

# Synthesis of 6,7-Dihydrodibenzo[*b,j*]phenanthroline Derivatives by Pfitzinger Condensation of Isatin and Cyclic Diketones

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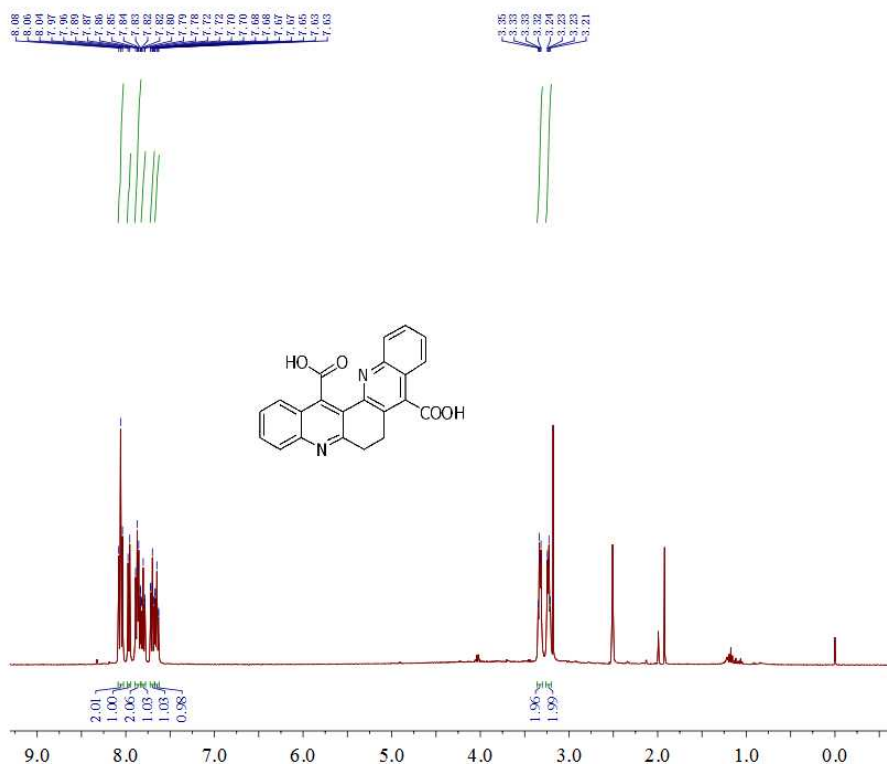
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1H SOLVENT:DMSO NO:1 2012-09-30



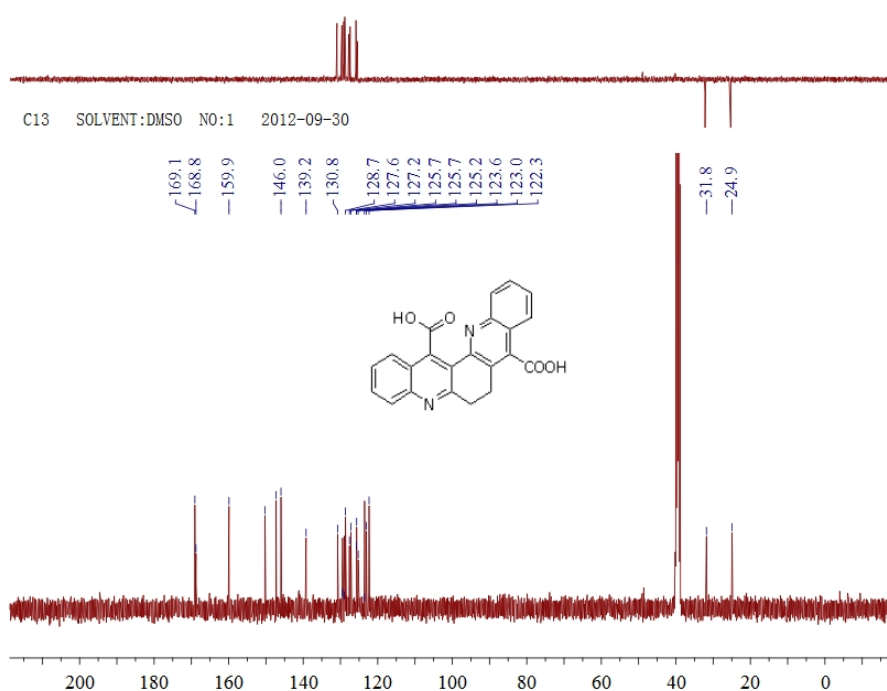
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SWH 8012.820 Hz  
FIDRES 0.244532 Hz  
AQ 2.0447731 sec  
RG 287.4  
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DE 6.00 usec  
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MCREST 0.00000000 sec  
MCWRK 0.01500000 sec

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F2 - Processing parameters  
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SF 400.2000066 MHz  
WDW EM  
SSB 0  
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GB 0  
PC 1.00

dept135 SOLVENT:DMSO NO:1 2012-09-30



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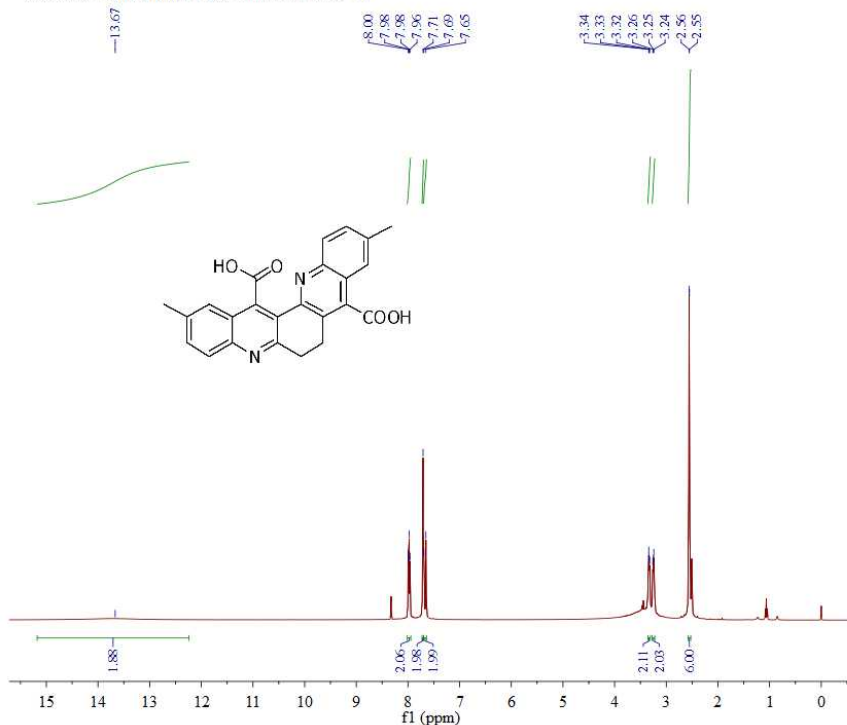
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SWH 26178.010 Hz  
FIDRES 0.798889 Hz  
AQ 0.6259188 sec  
RG 14596.5  
DW 19.100 usec  
DE 6.00 usec  
TE 299.5 K  
D1 10.00000000 sec  
d11 0.03000000 sec  
MCREST 0.00000000 sec  
MCWRK 0.01500000 sec

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NUC1 13C  
P1 9.30 usec  
PL1 -3.00 dB  
SF01 100.6411445 MHz

===== CHANNEL f2 =====  
CPDPRG2 waltz16  
NUC2 1H  
PCPD2 80.00 usec  
PL2 -3.00 dB  
PL12 14.23 dB  
SF02 400.2019910 MHz

Figure S1. <sup>1</sup>H and <sup>13</sup>C NMR spectra of 5a.

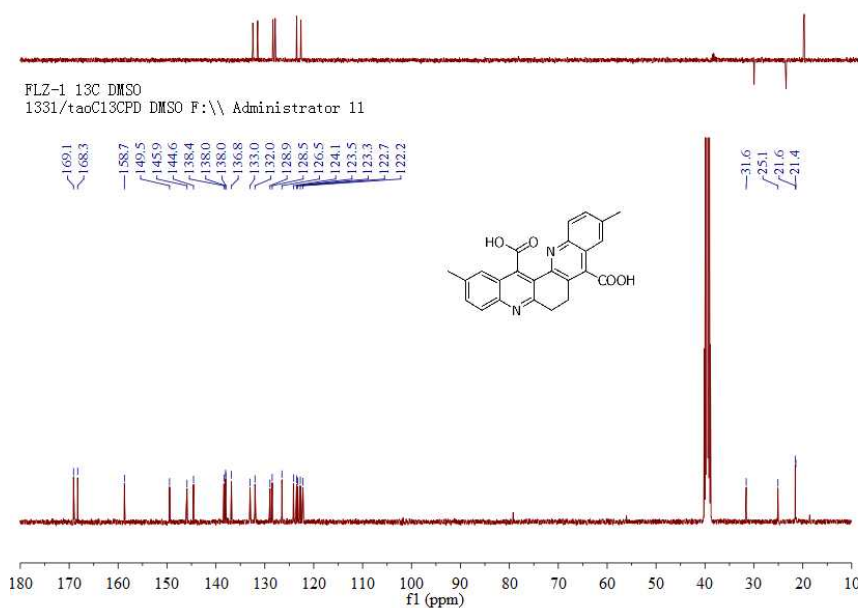
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1317/tao PROTON DMSO F:\\ Administrator 11



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PROCNO 1  
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INSTRUM spect  
PROBHD 5 mm PABBO BB-  
PULPROG zg30  
TD 65536  
SOLVENT DMSO  
NS 16  
DS 2  
SWH 8223.685 Hz  
FIDRES 0.125483 Hz  
AQ 3.9846387 sec  
RG 114  
DW 60.800 usec  
DE 6.50 usec  
TE 295.1 K  
D1 1.00000000 sec  
TDO 1

===== CHANNEL f1 =====  
NUC1 1H  
P1 12.00 usec  
PL1 -2.50 dB  
PL1W 13.53451252 W  
SFO1 400.1324710 MHz  
SI 32768  
SF 400.1300004 MHz  
WDW EM  
SSB 0  
LB 0.30 Hz  
GB 0  
PC 1.00

FLZ-1 dept135 DMSO  
1331/tao C13DEPT135 DMSO F:\\ Administrator 11



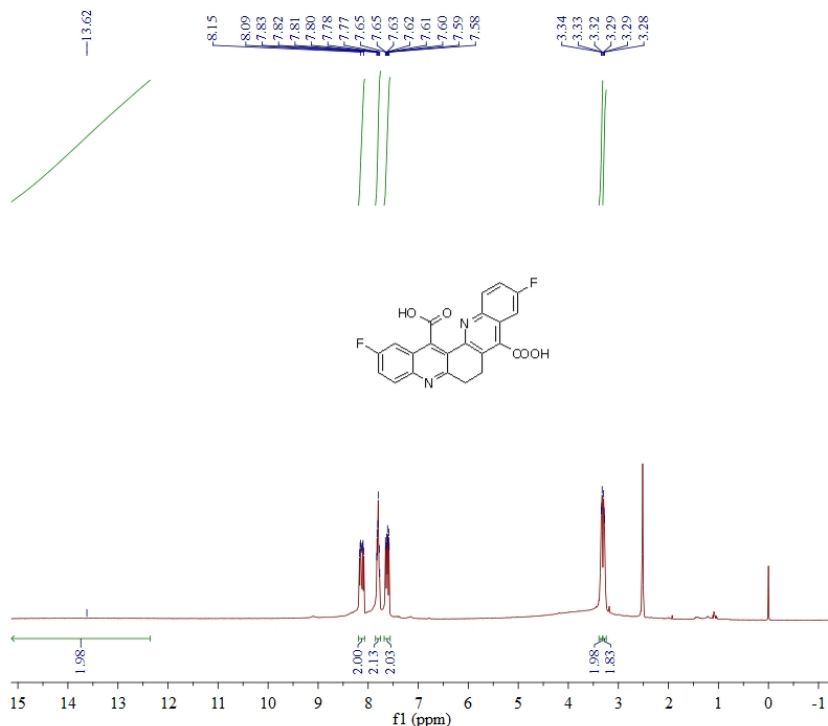
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PROCNO 1  
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PULPROG zgpg30  
TD 65536  
SOLVENT DMSO  
NS 512  
DS 4  
SWH 24038.461 Hz  
FIDRES 0.366798 Hz  
AQ 1.3631988 sec  
RG 912  
DW 20.800 usec  
DE 6.50 usec  
TE 295.2 K  
D1 2.00000000 sec  
D11 0.03000000 sec  
TDO 1

===== CHANNEL f1 =====  
NUC1 13C  
P1 11.25 usec  
PL1 -0.50 dB  
PL1W 35.22978592 W  
SFO1 100.6240377 MHz

===== CHANNEL f2 =====  
CPDPRG2 waltz16  
NUC2 1H  
PCPD2 80.00 usec  
PL2 -2.50 dB  
PL12 13.98 dB  
PL13 13.00 dB  
PL2W 13.53451252 W

Figure S2. <sup>1</sup>H and <sup>13</sup>C NMR spectra of **5b**

FLZ-2 1H DMSO  
1318/tao PROTON DMSO F:\Administrator 12



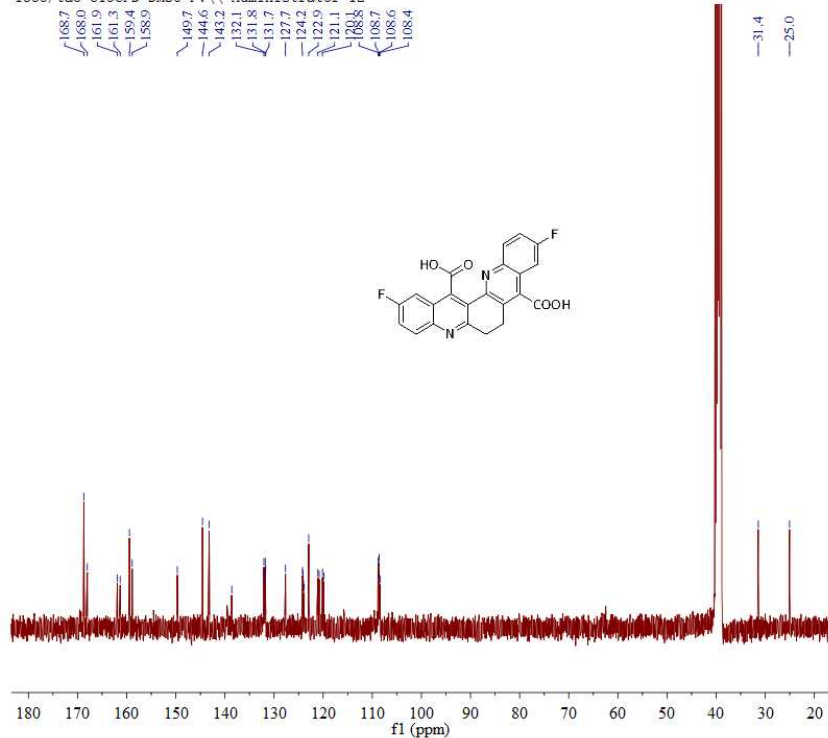
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Date_         20130514
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PULPROG       zg30
TD            65536
SOLVENT       DMSO
NS            16
DS            2
SWH           8223.685 Hz
FIDRES        0.125483 Hz
AQ            3.9846387 sec
RG            256
DW            60.800 usec
DE            6.50 usec
TE            295.1 K
D1            1.00000000 sec
TDO           1
  
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```

===== CHANNEL f1 =====
NUC1          1H
P1            12.00 usec
PL1           -2.50 dB
PL1W          13.53451252 W
SF01          400.1324710 MHz
SI            32768
SF            400.1299982 MHz
WDW           EM
SSB           0
LB            0.30 Hz
GB            0
PC            1.00
  
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FLZ-2 13C DMSO  
1333/tao C13CPD DMSO F:\Administrator 12



```

NAME          tao
EXPNO         1333
PROCNO        1
Date_         20130515
Time          6.04
INSTRUM       spect
PROBHD        5 mm PABBO BB-
PULPROG       zgpg30
TD            65536
SOLVENT       DMSO
NS            512
DS            4
SWH           24038.461 Hz
FIDRES        0.366798 Hz
AQ            1.3631988 sec
RG            64
DW            20.800 usec
DE            6.50 usec
TE            295.2 K
D1            2.00000000 sec
D11           0.03000000 sec
TDO           1
  
```

```

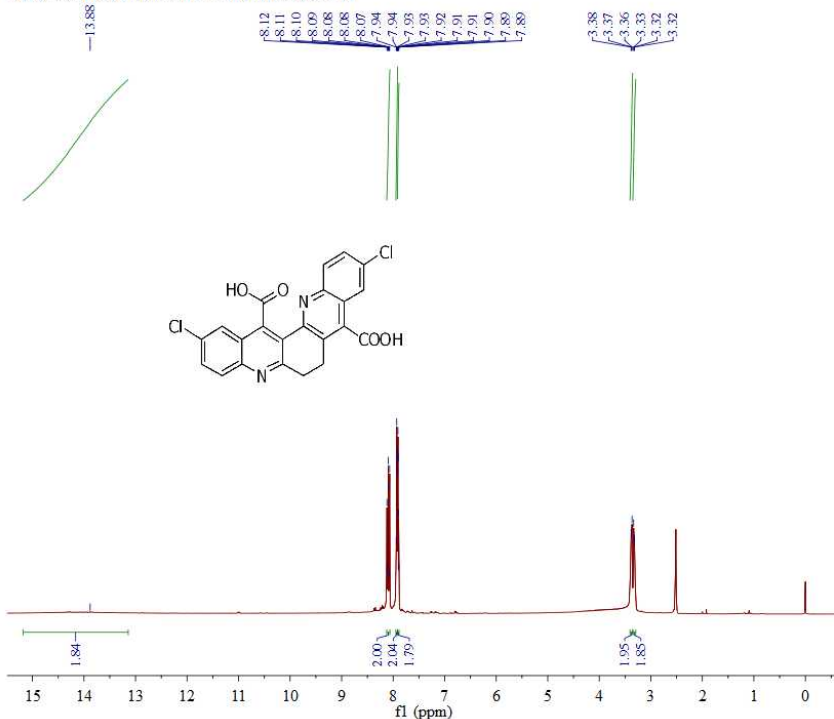
===== CHANNEL f1 =====
NUC1          13C
P1            11.25 usec
PL1           -0.50 dB
PL1W          35.22978592 W
SF01          100.6240377 MHz
  
```

```

===== CHANNEL f2 =====
CPDPRG2       waltz16
NUC2          1H
PCPD2         80.00 usec
PL2           -2.50 dB
PL12          13.98 dB
PL13          13.00 dB
PL2W          13.53451252 W
PL12W         0.30439863 W
PL13W         0.38145441 W
SF02          400.1316005 MHz
  
```

Figure S3. <sup>1</sup>H and <sup>13</sup>C NMR spectra of 5c

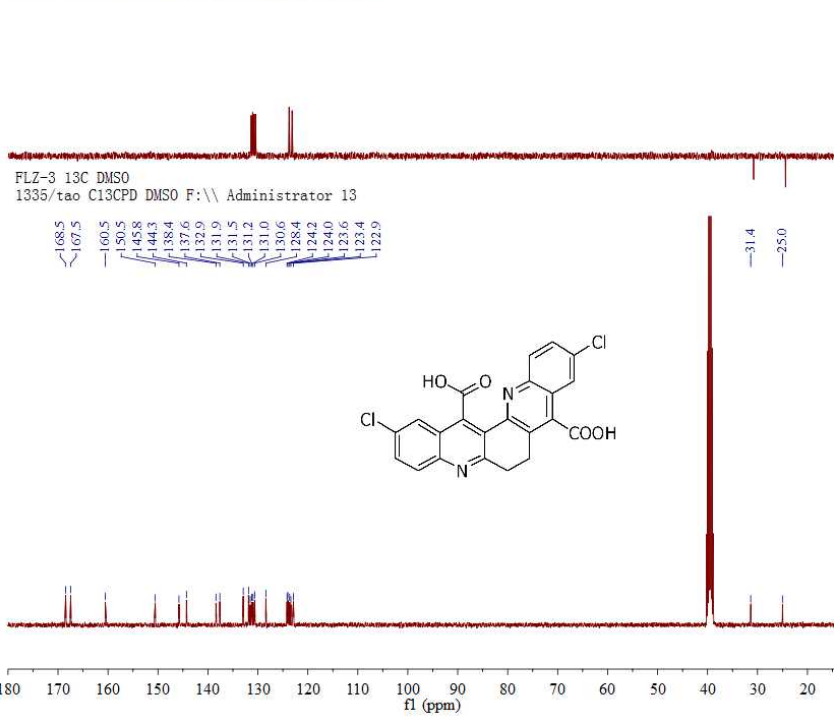
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EXPNO 1319  
PROCNO 1  
Date\_ 20130514  
Time 18.13  
INSTRUM spect  
PROBHD 5 mm PABBO BB-  
PULPROG zg30  
TD 65536  
SOLVENT DMSO  
NS 16  
DS 2  
SWH 8223.685 Hz  
FIDRES 0.125483 Hz  
AQ 3.9846387 sec  
RG 287  
DW 60.800 usec  
DE 6.50 usec  
TE 295.2 K  
D1 1.0000000 sec  
TDO 1

===== CHANNEL f1 =====  
NUC1 1H  
P1 12.00 usec  
PL1 -2.50 dB  
PL1W 13.53451252 W  
SFO1 400.1324710 MHz  
SI 32768  
SF 400.1299978 MHz  
WDW EM  
SSB 0  
LB 0.30 Hz  
GB 0  
PC 1.00

FLZ-3 dept135 DMSO  
1336/tao C13CPD DMSO F:\\ Administrator 13

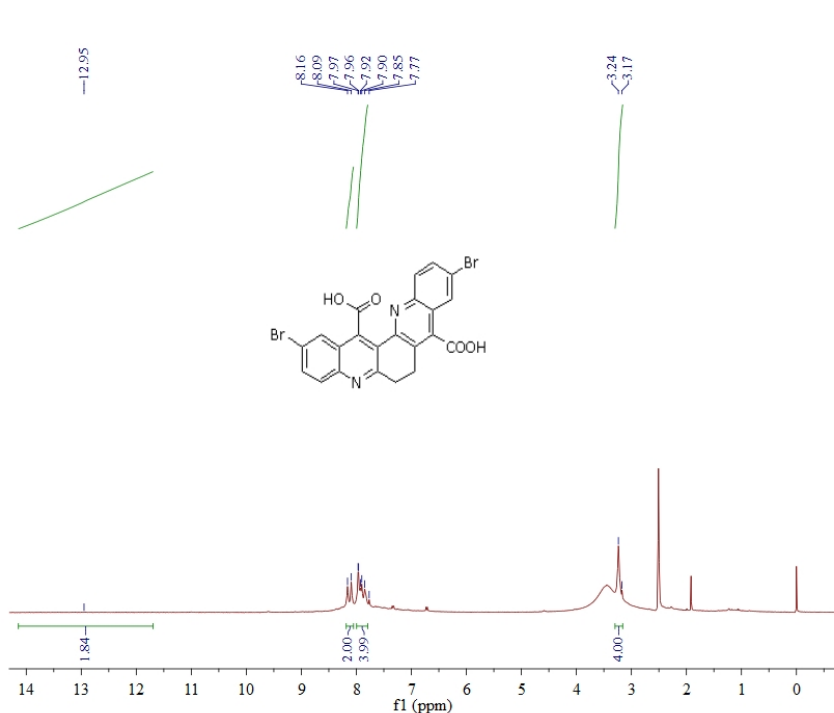


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PROCNO 1  
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PULPROG zgpg30  
TD 65536  
SOLVENT DMSO  
NS 512  
DS 4  
SWH 24038.461 Hz  
FIDRES 0.366798 Hz  
AQ 1.3631988 sec  
RG 1030  
DW 20.800 usec  
DE 6.50 usec  
TE 295.2 K  
D1 2.0000000 sec  
D11 0.0300000 sec  
TDO 1

===== CHANNEL f1 =====  
NUC1 13C  
P1 11.25 usec  
PL1 -0.50 dB  
PL1W 35.22978592 W  
SFO1 100.6240377 MHz  
===== CHANNEL f2 =====  
CPDPRG2 waltz16  
NUC2 1H  
PCPD2 80.00 usec  
PL2 -2.50 dB  
PL12 13.98 dB  
PL13 13.00 dB  
PL2W 13.53451252 W  
PL12W 0.30439863 W  
PL13W 0.38145441 W  
SFO2 400.1316005 MHz  
SI 32768  
SF 100.6128193 MHz

. Figure S4. <sup>1</sup>H and <sup>13</sup>C NMR spectra of 5d

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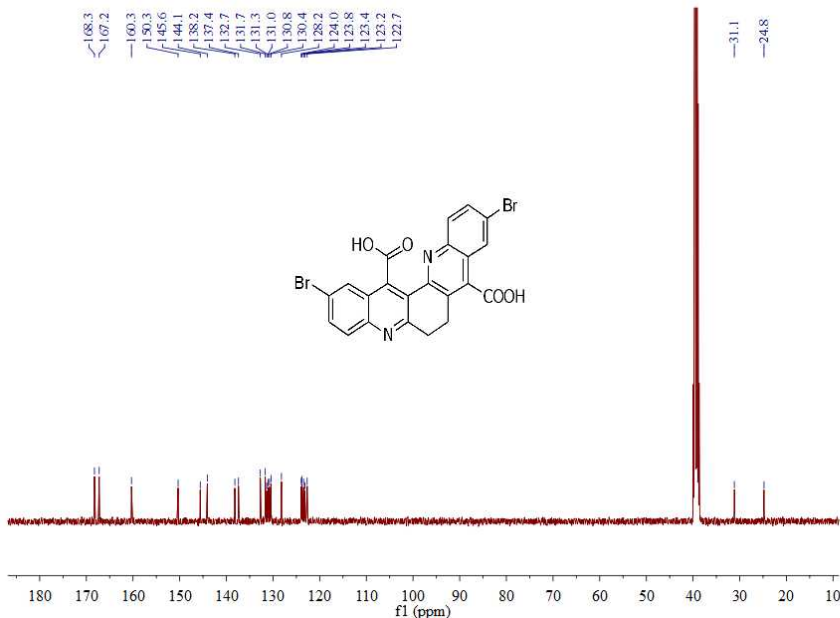
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EXPNO         1320
PROCNO        1
Date_         20130514
Time          18.18
INSTRUM       spect
PROBHD        5 mm PABBO BB-
PULPROG       zg30
TD            65536
SOLVENT       DMSO
NS            16
DS            2
SWH           8223.685 Hz
FIDRES        0.125483 Hz
AQ            3.9846387 sec
RG            322
DW            60.800 usec
DE            6.50 usec
TE            295.1 K
D1            1.0000000 sec
TD0           1
  
```

```

===== CHANNEL f1 =====
NUC1          1H
P1            12.00 usec
PL1           -2.50 dB
PL1W          13.53451252 W
SF01          400.1324710 MHz
SI            32768
SF            400.1300008 MHz
WDW           EM
SSB           0
LB            0.30 Hz
GB            0
PC            1.00
  
```

FLZ-4 13C DMSO  
1337/tao C13CPD DMSO F:\ Administrator 13



```

NAME          tao
EXPNO         1337
PROCNO        1
Date_         20130515
Time          7.25
INSTRUM       spect
PROBHD        5 mm PABBO BB-
PULPROG       zgpg30
TD            65536
SOLVENT       DMSO
NS            512
DS            4
SWH           24038.461 Hz
FIDRES        0.366798 Hz
AQ            1.3631988 sec
RG            1030
DW            20.800 usec
DE            6.50 usec
TE            295.2 K
D1            2.0000000 sec
D11           0.0300000 sec
TD0           1
  
```

```

===== CHANNEL f1 =====
NUC1          13C
P1            11.25 usec
PL1           -0.50 dB
PL1W          35.22978592 W
SF01          100.6240377 MHz
  
```

```

===== CHANNEL f2 =====
CPDPRG2       waltz16
NUC2          1H
PCPD2         80.00 usec
PL2           -2.50 dB
PL12          13.98 dB
PL13          13.00 dB
PL2W          13.53451252 W
PL12W         0.30439863 W
PL13W         0.38145441 W
SF02          400.1316005 MHz
SI            32768
SF            100.6128193 MHz
  
```

Figure S5. <sup>1</sup>H and <sup>13</sup>C NMR spectra of **5e**

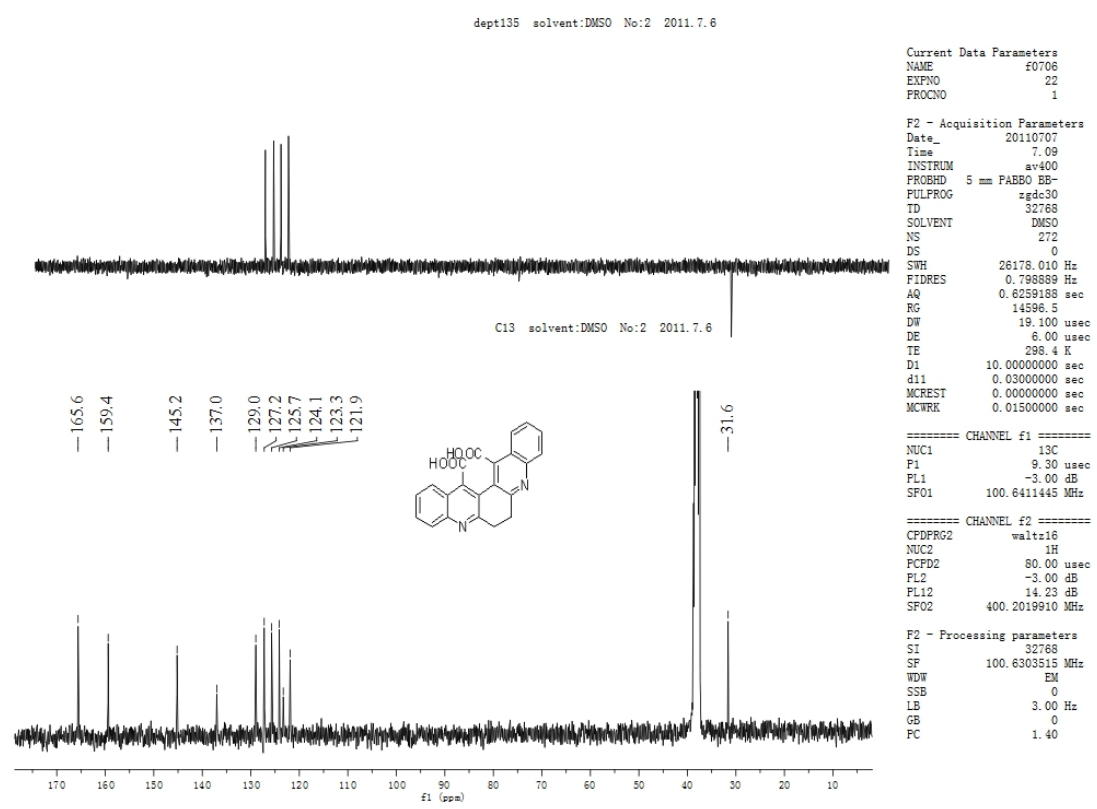
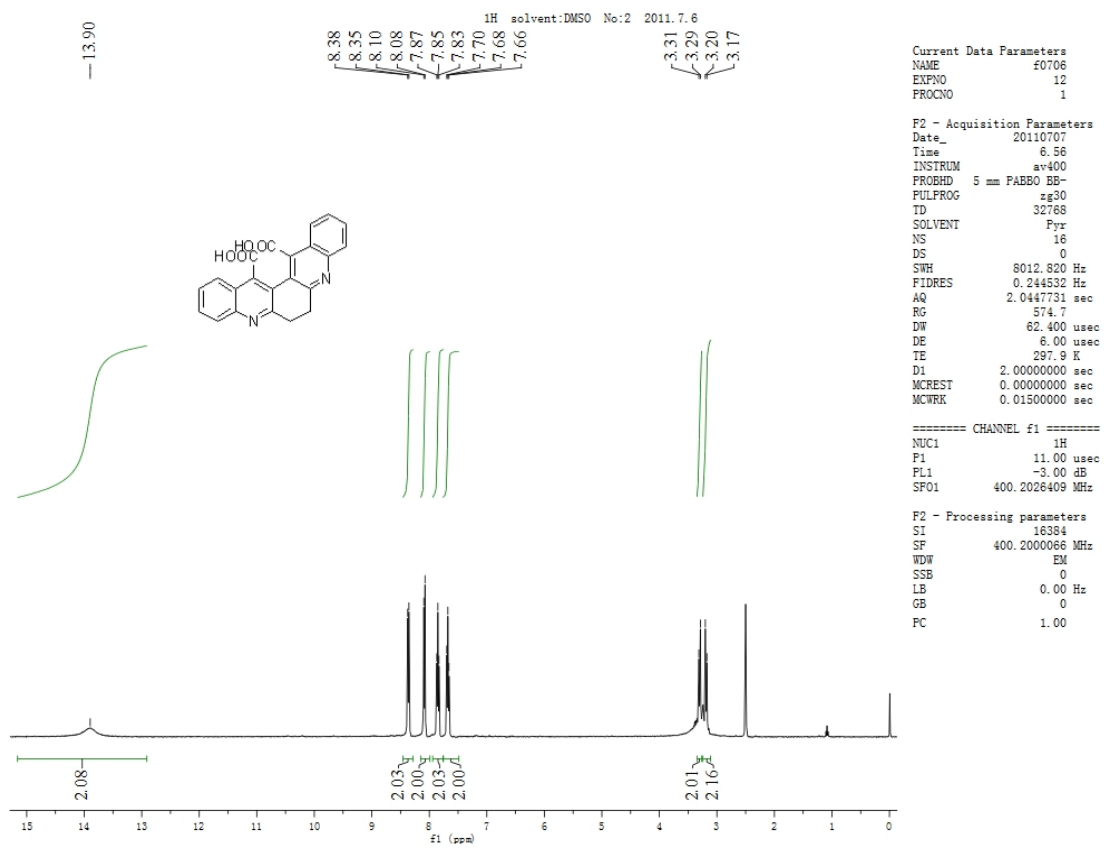


Figure S6. <sup>1</sup>H and <sup>13</sup>C NMR spectra of **6a**.

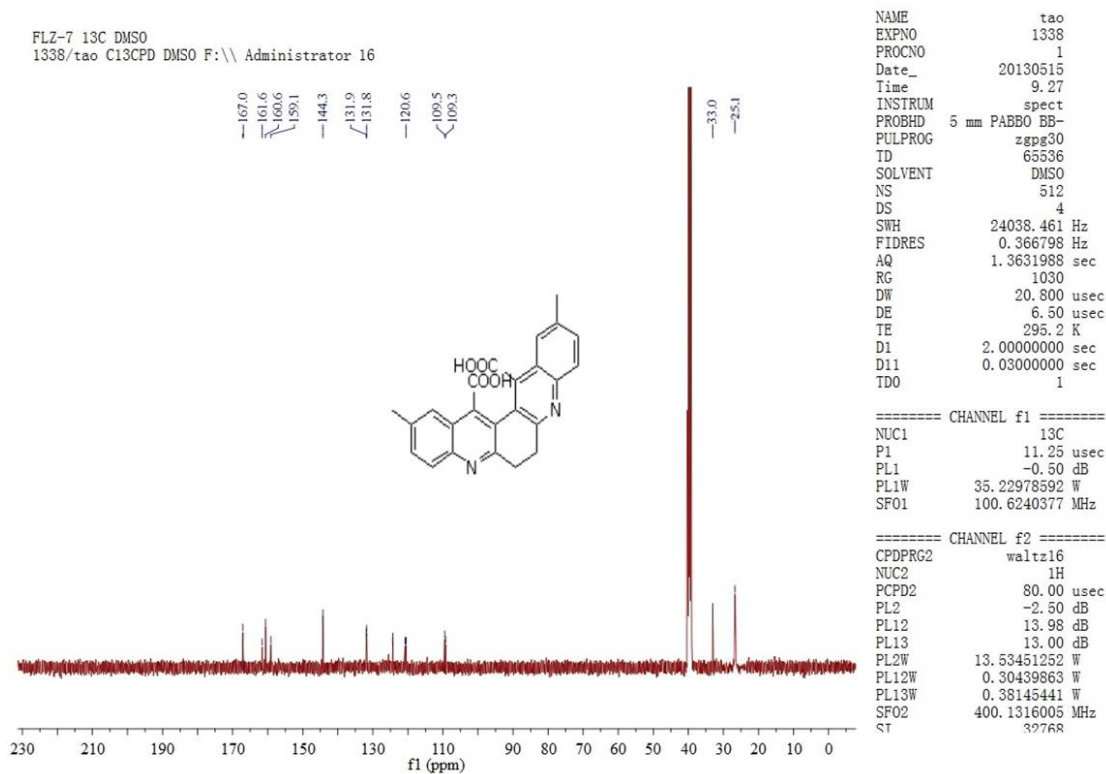
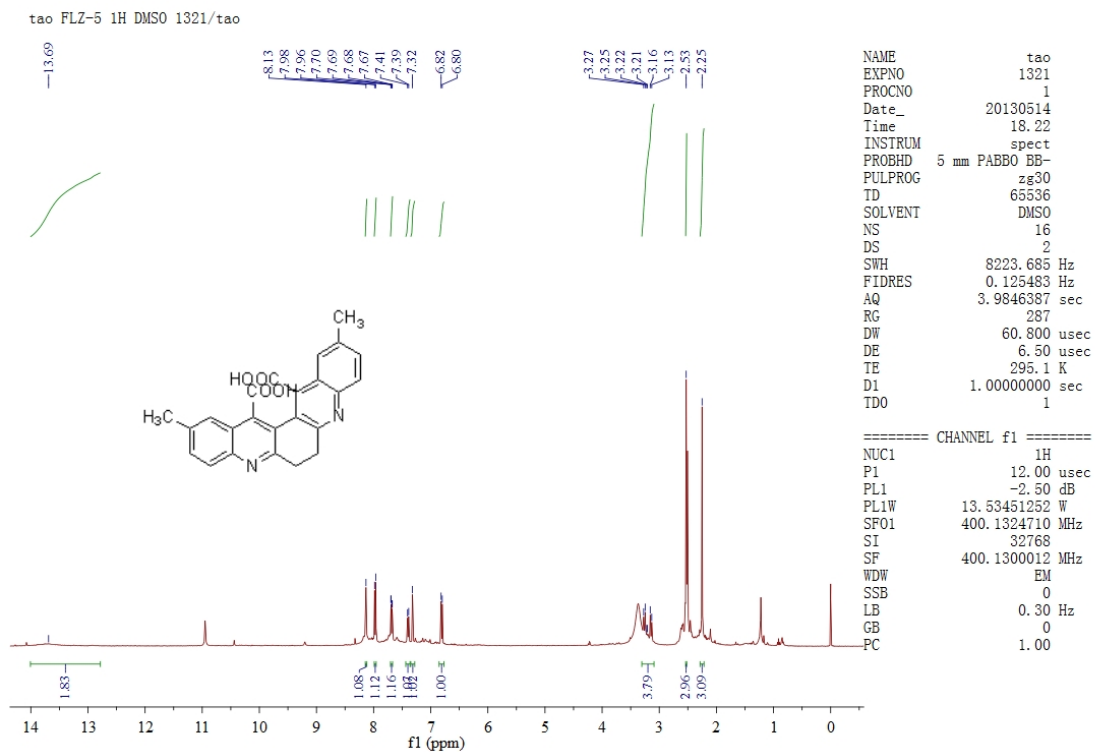
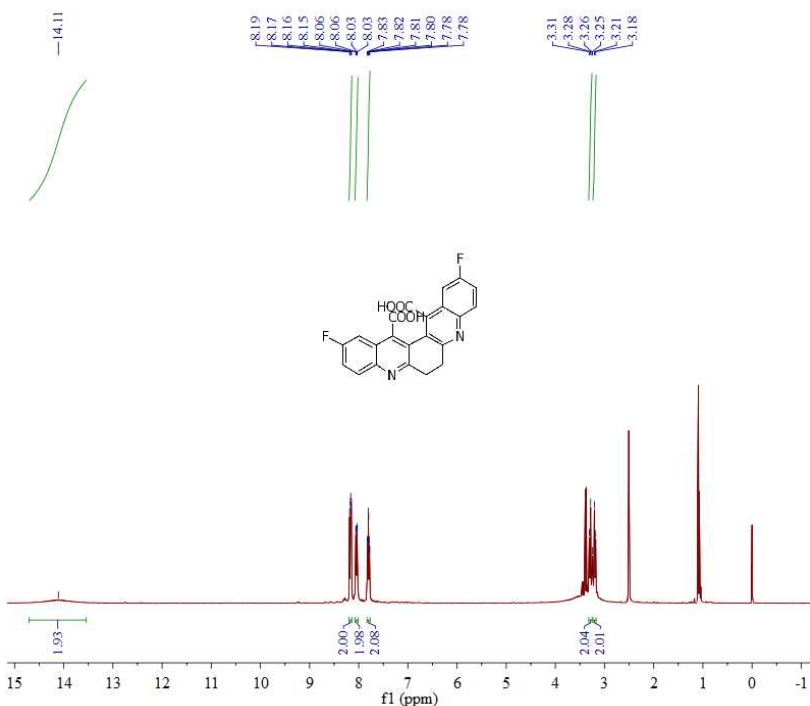


Figure S7. <sup>1</sup>H and <sup>13</sup>C NMR spectra of **6b**

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DMSO 1322/tao PROTON DMSO F:\ Administrator 16



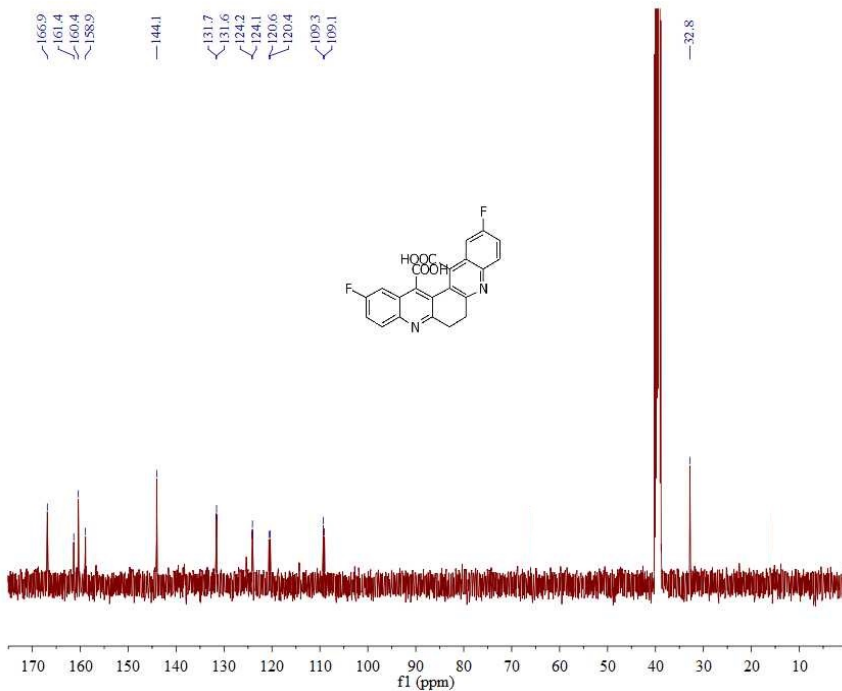
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Time          18.27
INSTRUM       spect
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PULPROG       zg30
ID            65536
SOLVENT       DMSO
NS            16
DS            2
SWH           8223.685 Hz
FIDRES        0.125483 Hz
AQ            3.9846387 sec
RG            406
DW            60.800 usec
DE            6.50 usec
TE            295.2 K
D1            1.00000000 sec
D10           1
TDO           1
  
```

```

===== CHANNEL f1 =====
NUC1          1H
P1            12.00 usec
PL1           -2.50 dB
PL1W         13.53451252 W
SF01         400.1324710 MHz
SI            32768
SF           400.1300008 MHz
WDW           EM
SSB           0
LB            0.30 Hz
GB            0
PC            1.00
  
```

FLZ-6 13C DMSO  
1337/tao C13CPD DMSO F:\ Administrator 16



```

NAME          tao
EXPNO         1337
PROCNO        1
Date_         20130515
Time          7.27
INSTRUM       spect
PROBHD        5 mm PABBO BB-
PULPROG       zgpg30
ID            65536
SOLVENT       DMSO
NS            512
DS            4
SWH           24038.461 Hz
FIDRES        0.366798 Hz
AQ            1.3631988 sec
RG            1030
DW            20.800 usec
DE            6.50 usec
TE            295.2 K
D1            2.00000000 sec
D11           0.03000000 sec
D10           1
TDO           1
  
```

```

===== CHANNEL f1 =====
NUC1          13C
P1            11.25 usec
PL1           -0.50 dB
PL1W         35.22978592 W
SF01         100.6240377 MHz
  
```

```

===== CHANNEL f2 =====
CPDPRG2       waltz16
NUC2          1H
PCPD2         80.00 usec
PL2           -2.50 dB
PL12          13.98 dB
PL13          13.00 dB
PL2W         13.53451252 W
PL12W        0.30439863 W
PL13W        0.38145441 W
SF02         400.1316005 MHz
SI            32768
  
```

Figure S8. <sup>1</sup>H and <sup>13</sup>C NMR spectra of 6c.

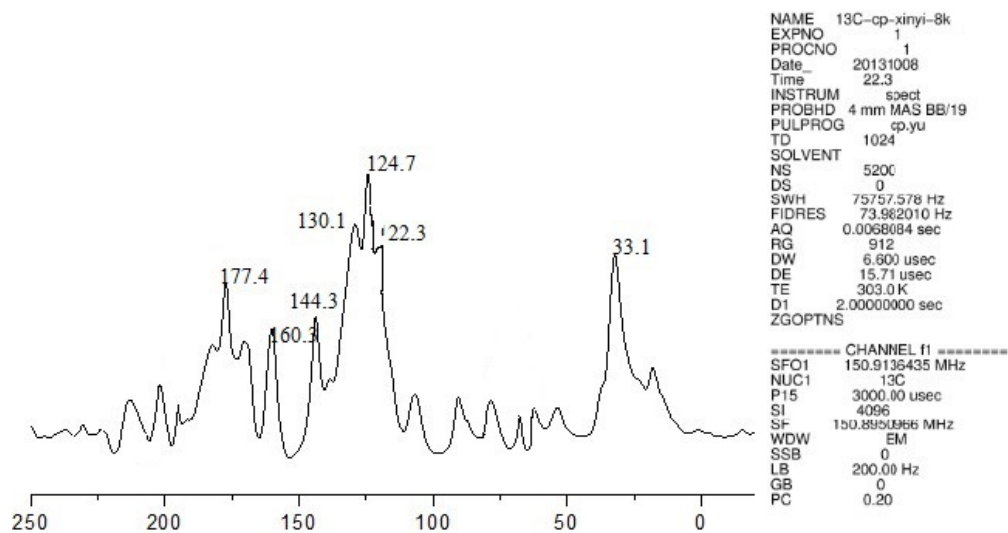


Figure S9  $^{13}\text{C}$  CP/MAS NMR spectra of **6d**.

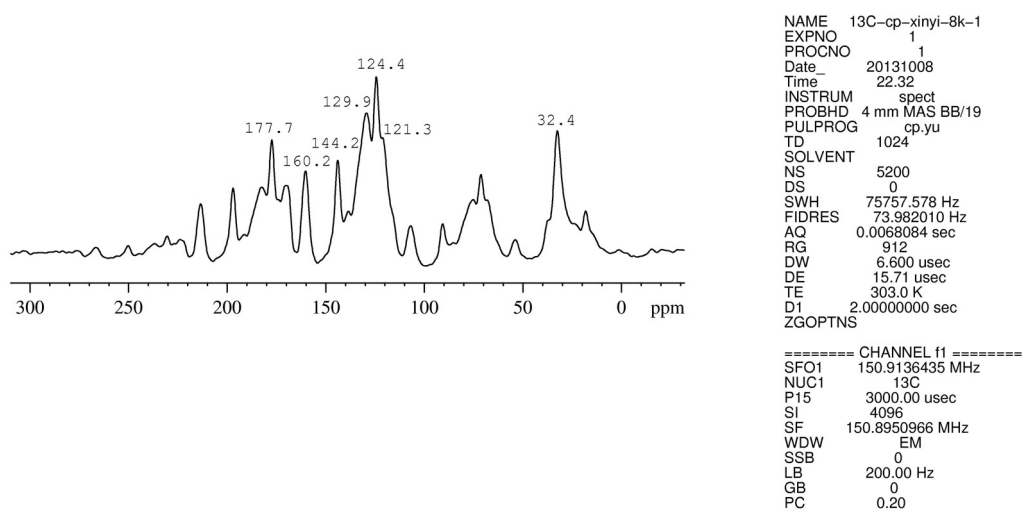


Figure S10  $^{13}\text{C}$  CP/MAS NMR spectra of **6e**.