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

Hong-Wu Zhao,* Hai-Liang Pang, Bo Li, Ting Tian, Xiao-Qin Chen, Xiu-Qing Song, Wei Meng, Zhao Yang, Yue-Yang Liu, Yu-Di Zhao

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*

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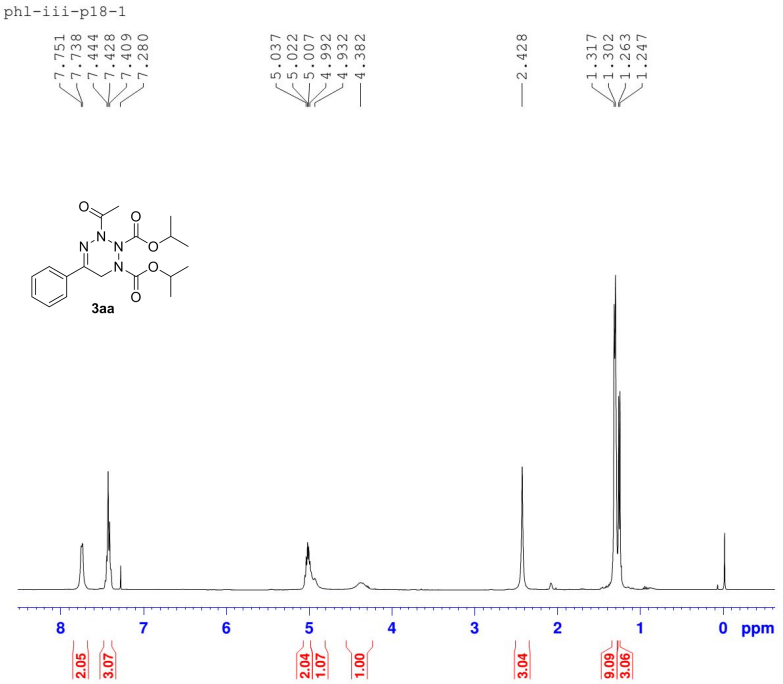
1. ^{1}H and ^{13}C NMR spectra

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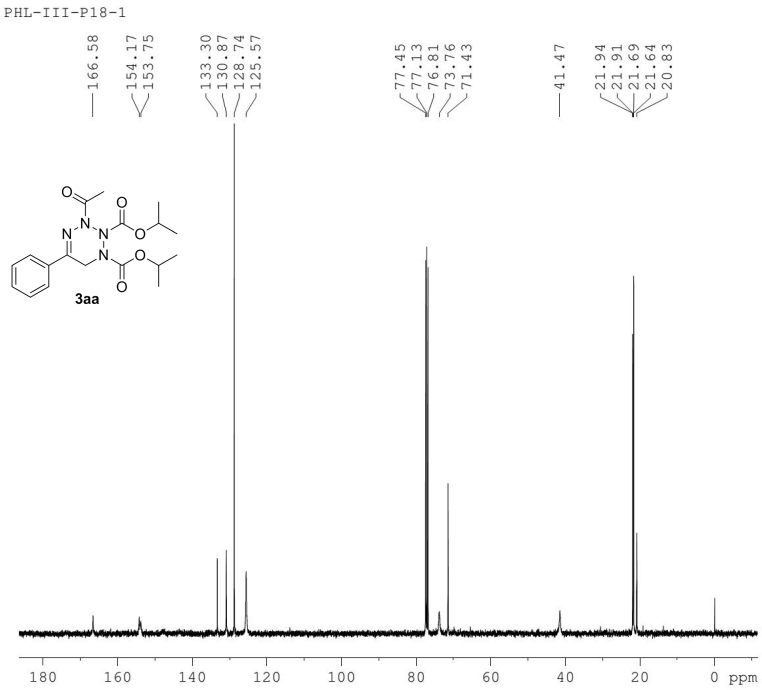
Time              10.46
INSTRUM           spect
PROBHD   5 mm PABBO BB-
PULPROG            zg30
TD                65536
SOLVENT           CDCl3
NS                   16
DS                    2
SWH            8223.685 Hz
FIDRES         0.125483 Hz
AQ             3.9846387 sec
RG                71.8
DW               60.800 usec
DE                6.50 usec
TE               673.2 K
D1             1.00000000 sec
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
```

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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            0.03000000 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
```
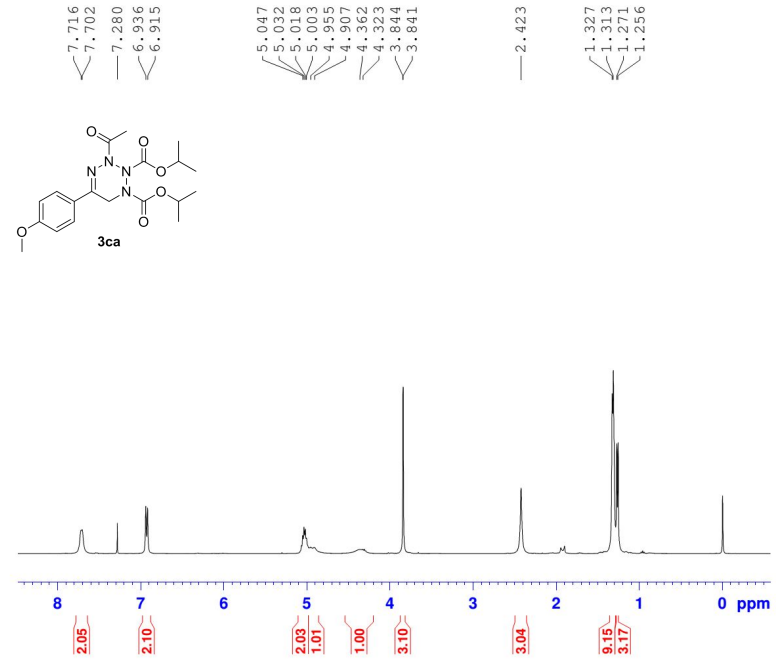

PHL-III-P31-1

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PROCNO                 1
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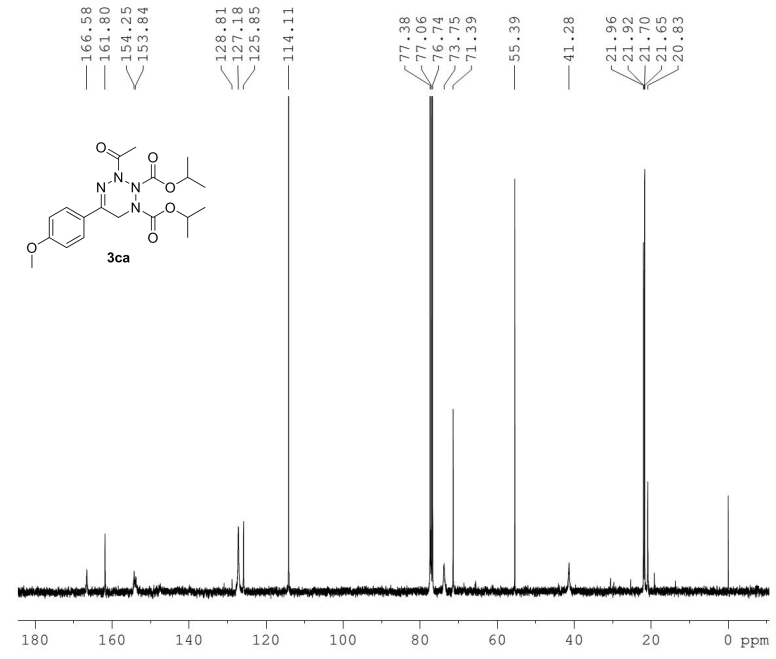
PULPROG            zg30
TD                65536
SOLVENT           CDCl3
NS                    9
DS                    2
SWH            8223.685 Hz
FIDRES         0.125483 Hz
AQ             3.9846387 sec
RG                  101
DW              60.800 usec
DE               6.50 usec
TE              673.2 K
D1           1.00000000 sec
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-P31-1 13C

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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                 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        0.55629748 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
```
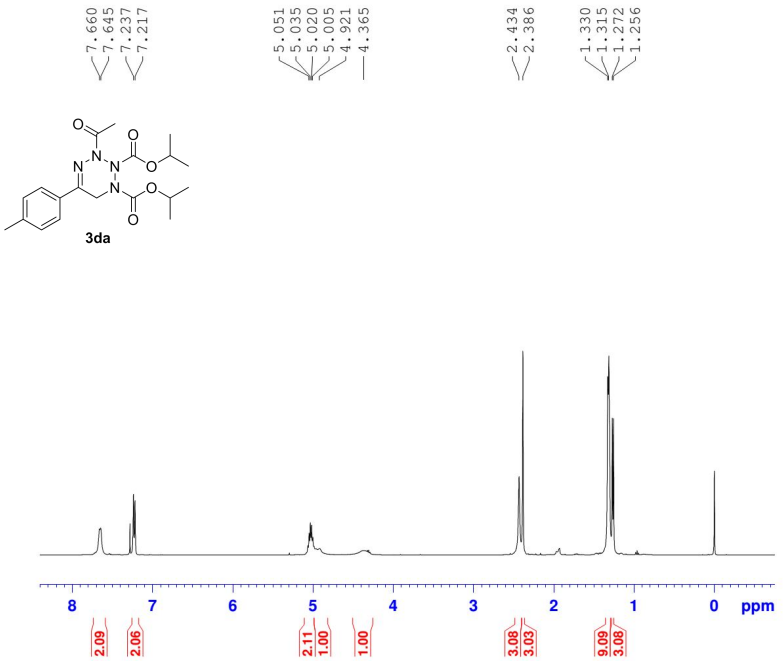

PHL-III-P31-7

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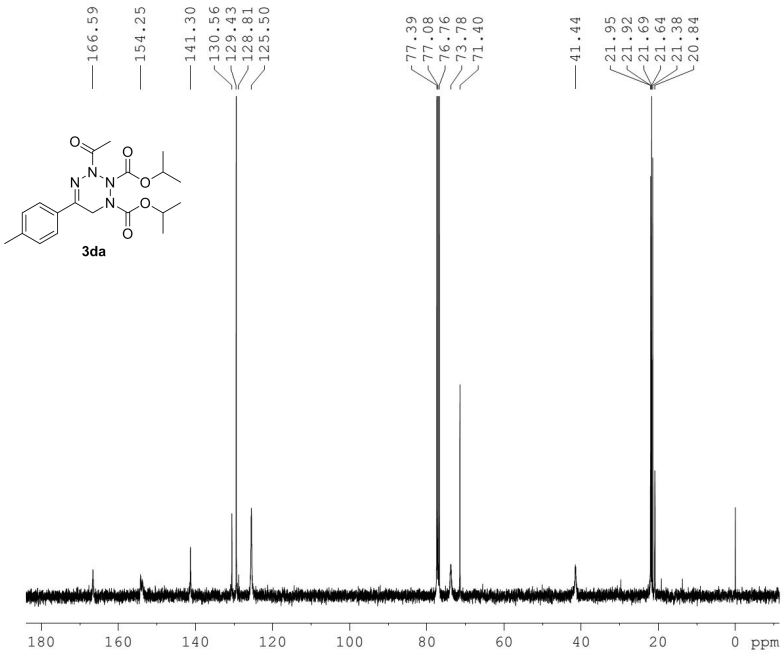
EXPNO              3428
PROCNO                1
Date_          20150908
Time               9.23
INSTRUM           spect
PROBHD   5 mm PABBO BB-
PULPROG            zg30
TD                65536
SOLVENT           CDCl3
NS                   16
DS                    2
SWH            8223.685 Hz
FIDRES        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
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-P31-7 13C




```
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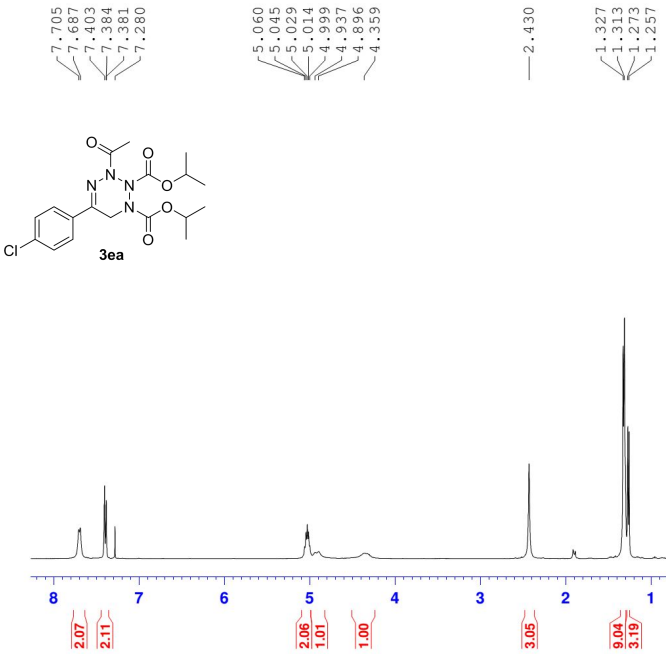
EXPNO              3432
PROCNO                1
Date_          20150909
Time              16.20
INSTRUM           spect
PROBHD   5 mm PABBO BB-
PULPROG          zgpg30
TD                65536
SOLVENT           CDCl3
NS                  401
DS                    4
SWH           24038.461 Hz
FIDRES        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           0.03000000 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-P31-2




```
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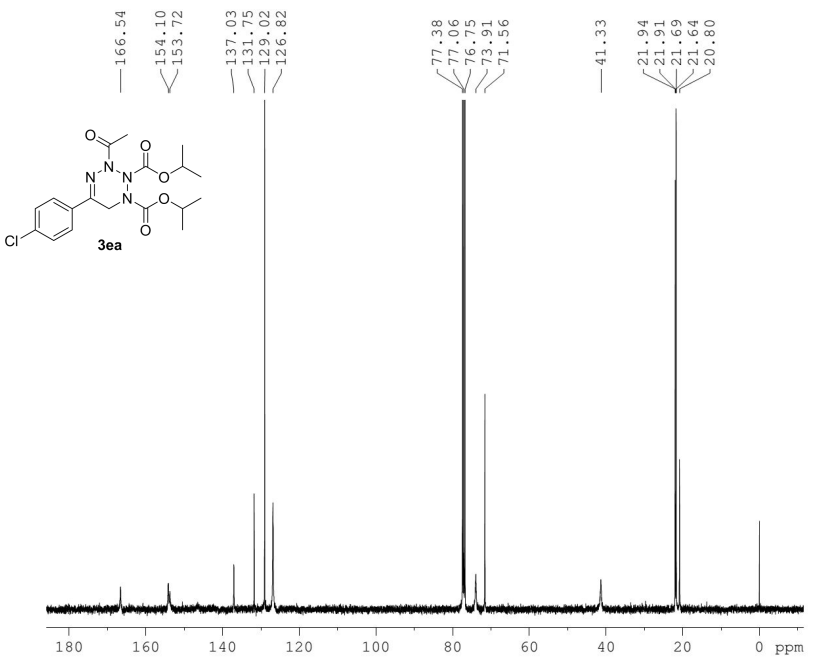
EXPNO              3426
PROCNO                1
Date_          20150908
Time               9.11
INSTRUM           spect
PROBHD    5 mm PABBO BB-
PULPROG            zg30
TD                65536
SOLVENT           CDCl3
NS                   16
DS                    2
SWH            8223.685 Hz
FIDRES         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         400.1724712 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

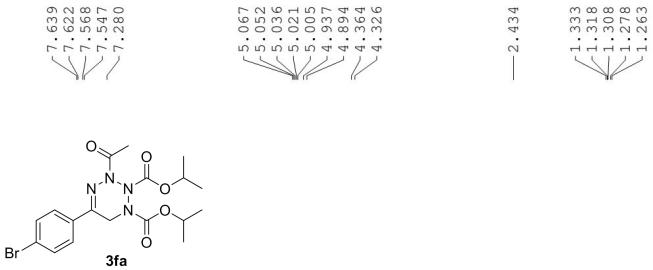


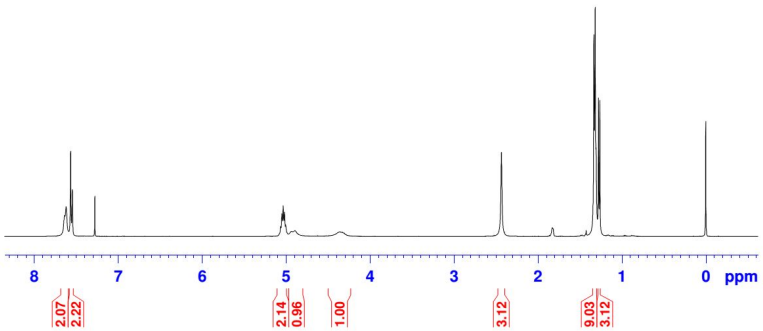

```
NAME                 C1
EXPNO              3433
PROCNO                1
Date_          20150909
Time              20.56
INSTRUM           spect
PROBHD    5 mm PABBO BB-
PULPROG           zgpg30
TD                65536
SOLVENT           CDCl3
NS                 1024
DS                    4
SWH           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
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-P31-3



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PROCNO                1
Date_          20150909
Time              16.08
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                  114
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
```
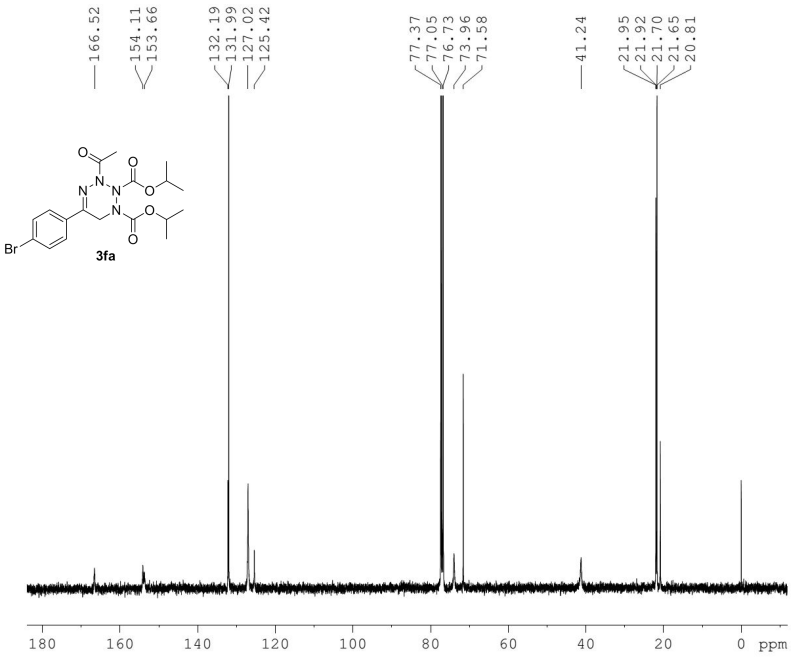
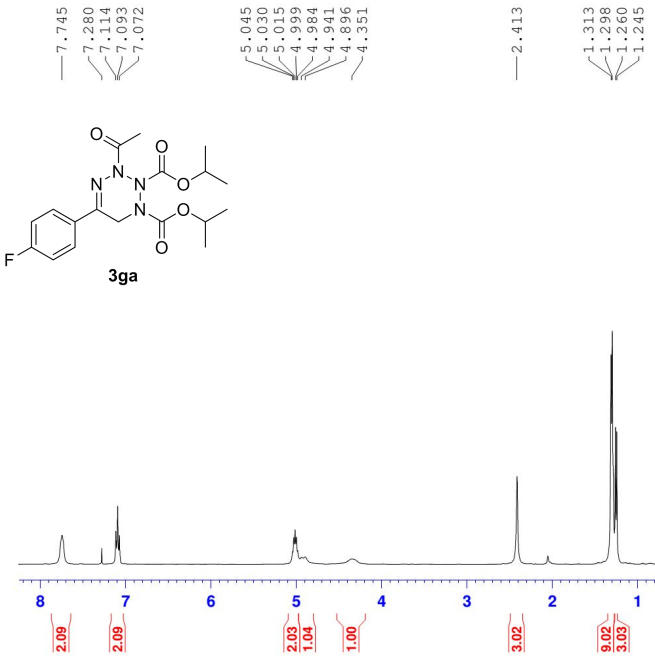

PHL-III-P31-3 13C




```
NAME                 C1
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
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-P31-6




```
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PROCNO                1
Date_          20150915
Time               9.29
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
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
```
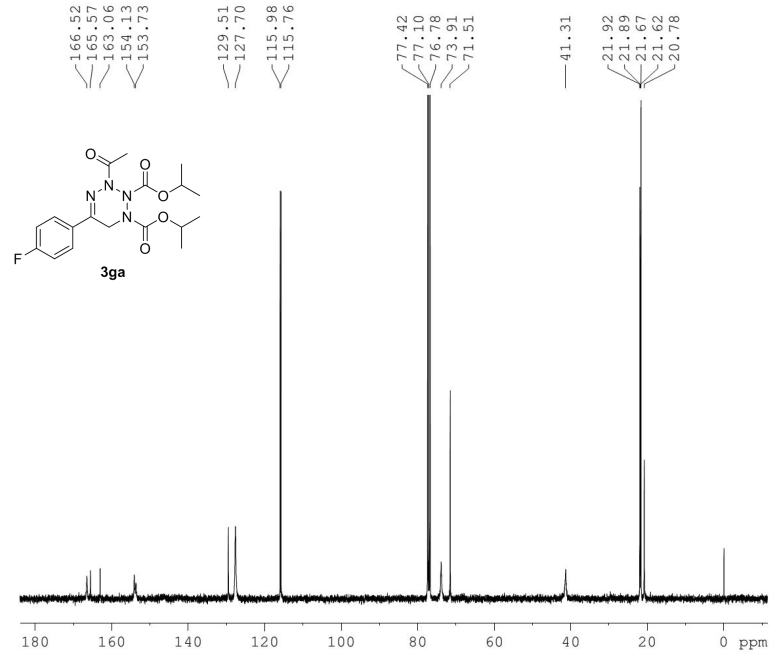

PHL-III-P31-6 13c




```
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PROCNO                1
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Time              15.41
INSTRUM           spect
PROBHD    5 mm PABBO 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
```
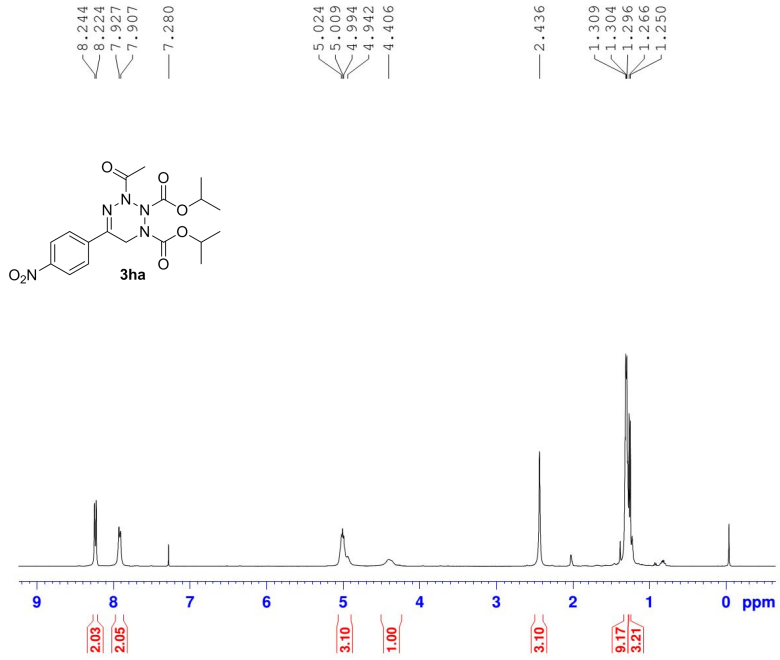

PHL-III-P31-4

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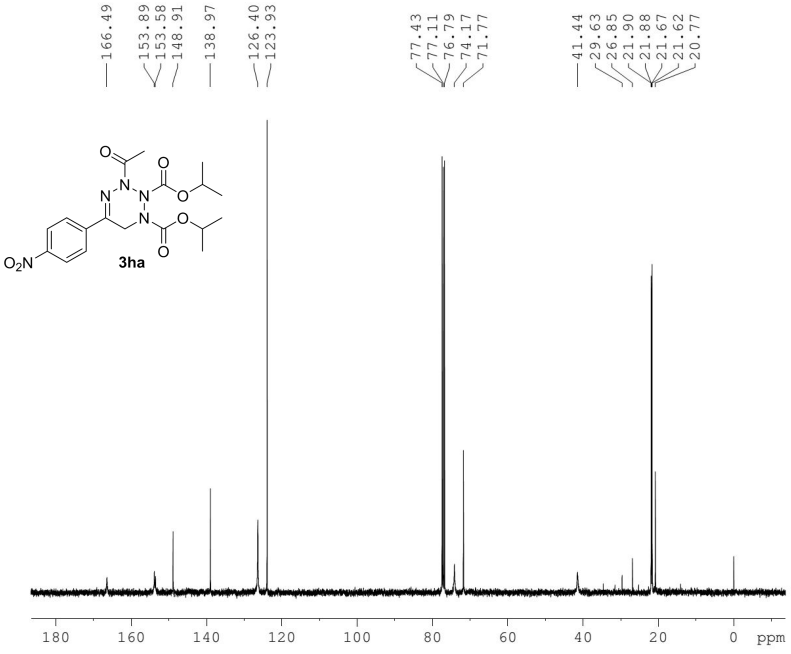
EXPNO              3455
PROCNO                1
Date_          20150914
Time               9.54
INSTRUM           spect
PROBHD   5 mm PABBO BB-
PULPROG            zg30
TD                65536
SOLVENT           CDCl3
NS                    9
DS                    2
SWH            8223.685 Hz
FIDRES        0.125483 Hz
AQ            3.9846387 sec
RG                    4
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-P31-4 13c

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NAME                 C1
EXPNO              3467
PROCNO                1
Date_          20150915
Time              10.27
INSTRUM           spect
PROBHD   5 mm PABBO BB-
PULPROG         zgpg30
TD                65536
SOLVENT           CDCl3
NS                  277
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        0.55629748 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
```
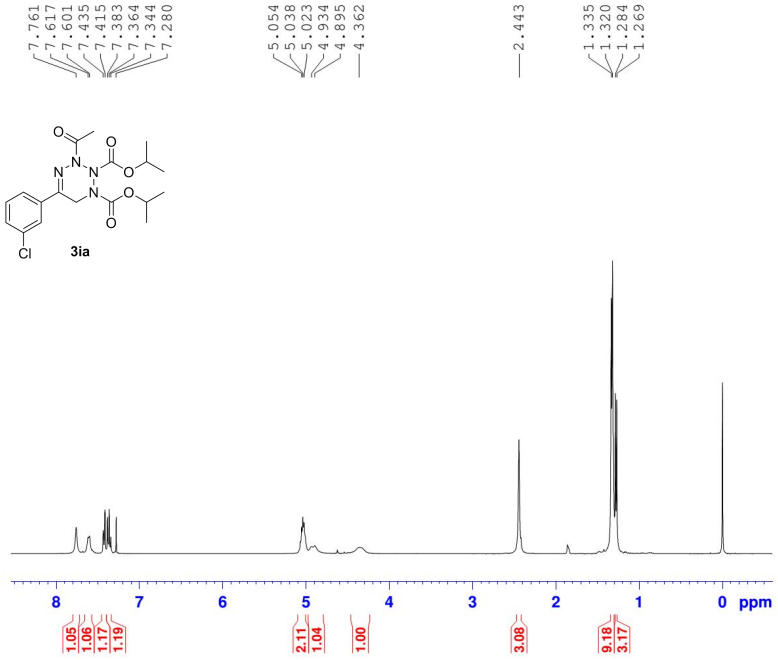
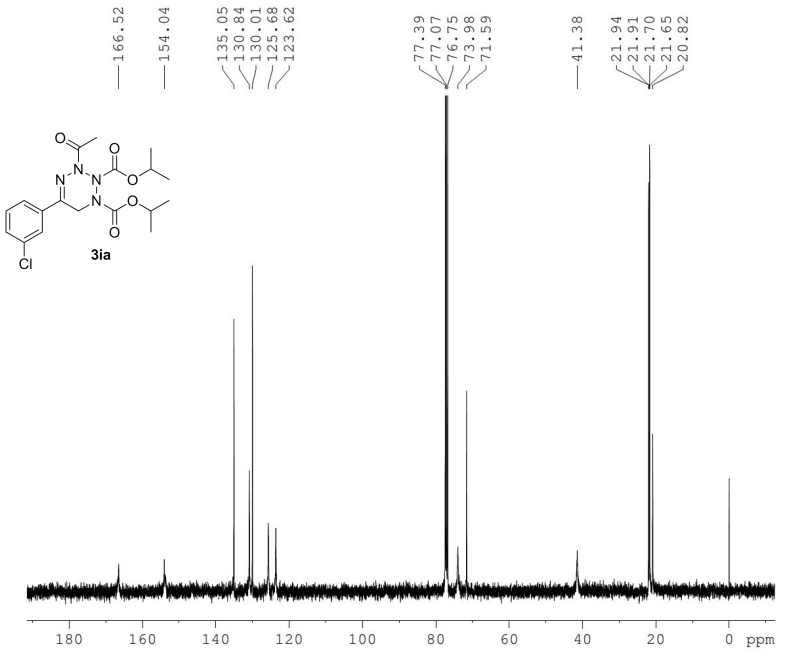

PHL-III-P31-8




```
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
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-P31-8 13C




```
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EXPNO              3461
PROCNO                1
Date_          20150914
Time              15.02
INSTRUM           spect
PROBHD   5 mm PABBO BB-
PULPROG         zgpg30
TD                65536
SOLVENT           CDCl3
NS                  392
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        0.55629748 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
```
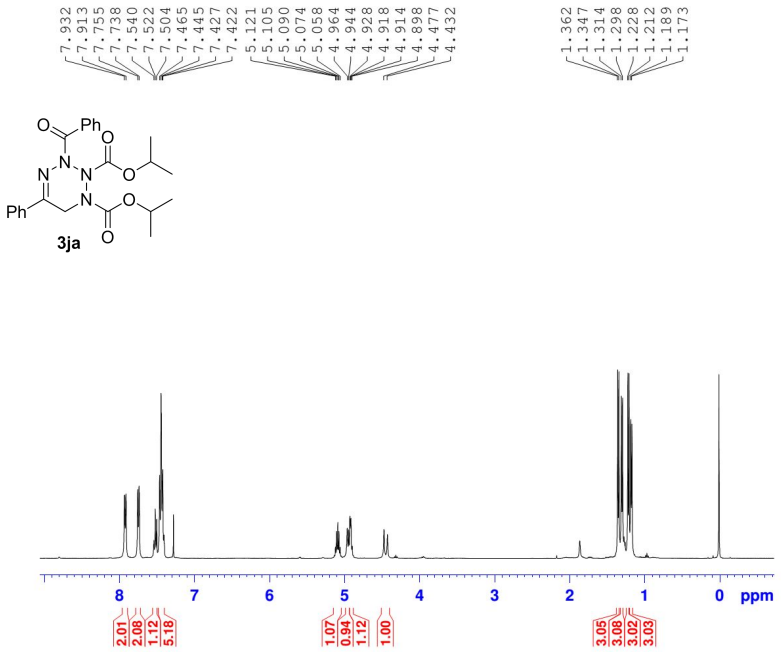
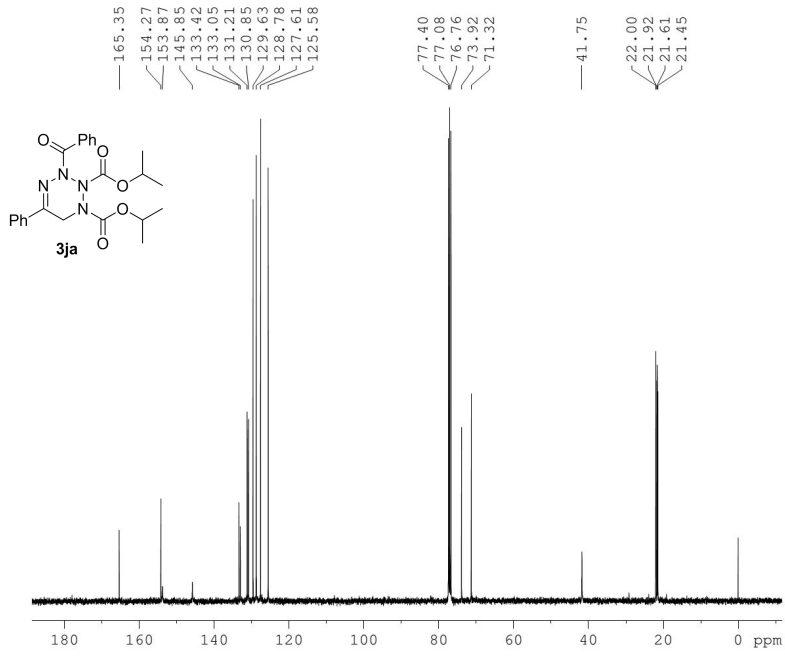

PHL-III-P36-1



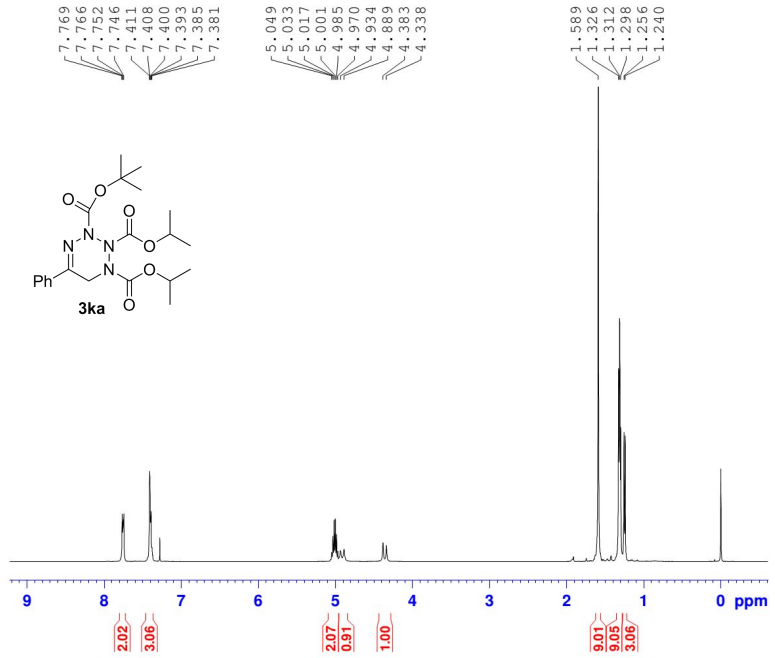
NAME	C1
EXPNO	3439
PROCNO	1
Date_	20150910
Time	11.45
INSTRUM	spect
PROBHD	5 mm PABBO BB-
PULPROG	zg30
TD	65536
SOLVENT	CDCl3
NS	10
DS	2
SWH	8223.685 Hz
FIDRES	0.125483 Hz
AQ	3.9846387 sec
RG	114
DW	60.800 usec
DE	6.50 usec
TE	673.2 K
D1	1.00000000 sec
TD0	1
======== CHANNEL f1 ========	
NUC1	1H
P1	14.50 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-P36-1 13c



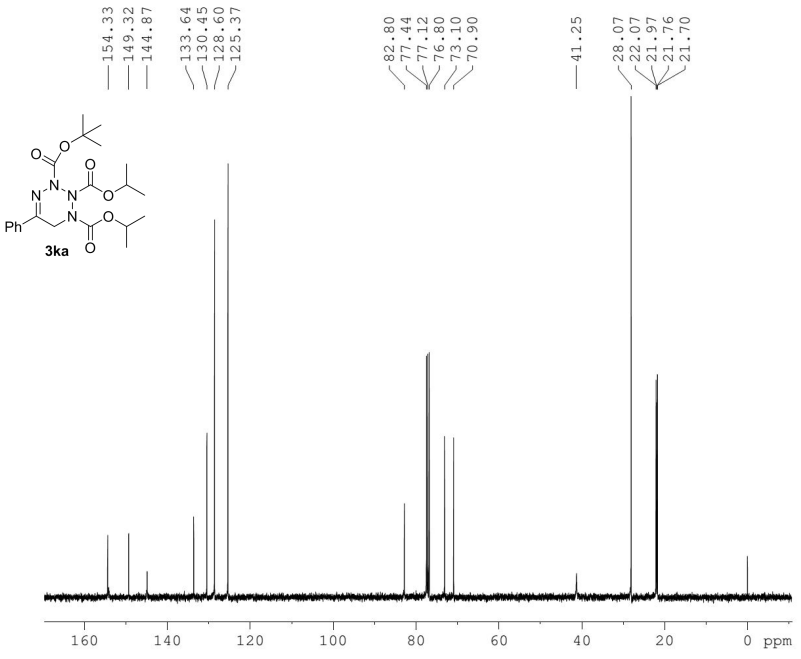
NAME	C1
EXPNO	3466
PROCNO	1
Date_	20150915
Time	9.47
INSTRUM	spect
PROBHD	5 mm PABBO BB-
PULPROG	zgpg30
TD	65536
SOLVENT	CDCl3
NS	504
DS	4
SWH	24038.461 Hz
FIDRES	0.366798 Hz
AQ	1.3631988 sec
RG	2050
DW	20.800 usec
DE	6.50 usec
TE	673.2 K
D1	2.00000000 sec
D11	0.03000000 sec
TD0	1
======== CHANNEL f1 ========	
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-P55-1



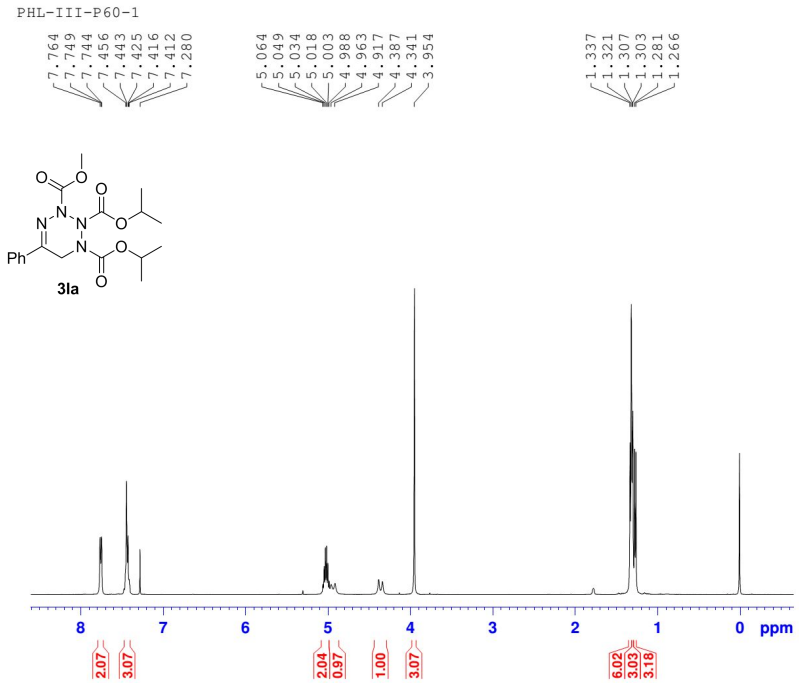

```
NAME                  C1
EXPNO               3563
PROCNO                 1
Date_           20151014
Time               10.25
INSTRUM            spect
PROBHD   5 mm PABBO BB-
PULPROG            zg30
TD                65536
SOLVENT           CDCl3
NS                    10
DS                     2
SWH             8223.685 Hz
FIDRES          0.125483 Hz
AQ              3.9846387 sec
RG                    64
DW                60.800 usec
DE                 6.50 usec
TE                296.8 K
D1             1.00000000 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-P55-1



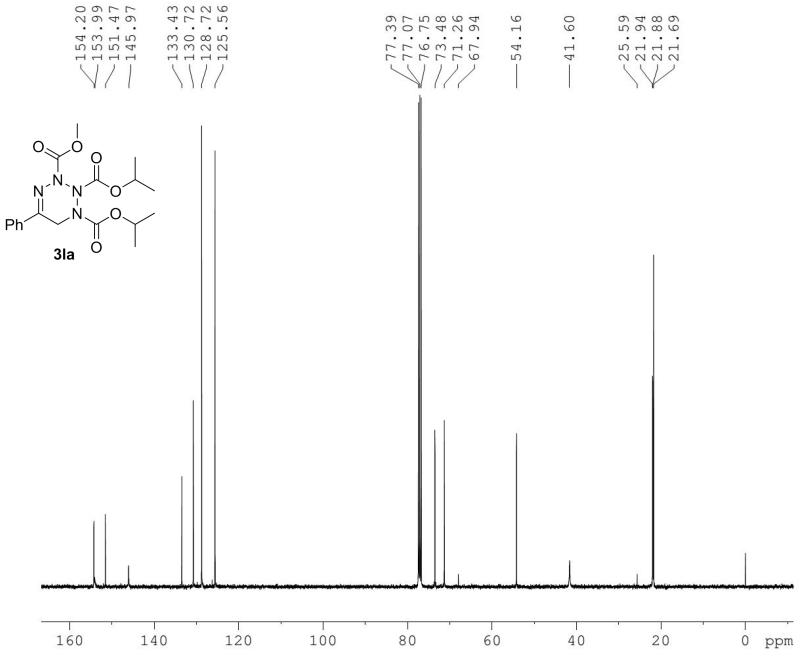

```
NAME                  C1
EXPNO               3573
PROCNO                 1
Date_           20151019
Time               10.33
INSTRUM            spect
PROBHD   5 mm PABBO BB-
PULPROG          zgpg30
TD                65536
SOLVENT           CDCl3
NS                   106
DS                     4
SWH            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
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-P60-1



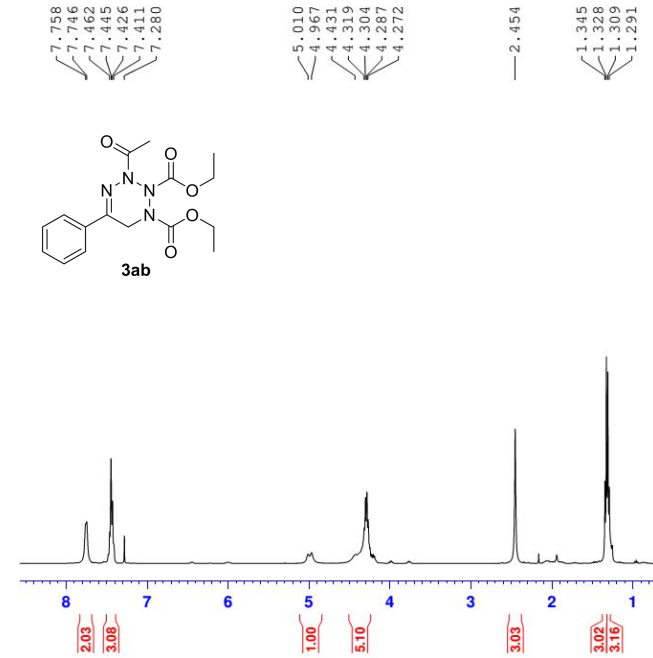

```
NAME                  C1
EXPNO               3526
PROCNO                 1
Date_           20151009
Time               10.01
INSTRUM            spect
PROBHD   5 mm PABBO BB-
PULPROG             zg30
TD                 65536
SOLVENT             CDCl3
NS                    16
DS                     2
SWH             8223.685 Hz
FIDRES          0.125483 Hz
AQ              3.9846387 sec
RG                   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-P60-1



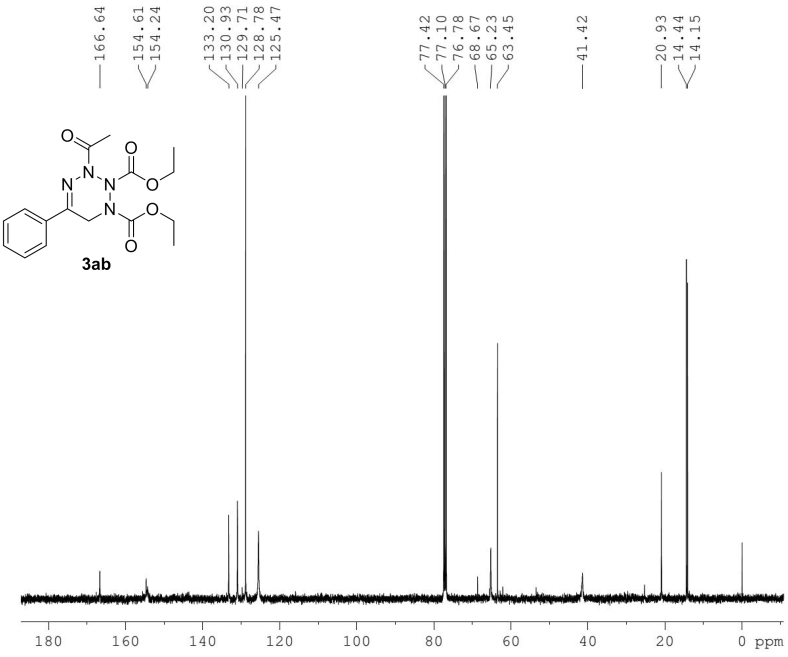

```
NAME                  C1
EXPNO               3544
PROCNO                 1
Date_           20151010
Time               16.40
INSTRUM            spect
PROBHD   5 mm PABBO BB-
PULPROG           zgpg30
TD                 65536
SOLVENT             CDCl3
NS                  1024
DS                     4
SWH            24038.461 Hz
FIDRES          0.366798 Hz
AQ              1.3631988 sec
RG                  2050
DW                20.800 usec
DE                 6.50 usec
TE                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-P35-1



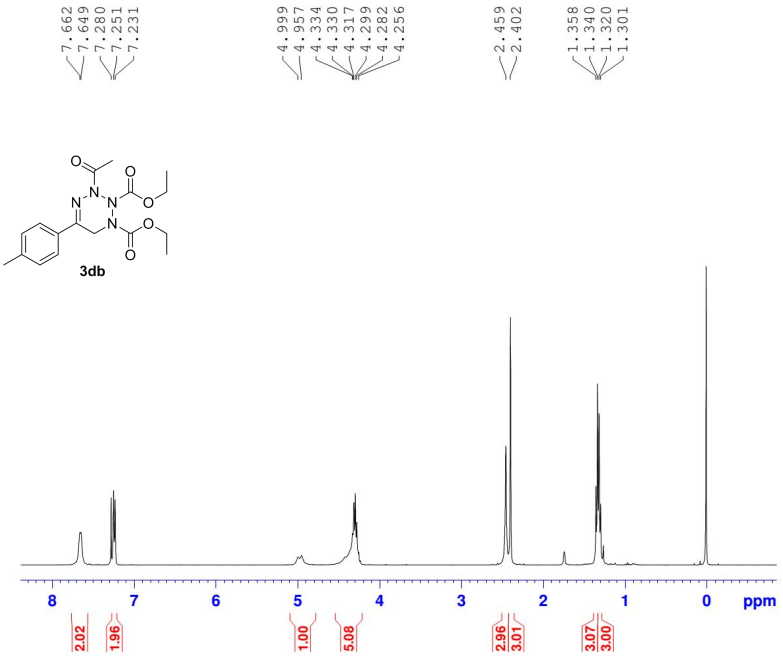

```
NAME                 C1
EXPNO              3537
PROCNO                1
Date_          20151010
Time              14.35
INSTRUM           spect
PROBHD   5 mm PABBO BB-
PULPROG           zg30
TD               65536
SOLVENT          CDCl3
NS                   8
DS                   2
SWH           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



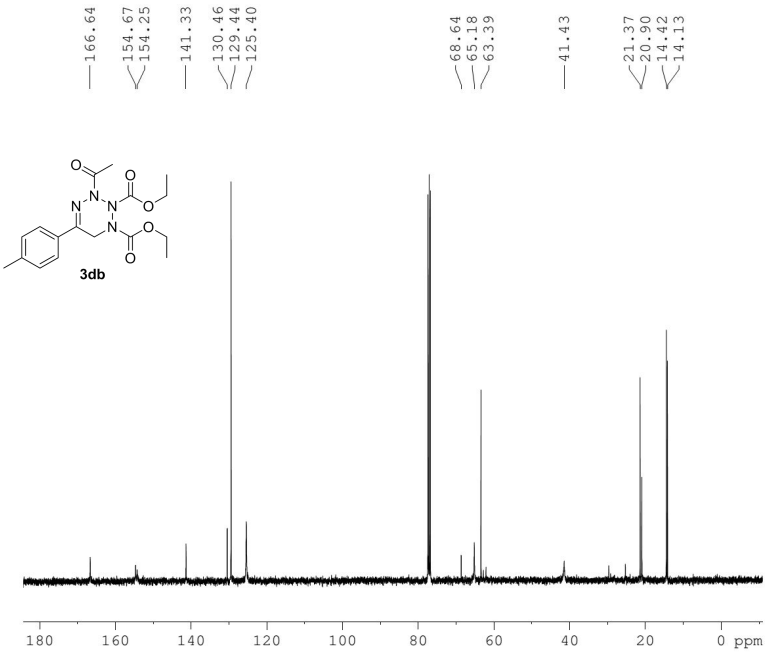

```
NAME                 C1
EXPNO              3481
PROCNO                1
Date_          20150918
Time              9.50
INSTRUM           spect
PROBHD   5 mm PABBO BB-
PULPROG         zgpg30
TD               65536
SOLVENT          CDCl3
NS                 350
DS                   4
SWH          24038.461 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



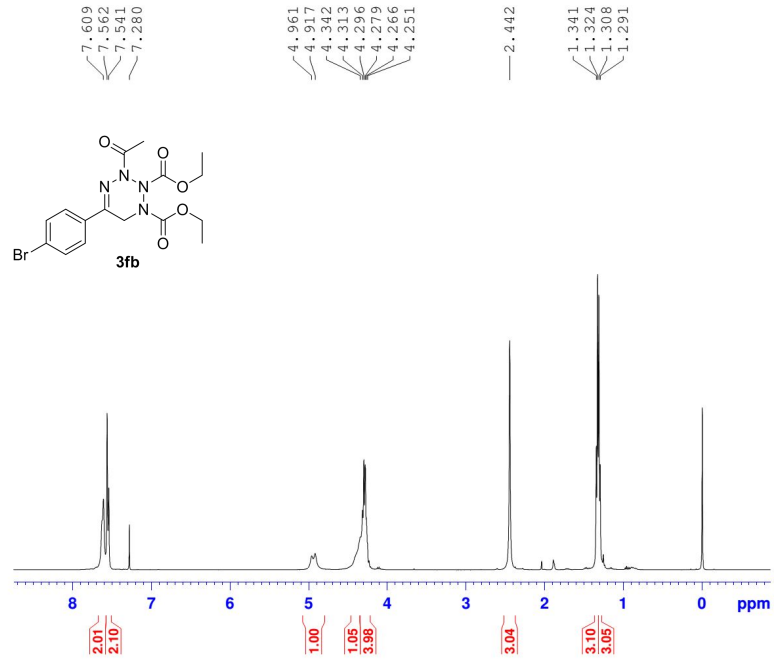

```
NAME                 C1
EXPNO              3528
PROCNO                1
Date_          20151009
Time              10.19
INSTRUM           spect
PROBHD   5 mm PABBO BB-
PULPROG             zg30
TD                 65536
SOLVENT            CDCl3
NS                    16
DS                     2
SWH             8223.685 Hz
FIDRES          0.125483 Hz
AQ              3.9846387 sec
RG                   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-P35-2 13C



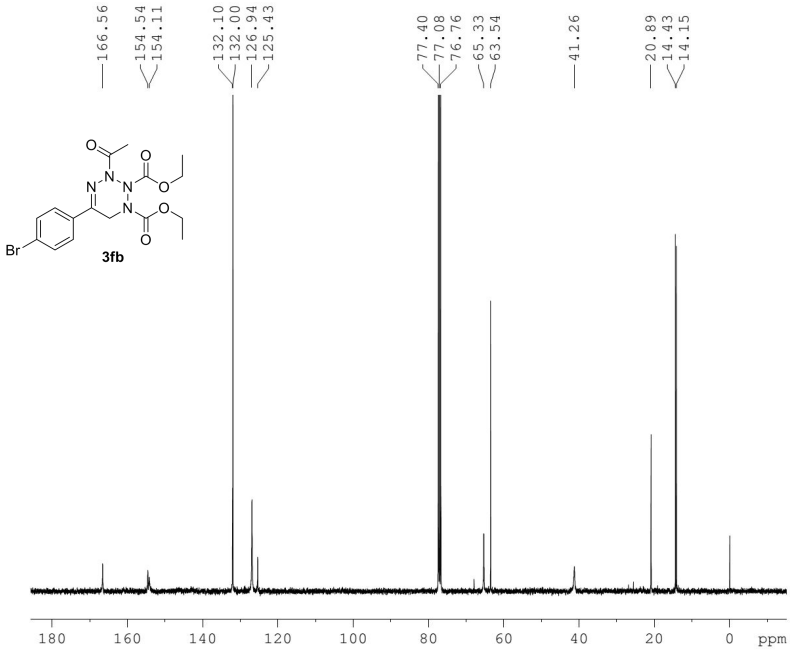

```
NAME                 C1
EXPNO              3359
PROCNO                1
Date_          20150727
Time              11.48
INSTRUM           spect
PROBHD   5 mm PABBO BB-
PULPROG           zgpg30
TD                 65536
SOLVENT            CDCl3
NS                   309
DS                     4
SWH            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
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-P35-3



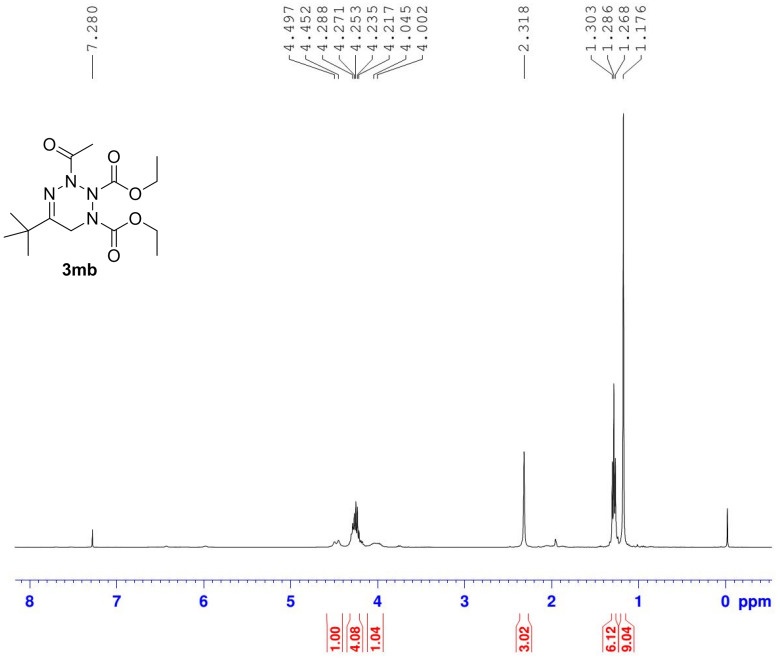

```
NAME                 C1
EXPNO              3527
PROCNO                1
Date_          20151009
Time              10.15
INSTRUM           spect
PROBHD    5 mm PABBO BB-
PULPROG            zg30
TD                65536
SOLVENT           CDCl3
NS                   16
DS                    2
SWH            8223.685 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-p35-3 13C



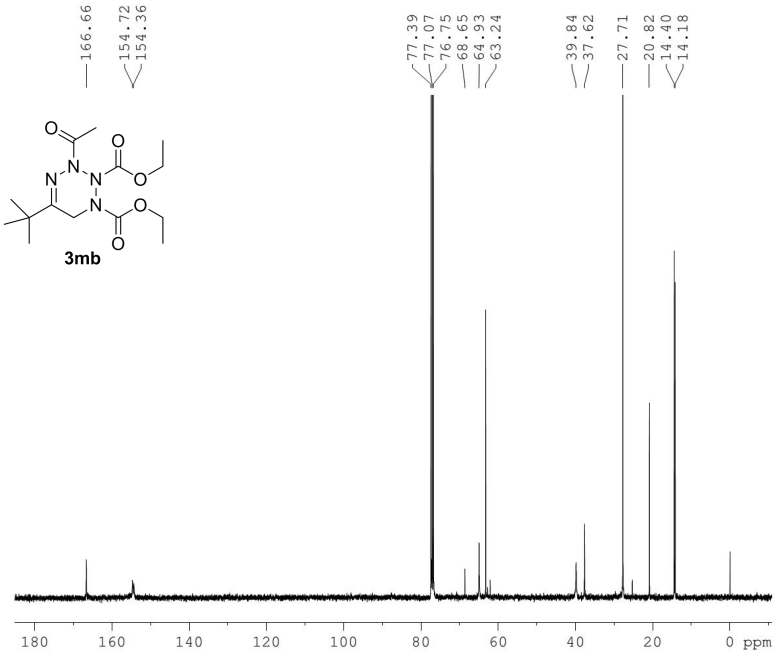

```
NAME                 C1
EXPNO              3491
PROCNO                1
Date_          20150923
Time               8.58
INSTRUM           spect
PROBHD    5 mm PABBO BB-
PULPROG          zgpg30
TD                65536
SOLVENT           CDCl3
NS                 1024
DS                    4
SWH           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
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-P35-4



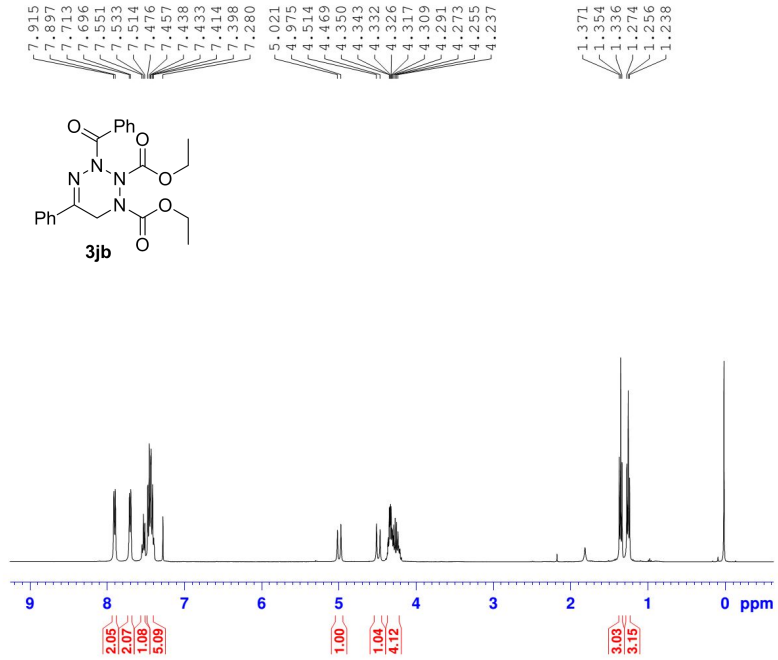

```
NAME                 C1
EXPNO              3470
PROCNO                1
Date_          20150916
Time              14.29
INSTRUM           spect
PROBHD    5 mm PABBO BB-
PULPROG            zg30
TD                65536
SOLVENT           CDCl3
NS                    6
DS                    2
SWH            8223.685 Hz
FIDRES         0.125483 Hz
AQ             3.9846387 sec
RG                 90.5
DW                60.800 usec
DE                 6.50 usec
TE                297.3 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-P35-4 13c



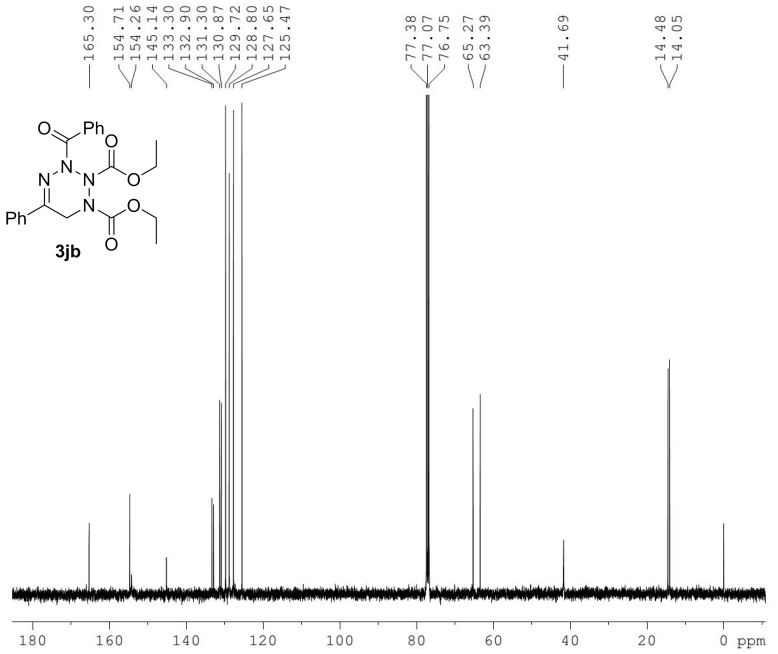

```
NAME                 C1
EXPNO              3478
PROCNO                1
Date_          20150917
Time              14.30
INSTRUM           spect
PROBHD    5 mm PABBO BB-
PULPROG          zgpg30
TD                65536
SOLVENT           CDCl3
NS                  721
DS                    4
SWH           24038.461 Hz
FIDRES         0.366798 Hz
AQ             1.3631988 sec
RG                 2050
DW                20.800 usec
DE                 6.50 usec
TE                296.9 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
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-P36-2

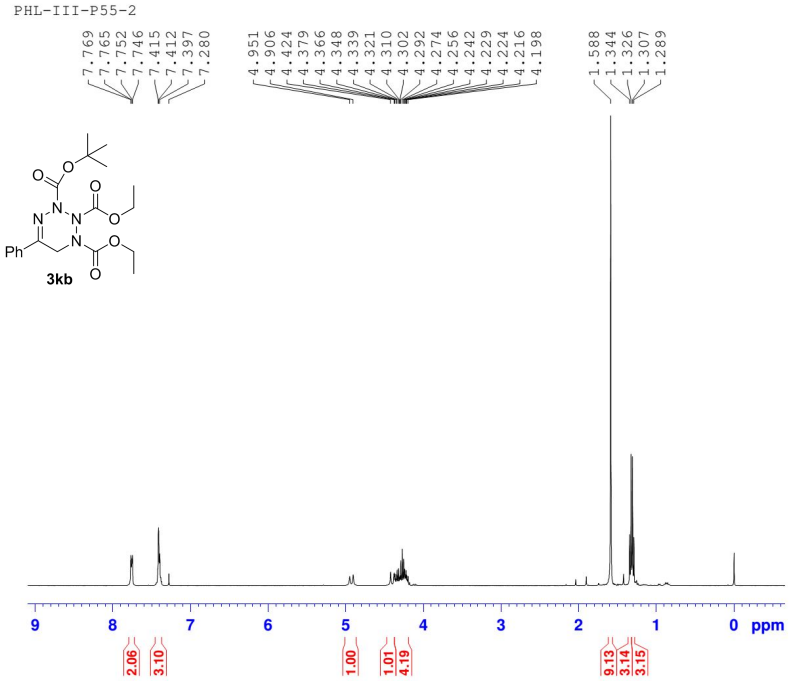



```
NAME                 C1
EXPNO              3452
PROCNO                1
Date_          20150910
Time              16.37
INSTRUM           spect
PROBHD   5 mm PABBO BB-
PULPROG            zg30
TD                65536
SOLVENT           CDCl3
NS                   16
DS                    2
SWH            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
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
```

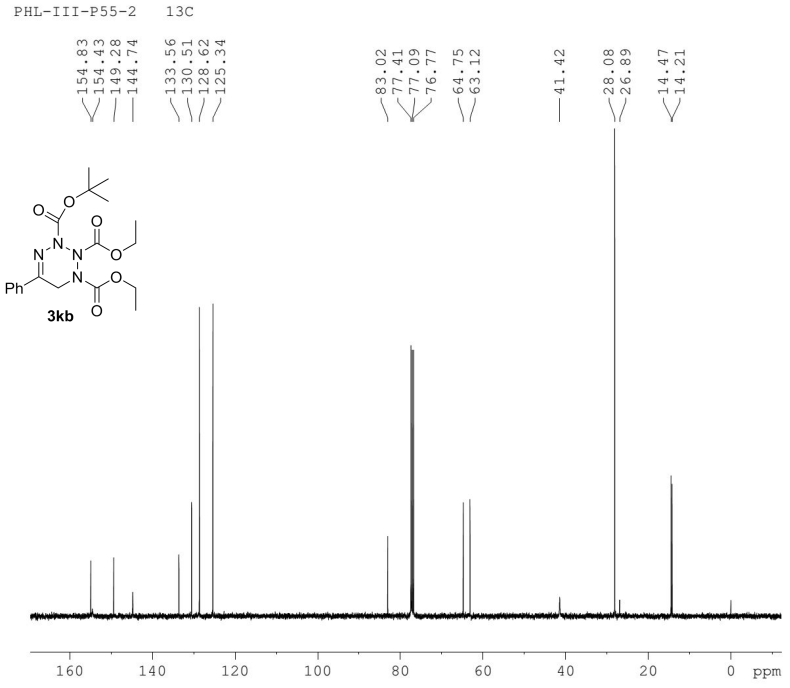
PHL-III-P36-2 13c




```
NAME                 C1
EXPNO              3468
PROCNO                1
Date_          20150915
Time              10.41
INSTRUM           spect
PROBHD   5 mm PABBO BB-
PULPROG          zgpg30
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
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
```

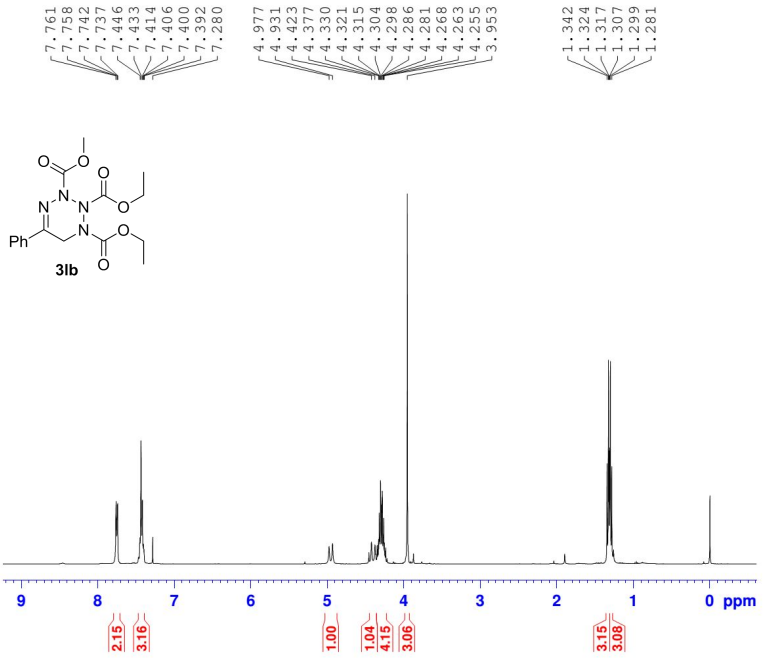



```
NAME                 C1
EXPNO              3554
PROCNO                1
Date_          20151012
Time              10.56
INSTRUM           spect
PROBHD    5 mm PABBO BB-
PULPROG            zg30
TD                65536
SOLVENT           CDCl3
NS                    6
DS                    2
SWH            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
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
```



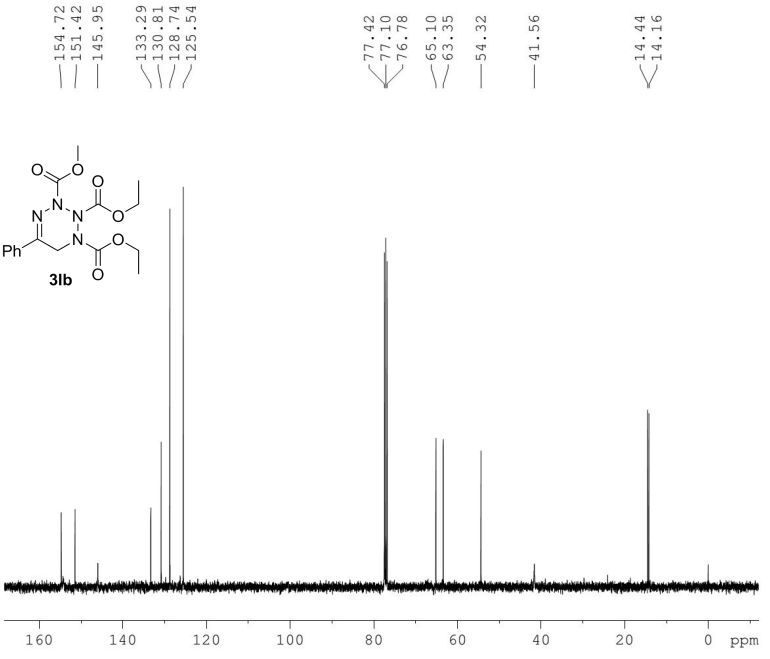

```
NAME                 C1
EXPNO              3557
PROCNO                1
Date_          20151012
Time              14.47
INSTRUM           spect
PROBHD    5 mm PABBO BB-
PULPROG          zgpg30
TD                65536
SOLVENT           CDCl3
NS                  128
DS                    4
SWH           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
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-P60-2



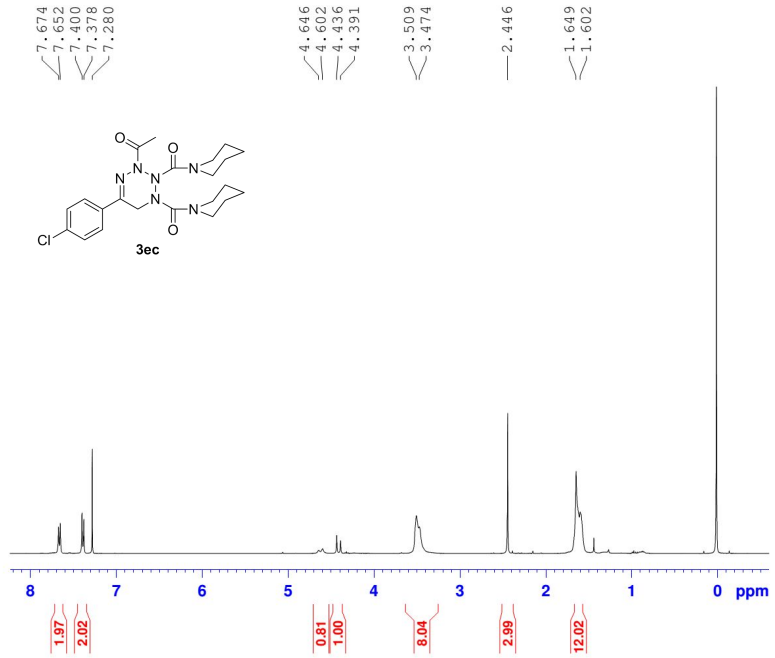

```
NAME                 C1
EXPNO              3518
PROCNO                1
Date_          20151008
Time              15.01
INSTRUM           spect
PROBHD    5 mm PABBO BB-
PULPROG            zg30
TD                65536
SOLVENT           CDCl3
NS                    4
DS                    2
SWH            8223.685 Hz
FIDRES        0.125483 Hz
AQ            3.9846387 sec
RG                  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-P60-2 13C



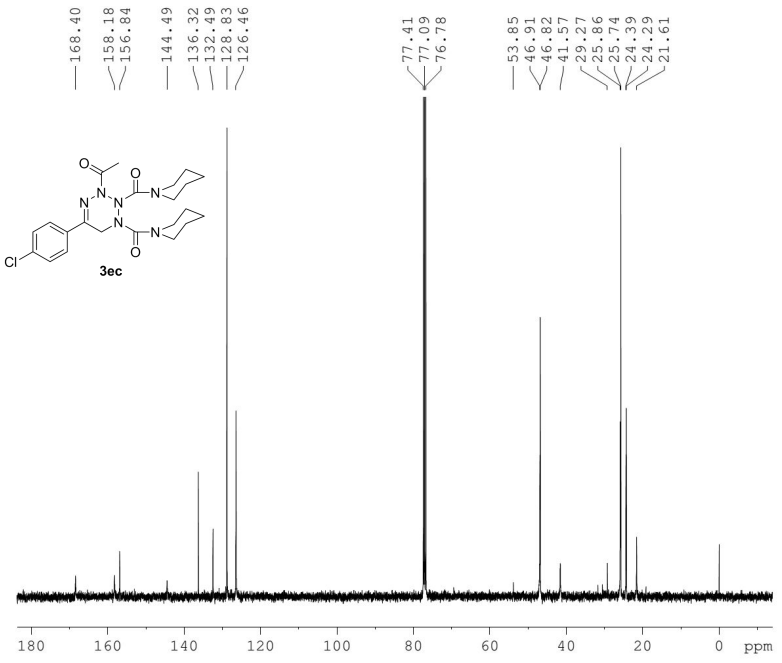

```
NAME                 C1
EXPNO              3530
PROCNO                1
Date_          20151009
Time              10.38
INSTRUM           spect
PROBHD    5 mm PABBO BB-
PULPROG          zgpg30
TD                65536
SOLVENT           CDCl3
NS                   64
DS                    4
SWH           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       0.55629748 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-P49-1



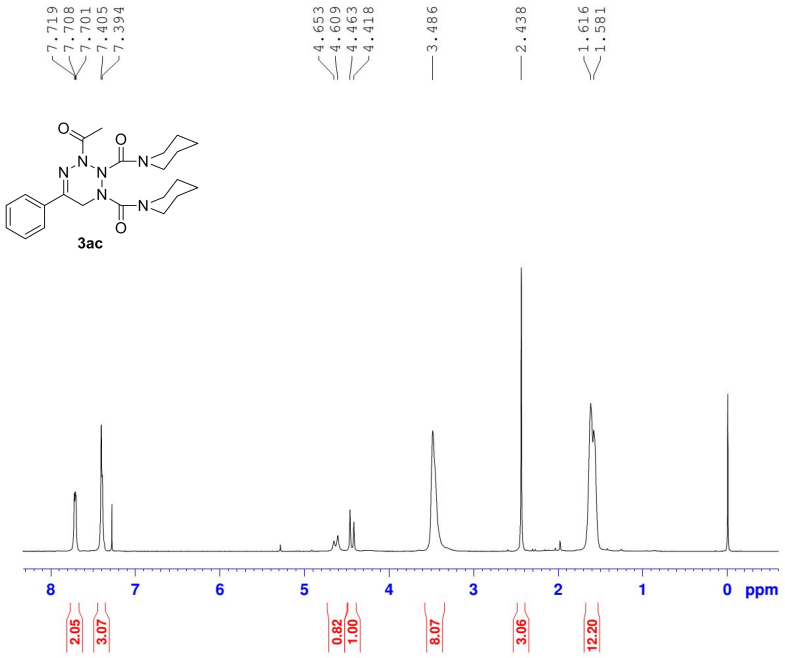

```
NAME                 C1
EXPNO              3507
PROCNO                1
Date_          20150930
Time              14.26
INSTRUM            spect
PROBHD   5 mm PABBO BB-
PULPROG            zg30
TD                65536
SOLVENT           CDC13
NS                   16
DS                    2
SWH            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
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-P49-1



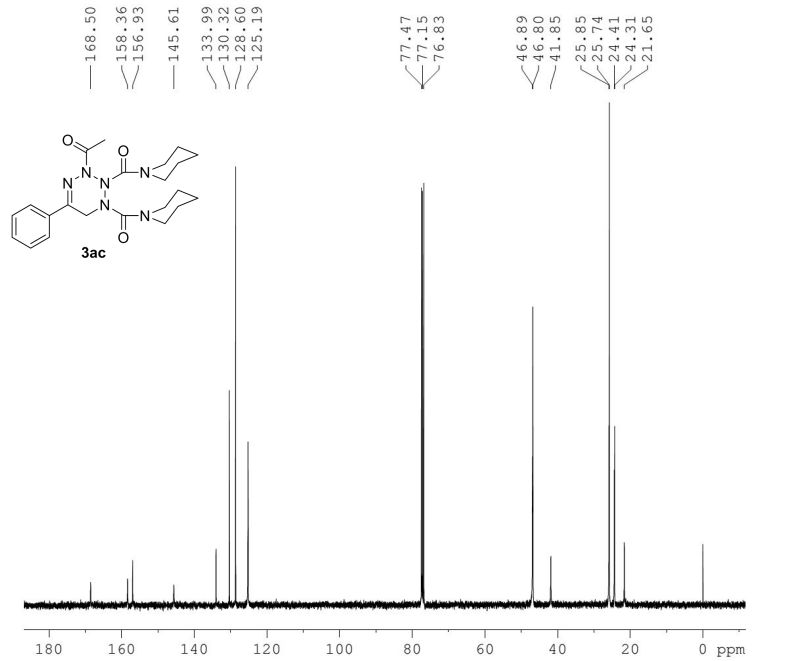

```
NAME                 C1
EXPNO              3543
PROCNO                1
Date_          20151010
Time              16.16
INSTRUM            spect
PROBHD   5 mm PABBO BB-
PULPROG         zgpg30
TD                65536
SOLVENT           CDC13
NS                  298
DS                    4
SWH           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
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-P38-1



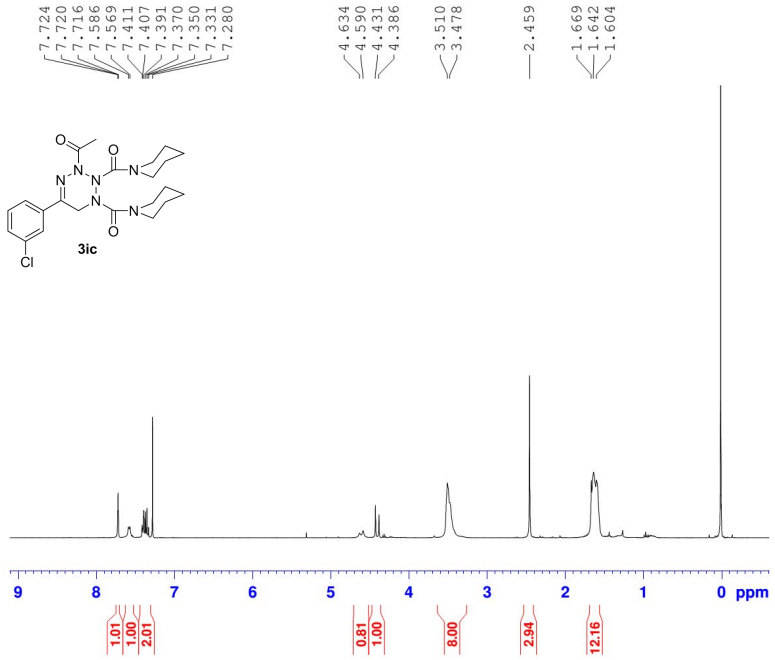

```
NAME                 C1
EXPNO              3561
PROCNO                1
Date_          20151014
Time              10.13
INSTRUM           spect
PROBHD    5 mm PABBO BB-
PULPROG            zg30
TD                65536
SOLVENT           CDCl3
NS                    6
DS                    2
SWH            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           1.00000000 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-P38-1



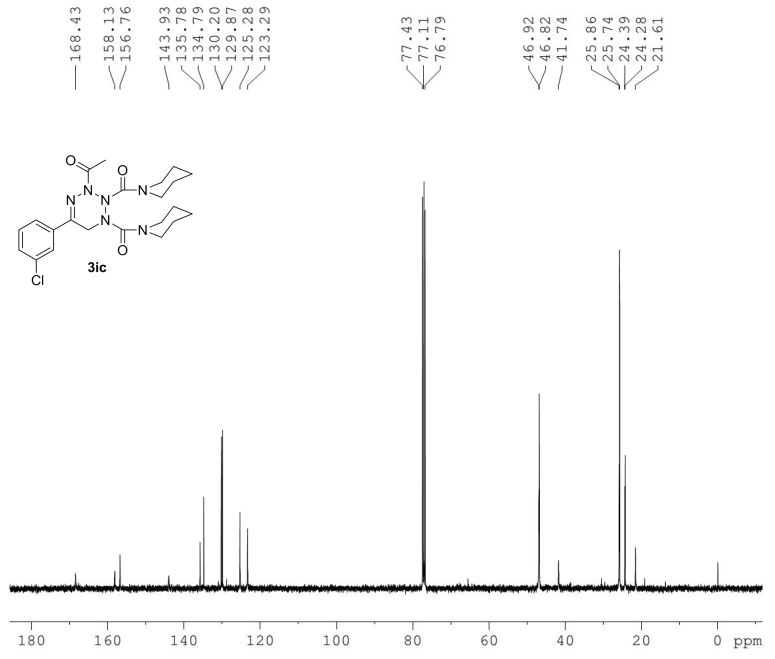

```
NAME                 C1
EXPNO              3572
PROCNO                1
Date_          20151019
Time              10.14
INSTRUM           spect
PROBHD    5 mm PABBO BB-
PULPROG         zgpg30
TD                65536
SOLVENT           CDCl3
NS                  260
DS                    4
SWH           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           2.00000000 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-P50-2



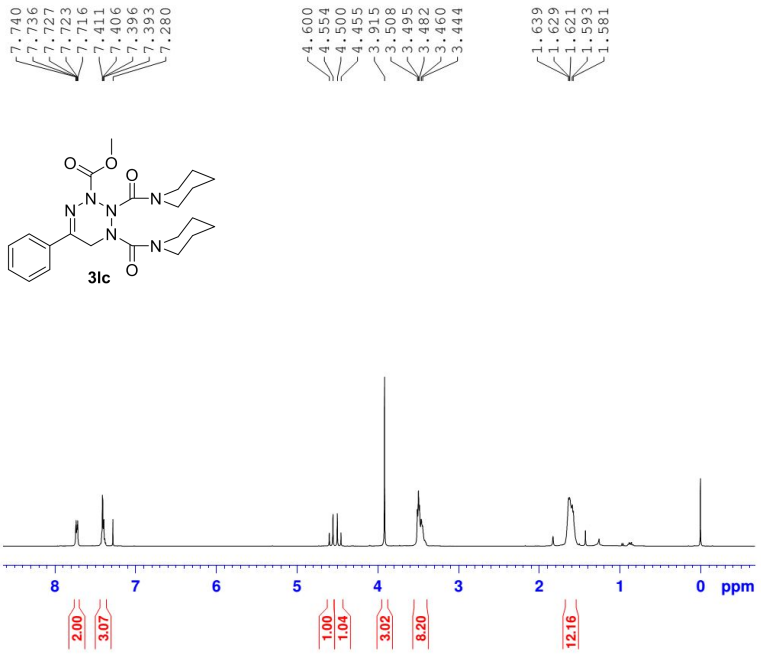

```
NAME                 C1
EXPNO              3517
PROCNO                1
Date_          20151008
Time              14.56
INSTRUM           spect
PROBHD   5 mm PABBO BB-
PULPROG            zg30
TD                65536
SOLVENT           CDCl3
NS                    5
DS                    2
SWH            8223.685 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



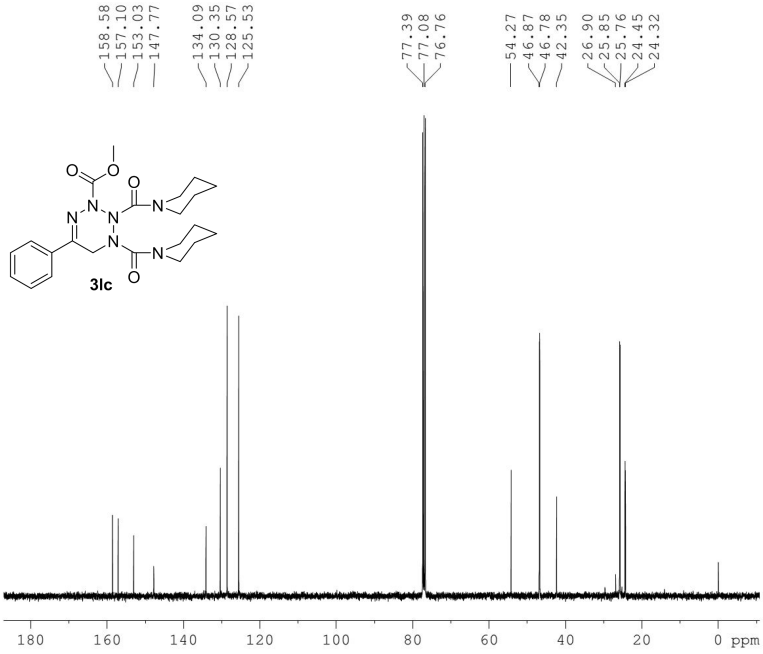

```
NAME                 C1
EXPNO              3550
PROCNO                1
Date_          20151012
Time              10.13
INSTRUM           spect
PROBHD   5 mm PABBO BB-
PULPROG           zgpg30
TD                65536
SOLVENT           CDCl3
NS                  230
DS                    4
SWH           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
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-P64-2




```
NAME                 C1
EXPNO              3598
PROCNO                1
Date_          20151022
Time              16.31
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                  128
DW             60.800 usec
DE              6.50 usec
TE              293.6 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-P64-2 13C




```
NAME                 C1
EXPNO              3610
PROCNO                1
Date_          20151029
Time              17.09
INSTRUM           spect
PROBHD   5 mm PABBO BB-
PULPROG          zgpg30
TD                65536
SOLVENT           CDCl3
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
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
```

2. X-Ray crystal data of compound 3aa

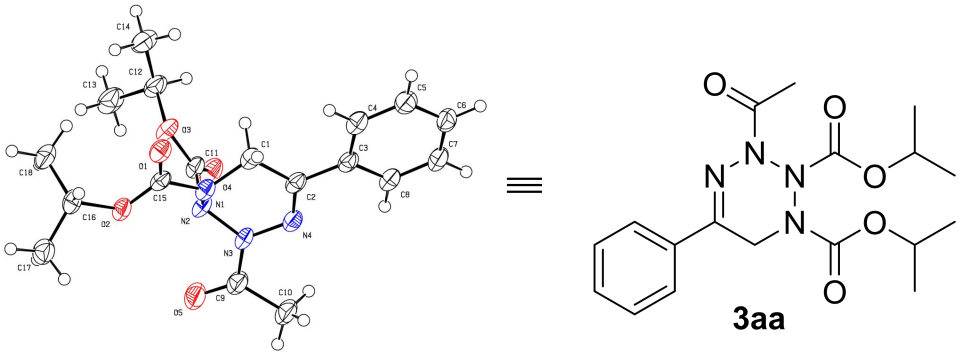


Figure 1.X-ray single crystal structure of 3aa (with thermal ellipsoils shown at the 50% probability level)

Identification code	3aa
Empirical formula	$C_{18}H_{24}N_4O_5$
Formula weight	376.41
Temperature	113(2) K
Wavelength	0.71073 A
Crystal system, space group	Monoclinic, P2(1)/c
Unit cell dimensions	a = 10.454(5) A alpha = 90 deg. b = 21.985(9) A beta = 102.524(11) deg. c = 8.731(4) A gamma = 90 deg.
Volume	1958.8(15) A^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>2sigma(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.A^-3
CCDC deposition number	1433224