Single isomer trisubstituted olefins from a novel reaction of *E*-β-chloro-α-iodo-α,β-unsaturated esters and amides

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Supplementary Material

General Procedure for the Preparation of Isomeric Trisubstituted Alkenes. (*Z*)-Ethyl 3-phenylbut-2-enoate (4).[1] A solution of (*E*)-ethyl 3-phenylbut-2-enoate **3** (25 mg, 0.22 mmol) in CH_2Cl_2 (2mL) was photolyzed using a UV lamp (λ = 350 nm) for 3 h. The CH_2Cl_2 was removed *in vacuo* to

(1) Mueller, A.J.; Jennings, M.P. Org. Lett. 2007, 9, 5327.

afford a mixture of **3** and **4**[1] (**3:4** = 5:1) (34 mg, 99 %). ^{1}H NMR for compound **4** (400 MHz, acetone-d_6) δ 7.38 – 7.30 (m, 2H), 7.24-7.22 (m, 3H), 5.92 (q, *J* = 1.3 Hz, 1H), 3.92 (q, *J* = 7.0 Hz, 2H), 2.17 (d, *J* = 1.4 Hz, 3H), 1.03 (t, *J* = 7.0 Hz, 3H); ^{13}C NMR for compound **4** (100 MHz, acetone-d_6) δ 166.1(C), 155.1(C), 142.0(C), 129.1(CH), 128.6(CH), 126.2(CH), 116.8(CH), 59.4(CH_2), 26.2(CH_3), 13.8(CH_3); IR(neat) 1712, 1628 cm^{-1}; MS 190.1 (M^+); HRMS calcd for $C_{12}H_{14}O_2$ (M^+) 190.0994, found 190.0989.

(*Z*)-Ethyl 3-(4-methoxyphenyl)but-2-enoate. Prepared from (*E*)-ethyl 3-(4-methoxyphenyl)but-2-enoate **5** (12 mg, 0.05 mmol) using the procedure described above for compound **4**. The product was obtained as an inseparable mixture of *E* and *Z* isomers (*E:Z* = 1:1.25, 12 mg, 99 %). ^{1}H NMR (*Z* isomer, 400 MHz, acetone-d_6) δ 7.22 – 7.20 (m, 2H), 6.90 – 6.87 (m, 2H), 5.86 (q, *J* = 1.2 Hz, 1H), 3.96 (q, *J* = 7.2 Hz, 2H), 3.81 (s, 3H), 2.16 (d, *J* = 1.2 Hz, 3H), 1.08 (t, *J* = 7.2 Hz, 3H); ^{13}C NMR (*Z* isomer, 100 MHz, acetone-d_6) δ 167.2 (C), 161.5 (C), 155.8 (C), 134.5 (C), 130.6 (CH), 118.7 (CH), 114.9 (CH), 60.9 (CH_2), 56.5 (CH_3), 27.9 (CH_3), 15.4 (CH_3); IR (neat) 1711, 1627 cm^{-1}; MS 220.1(M^+); HRMS calcd for $C_{13}H_{16}O_3$ (M^+) 220.1099, found 220.1106.

(*Z*)-Ethyl 3-(3-methoxyphenyl)but-2-enoate. Prepared from (*E*)-ethyl 3-(3-methoxyphenyl)but-2-enoate **6** (18 mg, 0.08 mmol) using a procedure similar to that described above for compound **4**. the title compound was after separation by column chromatography (7 mg, 39 %). ^{1}H NMR (400 MHz, acetone-d_6) δ 7.24 (dd, *J* = 8.2, 0.3 Hz, 1H), 6.86 (ddd, *J* = 8.3, 3.5, 1.2 Hz, 1H), 6.79 – 6.77 (m, 2H), 5.90 (q, 1.4 Hz, 1H), 3.94 (q, *J* = 7.2 Hz, 2H), 3.79 (s, 3H), 2.16 (d, *J* = 1.5 Hz, 3H), 1.05 (t, *J* = 7.2 Hz, 3H); ^{13}C NMR (100 MHz, acetone-d_6) δ 167.1 (C), 161.4 (C), 156.3 (C), 144.5 (C), 130.9 (CH), 121.3 (CH), 119.8 (CH), 114.9 (CH), 114.9 (CH), 61.2 (CH_2), 56.7 (CH_3), 28.1 (CH_3), 15.5 (CH_3); IR (neat) 1725, 1642 cm^{-1}; MS 220.1 (M^+); HRMS calcd for $C_{13}H_{16}O_3$ (M^+) 220.1099, found 220.1087.

(*Z*)-Ethyl 3-(2-methoxyphenyl)but-2-enoate. Prepared from (*E*)-ethyl 3-(2-methoxyphenyl)but-2-enoate **7** (24 mg, 0.11 mmol) using a procedure similar to that described above for **4** that afforded an inseparable mixture of *E* and *Z* (*E:Z* = 1:3.7) isomers (23 mg, 96 %). ^{1}H NMR (*Z* isomer, 400 MHz, acetone-d_6) δ 7.26 – 7.23 (m, 1H), 7.00 (dd, *J* = 7.6, 1.6 Hz, 1H), 6.98 (d, *J* = 8.4 Hz, 1H), 6.92 – 6.88 (m, 1H), 5.91 (q, 1.2 Hz, 1H), 3.89 (q, *J* = 7.2 Hz, 2H), 3.77 (s, 3H), 2.11 (d, *J* = 1.2 Hz, 3H), 1.01 (t, *J*

= 7.2 Hz, 3H); ^{13}C NMR (*Z* isomer, 100 MHz, acetone-d_6) δ 166.7 (C), 157.5 (C), 157.4 (C), 132.3 (C), 130.4 (CH), 129.9 (CH), 121.9 (CH), 120.7 (CH), 112.7 (CH), 60.7 (CH_2), 56.8 (CH_3), 27.2 (CH_3), 15.3 (CH_3); IR (neat) 1714, 1645 cm^{-1}; MS 220.1 (M^+); HRMS calcd for $C_{13}H_{16}O_3$ (M^+) 220.1099, found 220.1088.

(*Z*)-Ethyl 3-*p*-tolylbut-2-enoate. Prepared from (*E*)-ethyl 3-*p*-tolylbut-2-enoate **8** (12 mg, 0.06 mmol) using a procedure similar to that described above for **4** that afforded an inseparable mixture of *E* and *Z* isomers (*E:Z* = 2:1) (12 mg, 99 %). ^{1}H NMR (*Z* isomer, 400 MHz, acetone-d_6) δ 7.16 – 7.11 (m, 4H), 5.88 (q, *J* = 1.2 Hz, 1H), 3.94 (q, *J* = 7.2 Hz, 2H), 2.32 (s, 3H), 2.15 (d, *J* = 1.2 Hz, 3H), 1.11 (t, *J* = 7.2 Hz, 3H); ^{13}C NMR (*Z* isomer, 100 MHz, acetone-d_6) δ 167.0 (C), 156.3 (C), 139.8 (C), 139.1 (C), 130.1 (CH), 130.0 (CH), 119.2 (CH), 60.9 (CH_2), 28.0 (CH_3), 22.2 (CH_3), 15.3 (CH_3); IR (neat) 1713, 1628 cm^{-1}; MS 204.1 (M^+); HRMS calcd for $C_{13}H_{16}O_2$ (M^+) 204.1150, found 204.1165.

(*Z*)-Ethyl 3-(4-fluorophenyl)but-2-enoate. Prepared from (*E*)-ethyl 3-(4-fluorophenyl)but-2-enoate **9** (7.5 mg, 0.036 mmol) using a procedure similar to that described above for **4** that afforded an inseparable mixture of *E* and *Z* isomers (*E:Z* = 1.6:1) (7.5 mg, 99 %). ^{1}H NMR (Z isomer, 400 MHz, acetone-d_6) δ 7.31 – 7.27 (m, 2H), 7.12-7.08 (m, 2H), 5.93 (q, *J* = 1.2 Hz, 1H), 3.95 (q, *J* = 7.2 Hz, 2H), 2.17 (d, *J* = 1.2 Hz, 3H), 1.07 (t, *J* = 7.2 Hz, 3H); ^{13}C NMR (*Z* isomer, 100 MHz, acetone-d_6) δ 166.8 (C), 165.4 (d, *J* = 245Hz, C), 155.5 (C), 138.9 (d, *J* = 3.3Hz, C), 131.1 (d, *J* = 8.1Hz, CH), 119.8 (CH), 116.2 (d, *J* = 21.5Hz, CH), 61.0 (CH_2), 28.0 (CH_3), 15.3 (CH_3); IR (neat) 1715, 1632 cm^{-1}; MS 208.1 (M^+); HRMS calcd for $C_{12}H_{13}FO_2$ (M^+) 208.0900, found 208.0887.

(*Z*)-Ethyl 3-(thiophen-2-yl)but-2-enoate. Prepared from (*E*)-ethyl 3-(thiophen-2-yl)but-2-enoate **11** (17 mg, 0.09 mmol) using a procedure similar to that described above for compound **4**, that afforded the *Z* isomer after separation by column chromatography (6 mg, 35 %). ^{1}H NMR (400 MHz, acetone-d_6) δ 7.56-7.54 (m, 2H), 7.06 (dd, *J* = 5.1, 3.8 Hz, 1H), 5.88 (dd, *J* = 1.3, 1.3 Hz, 1H), 4.09 (q, *J* = 7.1 Hz, 2H), 2.28 (dd, *J* = 1.4, 1.4 Hz, 3H), 1.19 (t, *J* = 7.1 Hz, 3H); ^{13}C NMR (100 MHz, acetone-d_6) δ 165.4 (C), 143.4 (C), 140.2 (C), 129.4 (CH), 127.8 (CH), 126.7 (CH), 116.4 (CH), 59.5 (CH_2), 26.4 (CH_3),

13.6 (CH_3); IR (neat) 1705, 1610 cm^{-1}; MS 196.1 (M^+); HRMS calcd for $C_{10}H_{12}O_2S$ (M^+) 196.0558, found 196.0556.

(*Z*)-Ethyl 3-(naphthalen-1-ylbut-2-enoate. Prepared from (*E*)-ethyl 3-(naphthalen-1-ylbut-2-enoate **12** (21 mg, 0.09 mmol) using a procedure similar to that described above for compound **4**, that afforded the *Z* isomer after separation by column chromatography (15 mg, 71 %). ^{1}H NMR (400 MHz, acetone-d_6) δ 7.91-7.76 (m, 3H), 7.50-7.43 (m, 3H), 7.20 (dd, *J* = 7.0, 1.1 Hz, 1H), 6.22 (q, *J* = 1.4 Hz, 1H), 3.71 (q, *J* = 7.1 Hz, 2H), 2.27 (d, *J* = 1.4 Hz, 3H), 0.76 (t, *J* = 7.1 Hz, 3H); ^{13}C NMR (100 MHz, acetone-d_6) δ 164.4 (C), 154.3 (C), 140.0 (C), 133.6 (C), 130.1 (C), 128.3 (CH), 127.1 (CH), 125.9 (CH), 125.6 (CH), 125.3 (CH), 124.7 (CH), 123.3 (CH), 120.1 (CH), 58.9 (CH_2), 26.9 (CH_3), 13.2 (CH_3); IR (neat) 1726, 1647 cm^{-1}; MS 240.1 (M^+); HRMS calcd for $C_{16}H_{16}O_2$ (M^+) 240.1150, found 240.1150.

(*Z*)-Ethyl 3-(naphthalen-2-ylbut-2-enoate. Prepared from (*E*)-ethyl 3-(naphthalen-2-ylbut-2-enoate **13** (16 mg, 0.07 mmol) using a procedure similar to that described above for **4** that afforded an inseparable mixture of *E* and *Z* isomers (*E:Z* = 1:10) (16 mg, 99 %). ^{1}H NMR (*Z* isomer, 400 MHz, acetone-d_6) δ 7.91-7.85 (m, 3H), 7.76 (d, *J* = 1.3 Hz, 1H), 7.52-7.48 (m, 2H), 7.38 (dd, *J* = 8.5, 1.8 Hz, 1H), 6.02 (dd, *J* = 1.4, 1.4 Hz, 1H), 3.92 (q, *J* = 7.1 Hz, 2H), 2.28 (d, *J* = 1.5 Hz, 3H), 0.99 (t, *J* = 7.1 Hz, 3H); ^{13}C NMR (*Z* isomer, 100 MHz, acetone-d_6) δ 165.1 (C), 154.6 (C), 138.6 (C), 133.2 (C), 133.0 (C), 128.0 (CH), 127.6 (CH), 127.1 (CH), 126.0 (CH), 126,0 (CH), 125.8 (CH), 125.8 (CH), 118.0 (CH), 59.1 (CH_2), 26.2 (CH_3), 13.4 (CH_3); IR (neat) 1709, 1622 cm^{-1}; MS 240.1 (M^+); HRMS calcd for $C_{16}H_{16}O_2$ (M^+) 240.1150, found 240.1150.

(*Z*)-Ethyl 3-cyclohexyl-3-phenylacrylate. Prepared from (*E*)-Ethyl 3-cyclohexyl-3-phenylacrylate **15** (15 mg, 0.07 mmol) using a procedure similar to that described above for **4** that afforded an inseparable mixture of *E* and *Z* isomers (*E:Z* = 3:1) (15 mg, 99 %). ^{1}H NMR (*Z* isomer, 400 MHz, acetone-d_6) δ 7.34 – 7.28 (m, 3H), 7.12 – 7.09 (m, 2H), 5.81 (d, *J* = 0.9 Hz, 1H), 3.87 (q, *J* = 7.1 Hz, 2H), 2.32 (ddt, *J* = 11.6, 10.6, 2.8 Hz, 1H), 1.83-1.63 (m, 5H), 1.20-1.11 (m, 5H), 0.98 (t, *J* = 7.1 Hz, 3H); ^{13}C NMR (*Z* isomer, 100 MHz, acetone-d_6) δ 165.3 (C), 164.9 (C), 143.2 (C), 129.4 (CH), 129.3 (CH), 128.8 (CH),

117.9 (CH), 60.9 (CH_2), 48.9 (CH), 33.5 (CH_2), 28.1 (CH_2), 27.8 (CH_2), 15.3 (CH_3); IR (neat) 1715, 1629 cm^{-1}; MS 258.2 (M^+); HRMS calcd for $C_{17}H_{22}O_2$ (M^+) 258.1620, found 258.1626.

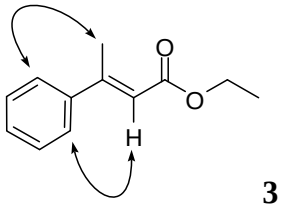
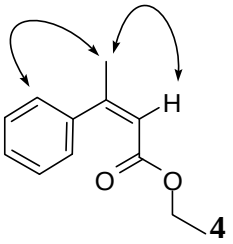
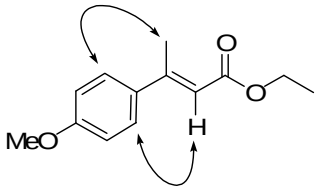
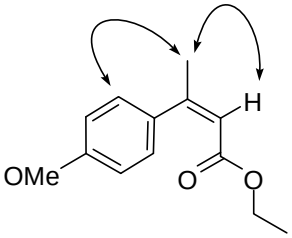
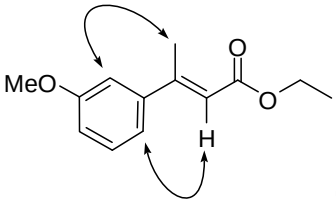
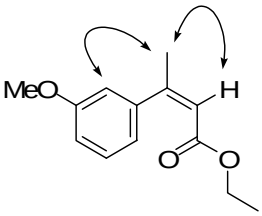
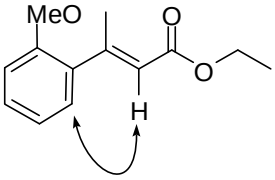
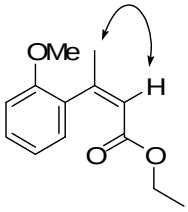
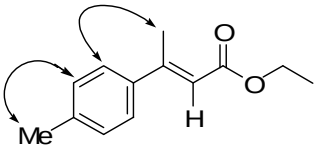
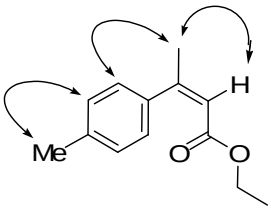
(*Z*)-Ethyl 5-(*tert*-butyldimethylsiloxy)-3-phenylpent-2-enoate. Prepared from (*E*)-Ethyl 5-(*tert*-butyldimethylsiloxy)-3-phenylpent-2-enoate **17** (13 mg, 0.05 mmol) using a procedure similar to that described above for compound **4** that afforded an inseparable mixture of *E* and *Z* isomers (*E:Z* = 2:1) (13 mg, 99 %). ^{1}H NMR (*Z* isomer, 400 MHz, acetone-d_6) δ 7.34–7.30 (m, 3H), 7.23–7.20 (m, 2H), 5.94 (s, 1H), 3.93 (q, *J* = 7.2 Hz, 2H), 3.65 (dd, *J* = 6.4, 6.4 Hz 2H), 2.68 (dd, *J* = 6.3, 6.3 Hz, 2H), 1.02 (t, *J* = 7.2 Hz, 3H), 0.89 (s, 9H), 0.01 (s, 6H); ^{13}C NMR (*Z* isomer, 100 MHz, acetone-d_6) δ 167.0 (C), 156.5 (C), 141.8 (C), 129.5 (CH), 129.4 (CH), 129.3 (CH), 120.9 (CH), 62.2 (CH_2), 60.9 (CH_2), 44.9 (CH_2), 27.2 (CH_3), 19.7 (CH), 15.3 (CH_3), -4.3 (CH_3); IR (neat) 1714, 1624 cm^{-1}; MS 277.1 (M^+-C_4H_9); HRMS calcd for $C_{15}H_{21}O_3Si(MH^+$-$C_4H_9)$ 278.1338, found 278.1296.

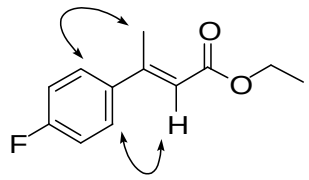
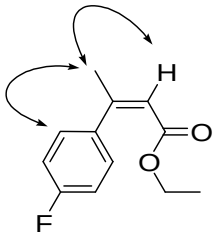
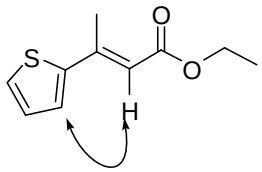
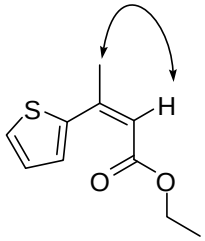
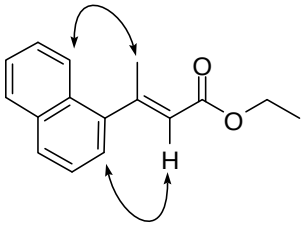
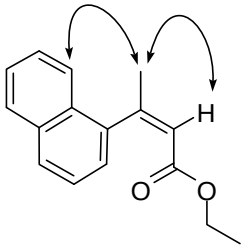
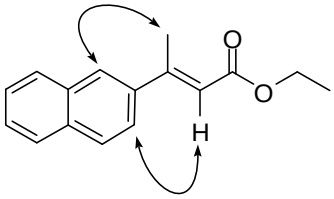
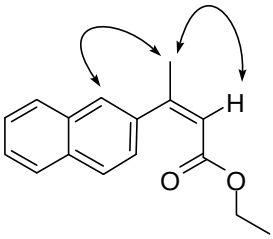
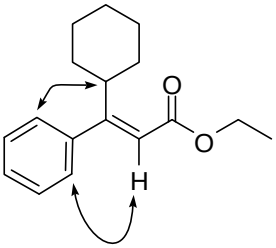
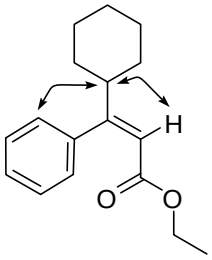
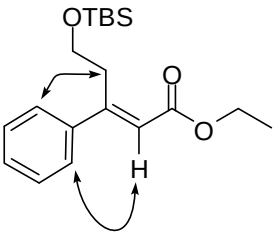
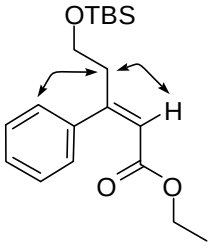
(*Z*)-Ethyl 5-(benzyloxy)-3-phenylpent-2-enoate. Prepared from **(***E*)-ethyl 5-(benzyloxy)-3-phenylpent-2-enoate **19** (37 mg, 0.12 mmol) using a procedure similar to that described above for compound **4** that afforded an inseparable mixture of *E* and *Z* isomers (*E:Z* = 0.7:1) (37 mg, 99 %). ^{1}H NMR (400 MHz, acetone-d_6) δ 7.34–7.18 (m, 10H), 5.97 (s, 1H), 4.45 (s, 2H), 3.91 (q, *J* = 7.1 Hz, 2H), 3.51-3.46 (m, 2H), 2.78 (ddd, *J* = 6.3, 6.3, 1.2 Hz, 2H), 1.02 (t, *J* = 7.1 Hz, 3H); ^{13}C NMR (100 MHz, acetone-d_6) δ 166.9 (C), 157.5 (C), 141.7 (C), 140.7 (C), 130.0 (CH), 129.5 (CH), 129.4 (CH), 129.3 (CH), 129.3 (CH), 129.2 (CH), 120.7 (CH), 74.2 (CH_2), 69.1 (CH_2), 60.0 (CH_2), 41.9 (CH_2), 15.3 (CH_3); IR (neat) 1711, 1626 cm^{-1}; this compound would not give satisfactory mass spectra.

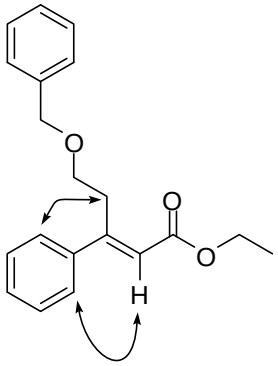
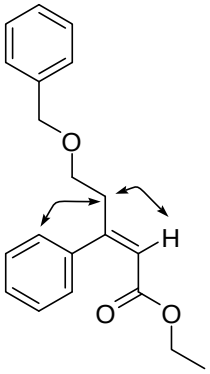
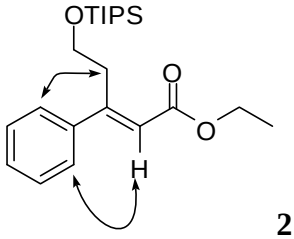
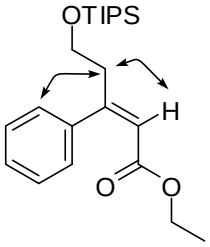
(*Z*)-Ethyl 3-phenyl-5-(triisopropylsiloxy)pent-2-enoate. Prepared from (*E*)-Ethyl 3-phenyl-5-(triisopropylsiloxy)pent-2-enoate **21** (39 mg, 0.10 mmol using a procedure similar to that described above for compound **4** that afforded an inseparable mixture of *E* and *Z* isomers (*E:Z* = 2:1) (39 mg, 99 %). ^{1}H NMR (400 MHz, acetone-d_6) δ 7.37-7.29 (m, 3H), 7.23-7.21 (m, 2H), 5.96 (s, 1H), 3.91 (q, *J* = 7.1 Hz, 2H), 3.74 (dd, *J* = 6.4, 6.4 Hz, 2H), 2.73 (ddd, *J* = 6.4, 6.4, 1.0 Hz, 2H), 1.06-0.99 (m, 24H); ^{13}C NMR (100 MHz, acetone-d_6) δ 166.9 (C), 157.3 (C), 141.3 (C), 129.5 (CH), 129.4 (CH), 129.3 (CH),

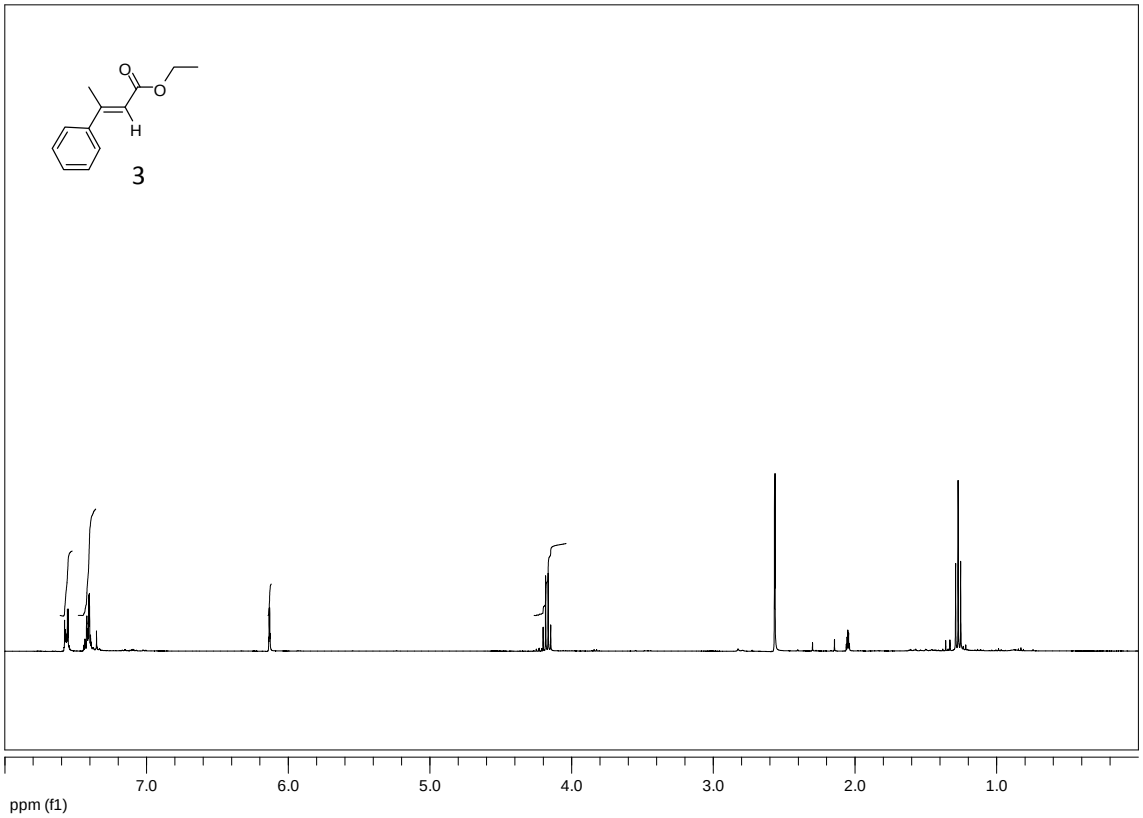
121.0 (CH), 62.7 (CH_2), 60.9 (CH_2), 45.2 (CH_2), 19.3 (CH_3), 15.3 (CH), 13.7 (CH_3); IR (neat) 1715, 1623 cm^{-1}; MS 333.2 (M^+-C_3H_7); HRMS calcd for $C_{19}H_{29}O_3Si$(M^+-C_3H_7), 333.1886, found 333.1868.

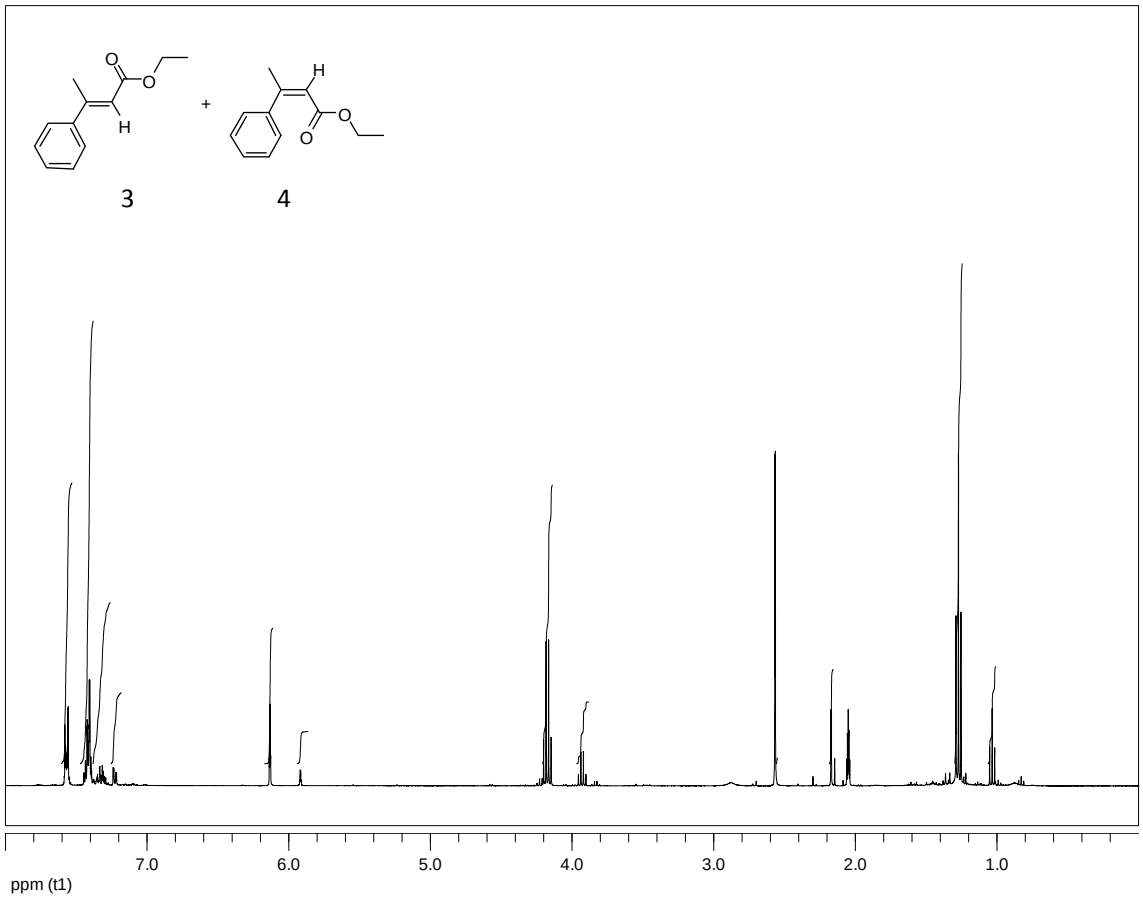
Table 1. NOE analyses of trisubstituted α-aryl-β-chloro-α,β-unsaturated ester olefin templates.

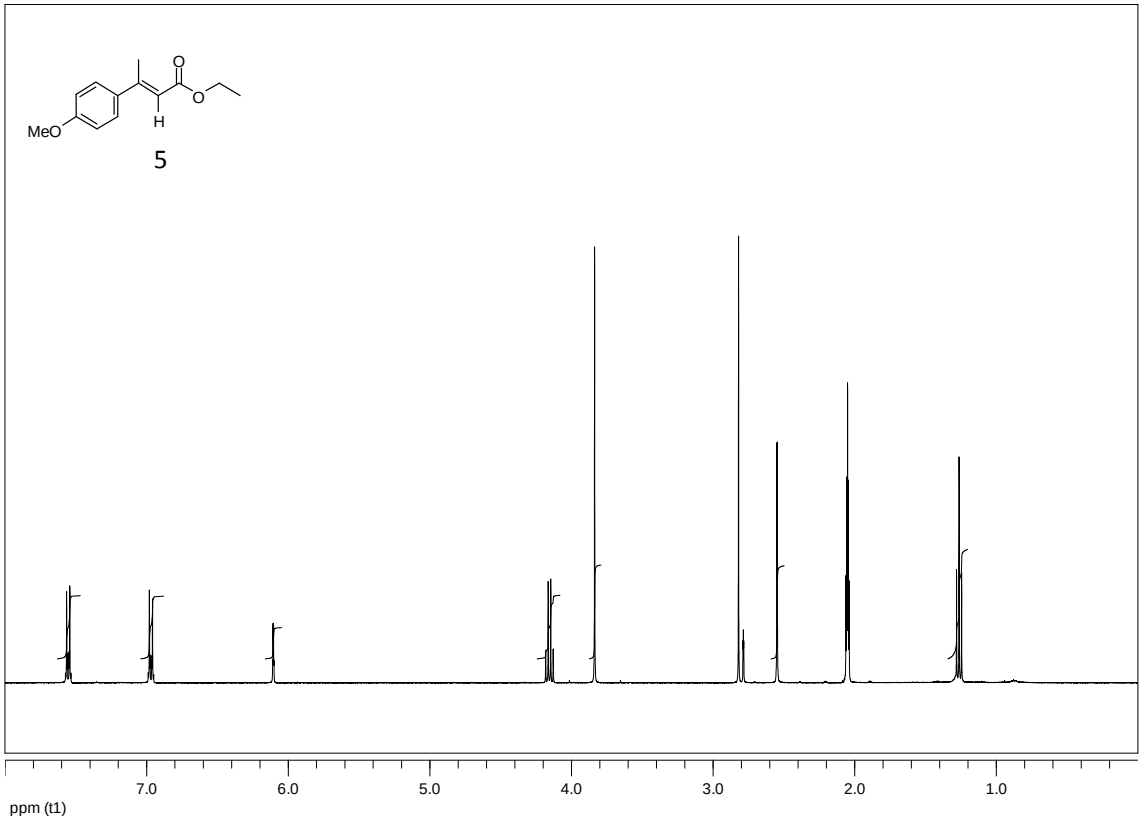
Original Isomer (*E*)	(*Z*) isomer
 **3**	 **4**
 **5**	
 **6**	
 **7**	
 **8**	

 **9**	
 **11**	
 **12**	
 **13**	
 **15**	
 **17**	

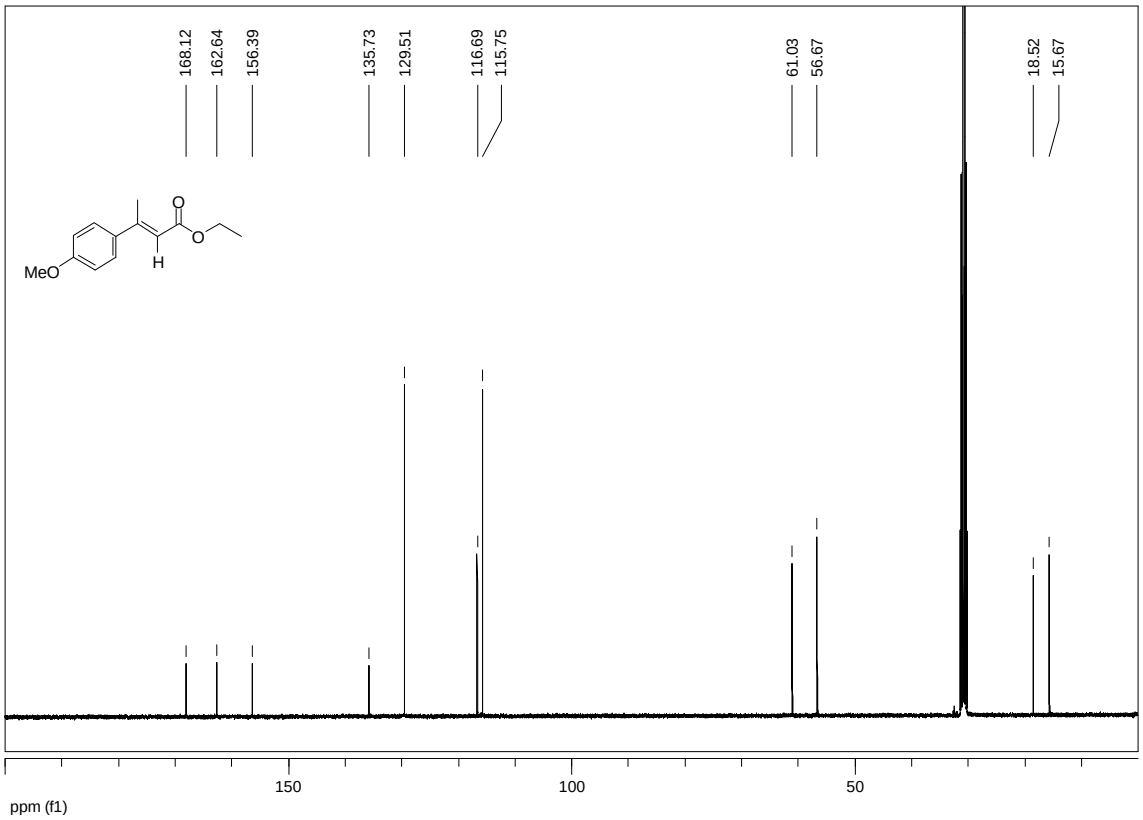
 **19**	
 **21**	

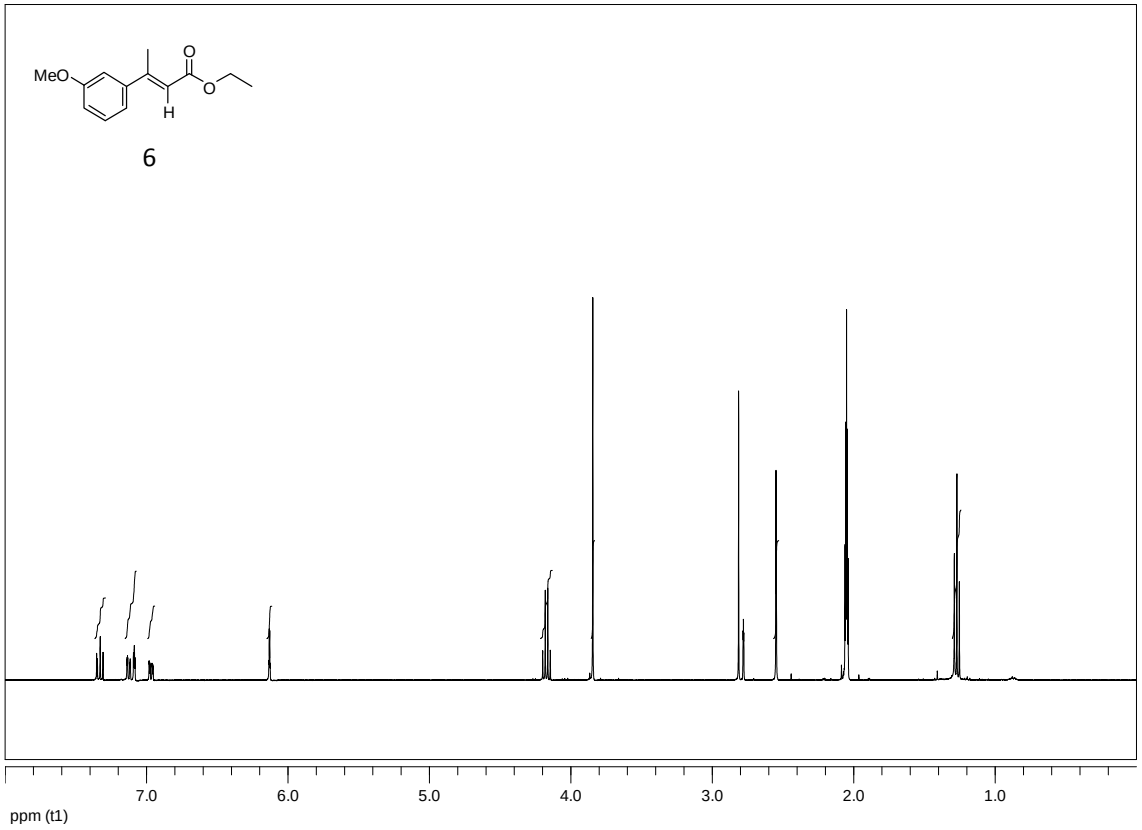

O
O
H
3
7.0
6.0
5.0
4.0
3.0
2.0
1.0
ppm (f1)


O
O
H
+
H
O
O
3
4
7.0
6.0
5.0
4.0
3.0
2.0
1.0
ppm (t1)

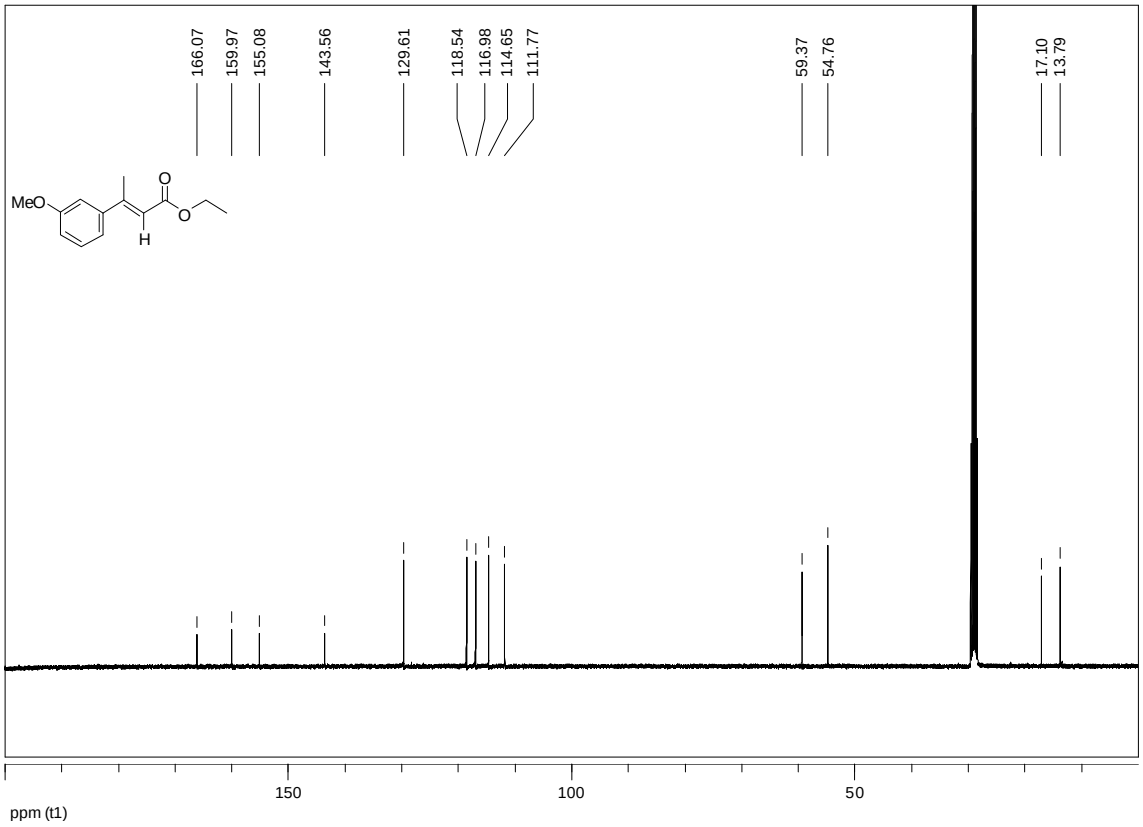

O
O
H
MeO
7.0
6.0
5.0
4.0
3.0
2.0
1.0
ppm (t1)

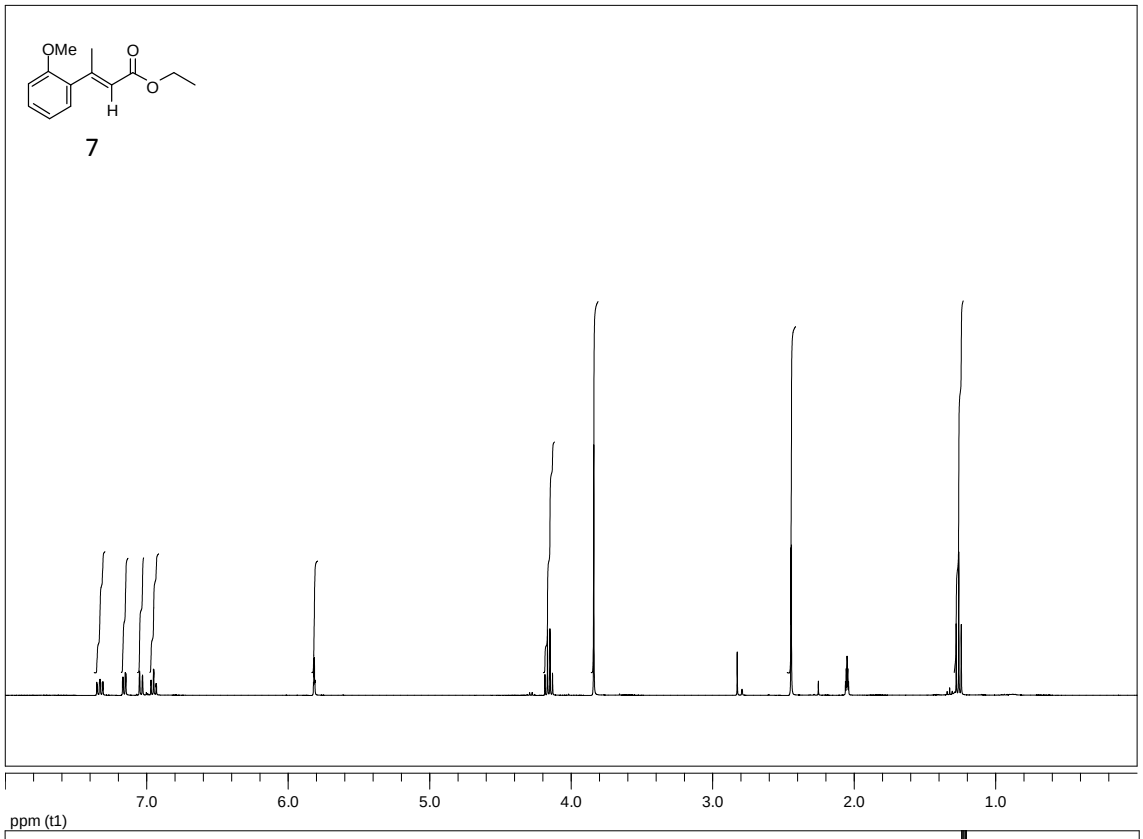
5

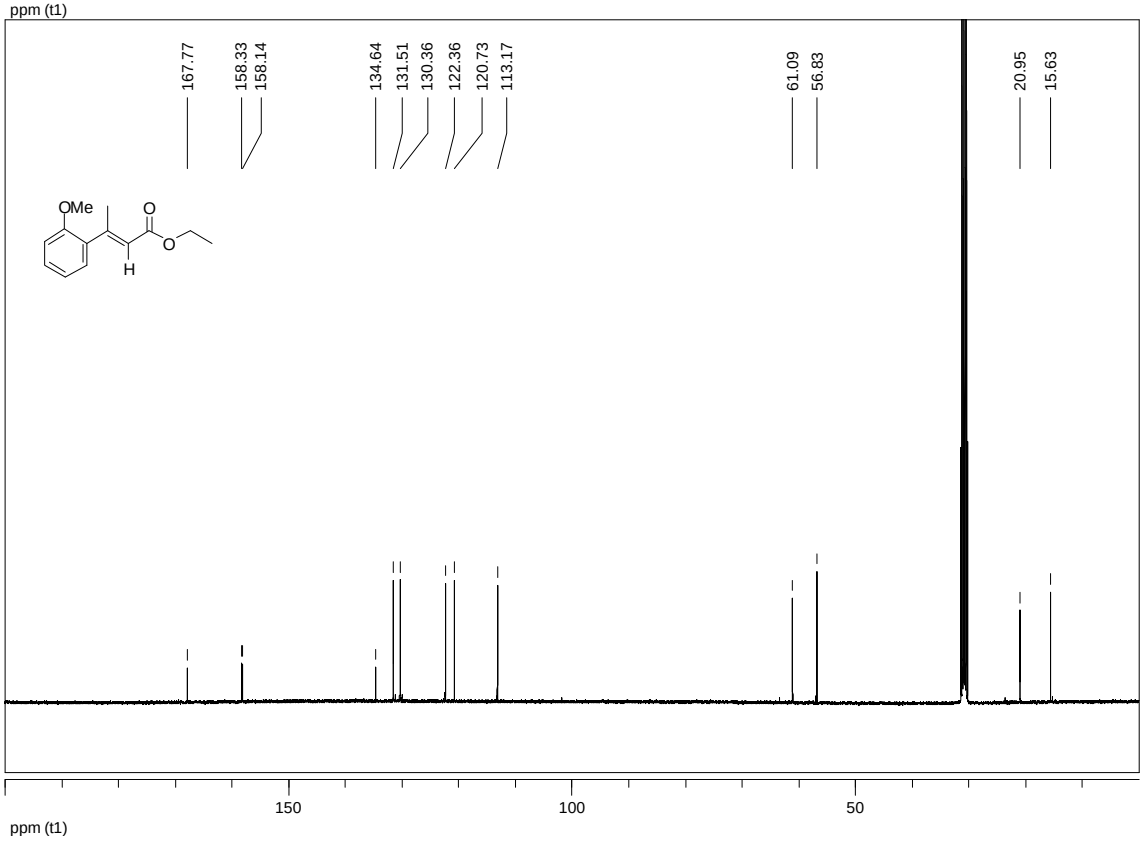

168.12
162.64
156.39
135.73
129.51
116.69
115.75
61.03
56.67
18.52
15.67
O
O
H
MeO
150
100
50
ppm (f1)

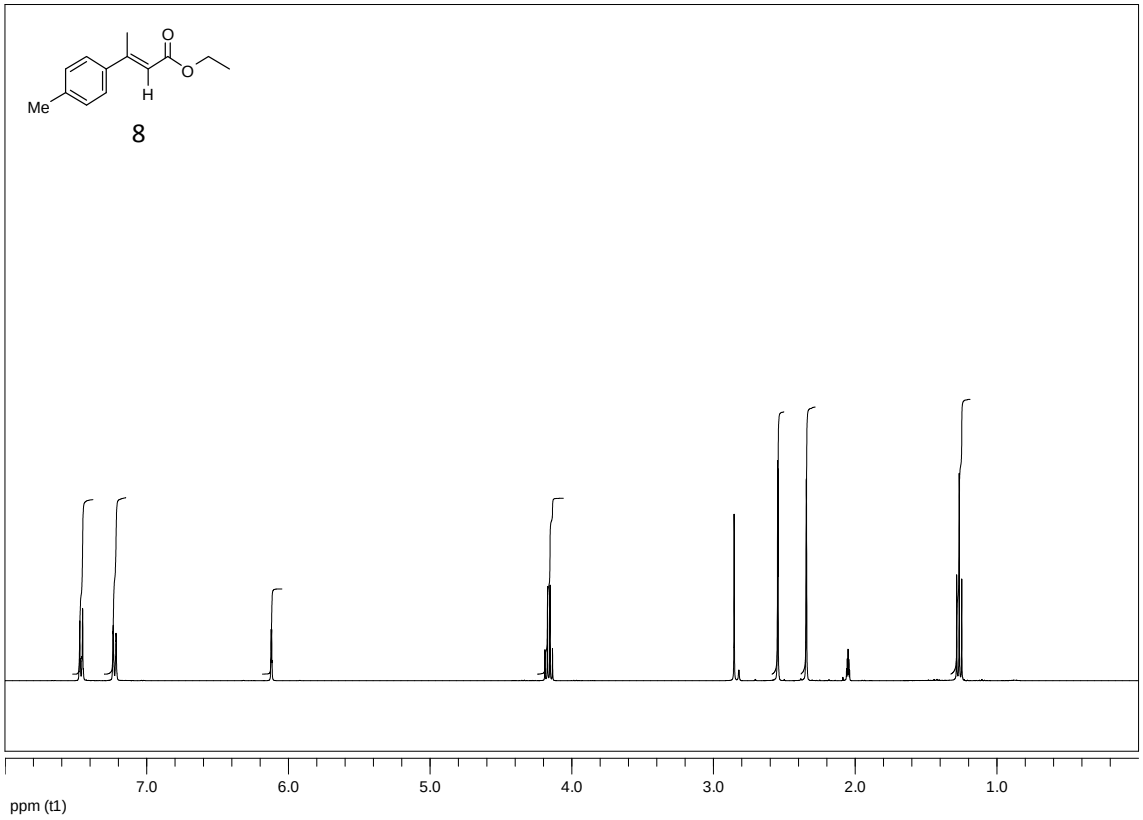

MeO
O
O
H
7.0
6.0
5.0
4.0
3.0
2.0
1.0
ppm (t1)

6

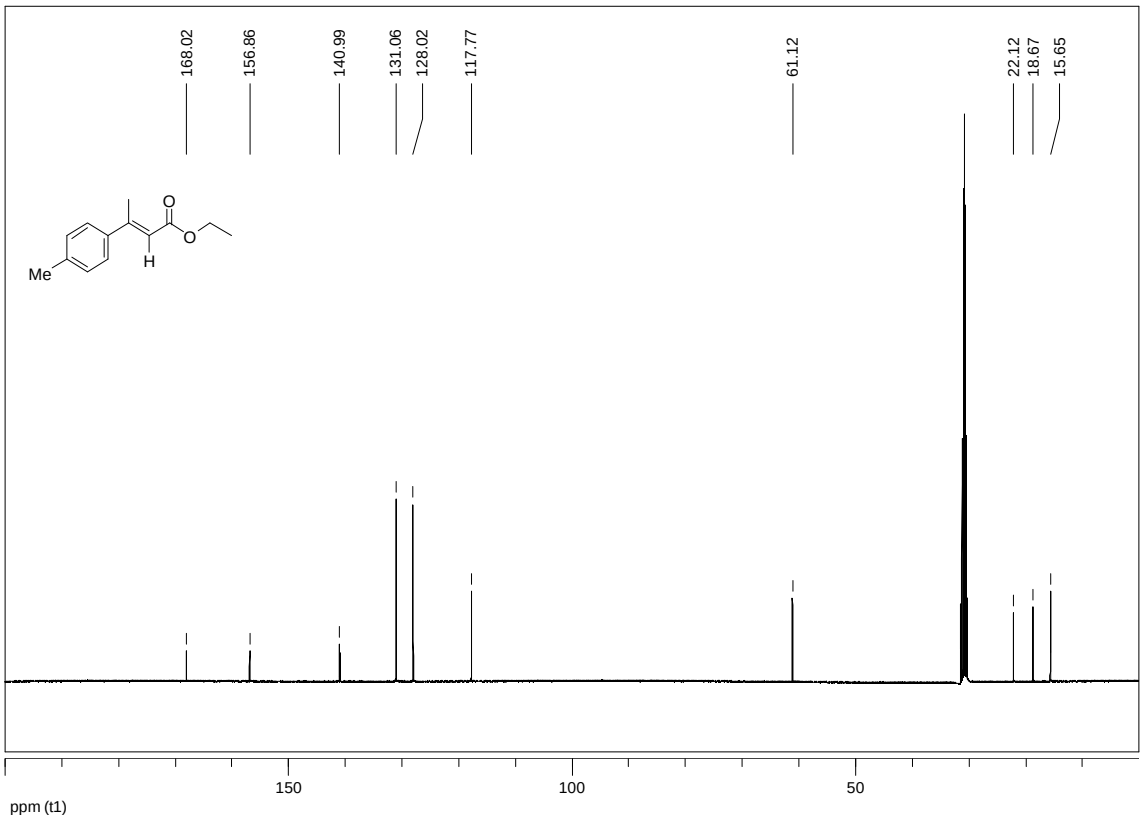

166.07
159.97
155.08
143.56
129.61
118.54
116.98
114.65
111.77
59.37
54.76
17.10
13.79
MeO
O
O
H
150
100
50
ppm (t1)

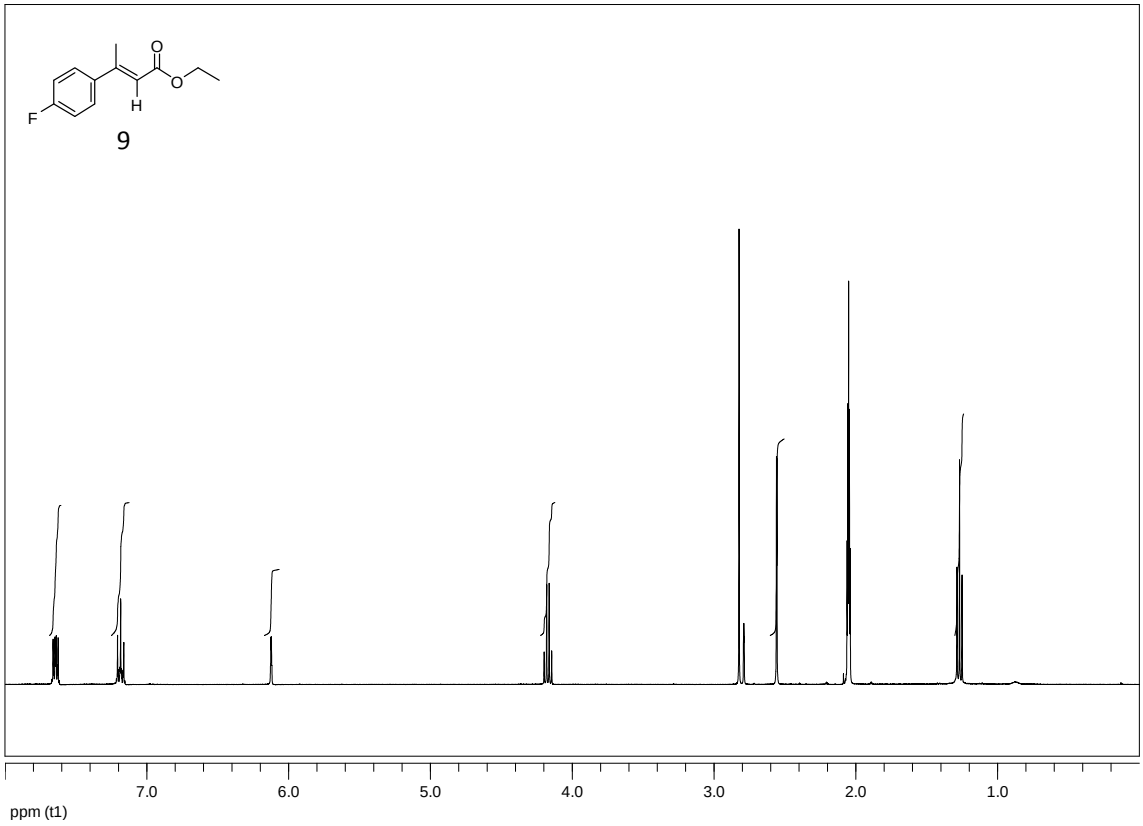

OMe
O
O
H
7
7.0
6.0
5.0
4.0
3.0
2.0
1.0
ppm (t1)

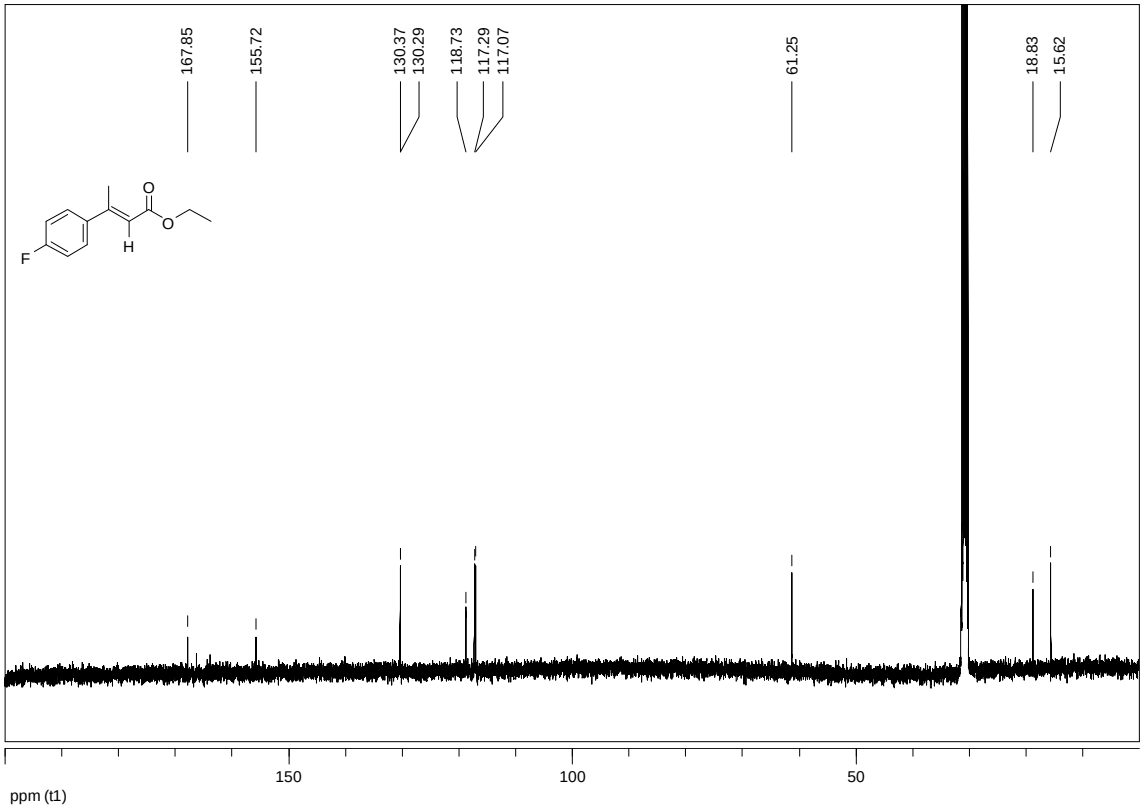

167.77
158.33
158.14
134.64
131.51
130.36
122.36
120.73
113.17
61.09
56.83
20.95
15.63
OMe
O
O
H
150
100
50
ppm (t1)

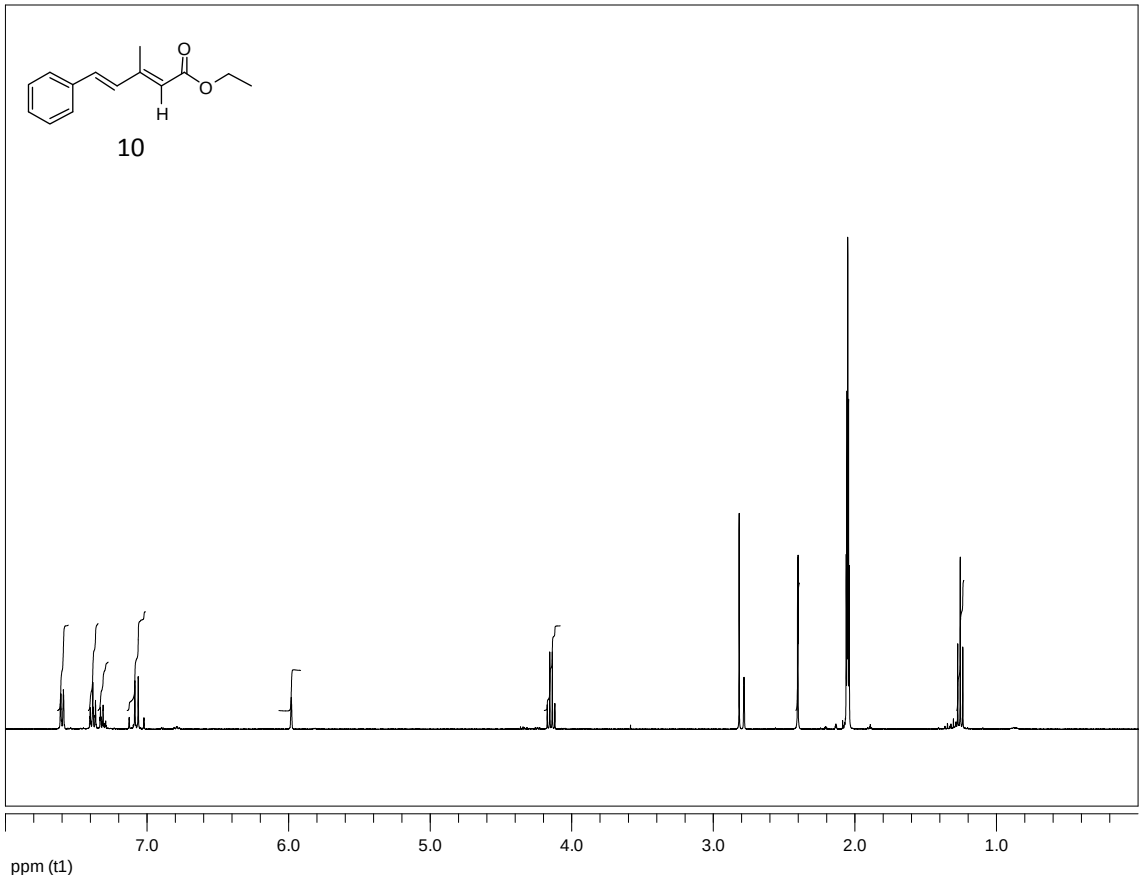

O
O
H
Me
7.0
6.0
5.0
4.0
3.0
2.0
1.0
ppm (t1)

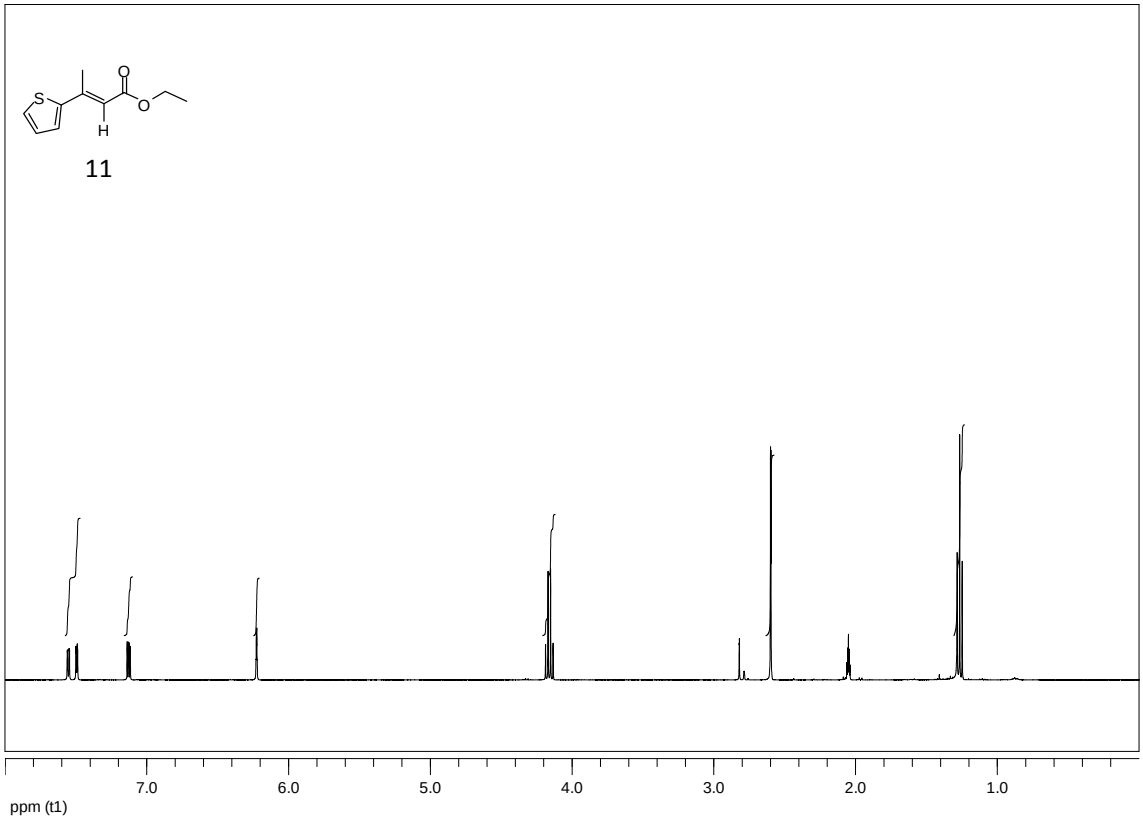
8

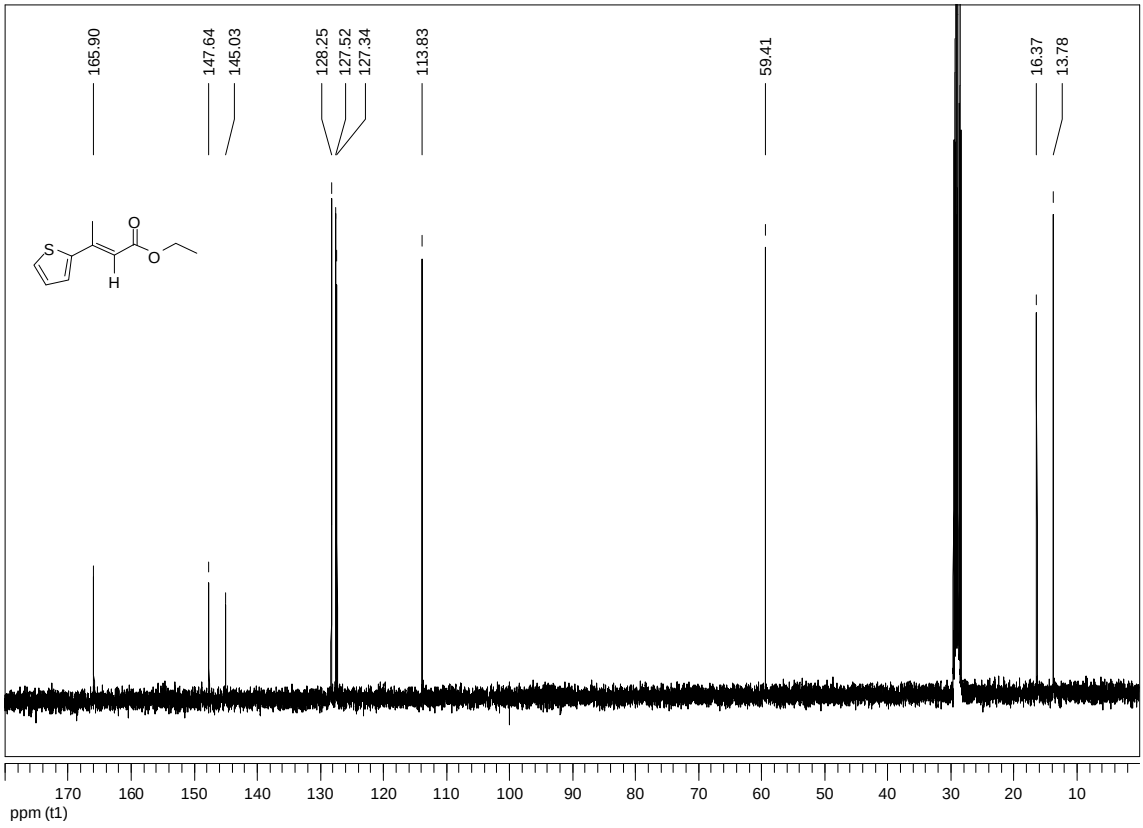

168.02
156.86
140.99
131.06
128.02
117.77
61.12
22.12
18.67
15.65
O
O
H
Me
150
100
50
ppm (t1)

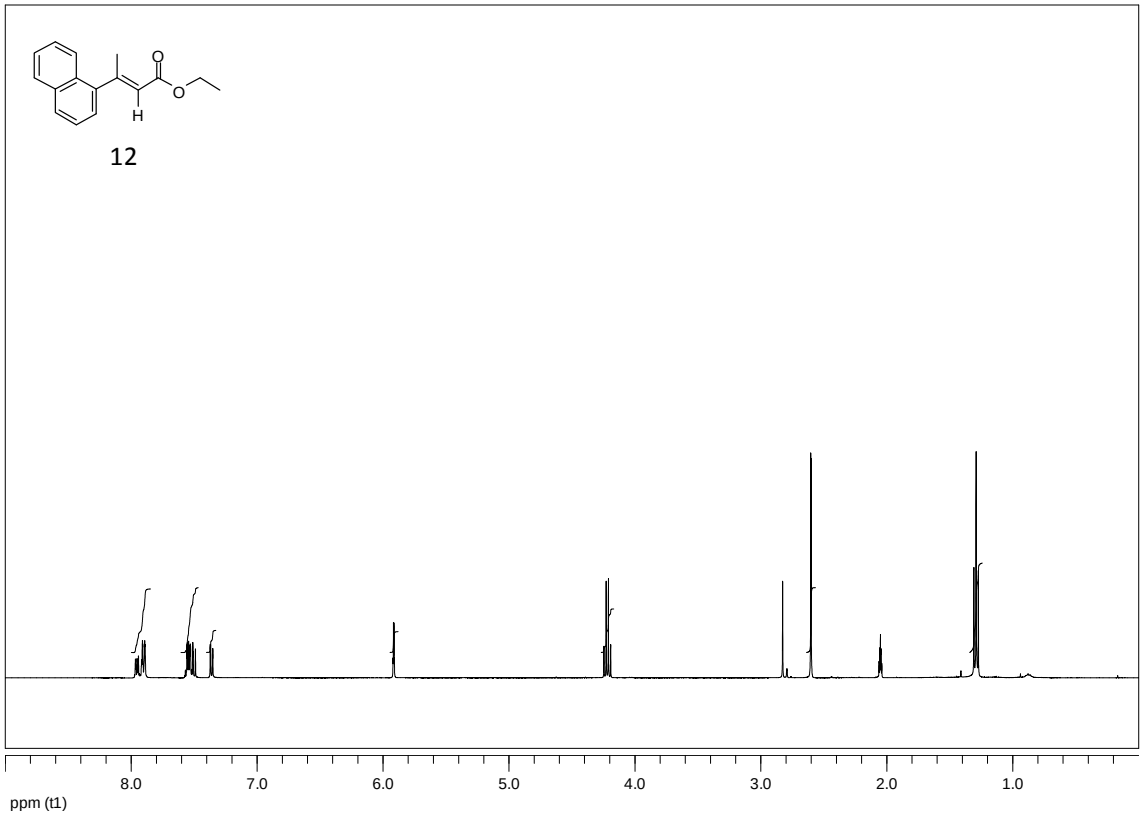

O
O
H
F
9
7.0
6.0
5.0
4.0
3.0
2.0
1.0
ppm (t1)


167.85
155.72
130.37
130.29
118.73
117.29
117.07
61.25
18.83
15.62
O
O
H
F
150
100
50
ppm (t1)

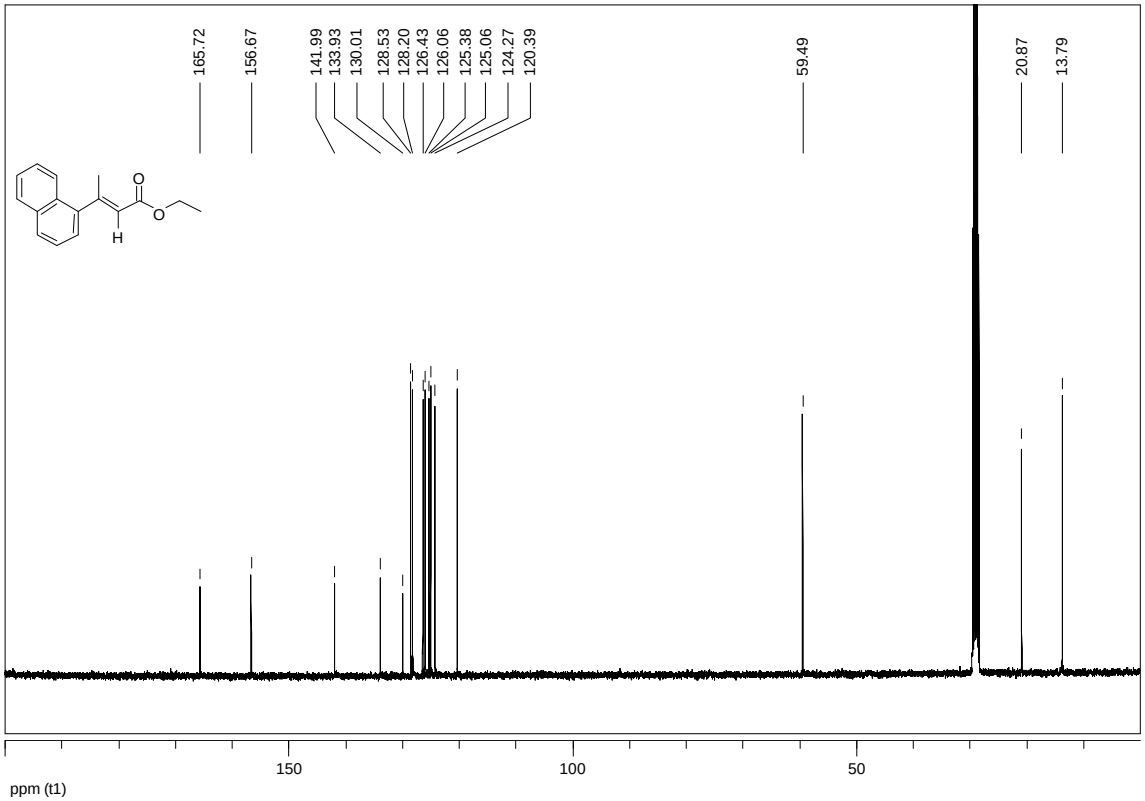

O
O
H
10
7.0
6.0
5.0
4.0
3.0
2.0
1.0
ppm (t1)

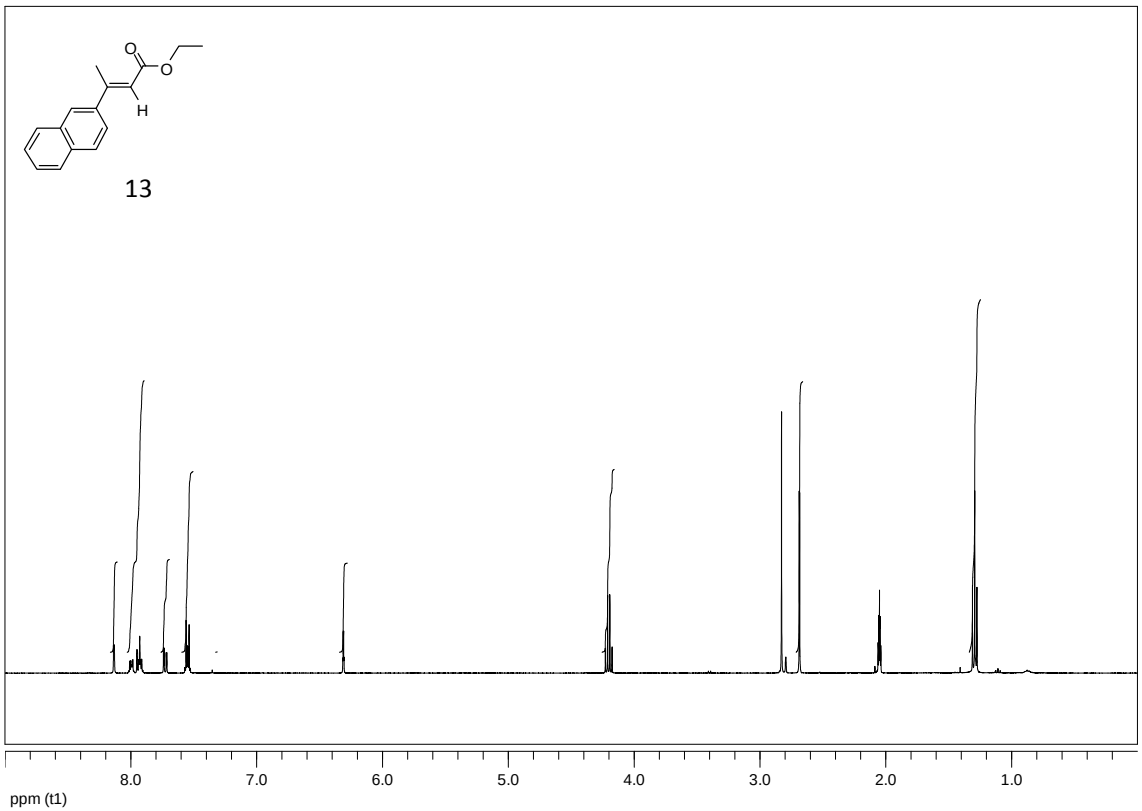

S
O
O
H
11
7.0
6.0
5.0
4.0
3.0
2.0
1.0
ppm (t1)

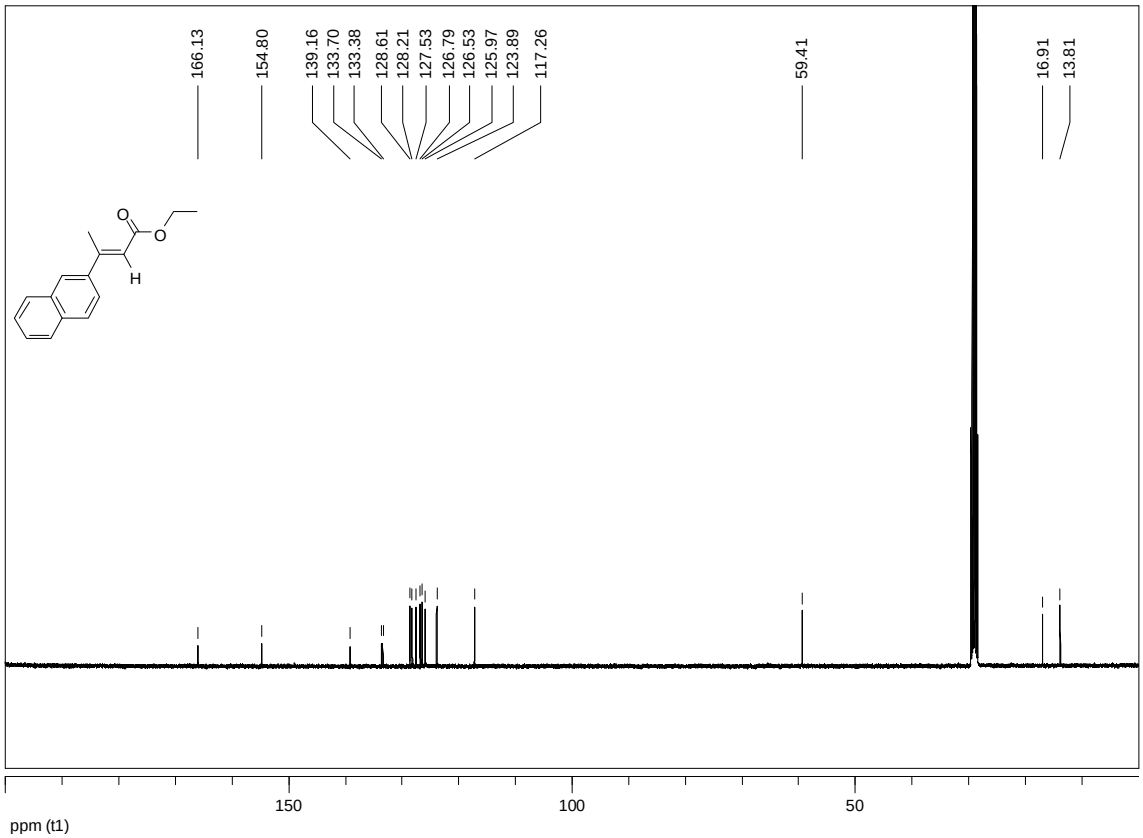

165.90
147.64
145.03
128.25
127.52
127.34
113.83
59.41
16.37
13.78
S
O
O
H
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
ppm (t1)

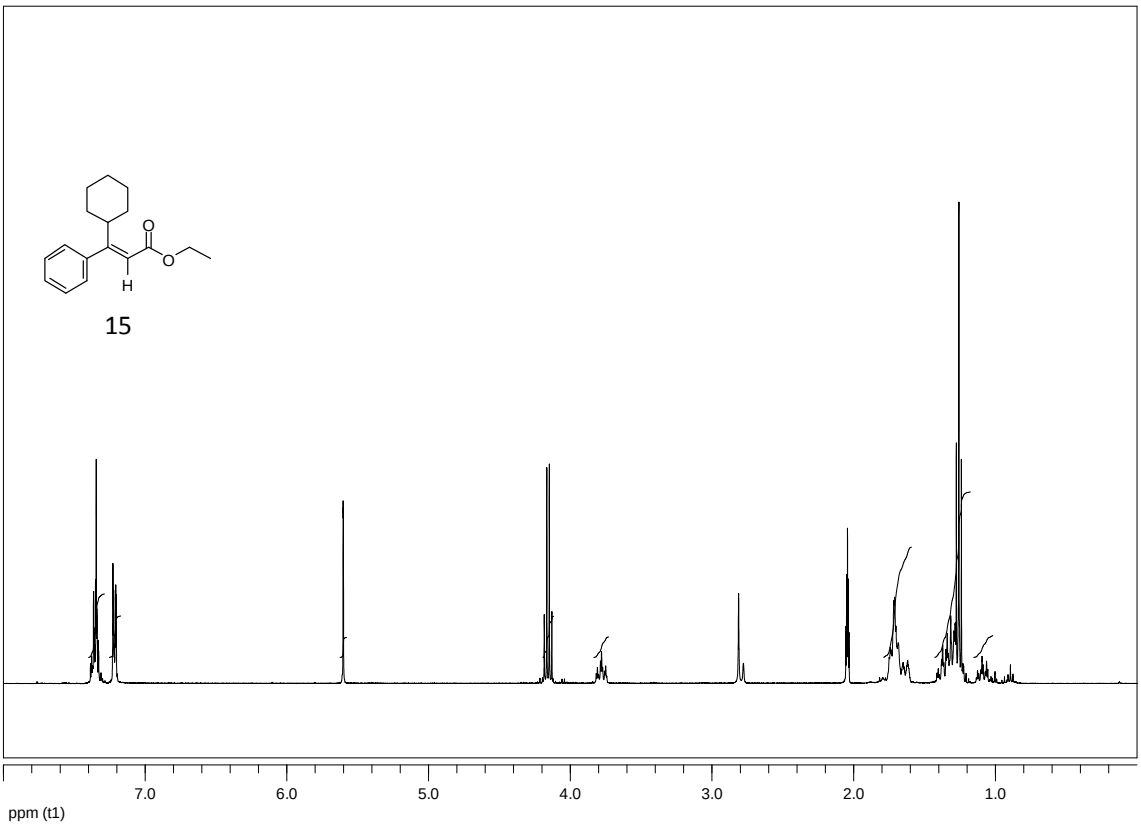

O
O
H
8.0
7.0
6.0
5.0
4.0
3.0
2.0
1.0
ppm (t1)

12

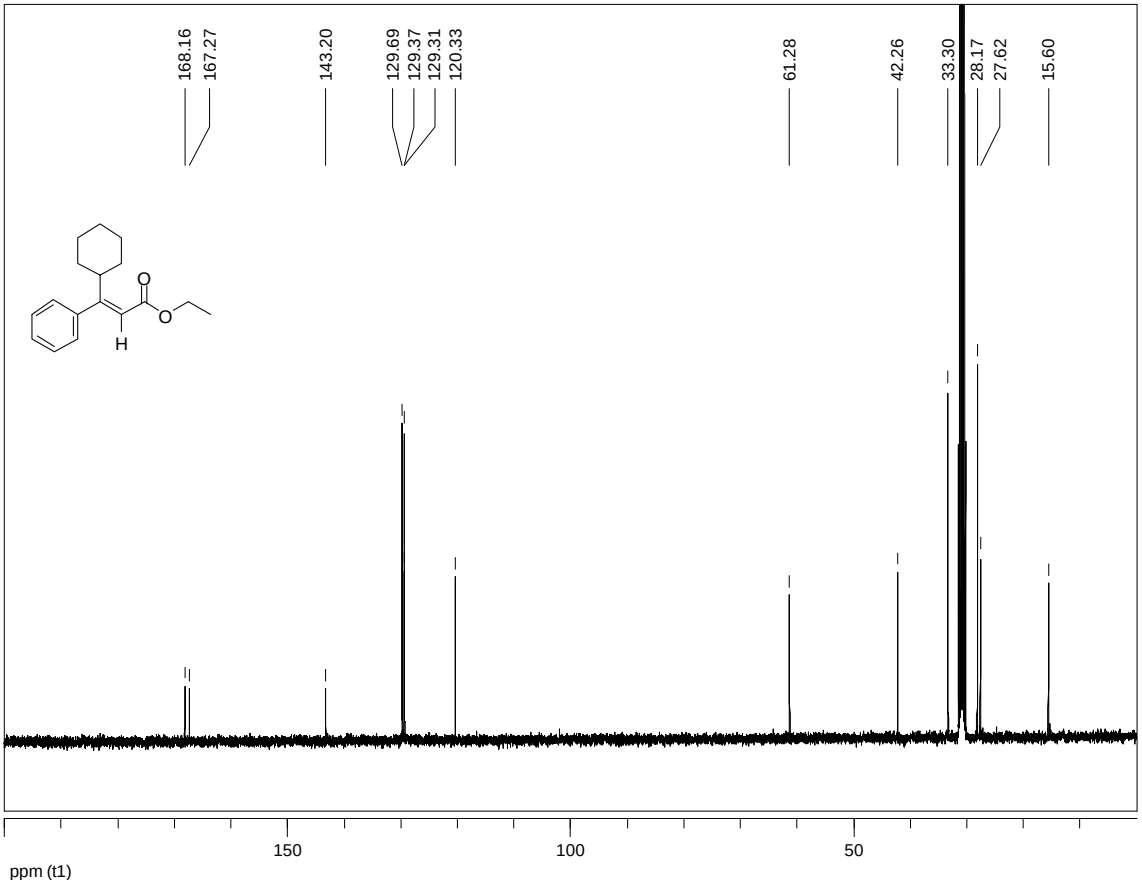

165.72
156.67
141.99
133.93
130.01
128.53
128.20
126.43
126.06
125.38
125.06
124.27
120.39
59.49
20.87
13.79
O
O
H
150
100
50
ppm (t1)

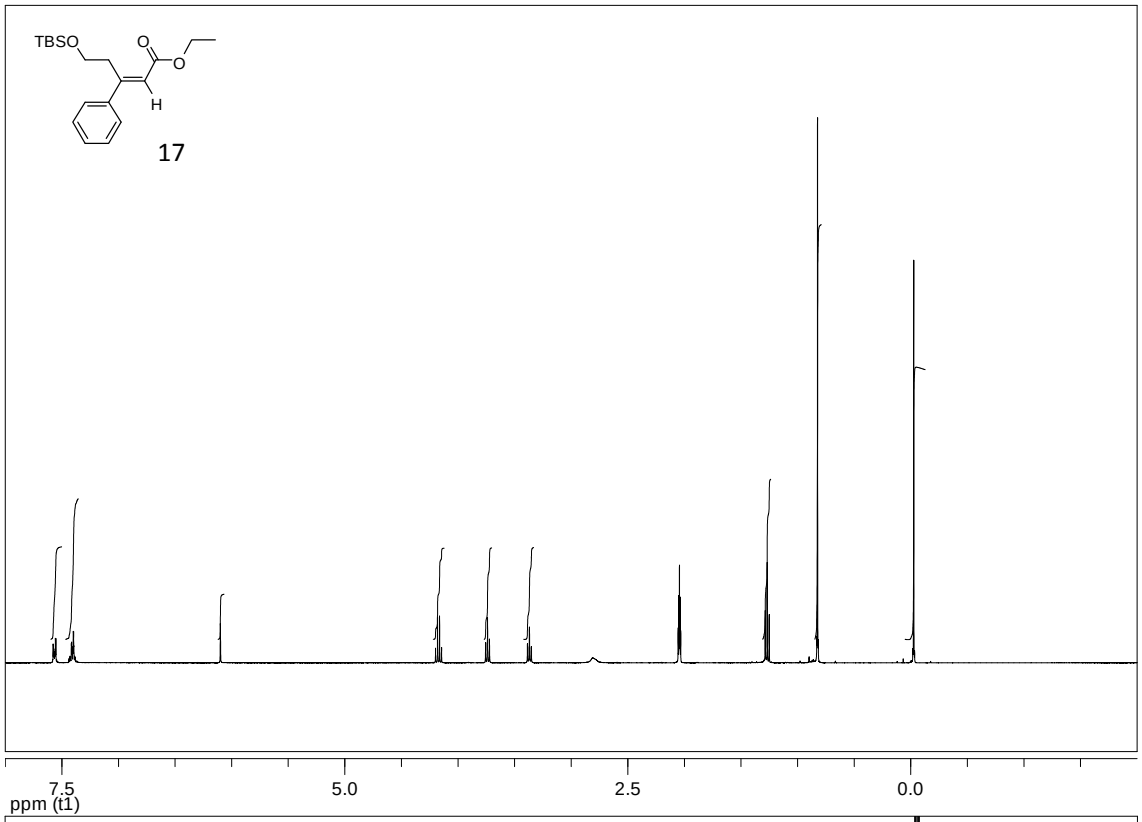

O
O
H
13
8.0
7.0
6.0
5.0
4.0
3.0
2.0
1.0
ppm (t1)

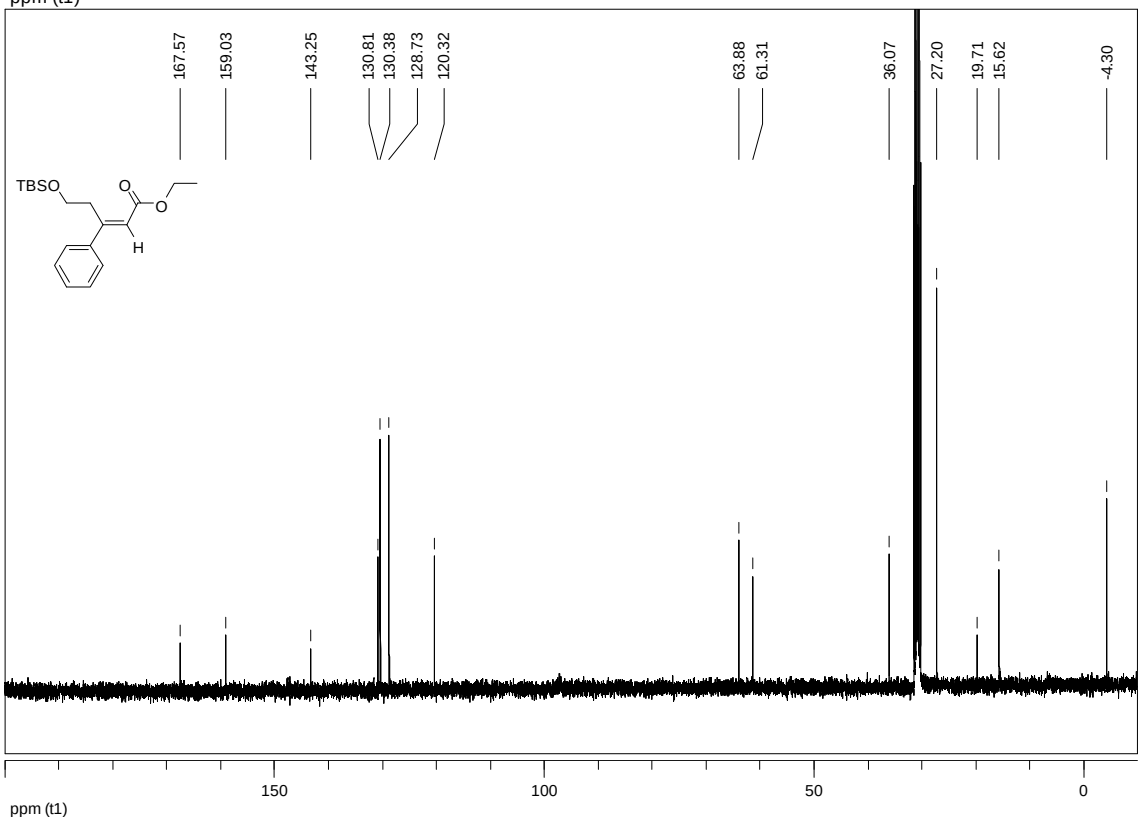

166.13
154.80
139.16
133.70
133.38
128.61
128.21
127.53
126.79
126.53
125.97
123.89
117.26
59.41
16.91
13.81
O
O
H
150
100
50
ppm (t1)

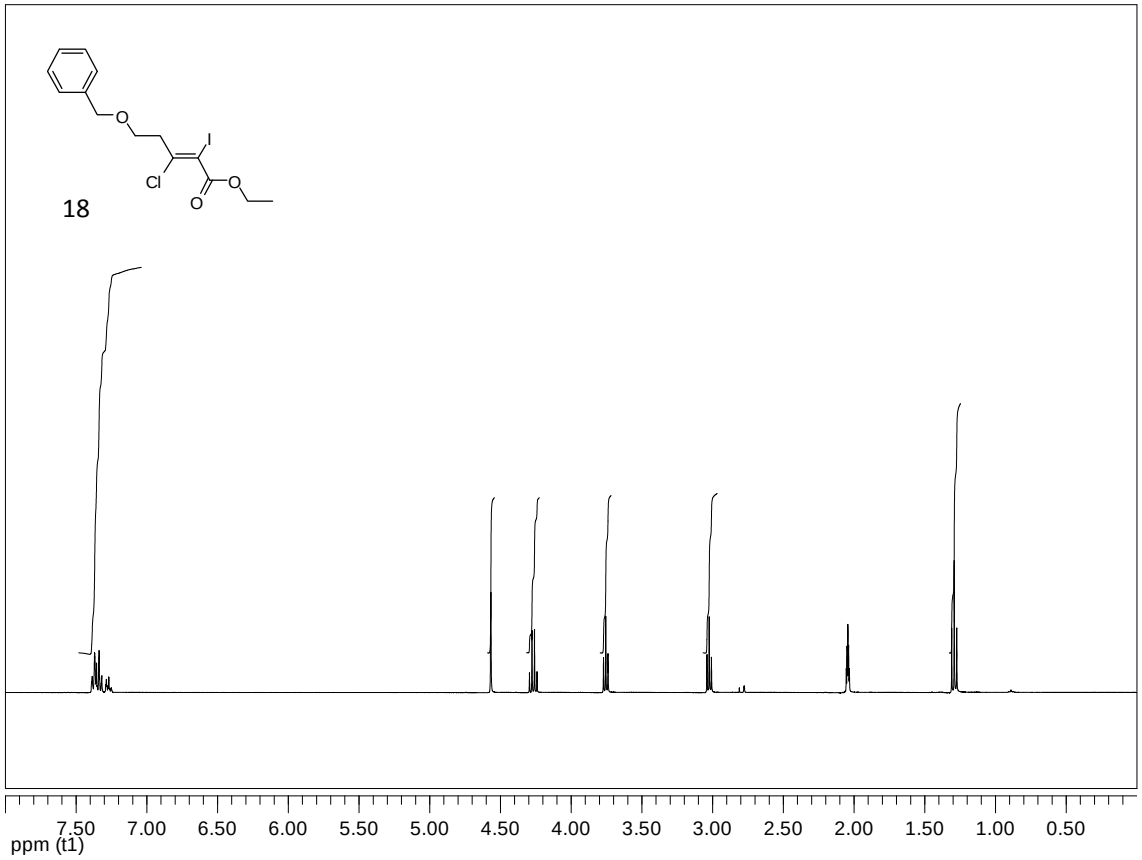

O
O
H
7.0
6.0
5.0
4.0
3.0
2.0
1.0
ppm (t1)

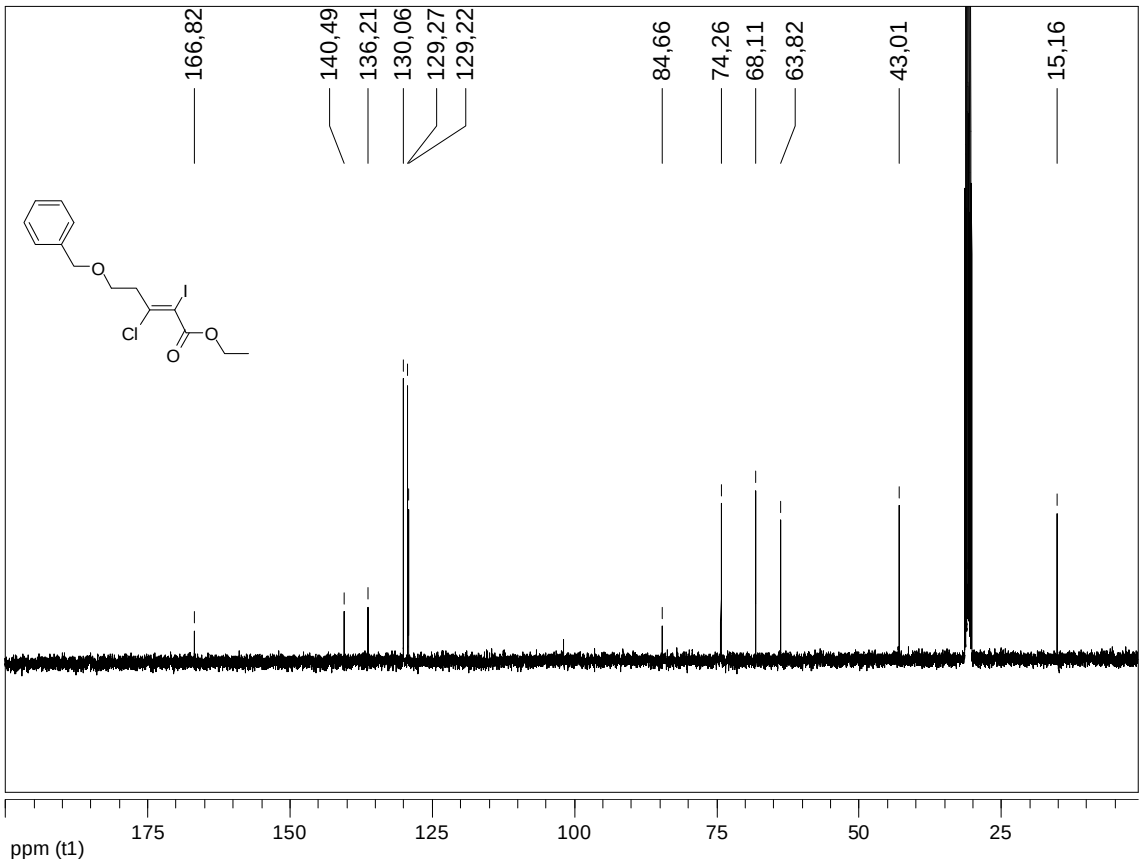
15

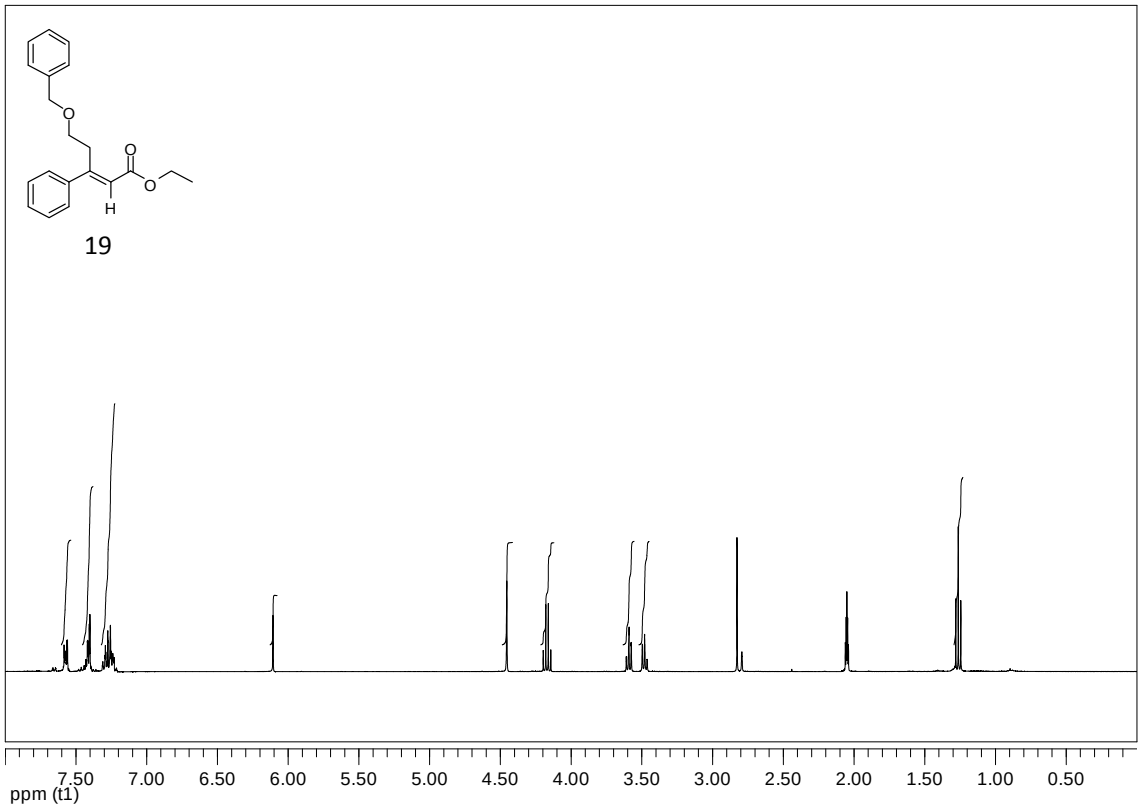

168.16
167.27
143.20
129.69
129.37
129.31
120.33
61.28
42.26
33.30
28.17
27.62
15.60
O
O
H
150
100
50
ppm (t1)

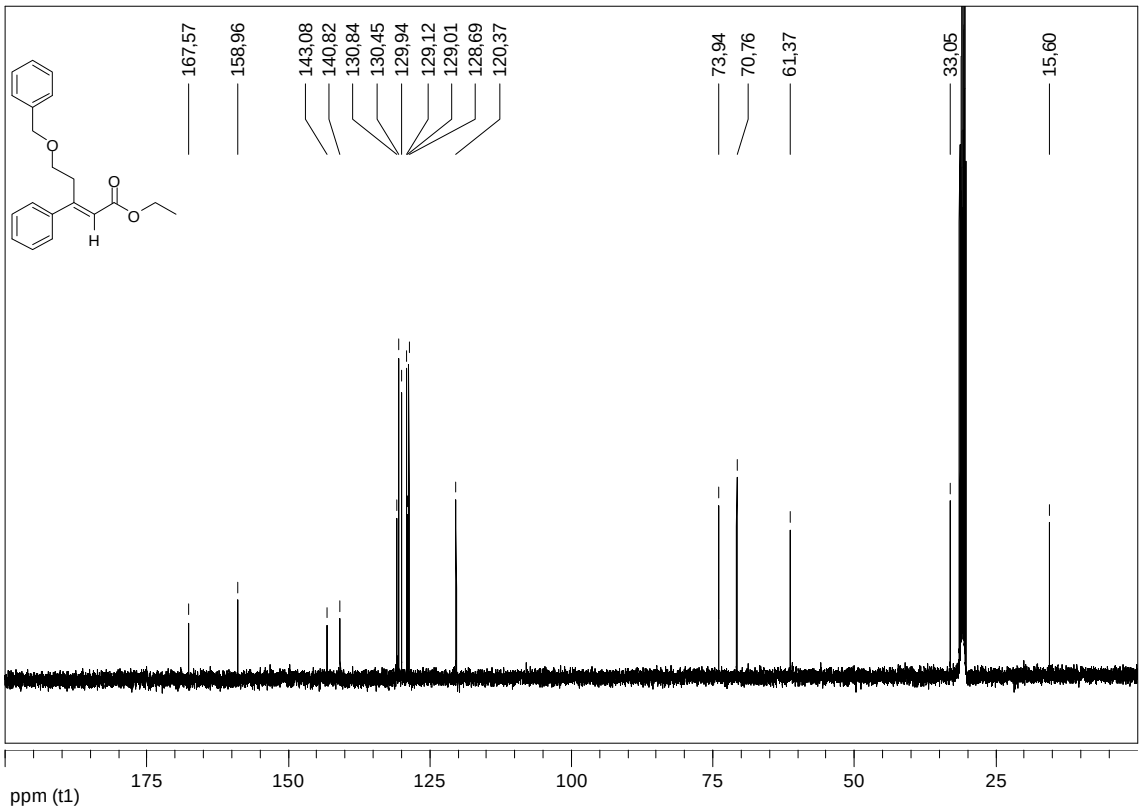

TBSO
O
O
H
17
7.5
5.0
2.5
0.0
ppm (t1)

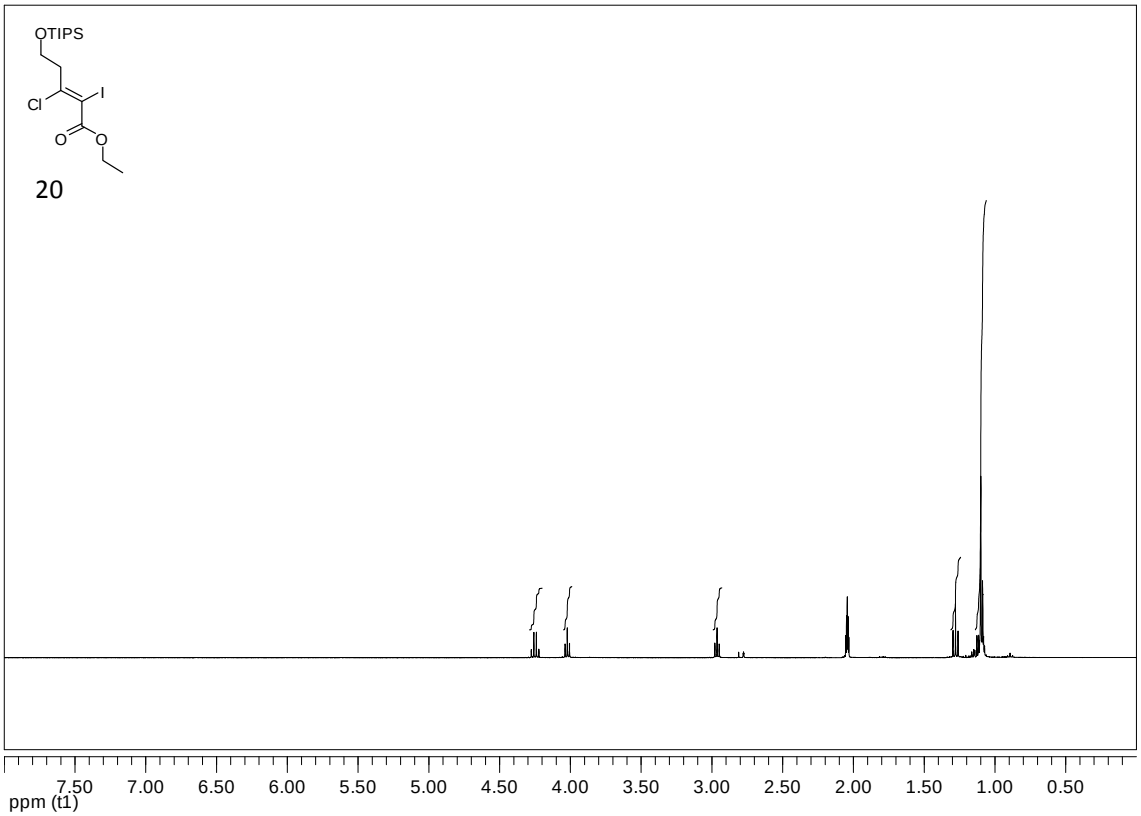

167.57
159.03
143.25
130.81
130.38
128.73
120.32
63.88
61.31
36.07
27.20
19.71
15.62
-4.30
TBSO
O
O
H
150
100
50
0
ppm (t1)

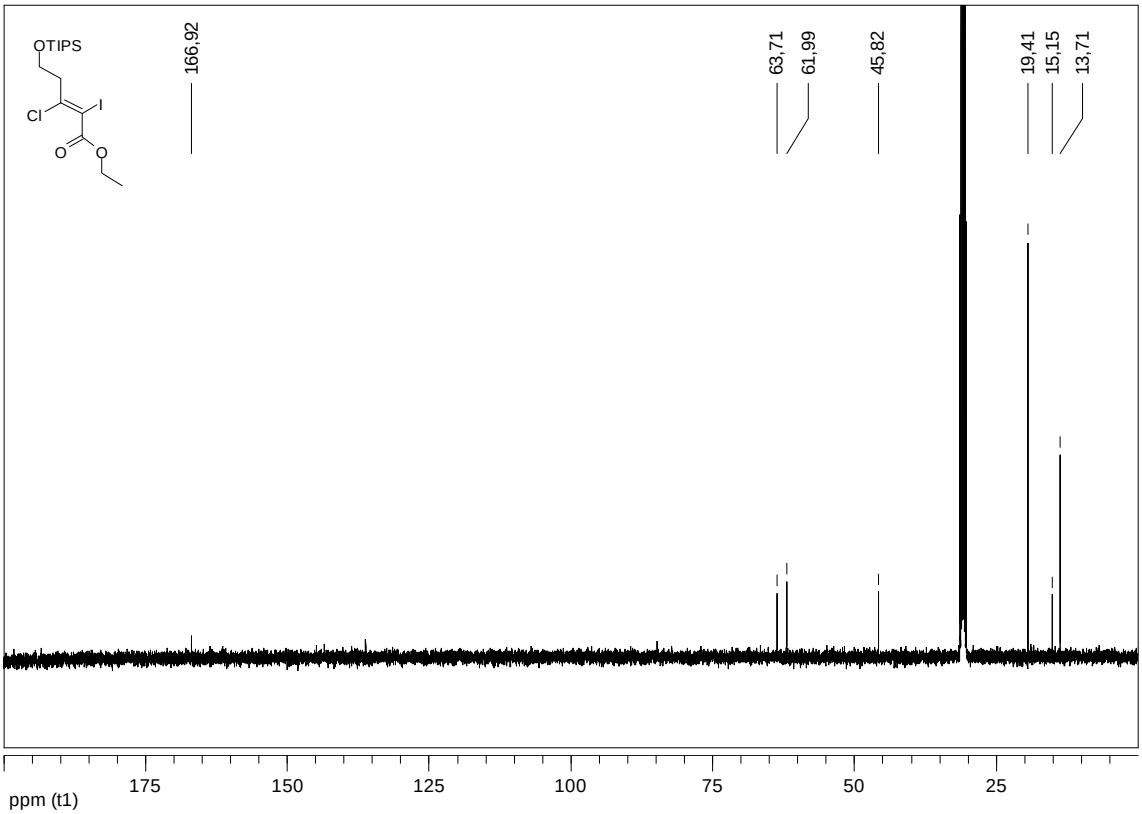

18
O
I
Cl
O
O
7.50
7.00
6.50
6.00
5.50
5.00
4.50
4.00
3.50
3.00
2.50
2.00
1.50
1.00
0.50
ppm (t1)

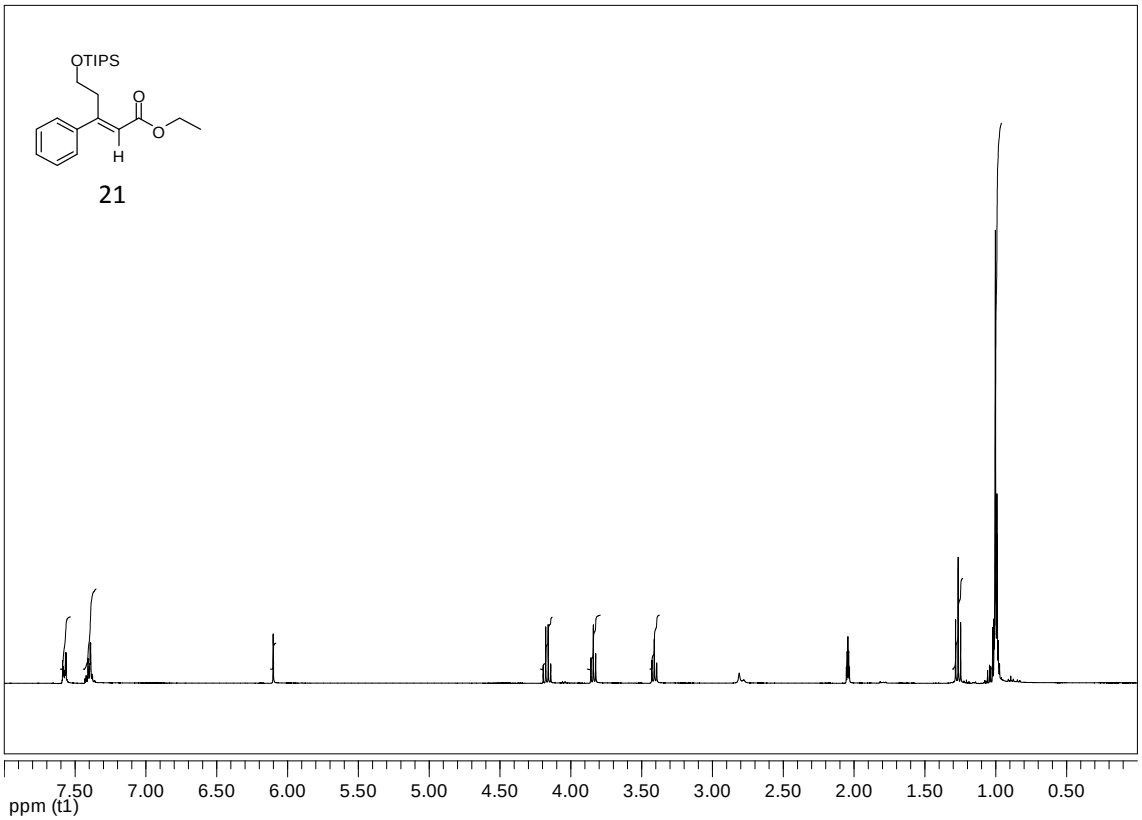

166,82
140,49
136,21
130,06
129,27
129,22
84,66
74,26
68,11
63,82
43,01
15,16
O
I
Cl
O
O
175
150
125
100
75
50
25
ppm (t1)

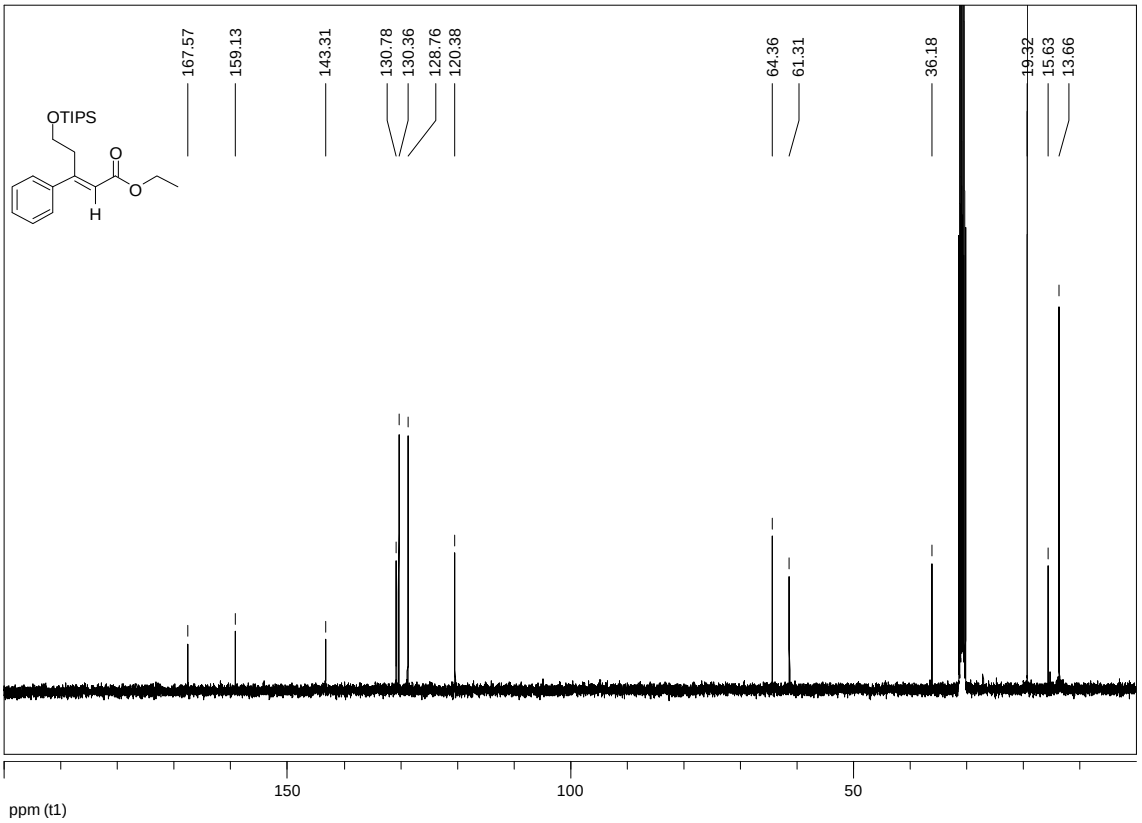

O
O
O
H
19
7.50
7.00
6.50
6.00
5.50
5.00
4.50
4.00
3.50
3.00
2.50
2.00
1.50
1.00
0.50
ppm (t1)

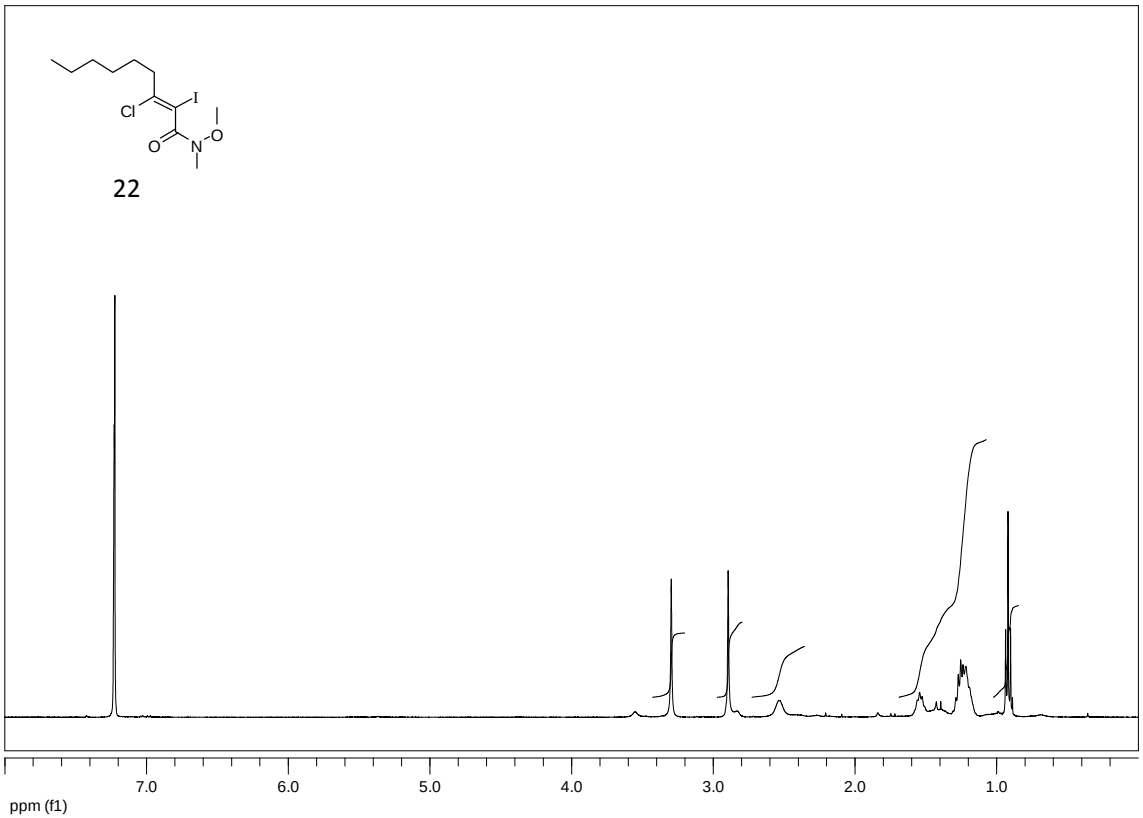

167,57
158,96
143,08
140,82
130,84
130,45
129,94
129,12
129,01
128,69
120,37
73,94
70,76
61,37
33,05
15,60
O
O
O
H
175
150
125
100
75
50
25
ppm (t1)


OTIPS
Cl
I
O
O
20
7.50
7.00
6.50
6.00
5.50
5.00
4.50
4.00
3.50
3.00
2.50
2.00
1.50
1.00
0.50
ppm (t1)

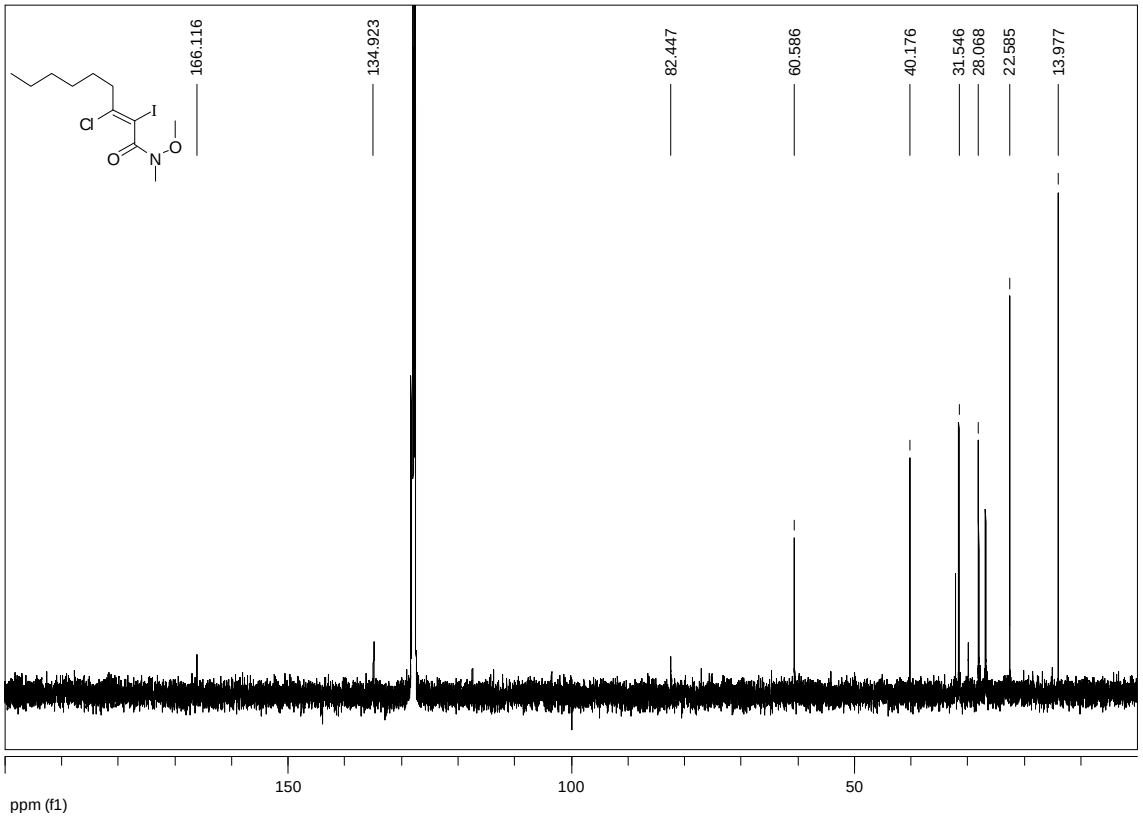

OTIPS
Cl
I
O
O
166,92
63,71
61,99
45,82
19,41
15,15
13,71
175
150
125
100
75
50
25
ppm (t1)

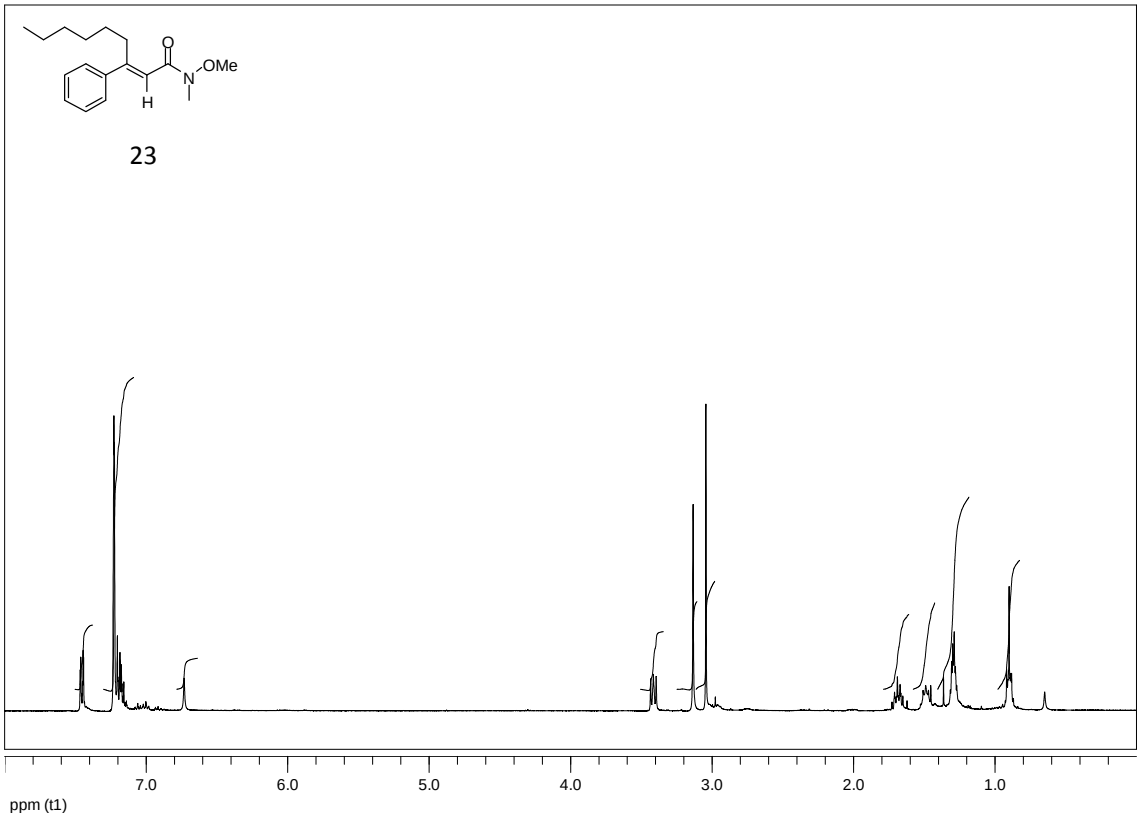

OTIPS
O
O
H
21
7.50
7.00
6.50
6.00
5.50
5.00
4.50
4.00
3.50
3.00
2.50
2.00
1.50
1.00
0.50
ppm (t1)


167.57
159.13
143.31
130.78
130.36
128.76
120.38
64.36
61.31
36.18
19.32
15.63
13.66
OTIPS
O
O
H
150
100
50
ppm (t1)

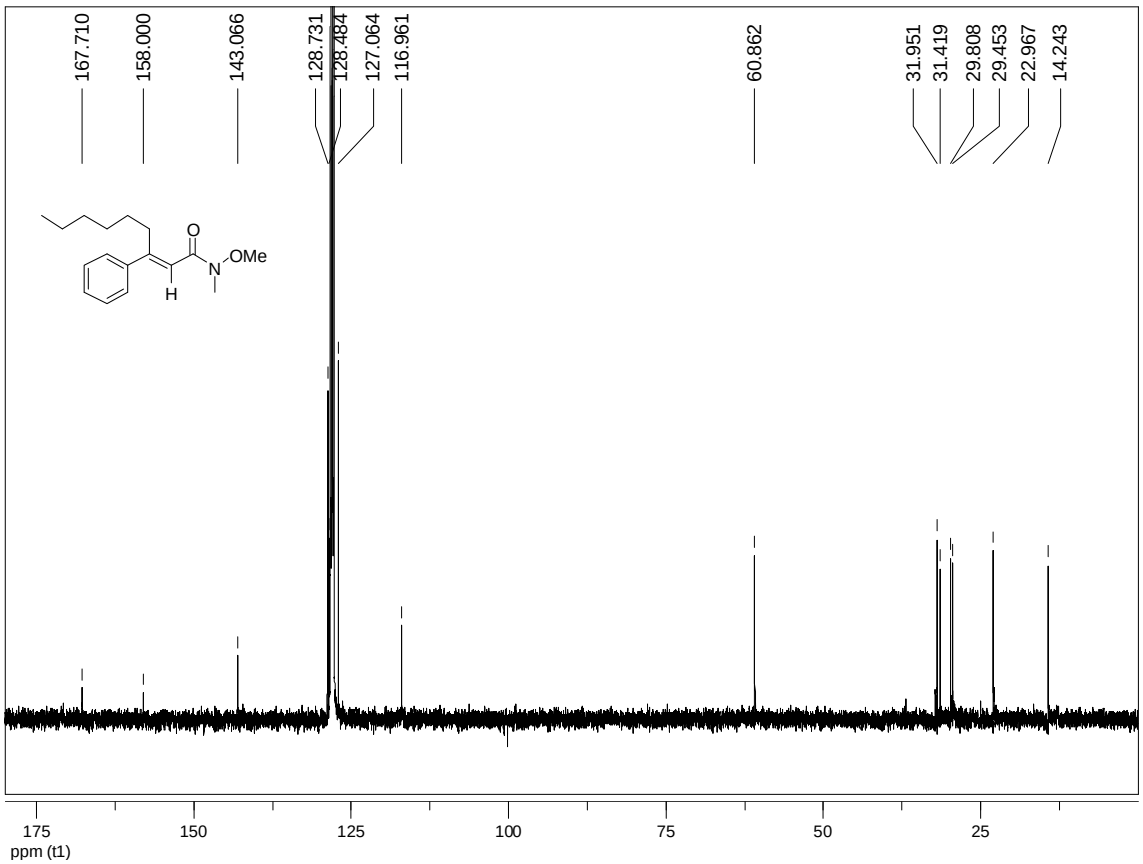

Cl
I
O
N
O
7.0
6.0
5.0
4.0
3.0
2.0
1.0
ppm (f1)

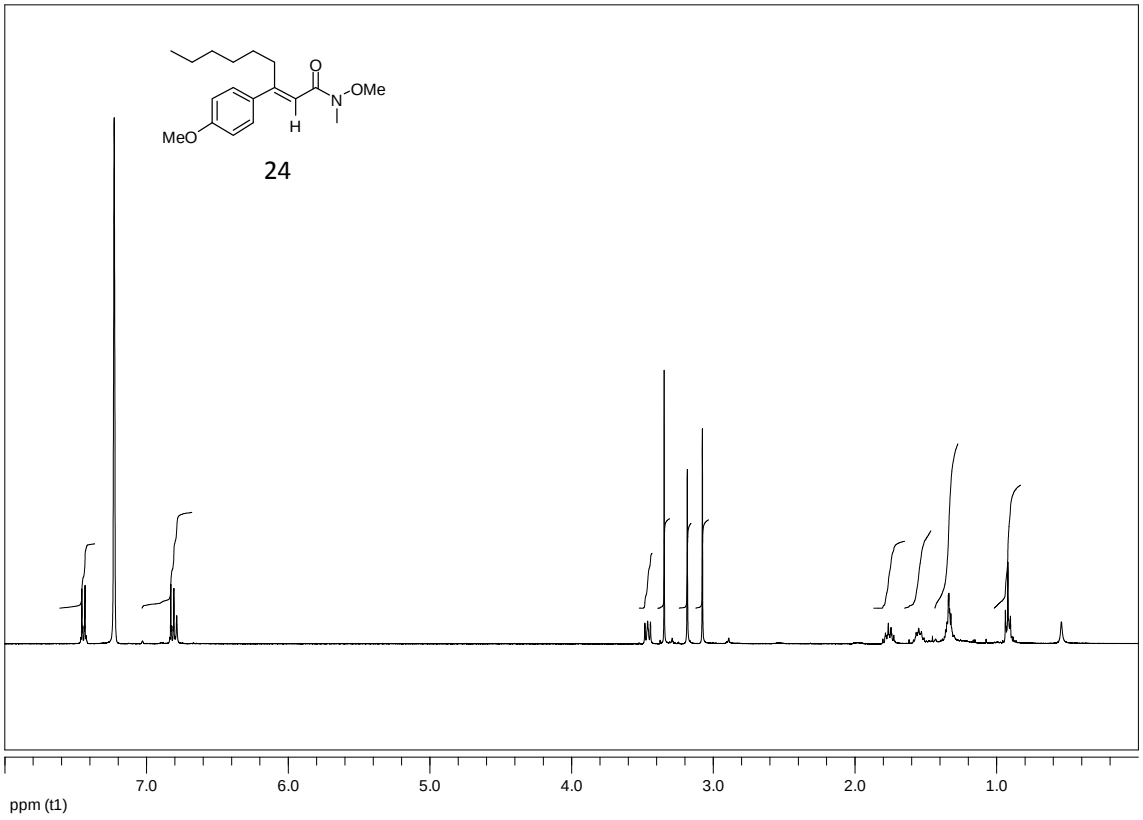
22


166.116
134.923
82.447
60.586
40.176
31.546
28.068
22.585
13.977
Cl
I
O
N
O
150
100
50
ppm (f1)

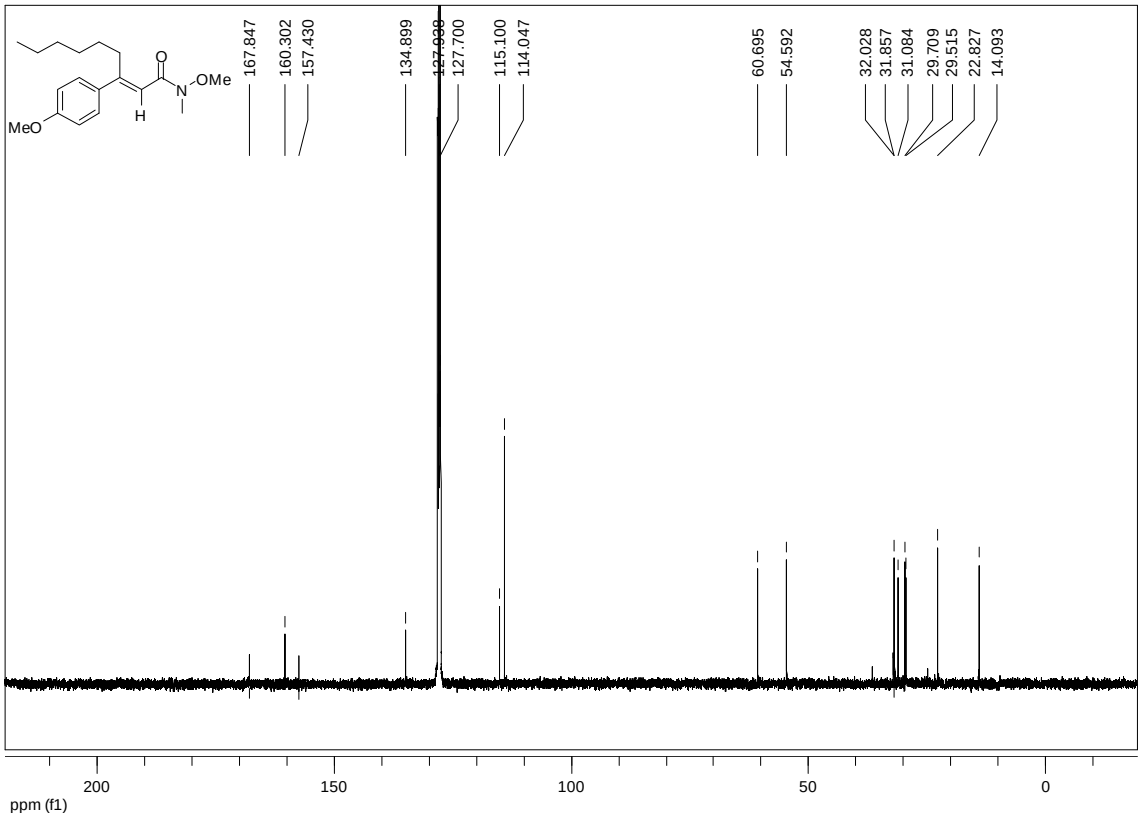

O
N
OMe
H
7.0
6.0
5.0
4.0
3.0
2.0
1.0
ppm (t1)

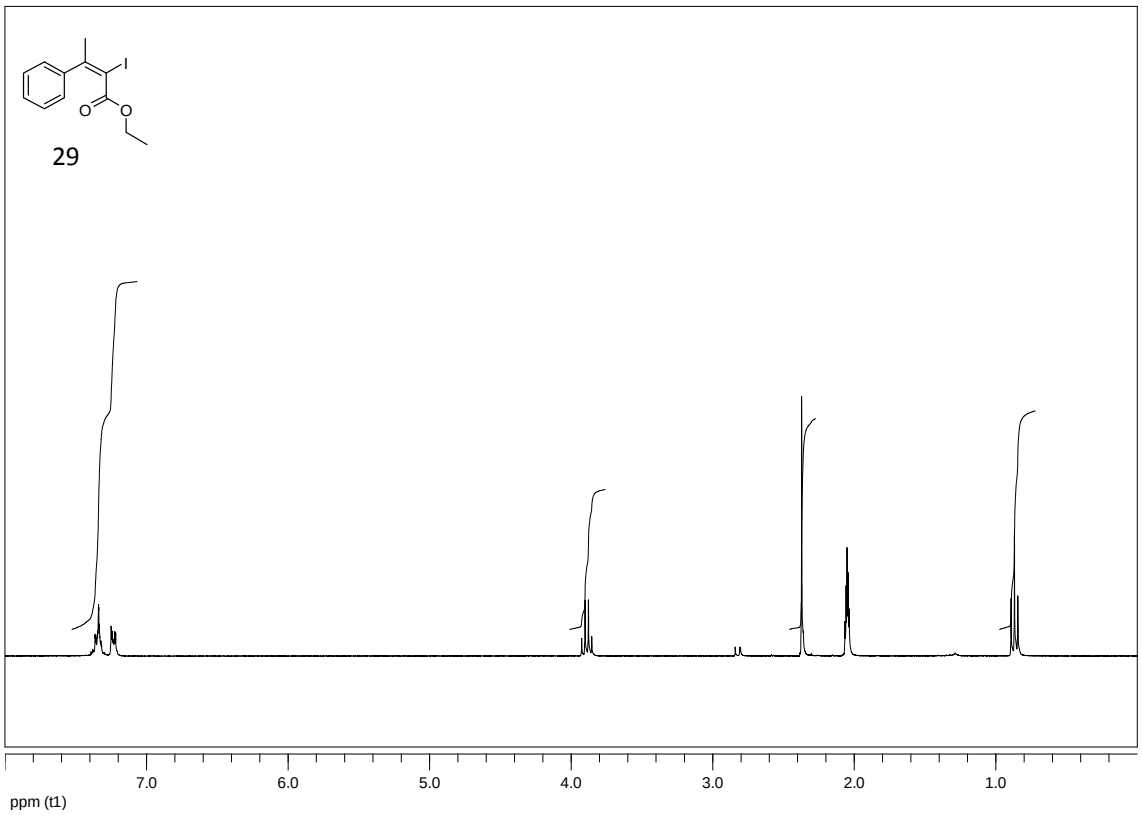
23

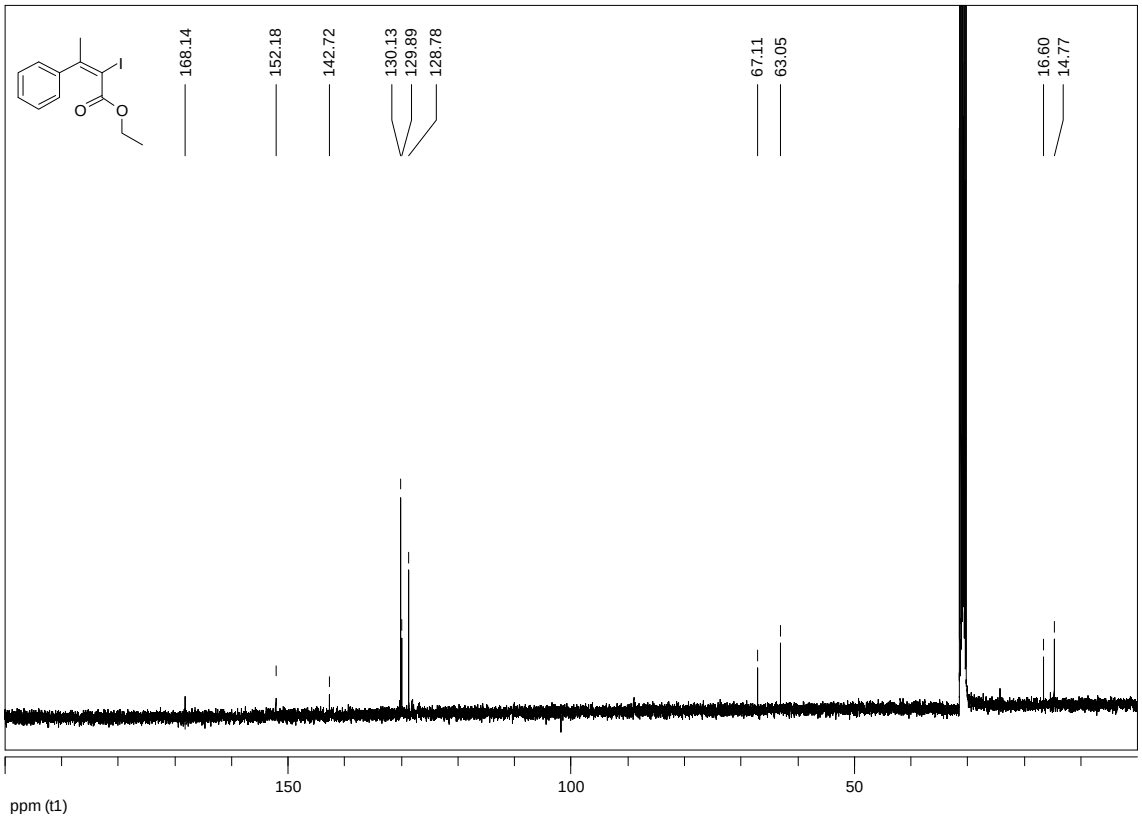

167.710
158.000
143.066
128.731
128.484
127.064
116.961
60.862
31.951
31.419
29.808
29.453
22.967
14.243
O
N
OMe
H
175
150
125
100
75
50
25
ppm (t1)

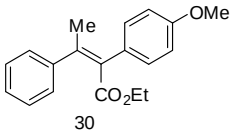

O
N
OMe
H
MeO
7.0
6.0
5.0
4.0
3.0
2.0
1.0
ppm (t1)

24

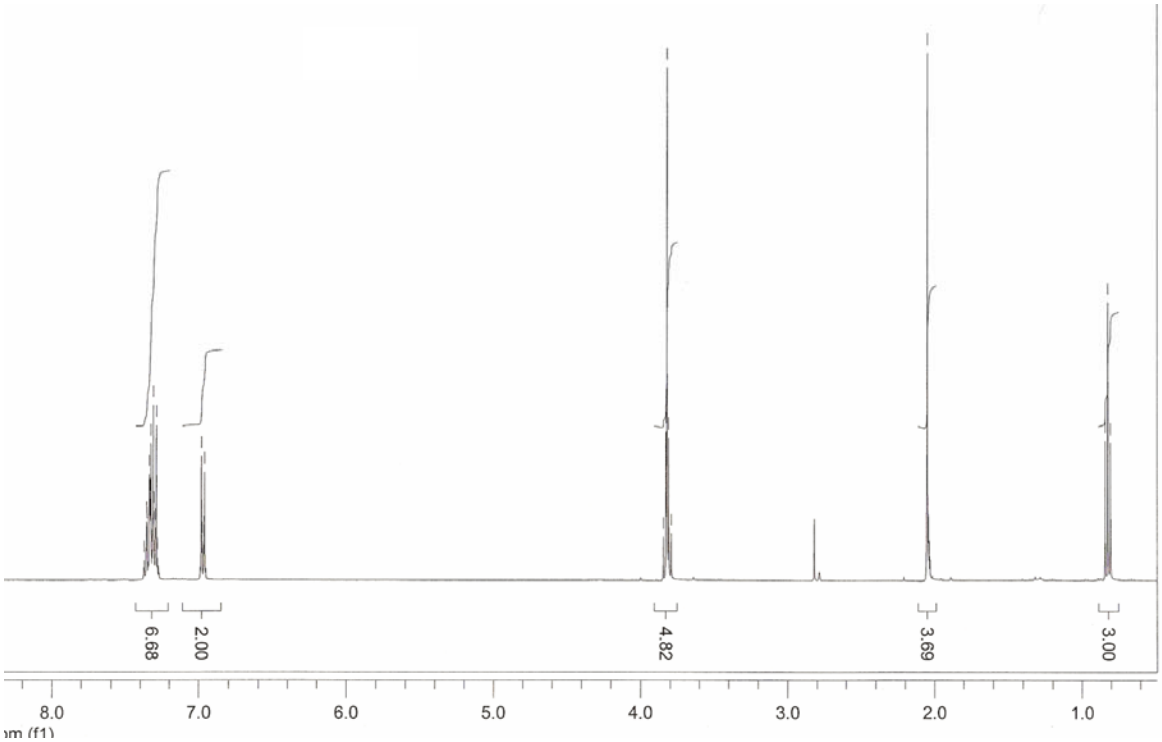

167.847
160.302
157.430
134.899
127.700
115.100
114.047
60.695
54.592
32.028
31.857
31.084
29.709
29.515
22.827
14.093
200
150
100
50
0
ppm (f1)

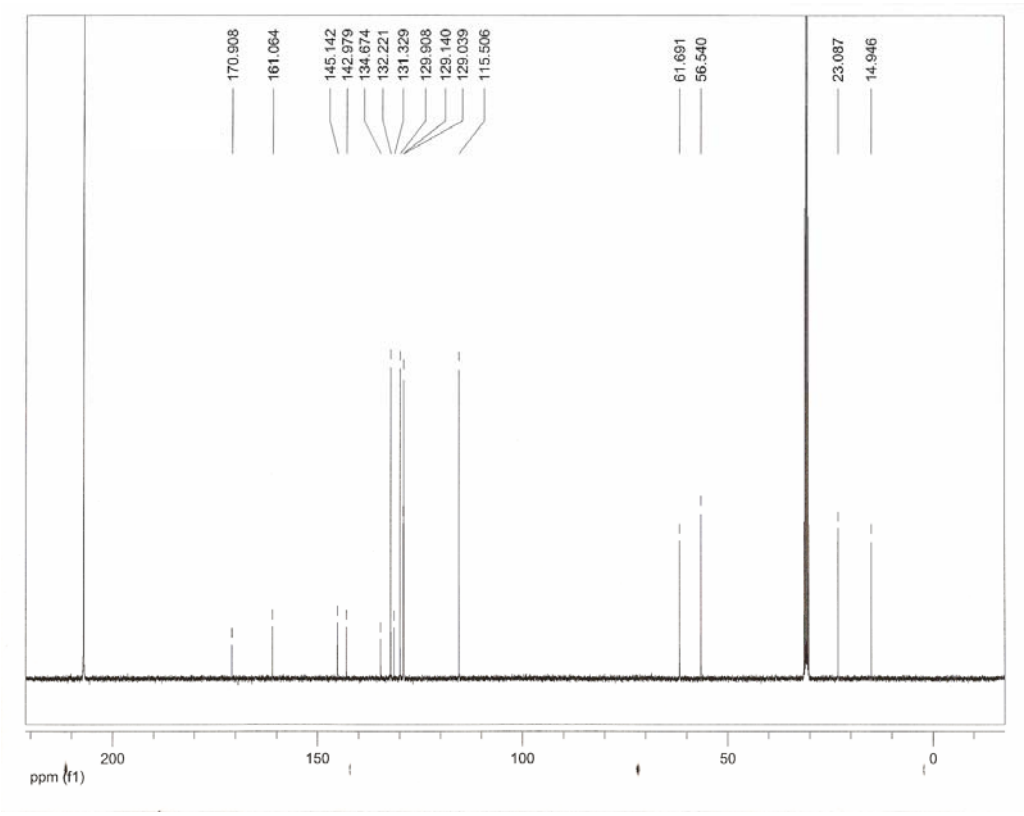

I
O
O
29
7.0
6.0
5.0
4.0
3.0
2.0
1.0
ppm (t1)

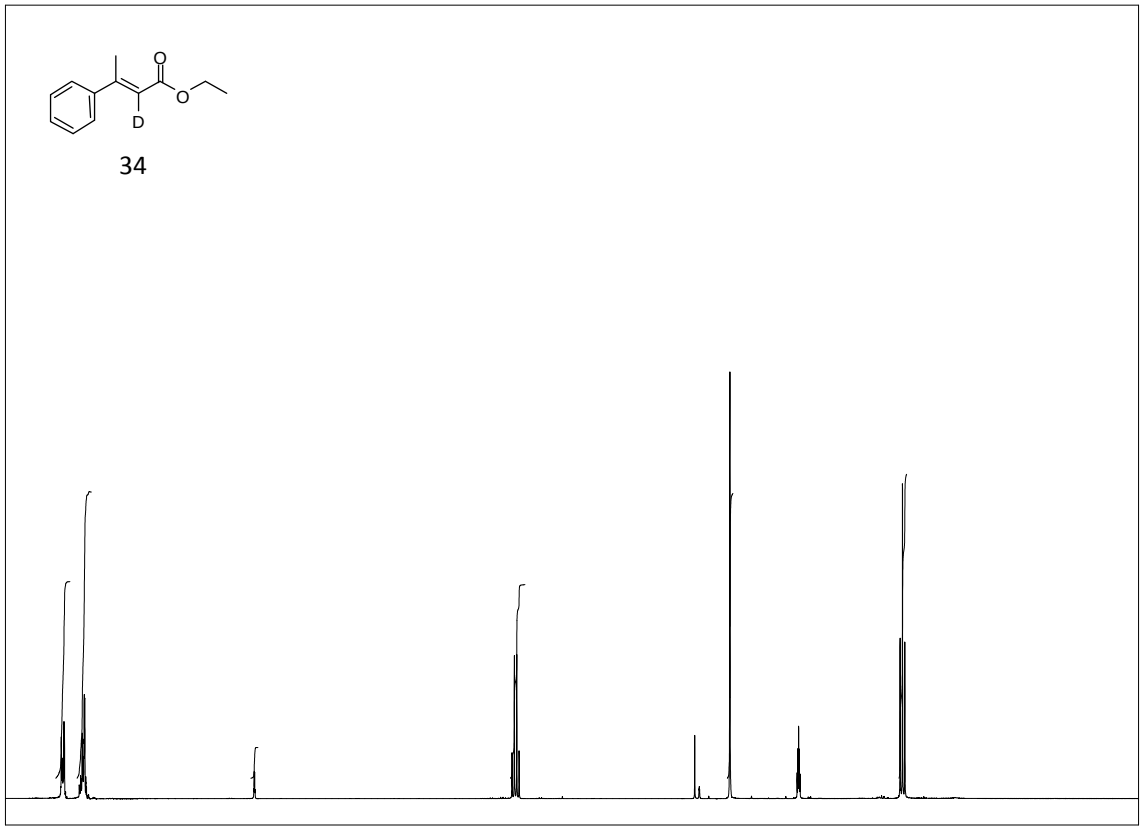

I
O
O
168.14
152.18
142.72
130.13
129.89
128.78
67.11
63.05
16.60
14.77
150
100
50
ppm (t1)


Me
OMe
CO2Et

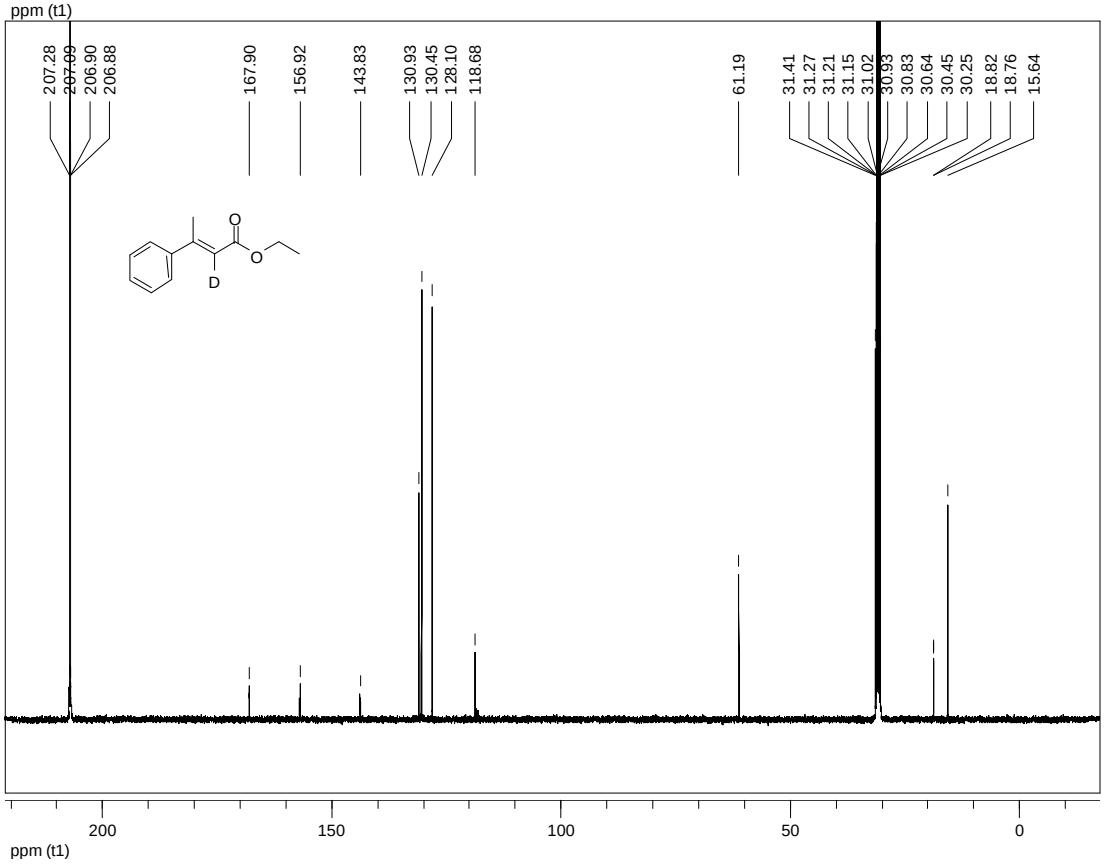
30

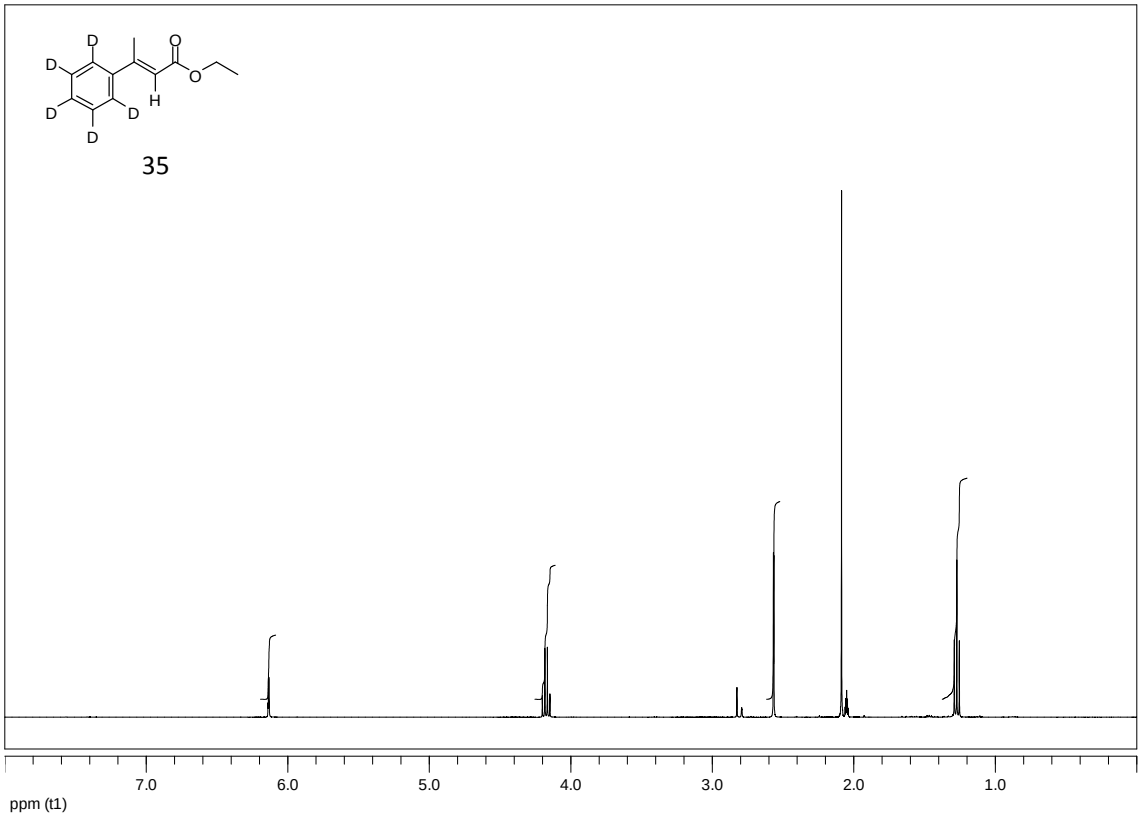

6.68
2.00
4.82
3.69
3.00
8.0
7.0
6.0
5.0
4.0
3.0
2.0
1.0

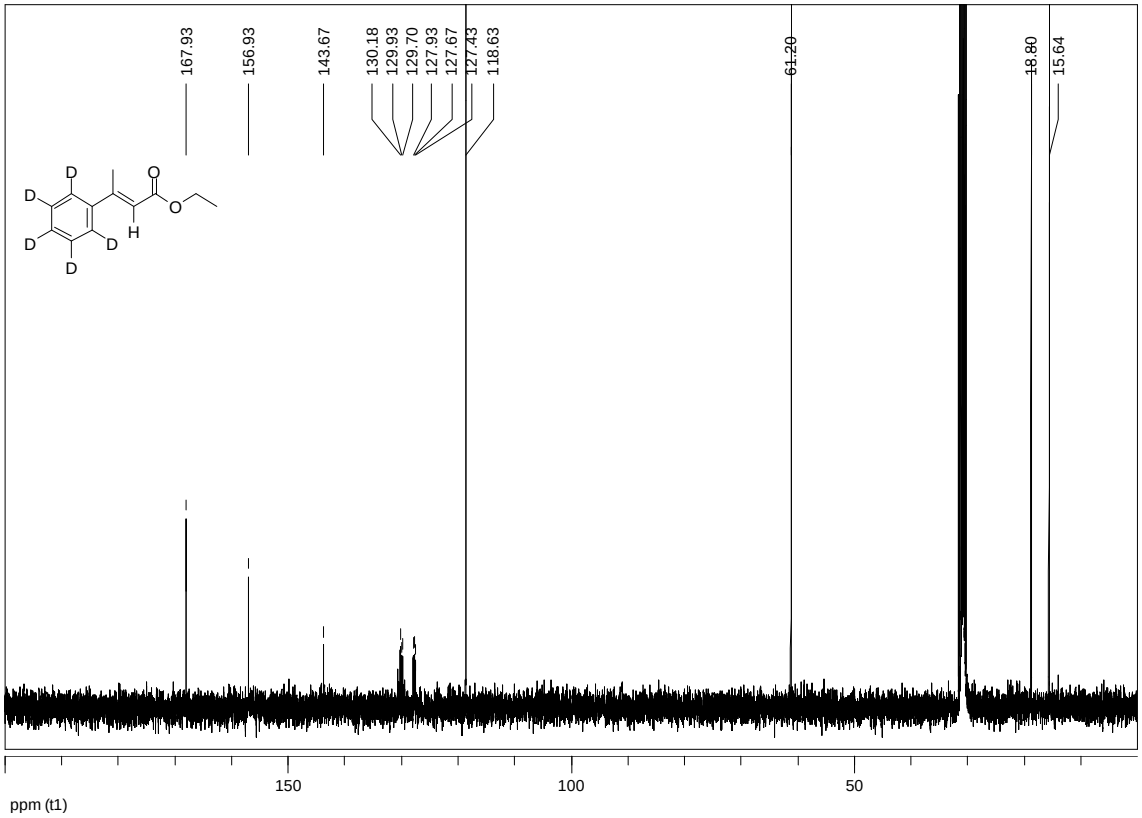

170.908
161.064
145.142
142.979
134.674
132.221
131.329
129.908
129.140
129.039
115.506
61.691
56.540
23.087
14.946
200
150
100
50
0
ppm (f1)

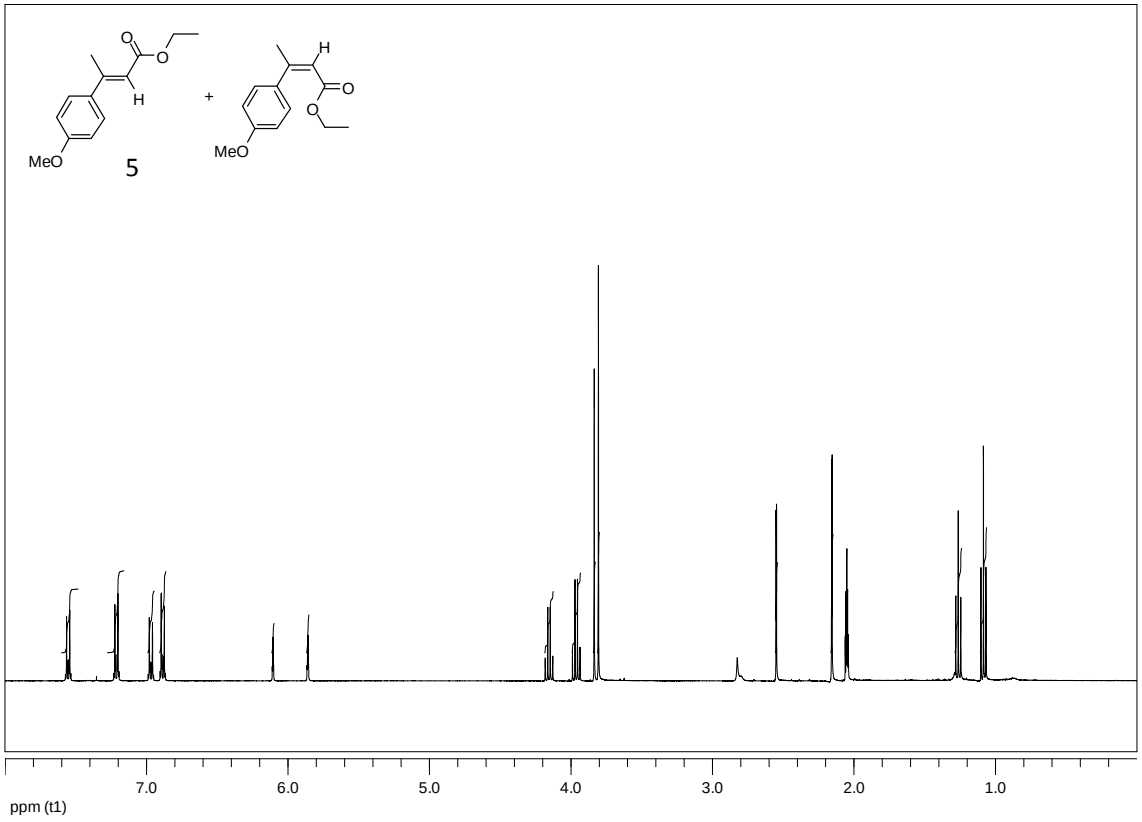

O
O
D
7.0
6.0
5.0
4.0
3.0
2.0
1.0
0.0
ppm (t1)

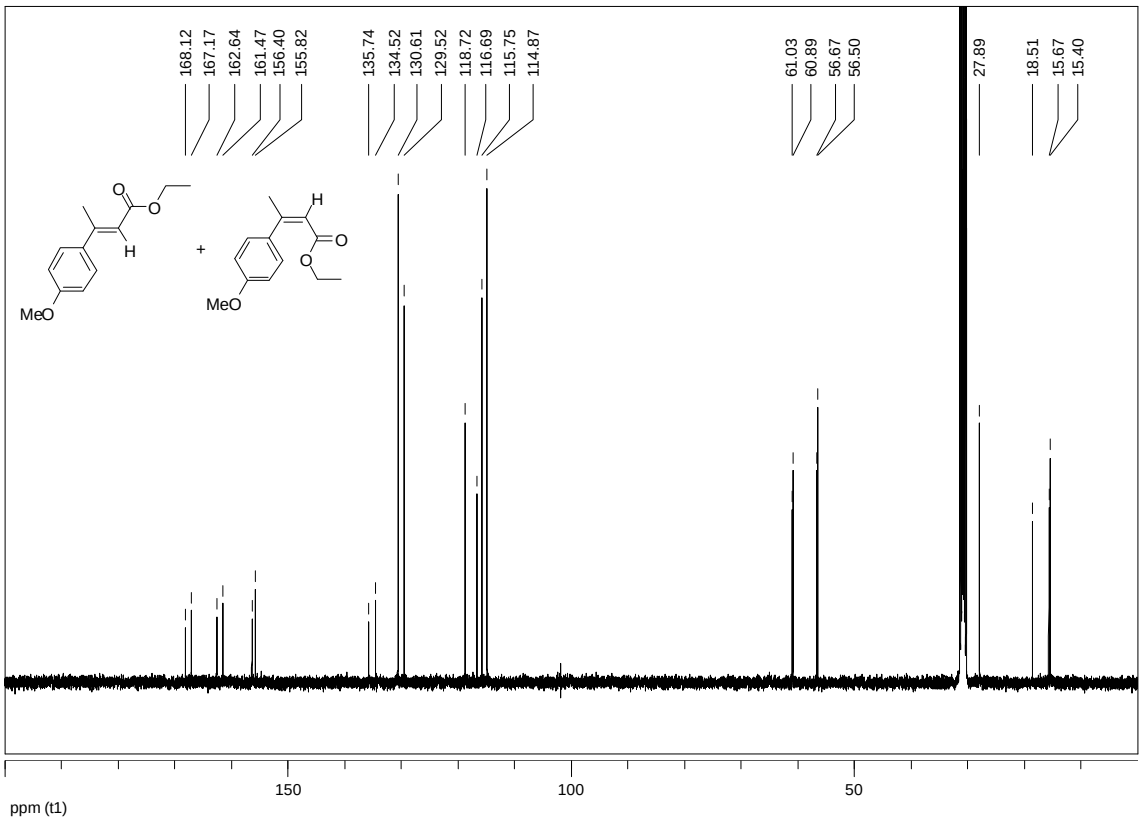
34

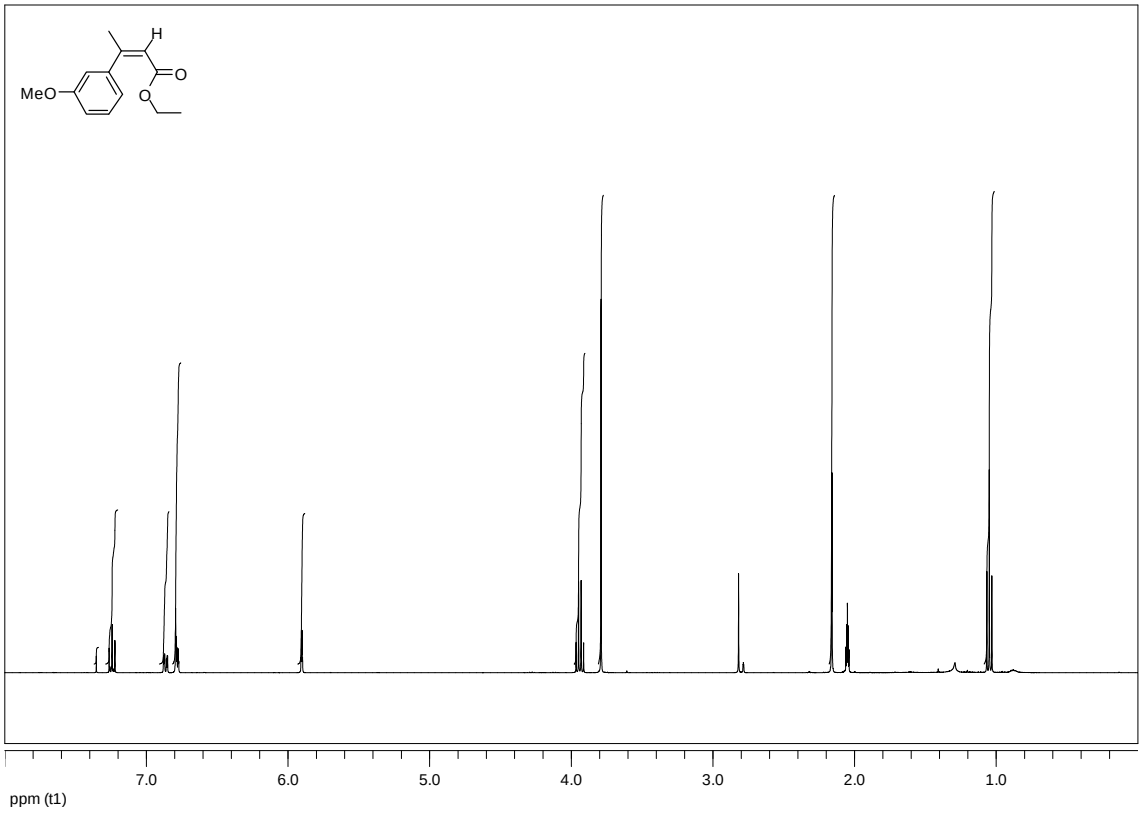

207.28
207.00
206.90
206.88
167.90
156.92
143.83
130.93
130.45
128.10
118.68
61.19
31.41
31.27
31.21
31.15
31.02
30.93
30.83
30.64
30.45
30.25
18.82
18.76
15.64
O
O
D
200
150
100
50
0
ppm (t1)

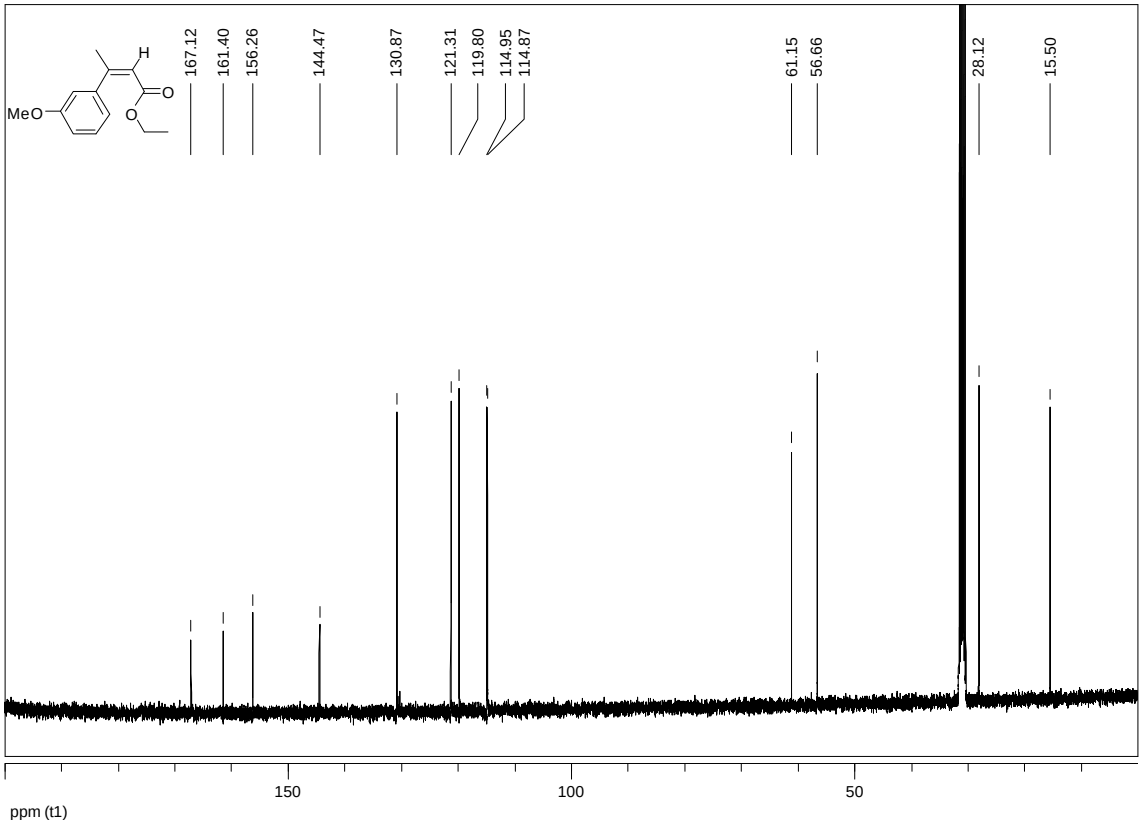

D
D
D
D
D
O
O
H
35
7.0
6.0
5.0
4.0
3.0
2.0
1.0
ppm (t1)

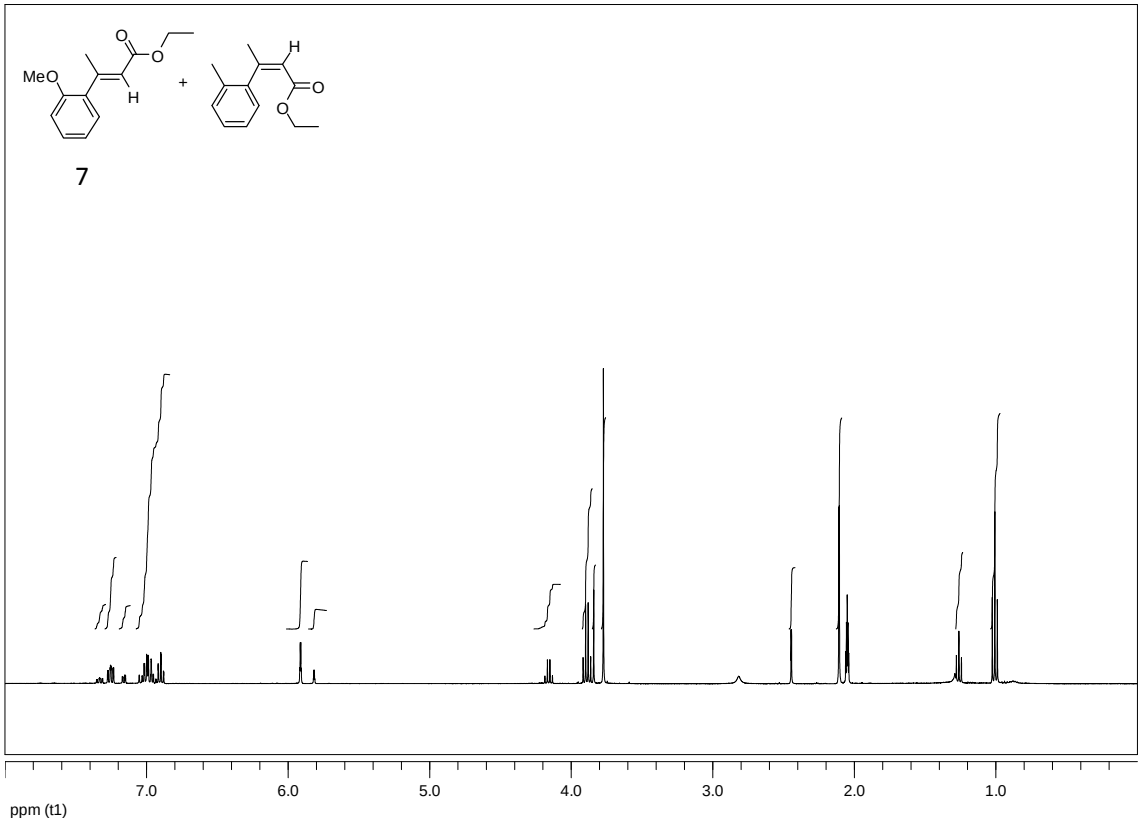

167.93
156.93
143.67
130.18
129.93
129.70
127.93
127.67
127.43
118.63
61.20
18.80
15.64
D
D
D
D
D
O
O
H
150
100
50
ppm (t1)

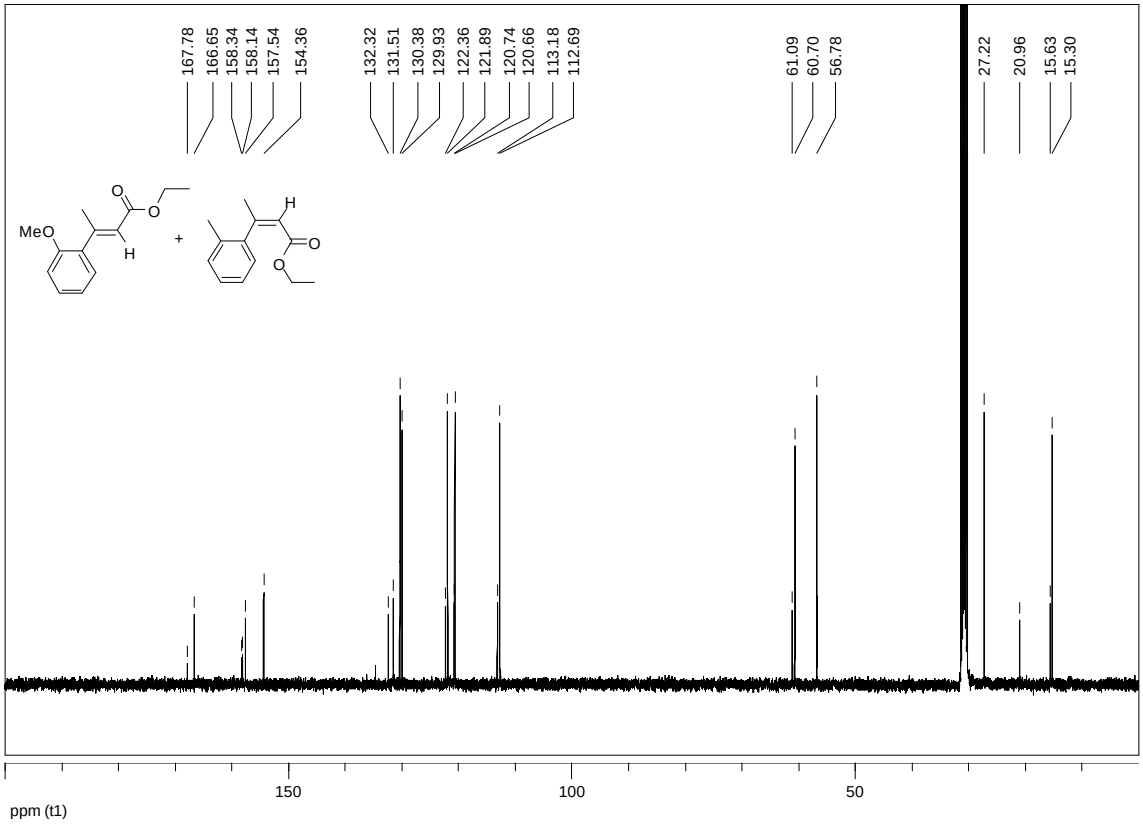

MeO
5
MeO
7.0
6.0
5.0
4.0
3.0
2.0
1.0
ppm (t1)

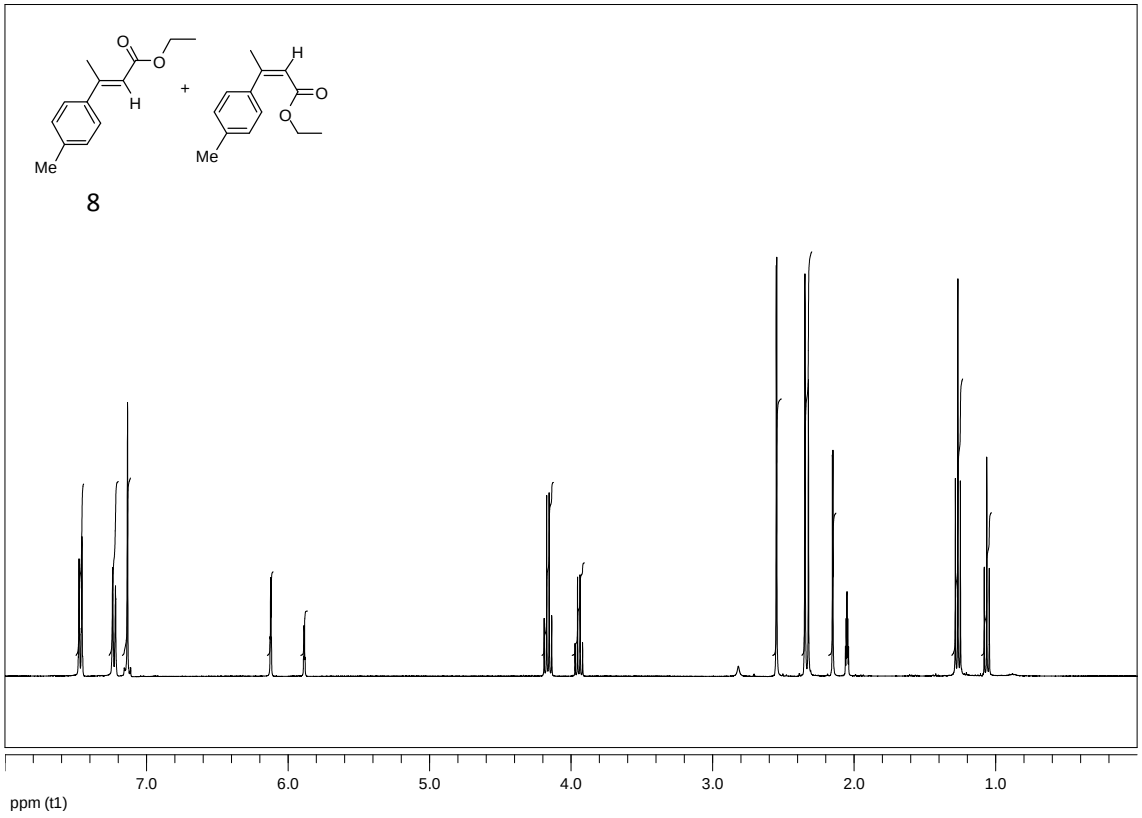

168.12
167.17
162.64
161.47
156.40
155.82
135.74
134.52
130.61
129.52
118.72
116.69
115.75
114.87
61.03
60.89
56.67
56.50
27.89
18.51
15.67
15.40
MeO
MeO
150
100
50
ppm (t1)


MeO
H
O
O
7.0
6.0
5.0
4.0
3.0
2.0
1.0
ppm (t1)

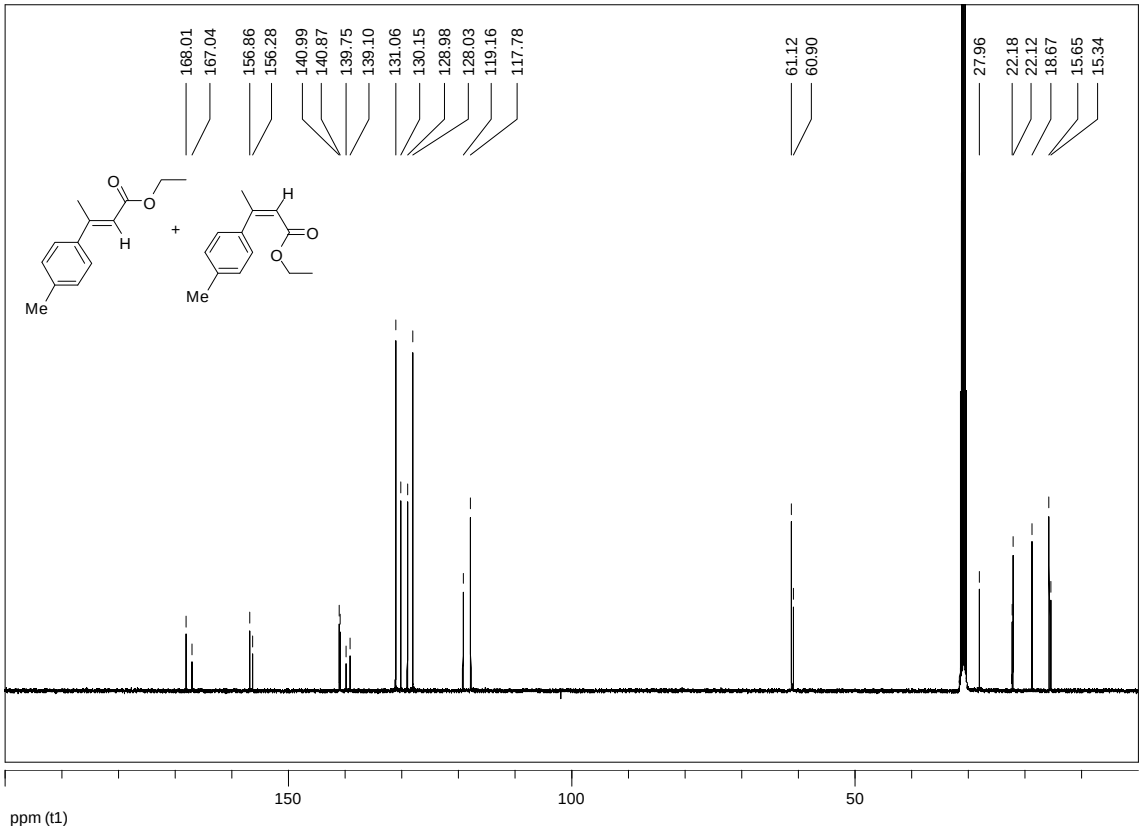

MeO
H
O
O
167.12
161.40
156.26
144.47
130.87
121.31
119.80
114.95
114.87
61.15
56.66
28.12
15.50
150
100
50
ppm (t1)

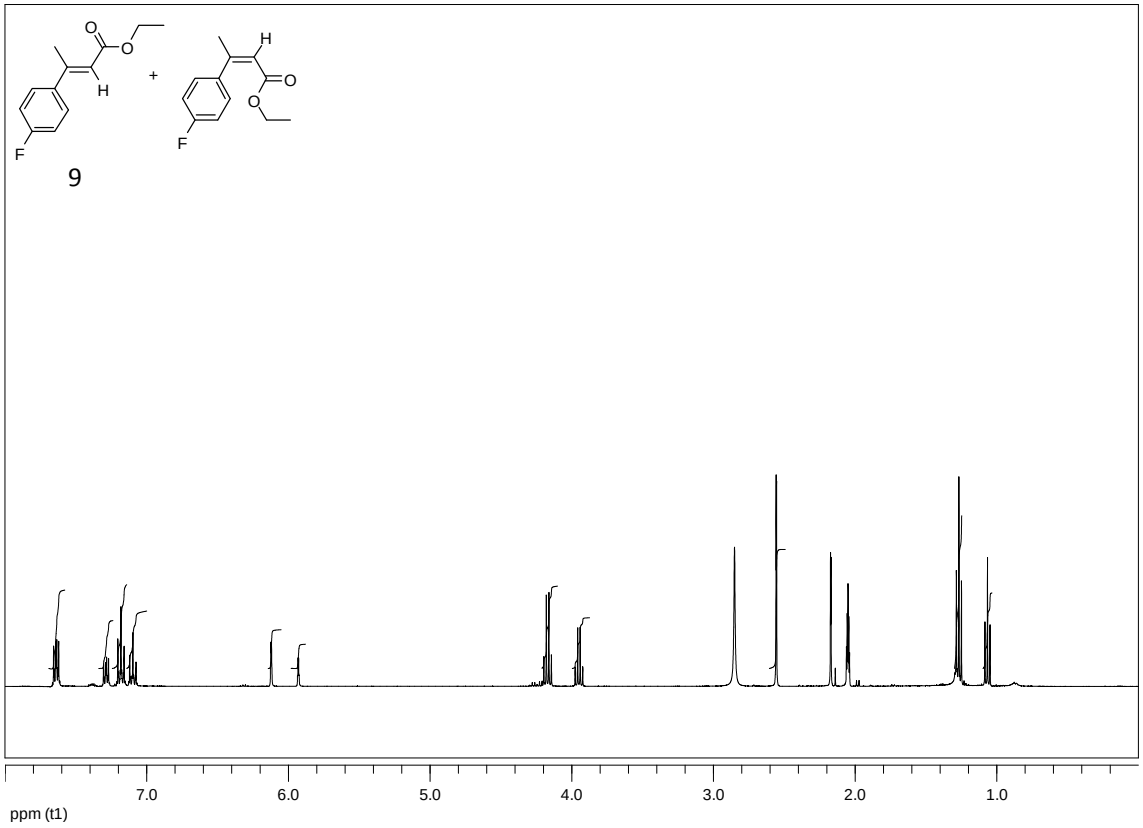

MeO
O
O
H
+
H
O
O
7
7.0
6.0
5.0
4.0
3.0
2.0
1.0
ppm (t1)

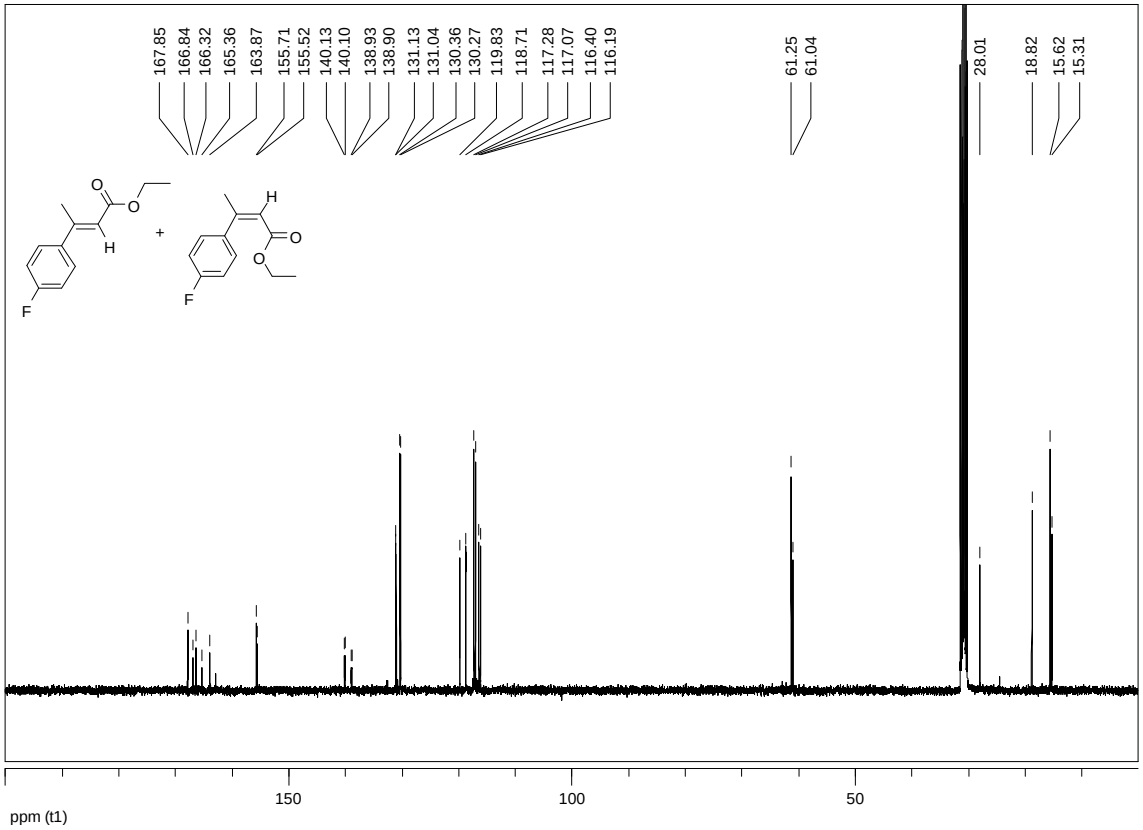

167.78
166.65
158.34
158.14
157.54
154.36
132.32
131.51
130.38
129.93
122.36
121.89
120.74
120.66
113.18
112.69
61.09
60.70
56.78
27.22
20.96
15.63
15.30
MeO
150
100
50
ppm (t1)

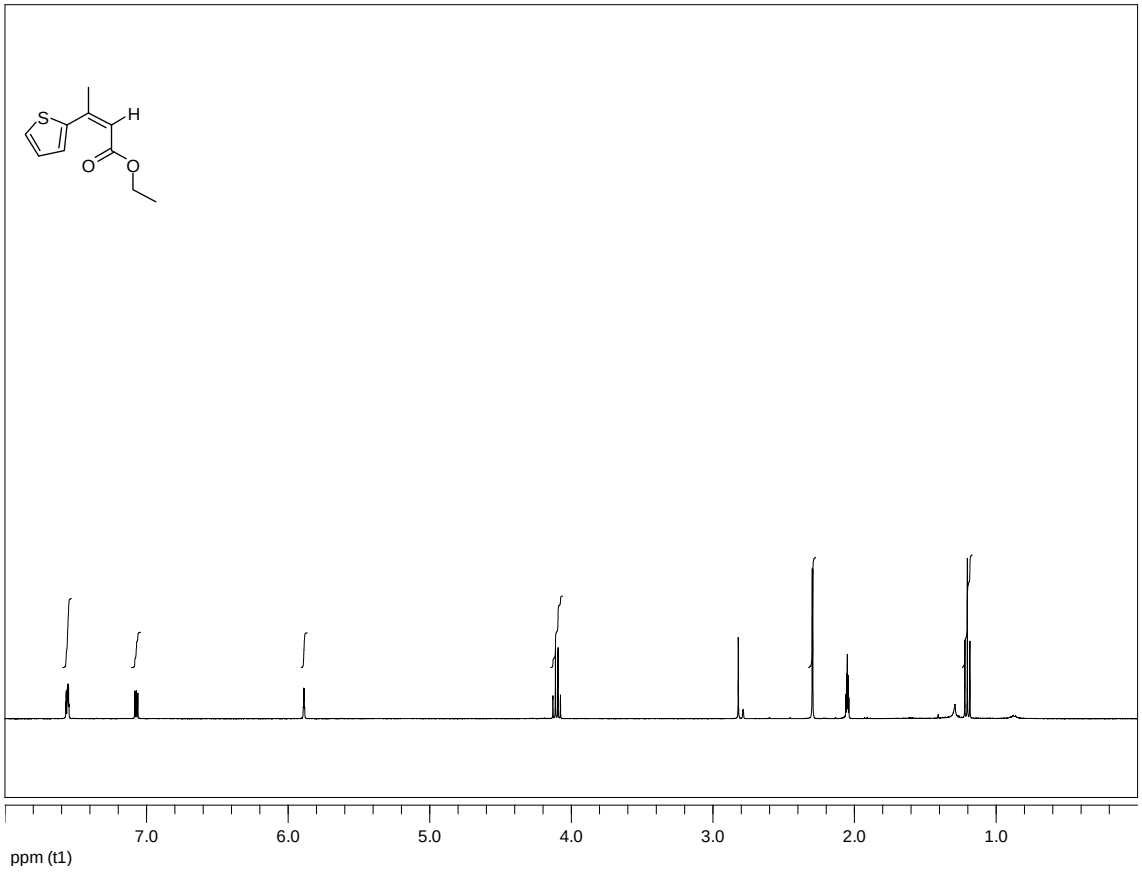

O
O
H
+
H
O
O
Me
Me
7.0
6.0
5.0
4.0
3.0
2.0
1.0
ppm (t1)

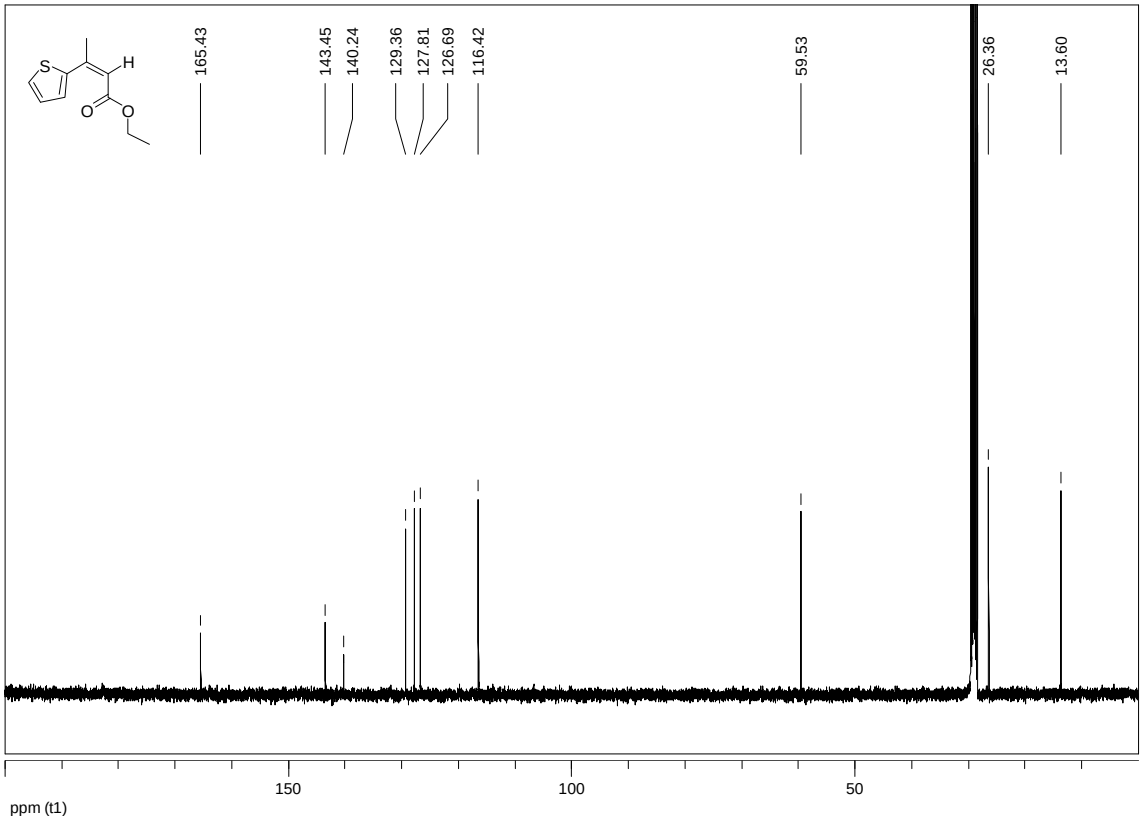
8

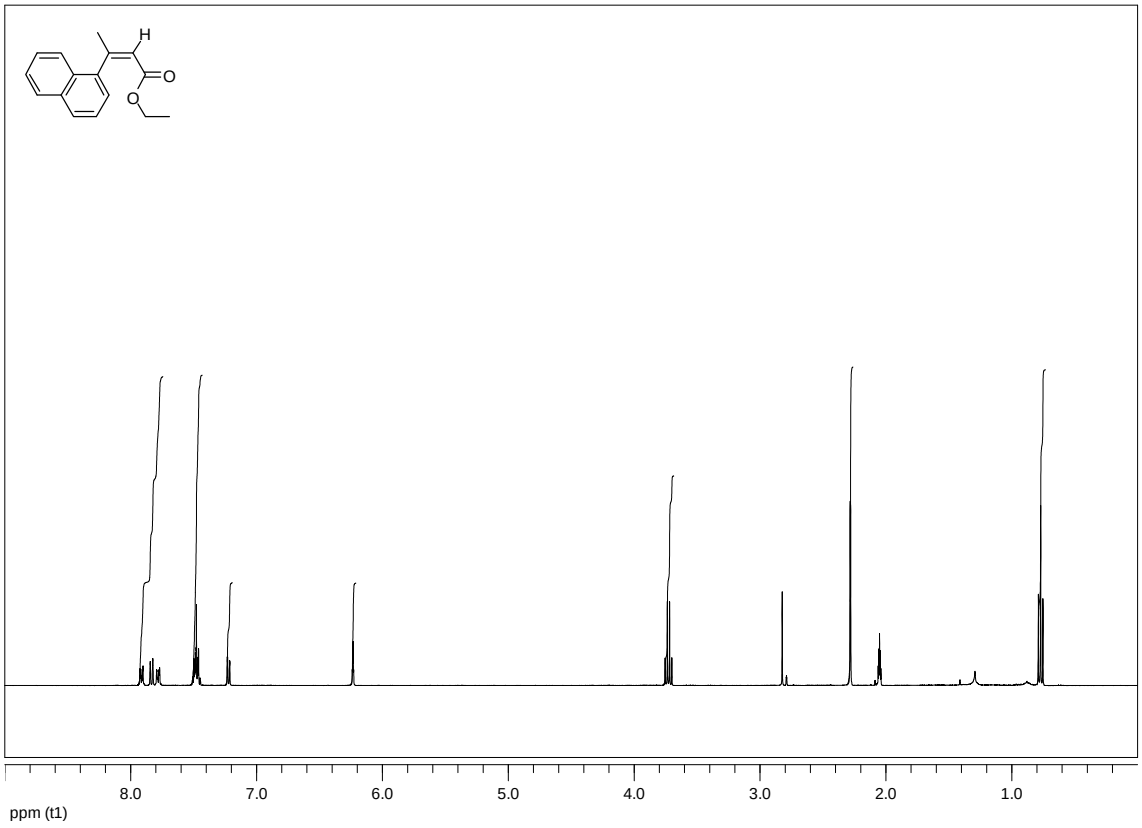

168.01
167.04
156.86
156.28
140.99
140.87
139.75
139.10
131.06
130.15
128.98
128.03
119.16
117.78
61.12
60.90
27.96
22.18
22.12
18.67
15.65
15.34
O
O
H
+
H
O
O
Me
Me
150
100
50
ppm (t1)

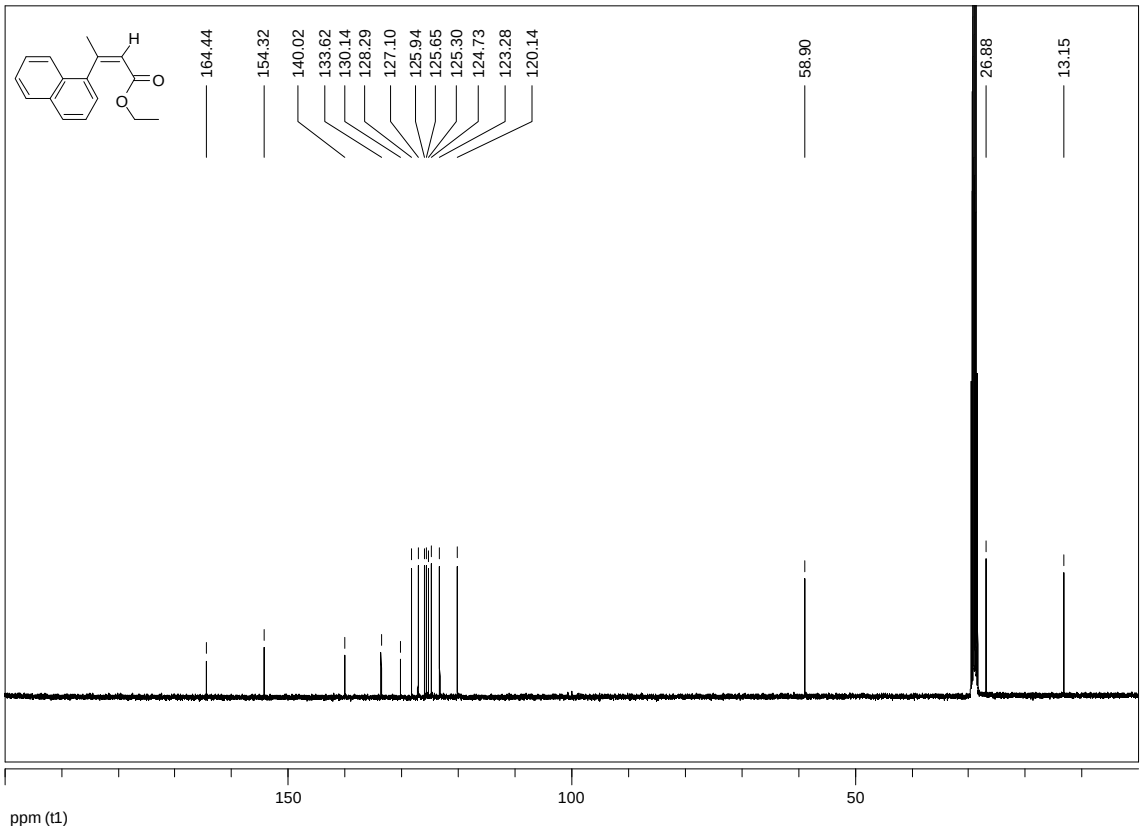

9
7.0
6.0
5.0
4.0
3.0
2.0
1.0
ppm (t1)

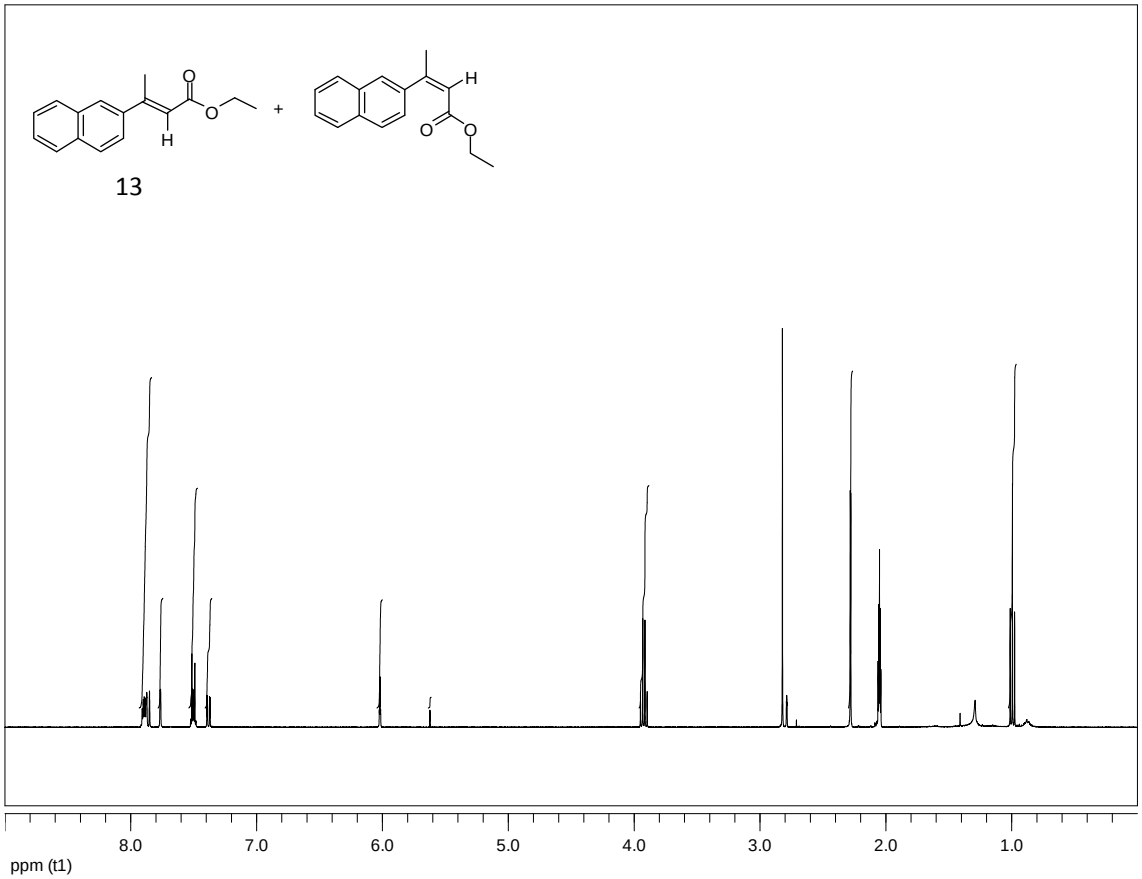

167.85
166.84
166.32
165.36
163.87
155.71
155.52
140.13
140.10
138.93
138.90
131.13
131.04
130.36
130.27
119.83
118.71
117.28
117.07
116.40
116.19
61.25
61.04
28.01
18.82
15.62
15.31
150
100
50
ppm (t1)

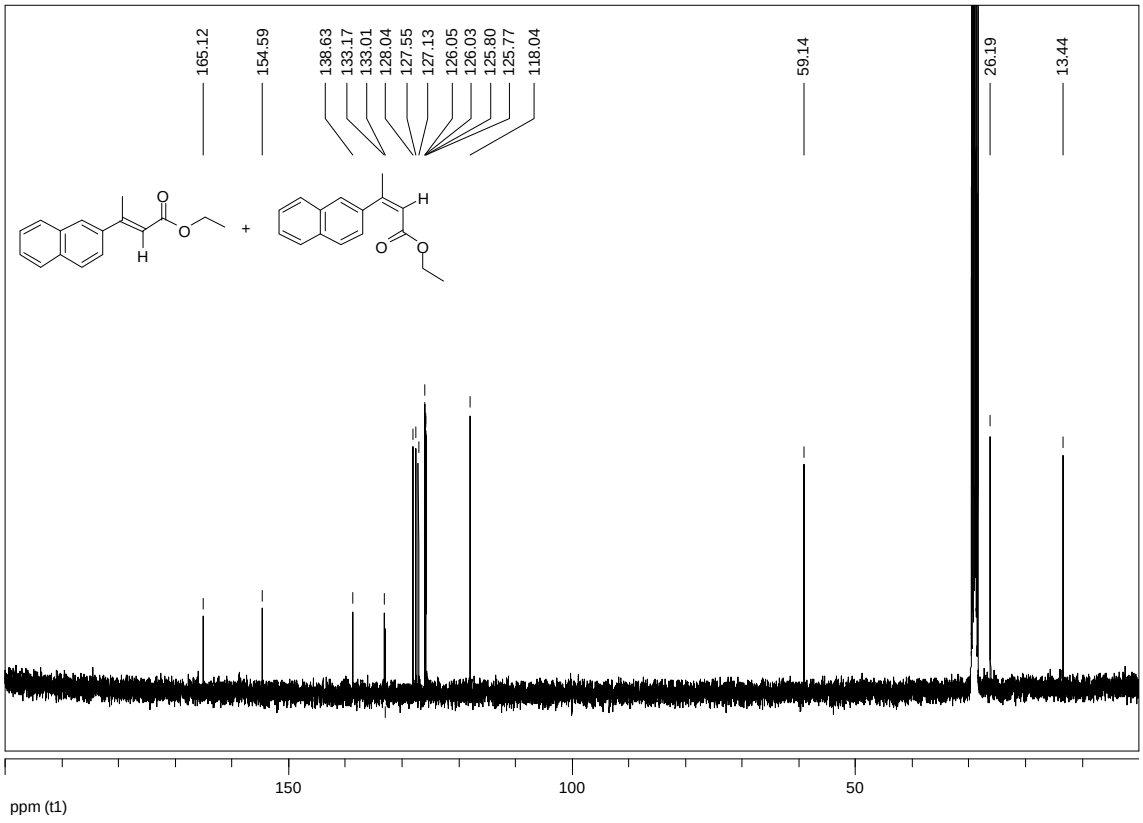

S
H
O
O
7.0
6.0
5.0
4.0
3.0
2.0
1.0
ppm (t1)

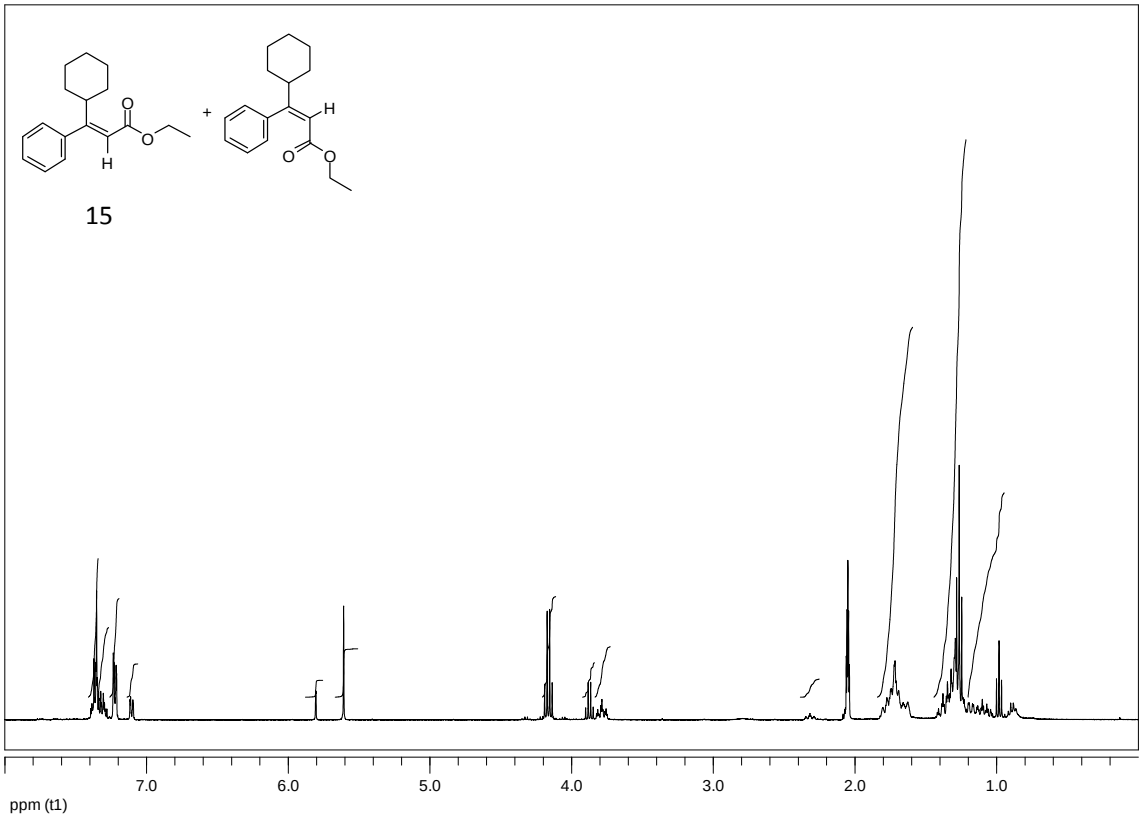

S
H
O
O
165.43
143.45
140.24
129.36
127.81
126.69
116.42
59.53
26.36
13.60
150
100
50
ppm (t1)


H
O
O
8.0
7.0
6.0
5.0
4.0
3.0
2.0
1.0
ppm (t1)

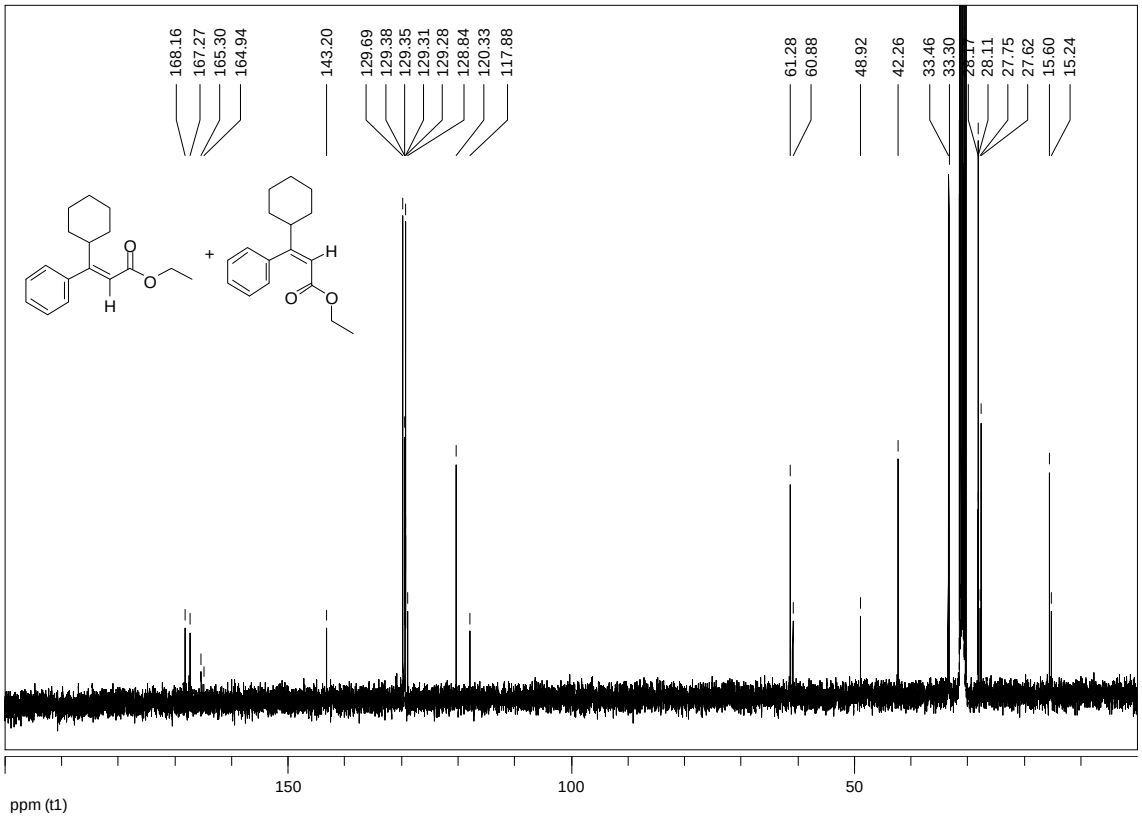

H
O
O
164.44
154.32
140.02
133.62
130.14
128.29
127.10
125.94
125.65
125.30
124.73
123.28
120.14
58.90
26.88
13.15
150
100
50
ppm (t1)

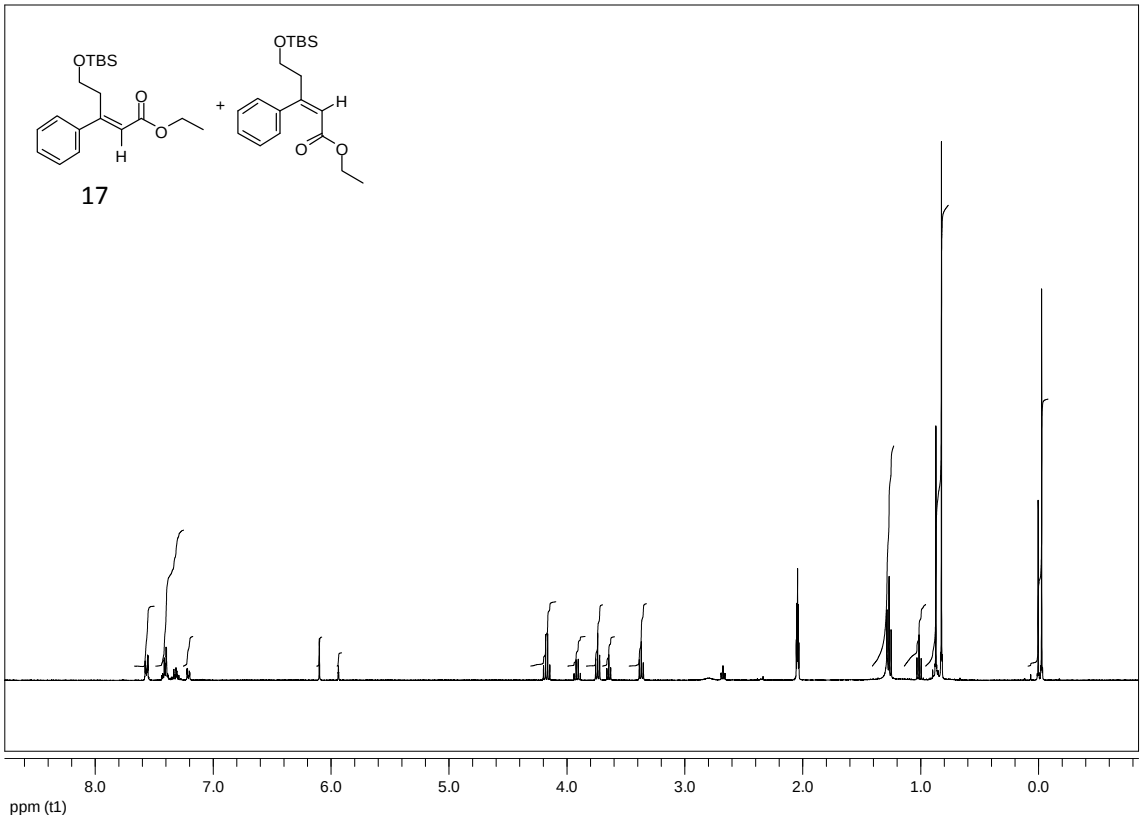

13
8.0
7.0
6.0
5.0
4.0
3.0
2.0
1.0
ppm (t1)

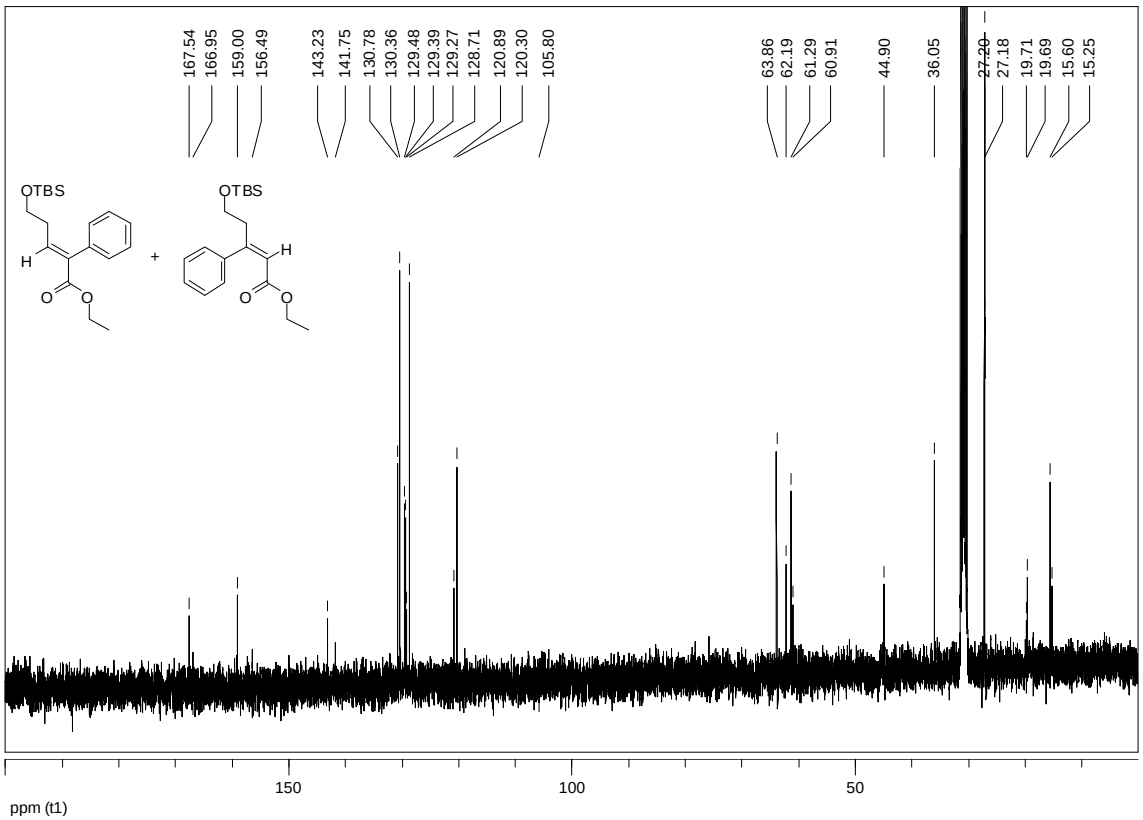

165.12
154.59
138.63
133.17
133.01
128.04
127.55
127.13
126.05
126.03
125.80
125.77
118.04
59.14
26.19
13.44
150
100
50
ppm (t1)

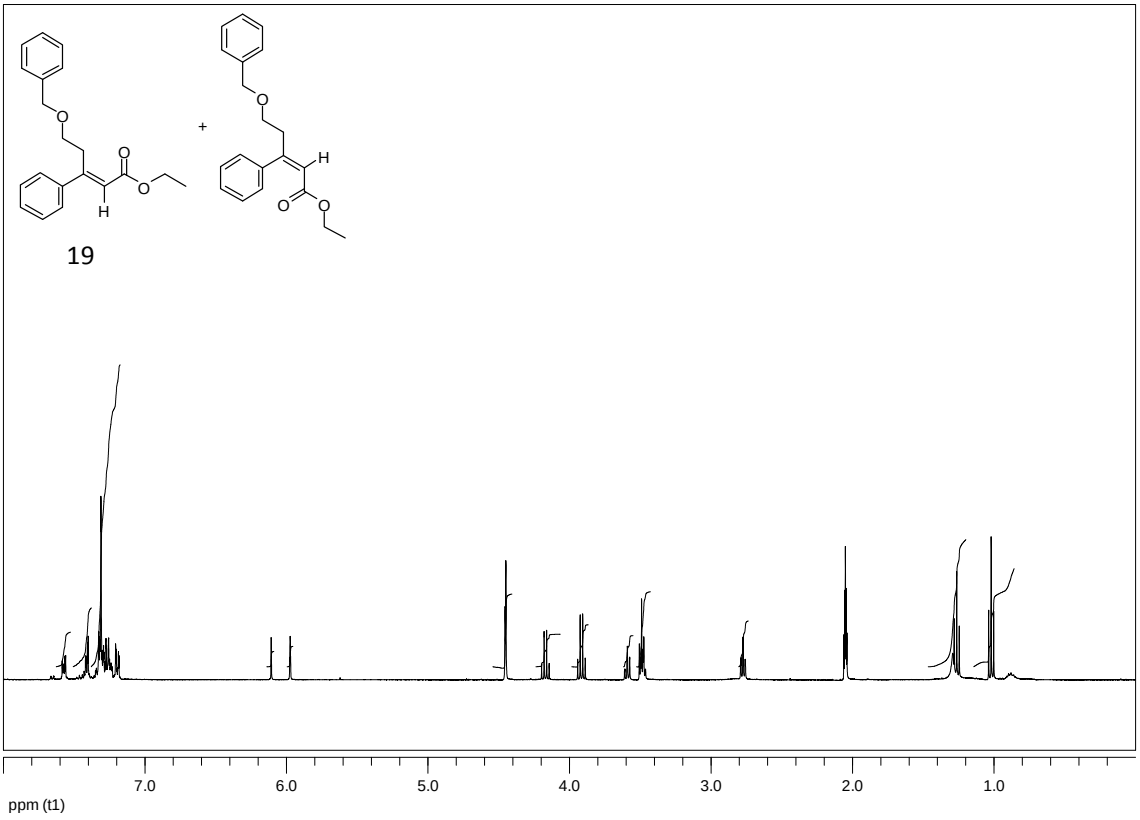

O
O
H
+
H
O
O
7.0
6.0
5.0
4.0
3.0
2.0
1.0
ppm (t1)

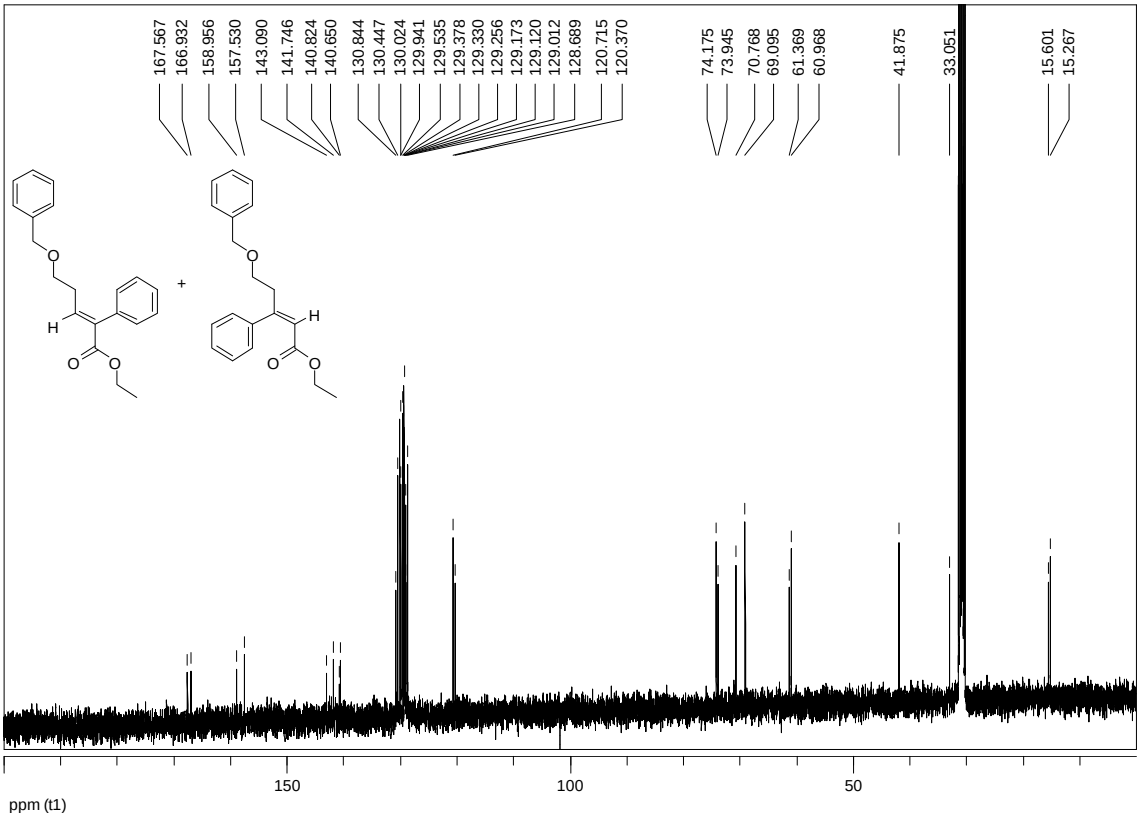
15

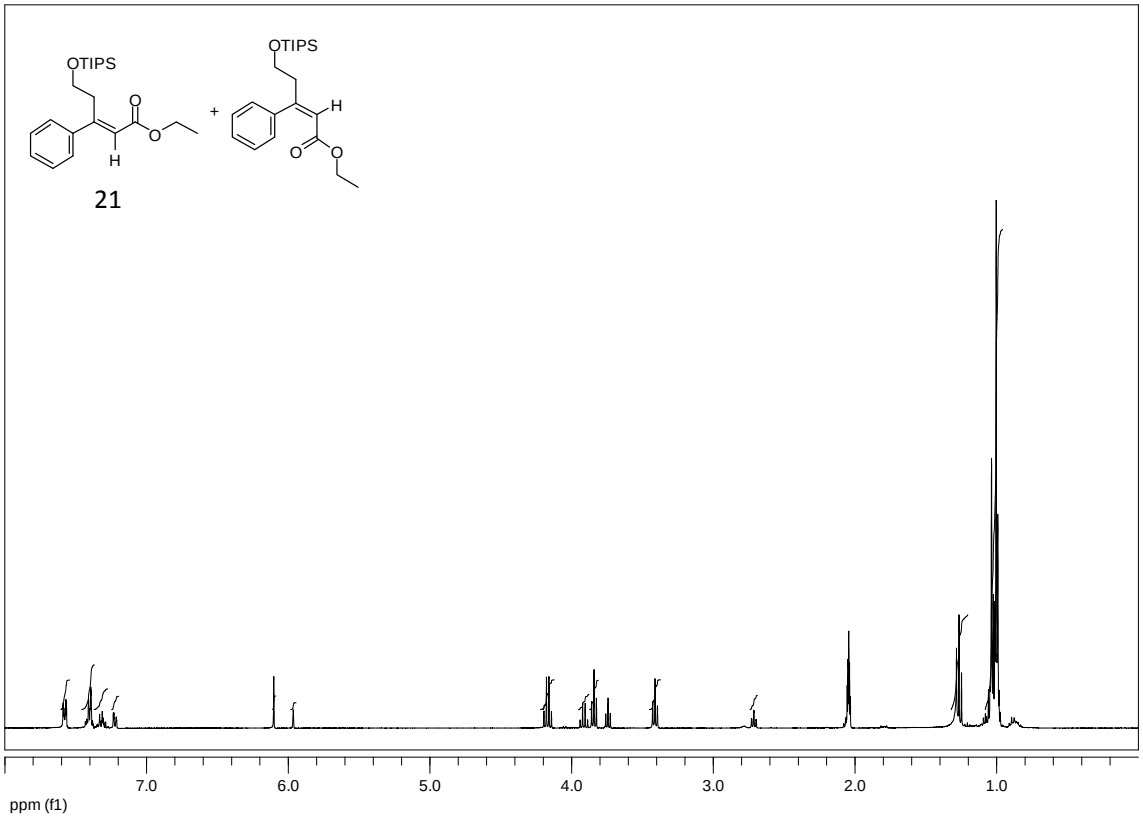

168.16
167.27
165.30
164.94
143.20
129.69
129.38
129.35
129.31
129.28
128.84
120.33
117.88
61.28
60.88
48.92
42.26
33.46
33.30
28.11
27.75
27.62
15.60
15.24
150
100
50
ppm (t1)


OTBS
O
O
H
+
OTBS
H
O
O
17
8.0
7.0
6.0
5.0
4.0
3.0
2.0
1.0
0.0
ppm (t1)

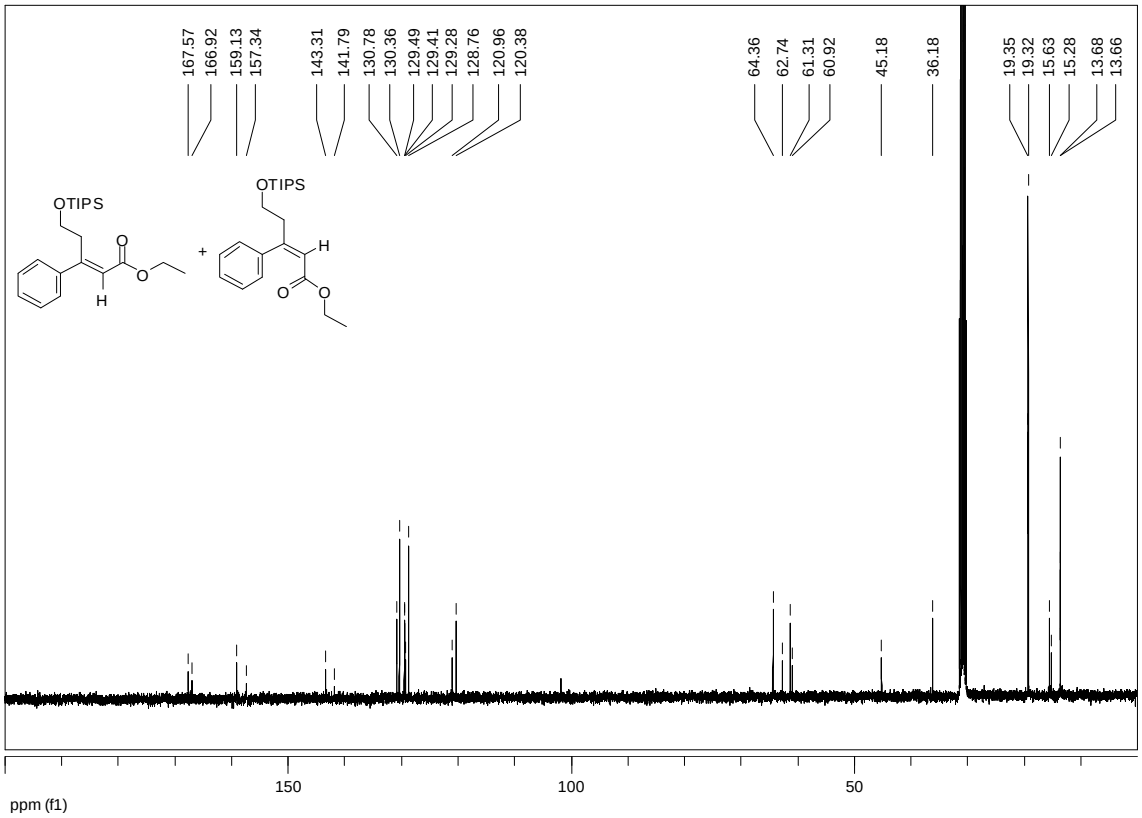

167.54
166.95
159.00
156.49
143.23
141.75
130.78
130.36
129.48
129.39
129.27
128.71
120.89
120.30
105.80
63.86
62.19
61.29
60.91
44.90
36.05
27.20
27.18
19.71
19.69
15.60
15.25
OTBS
H
O
O
+
OTBS
H
O
O
150
100
50
ppm (t1)


19
7.0
6.0
5.0
4.0
3.0
2.0
1.0
ppm (t1)


167.567
166.932
158.956
157.530
143.090
141.746
140.824
140.650
130.844
130.447
130.024
129.941
129.535
129.378
129.330
129.256
129.173
129.120
129.012
128.689
120.715
120.370
74.175
73.945
70.768
69.095
61.369
60.968
41.875
33.051
15.601
15.267
150
100
50
ppm (t1)


OTIPS
O
O
H
+
OTIPS
H
O
O
7.0
6.0
5.0
4.0
3.0
2.0
1.0
ppm (f1)

21


167.57
166.92
159.13
157.34
143.31
141.79
130.78
130.36
129.49
129.41
129.28
128.76
120.96
120.38
64.36
62.74
61.31
60.92
45.18
36.18
19.35
19.32
15.63
15.28
13.68
13.66
OTIPS
O
O
H
+
OTIPS
H
O
O
150
100
50
ppm (f1)