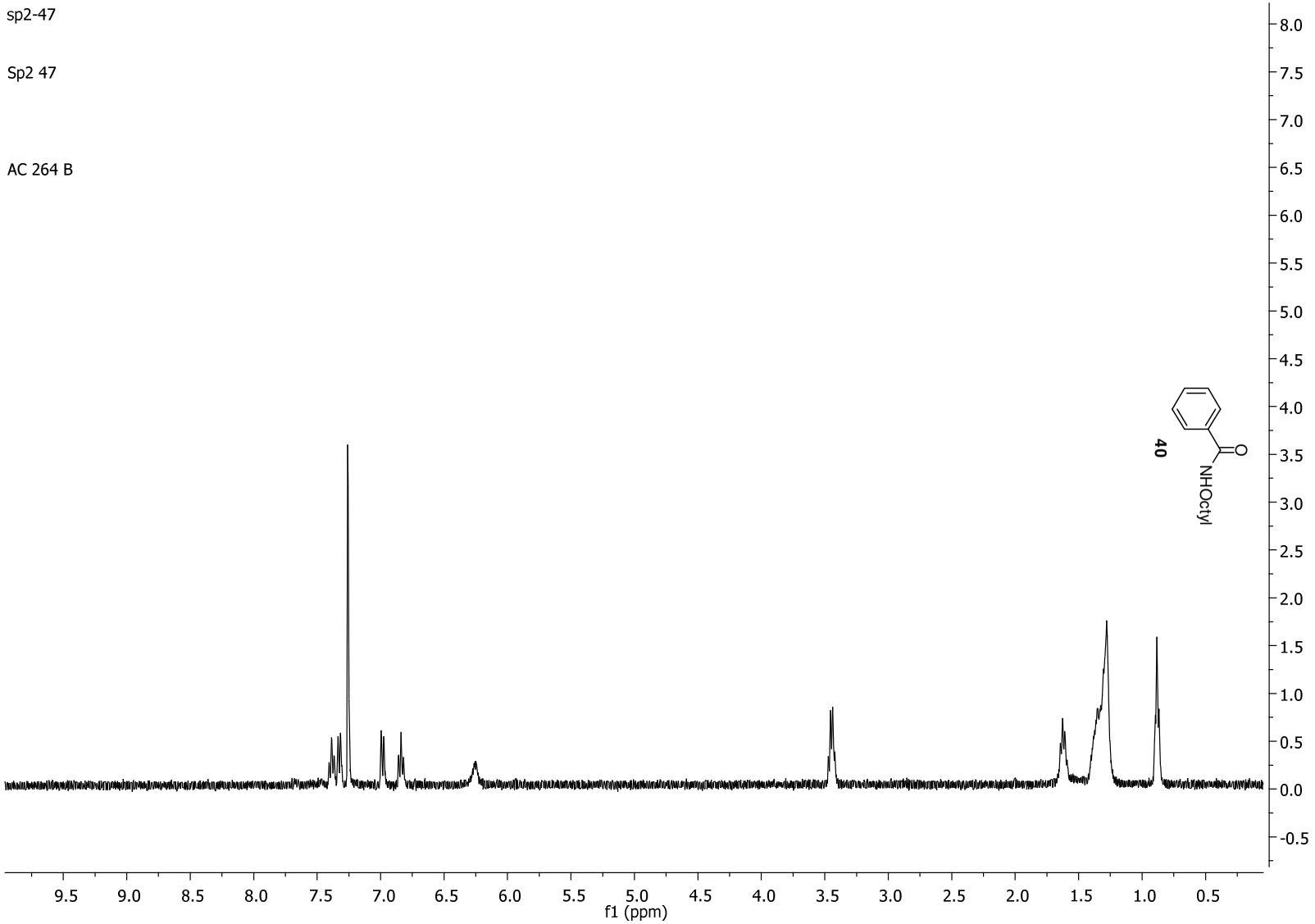

SP-44-Carbono
SP-44
O
NHOctyl
7
37
f1 (ppm)

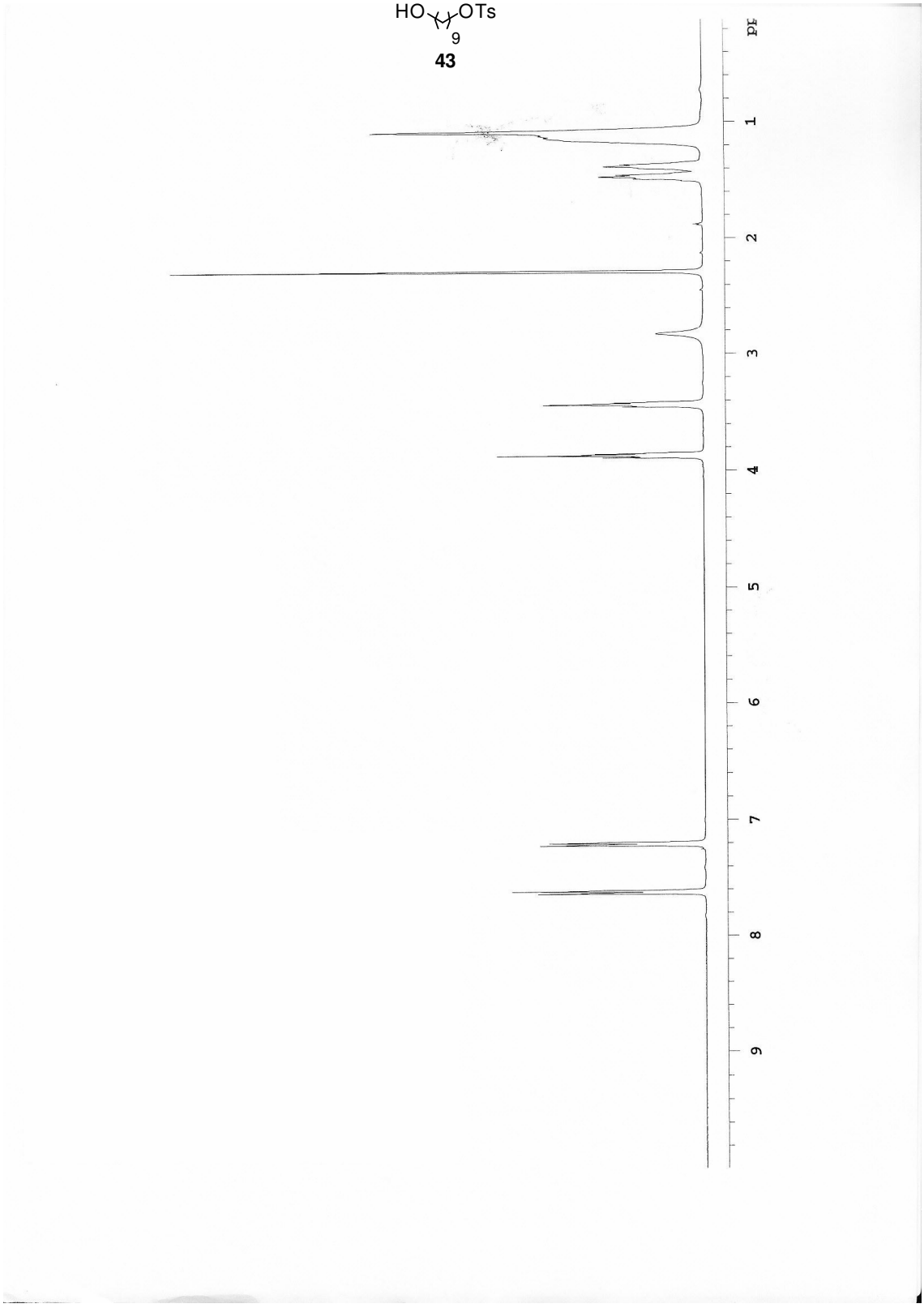

sp2-46b
Sp2 46B
AC 264 B
Cl
7
O
NHOctyl
38
10.0
9.5
9.0
8.5
8.0
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
0.5
0.0
f1 (ppm)
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
0.5
0.0
-0.5

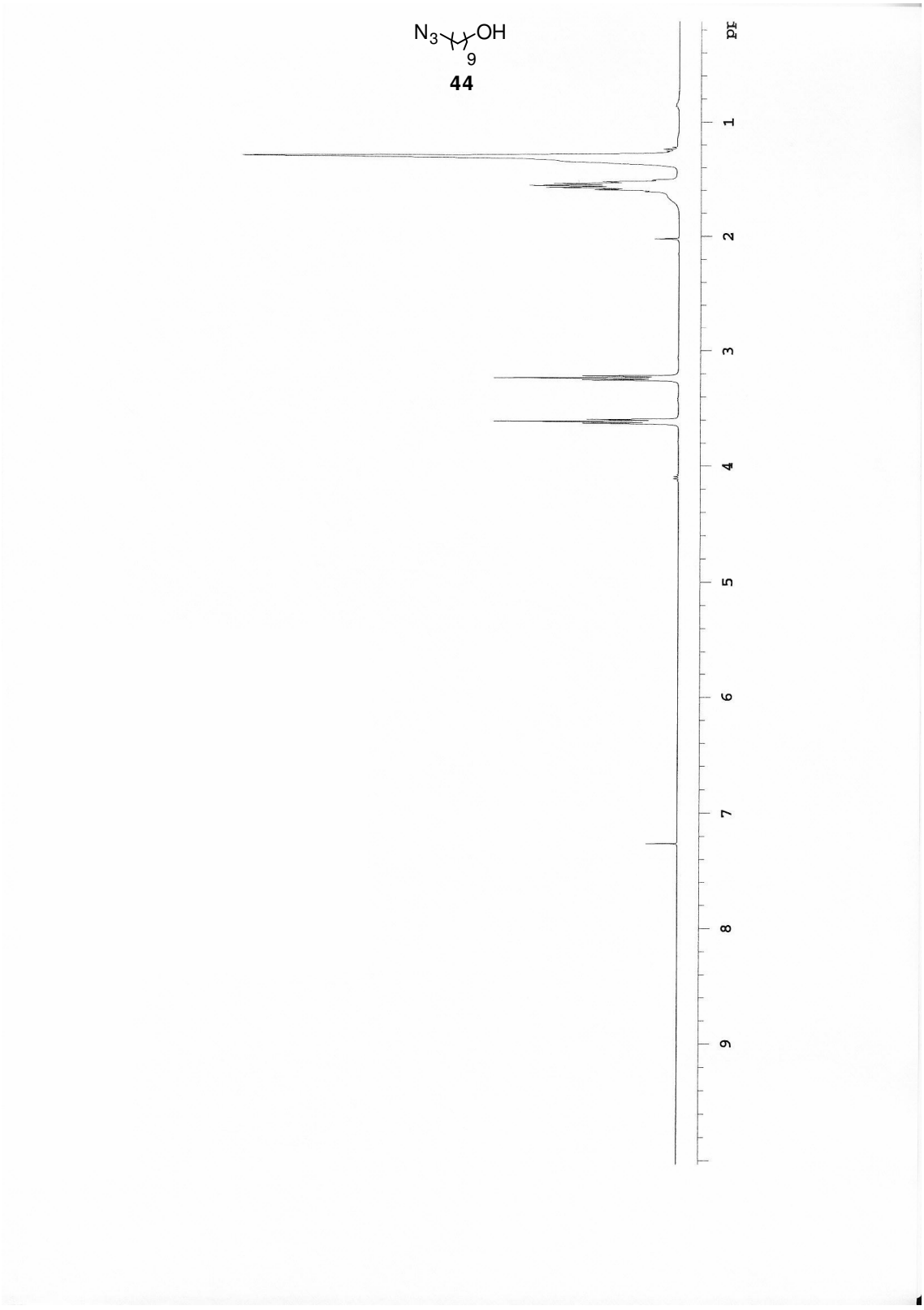

SP2-46B-C
SP2 46B
Cl
7
O
NHOctyl
38
f1 (ppm)


OctylHN
O
39
proton
SP2-45
f1 (ppm)


carbono
OctylHN
O
39
f1 (ppm)


sp2-47
Sp2 47
AC 264 B
40
NHOctyl
f1 (ppm)


HO
OTs
9
43
PF
1
2
3
4
5
6
7
8
9


N3
OH
9
44
ppm
1
2
3
4
5
6
7
8
9