Exploration of the Mitsunobu Reaction with Tosyl- and Boc-Hydrazones as Nucleophillic Agents

John M. Keith* and Leslie Gomez

Supplementary Information

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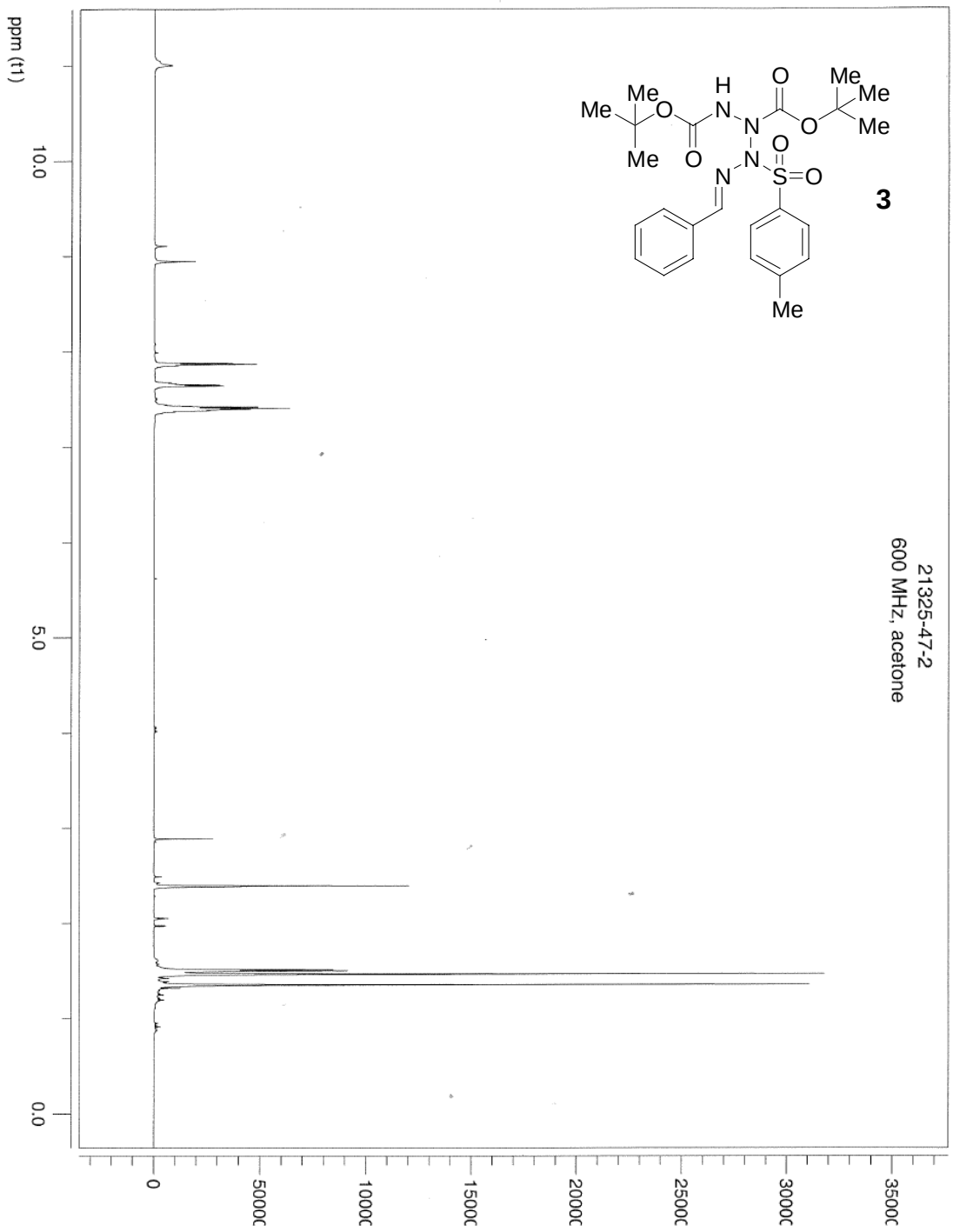
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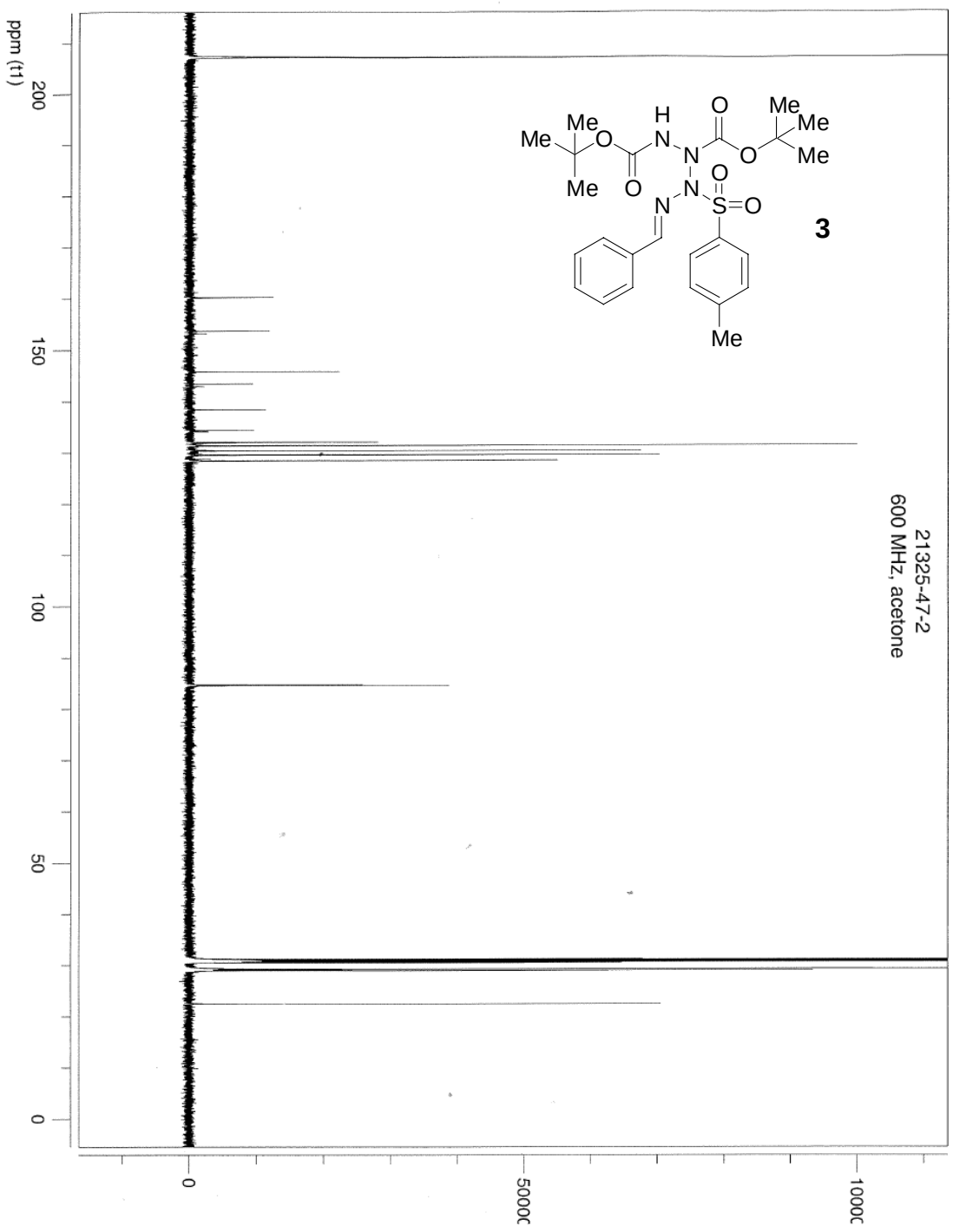
0.0
0
5000
10000
15000
20000
25000
30000
35000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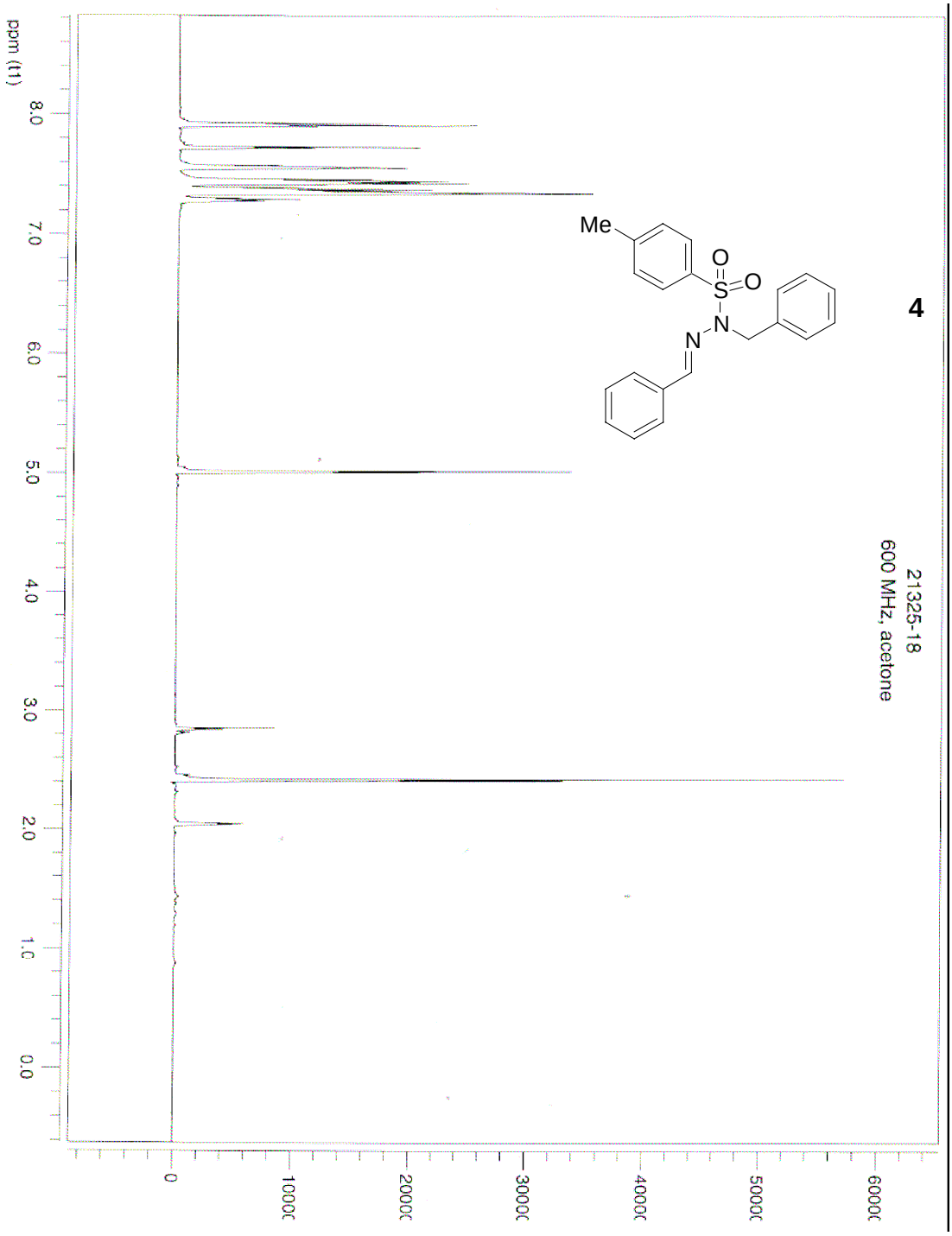
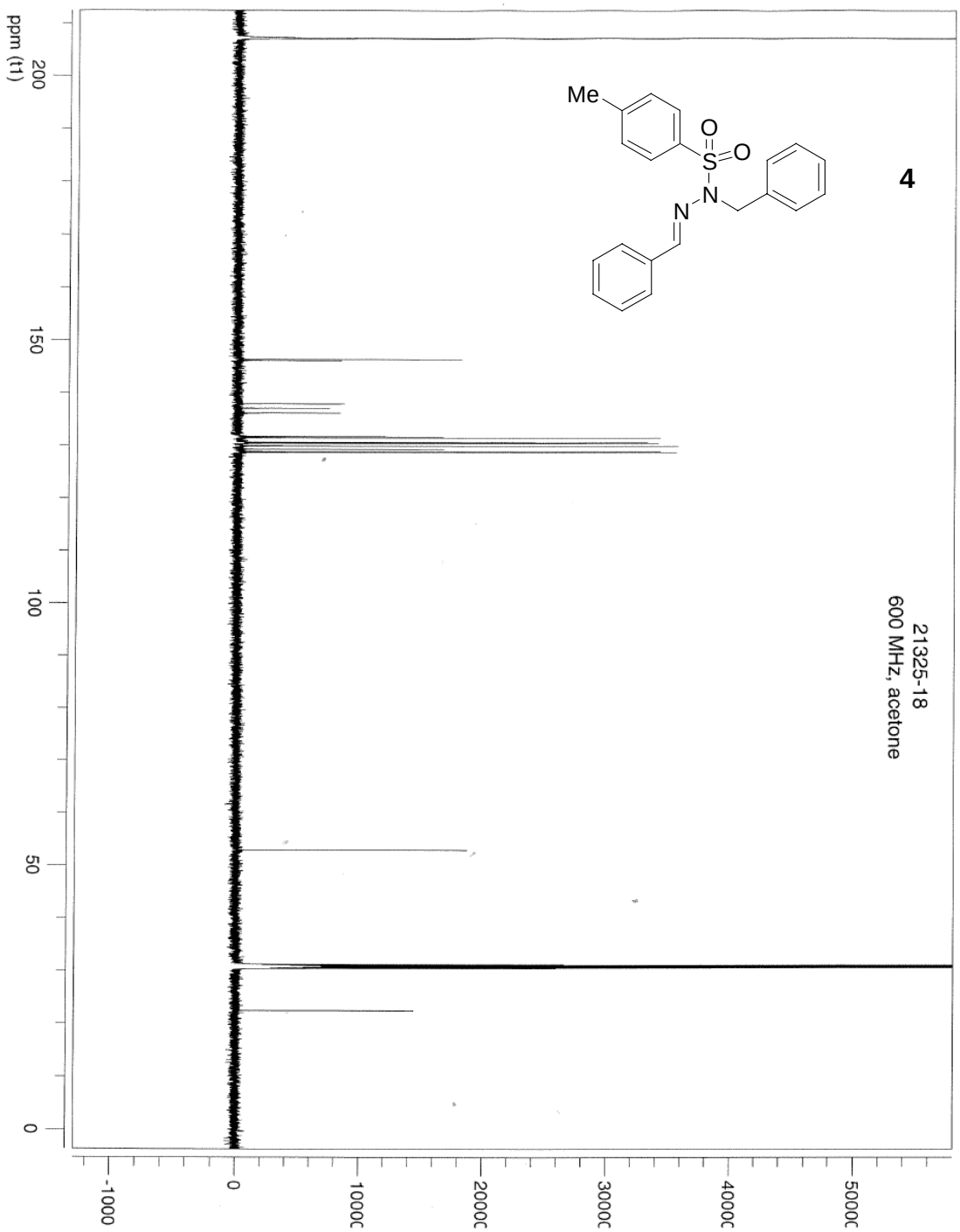
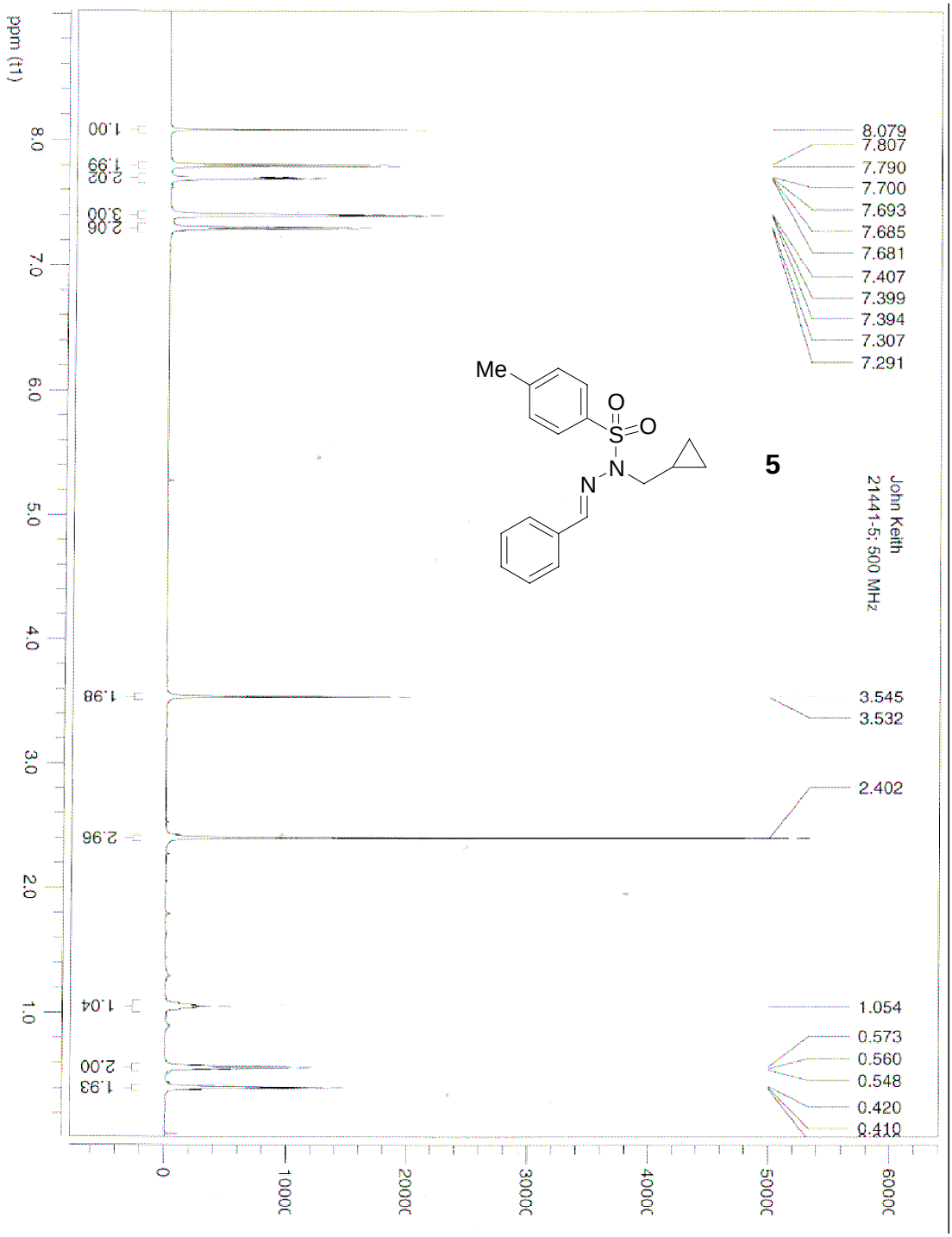
600 MHz, acetone
3
ppm (t1)
200
150
100
50
0
0
50000
100000


4
21325-18
600 MHz, acetone
Me
O
S
O
N
N
ppm (t1)
8.0
7.0
6.0
5.0
4.0
3.0
2.0
1.0
0.0
0
10000
20000
30000
40000
50000
60000


21325-18
600 MHz, acetone
4
Me
O
S
O
N
N
ppm (t1)
200
150
100
50
0
-1000
0
1000
2000
3000
4000
5000


ppm (t1)
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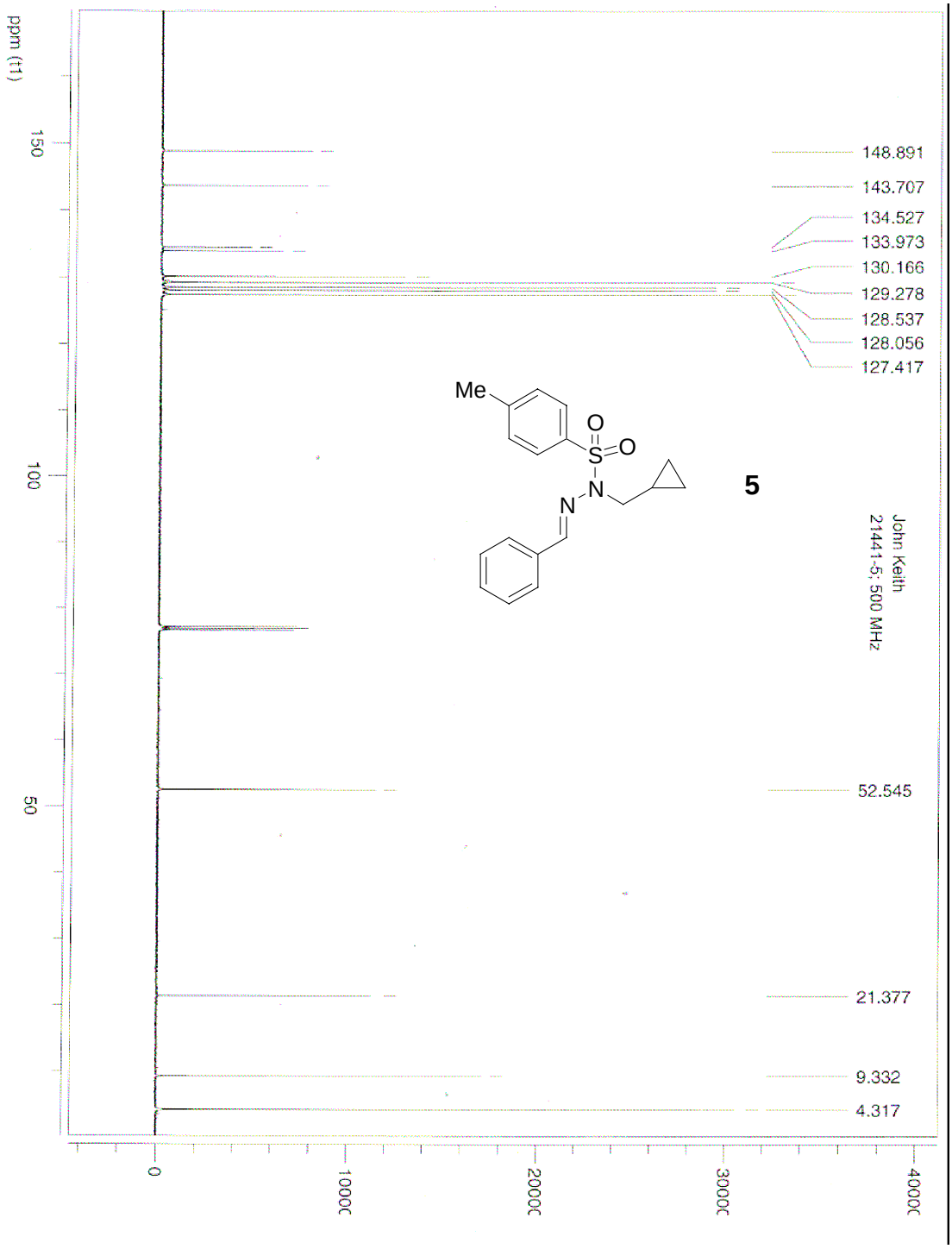
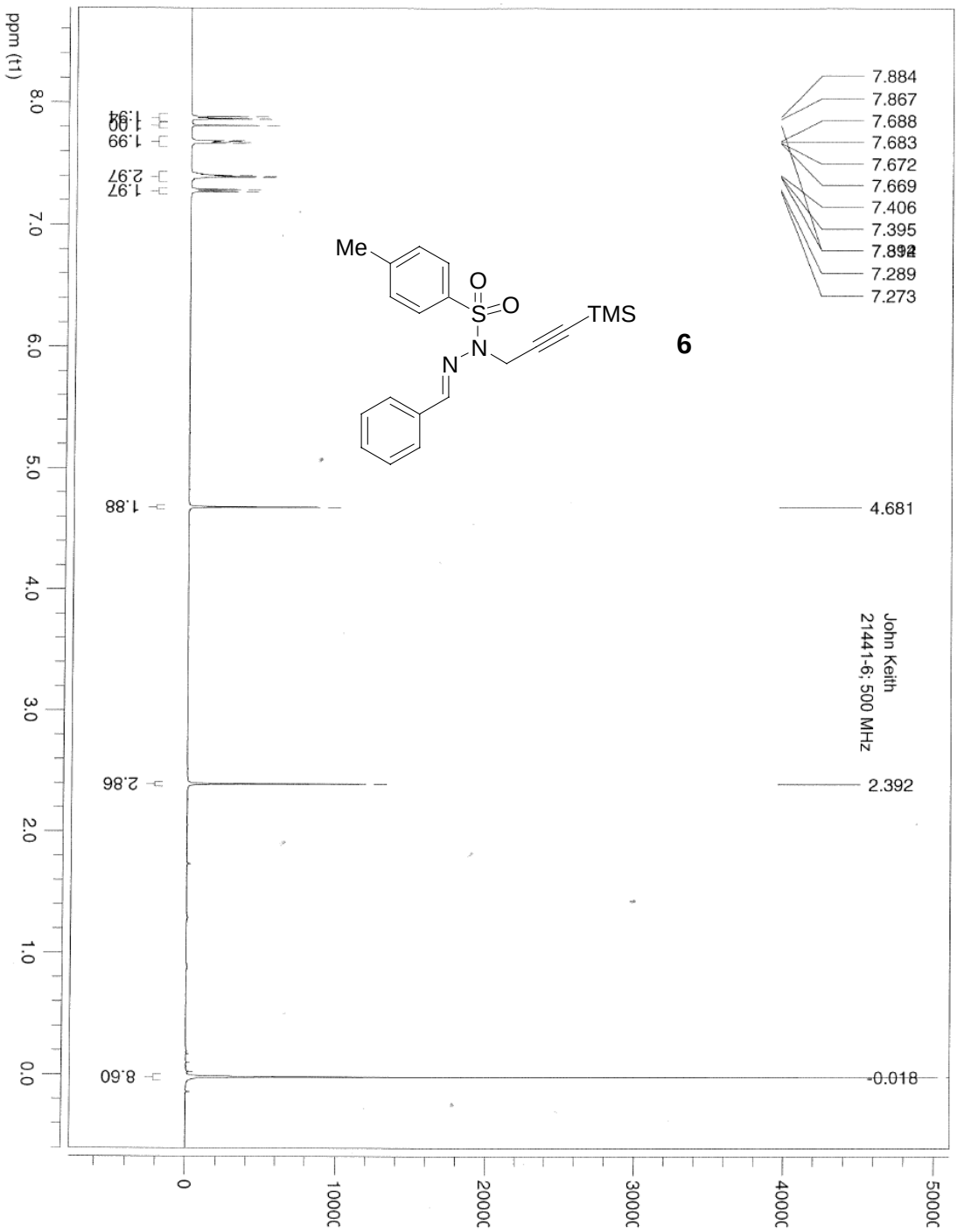
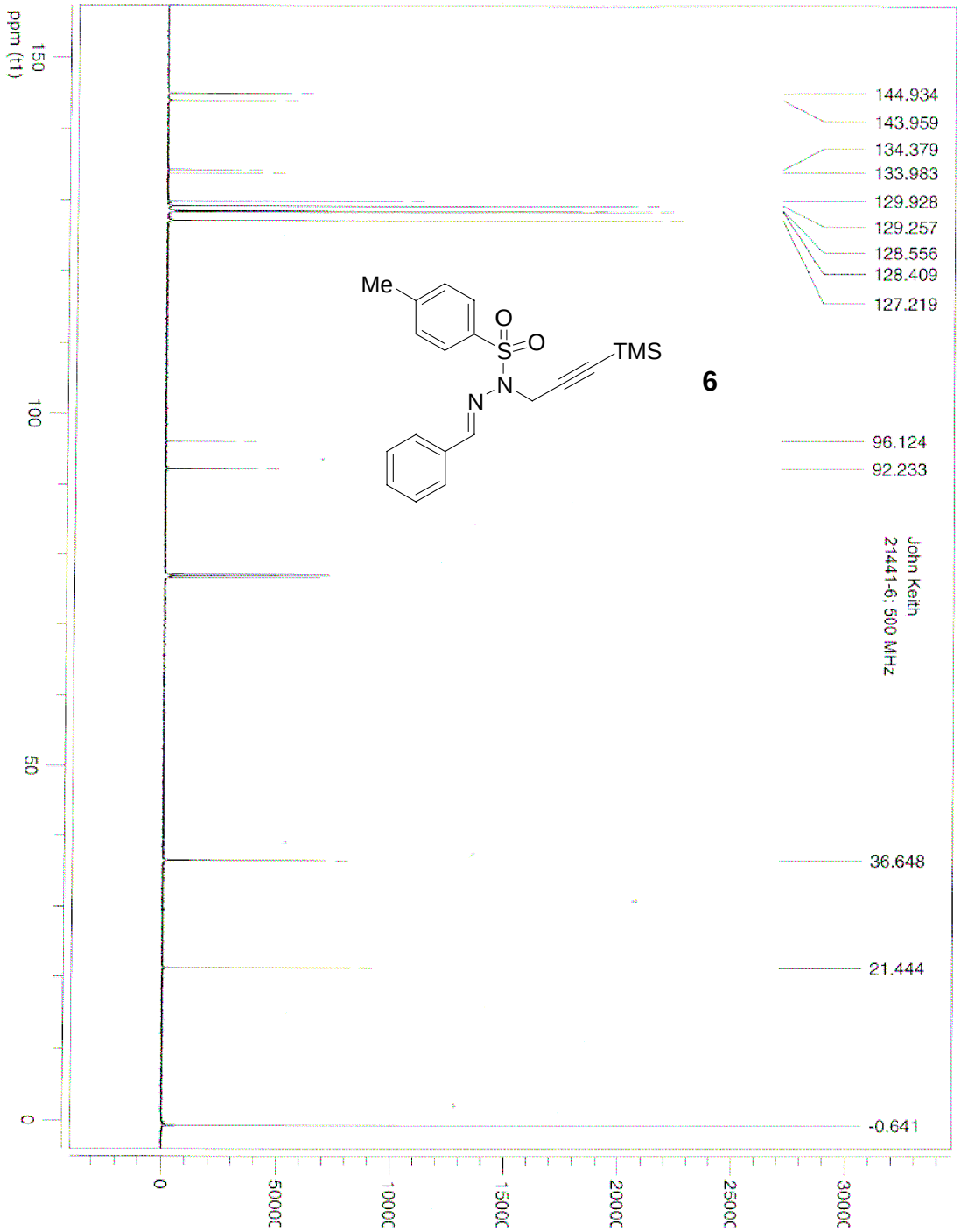
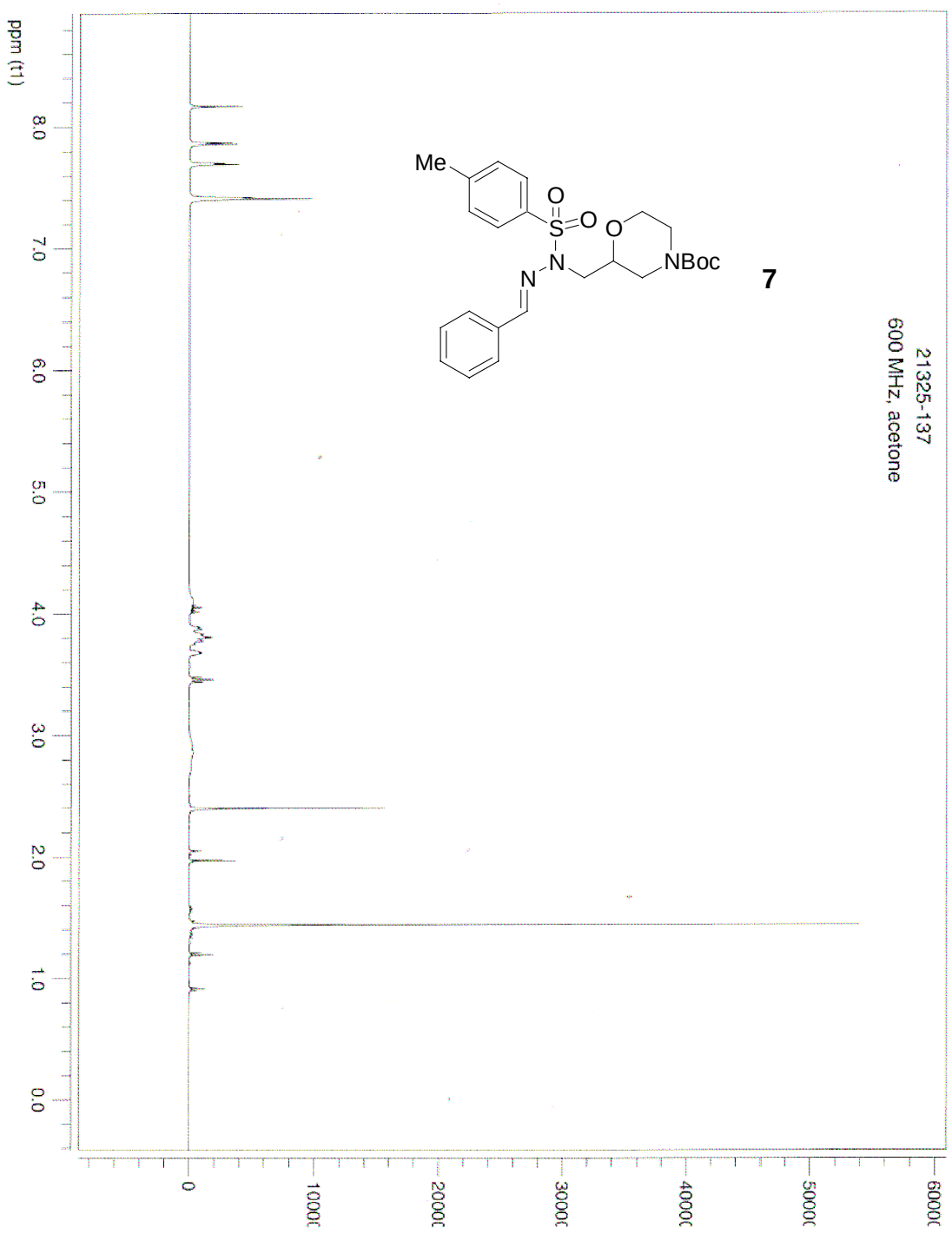
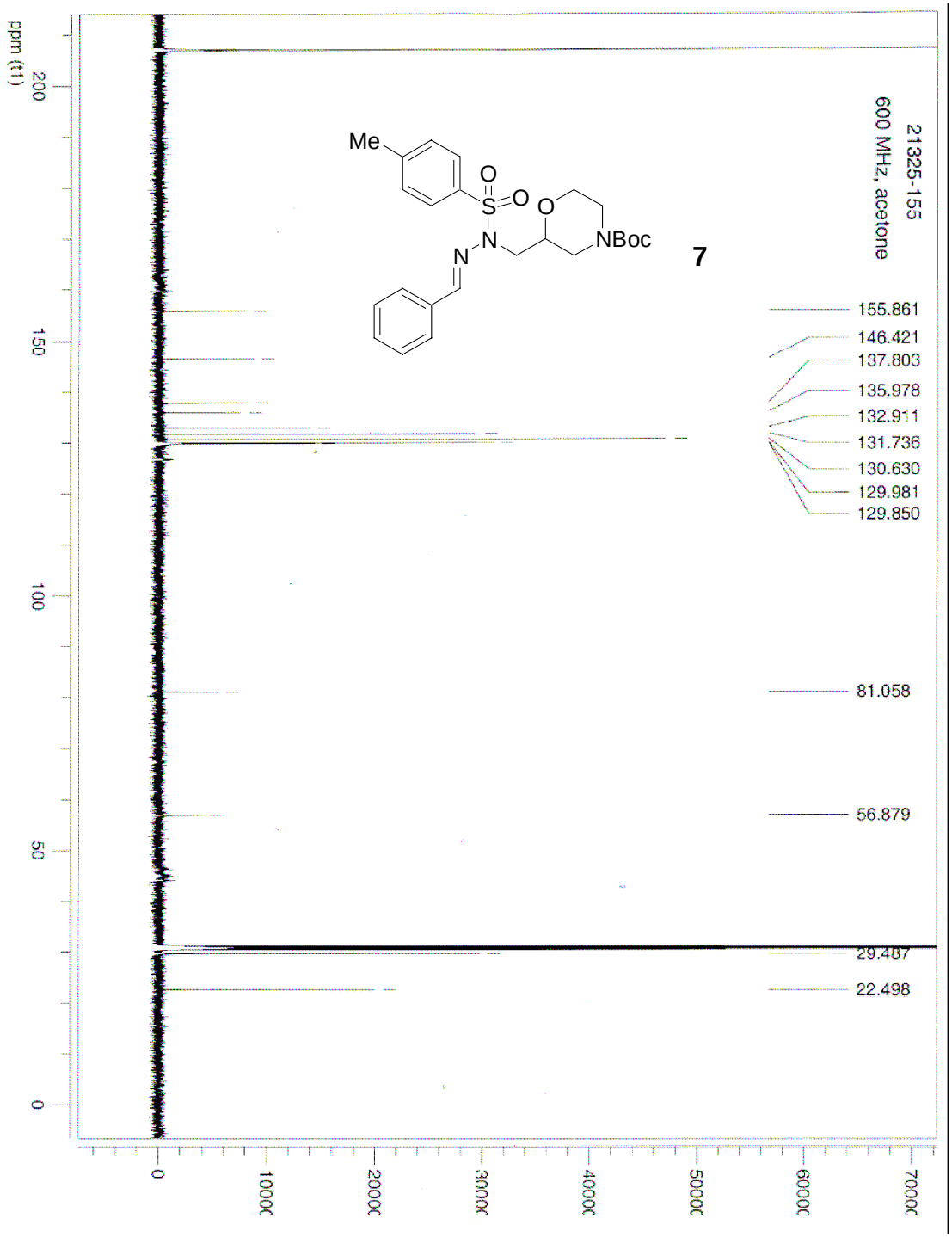
7.0
6.0
5.0
4.0
3.0
2.0
1.0
1.00
1.99
2.02
3.00
2.06
1.98
2.96
1.04
2.00
1.93
8.079
7.807
7.790
7.700
7.693
7.685
7.681
7.407
7.399
7.394
7.307
7.291
3.545
3.532
2.402
1.054
0.573
0.560
0.548
0.420
0.410
Me
O
S
O
N
N
5
John Keith
21441-5; 500 MHz
0
10000
20000
30000
40000
50000
60000

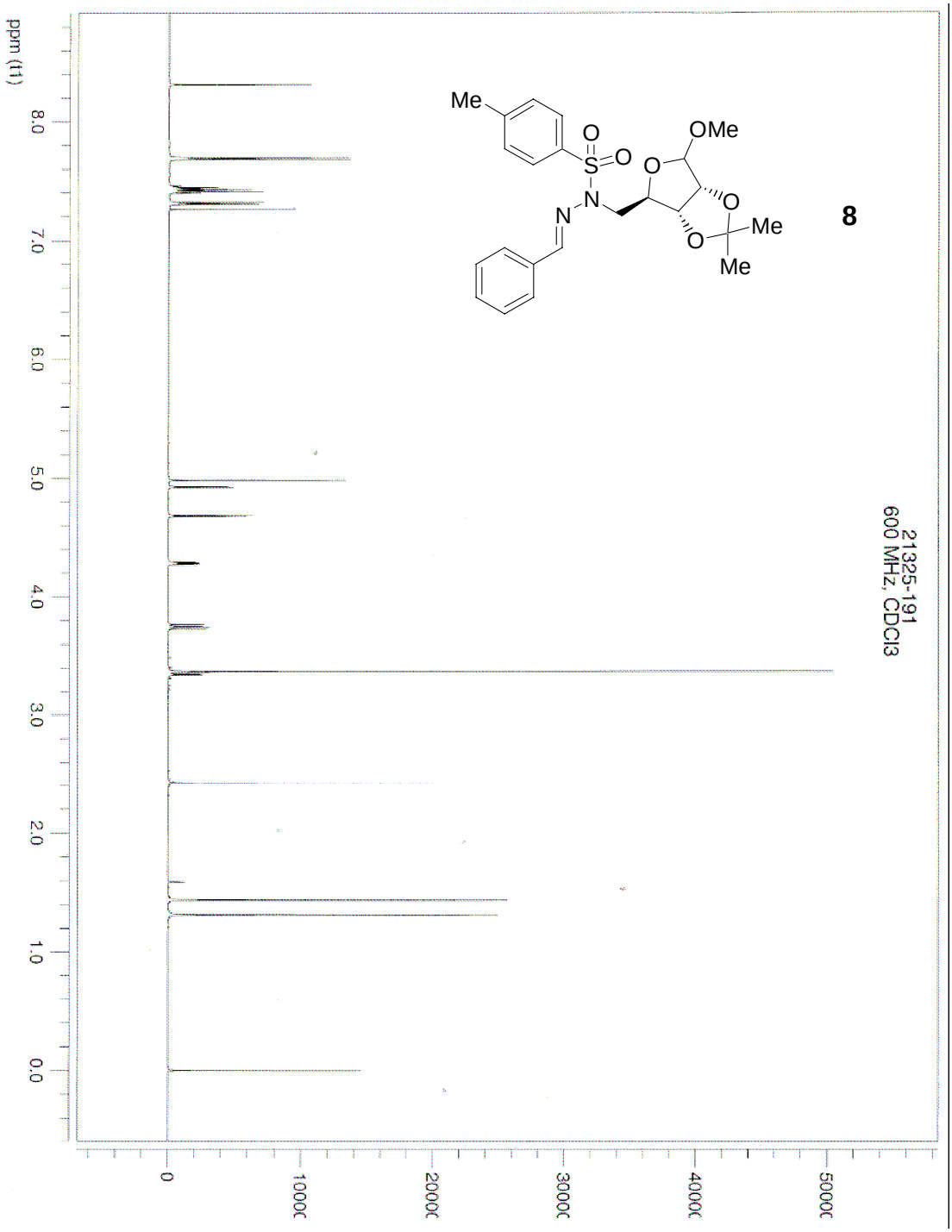

148.891
143.707
134.527
133.973
130.166
129.278
128.537
128.056
127.417
52.545
21.377
9.332
4.317
Me
O
S
O
N
N
5
John Keith
21441-5; 500 MHz
ppm (t1)
150
100
50
0
10000
20000
30000
40000

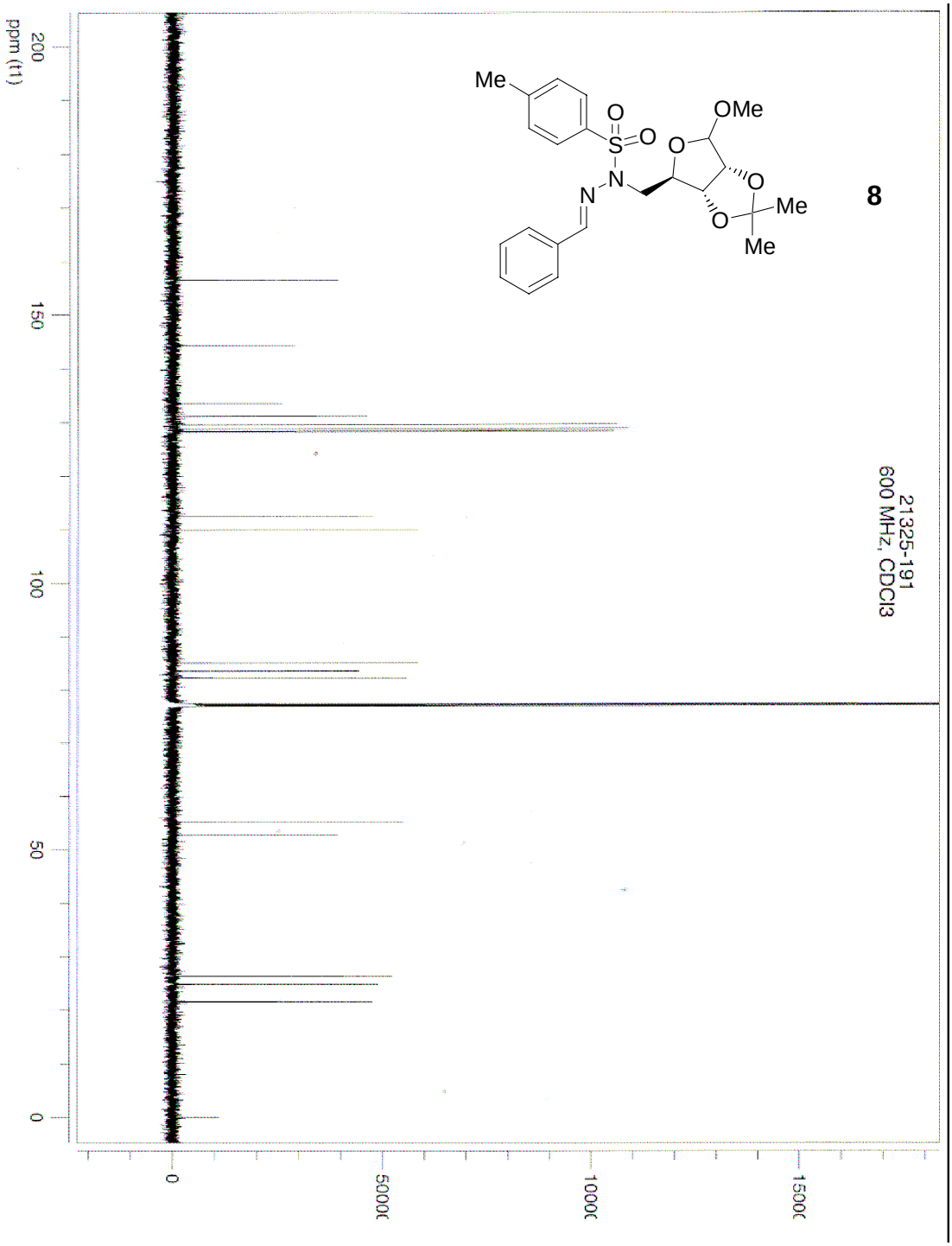

Me
O
S
O
N
N
TMS
6
7.884
7.867
7.688
7.683
7.672
7.669
7.406
7.395
7.289
7.273
4.681
2.392
-0.018
1.88
2.86
8.60
1.99
2.97
1.97
John Keith
21441-6; 500 MHz
ppm (t1)

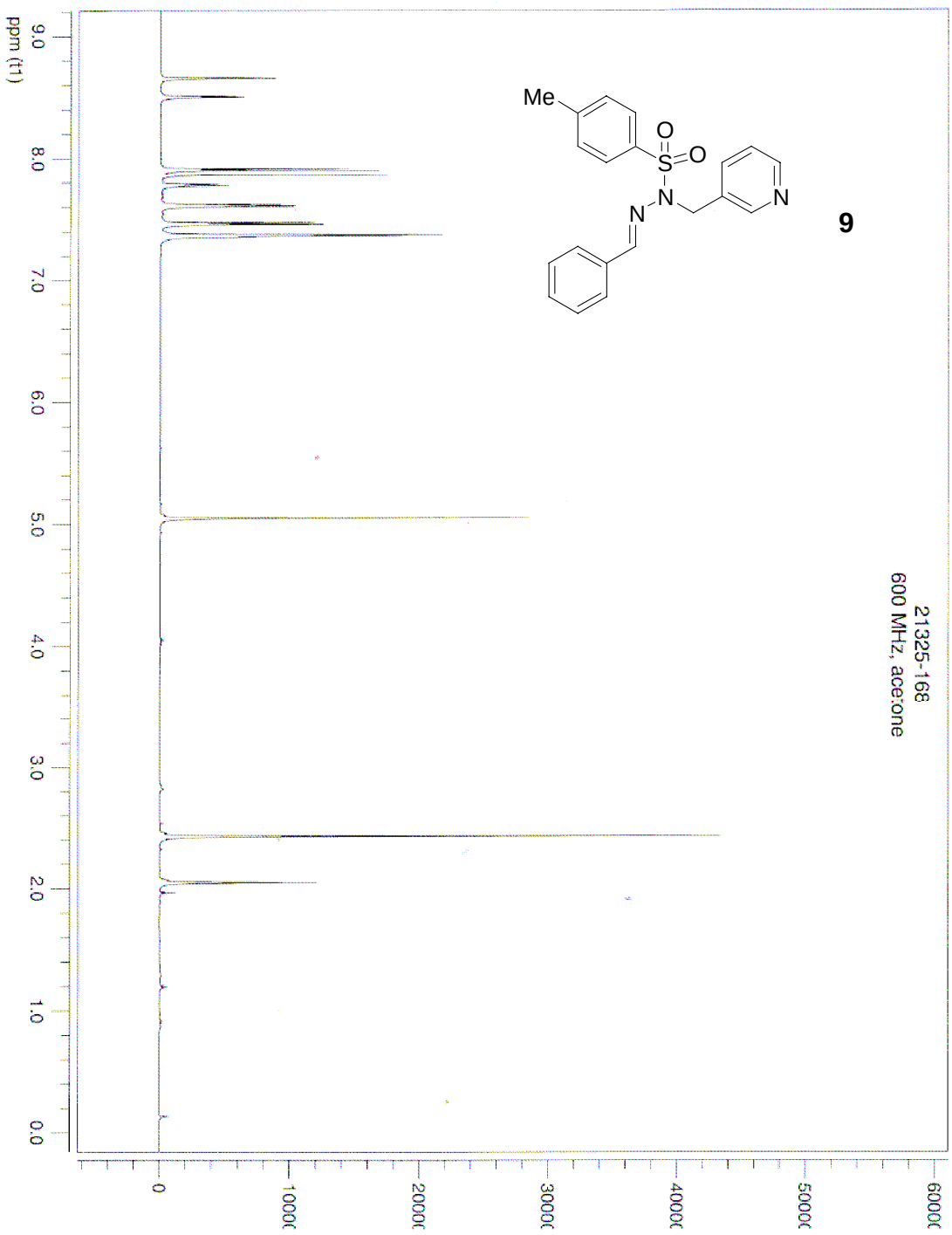

Me
O
S
O
TMS
N
N
6
144.934
143.959
134.379
133.983
129.928
129.257
128.556
128.409
127.219
96.124
92.233
36.648
21.444
-0.641
John Keith
21441-6: 500 MHz
ppm (t1)
150
100
50
0
0
5000
10000
15000
20000
25000
30000

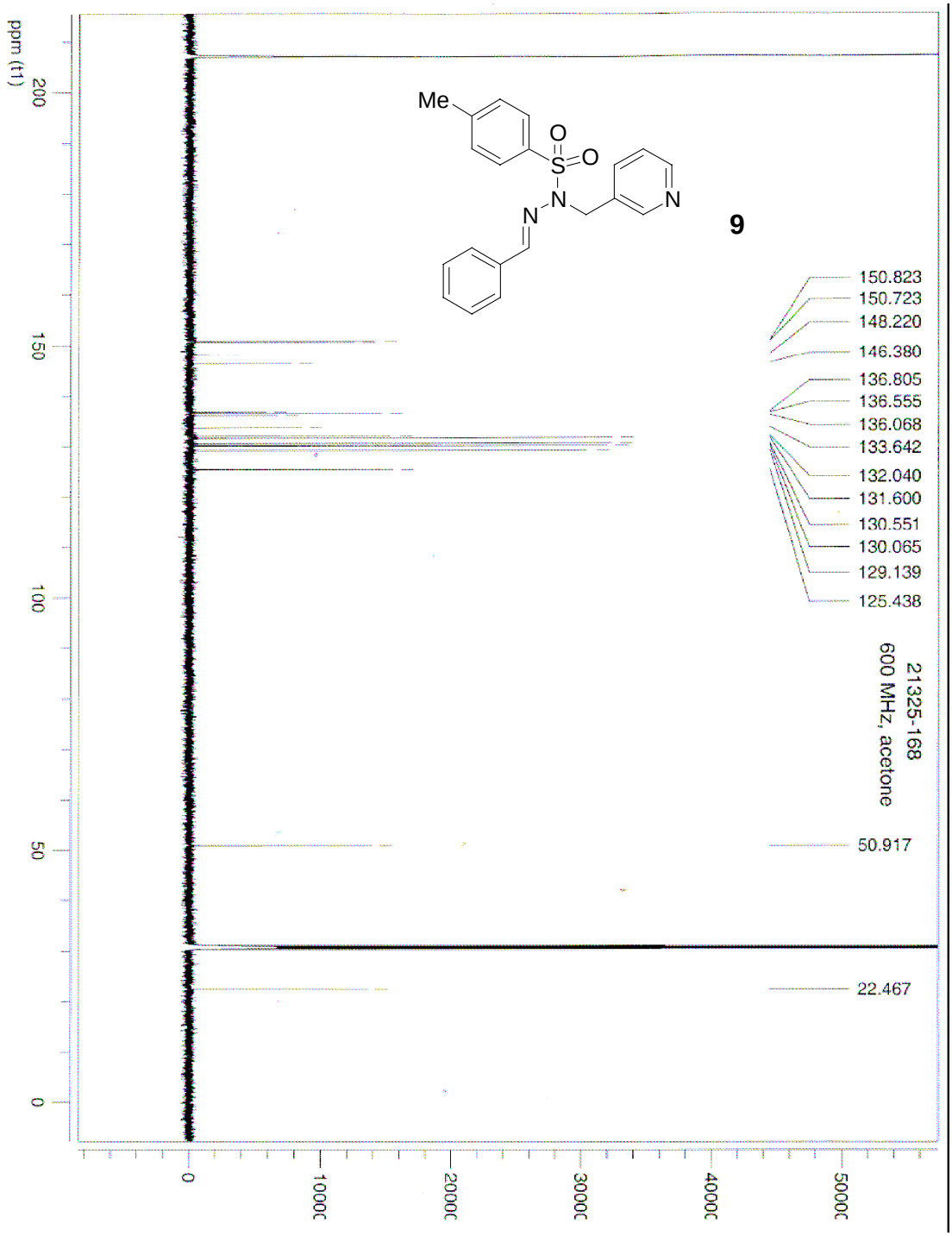

21325-137
600 MHz, acetone
Me
O
S
O
O
N
N
NBoc
7
ppm (t1)
8.0
7.0
6.0
5.0
4.0
3.0
2.0
1.0
0.0
0
10000
20000
30000
40000
50000
60000

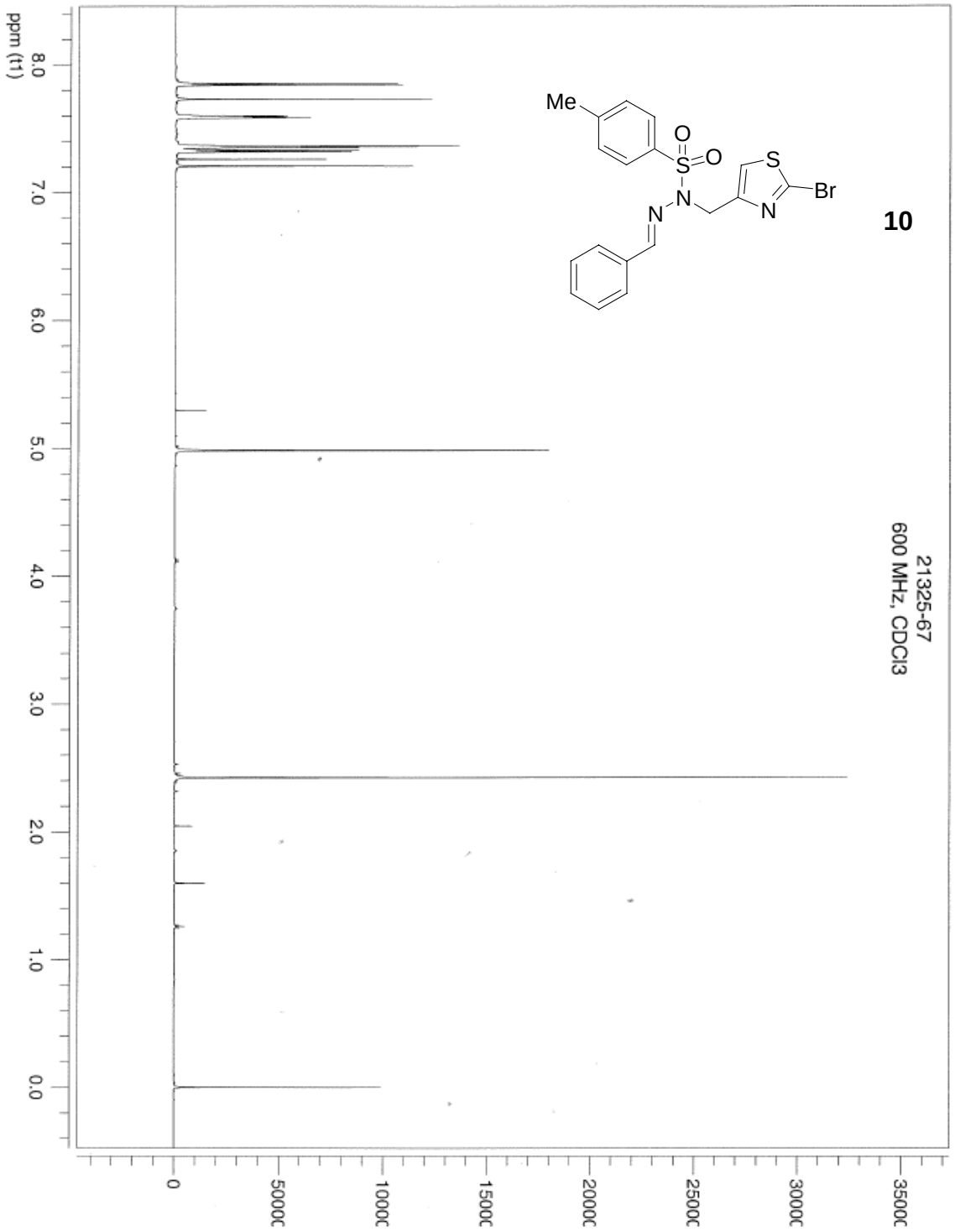

21325-155
600 MHz, acetone
Me
O
S
O
O
N
N
NBoc
7
155.861
146.421
137.803
135.978
132.911
131.736
130.630
129.981
129.850
81.058
56.879
29.487
22.498
ppm (t1)
200
150
100
50
0
0
10000
20000
30000
40000
50000
60000
70000

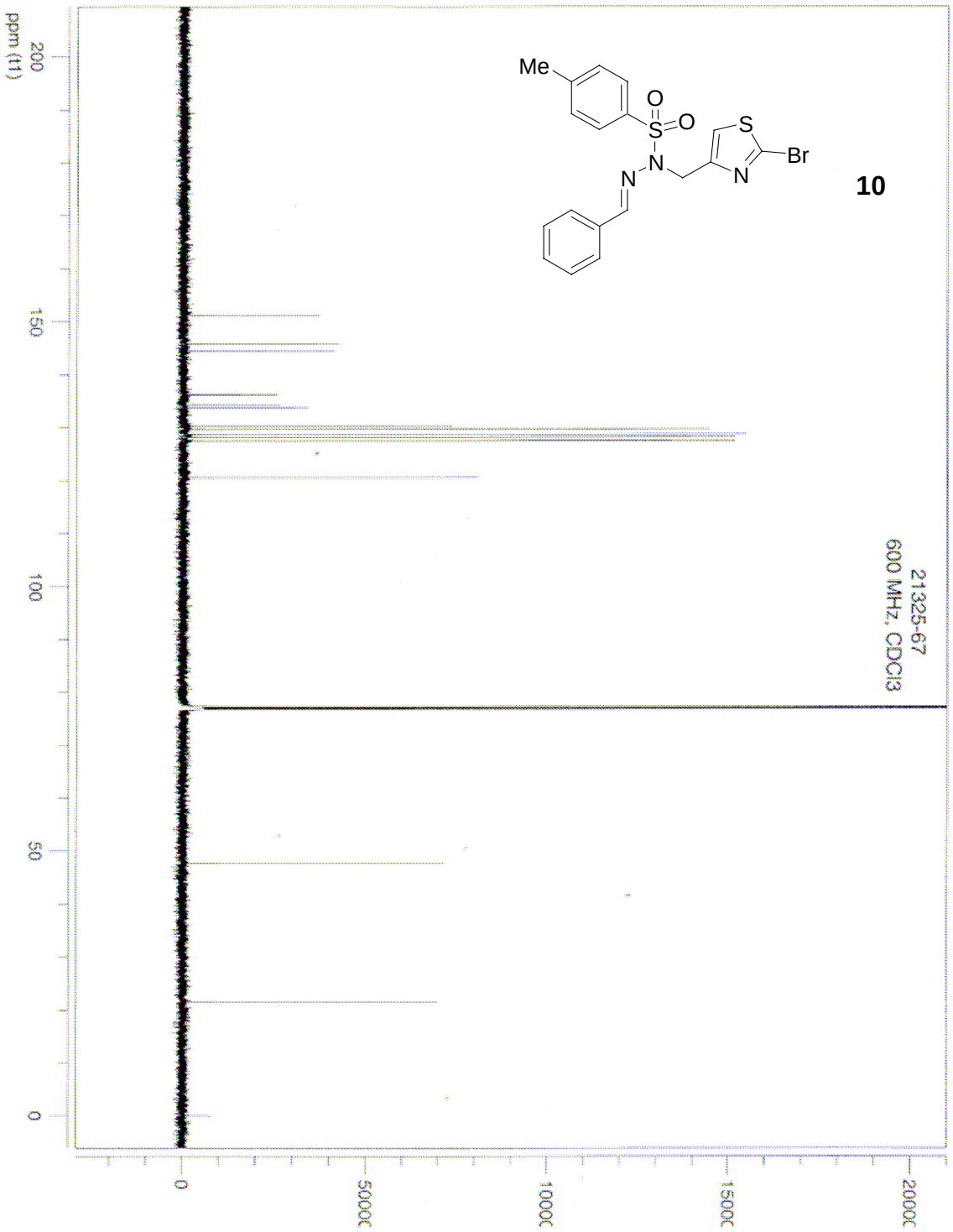

21325-191
600 MHz, CDCl3
8
Me
OMe
Me
Me
ppm (t1)
8.0
7.0
6.0
5.0
4.0
3.0
2.0
1.0
0.0
0
10000
20000
30000
40000
50000

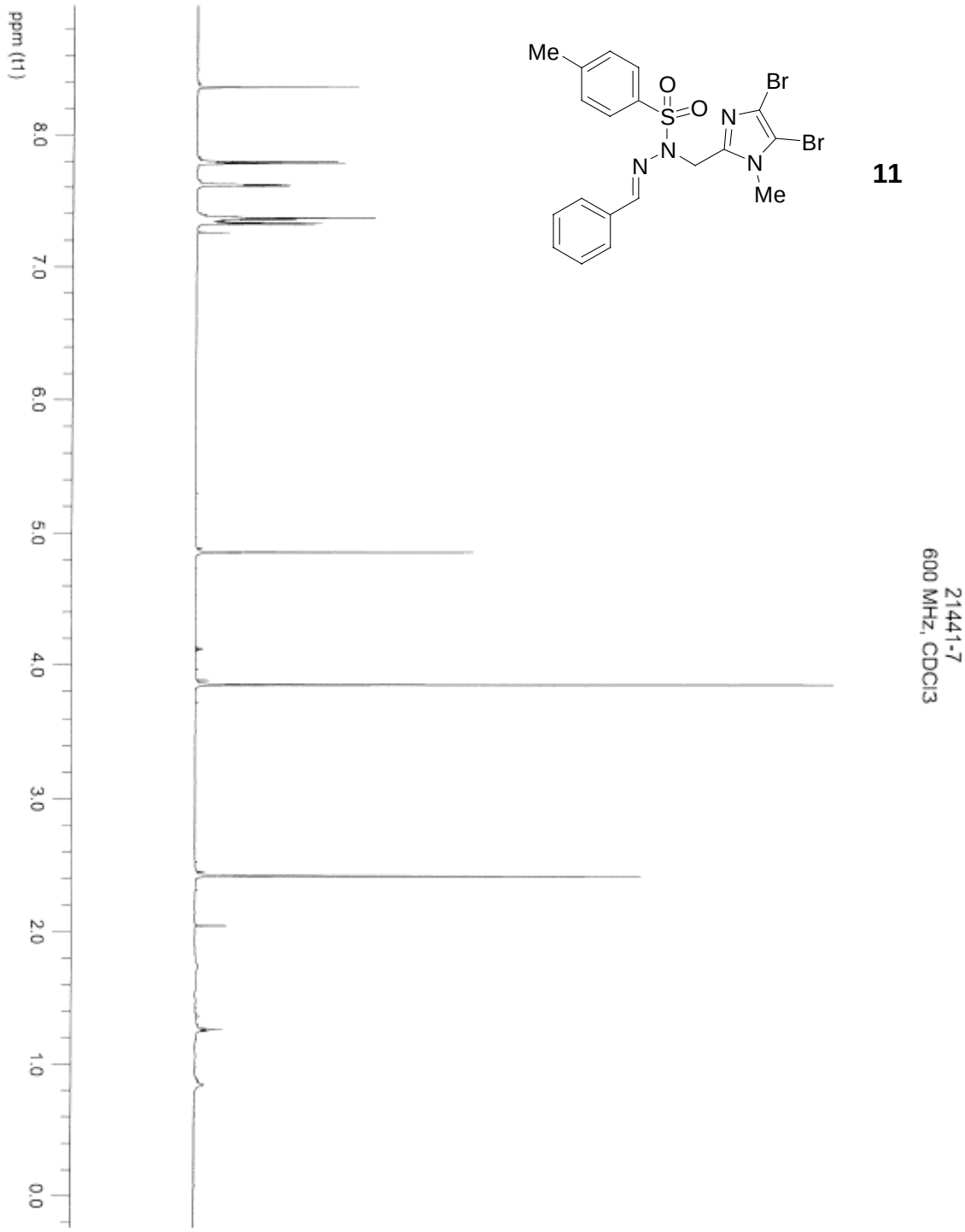

Me
O
S
O
N
N
OMe
O
O
O
Me
Me
8
21325-191
600 MHz, CDCl3
ppm (t1)
200
150
100
50
0
0
50000
100000
150000


21325-168
600 MHz, acetone
9
Me
O
S
O
N
N
N
ppm (t1)
9.0
8.0
7.0
6.0
5.0
4.0
3.0
2.0
1.0
0.0
0
10000
20000
30000
40000
50000
60000


Me
O
S
O
N
N
N
9
150.823
150.723
148.220
146.380
136.805
136.555
136.068
133.642
132.040
131.600
130.551
130.065
129.139
125.438
21325-168
600 MHz, acetone
50.917
22.467
ppm (t1)
200
150
100
50
0
0
10000
20000
30000
40000
50000


21325-67
600 MHz, CDCl3
10
Me
O
O
S
N
N
S
Br
N
ppm (t1)
8.0
7.0
6.0
5.0
4.0
3.0
2.0
1.0
0.0
0
5000
10000
15000
20000
25000
30000
35000


Me
O
S
O
N
N
S
Br
N
10
21325-67
600 MHz, CDCl3
200
150
100
50
0
ppm (t1)
0
50000
100000
150000
200000


21441-7
600 MHz, CDCl3
11
Me
O
S
O
N
N
N
Br
Br
N
Me
ppm (t1)
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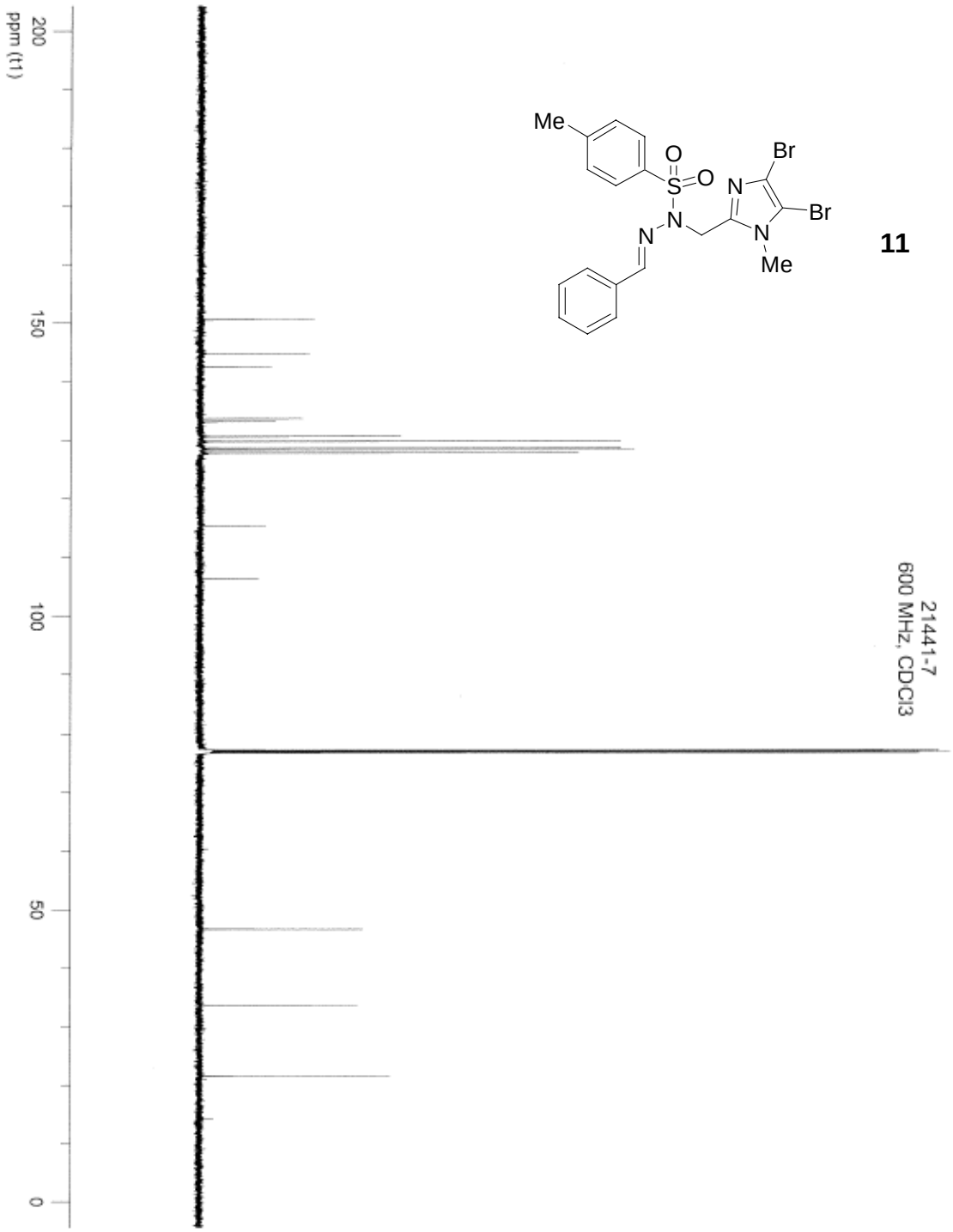
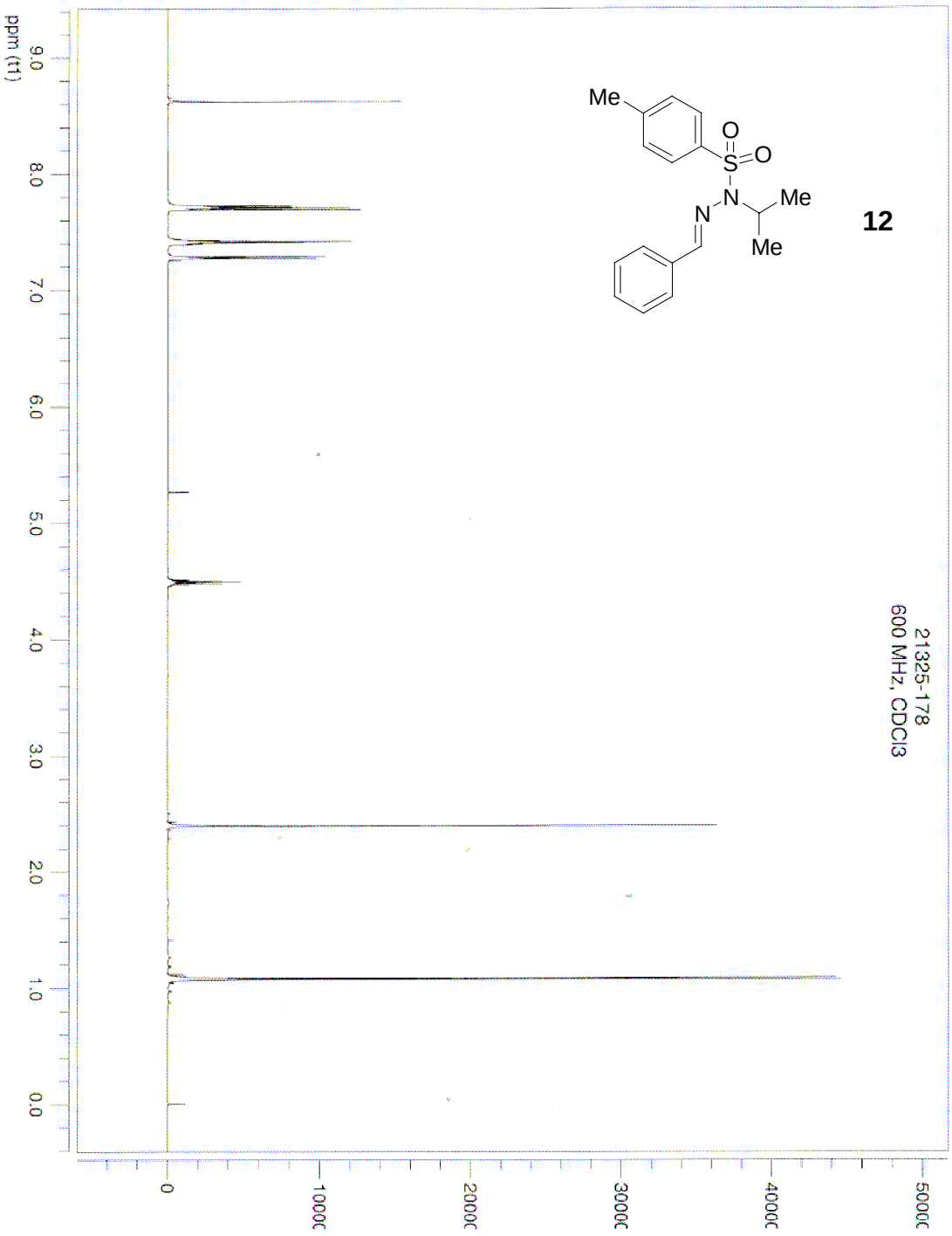
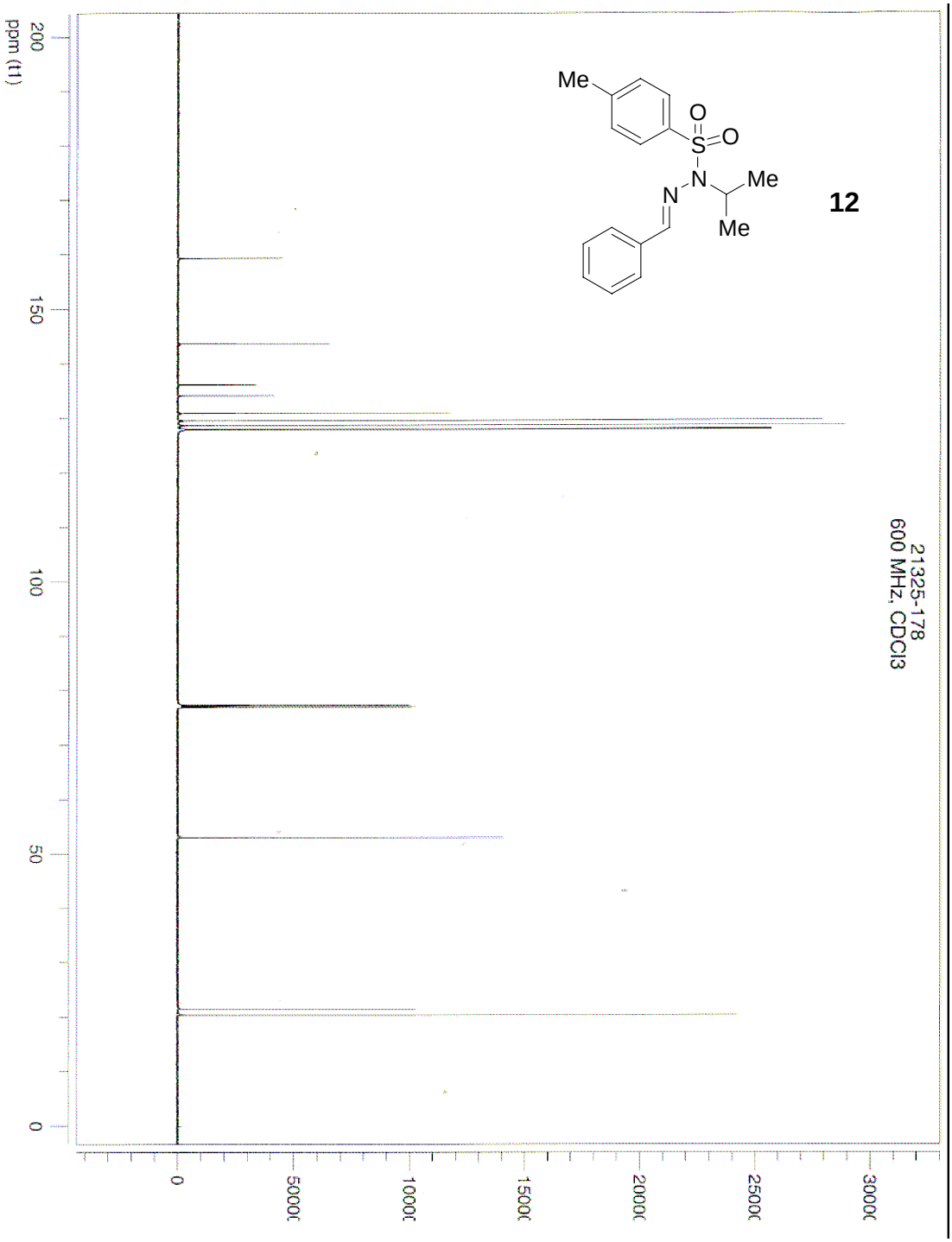
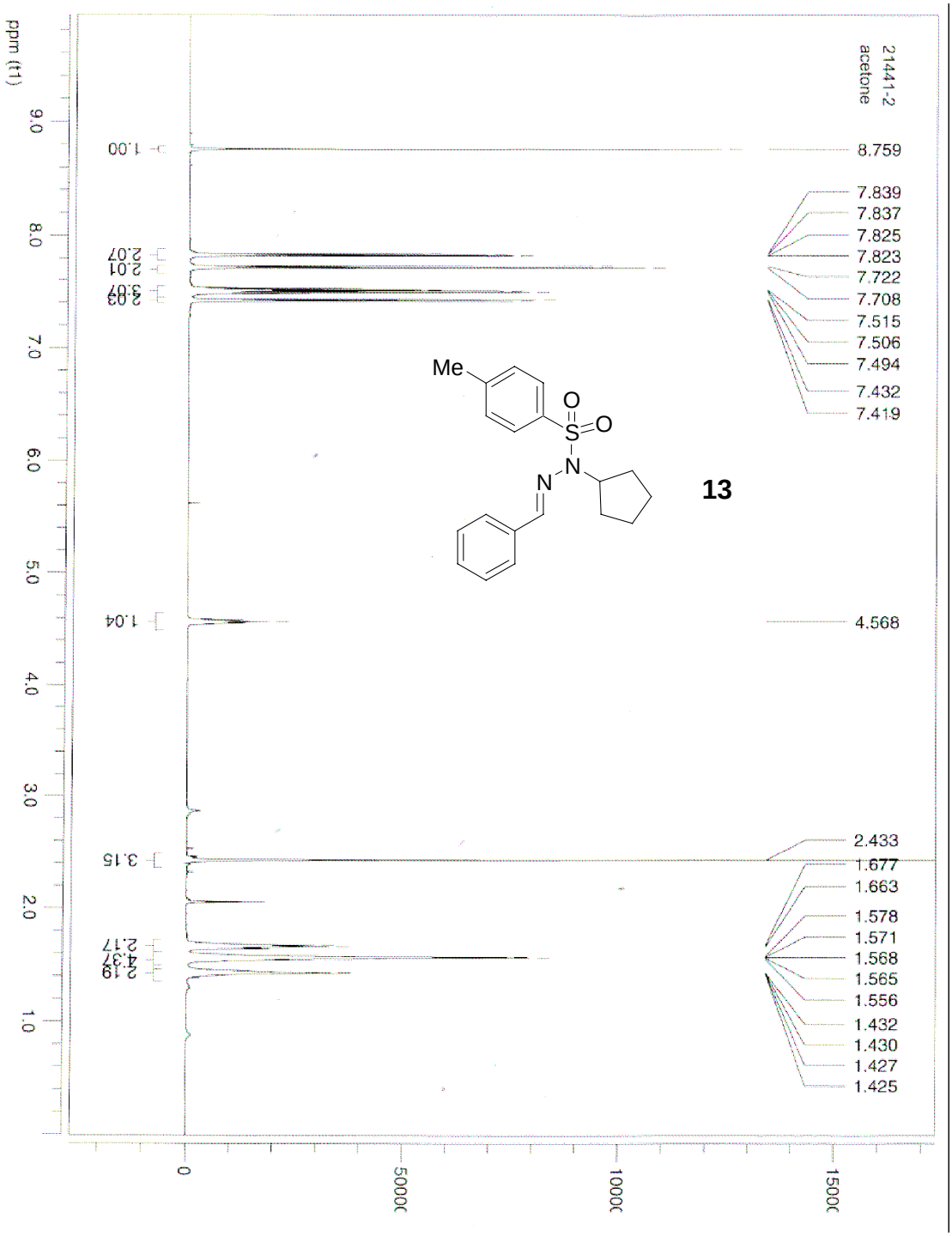
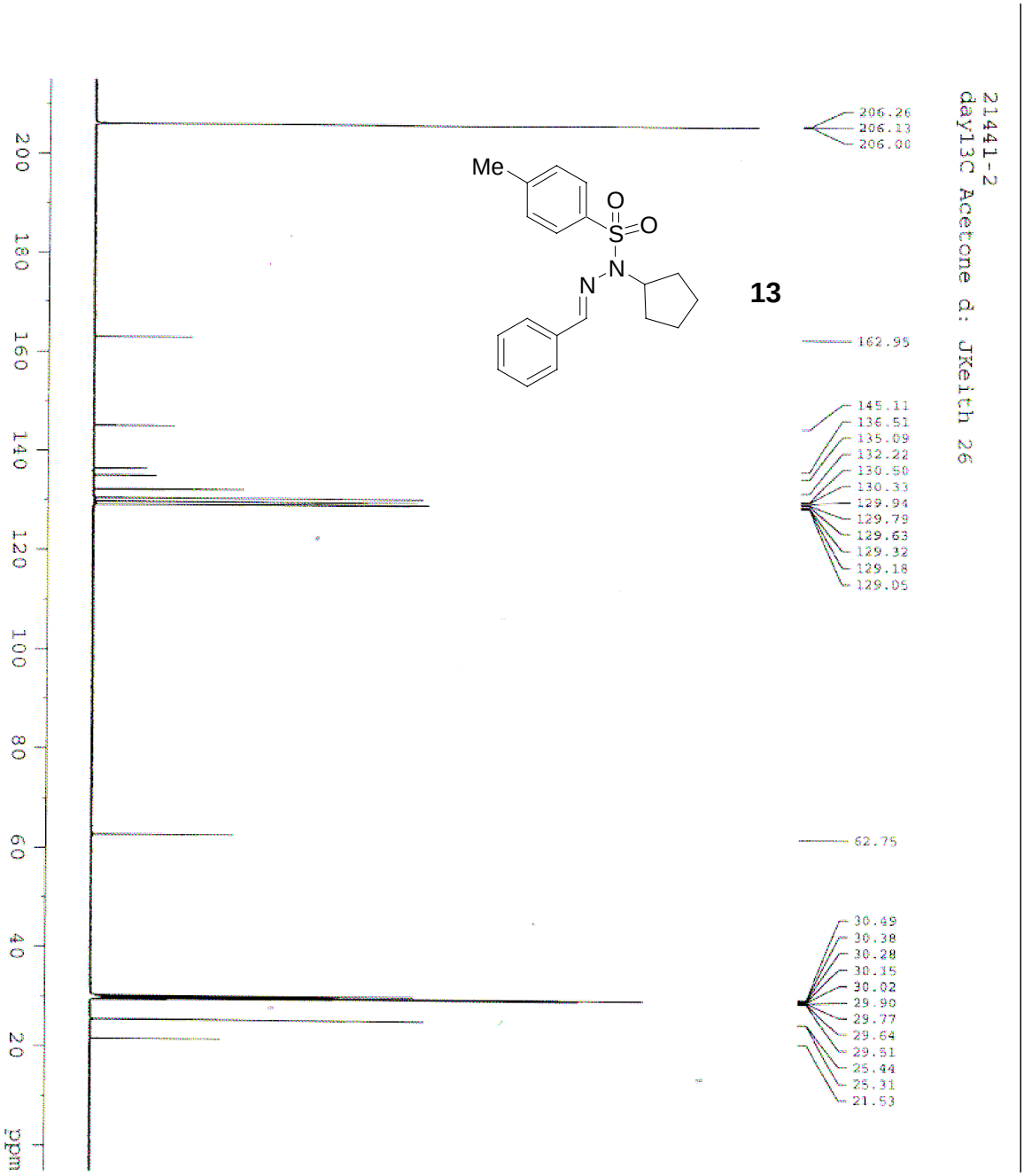
7.0
6.0
5.0
4.0
3.0
2.0
1.0
0.0

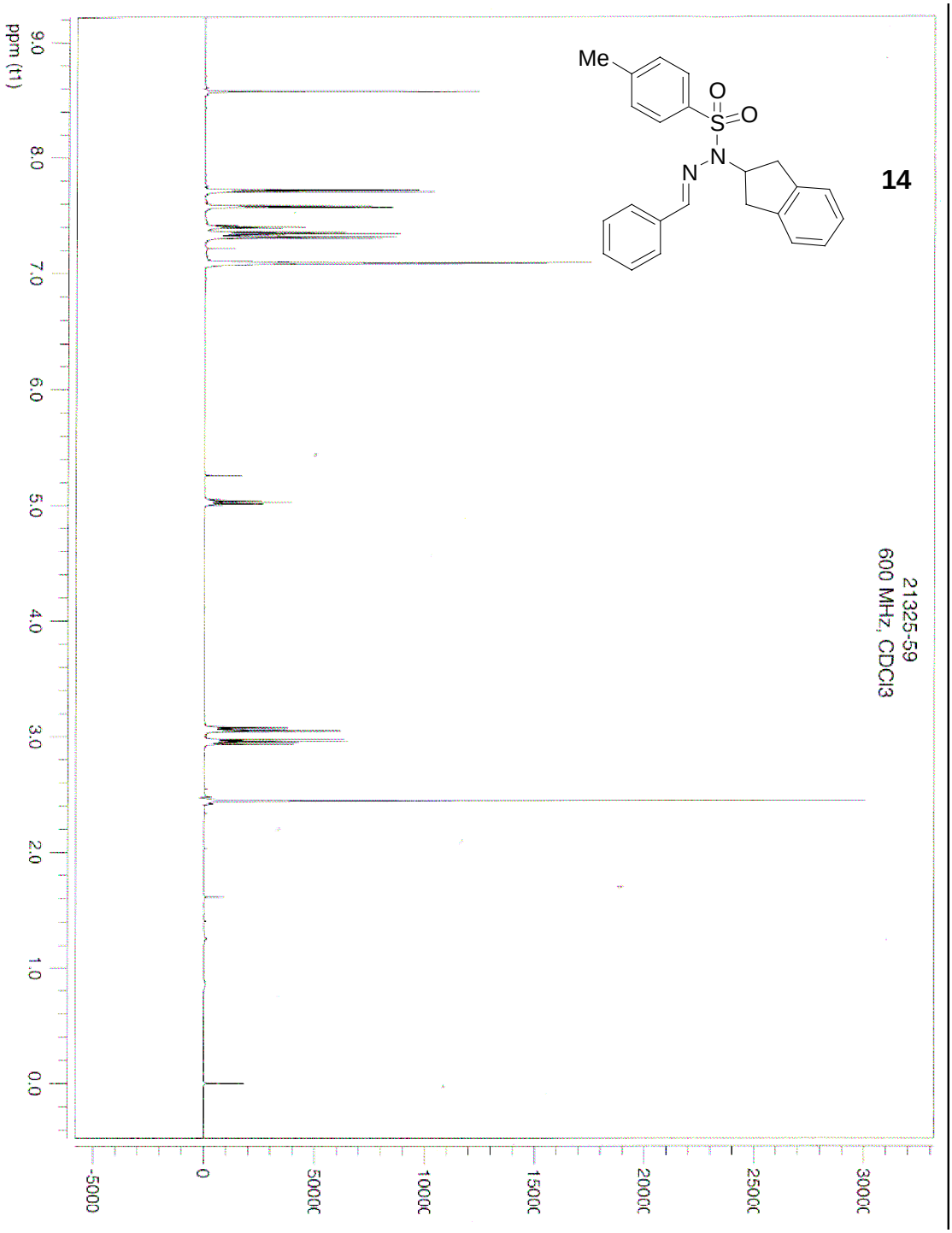

Me
O
S
O
N
N
N
Br
Br
N
Me
11
21441-7
600 MHz, CDCl3
200
150
100
50
0
ppm (t1)

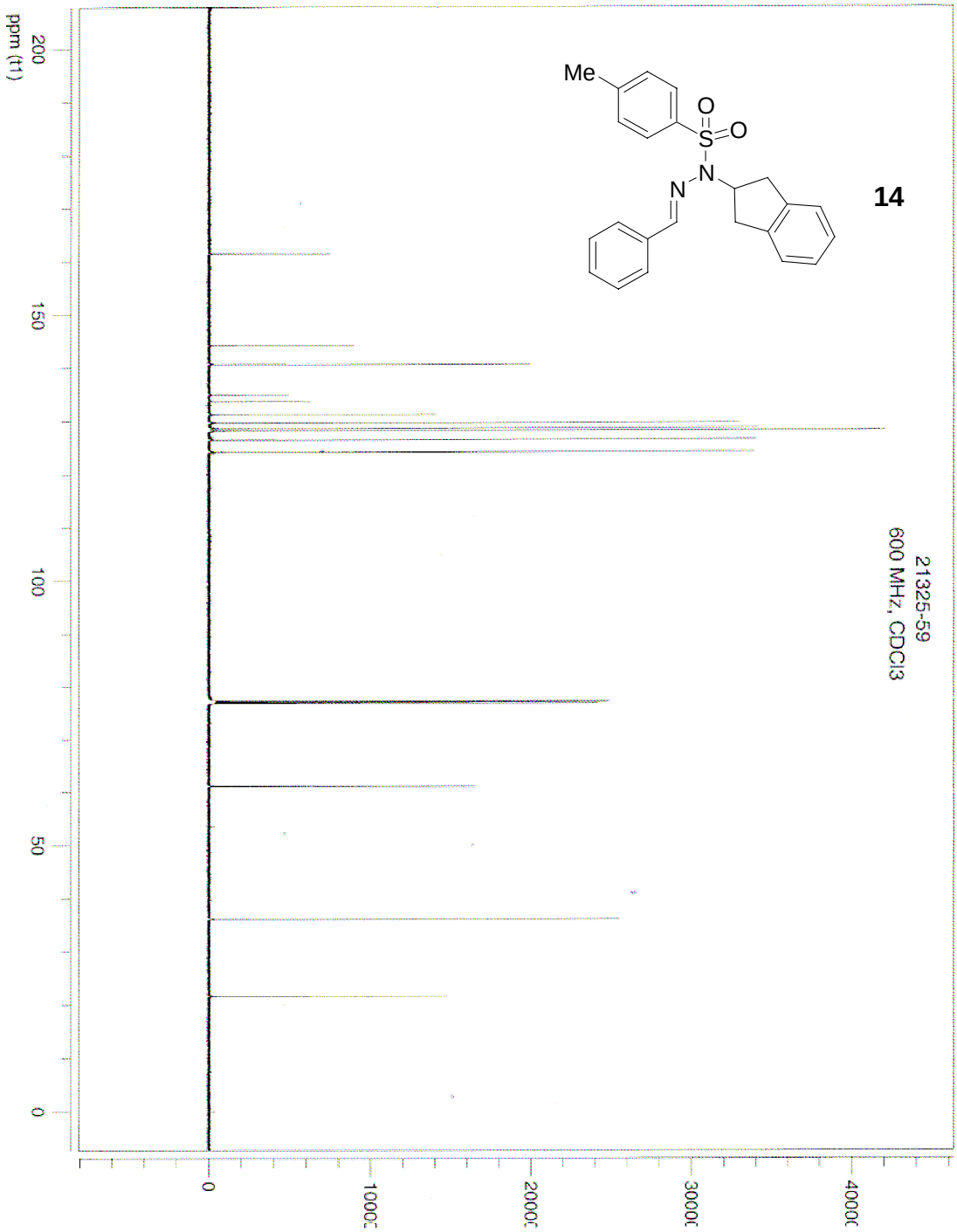

21325-178
600 MHz, CDCl3
12
Me
O
S
O
N
N
Me
Me
ppm (t1)
9.0
8.0
7.0
6.0
5.0
4.0
3.0
2.0
1.0
0.0
0
10000
20000
30000
40000
50000

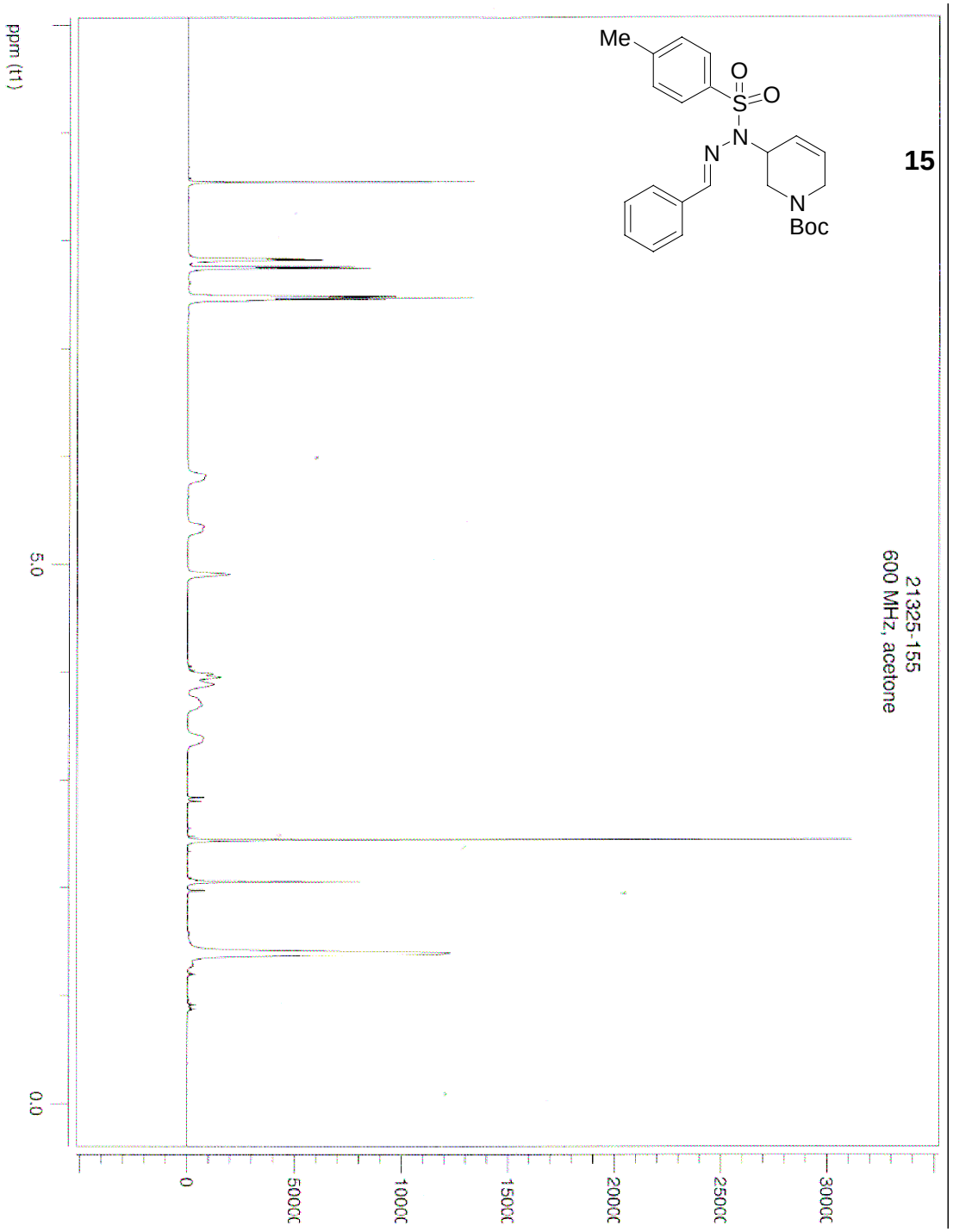

21325-178
600 MHz, CDCl3
12
Me
O
S
O
N
N
Me
Me
ppm (t1)
200
150
100
50
0
30000
25000
20000
15000
10000
5000
0

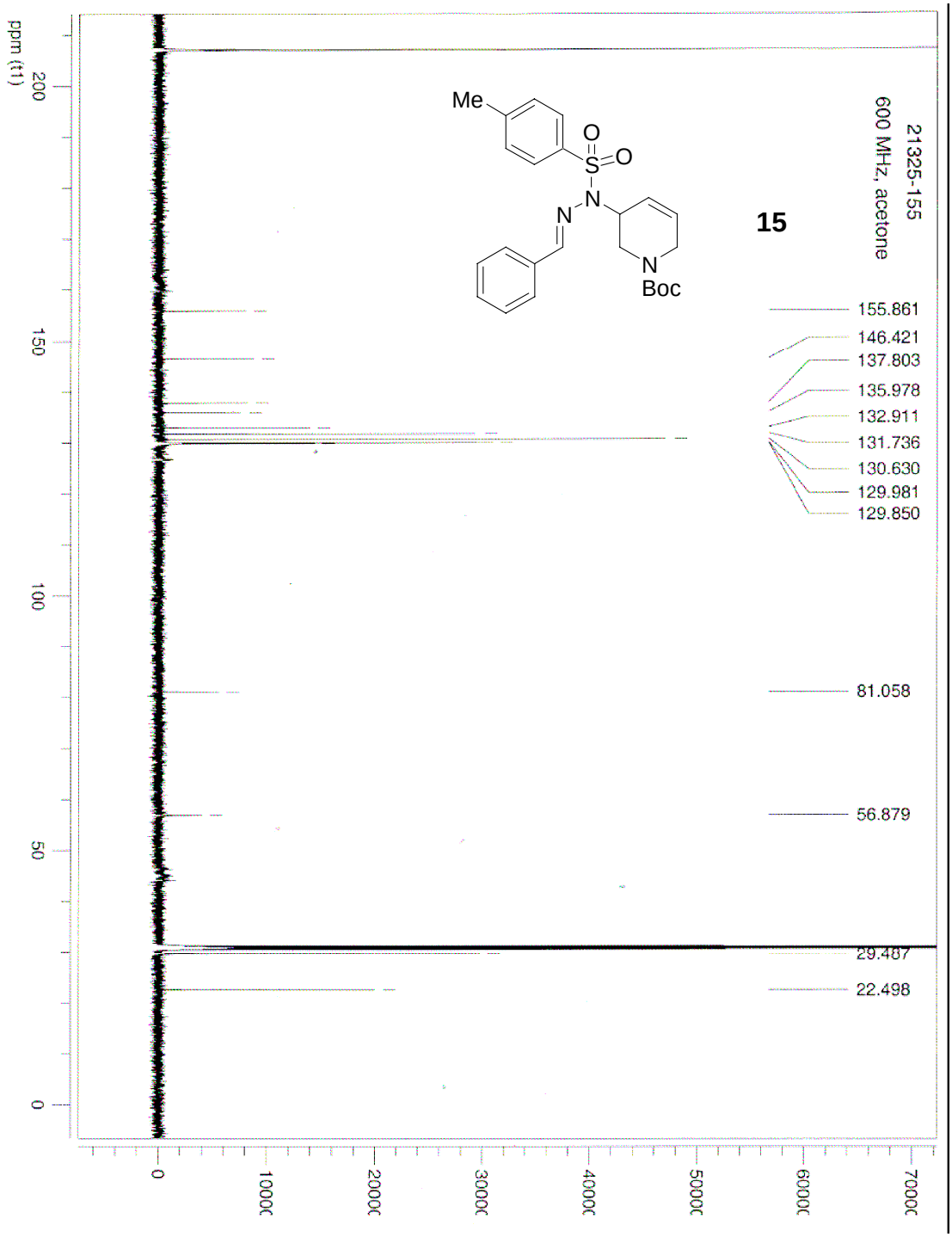

21441-2
acetone
8.759
7.839
7.837
7.825
7.823
7.722
7.708
7.515
7.506
7.494
7.432
7.419
4.568
2.433
1.677
1.663
1.578
1.571
1.568
1.565
1.556
1.432
1.430
1.427
1.425
1.00
2.07
2.01
3.07
2.03
1.04
3.15
2.17
4.37
2.19
Me
O
S
O
N
N
13
ppm (f1)
9.0
8.0
7.0
6.0
5.0
4.0
3.0
2.0
1.0
0
5000
10000
15000

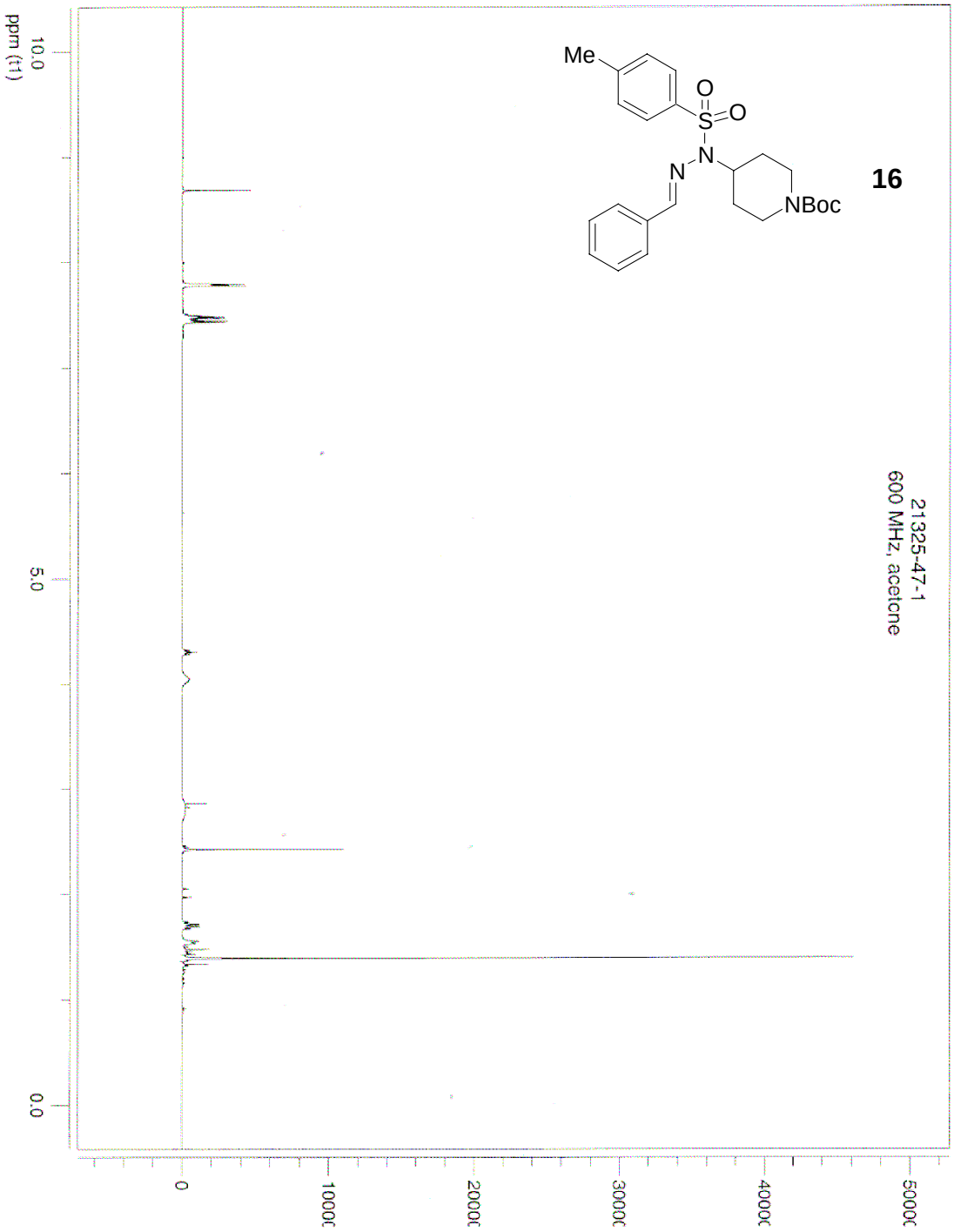

21441-2
day13C Acetone d: JKeith 26
206.26
206.13
206.00
162.95
145.11
136.51
135.09
132.22
130.50
130.33
129.94
129.79
129.63
129.32
129.18
129.05
62.75
30.49
30.38
30.28
30.15
30.02
29.90
29.77
29.64
29.51
25.44
25.31
21.53
Me
O
S
O
N
N
13
200
180
160
140
120
100
80
60
40
20
ppm

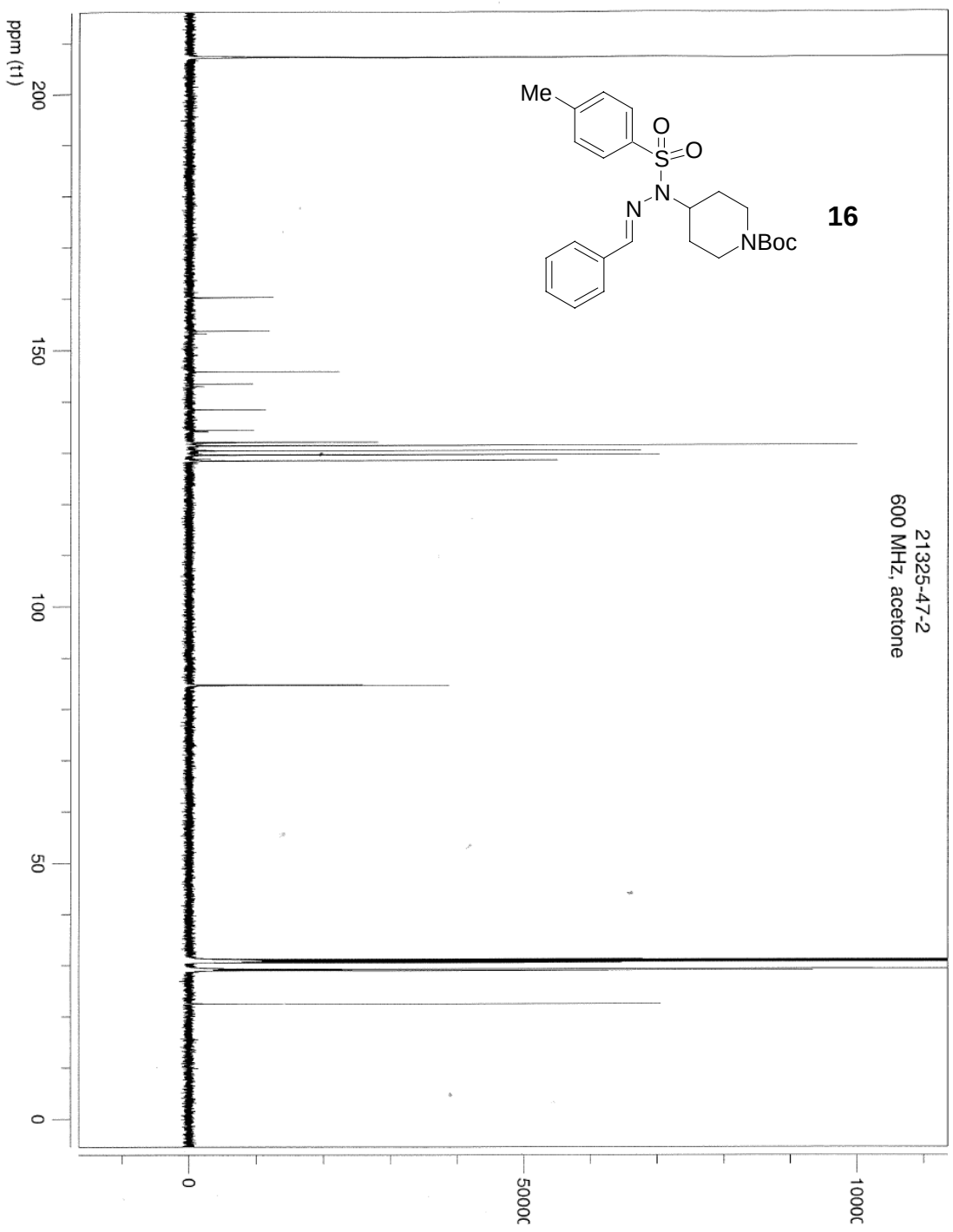

14
Me
O
S
O
N
N
21325-59
600 MHz, CDCl3
ppm (t1)
9.0
8.0
7.0
6.0
5.0
4.0
3.0
2.0
1.0
0.0
-5000
0
5000
10000
15000
20000
25000
30000

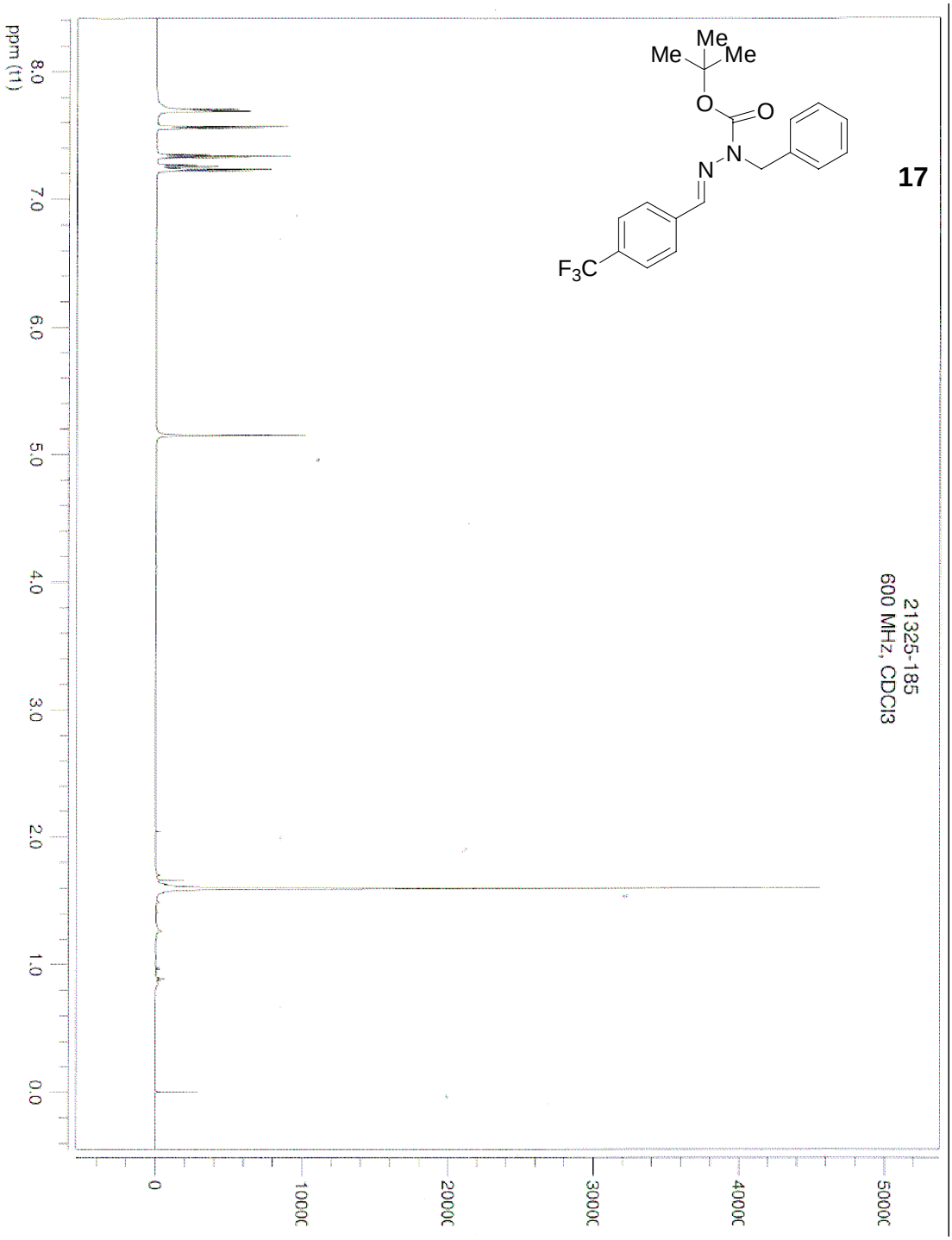

21325-59
600 MHz, CDCl3
14
Me
O
S
O
N
N
ppm (t1)
200
150
100
50
0
0
10000
20000
30000
40000

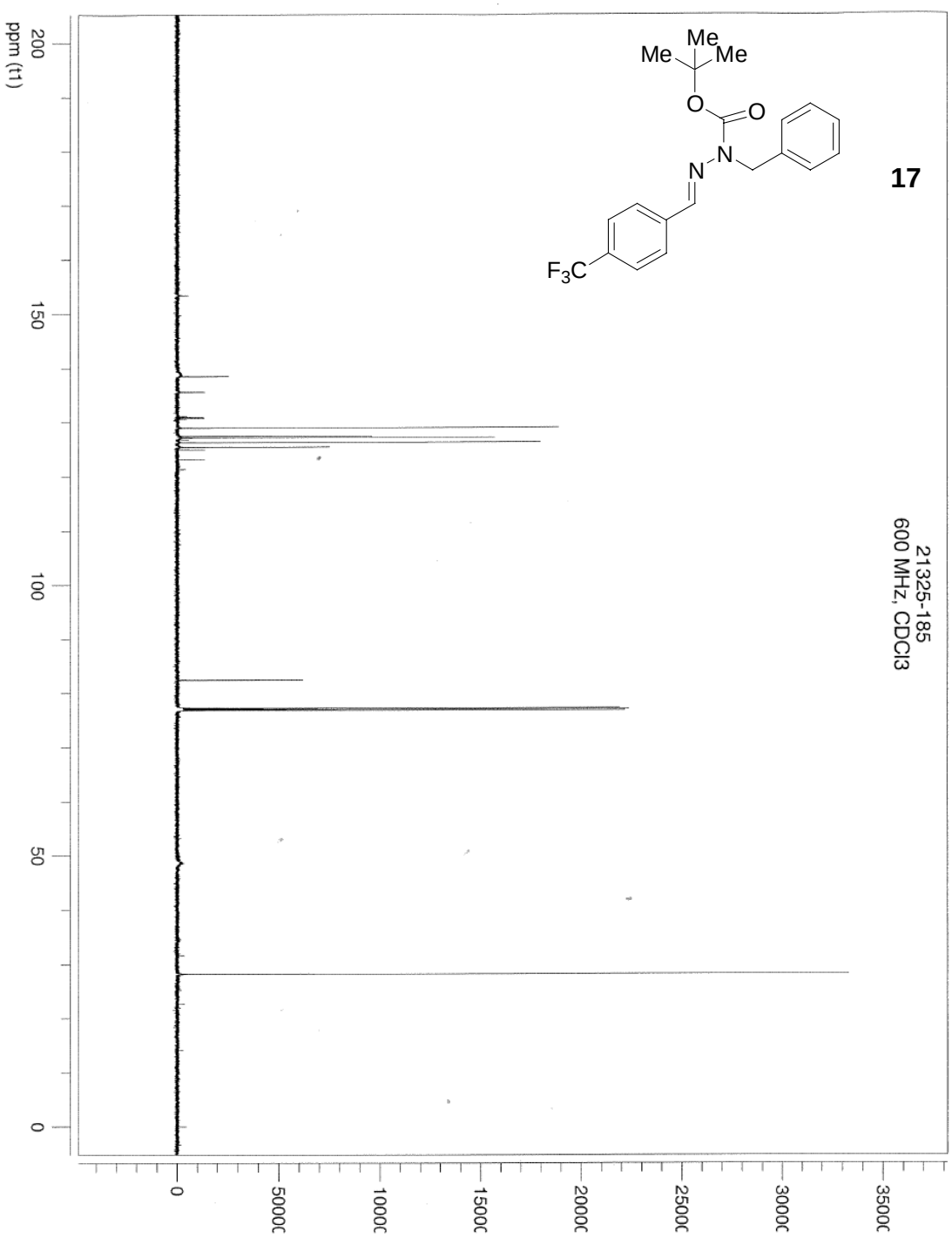

15
21325-155
600 MHz, acetone
Me
O
S
O
N
N
N
Boc
ppm (t1)
5.0
0.0
0
5000
10000
15000
20000
25000
30000

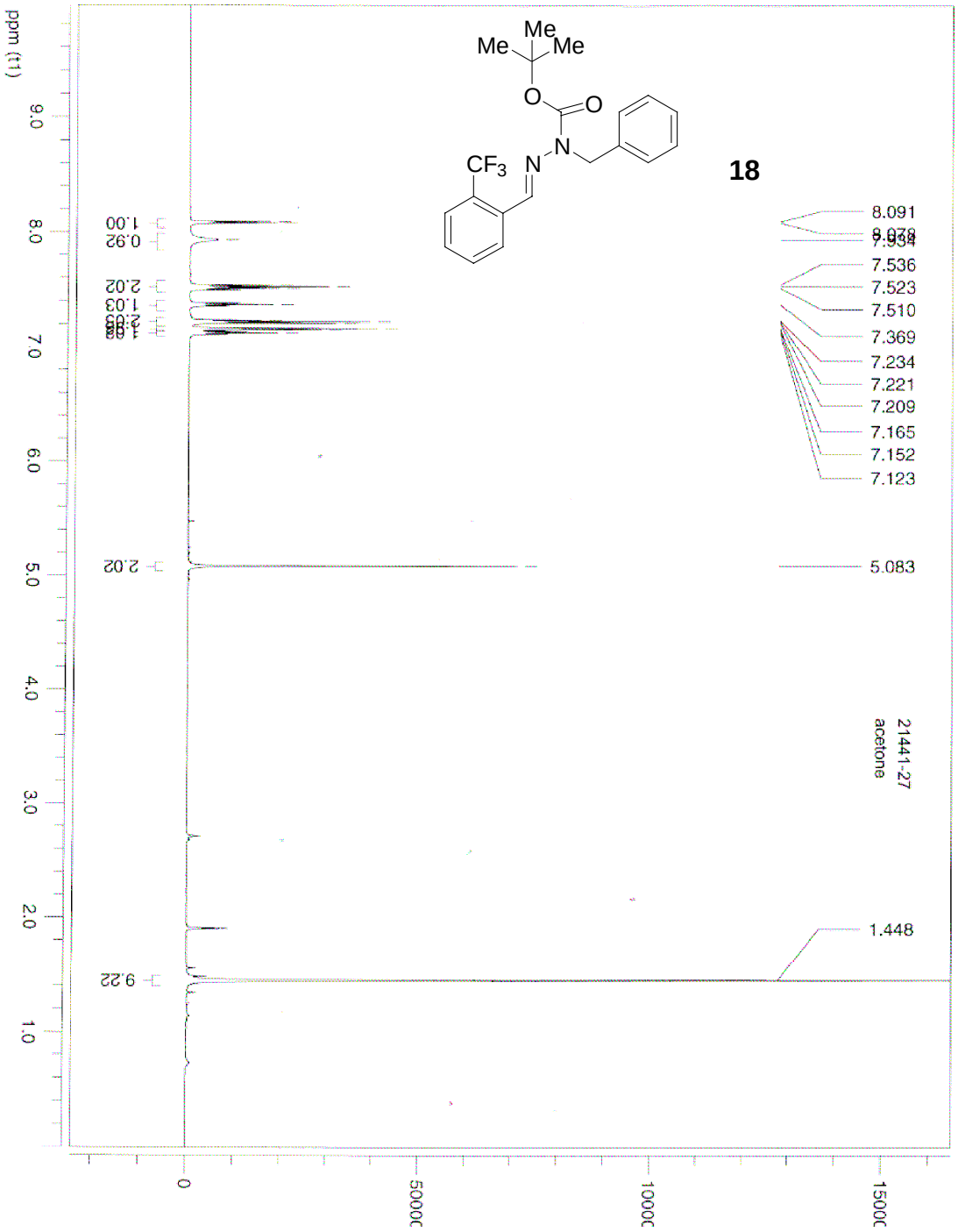

21325-155
600 MHz, acetone
15
Me
O
S
O
N
N
N
Boc
155.861
146.421
137.803
135.978
132.911
131.736
130.630
129.981
129.850
81.058
56.879
29.487
22.498
ppm (t1)
200
150
100
50
0
0
10000
20000
30000
40000
50000
60000
70000

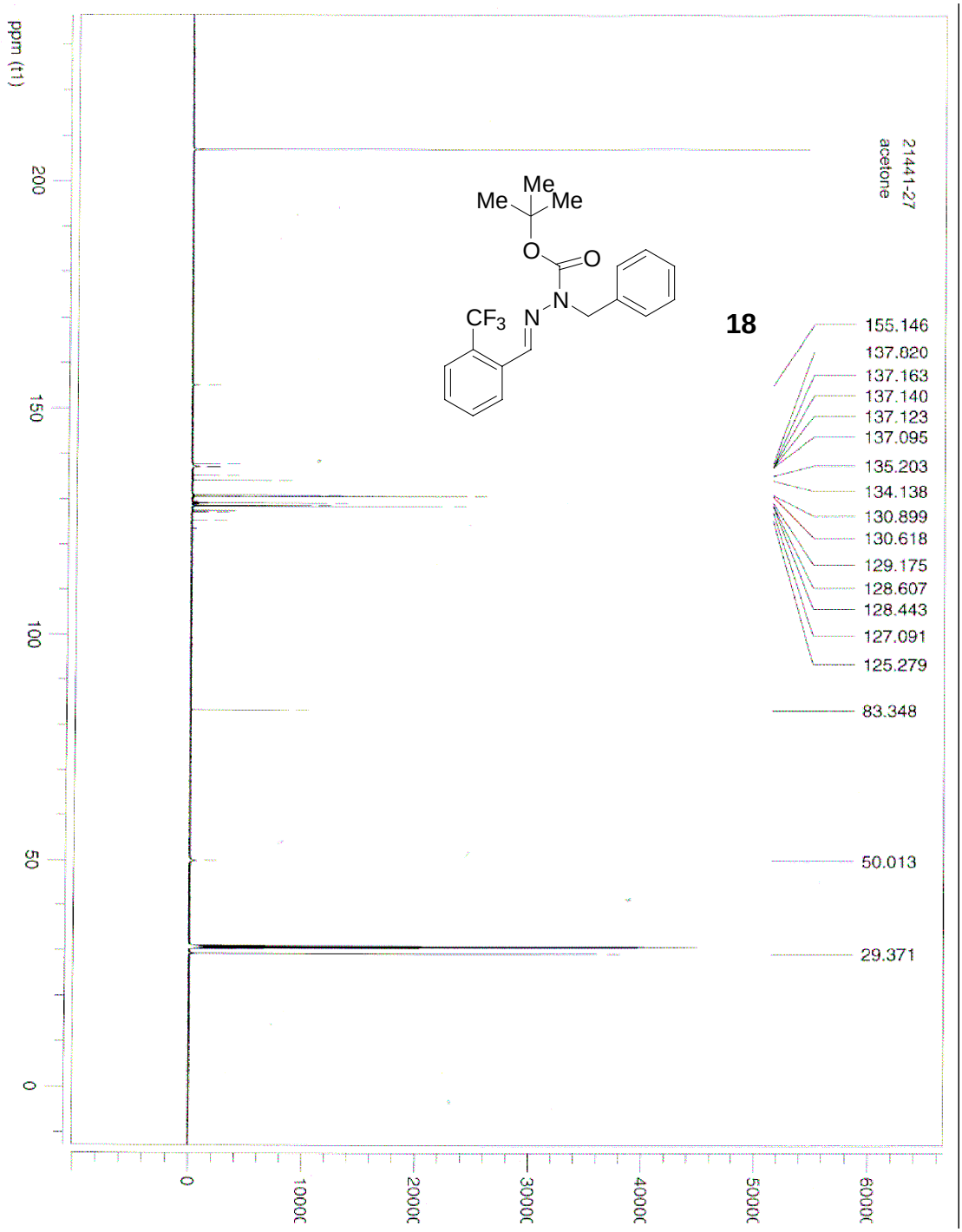

21325-47-1
600 MHz, acetone
16
Me
O
S
O
N
N
NBoc
5000
4000
3000
2000
1000
0
0.0
5.0
10.0
ppm (t1)

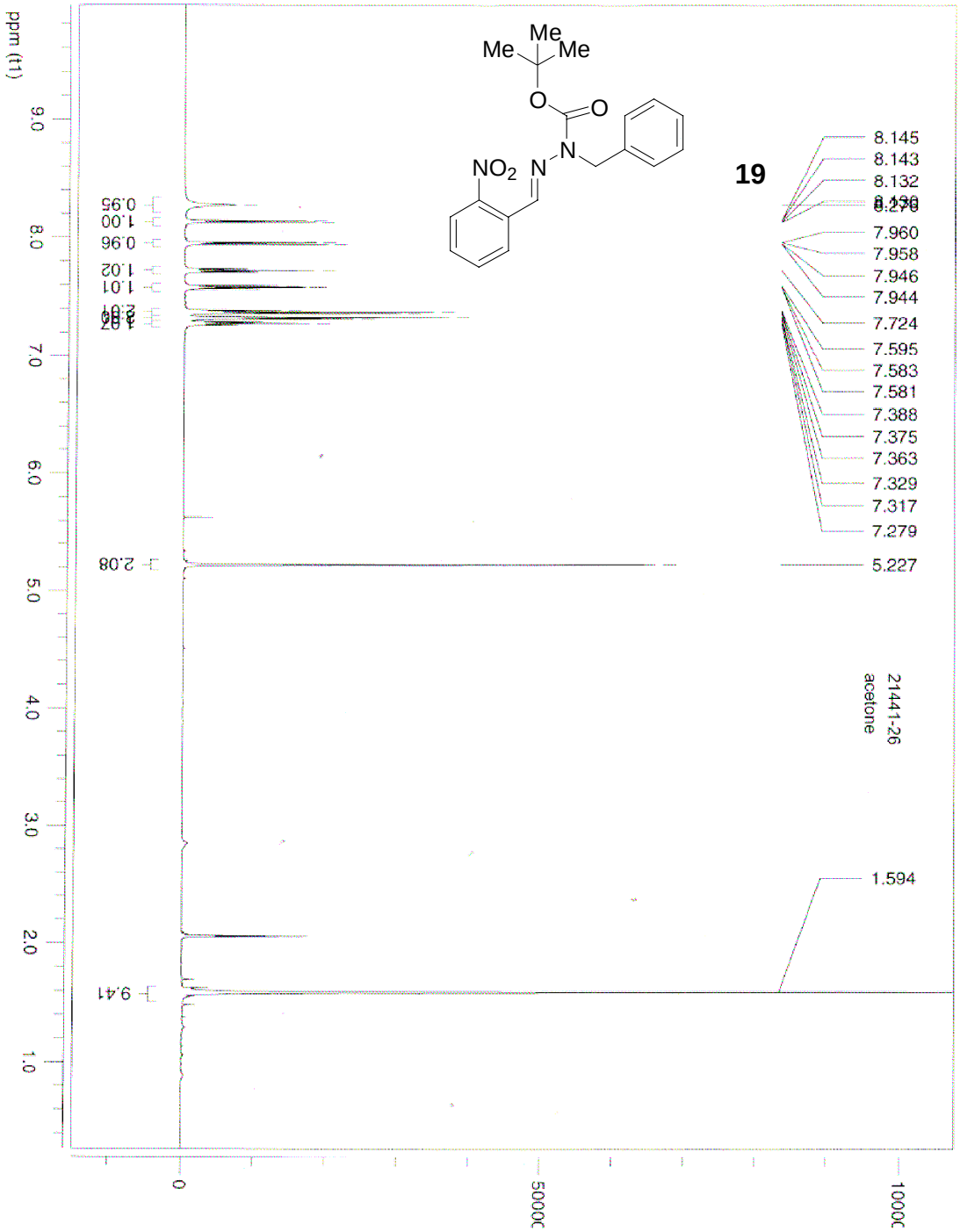

Me
O
S
O
N
N
NBoc
16
21325-47-2
600 MHz, acetone
ppm (t1)
200
150
100
50
0
0
50000
100000

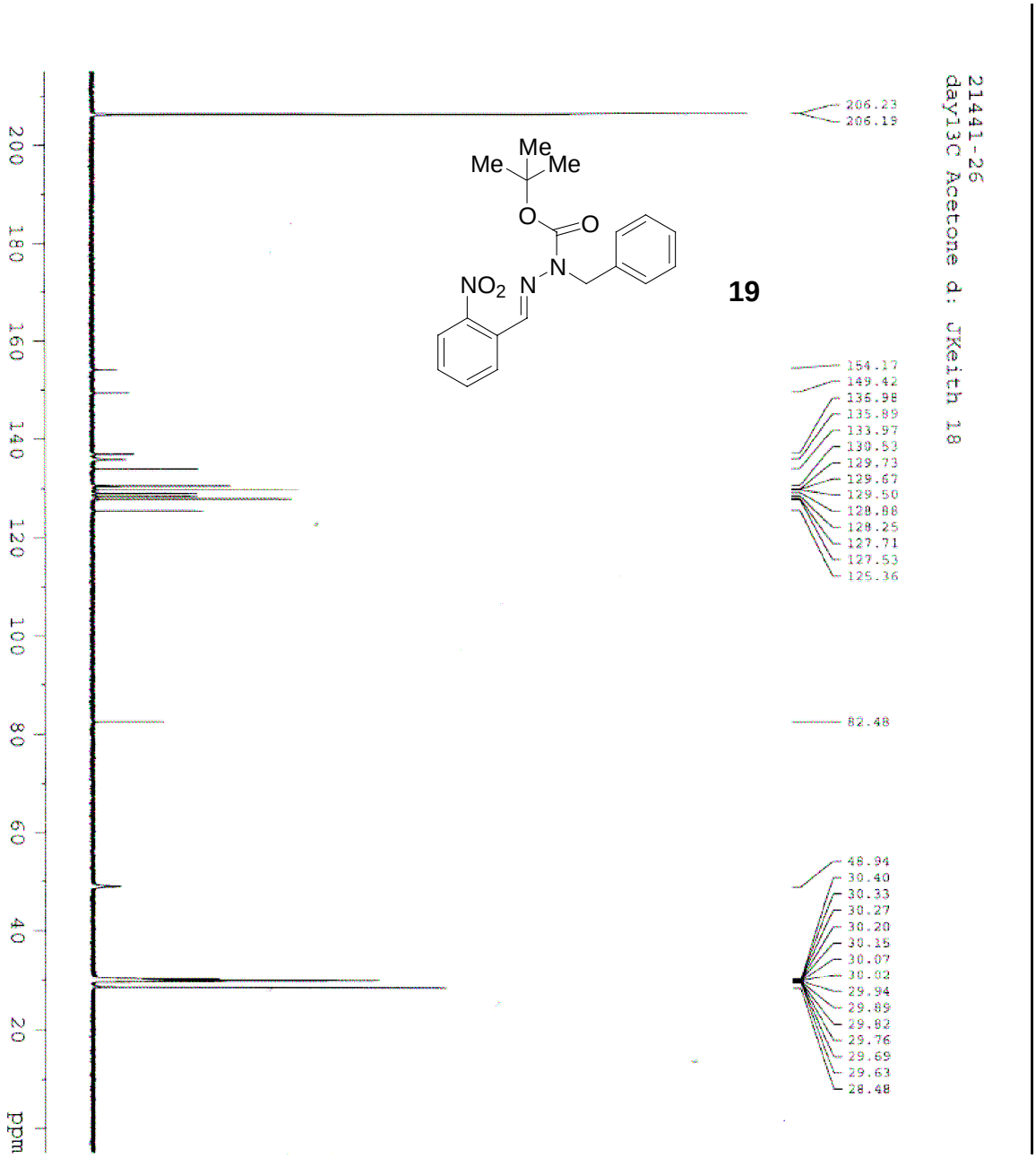

17
21325-185
600 MHz, CDCl3
Me
Me
Me
O
O
N
N
F3C
ppm (t1)
8.0
7.0
6.0
5.0
4.0
3.0
2.0
1.0
0.0
0
10000
20000
30000
40000
50000

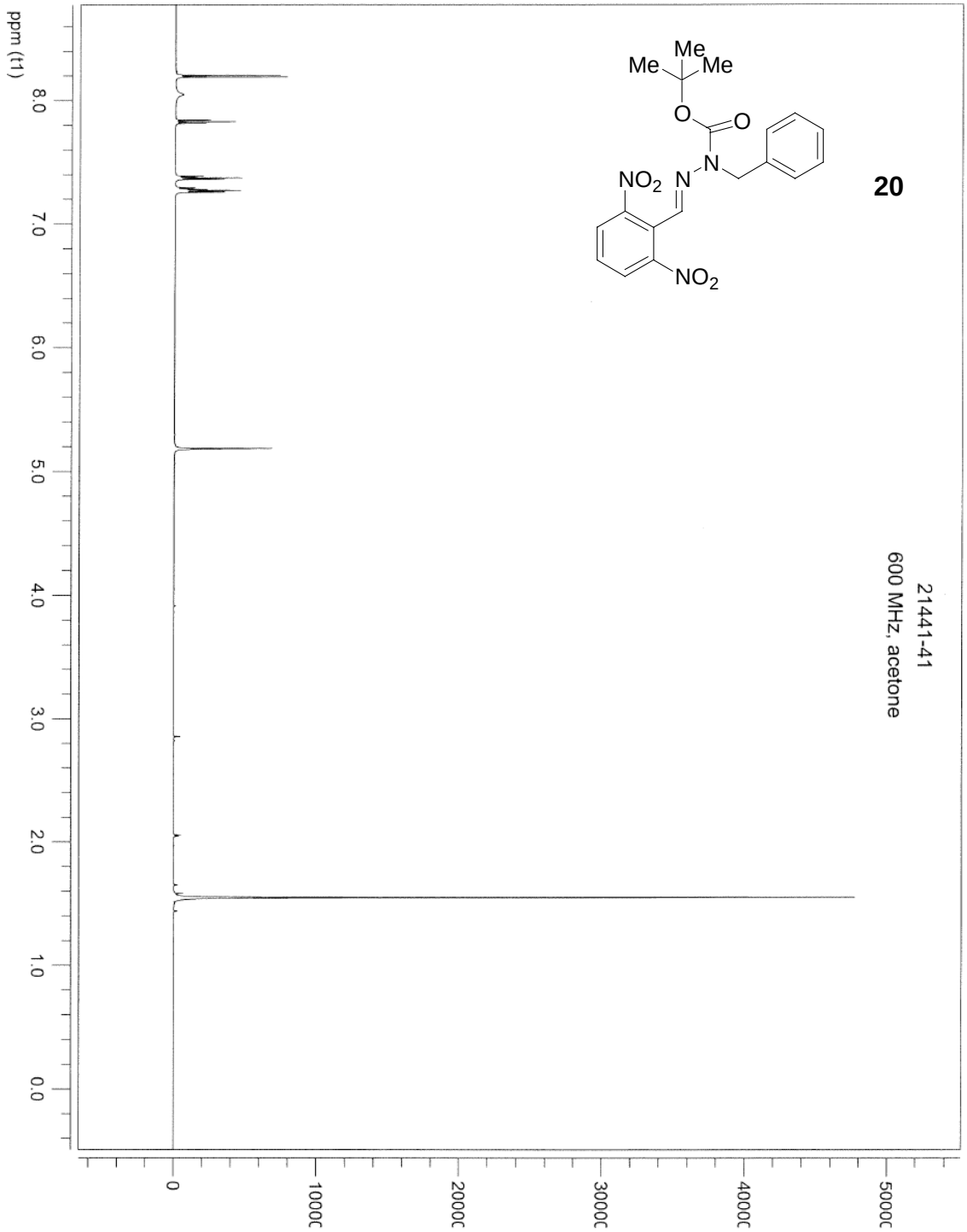

17
21325-185
600 MHz, CDCl3
ppm (t1)
200
150
100
50
0
0
5000
10000
15000
20000
25000
30000
35000

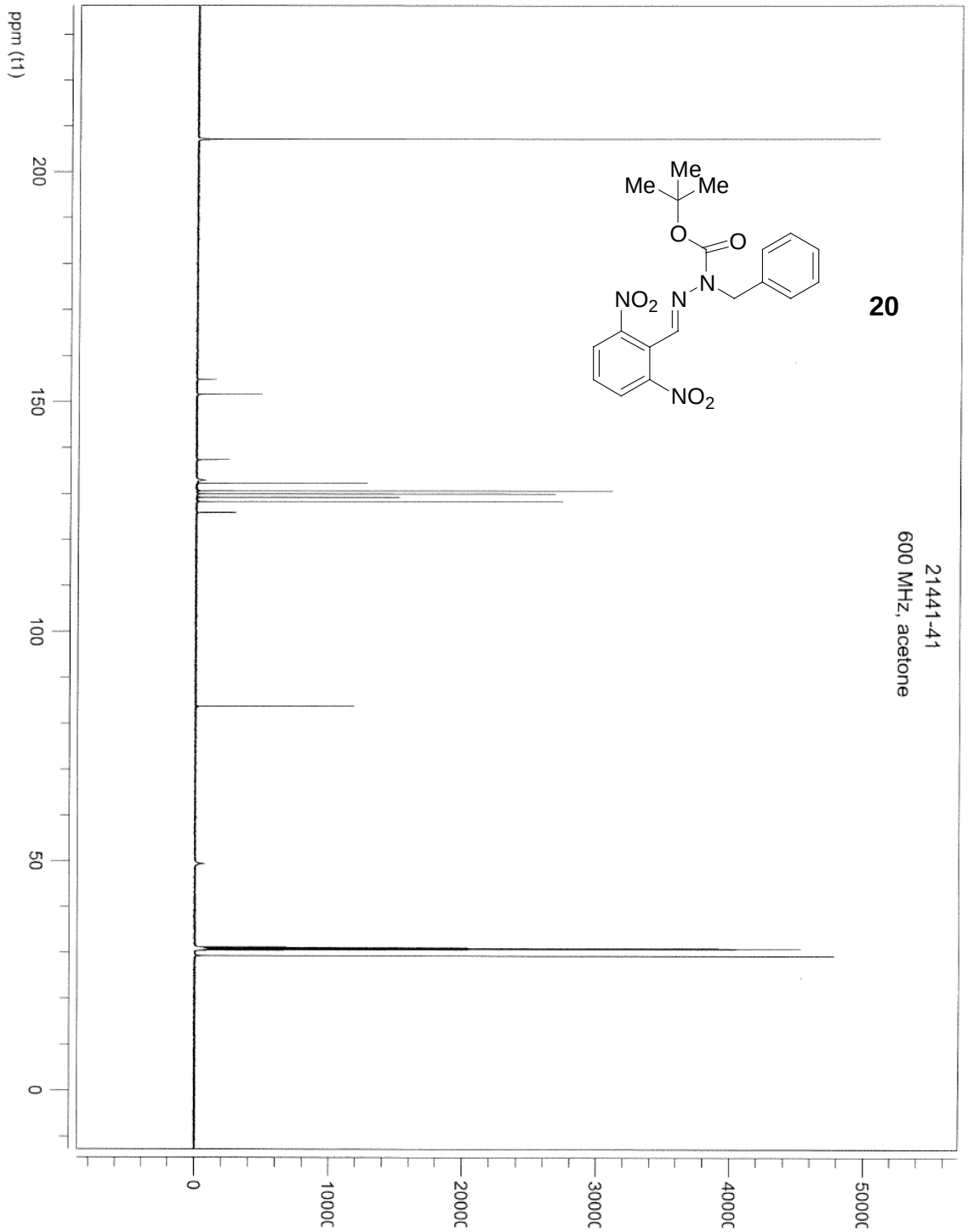

18
8.091
8.078
7.934
7.536
7.523
7.510
7.369
7.234
7.221
7.209
7.165
7.152
7.123
5.083
1.448
21441-27
acetone
1.00
0.92
2.02
1.03
2.05
1.02
2.02
9.22
ppm (t1)
9.0
8.0
7.0
6.0
5.0
4.0
3.0
2.0
1.0
0
5000
10000
15000


21441-27
acetone
18
155.146
137.820
137.163
137.140
137.123
137.095
135.203
134.138
130.899
130.618
129.175
128.607
128.443
127.091
125.279
83.348
50.013
29.371
ppm (t1)
200
150
100
50
0
0
10000
20000
30000
40000
50000
60000


19
Me
Me
Me
O
O
N
N
NO2
8.145
8.143
8.132
7.960
7.958
7.946
7.944
7.724
7.595
7.583
7.581
7.388
7.375
7.363
7.329
7.317
7.279
5.227
1.594
21441-26
acetone
2.08
9.41
0.95
1.00
0.96
1.02
1.01
ppm (t1)
9.0
8.0
7.0
6.0
5.0
4.0
3.0
2.0
1.0
0
5000
10000


21441-26
day13C Acetone d: JKeith 18
206.23
206.19
154.17
149.42
136.98
135.89
133.97
130.53
129.73
129.67
129.50
128.88
128.25
127.71
127.53
125.36
82.48
48.94
30.40
30.33
30.27
30.20
30.15
30.07
30.02
29.94
29.89
29.82
29.76
29.69
29.63
28.48
Me
Me
Me
O
O
N
N
NO2
19
200
180
160
140
120
100
80
60
40
20
ppm


Me
Me
Me
O
O
N
N
NO2
NO2
20
21441-41
600 MHz, acetone
ppm (t1)
8.0
7.0
6.0
5.0
4.0
3.0
2.0
1.0
0.0
0
10000
20000
30000
40000
50000


21441-41
600 MHz, acetone
20
Me
Me
Me
O
O
N
N
NO2
NO2
ppm (t1)
200
150
100
50
0
0
10000
20000
30000
40000
50000