NIS-Mediated Electrophilic Cyclization of 3-Silyloxy-1,n-enynes

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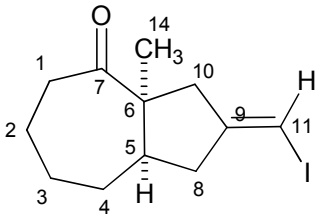
2D-NMR for selected examples

***rel*-(3a*R*, 8a*S*, *E*)-2-(Iodomethylene)-3a-methyloctahydroazulen-4(2*H*)-one (6g).**

Confirmation of the azulentype structure and the *syn* Konfiguration:

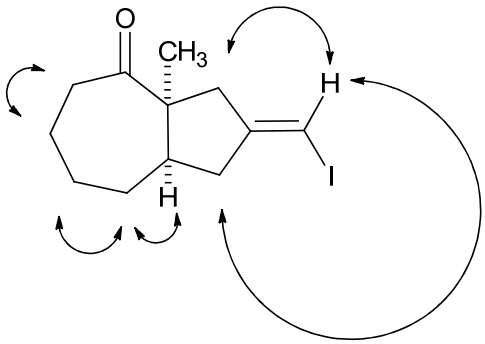
HSQC (Nr. → ^{13}C → ^{1}H, ppm)



1:	40.7	2.70-2.78/2.32-2.35,
2/3:	27.9	1.86-1.94/1.37-1.51,
2/3:	30.4	1.86-1.94/1.37-1.51,
4:	35.7	1.58-1.62/1.21-1.30,

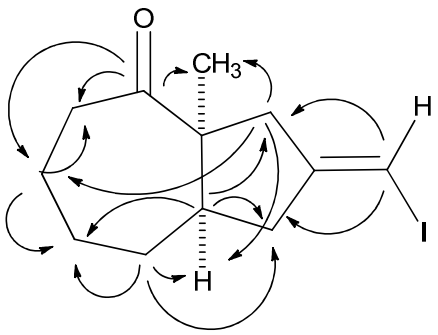
5:	46.2	2.01-2.05,
6:	60.3	-,
7:	216.4	-,
8:	44.7	2.70-2.78/2.07-2.11,
9:	152.6	-,
10:	41.2	2.70-2.78/2.26-2.29,
11:	70.4	5.93,
14:	24.9	1.16,

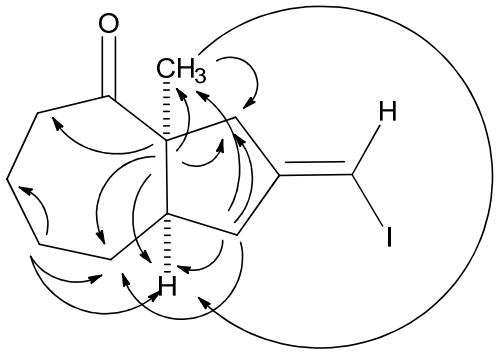
COSY (^{1}H ↔ ^{1}H, ppm)



1.16:	- ,
1.21-1.30:	1.58.1.62, 2.01-2.05,
1.37-1.51:	1.86-1.94, 2.70-2.78,
1.58-1.62:	1.21-1.30, 1.86-1.94,
1.86-1.94:	1.37-1.51, 2.32-2.35,
2.01-2.05:	1.21-1.30,
2.07-2.11:	2.70-2.78, 5.93,
2.26-2.29:	2.70-2.78,
2.32-2.35:	2.70-2.78,
2.70-2.78:	1.37-1.51, 2.07-2.11, 2.26-2.29, 5.93,
5.93:	2.07-2.11, 2.70-2.78.

HMBC (^{13}C → ^{1}H, [ppm])





24.9: 2.01-2.05, 2.26-2.29, 2.70-2.78.

27.9: 1.21-1.30, 1.37-1.51, 1.58-1.62, 1.86-1.94, 2.01-2.05, 2.32-2.35, 2.70-2.78.

30.4: 1.21-1.30, 1.37-1.51, 1.58-1.62, 1.86-1.94, 2.01-2.05, 2.32-2.35, 2.70-2.78,

35.7: 1.37-1.51, 1.86-1.94, 2.01-2.05, 2.07-2.11,

40.7: 1.37-1.51, 1.86-1.94,

41.2: 1.16, 2.01-2.05, 2.32-2.35,

44.7: 1.58-1.62, 2.01-2.05, 2.26-2.29,

46.2: 1.16, 1.21-1.30, 1.37-1.51, 1.58-1.62, 1.86-1.94, 2.07-2.11, 2.26-2.29, 2.70-2.78,

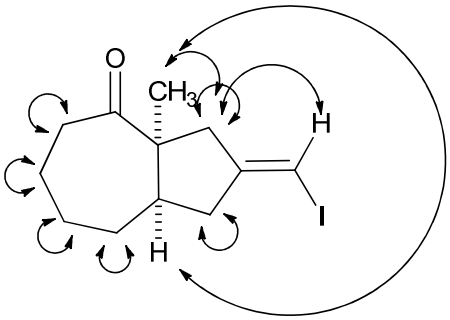
60.3: 1.16, 1.21-1.30, 2.01-2.05, 2.26-2.29, 2.32-2.35, 2.70-2.78,

70.4: 2.07-2.11, 2.26-2.29,

152.6: 2.07-2.11, 2.26-2.29, 2.70-2.78,

216.4: 1.16, 1.37-1.51, 1.86-1.94, 2.32-2.35, 2.70-2.78.

NOESY (^{1}H ↔ ^{1}H, ppm)



1.16: 2.01-2.05, 2.26-2.29,

1.21-1.30: 1.58-1.62,

1.37-1.51: 1.86-1.94,

1.58-1.62: 1.21-1.30,

1.86-1.94: 1.37-1.51,

2.01-2.05: 1.16,

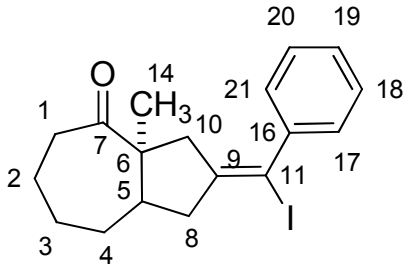
2.07-2.11: 2.70-2.78,

2.26-2.29:	1.16, 2.70-2.78, 5.93,
2.32-2.35:	2.70-2.78,
2.70-2.78:	2.07-2.11, 2.26-2.29, 2.32-2.35, 5.93,
5.93:	2.26-2.29, 2.70-2.78.

***rel*-(3a*R*, 8a*S*, *E*)-2-(Iodo(phenyl)methylene)-3a-methyloctahydroazulen-4(2*H*)-one (6a)**

Confirmation of the *E*-Konfiguration (Iodine to carbonyl-function):

HSQC (Nr. → ^{13}C → ^{1}H, ppm)



1:	40.7	2.23-2.33/2.68-2.76,
2/3:	27.9	1.31-1.53/1.90-1.93,
2/3:	30.4	1.31-1.53/1.90-1.93,
4:	35.8	1.31-1.53/1.66-1.70,
5:	46.0	2.02-2.06,
6:	60.7	-,
7:	216.6	-,
8:	49.2	2.23-2.33/2.99-3.05,
9:	148.3	-,
10:	39.3	2.23-2.33/2.68-2.76,
11:	91.8	-,
14:	24.8	1.13,
16:	143.6	-,
17/18/20/21:	128.3	7.26-7.29,
17/18/20/21:	129.0	7.26-7.29,
19:	127.7	7.19-7.22.

COSY (^{1}H ↔ ^{1}H, ppm)

1.13: -

1.31-1.53: 1.66-1.70, 1.90-1.93, 2.02-2.06, 2.68-2.76,

1.66-1.70: 1.31-1.53,

1.90-1.93: 1.31-1.53, 2.23-2.33,

2.02-2.06: 1.31-1.53, 2.99-3.05,

2.23-2.33: 1.90-1.93, 2.68-2.76, 2.99-3.05,

2.68-2.76: 1.31-1.53, 2.23-2.33,

2.99-3.05: 2.02-2.06, 2.23-2.33,

7.19-7.22: -

7.26-7.29: -

HMBC ($^{13}C \rightarrow {}^{1}H$, [ppm])

24.8: 2.02-2.06, 2.23-2.33, 2.68-2.76,

27.9: 1.31-1.53, 1.66-1.70, 1.90-1.93, 2.23-2.33, 2.68-2.76,

30.4: 1.31-1.53, 1.66-1.70, 1.90-1.93, 2.23-2.33, 2.68-2.76,

35.8: 1.31-1.53, 2.23-2.33,

39.3: 1.13, 2.02-2.06, 2.23-2.33, 2.99-3.05,

40.7: 1.31-1.53, 1.90-1.93,

46.0: 1.13, 1.31-1.53, 1.66-1.70, 1.90-1.93, 2.23-2.33, 2.99-3.05,

49.2: 1.66-1.70, 2.02-2.06, 2.23-2.33,

60.7: 1.13, 1.31-1.53, 1.66-1.70, 2.02-2.06, 2.23-2.33, 2.68-2.76,

91.8: 2.23-2.33, 2.68-2.76, 2.99-3.05,

127.7: 7.26-7.29,

128.3: 2.23-2.33, 2.68-2.76, 2.99-3.05,

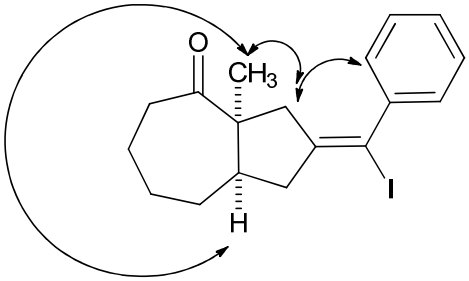
129.0: 2.23-2.33, 2.68-2.76, 2.99-3.05,

143.6: 2.23-2.33, 2.68-2.76, 2.99-3.05,

148.3: 2.02-2.06, 2.23-2.33, 2.68-2.76, 2.99-3.05,

216.6: 1.13, 1.90-1.93, 2.23-2.33, 2.68-2.76.

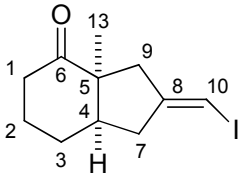
NOESY (^{1}H ↔ ^{1}H, ppm)



1.13:	2.02-2.06, 2.68-2.76, 2.99-3.05,
1.31-1.53:	1.66-1.70, 1.90-1.93,
1.66-1.70:	1.31-1.53,
1.90-1.93:	1.31-1.53,
2.02-2.06:	-
2.23-2.33:	2.99-3.05, 7.26-7.29,
2.68-2.76:	2.23-2.33, 7.26-7.29,
2.99-3.05:	-
7.19-7.22:	-
7.26-7.29:	2.23-2.33, 2.68-2.76.

***rel*-(3a*S*, 7a*R*, *E*)-2-(Iodomethylene)-3a-methylhexahydro-1H-inden-4(2*H*)-one (6j)**

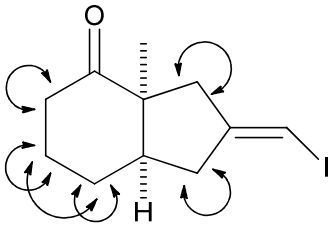
HSQC (Nr. → ^{13}C → ^{1}H, ppm)



1:	38.1	2.42-2.50 / 2.32-2.36,
2:	23.6	1.91-1.97 / 1.80-1.87,
3:	25.9	1.91-1.97 / 1.60-1.67,
4:	48.0	2.26-2.31,
5:	56.5	-,
6:	214.2	-.
7:	41.4	2.42-2.50 / 2.08-2.14,
8:	152.0	-,
9:	43.6	3.09 / 2.00-2.03,
10:	71.0	5.96-5.97,

13: 22.9 1.20.

COSY (^{1}H ↔ ^{1}H, ppm)



1.20: - ,

1.60-1.67: 1.91-1.97,

1.80-1.87: 1.91-1.97,

1.91-1.97: 1.60-1.67, 1.80-1.87,

2.00-2.03: 3.09,

2.08-2.14: 2.42-2.50,

2.26-2.31: - ,

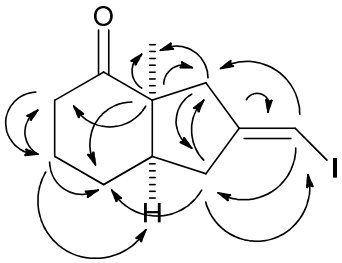
2.32-2.36: 2.42-2.50,

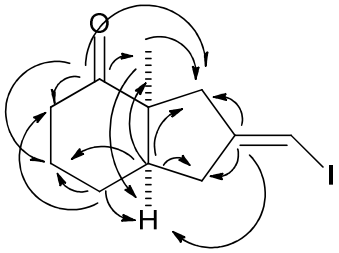
2.42-2.50: 2.08-2.14, 2.32-2.36,

3.09: 2.00-2.03,

5.96-5.97: - .

HMBC (^{13}C → ^{1}H, [ppm])





22.9: 2.00-2.03, 2.26-2.31, 2.42-2.50, 3.09,

23.6: 1.60-1.67, 1.91-1.97, 2.26-2.31, 2.32-2.36, 2.42-2.50,

25.9: 1.80-1.87, 1.91-1.97, 2.08-2.14, 2.26-2.31, 2.32-2.36, 2.42-2.50,

38.1: 1.60-1.67, 1.91-1.97,

41.4: 1.60-1.67, 2.00-2.03, 3.09, 5.96-5.97,

43.6: 1.20, 2.08-2.14, 5.96-5.97,

48.0: 1.20, 1.60-1.67, 1.80-1.87, 1.91-1.97, 2.08-2.14, 3.09,

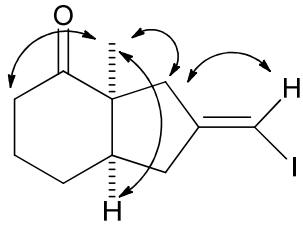
56.5: 1.20, 1.60-1.67, 1.91-1.97, 2.00-2.03, 2.26-2.31, 2.42-2.50, 3.09,

71.0: 2.00-2.03, 2.08-2.14, 2.42-2.50, 3.09,

152.0: 2.00-2.03, 2.08-2.14, 2.26-2.31, 2.42-2.50, 3.09,

214.2: 1.20, 1.80-1.87, 1.91-1.97, 2.00-2.03, 2.32-2.36, 2.42-2.50, 3.09.

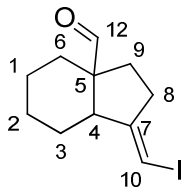
NOESY (^{1}H ↔ ^{1}H, ppm)



1.20:	2.00-2.03, 2.26-2.31, 2.32-2.36, 2.42-2.50, 3.09,
1.60-1.67:	- ,
1.80-1.87:	- ,
1.91-1.97:	- ,
2.00-2.03:	1.20,
2.08-2.14:	- ,
2.26-2.31:	1.20,
2.32-2.36:	1.20,
2.42-2.50:	1.20,
3.09:	1.20, 2.00-2.03,
5.96-5.97:	2.00-2.03, 3.09.

***rel*-(3a*S*, 7a*R*, *E*)-1-(Iodomethylene)octahydro-1*H*-indene-3a-carbaldehyde (6l)**

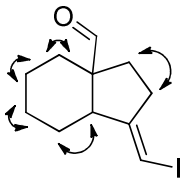
HSQC (Nr. → ^{13}C → ^{1}H, ppm)



1/2:	21.0	1.28-1.38,
1/2:	22.4	1.28-1.38/1.49-1.55,
3:	25.1	1.49-1.55/1.76-1.85,
4:	46.6	2.81,
5:	57.9	-,
6:	26.5	1.28-1.38/1.76-1.85,
7:	155.2	-,

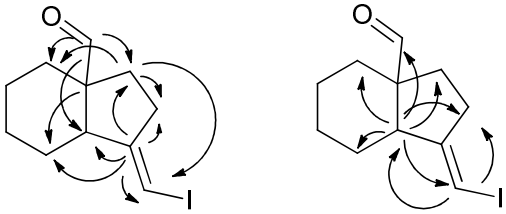
8:	34.9	2.39-2.43,
9:	29.8	1.59-1.64/1.76-1.85,
10:	71.0	5.95,
12:	204.6	out of meassured area (allocation by chemical shift)

COSY (^{1}H ↔ ^{1}H, ppm)



1.28-1.38:	1.49-1.55, 1.76-1.85,
1.49-1.55:	1.28-1.38, 1.76-1.85, 2.81,
1.59-1.64:	2.39-2.43,
1.76-1.85:	1.28-1.38, 1.49-1.55, 2.39-2.43, 2.81,
2.39-2.43:	1.59-1.64, 1.76-1.85,
2.81:	1.49-1.55, 1.76-1.85,
5.95:	- ,
9.49:	- .

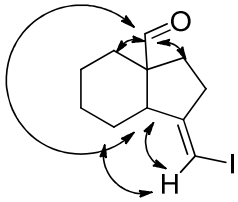
HMBC (^{13}C → ^{1}H, [ppm])



21.0:	1.28-1.38, 1.49-1.55, 1.76-1.85, 2.81,
22.4:	1.28-1.38, 1.49-1.55, 1.76-1.85,
25.1:	1.28-1.38, 1.49-1.55, 1.59-1.64, 2.81,
26.5:	1.28-1.38, 1.49-1.55, 1.76-1.85, 9.49,
29.8:	1.28-1.38, 1.76-1.85, 2.39-2.43, 9.49,
34.9:	1.28-1.38, 1.59-1.64, 1.76-1.85, 5.95,
46.6:	1.28-1.38, 1.49-1.55, 1.59-1.64, 1.76-1.85, 2.39-2.43, 5.95, 9.49,
57.9:	1.28-1.38, 1.49-1.55, 1.59-1.64, 1.76-1.85, 2.39-2.43, 2.81, 9.49,
71.0:	2.39-2.43, 2.81,
155.2:	1.49-1.55, 1.59-1.64, 1.76-1.85, 2.39-2.43, 2.81, 5.95,

204.6: 1.28-1.38, 1.59-1.64, 1.76-1.85, 2.81.

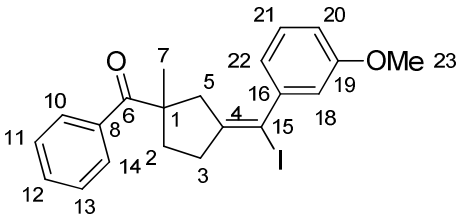
NOESY (^{1}H ↔ ^{1}H, ppm)



1.28-1.38:	- ,
1.49-1.55:	- ,
1.59-1.64:	- ,
1.76-1.85:	5.95, 9.49,
2.39-2.43:	- ,
2.81:	5.95, 9.49,
5.95:	1.76-1.85, 2.81,
9.49:	1.76-1.85, 2.81.

***rel*-(*E*)-(3-(Iodo(3-methoxyphenyl)methylene)-1-methylcyclopentyl)(phenyl)methanone (6m)**

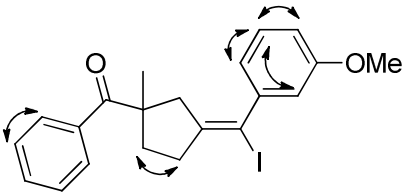
HSQC (Nr. → ^{13}C → ^{1}H, ppm)



1:	56.6	-,
2:	36.1	1.98-2.04/2.35-2.39,
3:	39.6	2.54-2.66,
4:	149.4	-,
5:	44.2	3.01/2.41-2.45,
6:	205.7	-,
7:	24.6	1.39,
8:	136.9	-,
10/14:	128.7	7.76-7.78,
11/13:	128.4	7.39-7.42,
12:	132.0	7.47-7.50,

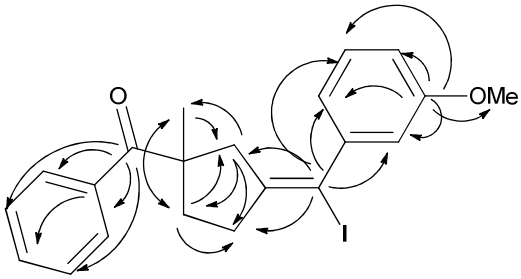
15:	91.2	-,
16:	145.1	-,
18:	121.2	6.85-6.87,
19:	159.4	-,
20:	113.5	6.76-6.78,
21:	129.3	7.20,
22:	114.5	6.83-6.84,
23:	55.4	3.80.

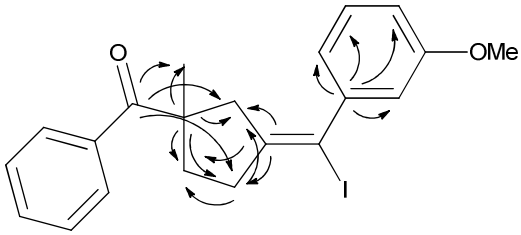
COSY (^{1}H ↔ ^{1}H, ppm)



1.39:	- ,
1.98-2.04:	2.35-2.39, 2.54-2.66,
2.35-2.39:	1.98-2.04, 2.54-2.66,
2.41-2.45:	3.01,
2.54-2.66:	1.98-2.04, 2.35-2.39,
3.01:	2.41-2.45,
3.80:	- ,
6.76-6.78:	7.20,
6.83-6.84:	7.20,
6.85-6.87:	7.20,
7.20:	6.76-6.78, 6.85-6.87, 6.83-6.84,
7.39-7.42:	7.76-7.78,
7.47-7.50:	- ,
7.76-7.78:	7.39-7.42.

HMBC (^{13}C → ^{1}H, [ppm])





24.6: 1.98-2.04, 2.35-2.39, 2.41-2.45, 3.01,

36.1: 1.39, 2.41-2.45, 2.54-2.66, 3.01,

39.6: 1.98-2.04, 2.35-2.39, 2.41-2.45, 3.01,

44.2: 1.39, 1.98-2.04, 2.35-2.39, 2.54-2.66,

55.4: -,

56.6: 1.39, 1.98-2.04, 2.35-2.39, 2.41-2.45, 2.54-2.66, 3.01,

91.2: 2.41-2.45, 2.54-2.66, 3.01, 6.83-6.84, 6.85-6.87, 7.20,

113.5: 6.83-6.84, 6.85-6.87, 7.20,

114.5: 2.41-2.45, 2.54-2.66, 3.01, 6.76-6.78, 6.85-6.87, 7.20,

121.2: 2.41-2.45, 2.54-2.66, 3.01, 6.76-6.78, 6.83-6.84, 7.20,

128.4: 7.47-7.50, 7.76-7.78,

128.7: 7.39-7.42, 7.47-7.50,

129.3: 6.85-6.87,

132.0: 7.76-7.78,

136.9: 7.39-7.42, 7.47-7.50,

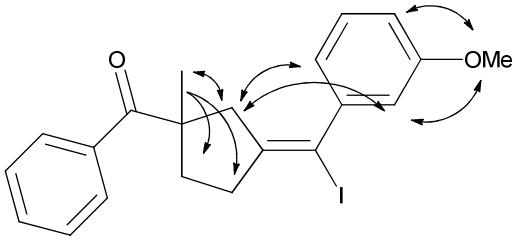
145.1: 6.76-6.78, 6.83-6.84, 6.85-6.87, 7.20,

149.4: 1.98-2.04, 2.35-2.39, 2.41-2.45, 2.54-2.66, 3.01,

159.4: 3.80, 6.76-6.78, 6.83-6.84, 6.85-6.87, 7.20,

205.7: 1.39, 1.98-2.04, 2.35-2.39, 2.41-2.45, 3.01, 7.39-7.42, 7.76-7.78.

NOESY (^{1}H ↔ ^{1}H, ppm)



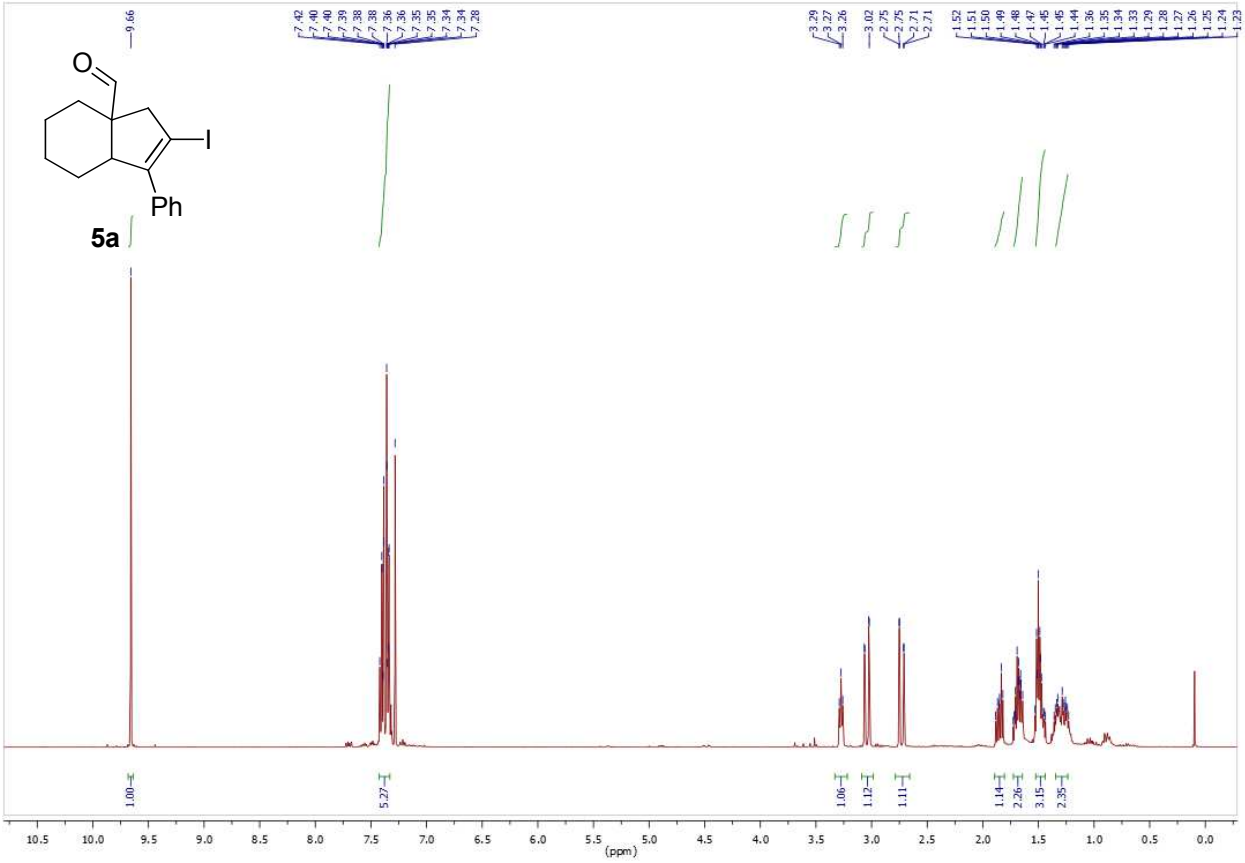
1.39: 1.98-2.04, 2.35-2.39, 2.41-2.45, 2.54-2.66, 7.76-7.78,

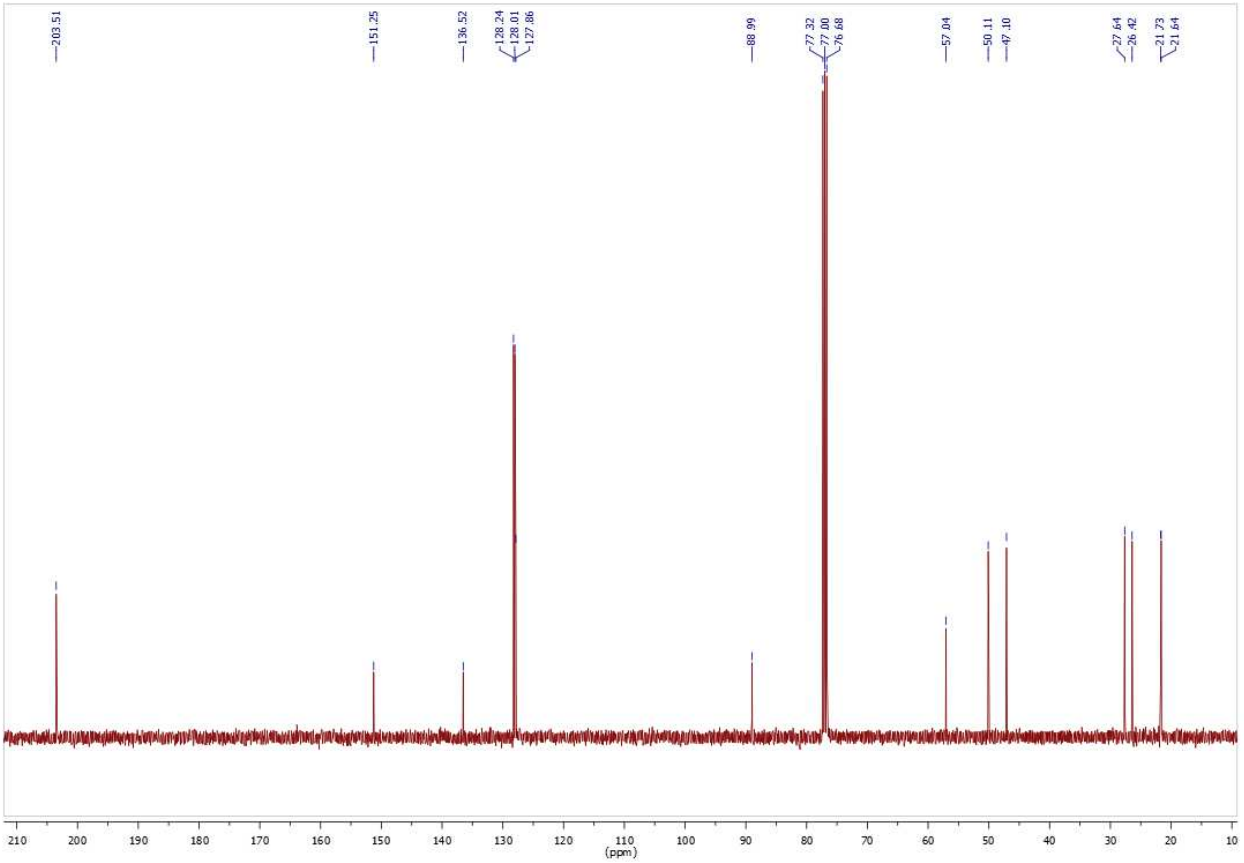
1.98-2.04: 1.39, 2.54-2.66,

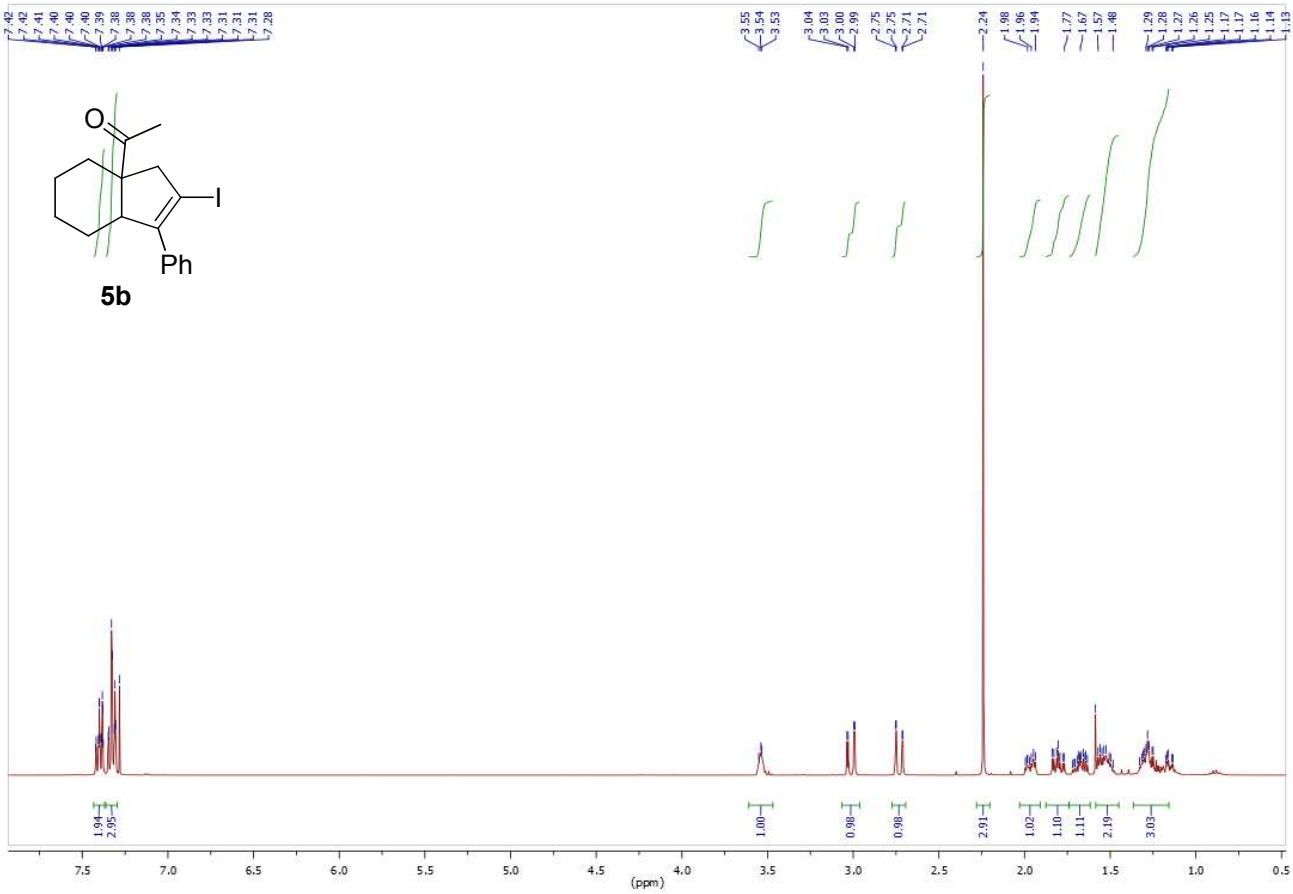
2.35-2.39: 1.98-2.04, 2.54-2.66,

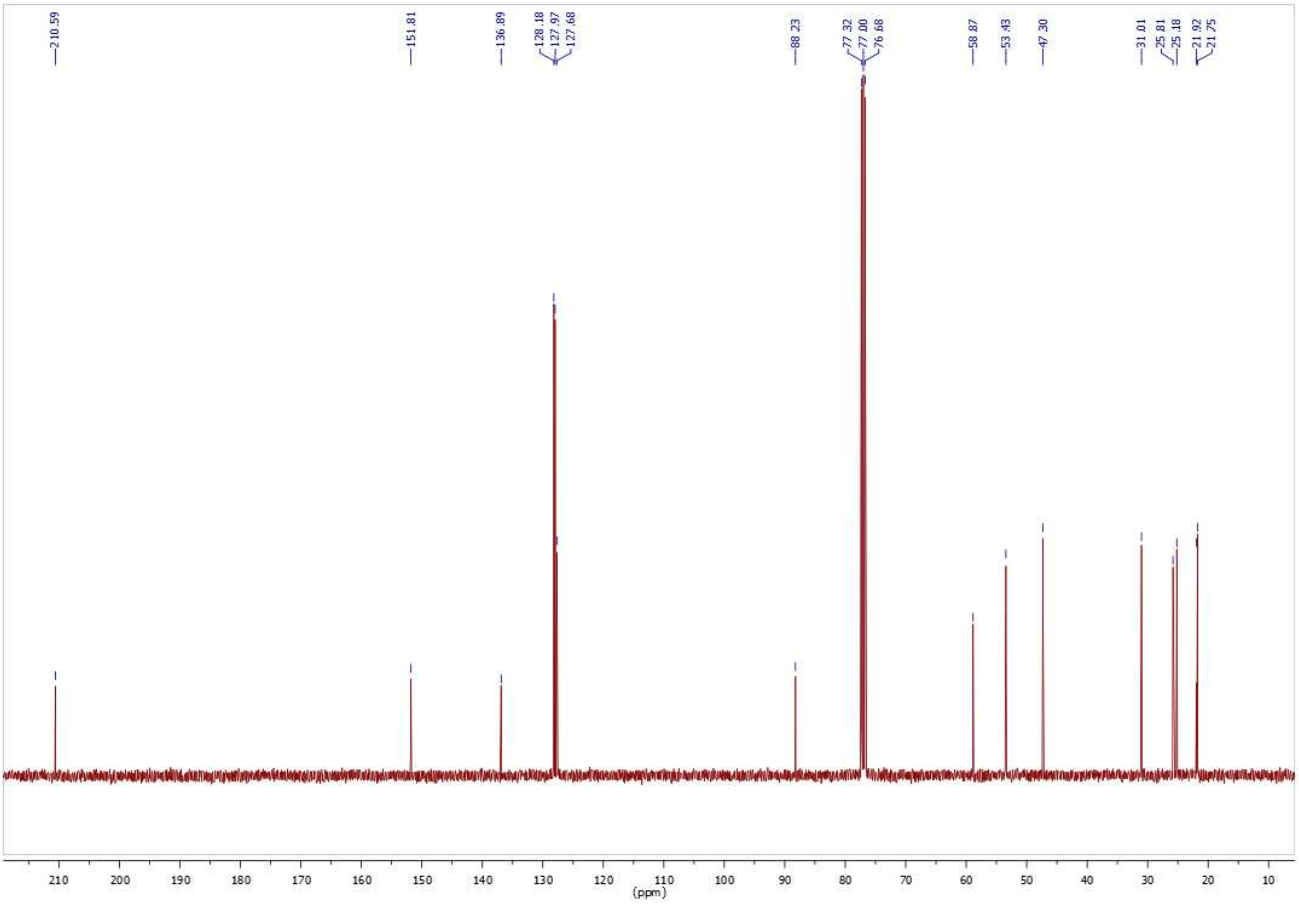
2.41-2.45:	1.39, 3.01, 6.83-6.84, 6.85-6.87,
2.54-2.66:	1.39, 1.98-2.04, 2.35-2.39,
3.01:	2.41-2.45, 6.83-6.84, 6.85-6.87,
3.80:	6.76-6.78, 6.83-6.84,
6.76-6.78:	3.80,
6.83-6.84:	2.41-2.45, 3.01, 3.80,
6.85-6.87:	2.41-2.45, 3.01,
7.20:	- ,
7.39-7.42:	7.76-7.78,
7.47-7.50:	7.76-7.78,
7.76-7.78:	1.39, 7.39-7.42, 7.47-7.50.

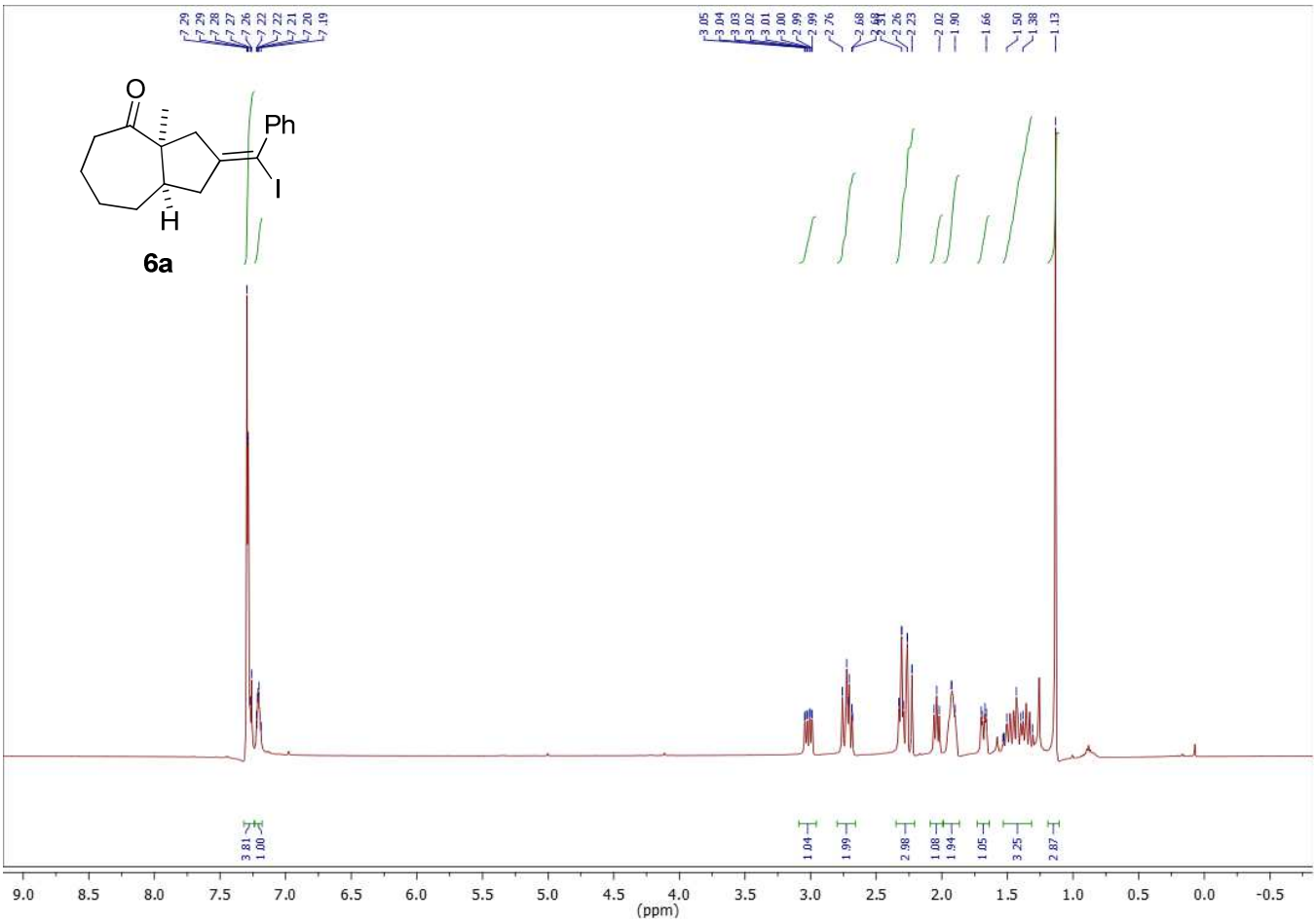
NMR-Spectra

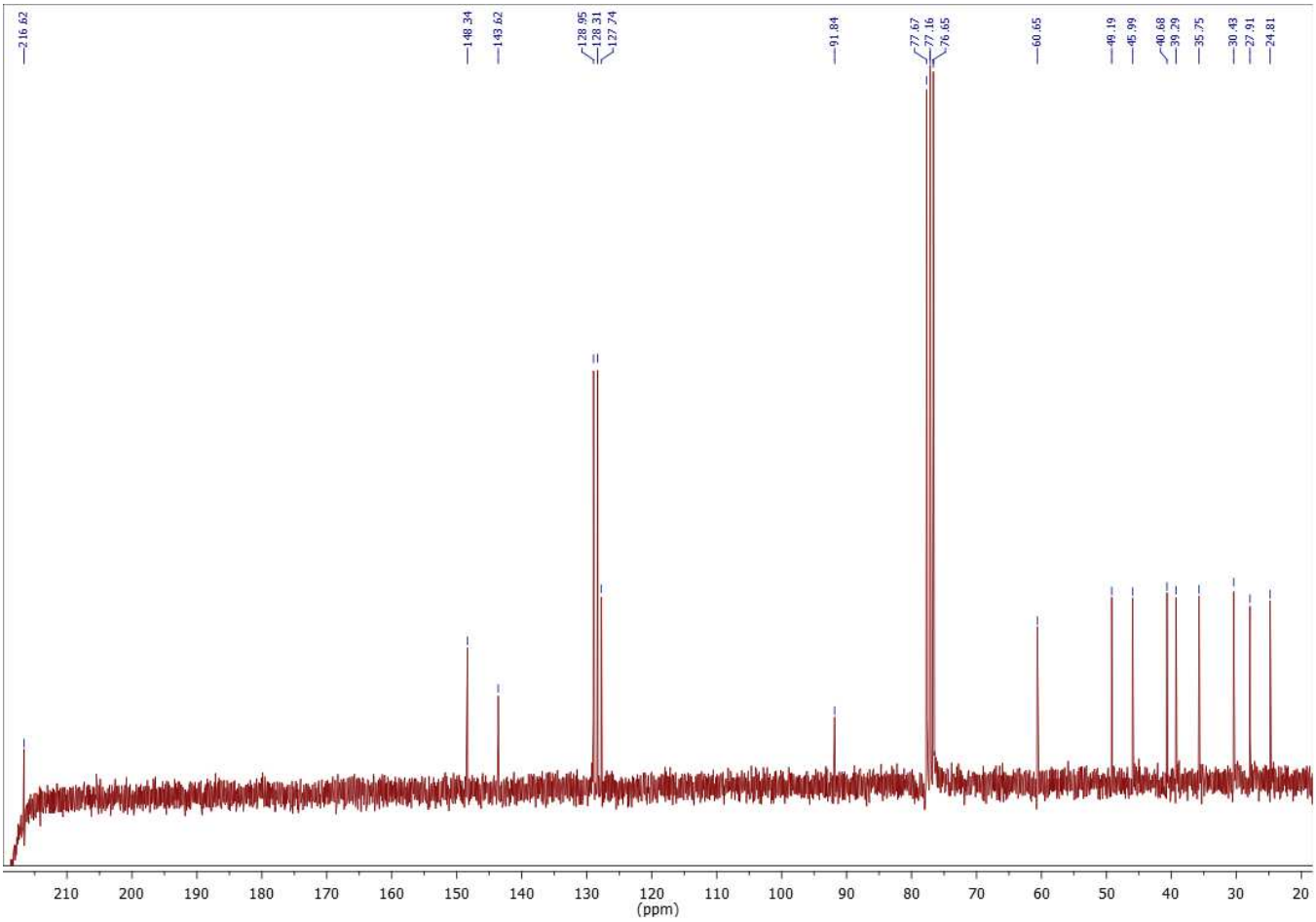

O
I
Ph
5a
9.66
7.42 7.40 7.40 7.39 7.38 7.38 7.36 7.36 7.35 7.35 7.34 7.34 7.28
3.29 3.27 3.26
3.02
2.75 2.75 2.71 2.71
1.52 1.51 1.50 1.49 1.48 1.47 1.45 1.45 1.44 1.36 1.35 1.34 1.33 1.29 1.28 1.27 1.26 1.25 1.24 1.23
1.00
5.27
1.06
1.12
1.11
1.14
2.26
3.15
2.35
10.5 10.0 9.5 9.0 8.5 8.0 7.5 7.0 6.5 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
(ppm)

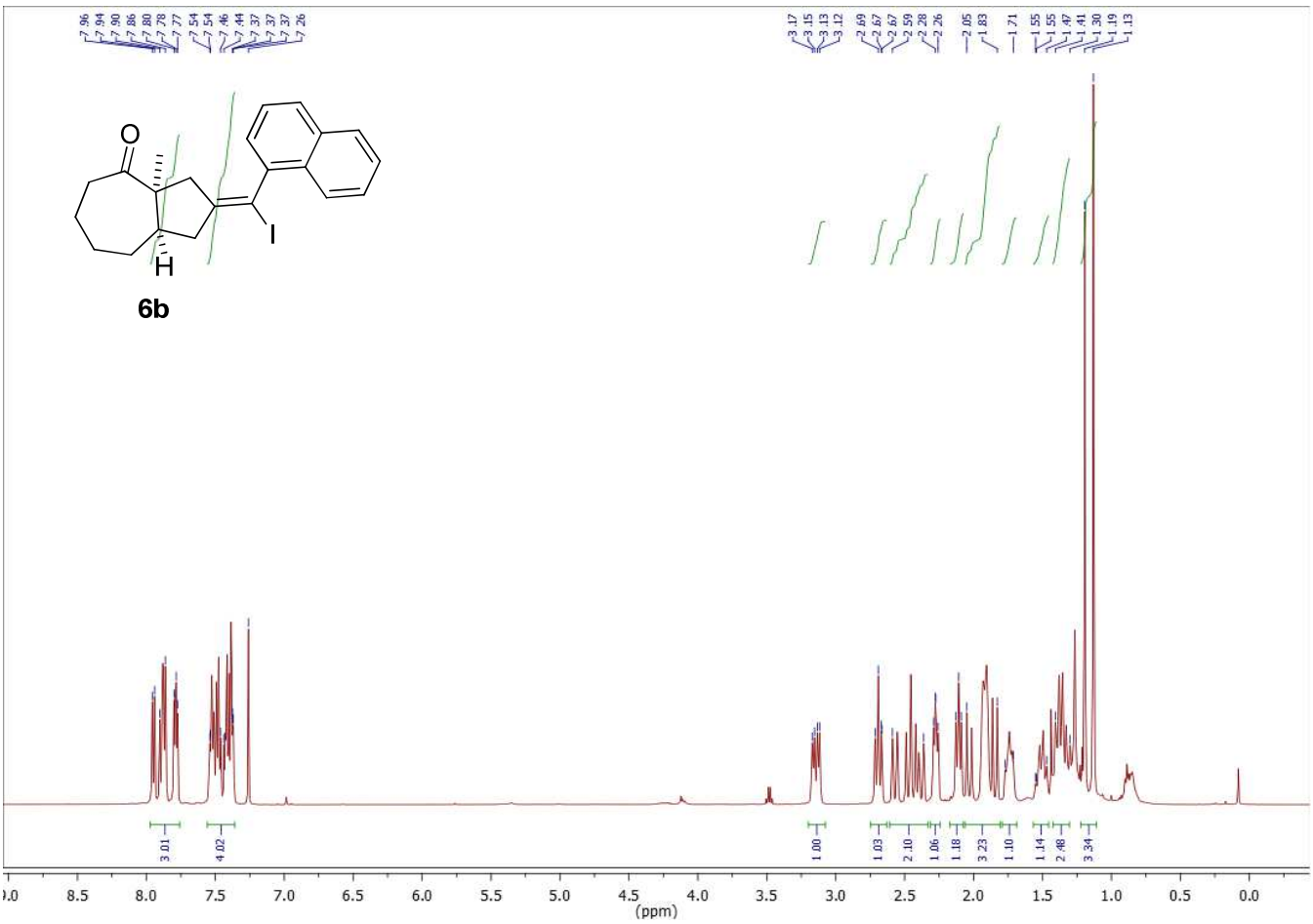

203.51
151.25
136.52
128.24
128.01
127.86
88.99
77.32
77.00
76.68
57.04
50.11
47.10
27.64
26.42
21.73
21.64
210 200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10
(ppm)

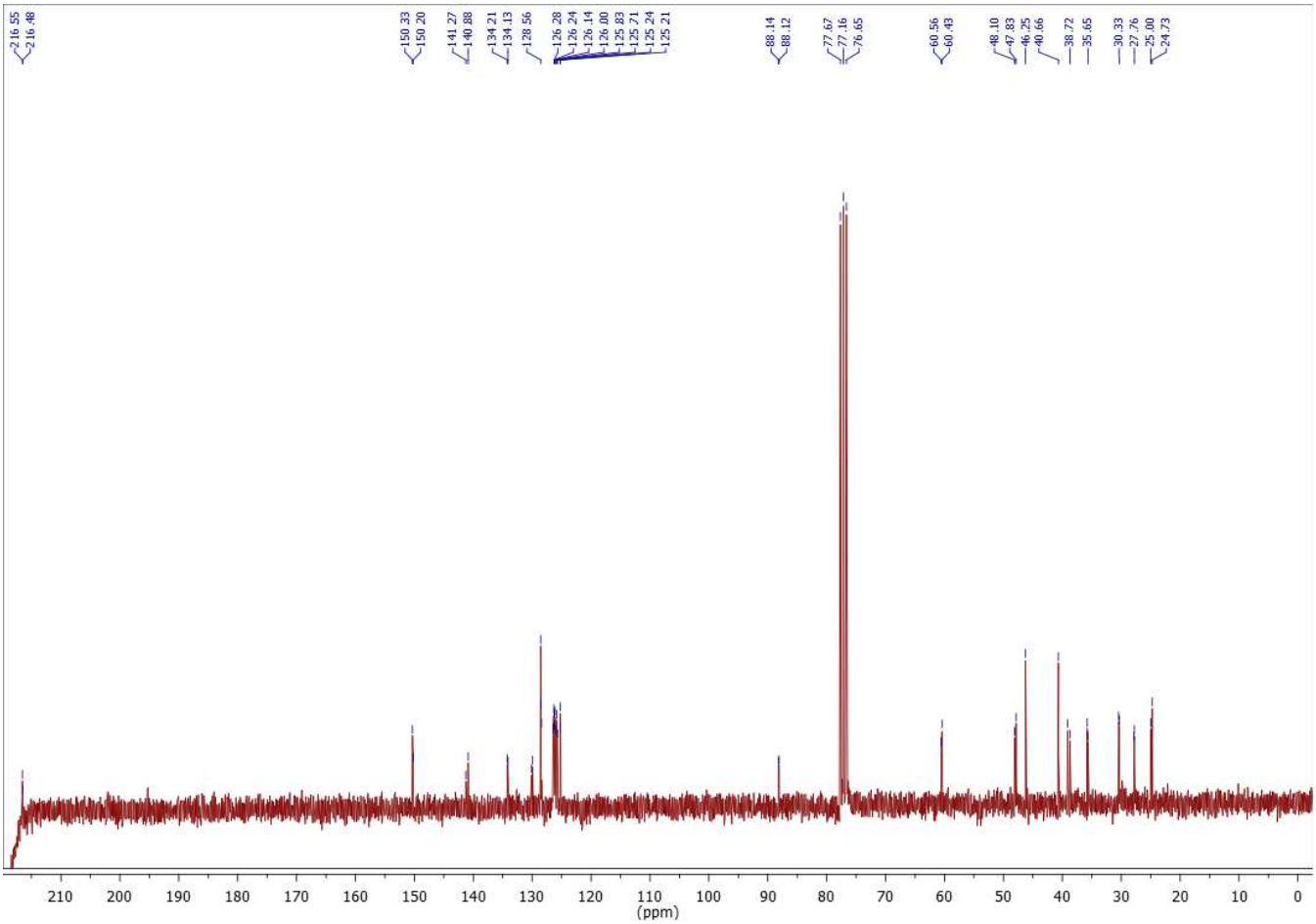

O
I
Ph
5b
7.42 7.42 7.41 7.40 7.40 7.40 7.39 7.38 7.38 7.35 7.34 7.33 7.31 7.31 7.28
3.55 3.54 3.53
3.04 3.03 3.00 2.99
2.75 2.75 2.71 2.71
2.24
1.98 1.96 1.94
1.77 1.67 1.57 1.48
1.29 1.28 1.27 1.26 1.25 1.17 1.17 1.16 1.14 1.13
1.94 2.95 1.00 0.98 0.98 2.91 1.02 1.10 1.11 2.19 3.03
7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 0.5
(ppm)

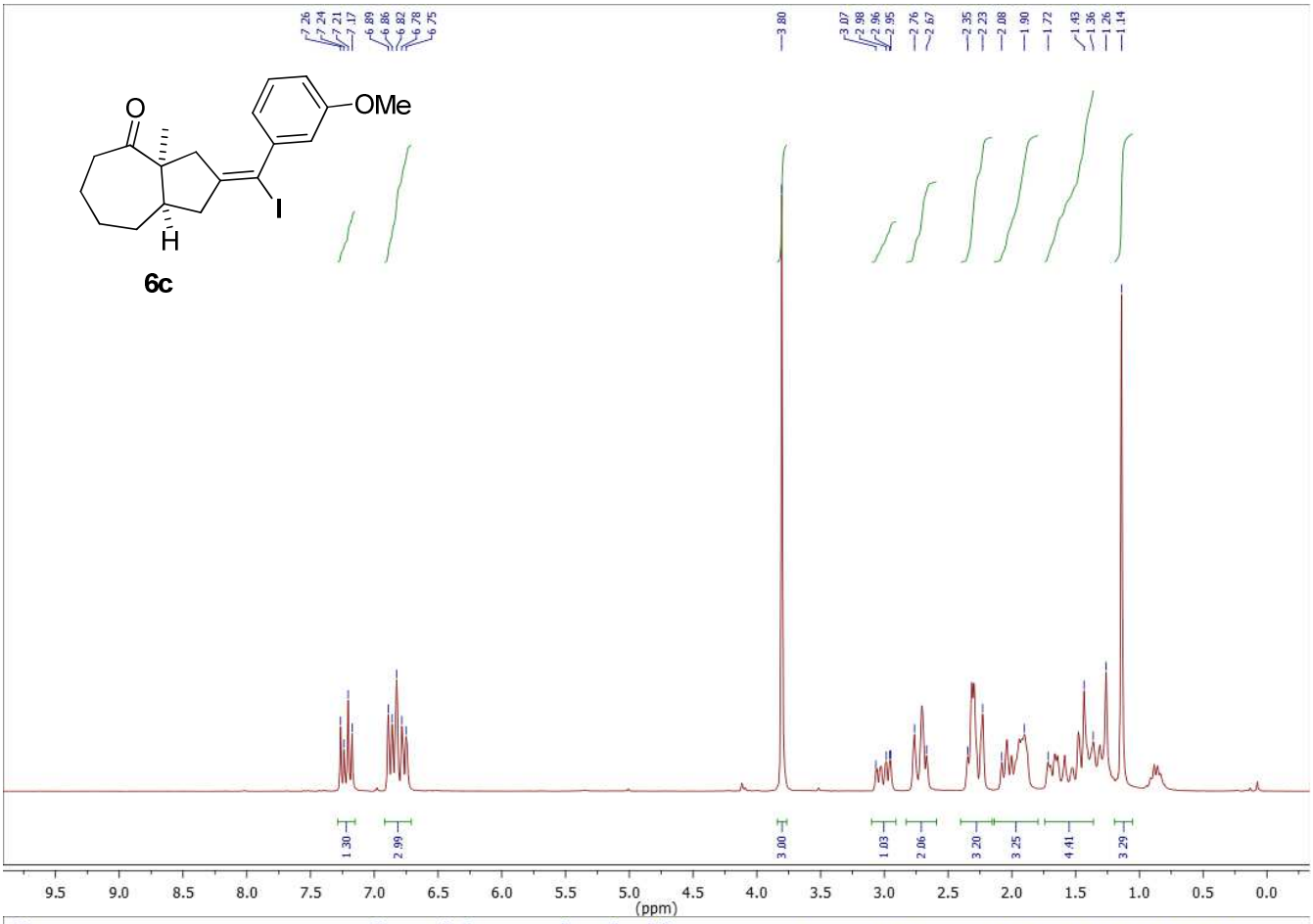

210.59
151.81
136.89
128.18 127.97 127.68
88.23
77.32 77.00 76.68
58.87
53.43
47.30
31.01
25.81 25.18
21.92 21.75
210 200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10
(ppm)

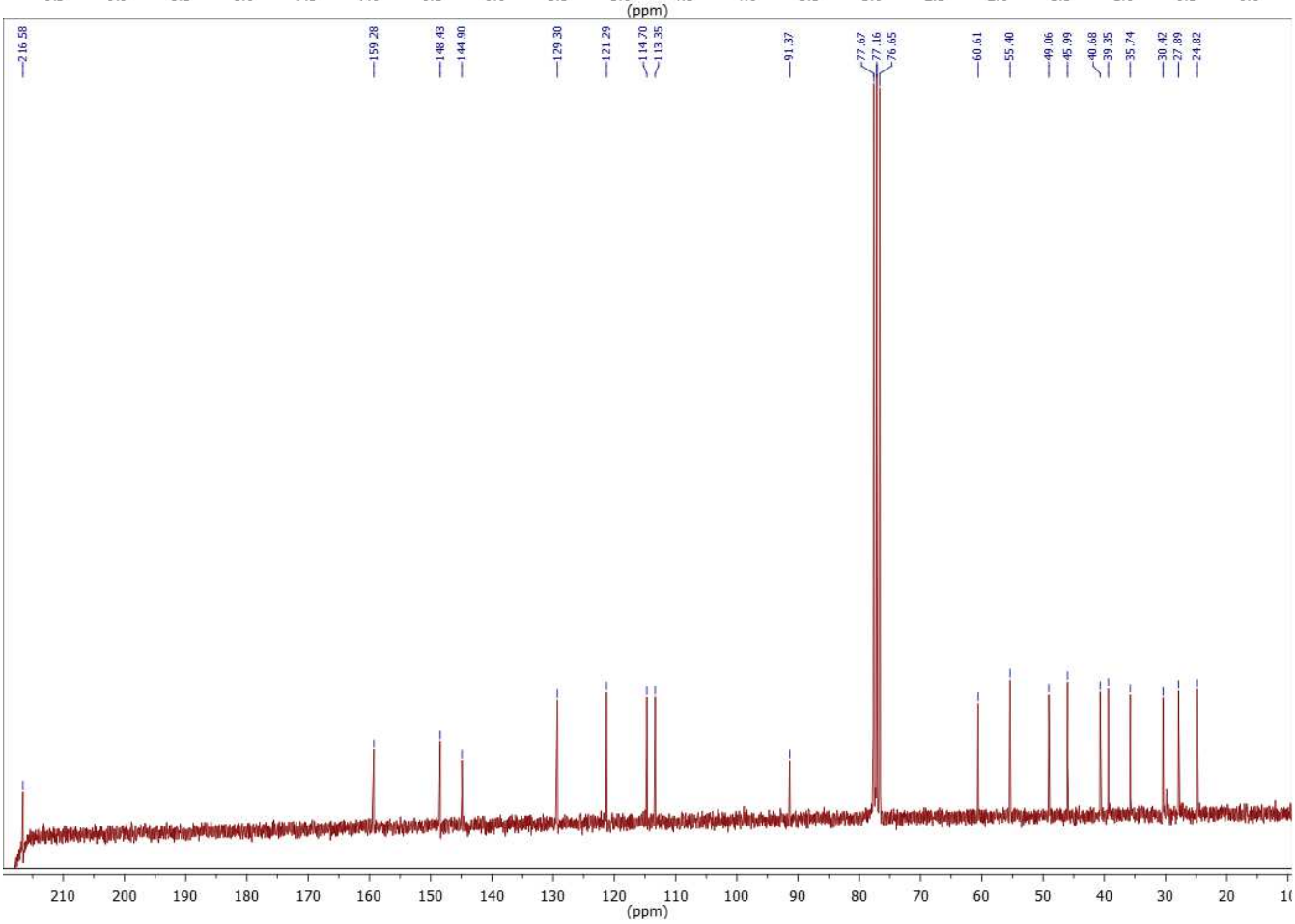

O
Ph
I
H
6a
7.29 7.29 7.28 7.27 7.26 7.22 7.22 7.21 7.20 7.19
3.05 3.04 3.03 3.02 3.01 3.00 2.99 2.99 2.76 2.68 2.64 2.26 2.23 2.02 1.90 1.66 1.50 1.38 1.13
3.81 1.00 1.04 1.99 2.98 1.08 1.94 1.05 3.25 2.87
9.0 8.5 8.0 7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 0.5 0.0 -0.5
(ppm)

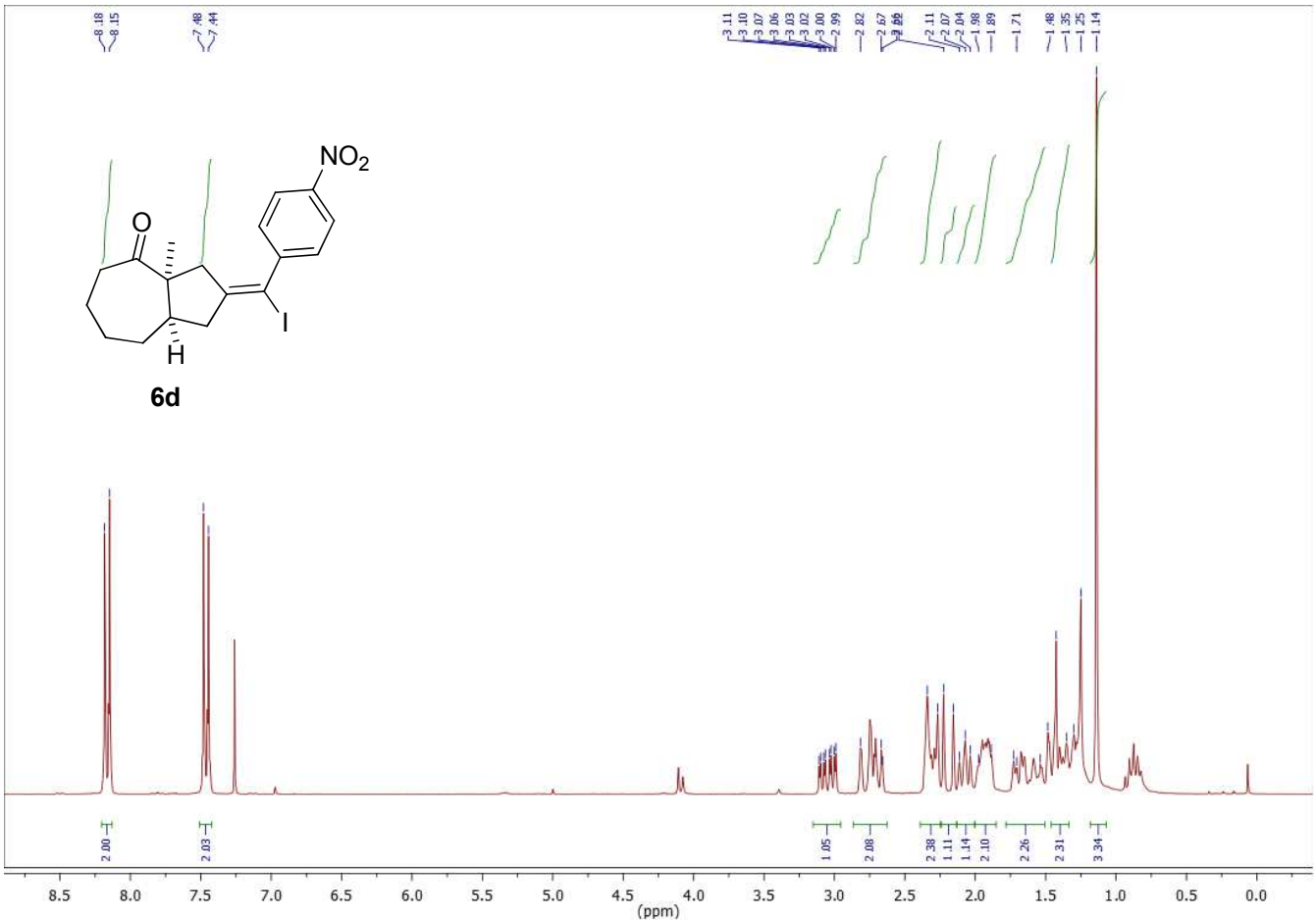

216.62
148.34 143.62
128.95 128.31 127.74
91.84
77.67 77.16 76.65
60.65
49.19 45.99 40.68 39.29 35.75 30.43 27.91 24.81
210 200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20
(ppm)

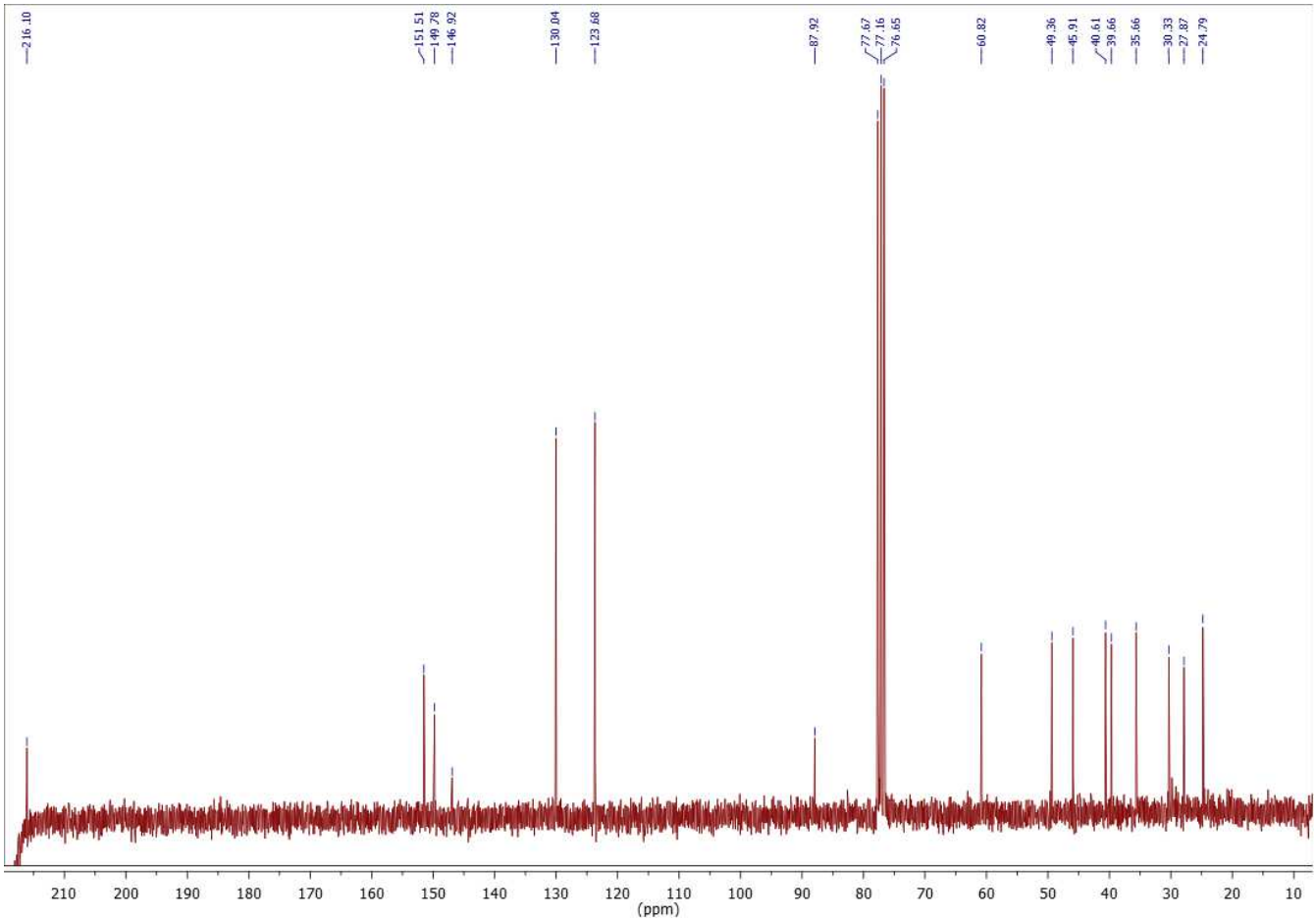

O
I
H
6b
(ppm)

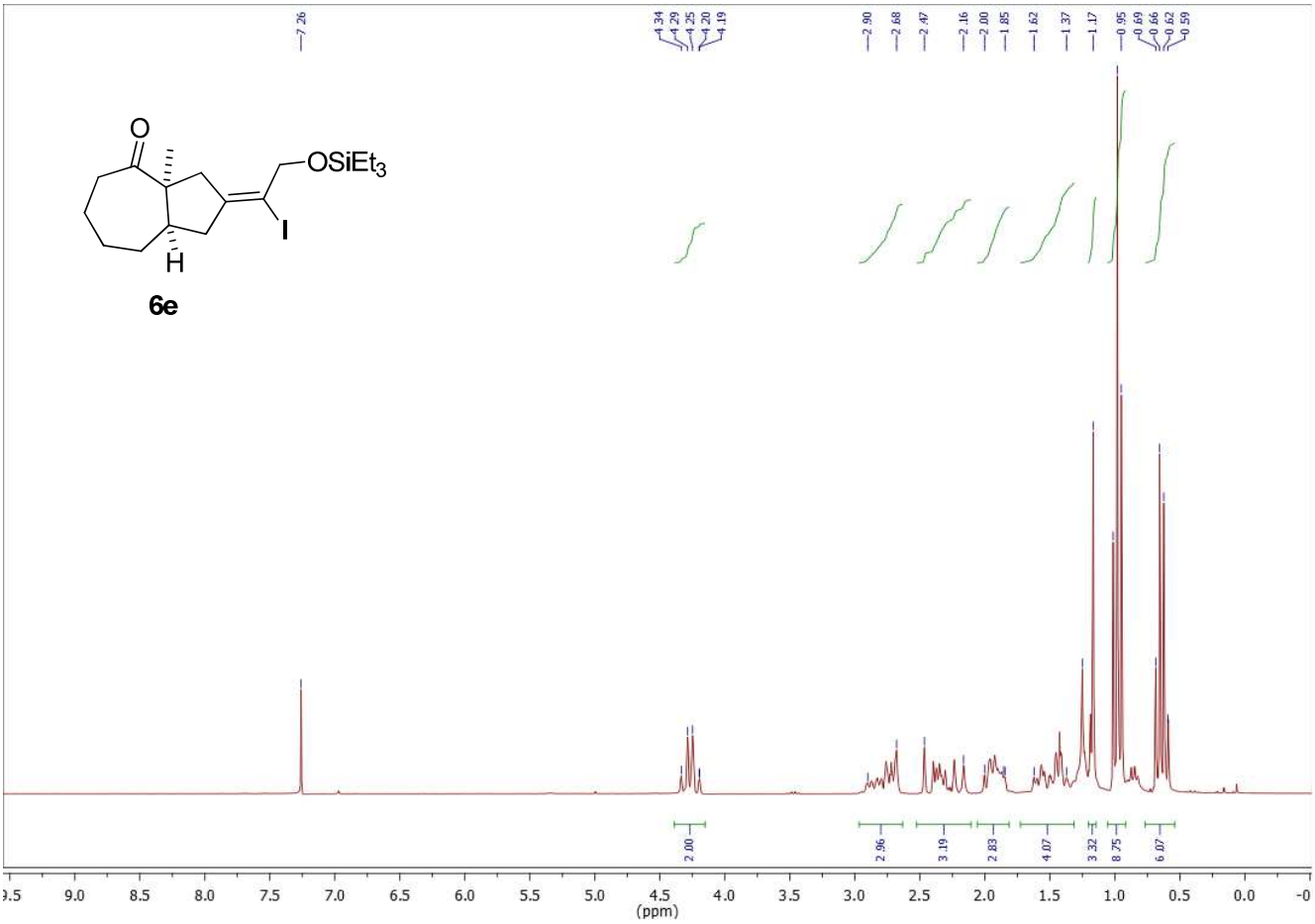

(ppm)

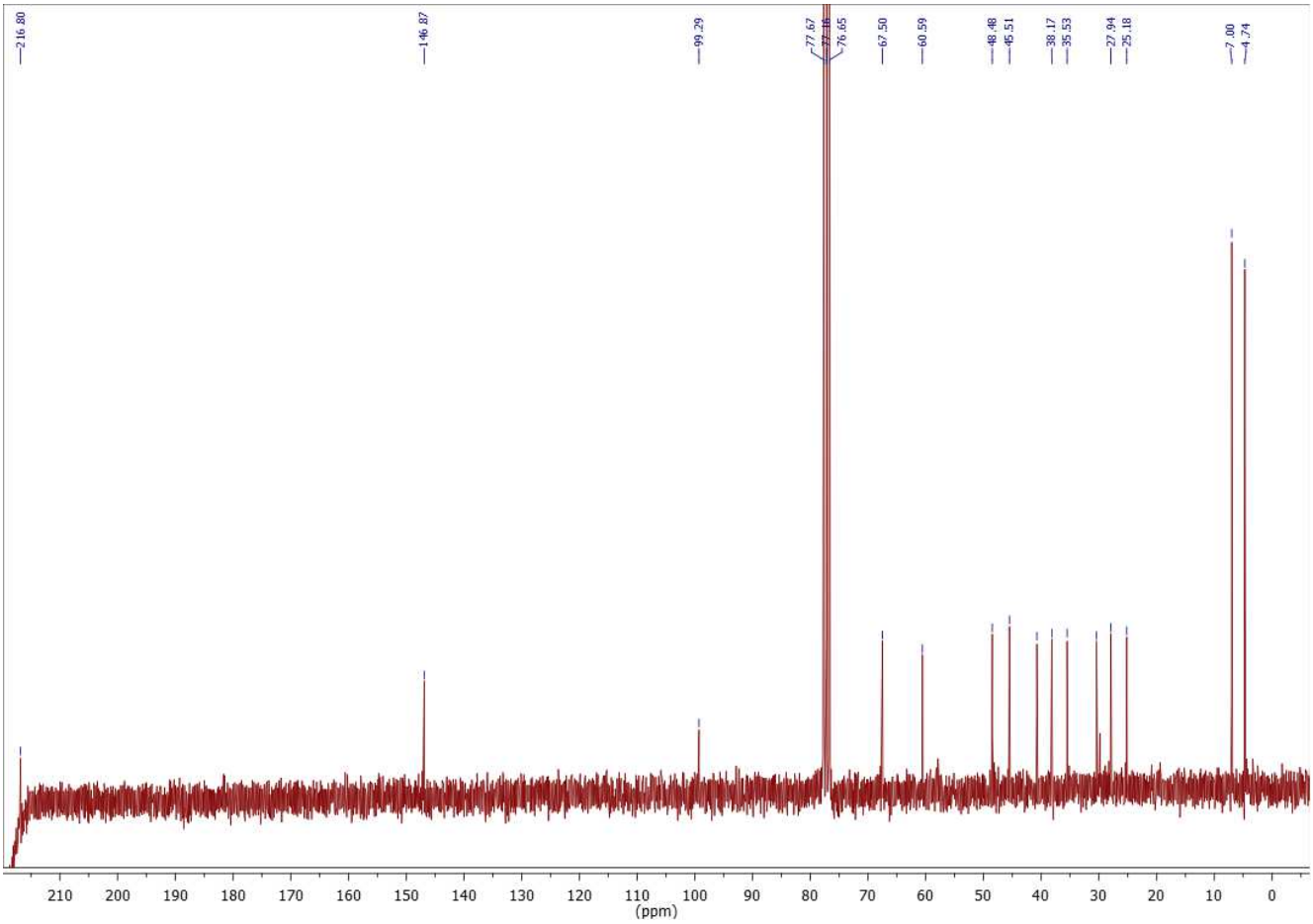

O
OMe
I
H
6c
7.26
7.24
7.21
7.17
6.89
6.86
6.82
6.78
6.75
3.80
3.07
2.98
2.96
2.95
2.76
2.67
2.35
2.23
2.08
1.90
1.72
1.43
1.36
1.26
1.14
1.30
2.99
3.00
1.03
2.06
3.20
3.25
4.41
3.29
9.5 9.0 8.5 8.0 7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 0.5 0.0
(ppm)

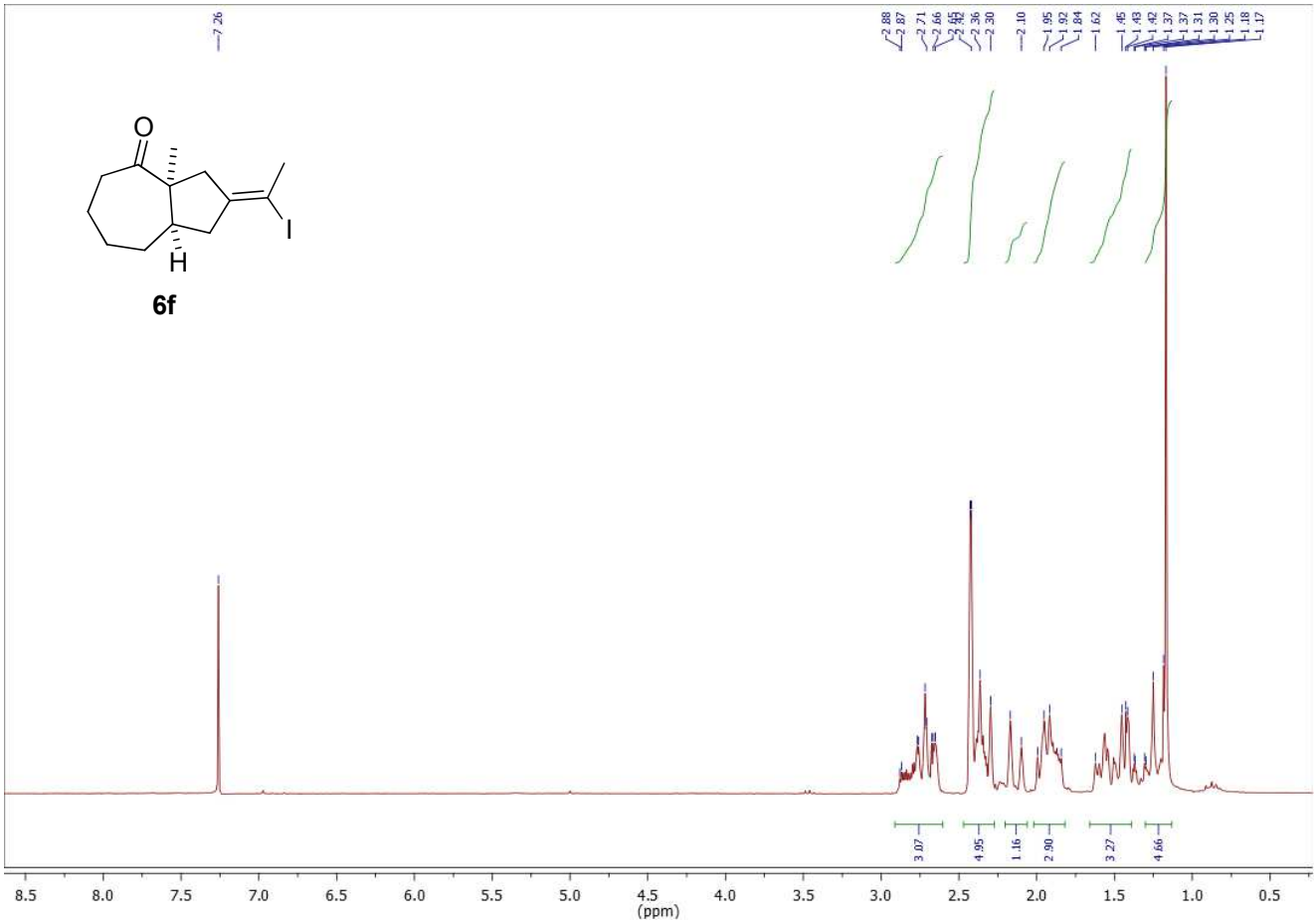

216.58
159.28
148.43
144.90
129.30
121.29
114.70
113.35
91.37
77.67
77.16
76.65
60.61
55.40
49.06
45.99
40.68
39.35
35.74
30.42
27.89
24.82
210 200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20
(ppm)

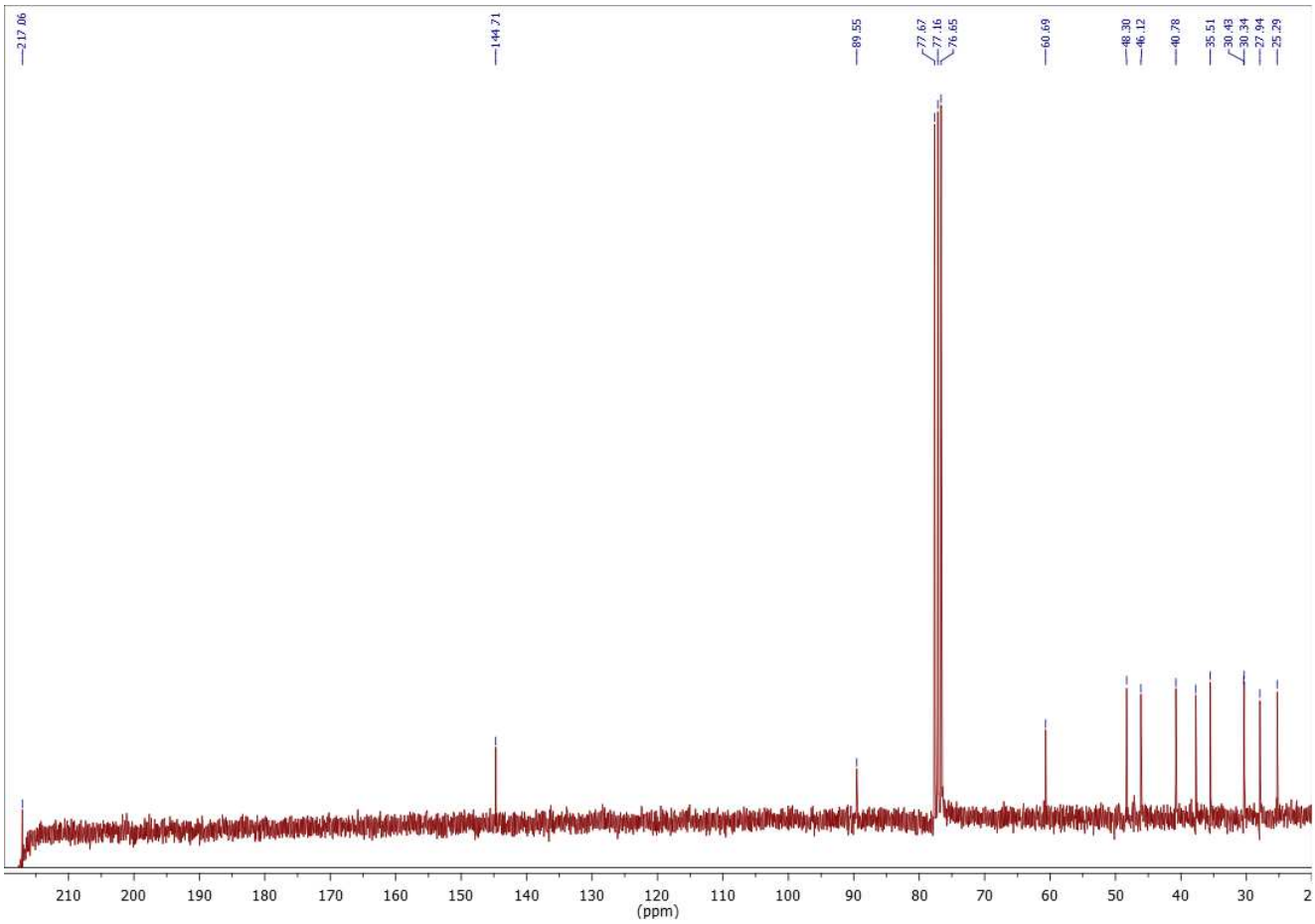

NO2
O
I
H
6d
8.18
8.15
7.48
7.44
3.11
3.10
3.07
3.06
3.03
3.02
3.00
2.99
2.82
2.67
2.11
2.07
2.04
1.98
1.89
1.71
1.48
1.35
1.25
1.14
2.00
2.03
1.05
2.08
2.38
1.11
1.14
2.10
2.26
2.31
3.34
8.5
8.0
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
0.5
0.0
(ppm)

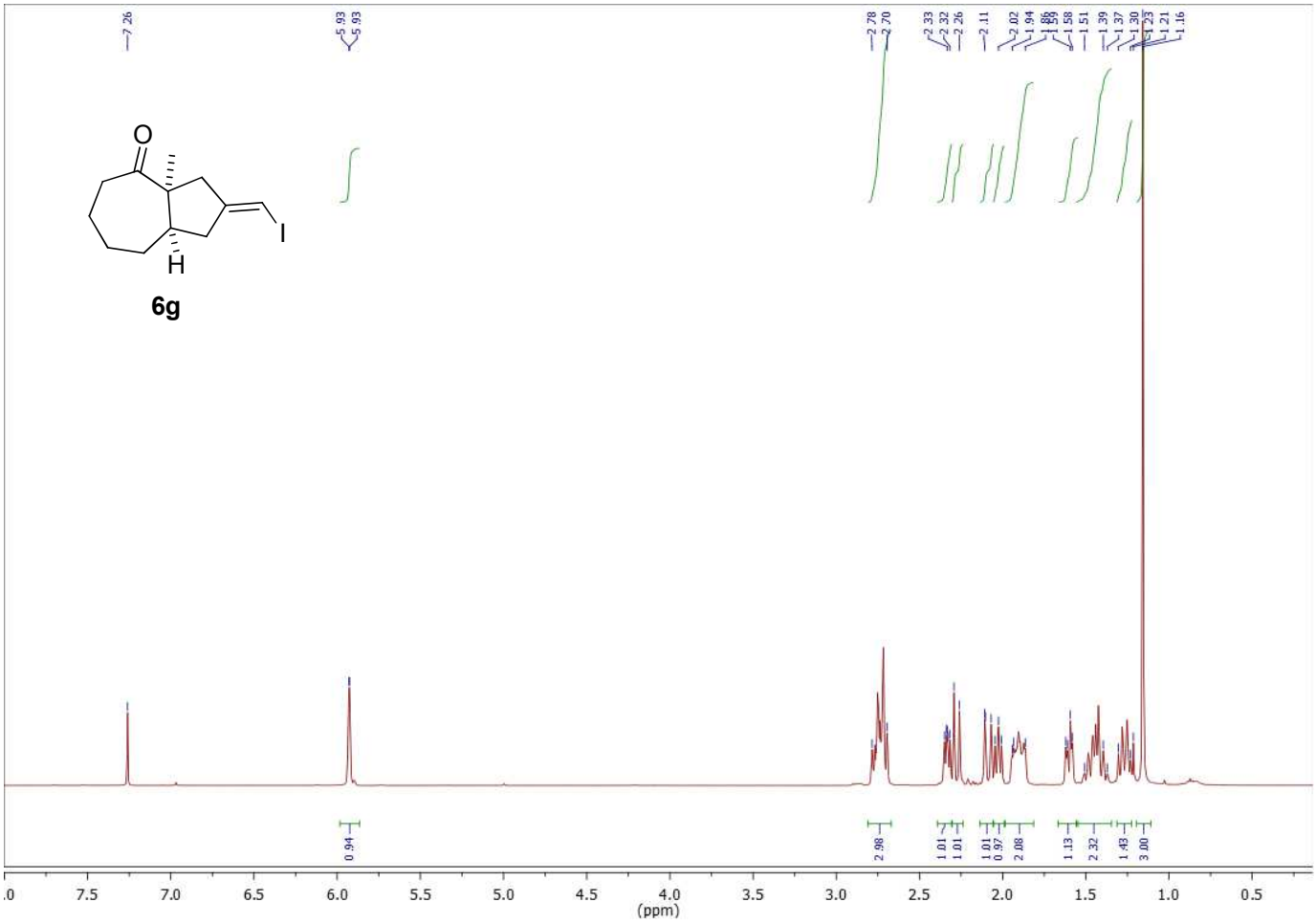

216.10
151.51
149.78
146.92
130.04
123.68
87.92
77.67
77.16
76.65
60.82
49.36
45.91
40.61
39.66
35.66
30.33
27.87
24.79
210
200
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
(ppm)

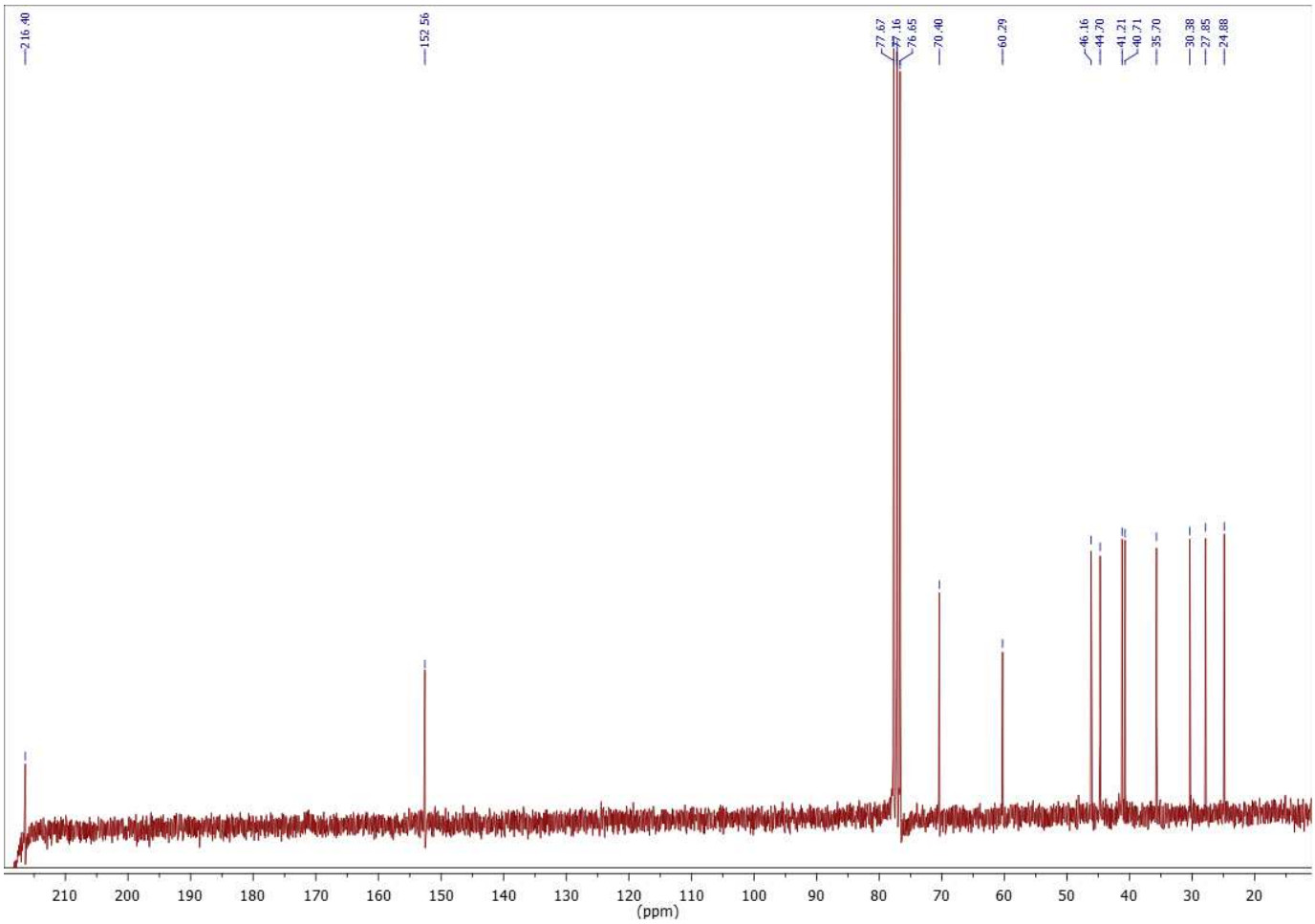

O
OSiEt3
I
H
6e
7.26
4.34
4.29
4.25
4.20
4.19
2.90
2.68
2.47
2.16
2.00
1.85
1.62
1.37
1.17
0.95
0.69
0.66
0.62
0.59
2.00
2.96
3.19
2.83
4.07
3.32
8.75
6.07
(ppm)

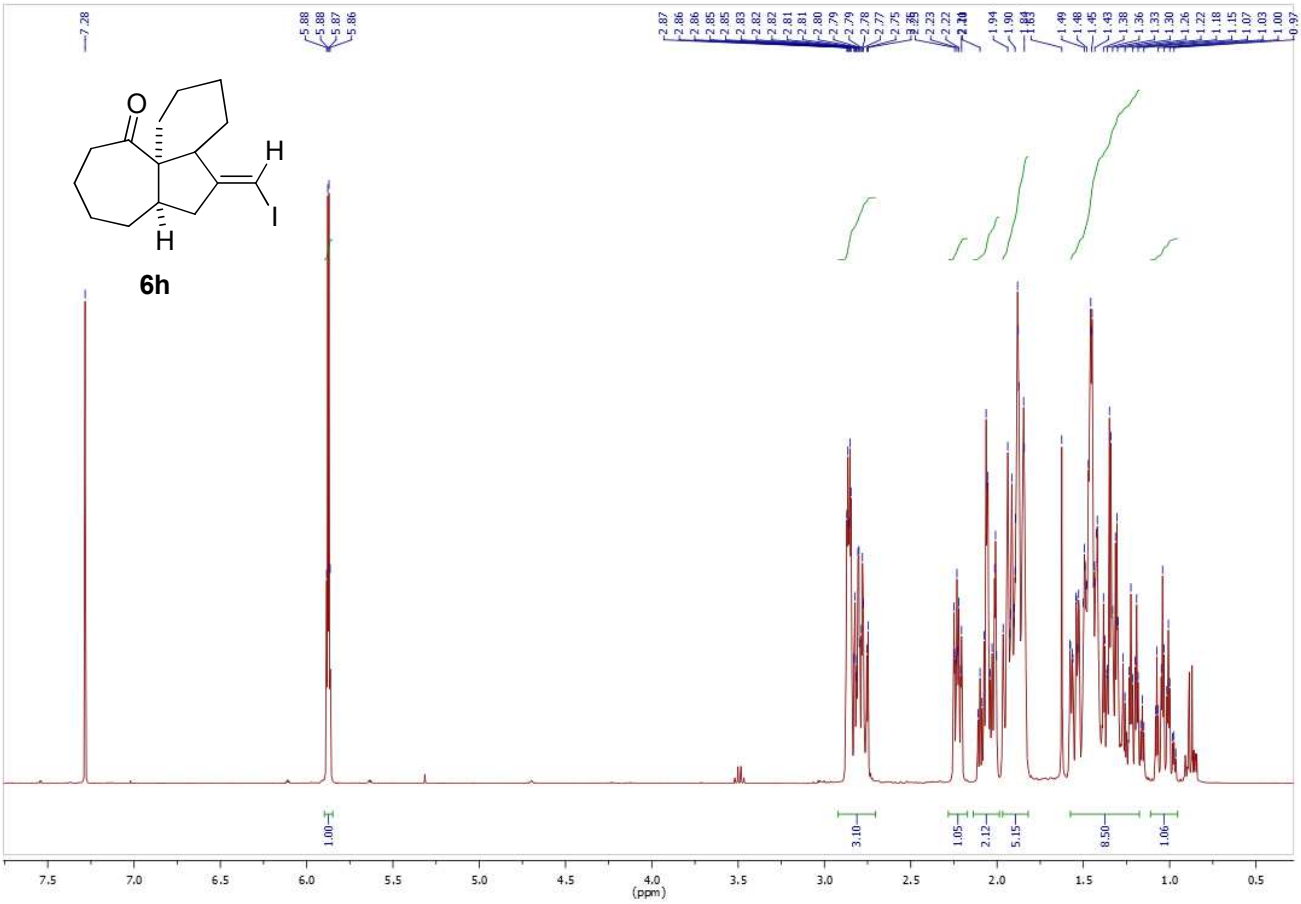

216.80
146.87
99.29
77.67
77.16
76.65
67.50
60.59
48.48
45.51
38.17
35.53
27.94
25.18
7.00
4.74
(ppm)

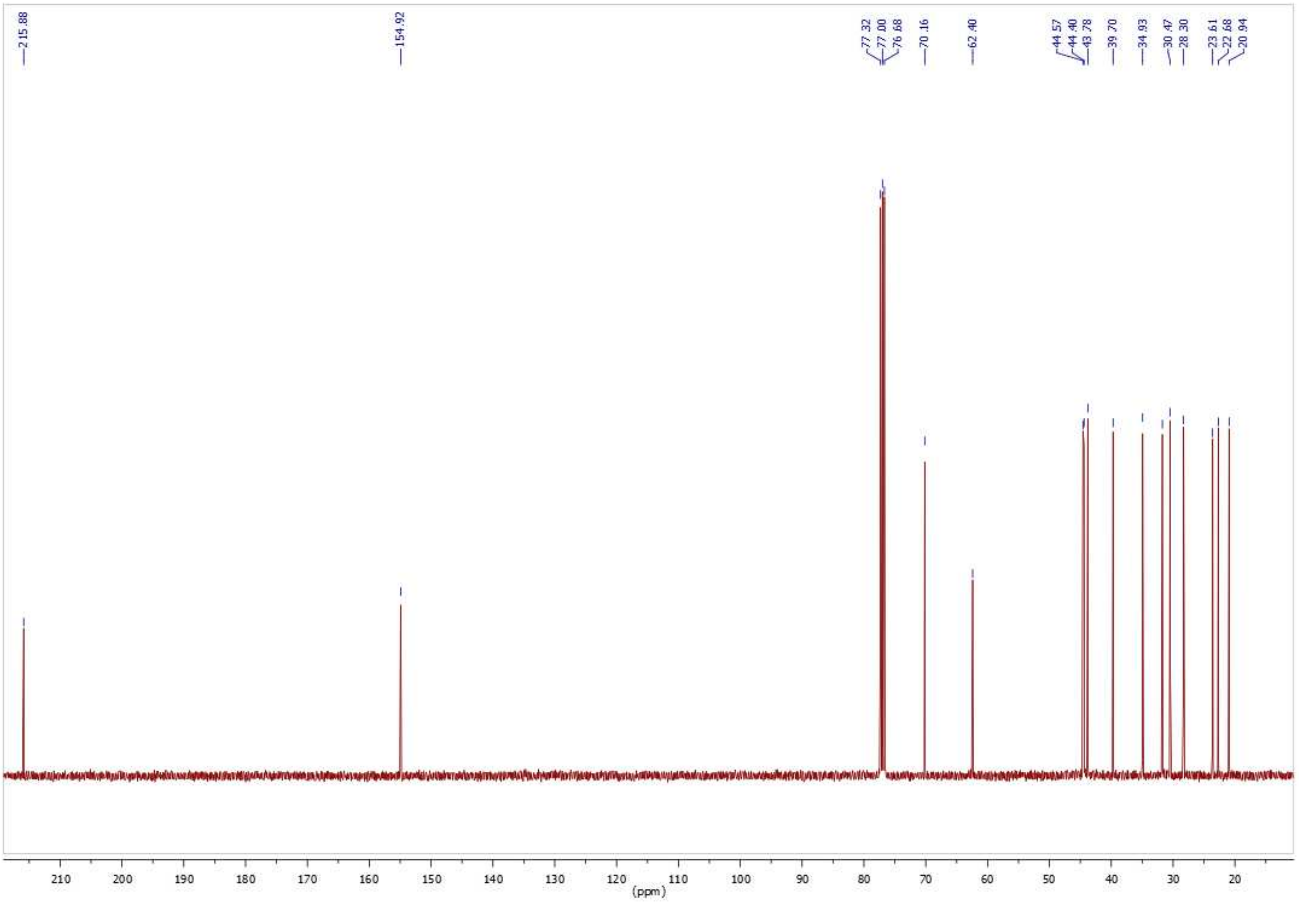

7.26
2.88
2.87
2.71
2.66
2.45
2.36
2.30
2.10
1.95
1.92
1.84
1.62
1.45
1.43
1.42
1.37
1.37
1.31
1.30
1.25
1.18
1.17
O
I
H
6f
3.07
4.95
1.16
2.90
3.27
4.66
8.5
8.0
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
0.5
(ppm)

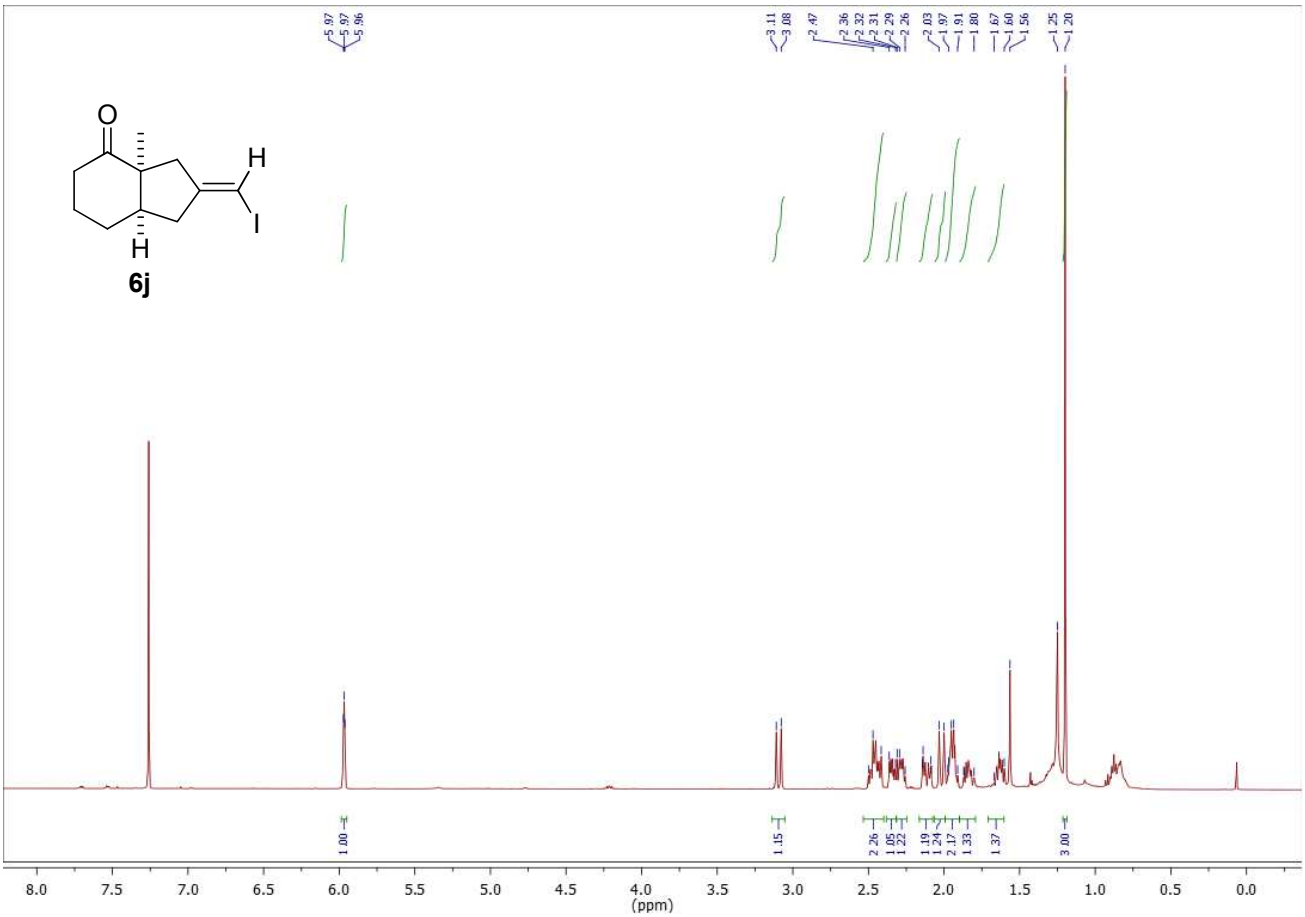

217.06
144.71
89.55
77.67
77.16
76.65
60.69
48.30
46.12
40.78
35.51
30.43
30.34
27.94
25.29
210
200
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
(ppm)

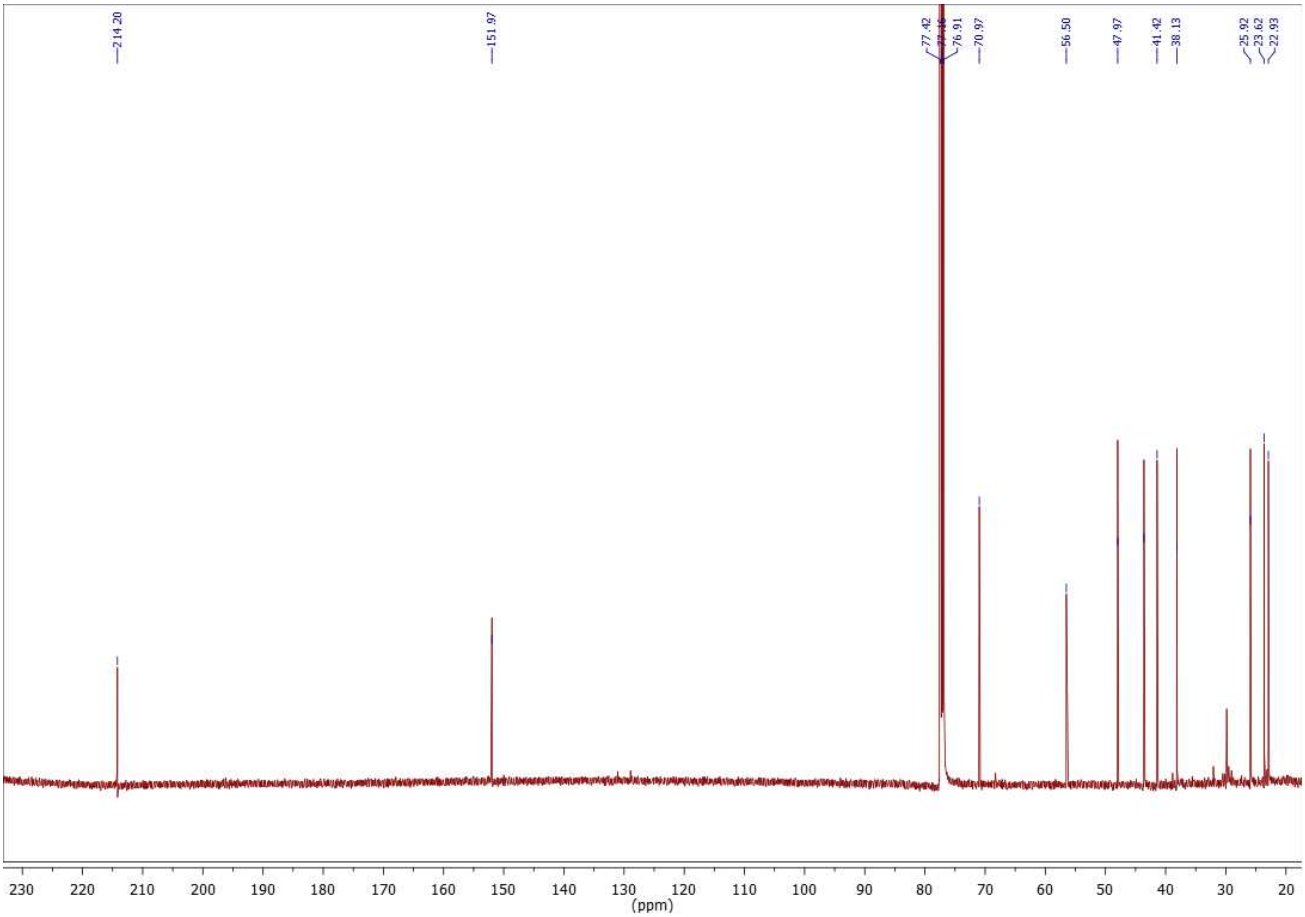

O
I
H
6g
7.26
5.93
5.93
2.78
2.70
2.33
2.32
2.26
2.11
2.02
1.94
1.58
1.51
1.39
1.37
1.30
1.23
1.21
1.16
0.94
2.98
1.01
1.01
1.01
0.97
2.08
1.13
2.32
1.43
3.00
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
0.5
(ppm)

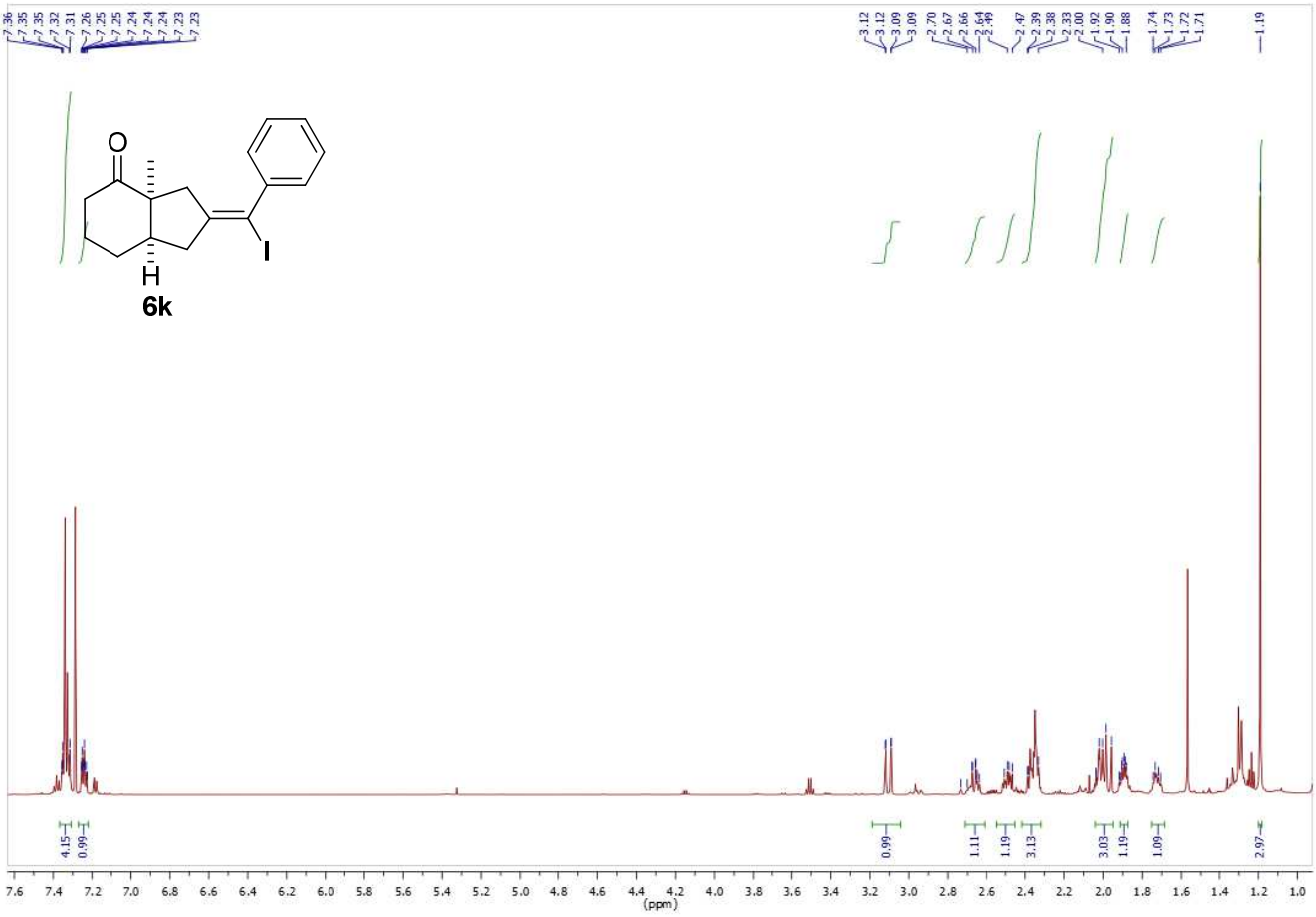

216.40
152.56
77.67
77.16
76.65
70.40
60.29
46.16
44.70
41.21
40.71
35.70
30.38
27.85
24.88
210
200
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
(ppm)

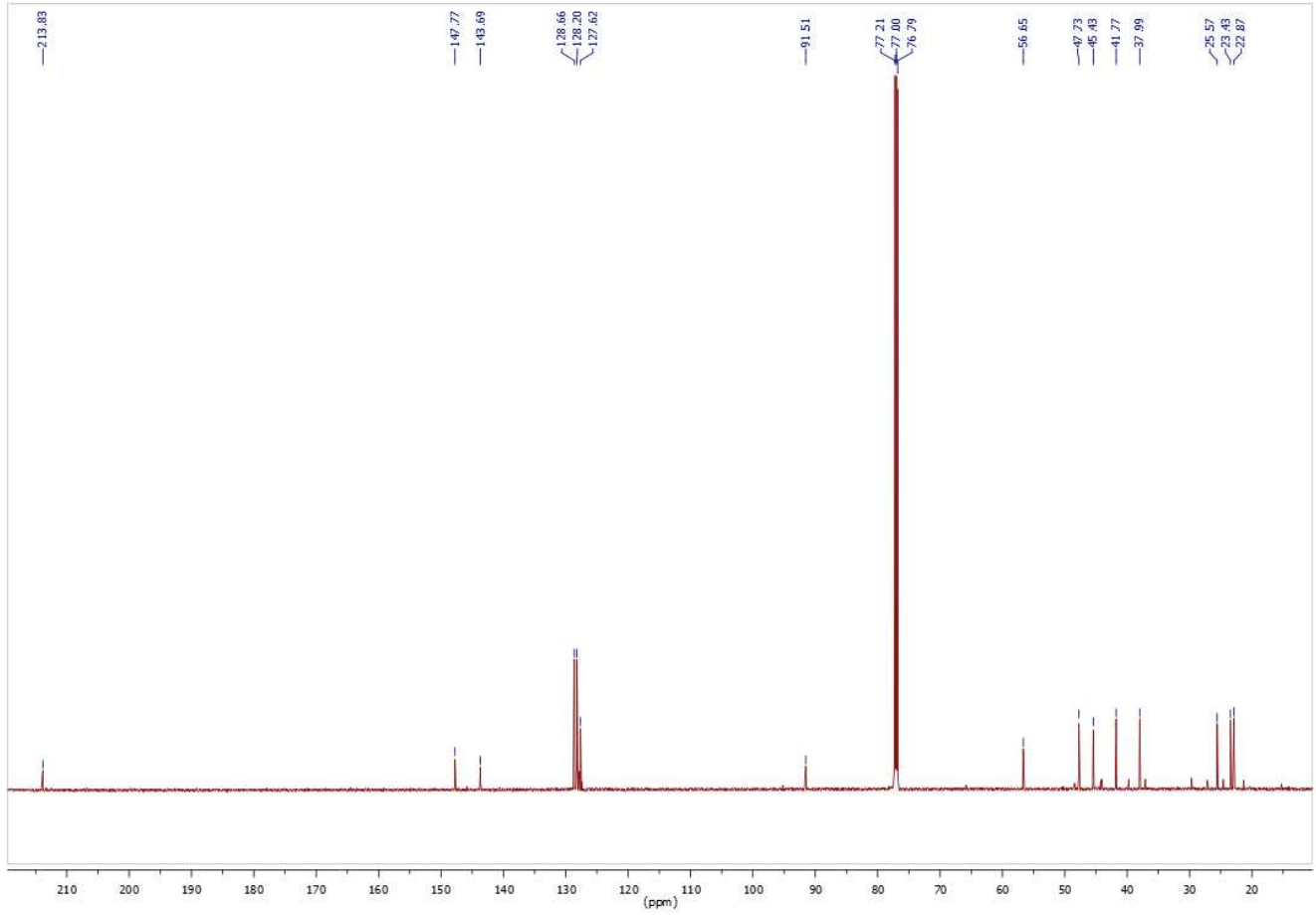

O
H
I
H
6h
7.26
5.88
5.87
5.86
1.00
3.10
1.05
2.12
5.15
8.50
1.06
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
0.5
(ppm)

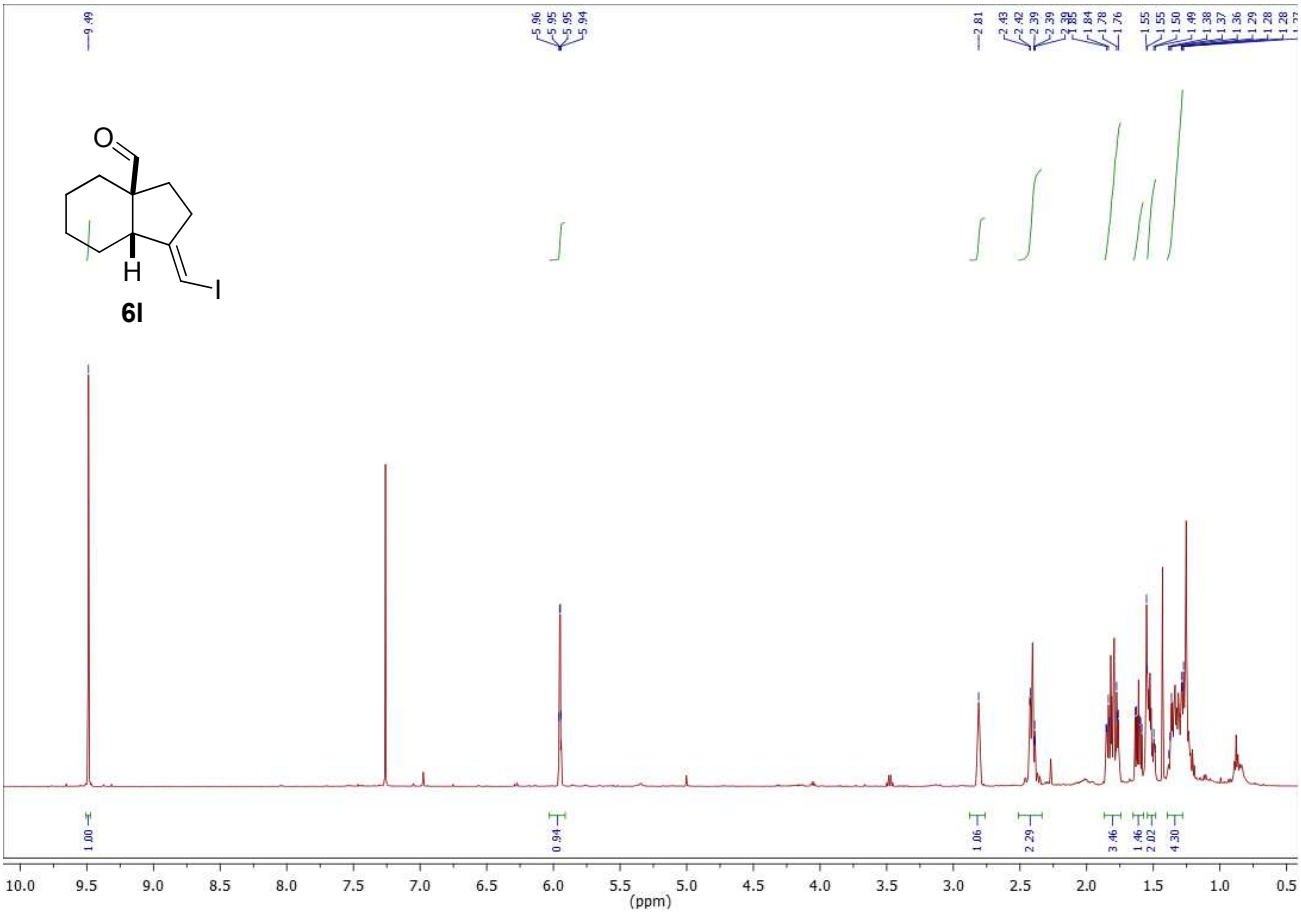

215.88
154.92
77.32
77.00
76.68
70.16
62.40
44.57
44.40
43.78
39.70
34.93
30.47
28.30
23.61
22.68
20.94
210
200
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
(ppm)

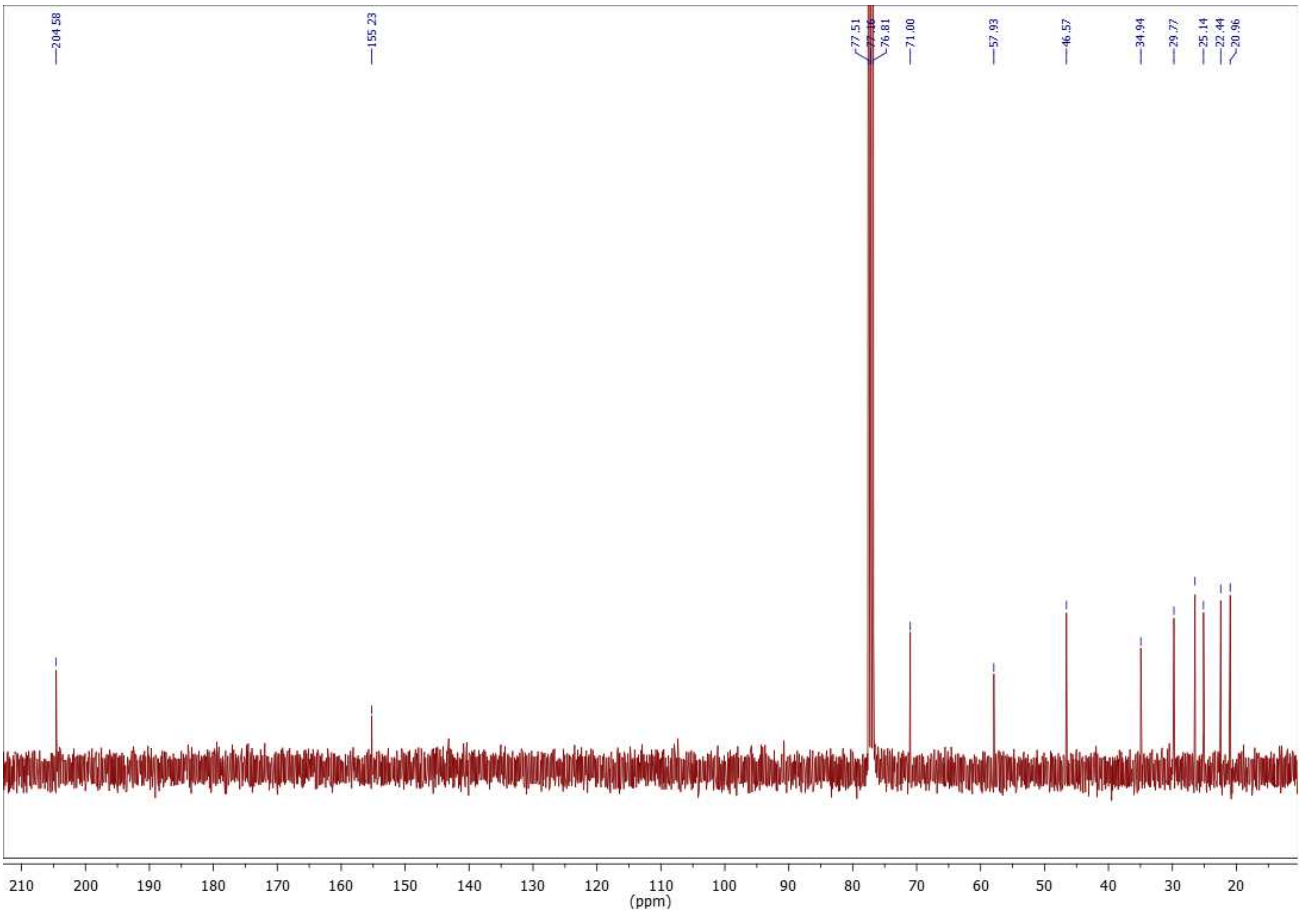

O
H
I
H
6j
5.97
5.97
5.96
3.11
3.08
2.47
2.36
2.32
2.31
2.29
2.26
2.03
1.97
1.91
1.80
1.67
1.60
1.56
1.25
1.20
1.00
1.15
2.26
1.05
1.22
1.19
1.24
2.17
1.33
1.37
3.00
8.0
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
0.5
0.0
(ppm)

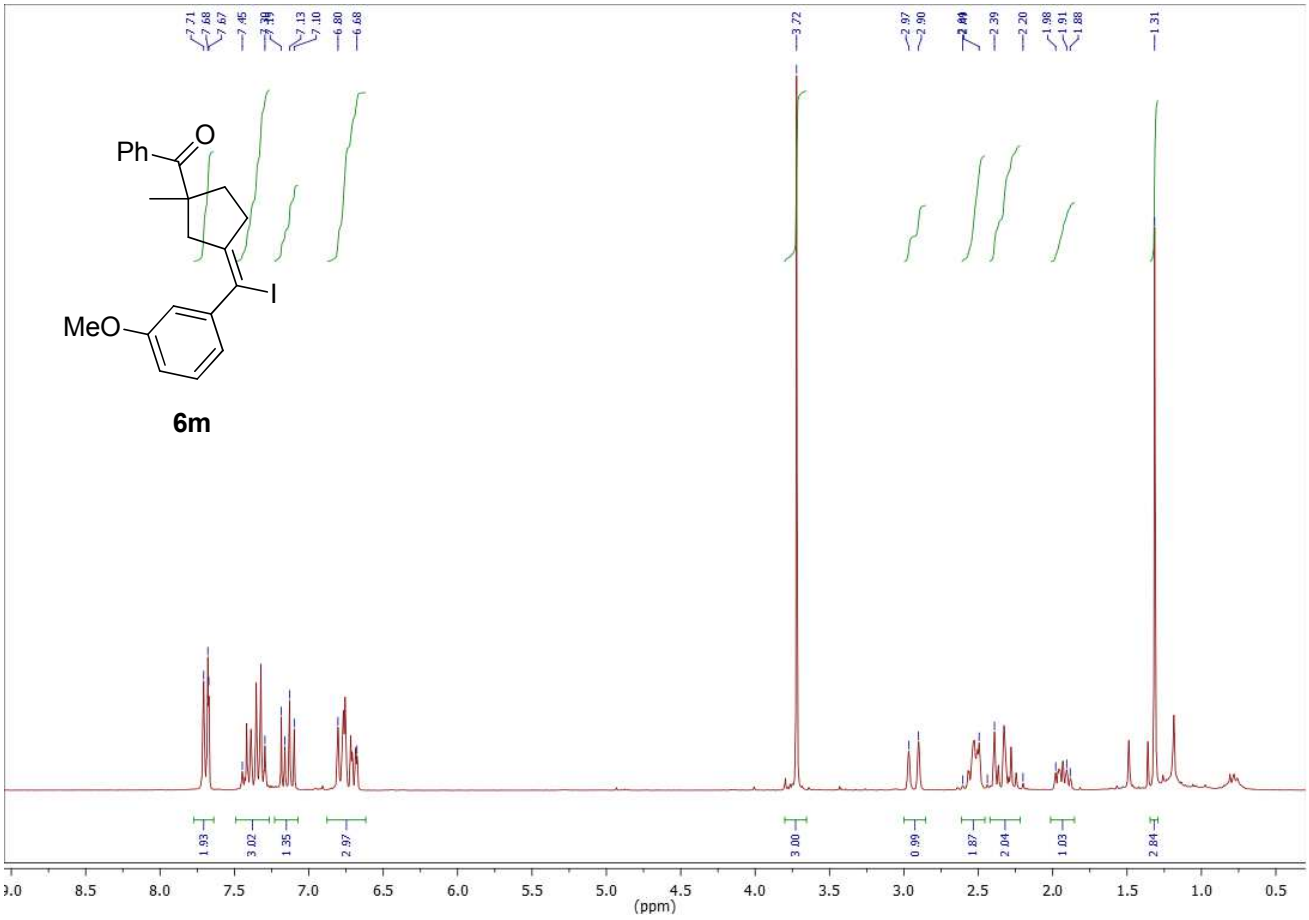

214.20
151.97
77.42
76.91
70.97
56.50
47.97
41.42
38.13
25.92
23.62
22.93
230
220
210
200
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
(ppm)

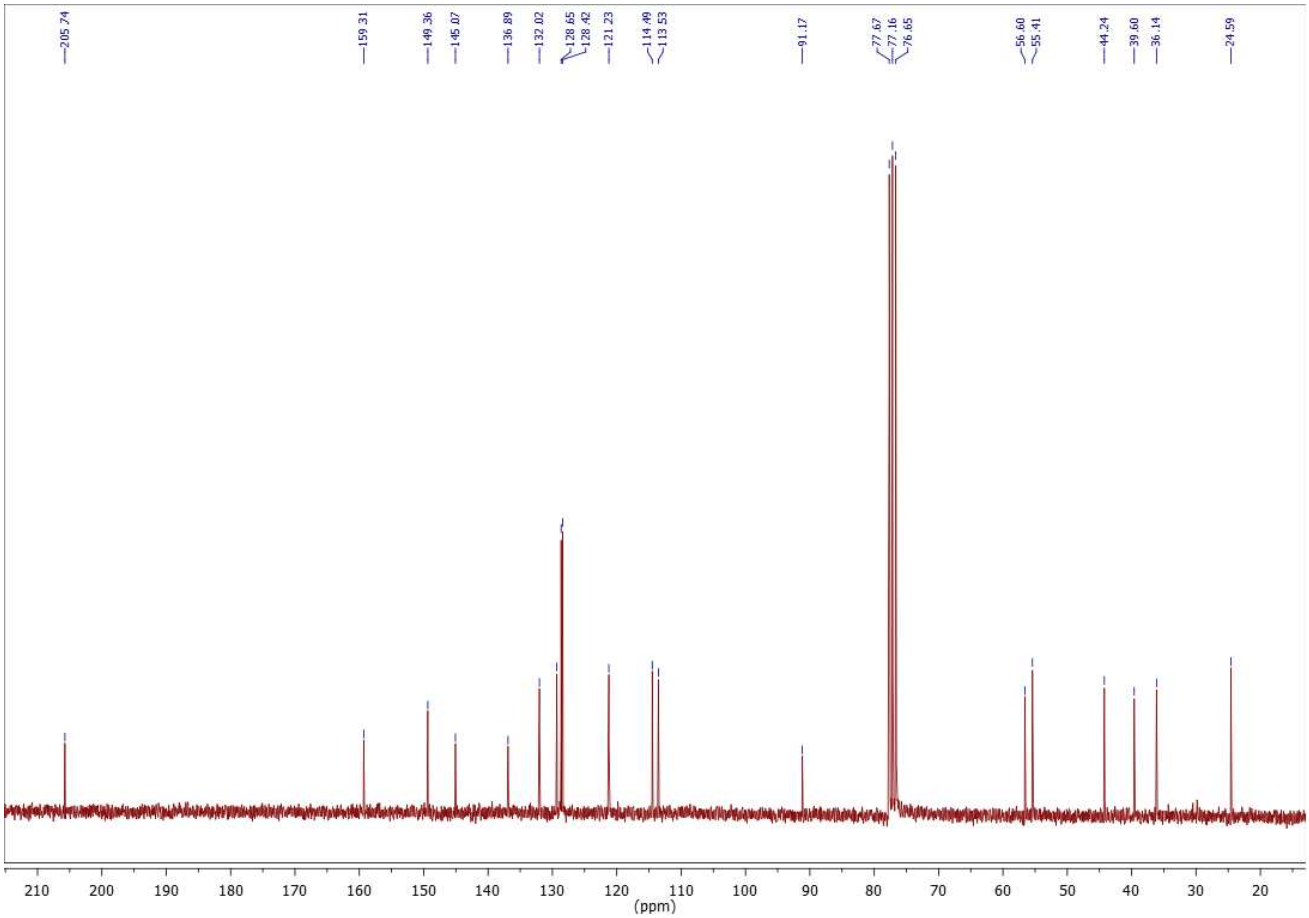

O
H
I
6k
7.36 7.35 7.35 7.32 7.31 7.26 7.25 7.25 7.24 7.24 7.24 7.23 7.23
3.12 3.12 3.09 3.09 2.70 2.67 2.66 2.64 2.49 2.47 2.39 2.38 2.33 2.00 1.92 1.90 1.88 1.74 1.73 1.72 1.71 1.19
4.15 0.99 0.99 1.11 1.19 3.13 3.03 1.19 1.09 2.97
7.6 7.4 7.2 7.0 6.8 6.6 6.4 6.2 6.0 5.8 5.6 5.4 5.2 5.0 4.8 4.6 4.4 4.2 4.0 3.8 3.6 3.4 3.2 3.0 2.8 2.6 2.4 2.2 2.0 1.8 1.6 1.4 1.2 1.0
(ppm)

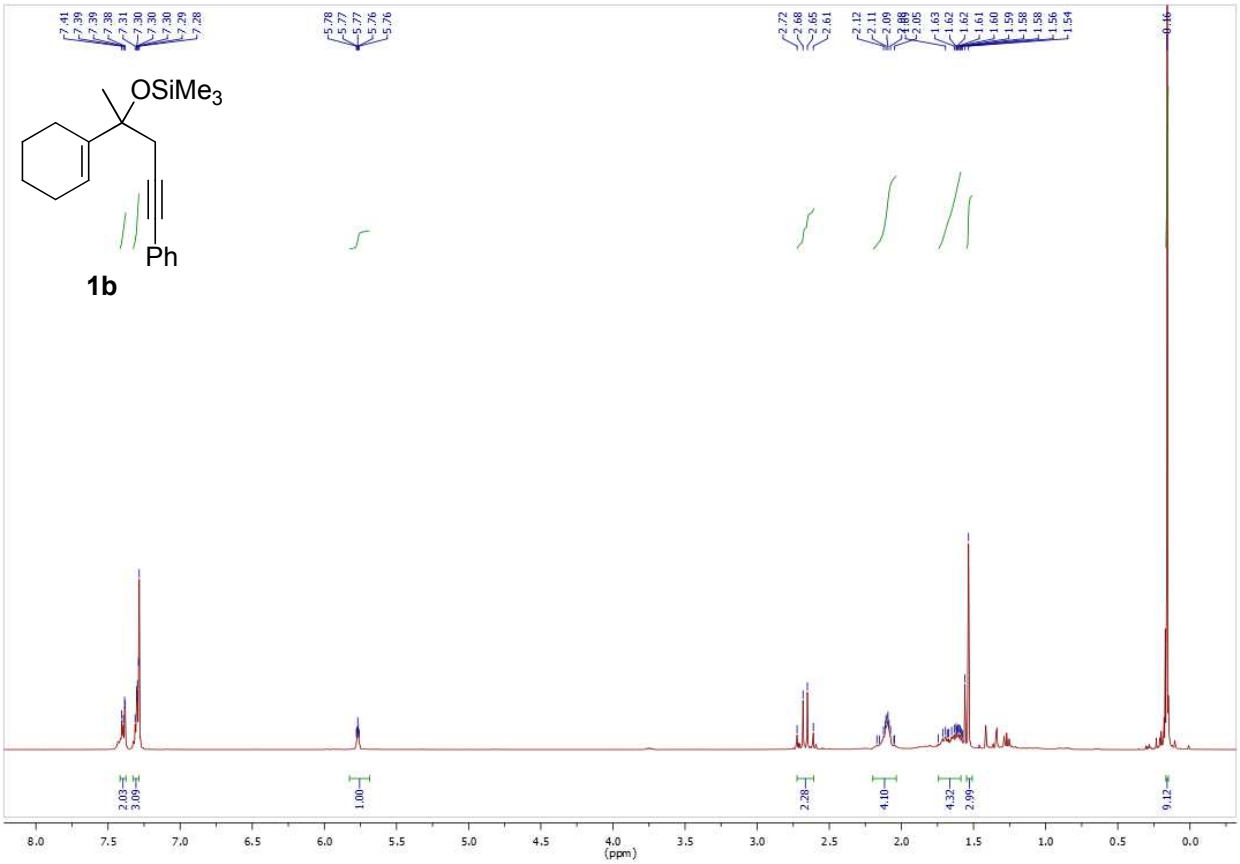

213.83
147.77
143.69
128.66
128.20
127.62
91.51
77.21
77.00
76.79
56.65
47.73
45.43
41.77
37.99
25.57
23.43
22.87
210 200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20
(ppm)

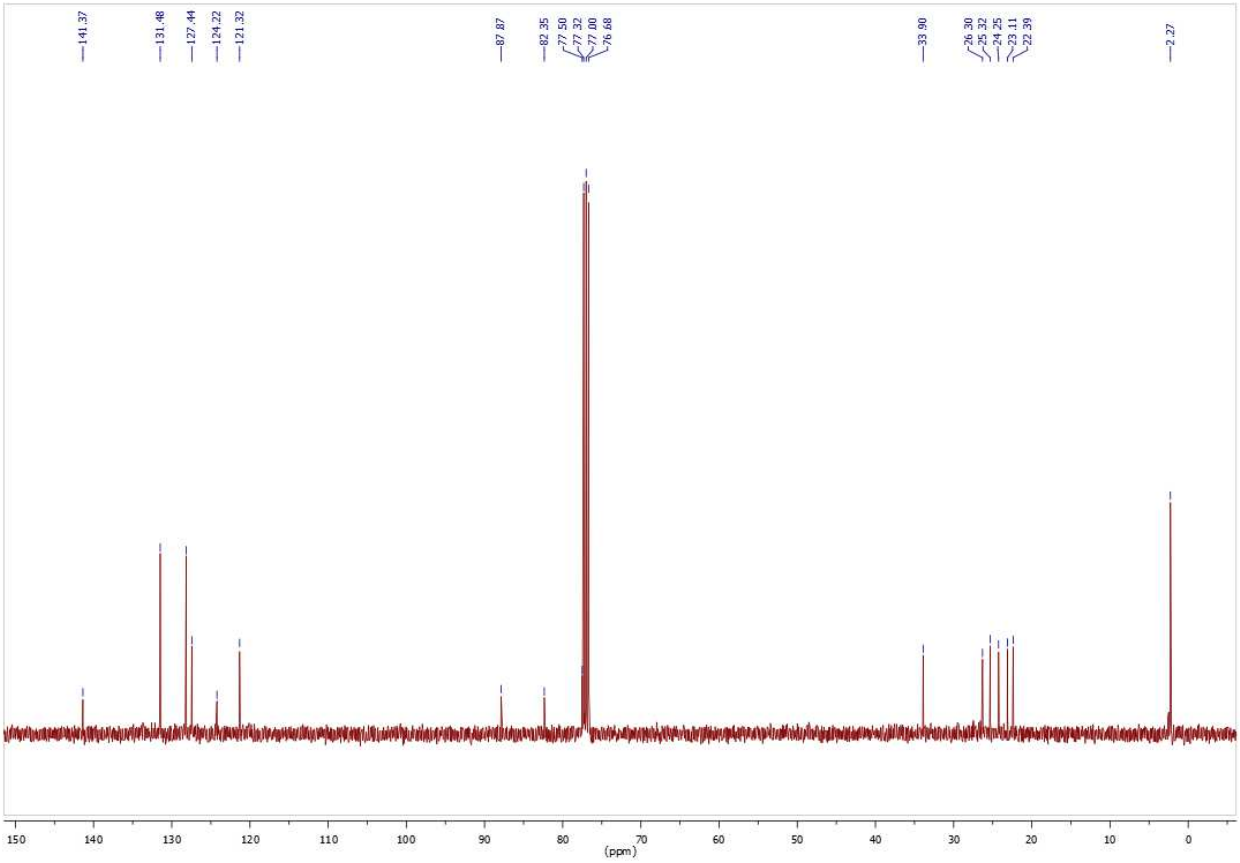

6l
(ppm)

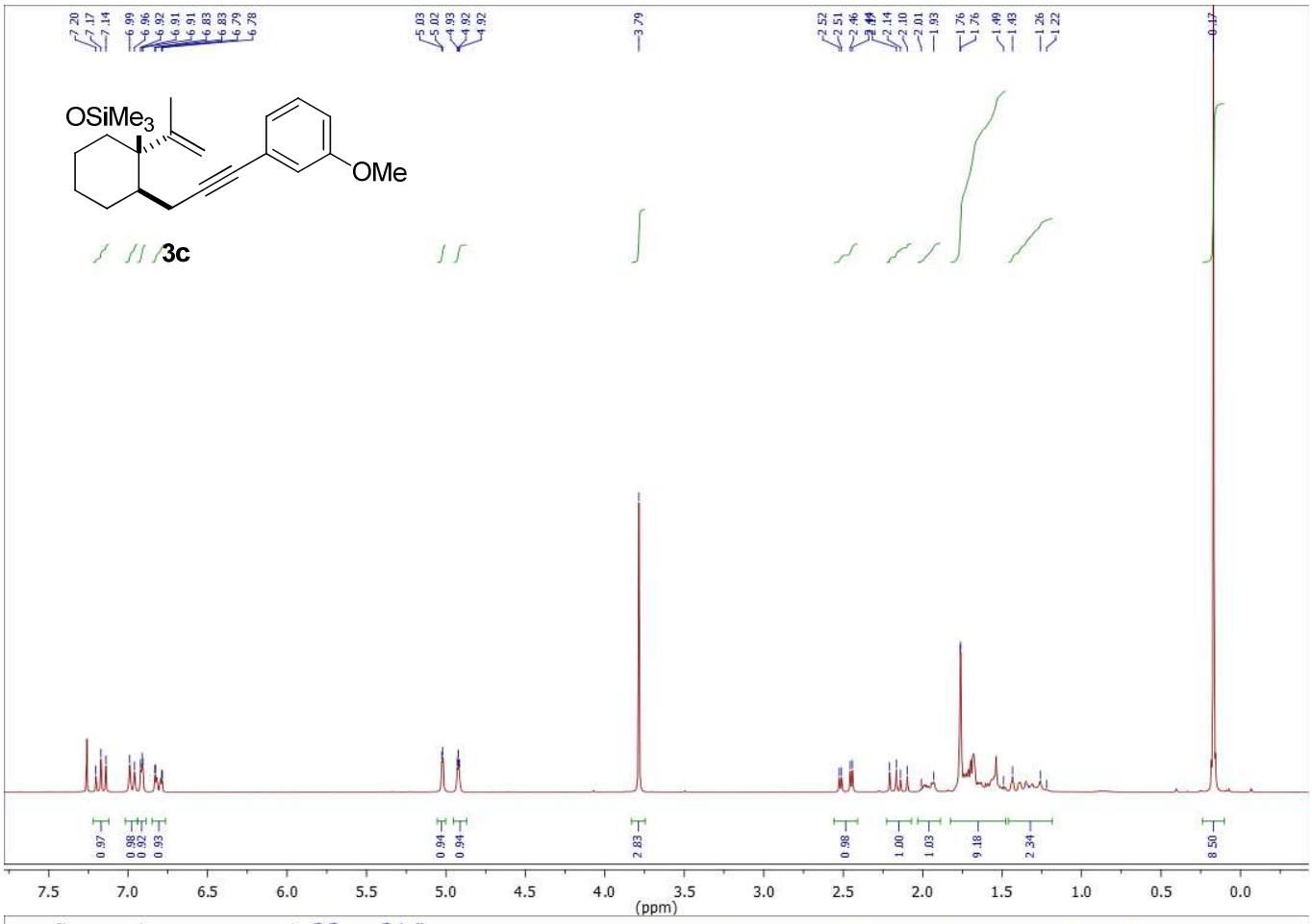

(ppm)

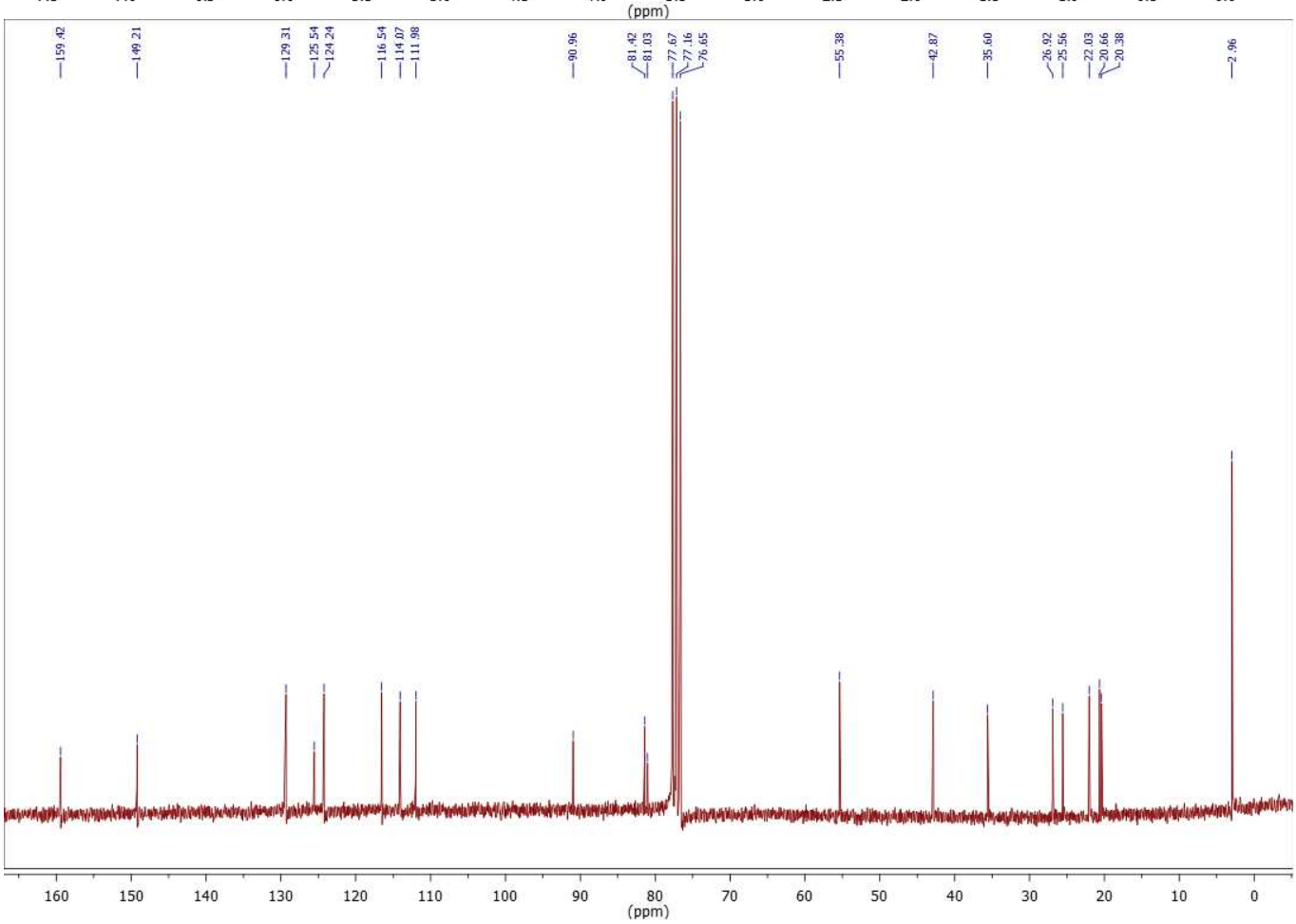

Ph
O
I
MeO
6m
7.71
7.68
7.67
7.45
7.13
7.10
6.80
6.68
3.72
2.97
2.90
2.39
2.20
1.98
1.91
1.88
1.31
1.93
3.02
1.35
2.97
3.00
0.99
1.87
2.04
1.03
2.84
8.5
8.0
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
0.5
(ppm)

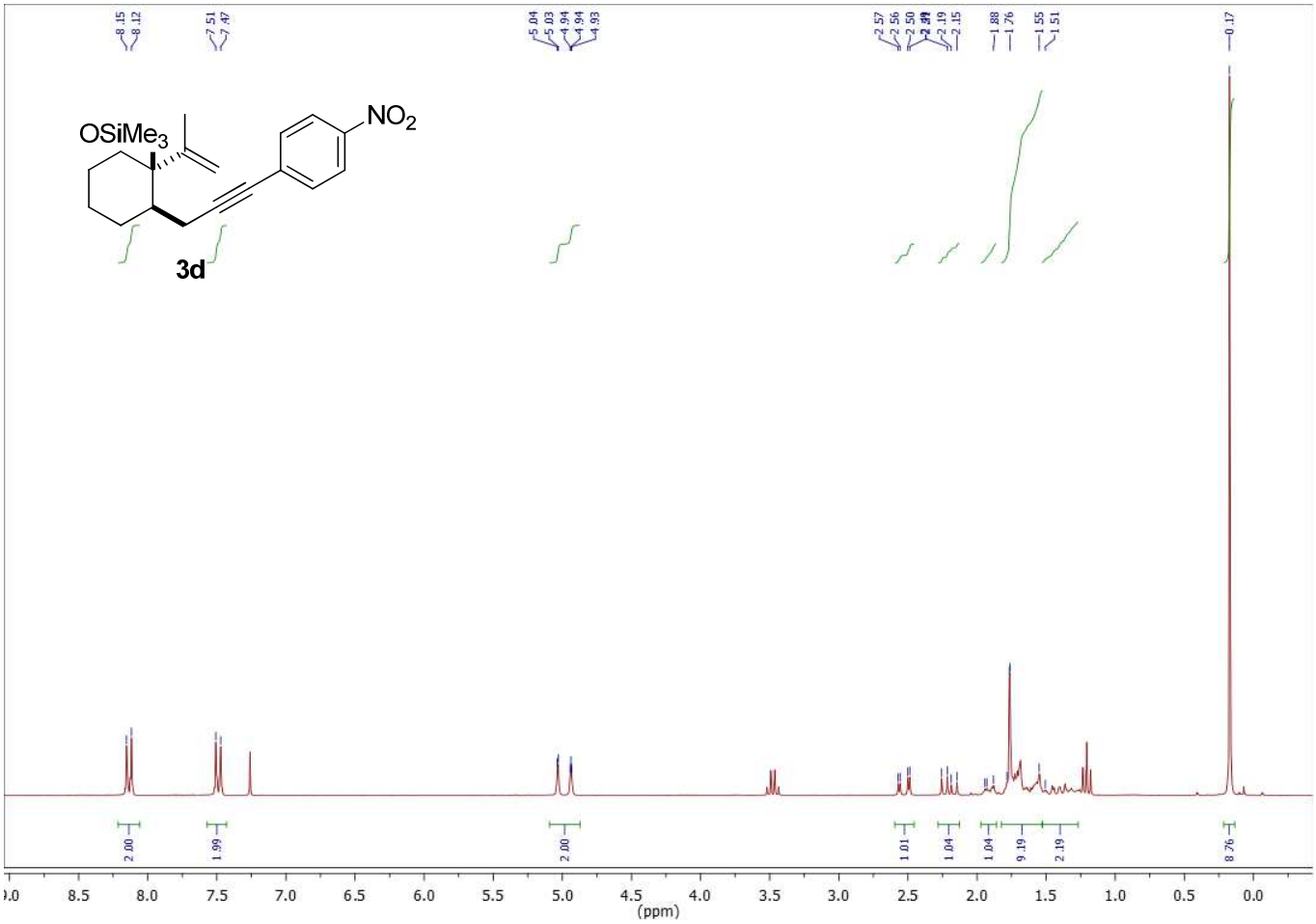

205.74
159.31
149.36
145.07
136.89
132.02
128.65
128.42
121.23
114.49
113.53
91.17
77.67
77.16
76.65
56.60
55.41
44.24
39.60
36.14
24.59
210
200
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
(ppm)

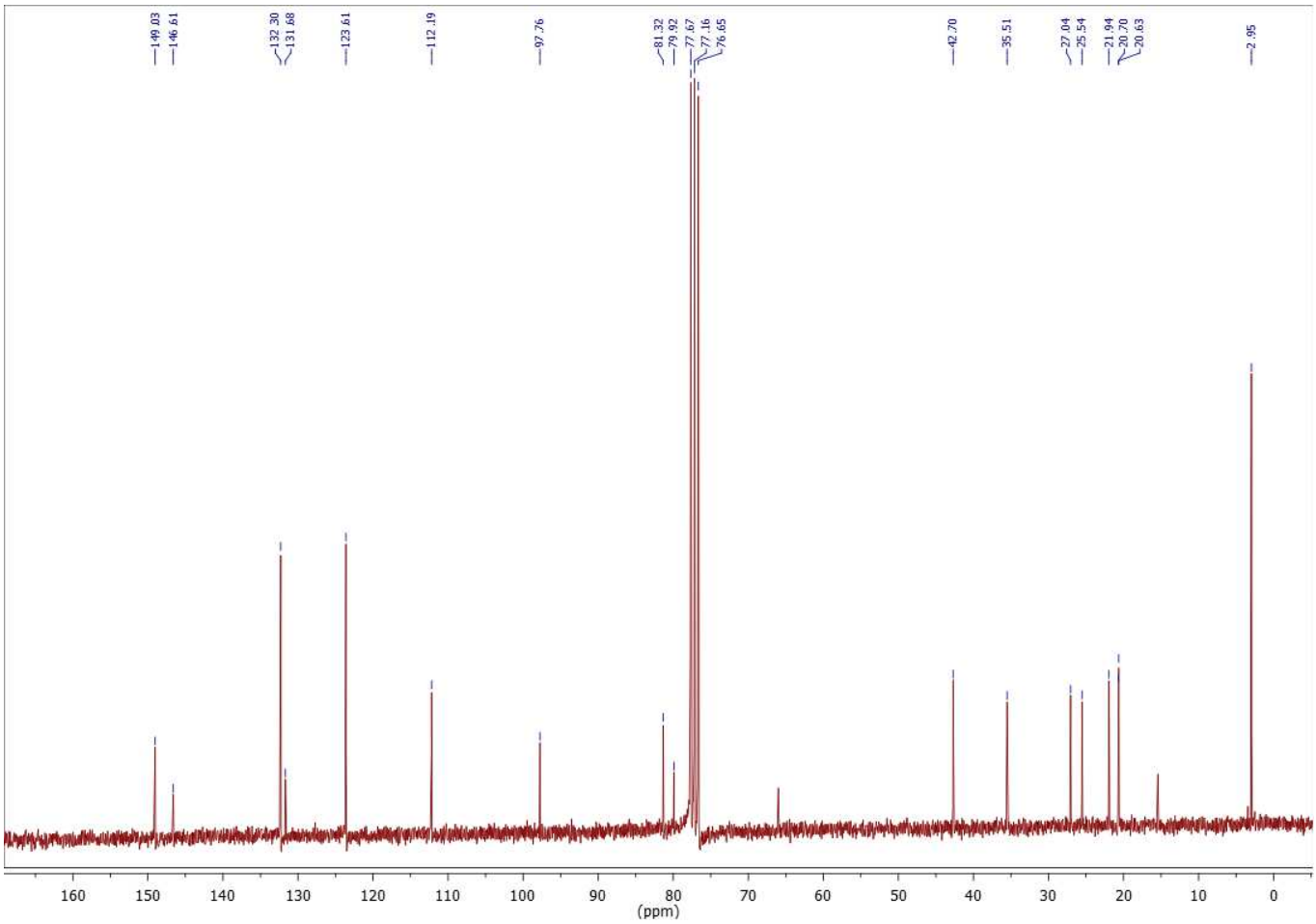

OSiMe3
Ph
1b
7.41 7.39 7.39 7.38 7.31 7.30 7.30 7.30 7.29 7.28
5.78 5.77 5.77 5.76 5.76
2.72 2.68 2.65 2.61
2.12 2.11 2.09 2.05
1.63 1.62 1.62 1.61 1.60 1.59 1.58 1.58 1.56 1.54
0.16
2.03 3.09 1.00 2.28 4.10 4.32 2.99 9.12
8.0 7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 0.5 0.0
(ppm)

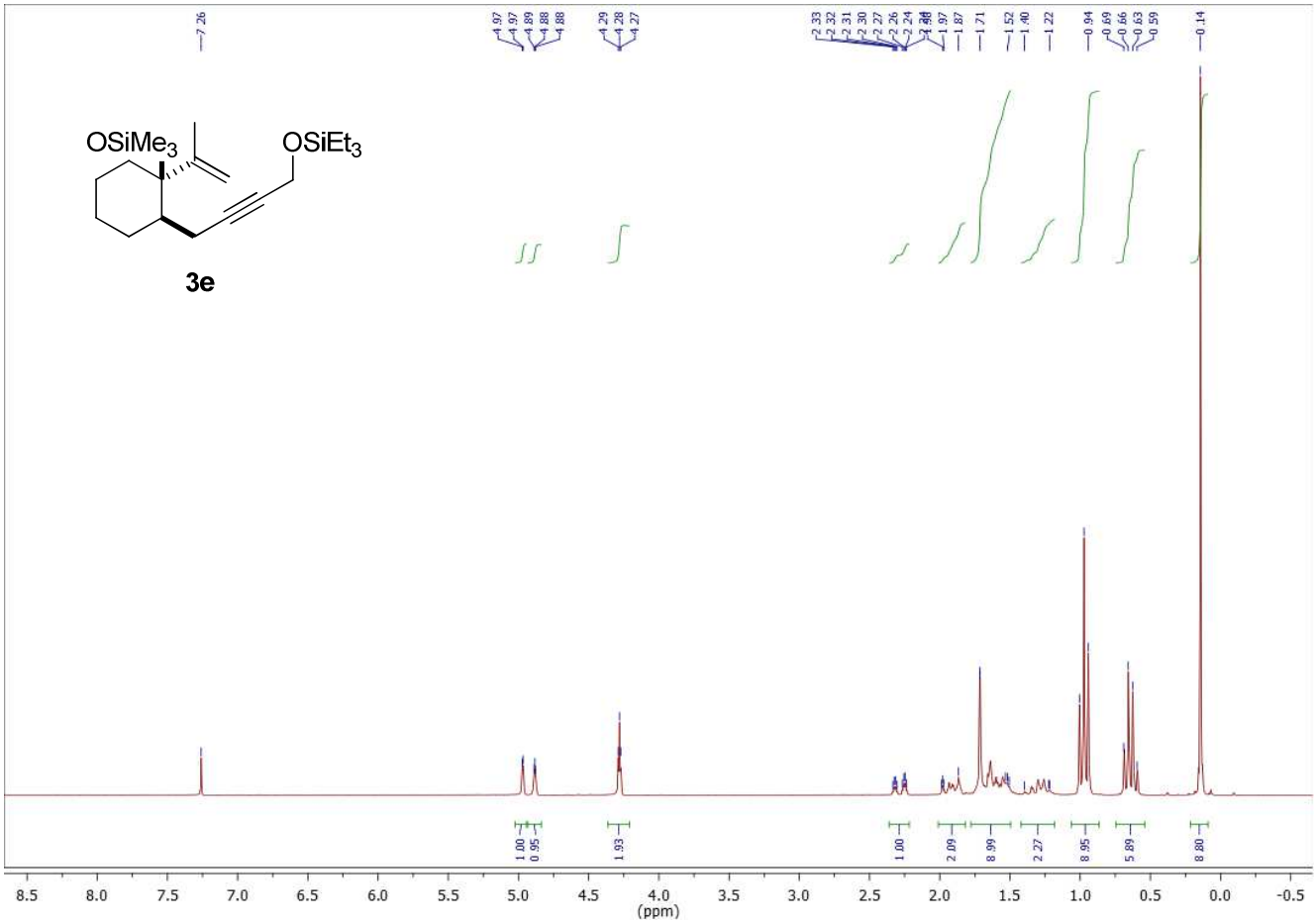

141.37
131.48
127.44
124.22
121.32
87.87
82.35
77.50
77.32
77.00
76.68
33.90
26.30
25.32
24.25
23.11
22.39
2.27
150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0
(ppm)

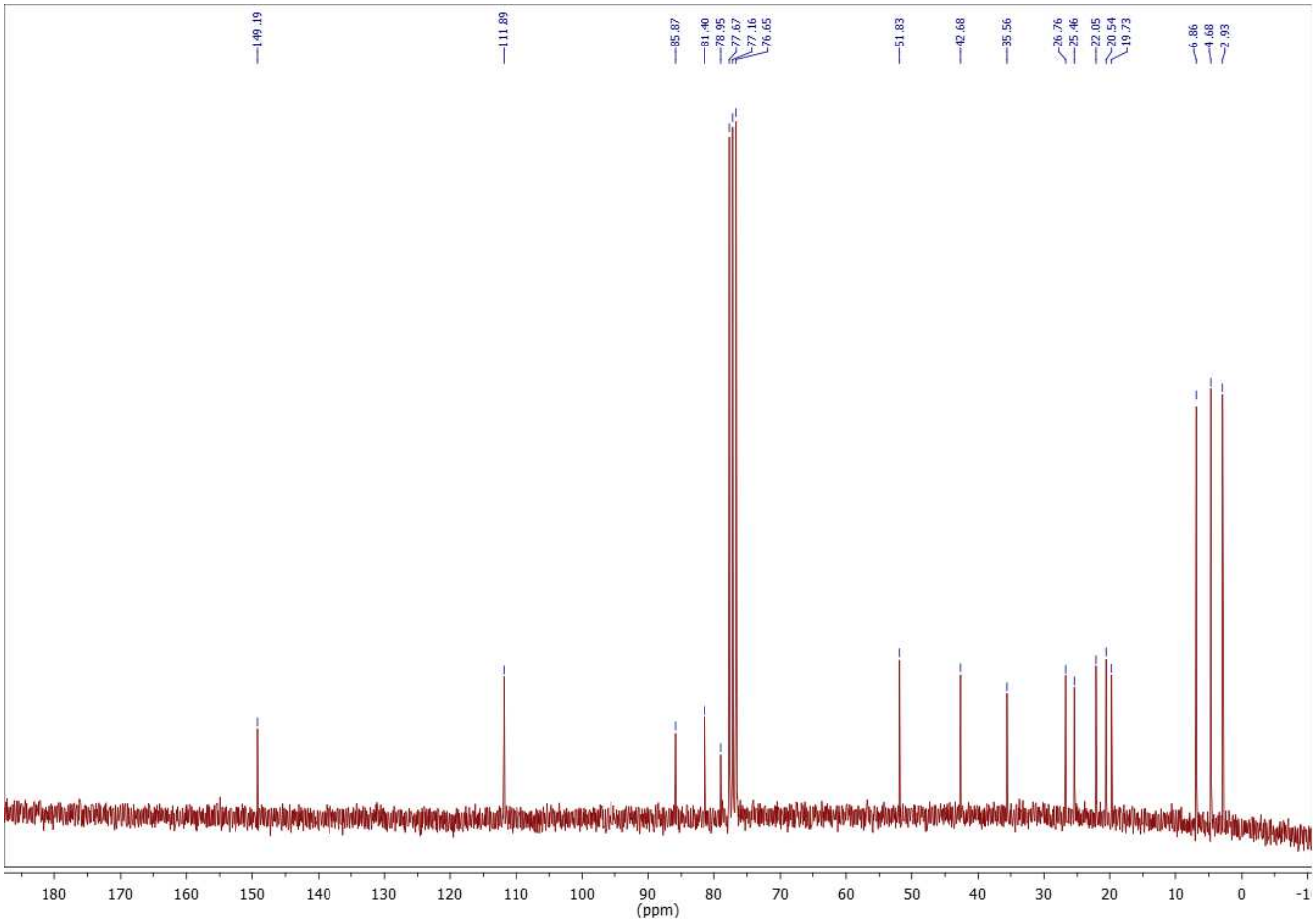

OSiMe3
OMe
3c
7.20
7.17
7.14
6.99
6.96
6.92
6.91
6.91
6.83
6.83
6.79
6.78
5.03
5.02
4.93
4.92
4.92
3.79
2.52
2.51
2.46
2.14
2.10
2.01
1.93
1.76
1.76
1.49
1.48
1.26
1.22
0.17
0.97
0.98
0.92
0.93
0.94
0.94
2.83
0.98
1.00
1.03
9.18
2.34
8.50
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
0.5
0.0
(ppm)

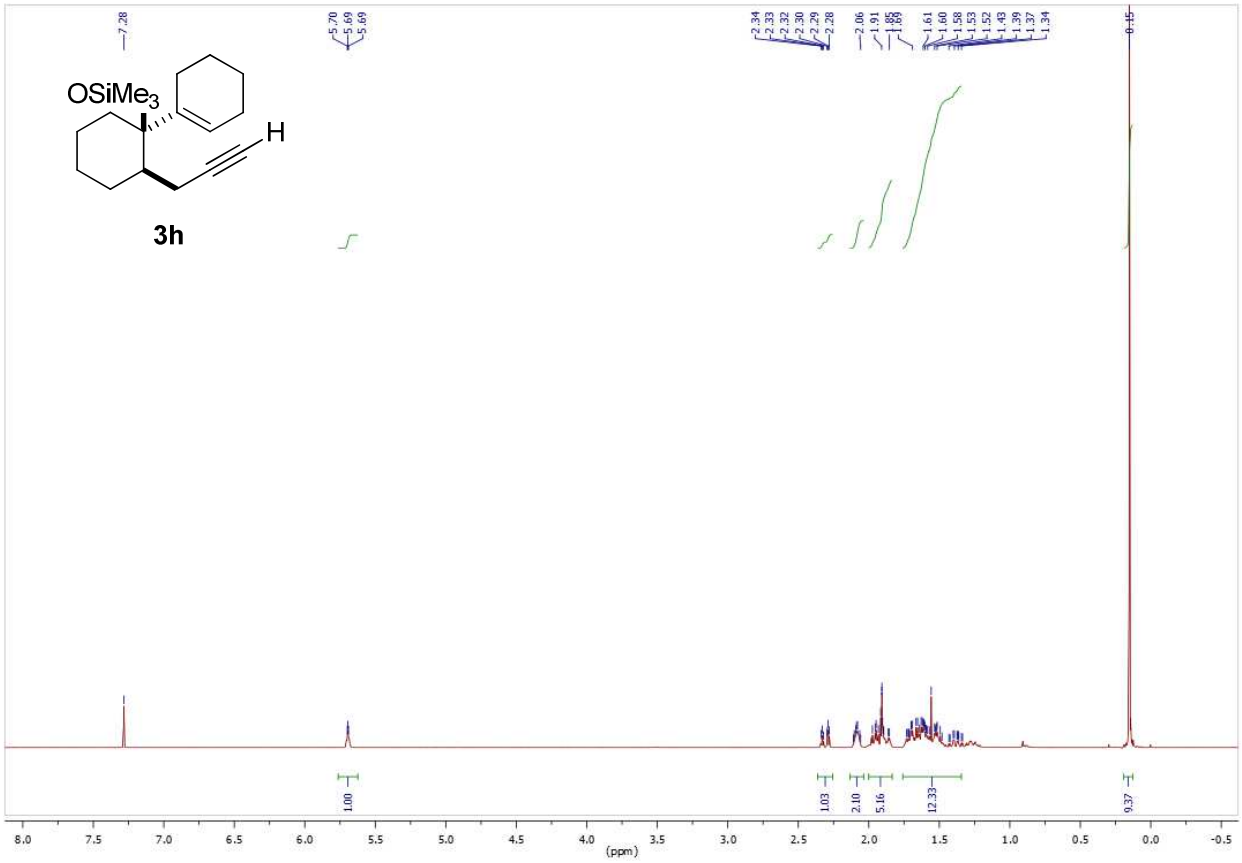

159.42
149.21
129.31
125.54
124.24
116.54
114.07
111.98
90.96
81.42
81.03
77.67
77.16
76.65
55.38
42.87
35.60
26.92
25.56
22.03
20.66
20.38
2.96
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
(ppm)

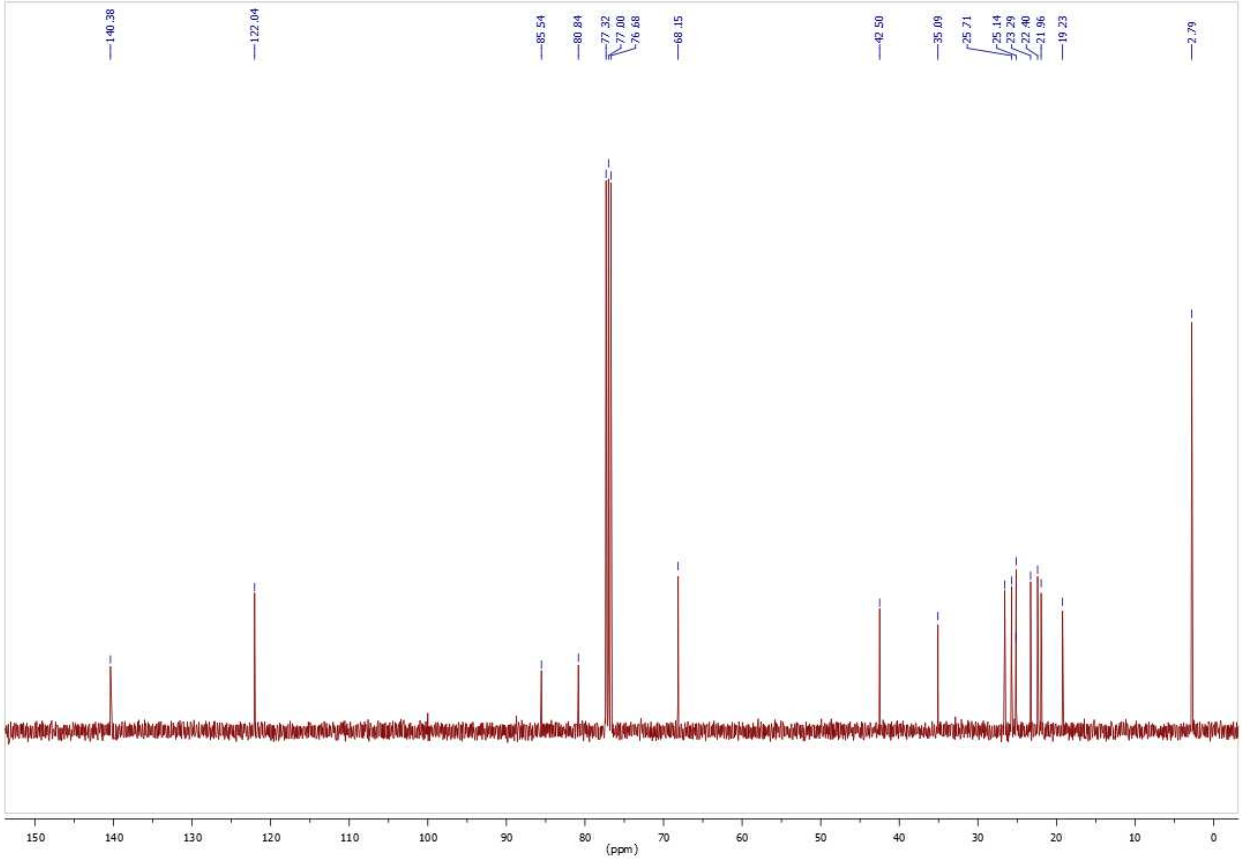

OSiMe3
NO2
3d
8.15
8.12
7.51
7.47
5.04
5.03
4.94
4.94
4.93
2.57
2.56
2.50
2.19
2.15
1.88
1.76
1.55
1.51
0.17
2.00
1.99
2.00
1.01
1.04
1.04
9.19
2.19
8.76
(ppm)

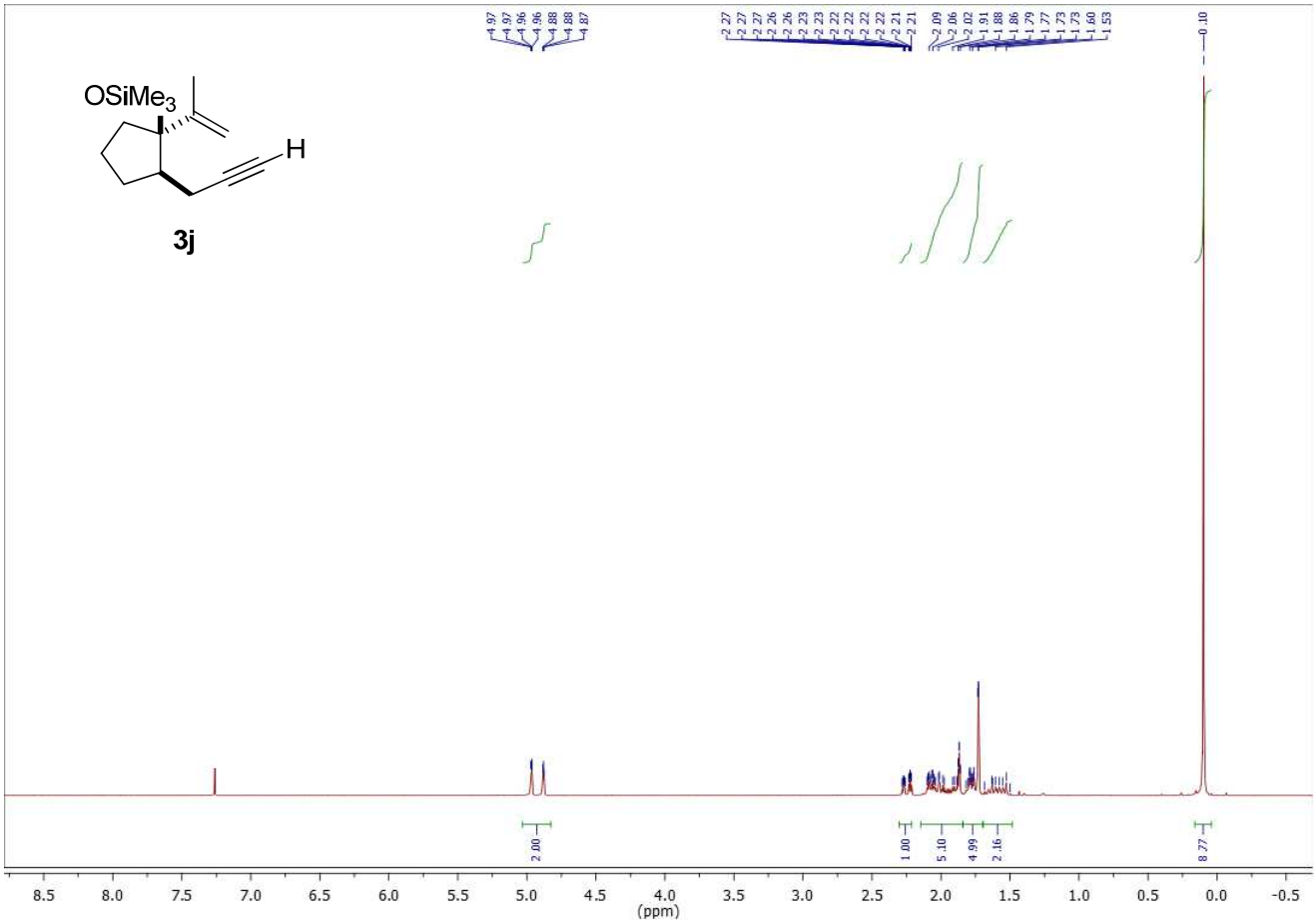

149.03
146.61
132.30
131.68
123.61
112.19
97.76
81.32
79.92
77.67
77.16
76.65
42.70
35.51
27.04
25.54
21.94
20.70
20.63
2.95
(ppm)

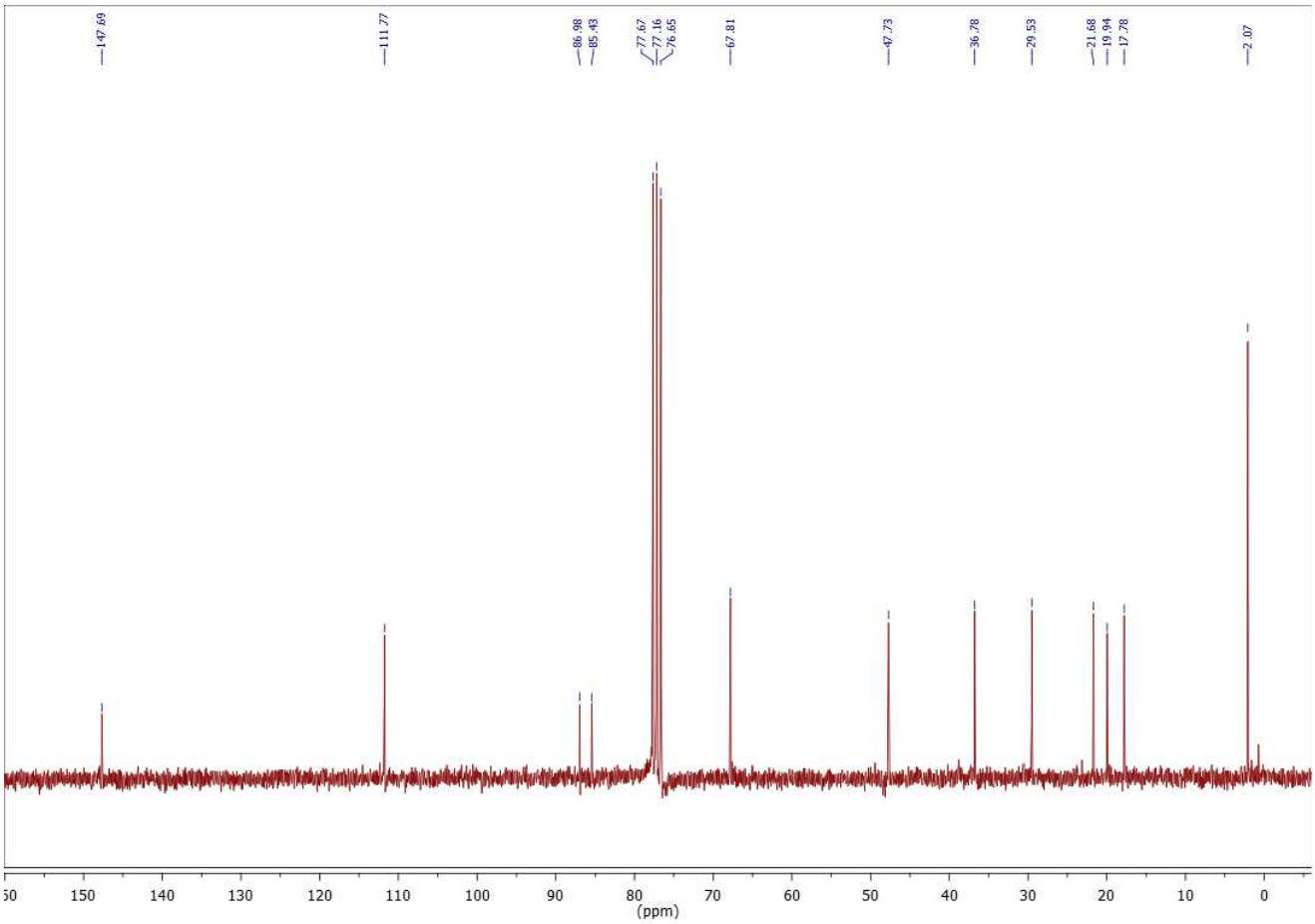

OSiMe3
OSiEt3
3e
7.26
4.97
4.97
4.89
4.88
4.88
4.29
4.28
4.27
2.33
2.32
2.31
2.30
2.27
2.26
2.24
1.97
1.87
1.71
1.52
1.40
1.22
0.94
0.69
0.66
0.63
0.59
0.14
1.00
0.95
1.93
1.00
2.09
8.99
2.27
8.95
5.89
8.80
8.5
8.0
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
0.5
0.0
-0.5
(ppm)

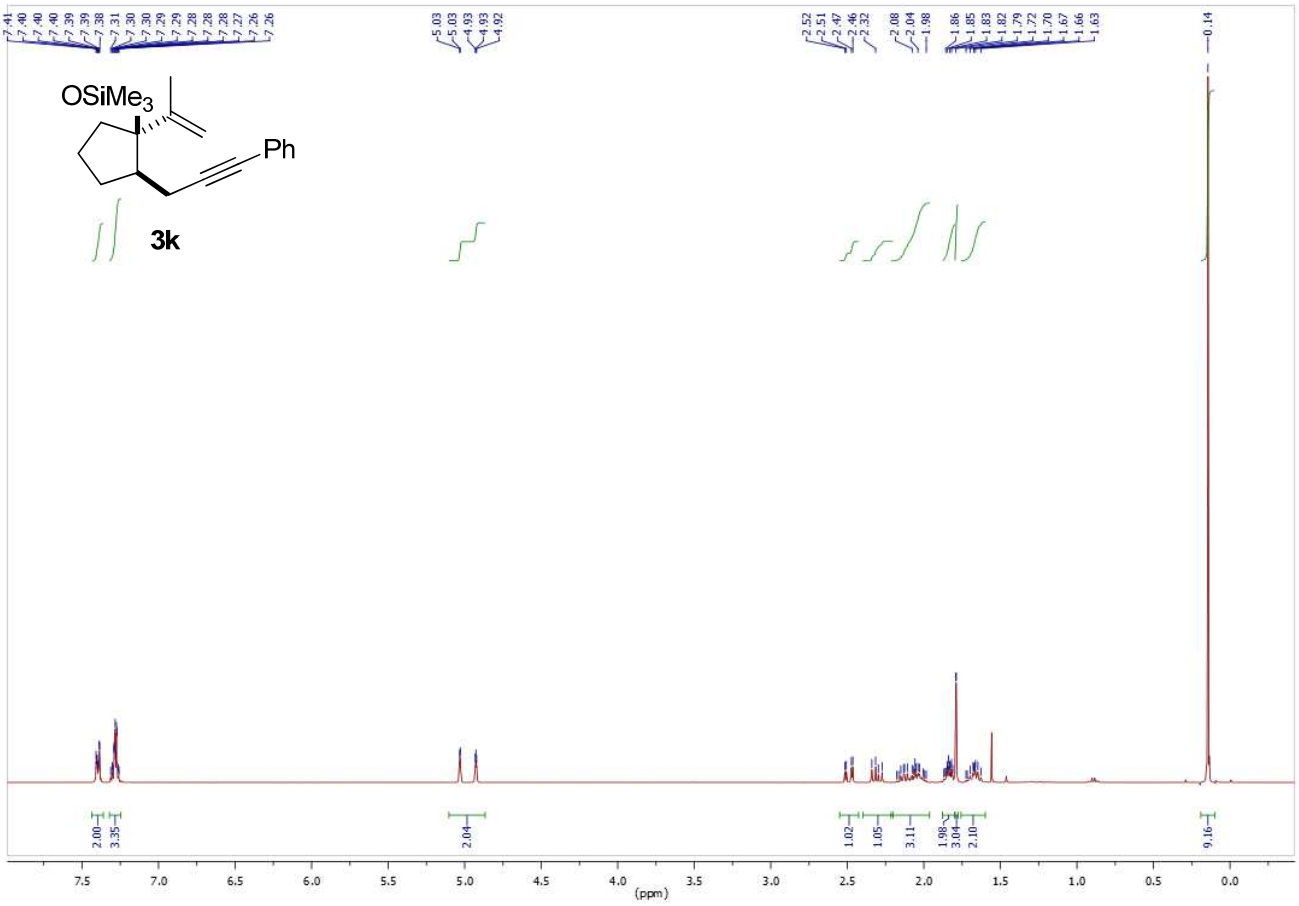

149.19
111.89
85.87
81.40
78.95
77.67
77.16
76.65
51.83
42.68
35.56
26.76
25.46
22.05
20.54
19.73
6.86
4.68
2.93
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
-1
(ppm)

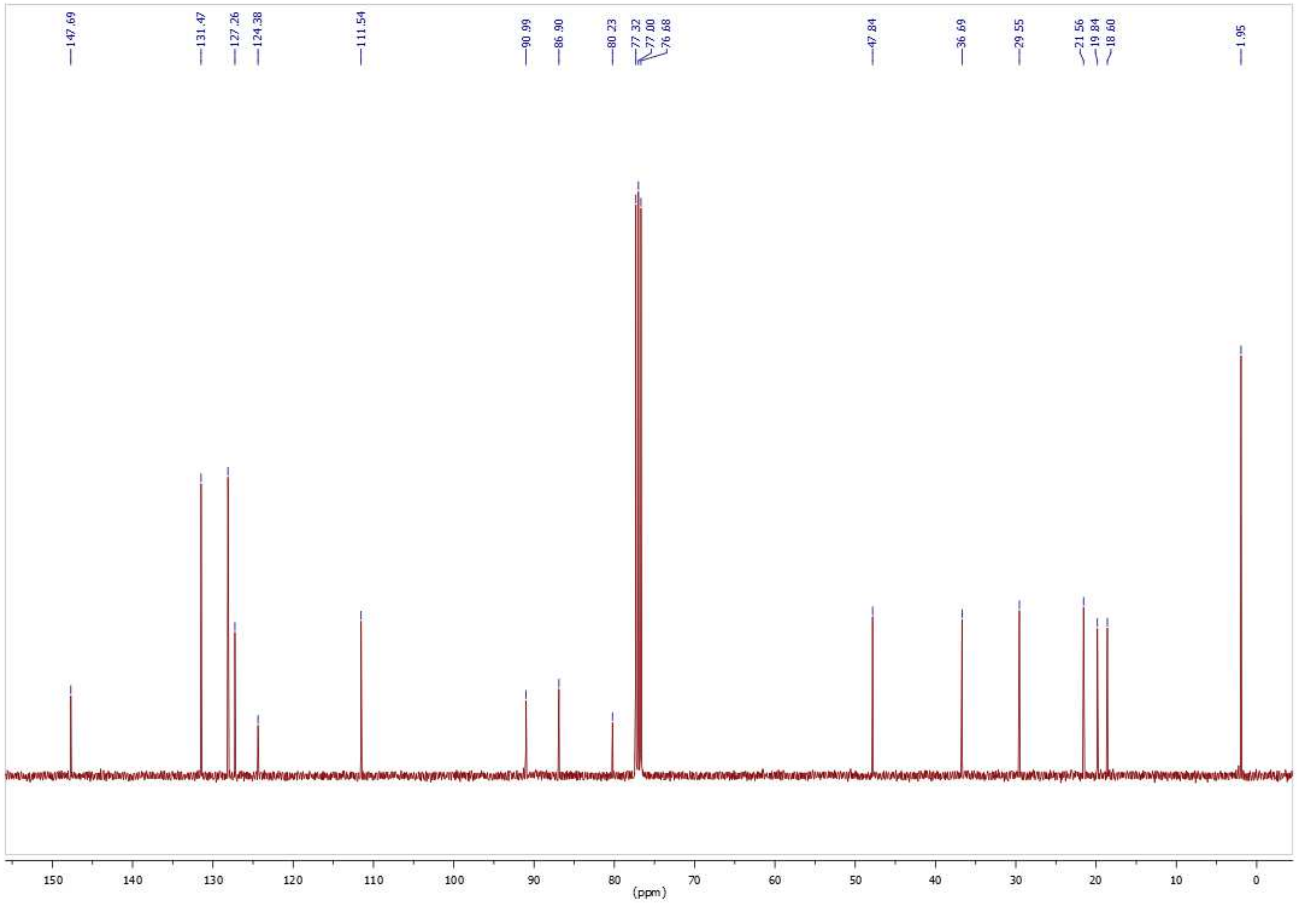

OSiMe3
H
3h
7.28
5.70
5.69
5.69
2.34
2.33
2.32
2.30
2.29
2.28
2.06
1.91
1.85
1.83
1.61
1.60
1.58
1.53
1.52
1.43
1.39
1.37
1.34
0.15
1.00
1.03
2.10
5.16
12.33
9.37
(ppm)

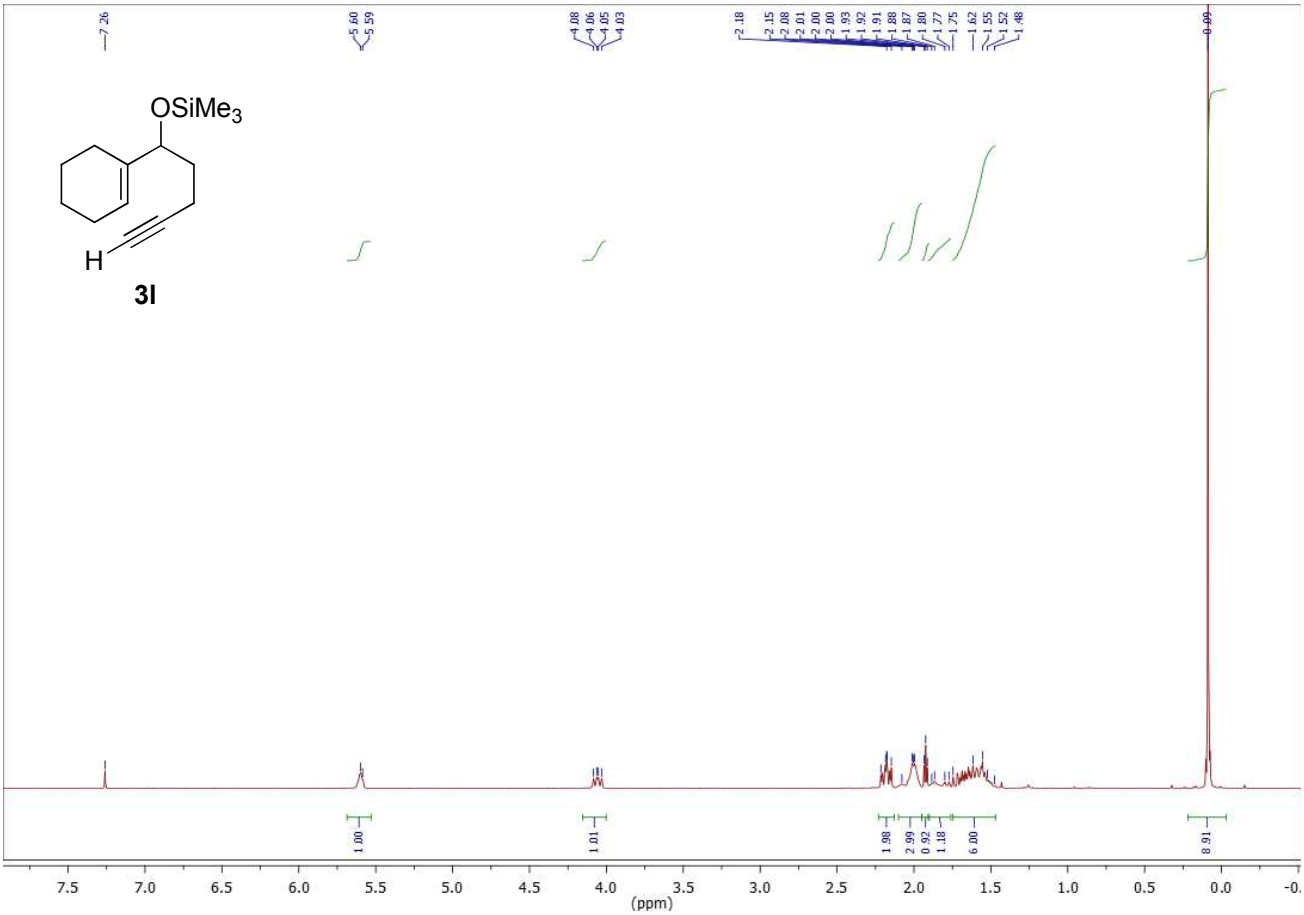

140.38
122.04
85.54
80.84
77.32
77.00
76.68
68.15
42.50
35.09
25.71
25.14
23.29
22.40
21.96
19.23
2.79
(ppm)

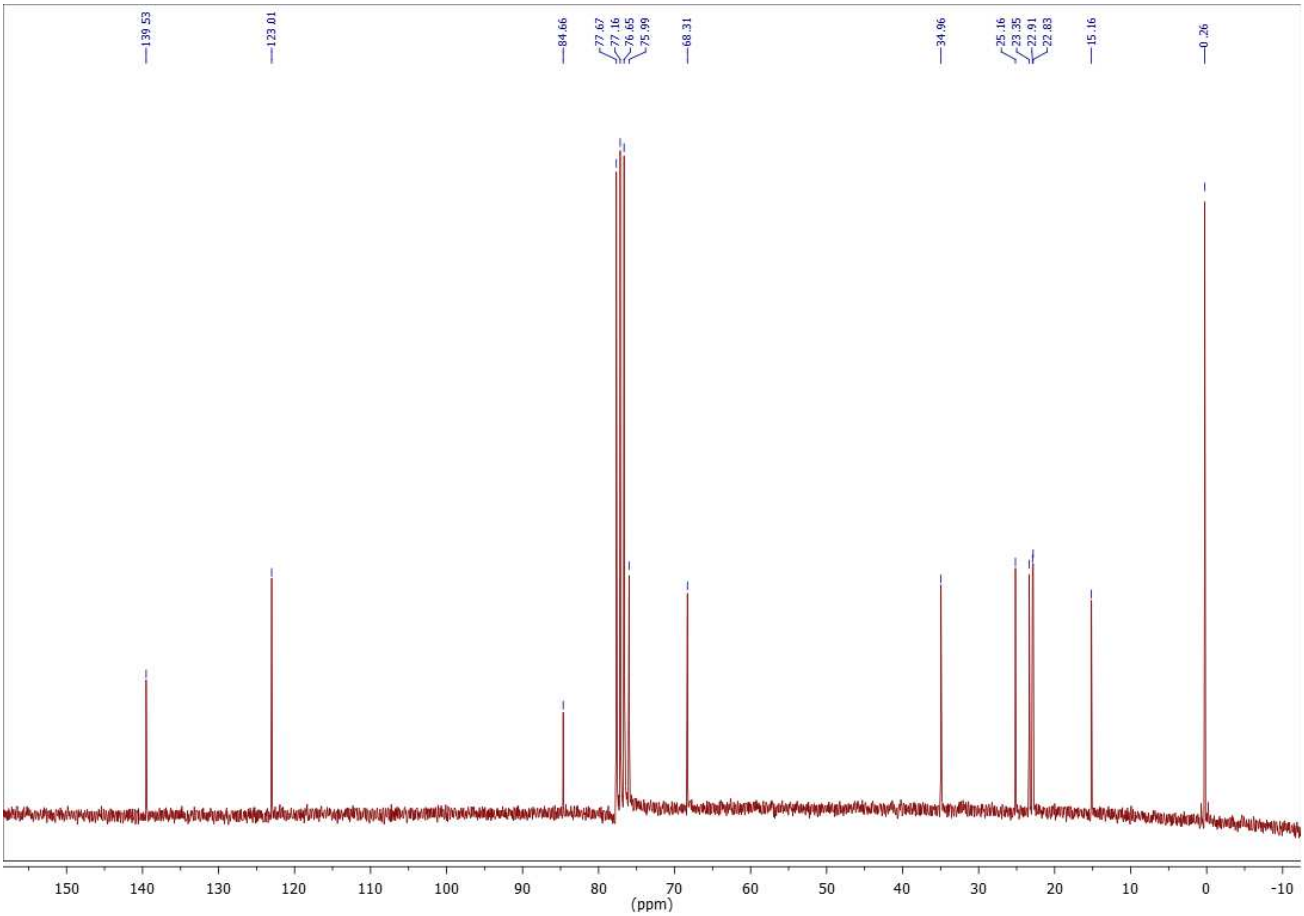

OSiMe3
H
3j
(ppm)

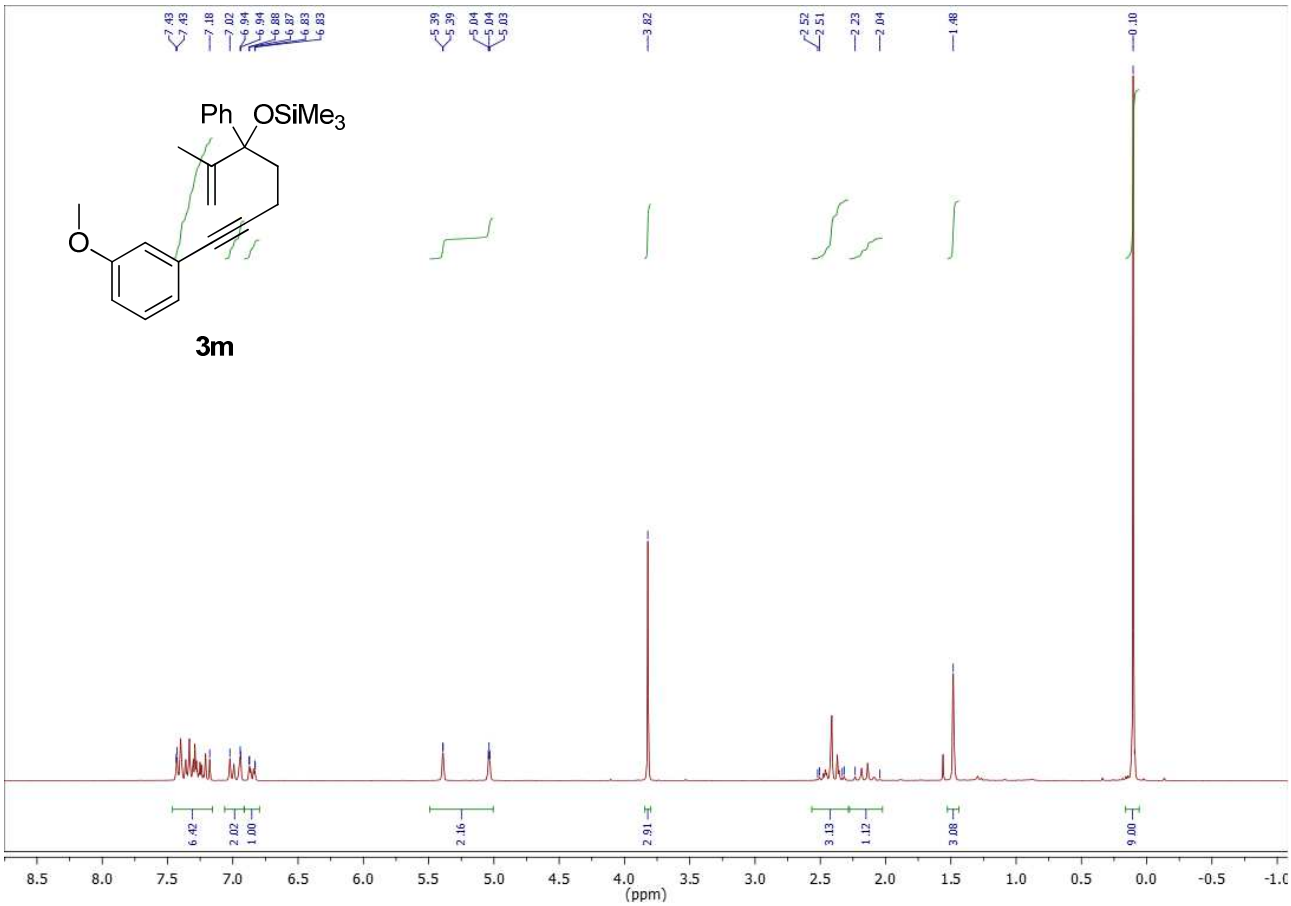

(ppm)

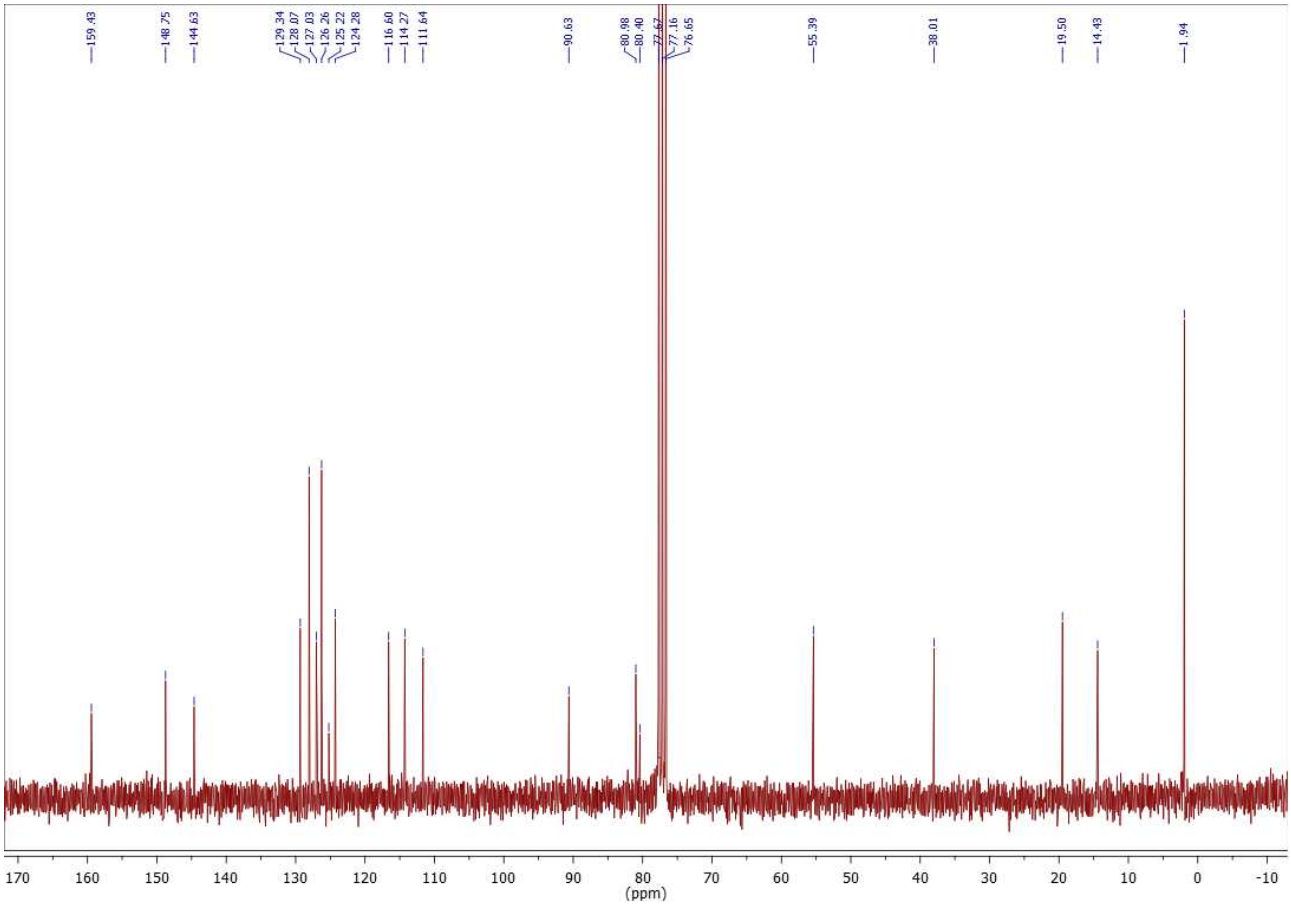

OSiMe3
Ph
3k
7.41 7.40 7.40 7.40 7.39 7.38 7.31 7.30 7.30 7.29 7.29 7.28 7.28 7.28 7.27 7.26 7.26
5.03 5.03 4.93 4.93 4.92
2.52 2.51 2.47 2.46 2.32 2.08 2.04 1.98 1.86 1.85 1.83 1.82 1.79 1.72 1.70 1.67 1.66 1.63
0.14
2.00 3.35 2.04 1.02 1.05 3.11 1.98 3.04 2.10 9.16
7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 0.5 0.0
(ppm)


147.69 131.47 127.26 124.38 111.54 90.99 86.90 80.23 77.32 77.00 76.68 47.84 36.69 29.55 21.56 19.84 18.60 1.95
150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0
(ppm)


OSiMe3
H
3l
7.26
5.60
5.59
4.08
4.06
4.05
4.03
2.18
2.15
2.08
2.01
2.00
2.00
1.93
1.92
1.91
1.88
1.87
1.80
1.77
1.75
1.62
1.55
1.52
1.48
0.09
1.00
1.01
1.98
2.99
0.92
1.18
6.00
8.91
(ppm)


139.53
123.01
84.66
77.67
77.16
76.65
75.99
68.31
34.96
25.16
23.35
22.91
22.83
15.16
0.26
(ppm)


Ph
OSiMe3
O
3m
7.43
7.43
7.18
7.02
6.94
6.88
6.87
6.83
6.83
5.39
5.39
5.04
5.04
5.03
3.82
2.52
2.51
2.23
2.04
1.48
0.10
6.42
2.02
1.00
2.16
2.91
3.13
1.12
3.08
9.00
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
-0.5
(ppm)


159.43
148.75
144.63
129.34
128.07
127.03
126.26
125.22
124.28
116.60
114.27
111.64
90.63
80.98
80.40
77.67
77.16
76.65
55.39
38.01
19.50
14.43
1.94
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
-10
(ppm)