

Regioselectivity-tunable Self-1,3-dipolar [3+3] Cyclizations of Azomethine Ylides to Assemble Dispirooxindole-piperazines

Peng-Ju Xia,^{†,§} Yan-Hua Sun,^{†,§} Jun-An Xiao,[†] Zhao-Fang Zhou,[†]

Sai-shuai Wen,[†] Yu Xiong,[†] Guang-Chuan Ou,[‡] Xiao-Qing Chen,[†] and

Hua Yang^{*,†}

[†] College of Chemistry and Chemical Engineering, Central South University, Changsha 410083, P.

R. China

[‡] Department of Biology and Chemistry, Hunan University of Science and Engineering, Yongzhou,

Hunan 425100, P. R. China

§Peng-Ju Xia and Yan-Hua Sun contributed equally.

e-mail: hyangchem@csu.edu.cn

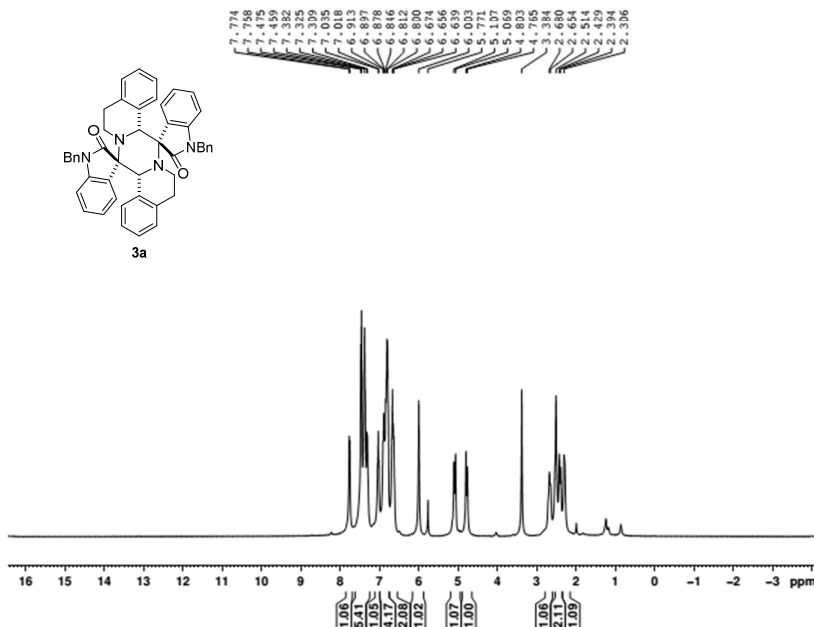
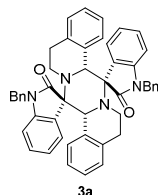
¹H NMR and ¹³C NMR spectra for compounds **3a**, **3a'**, **3b**, **3b'**, **3c**, **3c'**, **3d**, **3d'**, **3e**, **3e'**, **3f**, **3f'**, **3g**, **3h**, **3i**, **3i'**, **5a**, **5b**, **5c**, **5d**, **5e**, **5f**, **7a**, **7b**, **7c**, **7d**; X-ray structure of dispirooxindole—piperazine **3a**, **3a'**, **7a**.

Dispirooxindole-piperazine 3a	S3
Dispirooxindole-piperazine 3a'	S4
Dispirooxindole-piperazine 3b	S5
Dispirooxindole-piperazine 3b'	S6
Dispirooxindole-piperazine 3c	S7
Dispirooxindole-piperazine 3c'	S8
Dispirooxindole-piperazine 3d	S9
Dispirooxindole-piperazine 3d'	S10
Dispirooxindole-piperazine 3e	S11
Dispirooxindole-piperazine 3e'	S12
Dispirooxindole-piperazine 3f	S13
Dispirooxindole-piperazine 3f'	S14
Dispirooxindole-piperazine 3g	S15
Dispirooxindole-piperazine 3h	S16
Dispirooxindole-piperazine 3i	S17

Dispirooxindole-piperazine 3i'	S18
Dispirooxindole-piperazine 5a	S19
Dispirooxindole-piperazine 5b	S20
Dispirooxindole-piperazine 5c	S21
Dispirooxindole-piperazine 5d	S22
Dispirooxindole-piperazine 5e	S23
Dispirooxindole-piperazine 5f	S24
Dispirooxindole-piperazine 7a	S25
Dispirooxindole-piperazine 7b	S26
Dispirooxindole-piperazine 7c	S27
Dispirooxindole-piperazine 7d	S28
Crystal structure of dispirooxindole-piperazine 3a	S29
Crystal structure of dispirooxindole-piperazine 3a'	S31
Crystal structure of dispirooxindole-piperazine 7a	S33

^1H NMR (400 MHz; DMSO- d_6), ^{13}C NMR (100 MHz; DMSO- d_6)

H195-2



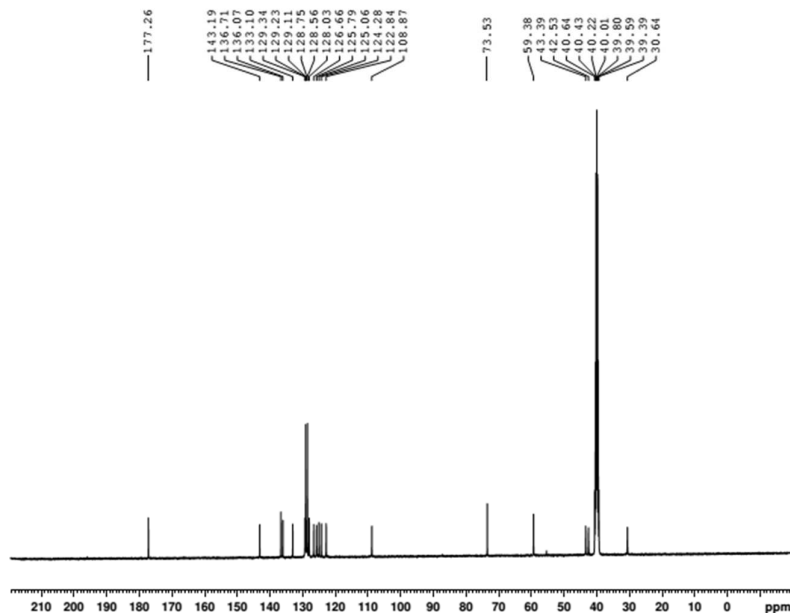
Current Data Parameters
NAME 01-28-2015
EXPNO 7
PROCNO 1

F2 - Acquisition Parameters
Date_ 20150128
Time 15.57
INSTRUM spect
PROBHD 5 mm PABBO BB-
PULPROG zg30
TD 65536
SOLVENT DMSO
NS 84
DS 2
SWH 8223.685 Hz
FIDRES 0.125483 Hz
AQ 3.9846387 sec
RG 79.3
DW 60.800 usec
DE 6.50 usec
TE 292.3 K
D1 1.00000000 sec

***** CHANNEL f1 *****
NUC1 ^1H
P1 14.00 usec
PLW1 9.72999954 W
SFO1 400.1324710 MHz

F2 - Processing parameters
SI 65536
SF 400.1300000 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00

H195-2



Current Data Parameters
NAME 01-28-2015
EXPNO 11
PROCNO 1

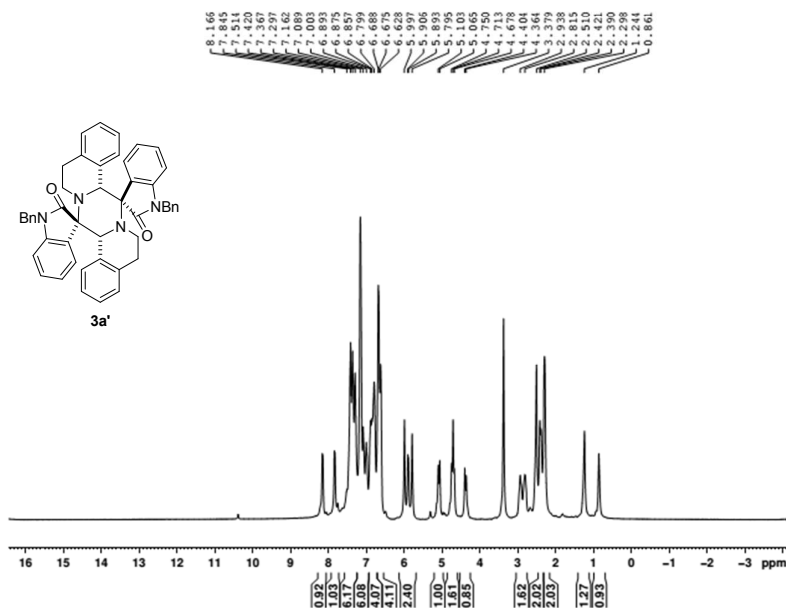
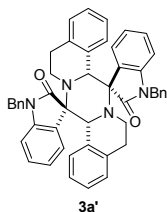
F2 - Acquisition Parameters
Date_ 20150204
Time 10.55
INSTRUM spect
PROBHD 5 mm PABBO BB-
PULPROG zgpg30
TD 65536
SOLVENT DMSO
NS 3852
DS 4
SWH 24038.461 Hz
FIDRES 0.366798 Hz
AQ 1.3631988 sec
RG 168.03
DW 20.800 usec
DE 6.50 usec
TE 292.3 K
D1 2.00000000 sec
D11 0.03000000 sec

***** CHANNEL f1 *****
NUC1 ^{13}C
P1 9.00 usec
PLW1 50.09999847 W
SFO1 100.6228293 MHz

***** CHANNEL f2 *****
CPDPRG2 waltz16
NUC2 ^1H
PCPD2 90.00 usec
PLW2 9.36999989 W
PLW12 0.22673000 W
PLW13 0.18945000 W
SFO2 400.1316005 MHz

F2 - Processing parameters
SI 32768
SF 100.6127690 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40

H195-1



```

Current Data Parameters
=====
NAME      01-28-2015
EXPNO     6
PROCNO    1

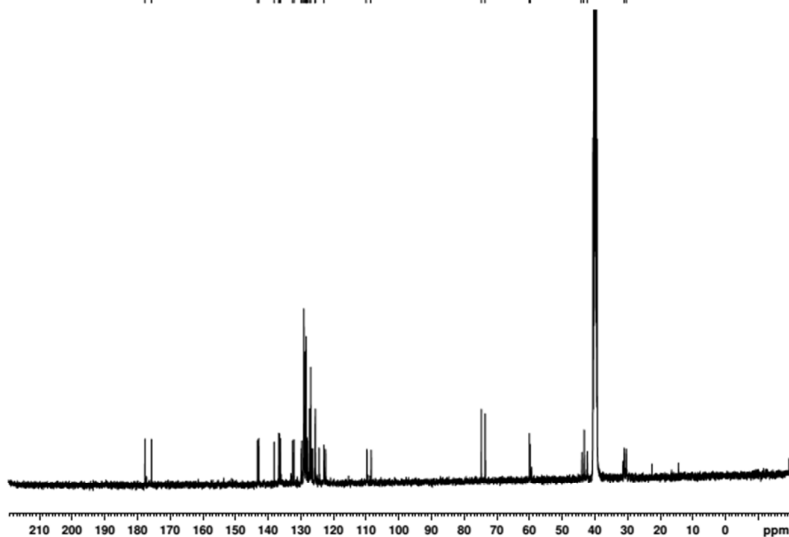
F2 - Acquisition Parameters
=====
Date_     20150128
Time      15.37
INSTRUM    spect
PROBHD     5 mm PABBO BB-
PULPROG    zg30
TD          65536
SOLVENT
NS          72
DS          2
FIDRES     0.125483 Hz
AQ          3.9846387 sec
WDW         79.3 Hz
DE          60.800 usec
CW          6.50 usec
DI          292.1 K
D1          1.00000000 sec

===== CHANNEL f1 =====
NUC1        1H
P1          14.00 usec
PLM1        9.72999954 Hz
SFO1        400.1324710 MHz

F2 - Processing parameters
=====
SI          65536
SF          400.1300000 MHz
WDW         EM
SS          0
LB          0.30 Hz
GB          0
PC          1.00

```

H195-1



```

Current Data Parameters
NAME          01-28-2015
EXPNO         10
PROCNO        1

F2 - Acquisition Parameters
Date_         20150202
Time          16:54
INSTRUM       spect
PROBHD        5 mm PABBO BBO
PULPROG       zgpg30
TD            65536
SOLVENT       DMSO
DS            3164
NS            24038
SWH           461 Hz
FIDRES        0.366798 Hz
AQ            1.363198 sec
RG            152.51
SQ            20.800 usec
DE            6.50 usec
TE            293.4 K
D1            2.0000000 sec
D11           0.0300000 sec

***** CHANNEL f1 *****
NUC1          P1          13C
NUC2          F2          9.00 usec
PLW1          50.999984 MHz
SFO1          100.6228247 MHz

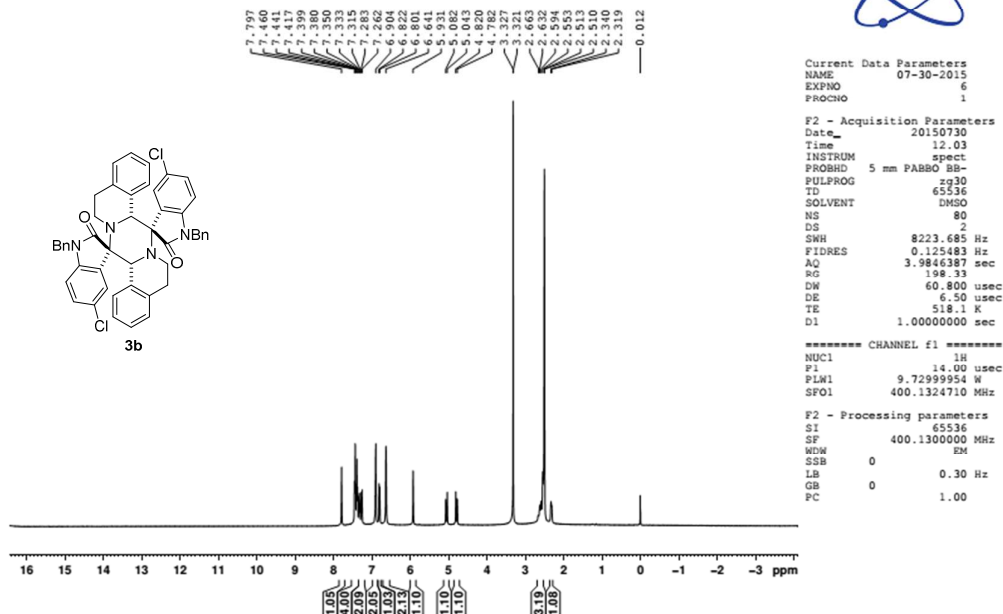
***** CHANNEL f2 *****
NUCPRG2       waltz16
NUC2          1H
PCPD2         P1          90.00 usec
PLW2          9.36999989 MHz
PLM12         0.2267300 MHz
PLM13         0.1836500 MHz
SFO2          400.1316005 MHz

F2 - Processing parameters
SI            32768
SF            100.6127690 MHz
WDW           EM
SSB           0
GB            1.00 Hz
LB            0
GB            0
PC            1.40

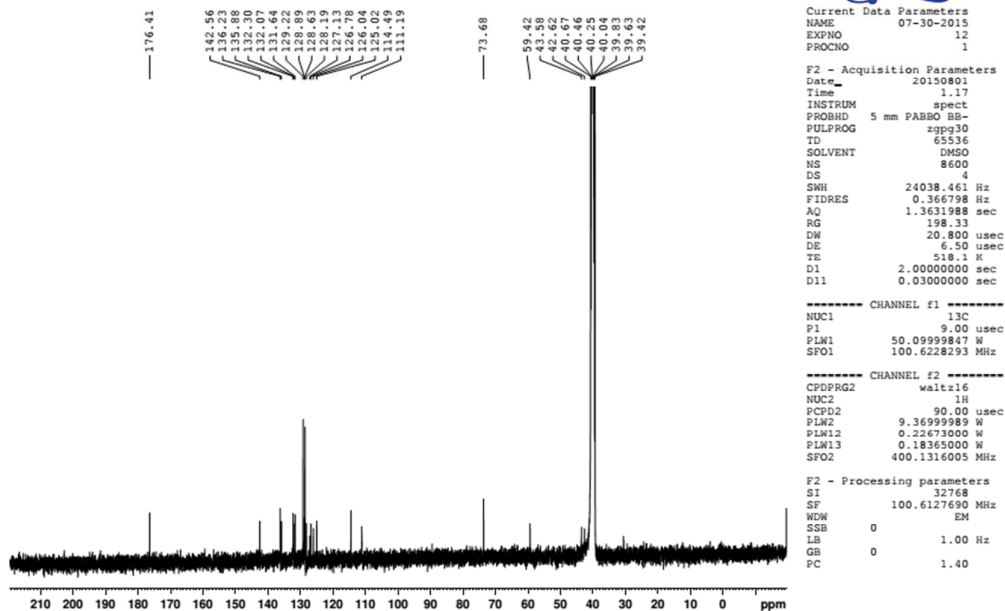
```


^1H NMR (400 MHz; $\text{DMSO-}d_6$), ^{13}C NMR (100 MHz; $\text{DMSO-}d_6$)

XP-16



XP-16



BRUKER

XP-15

The figure displays a ¹H NMR spectrum of compound 3b'. The x-axis represents the chemical shift in ppm, ranging from -4 to 16. The spectrum shows several distinct signals: aromatic protons between 7.0 and 8.3 ppm, aliphatic protons between 2.3 and 3.8 ppm, and a small peak at approximately 1.0 ppm. Integration values are provided below the baseline.

Chemical structure of 3b':

C1=CC=C(C=C1)C(=O)N2[C@H](c3ccccc3)[C@@H](c4cc(Cl)cc4)O[C@H]2Cc5ccccc5Cl

Current Data Parameters
NAME 07-24-2015
EXPNO 2
PROCNO 1

F2 - Acquisition Parameters
Date_ 20150724
Time 15.27
INSTRUM spect
PROBHD 5 mm PABBO BB-
PULPROG zg30
TD 65536
SOLVENT DMSO
NS 54
DS 2
SWH 8223.685 Hz
FIDRES 0.125483 Hz
AQ 3.9846387 sec
RG 124.55
DW 60.800 usec
DE 6.50 usec
TE 518.1 K
D1 1.00000000 sec

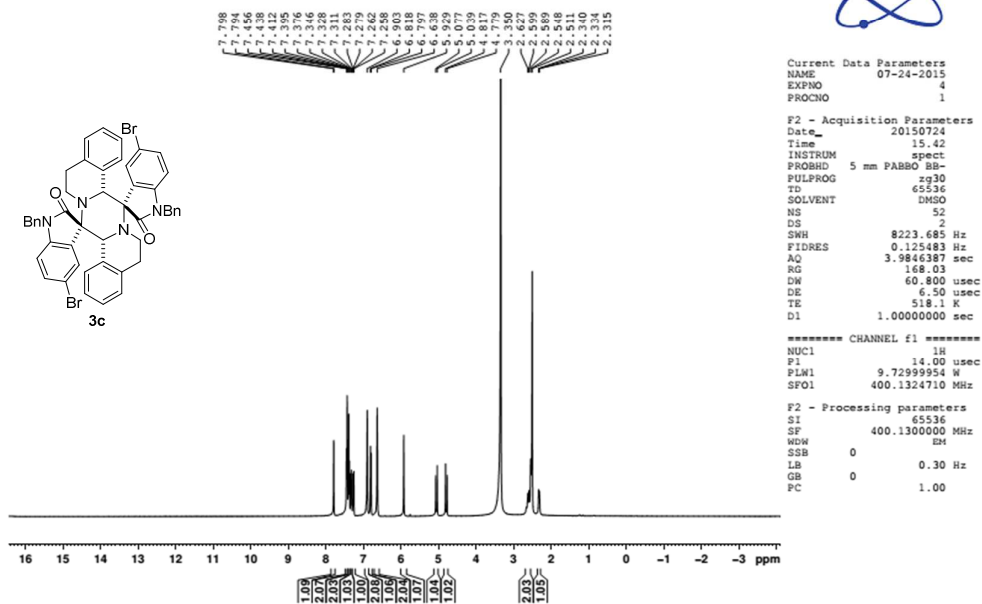
***** CHANNEL f1 *****
NUC1 1H
P1 14.00 usec
PLW1 9.72999954 W
SF01 400.1324710 MHz

F2 - Processing parameters
SI 65536
SF 400.1300000 MHz
WDW EM
SSR 0
LB 0.30 Hz
GB 0
PC 1.00

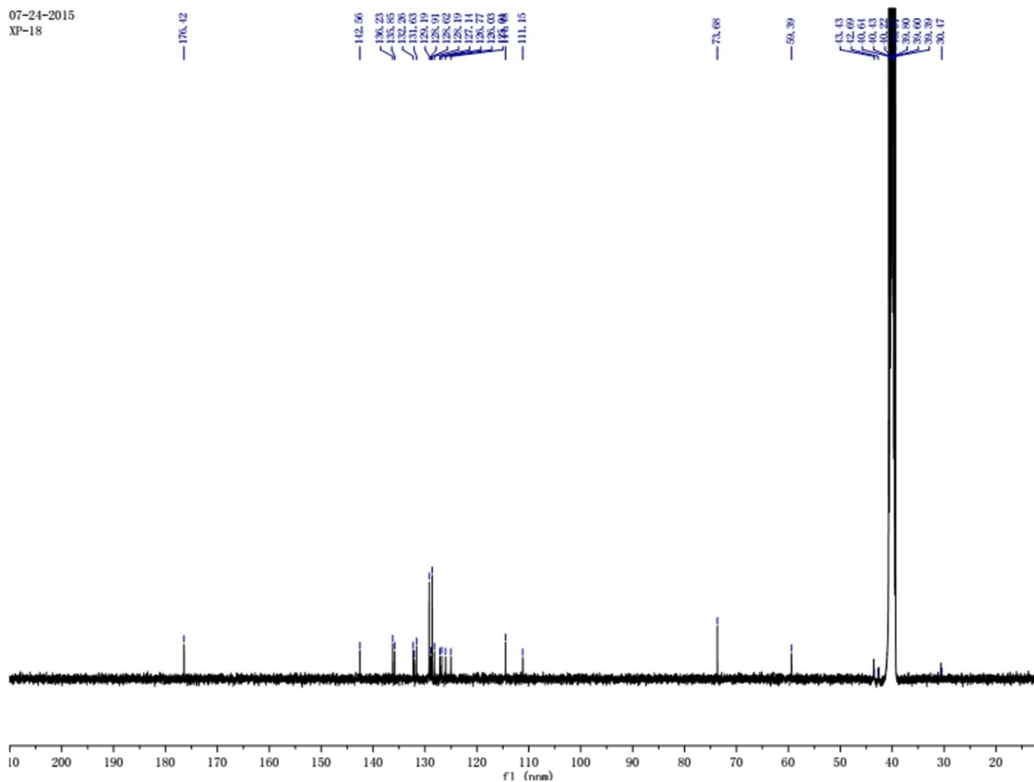


^1H NMR (400 MHz; $\text{DMSO-}d_6$), ^{13}C NMR (100 MHz; $\text{DMSO-}d_6$)

XP-18

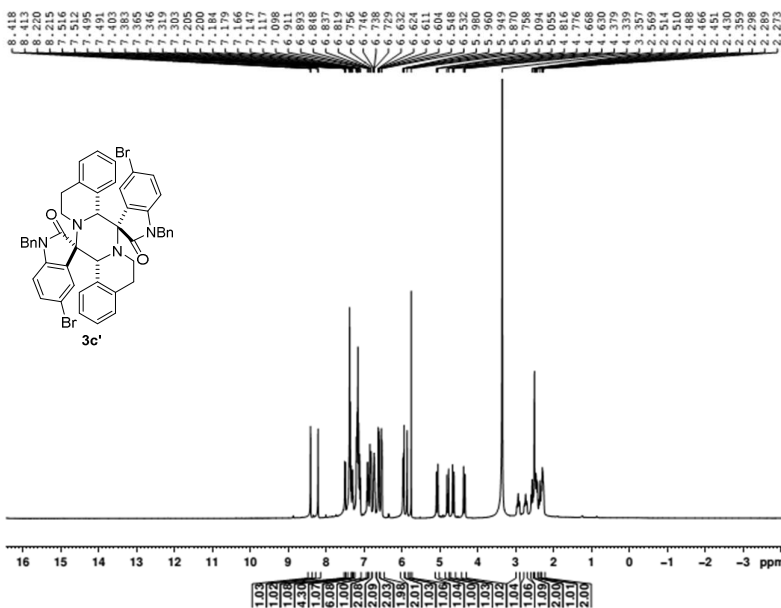


07-24-2015
XP-18



^1H NMR (400 MHz; $\text{DMSO-}d_6$), ^{13}C NMR (100 MHz; $\text{DMSO-}d_6$)

XP-17



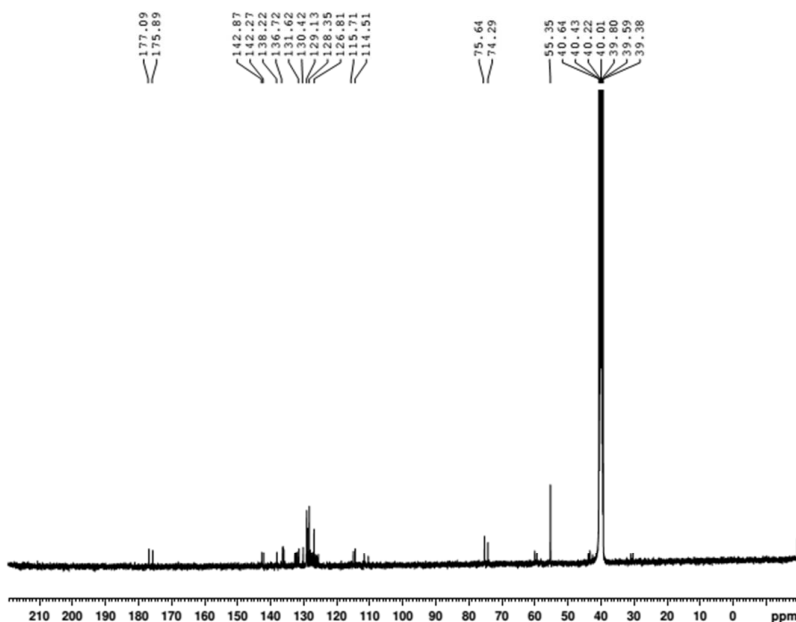
Current Data Parameters
NAME 07-24-2015
EXPNO 3
PROCNO 1

F2 - Acquisition Parameters
Date_ 20150724
Time 15.33
INSTRUM spect
PROBHD 5 mm PABBO BB-
PULPROG zg30
TD 65536
SOLVENT DMSO
NS 64
DS 2
SWH 8223.685 Hz
FIDRES 0.125483 Hz
AQ 3.9846387 sec
RG 124.55
DW 60.800 usec
DE 6.50 usec
TE 518.1 K
D1 1.00000000 sec

***** CHANNEL f1 *****
NUC1 ^1H
P1 14.00 usec
PLW1 9.7299954 W
SFO1 400.1324710 MHz

F2 - Processing parameters
SI 65536
SF 400.1300000 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00

XP-17



Current Data Parameters
NAME 07-24-2015
EXPNO 22
PROCNO 1

F2 - Acquisition Parameters
Date_ 20150730
Time 7.45
INSTRUM spect
PROBHD 5 mm PABBO BB-
PULPROG zgpg30
TD 65536
SOLVENT DMSO
NS 15600
DS 4
SWH 24038.461 Hz
FIDRES 0.366798 Hz
AQ 1.3631988 sec
RG 198.33
DW 20.800 usec
DE 6.50 usec
TE 518.1 K
D1 2.00000000 sec
D11 0.03000000 sec

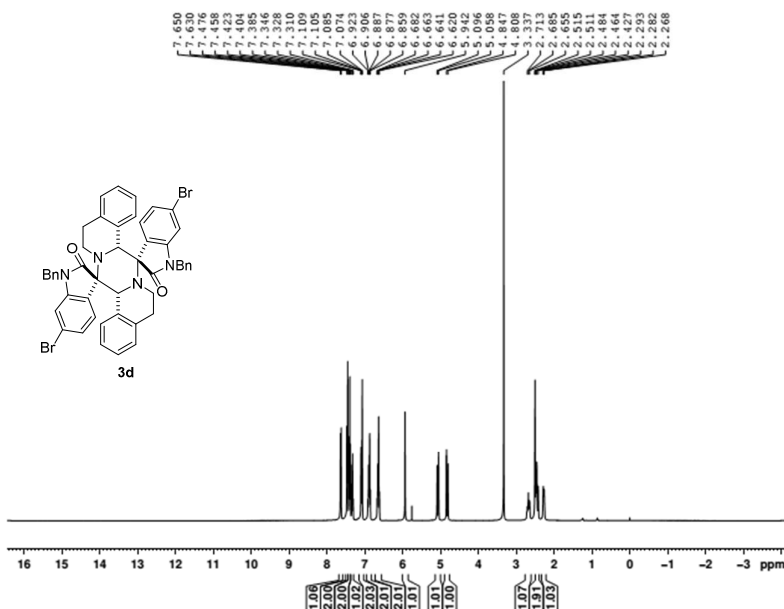
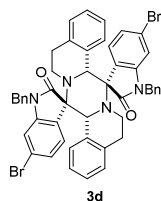
***** CHANNEL f1 *****
NUC1 ^{13}C
P1 9.00 usec
PLW1 50.09999847 W
SFO1 100.6228293 MHz

***** CHANNEL f2 *****
CPDPRG2 waltz16
NUC2 ^1H
PCPD2 90.00 usec
PLW2 9.36999989 W
PLW12 0.22673000 W
PLW13 0.18365000 W
SFO2 400.1316005 MHz

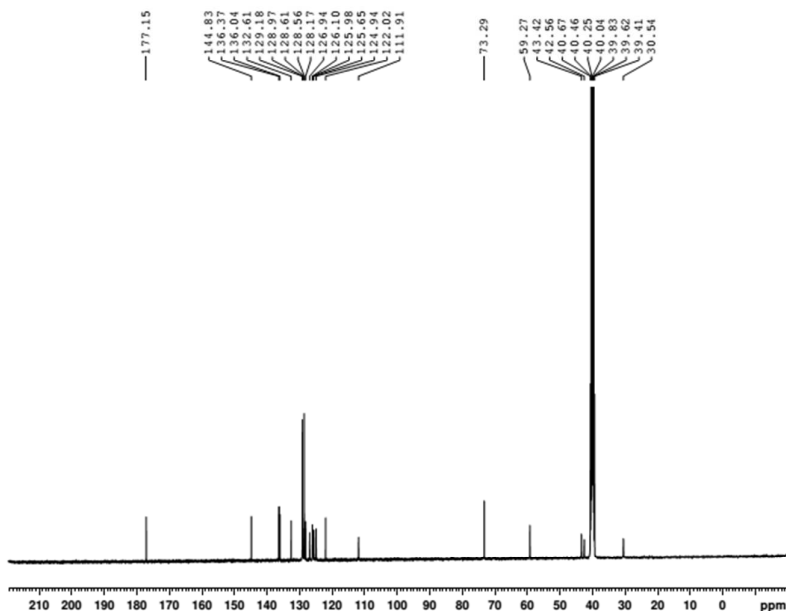
F2 - Processing parameters
SI 32768
SF 100.6127690 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40

^1H NMR (400 MHz; $\text{DMSO-}d_6$), ^{13}C NMR (100 MHz; $\text{DMSO-}d_6$)

XP-33



XP-33

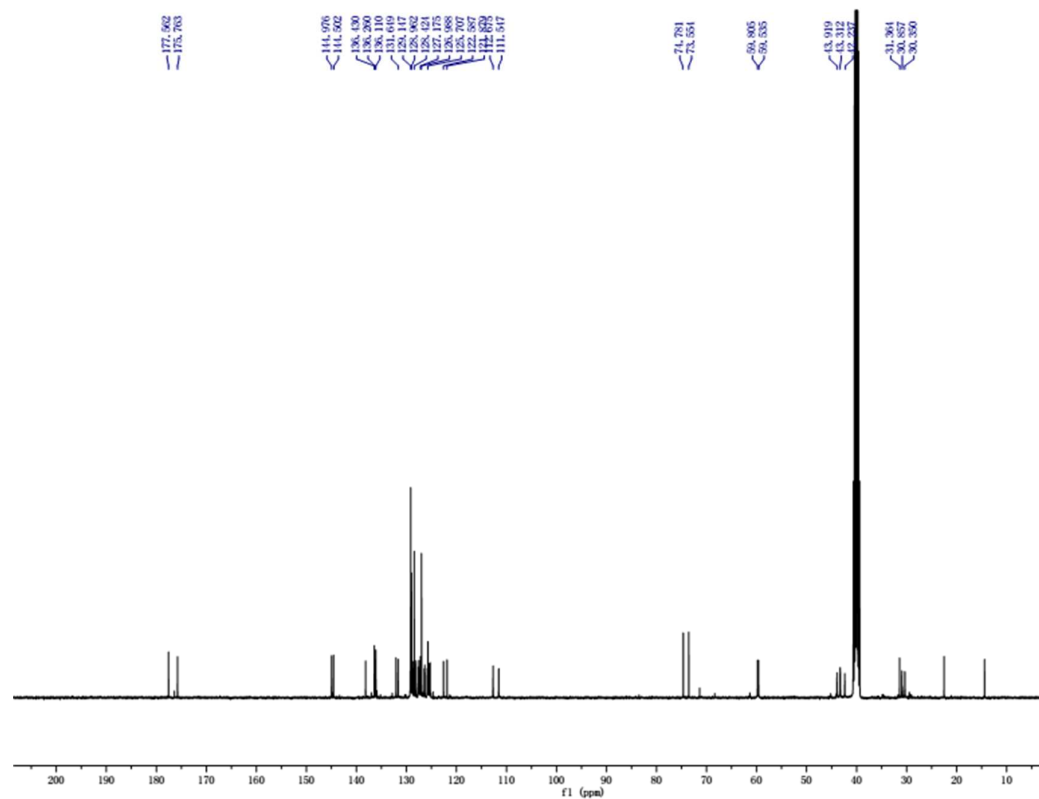


Chemical structure of **3d'** is shown as an inset. The structure is a complex polycyclic molecule featuring a central quaternary carbon atom bonded to two nitrogen atoms (each part of a five-membered ring containing a carbonyl group), two phenyl rings (one substituted with a bromine atom), and a benzyl group.

¹H NMR spectrum (CDCl₃) of compound **3d'**. The x-axis represents the chemical shift in ppm (δ), ranging from 0 to 16. The spectrum shows several multiplets and singlets, with integrations provided below the baseline. The chemical shifts (δ) for the peaks are listed at the top of the spectrum.

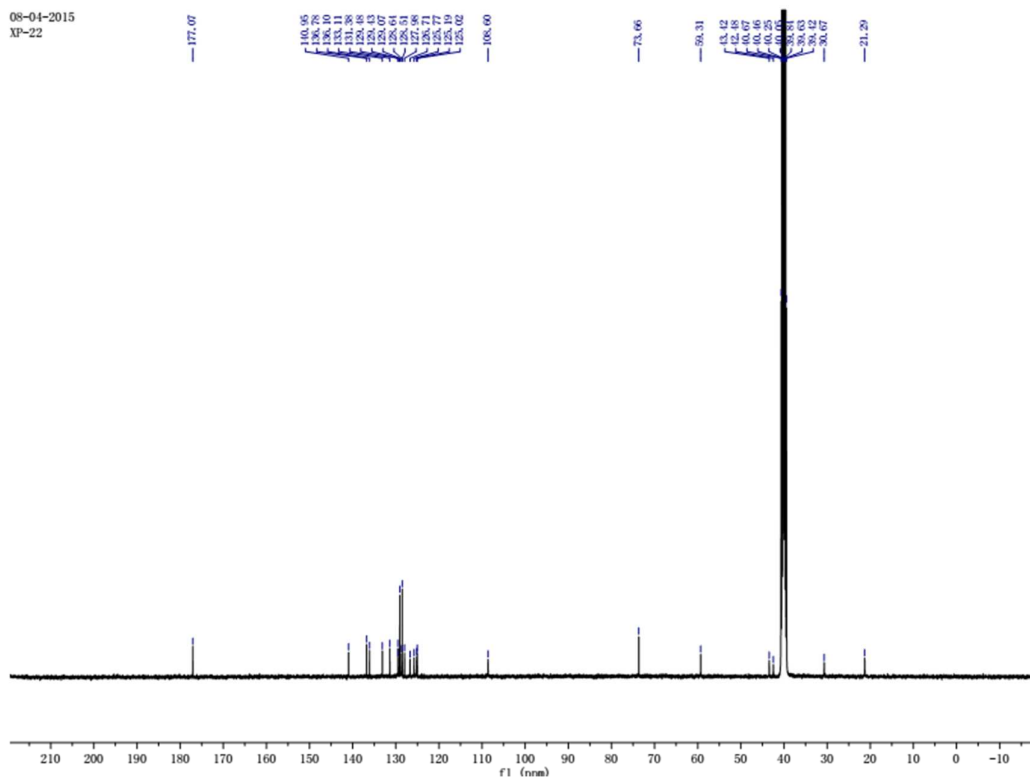
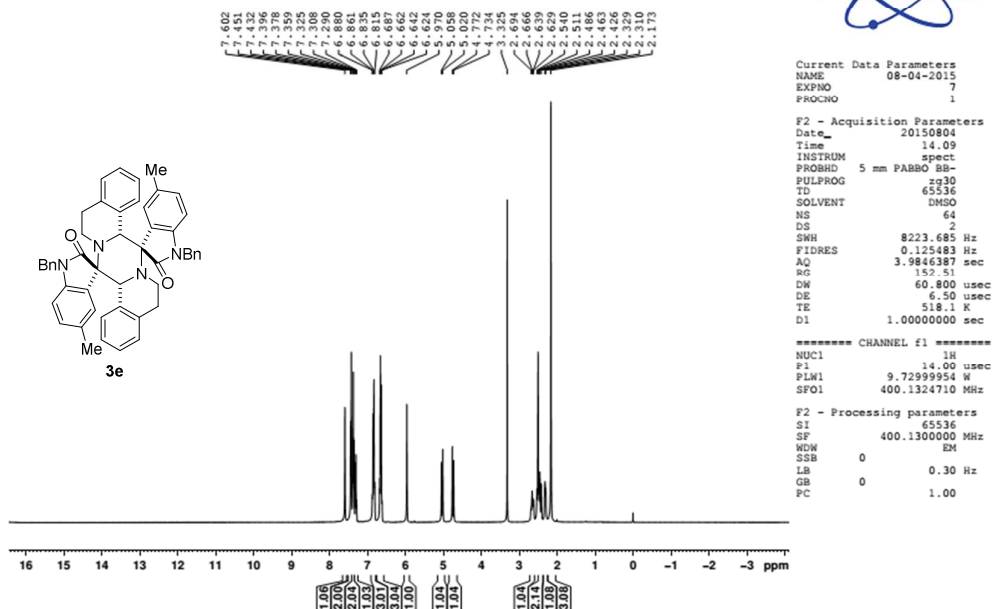
Chemical shifts (δ) listed at the top of the spectrum (from left to right): 7.071, 7.051, 7.030, 7.010, 6.990, 6.970, 6.951, 6.931, 6.910, 6.890, 6.870, 6.851, 6.831, 6.810, 6.790, 6.770, 6.751, 6.731, 6.710, 6.690, 6.670, 6.651, 6.631, 6.610, 6.590, 6.570, 6.551, 6.531, 6.510, 6.490, 6.470, 6.451, 6.431, 6.410, 6.390, 6.370, 6.351, 6.331, 6.310, 6.290, 6.270, 6.251, 6.231, 6.210, 6.190, 6.170, 6.151, 6.131, 6.110, 6.090, 6.070, 6.051, 6.031, 6.010, 5.990, 5.970, 5.951, 5.931, 5.910, 5.890, 5.870, 5.851, 5.831, 5.810, 5.790, 5.770, 5.751, 5.731, 5.710, 5.690, 5.670, 5.651, 5.631, 5.610, 5.590, 5.570, 5.551, 5.531, 5.510, 5.490, 5.470, 5.451, 5.431, 5.410, 5.390, 5.370, 5.351, 5.331, 5.310, 5.290, 5.270, 5.251, 5.231, 5.210, 5.190, 5.170, 5.151, 5.131, 5.110, 5.090, 5.070, 5.051, 5.031, 5.010, 4.990, 4.970, 4.951, 4.931, 4.910, 4.890, 4.870, 4.851, 4.831, 4.810, 4.790, 4.770, 4.751, 4.731, 4.710, 4.690, 4.670, 4.651, 4.631, 4.610, 4.590, 4.570, 4.551, 4.531, 4.510, 4.490, 4.470, 4.451, 4.431, 4.410, 4.390, 4.370, 4.351, 4.331, 4.310, 4.290, 4.270, 4.251, 4.231, 4.210, 4.190, 4.170, 4.151, 4.131, 4.110, 4.090, 4.070, 4.051, 4.031, 4.010, 3.990, 3.970, 3.951, 3.931, 3.910, 3.890, 3.870, 3.851, 3.831, 3.810, 3.790, 3.770, 3.751, 3.731, 3.710, 3.690, 3.670, 3.651, 3.631, 3.610, 3.590, 3.570, 3.551, 3.531, 3.510, 3.490, 3.470, 3.451, 3.431, 3.410, 3.390, 3.370, 3.351, 3.331, 3.310, 3.290, 3.270, 3.251, 3.231, 3.210, 3.190, 3.170, 3.151, 3.131, 3.110, 3.090, 3.070, 3.051, 3.031, 3.010, 2.990, 2.970, 2.951, 2.931, 2.910, 2.890, 2.870, 2.851, 2.831, 2.810, 2.790, 2.770, 2.751, 2.731, 2.710, 2.690, 2.670, 2.651, 2.631, 2.610, 2.590, 2.570, 2.551, 2.531, 2.510, 2.490, 2.470, 2.451, 2.431, 2.410, 2.390, 2.370, 2.351, 2.331, 2.310, 2.290, 2.270, 2.251, 2.231, 2.210, 2.190, 2.170, 2.151, 2.131, 2.110, 2.090, 2.070, 2.051, 2.031, 2.010, 1.990, 1.970, 1.951, 1.931, 1.910, 1.890, 1.870, 1.851, 1.831, 1.810, 1.790, 1.770, 1.751, 1.731, 1.710, 1.690, 1.670, 1.651, 1.631, 1.610, 1.590, 1.570, 1.551, 1.531, 1.510, 1.490, 1.470, 1.451, 1.431, 1.410, 1.390, 1.370, 1.351, 1.331, 1.310, 1.290, 1.270, 1.251, 1.231, 1.210, 1.190, 1.170, 1.151, 1.131, 1.110, 1.090, 1.070, 1.051, 1.031, 1.010, 0.990, 0.970, 0.951, 0.931, 0.910, 0.890, 0.870, 0.851, 0.831, 0.810, 0.790, 0.770, 0.751, 0.731, 0.710, 0.690, 0.670, 0.651, 0.631, 0.610, 0.590, 0.570, 0.551, 0.531, 0.510, 0.490, 0.470, 0.451, 0.431, 0.410, 0.390, 0.370, 0.351, 0.331, 0.310, 0.290, 0.270, 0.251, 0.231, 0.210, 0.190, 0.170, 0.151, 0.131, 0.110, 0.090, 0.070, 0.051, 0.031, 0.010, 0.000.

Integrations (from left to right): 1.00, 0.98, 4.11, 4.08, 4.05, 4.02, 4.00, 3.98, 3.95, 3.92, 3.90, 3.88, 3.85, 3.82, 3.80, 3.78, 3.75, 3.72, 3.70, 3.68, 3.65, 3.62, 3.60, 3.58, 3.55, 3.52, 3.50, 3.48, 3.45, 3.42, 3.40, 3.38, 3.35, 3.32, 3.30, 3.28, 3.25, 3.22, 3.20, 3.18, 3.15, 3.12, 3.10, 3.08, 3.05, 3.02, 3.00, 2.98, 2.95, 2.92, 2.90, 2.88, 2.85, 2.82, 2.80, 2.78, 2.75, 2.72, 2.70, 2.68, 2.65, 2.62, 2.60, 2.58, 2.55, 2.52, 2.50, 2.48, 2.45, 2.42, 2.40, 2.38, 2.35, 2.32, 2.30, 2.28, 2.25, 2.22, 2.20, 2.18, 2.15, 2.12, 2.10, 2.08, 2.05, 2.02, 2.00, 1.98, 1.95, 1.92, 1.90, 1.88, 1.85, 1.82, 1.80, 1.78, 1.75, 1.72, 1.70, 1.68, 1.65, 1.62, 1.60, 1.58, 1.55, 1.52, 1.50, 1.48, 1.45, 1.42, 1.40, 1.38, 1.35, 1.32, 1.30, 1.28, 1.25, 1.22, 1.20, 1.18, 1.15, 1.12, 1.10, 1.08, 1.05, 1.02, 1.00, 0.98, 0.95, 0.92, 0.90, 0.88, 0.85, 0.82, 0.80, 0.78, 0.75, 0.72, 0.70, 0.68, 0.65, 0.62, 0.60, 0.58, 0.55, 0.52, 0.50, 0.48, 0.45, 0.42, 0.40, 0.38, 0.35, 0.32, 0.30, 0.28, 0.25, 0.22, 0.20, 0.18, 0.15, 0.12, 0.10, 0.08, 0.05, 0.02, 0.00.

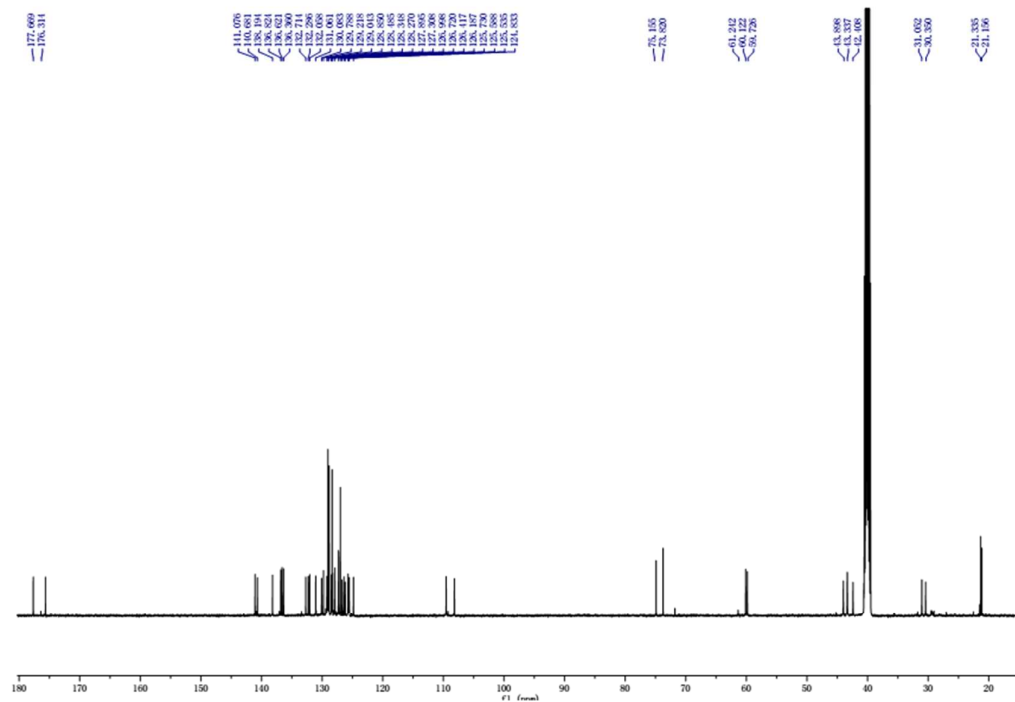
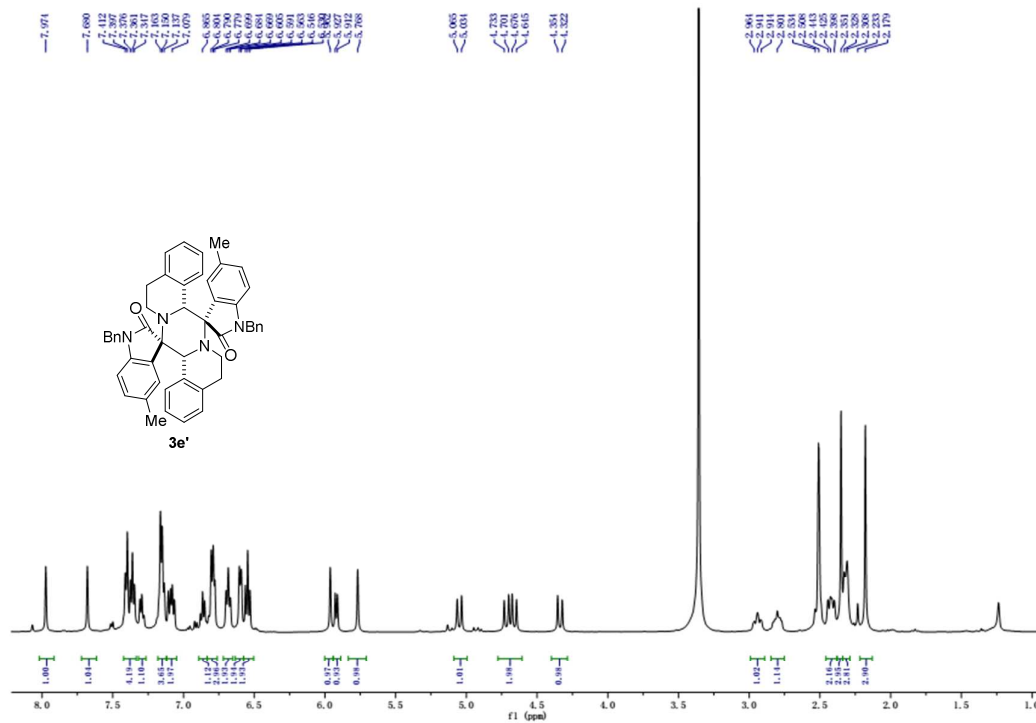


^1H NMR (400 MHz; $\text{DMSO}-d_6$), ^{13}C NMR (100 MHz; $\text{DMSO}-d_6$)

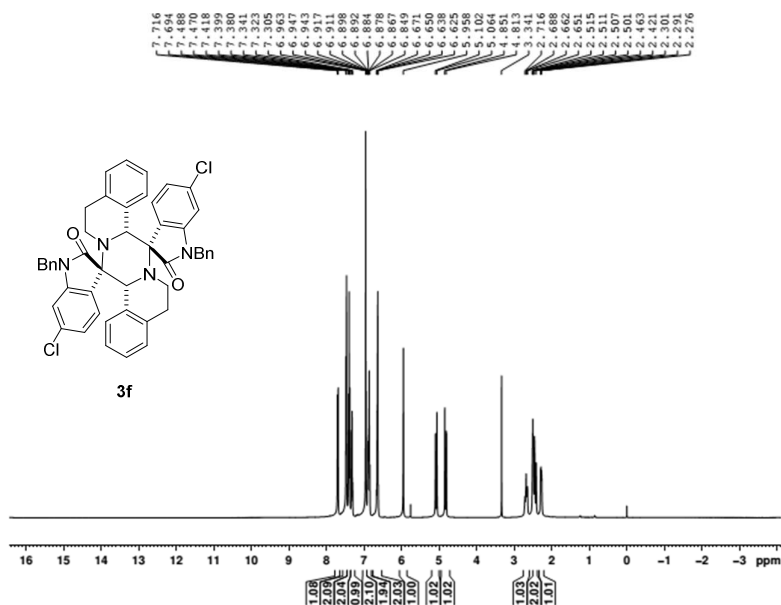
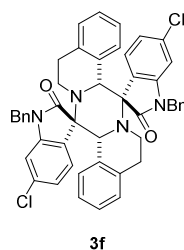
XP-22



^1H NMR (500 MHz; $\text{DMSO-}d_6$), ^{13}C NMR (100 MHz; $\text{DMSO-}d_6$)



XP-31



```

Current Data Parameters
Name          07-30-2015
EXPNO         8
PROCNO        1

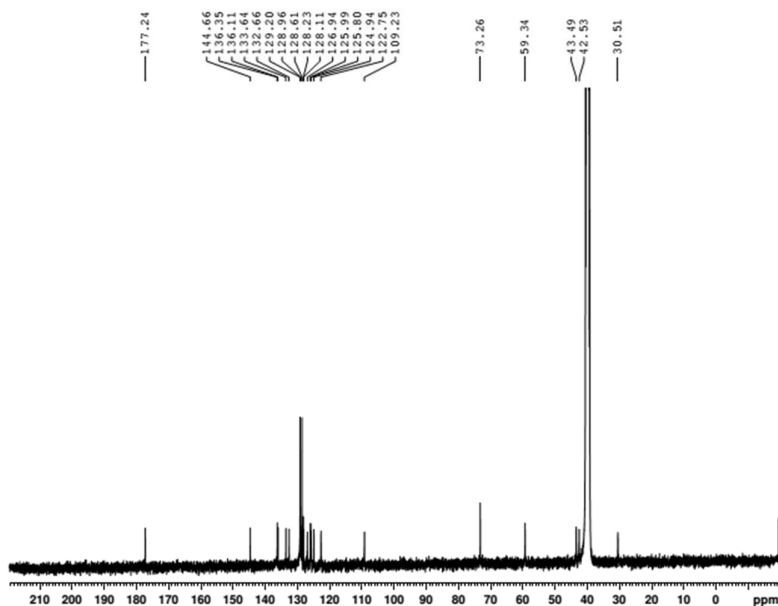
F2 - Acquisition Parameters
Date_         20150730
Time          12.26
INSTRUM       spect
PROBHD        5 mm PABBO BB-
PULPROG       zg30
TD            65536
SOLVENT       DMSO
NS            76
DS            2
SW            8223.65 Hz
FIDRES        0.125483 Hz
AQ            3.9846387 sec
RG            64.96
DE            60.800 usec
DW            6.50 usec
TE            518.1 K
D1            1.00000000 sec

===== CHANNEL f1 =====
NUC1          13C          14.00 usec
PLW1          9.72999954 W
P1           400.1324710 MHz

F2 - Processing parameters
S1            51536
SF            400.1300000 MHz
RG            EM
SSB           0
PC            0          0.30 Hz
LB           0          1.00

```

XP-31



```

Current Data Parameters
NAME          07-30-2015
EXPNO         16
PROCNO        1

F2 - Acquisition Parameters
Date_         20150805
Time          8.01
INSTRUM       spect
PROBHD        5 mm PABBO BB-
PULPROG       zgpg30
TD            65536
SOLVENT       DMSO
NS            15600
DS            4
SWH            24038.461 Hz
FIDRES        0.366798 Hz
AQ            1.3631988 sec
RG            198.33
CW            20.800 usec
DE            6.50 usec
TE            318.1 K
D11           2.00000000 sec
D12           0.03000000 sec

***** CHANNEL f1 *****
NUC1          F1          13C
P1            9.00 usec
PLW1          10.9999847 W
SFO1          100.6228297 MHz

***** CHANNEL f2 *****
CPDPRG2       waltz16
NUC2           1H
PCPD2         90.00 usec
P12           9.36999989 W
PLW12         0.2267300 W
PLW13         0.1836500 W
SFO2          400.1316005 MHz

F2 - Processing parameters
SI            32768
SF            100.6127690 MHz
WDW           EM
SSB           0
GB            0          1.00 Hz
PC            1.40

```

XP-30

Chemical structure of **3f** is shown, a complex polycyclic molecule with two chlorine atoms and two N-benzyl groups.

1H NMR Data (DMSO-d₆):

Chemical Shift (ppm)	Integration
8.150	0.139
7.931	0.111
7.741	0.121
7.541	0.102
7.384	0.065
7.241	0.114
7.196	0.128
7.082	0.111
6.992	0.092
6.899	0.089
6.765	0.065
6.665	0.065
6.532	0.032
6.514	0.034
6.496	0.034
6.444	0.034
6.326	0.036
6.295	0.036
6.161	0.036
6.000	0.000
5.773	0.036
5.557	0.036
4.734	0.036
4.566	0.036
4.332	0.036
4.302	0.036
4.345	0.036
4.261	0.036
4.166	0.036
4.112	0.036
4.095	0.036
3.922	0.036
3.345	0.036
3.261	0.036
3.166	0.036
3.112	0.036
3.095	0.036
2.922	0.036
2.445	0.036
2.410	0.036
2.395	0.036
2.297	0.036
2.275	0.036
2.246	0.036
2.161	0.036
2.112	0.036
2.095	0.036
2.036	0.036
2.018	0.036

Current Data Parameters

NAME	08-12-2015
EXPNO	4
PROCNO	1

F2 - Acquisition Parameters

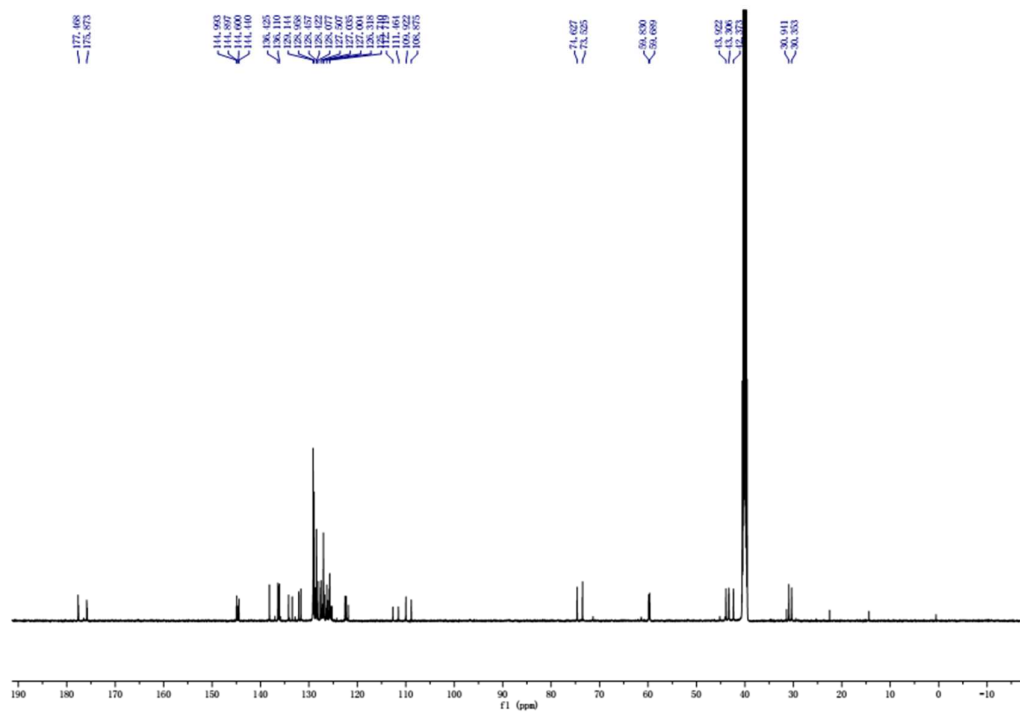
Date_	20150812
Time	10.56
INSTRUM	spect
PROBHD	5 mm PABBO BB-
PULPROG	zg30
TD	65536
SOLVENT	DMSO
NS	116
DS	2
SWH	8223.685 Hz
FIDRES	0.125483 Hz
AQ	3.9846387 sec
RG	124.55
DW	60.800 usec
DE	6.50 usec
TE	518.1 K
D1	1.00000000 sec

===== CHANNEL f1 =====

NUC1	1H
P1	14.00 usec
PLW1	9.72999954 W
SFO1	400.1324710 MHz

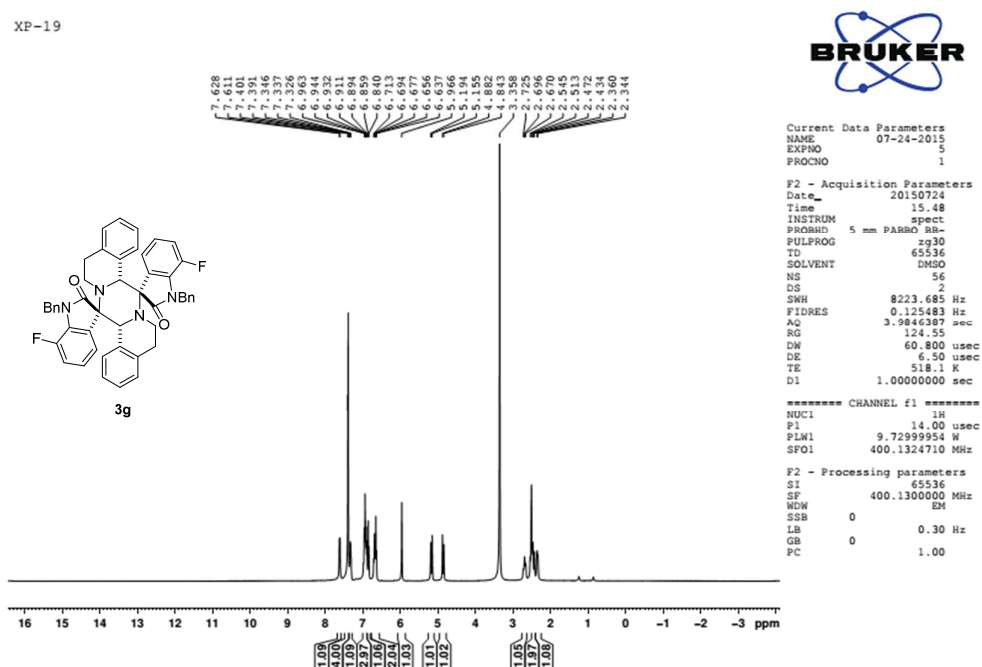
F2 - Processing parameters

SI	65536
SF	400.1300000 MHz
WDW	EM
SF	
LB	0.30 Hz
GB	0
PC	1.00

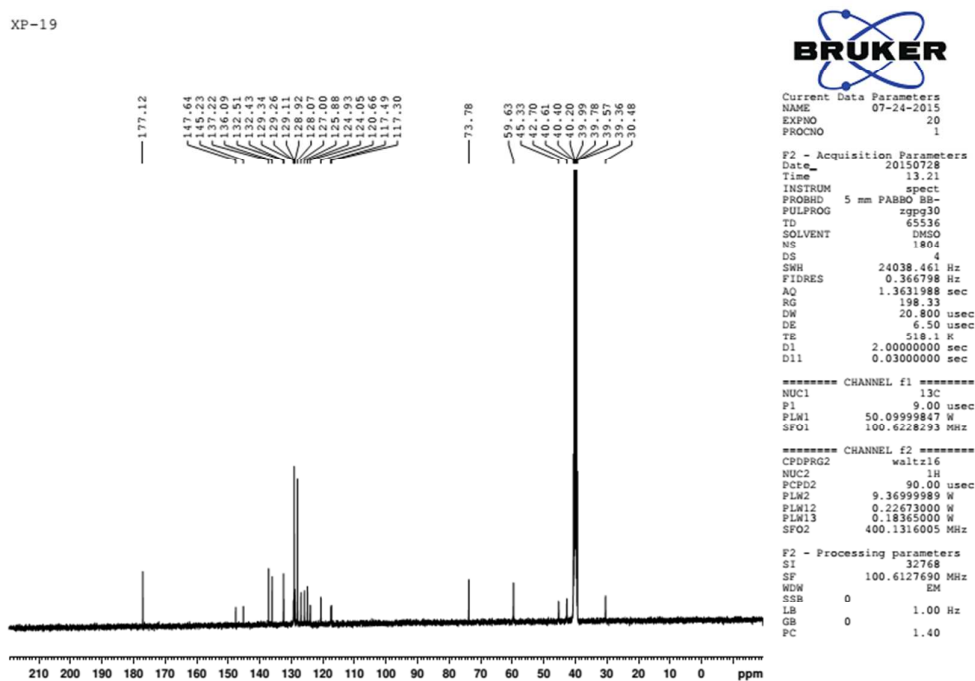


^1H NMR (400 MHz; $\text{DMSO-}d_6$), ^{13}C NMR (100 MHz; $\text{DMSO-}d_6$)

XP-19

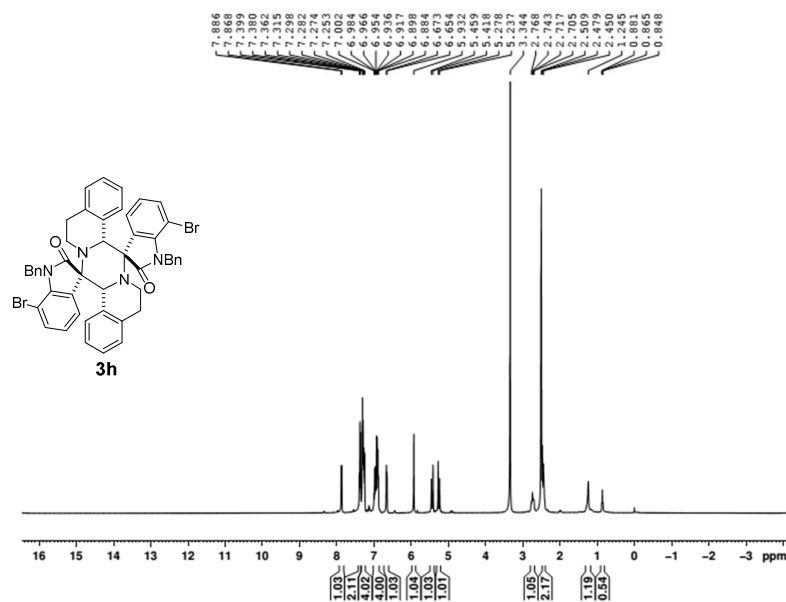


XP-19



^1H NMR (400 MHz; $\text{DMSO-}d_6$), ^{13}C NMR (100 MHz; $\text{DMSO-}d_6$)

XP-61



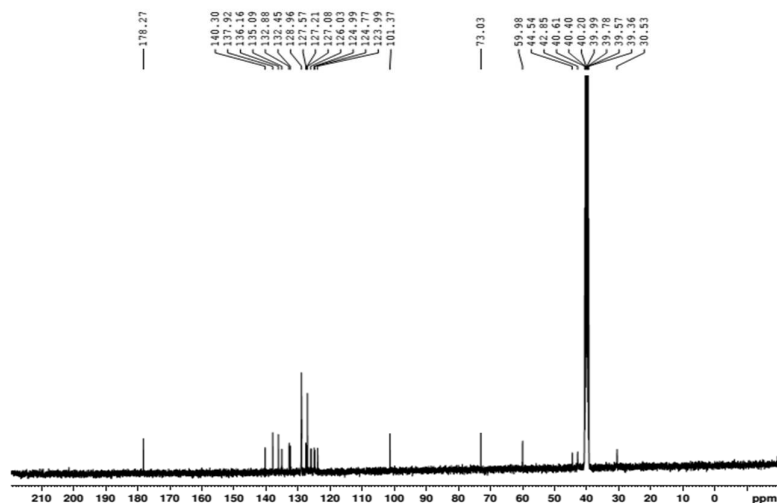
Current Data Parameters
NAME 10-19-2015
EXPNO 1
PROCNO 1

F2 - Acquisition Parameters
Date_ 20151019
Time 17.44
INSTRUM spect
PROBHD 5 mm PABBO BB-
PULPROG zgpg30
TD 65536
SOLVENT DMSO
NS 2
DS 2
SWH 8223.685 Hz
FIDRES 0.125483 Hz
AQ 3.9846387 sec
RG 198.33
DW 60.800 usec
DE 6.50 usec
TE 295.8 K
D1 1.00000000 sec

----- CHANNEL f1 -----
NUC1 ^1H
P1 14.00 usec
PLW1 9.72999954 W
SFO1 400.1324710 MHz

F2 - Processing parameters
SI 65536
SF 400.1300000 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00

XP-61



Current Data Parameters
NAME 10-19-2015
EXPNO 2
PROCNO 1

F2 - Acquisition Parameters
Date_ 20151020
Time 11.12
INSTRUM spect
PROBHD 5 mm PABBO BB-
PULPROG zgpg30
TD 65536
SOLVENT DMSO
NS 4
DS 4
SWH 24038.461 Hz
FIDRES 0.366798 Hz
AQ 1.3631988 sec
RG 198.33
DW 20.800 usec
DE 6.50 usec
TE 296.3 K
D1 2.00000000 sec
D11 0.03000000 sec

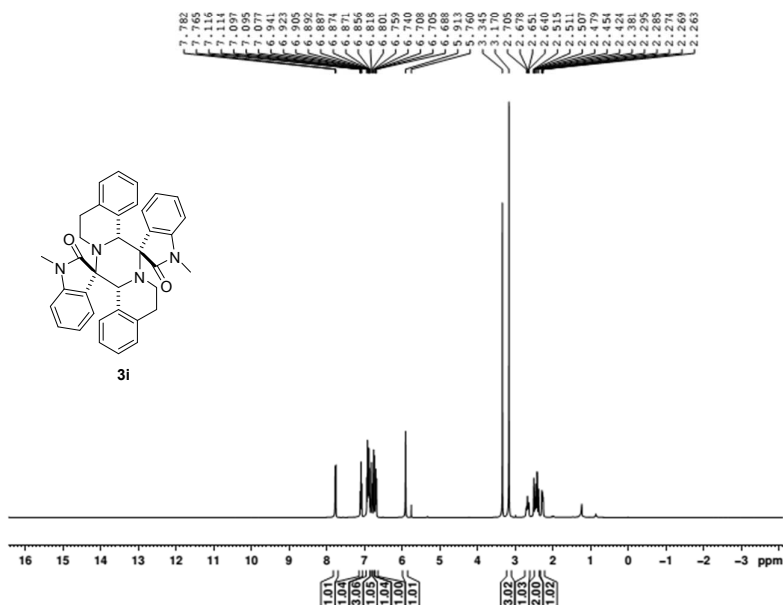
----- CHANNEL f1 -----
NUC1 ^{13}C
P1 9.00 usec
PLW1 50.09999847 W
SFO1 100.6228293 MHz

----- CHANNEL f2 -----
CPDPRG2 waltz16
NUC2 ^1H
PCPD2 90.00 usec
PLW2 9.36999989 W
PLW12 0.22673000 W
PLW13 0.18365000 W
SFO2 400.1316005 MHz

F2 - Processing parameters
SI 32768
SF 100.6127690 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40

^1H NMR (400 MHz; $\text{DMSO-}d_6$), ^{13}C NMR (100 MHz; $\text{DMSO-}d_6$)

XP-27



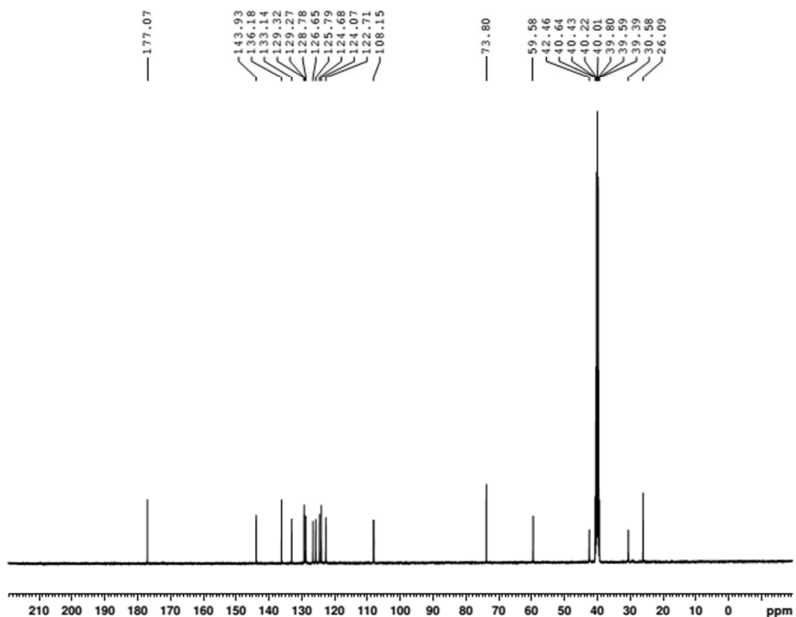
Current Data Parameters
NAME 08-12-2015
EXPNO 3
PROCNO 1

F2 - Acquisition Parameters
Date_ 20150812
Time 10.28
INSTRUM spect
PROBHD 5 mm PABBO BB-
PULPROG zg30
TD 65536
SOLVENT DMSO
NS 128
DS 2
SWH 8223.685 Hz
FIDRES 0.125483 Hz
AQ 3.9846387 sec
RG 106.96
DW 60.800 usec
DE 6.50 usec
TE 518.1 K
D1 1.00000000 sec

***** CHANNEL f1 *****
NUC1 ^1H
P1 14.00 usec
PLW1 9.72999954 W
SFO1 400.1324710 MHz

F2 - Processing parameters
SI 65536
SF 400.1300000 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00

XP-27



Current Data Parameters
NAME 08-12-2015
EXPNO 9
PROCNO 1

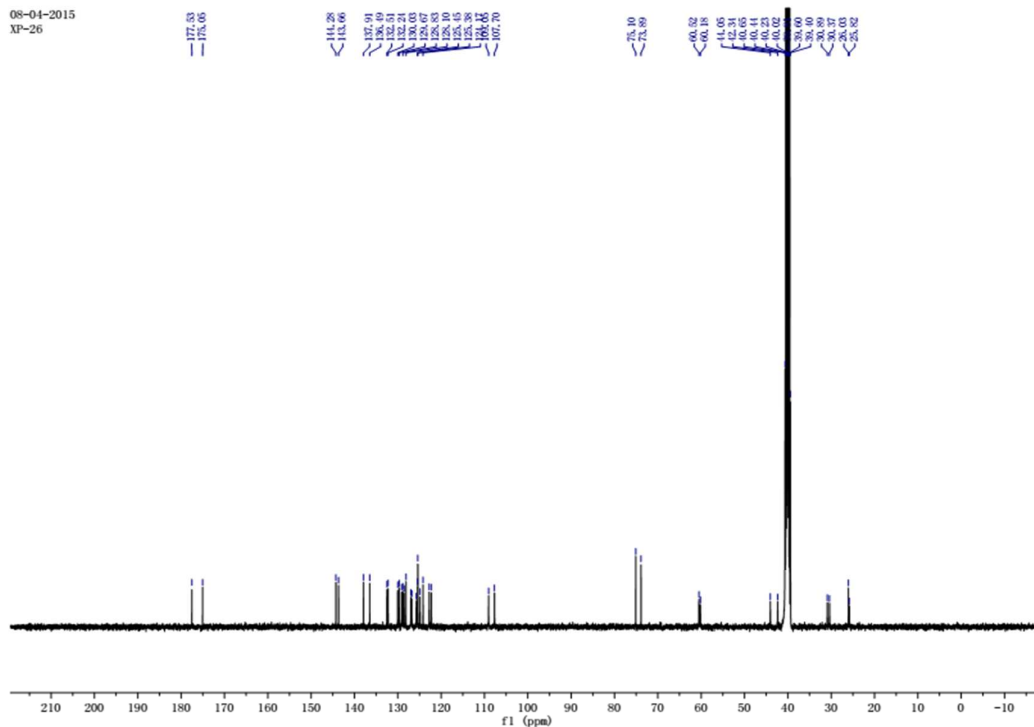
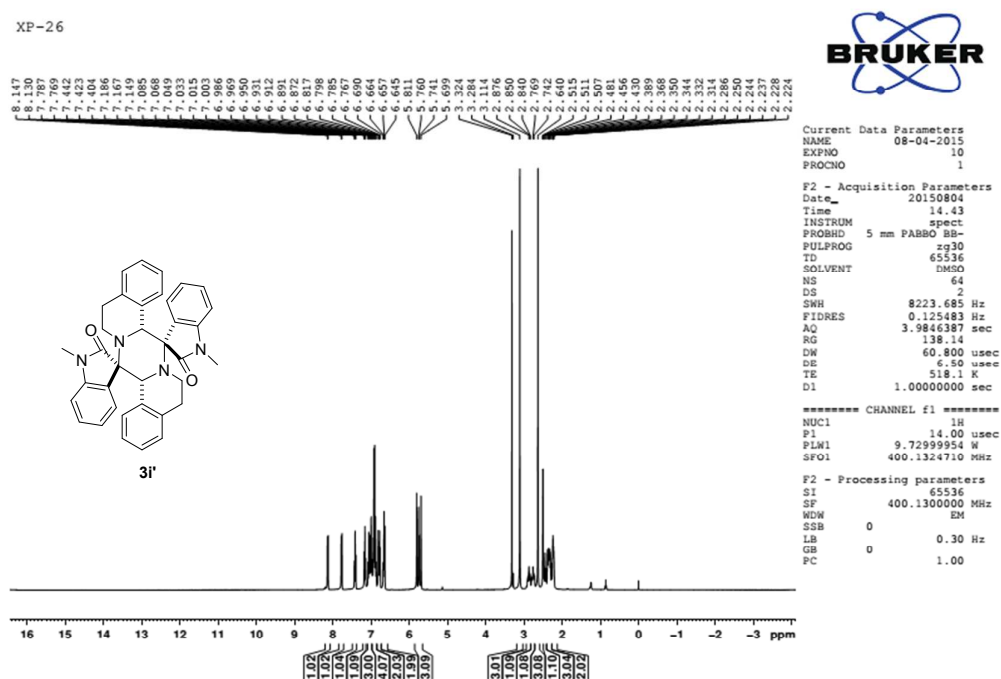
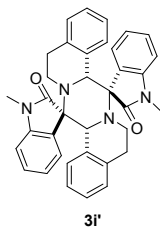
F2 - Acquisition Parameters
Date_ 20150817
Time 15.12
INSTRUM spect
PROBHD 5 mm PABBO BB-
PULPROG zgpg30
TD 65536
SOLVENT DMSO
NS 1892
DS 4
SWH 24038.461 Hz
FIDRES 0.366798 Hz
AQ 1.3631988 sec
RG 198.33
DW 20.800 usec
DE 6.50 usec
TE 518.1 K
D1 2.00000000 sec
D11 0.03000000 sec

***** CHANNEL f1 *****
NUC1 ^{13}C
P1 9.00 usec
PLW1 50.09999847 W
SFO1 100.6228293 MHz

***** CHANNEL f2 *****
CPDPRG2 waltz16
NUC2 ^1H
PCPD2 90.00 usec
PLW2 9.36999989 W
PLW12 0.22673000 W
PLW13 0.18365000 W
SFO2 400.1316005 MHz

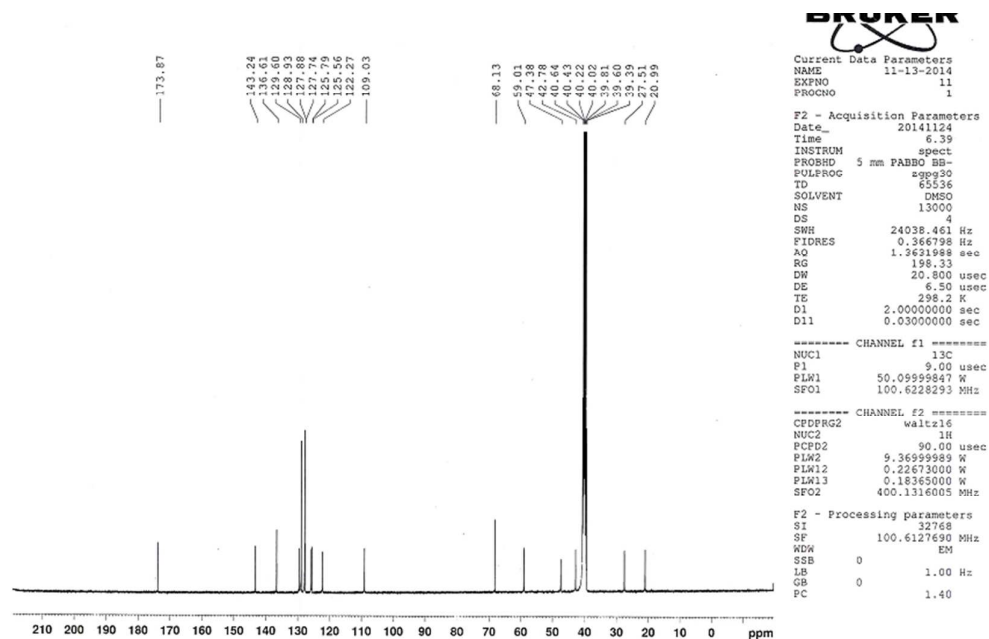
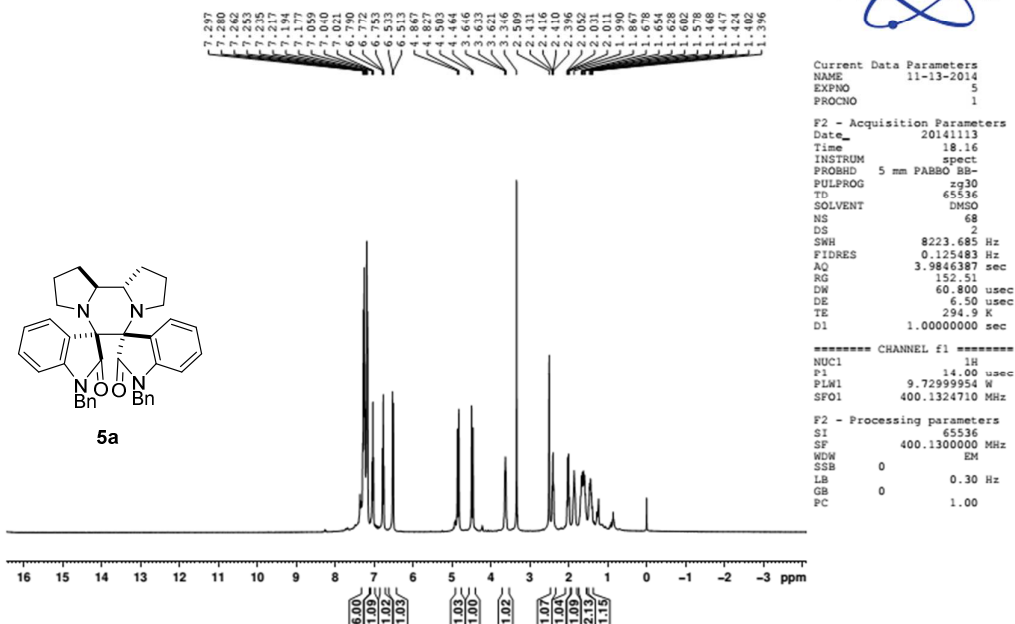
F2 - Processing parameters
SI 32768
SF 100.6127690 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40

XP-26



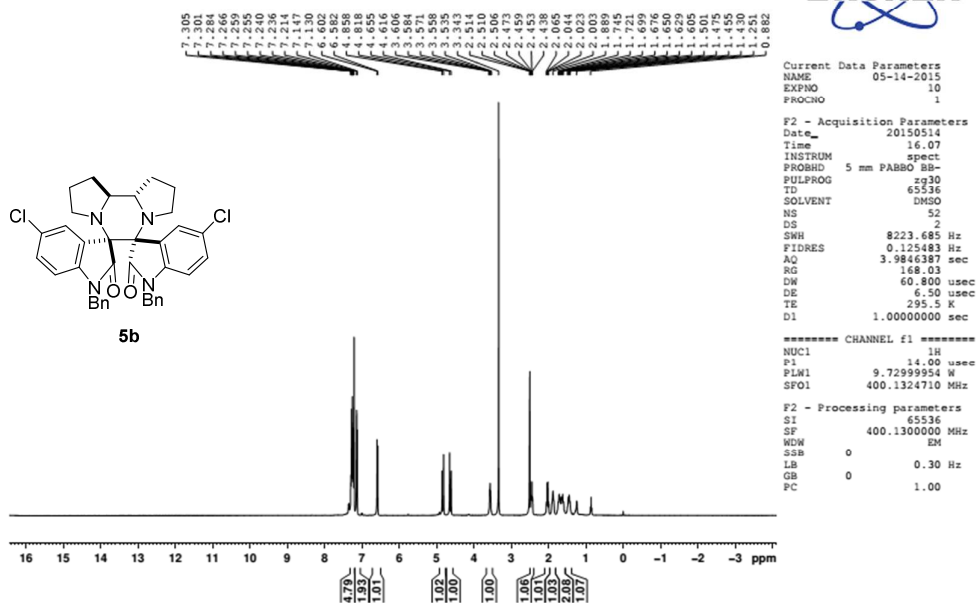
^1H NMR (400 MHz; $\text{DMSO-}d_6$), ^{13}C NMR (100 MHz; $\text{DMSO-}d_6$)

H76

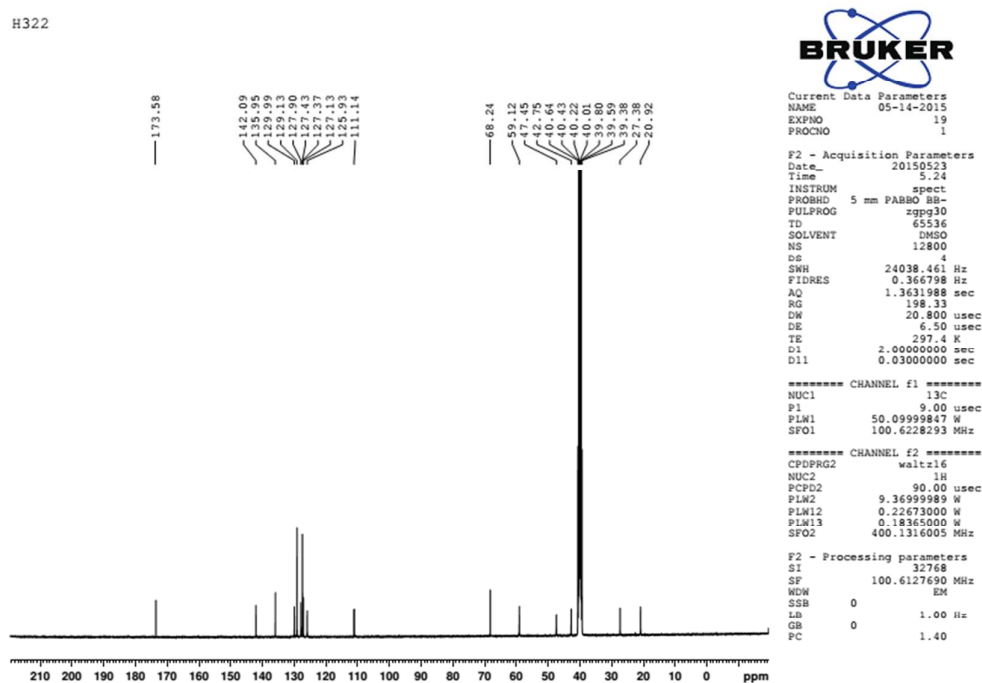


^1H NMR (400 MHz; DMSO- d_6), ^{13}C NMR (100 MHz; DMSO- d_6)

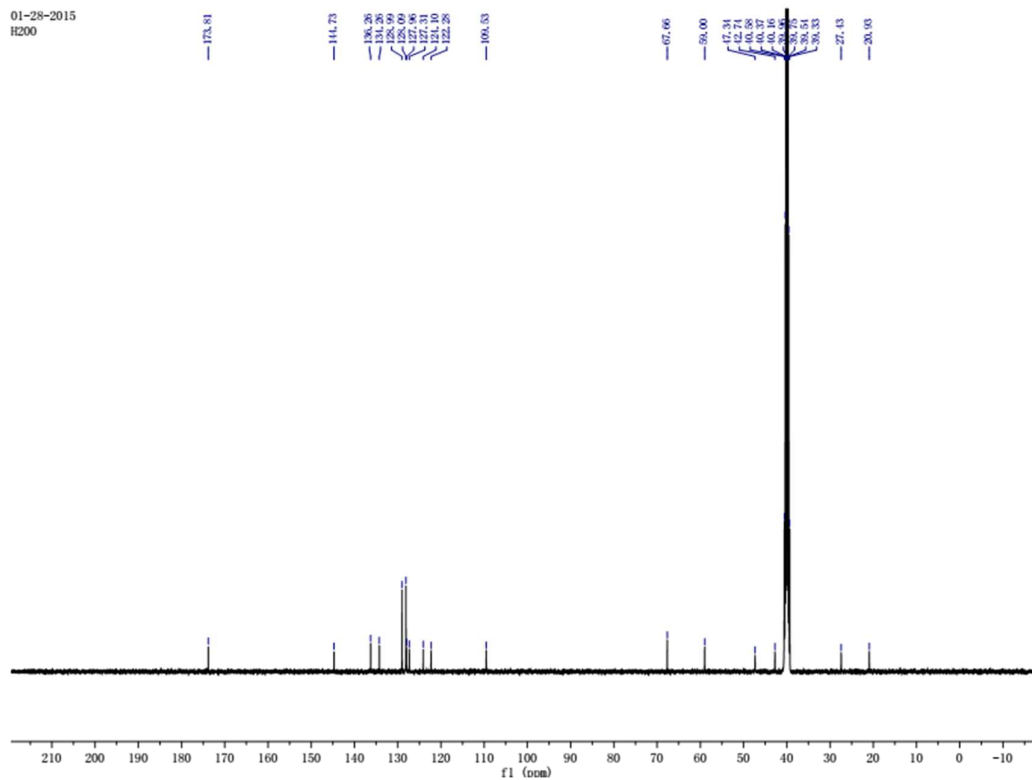
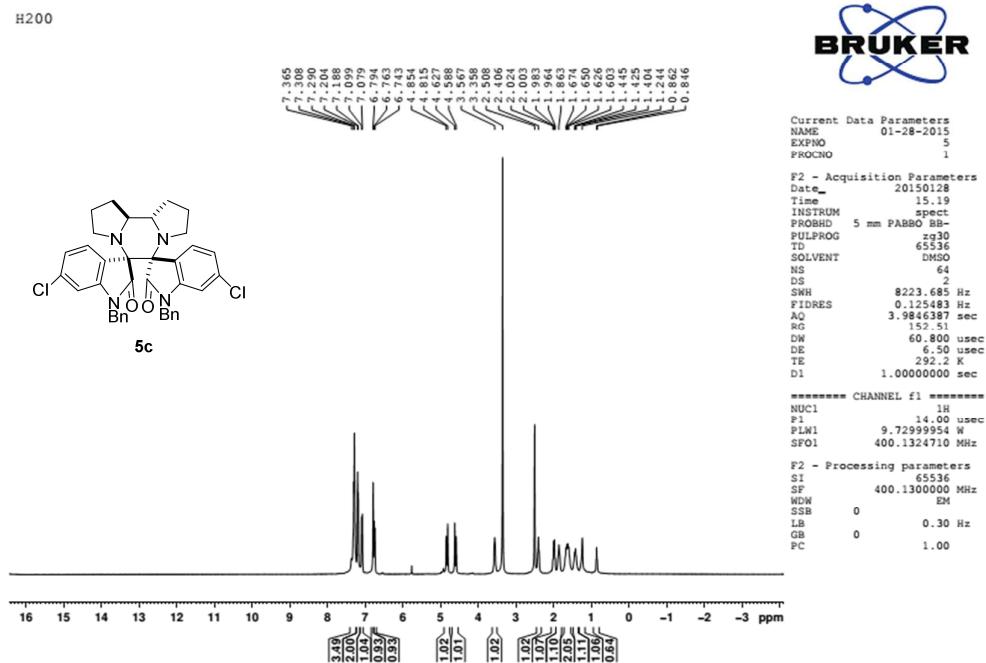
H322



H322

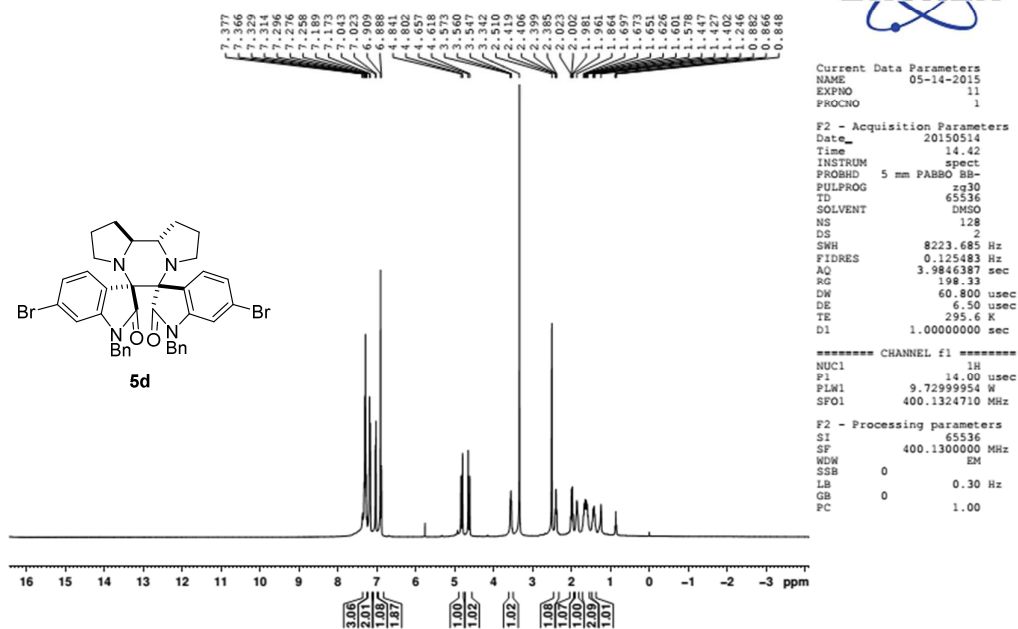


^1H NMR (400 MHz; $\text{DMSO-}d_6$), ^{13}C NMR (100 MHz; $\text{DMSO-}d_6$)

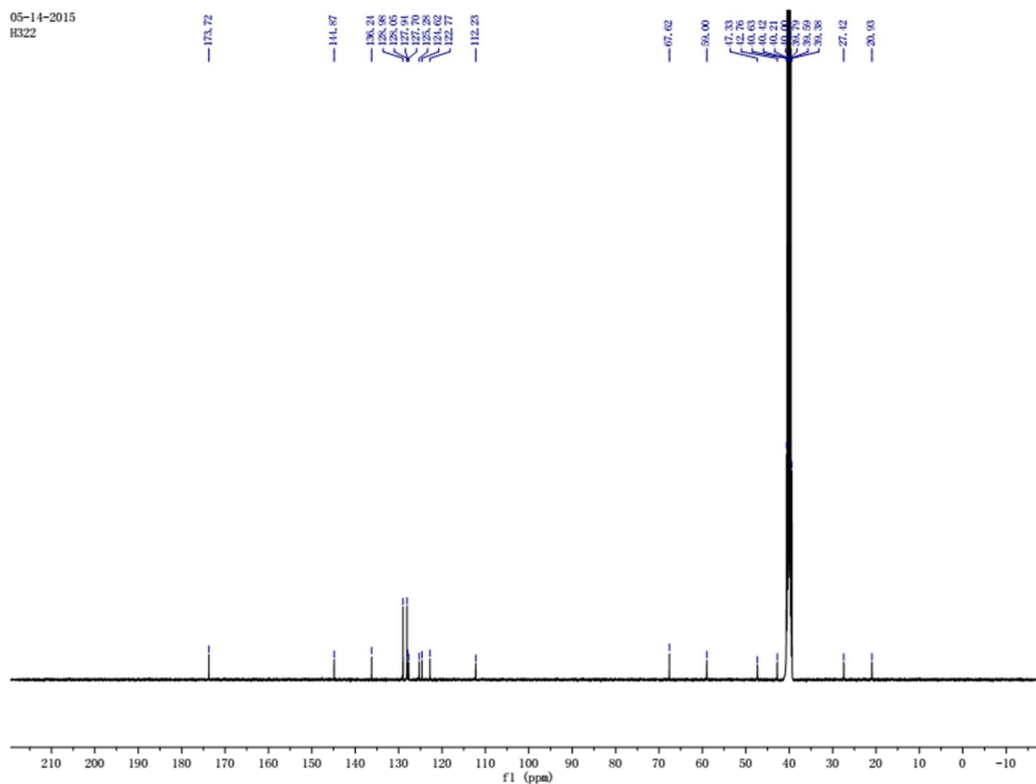


^1H NMR (400 MHz; $\text{DMSO-}d_6$), ^{13}C NMR (100 MHz; $\text{DMSO-}d_6$)

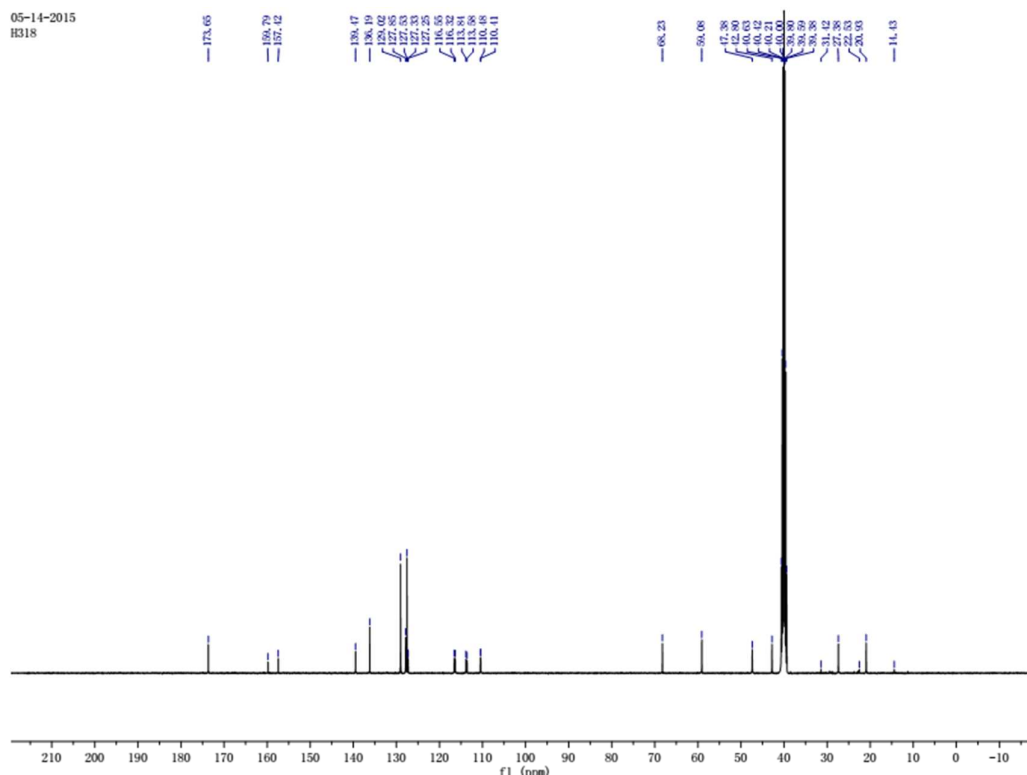
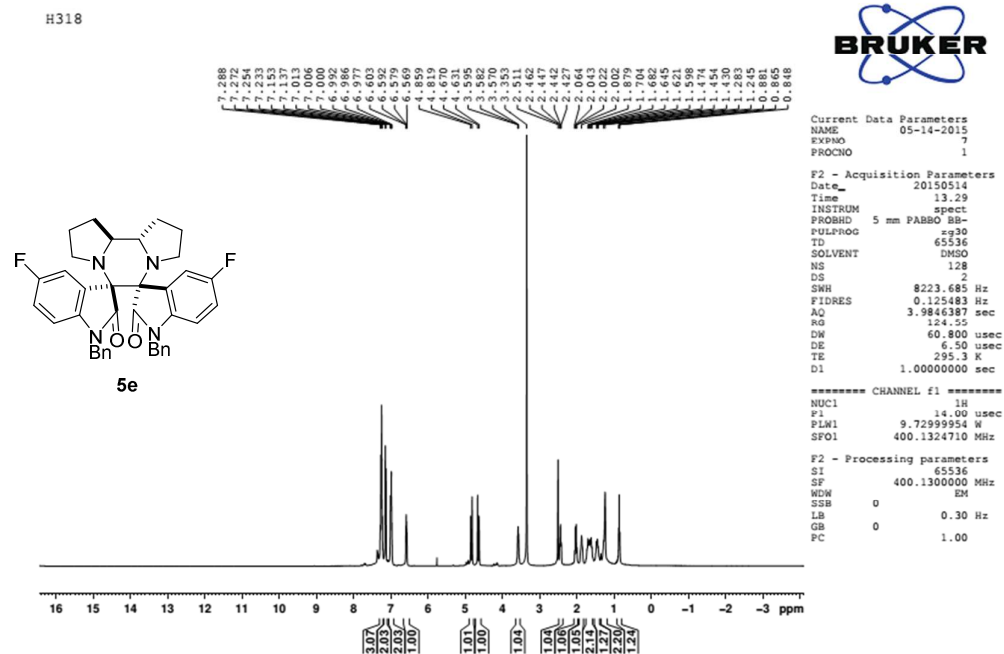
H323



05-14-2015
H322

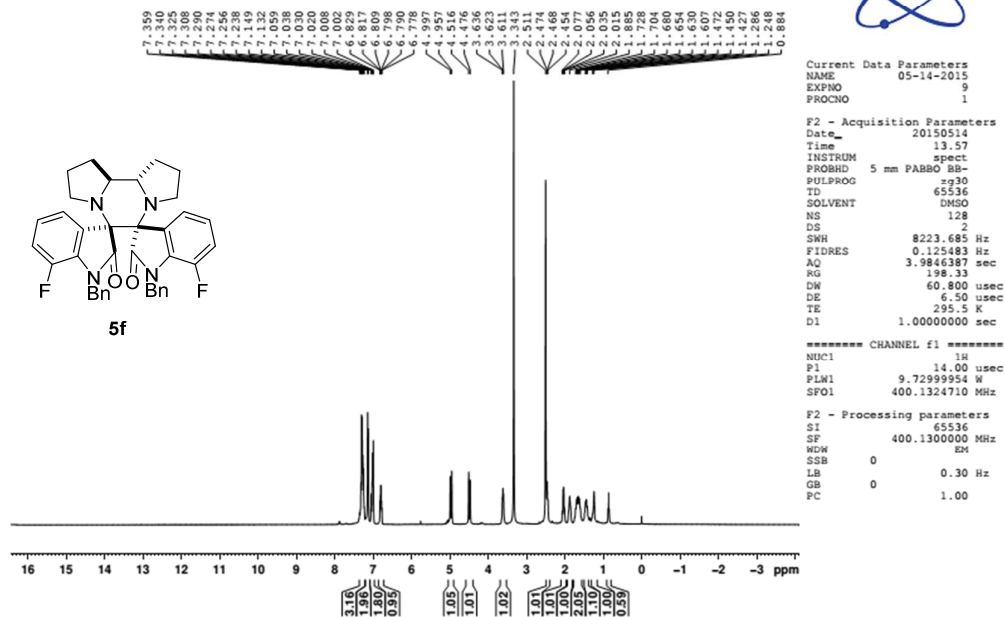


^1H NMR (400 MHz; $\text{DMSO}-d_6$), ^{13}C NMR (100 MHz; $\text{DMSO}-d_6$)



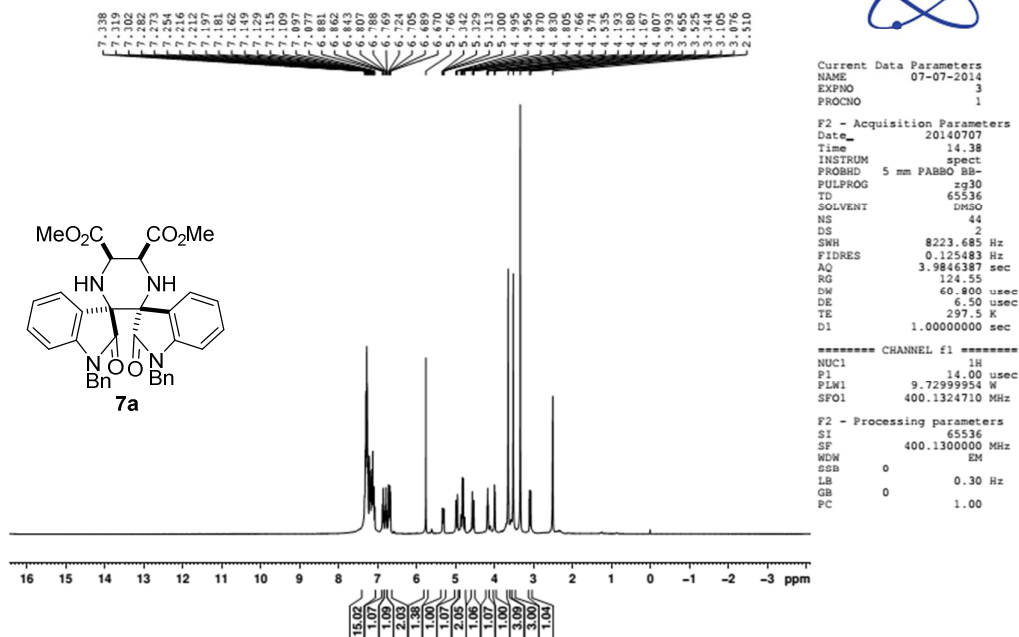
^1H NMR (400 MHz; $\text{DMSO}-d_6$), ^{13}C NMR (100 MHz; $\text{DMSO}-d_6$)

H321

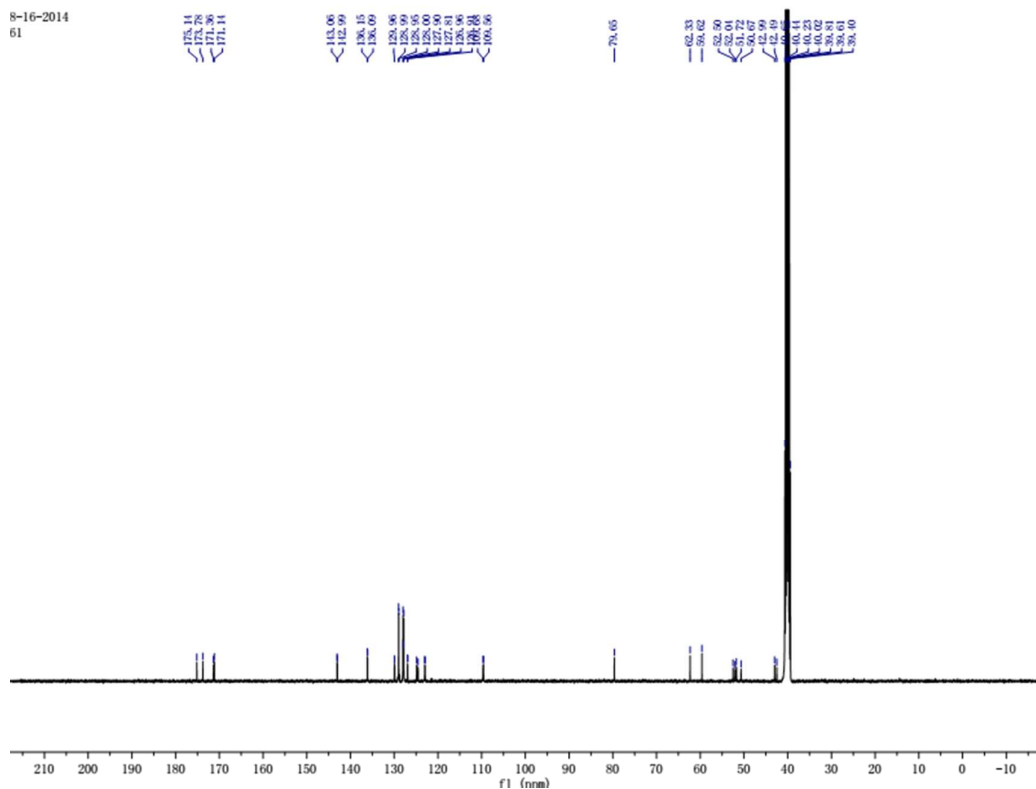


^1H NMR (400 MHz; $\text{DMSO}-d_6$), ^{13}C NMR (100 MHz; $\text{DMSO}-d_6$)

S61

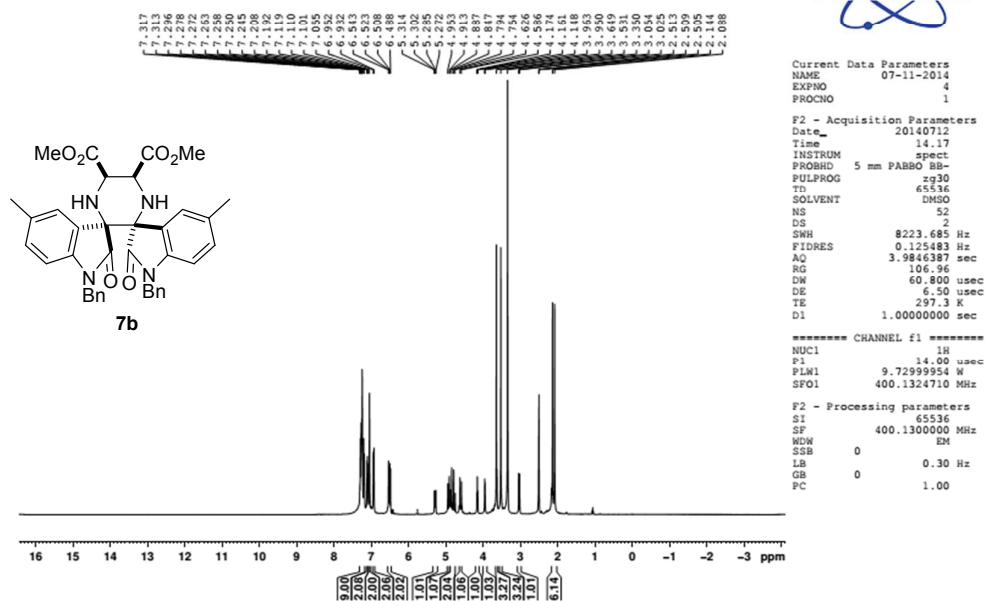


8-16-2014
61

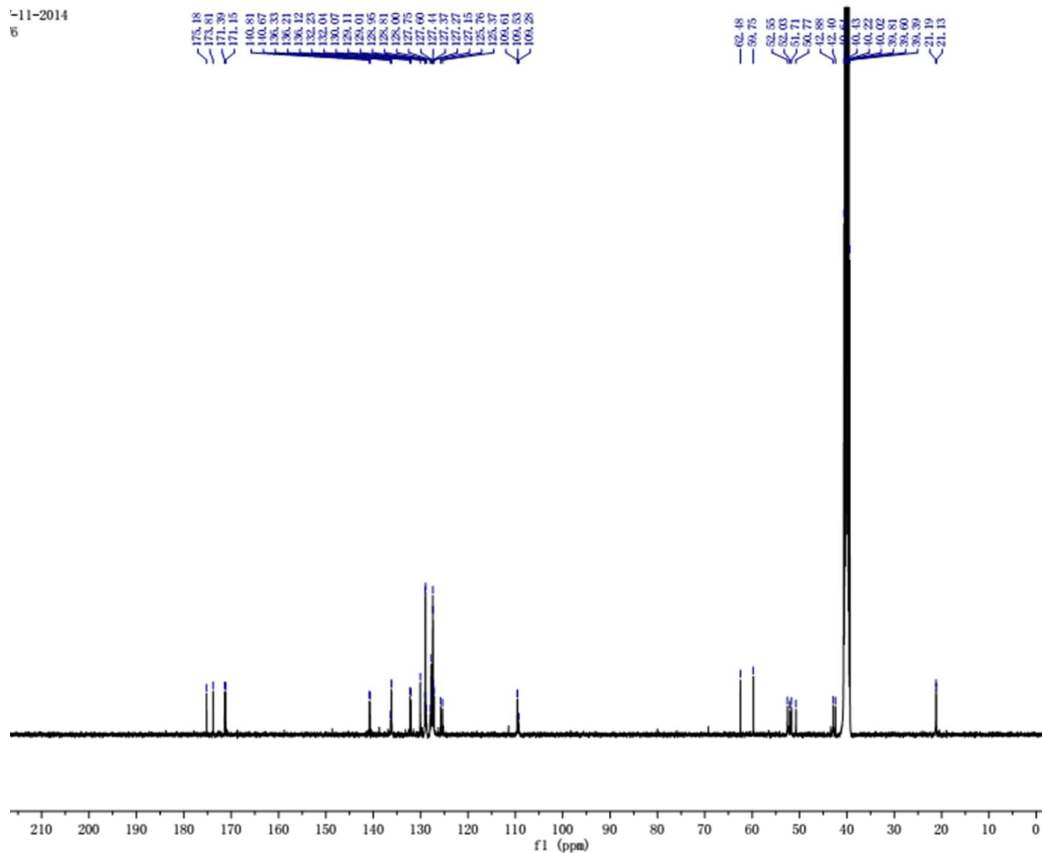


^1H NMR (400 MHz; DMSO- d_6), ^{13}C NMR (100 MHz; DMSO- d_6)

S76



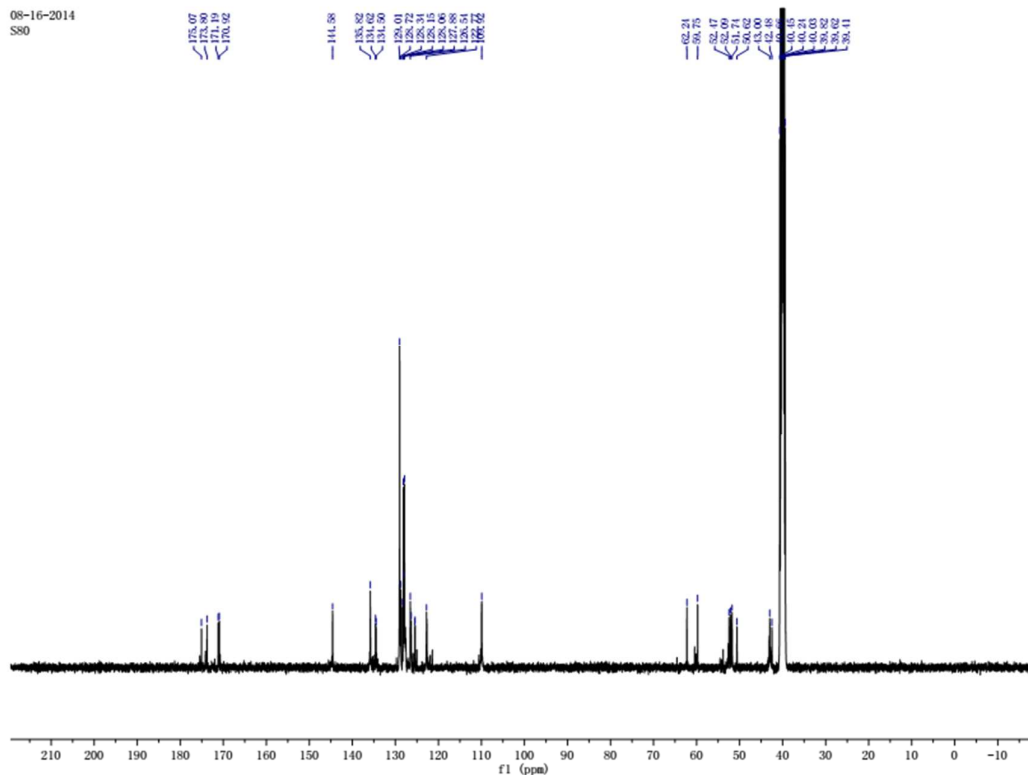
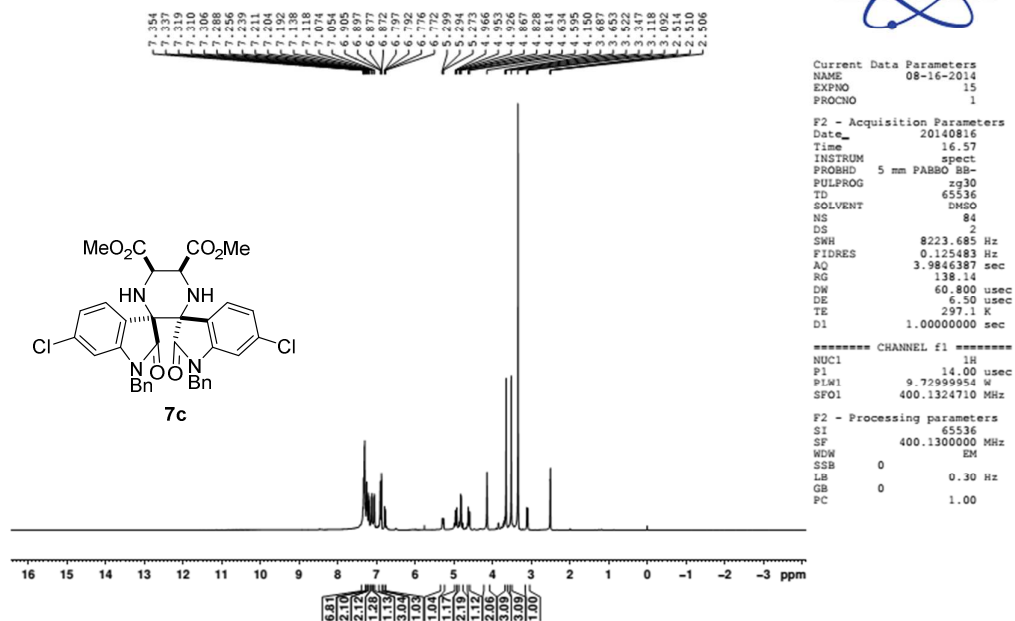
07-11-2014
6



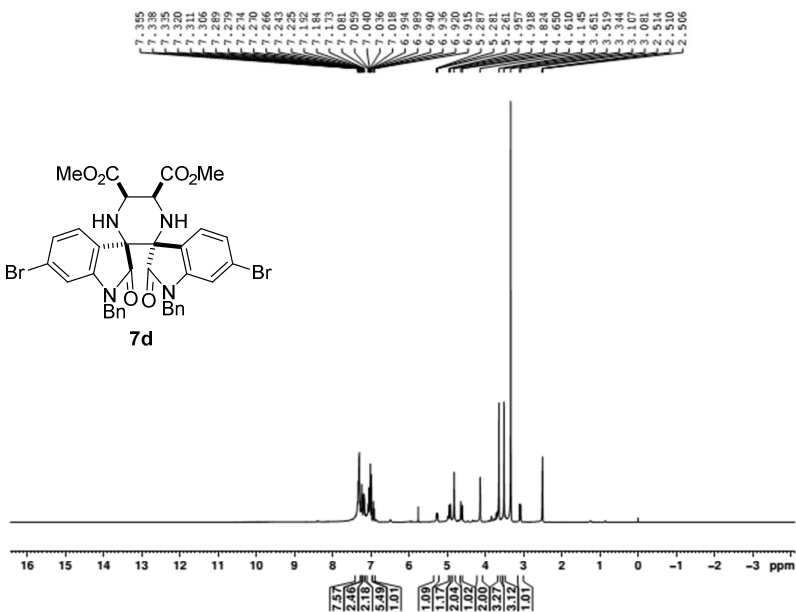
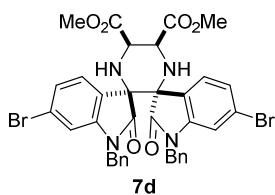
S26

^1H NMR (400 MHz; $\text{DMSO-}d_6$), ^{13}C NMR (100 MHz; $\text{DMSO-}d_6$)

S80



\$79



```

Current Data Parameters
NAME          08-16-2014
EXPNO        14
PROCNO       1

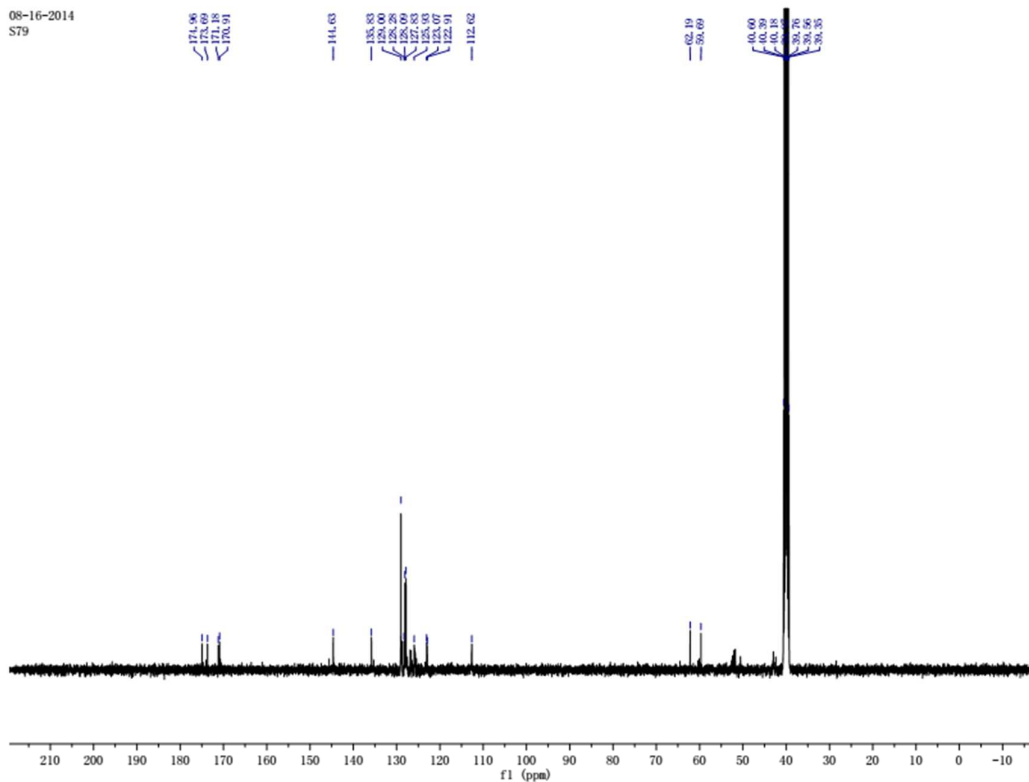
F2 - Acquisition Parameters
Date_         20140816
Time          16.44
INSTRUM       spect
PROBHD        5 mm PABBO BB-
PULPROG       zg30
TD            65536
SOLVENT       DMSO
DS            2
NS            2
SWH            8223.65 Hz
FIDRES        0.125483 Hz
AQ            3.9846377 sec
DE            152.51 Hz
CW            60.800 usec
DI            6.50 usec
TE            297.2 K
D1            1.00000000 sec

===== CHANNEL f1 =====
NUC1          13
P1            14.00 usec
PL1           9.72999954 uW
SFO1          400.1324710 MHz

F2 - Processing parameters
SI            65536
SF            400.1300000 MHz
RG            EM
PC            0
LB            0.30 Hz
FC            0
GB            1.00

```

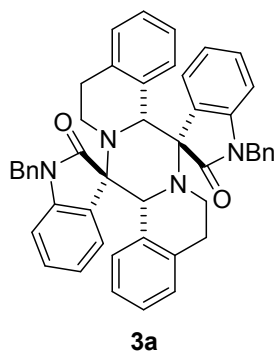
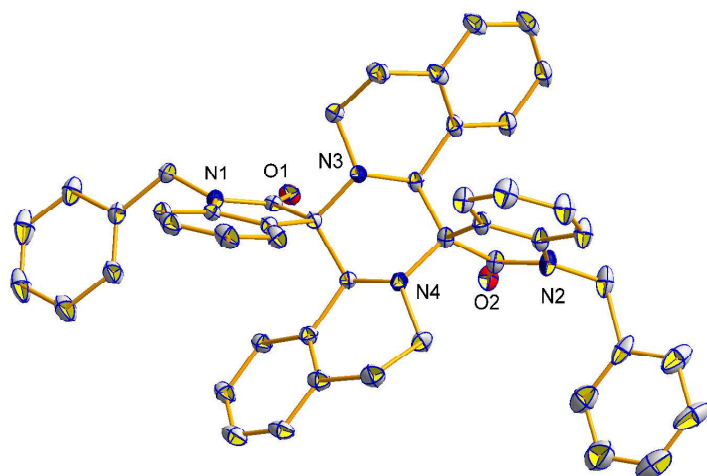
08-16-2014
S79



S28

Crystal structure of dispirooxindole-piperazine 3a

X-Ray crystallographic analysis of dispirooxindole-piperazine 3a (CCDC 1421682) showing the thermal ellipsoids at 30% probability level.



Bond precision: C-C = 0.0035 Å Wavelength=0.71073

Cell: a=9.2740(4) b=11.7097(4) c=20.2631(7)
 alpha=84.558(2) beta=85.736(2) gamma=69.051(2)
 Temperature: 295 K

	Calculated	Reported
Volume	2043.83(14)	2043.83(13)
Space group	P -1	P-1
Hall group	-P 1	?
Moiety formula	C48 H40 N4 O2	?
Sum formula	C48 H40 N4 O2	C48 H40 N4 O2
Mr	704.84	704.84
Dx, g cm ⁻³	1.145	1.145
Z	2	2
Mu (mm ⁻¹)	0.070	0.070
F000	744.0	744.0
F000'	744.28	
h,k,lmax	11,13,24	11,13,24
Nref	7189	7171
Tmin,Tmax	0.986,0.986	0.986,0.986
Tmin'	0.986	

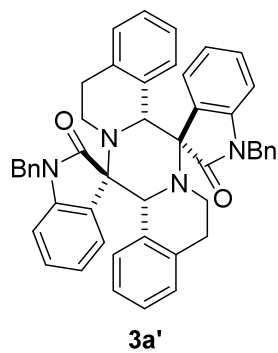
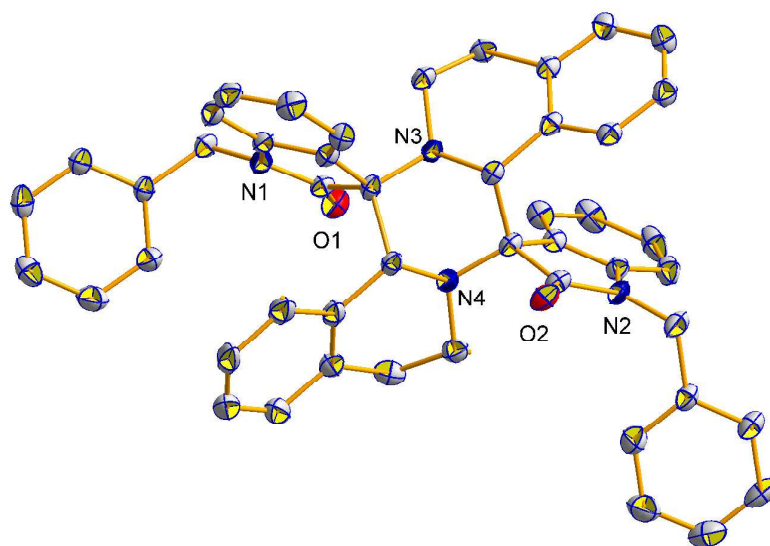
Correction method= # Reported T Limits: Tmin=0.986 Tmax=0.986
 AbsCorr = ?

Data completeness= 0.997 Theta(max)= 25.010

R(reflections)= 0.0491(4820) wR2(reflections)= 0.1595(7171)

S = 1.064 Npar= 518

X-Ray crystallographic analysis of dispirooxindole-piperazine 3a' (CCDC 1421681) showing the thermal ellipsoids at 30% probability level.



Bond precision: C-C = 0.0040 Å Wavelength=0.71073

Cell: a=12.4855(14) b=12.3203(14) c=29.040(3)
 alpha=90 beta=100.910(6) gamma=90
 Temperature: 296 K

	Calculated	Reported
Volume	4386.3(8)	4386.4(9)
Space group	P 21/c	P2(1)/c
Hall group	-P 2ybc	?
Moiety formula	C48 H40 N4 O2	?
Sum formula	C48 H40 N4 O2	C48 H40 Cl0 N4 O2
Mr	704.84	704.84
Dx, g cm ⁻³	1.067	1.067
Z	4	4
Mu (mm ⁻¹)	0.066	0.066
F000	1488.0	1488.0
F000'	1488.56	
h, k, lmax	14, 14, 34	14, 14, 34
Nref	7752	7679
Tmin, Tmax	0.987, 0.987	
Tmin'	0.987	

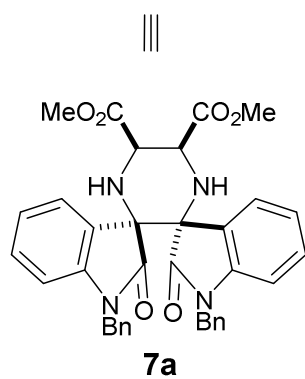
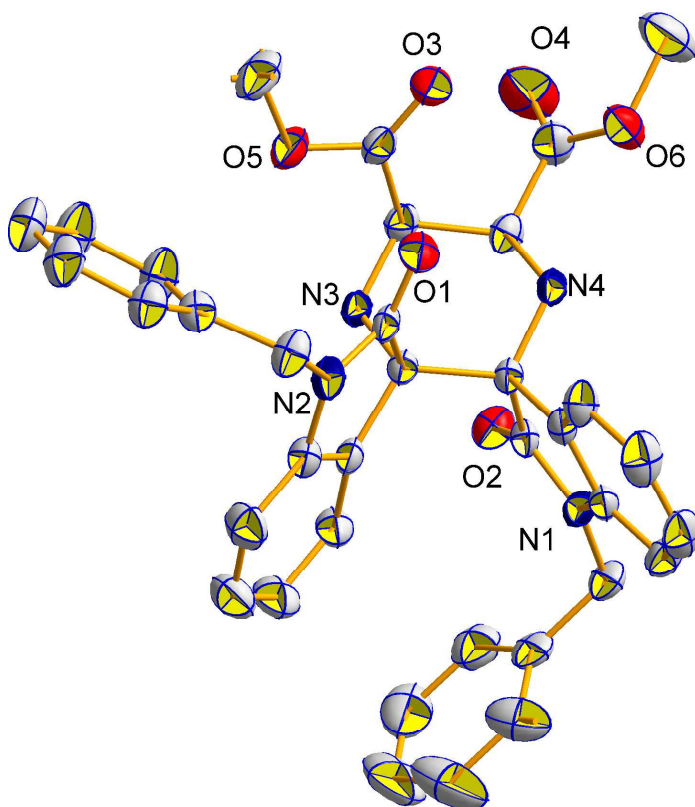
Correction method= Not given

Data completeness= 0.991 Theta(max)= 25.030

R(reflections)= 0.0593(4329) wR2(reflections)= 0.1809(7679)

S = 0.956 Npar= 487

X-Ray crystallographic analysis of dispirooxindole-piperazine 7a (CCDC 1021637) showing the thermal ellipsoids at 30% probability level.



Bond precision: C-C = 0.0056 Å Wavelength=0.71073

Cell: a=13.369(18) b=21.18(3) c=10.884(15)
 alpha=90 beta=106.287(15) gamma=90
 Temperature: 296 K

	Calculated	Reported
Volume	2958(7)	2958(7)
Space group	P 21/c	P2(1)/c
Hall group	-P 2ybc	?
Moiety formula	C36 H32 N4 O6	?
Sum formula	C36 H32 N4 O6	C2.44 H2.17 N0.27 O0.41
Mr	616.66	41.81
Dx, g cm ⁻³	1.385	1.385
Z	4	59
Mu (mm ⁻¹)	0.096	0.096
F000	1296.0	1296.0
F000'	1296.60	
h, k, lmax	17, 27, 14	17, 27, 14
Nref	7039	6912
Tmin, Tmax	0.977, 0.990	
Tmin'	0.972	

Correction method= Not given

Data completeness= 0.982 Theta(max)= 27.840

R(reflections)= 0.0844(4042) wR2(reflections)= 0.2199(6912)

S = 1.046 Npar= 421
