10.1071/CH13654_AC

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Supplementary Material

Regioselective Multicomponent Sequential Synthesis of Oxa-Aza[3.3.3]propellanes

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[B]Department of Chemistry, Zhejiang University, Hangzhou 310027, PR China.

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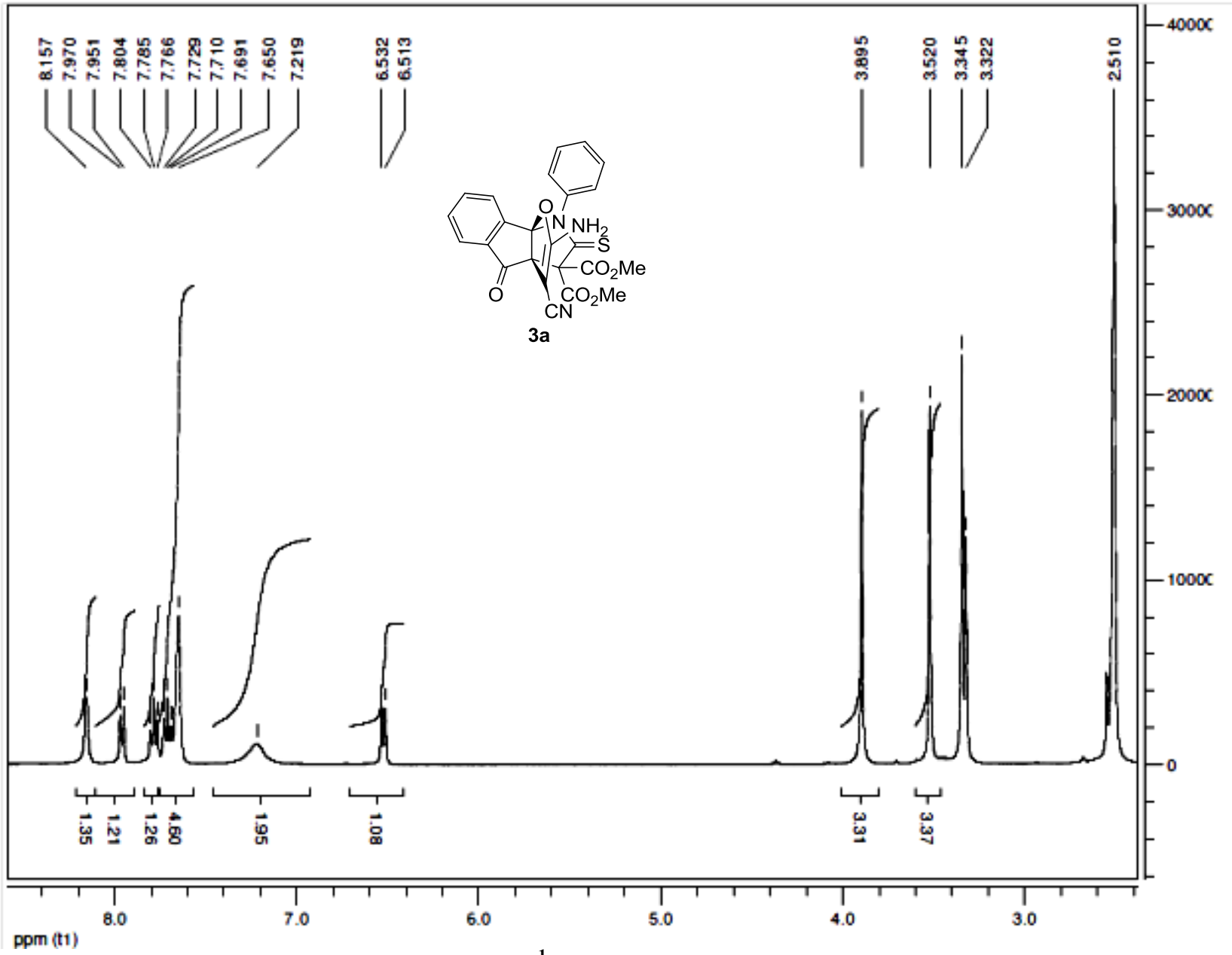
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6.513
3.895
3.520
3.345
3.322
2.510
O
N
NH2
S
CO2Me
CO2Me
CN
O
3a
1.35
1.21
1.26
4.60
1.95
1.08
3.31
3.37
8.0
7.0
6.0
5.0
4.0
3.0
ppm (t1)
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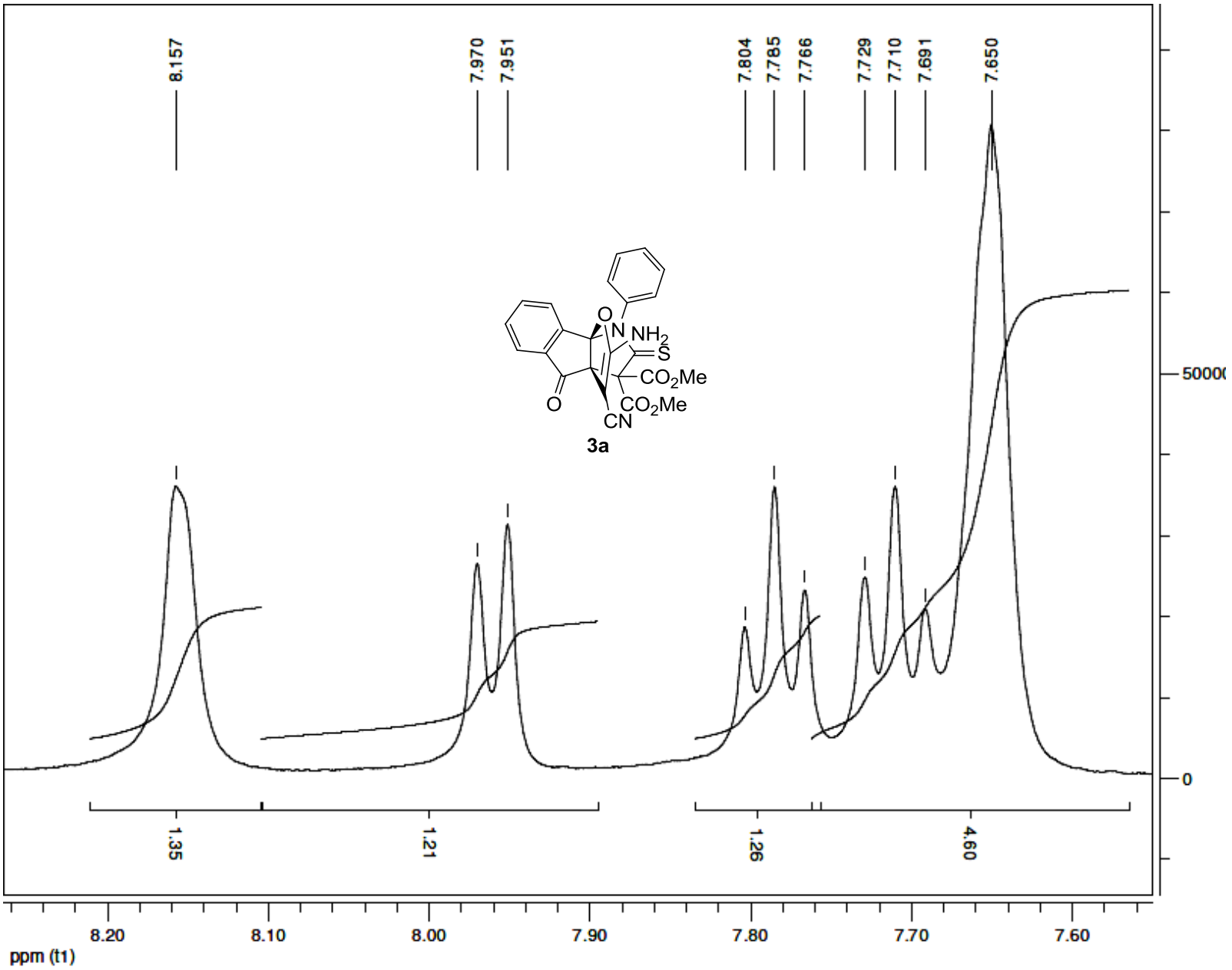
3000
2000
1000
0

^{1}H NMR of 3a



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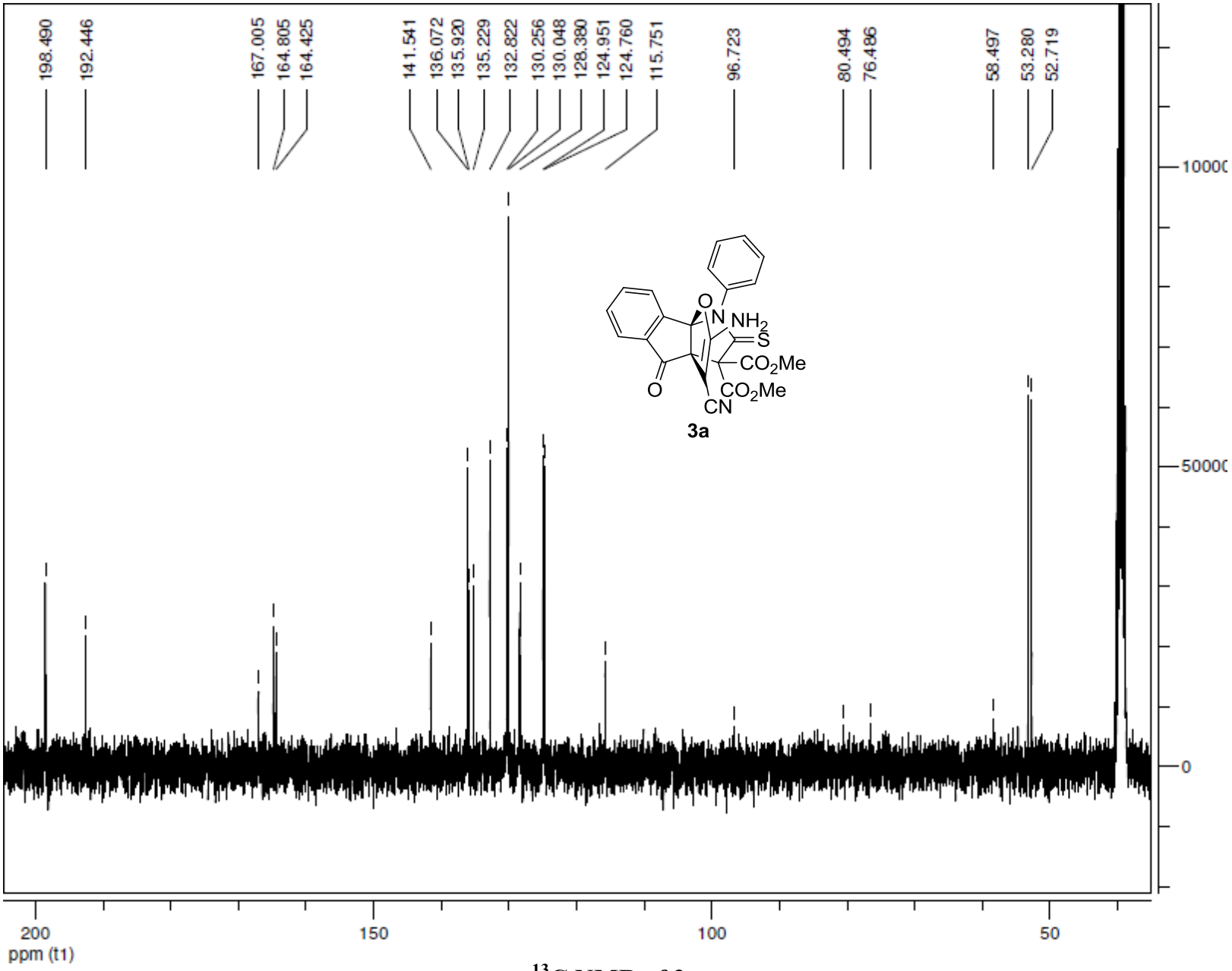
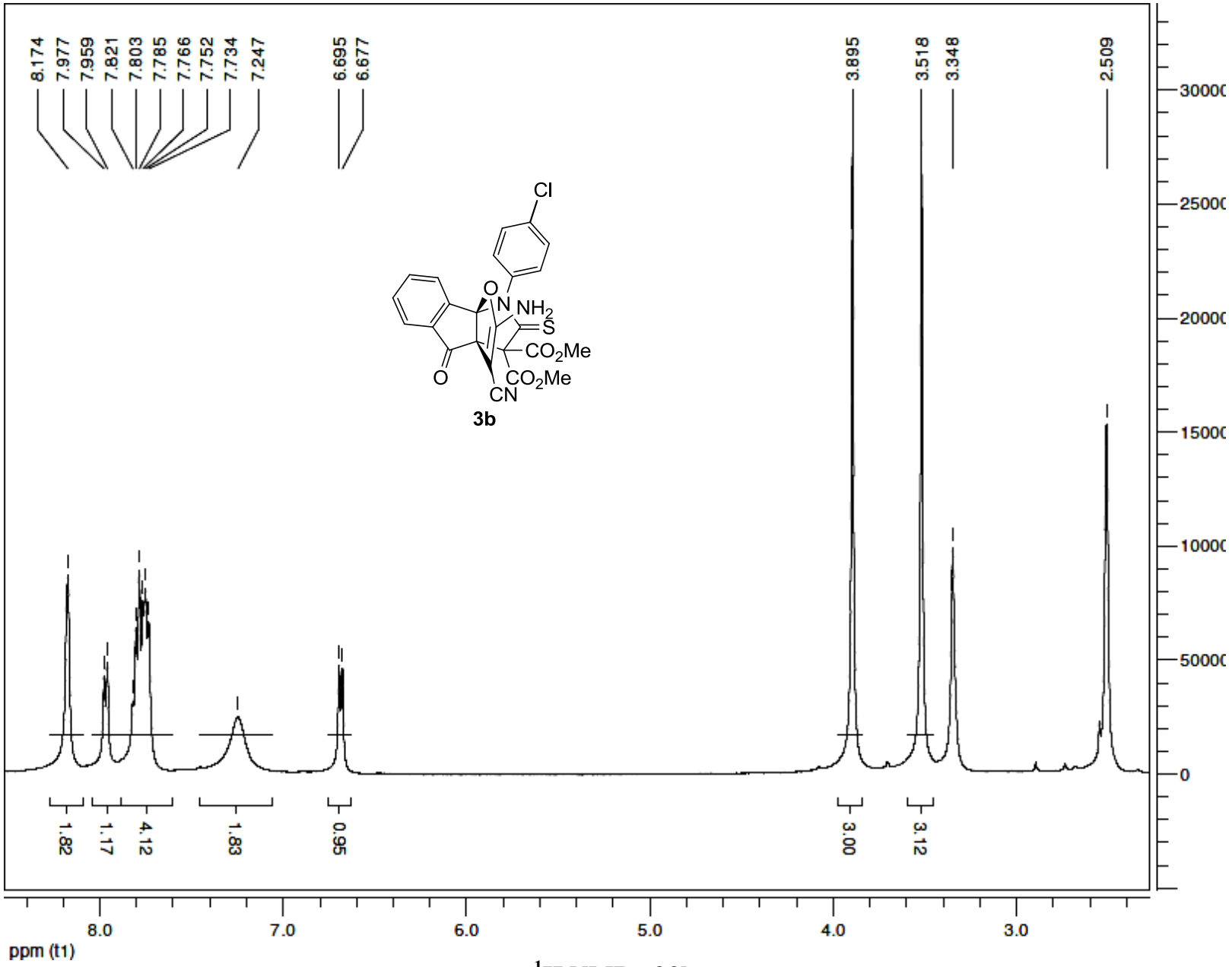


3a

^{13}C NMR of 3a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7.821
7.803
7.785
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7.734
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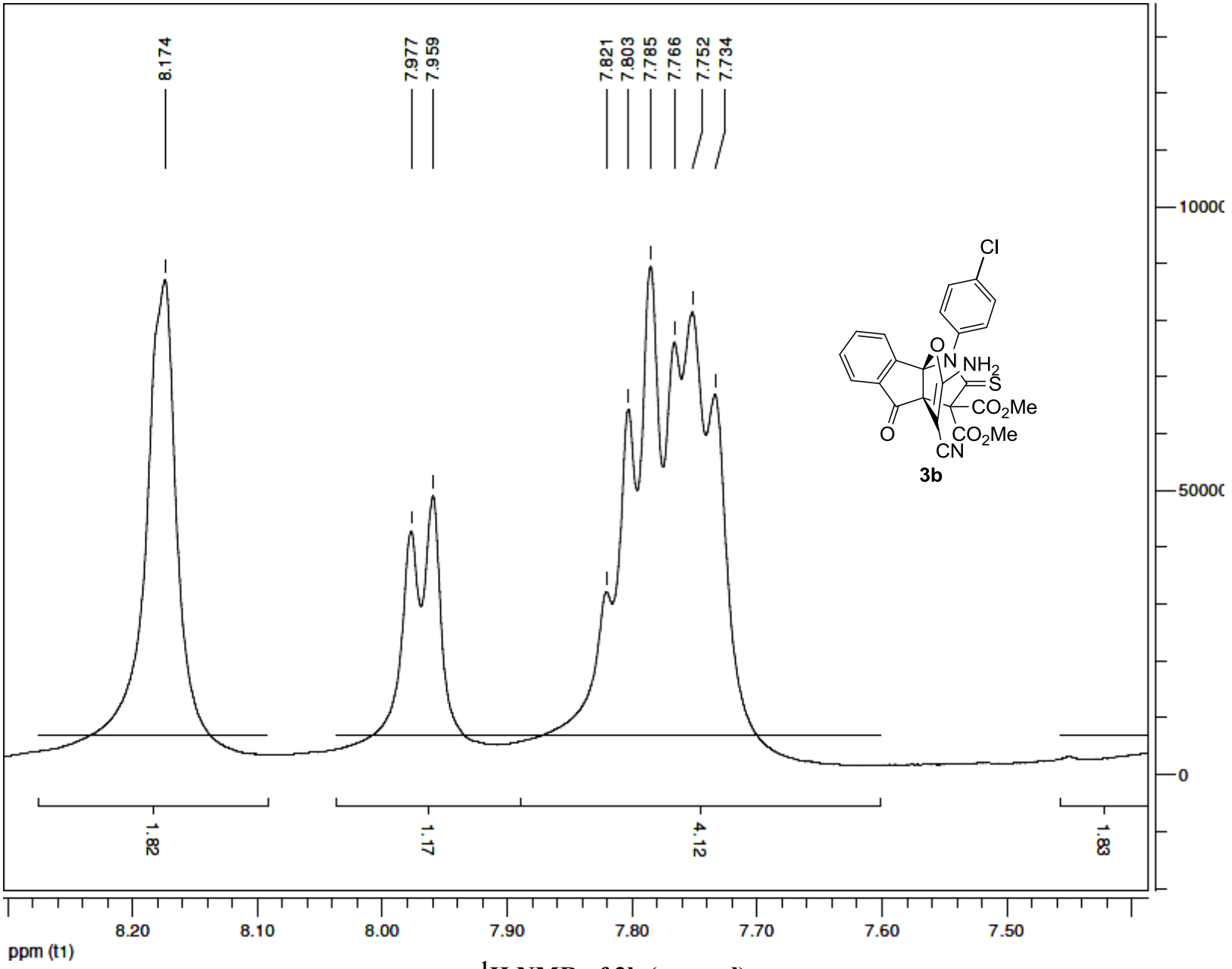
6.677
3.895
3.518
3.348
2.509
Cl
O
N
NH2
S
CO2Me
O
CO2Me
CN
3b
1.82
1.17
4.12
1.83
0.95
3.00
3.12
30000
25000
20000
15000
10000
5000
0
8.0
7.0
6.0
5.0
4.0
3.0
ppm (t1)

^{1}H NMR of 3b



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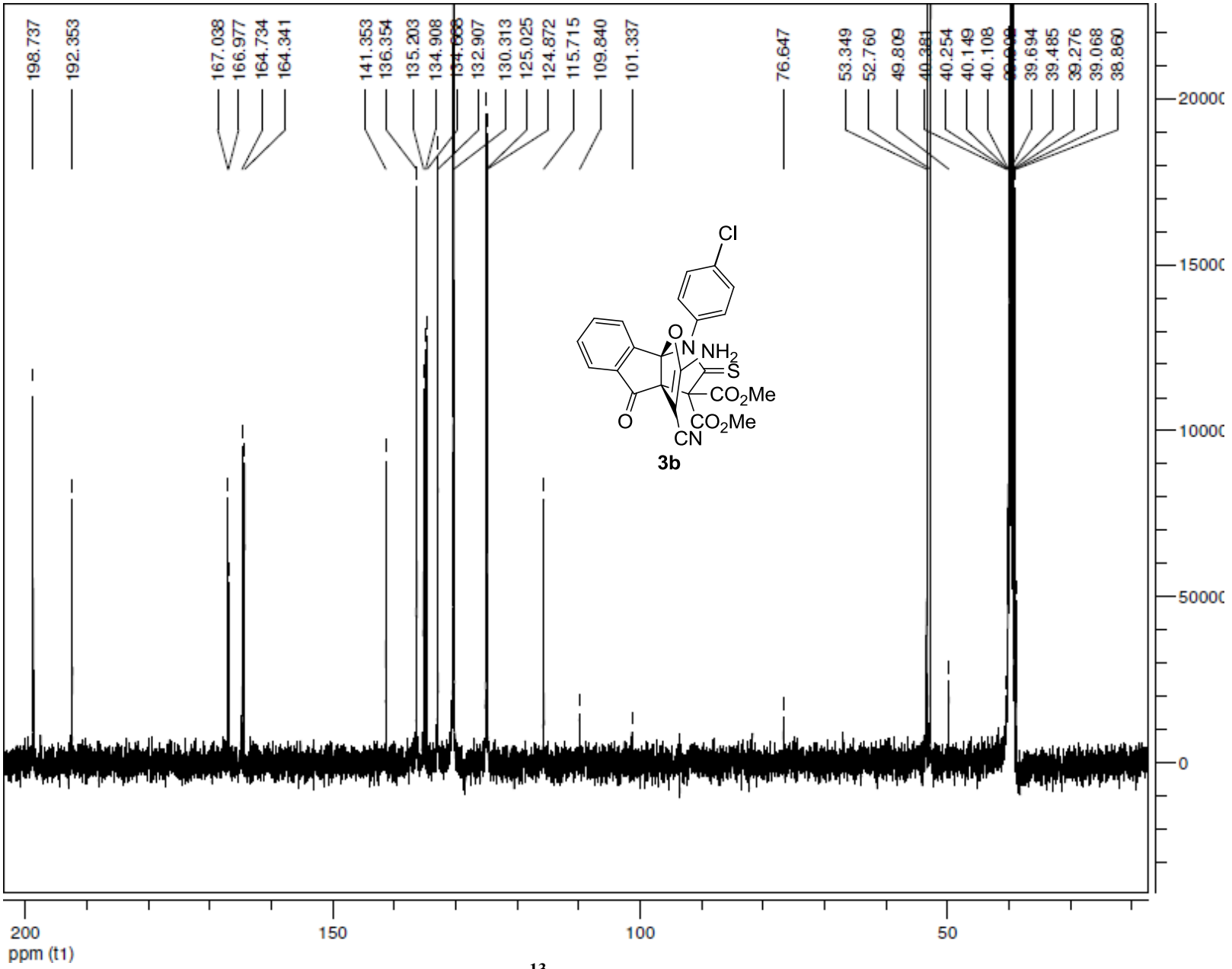
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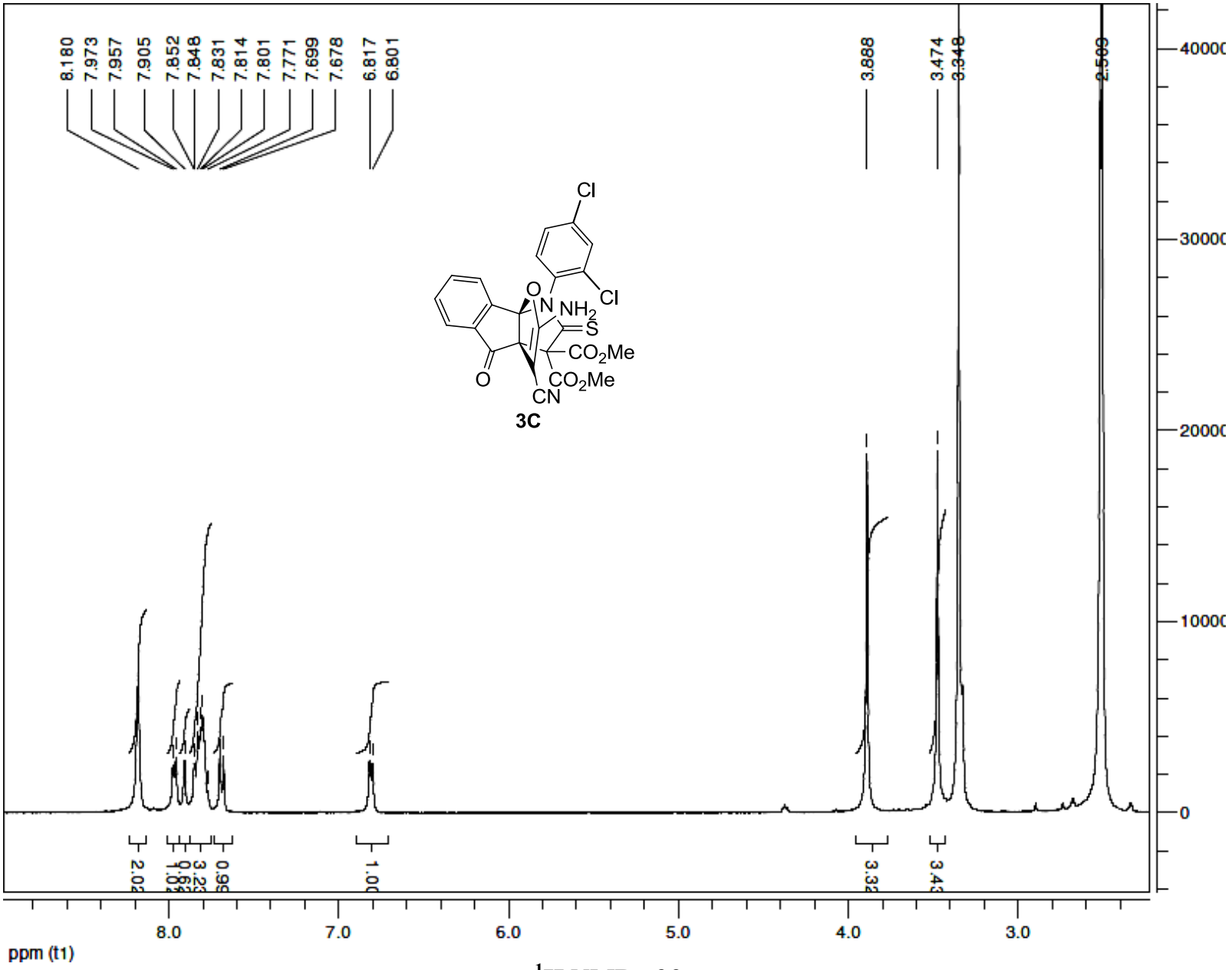
124.872
115.715
109.840
101.337
76.647
53.349
52.760
49.809
40.254
40.149
40.108
39.694
39.485
39.276
39.068
38.860
Cl
O
N
NH2
S
CO2Me
CO2Me
O
CN
3b
200
150
100
50
ppm (t1)
20000
15000
10000
50000
0

^{13}C NMR of 3b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7.801
7.771
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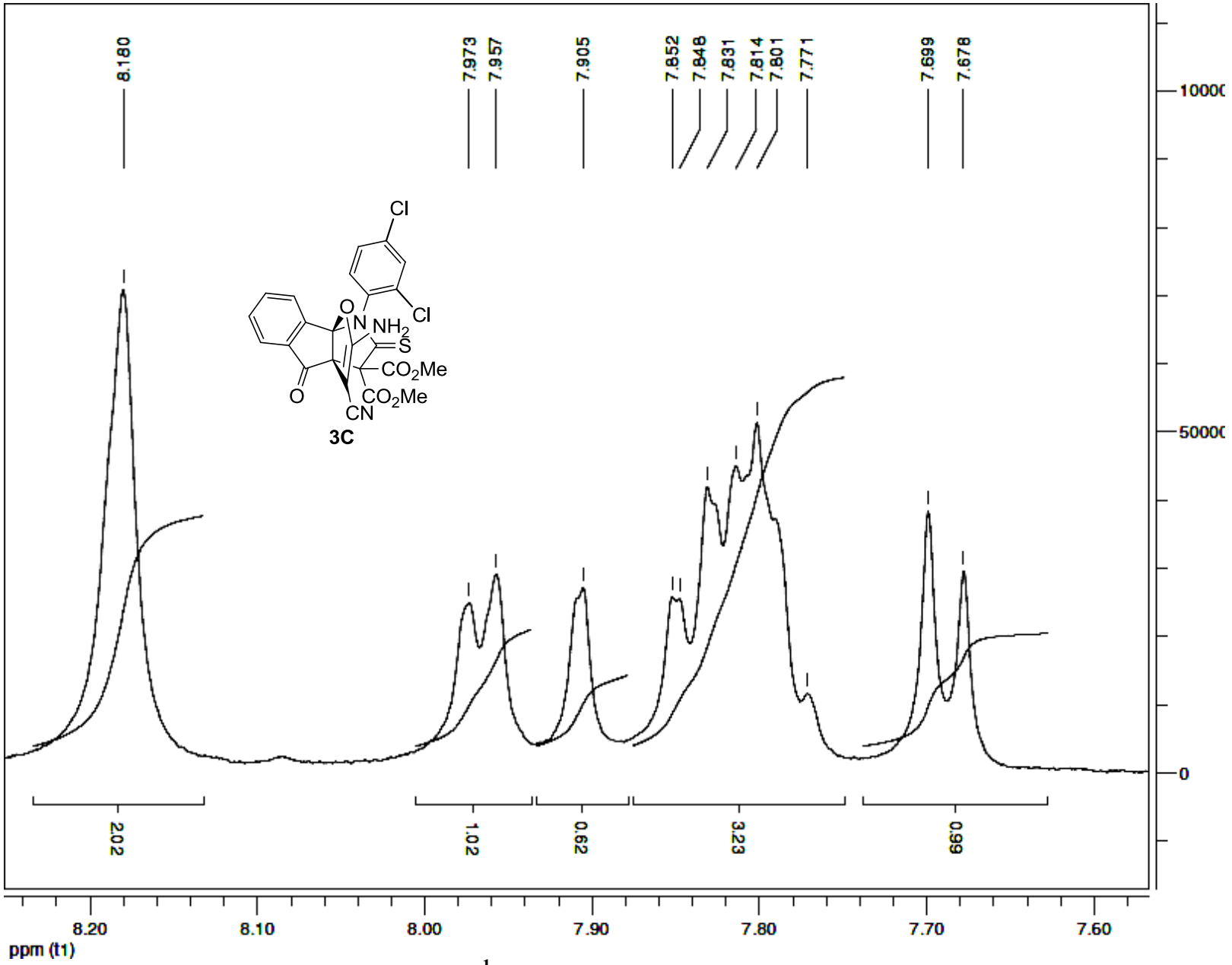
7.678
6.817
6.801
3.888
3.474
3.348
2.503
Cl
Cl
O
N
NH2
S
CO2Me
CO2Me
O
CN
3C
40000
30000
20000
10000
0
2.02
1.04
0.62
3.23
0.99
1.00
3.32
3.43
8.0
7.0
6.0
5.0
4.0
3.0
ppm (t1)

^{1}H NMR of 3c



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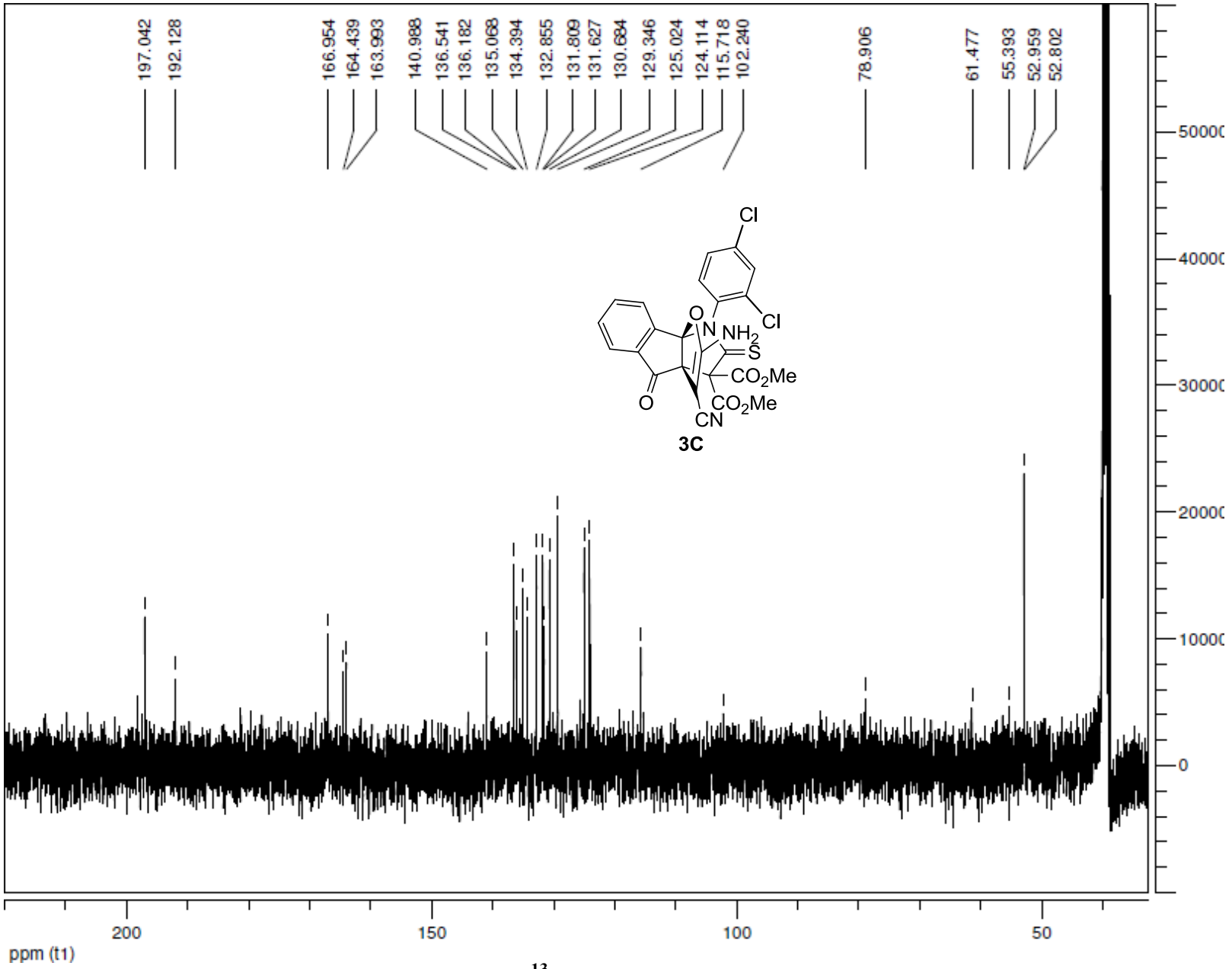
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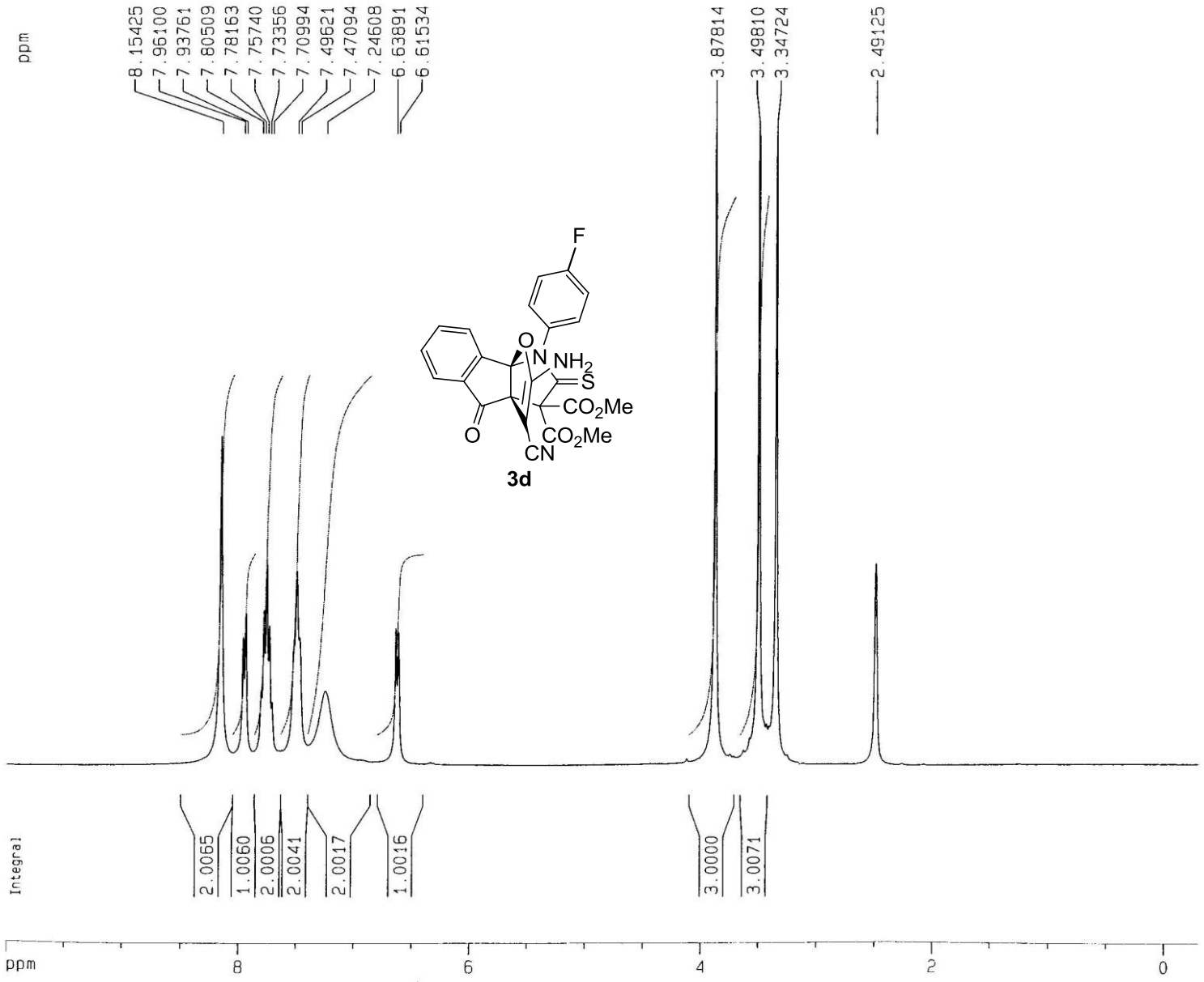
140.988
136.541
136.182
135.068
134.394
132.855
131.809
131.627
130.684
129.346
125.024
124.114
115.718
102.240
78.906
61.477
55.393
52.959
52.802
Cl
Cl
O
N
NH2
S
CO2Me
CO2Me
CN
O
3C
50000
40000
30000
20000
10000
0
200
150
100
50
ppm (t1)

^{13}C NMR of 3c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6.63891
6.61534
3.87814
3.49810
3.34724
2.49125
F
O
N
NH2
S
CO2Me
CO2Me
O
CN
3d
Integral
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1.0060
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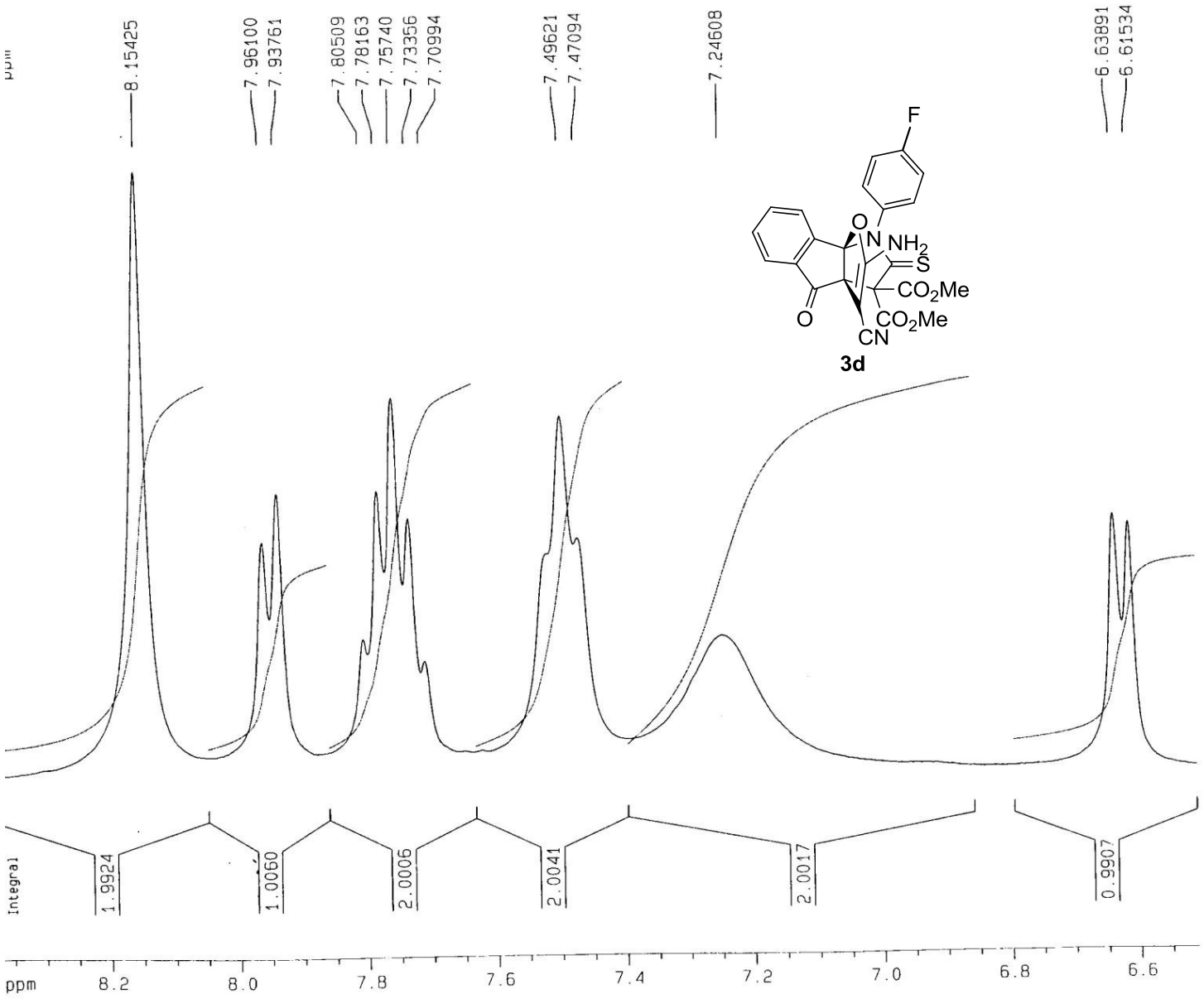
1.0016
3.0000
3.0071
ppm
8
6
4
2
0

^{1}H NMR of 3d



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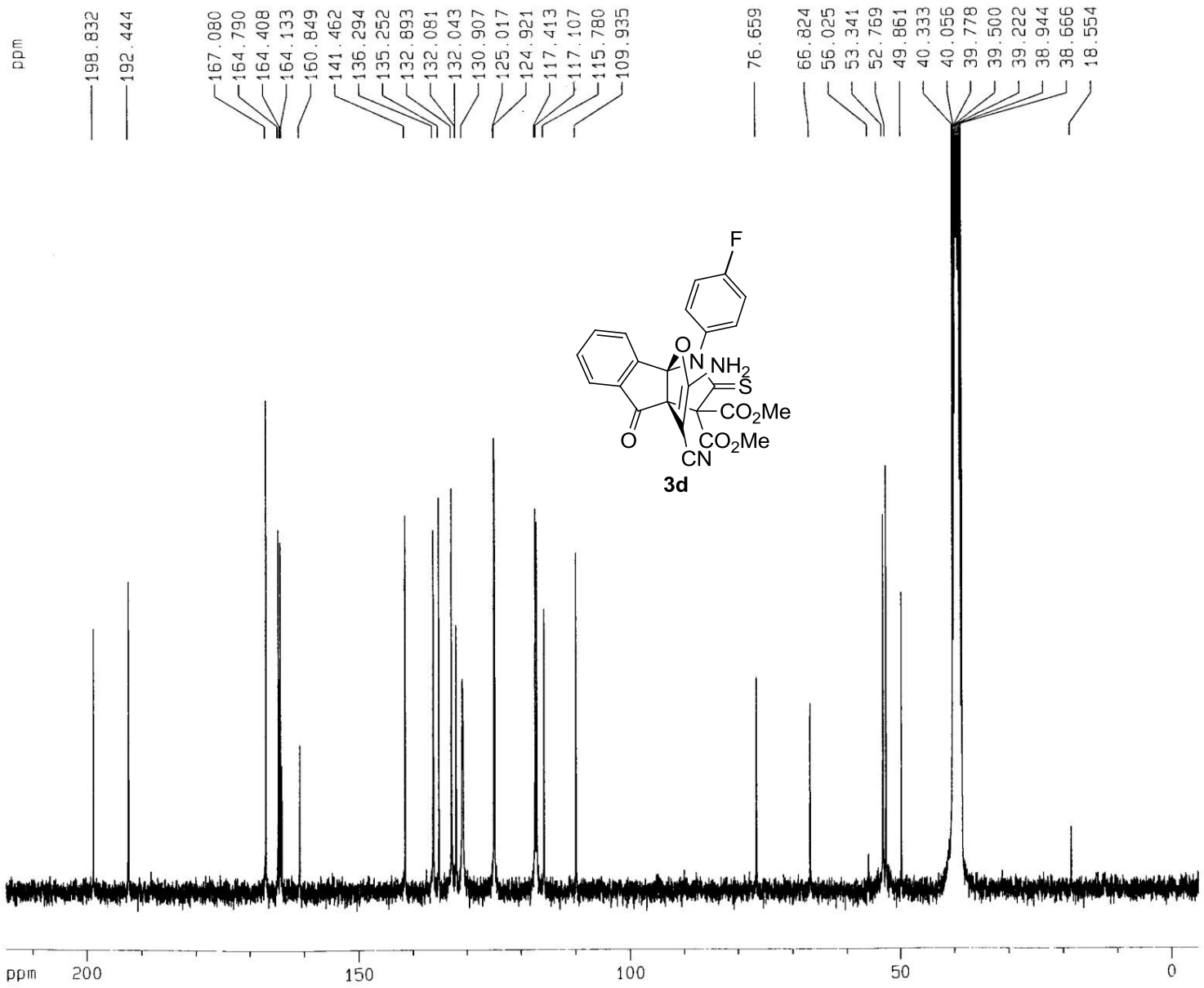
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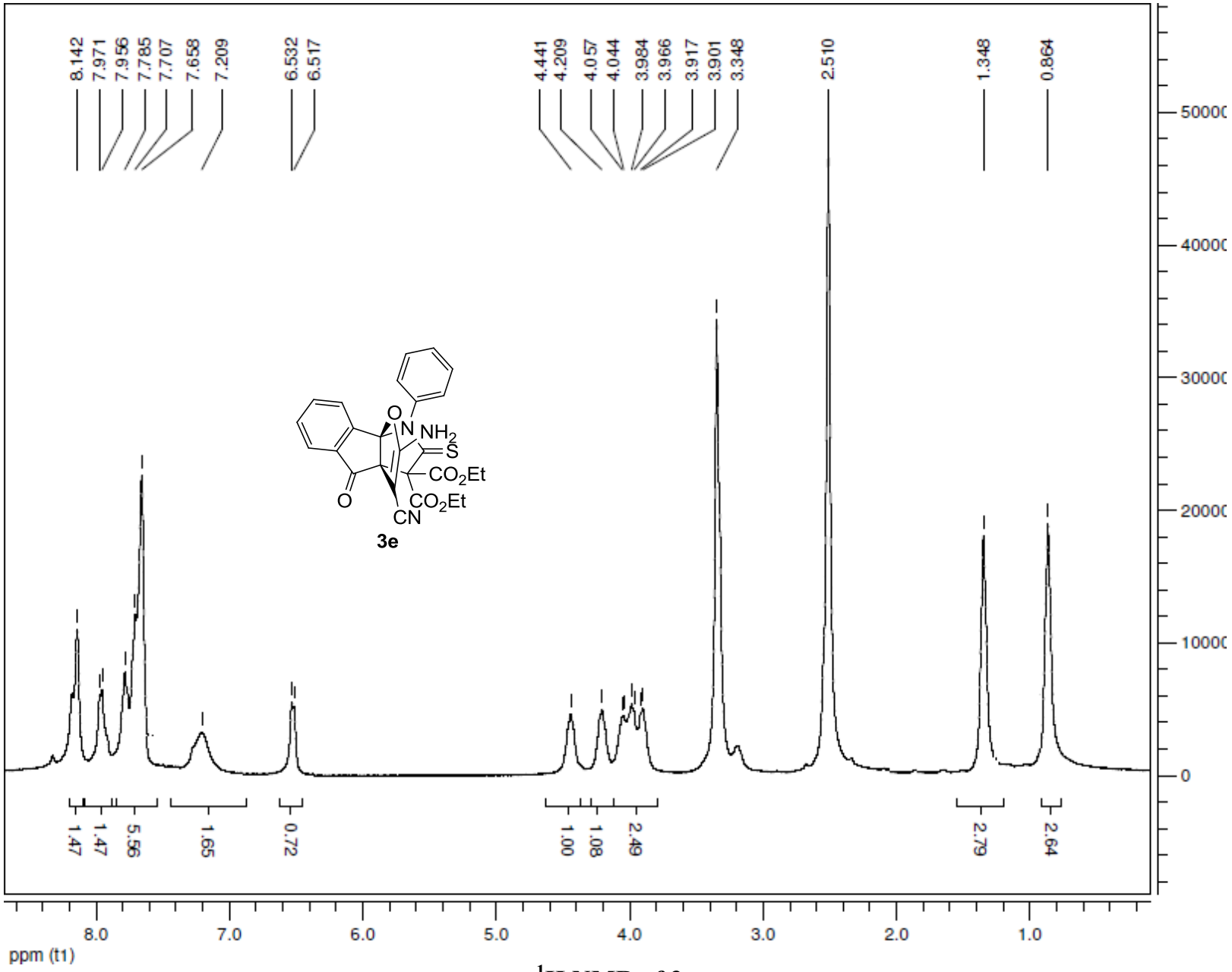
132.043
130.907
125.017
124.921
117.413
117.107
115.780
109.935
76.659
66.824
56.025
53.341
52.769
49.861
40.333
40.056
39.778
39.500
39.222
38.944
38.666
18.554
F
O
N
NH2
S
CO2Me
O
CO2Me
CN
3d
ppm
200
150
100
50
0

^{13}C NMR of 3d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4.441
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4.044
3.984
3.966
3.917
3.901
3.348
2.510
1.348
0.864
O
N
NH2
S
CO2Et
CO2Et
O
CN
3e
1.47
1.47
5.56
1.65
0.72
1.00
1.08
2.49
2.79
2.64
50000
40000
30000
20000
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6.0
5.0
4.0
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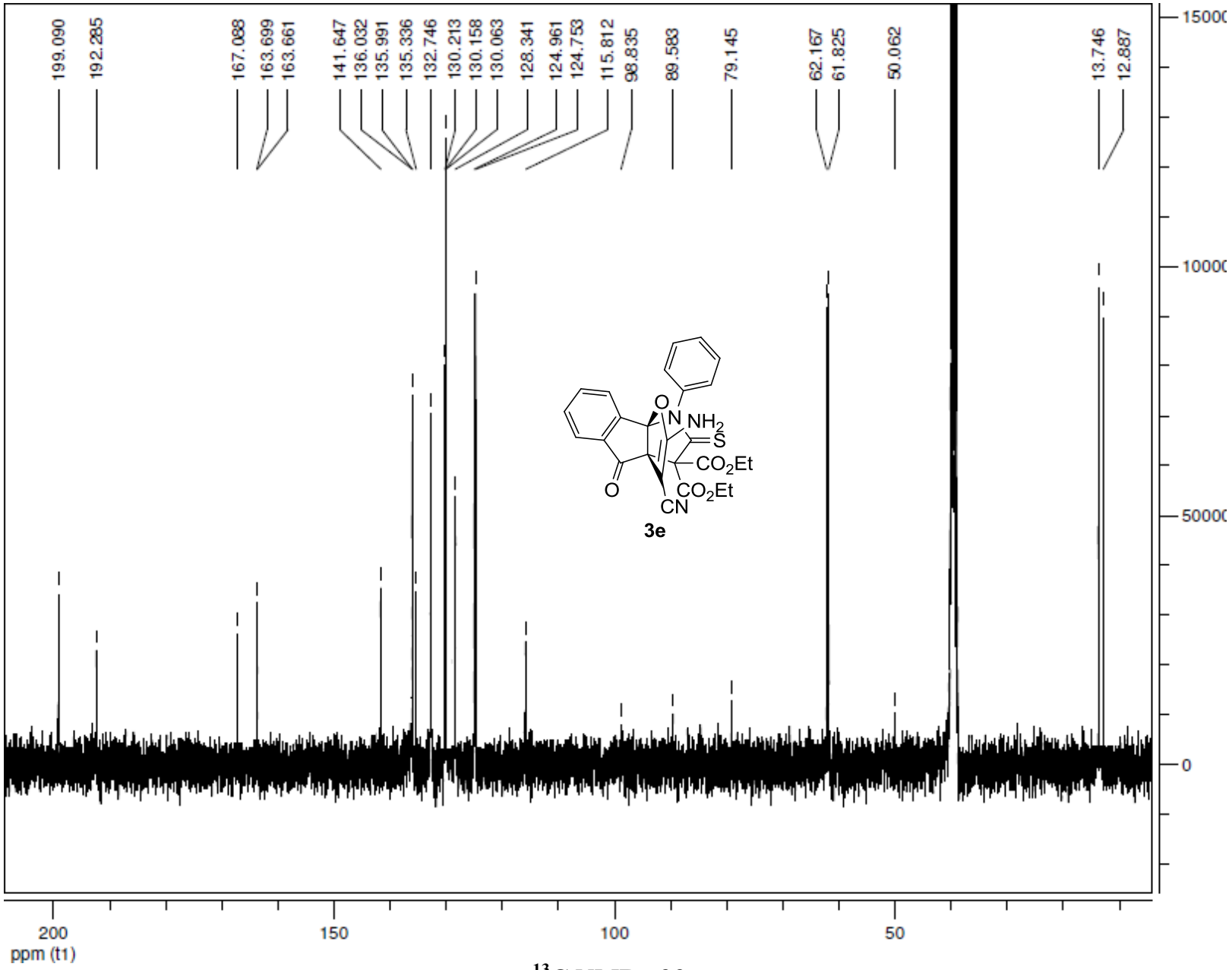
2.0
1.0
ppm (t1)

^{1}H NMR of 3e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130.063
128.341
124.961
124.753
115.812
98.835
89.583
79.145
62.167
61.825
50.062
13.746
12.887
15000
10000
50000
0
O
N
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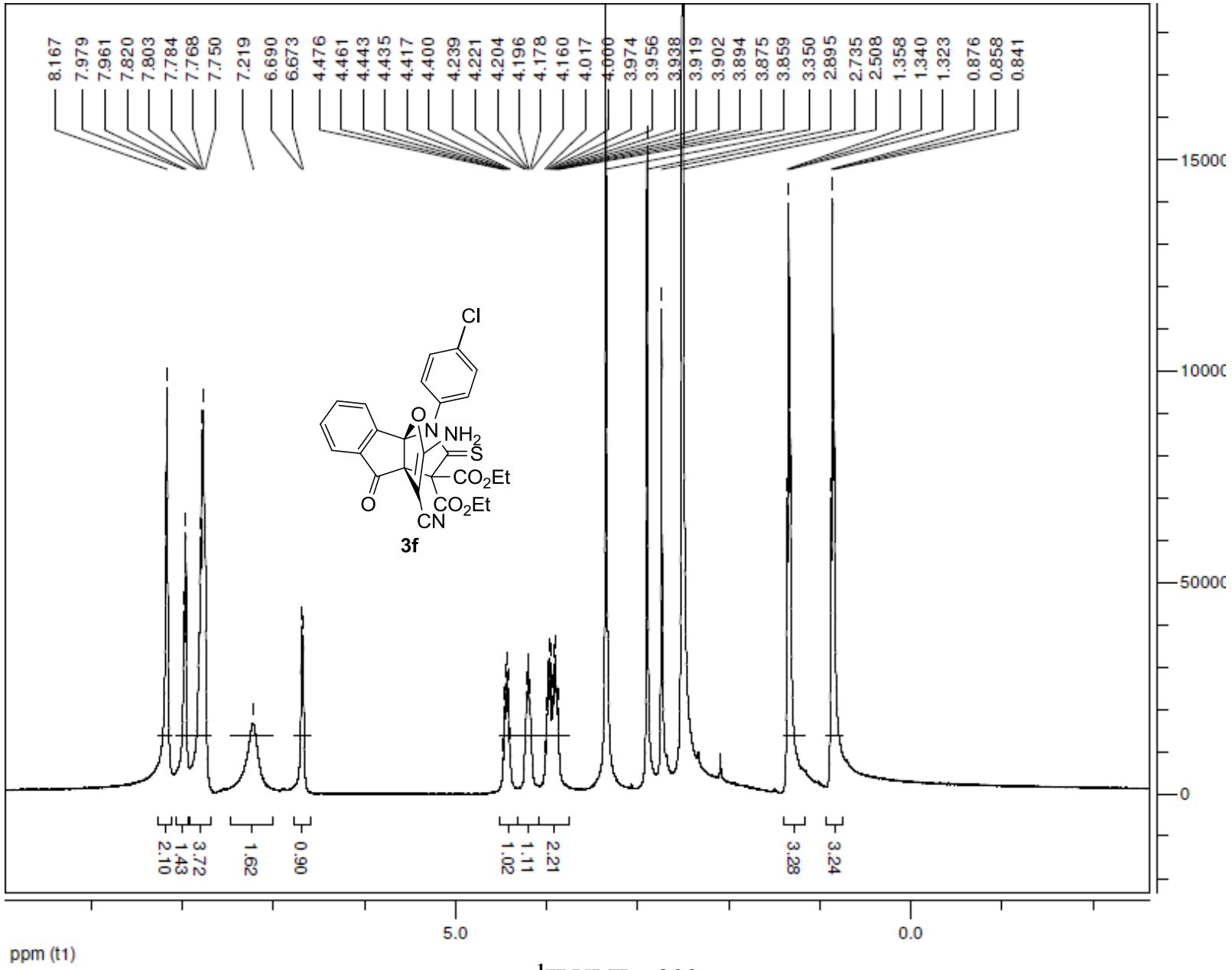
S
CO2Et
O
CO2Et
CN
3e
200
150
100
50
ppm (t1)

^{13}C NMR of 3e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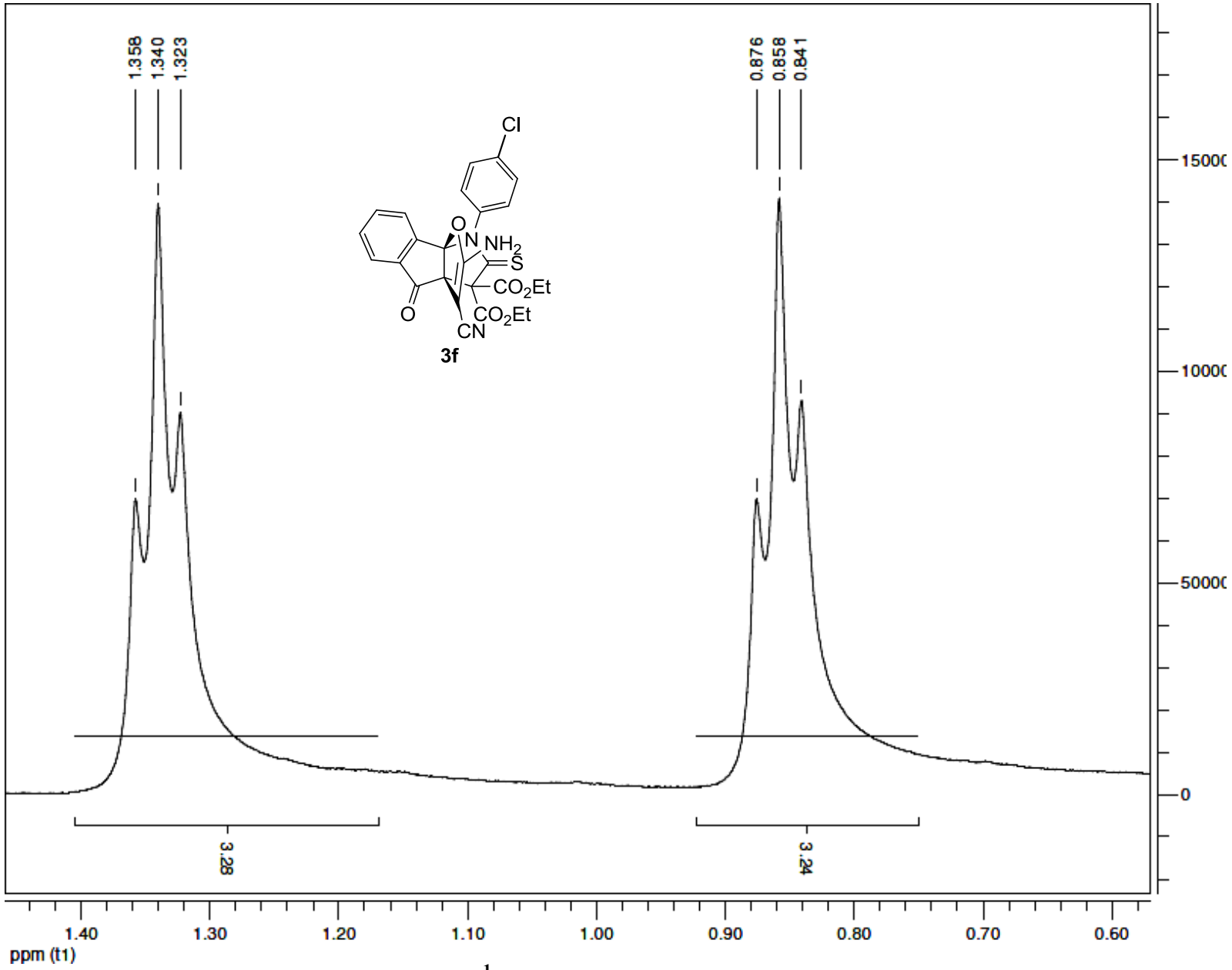
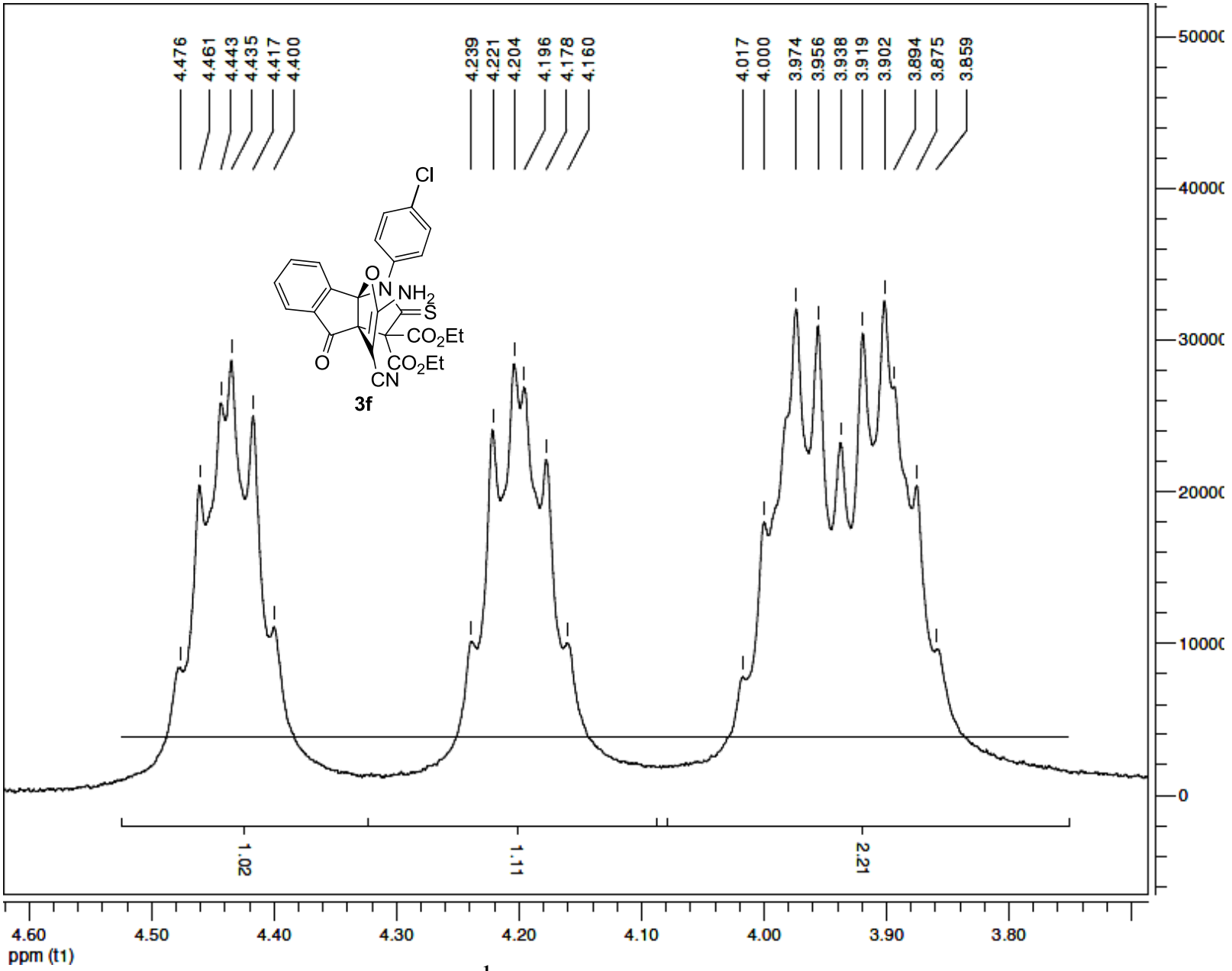
7.820
7.803
7.784
7.768
7.750
7.219
6.690
6.673
4.476
4.461
4.443
4.435
4.417
4.400
4.239
4.221
4.204
4.196
4.178
4.160
4.017
4.000
3.974
3.956
3.938
3.919
3.902
3.894
3.875
3.859
3.350
2.895
2.735
2.508
1.358
1.340
1.323
0.876
0.858
0.841
Cl
O
N
NH2
S
CO2Et
O
CO2Et
CN
3f
15000
10000
50000
0
2.10
1.43
3.72
1.92
0.90
1.02
1.11
2.21
3.28
3.24
5.0
0.0
ppm (t1)

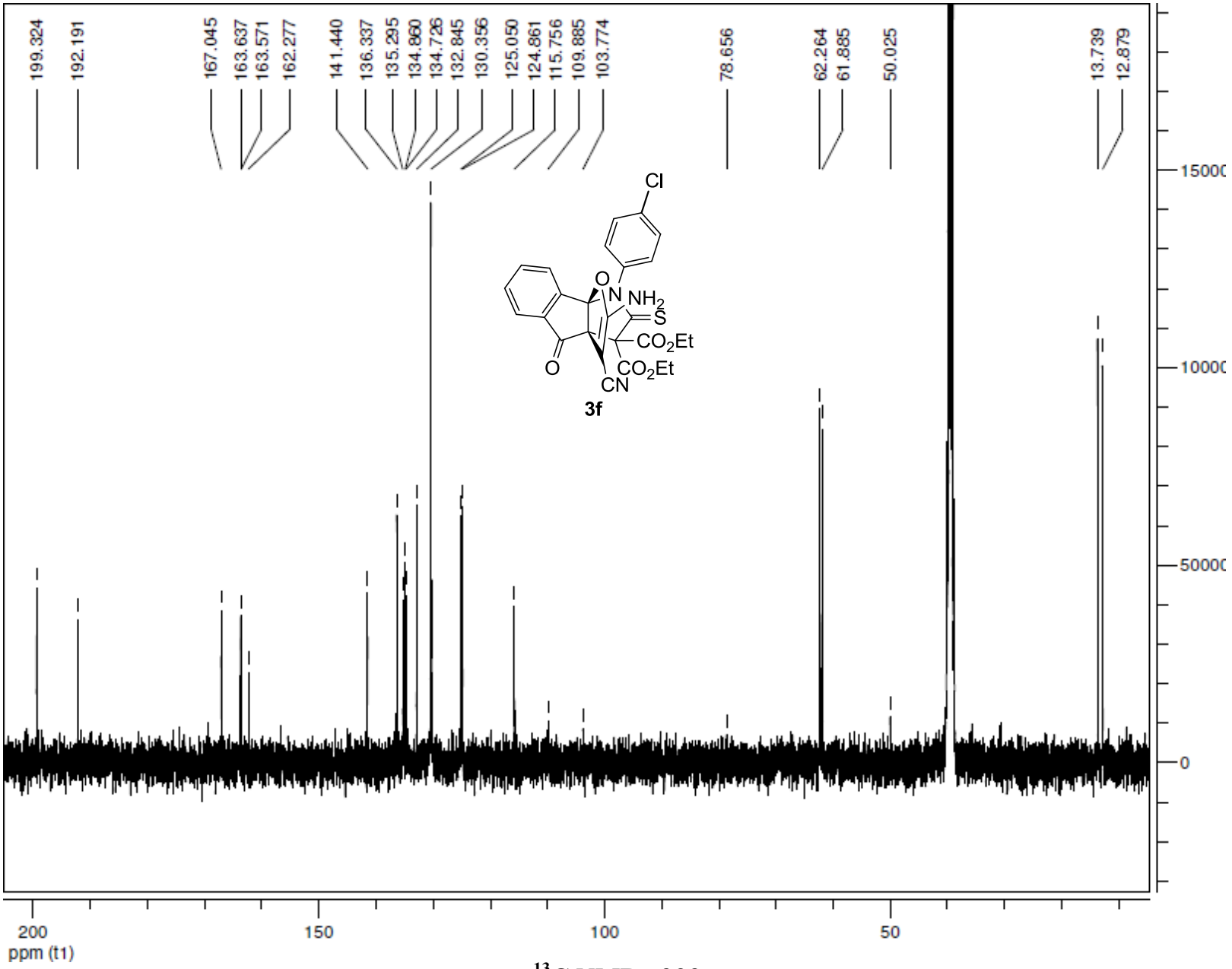
^{1}H NMR of 3f



^{1}H NMR of 3f (expand)



^{1}H NMR of 3f (expand)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167.045
163.637
163.571
162.277
141.440
136.337
135.295
134.860
134.726
132.845
130.356
125.050
124.861
115.756
109.885
103.774
78.656
62.264
61.885
50.025
13.739
12.879
Cl
O
N
NH2
S
CO2Et
CO2Et
O
CN
3f
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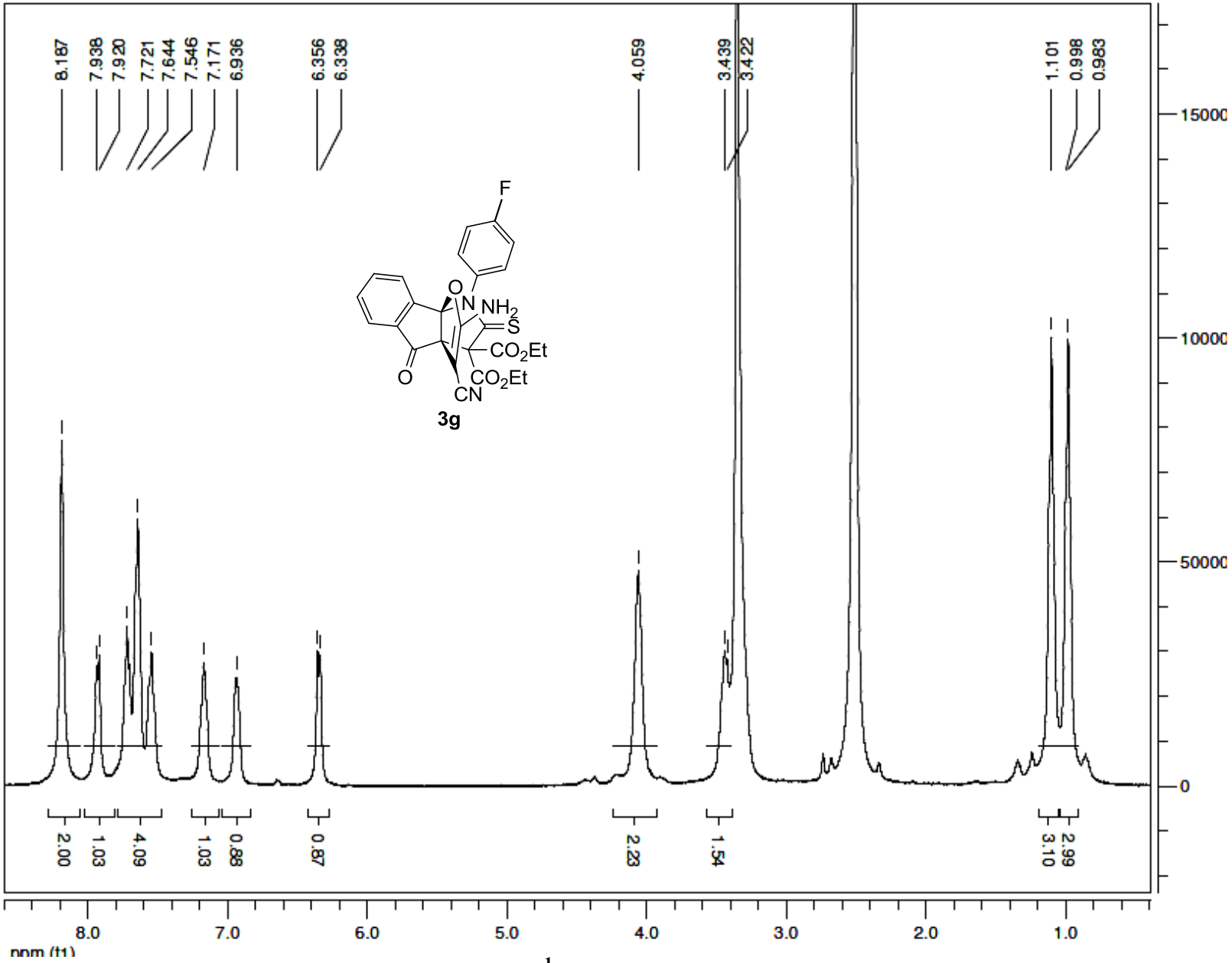
10000
50000
0
200
150
100
50
ppm (t1)

^{13}C NMR of 3f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CO2Et
CO2Et
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CN
3g
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4.09
1.03
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15000
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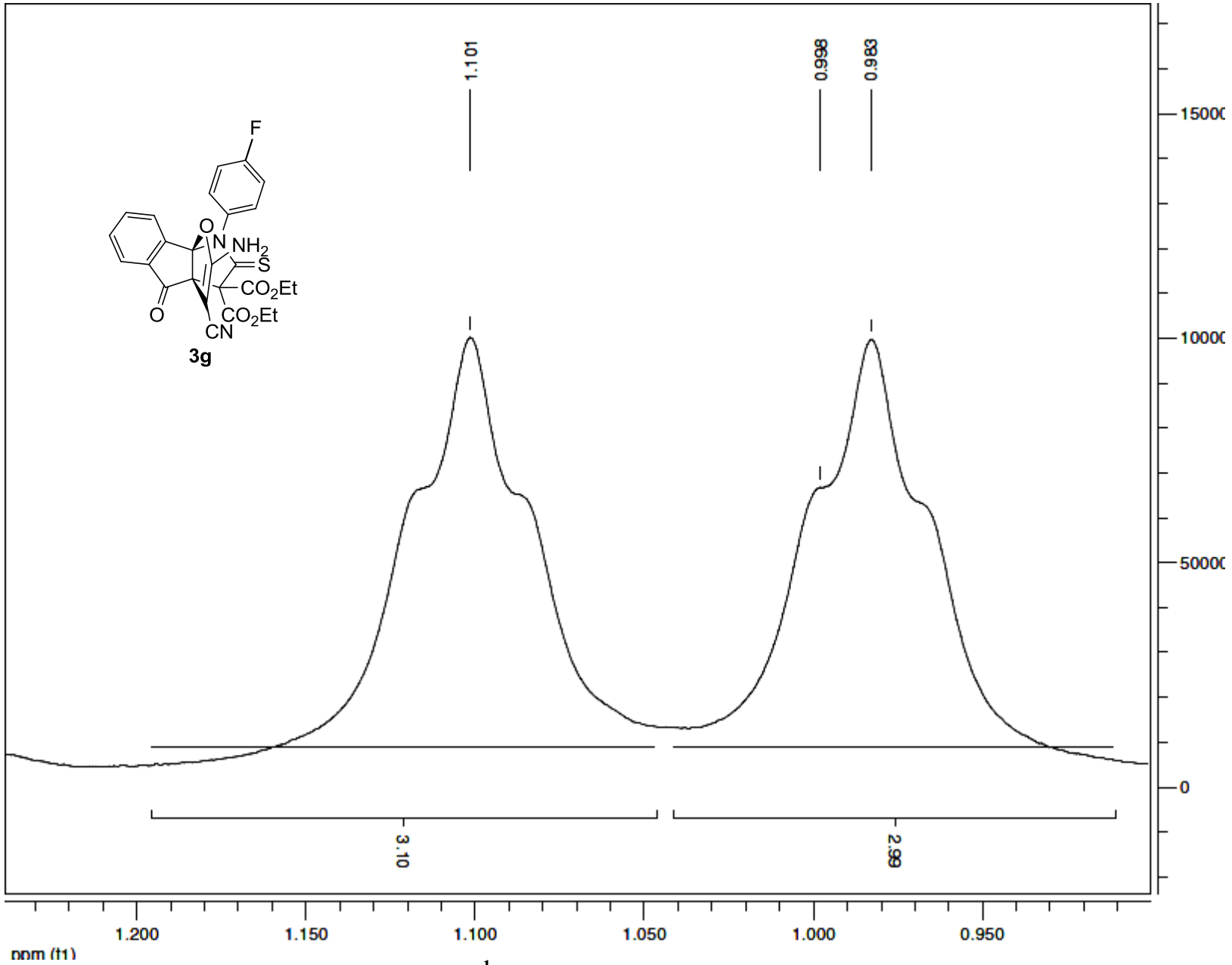
1.0
ppm (t1)

^{1}H NMR of 3g



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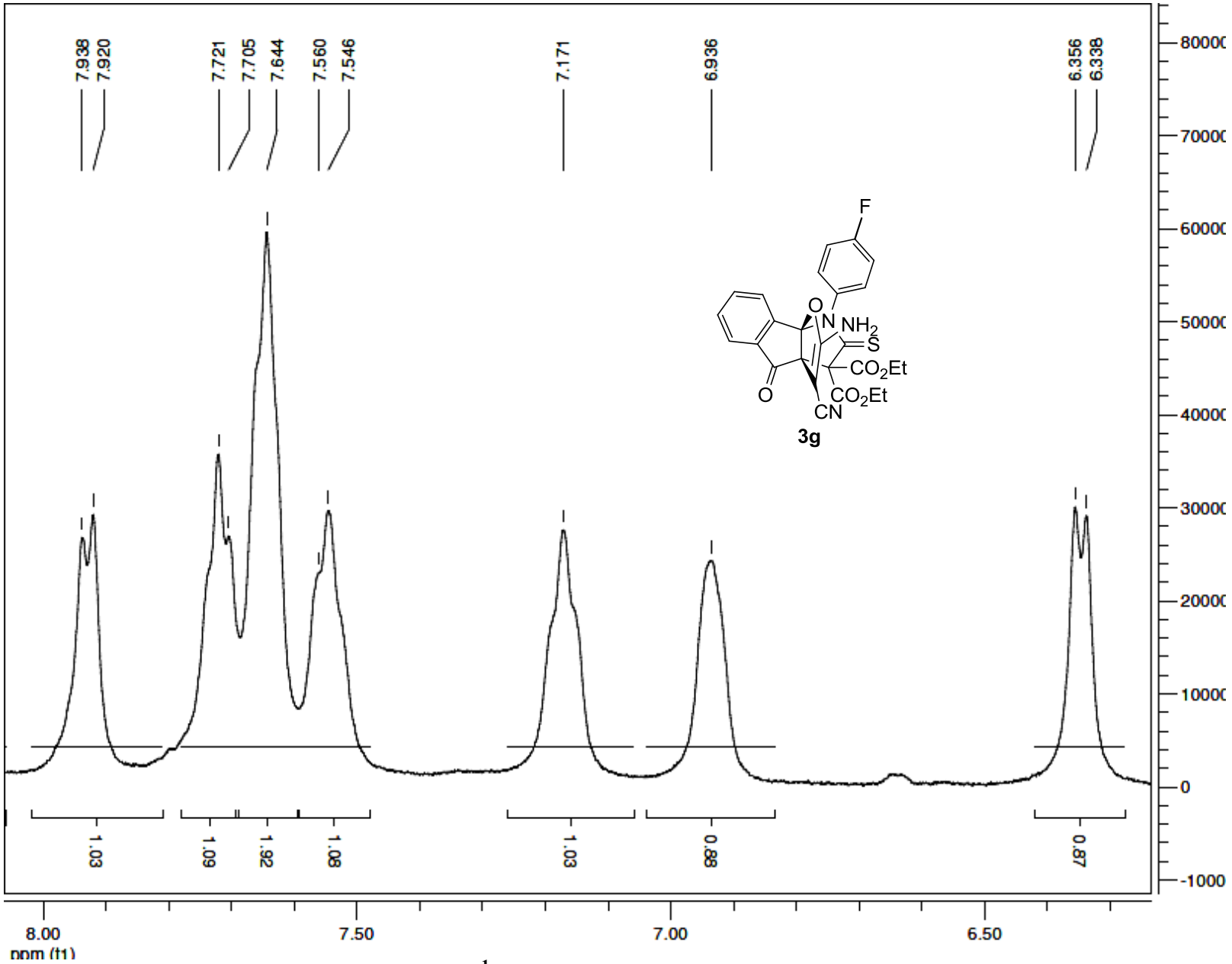
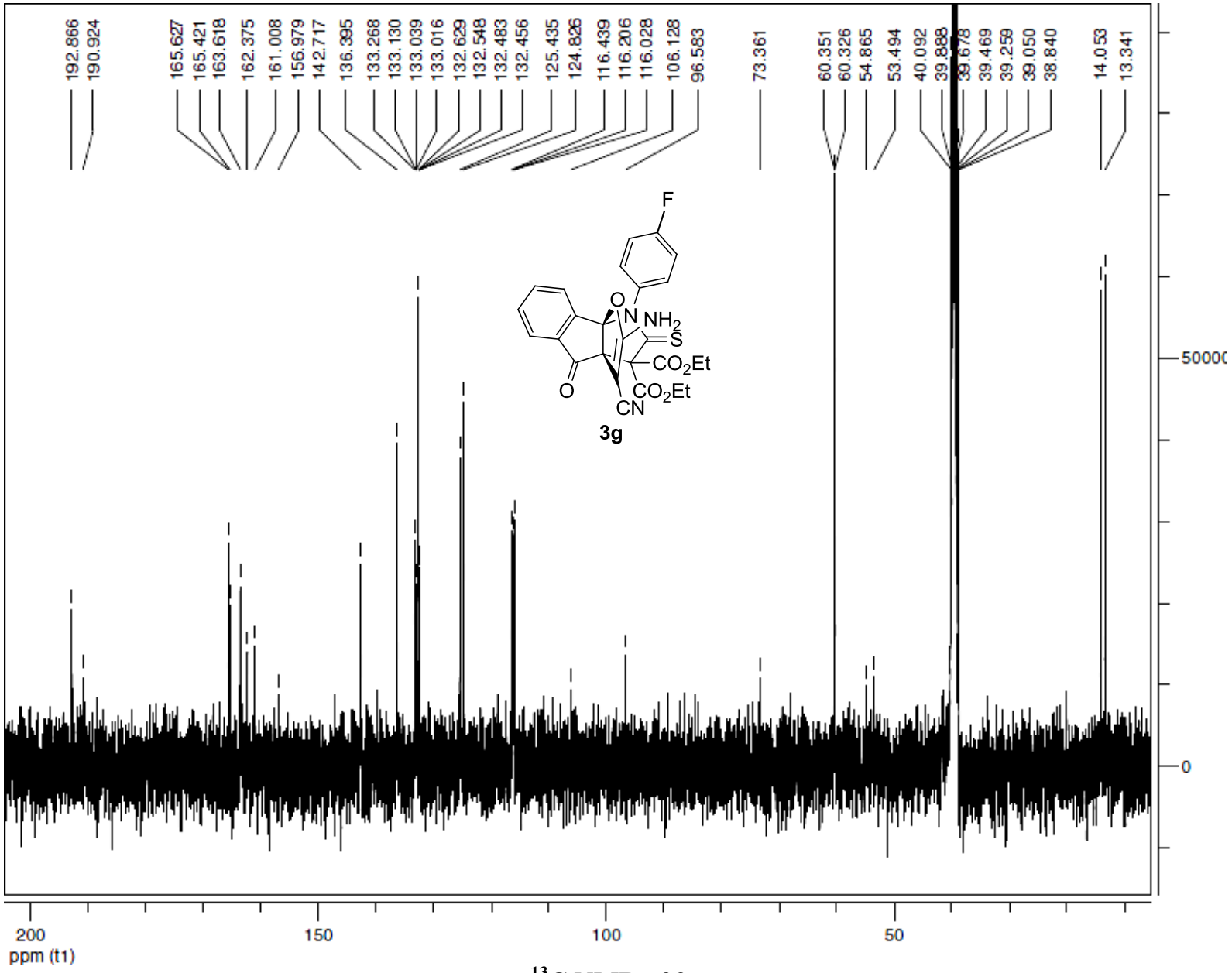


^{1}H NMR of 3g (expand)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165.421
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161.008
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142.717
136.395
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133.130
133.039
133.016
132.629
132.548
132.483
132.456
125.435
124.826
116.439
116.206
116.028
106.128
96.583
73.361
60.351
60.326
54.865
53.494
40.092
39.888
39.678
39.469
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39.050
38.840
14.053
13.341
F
O
N
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3g
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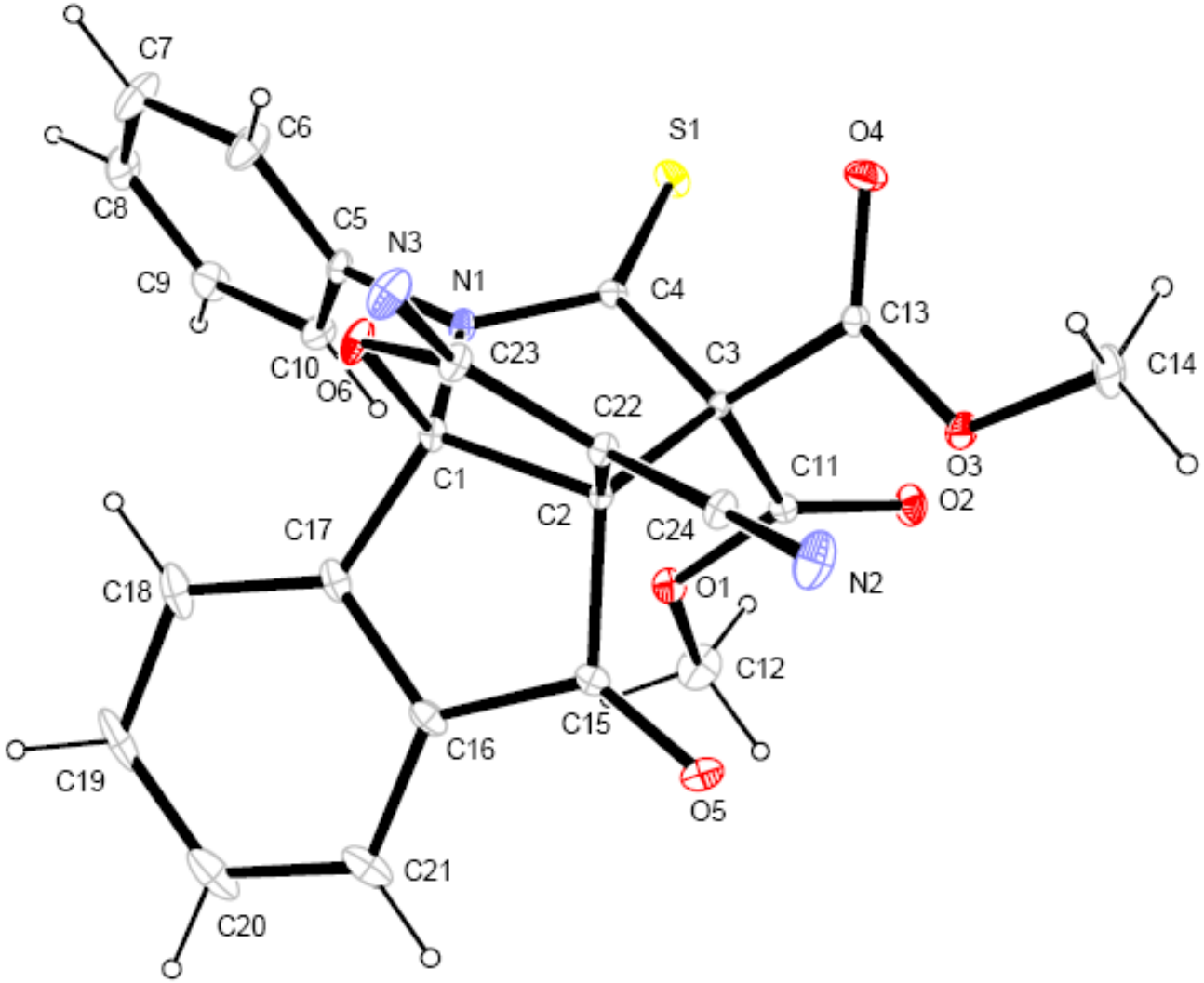
200
150
100
50
ppm (t1)

^{13}C NMR of 3g



ORTEP diagram for 3a

Crystal data for **3a** $C_{24}H_{17}N_3O_6S'$ (CCDC 941274): M_W = 473.45, monoclinic, space group P21/c, a = 12.7640(12) Å, b = 10.2238(6) Å, c = 18.2787(14) Å, α = 90.00 β = 108.332(9), γ = 90.00, V = 2264.3(3) $Å^3$, Z = 4, Dc = 1.389 mg/m^3, F (000) = 976, crystal dimension 0.48×0.38×0.35 mm, radiation, Mo Kα (λ = 0.71073Å), 3.13≤2θ≤25.10, intensity data were collected at 295(2) K with a Bruker APEX-II CCD area-detector diffractometer, and employing $\omega/2\theta$ scanning technique, in the range of -14≤h≤15, -11≤k≤12, -16≤l≤21; the structure was solved by a direct method, all non-hydrogen atoms were positioned and anisotropic thermal parameters refined from 4024 observed reflections with R (into) = 0.0783 by a full-matrix least-squares technique converged to R = 0.0500 and Raw = 0.1165 [I>2sigma(I)].