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Supporting Information
for DOI: 10.1055/s-0036-1590942

Thieme

Supporting information

Easy Access to Isomeric 7-Deazapurine - 1,2,3-Triazole Conjugates *via* S_NAr and CuAAC Reactions of 2,6-Diazido-7-deazapurines.

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General information

All reactions and purity of the synthesized compounds were monitored by TLC using Silica gel 60 F254 aluminum plates (Merck). Visualization was accomplished by UV light. Column chromatography was performed using Silica gel 60 (0.040–0.063 mm) (Merck). NMR spectra were recorded on a Bruker Ascend 400 spectrometer (400 MHz and 100 MHz for ^{1}H and ^{13}C, respectively). ^{1}H NMR and ^{13}C NMR were referenced to residual solvent peaks. High Resolution Mass Spectrometry (HRMS) analyses were carried out on a time-of-flight mass spectrometer Dual-ESI Q-TOF 6520 (Agilent Technologies). Melting points were determined in open capillaries with a digital melting point IA9100 series apparatus (ThermoFischerScientific).

General procedures for 9-alkyl-2,6-dichloro-7-deazapurines (1b-c):

A mixture of 2,4-dichloro-7*H*-pyrrolo[2,3-*d*]pyrimidine (2.0 g, 10.64 mmol), 1-iodobutane (2.4 mL , 21.28 mmol) or 1-bromoheptane (3.3 mL , 21.28 mmol), DMSO (20 mL), aqueous 50% K_2CO_3 (20 mL) and catalytic amount of benzyltriethylammonium chloride (242 mg, 1.06 mmol) was vigorously stirred for 16 hours at r. t. Then the reaction mixture was poured into water (100 mL) and extracted with EtOAc (3 x 50 mL). The extracts were combined, washed with water (2 x 50 mL), dried over Na_2SO_4 and filtered. Evaporation of the solvent under reduced pressure gave the crude product, which was purified by silica gel column chromatography (eluent – $CHCl_3$). Yield of **1b** 73% (1.9 g), yellow oil. Yield of **1c** 69% (2.1 g), yellow oil.

General procedure for 9-alkyl-2,6-diazido-7-deazapurines (2a-c):

A mixture of corresponding 9-alkyl-2,6-dichloro-7-deazapurine (**2a-c**) (1 eq.) and NaN_3 (2.2 eq.) in DMF (20 mL) was stirred at r. t. in a vessel protected from light for 24 h. Then the reaction mixture was poured into water (100 mL). Precipitate was filtered off, dried at r. t. in a dark place and stored in a vessel protected from sunlight. Products obtained as light brown solids. Yield of **2a** 78%; **2b** 88%; **2c** 85%.

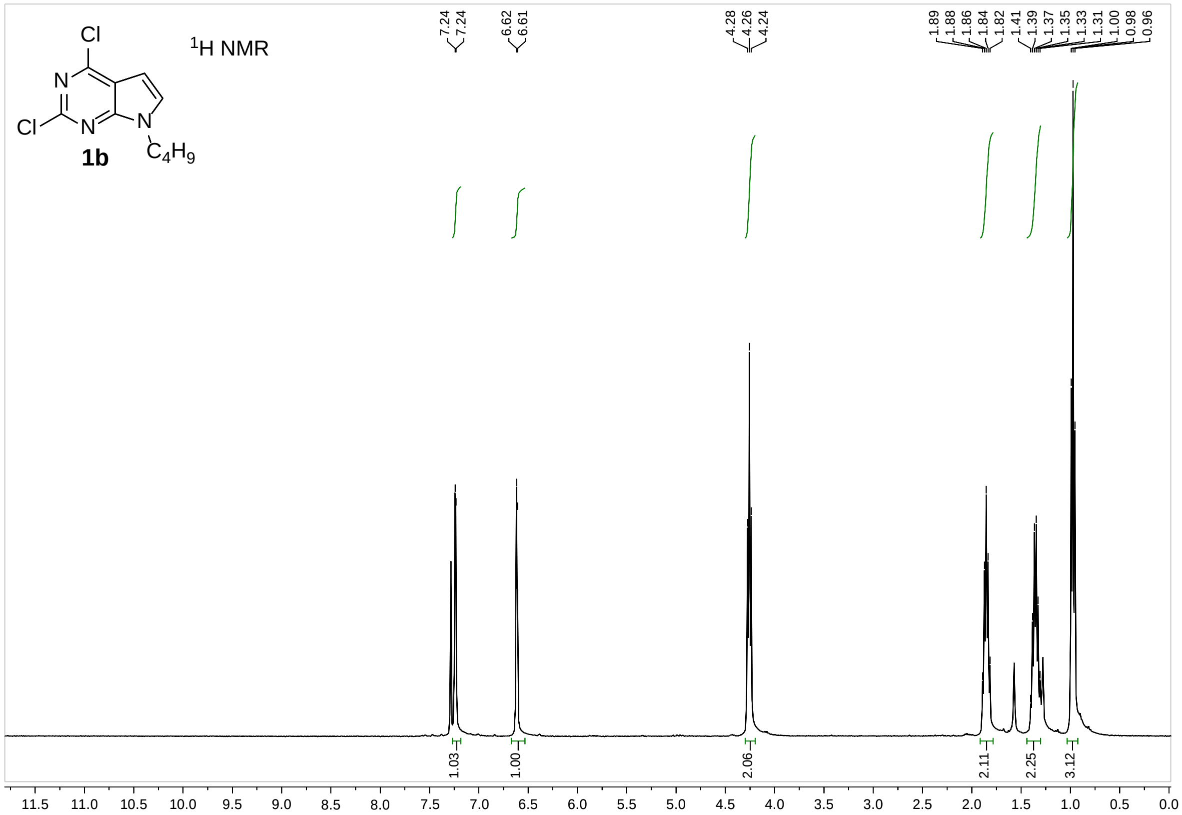
General procedure for 9-alkyl- 2-dialkylamino-6-azido-7-deazapurines (3a-d)

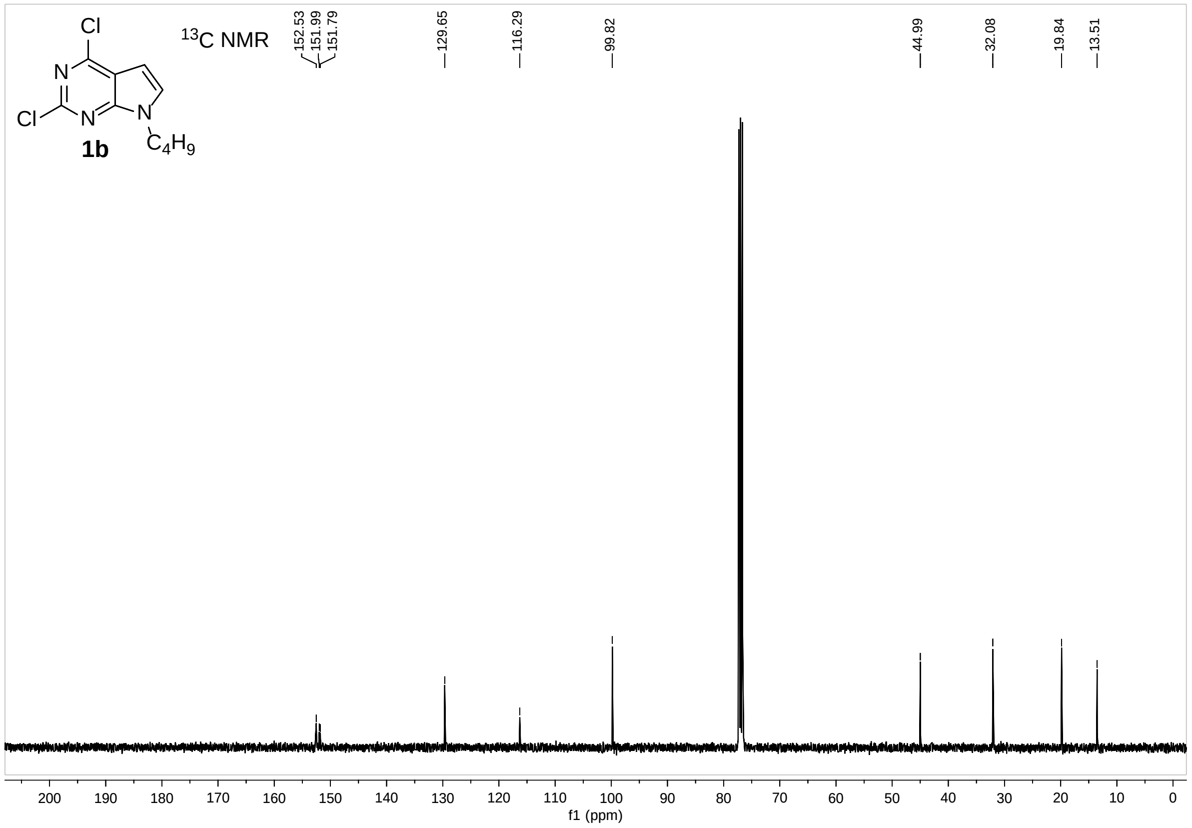
A mixture of the corresponding diazide (**2a-c**) (0.4 mmol) and secondary amine (1.2 mmol) in DMF (2 mL) were stirred for 2-4 h at 40 ^{o}C in a reaction vessel protected from light. Completion of the reaction was monitored by TLC. Then the reaction mixture was poured into water (20 mL) and precipitate was filtered (**3a-c**) or extracted with EtOAc (2 x 20 mL) (**3d**). The extracts were combined, washed with water (20 mL), dried over Na_2SO_4, filtered and solvent was removed on rotary evaporator. The obtained crude products were used further without purification. The obtained azides **3a-d** are highly sensitive to light and should be exposed as short as possible. Yields of crude products: **3a** 77%; **3b** 83%; **3c** 73%; **3d** 54%.

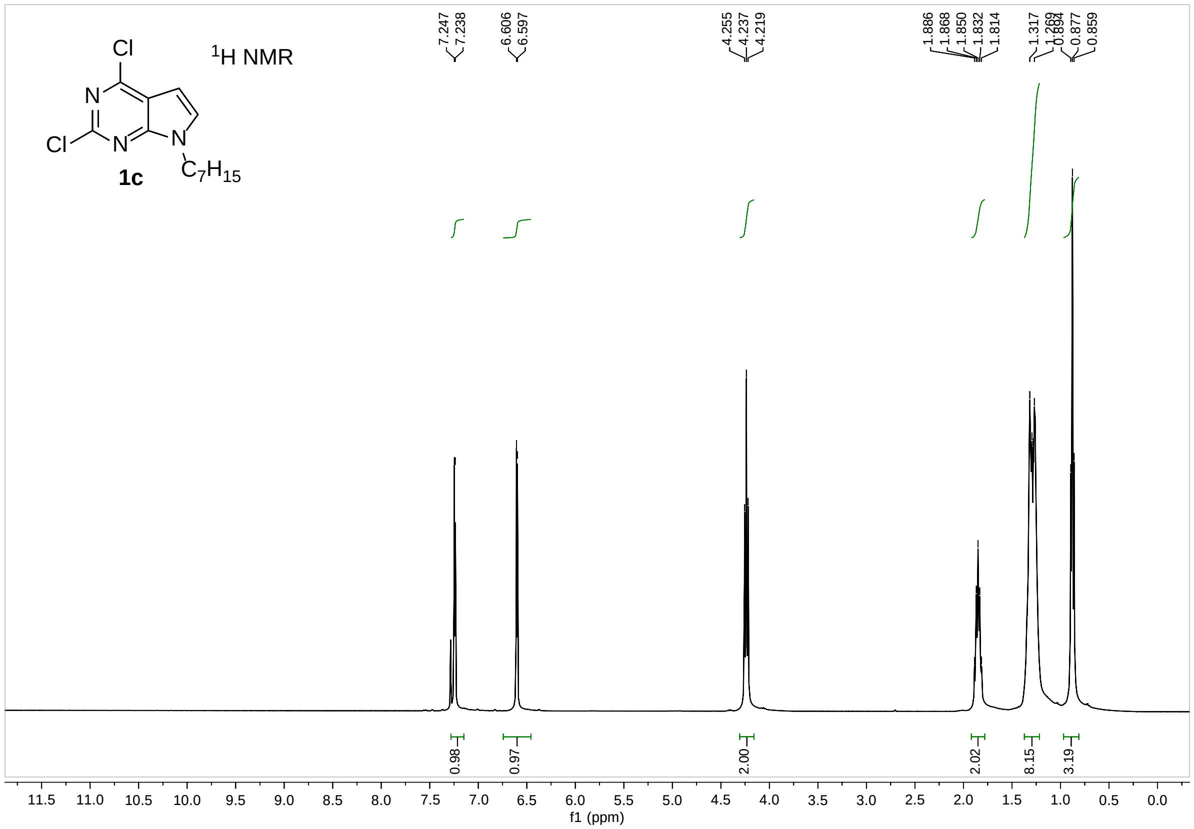
Procedure for 2-azido-9-methyl-6-piperidino-7-deazapurine (5):

A mixture of 9-methyl-2,6-dichloro-7-deazapurine (1**a**) (300 mg, 1.48 mmol) and piperidine (444 µL, 4.5 mmol) in CH_3CN (5mL) was stirred at r. t. for 3 h. Then the reaction mixture was poured into water (25 mL) and extracted with $CHCl_3$ (2 x 30mL). The extracts were combined, dried over Na_2SO_4 and filtered. Evaporation of the solvent under reduced pressure gave the brown pale oil, which was purified by silica gel column chromatography ($CHCl_3$ - EtOAc, 9:1) to afford the title compound as a yellow pale oil (326 mg, 88%).

Copies of NMR spectra for all products:







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Cl
N
N
C7H15
1c
1H NMR
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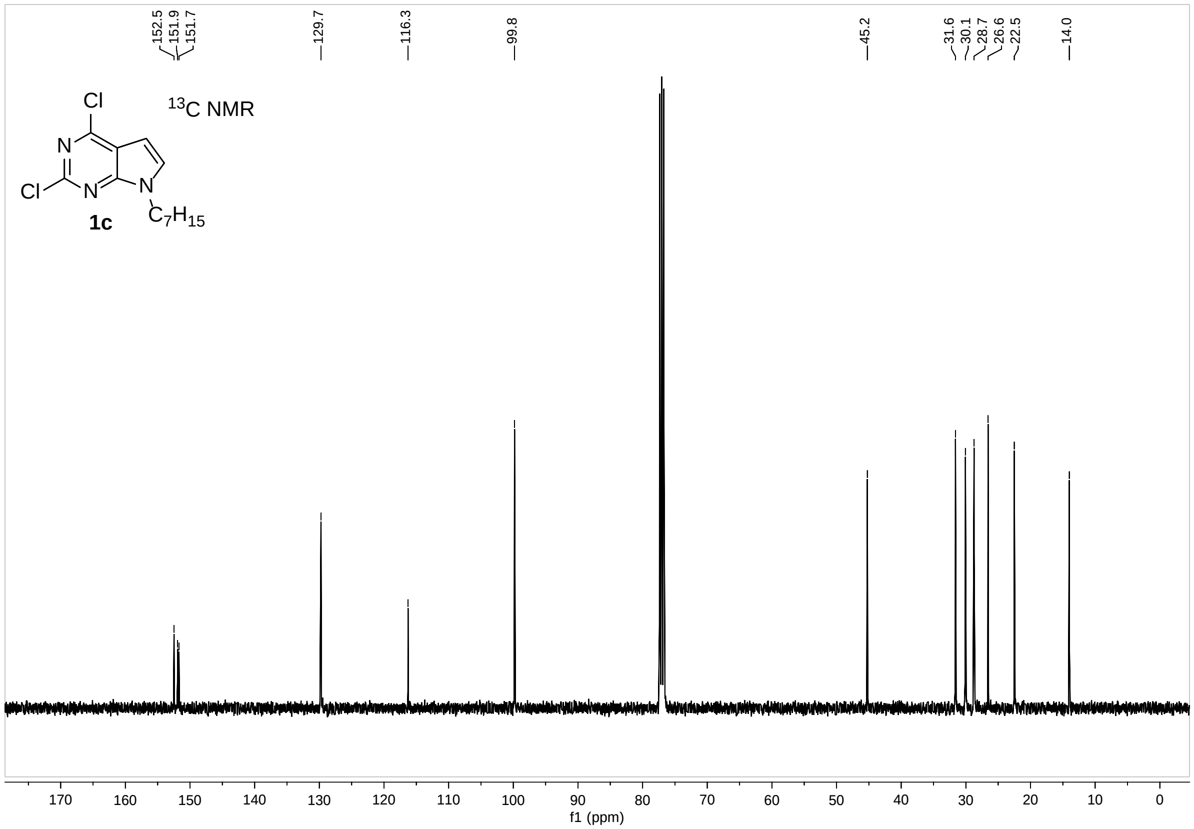
7.238
6.606
6.597
4.255
4.237
4.219
1.886
1.868
1.850
1.832
1.814
1.317
0.877
0.859
0.98
0.97
2.00
2.02
8.15
3.19
11.5 11.0 10.5 10.0 9.5 9.0 8.5 8.0 7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 0.5 0.0
f1 (ppm)



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N
Cl
N
N
C7H15
1c
13C NMR
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151.9
151.7
129.7
116.3
99.8
45.2
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28.7
26.6
22.5
14.0
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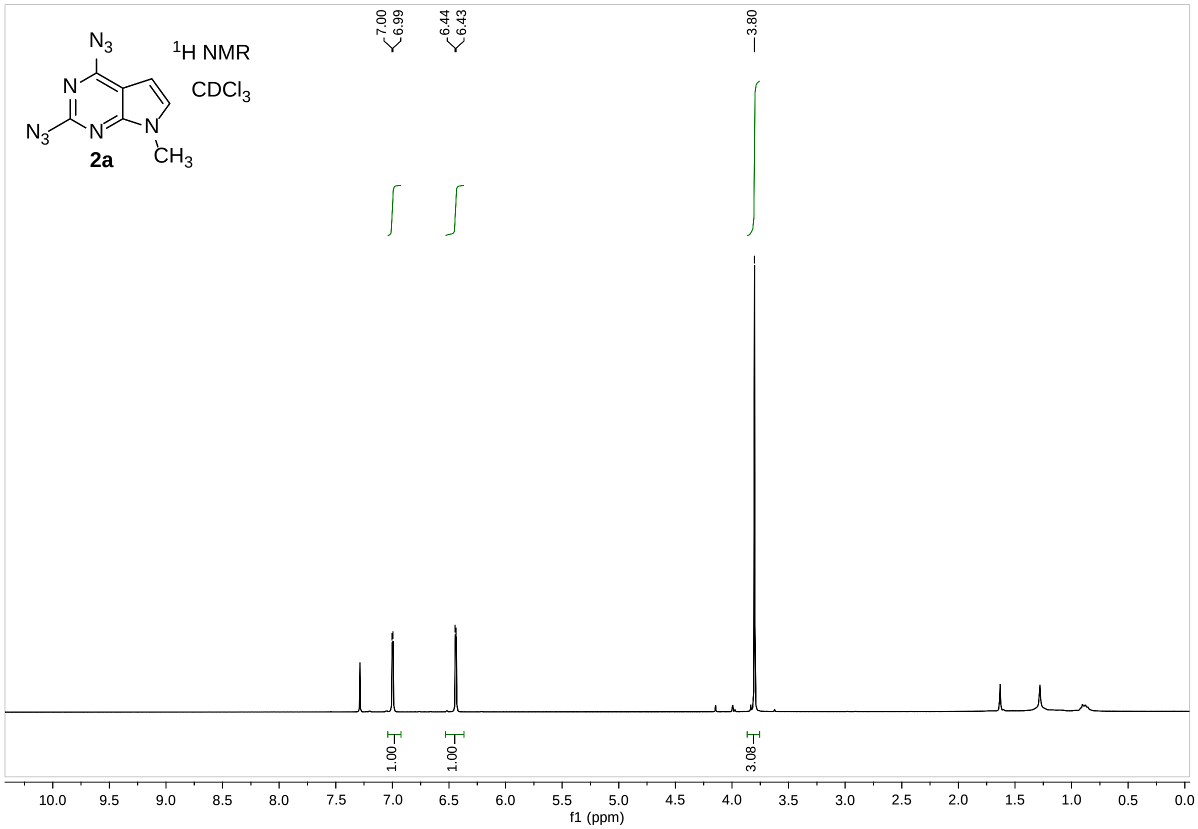
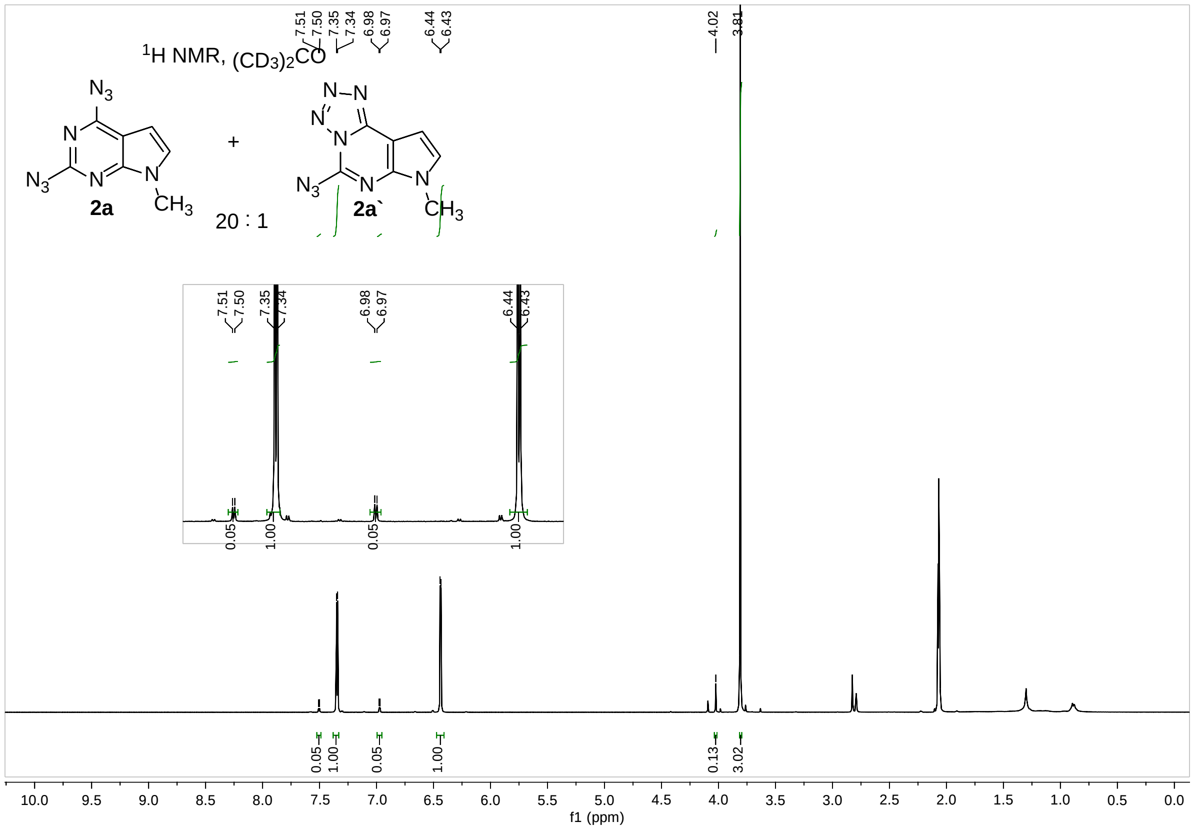
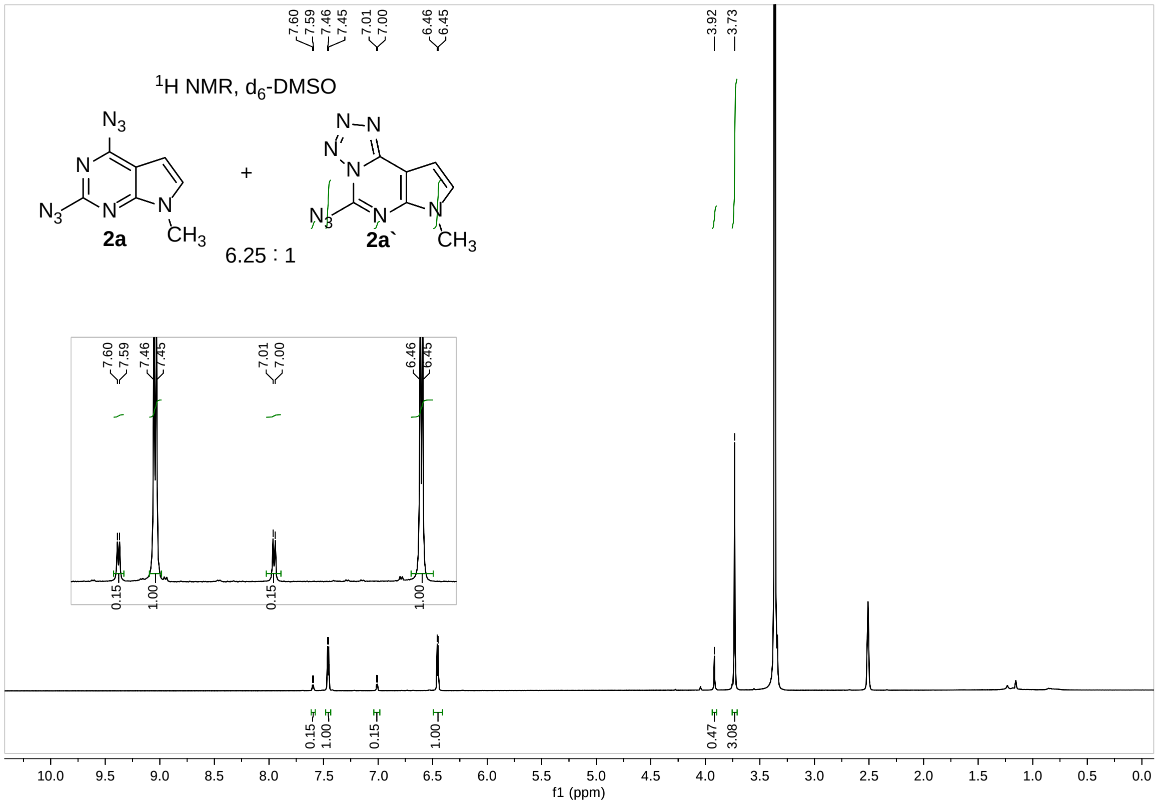
f1 (ppm)



1H NMR
CDCl3
N3
N
N3
N
N
CH3
2a
7.00
6.99
6.44
6.43
3.80
1.00
1.00
3.08
10.0 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 0.5 0.0
f1 (ppm)



1H NMR, (CD3)2CO
N3
N
N3
N
N
CH3
2a
+
N–N
N
N
N3
N
N
CH3
2a`
20 : 1
7.51
7.50
7.35
7.34
6.98
6.97
6.44
6.43
4.02
3.81
0.05
1.00
0.05
1.00
0.05
1.00
0.05
1.00
0.13
3.02
10.0 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 0.5 0.0
f1 (ppm)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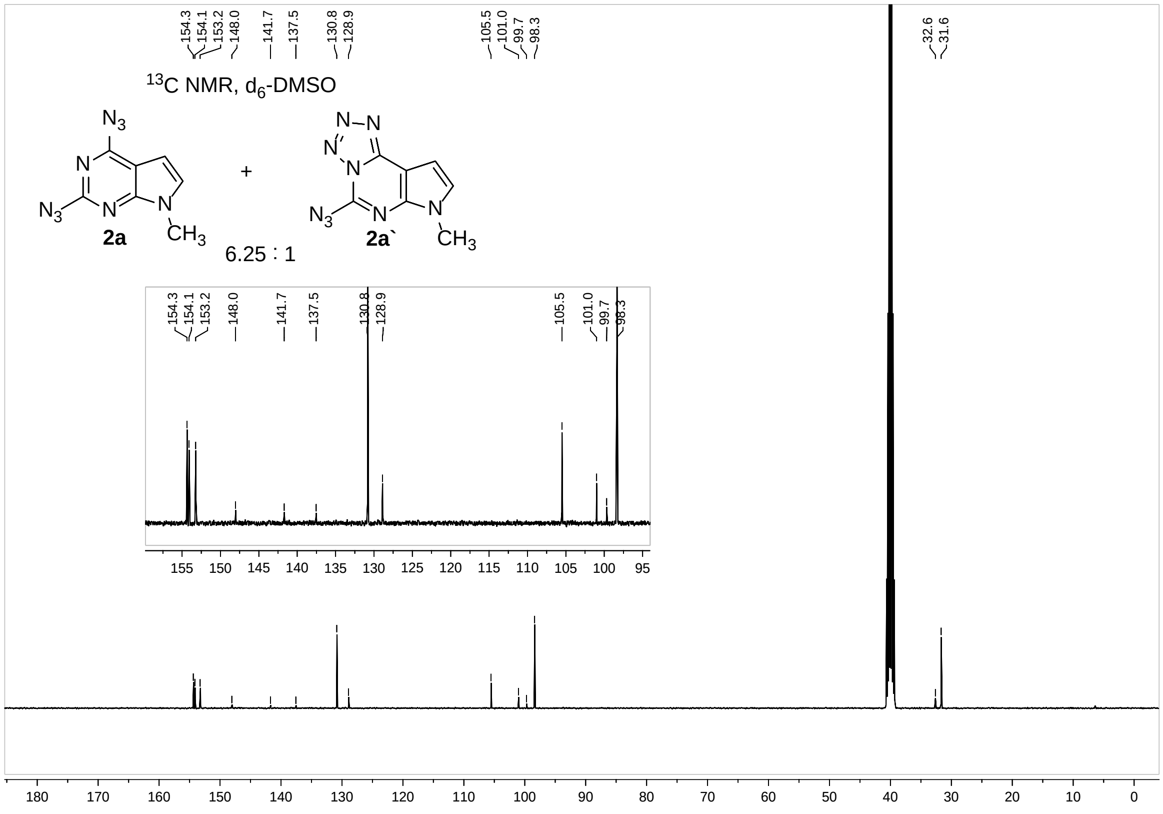
7.59
7.46
7.45
7.01
7.00
6.46
6.45
3.92
3.73
^{1}H NMR, d$_6$-DMSO
N$_3$
N$_3$
2a
CH$_3$
+
N–N
N$_3$
2a`
CH$_3$
6.25 : 1
0.15
1.00
0.15
1.00
0.47
3.08
10.0 9.5 9.0 8.5 8.0 7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 0.5 0.0
f1 (ppm)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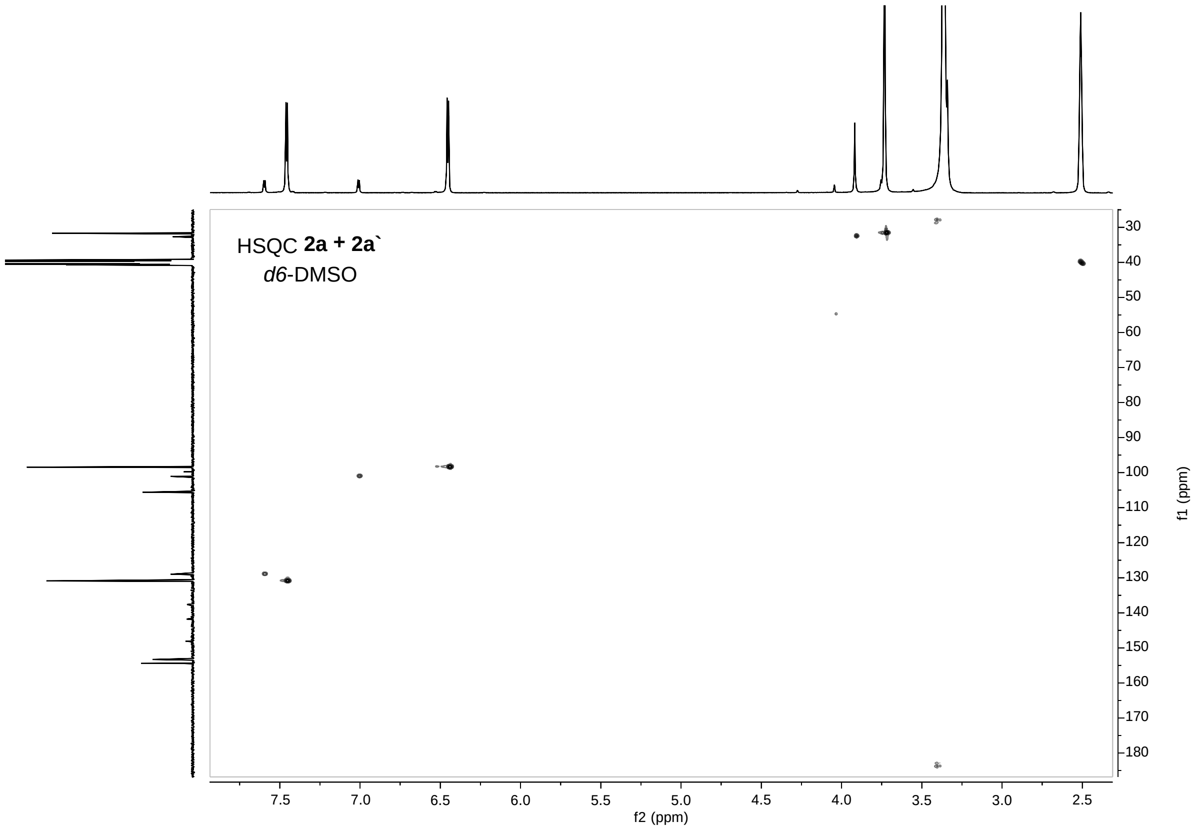
153.2
148.0
141.7
137.5
130.8
128.9
105.5
101.0
99.7
98.3
32.6
31.6
^{13}C NMR, d$_6$-DMSO
N$_3$
N$_3$
2a
CH$_3$
+
N–N
N$_3$
2a`
CH$_3$
6.25 : 1
155 150 145 140 135 130 125 120 115 110 105 100 95
180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0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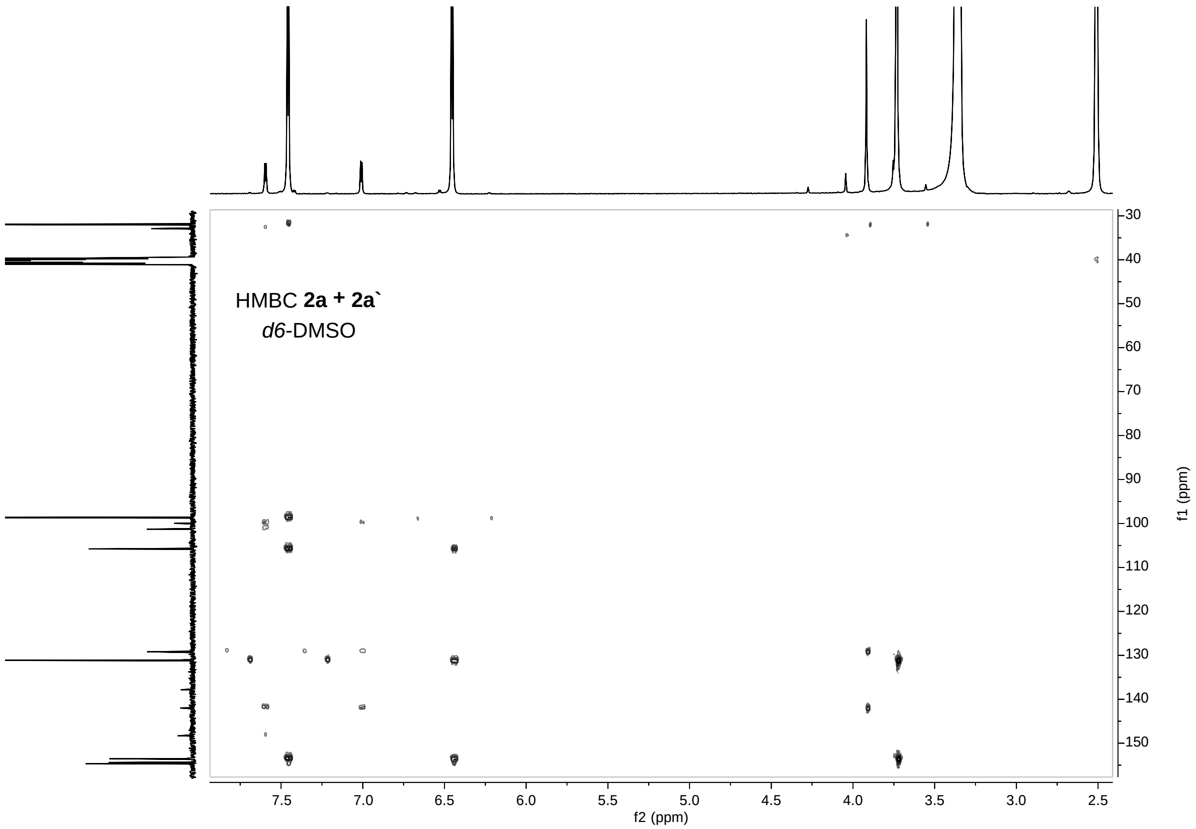
d6-DMSO
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
f2 (ppm)
30
40
50
60
70
80
90
100
110
120
130
140
150
160
170
180
f1 (ppm)


HMBC 2a + 2a`
d6-DMSO
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
f2 (ppm)
30
40
50
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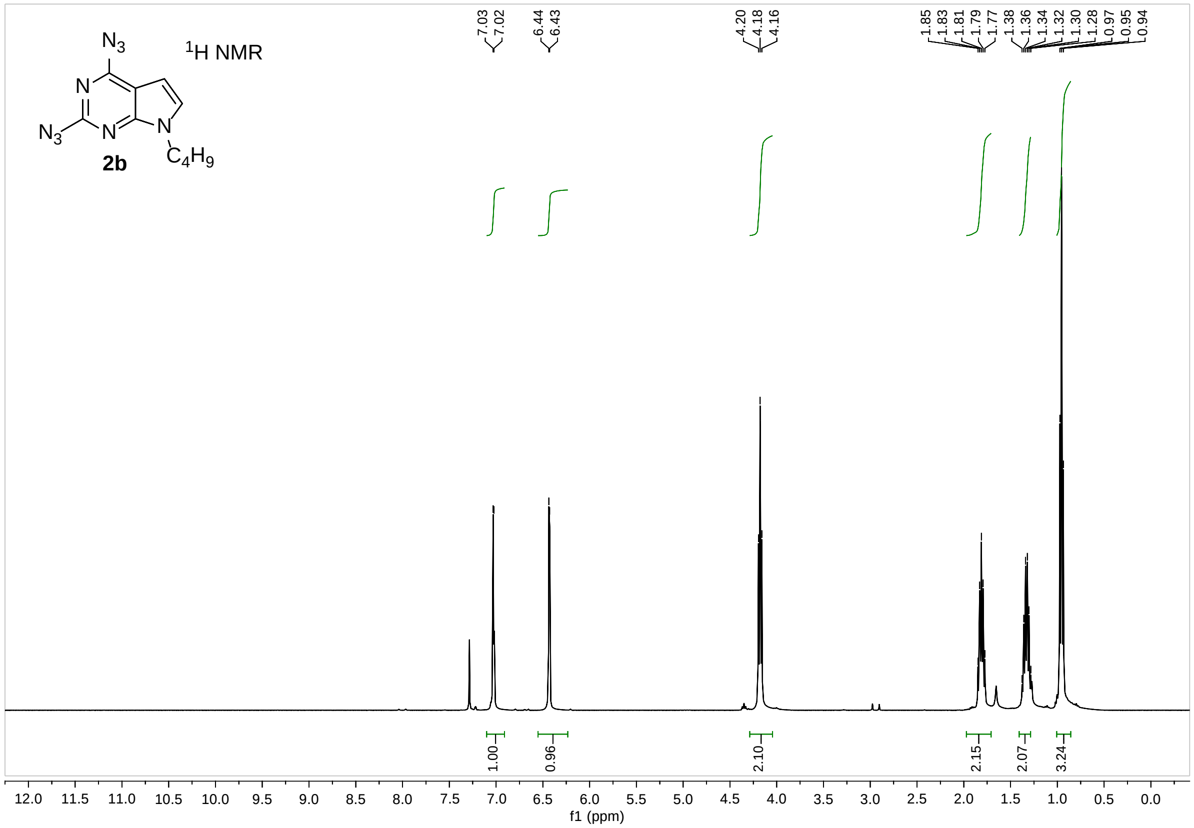
70
80
90
100
110
120
130
140
150
f1 (ppm)


N_3
N
N_3
N
N
C_4H_9
2b
^1H NMR
7.03
7.02
6.44
6.43
4.20
4.18
4.16
1.85
1.83
1.81
1.79
1.77
1.38
1.36
1.34
1.32
1.30
1.28
0.97
0.95
0.94
1.00
0.96
2.10
2.15
2.07
3.24
12.0 11.5 11.0 10.5 10.0 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 0.5 0.0
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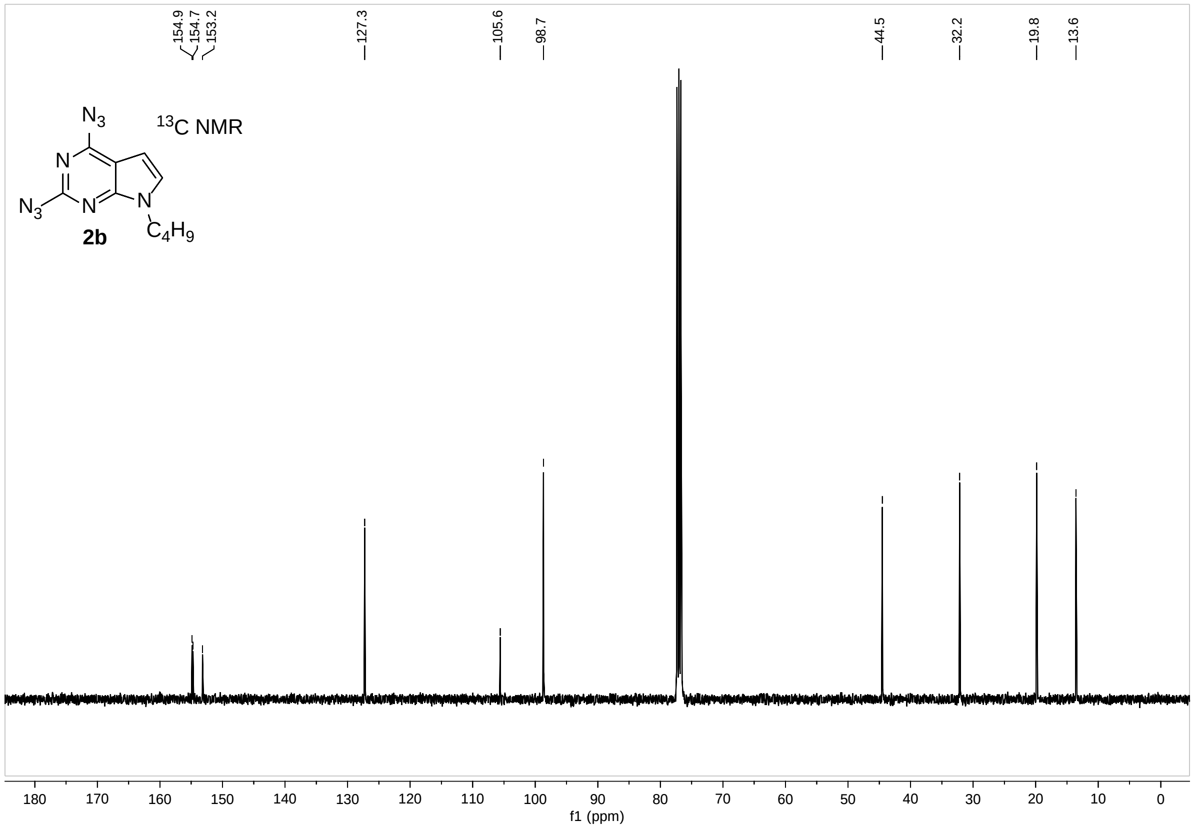
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105.6
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19.8
13.6
N_3
N
N_3
N
N
C_4H_9
2b
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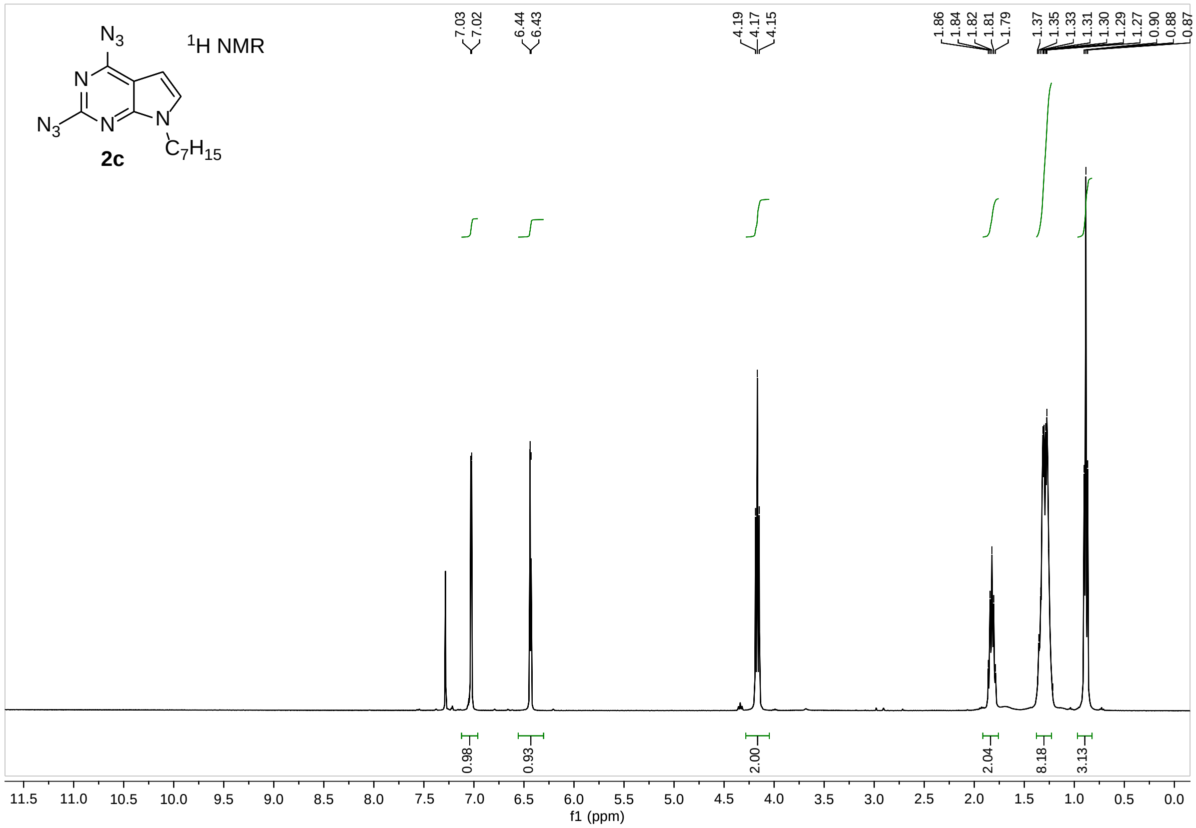
f1 (ppm)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N3
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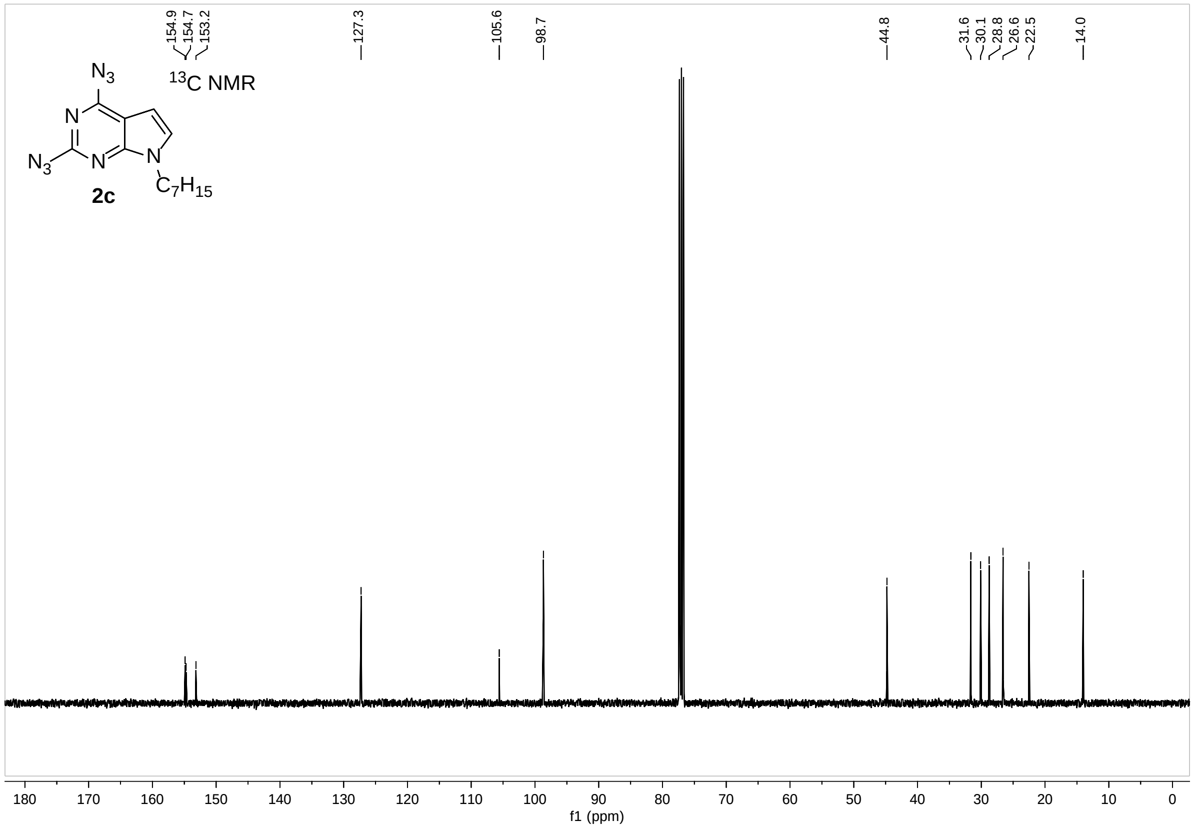
4.15
1.86
1.84
1.82
1.81
1.79
1.37
1.35
1.33
1.31
1.30
1.29
1.27
0.90
0.88
0.87
0.98
0.93
2.00
2.04
8.18
3.13
11.5 11.0 10.5 10.0 9.5 9.0 8.5 8.0 7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 0.5 0.0
f1 (ppm)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N
N3
N
C7H15
2c
13C NMR
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154.7
153.2
127.3
105.6
98.7
44.8
31.6
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28.8
26.6
22.5
14.0
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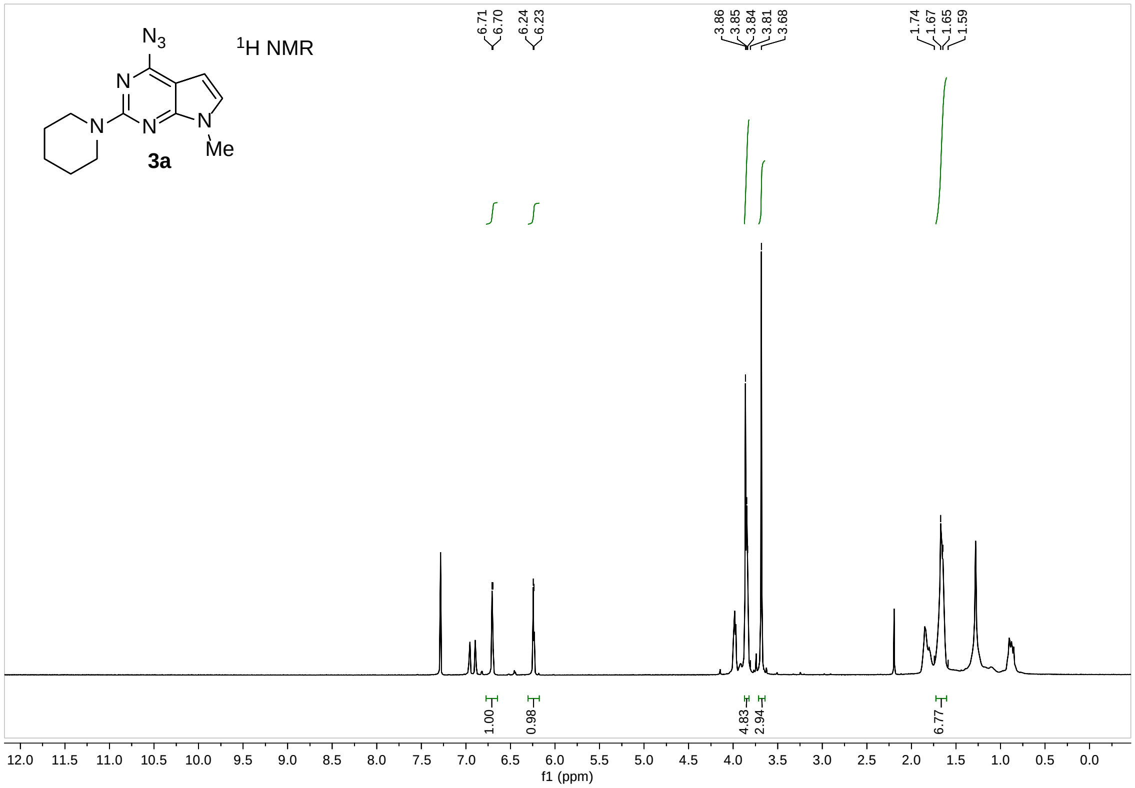
f1 (ppm)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Me
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3.84
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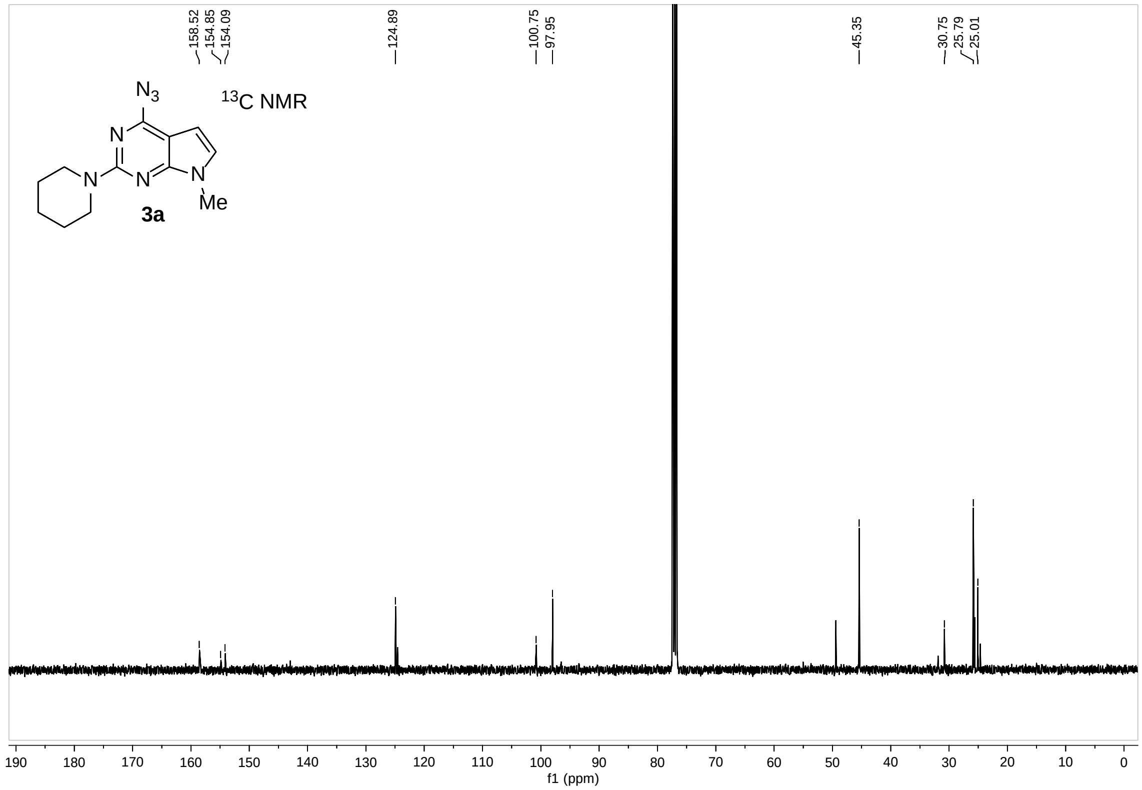
3.68
1.74
1.67
1.65
1.59
1.00
0.98
4.83
2.94
6.77
12.0 11.5 11.0 10.5 10.0 9.5 9.0 8.5 8.0 7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 0.5 0.0
f1 (ppm)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Me
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25.79
25.01
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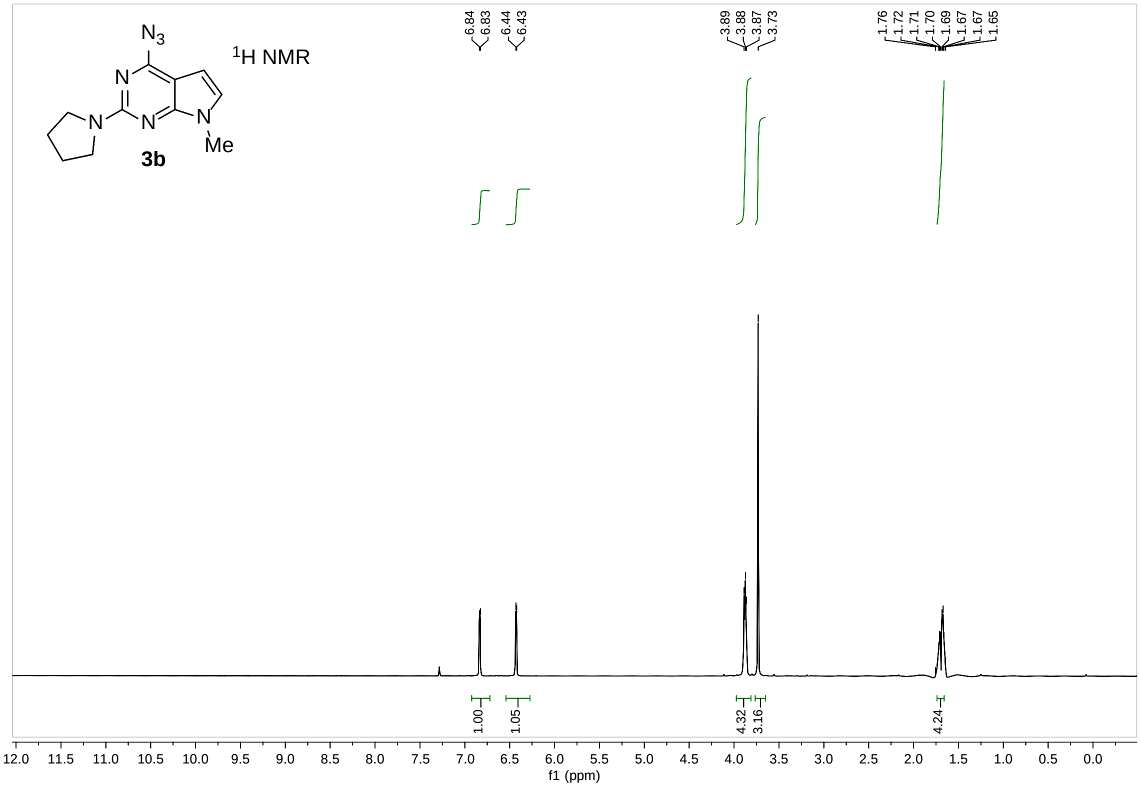
f1 (ppm)


N3
N
N
N
N
Me
3b
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6.43
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3.73
1.76
1.72
1.71
1.70
1.69
1.67
1.67
1.65
1.00
1.05
4.32
3.16
4.24
12.0
11.5
11.0
10.5
10.0
9.5
9.0
8.5
8.0
7.5
7.0
6.5
6.0
5.5
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1.0
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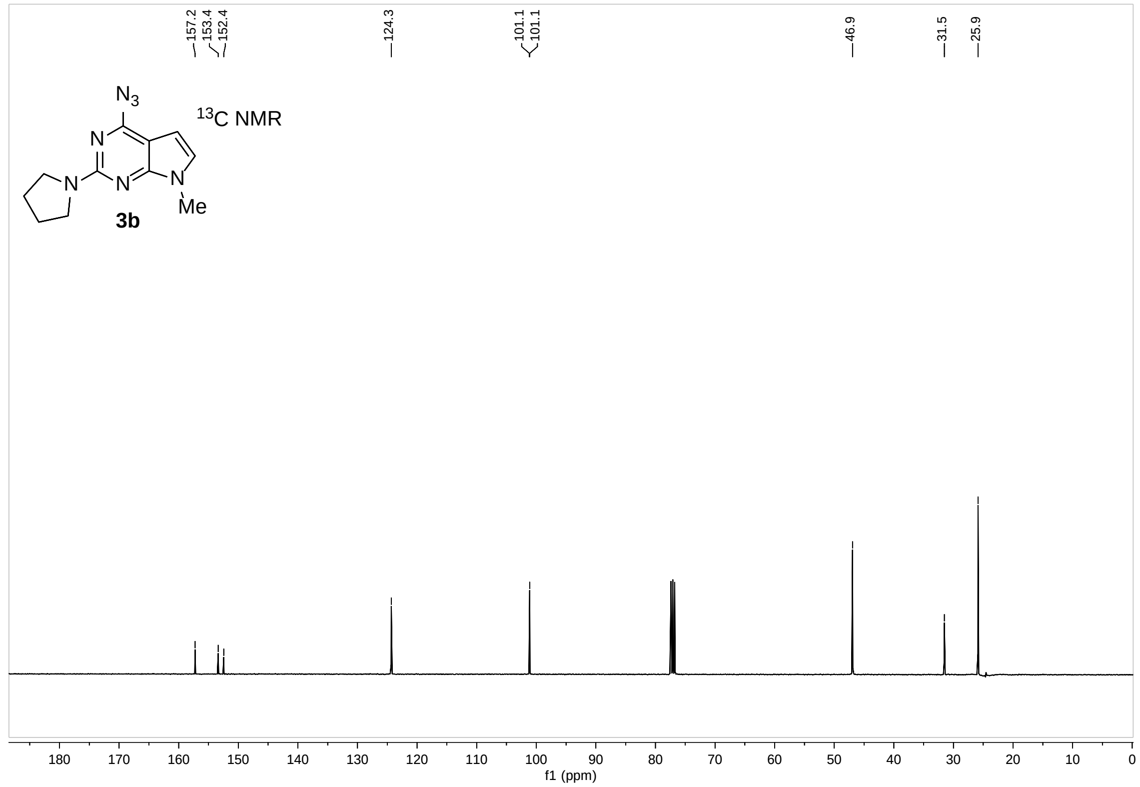
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152.4
124.3
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N3
13C NMR
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N
N
Me
3b
180
170
160
150
140
130
120
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80
70
60
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40
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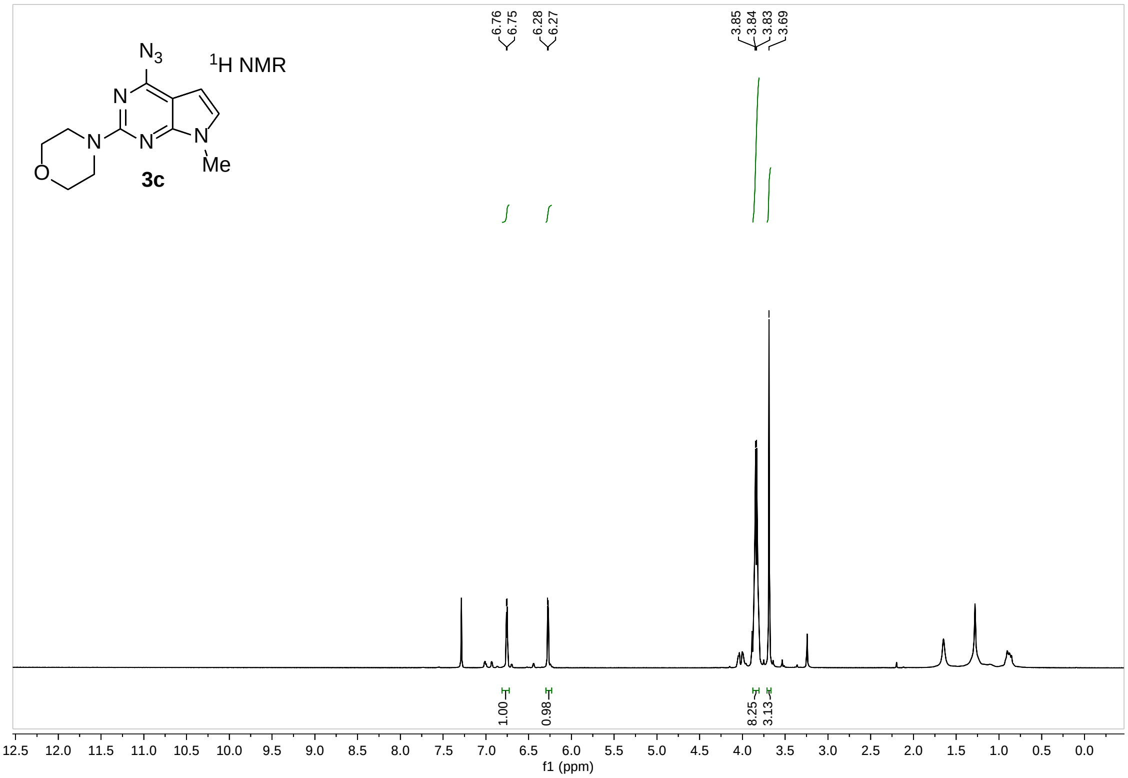
20
10
0
f1 (ppm)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O
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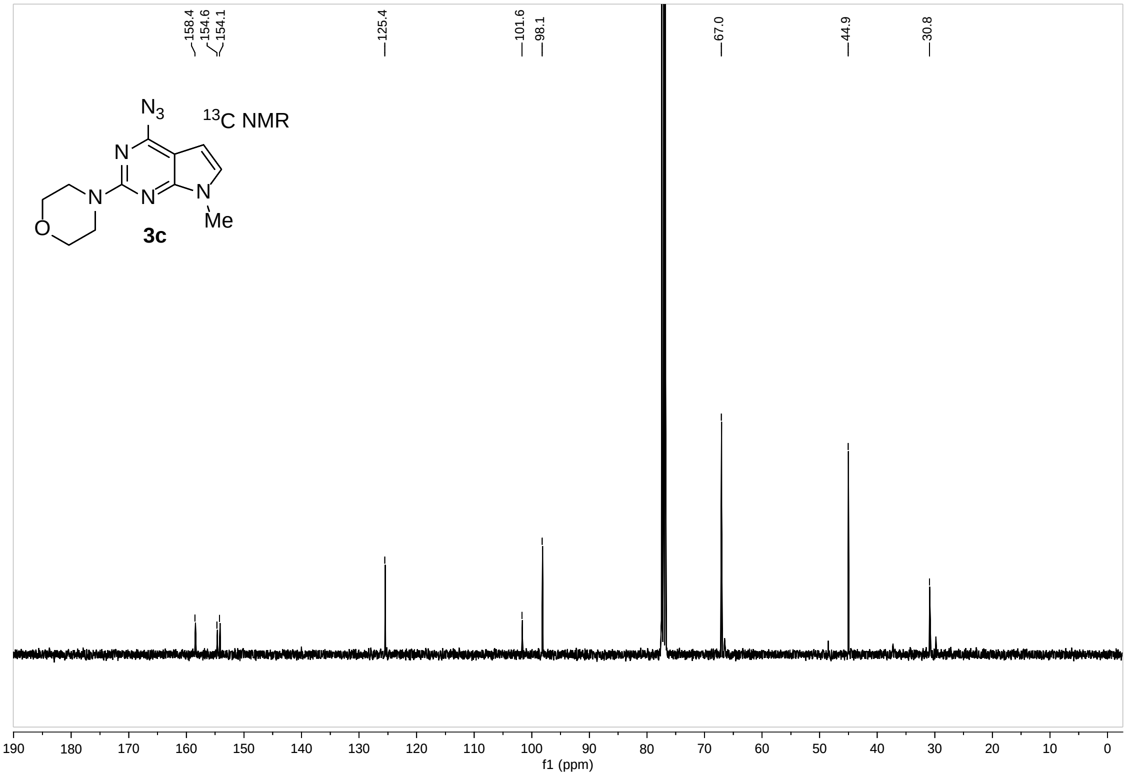
3.84
3.83
3.69
1.00
0.98
8.25
3.13
12.5 12.0 11.5 11.0 10.5 10.0 9.5 9.0 8.5 8.0 7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 0.5 0.0
f1 (ppm)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30.8
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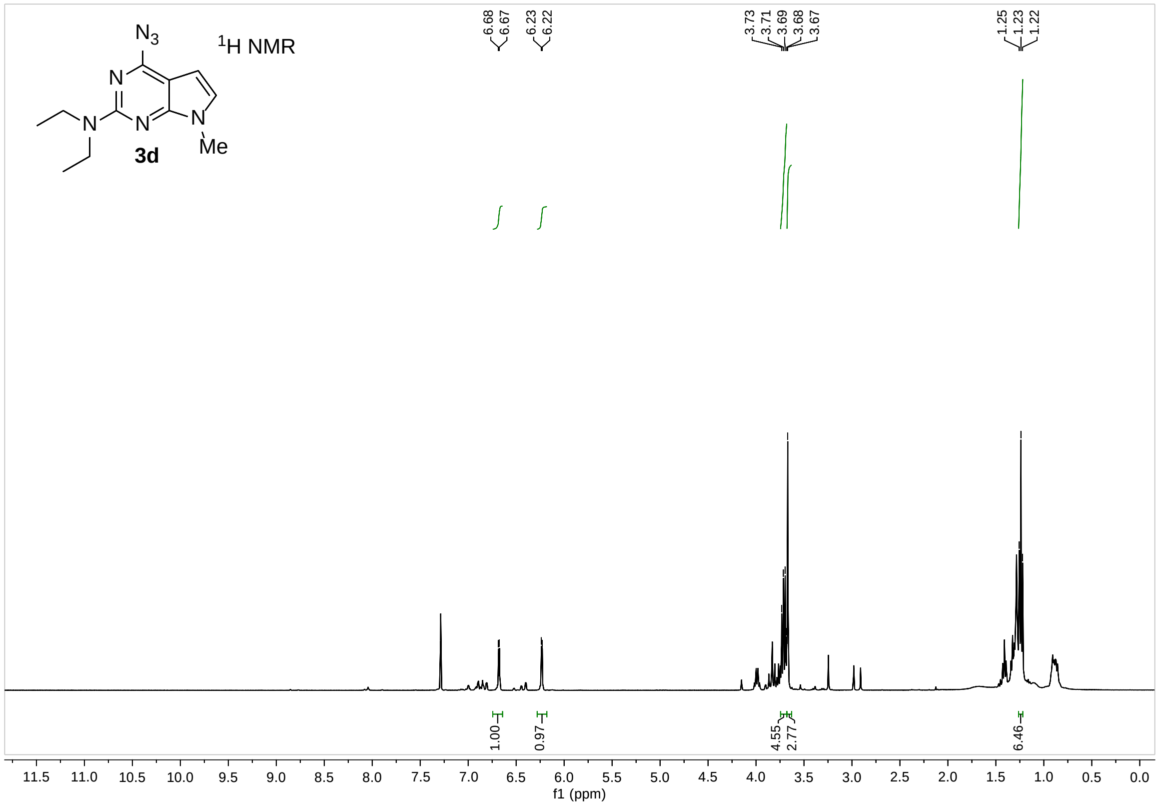
f1 (ppm)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N
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N
Me
3d
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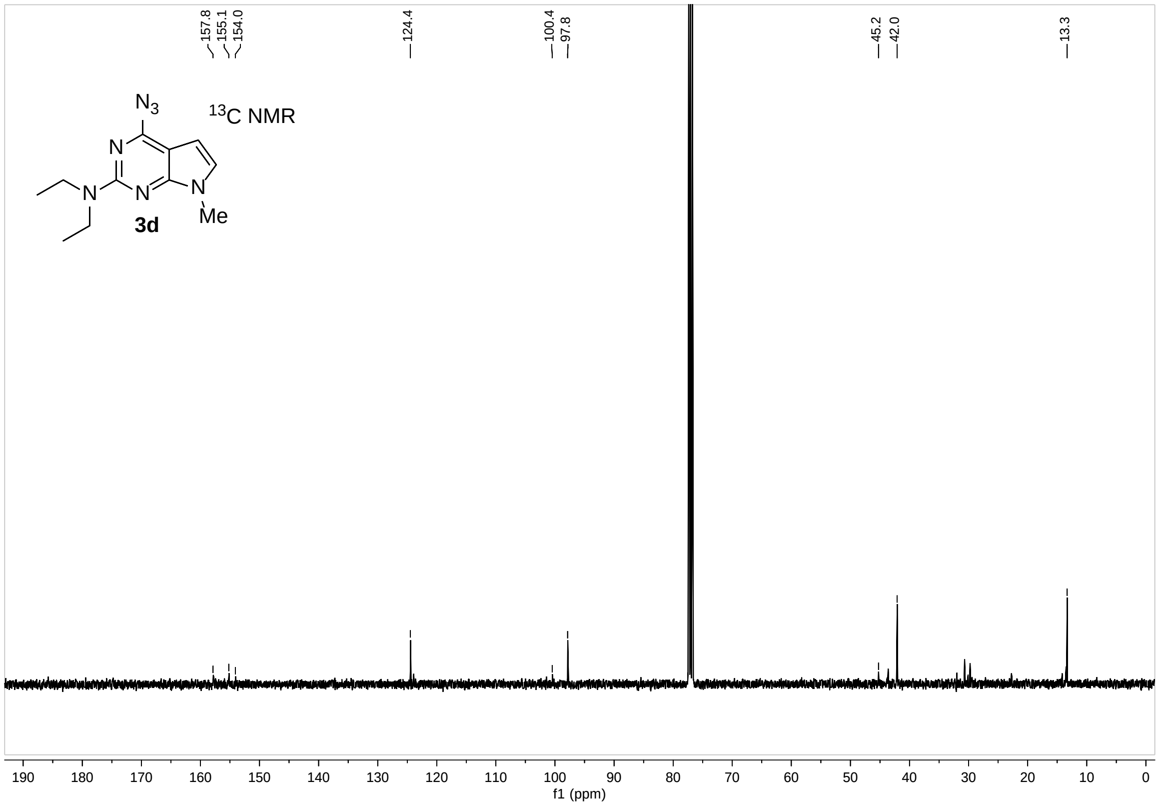
3.69
3.68
3.67
1.25
1.23
1.22
1.00
0.97
4.55
2.77
6.46
11.5 11.0 10.5 10.0 9.5 9.0 8.5 8.0 7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 0.5 0.0
f1 (ppm)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N
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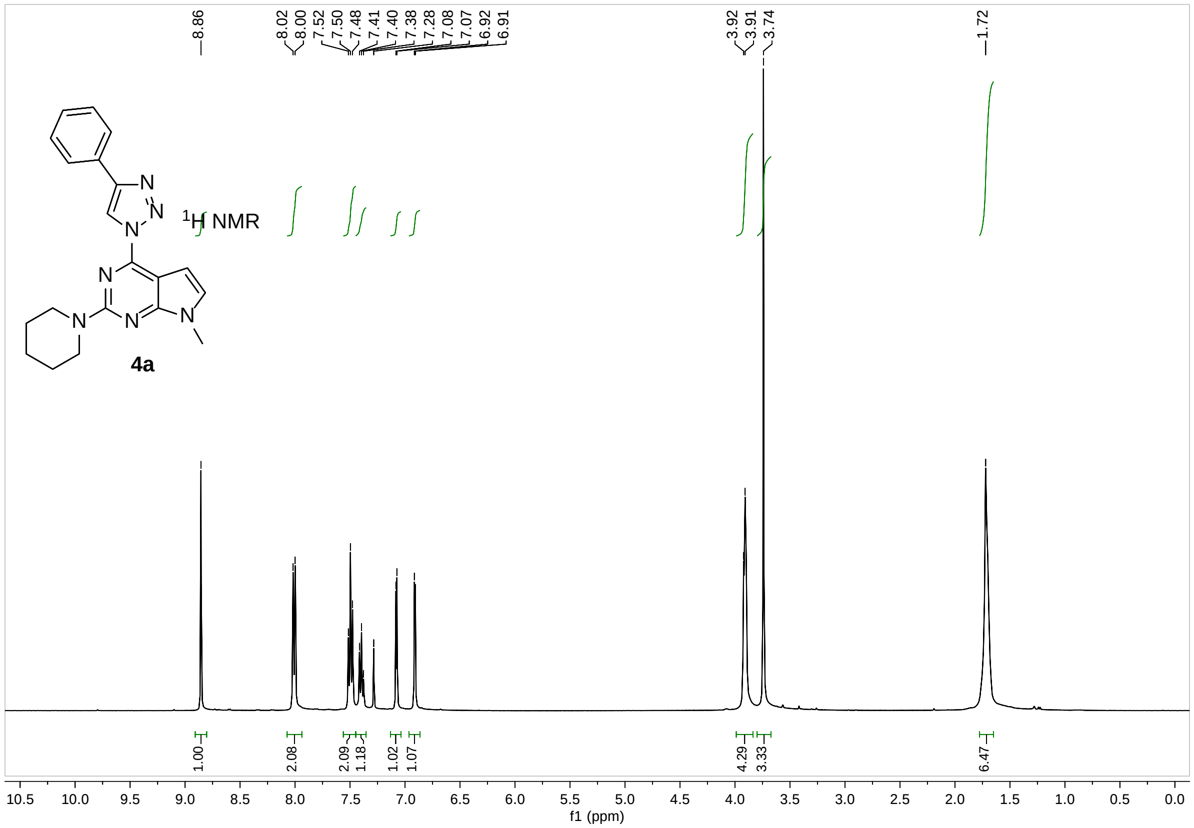
N
N
Me
3d
13C NMR
190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0
f1 (ppm)



8.86
8.02
8.00
7.52
7.50
7.48
7.41
7.40
7.38
7.28
7.08
7.07
6.92
6.91
3.92
3.91
3.74
1.72
^{1}H NMR
4a
N
N
N
N
N
N
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1.00
2.08
2.09
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1.07
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8.0
7.5
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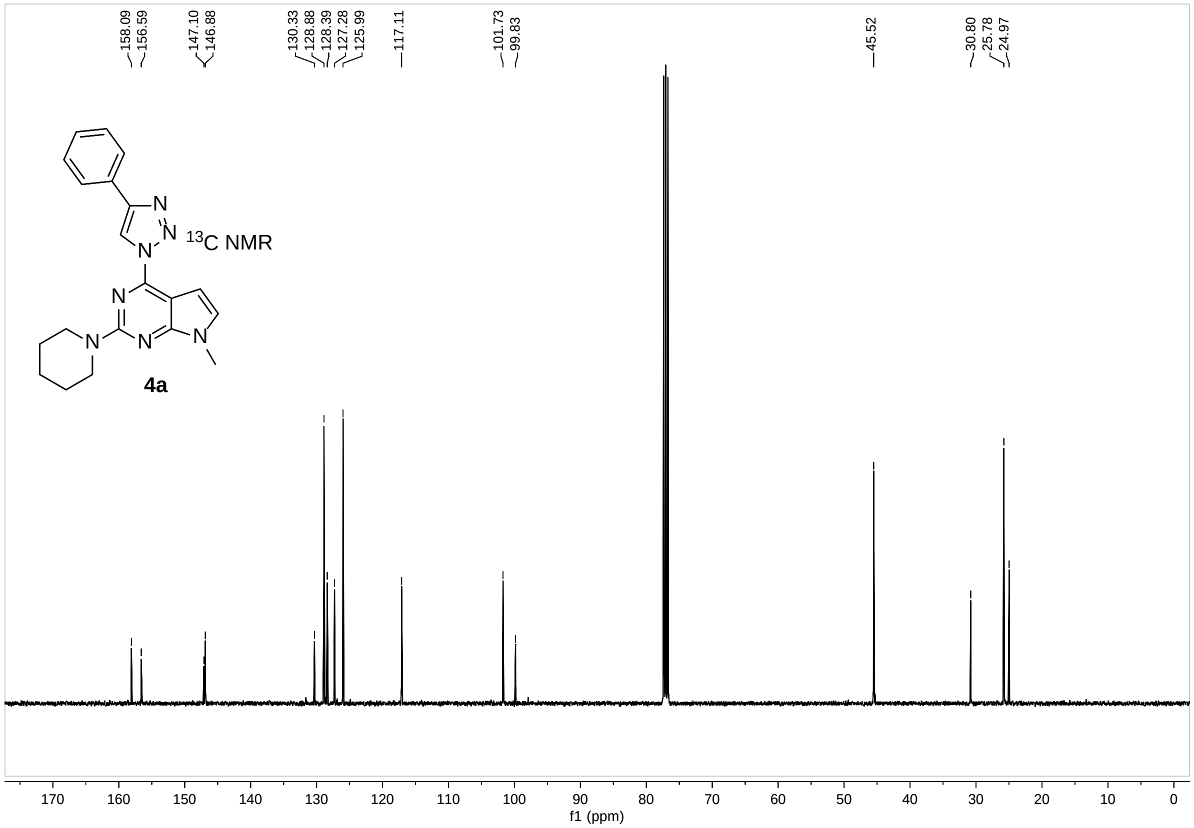
f1 (ppm)



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128.39
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30.80
25.78
24.97
^{13}C NMR
4a
N
N
N
N
N
N
N
170
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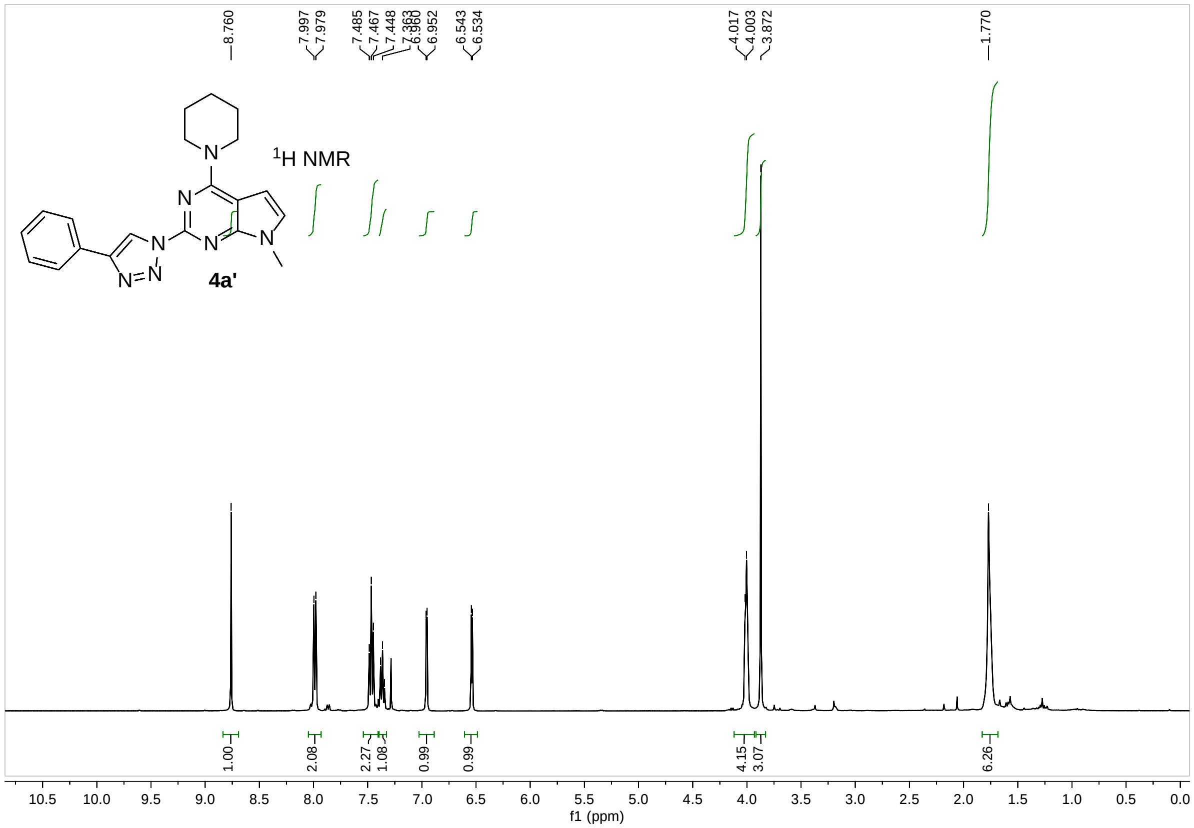
10
0
f1 (ppm)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3.872
1.770
^{1}H NMR
N
N
N
N
N
N=N
4a'
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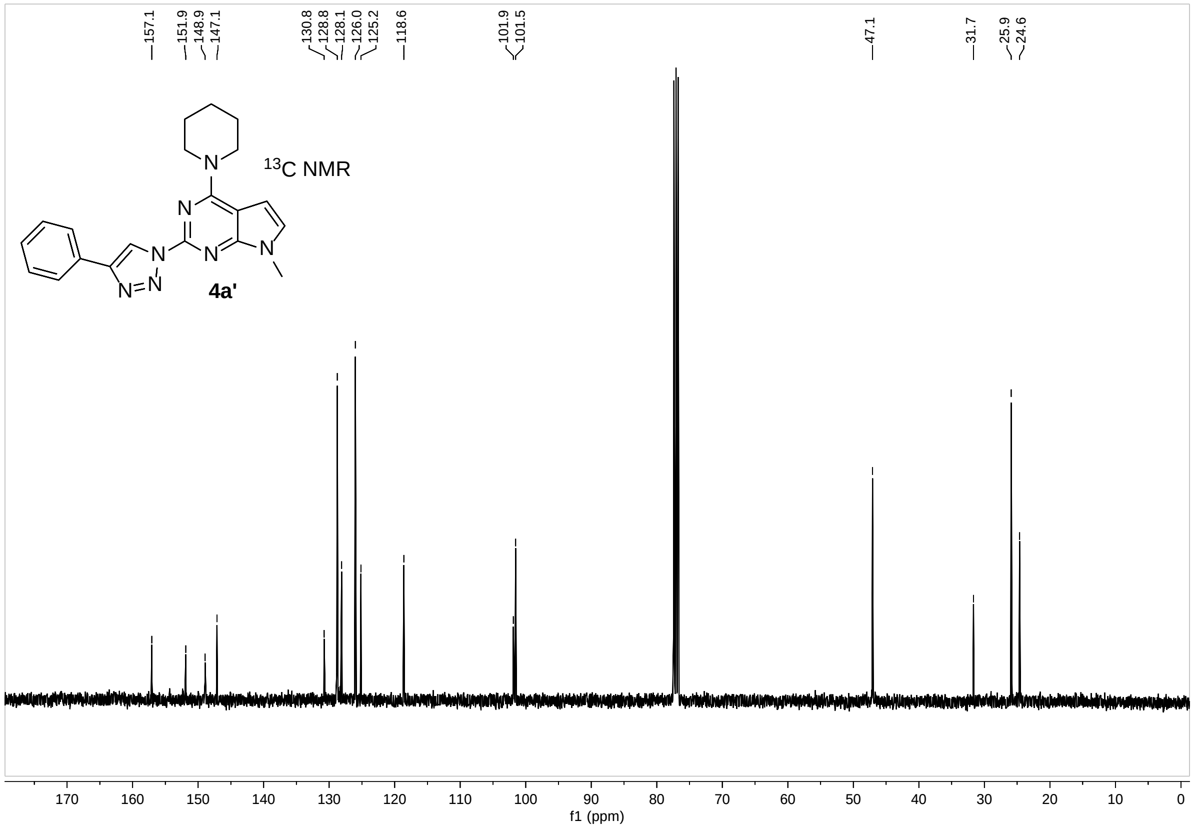
2.08
2.27
1.08
0.99
0.99
4.15
3.07
6.26
10.5 10.0 9.5 9.0 8.5 8.0 7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 0.5 0.0
f1 (ppm)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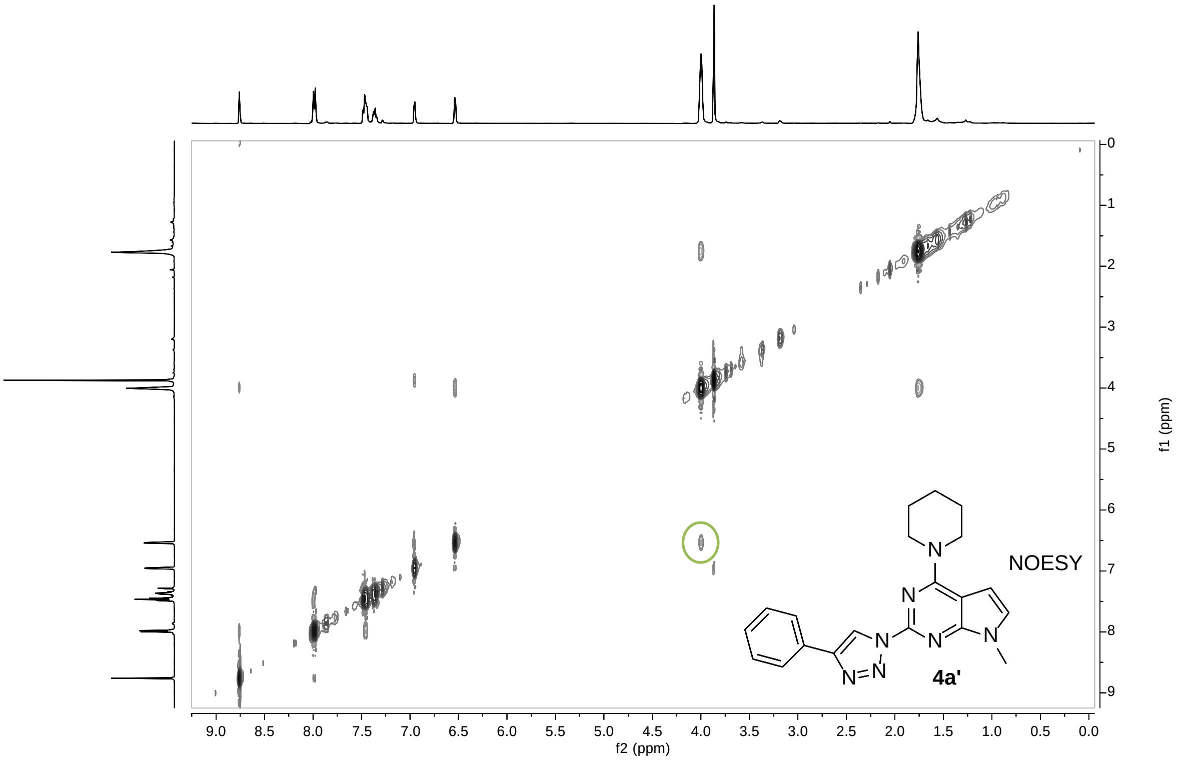
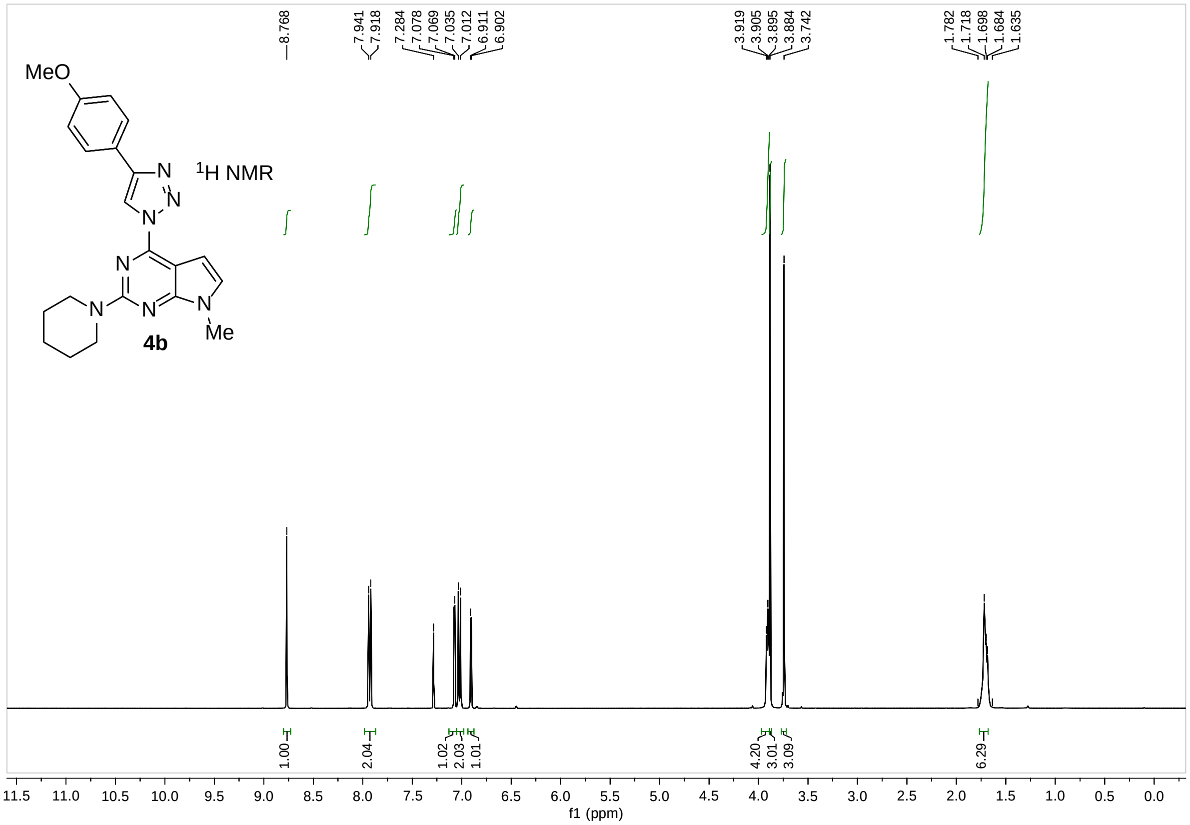
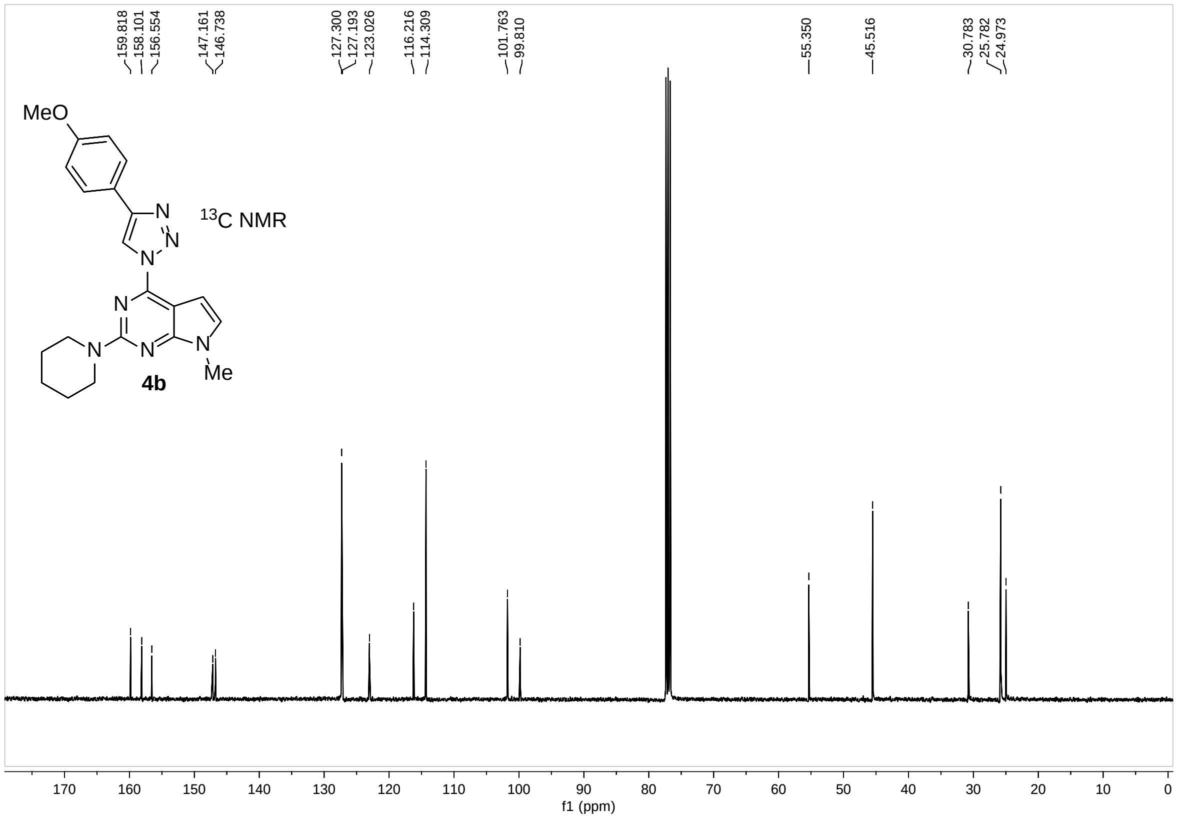
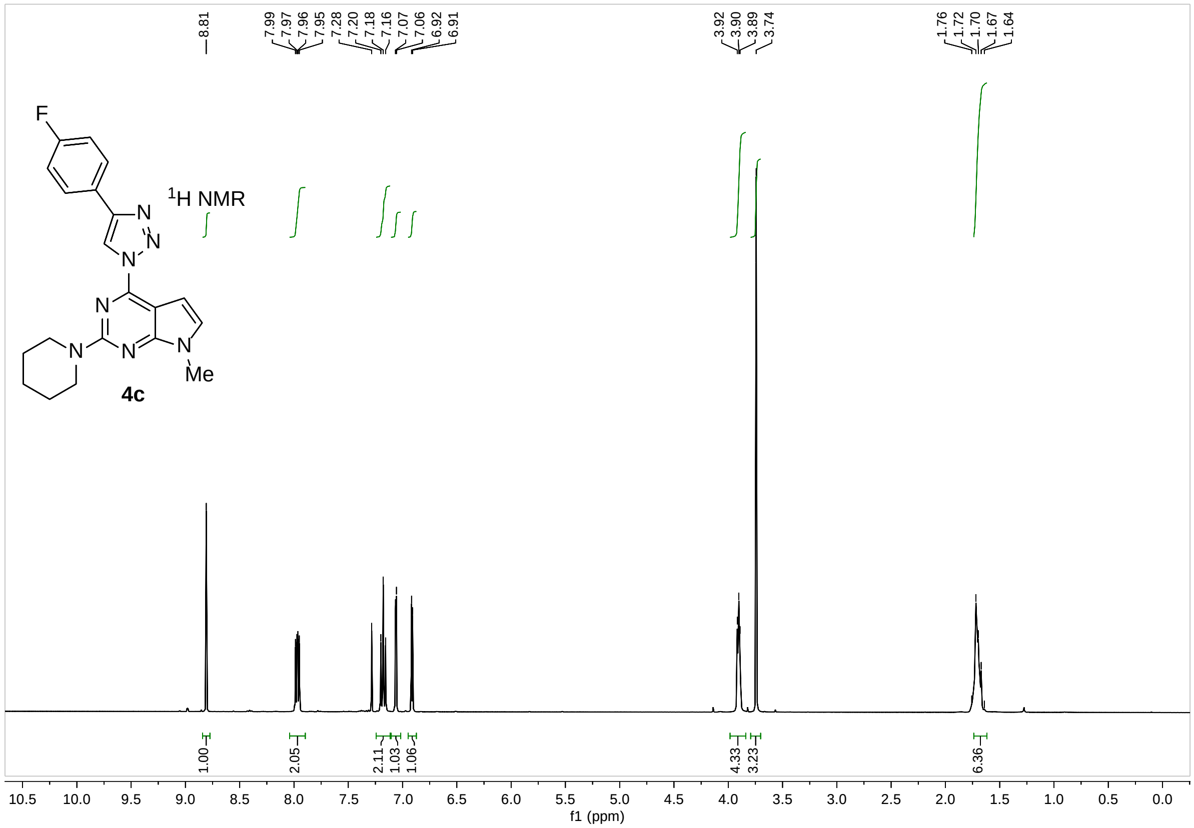
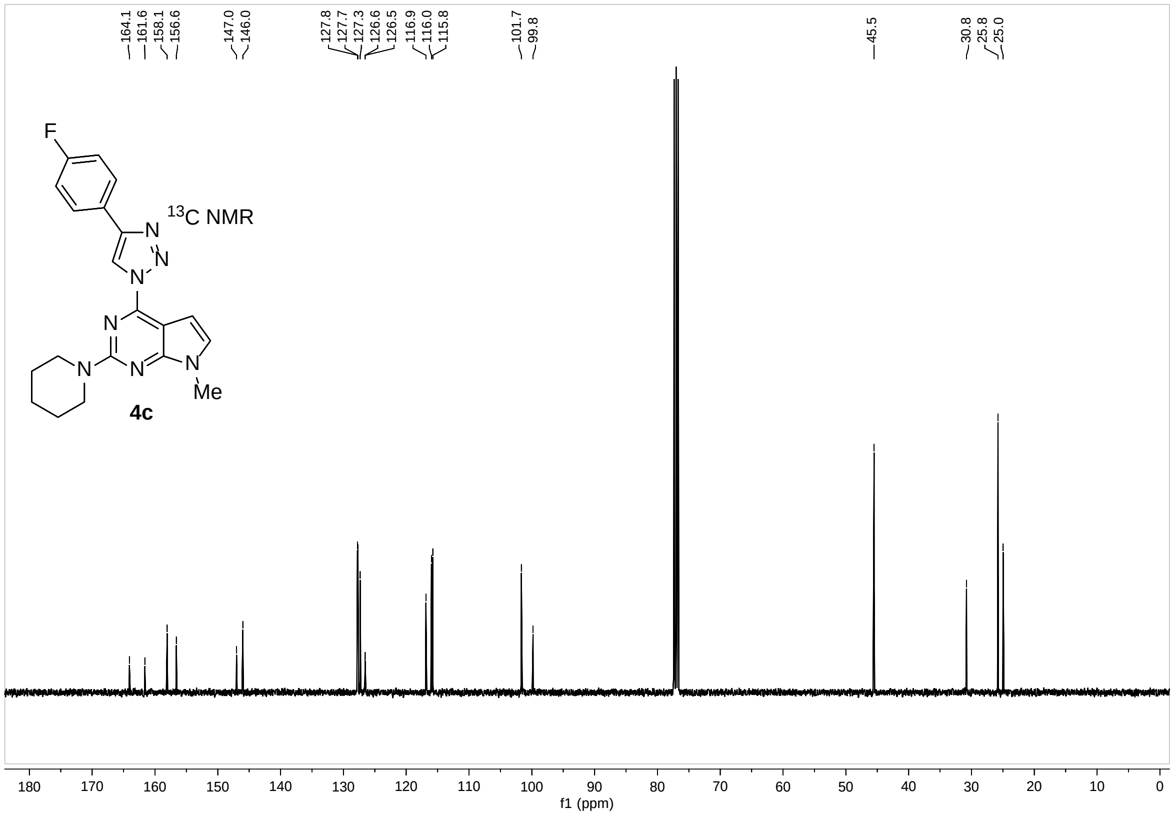
128.1
126.0
125.2
118.6
101.9
101.5
47.1
31.7
25.9
24.6
^{13}C NMR
N
N
N
N
N
N=N
4a'
170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0
f1 (ppm)

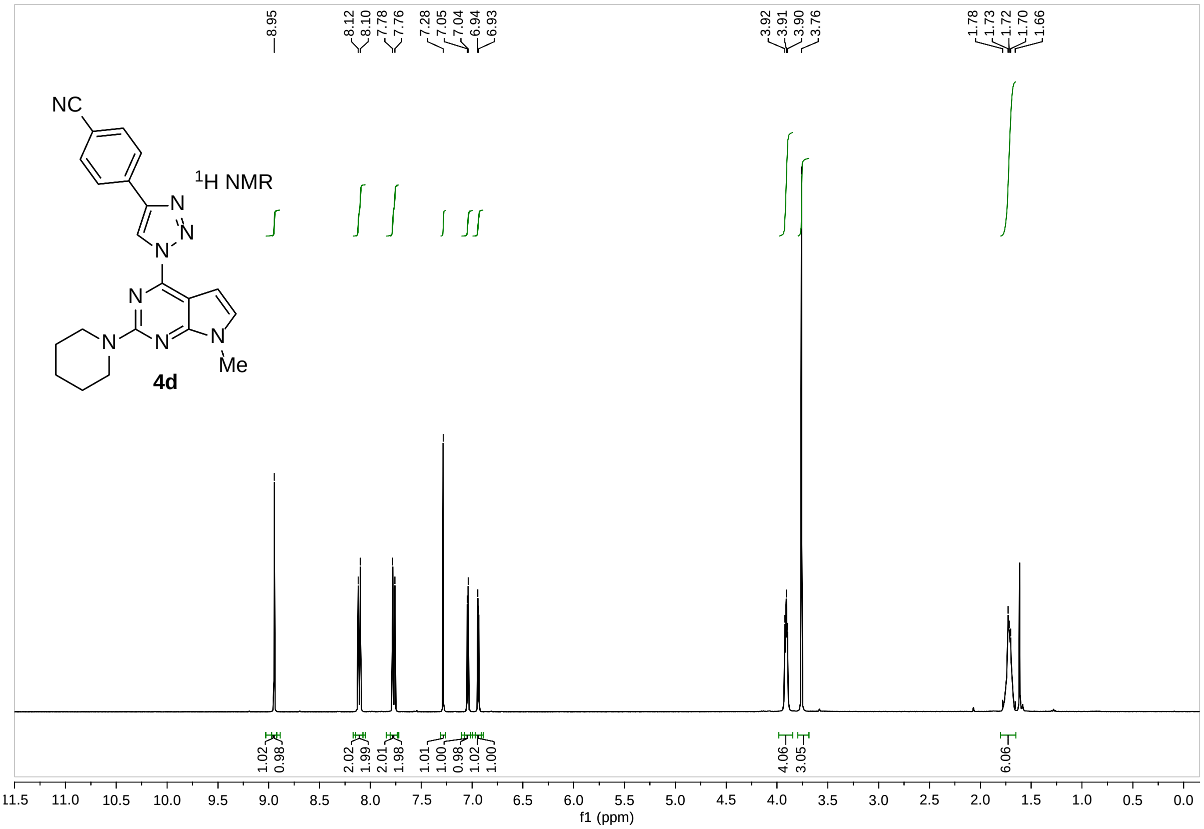

NOESY
4a'
N
N
N
N
N
N=N
f2 (ppm)
9.0 8.5 8.0 7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 0.5 0.0
f1 (ppm)
0 1 2 3 4 5 6 7 8 9

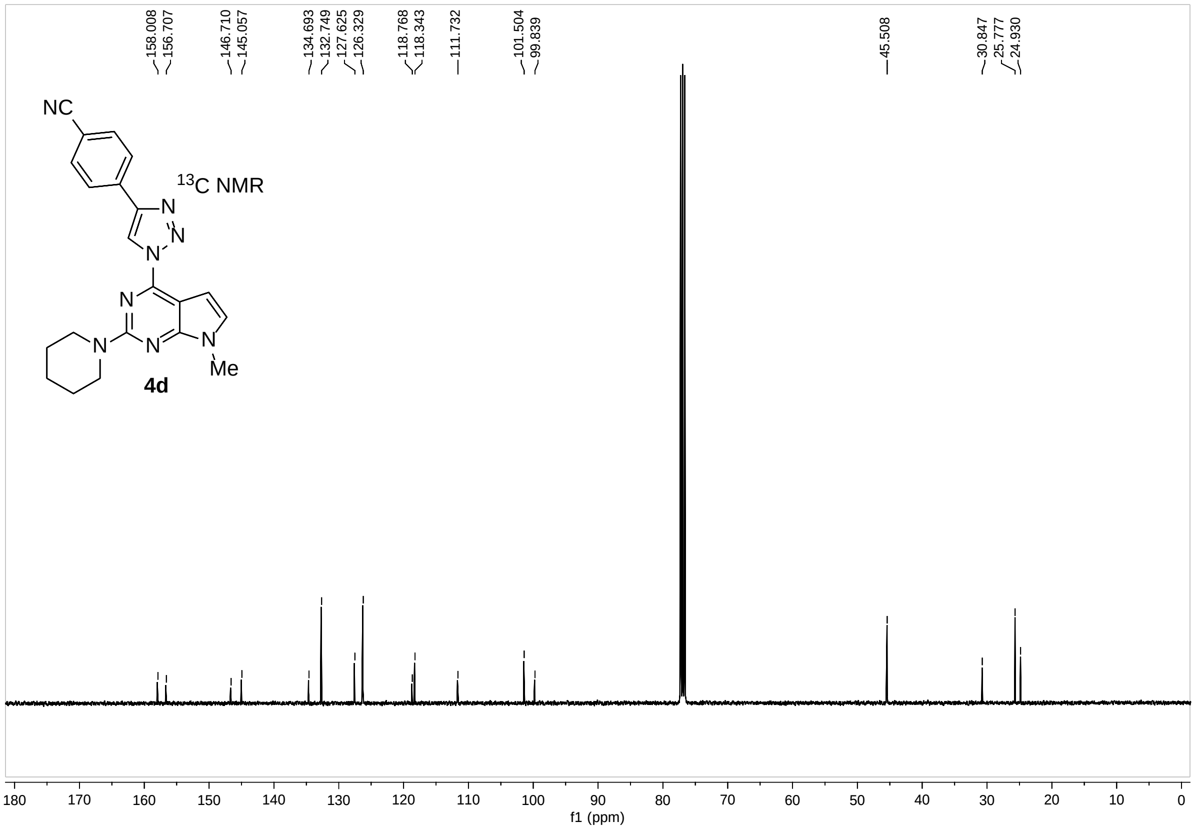

MeO
N
N
N
N
N
N
N
Me
4b
1H NMR
8.768
7.941
7.918
7.284
7.078
7.069
7.035
7.012
6.911
6.902
3.919
3.905
3.895
3.884
3.742
1.782
1.718
1.698
1.684
1.635
1.00
2.04
1.02
2.03
1.01
4.20
3.01
3.09
6.29
11.5 11.0 10.5 10.0 9.5 9.0 8.5 8.0 7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 0.5 0.0
f1 (ppm)

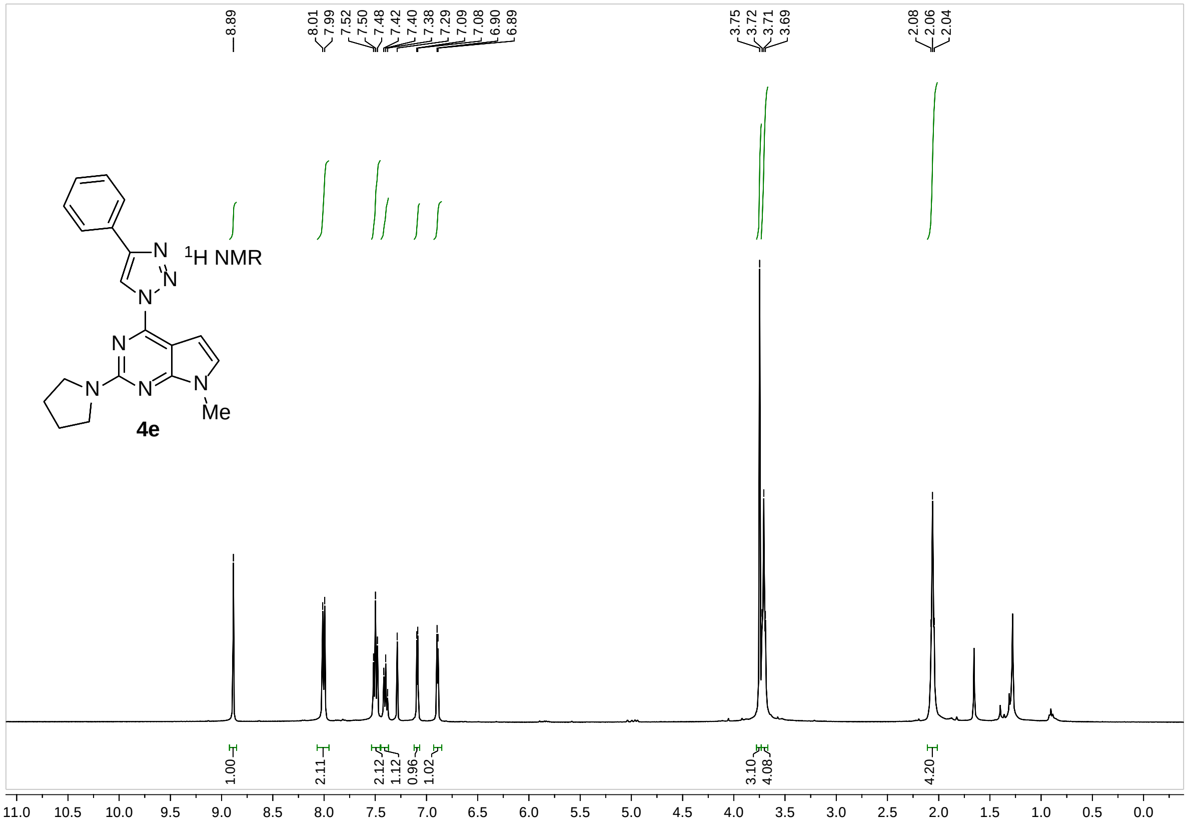

159.818
158.101
156.554
147.161
146.738
127.300
127.193
123.026
116.216
114.309
101.763
99.810
55.350
45.516
30.783
25.782
24.973
MeO
N
N
N
N
N
N
N
Me
4b
^{13}C NMR
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
f1 (ppm)

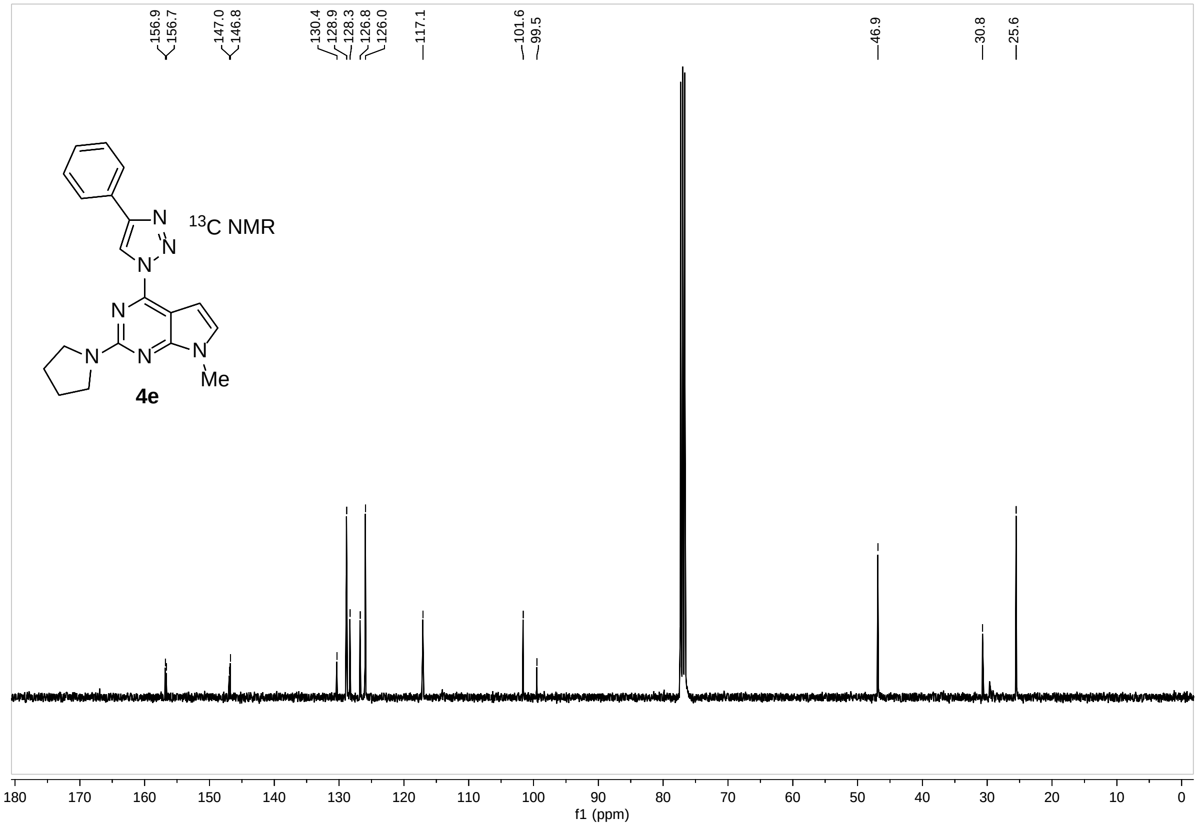

8.81
7.99
7.97
7.96
7.95
7.28
7.20
7.18
7.16
7.07
7.06
6.92
6.91
3.92
3.90
3.89
3.74
1.76
1.72
1.70
1.67
1.64
F
N
N
N
N
N
N
N
Me
4c
^{1}H NMR
1.00
2.05
2.11
1.03
1.06
4.33
3.23
6.36
10.5
10.0
9.5
9.0
8.5
8.0
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
0.5
0.0
f1 (ppm)

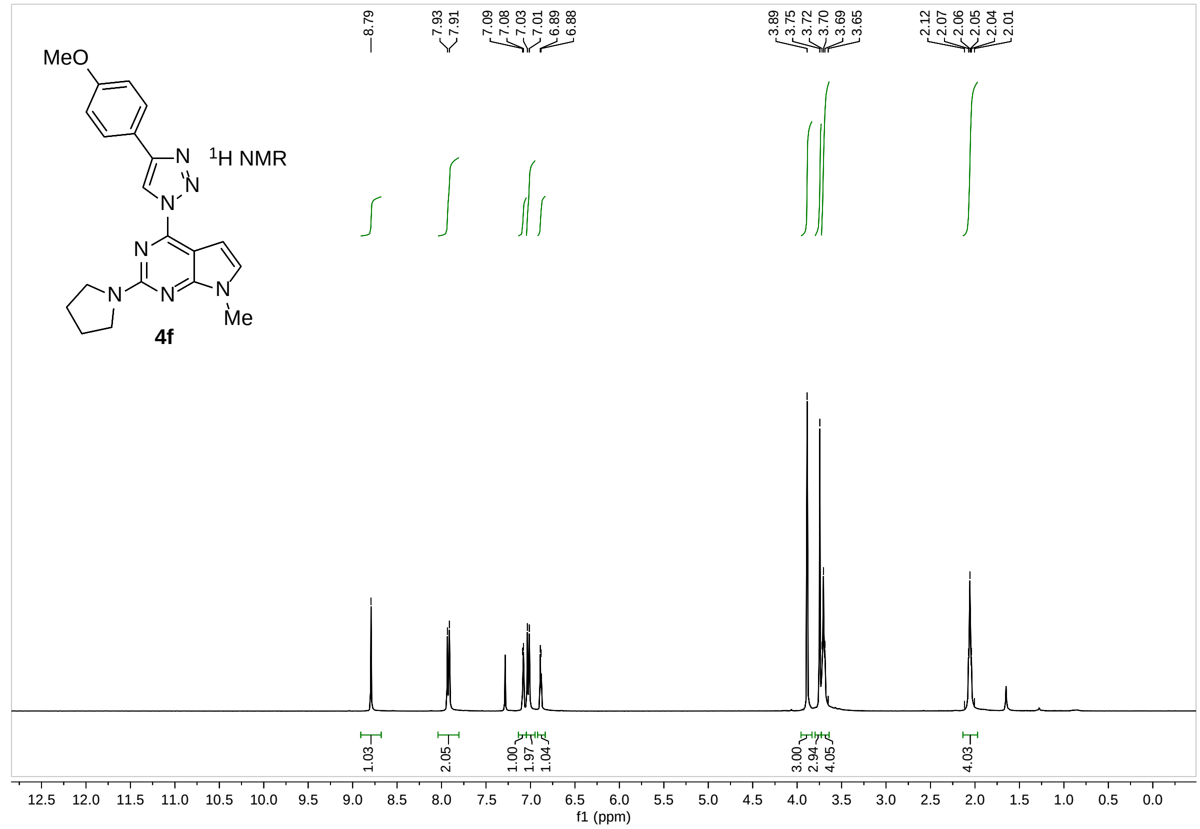

164.1
161.6
158.1
156.6
147.0
146.0
127.8
127.7
127.3
126.6
126.5
116.9
116.0
115.8
101.7
99.8
45.5
30.8
25.8
25.0
F
N
N
N
N
N
N
N
Me
4c
^{13}C NMR
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
f1 (ppm)

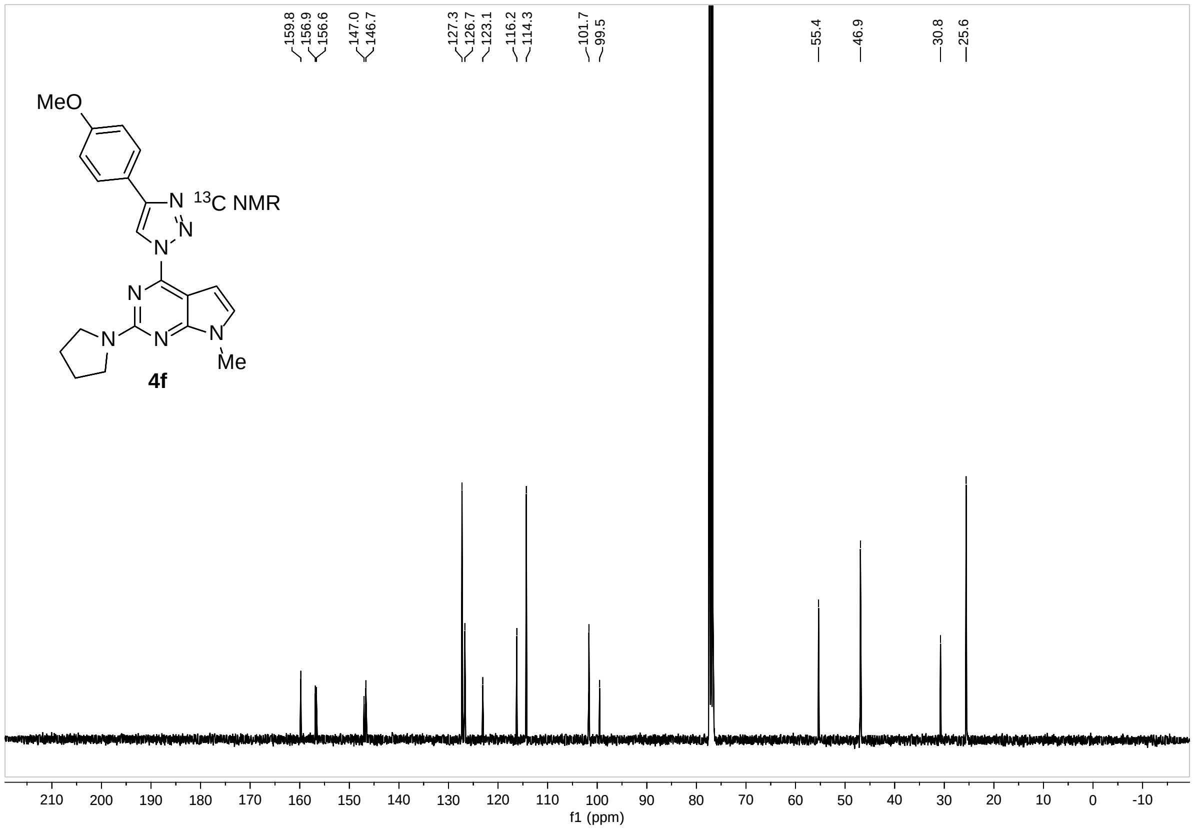

8.95
8.12
8.10
7.78
7.76
7.28
7.05
7.04
6.94
6.93
3.92
3.91
3.90
3.76
1.78
1.73
1.72
1.70
1.66
NC
N
N
N
N
N
N
N
Me
4d
^{1}H NMR
1.02
0.98
2.02
1.99
2.01
1.98
1.01
1.00
0.98
1.02
1.00
4.06
3.05
6.06
11.5
11.0
10.5
10.0
9.5
9.0
8.5
8.0
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
0.5
0.0
f1 (ppm)


158.008
156.707
146.710
145.057
134.693
132.749
127.625
126.329
118.768
118.343
111.732
101.504
99.839
45.508
30.847
25.777
24.930
NC
N
N
N
N
N
N
N
Me
4d
13C NMR
180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0
f1 (ppm)

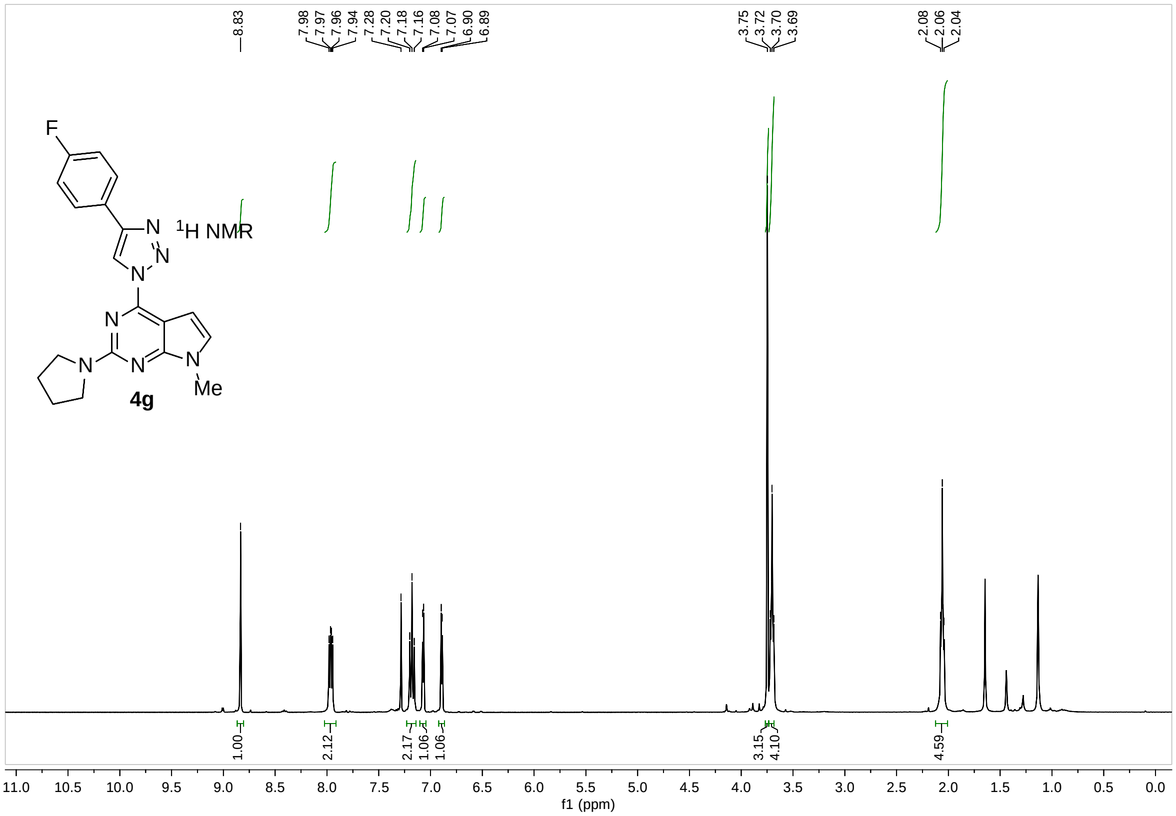

8.89
8.01
7.99
7.52
7.50
7.48
7.42
7.40
7.38
7.29
7.09
7.08
6.90
6.89
3.75
3.72
3.71
3.69
2.08
2.06
2.04
1H NMR
N
N
N
N
N
N
N
Me
4e
1.00
2.11
2.12
1.12
0.96
1.02
3.10
4.08
4.20
11.0 10.5 10.0 9.5 9.0 8.5 8.0 7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 0.5 0.0


156.9
156.7
147.0
146.8
130.4
128.9
128.3
126.8
126.0
117.1
101.6
99.5
46.9
30.8
25.6
^{13}C NMR
N
N
N
N
N
N
N
Me
4e
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
f1 (ppm)

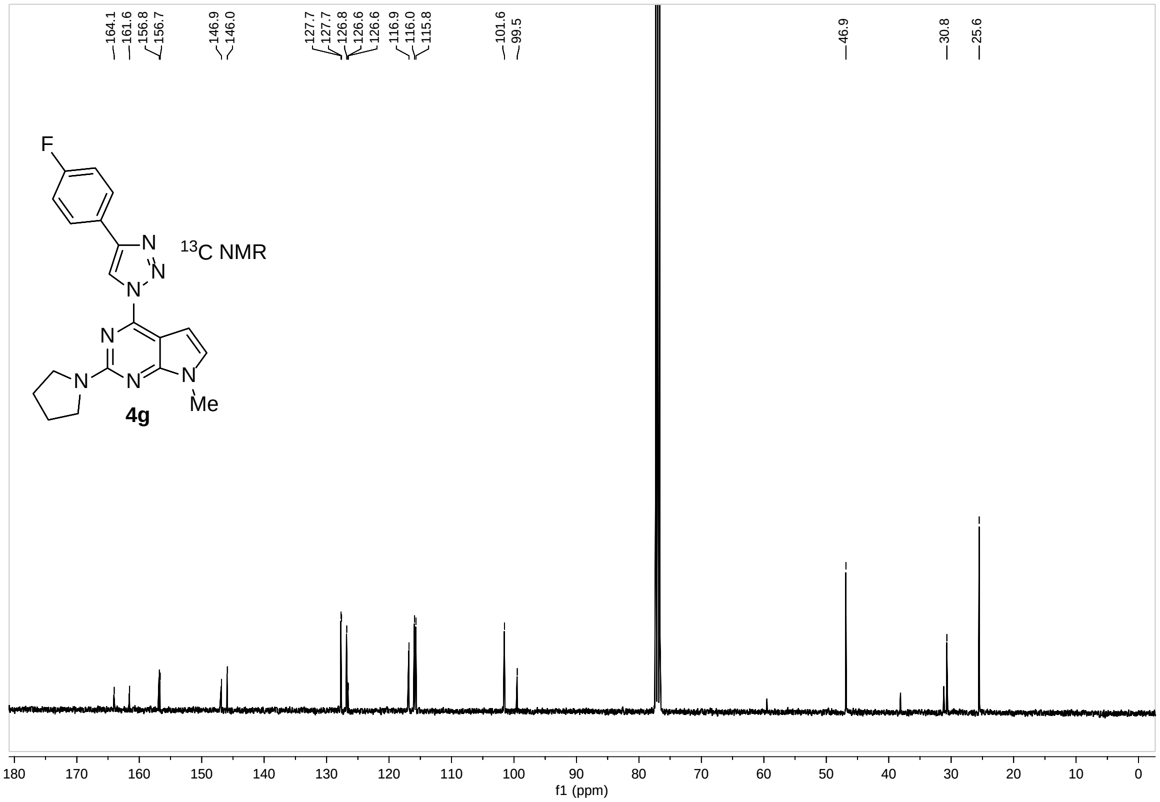

8.79
7.93
7.91
7.09
7.08
7.03
7.01
6.89
6.88
3.89
3.75
3.72
3.70
3.69
3.65
2.12
2.07
2.06
2.05
2.04
2.01
MeO
^{1}H NMR
N
N
N
N
N
N
N
Me
4f
1.03
2.05
1.00
1.97
1.04
3.00
2.94
4.05
4.03
12.5
12.0
11.5
11.0
10.5
10.0
9.5
9.0
8.5
8.0
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
0.5
0.0
f1 (ppm)

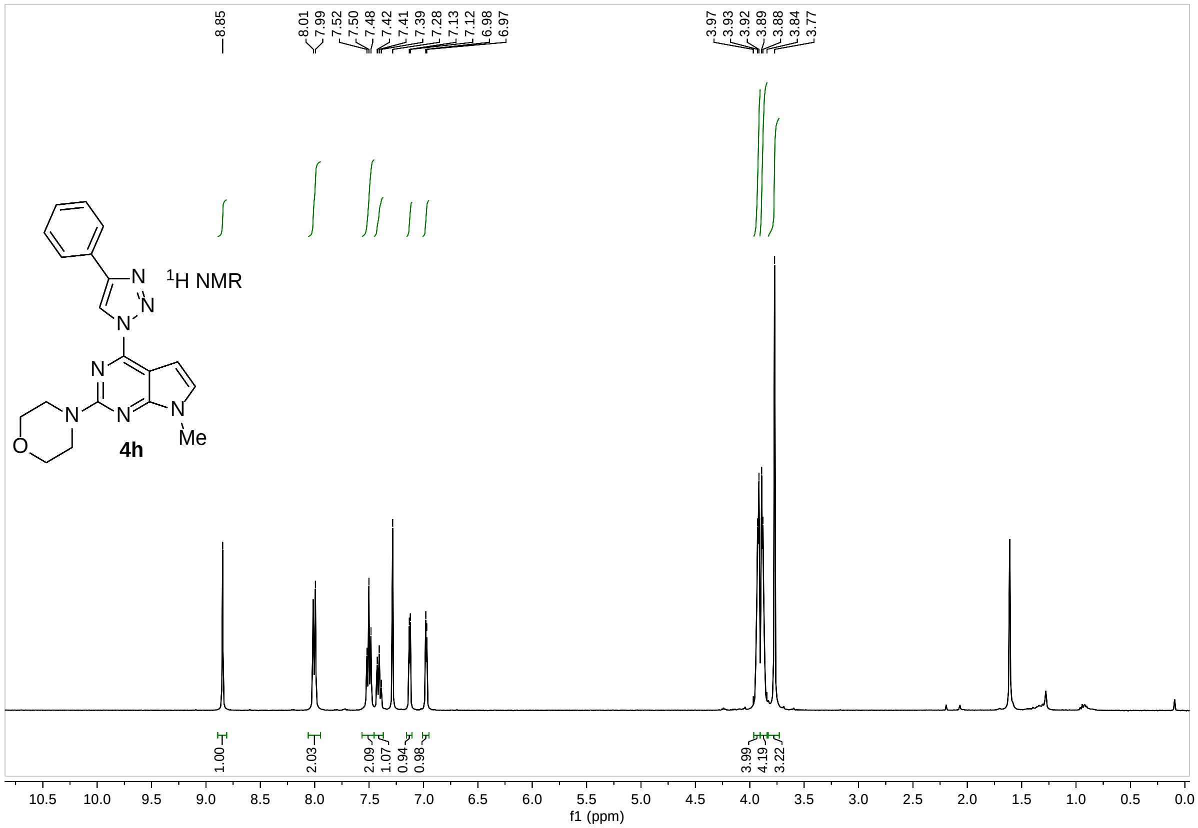

159.8
156.9
156.6
147.0
146.7
127.3
126.7
123.1
116.2
114.3
101.7
99.5
55.4
46.9
30.8
25.6
MeO
N
N
N
N
N
N
N
Me
^{13}C NMR
210 200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0 -10
f1 (ppm)

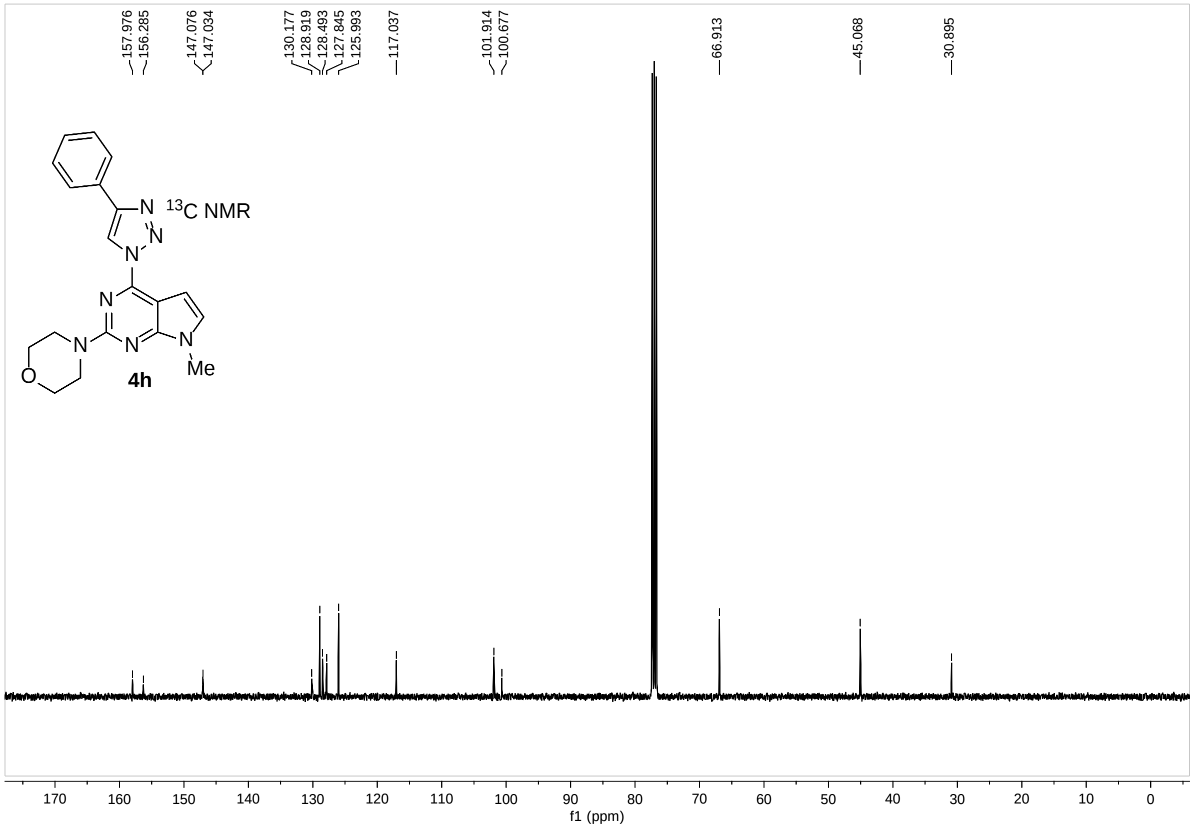
4f


8.83
7.98
7.97
7.96
7.94
7.28
7.20
7.18
7.16
7.08
7.07
6.90
6.89
3.75
3.72
3.70
3.69
2.08
2.06
2.04
F
N
N
N
N
N
N
N
Me
^{1}H NMR
1.00
2.12
2.17
1.06
1.06
3.15
4.10
4.59
11.0 10.5 10.0 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 0.5 0.0
f1 (ppm)

4g


164.1
161.6
156.8
156.7
146.9
146.0
127.7
127.7
126.8
126.6
126.6
116.9
116.0
115.8
101.6
99.5
46.9
30.8
25.6
F
N
N
N
N
N
N
Me
4g
^{13}C NMR
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
f1 (ppm)


8.85
8.01
7.99
7.52
7.50
7.48
7.42
7.41
7.39
7.28
7.13
7.12
6.98
6.97
3.97
3.93
3.92
3.89
3.88
3.84
3.77
N
N
N
N
N
N
O
Me
4h
^{1}H NMR
1.00
2.03
2.09
1.07
0.94
0.98
3.99
4.19
3.22
10.5
10.0
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
0.5
0.0
f1 (ppm)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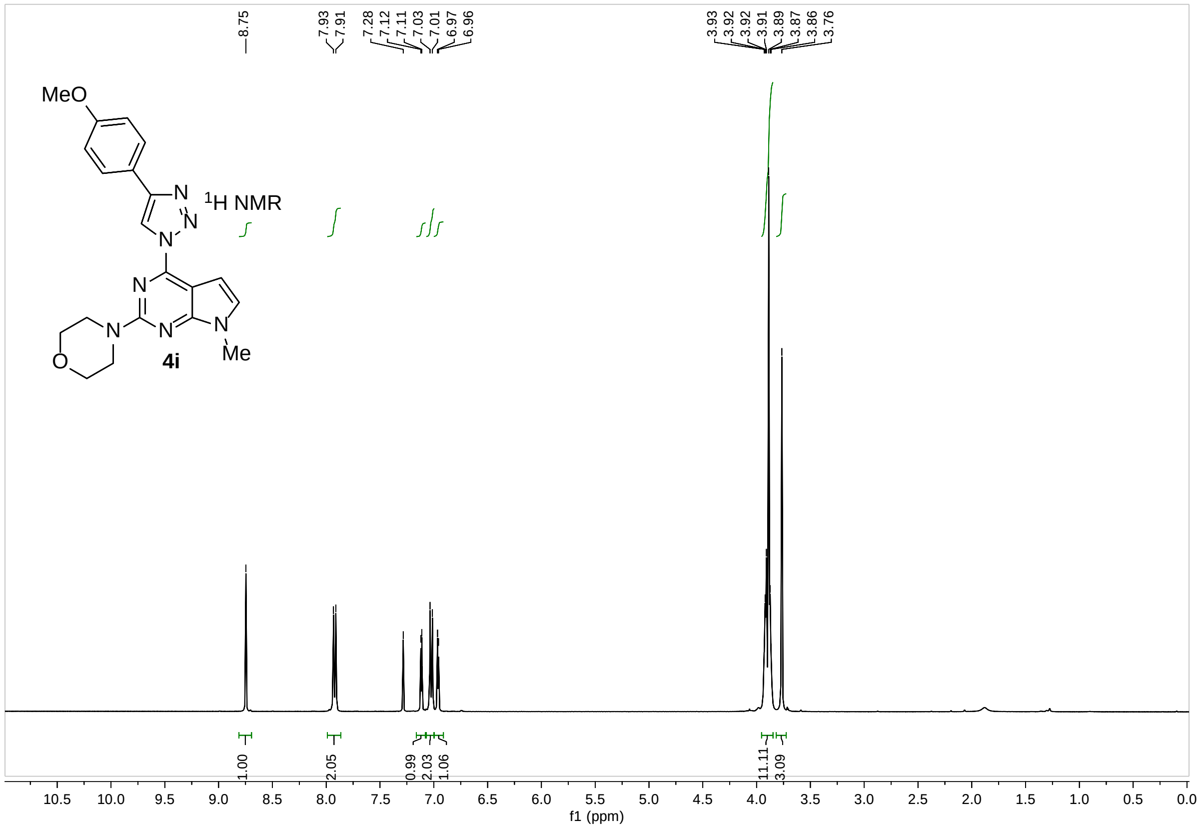
156.285
147.076
147.034
130.177
128.919
128.493
127.845
125.993
117.037
101.914
100.677
66.913
45.068
30.895
^{13}C NMR
N
N
N
N
N
N
N
O
Me
4h
170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0
f1 (ppm)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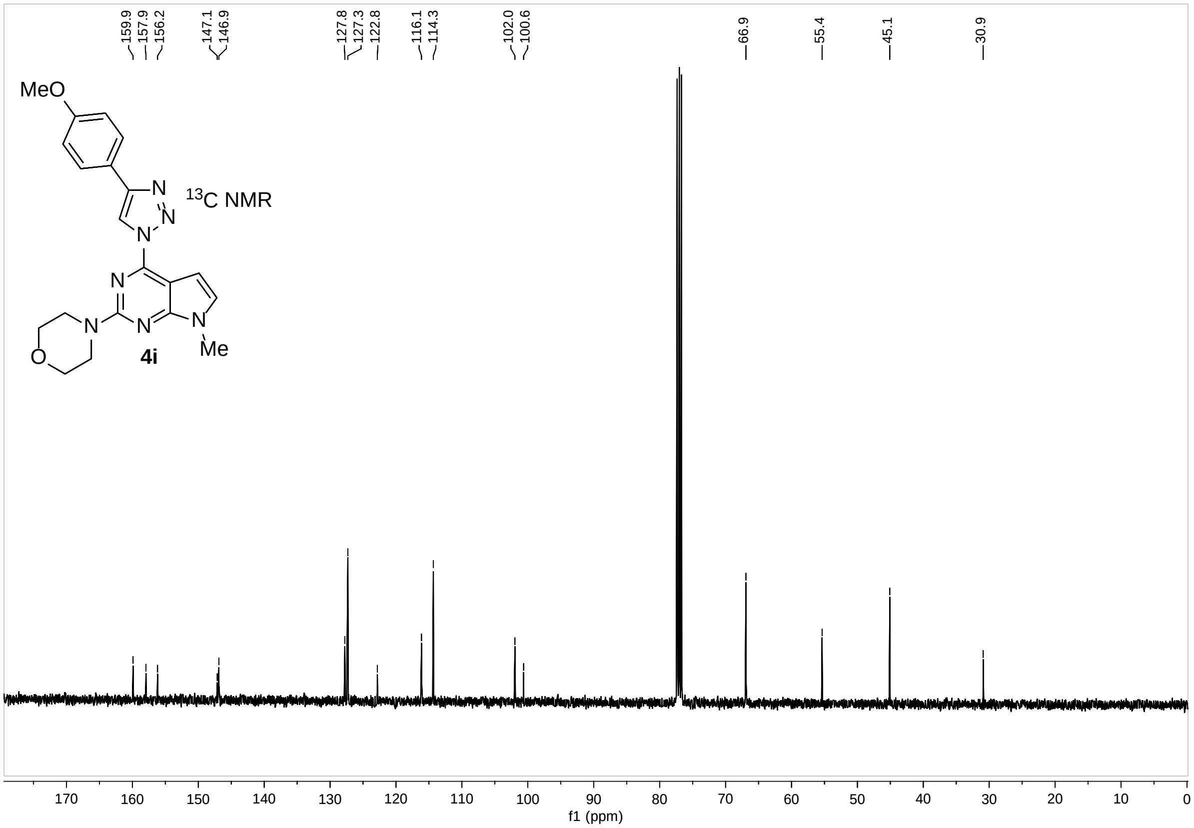
7.03
7.01
6.97
6.96
3.93
3.92
3.92
3.91
3.89
3.87
3.86
3.76
MeO
^{1}H NMR
N
N
N
N
N
N
N
O
Me
4i
1.00
2.05
0.99
2.03
1.06
11.11
3.09
10.5 10.0 9.5 9.0 8.5 8.0 7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 0.5 0.0
f1 (ppm)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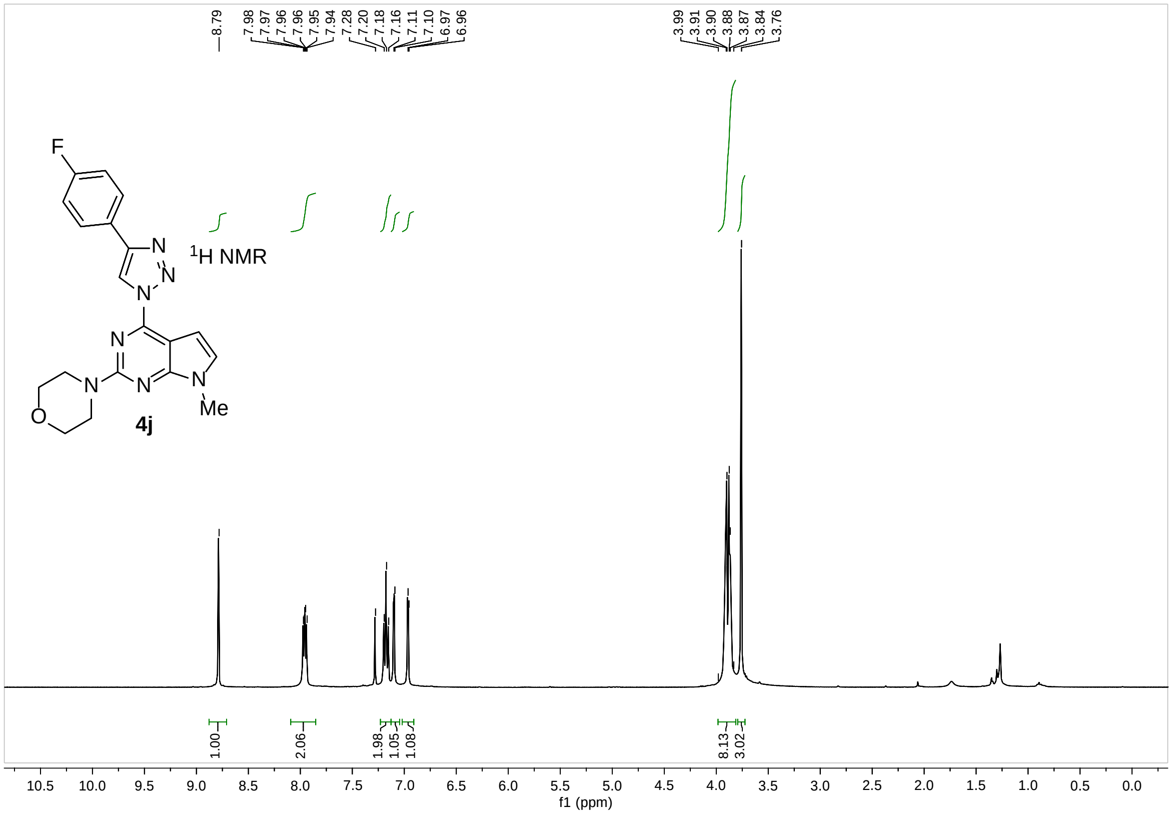
157.9
156.2
147.1
146.9
127.8
127.3
122.8
116.1
114.3
102.0
100.6
66.9
55.4
45.1
30.9
MeO
N
N
N
N
N
N
N
O
Me
4i
13C NMR
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
f1 (ppm)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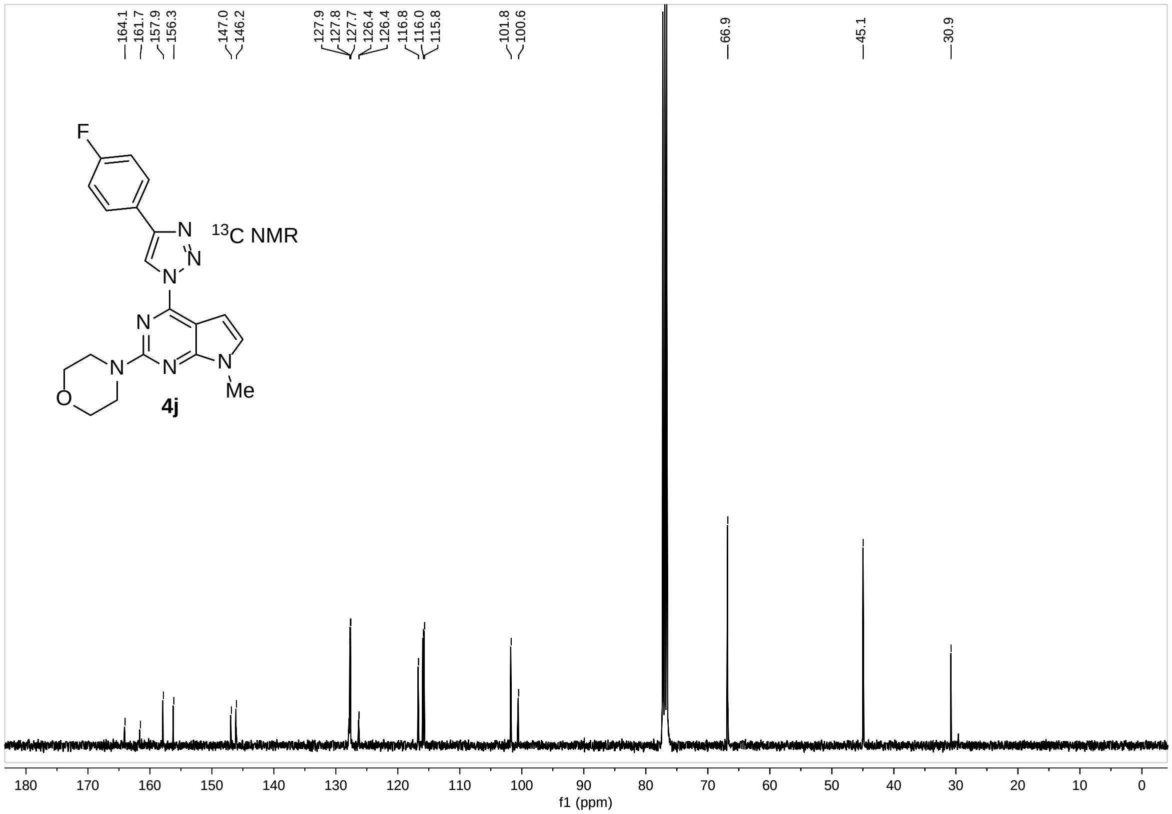
7.96
7.96
7.95
7.94
7.28
7.20
7.18
7.16
7.11
7.10
6.97
6.96
3.99
3.91
3.90
3.88
3.87
3.84
3.76
F
N
N
N
N
N
N
N
O
Me
4j
1H NMR
1.00
2.06
1.98
1.05
1.08
8.13
3.02
10.5
10.0
9.5
9.0
8.5
8.0
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
0.5
0.0
f1 (ppm)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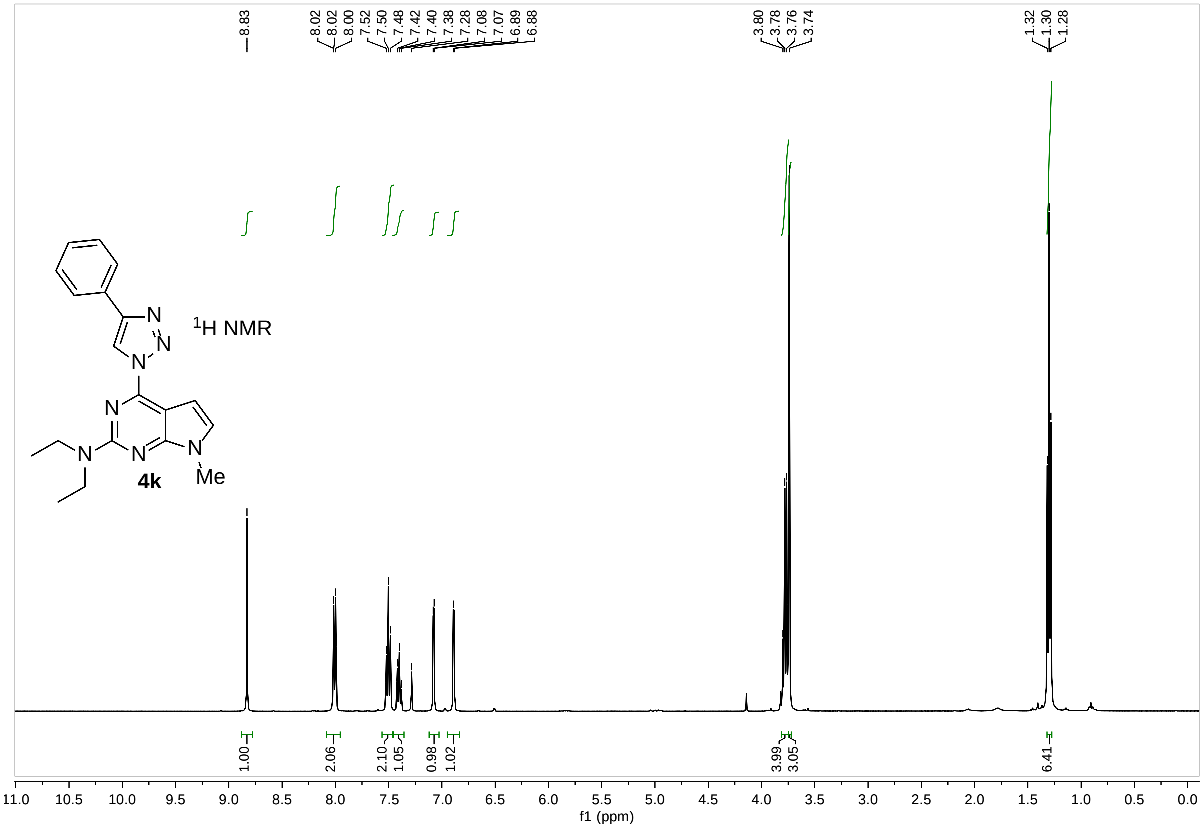
161.7
157.9
156.3
147.0
146.2
127.9
127.8
127.7
126.4
126.4
116.8
116.0
115.8
101.8
100.6
66.9
45.1
30.9
F
N
N
N
N
N
N
O
N
Me
4j
^{13}C NMR
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
f1 (ppm)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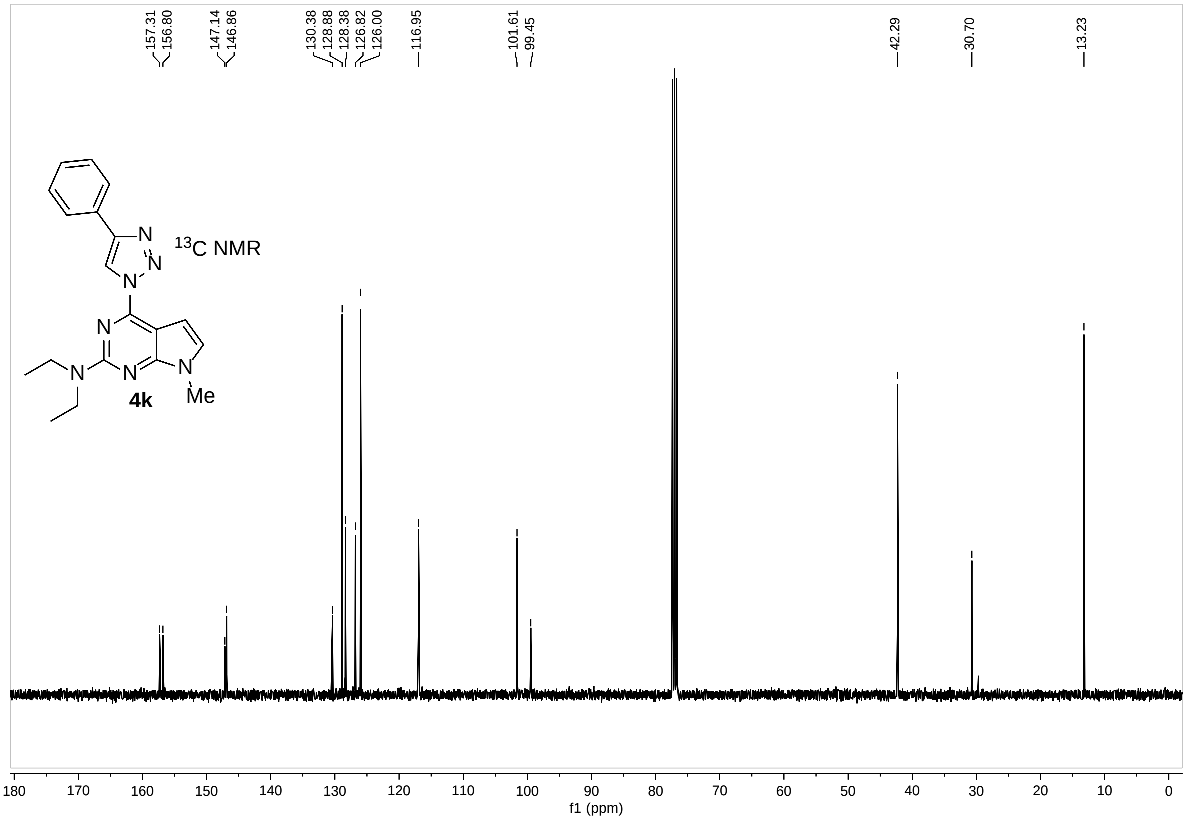
8.02
8.00
7.52
7.50
7.48
7.42
7.40
7.38
7.28
7.08
7.07
6.89
6.88
3.80
3.78
3.76
3.74
1.32
1.30
1.28
N
N
N
N
N
N
N
Me
4k
^{1}H NMR
1.00
2.06
2.10
1.05
0.98
1.02
3.99
3.05
6.41
11.0
10.5
10.0
9.5
9.0
8.5
8.0
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
0.5
0.0
f1 (ppm)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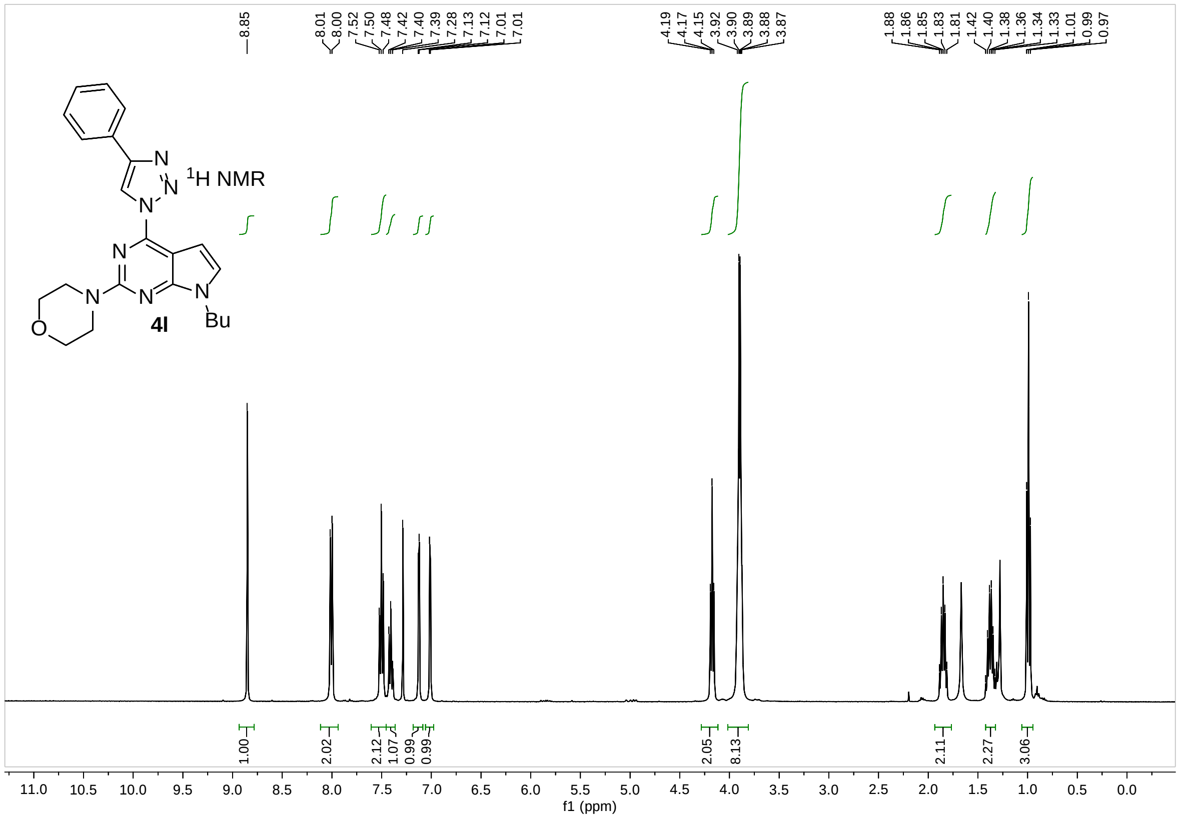
156.80
147.14
146.86
130.38
128.88
128.38
126.82
126.00
116.95
101.61
99.45
42.29
30.70
13.23
13C NMR
4k
Me
180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0
f1 (ppm)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8.00
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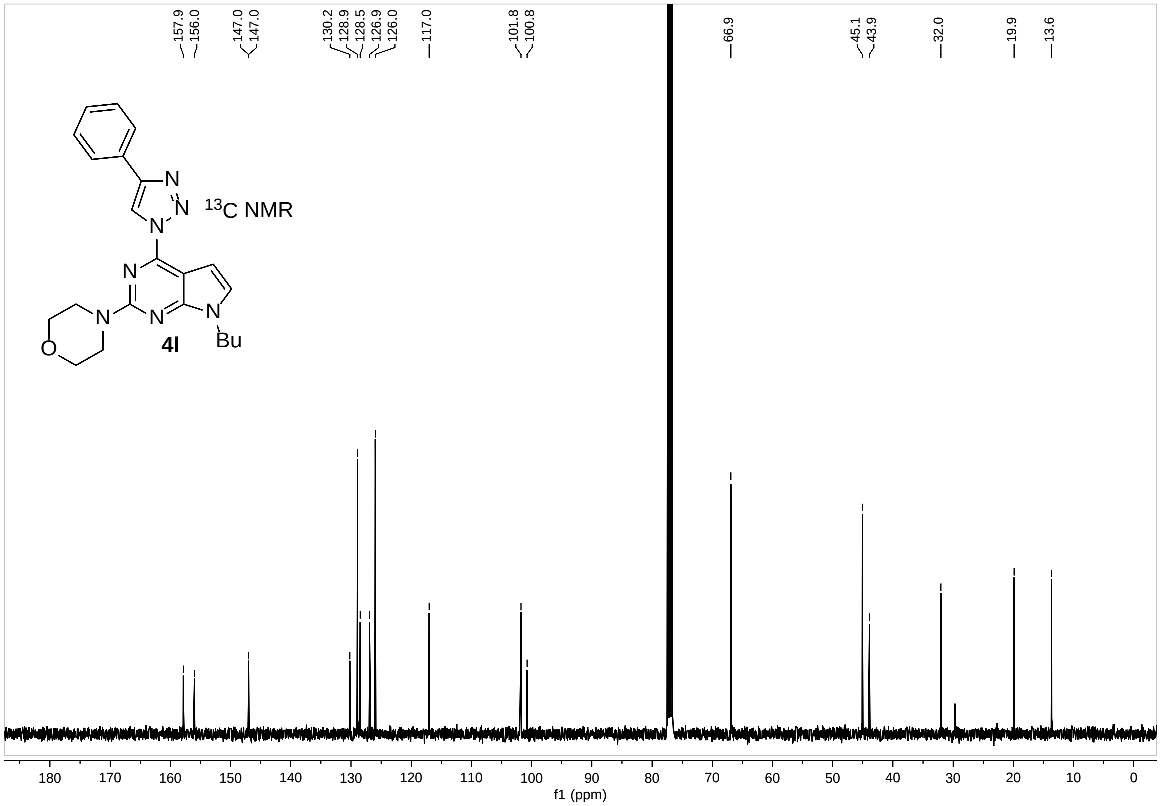
7.50
7.48
7.42
7.40
7.39
7.28
7.13
7.12
7.01
7.01
4.19
4.17
4.15
3.92
3.90
3.89
3.88
3.87
1.88
1.86
1.85
1.83
1.81
1.42
1.40
1.38
1.36
1.34
1.33
1.01
0.99
0.97
1H NMR
4l
Bu
1.00
2.02
2.12
1.07
0.99
0.99
2.05
8.13
2.11
2.27
3.06
11.0 10.5 10.0 9.5 9.0 8.5 8.0 7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 0.5 0.0
f1 (ppm)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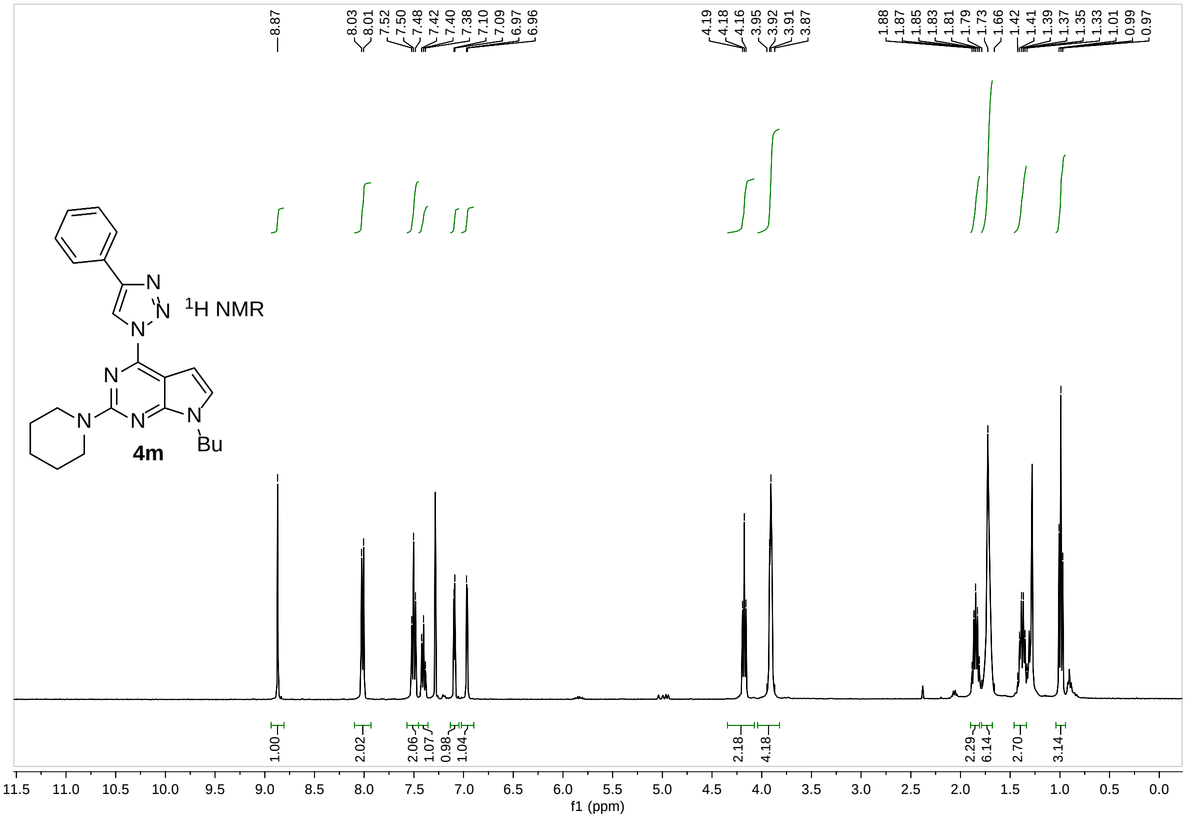
147.0
130.2
128.9
128.5
126.9
126.0
117.0
101.8
100.8
66.9
45.1
43.9
32.0
19.9
13.6
^{13}C NMR
N
N
N
N
N
N
O
N
4l
Bu
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
f1 (ppm)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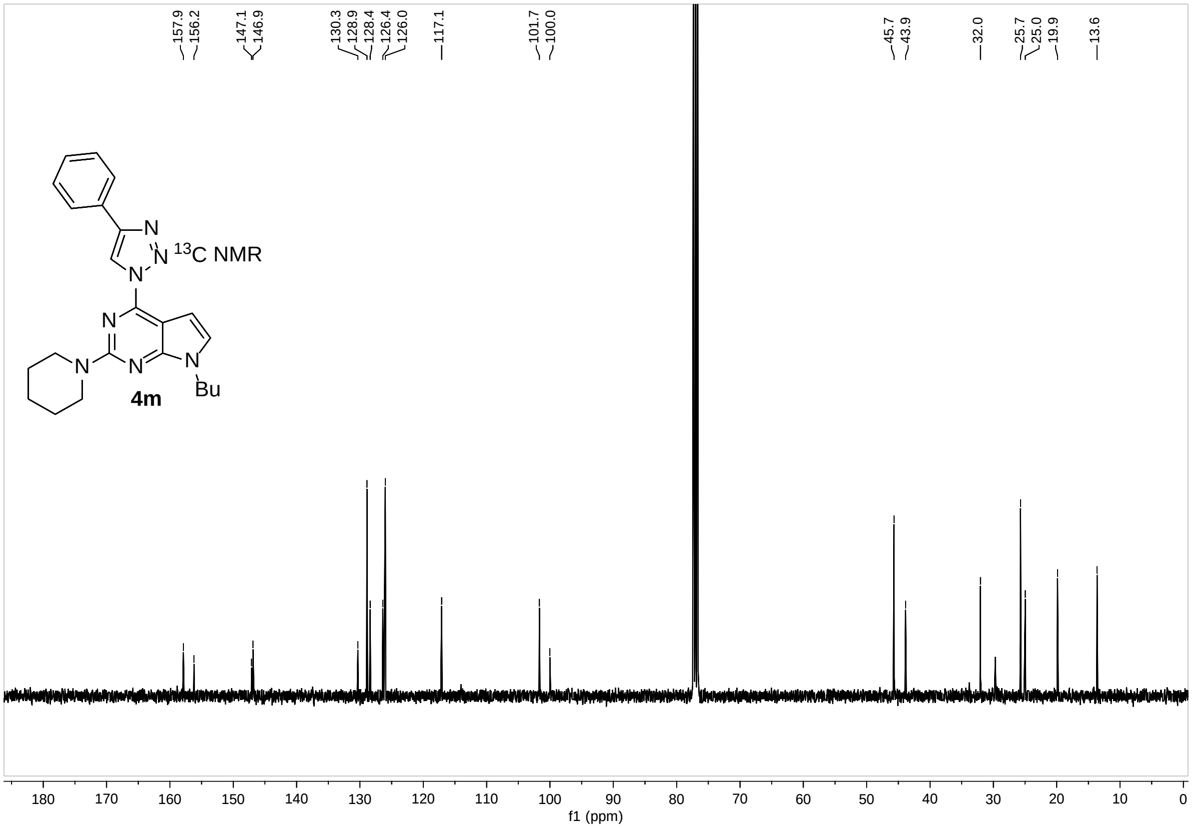
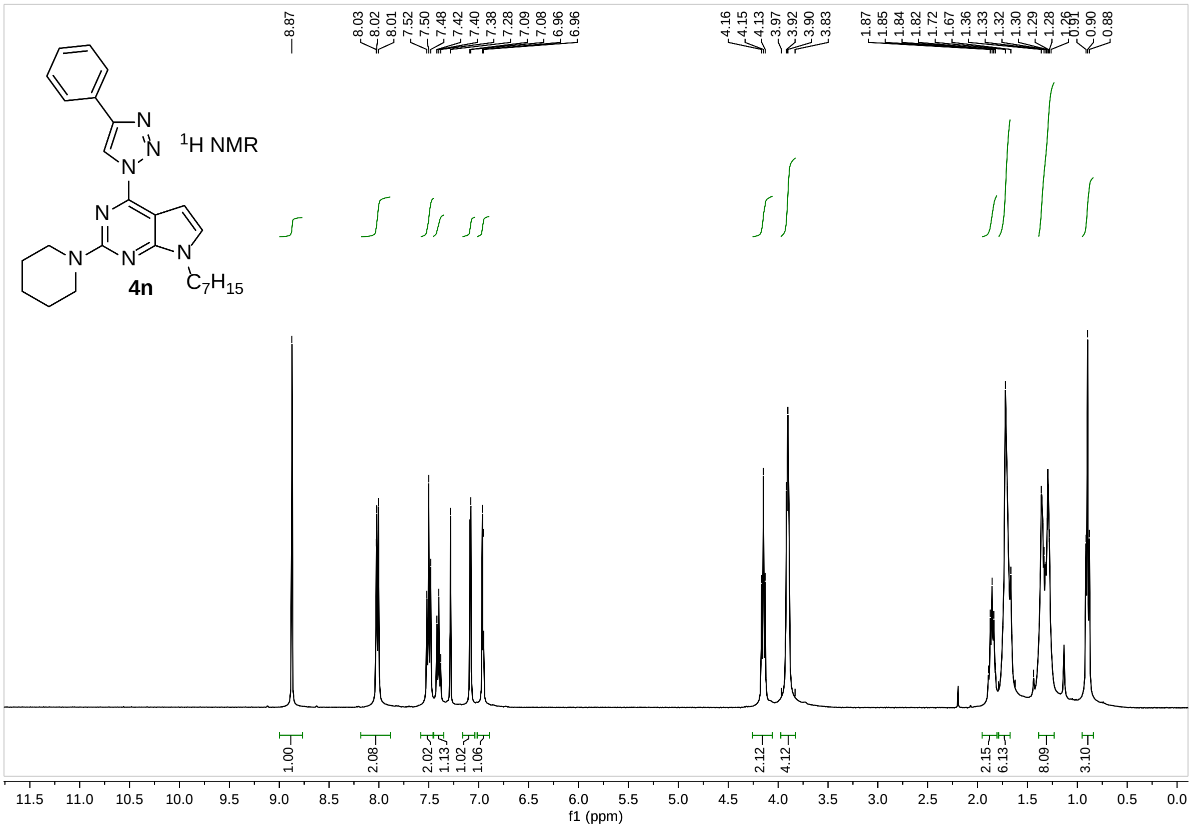
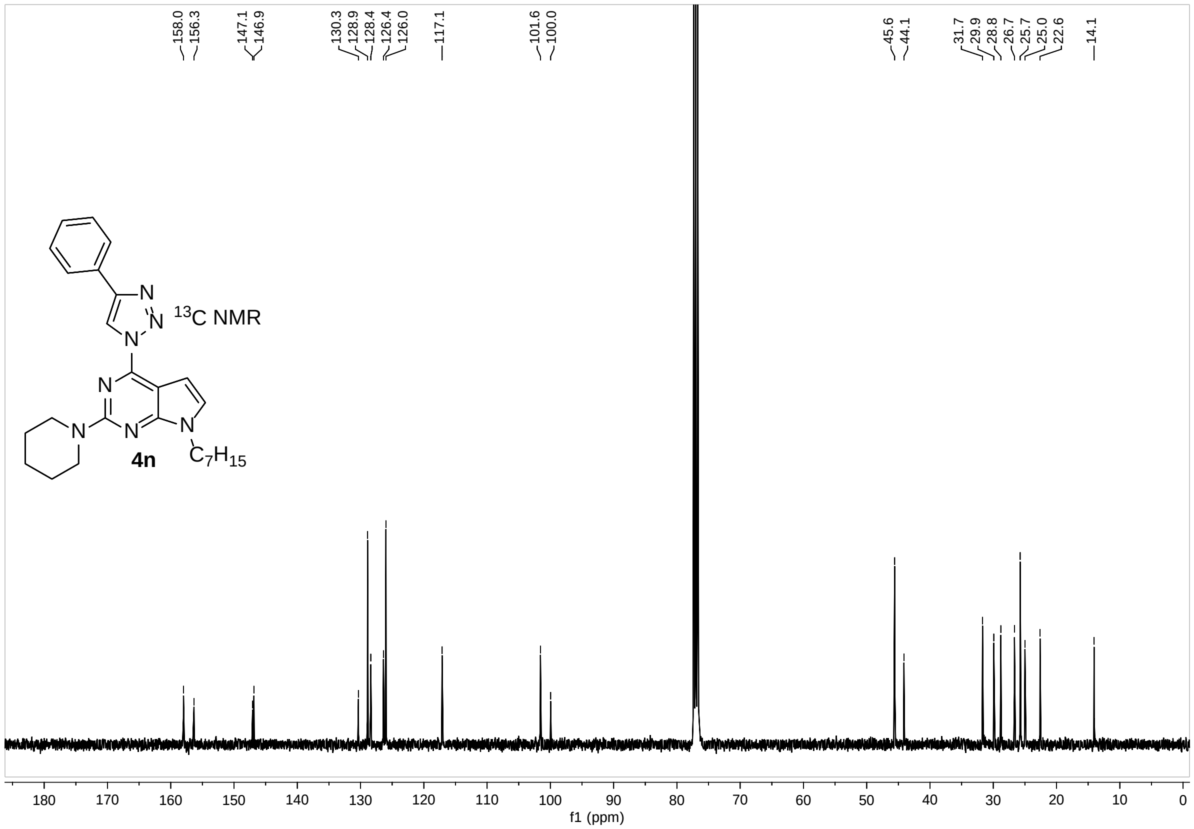
8.01
7.52
7.50
7.48
7.42
7.40
7.38
7.10
7.09
6.97
6.96
4.19
4.18
4.16
3.95
3.92
3.91
3.87
1.88
1.87
1.85
1.83
1.81
1.79
1.73
1.66
1.42
1.41
1.39
1.37
1.35
1.33
1.01
0.99
0.97
^{1}H NMR
N
N
N
N
N
N
N
4m
Bu
1.00
2.02
2.06
1.07
0.98
1.04
2.18
4.18
2.29
6.14
2.70
3.14
11.5
11.0
10.5
10.0
9.5
9.0
8.5
8.0
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
0.5
0.0
f1 (ppm)


157.9
156.2
147.1
146.9
130.3
128.9
128.4
126.4
126.0
117.1
101.7
100.0
45.7
43.9
32.0
25.7
25.0
19.9
13.6
N
N
N
N
N
N
^{13}C NMR
4m
Bu
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
f1 (ppm)


8.87
8.03
8.02
8.01
7.52
7.50
7.48
7.42
7.40
7.38
7.28
7.09
7.08
6.96
6.96
4.16
4.15
4.13
3.97
3.92
3.90
3.83
1.87
1.85
1.84
1.82
1.72
1.67
1.36
1.33
1.32
1.30
1.29
1.28
1.26
0.91
0.90
0.88
N
N
N
N
N
N
^{1}H NMR
4n
C_7H_{15}
1.00
2.08
2.02
1.13
1.02
1.06
2.12
4.12
2.15
6.13
8.09
3.10
11.5
11.0
10.5
10.0
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
0.5
0.0
f1 (ppm)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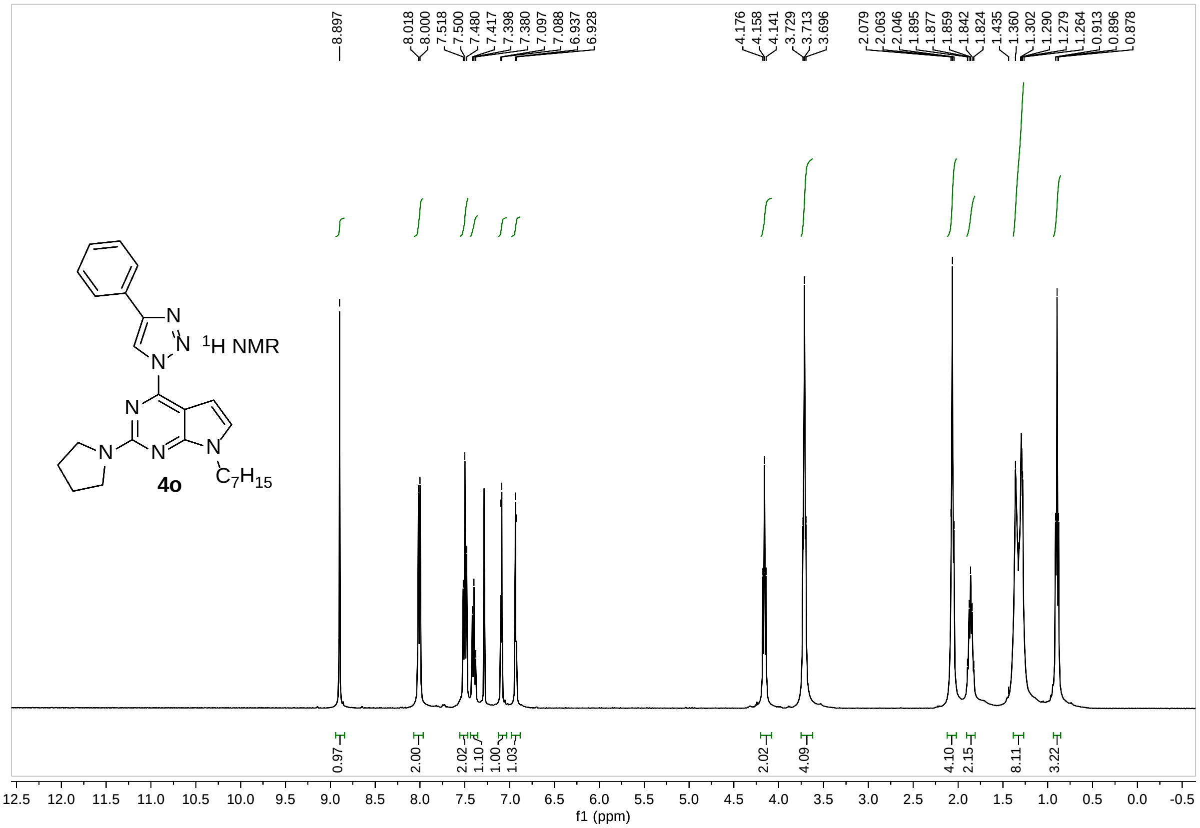
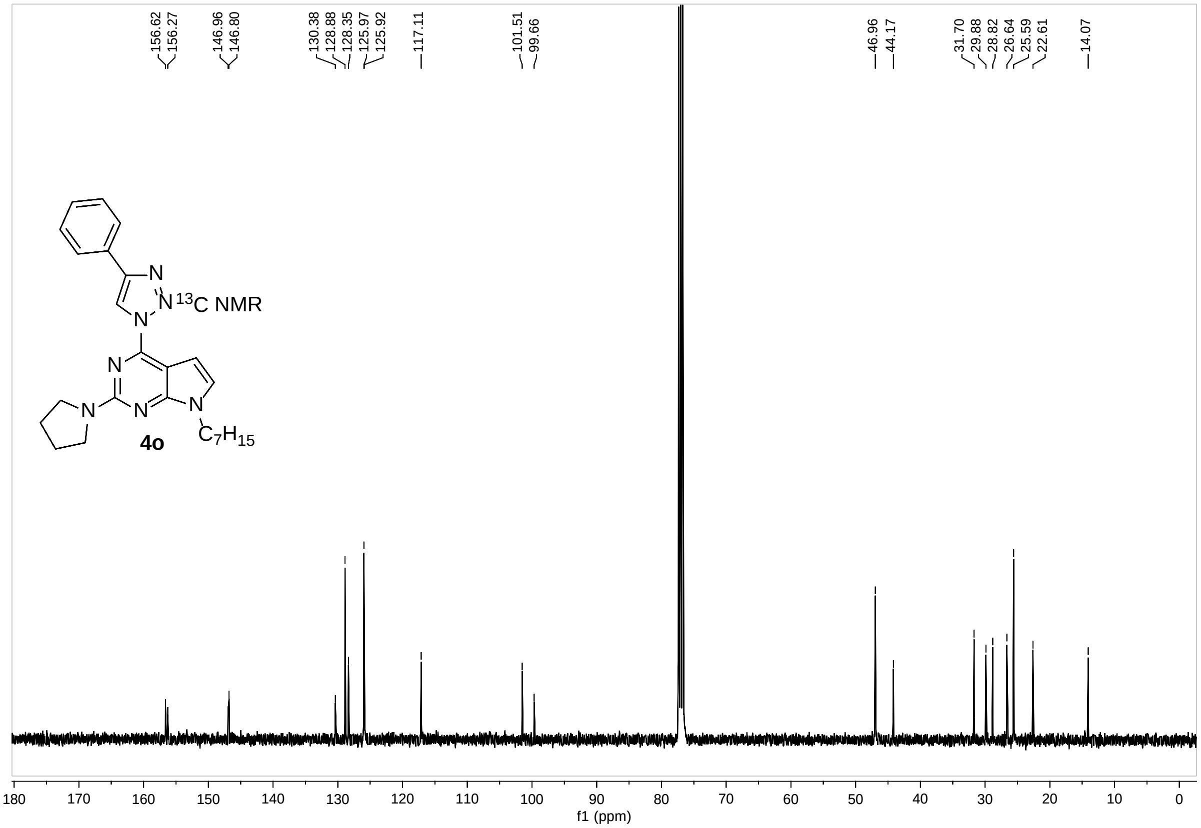
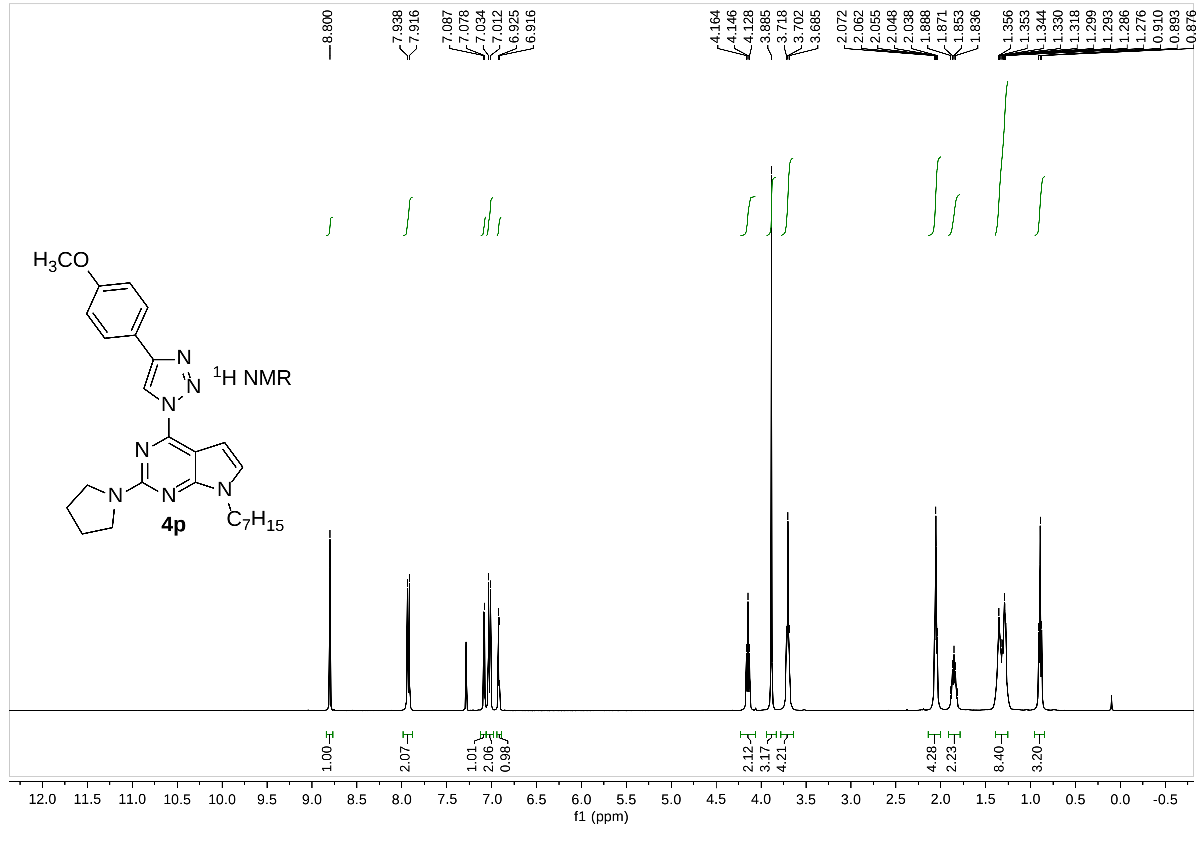
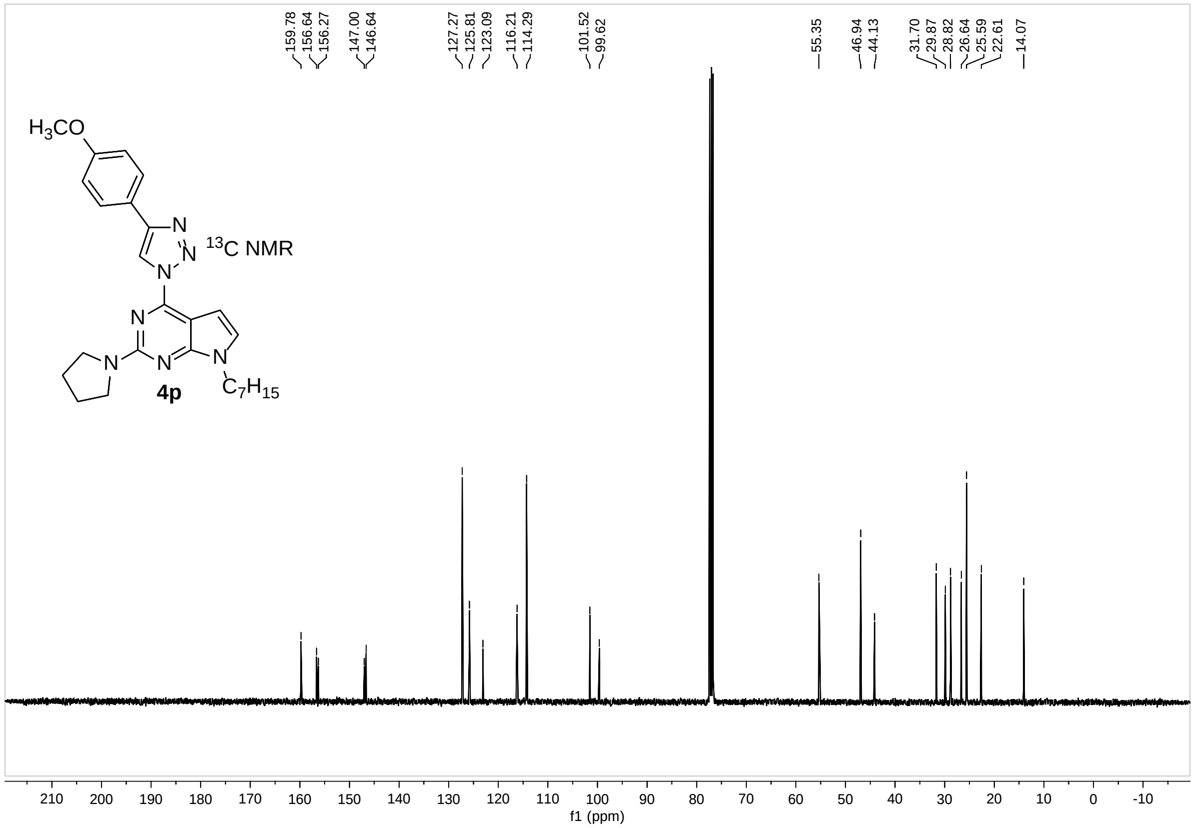
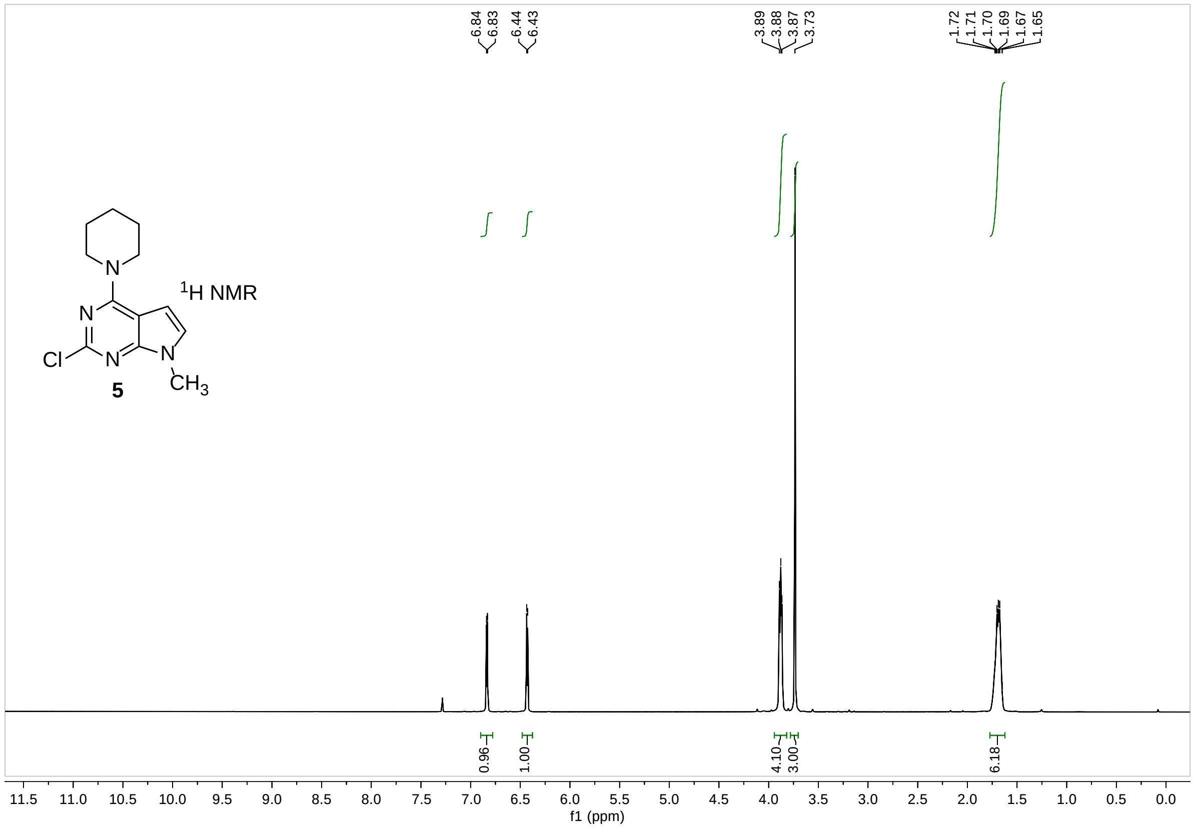
156.3
147.1
146.9
130.3
128.9
128.4
126.4
126.0
117.1
101.6
100.0
45.6
44.1
31.7
29.9
28.8
26.7
25.7
25.0
22.6
14.1
N
N
N
^{13}C NMR
N
N
N
N
4n
C_7H_{15}
180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0
f1 (ppm)

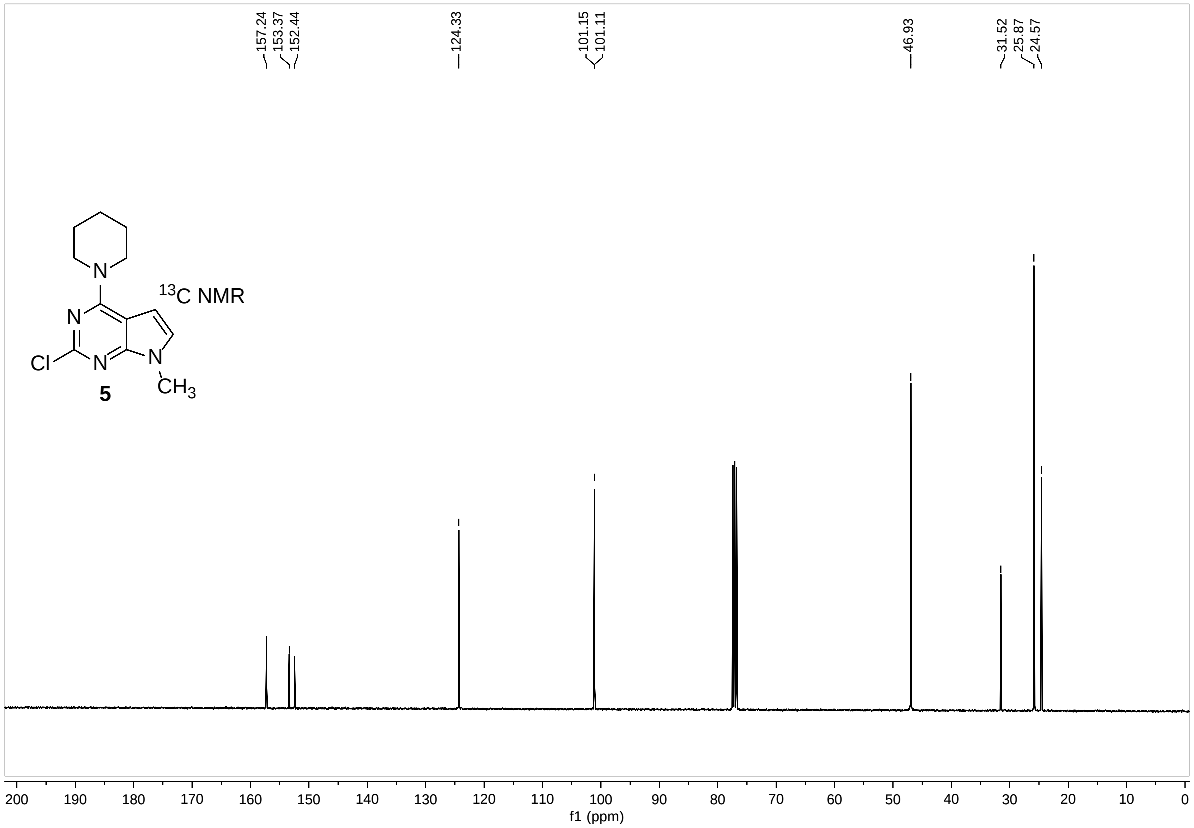

8.897
8.018
8.000
7.518
7.500
7.480
7.417
7.398
7.380
7.097
7.088
6.937
6.928
4.176
4.158
4.141
3.729
3.713
3.696
2.079
2.063
2.046
1.895
1.877
1.859
1.842
1.824
1.435
1.360
1.302
1.290
1.279
1.264
0.913
0.896
0.878
N
N
N
^{1}H NMR
N
N
N
N
4o
C_7H_{15}
0.97
2.00
2.02
1.10
1.00
1.03
2.02
4.09
4.10
2.15
8.11
3.22
12.5 12.0 11.5 11.0 10.5 10.0 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 0.5 0.0 -0.5
f1 (ppm)

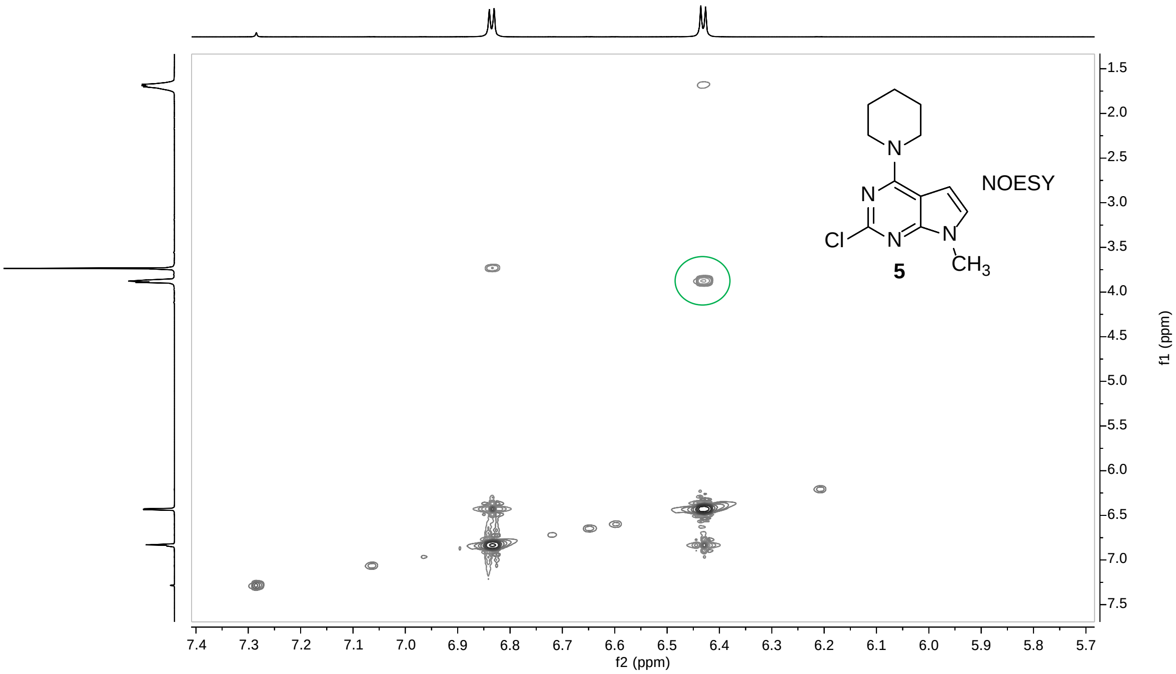

156.62
156.27
146.96
146.80
130.38
128.88
128.35
125.97
125.92
117.11
101.51
99.66
46.96
44.17
31.70
29.88
28.82
26.64
25.59
22.61
14.07
^{13}C NMR
N
N
N
N
N
N
N
C_7H_{15}
4o
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
f1 (ppm)

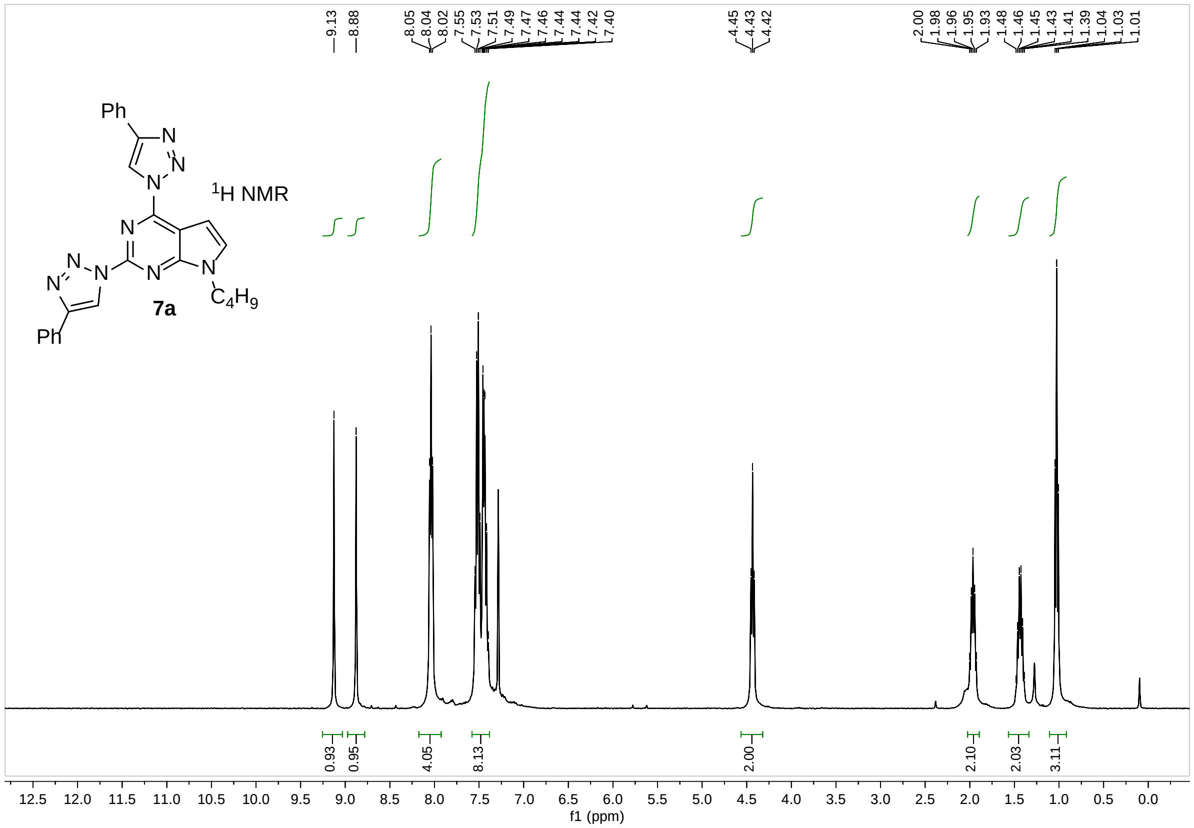

8.800
7.938
7.916
7.087
7.078
7.034
7.012
6.925
6.916
4.164
4.146
4.128
3.885
3.718
3.702
3.685
2.072
2.062
2.055
2.048
2.038
1.888
1.871
1.853
1.836
1.356
1.353
1.344
1.330
1.318
1.299
1.293
1.286
1.276
0.910
0.893
0.876
H_3CO
^{1}H NMR
N
N
N
N
N
N
N
C_7H_{15}
4p
1.00
2.07
1.01
2.06
0.98
2.12
3.17
4.21
4.28
2.23
8.40
3.20
12.0
11.5
11.0
10.5
10.0
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
0.5
0.0
-0.5
f1 (ppm)

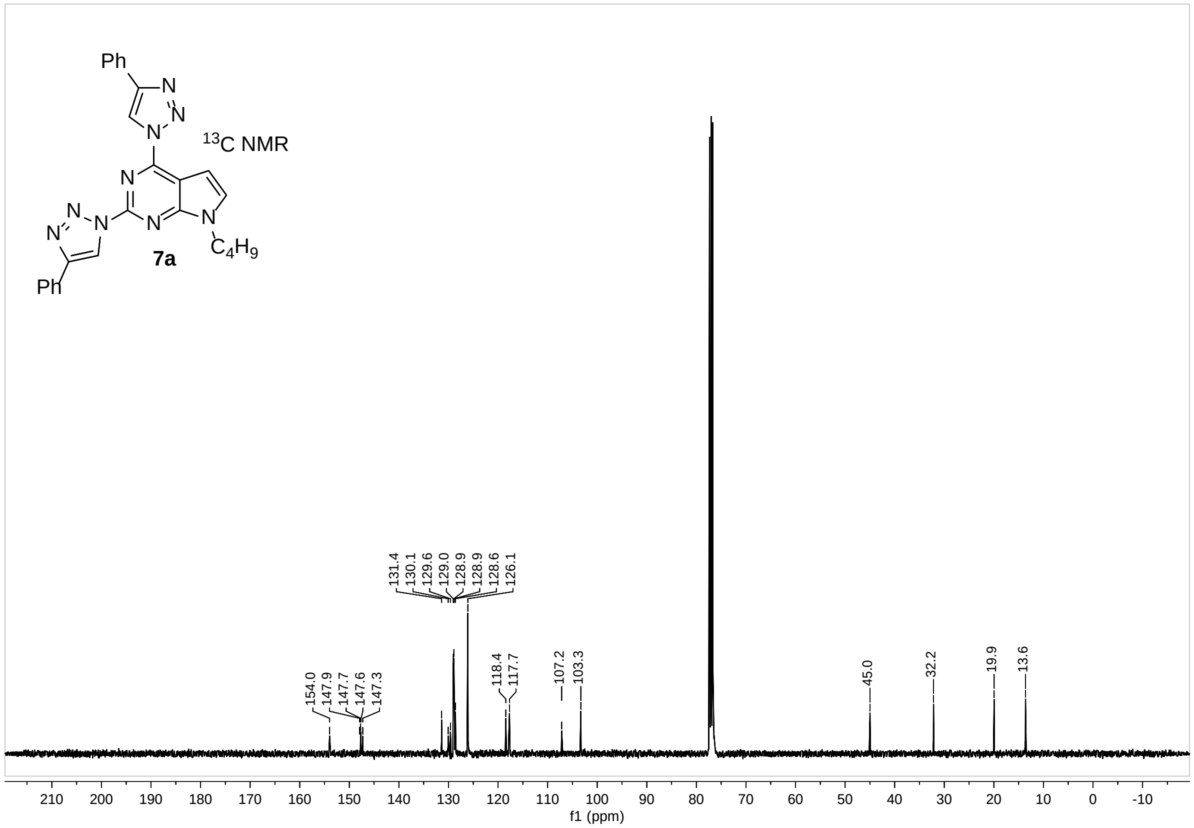

159.78
156.64
156.27
147.00
146.64
127.27
125.81
123.09
116.21
114.29
101.52
99.62
55.35
46.94
44.13
31.70
29.87
28.82
26.64
25.59
22.61
14.07
H3CO
N
N
N
N
N
N
N
^{13}C NMR
4p
C7H15
210 200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0 -10
f1 (ppm)

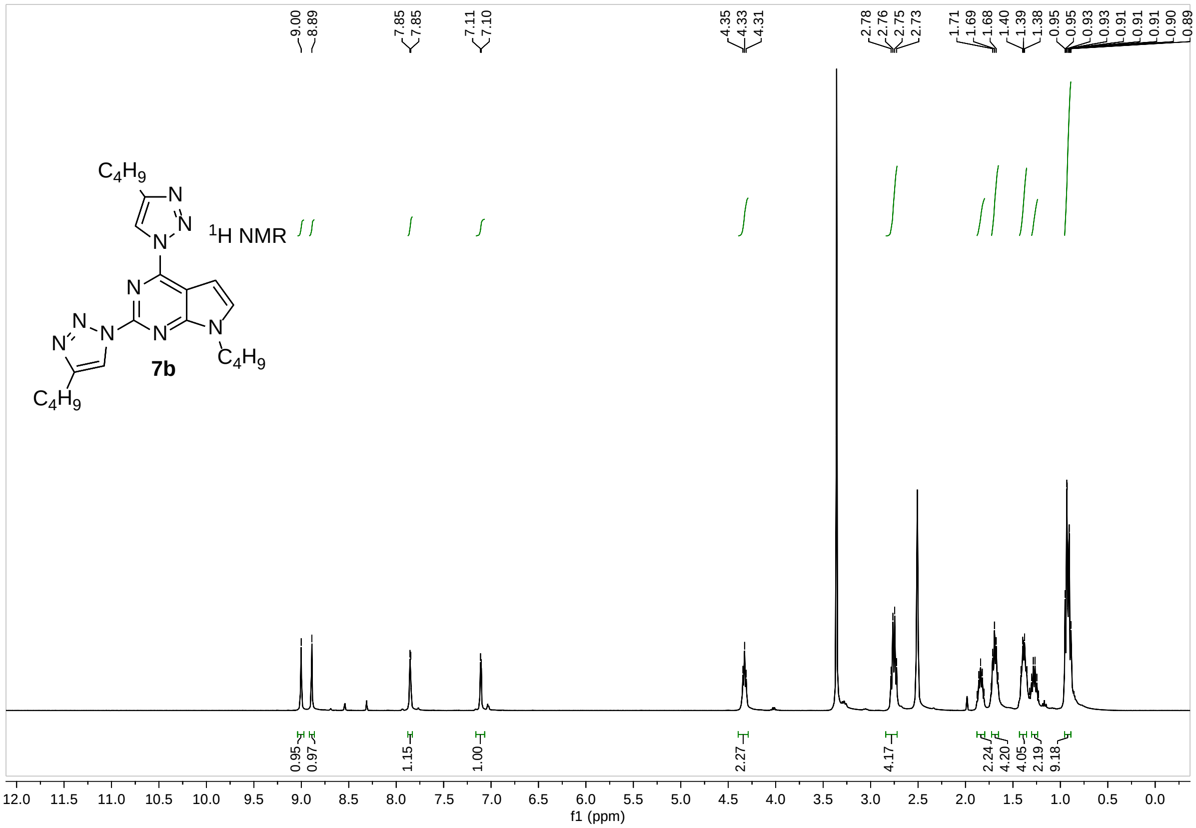

6.84
6.83
6.44
6.43
3.89
3.88
3.87
3.73
1.72
1.71
1.70
1.69
1.67
1.65
N
N
N
N
Cl
CH3
^{1}H NMR
5
0.96
1.00
4.10
3.00
6.18
11.5 11.0 10.5 10.0 9.5 9.0 8.5 8.0 7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 0.5 0.0
f1 (ppm)


157.24
153.37
152.44
124.33
101.15
101.11
46.93
31.52
25.87
24.57
^{13}C NMR
N
N
N
N
Cl
CH_3
5
200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0
f1 (ppm)

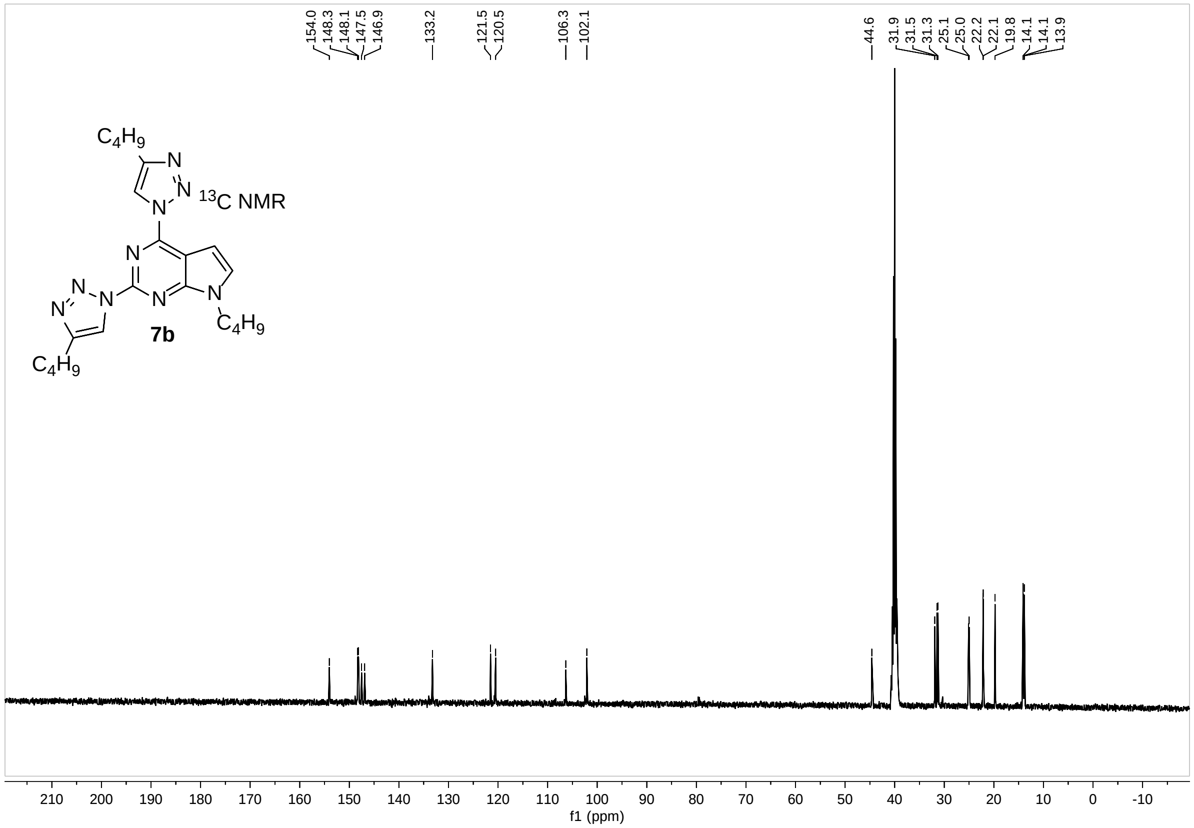

NOESY
N
N
N
N
Cl
CH_3
5
7.4 7.3 7.2 7.1 7.0 6.9 6.8 6.7 6.6 6.5 6.4 6.3 6.2 6.1 6.0 5.9 5.8 5.7
f2 (ppm)
1.5 2.0 2.5 3.0 3.5 4.0 4.5 5.0 5.5 6.0 6.5 7.0 7.5
f1 (ppm)


Ph
N
N
N
N
N
N
N
N
N
N
C4H9
Ph
7a
1H NMR
9.13
8.88
8.05
8.04
8.02
7.55
7.53
7.51
7.49
7.47
7.46
7.44
7.44
7.42
7.40
4.45
4.43
4.42
2.00
1.98
1.96
1.95
1.93
1.48
1.46
1.45
1.43
1.41
1.39
1.04
1.03
1.01
0.93
0.95
4.05
8.13
2.00
2.10
2.03
3.11
12.5 12.0 11.5 11.0 10.5 10.0 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 0.5 0.0
f1 (ppm)

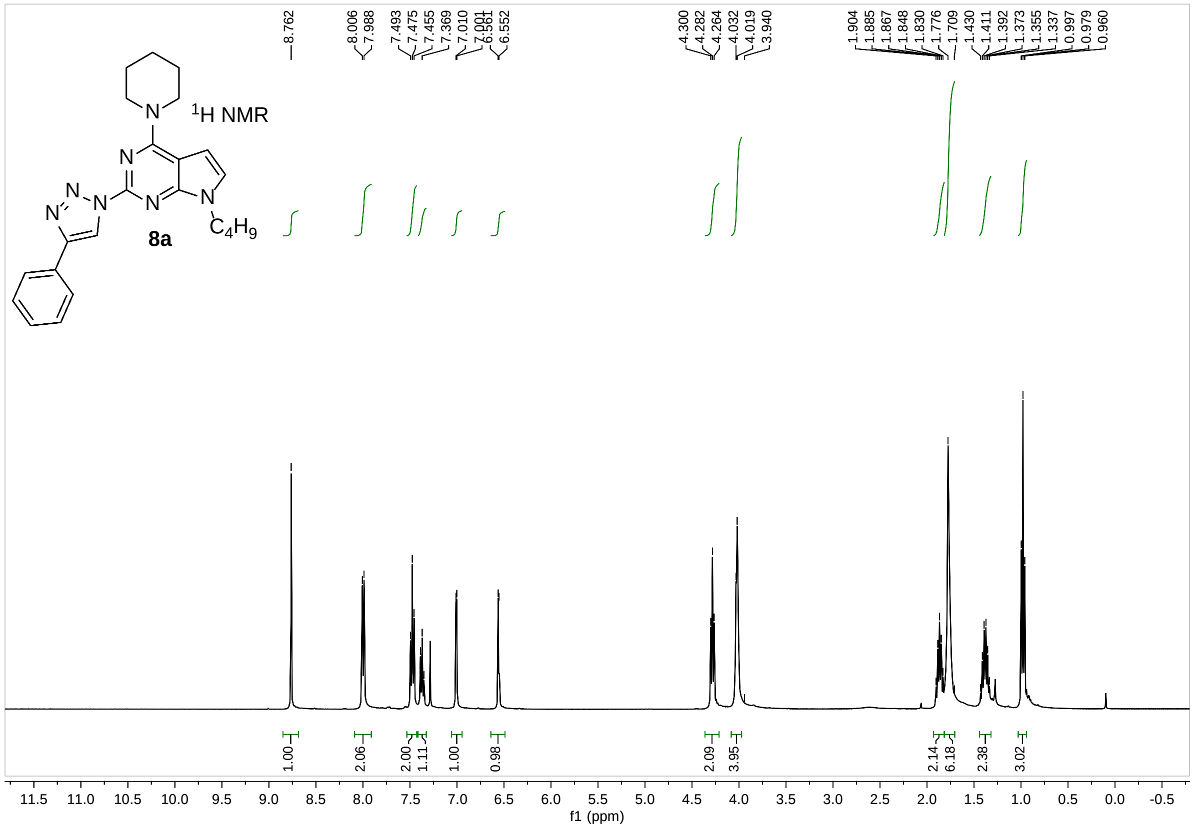

Ph
N
N
N
N
N
N
N
N
N
N
C4H9
Ph
7a
13C NMR
154.0
147.9
147.7
147.6
147.3
131.4
130.1
129.6
129.0
128.9
128.9
128.6
126.1
118.4
117.7
107.2
103.3
45.0
32.2
19.9
13.6
210 200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0 -10
f1 (ppm)

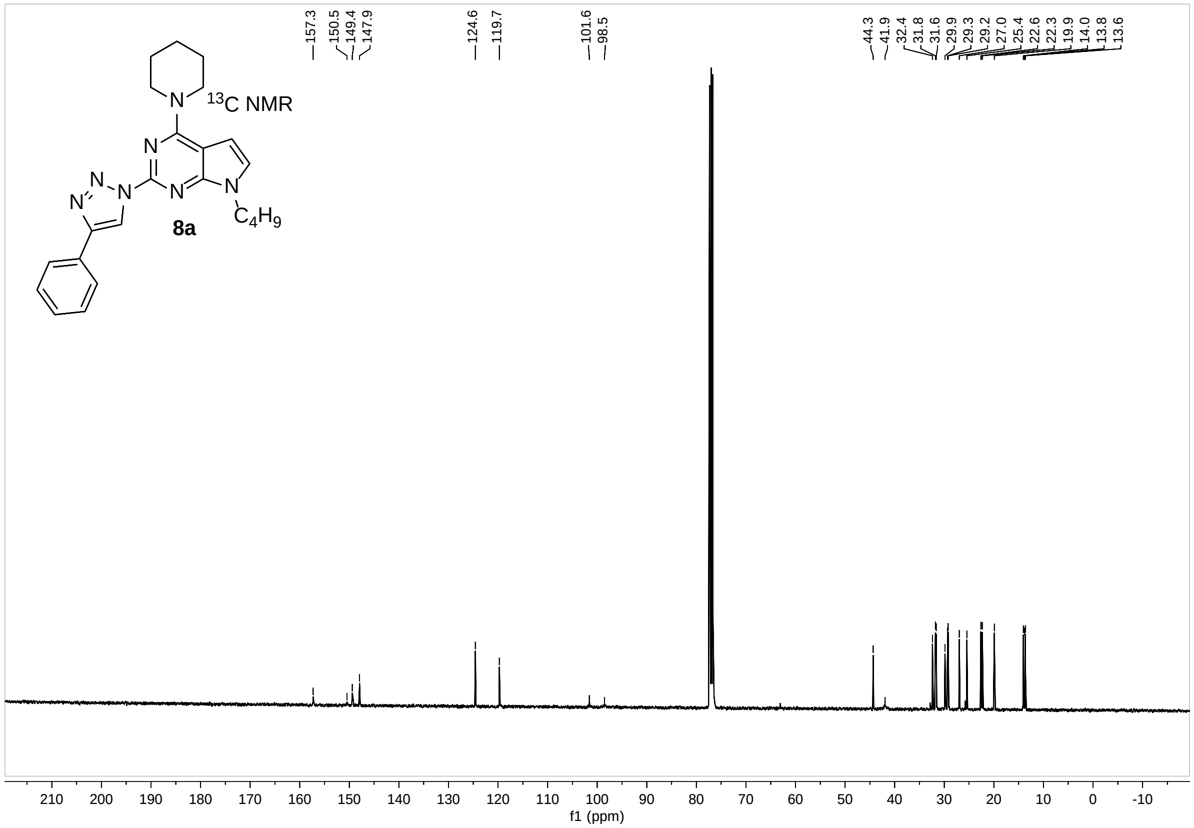


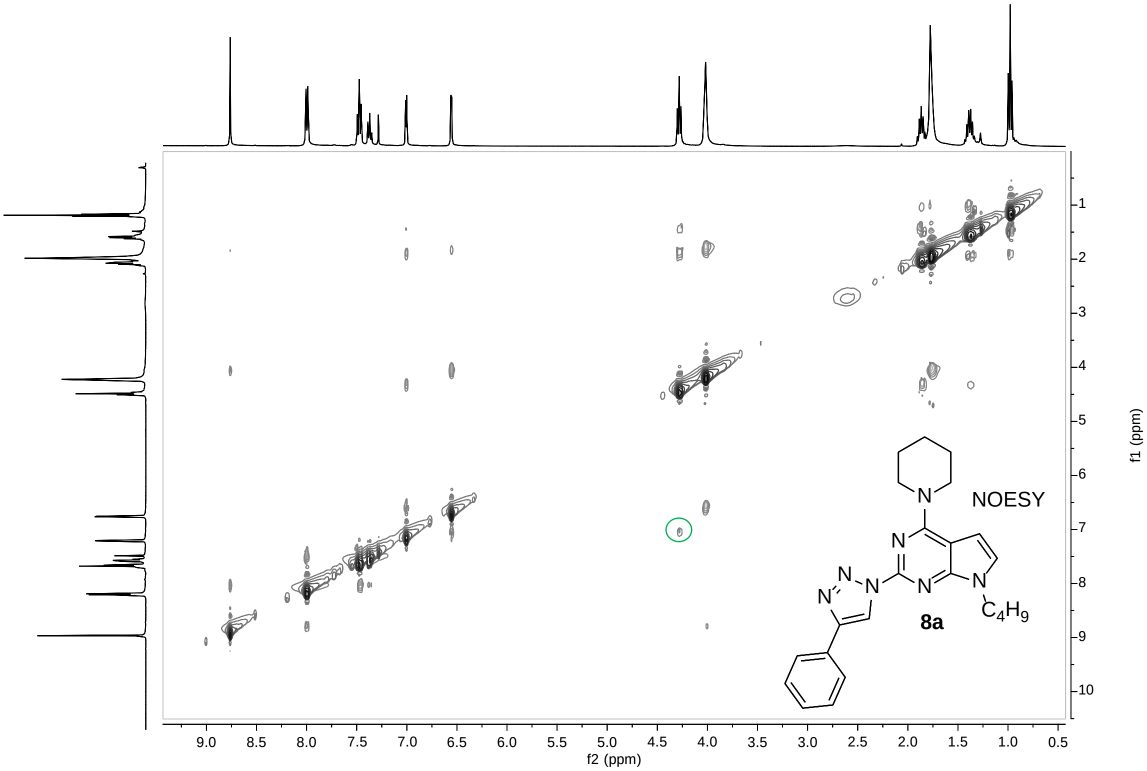
9.00
8.89
7.85
7.85
7.11
7.10
4.35
4.33
4.31
2.78
2.76
2.75
2.73
1.71
1.69
1.68
1.40
1.39
1.38
0.95
0.95
0.93
0.93
0.91
0.91
0.91
0.90
0.89
C_4H_9
^{1}H NMR
C_4H_9
7b
C_4H_9
0.95
0.97
1.15
1.00
2.27
4.17
2.24
4.20
4.05
2.19
9.18
12.0
11.5
11.0
10.5
10.0
9.5
9.0
8.5
8.0
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
0.5
0.0
f1 (ppm)

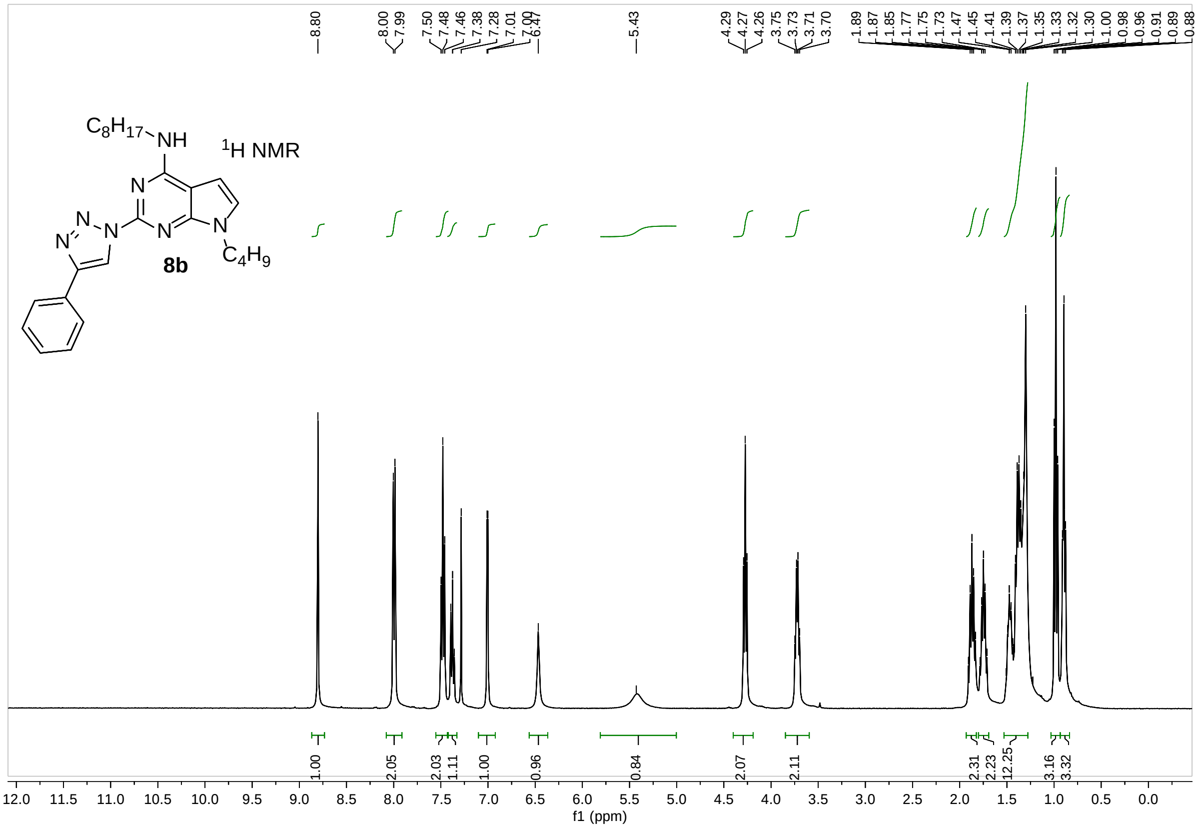


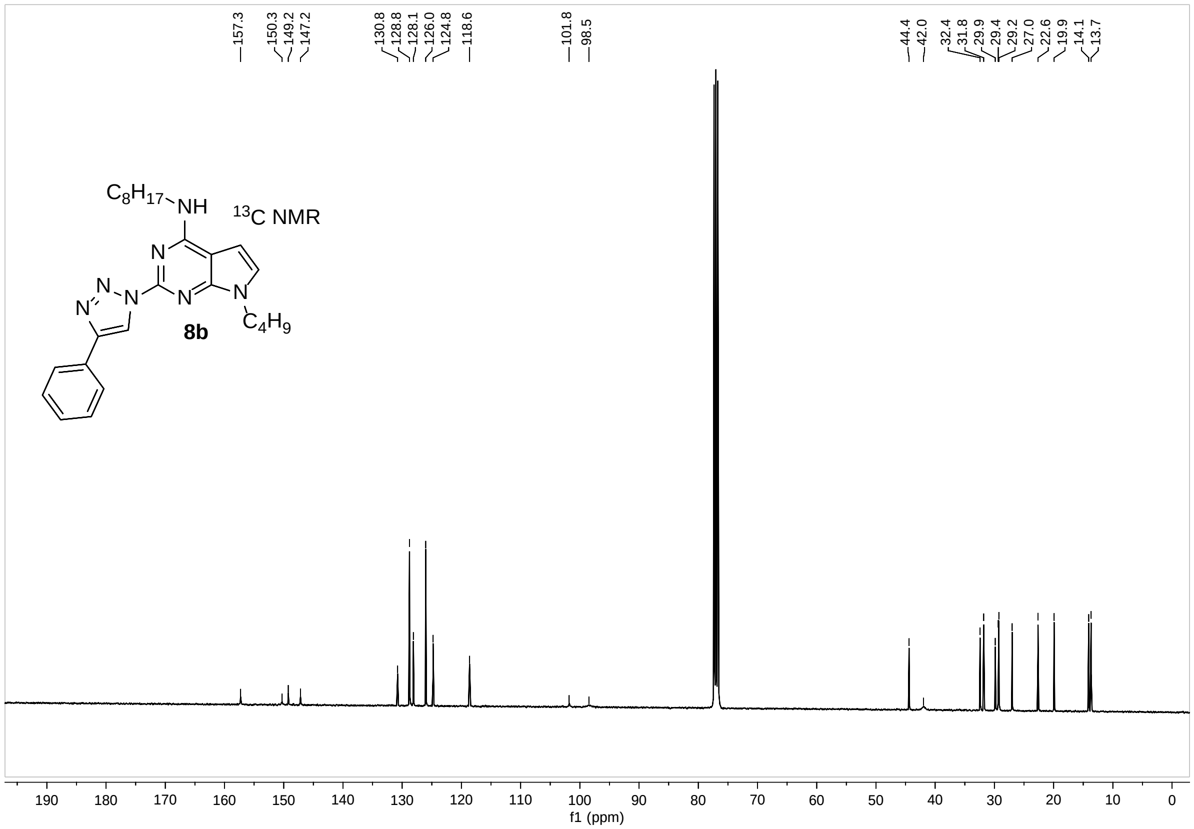
154.0
148.3
148.1
147.5
146.9
133.2
121.5
120.5
106.3
102.1
44.6
31.9
31.5
31.3
25.1
25.0
22.2
22.1
19.8
14.1
14.1
13.9
C_4H_9
^{13}C NMR
C_4H_9
7b
C_4H_9
210
200
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
-10
f1 (ppm)

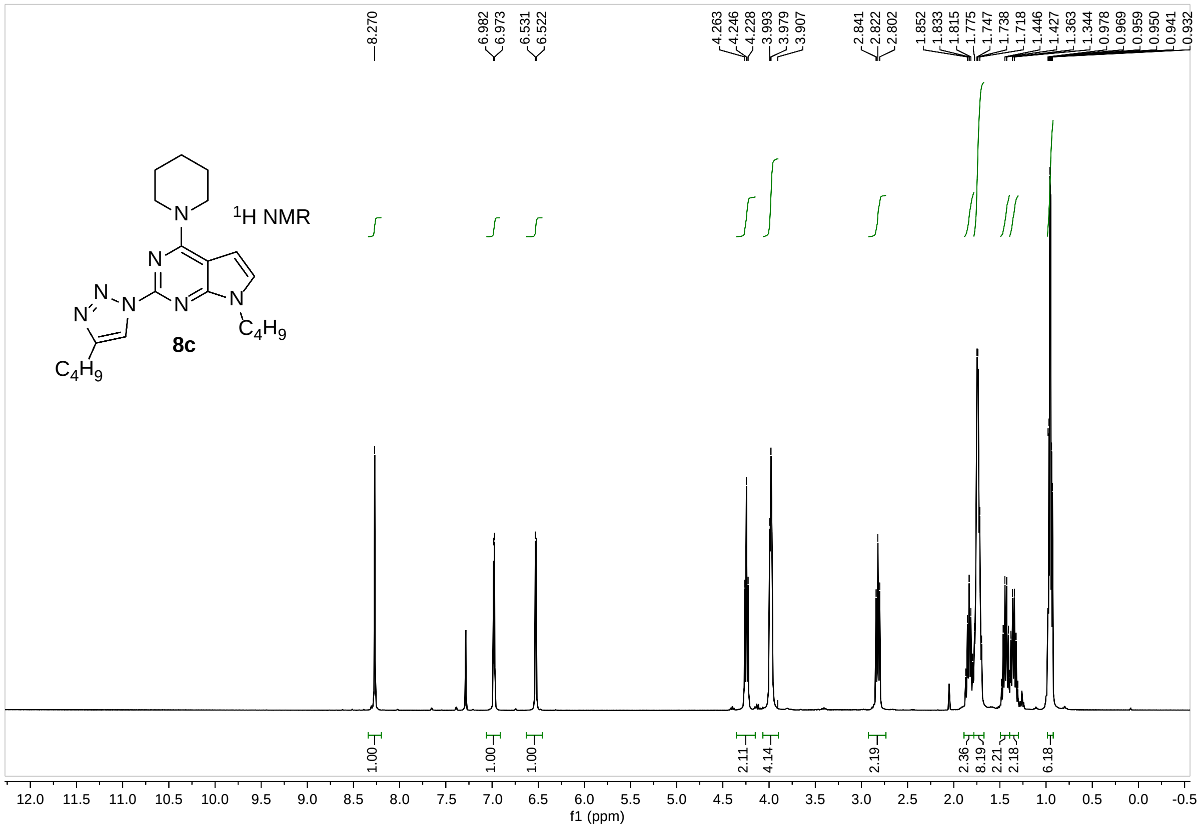

1H NMR
N
N
N
N
N
N
N
C4H9
8a
8.762
8.006
7.988
7.493
7.475
7.455
7.369
7.010
7.001
6.561
6.552
4.300
4.282
4.264
4.032
4.019
3.940
1.904
1.885
1.867
1.848
1.830
1.776
1.709
1.430
1.411
1.392
1.373
1.355
1.337
0.997
0.979
0.960
1.00
2.06
2.00
1.11
1.00
0.98
2.09
3.95
2.14
6.18
2.38
3.02
11.5
11.0
10.5
10.0
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
0.5
0.0
-0.5
f1 (ppm)

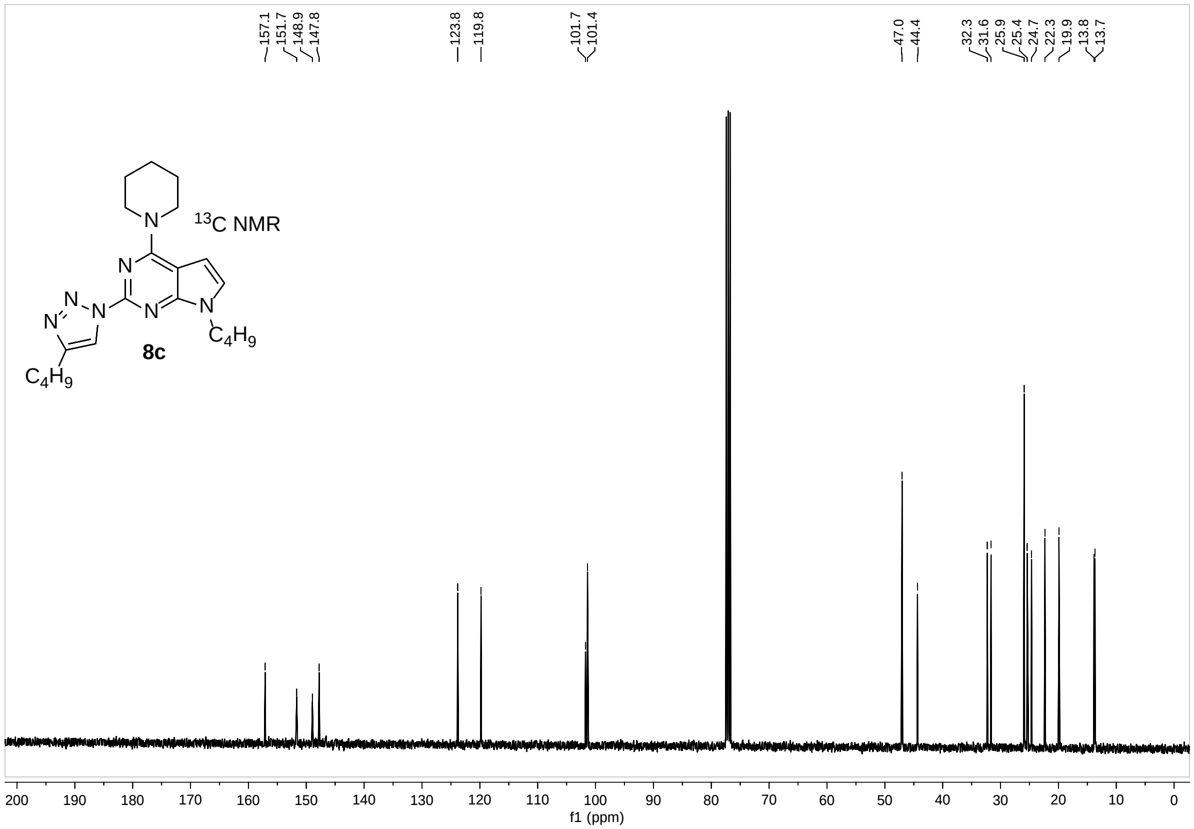

13C NMR
N
N
N
N
N
N
N
C4H9
8a
157.3
150.5
149.4
147.9
124.6
119.7
101.6
98.5
44.3
41.9
32.4
31.8
31.6
29.9
29.3
29.2
27.0
25.4
22.6
22.3
19.9
14.0
13.8
13.6
210
200
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
-10
f1 (ppm)


NOESY
8a
N
N
N
N
N
N
C4H9
f1 (ppm)
f2 (ppm)
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 0.5
1 2 3 4 5 6 7 8 9 10

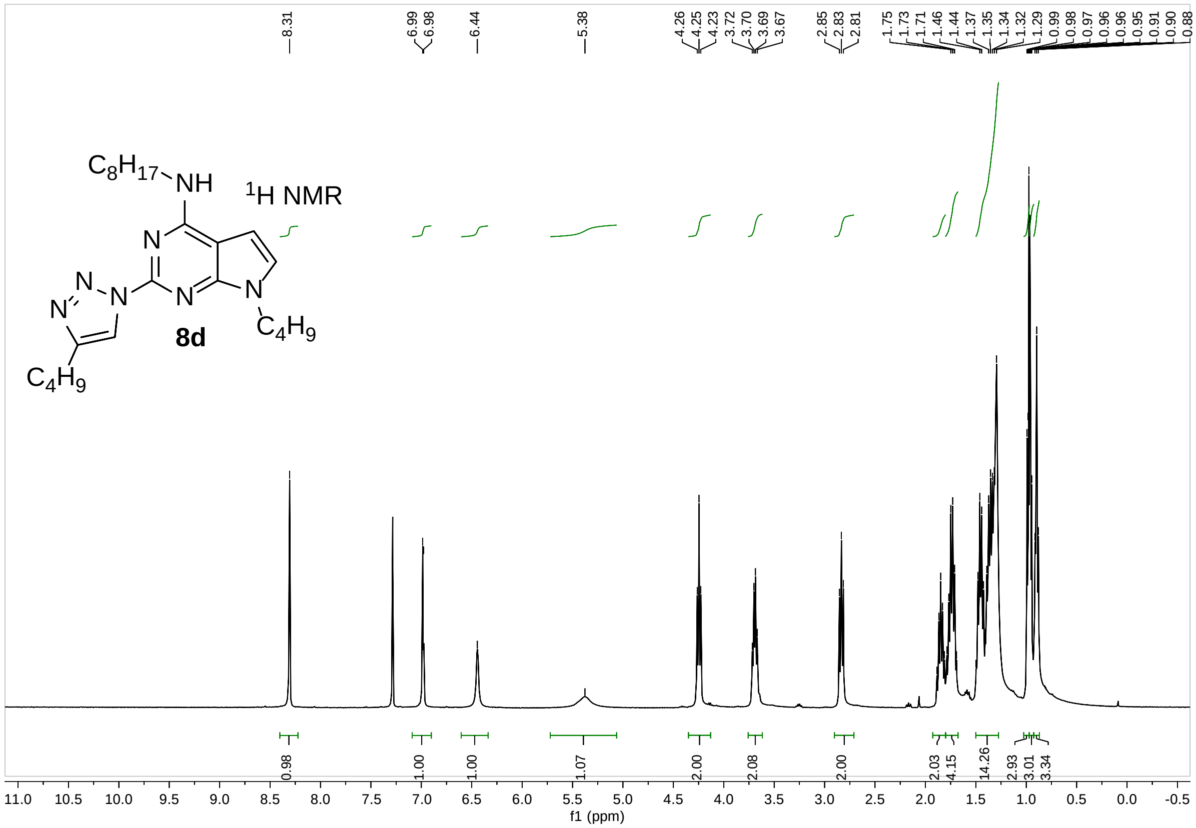

1H NMR
8b
C8H17-NH
N
N
N
N
N
N
C4H9
8.80
8.00
7.99
7.50
7.48
7.46
7.38
7.28
7.01
7.00
6.47
5.43
4.29
4.27
4.26
3.75
3.73
3.71
3.70
1.89
1.87
1.85
1.77
1.75
1.73
1.47
1.45
1.41
1.39
1.37
1.35
1.33
1.32
1.30
1.00
0.98
0.96
0.91
0.89
0.88
1.00
2.05
2.03
1.11
1.00
0.96
0.84
2.07
2.11
2.31
2.23
12.25
3.16
3.32
12.0 11.5 11.0 10.5 10.0 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 0.5 0.0
f1 (ppm)


157.3
150.3
149.2
147.2
130.8
128.8
128.1
126.0
124.8
118.6
101.8
98.5
44.4
42.0
32.4
31.8
29.9
29.4
29.2
27.0
22.6
19.9
14.1
13.7
C_8H_{17}
NH
N
N
N
N
N
N
N
C_4H_9
8b
^{13}C NMR
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
f1 (ppm)

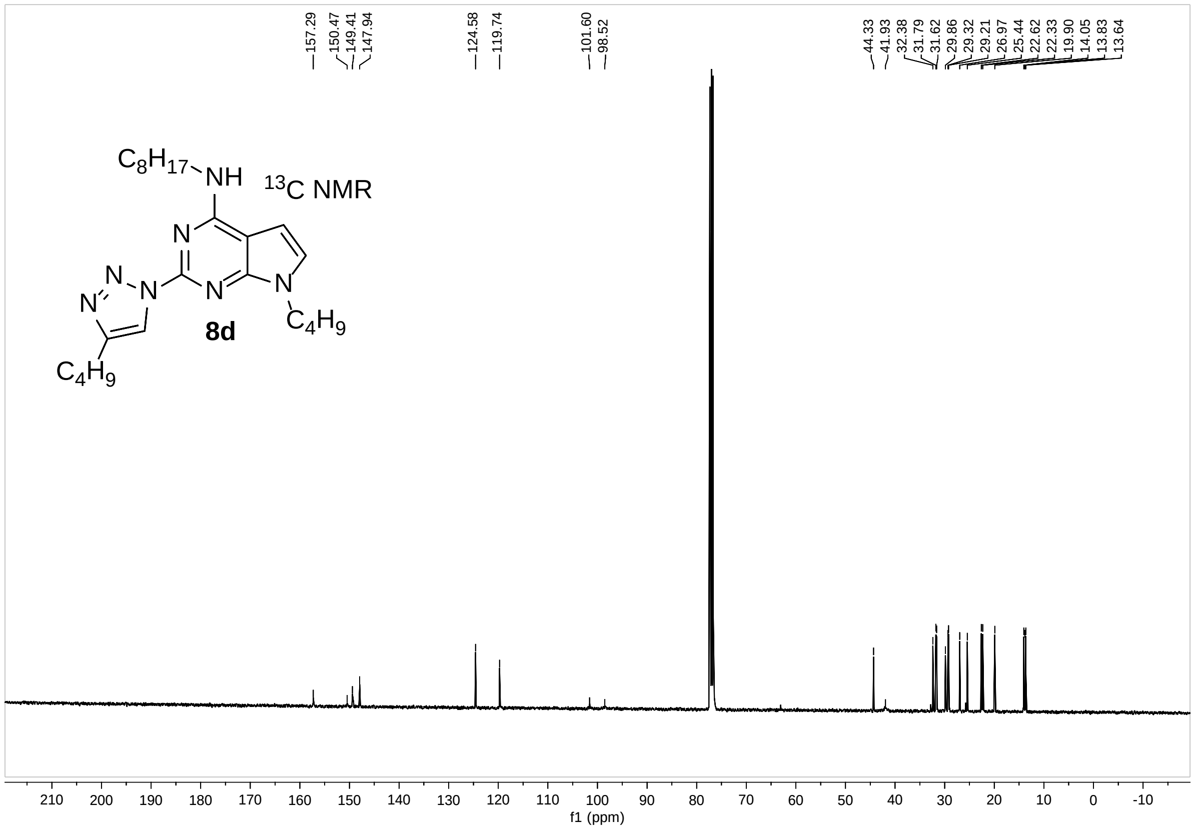

8.270
6.982
6.973
6.531
6.522
4.263
4.246
4.228
3.993
3.979
3.907
2.841
2.822
2.802
1.852
1.833
1.815
1.775
1.747
1.738
1.718
1.446
1.427
1.363
1.344
0.978
0.969
0.959
0.950
0.941
0.932
N
N
N
N
N
N
N
N
C_4H_9
C_4H_9
8c
^{1}H NMR
1.00
1.00
1.00
2.11
4.14
2.19
2.36
8.19
2.21
2.18
6.18
12.0
11.5
11.0
10.5
10.0
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
0.5
0.0
-0.5
f1 (ppm)



157.1
151.7
148.9
147.8
123.8
119.8
101.7
101.4
47.0
44.4
32.3
31.6
25.9
25.4
24.7
22.3
19.9
13.8
13.7
N
N
N
N
N
N
N
C4H9
C4H9
8c
13C NMR
200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0
f1 (ppm)



8.31
6.99
6.98
6.44
5.38
4.26
4.25
4.23
3.72
3.70
3.69
3.67
2.85
2.83
2.81
1.75
1.73
1.71
1.46
1.44
1.37
1.35
1.34
1.32
1.29
0.99
0.98
0.97
0.96
0.96
0.95
0.91
0.90
0.88
C8H17
NH
1H NMR
N
N
N
N
N
N
C4H9
C4H9
8d
0.98
1.00
1.00
1.07
2.00
2.08
2.00
2.03
4.15
14.26
2.93
3.01
3.34
11.0 10.5 10.0 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 0.5 0.0 -0.5
f1 (ppm)


C8H17
NH
13C NMR
N
N
N
N
N
N
8d
C4H9
C4H9
157.29
150.47
149.41
147.94
124.58
119.74
101.60
98.52
44.33
41.93
32.38
31.79
31.62
29.86
29.32
29.21
26.97
25.44
22.62
22.33
19.90
14.05
13.83
13.64
210
200
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
-10
f1 (ppm)