A Hetero-Diels-Alder Approach to Complex Pyrones: An Improved Synthesis of the Spongistatin AB Spiroketal

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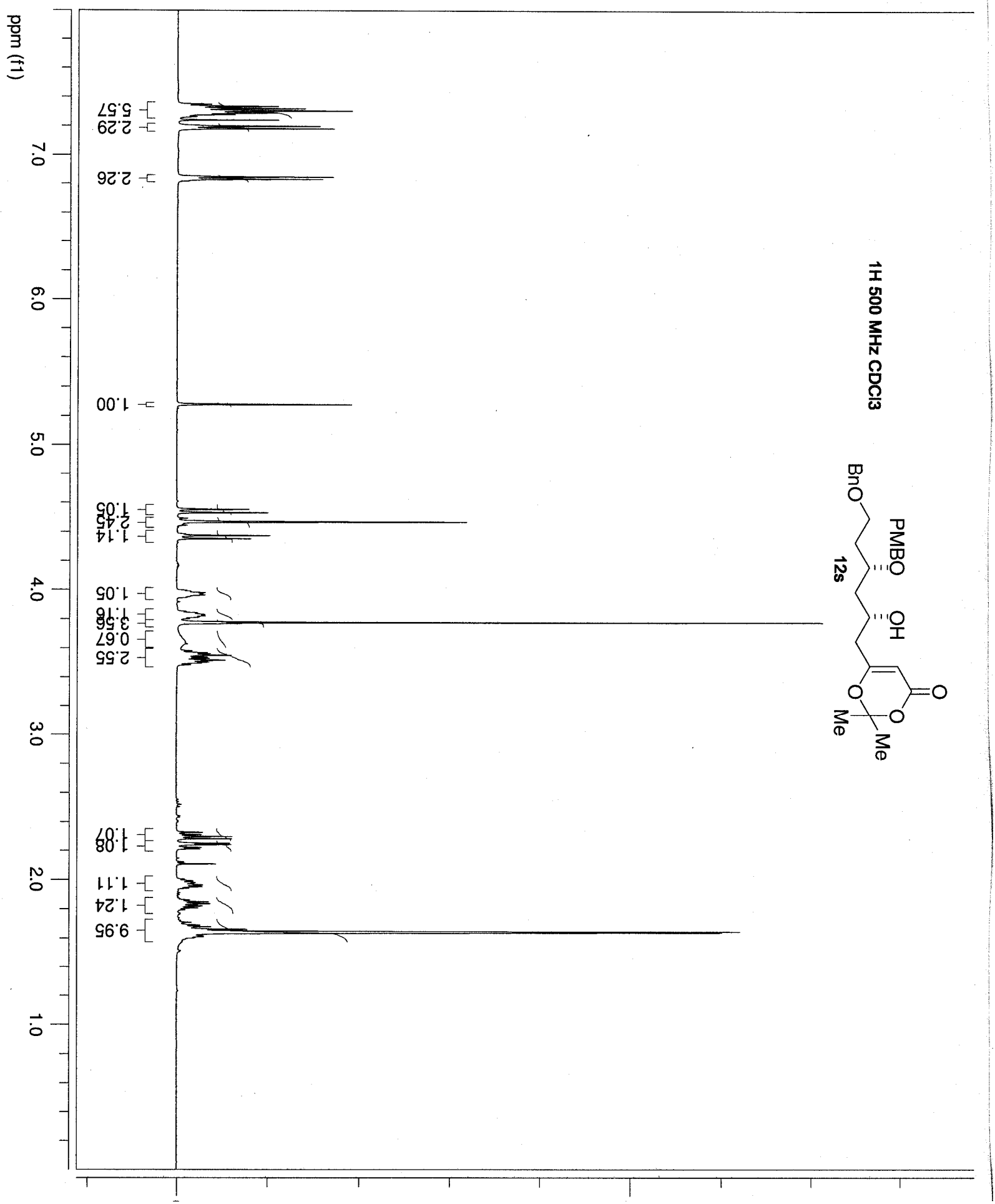
Supporting Informaiton: NMR Spectra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12s
BnO
PMBO
OH
O
O
O
Me
Me
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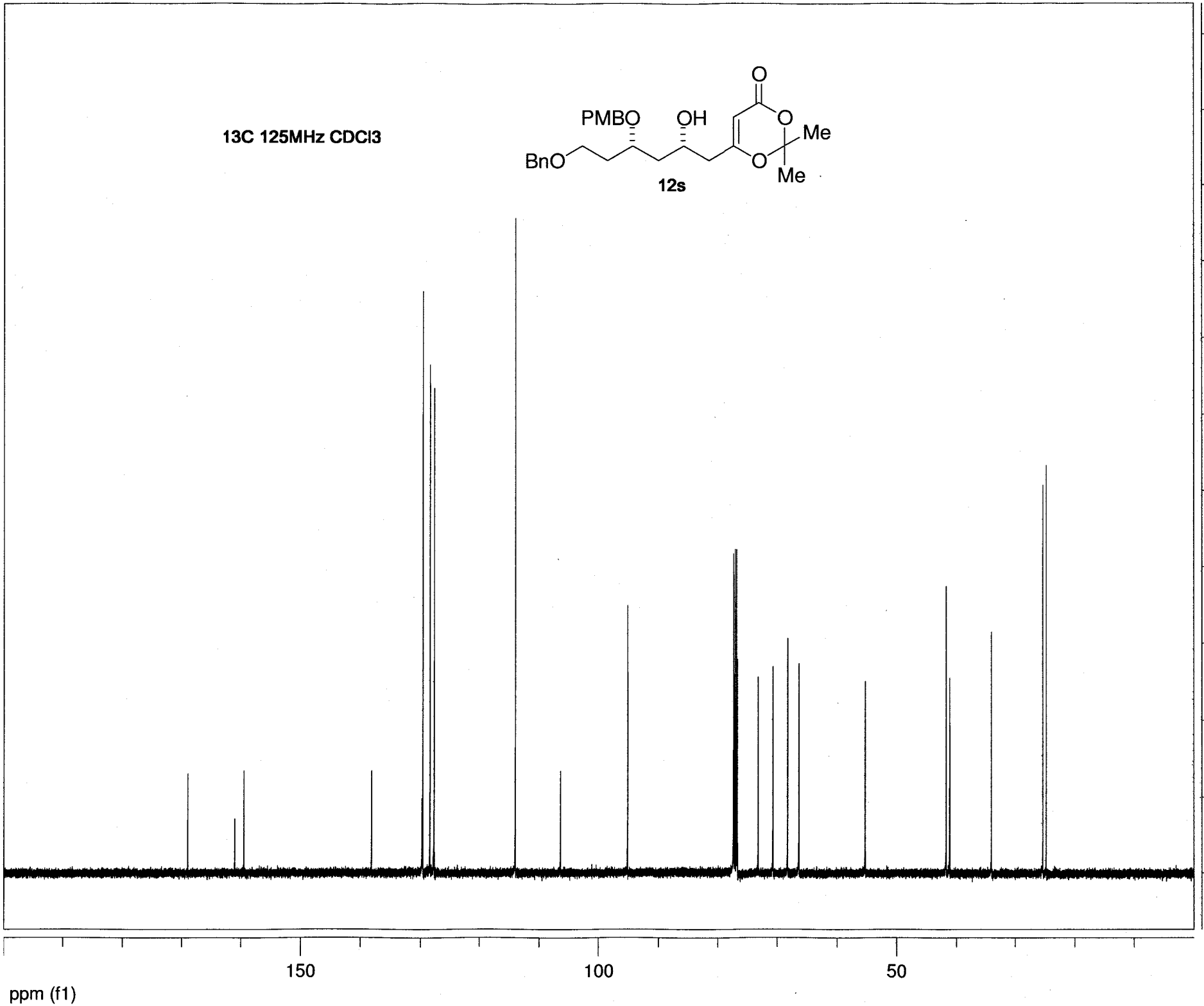
6.0
5.0
4.0
3.0
2.0
1.0
5.57
2.29
2.26
1.00
1.05
2.45
1.14
1.05
1.16
3.56
0.67
2.55
1.08
1.07
1.11
1.24
9.95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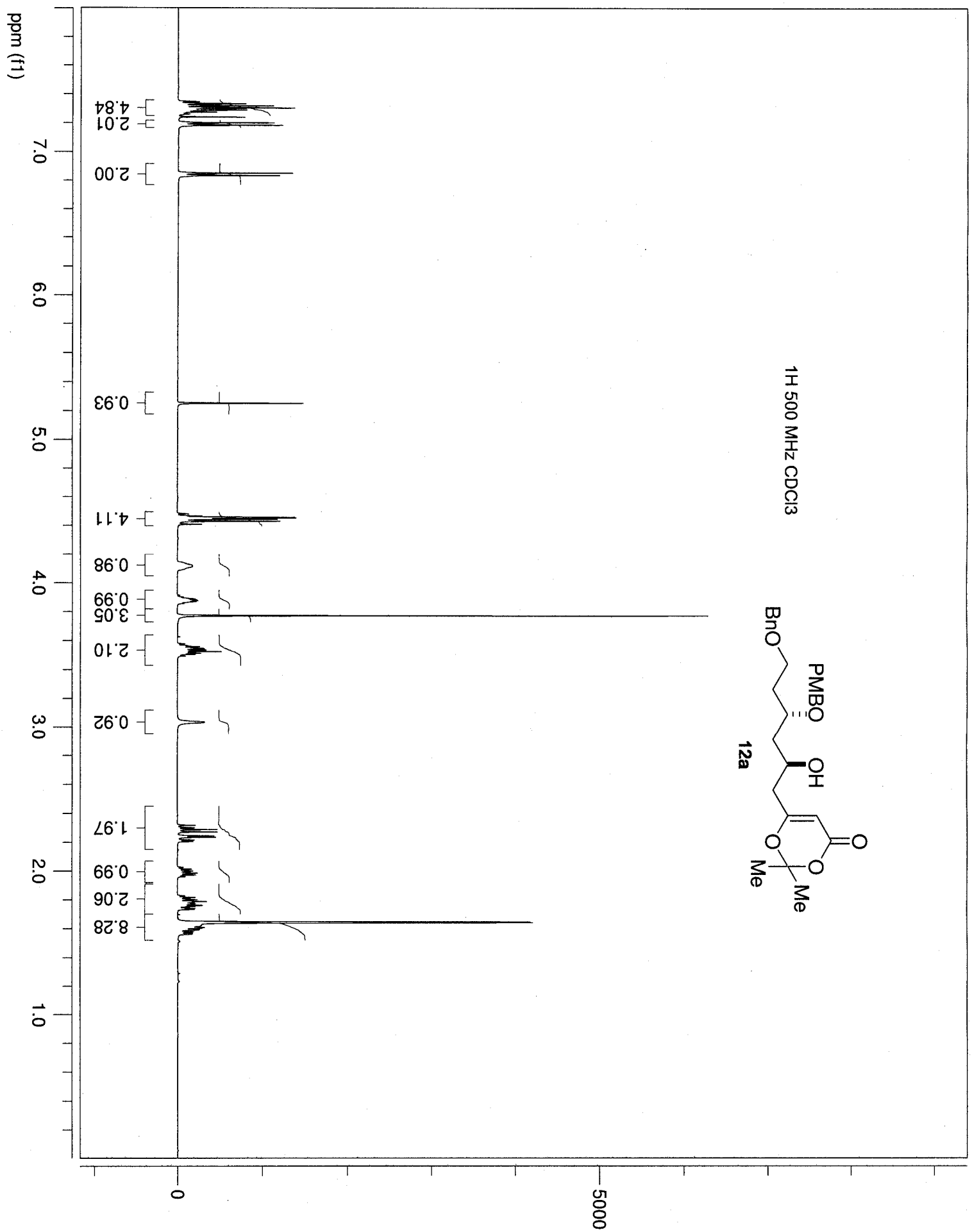
OH
O
O
Me
BnO
O
Me
12s
150
100
50
ppm (f1)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PMBO
OH
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O
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Me
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2.00
0.93
4.11
0.98
0.99
3.05
2.10
0.92
1.97
0.99
2.06
8.28
ppm (f1)
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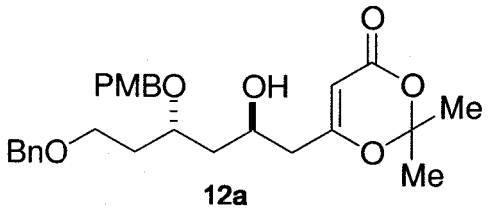
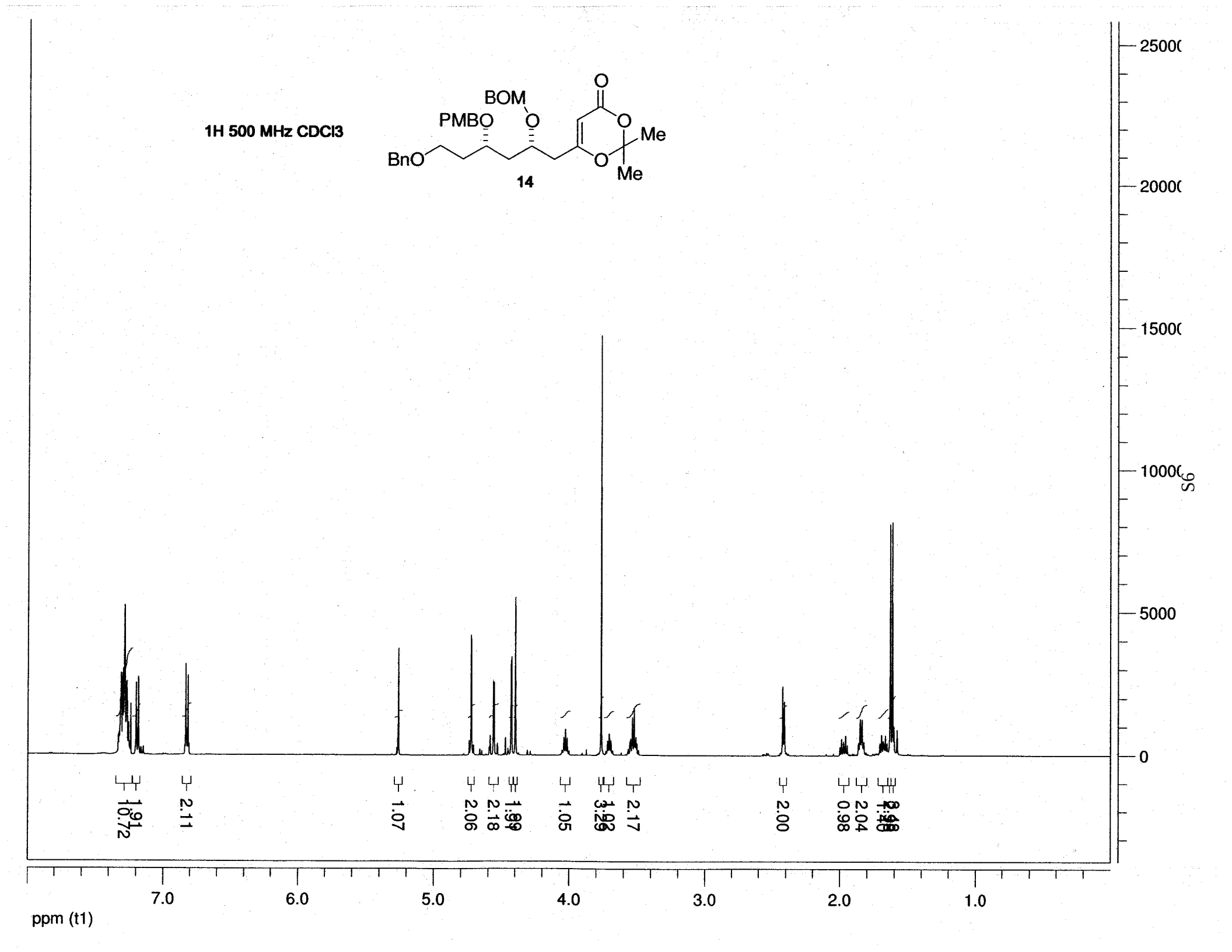
5.0
4.0
3.0
2.0
1.0
0
5000

13C 125 MHz CDCl3



12a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BOM
PMBO
O
BnO
O
O
Me
O
Me
14
25000
20000
15000
10000
5000
0
10.72
1.91
2.11
1.07
2.06
2.18
1.99
1.05
3.29
2.17
2.00
0.98
2.04
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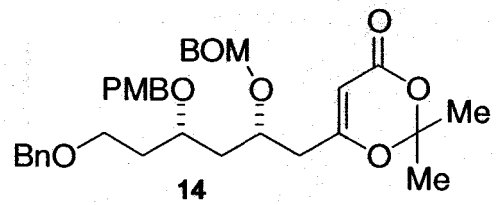
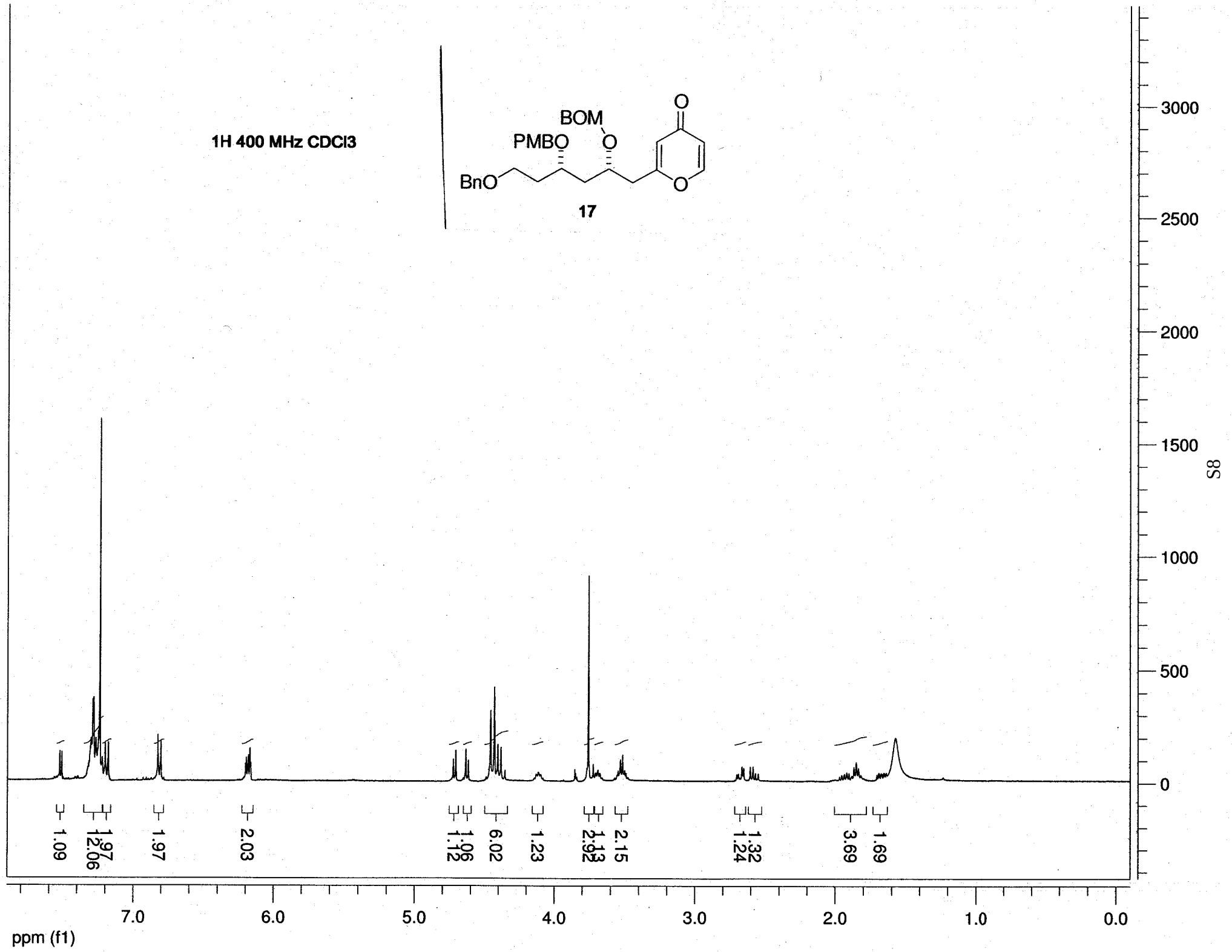
6.0
5.0
4.0
3.0
2.0
1.0
ppm (t1)

13C 125 MHz CDCl3



14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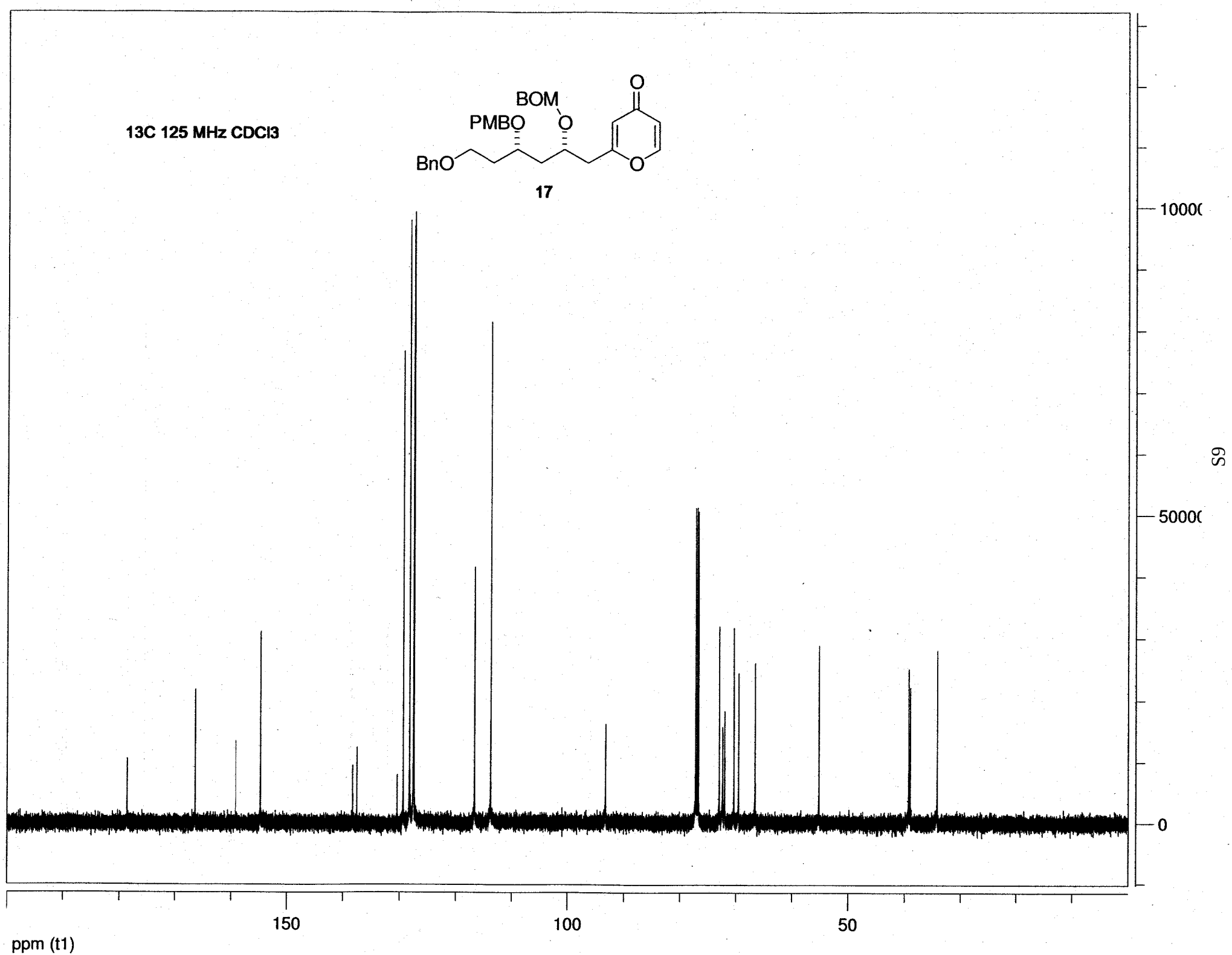
BOM
PMBO
O
BnO
O
O
17
3000
2500
2000
1500
1000
500
0
1.09
12.06
1.97
1.97
2.03
1.12
1.06
6.02
1.23
2.92
1.13
2.15
1.24
1.32
3.69
1.69
7.0
6.0
5.0
4.0
3.0
2.0
1.0
0.0
ppm (f1)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PMBO
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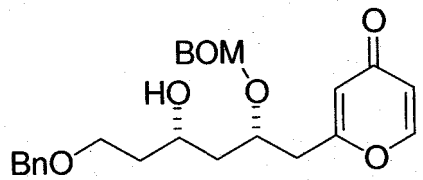
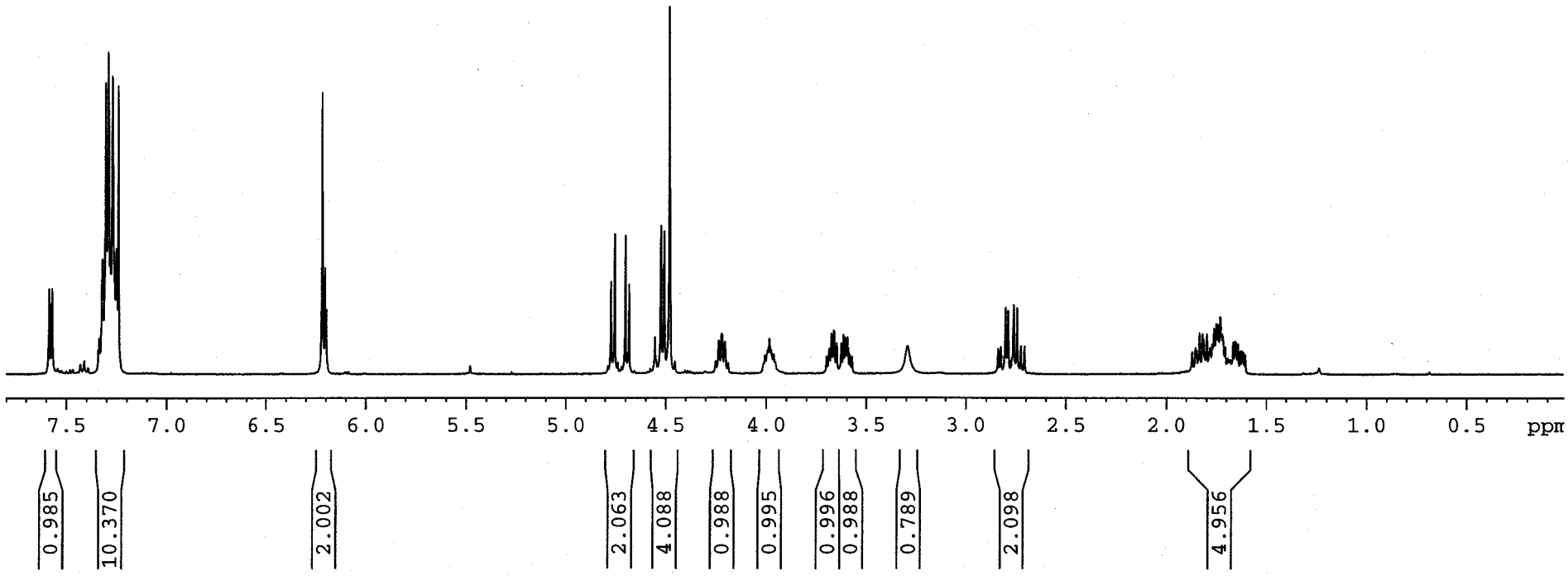
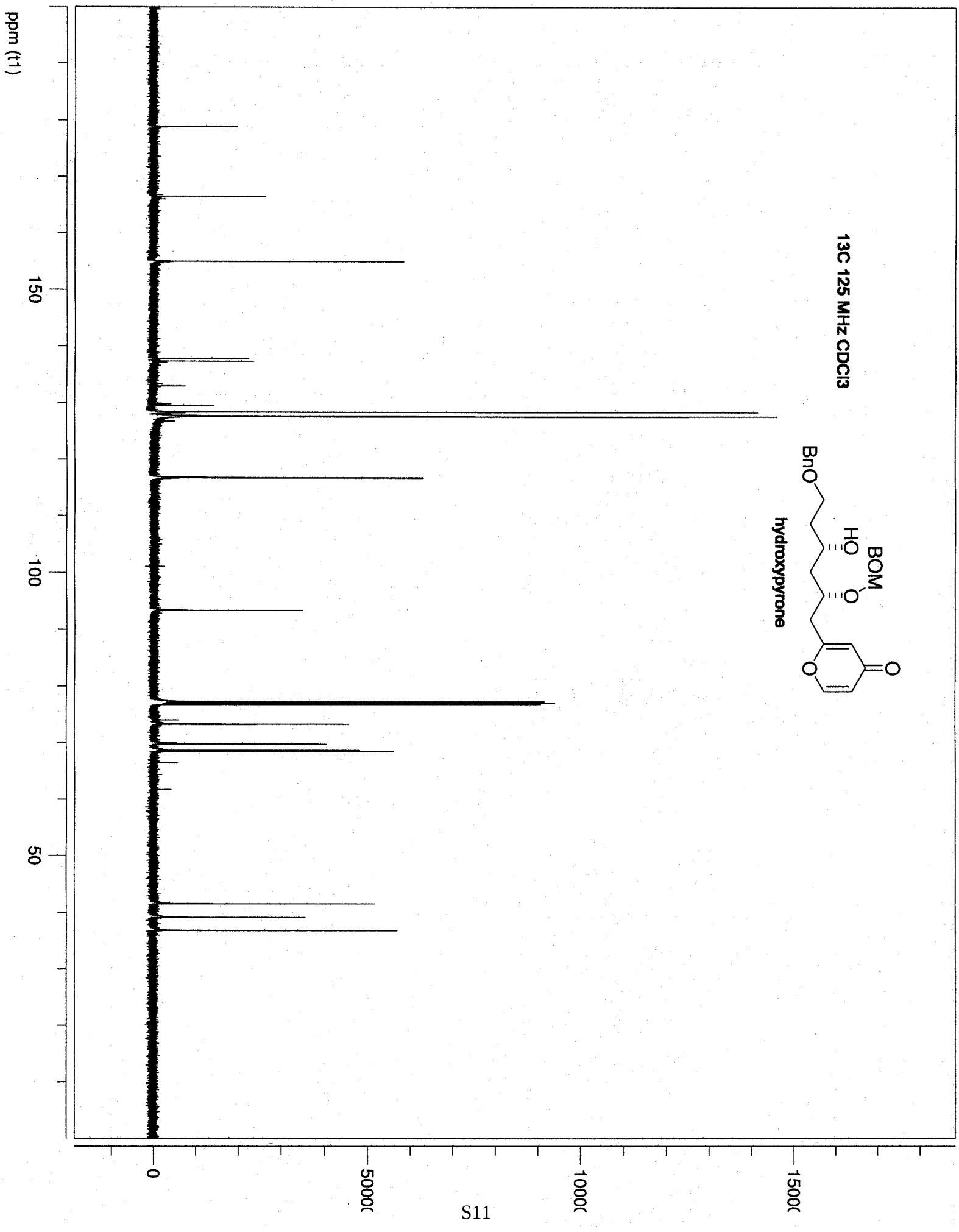
BnO
O
O
17
10000
50000
0
150
100
50
ppm (t1)

1H 400 MHz CDCl3



hydroxypyrone

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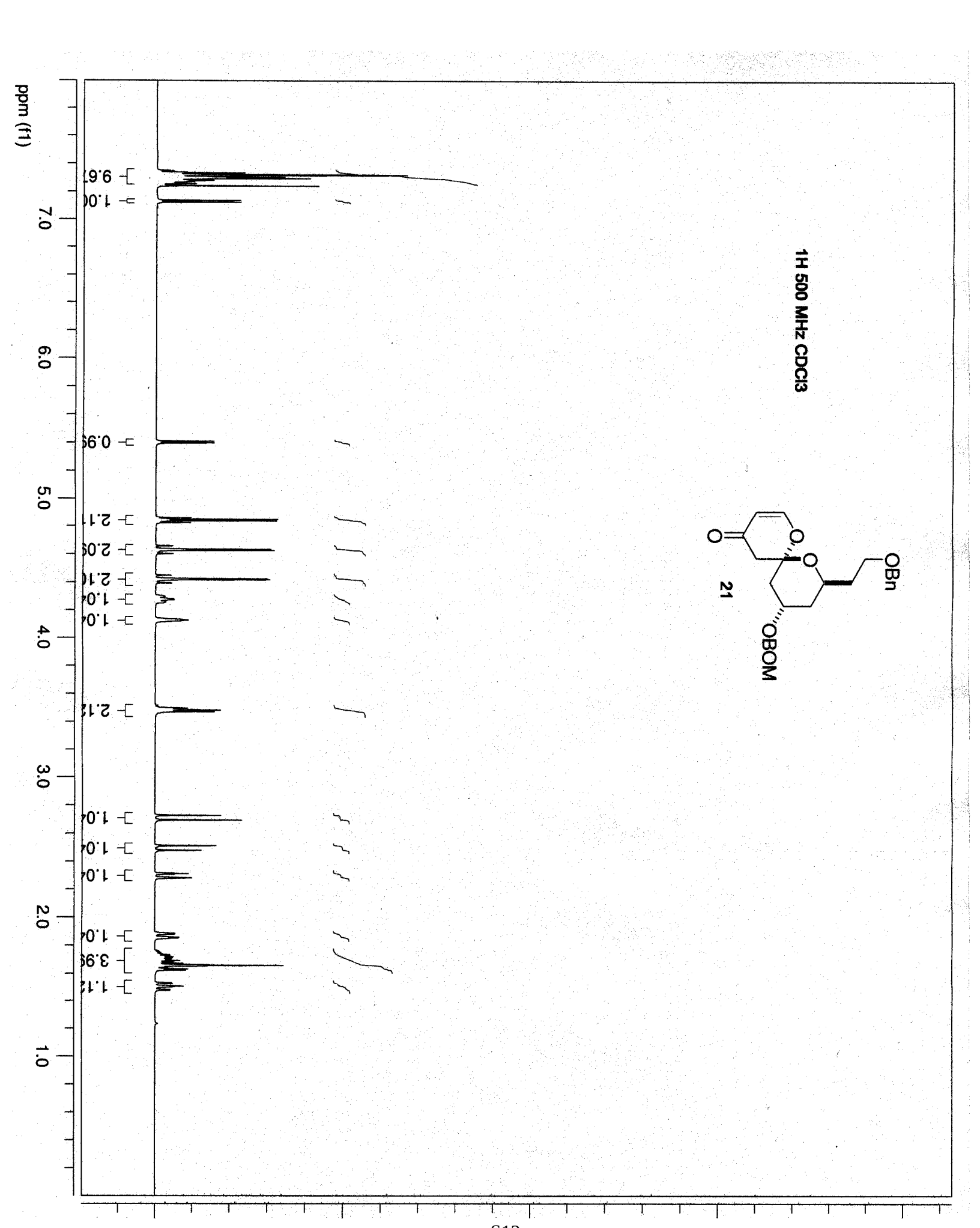
hydroxypyrone
BnO
HO
BOM
O
O
O
ppm (t1)
150
100
50
0
5000
10000
15000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OBn
OBOM
ppm (f1)
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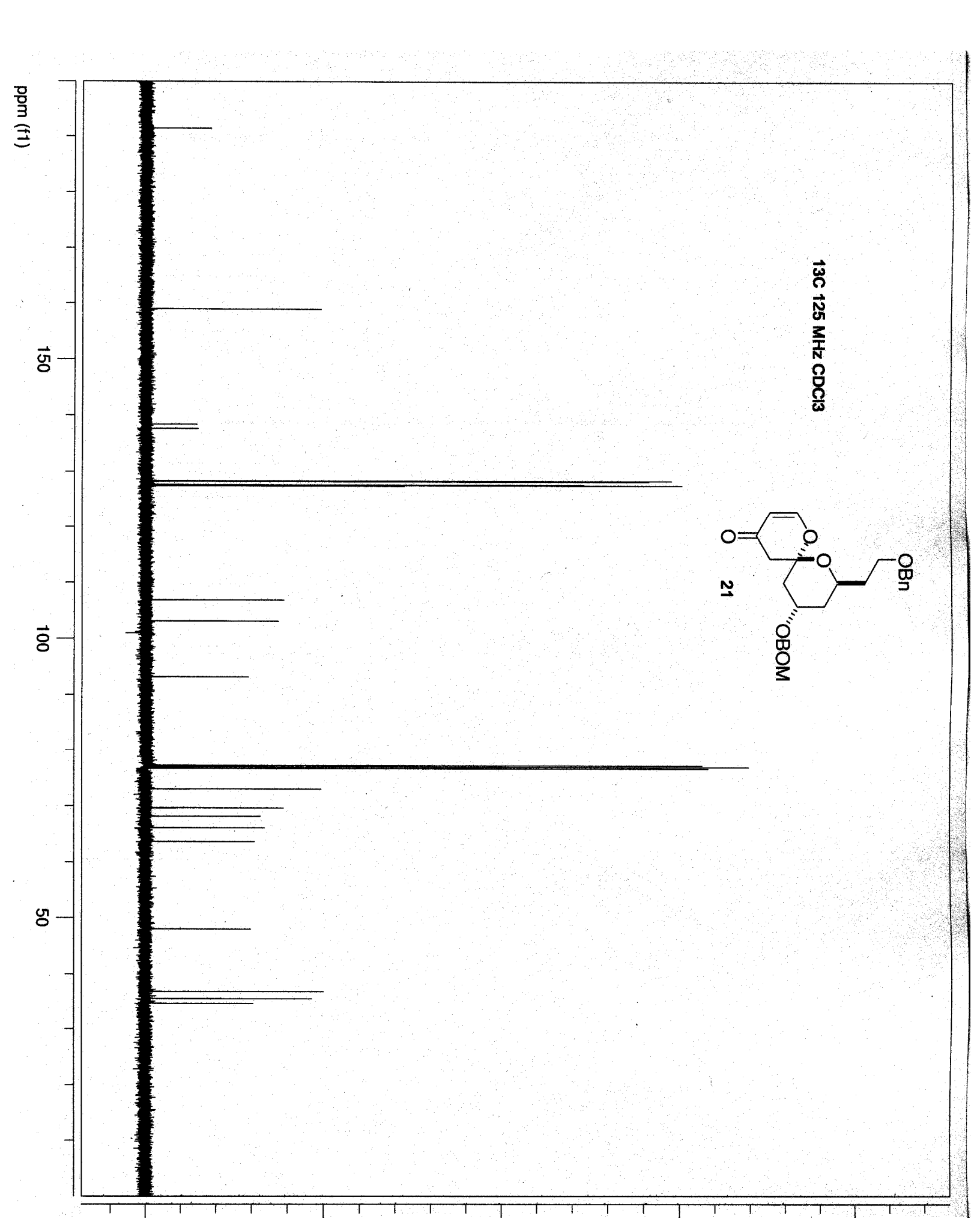
6.0
5.0
4.0
3.0
2.0
1.0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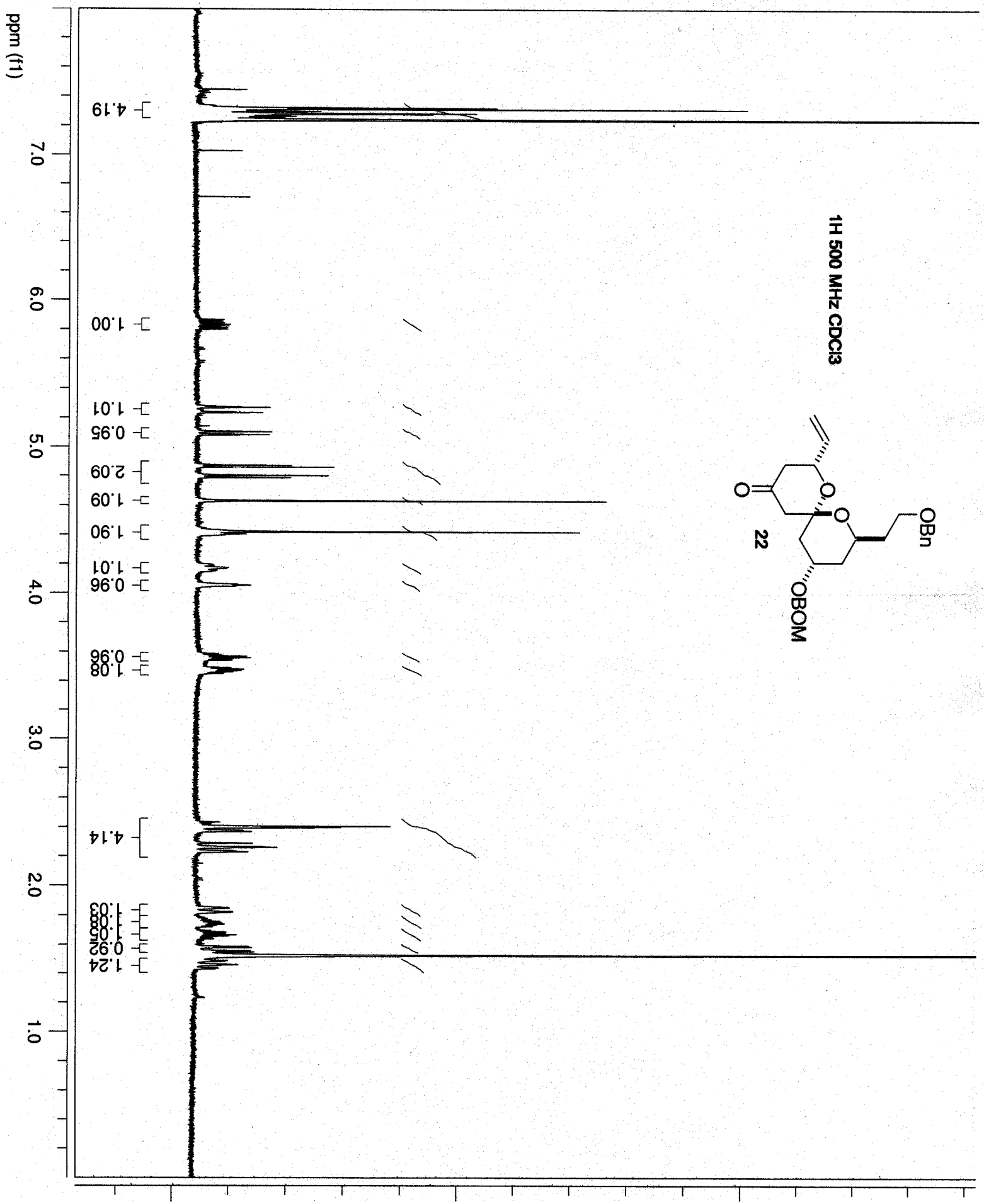
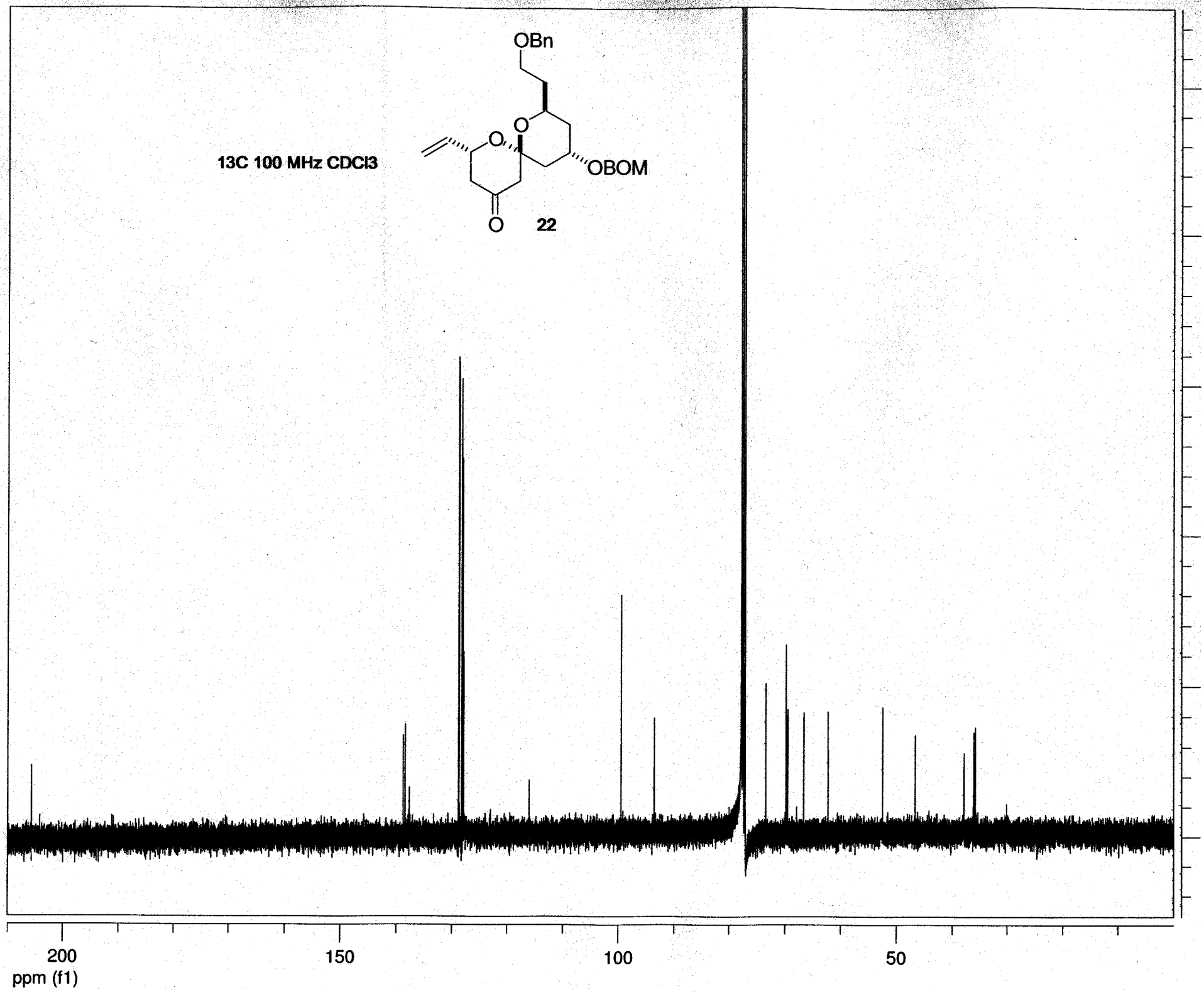
OBn
OBOM
O
O
O
ppm (f1)
150
100
50


1H 500 MHz CDCl3
22
OBn
OBOM
ppm (f1)
7.0
6.0
5.0
4.0
3.0
2.0
1.0
4.19
1.00
1.01
0.95
2.09
1.09
1.90
1.01
0.96
1.08
0.96
4.14
1.03
1.24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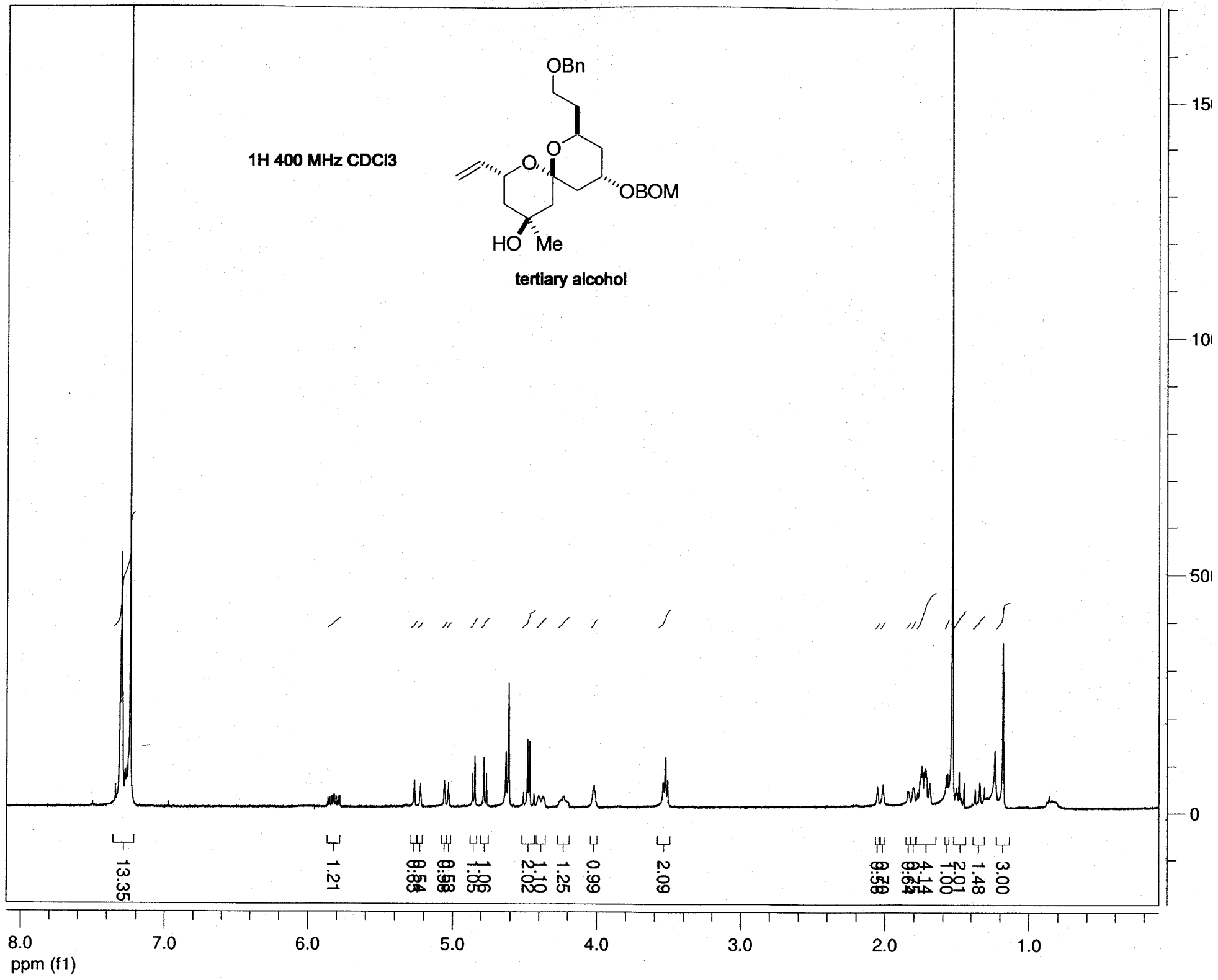
O
O
OBOM
O
22
200
150
100
50
ppm (f1)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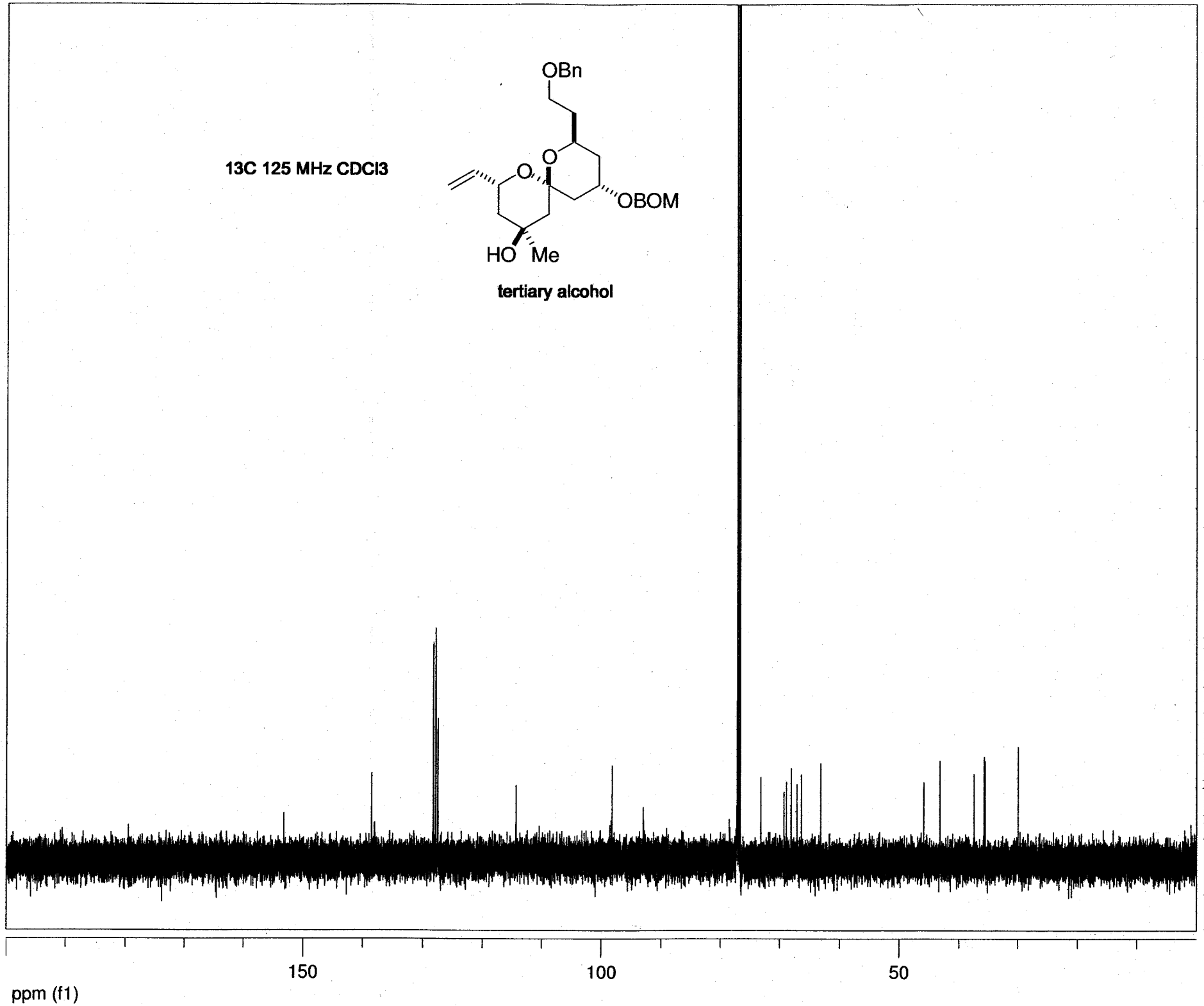
OBn
O
O
OBOM
HO
Me
tertiary alcohol
13.35
1.21
0.54
0.58
1.05
2.02
1.25
0.99
2.09
0.58
4.14
1.00
1.48
3.00
8.0
7.0
6.0
5.0
4.0
3.0
2.0
1.0
ppm (f1)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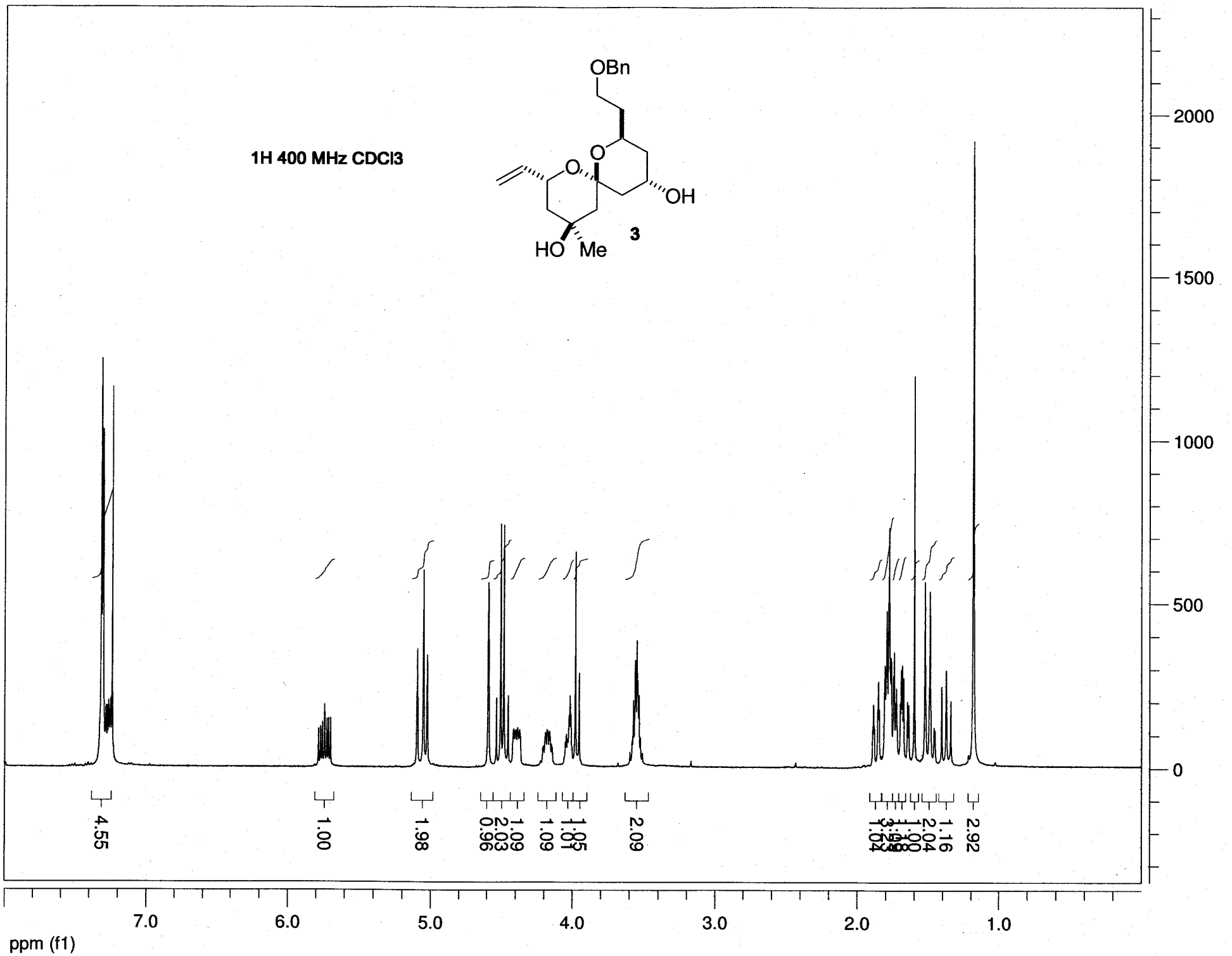
O
O
OBOM
HO
Me
tertiary alcohol
150
100
50
ppm (f1)
0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OBn
O
O
OH
HO
Me
3
2000
1500
1000
500
0
4.55
1.00
1.98
0.96
2.03
1.09
1.09
1.01
1.05
2.09
1.04
3.92
3.88
1.00
2.04
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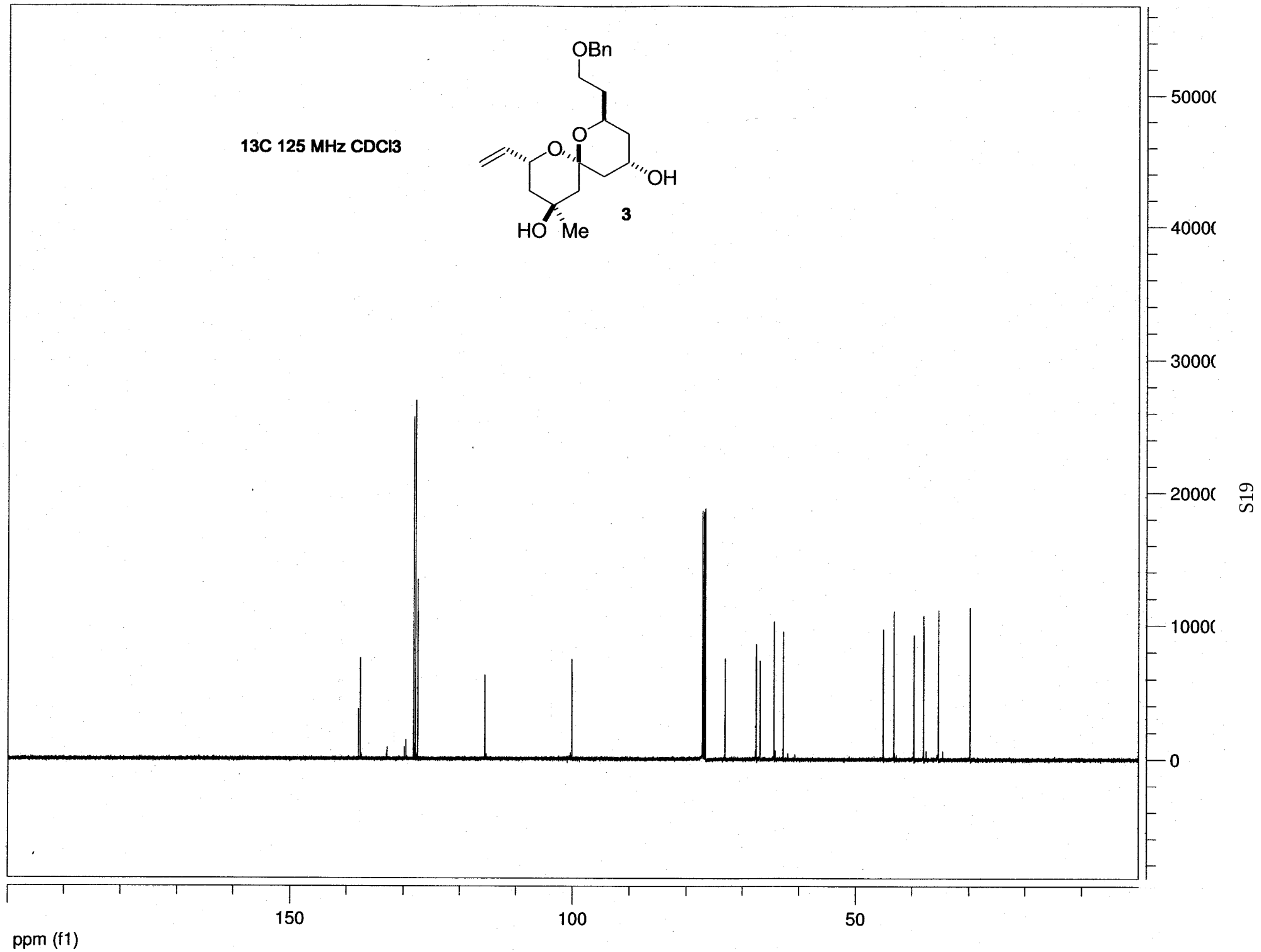
2.92
7.0
6.0
5.0
4.0
3.0
2.0
1.0
ppm (f1)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OBn
O
O
OH
HO
Me
3
50000
40000
30000
20000
10000
0
150
100
50
ppm (f1)