Direct metallation of thienopyrimidines using a mixed lithium-cadmium base and antitumor activity of functionalized derivatives

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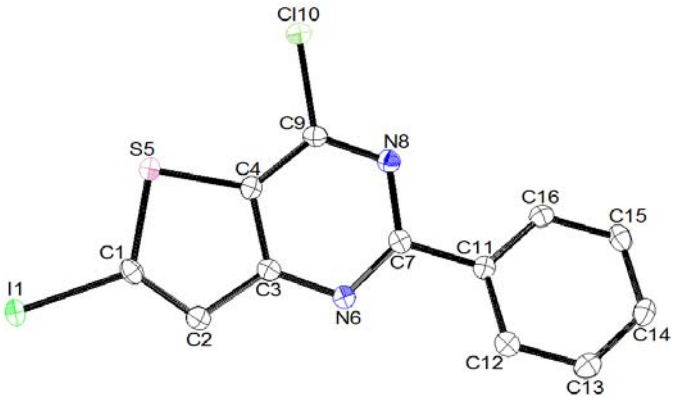
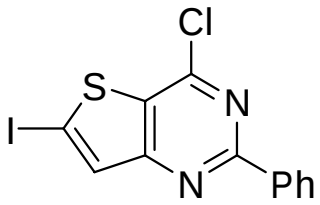
X-ray diffraction analysis

The sample was studied with graphite monochromatized $Mo_{K\alpha}$ radiation (λ = 0.71073 Å). X-ray diffraction data were collected at *T* = 100(2) K using APEXII Bruker-AXS diffractometer. The structure was solved by direct methods using the SIR97 program,[1] and then refined with full-matrix least-square methods based on F^2 (SHELX-97)[2] with the aid of the WINGX program.[3] All non-hydrogen atoms were refined with anisotropic thermal parameters. H atoms were finally included in their calculated positions. Except N-linked hydrogen that was introduced in the structural model through Fourier difference maps analysis, H atoms were finally included in their calculated positions. The molecular diagram was generated by ORTEP-3 (version 1.08).[4]

ORTEP figure





10a

Crystal data

10a: $C_{12}H_6ClIN_2S$; M_r = 372.60; *T* = 100(2) K; λ = 0.71073 Å; orthorombic; space group *P c a b*, *a* = 5.8116(2) Å, *b* = 16.7820(6) Å, *c* = 24.6525(9) Å, *V* = 2404.36(15) $Å^3$; *Z* = 8; D_c = 2.059 g cm^{-3}; μ = 3.035 mm^{-1}; *F*(000) = 1424; crystal size = 0.22 x 0.08 x 0.06 mm; θ range for data collection: 3.52-27.46°, limiting indices: $-7 \le h \le 7$, $-21 \le k \le 19$, $-31 \le l \le 30$; 27353 reflections collected; 2743 reflections unique (R_{int} = 0.0587); $R_1(I>2\sigma(I))$= 0.0252; $wR_2(I>2\sigma(I))$= 0.0495; Crystallographic data were deposited in CSD under CCDC registration number 729714.

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[1] A. Altomare, M. C. Burla, M. Camalli, G. Cascarano, C. Giacovazzo, A. Guagliardi, A. G. G. Moliterni, G. Polidori, R. Spagna, *J. Appl. Crystallogr.*, 1999, **32**, 115.
[2] SHELX97, release 97-2 (Programs for Crystal Structure Analysis). G. M. Sheldrick, Institüt für Anorganische Chemie der Universität, Tammanstrasse 4, D-3400 Göttingen, Germany, 1998.
[3] L. J. Farrugia, *J. Appl. Crystallogr.*, 1999, **32**, 837.
[4] L. J. Farrugia, *J. Appl. Crystallogr.*, 1997, **30**, 565.

NMR data

Methyl 3-benzoylaminothiophene-2-carboxylate. ^{1}H NMR ($CDCl_3$, 300 MHz): δ 3.88 (s, 3H), 7.51 (m, 4H), 7.99 (m, 2H), 8.27 (d, 1H, J = 5.4 Hz), 11.1 (s, 1H); ^{13}C NMR ($CDCl_3$, 75 MHz): δ 52.1, 110.4, 122.3, 127.4, 128.9, 131.9, 132.3, 133.6, 145.2, 164.1, 165.2.

2-Phenyl-3*H*-thieno[3,2-*d*]pyrimidin-4-one. ^{1}H NMR ($(CD_3)_2SO$, 300 MHz): δ 7.48 (d, 1H, J = 5.2 Hz), 7.57 (m, 3H), 8.13 (m, 2H), 8.22 (d, 1H, J = 5.2 Hz), 12.7 (br s, 1H); ^{13}C NMR ($(CD_3)_2SO$, 75 MHz): δ 121.2, 125.4, 127.8, 128.6, 131.3, 132.5, 135.4, 154.3, 157.9, 158.5.

4-Chloro-2-phenylthieno[3,2-*d*]pyrimidine (2). ^{1}H NMR ($CDCl_3$, 300 MHz): δ 7.51 (m, 3H), 7.62 (d, 1H, J = 5.5 Hz), 8.02 (d, 1H, J = 5.2 Hz), 8.52 (m, 2H); ^{13}C NMR ($CDCl_3$, 75 MHz): δ 125.2, 128.3, 128.5, 128.7, 130.9, 136.7, 136.8, 154.9, 161.7, 162.8.

4-Chloro-2-phenylthieno[2,3-*d*]pyrimidine (3). ^{1}H NMR ($CDCl_3$, 300 MHz): δ 7.42 (d, 1H, J = 6.0 Hz), 7.50 (m, 3H), 7.54 (d, 1H, J = 6.0 Hz), 8.52 (m, 2H); ^{13}C NMR ($CDCl_3$, 75 MHz): δ 119.9, 127.4, 127.5, 128.6, 128.7, 131.1, 136.3, 155.1, 160.0, 169.7.

4-Methoxythieno[2,3-*d*]pyrimidine (4). ^{1}H NMR ($CDCl_3$, 300 MHz): δ 4.12 (s, 3H), 7.35 (d, 1H, J = 6.0 Hz), 7.37 (d, 1H, J = 6.0 Hz), 8.64 (s, 1H); ^{13}C NMR ($CDCl_3$, 75 MHz): δ 54.1, 118.6, 119.2, 124.8, 153.4, 164.3, 168.5.

4-Methoxy-2-phenylthieno[2,3-*d*]pyrimidine (6). ^{1}H NMR ($CDCl_3$, 300 MHz): δ 4.22 (s, 3H), 7.31 (d, 1H, J = 6.0 Hz), 7.35 (d, 1H, J = 6.0 Hz), 7.50 (m, 3H), 8.55 (m, 2H); ^{13}C NMR ($CDCl_3$, 75 MHz): δ 53.6, 117.1, 118.5, 124.1, 128.3, 128.4, 130.3, 137.5, 159.7, 163.8, 169.4.

4-(Pyrazol-1-yl)thieno[2,3-*d*]pyrimidine (5). ^{1}H NMR ($CDCl_3$, 300 MHz): δ 7.26 (s, 1H), 8.26 (d, 1H, J = 6.0 Hz), 8.59 (s, 1H), 9.15 (d, 1H, J = 6.0 Hz), 9.50 (d, 1H, J = 2.7 Hz), 9.57 (s, 1H); ^{13}C NMR ($CDCl_3$, 75 MHz): δ 108.7, 119.2, 123.4, 126.4, 129.0, 144.3, 151.0, 152.4, 171.7.

4-(Morpholin-4-yl)-2-phenylthieno[2,3-*d*]pyrimidine (7). ^{1}H NMR ($CDCl_3$, 300 MHz): δ 3.88 (m, 4H), 3.97 (m, 4H), 7.24 (d, 1H, J = 6.0 Hz), 7.31 (d, 1H, J = 6.0 Hz), 7.45-7.48 (m, 3H), 8.46 (m, 2H); ^{13}C NMR ($CDCl_3$, 75 MHz): δ 47.3, 66.7, 114.9, 120.2, 121.9, 128.2, 128.3, 130.1, 138.0, 158.7, 158.8, 170.7.

4-Chloro-6-iodothieno[2,3-*d*]pyrimidine (8). ^{1}H NMR ($CDCl_3$, 300 MHz): δ 7.70 (s, 1H), 7.80 (s, 1H); ^{13}C NMR ($CDCl_3$, 75 MHz): δ 82.2, 129.4, 131.3, 152.7, 152.8, 172.8.

6-Iodo-4-methoxythieno[2,3-*d*]pyrimidine (9). ^{1}H NMR ($CDCl_3$, 300 MHz): δ 4.10 (s, 3H), 7.60 (s, 1H), 8.56 (s, 1H); ^{13}C NMR ($CDCl_3$, 75 MHz): δ 54.4, 77.2, 121.1, 128.5, 153.5, 162.4, 172.5.

4-Chloro-6-iodo-2-phenylthieno[3,2-*d*]pyrimidine (10a). ^{1}H NMR ($CDCl_3$, 300 MHz): δ 7.49 (m, 3H), 7.83 (s, 1H), 8.47 (m, 2H); ^{13}C NMR ($CDCl_3$, 75 MHz): δ 92.2, 128.5, 128.7, 131.1, 133.3, 134.9, 136.3, 152.6, 162.0, 163.0.

4-Chloro-6-iodo-2-phenylthieno[2,3-*d*]pyrimidine (11). ^{1}H NMR ($CDCl_3$, 300 MHz): δ 7.50 (m, 3H), 7.68 (s, 1H), 8.48 (m, 2H); ^{13}C NMR ($CDCl_3$, 75 MHz): δ 80.9, 128.6, 128.7, 129.2, 129.5, 131.3, 135.9, 152.8, 159.9, 173.5.

4-Chloro-6,7-diiodo-2-phenylthieno[3,2-*d*]pyrimidine (10b). ^{1}H NMR ($CDCl_3$, 300 MHz): δ 7.48 (m, 3H), 8.54 (m, 2H); ^{13}C NMR ($CDCl_3$, 75 MHz): δ 98.7, 101.3, 128.7, 128.8, 131.4, 132.8, 135.9, 153.1, 162.0, 162.7.

6-Iodo-4-methoxy-2-phenylthieno[2,3-*d*]pyrimidine (12). ^{1}H NMR ($CDCl_3$, 300 MHz): δ 4.21 (s, 3H), 7.48 (m, 3H), 7.60 (s, 1H), 8.49 (m, 2H); ^{13}C NMR ($CDCl_3$, 75 MHz): δ 53.9, 76.1, 119.0, 128.4, 128.4, 128.5, 130.6, 137.2, 159.7, 162.1, 173.3.

6-Iodo-4-(morpholin-4-yl)-2-phenylthieno[2,3-*d*]pyrimidine (13). ^{1}H NMR ($CDCl_3$, 300 MHz): δ 3.89 (t, 4H, J = 4.5 Hz), 4.00 (t, 4H, J = 4.3 Hz), 7.48 (m, 3H), 7.55 (s, 1H), 8.44 (m, 2H); ^{13}C NMR ($CDCl_3$, 75 MHz): δ 46.5, 65.9, 76.1, 116.2, 127.7, 128.4, 130.3, 130.6, 137.0, 156.3, 157.3, 173.4.

4-(Morpholin-4-yl)-2-phenyl-6-(pyridin-2-yl)-thieno[2,3-*d*]pyrimidine (14). ^{1}H NMR ($CDCl_3$, 300 MHz): δ 3.93 (m, 4H), 4.06 (m, 4H), 7.23 (ddd, 1H, J = 2.0, 4.9 and 6.0 Hz), 7.48 (m, 3H), 7.73 (m, 2H), 7.88 (s, 1H), 8.47 (m, 2H), 8.64 (dt, 1H, J = 1.2 and 4.6 Hz); ^{13}C NMR ($CDCl_3$, 75 MHz): δ 47.3, 66.8, 116.2, 117.5, 119.6, 122.7, 128.3, 128.3, 130.2, 136.8, 138.0, 139.3, 149.8, 151.8, 158.7, 159.3, 171.2.

(*S*)-3-Phenyl-2-(2-phenylthieno[3,2-*d*]pyrimidin-4-ylamino)propanoic acid (15). ^{1}H NMR ($(CD_3)_2SO$, 300 MHz): δ 3.34 (AB part of an ABX system, 2H, J_{AX} = 3.7 Hz, J_{BX} = 8.9 Hz and J_{AB} = 13.6 Hz), 4.99 (m, 1H), 7.06 (t, 1H, J = 7.0 Hz), 7.15 (t, 2H, J = 7.2 Hz), 7.29 (d, 2H, J = 7.2 Hz), 7.44 (m, 4H), 7.76 (br s, 1H), 8.06 (d, 1H, J = 5.3 Hz), 8.39 (m, 2H); ^{13}C NMR ($(CD_3)_2SO$, 75 MHz): δ 37.0, 56.3, 113.4, 124.6, 125.8, 127.7, 127.8, 128.1, 129.2, 129.7, 132.9, 138.4, 139.1, 156.5, 159.5, 160.2, 173.7.

(*S*)-2-(6-Iodo-2-phenylthieno[3,2-*d*]pyrimidin-4-ylamino)-3-phenylpropanoic acid (16). ^{1}H NMR ($(CD_3)_2SO$, 300 MHz): δ 3.26 (AB part of an ABX system, 2H, J_{AX} = 4.0 Hz, J_{BX} = 9.9 Hz and J_{AB} = 13.6 Hz), 4.90 (m, 1H), 7.10 (t, 1H, J = 7.2 Hz), 7.18 (t, 2H, J =7.3 Hz), 7.31 (d, 2H, J = 7.2 Hz), 7.43 (m, 3H), 7.71 (s, 1H), 8.10 (br s, 1H), 8.33 (m, 2H); ^{13}C NMR ($(CD_3)_2SO$, 75 MHz): δ 36.7, 56.3, 89.3, 118.3, 126.0, 127.7, 128.0, 128.2, 129.1, 129.9, 133.9, 137.9, 138.8, 155.0, 159.7, 160.9, 174.4.

(*S*)-2-(6,7-Diiodo-2-phenylthieno[3,2-*d*]pyrimidin-4-ylamino)-3-phenylpropanoic acid (17). ^{1}H NMR ($(CD_3)_2SO$, 300 MHz): δ 3.20 (m, 2H), 4.85 (m, 1H), 7.07 (t, 1H, J = 7.2 Hz), 7.15 (t, 2H, J =7.3

Hz), 7.26 (br d, 2H, J = 7 Hz), 7.47 (m, 3H), 7.96 (br s, 1H), 8.40 (m, 2H); ^{13}C NMR ($(CD_3)_2SO$, 75 MHz): δ 36.7, 56.3, 99.9, 103.7, 125.8, 127.8, 127.9, 128.2, 129.1, 129.4, 130.2, 137.7, 139.1, 159.6, 160.6, 173.9.

(*S*)-3-Phenyl-2-(2-phenylthieno[2,3-*d*]pyrimidin-4-ylamino)propanoic acid (18). ^{1}H NMR ($(CD_3)_2SO$, 300 MHz): δ 3.29 (AB part of an ABX system, 2H, J_{AX} = 3.8 Hz, J_{BX} = 10 Hz and J_{AB} = 13.4 Hz), 3.71 (br s, 1H), 5.00 (m, 1H), 7.08 (t, 1H, J = 7.1 Hz), 7.17 (t, 2H, J =7.3 Hz), 7.33 (d, 2H, J = 7.3 HZ), 7.43 (m, 3H), 7.49 (d, 1H, J = 5.9 Hz), 7.71 (d, 1H, J = 5.9 Hz), 8.17 (d, 1H, J = 7.0 Hz), 8.36 (m, 2H); ^{13}C NMR ($(CD_3)_2SO$, 75 MHz): δ 37.1, 56.3, 114.8, 119.7, 122.3, 125.9, 127.7, 127.9, 128.2, 129.1, 129.9, 137.9, 139.0, 156.5, 158.5, 166.7.

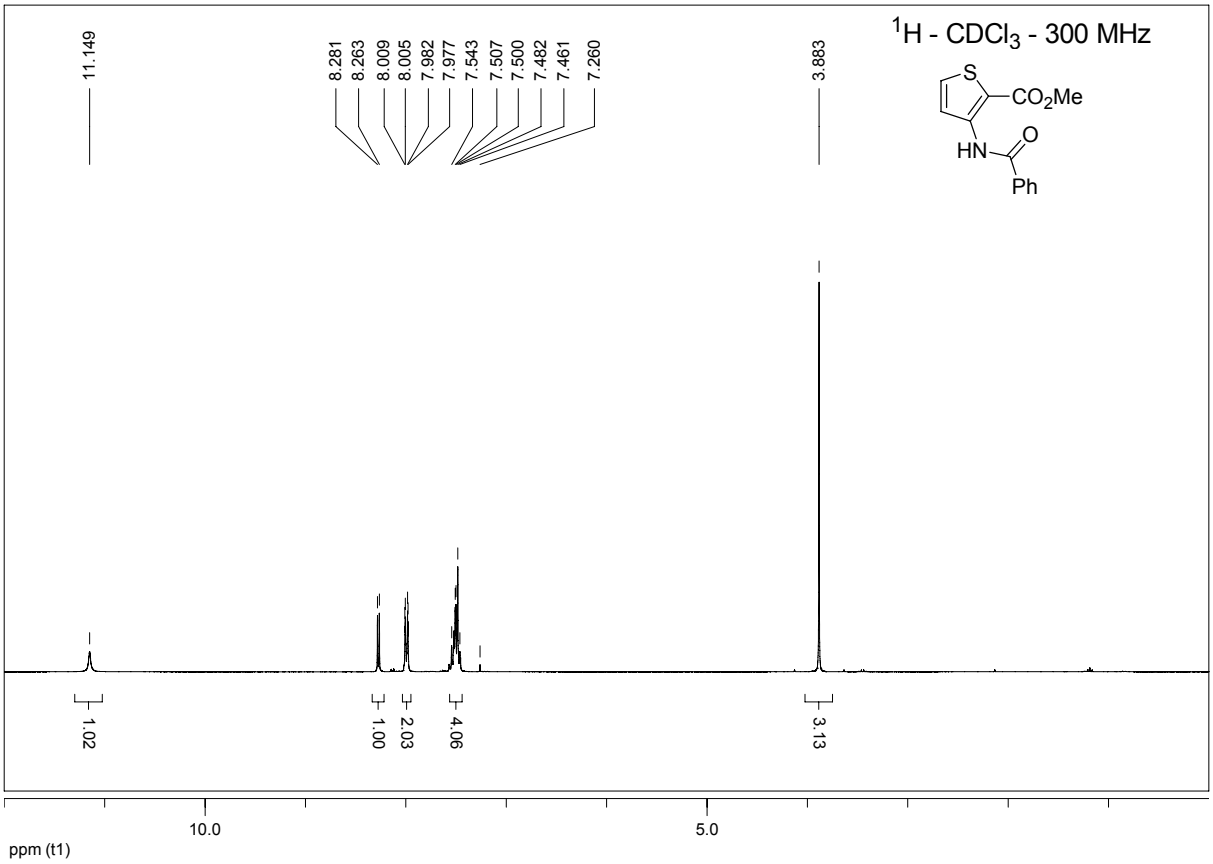
(*S*)-2-(6-Iodo-2-phenylthieno[2,3-*d*]pyrimidin-4-ylamino)-3-phenylpropanoic acid (19). ^{1}H NMR ($(CD_3)_2SO$, 300 MHz): δ 3.23 (AB part of an ABX system, 2H, J_{AX} = 4.5 Hz, J_{BX} = 10.3 Hz and J_{AB} = 13.7 Hz), 4.92 (m, 1H), 7.13 (t, 1H, J = 7.2 Hz), 7.23 (t, 2H, J =7.3 Hz), 7.35 (br d, 2H, J = 7.1 Hz), 7.43 (m, 3H), 8.06 (s, 1H), 8.30 (m, 3H); ^{13}C NMR ($(CD_3)_2SO$, 75 MHz): δ 36.6, 55.9, 75.8, 116.7, 126.2, 127.8, 128.1, 128.3, 129.0, 129.1, 130.2, 137.4, 138.4, 154.9, 158.5, 170.7, 173.8.

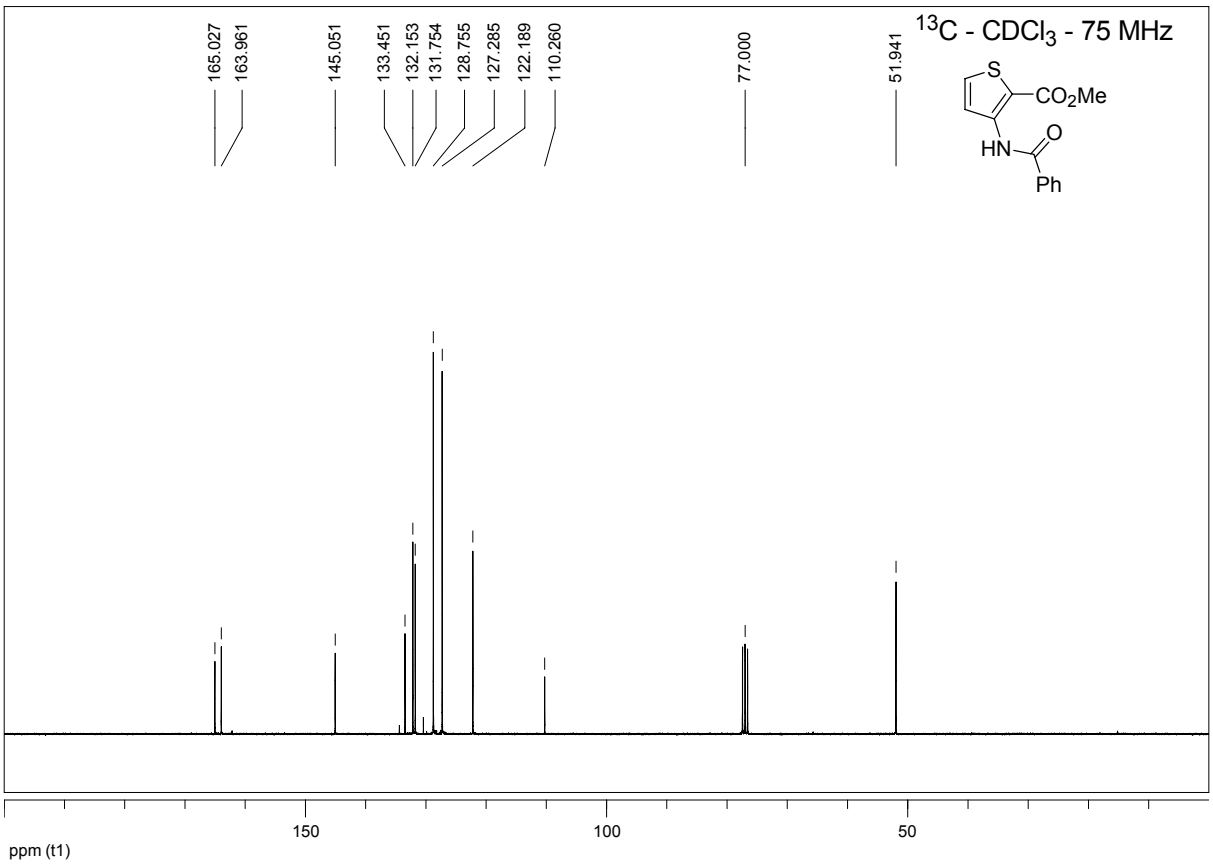
(*S*)-2-[(6-Iodo-2-phenylthieno[2,3-*d*]pyrimidin-4-yl)(methyl)amino]-3-phenylpropanoic acid (20). ^{1}H NMR ($CDCl_3$, 300 MHz): δ 3.12 (s, 3H), 3.40 (AB part of an ABX system, 2H, J_{AX} = 4.4 Hz, J_{BX} = 9.9 Hz and J_{AB} = 14 Hz), 5.11 (br s, 1H), 7.14 (m, 5H), 7.39 (m, 4H), 8.28 (br d, 2H); ^{13}C NMR ($CDCl_3$, 75 MHz): δ 29.7, 34.6, 73.2, 116.6, 126.8, 128.2, 128.4, 128.6, 128.9, 130.4, 130.7, 136.9, 137.0, 137.4, 156.0, 158.1, 174.5.

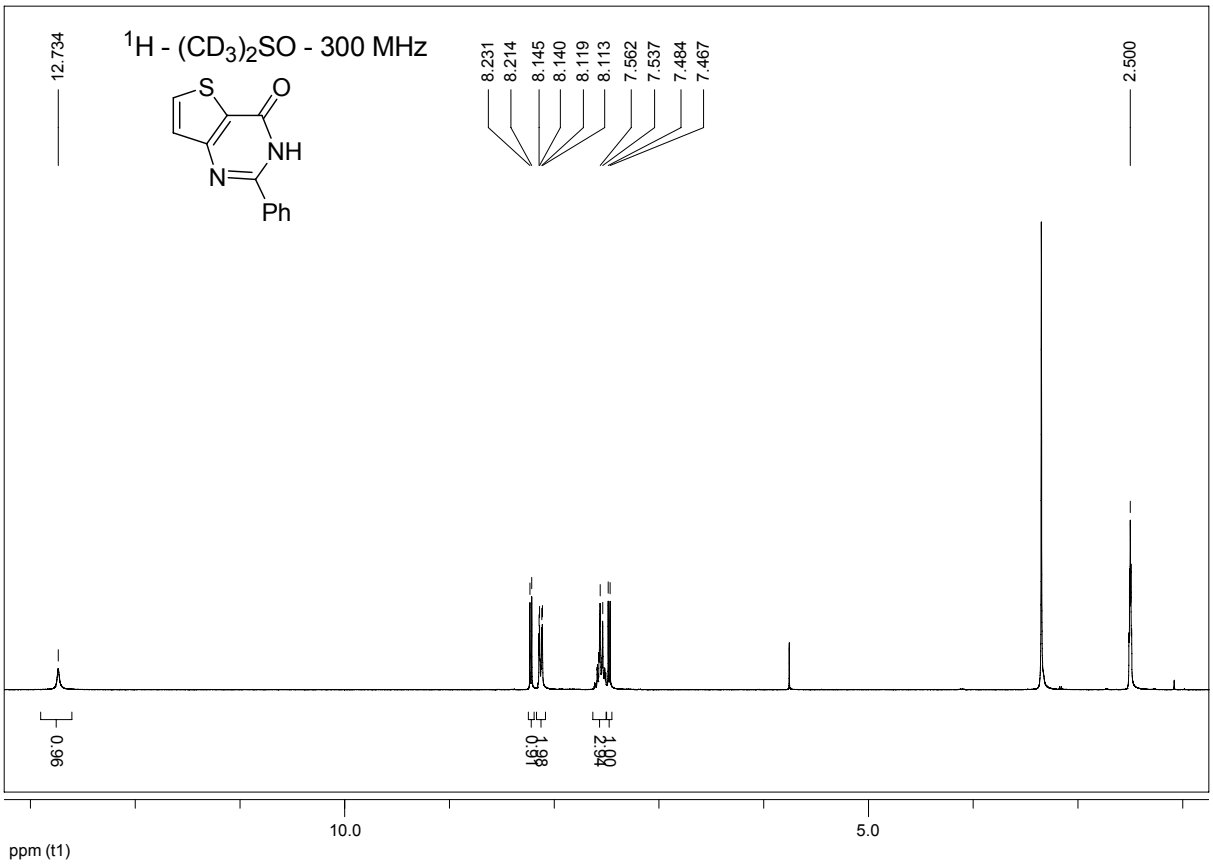
(*S*)-2-(6-Iodo-2-phenylthieno[2,3-*d*]pyrimidin-4-ylamino)-3-phenylpropan-1-ol (21). ^{1}H NMR ($(CD_3)_2SO$, 300 MHz): δ 2.98 (AB part of an ABX system, 2H, J_{AX} = 5.4 Hz, J_{BX} = 8.5 Hz and J_{AB} = 13.7 Hz), 3.60 (m, 2H), 4.66 (m, 1H), 4.93 (t, 1H, J = 5.6 Hz), 7.11 (tt, 1H, J = 7.2 Hz and 1.3 Hz), 7.23 (t, 2H, 7.4 Hz), 7.32 (m, 2H), 7.47 (m, 3H), 7.77 (d, 1H, J = 8.1 Hz), 8.07 (s, 1H), 8.33 (m, 2H); ^{13}C NMR ($(CD_3)_2SO$, 75 MHz): δ 36.6, 54.0, 62.2, 75.0, 116.7, 125.9, 127.7, 127.8, 128.1, 128.3, 128.7, 129.1, 129.3, 130.1, 130.8, 131.7, 137.7, 139.3, 155.1, 158.7, 170.5.

(*S*)-1-(6-Iodo-2-phenylthieno[2,3-*d*]pyrimidin-4-yl)pyrrolidine-2-carboxylic acid (22). ^{1}H NMR ($(CD_3)_2SO$, 300 MHz): δ 1.86-2.27 (m, 4H), 3.95 (m, 2H), 4.69 (m, 1H), 7.41 (m, 3H), 7.80 (s, 1H), 8.31 (m, 2H); ^{13}C NMR ($(CD_3)_2SO$, 75 MHz): δ 21.1, 24.7, 24.8, 28.4, 31.1, 49.3, 61.5, 63.1, 74.7, 116.0, 127.8, 128.3, 130.3, 130.6, 137.2, 157.6, 174.0.

Copies of NMR spectra



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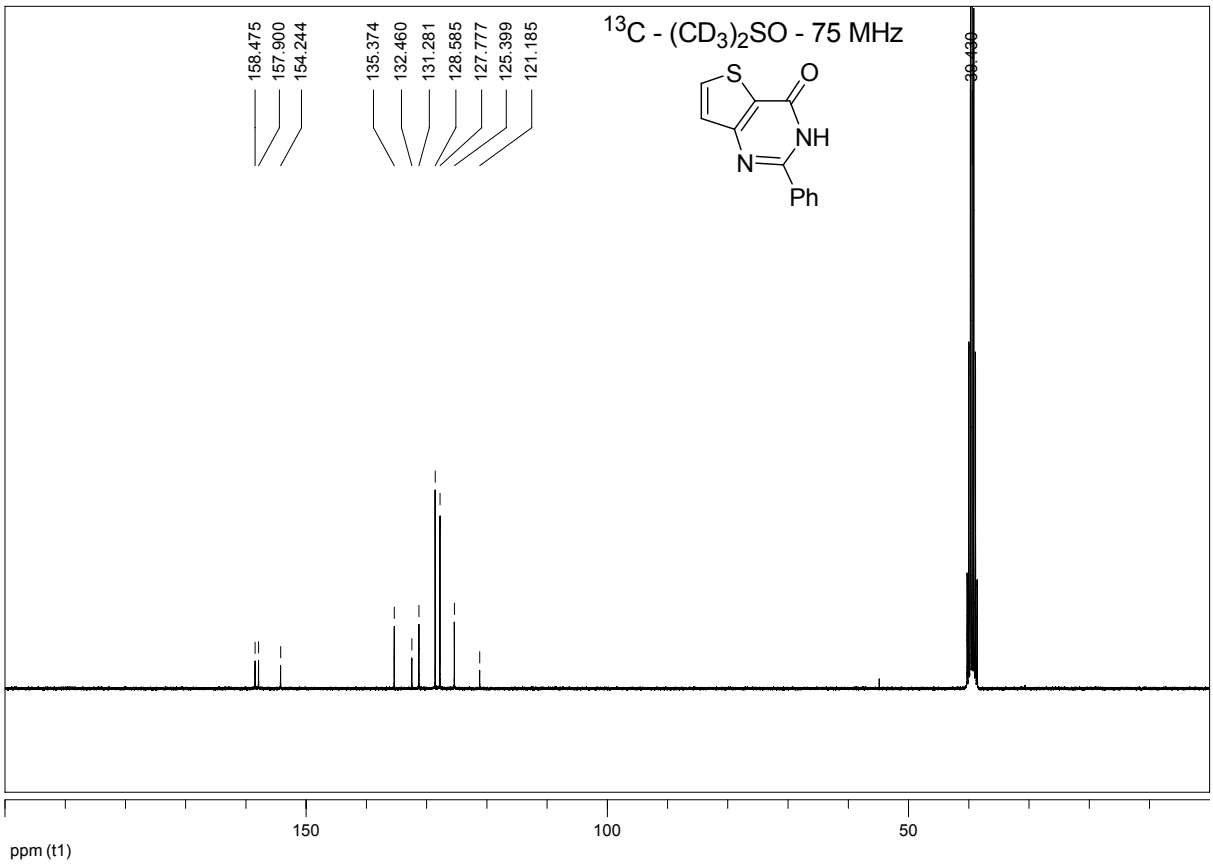
ppm (t1)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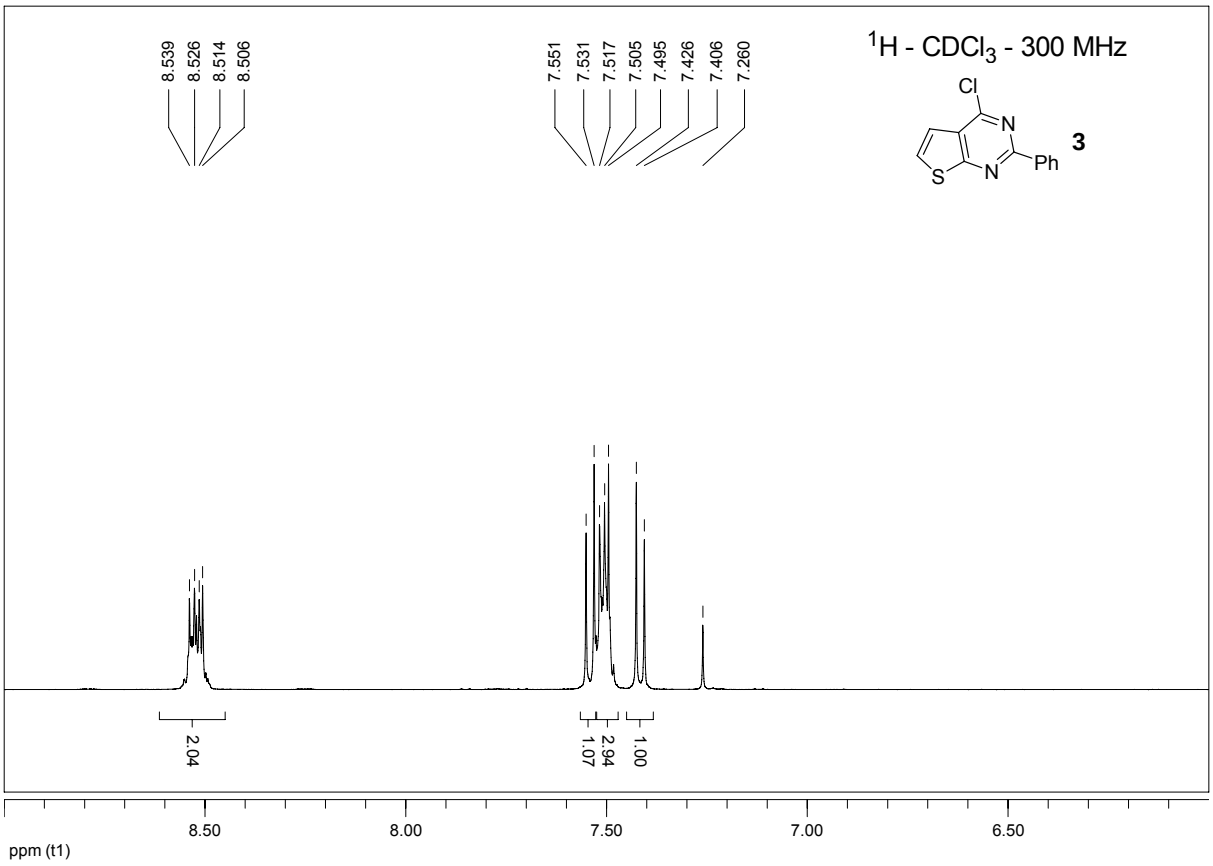
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Cl
N
Ph
S
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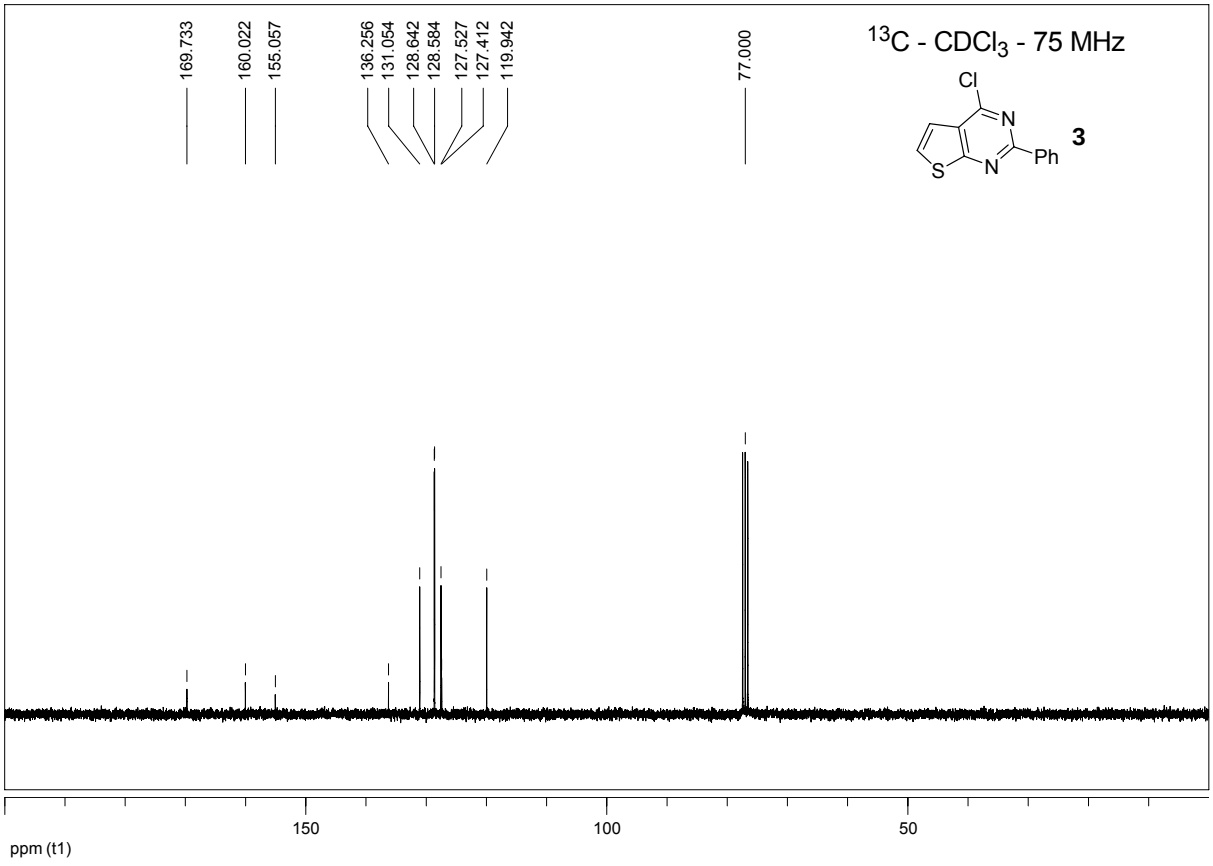
ppm (t1)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Ph
S
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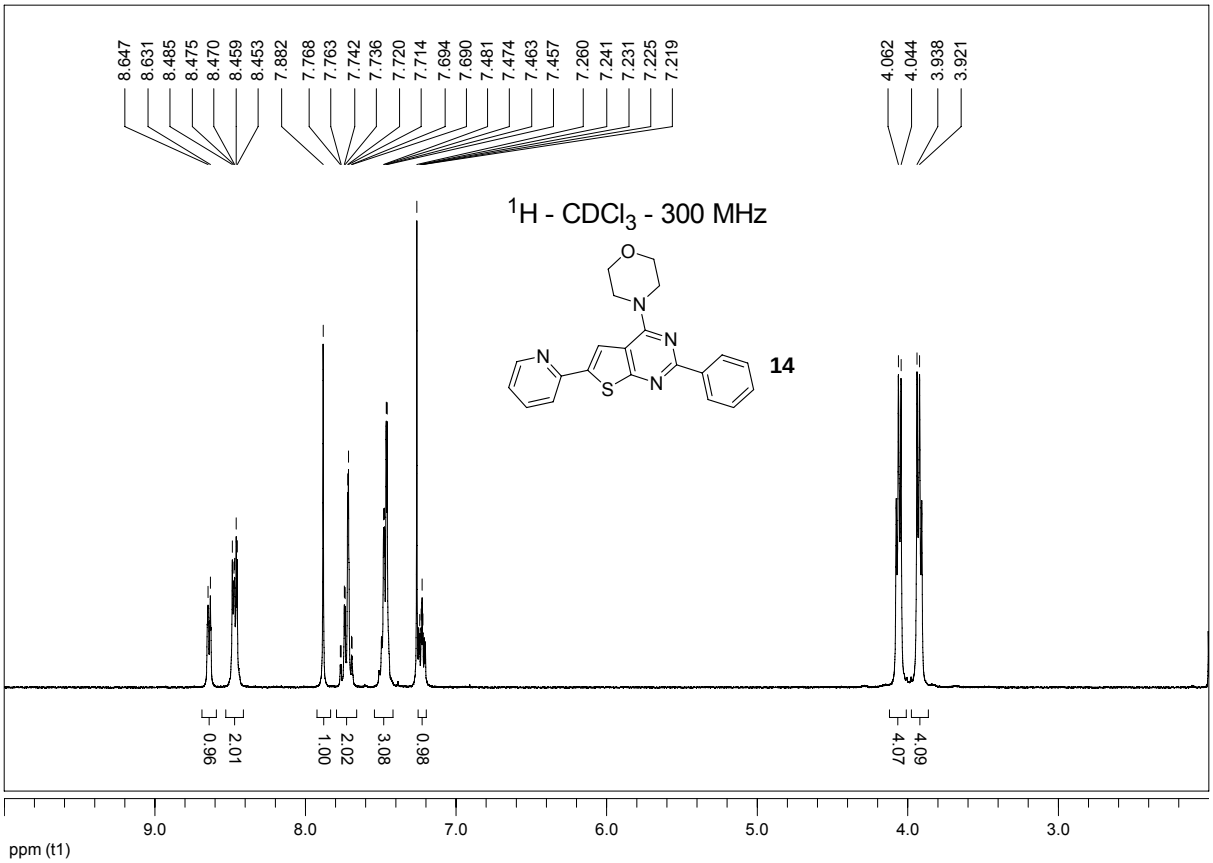
ppm (t1)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7.714
7.694
7.690
7.481
7.474
7.463
7.457
7.260
7.241
7.231
7.225
7.219
4.062
4.044
3.938
3.921
^{1}H - CDCl$_3$ - 300 MHz
14
0.96
2.01
1.00
2.02
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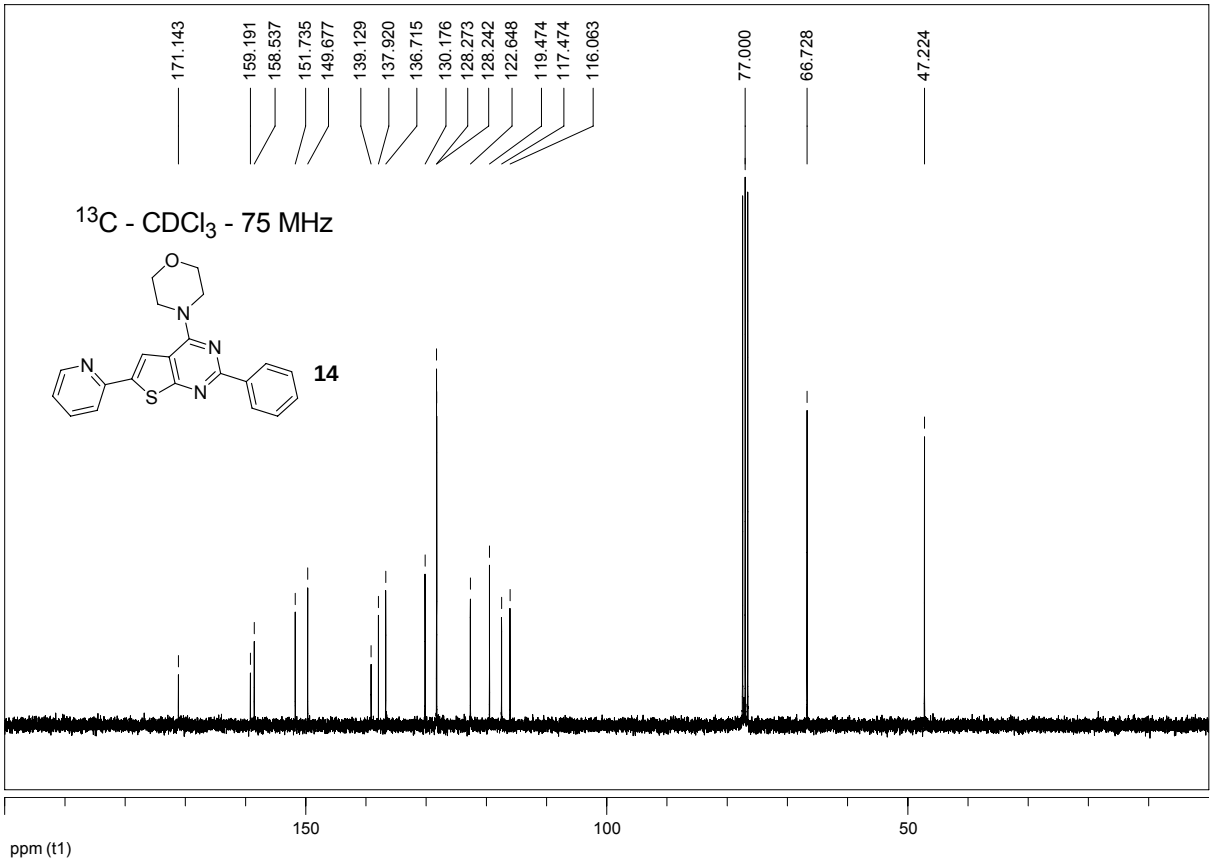
ppm (t1)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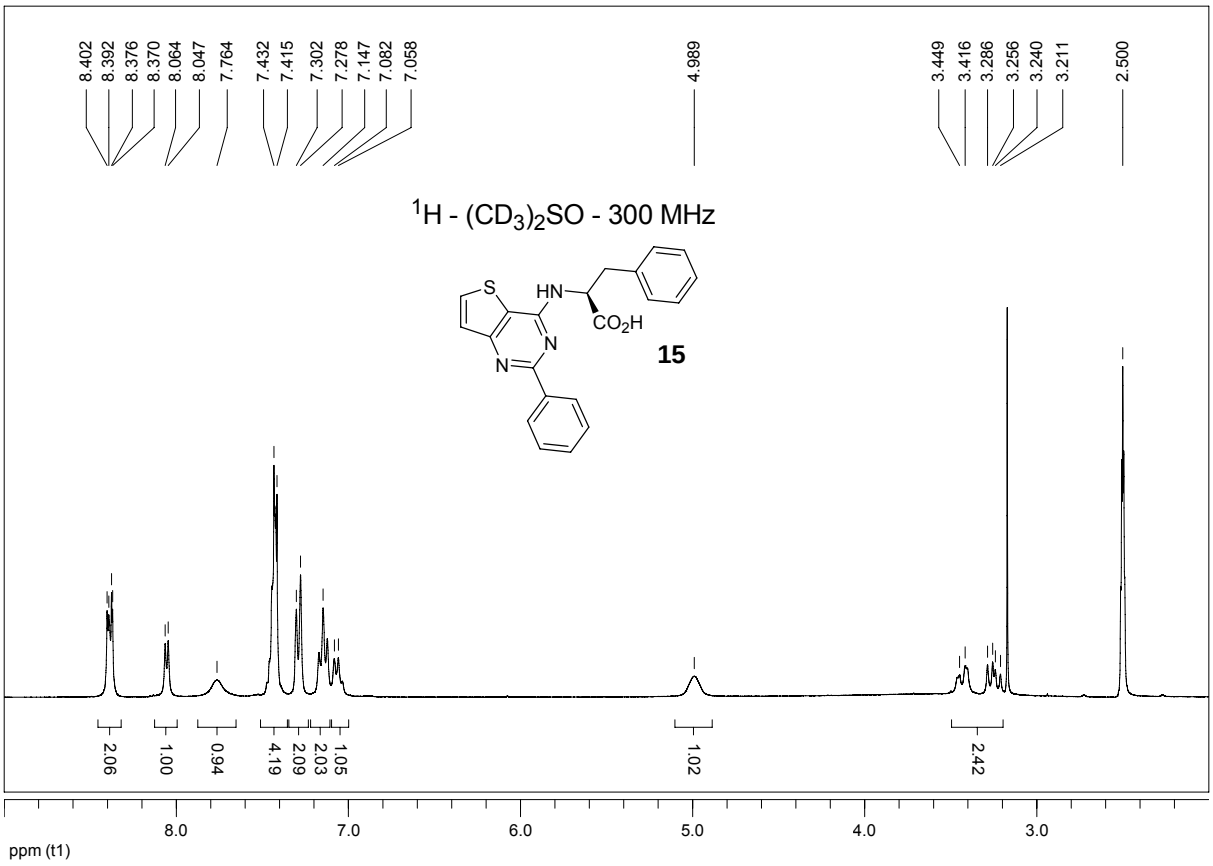
50
ppm (t1)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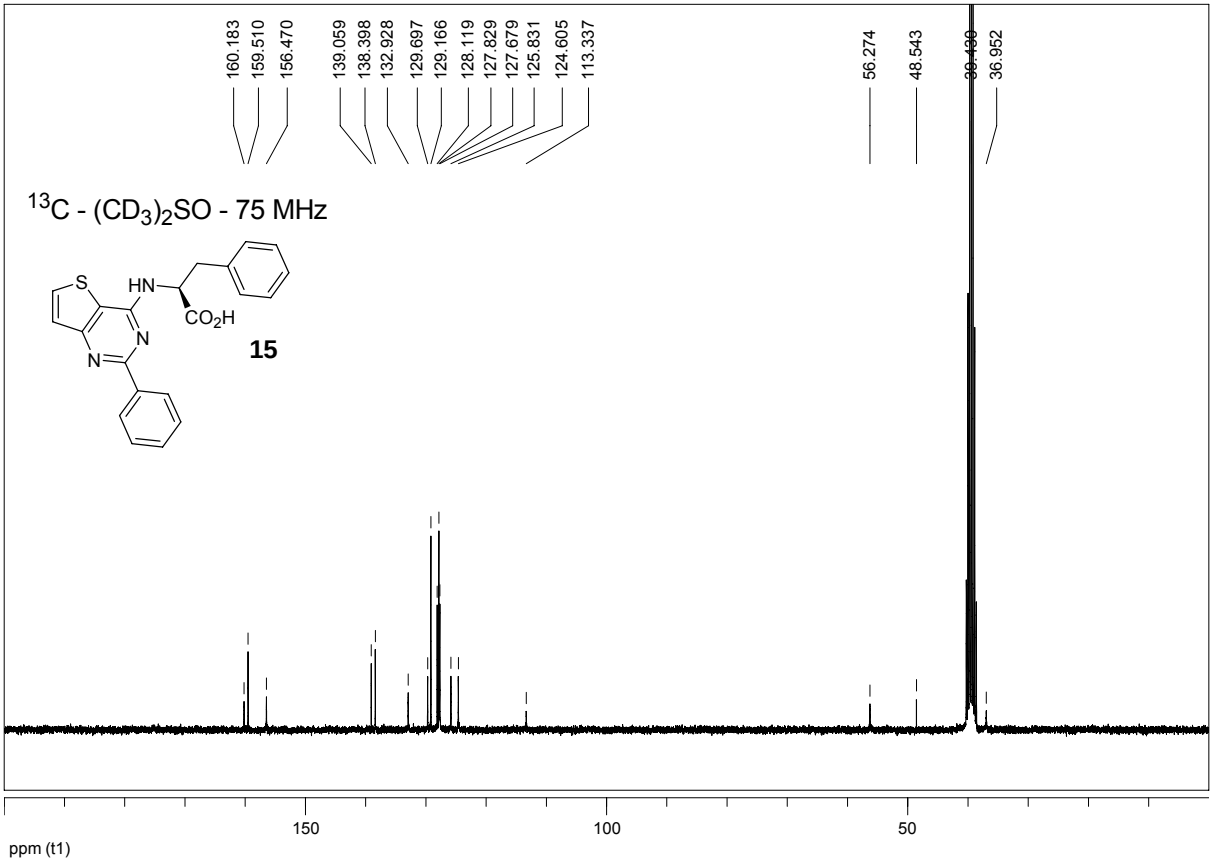
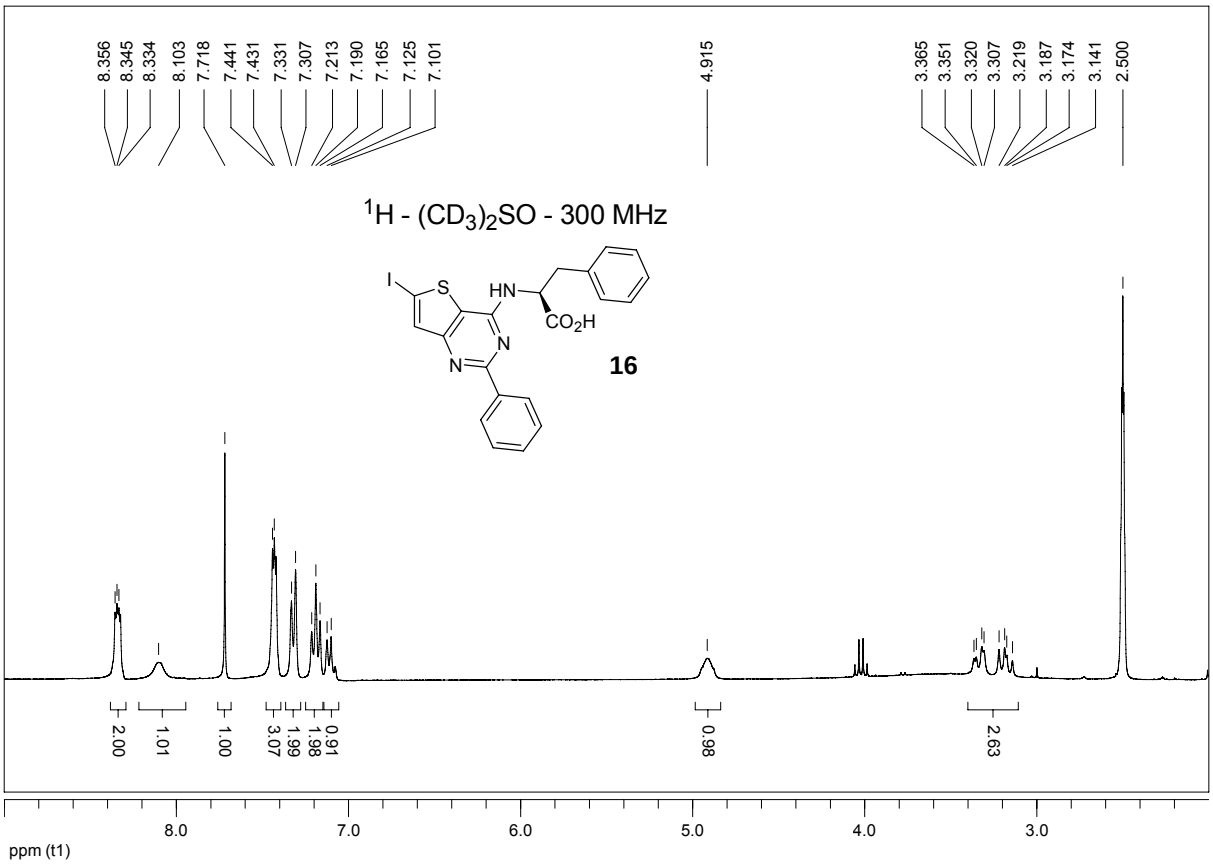
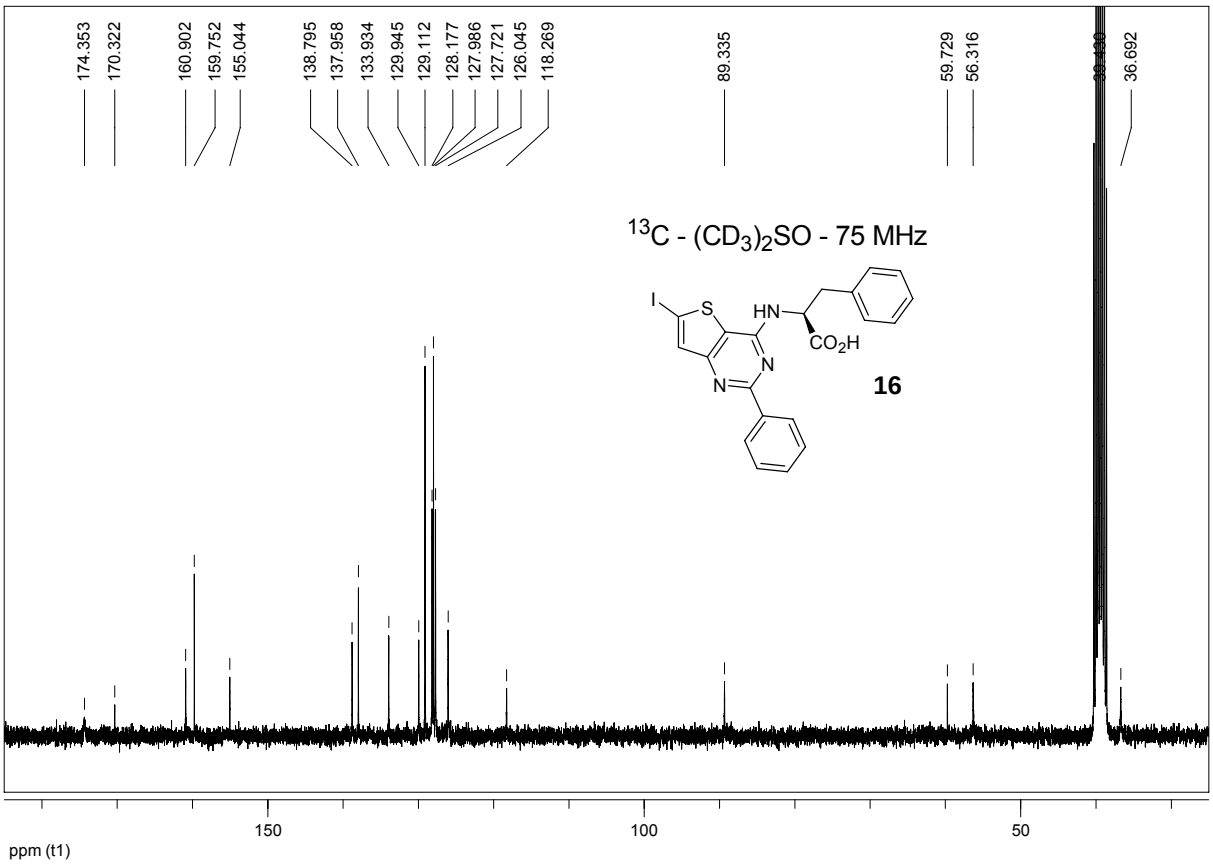
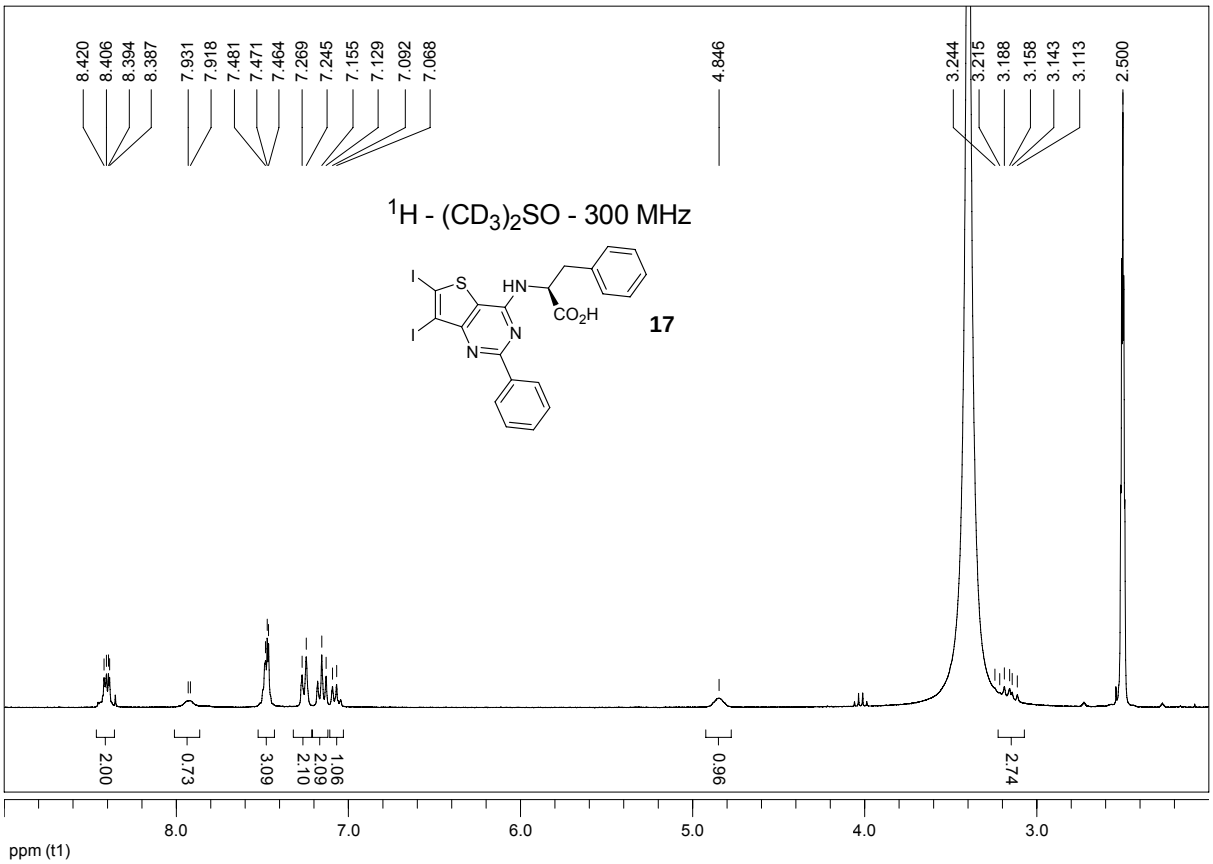
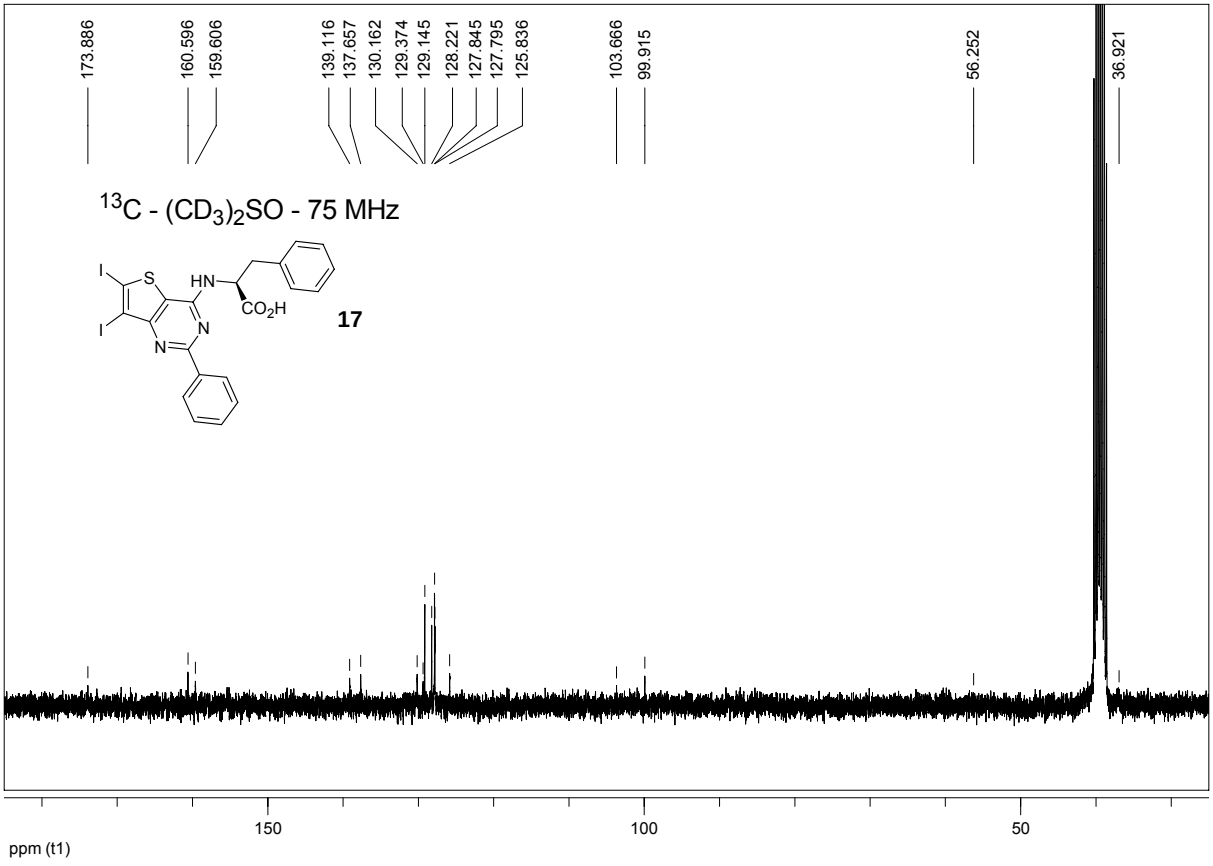
3.240
3.211
2.500
1H - (CD3)2SO - 300 MHz
S
HN
CO2H
N
N
15
2.06
1.00
0.94
4.19
2.09
2.03
1.05
1.02
2.42
8.0
7.0
6.0
5.0
4.0
3.0
ppm (t1)

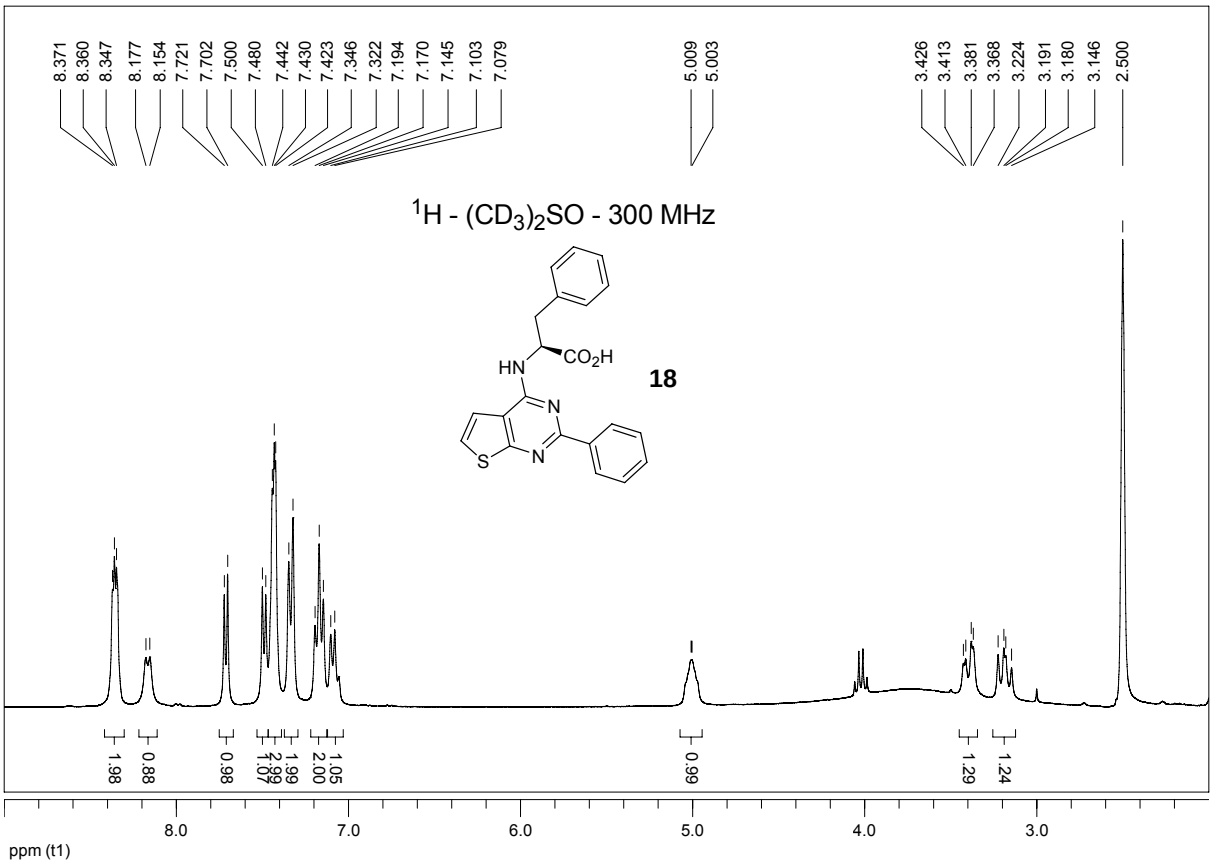

160.183
159.510
156.470
139.059
138.398
132.928
129.697
129.166
128.119
127.829
127.679
125.831
124.605
113.337
56.274
48.543
30.490
36.952
13C - (CD3)2SO - 75 MHz
S
HN
CO2H
N
N
15
150
100
50
ppm (t1)

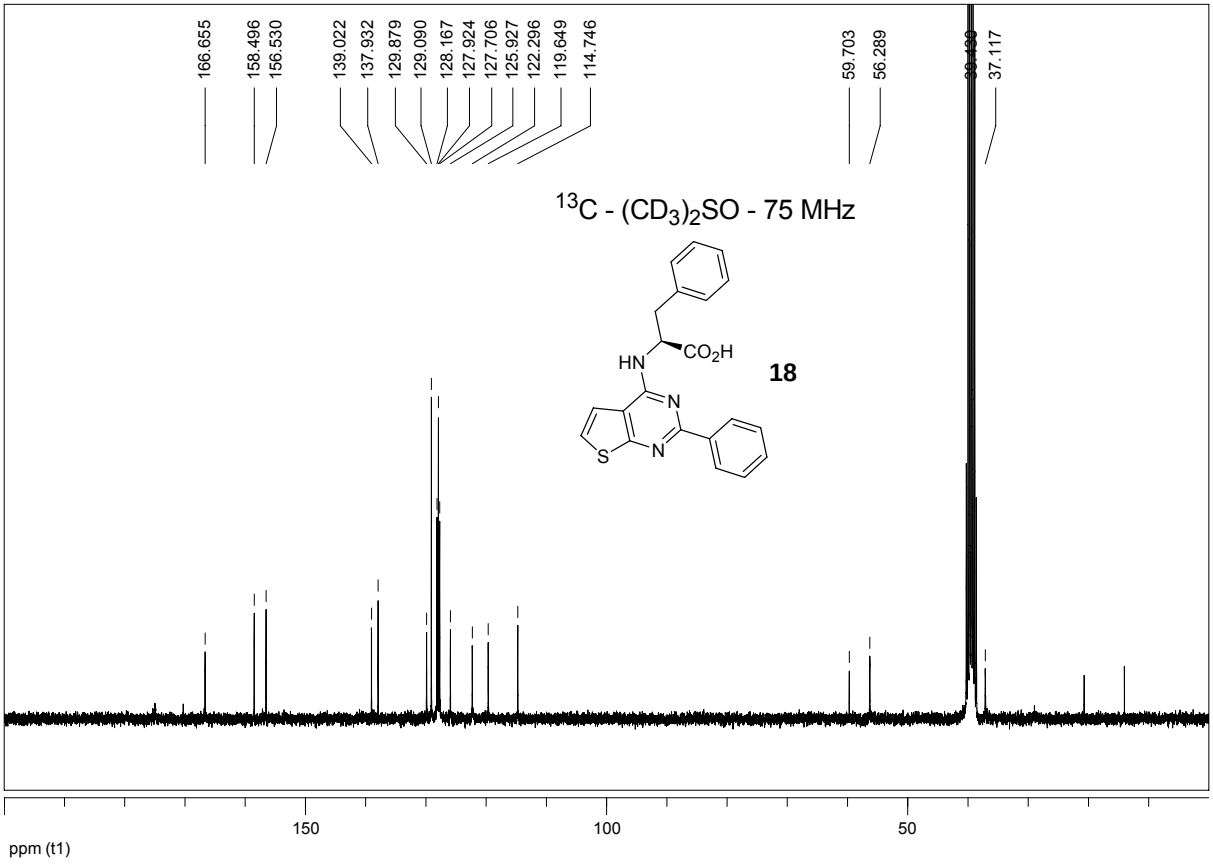

8.356
8.345
8.334
8.103
7.718
7.441
7.431
7.331
7.307
7.213
7.190
7.165
7.125
7.101
4.915
3.365
3.351
3.320
3.307
3.219
3.187
3.174
3.141
2.500
^{1}H - $(CD_3)_2SO$ - 300 MHz
I
S
HN
CO_2H
N
N
16
2.00
1.01
1.00
3.07
1.99
1.98
0.91
0.98
2.63
8.0
7.0
6.0
5.0
4.0
3.0
ppm (t1)

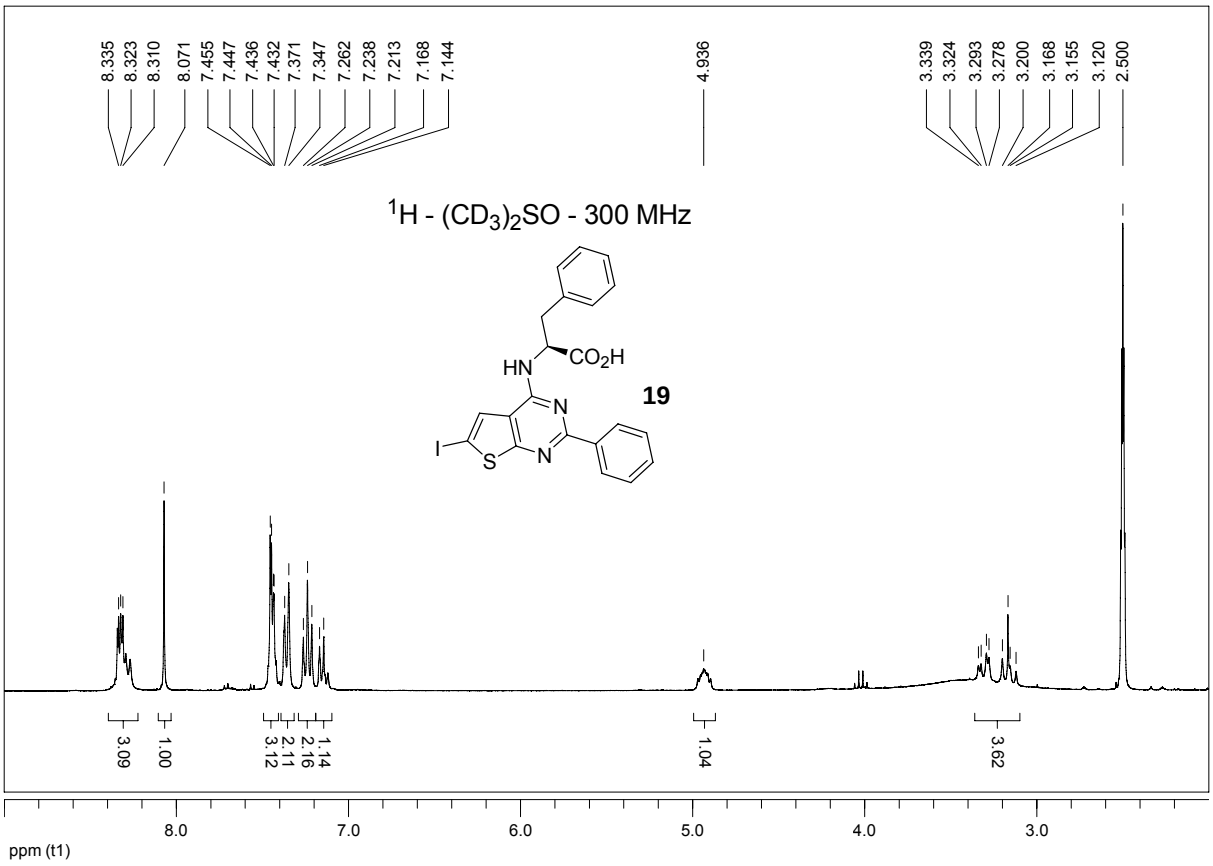

174.353
170.322
160.902
159.752
155.044
138.795
137.958
133.934
129.945
129.112
128.177
127.986
127.721
126.045
118.269
89.335
59.729
56.316
39.430
36.692
^{13}C - $(CD_3)_2SO$ - 75 MHz
I
S
HN
CO_2H
N
N
16
150
100
50
ppm (t1)

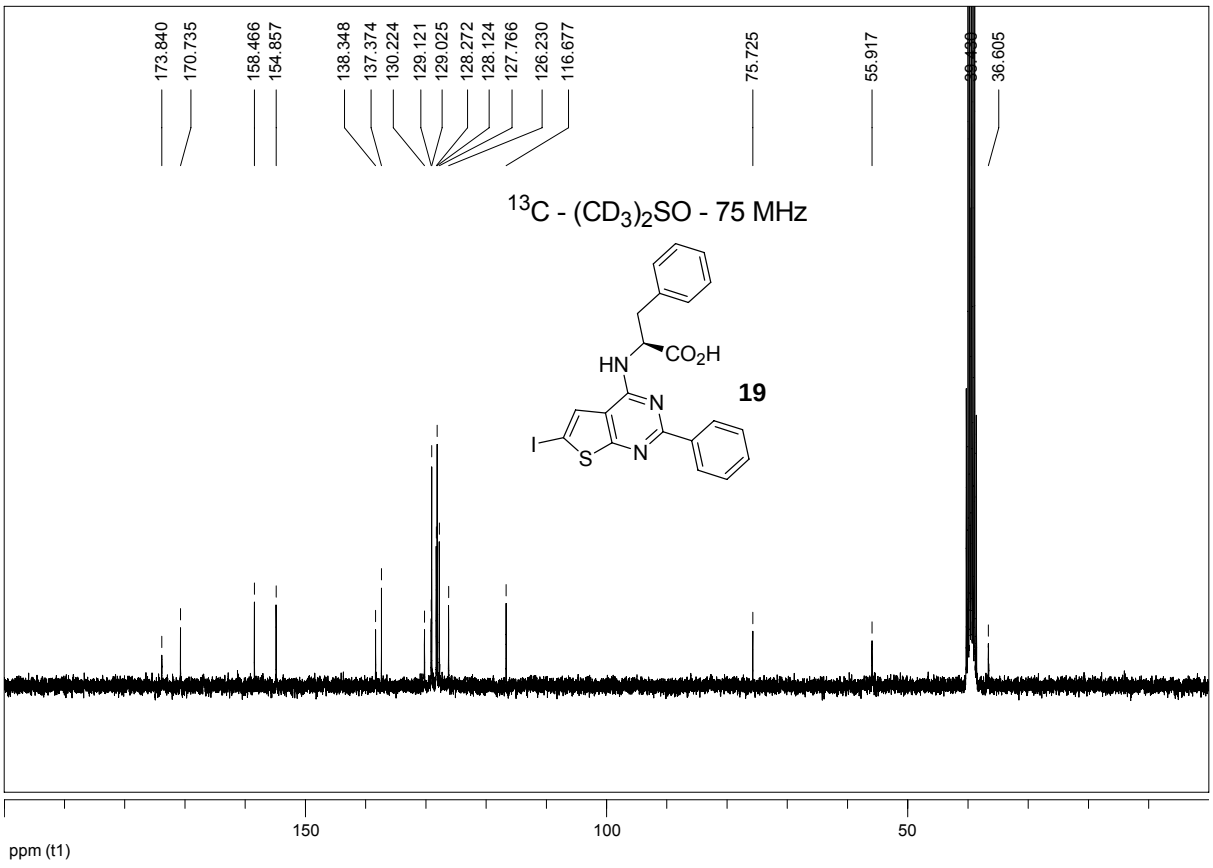

8.420
8.406
8.394
8.387
7.931
7.918
7.481
7.471
7.464
7.269
7.245
7.155
7.129
7.092
7.068
4.846
3.244
3.215
3.188
3.158
3.143
3.113
2.500
^{1}H - $(CD_3)_2SO$ - 300 MHz
17
2.00
0.73
3.09
2.10
2.09
1.06
0.96
2.74
8.0
7.0
6.0
5.0
4.0
3.0
ppm (t1)

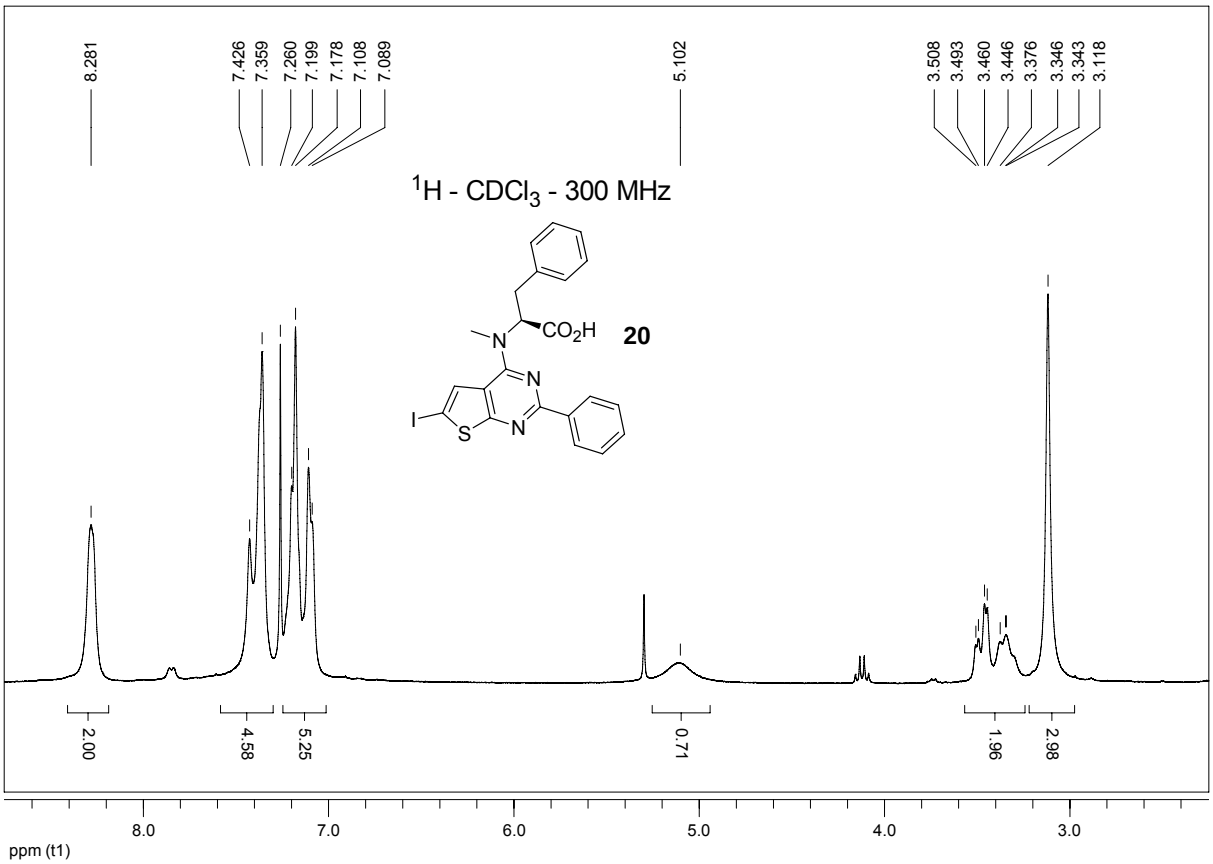

173.886
160.596
159.606
139.116
137.657
130.162
129.374
129.145
128.221
127.845
127.795
125.836
103.666
99.915
56.252
36.921
^{13}C - $(CD_3)_2SO$ - 75 MHz
17
150
100
50
ppm (t1)

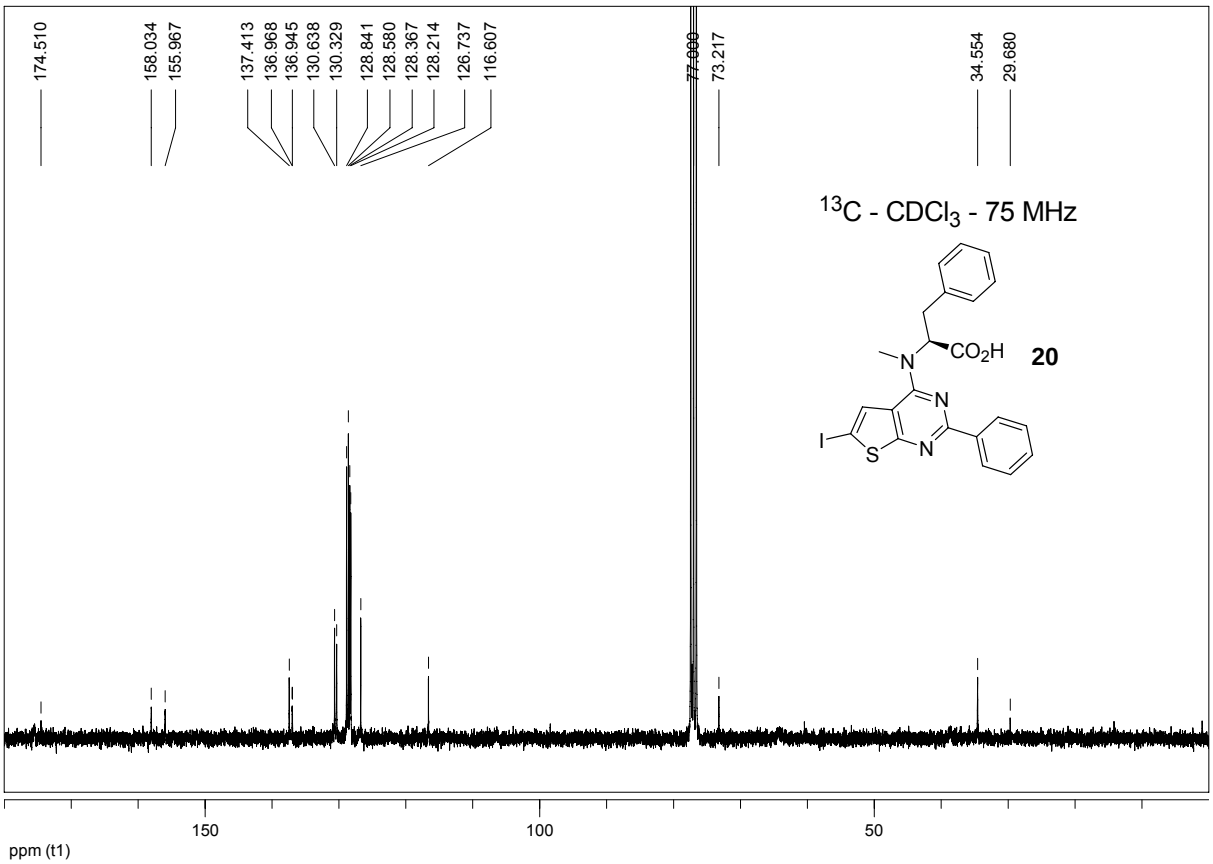

8.371
8.360
8.347
8.177
8.154
7.721
7.702
7.500
7.480
7.442
7.430
7.423
7.346
7.322
7.194
7.170
7.145
7.103
7.079
5.009
5.003
3.426
3.413
3.381
3.368
3.224
3.191
3.180
3.146
2.500
1H - (CD3)2SO - 300 MHz
HN
CO2H
N
S
N
18
1.98
0.88
0.98
0.98
1.99
2.00
1.05
0.99
1.29
1.24
8.0
7.0
6.0
5.0
4.0
3.0
ppm (t1)

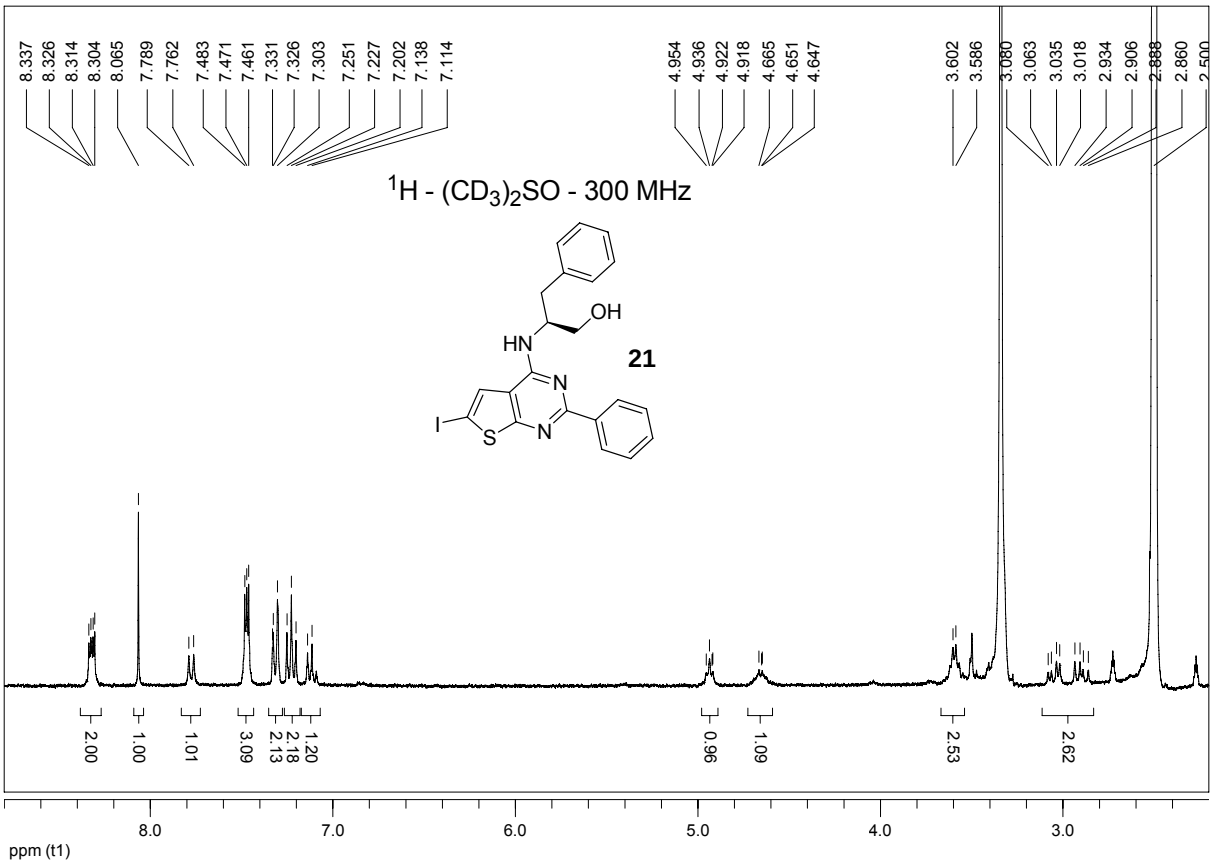

166.655
158.496
156.530
139.022
137.932
129.879
129.090
128.167
127.924
127.706
125.927
122.296
119.649
114.746
59.703
56.289
39.430
37.117
13C - (CD3)2SO - 75 MHz
HN
CO2H
N
S
N
18
150
100
50
ppm (t1)

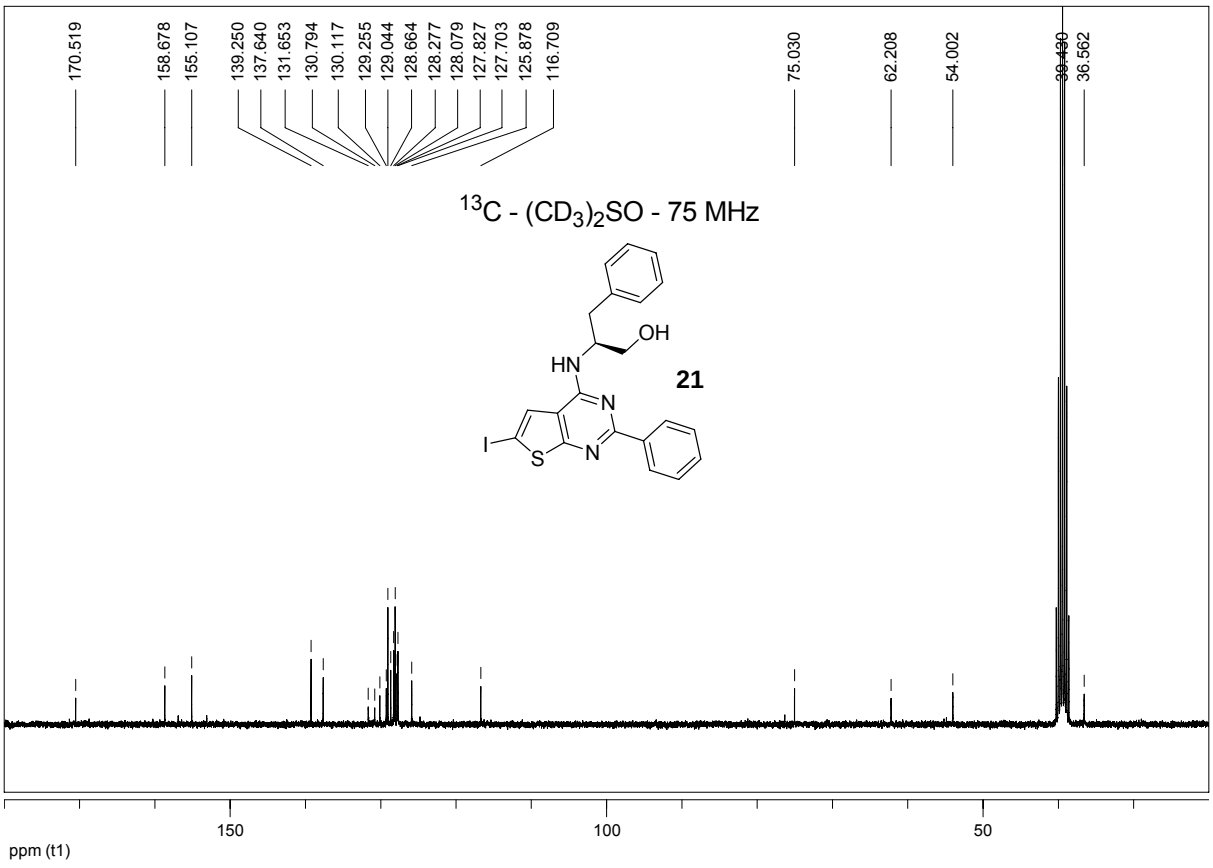

8.335
8.323
8.310
8.071
7.455
7.447
7.436
7.432
7.371
7.347
7.262
7.238
7.213
7.168
7.144
4.936
3.339
3.324
3.293
3.278
3.200
3.168
3.155
3.120
2.500
^{1}H - $(CD_3)_2SO$ - 300 MHz
HN
CO_2H
N
I
S
N
19
3.60
1.00
3.12
2.11
2.16
1.14
1.04
3.62
8.0
7.0
6.0
5.0
4.0
3.0
ppm (t1)

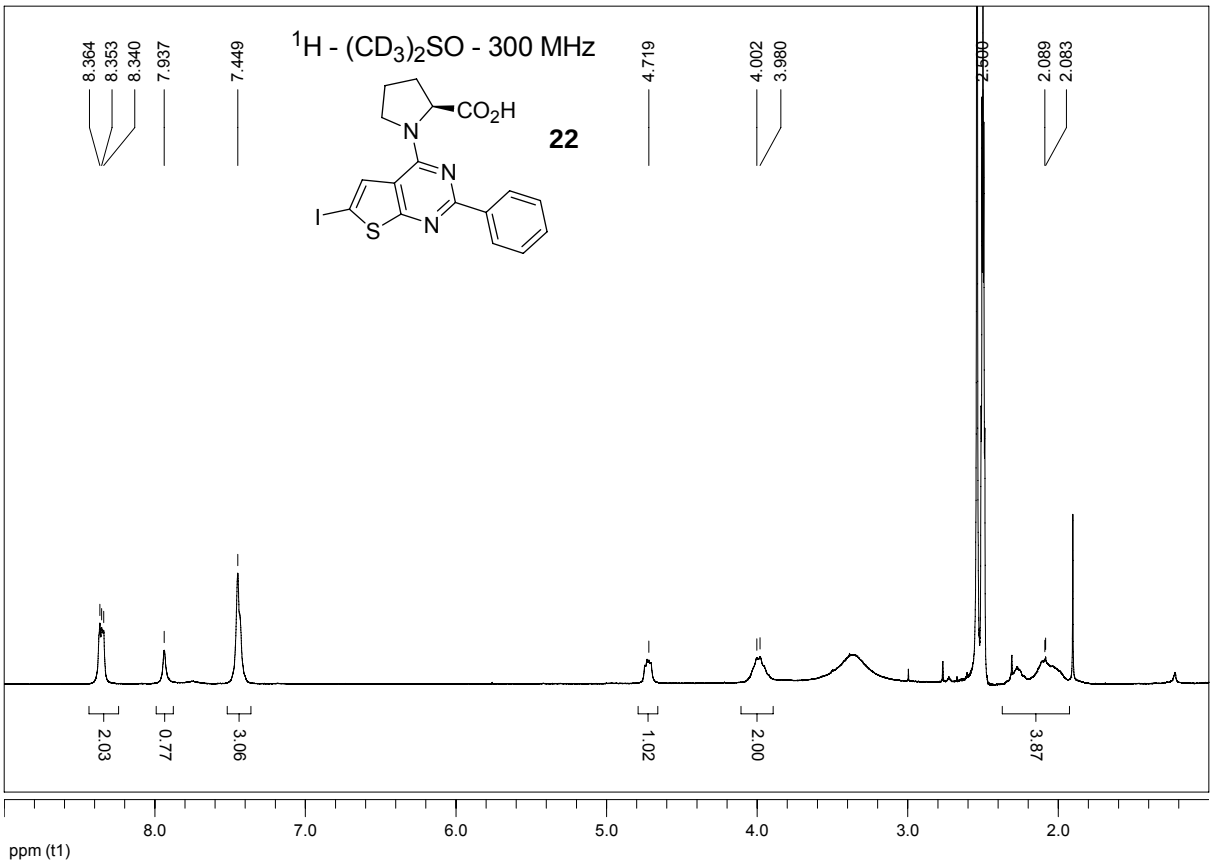

173.840
170.735
158.466
154.857
138.348
137.374
130.224
129.121
129.025
128.272
128.124
127.766
126.230
116.677
75.725
55.917
39.490
36.605
^{13}C - $(CD_3)_2SO$ - 75 MHz
HN
CO_2H
N
I
S
N
19
150
100
50
ppm (t1)

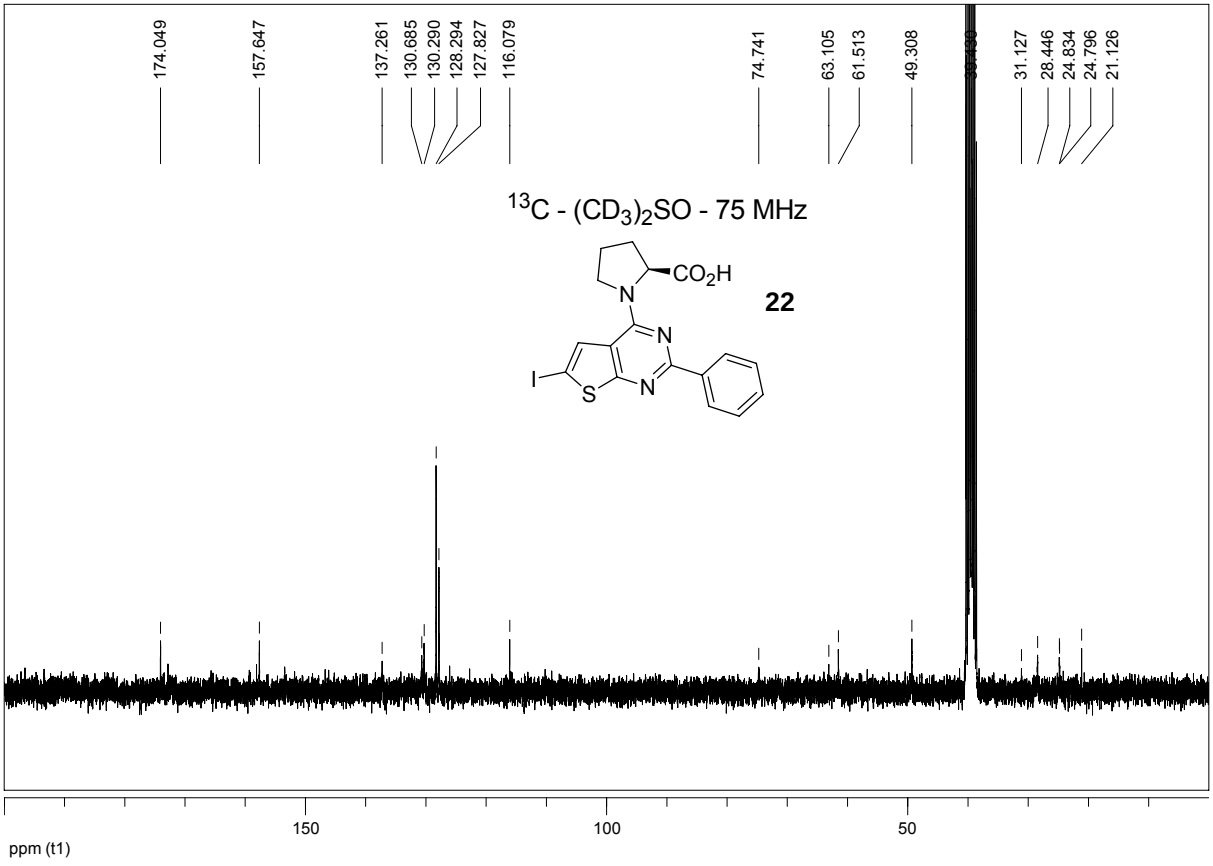

8.281
7.426
7.359
7.260
7.199
7.178
7.108
7.089
5.102
3.508
3.493
3.460
3.446
3.376
3.346
3.343
3.118
^{1}H - CDCl$_3$ - 300 MHz
CO$_2$H
20
2.00
4.58
5.25
0.71
1.96
2.98
8.0
7.0
6.0
5.0
4.0
3.0
ppm (t1)


174.510
158.034
155.967
137.413
136.968
136.945
130.638
130.329
128.841
128.580
128.367
128.214
126.737
116.607
77.000
73.217
34.554
29.680
^{13}C - CDCl$_3$ - 75 MHz
CO$_2$H
20
150
100
50
ppm (t1)


8.337
8.326
8.314
8.304
8.065
7.789
7.762
7.483
7.471
7.461
7.331
7.326
7.303
7.251
7.227
7.202
7.138
7.114
4.954
4.936
4.922
4.918
4.665
4.651
4.647
3.602
3.586
3.080
3.063
3.035
3.018
2.934
2.906
2.888
2.860
2.500
^{1}H - $(CD_3)_2SO$ - 300 MHz
OH
HN
N
21
I
S
N
2.00
1.00
1.01
3.09
2.13
2.18
1.20
0.96
1.09
2.53
2.92
8.0
7.0
6.0
5.0
4.0
3.0
ppm (t1)


170.519
158.678
155.107
139.250
137.640
131.653
130.794
130.117
129.255
129.044
128.664
128.277
128.079
127.827
127.703
125.878
116.709
75.030
62.208
54.002
39.430
36.562
^{13}C - $(CD_3)_2SO$ - 75 MHz
OH
HN
N
21
I
S
N
150
100
50
ppm (t1)


^{1}H - $(CD_3)_2SO$ - 300 MHz
22
8.364
8.353
8.340
7.937
7.449
4.719
4.002
3.980
2.500
2.089
2.083
2.03
0.77
3.06
1.02
2.00
3.87
8.0
7.0
6.0
5.0
4.0
3.0
2.0
ppm (t1)


^{13}C - $(CD_3)_2SO$ - 75 MHz
22
174.049
157.647
137.261
130.685
130.290
128.294
127.827
116.079
74.741
63.105
61.513
49.308
39.500
31.127
28.446
24.834
24.796
21.126
150
100
50
ppm (t1)