

Supporting Information

Direct Access to Non-Aromatic 1,2,3,6-Tetrahydro -1,2,3,4-Tetrazines via [4+2] Cycloaddition of α -Halogeno Hydrazones with Azodicarboxylic Acid Derivatives

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Wei Meng, Zhao Yang, Yue-Yang Liu, Yu-Di Zhao

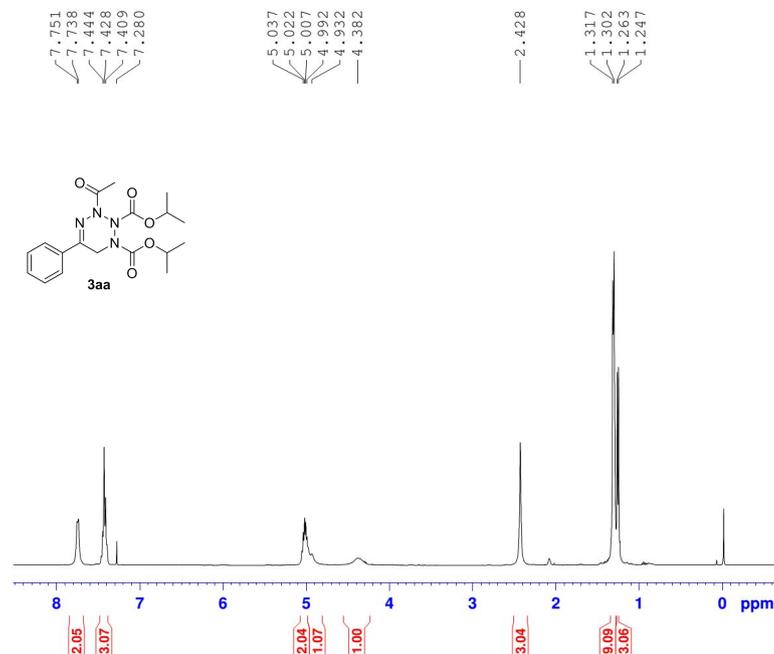
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1. ¹H and ¹³C NMR spectra

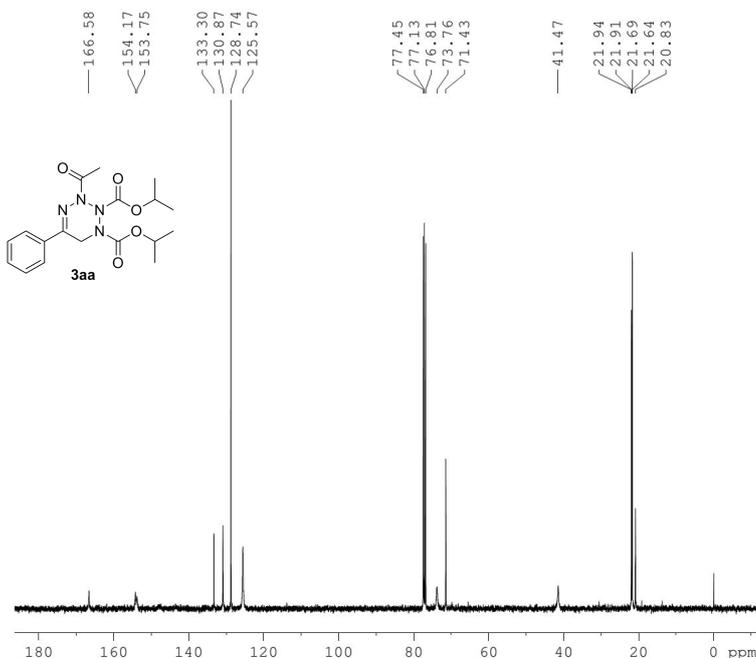
p1l-iii-p18-1



```

NAME          C1
EXPNO         3492
PROCNO        1
Date_         20150923
Time          10.46
INSTRUM       spect
PROBHD        5 mm PABBO BB-
PULPROG       zg30
TD            65536
SOLVENT       CDCl3
NS            16
DS            2
SWH           8223.685 Hz
FIDRES        0.125483 Hz
AQ            3.9846387 sec
RG            71.8
DW            60.800 usec
DE            6.50 usec
TE            673.2 K
D1            1.00000000 sec
TDO           1
===== CHANNEL f1 =====
NUC1          1H
P1            14.50 usec
PL1          -2.83 dB
PL1W         16.91636658 W
SFO1         400.1724712 MHz
SI           32768
SF           400.1700000 MHz
WDW          EM
SSB          0
LB           0.30 Hz
GB           0
PC           1.00
    
```

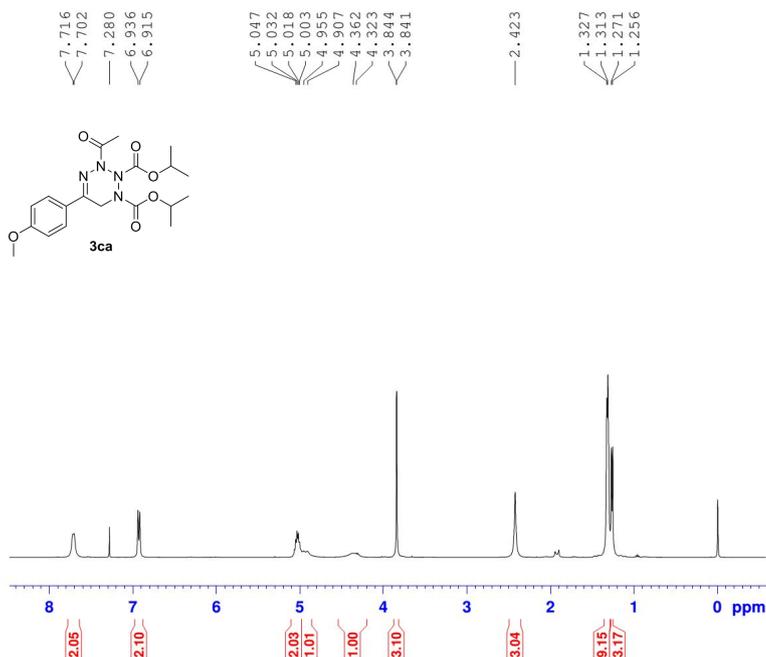
PHL-III-P18-1



```

NAME          C1
EXPNO         3497
PROCNO        1
Date_         20150924
Time          14.10
INSTRUM       spect
PROBHD        5 mm PABBO BB-
PULPROG       zgpg30
TD            65536
SOLVENT       CDCl3
NS            364
DS            4
SWH           24038.461 Hz
FIDRES        0.366798 Hz
AQ            1.3631988 sec
RG            2050
DW            20.800 usec
DE            6.50 usec
TE            673.2 K
D1            2.00000000 sec
D11           0.83000000 sec
TDO           1
===== CHANNEL f1 =====
NUC1          13C
P1            11.70 usec
PL1          -2.00 dB
PL1W         48.76612744 W
SFO1         100.6328888 MHz
===== CHANNEL f2 =====
CPDPRG2       waltz16
NUC2          1H
PCPD2         80.00 usec
PL2          -2.83 dB
PL12         12.00 dB
PL13         14.00 dB
PL2W         16.91636658 W
PL12W        0.55629748 W
PL13W        0.35100001 W
SFO2         400.1716007 MHz
SI           32768
SF           100.6228270 MHz
WDW          EM
SSB          0
LB           1.00 Hz
GB           0
PC           1.40
    
```

PHL-III-P31-1



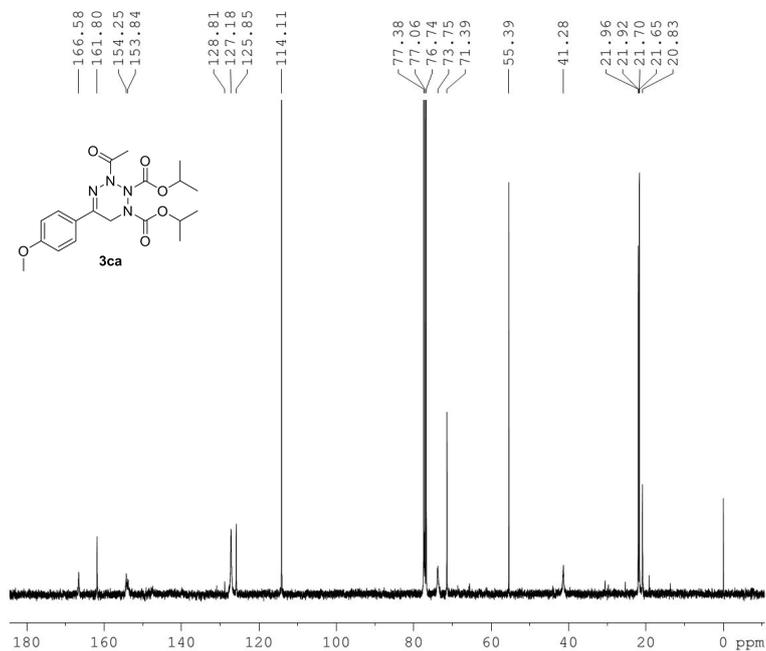
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NAME          C1
EXPNO         3425
PROCNO        1
Date_         20150908
Time          9.04
INSTRUM       spect
PROBHD        5 mm PABBO BB-
PULPROG       zg30
TD            65536
SOLVENT       CDCl3
NS            9
DS            2
SWH           8223.685 Hz
FIDRES        0.125483 Hz
AQ            3.9846387 sec
RG            101
DW            60.800 usec
DE            6.50 usec
TE            673.2 K
D1            1.00000000 sec
TDO           1
  
```

```

===== CHANNEL f1 =====
NUC1          1H
P1            14.50 usec
PL1           -2.83 dB
PL1W         16.91636658 W
SFO1         400.1724712 MHz
SI           32768
SF           400.1700000 MHz
WDW           EM
SSB           0
LB           0.30 Hz
GB            0
PC            1.00
  
```

PHL-III-P31-1 13C



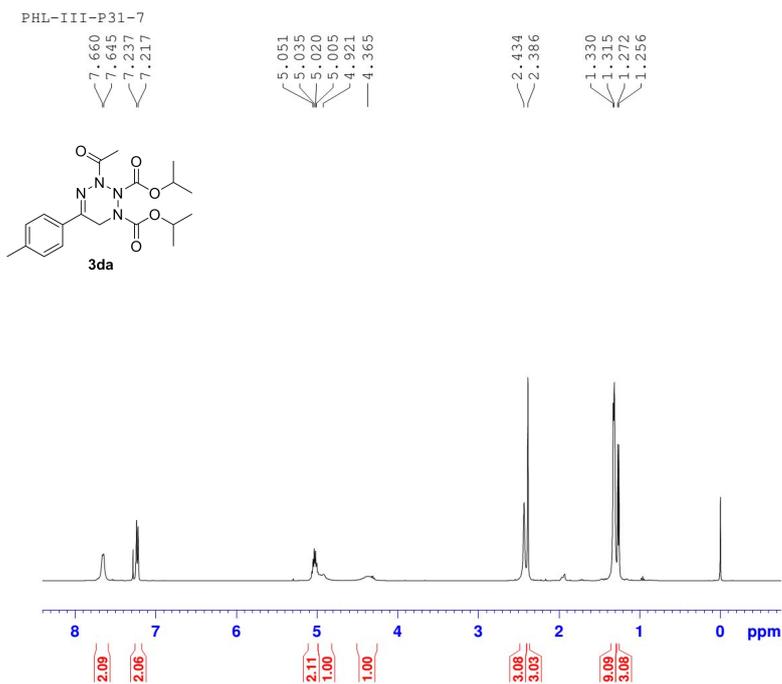
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NAME          C1
EXPNO         3429
PROCNO        1
Date_         20150908
Time          15.26
INSTRUM       spect
PROBHD        5 mm PABBO BB-
PULPROG       zgpg30
TD            65536
SOLVENT       CDCl3
NS            943
DS            4
SWH           24038.461 Hz
FIDRES        0.366798 Hz
AQ            1.3631988 sec
RG            2950
DW            20.800 usec
DE            6.50 usec
TE            673.2 K
D1            2.00000000 sec
D11           0.03000000 sec
TDO           1
  
```

```

===== CHANNEL f1 =====
NUC1          13C
P1            11.70 usec
PL1           -2.00 dB
PL1W         48.76812744 W
SFO1         100.6328888 MHz

===== CHANNEL f2 =====
CPDPRG2       waltz16
NUC2          1H
PCPD2         80.00 usec
PL2           -2.83 dB
PL12          12.00 dB
PL13          14.00 dB
PL2W         16.91636658 W
PL12W         0.55629748 W
PL13W         0.35100001 W
SFO2         400.1716007 MHz
SI           32768
SF           100.6228270 MHz
WDW           EM
SSB           0
LB            1.00 Hz
GB            0
PC            1.40
  
```



```

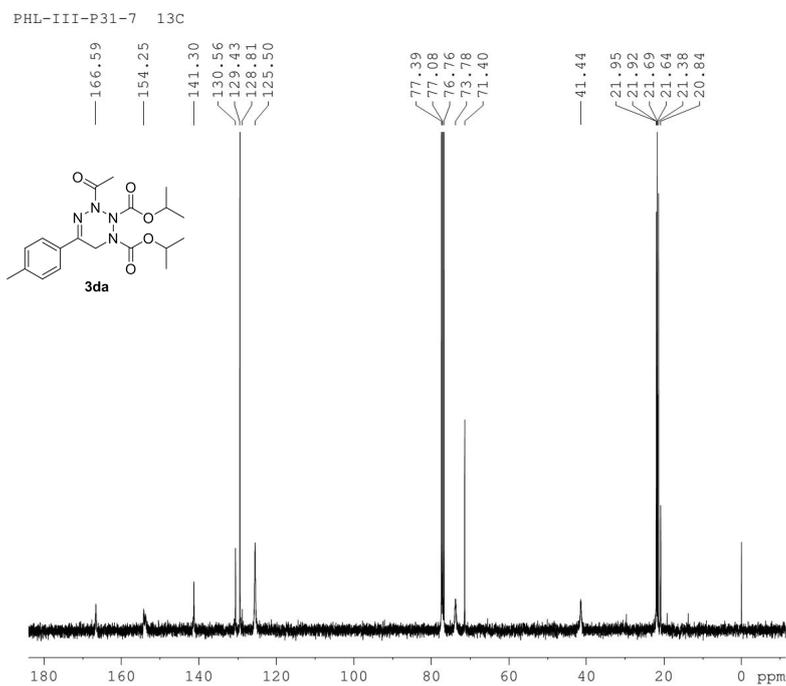
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EXPNO         3428
PROCNO        1
Date_         20150908
Time          9.23
INSTRUM       spect
PROBHD        5 mm PABBO BB-
PULPROG       zg30
TD            65536
SOLVENT       CDCl3
NS            16
DS            2
SWH           8223.685 Hz
FIDRES        0.125483 Hz
AQ            3.9846387 sec
RG            101
DW            60.800 usec
DE            6.50 usec
TE            673.2 K
D1            1.00000000 sec
TDO           1

```

```

===== CHANNEL f1 =====
NUC1          1H
P1            14.50 usec
PL1           -2.83 dB
PL1W          16.91636658 W
SF01          400.1724712 MHz
SI            32768
SF            400.1700000 MHz
WDW           EM
SSB           0
LB            0.30 Hz
GB            0
PC            1.00

```



```

NAME          C1
EXPNO         3432
PROCNO        1
Date_         20150909
Time          16.20
INSTRUM       spect
PROBHD        5 mm PABBO BB-
PULPROG       zgpg30
TD            65536
SOLVENT       CDCl3
NS            401
DS            4
SWH           24038.461 Hz
FIDRES        0.366798 Hz
AQ            1.3631988 sec
RG            2050
DW            20.800 usec
DE            6.50 usec
TE            673.2 K
D1            2.00000000 sec
D11           0.83000000 sec
TDO           1

```

```

===== CHANNEL f1 =====
NUC1          13C
P1            11.70 usec
PL1           -2.00 dB
PL1W          48.76612744 W
SF01          100.6328888 MHz

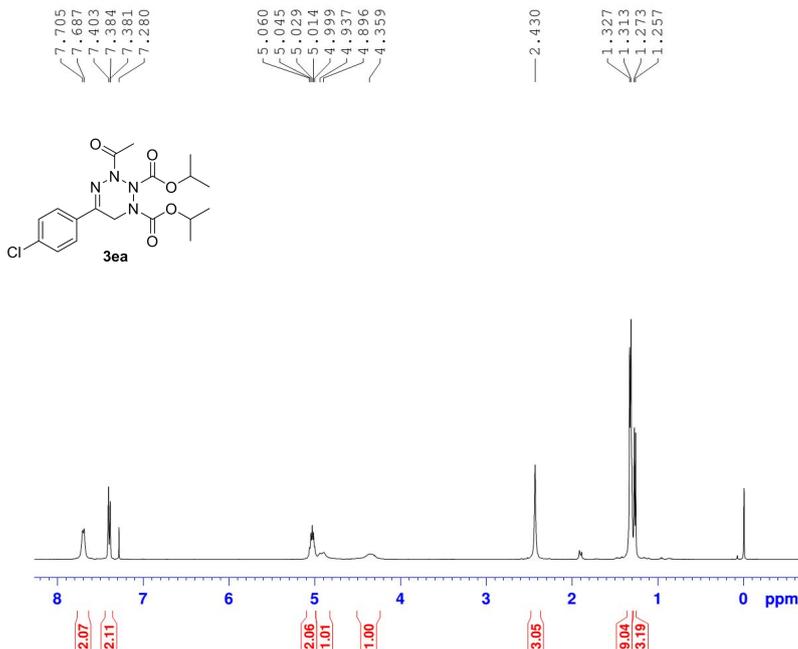
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```

===== CHANNEL f2 =====
CPDPRG2       waltz16
NUC2          1H
PCPD2         80.00 usec
PL2           -2.83 dB
PL12          12.00 dB
PL13          14.00 dB
PL2W          16.91636658 W
PL12W         0.55629748 W
PL13W         0.35100001 W
SF02          400.1716007 MHz
SI            32768
SF            100.6228270 MHz
WDW           EM
SSB           0
LB            1.00 Hz
GB            0
PC            1.40

```

PHL-III-P31-2



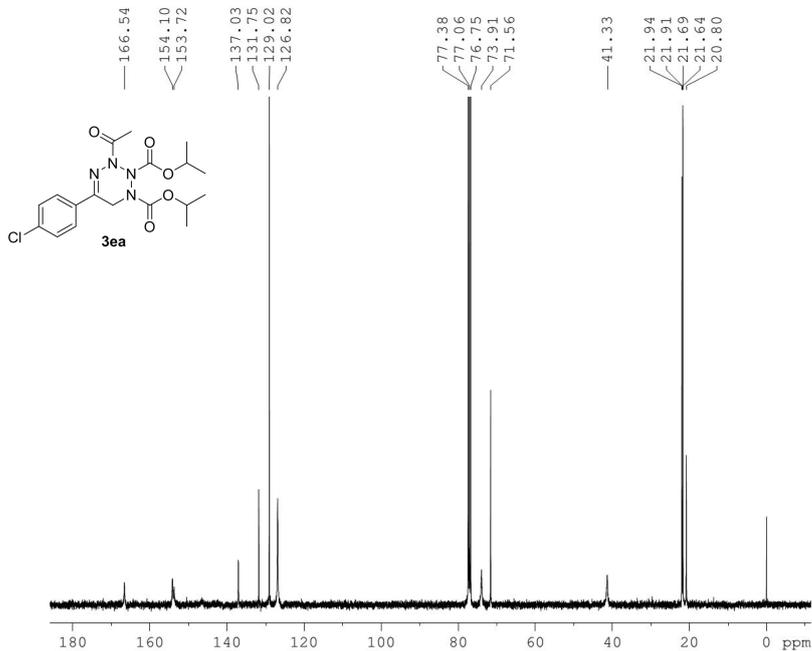
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NAME          C1
EXPNO         3426
PROCNO        1
Date_         20150908
Time          9.11
INSTRUM       spect
PROBHD        5 mm PABBO BB-
PULPROG       zg30
TD            65536
SOLVENT       CDCl3
NS            16
DS            4
SWH           8223.685 Hz
FIDRES        0.125483 Hz
AQ            3.9846387 sec
RG            101
DW            60.800 usec
DE            6.50 usec
TE            673.2 K
D1            1.00000000 sec
TD0           1
  
```

```

===== CHANNEL f1 =====
NUC1          1H
P1            14.50 usec
PL1           -2.83 dB
PL1W          16.91636658 W
SF01          400.1724712 MHz
SI            32768
SF            400.1700000 MHz
WDW           EM
SSB           0
LB            0.30 Hz
GB            0
PC            1.00
  
```

PHL-III-P31-2 13C



```

NAME          C1
EXPNO         3433
PROCNO        1
Date_         20150909
Time          20.56
INSTRUM       spect
PROBHD        5 mm PABBO BB-
PULPROG       zgpg30
TD            65536
SOLVENT       CDCl3
NS            1024
DS            4
SWH           24038.461 Hz
FIDRES        0.366798 Hz
AQ            1.3631988 sec
RG            2050
DW            20.800 usec
DE            6.50 usec
TE            673.2 K
D1            2.00000000 sec
D11           0.03000000 sec
TD0           1
  
```

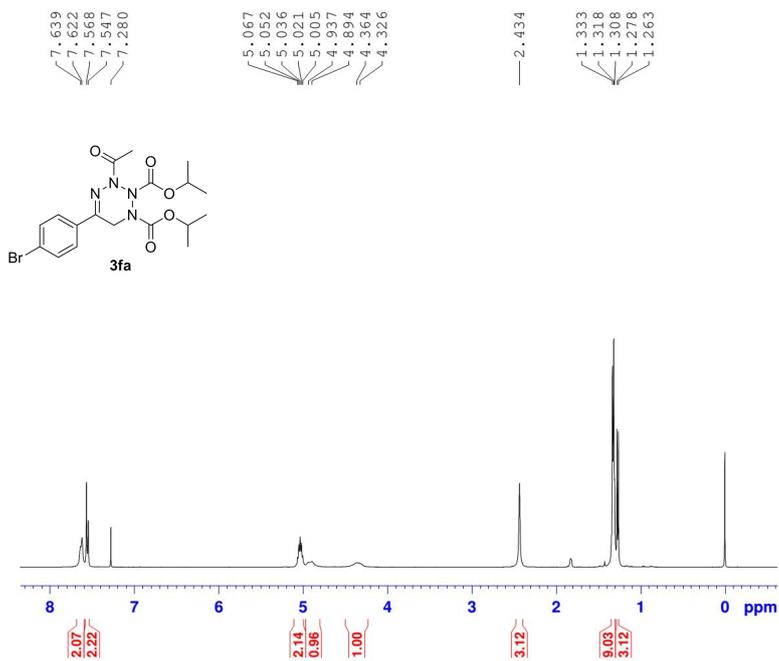
```

===== CHANNEL f1 =====
NUC1          13C
P1            11.70 usec
PL1           -2.00 dB
PL1W          48.76812744 W
SF01          100.6328888 MHz
  
```

```

===== CHANNEL f2 =====
CPDPRG2       waltz16
NUC2          1H
PCPD2         80.00 usec
PL2           -2.83 dB
PL12          12.00 dB
PL13          14.00 dB
PL1W          16.91636658 W
PL12W         0.55629748 W
PL13W         0.35100001 W
SF02          400.1716007 MHz
SI            32768
SF            100.6228270 MHz
WDW           EM
SSB           0
LB            1.00 Hz
GB            0
PC            1.40
  
```

PHL-III-P31-3



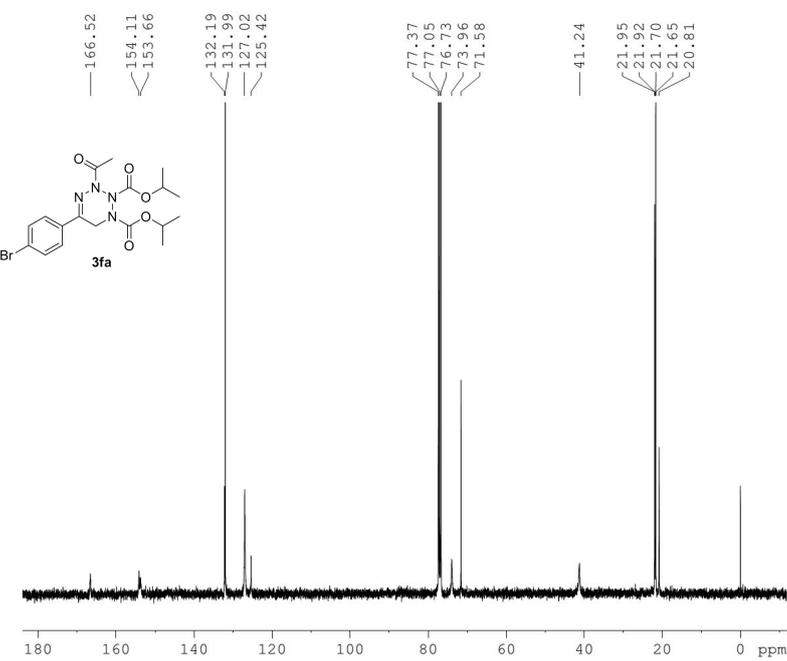
```

NAME          C1
EXPNO         3430
PROCNO        1
Date_         20150909
Time          16.08
INSTRUM       spect
PROBHD        5 mm PABBO BB-
PULPROG       zgpg30
TD            65536
SOLVENT       CDCl3
NS            4
DS            2
SWH           8223.685 Hz
FIDRES        0.125483 Hz
AQ            3.9846387 sec
RG            114
DW            60.800 usec
DE            6.50 usec
TE            673.2 K
D1            1.00000000 sec
D11           1
TD0           1
  
```

```

===== CHANNEL f1 =====
NUC1          1H
P1            14.50 usec
PL1           -2.83 dB
PL1W         16.91636658 W
SF01         400.1724712 MHz
SI           32768
SF           400.1700000 MHz
WDW           EM
SSB           0
LB            0.30 Hz
GB            0
PC            1.00
  
```

PHL-III-P31-3 13C



```

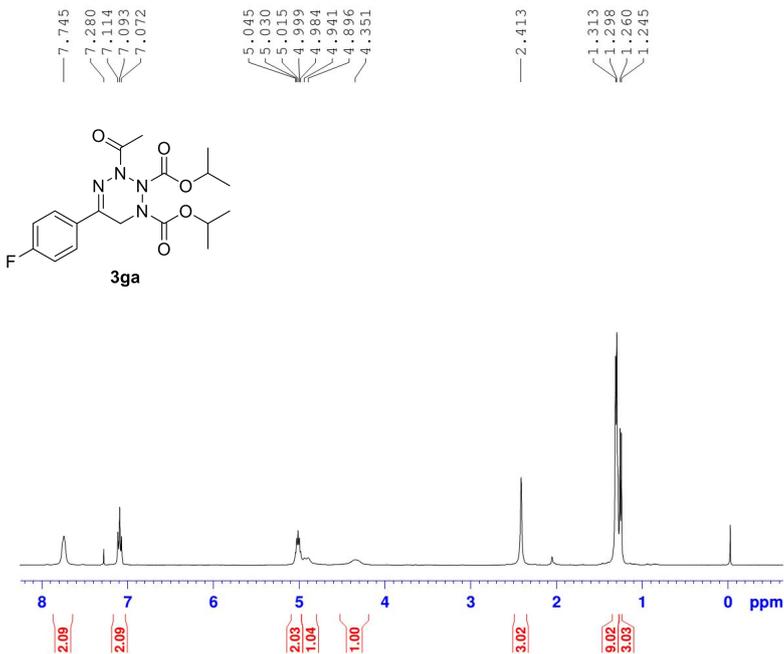
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EXPNO         3450
PROCNO        1
Date_         20150910
Time          15.28
INSTRUM       spect
PROBHD        5 mm PABBO BB-
PULPROG       zgpg30
TD            65536
SOLVENT       CDCl3
NS            1024
DS            4
SWH           24038.461 Hz
FIDRES        0.366798 Hz
AQ            1.3631988 sec
RG            2050
DW            20.800 usec
DE            6.50 usec
TE            673.2 K
D1            2.00000000 sec
D11           0.03000000 sec
TD0           1
  
```

```

===== CHANNEL f1 =====
NUC1          13C
P1            11.70 usec
PL1           -2.00 dB
PL1W         48.76812744 W
SF01         100.6328888 MHz

===== CHANNEL f2 =====
CPDPRG2       waltz16
NUC2          1H
PCPD2         80.00 usec
PL2           -2.83 dB
PL12         12.00 dB
PL13         14.00 dB
PL2W         16.91636658 W
PL12W        0.55629748 W
PL13W        0.35100001 W
SF02         400.1716007 MHz
SI           32768
SF           100.6228270 MHz
WDW           EM
SSB           0
LB            1.00 Hz
GB            0
PC            1.40
  
```

PHL-III-P31-6



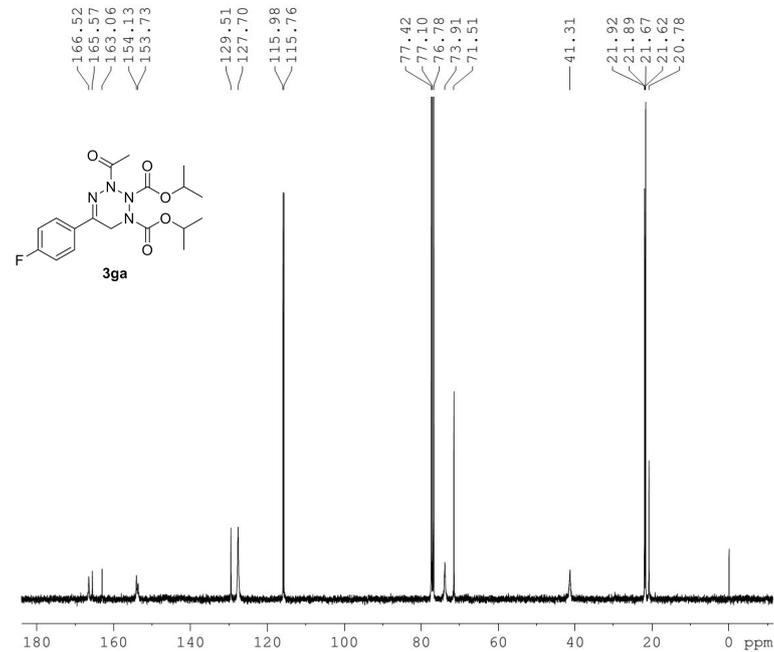
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NAME          C1
EXPNO         3464
PROCNO        1
Date_         20150915
Time          9.29
INSTRUM       spect
PROBHD        5 mm FABBO BB-
PULPROG       zg30
TD            65536
SOLVENT       CDCl3
NS            16
DS            2
SWH           8223.685 Hz
FIDRES        0.125483 Hz
AQ            3.9846387 sec
RG            101
DW            60.800 usec
DE            6.50 usec
TE            673.2 K
D1            1.00000000 sec
TD0           1
  
```

```

===== CHANNEL f1 =====
NUC1          1H
P1            14.50 usec
PL1           -2.83 dB
PL1W          16.91636658 W
SFO1          400.1724713 MHz
SI            32768
SF            400.1700000 MHz
WDW           EM
SSB           0
LB            0.30 Hz
GB            0
PC            1.00
  
```

PHL-III-P31-6 13c



```

NAME          C1
EXPNO         3473
PROCNO        1
Date_         20150916
Time          15.41
INSTRUM       spect
PROBHD        5 mm FABBO BB-
PULPROG       zgpg30
TD            65536
SOLVENT       CDCl3
NS            539
DS            4
SWH           24038.461 Hz
FIDRES        0.366798 Hz
AQ            1.3631988 sec
RG            2050
DW            20.800 usec
DE            6.50 usec
TE            298.8 K
D1            2.00000000 sec
D11           0.03000000 sec
TD0           1
  
```

```

===== CHANNEL f1 =====
NUC1          13C
P1            11.70 usec
PL1           -2.00 dB
PL1W          48.76812744 W
SFO1          100.6328888 MHz
  
```

```

===== CHANNEL f2 =====
CPDPRG2       waltz16
NUC2          1H
PCPD2         80.00 usec
PL2           -2.83 dB
PL12          12.00 dB
PL13          14.00 dB
PL2W          16.91636658 W
PL12W         0.55629748 W
PL13W         0.35100001 W
SFO2          400.1716007 MHz
SI            32768
SF            100.6228270 MHz
WDW           EM
SSB           0
LB            1.00 Hz
GB            0
PC            1.40
  
```

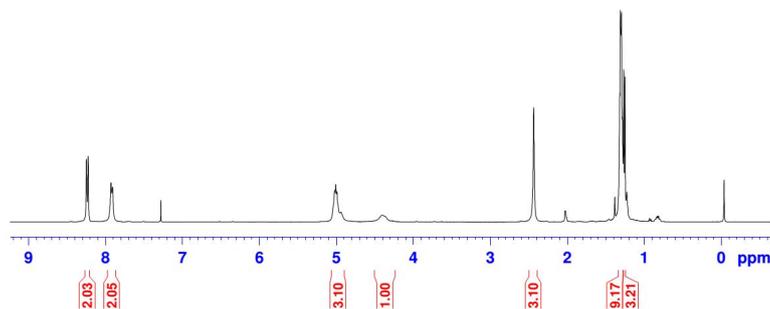
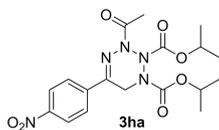
PHL-III-P31-4

8.244
8.224
7.927
7.907
7.280

5.024
5.009
4.994
4.942
4.406

2.436

1.309
1.304
1.296
1.266
1.250



```

NAME          C1
EXPNO         3455
PROCNO        1
Date_         20150914
Time          9.54
INSTRUM       spect
PROBHD        5 mm PABBO BB-
PULPROG       zg30
ID            65536
SOLVENT       CDCl3
NS            9
DS            2
SWH           8223.685 Hz
FIDRES        0.125483 Hz
AQ            3.9846387 sec
RG            4
DW            60.800 usec
DE            6.50 usec
TE            673.2 K
D1            1.0000000 sec
TD0           1

===== CHANNEL f1 =====
NUC1          1H
P1            14.50 usec
PL1          -2.83 dB
PL1W         16.91636658 W
SFO1         400.1724712 MHz
SI           32768
SF           400.1700000 MHz
WDW           EM
SSB           0
LB            0.30 Hz
GB            0
PC            1.00
    
```

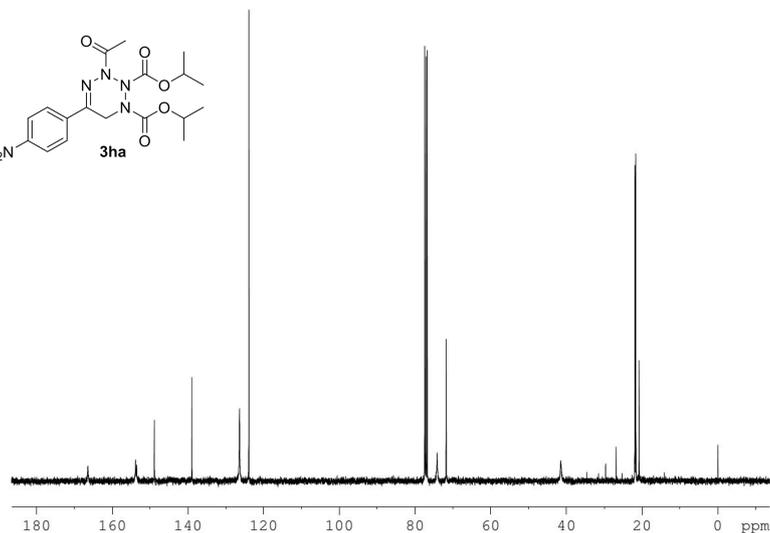
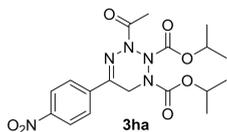
PHL-III-P31-4 13c

166.49
153.89
153.58
148.91
138.97

126.40
123.93

77.43
77.11
76.79
74.17
71.77

41.44
29.63
26.85
21.90
21.88
21.67
21.62
20.77



```

NAME          C1
EXPNO         3467
PROCNO        1
Date_         20150915
Time          10.27
INSTRUM       spect
PROBHD        5 mm PABBO BB-
PULPROG       zgpg30
ID            65536
SOLVENT       CDCl3
NS            277
DS            4
SWH           24038.461 Hz
FIDRES        0.136798 Hz
AQ            1.3631988 sec
RG            2050
DW            20.800 usec
DE            6.50 usec
TE            673.2 K
D1            2.0000000 sec
D11           0.8300000 sec
TD0           1

===== CHANNEL f1 =====
NUC1          13C
P1            11.70 usec
PL1          -2.00 dB
PL1W         48.76612744 W
SFO1         100.6328888 MHz

===== CHANNEL f2 =====
CPDPRG2       waltz16
NUC2          1H
PCPD2         80.00 usec
PL2          -2.83 dB
PL12         12.00 dB
PL13         14.00 dB
PL1W         16.91636658 W
PL12W        0.55629748 W
PL13W        0.35100001 W
SFO2         400.1716007 MHz
SI           32768
SF           100.6228270 MHz
WDW           EM
SSB           0
LB            1.00 Hz
GB            0
PC            1.40
    
```

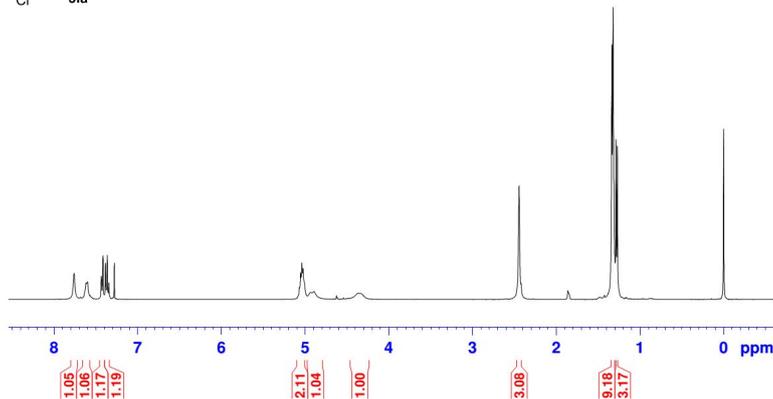
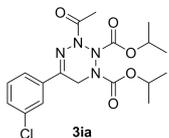
PHL-III-P31-8

7.761
7.617
7.601
7.435
7.415
7.383
7.364
7.344
7.280

5.054
5.038
5.023
4.934
4.895
4.362

2.443

1.335
1.320
1.284
1.269



```

NAME          C1
EXPNO         3451
PROCNO        1
Date_         20150910
Time          16.33
INSTRUM       spect
PROBHD        5 mm PABBO BB-
PULPROG       zg30
TD            65536
SOLVENT       CDCl3
NS            6
DS            2
SWH           8223.685 Hz
FIDRES        0.125483 Hz
AQ            3.9846387 sec
RG            114
DW            60.800 usec
DE            6.50 usec
TE            673.2 K
D1            1.00000000 sec
D11           1
TDO           1
  
```

```

===== CHANNEL f1 =====
NUC1          1H
P1            14.50 usec
PL1           -2.83 dB
PL1W         16.91636658 W
SF01         400.1724712 MHz
SI            32768
SF           400.1700000 MHz
WDW           EM
SSB           0
LB            0.30 Hz
GB            0
PC            1.00
  
```

PHL-III-P31-8 13C

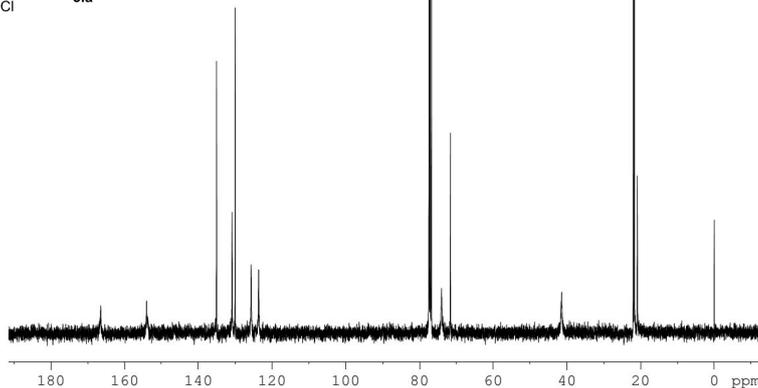
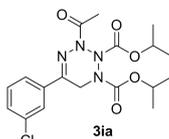
166.52
154.04

135.05
130.84
130.01
125.61
123.62

77.39
77.07
76.75
73.98
71.59

41.38

21.94
21.91
21.70
21.65
20.82



```

NAME          C1
EXPNO         3461
PROCNO        1
Date_         20150914
Time          15.02
INSTRUM       spect
PROBHD        5 mm PABBO BB-
PULPROG       zgpg30
TD            65536
SOLVENT       CDCl3
NS            4
DS            4
SWH           24038.461 Hz
FIDRES        0.366798 Hz
AQ            1.3631988 sec
RG            2050
DW            20.800 usec
DE            6.50 usec
TE            673.2 K
D1            2.00000000 sec
D11           0.03000000 sec
TDO           1
  
```

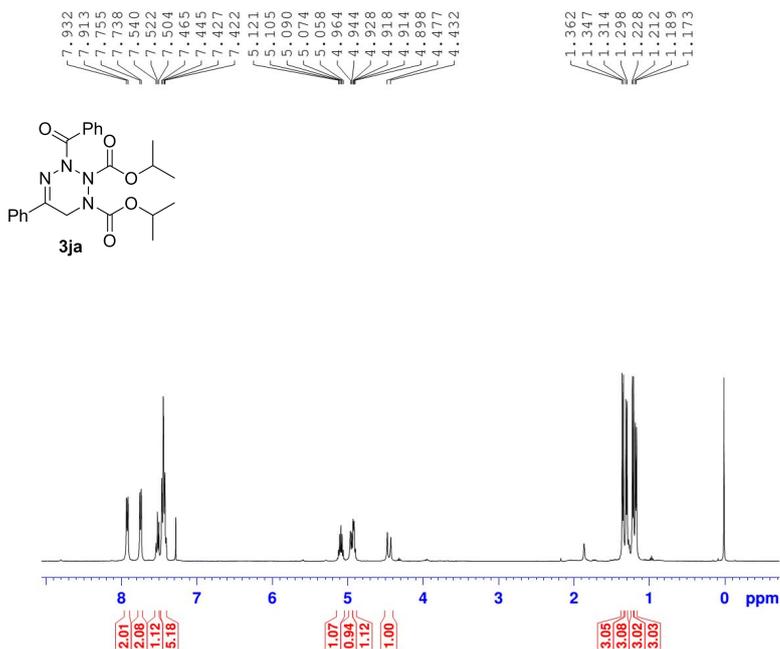
```

===== CHANNEL f1 =====
NUC1          13C
P1            11.70 usec
PL1           -2.00 dB
PL1W         48.76812744 W
SF01         100.6328888 MHz
  
```

```

===== CHANNEL f2 =====
CPDPRG2       waltz16
NUC2          1H
PCPD2         80.00 usec
PL2           -2.83 dB
PL12         12.00 dB
PL13         14.00 dB
PL2W         16.91636658 W
PL12W        0.55629748 W
PL13W        0.35100001 W
SF02         400.1716007 MHz
SI            32768
SF           100.6228270 MHz
WDW           EM
SSB           0
LB            1.00 Hz
GB            0
PC            1.40
  
```

PHL-III-P36-1



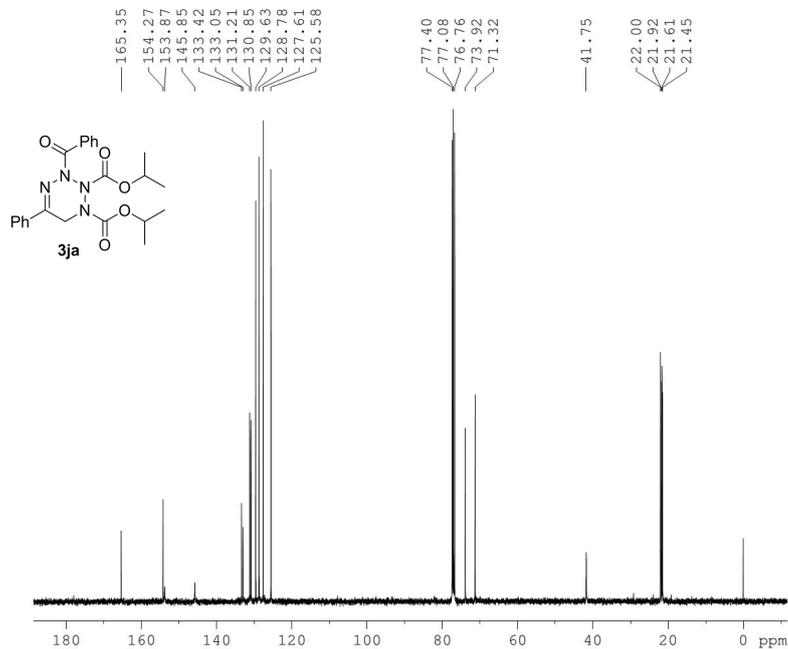
```

NAME          C1
EXPNO         3439
PROCNO        1
Date_         20150910
Time          11.45
INSTRUM       spect
PROBHD        5 mm PABBO BB-
PULPROG       zg30
TD            65536
SOLVENT       CDCl3
NS            10
DS            2
SWH           8223.685 Hz
FIDRES        0.125483 Hz
AQ            3.9846387 sec
RG            114
DW            60.800 usec
DE            6.50 usec
TE            673.2 K
D1            1.0000000 sec
TD0           1
  
```

```

===== CHANNEL f1 =====
NUC1          1H
P1            14.50 usec
PL1           -2.83 dB
PL1W          16.91636658 W
SFO1          400.1724712 MHz
SI            32768
SF            400.1700000 MHz
WDW           EM
SSB           0
LB            0.30 Hz
GB            0
PC            1.00
  
```

PHL-III-P36-1 13c



```

NAME          C1
EXPNO         3466
PROCNO        1
Date_         20150915
Time          9.47
INSTRUM       spect
PROBHD        5 mm PABBO BB-
PULPROG       zgpgq30
TD            65536
SOLVENT       CDCl3
NS            504
DS            4
SWH           24038.461 Hz
FIDRES        0.366798 Hz
AQ            1.3631988 sec
RG            2050
DW            20.800 usec
DE            6.50 usec
TE            673.2 K
D1            2.0000000 sec
D11           0.0300000 sec
TD0           1
  
```

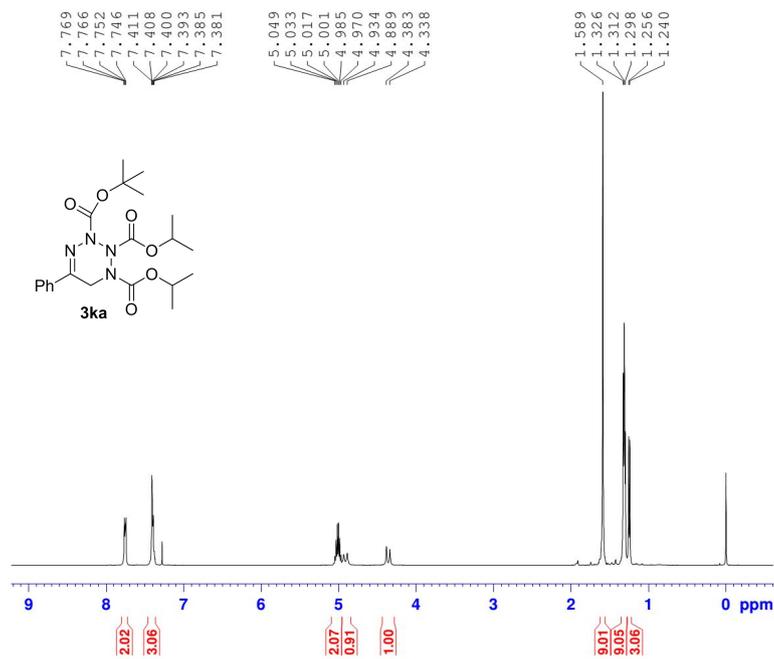
```

===== CHANNEL f1 =====
NUC1          13C
P1            11.70 usec
PL1           -2.00 dB
PL1W          48.76912744 W
SFO1          100.6328888 MHz
  
```

```

===== CHANNEL f2 =====
CPDPRG2       waltz16
NUC2          1H
PCPD2         80.00 usec
PL2           -2.83 dB
PL12          12.00 dB
PL13          14.00 dB
PL2W          16.91636658 W
PL12W         0.55629748 W
PL13W         0.35100001 W
SFO2          400.1716007 MHz
SI            32768
SF            100.6228270 MHz
WDW           EM
SSB           0
LB            1.00 Hz
GB            0
PC            1.40
  
```

PHL-III-P55-1

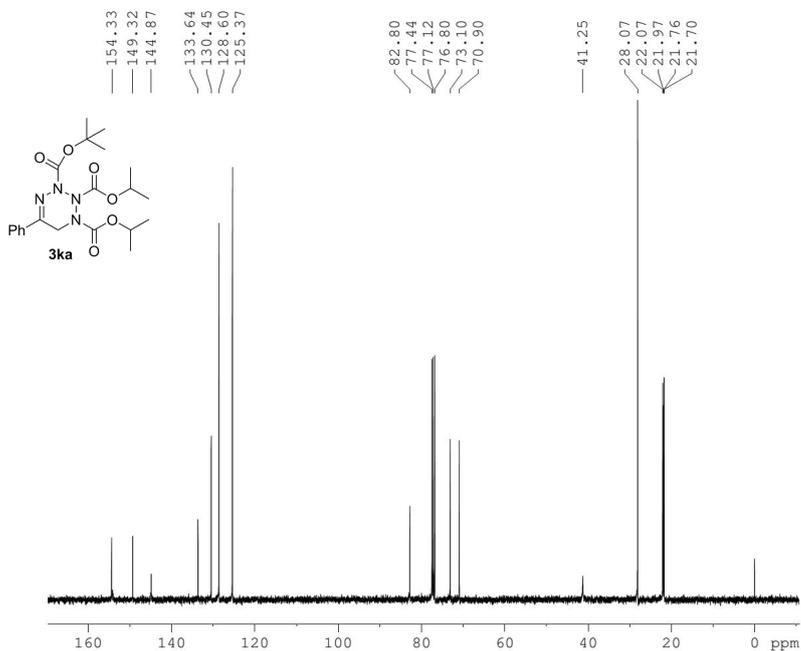


```

NAME          C1
EXPNO         3563
PROCNO        1
Date_         20151014
Time          10.25
INSTRUM       spect
PROBHD        5 mm PABBO BB-
PULPROG       zg30
TD            65536
SOLVENT       cdcl3
NS            10
DS            2
SWH           8223.665 Hz
FIDRES        0.125483 Hz
AQ            3.9846387 sec
RG            64
DW            60.800 usec
DE            6.50 usec
TE            296.8 K
D1            1.00000000 sec
D11           1
TDO           1

===== CHANNEL f1 =====
NUC1          1H
P1            14.50 usec
PL1           -2.83 dB
PL1W          16.91636658 W
SF01          400.1724712 MHz
SI            32768
SF            400.1700000 MHz
WDW           EM
SSB           0
LB            0.30 Hz
GB            0
PC            1.00
    
```

PHL-III-P55-1

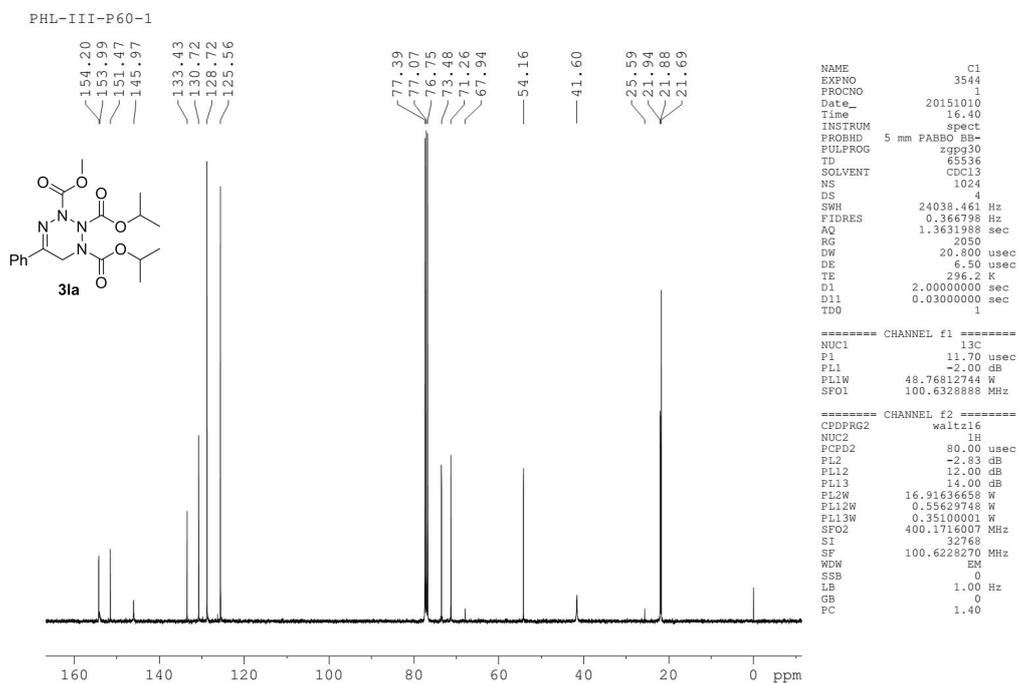
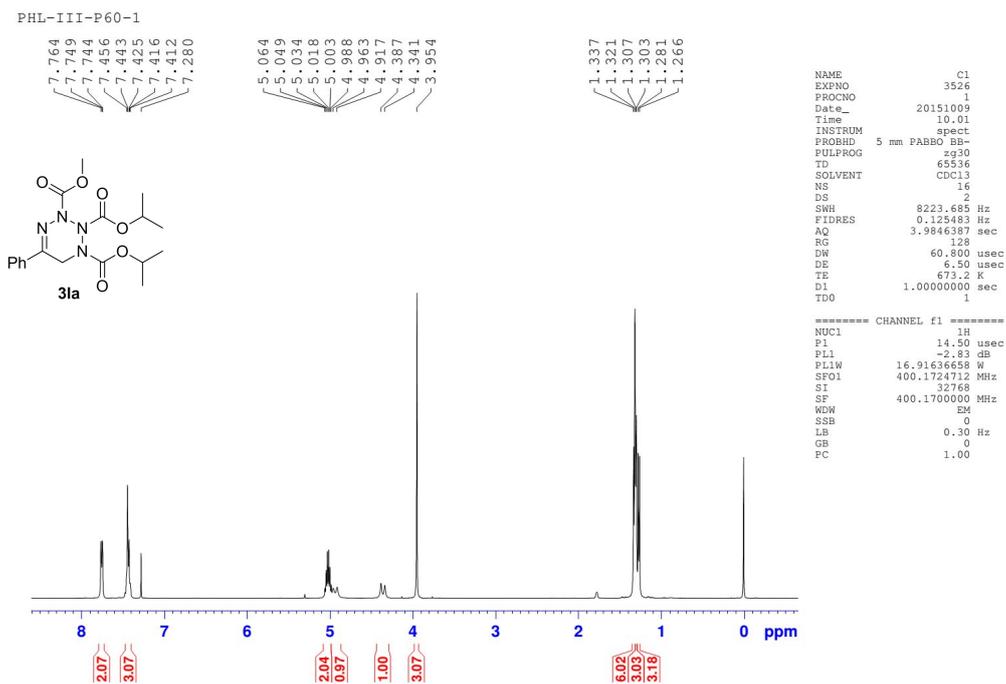


```

NAME          C1
EXPNO         3573
PROCNO        1
Date_         20151019
Time          10.33
INSTRUM       spect
PROBHD        5 mm PABBO BB-
PULPROG       zgpg30
TD            65536
SOLVENT       CDCl3
NS            106
DS            4
SWH           24038.461 Hz
FIDRES        0.366798 Hz
AQ            1.3631988 sec
RG            2050
DW            20.800 usec
DE            6.50 usec
TE            294.0 K
D1            2.00000000 sec
D11           0.03000000 sec
TDO           1

===== CHANNEL f1 =====
NUC1          13C
P1            11.70 usec
PL1           -2.00 dB
PL1W          48.76812744 W
SF01          100.6328888 MHz

===== CHANNEL f2 =====
CPDPRG2       waltz16
NUC2          1H
PCPD2         80.00 usec
PL2           -2.83 dB
PL12          12.00 dB
PL13          14.00 dB
PL1W          16.91636658 W
PL12W         0.55629748 W
PL13W         0.35100001 W
SF02          400.1716007 MHz
SI            32768
SF            100.6228270 MHz
WDW           EM
SSB           0
LB            1.00 Hz
GB            0
PC            1.40
    
```



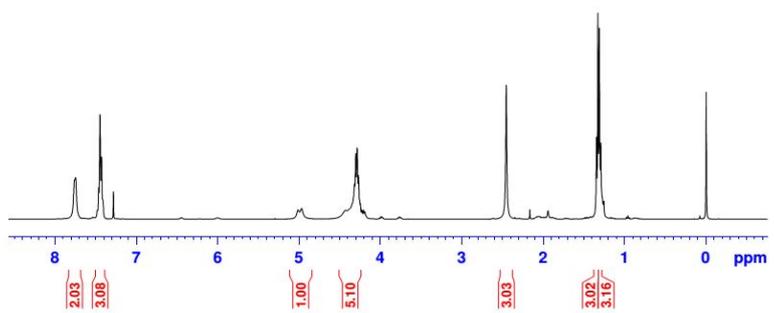
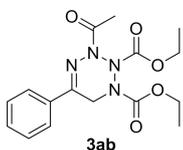
PHL-III-P35-1

7.758
7.746
7.462
7.445
7.426
7.411
7.280

5.010
4.967
4.431
4.319
4.304
4.287
4.272

— 2.454

1.345
1.328
1.309
1.291



```

NAME          C1
EXPNO         3537
PROCNO        1
Date_         20151010
Time          14.35
INSTRUM       spect
PROBHD        5 mm FABBO BB-
PULPROG       zg30
TD            65536
SOLVENT       CDCl3
NS            8
DS            2
SWH           8223.685 Hz
FIDRES        0.125483 Hz
AQ            3.9846387 sec
RG            144
DW            60.800 usec
DE            6.50 usec
TE            295.4 K
D1            1.00000000 sec
TD0           1

===== CHANNEL f1 =====
NUC1          1H
P1            14.50 usec
PL1           -2.83 dB
PL1W          16.91636658 W
SFO1          400.1724712 MHz
SI            32768
SF            400.1700000 MHz
WDW           EM
SSB           0
LB            0.30 Hz
GB            0
PC            1.00
    
```

PHL-III-P35-1 13C

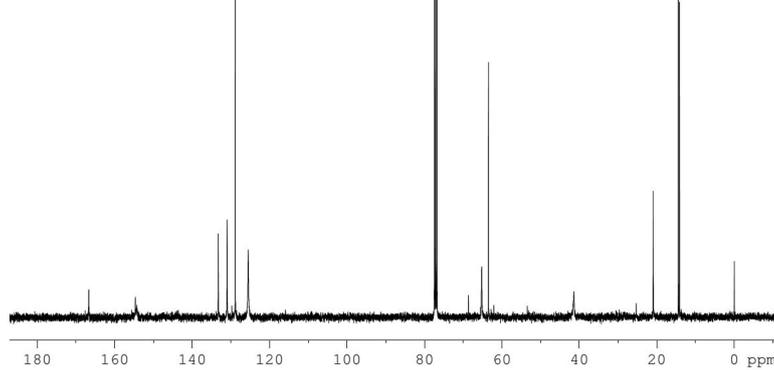
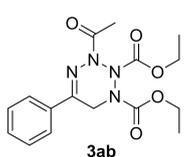
166.64
154.61
154.24

133.20
130.93
129.71
128.78
125.47

77.42
77.10
76.78
68.67
65.23
63.45

— 41.42

20.93
14.94
14.15



```

NAME          C1
EXPNO         3481
PROCNO        1
Date_         20150918
Time          9.50
INSTRUM       spect
PROBHD        5 mm PABBO BB-
PULPROG       zgpg30
TD            65536
SOLVENT       CDCl3
NS            350
DS            4
SWH           24038.461 Hz
FIDRES        0.366798 Hz
AQ            1.3631988 sec
RG            2050
DW            20.800 usec
DE            6.50 usec
TE            296.2 K
D1            2.00000000 sec
D11           0.03000000 sec
TD0           1

===== CHANNEL f1 =====
NUC1          13C
P1            11.70 usec
PL1           -2.00 dB
PL1W          48.76812744 W
SFO1          100.6328888 MHz

===== CHANNEL f2 =====
CPDPRG2       waltz16
NUC2          1H
PCPD2         80.00 usec
PL2           -2.83 dB
PL12          12.00 dB
PL13          14.00 dB
PL2W          16.91636658 W
PL12W         0.55629748 W
PL13W         0.35100001 W
SFO2          400.1716007 MHz
SI            32768
SF            100.6228270 MHz
WDW           EM
SSB           0
LB            1.00 Hz
GB            0
PC            1.40
    
```

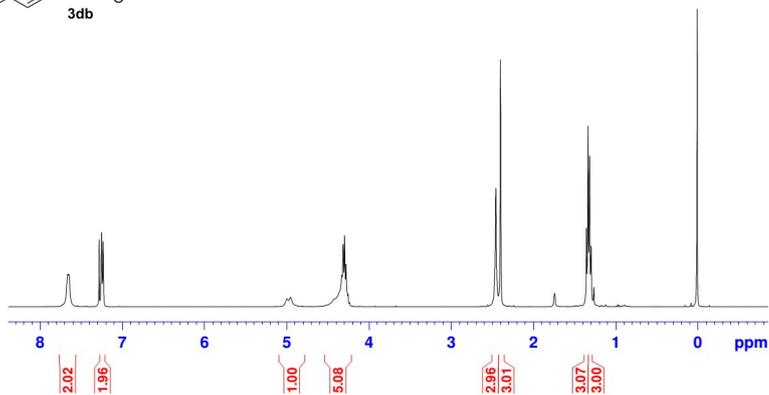
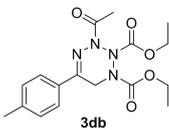
PHL-III-P35-2

7.662
7.649
7.280
7.251
7.231

4.999
4.957
4.334
4.330
4.317
4.299
4.282
4.256

2.459
2.402

1.358
1.340
1.320
1.301



```

NAME          C1
EXPNO         3528
PROCNO        1
Date_         20151009
Time          10.19
INSTRUM       spect
PROBHD        5 mm PABBO BB-
PULPROG       zgpg30
TD            65536
SOLVENT       CDCl3
NS            16
DS            2
SWH           8223.685 Hz
FIDRES        0.125483 Hz
AQ            3.9846387 sec
RG            128
DW            60.800 usec
DE            6.50 usec
TE            299.9 K
D1            1.00000000 sec
TD0           1
===== CHANNEL f1 =====
NUC1          1H
P1            14.50 usec
PL1           -2.83 dB
PL1W         16.91636658 W
SF01         400.1724712 MHz
SI           32768
SF           400.1700000 MHz
WDW           EM
SSB           0
LB            0.30 Hz
GB            0
PC            1.00
    
```

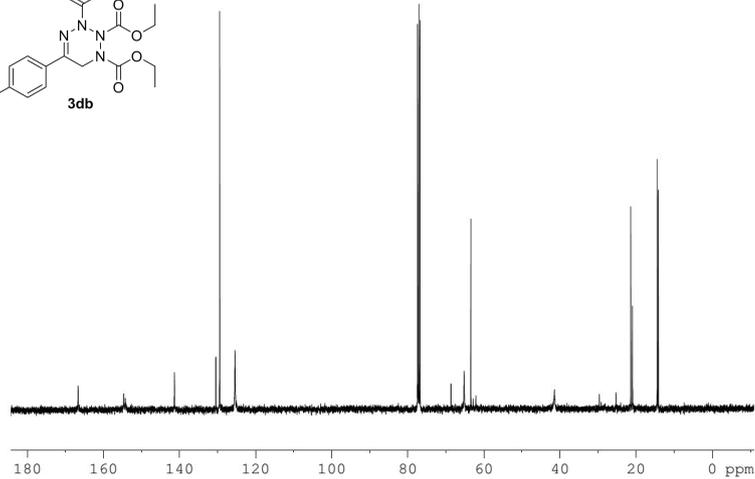
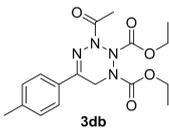
PHL-III-P35-2 13C

166.64
154.67
154.25
141.33
130.46
129.44
125.40

68.64
65.18
63.39

41.43

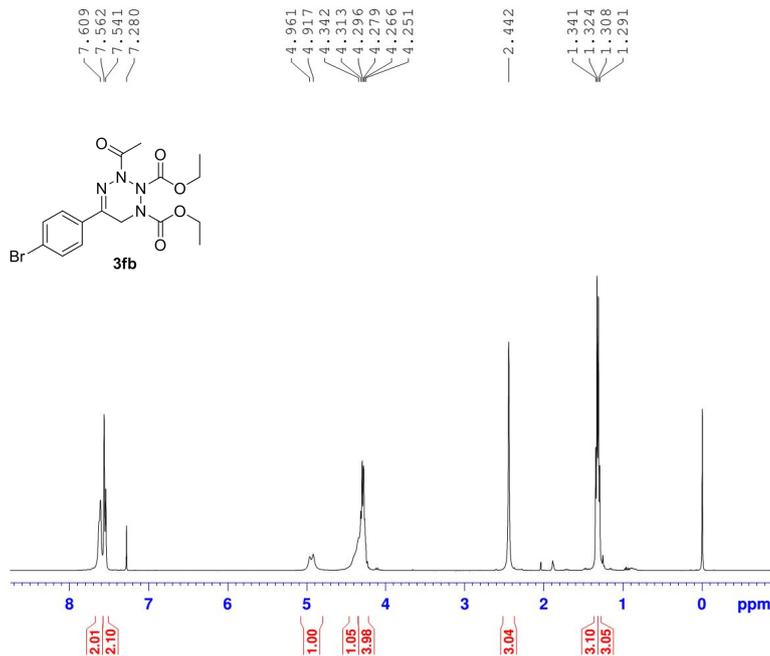
21.37
20.90
14.42
14.13



```

NAME          C1
EXPNO         3359
PROCNO        1
Date_         20150727
Time          11.48
INSTRUM       spect
PROBHD        5 mm PABBO BB-
PULPROG       zgpg30
TD            65536
SOLVENT       CDCl3
NS            309
DS            4
SWH           24038.461 Hz
FIDRES        0.366798 Hz
AQ            1.3631988 sec
RG            2050
DW            20.800 usec
DE            6.50 usec
TE            299.9 K
D1            2.00000000 sec
D11           0.03000000 sec
TD0           1
===== CHANNEL f1 =====
NUC1          13C
P1            11.70 usec
PL1           -2.00 dB
PL1W         48.76812744 W
SF01         100.6328888 MHz
===== CHANNEL f2 =====
CPDPRG2       waltz16
NUC2          1H
PCPD2         80.00 usec
PL2           -2.83 dB
PL12         12.00 dB
PL13         14.00 dB
PL2W         16.91636658 W
PL12W        0.55629748 W
PL13W        0.35100001 W
SF02         400.1716007 MHz
SI           32768
SF           100.6228270 MHz
WDW           EM
SSB           0
LB            1.00 Hz
GB            0
PC            1.40
    
```

PHL-III-P35-3

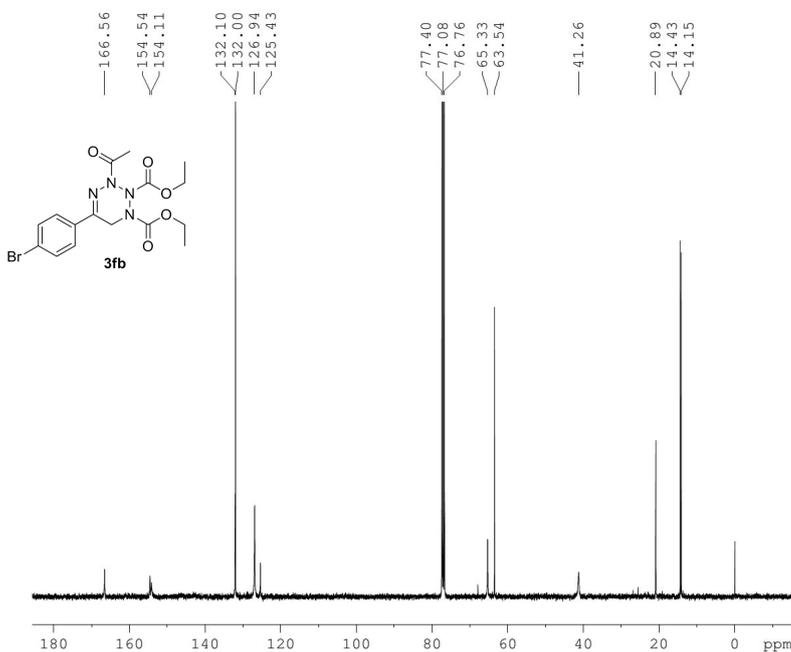


```

NAME          C1
EXPNO         3527
PROCNO        1
Date_         20151009
Time          10.15
INSTRUM       spect
PROBHD        5 mm PABBO BB-
PULPROG       zg30
TD            65536
SOLVENT       CDCl3
NS            16
DS            2
SWH           8223.683 Hz
FIDRES        0.125483 Hz
AQ            3.9846387 sec
RG            128
DW            60.500 usec
DE            6.50 usec
TE            673.2 K
D1            1.00000000 sec
TD0           1

===== CHANNEL f1 =====
NUC1          1H
P1            14.50 usec
PL1           -2.83 dB
PL1W          16.91636658 W
SFO1          400.1724712 MHz
SI            32768
SF            400.1700000 MHz
WDW           EM
SSB           0
LB            0.30 Hz
GB            0
PC            1.00
  
```

phl-iii-p35-3 13C



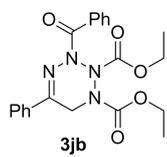
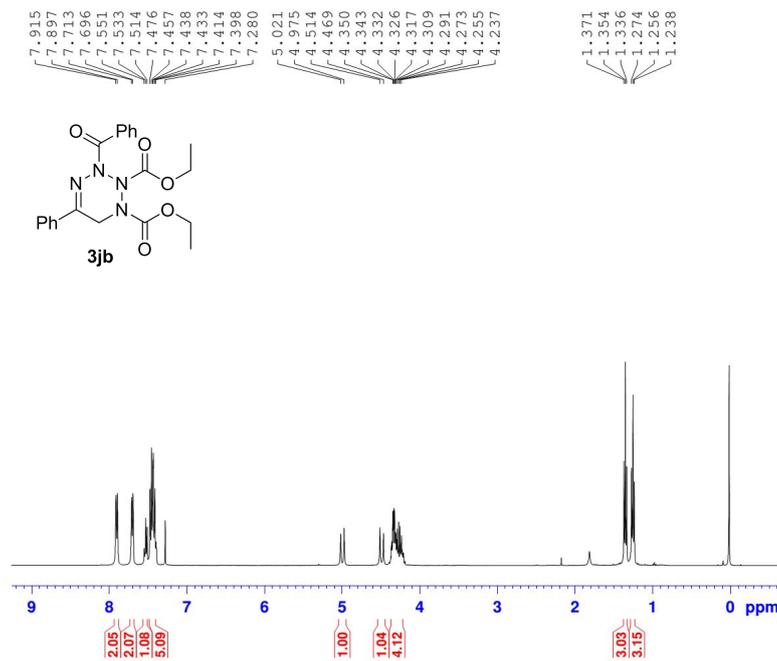
```

NAME          C1
EXPNO         3491
PROCNO        1
Date_         20150923
Time          8.58
INSTRUM       spect
PROBHD        5 mm PABBO BB-
PULPROG       zpg30
TD            65536
SOLVENT       CDCl3
NS            1024
DS            4
SWH           24038.461 Hz
FIDRES        0.366798 Hz
AQ            1.3621988 sec
RG            2050
DW            20.800 usec
DE            6.50 usec
TE            673.2 K
D1            2.00000000 sec
D11           0.03000000 sec
TD0           1

===== CHANNEL f1 =====
NUC1          13C
P1            11.70 usec
PL1           -2.00 dB
PL1W          48.76812744 W
SFO1          100.6328888 MHz

===== CHANNEL f2 =====
CPDPRG2       waltz16
NUC2          1H
PCPD2         80.00 usec
PL2           -2.83 dB
PL2W          12.00 dB
PL13          14.00 dB
PL2W          16.91636658 W
PL12W         0.55629748 W
PL13W         0.35100001 W
SFO2          400.1716007 MHz
SI            32768
SF            100.6228270 MHz
WDW           EM
SSB           0
LB            1.00 Hz
GB            0
PC            1.40
  
```


PHL-III-P36-2



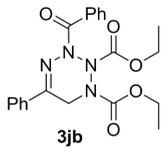
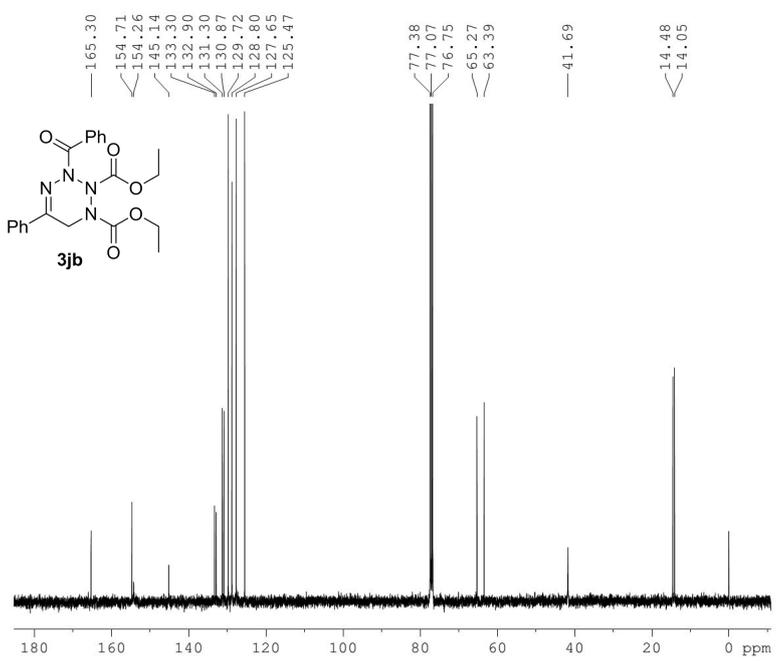
```

NAME          C1
EXPNO         3452
PROCNO        1
Date_         20150910
Time          16.37
INSTRUM       spect
PROBHD        5 mm PABBO BB-
PULPROG       zg30
TD            65536
SOLVENT       CDCl3
NS            16
DS            2
SWH           8223.685 Hz
FIDRES        0.125483 Hz
AQ            3.9846387 sec
RG            114
DW            60.800 usec
DE            6.50 usec
TE            673.2 K
D1            1.00000000 sec
D11           1
TD0           1
  
```

```

===== CHANNEL f1 =====
NUC1          1H
P1            14.50 usec
PL1           -2.83 dB
PL1W         16.91636658 W
SFO1         400.1724712 MHz
SI           32768
SF           400.1700000 MHz
WDW           EM
SSB           0
LB            0.30 Hz
GB            0
PC            1.00
  
```

PHL-III-P36-2 13c



```

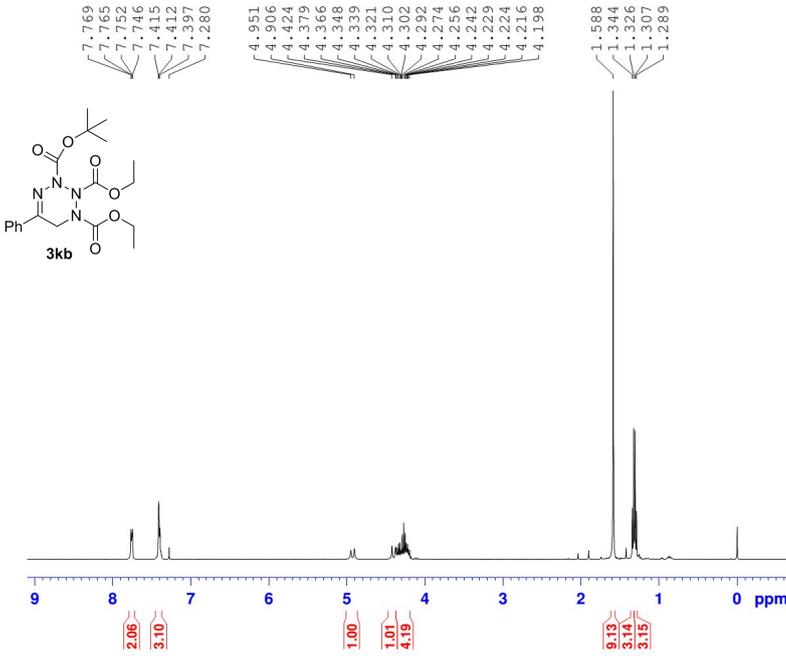
NAME          C1
EXPNO         3468
PROCNO        1
Date_         20150915
Time          10.41
INSTRUM       spect
PROBHD        5 mm PABBO BB-
PULPROG       zpgg30
TD            65536
SOLVENT       CDCl3
NS            188
DS            4
SWH           24038.461 Hz
FIDRES        0.366798 Hz
AQ            1.3631988 sec
RG            2050
DW            20.800 usec
DE            6.50 usec
TE            673.2 K
D1            2.00000000 sec
D11           0.03000000 sec
TD0           1
  
```

```

===== CHANNEL f1 =====
NUC1          13C
P1            11.70 usec
PL1           -2.00 dB
PL1W         48.76812744 W
SFO1         100.6328888 MHz

===== CHANNEL f2 =====
CPDPRG2       waltz16
NUC2          1H
PCPD2         80.00 usec
PL2           -2.83 dB
PL2W         12.00 dB
PL13         14.00 dB
PL2W         16.91636658 W
PL12W        0.55629748 W
PL13W        0.35100001 W
SFO2         400.1716007 MHz
SI           32768
SF           100.6228270 MHz
WDW           EM
SSB           0
LB            1.00 Hz
GB            0
PC            1.40
  
```

PHL-III-P55-2



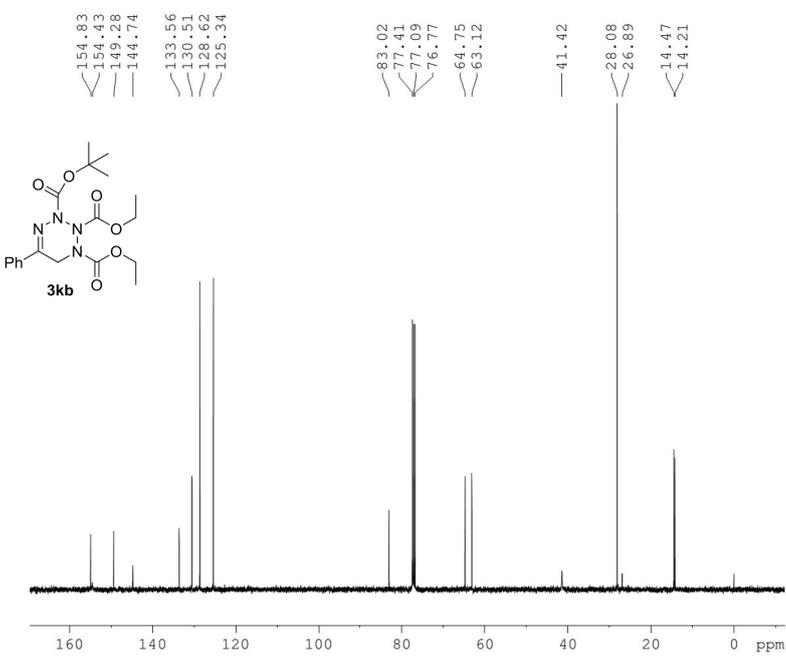
```

NAME          C1
EXPNO         3554
PROCNO        1
Date_         20151012
Time          10.56
INSTRUM       spect
PROBHD        5 mm PABBO BB-
PULPROG       zg30
TD            65536
SOLVENT       CDCl3
NS            6
DS            2
SWH           8223.685 Hz
FIDRES        0.125483 Hz
AQ           3.9846387 sec
RG            114
DW           60.800 usec
DE            6.50 usec
TE            294.8 K
D1            1.00000000 sec
D11           1
TD0           1
  
```

```

===== CHANNEL f1 =====
NUC1          1H
P1            14.50 usec
PL1           -2.83 dB
PL1W         16.91636658 W
SF01         400.1724712 MHz
SI            32768
SF           400.17000000 MHz
WDW           EM
SSB           0
LB            0.30 Hz
GB            0
PC            1.00
  
```

PHL-III-P55-2 13C



```

NAME          C1
EXPNO         3557
PROCNO        1
Date_         20151012
Time          14.47
INSTRUM       spect
PROBHD        5 mm PABBO BB-
PULPROG       zpgg30
TD            65536
SOLVENT       CDCl3
NS            128
DS            4
SWH           24038.461 Hz
FIDRES        0.366798 Hz
AQ           1.3631988 sec
RG            2050
DW           20.800 usec
DE            6.50 usec
TE            296.6 K
D1            2.00000000 sec
D11           0.03000000 sec
TD0           1
  
```

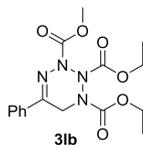
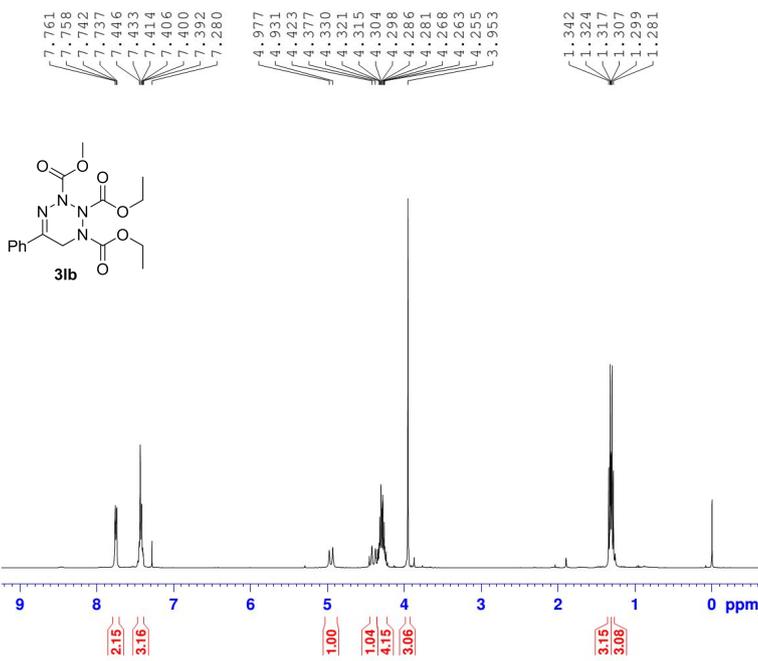
```

===== CHANNEL f1 =====
NUC1          13C
P1            11.70 usec
PL1           -2.00 dB
PL1W         48.76812744 W
SF01         100.6328888 MHz
  
```

```

===== CHANNEL f2 =====
CPDPRG2       waltz16
NUC2          1H
PCPD2         80.00 usec
PL2           -2.83 dB
PL12          12.00 dB
PL13          14.00 dB
PL1W         16.91636658 W
PL12W        0.55629748 W
PL13W        0.35100001 W
SF02         400.1716007 MHz
SI            32768
SF           100.6282270 MHz
WDW           EM
SSB           0
LB            1.00 Hz
GB            0
PC            1.40
  
```

PHL-III-P60-2

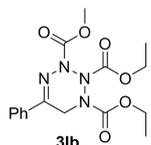
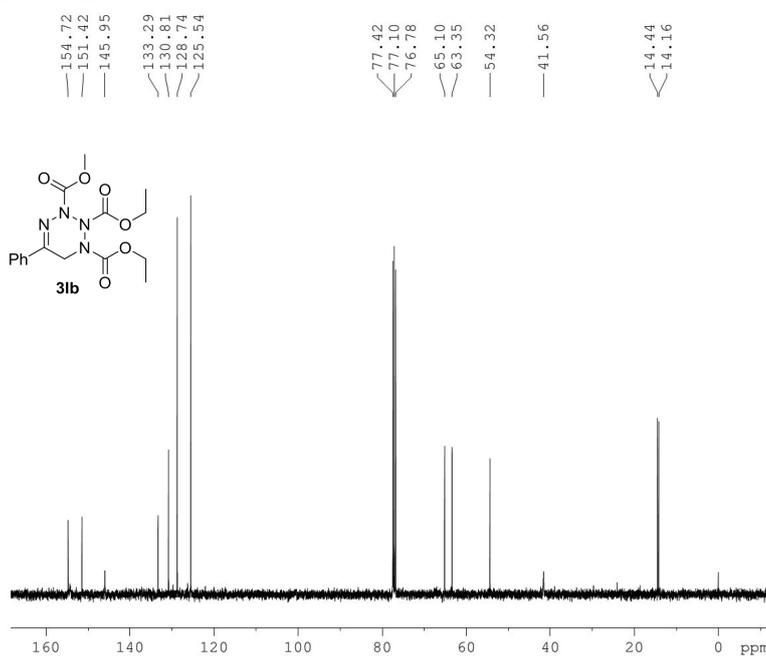


```

NAME          C1
EXPNO         3518
PROCNO        1
Date_         20151008
Time          15.01
INSTRUM       spect
PROBHD        5 mm PABBO BB-
PULPROG       zg30
TD            65536
SOLVENT       CDCl3
NS            4
DS            2
SWH           8223.685 Hz
FIDRES        0.125483 Hz
AQ            3.9846387 sec
RG            123
DW            60.800 usec
DE            6.50 usec
TE            673.2 K
D1            1.00000000 sec
TD0           1

===== CHANNEL f1 =====
NUC1          1H
P1            14.50 usec
PL1          -2.83 dB
PL1W         16.91636658 W
SFO1         400.1724712 MHz
SI           32768
SF           400.1700000 MHz
WDW           EM
SSB           0
LB           0.30 Hz
GB            0
PC            1.00
  
```

PHL-III-P60-2 13C



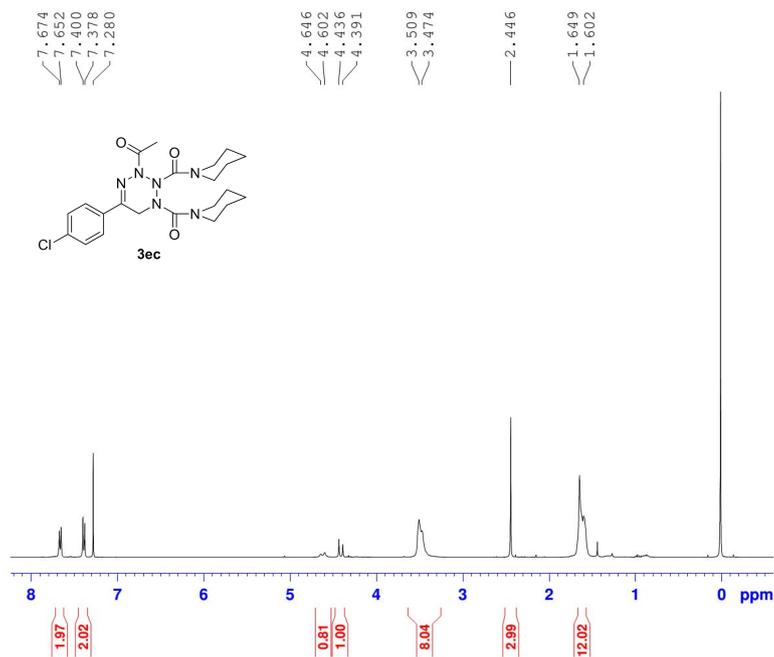
```

NAME          C1
EXPNO         3530
PROCNO        1
Date_         20151009
Time          10.38
INSTRUM       spect
PROBHD        5 mm PABBO BB-
PULPROG       zgpg30
TD            65536
SOLVENT       CDCl3
NS            64
DS            4
SWH           24038.461 Hz
FIDRES        0.366798 Hz
AQ            1.3631988 sec
RG            2050
DW            20.800 usec
DE            6.50 usec
TE            673.2 K
D1            2.00000000 sec
D11           0.03000000 sec
TD0           1

===== CHANNEL f1 =====
NUC1          13C
P1            11.70 usec
PL1          -2.00 dB
PL1W         48.76812744 W
SFO1         100.6328888 MHz

===== CHANNEL f2 =====
CPDPRG2       waltz16
NUC2          1H
PCPD2         80.00 usec
PL2          -2.83 dB
PL12         12.00 dB
PL13         14.00 dB
PL2W         16.91636658 W
PL12W        0.5529748 W
PL13W        0.35100001 W
SFO2         400.1716007 MHz
SI           32768
SF           100.6228270 MHz
WDW           EM
SSB           0
LB           1.00 Hz
GB            0
PC            1.40
  
```

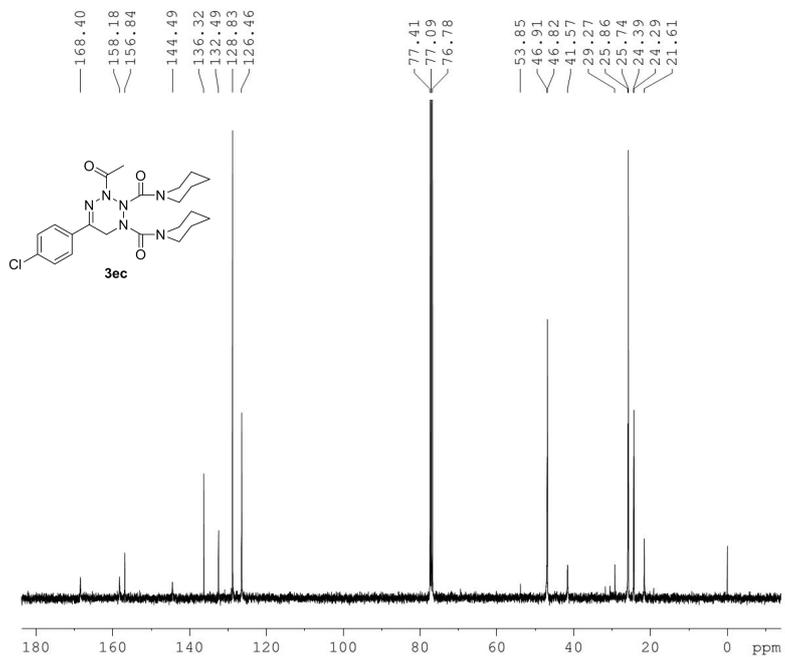
PHL-III-P49-1



```

NAME          C1
EXPNO         3507
PROCNO        1
Date_         20150930
Time          14.26
INSTRUM       spect
PROBHD        5 mm PABBO BB-
PULPROG       zgpg30
TD            65536
SOLVENT       CDCl3
NS            16
DS            2
SWH           8223.685 Hz
FIDRES        0.125483 Hz
AQ            3.9846387 sec
RG            144
DW            60.800 usec
DE            6.50 usec
TE            297.4 K
D1            1.00000000 sec
TDO           1
===== CHANNEL f1 =====
NUC1          1H
P1            14.50 usec
PL1           -2.83 dB
PL1W         16.91636658 W
SF01         400.1724712 MHz
SI            32768
SF           400.1700000 MHz
WDW           EM
SSB           0
LB            0.30 Hz
GB            0
PC            1.00
  
```

PHL-III-P49-1



```

NAME          C1
EXPNO         3543
PROCNO        1
Date_         20151010
Time          16.16
INSTRUM       spect
PROBHD        5 mm PABBO BB-
PULPROG       zgpg30
TD            65536
SOLVENT       CDCl3
NS            298
DS            4
SWH           24038.461 Hz
FIDRES        0.366798 Hz
AQ            1.3631988 sec
RG            2050
DW            20.800 usec
DE            6.50 usec
TE            295.2 K
D1            2.00000000 sec
D11           0.03000000 sec
TDO           1
===== CHANNEL f1 =====
NUC1          13C
P1            11.70 usec
PL1           -2.00 dB
PL1W         48.76812744 W
SF01         100.6328888 MHz
===== CHANNEL f2 =====
CPDPRG2      waltz16
NUC2          1H
PCPD2        80.00 usec
PL2           -2.83 dB
PL12         12.00 dB
PL13         14.00 dB
PL2W         16.91636658 W
PL12W        0.55629748 W
PL13W        0.55100001 W
SF02         400.1716007 MHz
SI            32768
SF           100.6228270 MHz
WDW           EM
SSB           0
LB            1.00 Hz
GB            0
PC            1.40
  
```

PHL-III-P38-1

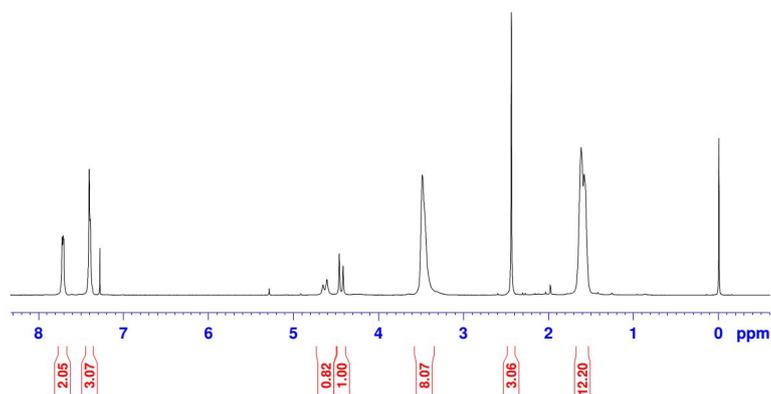
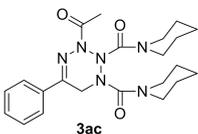
7.719
7.708
7.701
7.405
7.394

4.653
4.609
4.463
4.418

3.486

2.438

1.616
1.581



```

NAME          C1
EXPNO         3561
PROCNO        1
Date_         20151014
Time          10.13
INSTRUM       spect
PROBHD        5 mm PABBO BB-
PULPROG       zg30
TD            65536
SOLVENT       CDCl3
NS            6
DS            2
SWH           8223.685 Hz
FIDRES        0.125483 Hz
AQ            3.9846387 sec
RG            64
DW            60.800 usec
DE            6.50 usec
TE            296.8 K
D1            1.0000000 sec
TD0           1

```

```

===== CHANNEL f1 =====
NUC1          1H
P1            14.50 usec
PL1           -2.83 dB
PL1W          16.91636658 W
SF01          400.1724712 MHz
SI            32768
SF            400.1700000 MHz
WDW           EM
SSB           0
LB            0.30 Hz
GB            0
PC            1.00

```

PHL-III-P38-1

168.50
158.36
156.93
145.61
133.99
130.32
128.60
125.19

77.47

77.15

76.83

46.89

46.00

41.85

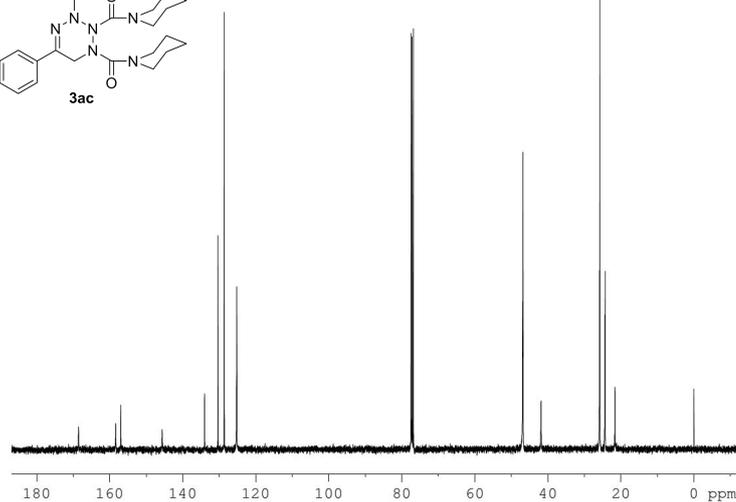
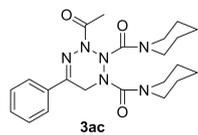
25.85

25.74

24.41

24.31

21.65



```

NAME          C1
EXPNO         3572
PROCNO        1
Date_         20151019
Time          10.14
INSTRUM       spect
PROBHD        5 mm PABBO BB-
PULPROG       zgpg30
TD            65536
SOLVENT       CDCl3
NS            260
DS            4
SWH           24038.461 Hz
FIDRES        0.366798 Hz
AQ            1.3631988 sec
RG            2050
DW            20.800 usec
DE            6.50 usec
TE            293.3 K
D1            2.0000000 sec
D11           0.0300000 sec
TD0           1

```

```

===== CHANNEL f1 =====
NUC1          13C
P1            11.70 usec
PL1           -2.00 dB
PL1W          48.76812744 W
SF01          100.6328888 MHz

```

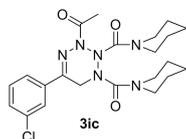
```

===== CHANNEL f2 =====
CPDPRG2       waltz16
NUC2          1H
PCPD2         80.00 usec
PL2           -2.83 dB
PL12          12.00 dB
PL13          14.00 dB
PL2W          16.91636658 W
PL12W         0.55629748 W
PL13W         0.35100001 W
SF02          400.1716007 MHz
SI            32768
SF            100.6228270 MHz
WDW           EM
SSB           0
LB            1.00 Hz
GB            0
PC            1.40

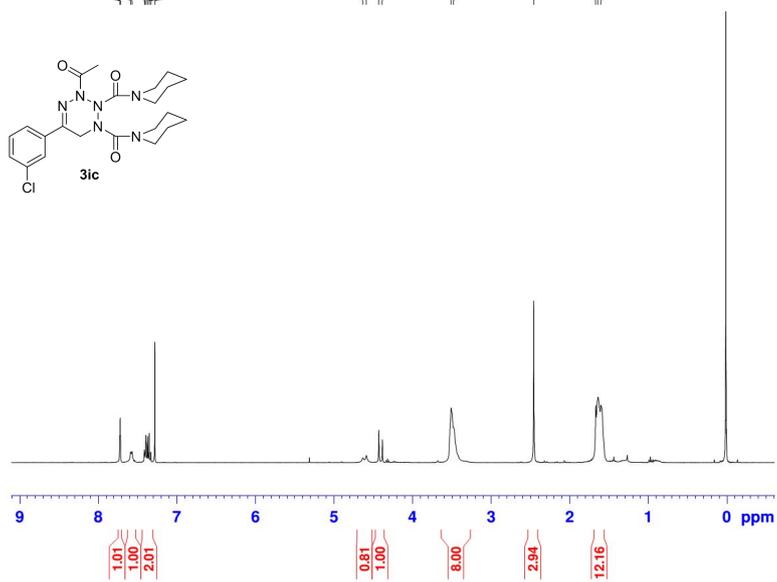
```

PHL-III-P50-2

7.724
7.720
7.716
7.586
7.569
7.411
7.407
7.391
7.370
7.350
7.331
7.280



4.634
4.590
4.431
4.386
3.510
3.478
2.459
1.669
1.642
1.604



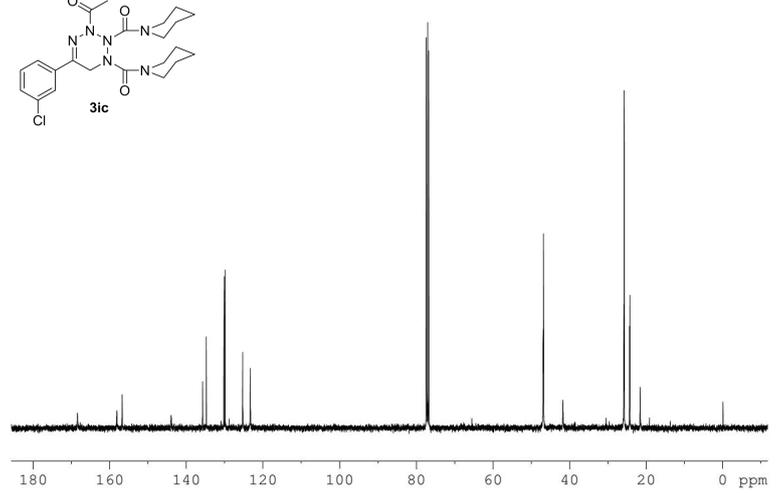
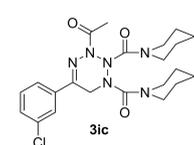
```

NAME          C1
EXPNO         3517
PROCNO        1
Date_         20151008
Time          14.56
INSTRUM       spect
PROBHD        5 mm PABBO BB-
PULPROG       zg30
TD            65536
SOLVENT       CDCl3
NS            5
DS            2
SWH           8223.665 Hz
FIDRES        0.125483 Hz
AQ            3.9846387 sec
RG            128
DW            60.800 usec
DE            6.50 usec
TE            673.2 K
D1            1.00000000 sec
TD0           1

===== CHANNEL f1 =====
NUC1          1H
P1            14.50 usec
PL1           -2.83 dB
PL1W         16.91636658 W
SFO1         400.1724712 MHz
SI            32768
SF           400.1700000 MHz
WDW           EM
SSB           0
LB            0.30 Hz
GB            0
PC            1.00
    
```

PHL-III-P50-2 13C

168.43
158.13
156.76
143.93
135.78
134.79
130.20
129.87
125.28
123.29
77.43
77.11
76.79
46.82
46.82
41.74
25.86
25.74
24.59
24.28
21.61



```

NAME          C1
EXPNO         3550
PROCNO        1
Date_         20151012
Time          10.13
INSTRUM       spect
PROBHD        5 mm PABBO BB-
PULPROG       zgpg30
TD            65536
SOLVENT       CDCl3
NS            4
DS            4
SWH           24038.461 Hz
FIDRES        0.366798 Hz
AQ            1.3631988 sec
RG            2050
DW            20.800 usec
DE            6.50 usec
TE            294.5 K
D1            2.00000000 sec
D11           0.03000000 sec
TD0           1

===== CHANNEL f1 =====
NUC1          13C
P1            11.70 usec
PL1           -2.00 dB
PL1W         48.76812744 W
SFO1         100.6328888 MHz

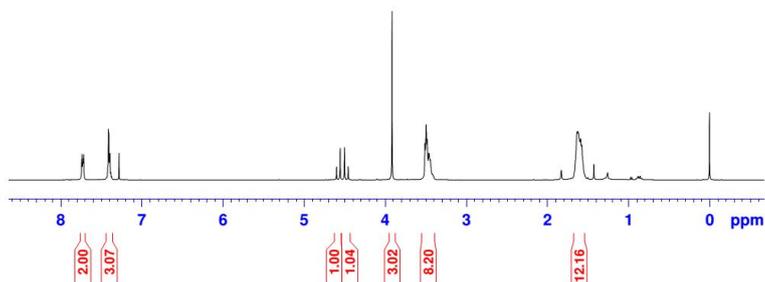
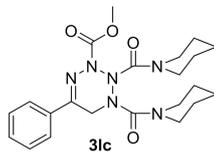
===== CHANNEL f2 =====
CPDPRG2      waltz16
NUC2          1H
PCPD2        80.00 usec
PL2           -2.83 dB
PL2W         12.00 dB
PL13         14.00 dB
PL13W        16.91636658 W
PL12W        0.55629748 W
PL13W        0.35100001 W
SFO2         400.1716007 MHz
SI            32768
SF           100.6228270 MHz
WDW           EM
SSB           0
LB            1.00 Hz
GB            0
PC            1.40
    
```

PHL-III-P64-2

7.740
7.736
7.727
7.723
7.716
7.411
7.406
7.396
7.393
7.280

4.600
4.554
4.500
4.455
3.915
3.508
3.495
3.482
3.460
3.444

1.639
1.629
1.621
1.593
1.581



```

NAME          C1
EXPNO         3598
PROCNO        1
Date_         20151022
Time          16.31
INSTRUM       spect
PROBHD        5 mm PABBO BB-
PULPROG       zg30
TD            65536
SOLVENT       CDC13
NS            6
DS            2
SWH           8223.685 Hz
FIDRES        0.125483 Hz
AQ            3.9846387 sec
RG            182
DW            60.800 usec
DE            6.50 usec
TE            293.6 K
D1            1.00000000 sec
TDO           1
  
```

```

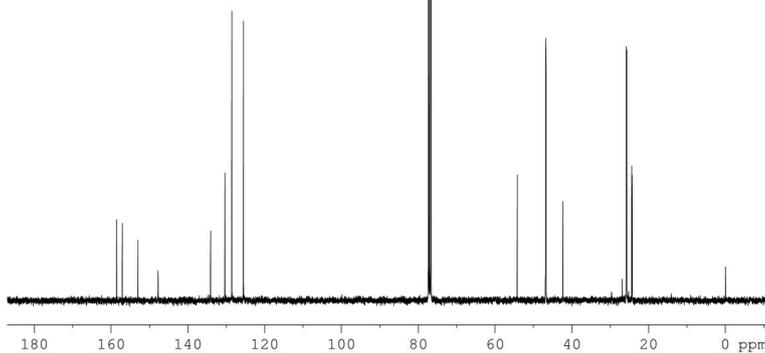
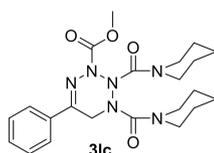
===== CHANNEL f1 =====
NUC1          1H
P1            14.50 usec
PL1          -2.83 dB
PL1W         16.91636658 W
SFO1         400.1724712 MHz
SI            32768
SF           400.1700000 MHz
WDW           EM
SSB           0
LB            0.30 Hz
GB            0
PC            1.00
  
```

PHL-III-P64-2 13C

158.58
157.10
153.03
147.77
134.09
130.35
128.57
125.53

77.39
77.08
76.76

54.27
46.87
46.78
42.35
26.90
25.85
25.76
24.45
24.32



```

NAME          C1
EXPNO         3610
PROCNO        1
Date_         20151029
Time          17.09
INSTRUM       spect
PROBHD        5 mm PABBO BB-
PULPROG       zgpg30
TD            65536
SOLVENT       CDC13
NS            311
DS            4
SWH           24038.461 Hz
FIDRES        0.366798 Hz
AQ            1.3631988 sec
RG            1620
DW            20.800 usec
DE            6.50 usec
TE            293.4 K
D1            2.00000000 sec
D11           0.03000000 sec
TDO           1
  
```

```

===== CHANNEL f1 =====
NUC1          13C
P1            11.70 usec
PL1          -2.00 dB
PL1W         48.76812744 W
SFO1         100.6328888 MHz

===== CHANNEL f2 =====
CPDPRG2       waltz16
NUC2          1H
PCPD2         80.00 usec
PL2          -2.83 dB
PL12         12.00 dB
PL13         14.00 dB
PL12W        16.91636658 W
PL13W        0.55629748 W
SFO2         400.1716007 MHz
SI            32768
SF           100.6228270 MHz
WDW           EM
SSB           0
LB            1.00 Hz
GB            0
PC            1.40
  
```

2. X-Ray crystal data of compound 3aa

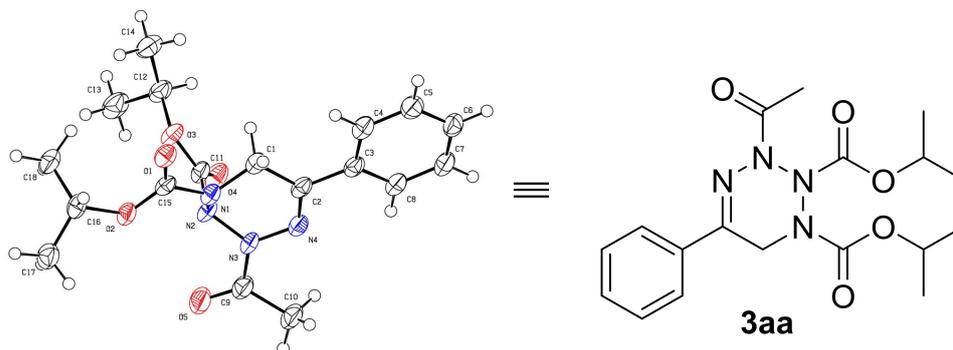


Figure 1. X-ray single crystal structure of 3aa (with thermal ellipsoids shown at the 50% probability level)

Identification code	3aa
Empirical formula	C ₁₈ H ₂₄ N ₄ O ₅
Formula weight	376.41
Temperature	113(2) K
Wavelength	0.71073 Å
Crystal system, space group	Monoclinic, P2(1)/c
Unit cell dimensions	a = 10.454(5) Å alpha = 90 deg. b = 21.985(9) Å beta = 102.524(11) deg. c = 8.731(4) Å gamma = 90 deg.
Volume	1958.8(15) Å ³
Z, Calculated density	4, 1.276 Mg/m ³
Absorption coefficient	0.095 mm ⁻¹
F(000)	800
Crystal size	0.20 x 0.18 x 0.12 mm
Theta range for data collection	3.02 to 25.02 deg.
Limiting indices	-12 ≤ h ≤ 12, -26 ≤ k ≤ 25, -10 ≤ l ≤ 10

Reflections collected / unique	19595 / 3441 [R(int) = 0.1027]
Completeness to theta = 25.02	99.4%
Absorption correction	Semi-empirical from equivalents
Max. and min. transmission	0.9887 and 0.9813
Refinement method	Full-matrix least-squares on F ²
Data / restraints / parameters	3441 / 0 / 249
Goodness-of-fit on F ²	1.000
Final R indices [I > 2σ(I)]	R1 = 0.0624, wR2 = 0.1493
R indices (all data)	R1 = 0.0744, wR2 = 0.1558
Largest diff. peak and hole	0.403 and -0.268 e.Å ⁻³
CCDC deposition number	1433224