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Base-Promoted Cascade Reaction of α,β -Unsaturated *N*-Tosylhydrazones with *o*-Hydroxybenzyl Alcohols: Highly Regioselective Synthesis of *N*-sec-Alkylpyrazoles

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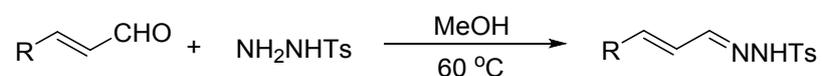
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I-General information

¹H NMR spectra were determined on a 400 MHz spectrometer in CDCl₃ solution. Chemical shifts are expressed in parts per million (δ), and the signals were reported as s (singlet), d (doublet), t (triplet), m (multiplet), and coupling constants J were given in Hz. ¹³C NMR spectra were recorded at 100 MHz in CDCl₃ solution. Chemical shifts are expressed in parts per million (δ) and are referenced to CDCl₃ (δ = 77.16) as an internal standard. TLC was done on silica gel coated glass slide (Silica gel G for TLC). Silica gel (60-120 mesh) was used for column chromatography. Petroleum ether refers to the fraction boiling in the range of 60-90 °C unless otherwise mentioned. All solvents were dried and distilled before use. Commercially available substrates were freshly distilled before the reaction. Solvents, reagents and chemicals were commercially purchased. All reactions involving moisture sensitive reactants were executed using oven-dried glassware. The α,β-unsaturated tosylhydrazones **1** and ortho-hydroxymethyl phenols **2** were synthesized according to the reported procedure.¹⁻⁴ The relative configuration of **4I** was assigned by the X-ray analysis.

II-Experimental Section

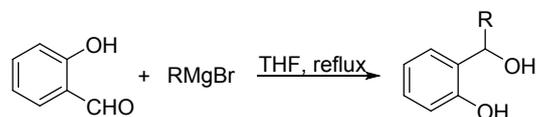
1. Synthesis of α,β -unsaturated tosylhydrazones **1**



α,β -unsaturated tosylhydrazones were prepared according to literature procedure.³

A solution of pure 4-methylbenzenesulfonylhydrazide (5 mmol) in methanol (5 mL) was stirred and heated to 60 °C until the 4-methylbenzenesulfonylhydrazide was completely dissolved. Then α,β -unsaturated aldehydes were slowly dropped to the mixture. After approximately 30 min, the crude products as precipitates were obtained. The precipitates were washed by petroleum ether and dried in *vacuo* to afford the pure products **1a-k**.

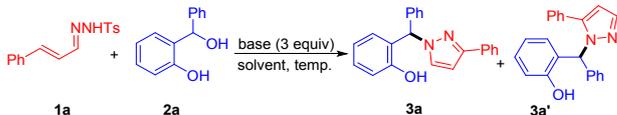
2. Synthesis of *ortho*-hydroxymethyl phenols **2**



General procedure for *ortho*-hydroxymethyl phenols.⁴ RMgBr (2.0 equiv) was added into a solution of salicylaldehyde (1 equiv) in THF at 0 °C. The mixture was refluxed for about 5 hours. The reaction was quenched with 2mL saturated NH₄Cl solution, and the mixture solution was transferred to a separatory funnel. The aqueous layer was extracted with 4mL EtOAc. The organic layer was separated. The combined organic layers were dried over Na₂SO₄ and concentrated under reduced pressure. The residue was purified by recrystallization to provide the desired products **2** (40-80% yields).

III-Optimization of the Reaction conditions

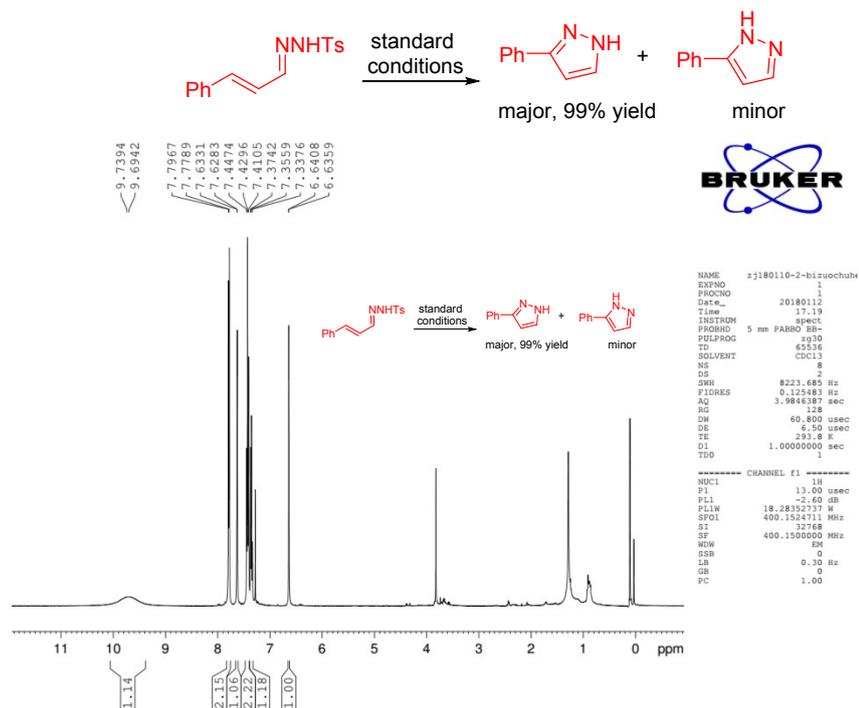
Table 1. Optimization of reaction conditions

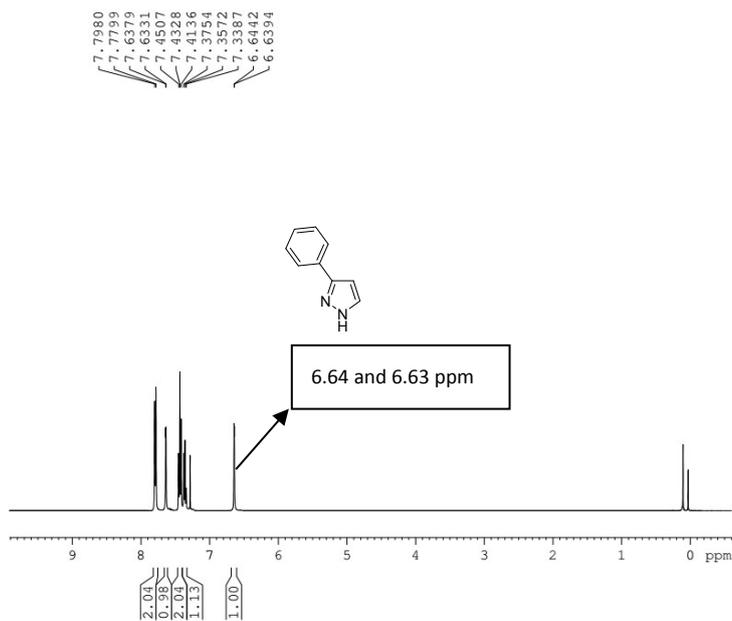


entry ^a	base	solvent/temp(°C)	3a yield (%) ^b	3a/3a' ^c
1	Cs ₂ CO ₃	1,4-dioxane/60	70	>20:1
2	K ₂ CO ₃	1,4-dioxane/60	68	>20:1
3	Na ₂ CO ₃	1,4-dioxane/60	55	>20:1
4	Cs ₂ CO ₃	THF/60	62	>20:1
5	Cs ₂ CO ₃	Toluene/60	48	>20:1
6	Cs ₂ CO ₃	(CH ₂ Cl) ₂ /60	23	>20:1
7 ^[d]	Cs ₂ CO ₃	1,4-dioxane/90	92	>20:1

^aReaction conditions: α,β -unsaturated *N*-tosylhydrazone **1a** (0.22 mmol, 1.1 equiv), *ortho*-hydroxybenzyl alcohol **2a** (0.2 mmol, 1 equiv), base (0.6 mmol, 3.0 equiv) in 1 mL solvent for 12 hours. ^bIsolated yield. ^cDetermined by ¹H NMR analysis of the crude reaction mixture. ^dFor 4 hours.

¹H NMR spectroscopy of the crude reaction mixture (1a)



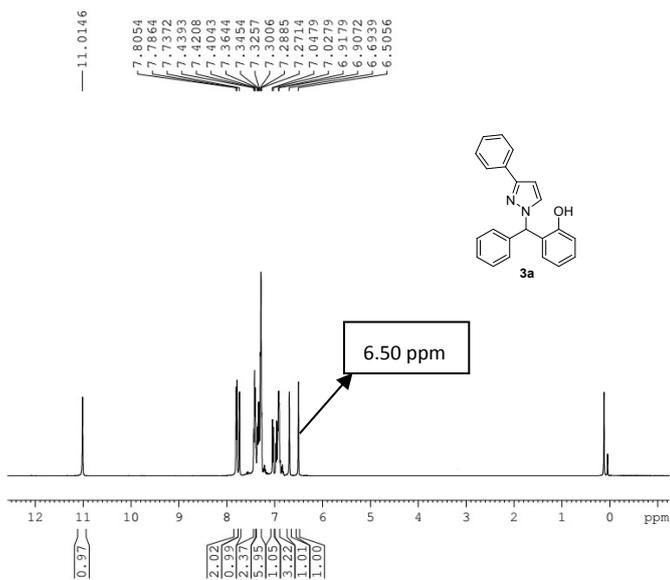


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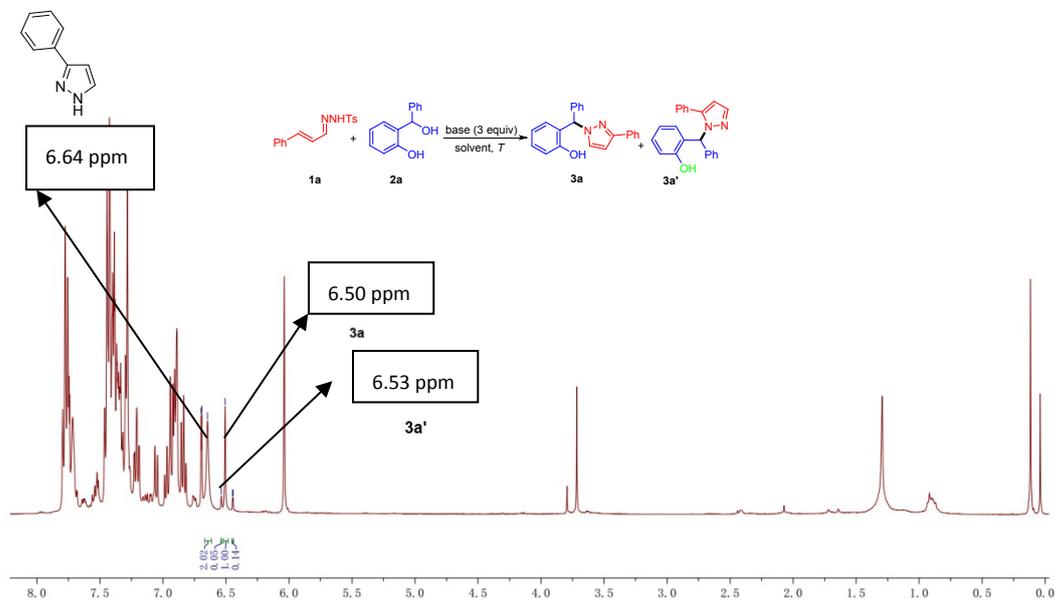
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¹H NMR spectroscopy of the crude reaction mixture (1a +2a)

Zj171226-chun-xizong
Zj171226



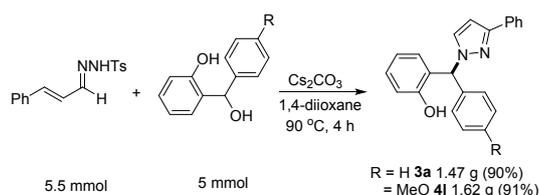
the value of 3a/3a' was about > 20:1.

IV- General Procedure

1. Procedure for the cascade Reaction

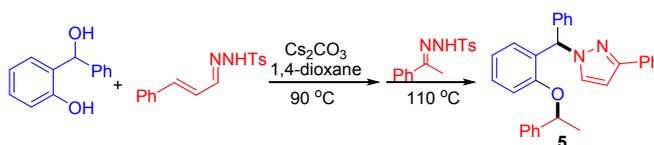
α,β -unsaturated *N*-tosylhydrazones **1** (0.22 mmol), *ortho*-hydroxymethyl phenols **2** (0.2 mmol), Cs₂CO₃ (0.6 mmol) and 1,4-dioxane (1 mL) were added into a tube. The mixture was stirred at 90 °C for 4-8 hours. After cooling to room temperature, the mixture was quenched with NH₄Cl saturated aqueous solution (2 mL) and extracted with CH₂Cl₂ (3 × 2 mL). The combined organic phase was dried over Na₂SO₄, and then organic solvents were removed in *vacuo*. The residue was purified by flash column chromatography on silica gel with ethyl acetate/petroleum ether (1:15-1:10, V/V) as the eluent, affording the desired products **3** and **4**, which were characterized by ¹H and ¹³C NMR spectroscopy, HRMS.

2. Procedure for the gram scale reaction (for Scheme 5)



α,β -unsaturated tosylhydrazones **1** (5.5 mmol), *ortho*-hydroxymethyl phenols **2** (5 mmol), Cs₂CO₃ (15 mmol) were suspended in 1,4-dioxane (10 mL) in a flask. The resulting solution was stirred at 90 °C for 4 h. After cooling to room temperature, the mixture was quenched with NH₄Cl saturated aqueous solution (40 mL) and extracted with CH₂Cl₂ (3 × 40 mL). The combined organic phase was dried over Na₂SO₄, and then the solvent was removed in *vacuo* to give a residue. The residue was purified by flash column chromatography (EtOAc/hexane) to give the compounds **3a** (90% yield) or **4I** (91% yield).

3. Synthesis of compound 5 (for Scheme 4)

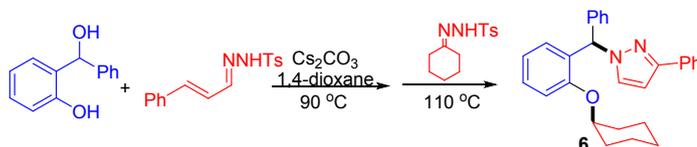


The compound **5** was prepared according to the reported literature procedure.⁵

α,β -unsaturated tosylhydrazones **1** (0.11 mmol), *ortho*-hydroxymethyl phenols **2** (0.1 mmol),

Cs₂CO₃ (0.4 mmol) and 1,4-dioxane (0.5 mL) were added into a tube. The mixture was stirred at 90 °C for 4 h. Next, (E)-4-methyl-N'-(1-phenylethylidene)benzenesulfonylhydrazide (0.1 mmol) was added. The system was heated at 110 °C with stirring and refluxing. After the reaction was completed, the reaction mixture was cooled to room temperature, and the solvent was eliminated under reduced pressure. 2 mL NaOH solution (2M) and 2 mL dichloromethane were added to the residue. The aqueous phase was extracted three times with dichloromethane. The combined organic layer was washed with brine and dried over MgSO₄. The organic solvent was removed under reduced pressure. The residue was purified by chromatography on silica gel or alumina eluted with EtOAc/hexane to afford the product **5** in 85% yield.

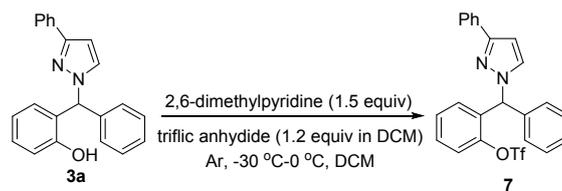
4. Synthesis of compound **6** (for Scheme 4)



The compound **6** was prepared according to the literature procedure.⁵

α,β -unsaturated tosylhydrazones **1** (0.11 mmol), ortho-hydroxymethyl phenols **2** (0.1 mmol), Cs₂CO₃ (0.4 mmol) and 1,4-dioxane (0.5 mL) were added into a tube. The mixture was stirred at 90 °C for 4 h. Next, (E)-4-methyl-N'-(1-phenylethylidene)benzenesulfonylhydrazide (0.1 mmol) was added. The system was heated at 110 °C with stirring and refluxing. After the reaction was completed, the crude reaction mixture was cooled to room temperature. The solvent was eliminated. 2 mL NaOH solution (2M) and 2 mL dichloromethane were added. The layers were separated. The aqueous phase was extracted with dichloromethane three times. The combined organic layer was washed with brine and dried over MgSO₄. After the solvent was removed under reduced pressure, the residue was purified by chromatography on silica gel with EtOAc/hexane. The product **6** was obtained in 80% yield.

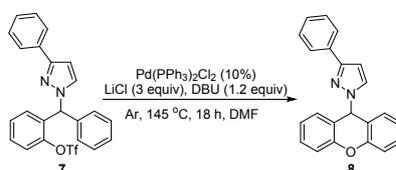
5. Synthesis of compound **7** (for Scheme 5 b)



The compound **7** was synthesized according to the reported literature.⁶

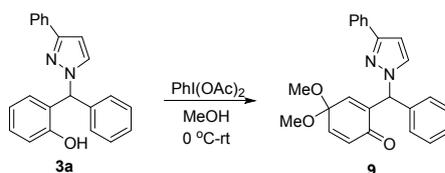
Triflic anhydride (21.8 μ L, 0.12 mmol) in CH_2Cl_2 (0.5 mL) was added to a solution of **3a** (32.6 mg, 0.1 mmol) and 2,6-lutidine (18.6 μ L, 0.15 mmol) in dry CH_2Cl_2 (0.5 mL) under argon at -30°C for 1 hour. And then, the reaction system reacted at 0°C for 1 hour. The reaction was quenched by addition of water (0.5 mL). The solution was allowed to warm to room temperature. The layers were separated. The aqueous phase was extracted with dichloromethane three times. The combined organic layer was washed with brine and dried over MgSO_4 . After the solvent was removed under reduced pressure, the residue was purified by chromatography on silica gel with EtOAc/hexane. The product **7** was obtained in 95% yield.

6. Synthesis of compound **8** (for Scheme 5 b)



The compound **8** was prepared according to literature procedure.⁶ A solution of **7** (45.8 mg, 0.1 mmol), $\text{Pd}(\text{PPh}_3)_2\text{Cl}_2$ (7.01 mg, 0.01 mmol), LiCl (12.7 mg, 0.3 mmol), and DBU (18 μ L, 0.12 mmol) in dry DMF (0.5 mL), was heated at 145°C under argon for 18 h. After cooling, the dark brown solution was transferred to a separatory funnel. Water and ether was added. The ether layer was washed 3 times with water, dried over MgSO_4 , and evaporated to dryness. The residue was chromatographed on a column of silica gel eluted with EtOAc/hexane to **8** (60% yield).

7. Synthesis of compound **9** (for Scheme 5 b)

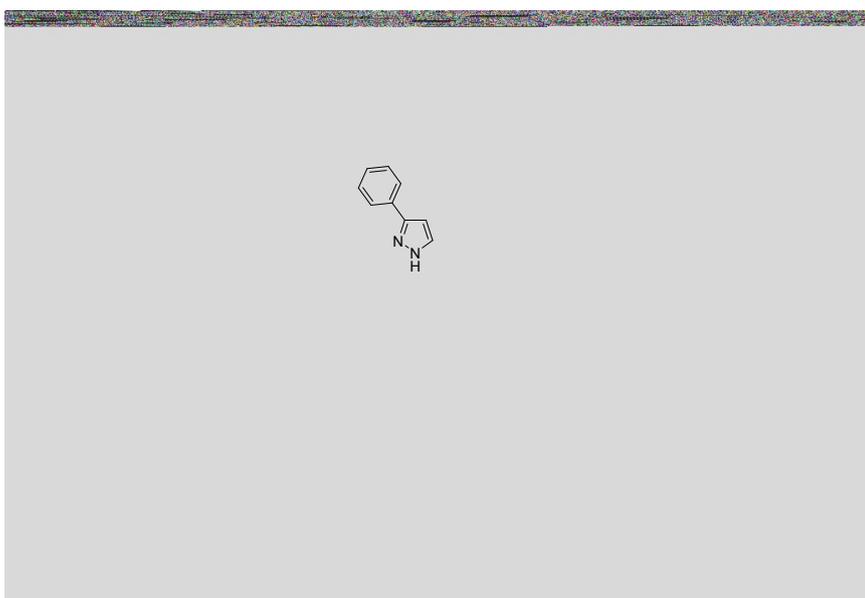
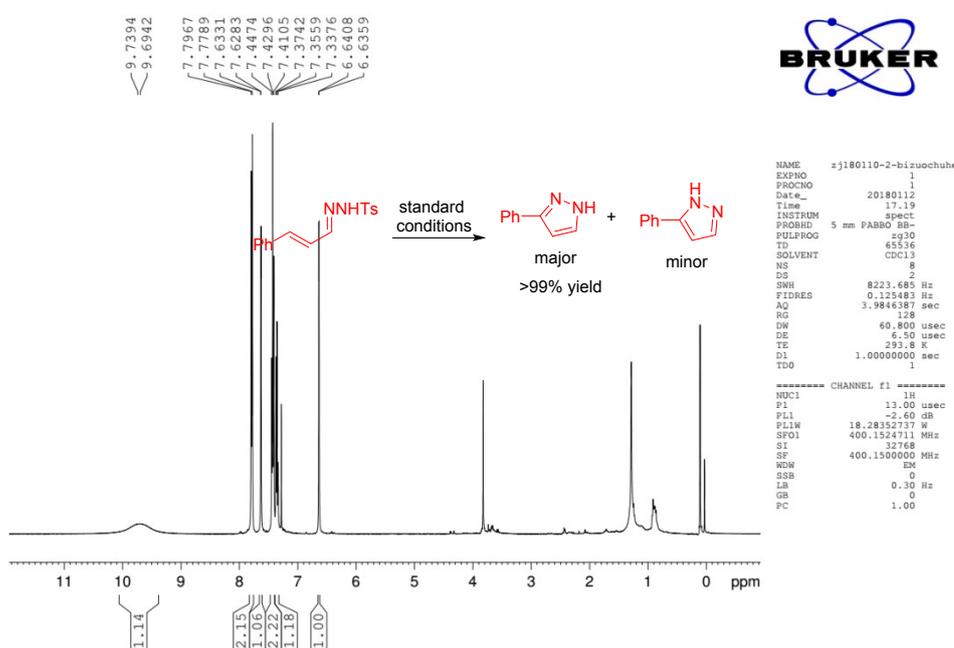


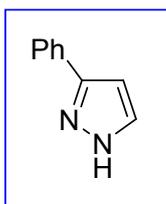
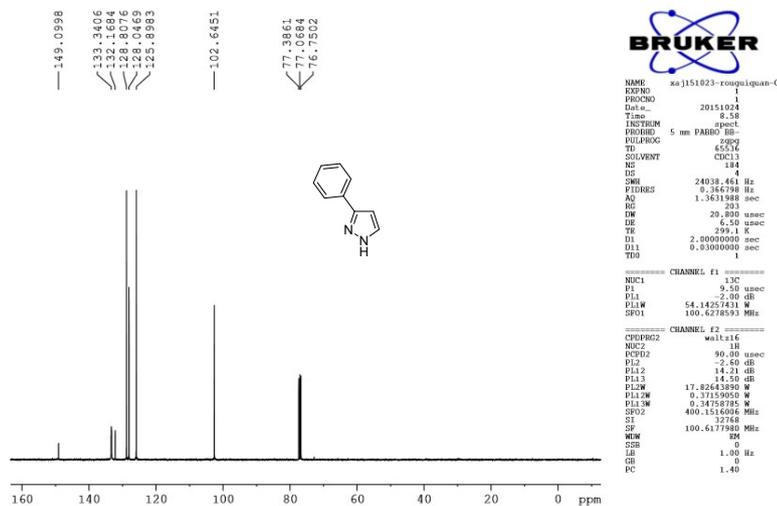
A modified literature procedure was followed.⁷

PhI(OAc)₂ (32.2 mg, 0.1 mmol) was added to the solution of **3a** (32.6 mg, 0.1 mmol) in MeOH (1 mL) at 0 °C. The mixture was stirred at room temperature for 1 h. Then, 5 mL saturated aqueous NaHCO₃ solution was added. The mixture was extracted with DCM (3×10 mL). The combined organic layer was dried over Na₂SO₄, filtered, and concentrated. The residue was purified by silica gel chromatography (hexanes/EtOAc) to afford the product **9** as a colorless oil in 66% yield.

8. Some complementary experiments (for Scheme 5 a)

¹H NMR spectroscopy of the crude reaction mixture (Scheme 4 a)



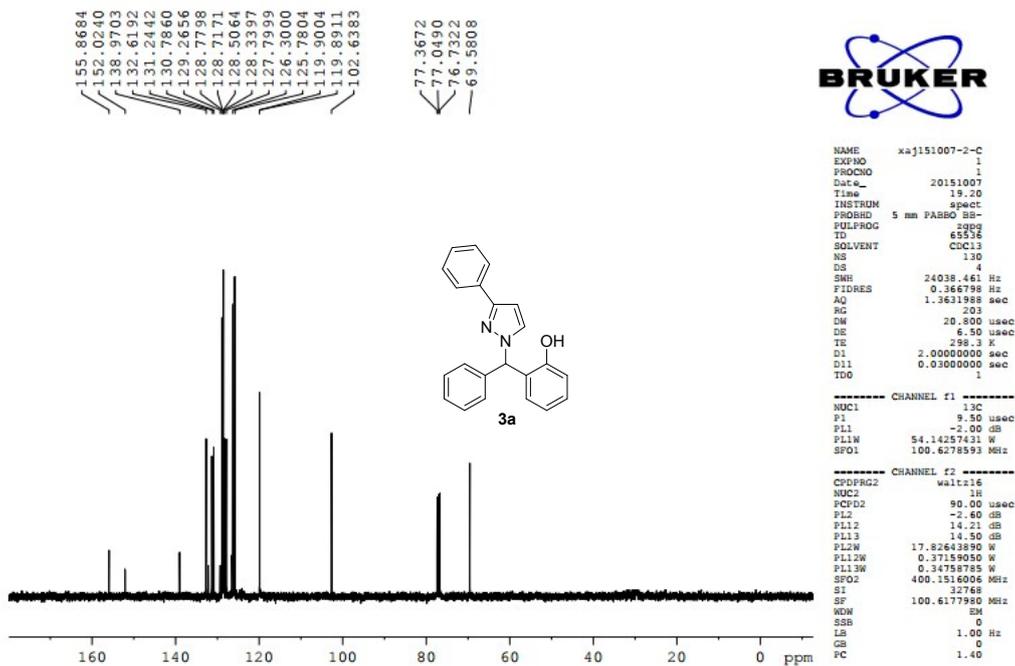
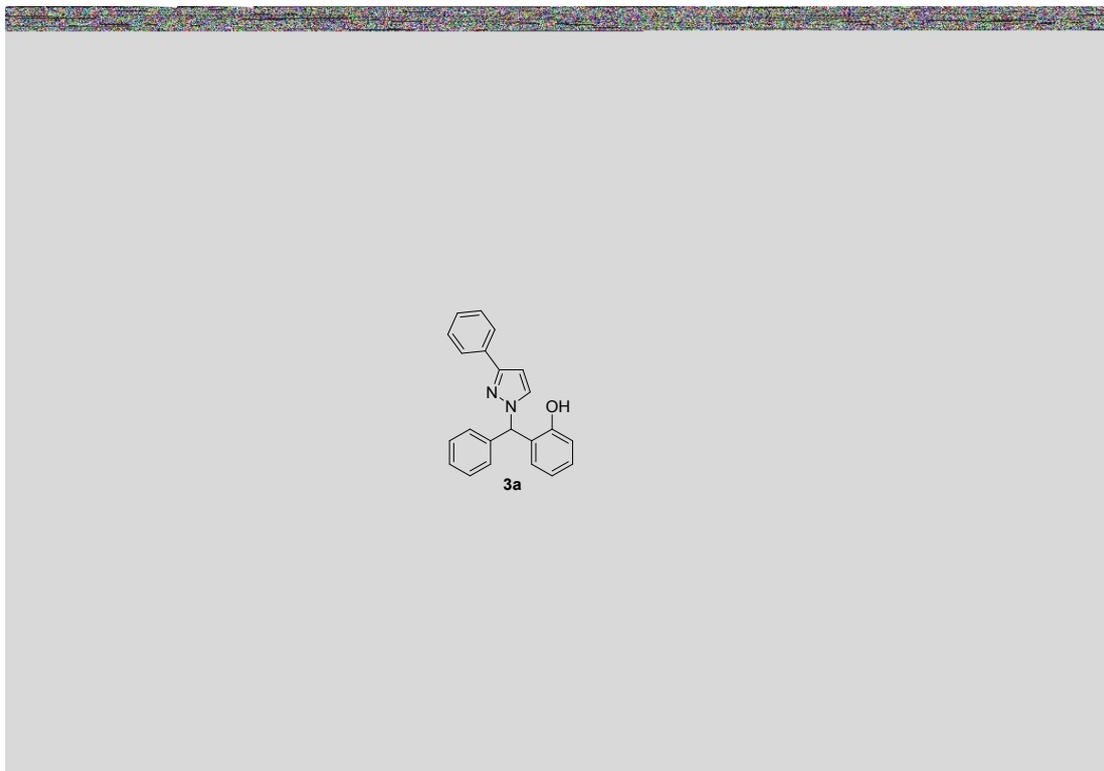


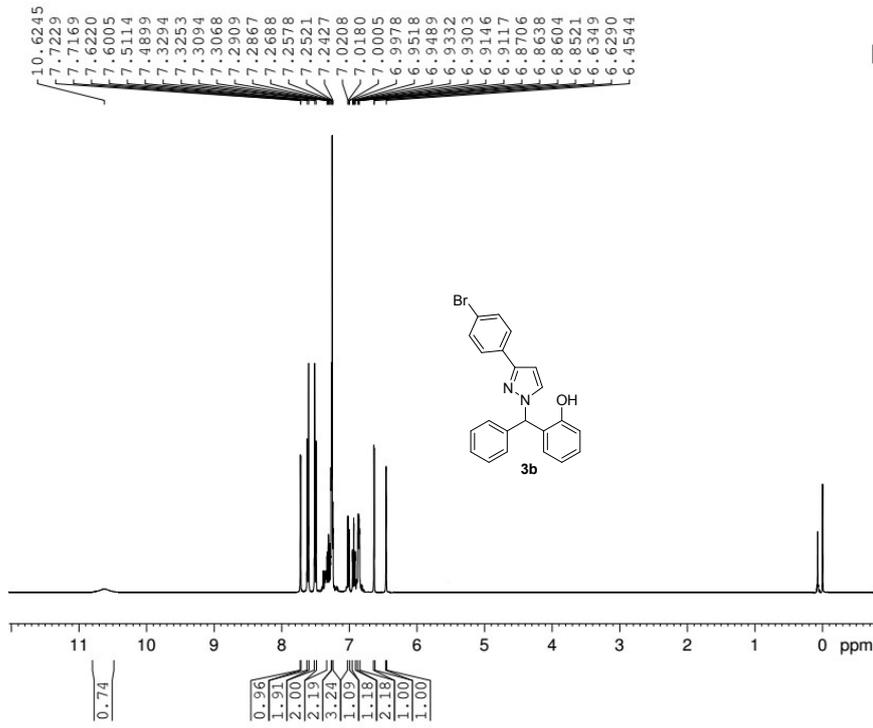
¹H NMR (400 MHz, CDCl₃): δ (ppm) 7.80-7.78 (m, 2H), 7.64 (d, *J* = 1.9Hz, 1H), 7.45-7.41 (m, 2H), 7.38-7.34 (m, 1H), 6.64 (d, *J* = 1.9Hz, 1H). ¹³C NMR (CDCl₃, 100 MHz): δ (ppm) 149.1, 133.3, 132.2, 128.8, 128.0, 125.9, 102.6.

References

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V-NMR spectra for compounds

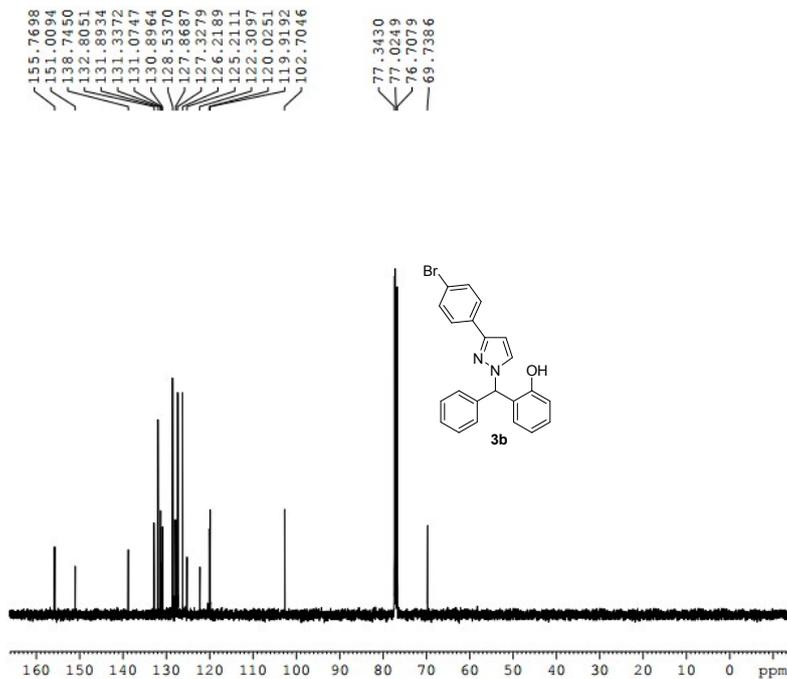




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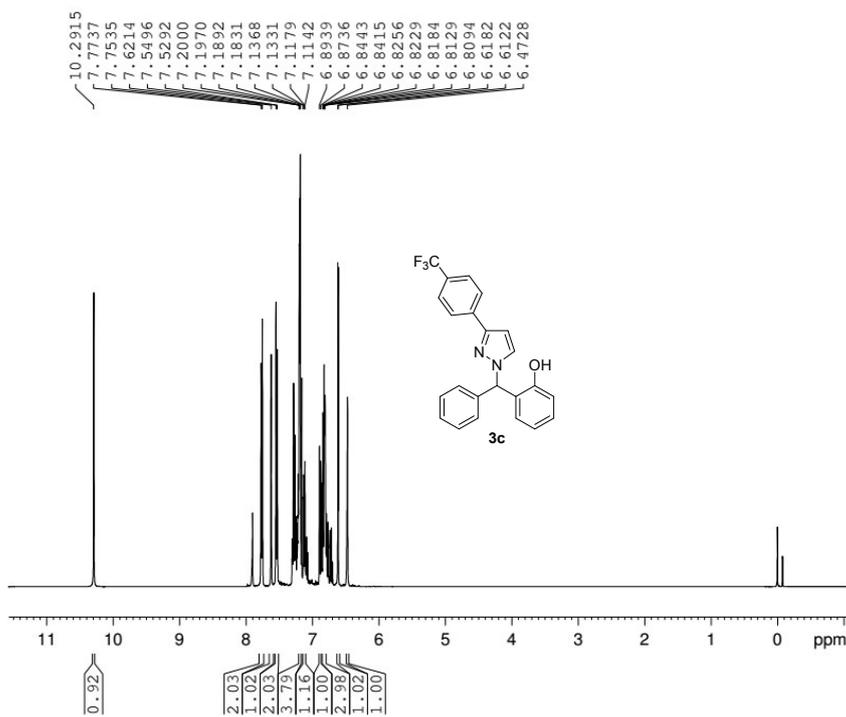
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NAME      160512-4Brzong
EXPNO     2
PROCNO    1
Date_     20160524
Time      3.06
INSTRUM   spect
PROBHD    5 mm PABBO BB/
PULPROG   zgpg30
TD         65536
SOLVENT   CDCl3
NS         512
DS         0
SWH       24038.461 Hz
FIDRES    0.366798 Hz
AQ         1.361988 sec
RG         194.26
DW         20.800 us
DE         6.50 us
TE         300.0 K
D1         2.00000000 sec
D11        0.03000000 sec
D12        1
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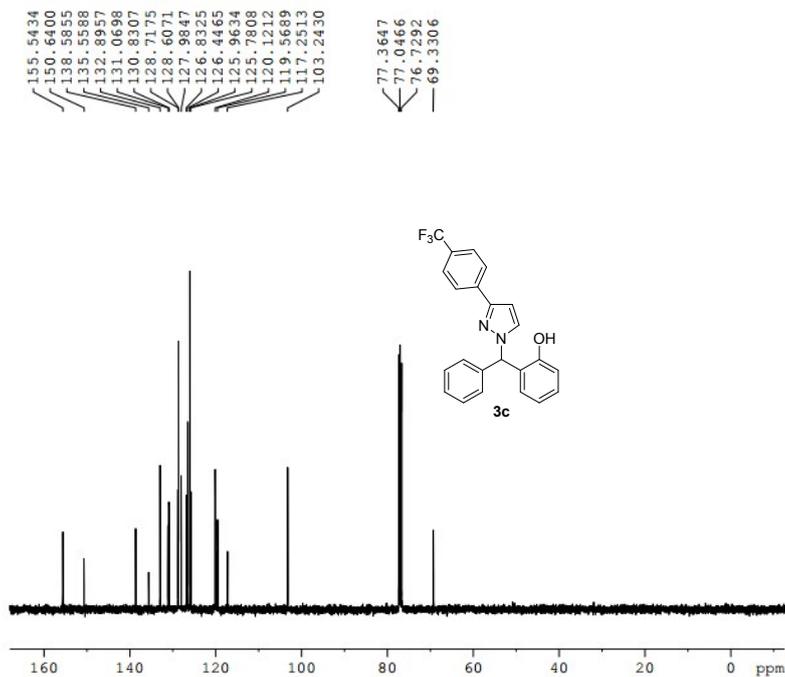
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NAME      160730-4CF3
EXPNO    1
PROCNO   1
Date_    20160730
Time     7.47
INSTRUM  spect
PROBHD   5 mm PABBO BB/
PULPROG  zg30
TD       65536
SOLVENT  CDCl3
NS       16
DS       2
SWH      8012.820 Hz
FIDRES   0.122266 Hz
AQ       4.0894966 sec
RG       32.77
DW       62.400 usec
DE       6.50 usec
TE       300.0 K
D1       1.00000000 sec
TDO      1

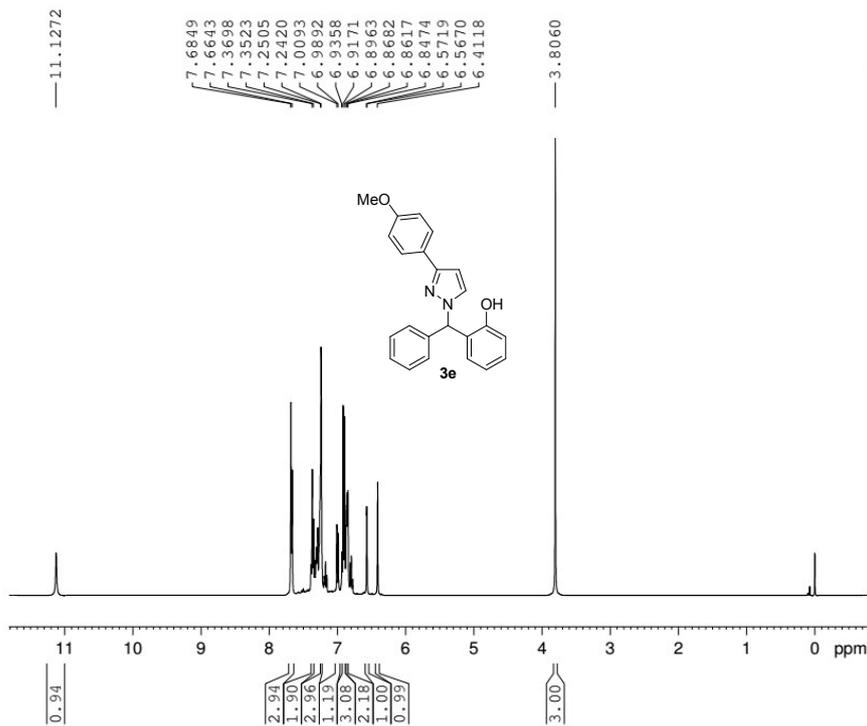
----- CHANNEL f1 -----
SFO1     400.1324710 MHz
NUC1     1H
P1       8.97 usec
SI       65536
SF       400.1300491 MHz
WDW      EM
SSB      0
LB       0.30 Hz
GB       0
PC       1.00
  
```



```

NAME      160730-4CF3
EXPNO    2
PROCNO   1
Date_    20160730
Time     8.03
INSTRUM  spect
PROBHD   5 mm PABBO BB/
PULPROG  zgpg30
TD       65536
SOLVENT  CDCl3
NS       256
DS       0
SWH      24038.463 Hz
FIDRES   0.366798 Hz
AQ       1.3631988 sec
RG       394.26
DW       20.800 usec
DE       6.50 usec
TE       300.0 K
D1       2.00000000 sec
D11      0.03000000 sec
TDO      1

----- CHANNEL f1 -----
SFO1     100.6228293 MHz
NUC1     13C
P1       8.59 usec
SI       32768
SF       100.6127685 MHz
WDW      EM
SSB      0
LB       1.00 Hz
GB       0
PC       1.40
  
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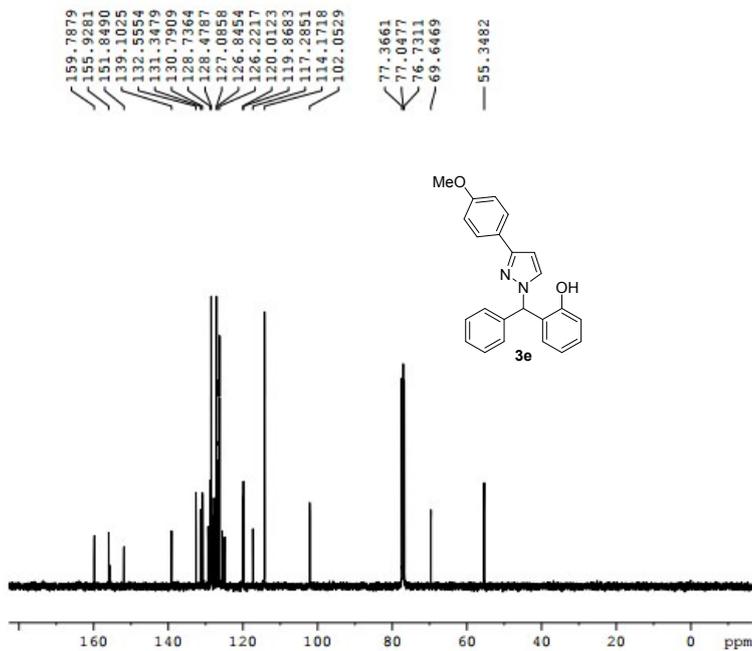
NAME      4e-4ome
EXPNO     1
PROCNO    1
Date_     20160329
Time      23.46
INSTRUM   spect
PROBHD    5 mm PABBO BB/
PULPROG   zg30
TD         65536
SOLVENT   CDCl3
NS         16
DS         2
SWH       8012.820 Hz
FIDRES    0.122266 Hz
AQ         4.0894966 se
RG         32.77
DW         62.400 us
DE         6.50 us
TE         298.2 K
D1         1.00000000 se
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D12        1
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D93        1
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D95        1
D96        1
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D98        1
D99        1
D100       1

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----- CHANNEL f1 -----
SFO1     400.1324710 MHz
NUC1      1H
P1        8.97 us
SI        65536
SF        400.1300164 MHz
WDW       EM
SSB       0
LB        0.30 Hz
GB        0
PC        1.00

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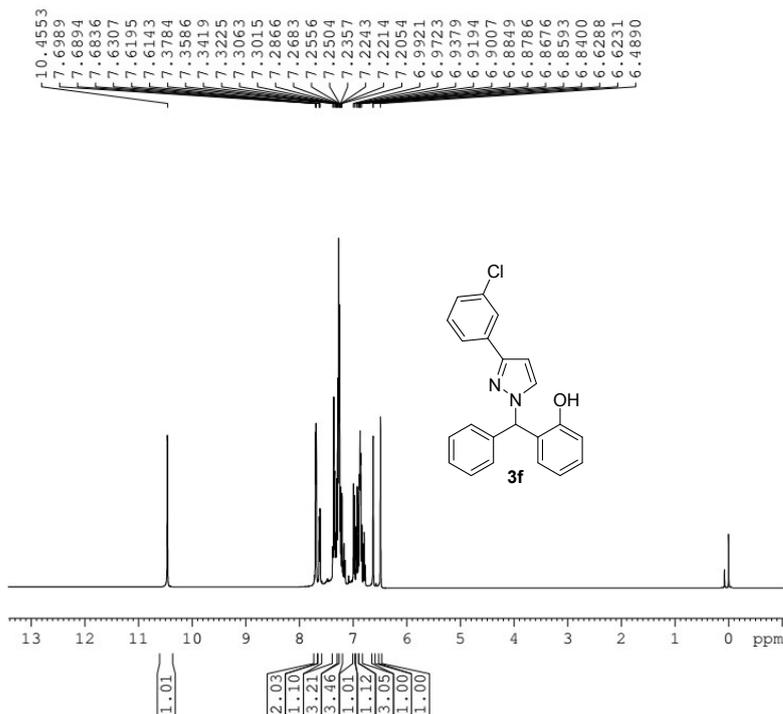
NAME      4e-4ome
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PROCNO    1
Date_     20160330
Time      0.16
INSTRUM   spect
PROBHD    5 mm PABBO BB/
PULPROG   zgpg30
TD         65536
SOLVENT   CDCl3
NS         512
DS         4
SWH       24038.461 Hz
FIDRES    0.366798 Hz
AQ         1.3631988 sec
RG         194.26
DW         20.800 usec
DE         6.50 usec
TE         298.3 K
D1         2.00000000 sec
D11        0.03000000 sec
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D68        1
D69        1
D70        1
D71        1
D72        1
D73        1
D74        1
D75        1
D76        1
D77        1
D78        1
D79        1
D80        1
D81        1
D82        1
D83        1
D84        1
D85        1
D86        1
D87        1
D88        1
D89        1
D90        1
D91        1
D92        1
D93        1
D94        1
D95        1
D96        1
D97        1
D98        1
D99        1
D100       1

```

```

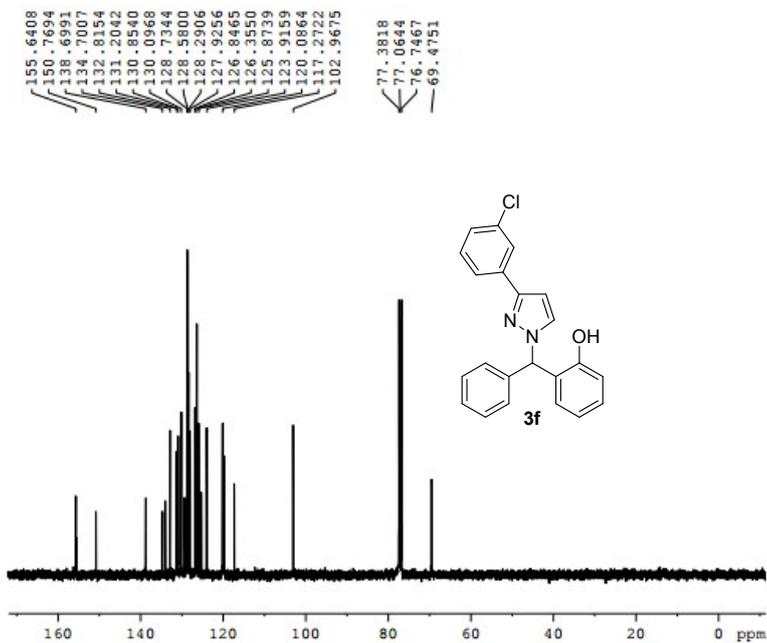
----- CHANNEL f1 -----
SFO1     100.6228293 MHz
NUC1      13C
P1        8.55 usec
SI        32768
SF        100.6127685 MHz
WDW       EM
SSB       0
LB        1.00 Hz
GB        0
PC        1.40

```



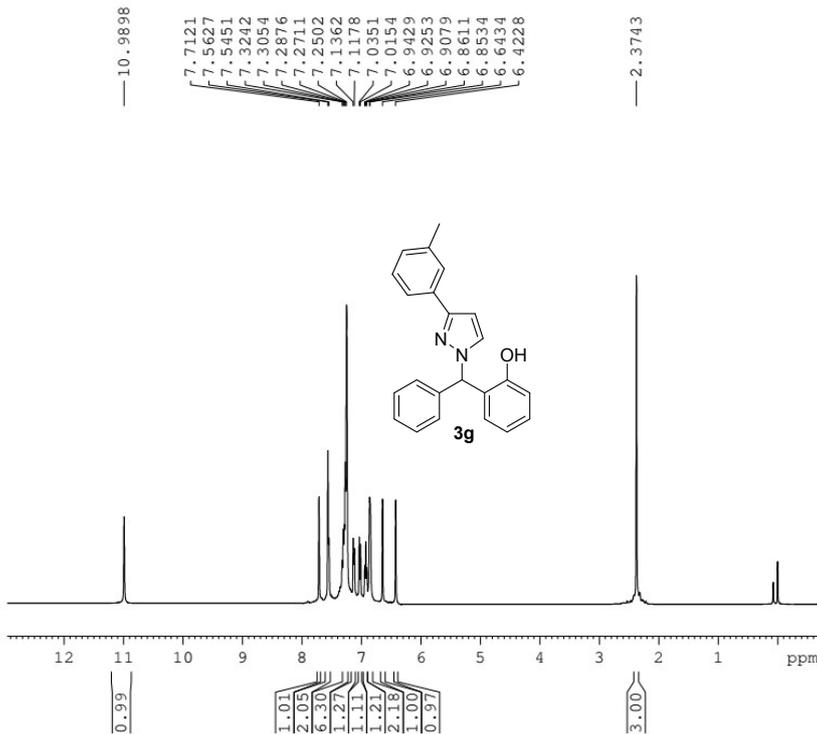
```

NAME          4c-3c1
EXPNO         1
PROCNO        1
Date_         20160329
Time          13.49
INSTRUM       spect
PROBHD        5 mm PABBO BB/
PULPROG       zg30
TD            65536
SOLVENT       CDCl3
NS            16
DS            2
SWH           8012.820 Hz
FIDRES       0.122266 Hz
AQ           4.0894966 sec
RG            32.77
DW           62.400 usec
DE           6.50 usec
TE           298.3 K
D1           1.0000000 sec
D10           1
----- CHANNEL f1 -----
SFO1          400.1324710 MHz
NUC1          1H
P1            8.97 usec
SI            65536
SF           400.1300198 MHz
WDW           EM
SSB           0
LB            0.30 Hz
GB            0
PC            1.00
  
```



```

NAME          4c-3c1
EXPNO         2
PROCNO        1
Date_         20160329
Time          14.04
INSTRUM       spect
PROBHD        5 mm PABBO BB/
PULPROG       zgpg30
TD            65536
SOLVENT       CDCl3
NS            242
DS            4
SWH          24038.461 Hz
FIDRES       0.366798 Hz
AQ           1.3631988 sec
RG            194.26
DW           20.800 usec
DE           6.50 usec
TE           298.3 K
D1           2.0000000 sec
D11          0.0300000 sec
D10           1
----- CHANNEL f1 -----
SFO1          100.6228293 MHz
NUC1          13C
P1            8.59 usec
SI            32768
SF           100.6127685 MHz
WDW           EM
SSB           0
LB            1.00 Hz
GB            0
PC            1.40
  
```

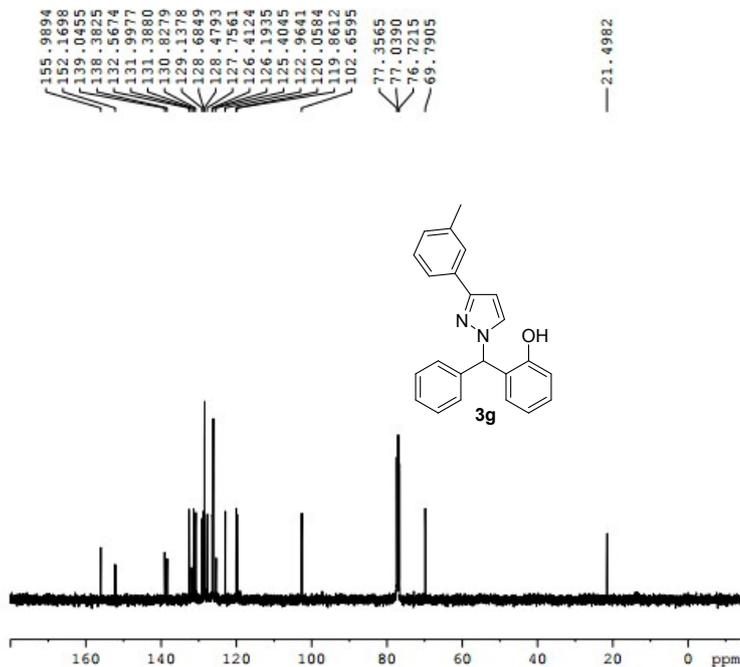


```

NAME          4d-3me
EXPNO         1
PROCNO        1
Date_         20160329
Time          22.34
INSTRUM       spect
PROBHD        5 mm PABBO BB/
PULPROG       zg30
TD            65536
SOLVENT       CDCl3
NS            16
DS            2
SWH           8012.820 Hz
FIDRES        0.122266 Hz
AQ            4.0894966 sec
RG            63
DE            62.400 usec
TE            298.3 K
D1            1.00000000 sec
TDO           1
  
```

```

----- CHANNEL f1 -----
SFO1          400.1324710 MHz
NUC1           1H
P1            8.97 usec
SI            65536
SF            400.1300154 MHz
WDW           EM
SSB           0
LB            0.30 Hz
GB            0
PC            1.00
  
```

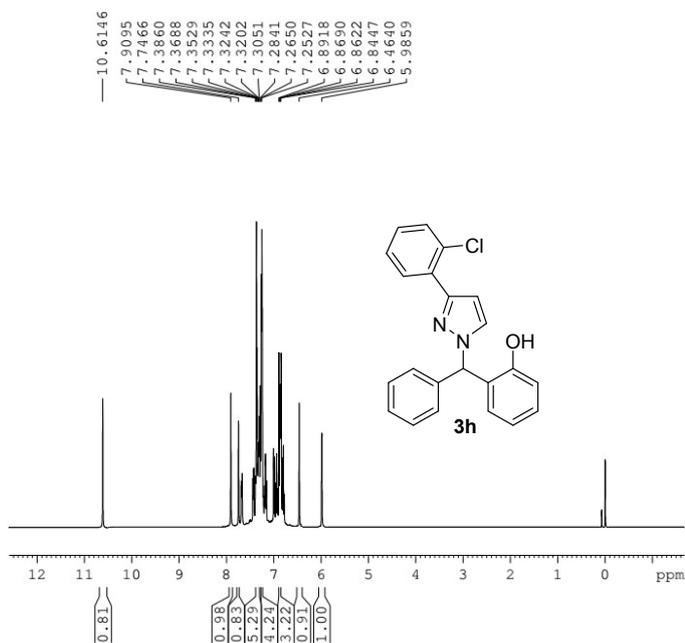


```

NAME          4d-3me
EXPNO         2
PROCNO        1
Date_         20160329
Time          23.04
INSTRUM       spect
PROBHD        5 mm PABBO BB/
PULPROG       zgpg30
TD            65536
SOLVENT       CDCl3
NS            12
DS            4
SWH           24038.461 Hz
FIDRES        0.366798 Hz
AQ            1.3631988 sec
RG            194.26
DE            20.800 usec
TE            298.3 K
D1            2.00000000 sec
D11           0.03000000 sec
TDO           1
  
```

```

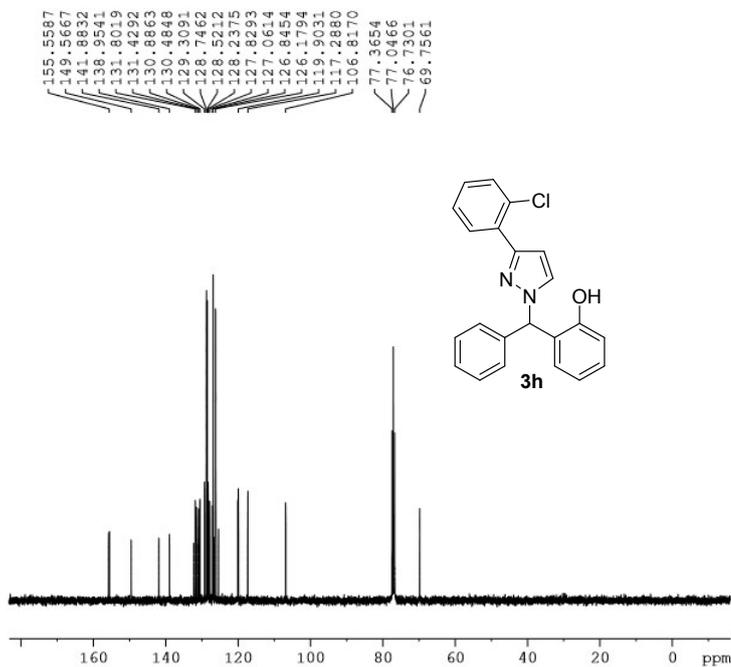
----- CHANNEL f1 -----
SFO1          100.6278293 MHz
NUC1           13C
P1            8.59 usec
SI            32768
SF            100.6127685 MHz
WDW           EM
SSB           0
LB            1.00 Hz
GB            0
PC            1.40
  
```



```

NAME          4a-2c1
EXPNO         1
PROCNO       2
Date_         20160329
Time         20.54
INSTRUM      spect
PROBHD       5 mm PABBO BB/
PULPROG      zgpg30
TD           65536
SOLVENT      CDCl3
NS           16
DS           2
SWH          8012.820 Hz
FIDRES      0.122266 Hz
AQ          4.0894966 sec
RG           57.79
DW          62.400 usec
DE           6.50 usec
TE          298.3 K
D1          1.00000000 sec
D11         1
TD0         1

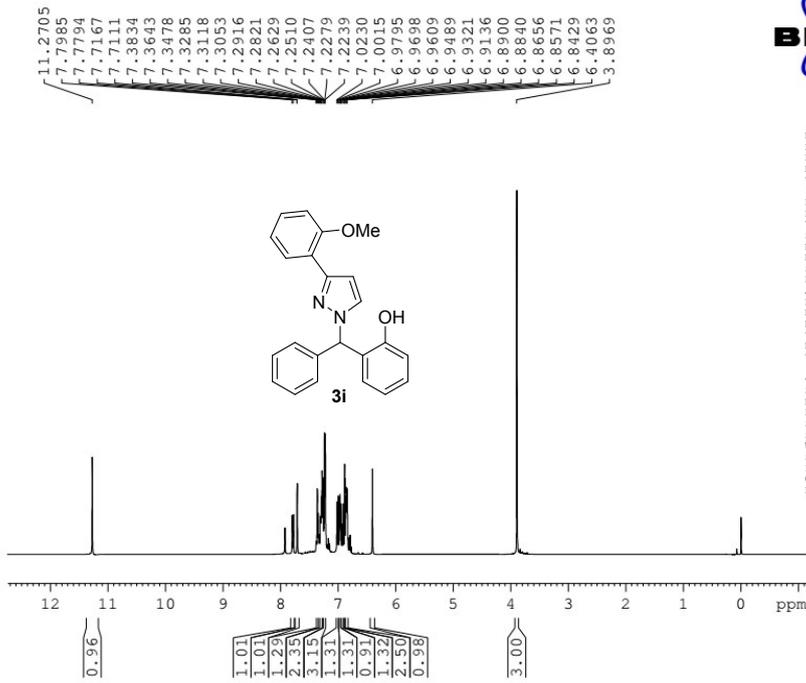
===== CHANNEL f1 =====
SF01        400.1324710 MHz
NUC1        1H
P1          8.97 usec
SI          65536
SF          400.1300164 MHz
WDW         EM
SSB         0
LB          0.30 Hz
GB          0
PC          1.00
  
```



```

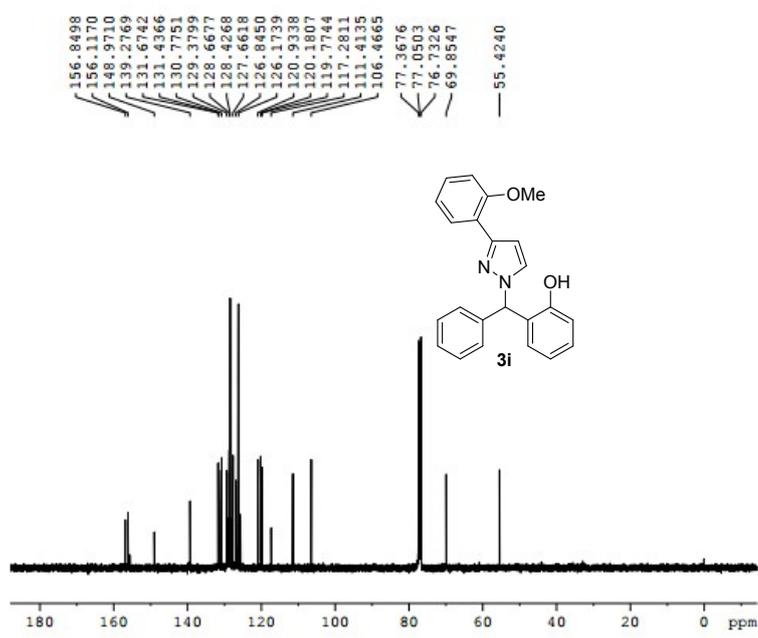
NAME          4a-2c1
EXPNO         1
PROCNO       2
Date_         20160329
Time         20.56
INSTRUM      spect
PROBHD       5 mm PABBO BB/
PULPROG      zgpg30
TD           65536
SOLVENT      CDCl3
NS           256
DS           2
SWH          24038.464 Hz
FIDRES      0.366798 Hz
AQ          1.3631988 sec
RG           194.26
DW          20.800 usec
DE           6.50 usec
TE          298.5 K
D1          2.00000000 sec
D11         0.03000000 sec
TD0         1

===== CHANNEL f1 =====
SF01        100.6228293 MHz
NUC1        13C
P1          8.59 usec
SI          32768
SF          100.6127685 MHz
WDW         EM
SSB         0
LB          1.00 Hz
GB          0
PC          1.40
  
```



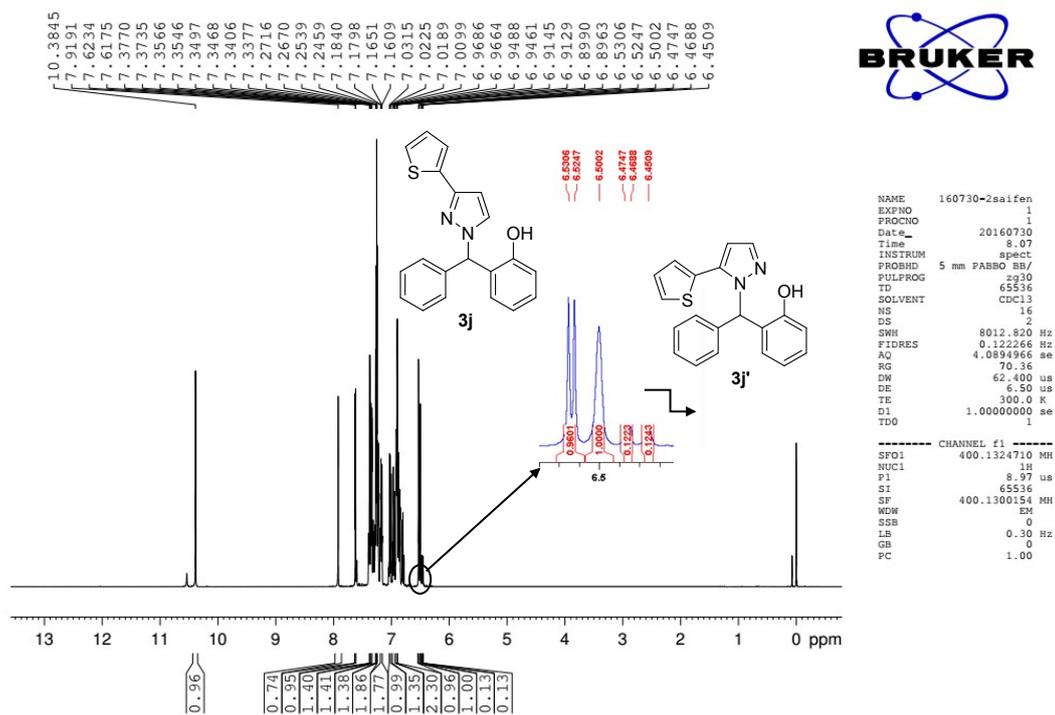
```

NAME          4b-2ome
EXPNO         1
PROCNO        1
Date_         20160329
Time          21.35
INSTRUM       spect
PROBHD        5 mm PABBO BB/
PULPROG       zg30
TD            65536
SOLVENT       CDCl3
NS            16
DS            2
SWH           8012.820 Hz
FIDRES        0.122266 Hz
AQ            4.0894966 sec
RG            51.19
DW            62.400 usec
DE            6.50 usec
TE            298.3 K
D1            1.00000000 sec
TD0           1
----- CHANNEL f1 -----
SFO1          400.1324710 MHz
NUC1          1H
P1            8.97 usec
SI            65536
SF            400.1300175 MHz
WDW           EM
SSB           0
LB            0.30 Hz
GB            0
PC            1.00
  
```

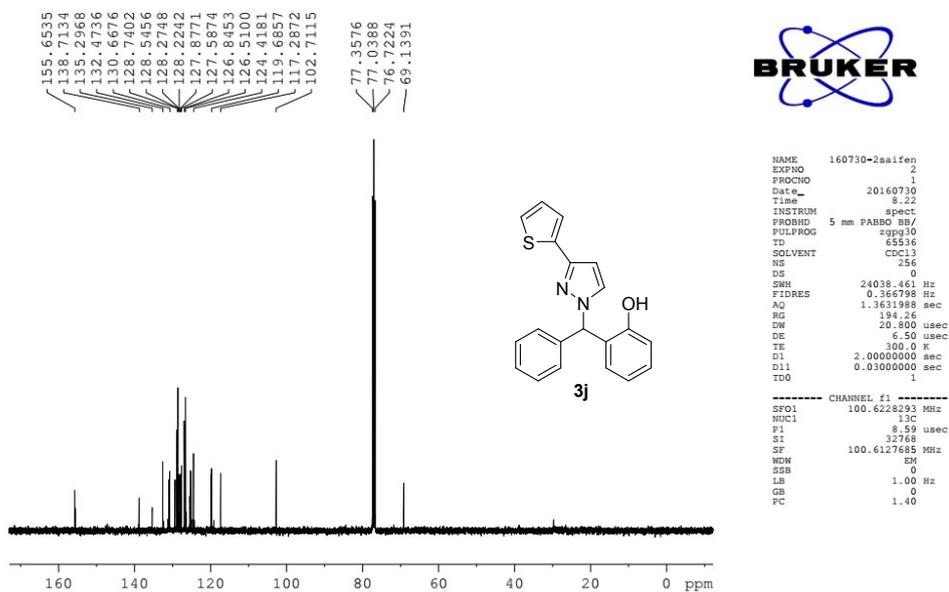


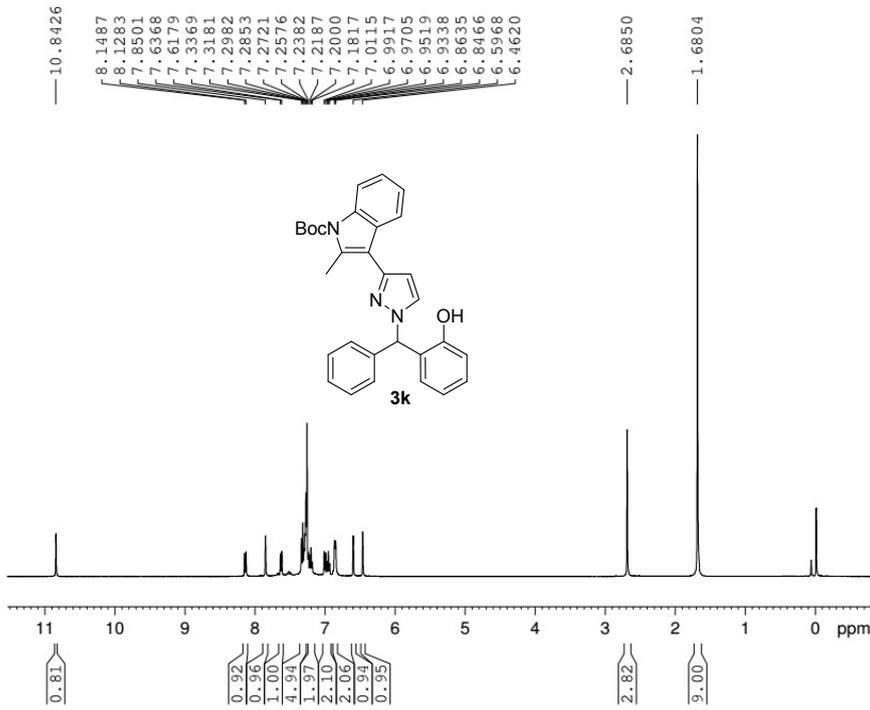
```

NAME          4b-2ome
EXPNO         2
PROCNO        1
Date_         20160329
Time          22.06
INSTRUM       spect
PROBHD        5 mm PABBO BB/
PULPROG       zgpg30
TD            65536
SOLVENT       CDCl3
NS            512
DS            4
SWH           24038.461 Hz
FIDRES        0.364798 Hz
AQ            1.3631988 sec
RG            194.26
DW            20.800 usec
DE            6.50 usec
TE            298.3 K
D1            2.00000000 sec
D11           0.03000000 sec
TD0           1
----- CHANNEL f1 -----
SFO1          100.6228293 MHz
NUC1          13C
P1            8.59 usec
SI            32768
SF            100.6127685 MHz
WDW           EM
SSB           0
LB            1.00 Hz
GB            0
PC            1.40
  
```



the ratio of 3j:3j' was about 8:1



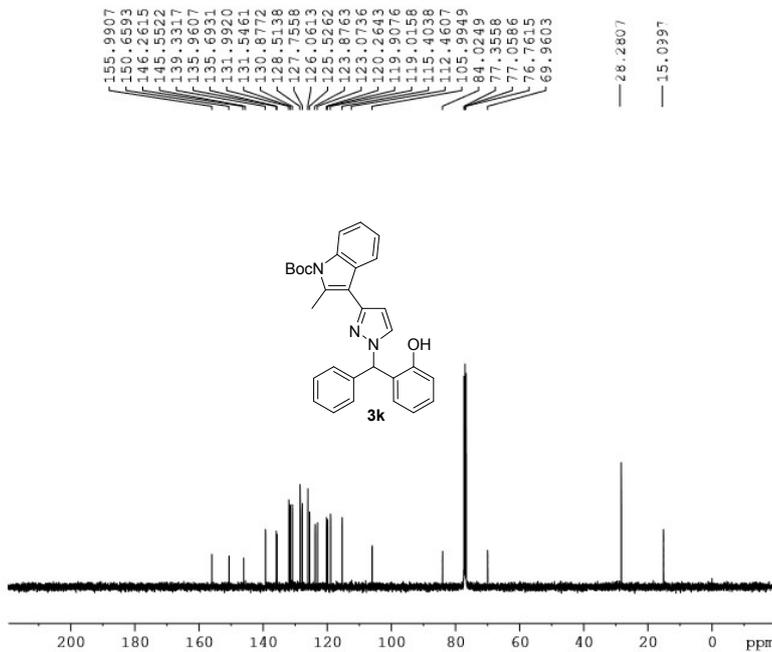


```

NAME      4f-2mebocindole
EXPNO     1
PROCNO    1
Date_     20160330
Time      5:10
INSTRUM   spect
PROBHD    5 mm PABBO BB/
PULPROG   zg30
TD         65536
SOLVENT   CDCl3
NS         16
DS         2
SWH        8012.820 Hz
FIDRES     0.122266 Hz
AQ         4.0894966 sec
RG         77.71
DW         62.400 usec
DE         6.50 usec
TE         298.3 K
D1         1.00000000 sec
TD0        1
  
```

```

----- CHANNEL f1 -----
SFO1      400.1324710 MHz
NUC1       1H
P1         8.97 usec
SI         65536
SF         400.1300112 MHz
WDW        EM
SSB        0
LB         0.30 Hz
GB         0
PC         1.00
  
```

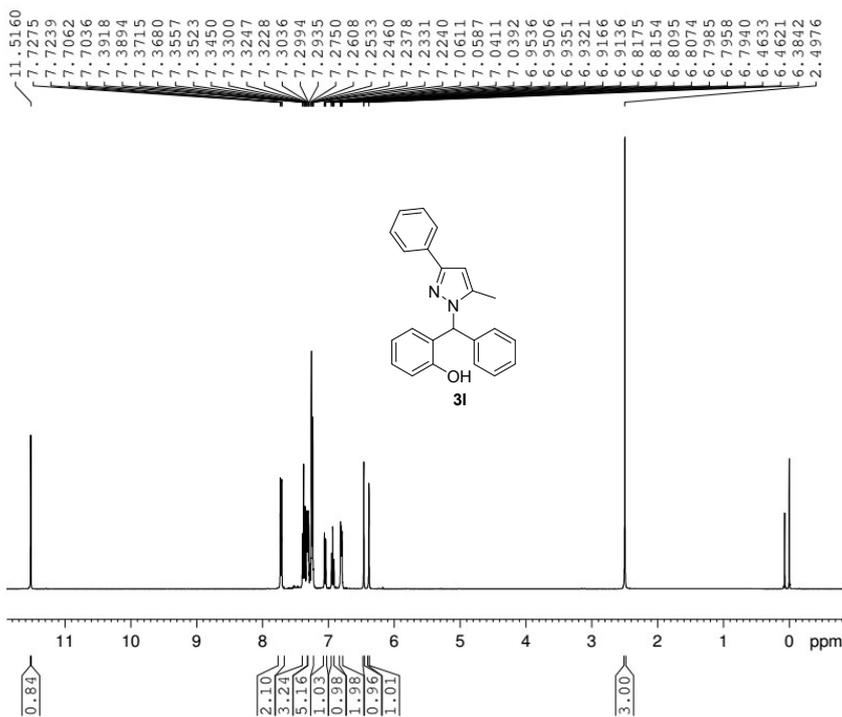


```

NAME      4f-2mebocindole
EXPNO     2
PROCNO    1
Date_     20160330
Time      5:41
INSTRUM   spect
PROBHD    5 mm PABBO BB/
PULPROG   zgpg30
TD         65536
SOLVENT   CDCl3
NS         512
DS         4
SWH        24038.461 Hz
FIDRES     0.366798 Hz
AQ         1.3631988 sec
RG         194.26
DW         20.800 usec
DE         6.50 usec
TE         298.3 K
D1         2.00000000 sec
D11        0.03000000 sec
TD0        1
  
```

```

----- CHANNEL f1 -----
SFO1      100.6228293 MHz
NUC1       13C
P1         8.59 usec
SI         32768
SF         100.6127685 MHz
WDW        EM
SSB        0
LB         1.00 Hz
GB         0
PC         1.40
  
```

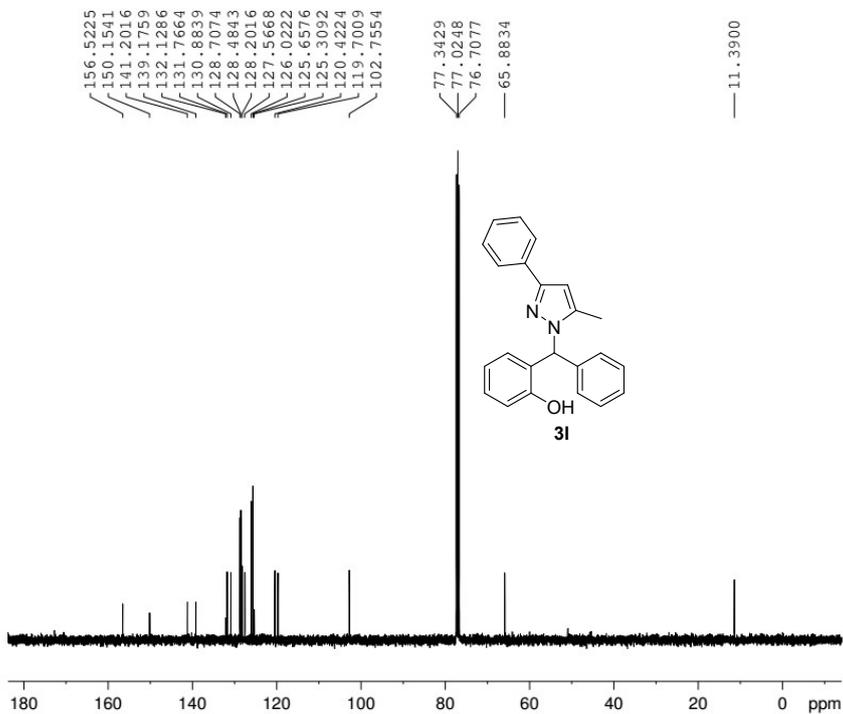


```

NAME      160730-MeTong
EXPNO    1
PROCNO   1
Date_    20160730
Time     9.07
INSTRUM  spect
PROBHD   5 mm PABBO BB/
PULPROG  zgpg30
TD        65536
SOLVENT  CDCl3
NS        16
DS        2
SWH      8012.820 Hz
FIDRES   0.122266 Hz
AQ        4.0894966 sec
RG        77.71
DW        62.400 usec
DE        6.50 usec
TE        300.0 K
D1        1.00000000 sec
TD0       1
  
```

```

----- CHANNEL f1 -----
SF01    400.1324710 MHz
NUC1     1H
P1       8.97 usec
SI       65536
SF       400.1300124 MHz
WDW      EM
SSB      0
LB       0.30 Hz
GB       0
PC       1.00
  
```

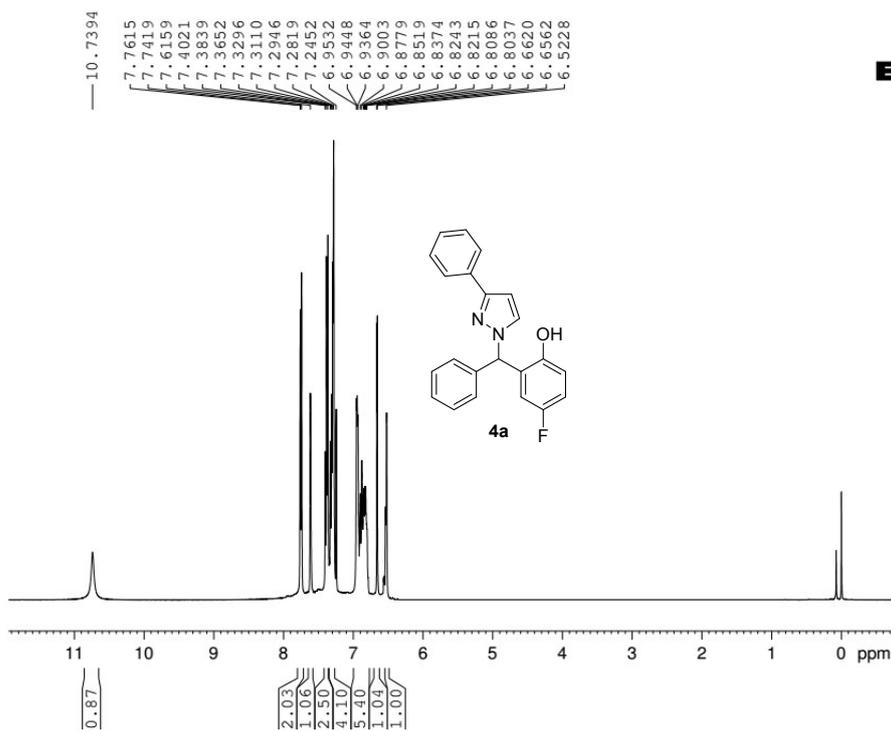


```

NAME      160730-MeTong
EXPNO    2
PROCNO   1
Date_    20160730
Time     9.23
INSTRUM  spect
PROBHD   5 mm PABBO BB/
PULPROG  zgpg30
TD        65536
SOLVENT  CDCl3
NS        256
DS        0
SWH      24038.461 Hz
FIDRES   0.366798 Hz
AQ        1.363198 sec
RG        194.26
DW        20.800 usec
DE        6.50 usec
TE        300.0 K
D1        2.00000000 sec
D11       0.03000000 sec
TD0       1
  
```

```

----- CHANNEL f1 -----
SF01    100.6228293 MHz
NUC1     13C
P1       8.59 usec
SI       32768
SF       100.6127685 MHz
WDW      EM
SSB      0
LB       1.00 Hz
GB       0
PC       1.40
  
```

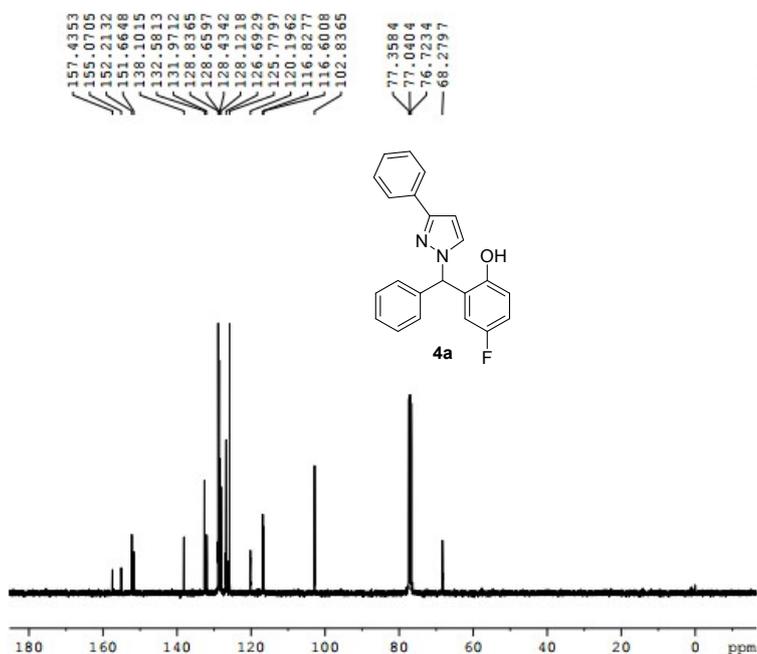
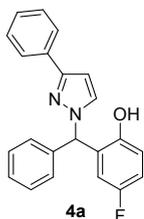


```

NAME          3f-5F
EXPNO         1
PROCNO        1
Date_         20160327
Time         6.25
INSTRUM       spect
PROBHD        5 mm PABBO BB/
PULPROG       zg30
TD            65536
SOLVENT       CDCl3
NS           16
DS           2
SWH          8012.820 Hz
FIDRES       0.122266 Hz
AQ          4.0894966 sec
RG           57.79
DW          62.400 usec
DE          6.50 usec
TE           298.3 K
D1          1.00000000 sec
D11          1
TDO          1

----- CHANNEL f1 -----
SFO1         400.1324710 MHz
NUC1         1H
P1           8.97 usec
SI          65536
SF          400.1300159 MHz
WDW          EM
SSB          0
LB          0.30 Hz
GB          0
PC          1.00

```

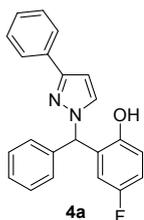


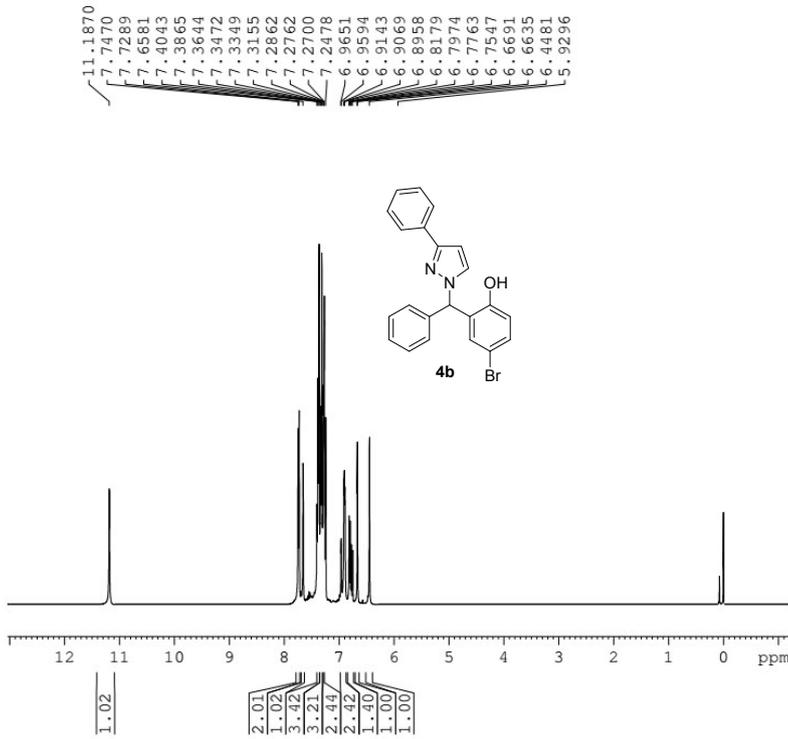
```

NAME          3f-5F
EXPNO         2
PROCNO        1
Date_         20160327
Time         7.25
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           194.26
DW          20.800 usec
DE          6.50 usec
TE           298.3 K
D1          2.00000000 sec
D11          0.03000000 sec
TDO          1

----- CHANNEL f1 -----
SFO1         100.6228293 MHz
NUC1         13C
P1           8.59 usec
SI          32768
SF          100.6127685 MHz
WDW          EM
SSB          0
LB          1.00 Hz
GB          0
PC          1.40

```

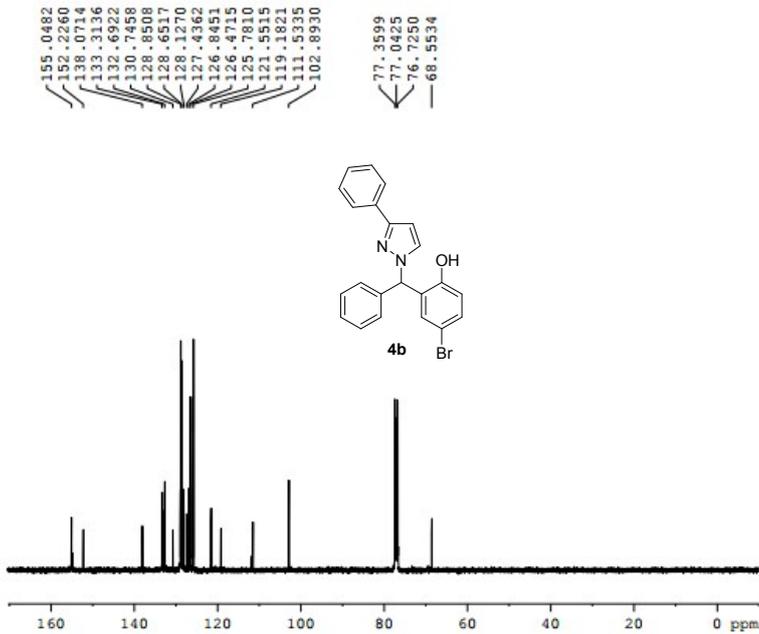




```

NAME          3q-5br
EXPNO         1
PROCNO        1
Date_         20160327
Time          7.29
INSTRUM       spect
PROBHD        5 mm PABBO BB/
PULPROG       zg30
TD            65536
SOLVENT       CDCl3
NS            16
DS            2
SWH           8012.820 Hz
FIDRES       0.122266 Hz
AQ           4.0894966 sec
RG           57.79
DM           62.400 usec
DE           6.50 usec
TE           298.3 K
D1           1.00000000 sec
TD0          1

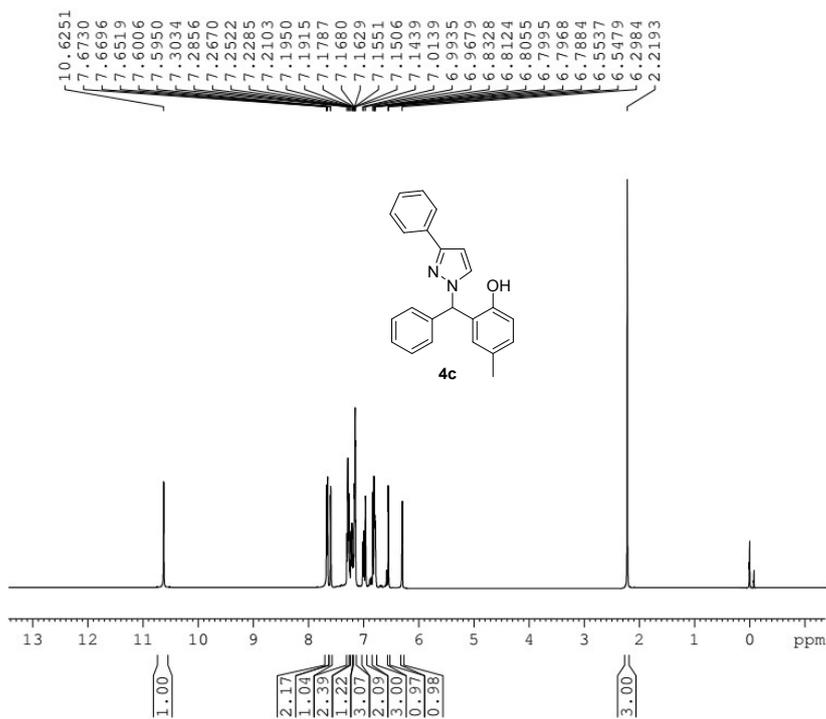
----- CHANNEL f1 -----
SFO1         400.1324710 MHz
NUC1         1H
P1           8.97 usec
SI           65536
SF           400.1300147 MHz
WOW         EX
SSB          0
LB           0.30 Hz
GB           0
PC           1.00
  
```



```

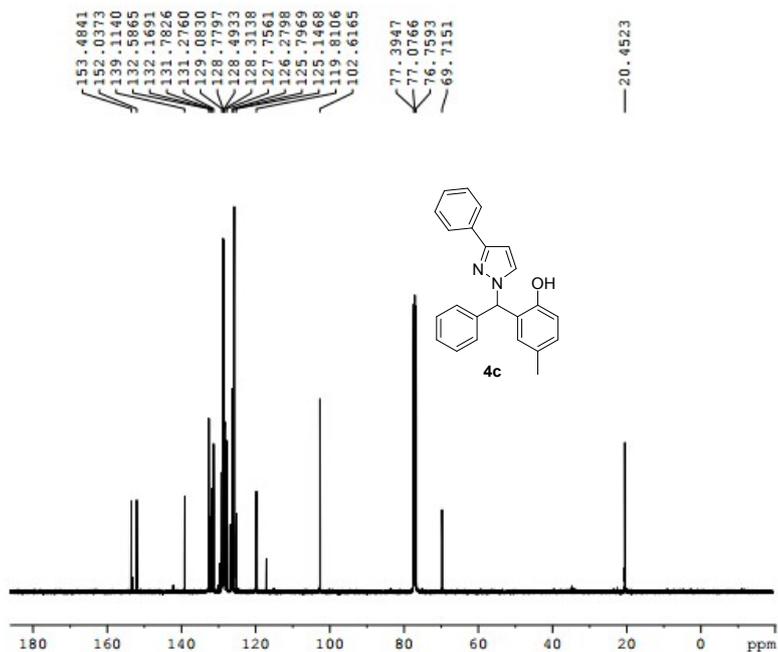
NAME          3q-5br
EXPNO         2
PROCNO        1
Date_         20160327
Time          8.39
INSTRUM       spect
PROBHD        5 mm PABBO BB/
PULPROG       zgpg30
TD            65536
SOLVENT       CDCl3
NS            1024
DS            4
SWH           24038.465 Hz
FIDRES       0.366798 Hz
AQ           1.3631988 sec
RG           194.26
DM           20.800 usec
DE           6.50 usec
TE           298.3 K
D1           2.00000000 sec
D11          0.03000000 sec
TD0          1

----- CHANNEL f1 -----
SFO1         100.6228293 MHz
NUC1         13C
P1           8.59 usec
SI           32768
SF           100.6127685 MHz
WOW         EX
SSB          0
LB           1.00 Hz
GB           0
PC           1.40
  
```



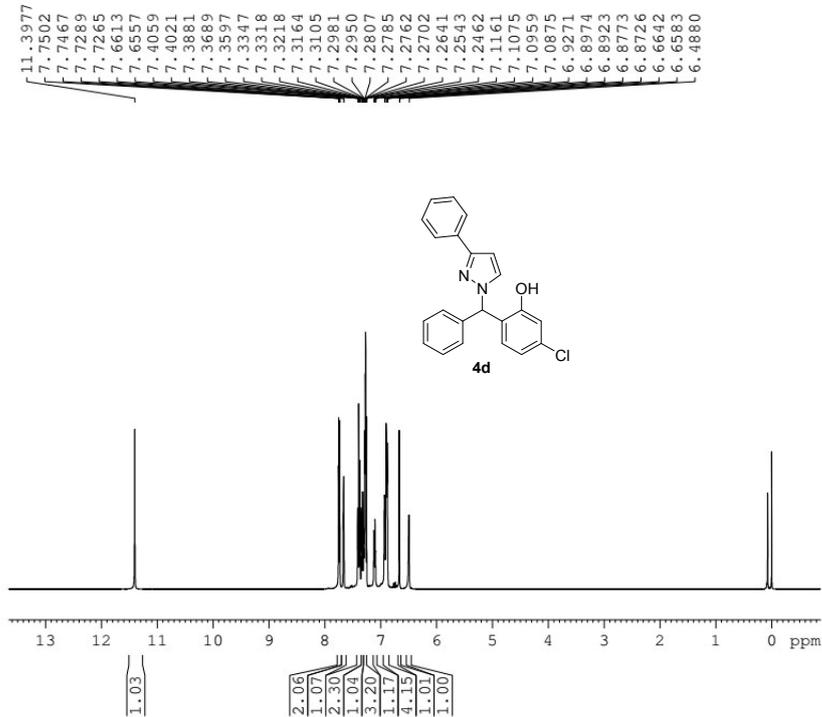
```

NAME          3h-5me
EXPNO         1
PROCNO        1
Date_         20160327
Time          9.38
INSTRUM       spect
PROBHD        5 mm PABBO BB/
PULPROG       zg30
TD            65536
SOLVENT       CDCl3
NS            16
DS            2
SWH           8012.820 Hz
FIDRES       0.122266 Hz
AQ           4.0894966 sec
RG           32.77
DW           62.400 usec
DE           6.50 usec
TE           298.3 K
D1           1.00000000 sec
TD0          1
----- CHANNEL f1 -----
SFO1         400.1324710 MHz
NUC1         1H
P1           8.97 usec
SI           65536
SF           400.1300563 MHz
WF           EM
SSB          0
LB           0.30 Hz
GB           0
PC           1.00
  
```



```

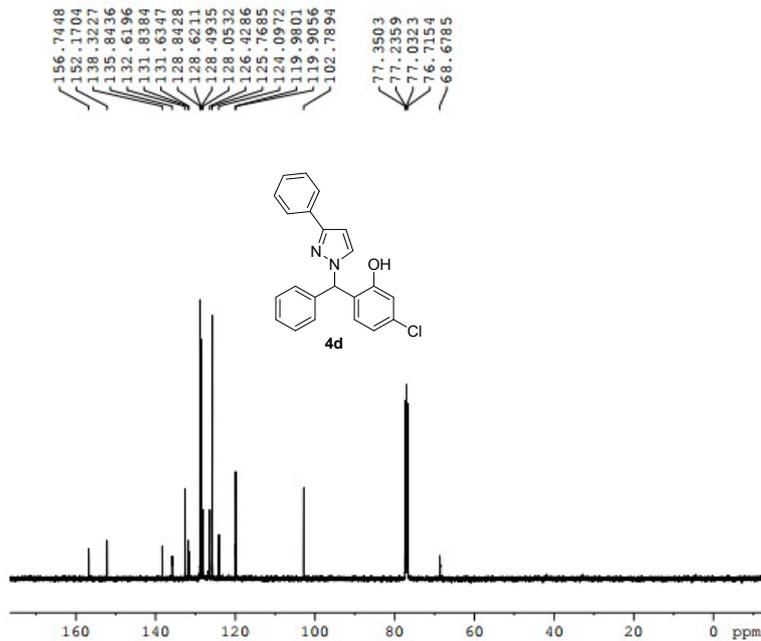
NAME          3h-5me
EXPNO         2
PROCNO        1
Date_         20160327
Time         10.38
INSTRUM       spect
PROBHD        5 mm PABBO BB/
PULPROG       zgpg30
TD            65536
SOLVENT       CDCl3
NS            1024
DS            4
SWH           24038.461 Hz
FIDRES       0.366798 Hz
AQ           1.3631988 sec
RG           194.26
DW           20.800 usec
DE           6.50 usec
TE           298.3 K
D1           2.00000000 sec
D11          0.03000000 sec
TD0          1
----- CHANNEL f1 -----
SFO1         100.6228293 MHz
NUC1         13C
P1           8.59 usec
SI           32768
SF           100.6127685 MHz
WF           EM
SSB          0
LB           1.00 Hz
GB           0
PC           1.40
  
```



```

NAME          3c
EXPNO         1
PROCNO        1
Date_         20160327
Time          3.12
INSTRUM       spect
PROBHD        5 mm PABBO BB/
PULPROG       zg30
TD            65536
SOLVENT       CDCl3
NS            16
DS            2
SWH           8012.820 Hz
FIDRES        0.122266 Hz
AQ            4.0894966 sec
RG            32.77
DW            62.400 usec
DE            6.50 usec
TE            298.3 K
D1            1.00000000 sec
TD0           1

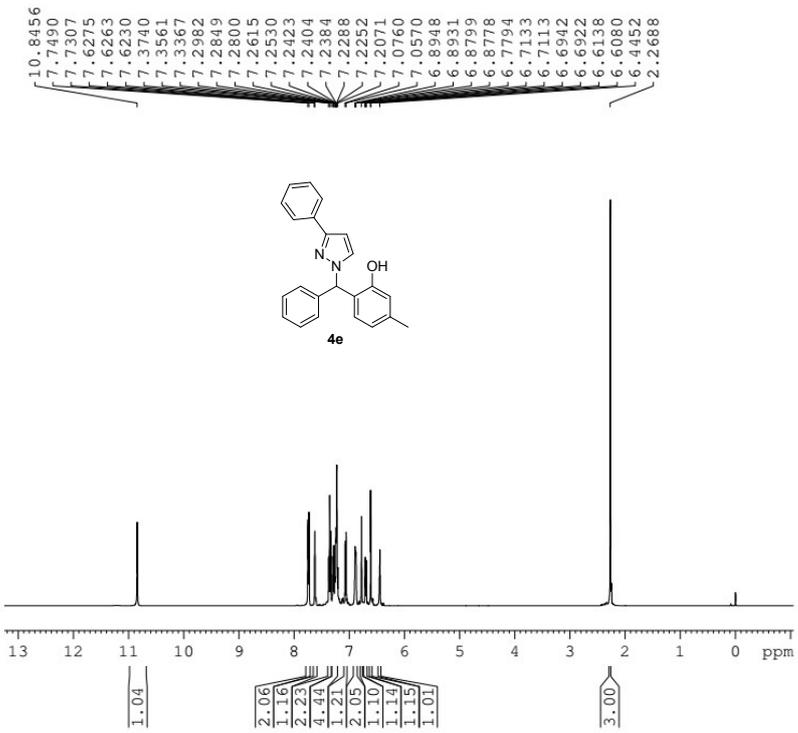
----- CHANNEL f1 -----
SFO1          400.1324710 MHz
NUC1          1H
P1            8.97 usec
SI            65536
SF            400.1300154 MHz
WDW           EM
SSB           0
LB            0.30 Hz
GB            0
PC            1.00
  
```



```

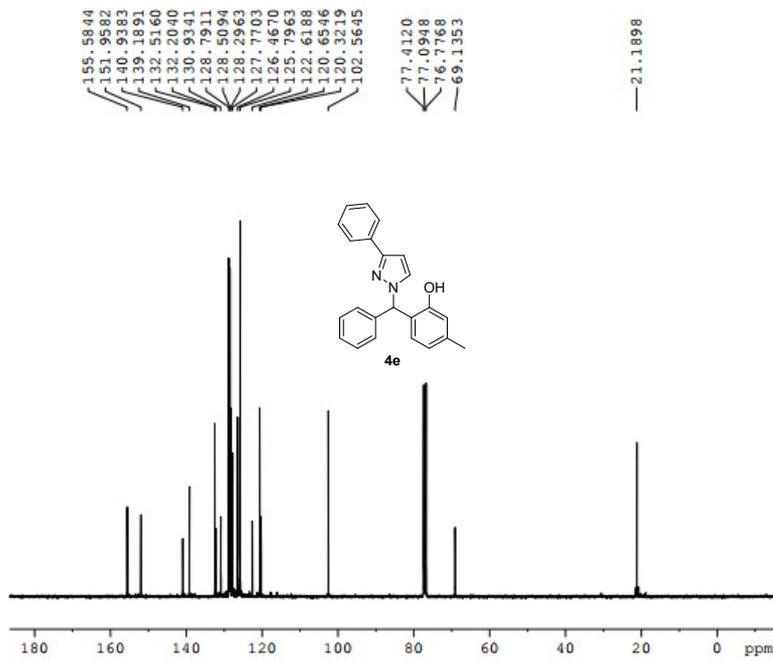
NAME          3c
EXPNO         2
PROCNO        1
Date_         20160327
Time          4.12
INSTRUM       spect
PROBHD        5 mm PABBO BB/
PULPROG       zg30
TD            65536
SOLVENT       CDCl3
NS            1024
DS            4
SWH           24038.461 Hz
FIDRES        0.366798 Hz
AQ            1.3631988 sec
RG            194.26
DW            20.800 usec
DE            6.50 usec
TE            298.3 K
D1            2.00000000 sec
D11           0.03000000 sec
TD0           1

----- CHANNEL f1 -----
SFO1          100.6228293 MHz
NUC1          13C
P1            8.59 usec
SI            32768
SF            100.6127685 MHz
WDW           EM
SSB           0
LB            1.00 Hz
GB            0
PC            1.40
  
```



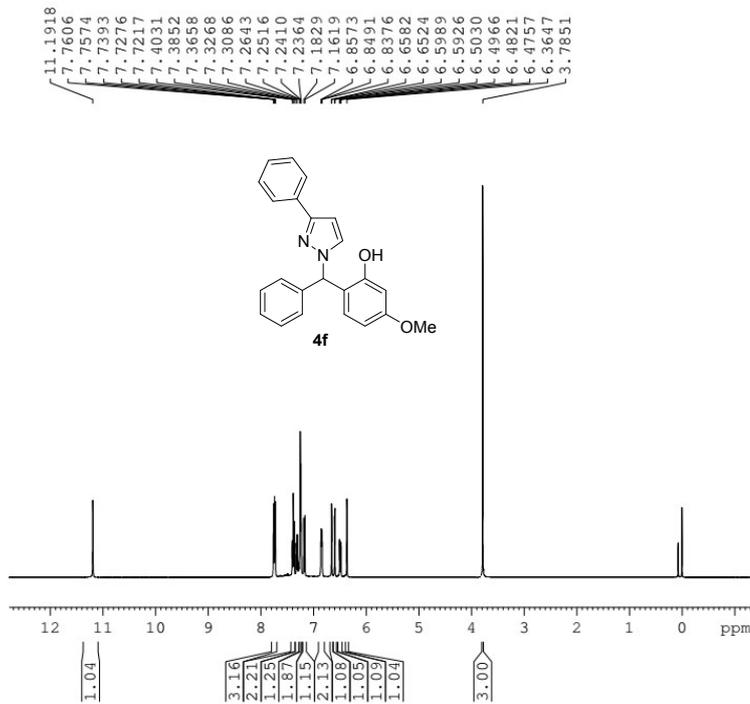
```

NAME          3d
EXPNO         1
PROCNO        1
Date_         20160327
Time          5.21
INSTRUM       spect
PROBHD        5 mm F4BBO BB/
PULPROG       zg30
TD            65536
SOLVENT       CDCl3
NS            16
DS            2
SWH           8012.820 Hz
FIDRES        0.122266 Hz
AQ            4.0894966 sec
RG            18.6
DW            62.400 usec
DE            6.50 usec
TE            298.3 K
D1            1.0000000 sec
TD0           1
----- CHANNEL f1 -----
SFO1          400.1324710 MHz
NUC1          1H
P1            8.97 usec
SI            65536
SF            400.1300309 MHz
WDW           EM
SSB           0
LB            0.30 Hz
GB            0
PC            1.00
  
```



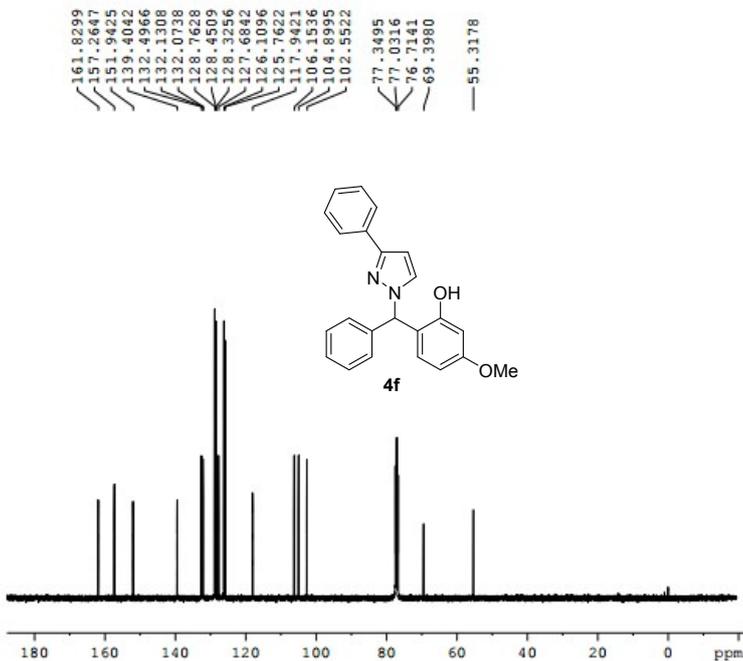
```

NAME          3d
EXPNO         2
PROCNO        1
Date_         20160327
Time          6.20
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           194.26
DW           20.800 usec
DE           6.50 usec
TE           298.3 K
D1           2.00000000 sec
D11          0.03000000 sec
TD0          1
----- CHANNEL f1 -----
SF01         100.6228293 MHz
NUC1          13C
P1            8.59 usec
SI           32768
SF           100.6127685 MHz
WDW          EM
SSB          0
LB           1.00 Hz
GB           0
PC           1.40
  
```



```

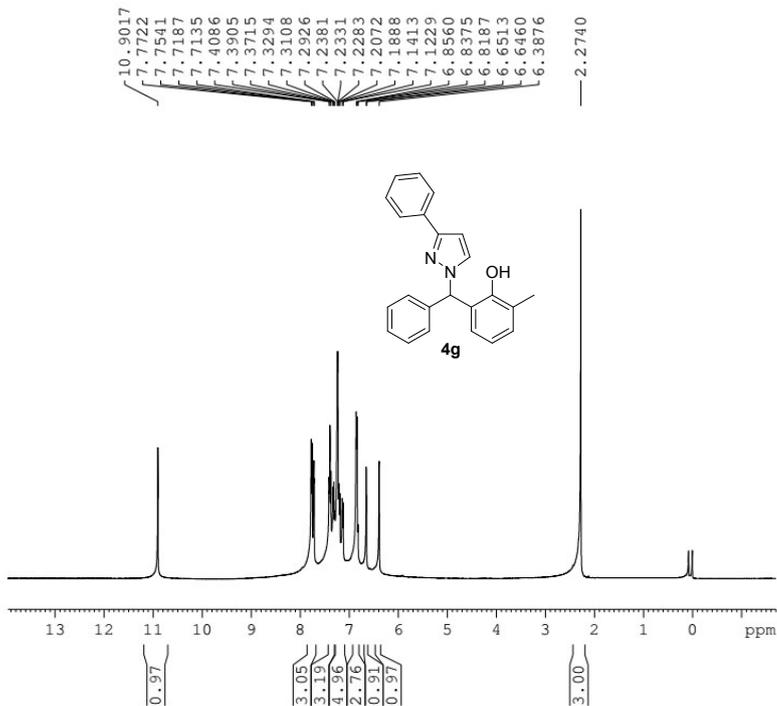
NAME          3e
EXPNO         1
PROCNO        1
Date_         20160327
Time          4.16
INSTRUM       spect
PROBHD        5 mm PABBO BB/
PULPROG       zg30
TD            65536
SOLVENT       CDCl3
NS            16
DS            2
SWH           8012.820 Hz
FIDRES       0.122266 Hz
AQ           4.0894966 sec
RG           70.36
DW           62.400 usec
DE           6.50 usec
TE           298.3 K
D1           1.00000000 sec
TD0          1
----- CHANNEL f1 -----
SF01         400.1324710 MHz
NUC1          1H
P1            8.97 usec
SI           65536
SF           400.1300133 MHz
WDW          EM
SSB          0
LB           0.30 Hz
GB           0
PC           1.00
  
```



```

NAME          3e
EXPNO         1
PROCNO        1
Date_         20160327
Time          3.16
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           194.26
DW           20.800 usec
DE           6.50 usec
TE            298.3 K
SI            2.0000000 sec
Sf1           0.8300000 sec
TD0           1
----- CHANNEL f1 -----
SF01         100.6228293 MHz
NUC1          13C
P1            8.59 usec
SI            32768
SF           100.6127485 MHz
WDW           EM
SSB           0
LB            1.00 Hz
GB            0
PC            1.40

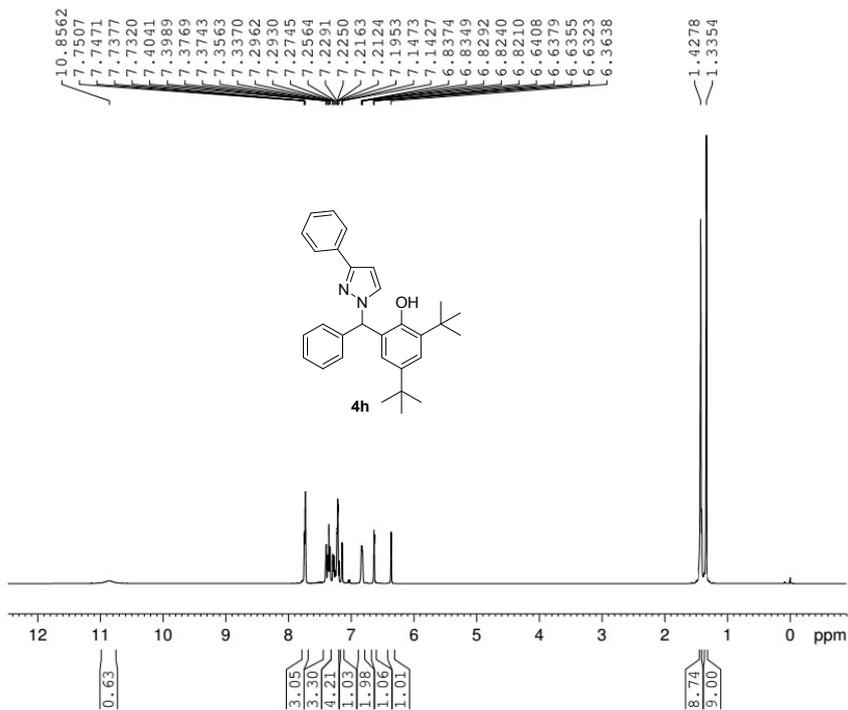
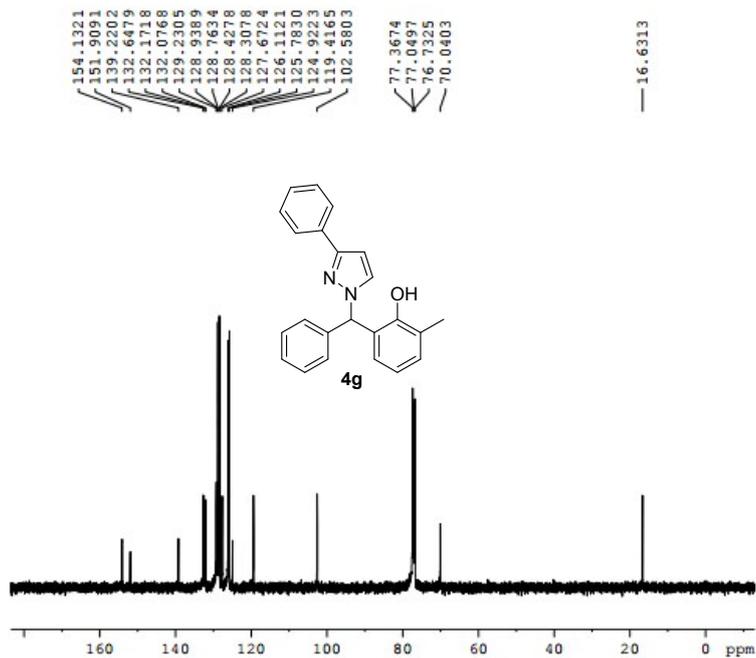
```

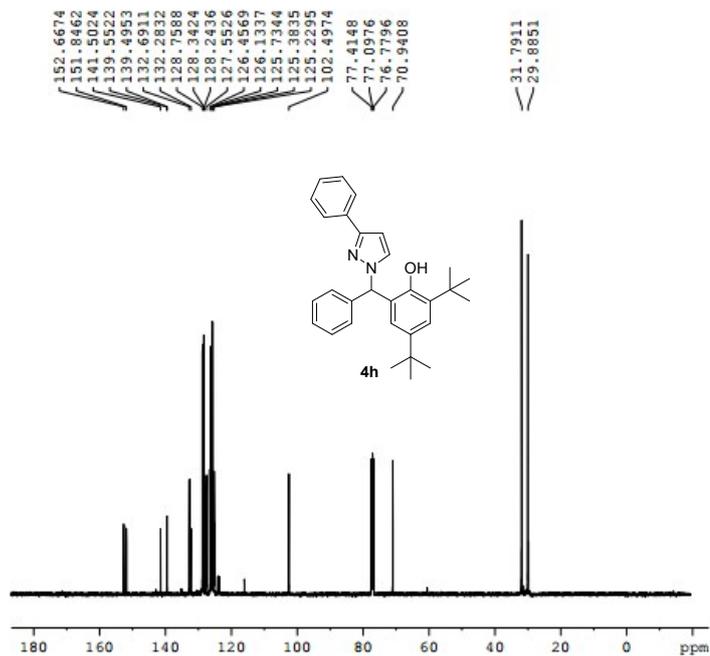


```

NAME          3b
EXPNO         1
PROCNO        1
Date_         20160327
Time          2.08
INSTRUM       spect
PROBHD        5 mm PABBO BB/
PULPROG       zg30
TD            65536
SOLVENT       CDCl3
NS            16
DS            2
SWH           8012.820 Hz
FIDRES       0.122266 Hz
AQ           4.0894966 sec
RG           57.79
DW           62.400 usec
DE           6.50 usec
TE            298.3 K
SI            1.0000000 sec
Sf1           400.1300180 MHz
WDW           EM
SSB           0
LB            0.30 Hz
GB            0
PC            1.00
----- CHANNEL f1 -----
SF01         400.1324710 MHz
NUC1          1H
P1            8.97 usec
SI            65536
SF           400.1300180 MHz
WDW           EM
SSB           0
LB            0.30 Hz
GB            0
PC            1.00

```

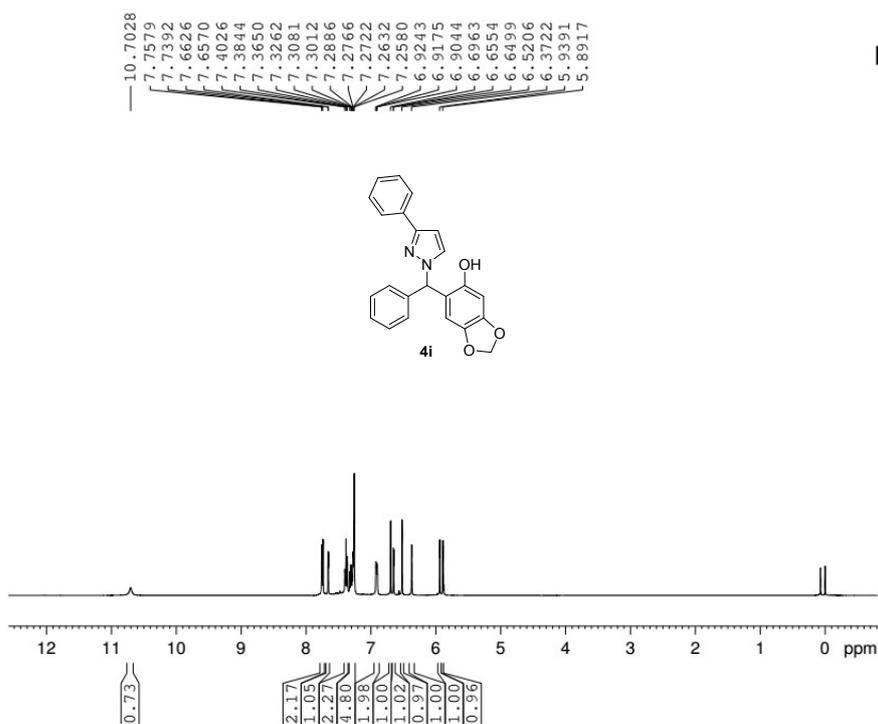




```

NAME          3i-tbu2
EXPNO         2
PROCNO        1
Date_         20160329
Time          9.35
INSTRUM       spect
PROBHD        5 mm PABBO BB/
PULPROG       zgpg30
TD            65536
SOLVENT       CDCl3
NS            512
DS            4
SWH           24038.4611
FIDRES        0.3667981
AQ            1.3619881
RG            194.26
SM           20.8001
DE            6.501
TE            298.31
D1            1.00000000
D11           0.03000000
TDO           1
----- CHANNEL f1 -----
SFO1          100.62282931
NUC1          13C
P1            8.591
SI            32768
SF            100.61276851
WOW           EM
SSB           0
LB            1.001
GB            0
PC            1.40

```



```

NAME          YJL-LQH-Z10419zmff
EXPNO         1
PROCNO        1
Date_         20160421
Time          13.18
INSTRUM       spect
PROBHD        5 mm PABBO BB/
PULPROG       zg30
TD            65536
SOLVENT       CDCl3
NS            16
DS            2
SWH           8012.820 Hz
FIDRES        0.122266 Hz
AQ            4.0894966 sec
RG            57.79
DW            62.400 usec
DE            6.50 usec
TE            298.0 K
D1            1.00000000 sec
TDO           1
----- CHANNEL f1 -----
SFO1          400.1324710 MHz
NUC1          1H
P1            8.97 usec
SI            65536
SF            400.1300109 MHz
WOW           EM
SSB           0
LB            0.30 Hz
GB            0
PC            1.00

```

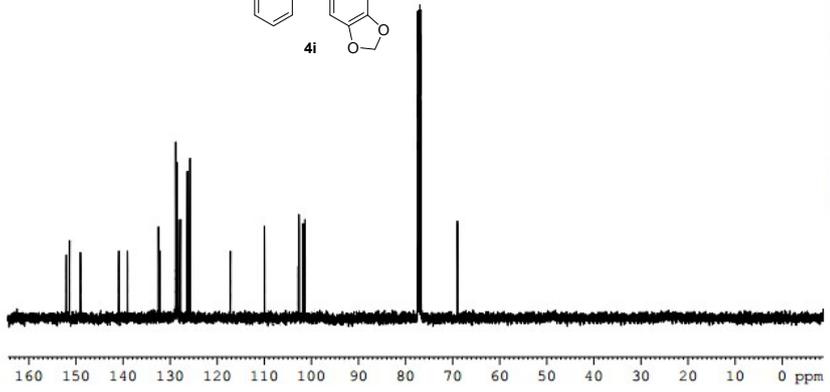
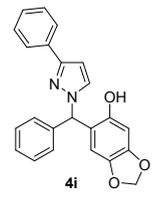
152.0281
 151.3366
 149.0098
 140.8680
 139.0482
 132.4338
 132.0885
 128.7649
 128.4834
 128.3210
 127.7832
 126.3173
 123.7562
 117.1810
 109.9349
 102.5931
 101.6881
 101.3023

77.3447
 77.0270
 76.7097
 68.9373



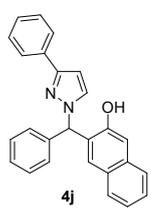
```

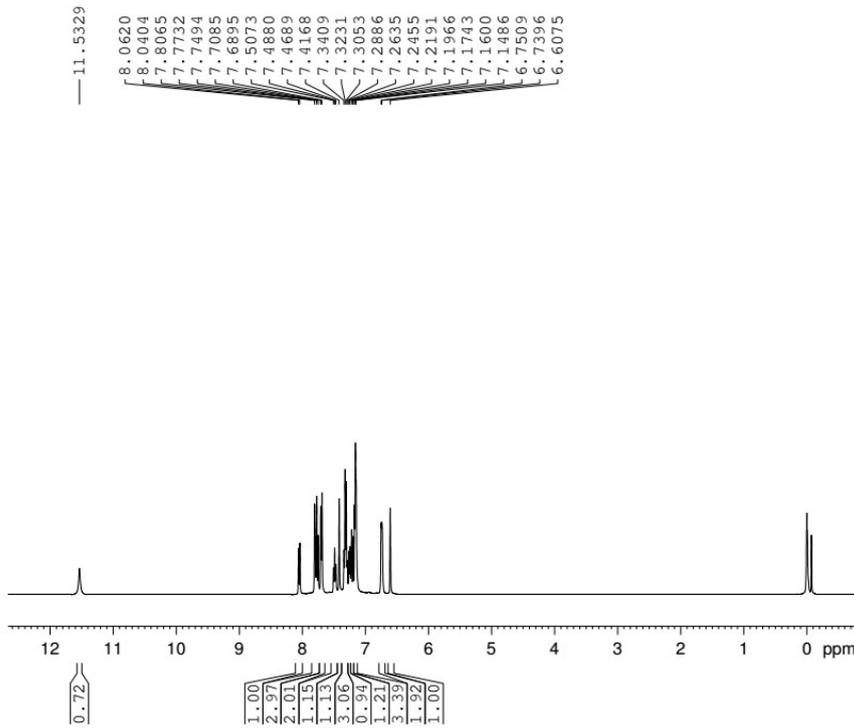
NAME      160730-2MF
EXPNO    4
PROCNO   1
Date_    20160730
Time     7.43
INSTRUM  spect
PROBHD   5 mm PABBO BB/
PULPROG  zgpg30
TD        65536
SOLVENT  CDCl3
NS        256
DS        0
SWH       24038.461 H
FIDRES   0.366798 H
AQ        1.3631988 s
RG        194.26
DW        20.600 u
DE        6.50 u
TE        300.0 K
D1        2.0000000 s
D11       0.0300000 s
TD0       1
  
```



```

----- CHANNEL f1 -----
SFO1    100.6228293 M
NUC1     13C
P1       8.59 u
S1       32768
SF       100.6127685 M
WDM      EM
SGB      0
LS       1.00 H
GB       0
PC       1.40
  
```



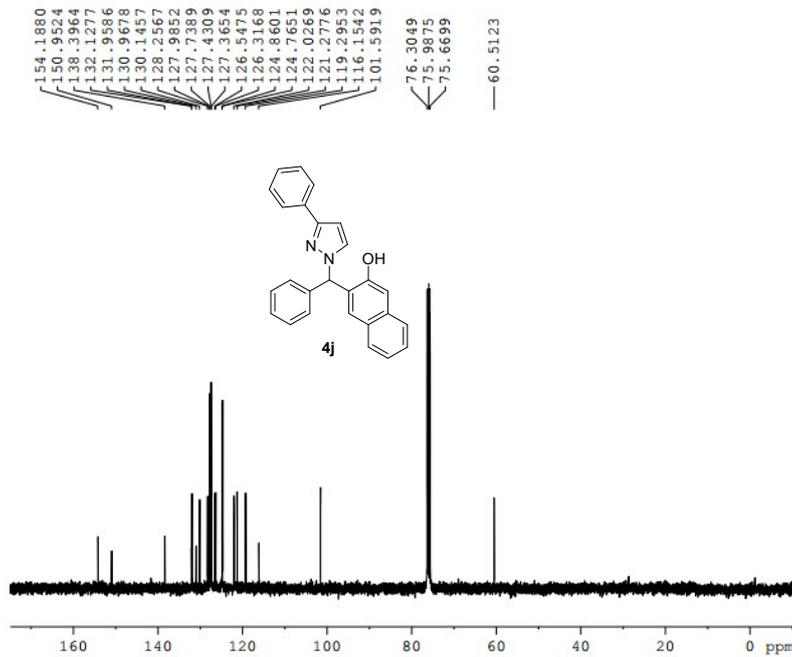


```

NAME      160512-na1
EXPNO     1
PROCNO    1
Date_     20160523
Time      21.52
INSTRUM   spect
PROBHD    5 mm PABBO BB/
PULPROG   zg30
TD         65536
SOLVENT   CDCl3
NS         16
DS         2
SWH        8012.820 Hz
FIDRES     0.122266 Hz
AQ         4.0894966 sec
RG         77.71
DW         62.400 usec
DE         6.50 usec
TE         300.0 K
D1         1.00000000 sec
TDO        1
  
```

```

----- CHANNEL f1 -----
SFO1      400.1324710 MHz
NUC1       1H
P1         8.97 usec
SI         65536
SF         400.1300437 MHz
WDW        EM
SSB        0
LB         0.30 Hz
GB         0
PC         1.00
  
```

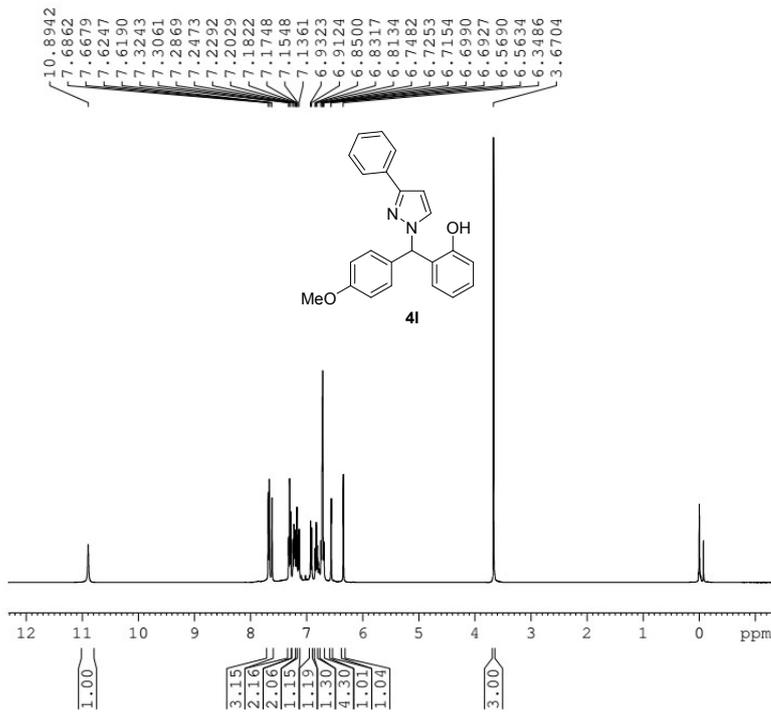


```

NAME      160512-na1
EXPNO     2
PROCNO    1
Date_     20160523
Time      22.22
INSTRUM   spect
PROBHD    5 mm PABBO BB/
PULPROG   zgpg30
TD         65536
SOLVENT   CDCl3
NS         512
DS         0
SWH        24038.461 Hz
FIDRES     0.366798 Hz
AQ         1.3631988 sec
RG         194.26
DW         20.880 usec
DE         6.50 usec
TE         300.0 K
D1         2.00000000 sec
D11        0.03000000 sec
TDO        1
  
```

```

----- CHANNEL f1 -----
SFO1      100.6228293 MHz
D1        13C
P1         8.59 usec
SI         32768
SF         100.6129732 MHz
WDW        EM
SSB        0
LB         1.00 Hz
GB         0
PC         1.40
  
```

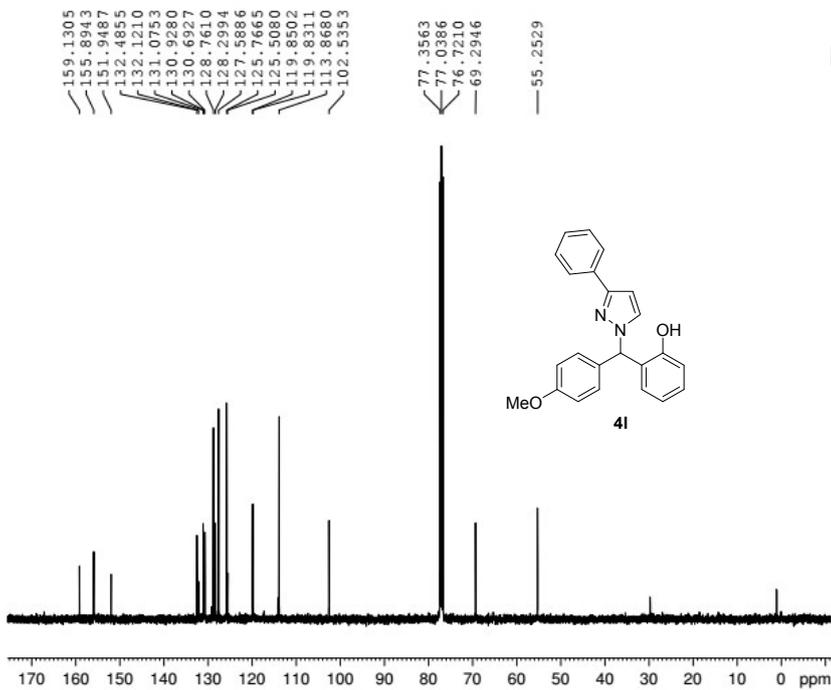



```

NAME      3j-m-ome
EXPNO    1
PROCNO   1
Date_    20160329
Time     18.46
INSTRUM  spect
PROBHD   5 mm PABBO BB/
PULPROG  zg30
TD       65536
SOLVENT  cdcl3
NS       16
DS       2
SWH      8012.820 Hz
FIDRES   0.122266 Hz
AQ       4.0894966 sec
RG       32.77
DW       62.400 usec
DE       6.50 usec
TE       298.3 K
D1       1.0000000 sec
TD0      1
  
```

```

----- CHANNEL f1 -----
SFO1    400.1324710 MHz
NUC1     1H
P1       8.97 usec
SI      65536
SF      400.1300440 MHz
WDW      EM
SSB      0
LB       0.30 Hz
GB       0
PC       1.00
  
```

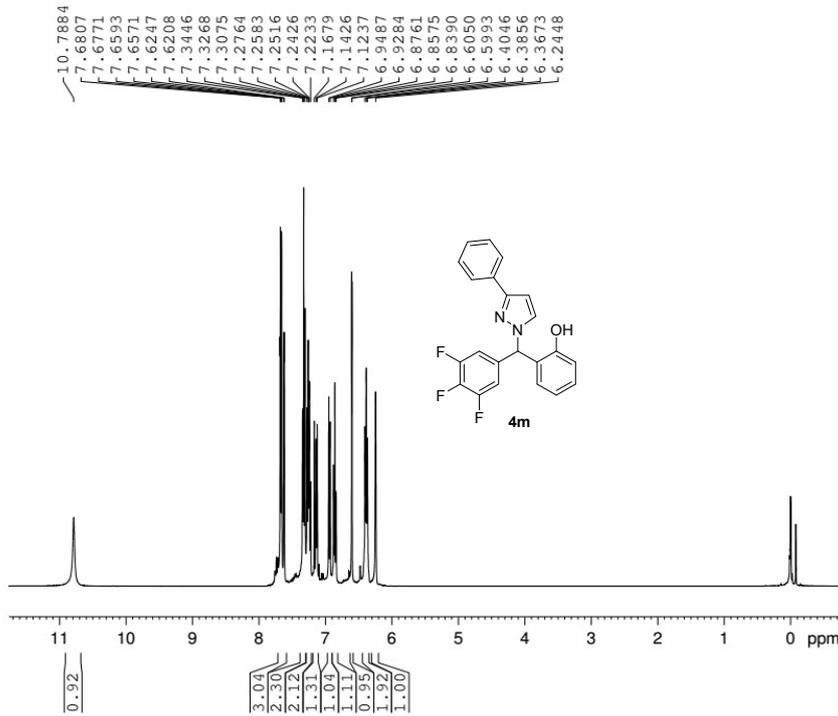


```

NAME      3j-p-ome
EXPNO    2
PROCNO   1
Date_    20160329
Time     19.17
INSTRUM  spect
PROBHD   5 mm PABBO BB/
PULPROG  zgpg30
TD       65536
SOLVENT  cdcl3
NS       512
DS       4
SWH      24038.461 H
FIDRES   0.366798 H
AQ       1.3631988 s
RG       194.26
DW       20.800 u
DE       6.50 u
TE       298.3 K
D1       2.0000000 s
D11      0.0300000 s
TD0      1
  
```

```

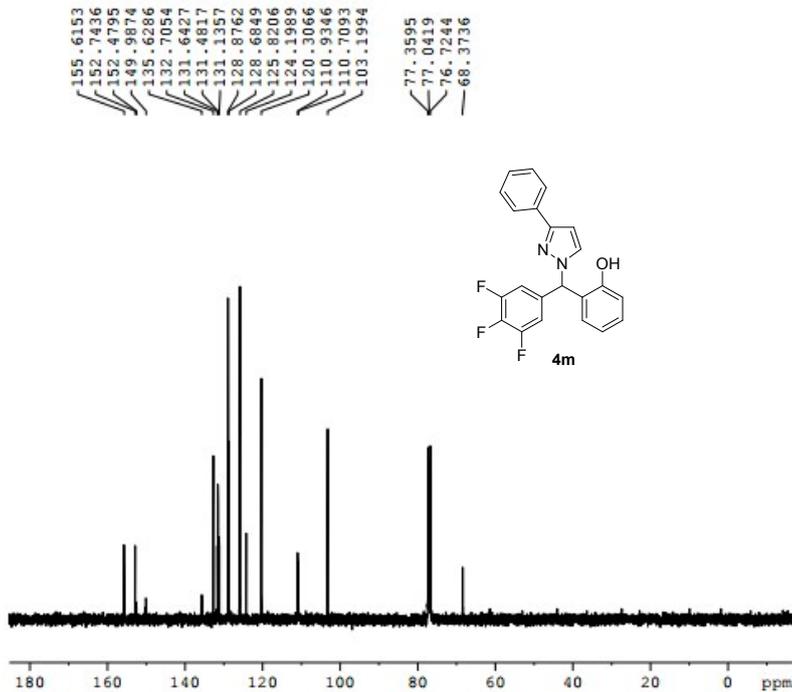
----- CHANNEL f1 -----
SFO1    100.6228293 M
NUC1     13C
P1       8.59 u
SI      32768
SF      100.6127685 M
WDW      EM
SSB      0
LB       1.00 H
GB       0
PC       1.40
  
```



```

NAME          31-345f
EXPNO         1
PROCNO        1
Date_         20160329
Time          20.28
INSTRUM       spect
PROBHD        5 mm FAPBBO BB/
PULPROG       zg30
TD            65536
SOLVENT       CDCl3
NS            16
DS            2
SWH           8012.820 Hz
FIDRES        0.122266 Hz
AQ            4.0894966 sec
RG            32.77
DW            62.400 usec
DE            6.50 usec
TE            298.3 K
D1            1.0000000 sec
D11           1
TDO           1

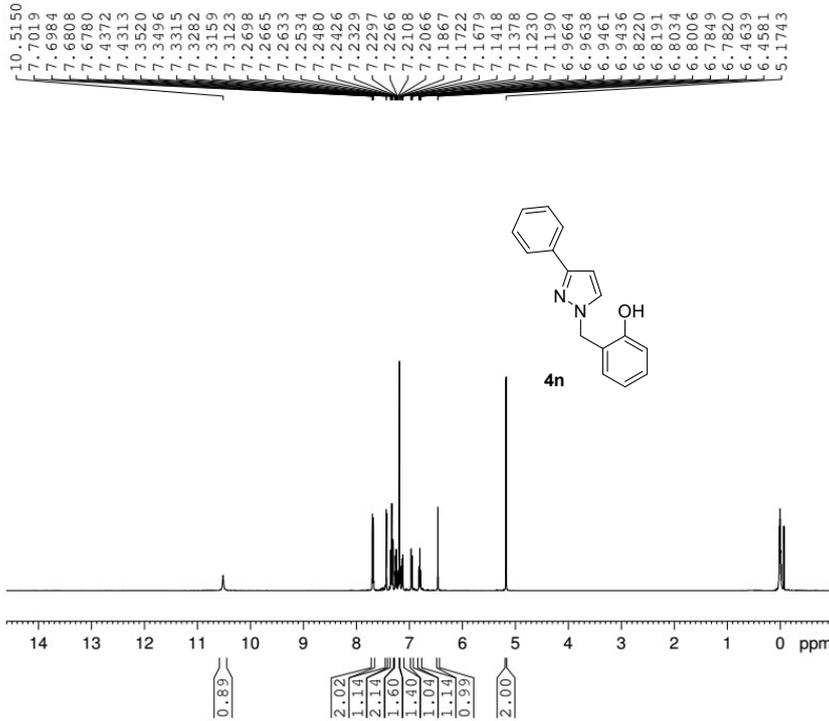
----- CHANNEL f1 -----
SFO1          400.1324710 MHz
NUC1          1H
P1            8.97 usec
SI            65536
SF            400.1300467 MHz
WDW           EM
SSB           0
LB            0.30 Hz
GB            0
PC            1.00
  
```



```

NAME          31-345f
EXPNO         2
PROCNO        1
Date_         20160329
Time          20.30
INSTRUM       spect
PROBHD        5 mm FAPBBO BB/
PULPROG       zgpg30
TD            65536
SOLVENT       CDCl3
NS            256
DS            4
SWH           24038.461 Hz
FIDRES        0.366798 Hz
AQ            1.3631988 sec
RG            194.26
DW            20.800 usec
DE            6.50 usec
TE            298.3 K
D1            2.8000000 sec
D11           0.0300000 sec
TDO           1

----- CHANNEL f1 -----
SFO1          100.6228293 MHz
NUC1          13C
P1            8.59 usec
SI            32768
SF            100.6127685 MHz
WDW           EM
SSB           0
LB            1.00 Hz
GB            0
PC            1.40
  
```

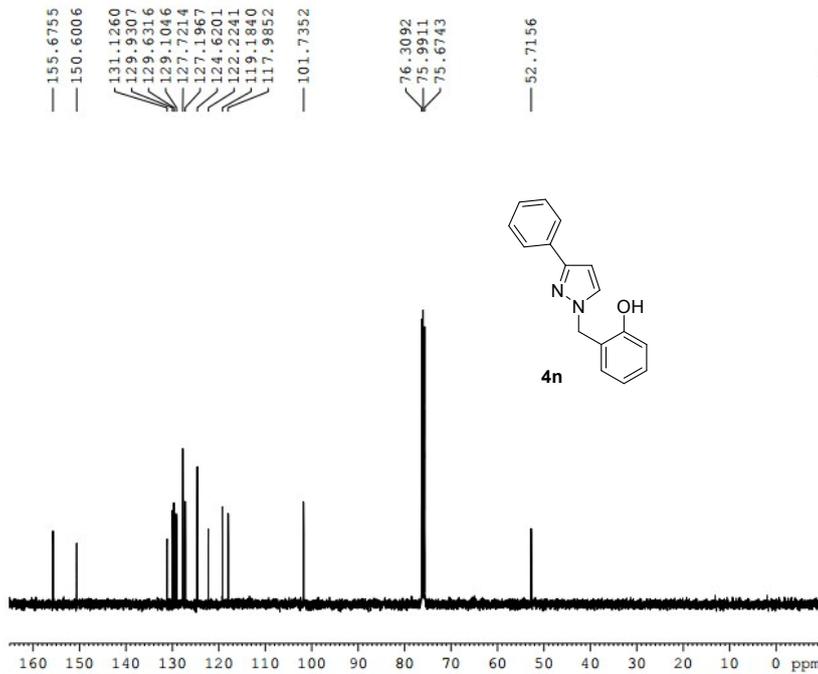


```

NAME      160730-SYC-up
EXPNO    1
PROCNO   1
Date_    20160730
Time     8.28
INSTRUM  spect
PROBHD   5 mm PABBO BB/
PULPROG  zgpg30
TD       65536
SOLVENT  CDCl3
NS       16
DS       2
SWH      8012.820 H
FIDRES   0.122266 H
AQ       4.089496 sec
RG       70.36
DW       62.400 u
DE       6.50 u
TE       300.0 K
D1       1.00000000 sec
TDO      1
  
```

```

----- CHANNEL f1 -----
SFO1     400.1324710 M
NUC1     1H
P1       8.97 u
SI       65536
SF       400.1300391 M
WDW      EM
SSB      0
LB       0.30 H
GB       0
PC       1.00
  
```

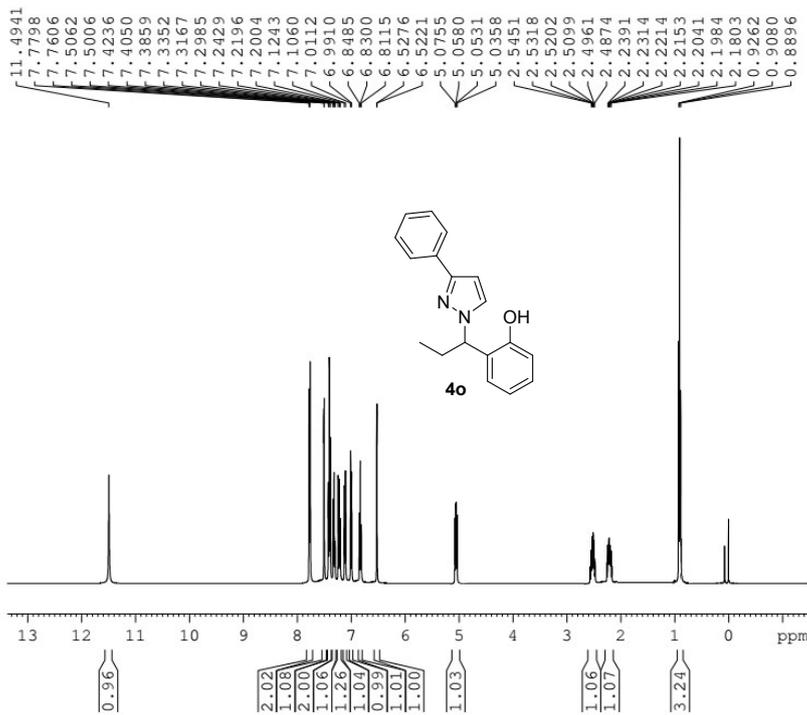


```

NAME      160730-SYC-up
EXPNO    2
PROCNO   1
Date_    20160730
Time     8.43
INSTRUM  spect
PROBHD   5 mm PABBO BB/
PULPROG  zgpg30
TD       65536
SOLVENT  CDCl3
NS       256
DS       0
SWH      24038.481 Hz
FIDRES   0.366798 Hz
AQ       1.3631988 sec
RG       194.26
DW       20.800 usec
DE       6.50 usec
TE       300.0 K
D1       2.00000000 sec
D11      0.03000000 sec
TDO      1
  
```

```

----- CHANNEL f1 -----
SFO1     100.6228293 MHz
NUC1     13C
P1       8.59 usec
SI       32768
SF       100.6128719 MHz
WDW      EM
SSB      0
LB       1.00 Hz
GB       0
PC       1.40
  
```

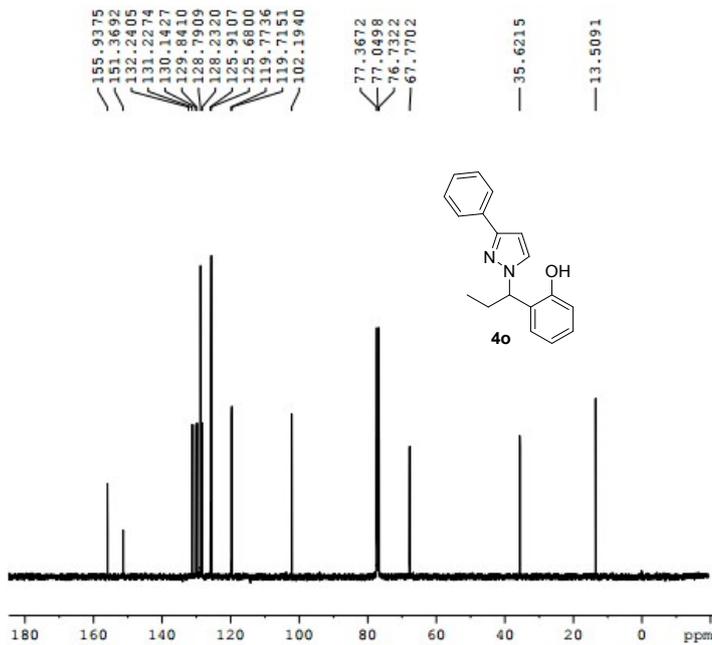


```

NAME          3m-et
EXPNO         1
PROCNO        1
Date_         20160329
Time          19.22
INSTRUM       spect
PROBHD        5 mm PABBO BB/
PULPROG       zg30
TD            65536
SOLVENT       CDCl3
NS            16
DS            2
SWH           8012.820 Hz
FIDRES        0.122266 Hz
AQ            4.0894966 sec
RG            32.77
DW            62.400 usec
DE            6.50 usec
TE            298.3 K
D1            1.00000000 sec
TD0           1
  
```

```

----- CHANNEL f1 -----
SFO1          400.1324710 MHz
NUC1           1H
P1            8.97 usec
SI            65536
SF            400.1300166 MHz
WDW           EM
SSB           0
LB            0.30 Hz
GB            0
PC            1.00
  
```

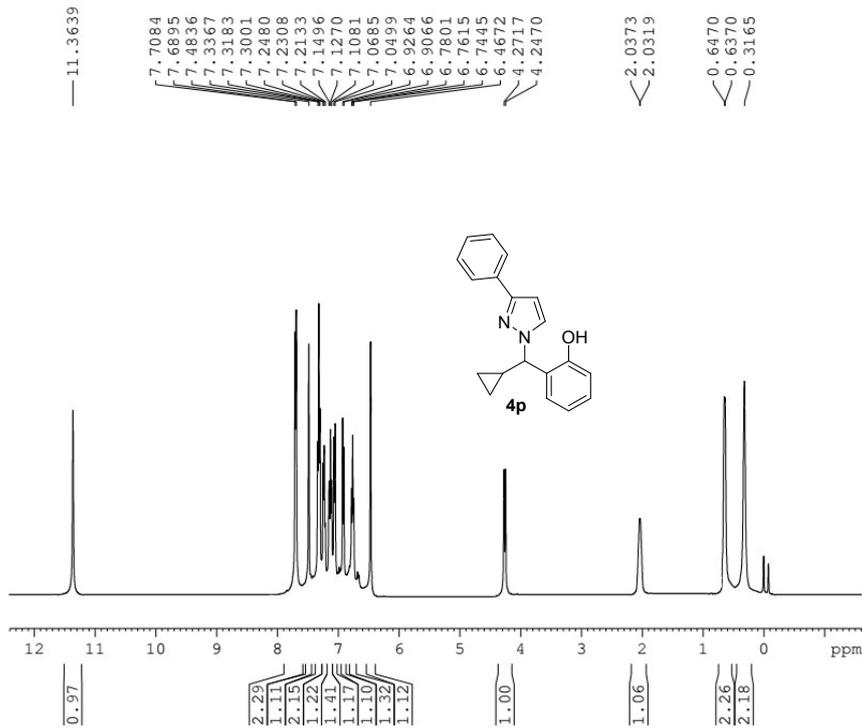


```

NAME          3m-et
EXPNO         1
PROCNO        1
Date_         20160329
Time          19.53
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            194.26
DW            20.800 usec
DE            6.50 usec
TE            298.3 K
D1            2.00000000 sec
D11           0.03000000 sec
TD0           1
  
```

```

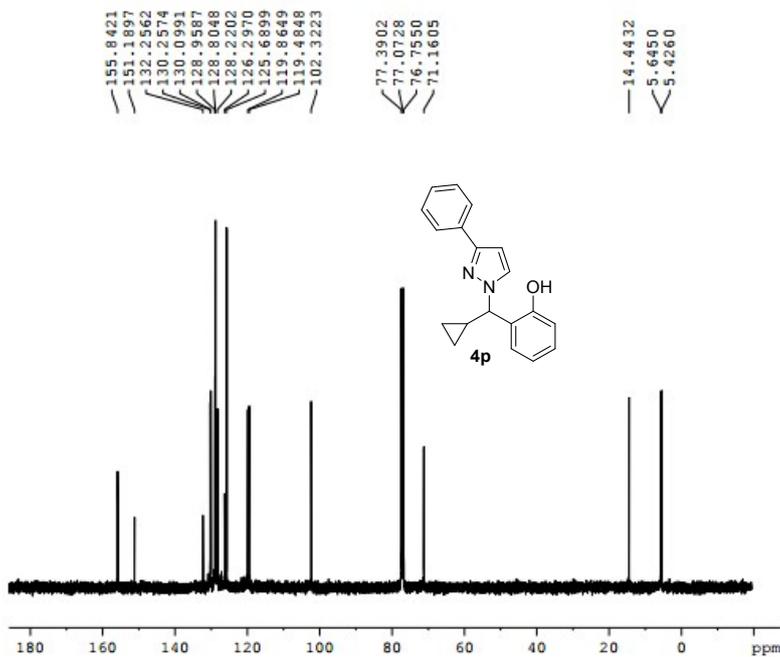
----- CHANNEL f1 -----
SFO1          100.6228293 MHz
NUC1          13C
P1            8.59 usec
SI            32768
SF            100.6127685 MHz
WDW           EM
SSB           0
LB            1.00 Hz
GB            0
PC            1.40
  
```



```

NAME          3n-cp
EXPNO         1
PROCNO        1
Date_         20160329
Time          20.00
INSTRUM       spect
PROBHD        5 mm PABBO BB/
PULPROG       zg30
TD            65536
SOLVENT       CDCl3
NS            16
DS            2
SWH           8012.820 Hz
FIDRES        0.122266 Hz
AQ            4.0894966 sec
RG            32.77
DW            62.400 usec
DE            6.50 usec
TE            298.3 K
D1            1.00000000 sec
TD0           1

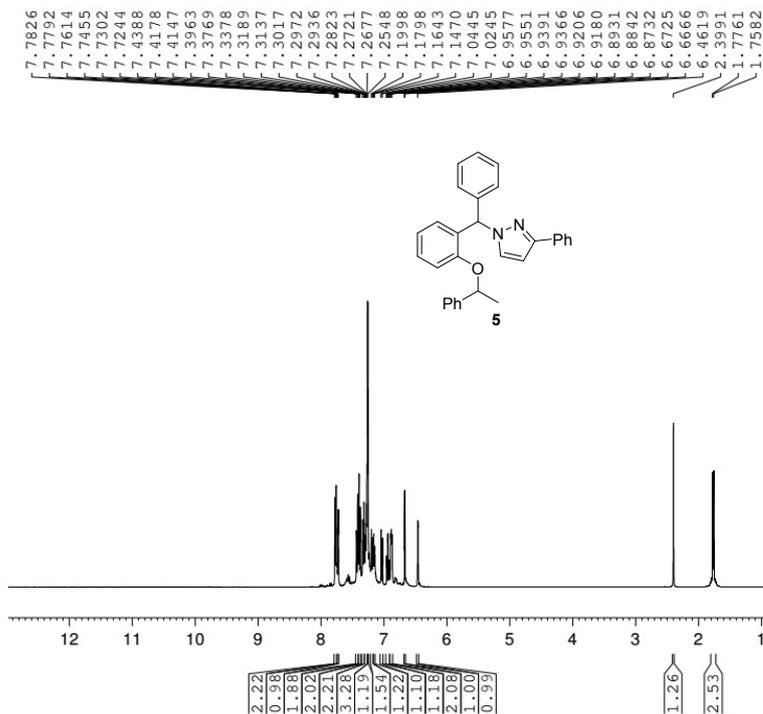
----- CHANNEL f1 -----
SFO1          400.1324710 MHz
NUC1          1H
P1            8.97 usec
SI            65536
SF            400.1300523 MHz
WDW           EM
SSB           0
LB            0.30 Hz
GB            0
PC            1.00
  
```



```

NAME          3n-cp
EXPNO         1
PROCNO        1
Date_         20160329
Time          20.07
INSTRUM       spect
PROBHD        5 mm PABBO BB/
PULPROG       zgpg30
TD            65536
SOLVENT       CDCl3
NS            256
DS            4
SWH           24038.461 Hz
FIDRES        0.366798 Hz
AQ            1.3651988 sec
RG            194.26
DW            20.800 usec
DE            6.50 usec
TE            298.3 K
D1            2.00000000 sec
D11           0.03000000 sec
TD0           1

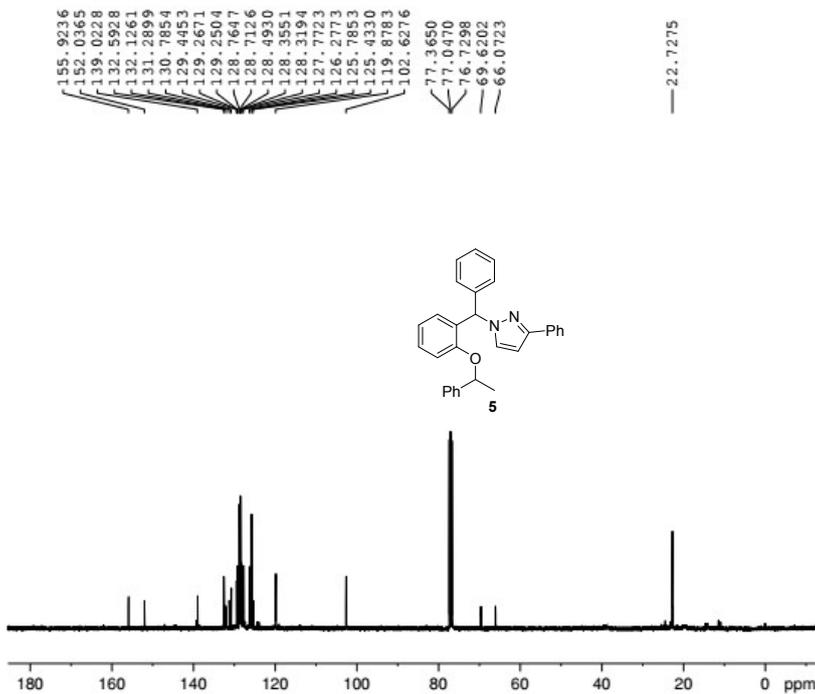
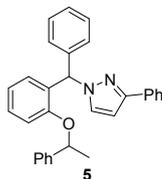
----- CHANNEL f1 -----
SFO1          100.6228293 MHz
NUC1          13C
P1            8.53 usec
SI            32768
SF            100.6127685 MHz
WDW           EM
SSB           0
LB            1.00 Hz
GB            0
PC            1.40
  
```



```

NAME      171025-J1-170901-6y
EXPNO     1
PROCNO    1
Date_     20171025
Time      14.10
INSTRUM   spect
PROBHD    5 mm PABBO BB/
PULPROG   zg30
TD        65536
SOLVENT   cdcl3
NS        12
DS        0
SWH       8012.820 Hz
FIDRES    0.122266 Hz
AQ        4.0894966 sec
RG        26.24
DW        62.400 usec
DE        6.50 usec
TE        300.0 K
D1        1.00000000 sec
TD0       1

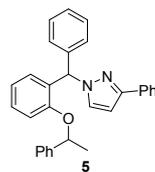
----- CHANNEL f1 -----
SF01      400.1324710 MHz
NUCL1     1H
F1        8.04 usec
SI        65536
SF        400.1300099 MHz
WDW       EM
SSB       0
LB        0.30 Hz
GB        0
PC        1.00
  
```



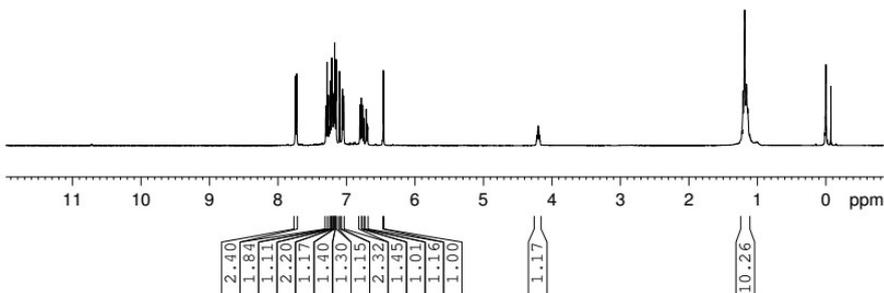
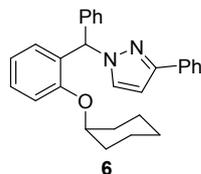
```

NAME      171025-J1-170901-6y
EXPNO     2
PROCNO    1
Date_     20171101
Time      19.43
INSTRUM   spect
PROBHD    5 mm PABBO BB/
PULPROG   zgpg30
TD        65536
SOLVENT   cdcl3
NS        432
DS        0
SWH       24038.461 Hz
FIDRES    0.366798 Hz
AQ        1.3631988 sec
RG        194.26
DW        20.800 usec
DE        6.50 usec
TE        300.0 K
D1        2.00000000 sec
D11       0.03000000 sec
TD0       1

----- CHANNEL f1 -----
SF01      100.6228293 MHz
NUCL1     13C
F1        8.54 usec
SI        32768
SF        100.6127685 MHz
WDW       EM
SSB       0
LB        1.00 Hz
GB        0
PC        1.40
  
```



7.7487
7.7453
7.7277
7.7250
7.3023
7.2983
7.2840
7.2329
7.2146
7.2097
7.2049
7.1924
7.1863
7.1737
7.1677
7.1569
7.1510
7.1020
7.0618
7.0579
7.0428
6.8035
6.7838
6.7678
6.7493
6.7472
6.7132
6.7090
6.6942
6.6900
6.4630
6.4571
4.2270
4.2186
4.2086
4.1996
4.1905
4.1805
4.1721
1.2108
1.1846
1.1757
1.1652
1.1579
1.1403

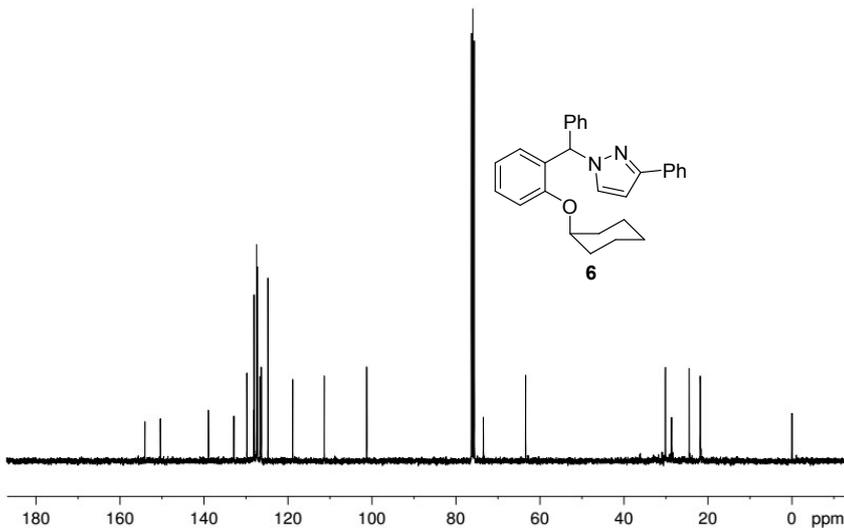
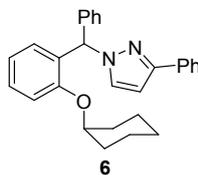


```

NAME      YSY-XAJ-N15-huanji
EXPNO    1
PROCNO   1
Date_    20180122
Time     19.28
INSTRUM  spect
PROBHD   5 mm PABBO BB/
PULPROG  zg30
TD       65536
SOLVENT  CDCl3
NS       12
DS       0
SWH      8012.820 Hz
FIDRES   0.122266 Hz
AQ       4.0894966 sec
RG       51.19
DW       62.400 usec
DE       6.50 usec
TE       300.0 K
D1       1.0000000 sec
TD0      1

----- CHANNEL f1 -----
SFO1    400.1324710 MHz
NUC1     1H
P1       8.04 usec
SI       65536
SF       400.1300441 MHz
WDW      EM
SSB      0
LB       0.30 Hz
GB       0
PC       1.00
  
```

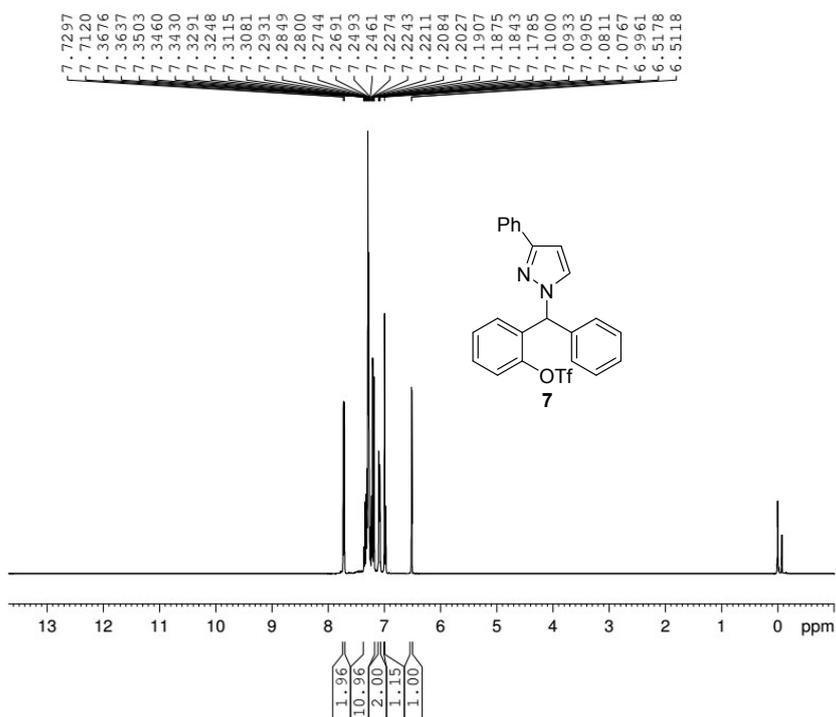
154.0348
150.3558
138.9108
132.8643
129.7517
128.1491
128.0250
127.4327
127.3394
127.1854
126.5408
126.3469
124.7302
118.8455
111.3402
101.2424
76.3111
75.9931
75.6759
73.4857
63.4159
30.1489
30.1309
28.6774
24.4955
21.8339



```

NAME      YSY-XAJ-N15-huanj
EXPNO    2
PROCNO   1
Date_    20180122
Time     19.58
INSTRUM  spect
PROBHD   5 mm PABBO BB/
PULPROG  zgpg30
TD       65536
SOLVENT  CDCl3
NS       512
DS       0
SWH      24038.461 Hz
FIDRES   0.366798 Hz
AQ       1.3631988 se
RG       194.26
DW       20.800 us
DE       6.50 us
TE       300.1 K
D1       2.0000000 se
D11      0.03000000 se
TD0      1

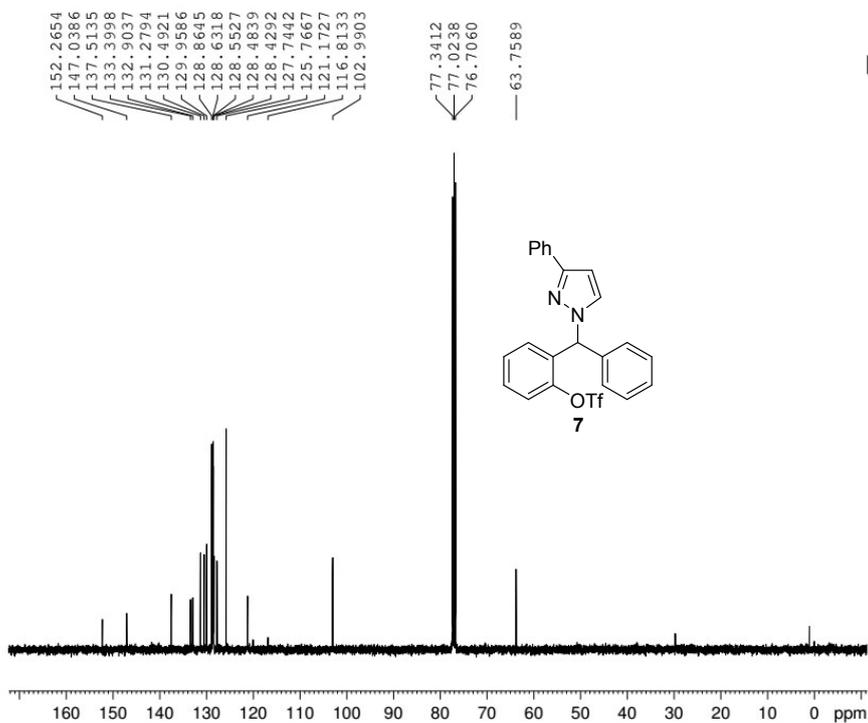
----- CHANNEL f1 -----
SFO1    100.6228293 MH
NUC1     13C
P1       8.54 us
SI       32768
SF       100.6128730 MH
WDW      EM
SSB      0
LB       1.00 Hz
GB       0
PC       1.40
  
```



```

NAME      160730-step1
EXPNO     5
PROCNO    1
Date_     20160730
Time      4.47
INSTRUM   spect
PROBHD    5 mm PABBO BB/
PULPROG   zg30
TD         65536
SOLVENT   CDCl3
NS         16
DS         2
SWH        8012.820 I
FIDRES     0.122266 I
AQ         4.0894966 I
RG         70.36
DW         62.400 I
DE         6.50 I
TE         300.1 I
D1         1.0000000 I
TDO        1
----- CHANNEL f1 -----
SF01      400.1324710 I
NUC1       1H
P1         8.97 I
SI         65536
SF         400.1300424 I
WDW        EM
SSB        0
LB         0.30 I
GB         0
PC         1.00

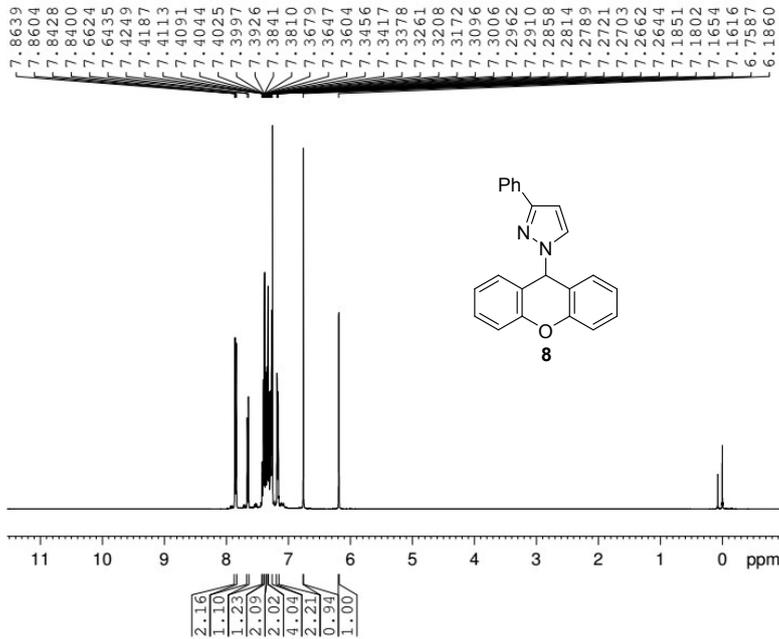
```



```

NAME      160730-step1
EXPNO     6
PROCNO    1
Date_     20160730
Time      5.03
INSTRUM   spect
PROBHD    5 mm PABBO BB/
PULPROG   zgpg30
TD         65536
SOLVENT   CDCl3
NS         256
DS         0
SWH        24038.461 H
FIDRES     0.366798 H
AQ         1.3631988 H
RG         194.26
DW         20.800 u
DE         6.50 u
TE         300.0 K
D1         2.0000000 H
D11        0.0300000 H
TDO        1
----- CHANNEL f1 -----
SF01      100.6228293 M
NUC1       13C
P1         8.59 u
SI         32768
SF         100.6127685 M
WDW        EM
SSB        0
LB         1.00 H
GB         0
PC         1.40

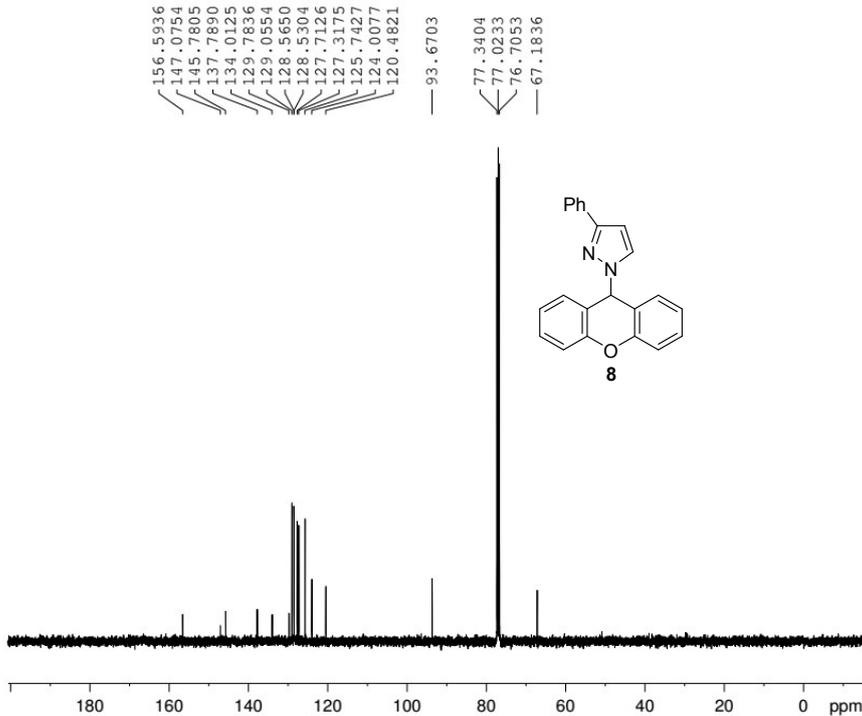
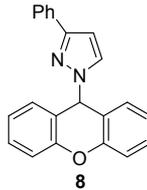
```



```

NAME 160730-step2-down:
EXPNO 5
PROCNO 1
Date_ 20160730
Time 3.27
INSTRUM spect
PROBHD 5 mm PABBO BB/
PULPROG zg30
TD 65536
SOLVENT CDCl3
NS 16
DS 2
SWH 8012.820 Hz
FIDRES 0.122266 Hz
AQ 4.0894966 sec
RG 77.71
DW 62.400 usec
DE 6.50 usec
TE 300.0 K
D1 1.00000000 sec
TD0 1

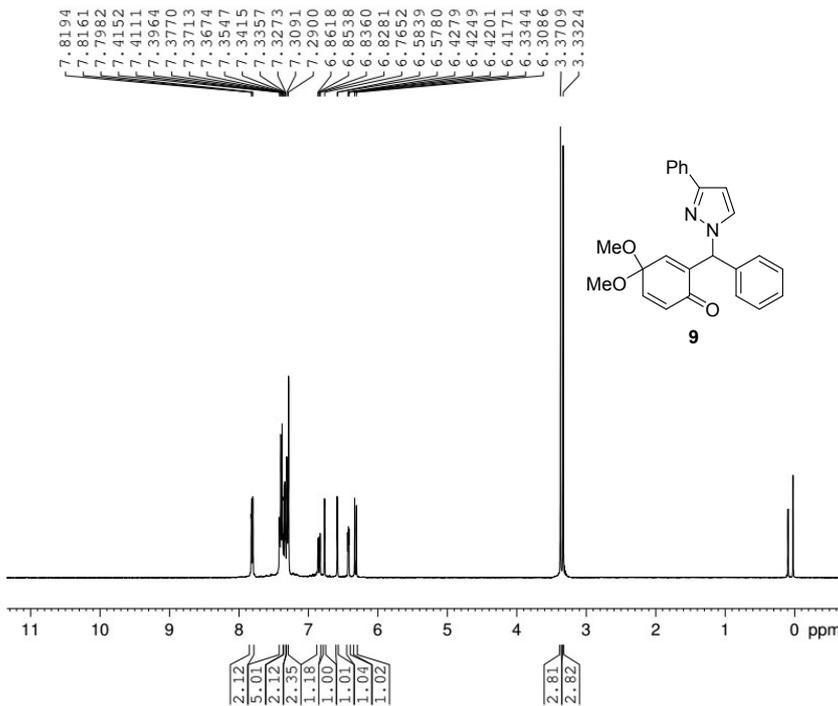
----- CHANNEL f1 -----
SFO1 400.1324710 MHz
NUC1 1H
P1 8.97 usec
SI 65536
SF 400.1300118 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00
  
```



```

NAME 160730-step2-down:
EXPNO 6
PROCNO 1
Date_ 20160730
Time 3.43
INSTRUM spect
PROBHD 5 mm PABBO BB/
PULPROG zgpg30
TD 65536
SOLVENT CDCl3
NS 256
DS 0
SWH 24038.461 Hz
FIDRES 0.366798 Hz
AQ 1.3631988 sec
RG 194.26
DW 20.800 usec
DE 6.50 usec
TE 300.0 K
D1 2.00000000 sec
D11 0.03000000 sec
TD0 1

----- CHANNEL f1 -----
SFO1 100.6228293 MHz
NUC1 13C
P1 8.59 usec
SI 32768
SF 100.6127685 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40
  
```

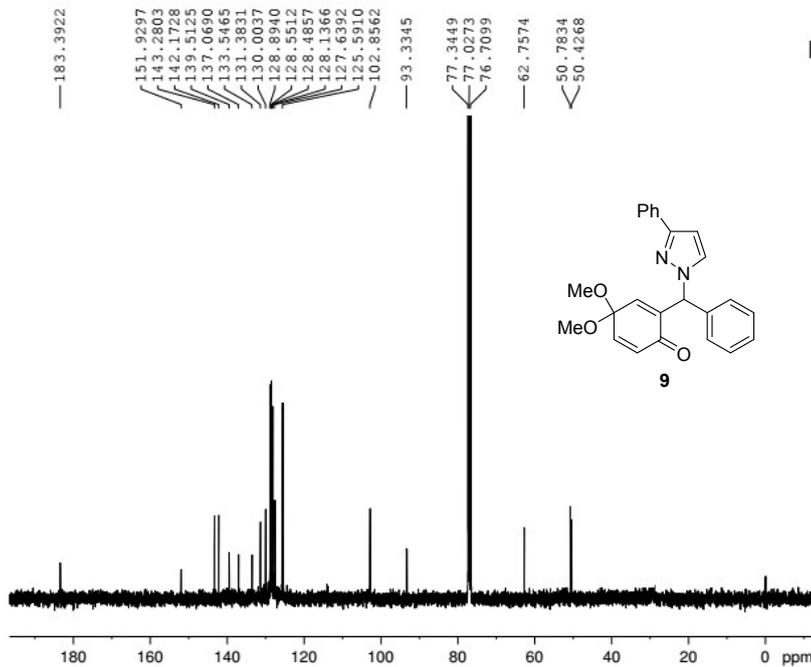


```

NAME      180128-yanshengyan
EXPNO    1
PROCNO   1
Date_    20180128
Time     14.41
INSTRUM  spect
PROBHD   5 mm PABBO BB-
PULPROG  zg30
TD       65536
SOLVENT  CDCl3
NS       2
DS       16
SWH      8223.685 Hz
FIDRES   0.125483 Hz
AQ       3.9846387 sec
RG       203
DW       60.800 usec
DE       6.50 usec
TE       295.1 K
D1       1.00000000 sec
TD0      1
  
```

```

----- CHANNEL f1 -----
NUC1     1H
P1       13.60 usec
PL1      -2.60 dB
PL1W     18.28352737 W
SFO1     400.1524711 MHz
SI       32768
SF       400.1500000 MHz
WDW      EM
SSB      0
LB       0.30 Hz
GB       0
PC       1.00
  
```



```

NAME      171025-J3-170901-1
EXPNO    2
PROCNO   1
Date_    20171110
Time     8.14
INSTRUM  spect
PROBHD   5 mm PABBO BB/
PULPROG  zgpg30
TD       65536
SOLVENT  CDCl3
NS       512
DS       0
SWH      24038.461 Hz
FIDRES   0.366798 Hz
AQ       1.3631988 sec
RG       194.26
DW       20.800 usec
DE       6.50 usec
TE       300.0 K
D1       2.00000000 sec
D11      0.03000000 sec
TD0      1
  
```

```

----- CHANNEL f1 -----
SFO1     100.6228293 MHz
NUC1     13C
P1       8.54 usec
SI       32768
SF       100.6127685 MHz
WDW      EM
SSB      0
LB       1.00 Hz
GB       0
PC       1.40
  
```

VI-Structure of compound 4I

CCDC 1582529 (compound **4I**) contains the supplementary crystallographic data for this paper. These data can be obtained by free of charge on application to the Director, CCDC 12 Union Road, Cambridge CB2 1EZ, UK (fax (+44) 1223-336033; or e-mail deposit@ccdc.cam.ac.uk) or via www.ccdc.cam.ac.uk/data_request/cif

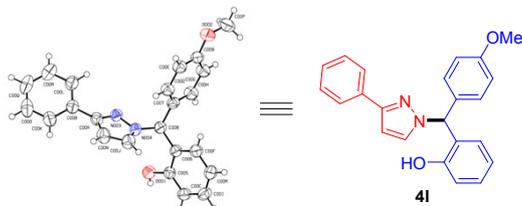


Table 1. Crystal data and structure refinement for Compound **4I**

Identification code	4I
Empirical formula	C ₂₃ H ₂₀ N ₂ O ₂
Formula weight	356.41
Temperature/K	293.1(3)
Crystal system	monoclinic
Space group	P2 ₁ /c
a/Å	9.34970(18)
b/Å	9.15478(16)
c/Å	21.8715(4)
α/°	90
β/°	92.8985(15)
γ/°	90
Volume/Å ³	1869.68(6)
Z	4
ρ _{calc} /cm ³	1.266
μ/mm ⁻¹	0.649
F(000)	752.0
Crystal size/mm ³	0.8 × 0.35 × 0.2
Radiation	CuKα (λ = 1.54184)
2θ range for data collection/°	9.472 to 145.056
Index ranges	-11 ≤ h ≤ 11, -7 ≤ k ≤ 11, -17 ≤ l ≤ 26
Reflections collected	10657
Independent reflections	3641 [R _{int} = 0.0325, R _{sigma} = 0.0244]
Data/restraints/parameters	3641/0/246
Goodness-of-fit on F ²	1.027
Final R indexes [I ≥ 2σ (I)]	R ₁ = 0.0505, wR ₂ = 0.1394
Final R indexes [all data]	R ₁ = 0.0574, wR ₂ = 0.1512
Largest diff. peak/hole / e Å ⁻³	0.22/-0.24