Electronic Supplementary Material (ESI) for RSC Advances.

Supporting Information

Synthesis of (–) Cephalosporolide E and (+) Cephalosporolide F Utilizing the Vinylogous Mukaiyama type Reaction with an α-Chloro Sulfide

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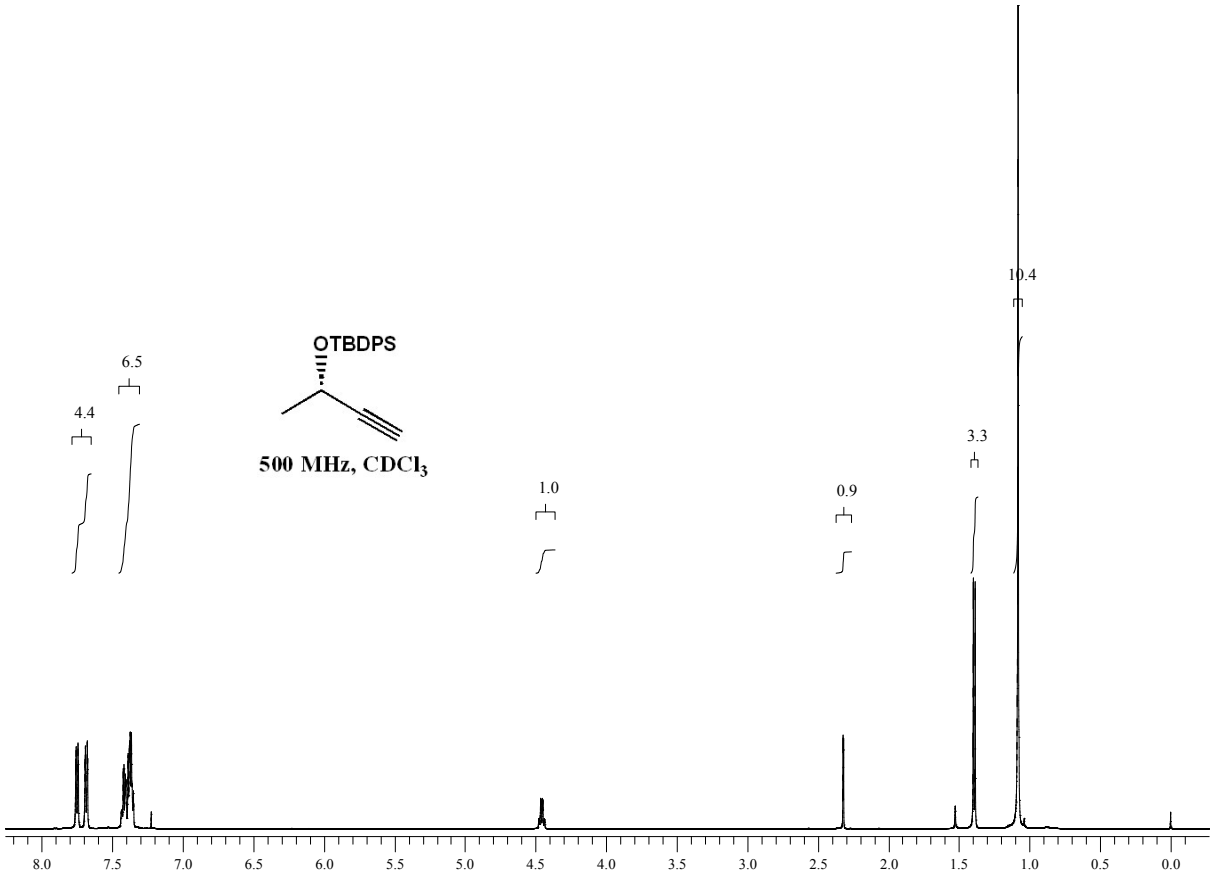
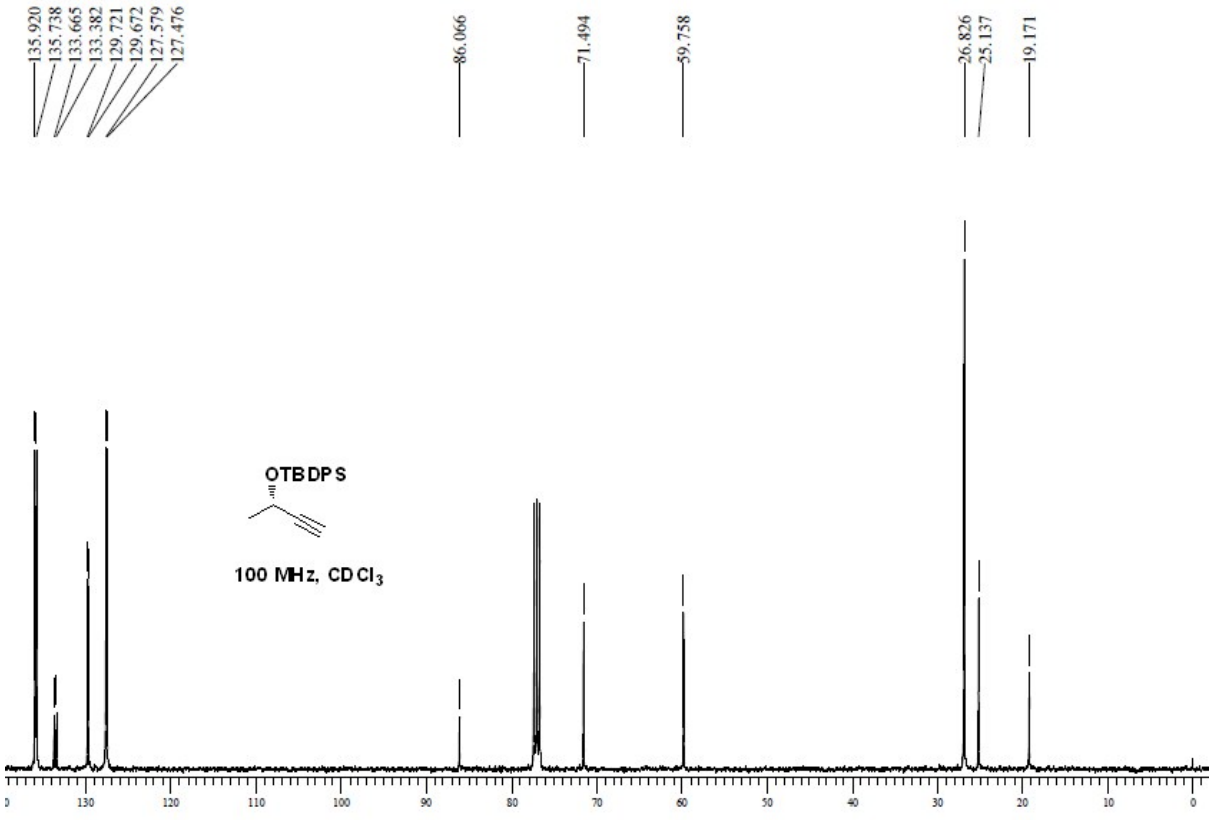
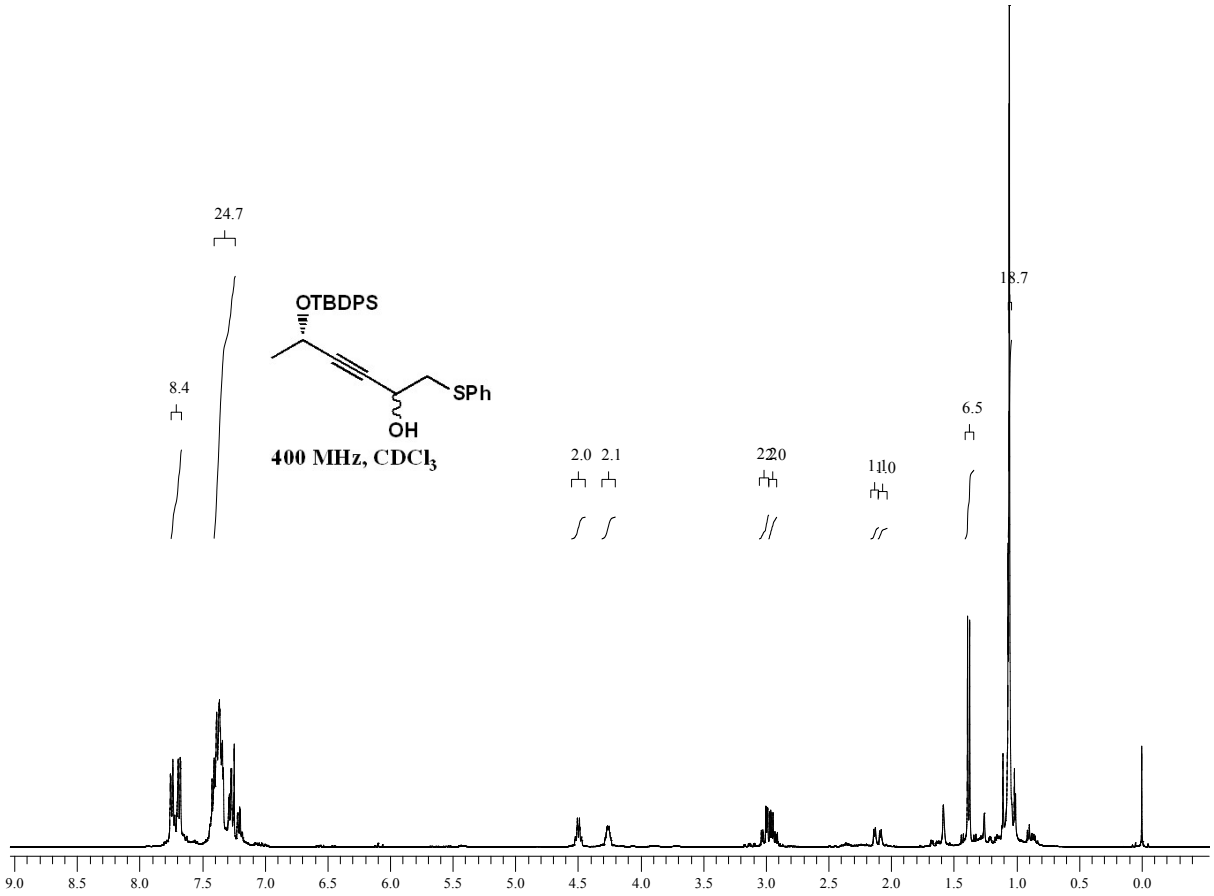
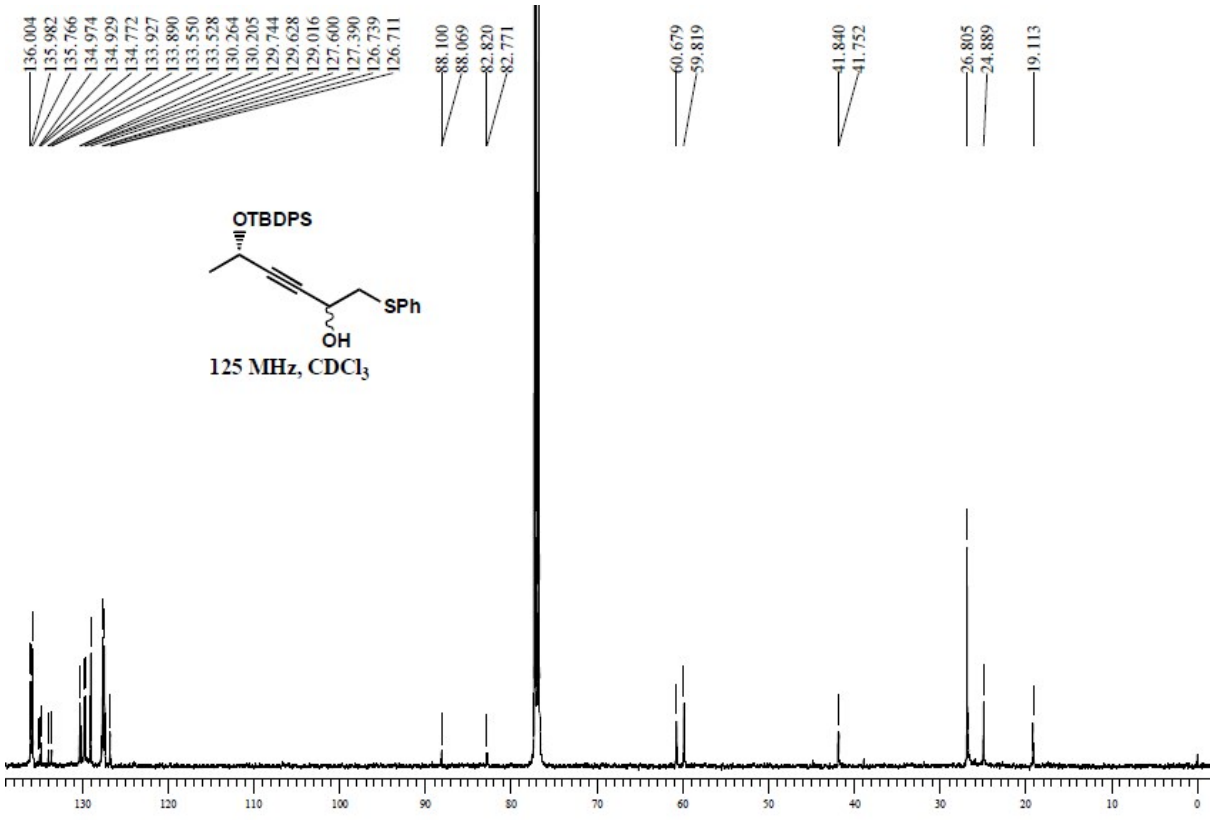
Compund 10:


OTBDPS
500 MHz, CDCl3
4.4
6.5
1.0
0.9
3.3
10.4
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

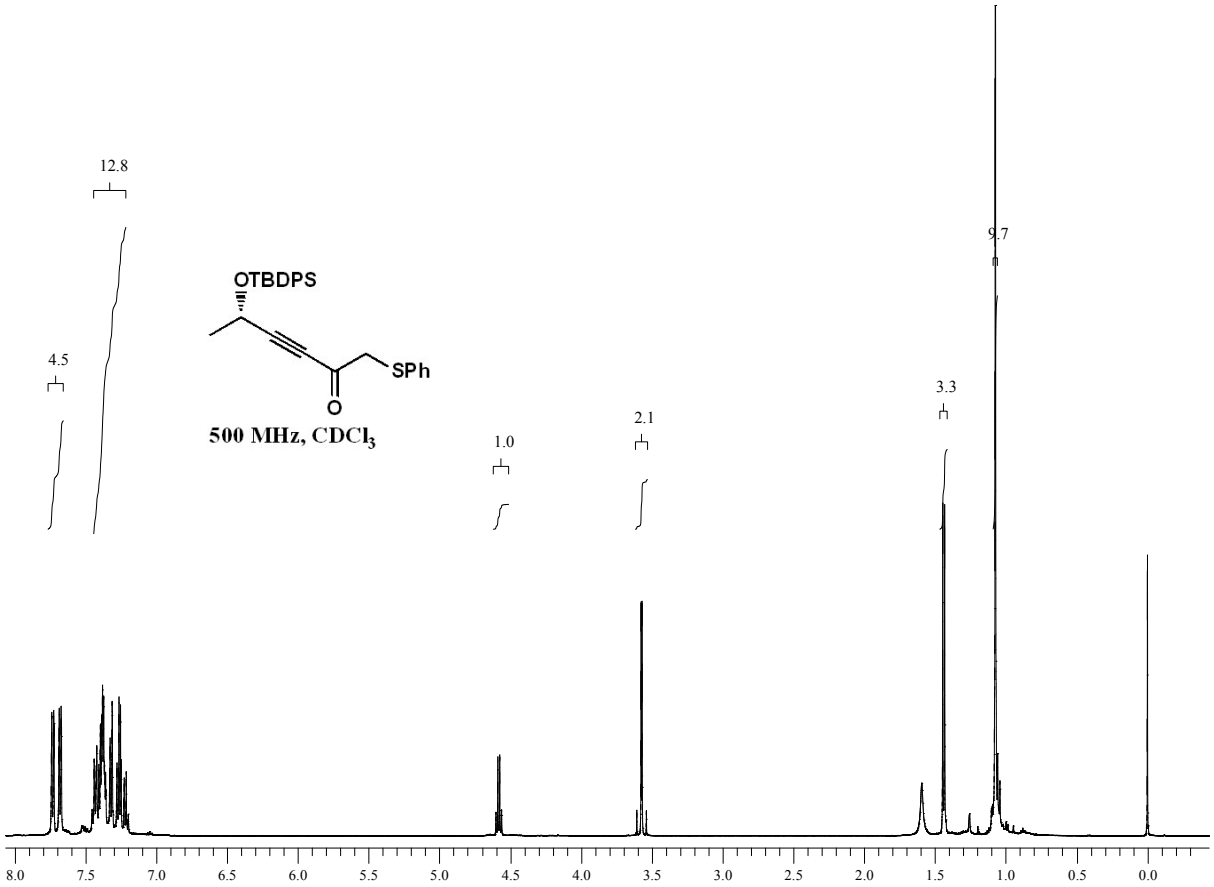

135.920
135.738
133.665
133.382
129.721
129.672
127.579
127.476
86.066
71.494
59.758
26.826
25.137
19.171
OTBDPS
100 MHz, CDCl3
130
120
110
100
90
80
70
60
50
40
30
20
10
0

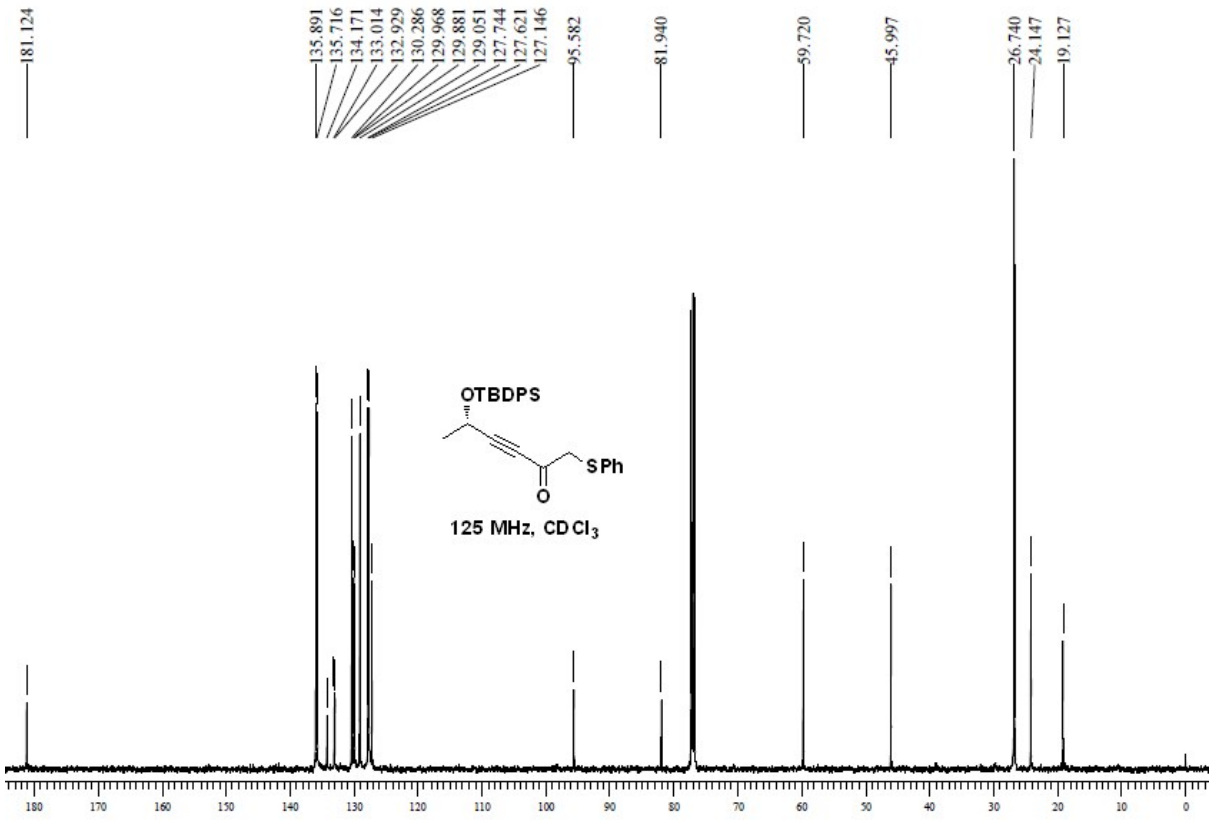
Compound 12:


24.7
18.7
8.4
6.5
2.0
2.1
2.2
2.0
1.1
1.0
OTBDPS
SPh
OH
400 MHz, CDCl3
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

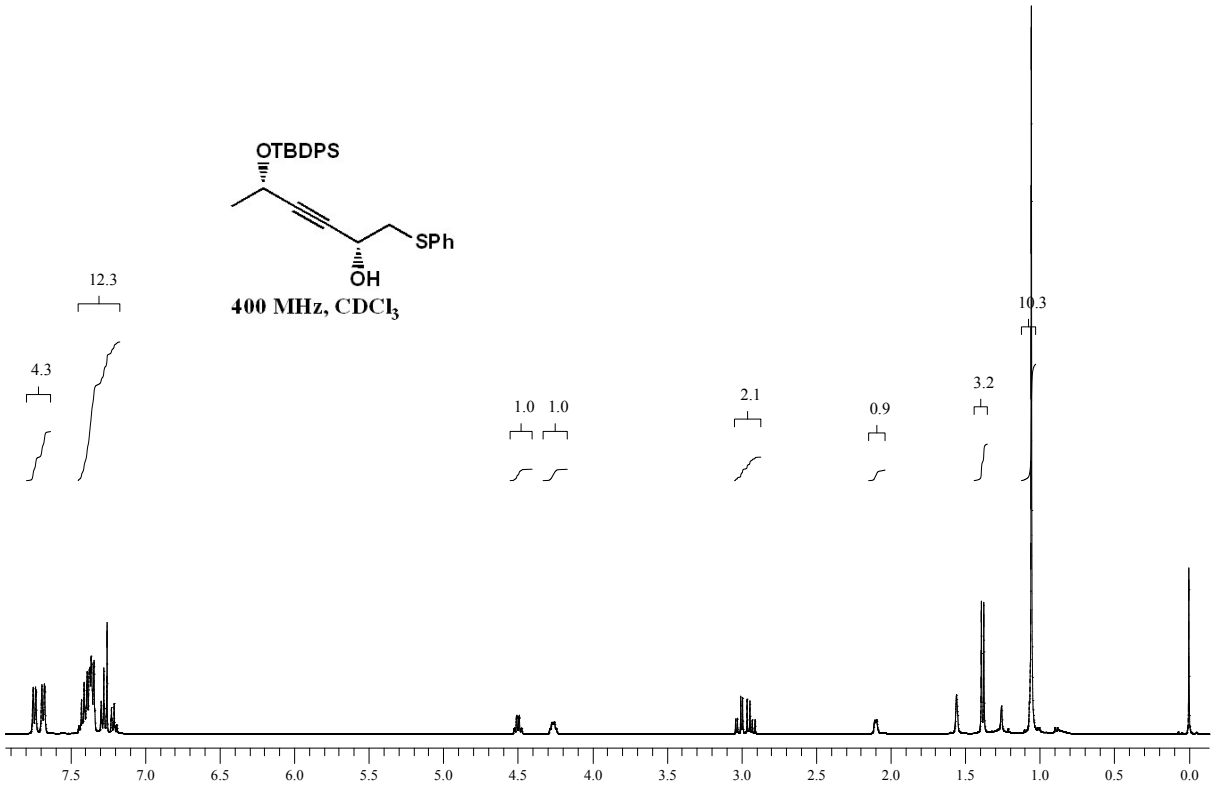

136.004
135.982
135.766
134.974
134.929
134.772
133.927
133.890
133.550
133.528
130.264
130.205
129.744
129.628
129.016
127.600
127.390
126.739
126.711
88.100
88.069
82.820
82.771
60.679
59.819
41.840
41.752
26.805
24.889
19.113
OTBDPS
SPh
OH
125 MHz, CDCl3
130
120
110
100
90
80
70
60
50
40
30
20
10
0

Compound 13:

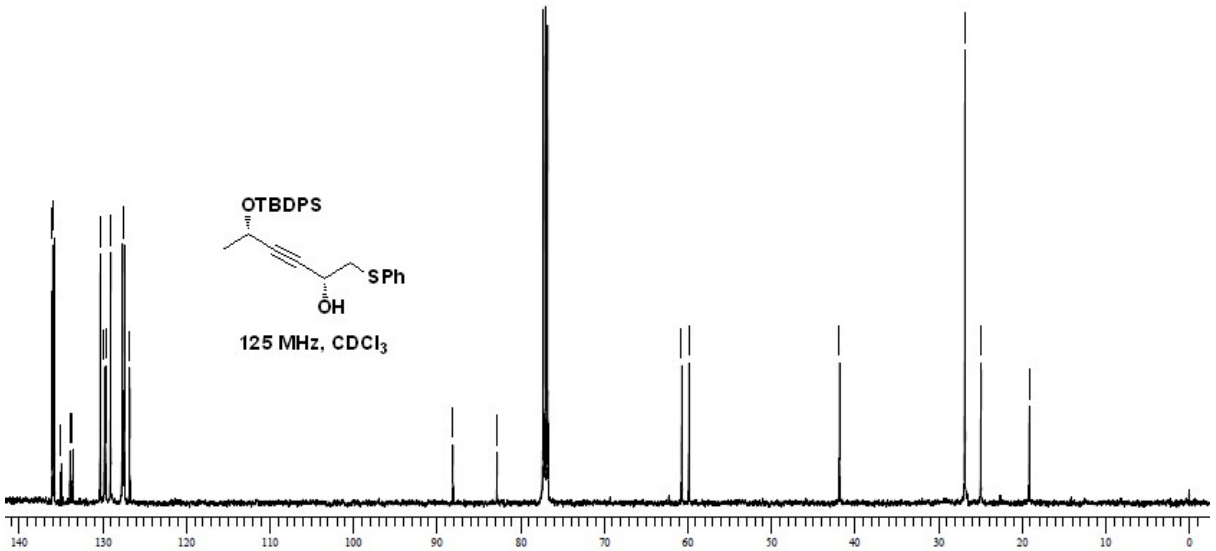

12.8
4.5
OTBDPS
SPh
O
500 MHz, CDCl3
1.0
2.1
3.3
9.7
8.0
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
0.5
0.0


181.124
135.891
135.716
134.171
133.014
132.929
130.286
129.968
129.881
129.051
127.744
127.621
127.146
95.582
81.940
59.720
45.997
26.740
24.147
19.127
OTBDPS
SPh
O
125 MHz, CDCl3
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0

Compound 14:


OTBDPS
SPh
OH
400 MHz, CDCl3
12.3
4.3
1.0
1.0
2.1
0.9
3.2
10.3
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


135.971
135.747
134.930
133.874
133.535
130.243
129.736
129.613
129.007
127.584
127.374
126.727
88.050
82.770
60.671
59.814
41.818
26.797
24.890
19.106


OTBDPS
SPh
OH
125 MHz, CDCl3
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0

HPLC Spectra of compound 14 :

10/16/2015 14:18:20 1 / 1

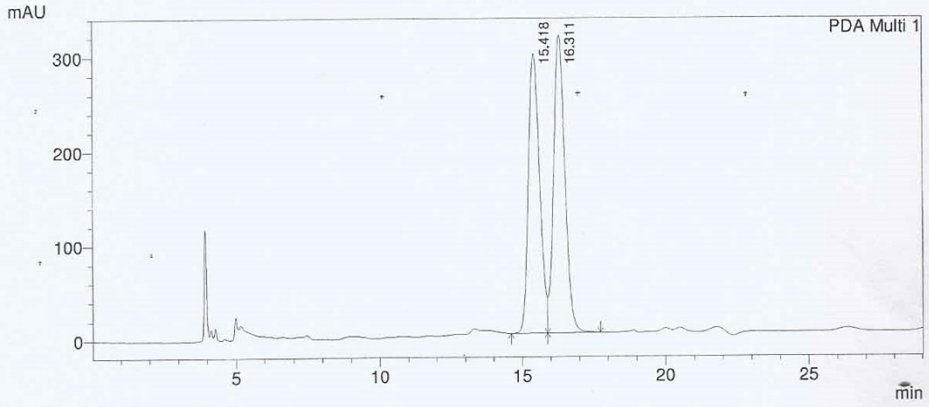
IICT NPL

Acquired by	: Admin
Sample Name	: SR-JP-SNR-RACEMIC
Sample ID	: SR-JP-SNR-RACEMIC
Vail #	: 16
Injection Volume	: 20 uL
Data File Name	: SR-JP-SNR-RACEMIC..5.lcd
Method File Name	: SR-JP.lcm
Batch File Name	:
Report File Name	: Report Format.lcr
Data Acquired	: 10/16/2015 11:21:23 AM
Data Processed	: 10/16/2015 11:50:28 AM

MOBILE PHASE: 2% IPA IN HEXANE
Column:: CHIRAL PAK IA (250mm X 4.6 mm, 5u)
Flowrate: 0.8mL/min
DETECTION : 210nm

<Chromatogram>

C:\LabSolutions\Data\Project1\SR-JP-SNR-RACEMIC..5.lcd



1 PDA Multi 1/210nm 4nm

PeakTable

PDA Ch1 210nm 4nm

Peak#	Ret. Time	Area	Height	Area %	Height %
1	15.418	8113751	296550	48.281	48.404
2	16.311	8691524	316100	51.719	51.596
Total		16805275	612650	100.000	100.000

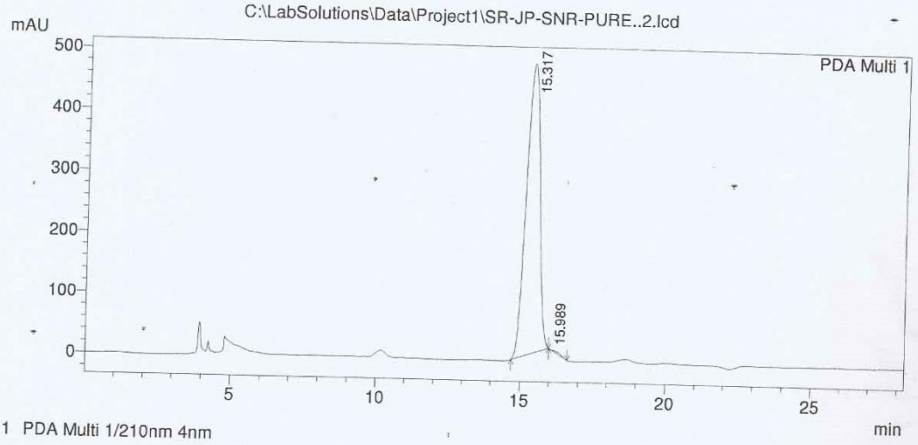
C:\LabSolutions\Data\Project1\SR-JP-SNR-RACEMIC..5.lcd

IICT NPL

Acquired by	: Admin
Sample Name	: SR-JP-SNR-PURE
Sample ID	: SR-JP-SNR-PURE
Vail #	: 17
Injection Volume	: 20 uL
Data File Name	: SR-JP-SNR-PURE..2.lcd
Method File Name	: SR-JP.lcm
Batch File Name	:
Report File Name	: Report Format.lcr
Data Acquired	: 10/16/2015 12:19:16 PM
Data Processed	: 10/16/2015 2:29:05 PM

MOBILE PHASE: 2% IPA IN HEXANE
Column:: CHIRAL PAK IA (250mm X 4.6 mm, 5u)
Flowrate: 0.8mL/min
DETECTION : 210nm

<Chromatogram>



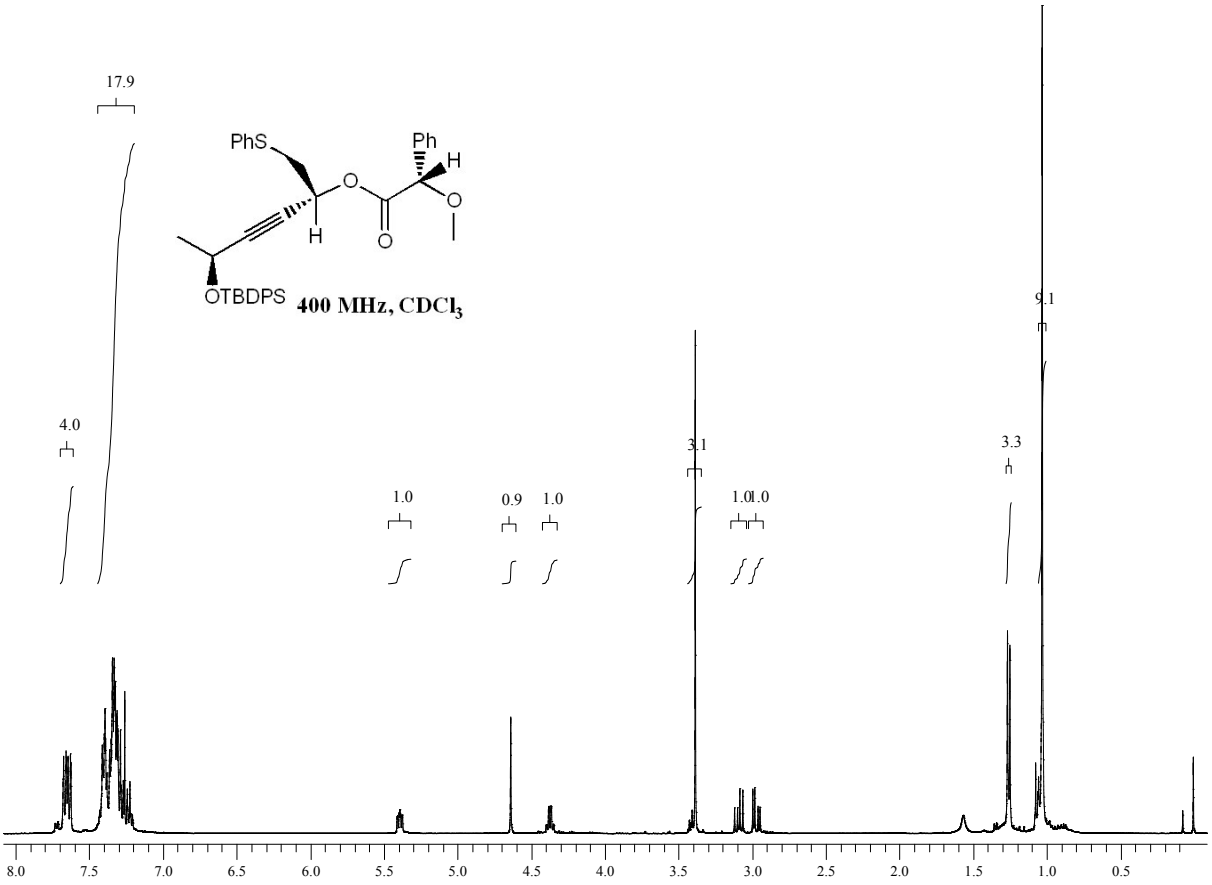
1 PDA Multi 1/210nm 4nm

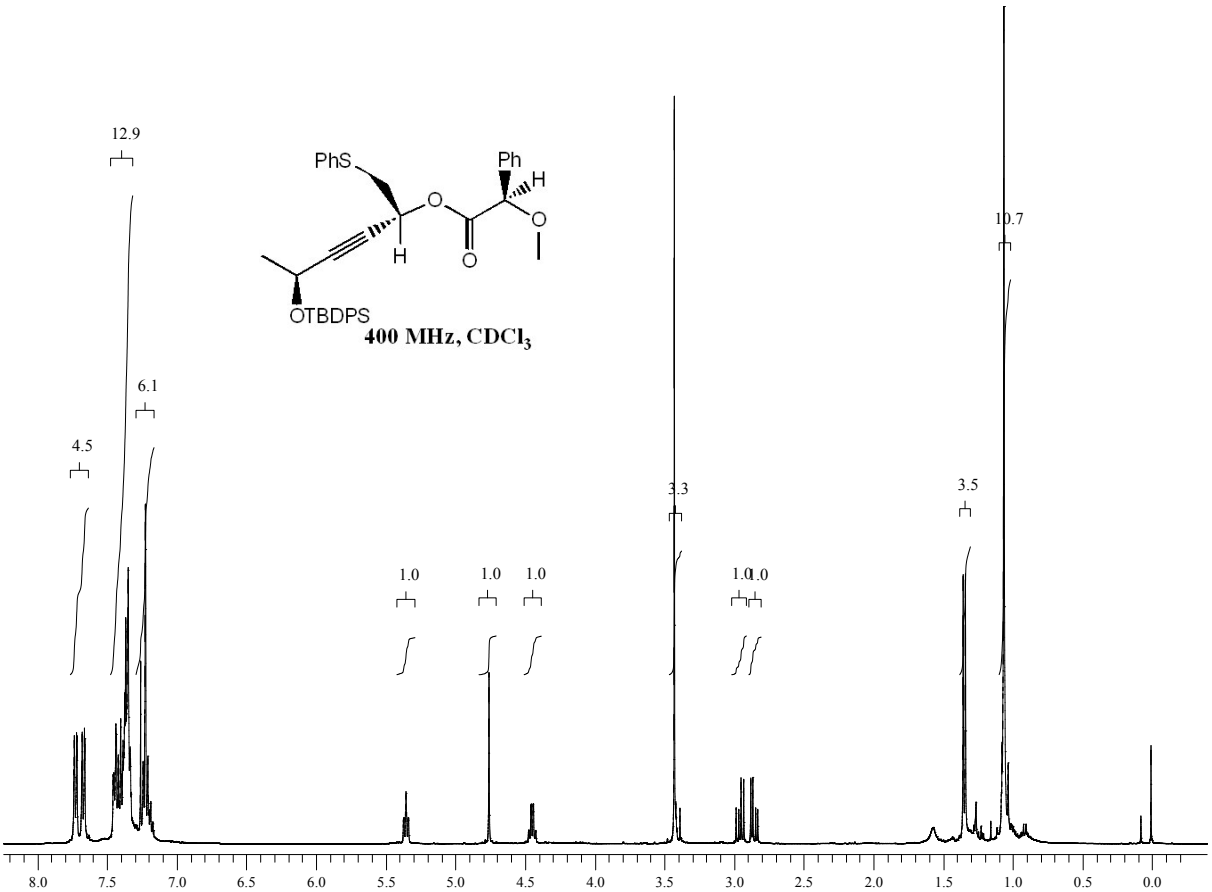
PeakTable

PDA Ch1 210nm 4nm

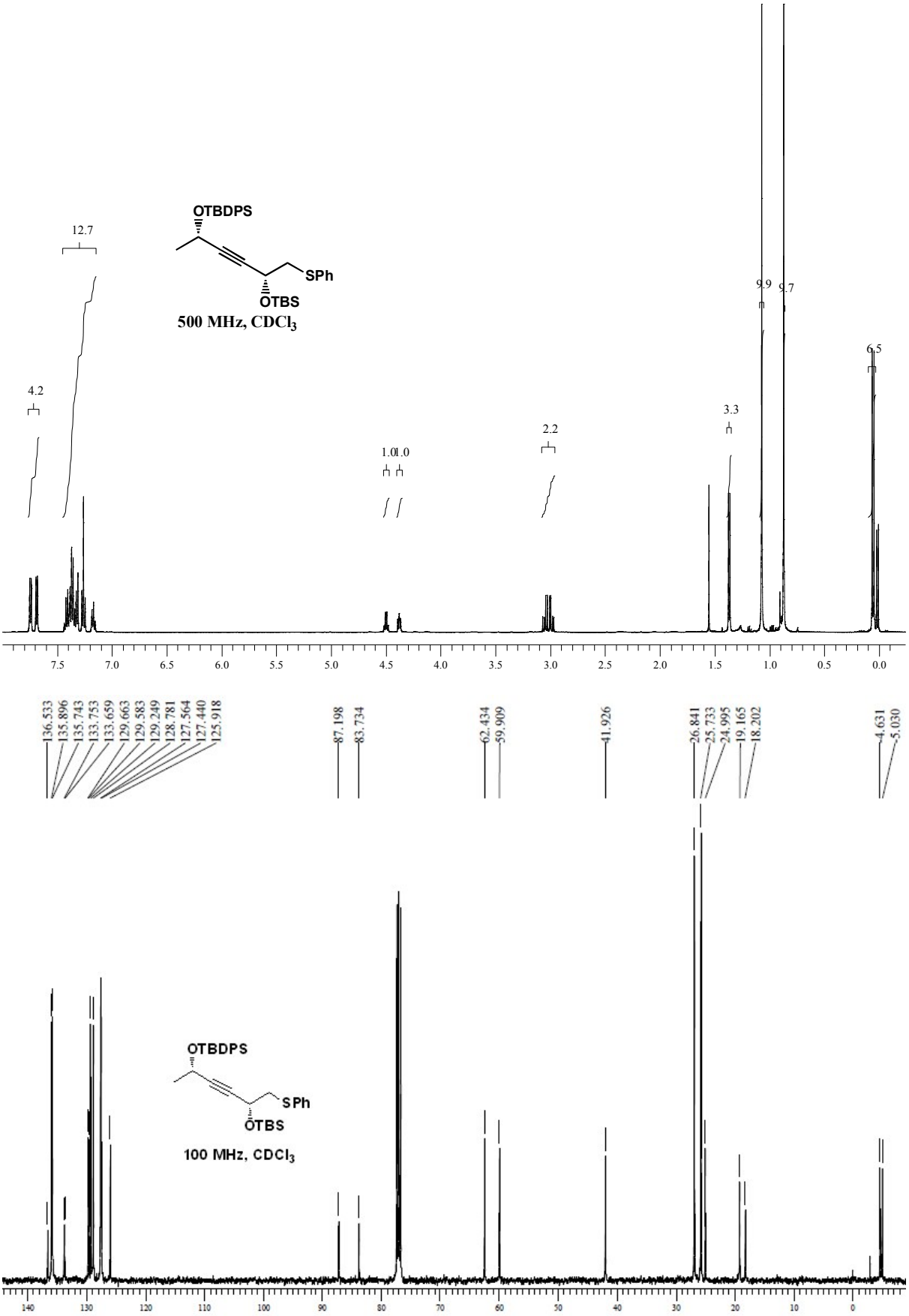
Peak#	Ret. Time	Area	Height	Area %	Height %
1	15.317	15330137	475657	99.672	100.031
2	15.989	50425	-148	0.328	-0.031
Total		15380562	475509	100.000	100.000

Mandelic esters of compound 14 :

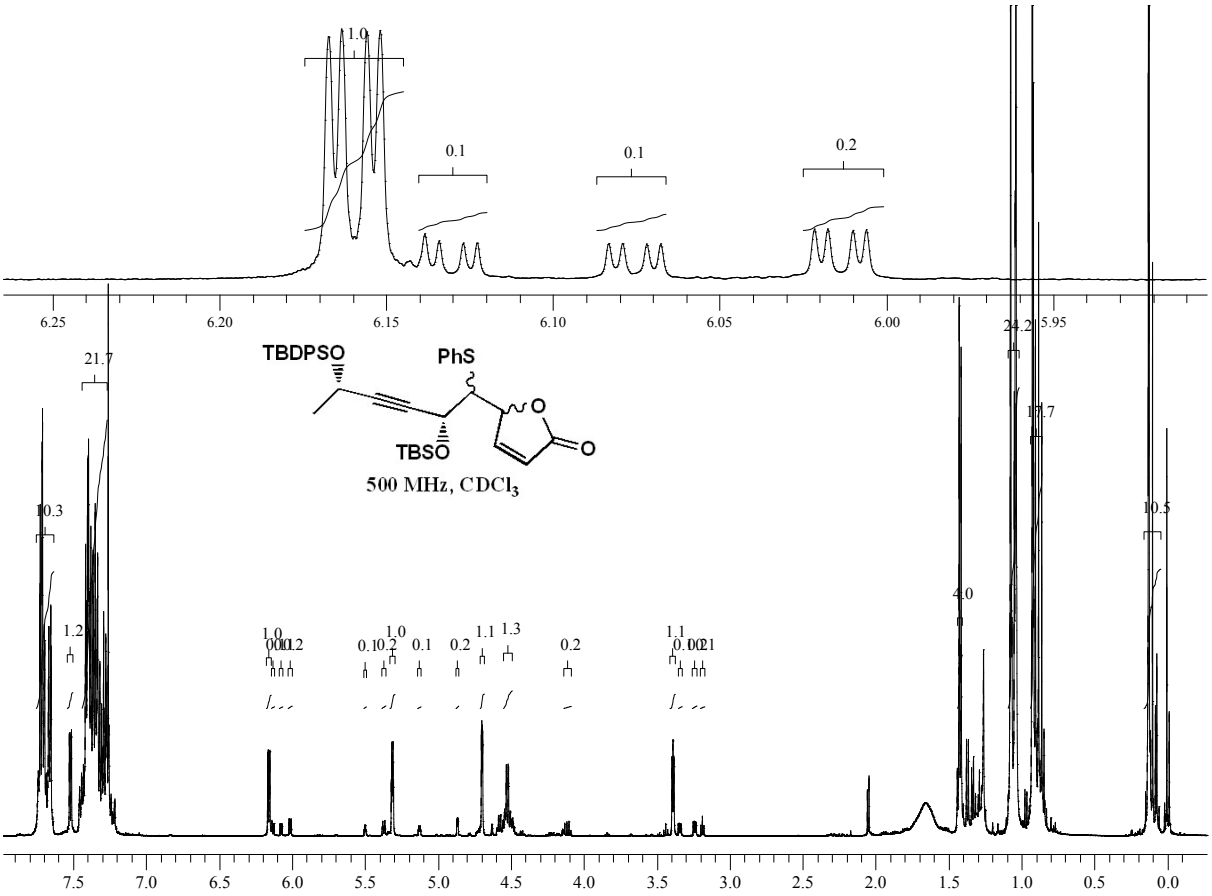




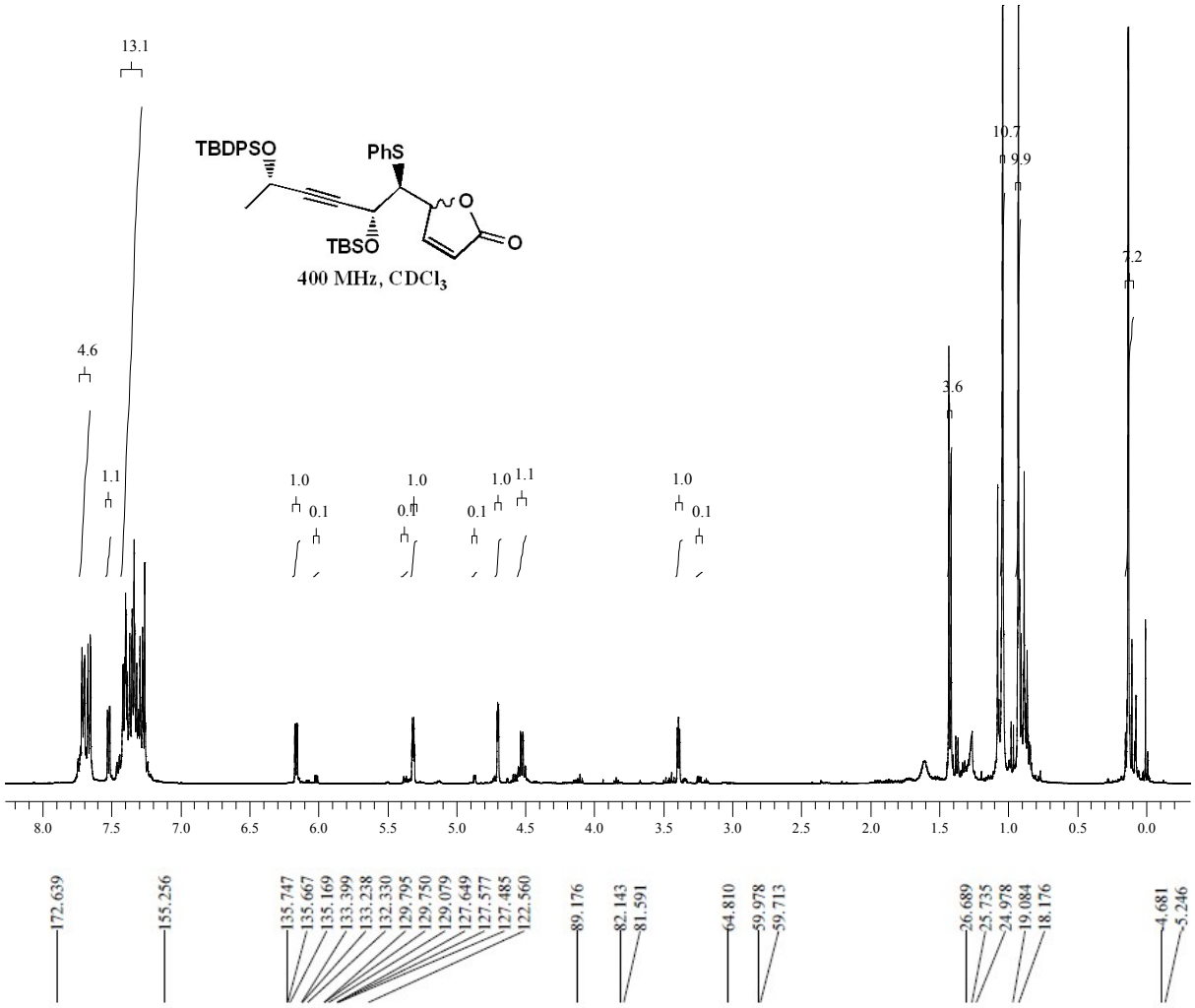
Compound 7 :

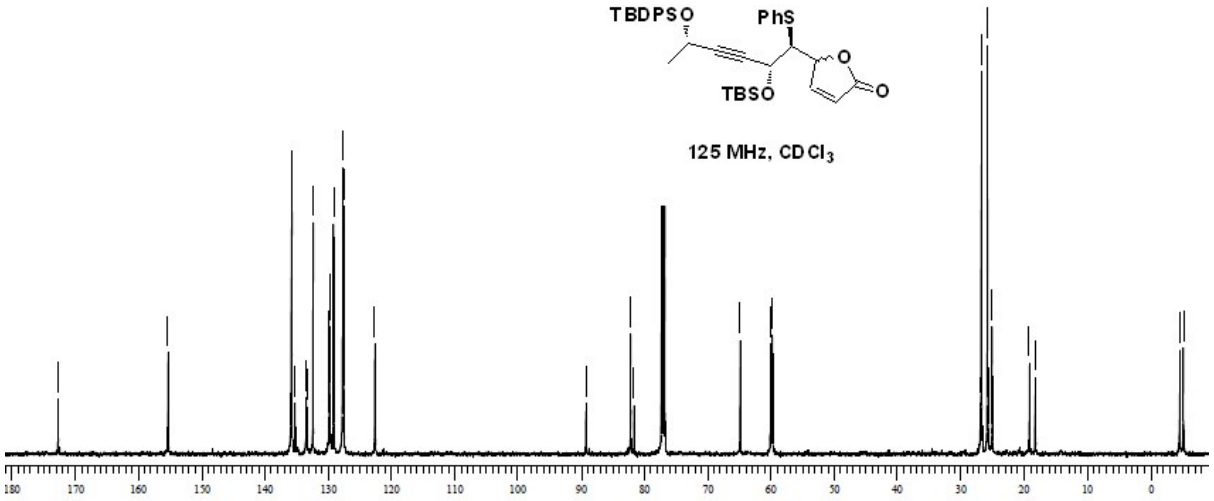

OTBDPS
SPh
OTBS
500 MHz, CDCl3
12.7
4.2
1.0
1.0
2.2
3.3
9.9
9.7
6.5
136.533
135.896
135.743
133.753
133.659
129.663
129.583
129.249
128.781
127.564
127.440
125.918
87.198
83.734
62.434
59.909
41.926
26.841
25.733
24.995
19.165
18.202
-4.631
-5.030
OTBDPS
SPh
OTBS
100 MHz, CDCl3

Crude compound (15 + 16 +17 +18) $ZnBr_2$ catalyzed reaction:

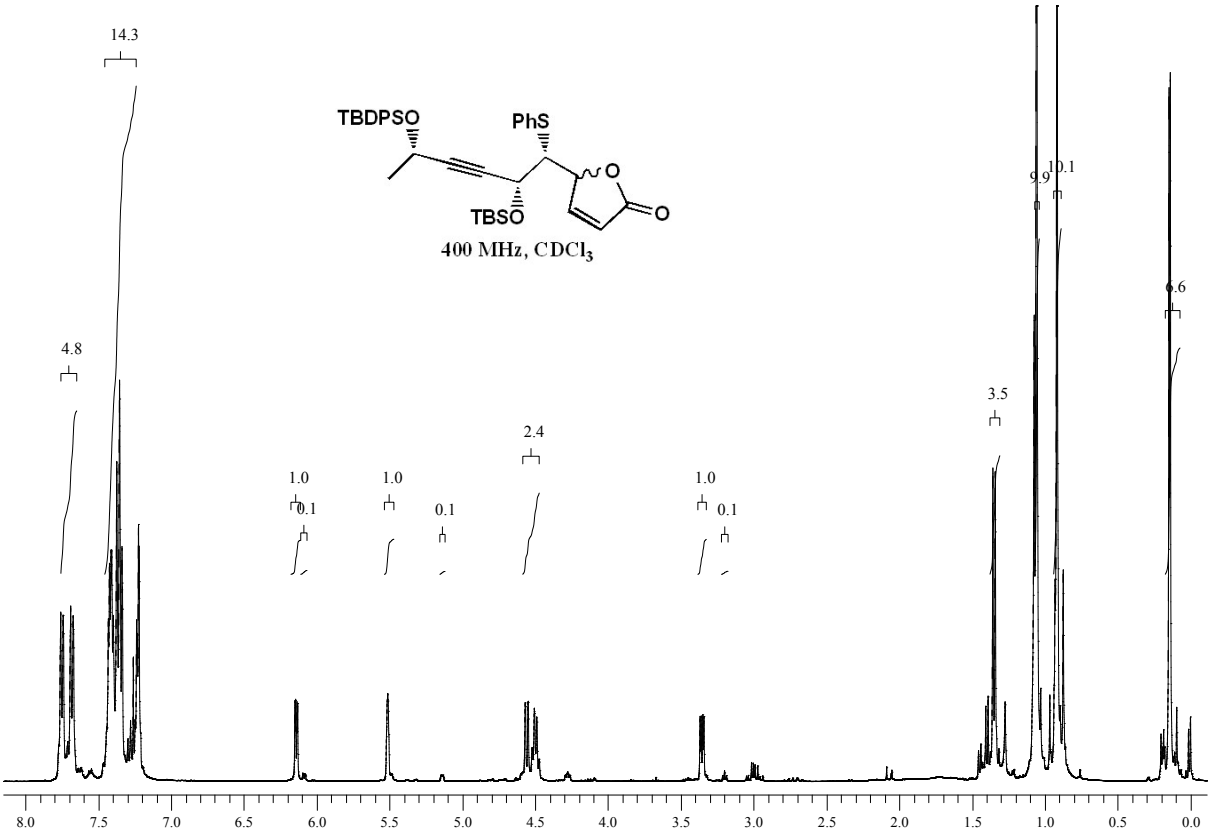


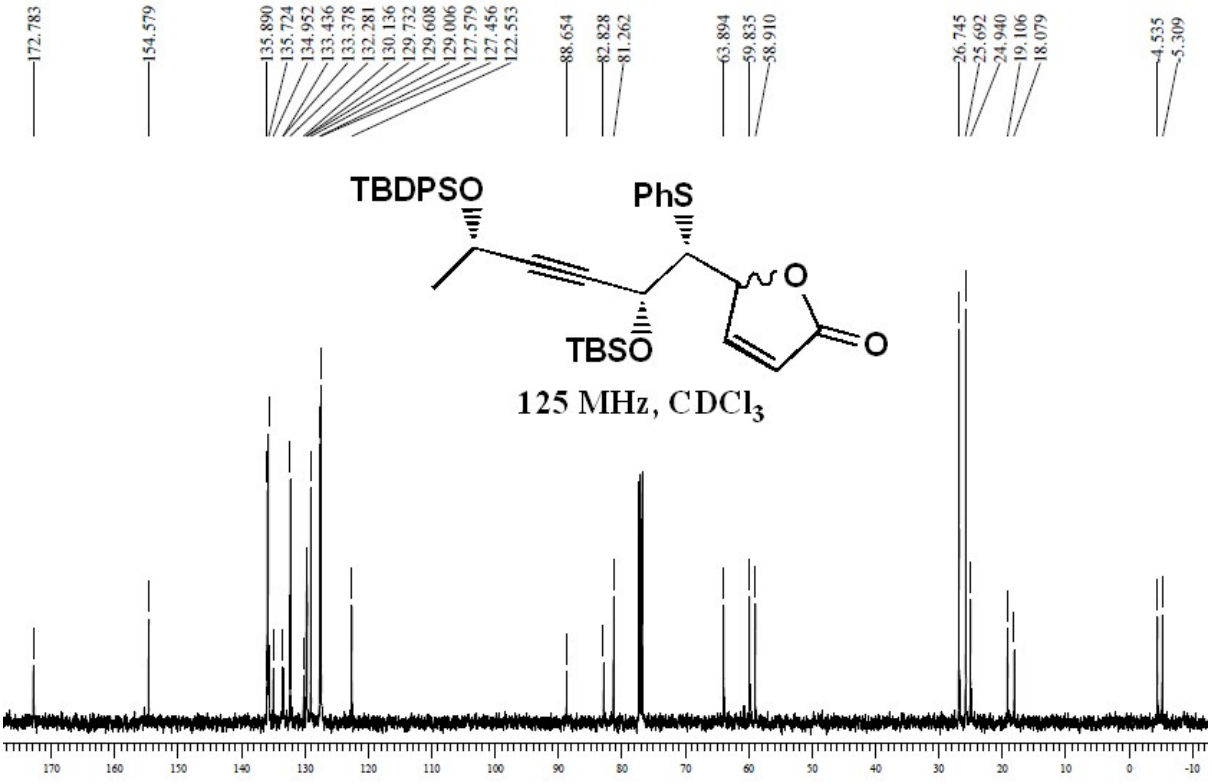
Compound (15 + 16) :



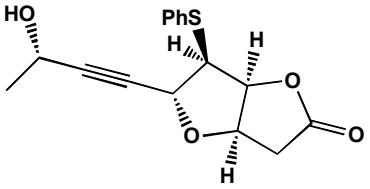


Compound (17 + 18) :

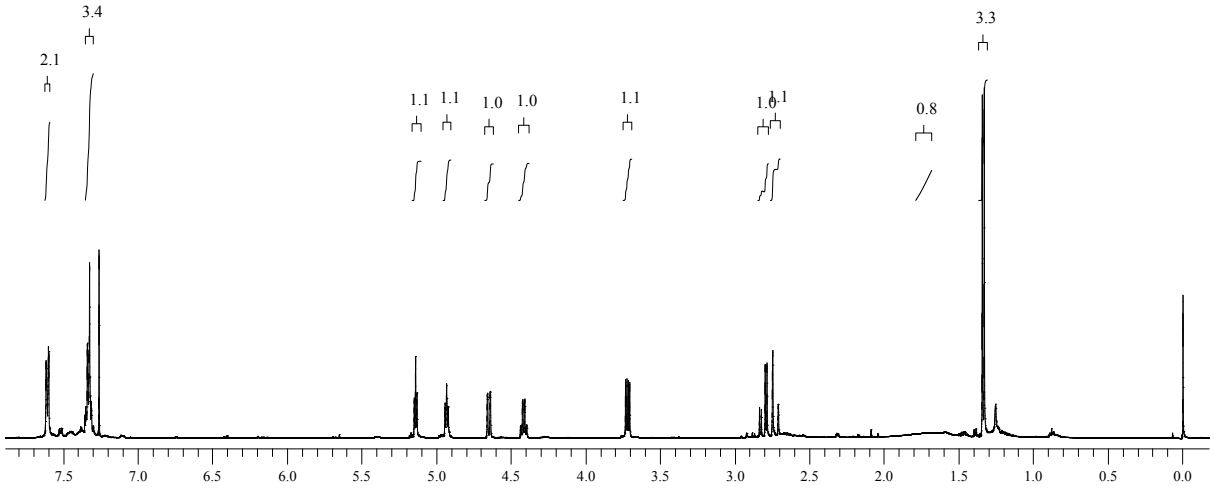


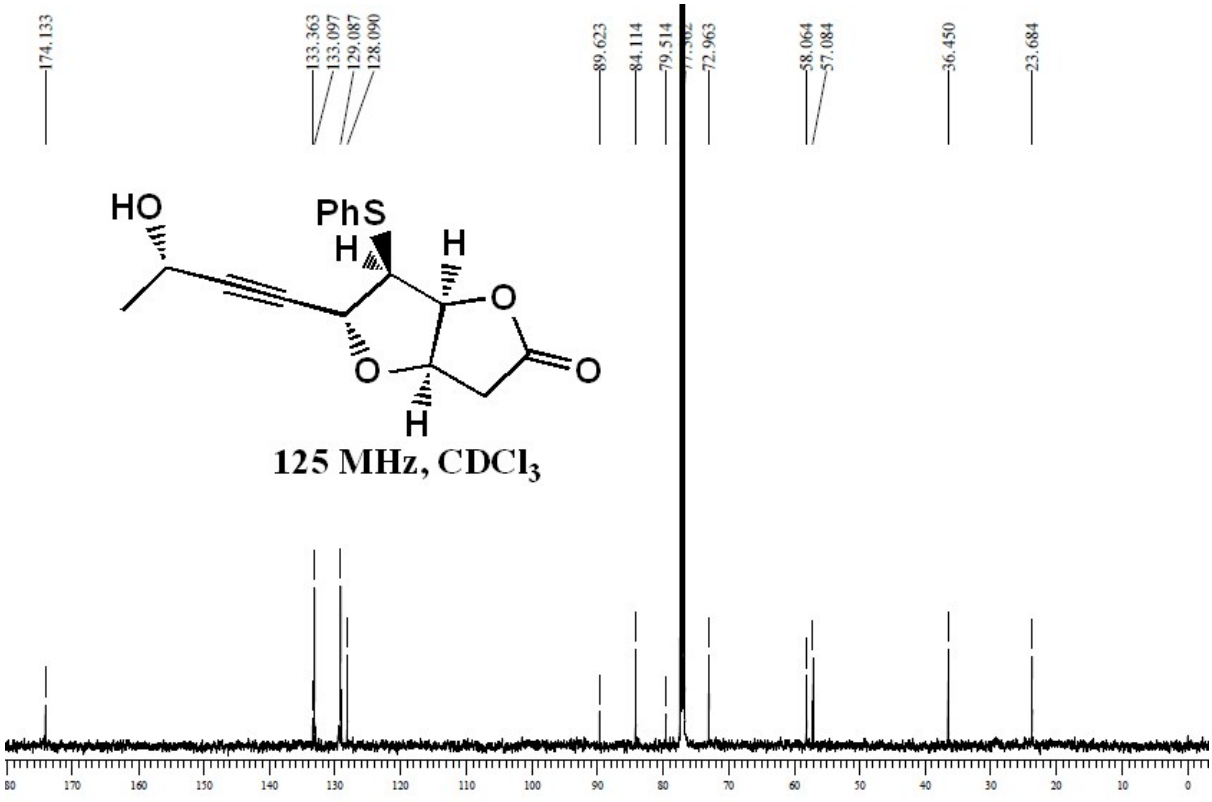


Compound 4 :

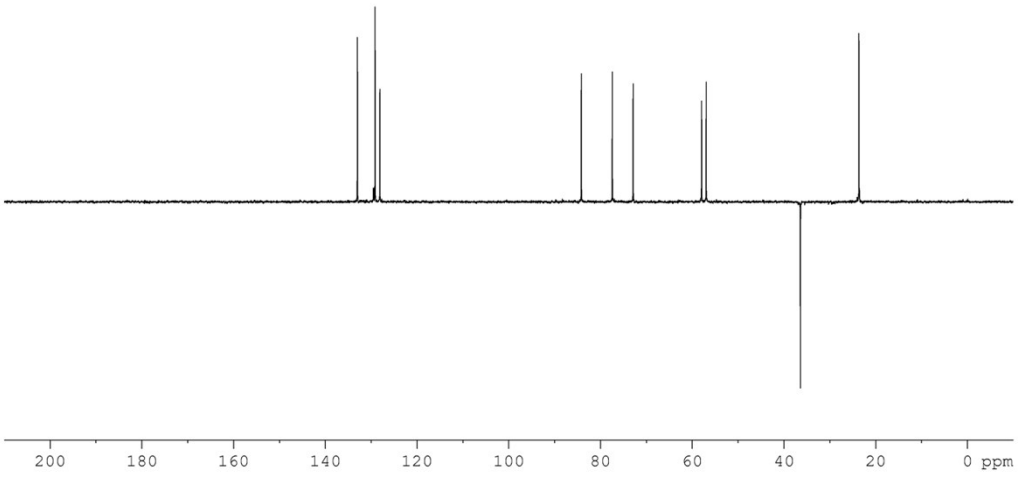


500 MHz, $CDCl_3$

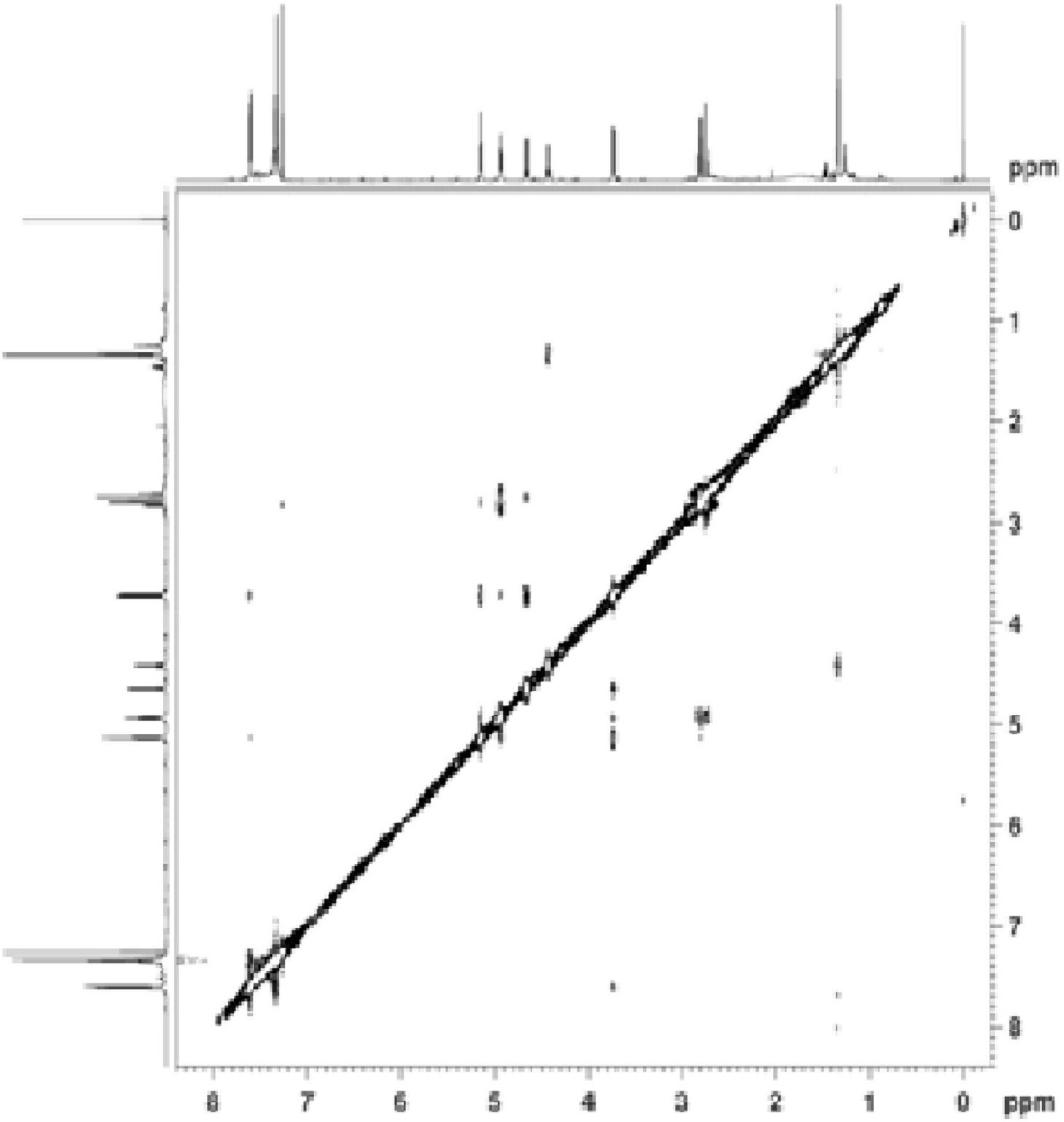




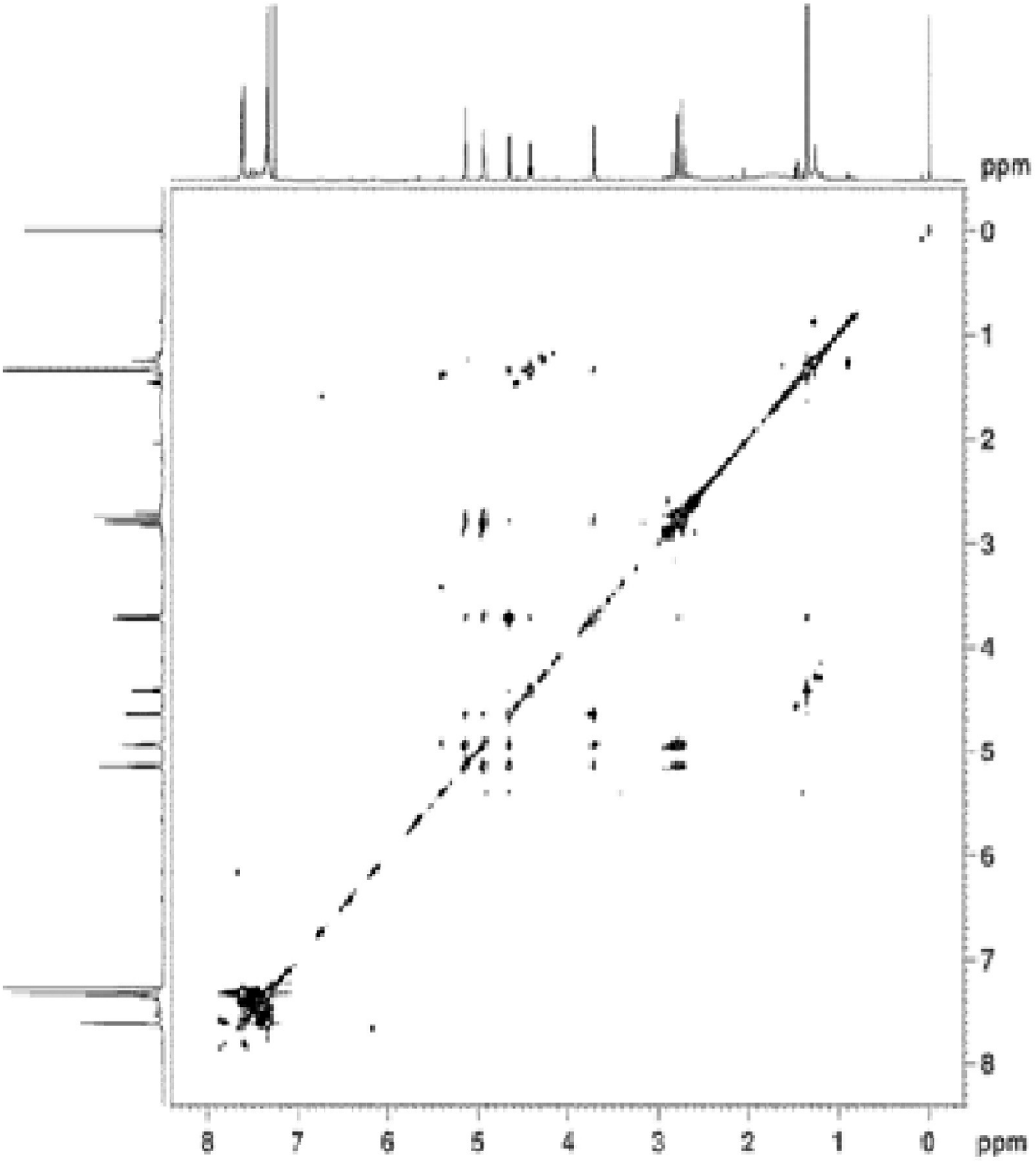
DEPT 135 of Compound 4:



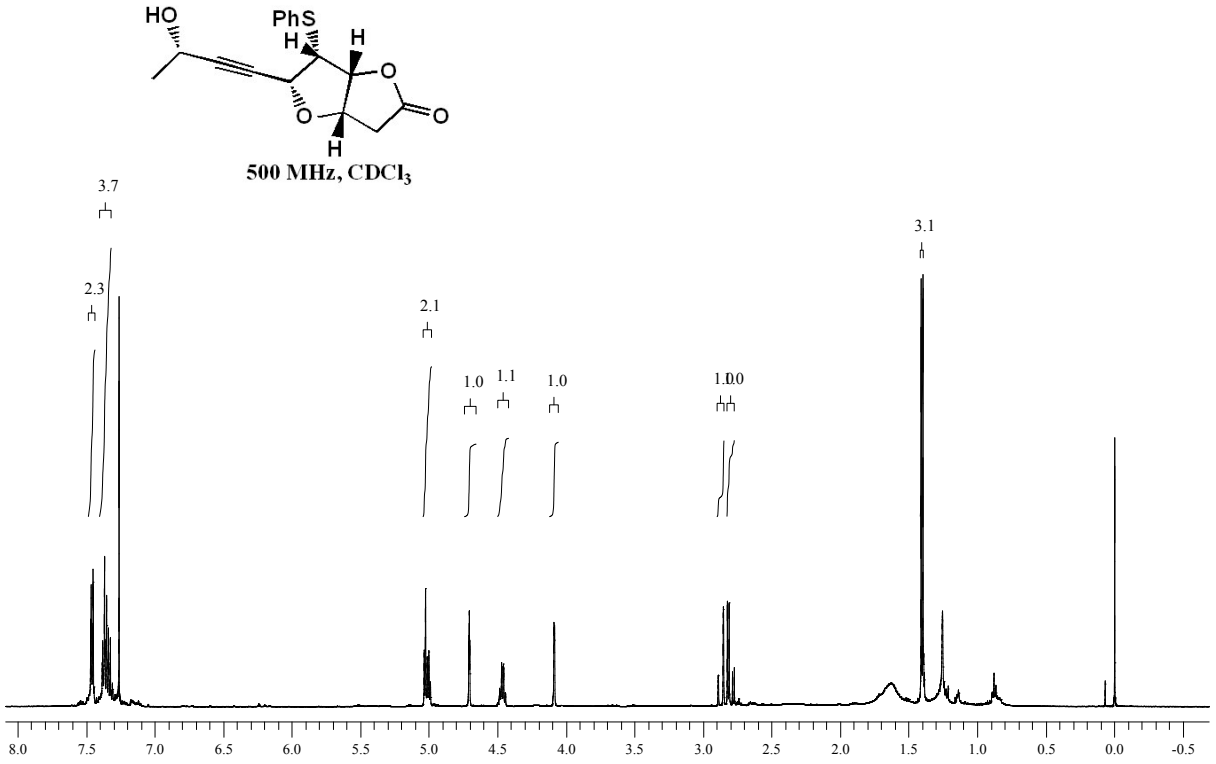
NOESY Spectrum of compound 4:

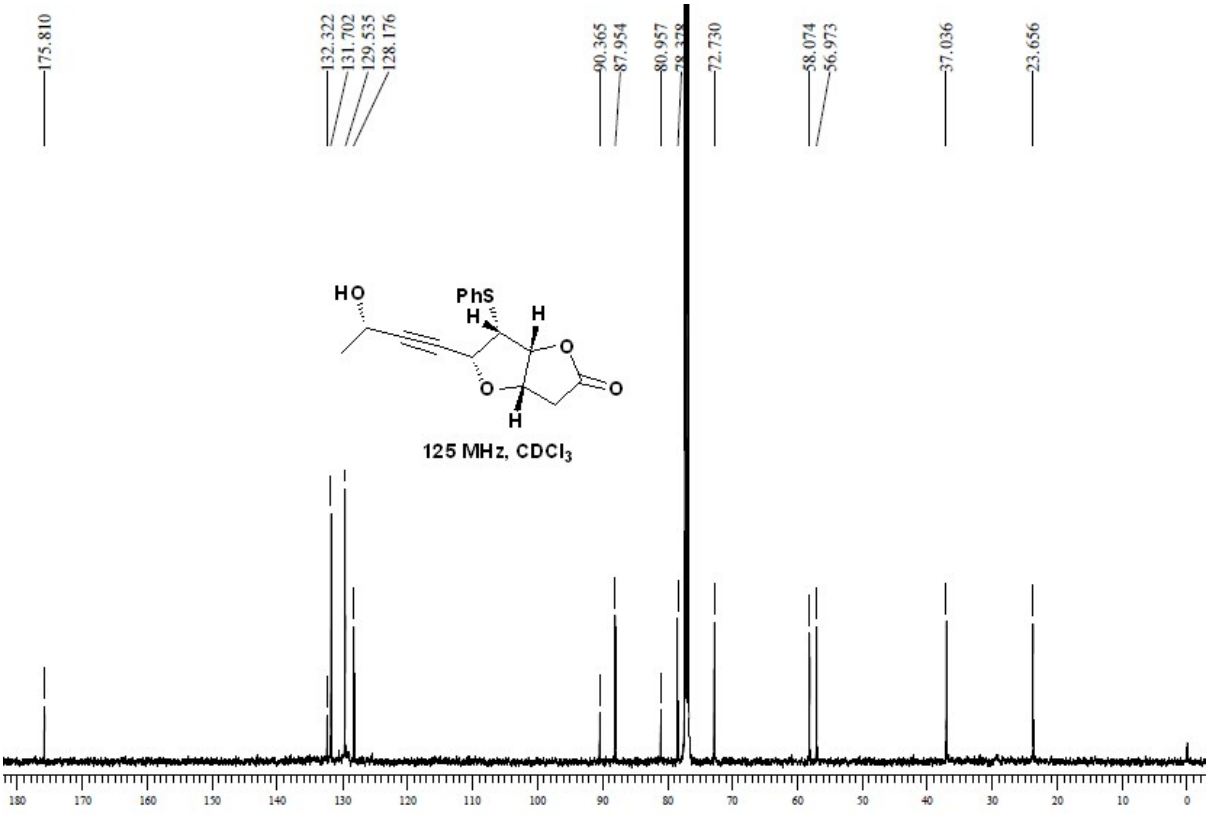


NOESY Spectrum of compound 4:

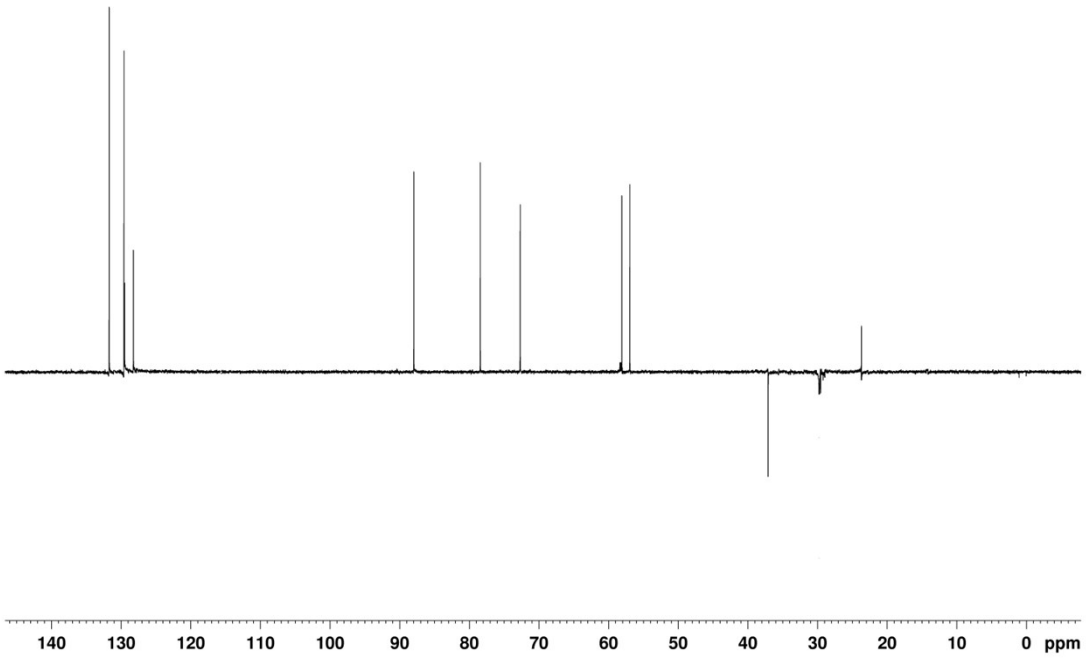


Compound 19:

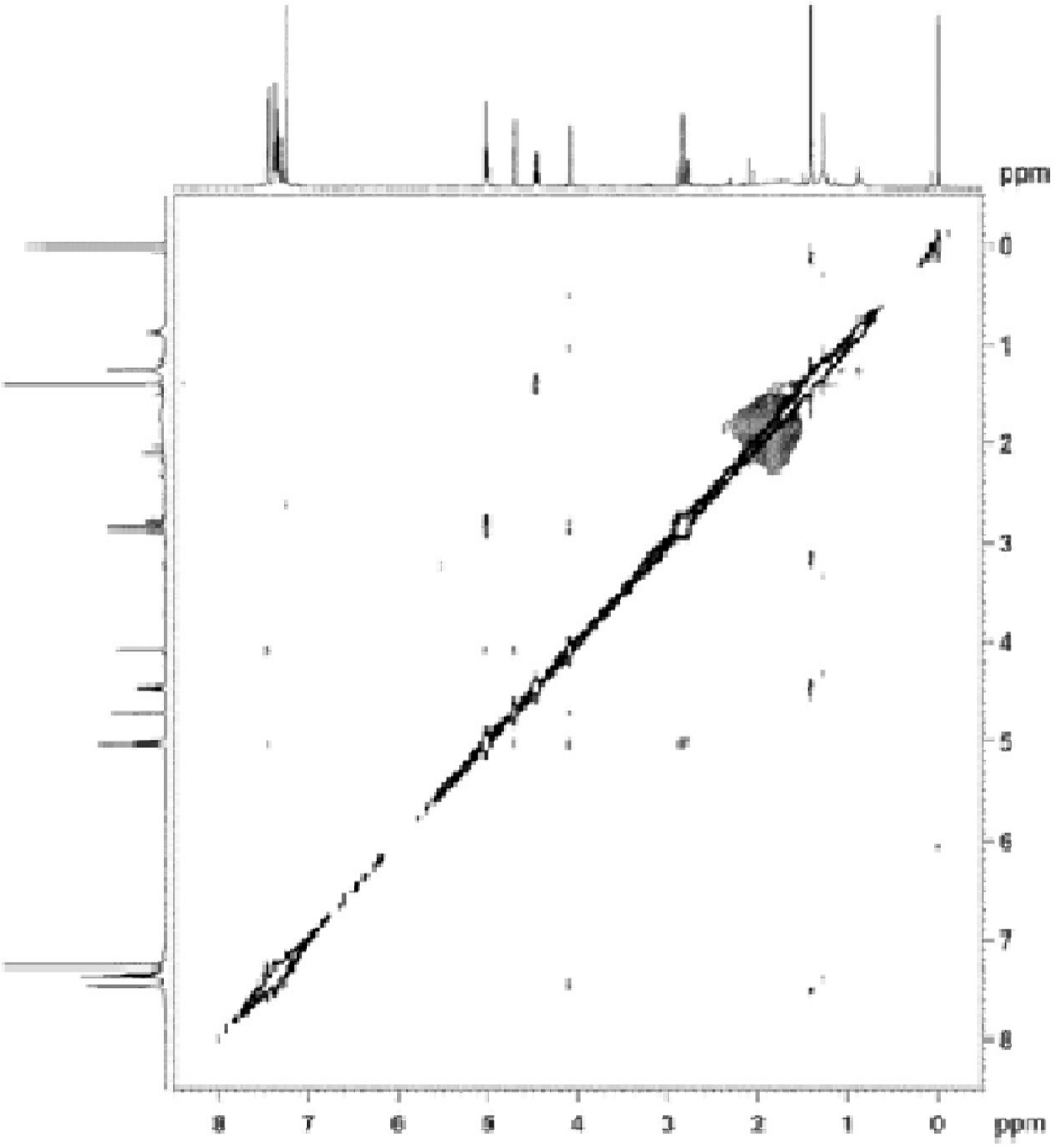

HO
PhS
H
H
O
O
O
H
500 MHz, CDCl3
3.7
2.3
2.1
1.0
1.1
1.0
1.0
1.0
3.1
8.0
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
0.5
0.0
-0.5



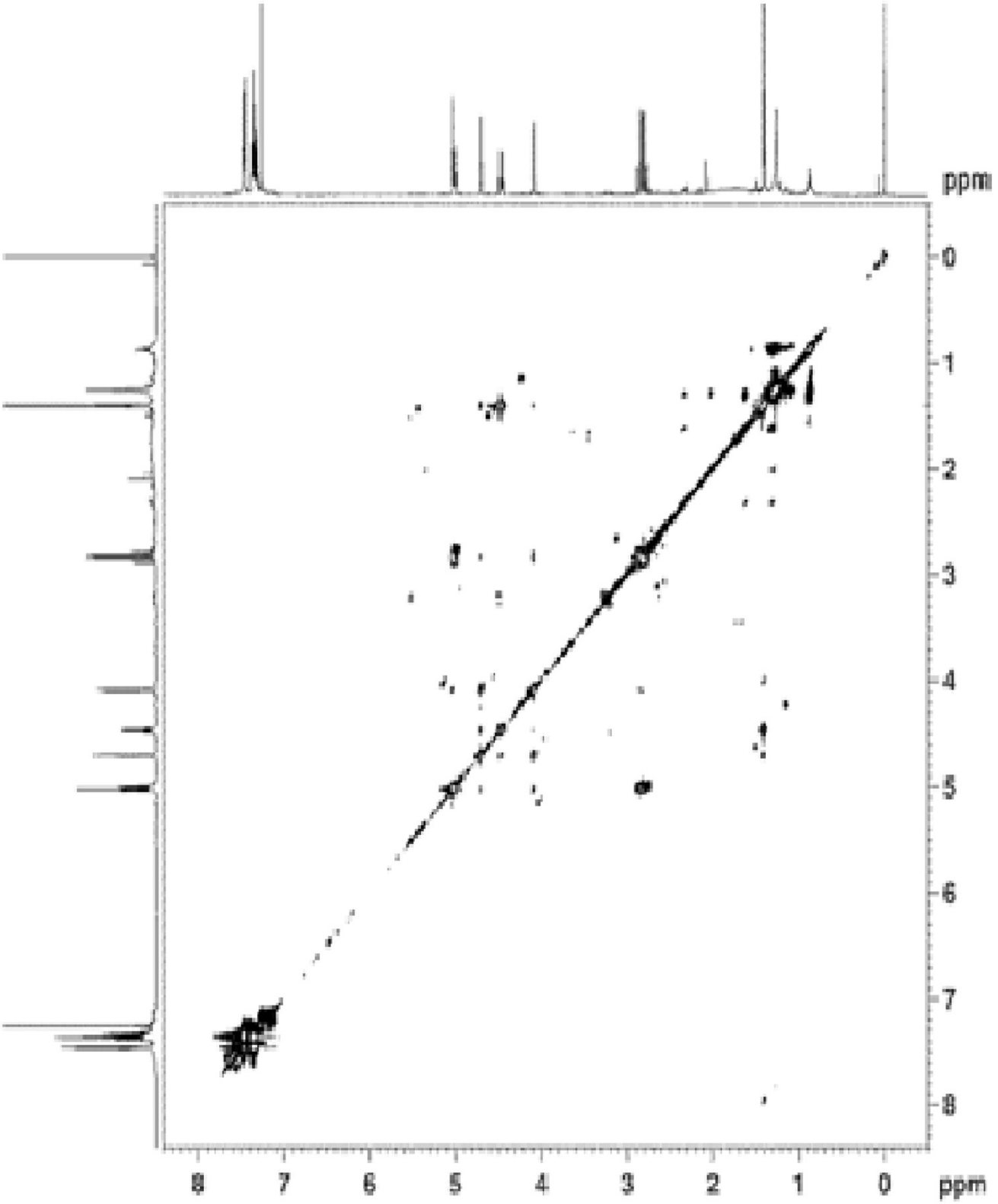
DEPT 135 of Compound 19:



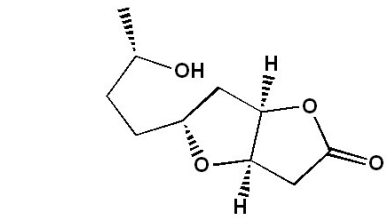
NOESY Spectrum of compound 19:



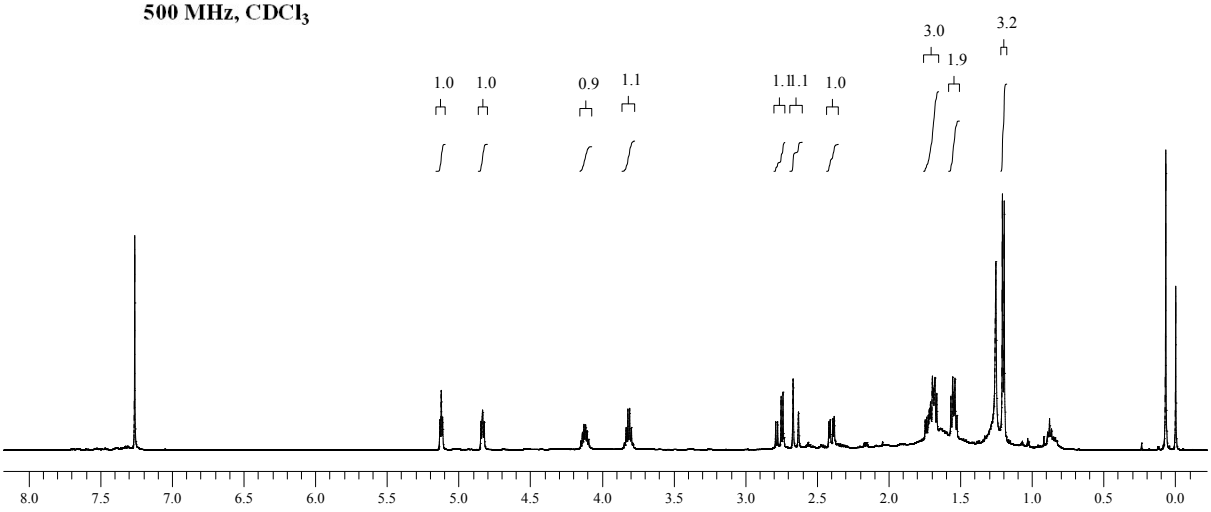
NOESY Spectrum of compound 19:

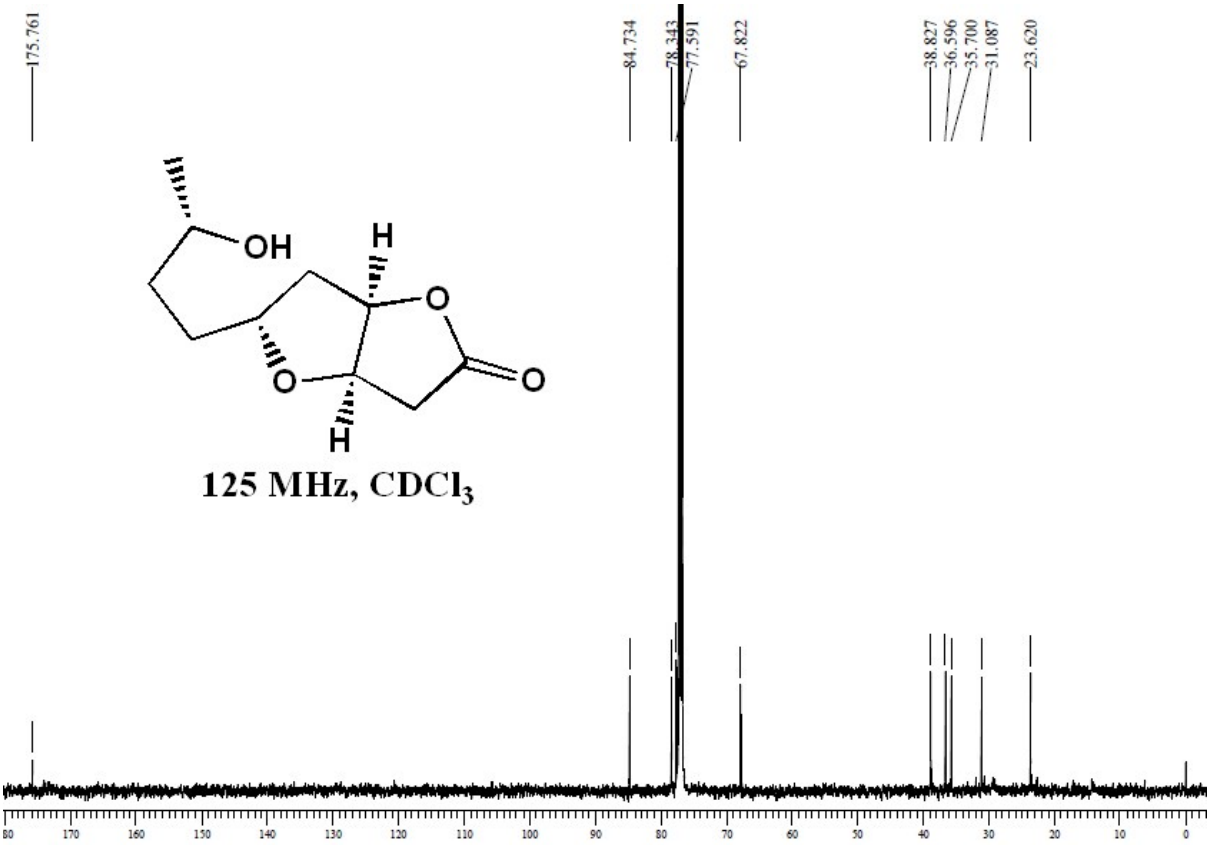


Compound 3 :



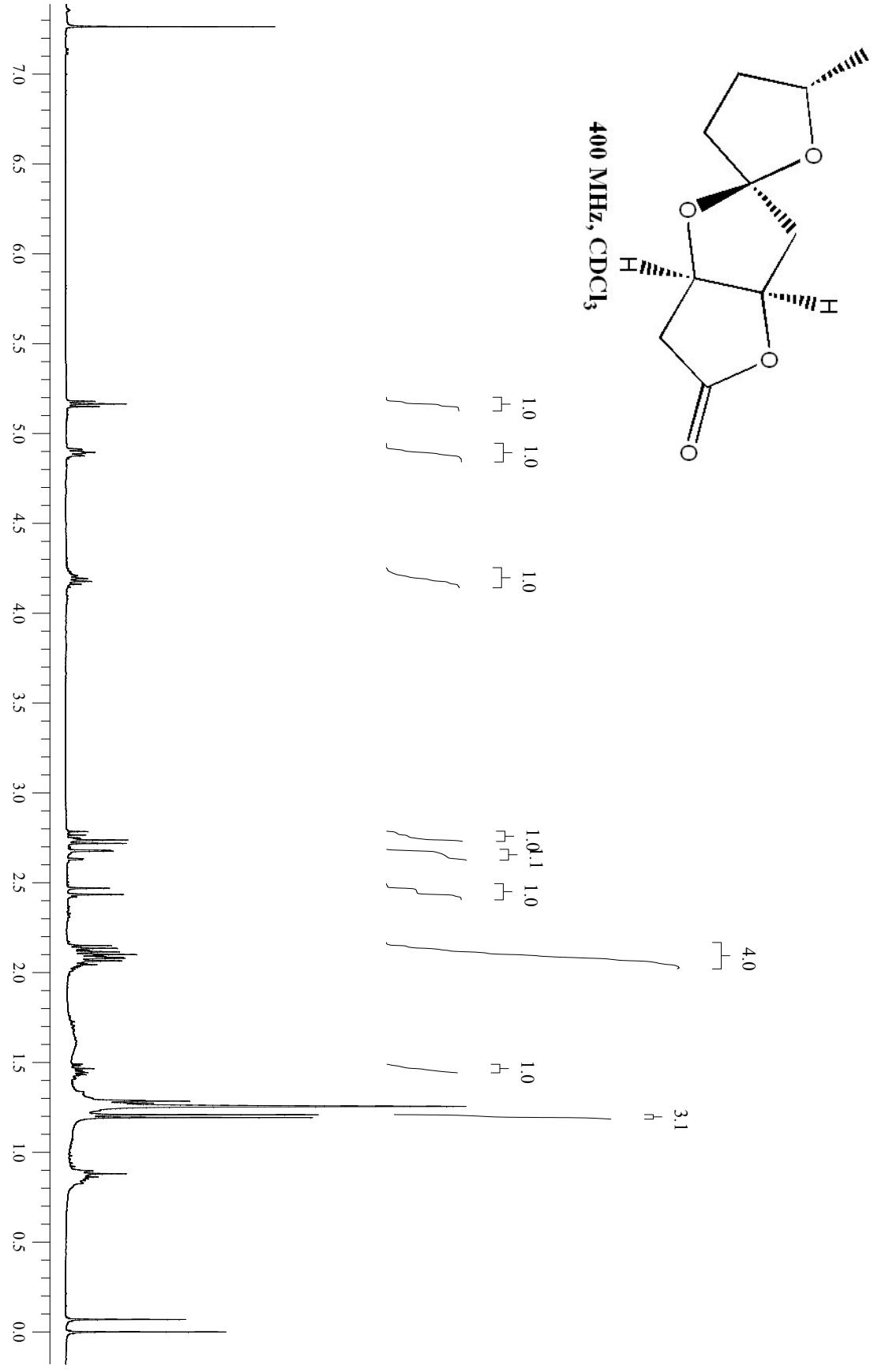
500 MHz, $CDCl_3$

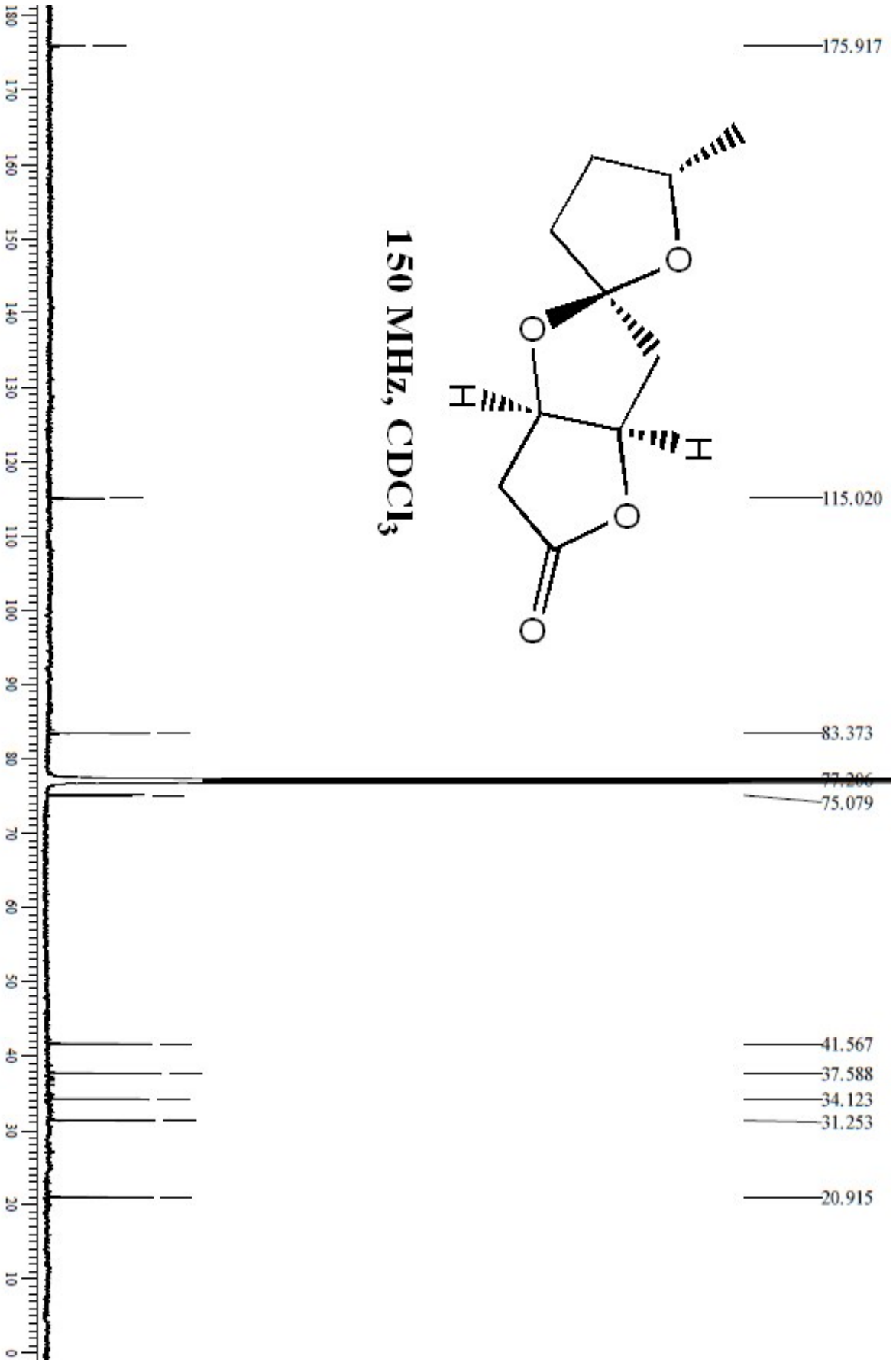




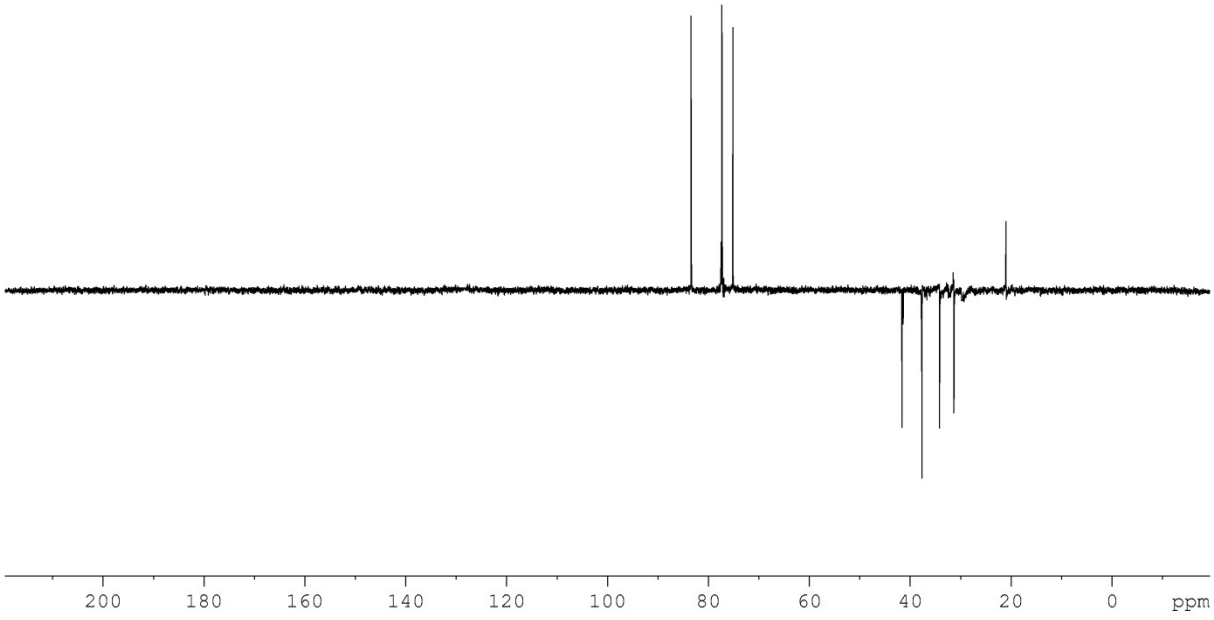
125 MHz, $CDCl_3$

(–) Cephalosporolide E :

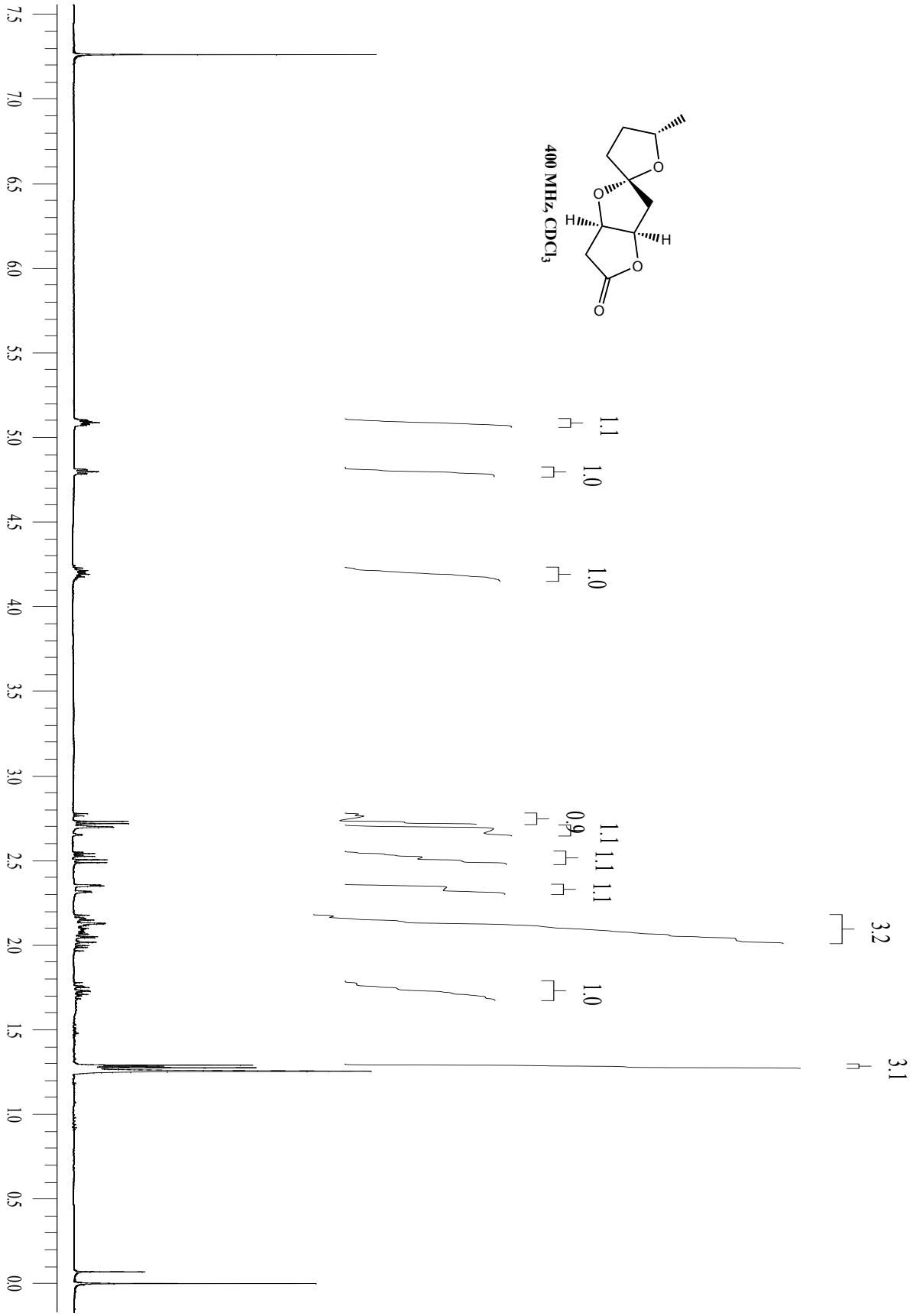


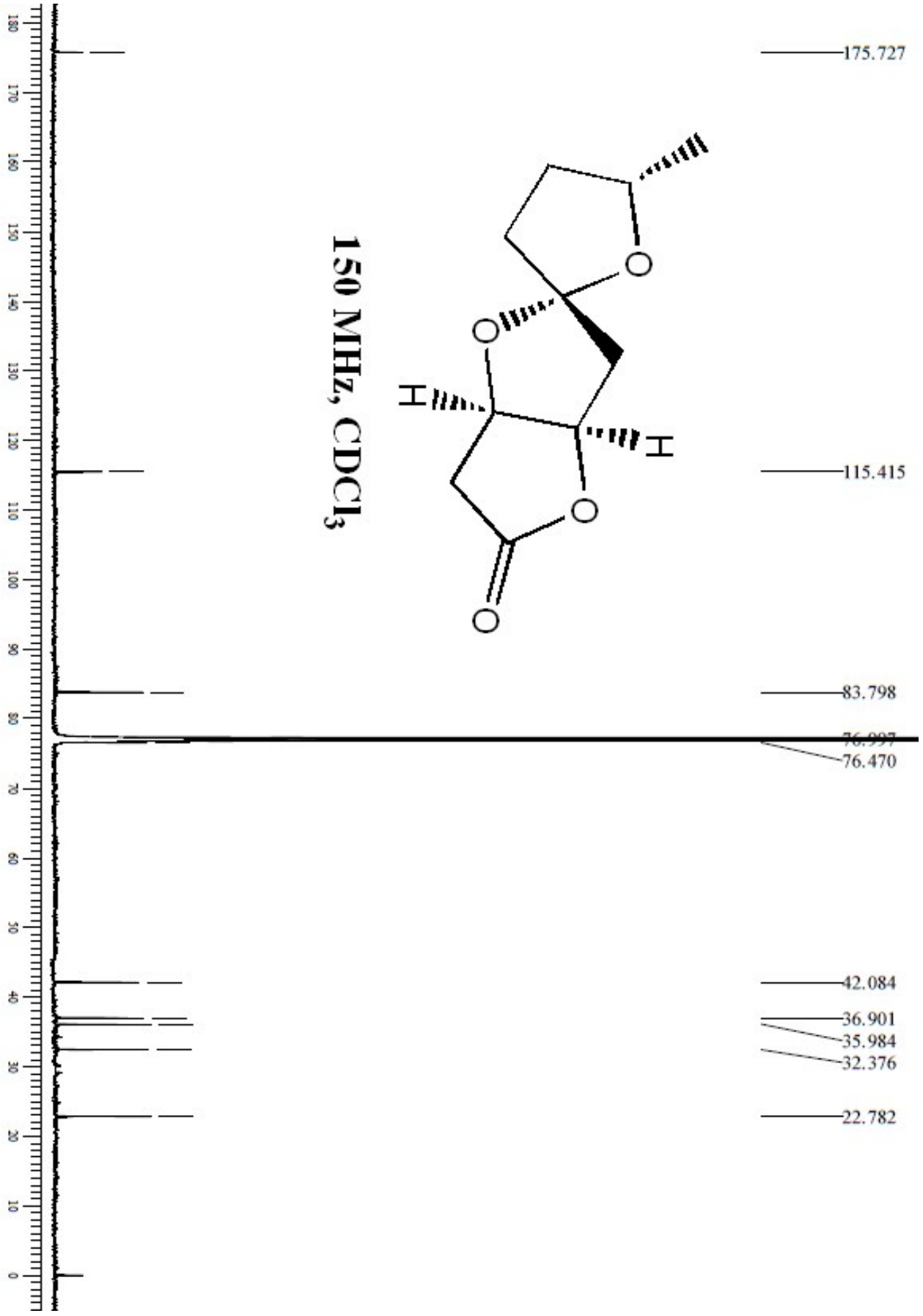

175.917
115.020
83.373
77.206
75.079
41.567
37.588
34.123
31.253
20.915
150 MHz, CDCl3
O
O
O
O
H
H

DEPT 135 of Cephalosporolide-E:



(+) Cephalosporolide F :




150 MHz, CDCl3
175.727
115.415
83.798
76.997
76.470
42.084
36.901
35.984
32.376
22.782