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Supporting Information

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What is Amphidinolide V? Report on a Likely Conquest

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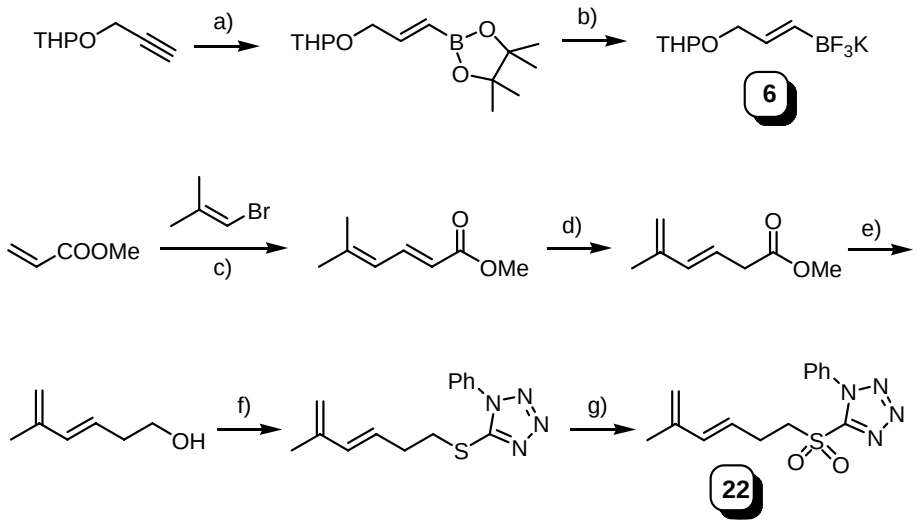
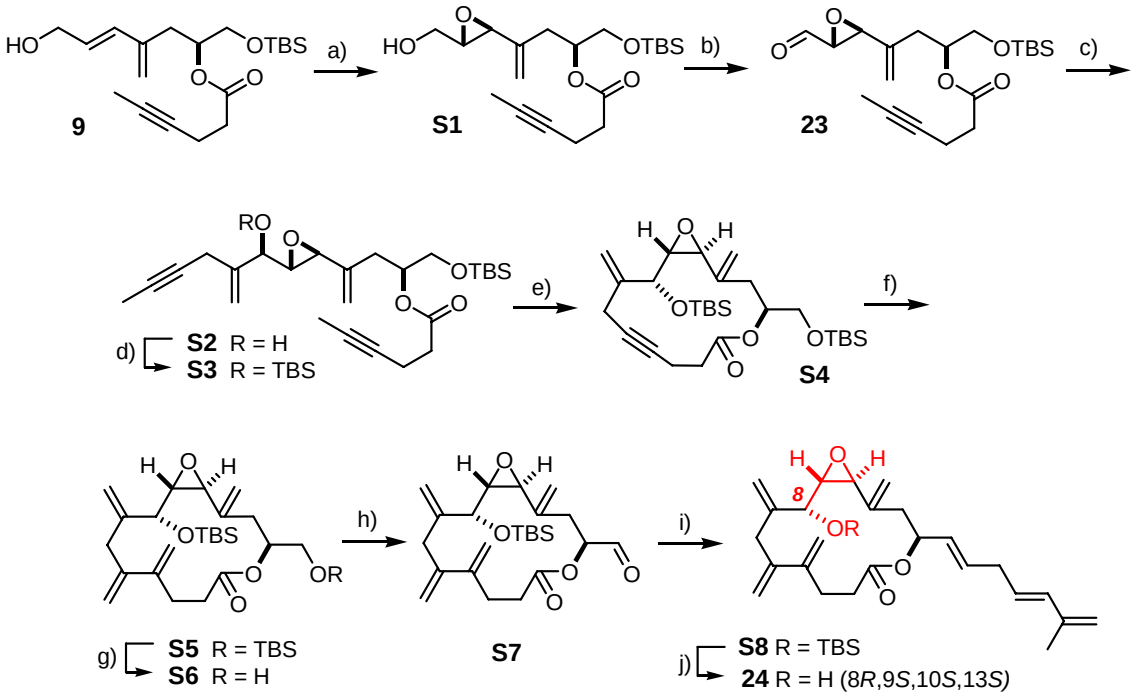
Building Blocks



Scheme S-1: a) Pinacolborane, $(cyclohexyl)_2BH$ cat., RT→35°C, 81%; b) KHF_2, $MeCN/H_2O$, 40°C, 83%; c) $Pd(OAc)_2$ cat., PPh_3 cat., Et_3N, 100°C, 86%; d) LiHMDS, THF/DMPU, −35°C, 99%; e) DIBAL-H, CH_2Cl_2, −78°C, 70%; f) DEAD, 1-phenyl-1*H*-tetrazol-5-thiol, THF, 63%; g) $(NH_4)_6Mo_7O_{24}{\cdot}4\ H_2O\ H_2O_2$, EtOH, 76%.

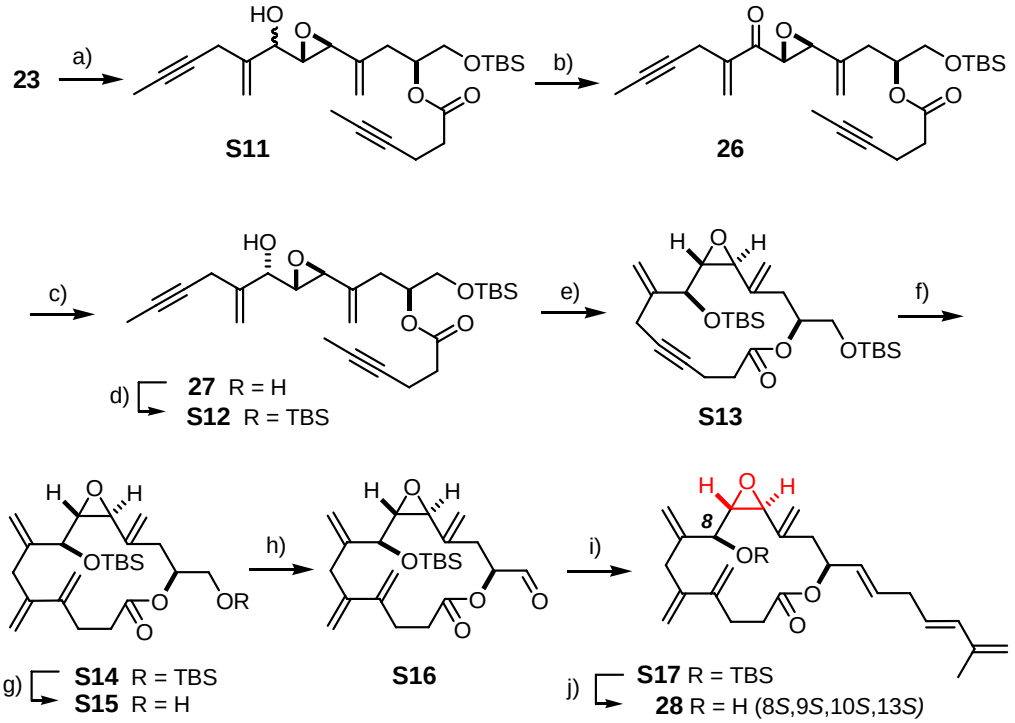
Preparation of the 8*R*,9*S*,10*S*,13*S*-Configured Isomer 24



Scheme S-2: (a) L-(+)-DET (40%), Ti(O*i*Pr)$_4$ (40%), *t*-BuOOH, MS 4 Å, CH_2Cl_2, –20°C, 92%; (b) Dess-Martin periodinane, $NaHCO_3$, CH_2Cl_2, 90%. (c) diorganozinc derivative **14**, toluene, (+)-*N*-methyl-ephedrine (60%), –25°C, 70% (dr ≥ 4:1); (d) TBSCl, imidazole, CH_2Cl_2, 10°C, 99%; (e) complex **20** (20 mol%), CH_2Cl_2/toluene, 85°C, 89%; (f) complex **21** (5 mol%), C_2H_4 (1.8 atm), toluene, 45°C, 96%; (g) catalytic PPTS, MeOH, 64%; (h) Dess-Martin periodinane, $NaHCO_3$, CH_2Cl_2, 89%; (i) sulfone **22**, KHMDS, THF, –78°C→RT, 66% (*E*:*Z*~3:1); (j) TASF, DMF, –5°C, 99%.

Isomer 24. $[\alpha]_D^{20}$ = –46.5 ($CHCl_3$, *c* = 0.31); ^{1}H NMR (400 MHz, $CDCl_3$): δ = 1.82 (3 H, s), 2.04 (1 H, dd, *J* = 10.5, 15.1 Hz), 2.19–2.25 (2 H, m), 2.29–2.41 (4 H, m), 2.81 (2 H, t, *J* = 6.4 Hz), 3.07 (2 H, s), 3.16 (1 H, dd, *J* = 2.0, 6.9 Hz), 3.33 (1 H, d, *J* = 2.0 Hz), 3.88 (1 H, dd, *J* = 2.8, 6.4 Hz), 4.85 (1 H, s), 4.87 (1 H, s), 4.88 (1 H, s), 5.00 (1 H, s), 5.02 (1 H, s), 5.16 (1 H, s), 5.21 (1 H, s), 5.25 (1 H, s), 5.31 (1 H, s), 5.34 (1 H, s), 5.37–5.43 (2 H, m), 5.58 (1 H, dt, *J* = 6.8, 15.6 Hz), 5.75 (1 H, dt, *J* = 6.4, 14.5 Hz), 6.11 (1 H, d, *J* = 15.6 Hz) ppm; ^{13}C NMR (100.6 MHz, $CDCl_3$): δ = 18.6 (CH_3), 30.4 (CH_2), 33.2 (CH_2), 34.3 (CH_2), 35.1 (CH_2), 37.2 (CH_2), 59.7 (CH), 59.8 (CH), 74.2 (CH), 76.2 (CH), 113.1 (CH_2), 114.0 (CH_2), 115.1 (CH_2), 115.8 (CH_2), 117.7 (CH_2), 127.2 (CH), 128.7 (CH), 132.1 (CH), 134.2 (CH), 140.6 (C), 141.8 (C), 144.0 (C), 145.0 (C), 146.5 (C), 172.1 (C) ppm; IR (NaCl): $\tilde{\nu}$ = 3450, 3080, 2920, 1732, 1644, 1607, 1435, 1263, 1234, 1157, 1131, 1032, 967, 891 cm^{-1}; MS (EI): *m/z* (%): 396 [M$^+$] (<1), 201 (8), 177, (21), 173 (12), 159 (21), 157 (13), 147 (17), 145 (18), 143 (19), 135 (18), 131 (44), 129 (21), 121 (34), 119 (46), 117 (31), 105 (65), 93 (66), 91 (100), 81 (55), 79 (86), 55 (60), 41 (58); HRMS (ESI+) calcd for $C_{25}H_{32}O_4Na$ [M+Na$^+$]: 419.2196, found: 419.2193.

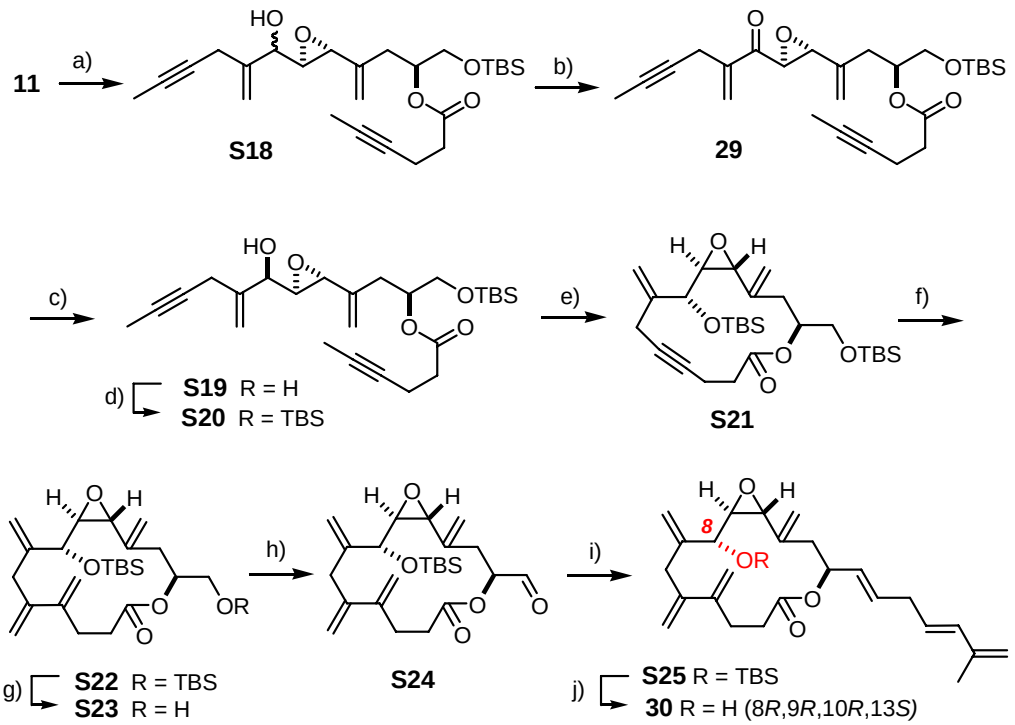
Preparation of the 8*S*,9*S*,10*S*,13*S*-Configured Isomer 28



Scheme S-4: (a) Grignard reagent **25**, THF, –10°C, 86%; (b) Dess-Martin periodinane, CH_2Cl_2; (c) $NaBH_4$, $CaCl_2$, MeOH, 0°C, 71% (over both steps, *dr* = 11:1); (d) TBSCl, imidazole, CH_2Cl_2, 10°C, 87%; (e) complex **20** (20 mol%), CH_2Cl_2/toluene, 85°C, 75%; (f) complex **21** (2 mol%), C_2H_4 (1.8 atm), toluene, 45°C, 92%; (g) catalytic PPTS, MeOH, 61%; (h) Dess-Martin periodinane, $NaHCO_3$, CH_2Cl_2, 97%; (i) sulfone **22**, KHMDS, DME/DMPU (50:1), –78°C→RT, 98% (*E*:*Z* > 10:1); (j) TASF, DMF, –5°C, 78%.

Isomer 28. $[\alpha]_D^{20}$ = +6.9 ($CHCl_3$, *c* = 0.345); ^{1}H NMR (400 MHz, $CDCl_3$): δ = 1.83 (3 H, s), 1.92–2.19 (2 H, m), 2.27–2.45 (2 H, m), 2.33–2.45 (2 H, m), 2.83 (2 H, dd, *J* = 6.5, 6.4 Hz), 3.04 (2 H, brs), 3.22 (1 H, dd, *J* = 2.2, 2.2 Hz), 3.59 (1 H, d, *J* = 2.2 Hz), 4.57 (1 H, s), 4.82 (1 H, s), 4.89 (2 H, brs), 5.02 (2 H, brs), 5.14 (1 H, s), 5.24 (1 H, s), 5.26 (1 H, s), 5.29 (1 H, s), 5.33 (1H, s), 5.40 (1 H, brs), 5.40–5.45 (1 H, m), 5.60 (1 H, dt, *J* = 15.6, 6.8 Hz), 5.77 (1 H, dt, *J* = 14.2, 6.3 Hz), 6.13 (1 H, d, *J* = 15.7 Hz) ppm; ^{13}C NMR (400 MHz, $CDCl_3$): δ = 18.6 (CH_3), 29.8 (CH_2), 31.7 (CH_2), 34.1 (CH_2), 35.1 (CH_2), 37.7 (CH_2), 56.4 (CH), 57.8 (CH), 70.4 (CH), 74.3 (CH), 112.6 (CH_2), 113.8 (CH_2), 115.1 (CH_2), 115.7 (CH_2), 117.9 (CH_2), 127.3 (CH), 128.9 (CH), 131.9 (CH), 134.3 (CH), 140.8 (C), 141.8 (C), 144.4 (C), 144.4 (C), 146.0 (C), 172.1 (C) ppm; IR (NaCl): $\tilde{\nu}$ = 3480, 3081, 3018, 2985, 2921, 1735, 1640, 1608, 1435, 1374, 1262, 1239, 1155, 1089, 969, 896 cm^{-1}; MS (EI): *m/z* (%): 159 (27), 131 (58), 105 (70), 91 (100), 79 (84), 55 (52); HRMS (ESI+) calcd for $C_{25}H_{32}O_4Na$ [M+Na$^+$]: 419.2193, found: 419.2196.

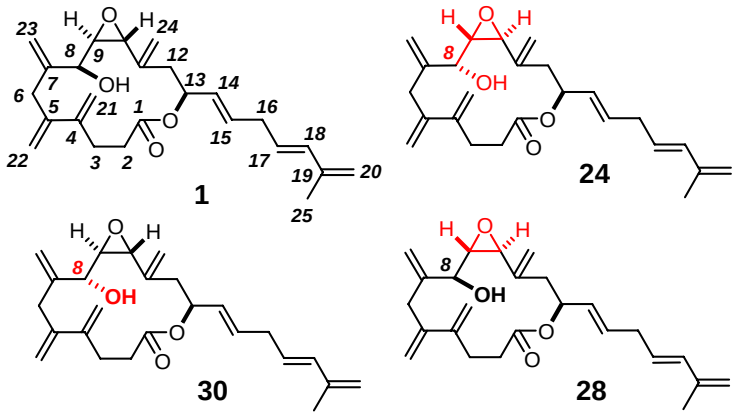
Preparation of the 8*R*,9*R*,10*R*,13*S*-Configured Isomer 30



Scheme S-5: (a) Grignard reagent **25**, THF, −10°C, 70%; (b) Dess-Martin periodinane, CH_2Cl_2; (c) $NaBH_4$, $CaCl_2$, MeOH, 0°C, 70% (over two steps); (d) TBSCl, imidazole, CH_2Cl_2, 10°C, 93%; (e) complex **20** (20 mol%), CH_2Cl_2/toluene, 85°C, 93%; (f) complex **21** (10 mol%), C_2H_4 (1.8 atm), toluene, 45°C, 73%; (g) catalytic PPTS, MeOH, 85%; (h) Dess-Martin periodinane, $NaHCO_3$, CH_2Cl_2, 60%; (i) sulfone **22**, KHMDS, THF, −78°C→RT, 75% (*E*:*Z* ~ 3:1); (j) TASF, DMF, −5°C, 60%.

Isomer 30. This compound was found to be unstable and could not be obtained in analytically pure form; the following characteristic data, however, could be extracted: ^{1}H NMR (400 MHz, $CDCl_3$): δ = 1.83 (3 H, s), 2.20–2.57 (2 H, m), 2.27–2.57 (2 H, m), 2.39–2.56 (2 H, m), 2.83 (2 H, dd, *J* = 6.4 Hz), 3.01 (1 H, d, *J* = 16.4 Hz), 3.02 (1 H, dd, *J* = 2.1, 2.1 Hz), 3.18 (1 H, d, *J* = 16.4 Hz), 3.59 (1 H, d, *J* = 1.7 Hz), 4.51 (1 H, br s), 4.90 (2 H, s), 4.94 (1 H, s), 5.04 (1 H, s), 5.08 (1 H, s), 5.12 (1 H, s), 5.18 (1 H, s), 5.24 (1 H, s), 5.26 (1 H, s), 5.29 (1 H, m), 5.36 (1 H, s), 5.42 (1 H, m), 5.60 (1 H, dt, *J* = 14.0, 6.7 Hz), 5.74 (1 H, dt, *J* = 6.7, 14.7 Hz), 6.13 (1 H, d, *J* = 15.0 Hz) ppm; ^{13}C NMR (100.6 MHz, $CDCl_3$): δ = 18.6 (CH_3), 29.7 (CH_2), 33.9 (CH_2), 35.1 (CH_2), 36.5 (CH_2), 37.9 (CH_2), 55.6 (CH), 62.3 (CH), 71.8 (CH), 74.1 (CH), 113.5 (CH_2), 115.0 (CH_2), 115.1 (CH_2), 115.8 (CH_2), 116.5 (CH_2), 127.4 (CH), 129.6 (CH), 131.6 (CH), 134.2 (CH), 140.6 (C), 141.1 (C), 144.2 (C), 146.6 (C), 172.0 (C) ppm; IR (NaCl): $\tilde{\nu}$ = 3444, 2922, 1732, 1438, 1259, 1156, 900 cm^{-1}; MS (ESI+): *m/z*: 419 (100) [M+Na$^+$], 396 (1) [M$^+$]; HRMS (ESI+) calcd for $C_{25}H_{32}NaO_4$ [M+Na$^+$]: 419.2193, found: 419.2195.

Table S1. Comparison of the ^{13}C NMR ($CDCl_3$) Data of Compounds **1**, **24**, **28** and **30** with those of Amphidinolide V Reported in the Literature;[1] Arbitrary Numbering Scheme as shown.



Nr	Lit.[1]	1	Δδ[a]	24	Δδ[a]	28	Δδ[a]	30	Δδ[a]
1	171.3	171.8	0.5	172.1	0.8	172.1	0.8	172.0	0.7
2	33.5	33.7	0.2	34.3	0.8	34.1	0.6	36.5	3.0
3	30.2	30.5	0.3	30.4	0.2	29.8	–0.4	29.7	–0.5
4	144.5	144.6	0.1	146.5	2.0	146.0	1.5	144.2	–0.3
5	141.4	141.8	0.4	144.0	2.6	144.4	3.0	140.6	–0.8
6	39.0	39.1	0.1	37.2	–1.8	37.7	–1.3	37.9	–1.1
7	144.7	144.9	0.2	145.0	0.3	144.4	–0.3	146.6	1.0
8	71.1	71.4	0.3	76.2	5.1	70.4	–0.7	71.8	0.7
9	63.2	63.2	0.0	59.8	–3.4	57.8	–5.4	62.3	–0.9
10	57.7	57.9	0.2	59.7	2.0	56.4	–1.3	55.6	–2.1
11	n.o.[b]	140.7		140.6		140.8		n.o.[b]	
12	38.9	39.0	0.1	33.2	–5.7	31.7	–7.2	35.1	–3.8
13	74.3	74.3	0.0	74.2	–0.1	74.3	0.0	74.1	–0.2
14	127.6	128.0	0.4	128.7	1.1	128.9	1.3	129.6	2.0
15	131.2	132.0	0.8	132.1	0.9	131.9	0.7	131.6	0.4
16	34.9	35.0	0.1	35.1	0.2	35.1	0.2	33.9	–1.0
17	127.1	127.4	0.3	127.2	0.1	127.3	0.2	127.4	0.3
18	133.3	134.1	0.8	134.2	0.9	134.3	1.0	134.2	0.9
19	141.5	141.8	0.3	141.8	0.3	141.8	0.3	141.1	–0.4
20	114.8	115.1	0.3	115.1	0.3	115.1	0.3	115.1	0.3
21	114.4	114.8	0.4	113.1	–1.3	112.6	–1.8	115.0	0.6
22	113.6	114.0	0.4	115.8	2.2	115.7	2.1	113.5	–0.1
23	114.4	114.8	0.4	114.0	–0.4	113.8	–0.6	115.8	1.4
24	114.8	115.0	0.2	117.7	2.9	117.9	3.1	116.5	1.7
25	18.3	18.6	0.3	18.6	0.3	18.6	0.3	18.6	0.3

[a] $\Delta\delta = \delta$(synthetic isomer)–δ(ref. 1) [b] n.o. = not observed; color code: black: $\Delta\delta_C < 1.0$ ppm; red: $\Delta\delta_C \geq 1.0$ ppm.

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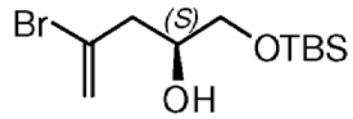
1 T. Kubota, M. Tsuda, J. Kobayashi, *Tetrahedron Lett.* **2000**, *41*, 713-716.

Table S2. Comparison of the ^{1}H NMR Data of Compound **1** with those of Amphidinolide V Reported in the Literature;[1] Arbitrary Numbering Scheme as Shown in the Previous Table.

Nr	C_6D_6, δ (*J*)		$CDCl_3$, δ (*J*)	
	Lit.	**1**	**Lit.**	**1**
2	2.17 (m)	2.08-2.18 (m)	2.45 (m)	2.43-2.47 (m)
	2.05 (ddd, *14.4, 4.7, 3.8*)	2.01 (ddd, *14.4, 5.6, 4.0*)		
3	2.57 (ddd, *14.1, 12.1, 3.8*)	2.52 (ddd, *14.1, 12.1, 4.0*)	2.73 (m)	2.70-2.77 (m)
			2.63 (m)	2.50-2.67 (m)
	2.38 (dt, *14.1, 4.7*)	2.34 (dt, *14.1, 4.2*)		
6	3.05 (d, *16.3*)	3.01 (d, *16.1*)	3.25 (d, *16.4*)	3.25 (d, *16.4*)
	2.88 (d, *16.3*)	2.86 (d, *16.1*)	3.11 (d, *16.4*)	3.11 (d, *16.4*)
8	3.97 (m)	3.93 (brs)	**4.50 (m)**	**4.00 (dd, *5.8, 5.4*)**
9	2.82 (dd, *6.5, 2.2*)	2.77 (dd, *6.5, 2.1*)	2.80 (m)	2.79-2.82 (m)
10	3.55 (d, *2.2*)	3.50 (brs)	3.46 (brs)	3.46 (brs)
12	2.29 (dd, *14.5, 3.3*)	2.24 (dd, *14.1, 2.6*)	2.41 (m)	2.39-2.41 (m)
	2.18 (m)	2.08-2.18 (m)		
13	5.62 (dt, *7.1, 3.3*)	5.53-5.59 (m)	5.43 (m)	5.43-5.45 (m)
14	5.47 (dd, *15.5, 7.1*)	5.42 (dd, *15.4, 6.6*)	5.42 (m)	5.40-5.43 (m)
15	5.74 (dt, *15.5, 6.6*)	5.69 (dt, *15.4, 6.6*)	5.72 (m)	5.67-5.77 (m)
16	2.70 (t, *6.6*)	2.64 (t, *6.5*)	2.83 (m)	2.82-2.85 (m)
17	5.57 (dt, *15.6, 6.6*)	5.52 (dd, *15.6, 6.6*)	5.60 (dt, *15.6, 6.7*)	5.60 (dt, *15.6, 6.7*)
18	6.21 (d, *15.6*)	6.15 (d, *15.6*)	6.12 (d, *15.6*)	6.12 (d, *15.6*)
20	5.01 (s)	4.95 (s)	4.89 (s)	4.89 (s)
	4.94 (s)	4.89 (s)		
21	5.03 (s)	4.98 (s)	5.13 (s)	5.13 (s)
	4.97 (s)	4.92 (s)	5.08 (s)	5.08 (s)
22	4.97 (s)	4.92 (s)	5.26 (s)	5.24 (s)
	4.92 (s)	4.89 (s)	5.10 (s)	5.10 (s)
23	5.68 (s)	5.62 (s)	5.46 (s)	5.46 (s)
	5.34 (s)	5.28 (s)	5.14 (s)	5.16 (s)
24	5.10 (s)	5.04 (s)	5.19 (s)	5.19 (s)
	4.76 (s)	4.71 (s)	4.93 (s)	4.93 (s)
25	1.78 (s)	1.73 (s)	1.83 (s)	1.83 (s)

FLS-FA-118-01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ppm (t1)

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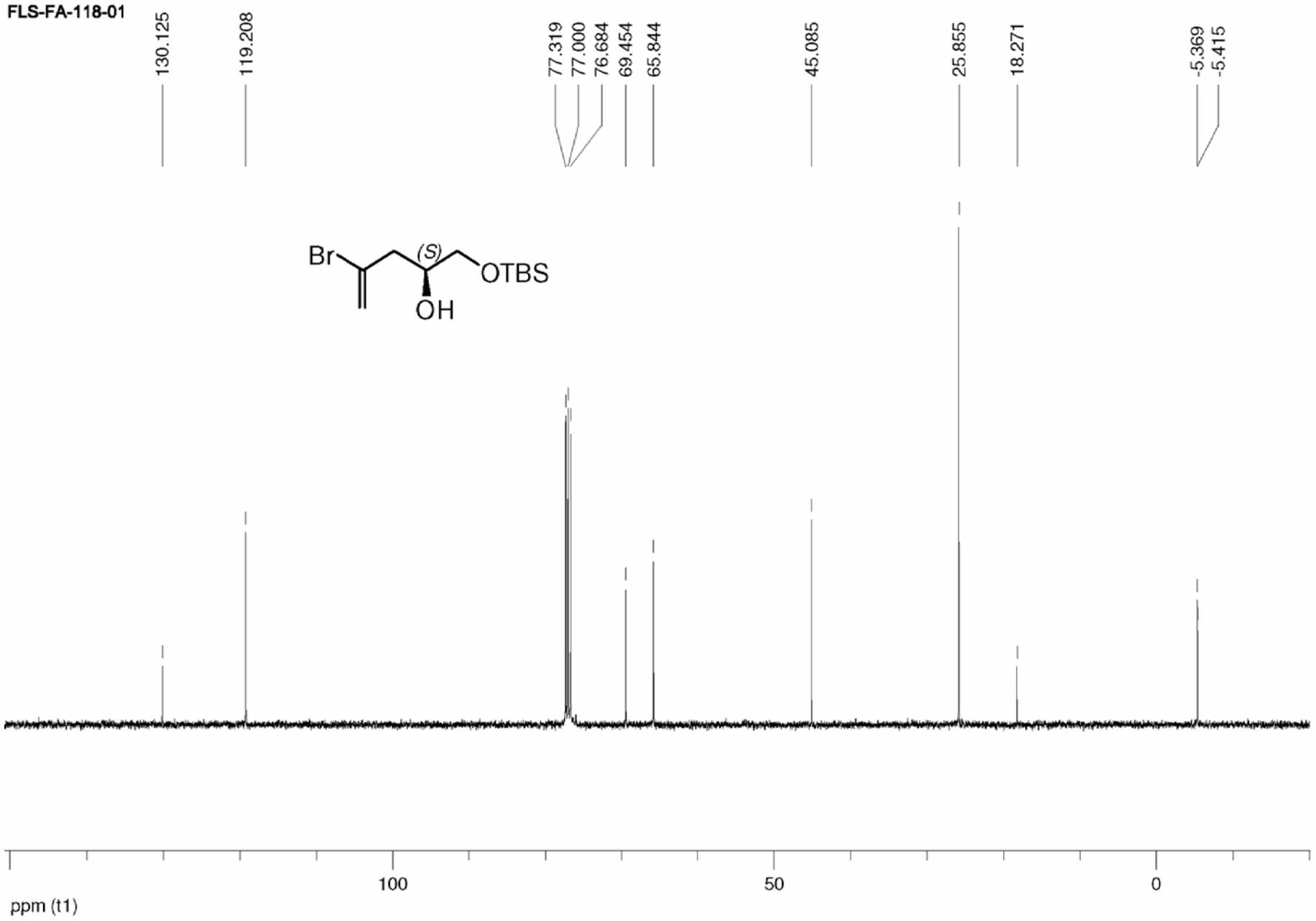
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OH
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50
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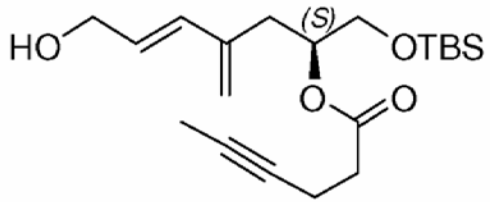

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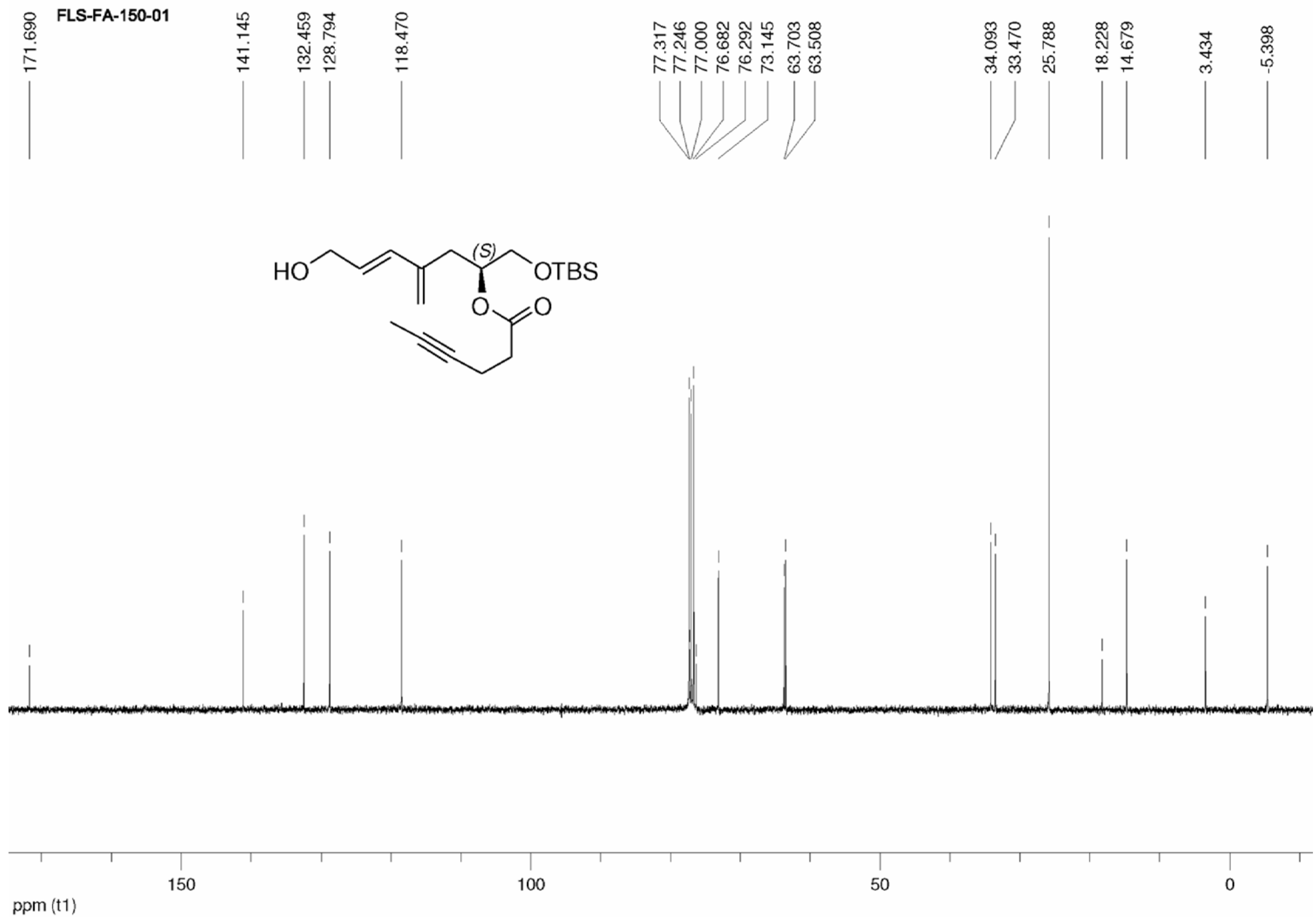
ppm (t1)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63.508
34.093
33.470
25.788
18.228
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ppm (t1)

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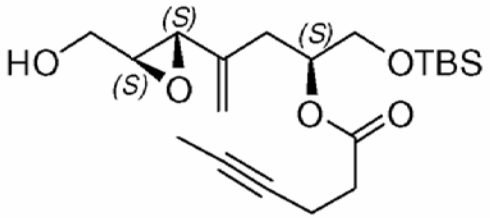
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OTBS

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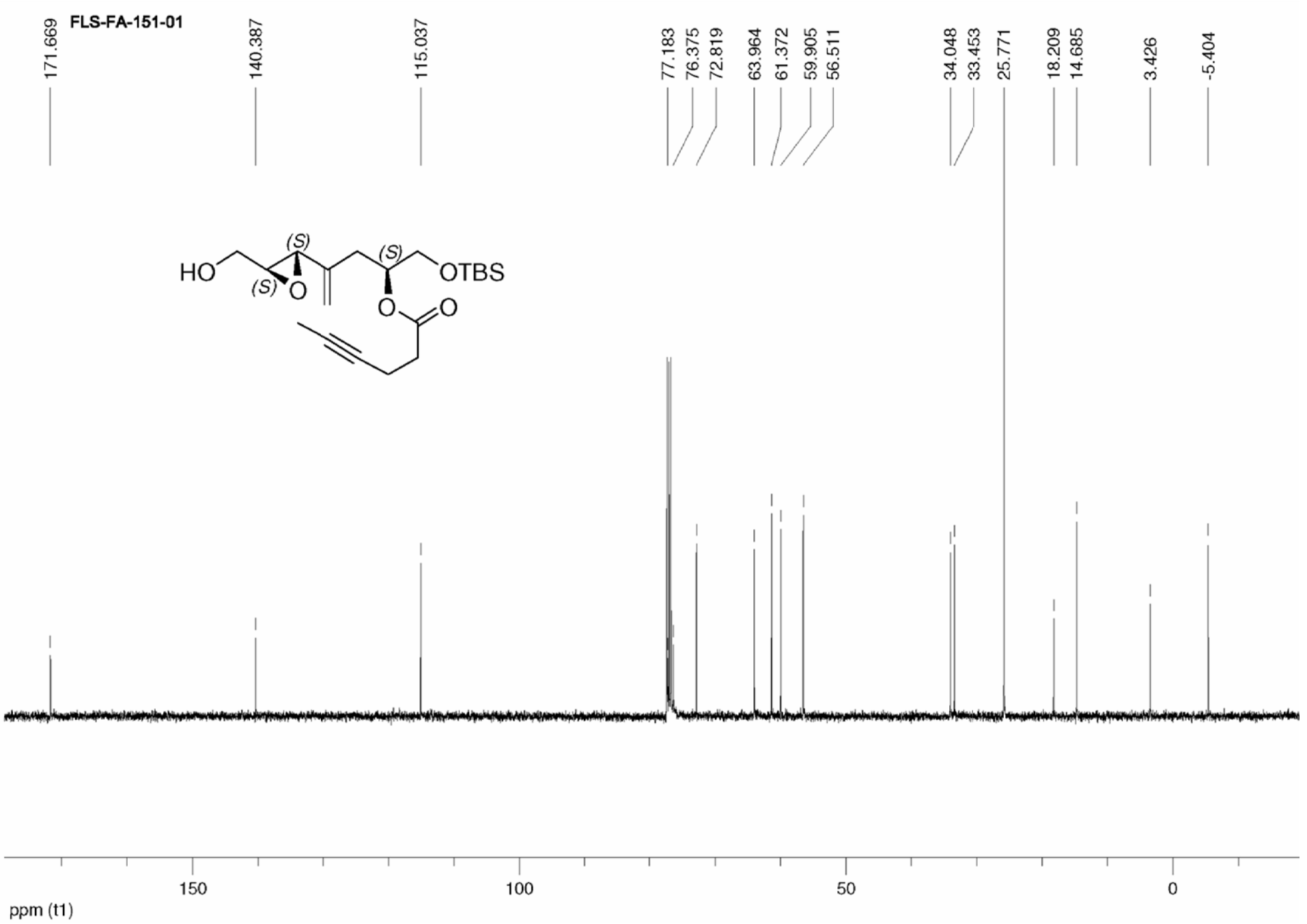
ppm (t1)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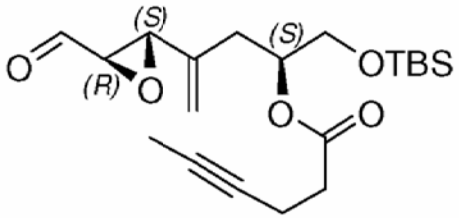
ppm (t1)

FLS-FA-153-01



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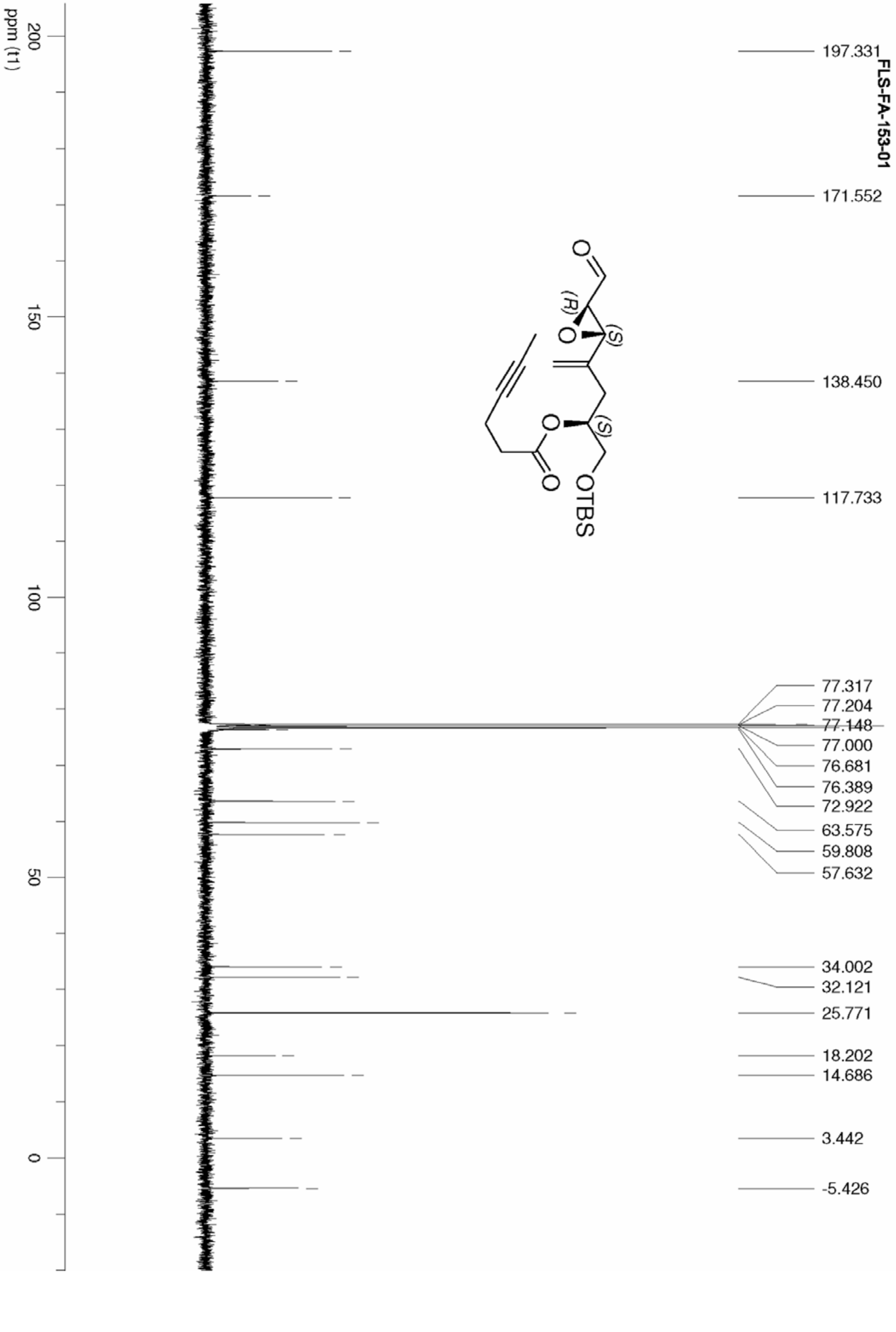
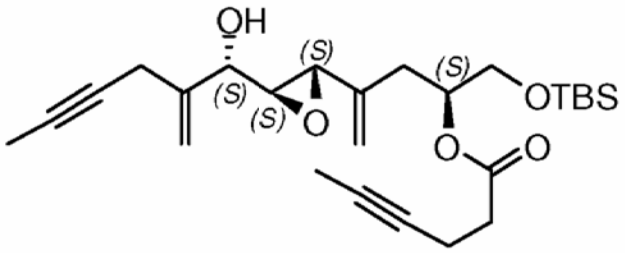
0.0

ppm (t1)



FLS-FA-153-01
197.331
171.552
138.450
117.733
77.317
77.204
77.148
77.000
76.681
76.389
72.922
63.575
59.808
57.632
34.002
32.121
25.771
18.202
14.686
3.442
-5.426
(R)
(S)
(S)
OTBS
ppm (t1)
200
150
100
50
0

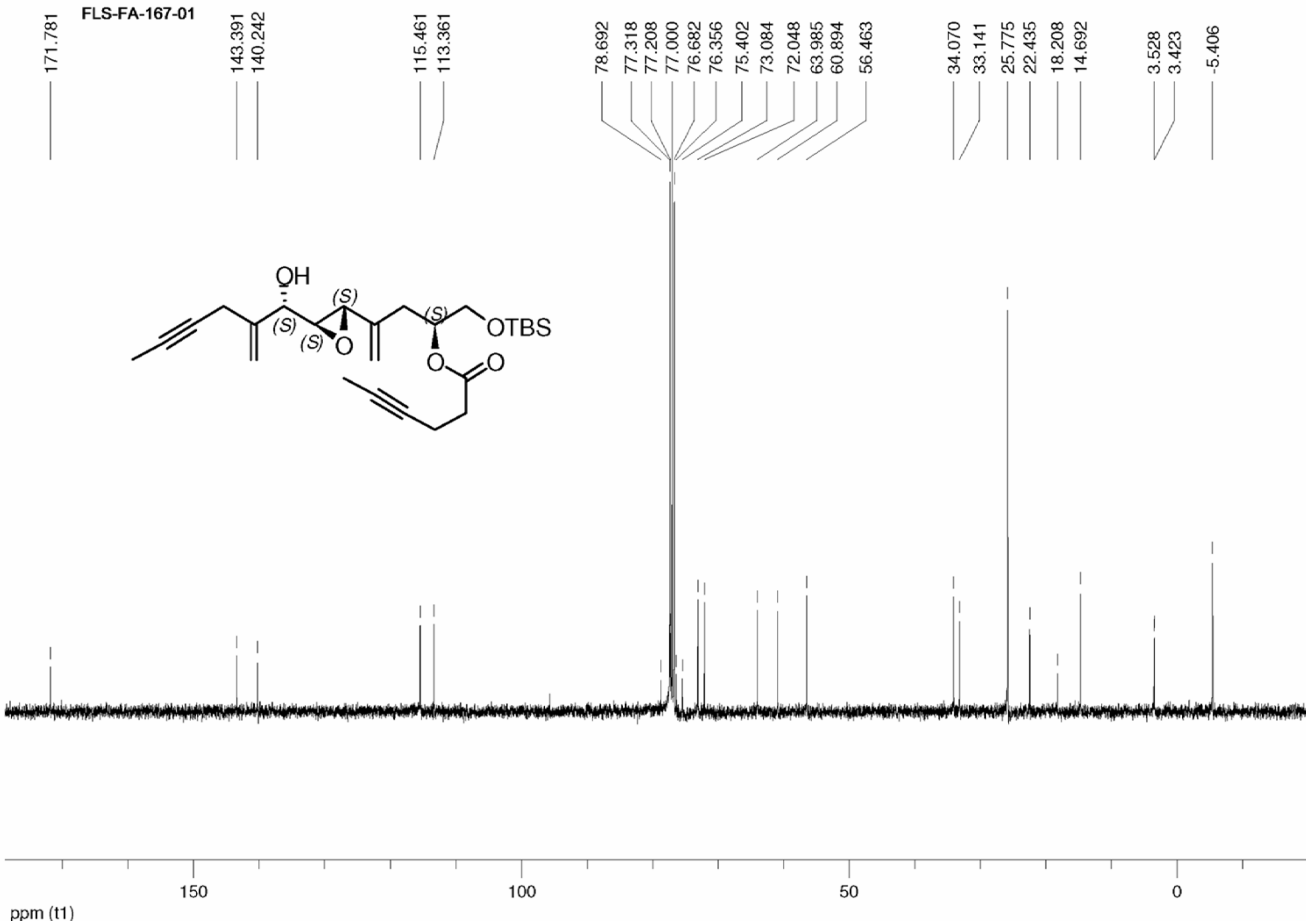
FLS-FA-167-01



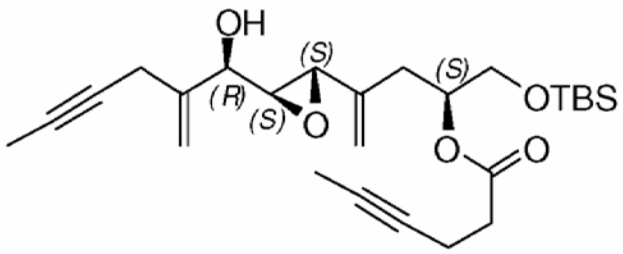
1.00
1.12
1.13
2.53
1.08
3.59
6.50
1.25
1.34
3.94
4.92
9.99
6.61

5.0
4.0
3.0
2.0
1.0
0.0

ppm (t1)

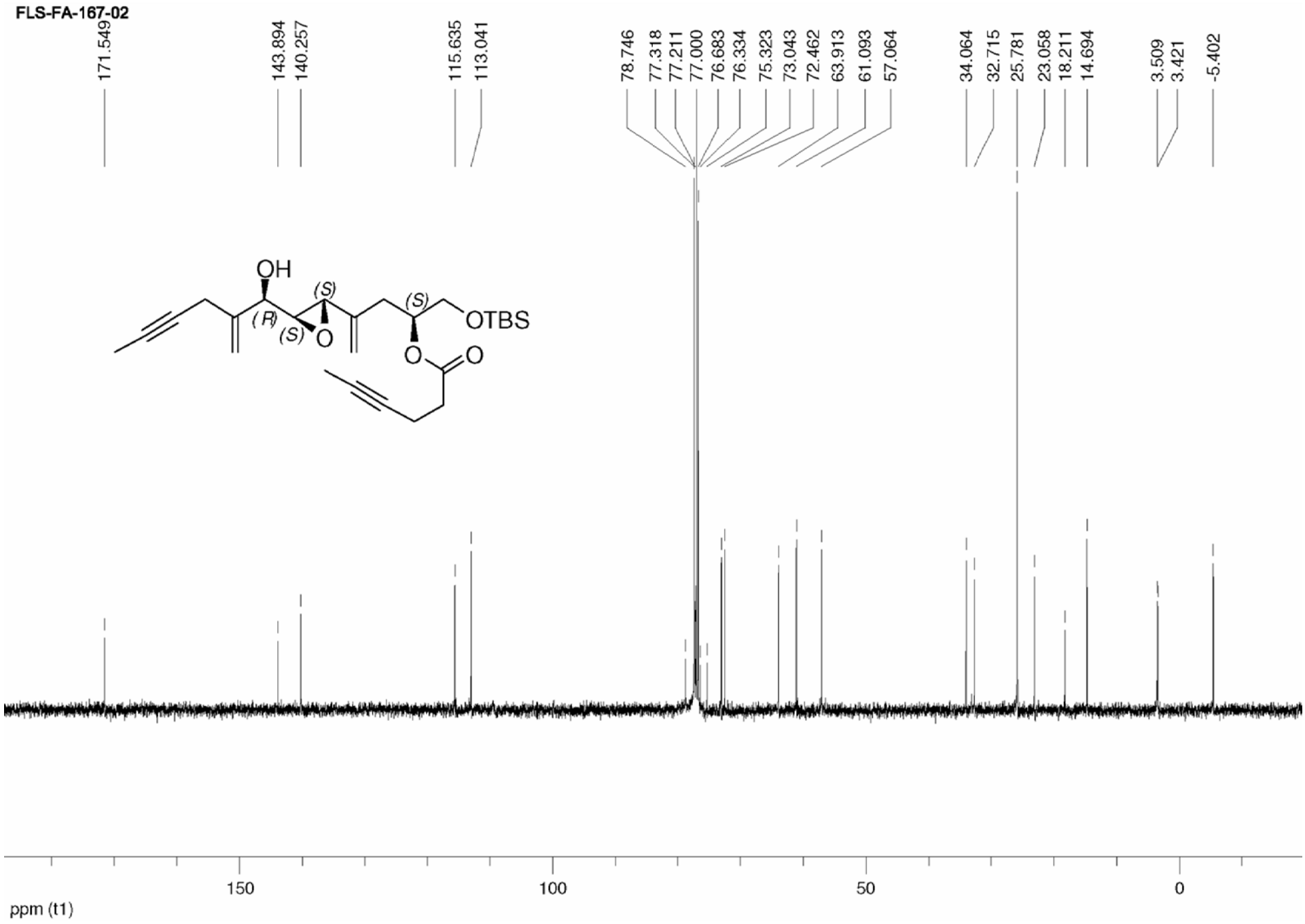

FLS-FA-167-01
171.781
143.391
140.242
115.461
113.361
78.692
77.318
77.208
77.000
76.682
76.356
75.402
73.084
72.048
63.985
60.894
56.463
34.070
33.141
25.775
22.435
18.208
14.692
3.528
3.423
-5.406
OH
(S)
(S)
(S)
(S)
O
OTBS
O
O
150
100
50
0
ppm (t1)

FLS-FA-167-02

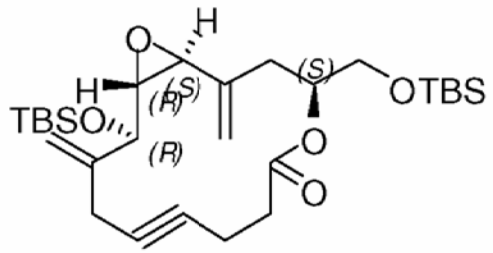

OH
(S)
(S)
(R)
(S)
O
OTBS
O
O

0.92
1.94
1.98
0.84
1.97
0.90
2.39
4.29
2.03
0.82
2.96
3.12
9.27
6.18
5.0
4.0
3.0
2.0
1.0
0.0
ppm (t1)

FLS-FA-167-02


171.549
143.894
140.257
115.635
113.041
78.746
77.318
77.211
77.000
76.683
76.334
75.323
73.043
72.462
63.913
61.093
57.064
34.064
32.715
25.781
23.058
18.211
14.694
3.509
3.421
-5.402
OH
(S)
(S)
(R)
(S)
O
OTBS
O
O
150
100
50
0
ppm (t1)

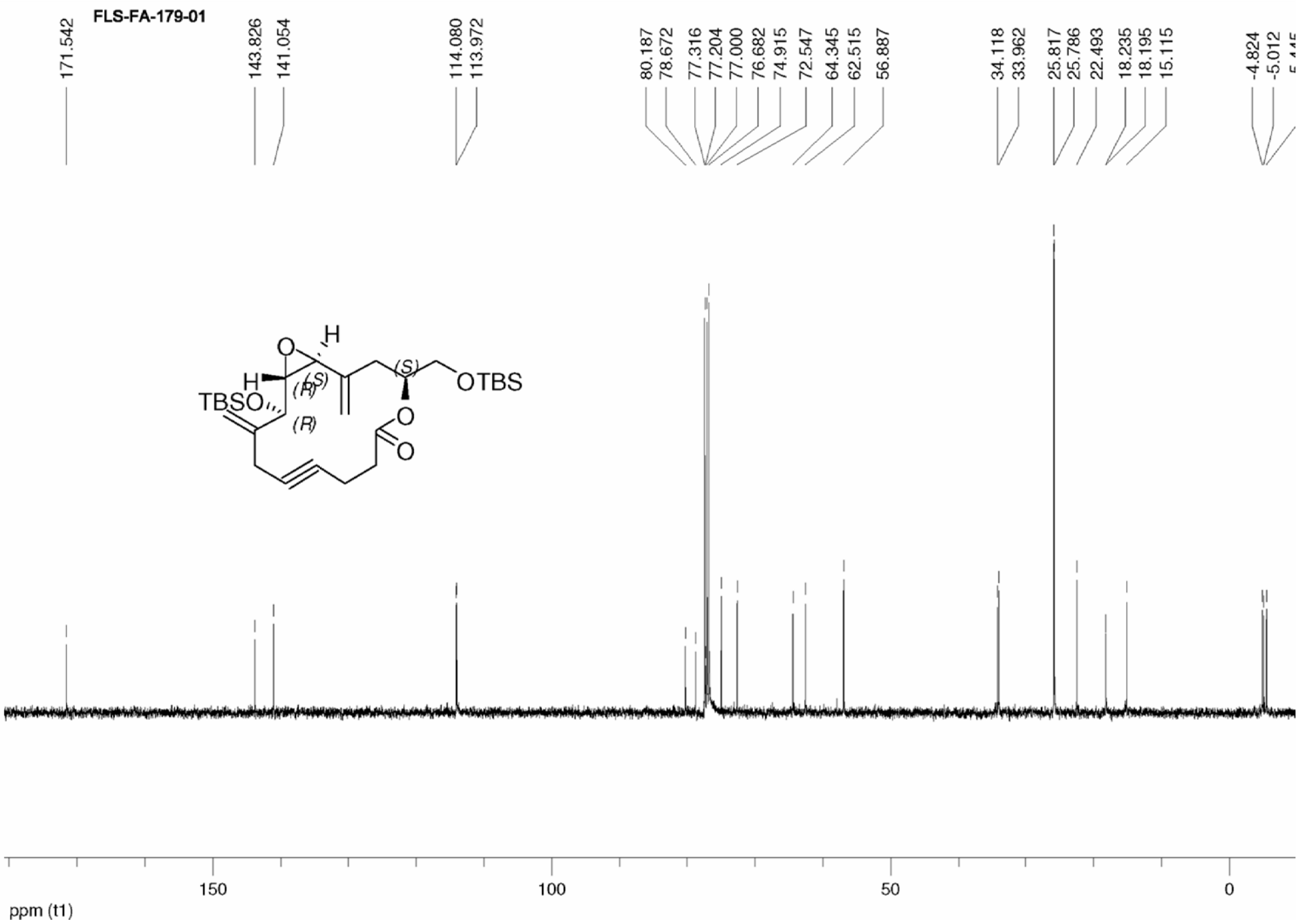
FLS-FA-179-01



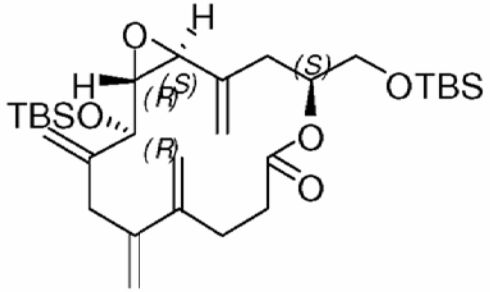
1.12
1.00
1.02
1.02
0.90
1.09
1.17
6.46
18.16
12.61

5.0
4.0
3.0
2.0
1.0
0.0

ppm (t1)


FLS-FA-179-01
171.542
143.826
141.054
114.080
113.972
80.187
78.672
77.316
77.204
77.000
76.682
74.915
72.547
64.345
62.515
56.887
34.118
33.962
25.817
25.786
22.493
18.235
18.195
15.115
-4.824
-5.012
TBSO
OTBS
(S)
(R)
(S)
(R)
150
100
50
0
ppm (t1)

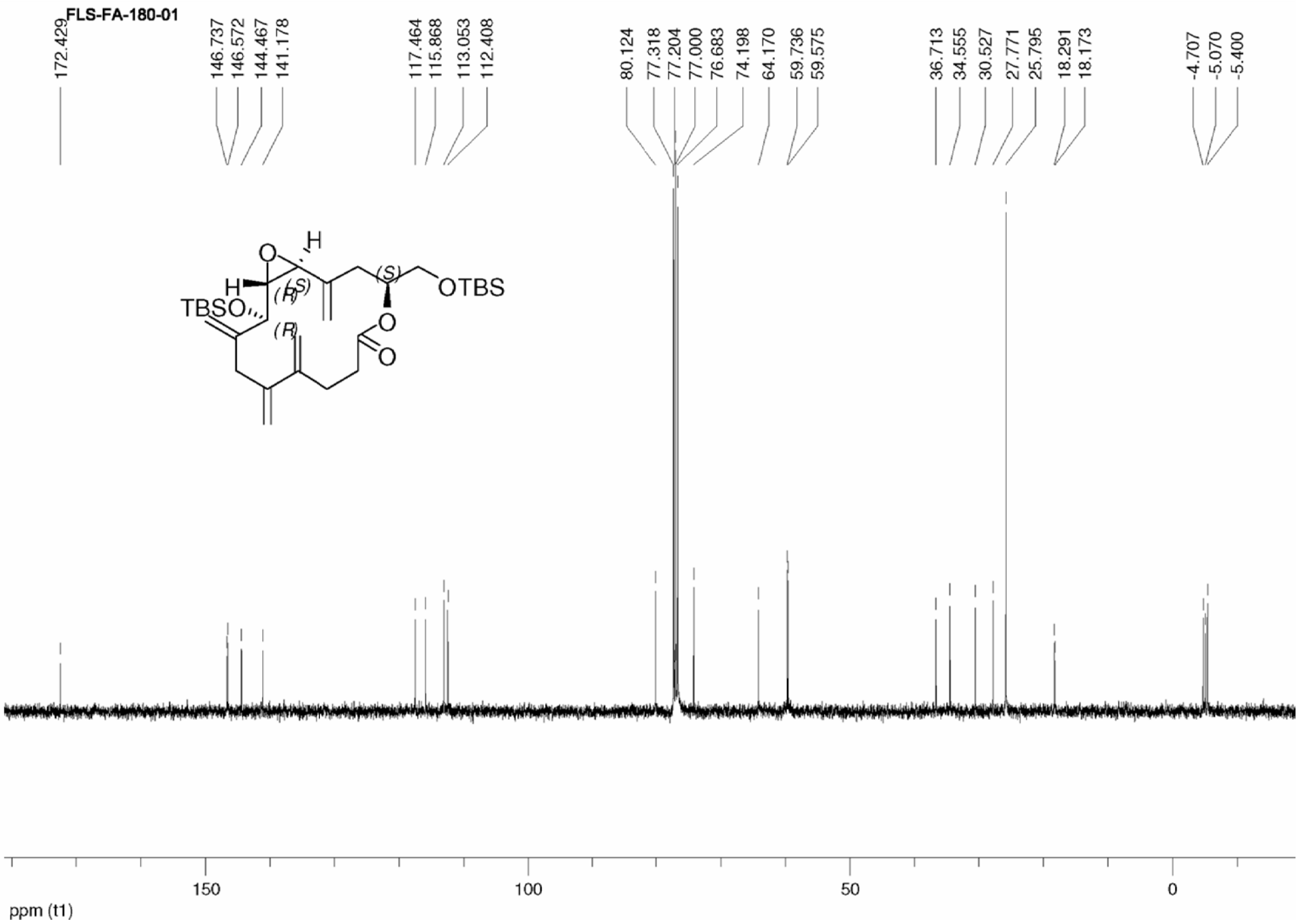
FLS-FA-180-01



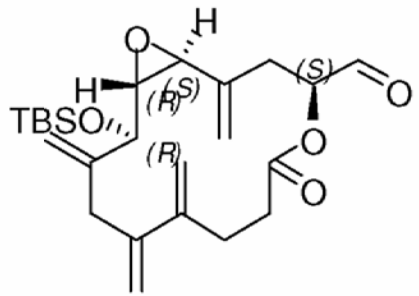
2.01 1.08 1.10 2.19 1.15 0.99 0.99 1.04 2.18 1.00 0.99 1.95 4.19 1.11 9.87 9.83 3.16 6.28

5.0 4.0 3.0 2.0 1.0 0.0

ppm (t1)

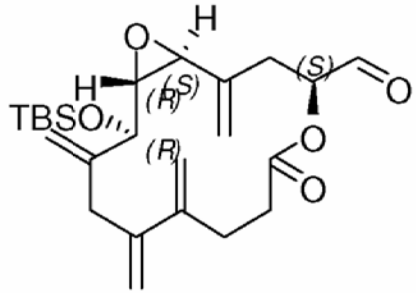

FLS-FA-180-01
172.429
146.737
146.572
144.467
141.178
117.464
115.868
113.053
112.408
80.124
77.318
77.204
77.000
76.683
74.198
64.170
59.736
59.575
36.713
34.555
30.527
27.771
25.795
18.291
18.173
-4.707
-5.070
-5.400
TBSO
H
O
H
(S)
(R)
(S)
(R)
O
O
OTBS
150
100
50
0
ppm (t1)

FLS-FA-192-01



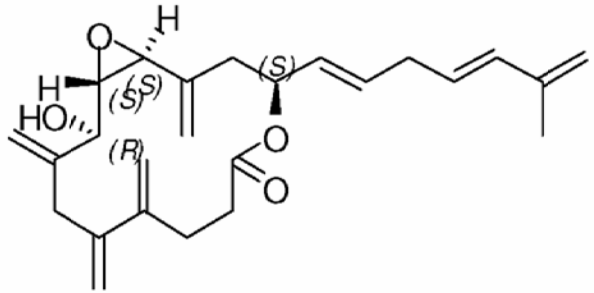
FLS-FA-192-01

197.296
172.350
146.244
146.149
144.080
139.896
117.836
115.836
113.684
112.977
79.197
78.493
77.318
77.207
77.000
76.868
76.684
76.446
76.372
60.288
58.939
36.968
33.695
30.393
27.351
25.818
18.308
-4.689
-5.065

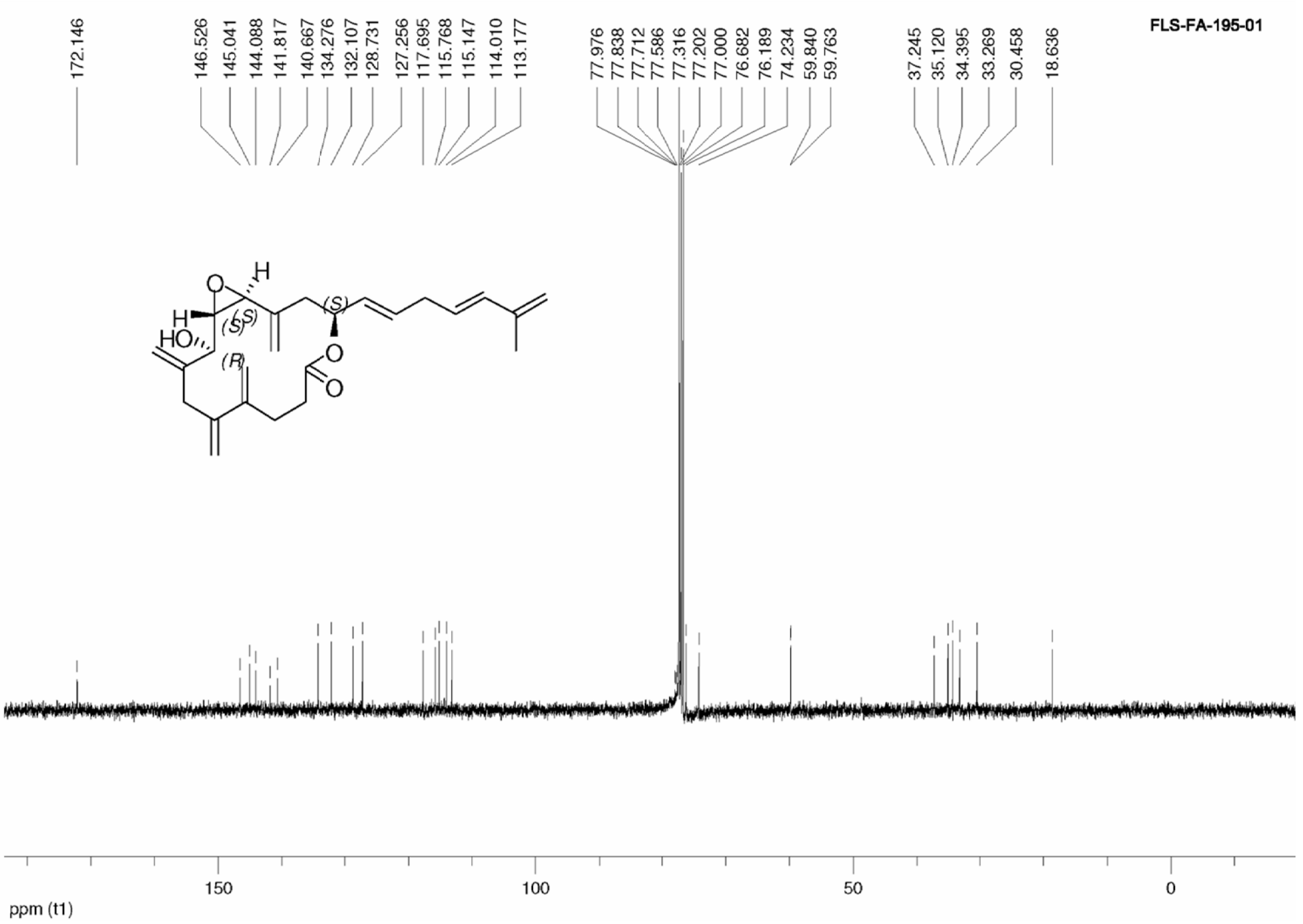


200
150
100
50
0
ppm (t1)

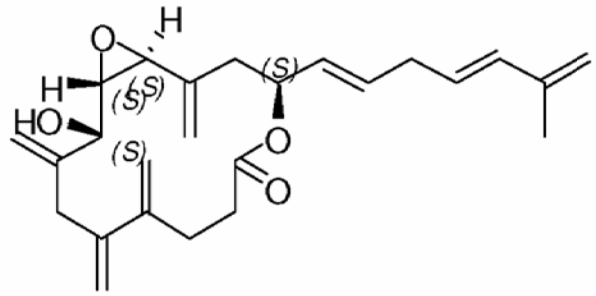
FLS-FA-195-01


O
H
H
HO
(S)
(S)
(S)
(R)
O
O

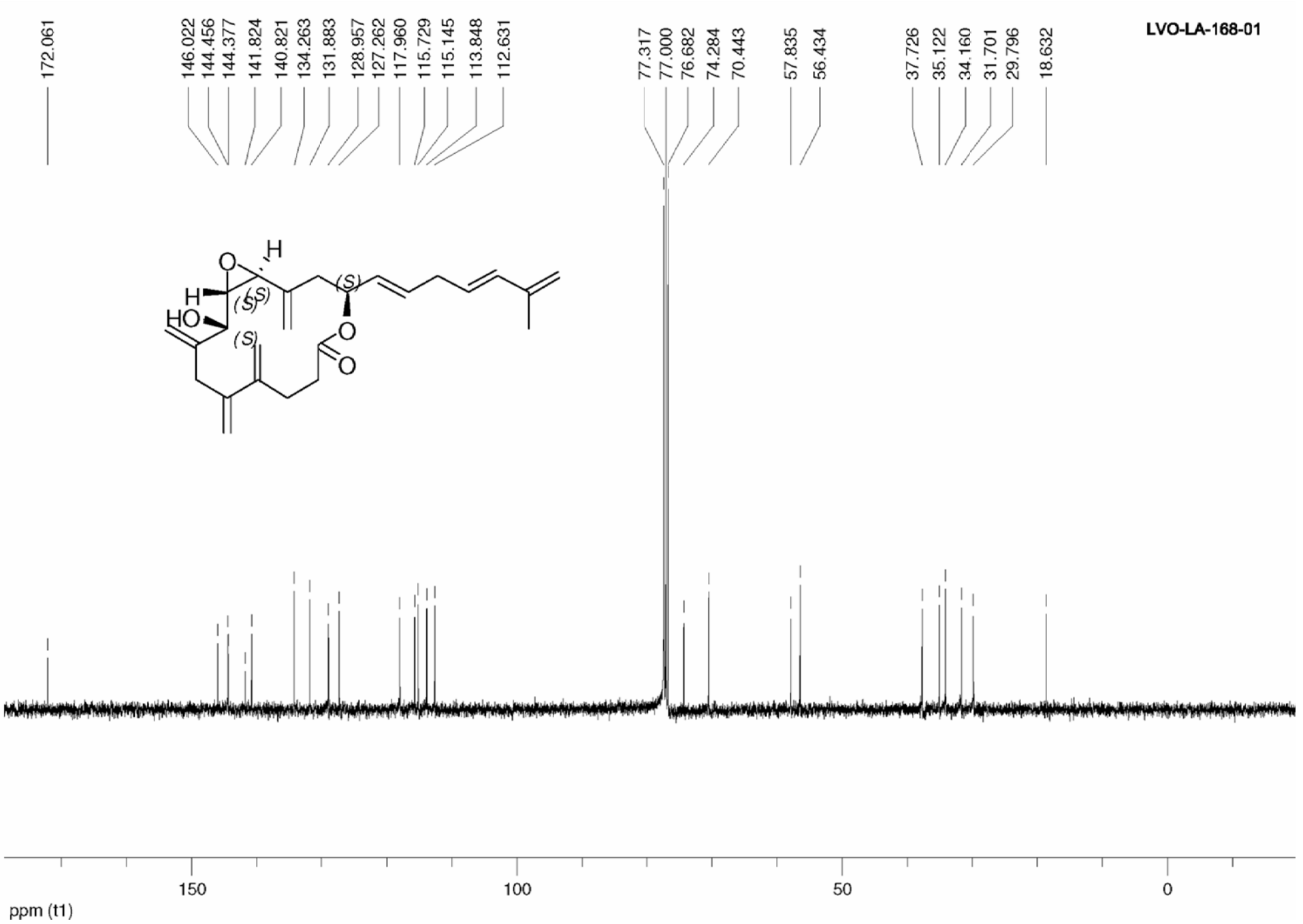
1.00
1.03
1.07
1.99
2.95
0.91
0.97
0.89
1.92
1.95
4.09
2.20
1.04
3.04
6.0
5.0
4.0
3.0
2.0
1.0
0.0
ppm (t1)


FLS-FA-195-01
172.146
146.526
145.041
144.088
141.817
140.667
134.276
132.107
128.731
127.256
117.695
115.768
115.147
114.010
113.177
77.976
77.838
77.712
77.586
77.316
77.202
77.000
76.682
76.189
74.234
59.840
59.763
37.245
35.120
34.395
33.269
30.458
18.636
O
H
HO
(S)
(S)
(S)
(R)
O
O
150
100
50
0
ppm (t1)

LVO-LA-168-01

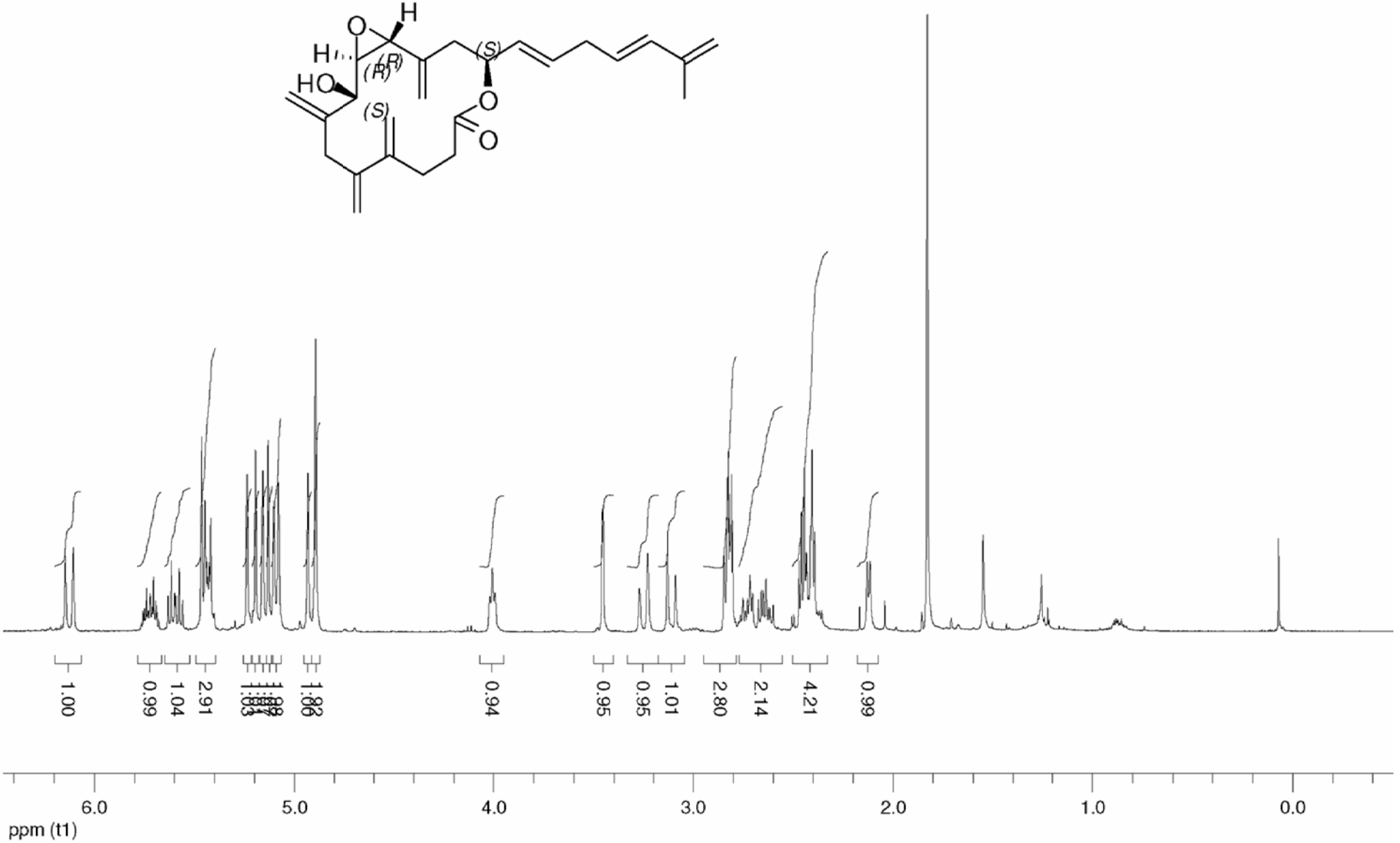

O
H
H
HO
(S)
(S)
(S)
(S)
O
O

0.98
0.99
1.10
2.90
2.11
1.97
0.99
1.00
1.02
0.96
2.00
1.89
4.22
2.01
1.27
3.22
6.0
5.0
4.0
3.0
2.0
1.0
0.0
ppm (t1)


LVO-LA-168-01
172.061
146.022
144.456
144.377
141.824
140.821
134.263
131.883
128.957
127.262
117.960
115.729
115.145
113.848
112.631
77.317
77.000
76.682
74.284
70.443
57.835
56.434
37.726
35.122
34.160
31.701
29.796
18.632
O
H
H
HO
(S)
(S)
(S)
(S)
O
O
150
100
50
0
ppm (t1)

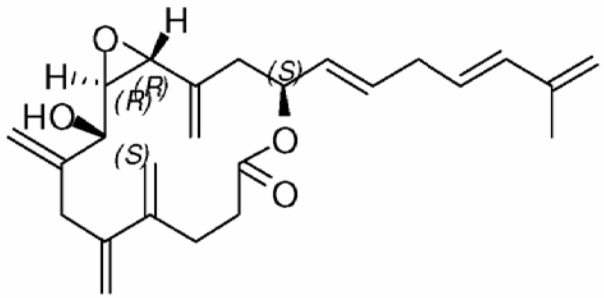
LVO-LA-182-01

in CDCl3



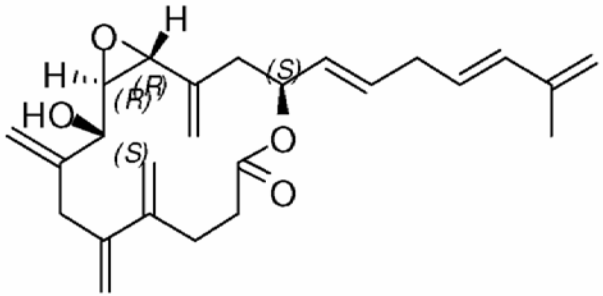
LVO-LA-182-01

in C6D6



LVO-LA-182-01
in CDCl3

171.869
144.959
144.627
142.112
141.853
140.748
134.130
132.021
128.057
127.447
115.147
115.089
114.868
114.814
114.036
77.318
77.204
77.000
76.684
74.326
71.436
63.260
57.966
39.169
39.089
35.091
33.706
30.518
18.629



150
100
50
ppm (t1)