Supporting Information

Construction of 2,3,4,5-tetrahydro-1,2,4-triazines via [4+2] cycloaddition of α-halogeno hydrazones with imines

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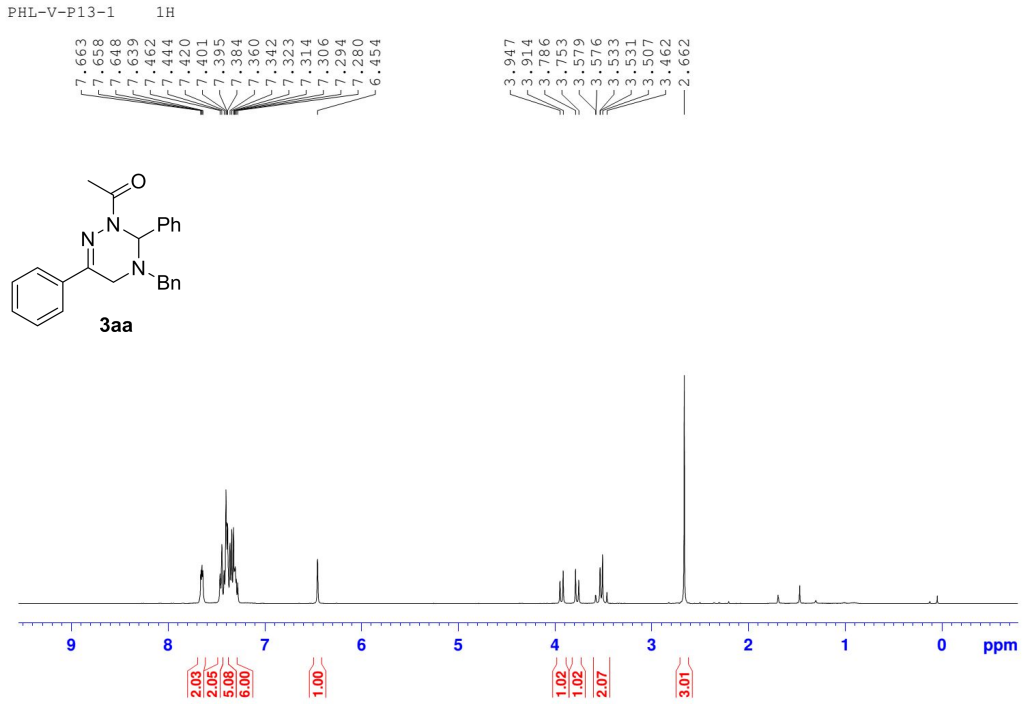
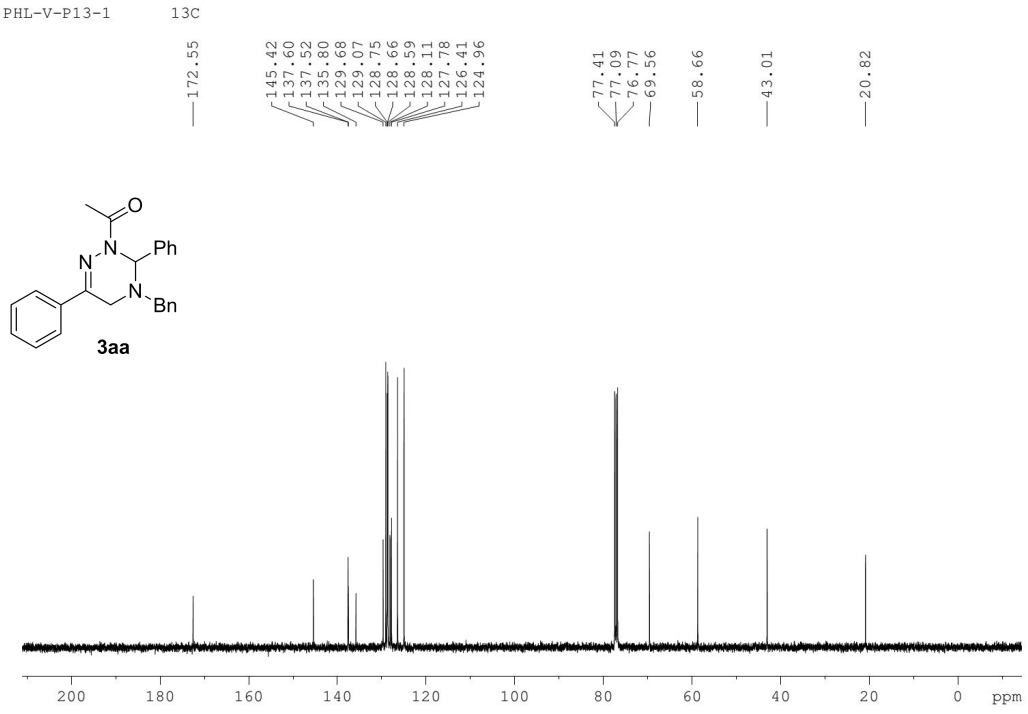
Table of Contents

1. ^{1}H and ^{13}C NMR spectra





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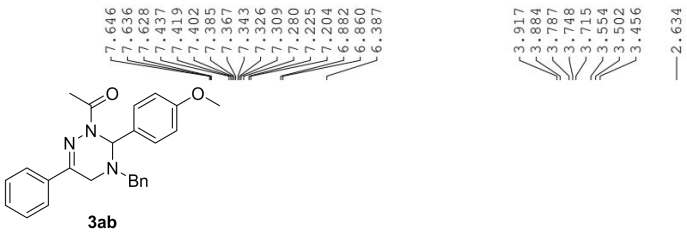

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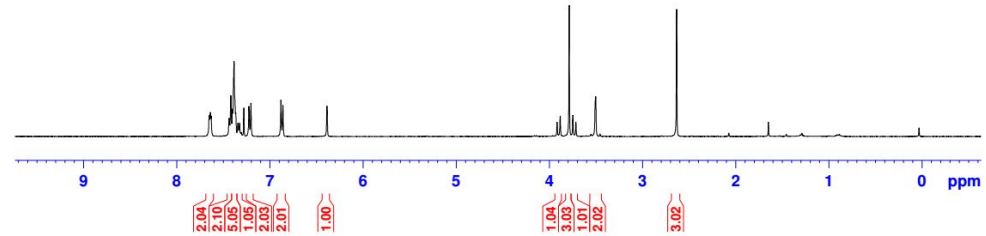

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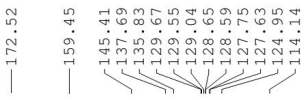

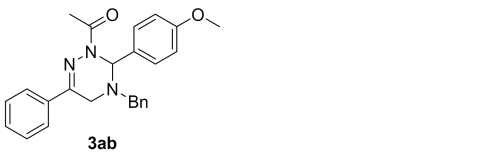

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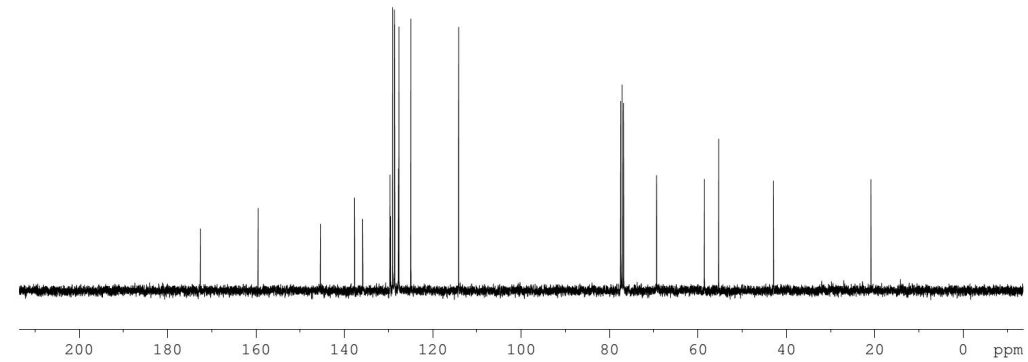
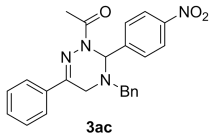


PHL-V-P17-5 NEW 1H

8.217
8.195
7.643
7.629
7.623
7.493
7.472
7.423
7.416
7.409
7.391
7.378
7.371
7.357
7.280
6.452

3.933
3.900
3.812
3.780
3.639
3.592
3.439
3.393

2.663



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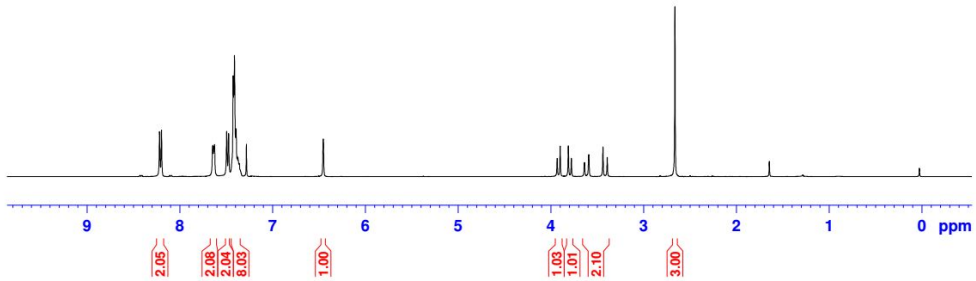

PHL-V-P17-5 13C

172.67

147.88
145.66
144.94
136.94
135.24
130.05
129.10
128.81
128.69
128.10
127.66
124.94
124.01

77.40
77.08
76.76
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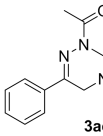
58.85
43.17
20.75



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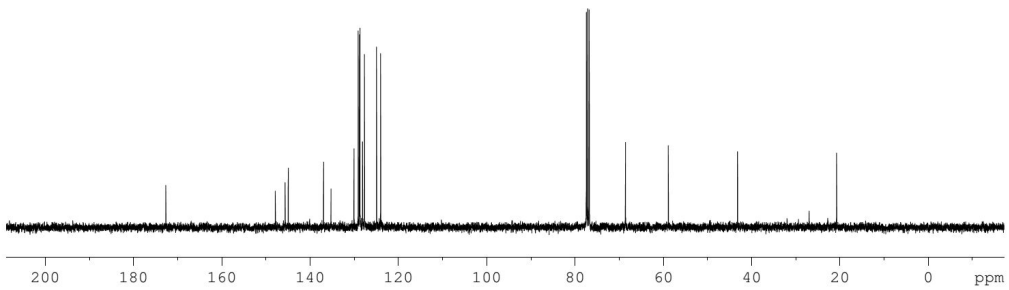

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7.638
7.630
7.606
7.554
7.534
7.514
7.449
7.432
7.410
7.396
7.380
7.375
7.358
7.280
6.462

3.950
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3.821
3.788
3.649
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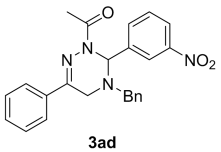
3.452
3.406

2.676



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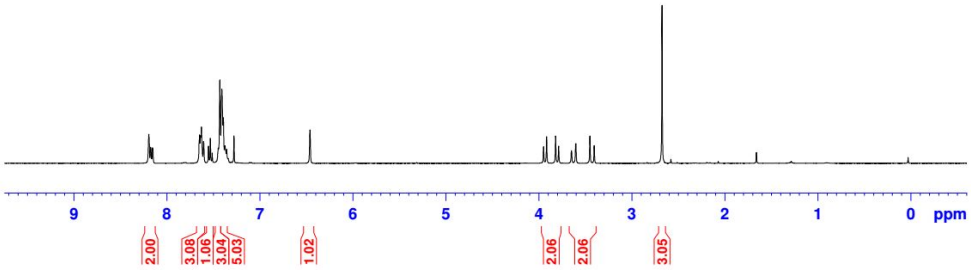

PHL-V-P17-6 13C

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145.70
140.11
136.92
135.28
132.71
130.04
129.86
129.15
128.83
128.69
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123.31
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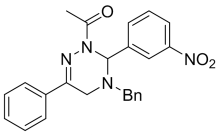
77.11
76.79

68.40

58.82

43.00

20.78



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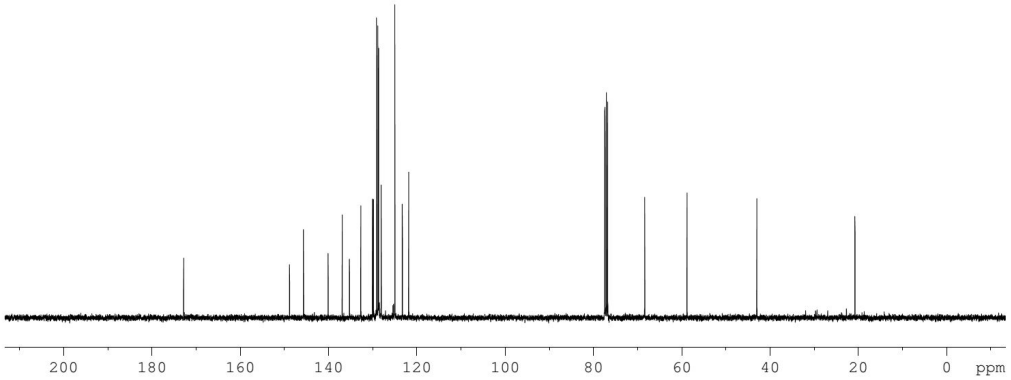

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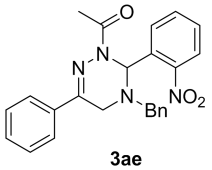

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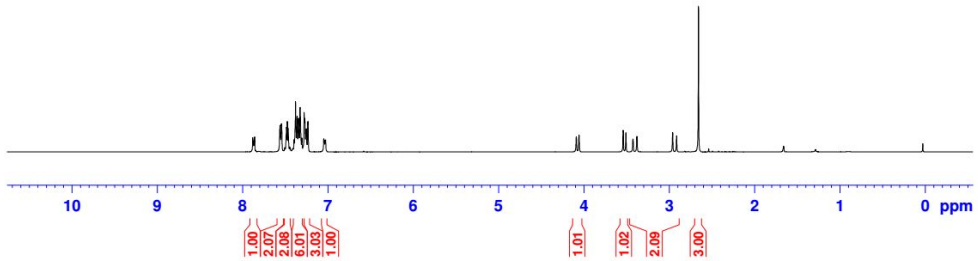

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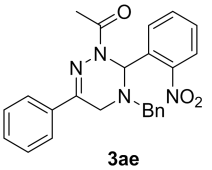

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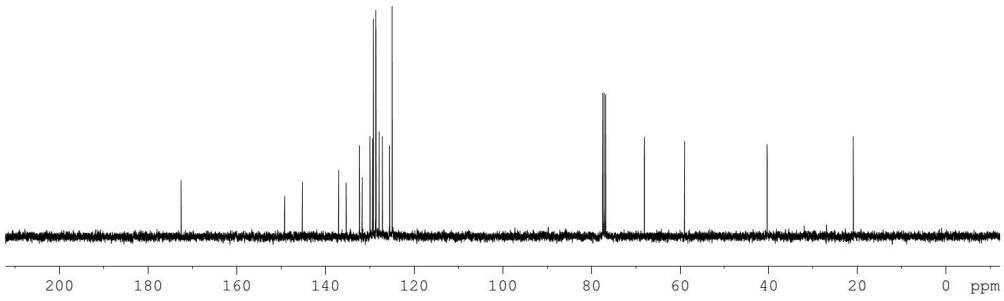

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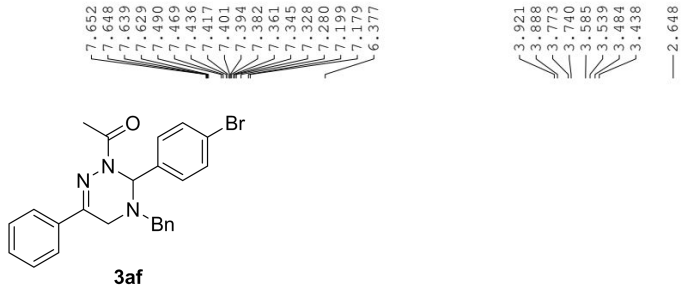

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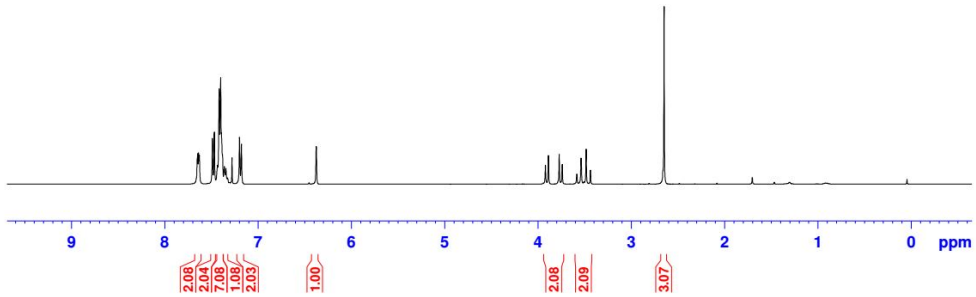

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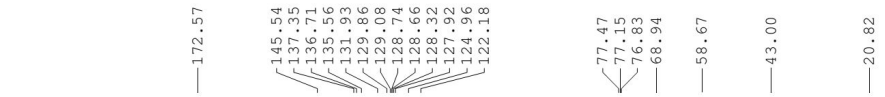

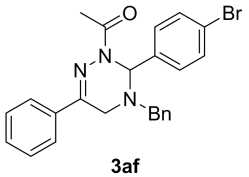

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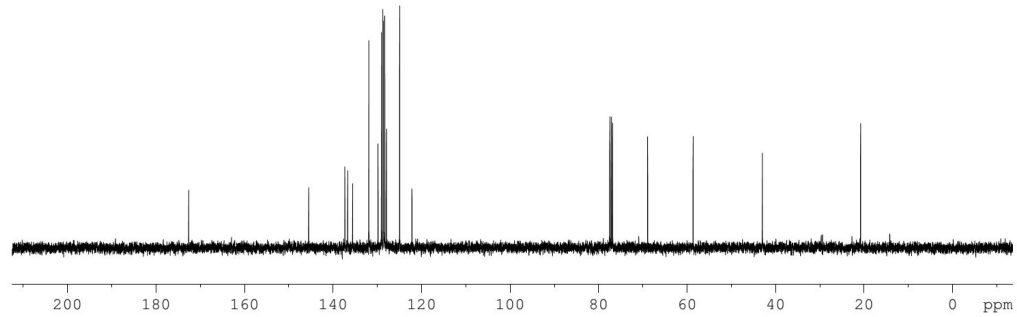

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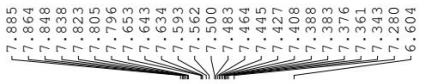

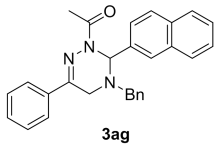

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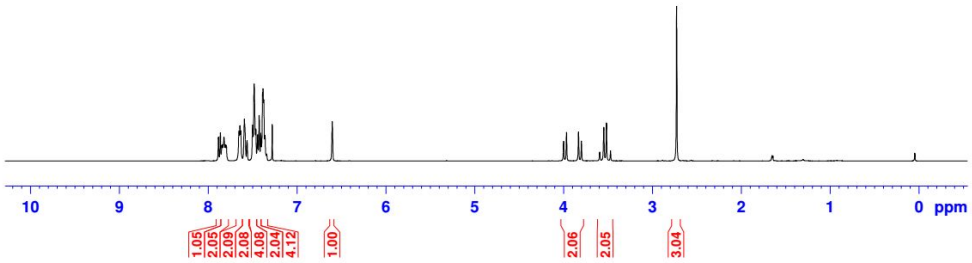

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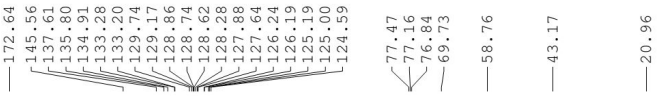

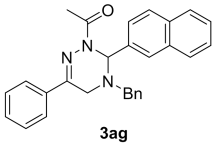

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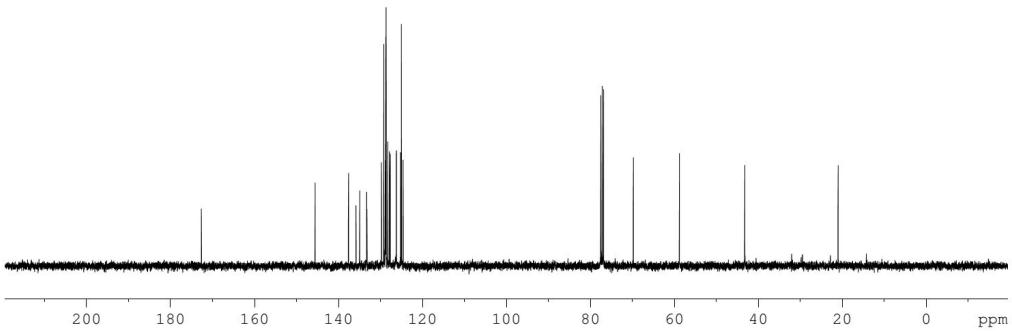

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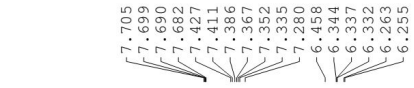

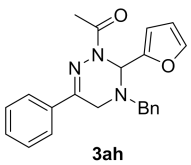

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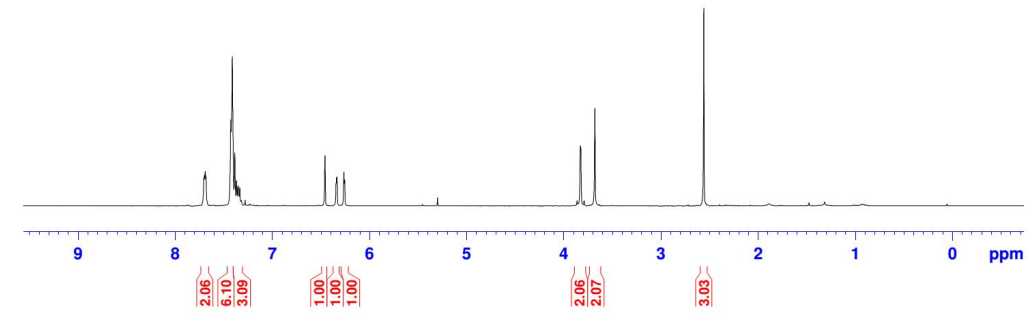

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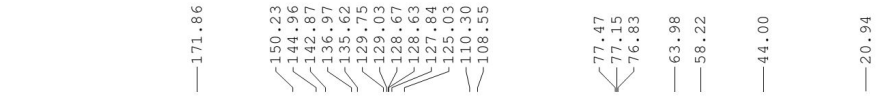

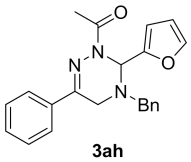

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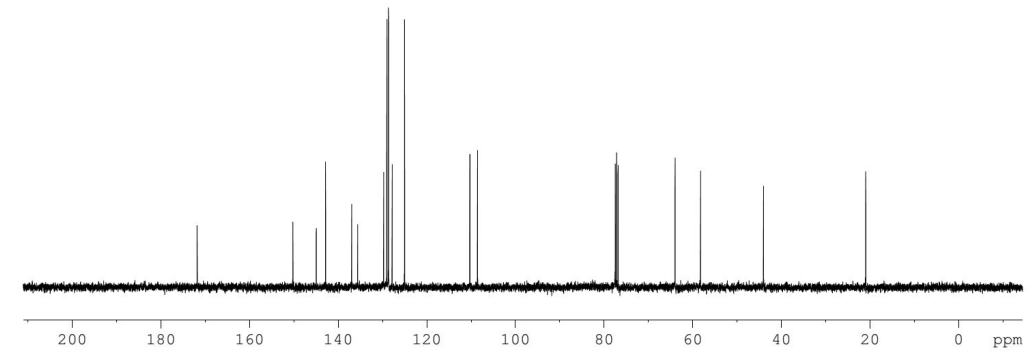

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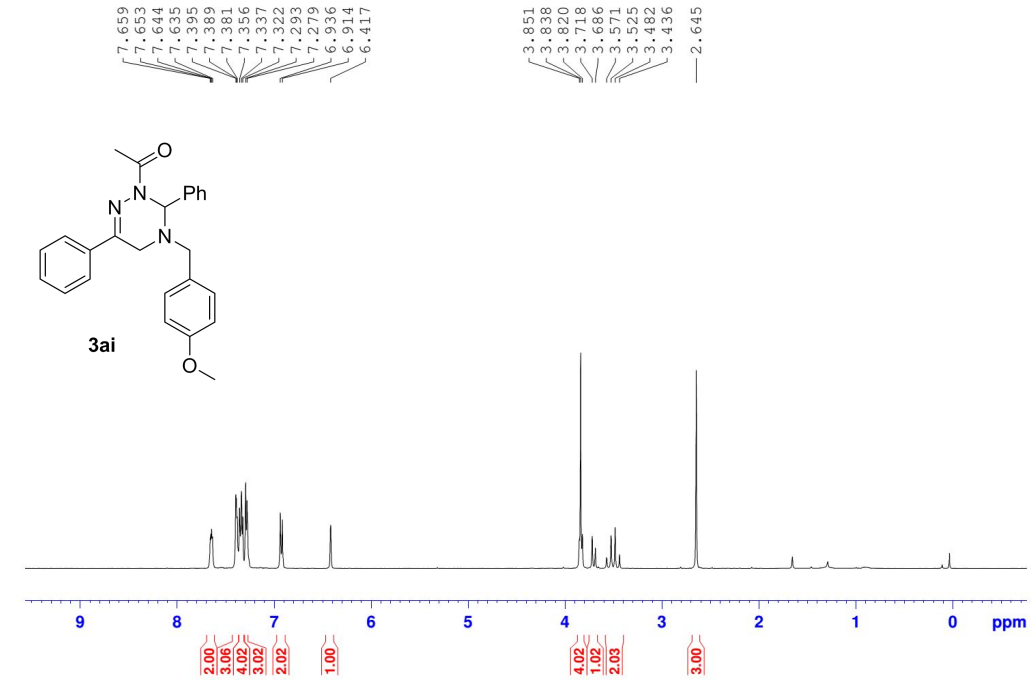

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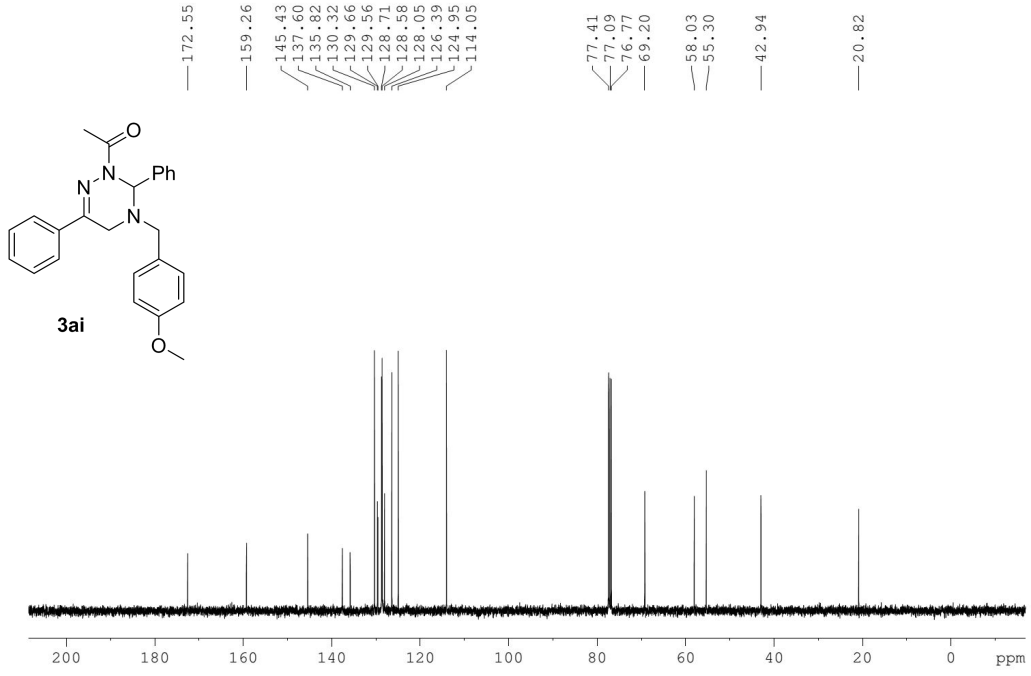

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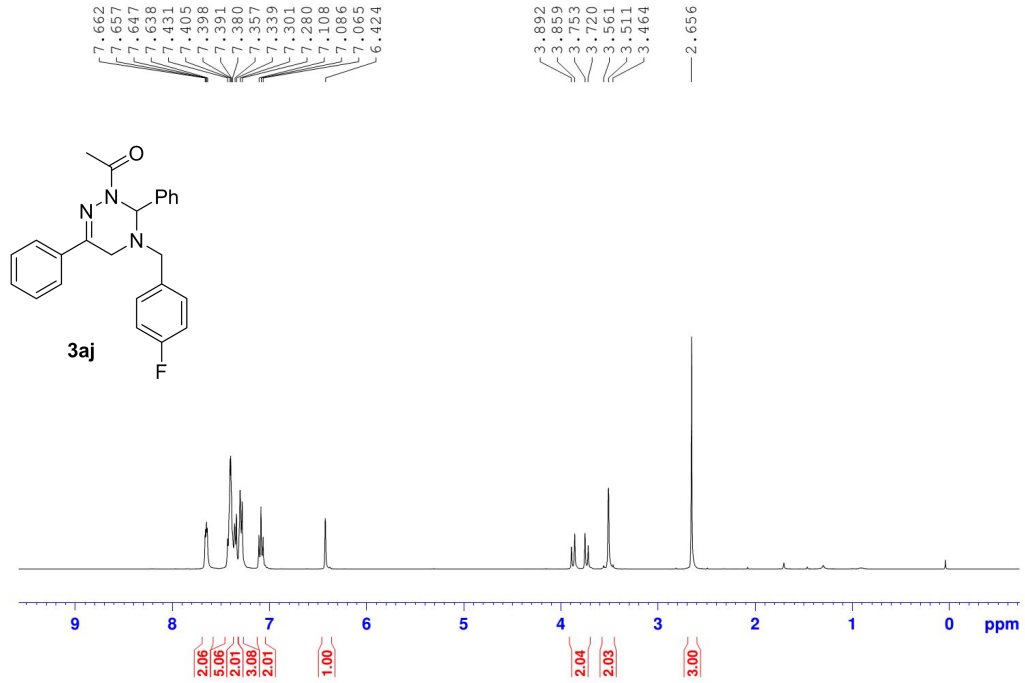

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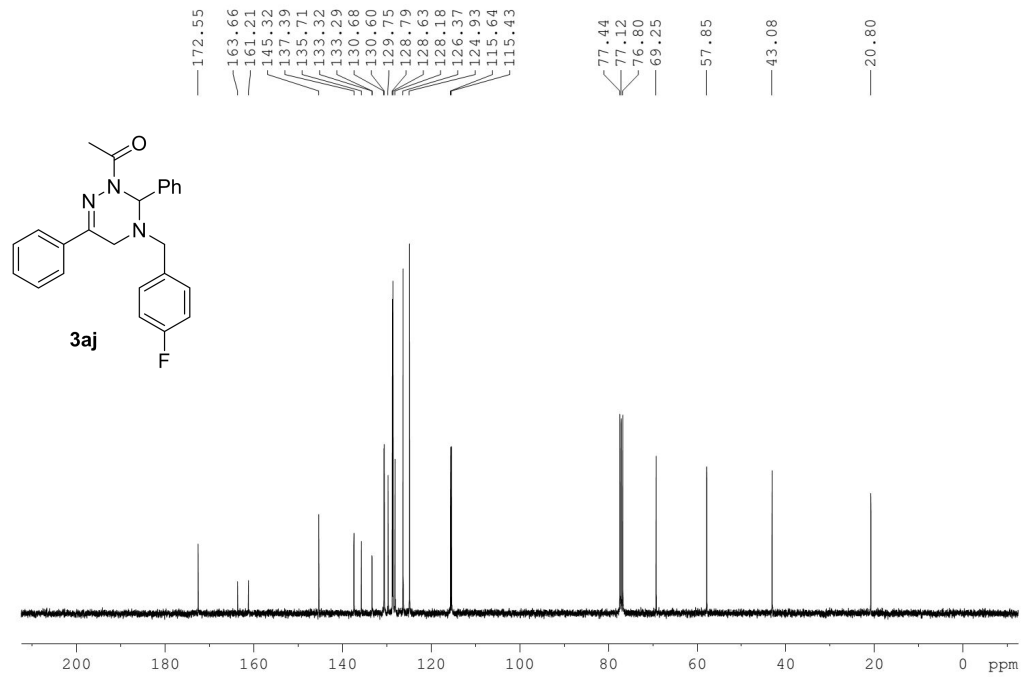

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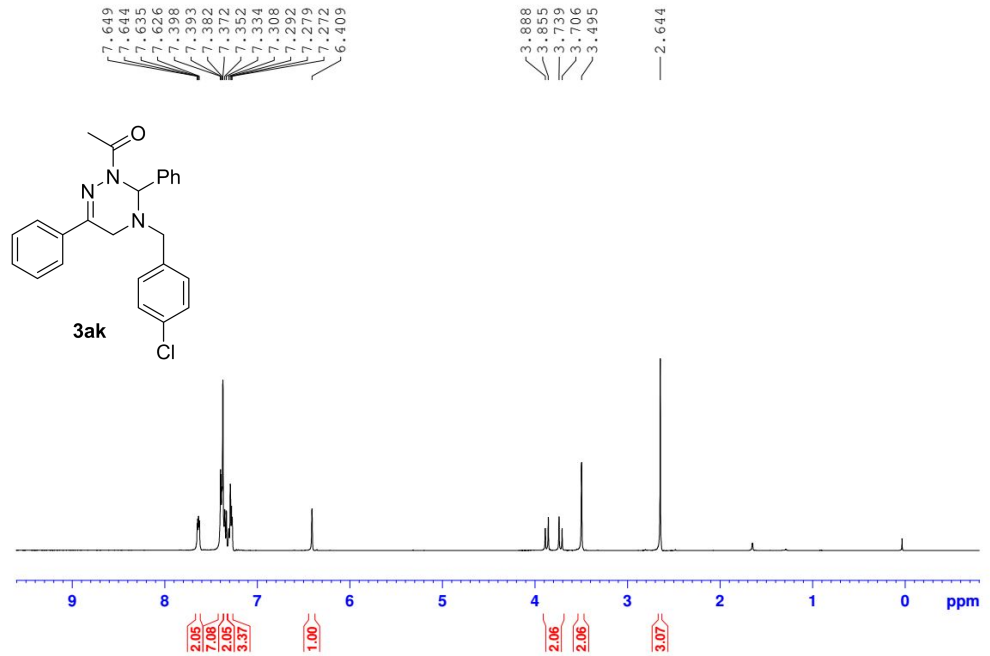

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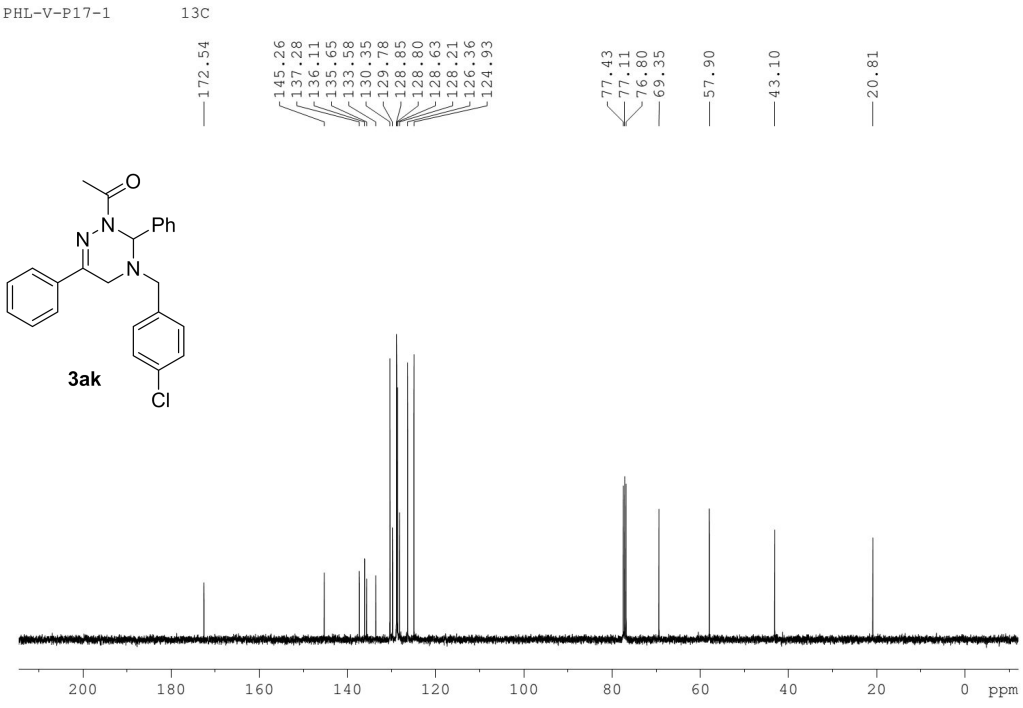

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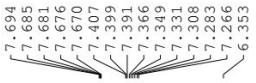

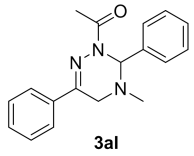

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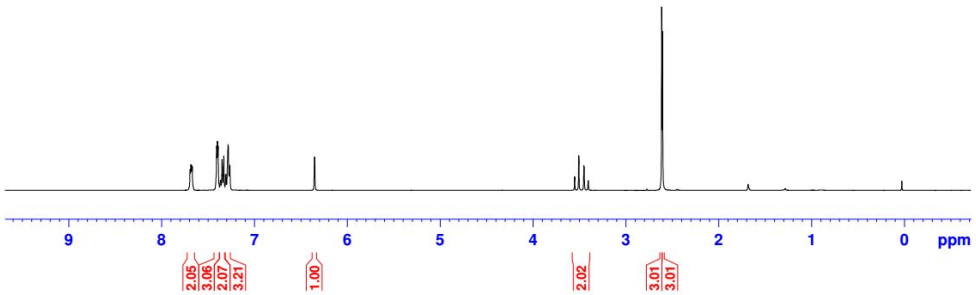

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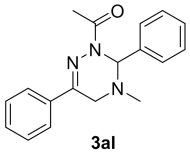

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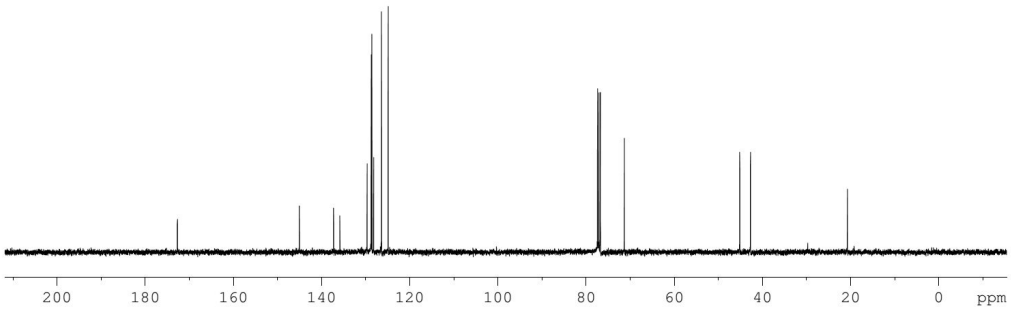

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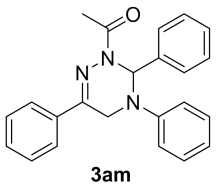

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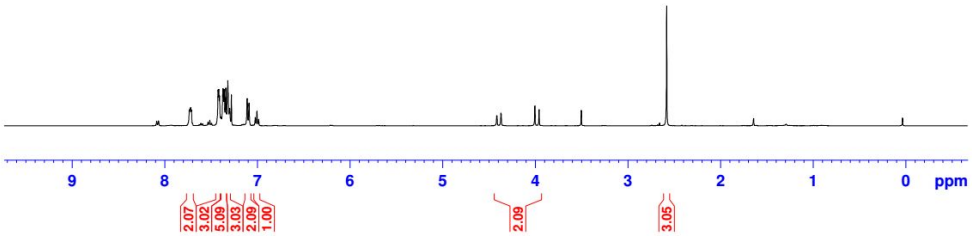

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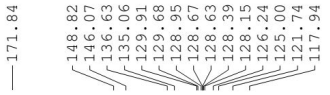

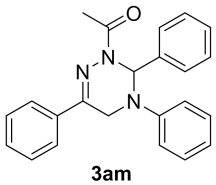

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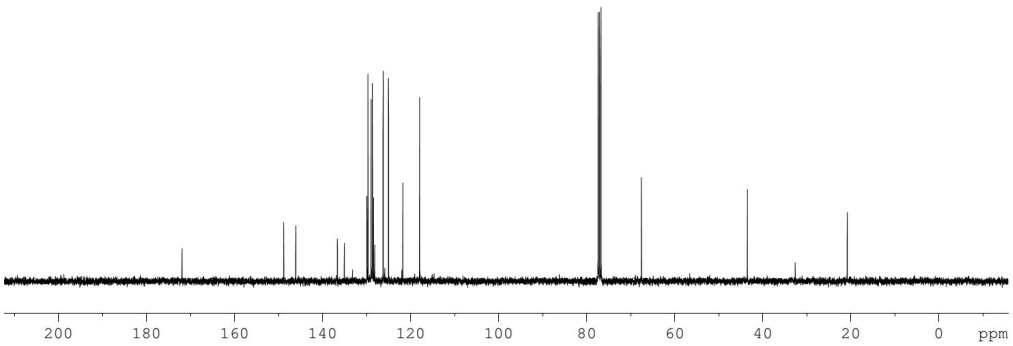

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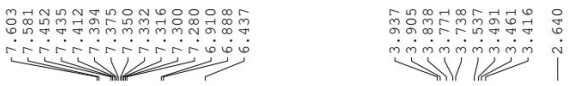
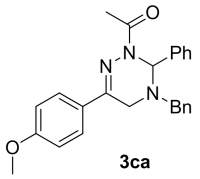
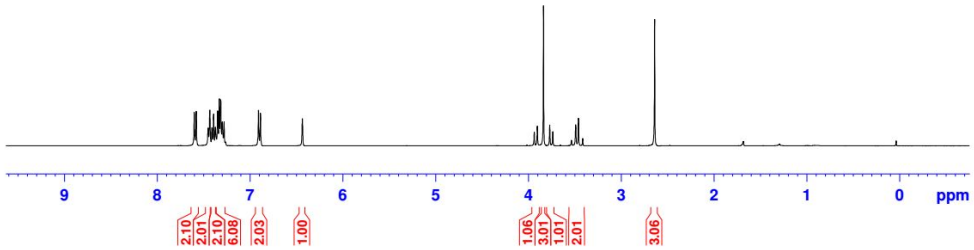
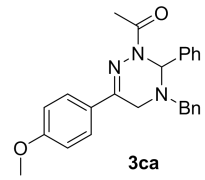




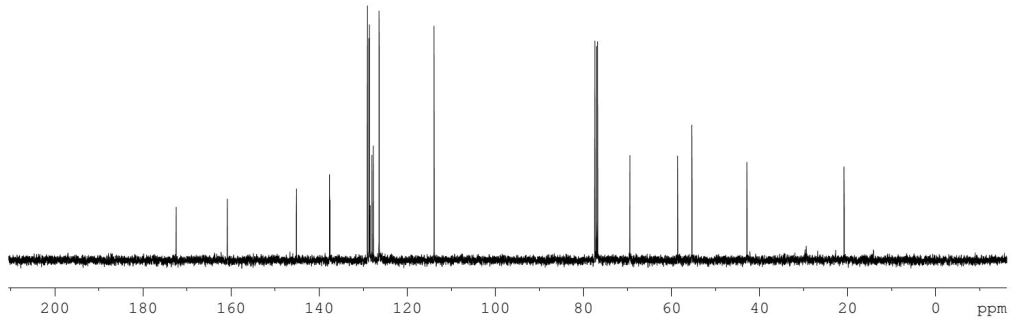
3ca



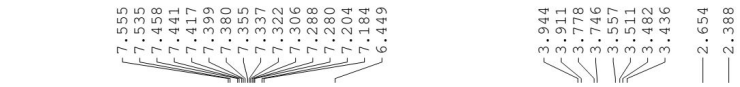
PHL-V-P13-2 13C

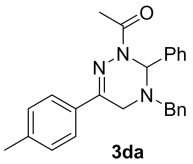


3ca

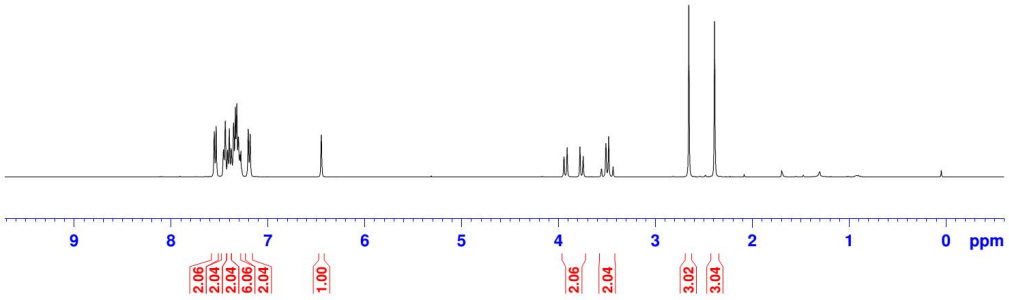


PHL-V-P13-3 NEW 1H

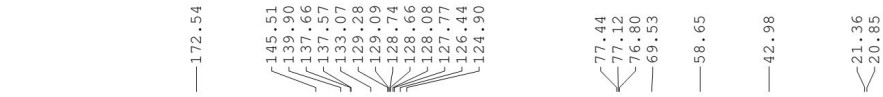


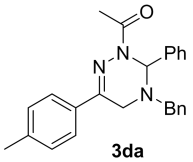


3da

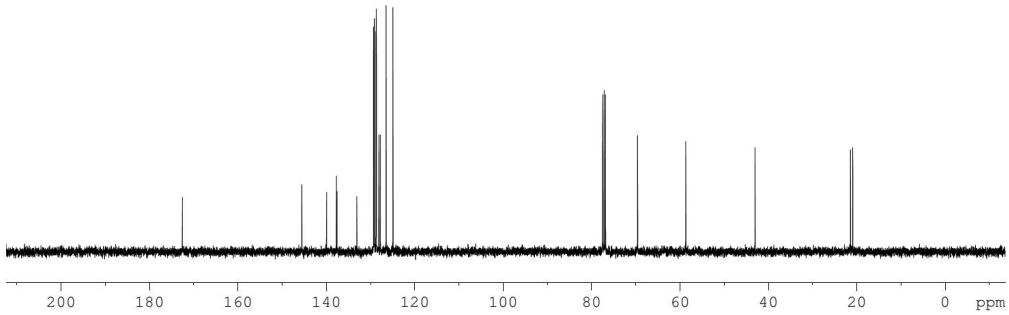


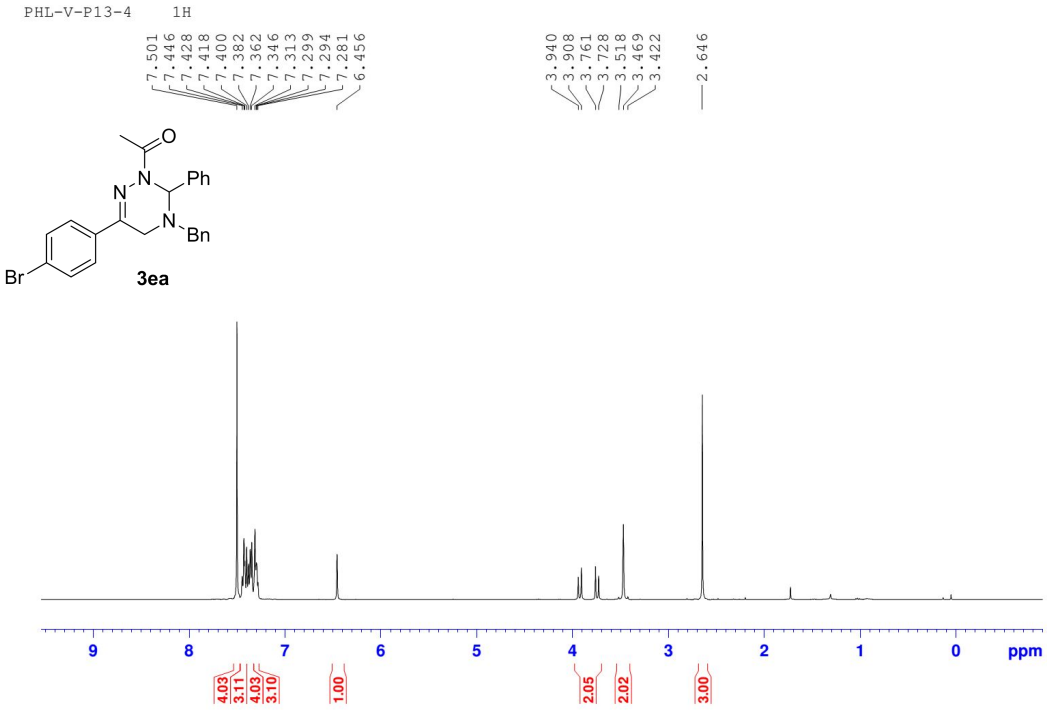
PHL-V-P13-3 13C

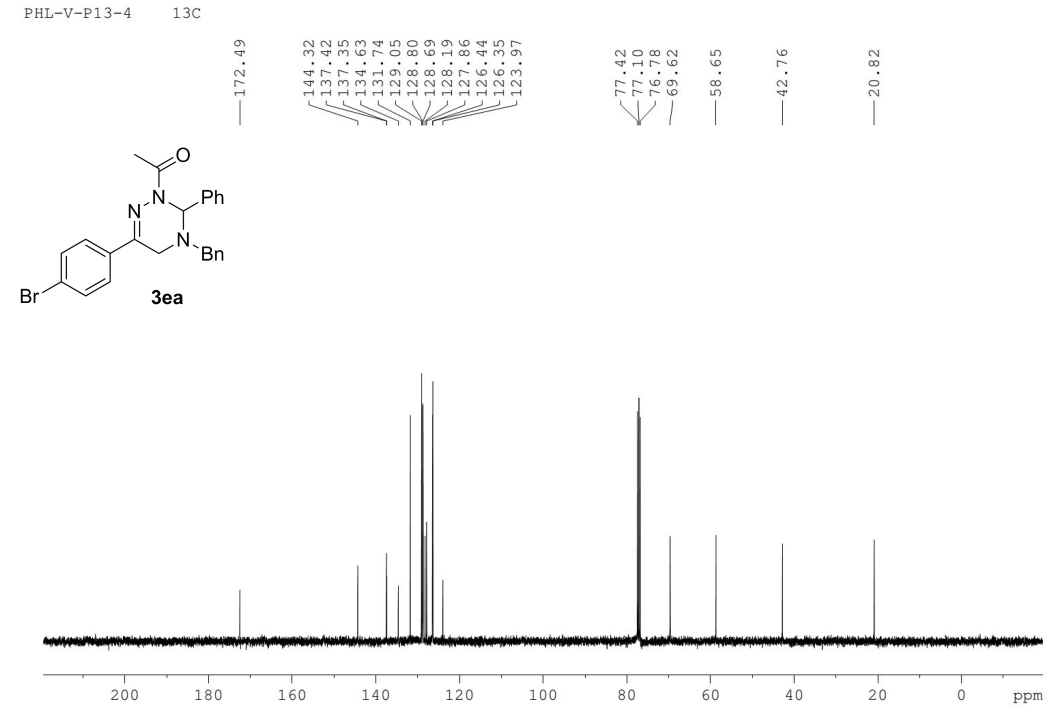




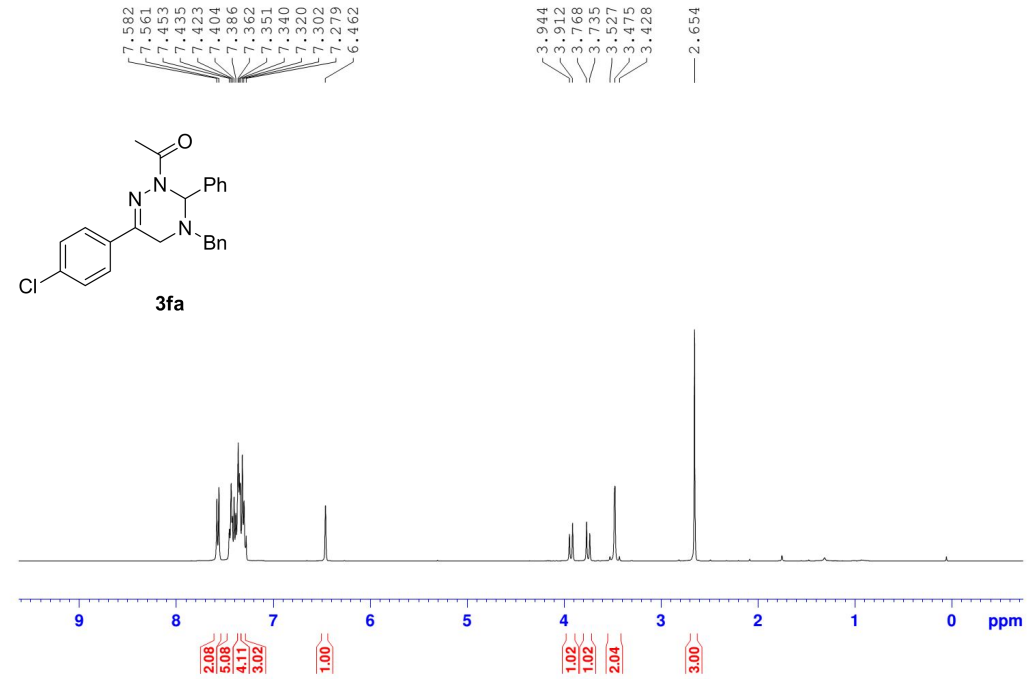
3da



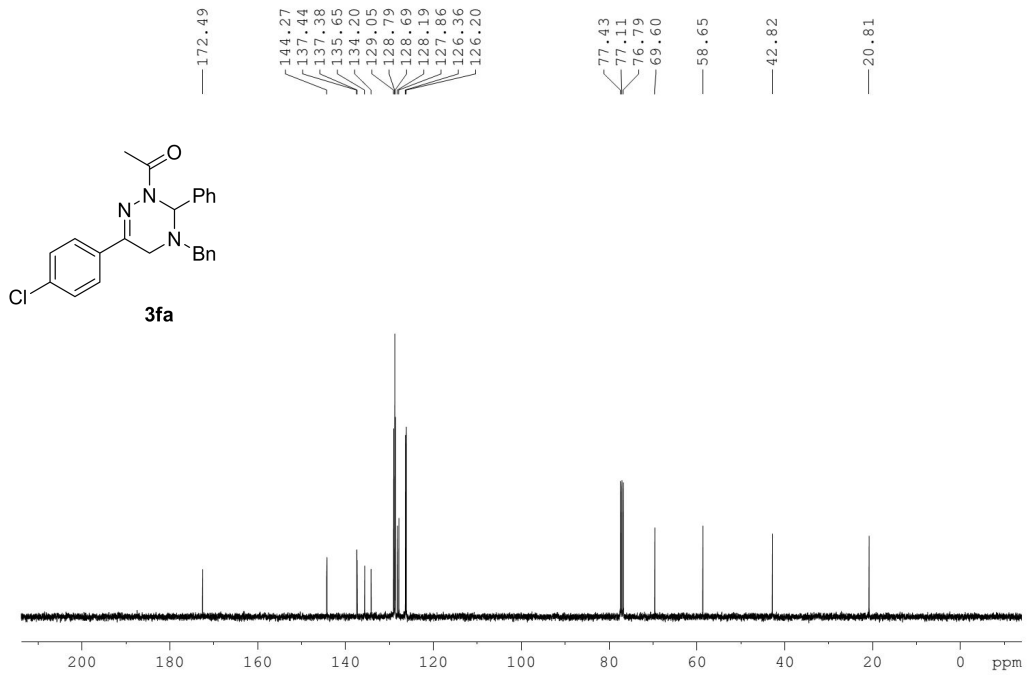

PHL-V-P13-4 1H
7.501
7.446
7.428
7.418
7.400
7.382
7.362
7.346
7.313
7.299
7.294
7.281
6.456
3.940
3.908
3.761
3.728
3.518
3.469
3.422
2.646
Ph
Bn
Br
3ea
9
8
7
6
5
4
3
2
1
0
ppm
4.03
3.11
4.03
3.10
1.00
2.05
2.02
3.00


PHL-V-P13-4 13C
172.49
144.32
137.42
137.35
134.63
131.74
129.05
128.80
128.69
128.19
127.86
126.44
126.35
123.97
77.42
77.10
76.78
69.62
58.65
42.76
20.82
Ph
Bn
Br
3ea
200
180
160
140
120
100
80
60
40
20
0
ppm

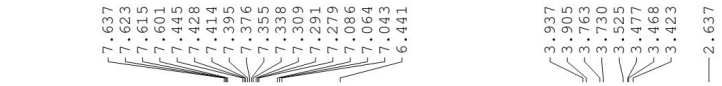
PHL-V-P13-5 NEW 1H

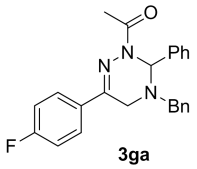


PHL-V-P13-5 13C

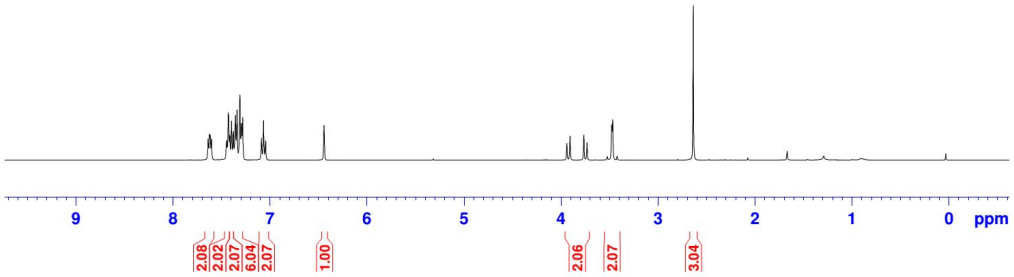


PHL-V-P13-6 NEW 1H

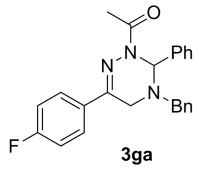




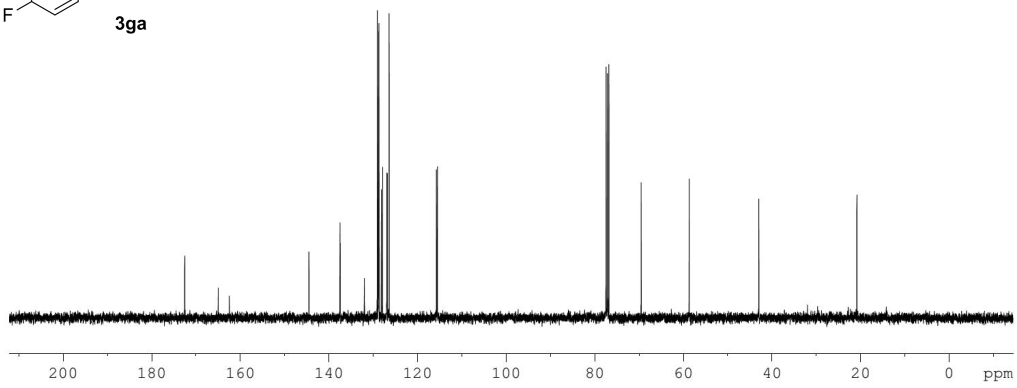
3ga



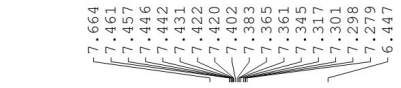
PHL-V-P13-6 13C

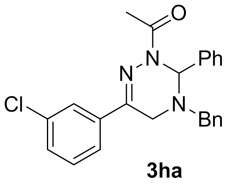


3ga

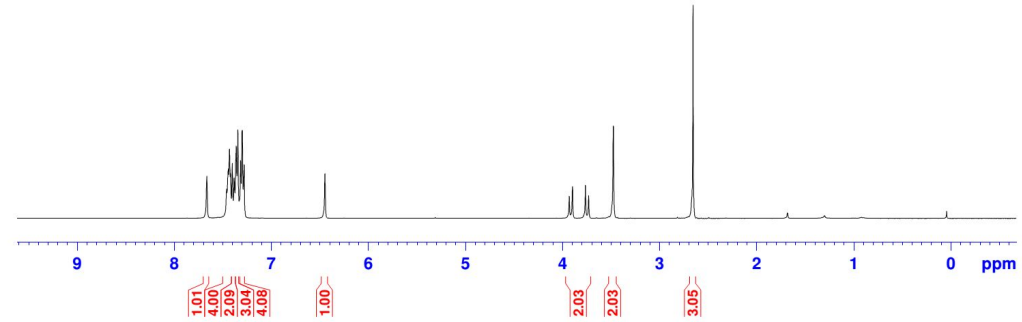


PHL-V-P13-7 NEW 1H

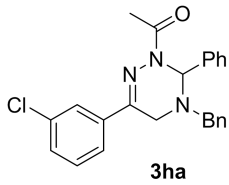




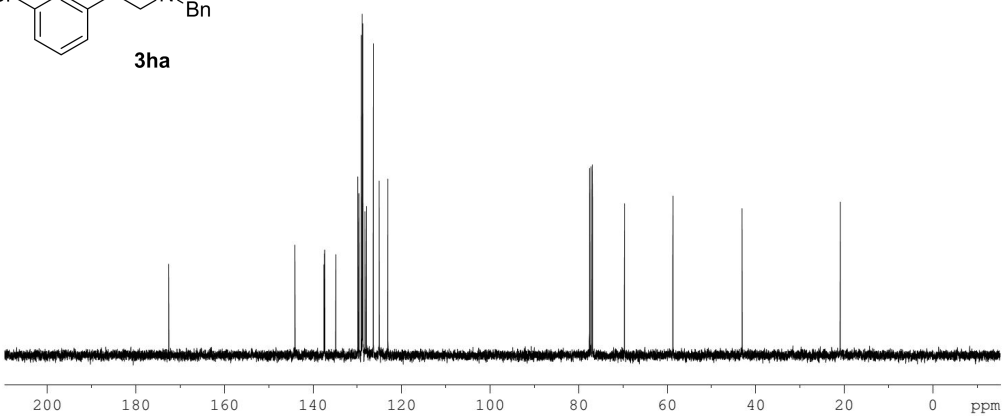
3ha



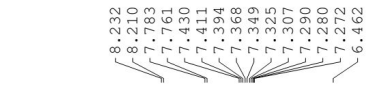
PHL-V-P13-7 13C

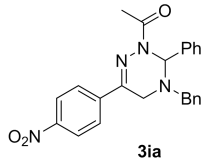


3ha

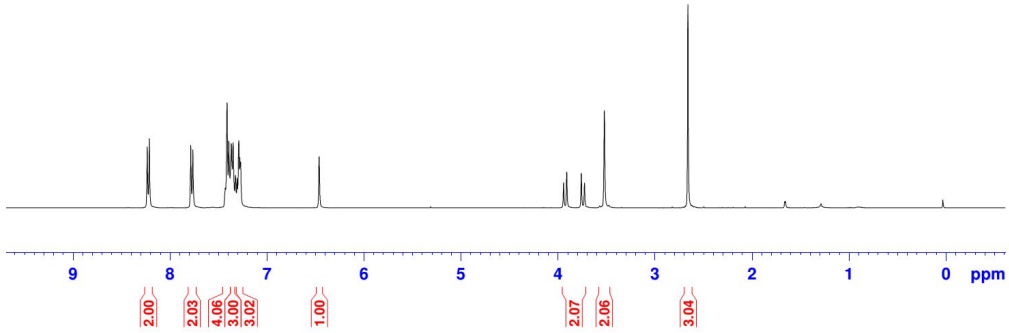


PHL-V-P13-9 NEW 1H

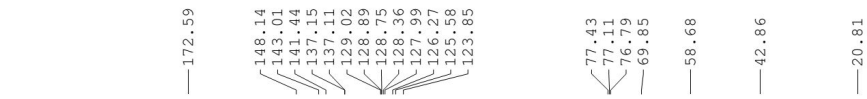


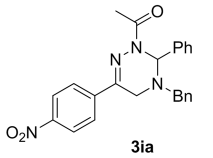


3ia



PHL-V-P13-9 13C





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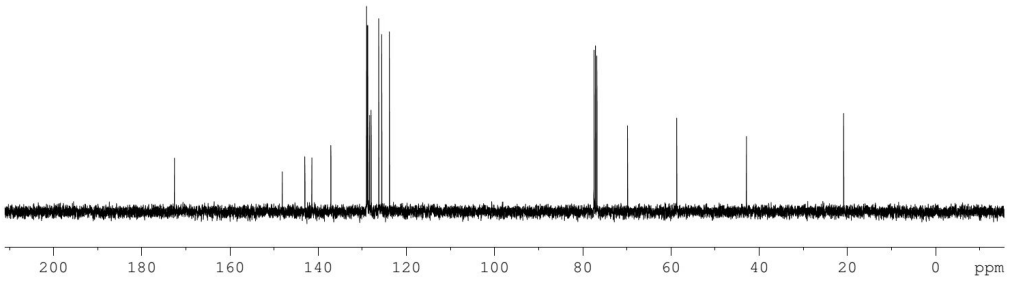

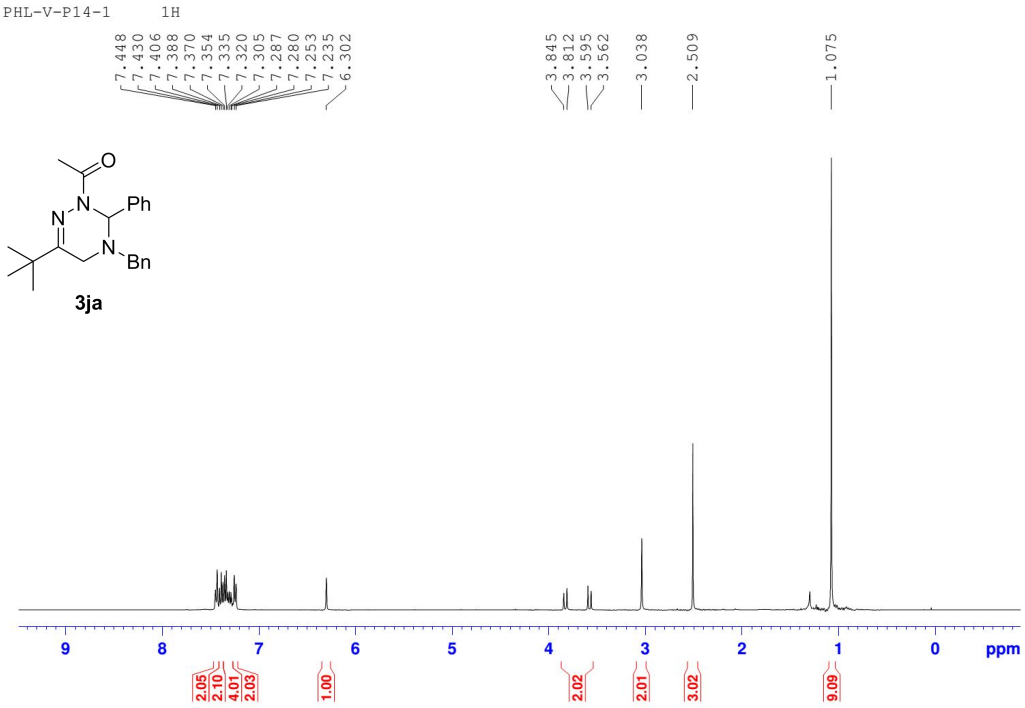
PHL-V-P14-1 1H
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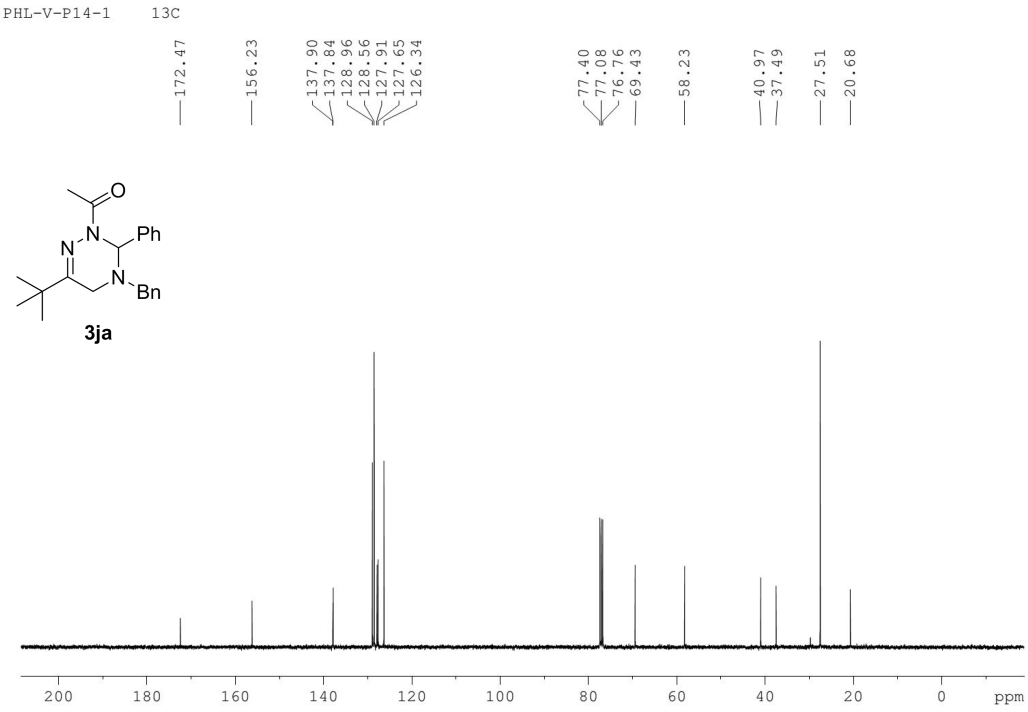
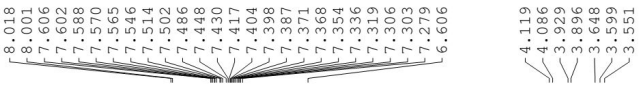
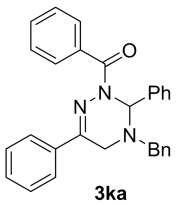
7.430
7.406
7.388
7.370
7.354
7.335
7.320
7.305
7.287
7.280
7.253
7.235
6.302
3.845
3.812
3.595
3.562
3.038
2.509
1.075
O
N
N
Ph
N
Bn
3ja
9
8
7
6
5
4
3
2
1
0
ppm
2.05
2.10
4.01
2.03
1.00
2.02
2.01
3.02
9.09

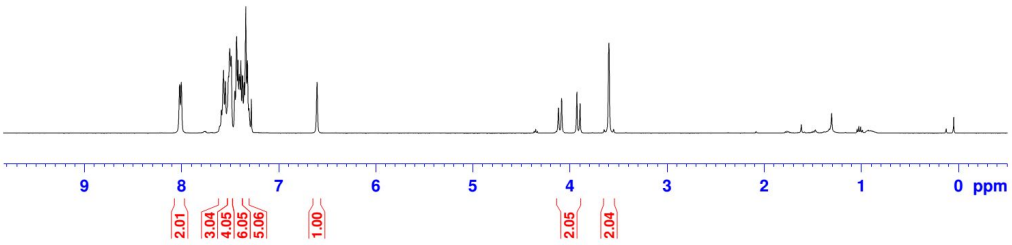

PHL-V-P14-1 13C
172.47
156.23
137.90
137.84
128.96
128.56
127.91
127.65
126.34
77.40
77.08
76.76
69.43
58.23
40.97
37.49
27.51
20.68
O
N
N
Ph
N
Bn
3ja
200
180
160
140
120
100
80
60
40
20
0
ppm

PHL-V-P14-2 NEW 1H

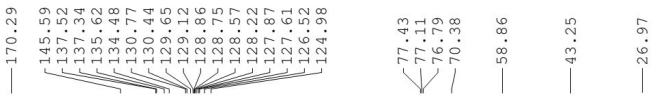


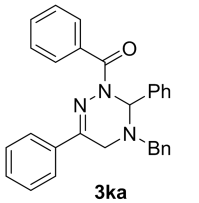


3ka

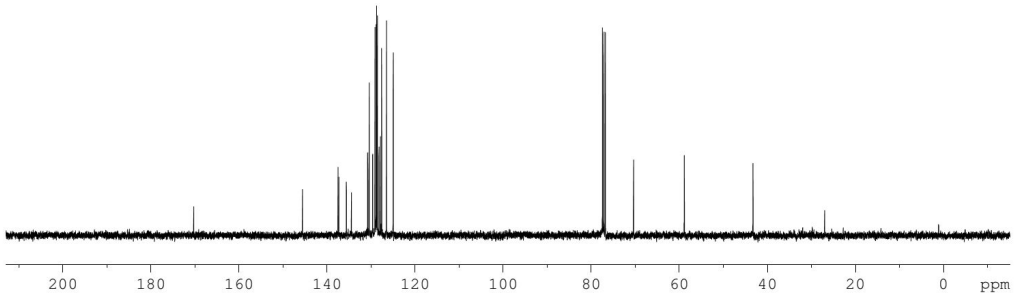


PHL-V-P14-2 13C

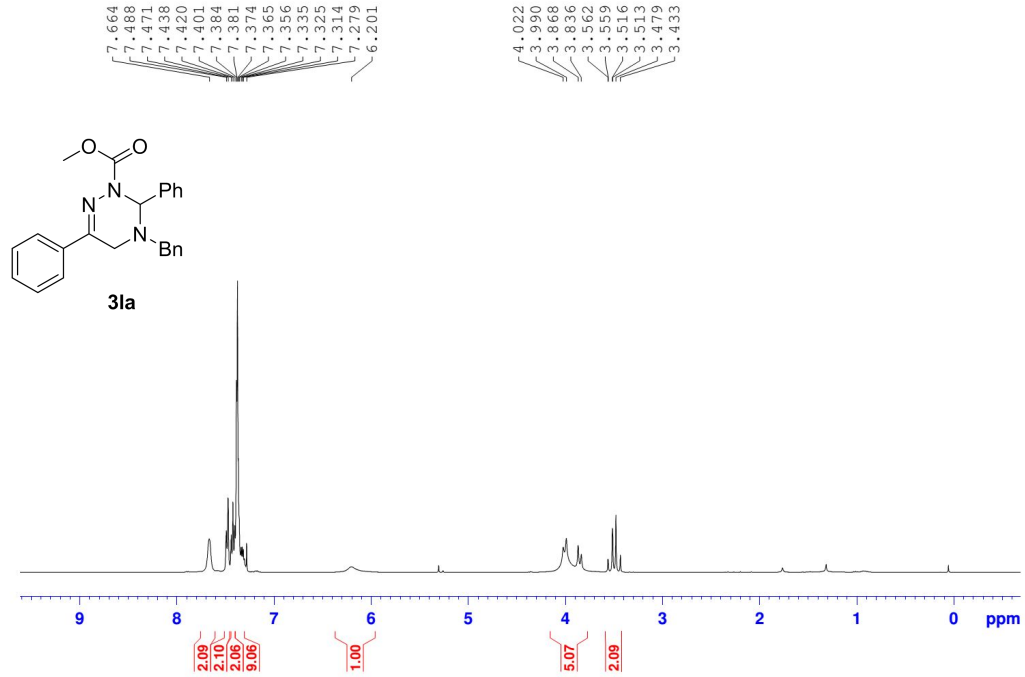




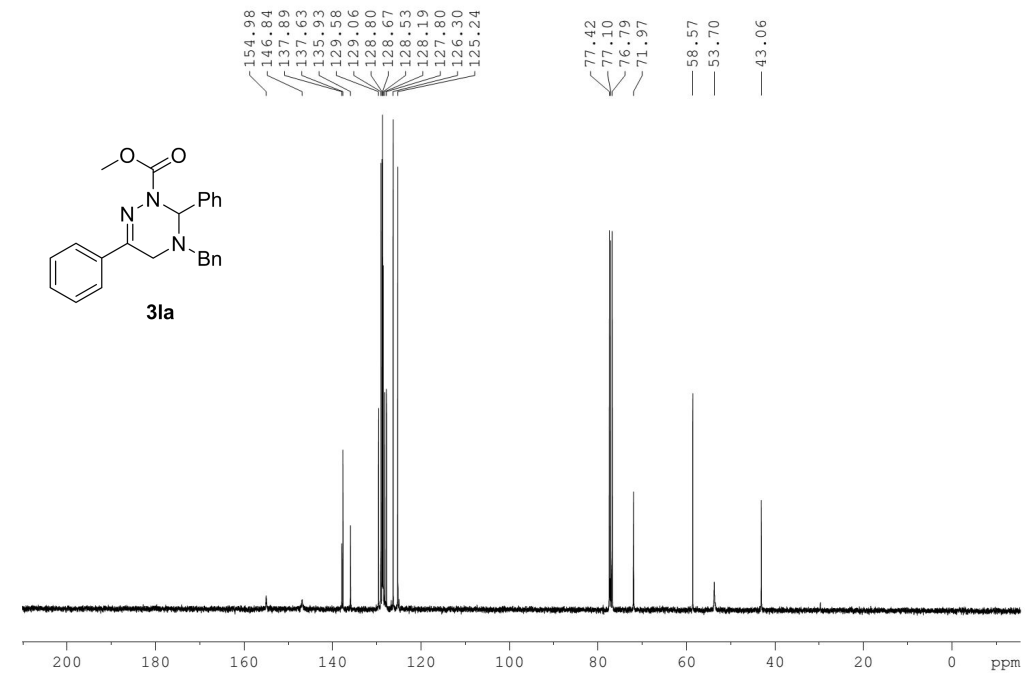
3ka



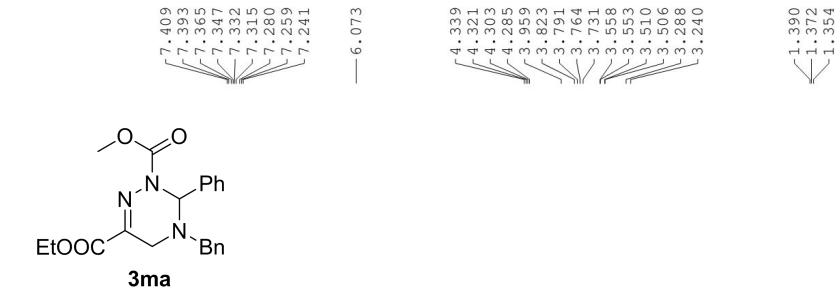
PHL-V-P14-4 NEW 1H



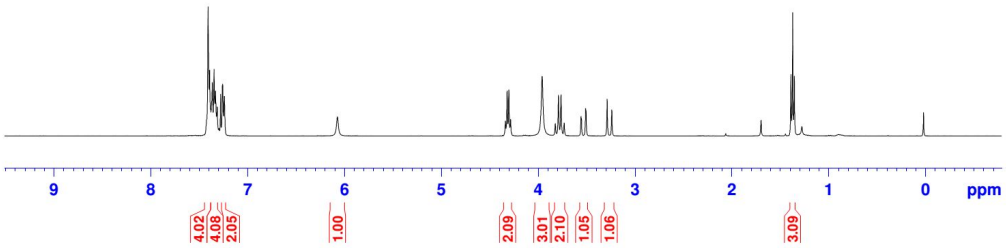
PHL-V-P14-4 NEW 13C



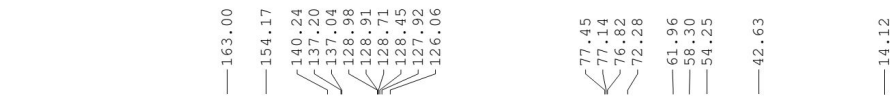
PHL-V-P14-5 NEW 1H



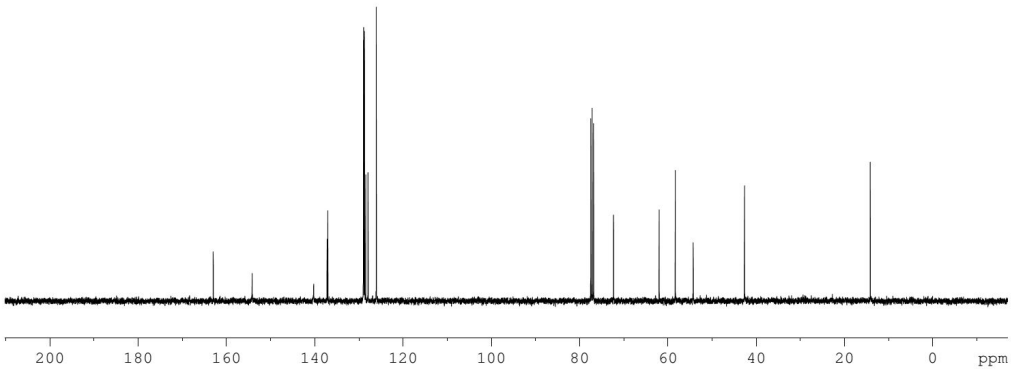
3ma



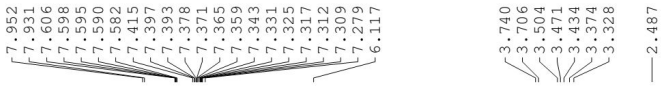
PHL-V-P14-5 13C

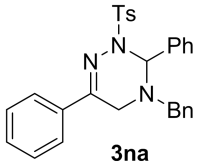


3ma

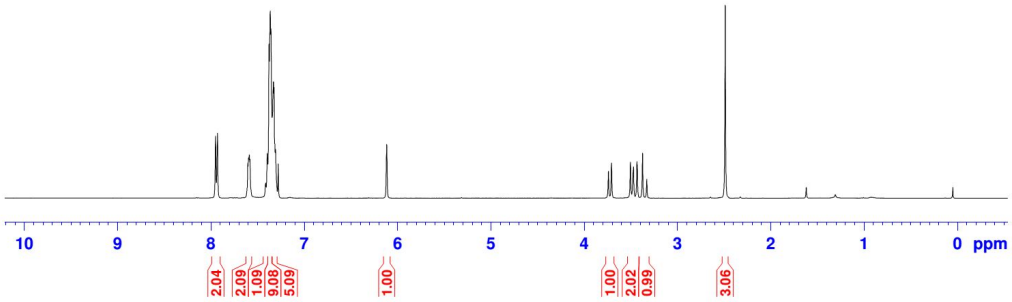


PHL-V-P14-7 NEW 1H

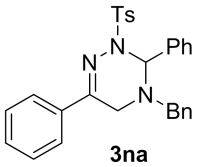




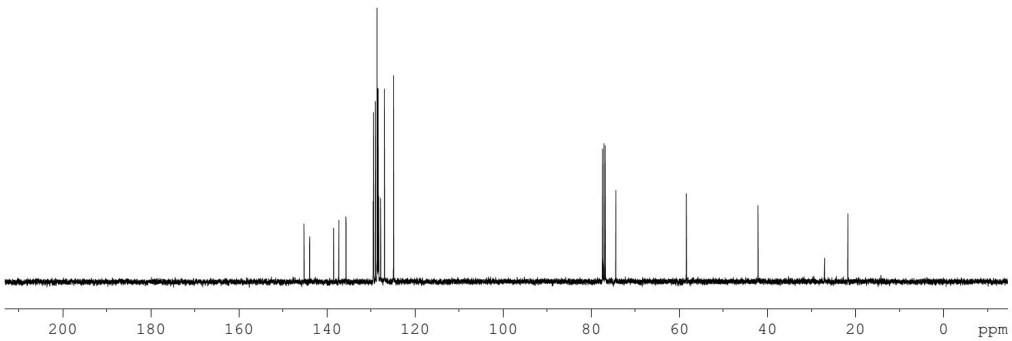
3na



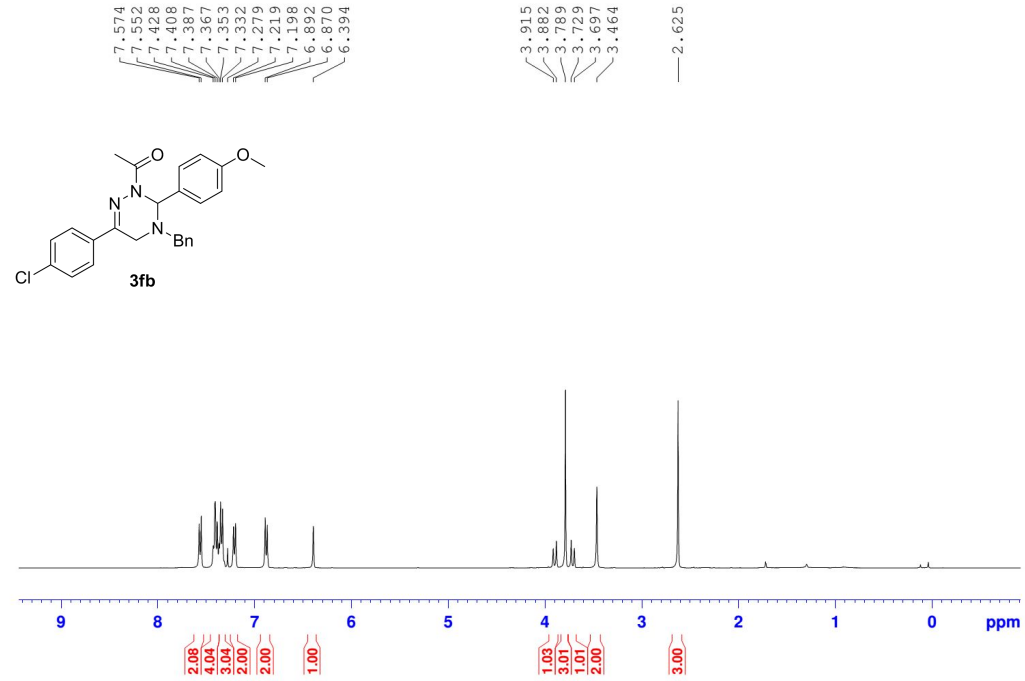
PHL-V-P14-7 13C



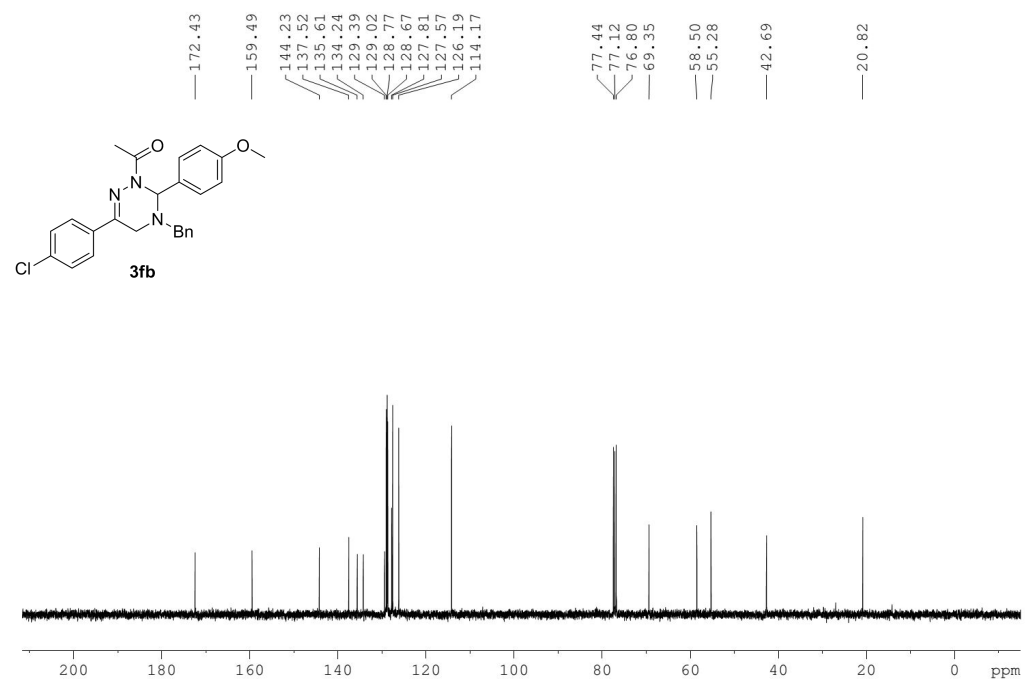
3na



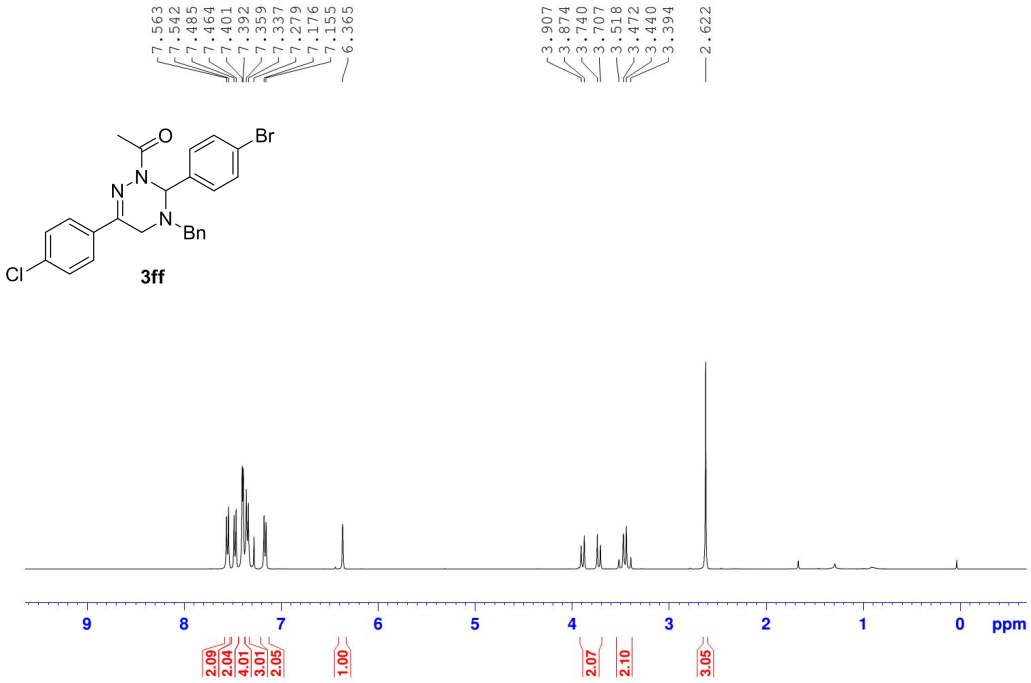
PHL-V-P21-1 NEW 1H



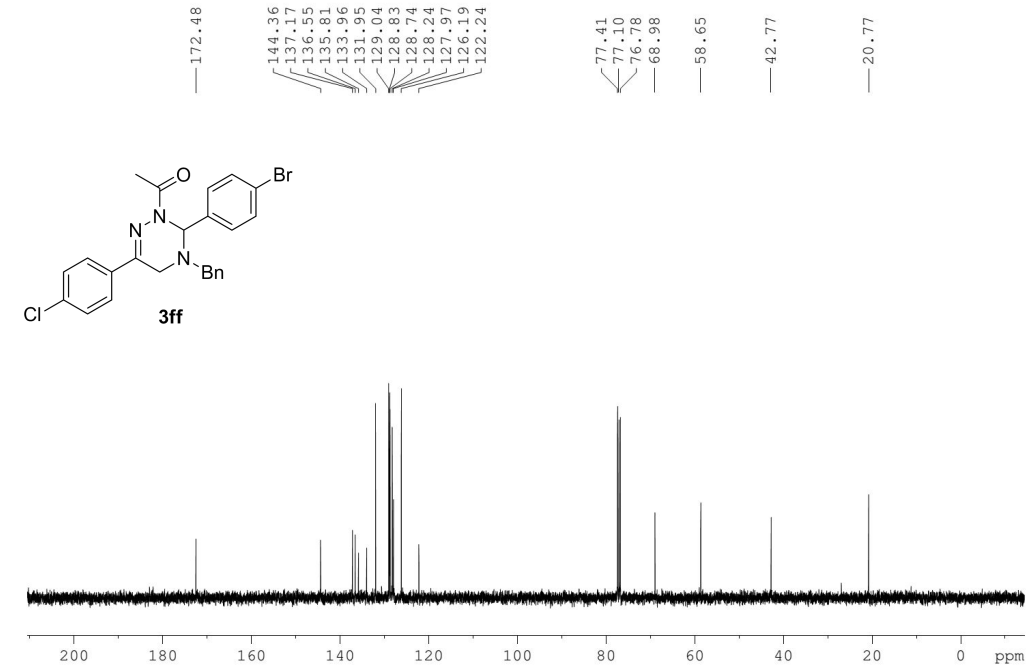
PHL-V-P21-1 13C



PHL-V-P21-2 NEW 1H



PHL-V-P21-2 13C



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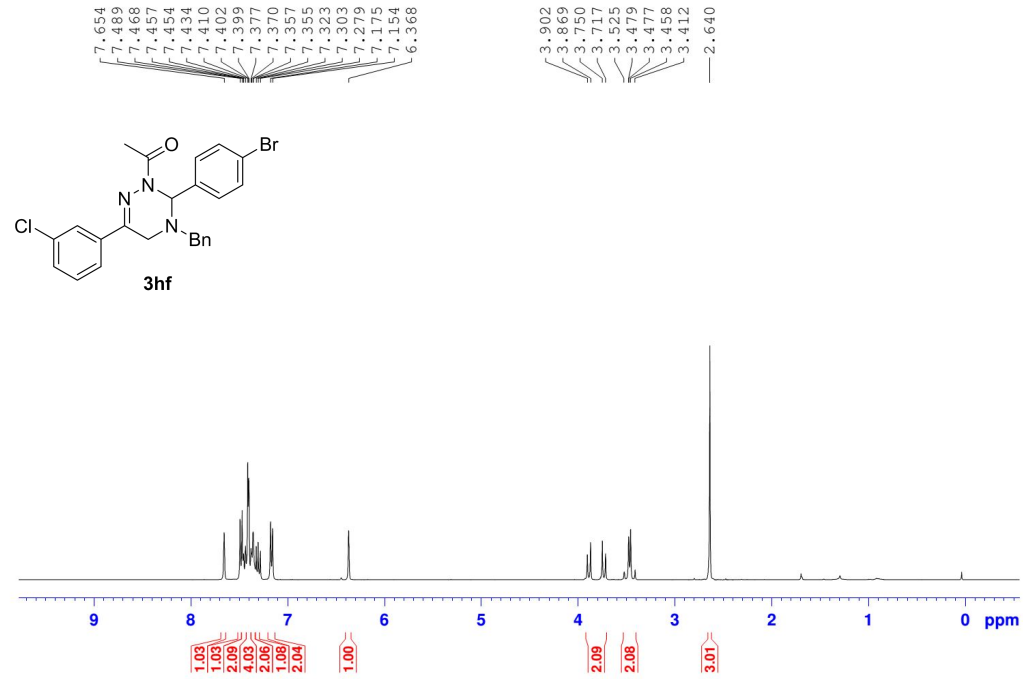

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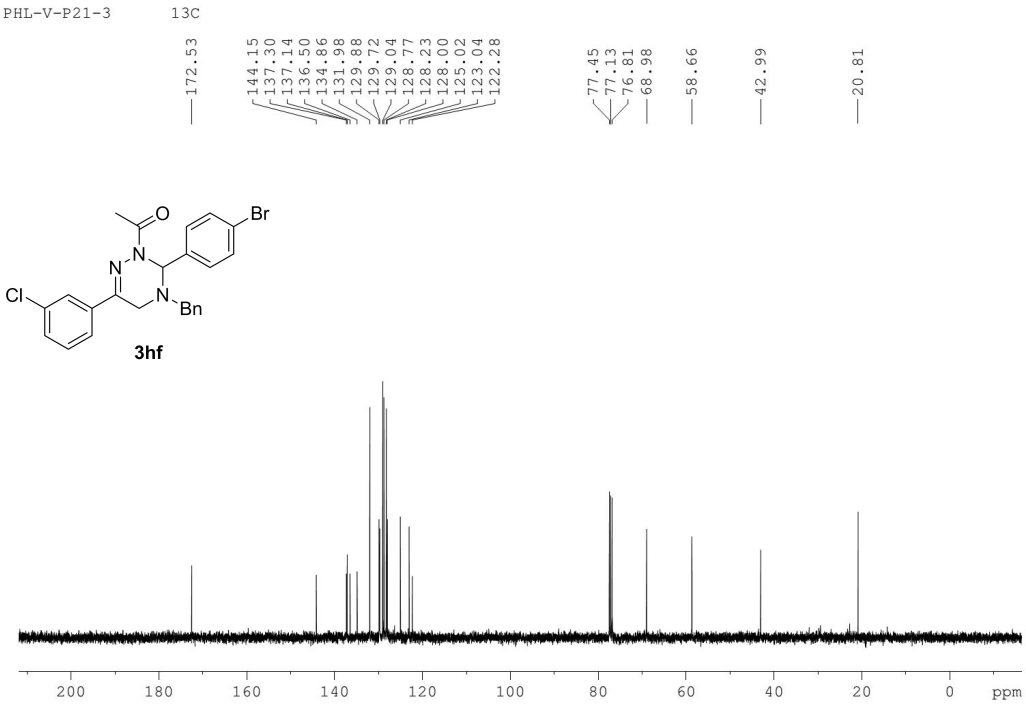

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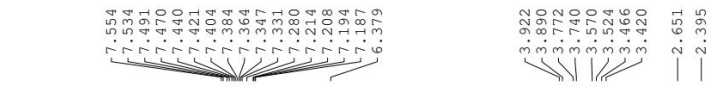
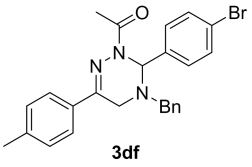
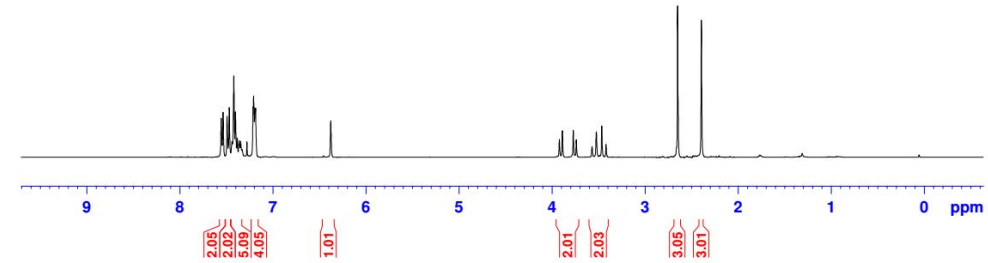




3df



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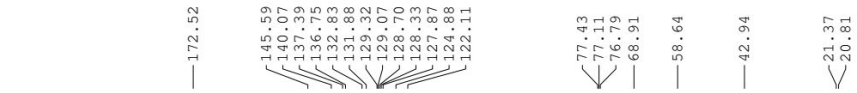

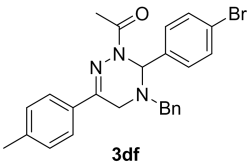

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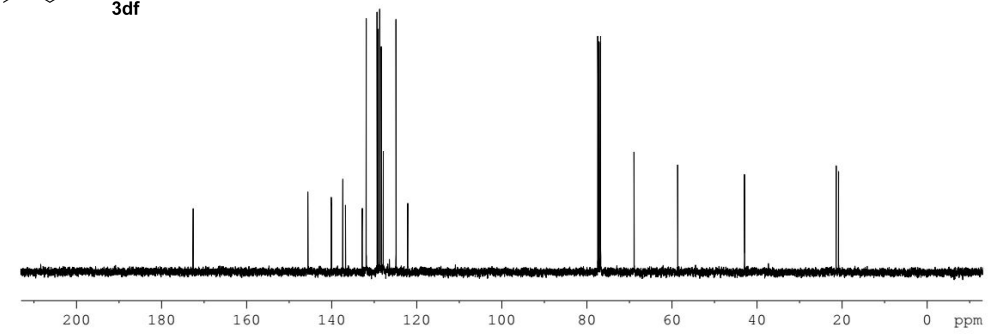

2. X-Ray crystal data of compound 3aa

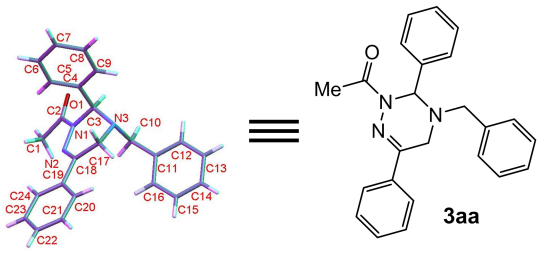

Figure 1. X-ray single crystal structure of 3aa (with thermal ellipsoid shown at the 50% probability level)

Identification code	**3aa**
Empirical formula	C_{24} H_{23} N_3 O
Formula weight	369.45
Temperature	113(2) K
Wavelength	0.71073 A
Crystal system, space group	Orthorhombic, Pbca
Unit cell dimensions	a = 13.3996(18) A alpha = 90 deg. b = 12.191(2) A beta = 90 deg. c = 22.958(3) A gamma = 90 deg.
Volume	3750.3(9) A^3
Z, Calculated density	8, 1.309 Mg/m^3
Absorption coefficient	0.081 mm^-1
F(000)	1568
Crystal size	0.20 x 0.18 x 0.12 mm
Theta range for data collection	3.04 to 27.62 deg.
Limiting indices	-17<=h<=17, -15<=k<=15, -29<=l<=29
Reflections collected / unique	45028 / 4327 [R(int) = 0.0237]

Completeness to theta = 25.02	99.4%
Absorption correction	Semi-empirical from equivalents
Max. and min. transmission	0.9903 and 0.9839
Refinement method	Full-matrix least-squares on F^2
Data / restraints / parameters	4327 / 0 / 254
Goodness-of-fit on F^2	1.069
Final R indices [I>2sigma(I)]	R1 = 0.0343, wR2 = 0.0915
R indices (all data)	R1 = 0.0377, wR2 = 0.0934
Largest diff. peak and hole	0.337 and -0.251 e.A^-3