

Supporting Information

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

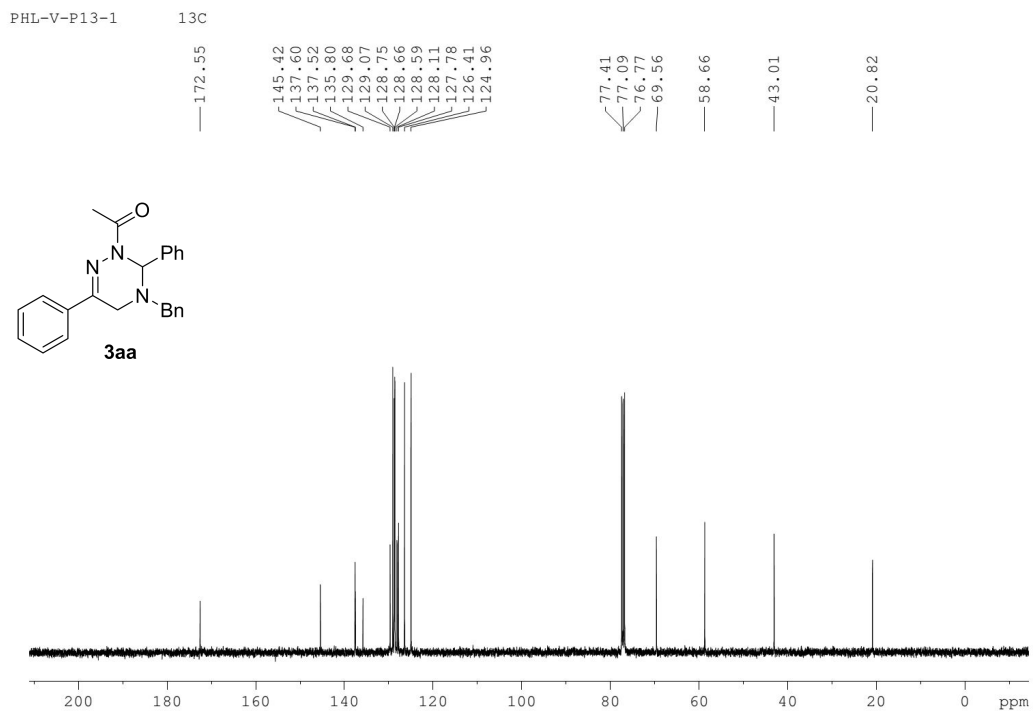
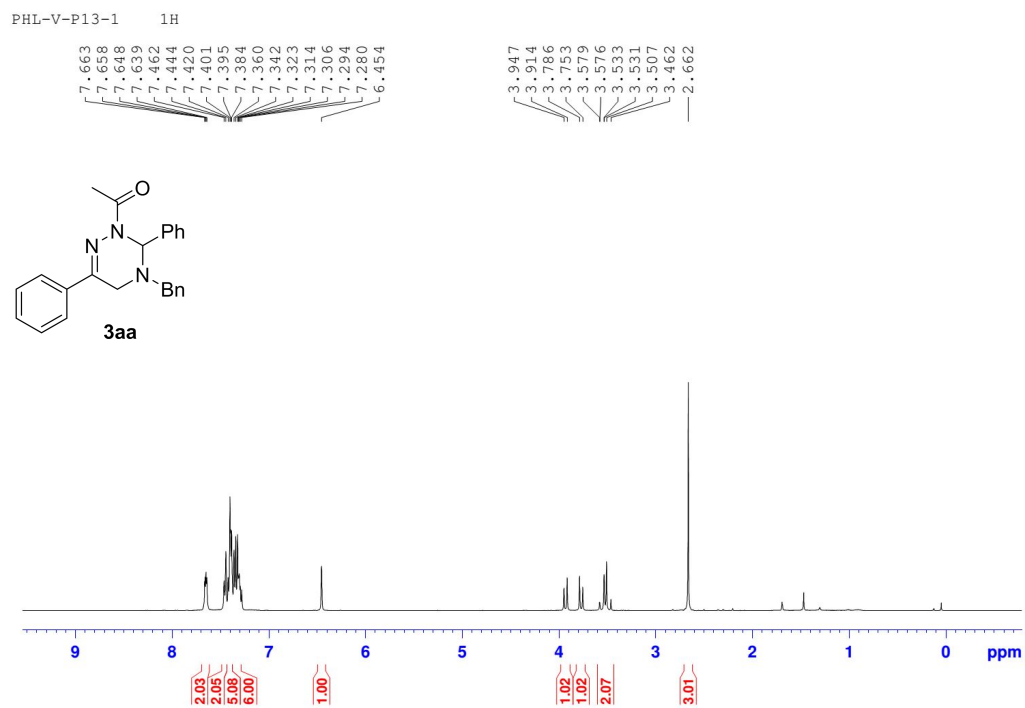
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Xiu-Qing Song, Ning-Ning Feng and Juan Du

College of Life Science and Bio-engineering, Beijing University of Technology, Beijing 100124,
P.R. China; *Phone: 86-10-6739-6211; Fax: 86-10-6739-6211; Email: hwzhao@bjut.edu.cn;*
zhaolijiao@bjut.edu.cn.

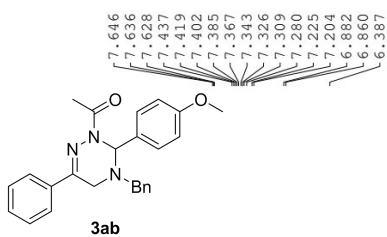
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1. ^1H and ^{13}C NMR spectra



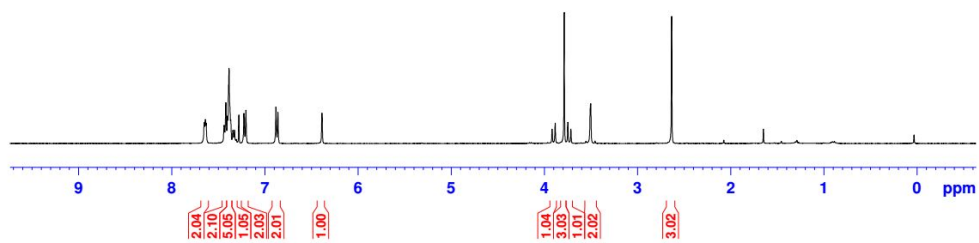
PHL-V-P17-4 NEW 1H



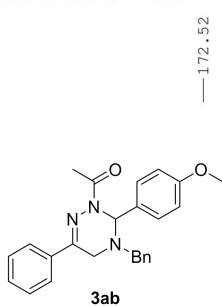
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7.437
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7.402
7.385
7.367
7.343
7.326
7.309
7.280
7.225
7.204
6.882
6.860
6.387

3.917
3.884
3.787
3.748
3.715
3.554
3.502
3.456

— 2.634



PHL-V-P17-4 13C



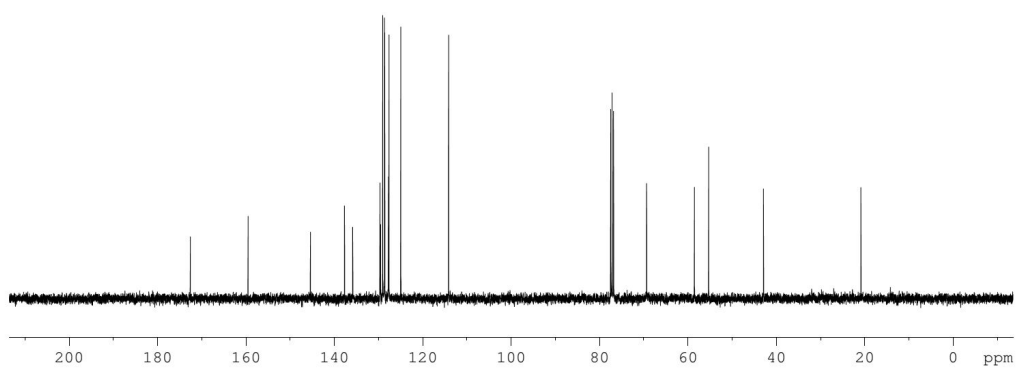
— 172.52
— 159.45
145.41
137.69
135.83
129.67
129.55
129.04
128.65
128.59
127.75
127.63
124.95
114.14

77.45
77.14
76.82
— 69.32

— 58.51
— 55.29

— 42.89

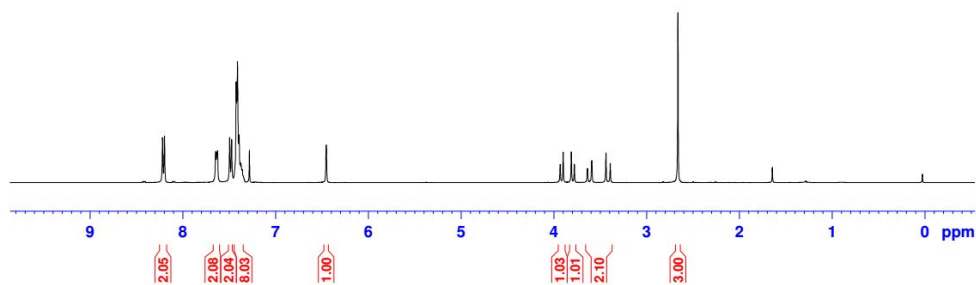
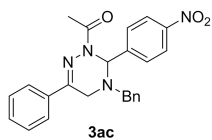
— 20.85



PHL-V-P17-5 NEW 1H

8.217
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

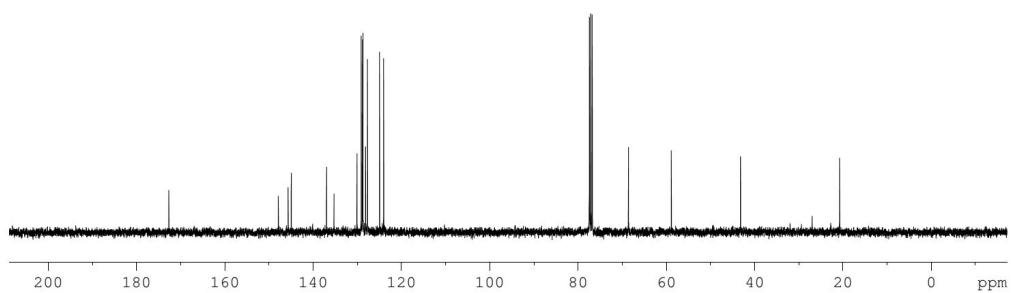
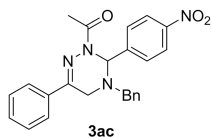
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3.900
3.812
3.780
3.639
3.592
3.439
3.393
—2.663



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

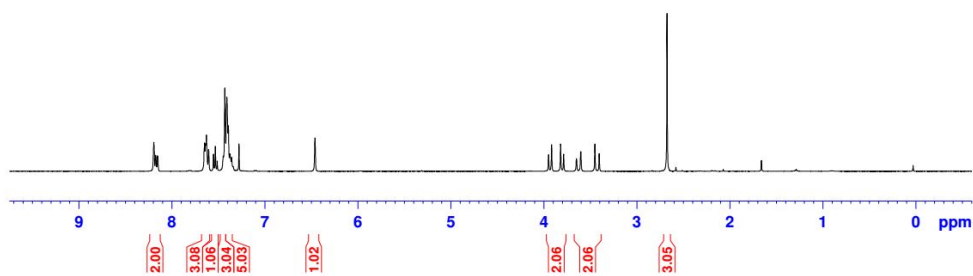
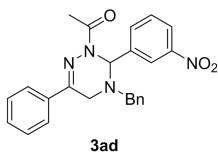
77.40
77.08
76.76
—68.55
—58.85
—43.17
—20.75



PHL-V-P17-6 NEW 1H

8.196
8.176
8.156
7.650
7.638
7.630
7.606
7.554
7.534
7.514
7.449
7.432
7.410
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7.380
7.375
7.358
7.280
6.462

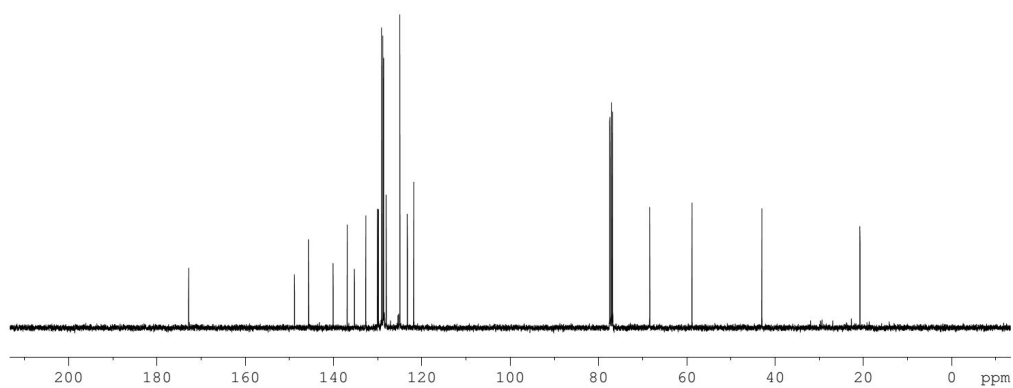
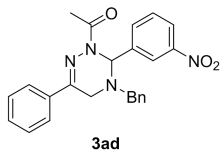
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3.649
3.603
3.452
3.406
— 2.676



PHL-V-P17-6 13C

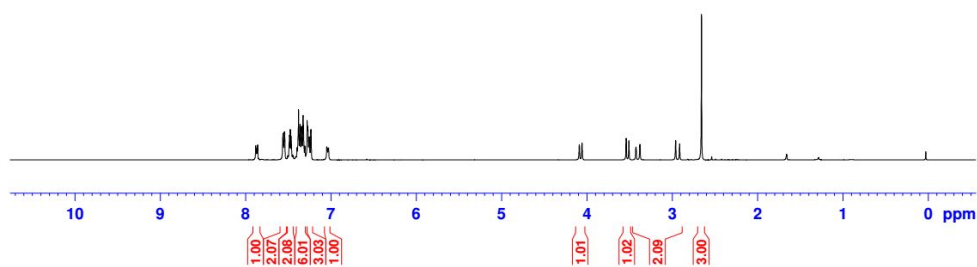
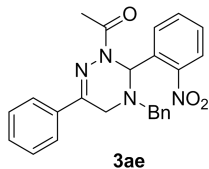
— 172.75
148.85
145.70
140.11
136.92
135.28
132.71
130.04
129.86
129.15
128.83
128.69
128.10
125.00
123.31
121.86

77.43
77.11
76.79
— 68.40
— 58.82
— 43.00
— 20.78



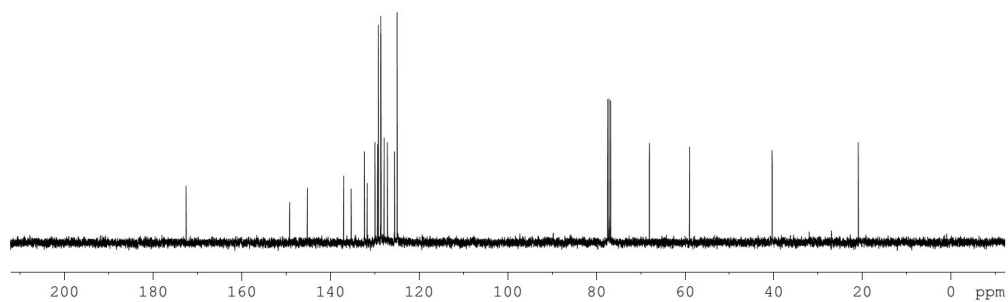
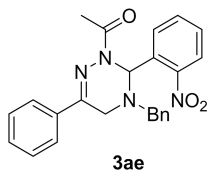
PHL-V-P17-7 NEW 1H

7.882
7.878
7.866
7.859
7.562
7.546
7.542
7.505
7.491
7.486
7.477
7.468
7.463
7.448
7.397
7.380
7.361
7.345
7.327
7.306
7.279
7.274
7.271
7.255
7.235
7.050
7.043
7.028
4.090
4.058
3.940
3.508
3.426
3.423
3.380
3.377
2.960
2.914
2.657



PHL-V-P17-7 13C

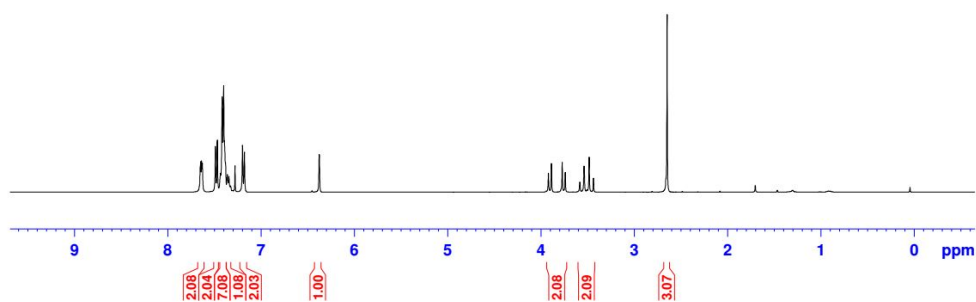
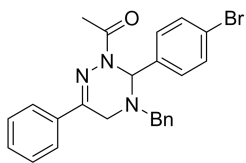
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137.04
135.33
132.31
131.73
129.95
129.37
129.21
128.68
128.60
127.89
127.19
125.52
124.95
77.44
77.12
76.80
68.07
58.98
40.35
20.90



PHL-V-P19-7 NEW 1H

7.652
7.648
7.639
7.629
7.490
7.469
7.436
7.417
7.401
7.394
7.382
7.361
7.345
7.328
7.280
7.199
7.179
6.377

3.921
3.888
3.773
3.740
3.585
3.539
3.484
3.438
— 2.648



PHL-V-P19-7 13C

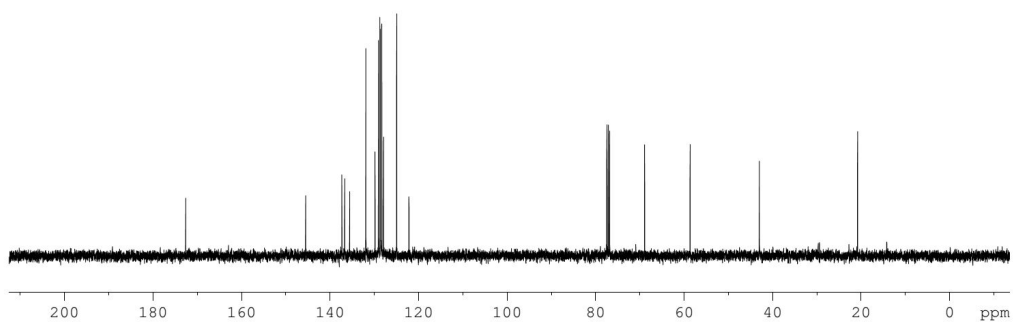
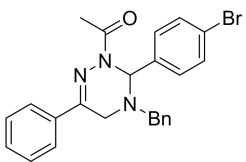
— 172.57

145.54
137.35
136.71
135.56
131.93
129.86
129.08
128.74
128.66
128.32
127.92
124.96
122.18

77.47
77.15
76.83
— 68.94
— 58.67

— 43.00

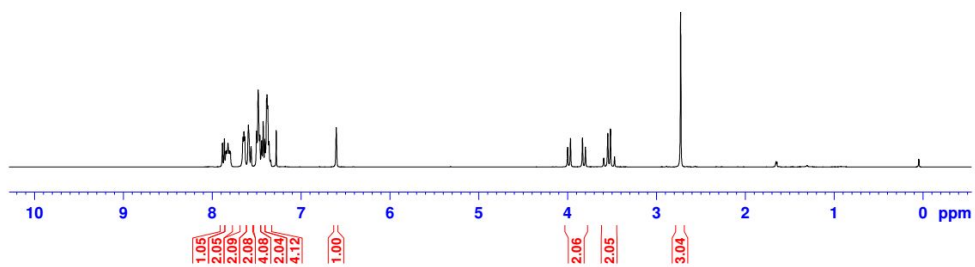
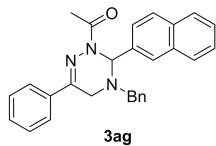
— 20.82



PHL-V-P19-4 NEW 1H

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7.864
7.848
7.838
7.823
7.805
7.796
7.653
7.643
7.634
7.593
7.562
7.500
7.483
7.464
7.445
7.427
7.408
7.388
7.383
7.376
7.361
7.343
7.280
6.604

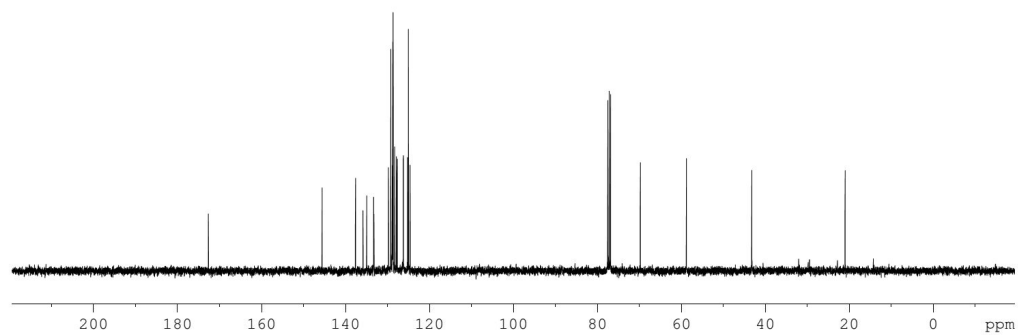
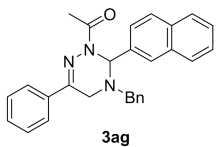
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3.967
3.832
3.799
3.593
3.547
3.517
3.472
— 2.728



PHL-V-P19-4 13C

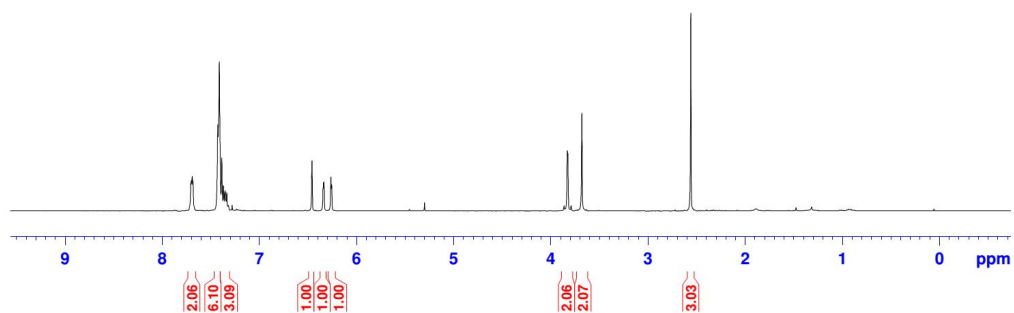
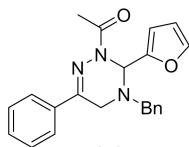
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137.61
135.80
134.91
133.28
133.20
129.74
129.17
128.86
128.74
128.62
128.28
127.88
127.64
126.24
126.19
125.19
125.00
124.59

77.47
77.16
76.84
69.73
— 58.76
— 43.17
— 20.96



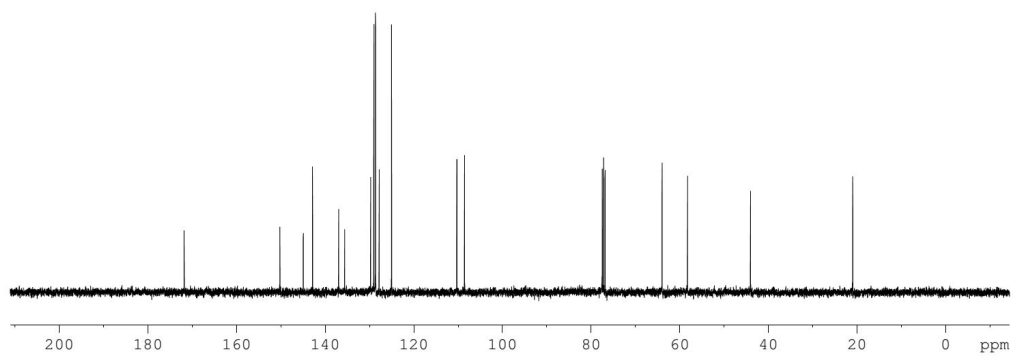
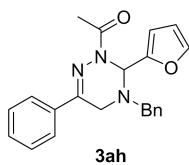
PHL-V-P19-5 1H

7.705
7.699
7.690
7.682
7.427
7.411
7.386
7.367
7.352
7.335
7.280
6.458
6.344
6.337
6.332
6.263
6.255
3.866
3.852
3.827
3.793
3.683
— 2.562

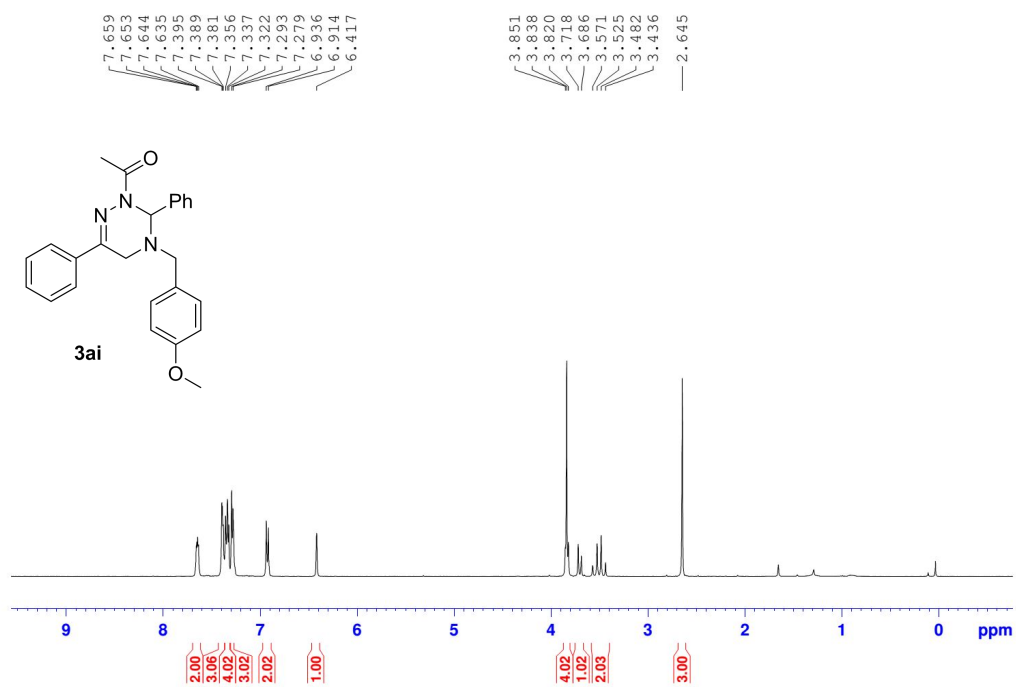


PHL-V-P19-5 13C

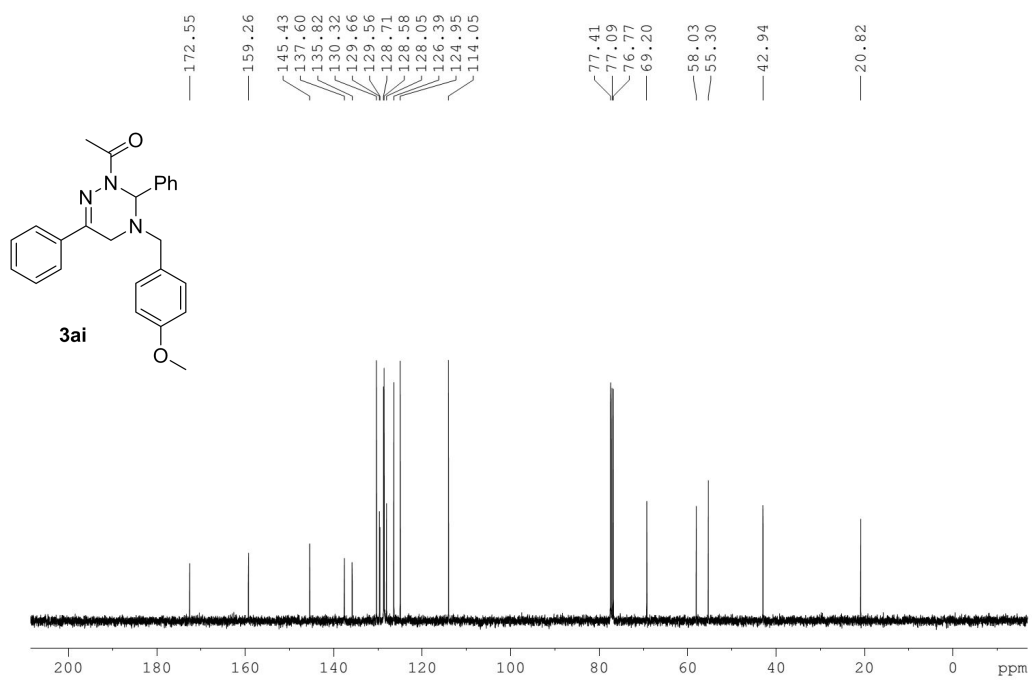
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150.23
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136.97
135.62
129.75
129.03
128.67
128.63
127.84
125.03
110.30
108.55
77.47
77.15
76.83
63.98
58.22
44.00
— 20.94



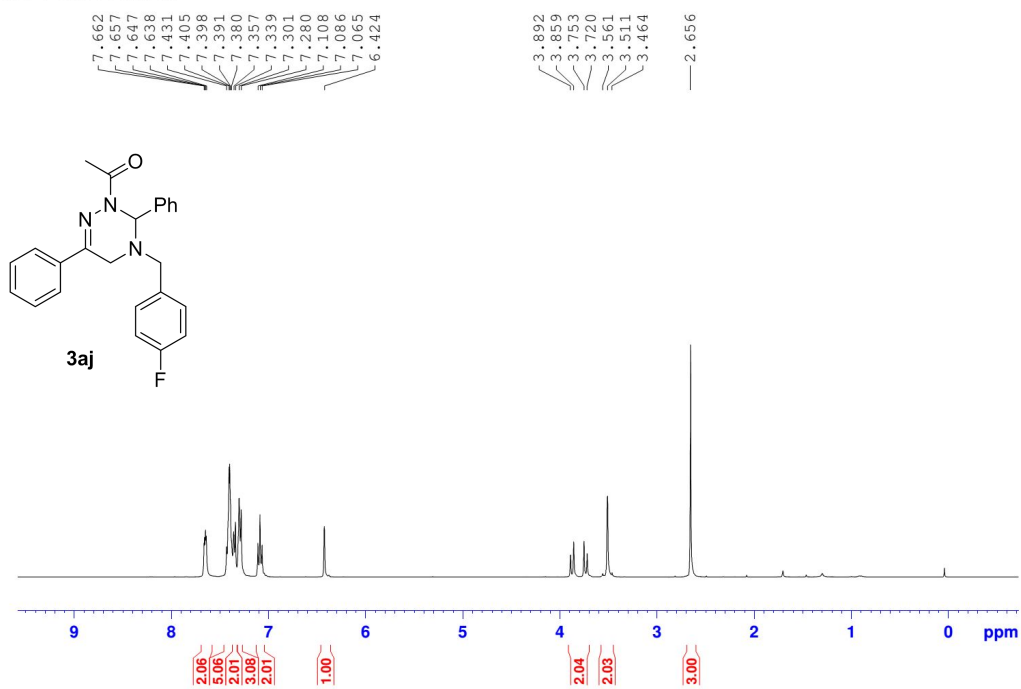
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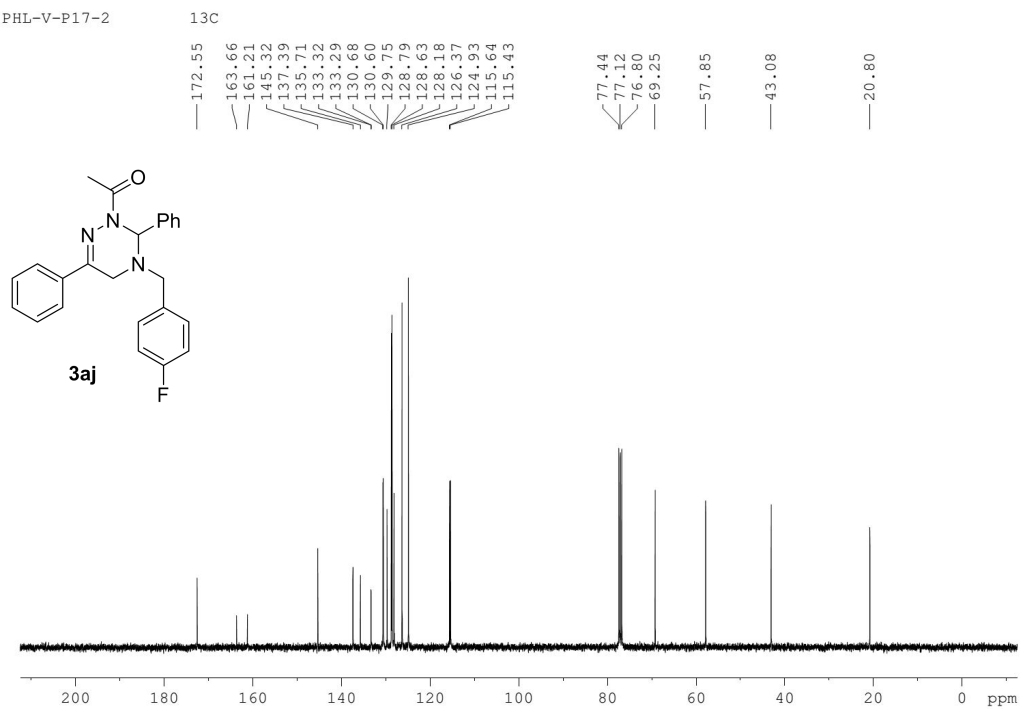
PHL-V-P17-3 13C



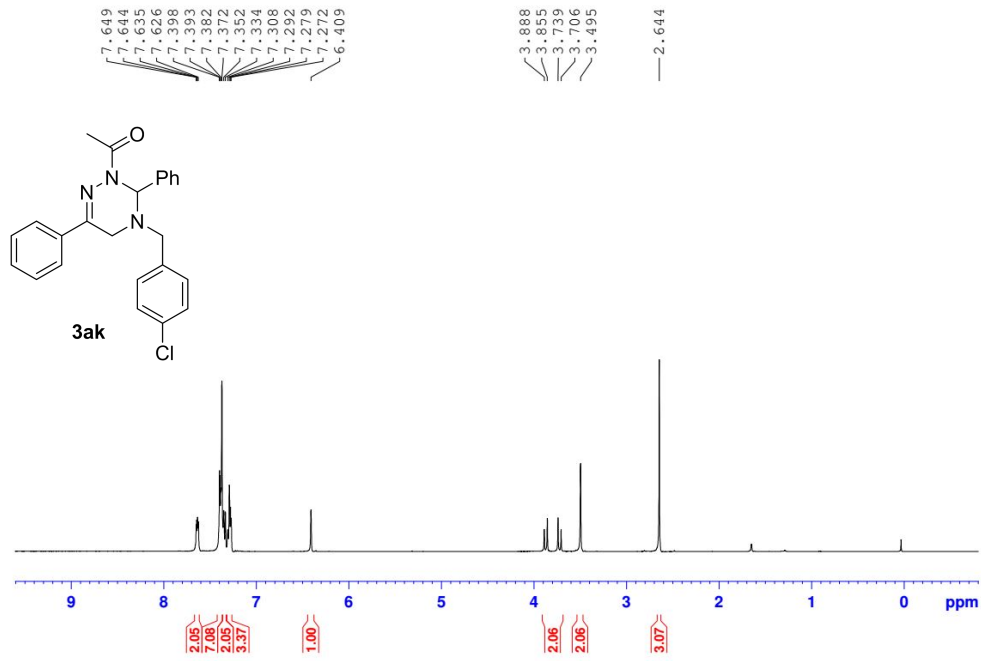
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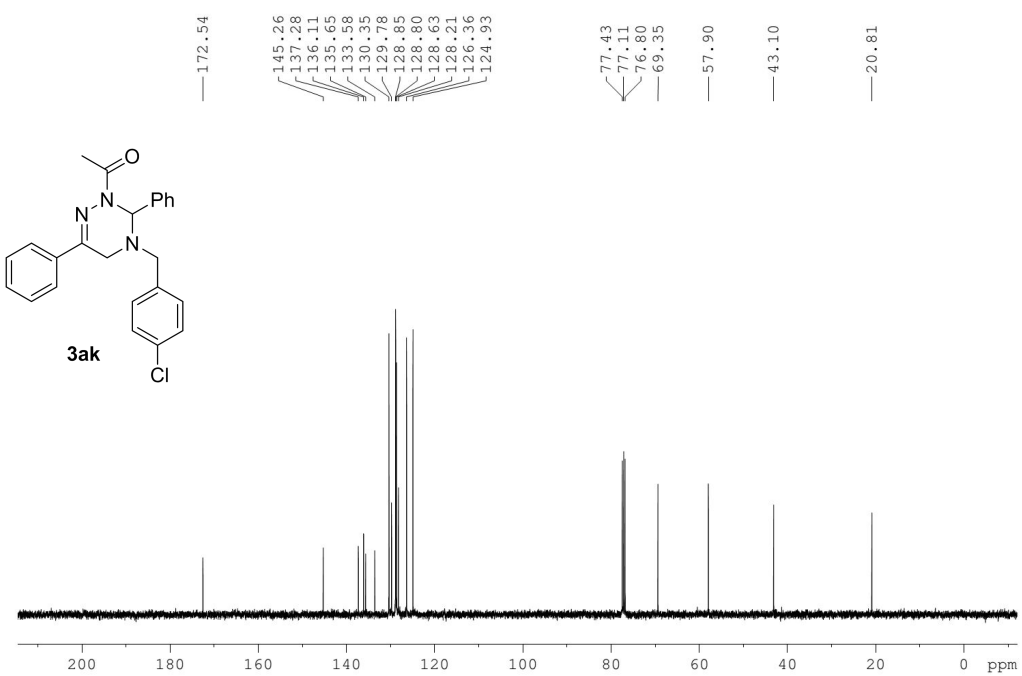
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PHL-V-P17-1 NEW 1H



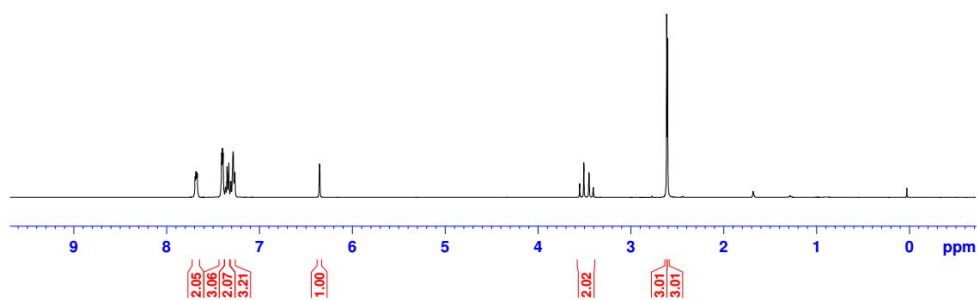
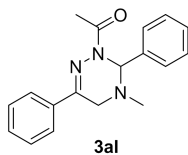
PHL-V-P17-1 13C



PHL-V-P19-2 NEW 1H

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7.676
7.670
7.607
7.399
7.391
7.366
7.349
7.331
7.308
7.283
7.266
6.353

3.551
3.506
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3.449
3.406
3.403
2.613
2.605



PHL-V-P19-2 13C

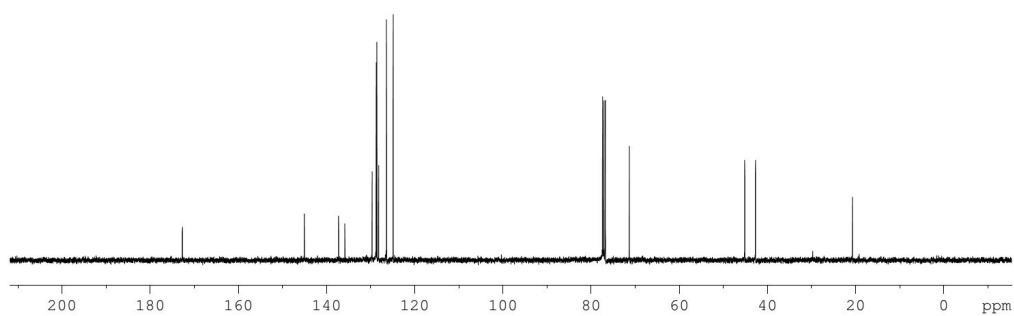
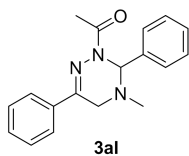
172.69

145.03
137.26
135.85
129.66
128.74
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128.17
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77.39
77.07
76.76
71.29

45.12
42.70

20.70

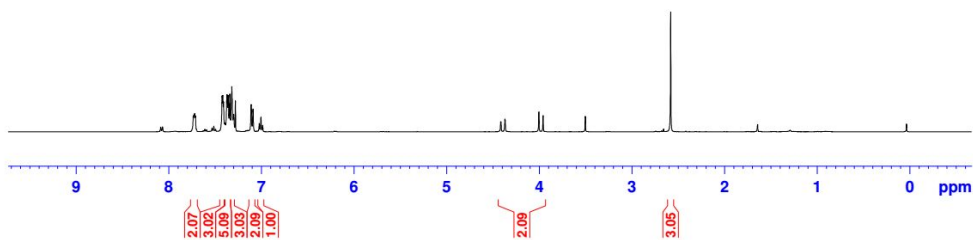
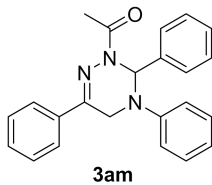


PHL-V-P19-1 NEW 1H

7.735
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7.724
7.720
7.711
7.711
7.424
7.416
7.408
7.370
7.357
7.340
7.319
7.298
7.280
7.109
7.089
7.022
7.004
6.985

4.415
4.412
4.370
4.366
4.003
3.957

— 2.579



PHL-V-P19-1 13C

— 171.84

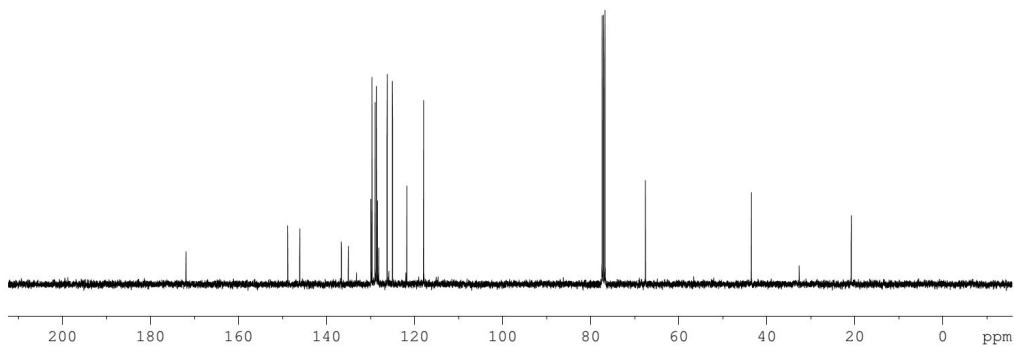
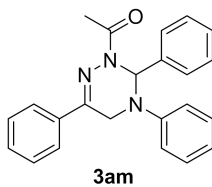
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146.07
136.63
135.06
129.91
129.68
128.95
128.67
128.63
128.39
128.15
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125.00
121.74
117.94

77.38
77.07
76.75
— 67.58

— 43.46

— 32.62

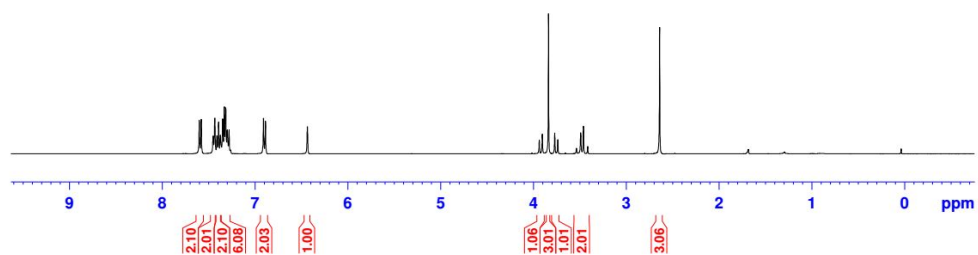
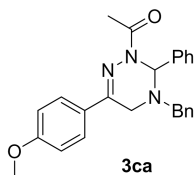
— 20.78



PHL-V-P13-2 NEW 1H

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7.581
7.452
7.435
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7.375
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6.437

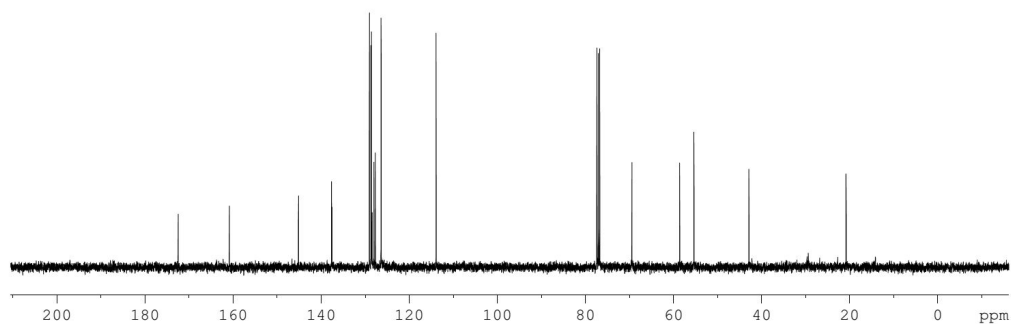
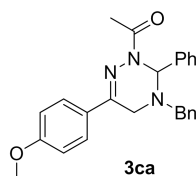
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3.771
3.738
3.537
3.491
3.461
3.416
— 2.640



PHL-V-P13-2 13C

172.44
160.88
145.18
137.68
137.58
129.08
128.72
128.64
128.46
128.05
127.75
126.44
126.41
113.94

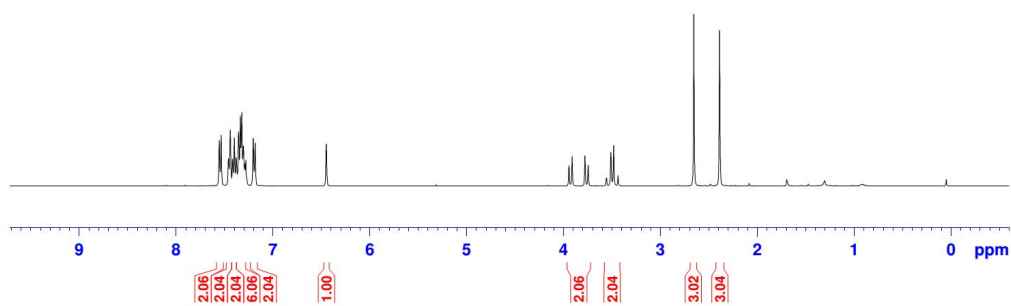
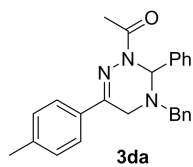
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55.38
42.99
20.83



PHL-V-P13-3 NEW 1H

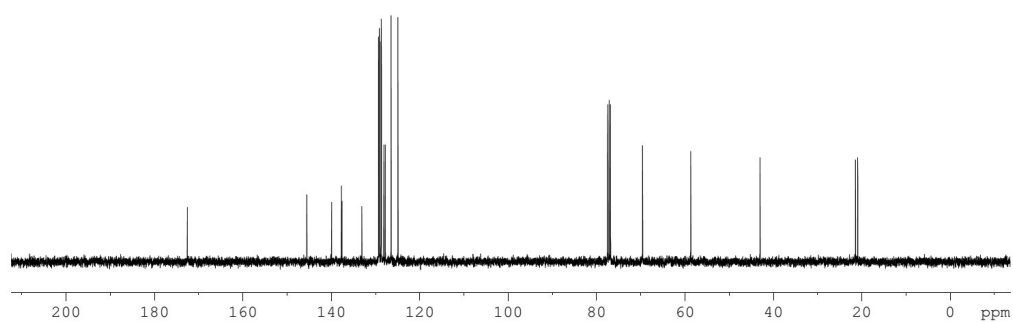
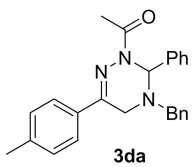
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7.535
7.458
7.441
7.417
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7.380
7.355
7.337
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7.306
7.288
7.280
7.204
7.184
6.449

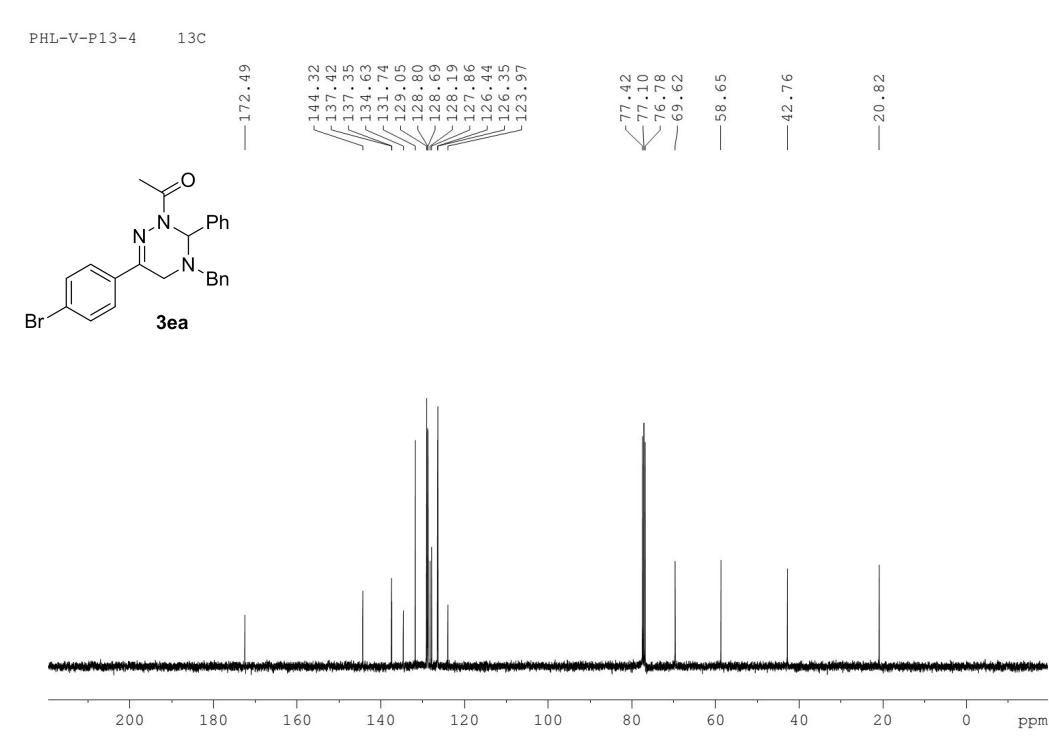
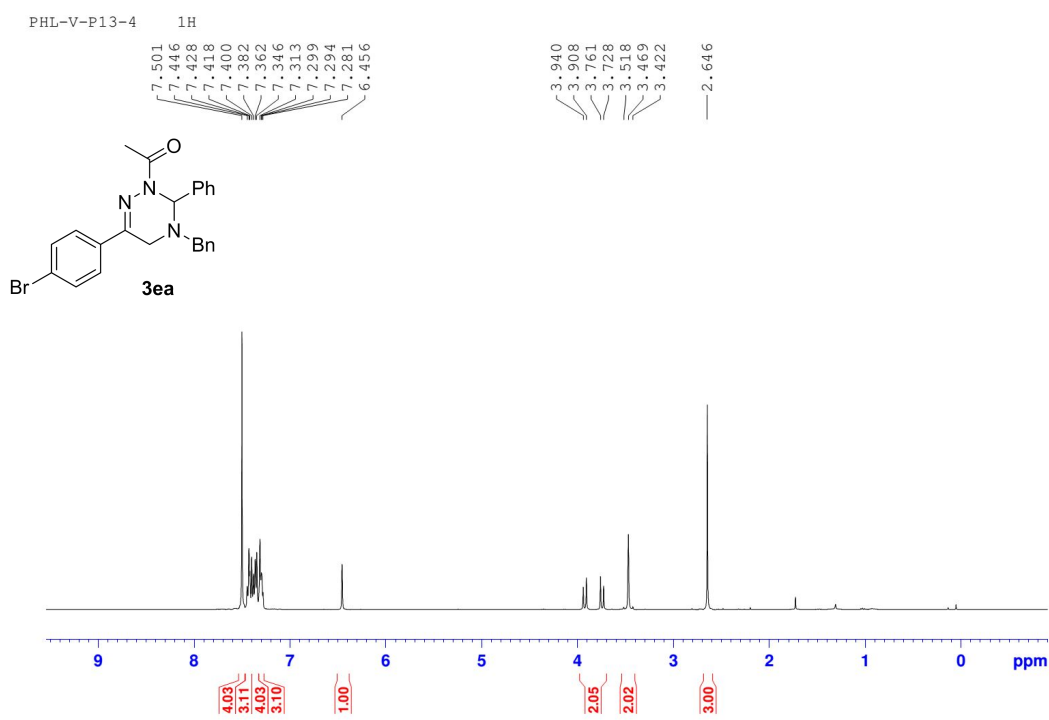
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2.388



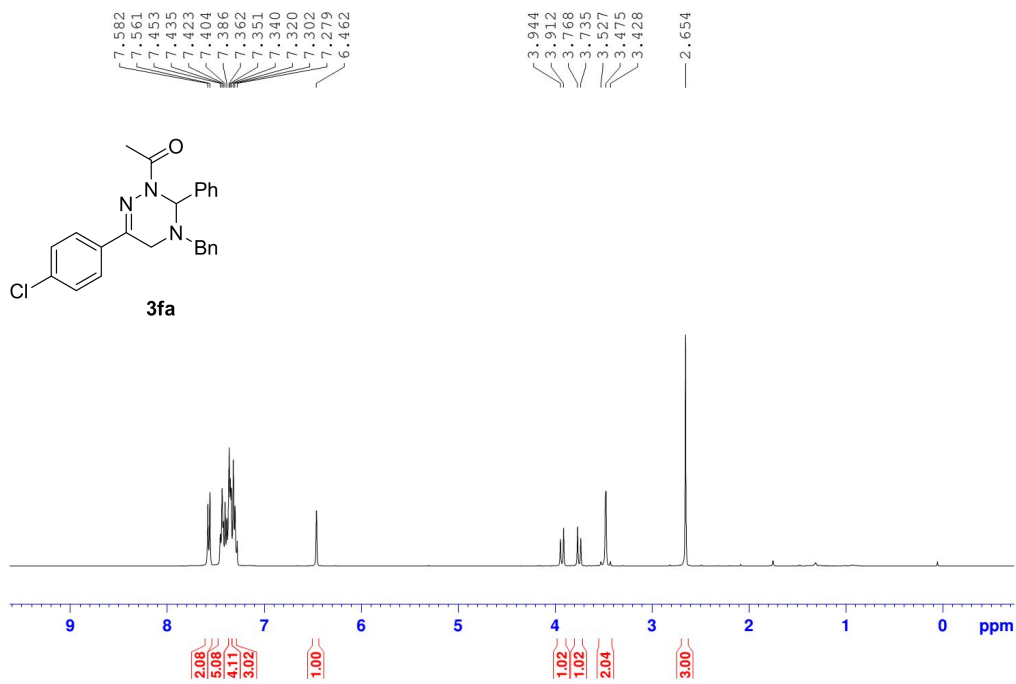
PHL-V-P13-3 13C

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139.90
137.66
137.57
133.07
129.28
129.09
128.74
128.66
128.08
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124.90
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77.12
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21.36
20.85

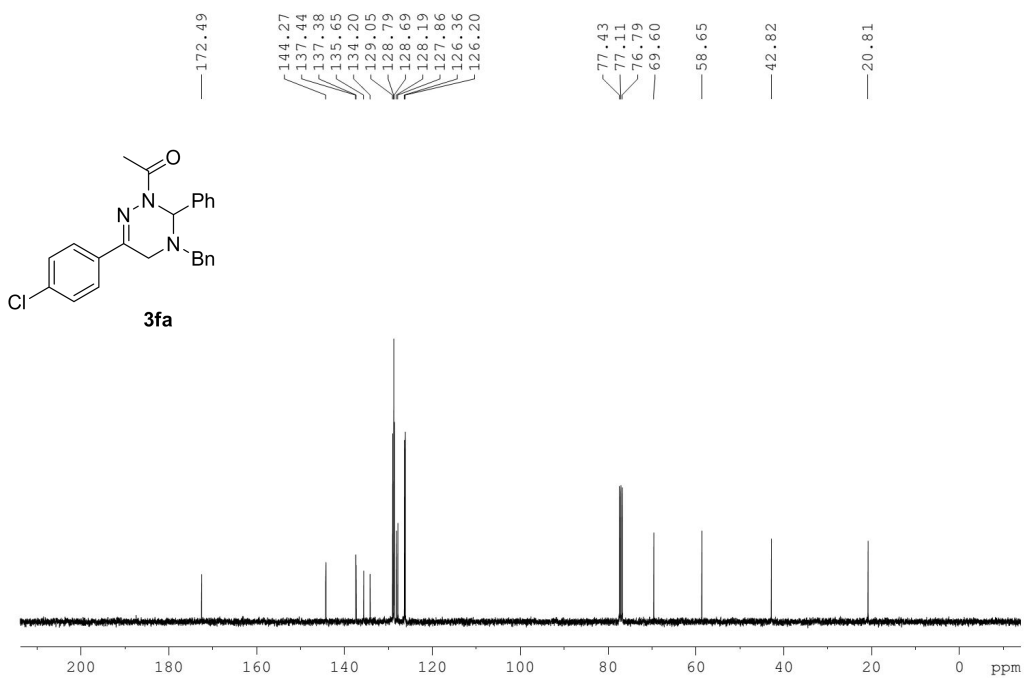




PHL-V-P13-5 NEW 1H



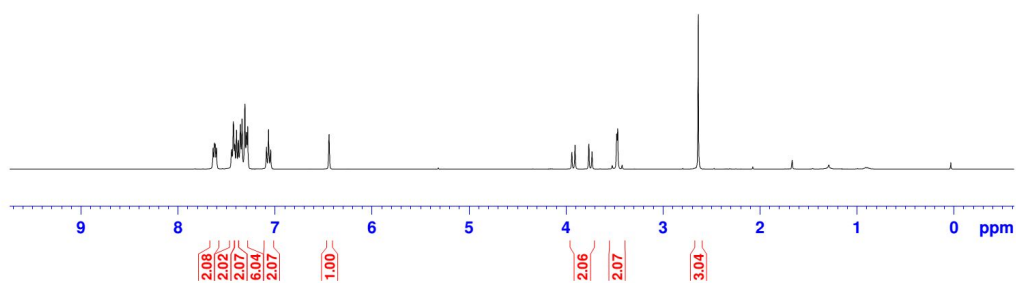
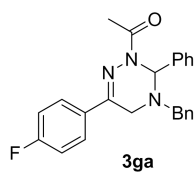
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PHL-V-P13-6 NEW 1H

7.637
7.623
7.615
7.601
7.445
7.428
7.414
7.395
7.376
7.355
7.338
7.309
7.291
7.279
7.086
7.064
7.043
6.441

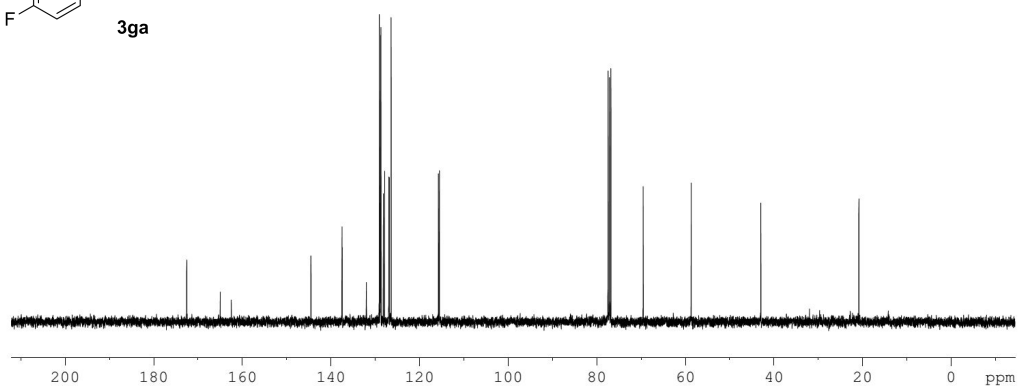
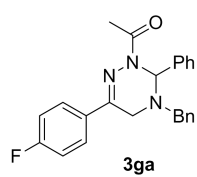
3.937
3.905
3.763
3.730
3.525
3.477
3.468
3.423
— 2.637



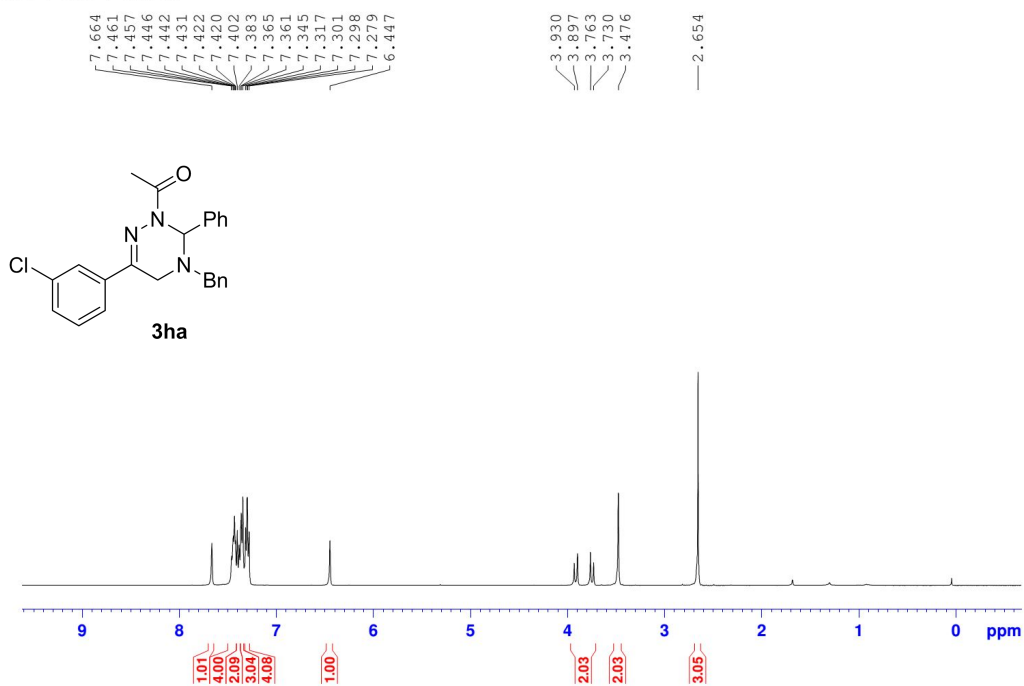
PHL-V-P13-6 13C

172.47
164.90
162.41
144.43
137.50
137.42
131.99
131.96
129.05
128.78
128.69
128.16
127.84
126.90
126.82
126.38
115.71
115.49

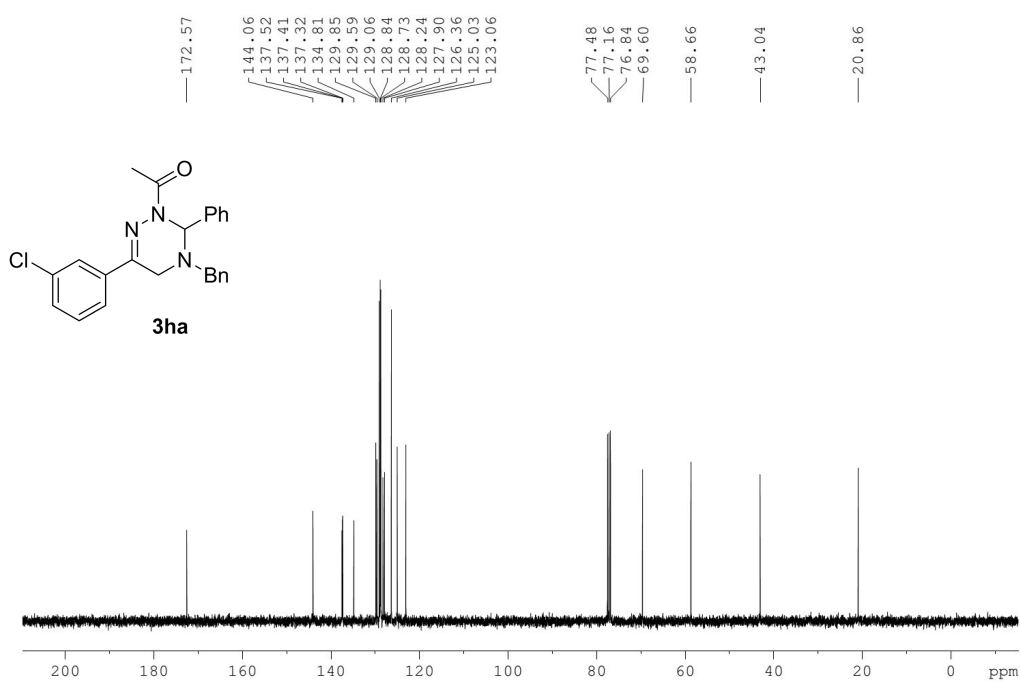
77.41
77.10
76.78
69.50
58.65
42.94
20.80



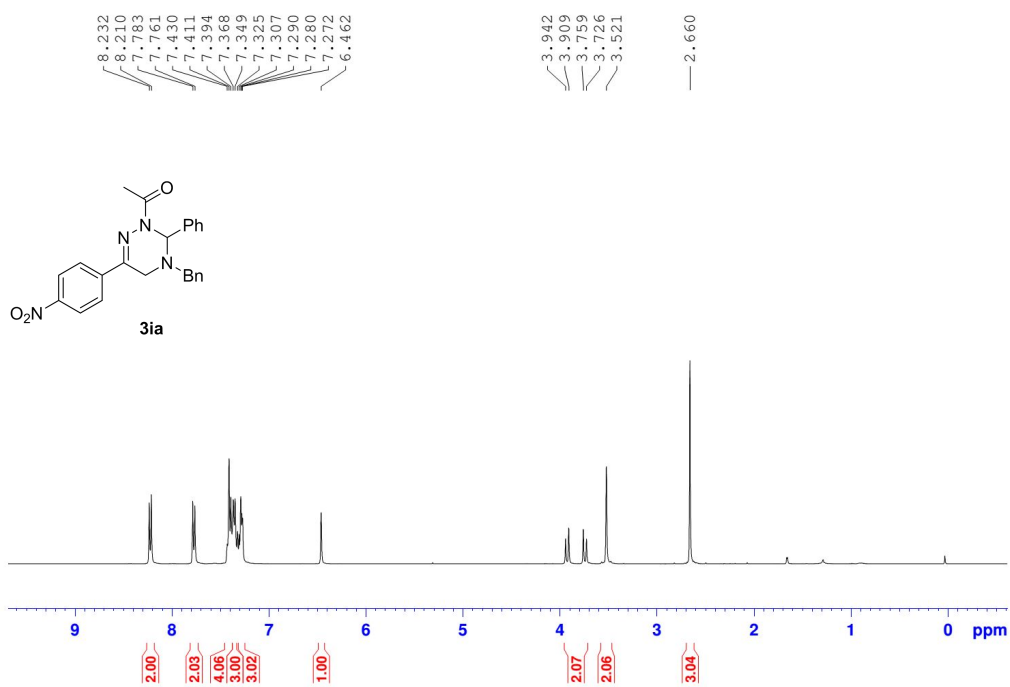
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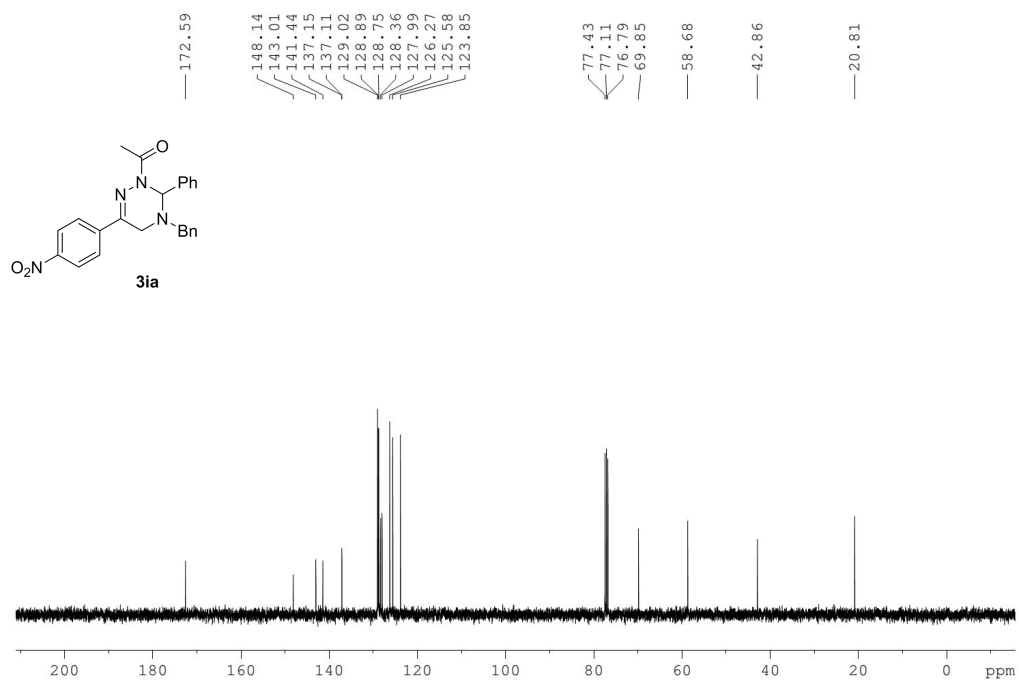
PHL-V-P13-7 13C

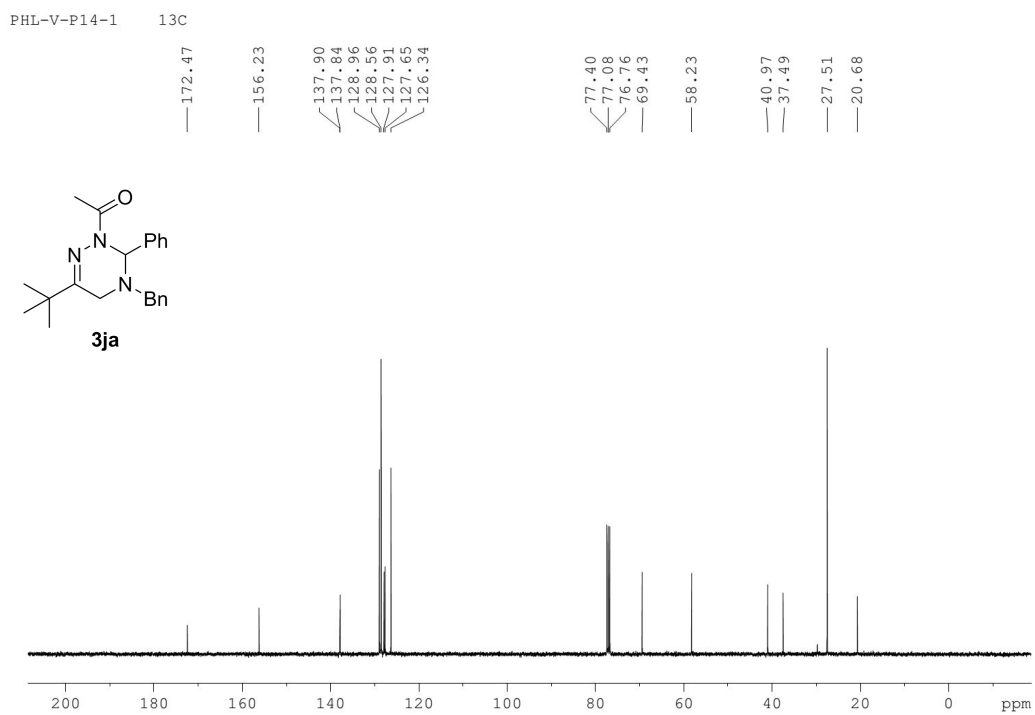
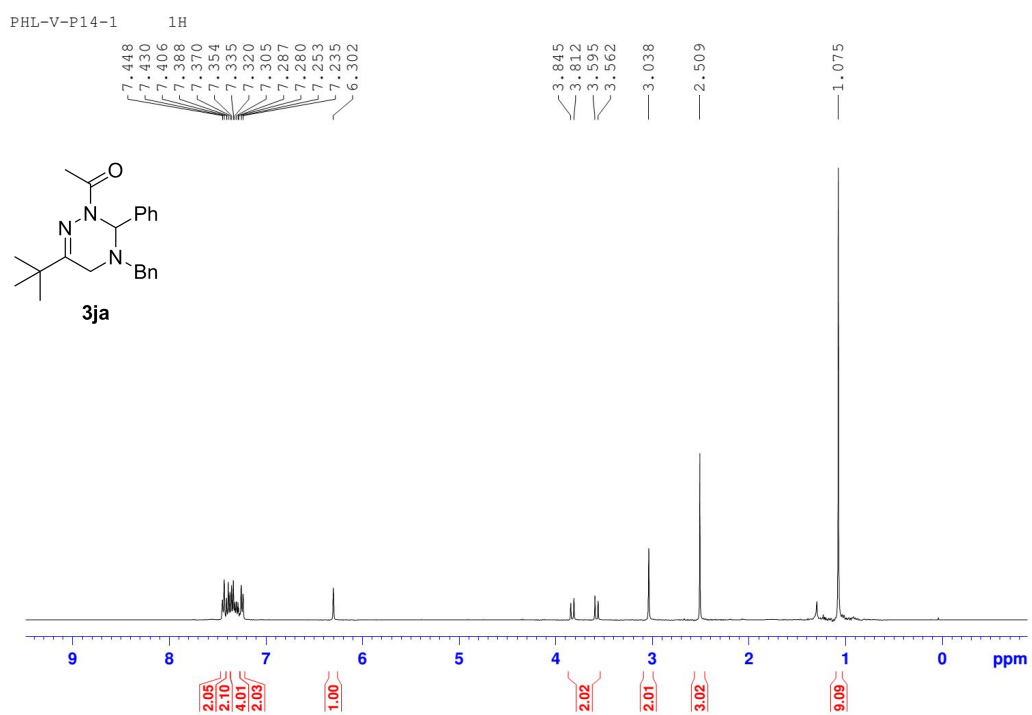


PHL-V-P13-9 NEW 1H

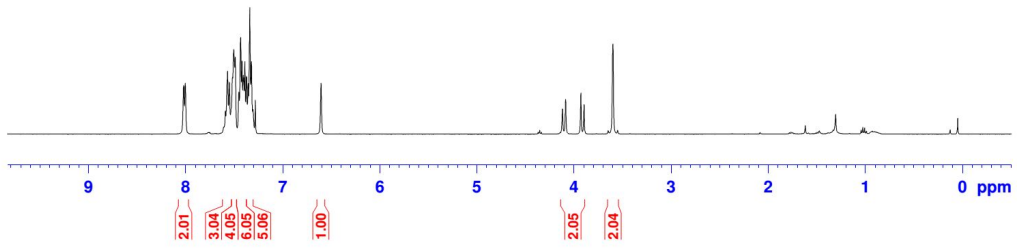
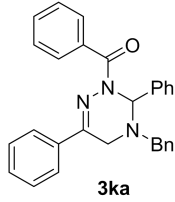
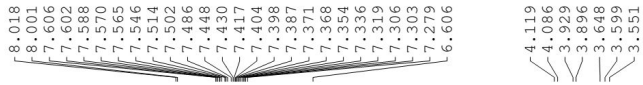


PHL-V-P13-9 13C

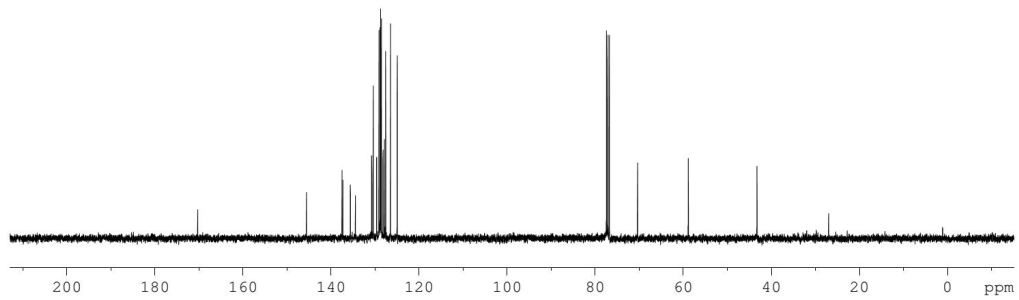
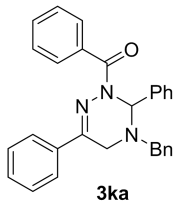
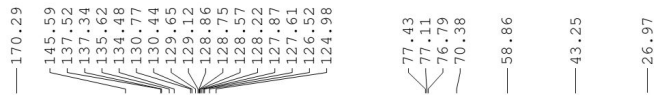




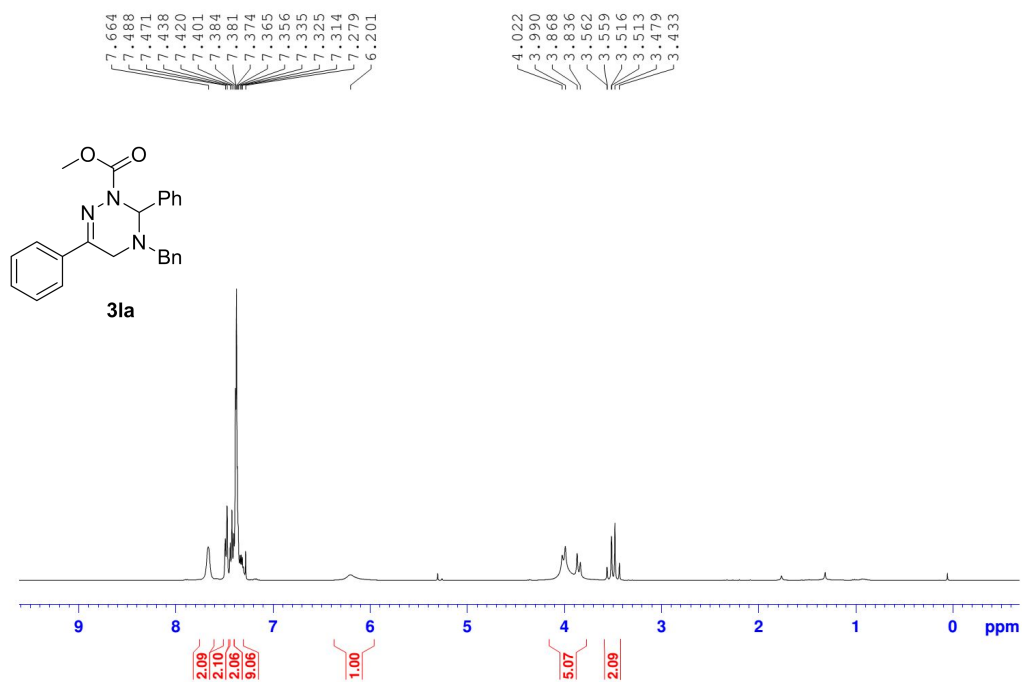
PHL-V-P14-2 NEW 1H



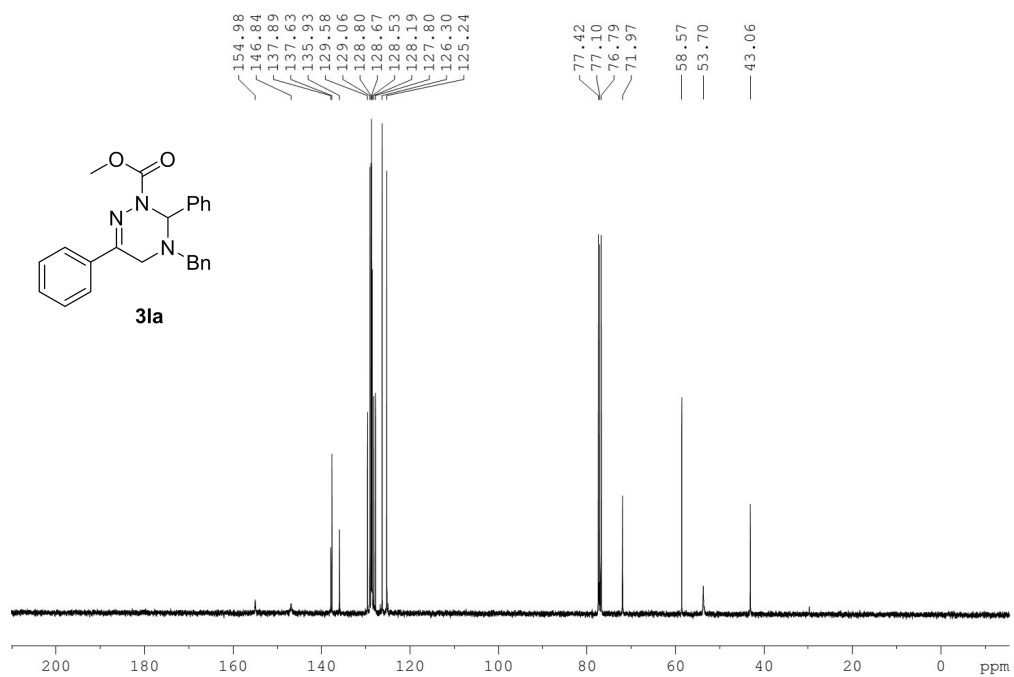
PHL-V-P14-2 13C



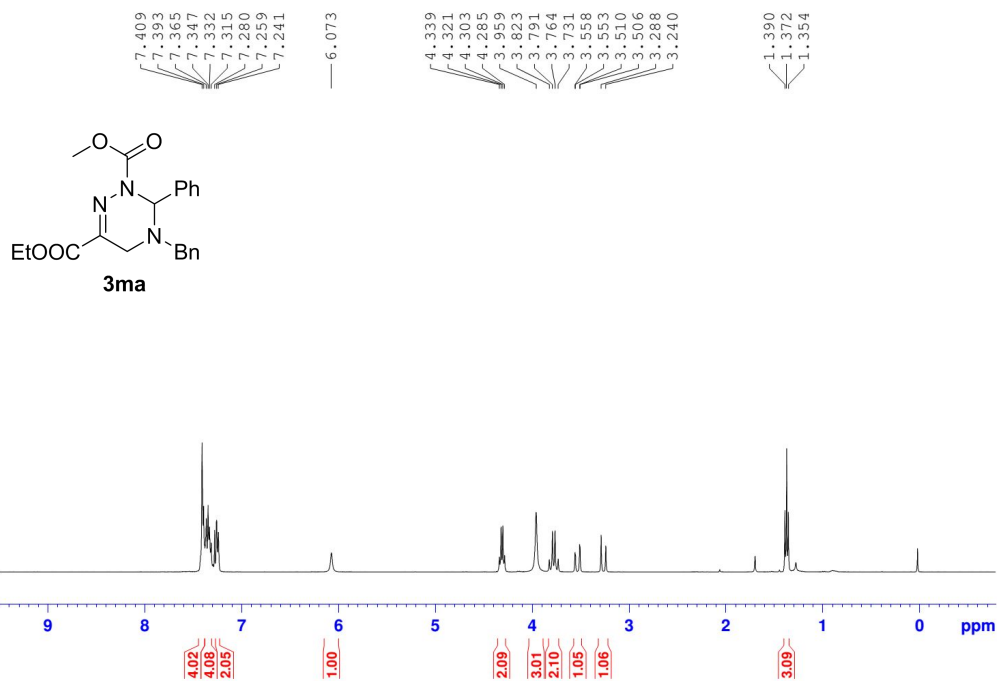
PHL-V-P14-4 NEW 1H



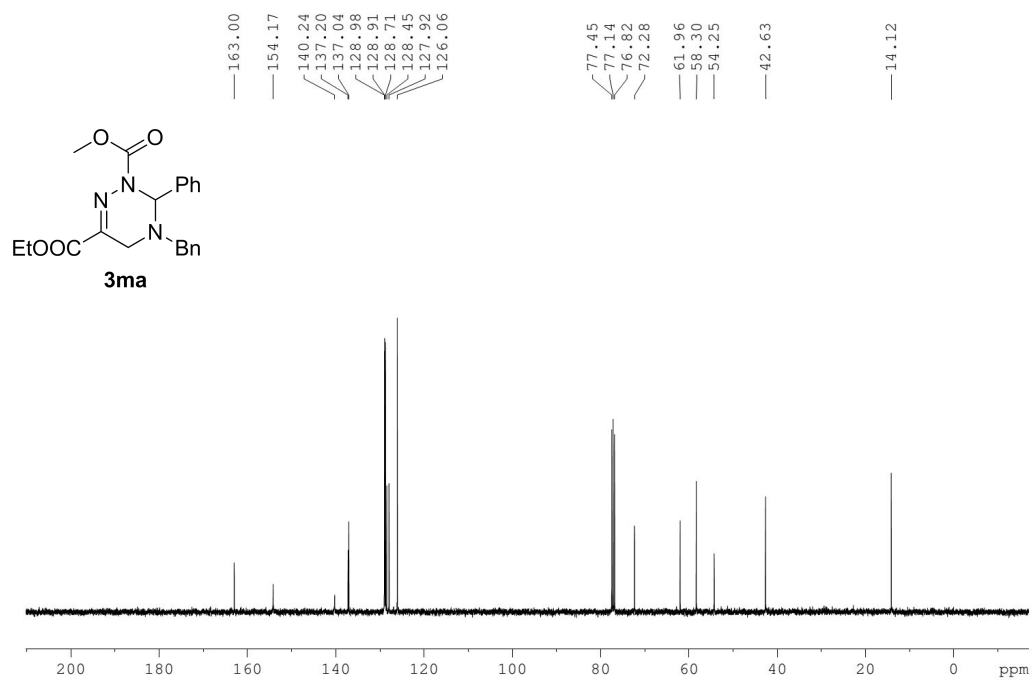
PHL-V-P14-4 NEW 13C



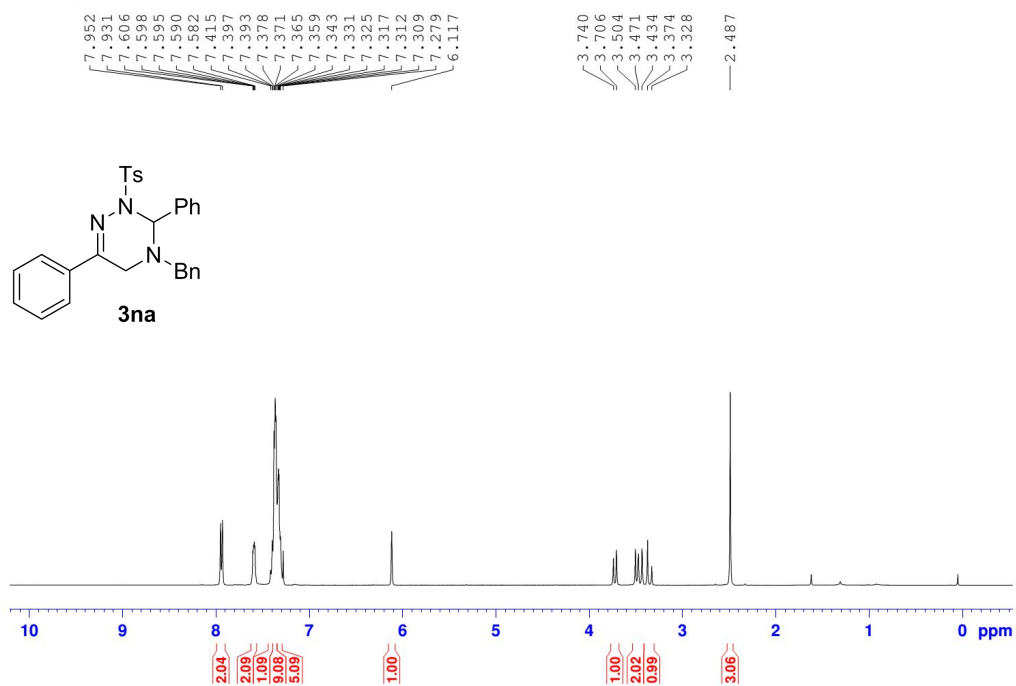
PHL-V-P14-5 NEW 1H



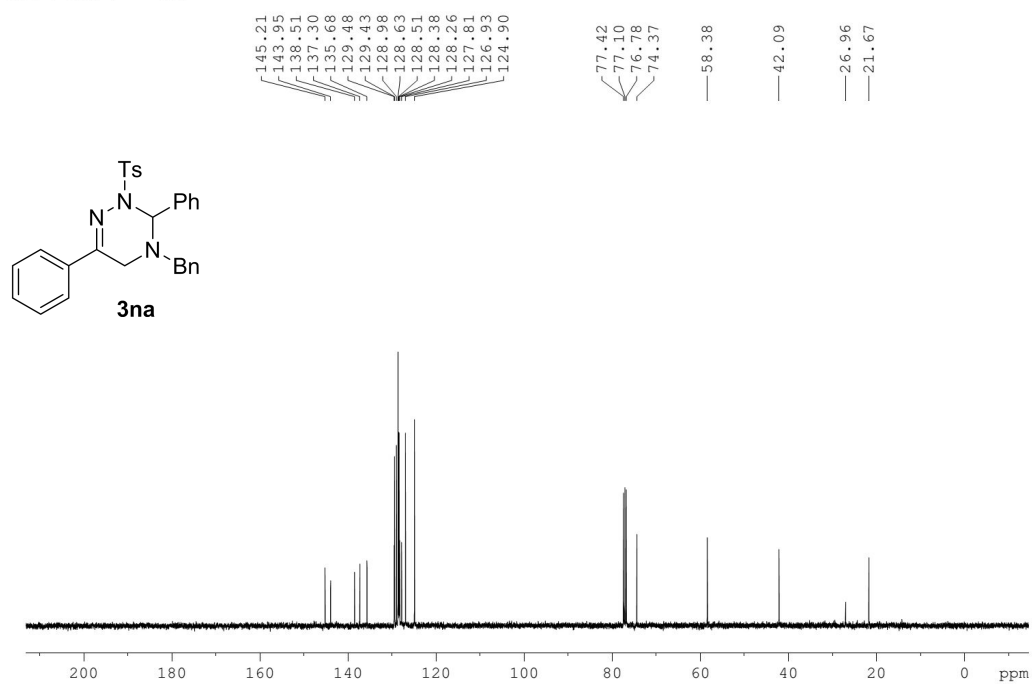
PHL-V-P14-5 13C



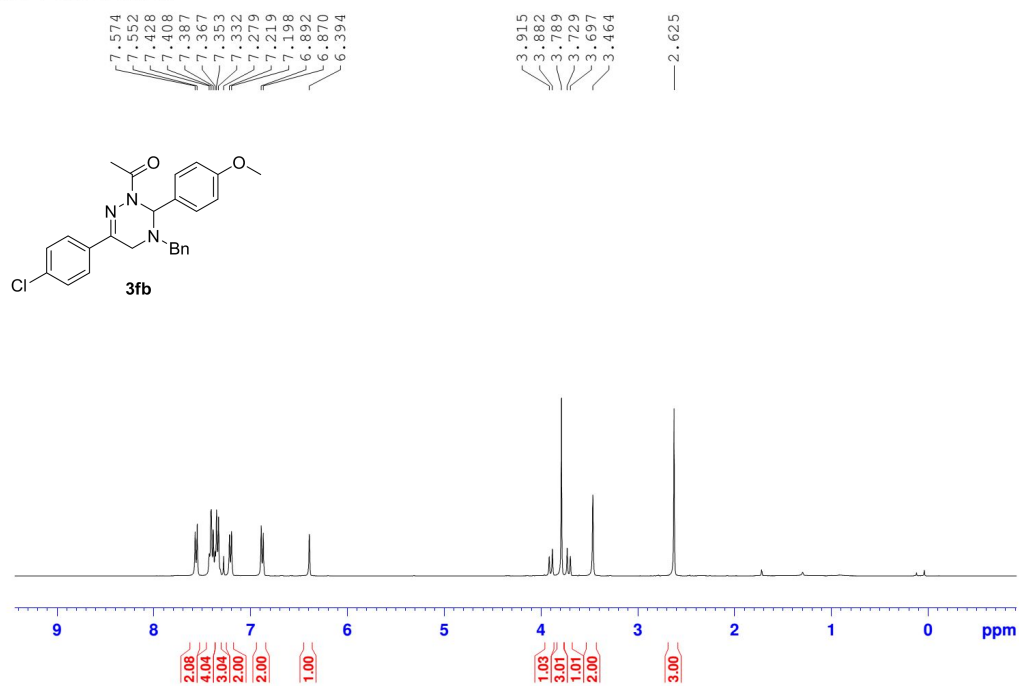
PHL-V-P14-7 NEW 1H



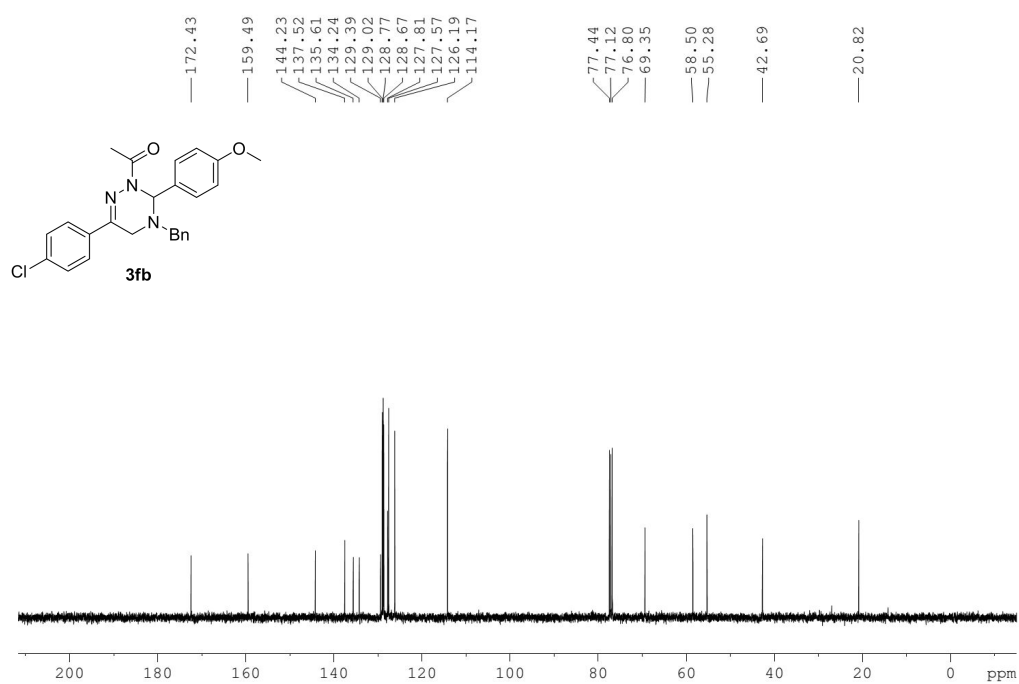
PHL-V-P14-7 13C



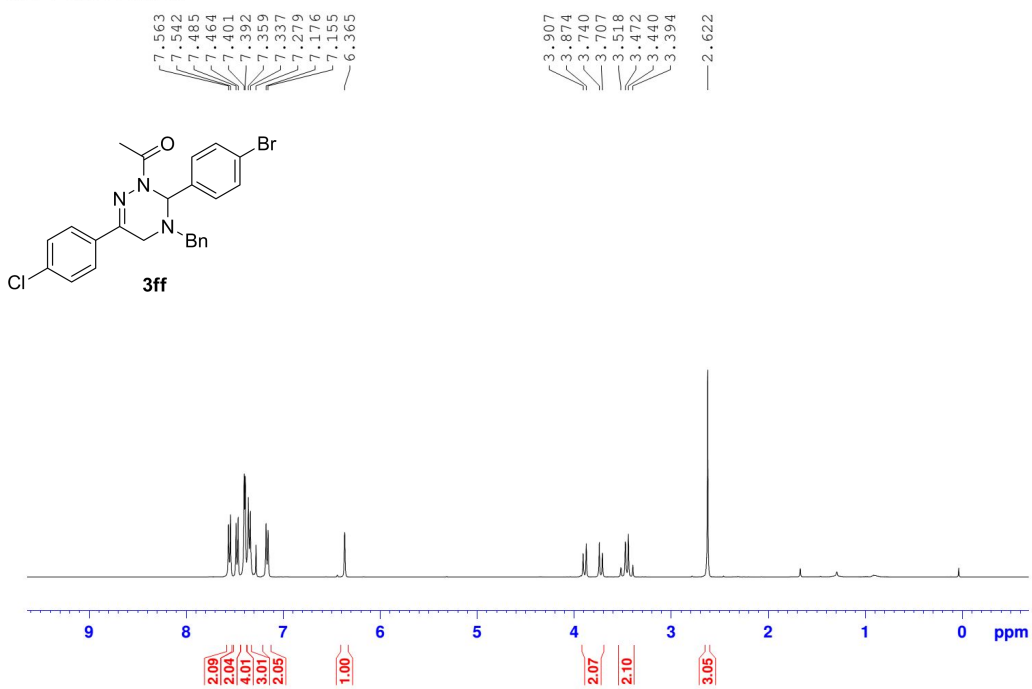
PHL-V-P21-1 NEW 1H



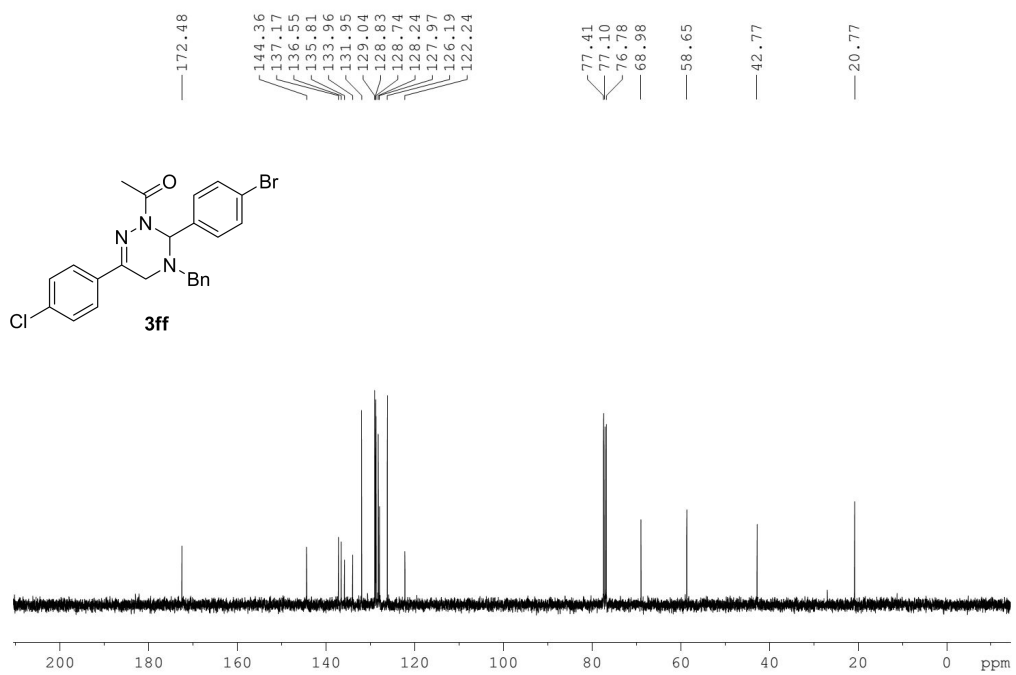
PHL-V-P21-1 13C



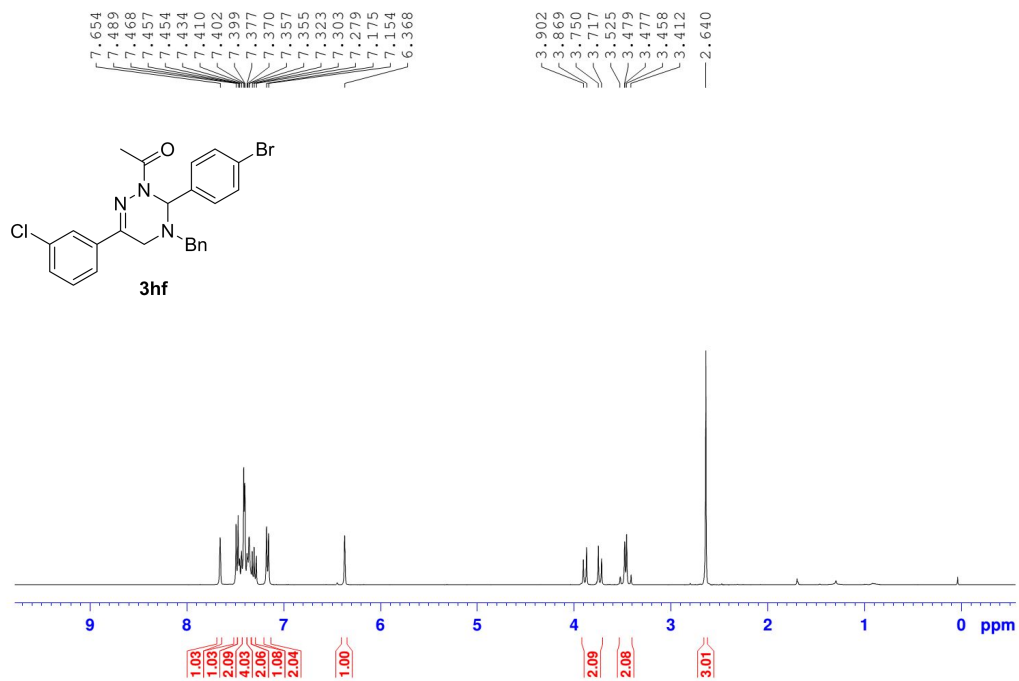
PHL-V-P21-2 NEW 1H



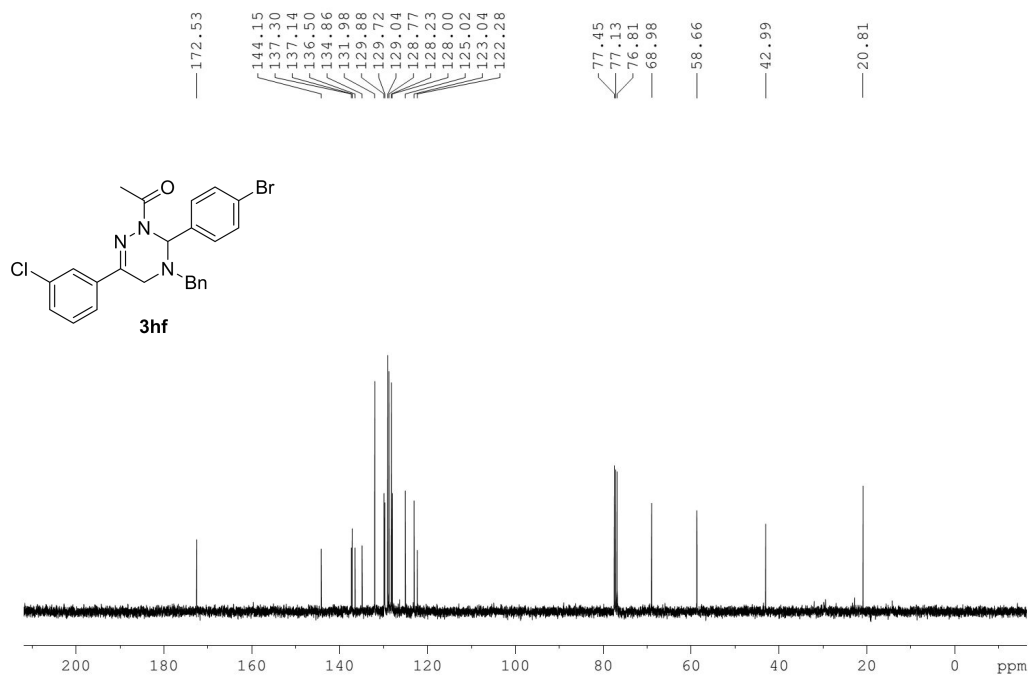
PHL-V-P21-2 13C



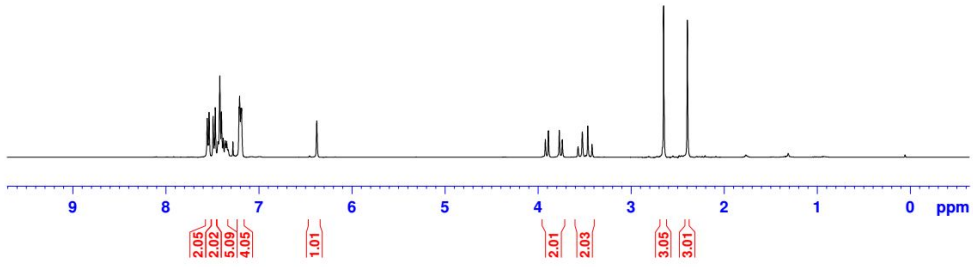
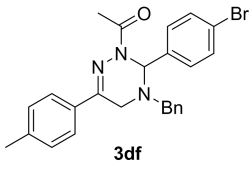
PHL-V-P21-3 NEW 1H



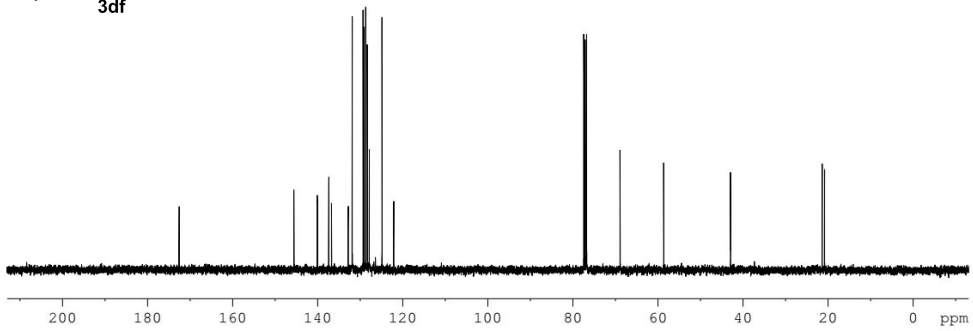
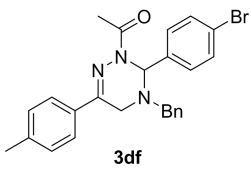
PHL-V-P21-3 13C



PHL-V-P21-4 NEW 1H



PHL-V-P21-4 NEW



2. X-Ray crystal data of compound 3aa

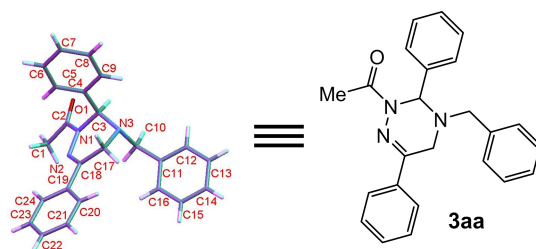


Figure 1. X-ray single crystal structure of 3aa (with thermal ellipsoid shown at the 50% probability level)

Identification code	3aa
Empirical formula	C ₂₄ H ₂₃ N ₃ O
Formula weight	369.45
Temperature	113(2) K
Wavelength	0.71073 Å
Crystal system, space group	Orthorhombic, Pbc _a
Unit cell dimensions	a = 13.3996(18) Å alpha = 90 deg. b = 12.191(2) Å beta = 90 deg. c = 22.958(3) Å gamma = 90 deg.
Volume	3750.3(9) Å 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 > 2σ(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.Å ⁻³