Electronic Supplementary Material (ESI) for Organic & Biomolecular Chemistry.

Supplementary Material for

Development of an efficient route toward meiogynin A-derived dual inhibitors of Bcl-xL and Mcl-1 anti-apoptotic proteins

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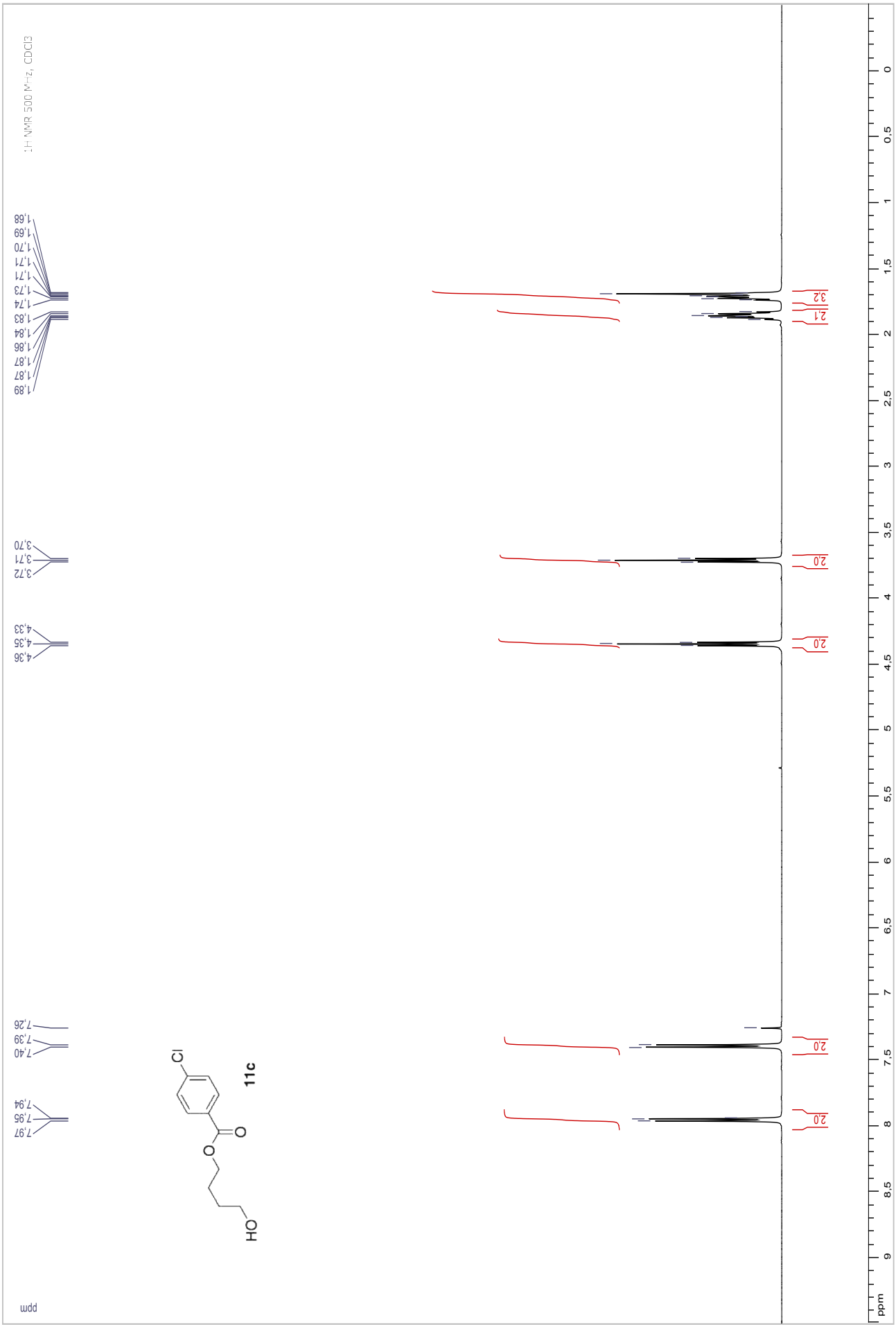
* Corresponding Author Email Adress: fanny.roussi@cnrs.fr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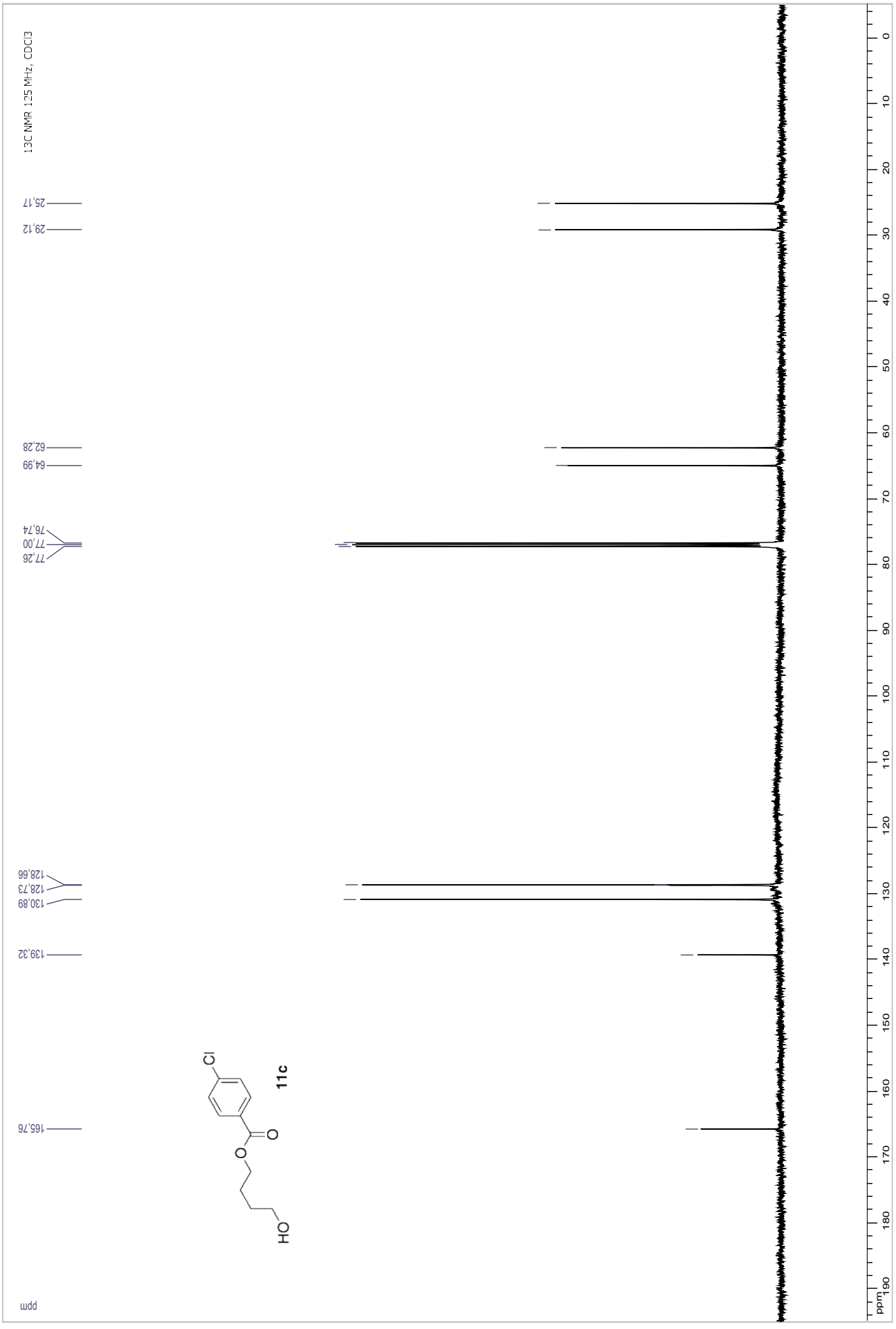
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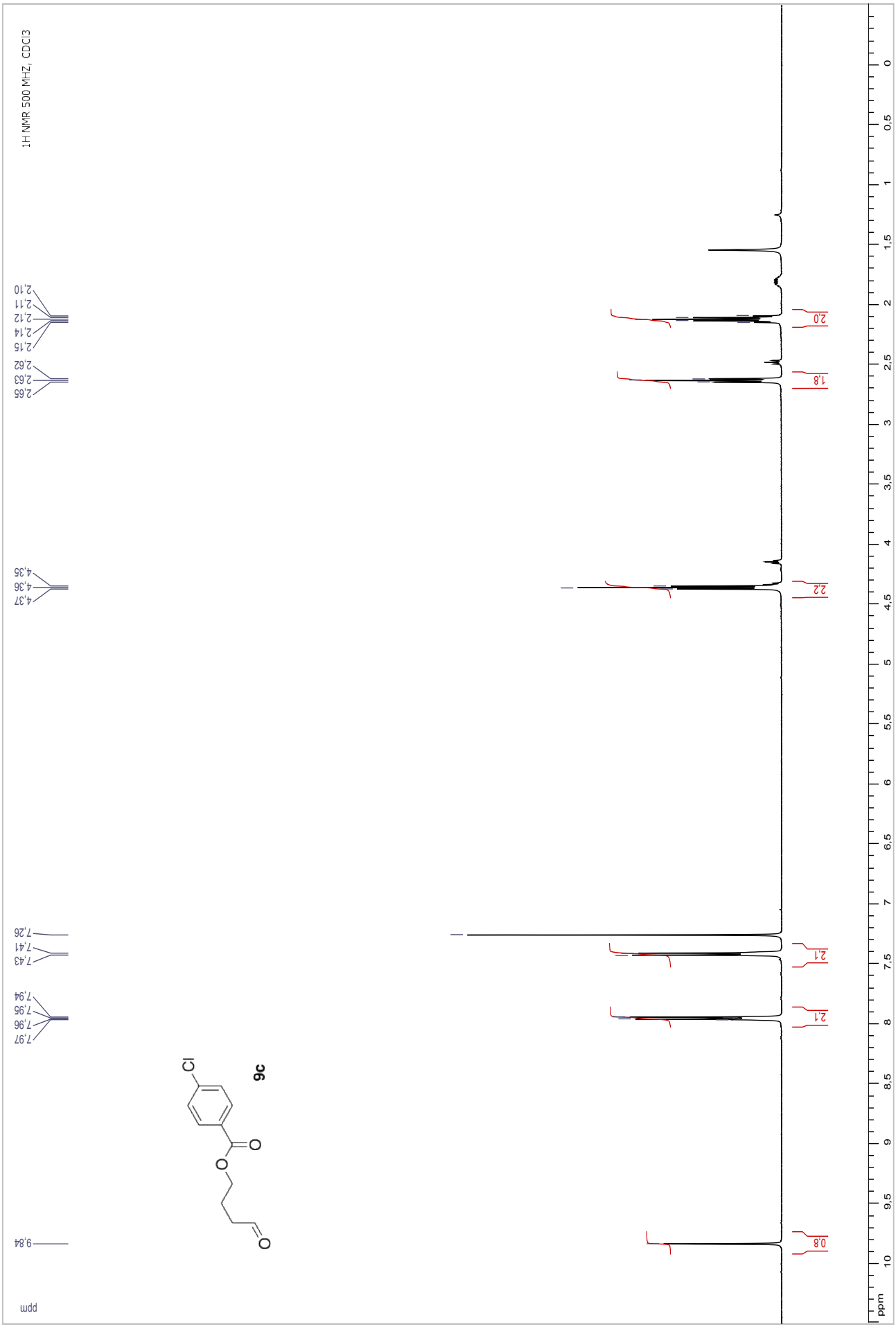
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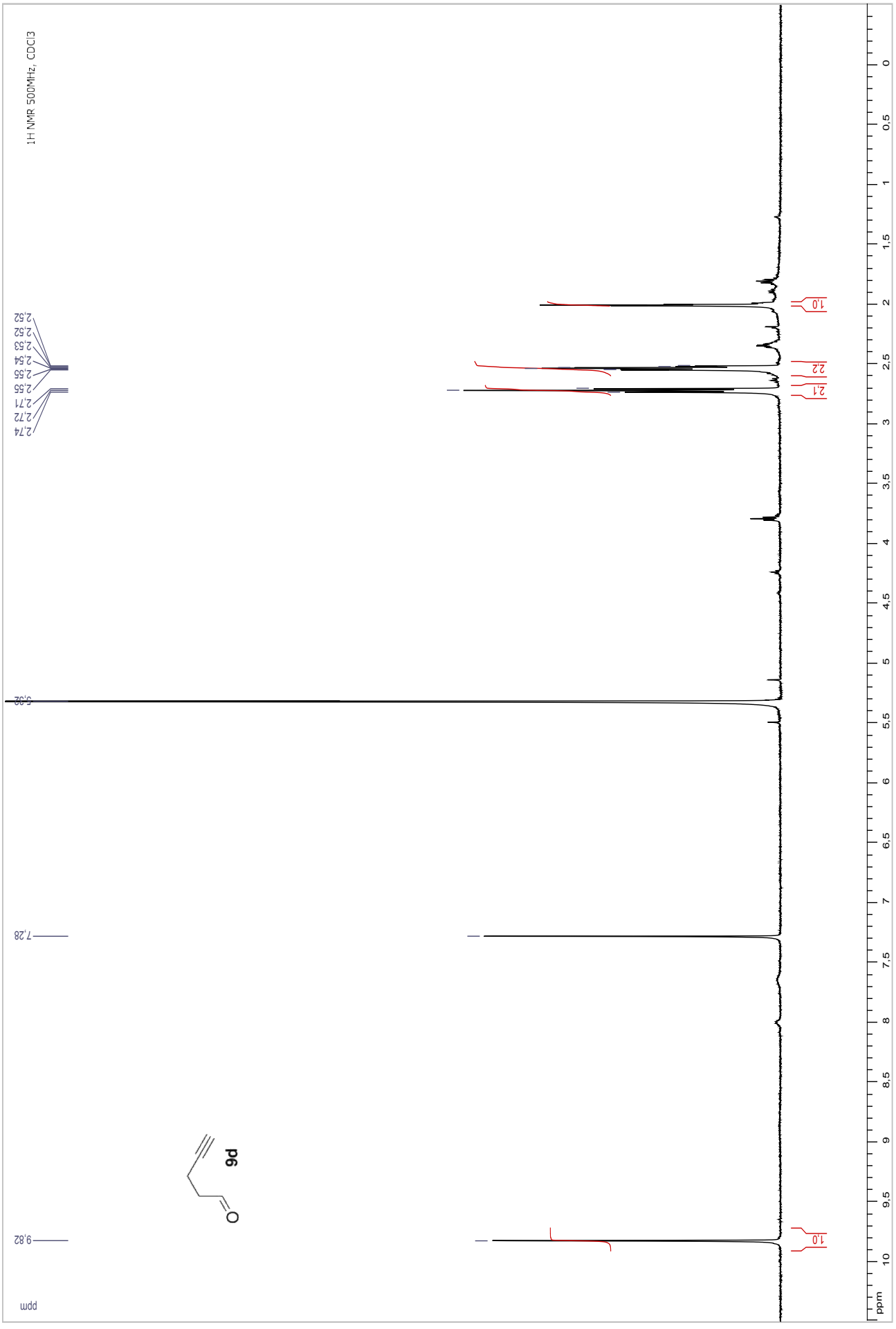
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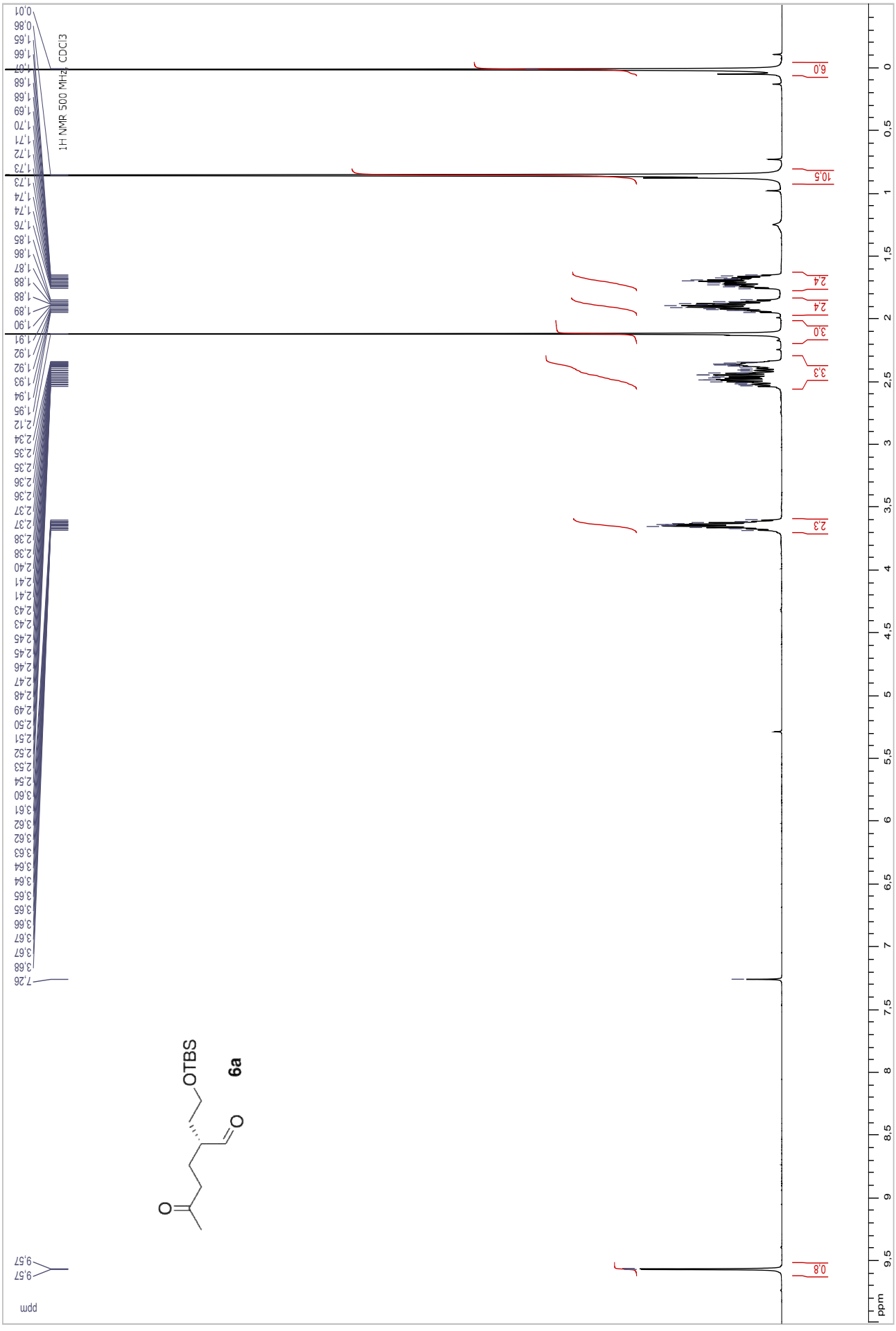
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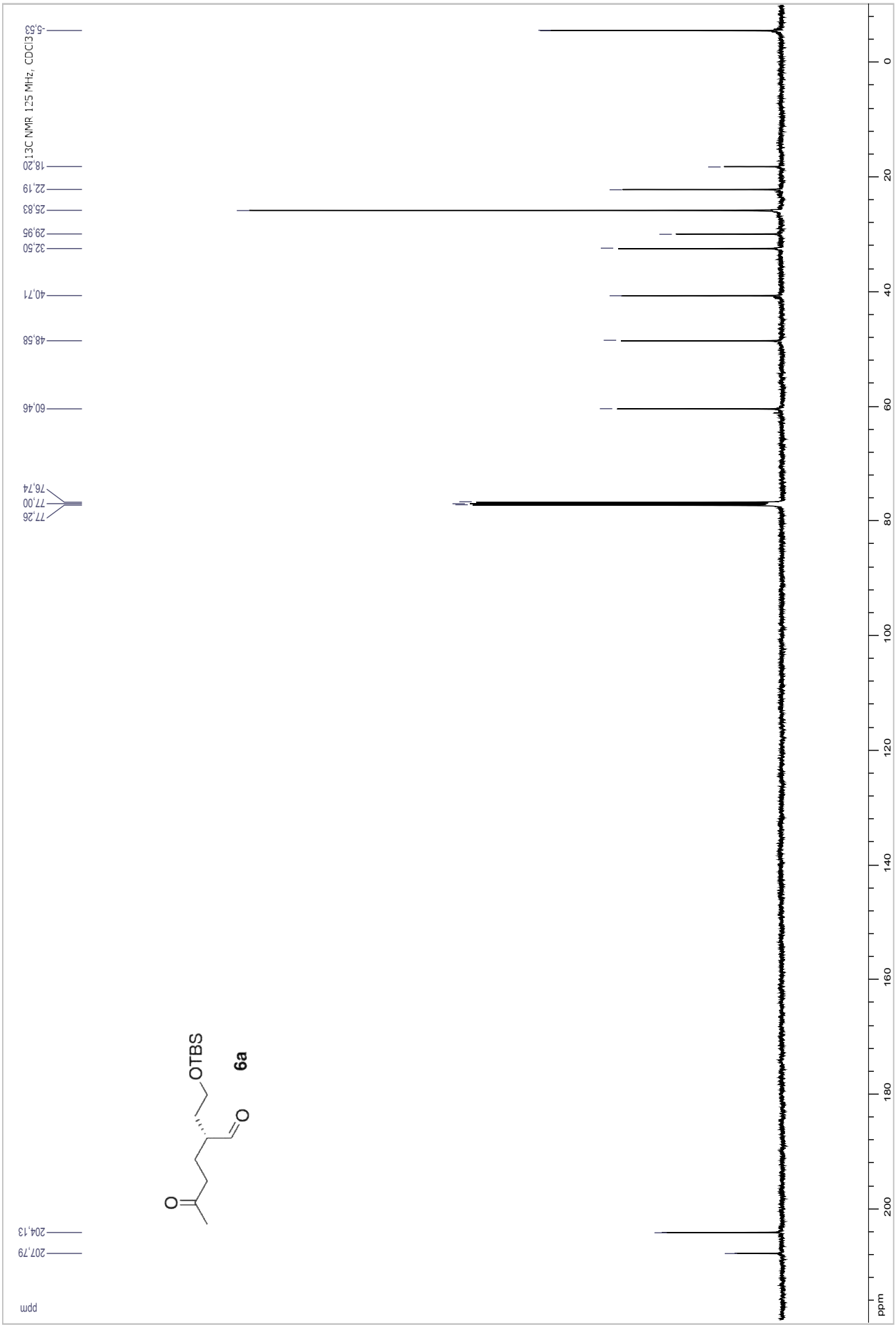
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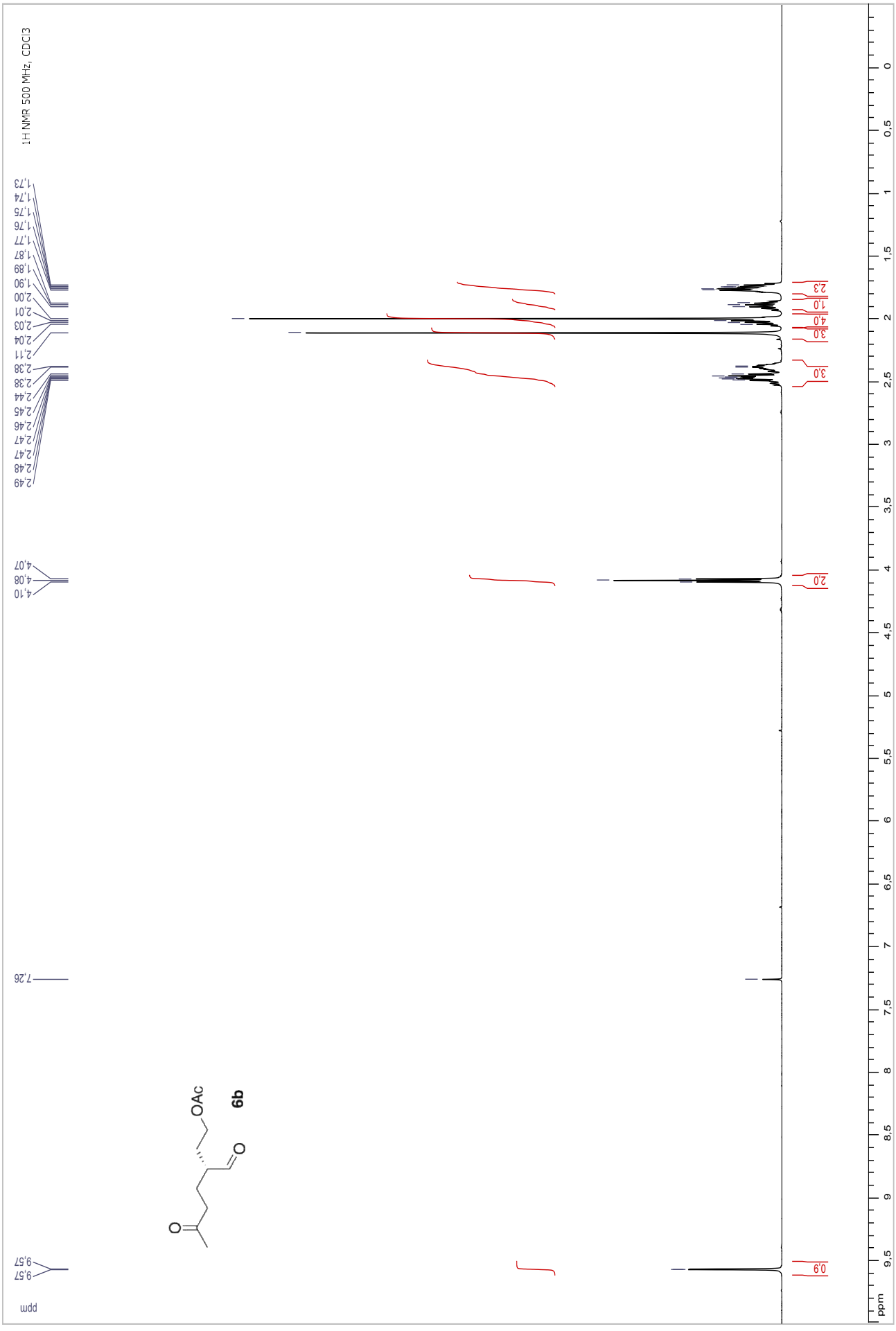
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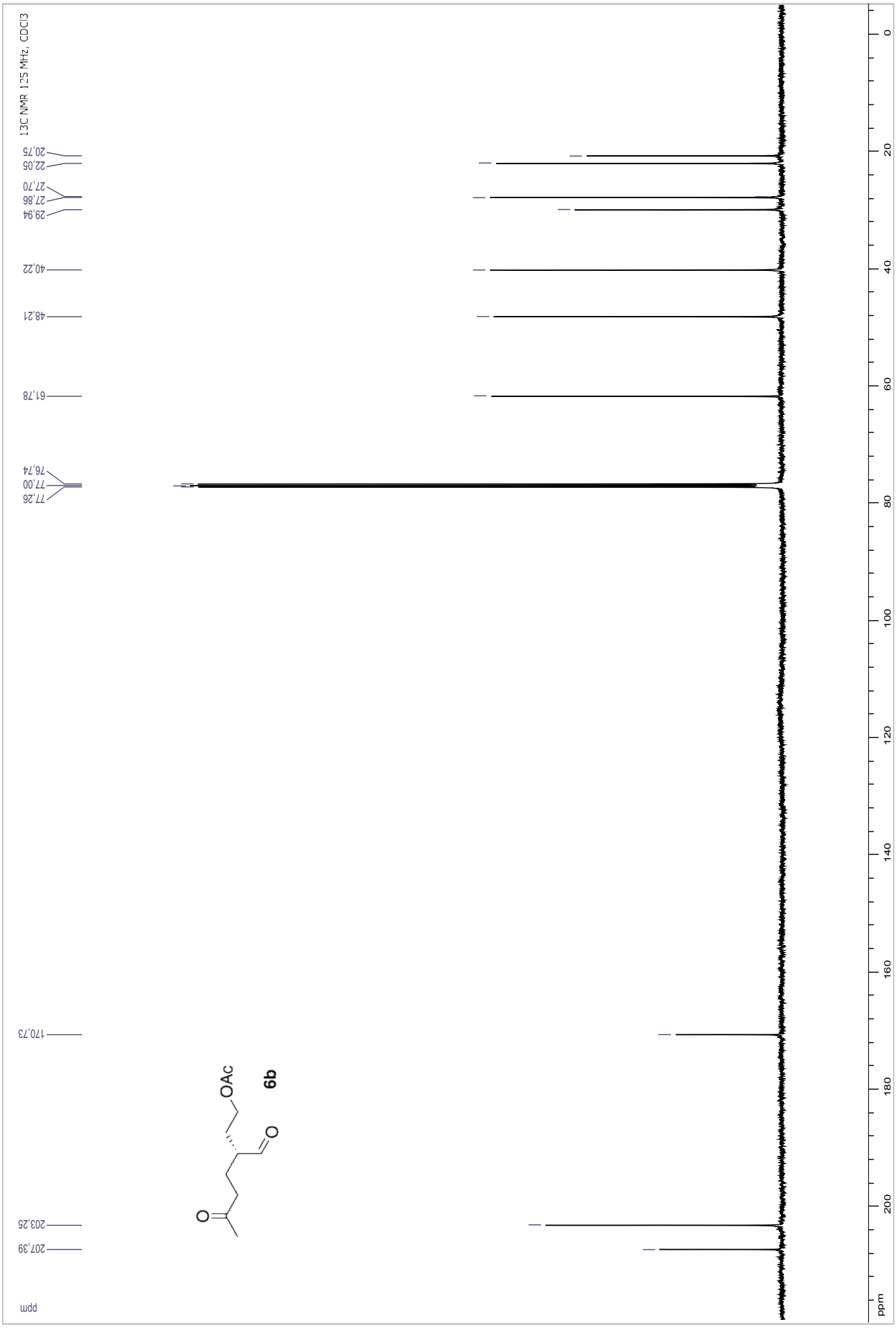
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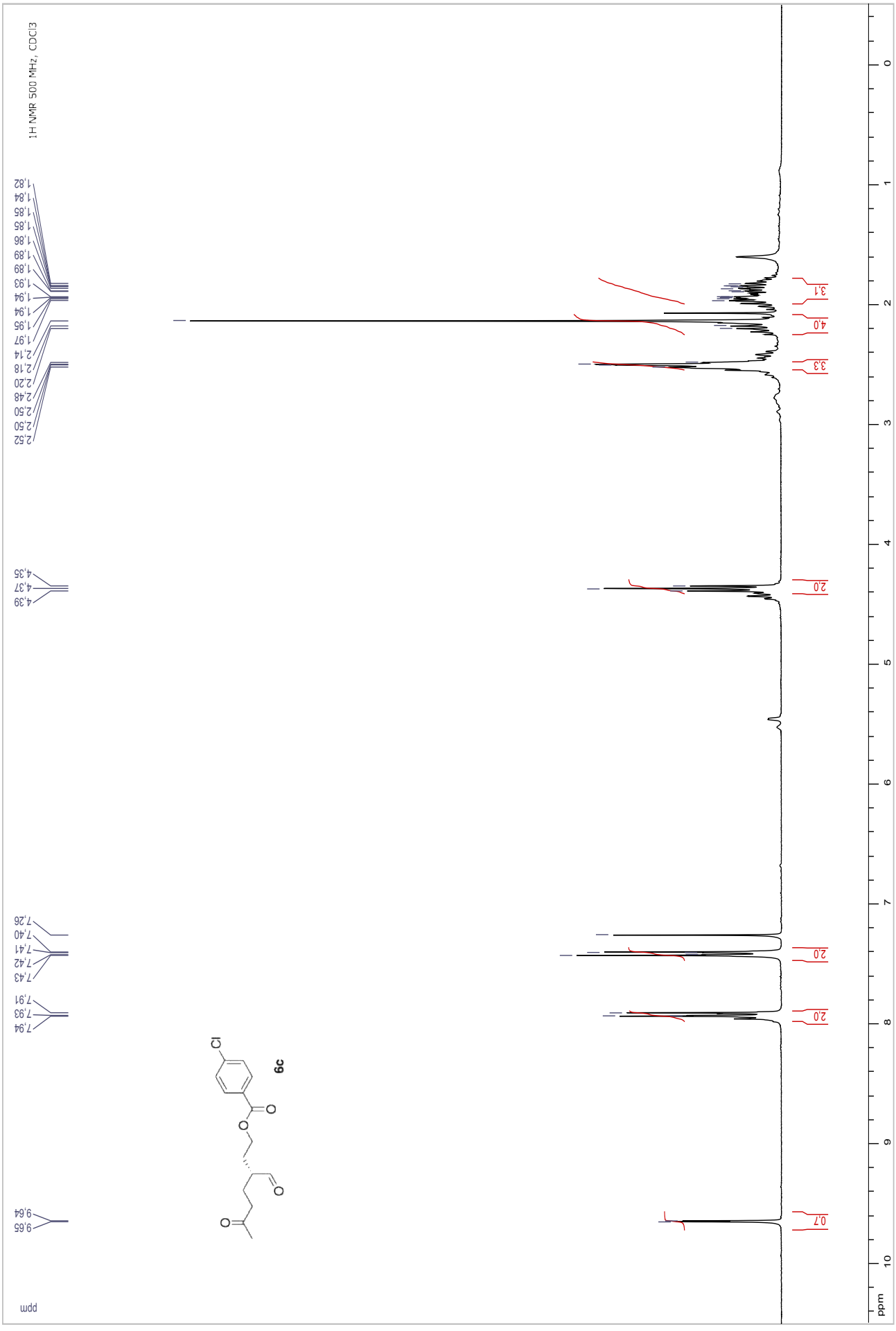
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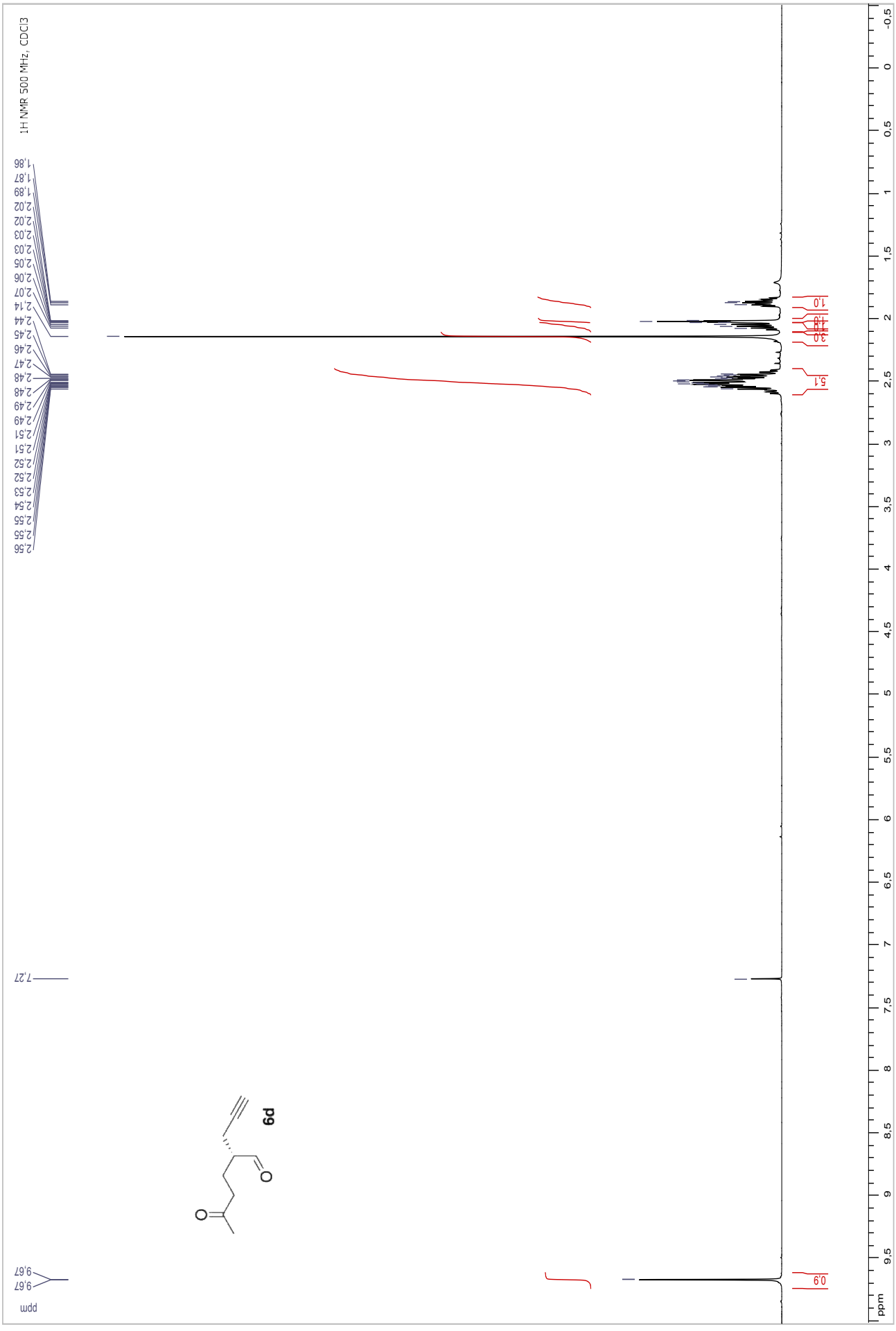
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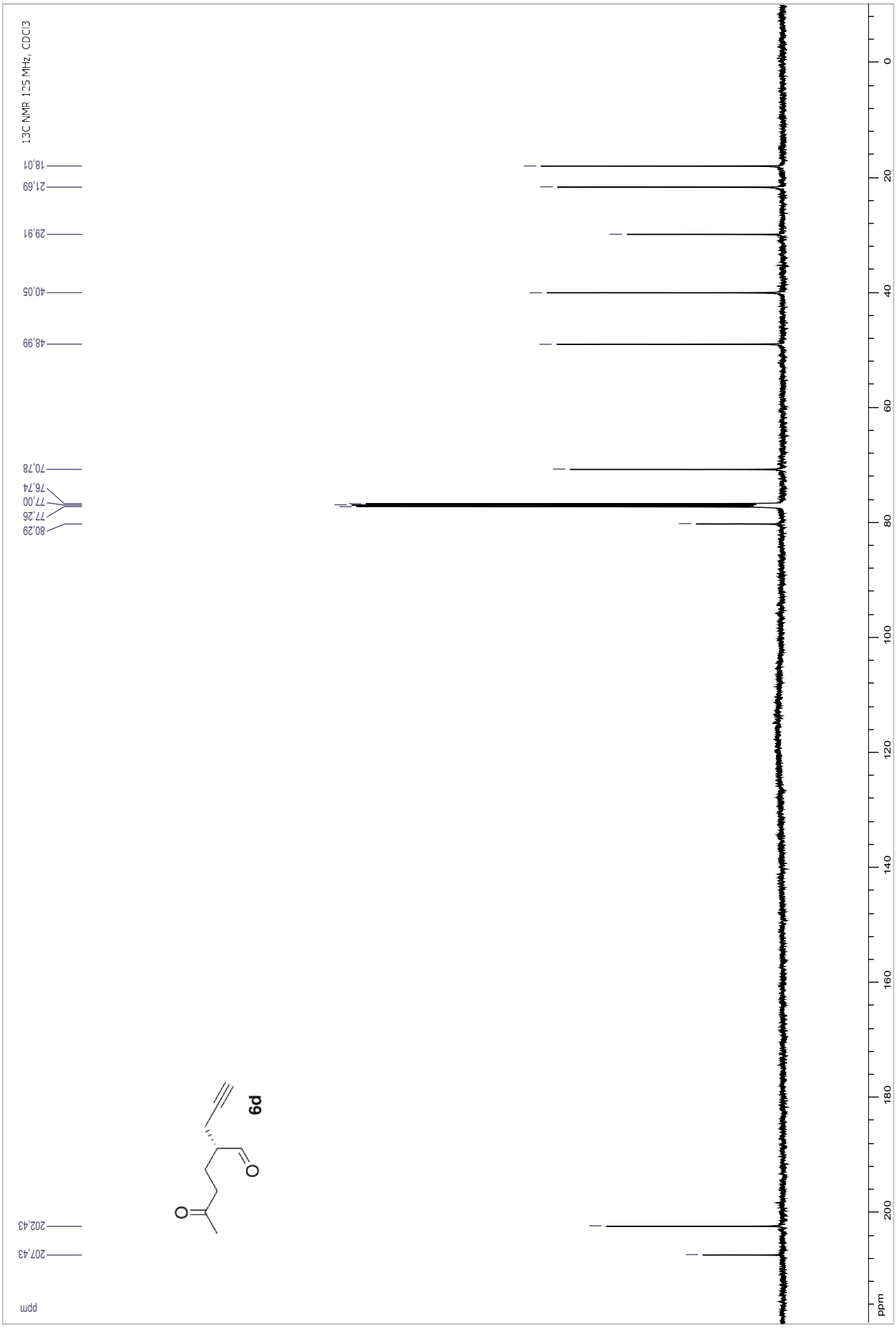
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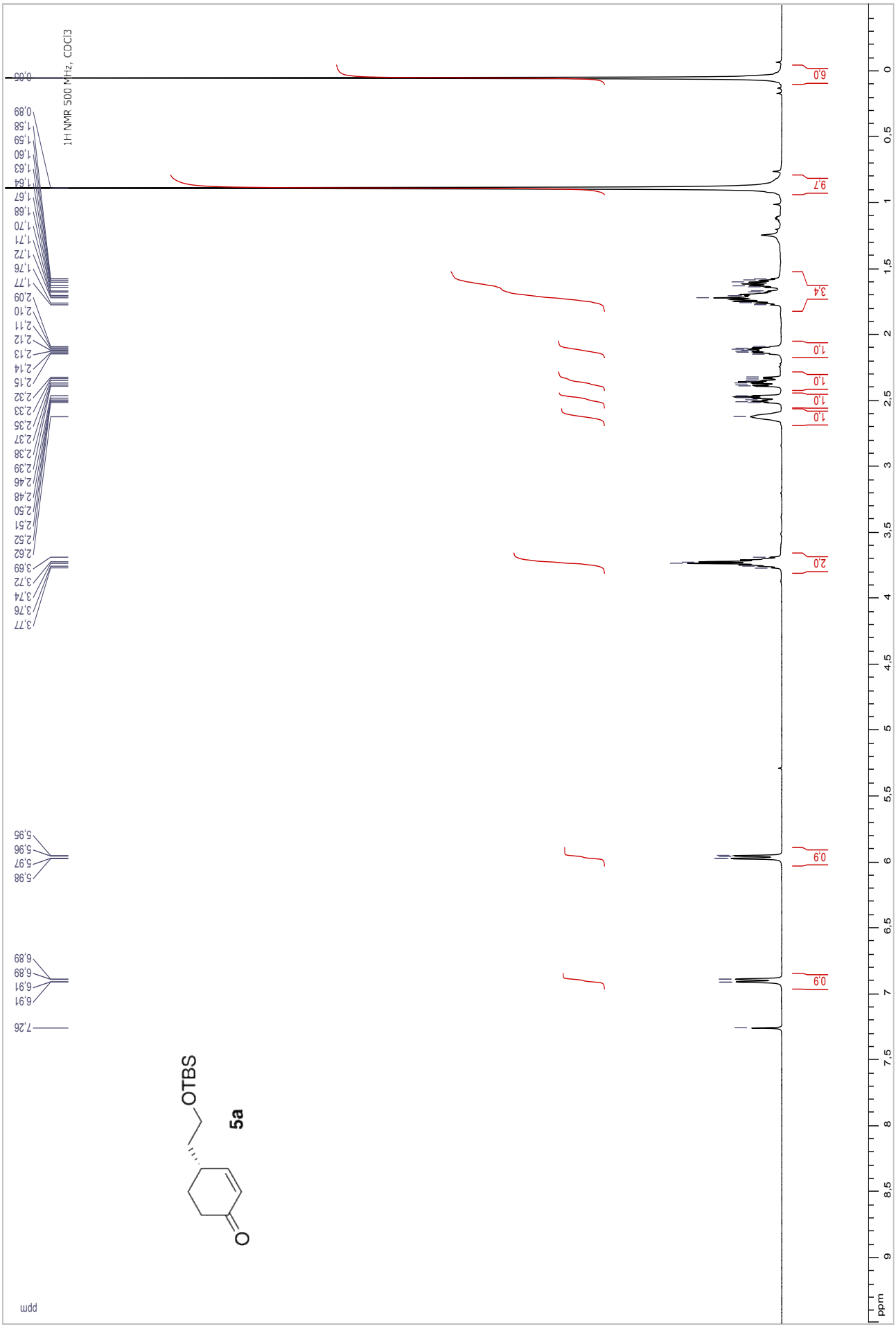
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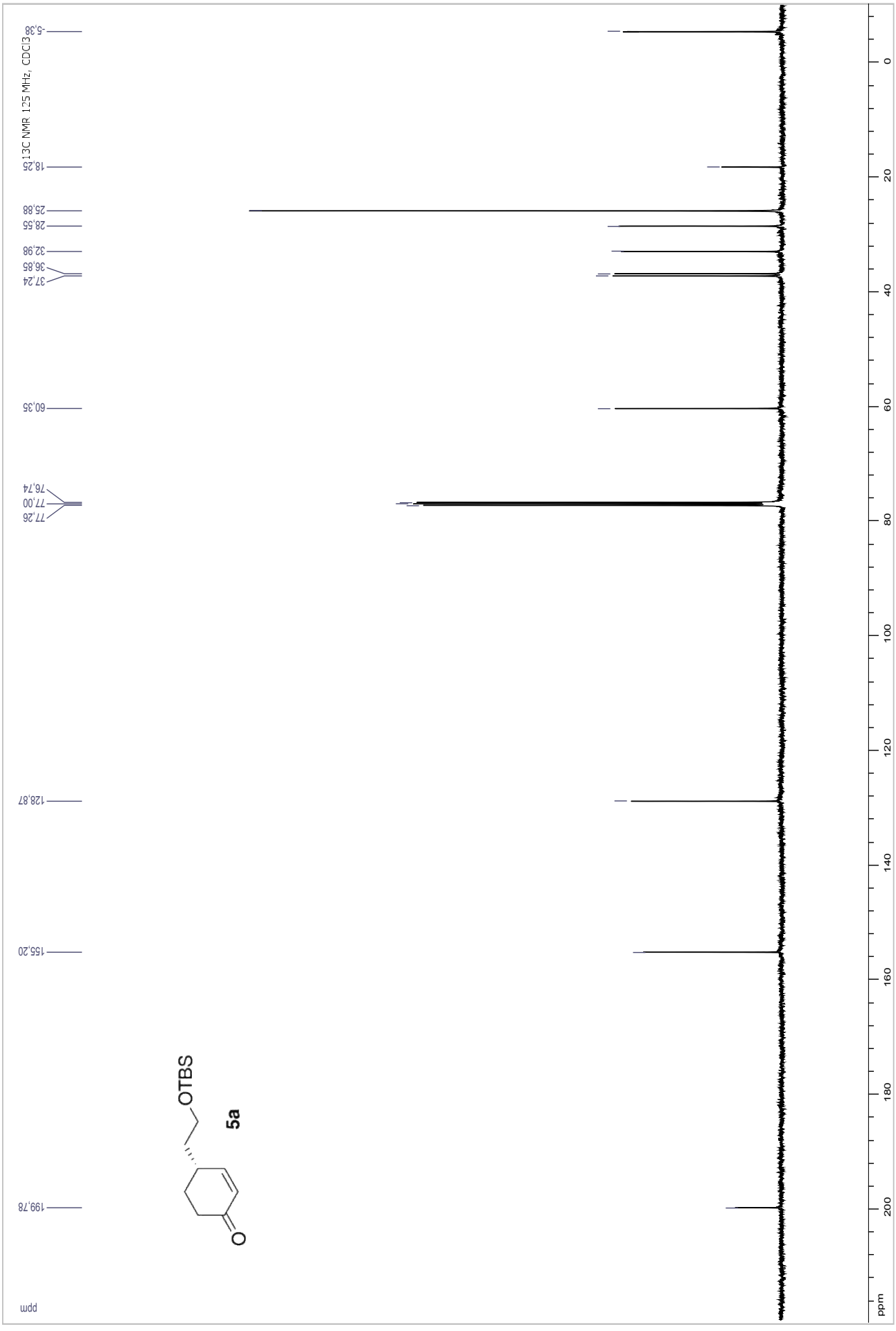
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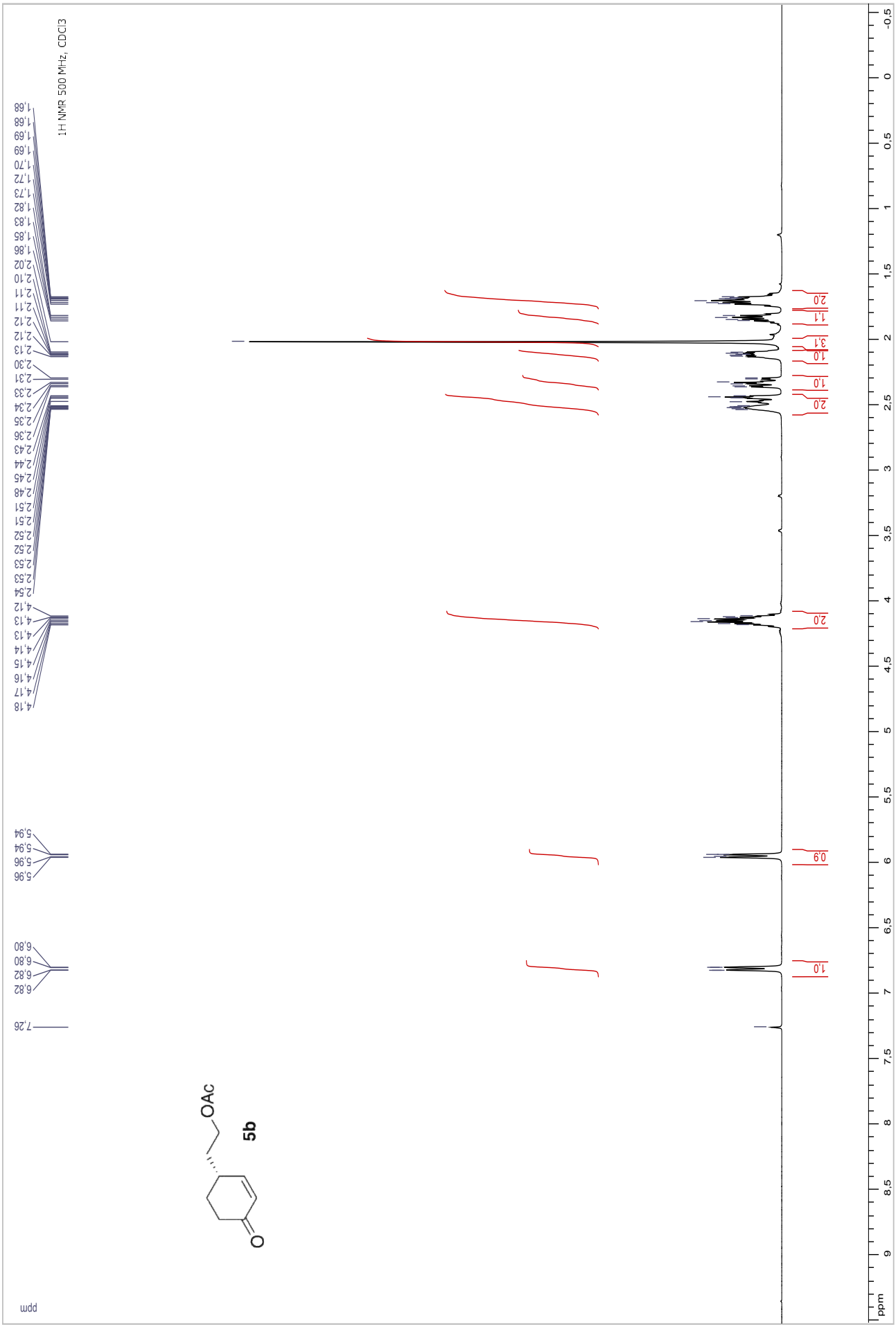
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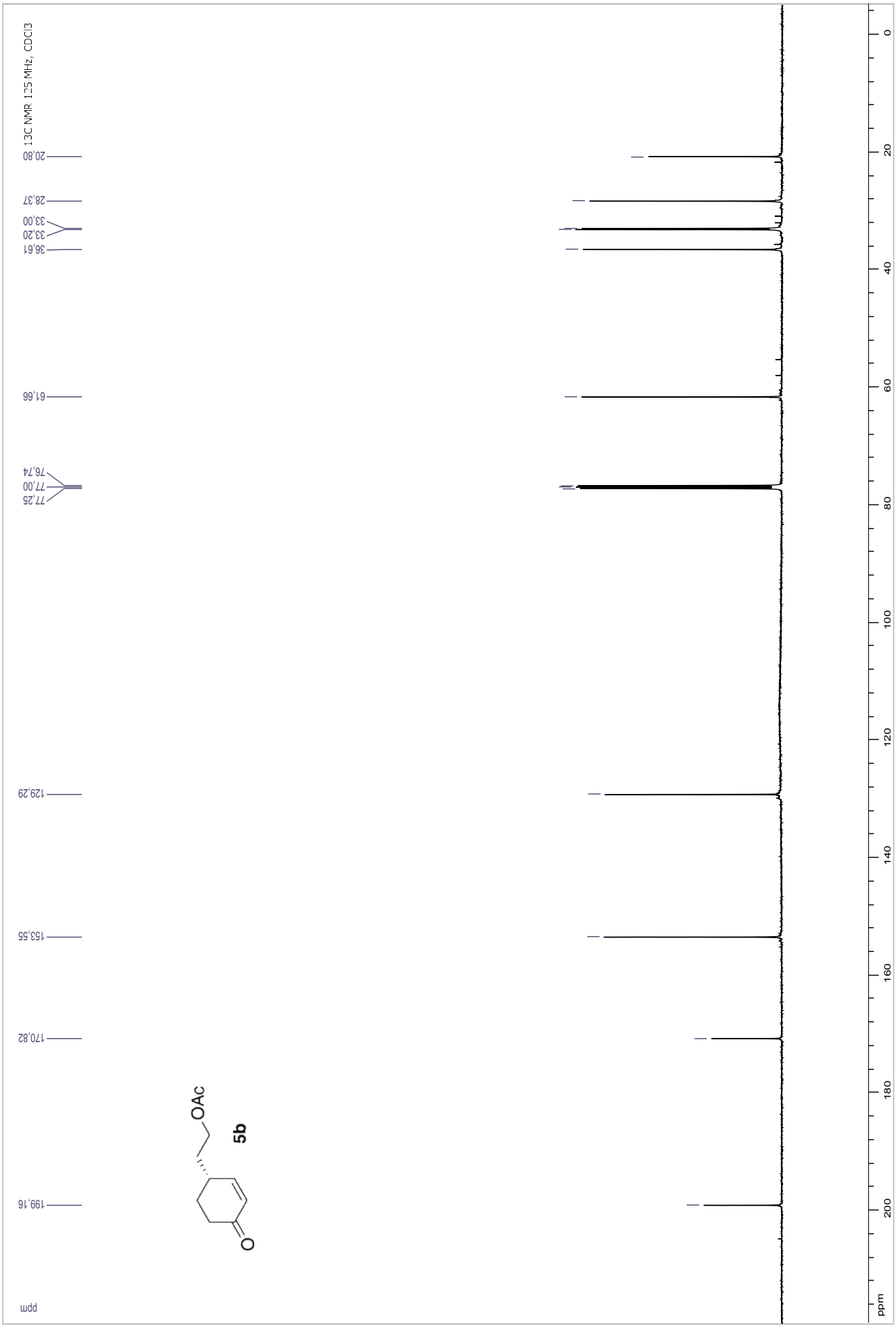
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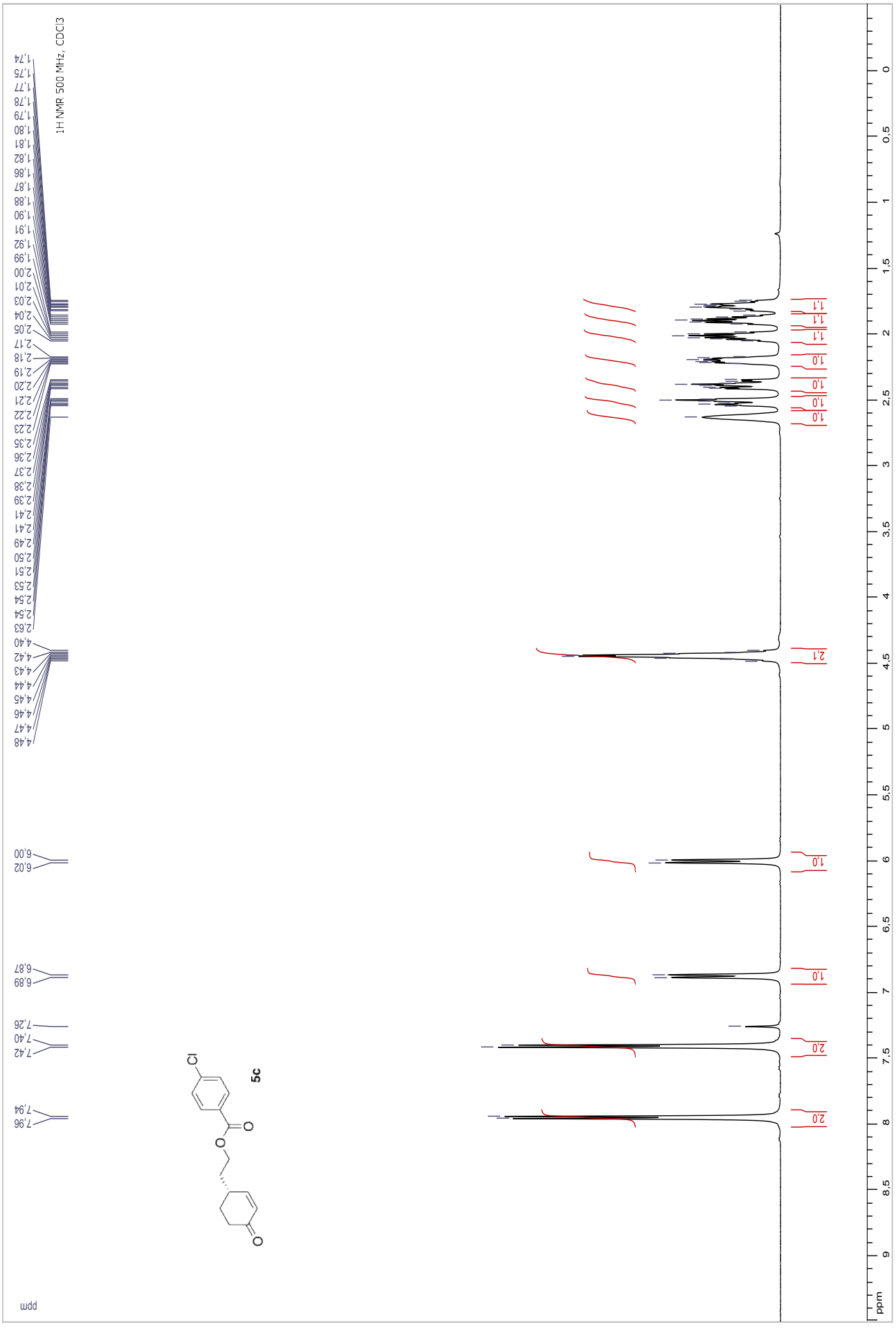
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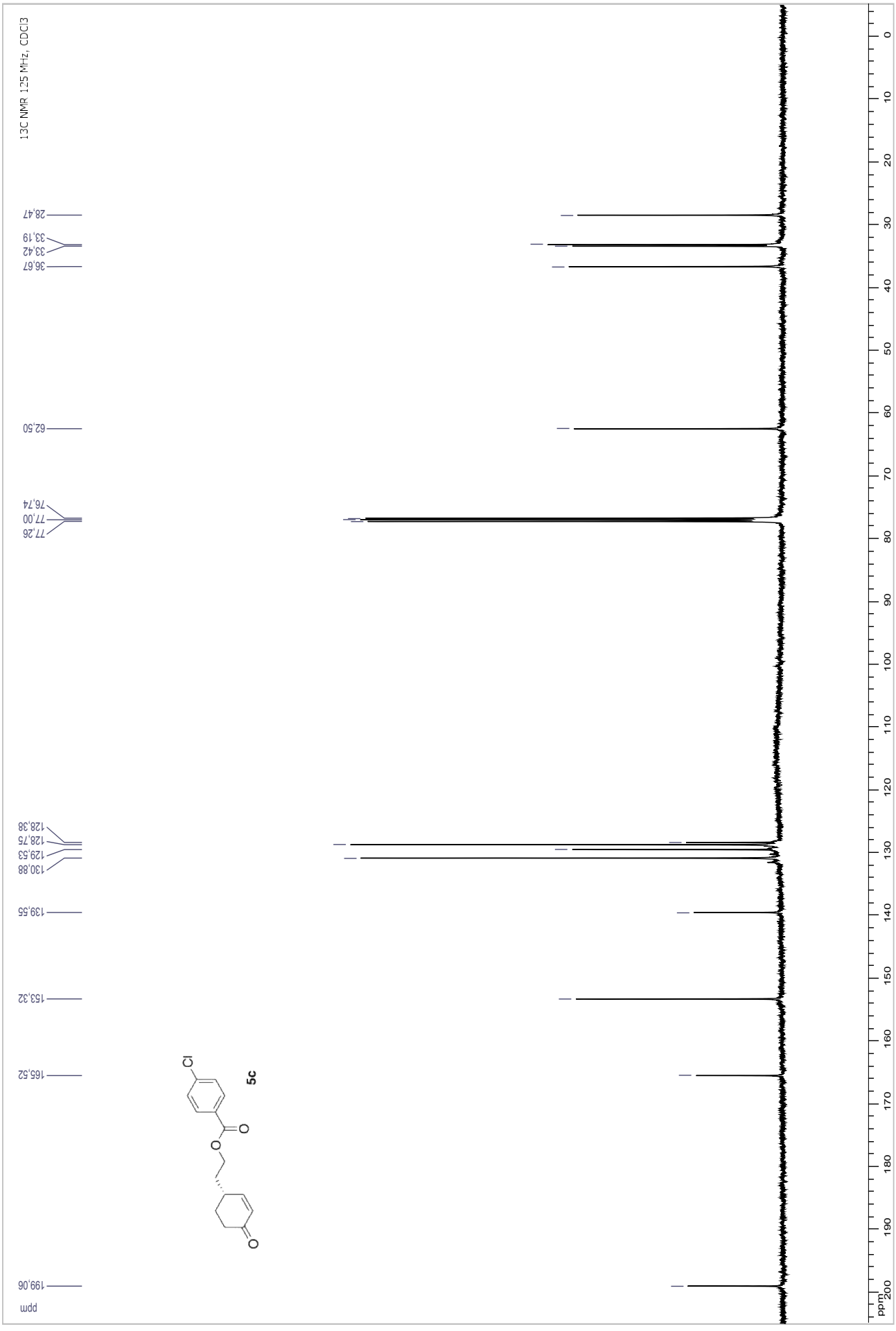
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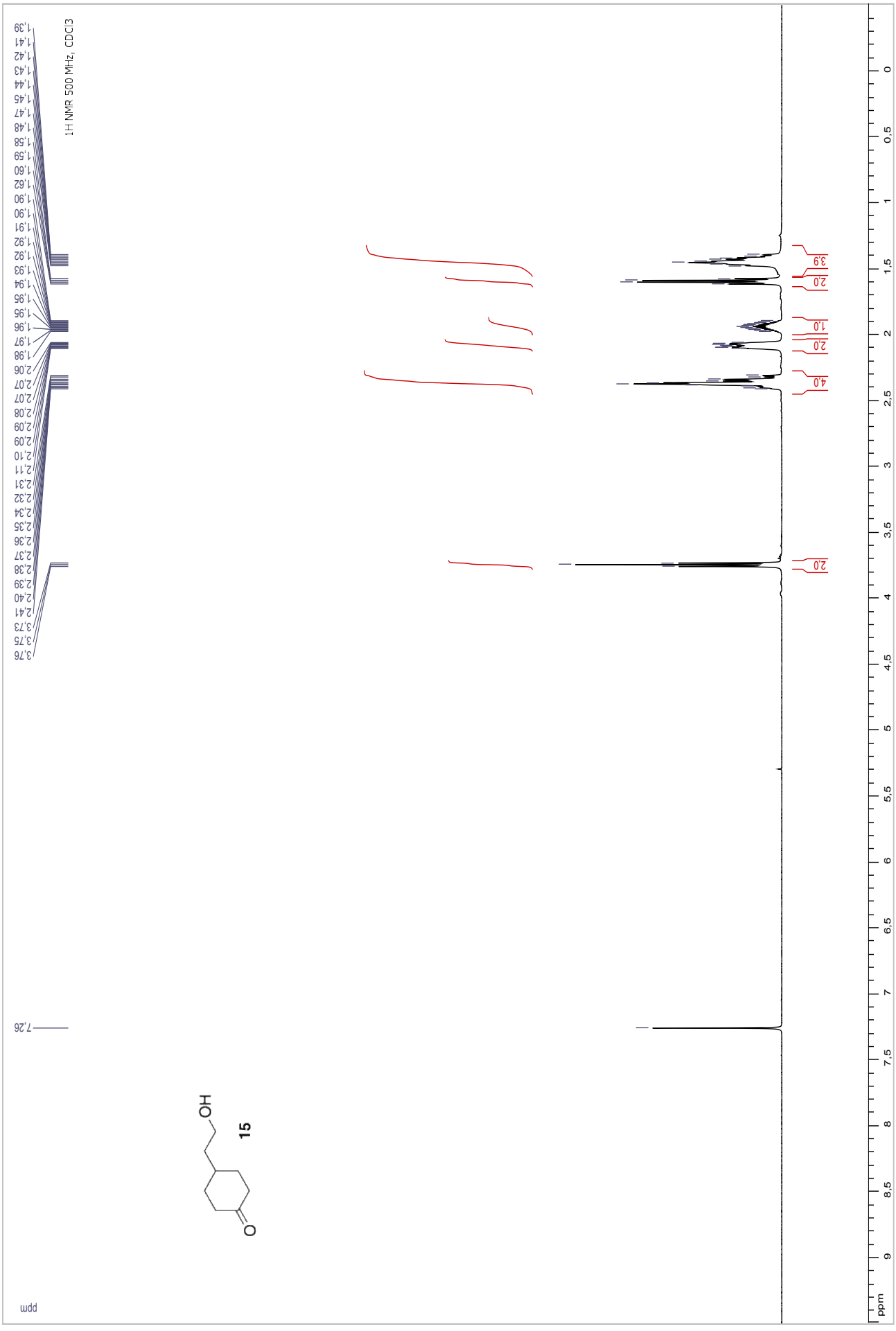
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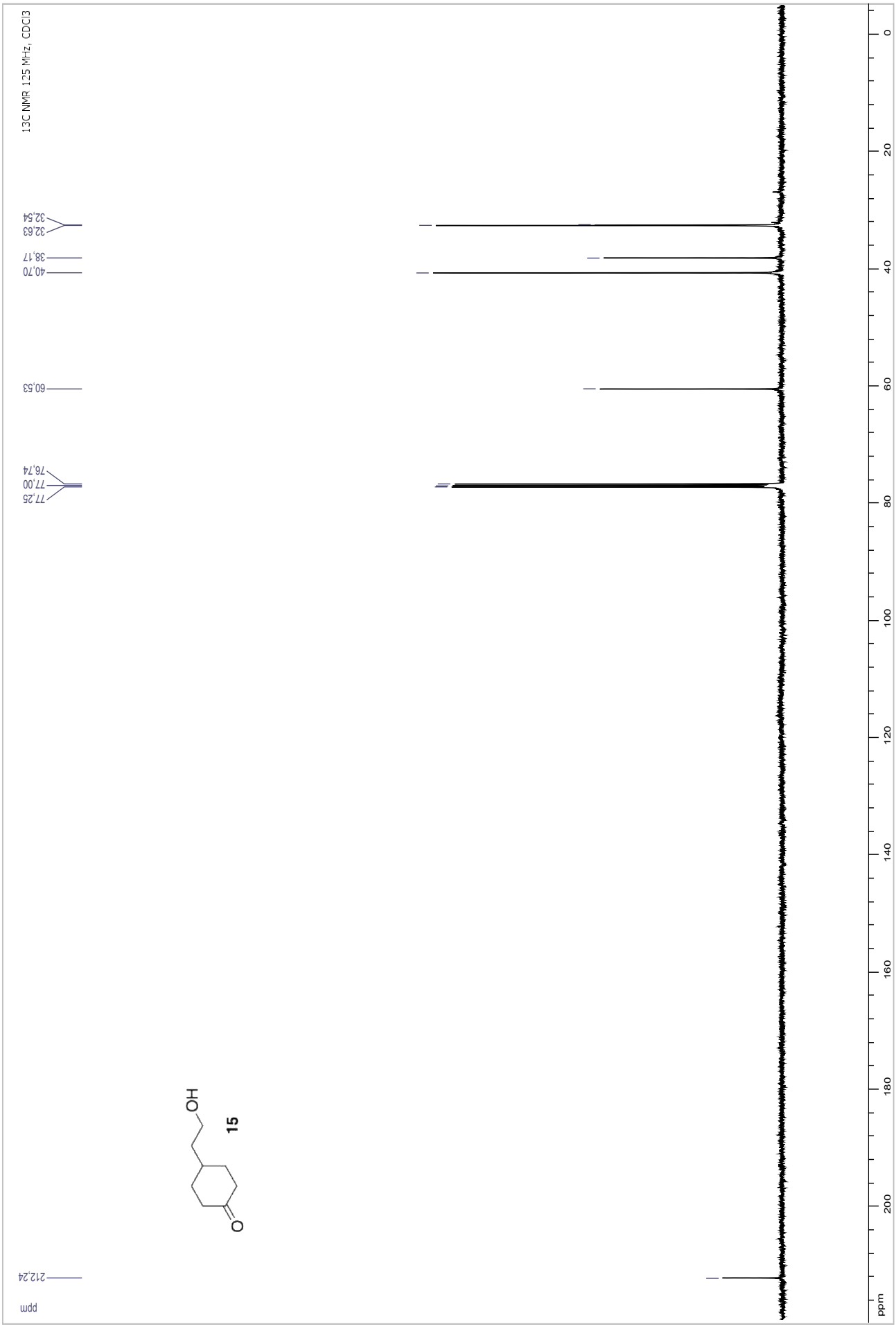
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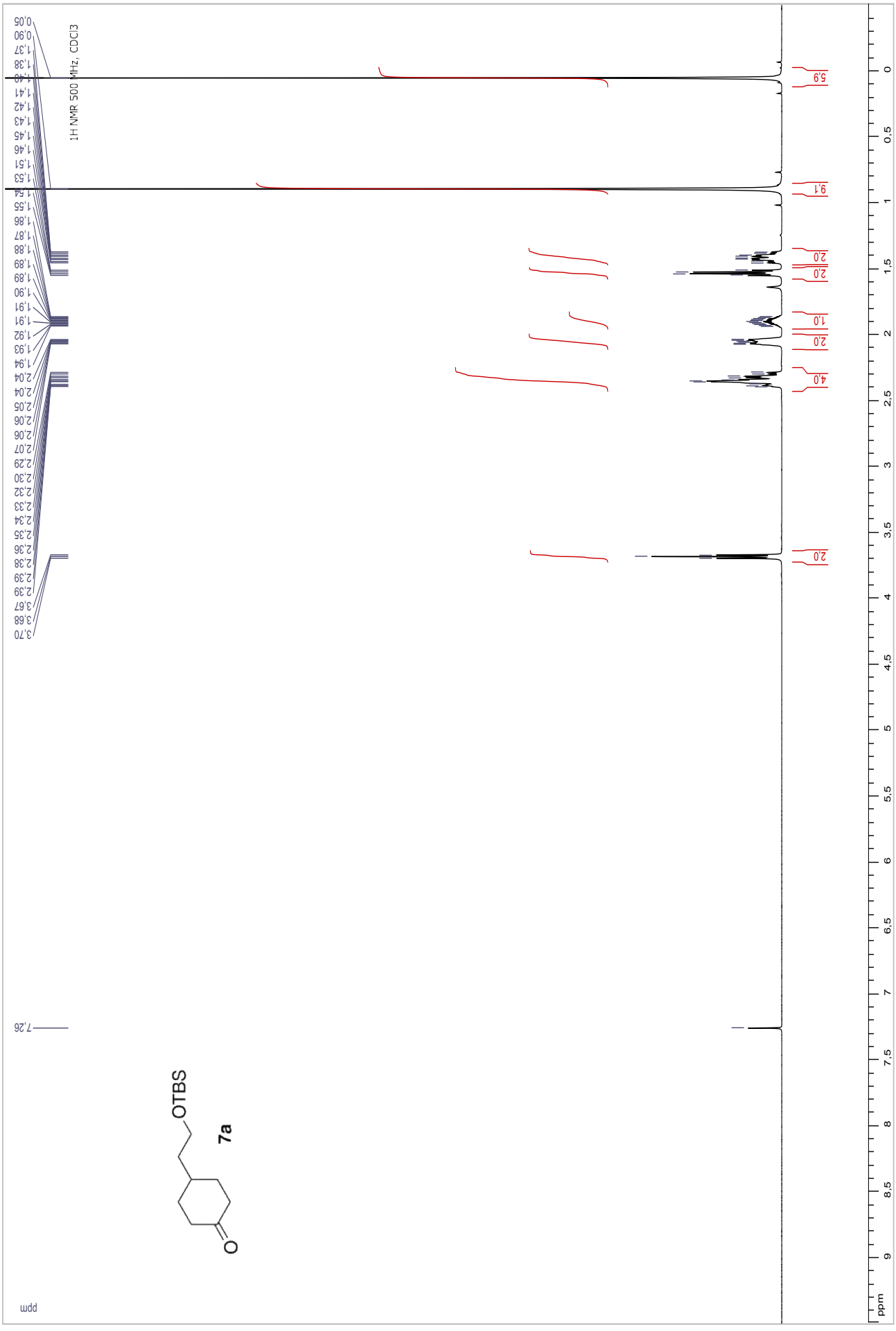
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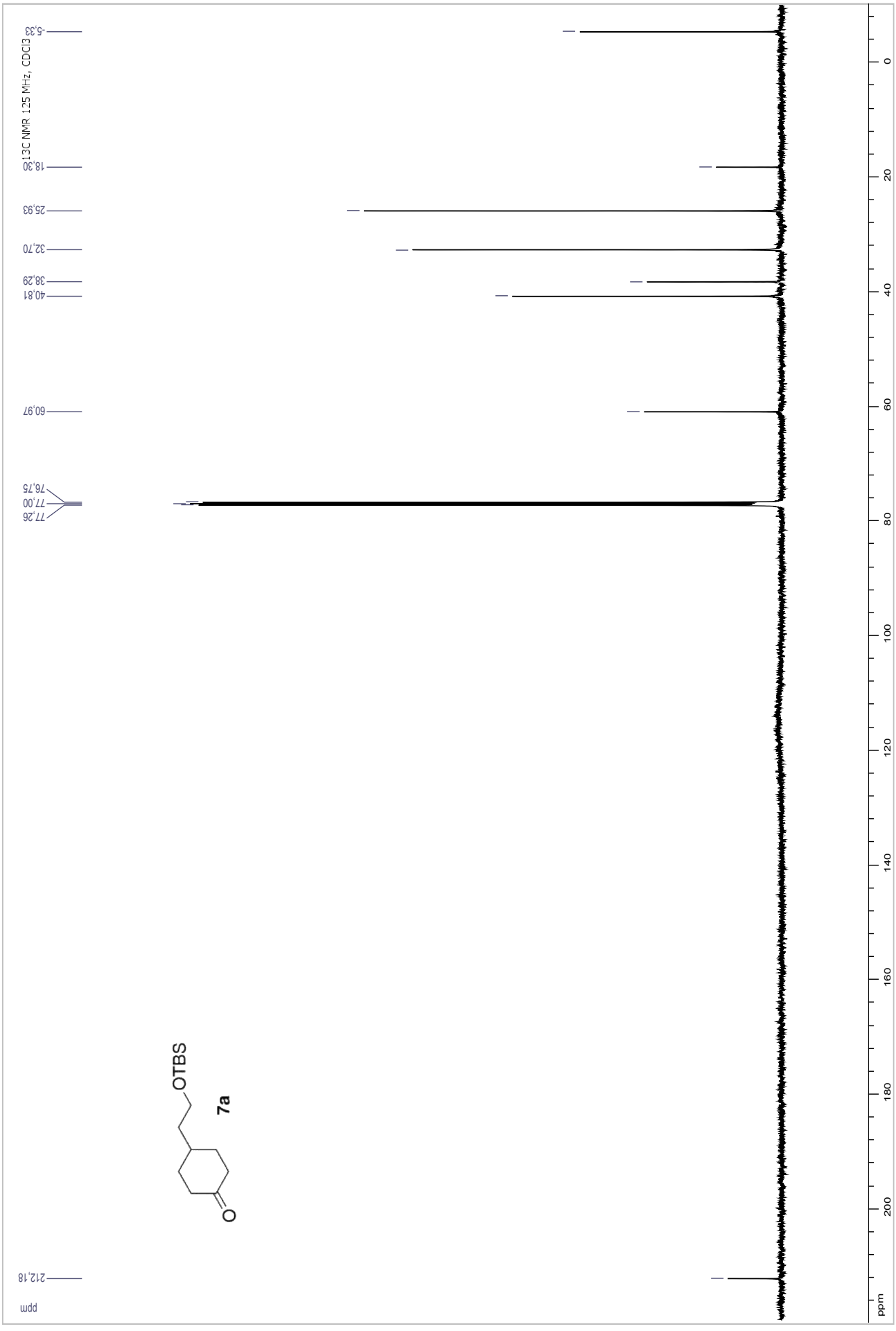
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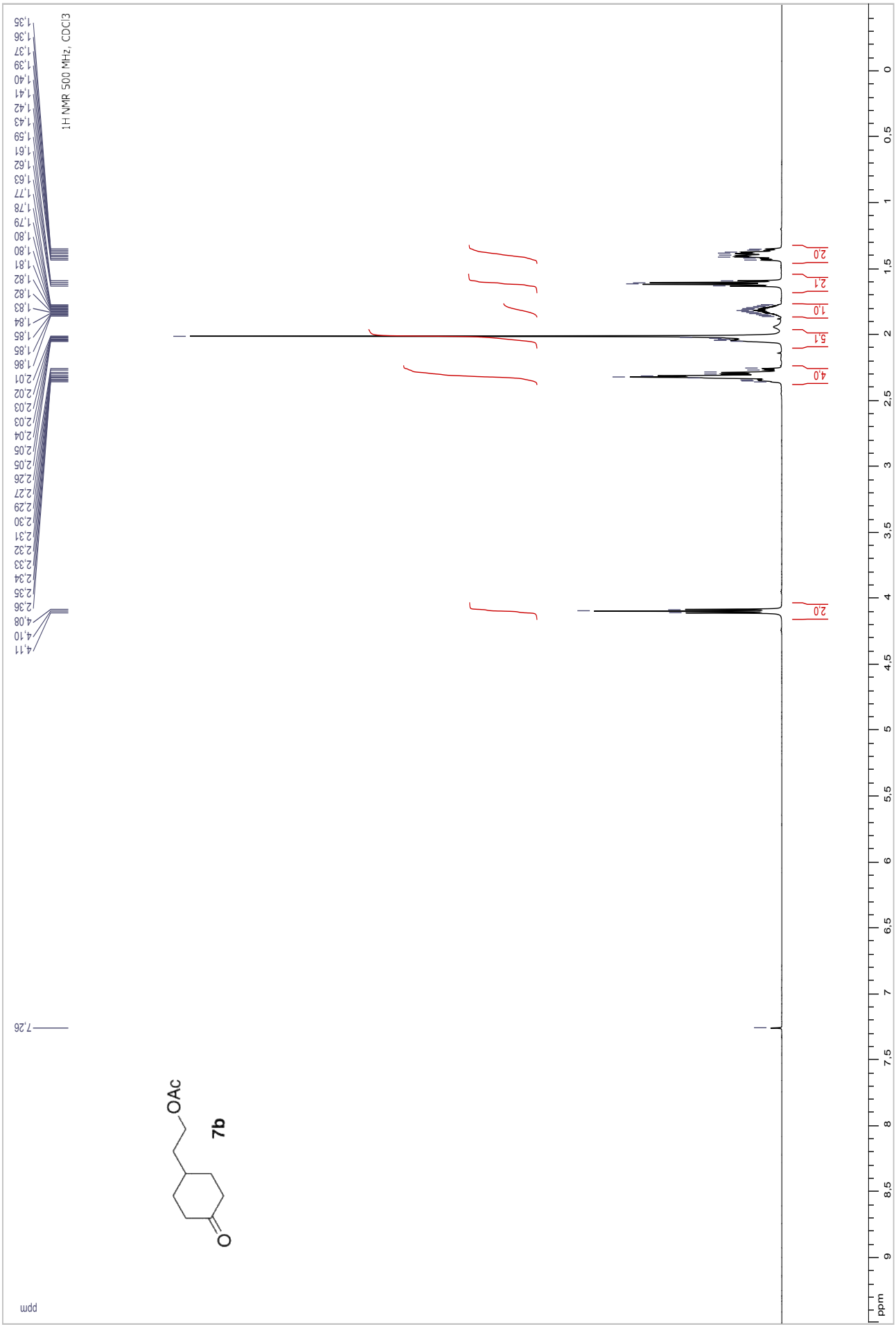
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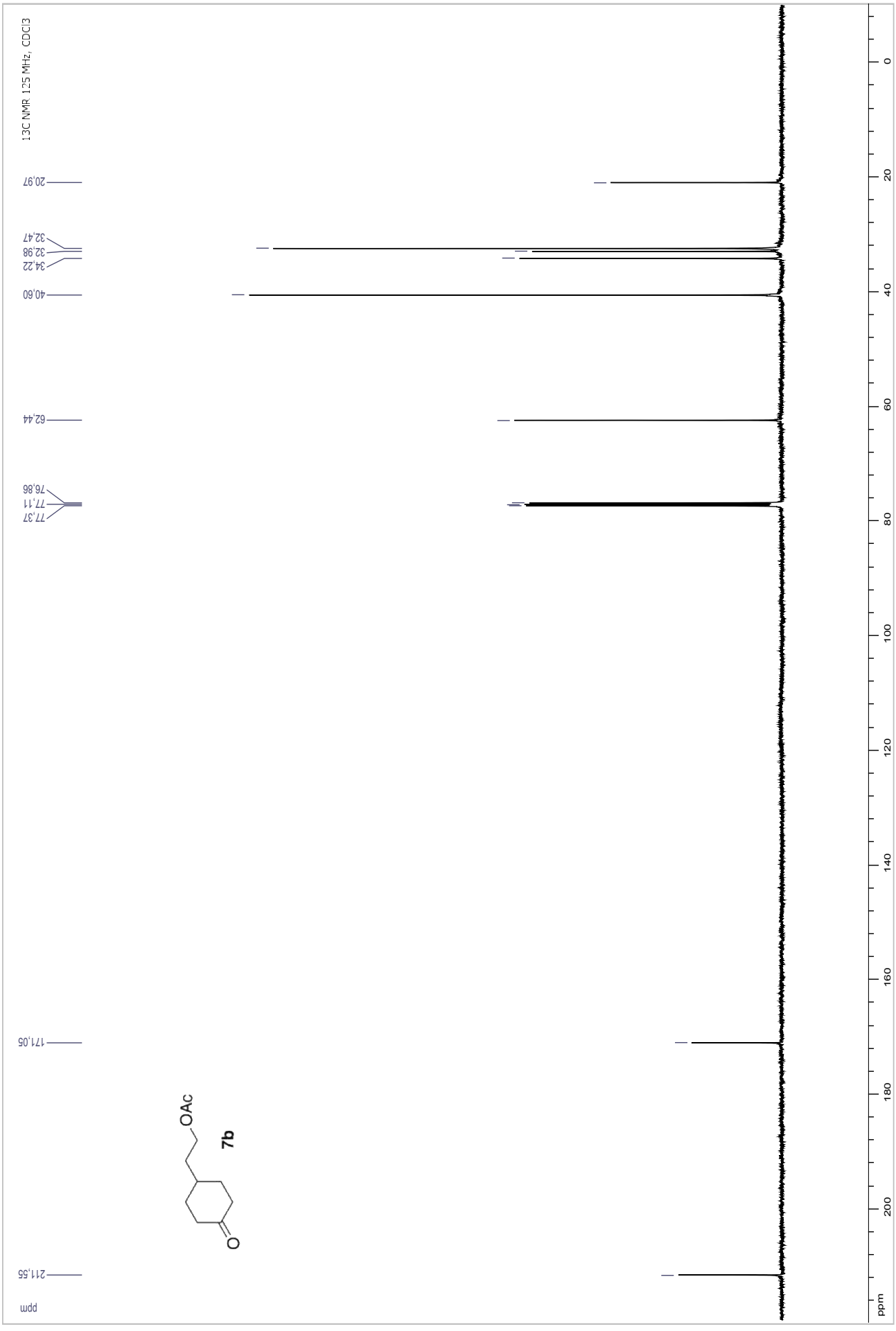
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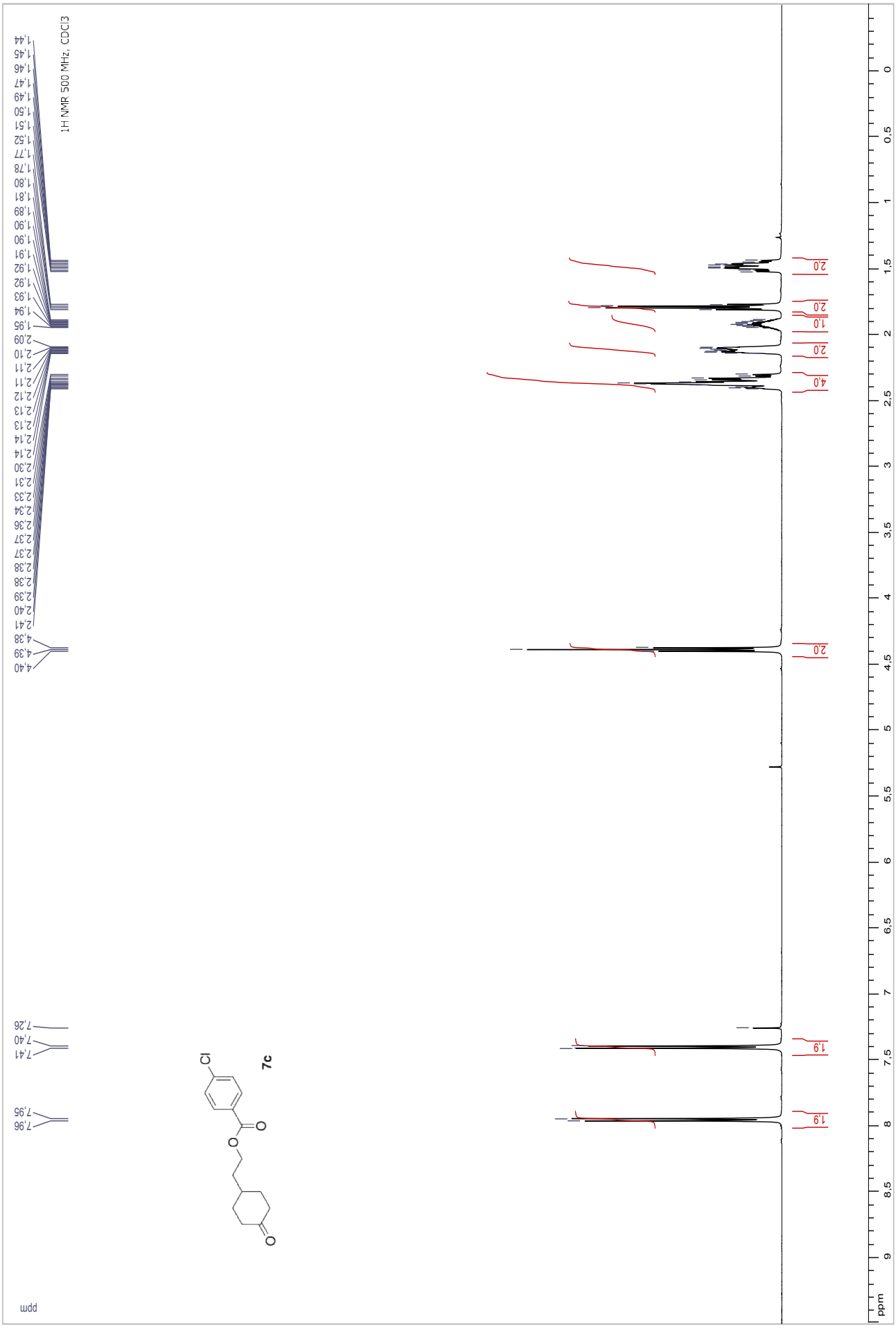
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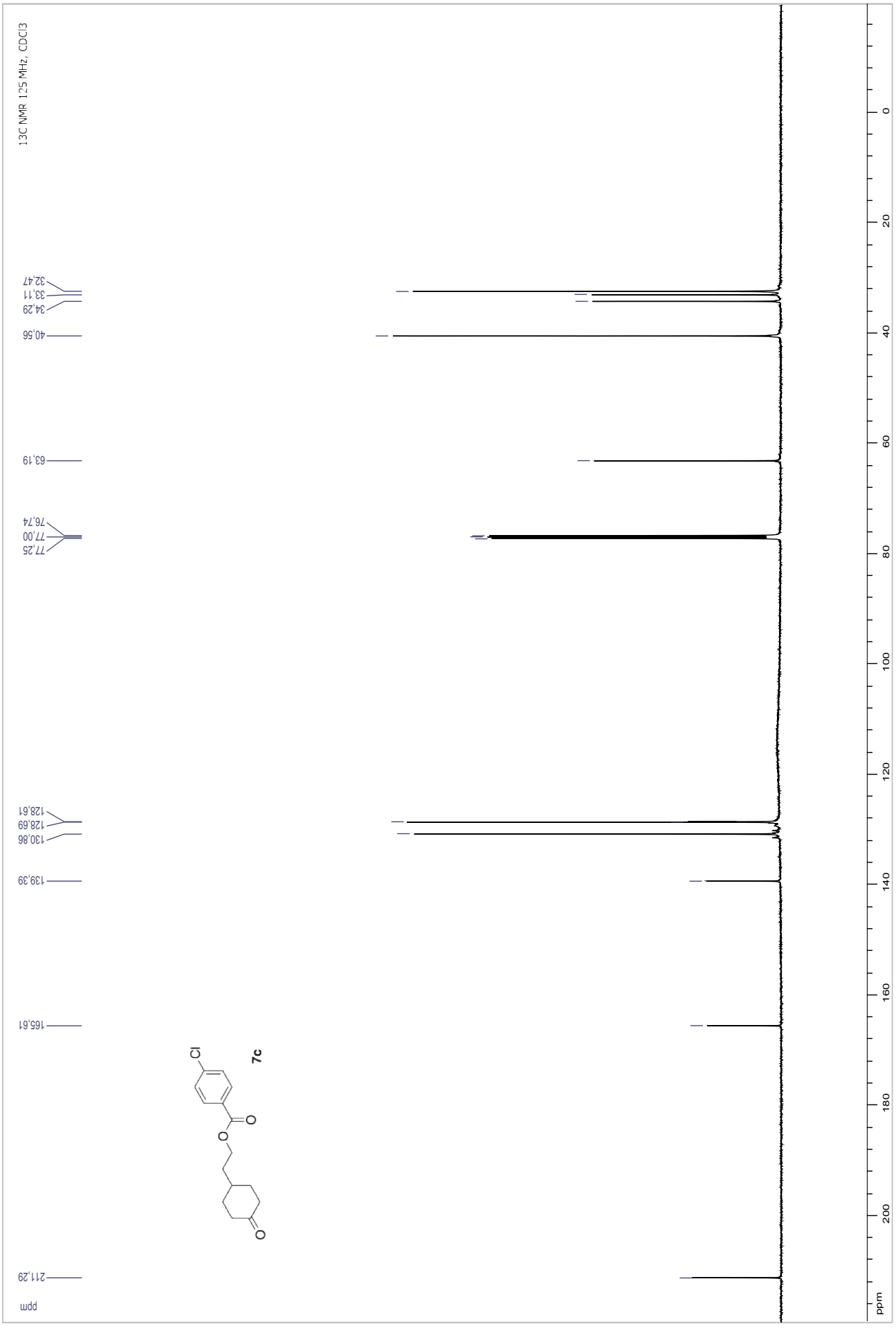
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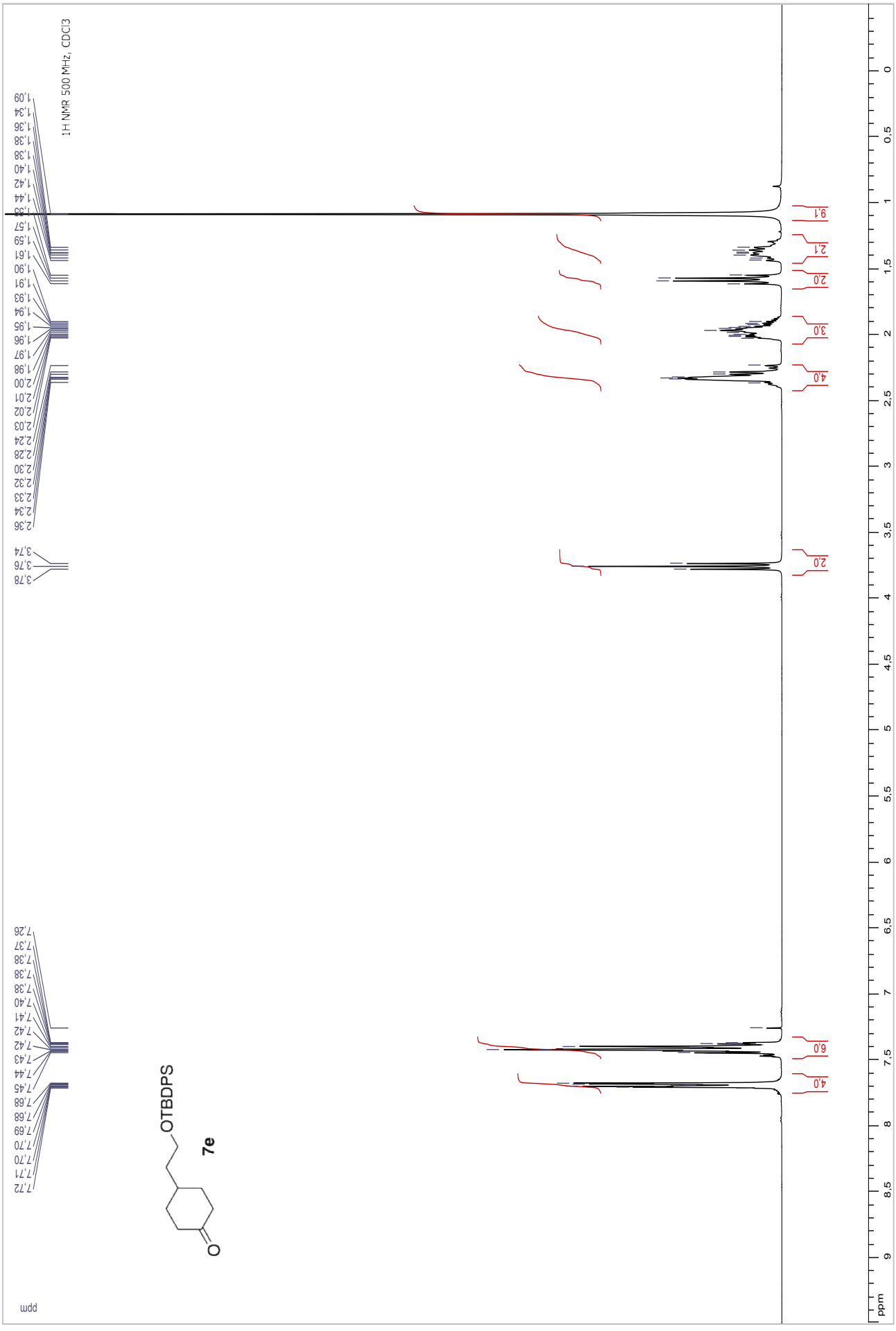
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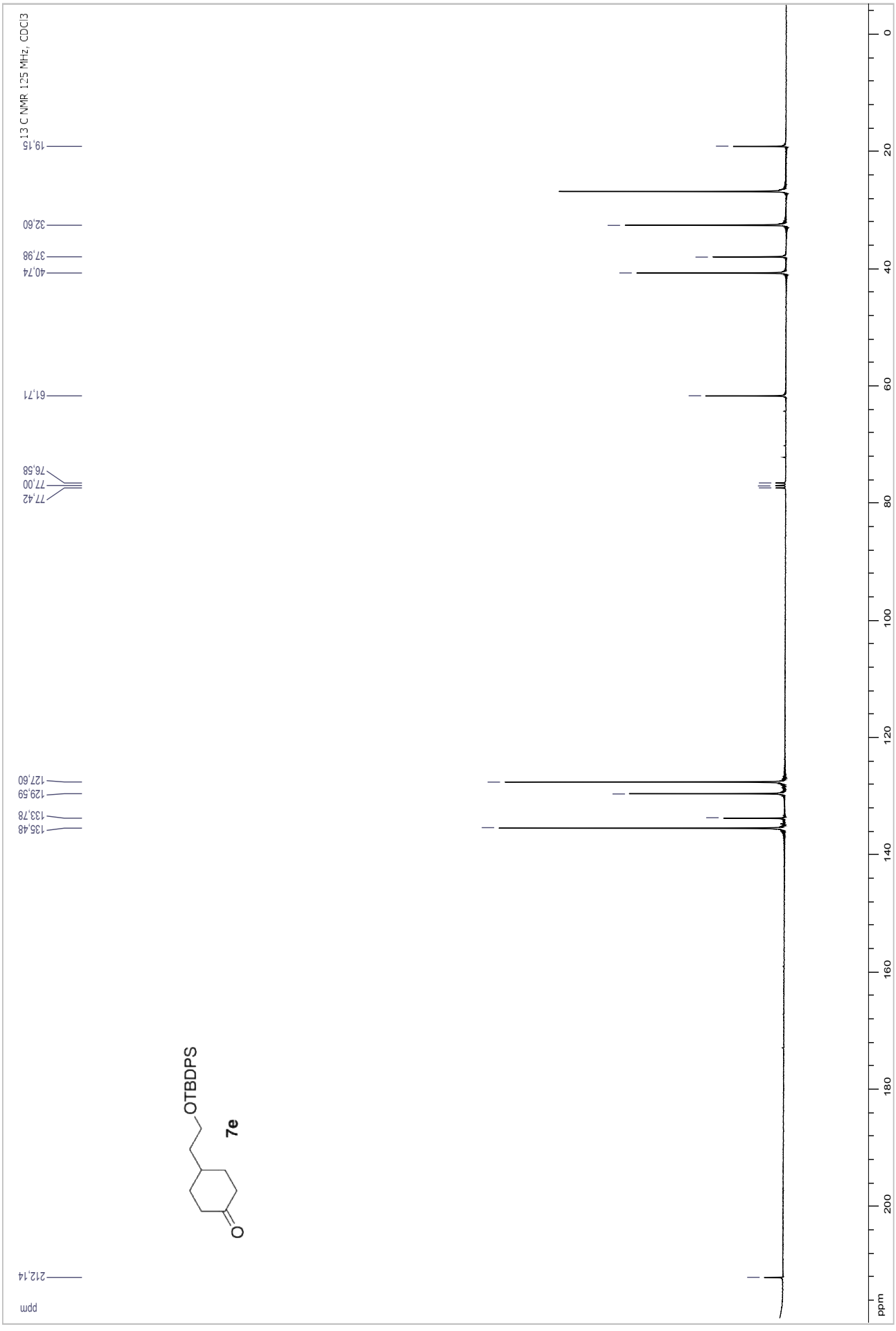
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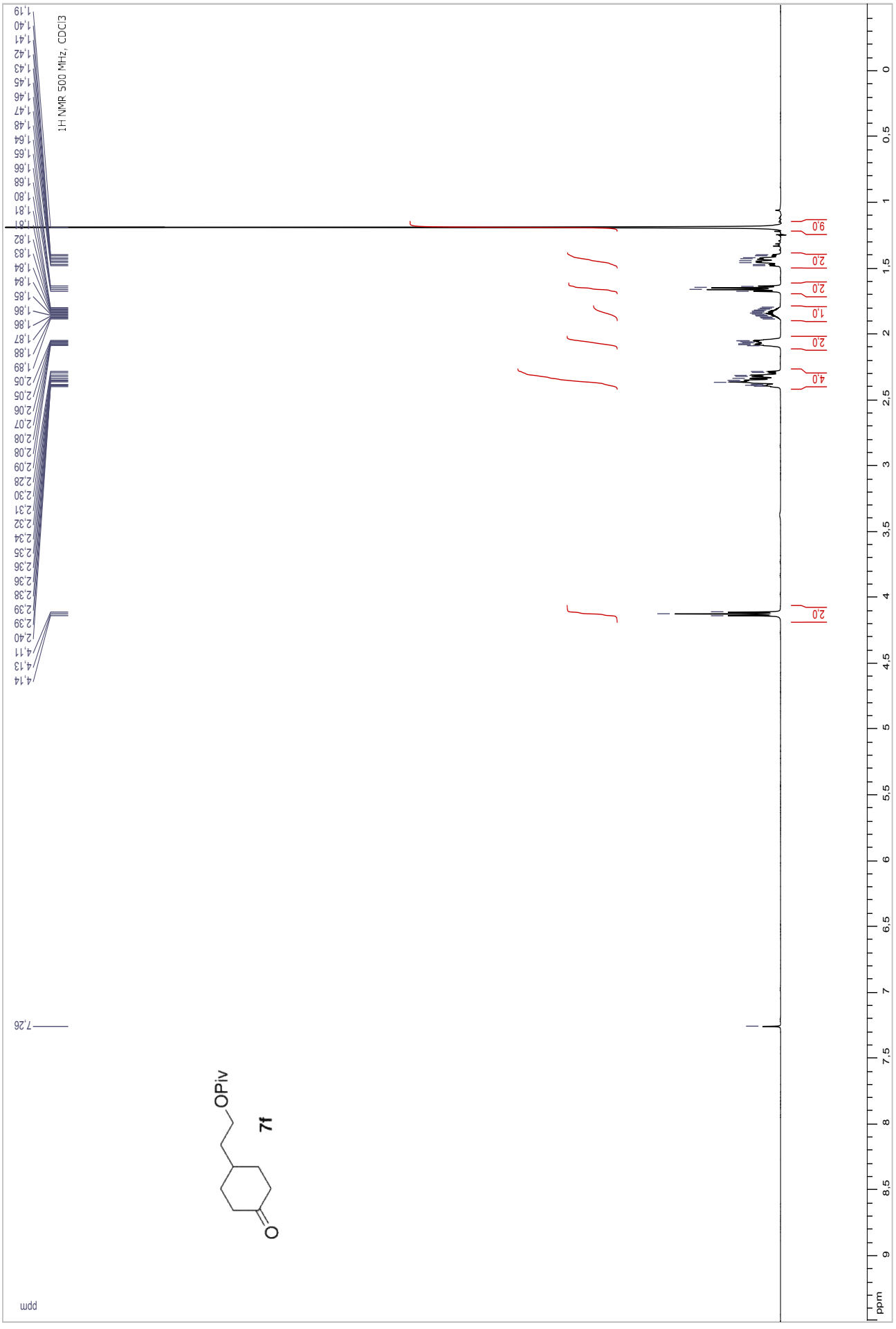
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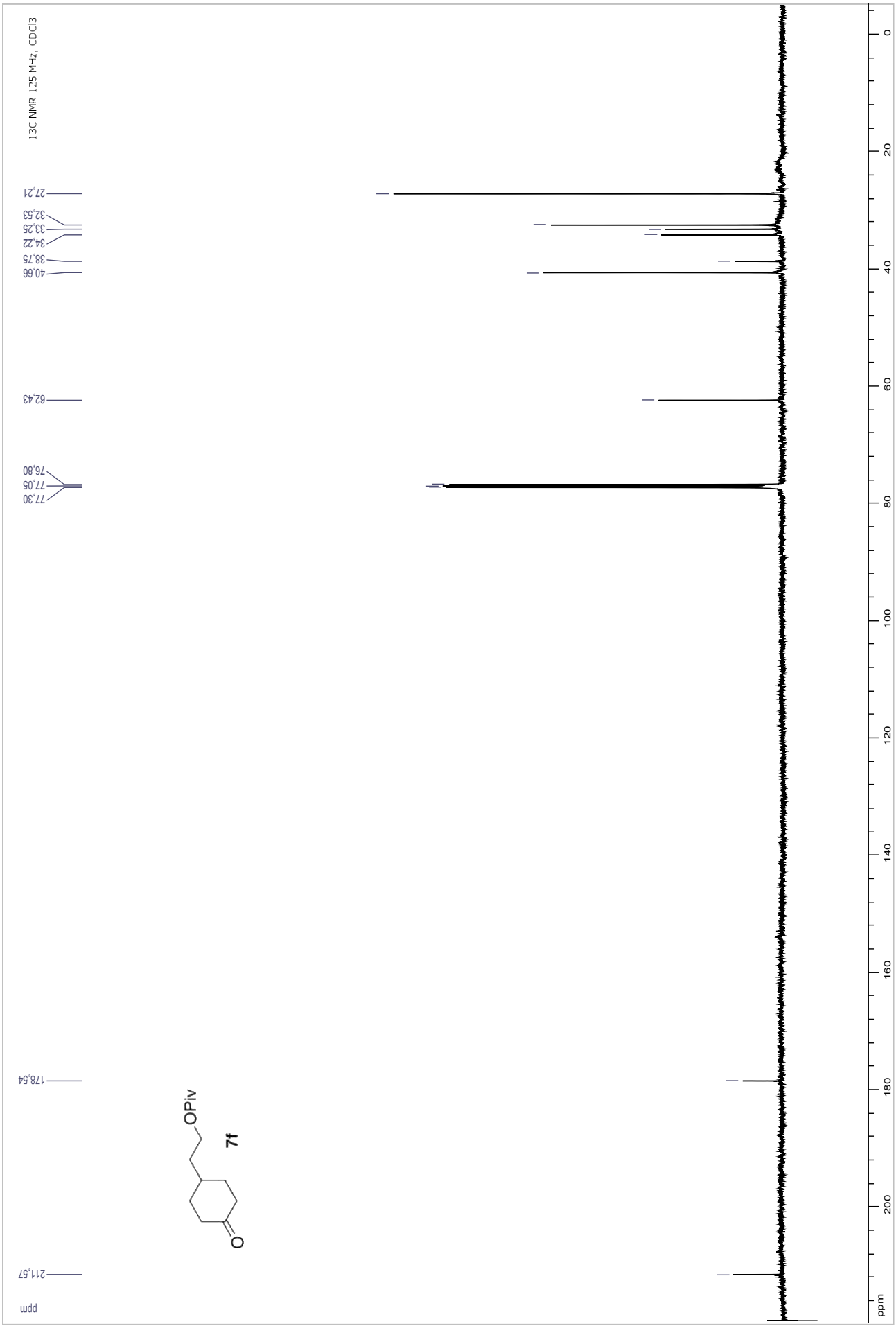
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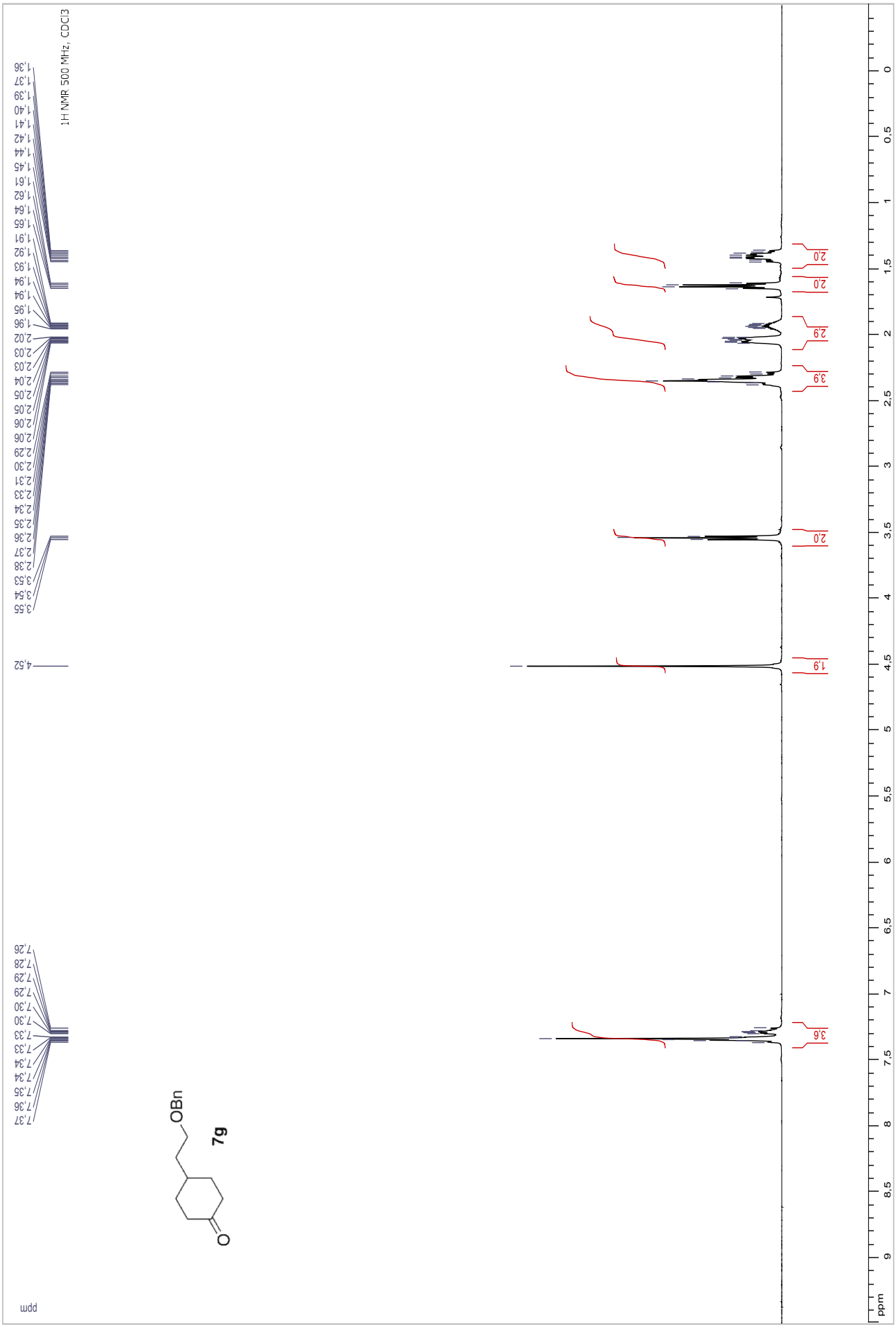
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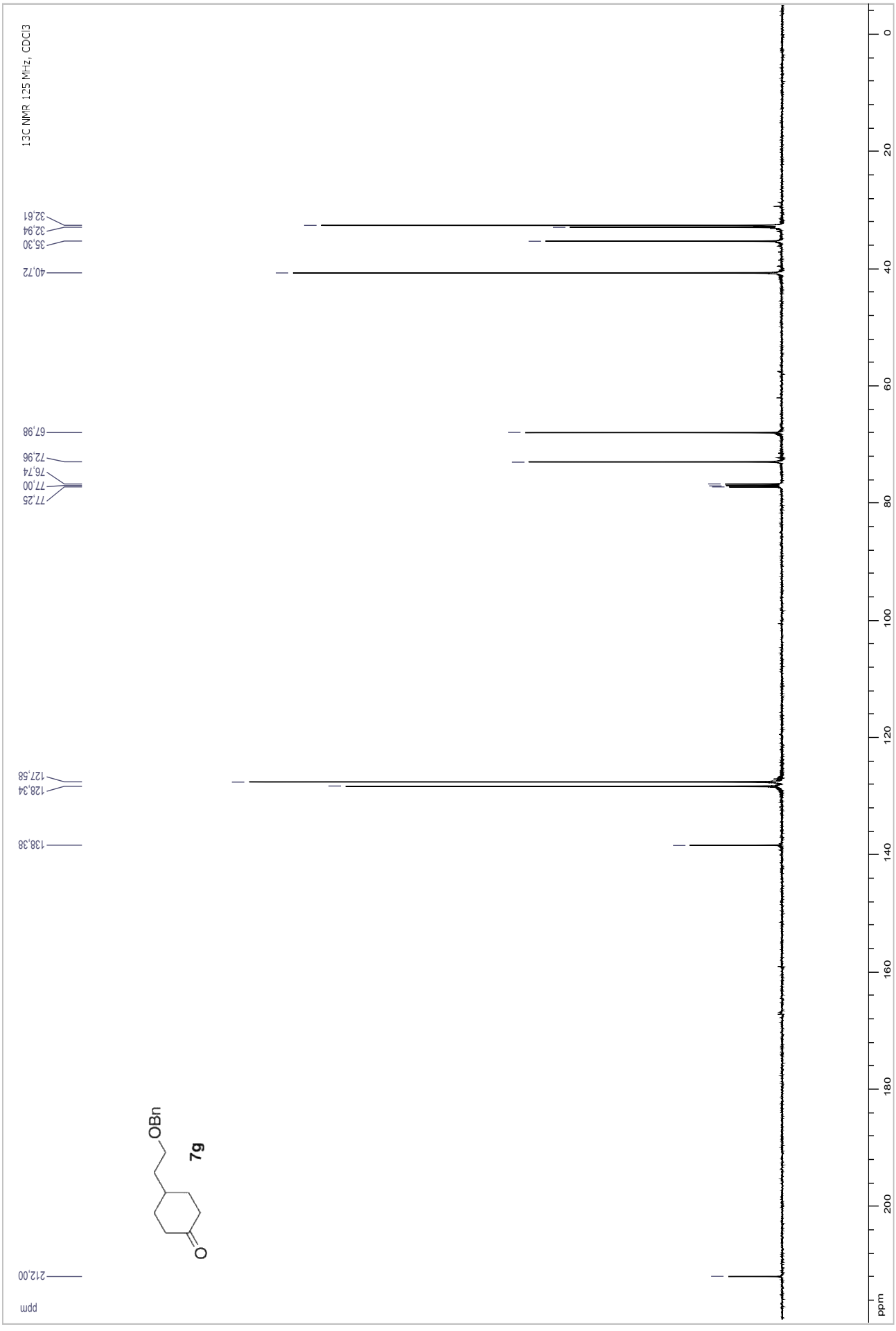
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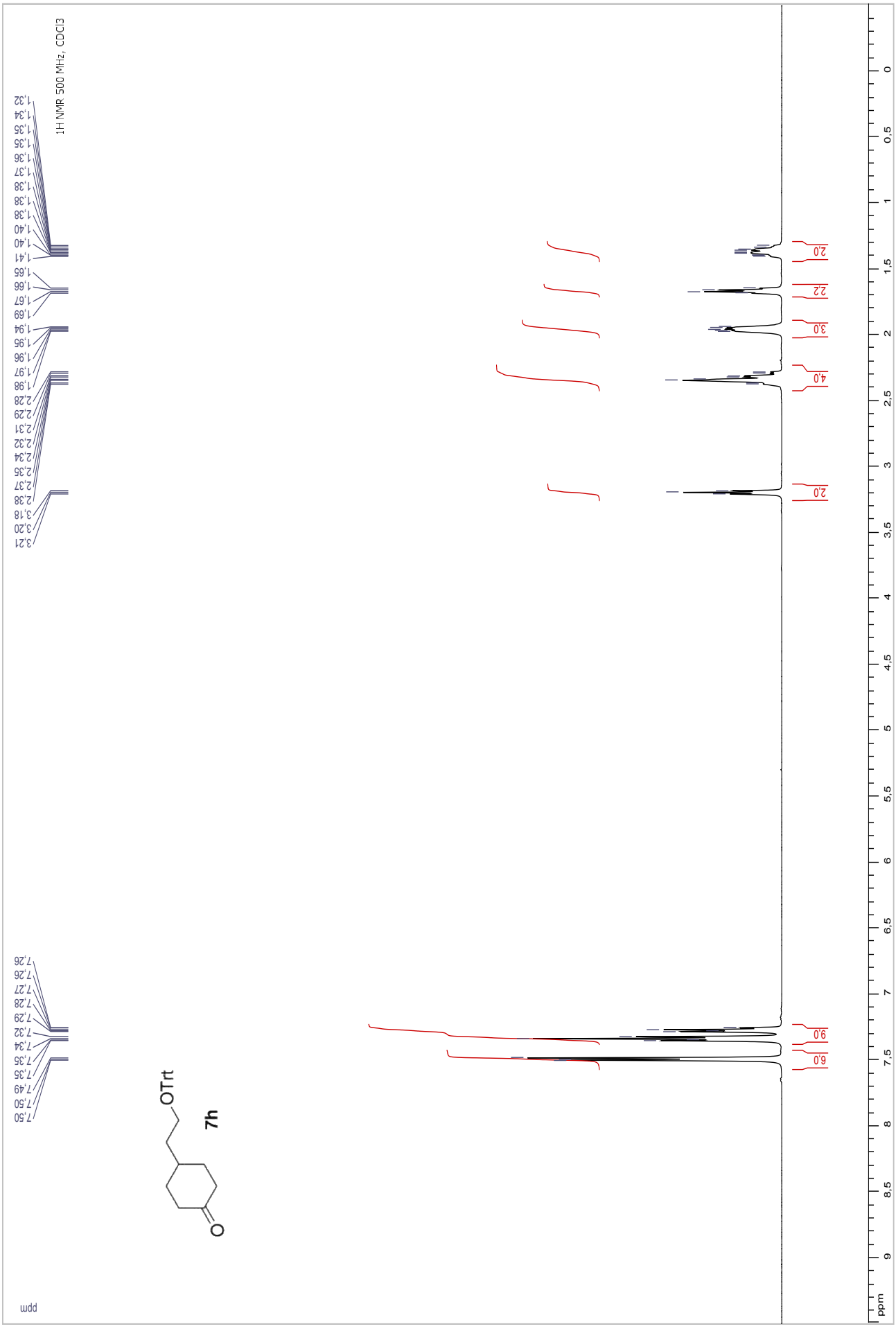
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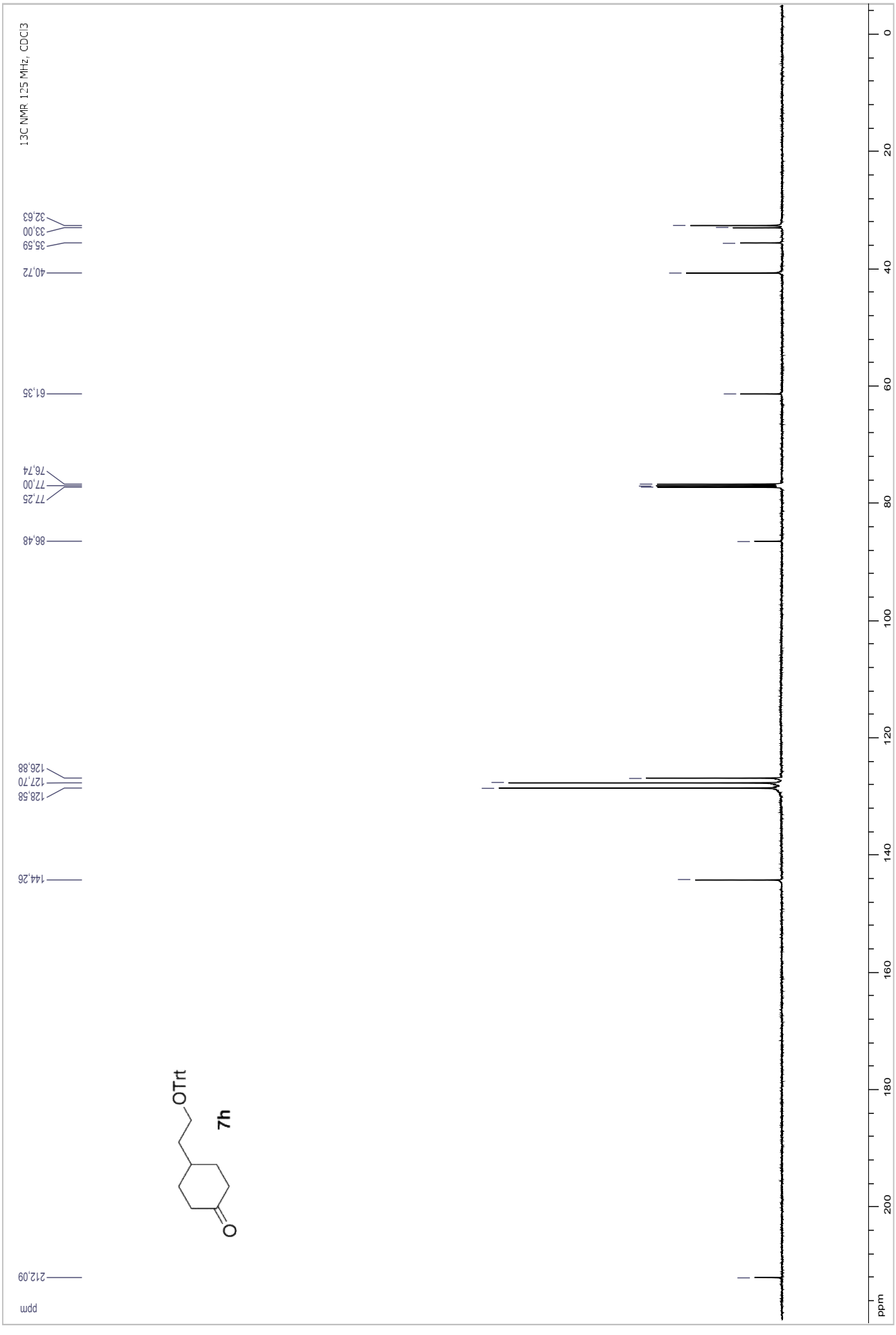
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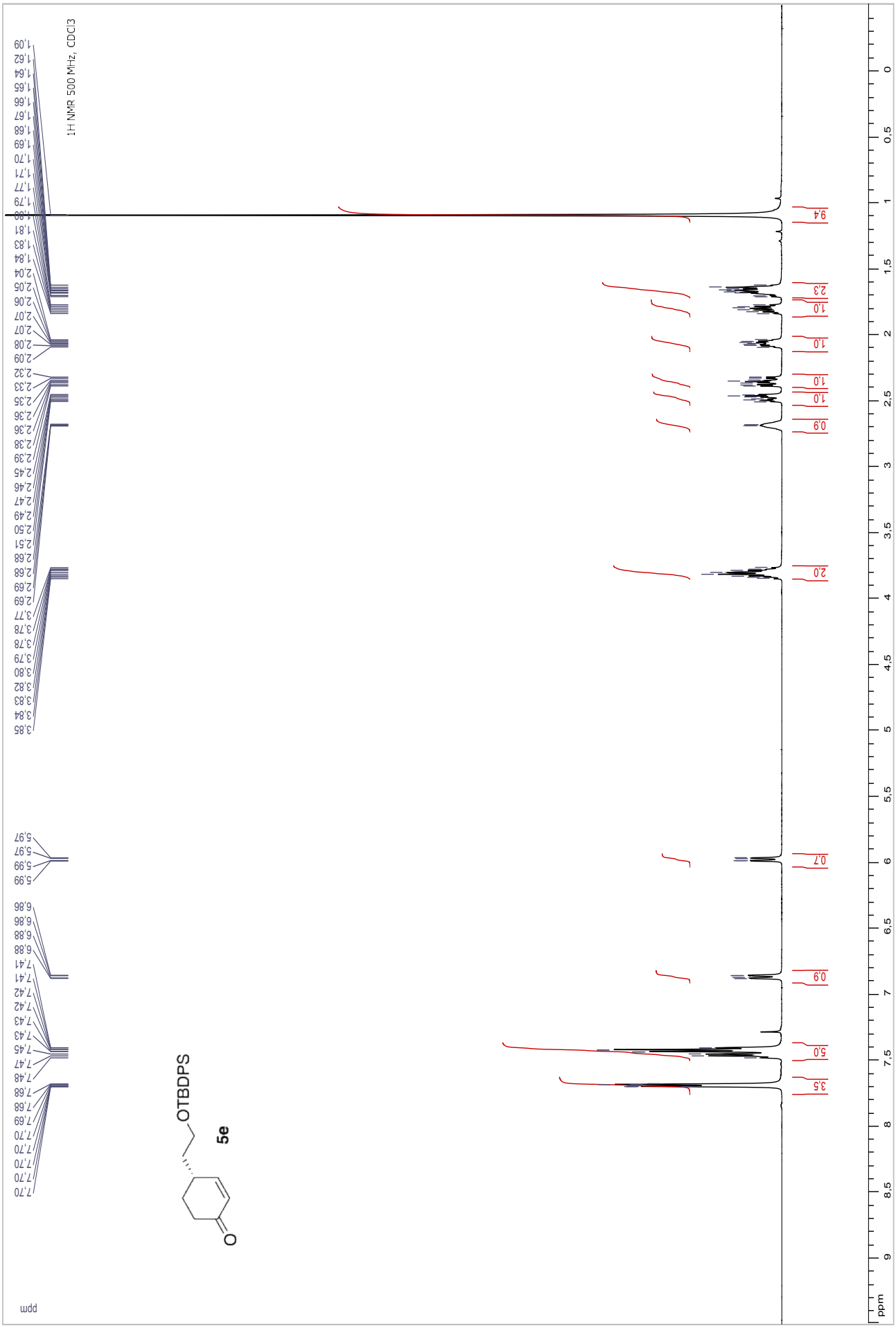
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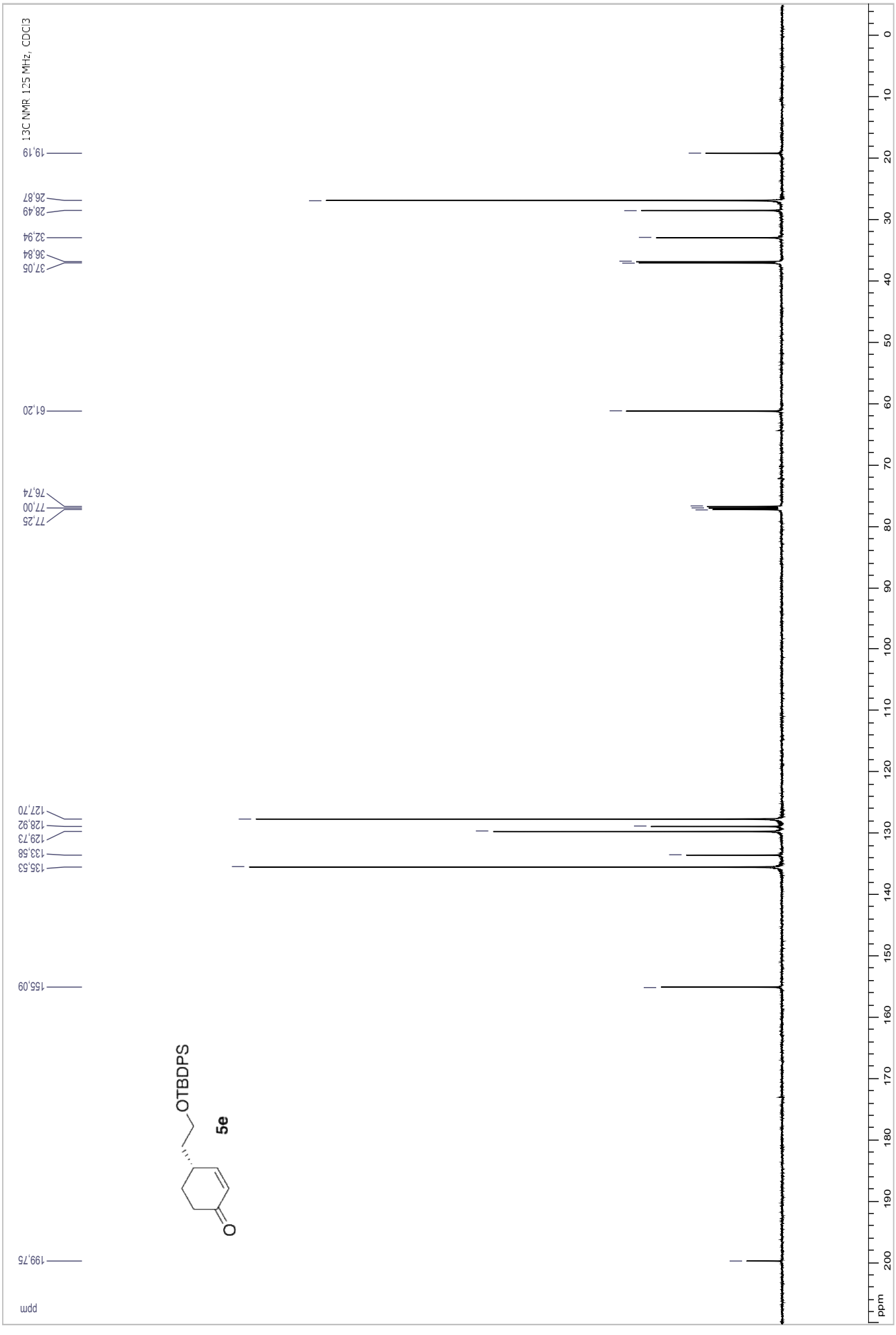
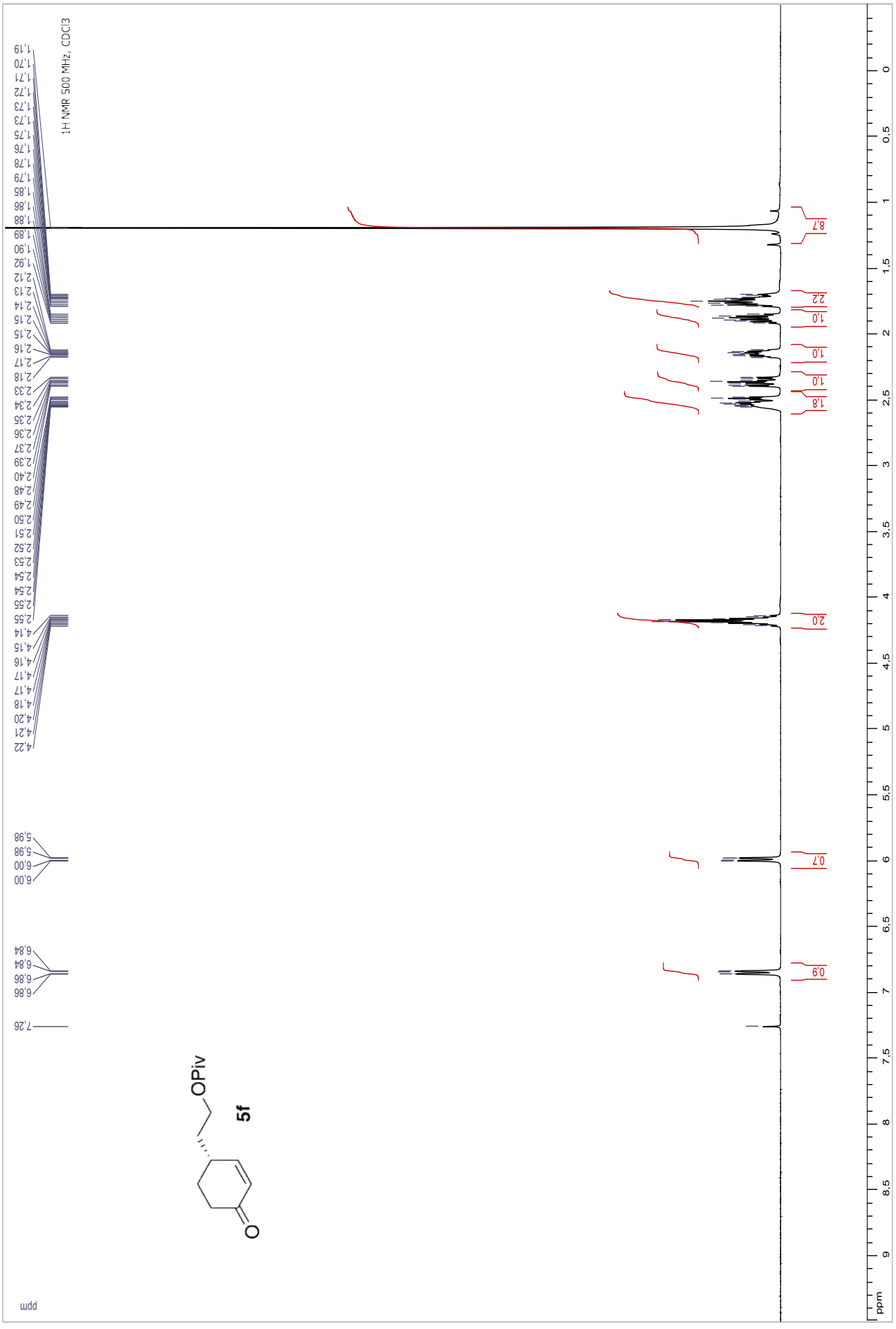
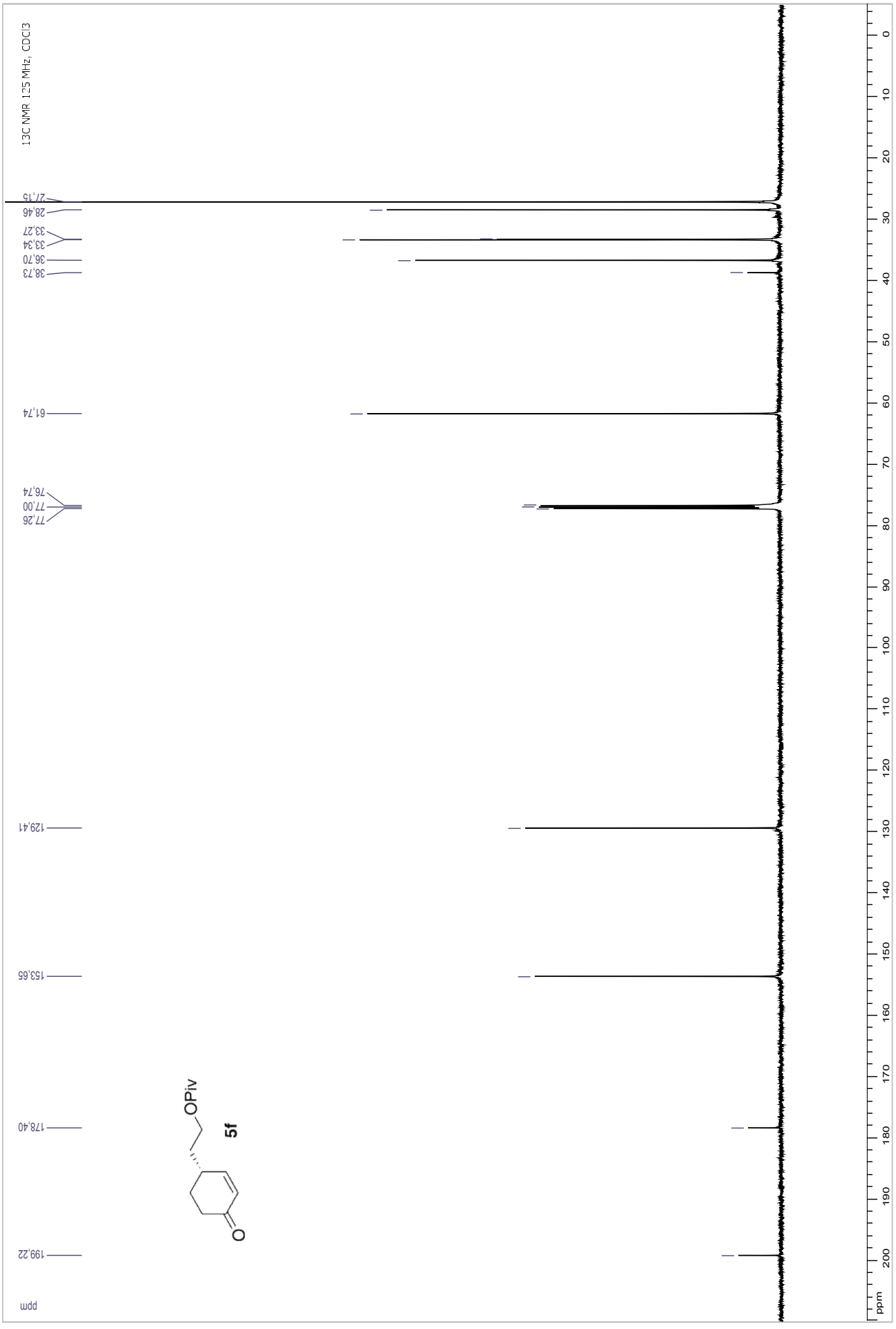
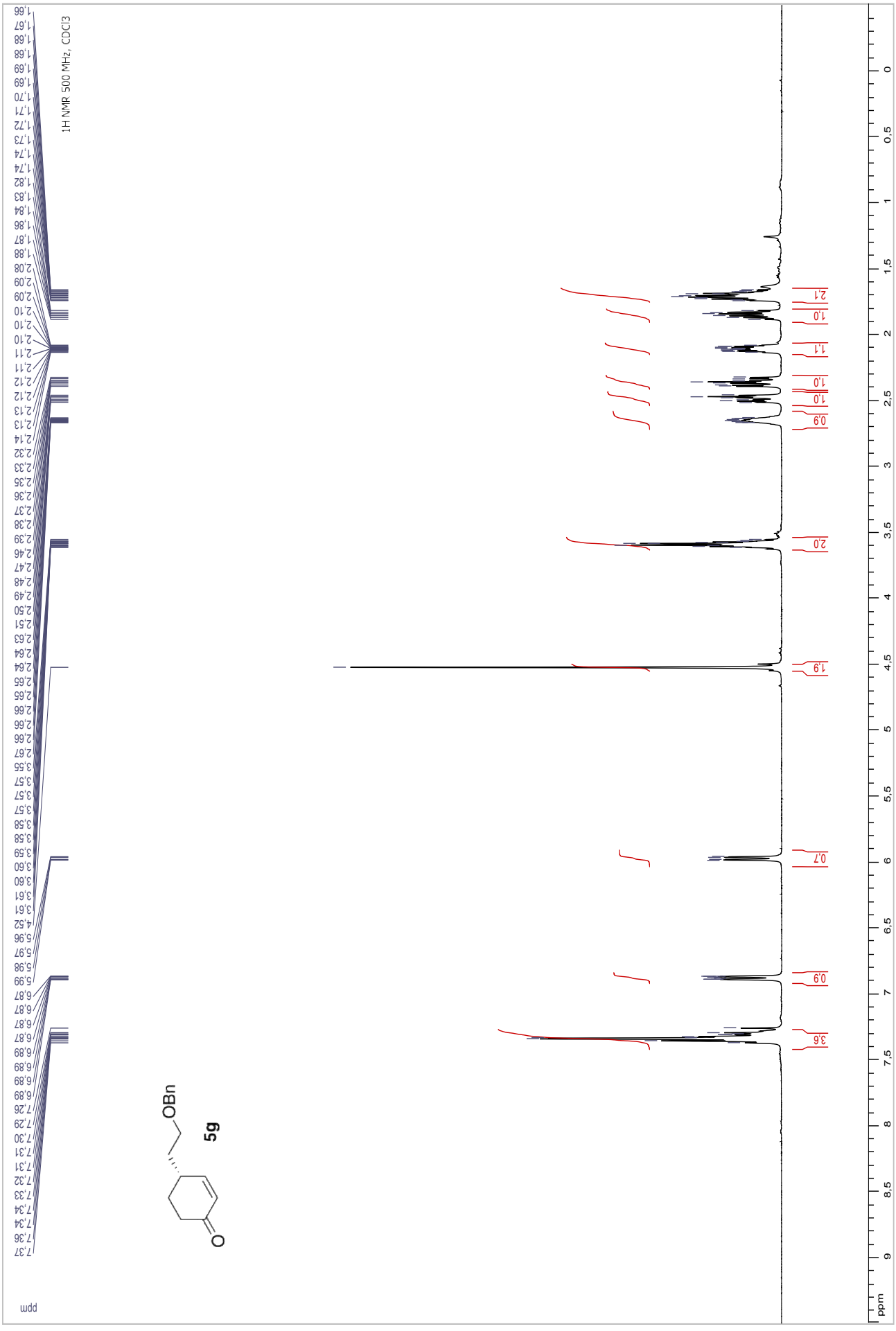
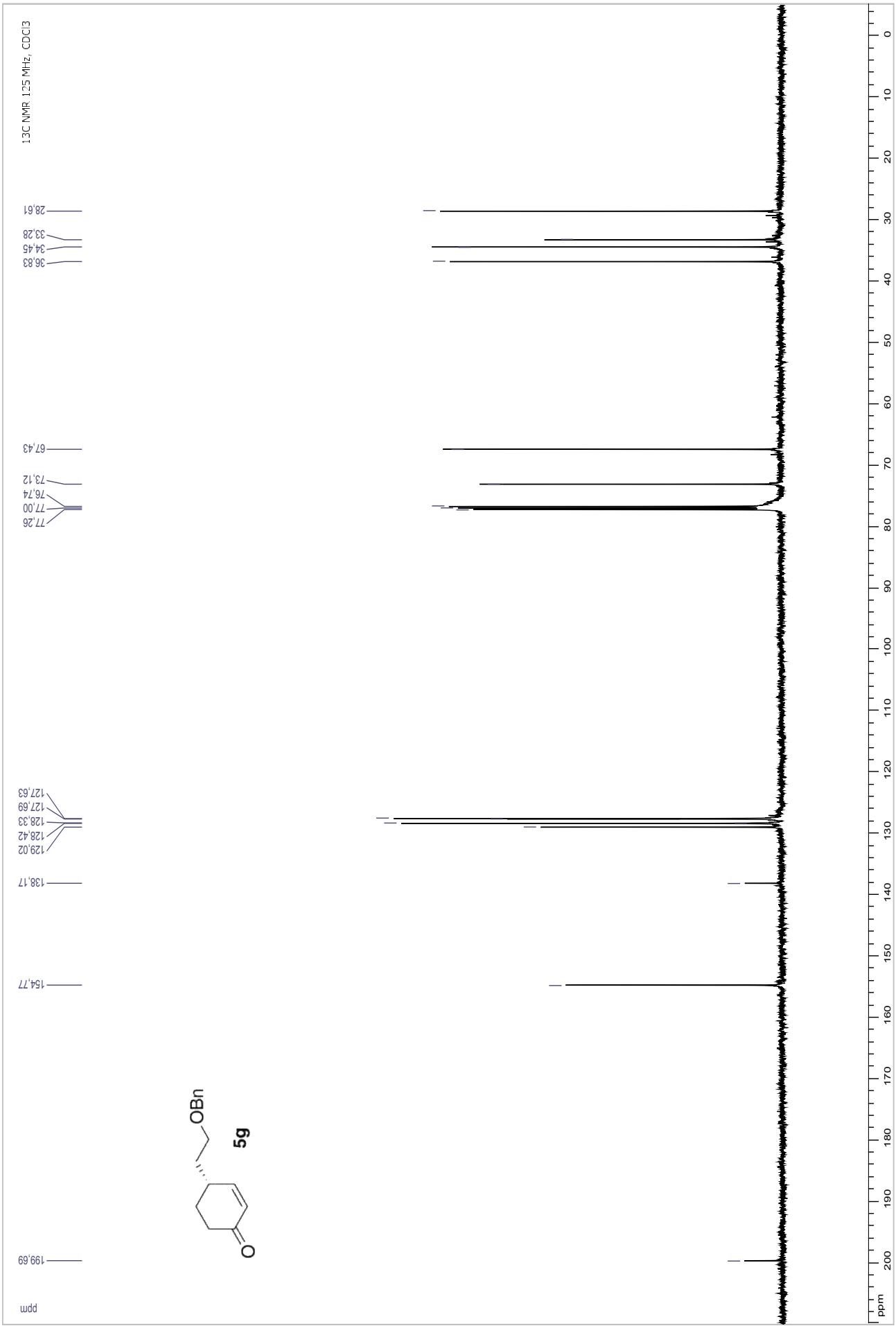
O
ppm

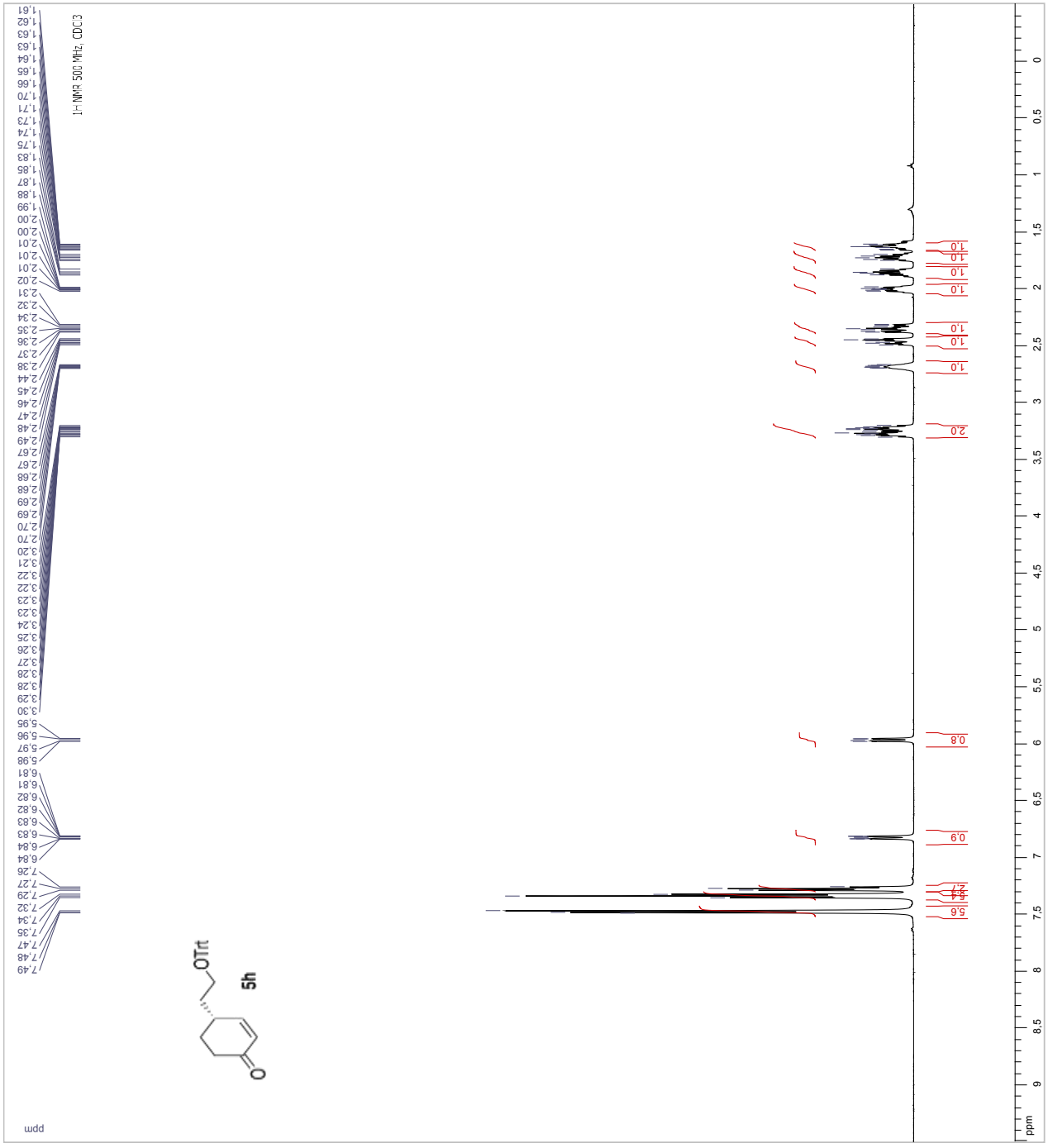

13C NMR 125 MHz, CDCl3
19.19
26.87
28.49
32.94
36.84
37.05
61.20
76.74
77.00
77.25
127.70
128.92
129.73
133.58
135.53
155.09
199.75
ppm
OTBDPS
5e
O

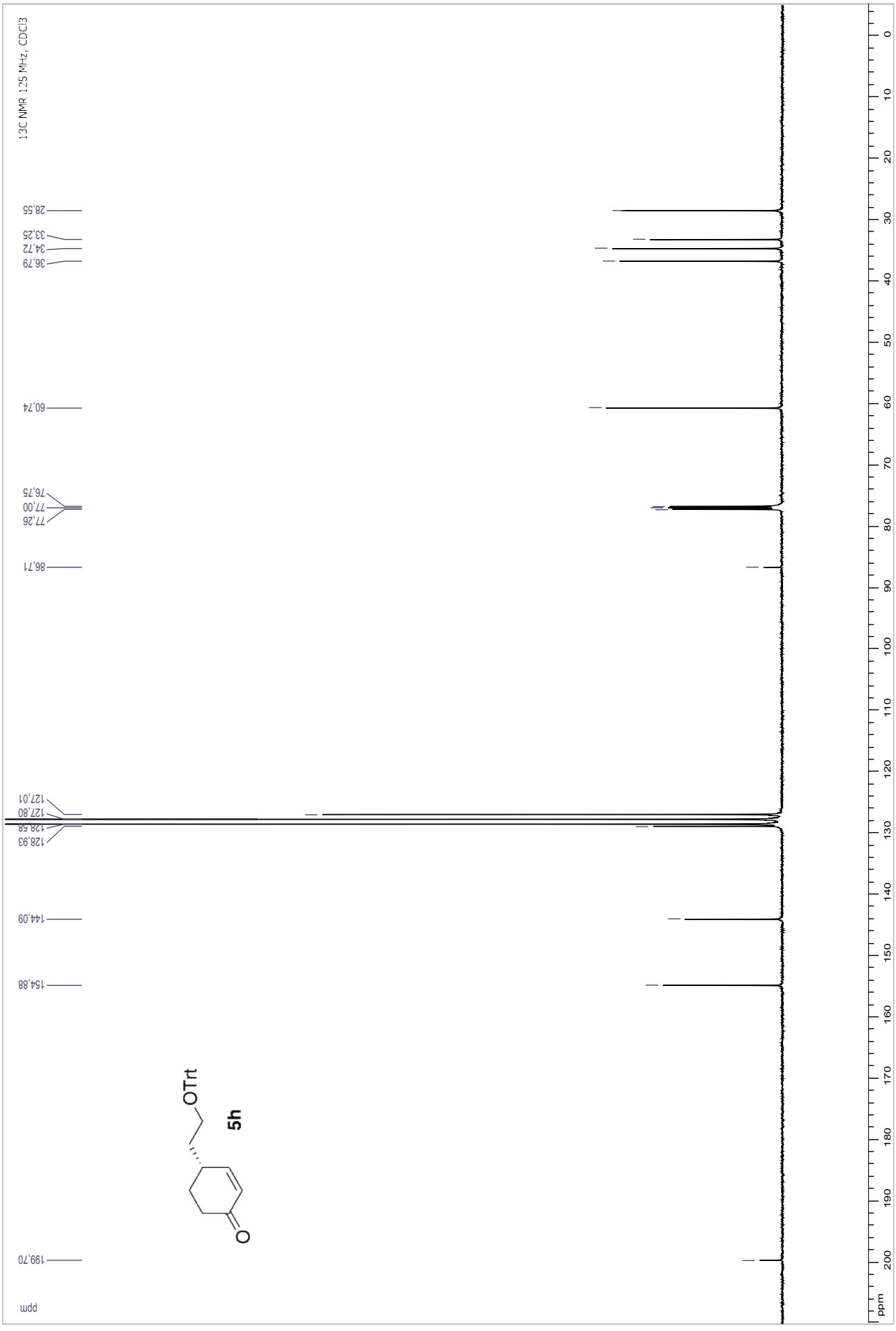

1H NMR 500 MHz, CDCl3
OPiv
5f
O
8.7
2.2
1.0
1.0
1.0
1.8
2.0
0.7
0.9
ppm

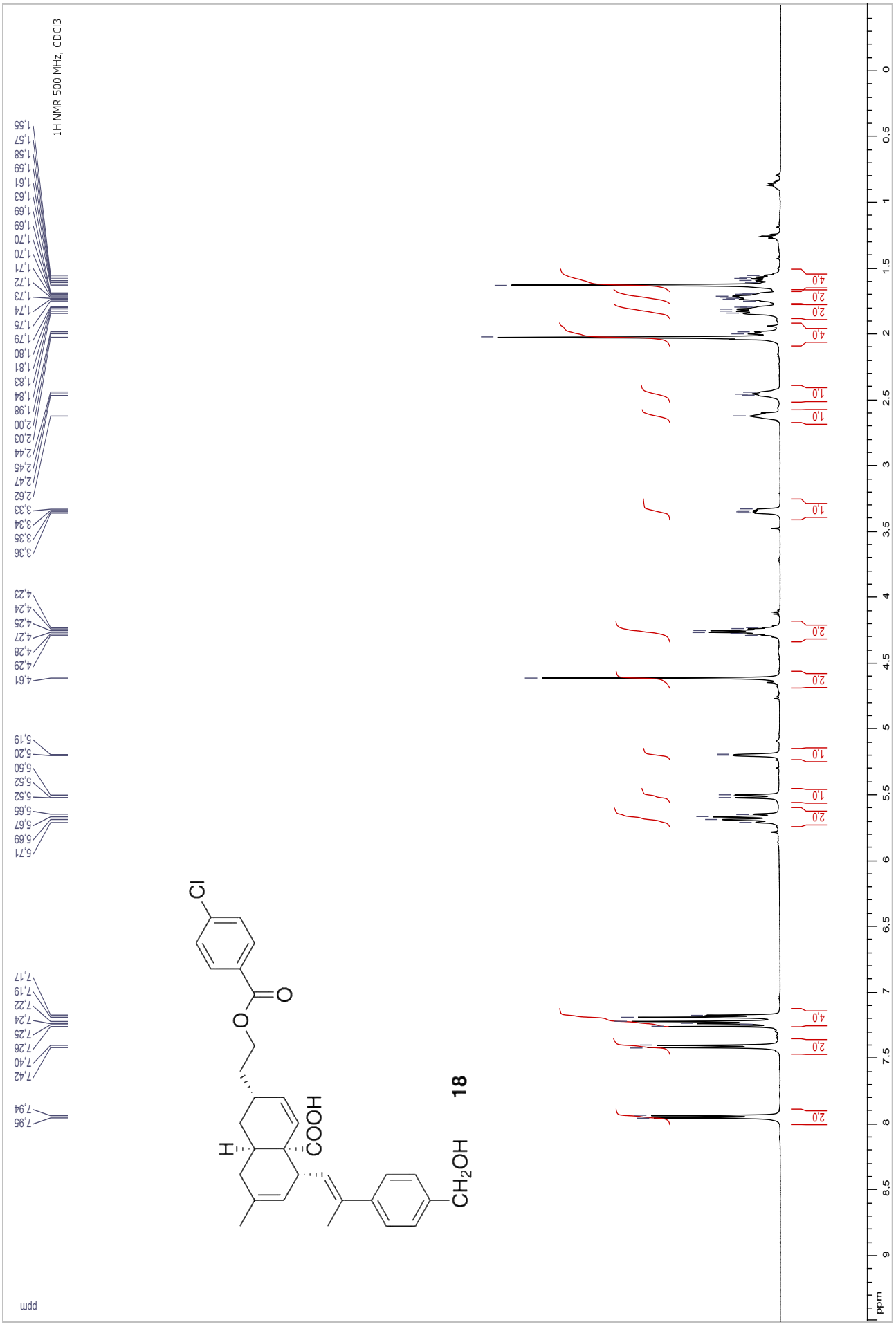

13C NMR 125 MHz, CDCl3
27.15
28.46
33.27
33.34
36.70
38.73
61.74
76.74
77.00
77.26
129.41
153.65
178.40
199.22
ppm
OPiv
5f
O

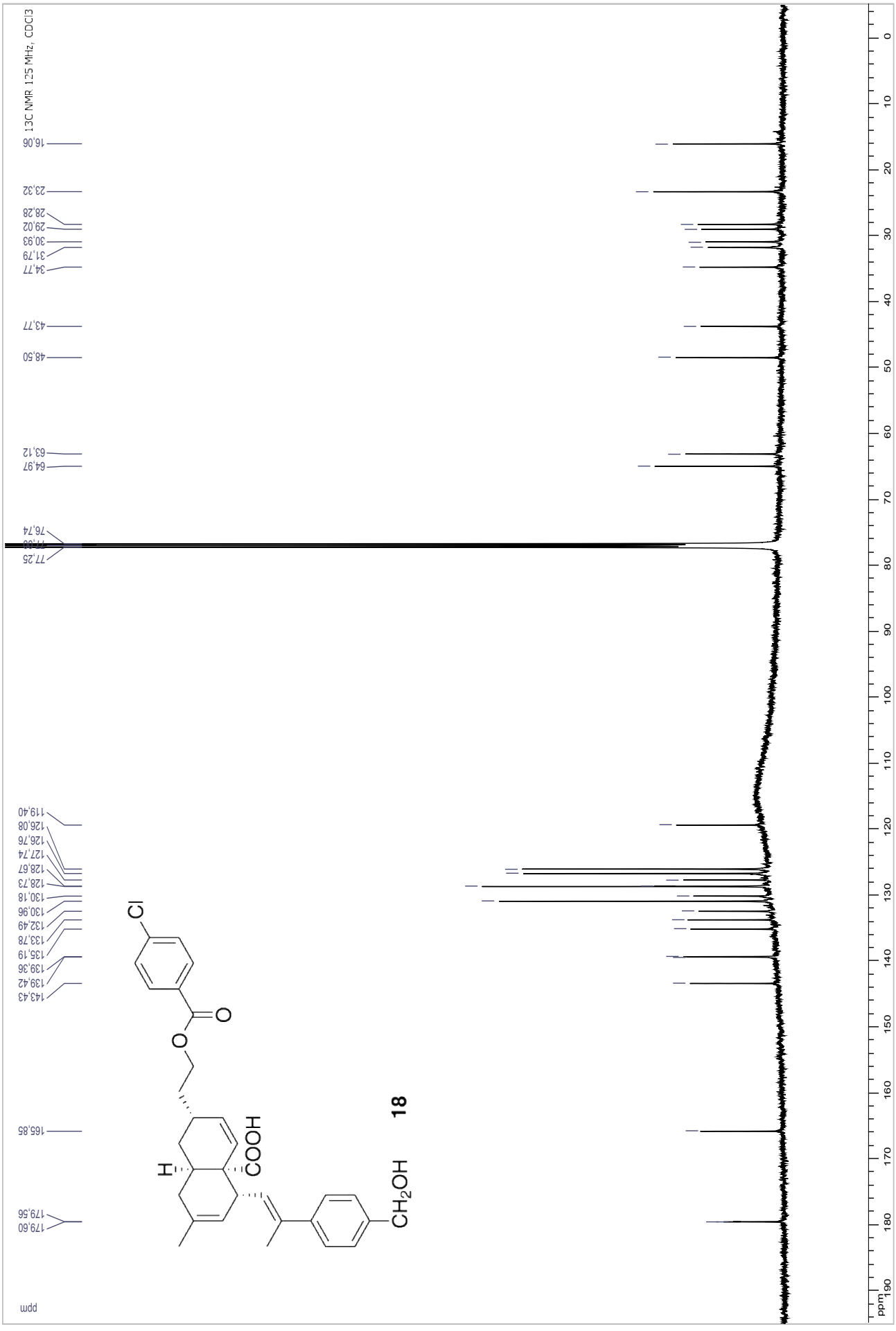

1H NMR 500 MHz, CDCl3
OBn
5g
O
ppm
3.6
0.9
0.7
1.9
2.0
0.9
1.0
1.0
1.1
1.0
2.1

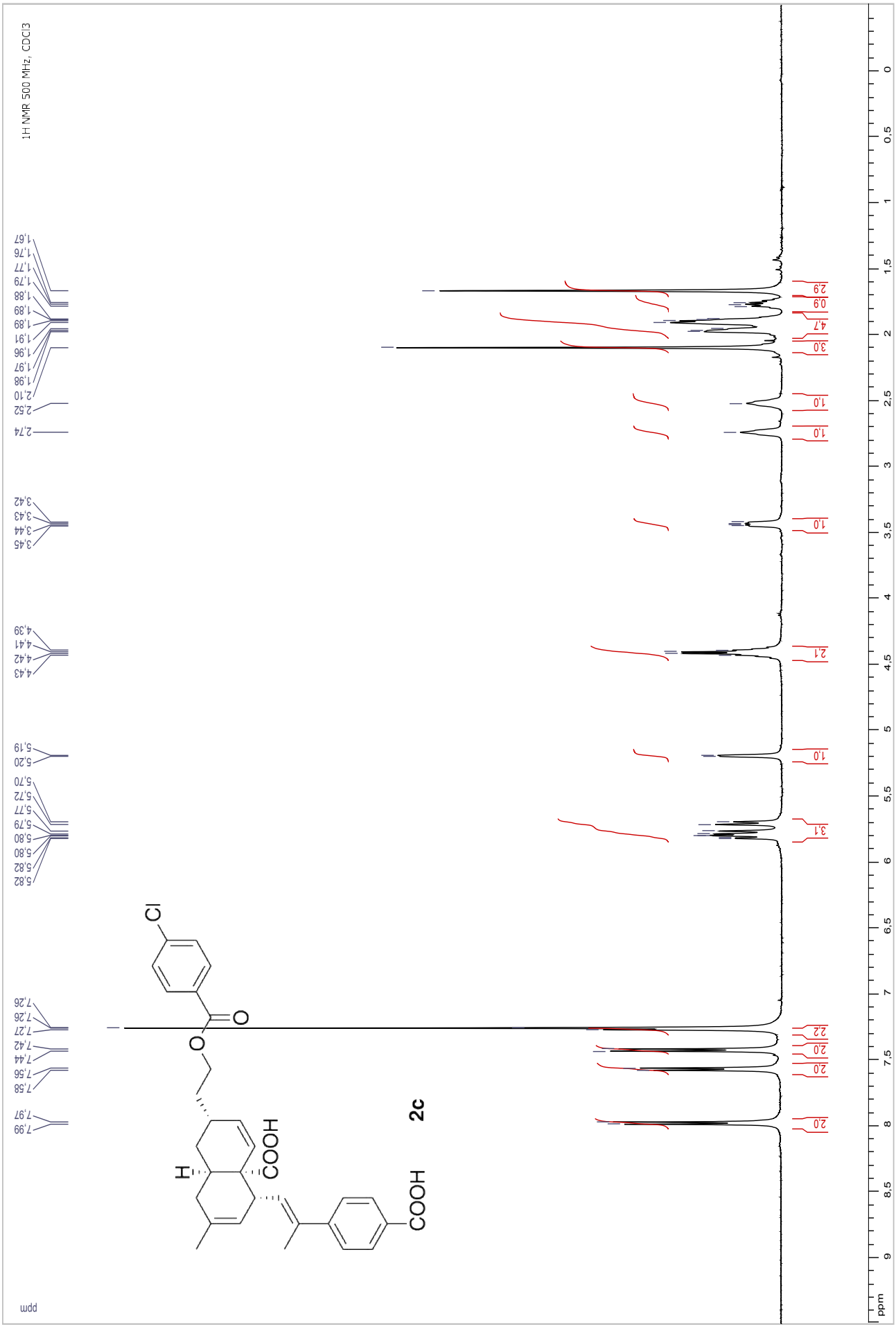

13C NMR 125 MHz, CDCl3
28.61
33.28
34.45
36.83
67.43
73.12
76.74
77.00
77.26
127.63
127.69
128.33
128.42
129.02
138.17
154.77
199.69
ppm
OBn
5g
O

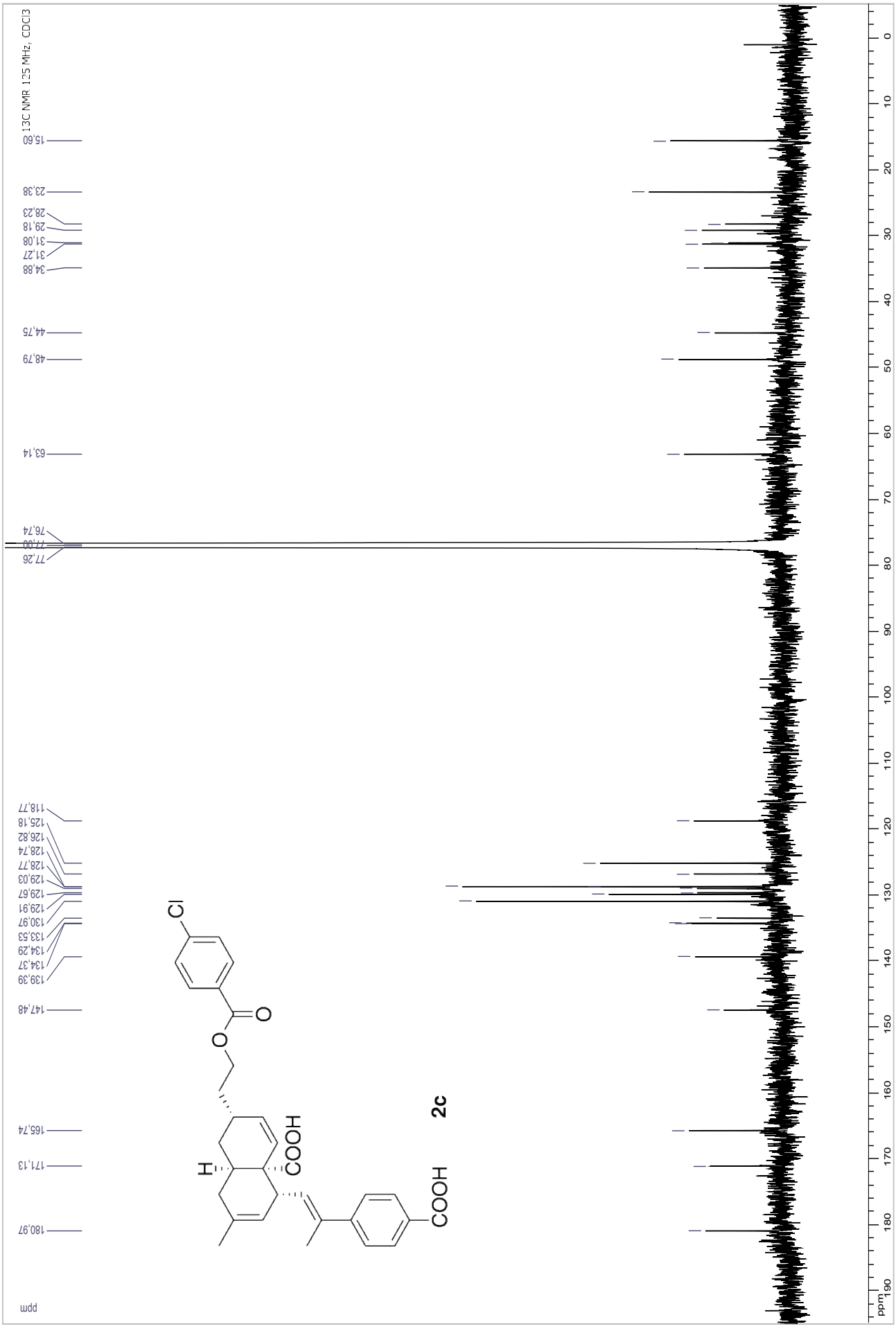

1H-NMR 500 MHz, CDCl3
OTrt
5h
O
ppm

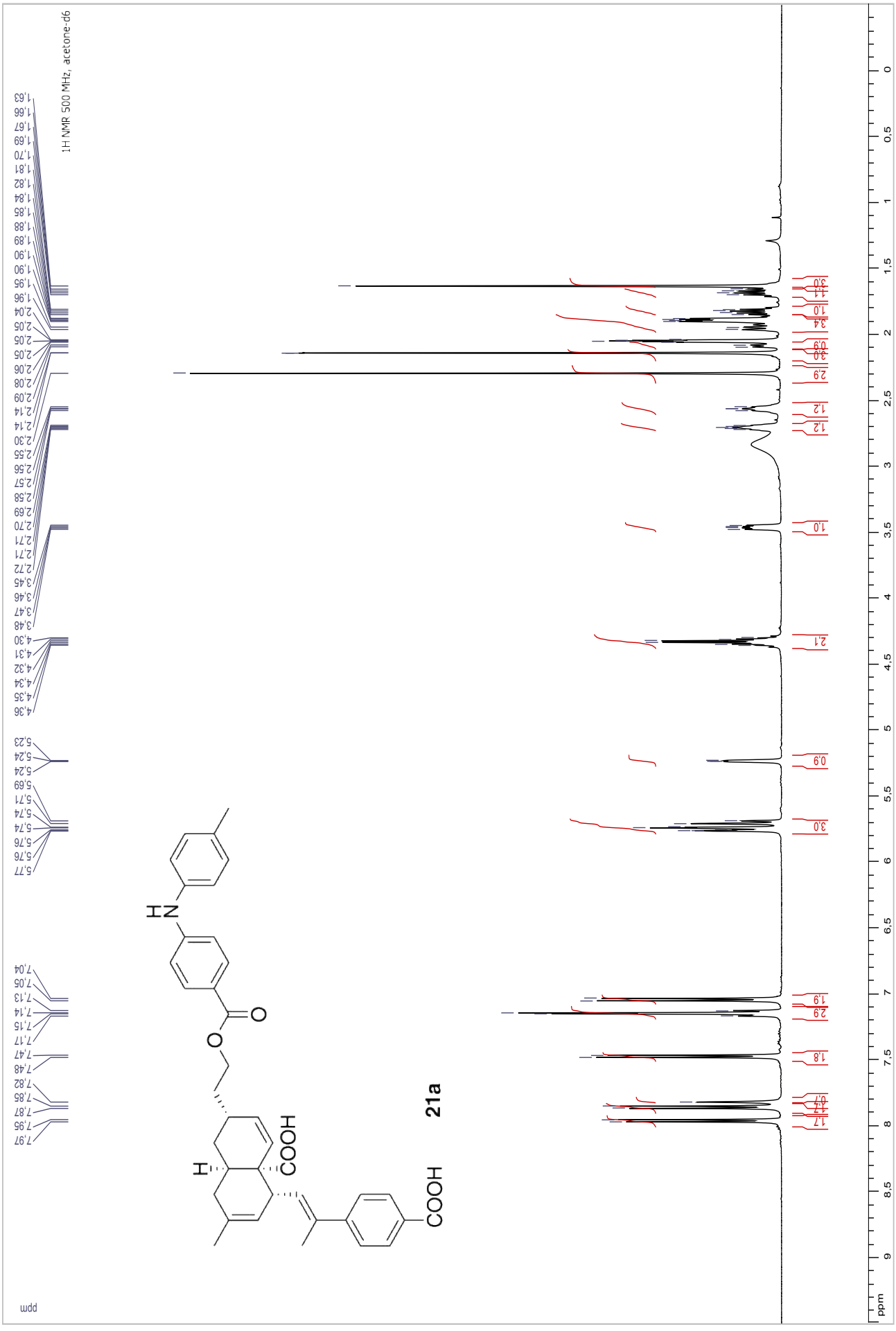

13C NMR 125 MHz, CDCl3
28.55
33.25
34.72
36.79
60.74
76.75
77.00
77.26
86.71
127.01
127.80
128.58
128.93
144.09
154.88
199.70
ppm
OTrt
5h
O

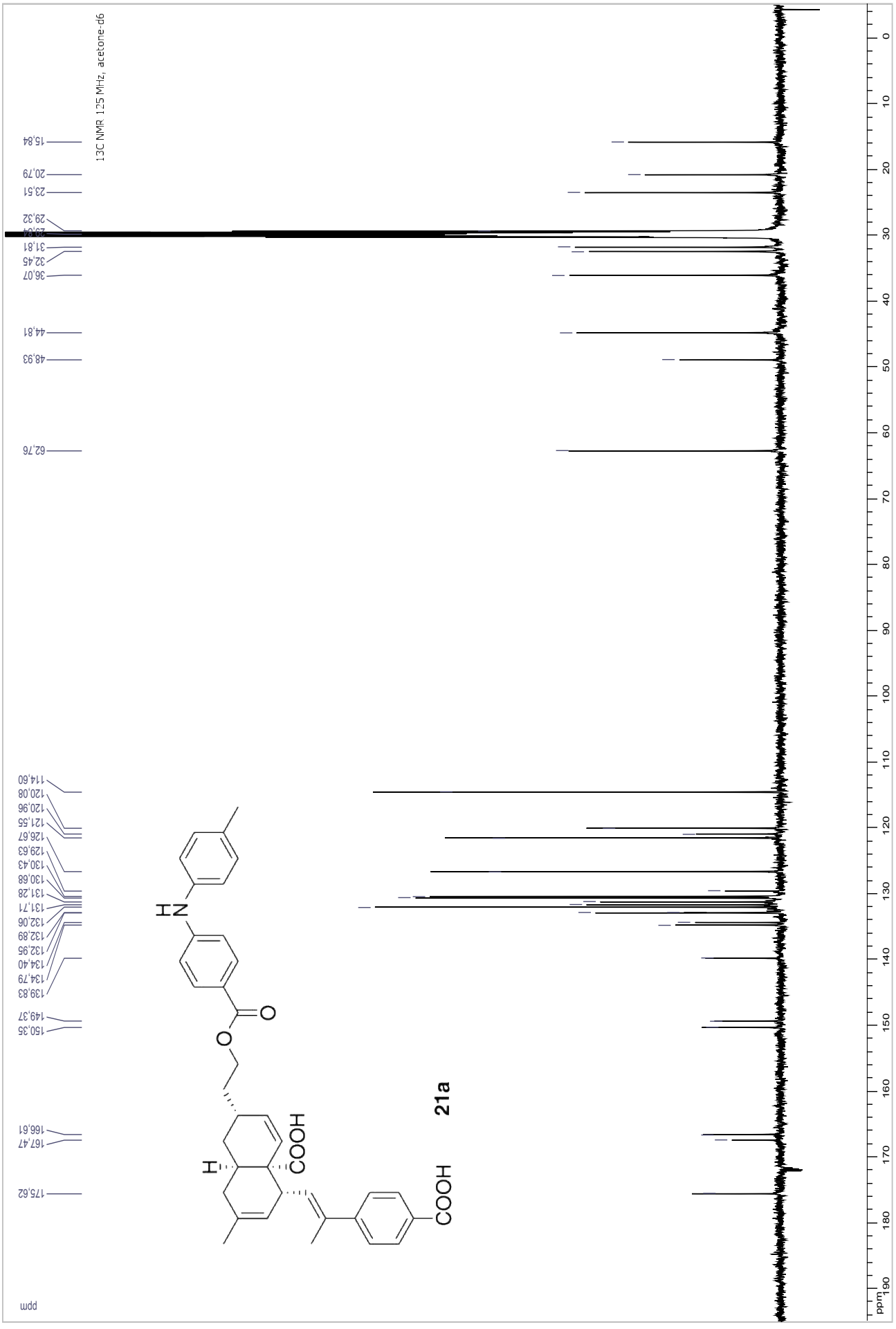

1H NMR 500 MHz, CDCl3
18
Cl
O
O
H
COOH
CH2OH
ppm

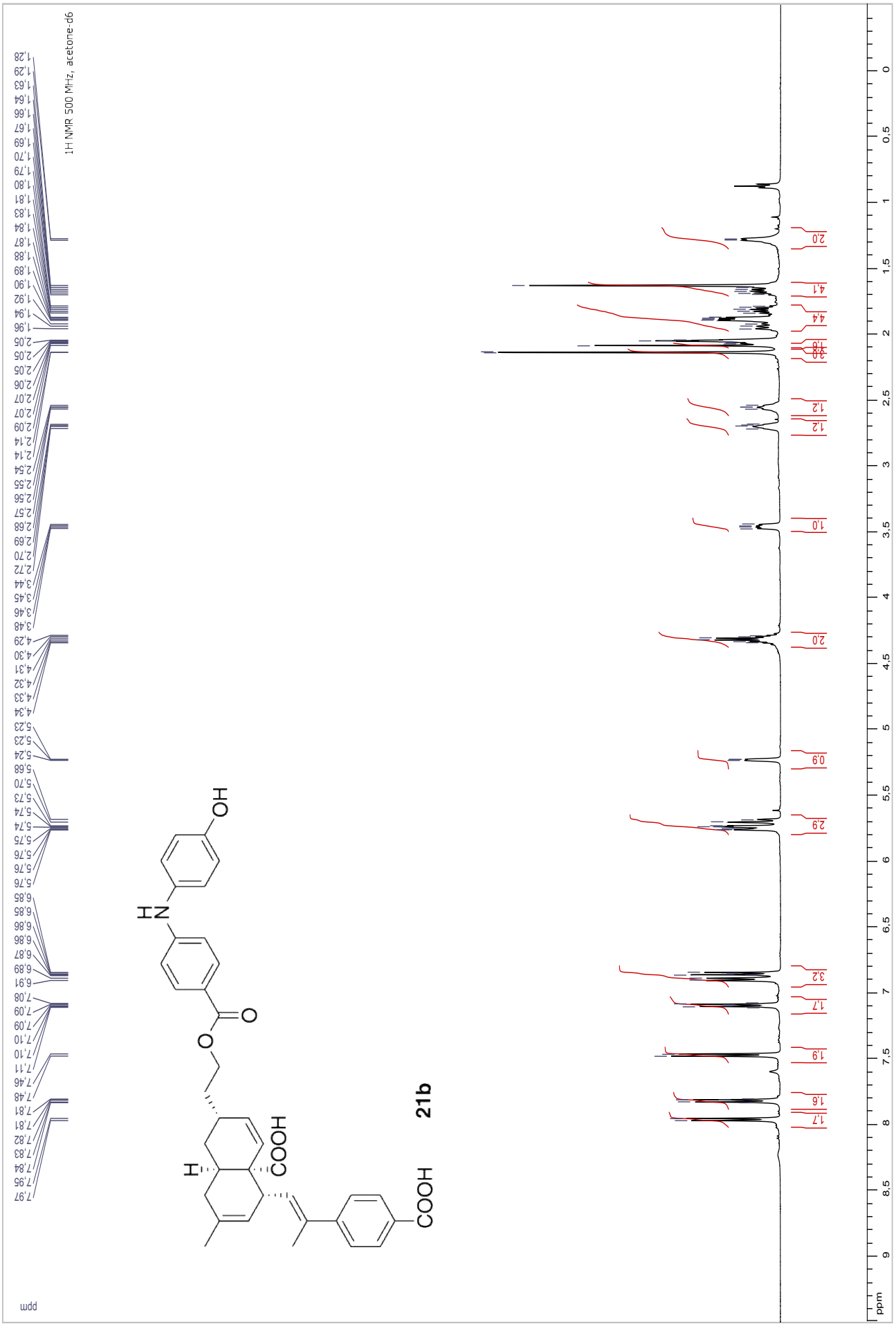

13C NMR 125 MHz, CDCl3
16,06
23,32
28,28
29,02
30,93
31,79
34,77
43,77
48,50
63,12
64,97
76,74
77,25
119,40
126,08
126,76
127,74
128,67
128,73
130,18
130,96
132,49
133,78
135,19
139,36
139,42
143,43
165,85
179,56
179,60
ppm
Cl
O
O
H
COOH
CH2OH
18

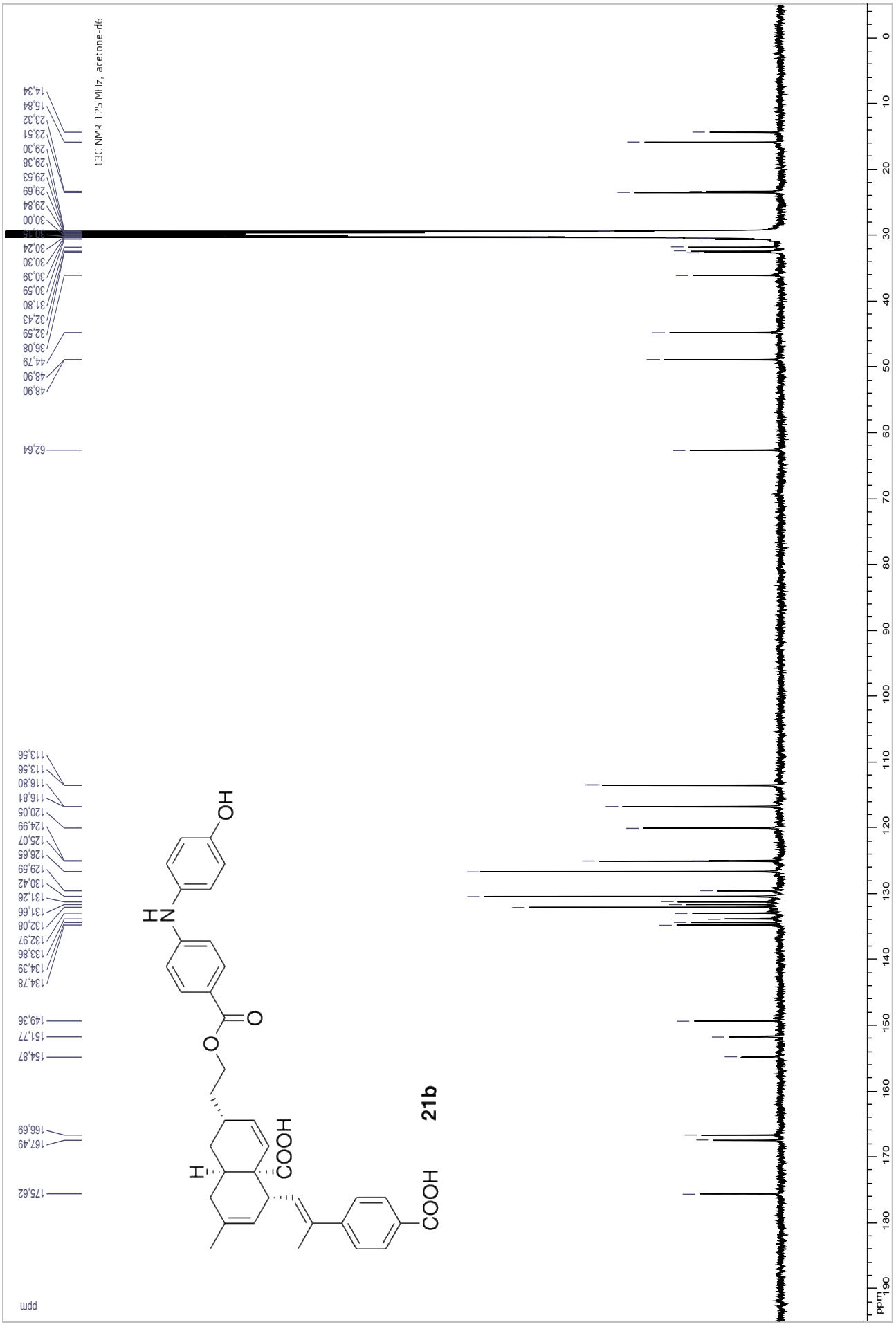

1H NMR 500 MHz, CDCl3
ppm
2c
Cl
O
O
H
COOH
COOH
1.67
1.76
1.77
1.79
1.88
1.89
1.89
1.91
1.96
1.97
1.98
2.10
2.52
2.74
3.42
3.43
3.44
3.45
4.39
4.41
4.42
4.43
5.19
5.20
5.70
5.72
5.77
5.79
5.80
5.80
5.82
5.82
7.26
7.26
7.27
7.42
7.44
7.56
7.58
7.97
7.99
2.9
0.9
4.7
3.0
1.0
1.0
1.0
2.1
1.0
3.1
2.2
2.0
2.0
2.0

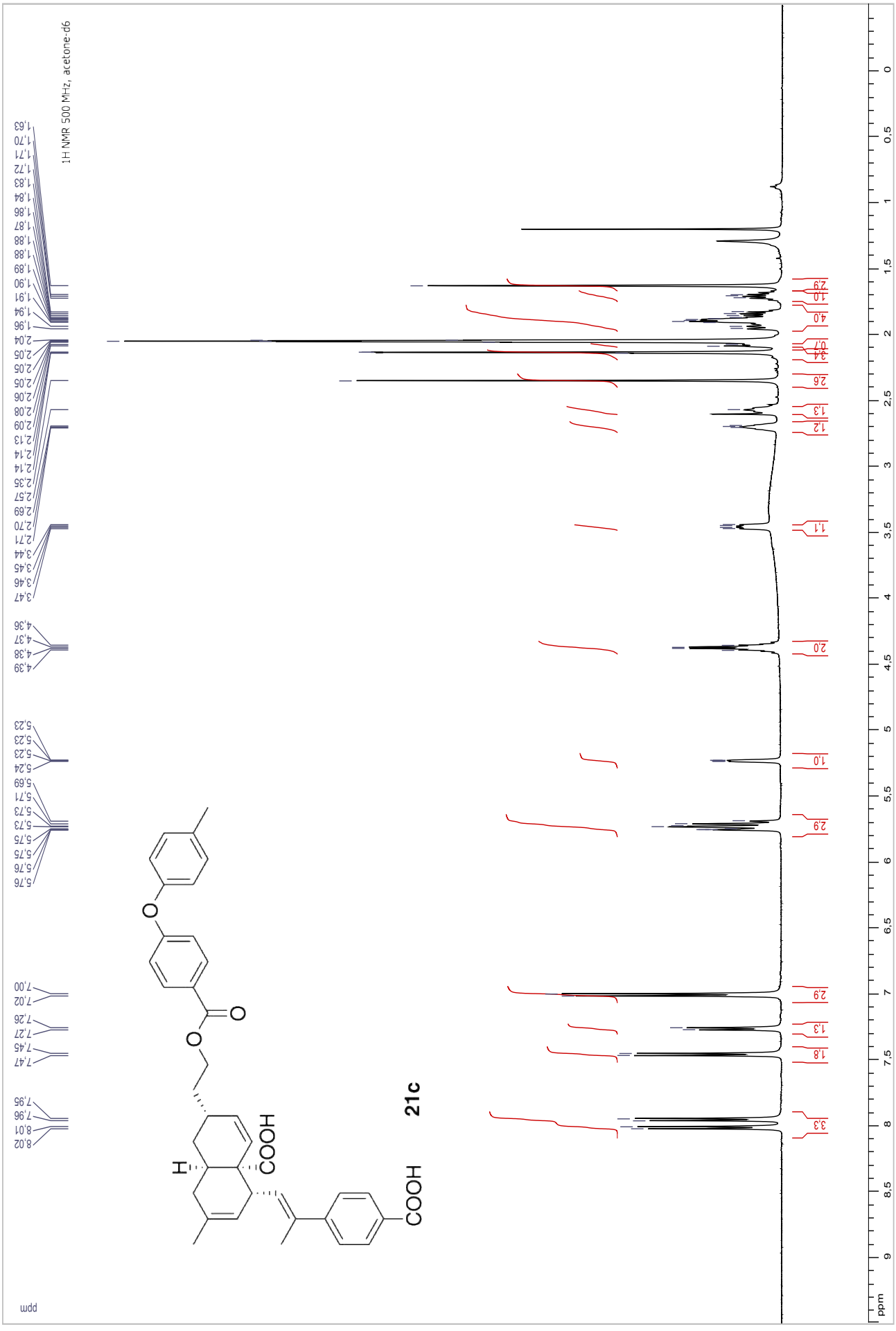

13C NMR 125 MHz, CDCl3
15.60
23.38
28.23
29.18
31.08
31.27
34.88
44.75
48.79
63.14
76.74
77.00
77.26
118.77
125.18
126.82
128.74
128.77
129.03
129.67
129.91
130.97
133.53
134.29
134.37
139.39
147.48
165.74
171.13
180.97
ppm
Cl
O
O
H
COOH
COOH
2c

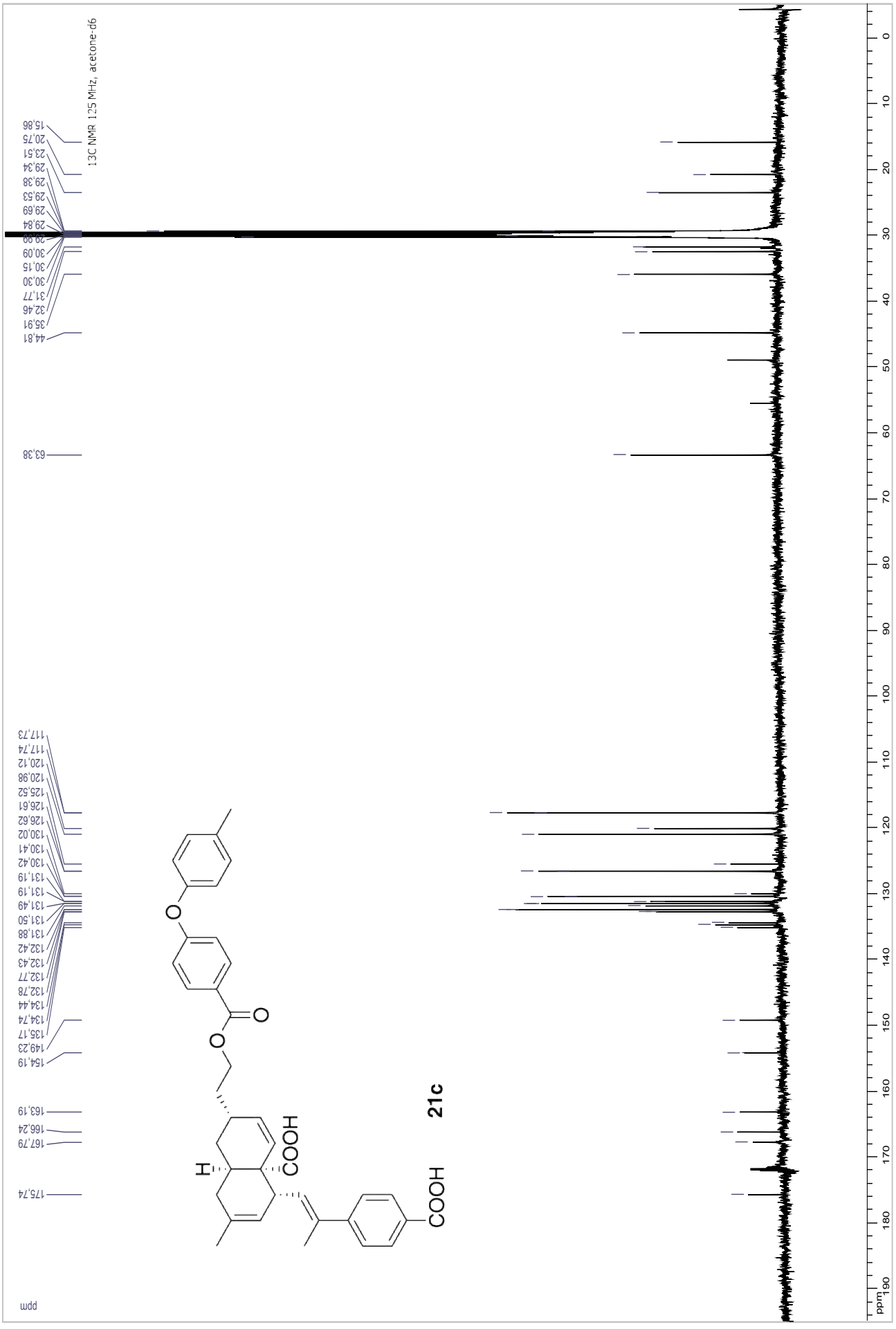

1H NMR 500 MHz, acetone-d6
21a
COOH
COOH
H
N
O
O
ppm

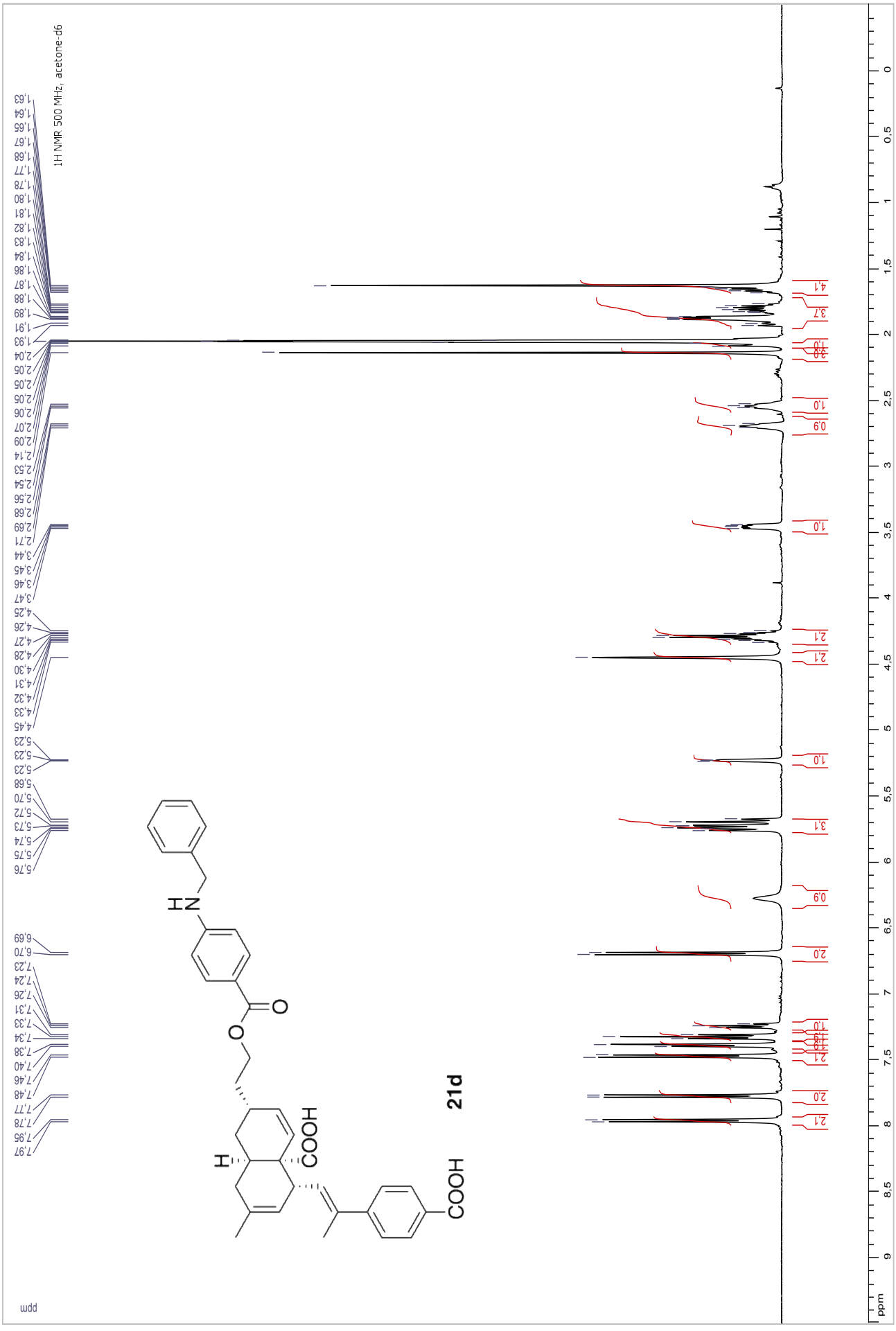

13C NMR 125 MHz, acetone-d6
15,84
20,79
23,51
29,32
29,84
31,81
32,45
36,07
44,81
48,93
62,76
114,60
120,08
120,96
121,55
126,67
129,63
130,43
130,68
131,28
131,71
132,06
132,89
132,95
134,40
134,79
139,83
149,37
150,35
166,61
167,47
175,62
ppm
21a
COOH
COOH
H
N
O
O

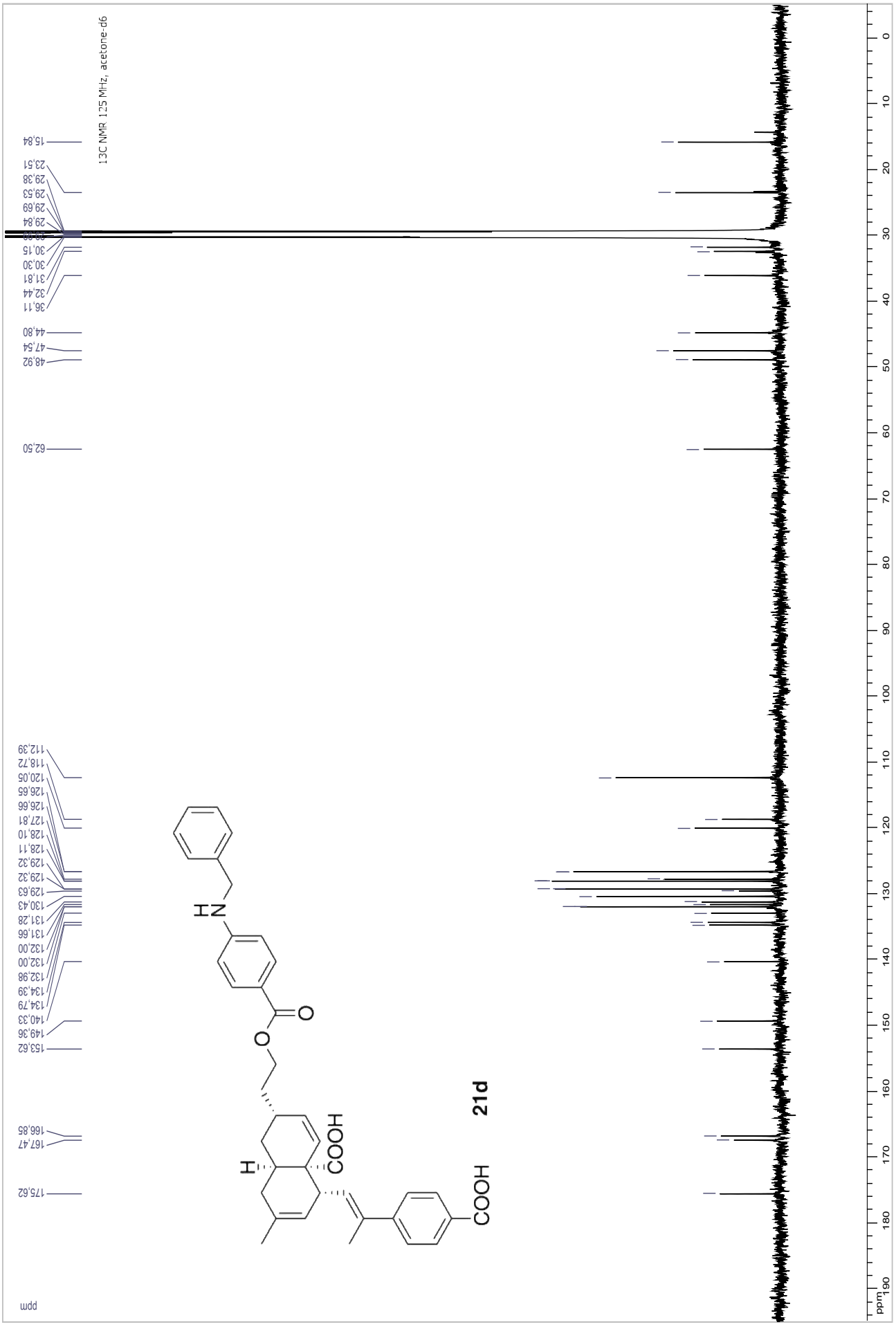

1H NMR 500 MHz, acetone-d6
21b
COOH
COOH
HO
N
H
O
O
ppm

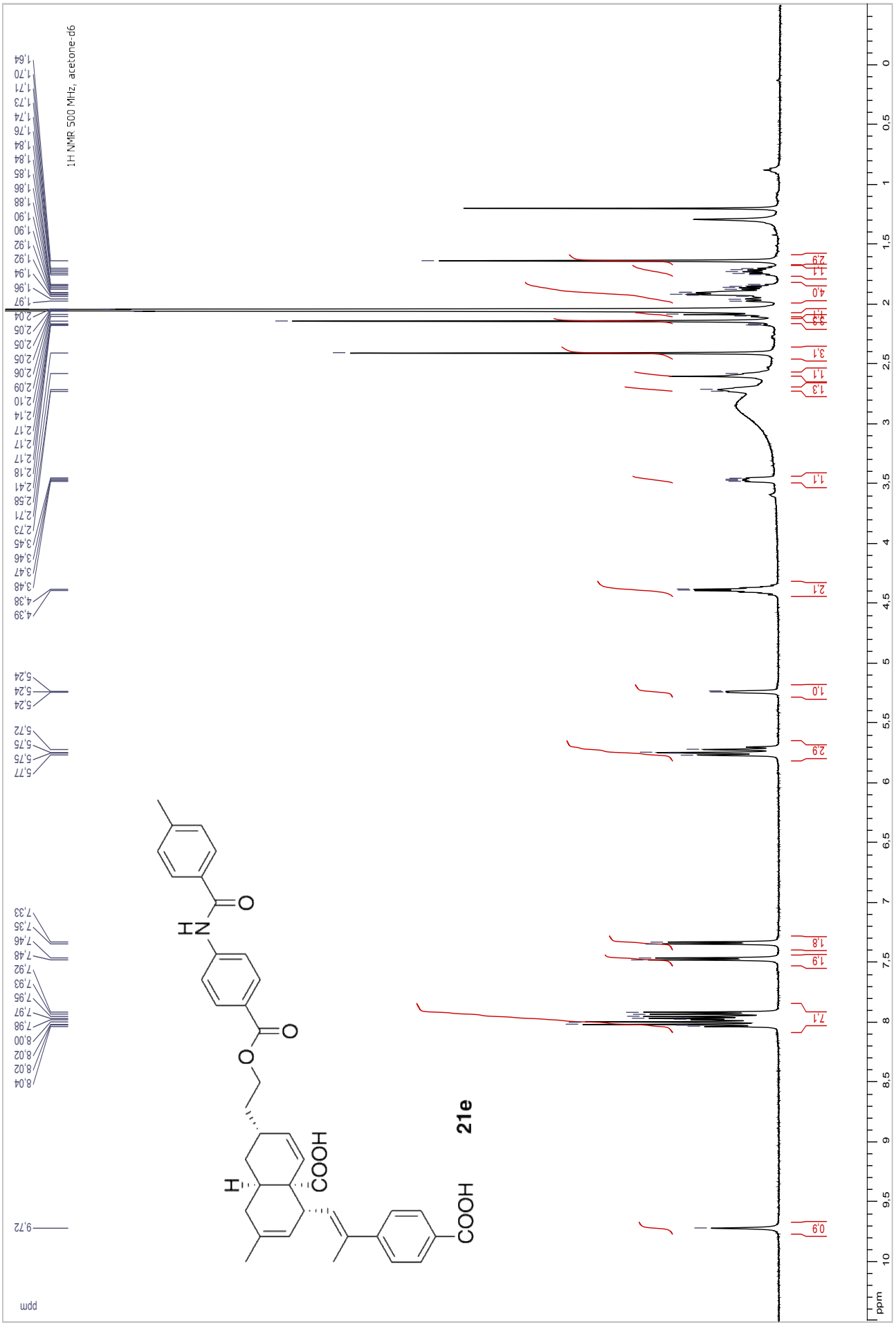

13C NMR 125 MHz, acetone-d6
14,34
15,84
23,32
23,51
29,30
29,38
29,53
29,69
29,84
30,00
30,24
30,30
30,39
30,59
31,80
32,43
32,59
36,08
44,79
48,90
48,90
62,64
113,56
113,56
116,80
116,81
120,05
124,99
125,07
126,65
129,59
130,42
131,26
131,66
132,08
132,97
133,86
134,39
134,78
149,36
151,77
154,87
166,69
167,49
175,62
ppm
0
10
20
30
40
50
60
70
80
90
100
110
120
130
140
150
160
170
180
190
ppm
OH
NH
O
O
H
COOH
COOH
21b

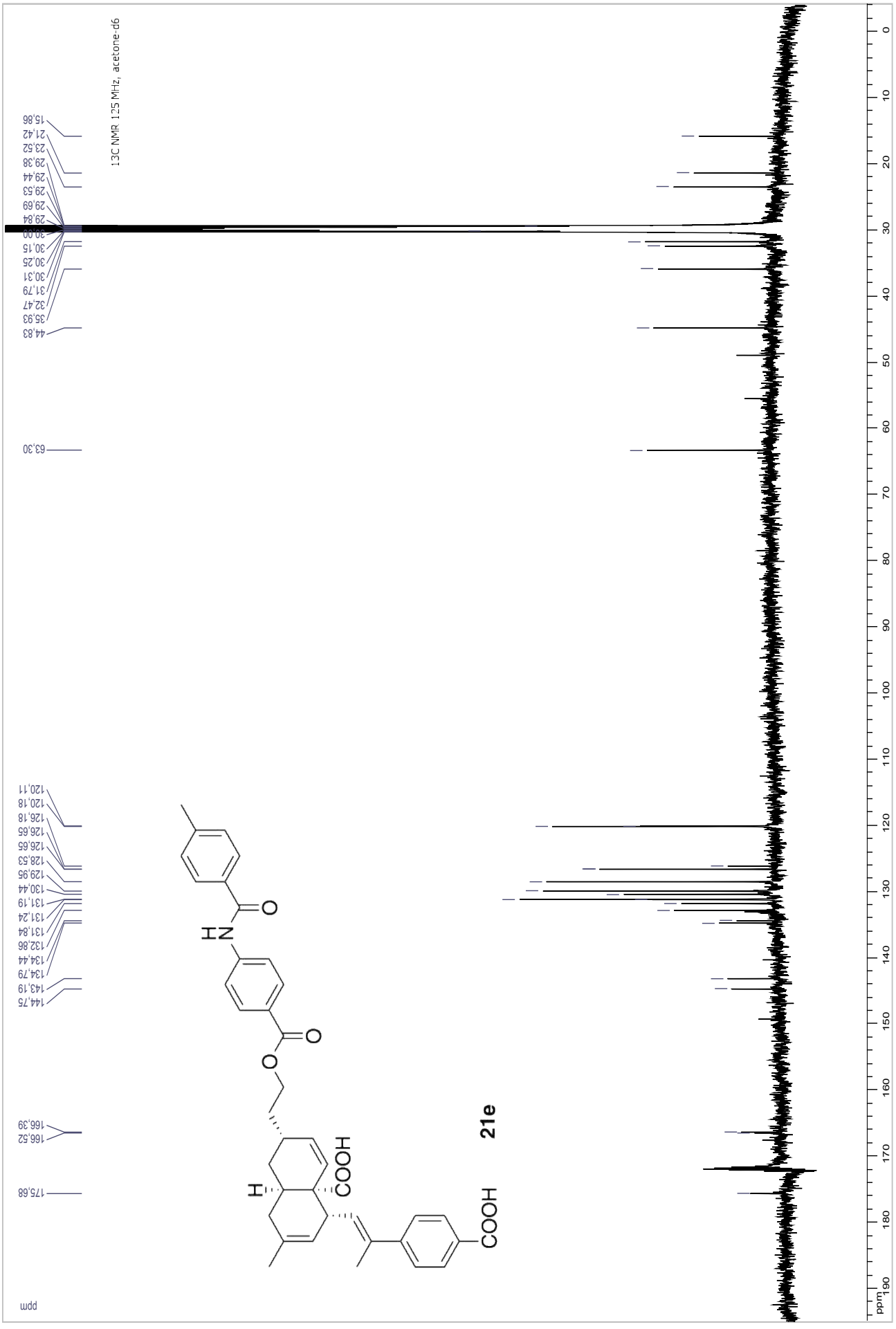

1H NMR 500 MHz, acetone-d6
21c
COOH
COOH
H
O
O
O
ppm


13C NMR 125 MHz, acetone-d6
15,86
20,75
23,51
29,34
29,38
29,53
29,69
29,84
29,99
30,09
30,15
30,30
31,77
32,46
35,91
44,81
63,38
117,73
117,74
120,12
120,98
125,52
126,61
126,62
130,02
130,41
130,42
131,19
131,19
131,49
131,50
131,88
132,42
132,43
132,77
132,78
134,44
134,74
135,17
149,23
154,19
163,19
166,24
167,79
175,74
ppm
H
COOH
COOH
21c


1H NMR 500 MHz, acetone-d6
21d
COOH
COOH
NH
O
O
H
ppm


13C NMR 125 MHz, acetone-d6
15.84
23.51
29.38
29.53
29.69
29.84
30.15
30.30
31.81
32.44
36.11
44.80
47.54
48.92
62.50
112.39
118.72
120.05
126.65
126.66
127.81
128.10
128.11
129.32
129.32
129.63
130.43
131.28
131.66
132.00
132.00
132.98
134.39
134.79
140.33
149.36
153.62
166.85
167.47
175.62
ppm
NH
O
O
H
COOH
COOH
21d


1H NMR 500 MHz, acetone-d6
21e
COOH
COOH
H
NH
O
O
O
ppm


13C NMR 125 MHz, acetone-d6
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29.53
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29.84
30.00
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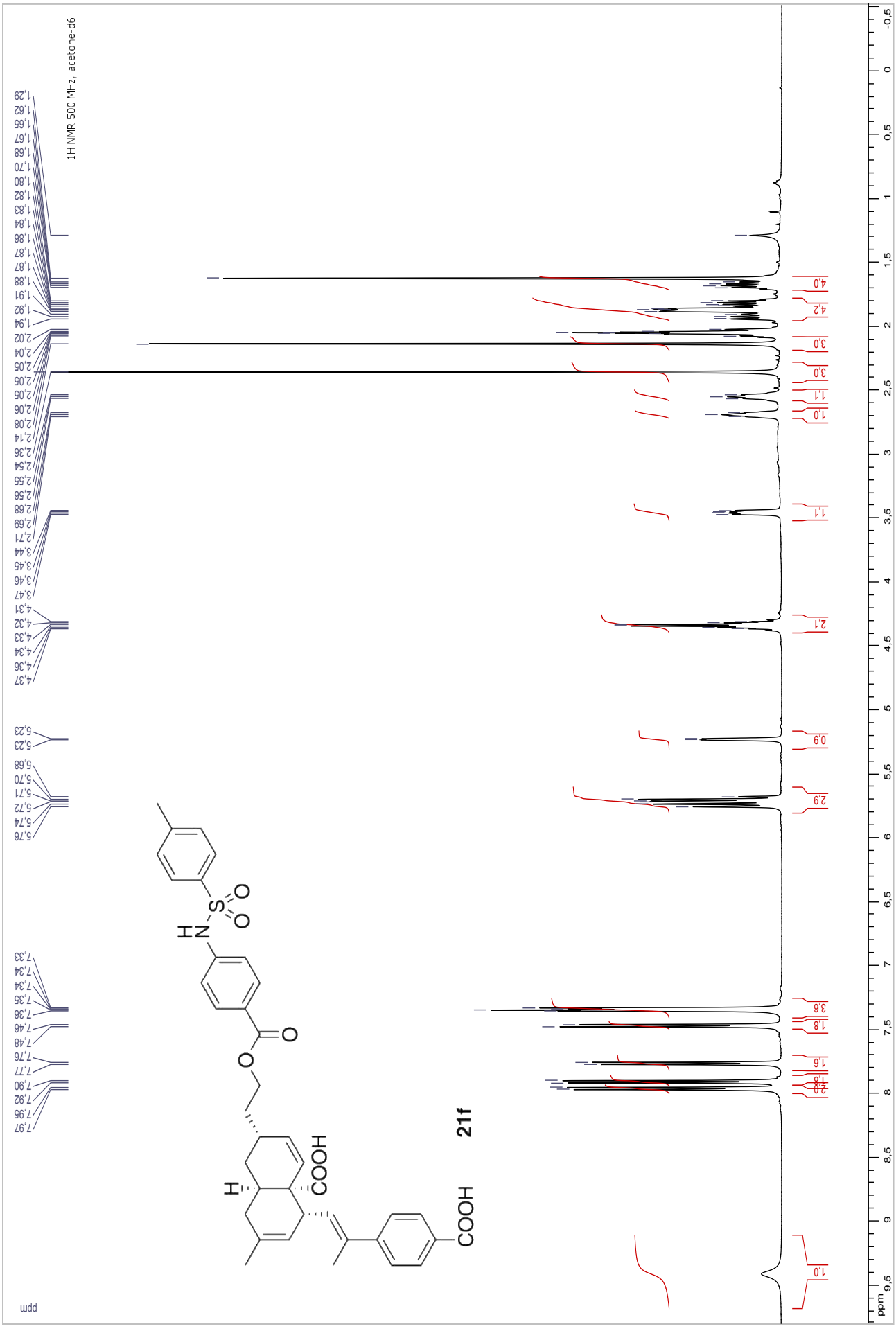
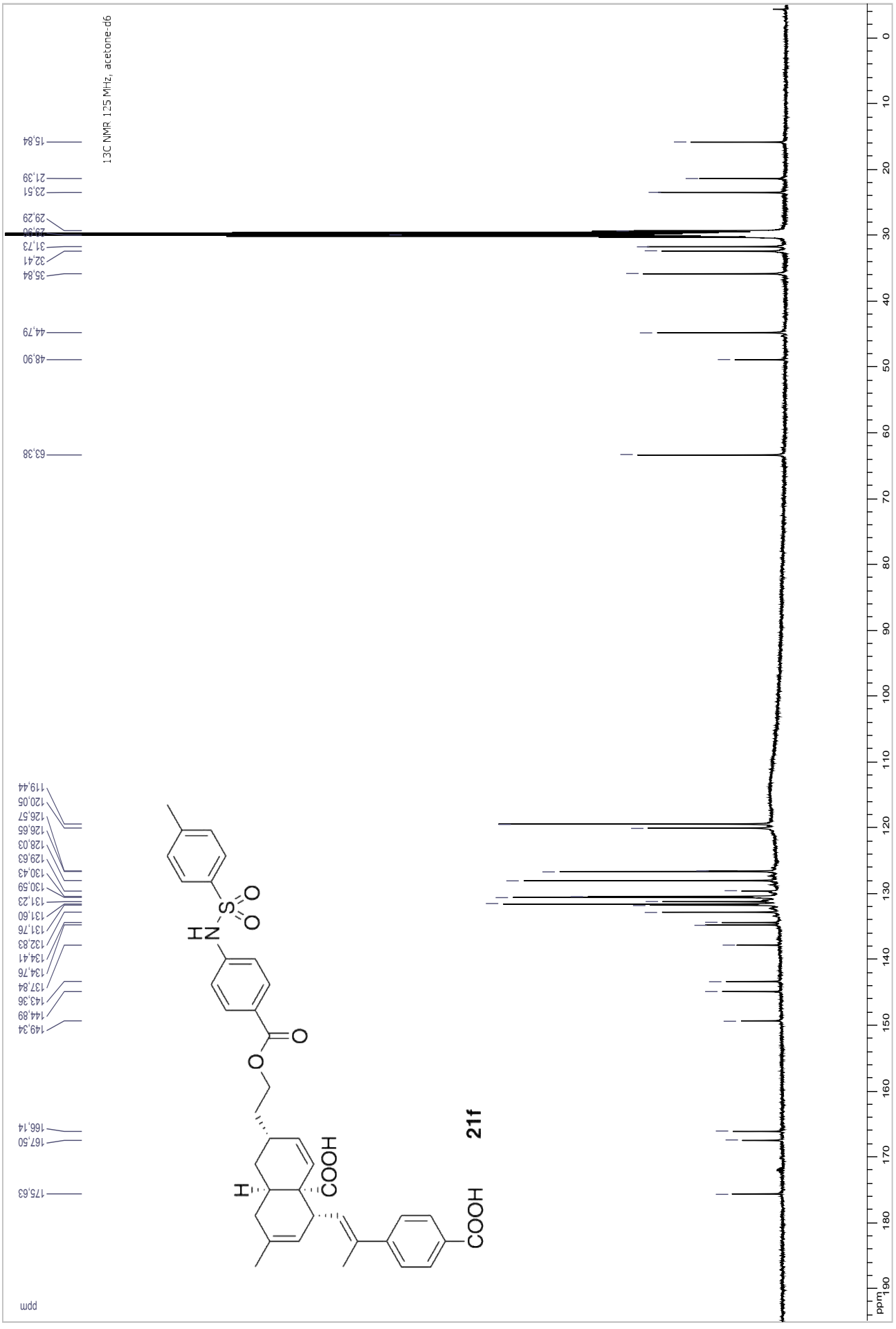
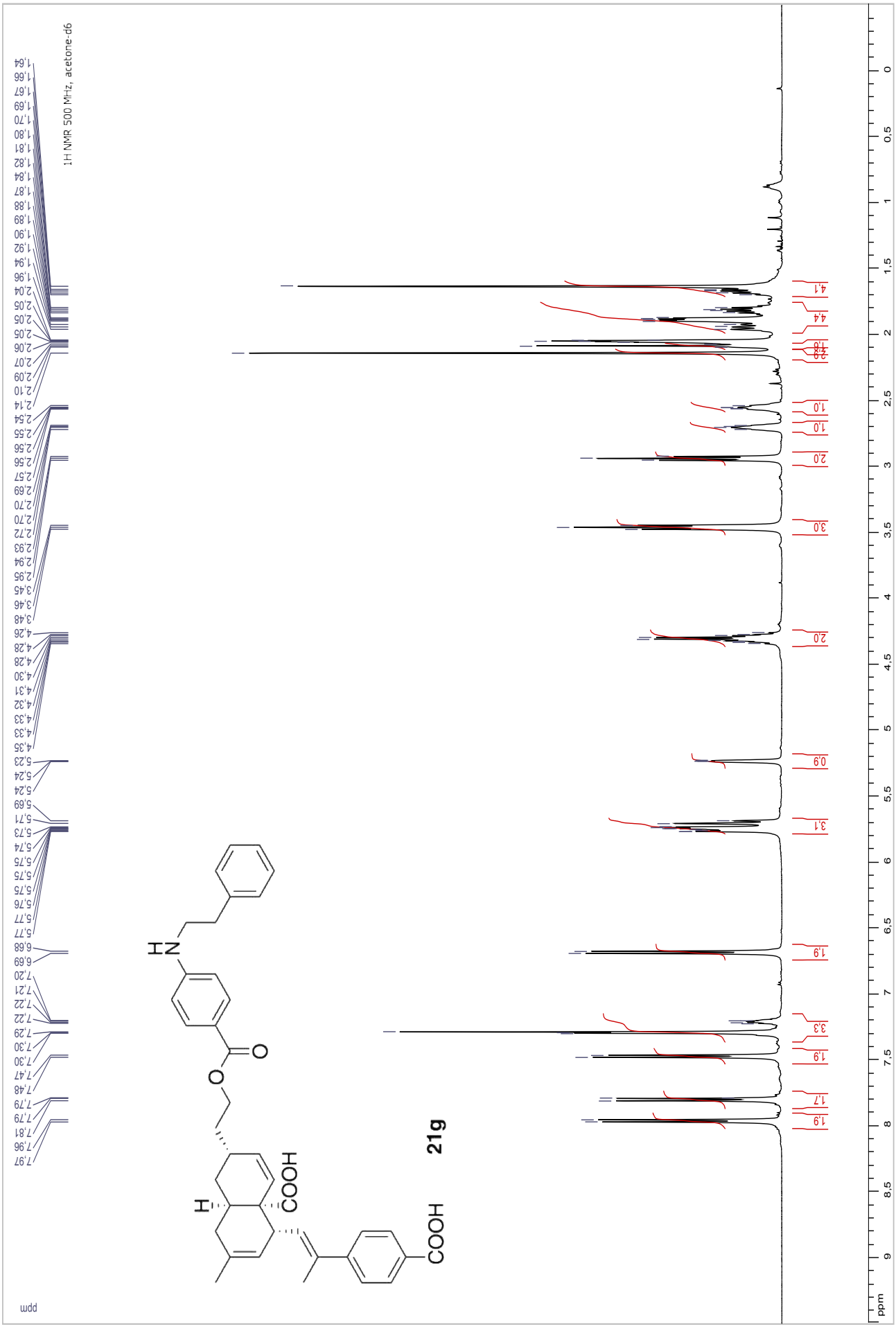
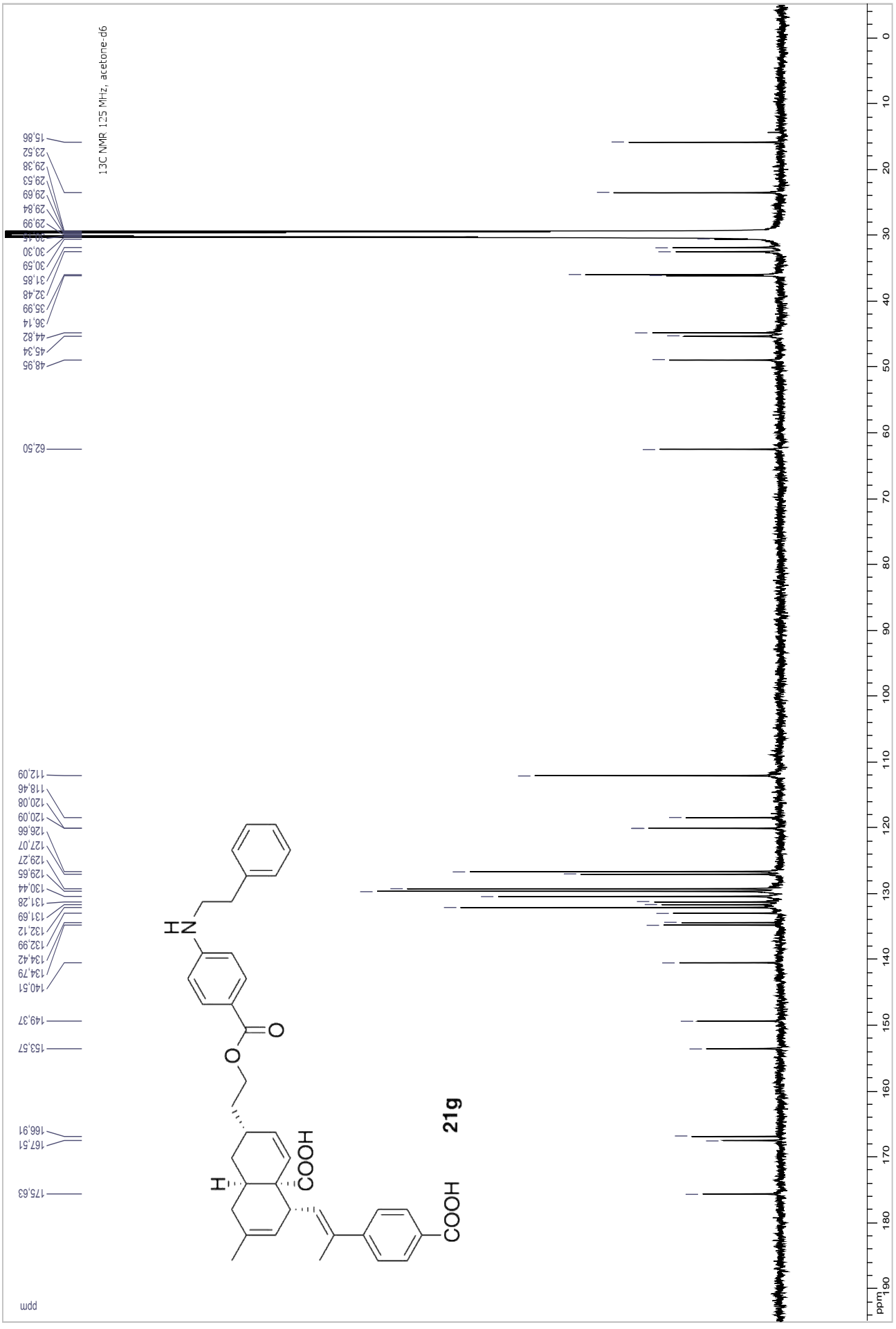
30.25
30.31
31.79
32.47
35.93
44.83
63.30
120.11
120.18
126.18
126.65
126.65
128.53
129.95
130.44
131.19
131.24
131.84
132.86
134.44
134.79
143.19
144.75
166.39
166.52
175.68
ppm
COOH
COOH
21e


1H NMR 500 MHz, acetone-d6
21f
COOH
COOH
H
N
H
O
O
S
O
O
ppm


13C NMR 125 MHz, acetone-d6
15,84
21,39
23,51
29,29
29,90
31,73
32,41
35,84
44,79
48,90
63,38
119,44
120,05
126,57
126,65
128,03
129,63
130,43
130,59
131,23
131,60
131,76
132,83
134,41
134,76
137,84
143,36
144,89
149,34
166,14
167,50
175,63
ppm
COOH
COOH
21f


1H NMR 500 MHz, acetone-d6
21g
COOH
COOH
H
N
O
O
ppm

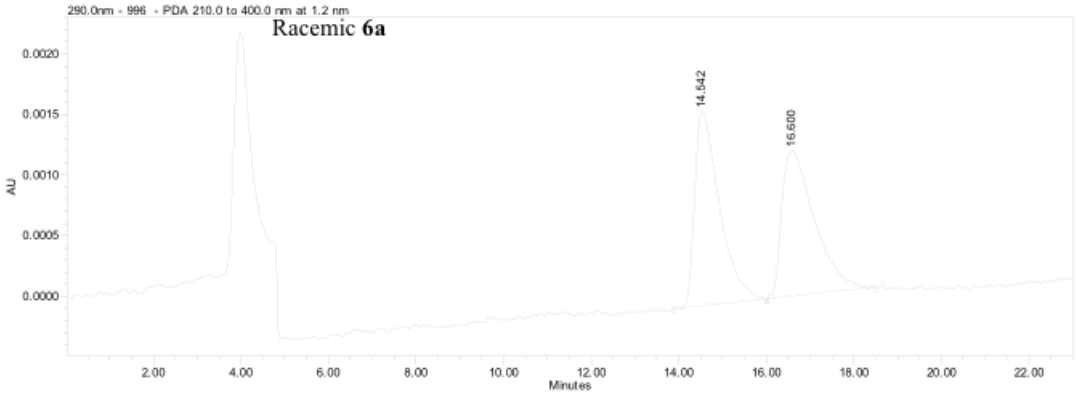

13C NMR 125 MHz, acetone-d6
15,86
23,52
29,38
29,53
29,69
29,84
29,99
30,15
30,30
30,59
31,85
32,48
35,99
36,14
44,82
45,34
48,95
62,50
112,09
118,46
120,08
120,09
126,66
127,07
129,27
129,65
130,44
131,28
131,69
132,12
132,99
134,42
134,79
140,51
149,37
153,57
166,91
167,51
175,63
ppm
21g
COOH
COOH
H
N
O
O

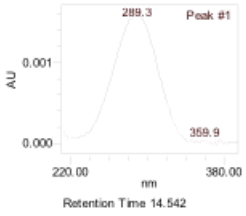
Chiral HPLC Data

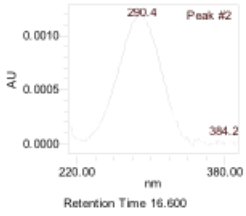
Synthesis of chiral 2-cyclohexenone 5 by intermolecular aldol condensation

Compound 6a

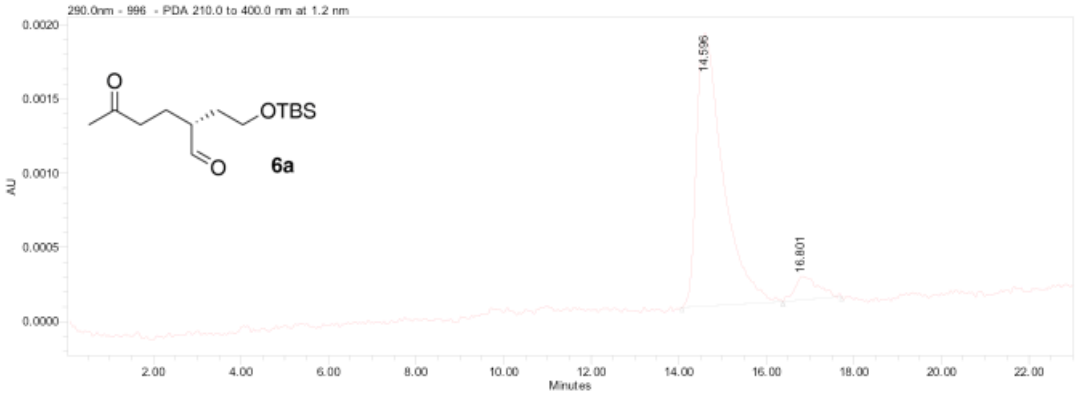
The *e.r.* of the keto-aldehyde **6a** was measured using a Daicel Chiralpal® IA column (5 µm, 4.6 x 250 mm) with a U.V. detection at 290 nm and at 25 °C. Flow = 1 mL/min with heptane/isopropanol 99.5:0.5.

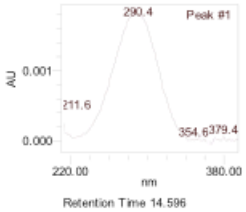


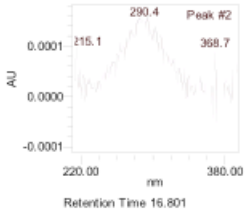




	Channel Description	RT	Area	% Area
1	PDA 210.0 to 400.0 nm at 1.2 nm	14.542	63255	50.63
2	PDA 210.0 to 400.0 nm at 1.2 nm	16.600	61687	49.37





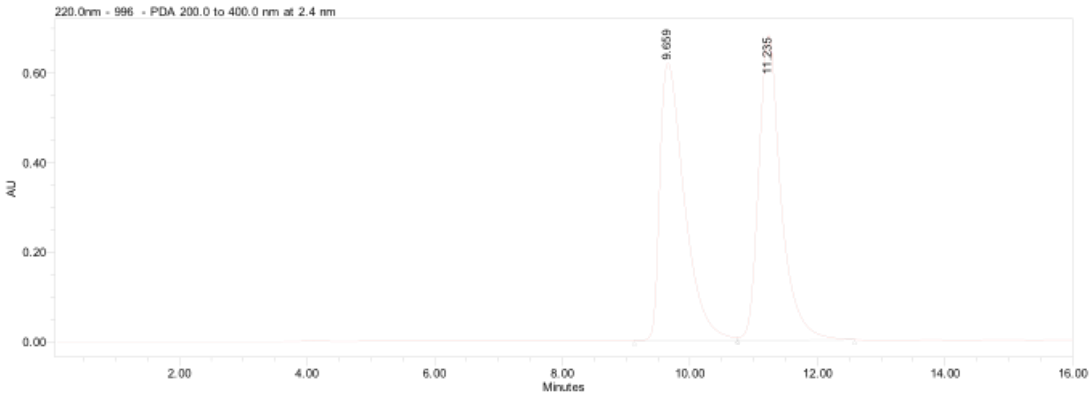


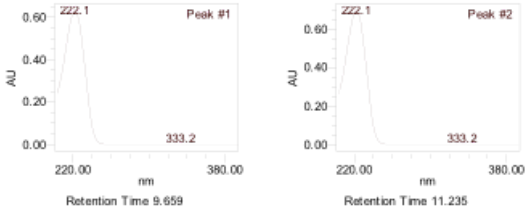
	Channel Description	RT	Area	% Area
1	PDA 210.0 to 400.0 nm at 1.2 nm	14.596	76938	92.95
2	PDA 210.0 to 400.0 nm at 1.2 nm	16.801	5840	7.05

Compound 5a

The *e.r.* of the 2-cyclohexenone **5a** was measured using a Daicel Chiralpal® IA column (5 µm, 4.6 x 250 mm) with a U.V. detection at 220 nm and at 25 °C. Flow = 1 mL/min with heptane/isopropanol 99.5:0.5.

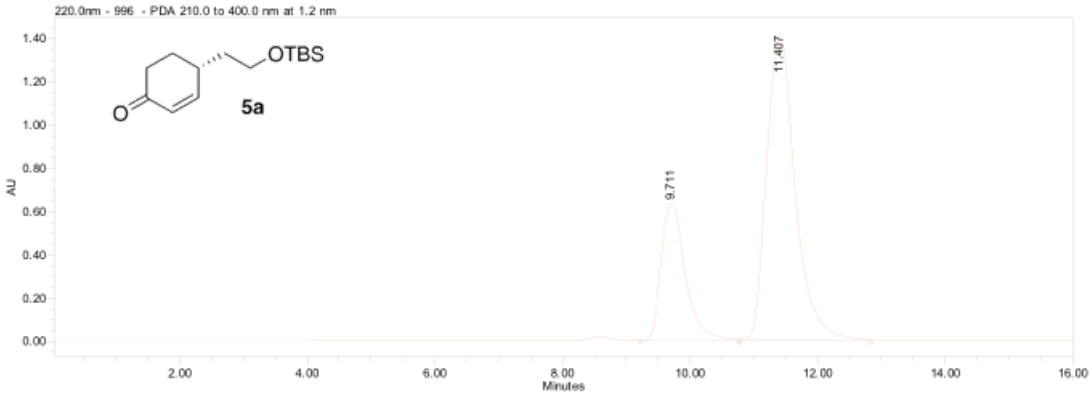
Racemic **5a**

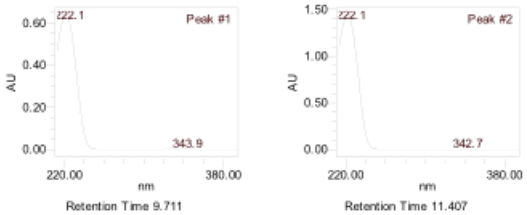




	Channel Description	RT	Area	% Area
1	PDA 200.0 to 400.0 nm at 2.4 nm	9.659	16587257	50.01
2	PDA 200.0 to 400.0 nm at 2.4 nm	11.235	16579553	49.99

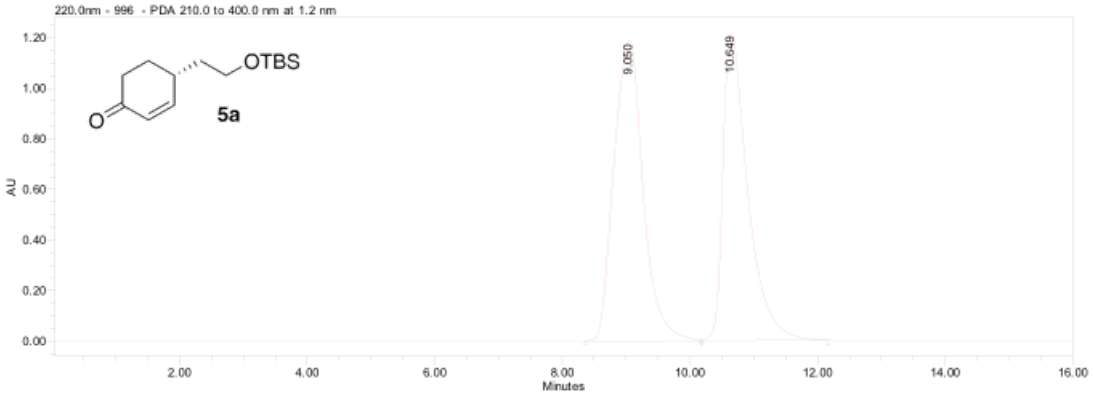
Conditions of Table1, entry 1 (LiOH)

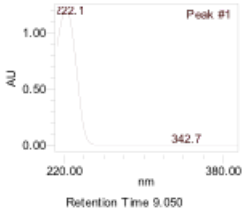


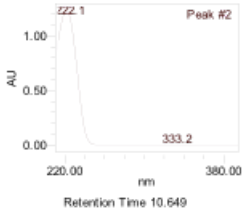


	Channel Description	RT	Area	% Area
1	PDA 210.0 to 400.0 nm at 1.2 nm	9.711	16464851	27.33
2	PDA 210.0 to 400.0 nm at 1.2 nm	11.407	43783513	72.67

Conditions of Table 1, entry 5 (McQuade's condition)





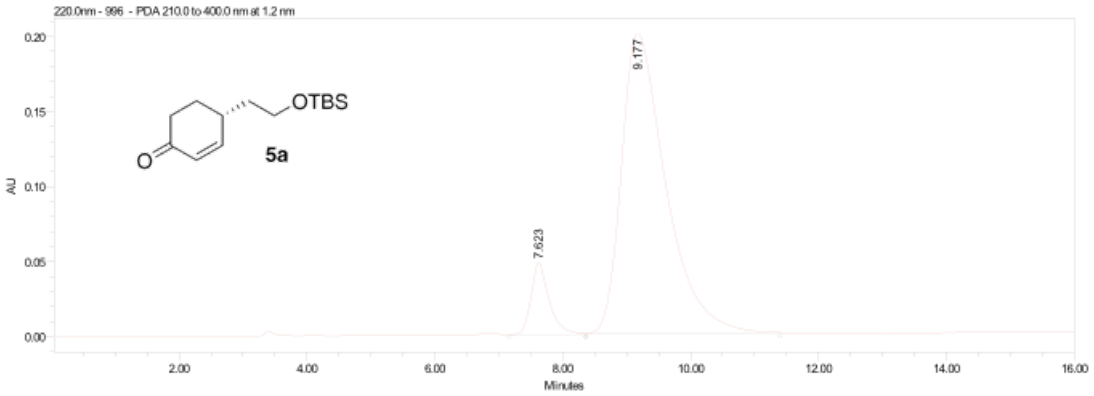


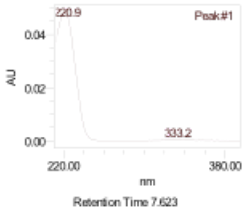
	Channel Description	RT	Area	% Area
1	PDA 210.0 to 400.0 nm at 1.2 nm	9.050	37635638	53.34
2	PDA 210.0 to 400.0 nm at 1.2 nm	10.649	32916565	46.66

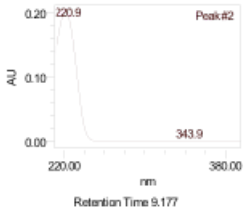
Synthesis of chiral 2-cyclohexenones 5a-c and 5e-h by desymmetrization

Compound 5a

The *e.r.* of the 2-cyclohexenone **5a** was measured using a Daicel Chiralpal® IA column (5 µm, 4.6 x 250 mm) with a U.V. detection at 220 nm and at 25 °C. Flow = 1 mL/min with heptane/isopropanol 99.5:0.5



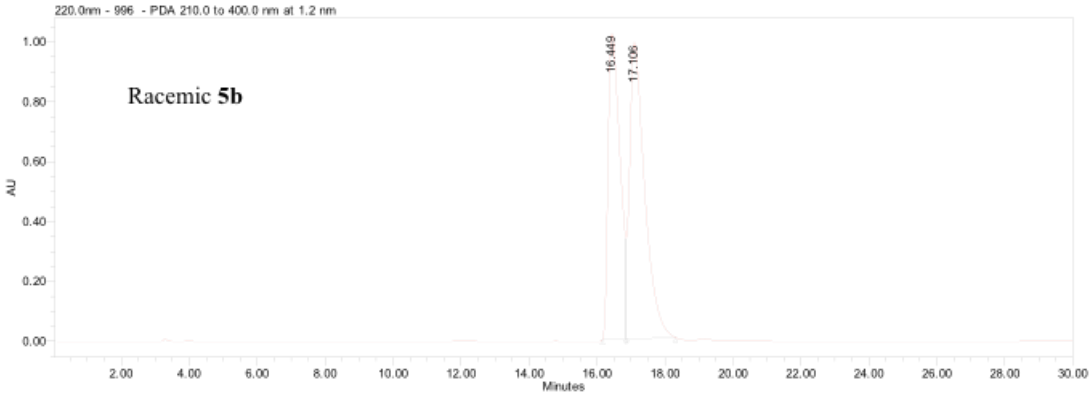


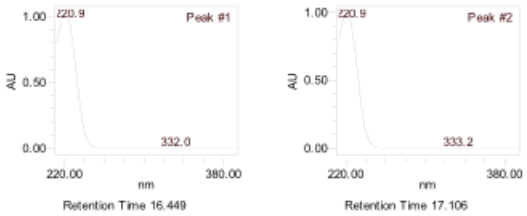


	Channel Description	RT	Area	% Area
1	PDA 210.0 to 400.0 nm at 1.2 nm	7.623	936537	8.85
2	PDA 210.0 to 400.0 nm at 1.2 nm	9.177	9645087	91.15

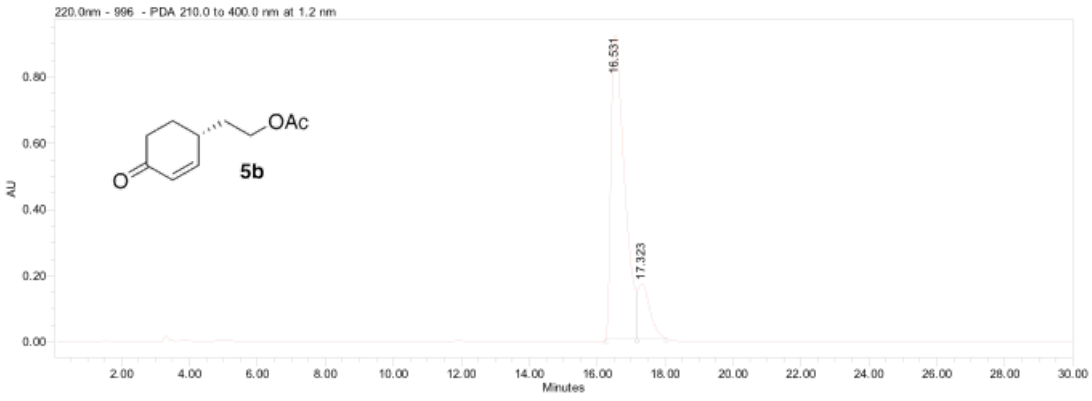
Compound 5b

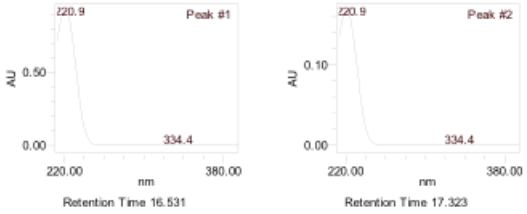
The *e.r.* of the 2-cyclohexenone **5b** was measured using a Daicel Chiralpal® IB column (5 µm, 4.6 x 250 mm) with a U.V. detection at 220 nm and at 25 °C. Flow = 1 mL/min with heptane/isopropanol 95:5.





	Channel Description	RT	Area	% Area
1	PDA 210.0 to 400.0 nm at 1.2 nm	16.449	24996124	44.90
2	PDA 210.0 to 400.0 nm at 1.2 nm	17.106	30670903	55.10

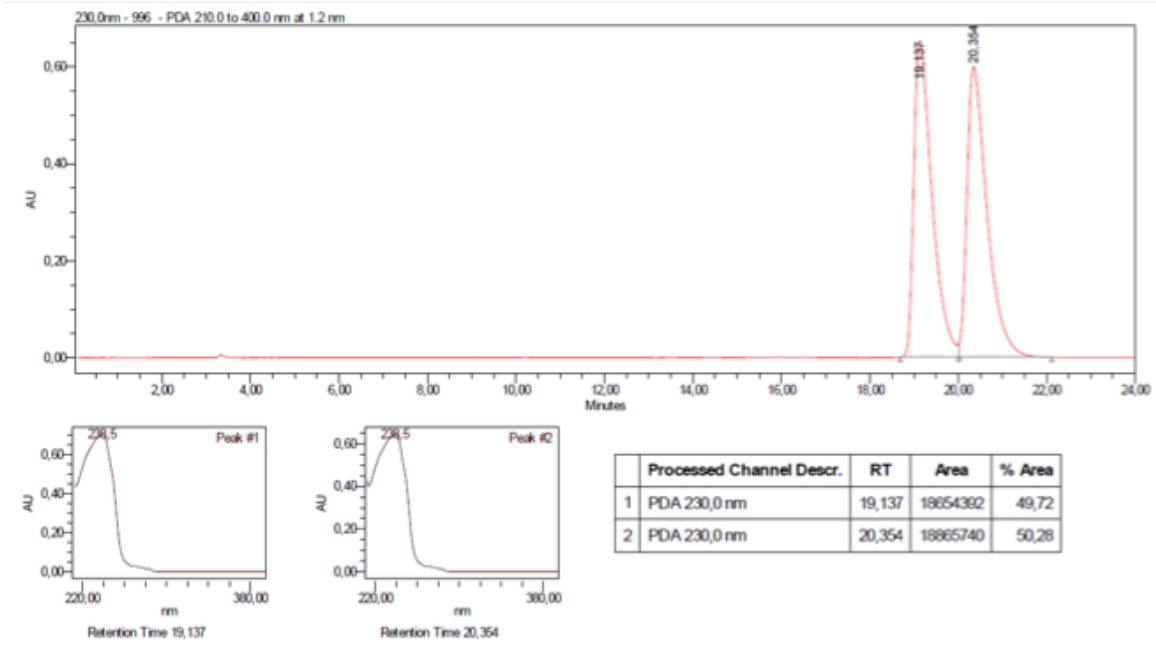




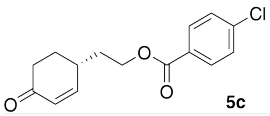
	Channel Description	RT	Area	% Area
1	PDA 210.0 to 400.0 nm at 1.2 nm	16.531	25376236	86.04
2	PDA 210.0 to 400.0 nm at 1.2 nm	17.323	4118971	13.96

Compound 5c

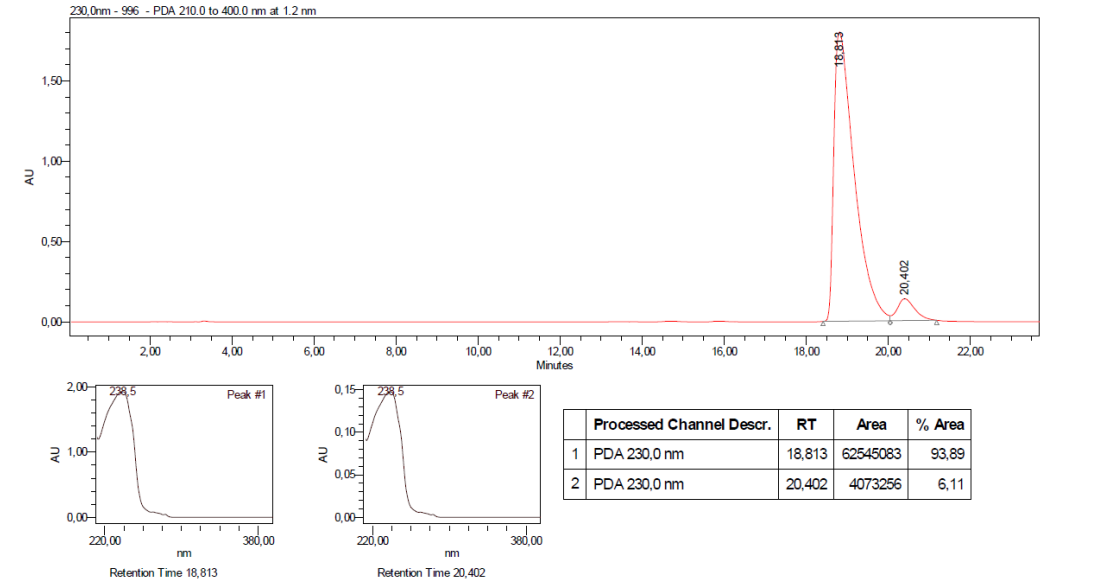
The *e.r.* of the 2-cyclohexenone **5c** was measured using a Daicel Chiralpal® IB column (5 µm, 4.6 x 250 mm) with a U.V. detection at 230 nm and at 25 °C. Flow = 1 mL/min with heptane/isopropanol 95:5.
racemic **5c**



	Processed Channel Descr.	RT	Area	% Area
1	PDA 230,0 nm	19,137	18654392	49,72
2	PDA 230,0 nm	20,354	18865740	50,28



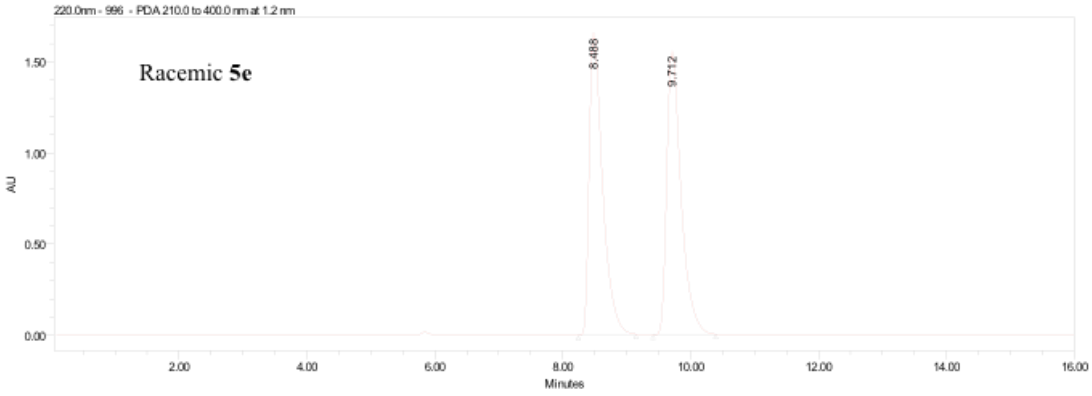
5c

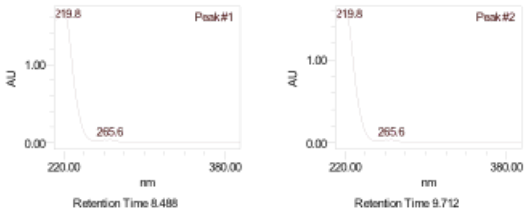


	Processed Channel Descr.	RT	Area	% Area
1	PDA 230,0 nm	18,813	62545083	93,89
2	PDA 230,0 nm	20,402	4073256	6,11

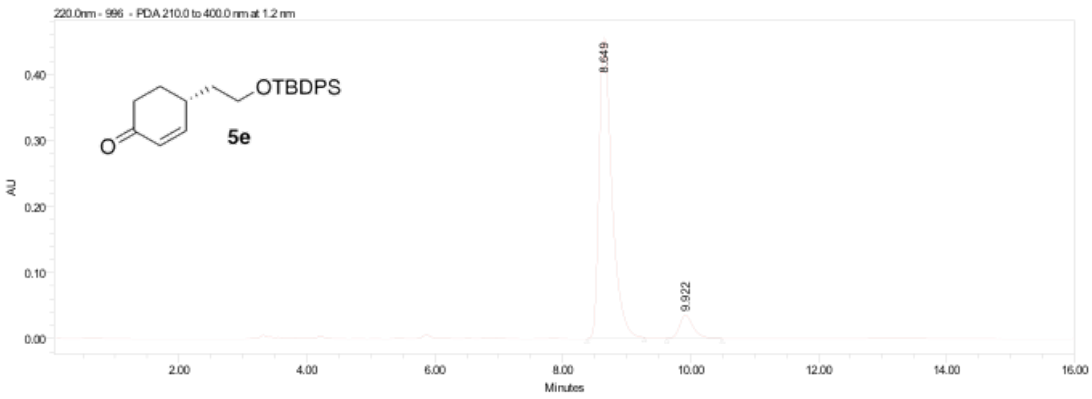
Compound 5e

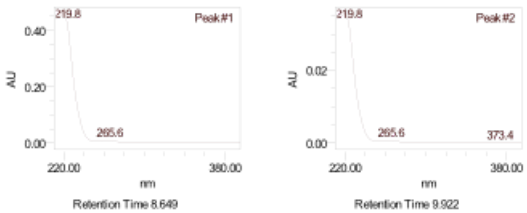
The *e.r.* of the 2-cyclohexenone **5e** was measured using a Daicel Chiralpal® IB column (5 µm, 4.6 x 250 mm) with a U.V. detection at 220 nm and at 25 °C. Flow = 1 mL/min with heptane/isopropanol 98:2.





	Channel Description	RT	Area	% Area
1	PDA 210.0 to 400.0 nm at 1.2 nm	8.488	24055307	49.87
2	PDA 210.0 to 400.0 nm at 1.2 nm	9.712	24183209	50.13

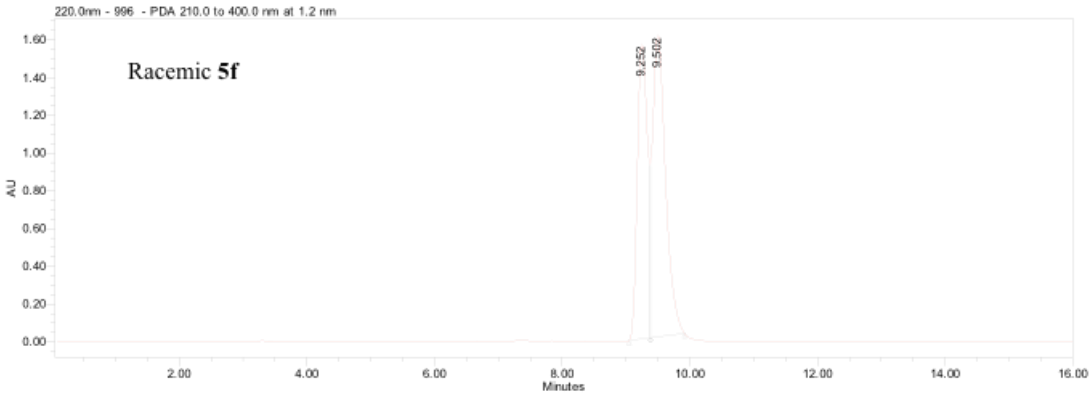


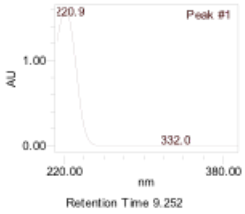


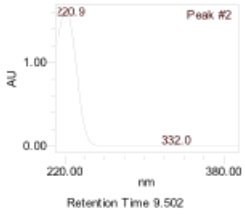
	Channel Description	RT	Area	% Area
1	PDA 210.0 to 400.0 nm at 1.2 nm	8.649	6332001	92.30
2	PDA 210.0 to 400.0 nm at 1.2 nm	9.922	527949	7.70

Compound 5f

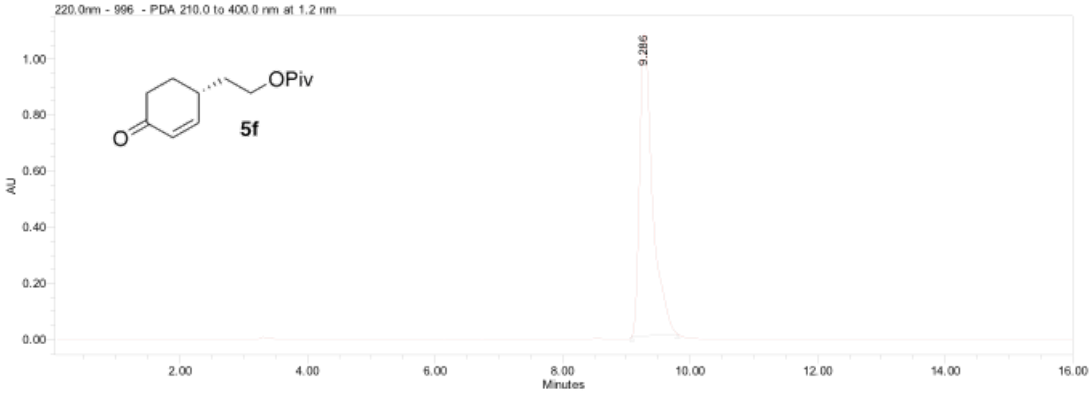
The *e.r.* of the 2-cyclohexenone **5f** was measured using a Daicel Chiralpal® IB column (5 µm, 4.6 x 250 mm) with a U.V. detection at 220 nm and at 25 °C. Flow = 1 mL/min with heptane/isopropanol 95:5.

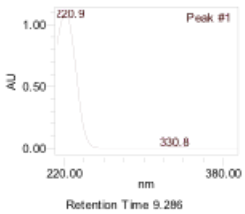






	Channel Description	RT	Area	% Area
1	PDA 210.0 to 400.0 nm at 1.2 nm	9.252	17544265	43.63
2	PDA 210.0 to 400.0 nm at 1.2 nm	9.502	22667644	56.37

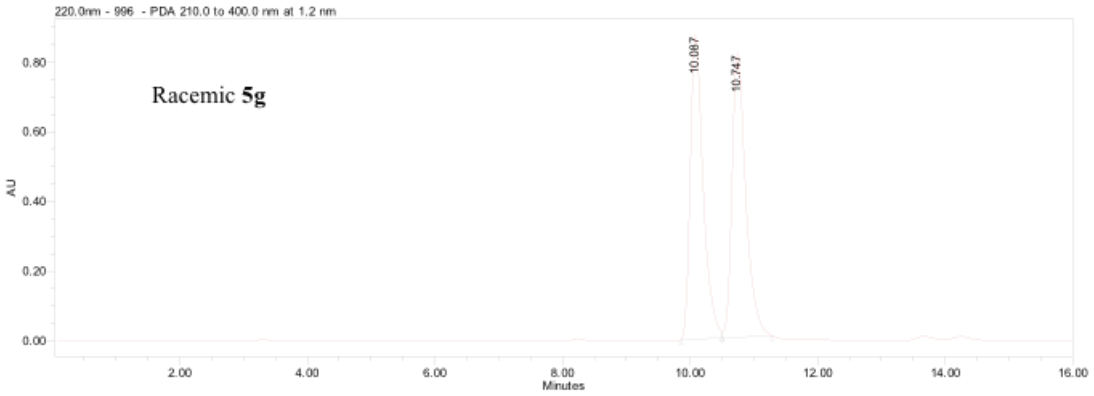


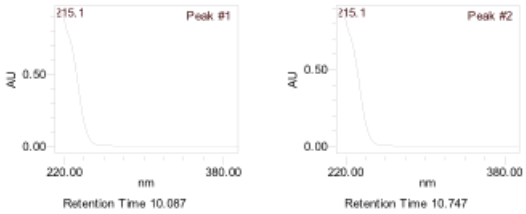


	Channel Description	RT	Area	% Area
1	PDA 210.0 to 400.0 nm at 1.2 nm	9.286	15124984	100.00

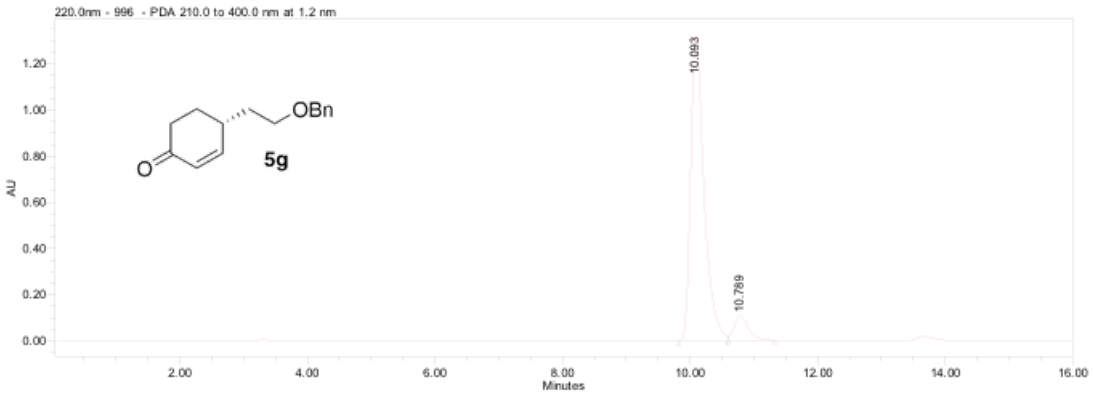
Compound 5g

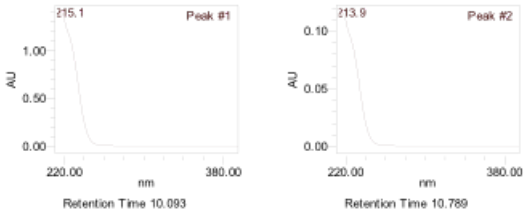
The *e.r.* of the 2-cyclohexenone **5g** was measured using a Daicel Chiralpal® IB column (5 µm, 4.6 x 250 mm) with a U.V. detection at 220 nm and at 25 °C. Flow = 1 mL/min with heptane/isopropanol 95:5.





	Channel Description	RT	Area	% Area
1	PDA 210.0 to 400.0 nm at 1.2 nm	10.087	12245647	49.99
2	PDA 210.0 to 400.0 nm at 1.2 nm	10.747	12252204	50.01

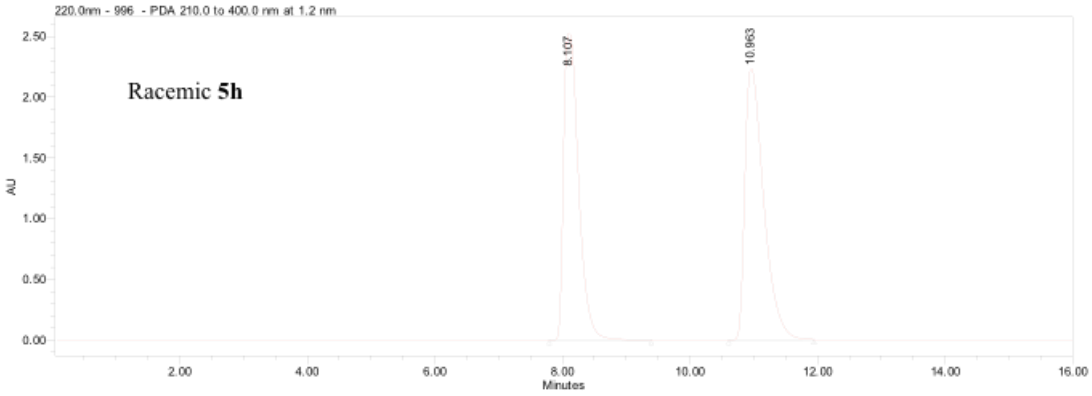


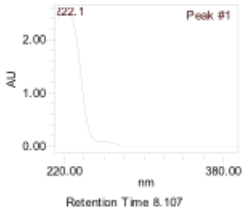


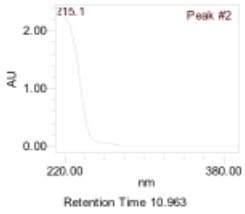
	Channel Description	RT	Area	% Area
1	PDA 210.0 to 400.0 nm at 1.2 nm	10.093	19322738	92.02
2	PDA 210.0 to 400.0 nm at 1.2 nm	10.789	1676341	7.98

Compound 5h

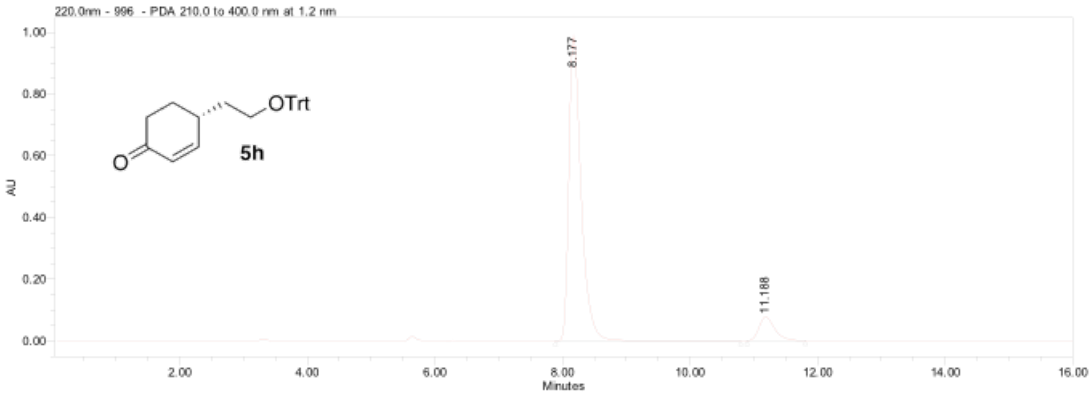
The *e.r.* of the 2-cyclohexenone **5h** was measured using a Daicel Chiralpal® IB column (5 µm, 4.6 x 250 mm) with a U.V. detection at 220 nm and at 25 °C. Flow = 1 mL/min with heptane/isopropanol 95:5

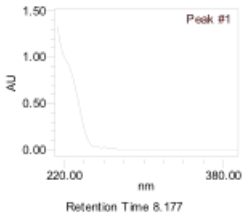


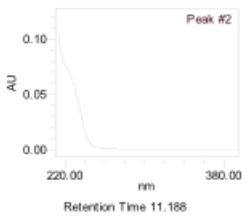




	Channel Description	RT	Area	% Area
1	PDA 210.0 to 400.0 nm at 1.2 nm	8.107	41421648	47.49
2	PDA 210.0 to 400.0 nm at 1.2 nm	10.963	45798326	52.51







	Channel Description	RT	Area	% Area
1	PDA 210.0 to 400.0 nm at 1.2 nm	8.177	13269487	90.85
2	PDA 210.0 to 400.0 nm at 1.2 nm	11.188	1336828	9.15

IR spectrum of compound **2c**