Supporting Information

Two-pot sequential multicomponent metal-free synthesis of pyrrolo[2,3-*d*]pyridazin-7-ones and pyrrolo[2,3-*d*]pyrizidines

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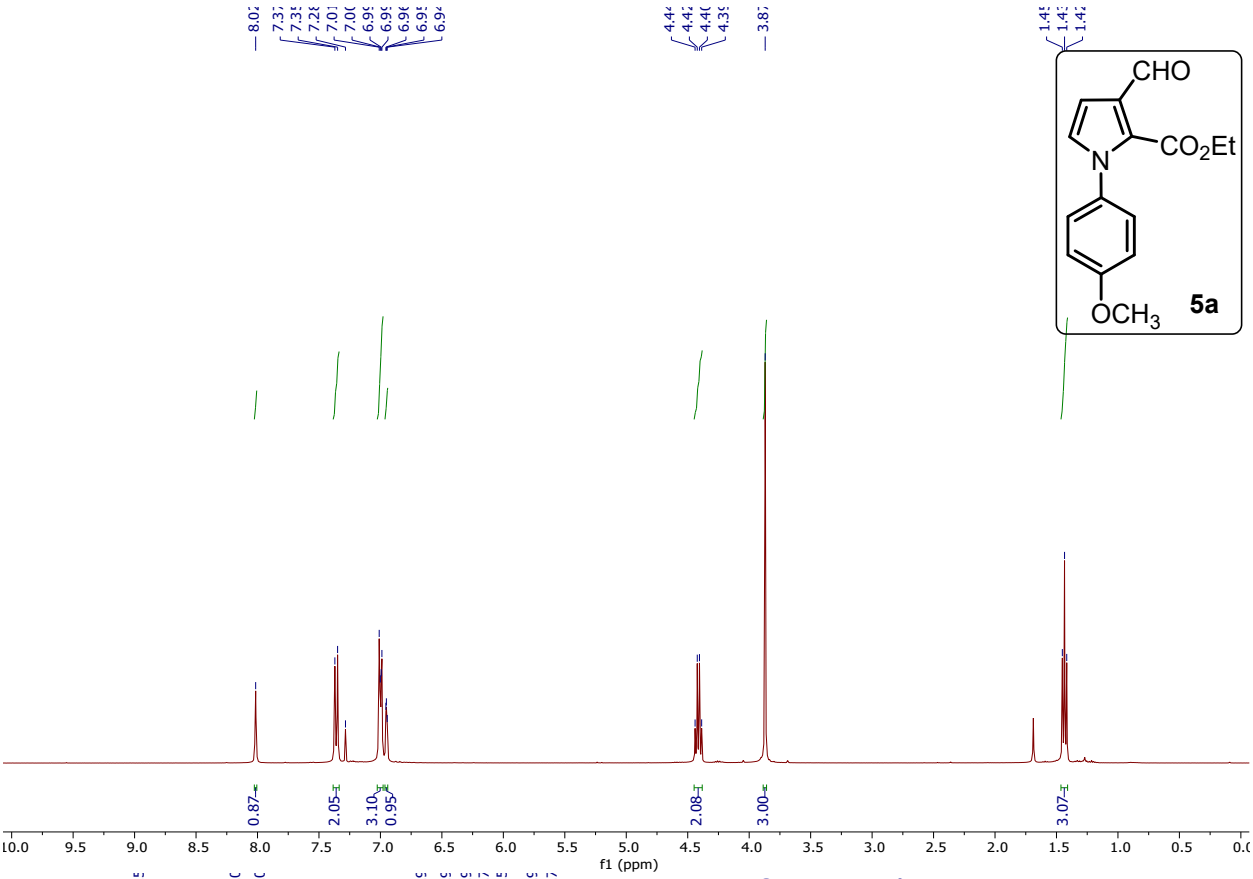
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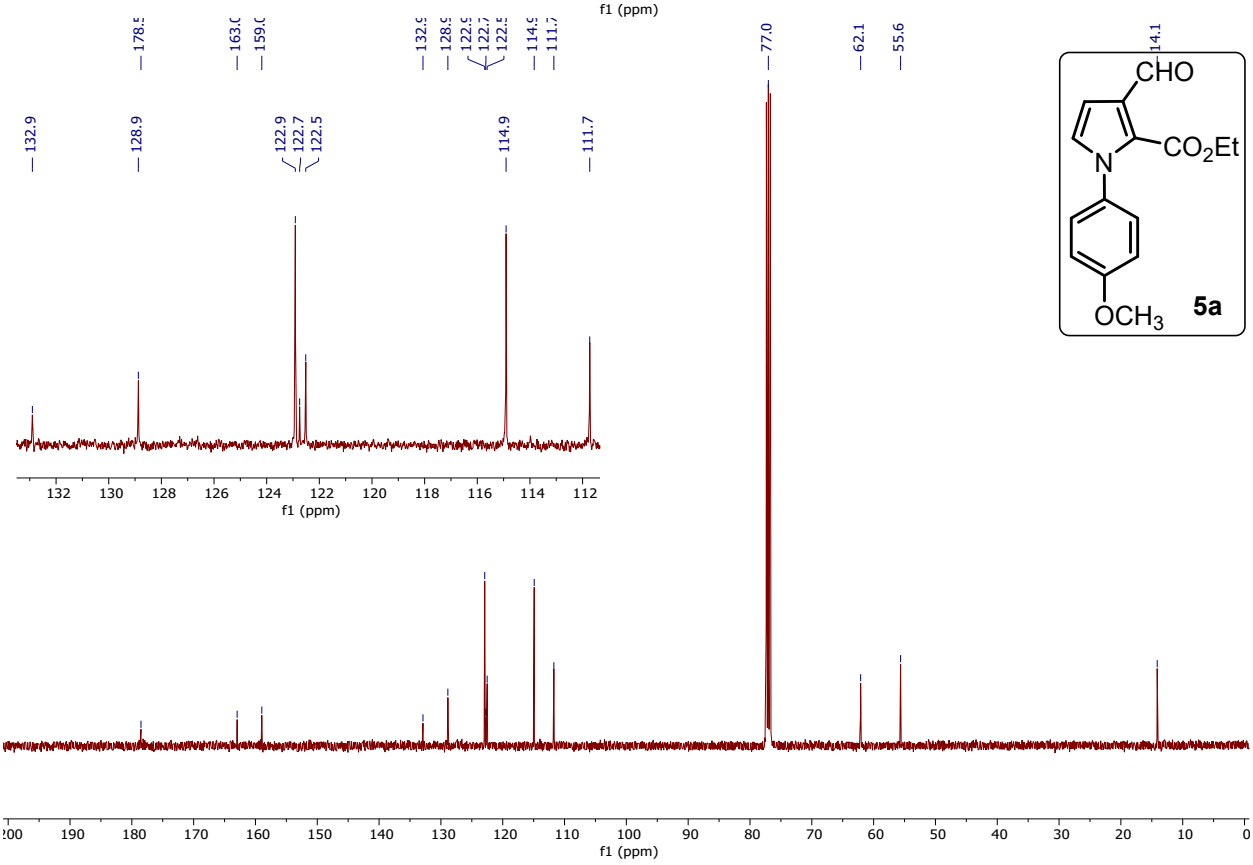
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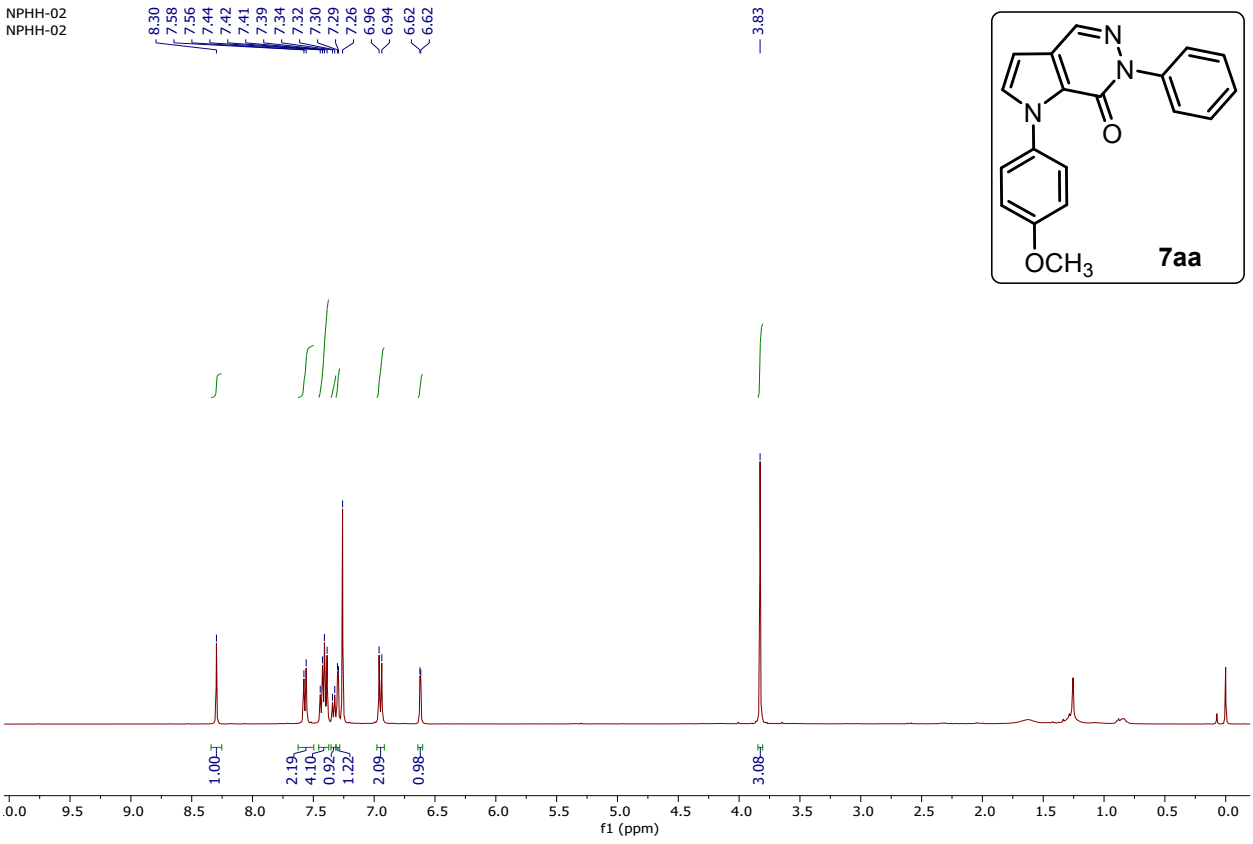
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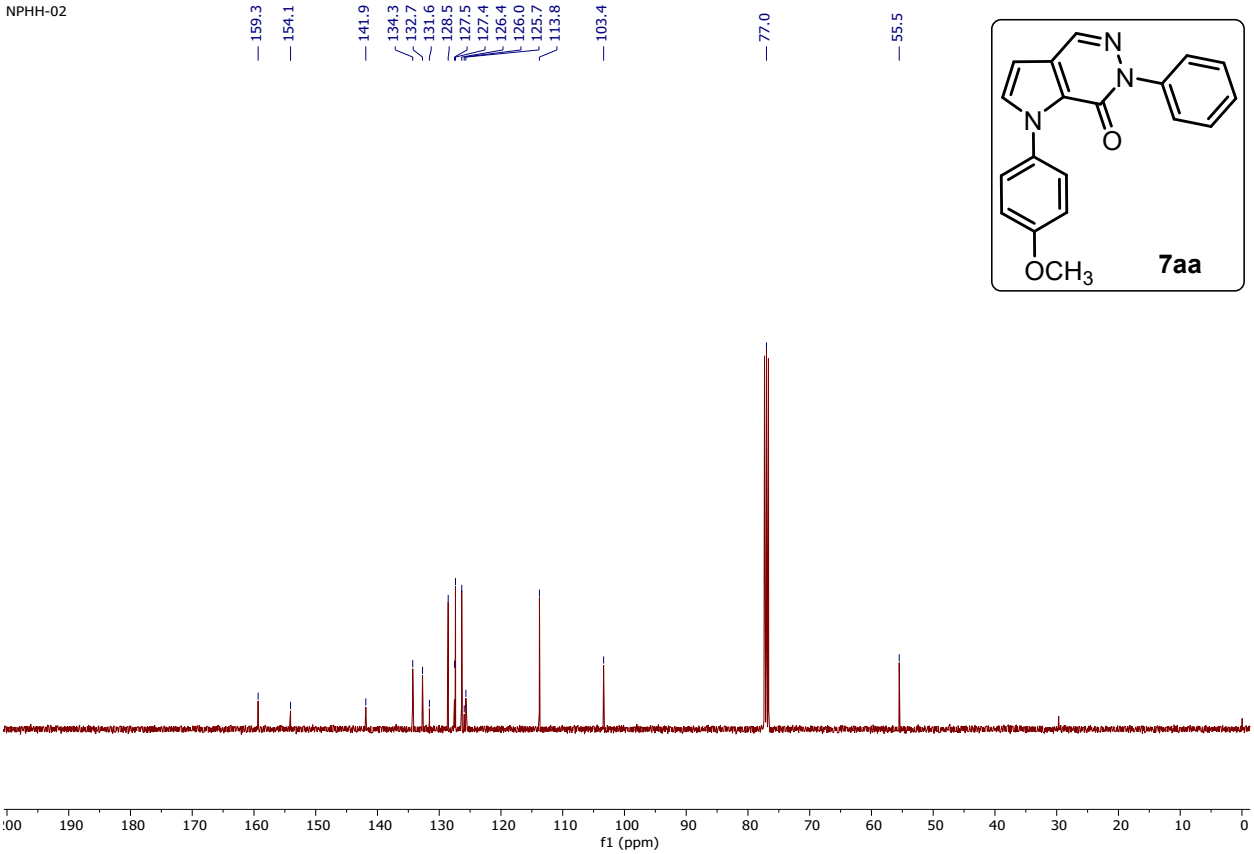
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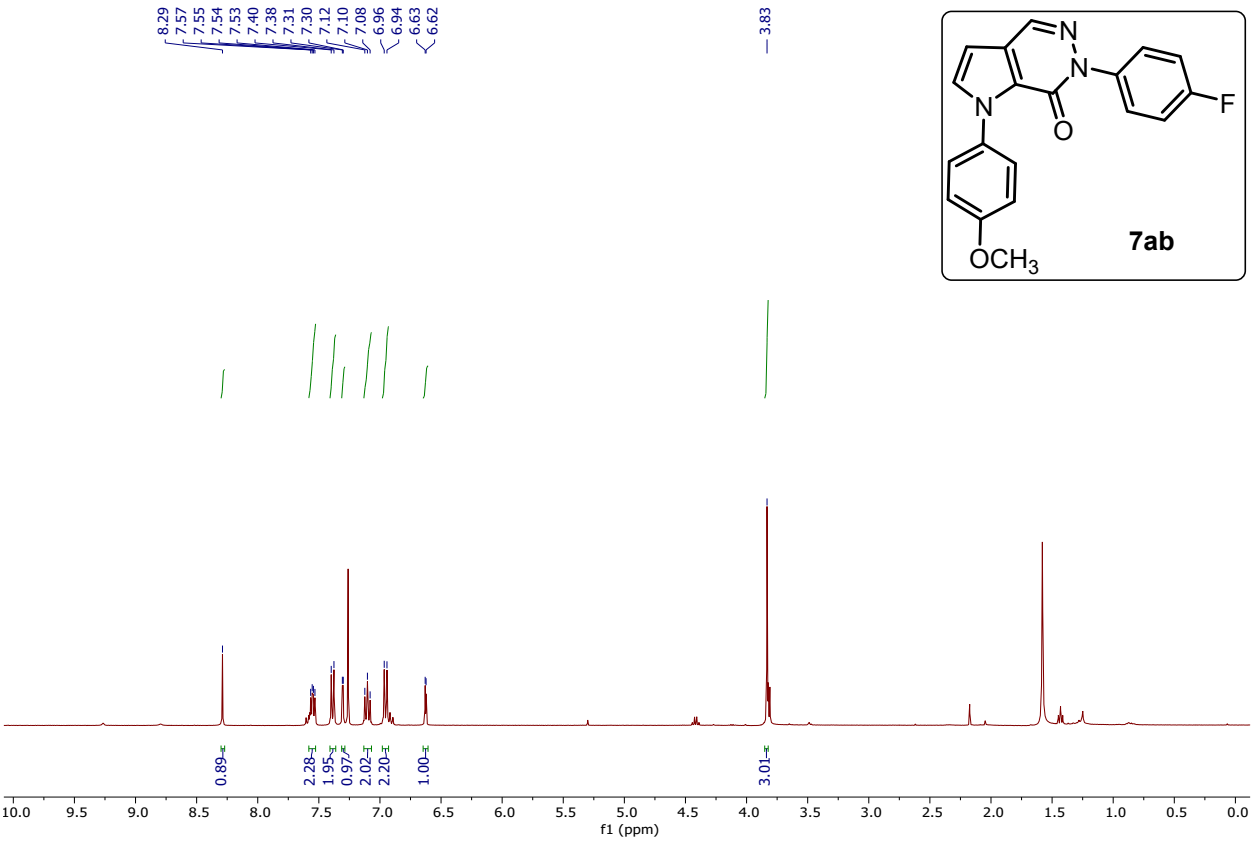
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F
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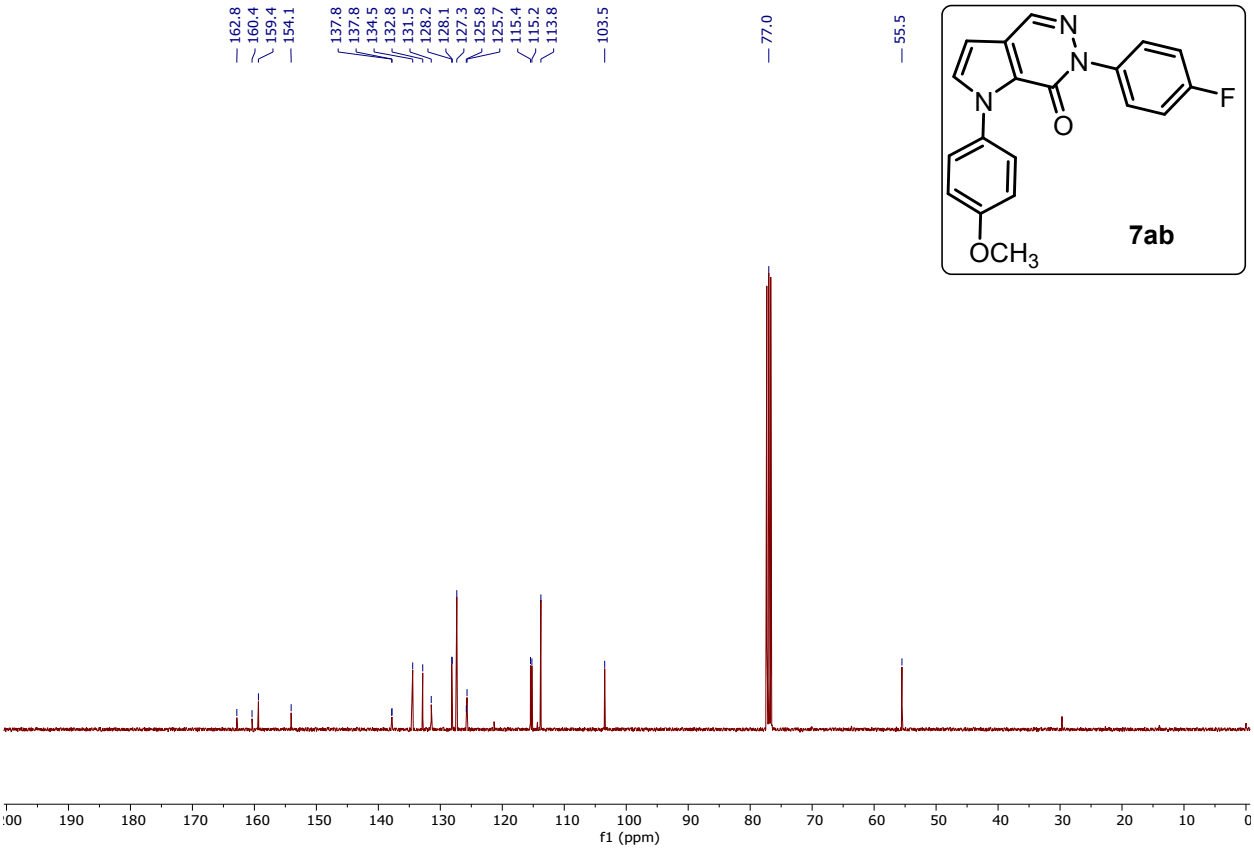
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O
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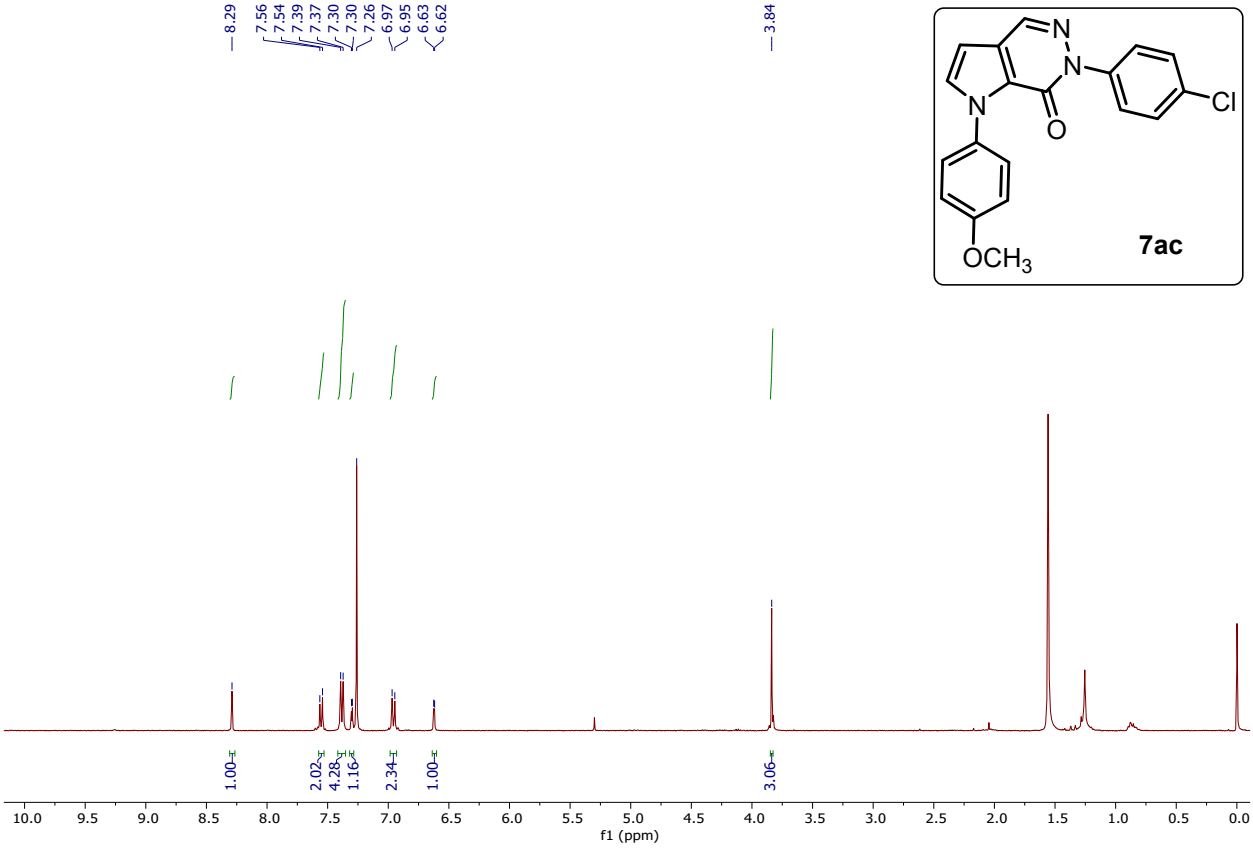
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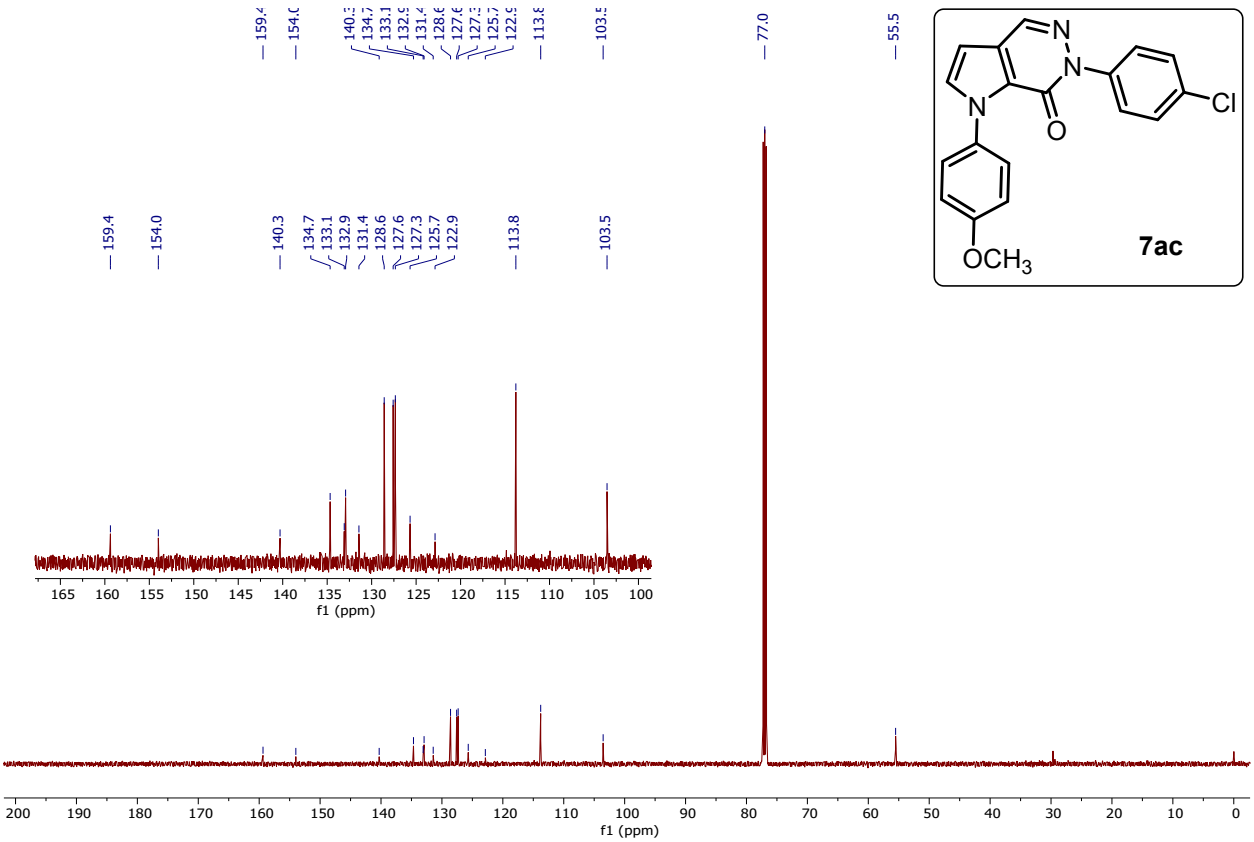
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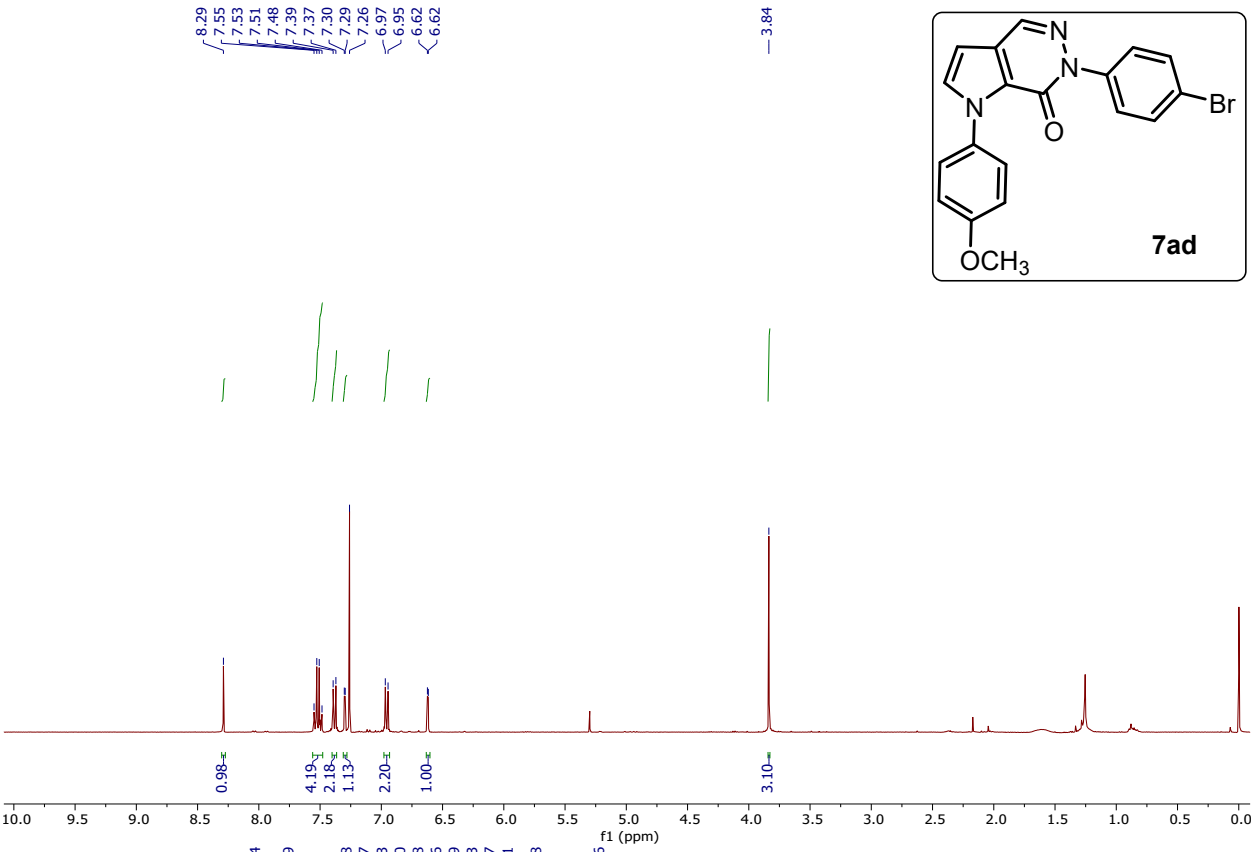
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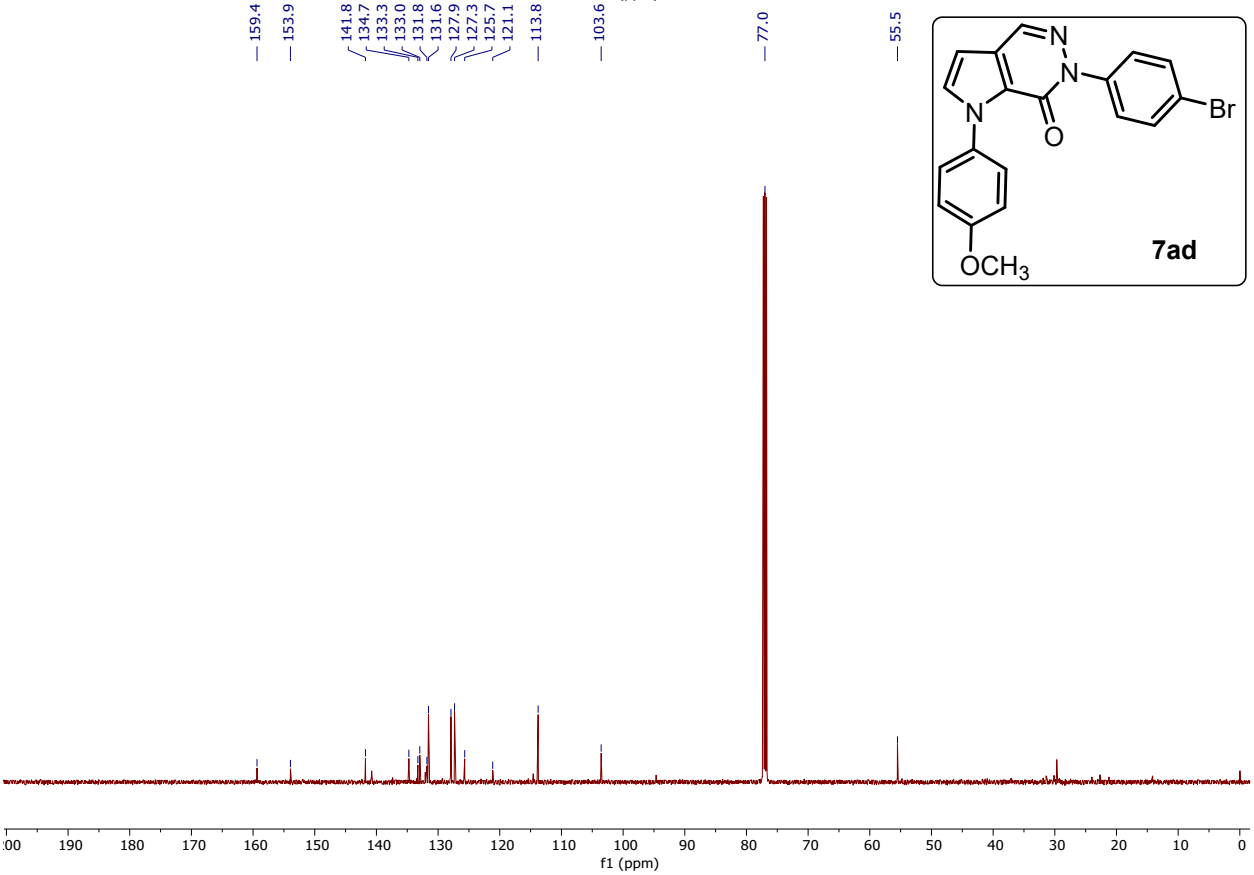
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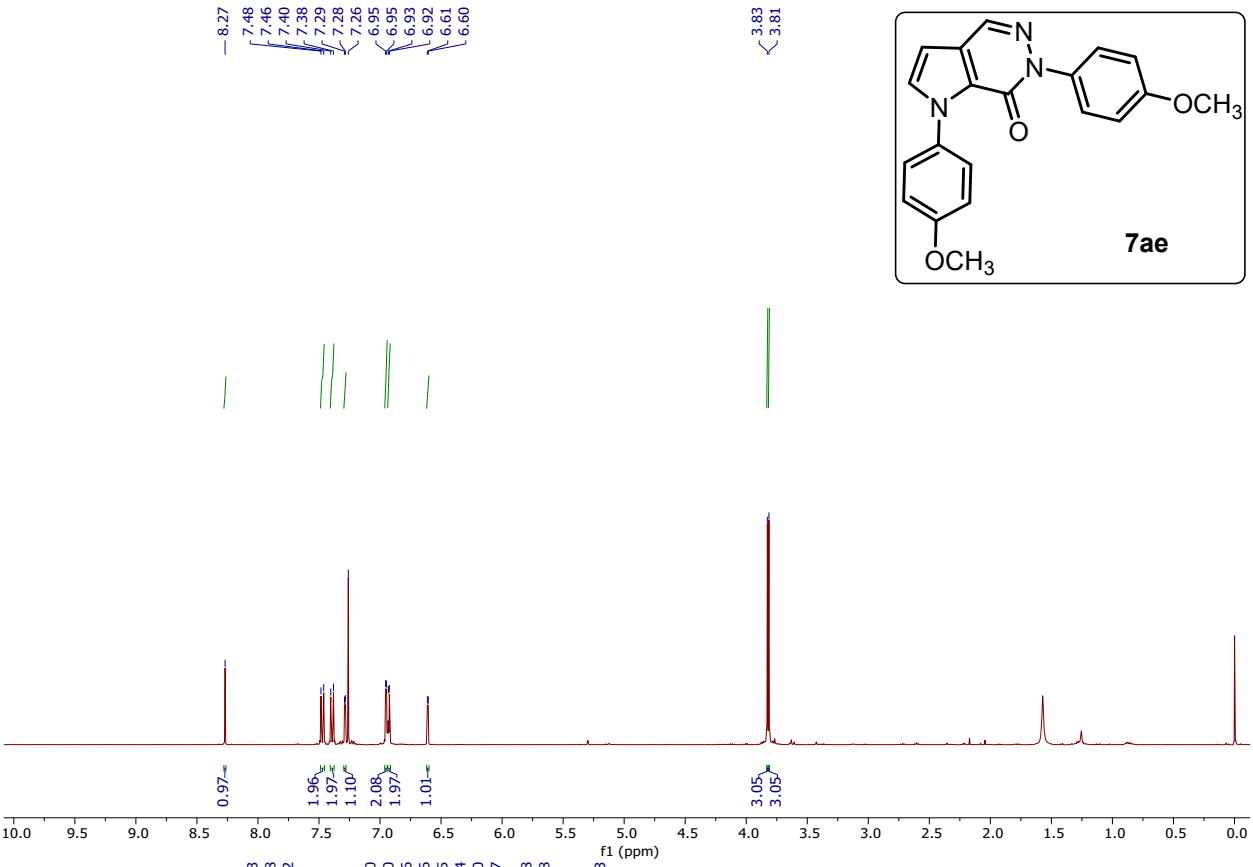
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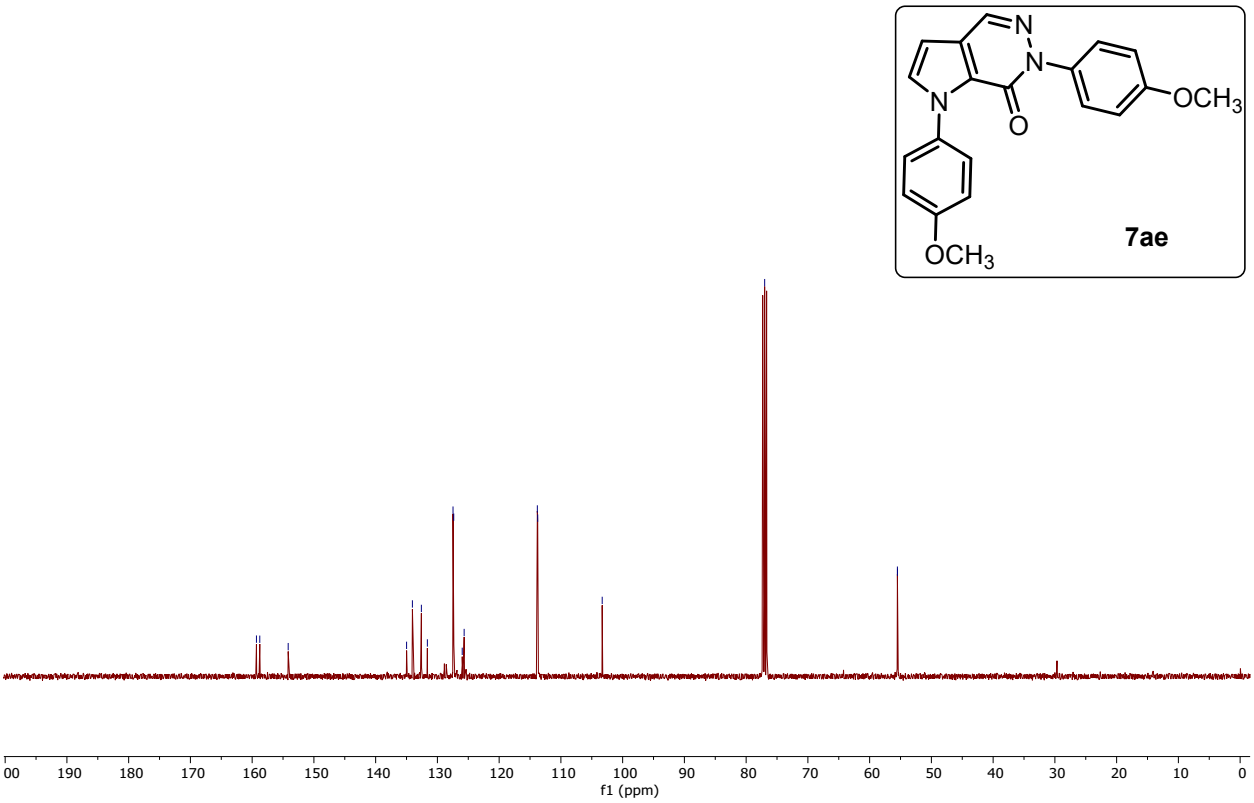
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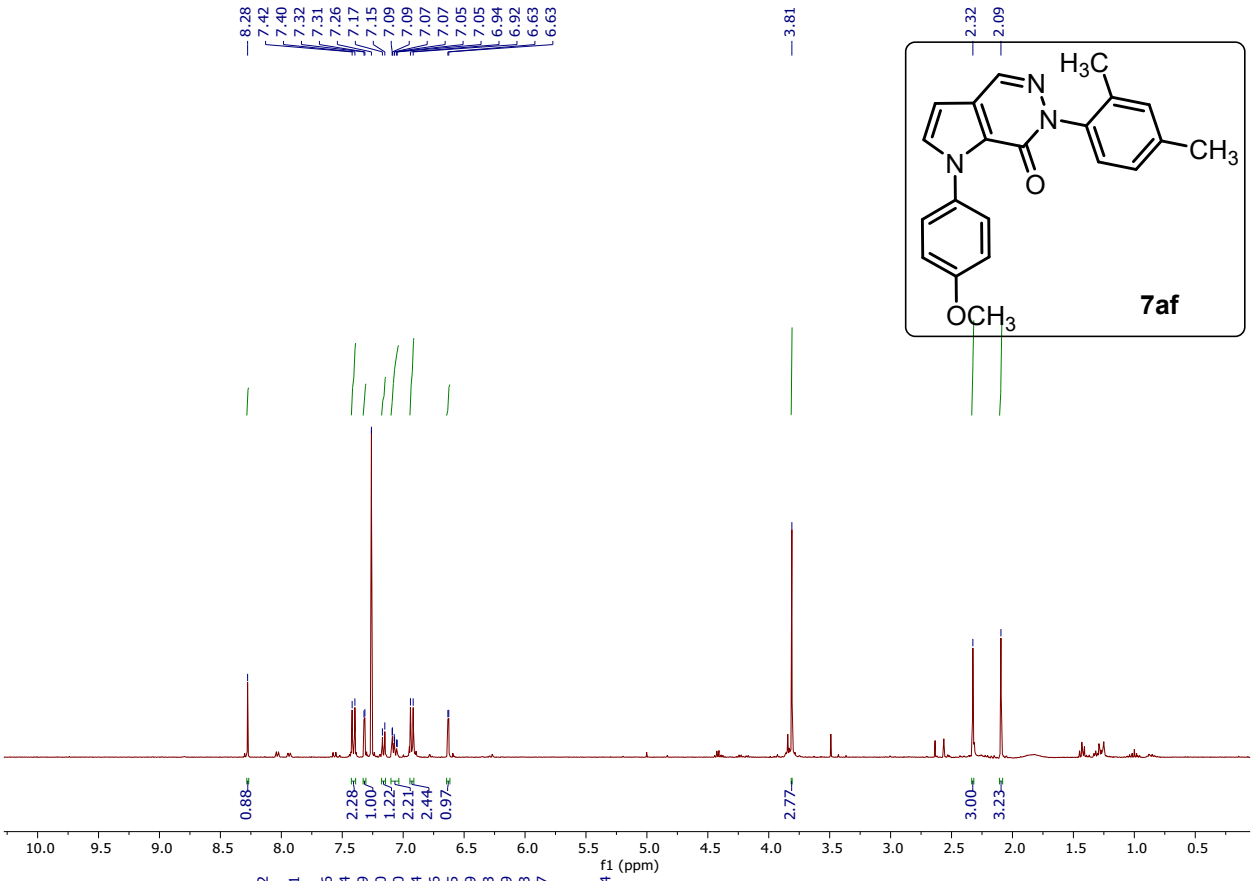
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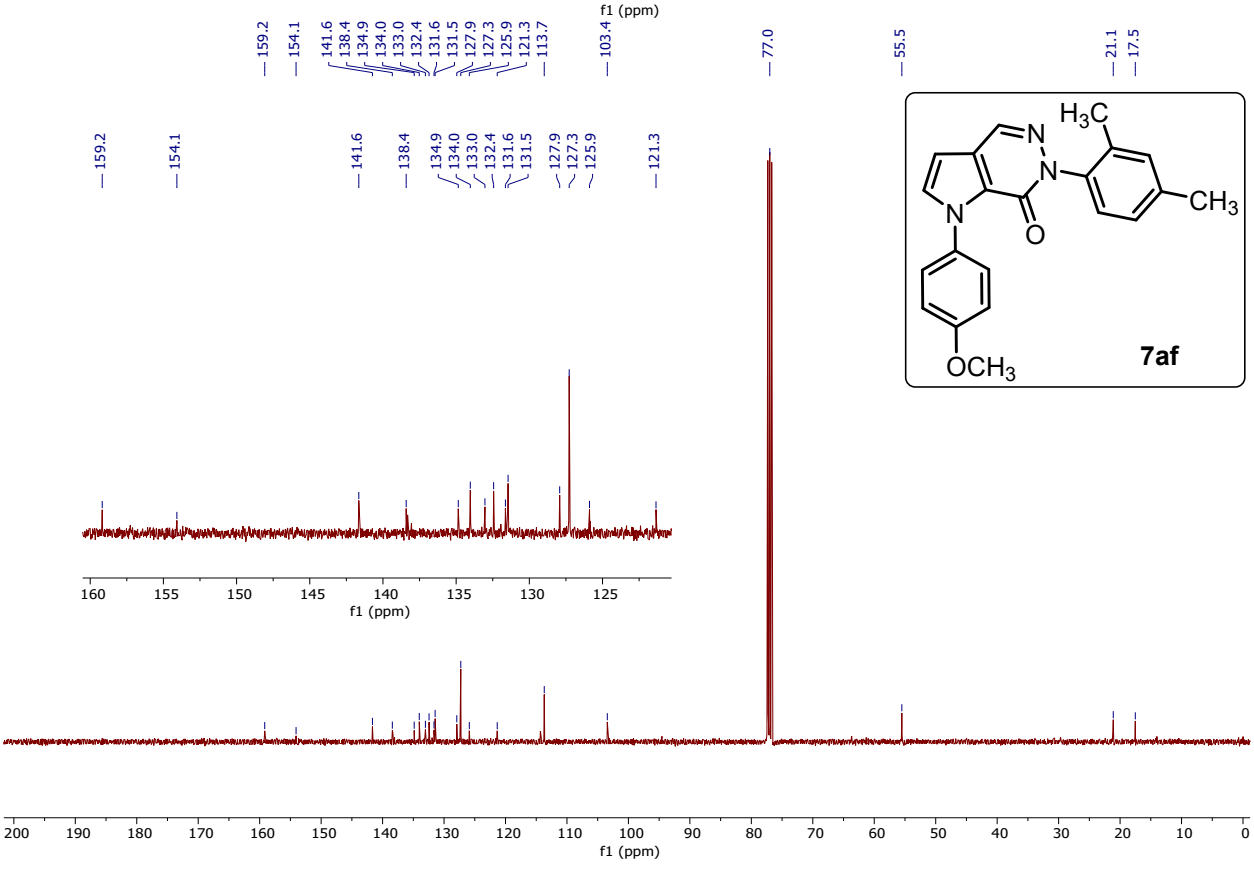
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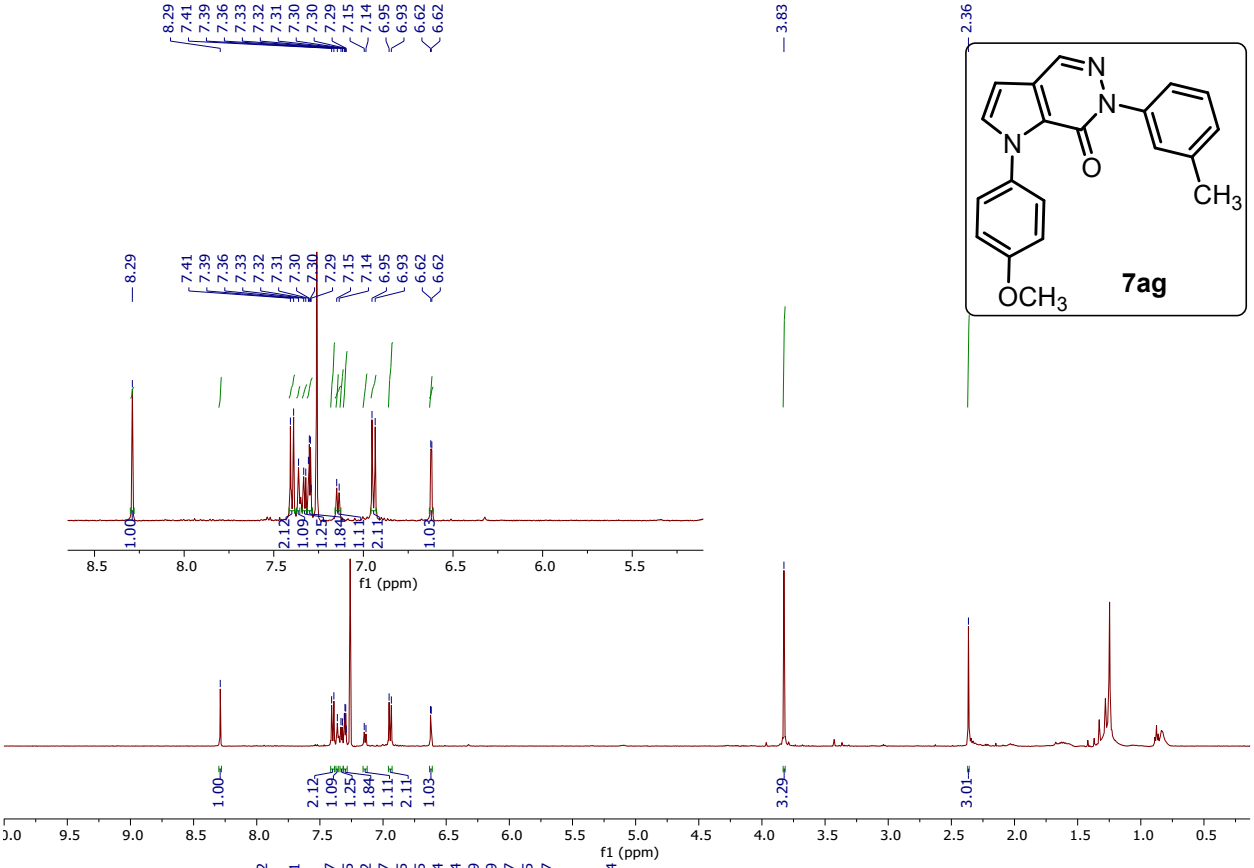
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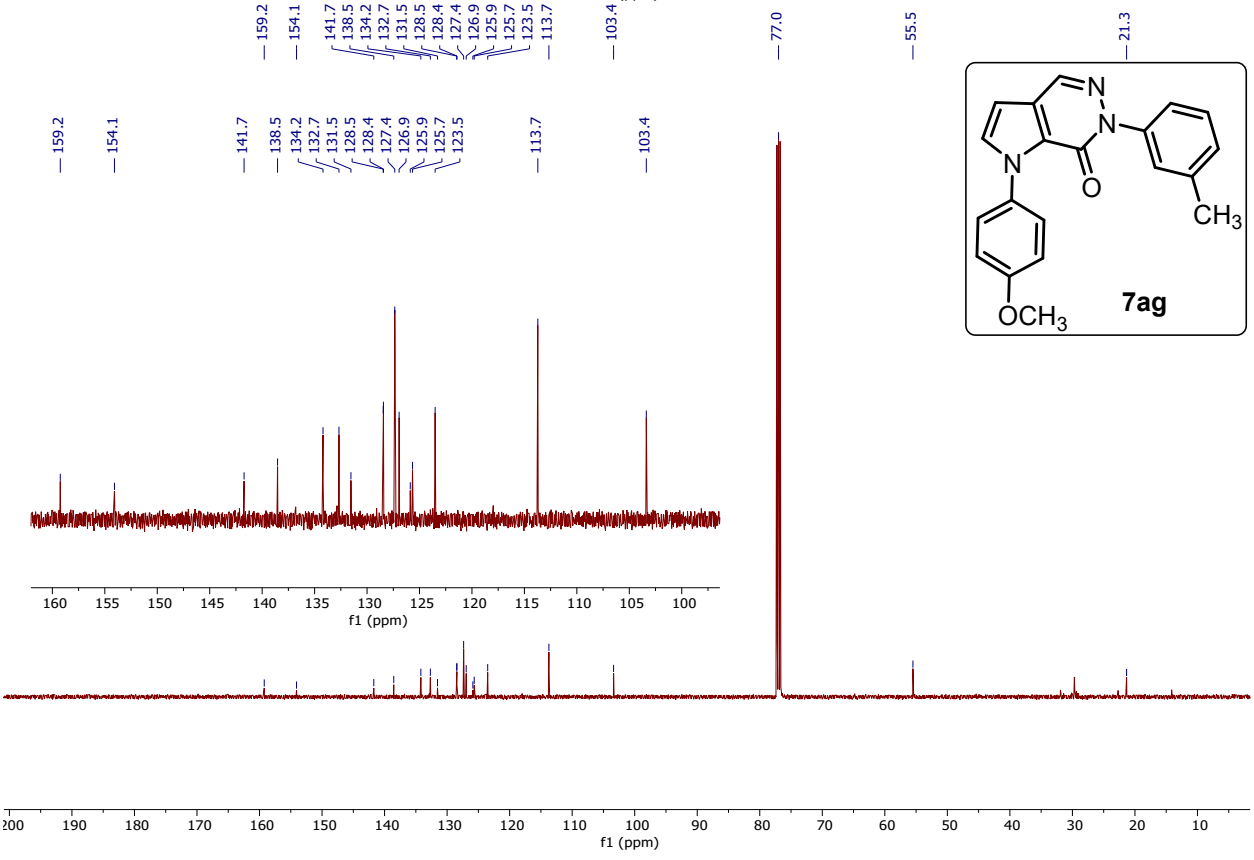
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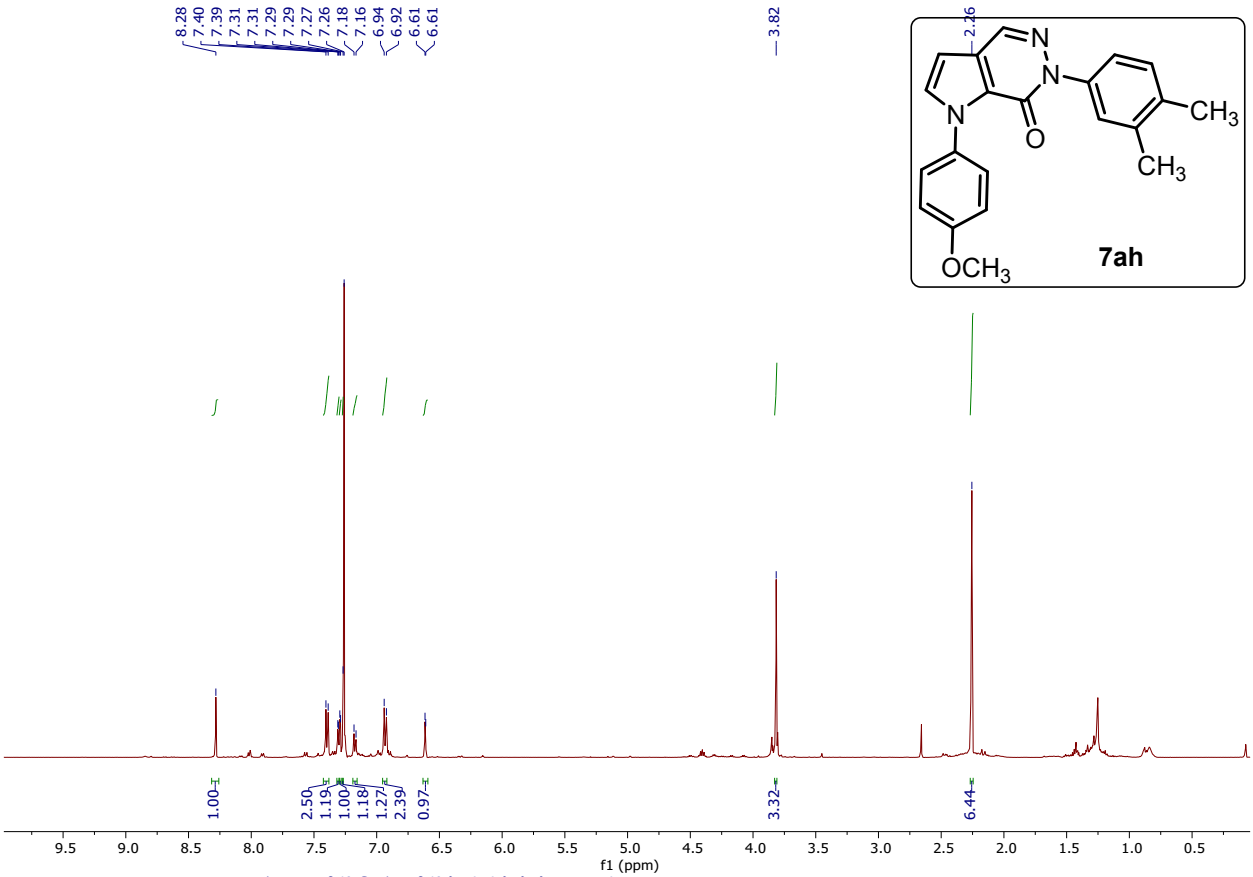
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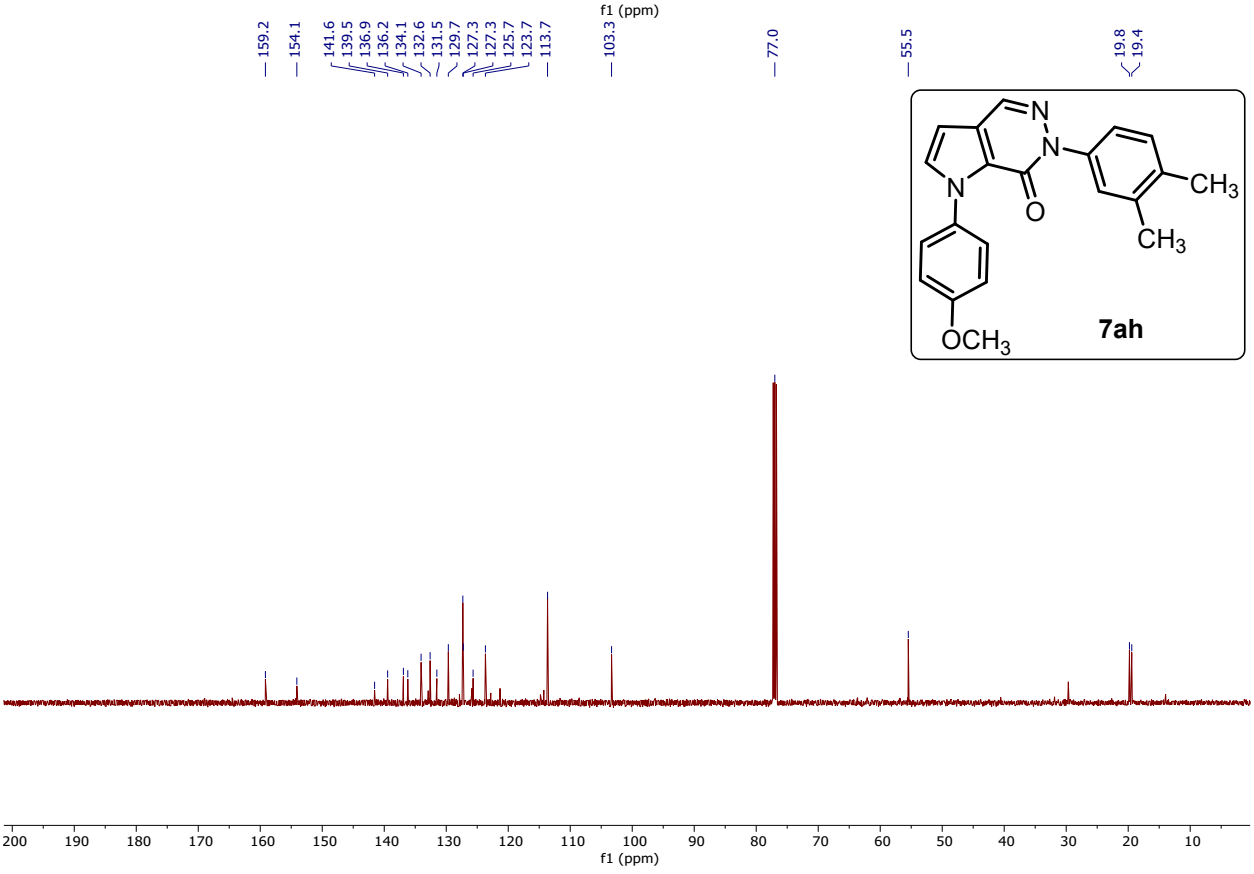
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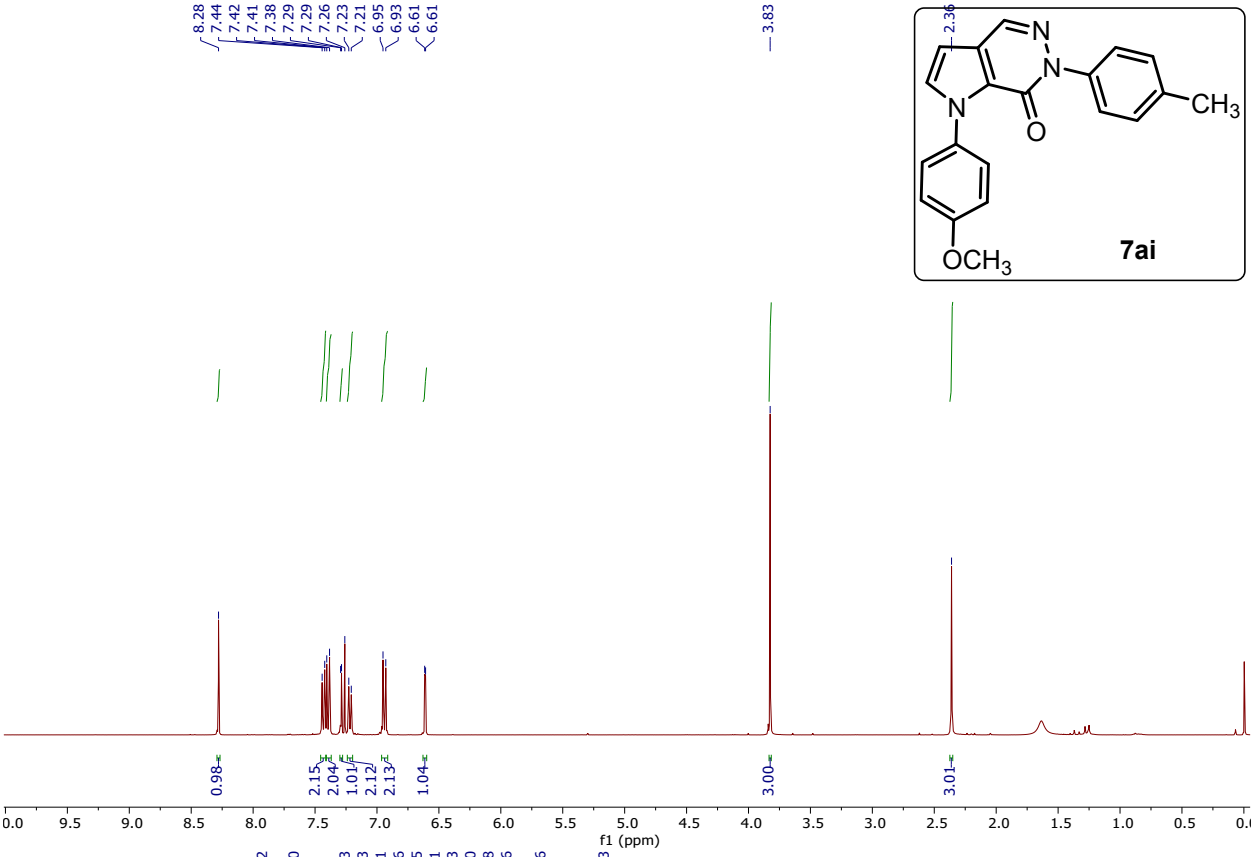
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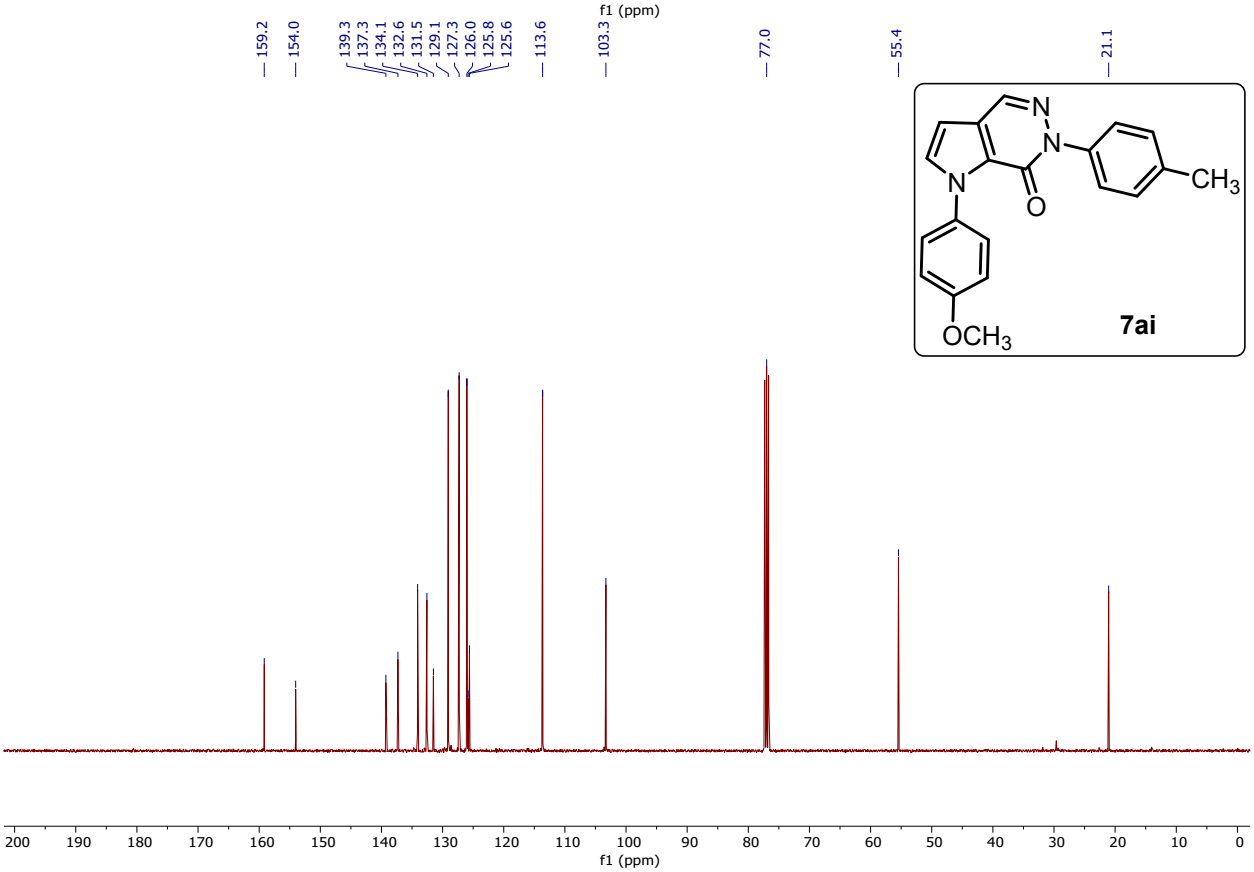
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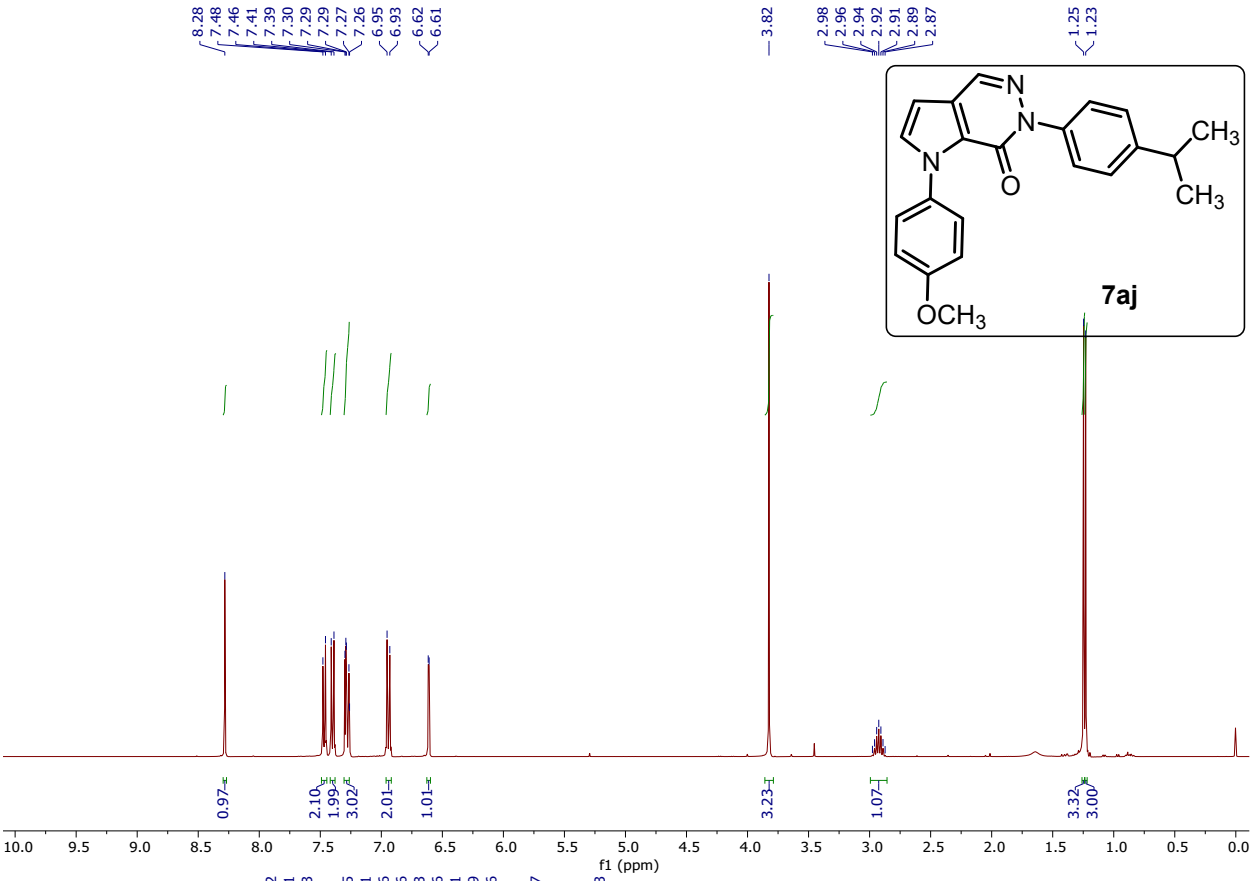
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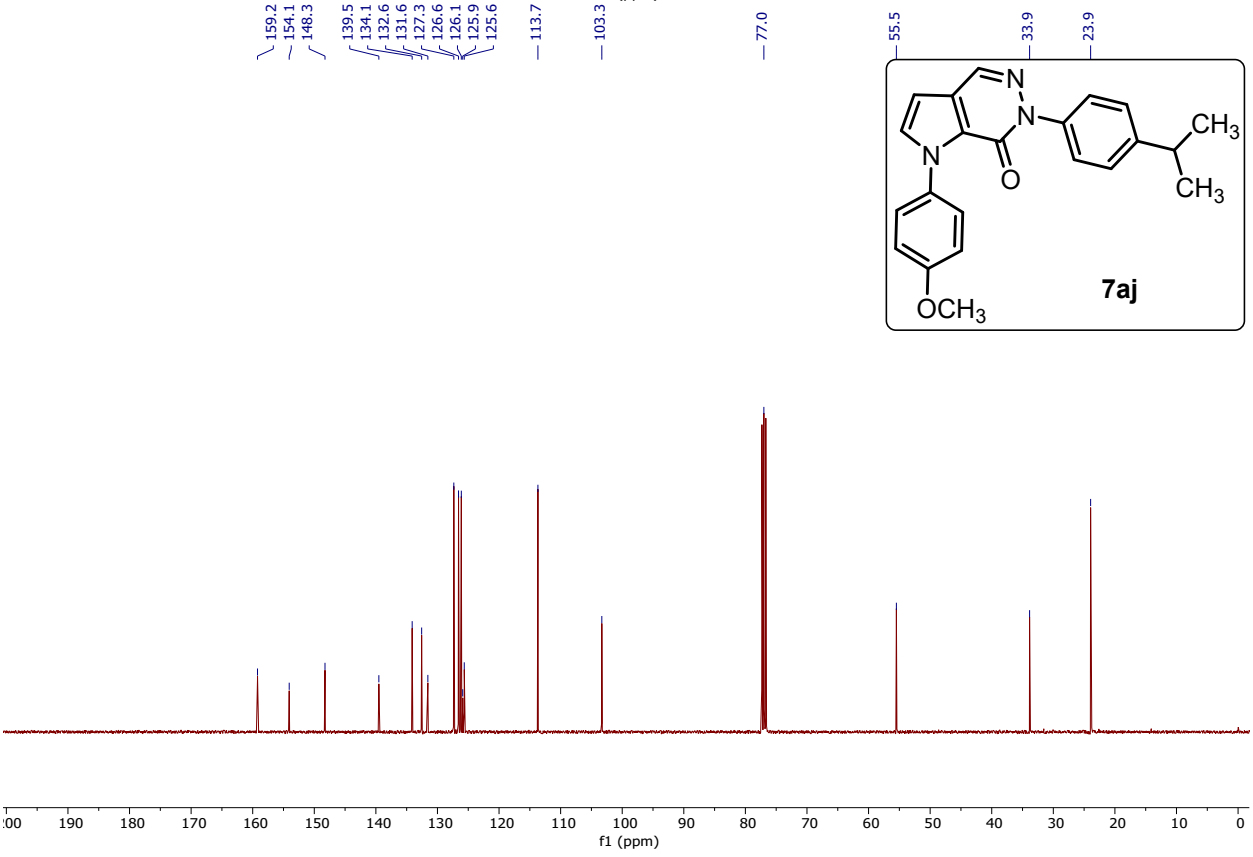
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CH3
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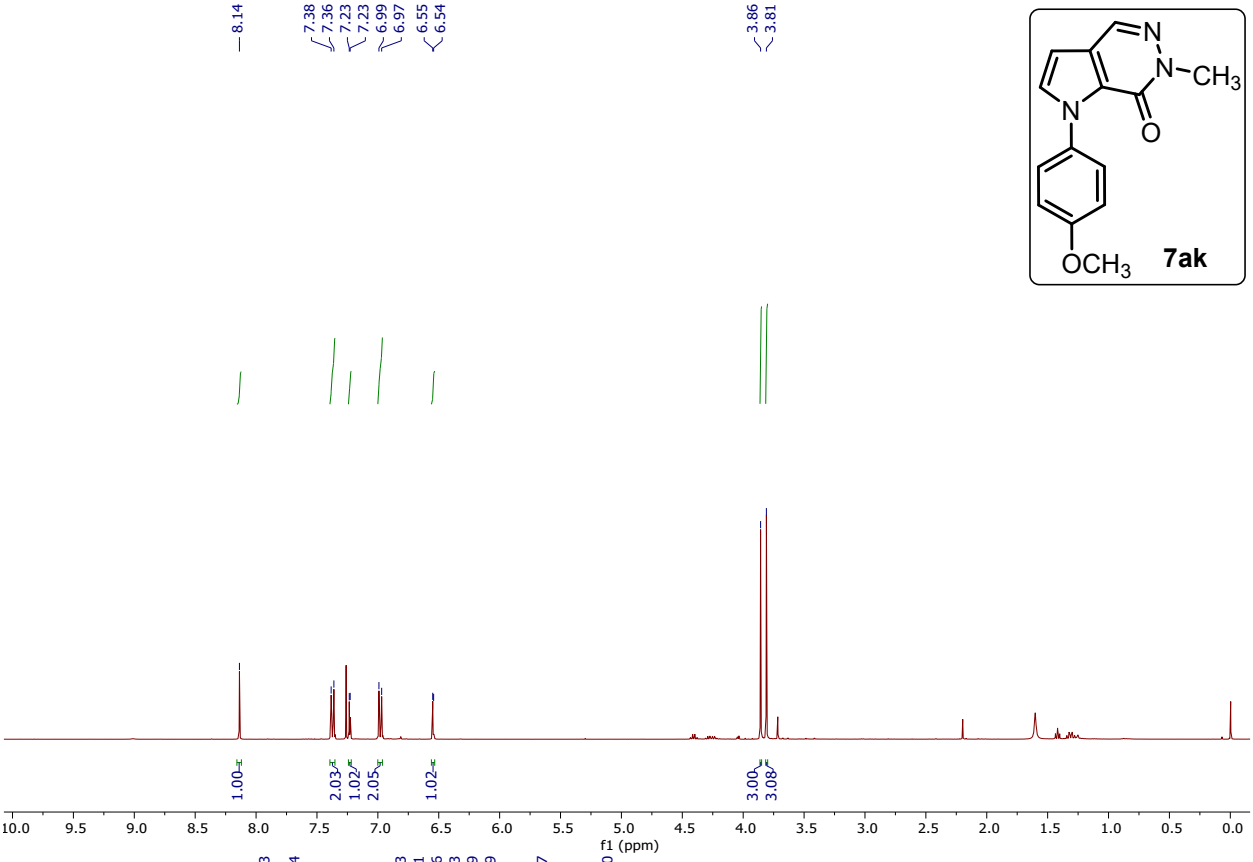
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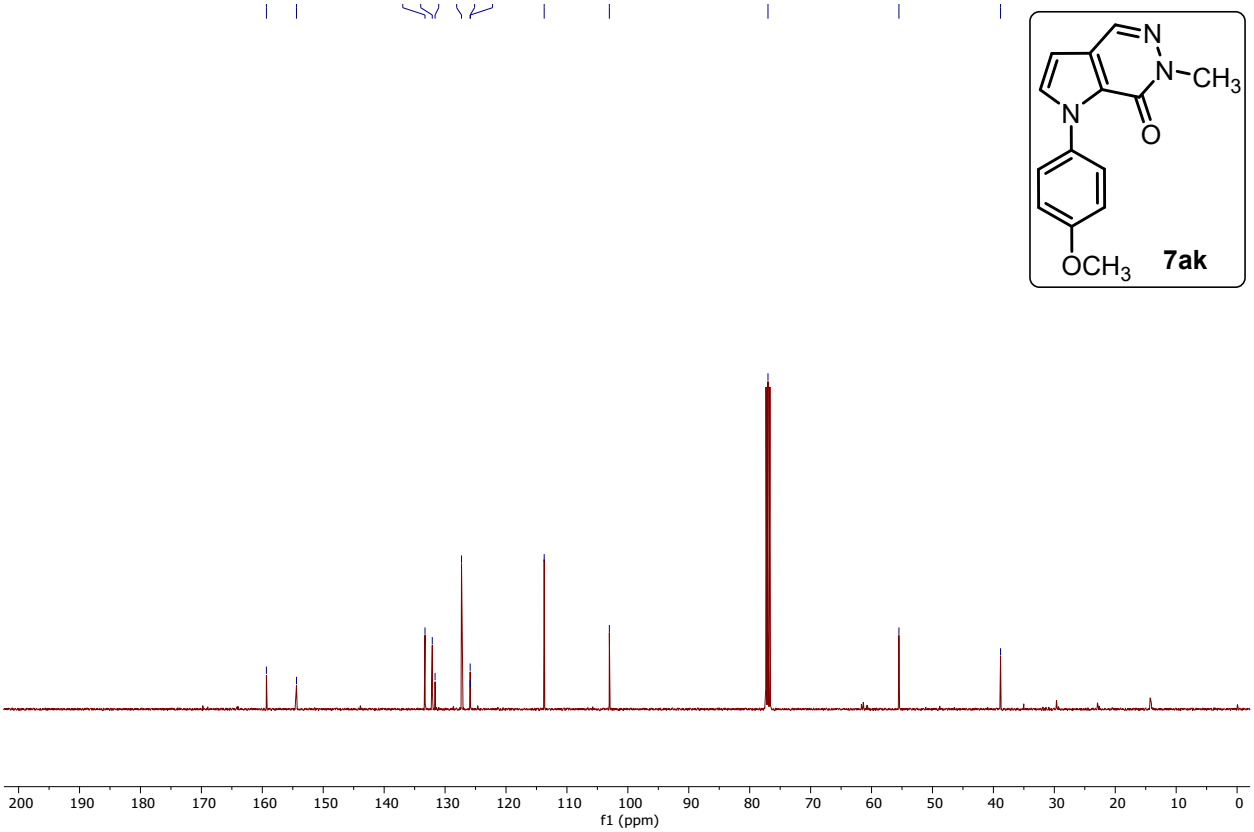
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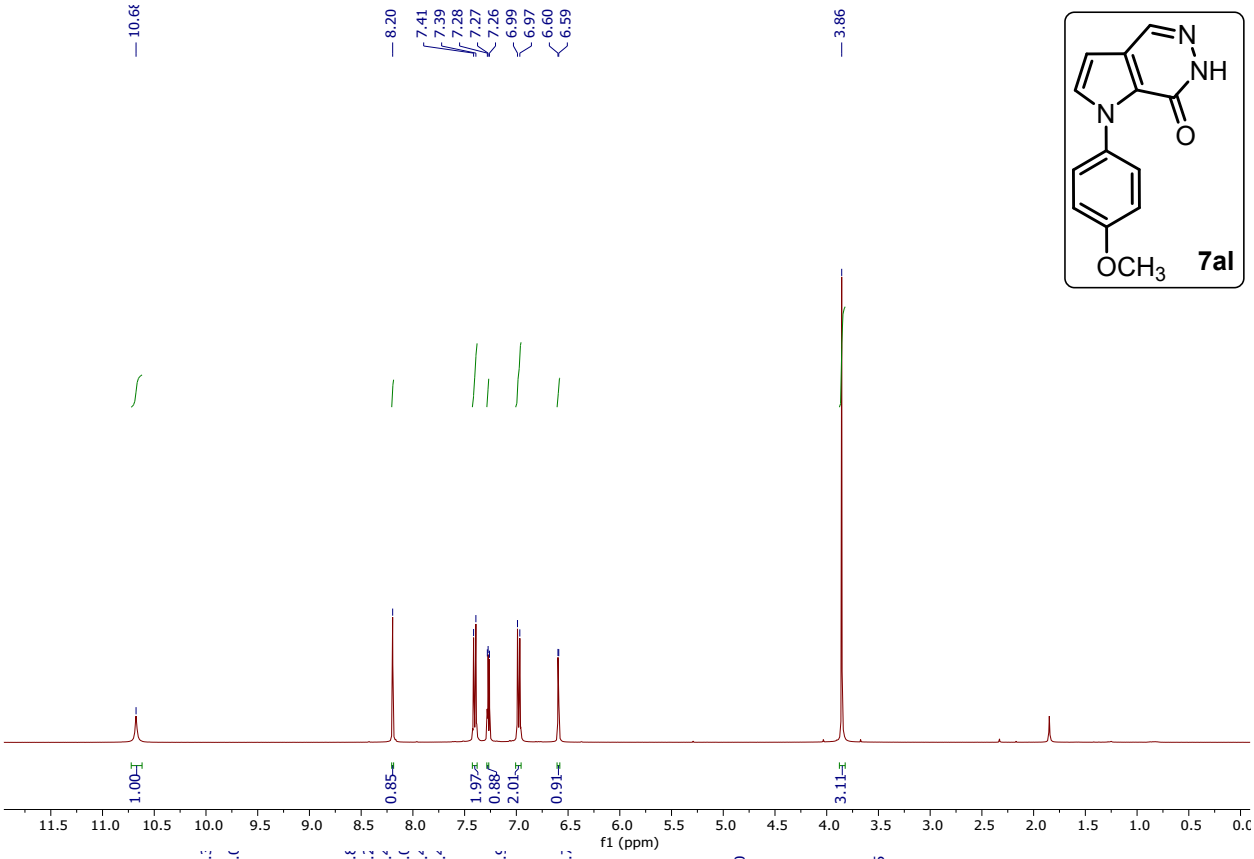
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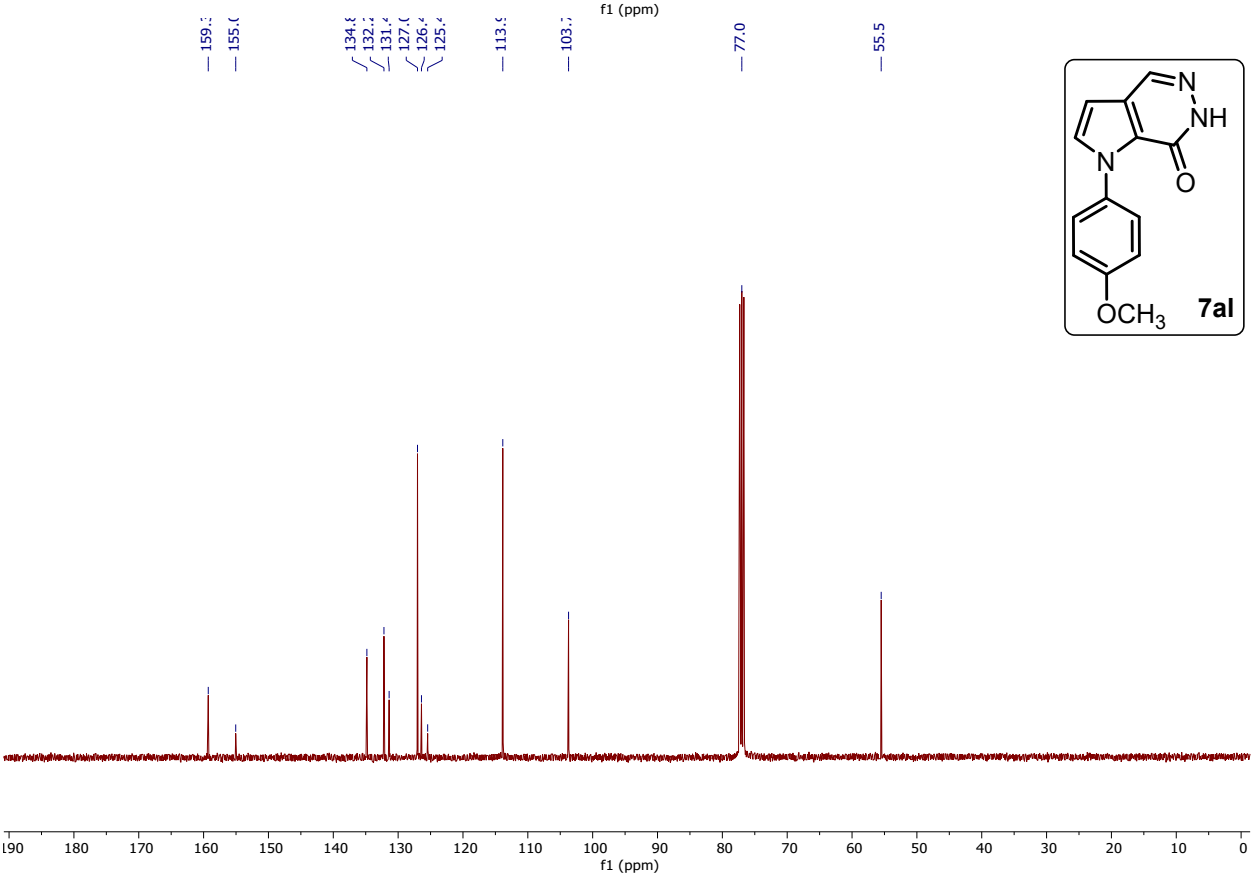
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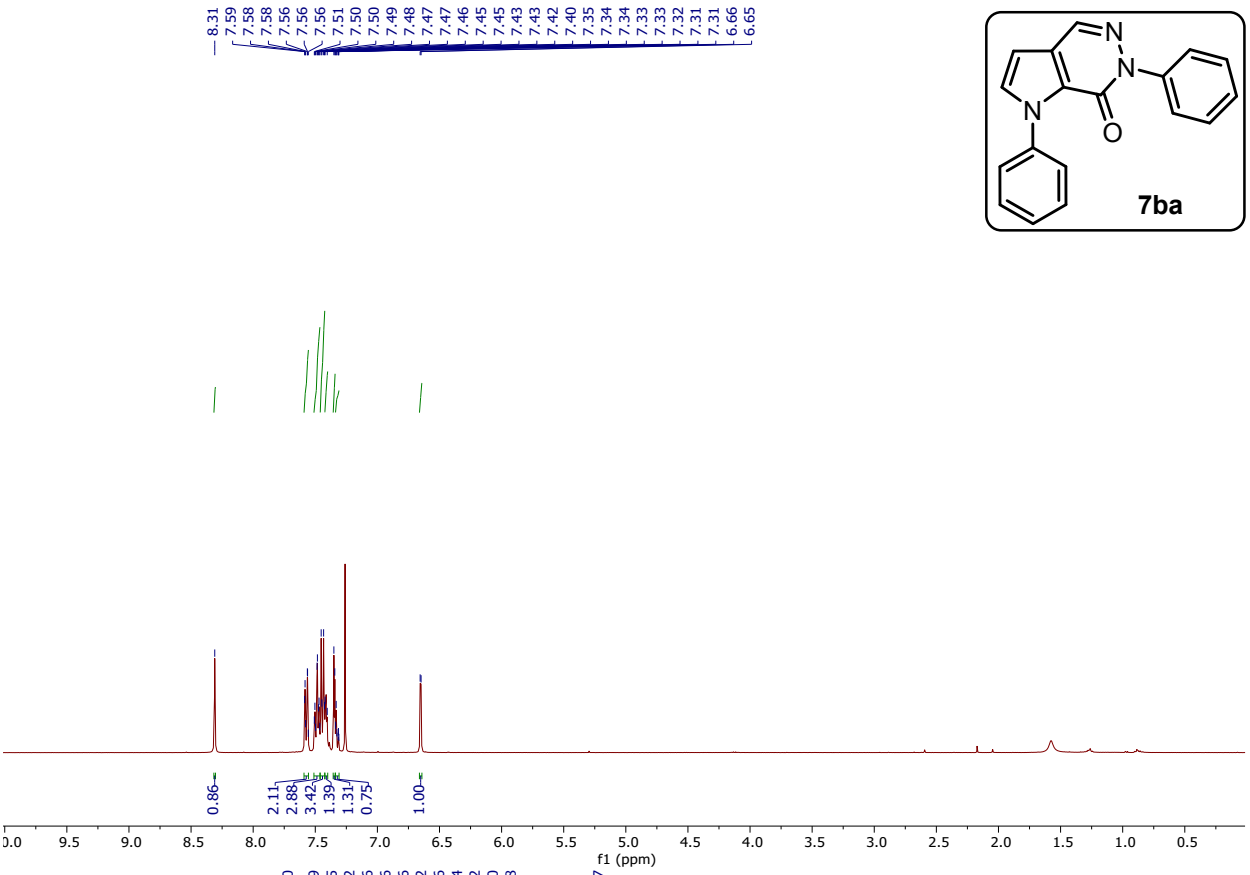
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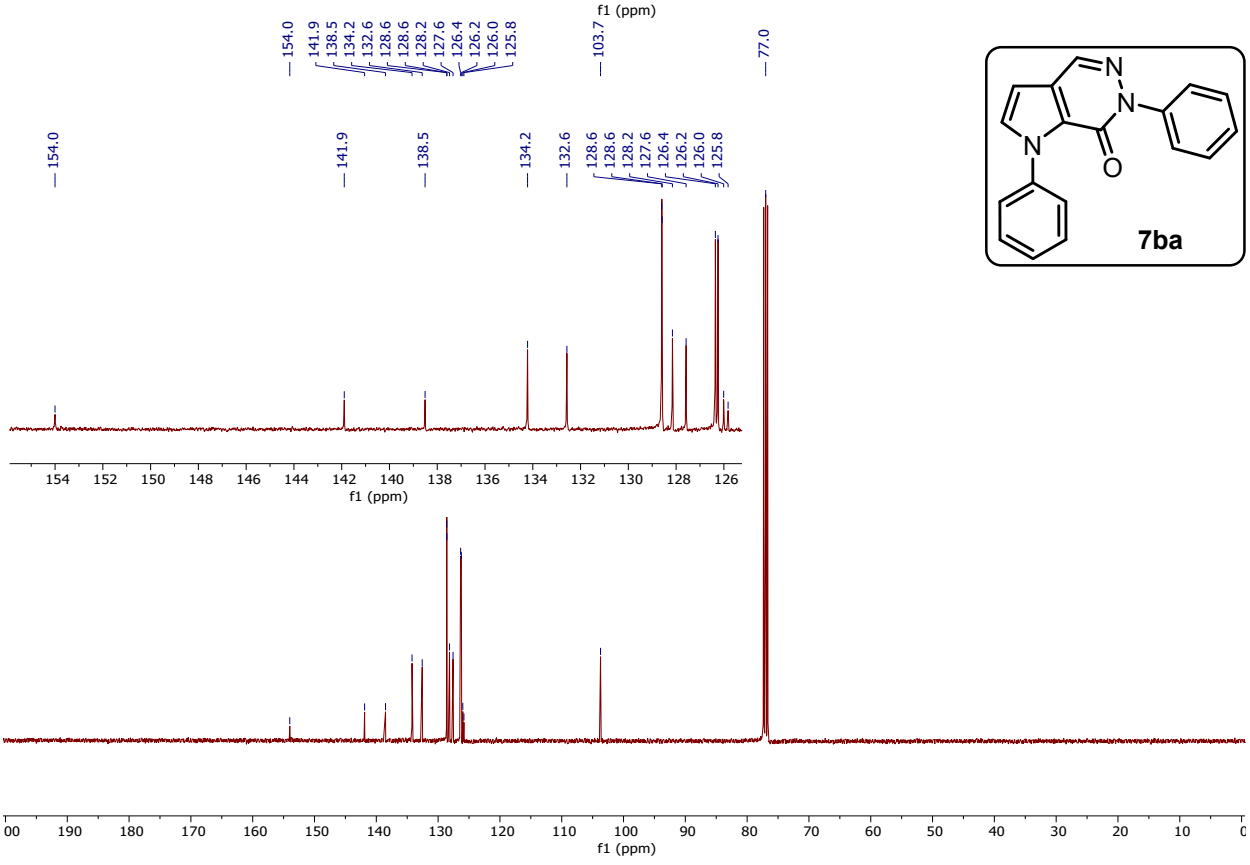
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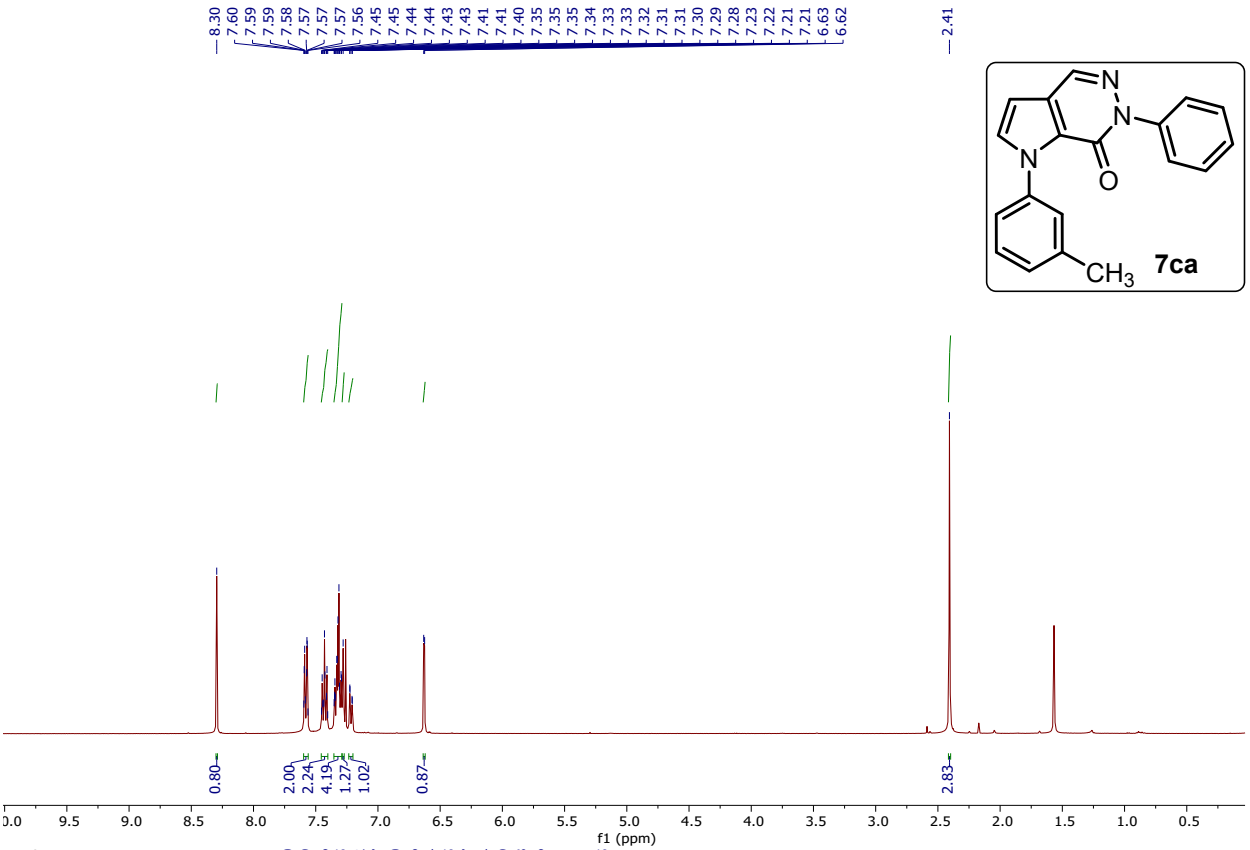
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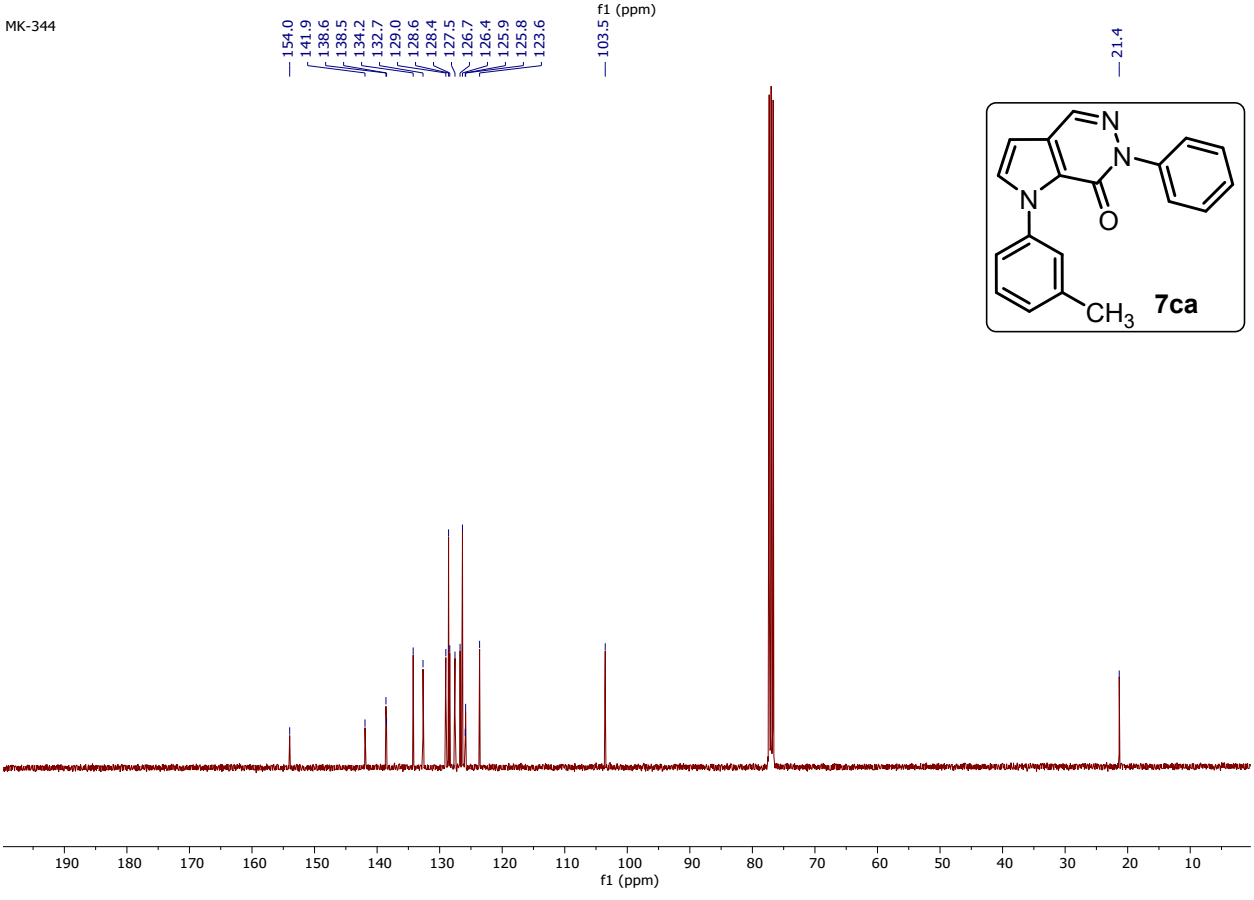
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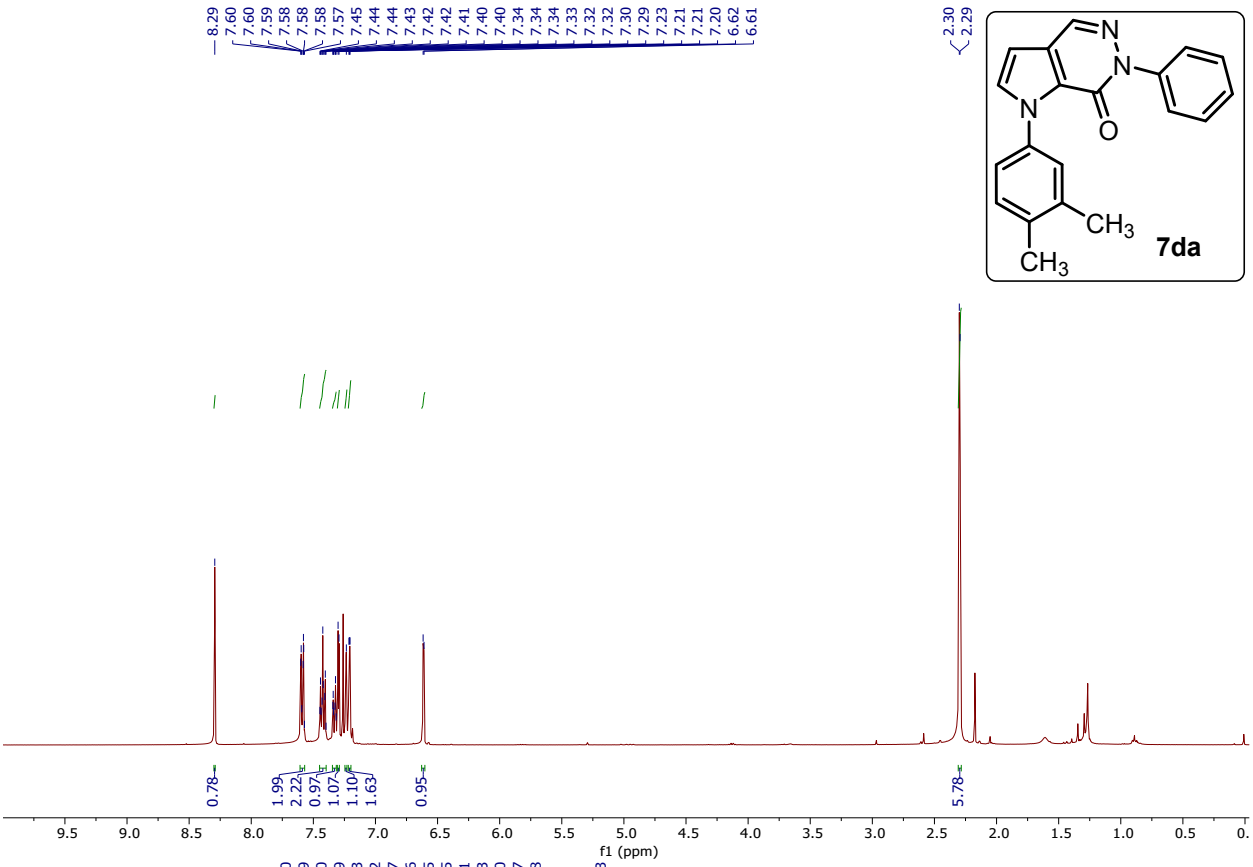
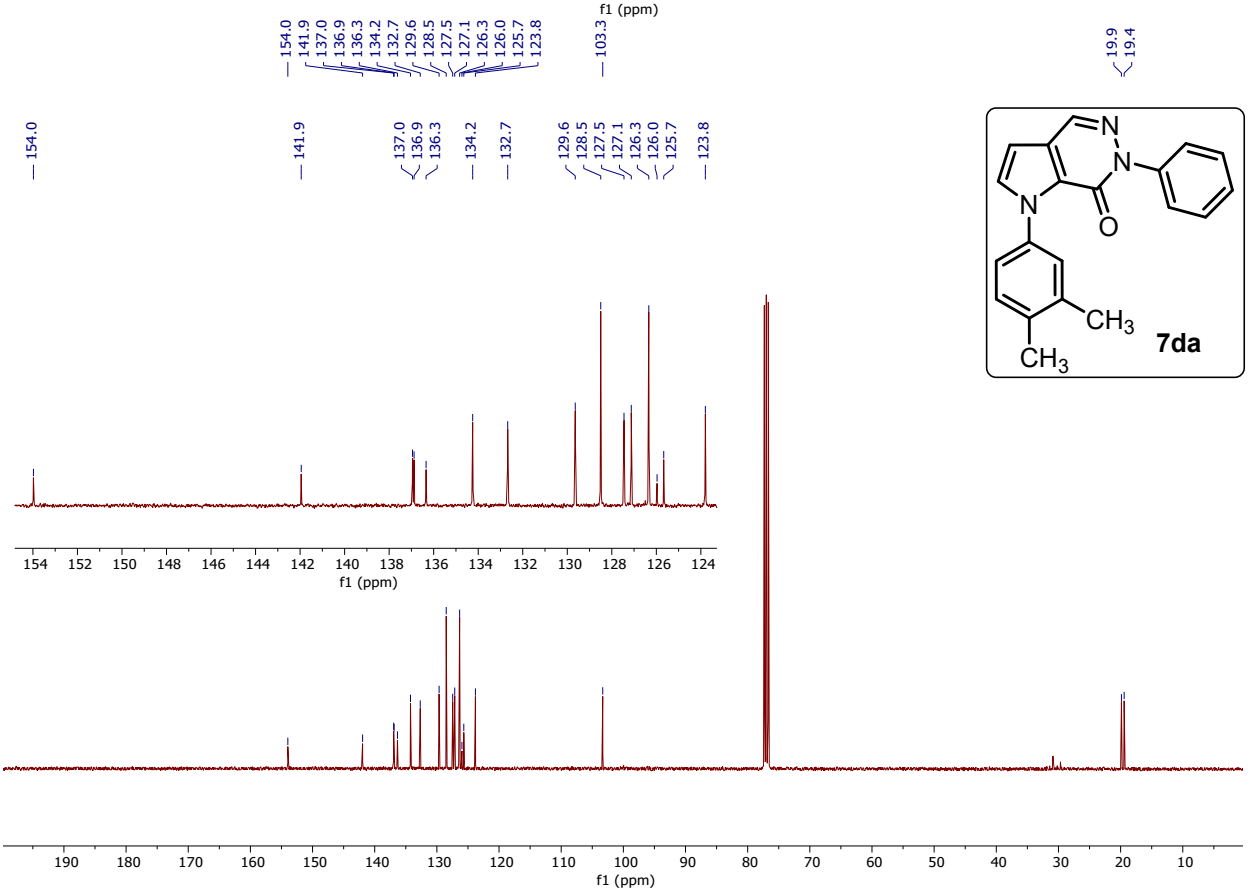
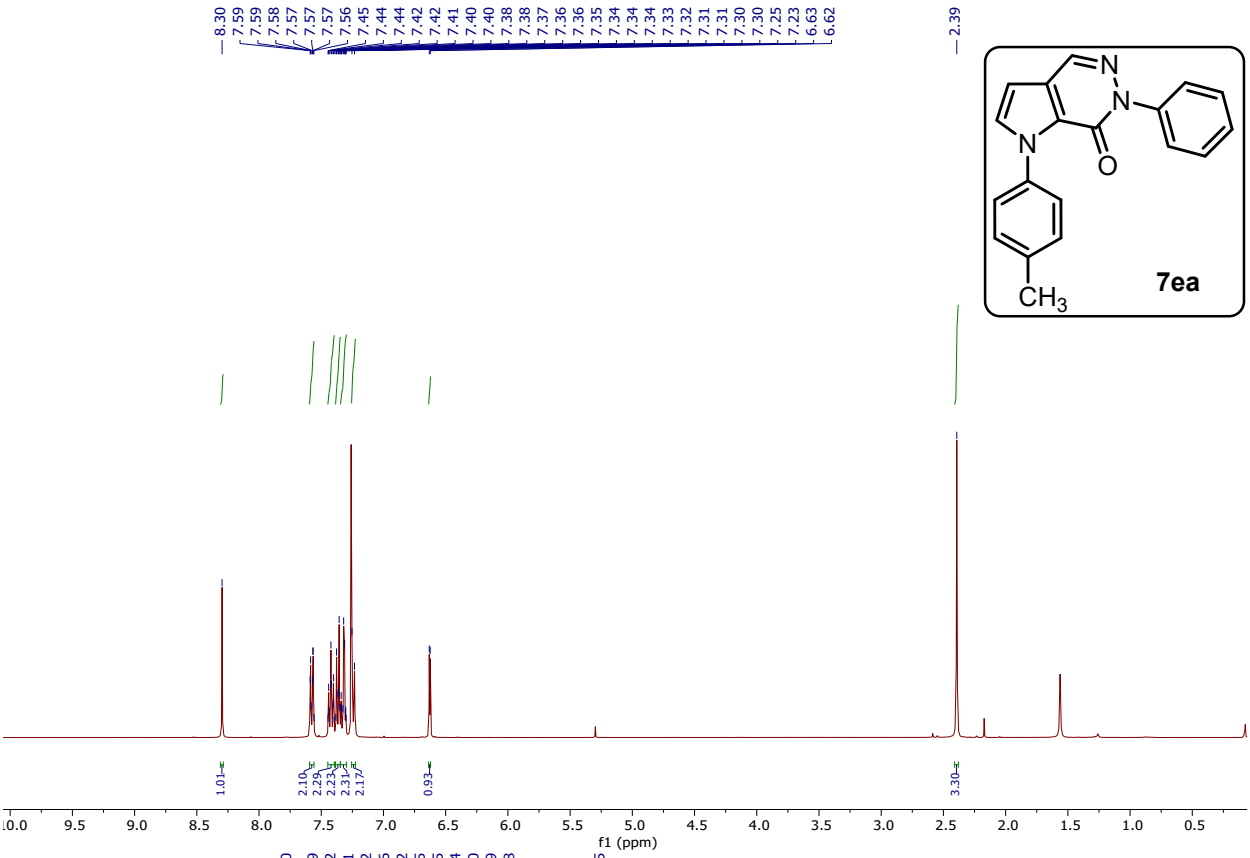
7ca
f1 (ppm)


8.29
7.60
7.60
7.59
7.58
7.58
7.58
7.57
7.45
7.44
7.44
7.43
7.42
7.42
7.41
7.40
7.40
7.34
7.34
7.34
7.33
7.32
7.32
7.30
7.29
7.23
7.21
7.21
7.20
6.62
6.61
2.30
2.29
0.78
1.99
2.22
0.97
1.07
1.10
1.63
0.95
5.78
f1 (ppm)
7da


154.0
141.9
137.0
136.9
136.3
134.2
132.7
129.6
128.5
127.5
127.1
126.3
126.0
125.7
123.8
103.3
19.9
19.4
f1 (ppm)
7da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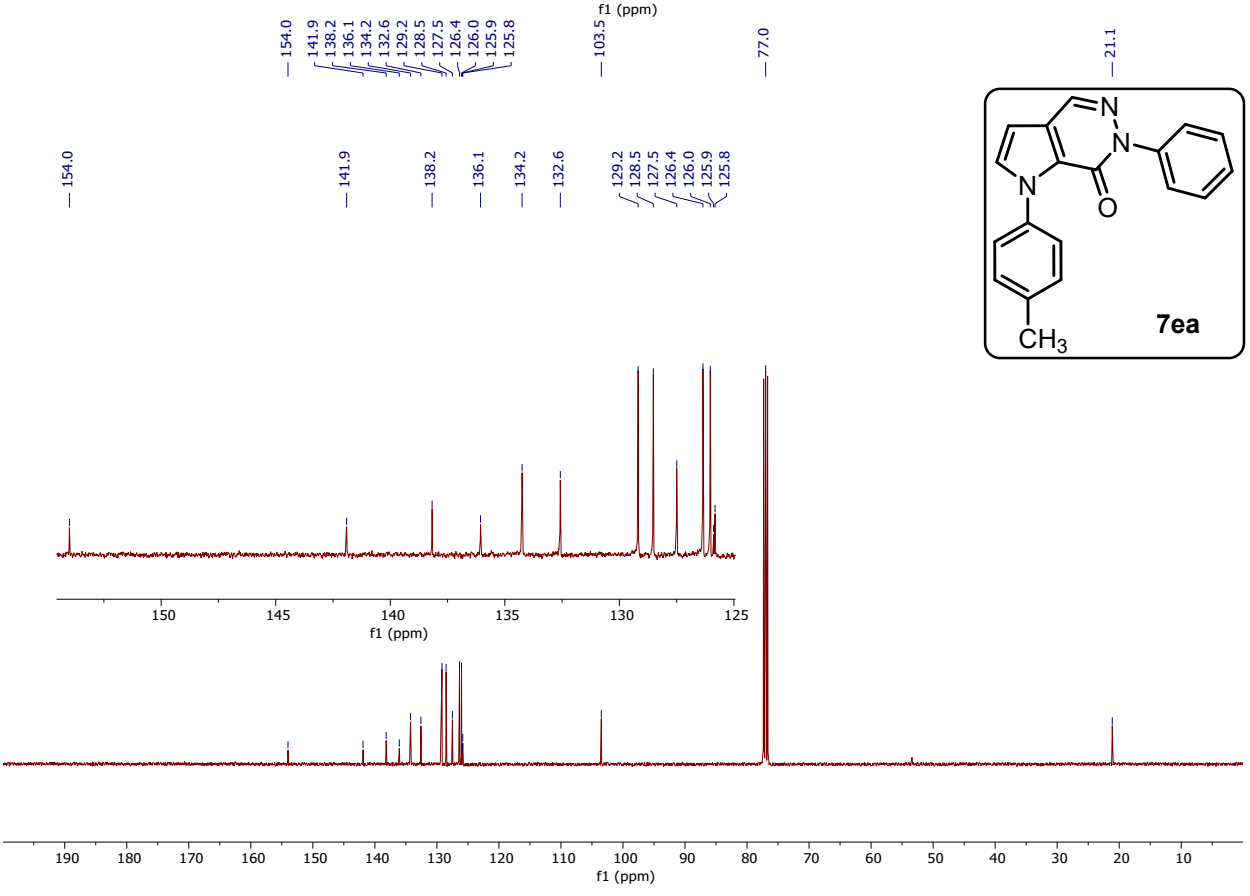
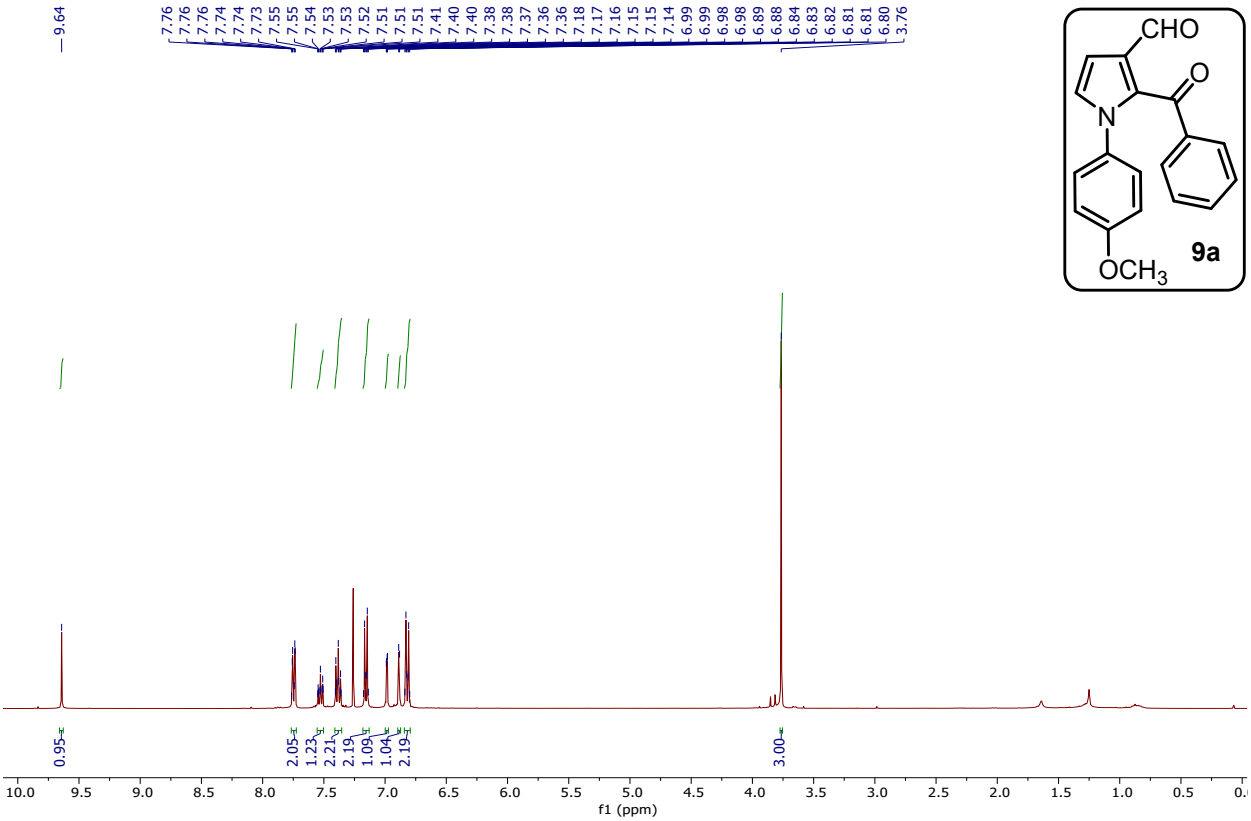
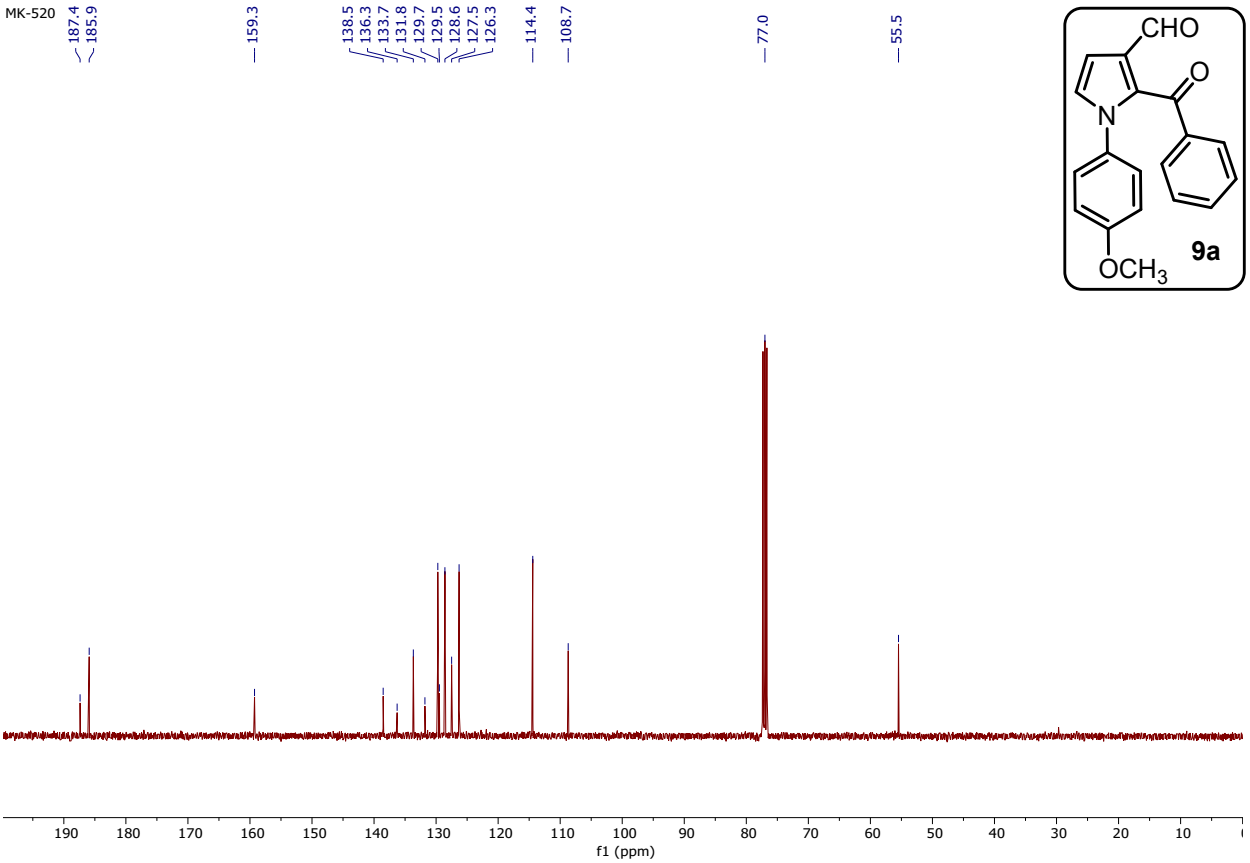
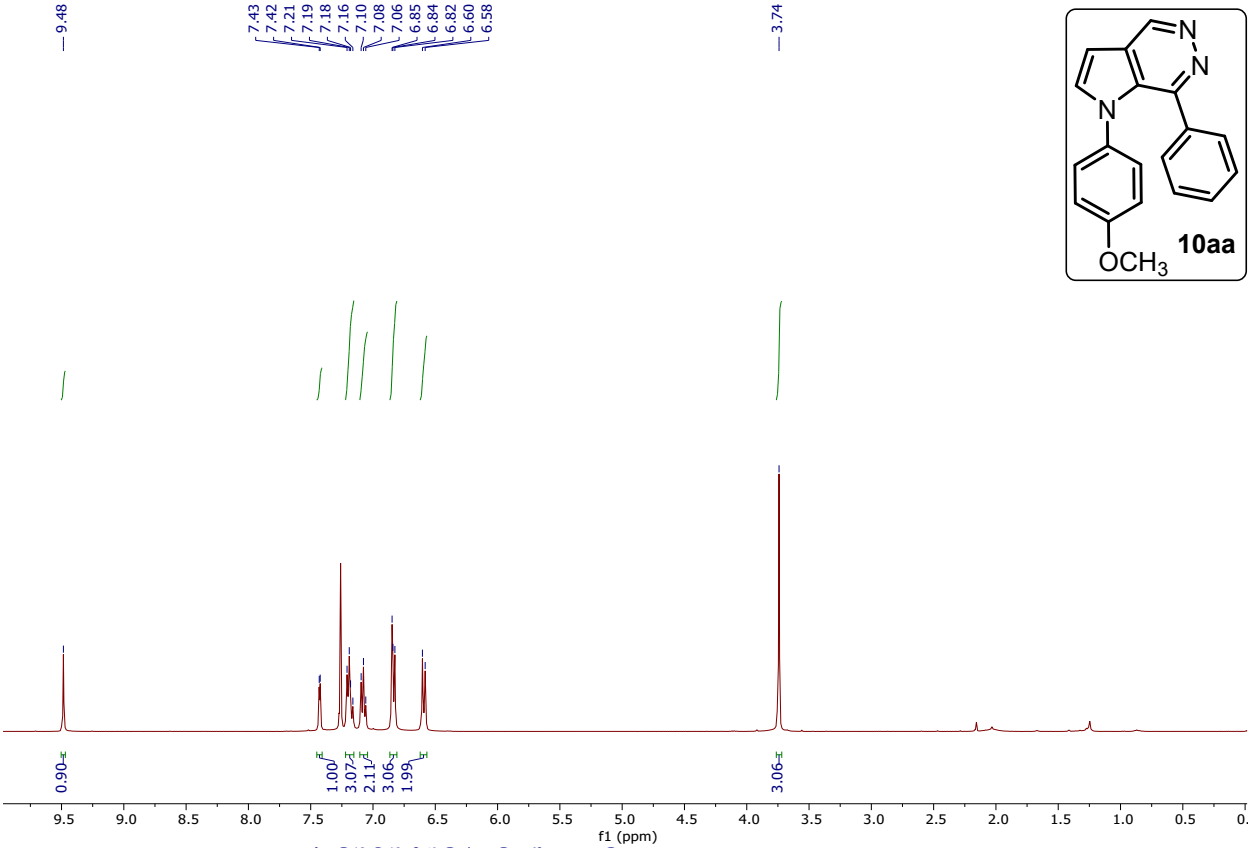
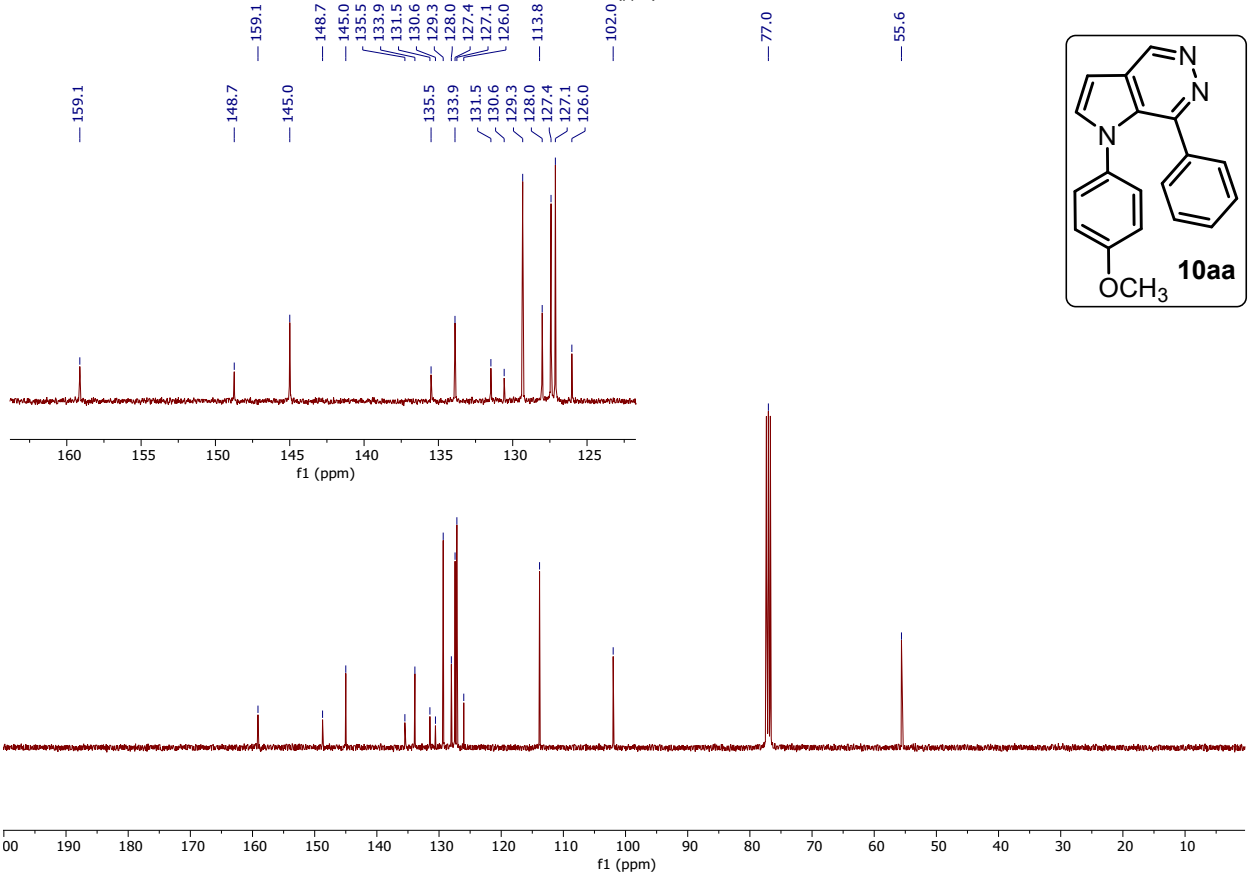
7.59
7.59
7.58
7.57
7.57
7.57
7.56
7.45
7.44
7.44
7.42
7.42
7.41
7.40
7.40
7.38
7.38
7.37
7.36
7.36
7.35
7.34
7.34
7.34
7.33
7.32
7.31
7.31
7.30
7.30
7.25
7.23
6.63
6.62
2.39
1.01
2.10
2.25
2.31
2.17
0.93
3.30
f1 (ppm)
7ea

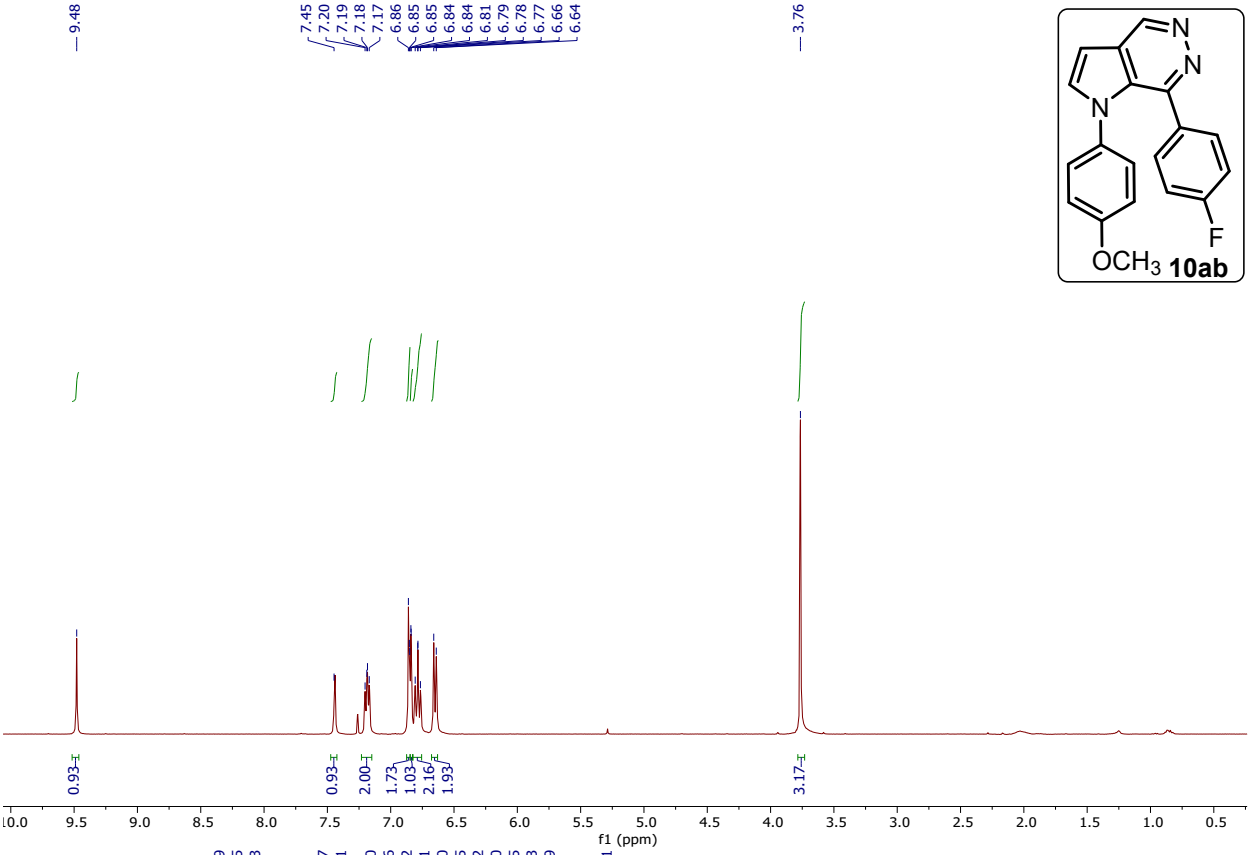

154.0
141.9
138.2
136.1
134.2
132.6
129.2
128.5
127.5
126.4
126.0
125.9
125.8
103.5
77.0
21.1
f1 (ppm)
7ea

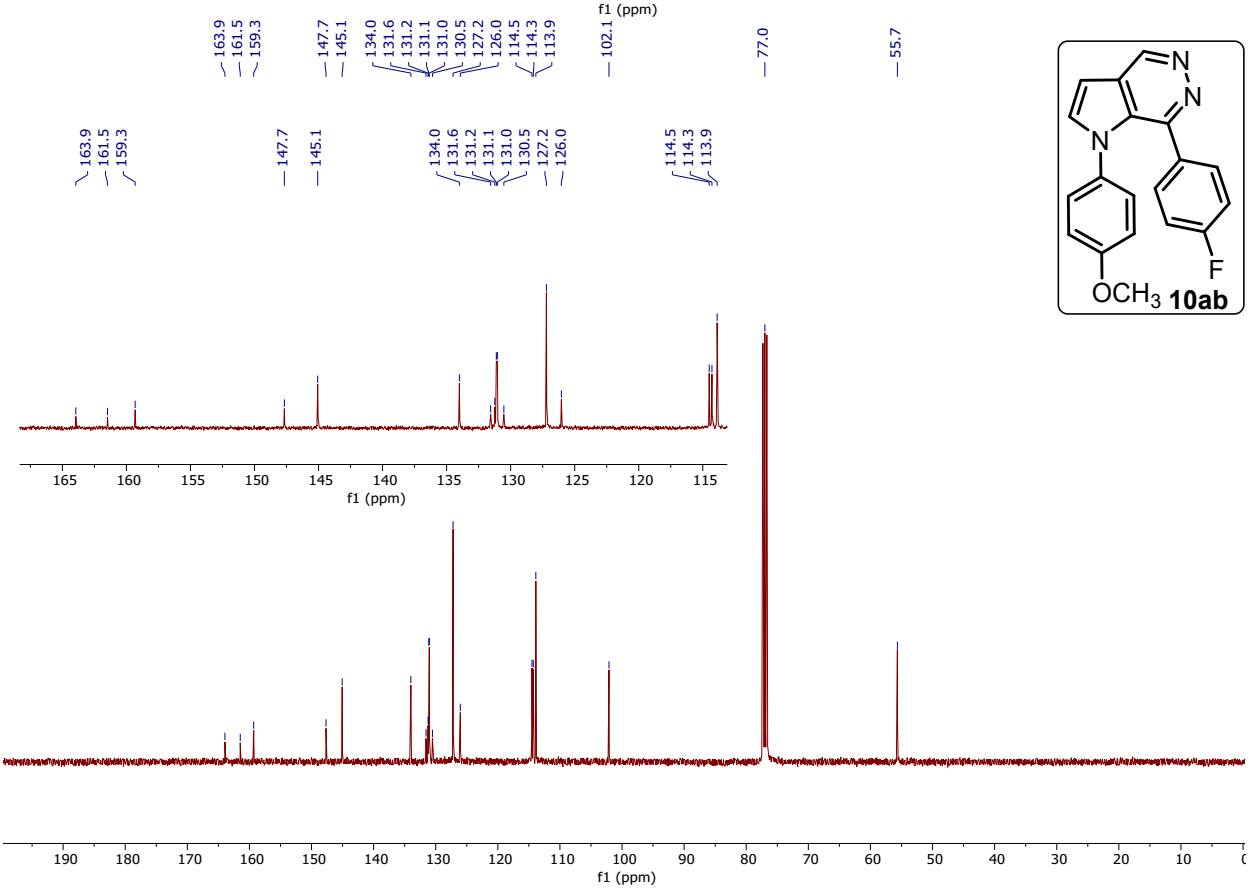

9.64
7.76
7.76
7.76
7.74
7.74
7.73
7.55
7.55
7.54
7.53
7.53
7.52
7.51
7.51
7.51
7.41
7.40
7.40
7.38
7.38
7.37
7.36
7.36
7.18
7.17
7.16
7.15
7.15
7.14
6.99
6.99
6.98
6.98
6.89
6.88
6.84
6.83
6.82
6.81
6.81
6.80
3.76
CHO
O
N
OCH3
9a
0.95
2.05
1.23
2.21
2.19
1.09
1.04
2.19
3.00
f1 (ppm)

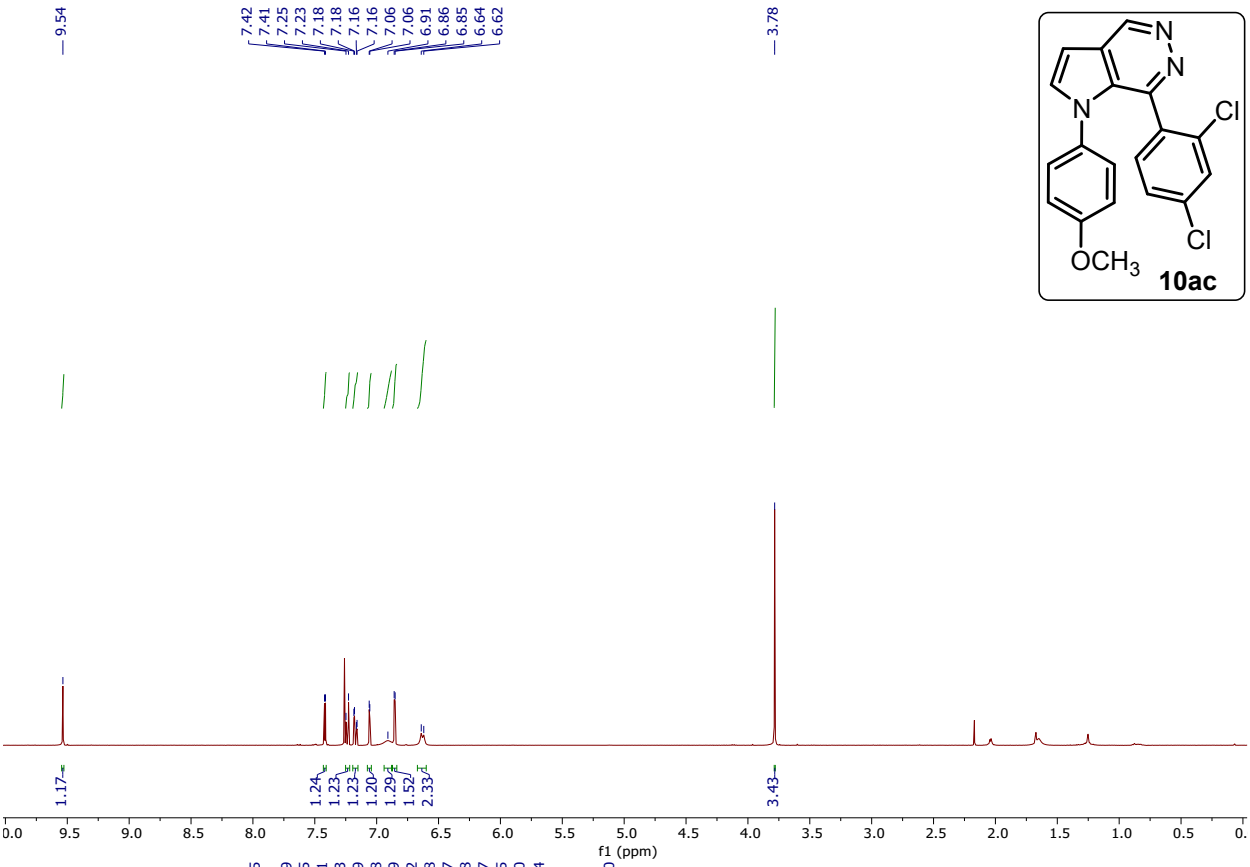

MK-520
187.4
185.9
159.3
138.5
136.3
133.7
131.8
129.7
129.5
128.6
127.5
126.3
114.4
108.7
77.0
55.5
CHO
O
N
OCH3
9a
f1 (ppm)

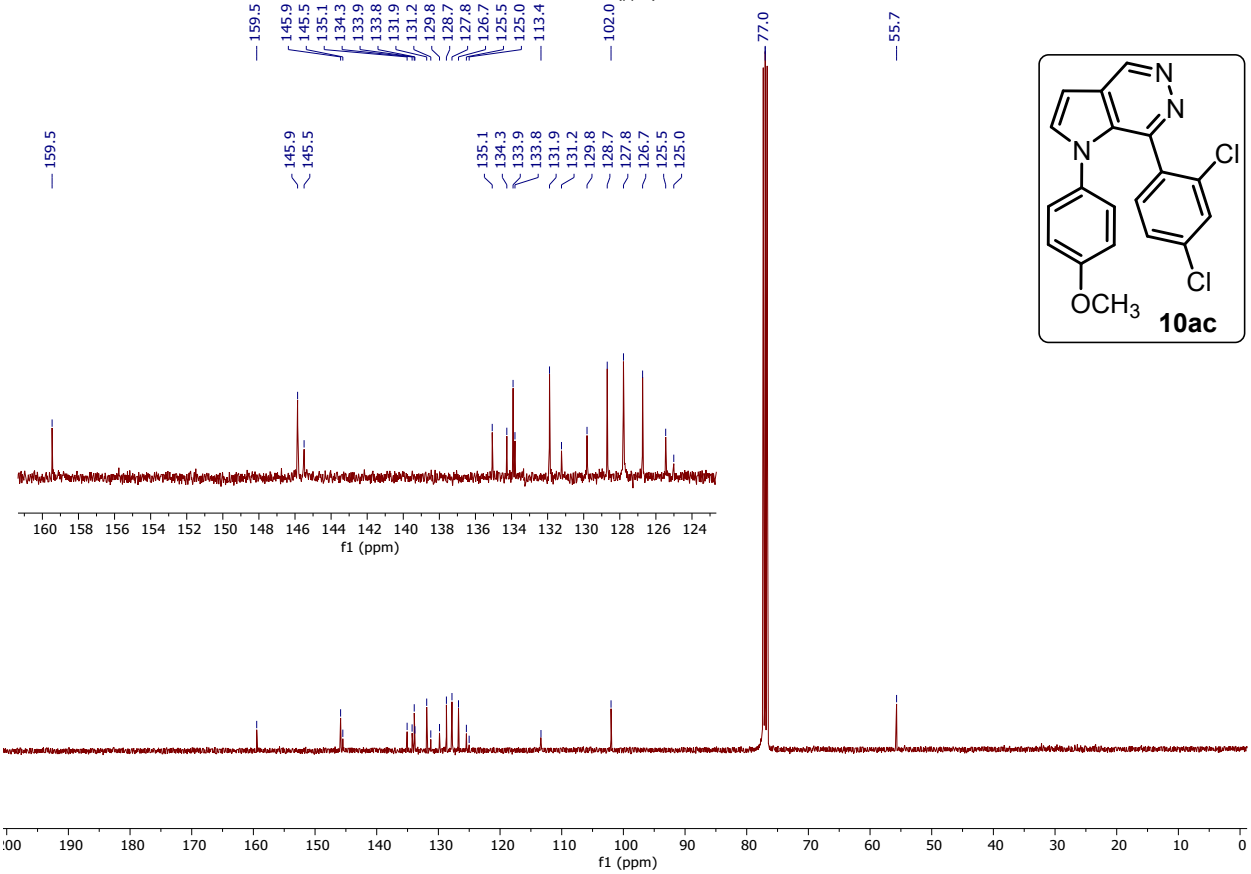

9.48
7.43
7.42
7.21
7.19
7.18
7.16
7.10
7.08
7.06
6.85
6.84
6.82
6.60
6.58
3.74
0.90
1.00
3.07
2.11
3.06
1.99
3.06
f1 (ppm)
10aa
OCH3


159.1
148.7
145.0
135.5
133.9
131.5
130.6
129.3
128.0
127.4
127.1
126.0
113.8
102.0
77.0
55.6
f1 (ppm)
10aa
OCH3


9.48
7.45
7.20
7.19
7.18
7.17
6.86
6.85
6.85
6.84
6.84
6.81
6.79
6.78
6.77
6.66
6.64
3.76
0.93
0.93
2.00
1.73
1.03
2.16
1.93
3.17
f1 (ppm)
OCH3 10ab


163.9
161.5
159.3
147.7
145.1
134.0
131.6
131.2
131.1
131.0
130.5
127.2
126.0
114.5
114.3
113.9
102.1
77.0
55.7
f1 (ppm)
OCH3 10ab


9.54
7.42
7.41
7.25
7.23
7.18
7.18
7.16
7.16
7.06
7.06
6.91
6.86
6.85
6.64
6.62
3.78
1.17
1.24
1.23
1.23
1.20
1.29
1.52
2.33
3.43
f1 (ppm)
10ac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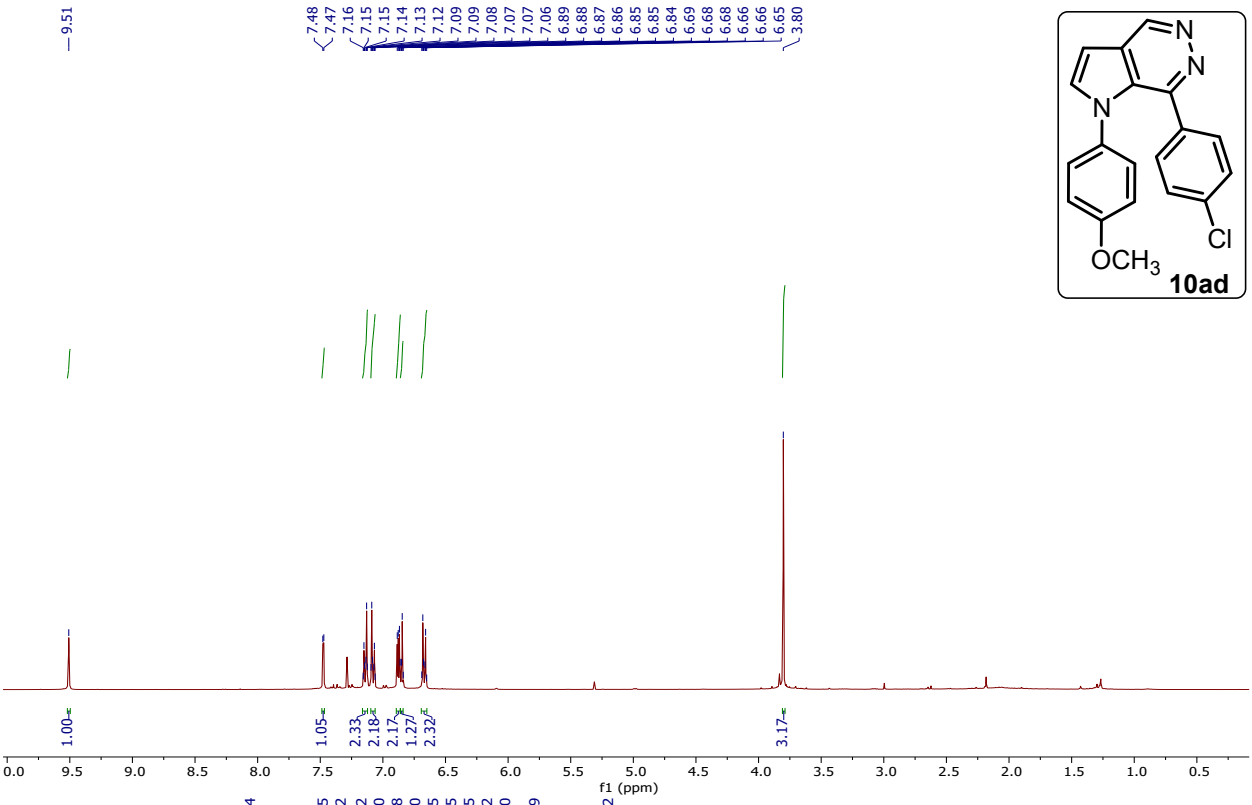
145.9
145.5
135.1
134.3
133.9
133.8
131.9
131.2
129.8
128.7
127.8
126.7
125.5
125.0
113.4
102.0
77.0
55.7
f1 (ppm)
10ac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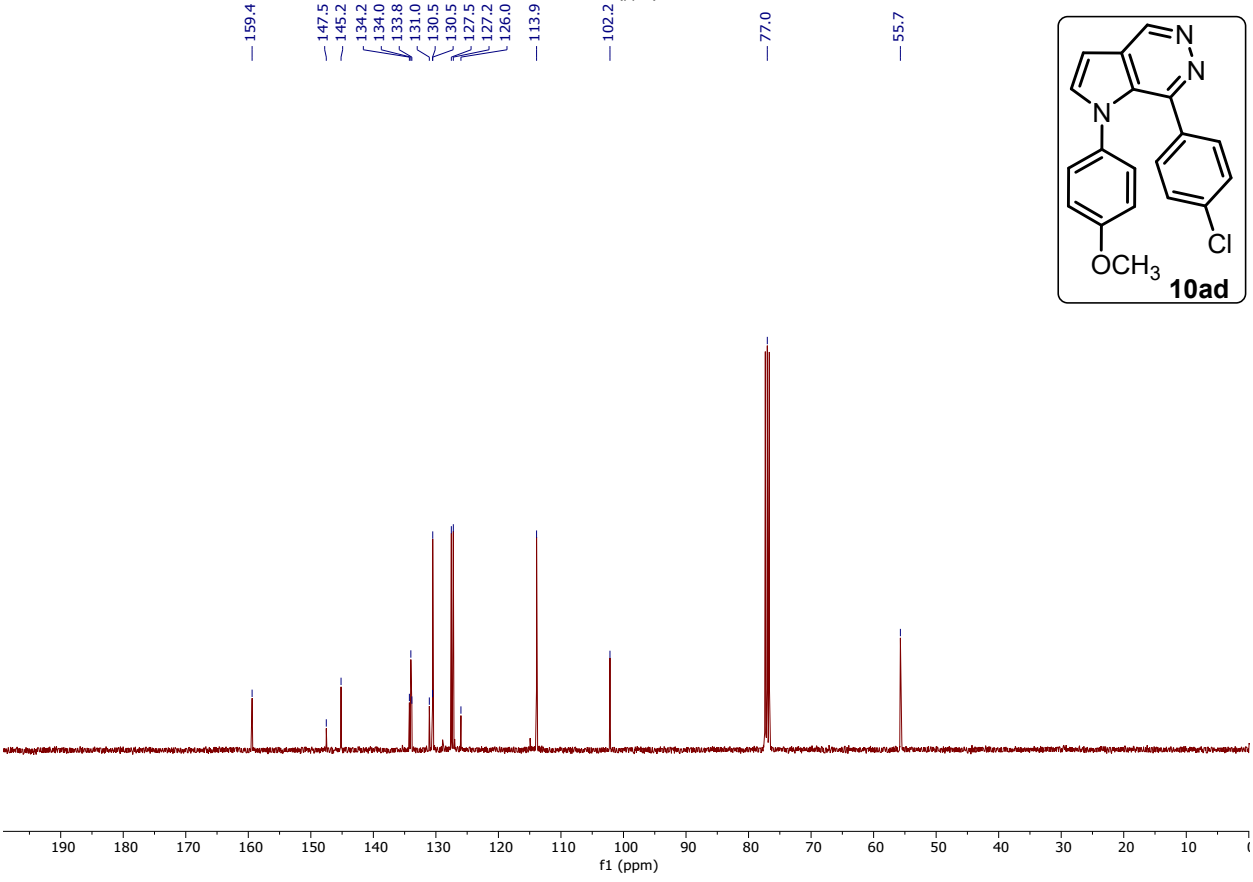
7.15
7.15
7.14
7.13
7.12
7.09
7.09
7.08
7.07
7.07
7.06
6.89
6.88
6.87
6.86
6.85
6.85
6.84
6.69
6.68
6.68
6.66
6.66
6.65
3.80
1.00
1.05
2.33
2.18
2.17
1.27
2.32
3.17
f1 (ppm)
N
N
N
Cl
OCH3
10ad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147.5
145.2
134.2
134.0
133.8
131.0
130.5
130.5
127.5
127.2
126.0
113.9
102.2
77.0
55.7
f1 (ppm)
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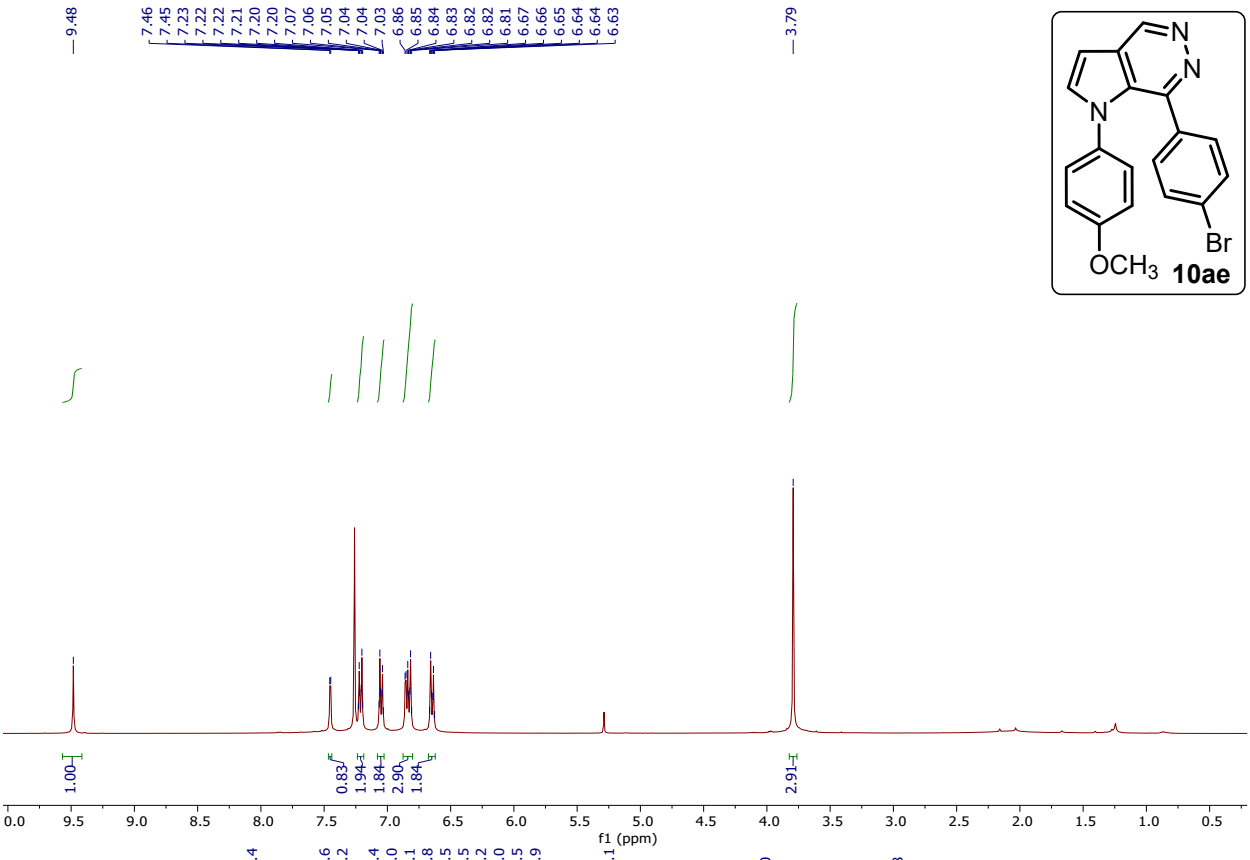
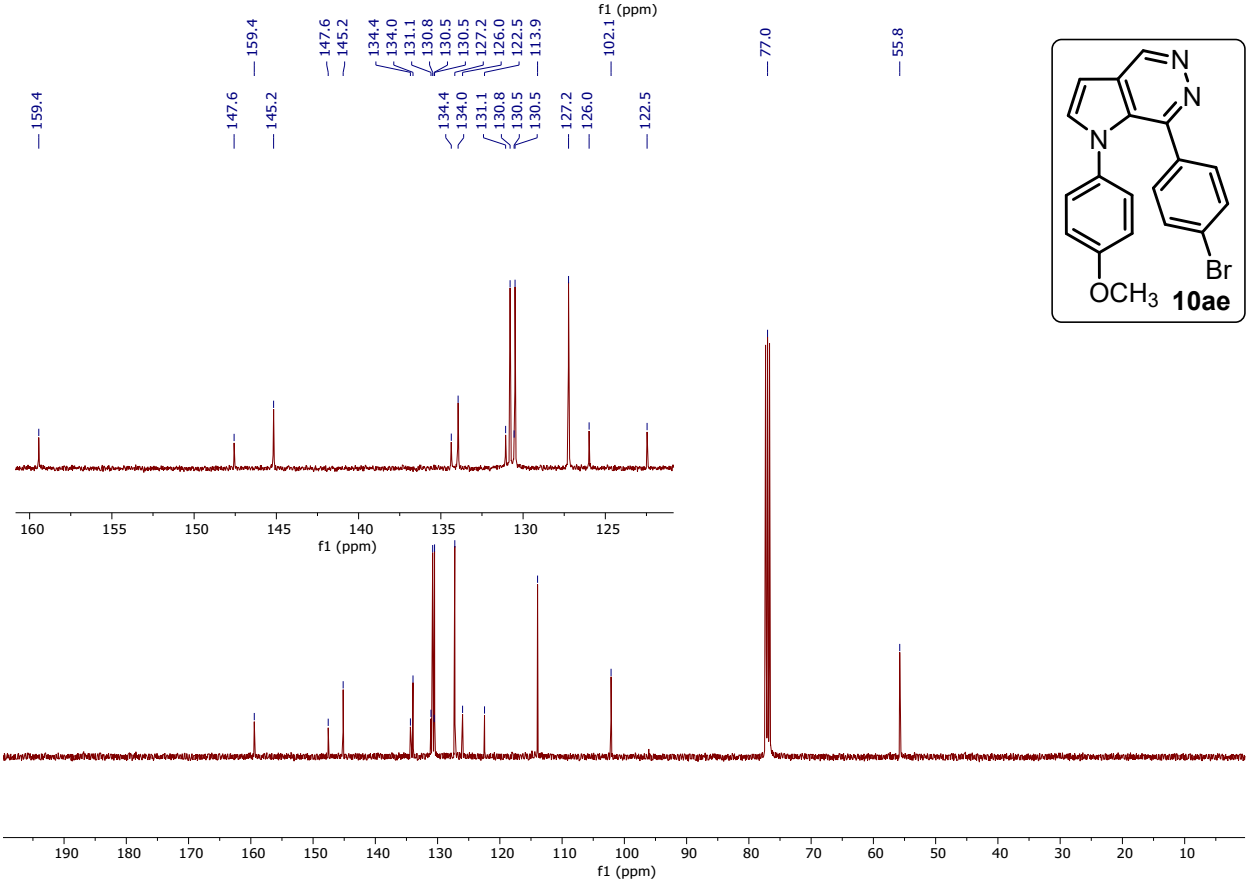
N
N
Cl
OCH3
10ad


9.48
7.46
7.45
7.23
7.22
7.22
7.21
7.20
7.20
7.07
7.06
7.05
7.04
7.04
7.03
6.86
6.85
6.84
6.83
6.82
6.82
6.81
6.67
6.66
6.65
6.64
6.64
6.63
3.79
1.00
0.83
1.94
1.84
2.90
1.84
2.91
f1 (ppm)
N
N
N
Br
OCH3
10ae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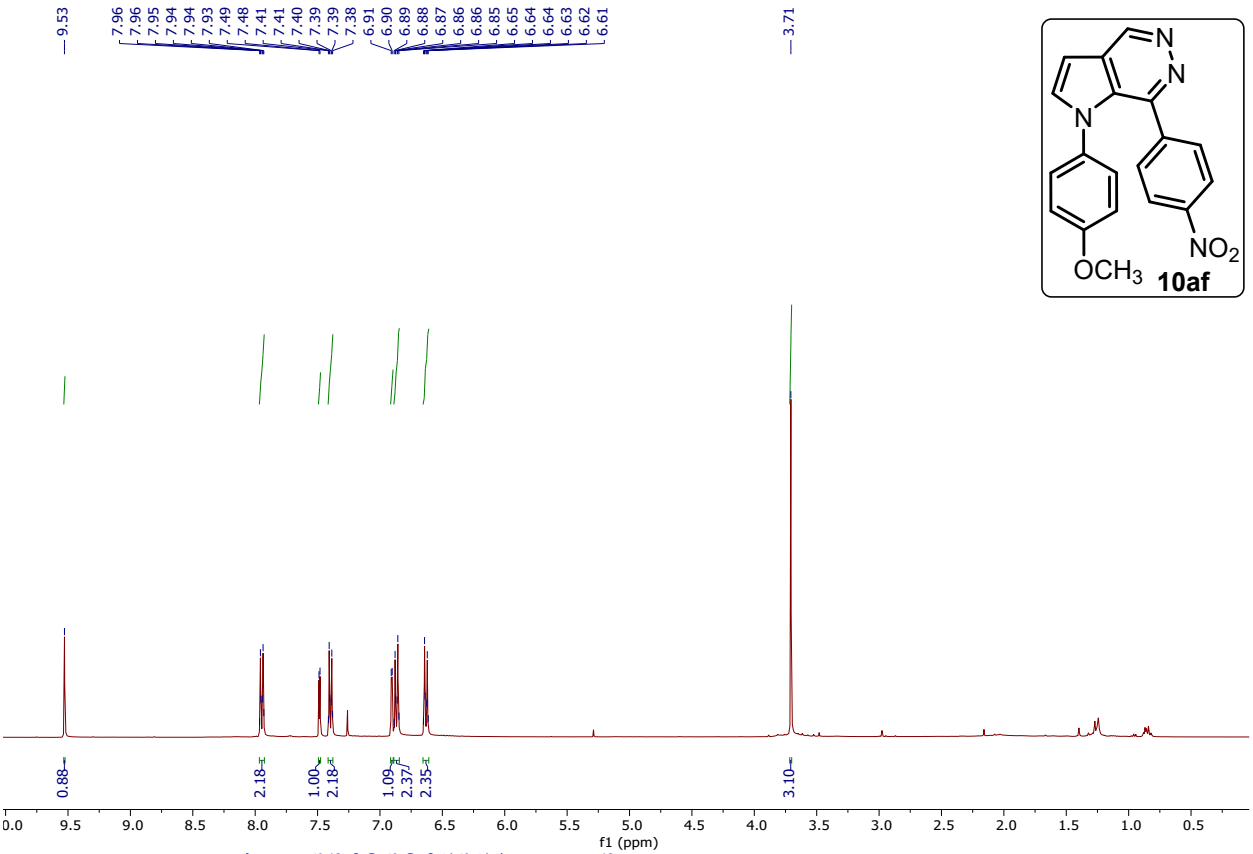
147.6
145.2
134.4
134.0
131.1
130.8
130.5
130.5
127.2
126.0
122.5
113.9
102.1
77.0
55.8
f1 (ppm)
N
N
N
Br
OCH3
10ae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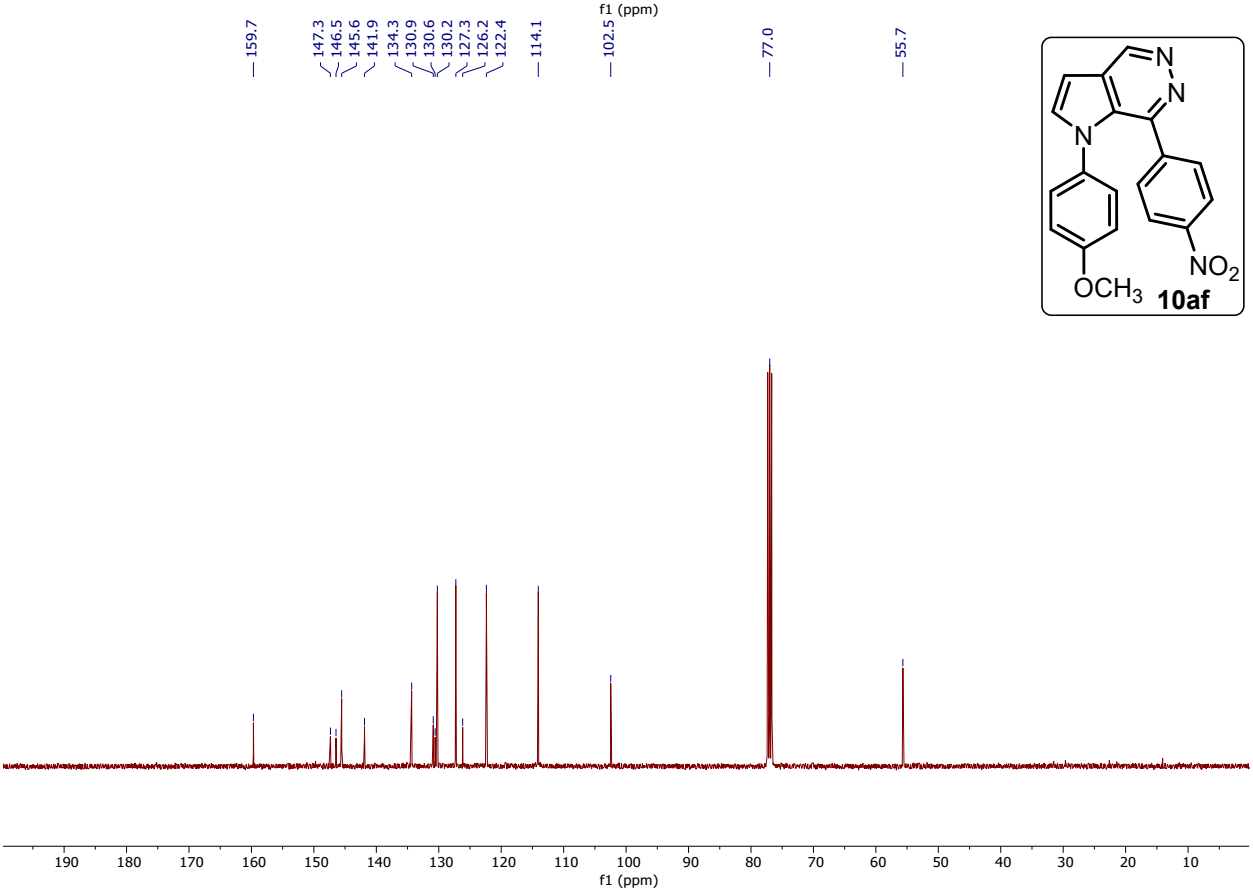
7.95
7.94
7.94
7.93
7.49
7.48
7.41
7.41
7.40
7.39
7.39
7.38
6.91
6.90
6.89
6.88
6.87
6.86
6.86
6.85
6.65
6.64
6.64
6.63
6.62
6.61
3.71
0.88
2.18
1.00
2.18
1.09
2.37
2.35
3.10
f1 (ppm)
N
N
N
NO2
OCH3
10af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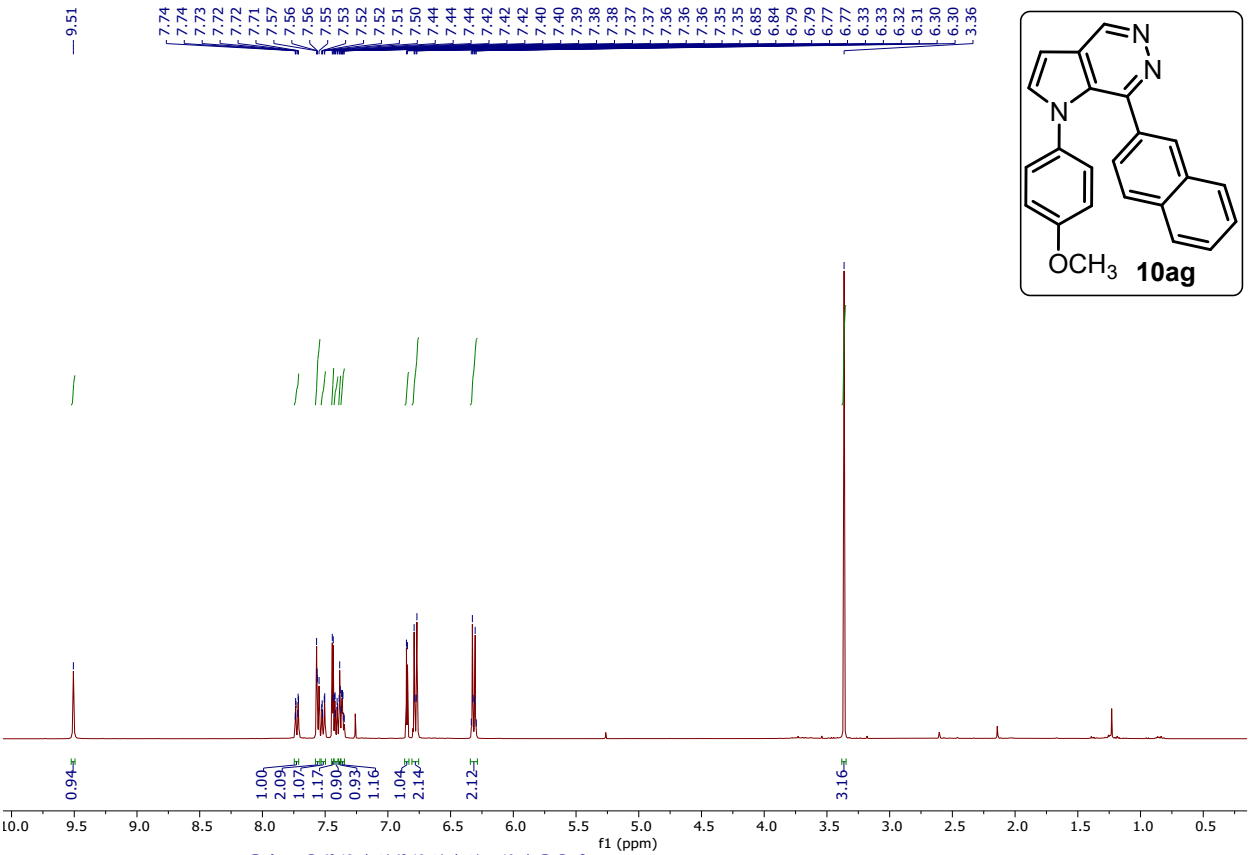
146.5
145.6
141.9
134.3
130.9
130.6
130.2
127.3
126.2
122.4
114.1
102.5
77.0
55.7
f1 (ppm)
N
N
N
NO2
OCH3
10af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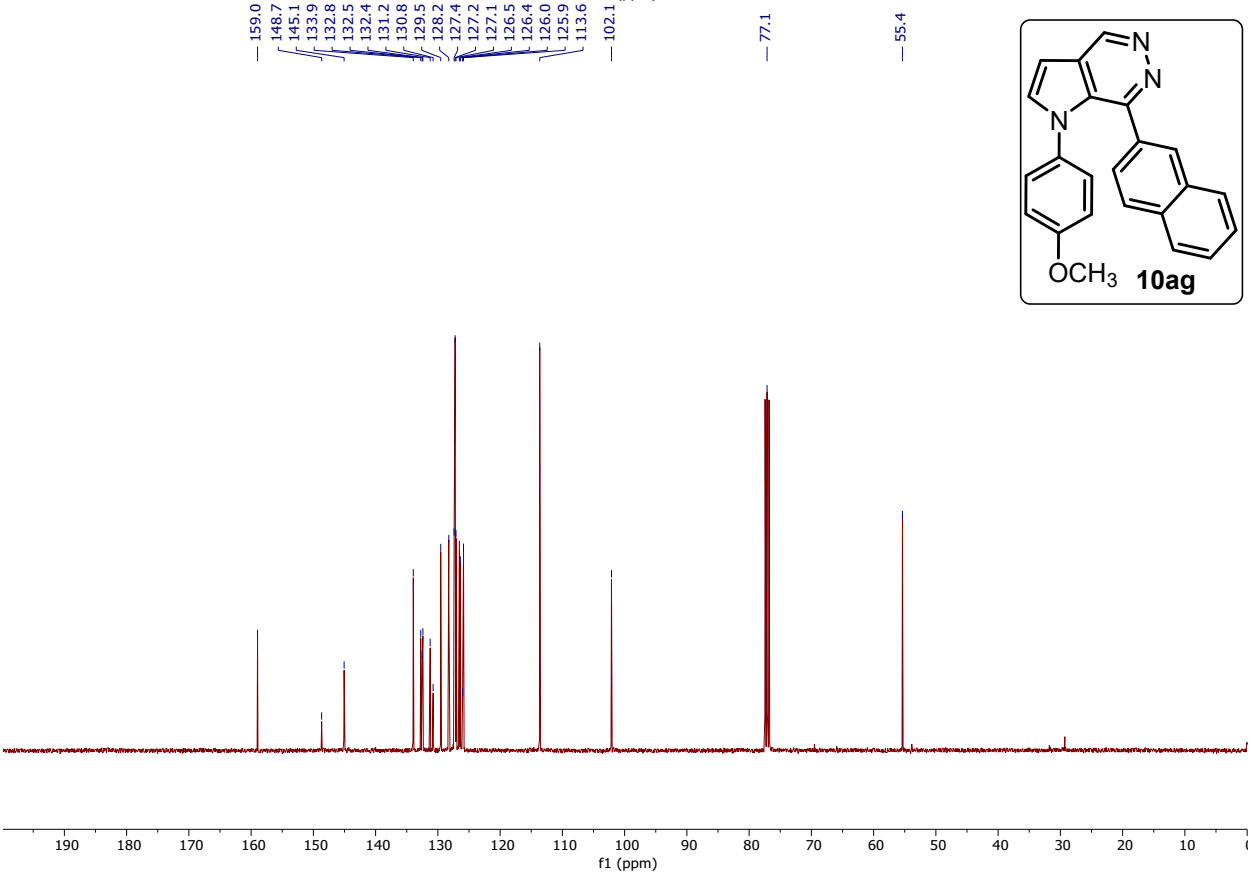
7.56
7.55
7.53
7.52
7.52
7.51
7.50
7.44
7.44
7.44
7.42
7.42
7.40
7.40
7.39
7.38
7.38
7.37
7.37
7.36
7.36
7.36
7.35
7.35
6.85
6.84
6.79
6.79
6.77
6.77
6.33
6.33
6.32
6.31
6.30
6.30
3.36
N
N
N
OCH3
10ag
0.94
1.00
2.09
1.07
1.17
0.90
0.93
1.16
1.04
2.14
2.12
3.16
f1 (ppm)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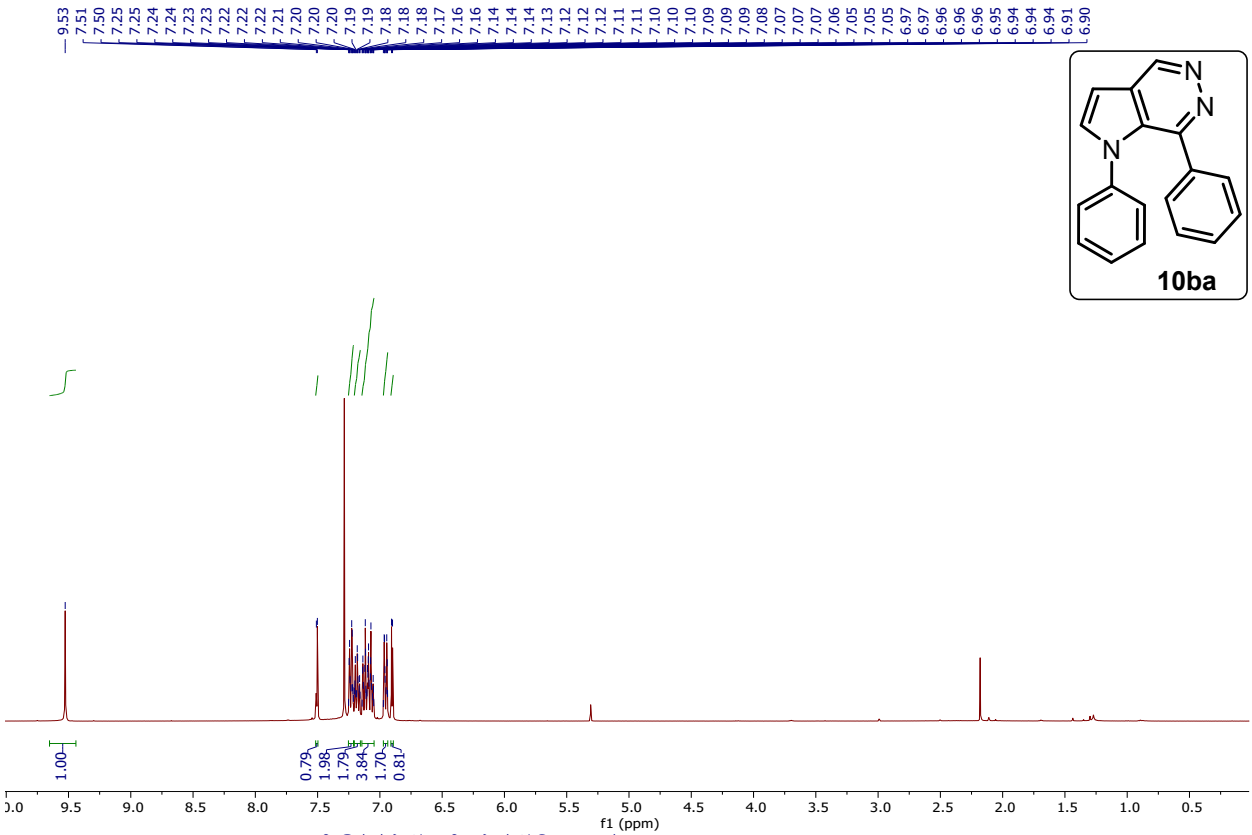
133.9
132.8
132.5
132.4
131.2
130.8
129.5
128.2
127.4
127.2
127.1
126.5
126.4
126.0
125.9
113.6
102.1
77.1
55.4
N
N
N
OCH3
10ag
f1 (ppm)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7.23
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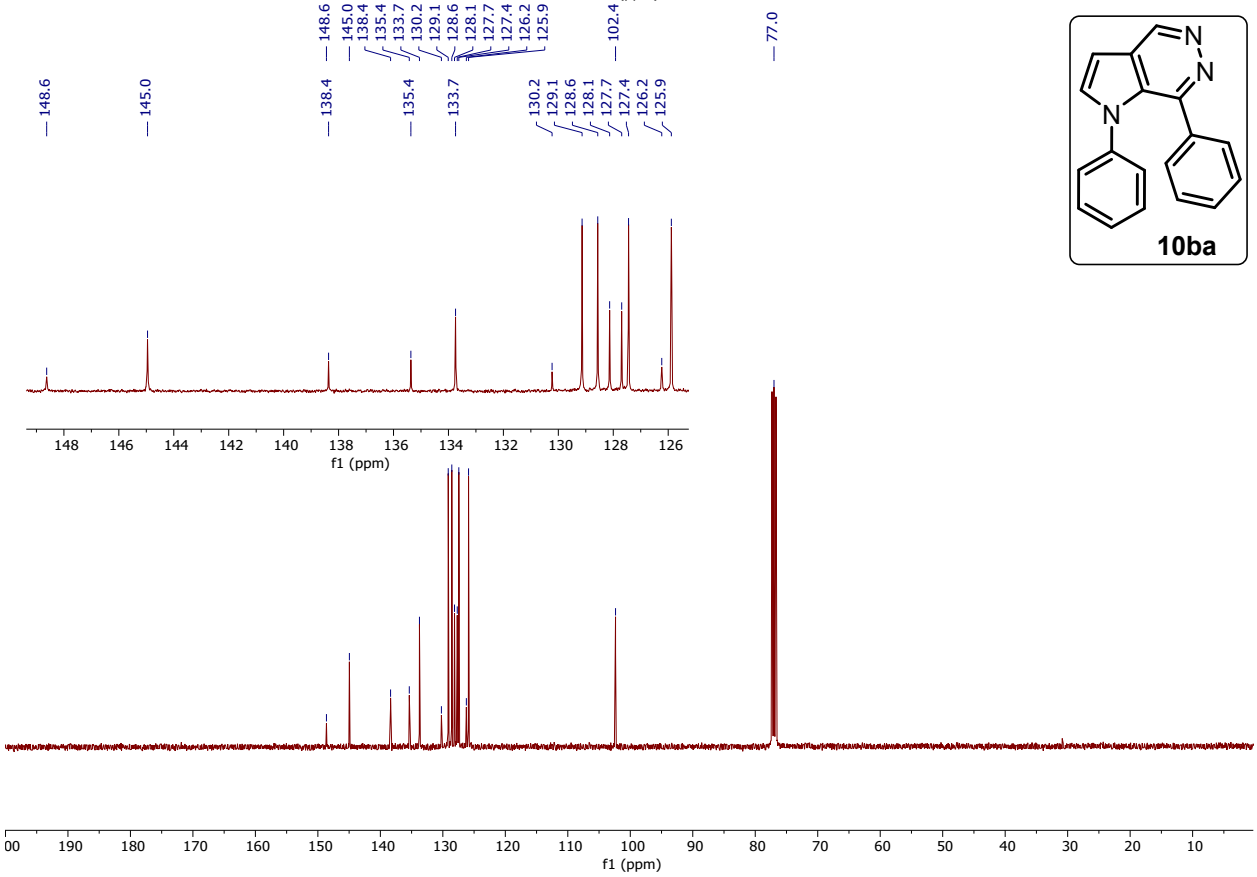
7.22
7.22
7.22
7.21
7.20
7.20
7.20
7.19
7.19
7.18
7.18
7.18
7.17
7.16
7.16
7.14
7.14
7.14
7.13
7.12
7.12
7.12
7.11
7.11
7.10
7.10
7.10
7.09
7.09
7.09
7.08
7.07
7.07
7.07
7.06
7.05
7.05
7.05
6.97
6.97
6.96
6.96
6.96
6.96
6.95
6.94
6.94
6.94
6.91
6.90
N
N
N
10ba
1.00
0.79
1.98
1.79
3.84
1.70
0.81
f1 (ppm)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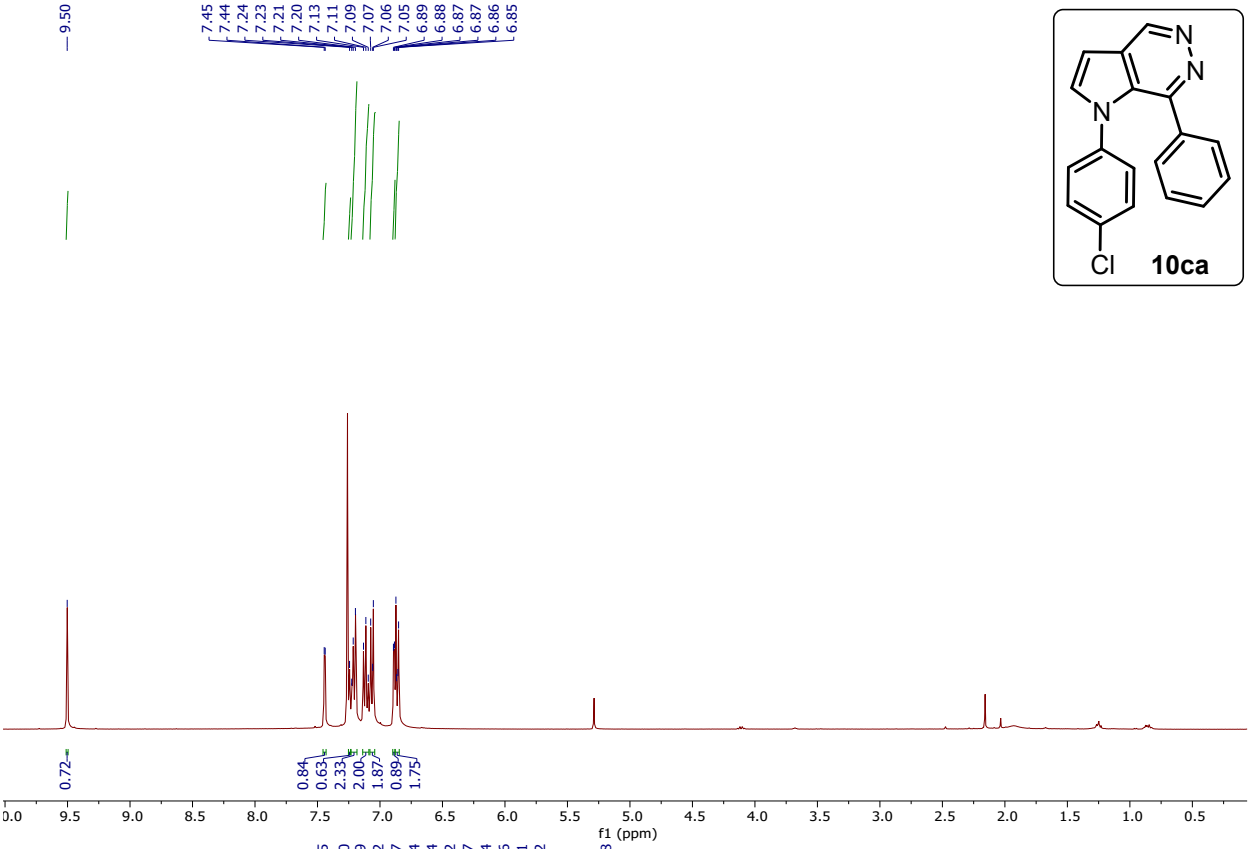
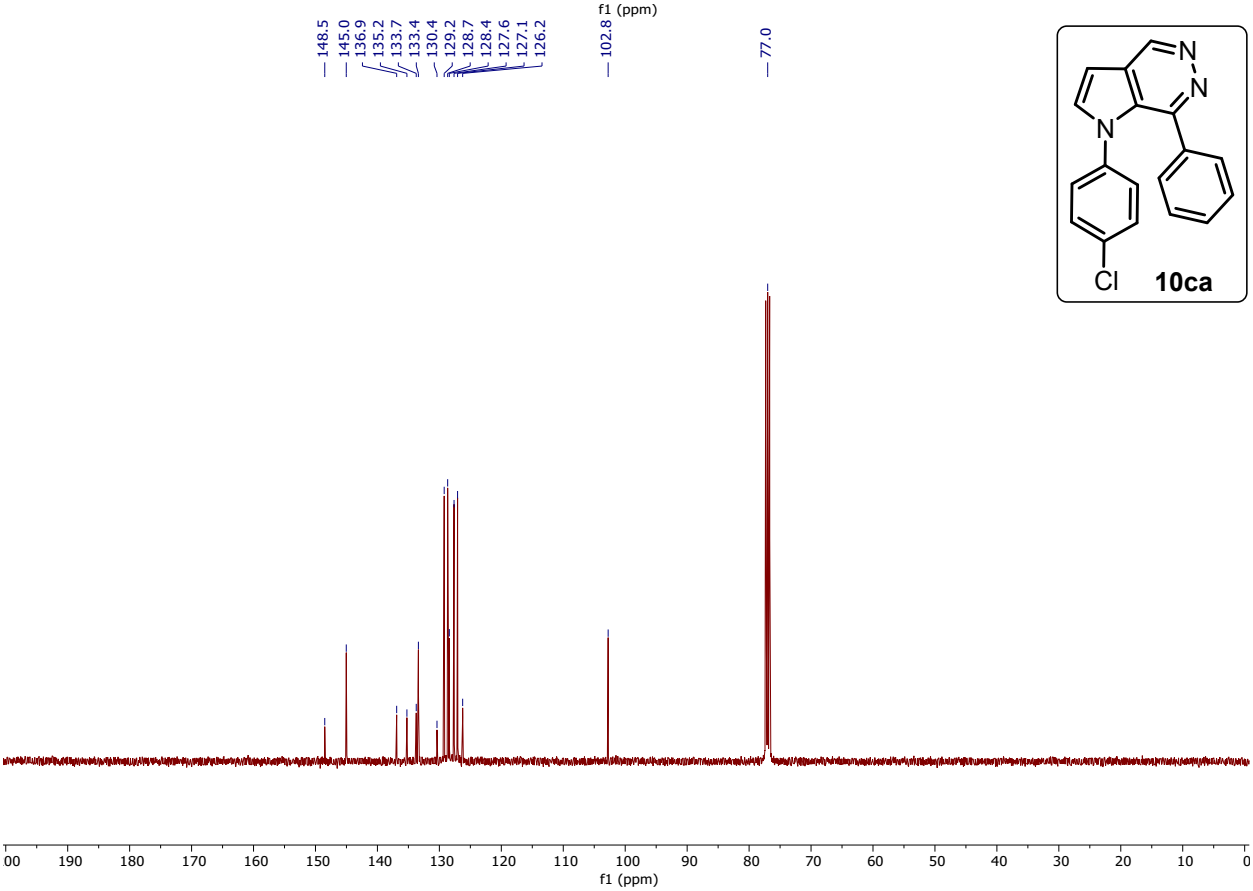
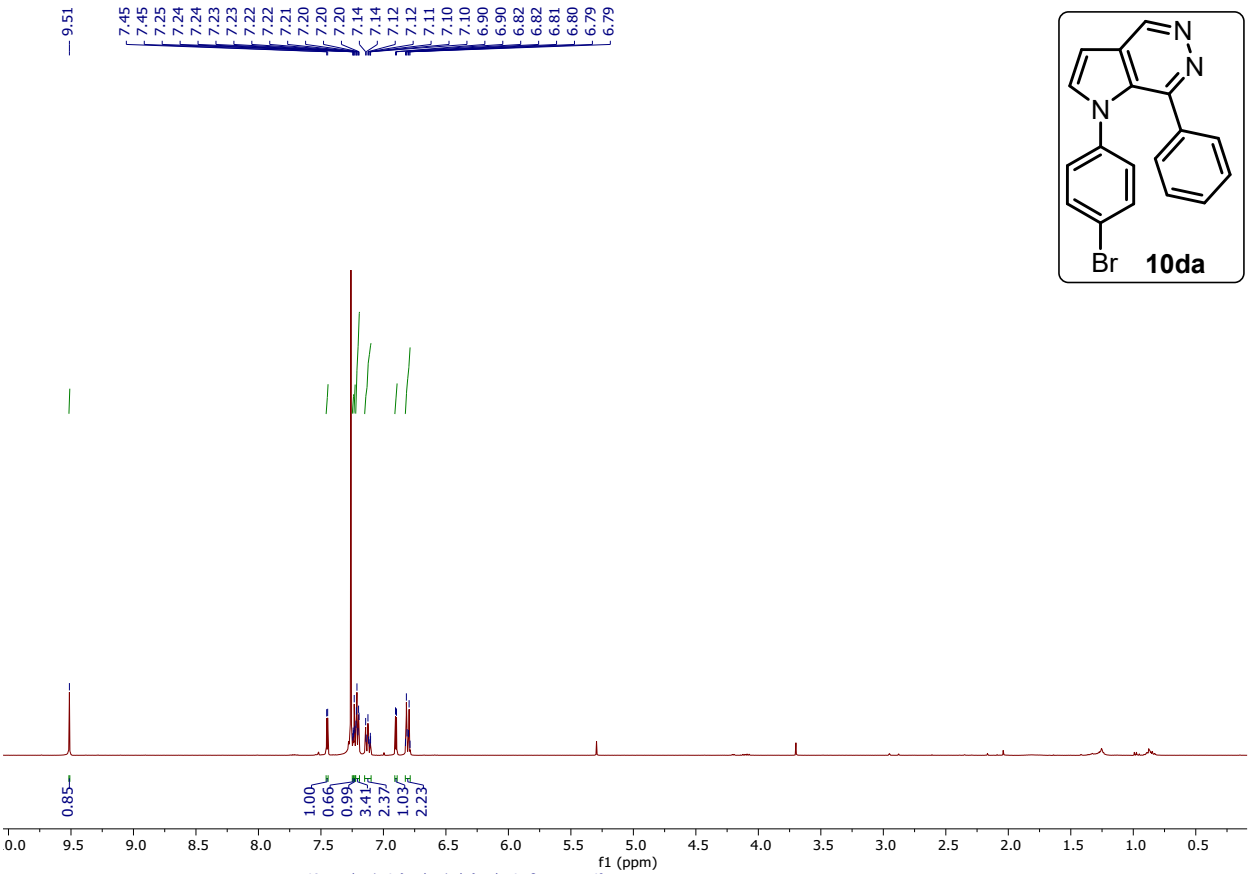
138.4
135.4
133.7
130.2
129.1
128.6
128.1
127.7
127.4
126.2
125.9
102.4
77.0
N
N
N
10ba
148
146
144
142
140
138
136
134
132
130
128
126
f1 (ppm)
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
f1 (ppm)


9.50
7.45
7.44
7.24
7.23
7.21
7.20
7.13
7.11
7.09
7.07
7.06
7.05
6.89
6.88
6.87
6.87
6.86
6.85
N
N
N
Cl
10ca
0.72
0.84
0.63
2.33
2.00
1.87
0.89
1.75
f1 (ppm)


148.5
145.0
136.9
135.2
133.7
133.4
130.4
129.2
128.7
128.4
127.6
127.1
126.2
102.8
77.0
N
N
N
Cl
10ca
f1 (ppm)


9.51
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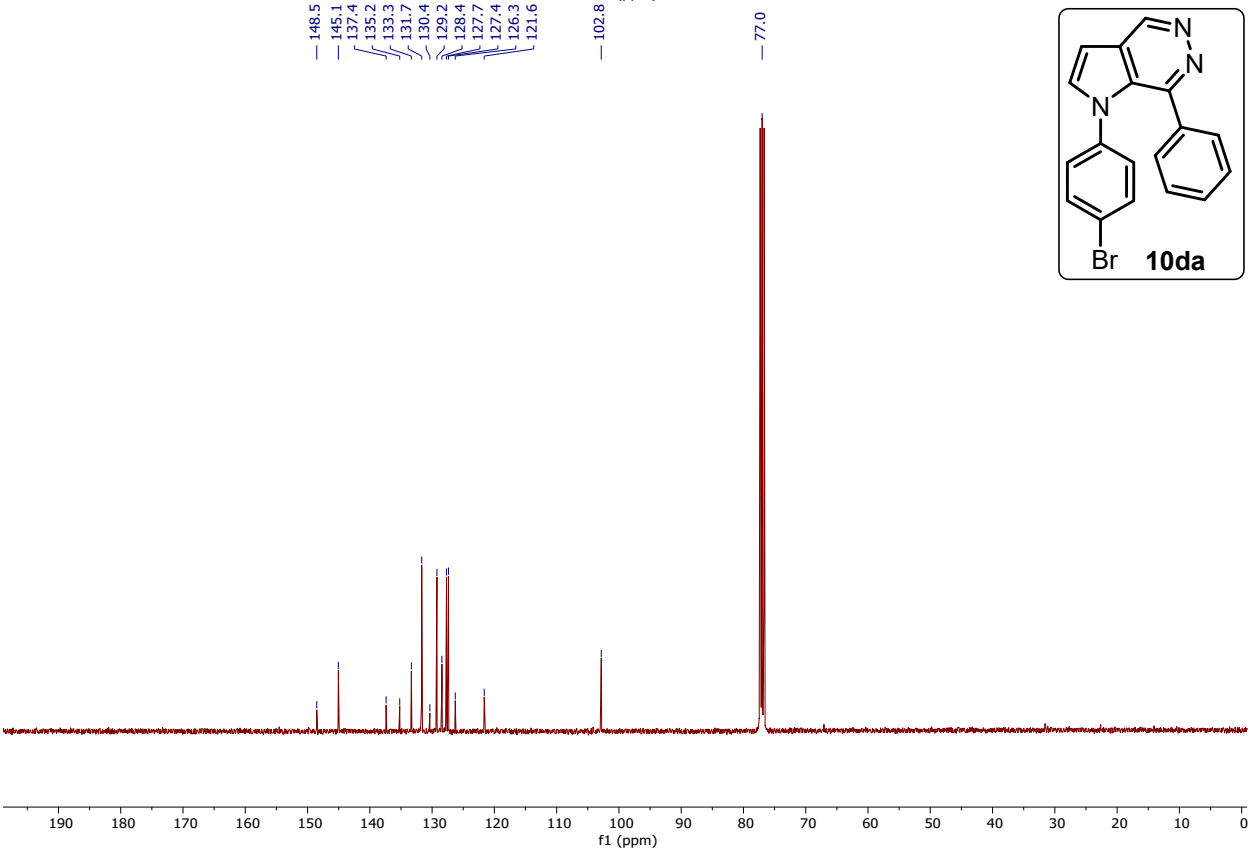
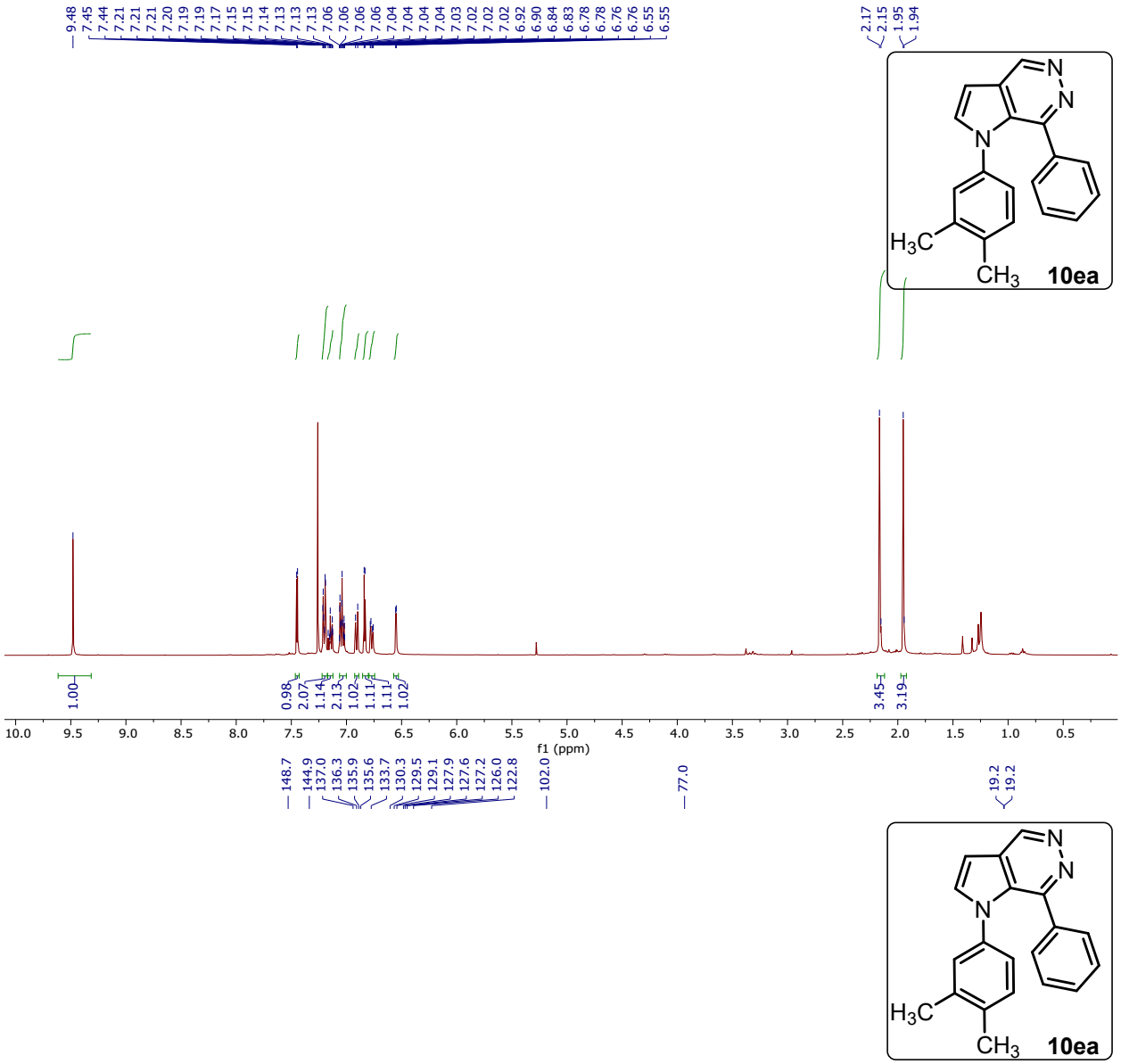
7.45
7.25
7.24
7.24
7.23
7.23
7.22
7.22
7.21
7.20
7.20
7.20
7.14
7.14
7.12
7.12
7.11
7.10
7.10
6.90
6.90
6.82
6.82
6.81
6.80
6.79
6.79
N
N
N
Br
10da
0.85
1.00
0.66
0.99
3.41
2.37
1.03
2.23
f1 (ppm)


148.5
145.1
137.4
135.2
133.3
131.7
130.4
129.2
128.4
127.7
127.4
126.3
121.6
102.8
77.0
N
N
N
Br
10da
f1 (ppm)


9.48
7.45
7.44
7.21
7.21
7.21
7.20
7.19
7.19
7.17
7.15
7.15
7.14
7.13
7.13
7.13
7.06
7.06
7.06
7.06
7.04
7.04
7.04
7.04
7.03
7.02
7.02
7.02
6.92
6.90
6.84
6.83
6.78
6.78
6.76
6.76
6.55
6.55
2.17
2.15
1.95
1.94
N
N
N
H3C
CH3
10ea
1.00
0.98
2.07
1.14
2.13
1.02
1.11
1.11
1.02
3.45
3.19
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
f1 (ppm)
148.7
144.9
137.0
136.3
135.9
135.6
133.7
130.3
129.5
129.1
127.9
127.6
127.2
126.0
122.8
102.0
77.0
19.2
19.2
N
N
N
H3C
CH3
10ea
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
f1 (ppm)

Single Crystal X-Ray Diffraction Experiment and Analysis for Compound 7aj

Single Crystal XRD Experiments for compound 7aj: The title compound, $C_{22}H_{21}N_3O_2$ crystallizes in the Triclinic space group *P-1* with unit cell parameters *a*= 7.1067(8), *b*= 9.6899(10), *c* = 14.5223(12) Å α = 84.346(8), β = 77.804(8), ϒ = 73.711(10) and Z=2. The crystal structure is stabilized by C-H…O inter-molecular hydrogen bonds responsible for the formation of independent layers of chains. The crystal structure is further stabilized by π–π stacking interactions between the Pyrrole and Pyridazine ring of the molecule.

Crystal Structure Determination and Refinement:

Block-shaped crystal selected for intensity data collection was of dimensions 0.30 x 0.20 x 0.10 mm. Accurate cell parameters were determined from 2042 reflections with 3.7< θ <27.31°. X-ray intensity data of 6364 reflections (of which 3685 were unique) were collected on a computer controlled single crystal X-ray diffractometer with graphite mono-chromated Mo *Kα* radiation (λ = 0.71073 Å) in ω scan mode. Data collection was carried out in the range 3.5< θ <26.0°. The number of reflections after applying the limiting criterion I > 2σ(I) converged to 1701 which were considered as observed (-8≤ *h* ≤ 8, -11≤ *k* ≤ 11, -16 ≤ *l* ≤ 17). Data were corrected for Lorentz-polarization and multi-scan absorption corrections.[1] Full-matrix least-squares refinement was carried out using SHELXL97.[2] All the hydrogen atoms were geometrically fixed and allowed to ride on their parent carbon atoms with C-H= 0.97-0.98 Å with $U_{iso}(H) = 1.2U_{eq}(C)$. The final refinement cycles converged to an R = 0.0589 and wR (F^2) = 0.0961 for the observed data. Residual electron densities ranged from -0.174 to 0.038 eÅ^{-3}. The ORTEP diagram as crystal structure of **7aj** with CCDC No. 1455374 is illustrated in Figure S1.[3] The crystallographic data are summarized in Table 1.

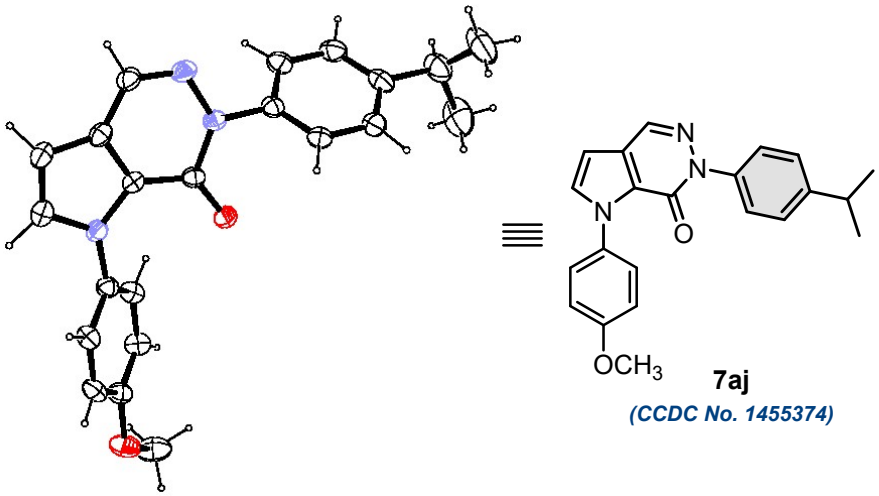


Figure S1: ORTEP plot of the molecule with 40% probability thermal ellipsoids. H-atoms are shown as small spheres of arbitrary radii.

Table S1: Crystal and experimental data

Crystal data	
Crystal description	Block
Crystal colour	White
Crystal size	0.3 x 0.2 x 0.1 mm
Empirical formula	$C_{22}H_{21}N_3O_2$
Formula weight	359.42
Radiation, Wavelength	Mo *Kα*, 0.71073 Å
Crystal system	Triclinic
Space group	*P-1*
Hall symbol	*-P 1*
No. of molecules per unit cell, Z	2
Unit cell dimensions	*a*= 7.1067(8), *b*= 9.6899(10), *c* = 14.5223(12) Å
	α = 84.346(8), β = 77.804(8), ϒ = 73.711(10)

Unit cell volume	937.70(16) $Å^3$
D_x	1.273 g cm^{-3}
Temperature	293 (2) K
Absorption coefficient	0.083 mm^{-1}
F(000)	380
θ range for collection of cell parameters	3.716 <θ<27.3100 º

Data collection

Measurement	X'calibur system–*Oxford diffraction make, U.K.* [Oxford Diffraction, 2010]
Structure determination	Direct methods
Range of indices	*h*=-8 to 8, *k*= -11 to 11, *l*= -16 to 17
Reflections collected / unique	6364 /3685
Reflections observed (I > 2□(I))	1701
R_{int}	0.042
R_{sigma}	0.1152
Scan mode	ω scan
θ_{max}	26.00°
θ_{min}	3.54°
T_{min}, T_{max}	0.39569, 1.0000
Absorption correction	multi-scan [*CrysAlis RED*; Oxford Diffraction, 2010]

Refinement

Refinement	Full-matrix least squares on F^2

No. of parameters refined	248
Final *R*	0 .0589
w*R*(F^2)	0.0961
Weight	$w=1/[\sigma^2(F_o^2)+(0.0254P)^2+0.00P]$
	where $P=[F_o^2+2F_c^2]/3$.
Goodness-of-fit	0.958
$(\Delta /\sigma)_{max}$	0.001
Final residual electron density	$-0.174 < \Delta\rho < 0.038$ e $Å^{-3}$
Software for structure solution	SHELXS97 [Sheldrick, 2008]
Software for refinement	SHELXL97 [Sheldrick, 2008]
Software for molecular plotting	ORTEP-3[Farrugia,2012]; PLATON [Spek,2009]
Software for geometrical calculation	PLATON [Spek, 2009]; PARST [Nardelli, 1995]

References:

1. G. M. Sheldrick, *Acta Cryst.*, **2008**, *A64*, 112.
2. L. J. Farrugia, *J. Appl. Cryst.*, **2012**, *45*, 849.
3. L. Spek, *Acta Cryst.*, **2009**, *D65*, 148.

Single Crystal X-Ray Diffraction Experiment and Analysis for Compound 10ba

Single Crystal XRD Experiments for compound 10ba (exp 1209_MK-206): The single crystal XRD data collection and data reduction were performed using CrysAlis PRO on a single crystal Rigaku Oxford XtaLab Pro Kappa dual home/near diffractometer. The crystals were kept at 133(2) K during data collection using CuKα (λ = 1.54184 Å) radiation. Using Olex2[1], the structure was solved with the ShelXT[2] structure solution program using Intrinsic Phasing and refined with the ShelXL[3] refinement package using Least Squares minimization.

Single Crystal structure, Cell parameters and structure data of compound 10ba (exp 1209_MK-206):

The single crystal of compound **10ba** ($C_{18}H_{13}N_3$) **(exp 1209_MK-206)**) was crystalized as a colorless block through the slow evaporation of chloroform solution at room temperature. The compound **10ba** crystallized in monoclinic crystal system with $P2_1/c$ space group. Two independent molecules with slightly different bond parameters appeared in the structure solution in an asymmetric unit (Z'=2) with the following crystal unit cell data.

Crystal Data for **10ba** ($C_{18}H_{13}N_3$)(M =271.31 g/mol): monoclinic, space group $P2_1/c$ (no. 14), a = 17.6435(6) Å, b = 9.6496(4) Å, c = 15.5396(4) Å, β = 91.290(3)°, V = 2644.99 (16) Å^3, Z = 8, T = 133(2) K, μ(Cu Kα) = 0.649 mm^{-1}, *Dcalc* = 1.363 g/cm^3, 16652 reflections measured (10.03° ≤ 2Θ ≤ 165.9°), 5587 unique (R_{int} = 0.0370, R_{sigma} = 0.0418) which were used in all calculations. The final R_1 was 0.0471 (I > 2σ(I)) and wR_2 was 0.1343 (all data). The crystallographic details of the compound **10ba** are deposited to the Cambridge Crystallographic (CCDC 2254911). The crystal data and structure refinement for the compound **10ba** is shown in Table S1. The ORTEP diagram as crystal structure of **10ba [exp 1209_MK-206]** is illustrated in Figure S1.

Table 1 Crystal data and structure refinement for 10ba (exp_1209_MK-206_20221017).

Identification code	exp_1209_MK-206_20221017
Empirical formula	$C_{18}H_{13}N_3$
Formula weight	271.31
Temperature/K	133(2)
Crystal system	monoclinic
Space group	$P2_1/c$
a/Å	17.6435(6)
b/Å	9.6496(4)
c/Å	15.5396(4)
α/°	90

β/°	91.290(3)
γ/°	90
Volume/Å^3	2644.99(16)
Z	8
ρ_{calc}g/cm^3	1.363
μ/mm^{-1}	0.649
F(000)	1136.0
Crystal size/mm^3	0.11 × 0.08 × 0.06
Radiation	Cu Kα (λ = 1.54184)
2Θ range for data collection/°	10.03 to 165.9
Index ranges	-21 ≤ h ≤ 14, -11 ≤ k ≤ 11, -19 ≤ l ≤ 17
Reflections collected	16652
Independent reflections	5587 [R_{int} = 0.0370, R_{sigma} = 0.0418]
Data/restraints/parameters	5587/0/379
Goodness-of-fit on F^2	1.091
Final R indexes [I>=2σ (I)]	R_1 = 0.0471, wR_2 = 0.1244
Final R indexes [all data]	R_1 = 0.0558, wR_2 = 0.1343
Largest diff. peak/hole / e Å^{-3}	0.33/-0.26

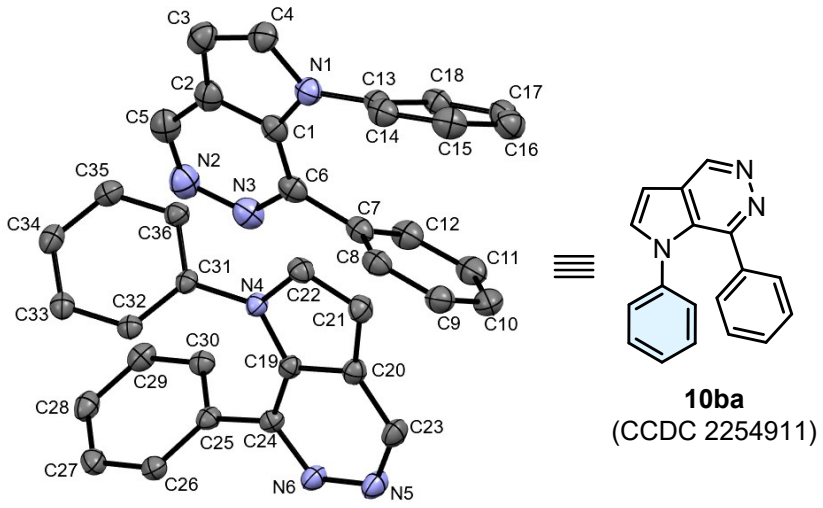


Figure S1: The ORTEP diagram of compound **10ba** (**exp 1209_MK-206**) (CCDC 2254911). Two molecules appeared in an asymmetric unit; hydrogen atoms are not shown for clarity. The thermal ellipsoid is drawn at the 50 % probability level.

Refinement model description.

Number of restraints - 0, number of constraints - unknown.

Details:

```
1. Fixed Uiso
 At 1.2 times of:
  All C(H) groups
2.a Aromatic/amide H refined with riding coordinates:
```

```
C3(H3), C4(H4), C5(H5), C8(H8), C9(H9), C10(H10), C11(H11), C12(H12),
C14(H14), C15(H15), C16(H16), C17(H17), C18(H18), C21(H21), C22(H22), C23(H23),
 C26(H26), C27(H27), C28(H28), C29(H29), C30(H30), C32(H32), C33(H33),
C34(H34), C35(H35), C36(H36)
```

This report has been created with Olex2, compiled on 2022.04.07 svn.rca3783a0 for OlexSys.

References:

1. Dolomanov, O.V., Bourhis, L.J., Gildea, R.J, Howard, J.A.K. & Puschmann, H., *J. Appl. Cryst*. **2009,** 42, 339-341.
2. Sheldrick, G.M. Acta Cryst. **2015**, A71, 3-8.
3. Sheldrick, G.M. Acta Cryst. **2015**, C71, 3-8.