SUPPORTING INFORMATION

Synthesis of α-Cyano and α-Sulfonyl Cyclic Ethers via Intramolecular Reactions of Peroxides with Sulfone- and Nitrile-Stabilized Carbanions

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2-(But-3-en-1-yl)octanenitrile (**1**)
^{1}H S6
^{13}C S7

2-(3-Methylbut-3-en-1-yl)octanenitrile (**2**)
^{1}H S8
^{13}C S9

2-Phenylpent-4-enenitrile (**3**)
^{1}H S10
^{13}C S11

2-Phenylhex-5-enenitrile (**4**)
^{1}H S12
^{13}C S13

2-Phenylhept-6-enenitrile (**5**)
^{1}H S14
^{13}C S15

2-Phenyloct-7-enenitrile (**6**)
^{1}H S16
^{13}C S17

5-Methyl-2-phenylhex-5-enenitrile (**7**)
^{1}H S18
^{13}C S19

2-(3-((Triethylsilyl)peroxy)butyl)octanenitrile (**1si**)
^{1}H S20
^{13}C S21

2-(3-Methyl-3-((triethylsilyl)peroxy)butyl)octanenitrile (**2si**)
^{1}H S22
^{13}C S23

2-Phenyl-4-((triethylsilyl)peroxy)pentanenitrile (**3si)**
^{1}H S24
^{13}C S25

2-Phenyl-5-((triethylsilyl)peroxy)hexanenitrile (**4si**)
^{1}H S26
^{13}C S27

2-Phenyl-6-((triethylsilyl)peroxy)heptanenitrile (**5si**)
^{1}H S28
^{13}C S29

2-Phenyl-7-((triethylsilyl)peroxy)octanenitrile (**6si**)
^{1}H S30
^{13}C S31

5-Methyl-2-phenyl-5-((triethylsilyl)peroxy)hexanenitrile (**7si**)
^{1}H S32
^{13}C S33

5-Hydroperoxy-2-phenylhexanenitrile (**4h**)
^{1}H S34
^{13}C S35

2-Phenyl-5-(tetrahydro-2*H*-pyran-2-yl)peroxy hexanenitrile (**4thp**)
^{1}H S36
^{13}C S37

2-(3-Chloropropyl)octanenitrile (**8**)
^{1}H S38
^{13}C S39

2-(3-Iodopropyl)octanenitrile (**9i**)
^{1}H S40
^{13}C S41

2-(3-(*tert*-Butylperoxy)propyl)octanenitrile (**9bu**)
^{1}H S42
^{13}C S43

3-Chloropropyl-phenyl sulfide (**10a**)
^{1}H S44
^{13}C S45

4-Chlorobutyl-phenyl sulfide (**11a**)
^{1}H S46
^{13}C S47

5-Chloropentyl-phenyl sulfide (**12a)**
^{1}H S48
^{13}C S49

6-Chlorohexyl-phenyl sulfide (**13a**)
^{1}H S50
^{13}C S51

3-Chloropropyl-sulfonyl benzene (**10b**)
^{1}H S52
^{13}C S53

4-Chlorobutyl-sulfonyl benzene (**11b**)
^{1}H S54
^{13}C S55

5-Chloropentyl-sulfonyl benzene (**12b**)
^{1}H S56
^{13}C S57

6-Chlorohexyl-sulfonyl benzene (**13b**)
^{1}H S58
^{13}C S59

3-Iodopropylsulfonyl benzene (**10c**)
^{1}H S60
^{13}C S61

4-Iodobutylsulfonyl benzene (**11c**)
^{1}H S62
^{13}C S63

5-Iodopentylsulfonyl benzene (**12c**)
^{1}H S64
^{13}C S65

6-Iodohexylsulfonyl benzene (**13c**)
^{1}H S66
^{13}C S67

3-(*tert*-Butylperoxy)propyl-sulfonyl benzene (**10bu**)
^{1}H S68
^{13}C S69

Cyclopropyl sulfonyl benzene
^{1}H S70
^{13}C S71

4-(*tert*-Butylperoxy)butyl-sulfonyl benzene (**11bu**)
^{1}H S72
^{13}C S73

2-((4-Phenylsulfonyl)butyl)peroxy)tetrahydro-2*H*-pyran (**11thp**)
^{1}H S74

^{13}C S75

5-(*tert*-Butylperoxy)pentyl-sulfonyl benzene (**12bu**)
^{1}H S76
^{13}C S77

6-(*tert*-Butylperoxy)hexyl-sulfonyl benzene (**13bu**)
^{1}H S78
^{13}C S79

Pent-4-en-1-yl(phenyl)sulfide (**14**)
^{1}H S80
^{13}C S81

Triethyl (5-phenylthiopentan-2-yl)peroxysilane (**15**)
^{1}H S82
^{13}C S83

Triethyl (5-phenylsulfonylpentan-2-yl peroxy) silane (**16si**)
^{1}H S84
^{13}C S85

(4-Hydroperoxypentyl)sulfonylbenzene (**16h**)
^{1}H S86
^{13}C S87

4-(Methylperoxy)pentylsulfonyl benzene (**16me**)
^{1}H S88
^{13}C S89

5-(Phenylsulfonyl)pentan-2-yl tetrahydro-2*H*-pyran-2-yl peroxide (**16thp**)
^{1}H S90
^{13}C S91

5-Methyl-2-phenyltetrahydrofuran-2-carbonitrile (**17ab**, from **4-si)**
^{1}H S92
^{13}C S93
17ab from **4-thp** (^{1}H) S94
17ab from **4-thp** (^{13}C) S95

5,5-Dimethyl-2-phenyltetrahydrofuran-2-carbonitrile (**18**)
^{1}H S96
^{13}C S97

4-Methyl-2-phenyloxetane-2-carbonitrile (**19**)
^{1}H S98
^{13}C S99

7-methyl-2-phenyloxepane-2-carbonitrile (**20**)
^{1}H S100

^{13}C S101

1-(5-Methyl-2-phenyltetrahydrofuran-2-yl)heptan-1-one (**21**)
^{1}H S102
NOESY spectra S103
Table S1: Peak positions and spectral correlations S104
^{13}C S105

2-Hexyltetrahydrofuran-2-carbonitrile (**22**)
^{1}H S106
^{13}C S107

2-Hexyl-5-methyltetrahydrofuran-2-carbonitrile (**23**)
^{1}H S108
^{13}C S109

2-Hexyl-5,5-dimethyltetrahydrofuran-2-carbonitrile (**24**).
^{1}H S110
^{13}C S111

6-(3-Hydroxy-3-methylbutyl)dodecan-5-one (**25**)
^{1}H S112
^{13}C S113

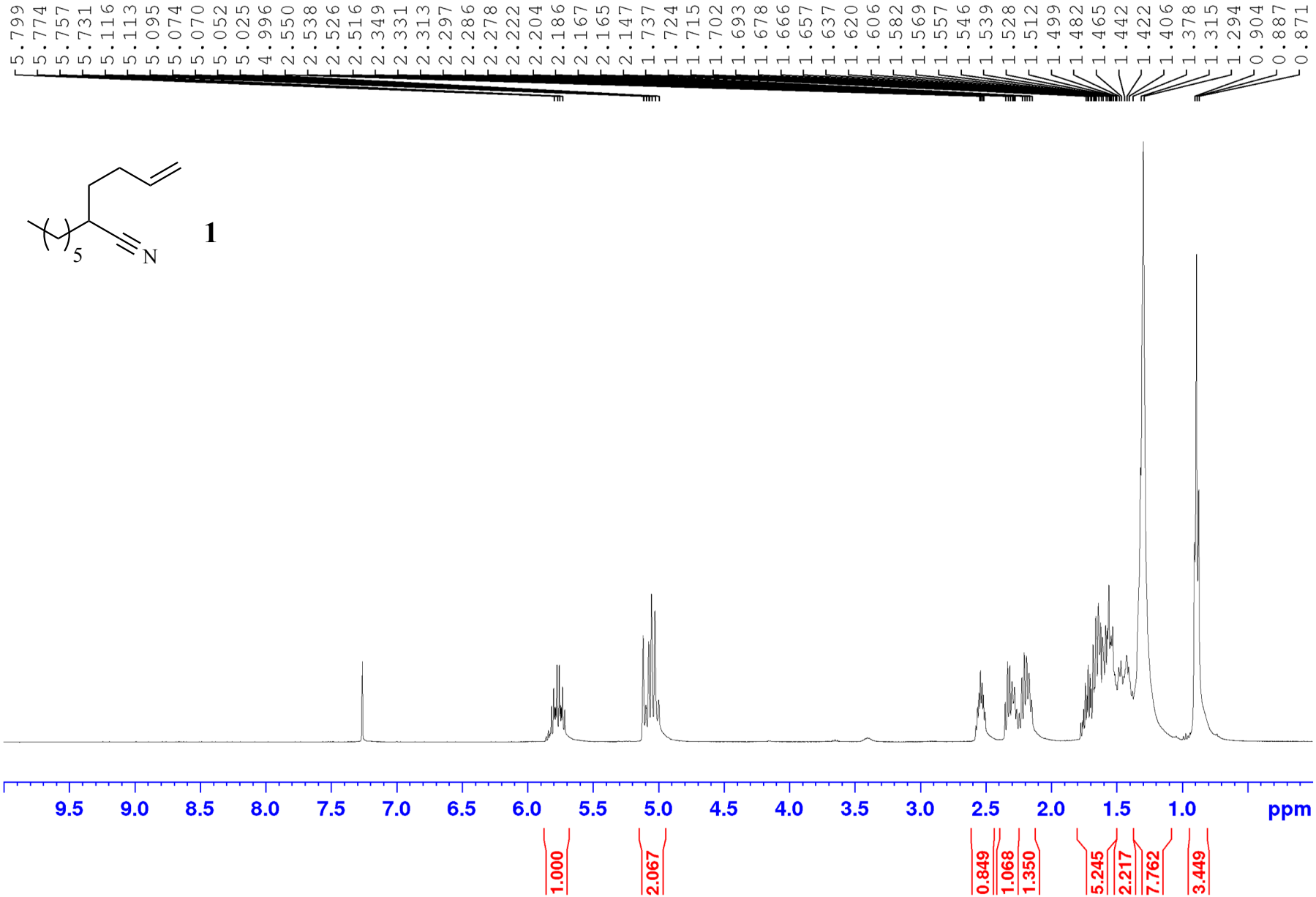
2-(Phenylsulfonyl)oxetane (**26**)
^{1}H S114
^{13}C S115

2-(Phenylsulfonyl)tetrahydrofuran (**27**)
^{1}H S116
^{13}C S117

2-(Phenylsulfonyl)tetrahydro-2*H*-pyran (**28**)
^{1}H S118
^{13}C S119

2-Methyl-5-phenylsulfonyl tetrahydrofuran (**29**)
^{1}H S120
^{13}C S121

5-(Phenylsulfonyl)pentan-2-ol (**30**)
^{1}H S122
^{13}C S123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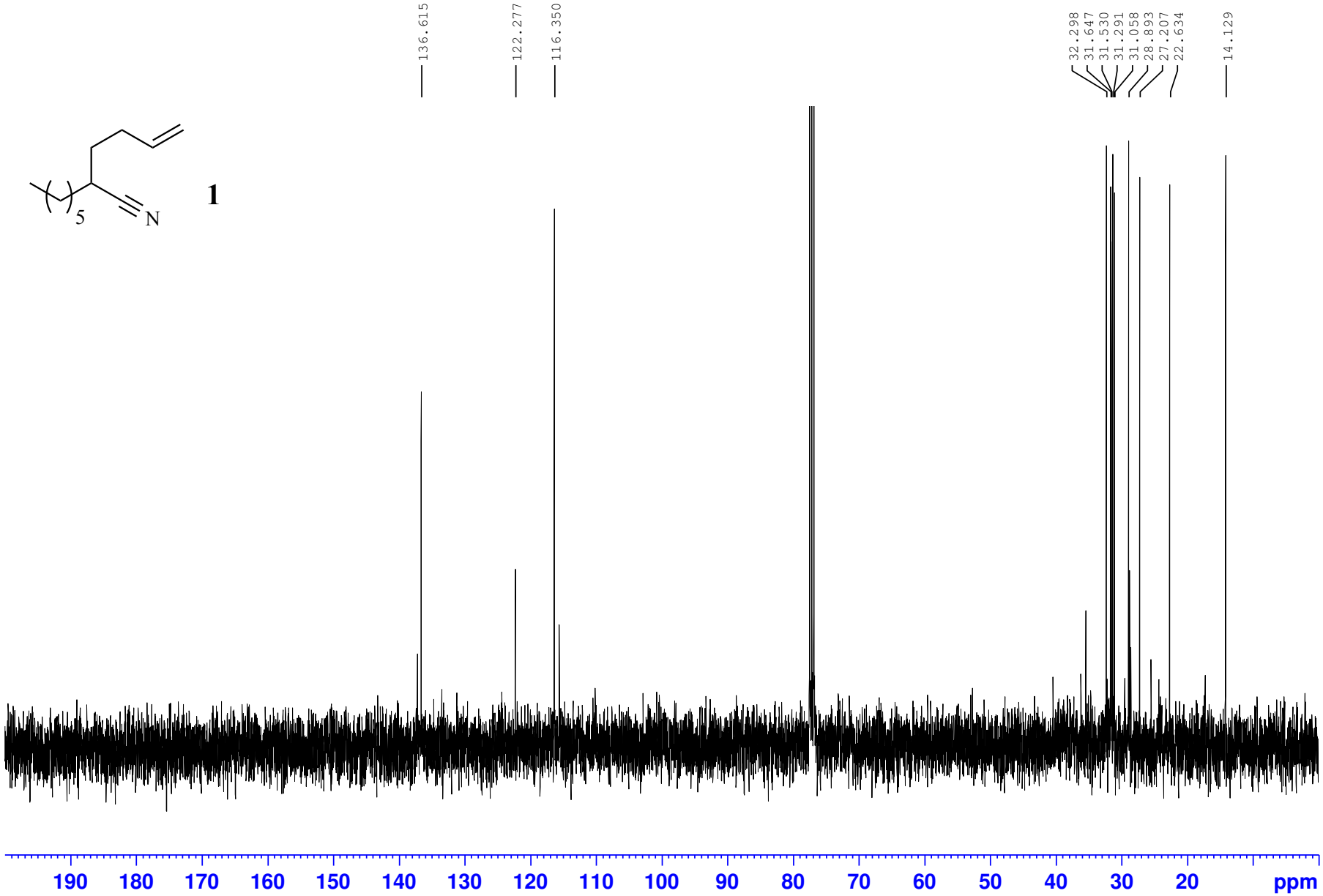
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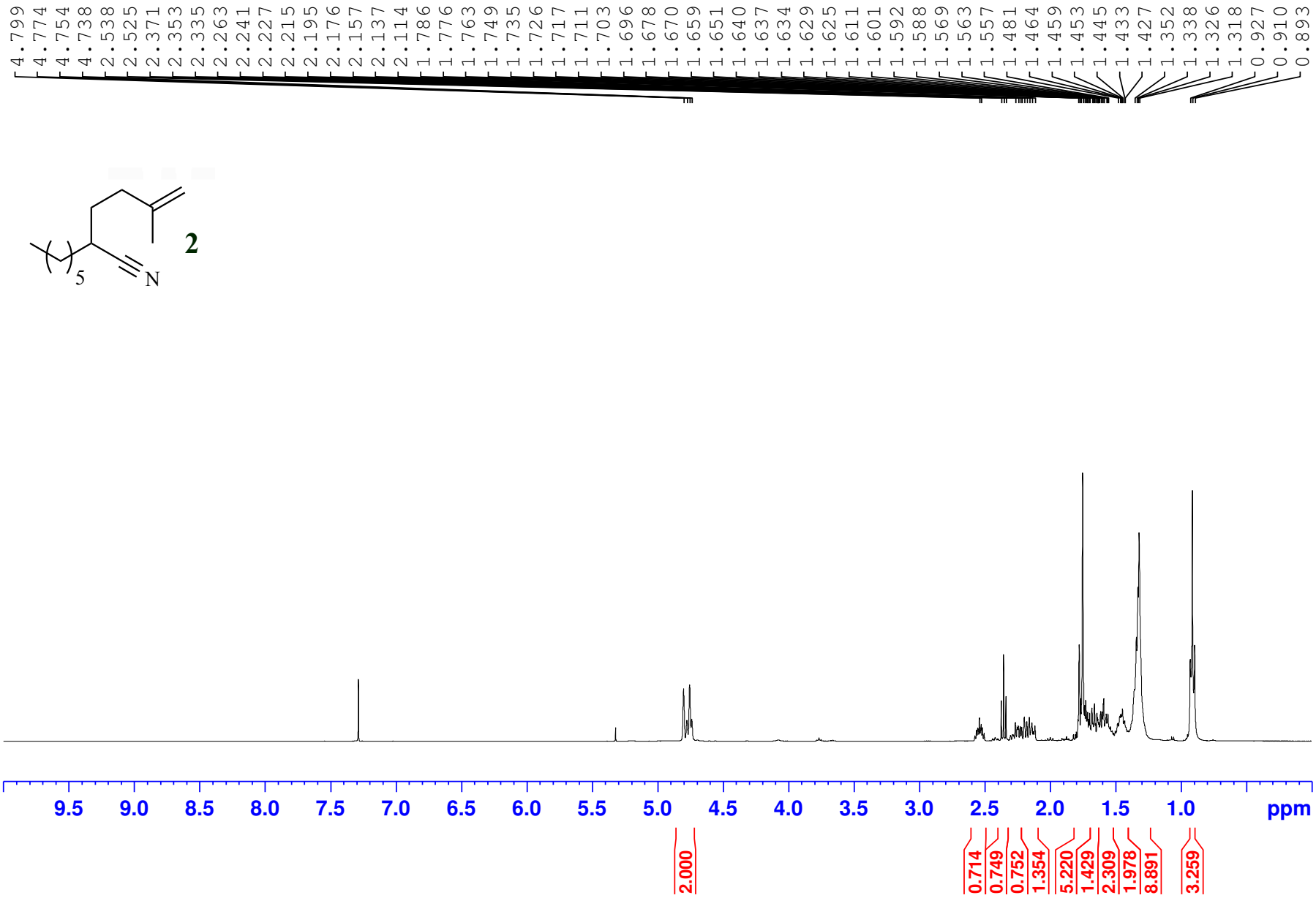
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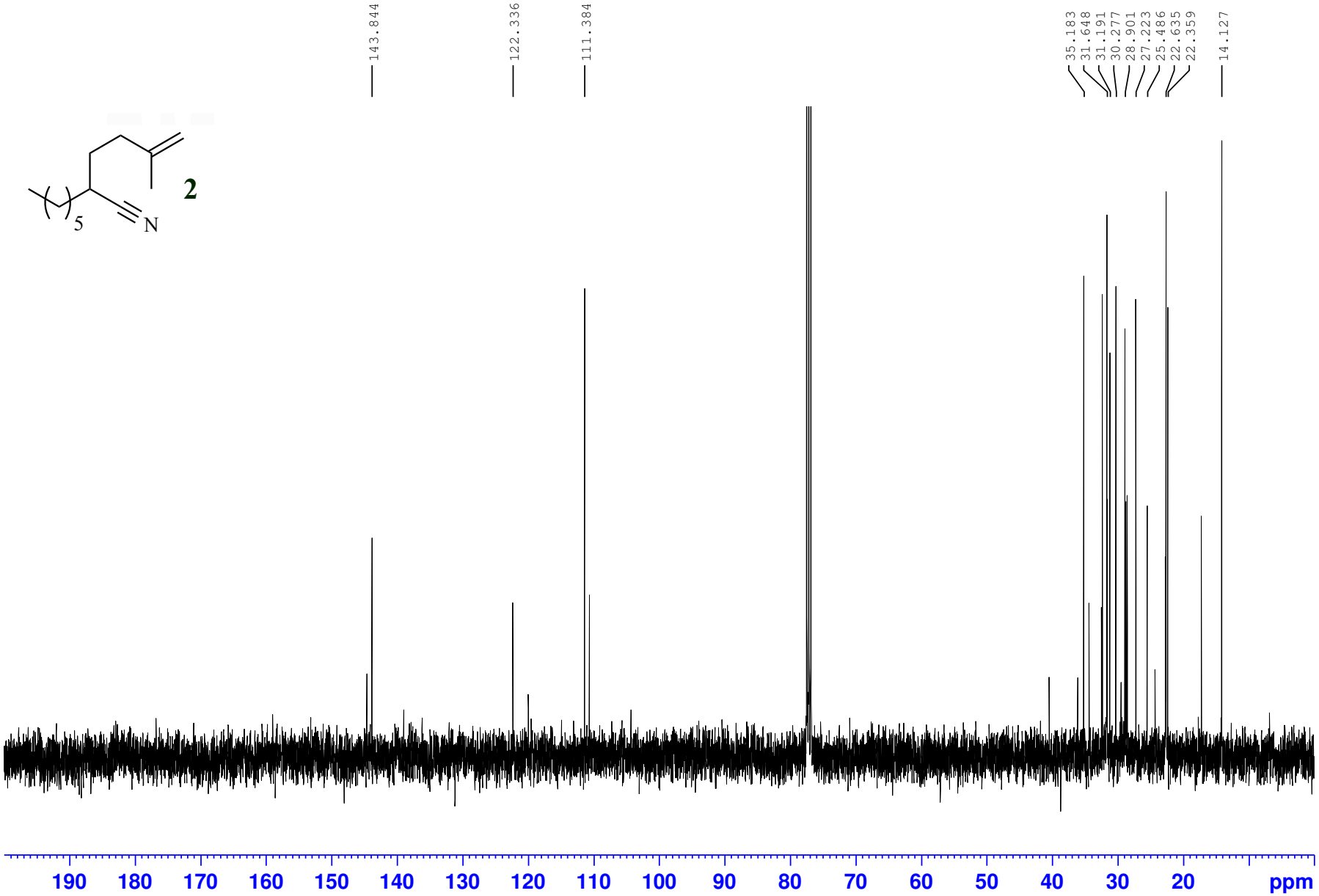
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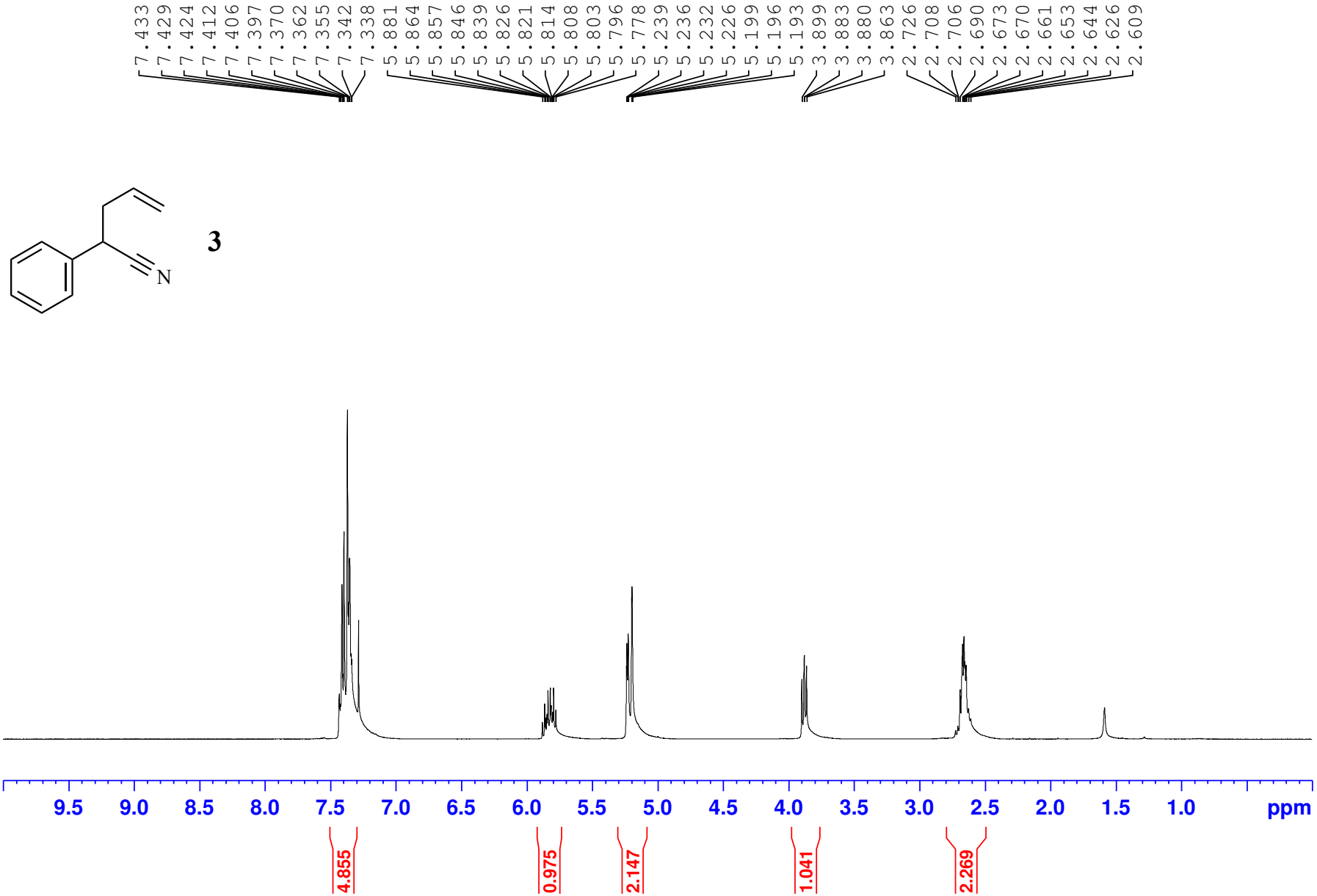
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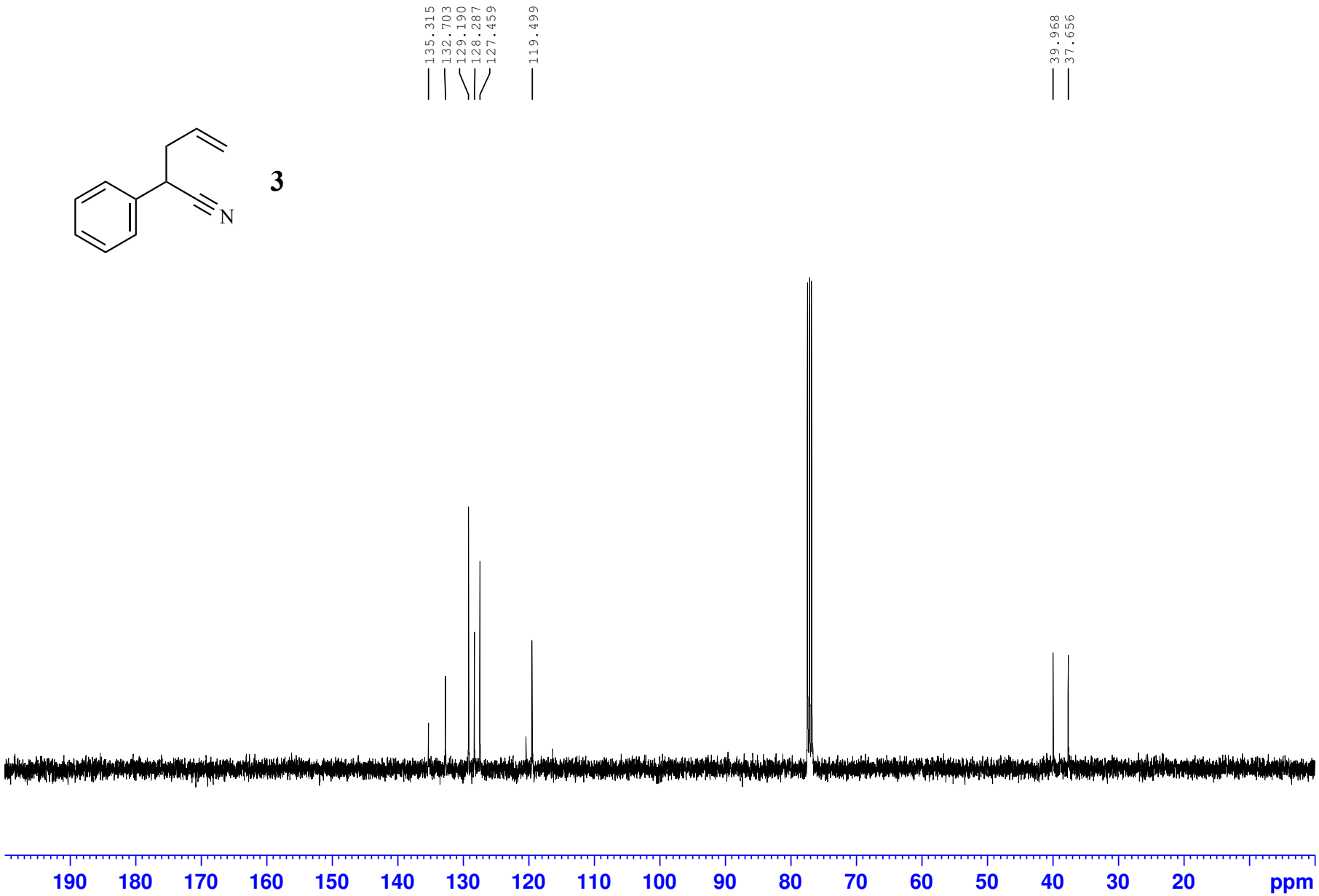
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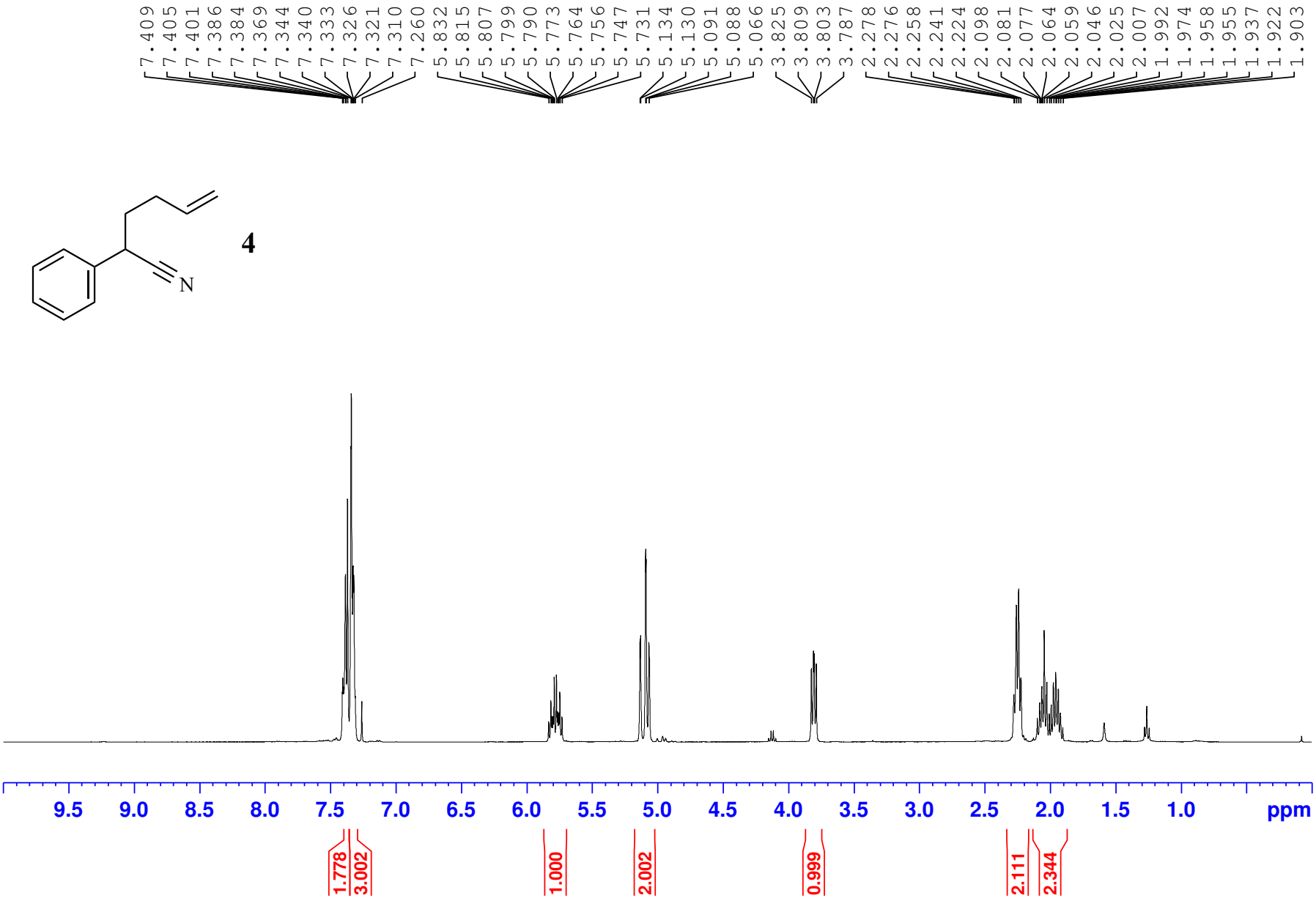
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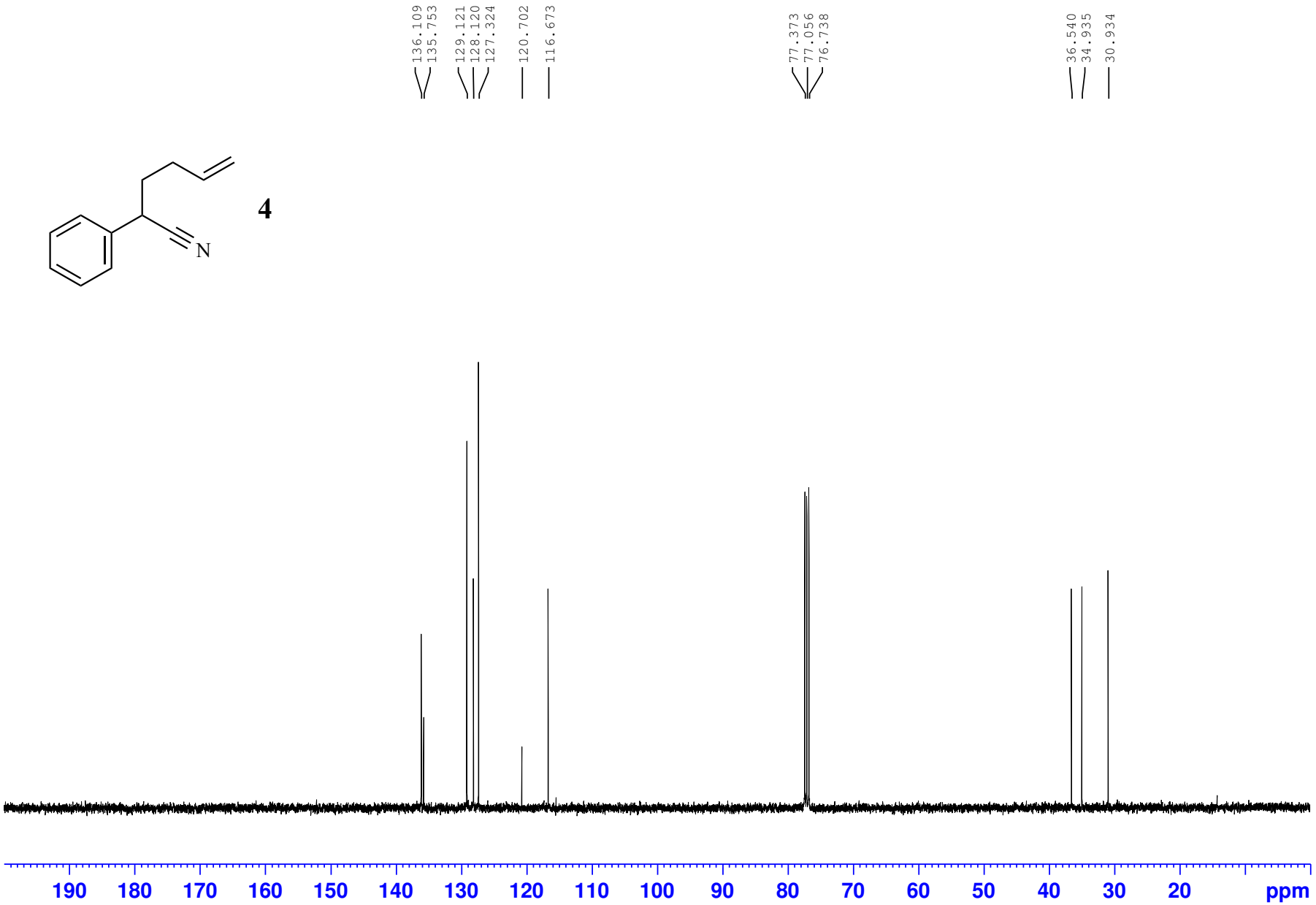
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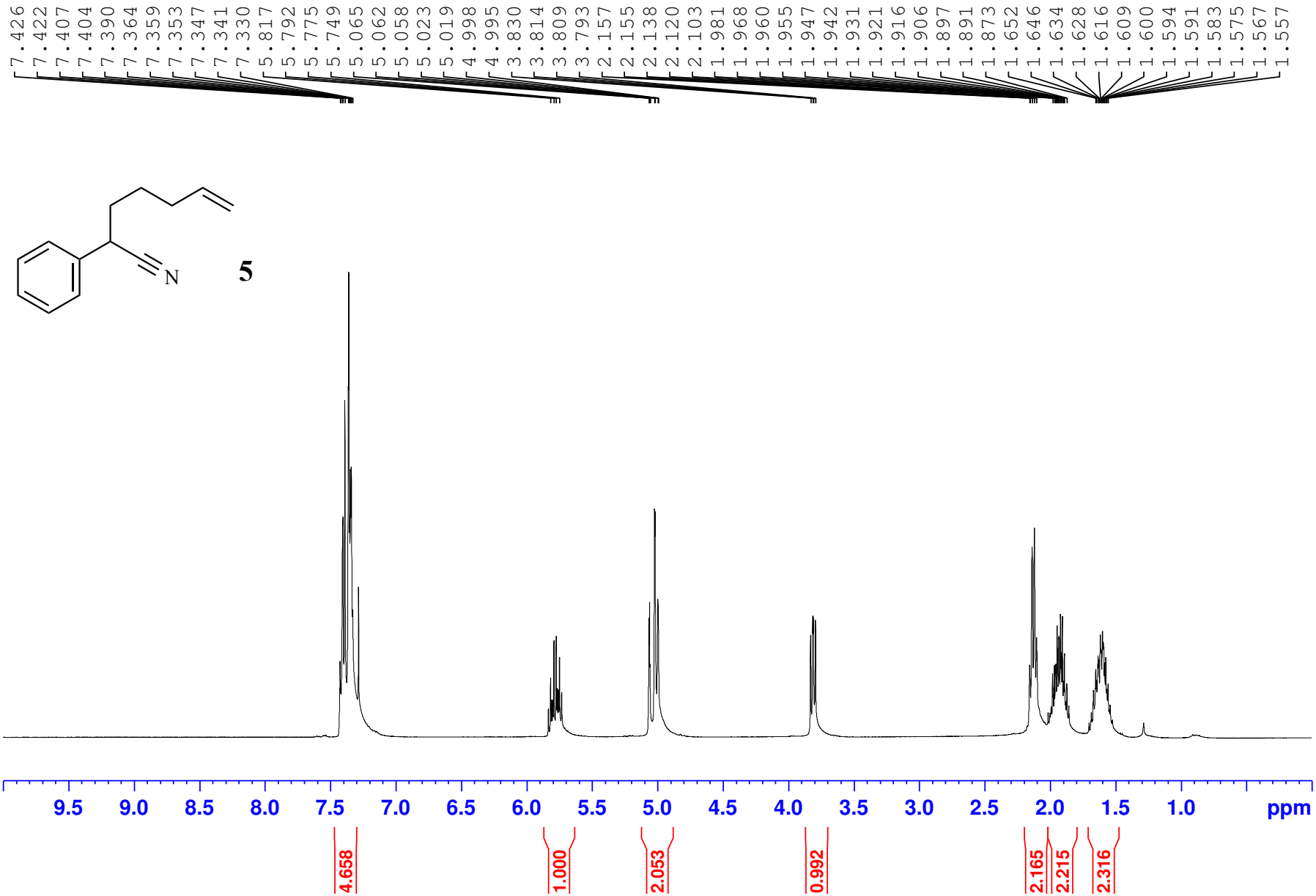
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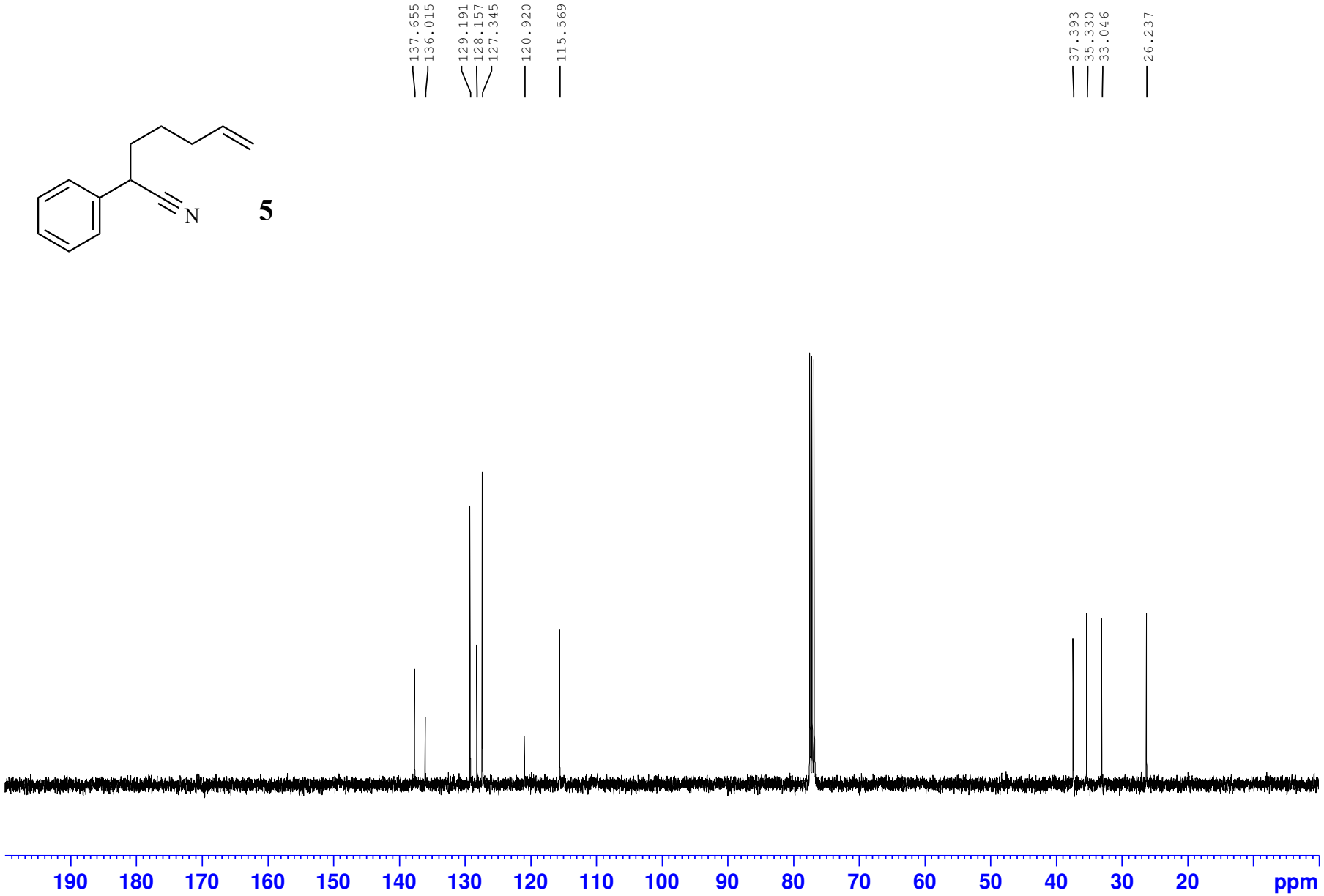
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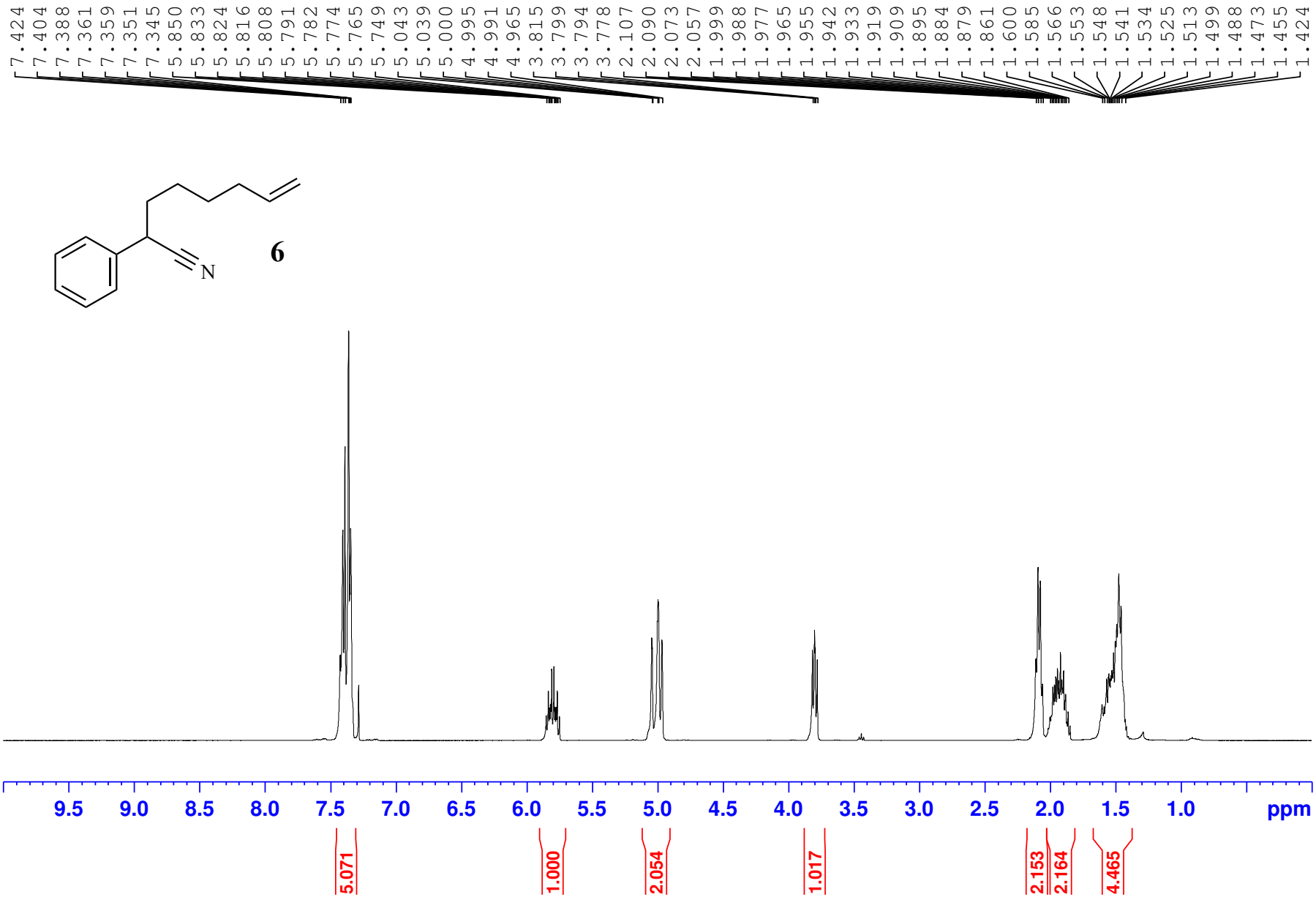
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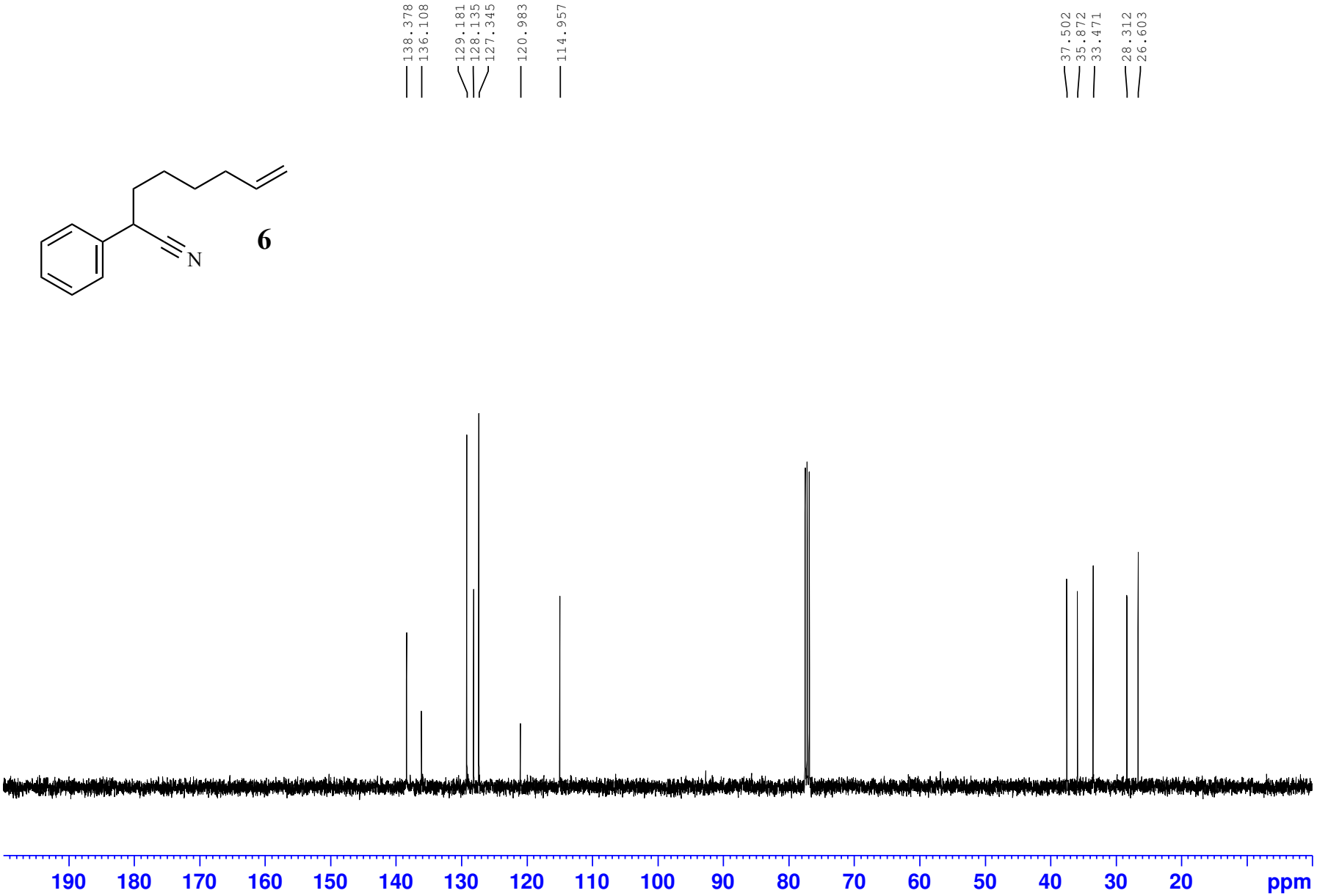
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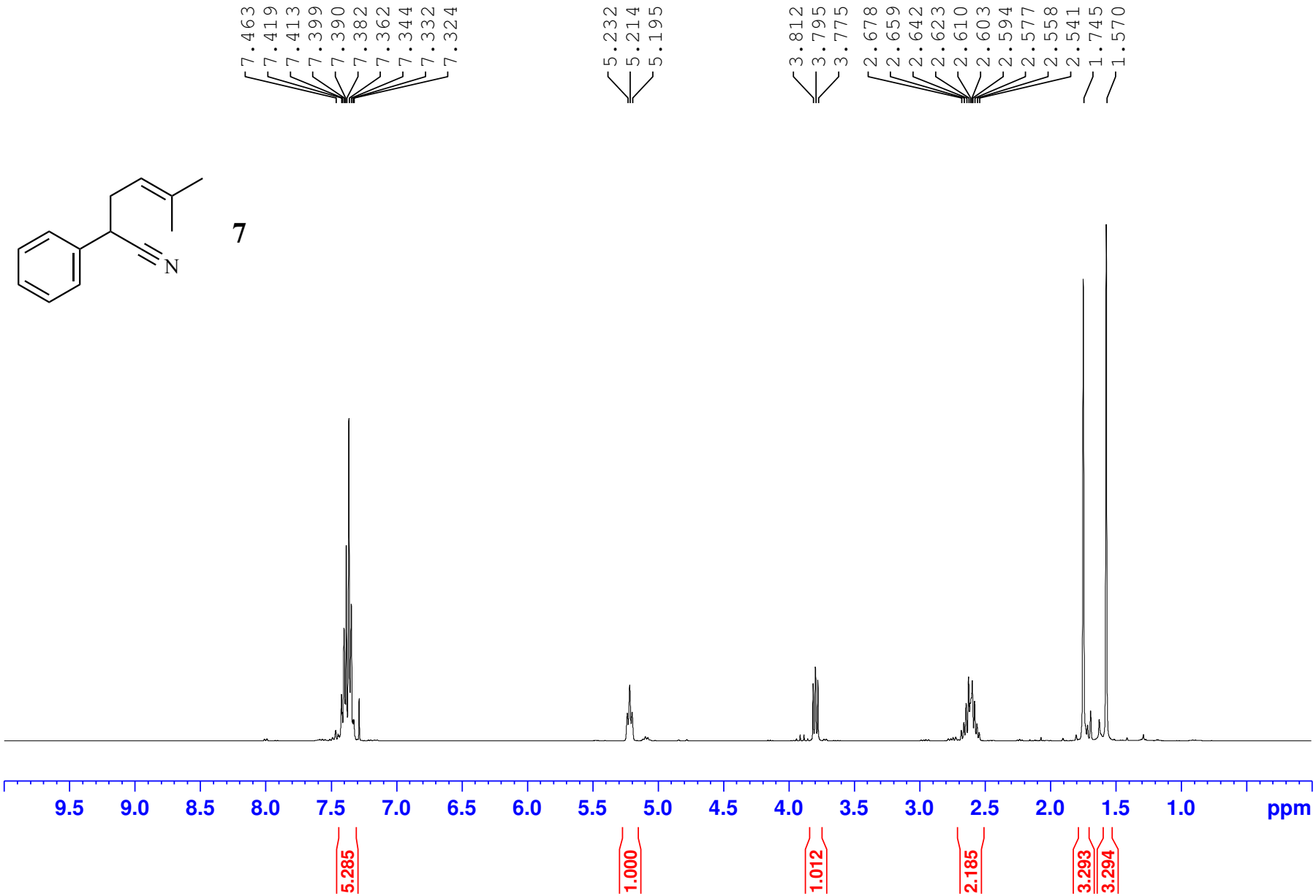
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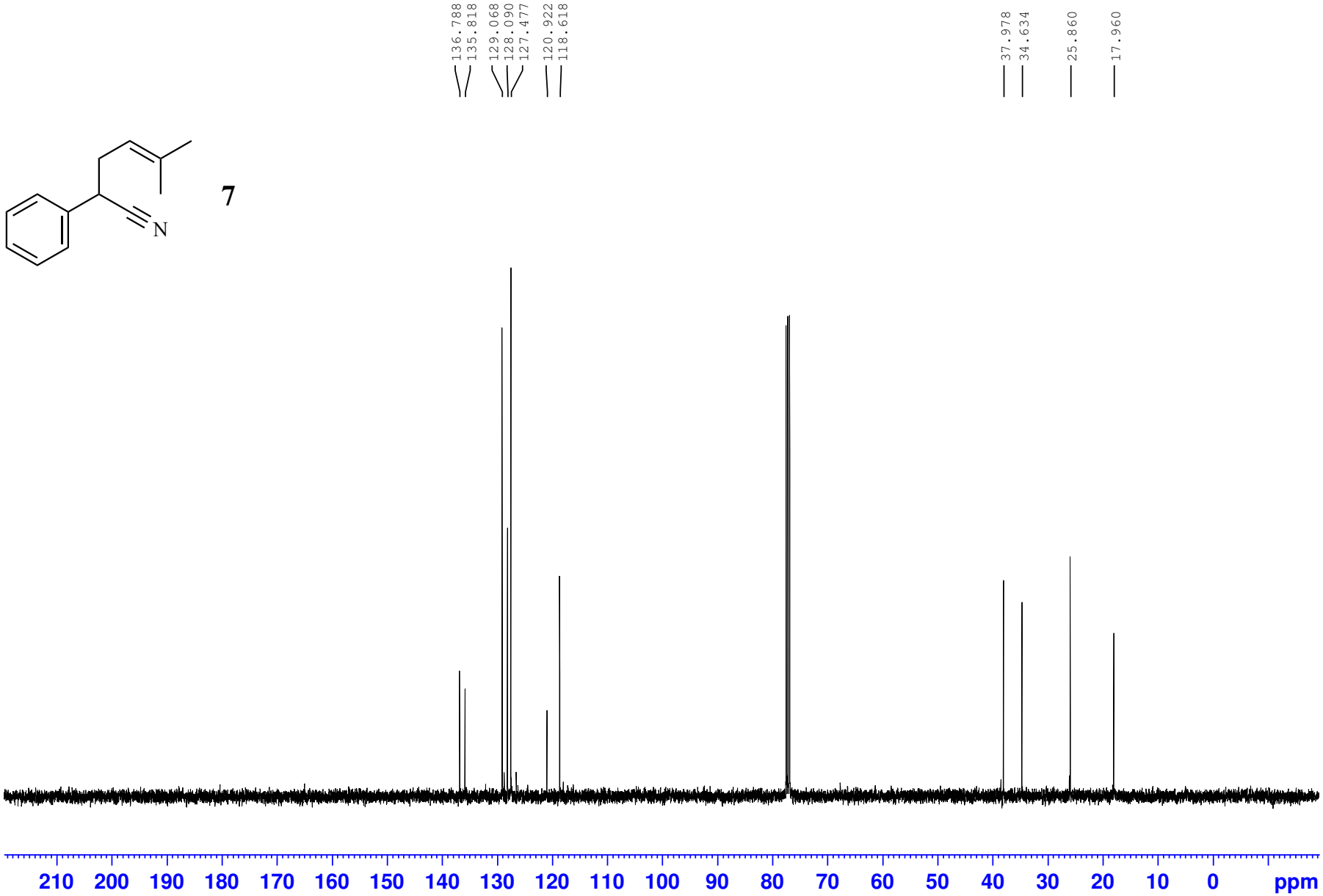
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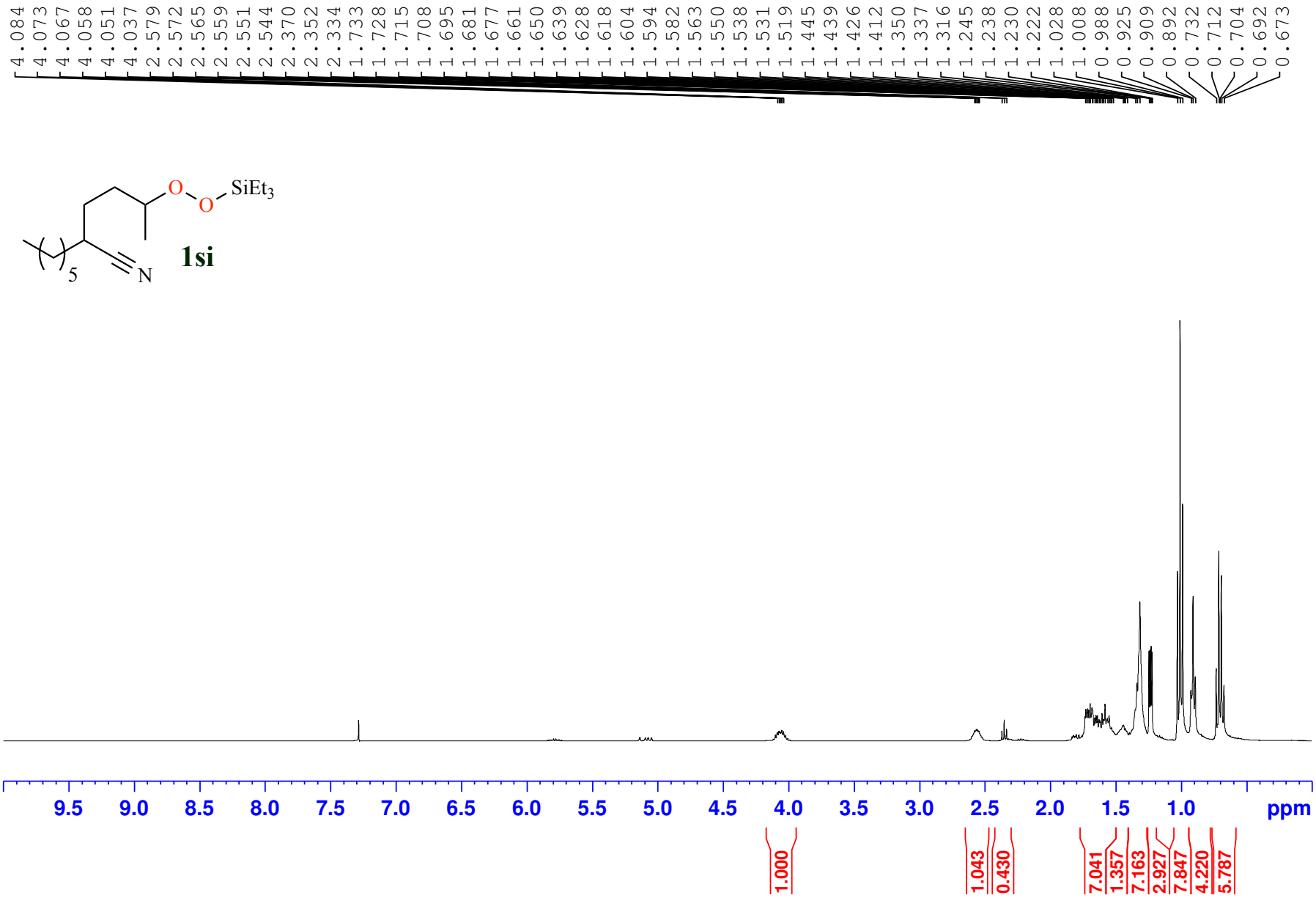
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0.732
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0.704
0.692
0.673
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O
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N
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7.5
7.0
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6.0
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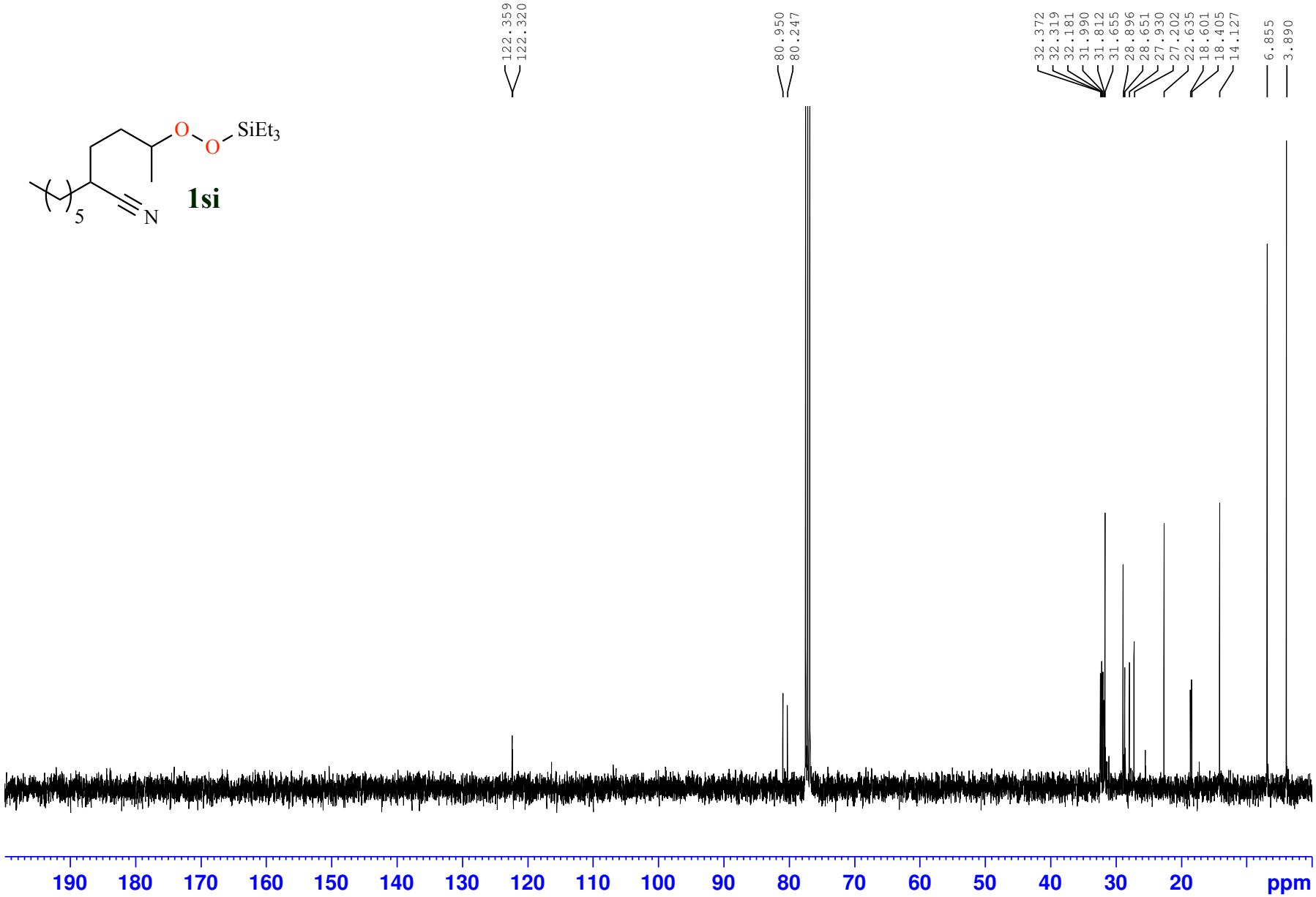
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O
O
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5
N
1si
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ppm

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SiEt3
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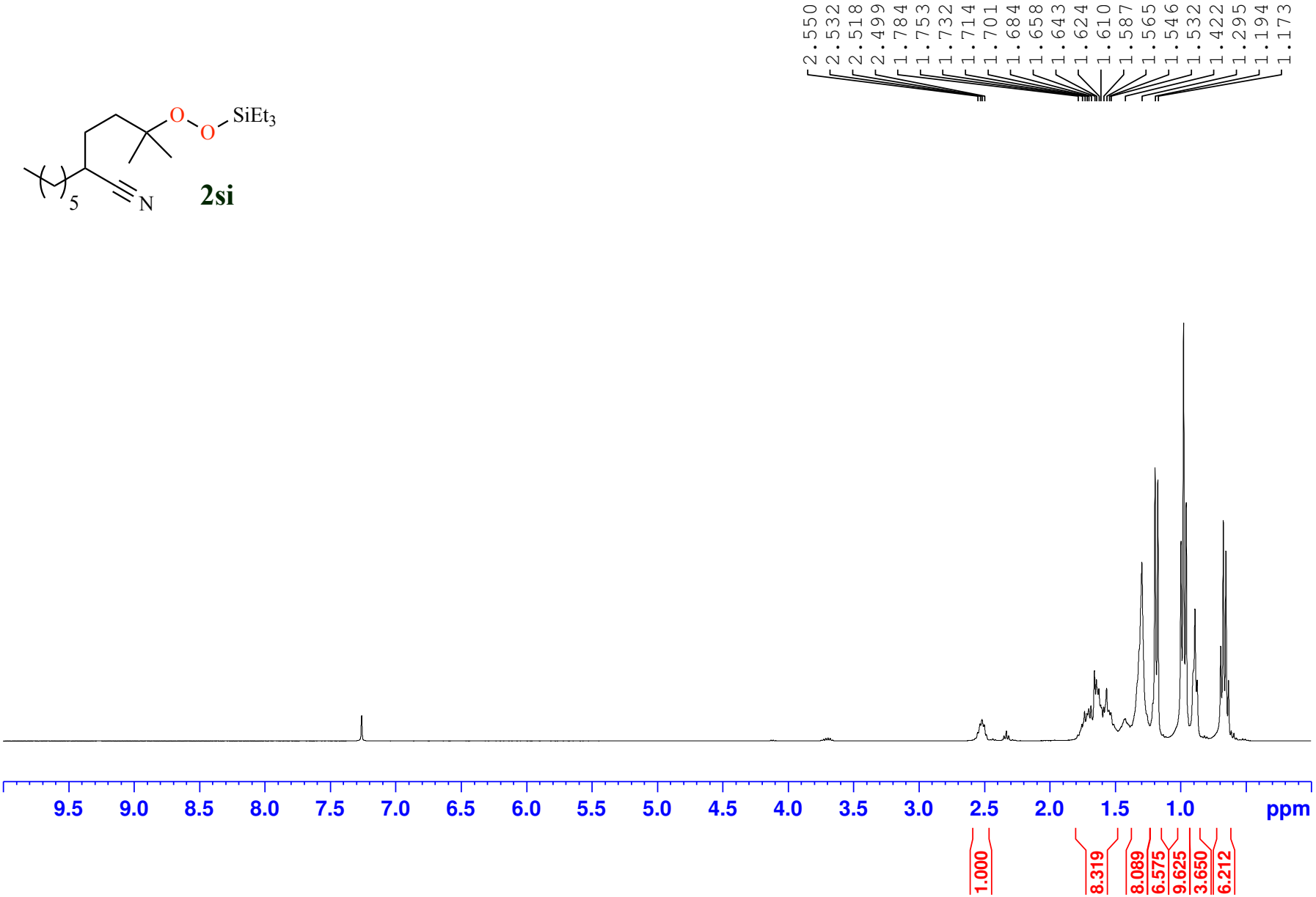
2si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1.610
1.587
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1.546
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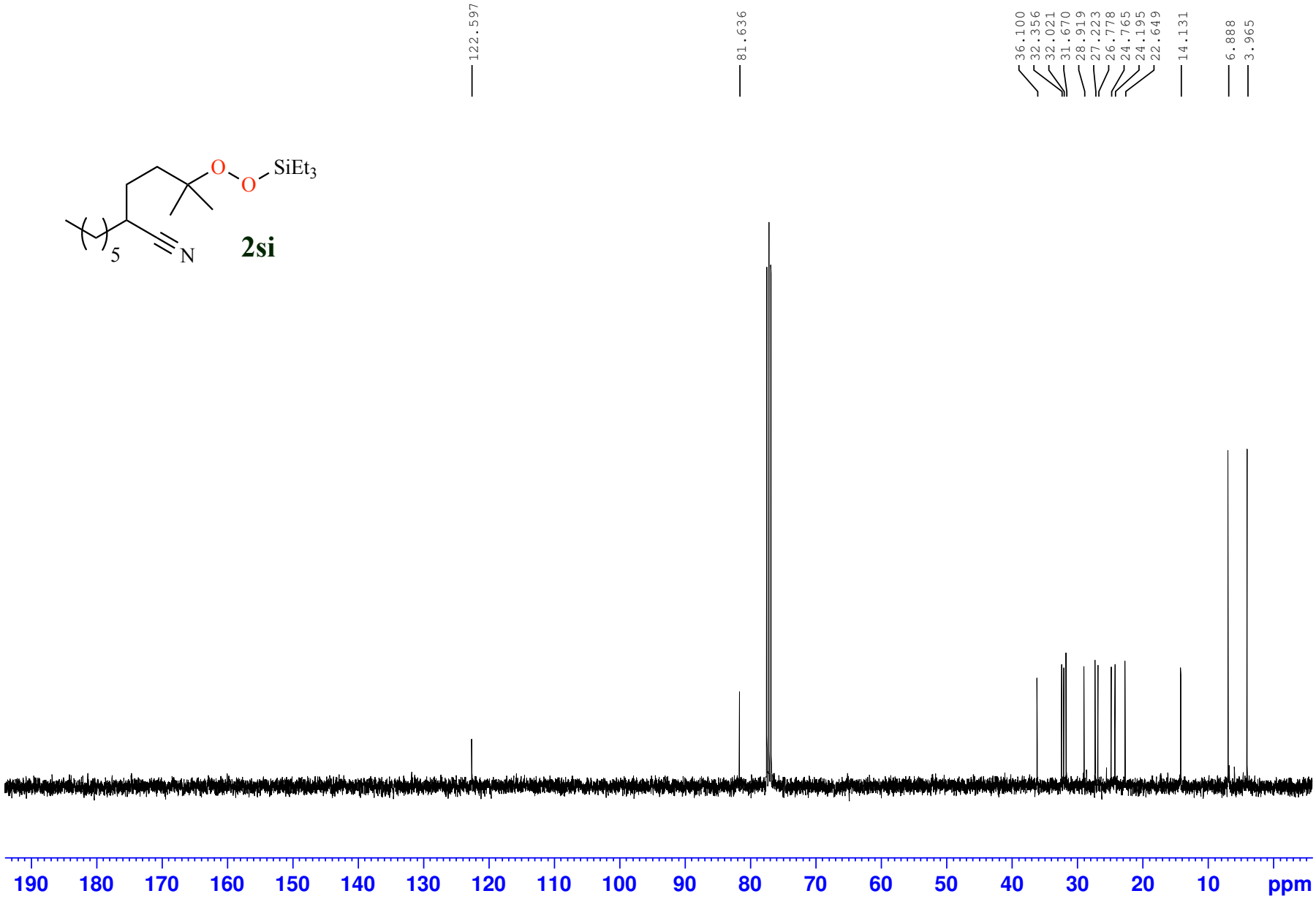
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6.888
3.965
SiEt3
O
O
N
5
2si
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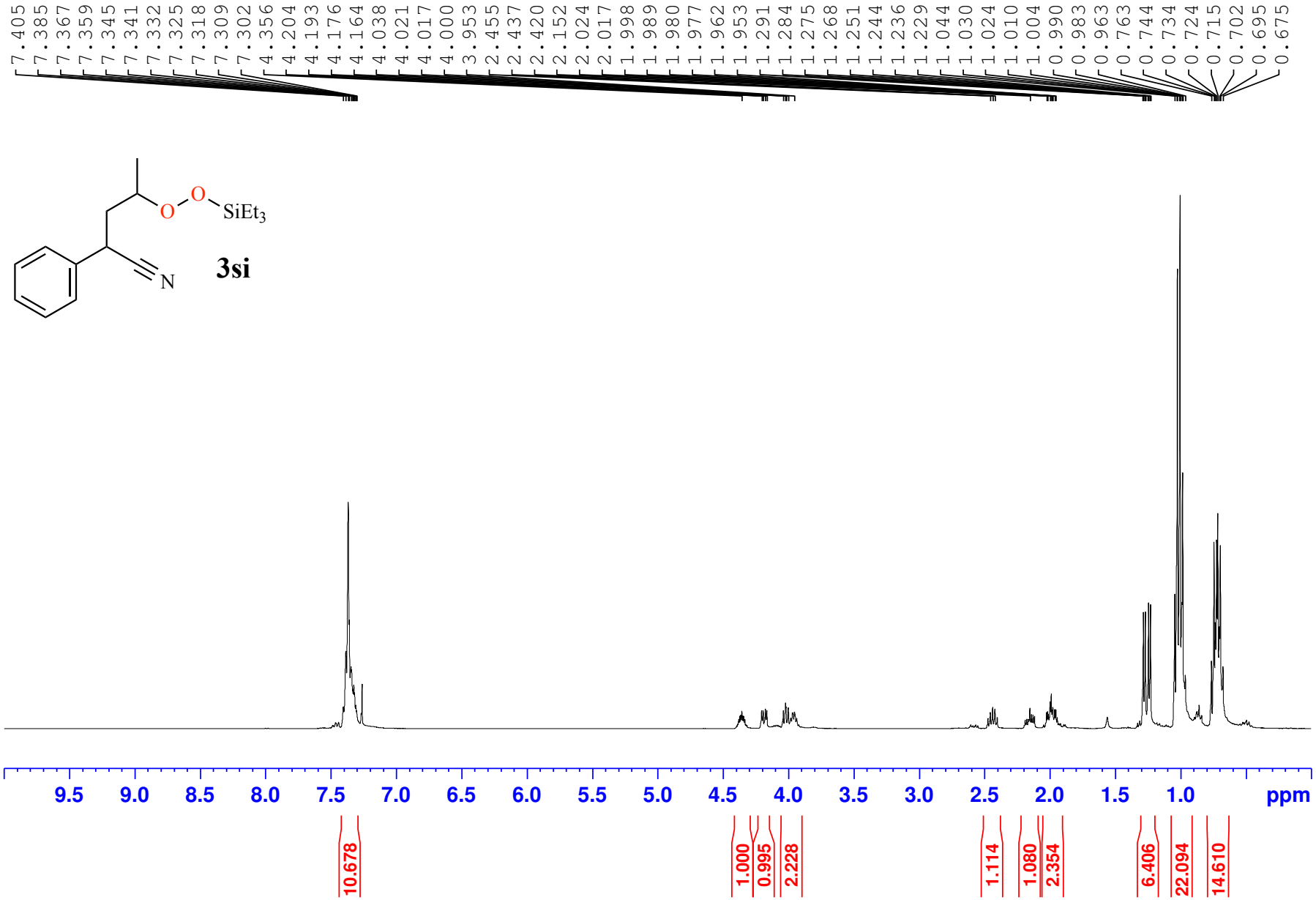
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2.017
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1.229
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0.702
0.695
0.675
O
O
SiEt3
N
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3.5
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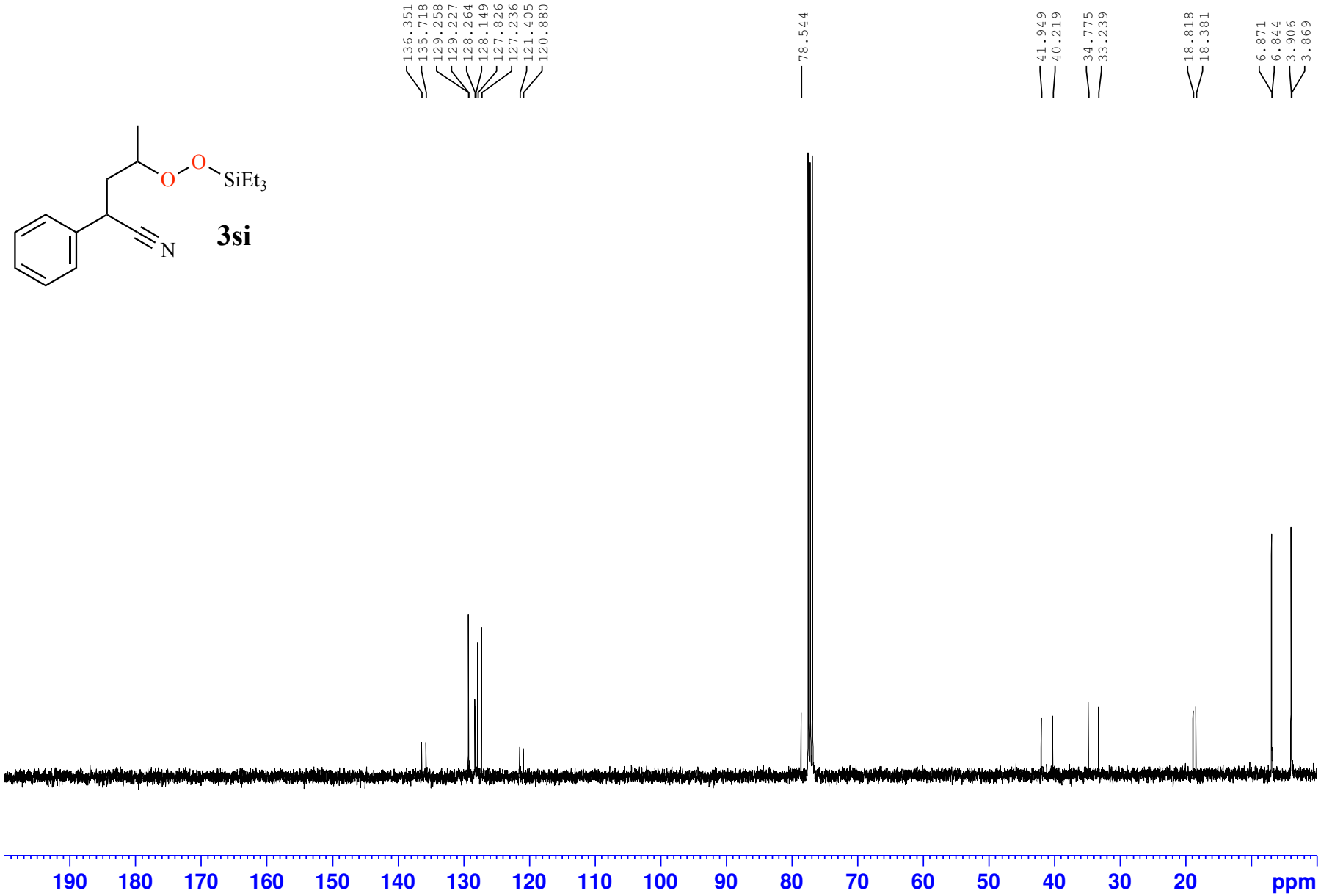
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34.775
33.239
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18.381
6.871
6.844
3.906
3.869
O
O
SiEt3
N
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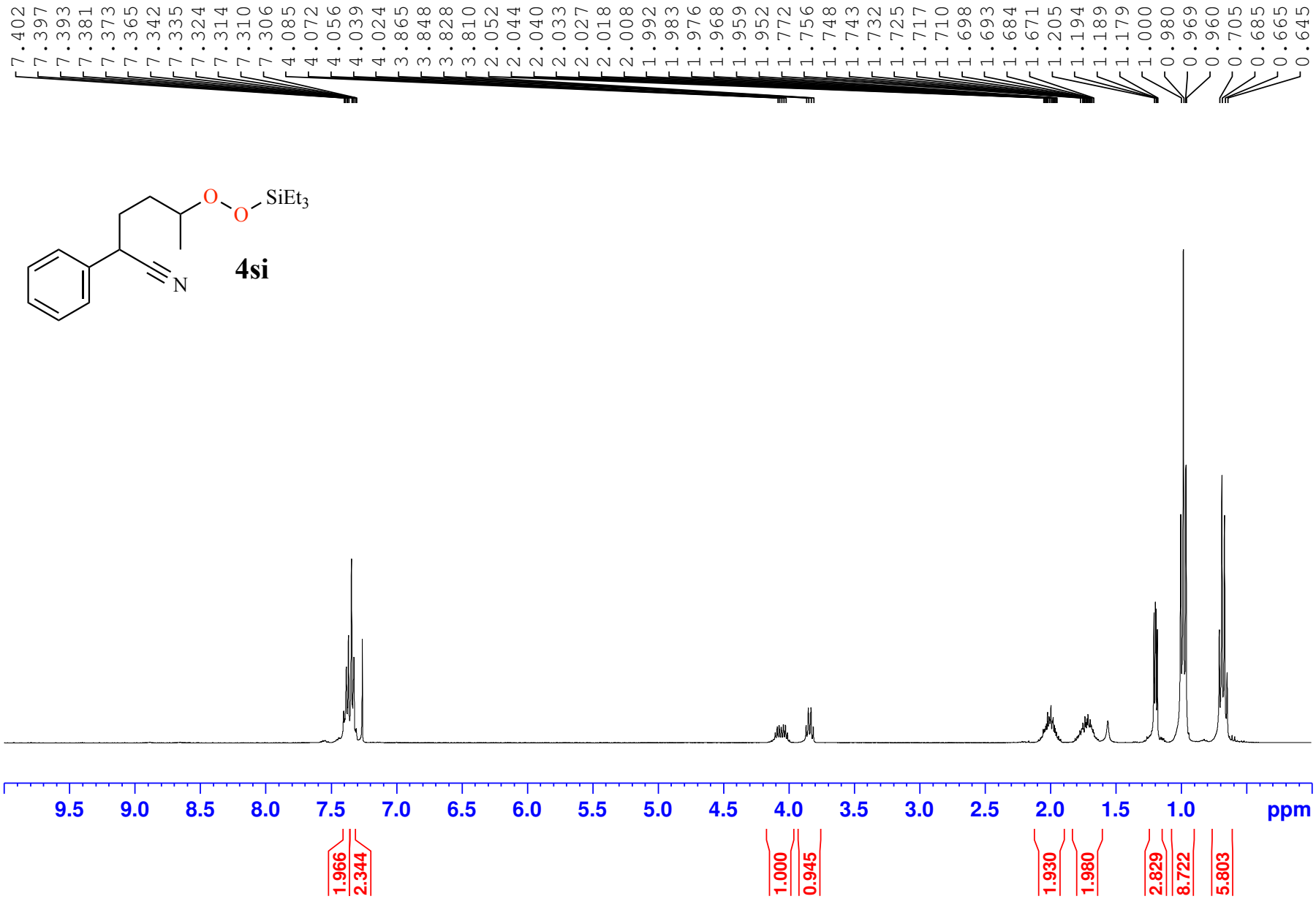
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7.335
7.324
7.314
7.310
7.306
4.085
4.072
4.056
4.039
4.024
3.865
3.848
3.828
3.810
2.052
2.044
2.040
2.033
2.027
2.018
2.008
1.992
1.983
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1.968
1.959
1.952
1.772
1.756
1.748
1.743
1.732
1.725
1.717
1.710
1.698
1.693
1.684
1.671
1.205
1.194
1.189
1.179
1.000
0.980
0.969
0.960
0.705
0.685
0.665
0.645
O
O
SiEt3
N
4si
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8.5
8.0
7.5
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4.0
3.5
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2.0
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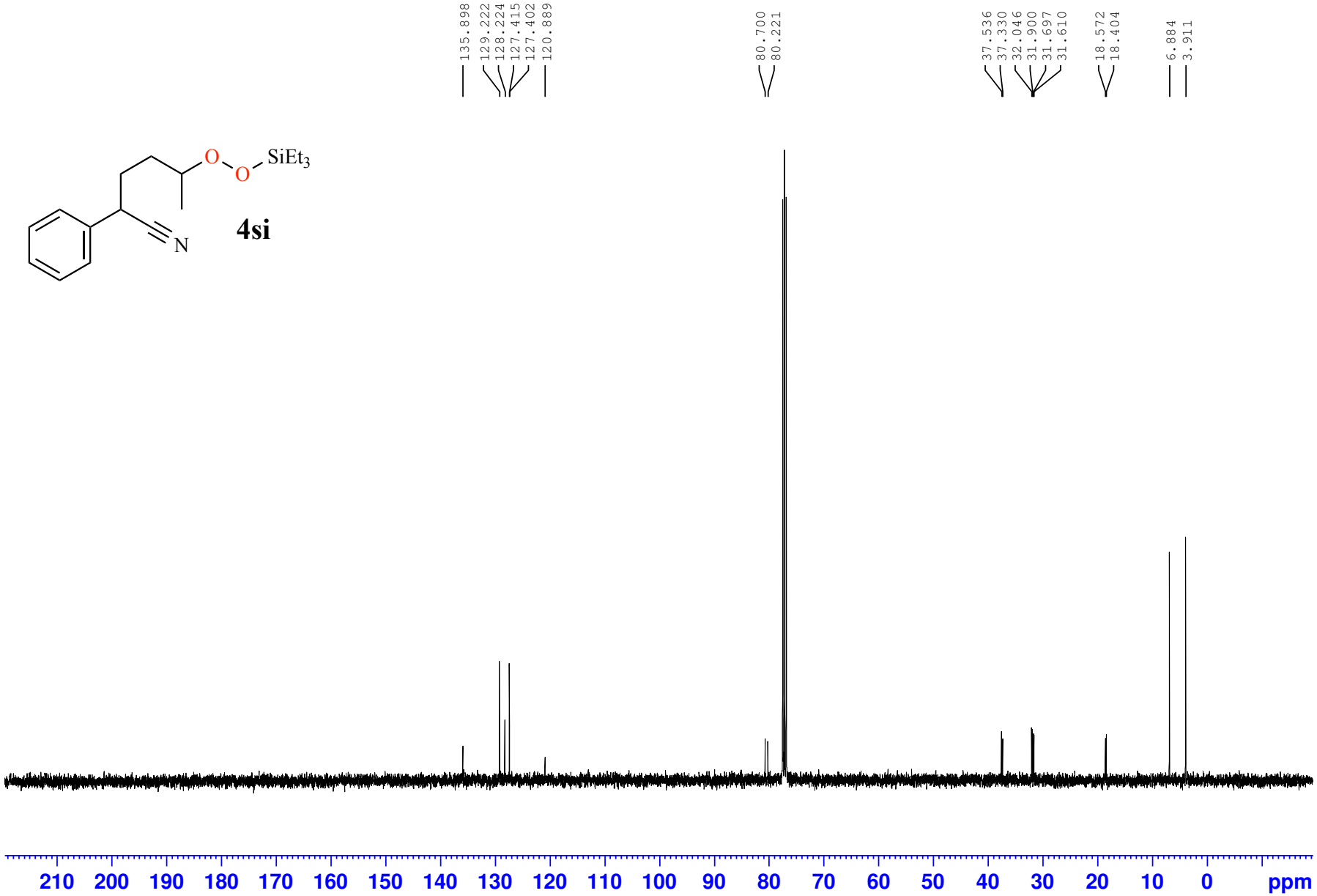
5.803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80.221
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31.697
31.610
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O
SiEt3
N
4si
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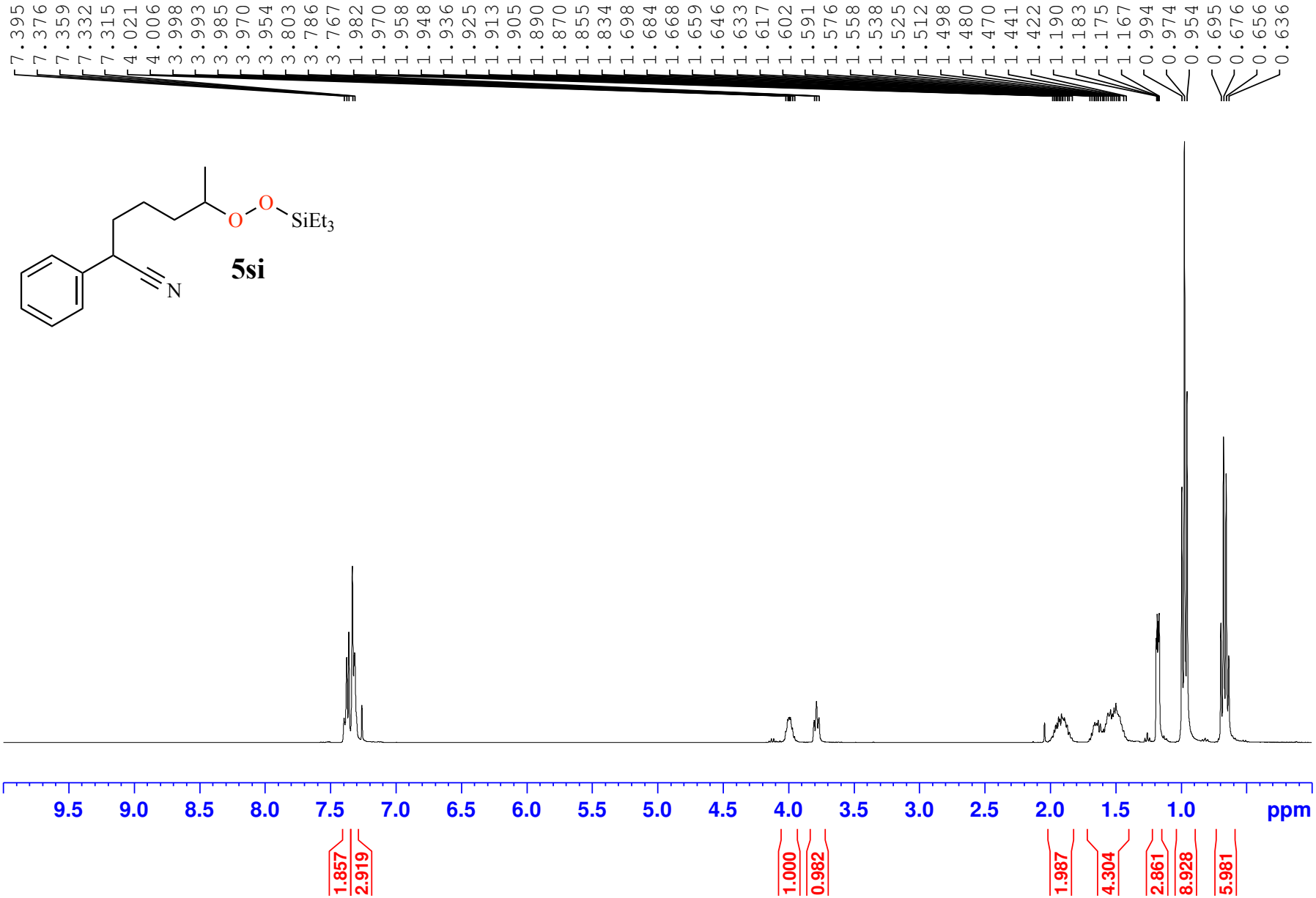
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3.767
1.982
1.970
1.958
1.948
1.936
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1.913
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1.659
1.646
1.633
1.617
1.602
1.591
1.576
1.558
1.538
1.525
1.512
1.498
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1.470
1.441
1.422
1.190
1.183
1.175
1.167
0.994
0.974
0.954
0.695
0.676
0.656
0.636
O
O
SiEt3
N
5si
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8.5
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7.5
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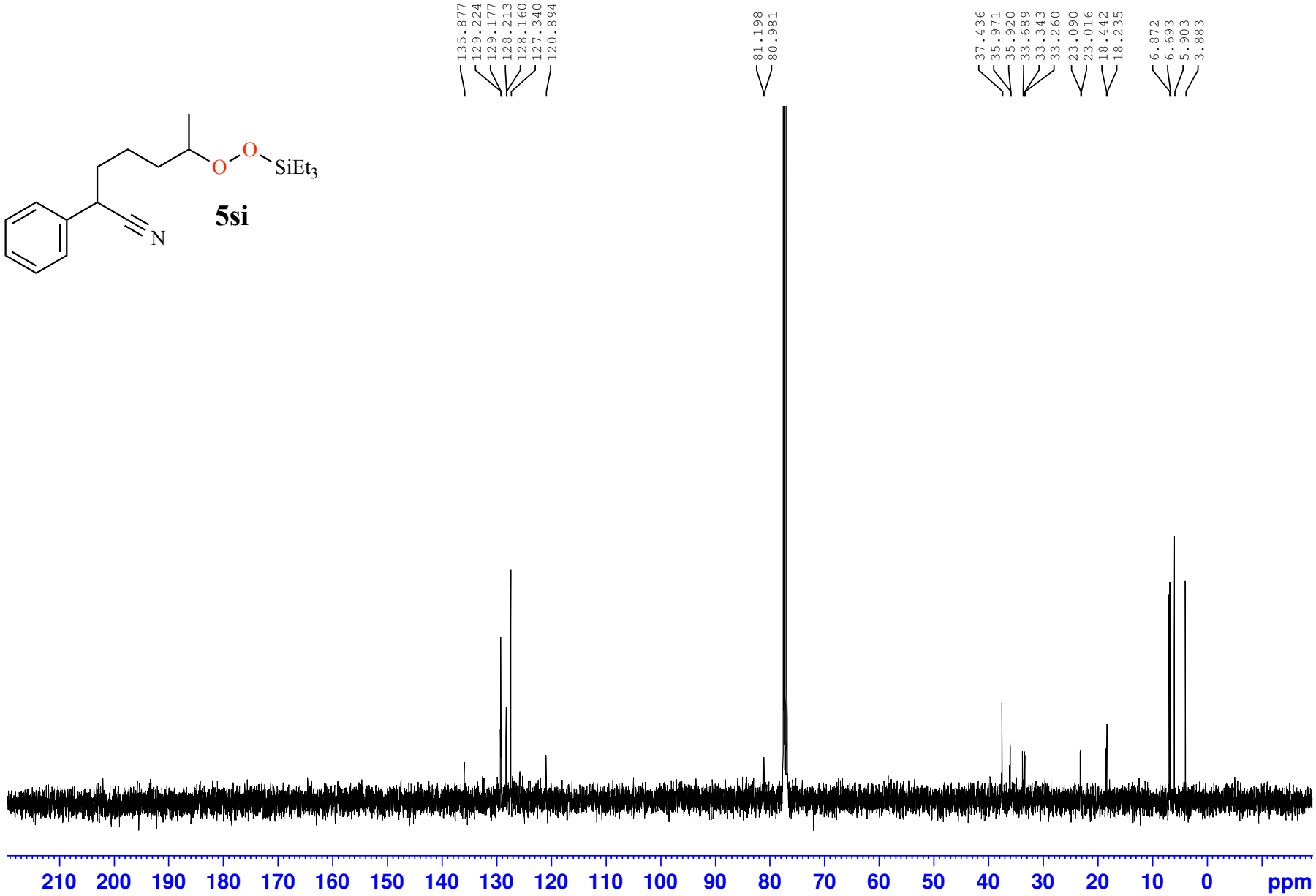
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O
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35.971
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33.260
23.090
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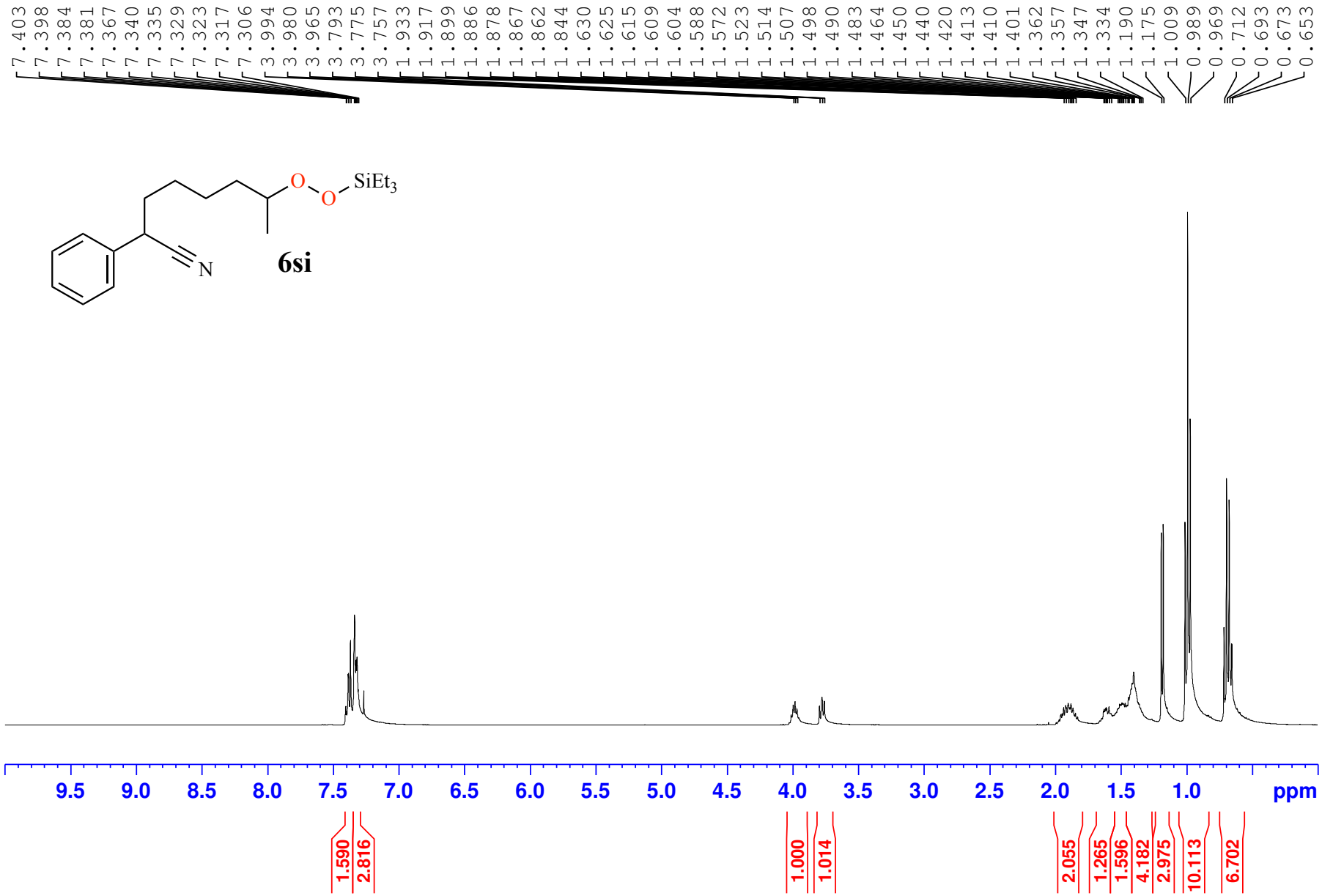
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3.775
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1.609
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1.523
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1.347
1.334
1.190
1.175
1.009
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0.969
0.712
0.693
0.673
0.653
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O
SiEt3
N
6si
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7.0
6.5
6.0
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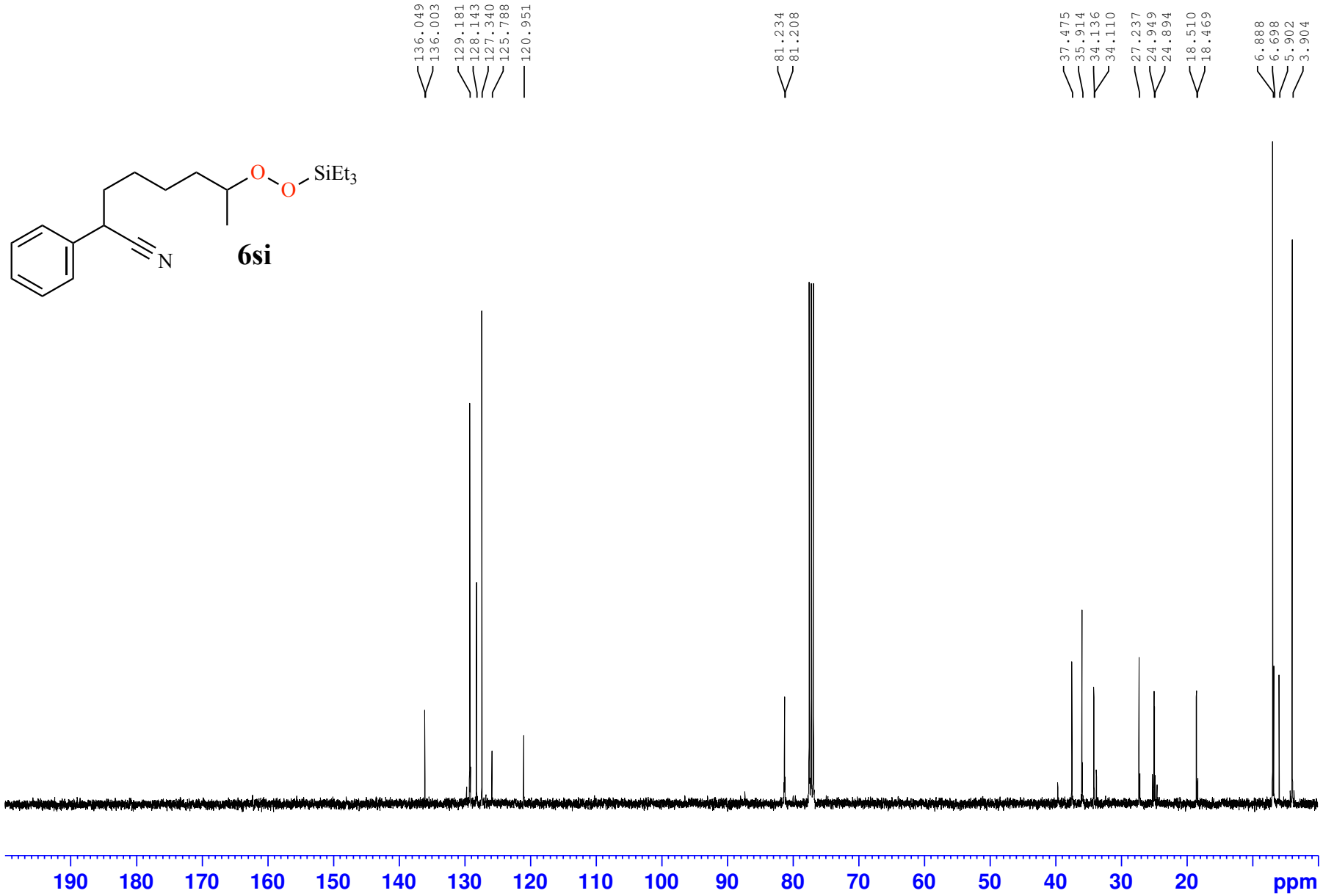
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27.237
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24.894
18.510
18.469
6.888
6.698
5.902
3.904
O
O
SiEt3
N
6si
190
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160
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90
80
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20
ppm

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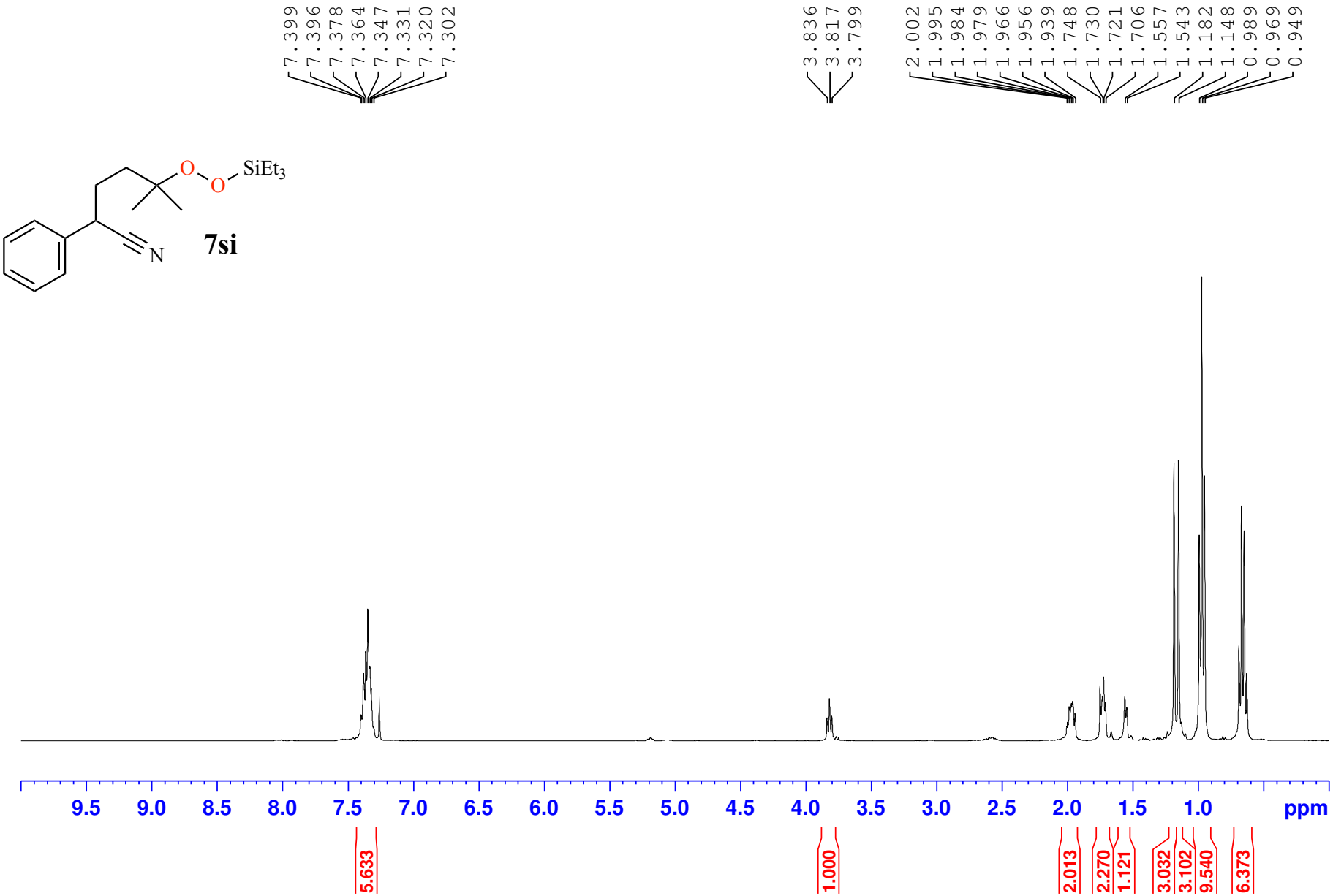
SiEt3
N

7si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1.984
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1.966
1.956
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7.0
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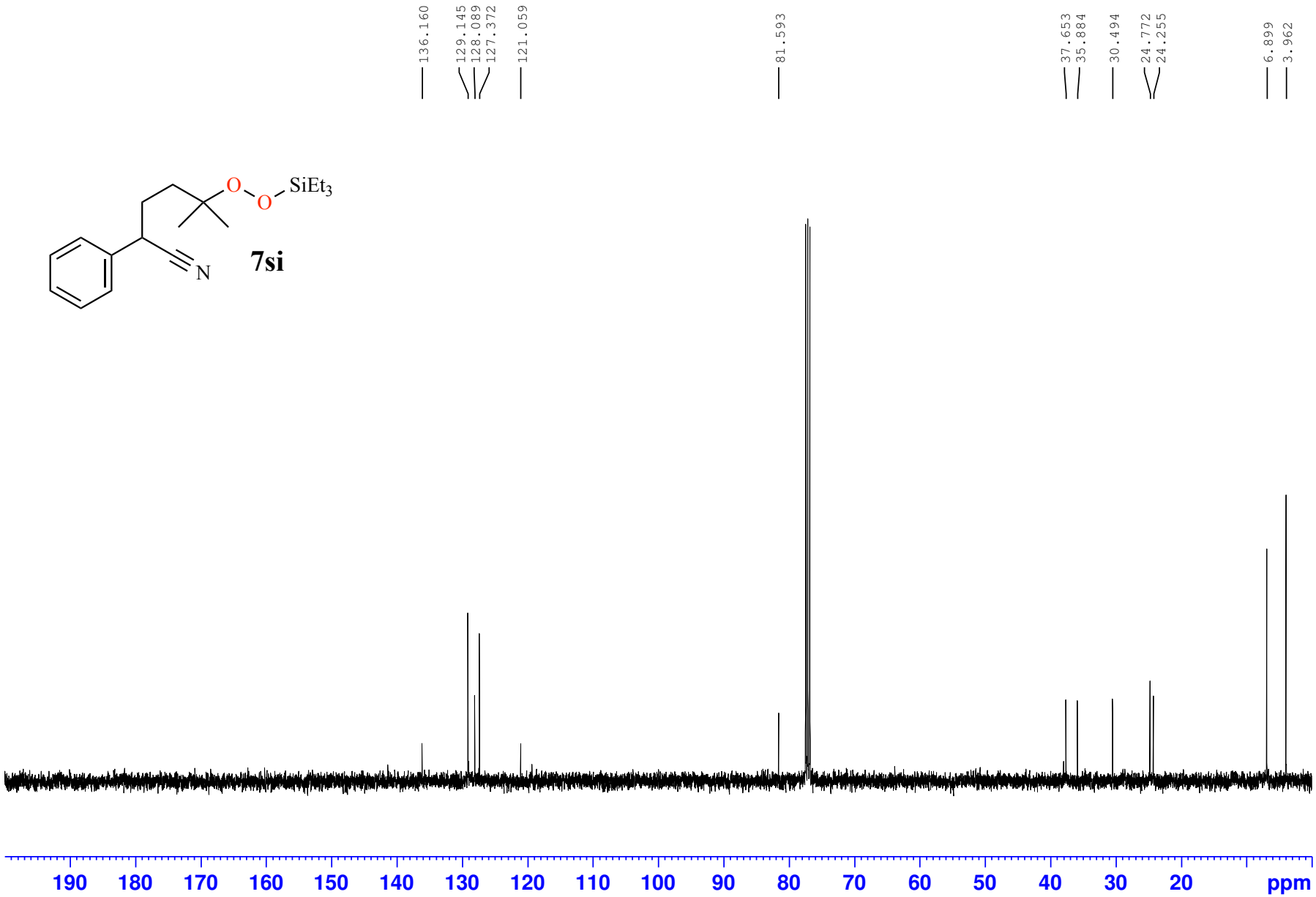
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O
SiEt3
N
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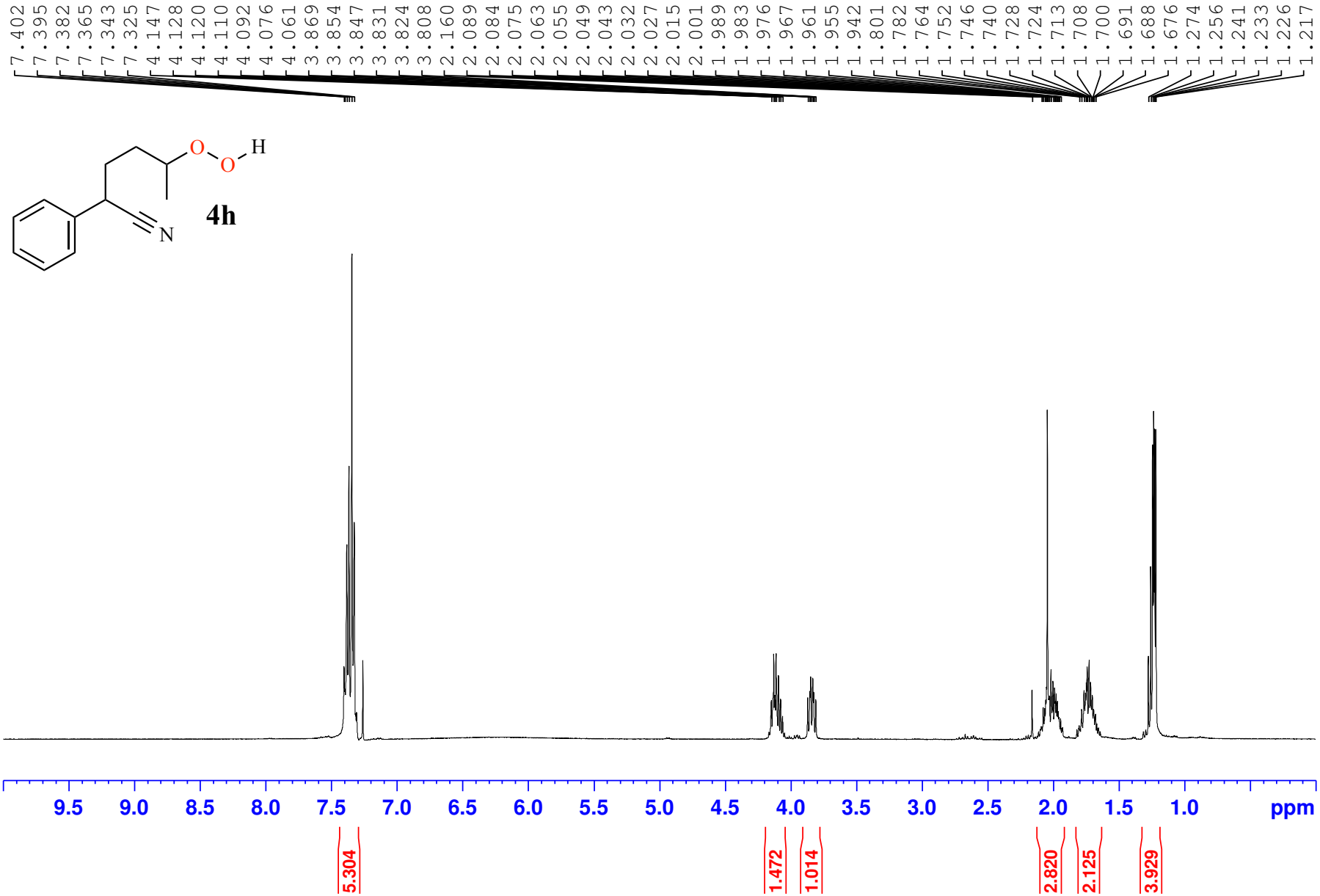
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N
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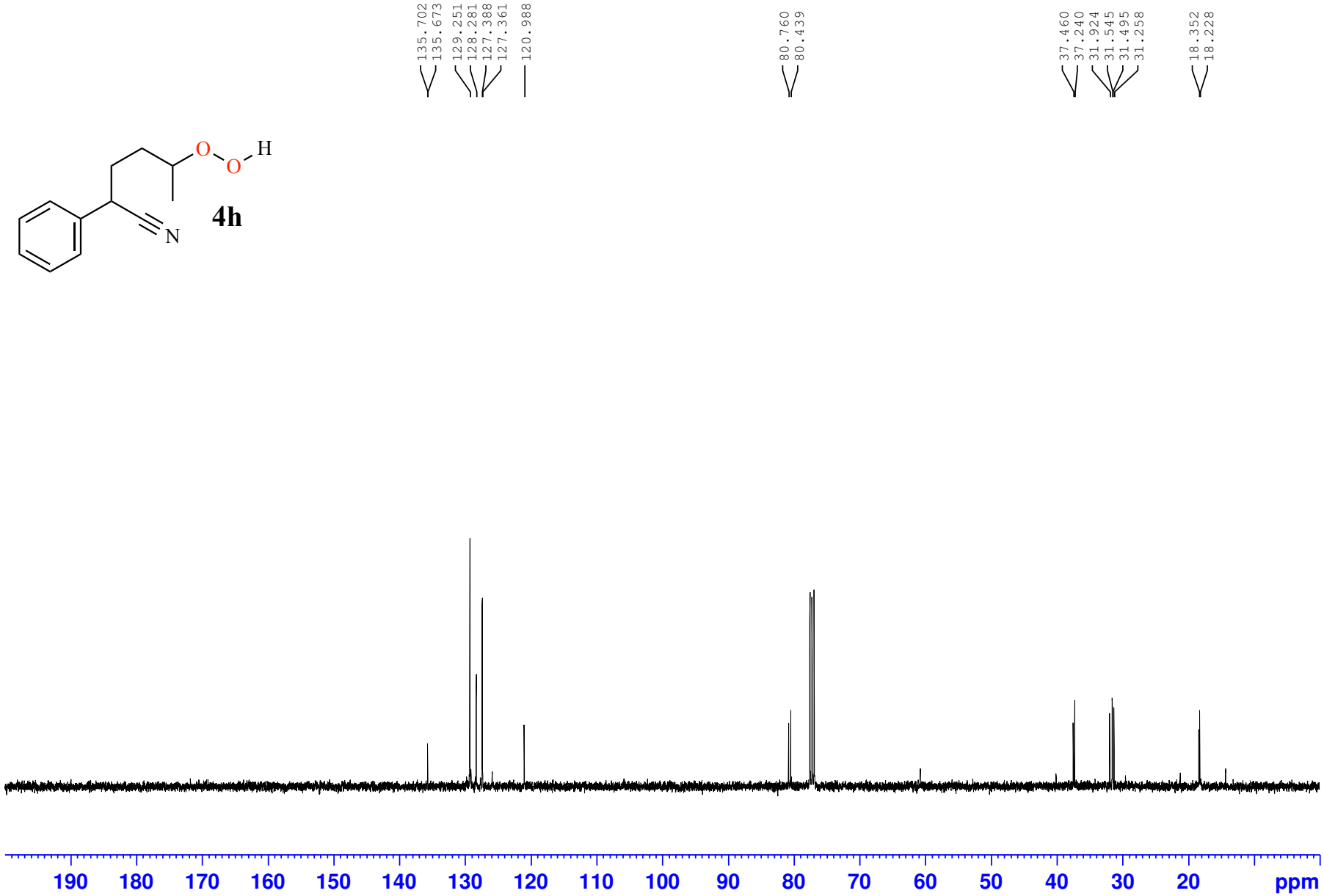
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O
H
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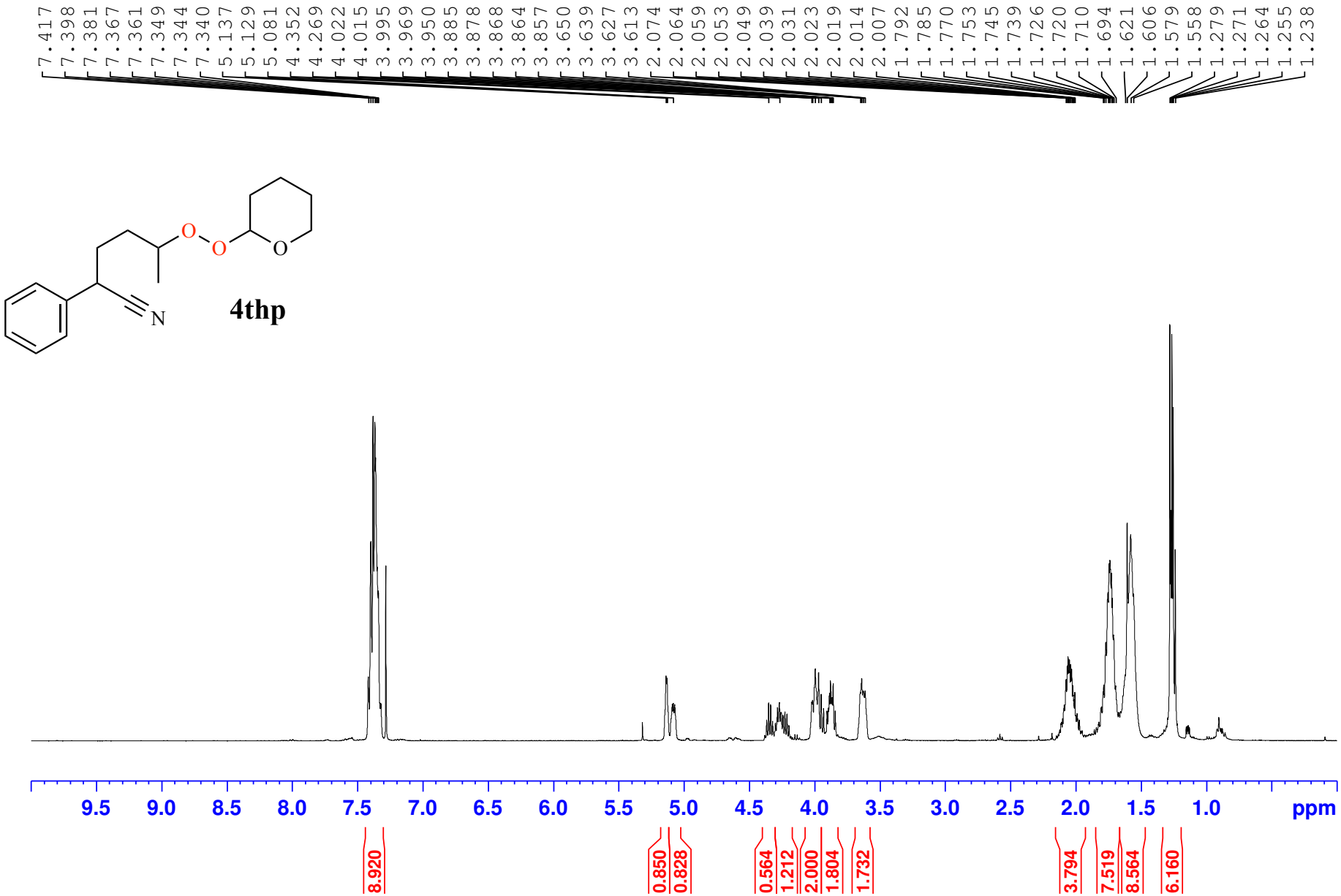
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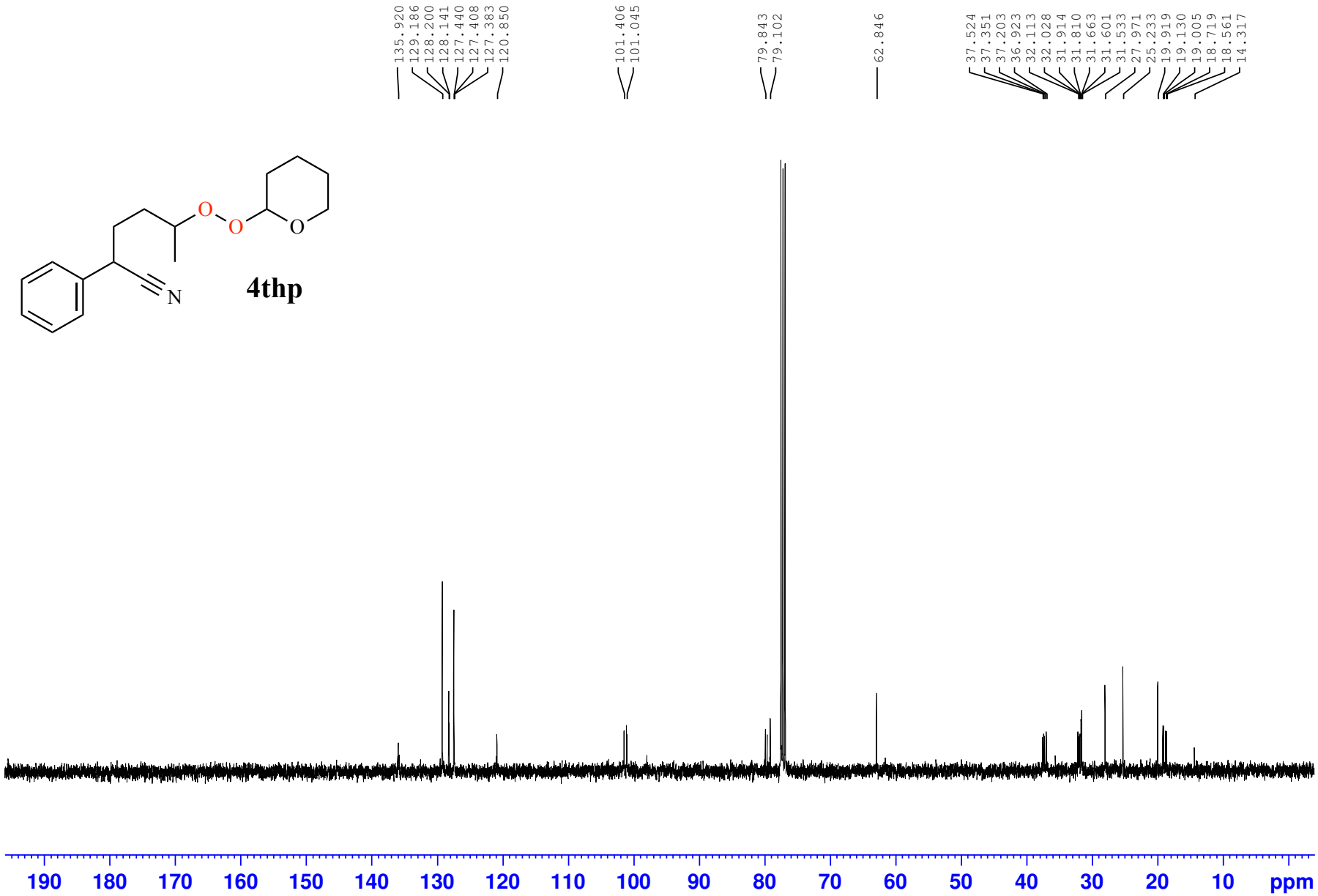
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37.351
37.203
36.923
32.113
32.028
31.914
31.810
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31.533
27.971
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19.130
19.005
18.719
18.561
14.317
O
O
O
N
4thp
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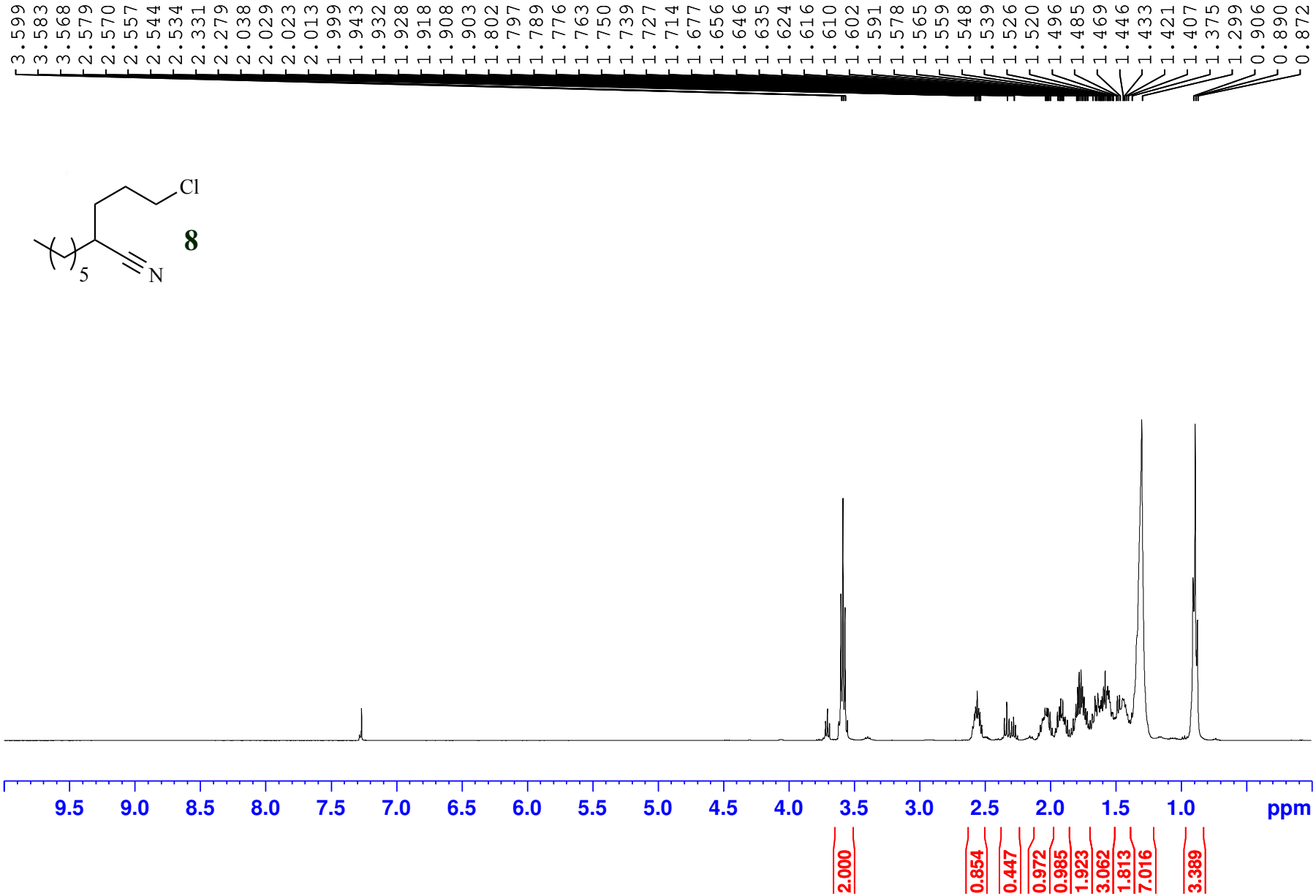
10
ppm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2.0
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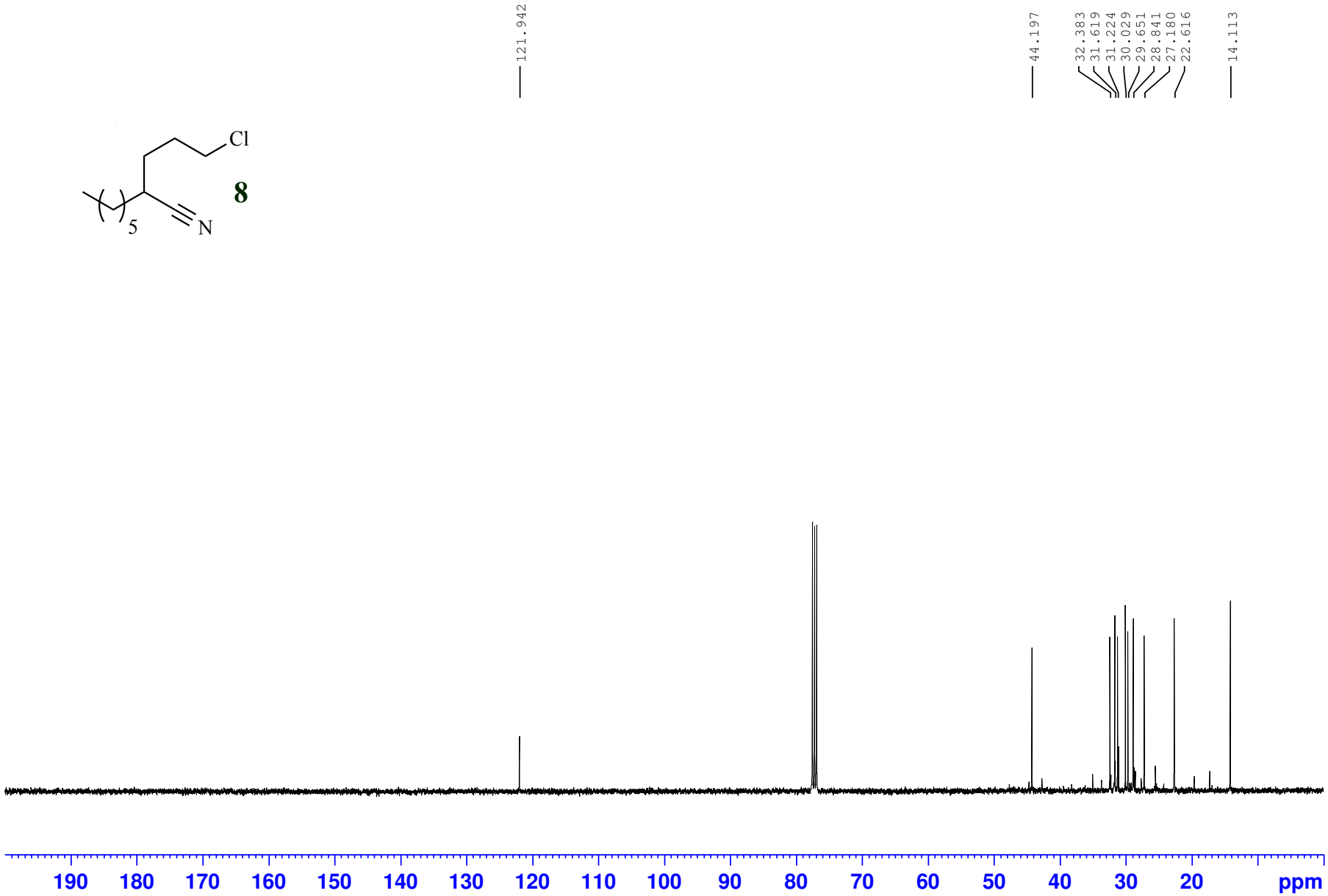
7.016
3.389

Cl
5
N

8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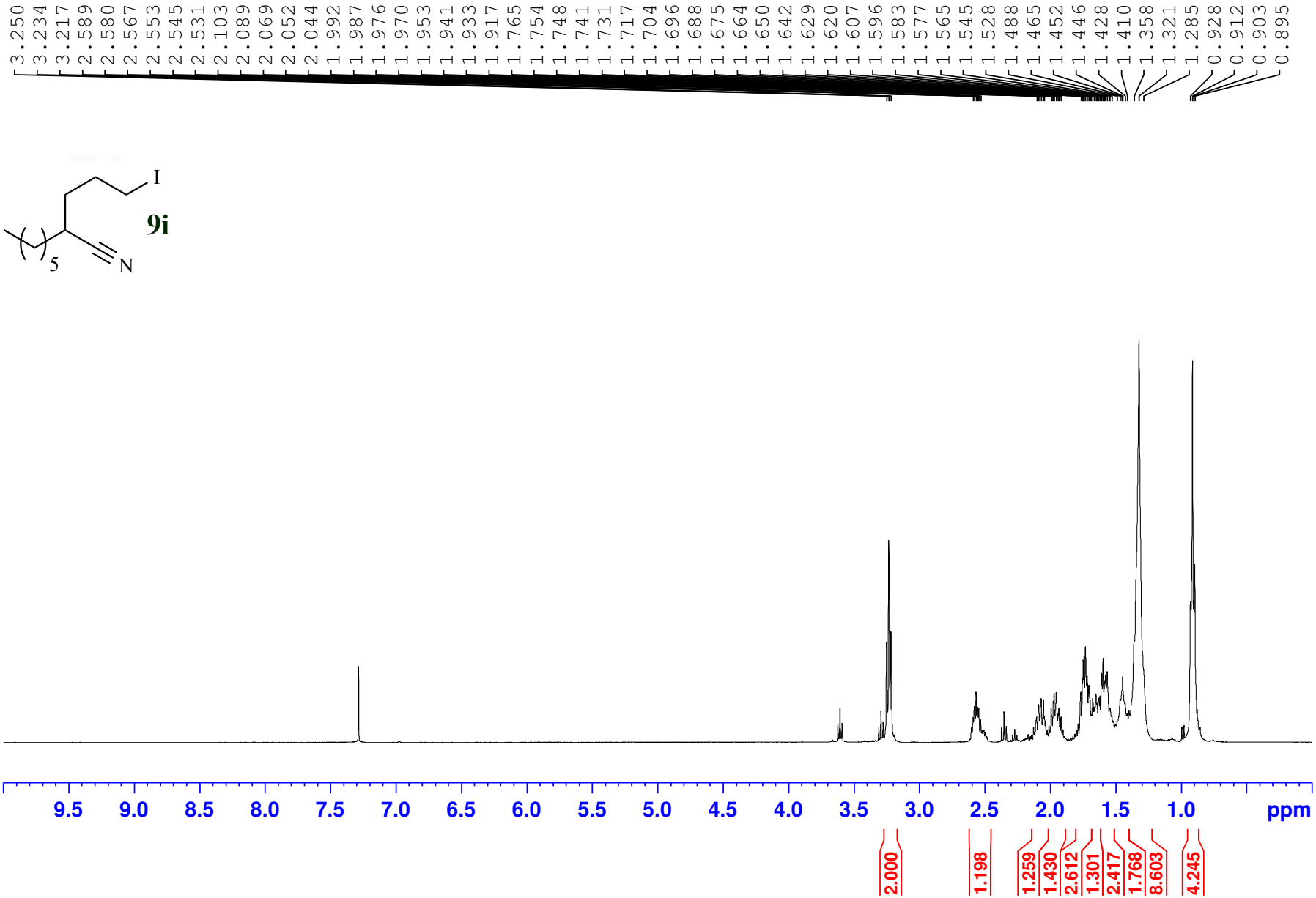
20
ppm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2.553
2.545
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2.103
2.089
2.069
2.052
2.044
1.992
1.987
1.976
1.970
1.953
1.941
1.933
1.917
1.765
1.754
1.748
1.741
1.731
1.717
1.704
1.696
1.688
1.675
1.664
1.650
1.642
1.629
1.620
1.607
1.596
1.583
1.577
1.565
1.545
1.528
1.488
1.465
1.452
1.446
1.428
1.410
1.358
1.321
1.285
0.928
0.912
0.903
0.895
I
9i
5
N
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
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1.430
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1.301
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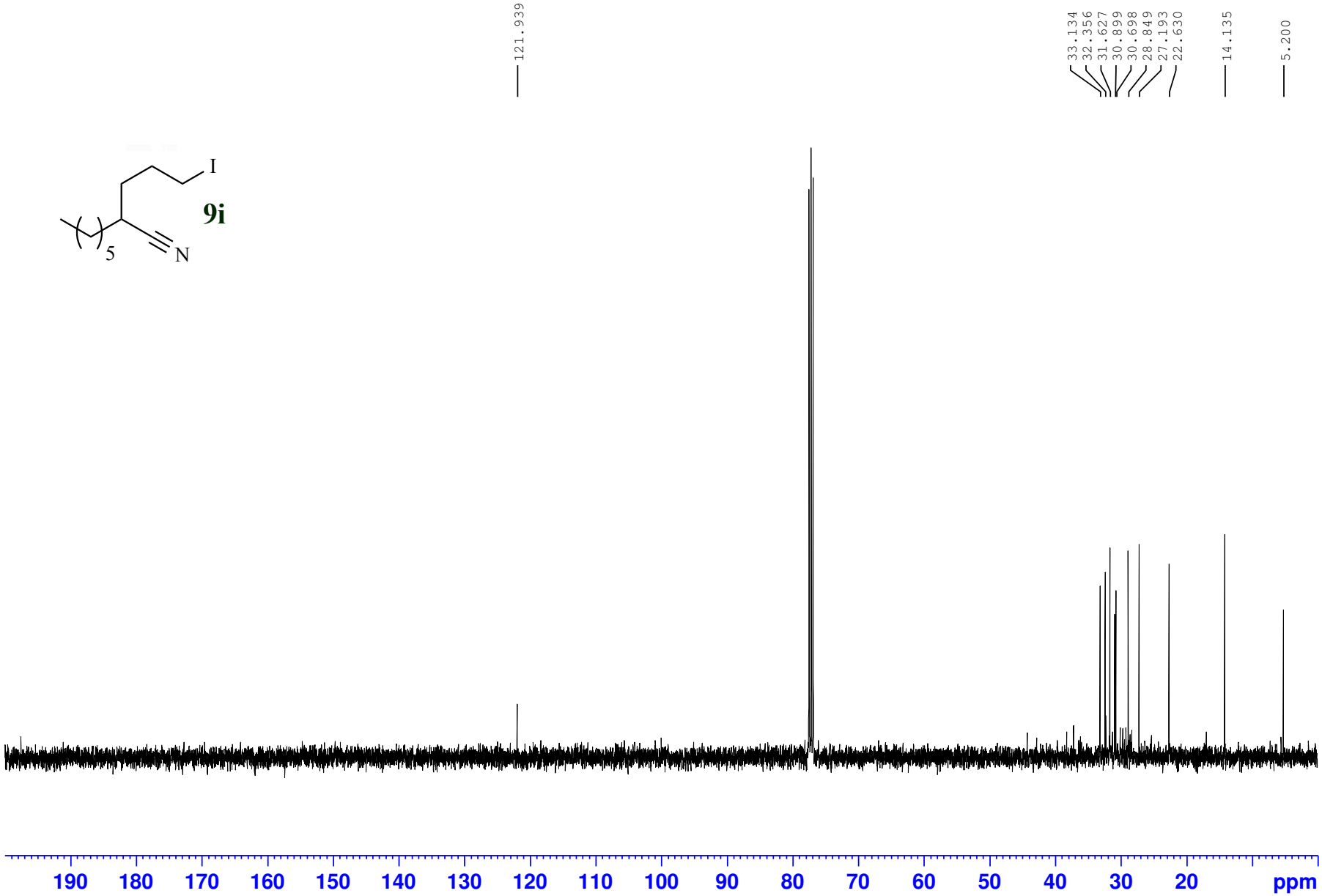
4.245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31.627
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30.698
28.849
27.193
22.630
14.135
5.200
I
9i
5
N
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180
170
160
150
140
130
120
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100
90
80
70
60
50
40
30
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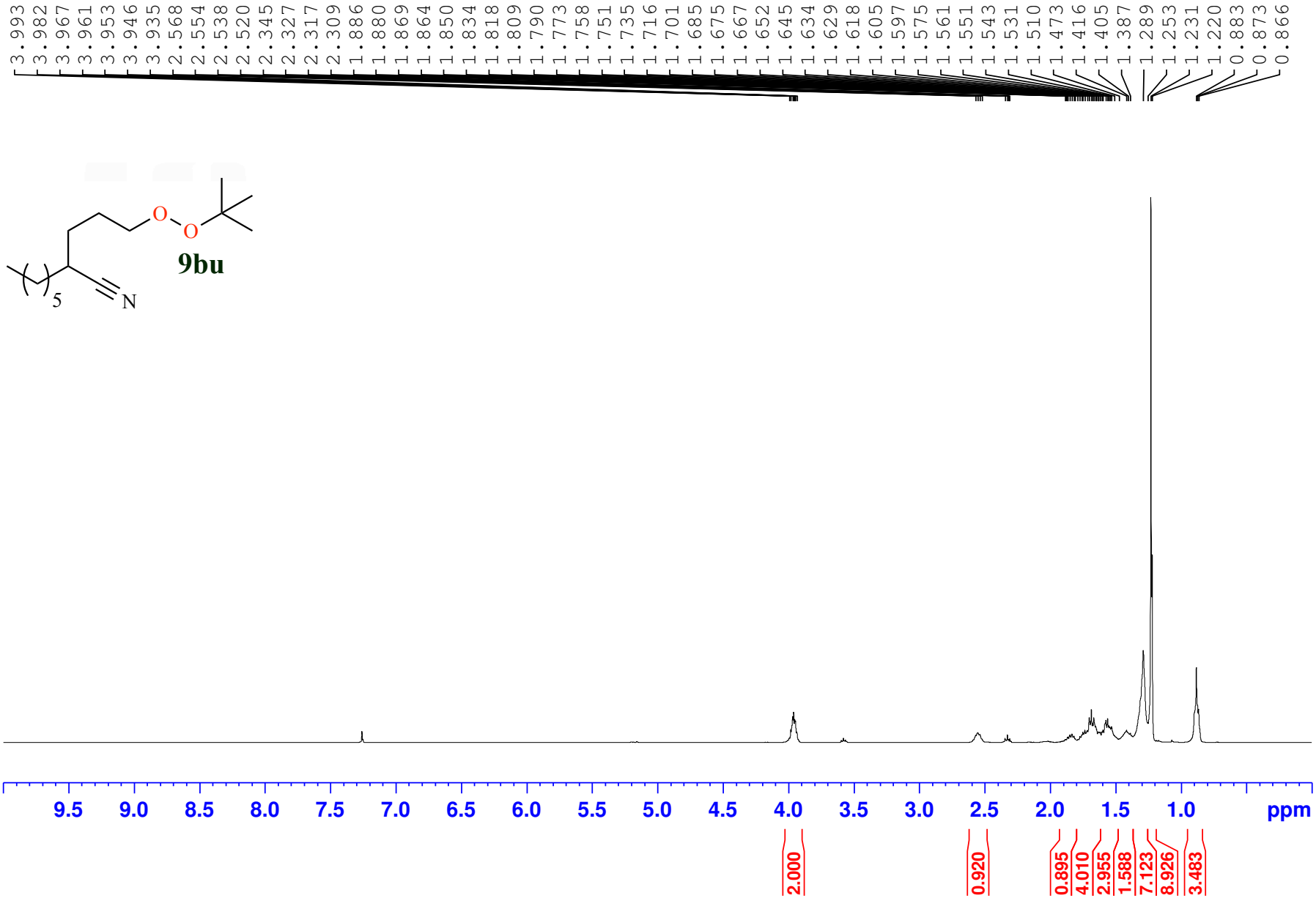
ppm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3.982
3.967
3.961
3.953
3.946
3.935
2.568
2.554
2.538
2.520
2.345
2.327
2.317
2.309
1.886
1.880
1.869
1.864
1.850
1.834
1.818
1.809
1.790
1.773
1.758
1.751
1.735
1.716
1.701
1.685
1.675
1.667
1.652
1.645
1.634
1.629
1.618
1.605
1.597
1.575
1.561
1.551
1.543
1.531
1.510
1.473
1.416
1.405
1.387
1.289
1.253
1.231
1.220
0.883
0.873
0.866
O
O
9bu
5
N
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
ppm
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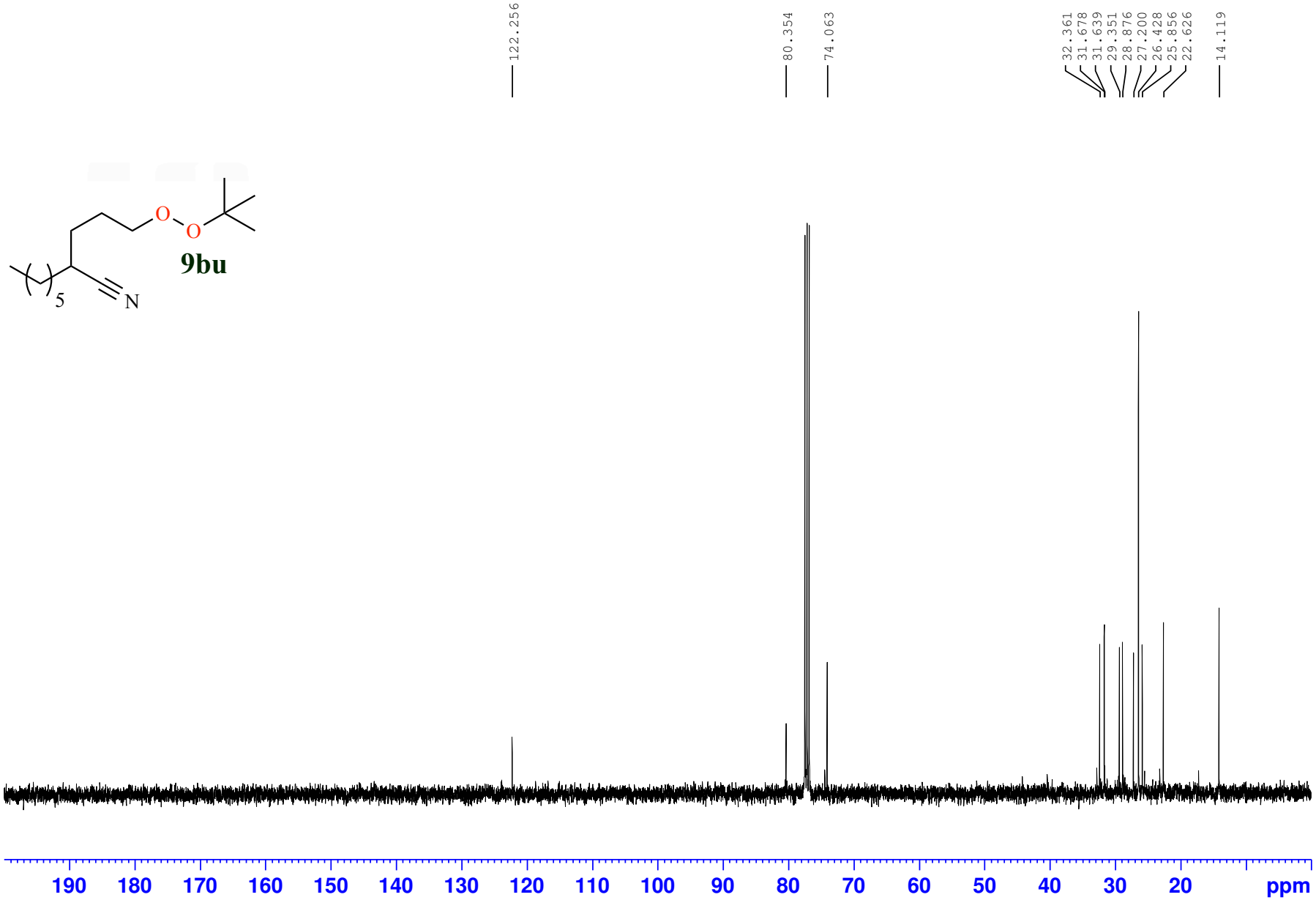
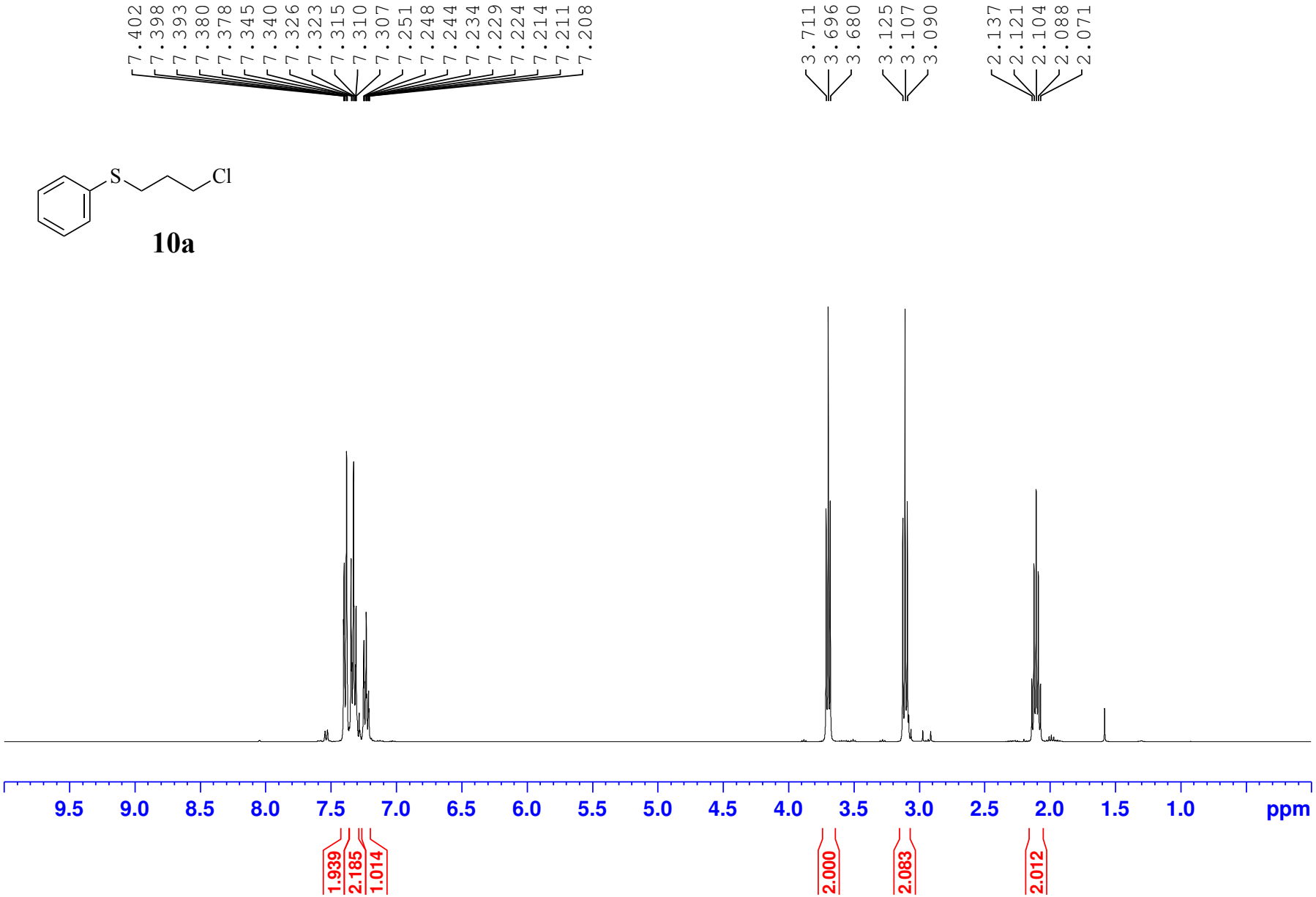
0.920
0.895
4.010
2.955
1.588
7.123
8.926
3.483


9bu
122.256
80.354
74.063
32.361
31.678
31.639
29.351
28.876
27.200
26.428
25.856
22.626
14.119
190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 ppm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7.398
7.393
7.380
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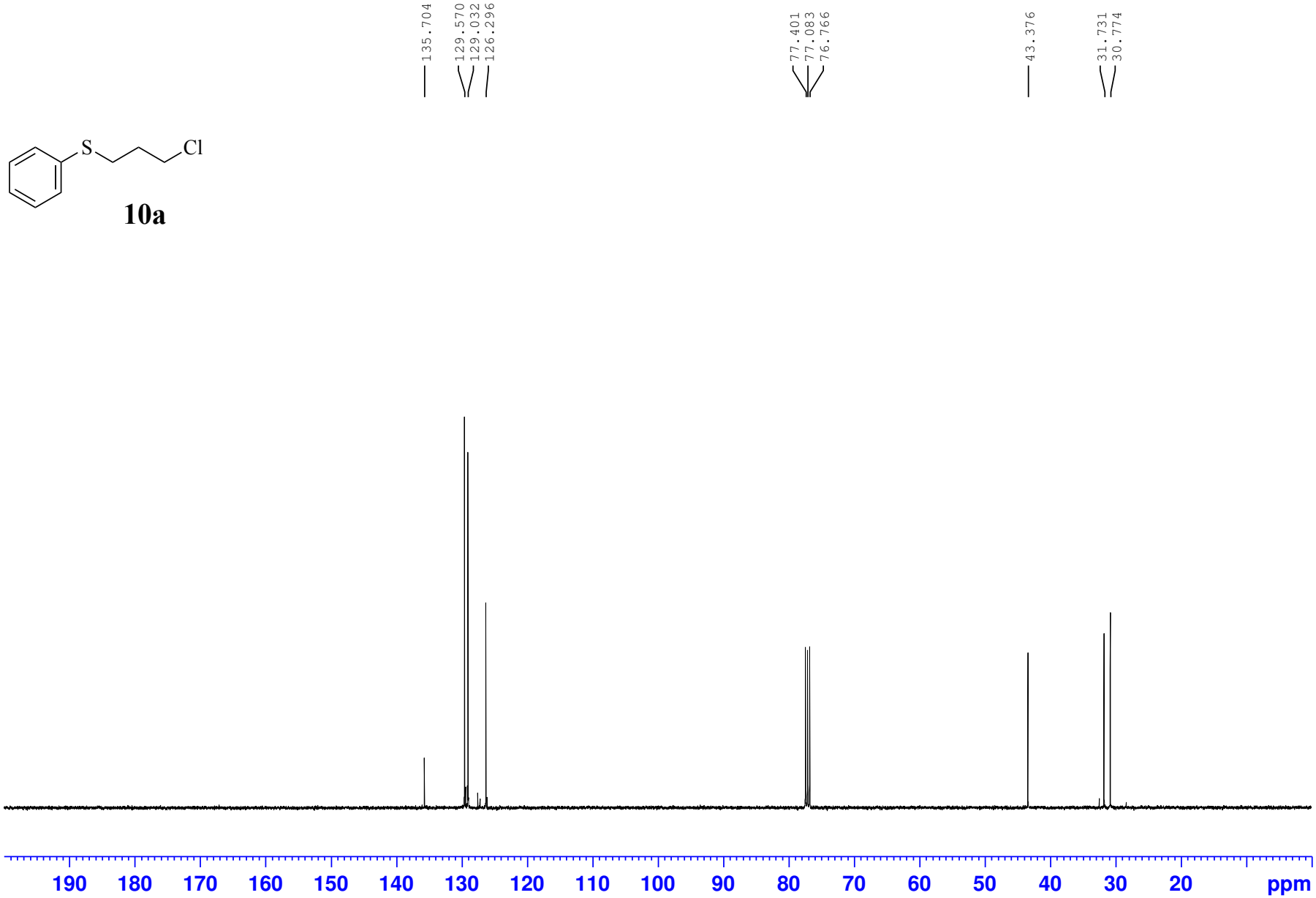
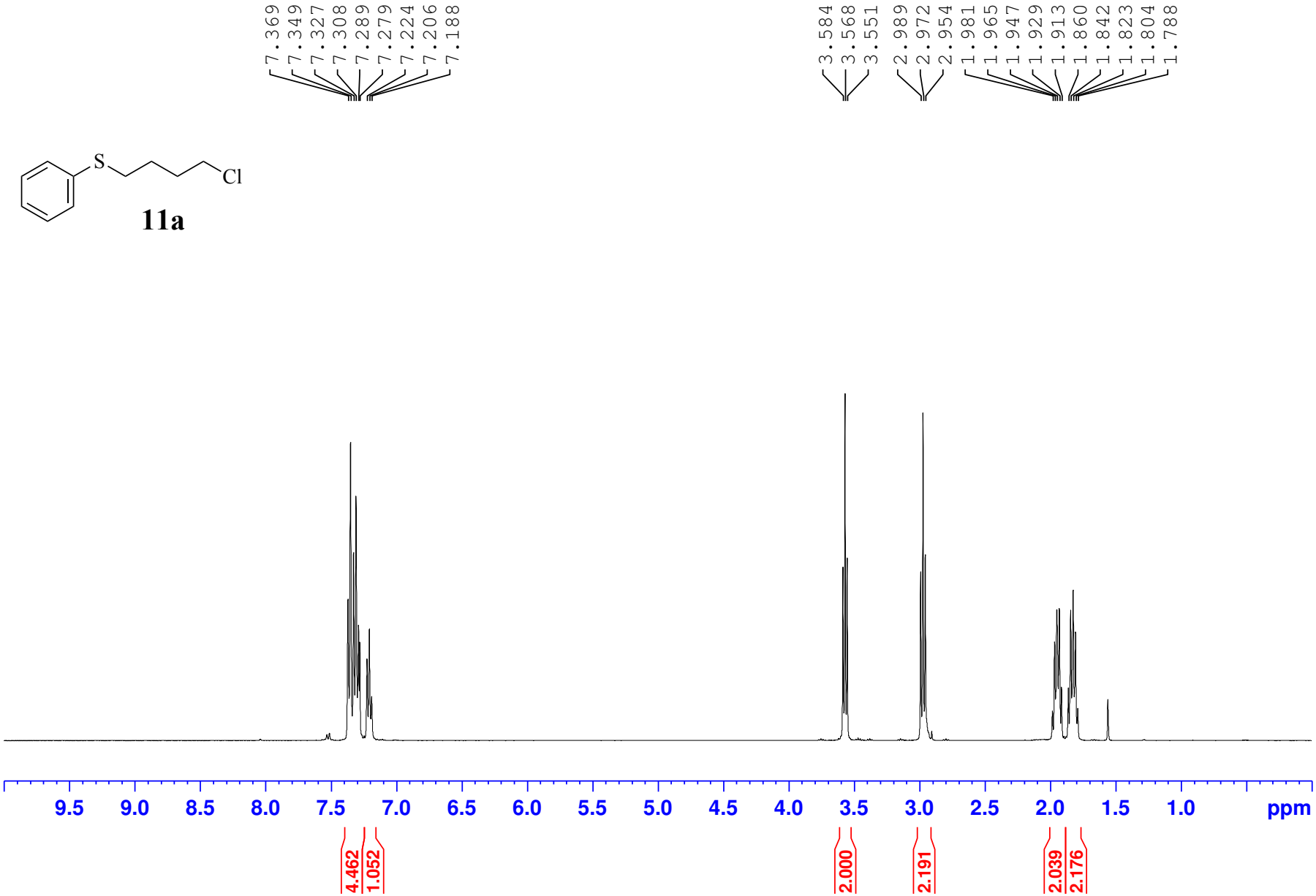
7.345
7.340
7.326
7.323
7.315
7.310
7.307
7.251
7.248
7.244
7.234
7.229
7.224
7.214
7.211
7.208
3.711
3.696
3.680
3.125
3.107
3.090
2.137
2.121
2.104
2.088
2.071
S
Cl
10a
9.5
9.0
8.5
8.0
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
ppm
1.939
2.185
1.014
2.000
2.083
2.012

S
Cl

10a


135.704
129.570
129.032
126.296
77.401
77.083
76.766
43.376
31.731
30.774
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
ppm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7.349
7.327
7.308
7.289
7.279
7.224
7.206
7.188
3.584
3.568
3.551
2.989
2.972
2.954
1.981
1.965
1.947
1.929
1.913
1.860
1.842
1.823
1.804
1.788
S
Cl
11a
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 ppm
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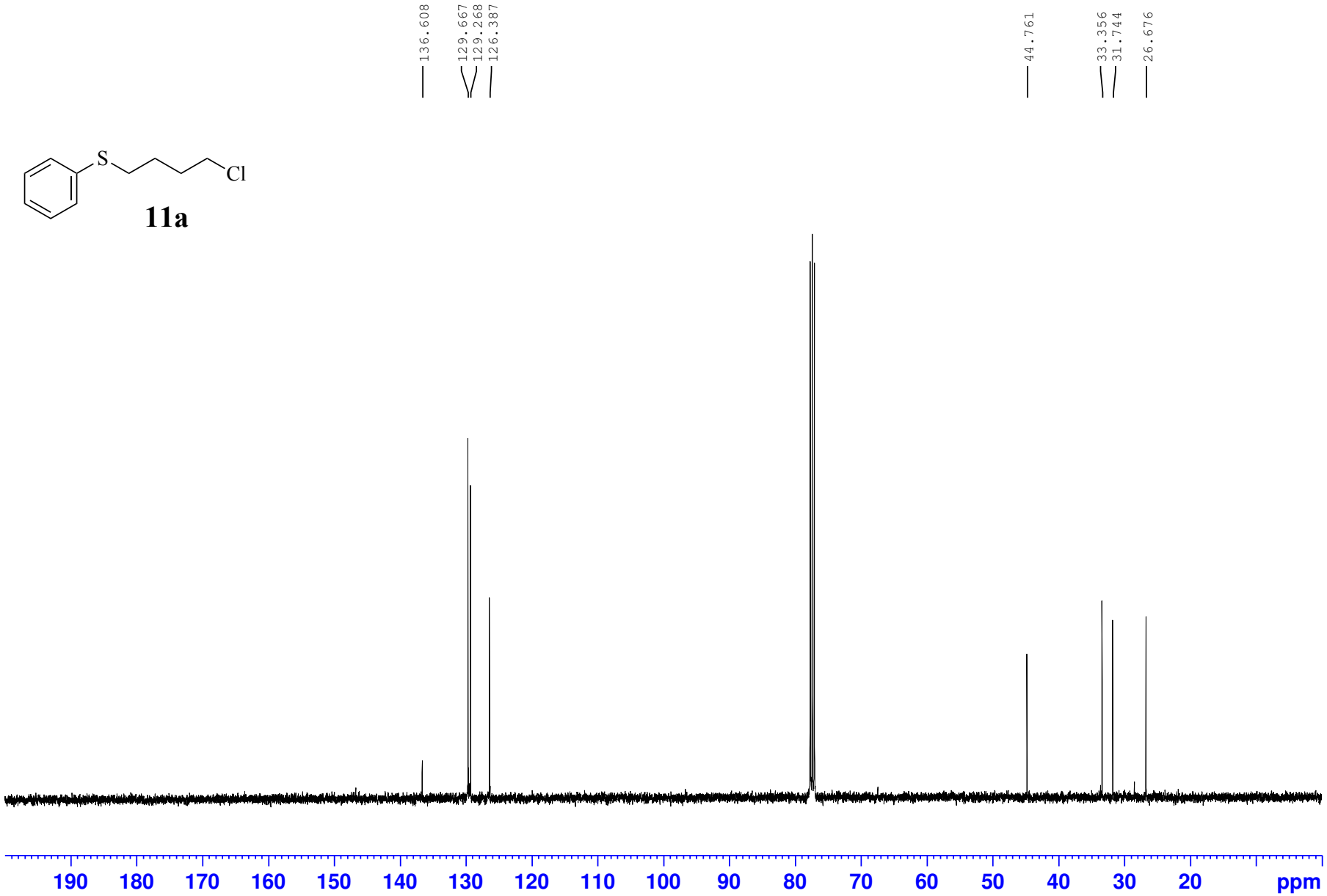
1.052
2.000
2.191
2.039
2.176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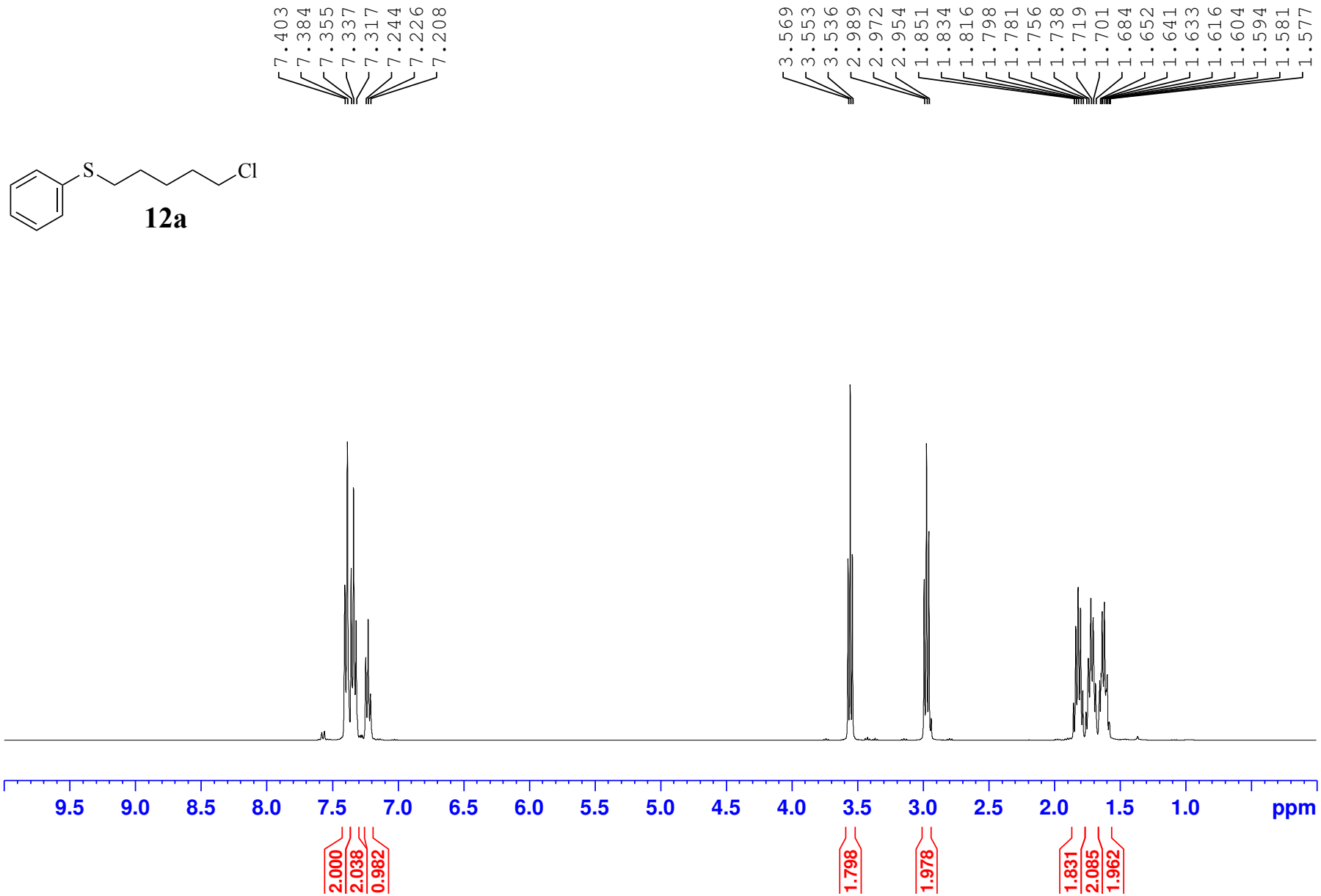
129.667
129.268
126.387
44.761
33.356
31.744
26.676
S
Cl
11a
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
ppm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7.384
7.355
7.337
7.317
7.244
7.226
7.208
3.569
3.553
3.536
2.989
2.972
2.954
1.851
1.834
1.816
1.798
1.781
1.756
1.738
1.719
1.701
1.684
1.652
1.641
1.633
1.616
1.604
1.594
1.581
1.577
S
Cl
12a
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
ppm
2.000
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0.982
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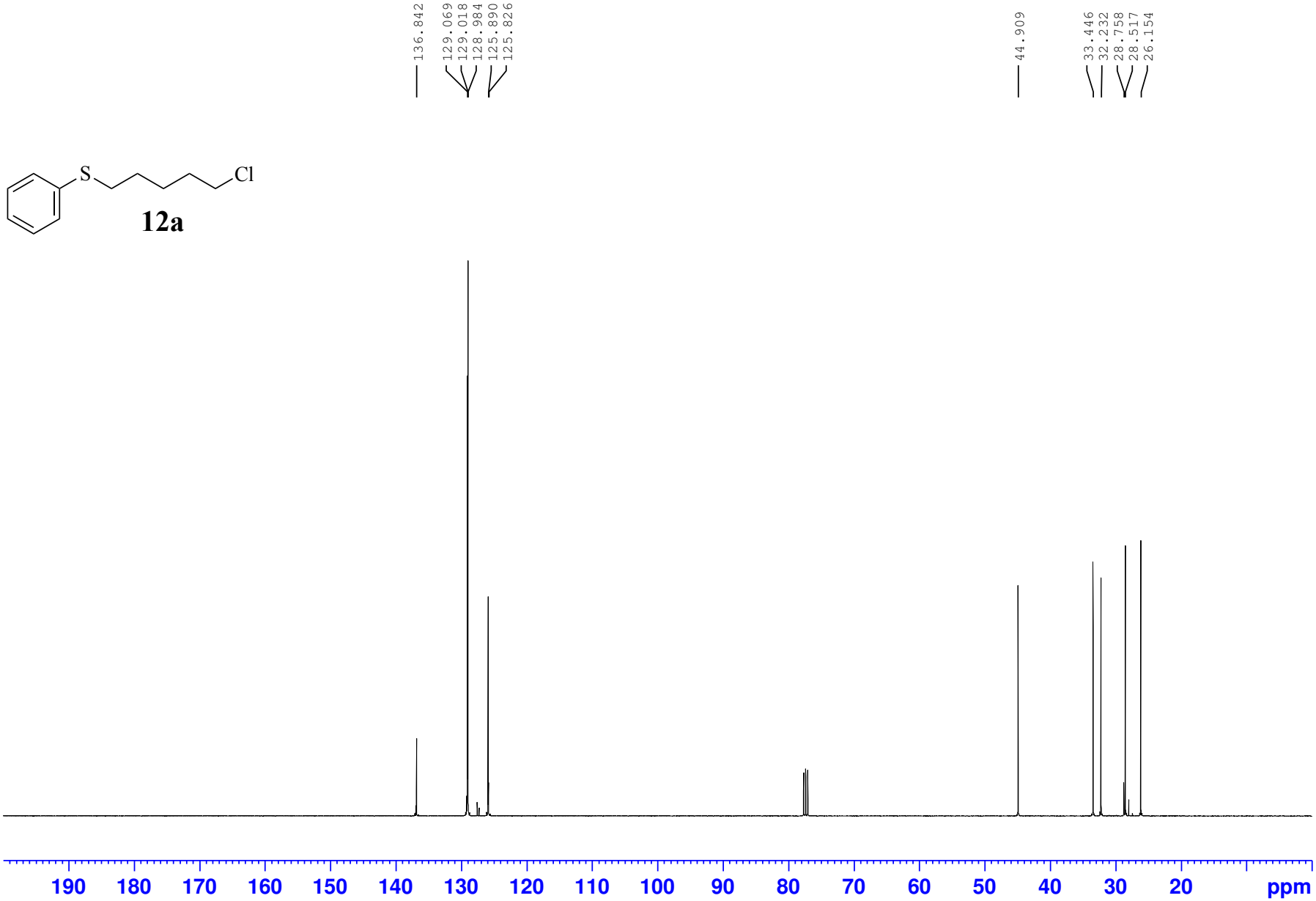
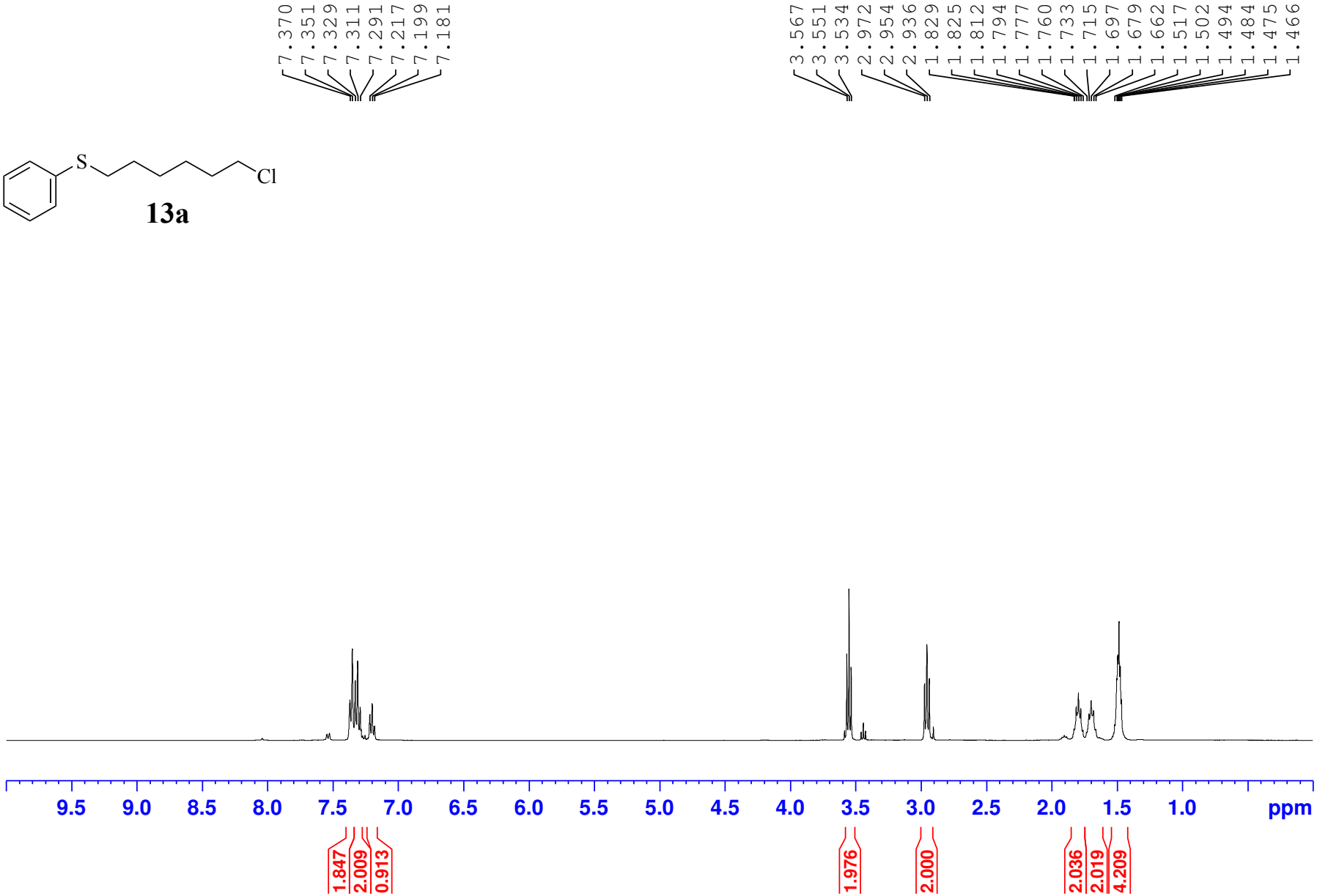
1.978
1.831
2.085
1.962


136.842
129.069
129.018
128.984
125.890
125.826
44.909
33.446
32.232
28.758
28.517
26.154
S
Cl
12a
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
ppm

S
Cl

13a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7.351
7.329
7.311
7.291
7.217
7.199
7.181
3.567
3.551
3.534
2.972
2.954
2.936
1.829
1.825
1.812
1.794
1.777
1.760
1.733
1.715
1.697
1.679
1.662
1.517
1.502
1.494
1.484
1.475
1.466
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
ppm
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2.009
0.913
1.976
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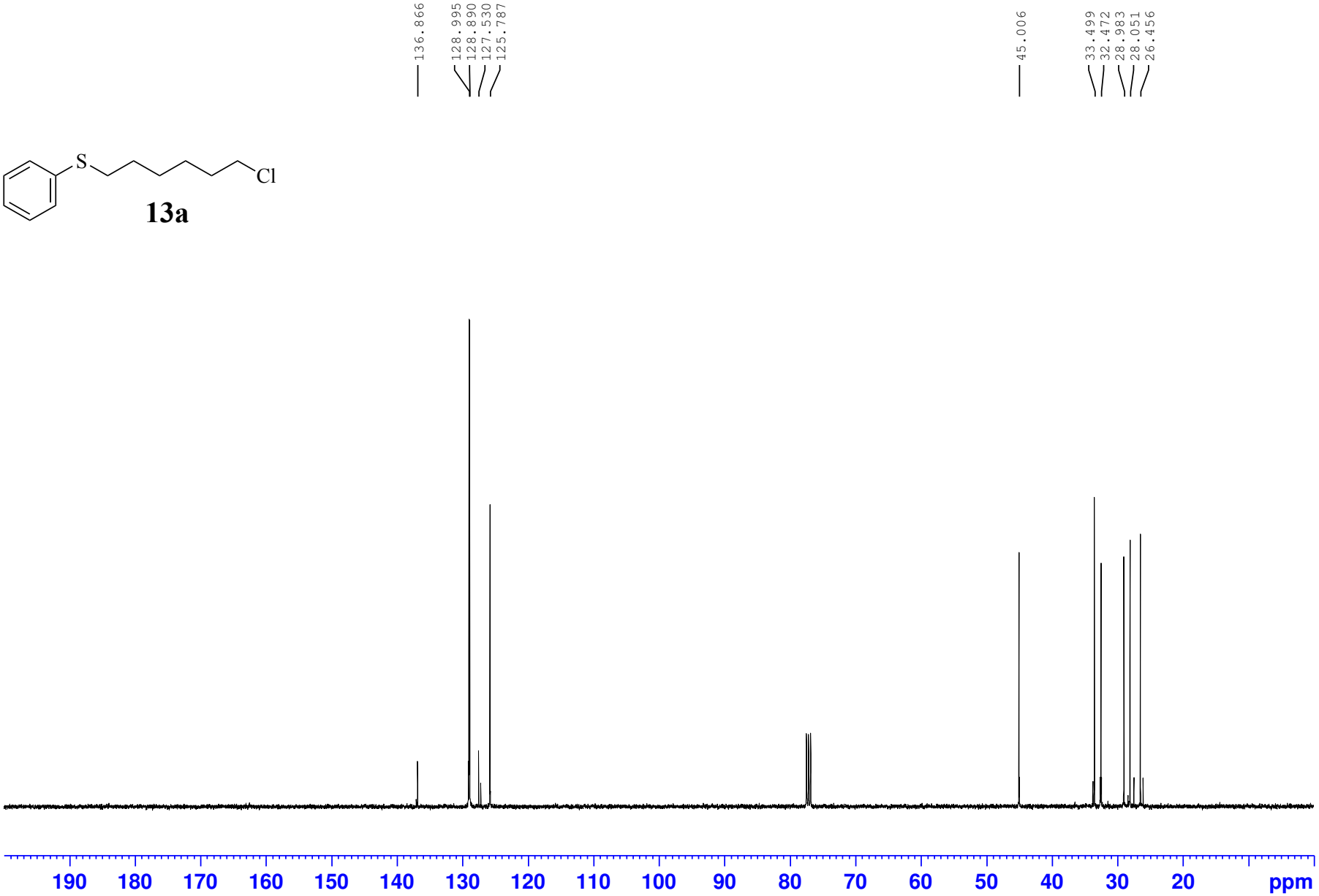
2.019
4.209

S
Cl

13a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128.995
128.890
127.530
125.787
45.006
33.499
32.472
28.983
28.051
26.456
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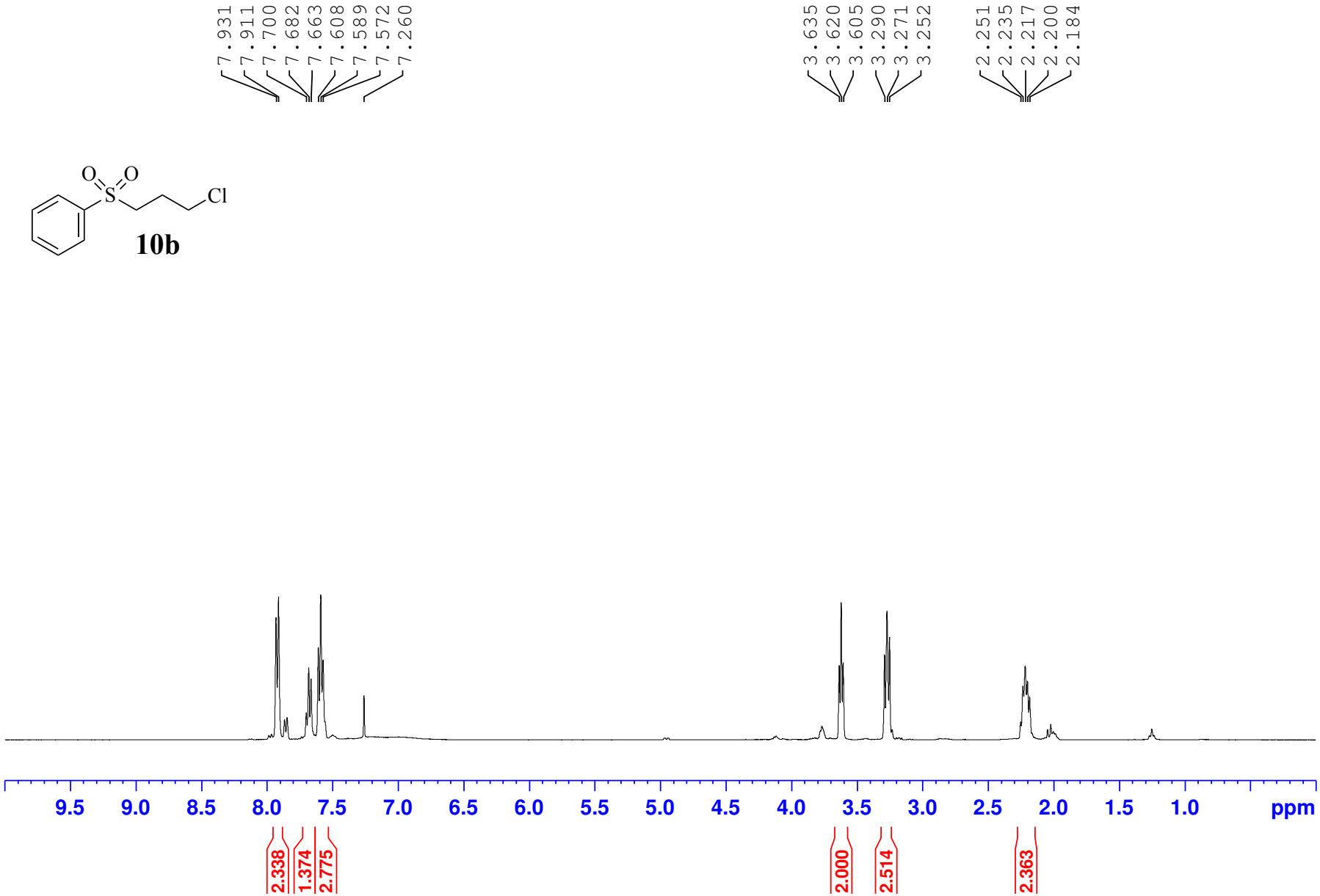
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
ppm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7.911
7.700
7.682
7.663
7.608
7.589
7.572
7.260
3.635
3.620
3.605
3.290
3.271
3.252
2.251
2.235
2.217
2.200
2.184
O
O
S
Cl
10b
9.5
9.0
8.5
8.0
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
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2.0
1.5
1.0
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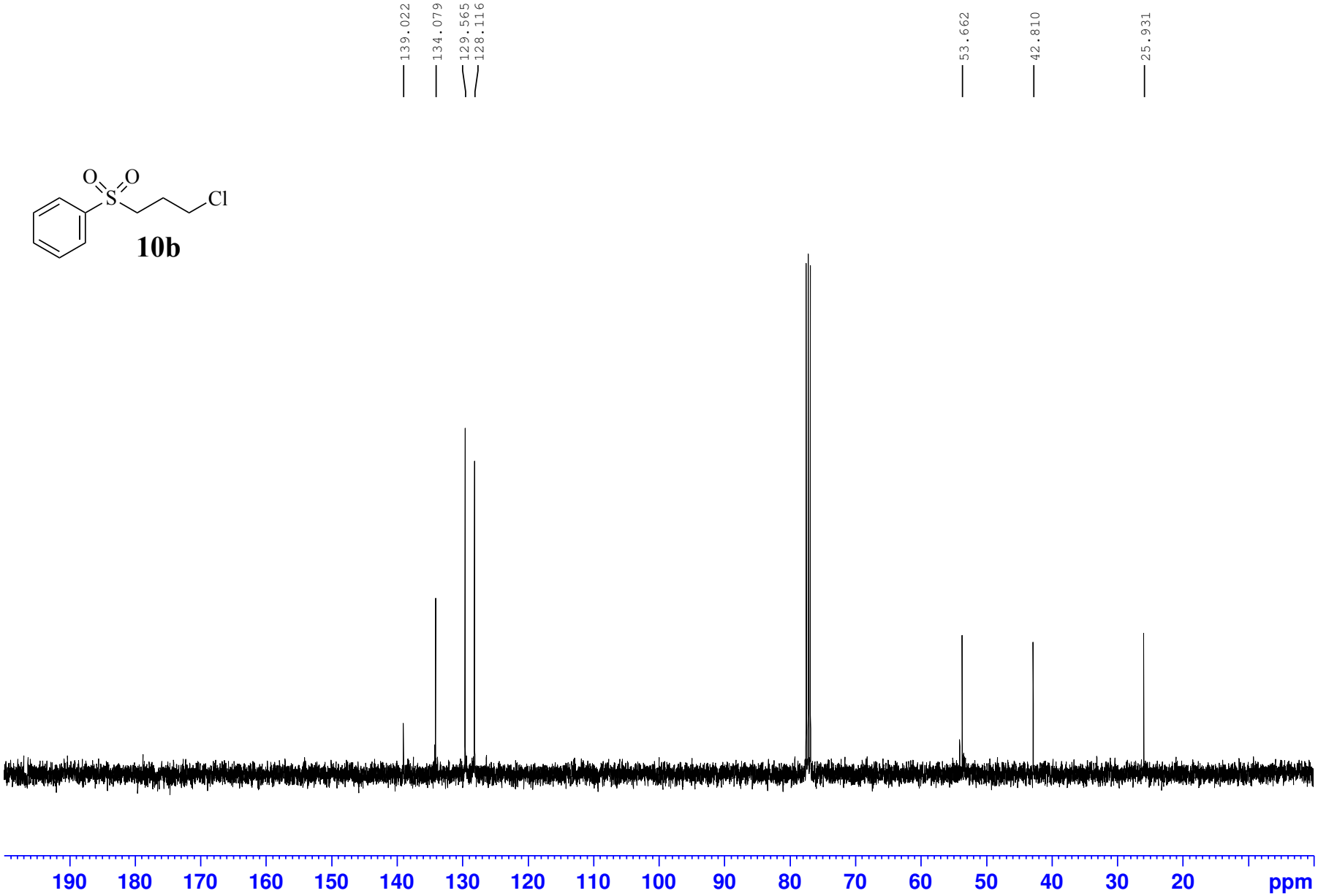
2.363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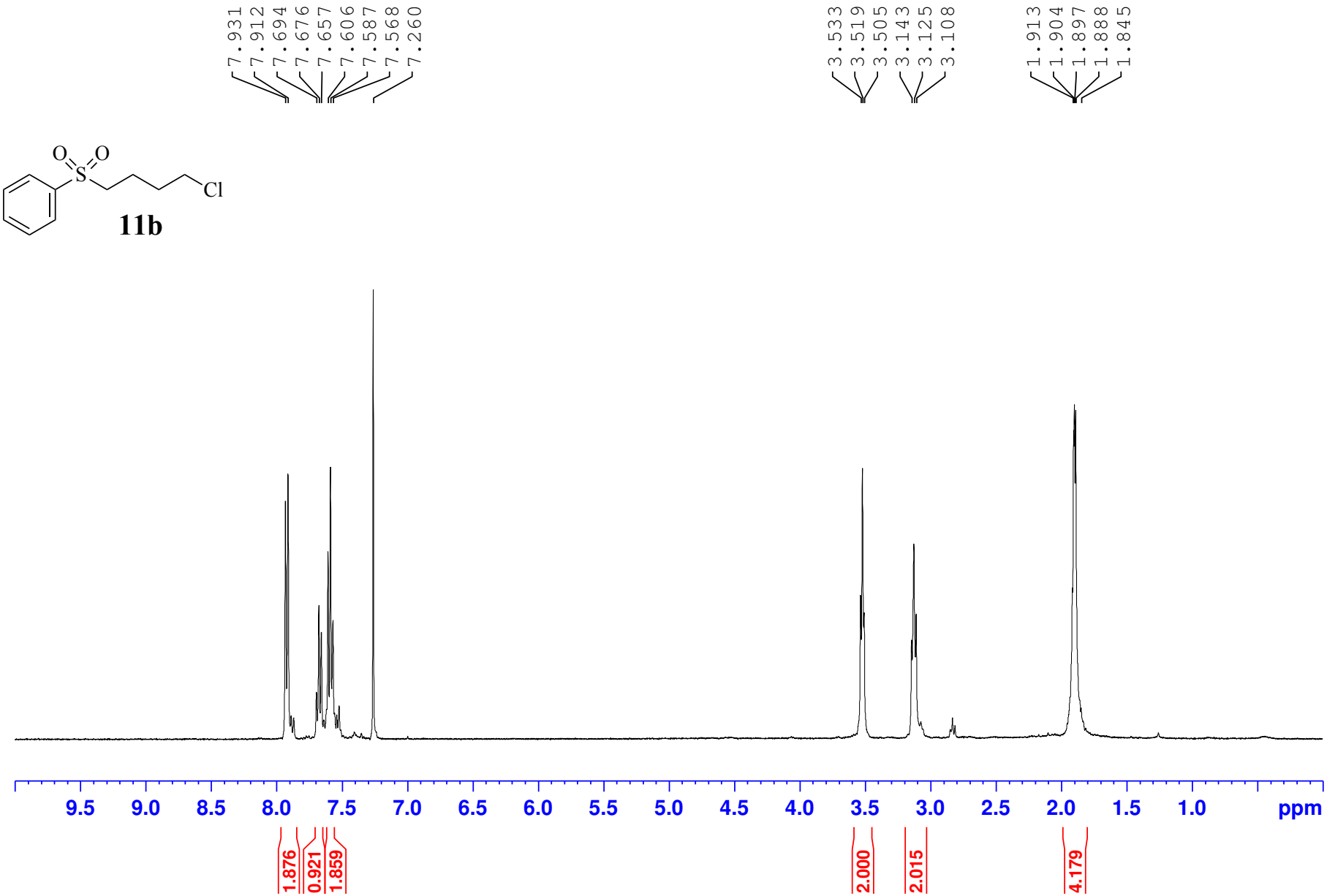
129.565
128.116
53.662
42.810
25.931
O
O
S
Cl
10b
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
ppm

O
O
S
Cl

11b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7.694
7.676
7.657
7.606
7.587
7.568
7.260
3.533
3.519
3.505
3.143
3.125
3.108
1.913
1.904
1.897
1.888
1.845
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
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0.921
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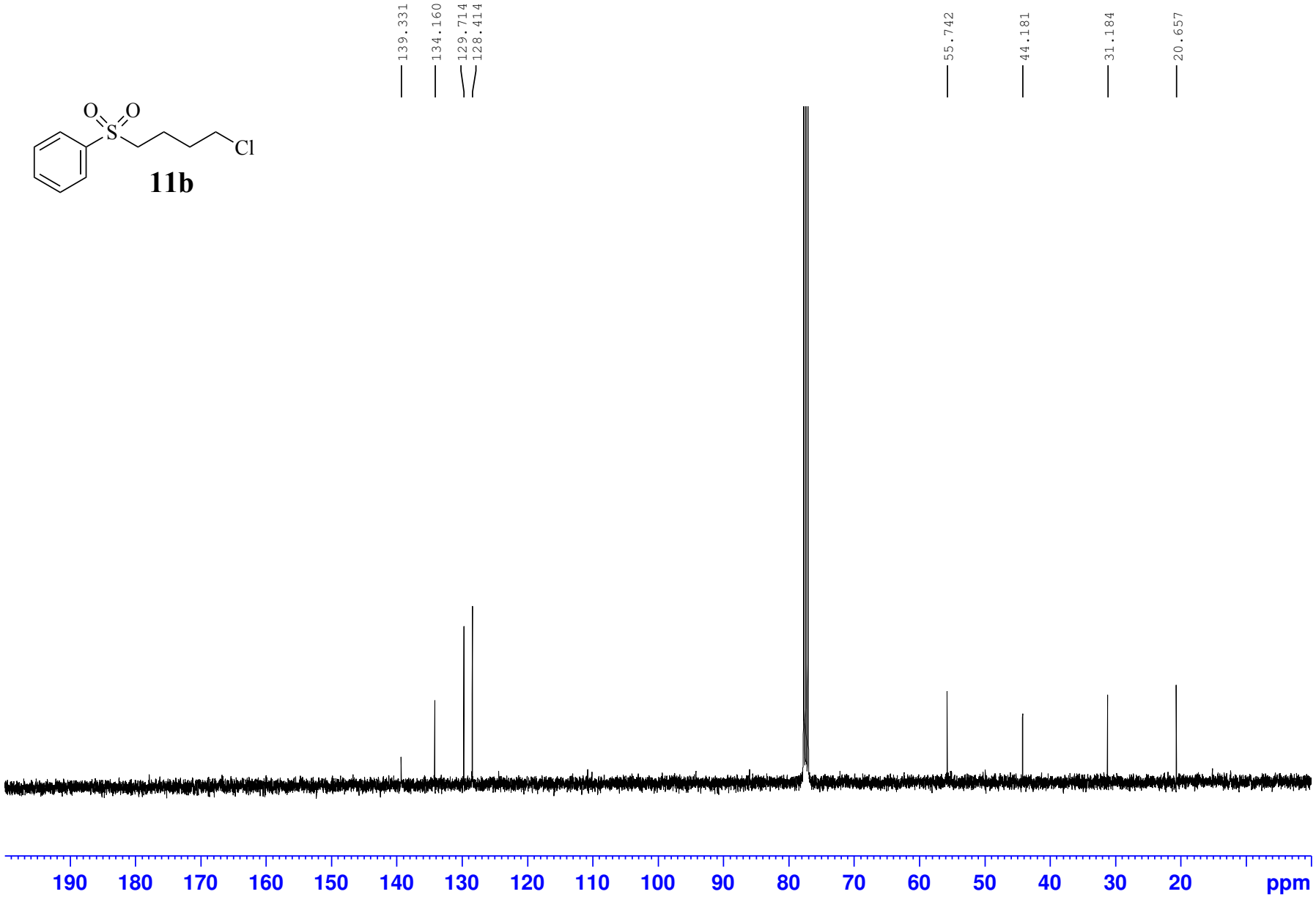
2.015
4.179

O
O
S
Cl

11b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31.184
20.657
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180
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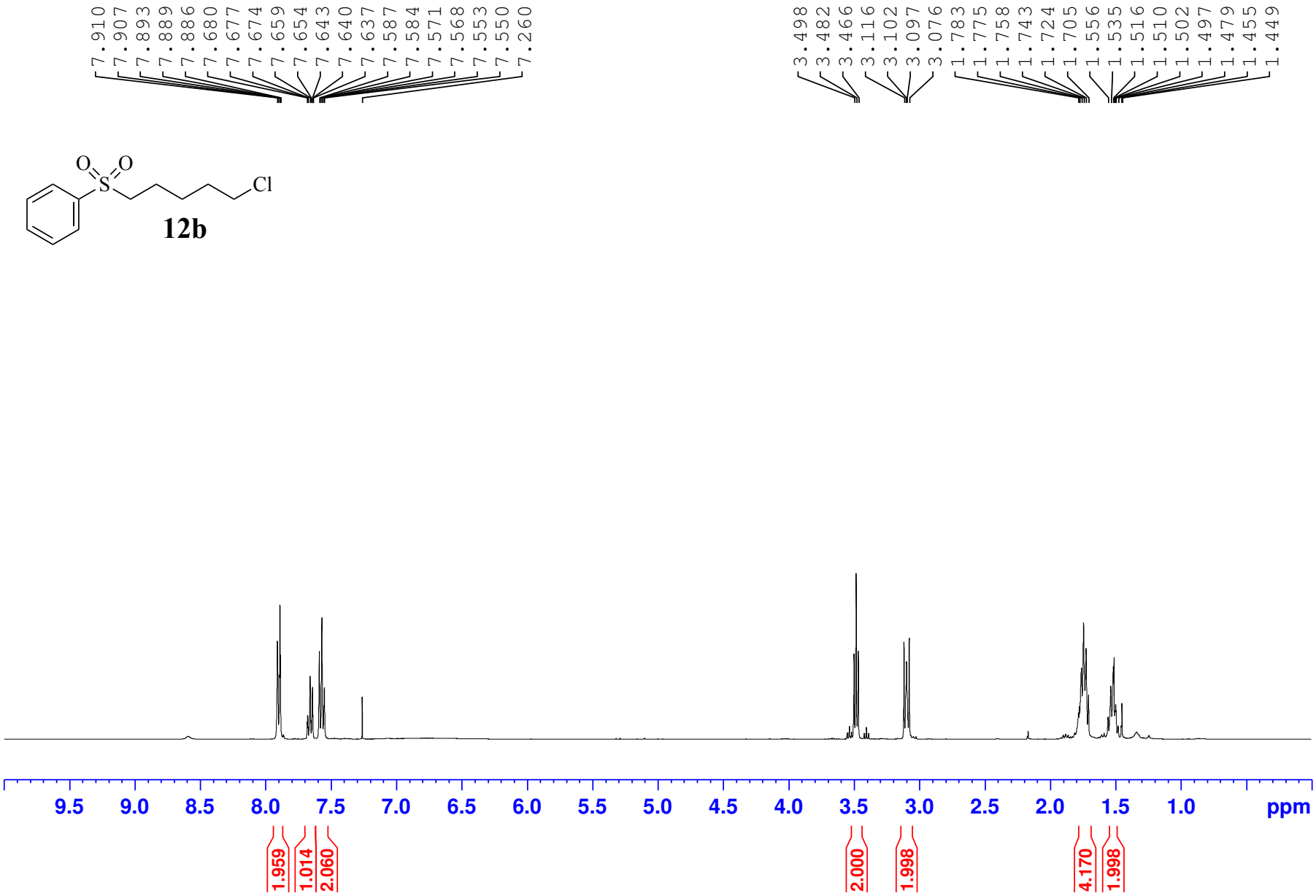
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
ppm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7.674
7.659
7.654
7.643
7.640
7.637
7.587
7.584
7.571
7.568
7.553
7.550
7.260
3.498
3.482
3.466
3.116
3.102
3.097
3.076
1.783
1.775
1.758
1.743
1.724
1.705
1.556
1.535
1.516
1.510
1.502
1.497
1.479
1.455
1.449
O
O
S
Cl
12b
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
ppm
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1.014
2.060
2.000
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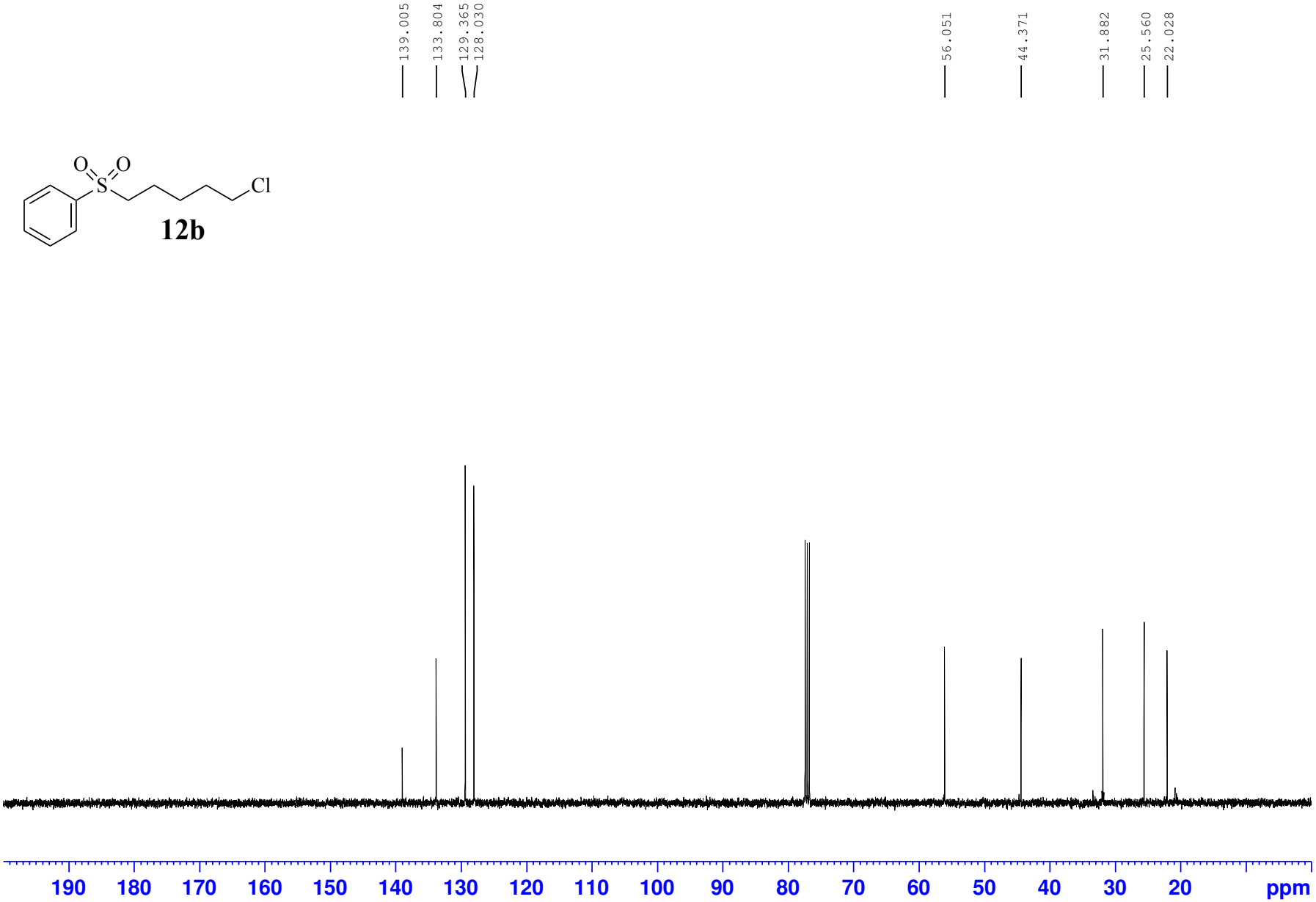
4.170
1.998

O
O
S
Cl

12b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129.365
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56.051
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31.882
25.560
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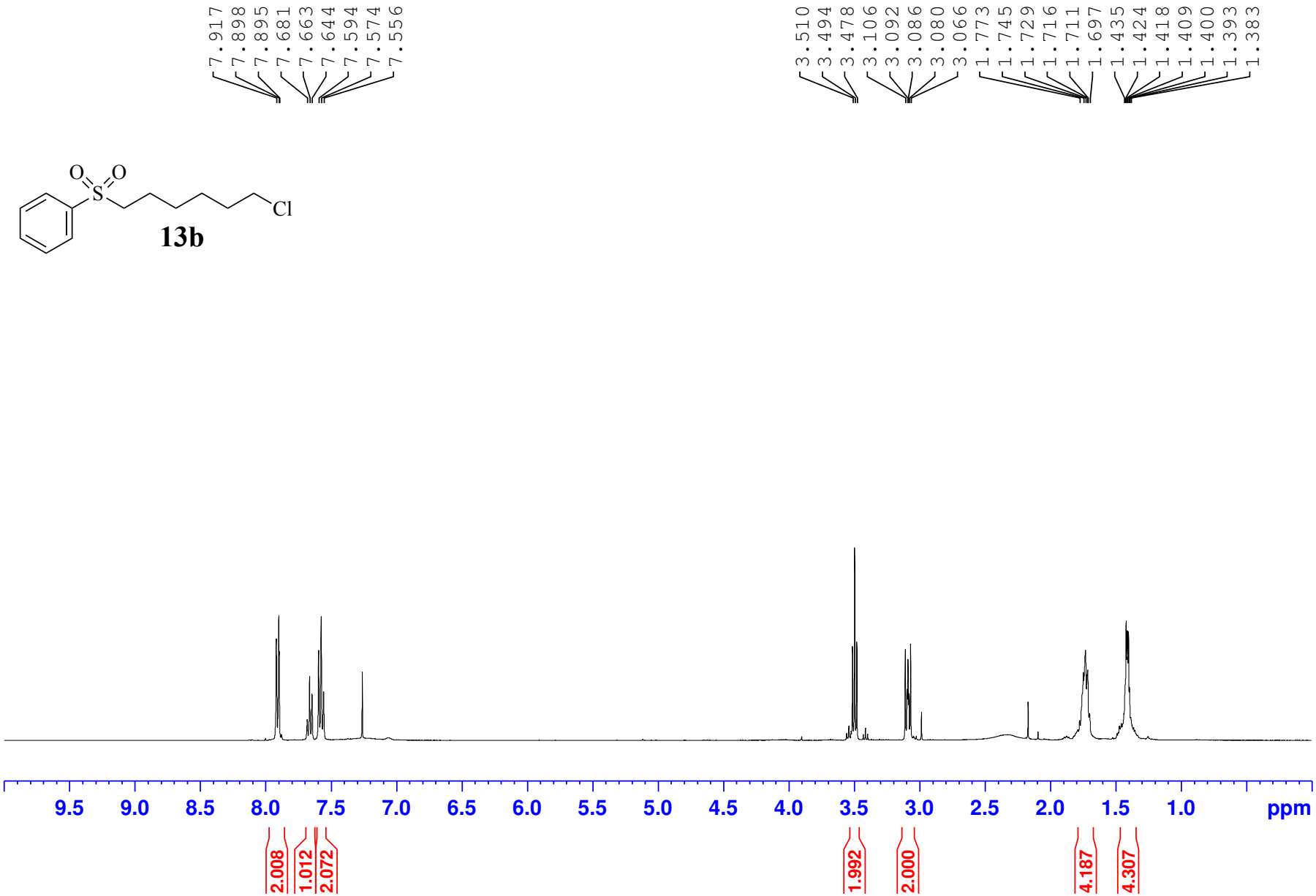
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
ppm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7.681
7.663
7.644
7.594
7.574
7.556
3.510
3.494
3.478
3.106
3.092
3.086
3.080
3.066
1.773
1.745
1.729
1.716
1.711
1.697
1.435
1.424
1.418
1.409
1.400
1.393
1.383
O
O
S
Cl
13b
9.5
9.0
8.5
8.0
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
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1.5
1.0
ppm
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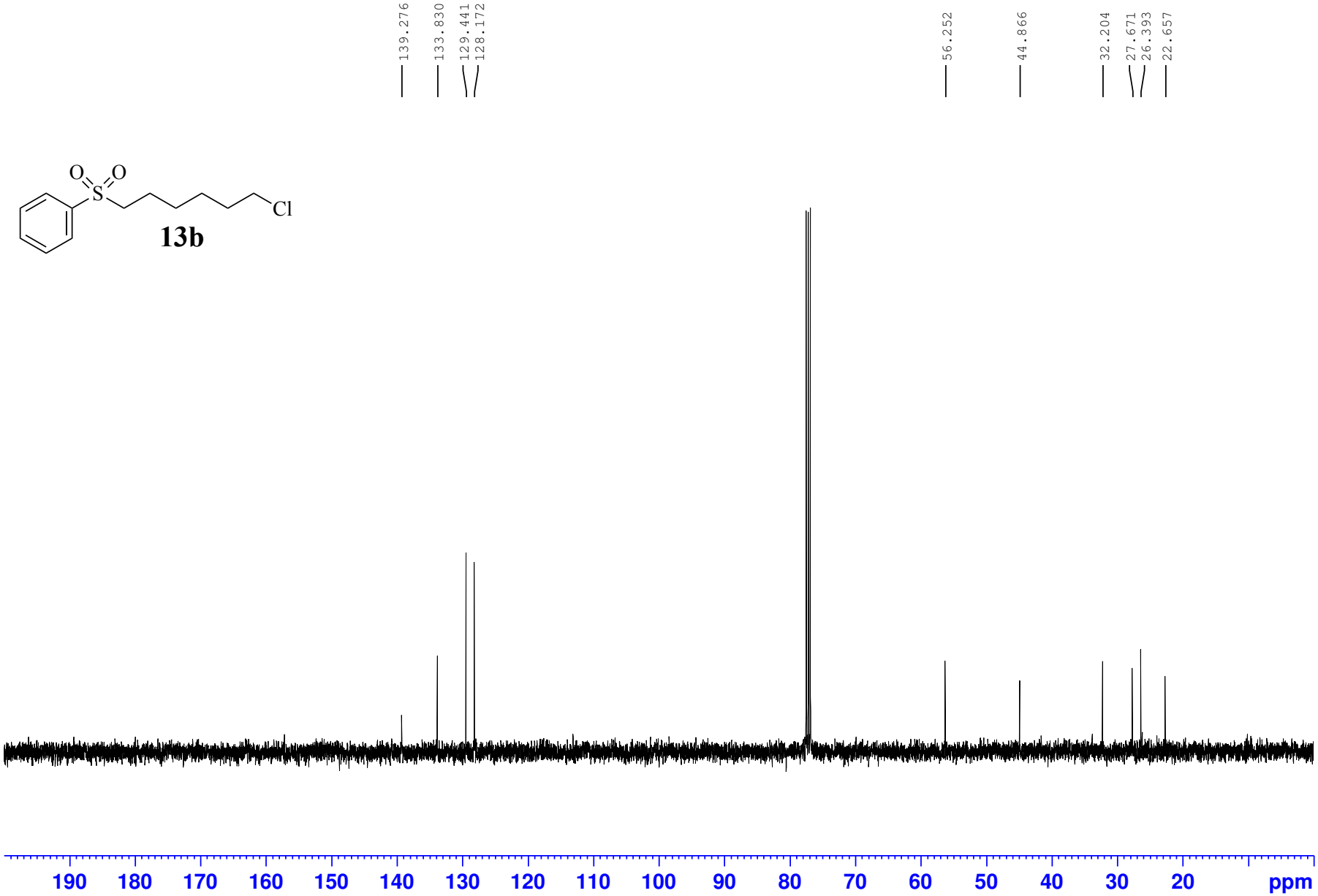
4.307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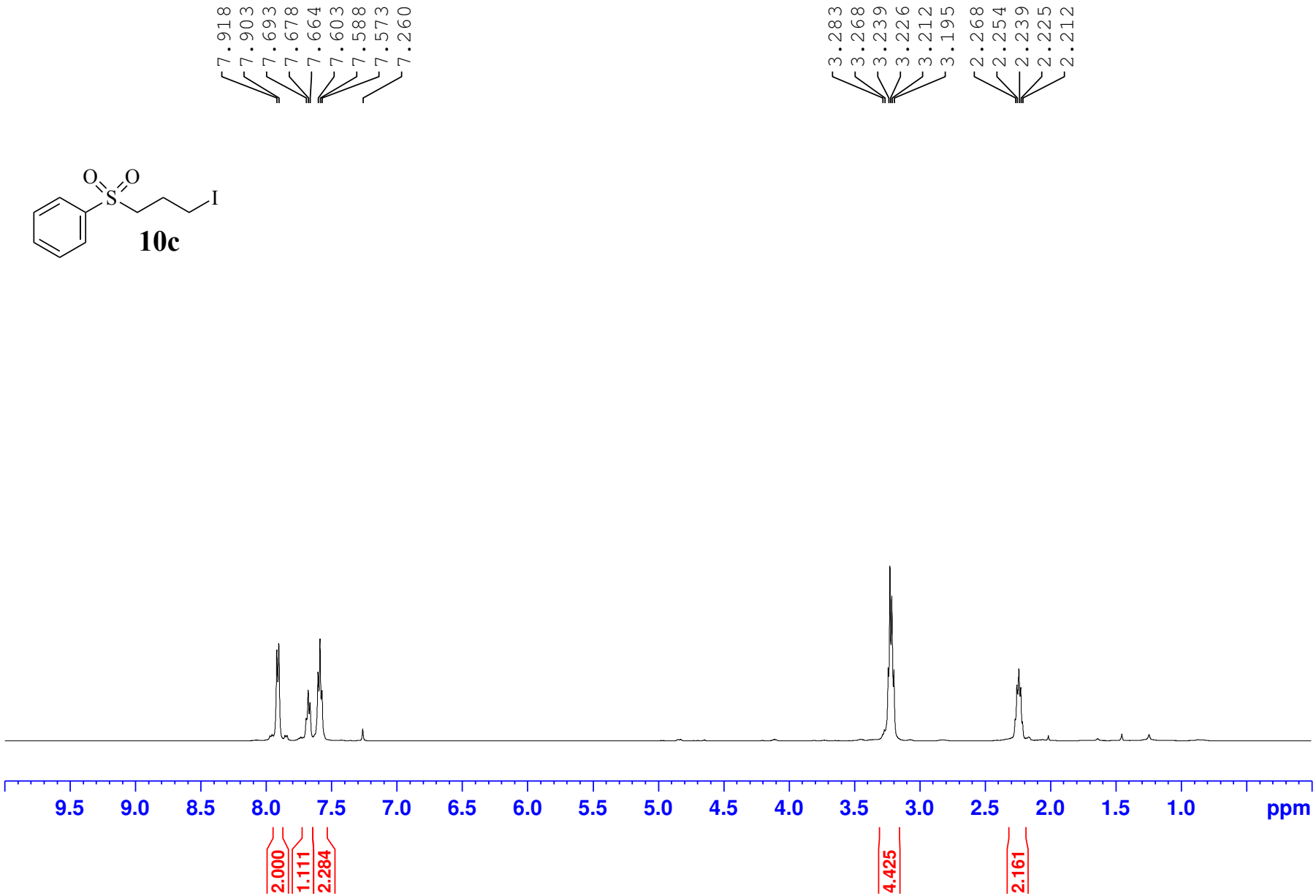
128.172
56.252
44.866
32.204
27.671
26.393
22.657
O
O
S
Cl
13b
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
ppm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7.664
7.603
7.588
7.573
7.260
3.283
3.268
3.239
3.226
3.212
3.195
2.268
2.254
2.239
2.225
2.212
O
O
S
I
10c
9.5
9.0
8.5
8.0
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
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2.5
2.0
1.5
1.0
ppm
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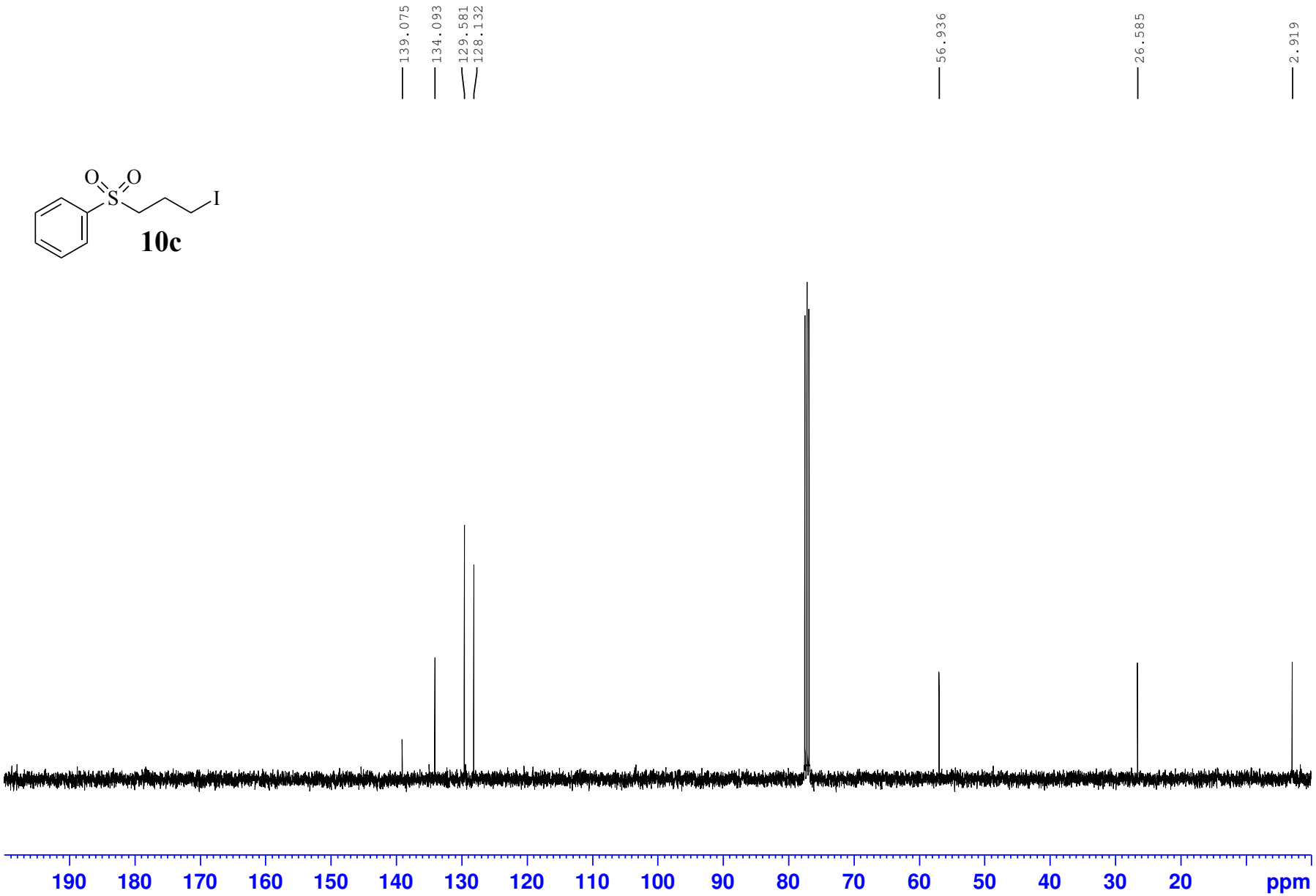
2.161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134.093
129.581
128.132
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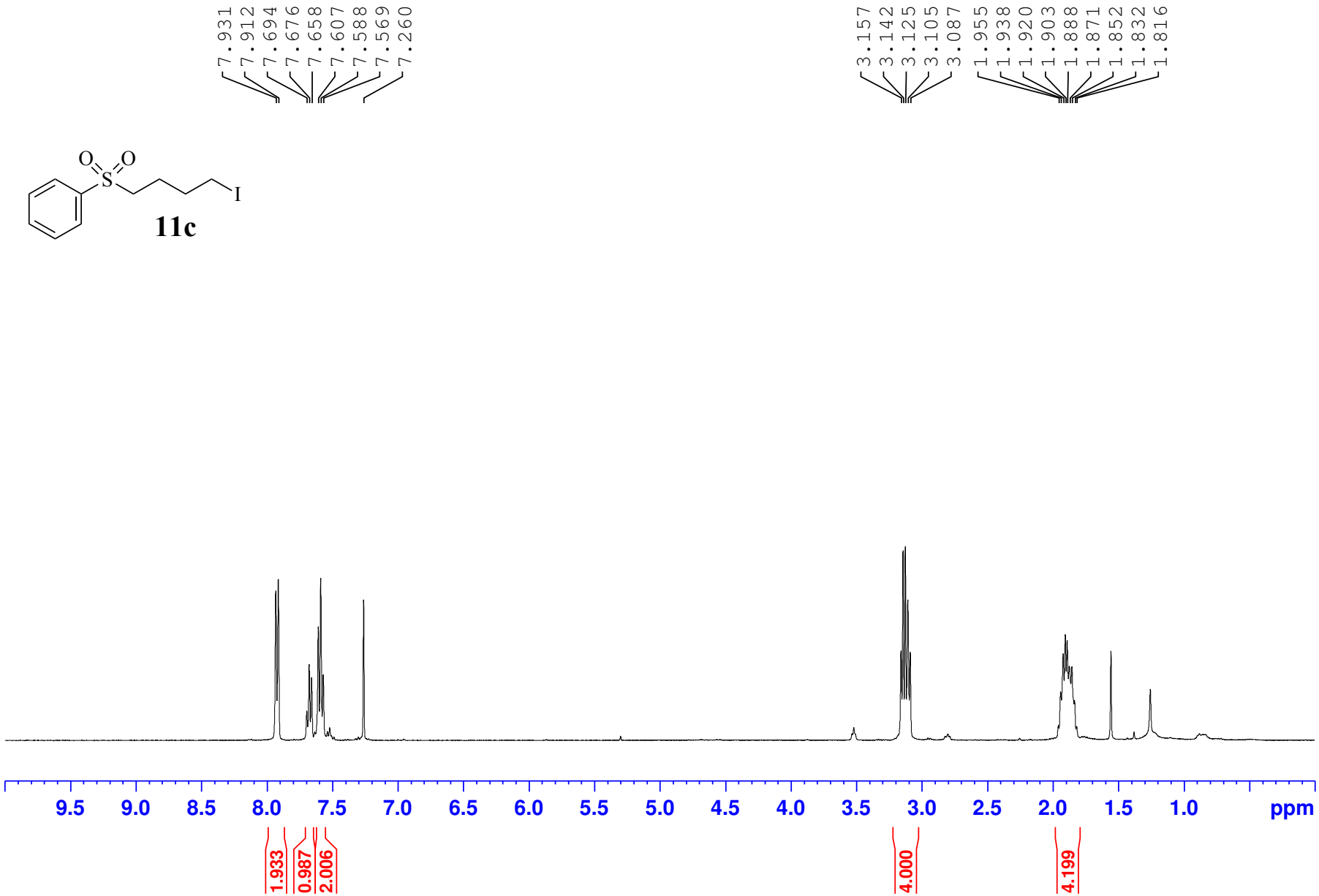
26.585
2.919
O
O
S
I
10c
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
ppm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3.125
3.105
3.087
1.955
1.938
1.920
1.903
1.888
1.871
1.852
1.832
1.816
O
O
S
I
11c
9.5
9.0
8.5
8.0
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
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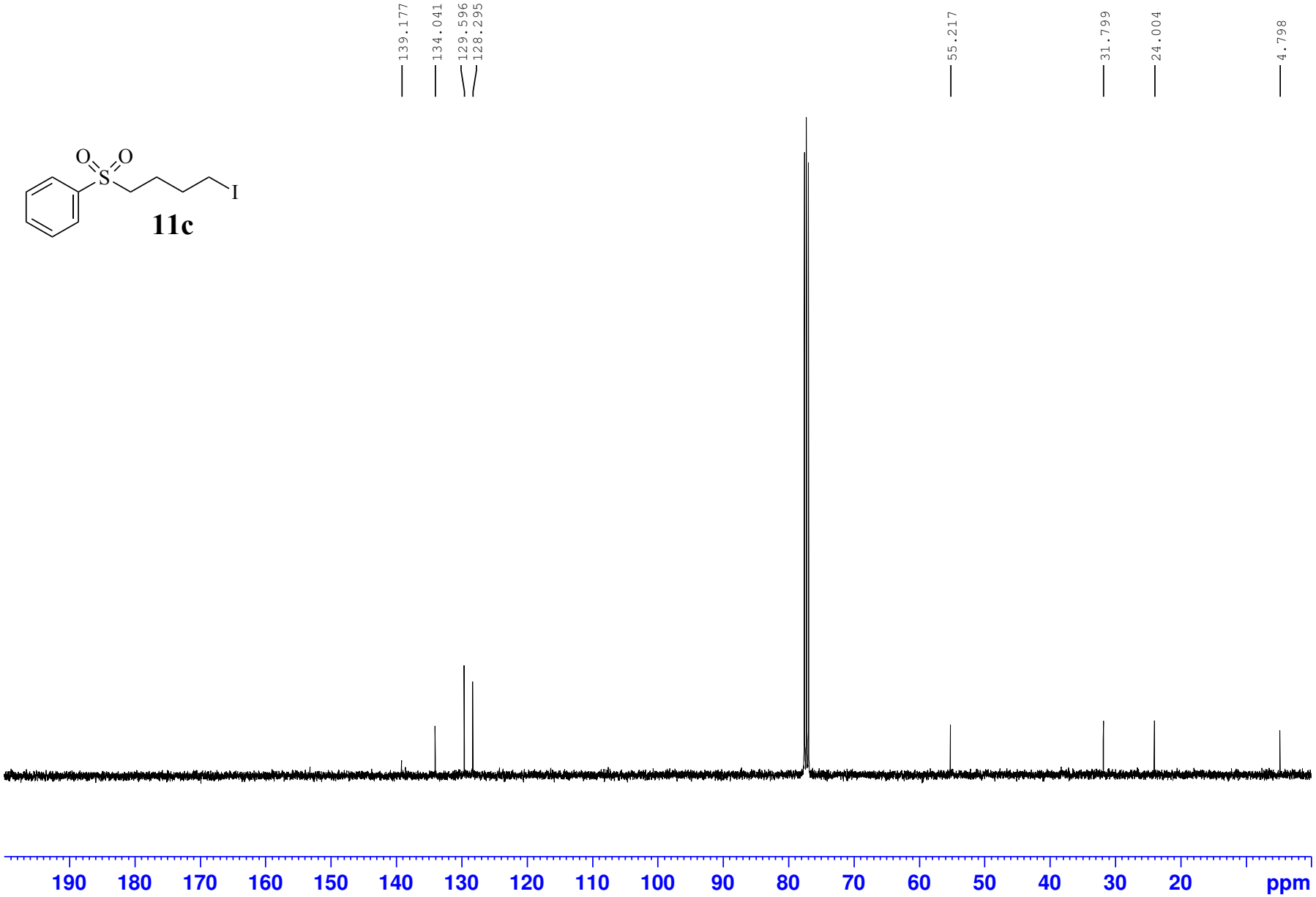
4.199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O
S
I
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80
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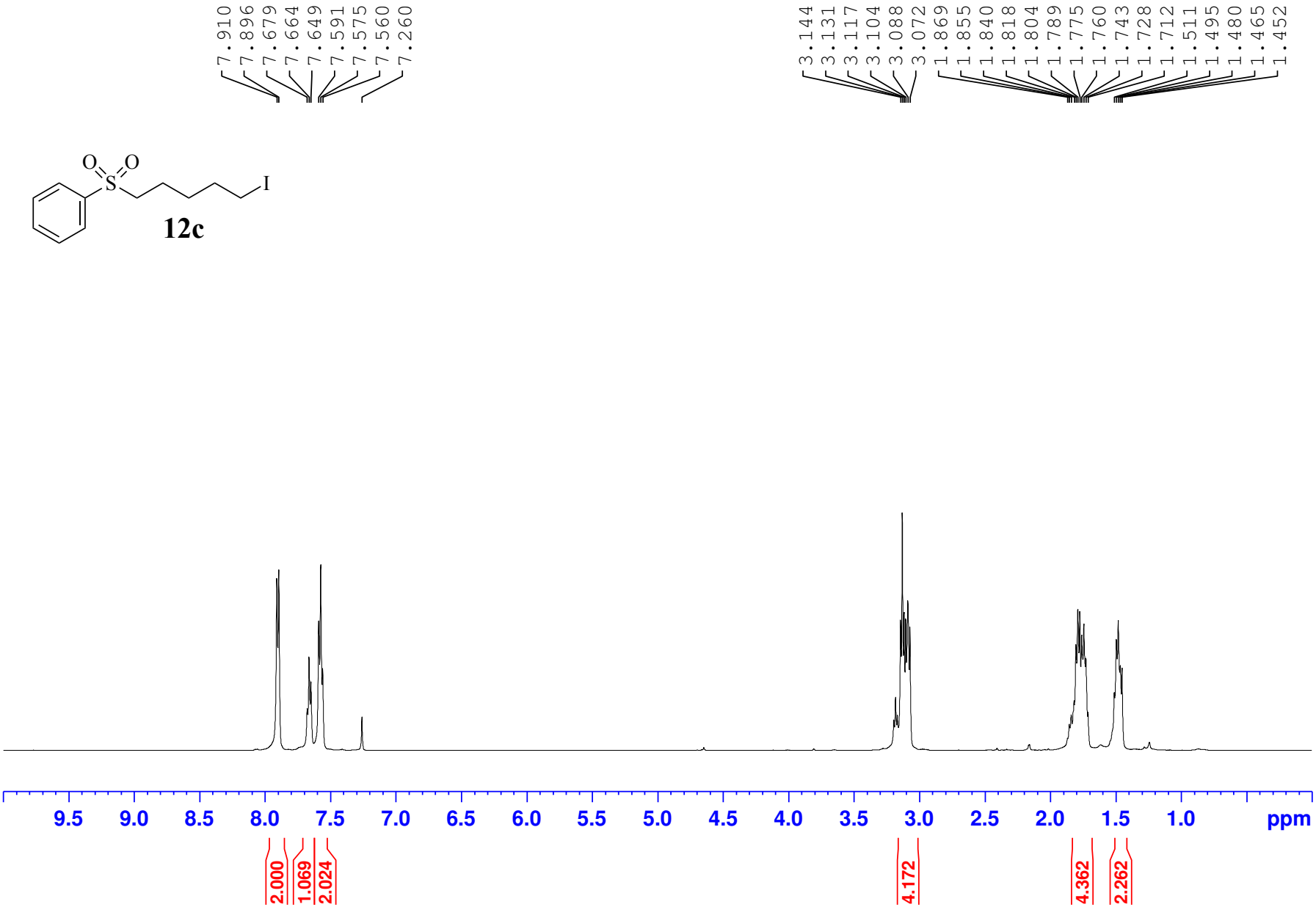
30
20
ppm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7.560
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3.104
3.088
3.072
1.869
1.855
1.840
1.818
1.804
1.789
1.775
1.760
1.743
1.728
1.712
1.511
1.495
1.480
1.465
1.452
O
O
S
I
12c
9.5
9.0
8.5
8.0
7.5
7.0
6.5
6.0
5.5
5.0
4.5
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3.0
2.5
2.0
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ppm
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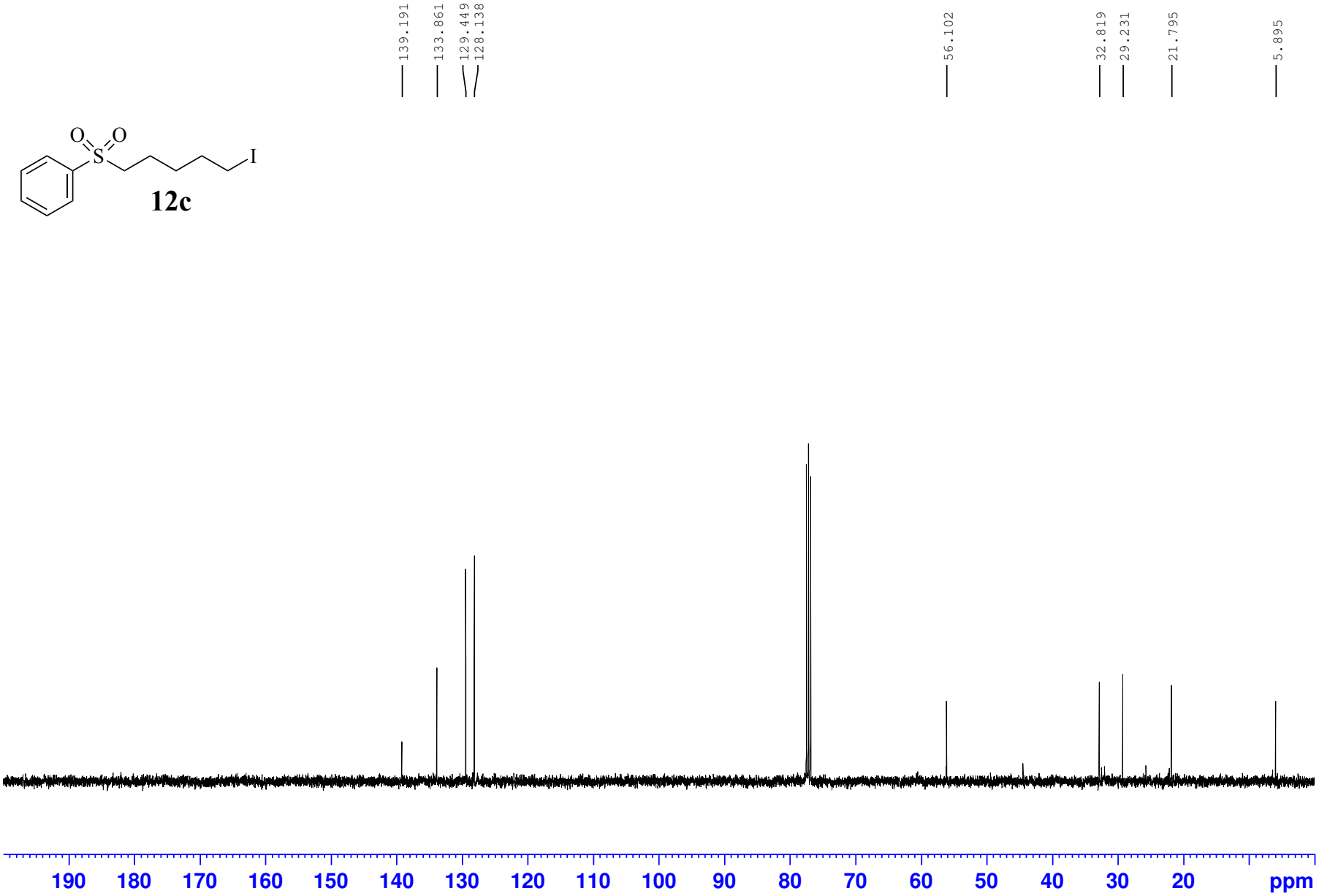
4.172
4.362
2.262

O
O
S
I

12c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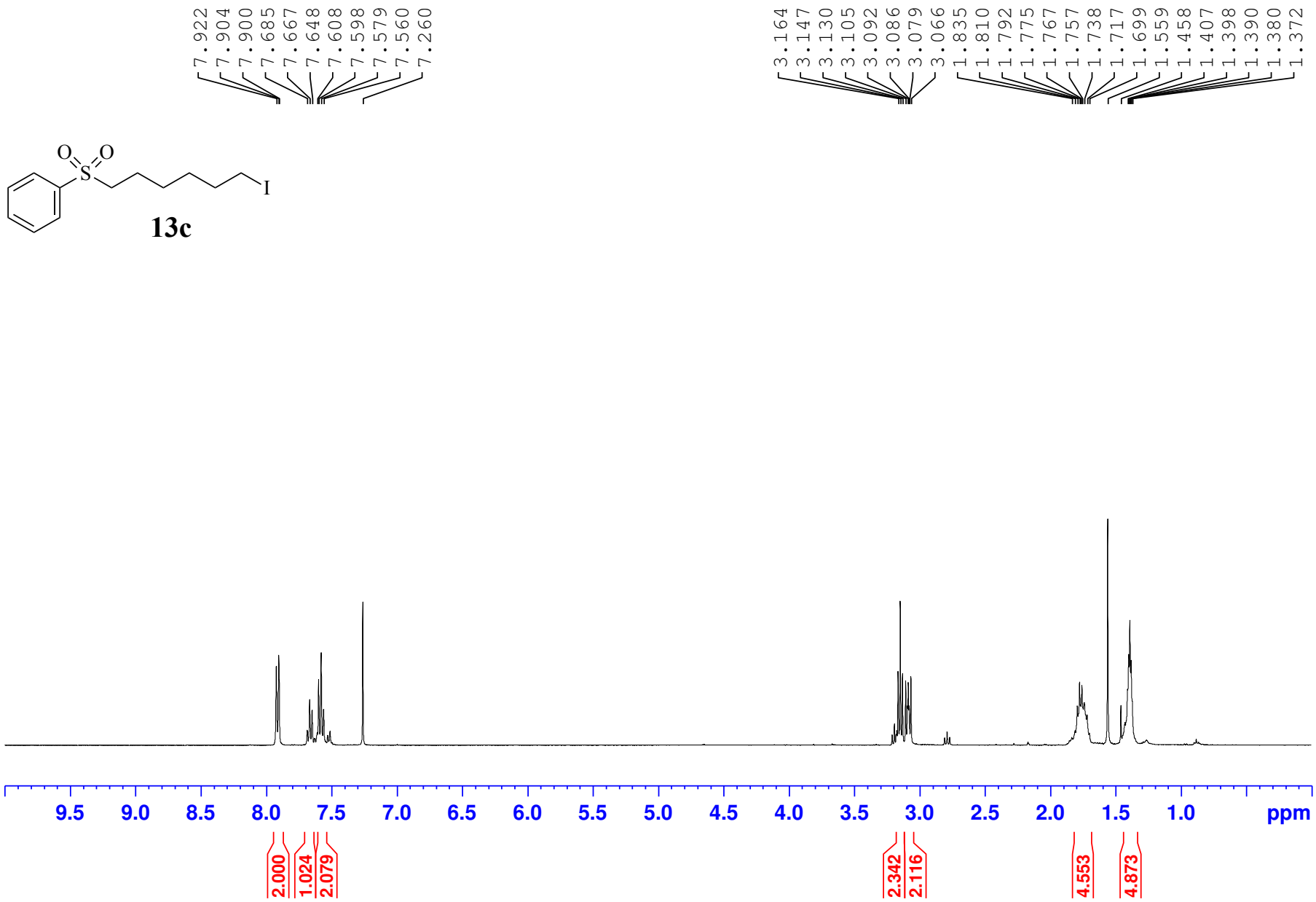
ppm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7.900
7.685
7.667
7.648
7.608
7.598
7.579
7.560
7.260
3.164
3.147
3.130
3.105
3.092
3.086
3.079
3.066
1.835
1.810
1.792
1.775
1.767
1.757
1.738
1.717
1.699
1.559
1.458
1.407
1.398
1.390
1.380
1.372
O
O
S
I
13c
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9.0
8.5
8.0
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
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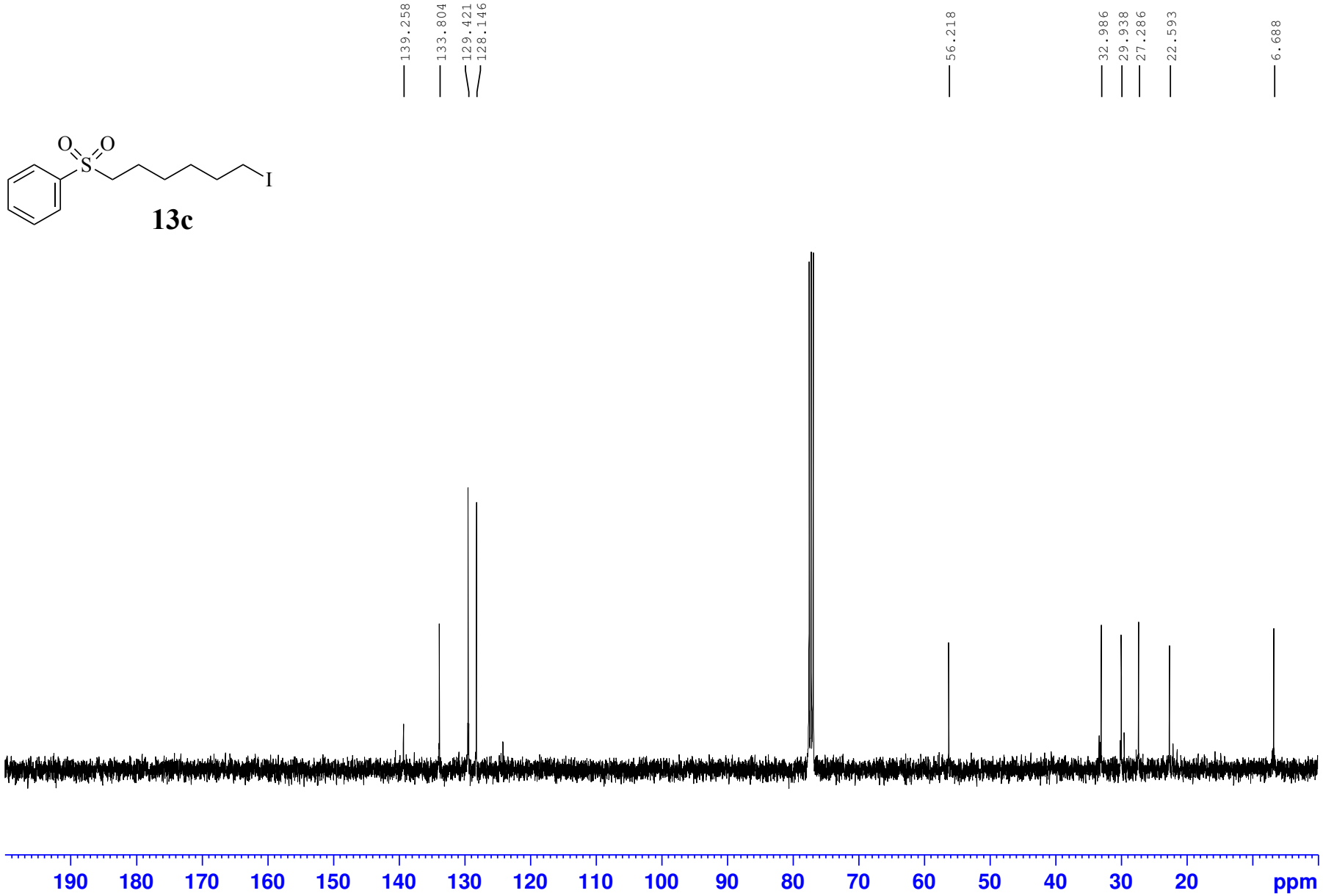
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27.286
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6.688
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O
S
I
13c
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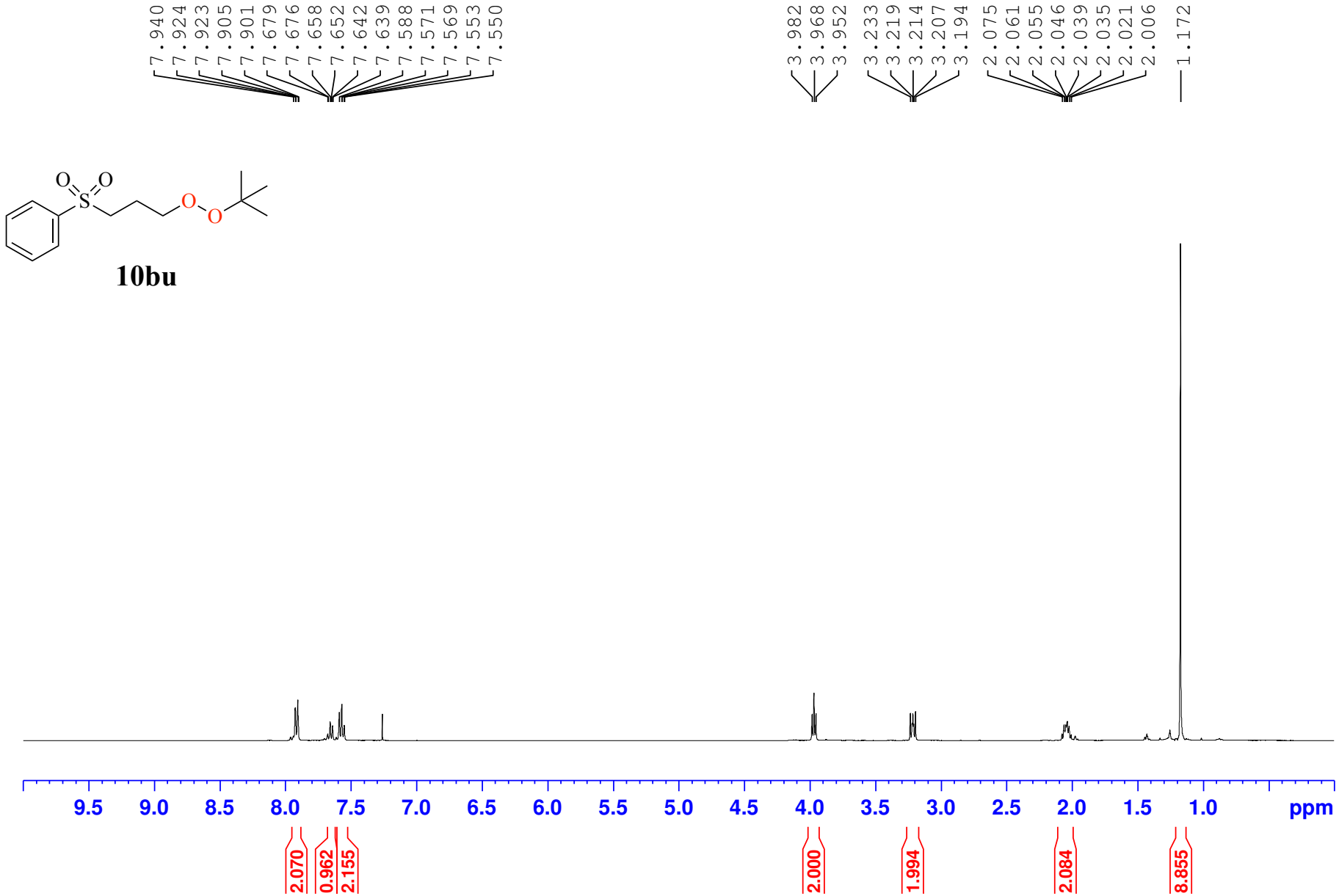
50
40
30
20
ppm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7.905
7.901
7.679
7.676
7.658
7.652
7.642
7.639
7.588
7.571
7.569
7.553
7.550
3.982
3.968
3.952
3.233
3.219
3.214
3.207
3.194
2.075
2.061
2.055
2.046
2.039
2.035
2.021
2.006
1.172
O
O
S
O
O
10bu
9.5
9.0
8.5
8.0
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
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1.0
ppm
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2.000
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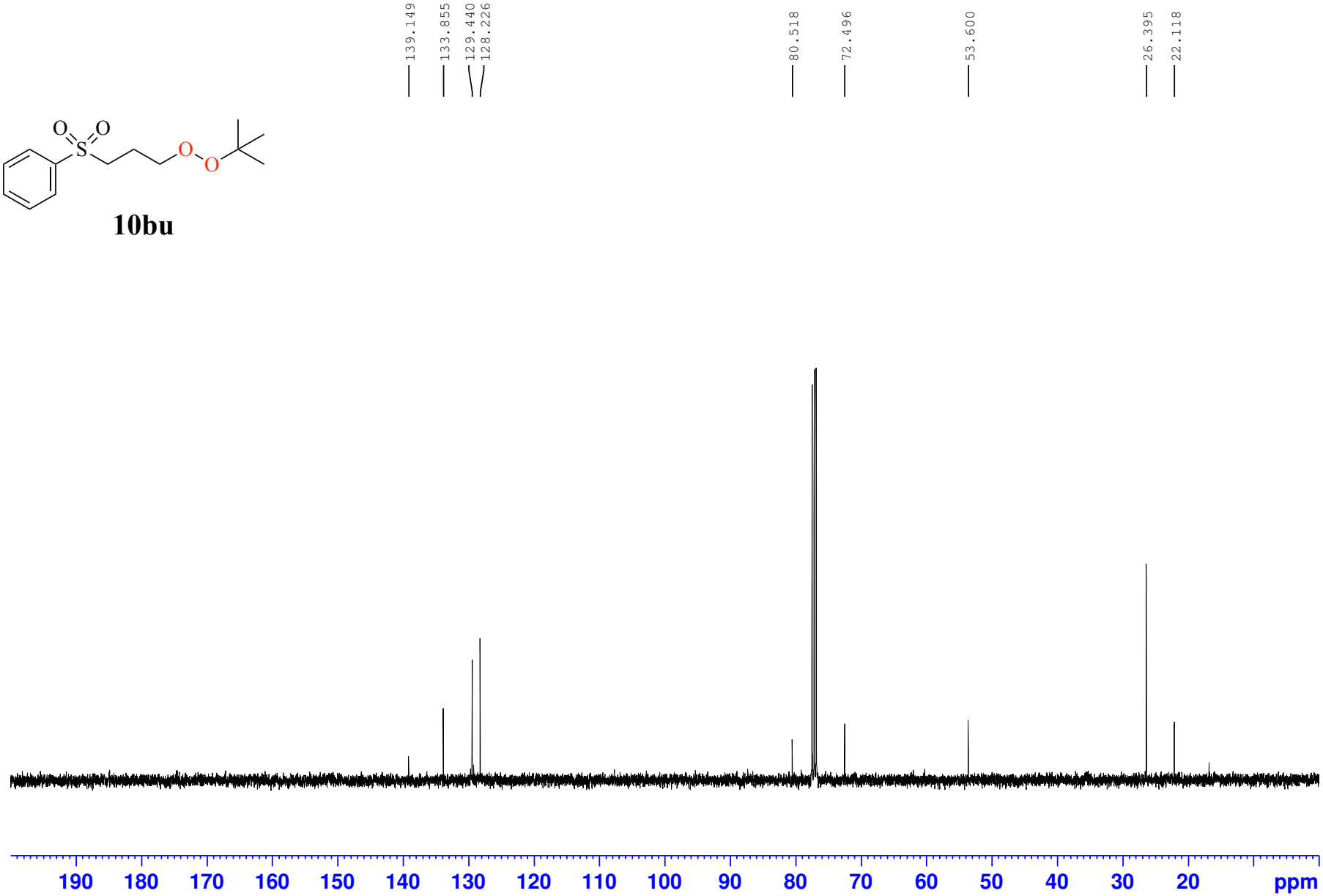
2.084
8.855

10bu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72.496
53.600
26.395
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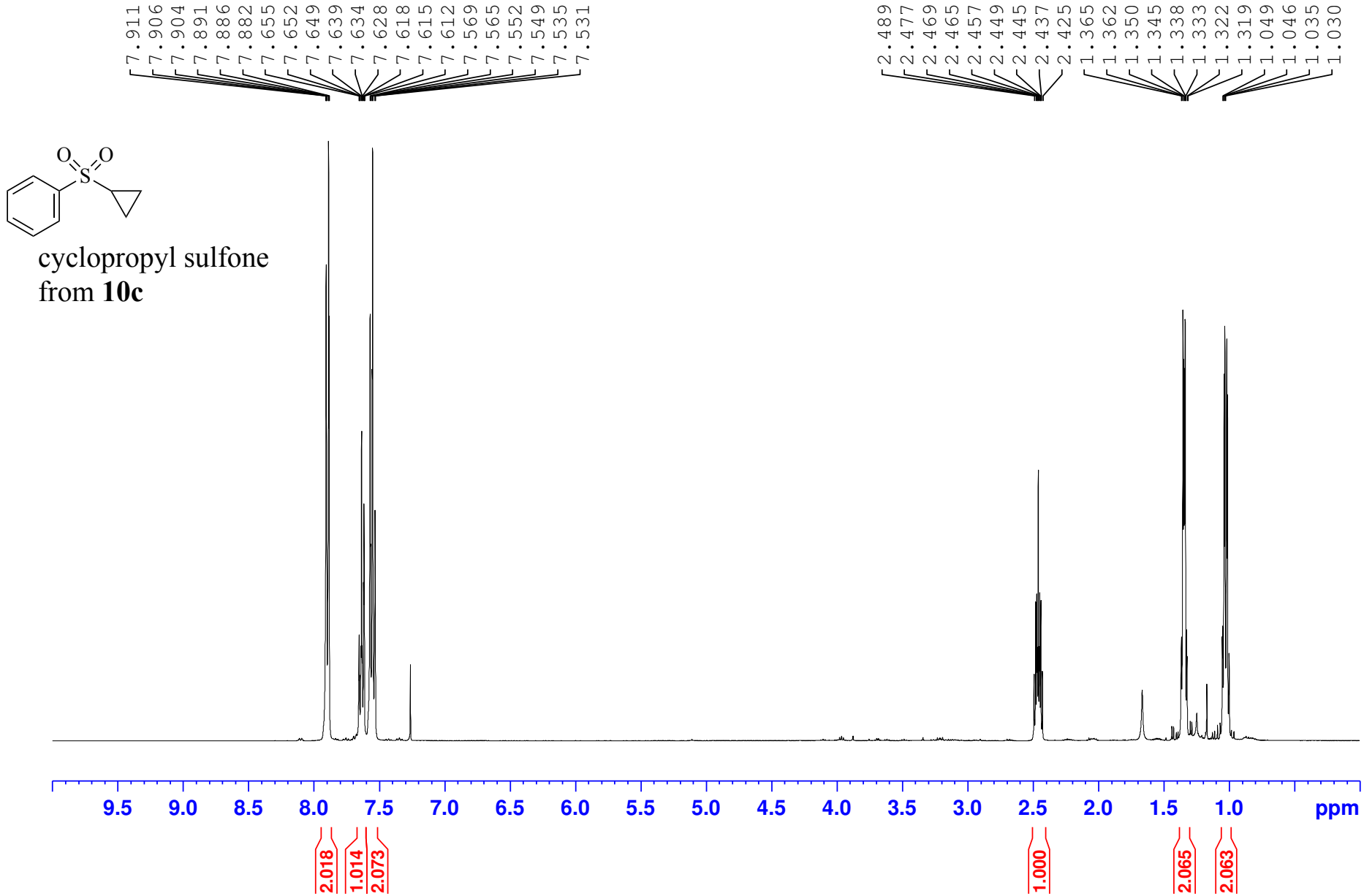
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
ppm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7.652
7.649
7.639
7.634
7.628
7.618
7.615
7.612
7.569
7.565
7.552
7.549
7.535
7.531
2.489
2.477
2.469
2.465
2.457
2.449
2.445
2.437
2.425
1.365
1.362
1.350
1.345
1.338
1.333
1.322
1.319
1.049
1.046
1.035
1.030
O
O
S
cyclopropyl sulfone
from 10c
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
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1.0
ppm
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1.000
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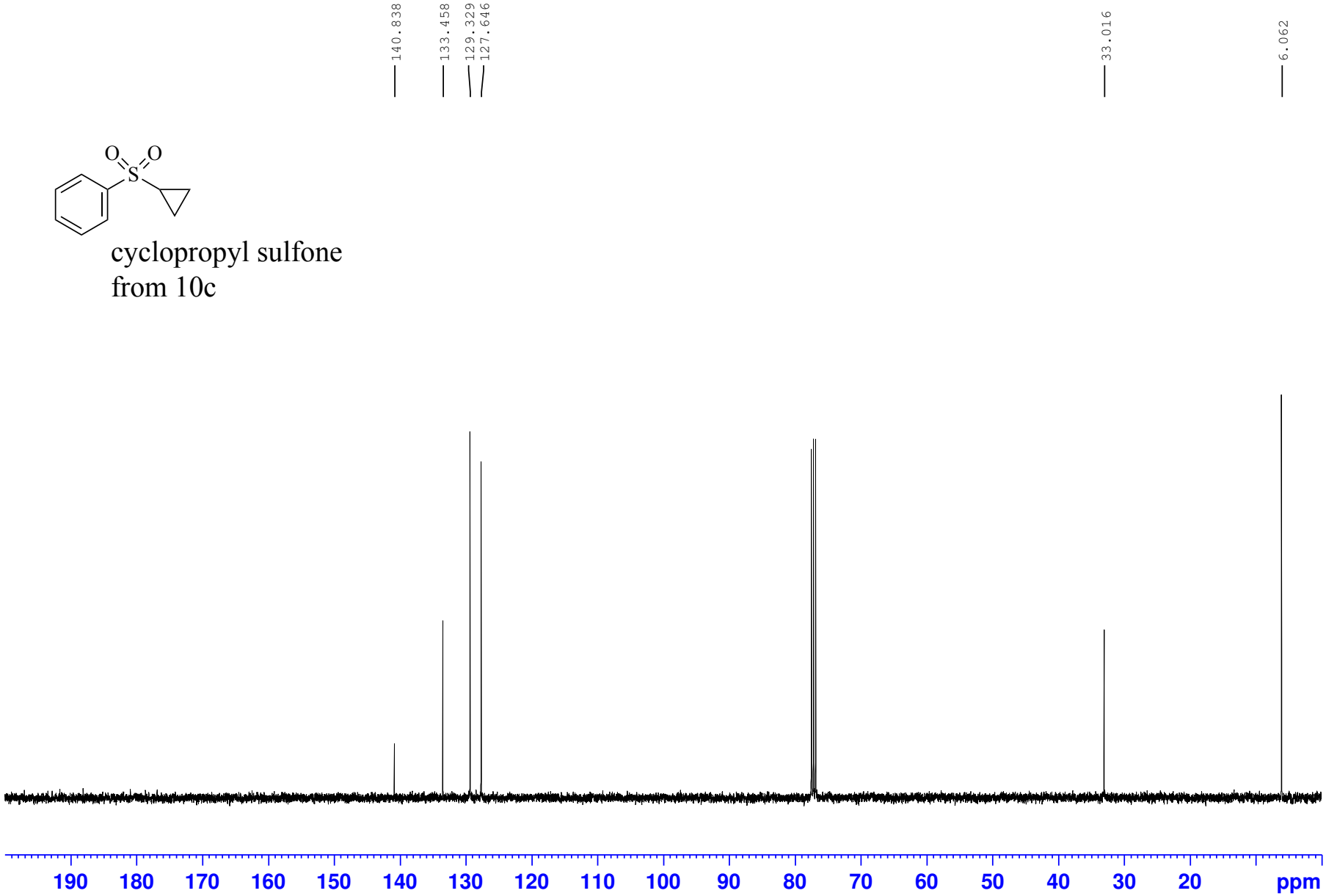
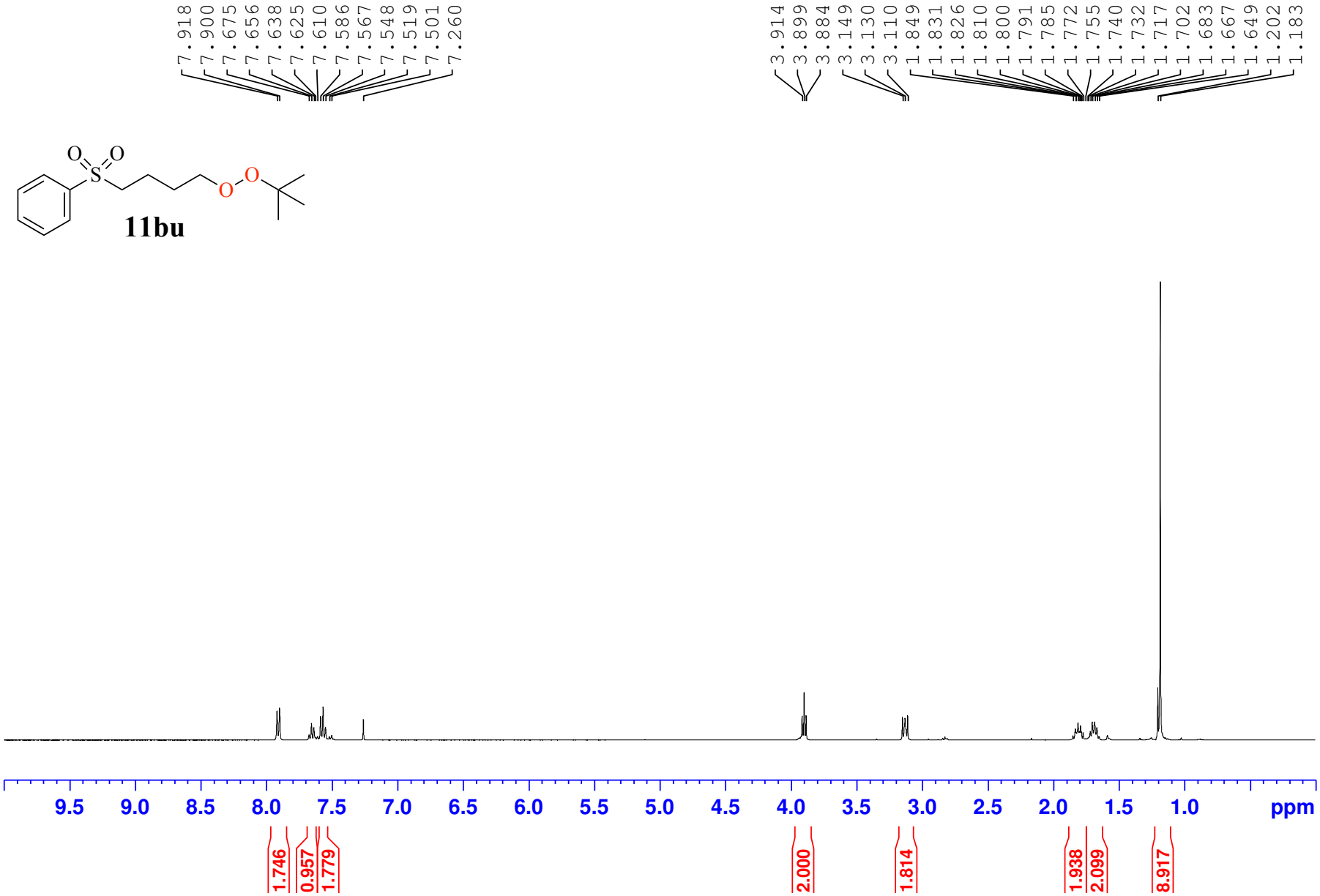
2.063

cyclopropyl sulfone
from 10c

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7.586
7.567
7.548
7.519
7.501
7.260
3.914
3.899
3.884
3.149
3.130
3.110
1.849
1.831
1.826
1.810
1.800
1.791
1.785
1.772
1.755
1.740
1.732
1.717
1.702
1.683
1.667
1.649
1.202
1.183
11bu
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
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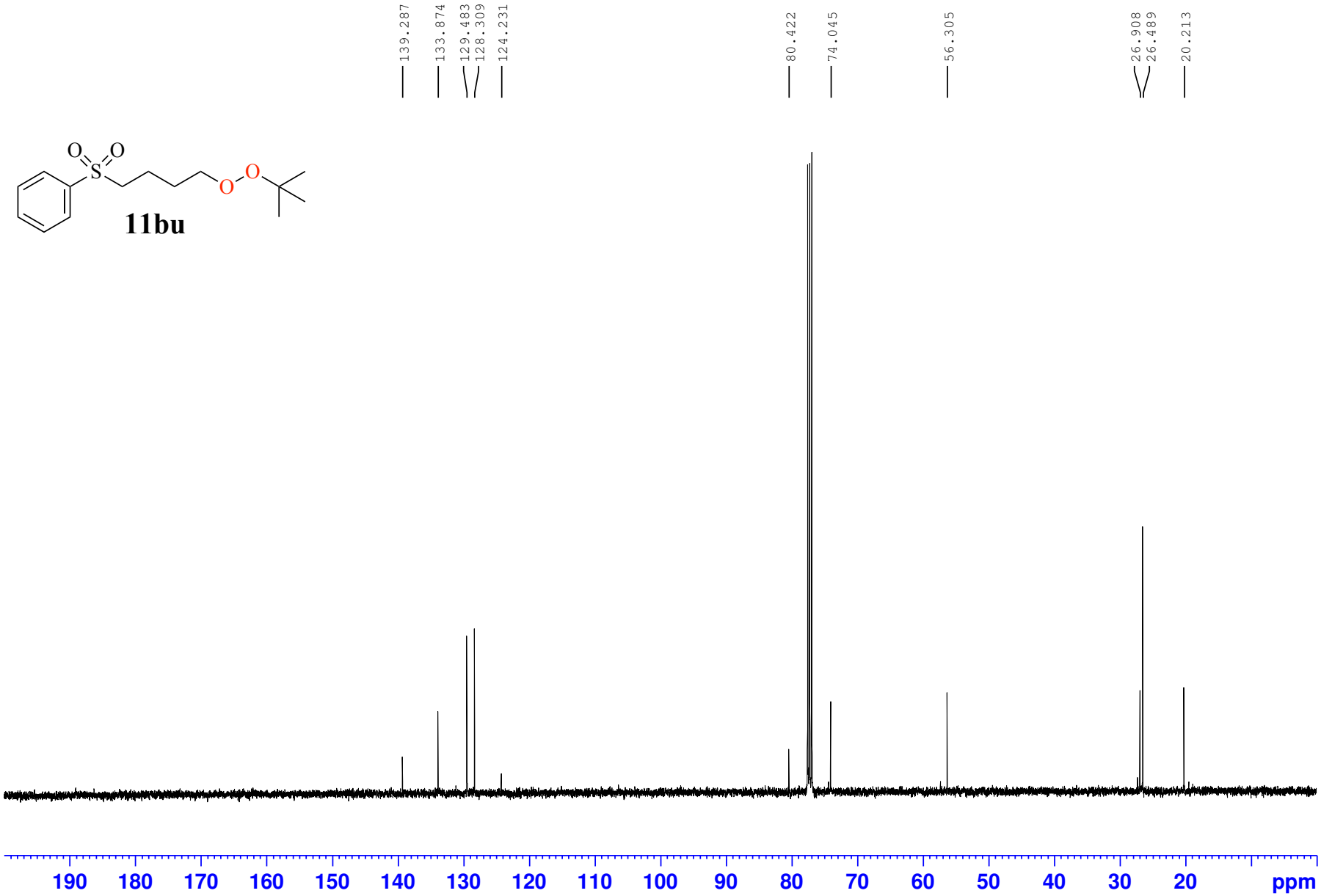
8.917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26.489
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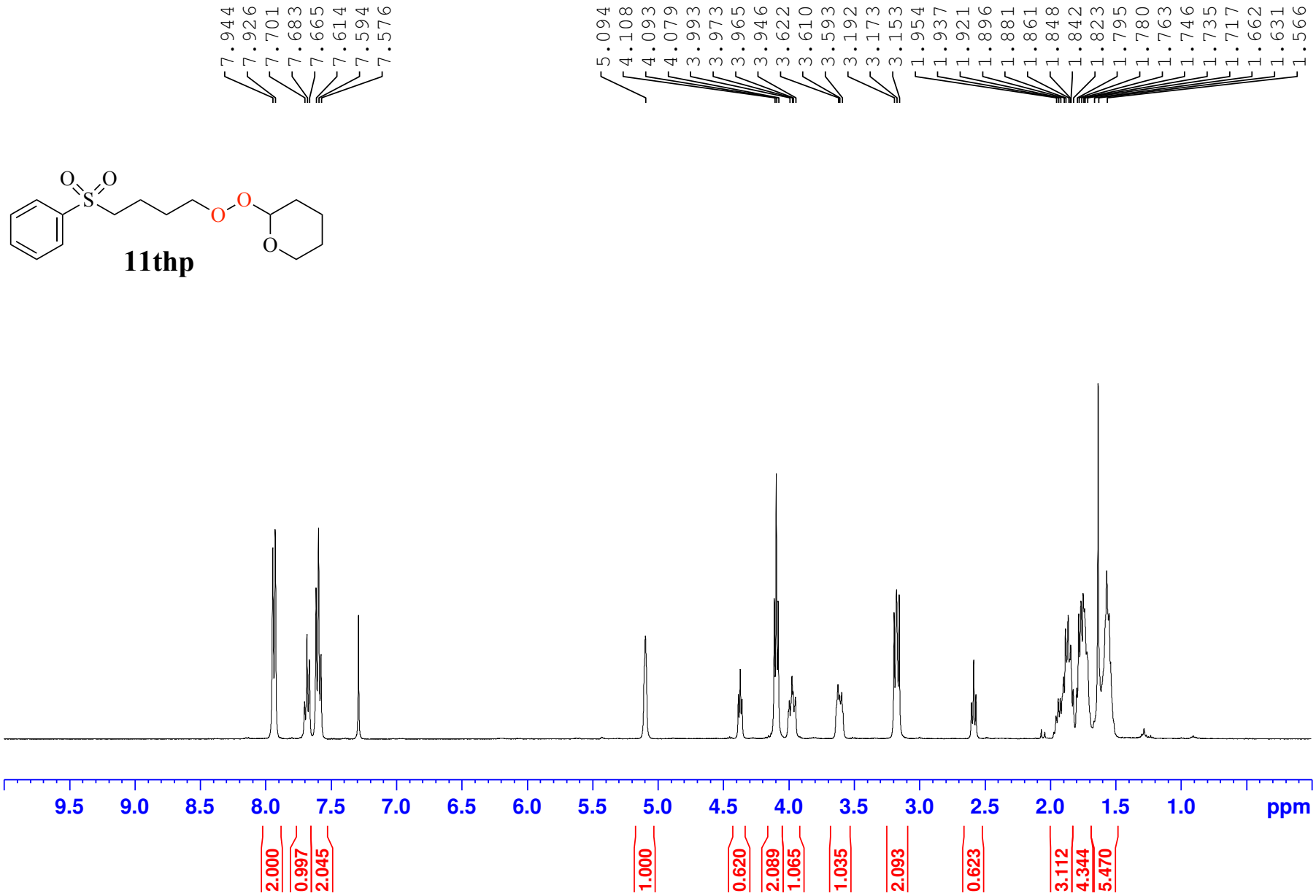
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
ppm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7.576
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4.093
4.079
3.993
3.973
3.965
3.946
3.622
3.610
3.593
3.192
3.173
3.153
1.954
1.937
1.921
1.896
1.881
1.861
1.848
1.842
1.823
1.795
1.780
1.763
1.746
1.735
1.717
1.662
1.631
1.566
O
O
S
O
O
O
11thp
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
ppm
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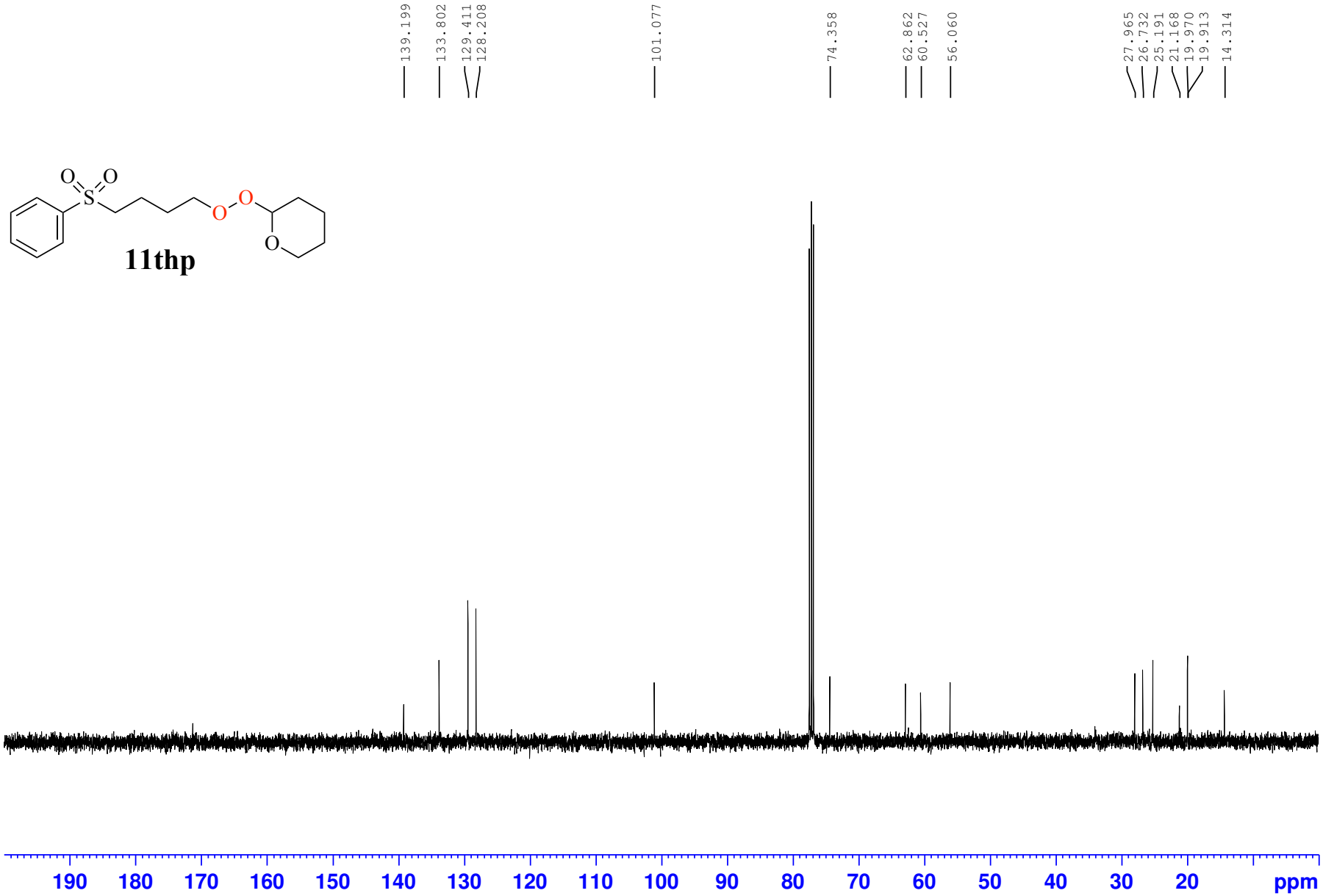
5.470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26.732
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21.168
19.970
19.913
14.314
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150
140
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80
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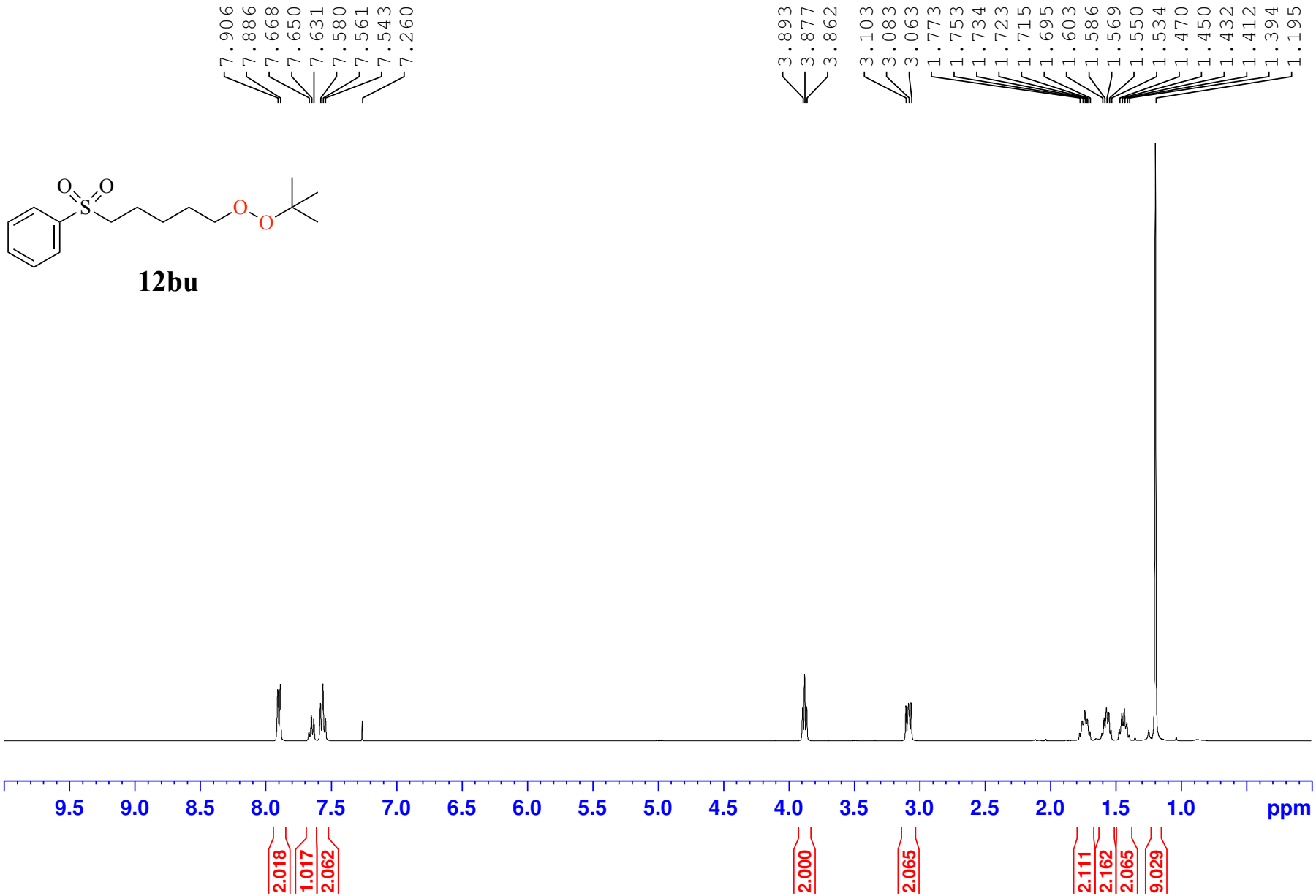
40
30
20
ppm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3.083
3.063
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1.753
1.734
1.723
1.715
1.695
1.603
1.586
1.569
1.550
1.534
1.470
1.450
1.432
1.412
1.394
1.195
O
O
S
O
O
12bu
9.5
9.0
8.5
8.0
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
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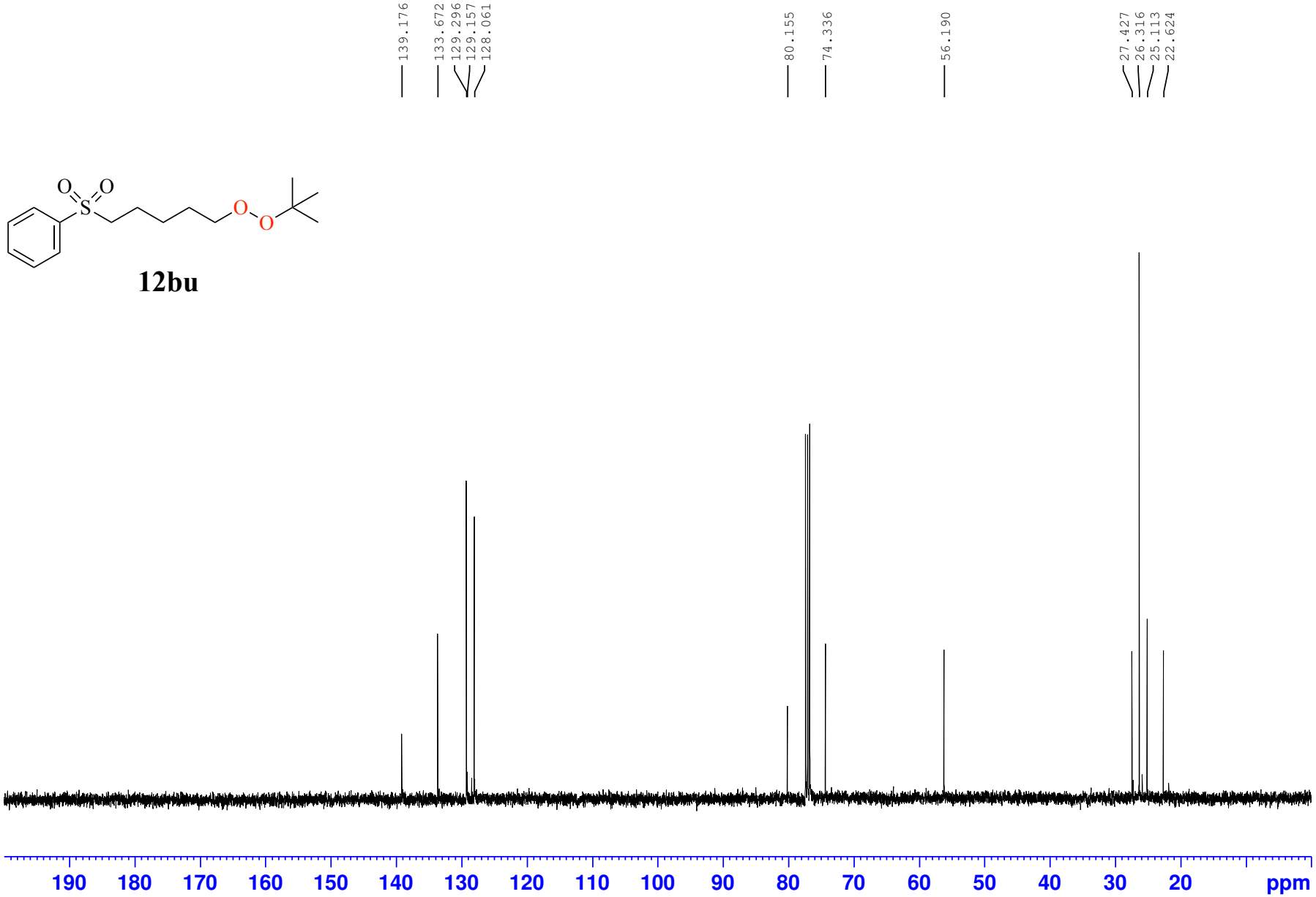
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80
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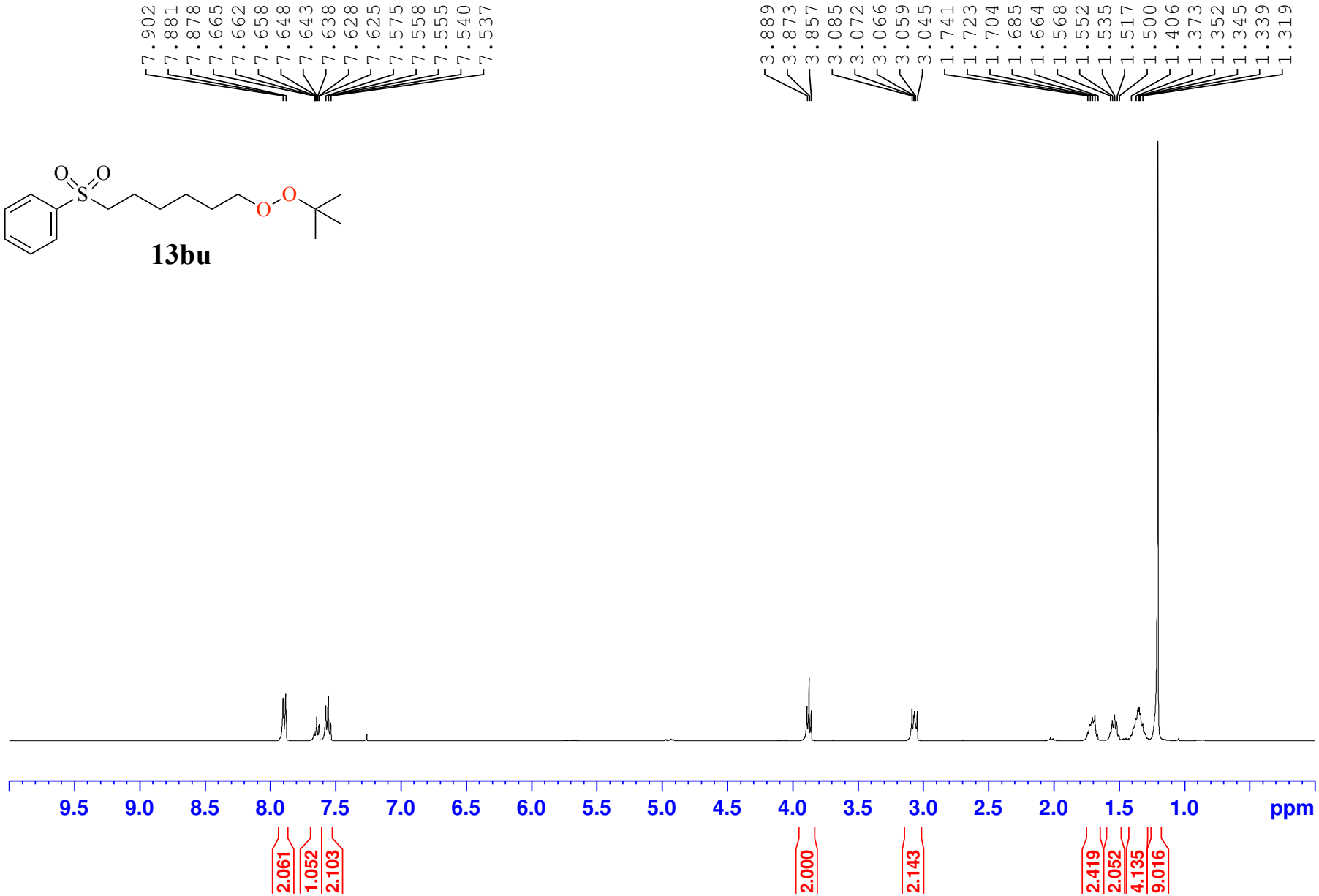
40
30
20
ppm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7.555
7.540
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3.857
3.085
3.072
3.066
3.059
3.045
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1.723
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1.685
1.664
1.568
1.552
1.535
1.517
1.500
1.406
1.373
1.352
1.345
1.339
1.319
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6.0
5.5
5.0
4.5
4.0
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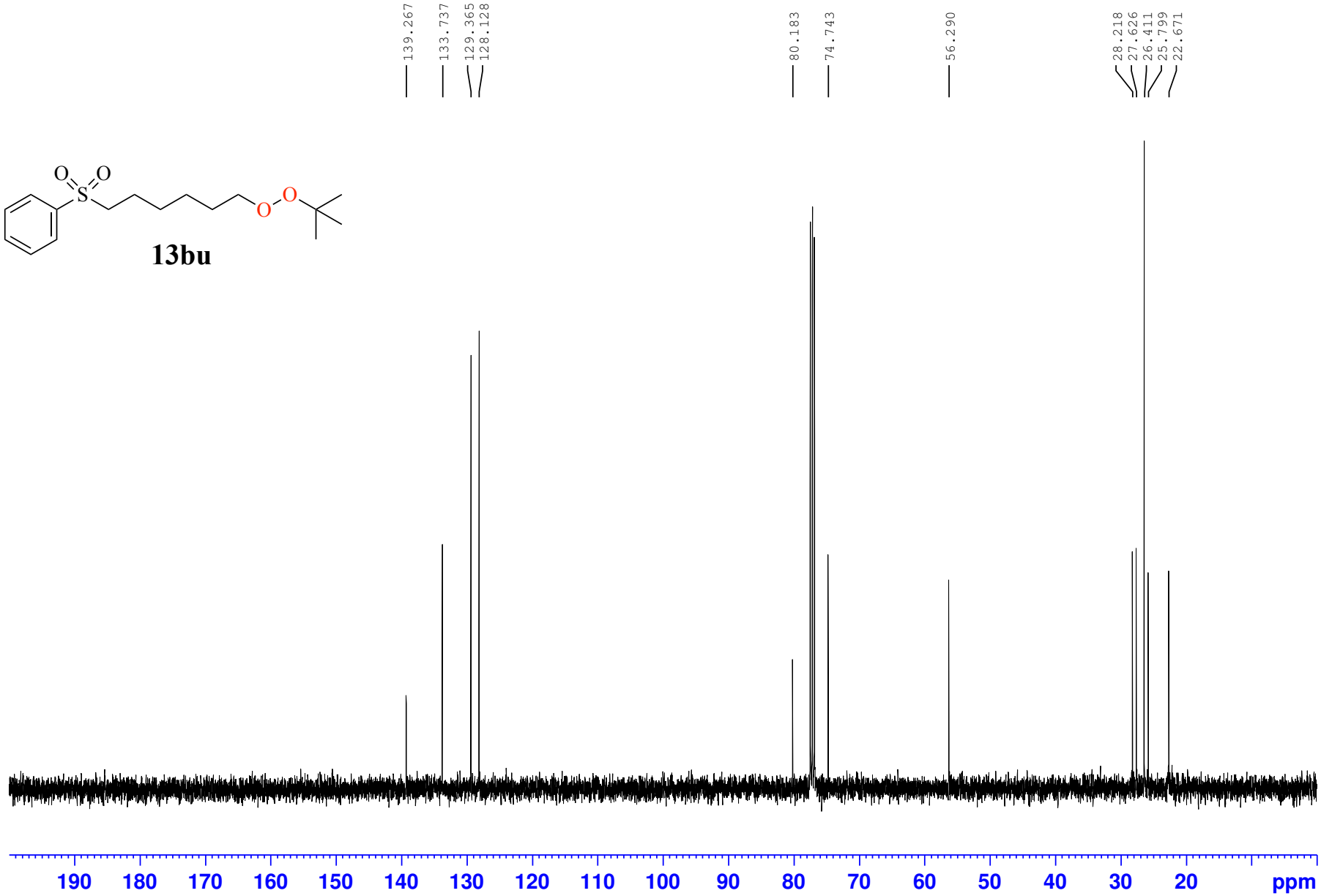
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22.671
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80
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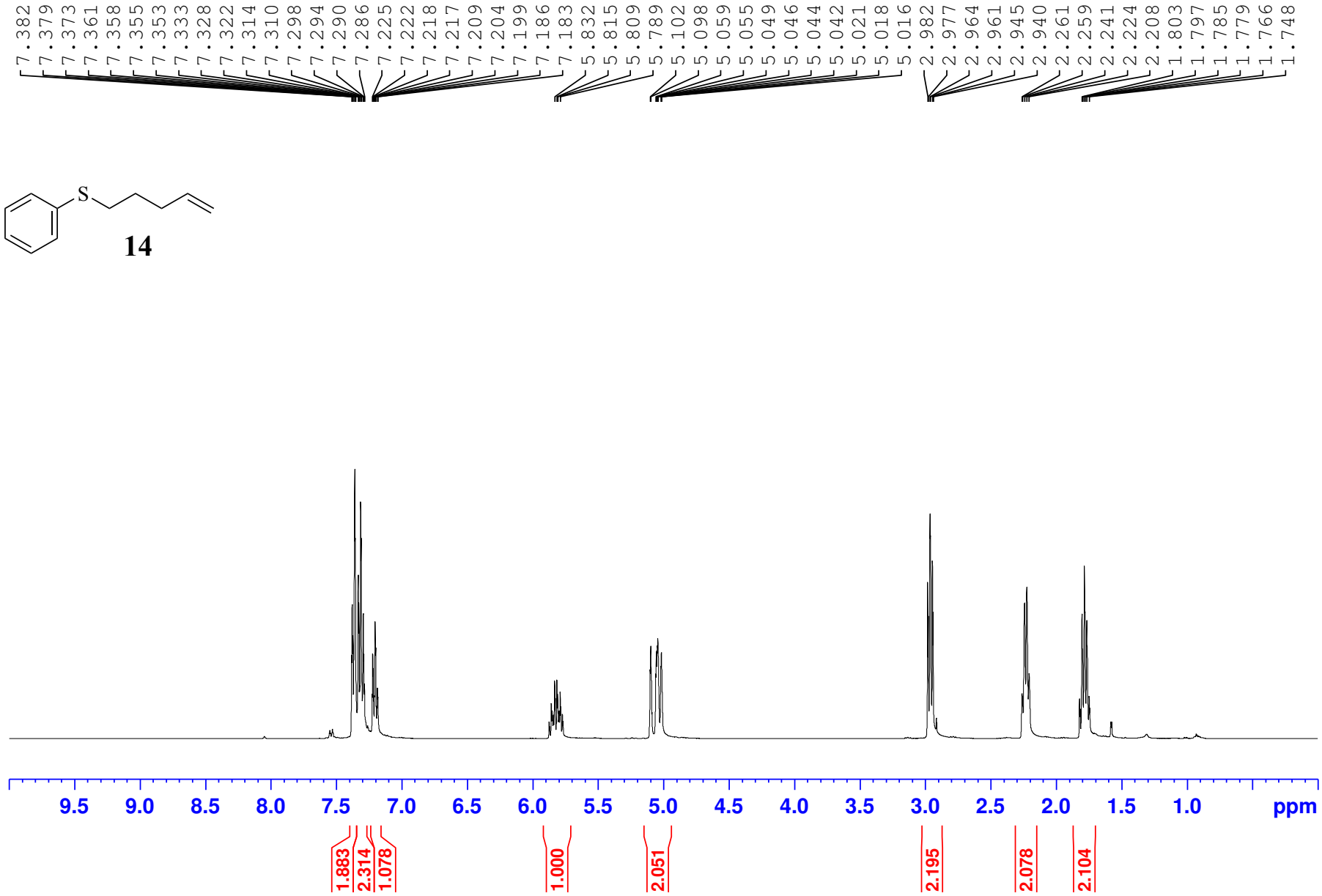
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7.218
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7.204
7.199
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5.815
5.809
5.789
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5.059
5.055
5.049
5.046
5.044
5.042
5.021
5.018
5.016
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2.945
2.940
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2.259
2.241
2.224
2.208
1.803
1.797
1.785
1.779
1.766
1.748
S
14
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9.0
8.5
8.0
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
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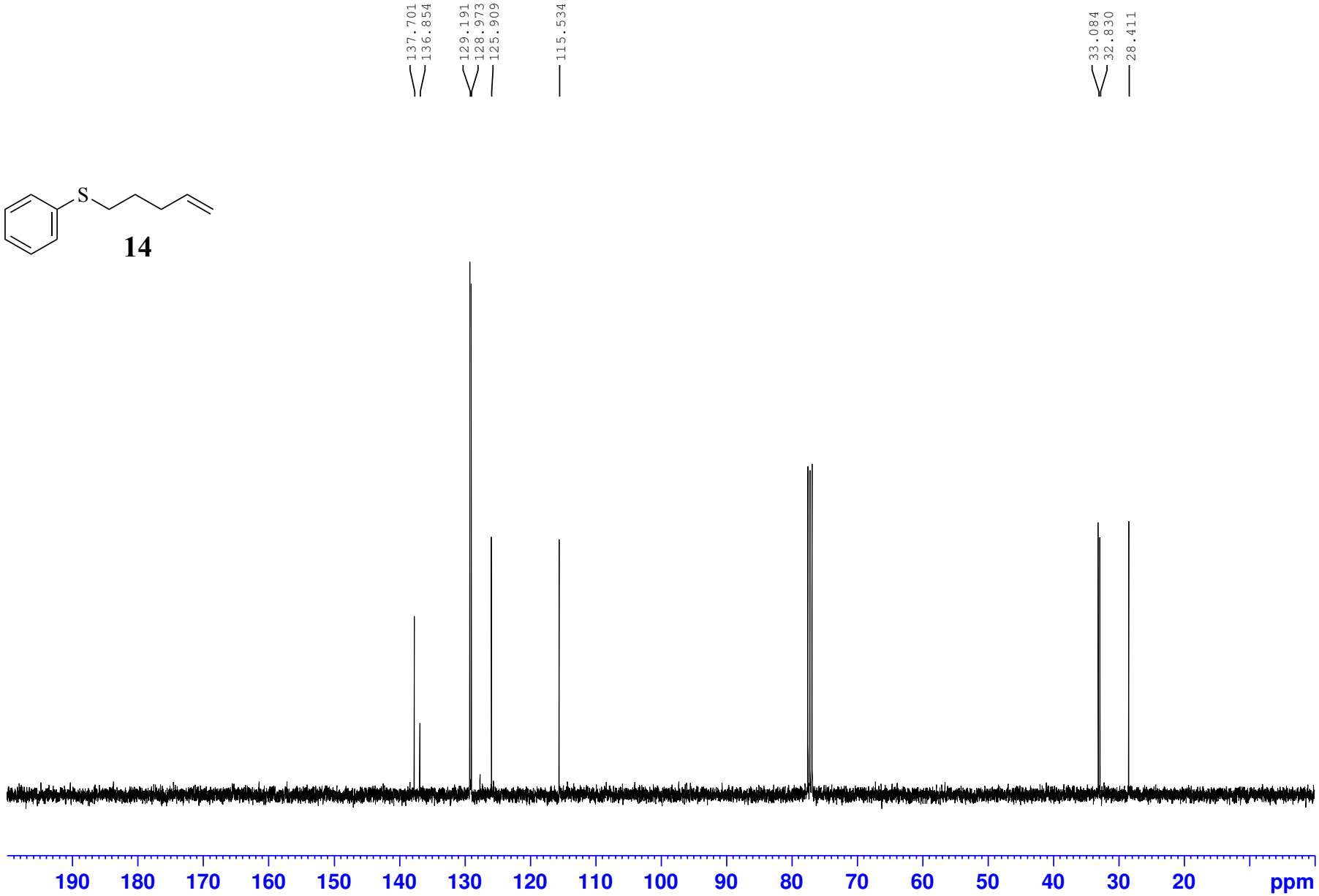
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180
170
160
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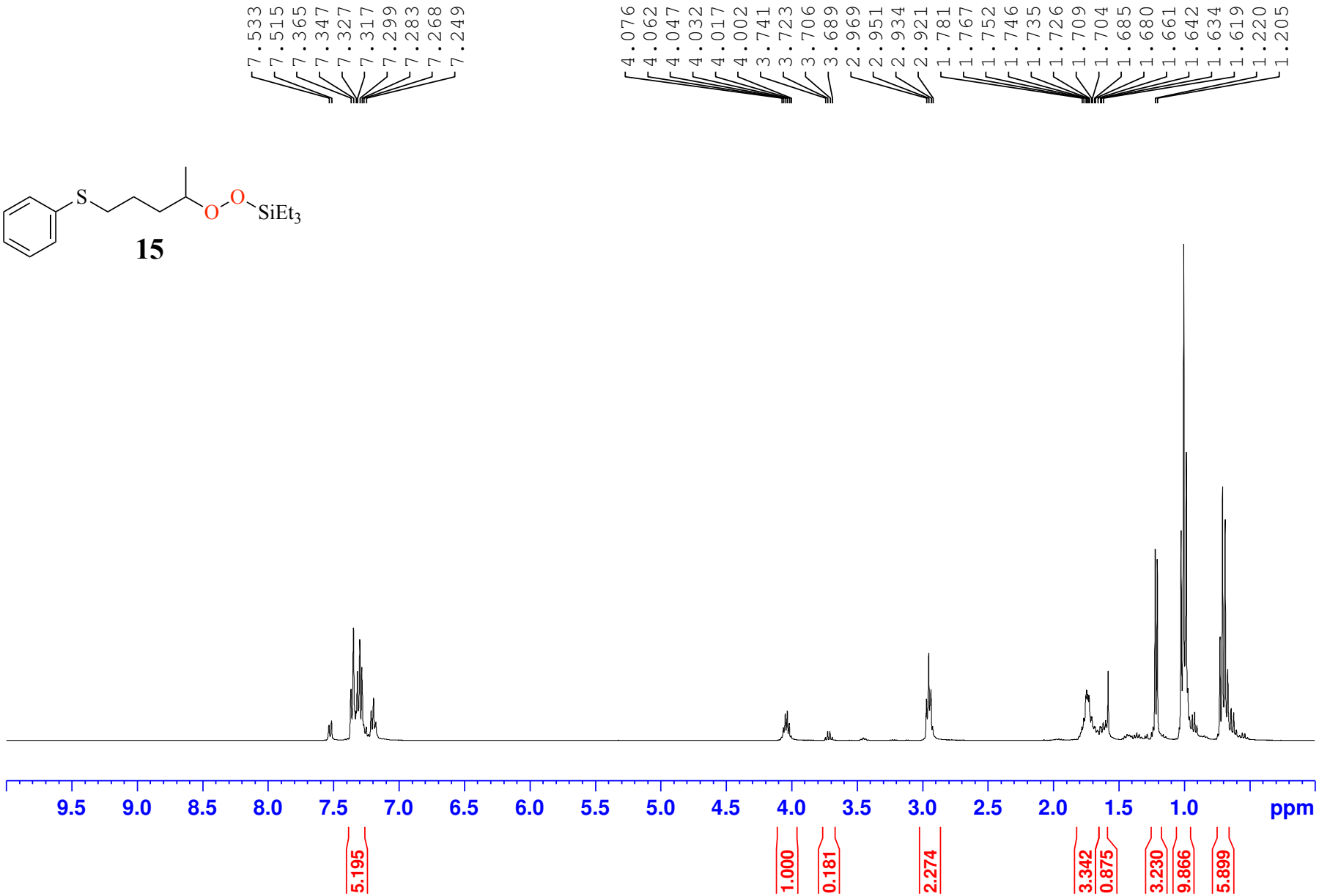
100
90
80
70
60
50
40
30
20
ppm

S
O
O
SiEt3

15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4.047
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4.002
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2.951
2.934
2.921
1.781
1.767
1.752
1.746
1.735
1.726
1.709
1.704
1.685
1.680
1.661
1.642
1.634
1.619
1.220
1.205
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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
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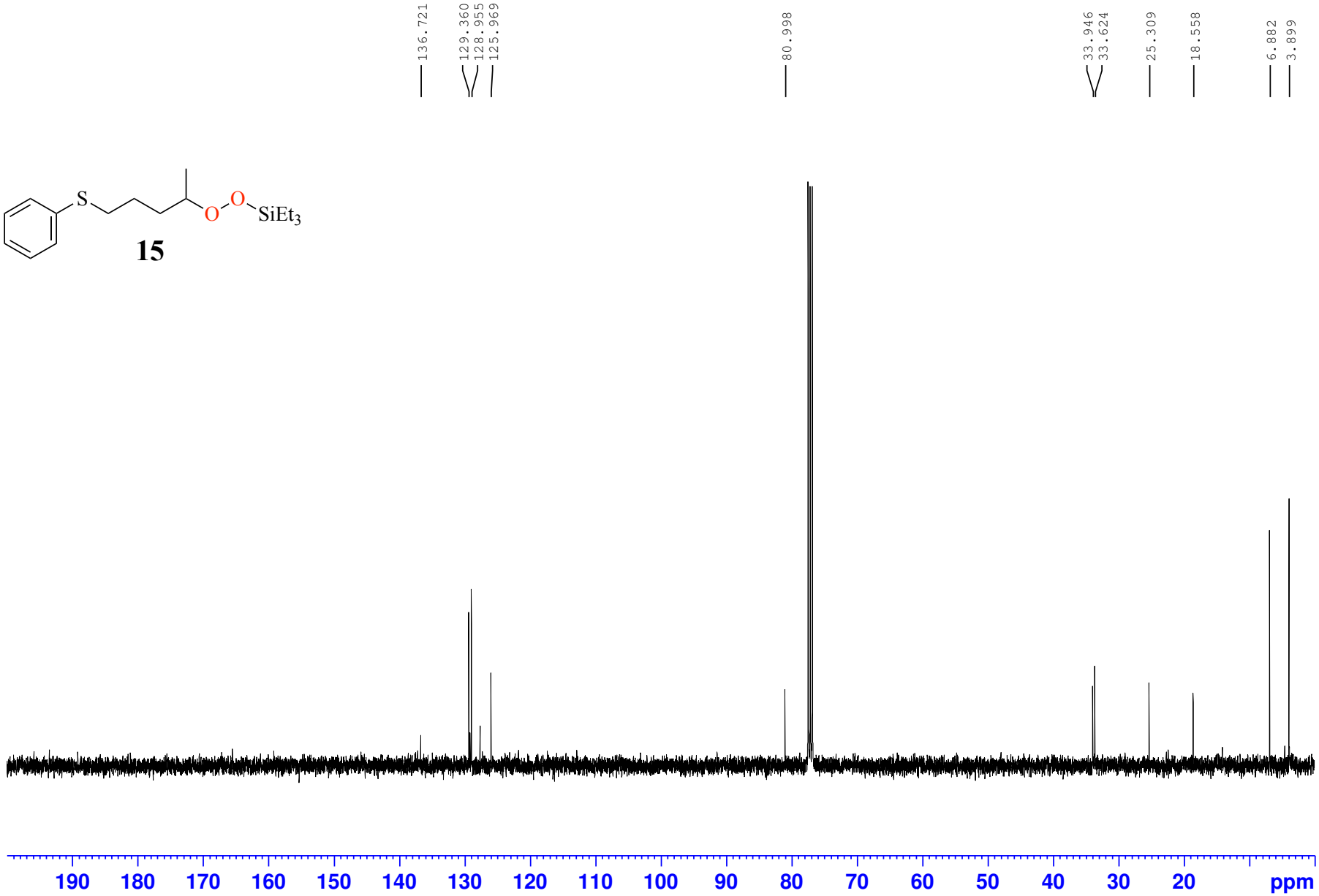
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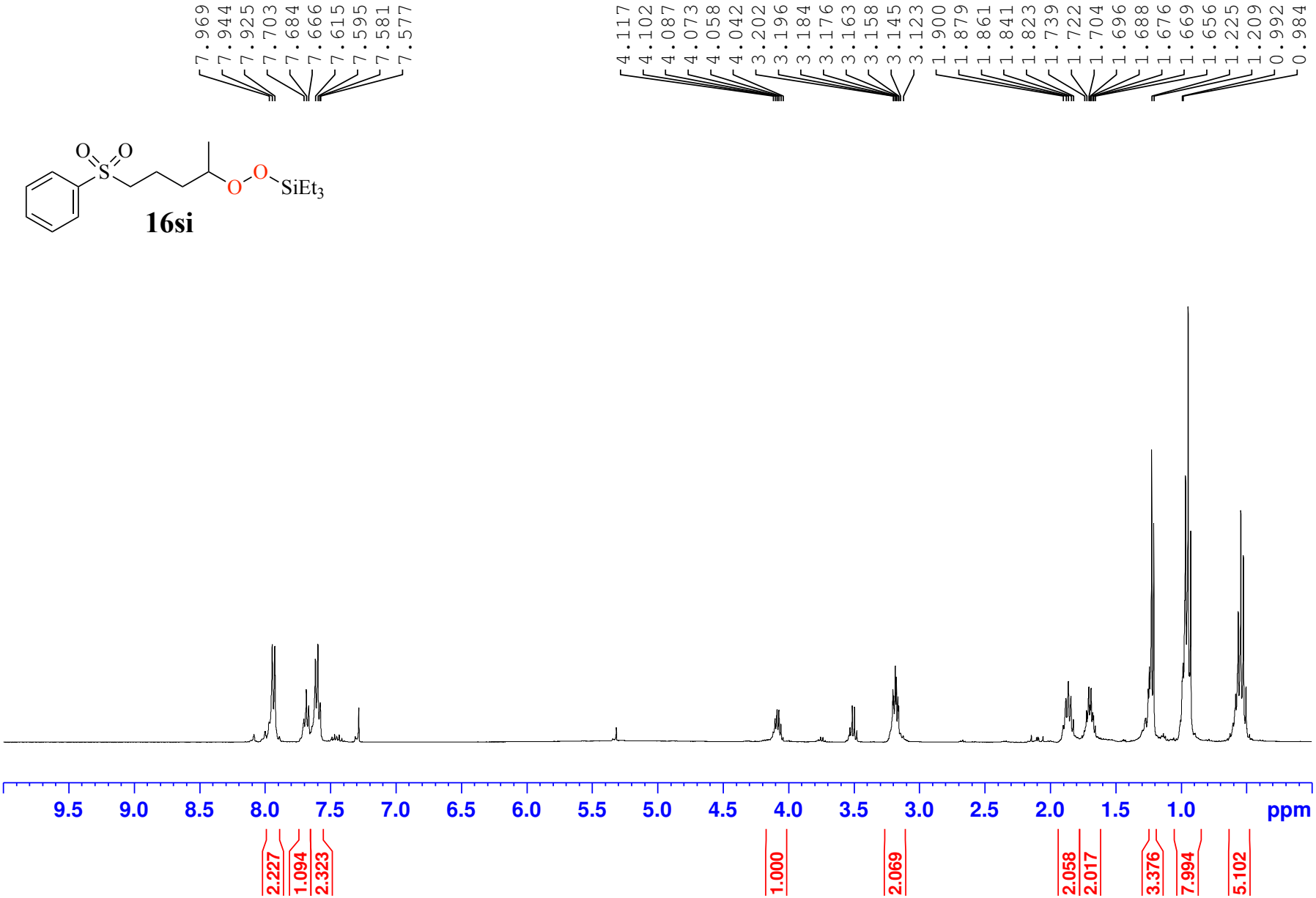
80.998
33.946
33.624
25.309
18.558
6.882
3.899
S
O
O
SiEt3
15
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
ppm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4.087
4.073
4.058
4.042
3.202
3.196
3.184
3.176
3.163
3.158
3.145
3.123
1.900
1.879
1.861
1.841
1.823
1.739
1.722
1.704
1.696
1.688
1.676
1.669
1.656
1.225
1.209
0.992
0.984
O
O
S
O
O
SiEt3
16si
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
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2.058
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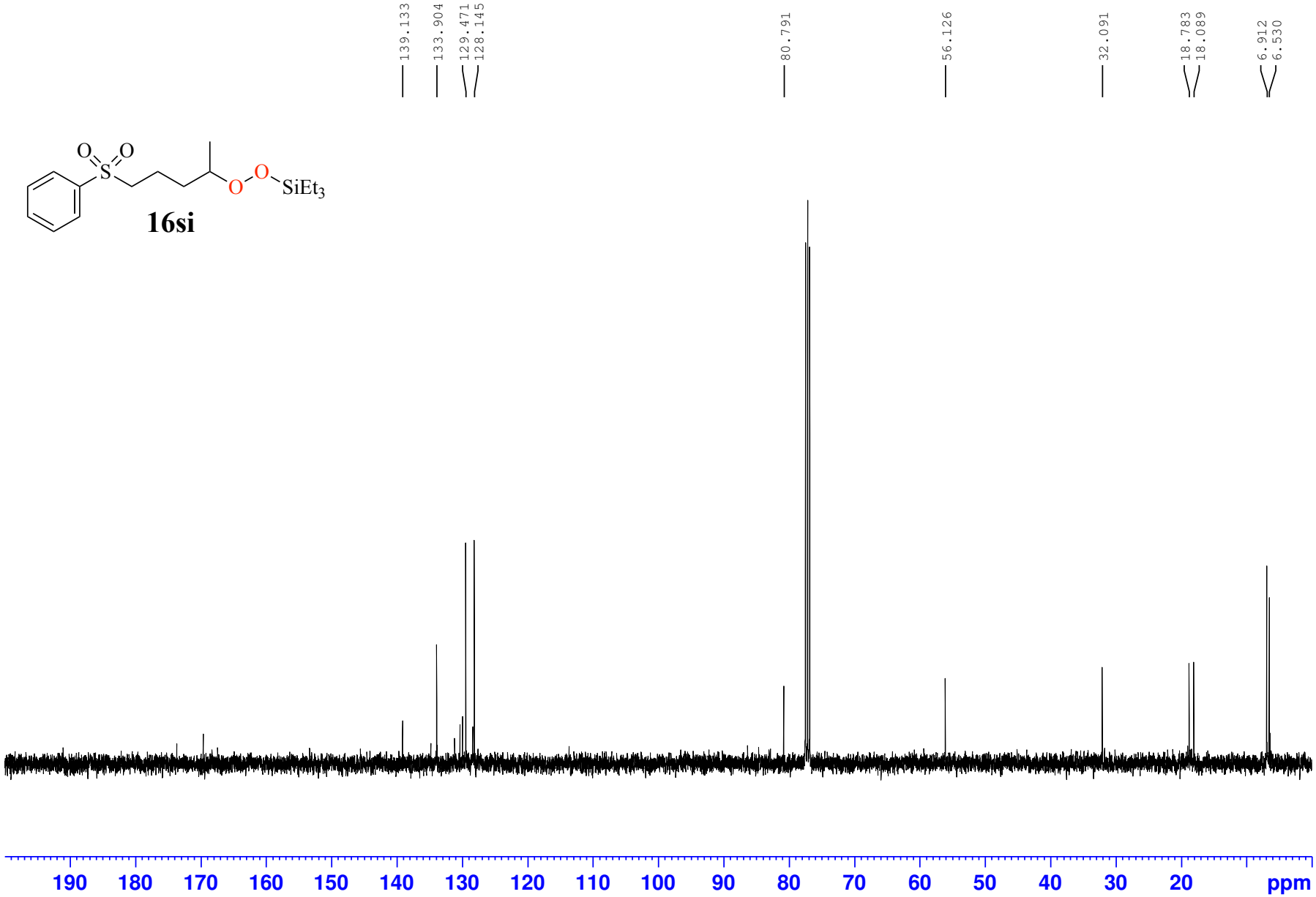
5.102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129.471
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6.912
6.530
O
O
S
O
O
SiEt3
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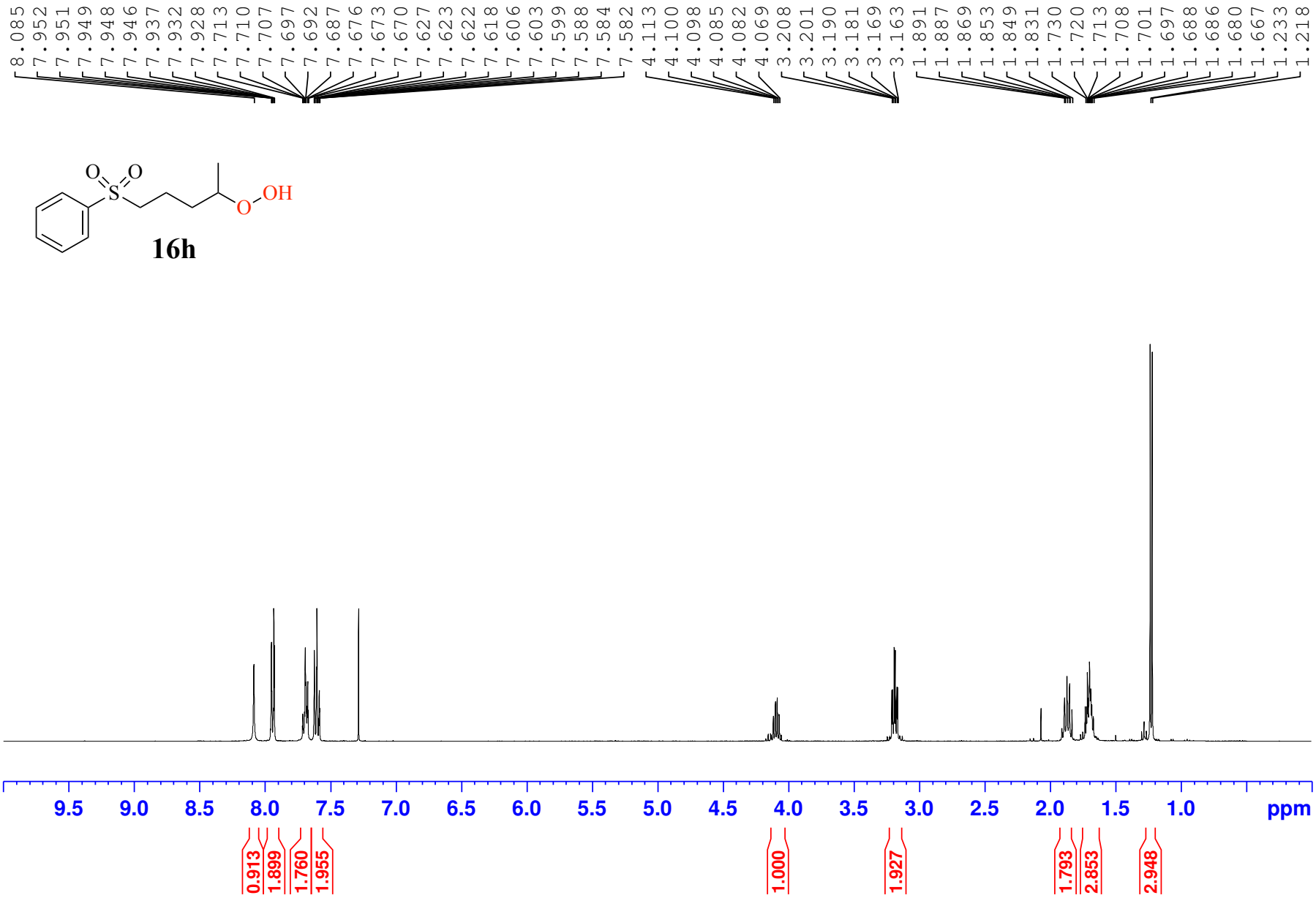
20
ppm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7.710
7.707
7.697
7.692
7.687
7.676
7.673
7.670
7.627
7.623
7.622
7.618
7.606
7.603
7.599
7.588
7.584
7.582
4.113
4.100
4.098
4.085
4.082
4.069
3.208
3.201
3.190
3.181
3.169
3.163
1.891
1.887
1.869
1.853
1.849
1.831
1.730
1.720
1.713
1.708
1.701
1.697
1.688
1.686
1.680
1.667
1.233
1.218
O
O
S
O
OH
16h
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
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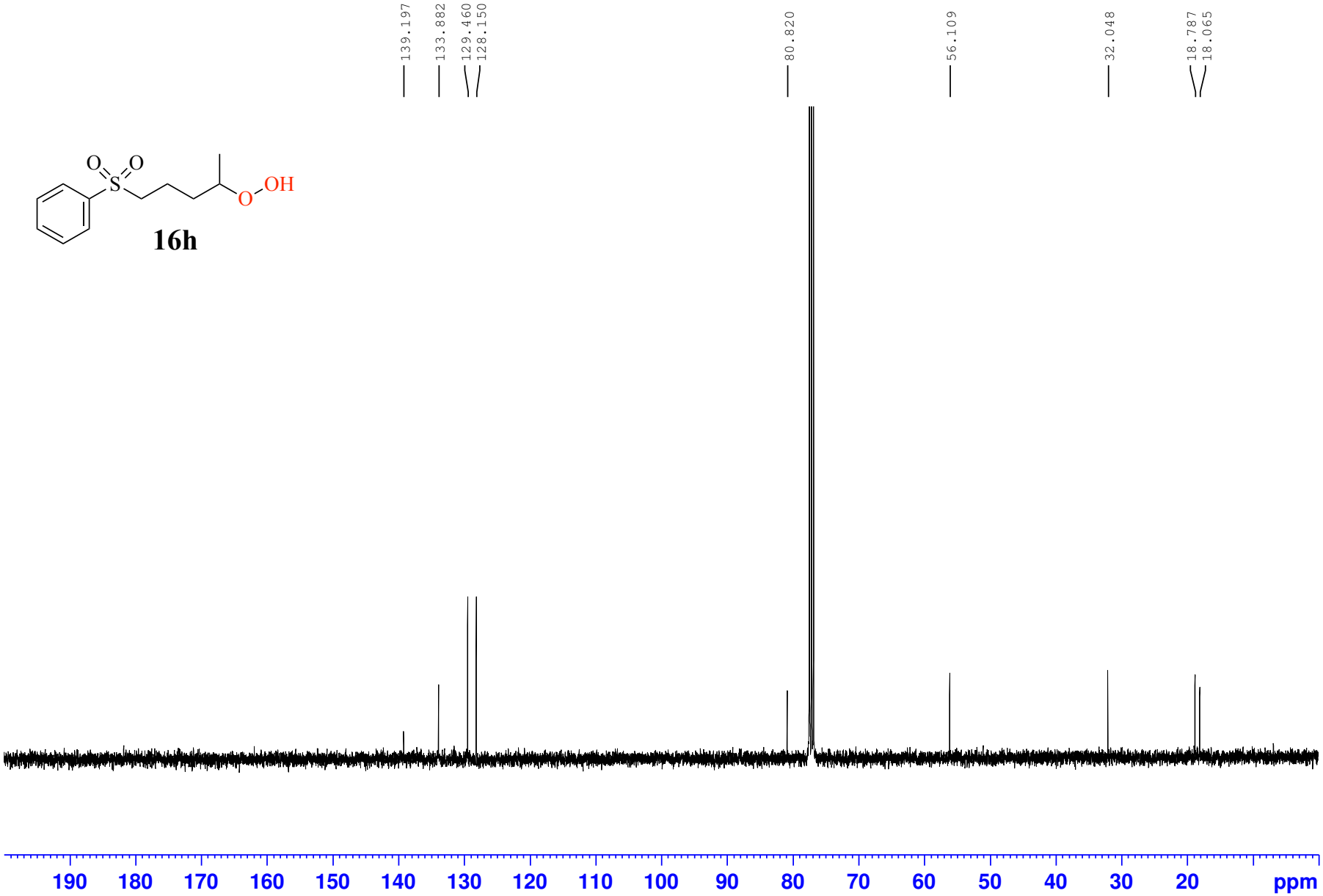
2.948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O
S
O
OH
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80
70
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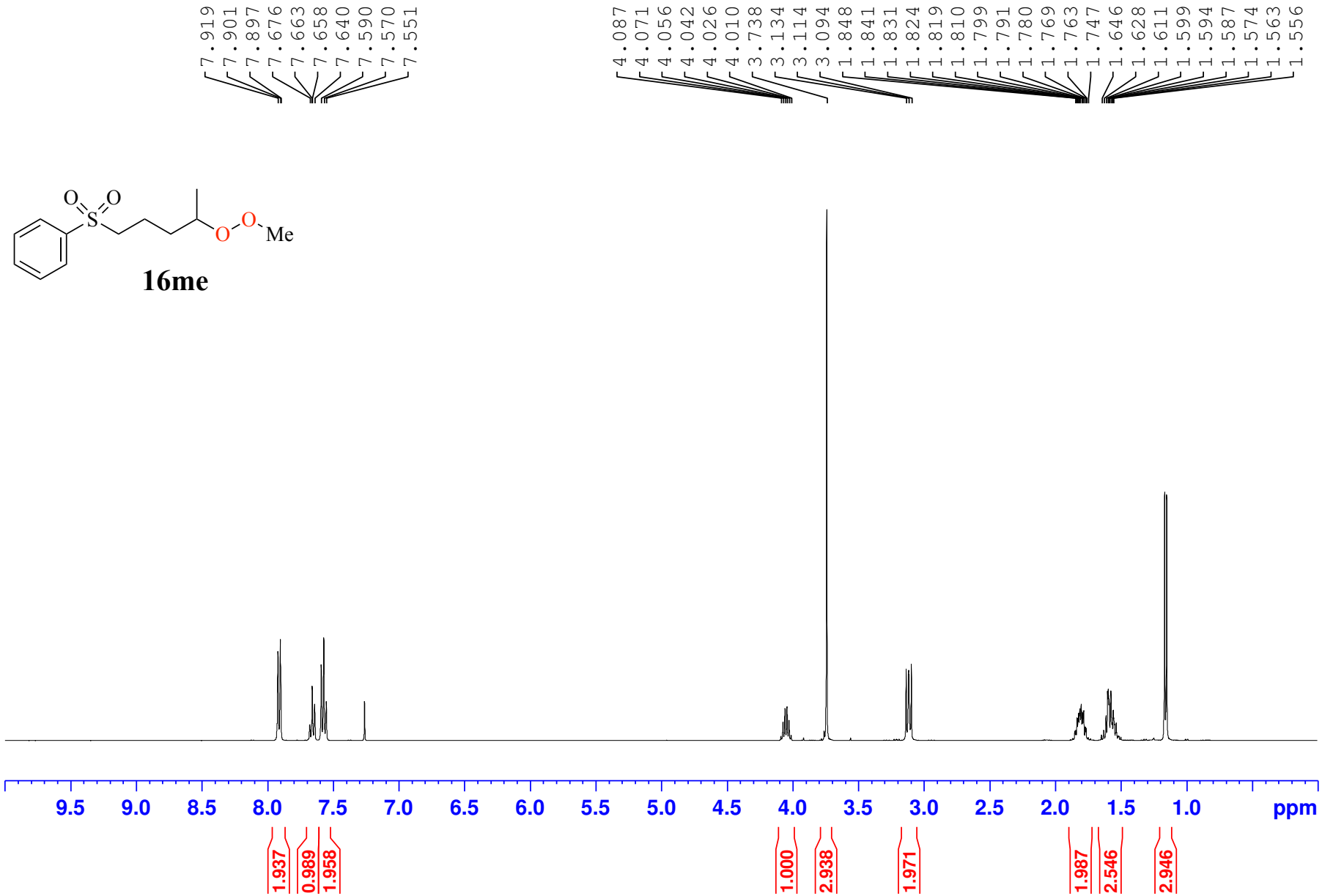
50
40
30
20
ppm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4.056
4.042
4.026
4.010
3.738
3.134
3.114
3.094
1.848
1.841
1.831
1.824
1.819
1.810
1.799
1.791
1.780
1.769
1.763
1.747
1.646
1.628
1.611
1.599
1.594
1.587
1.574
1.563
1.556
O
O
S
O
O
Me
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8.0
7.5
7.0
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5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
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1.000
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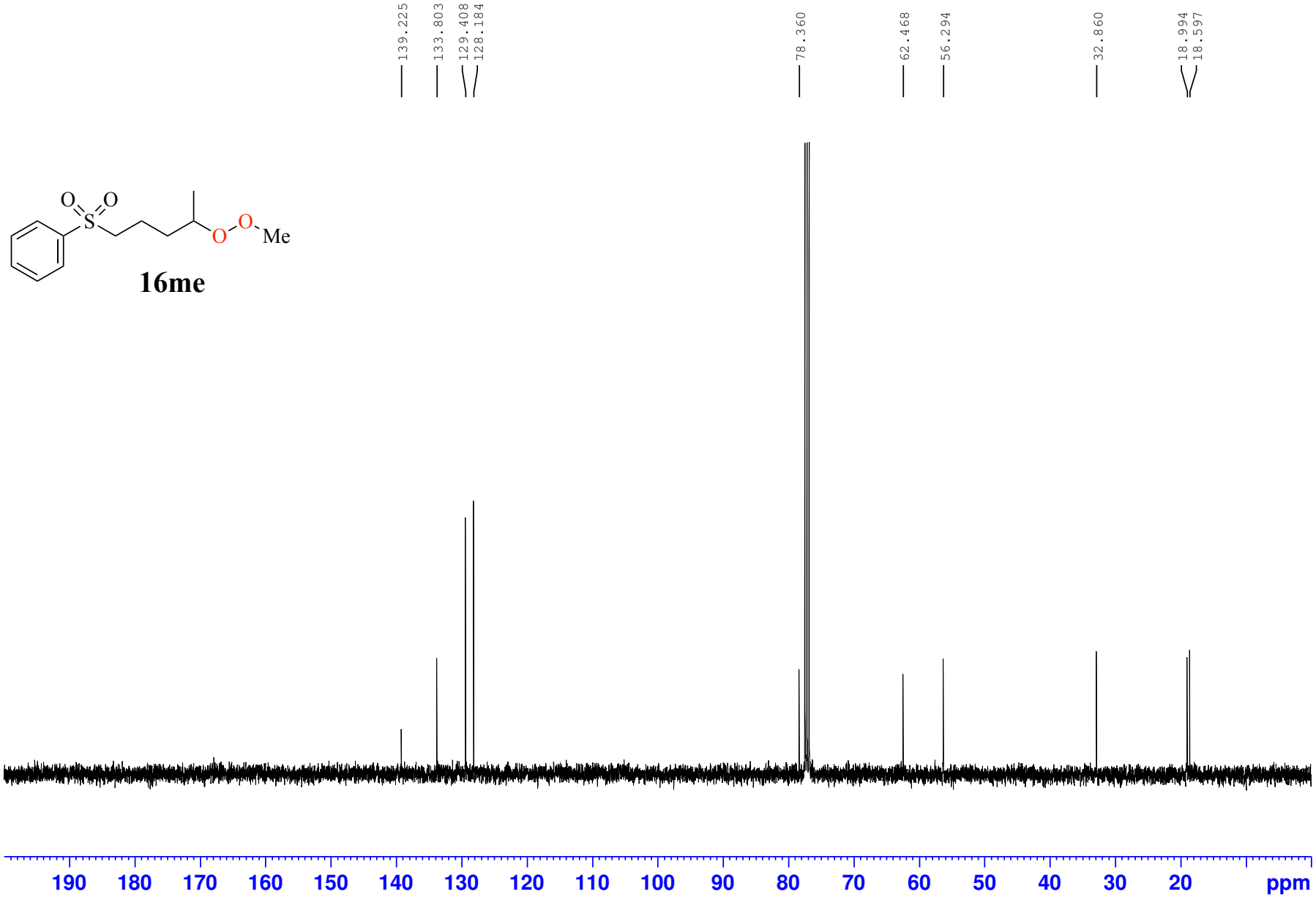
1.971
1.987
2.546
2.946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32.860
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18.597
O
O
S
O
O
Me
16me
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160
150
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130
120
110
100
90
80
70
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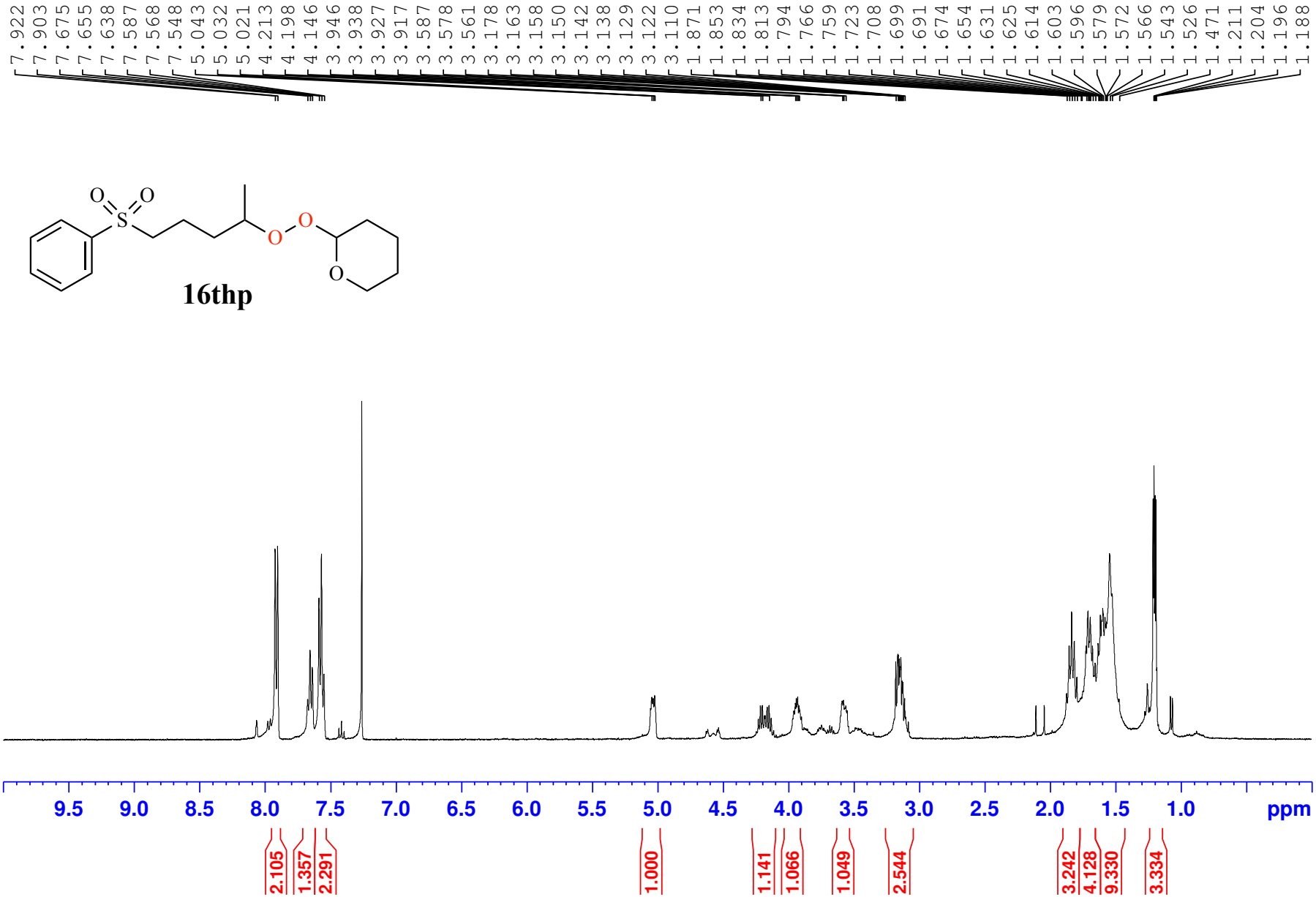
50
40
30
20
ppm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5.032
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4.198
4.146
3.946
3.938
3.927
3.917
3.587
3.578
3.561
3.178
3.163
3.158
3.150
3.142
3.138
3.129
3.122
3.110
1.871
1.853
1.834
1.813
1.794
1.766
1.759
1.723
1.708
1.699
1.691
1.674
1.654
1.631
1.625
1.614
1.603
1.596
1.579
1.572
1.566
1.543
1.526
1.471
1.211
1.204
1.196
1.188
O
O
S
O
O
O
16thp
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
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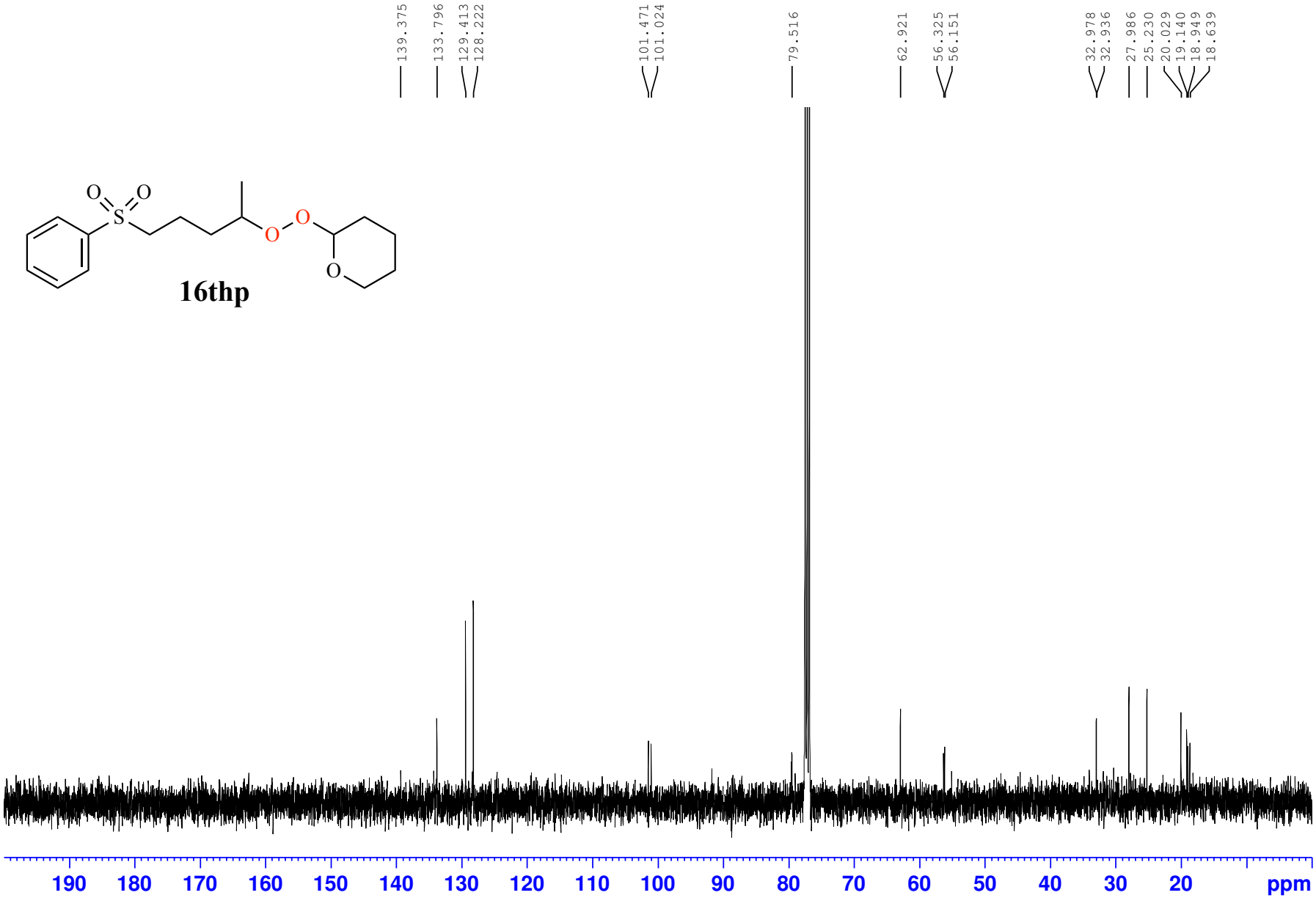
3.334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18.949
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O
S
O
O
O
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150
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120
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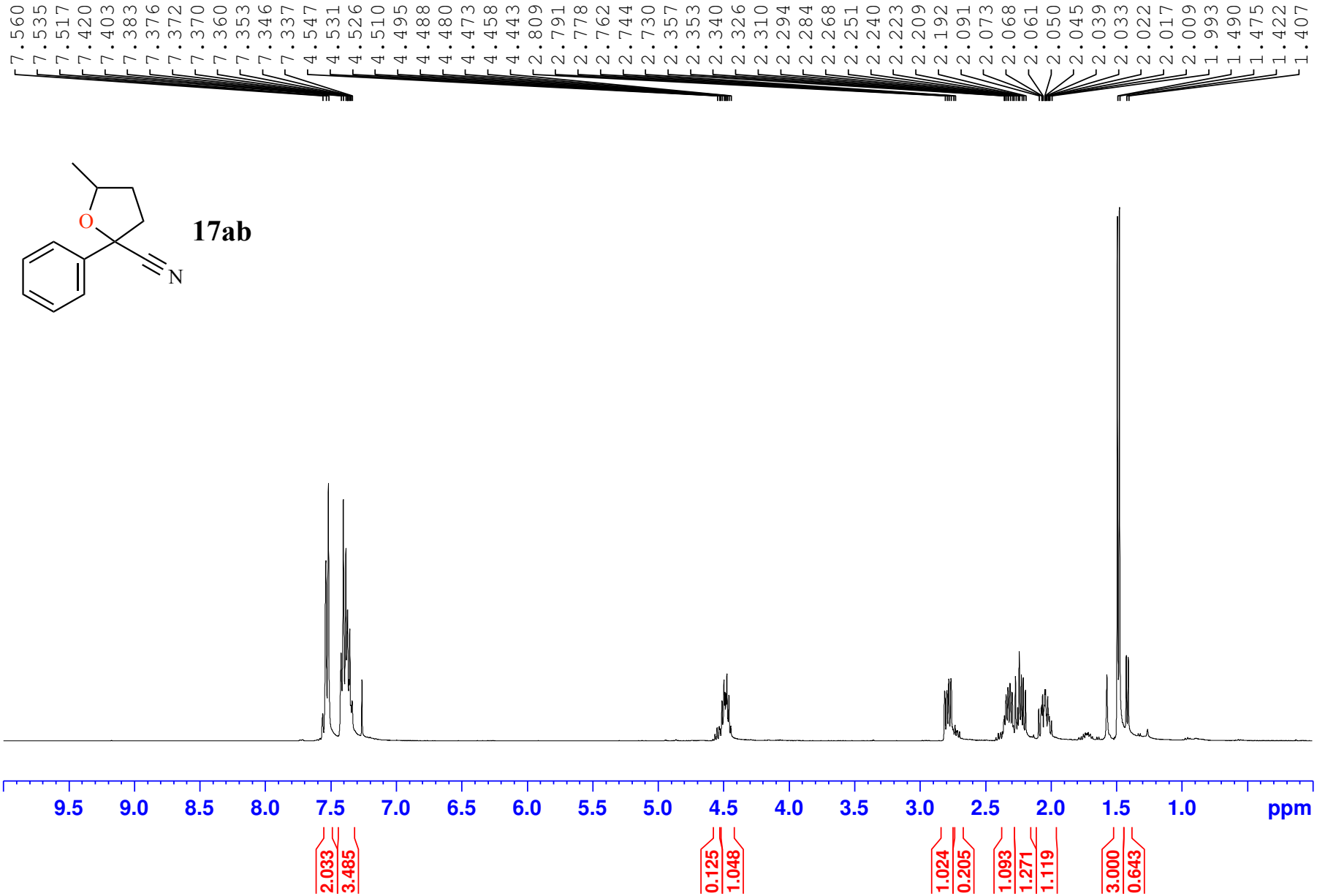
ppm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4.526
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4.495
4.488
4.480
4.473
4.458
4.443
2.809
2.791
2.778
2.762
2.744
2.730
2.357
2.353
2.340
2.326
2.310
2.294
2.284
2.268
2.251
2.240
2.223
2.209
2.192
2.091
2.073
2.068
2.061
2.050
2.045
2.039
2.033
2.022
2.017
2.009
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1.490
1.475
1.422
1.407
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N
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6.0
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5.0
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4.0
3.5
3.0
2.5
2.0
1.5
1.0
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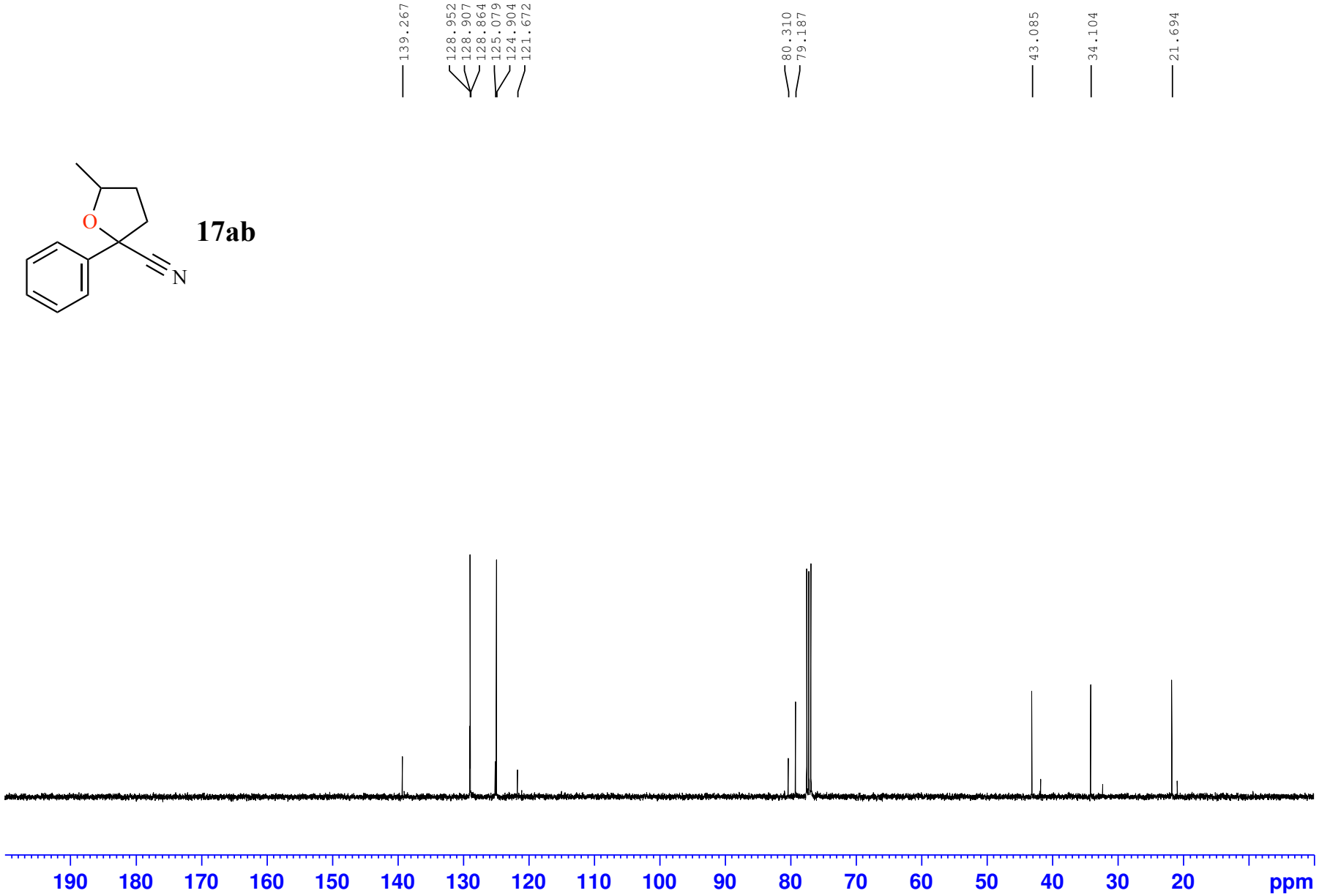
O
N

17ab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150
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80
70
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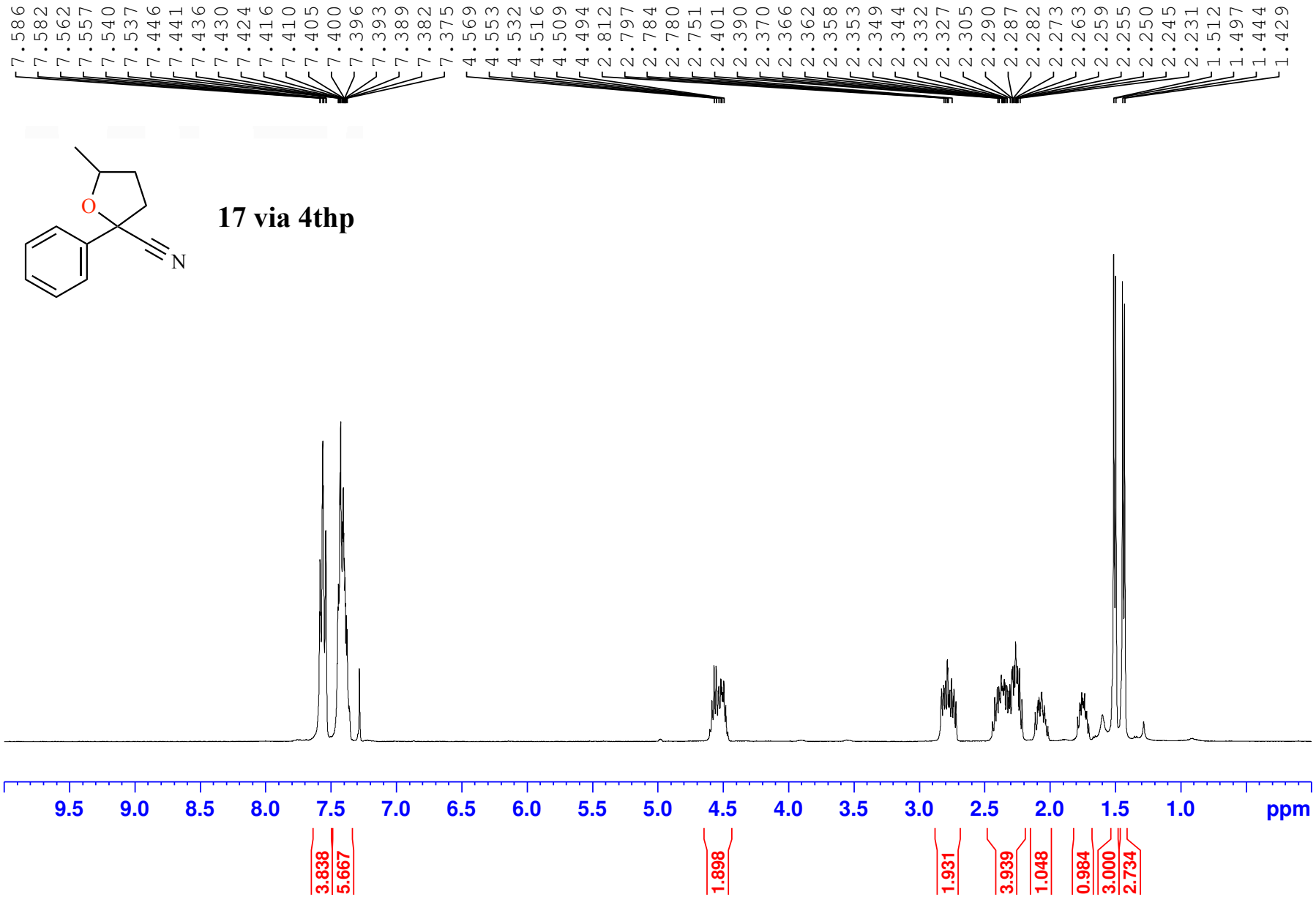
20
ppm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7.441
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7.410
7.405
7.400
7.396
7.393
7.389
7.382
7.375
4.569
4.553
4.532
4.516
4.509
4.494
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2.797
2.784
2.780
2.751
2.401
2.390
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2.366
2.362
2.358
2.353
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2.344
2.332
2.327
2.305
2.290
2.287
2.282
2.273
2.263
2.259
2.255
2.250
2.245
2.231
1.512
1.497
1.444
1.429
O
N
17 via 4thp
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8.5
8.0
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6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
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3.000
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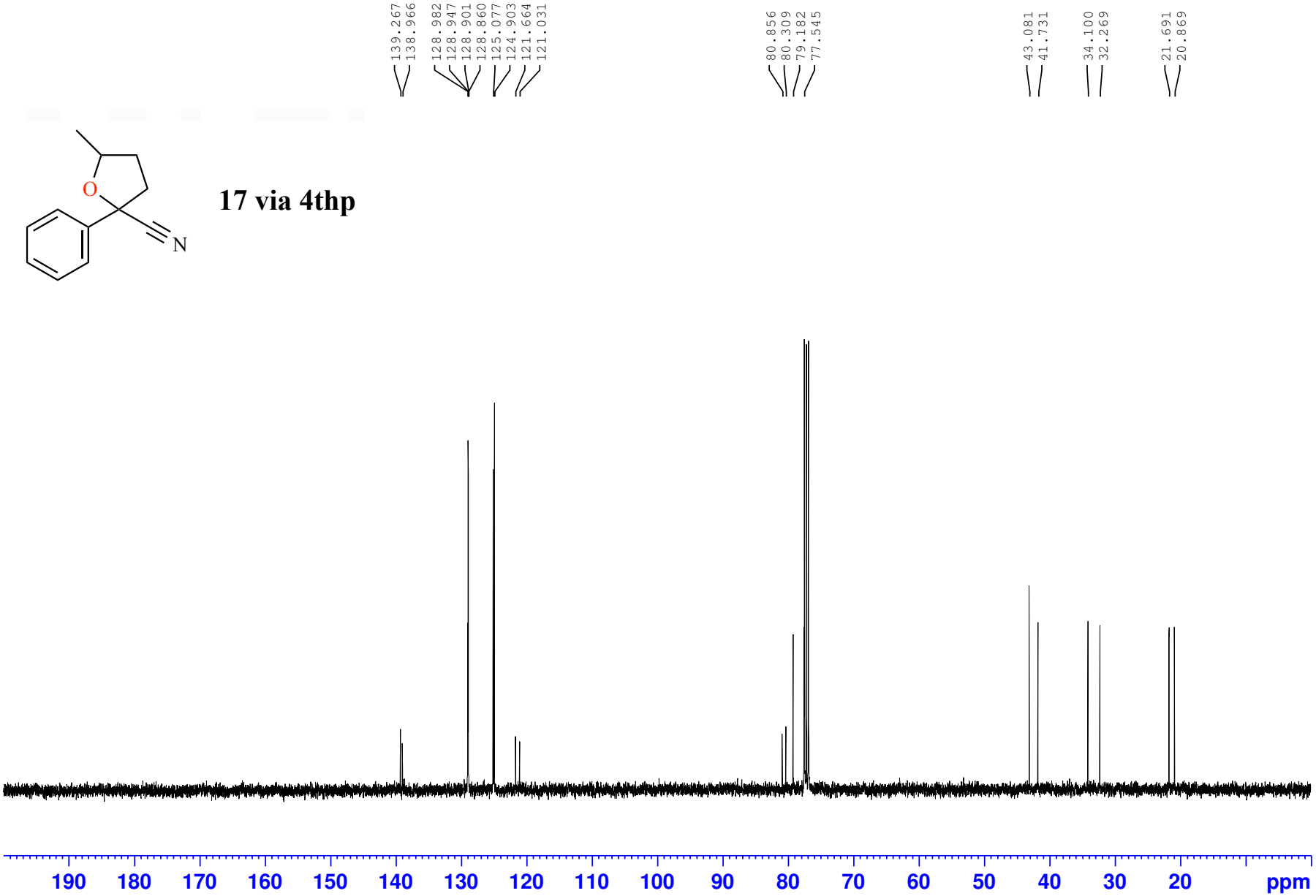
O
N

17 via 4thp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77.545
43.081
41.731
34.100
32.269
21.691
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20
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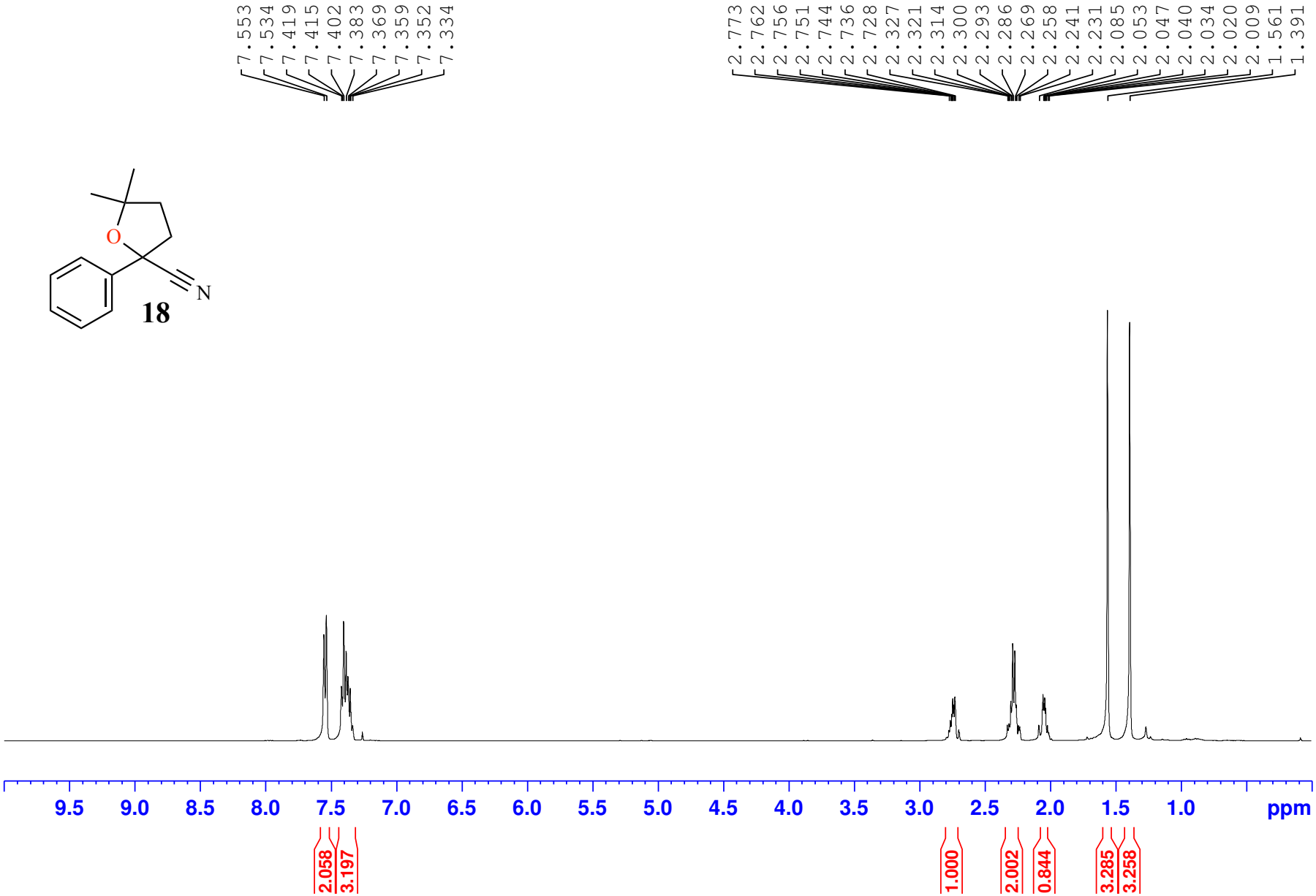
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2.751
2.744
2.736
2.728
2.327
2.321
2.314
2.300
2.293
2.286
2.269
2.258
2.241
2.231
2.085
2.053
2.047
2.040
2.034
2.020
2.009
1.561
1.391
O
N
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7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
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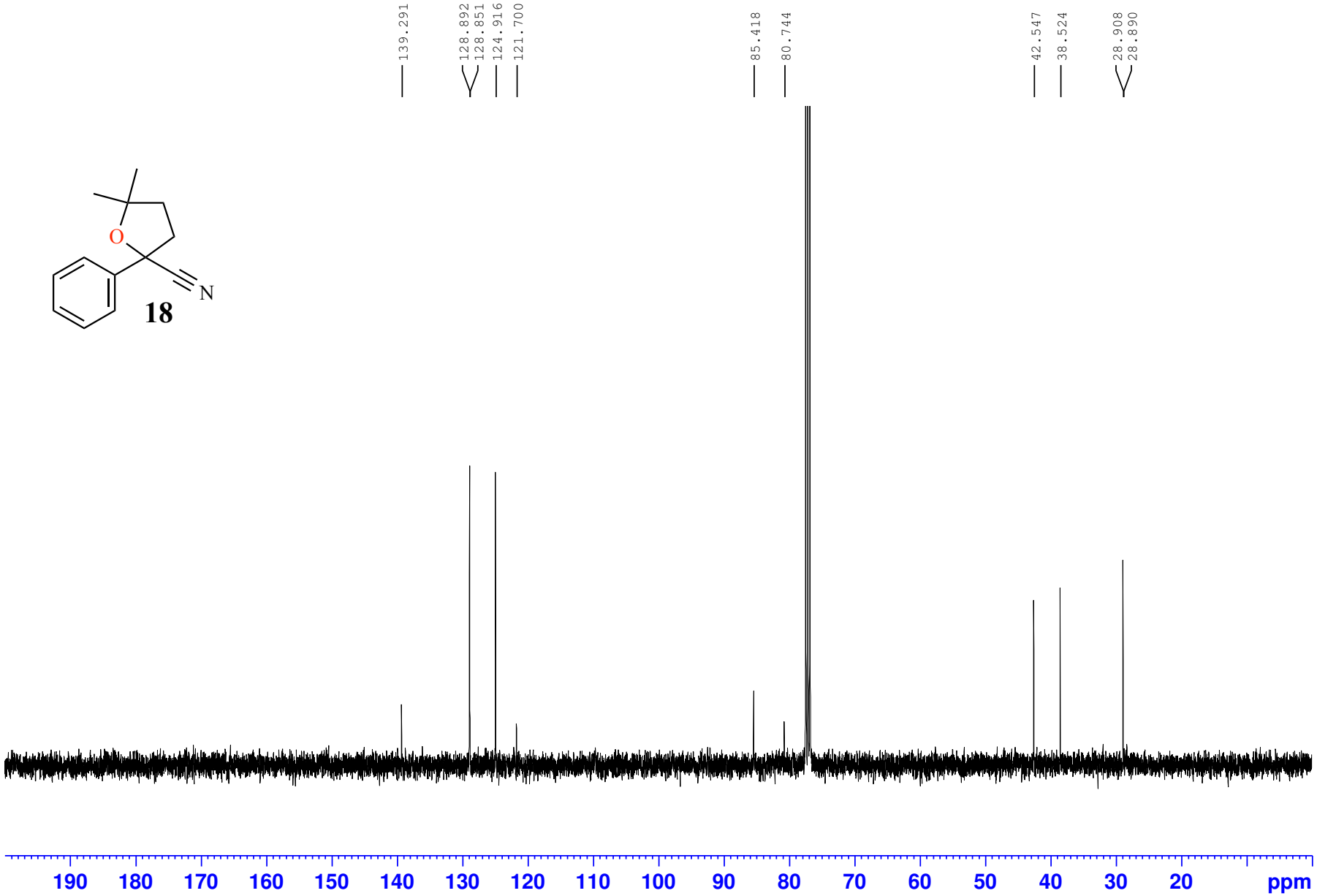
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28.890
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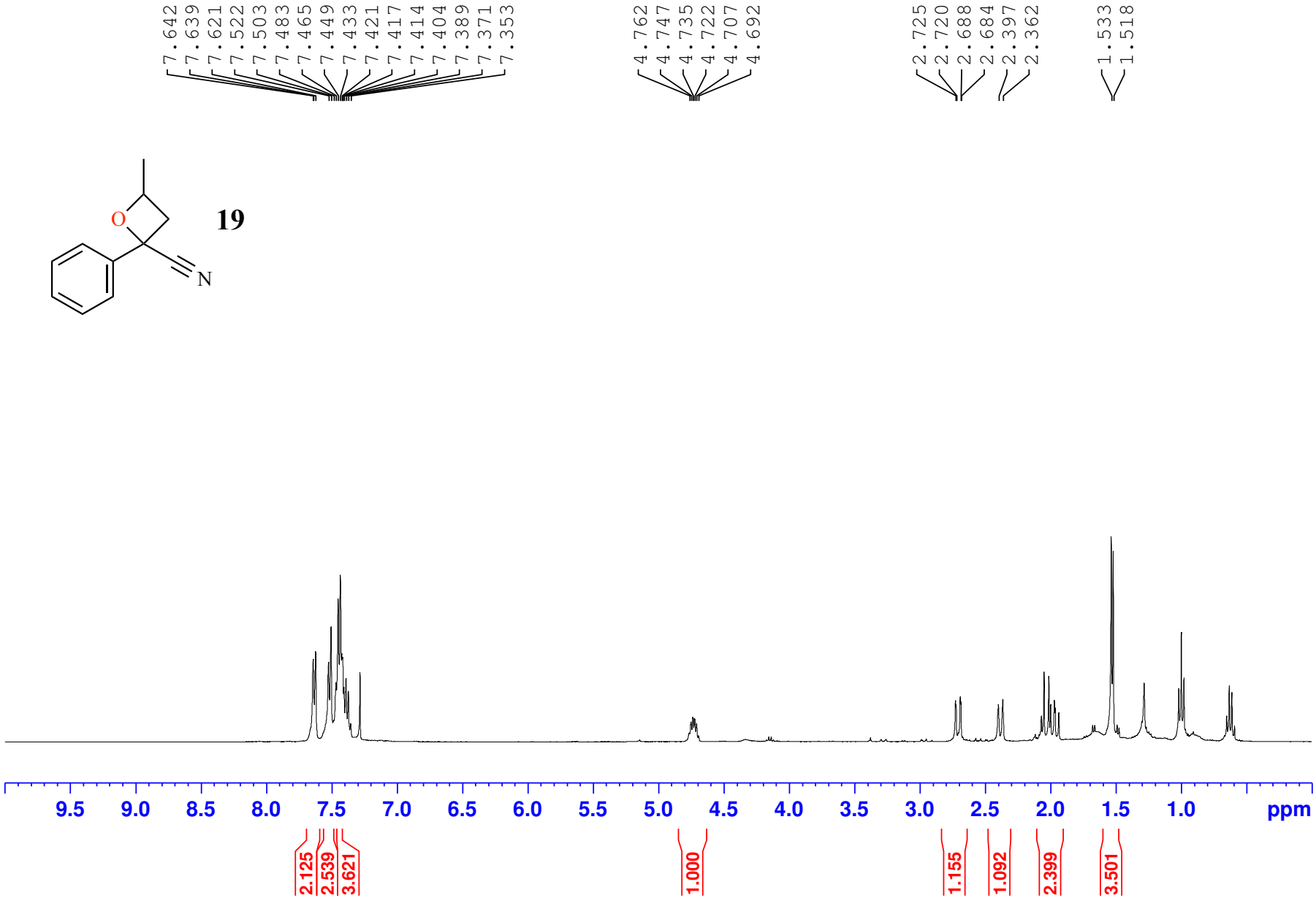
20
ppm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7.421
7.417
7.414
7.404
7.389
7.371
7.353
4.762
4.747
4.735
4.722
4.707
4.692
2.725
2.720
2.688
2.684
2.397
2.362
1.533
1.518
O
N
19
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8.0
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7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
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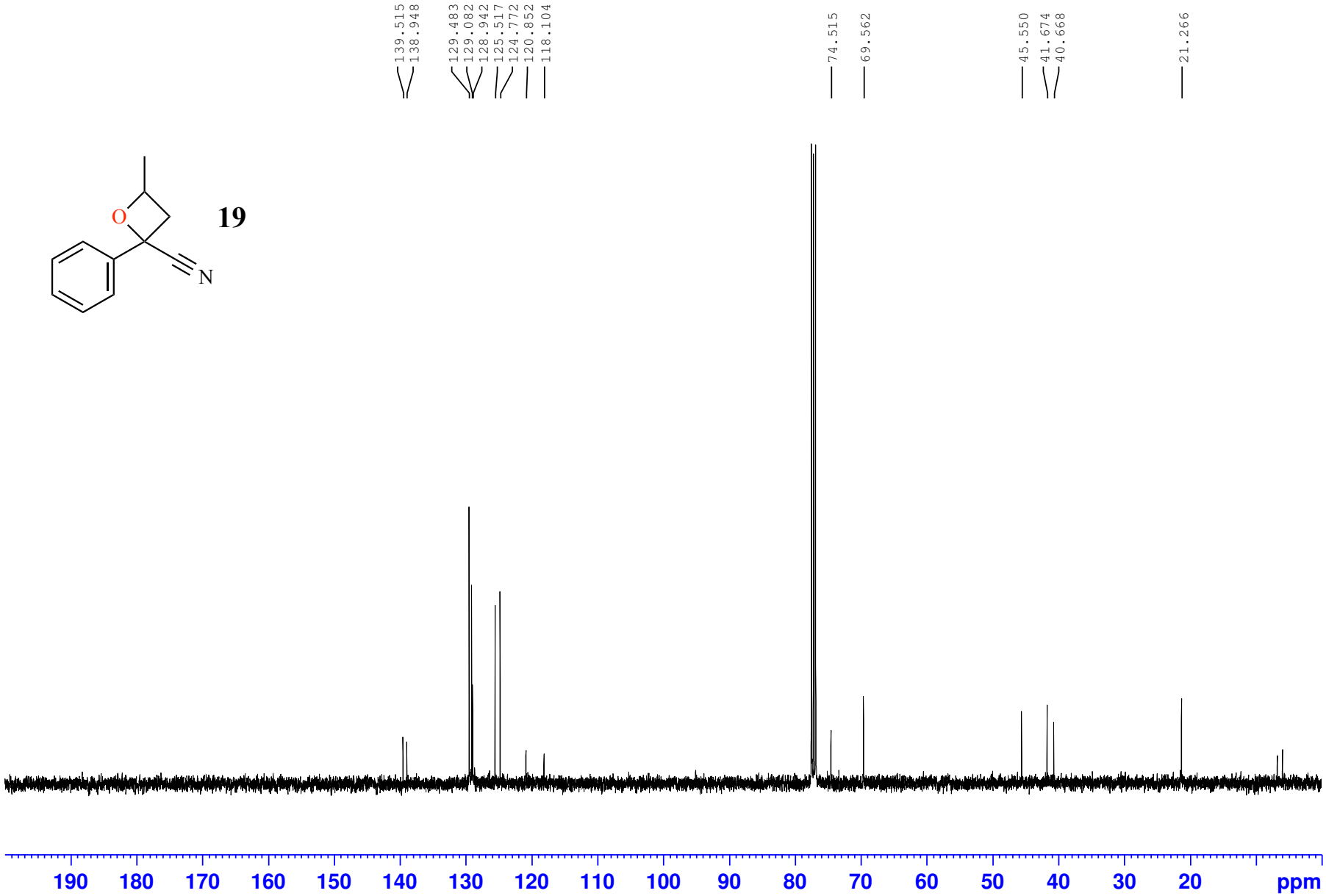
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O
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40
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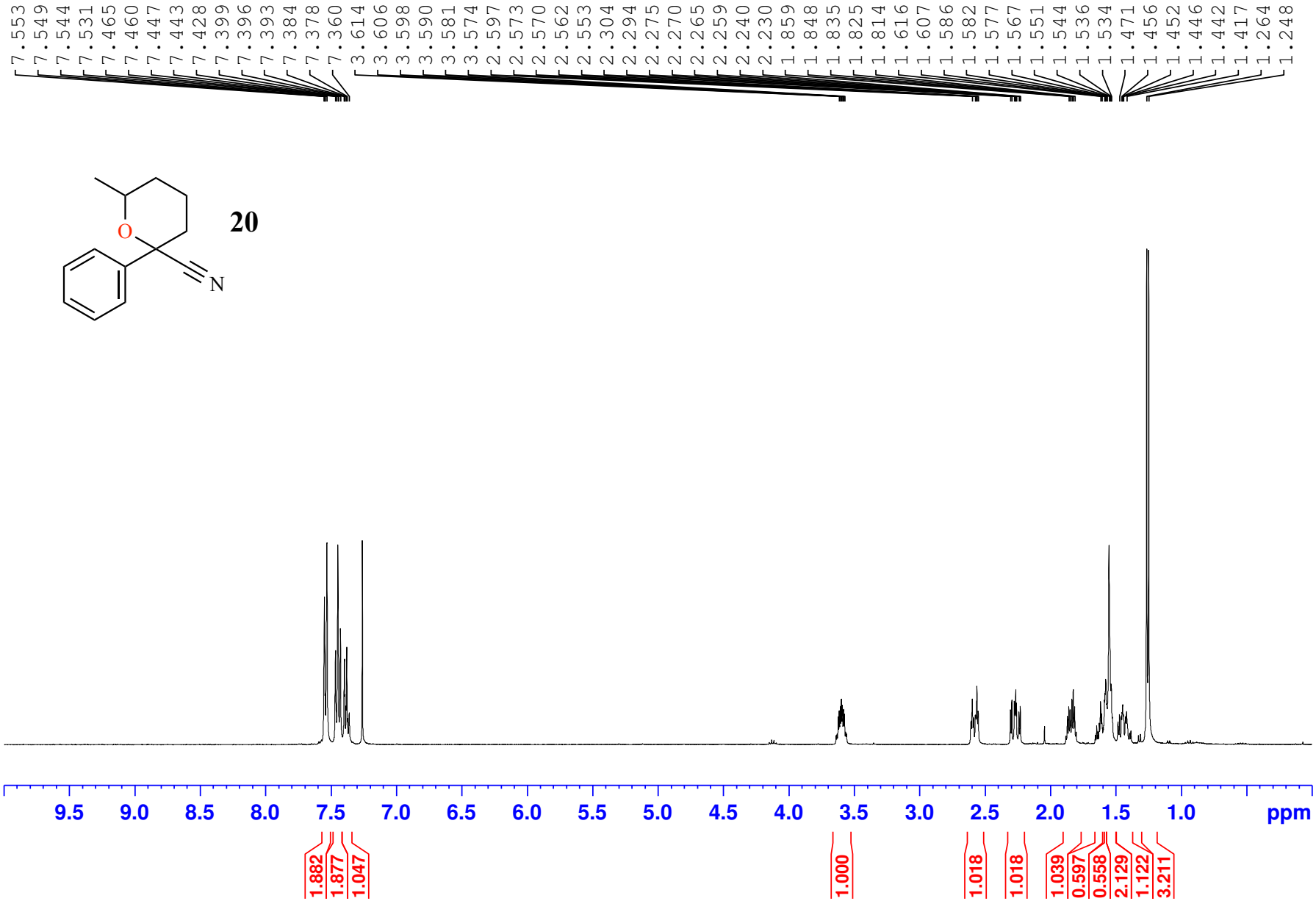
20
ppm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7.384
7.378
7.360
3.614
3.606
3.598
3.590
3.581
3.574
2.597
2.573
2.570
2.562
2.553
2.304
2.294
2.275
2.270
2.265
2.259
2.240
2.230
1.859
1.848
1.835
1.825
1.814
1.616
1.607
1.586
1.582
1.577
1.567
1.551
1.544
1.536
1.534
1.471
1.456
1.452
1.446
1.442
1.417
1.264
1.248
O
N
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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
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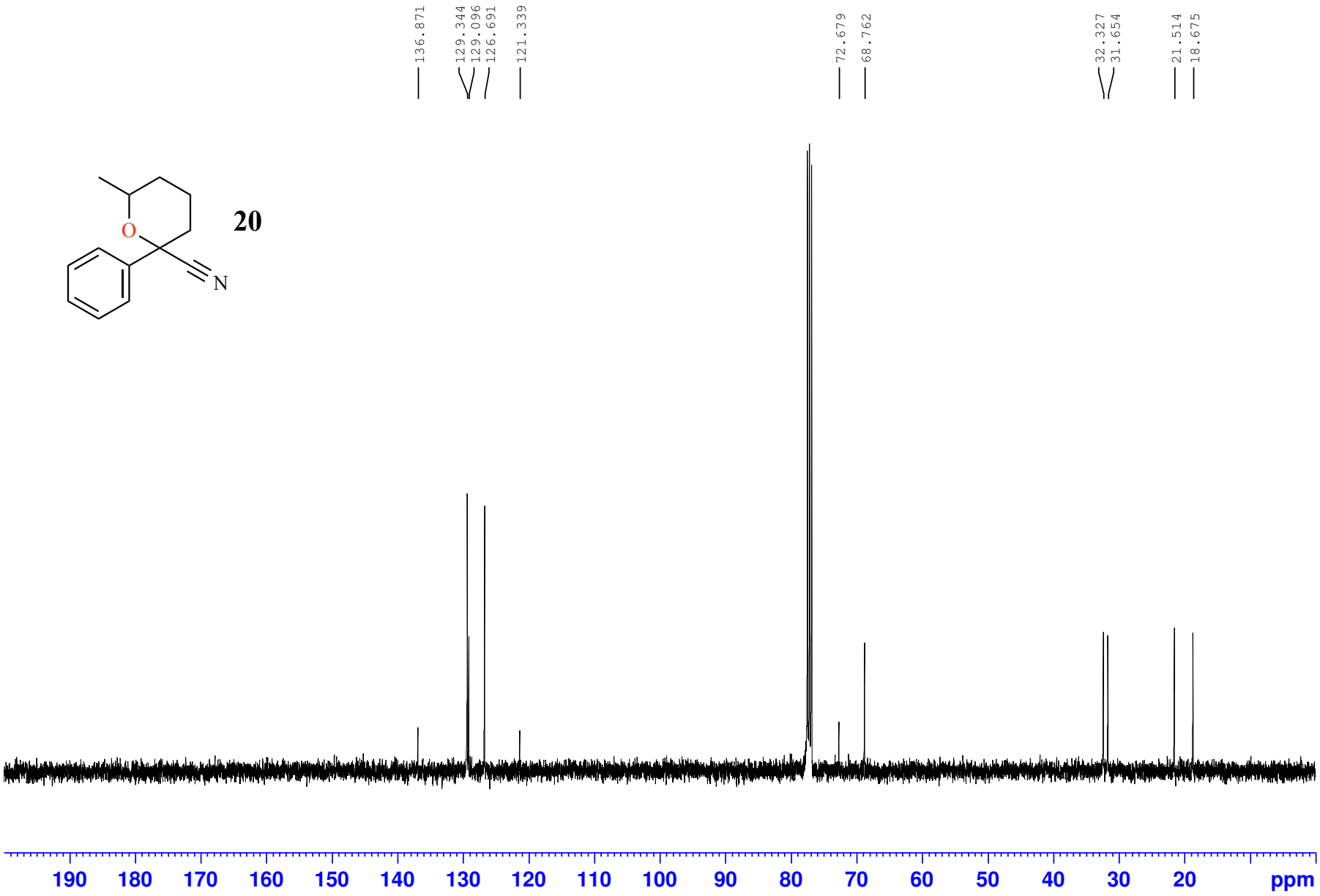
3.211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150
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120
110
100
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80
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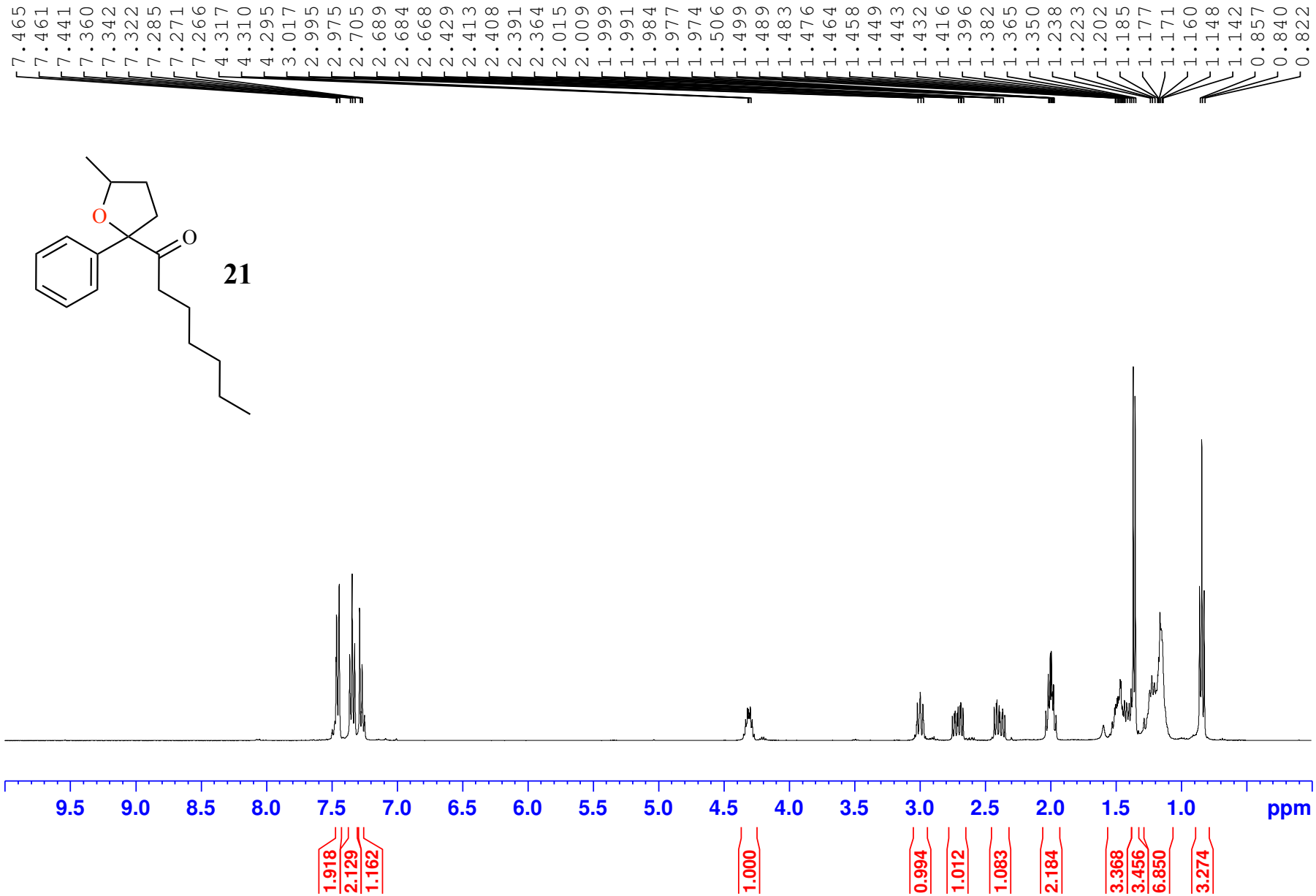
50
40
30
20
ppm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7.266
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4.310
4.295
3.017
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2.975
2.705
2.689
2.684
2.668
2.429
2.413
2.408
2.391
2.364
2.015
2.009
1.999
1.991
1.984
1.977
1.974
1.506
1.499
1.489
1.483
1.476
1.464
1.458
1.449
1.443
1.432
1.416
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1.177
1.171
1.160
1.148
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0.840
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O
O
21
9.5
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7.0
6.5
6.0
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5.0
4.5
4.0
3.5
3.0
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2.0
1.5
1.0
ppm
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0.994
1.012
1.083
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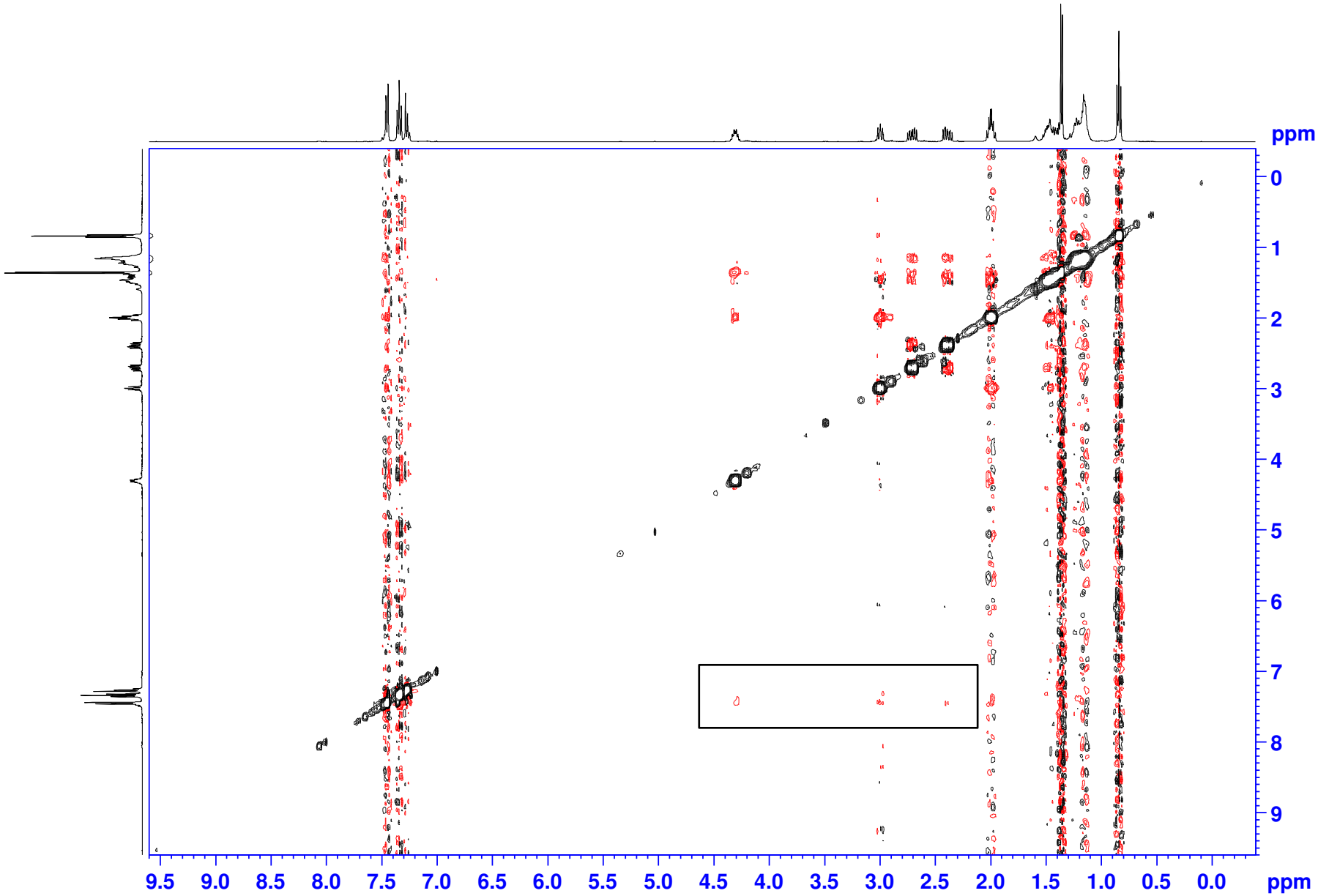
3.368
3.456
6.850
3.274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0
1
2
3
4
5
6
7
8
9
9.5
9.0
8.5
8.0
7.5
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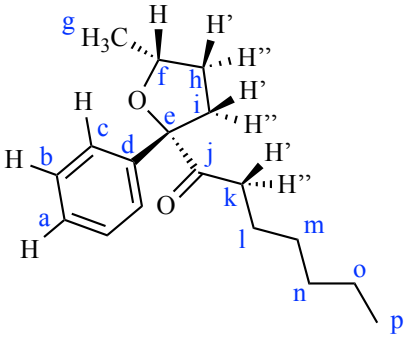
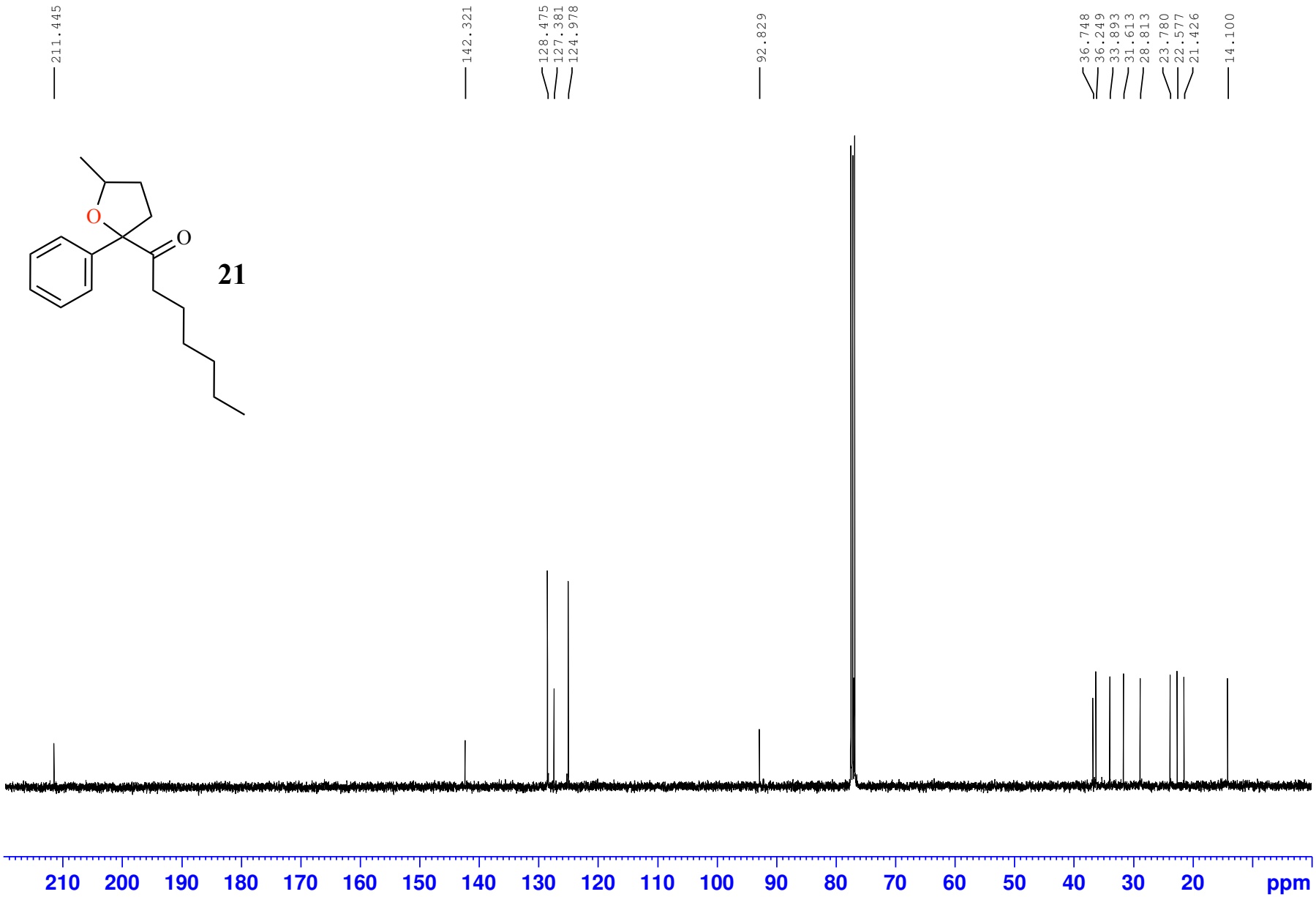
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
0.5
0.0
ppm

Table S1: Peak positions and spectral correlations for tetrahydrofuran **21**.

position	δ ^{13}C	^{13}C corr (HMBC)	δ^{1} H	COSY	NOESY
a	127.4	c	7.26*		
b	128.5	d	7.34*		
c	124.9	a,e	7.45*		f(w)
d	142.3	-	na		
e	92.8	-	na		
f	76.9	-	4.30*	g,h,h'(w)	c(w),g,h
g	21.4	f, h	1.35*	-	
h	33.9	e,f	h: 1.99*	h',f	f(w),h',i
			h': 1.45*	i'	i',k,i(w)
i	36.2	e,f,	i: 2.99*	h,h'(w)	h,h'
		h, j	i': 1.99*	h'	h'
j	211.4	-	na		
k	36.7	j, l, m	k: 2.71*	k',l	k'
			k': 2.38*	l	k
l	23.8	n	1.45*	i'	
m	28.8	o	1.12*	na	
n	31.6	l, o	1.16*	na	
o	22.6	m, p	1.23*	na	
p	14.1	n, o	0.84*	o	o



* HSQC correlation with associated ^{13}C
w: weak COSY or NOESY correlation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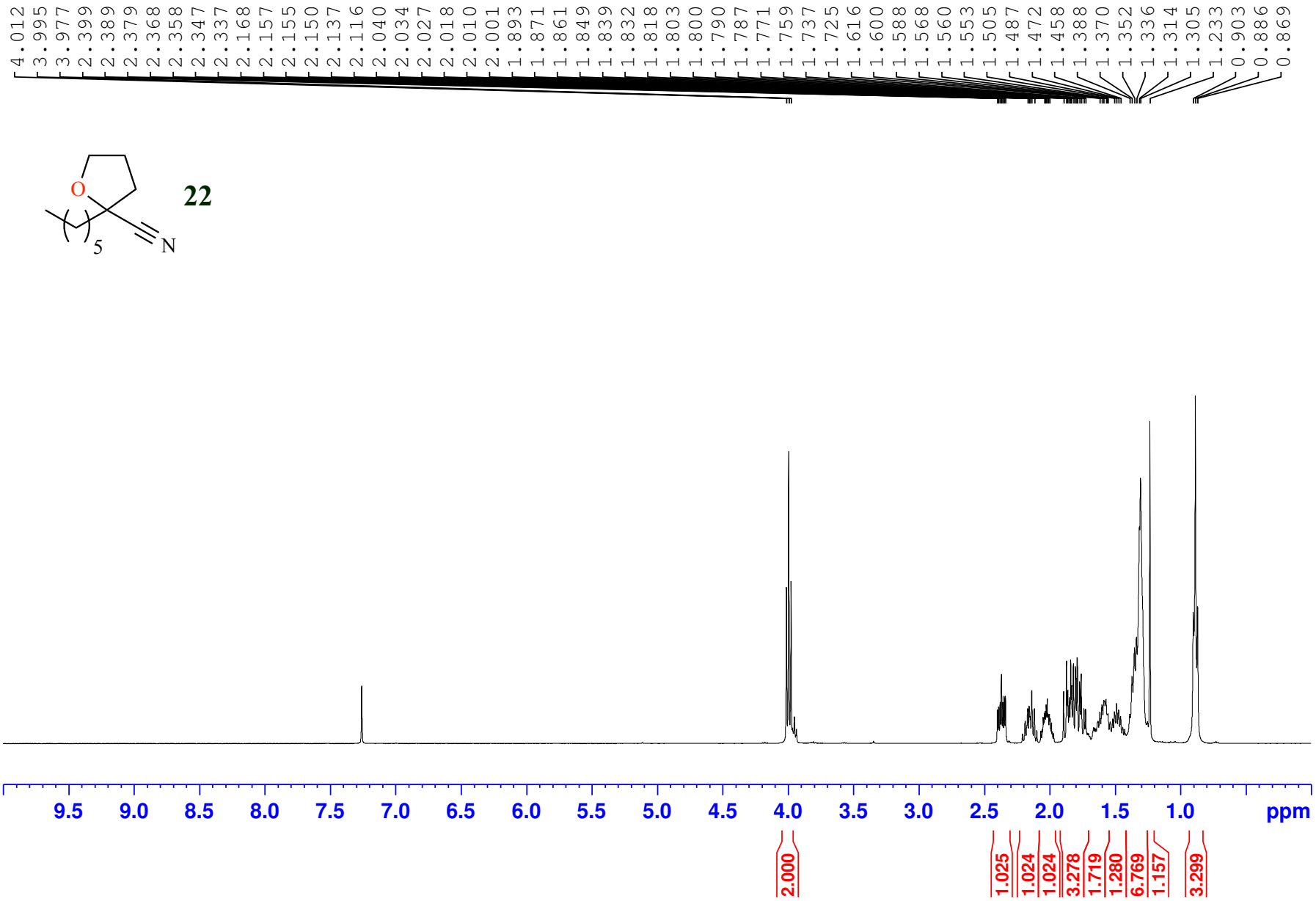
128.475
127.381
124.978
92.829
36.748
36.249
33.893
31.613
28.813
23.780
22.577
21.426
14.100
O
O
21
210 200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 ppm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3.977
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2.379
2.368
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2.168
2.157
2.155
2.150
2.137
2.116
2.040
2.034
2.027
2.018
2.010
2.001
1.893
1.871
1.861
1.849
1.839
1.832
1.818
1.803
1.800
1.790
1.787
1.771
1.759
1.737
1.725
1.616
1.600
1.588
1.568
1.560
1.553
1.505
1.487
1.472
1.458
1.388
1.370
1.352
1.336
1.314
1.305
1.233
0.903
0.886
0.869
O
5
N
22
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
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1.024
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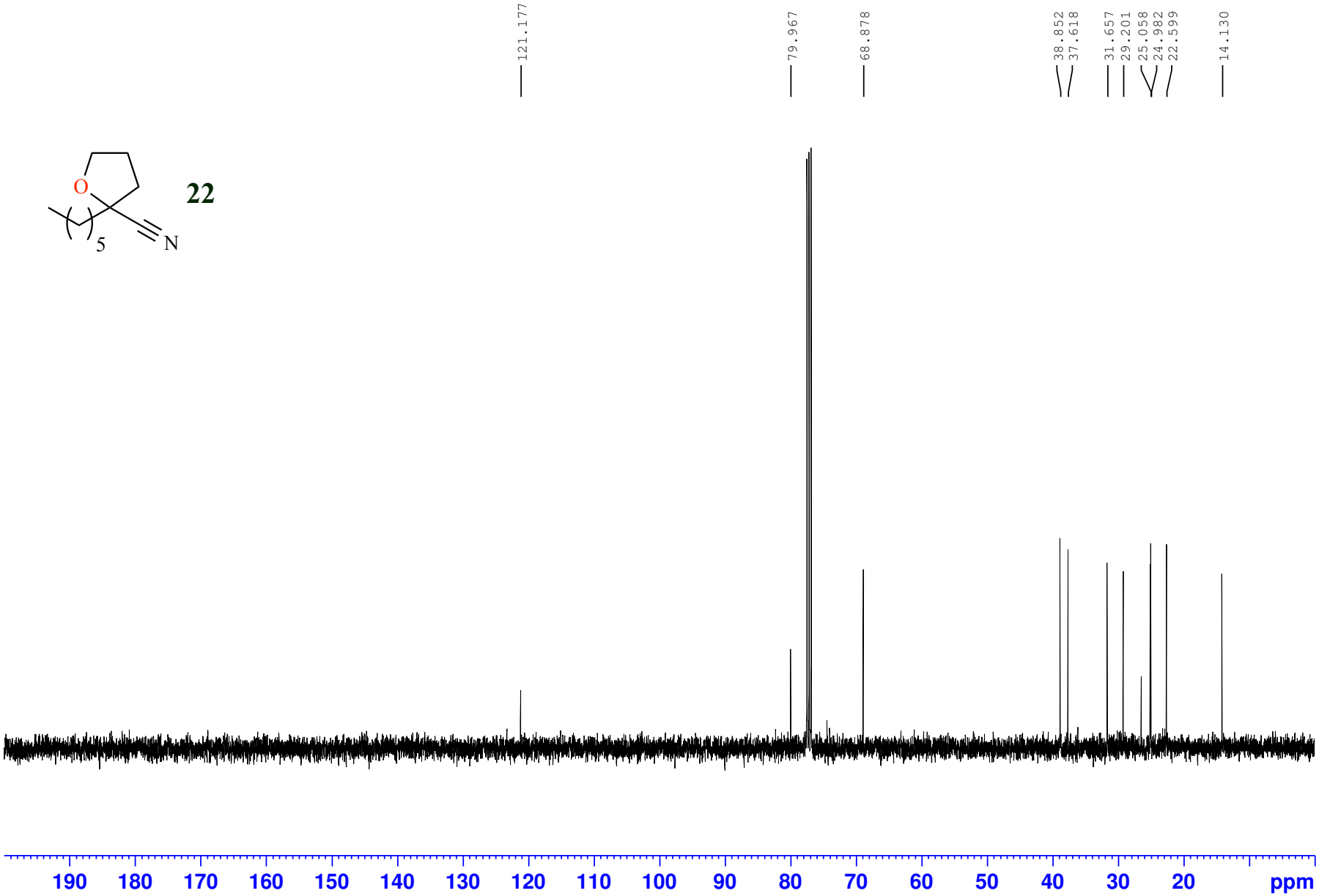
1.719
1.280
6.769
1.157
3.299


22
O
5
N
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79.967
68.878
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37.618
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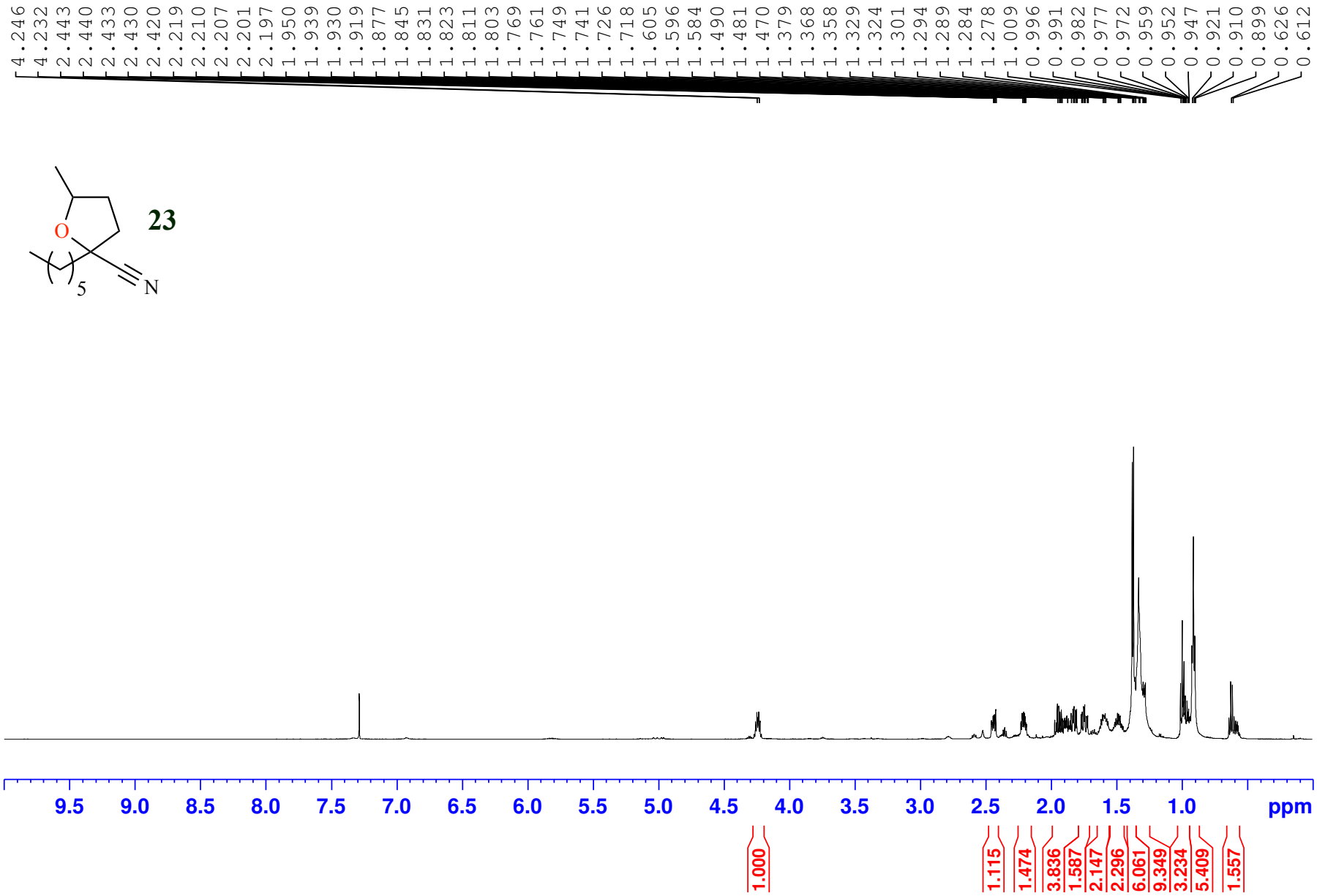
29.201
25.058
24.982
22.599
14.130
190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 ppm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4.232
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2.440
2.433
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2.219
2.210
2.207
2.201
2.197
1.950
1.939
1.930
1.919
1.877
1.845
1.831
1.823
1.811
1.803
1.769
1.761
1.749
1.741
1.726
1.718
1.605
1.596
1.584
1.490
1.481
1.470
1.379
1.368
1.358
1.329
1.324
1.301
1.294
1.289
1.284
1.278
1.009
0.996
0.991
0.982
0.977
0.972
0.959
0.952
0.947
0.921
0.910
0.899
0.626
0.612
O
5
N
23
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
ppm
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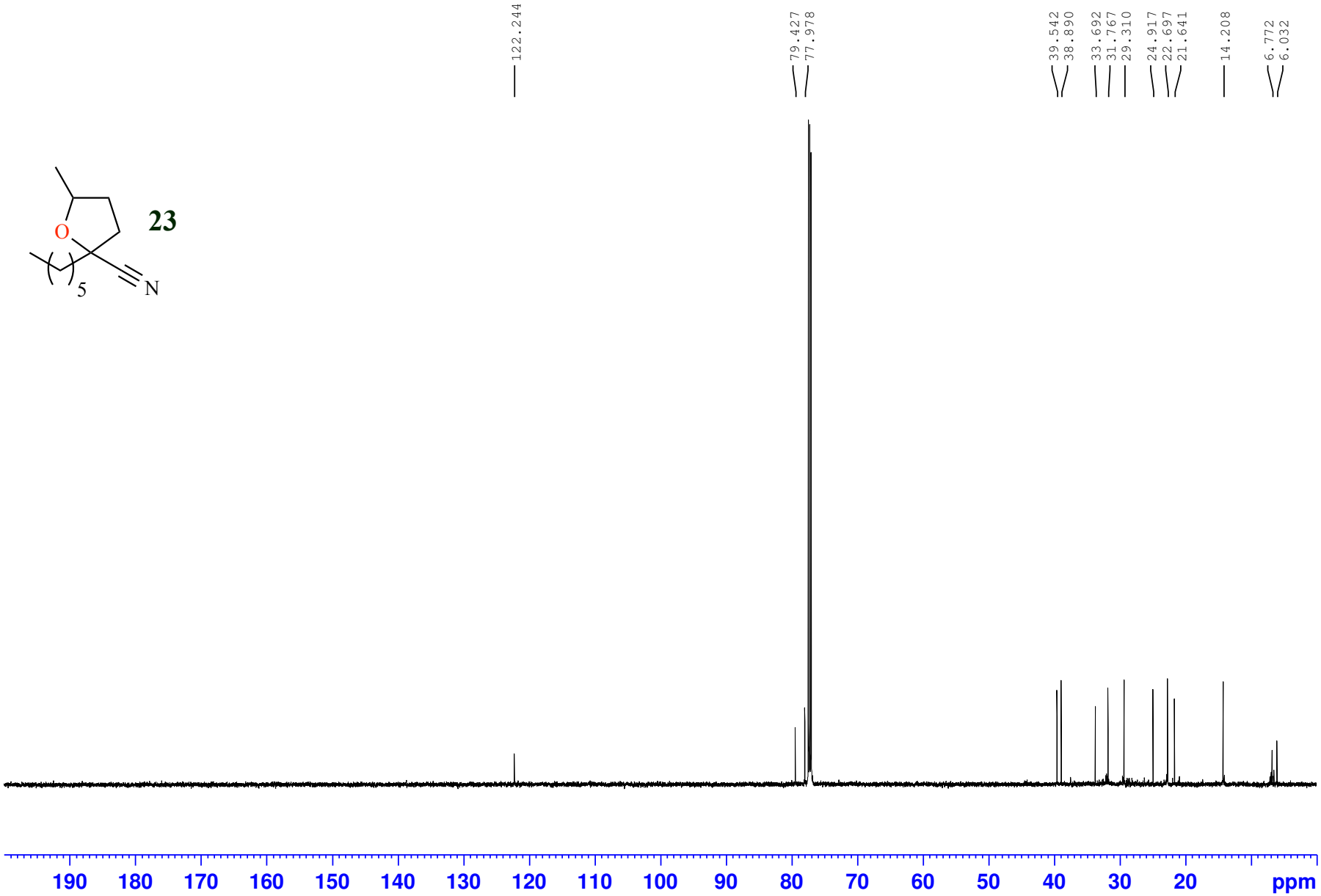
3.836
1.587
2.147
2.296
6.061
9.349
3.234
5.409
1.557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O
5
N
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77.978
39.542
38.890
33.692
31.767
29.310
24.917
22.697
21.641
14.208
6.772
6.032
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180
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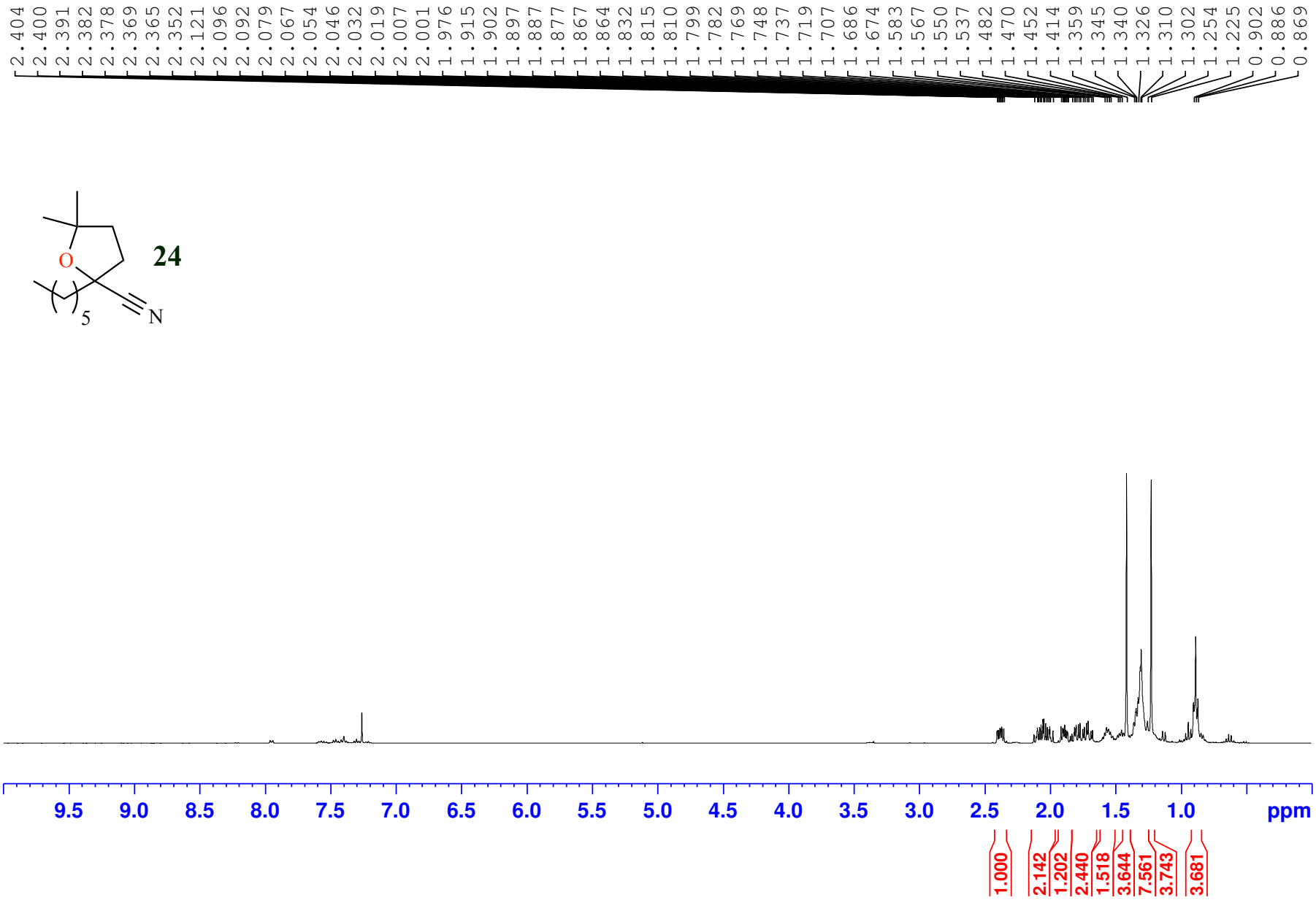
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
ppm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2.391
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2.365
2.352
2.121
2.096
2.092
2.079
2.067
2.054
2.046
2.032
2.019
2.007
2.001
1.976
1.915
1.902
1.897
1.887
1.877
1.867
1.864
1.832
1.815
1.810
1.799
1.782
1.769
1.748
1.737
1.719
1.707
1.686
1.674
1.583
1.567
1.550
1.537
1.482
1.470
1.452
1.414
1.359
1.345
1.340
1.326
1.310
1.302
1.254
1.225
0.902
0.886
0.869
O
5
N
24
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
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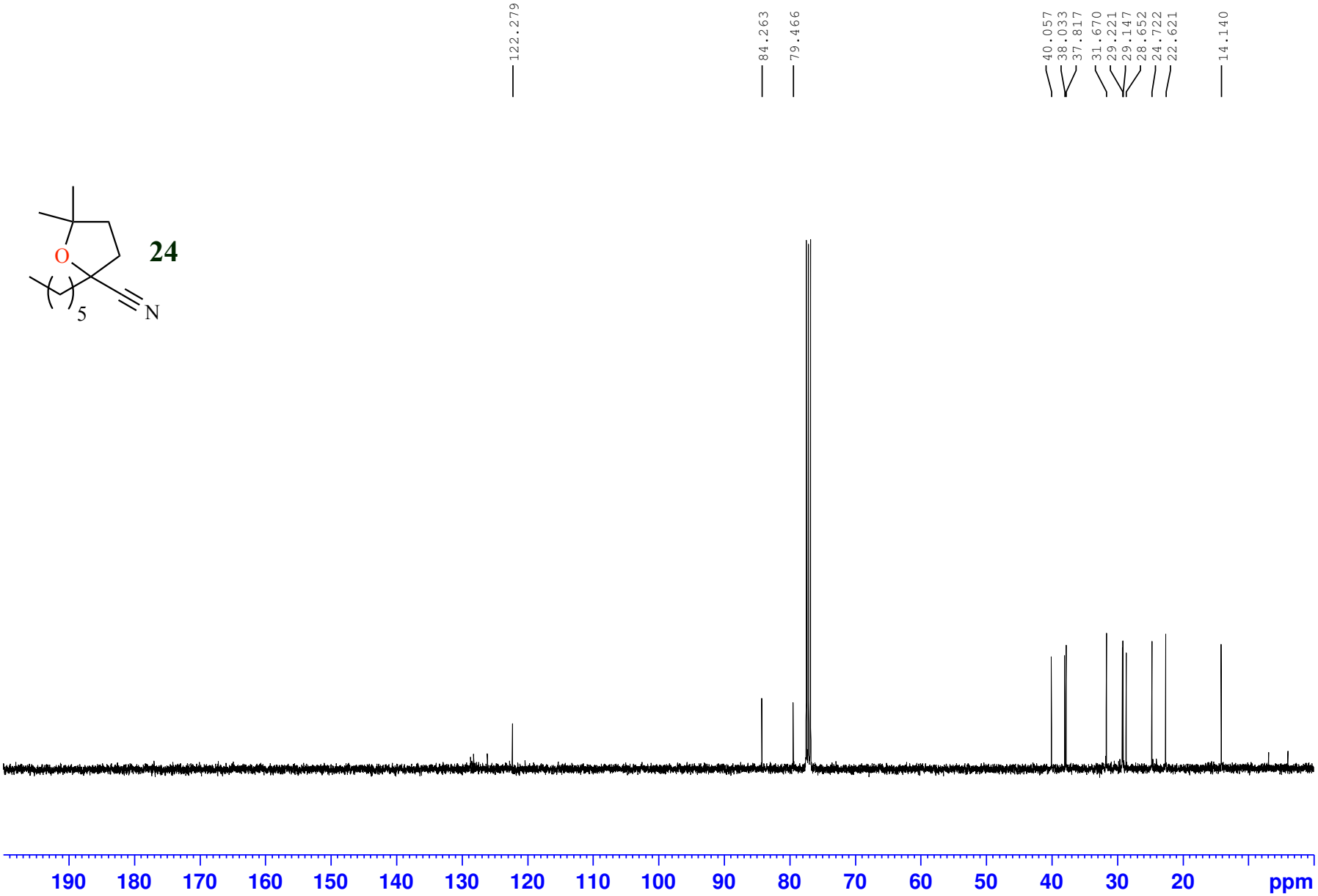
3.743
3.681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N
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37.817
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28.652
24.722
22.621
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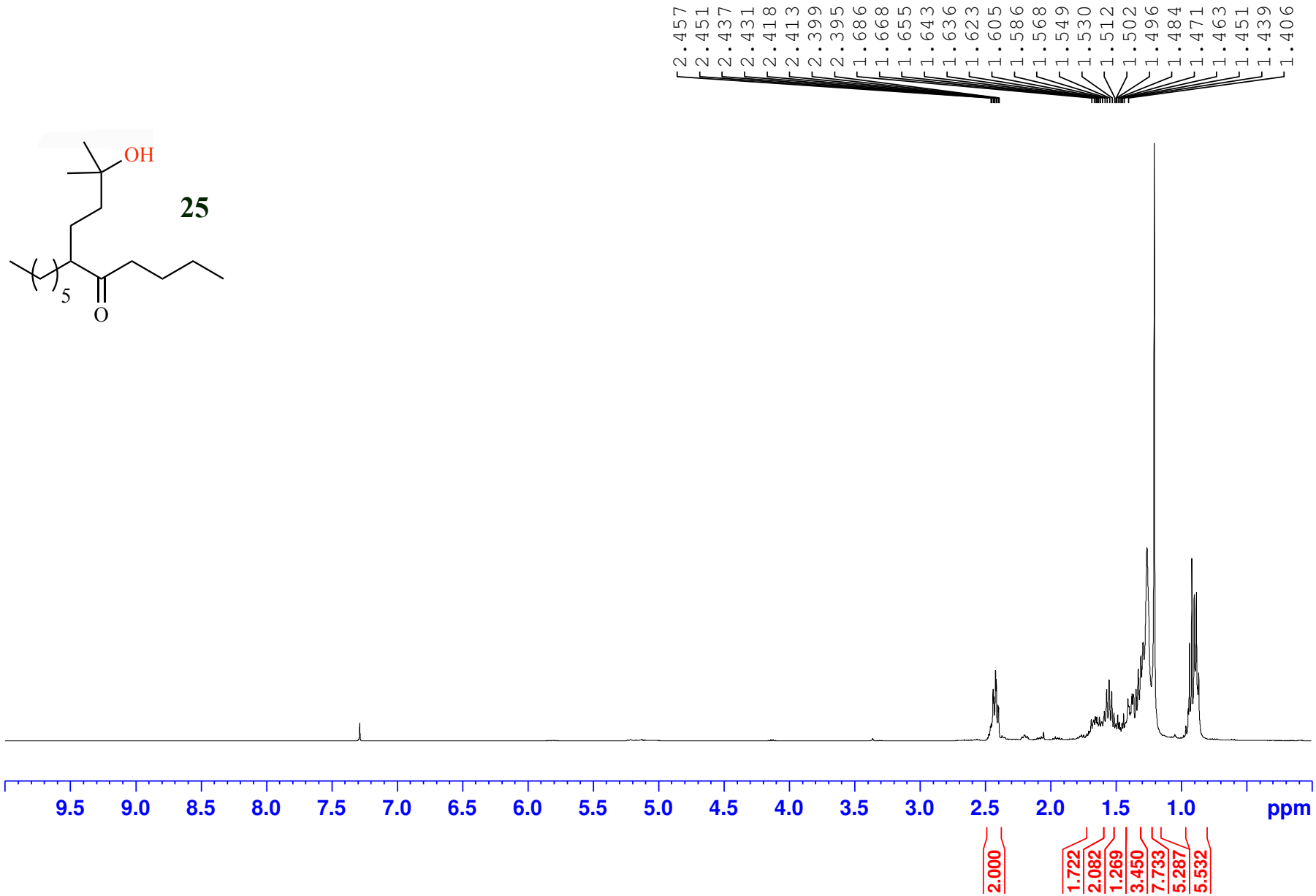
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
ppm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2.395
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1.643
1.636
1.623
1.605
1.586
1.568
1.549
1.530
1.512
1.502
1.496
1.484
1.471
1.463
1.451
1.439
1.406
OH
25
5
O
9.5
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8.5
8.0
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
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1.0
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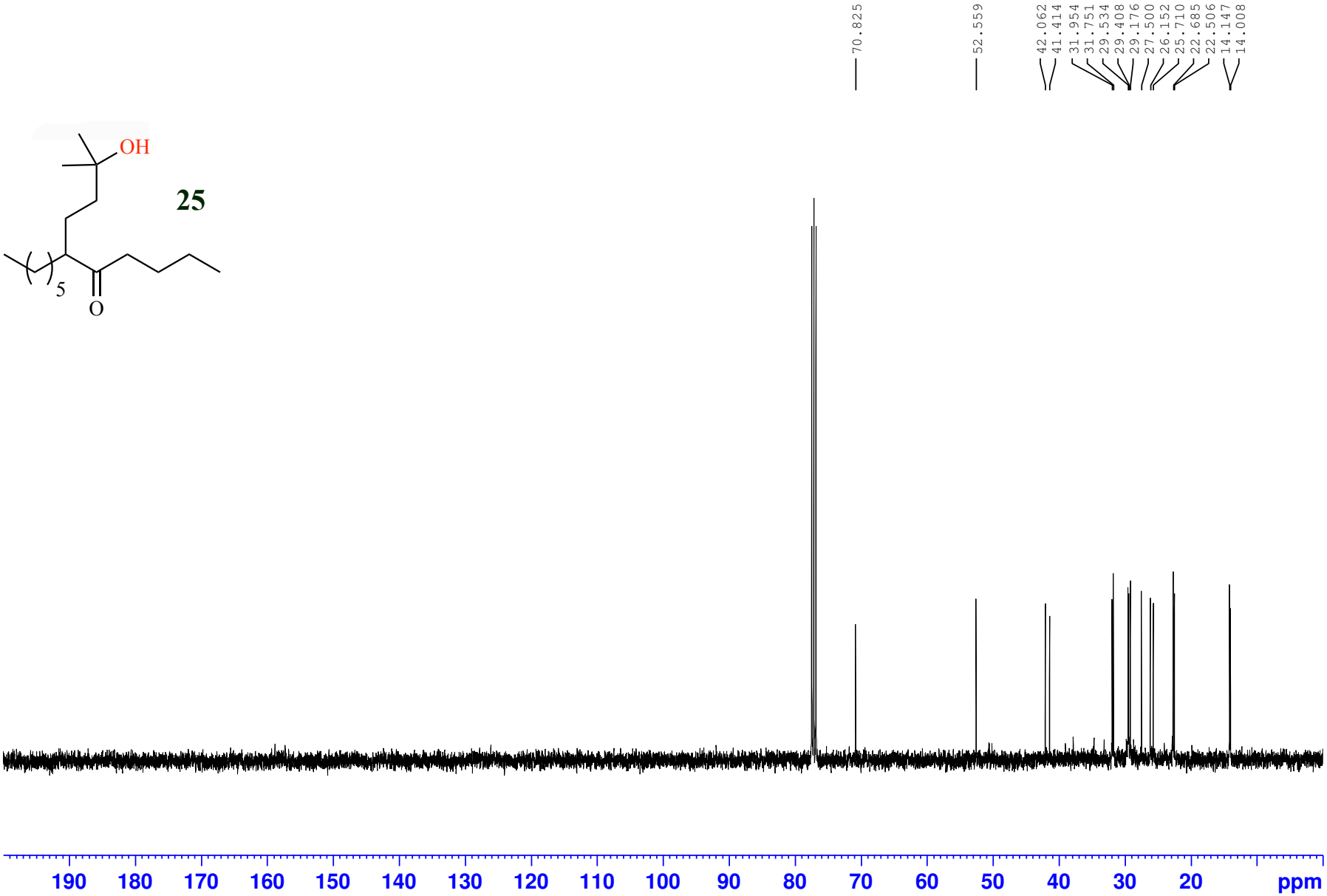
5.532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5
O
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31.954
31.751
29.534
29.408
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25.710
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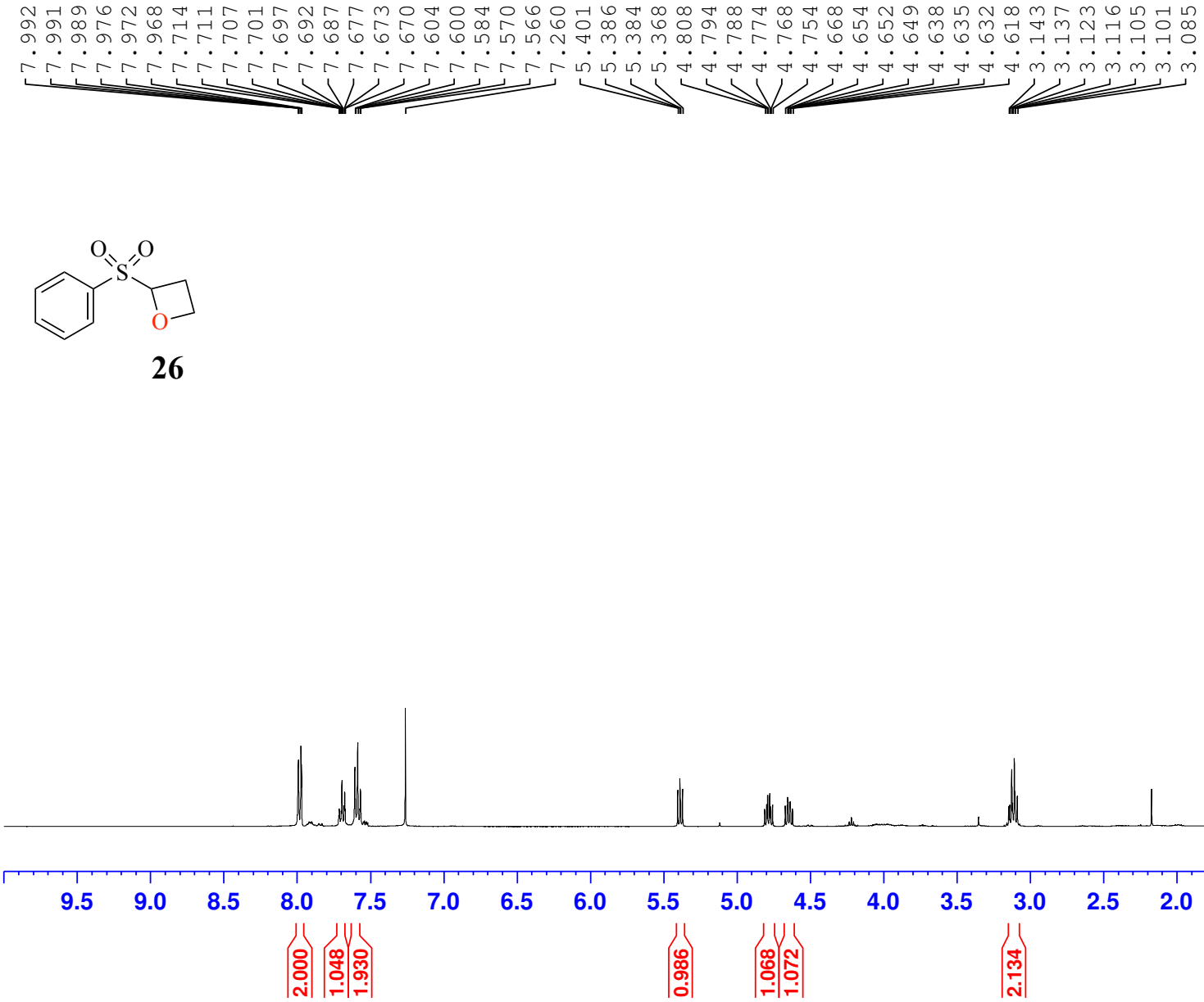
22.506
14.147
14.008
190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 ppm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7.711
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7.701
7.697
7.692
7.687
7.677
7.673
7.670
7.604
7.600
7.584
7.570
7.566
7.260
5.401
5.386
5.384
5.368
4.808
4.794
4.788
4.774
4.768
4.754
4.668
4.654
4.652
4.649
4.638
4.635
4.632
4.618
3.143
3.137
3.123
3.116
3.105
3.101
3.085
O
O
S
O
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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
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1.0
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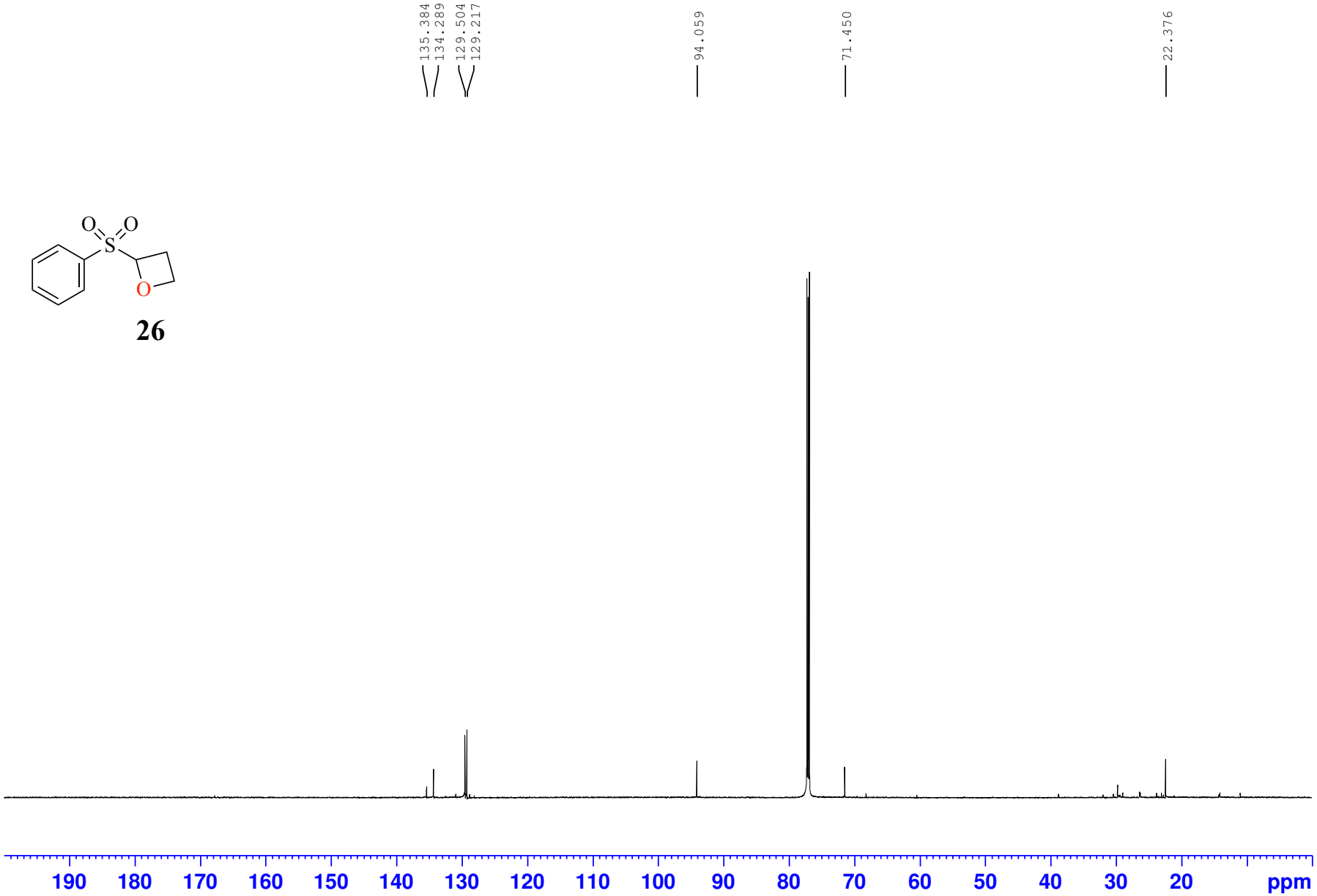
1.072
2.134

26

26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129.217
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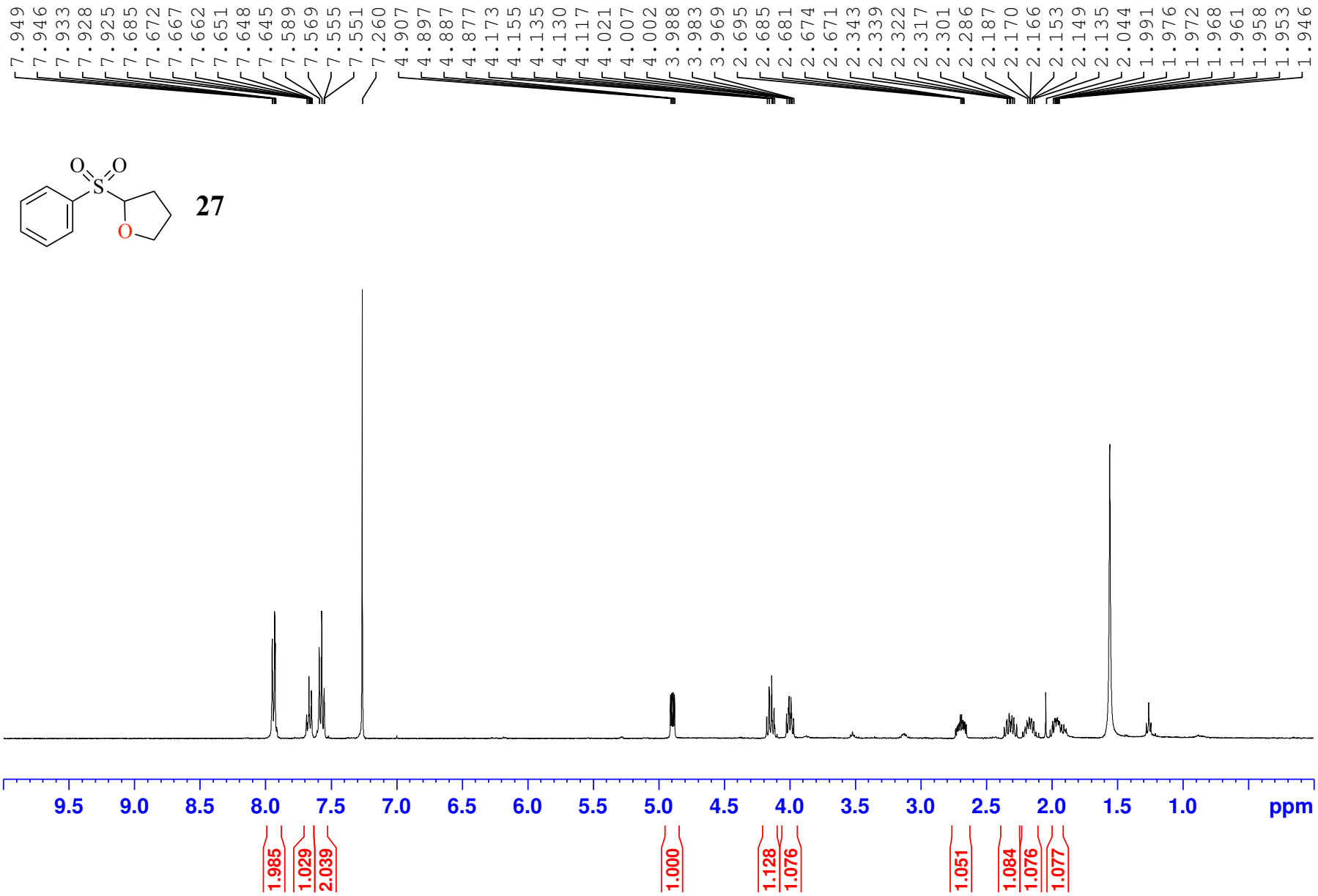
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
ppm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7.645
7.589
7.569
7.555
7.551
7.260
4.907
4.897
4.887
4.877
4.173
4.155
4.135
4.130
4.117
4.021
4.007
4.002
3.988
3.983
3.969
2.695
2.685
2.681
2.674
2.671
2.343
2.339
2.322
2.317
2.301
2.286
2.187
2.170
2.166
2.153
2.149
2.135
2.044
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1.976
1.972
1.968
1.961
1.958
1.953
1.946
O
O
S
O
27
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
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1.051
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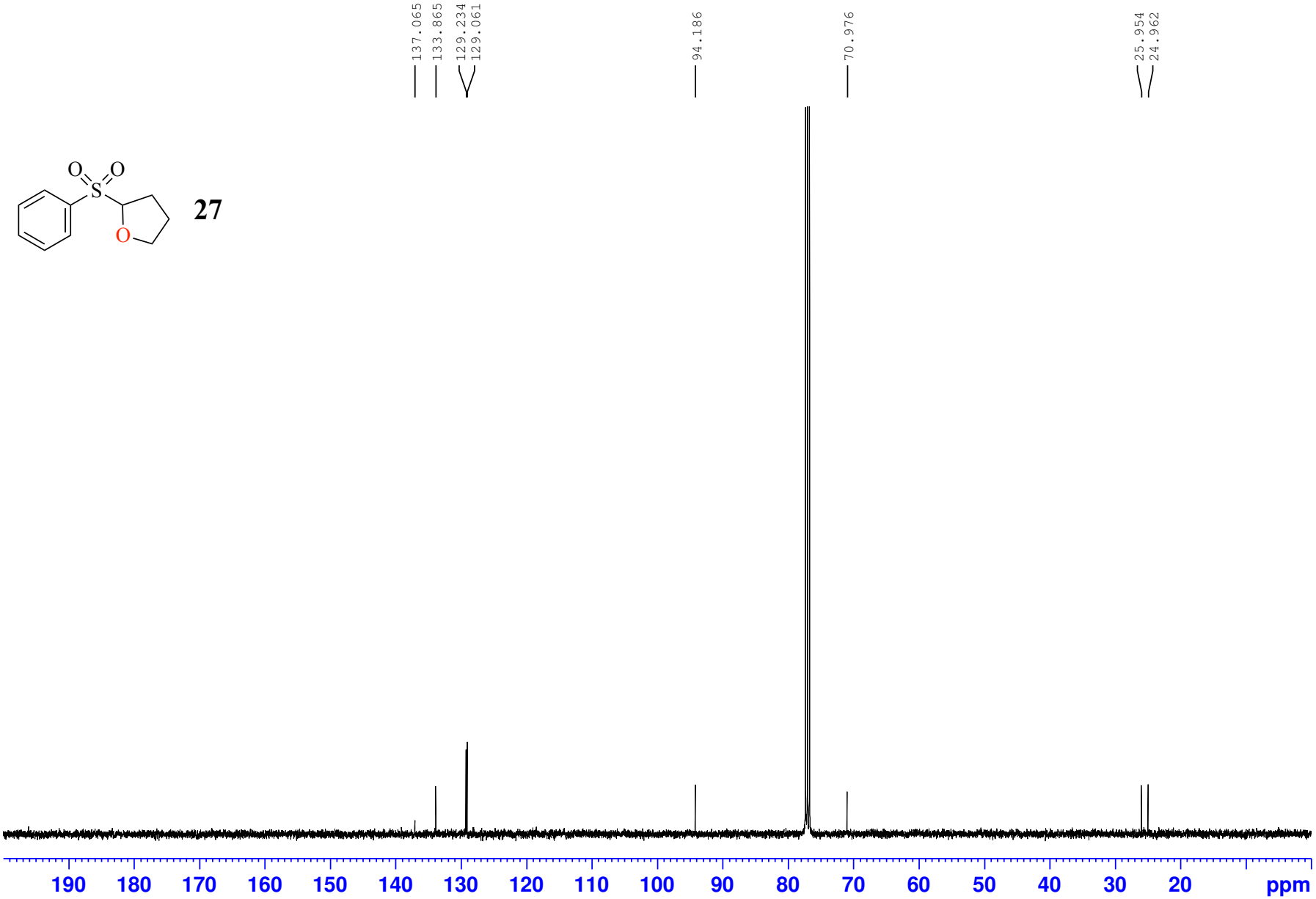
1.076
1.077

27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150
140
130
120
110
100
90
80
70
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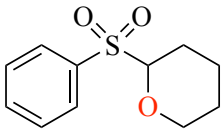
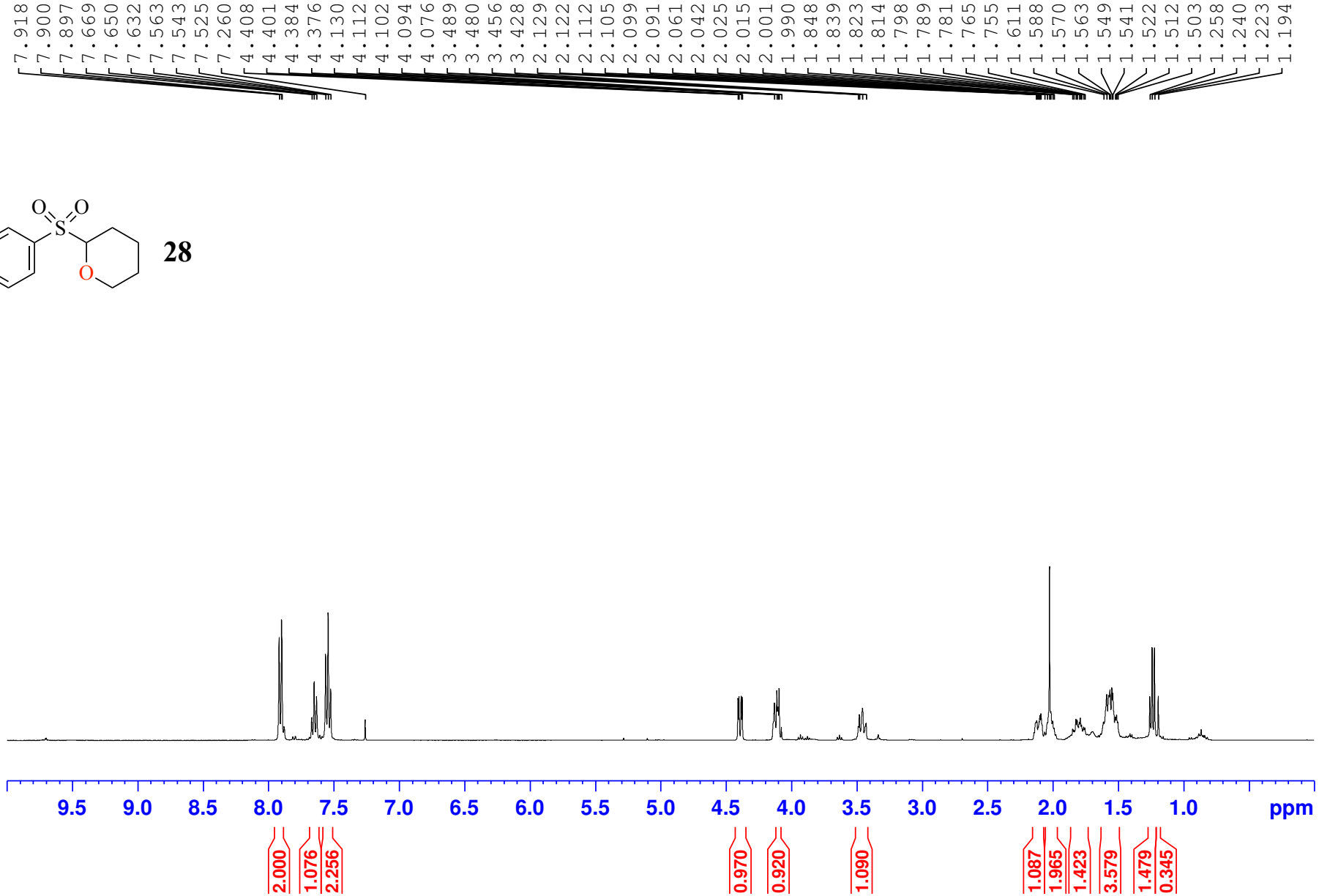
50
40
30
20
ppm



28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7.543
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7.260
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4.401
4.384
4.376
4.130
4.112
4.102
4.094
4.076
3.489
3.480
3.456
3.428
2.129
2.122
2.112
2.105
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2.015
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1.839
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1.755
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1.588
1.570
1.563
1.549
1.541
1.522
1.512
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1.240
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7.0
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6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
ppm
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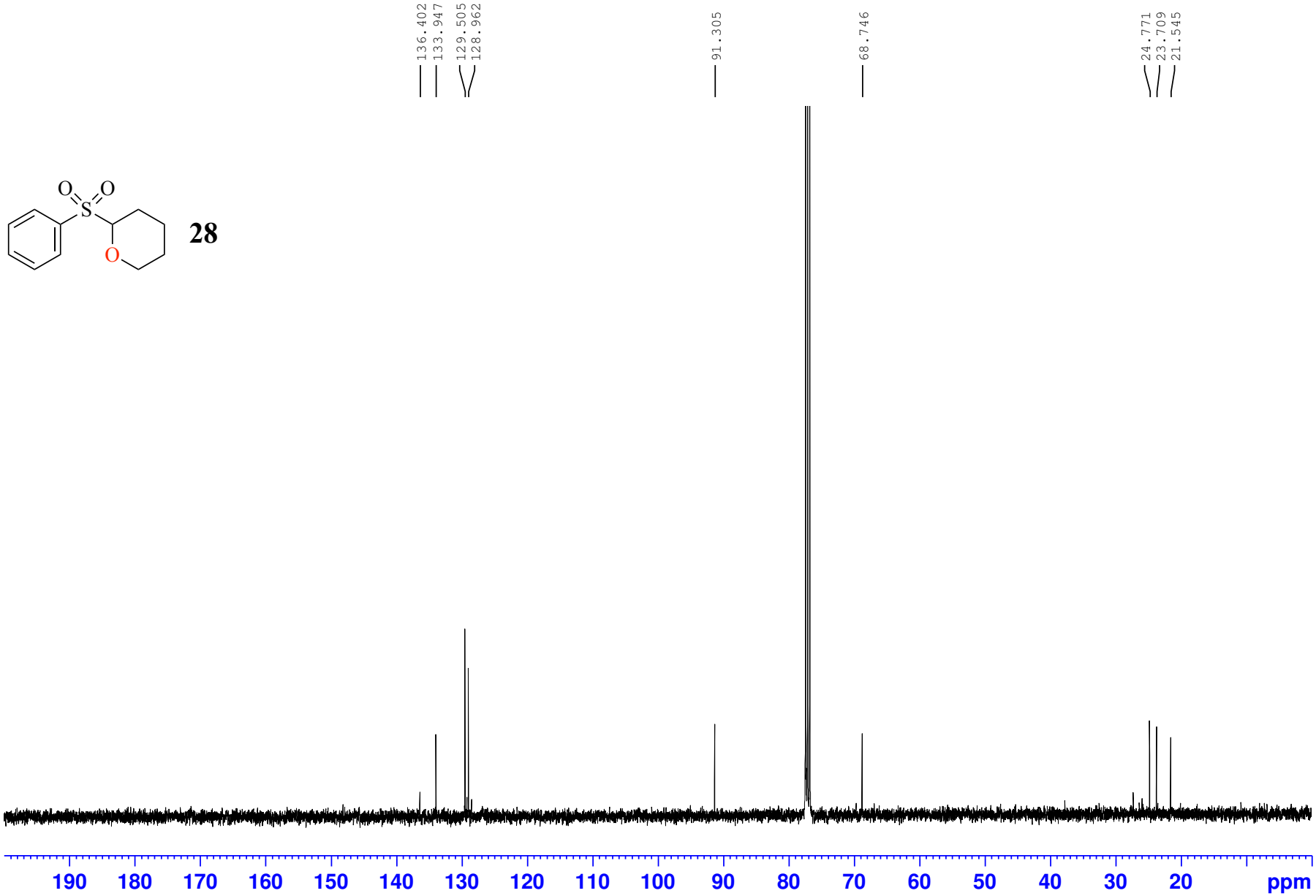
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O
O
S
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150
140
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120
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100
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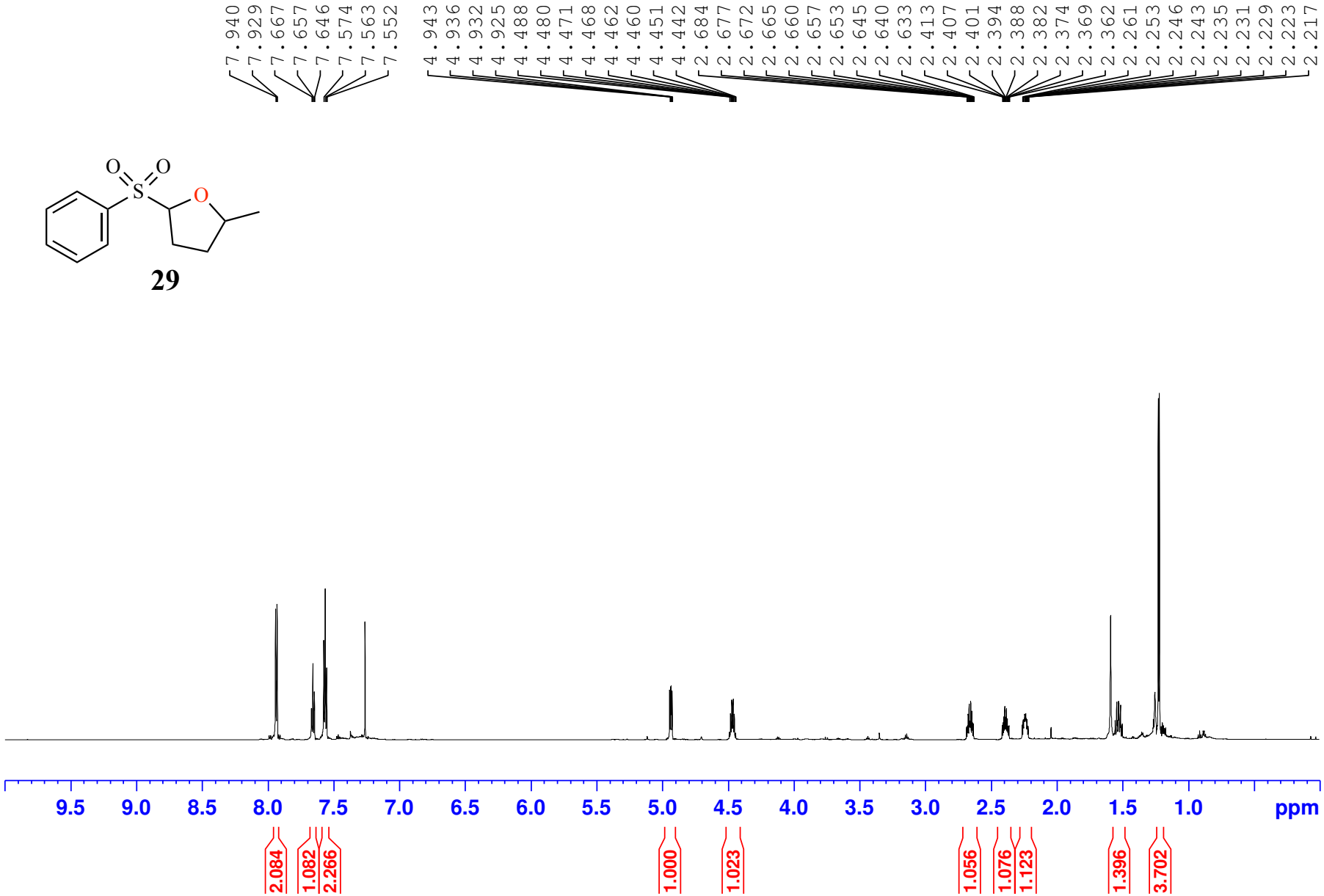
80
70
60
50
40
30
20
ppm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4.488
4.480
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4.468
4.462
4.460
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4.442
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2.677
2.672
2.665
2.660
2.657
2.653
2.645
2.640
2.633
2.413
2.407
2.401
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2.382
2.374
2.369
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2.253
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2.243
2.235
2.231
2.229
2.223
2.217
O
O
S
O
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8.0
7.5
7.0
6.5
6.0
5.5
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4.5
4.0
3.5
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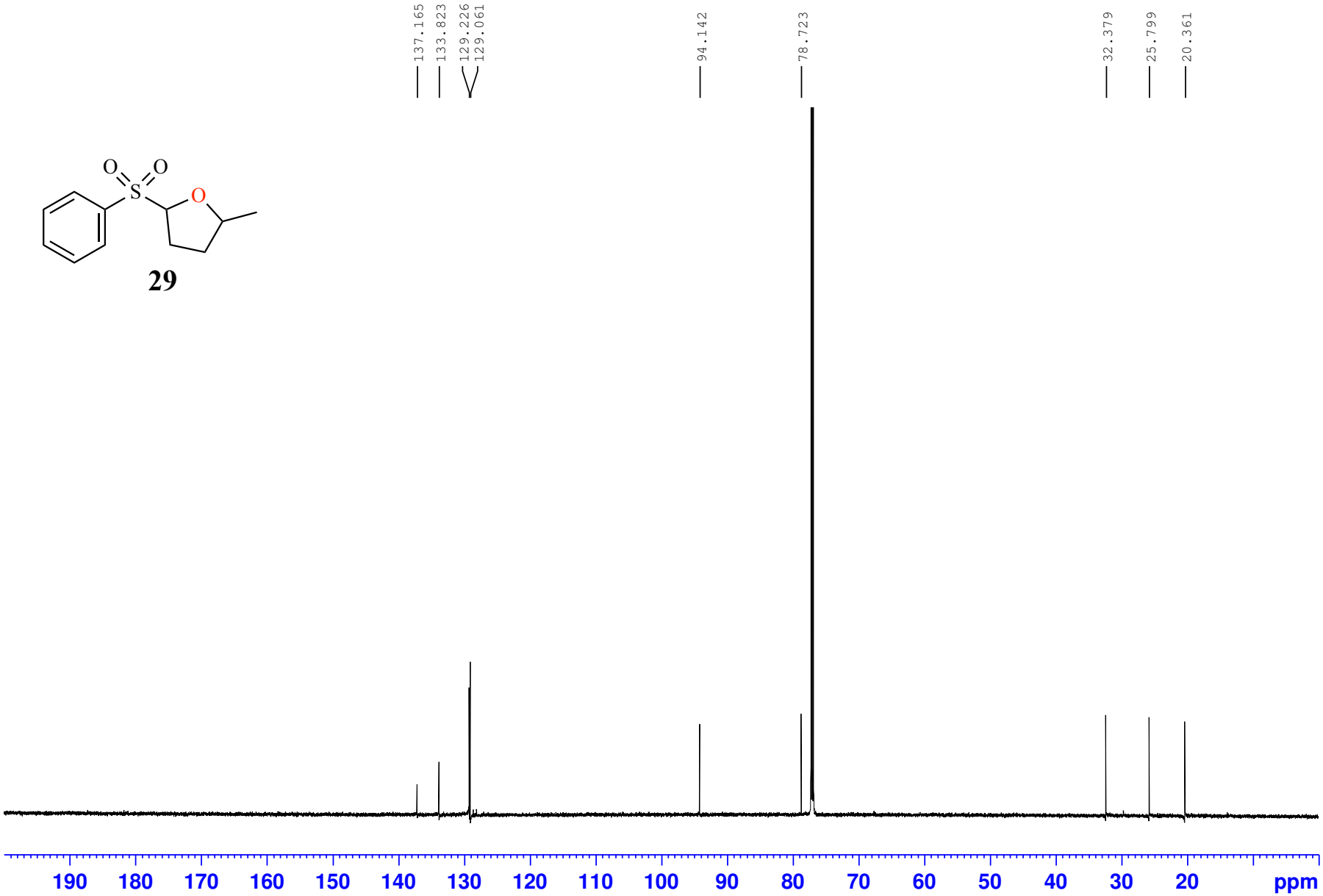
29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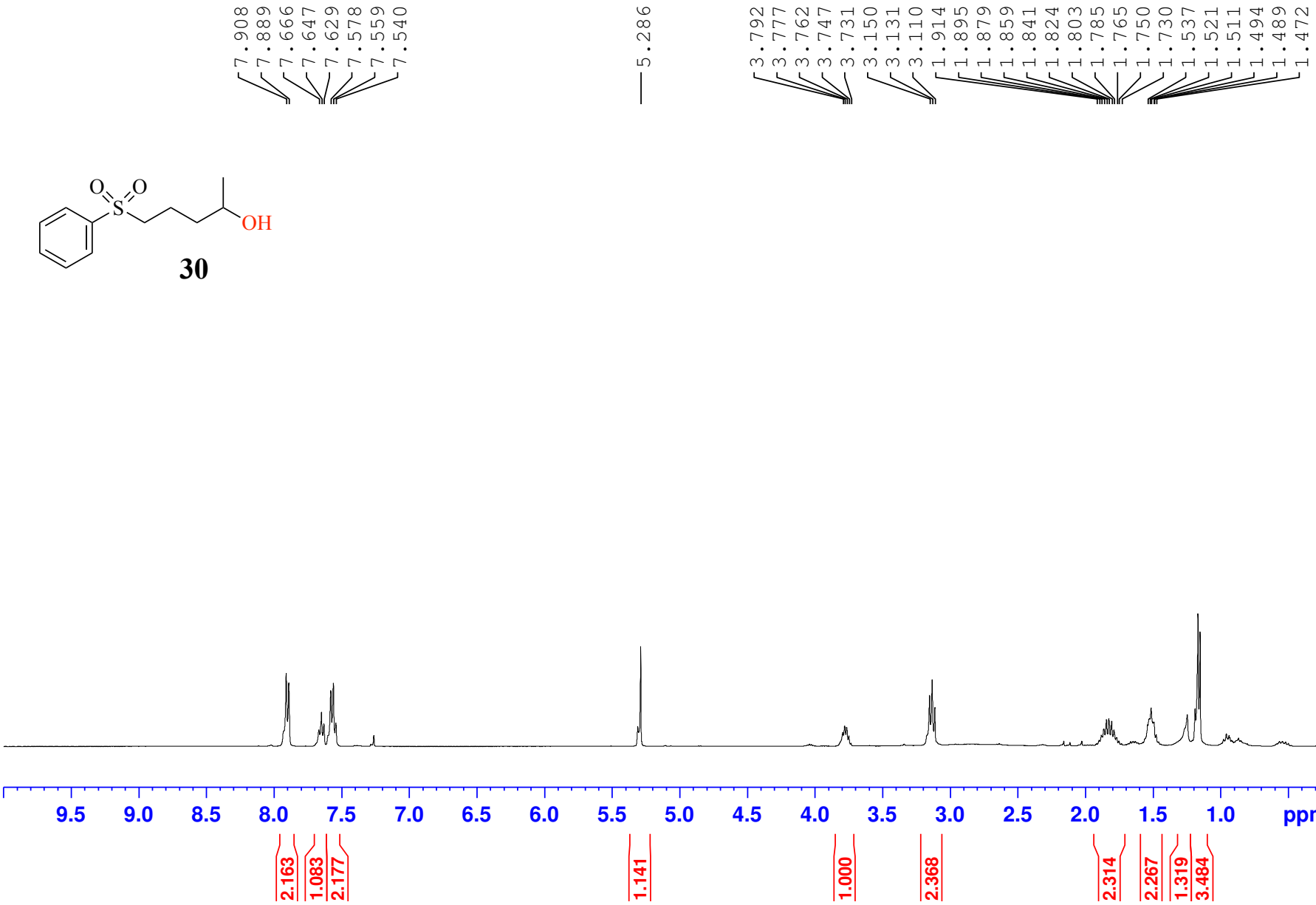
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
ppm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7.578
7.559
7.540
5.286
3.792
3.777
3.762
3.747
3.731
3.150
3.131
3.110
1.914
1.895
1.879
1.859
1.841
1.824
1.803
1.785
1.765
1.750
1.730
1.537
1.521
1.511
1.494
1.489
1.472
O
O
S
OH
30
9.5
9.0
8.5
8.0
7.5
7.0
6.5
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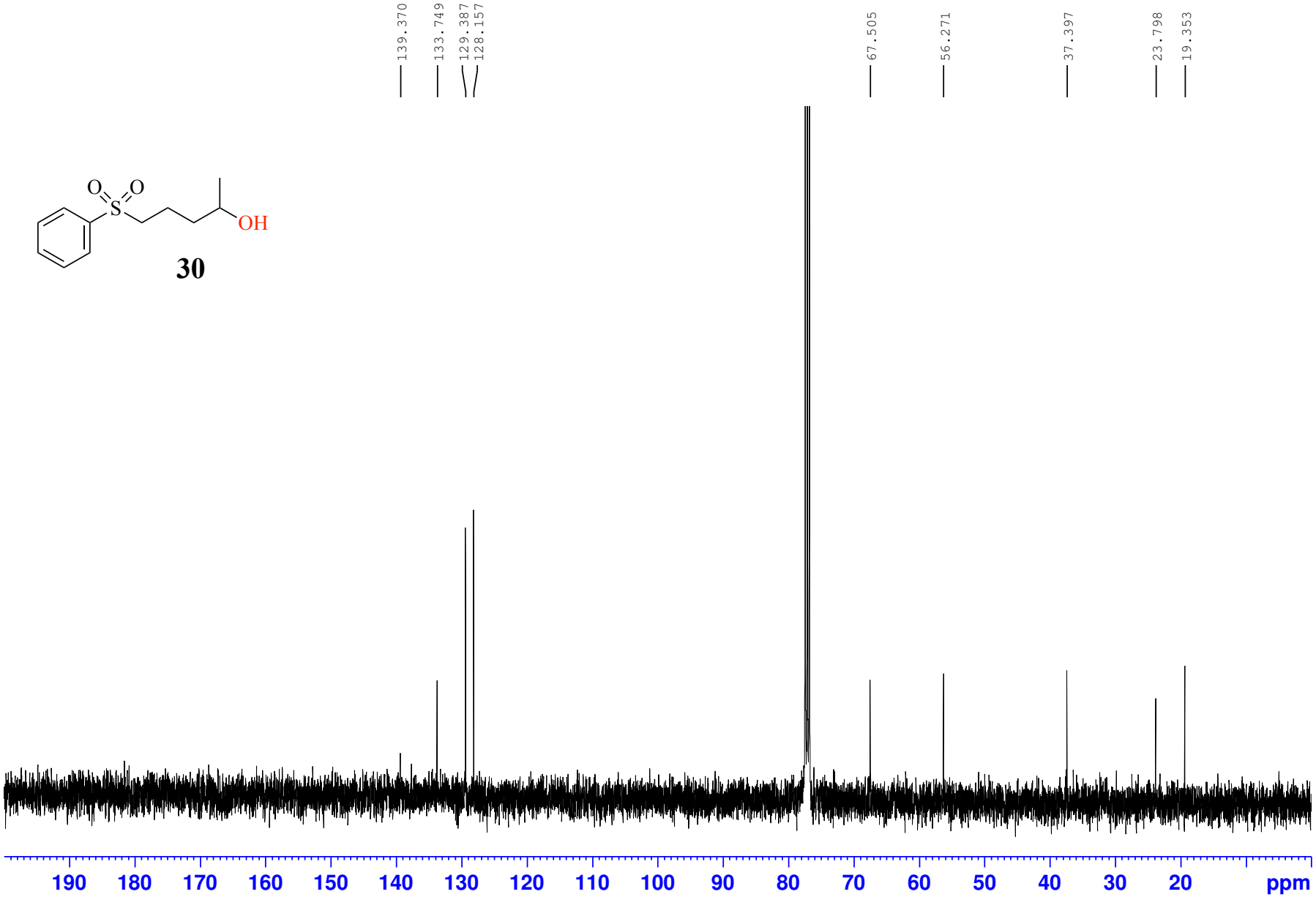
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ppm