Supporting Information for

Cu-Catalyzed Oxidative 3-Amination of Indoles via Formation of Indolyl(aryl)iodonium Imides Using *o*-Substituted (Diacetoxyiodo)arene as a High-Performance Hypervalent Iodine Compound

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1. X-ray Diffraction Analysis of 2a.

Crystal data for 2a: Formula $C_{27}H_{28}N_2O_5S_2$, colorless, crystal dimensions 0.30 × 0.20 × 0.20 mm^3, Triclinic, space group P -1, a = 9.475(2) Å, b = 10.144(2) Å, c = 13.660(3) Å, α = 98.118(3) °, β = 91.348(3) °, γ = 100.871(3) °, V = 1274.7(5) $Å^3$, Z = 2, ρ_{calc} = 1.367 g cm^{-3}, F(000) = 552, μ(MoKα) = 0.250 mm^{-1}, T = 173 K. 7413 reflections collected, 5602 independent reflections with $I > 2\sigma(I)$ ($2\theta_{max}$ = 27.572 °), and 330 parameters were used for the solution of the structure. The non-hydrogen atoms were refined anisotropically. R_1 = 0.0508 and wR_2 = 0.1031. GOF = 1.015. Crystallographic data (excluding structure factors) for the structure reported in this paper have been deposited with the Cambridge Crystallographic Data Centre as supplementary publication no. CCDC-1860598. Copies of the data can be obtained free of charge on application to CCDC, 12 Union Road, Cambridge CB2 1EZ, UK [Fax: int. code + 44(1223)336-033; E-mail: deposit@ccdc.cam.ac.uk].

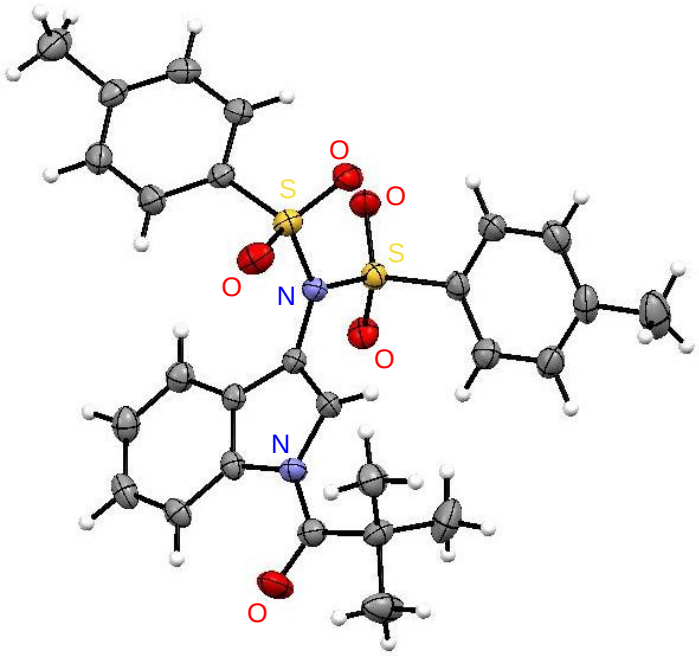
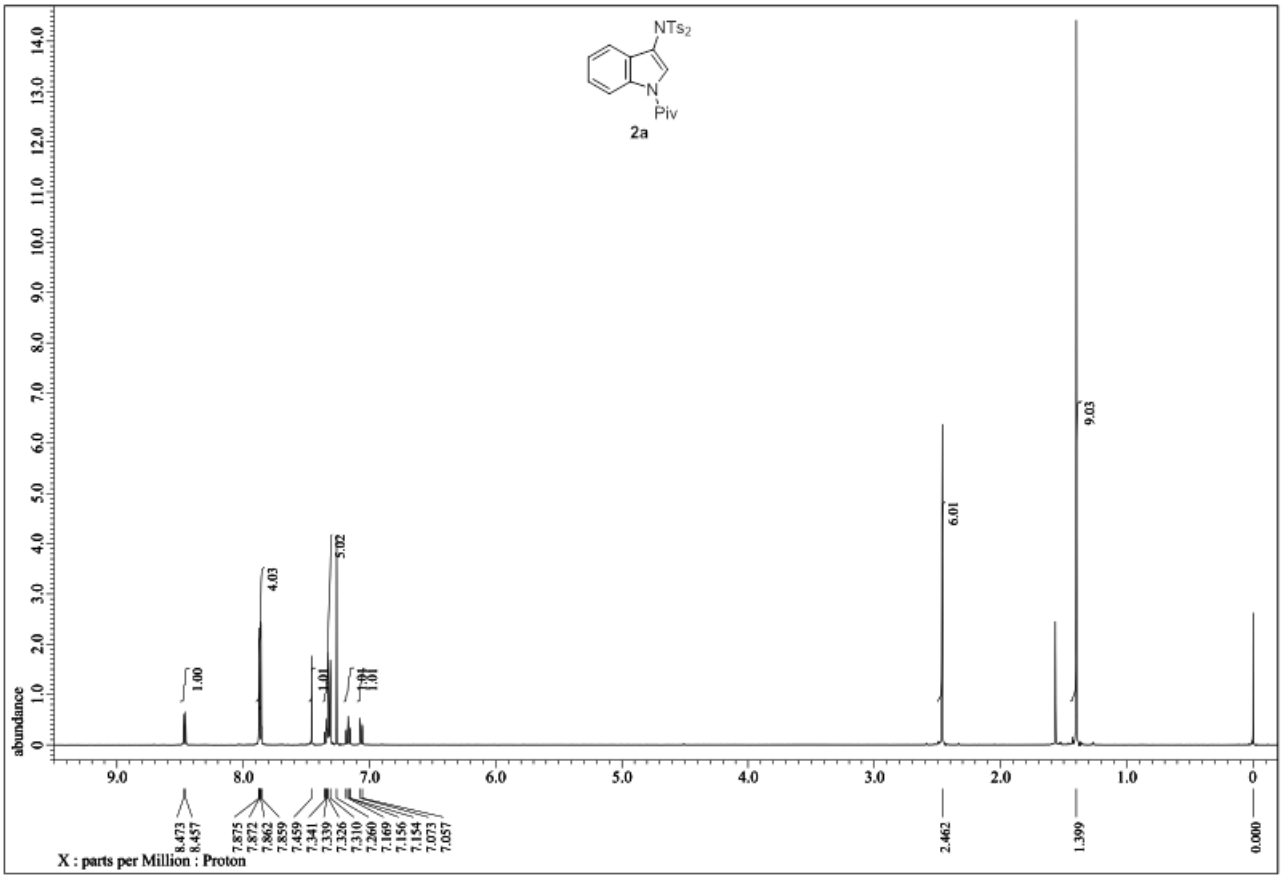


Figure S1. ORTEP drawing of **2a**. The ellipsoids correspond to 50% probability.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Piv
2a
abundance
X : parts per Million : Proton
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7.862
7.859
7.459
7.341
7.339
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7.154
7.073
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2.462
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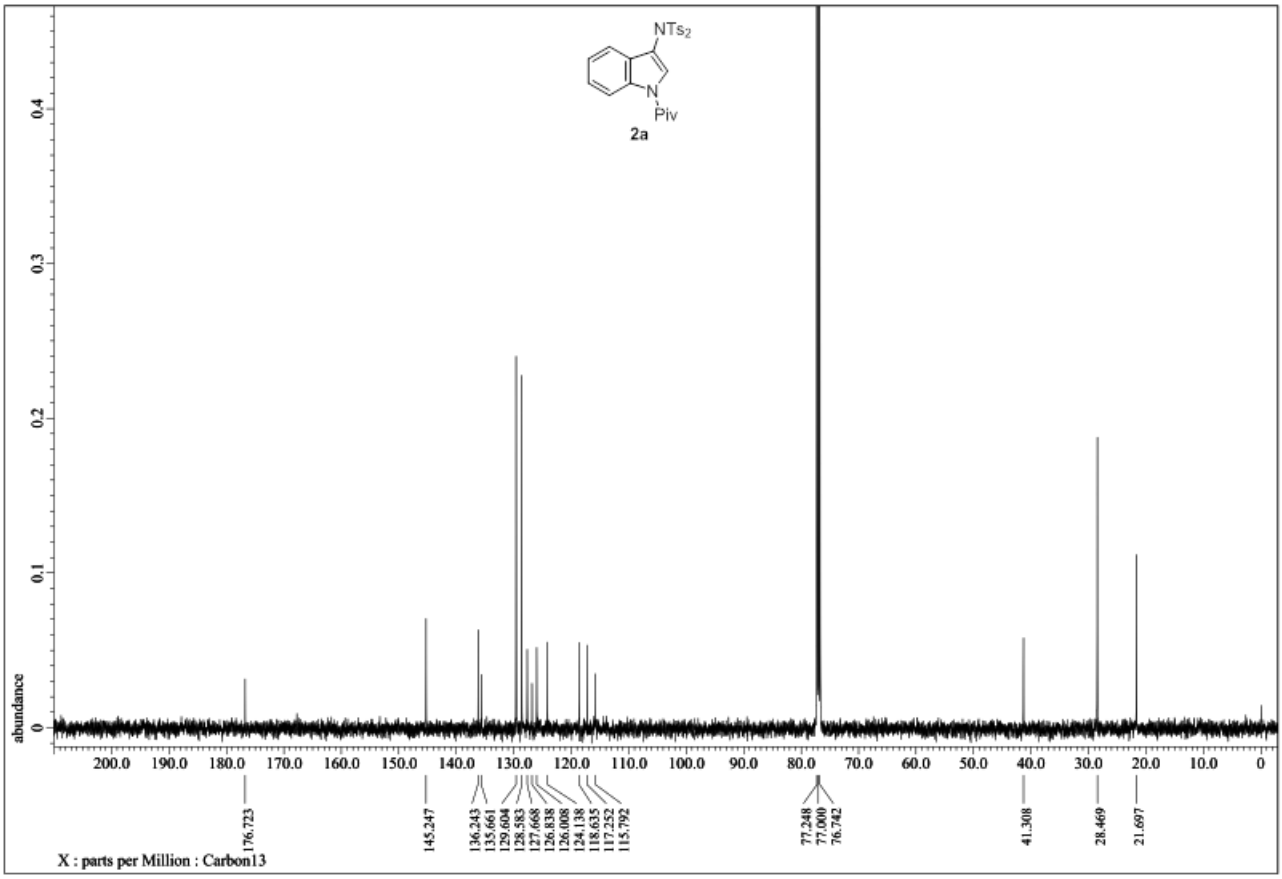
0.000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N
Piv
2a
abundance
X : parts per Million : Carbon13
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136.243
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127.668
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115.792
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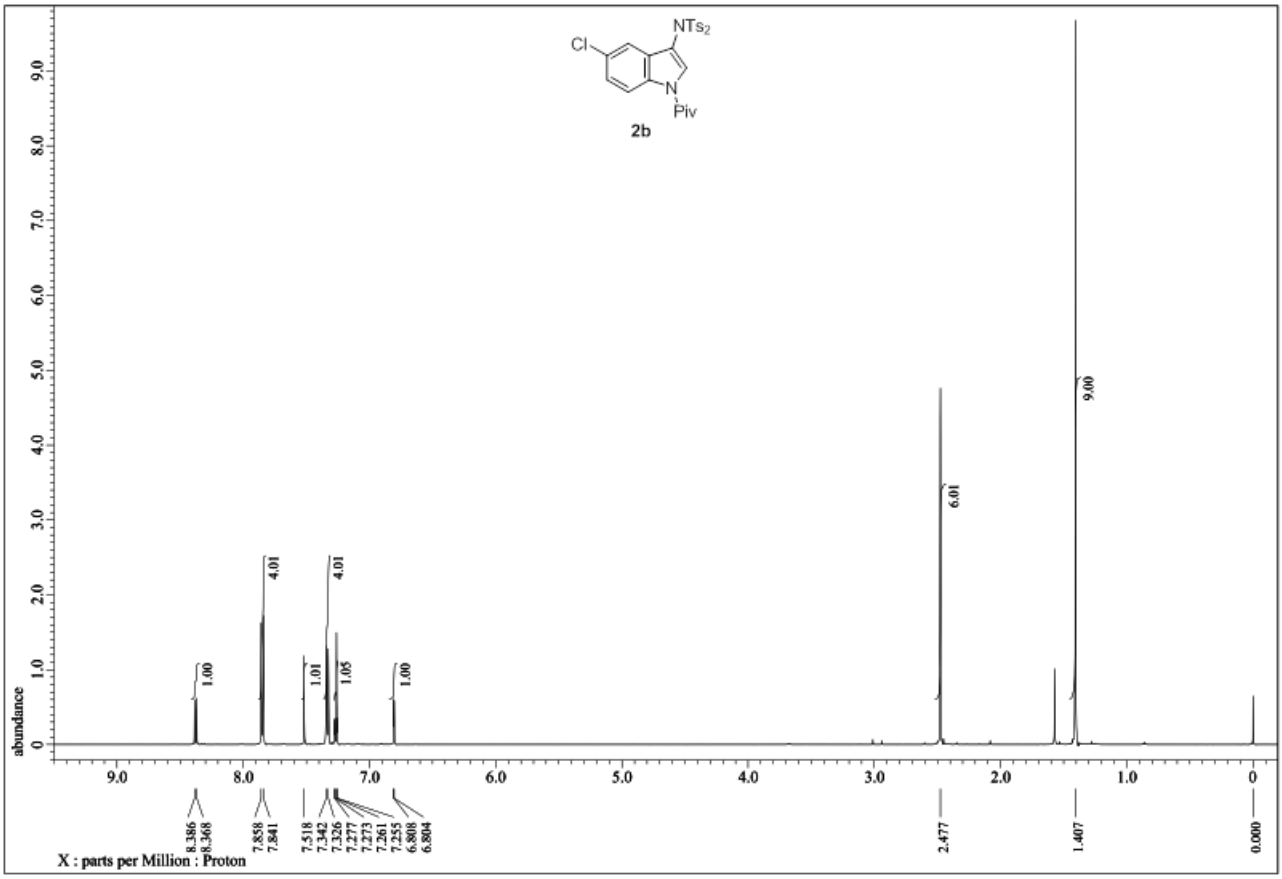
77.000
76.742
41.308
28.469
21.697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Piv
2b
abundance
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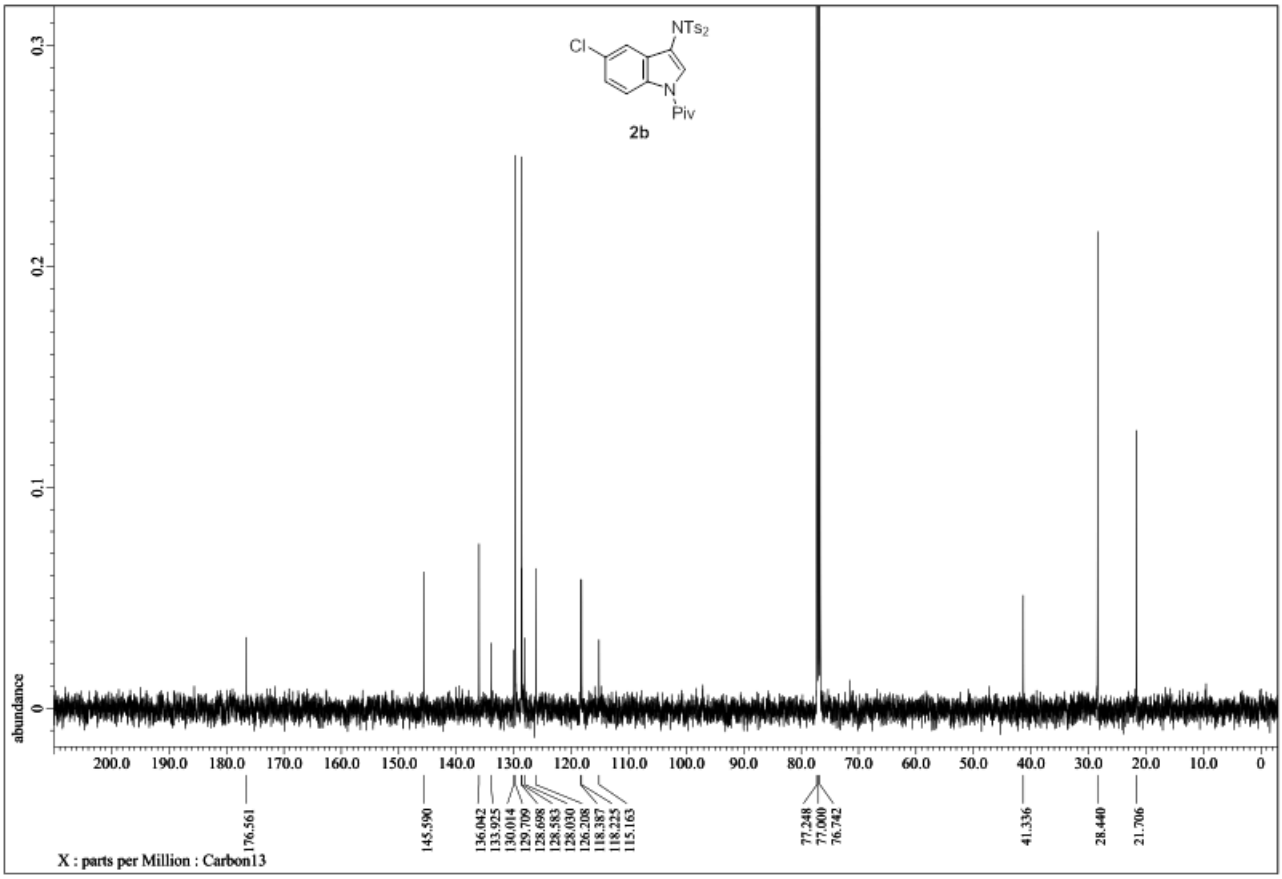
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N
Piv
2b
abundance
X : parts per Million : Carbon13
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145.590
136.042
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129.709
128.698
128.583
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126.208
118.387
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115.163
77.248
77.000
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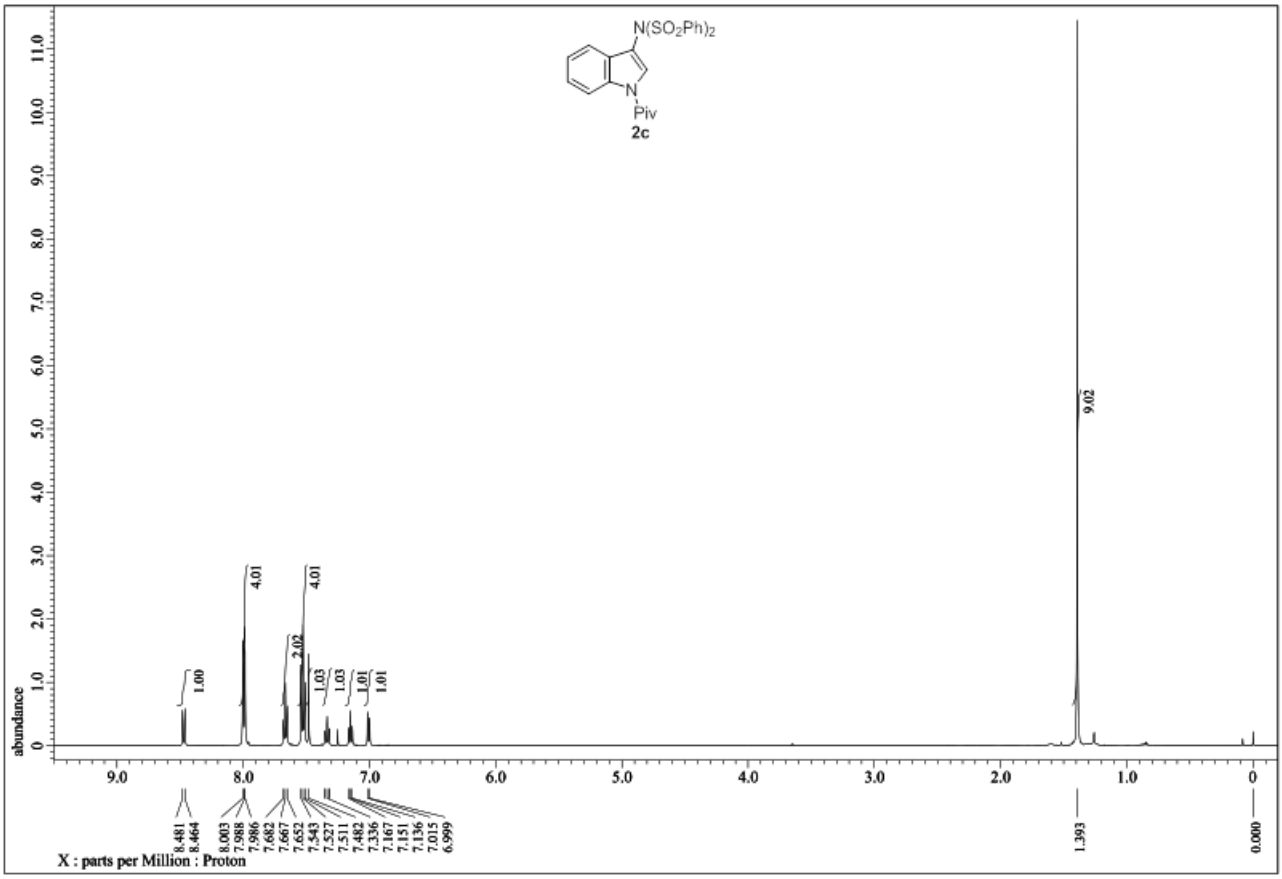
41.336
28.440
21.706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N
Piv
2c
abundance
X : parts per Million : Proton
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4.01
2.02
4.01
1.03
1.03
1.01
1.01
9.02
8.481
8.464
8.003
7.988
7.986
7.682
7.667
7.652
7.543
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7.511
7.482
7.336
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7.151
7.136
7.015
6.999
1.393
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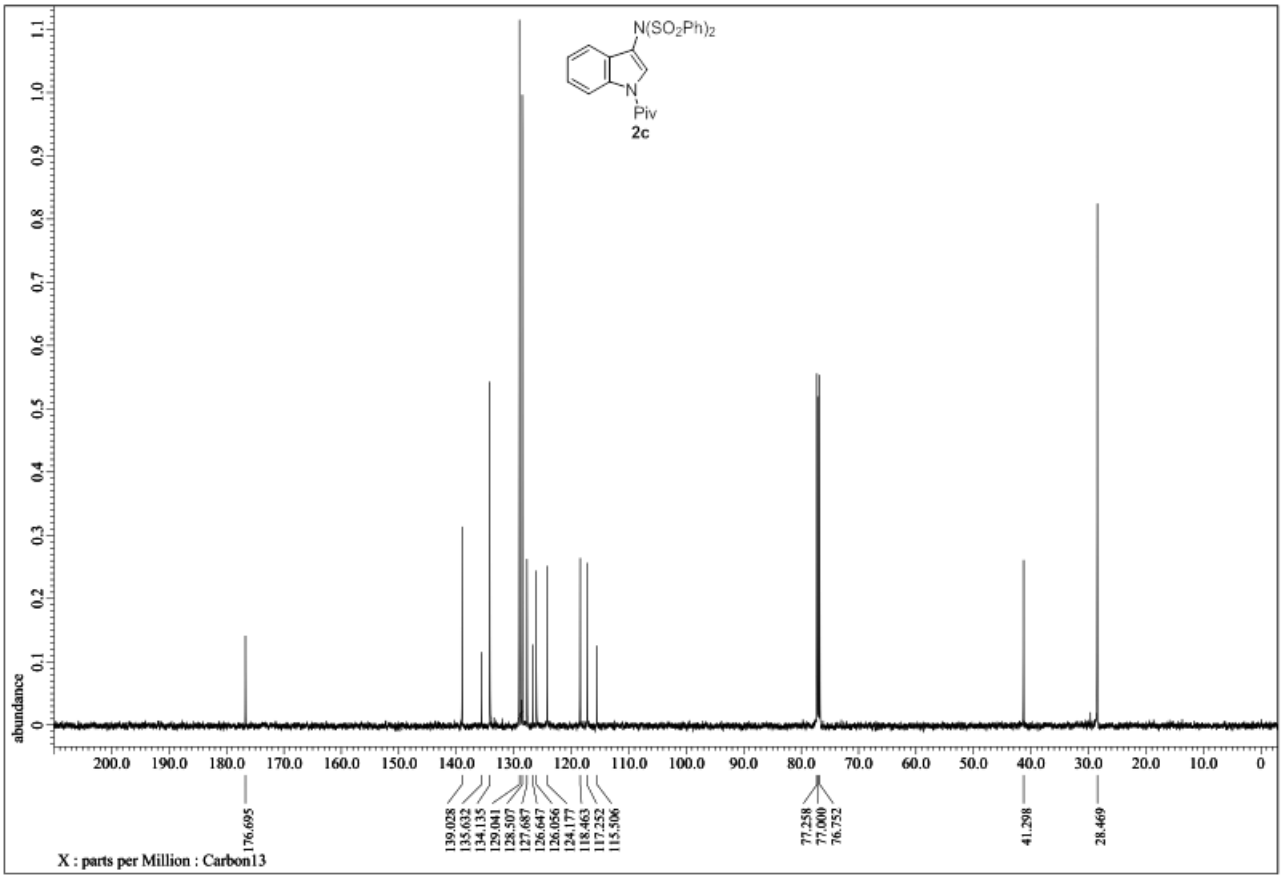
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N
Piv
2c
abundance
X : parts per Million : Carbon13
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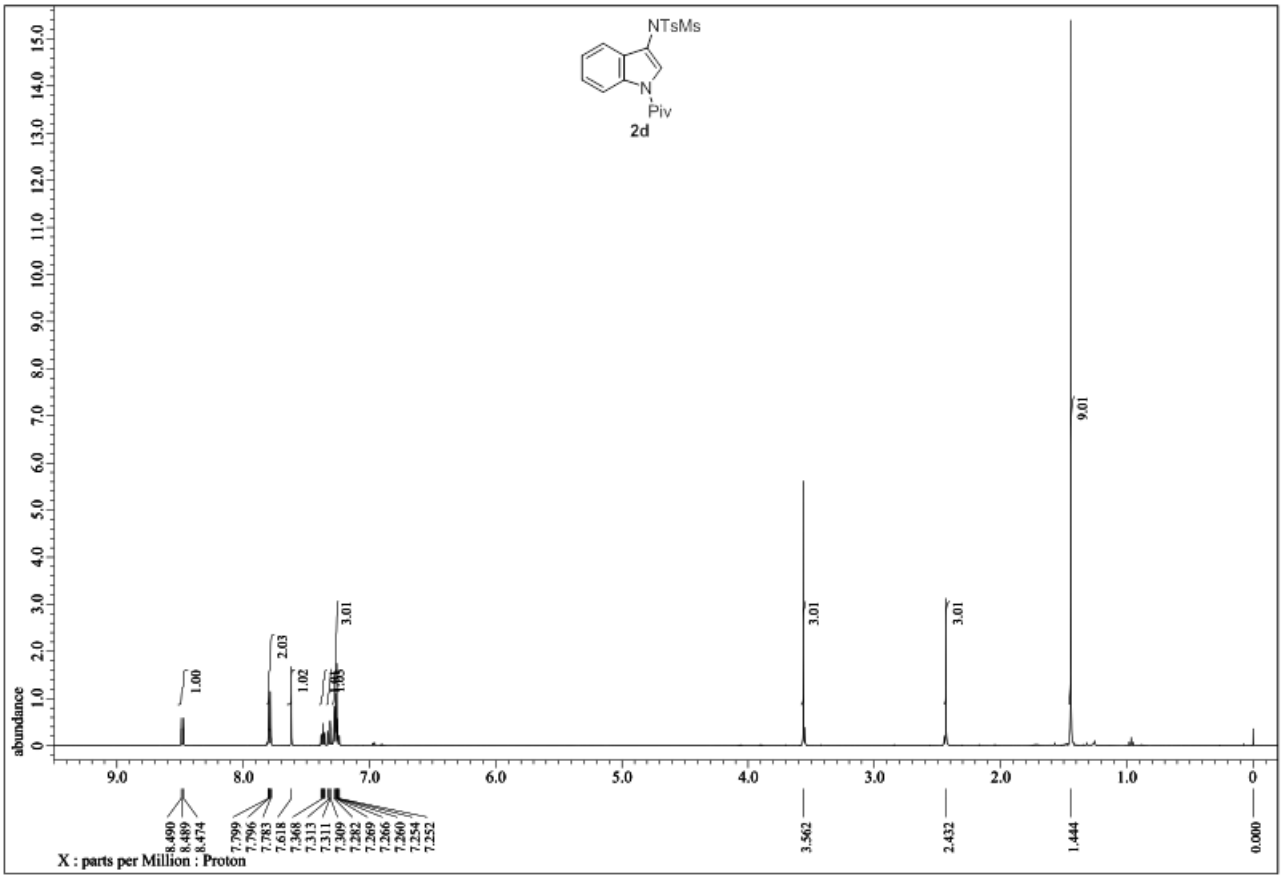
124.177
118.463
117.252
115.506
77.258
77.000
76.752
41.298
28.469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N
Piv
2d
abundance
X : parts per Million : Proton
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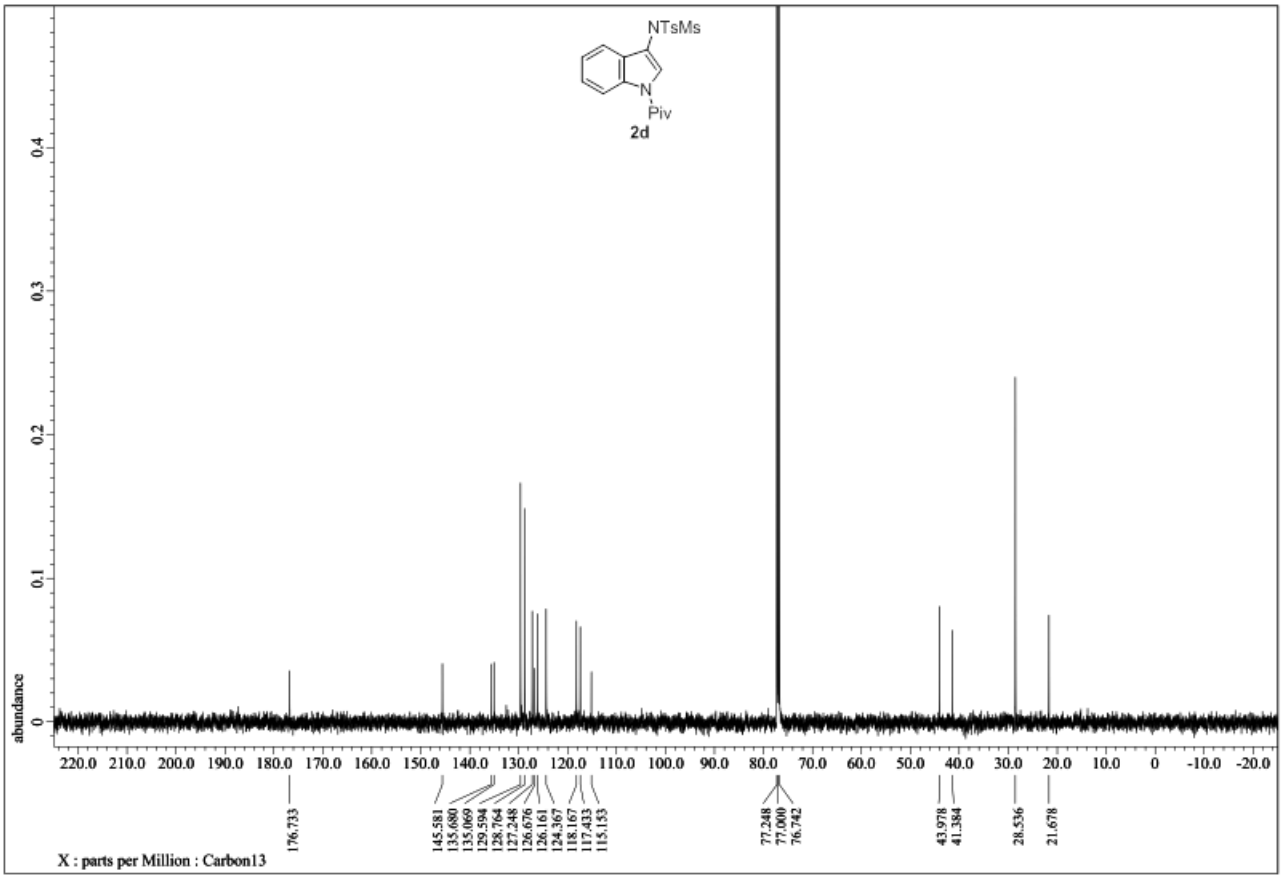
NTsMs
N
Piv
2d
abundance
X : parts per Million : Carbon13
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135.069
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128.764
127.248
126.676
126.161
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77.000
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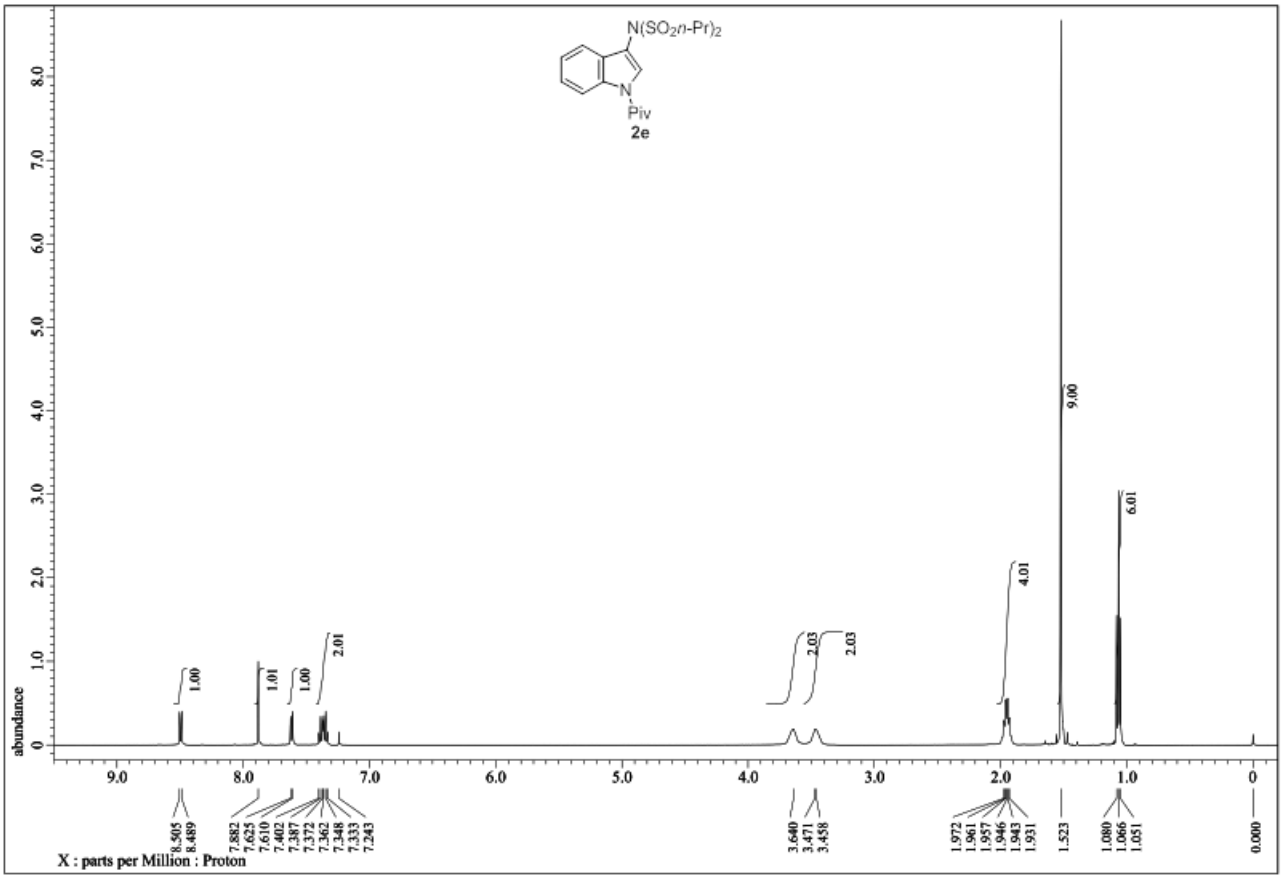
28.536
21.678


N(SO2n-Pr)2
N
Piv
2e
abundance
1.00
1.01
1.00
2.01
2.03
2.03
4.01
9.00
6.01
8.505
8.489
7.882
7.625
7.610
7.402
7.387
7.372
7.362
7.348
7.333
7.243
3.640
3.471
3.458
1.972
1.961
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1.943
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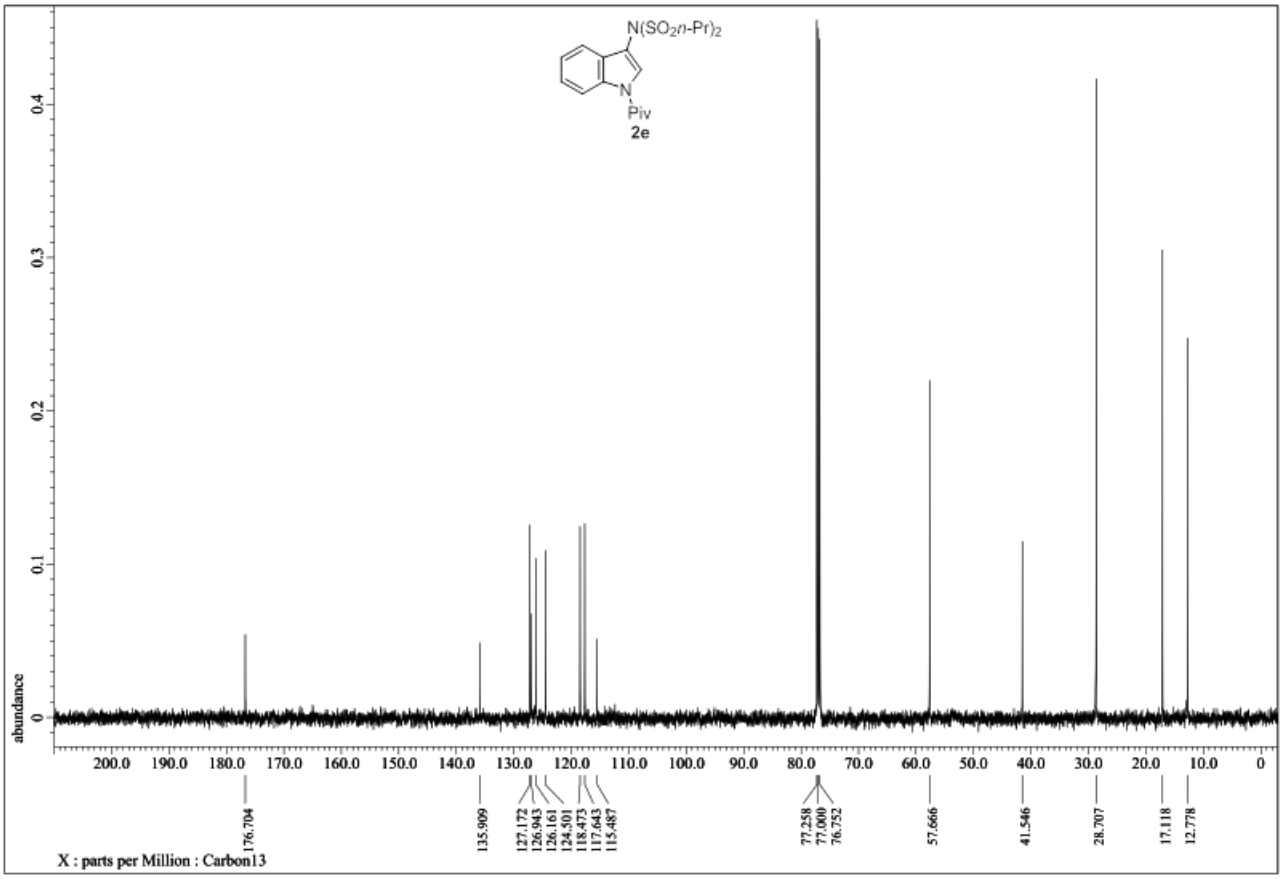
1.066
1.051
0.000
X : parts per Million : Proton


N(SO2n-Pr)2
N
Piv
2e
abundance
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126.161
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118.473
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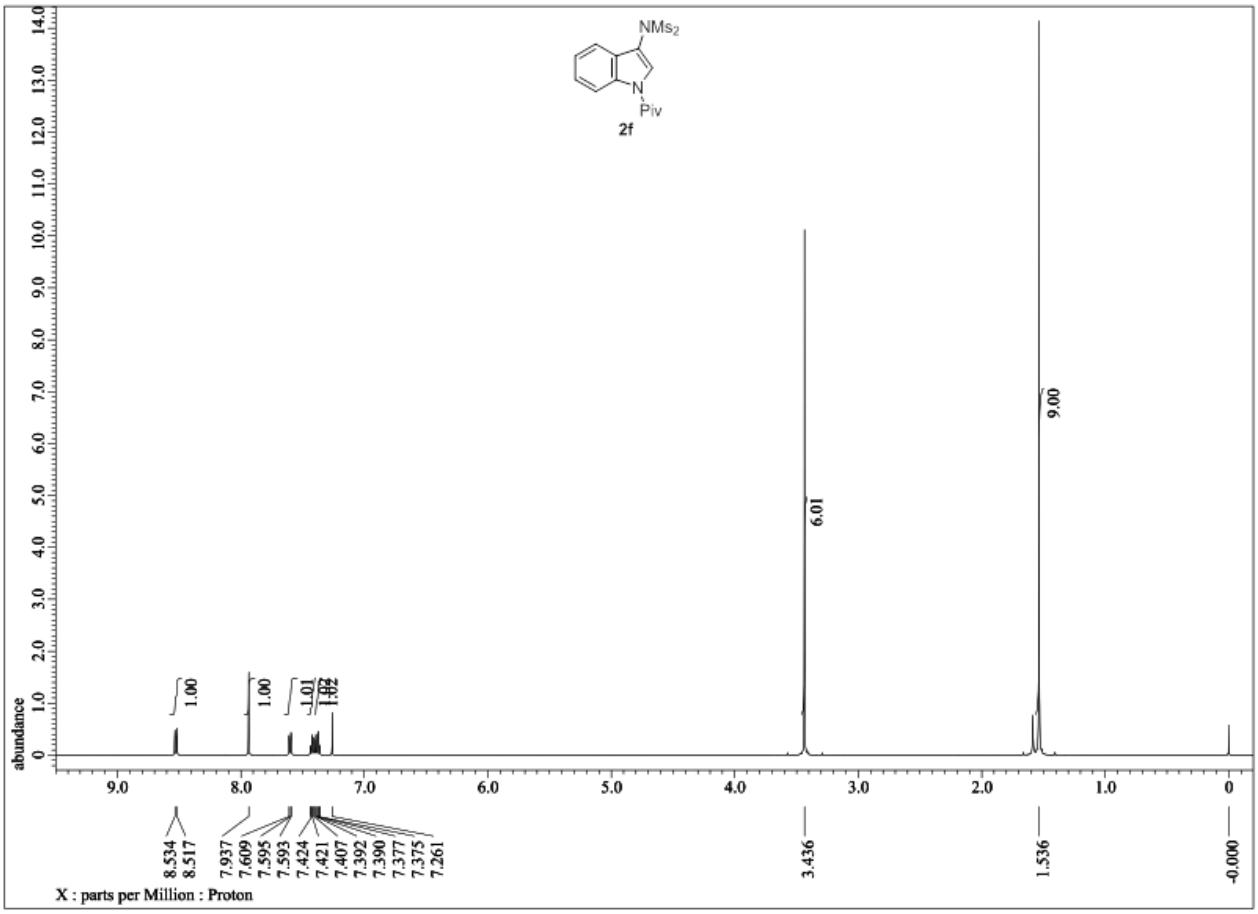
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N
Piv
2f
abundance
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1.00
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6.01
9.00
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7.609
7.595
7.593
7.424
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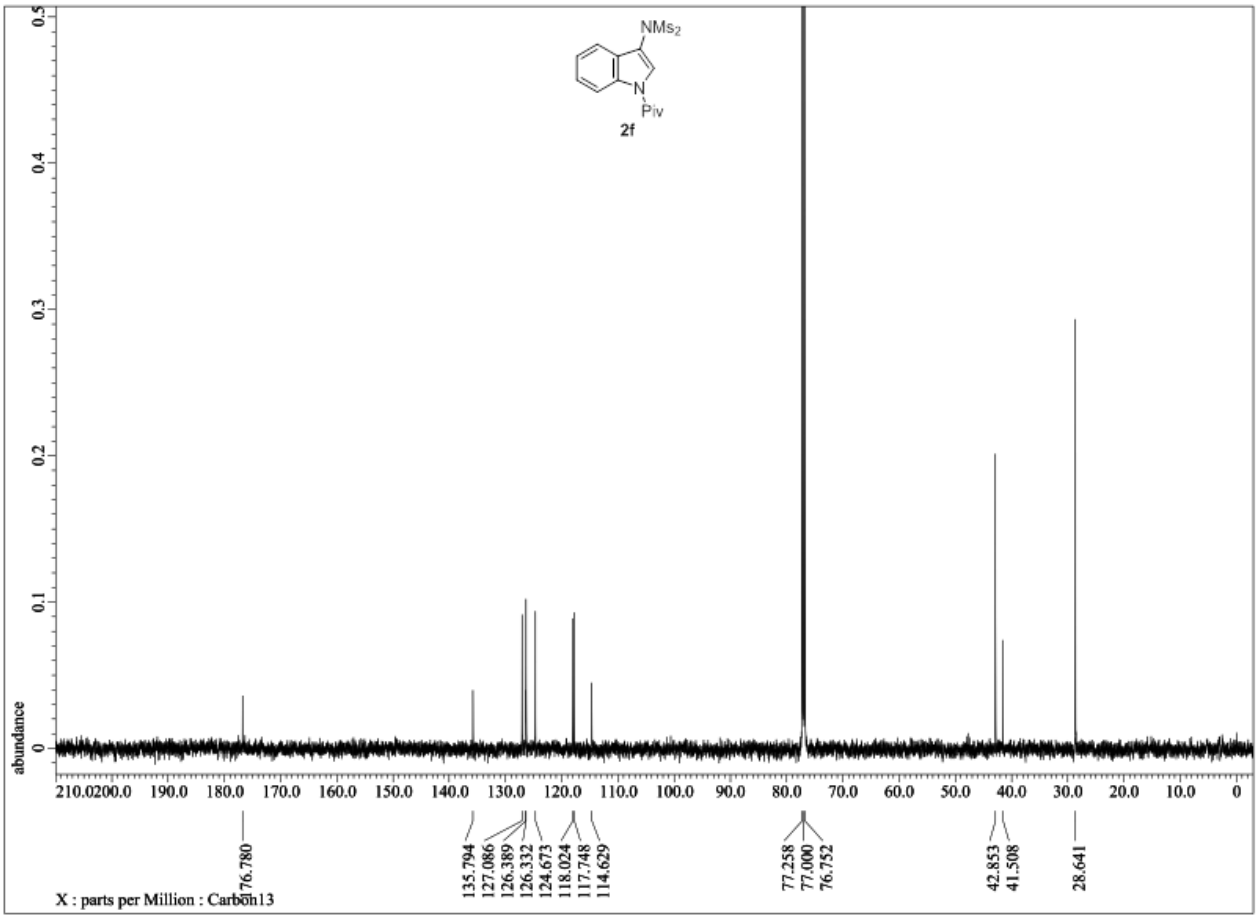
X : parts per Million : Proton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N
Piv
2f
abundance
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126.332
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77.000
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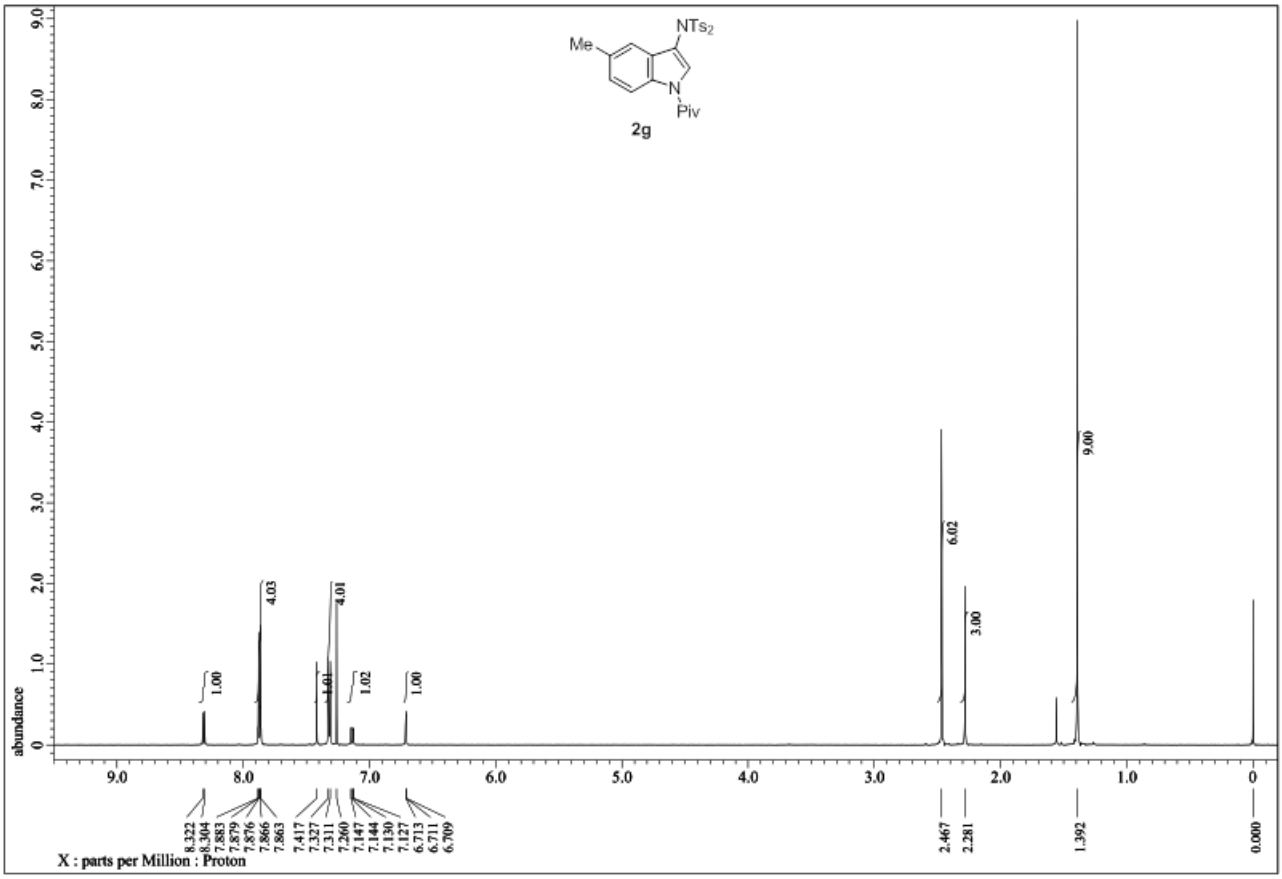
28.641
X : parts per Million : Carbon13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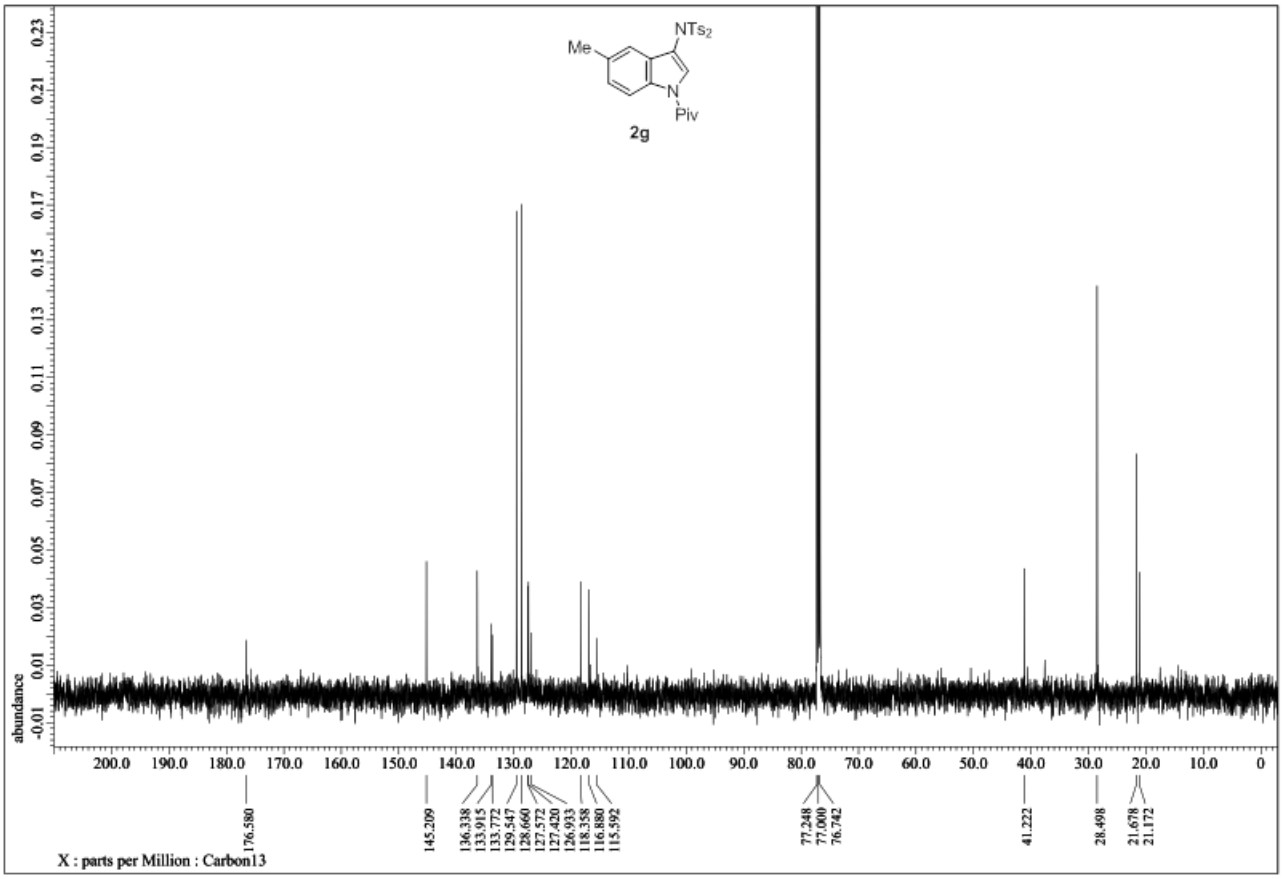
Me
N
Piv
2g
abundance
X : parts per Million : Proton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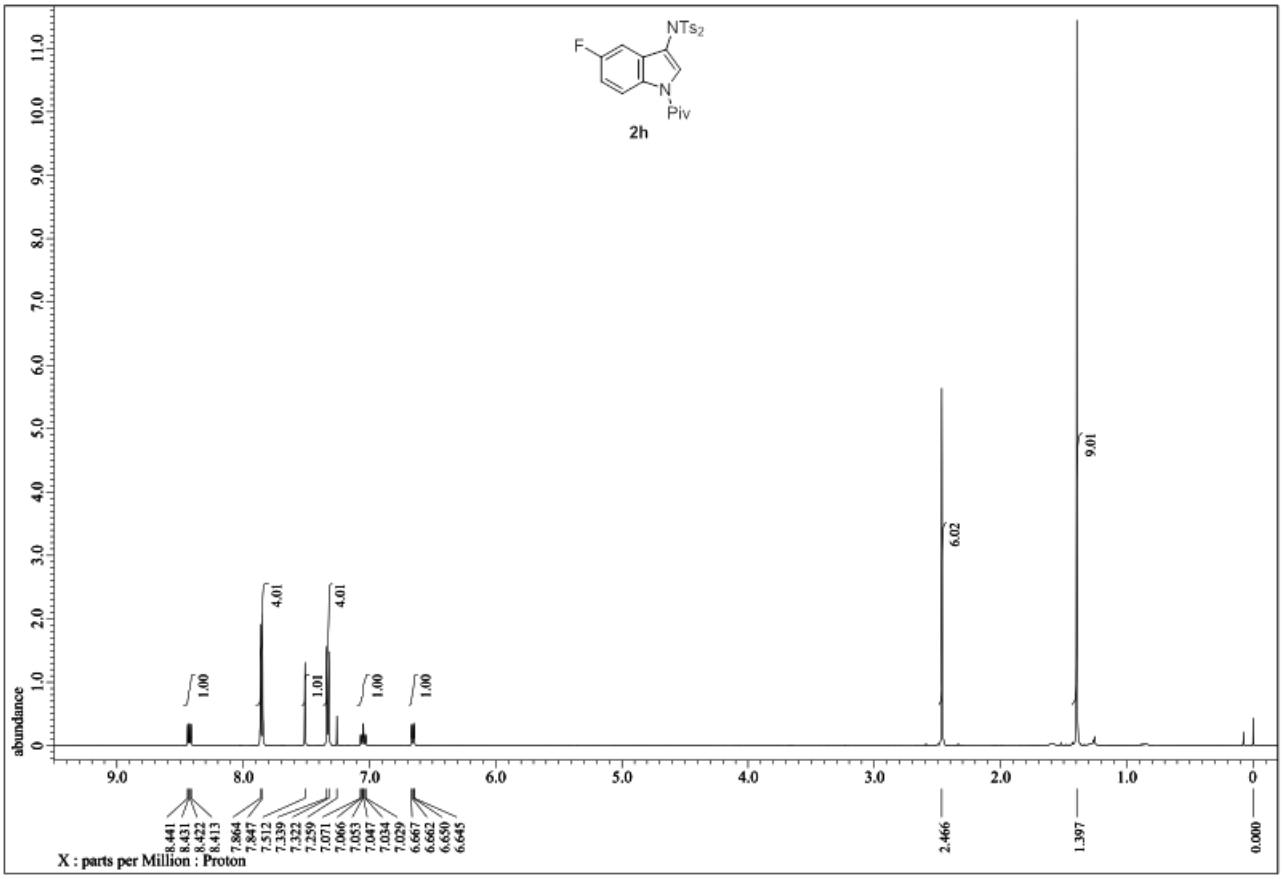
Me
N
Piv
2g
abundance
X : parts per Million : Carbon13


NTs2
F
N
Piv
2h
abundance
1.00
4.01
1.01
4.01
1.00
1.00
6.02
9.01
8.441
8.431
8.422
8.413
7.864
7.847
7.512
7.339
7.322
7.259
7.071
7.066
7.053
7.047
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7.029
6.667
6.662
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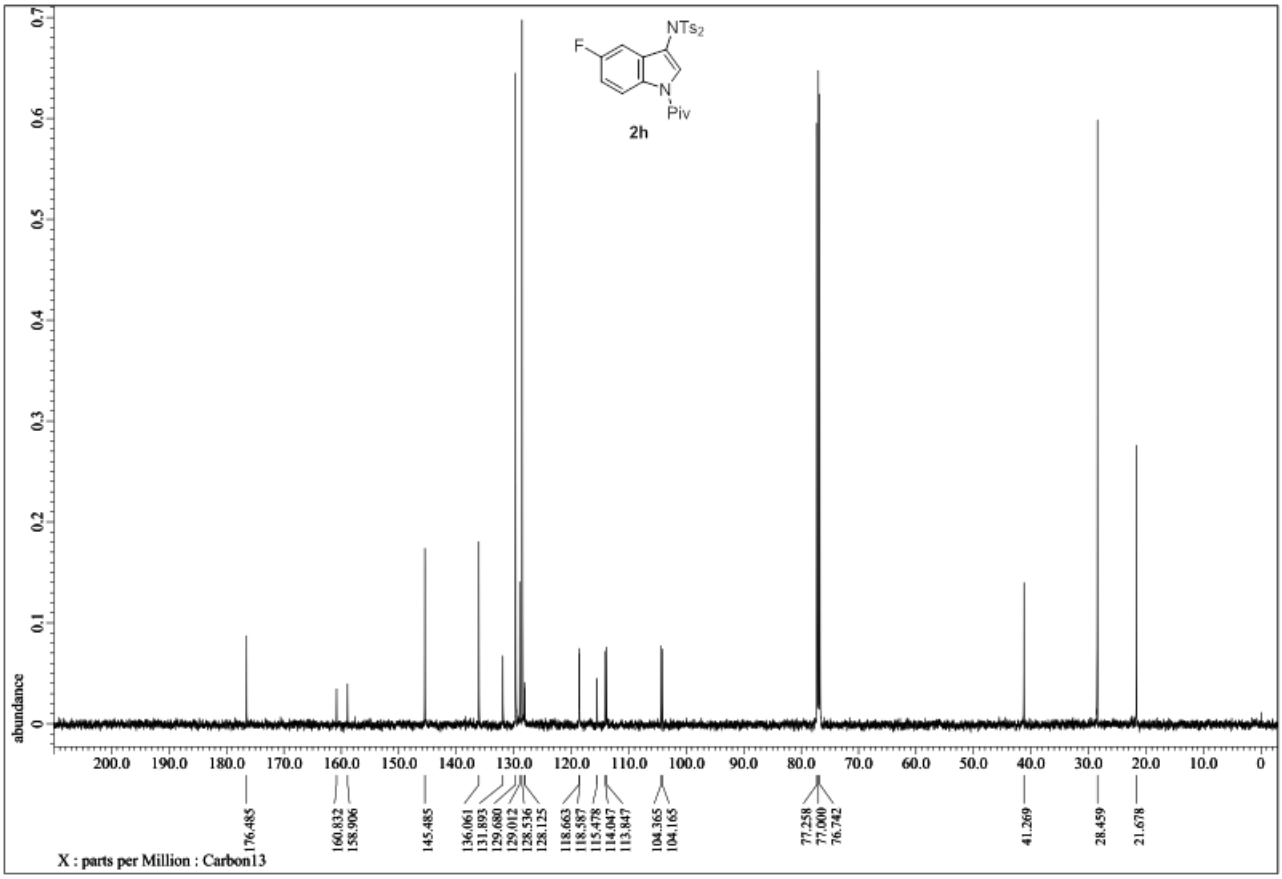
6.645
2.466
1.397
0.000
X : parts per Million : Proton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F
N
Piv
2h
abundance
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158.906
145.485
136.061
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129.680
129.012
128.536
128.125
118.663
118.587
115.478
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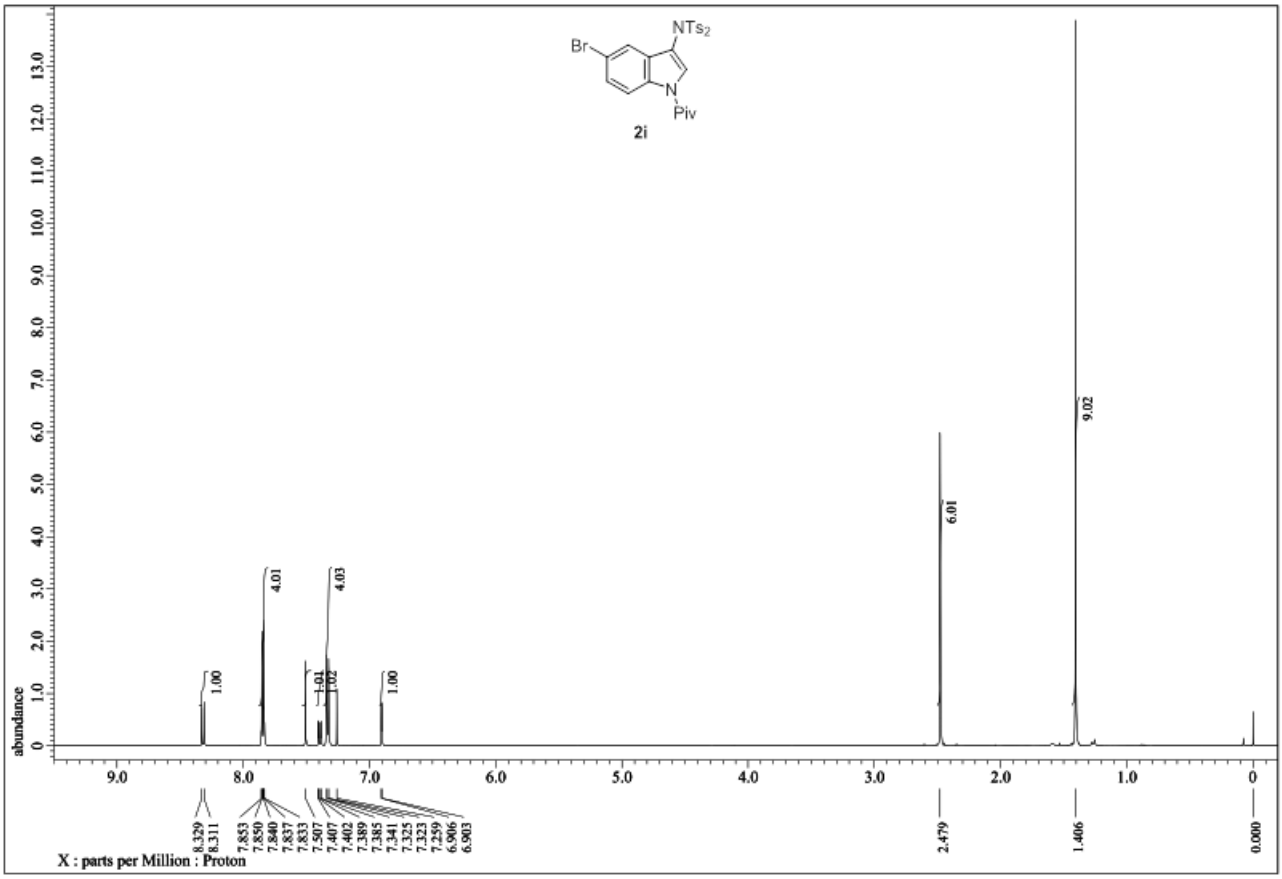
77.000
76.742
41.269
28.459
21.678
X : parts per Million : Carbon13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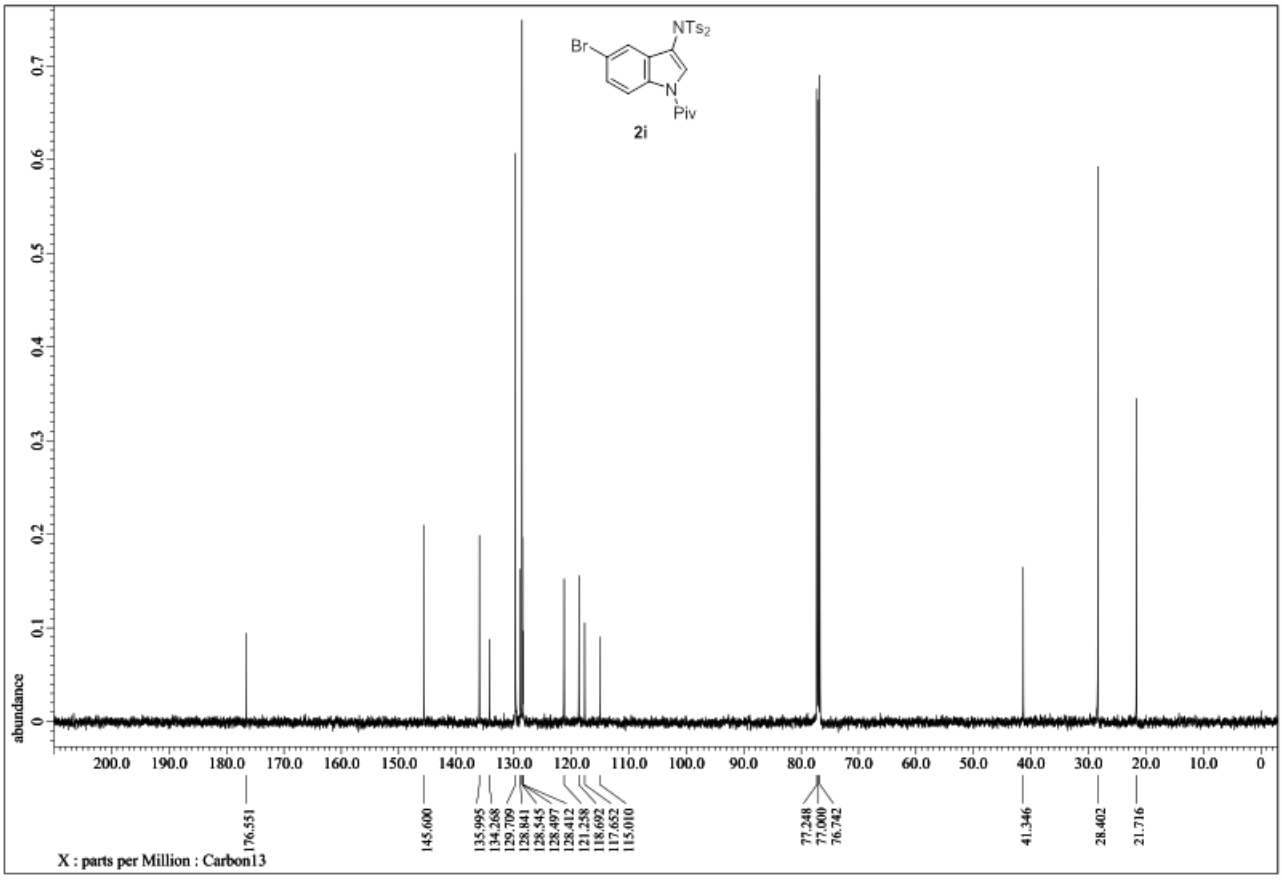
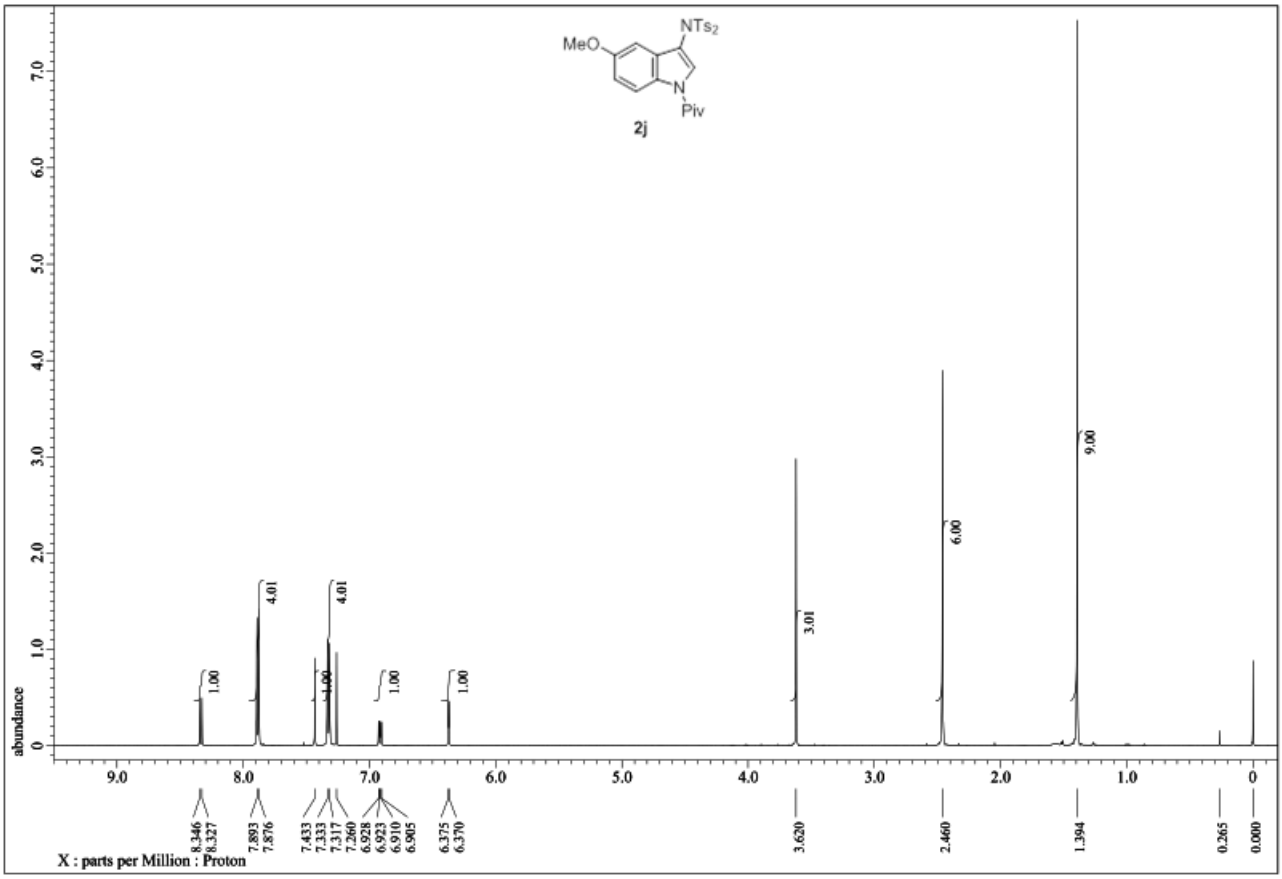
Br
N
Piv
2i
abundance
X : parts per Million : Proton


NTs2
Br
N
Piv
2i
abundance
X : parts per Million : Carbon13


MeO
NTs2
N
Piv
2j
abundance
X : parts per Million : Proton
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7.333
7.317
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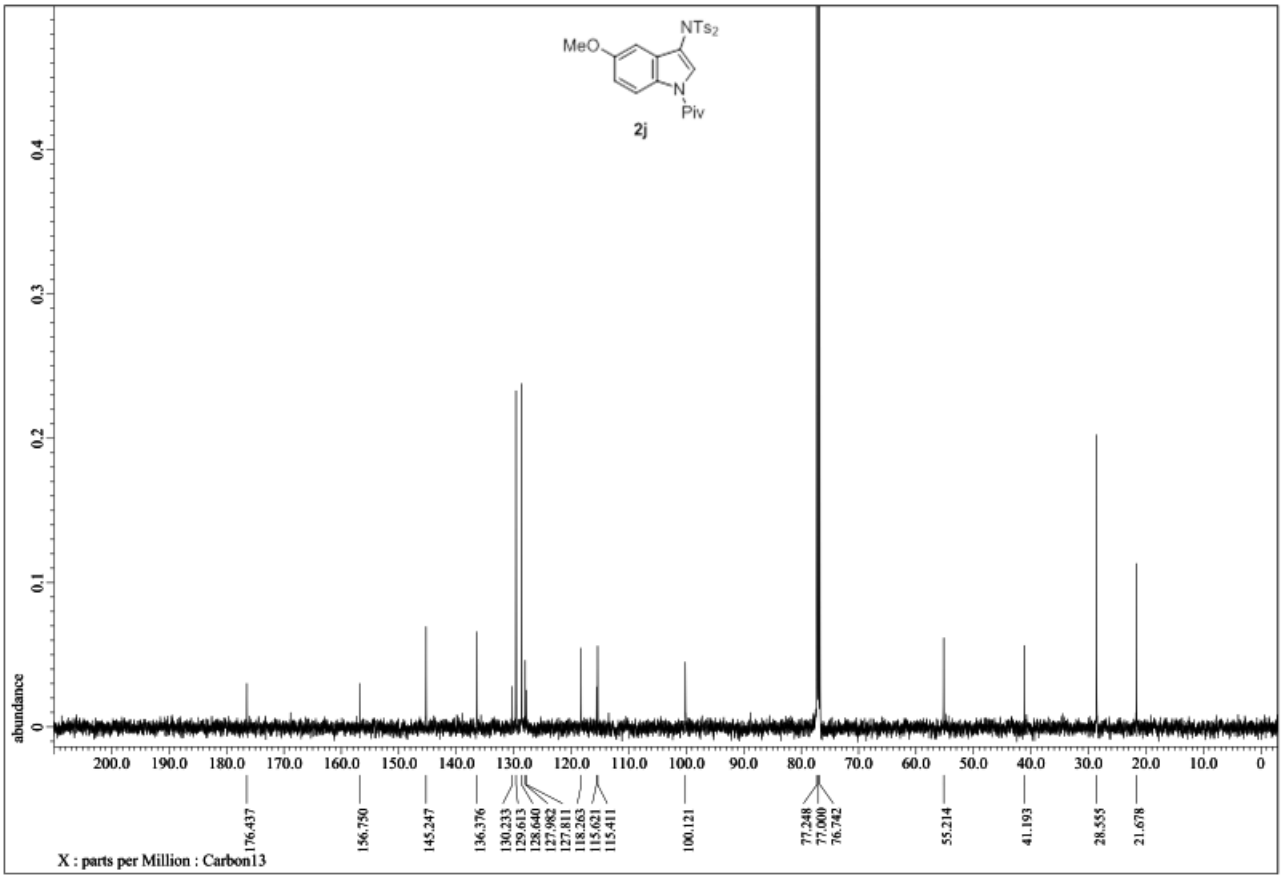
3.620
2.460
1.394
0.265
0.000


MeO
NTs2
N
Piv
2j
abundance
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128.640
127.982
127.811
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115.411
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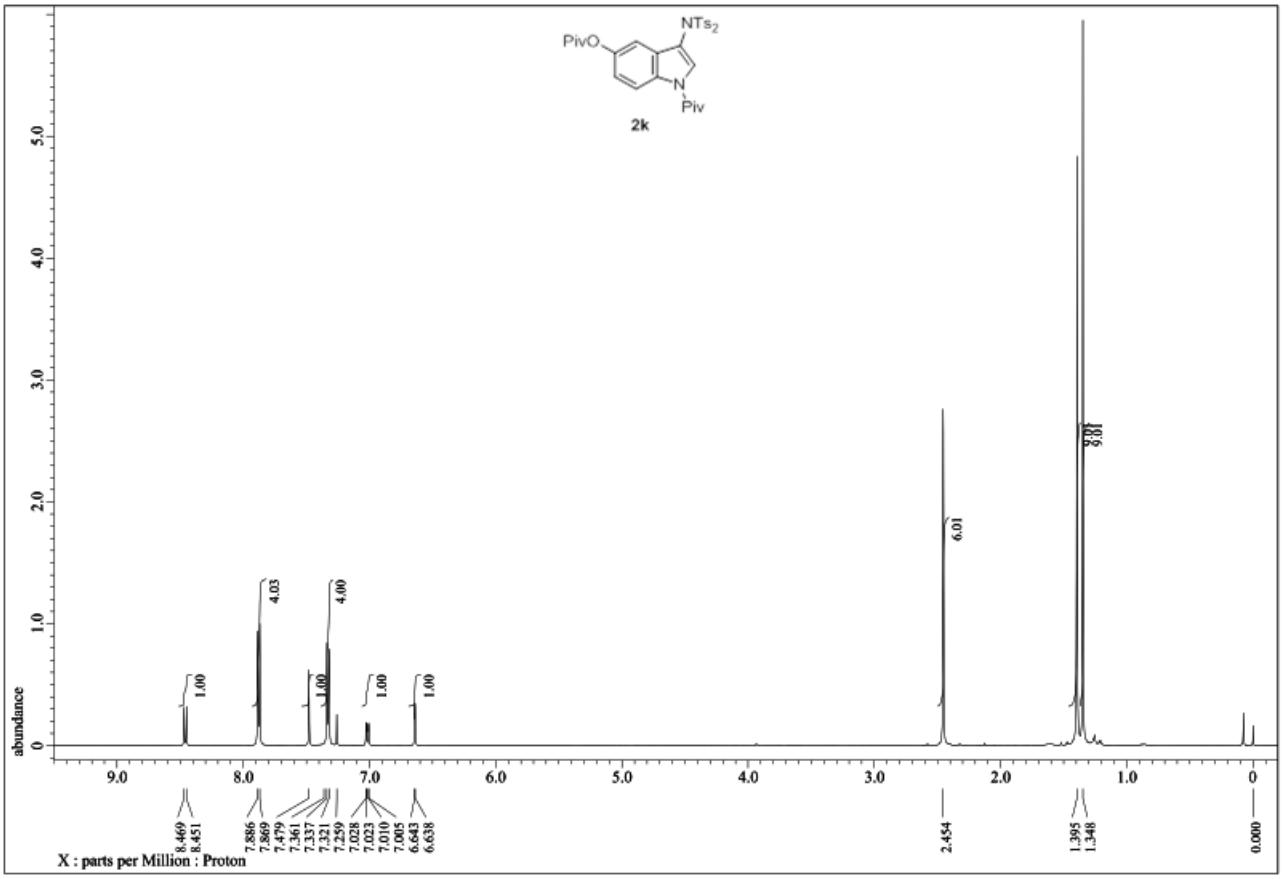
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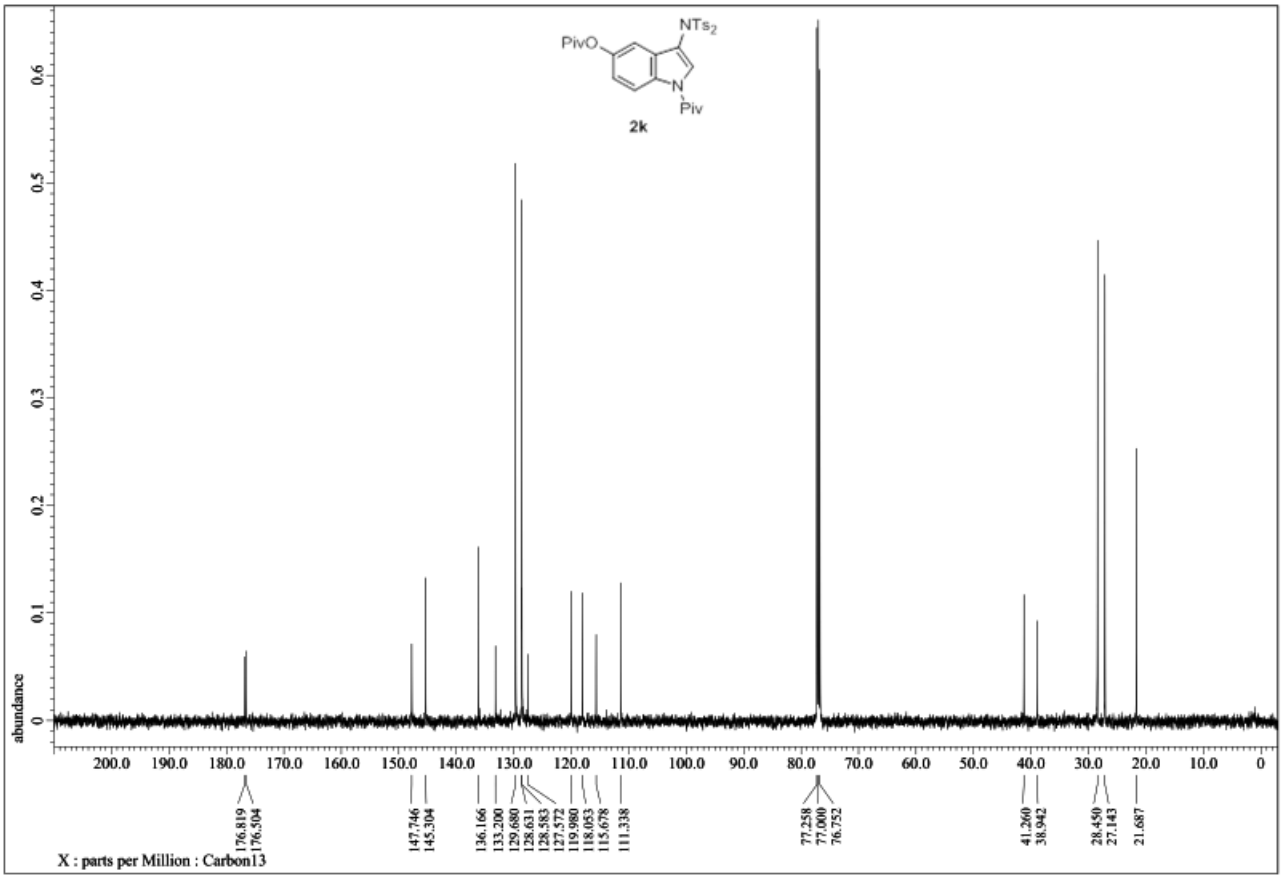
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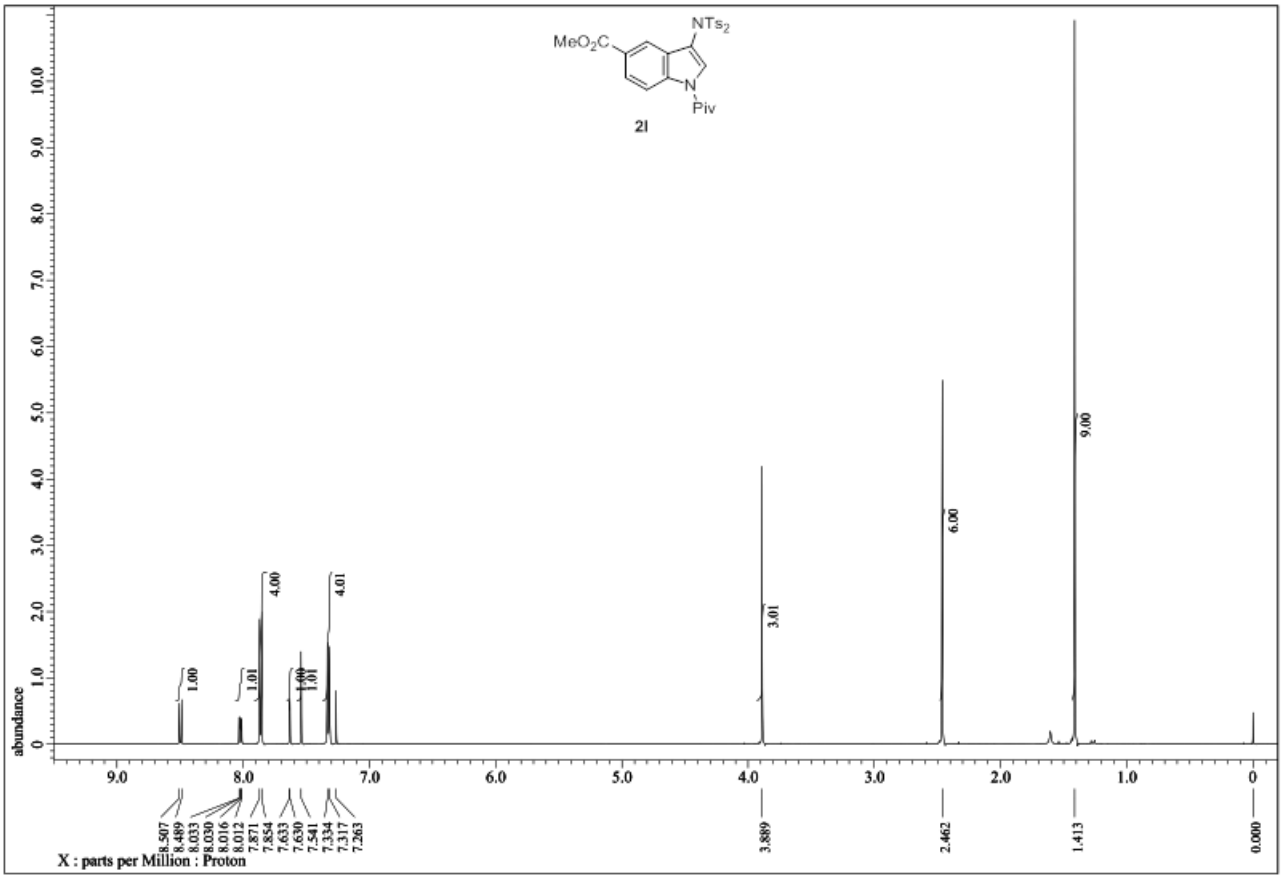
abundance
X : parts per Million : Carbon13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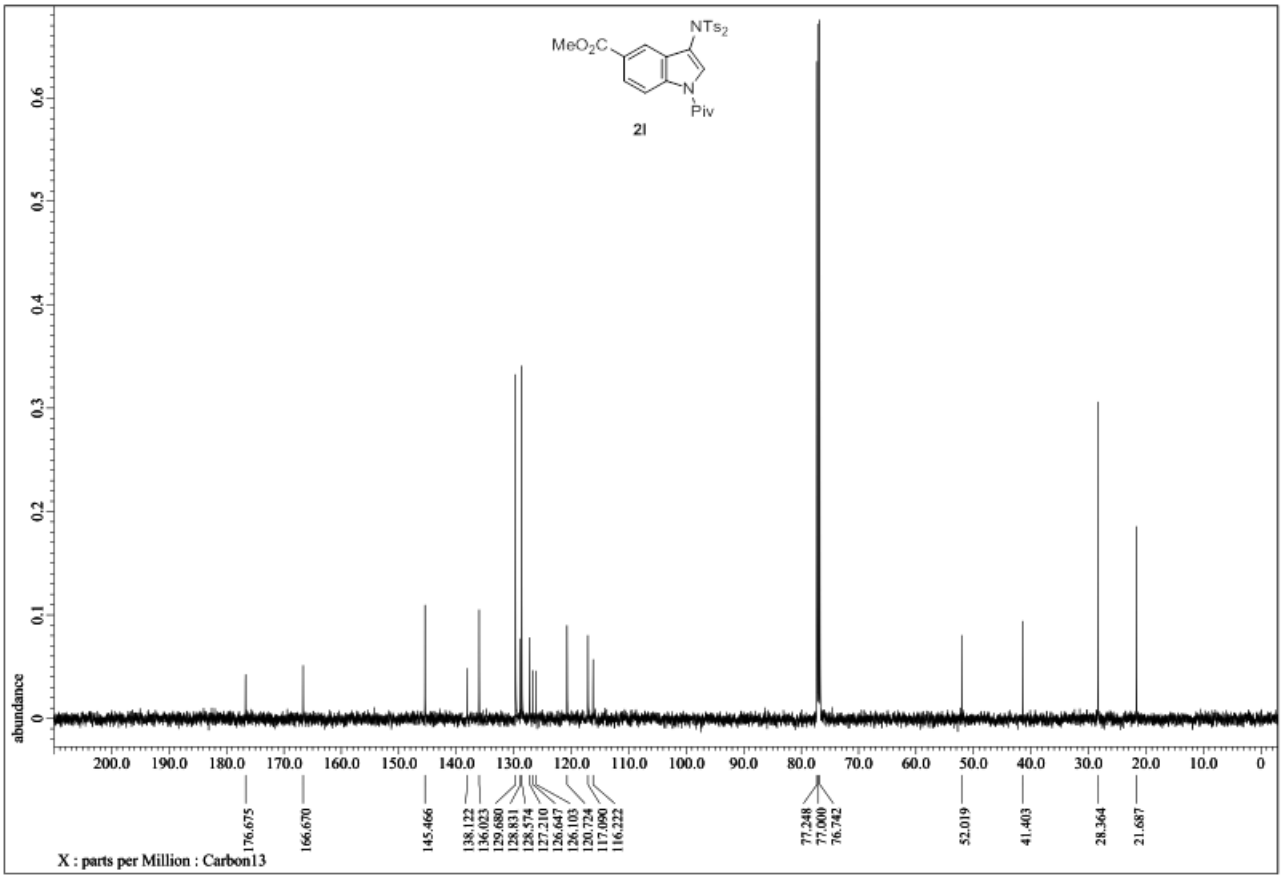
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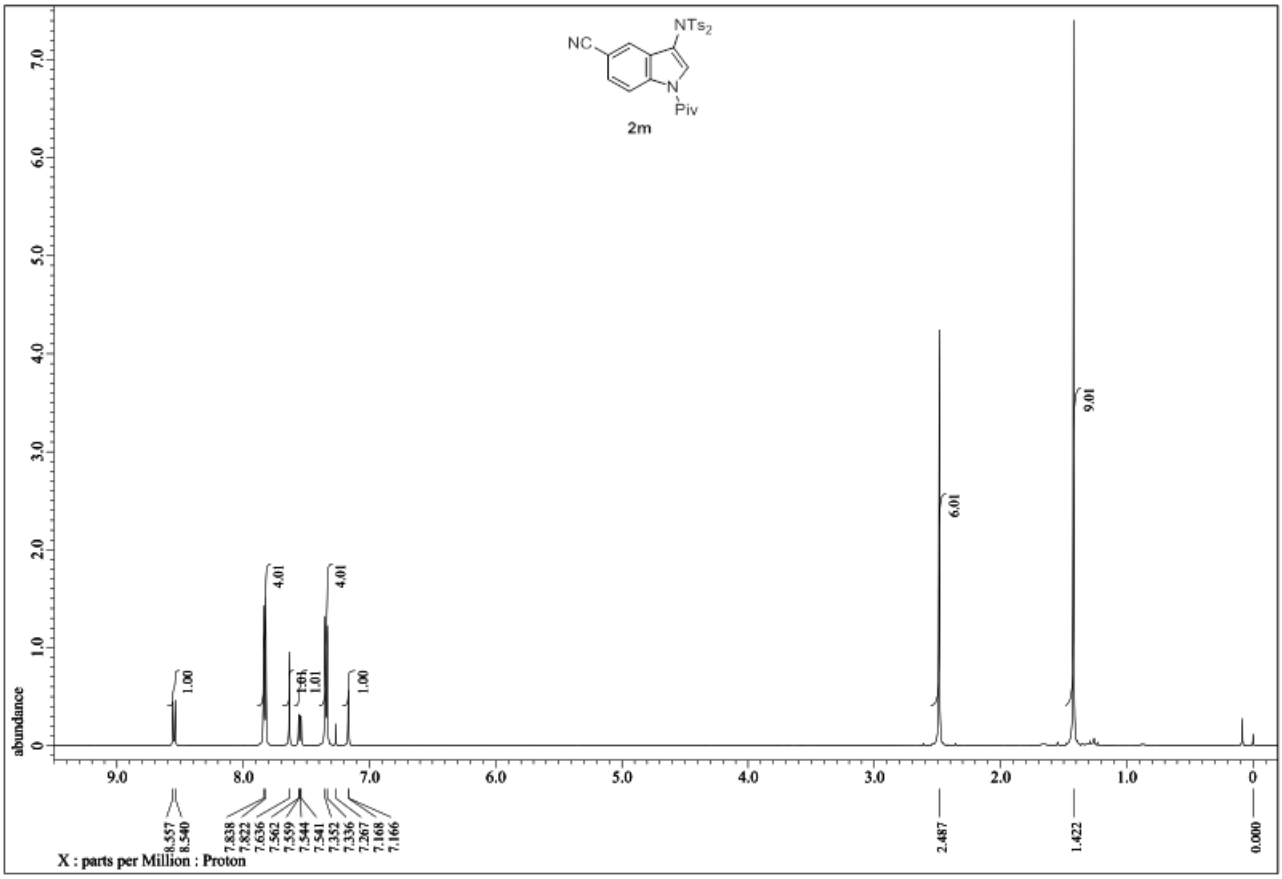
Piv
2l
abundance
X : parts per Million : Carbon13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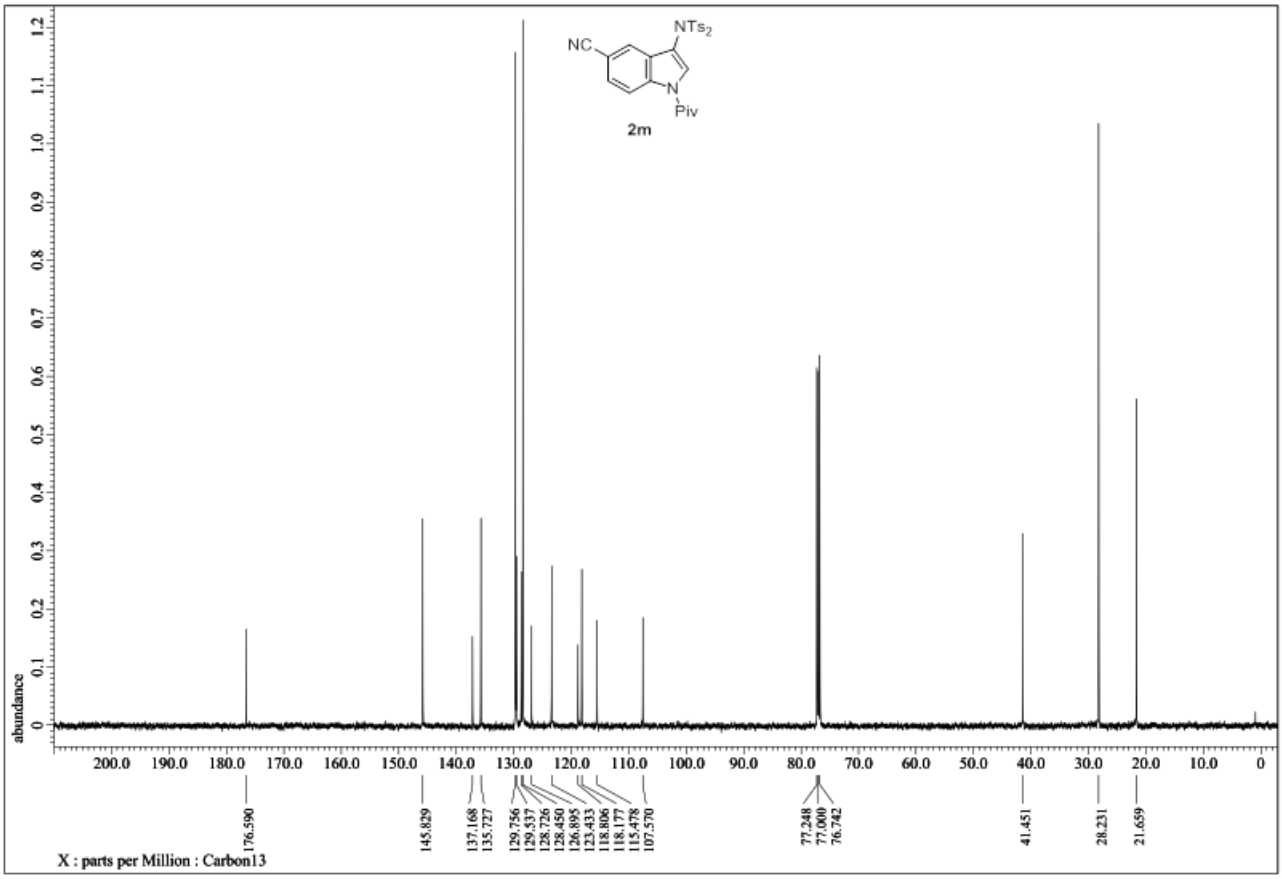
Piv
2m
abundance
X : parts per Million : Proton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abundance
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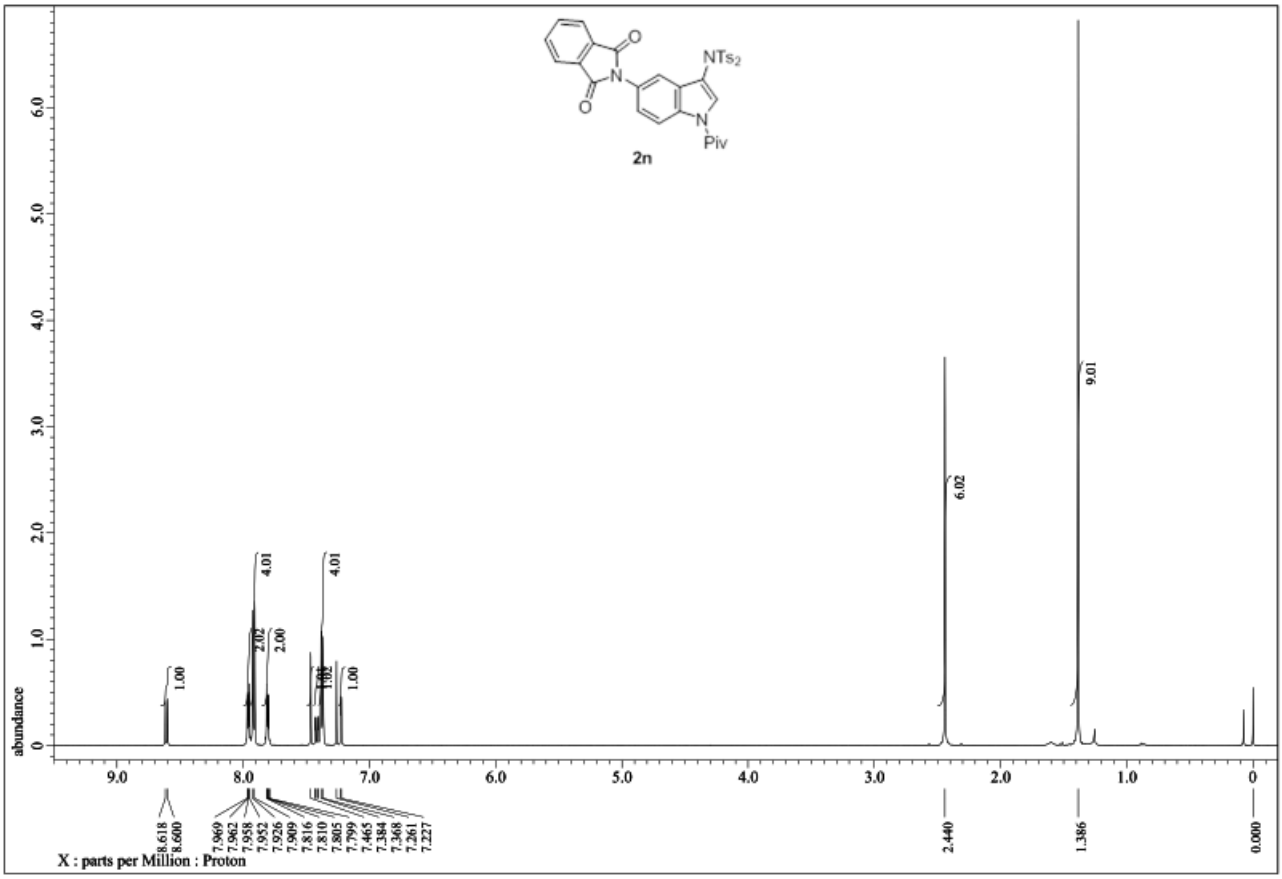
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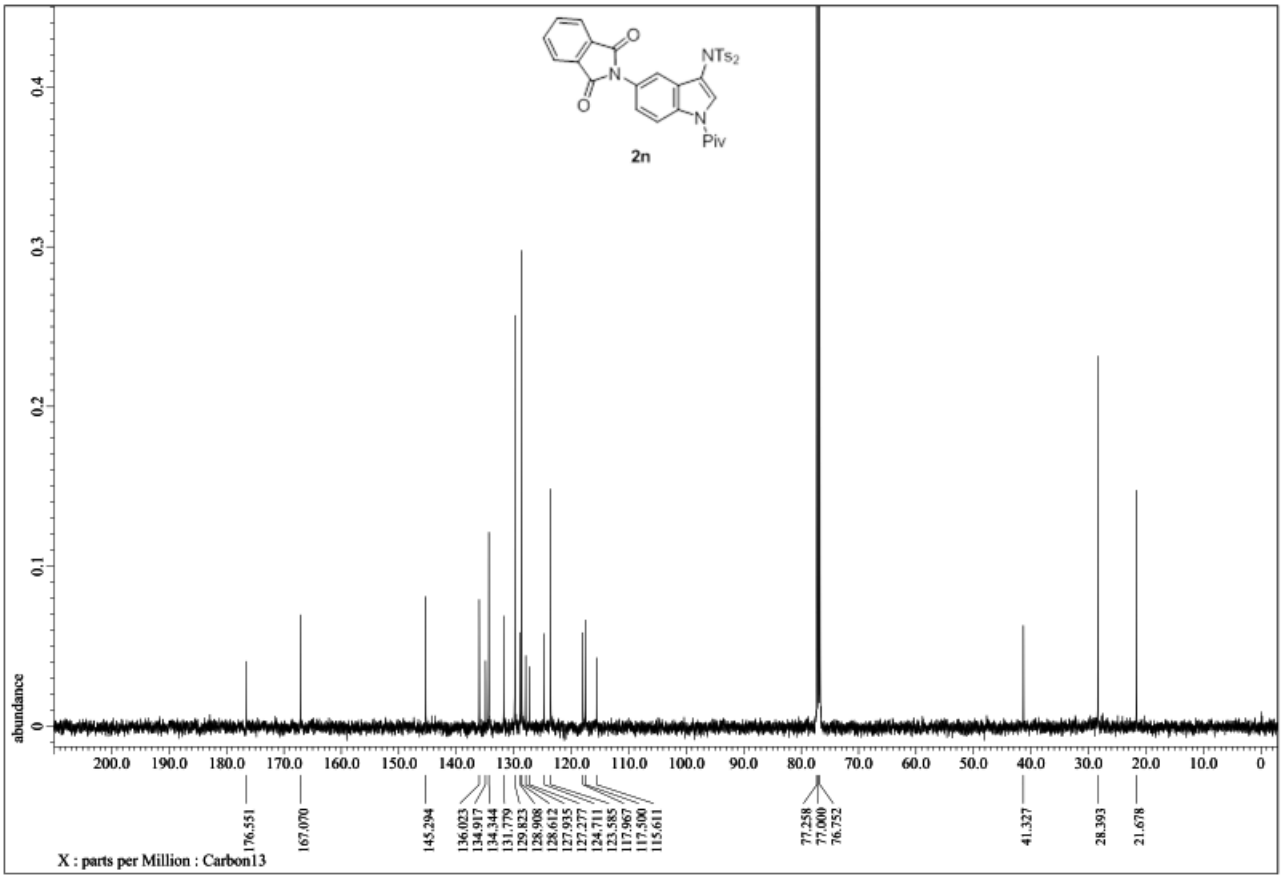
2n
abundance
X : parts per Million : Proton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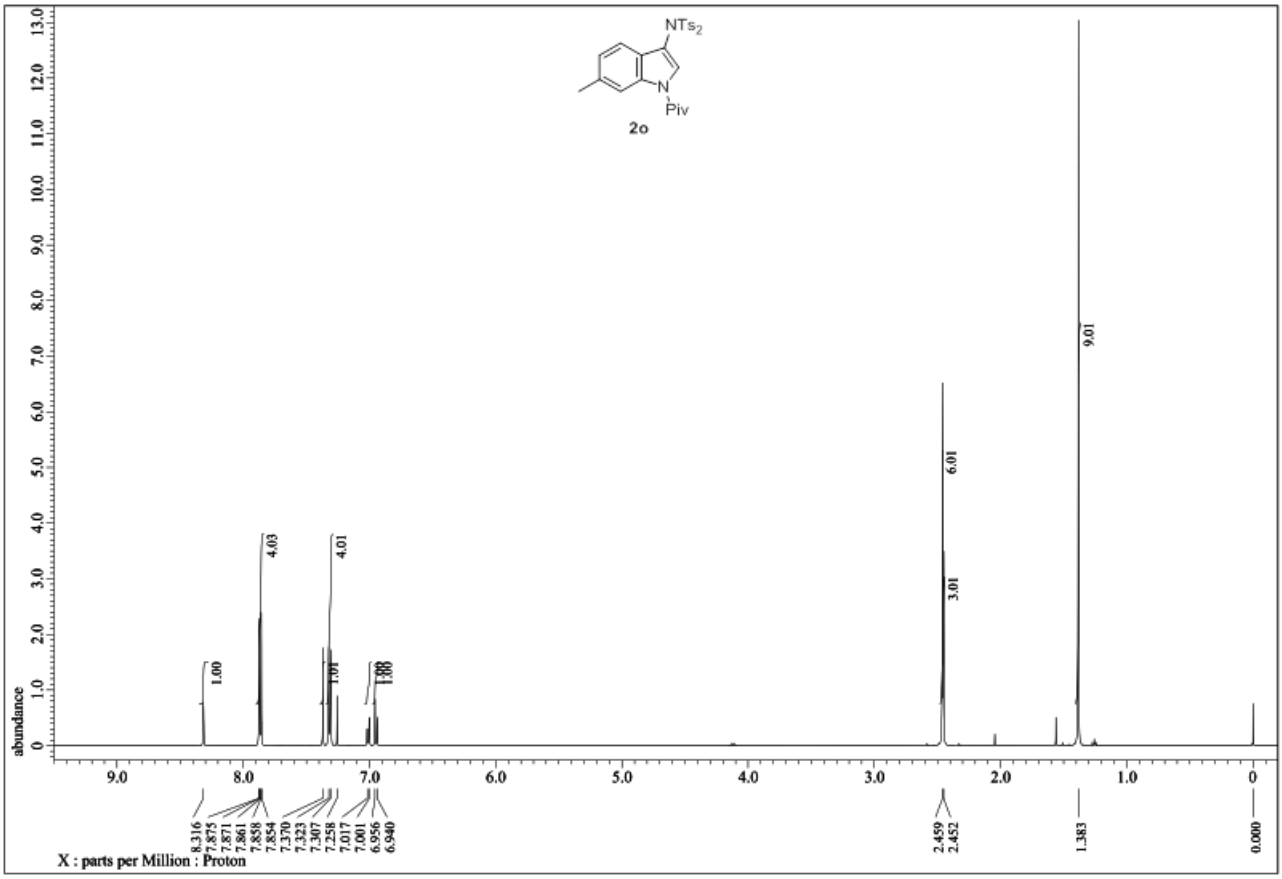
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N
Piv
2o
abundance
X : parts per Million : Proton
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6.01
4.03
4.01
3.01
1.00
1.01
8.316
7.875
7.871
7.861
7.858
7.854
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6.940
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2.452
1.383
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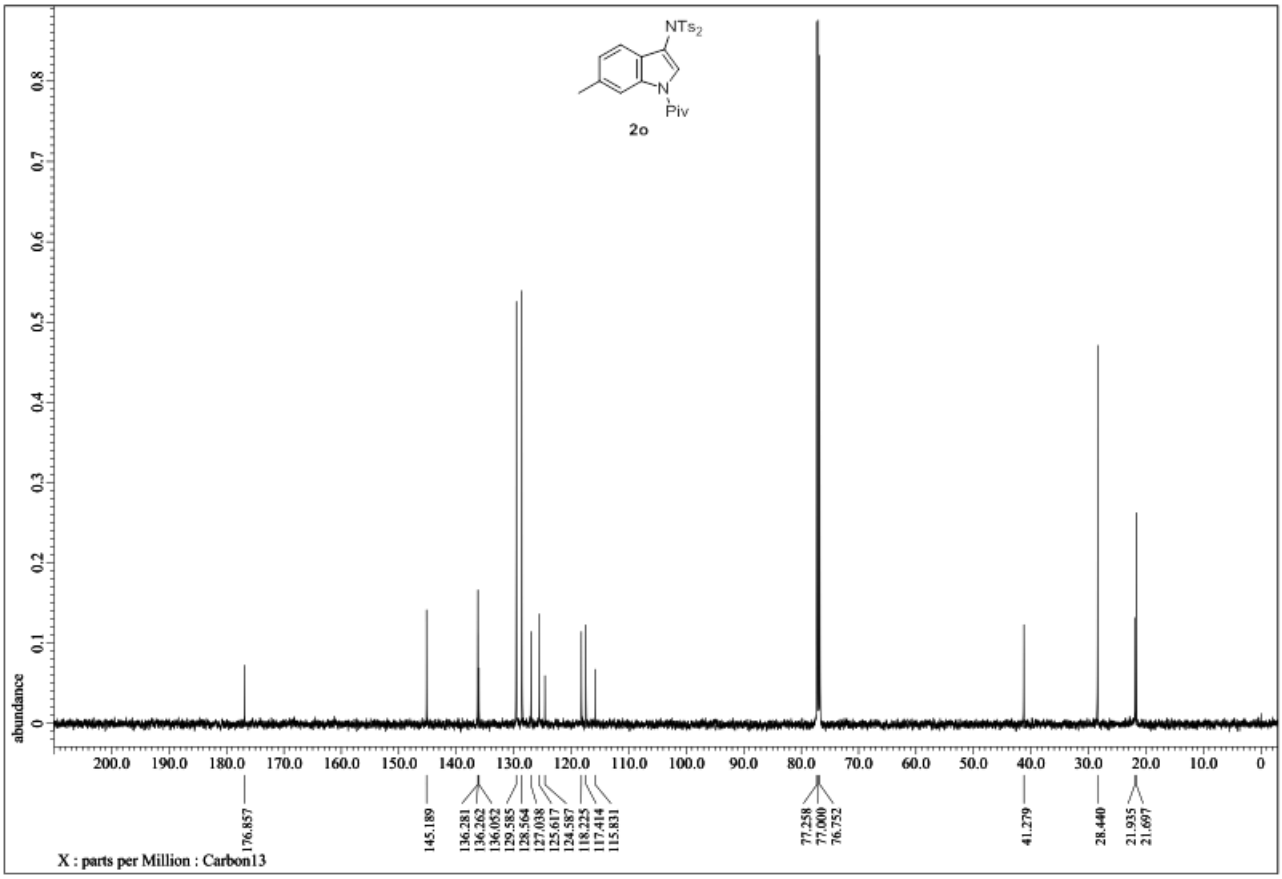
NTs2
N
Piv
2o
abundance
X : parts per Million : Carbon13
176.857
145.189
136.281
136.262
136.052
129.585
128.564
127.038
125.617
124.587
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117.414
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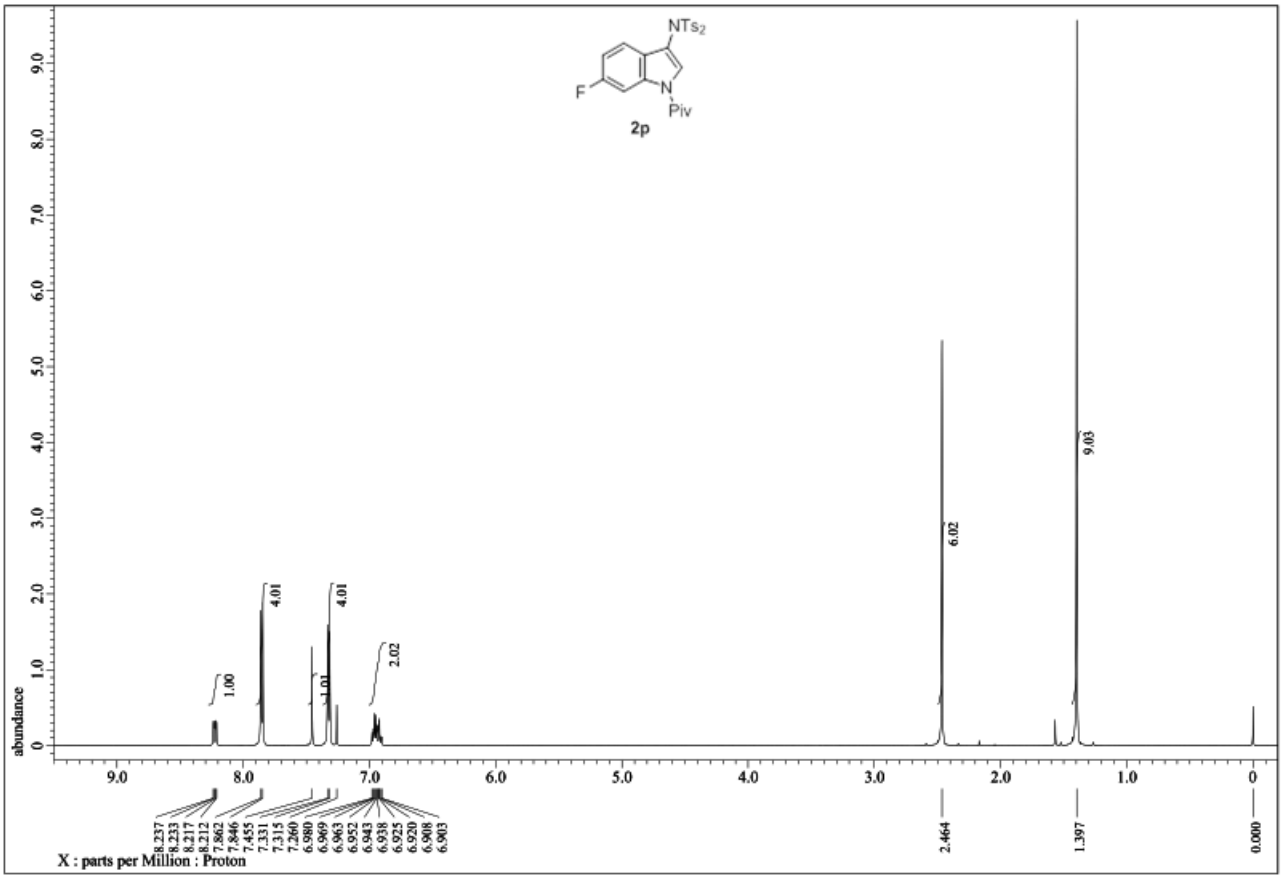
77.000
76.752
41.279
28.440
21.935
21.697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F
N
Piv
2p
abundance
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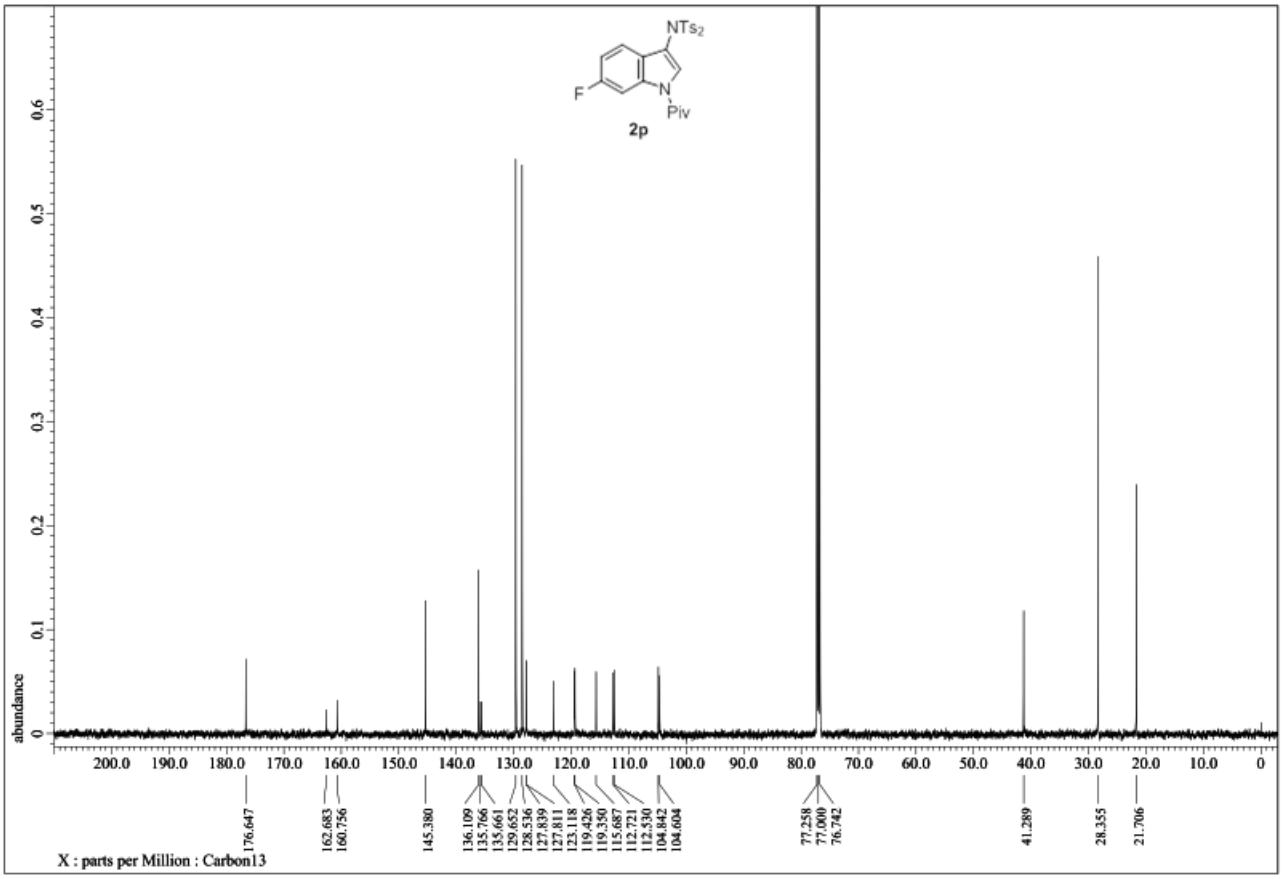
4.01
1.01
4.01
2.02
6.02
9.03
8.237
8.233
8.217
8.212
7.862
7.846
7.455
7.331
7.315
7.260
6.980
6.969
6.963
6.952
6.943
6.938
6.925
6.920
6.908
6.903
2.464
1.397
0.000
X : parts per Million : Proton


NTs2
F
N
Piv
2p
abundance
176.647
162.683
160.756
145.380
136.109
135.766
135.661
129.652
128.536
127.839
127.811
123.118
119.426
119.350
115.687
112.721
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77.258
77.000
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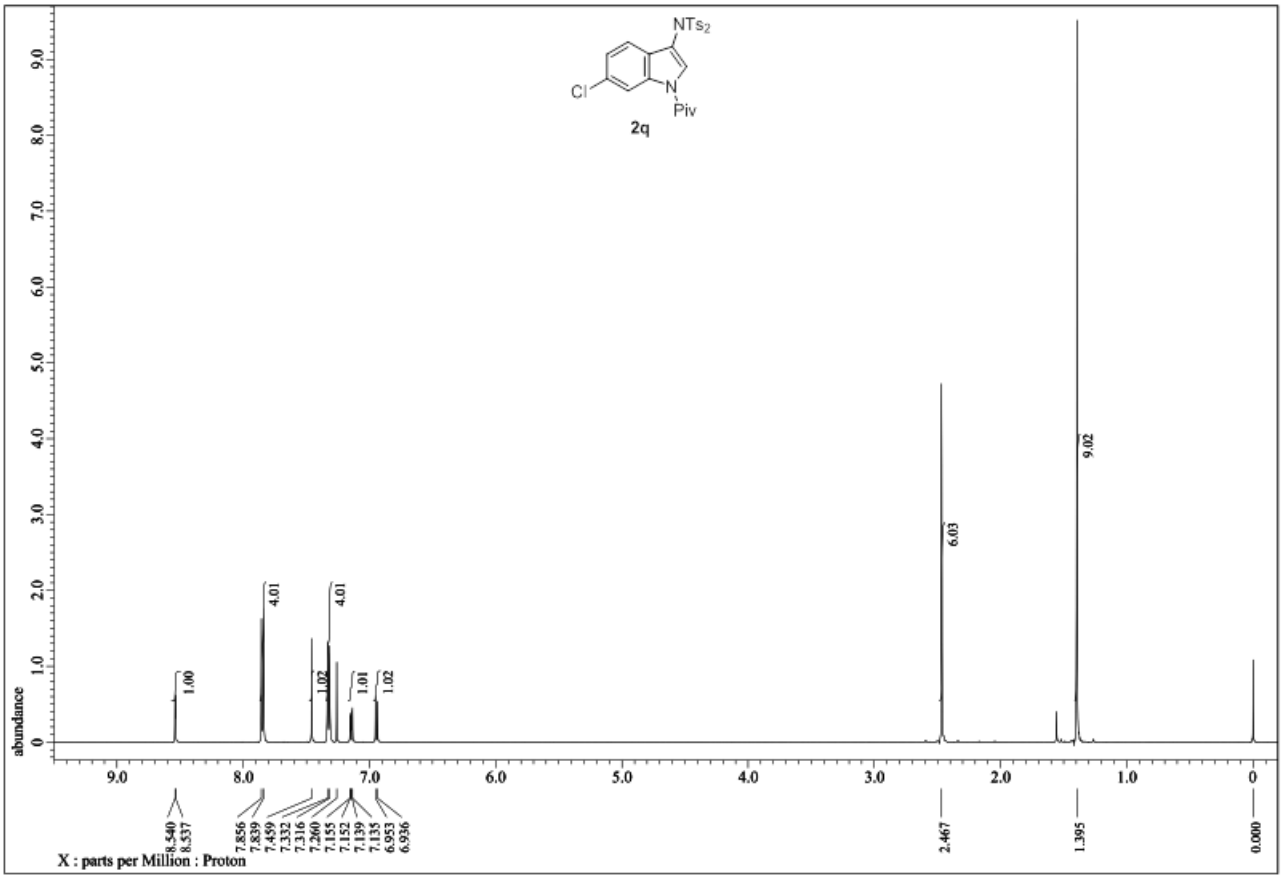
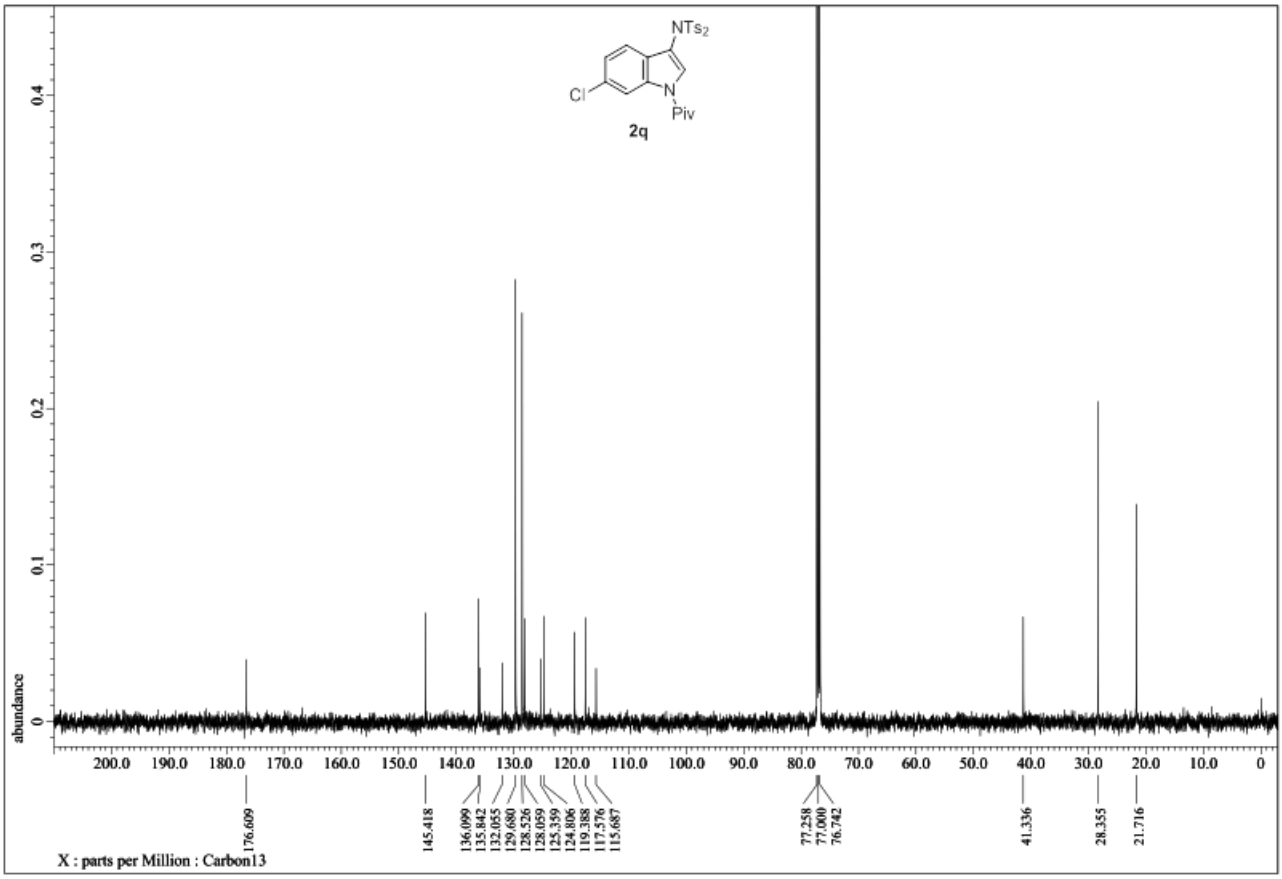
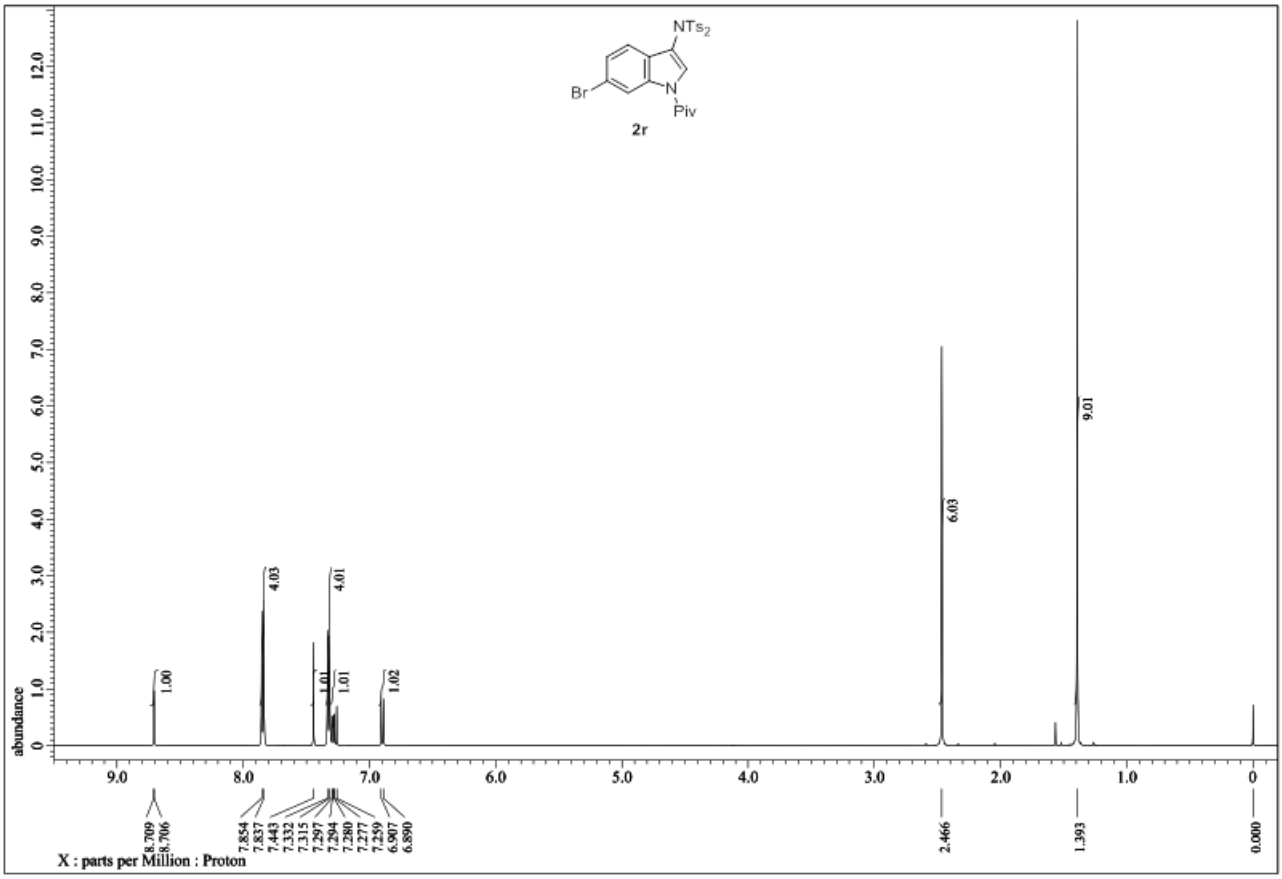
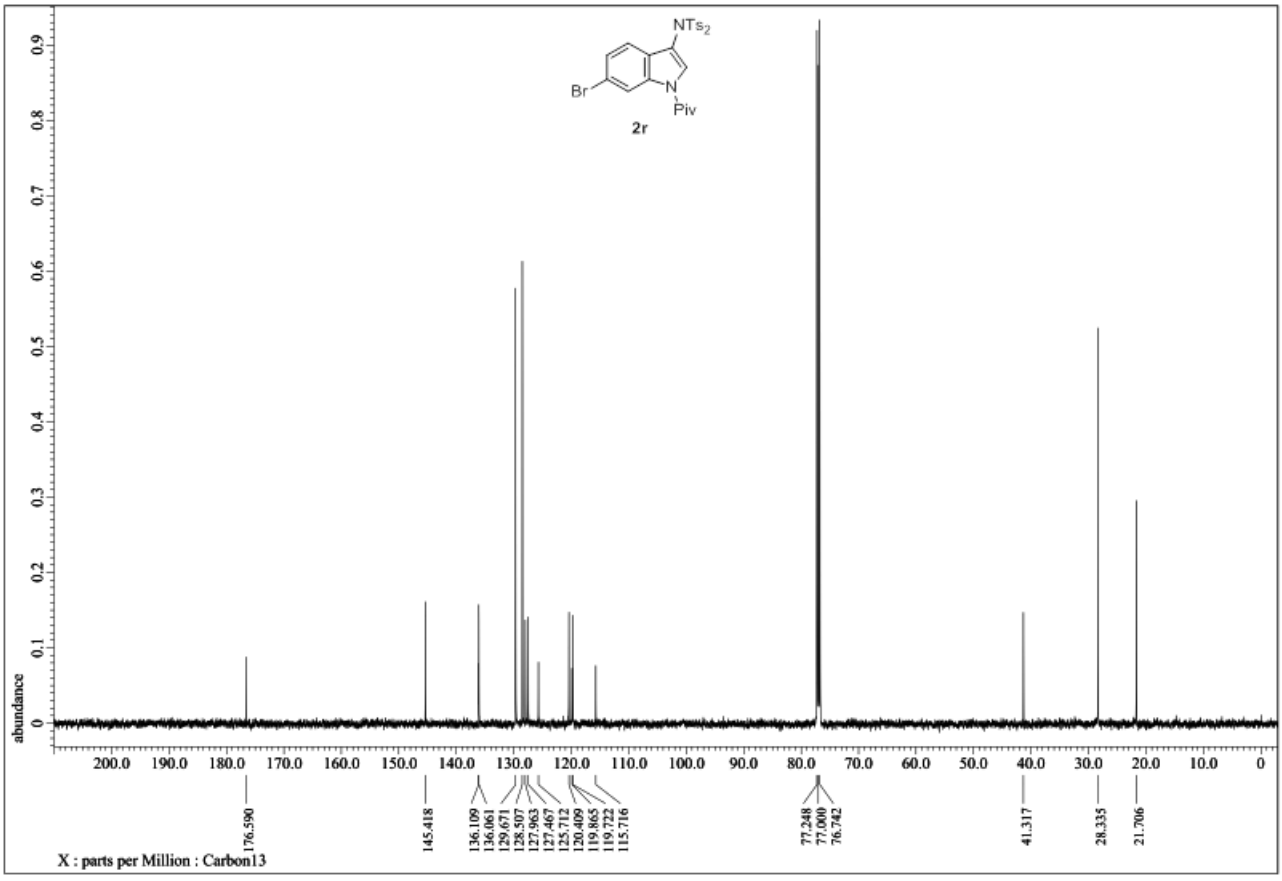
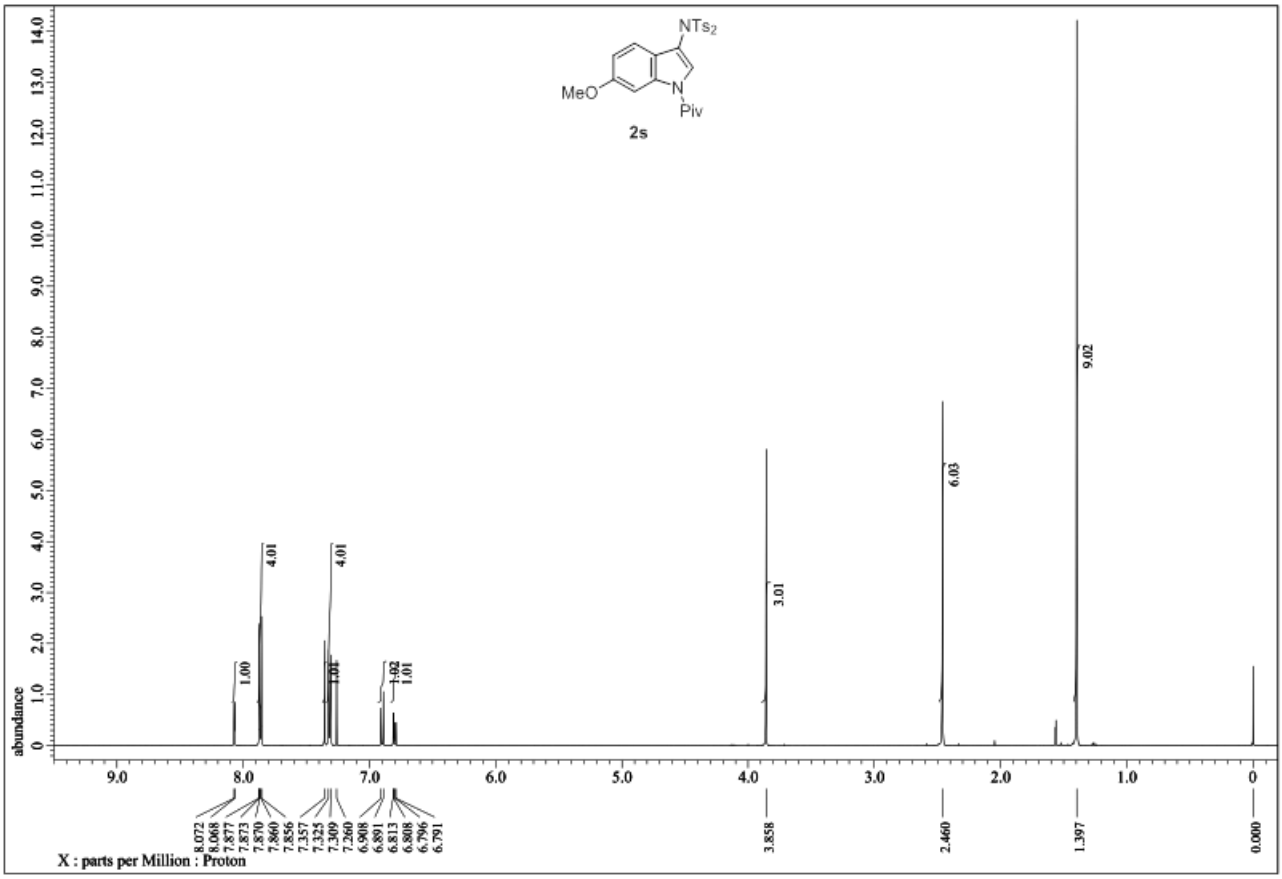
41.289
28.355
21.706
X : parts per Million : Carbon13

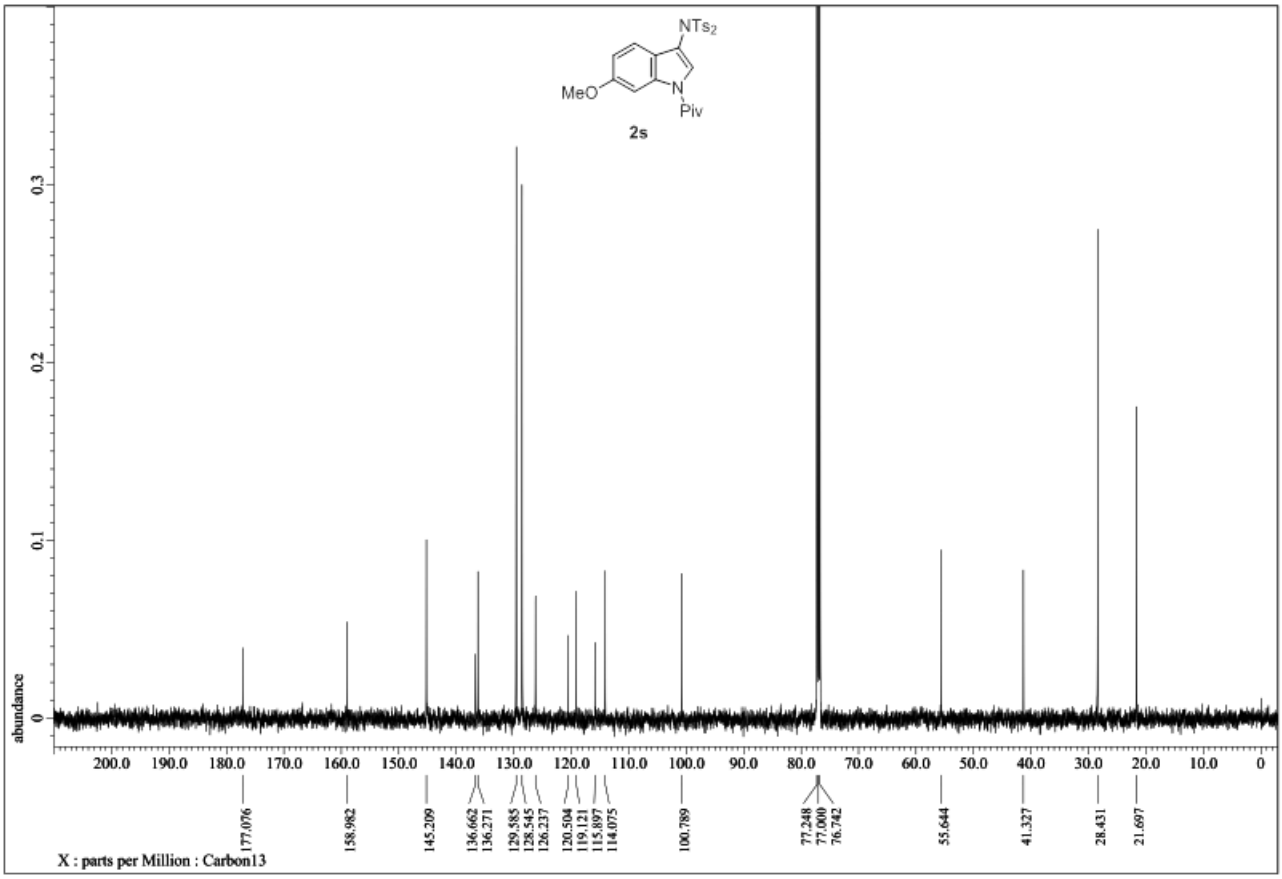

NTs2
Cl
N
Piv
2q
abundance
X : parts per Million : Proton
1.00
4.01
1.02
4.01
1.01
1.02
6.03
9.02
8.540
8.537
7.856
7.839
7.459
7.332
7.316
7.260
7.155
7.152
7.139
7.135
6.953
6.936
2.467
1.395
0.000

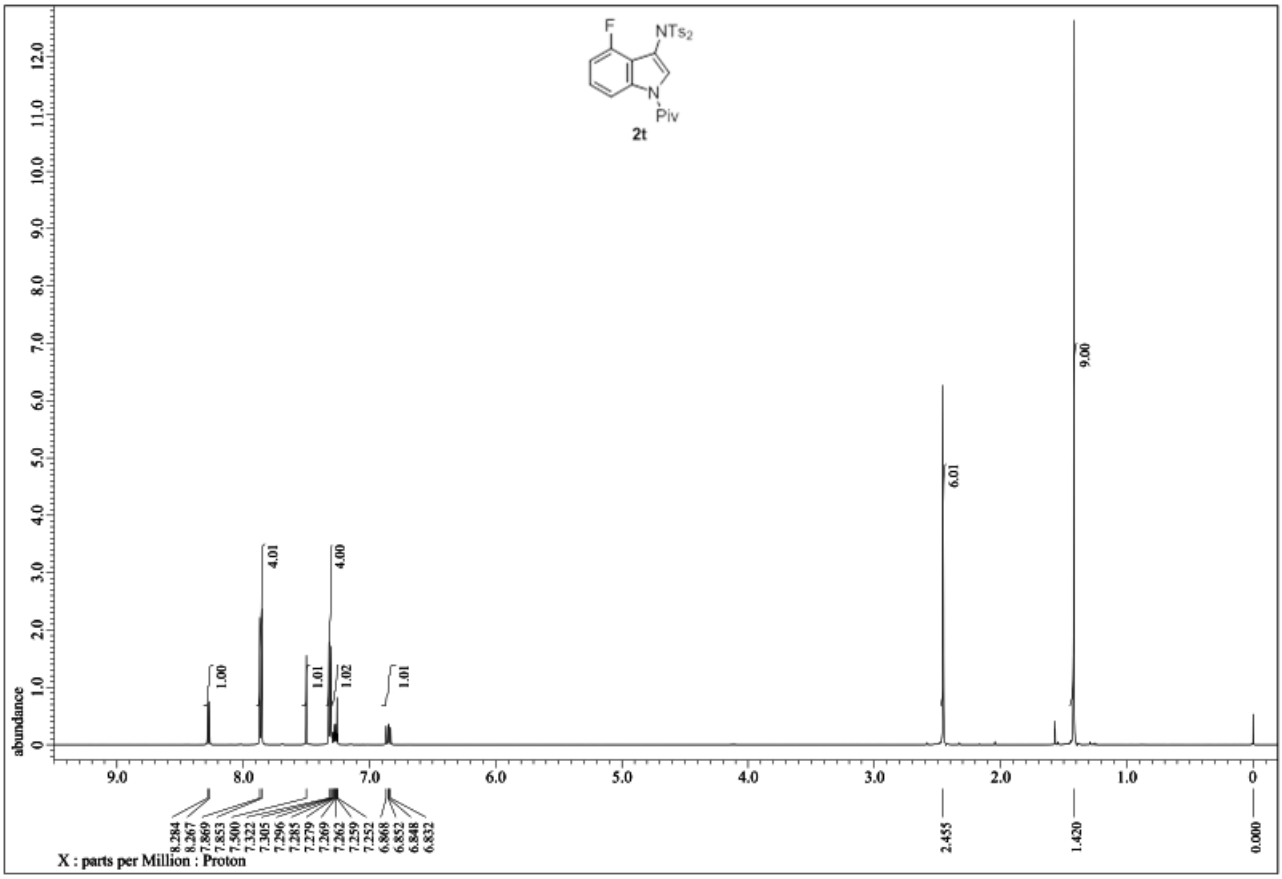

NTs2
Cl
N
Piv
2q
abundance
X : parts per Million : Carbon13
176.609
145.418
136.099
135.842
132.055
129.680
128.526
128.059
125.359
124.806
119.388
117.576
115.687
77.258
77.000
76.742
41.336
28.355
21.716

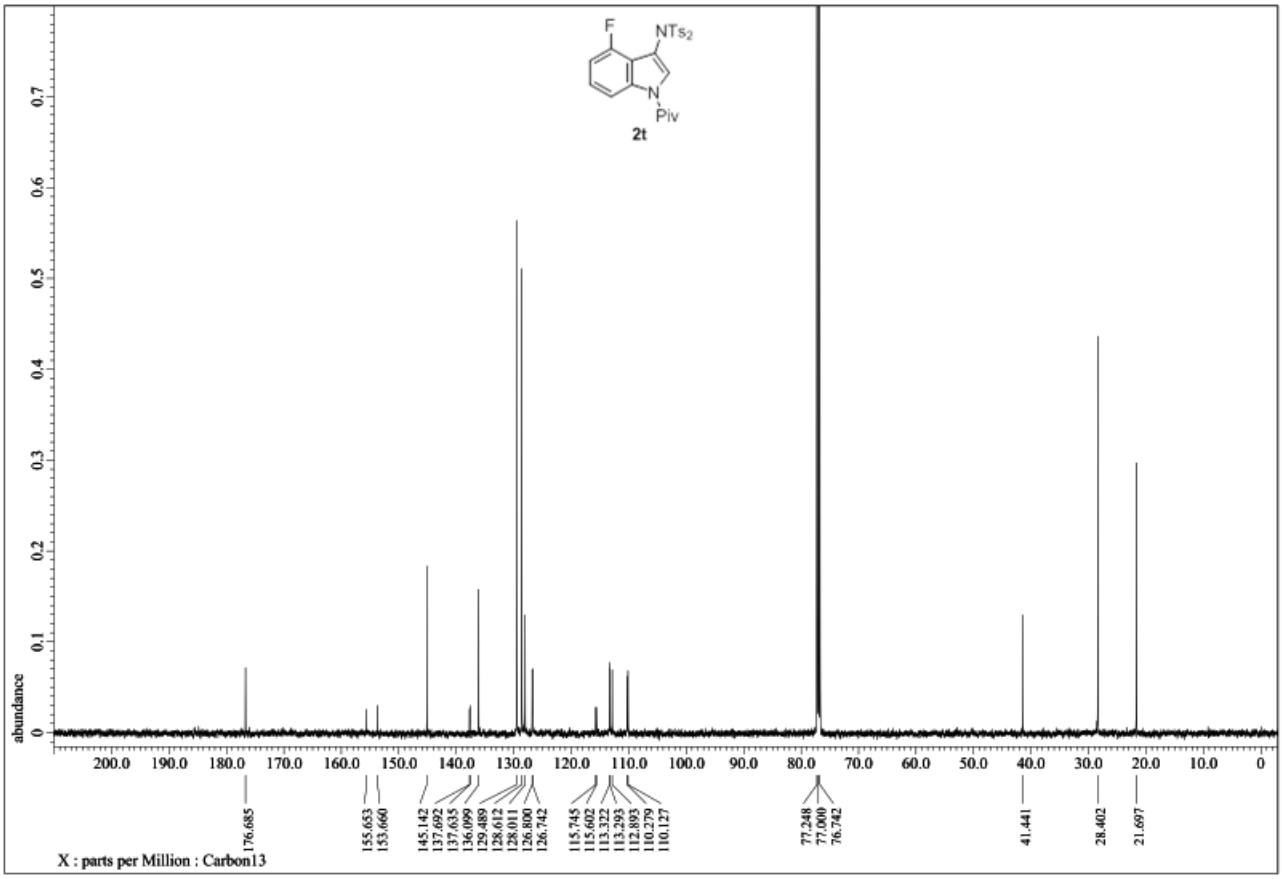

NTs2
Br
N
Piv
2r
abundance
X : parts per Million : Proton
1.00
4.03
4.01
1.01
1.01
1.02
6.03
9.01
8.709
8.706
7.854
7.837
7.443
7.332
7.315
7.297
7.294
7.280
7.277
7.259
6.907
6.890
2.466
1.393
0.000

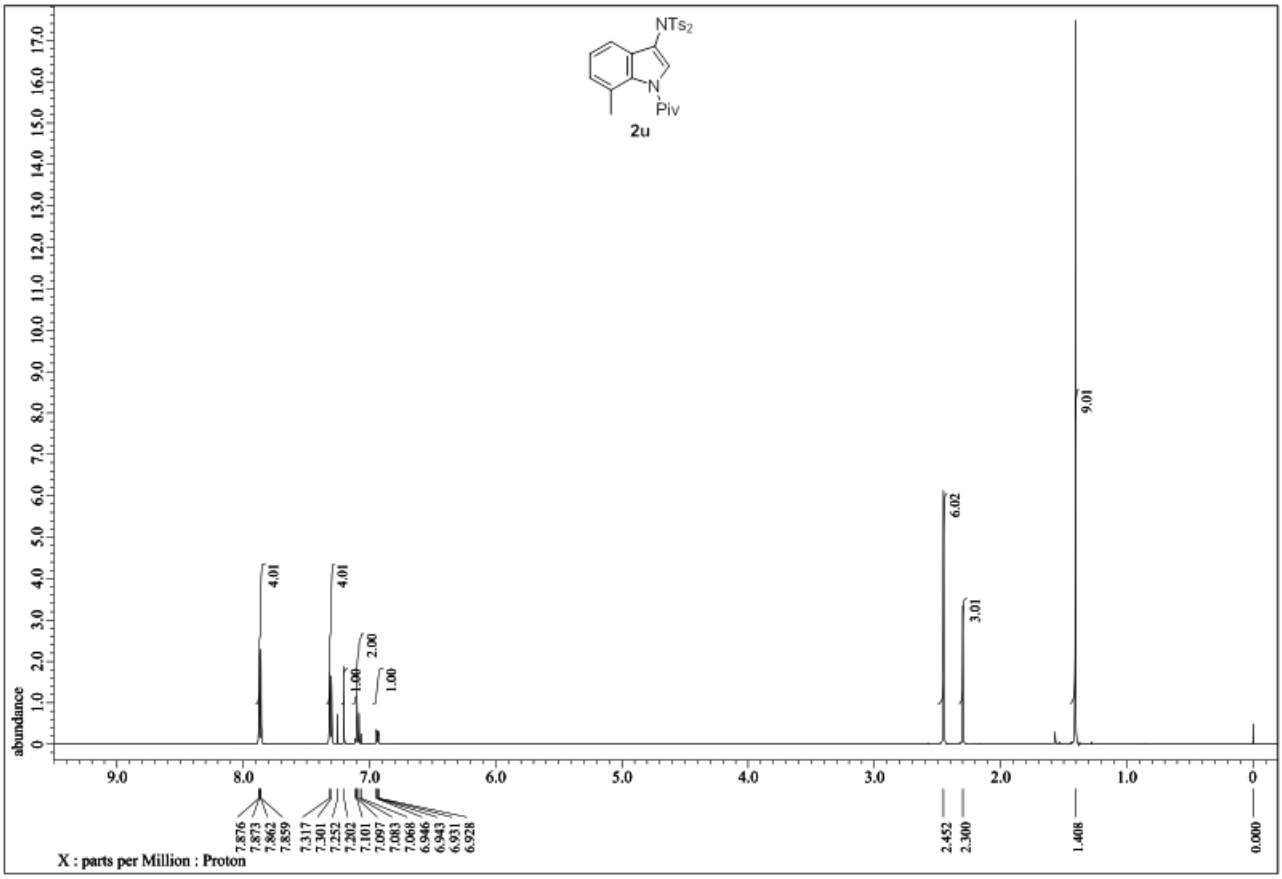

NTs2
Br
N
Piv
2r
abundance
X : parts per Million : Carbon13
176.590
145.418
136.109
136.061
129.671
128.597
127.963
127.467
125.712
120.409
119.865
119.722
115.716
77.248
77.000
76.742
41.317
28.335
21.706

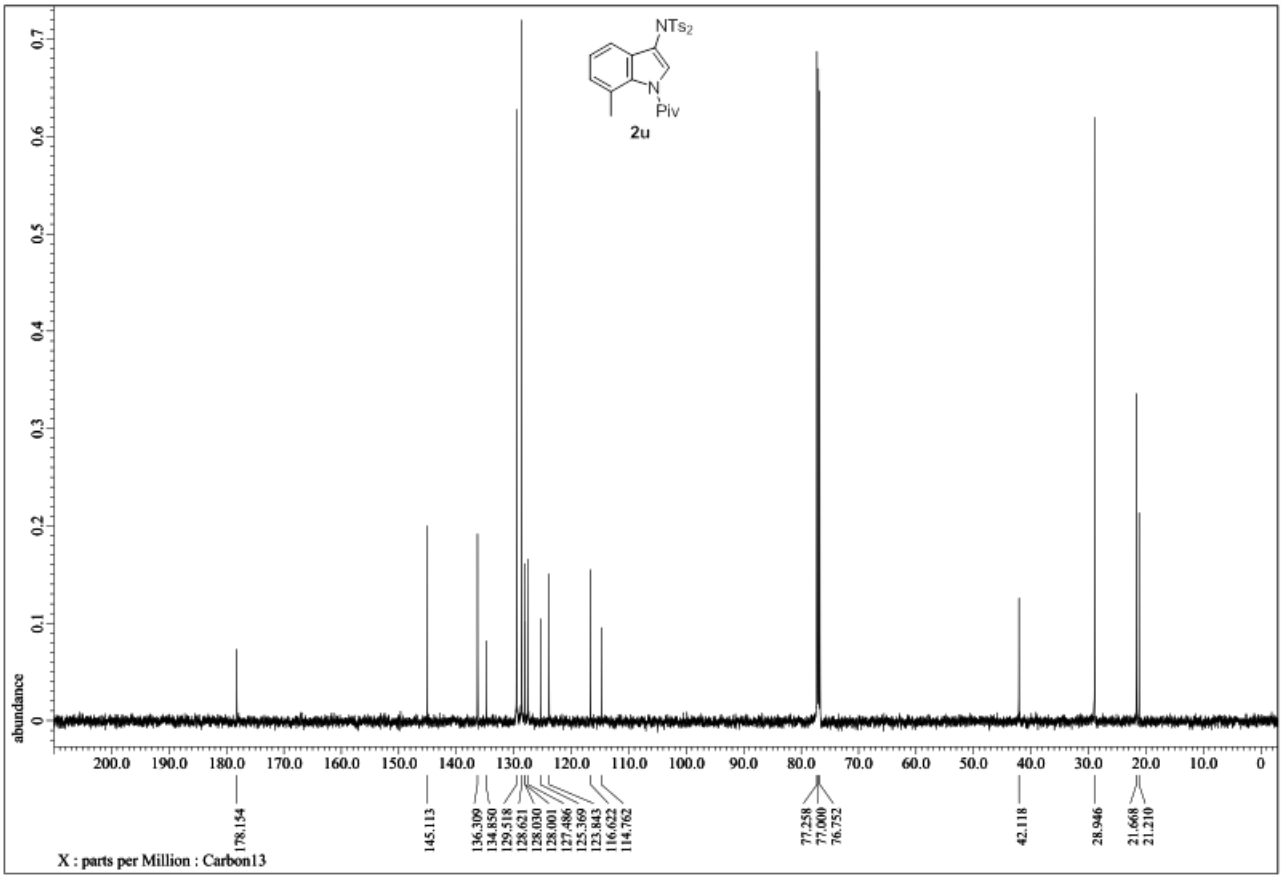

NTs2
MeO
N
Piv
2s
abundance
1.00
4.01
4.01
1.01
1.01
1.02
1.01
3.01
6.03
9.02
8.072
8.068
7.877
7.873
7.870
7.860
7.856
7.357
7.325
7.309
7.260
6.908
6.891
6.813
6.808
6.796
6.791
3.858
2.460
1.397
0.000
X : parts per Million : Proton

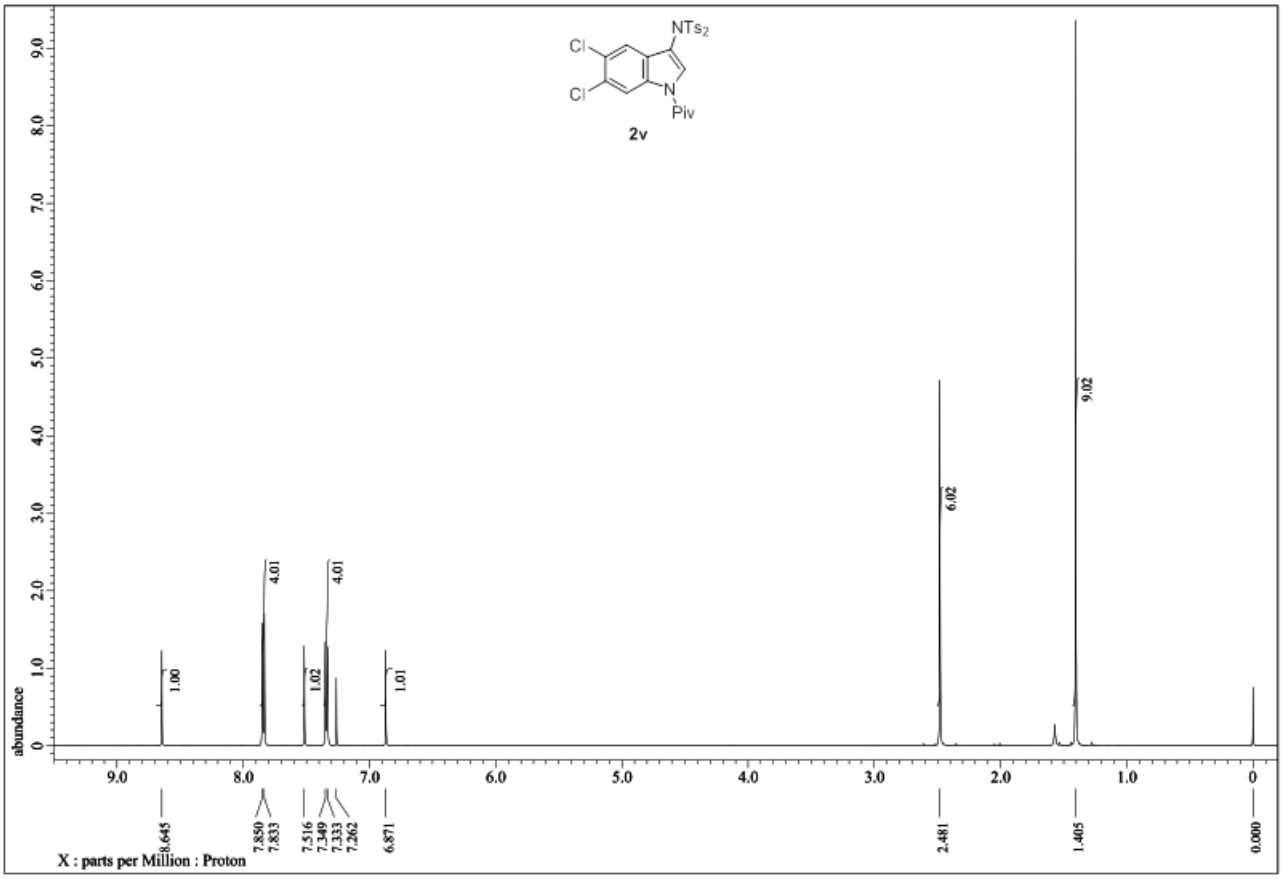

NTs2
MeO
N
Piv
2s
abundance
177.076
158.982
145.209
136.662
136.271
129.585
128.545
126.237
120.504
119.121
115.897
114.075
100.789
77.248
77.000
76.742
55.644
41.327
28.431
21.697
X : parts per Million : Carbon13

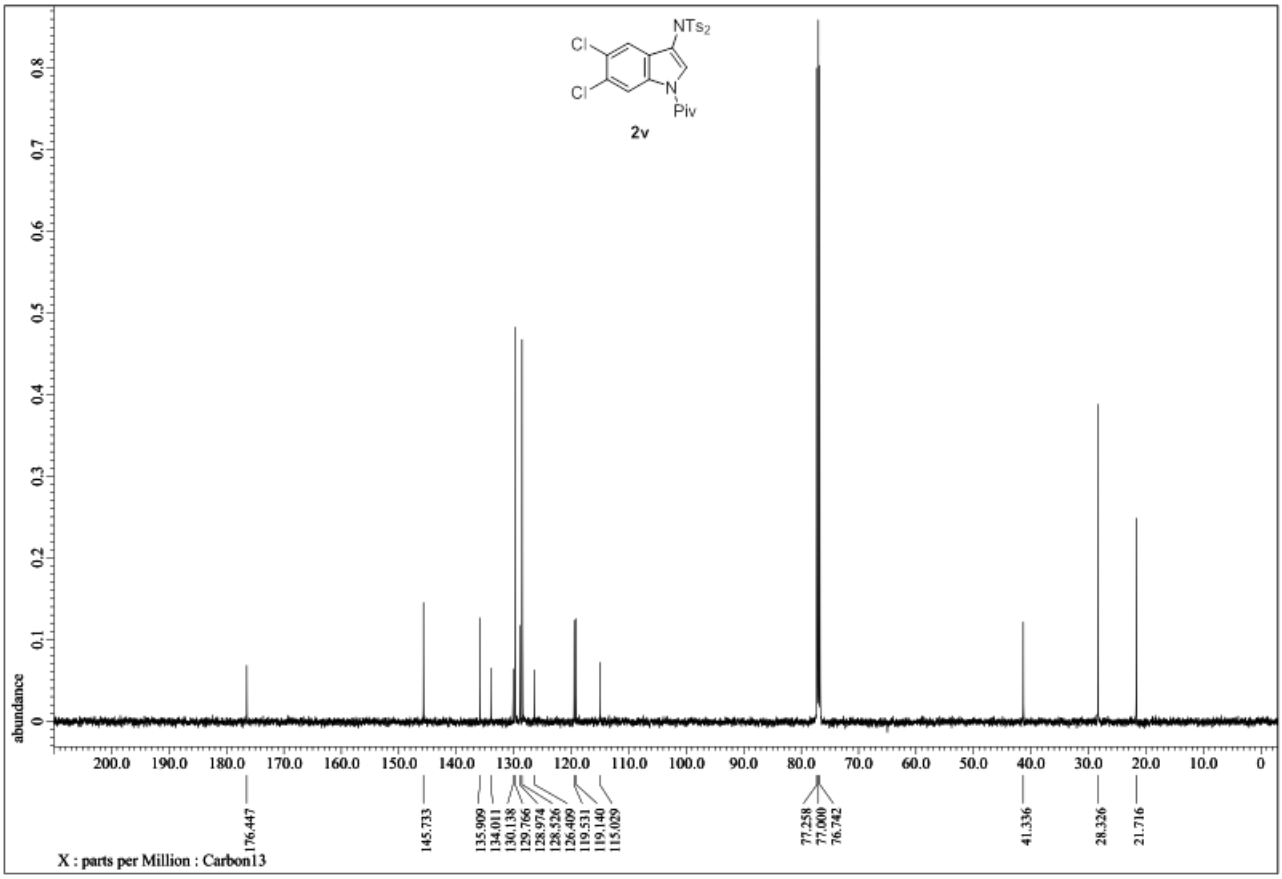

F
NTs2
N
Piv
2t
abundance
1.00
4.01
4.00
1.01
1.02
1.01
6.01
9.00
8.284
8.267
7.869
7.853
7.500
7.322
7.305
7.296
7.285
7.279
7.269
7.262
7.259
7.252
6.868
6.852
6.848
6.832
2.455
1.420
0.000
X : parts per Million : Proton

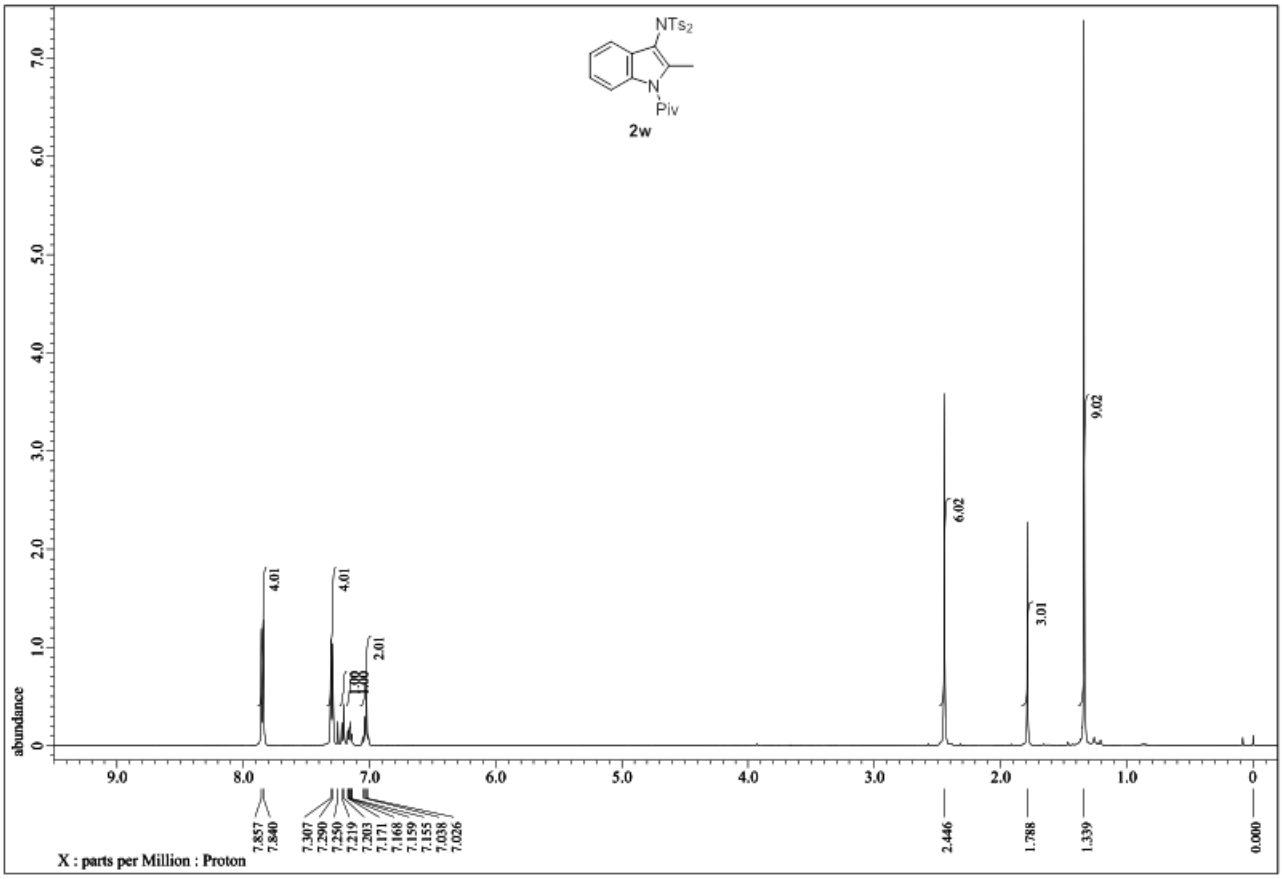

F
NTs2
N
Piv
2t
abundance
176.685
155.653
153.660
145.142
137.692
137.635
136.099
129.489
128.612
128.011
126.800
126.742
115.745
115.602
113.322
113.293
112.893
110.279
110.127
77.248
77.000
76.742
41.441
28.402
21.697
X : parts per Million : Carbon13

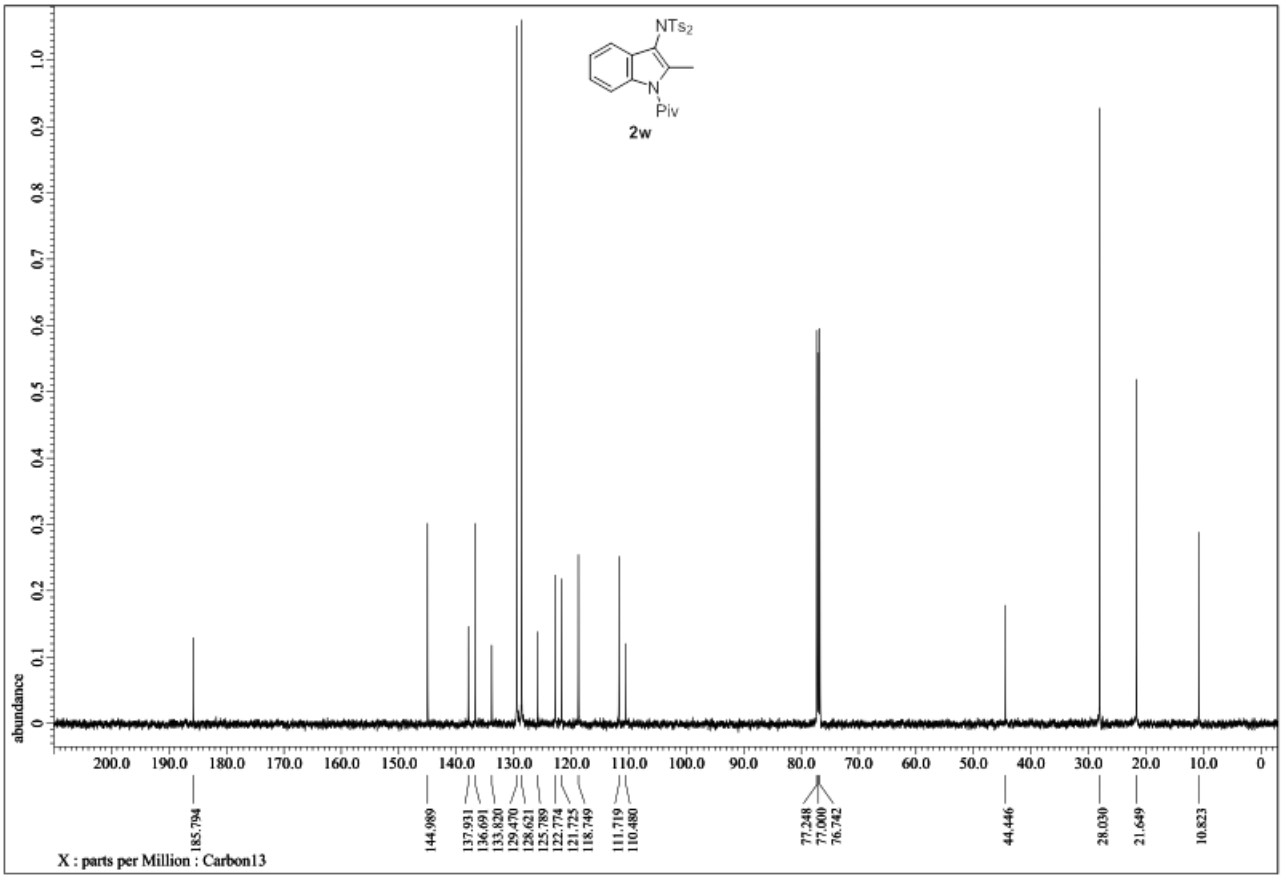

NTs2
N
Piv
2u
abundance
4.01
4.01
2.00
1.00
1.00
6.02
3.01
9.01
7.876
7.873
7.862
7.859
7.317
7.301
7.252
7.202
7.101
7.097
7.083
7.068
6.946
6.943
6.931
6.928
2.452
2.300
1.408
0.000
X : parts per Million : Proton

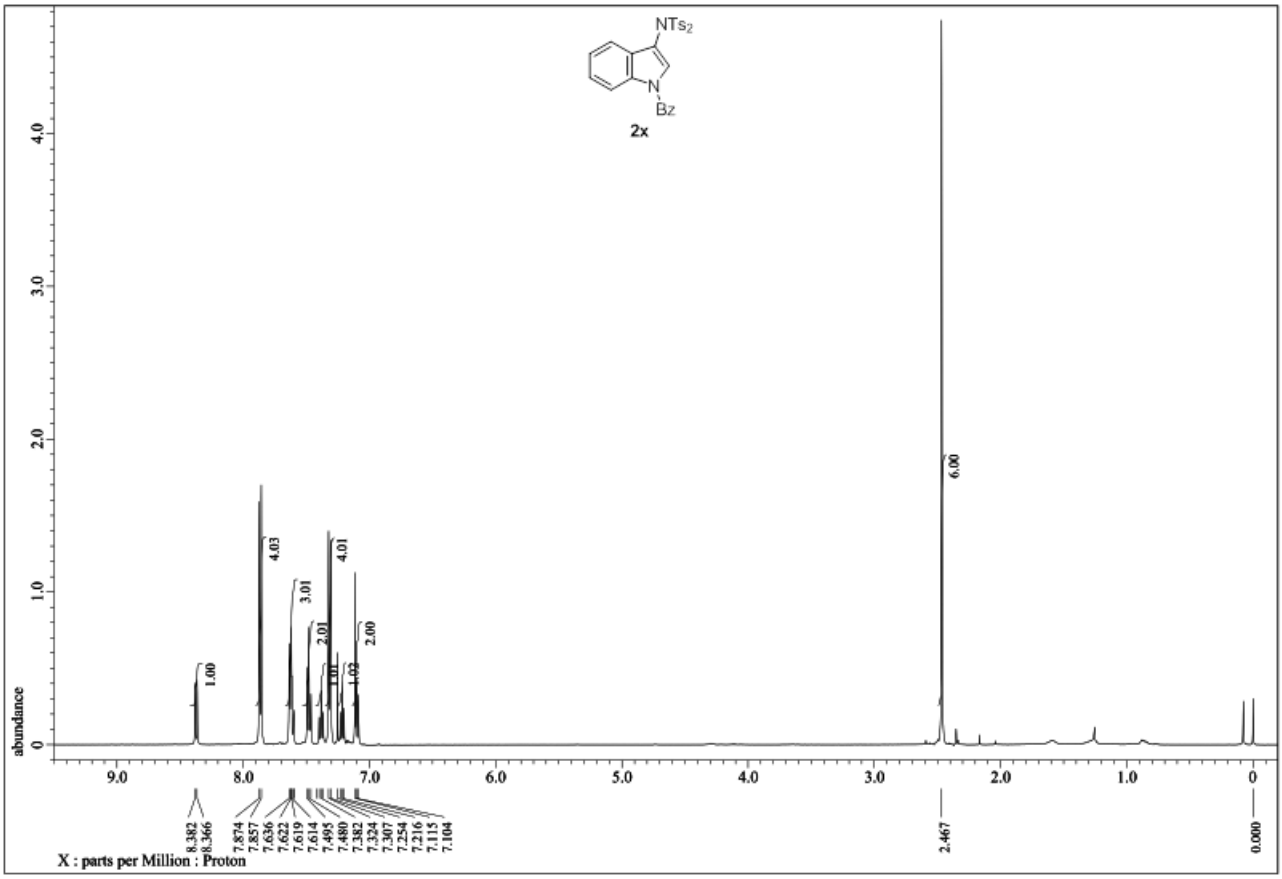

NTs2
N
Piv
2u
abundance
178.154
145.113
136.309
134.850
129.518
128.621
128.030
128.001
127.486
125.369
123.843
116.622
114.762
77.258
77.000
76.752
42.118
28.946
21.668
21.210
X : parts per Million : Carbon13

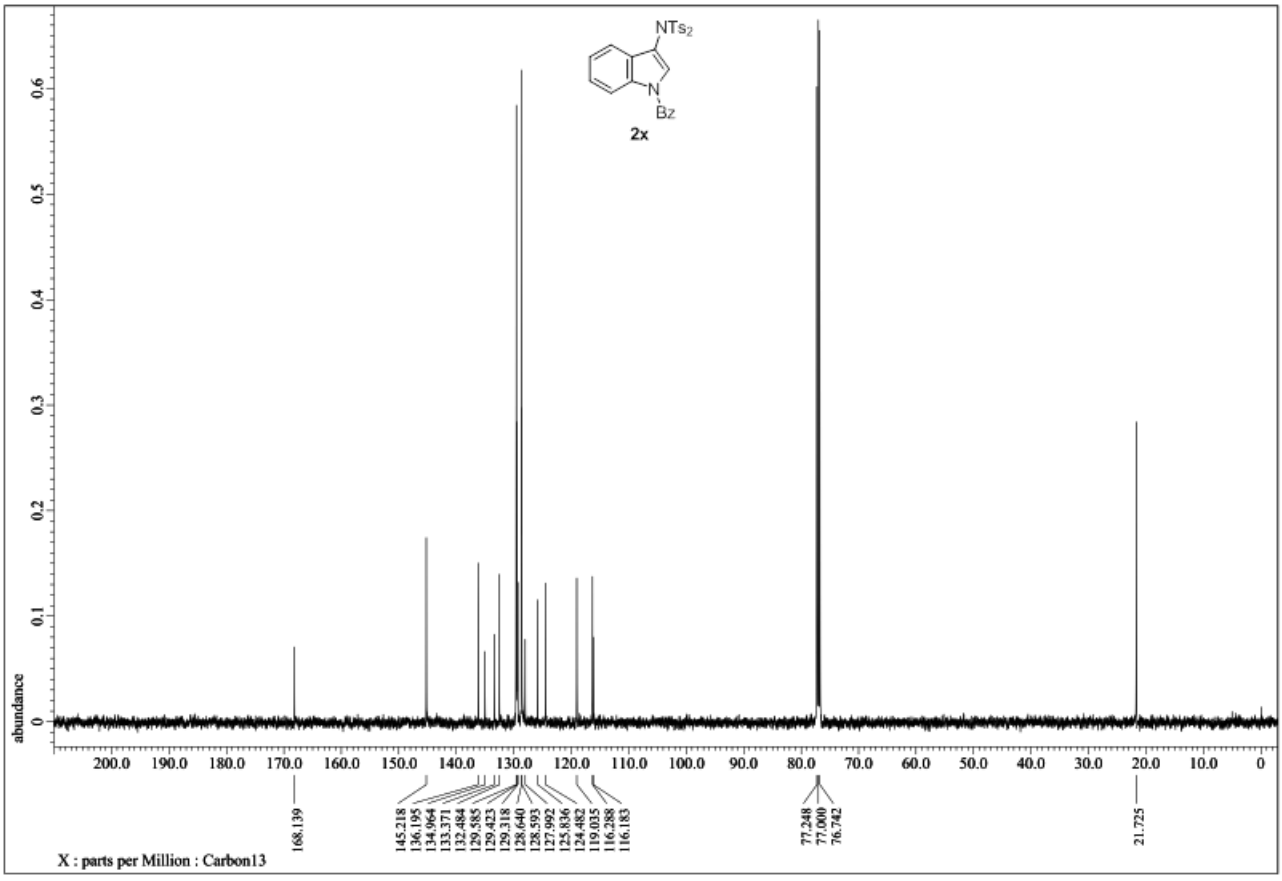

NTs2
Cl
Cl
N
Piv
2v
abundance
1.00
4.01
4.01
1.02
1.01
6.02
9.02
8.645
7.850
7.833
7.516
7.349
7.333
7.262
6.871
2.481
1.405
0.000
X : parts per Million : Proton

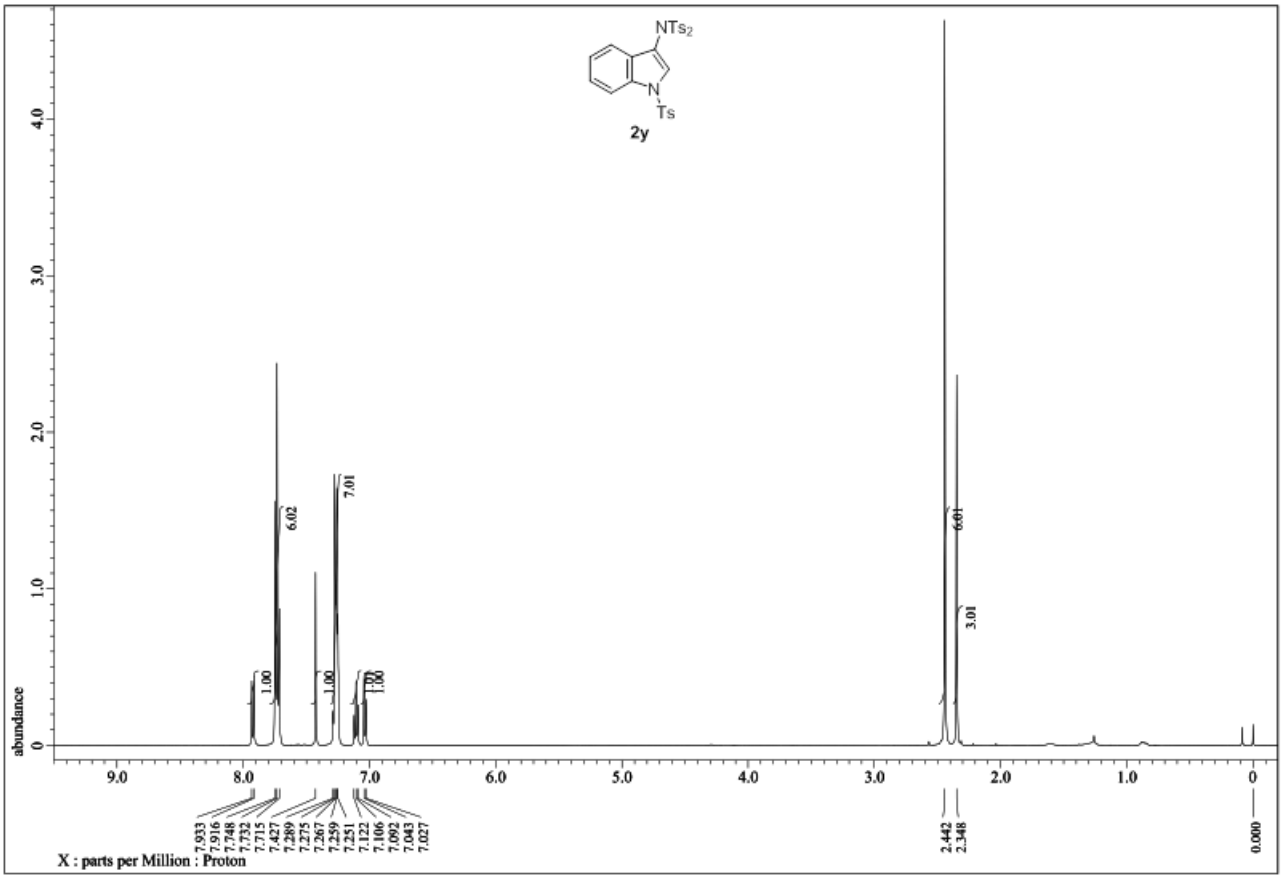

NTs2
Cl
Cl
N
Piv
2v
abundance
176.447
145.733
135.909
134.011
130.138
129.766
128.974
128.526
126.409
119.531
119.140
115.029
77.258
77.000
76.742
41.336
28.326
21.716
X : parts per Million : Carbon13

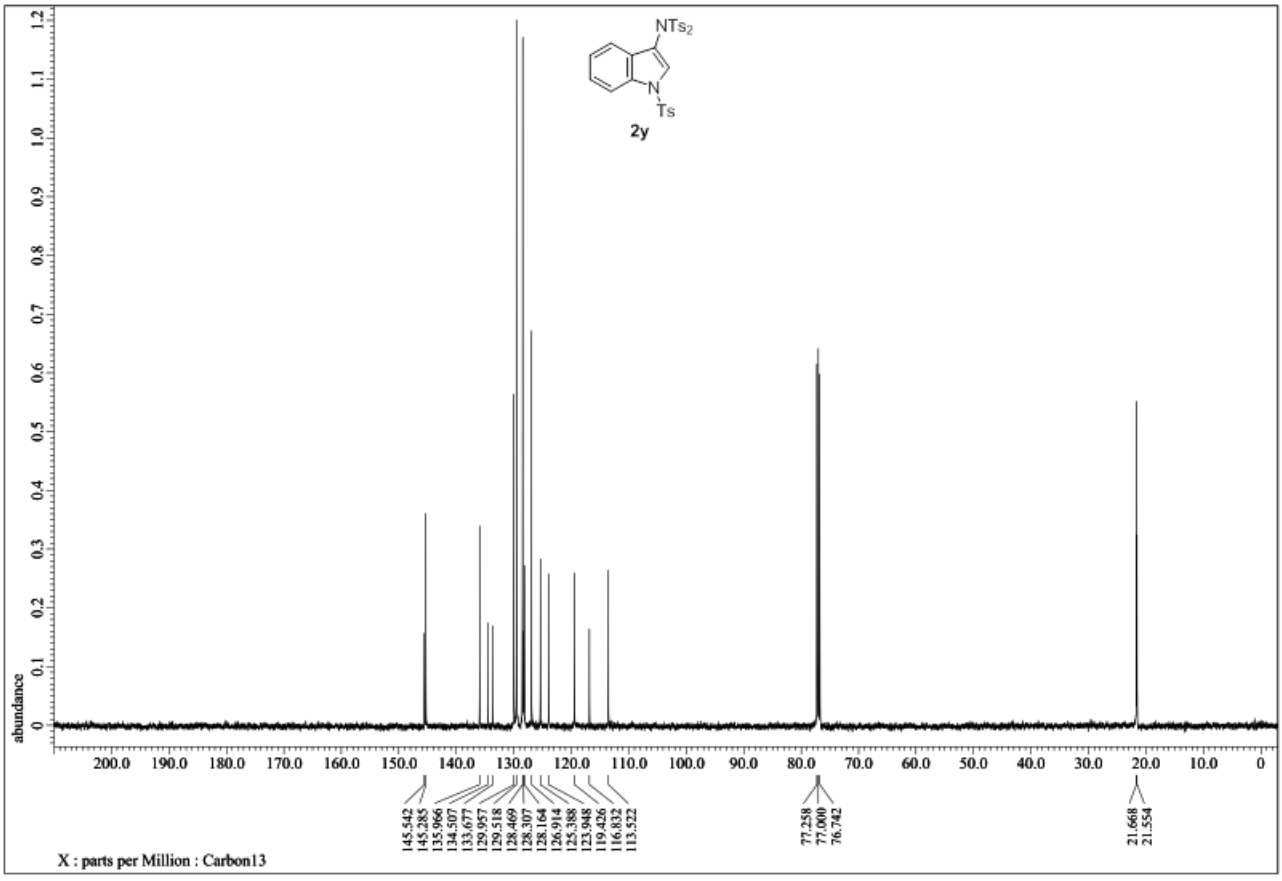

NTs2
N
Piv
2w
abundance
4.01
4.01
2.01
6.02
3.01
9.02
7.857
7.840
7.307
7.290
7.250
7.219
7.203
7.171
7.168
7.159
7.155
7.038
7.026
2.446
1.788
1.339
0.000
X : parts per Million : Proton


NTs2
N
Piv
2w
abundance
185.794
144.989
137.931
136.691
133.820
129.470
128.621
125.789
122.774
121.725
118.749
111.719
110.480
77.248
77.000
76.742
44.446
28.030
21.649
10.823
X : parts per Million : Carbon13


NTs2
N
Bz
2x
abundance
X : parts per Million : Proton


NTs2
N
Bz
2x
abundance
X : parts per Million : Carbon13


NTs2
N
Ts
2y
abundance
X : parts per Million : Proton


NTs2
N
Ts
2y
abundance
X : parts per Million : Carbon13