Supporting Information for

An Expedient Synthesis towards Fused Heteroacenes Bearing Pyrrolo[3,2-*b*]pyrrole Core

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Experimental Details

General: All glass wares were thoroughly oven-dried. Chemicals and solvents were either purchased from commercial suppliers or purified by standards techniques. Flash chromatography was carried out utilizing silica gel 100-200 mesh. HRMS were measured on a Bruke Maxis UHR TOF unless otherwise mentioned. ^{1}H NMR and ^{13}C NMR spectra were recorded on a Bruker AV600 with tetramethylsilane (TMS) as an internal standard. The infrared spectra were acquired on Nicolet 6700 FTIR Spectrometer and The UV-vis and fluorescence spectra were collected with a Hitachi U-4100 UV-vis spectrophotometer. Cyclic voltammetry was performed on an electrochemistry workstation (CHI660D, Chenhua Shanghai) using an anhydrous and argon–saturated solution of 0.1 M Tetrabutylammonium Perchlorate (TBAP) in N,N-dimethylformamide (DMF) as an electrolyte and an Ag/AgCl electrode as a reference electrode. The potential of Ag/AgCl in DMF was determined using ferrocene as internal standard. The OFET devices were made by sublimating the molecules (about 50-nm thickness) to OTS-treated SiO_2/Si substrates under a vacuum of ca. 6×10^{-4} Pa. The device performance was measured in a top-contact bottom-gate configuration (drain and source electrodes deposited above the semiconductor) with Au electrodes. Organic semiconductors were deposited 10 nm at a rate of 1 A/min, then deposited 30 nm at a rate of 10 A/min. Gold source and drain contacts were patterned 50 nm by thermal evaporation using shadow masks. The channel length (L) and width (W) were 80 and 8800 μm, respectively. The device characteristics of the OFET devices were determined using a Keithley 4200 SCS semiconductor parameter analyzer at room temperature in air.

Synthesis Details:

Dibenzo[b,f][1,5]diazocine-6,12(5H,11H)-dione (13).[1] Methyl anthranilate (4.70g, 31.0 mmol, 1.0 equiv.) was added dropwise to the suspension of NaH (2.48 g, 62.0 mmol, 2.0 equiv., 60%) in anhydrous THF (60 mL) at 25^{o} C. the resulting mixture was gradually heated to reflux, and stirred for 3 days. The mixture was cooled down to rt and then poured slowly into 300 mL of 0.1 M HCl and ice. After the ice had melted, the precipitated product was collected by filtration, washed several times with de-ionized water, and air-dried to yield the crude product, which was then recrystallized from aqueous EtOH to yield pure product(2.8 g, 75%) as a light yellow powder. δ_H (600 MHz; DMSO-d_6; Me_4Si) 10.20 (2H, s, NHCO), 7.36-7.30 (4H, m, Ar), 7.22 (2H, t, J =7.4Hz, Ar), 7.07 (2H, d, J = 7.9 Hz, Ar). δ_C (150 MHz; DMSO-d_6; Me_4Si) 169.7,135.2, 134.0,131.0,128.6, 127.7, 126.2. HRMS(EI) calcd. for $C_{14}H_{10}N_2O_2$ $[M+H]^+$, 239.0815, found 239.0819.

Dinaphtho[2,3-b:2',3'-f][1,5]diazocine-7,15(6H,14H)-dione (15).[2] Methyl 3-amino-2-naphthoate (3.40 g, 17.0 mmol, 1.0 equiv.) was added in portions to the suspension of NaH (2.48 g, 62.0 mmol, 2.0 equiv. , 60%) in anhydrous THF (60mL) at 25^{o} C. the resulting mixture was gradually heated to reflux, and stirred for 3 days. The mixture was cooled down to rt and then poured slowly into 300 mL of 0.1 M HCl and ice, After the ice had melted, the precipitated product was collected by filtration, washed several times

with deionized water, and air-dried to yield the crude product, which was then recrystallized from aqueous EtOH to yield pure product (2.8 g, 75%) as a yellow powder. δ_H (600 MHz; DMSO-d_6; Me_4Si) 10.48 (2H, s, NHCO), 7.96 (2H, s, Ar), 7.90 (2H, d, J =7.8Hz, Ar), 7.86 (2H, d, J =8.0Hz, Ar), 7.66 (2H, s, Ar), 7.50-7.46 (4H, m, Ar). δ_C (150 MHz; DMSO-d_6; Me_4Si) 170.0, 133.8, 133.6, 133.2, 131.6, 128.4, 128.3, 128.2, 127.8, 127.2, 124.5. HRMS(EI) calcd. for $C_{22}H_{14}N_2O_2$ $[M+Na]^+$: 361.0947, found: 361.1006.

6,12-Dichlorodibenzo[b,f][1,5]diazocine (11).[3] Finely powdered dibenzob,f][1,5] diazocine-6,12(5H,11H)-dione (0.90g, 3.77mmol, 1 equiv.) and phosphorus pentachloride (1.68 g, 8.12 mmol, 2.15 equiv.) were boiled together in chloroform (15 ml) for 4 hr. After filtration, the solid which separated on concentration of the filtrate was crystallized from light petroleum and afforded the desired product as a white solid (0.66 g , 64%). IR (KBr, cm^{-1}): 3160, 3034, 2900, 1649 (C=N), 1598, 1481, 1439, 1397, 1216, 948, 755(C-Cl). δ_H (600 MHz; $CDCl_3$; Me_4Si) 7.38-7.34 (4H, m, Ar), 7.15 (2H, t, J=7.6Hz, Ar), 6.99 (2H, d, J= 8.1Hz, Ar). δ_C (150 MHz; $CDCl_3$; Me_4Si) 156.3, 145.3, 131.6, 127.1, 126.1, 125.4, 122.0.

7,15-Dichlorodinaphtho[2,3-b:2',3'-f][1,5]diazocine (16). Finely powdered dinaphtho-[2,3-b:2',3'-f][1,5]diazocine-7,15(6H,14H)-dione (1.9g, 5.6 mmol, 1.0 equiv.) and phosphorus pentachloride (2.5g, 12mmol, 2.15equiv.) were boiled together in chloroform (15 ml) for 4 hr. After filtration, the solid which separated on concentration of the filtrate was crystallized from light petroleum and afforded the desired product as a light yellow solid(1.2 g , 60%). m.p. : not determined (hydrolysis in air). IR (KBr, cm^{-1}): 3054, 2928, 1654(C=N), 1594, 1495, 1389, 1174, 1114, 929, 772(C-Cl). δ_H (600 MHz; $CDCl_3$; Me_4Si) 7.88 (1H, s, Ar); 7.77 (1H, d, J=8.4Hz, Ar), 7.71 (1H, d, J=8.4Hz, Ar), 7.49-7.47 (1H, m, Ar), 7.43-7.41 (2H, m, Ar). δ_C (150 MHz; $CDCl_3$; Me_4Si) 156.6, 141.5, 134.3, 130.2, 128.4, 128.3, 127.5(overlapped), 126.7, 126.4, 118.9. HRMS(EI) calcd. for $C_{22}H_{12}Cl_2N_2$ $[M+Na]^+$: 397.0269, found: 397.0325

5,10-Dihydroindolo[3,2-b]indole, DBPP (10). To the solution of 6,12-dichloro-dibenzo[b,f][1,5]diazocine (0.66g, 2.4 mmol, 1.0 equiv.) in anhydrous THF (60 mL) was added activated zinc(1.92g, 29mmol, 12.0 equiv.) in one portion, then TFA(4.4ml, 58mmol, 24.0 equiv.) was added dropwise at rt. The resulting suspension was stirred for 8h at that temperature, then saturated aqueous NH_4Cl was added to quench the reaction. The mixture was extracted with EtOAc and dried over anhydrous Na_2SO_4. Removal of the solvent under under reduced pressure afforded the crude product, which was then purified by recrystallization from MeOH to give the desired product as a white solid (0.34 g, 1.7 mmol, 70% yield). m.p. > 300°C. IR (KBr, cm^{-1}): 3400（NH）, 1459, 1404, 1322, 737. δ_H (600 MHz; $CDCl_3$; Me_4Si) 11.09 (2H, s, NH), 7.75(2H, d, J=7.8Hz, Ar), 7.49(2H, d, J=8.0Hz, Ar), 7.18-7.16 (2H, m, Ar), 7.09-7.07 (2H, m, Ar). δ_C (150 MHz; $CDCl_3$; Me_4Si) 140.8, 125.7, 121.8, 118.4, 117.9, 115.1, 112.6. HRMS(ESI) calcd. for $C_{14}H_{10}N_2$[M+Na]$^+$: 229.0736, found: 229.0734

Dinaphtho [2,3-*b*:2',3'-*f*]pyrrolo [3,2-*b*]pyrrole, DNPP， (17). To the solution of 7,15-dichlorodinaphtho[2,3-b:2',3'-f][1,5] diazocine(0.17g, 0.45 mmol, 1.0 equiv.) in anhydrous THF (10mL) was added activated zinc(0.44g, 6.8 mmol, 15.0 equiv.) in

one portion, then TFA(0.8ml, 10.6mmol, 24 equiv.) was added dropwise at rt. The resulting suspension was stirred for 8h at that temperature, then saturated aqueous NH_4Cl was added to quench the reaction. The mixture was extracted with EtOAc to give a suspension in organic layer, which was separated and dried over anhydrous Na_2SO_4. Removal of the solvent under reduced pressure afforded the crude product, which was then purified by recrystallization from MeOH to give the desired product as a white solid (70.0 mg, 0.23 mmol, 50% yield). m.p. > 300°C. IR (KBr, cm^{-1}): 3392（NH）, 1629, 1485, 1446, 861, 739. δ_H (600 MHz; DMSO-d_6; Me_4Si) 11.25 (2H, s, NH), 8.31(2H, s, Ar); 8.06(2H, d, J = 8.3Hz, Ar), 8.00-7.99 (4H, m, Ar), 7.40-7.33 (4H, m, Ar). ^{13}C NMR (DMSO-d_6, DEPTQ) δ: 141.5 (s), 130.0 (s), 128.0 (d), 127.8 (s), 127.4 (s), 127.2 (d), 123.6 (d), 122.2 (d), 117.3 (s), 114.9 (d), 107.2 (d). FTMS(ESI, Bruker Apex IV FTMS) calcd. for $C_{22}H_{12}C_2N_2$: $[M]^+$: 306.1152， found: 306.1153.

[1] (*a*) S. W. Gordon-Wylie, E. Teplin, J. C. Morris, M. I. Trombley, S. M. McCarthy, W. M. Cleaver and G. R. Clark, *Cryst. Growth Des*., 2004, 4, 789; (*b*) A. Hoorfar, W. D. Ollis, J. A. Price, J. S. Stephanatou and J. F. Stoddart, *J. Chem. Soc., Perkin Trans. 1*, 1982, 1649.

[2] C. Pardo, C. Pirat and J. Elguero, *J. Heterocyclic Chem.,* 2007, 44, 1303.

[3] F. C. Cooper and M. W. Partridge, *J. Chem. Soc*., 1955, 991

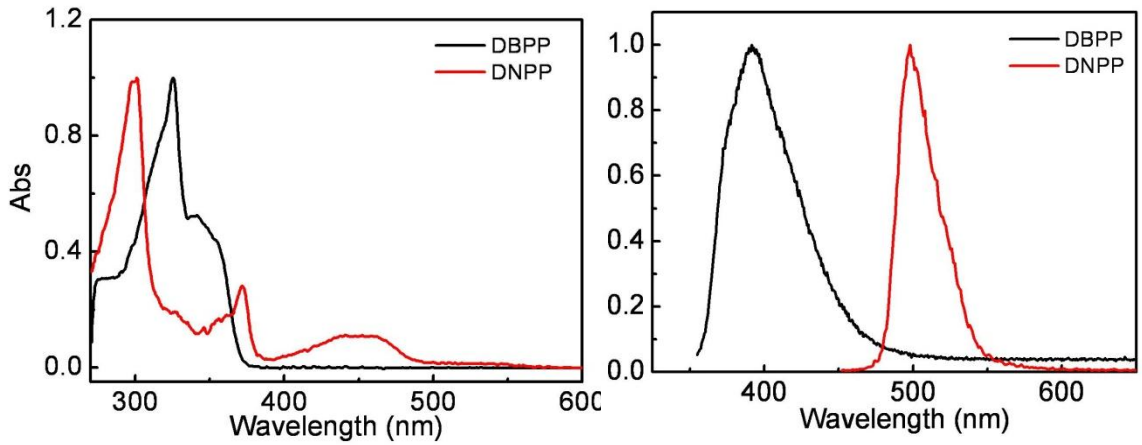


Fig. S1. Normalized UV-vis absorption (left) and fluorescence emission (right) of DBPP and DNPP in DMF.

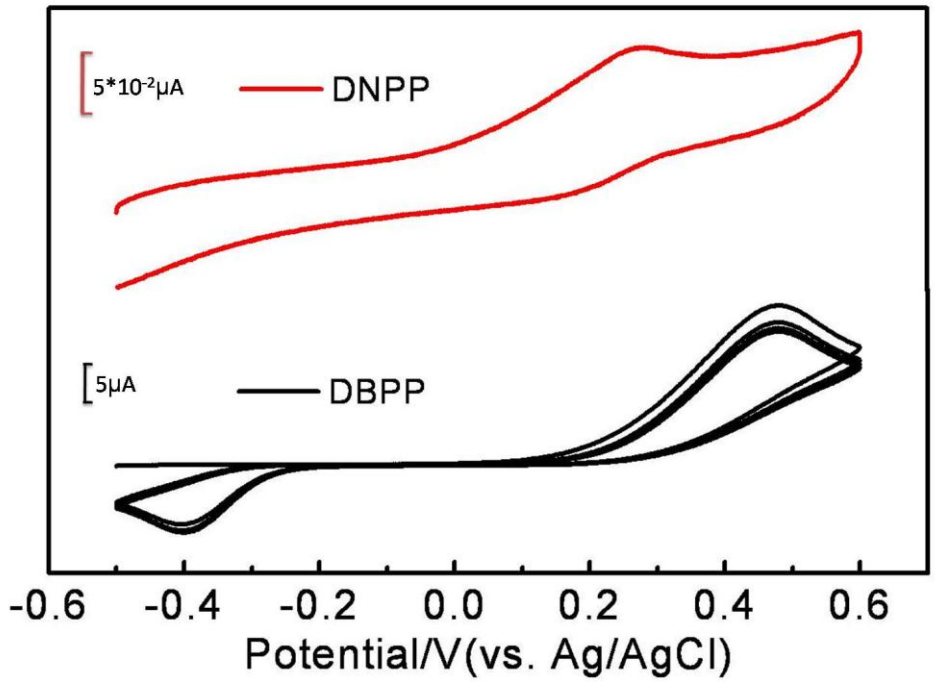


Figure S2. Cyclic Voltammetry of DBPP and DNPP in DMF (electrolyte: NBu_4ClO_4, 0.1 M， scan rate: 0.1 V/s). Five scans were recorded for DBPP to rule out the formation of polymer on the electrode.

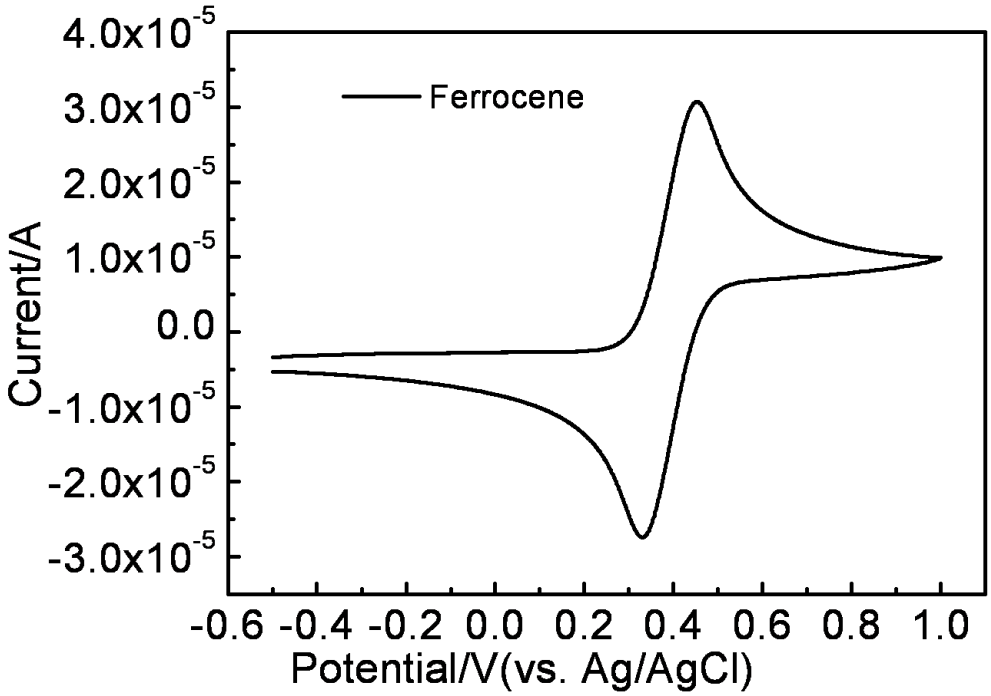


Figure S3. Cyclic voltammetry of ferrocene in DMF (electrolyte: NBu_4ClO_4, 0.1 M， scan rate: 0.1 V/s).

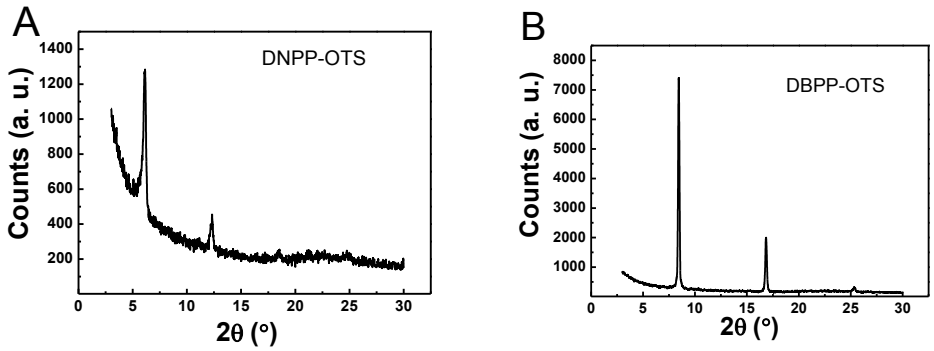


Figure S4. Thin-film XRD data of (A) DNPP and (B) DBPP.

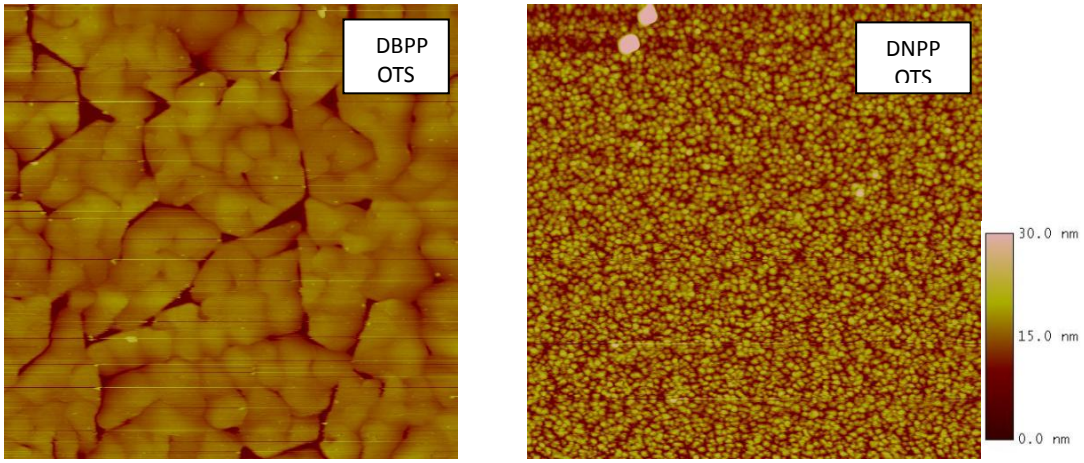


Figure S5. Thin- film AFM images of DBPP and DBPP.

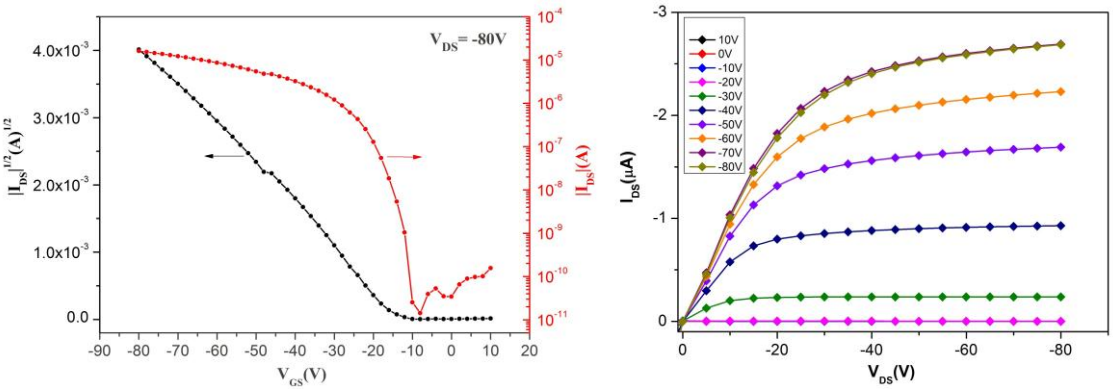
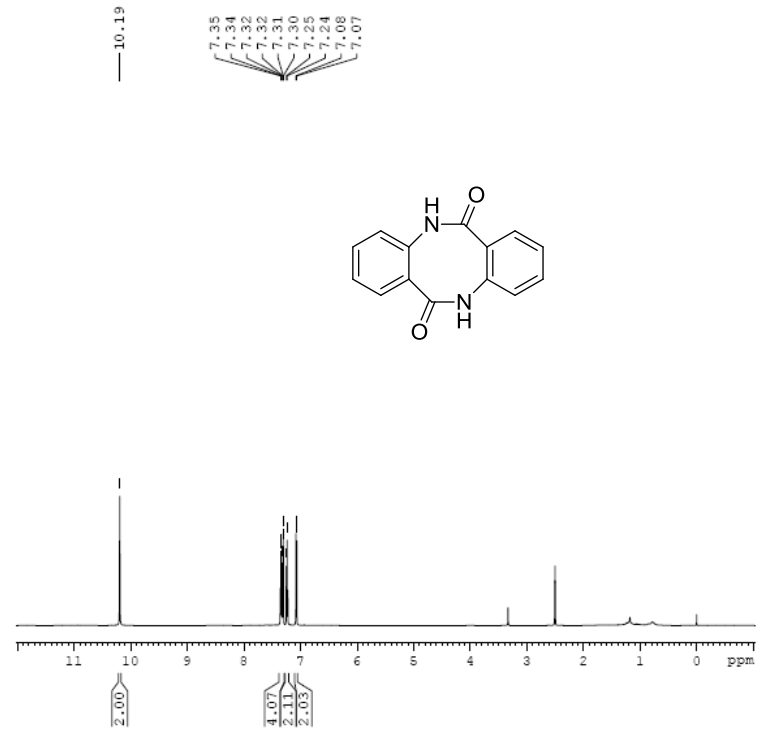


Fig. S6. FET characteristic and output curves of DBPP at various gate voltages.

ql01-100-3-2;1HNMR; DMSO



BRUKER

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PULPROG                      zg30
TD                          32768
SOLVENT                      DMSO
NS                              4
DS                              2
SWH                     12019.230 Hz
FIDRES                   0.366798 Hz
AQ                       1.3631988 sec
RG                           12.7
DW                         41.600 usec
DE                          6.50 usec
TE                         298.0 K
D1                      1.00000000 sec
TD0                             1

======== CHANNEL f1 ========
NUC1                           1H
P1                           7.80 usec
PL1                          3.85 dB
PL1W                 5.83820200 W
SFO1                600.1336008 MHz
SI                         65536
SF                  600.1300039 MHz
WDW                           EM
SSB                            0
LB                          0.30 Hz
GB                             0
PC                          1.00
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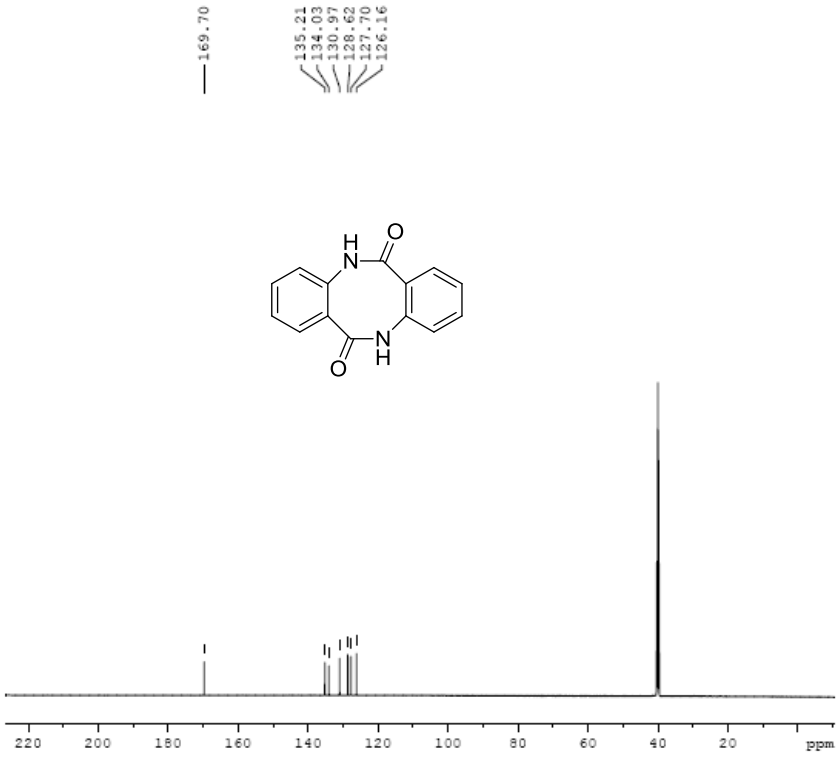
ql01-100-3-2;1HNMR; DMSO



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SOLVENT                      DMSO
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FIDRES                   0.544957 H
AQ                       0.9175540 s
RG                             57
DW                         14.000 u
DE                          14.00 u
TE                         298.0 K
D1                      2.00000000 s
D11                     0.03000000 s
TD0                            16

======== CHANNEL f1 =====
NUC1                           13C
P1                          11.80 u
PL1                         -0.60 d
PL1W                97.76866913 W
SFO1                150.9191065 M

======== CHANNEL f2 =====
CPDPRG2                   waltz16
NUC2                           1H
PCPD2                      80.00 u
PL2                         7.55 d
PL12                       24.00 d
PL13                       24.00 d
PL2W                 2.49045730 W
PL12W                0.05640000 W
PL13W                0.05640000 W
SFO2                600.1330607 M
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WDW                           EM
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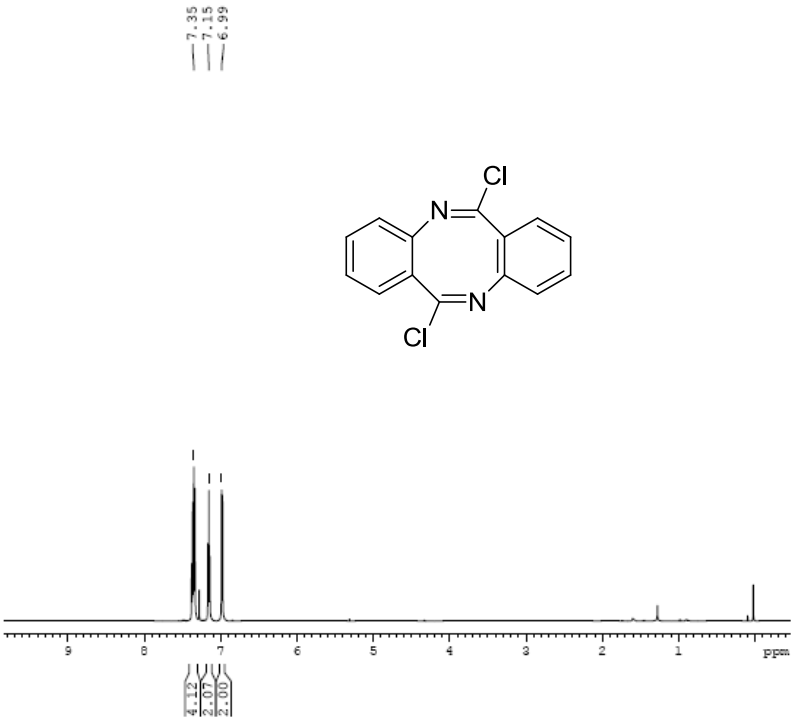
q101-96;1HNMR; CDCl3

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SOLVENT             CDCl3
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DS                       2
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FIDRES            0.366798 Hz
AQ                1.3631988 sec
RG                    20.2
DW                  41.600 usec
DE                   6.50 usec
TE                  298.0 K
D1                1.00000000 sec
TD0                      1

======== CHANNEL f1 ========
NUC1                    1H
P1                    7.80 usec
PL1                   3.85 dB
PL1W           5.83820200 W
SFO1          600.1336008 MHz
SI                   65536
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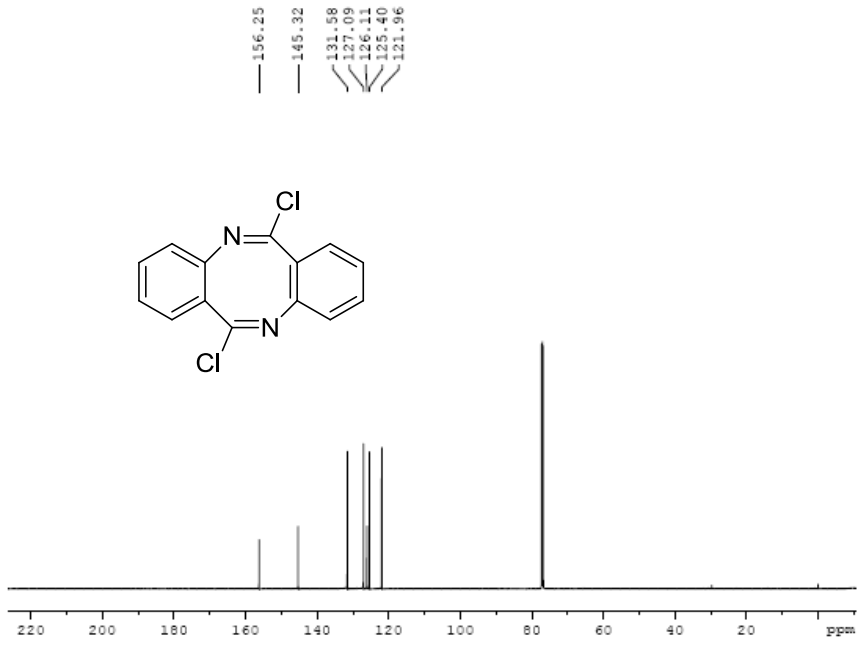
q101-96;13CNMR; CDCl3

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FIDRES            0.544957 H
AQ                0.9175540 s
RG                      57
DW                  14.000 u
DE                   14.00 u
TE                   298.0 K
D1                2.00000000 s
D11               0.03000000 s
TD0                     16

======== CHANNEL f1 =====
NUC1                   13C
P1                   11.80 u
PL1                  -0.60 d
PL1W          97.76866913 W
SFO1         150.9191065 M

======== CHANNEL f2 =====
CPDPRG2           waltz16
NUC2                    1H
PCPD2                80.00 u
PL2                   7.55 d
PL12                 24.00 d
PL13                 24.00 d
PL2W          2.49045730 W
PL12W         0.05640000 W
PL13W         0.05640000 W
SFO2         600.1322205 M
SI                  131072
SF           150.9028090 M
WDW                     EM
SSB                      0
LB                    1.00 H
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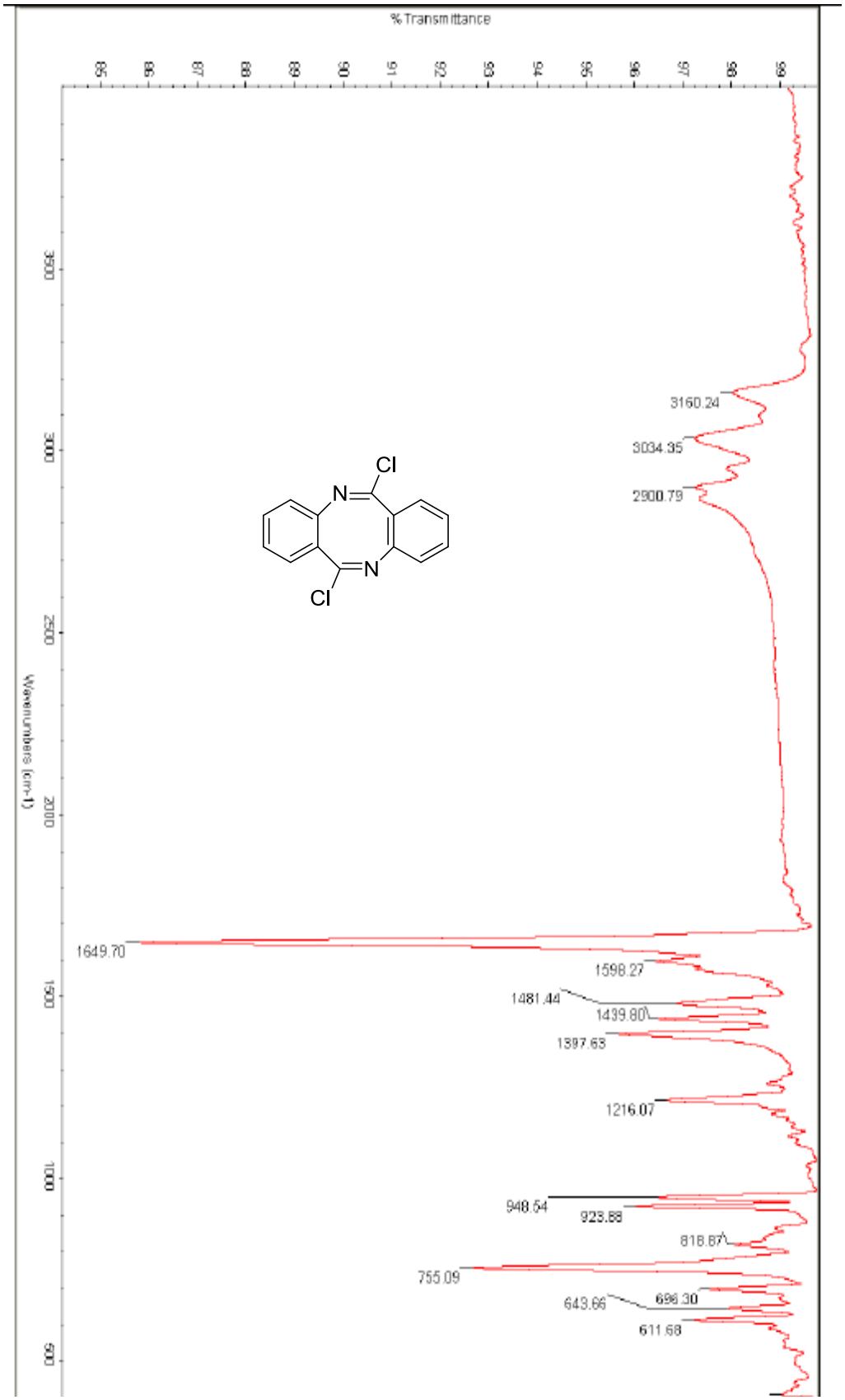

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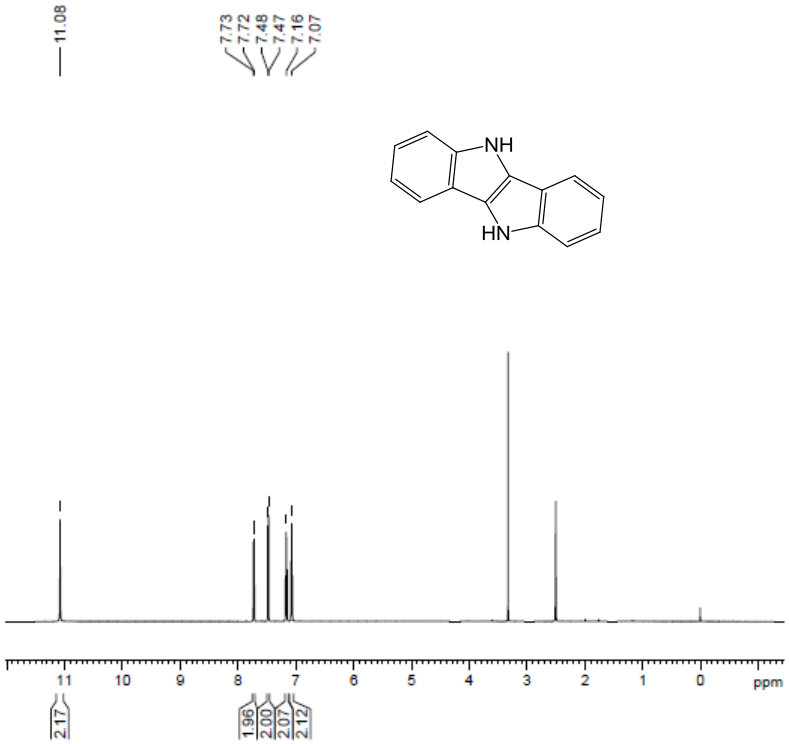


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TD                        32768
SOLVENT                    DMSO
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FIDRES               0.366798 Hz
AQ                   1.3631988 sec
RG                           16
DW                      41.600 usec
DE                       6.50 usec
TE                      297.9 K
D1                   1.00000000 sec
TD0                           1

======== CHANNEL f1 ========
NUC1                         1H
P1                        7.80 usec
PL1                       3.85 dB
PL1W                5.83820200 W
SFO1               600.1336008 MHz
SI                        65536
SF                 600.1300039 MHz
WDW                          EM
SSB                           0
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GB                            0
PC                         1.00
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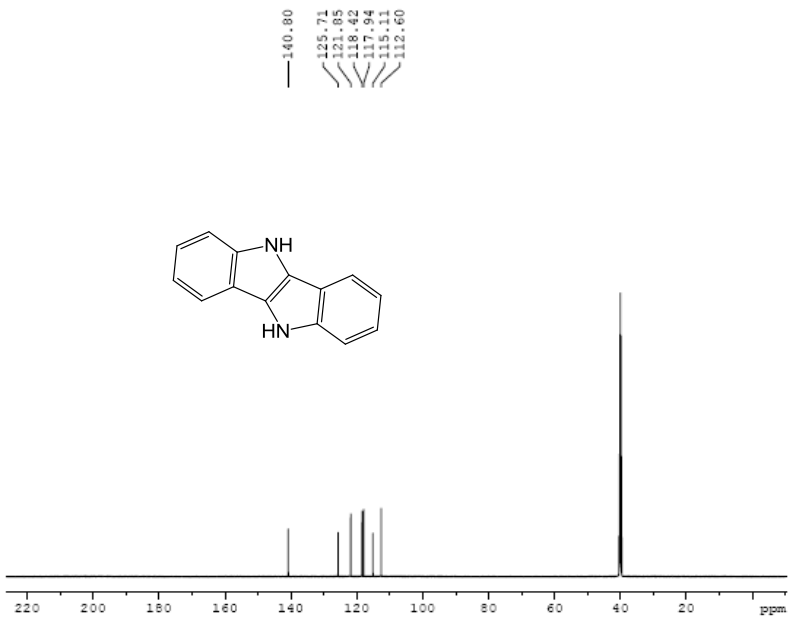


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TD                        65536
SOLVENT                    DMSO
NS                          344
DS                            4
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FIDRES               0.544957 H
AQ                   0.9175540 s
RG                           57
DW                      14.000 u
DE                      14.00 u
TE                      298.0 K
D1                   2.00000000 s
D11                  0.03000000 s
TD0                          16

======== CHANNEL f1 =====
NUC1                        13C
P1                       11.80 u
PL1                      -0.60 d
PL1W               97.76866913 W
SFO1              150.9191065 M

======== CHANNEL f2 =====
CPDPRG2                waltz16
NUC2                         1H
PCPD2                    80.00 u
PL2                       7.55 d
PL12                     24.00 d
PL13                     24.00 d
PL2W               2.49045730 W
PL12W              0.05640000 W
PL13W              0.05640000 W
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SI                       131072
SF                150.9028090 M
WDW                          EM
SSB                           0
LB                         1.00 H
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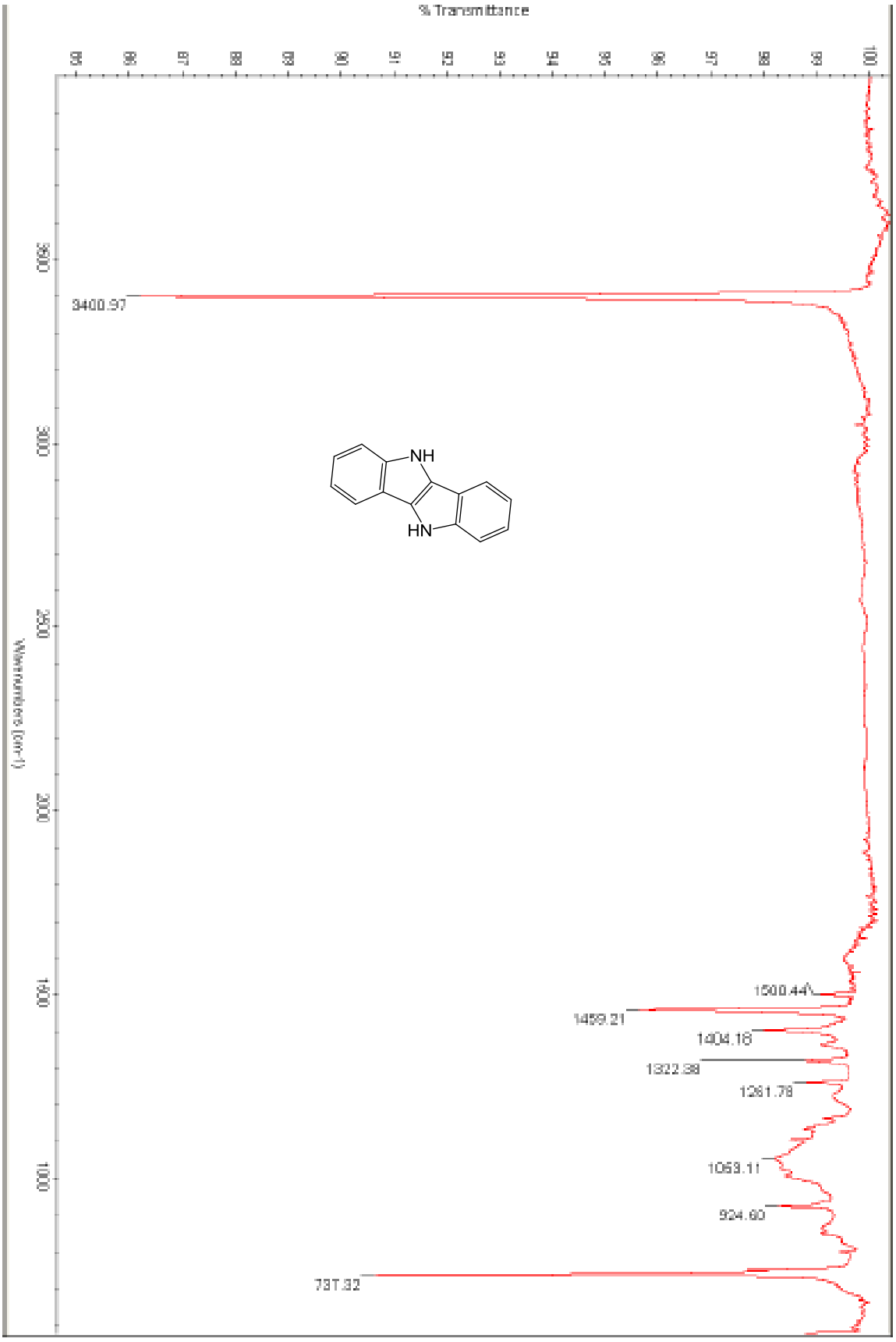
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1322.38
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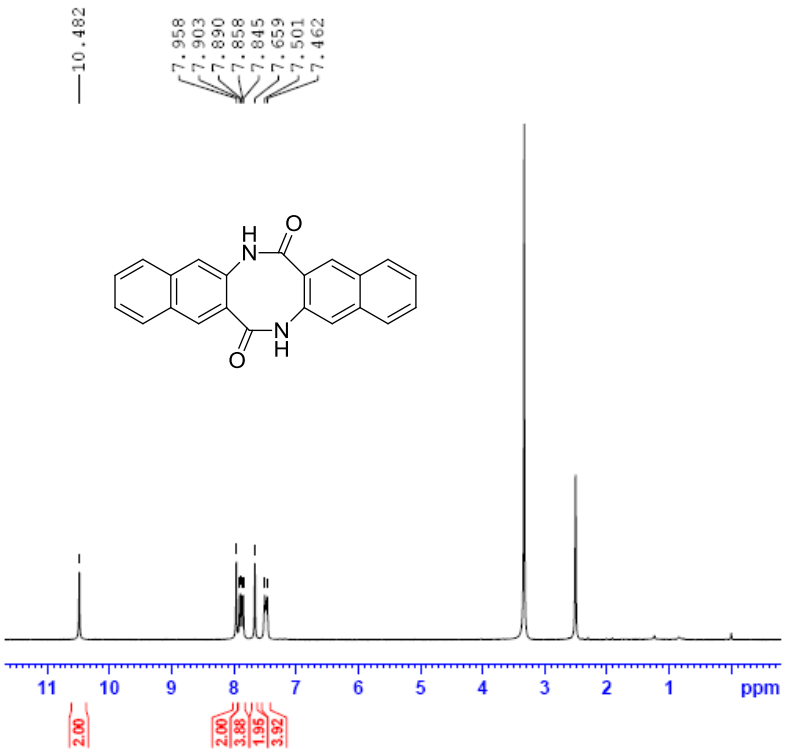
737.32
NH
HN

ql02-51-6;1HNMR;DMSO



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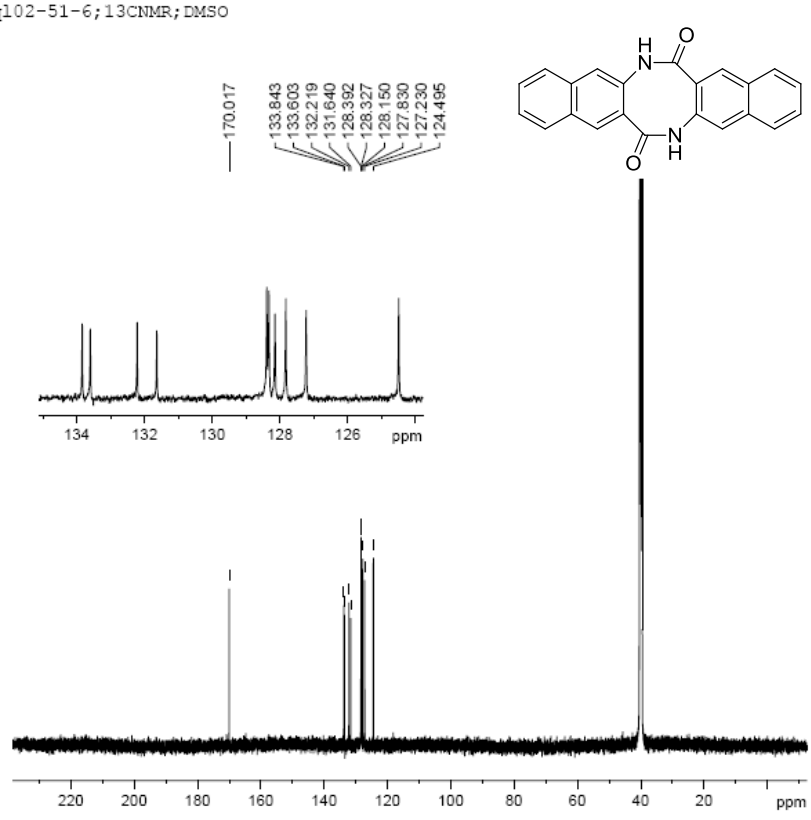
AQ 1.3631988 sec
RG 50.8
DW 41.600 usec
DE 6.50 usec
TE 298.0 K
D1 1.00000000 sec
TD0 1

======== CHANNEL f1 ========
NUC1 1H
P1 7.80 usec
PL1 3.85 dB
PL1W 5.83820200 W
SFO1 600.1336008 MHz
SI 65536
SF 600.1300039 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00

ql02-51-6;13CNMR;DMSO



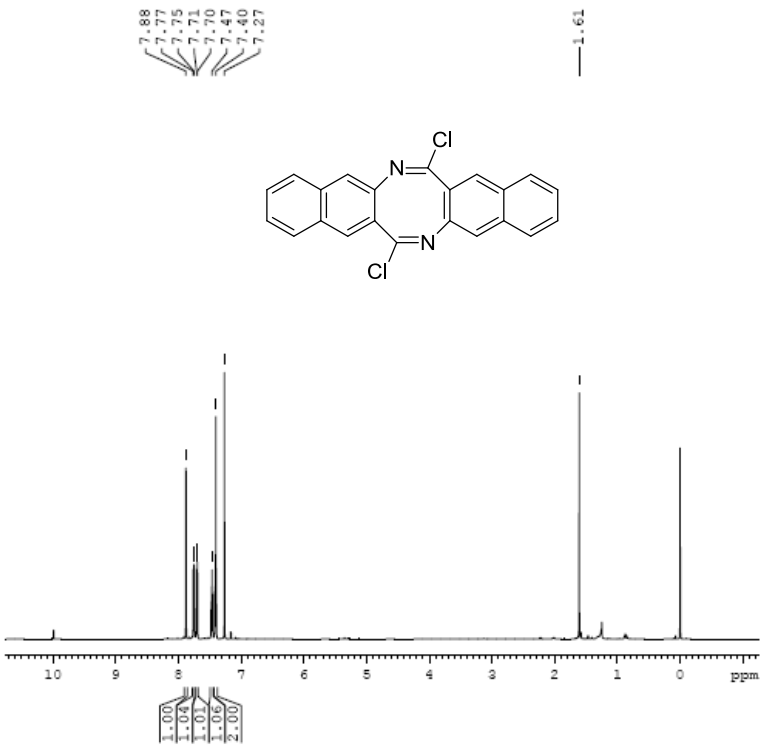
BRUKER

NAME Jul18-2012 ql02-51-6
EXPNO 2
PROCNO 1
Date_ 20120718
Time 11.10
INSTRUM spect
PROBHD 5 mm CPTCI 1H-
PULPROG zgpg30
TD 65536
SOLVENT DMSO
NS 910
DS 4
SWH 37878.789 Hz
FIDRES 0.577984 Hz
AQ 0.8651252 sec
RG 57
DW 13.200 usec
DE 14.00 usec
TE 298.0 K
D1 2.00000000 sec
D11 0.03000000 sec
TD0 16

======== CHANNEL f1 ========
NUC1 13C
P1 13.60 usec
PL1 -0.60 dB
PL1W 97.76866913 W
SFO1 150.9198610 MHz

======== CHANNEL f2 ========
CPDPRG2 waltz16
NUC2 1H
PCPD2 80.00 usec
PL2 7.55 dB
PL12 24.00 dB
PL13 24.00 dB
PL2W 2.49045730 W
PL12W 0.05640000 W
PL13W 0.05640000 W
SFO2 600.1331207 MHz
SI 131072
SF 150.9028090 MHz
WDW EM
SSB 0
LB 1.00 Hz

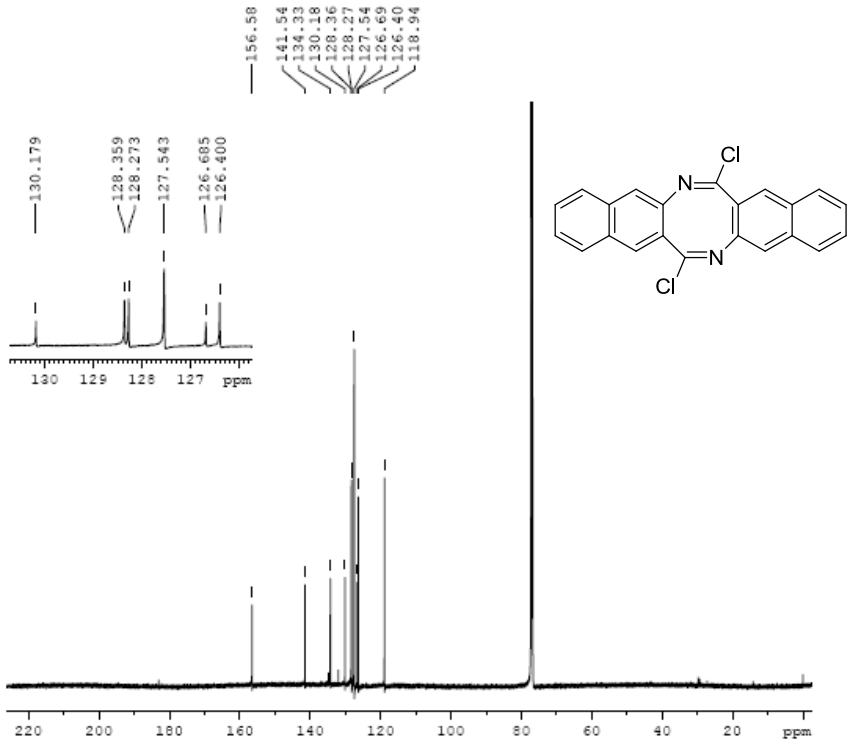
ql01-114;1HNMR;CDCl3




```
NAME          20120316 ql01-114
EXPNO                        1
PROCNO                       1
Date_                 20120316
Time                     16.23
INSTRUM                  spect
PROBHD    5 mm CPTCI 1H-
PULPROG                  zg30
TD                      32768
SOLVENT                 CDCl3
NS                        128
DS                          2
SWH                 12019.230 Hz
FIDRES                0.366798 Hz
AQ                    1.3631988 sec
RG                         25.4
DW                       41.600 usec
DE                        6.50 usec
TE                       280.0 K
D1                    1.00000000 sec
TD0                          1

======== CHANNEL f1 ========
NUC1                        1H
P1                        7.80 usec
PL1                       3.85 dB
PL1W                5.83820200 W
SFO1               600.1336008 MHz
SI                        65536
SF                 600.1300129 MHz
WDW                          EM
SSB                           0
LB                         0.30 Hz
GB                            0
PC                         1.00
```

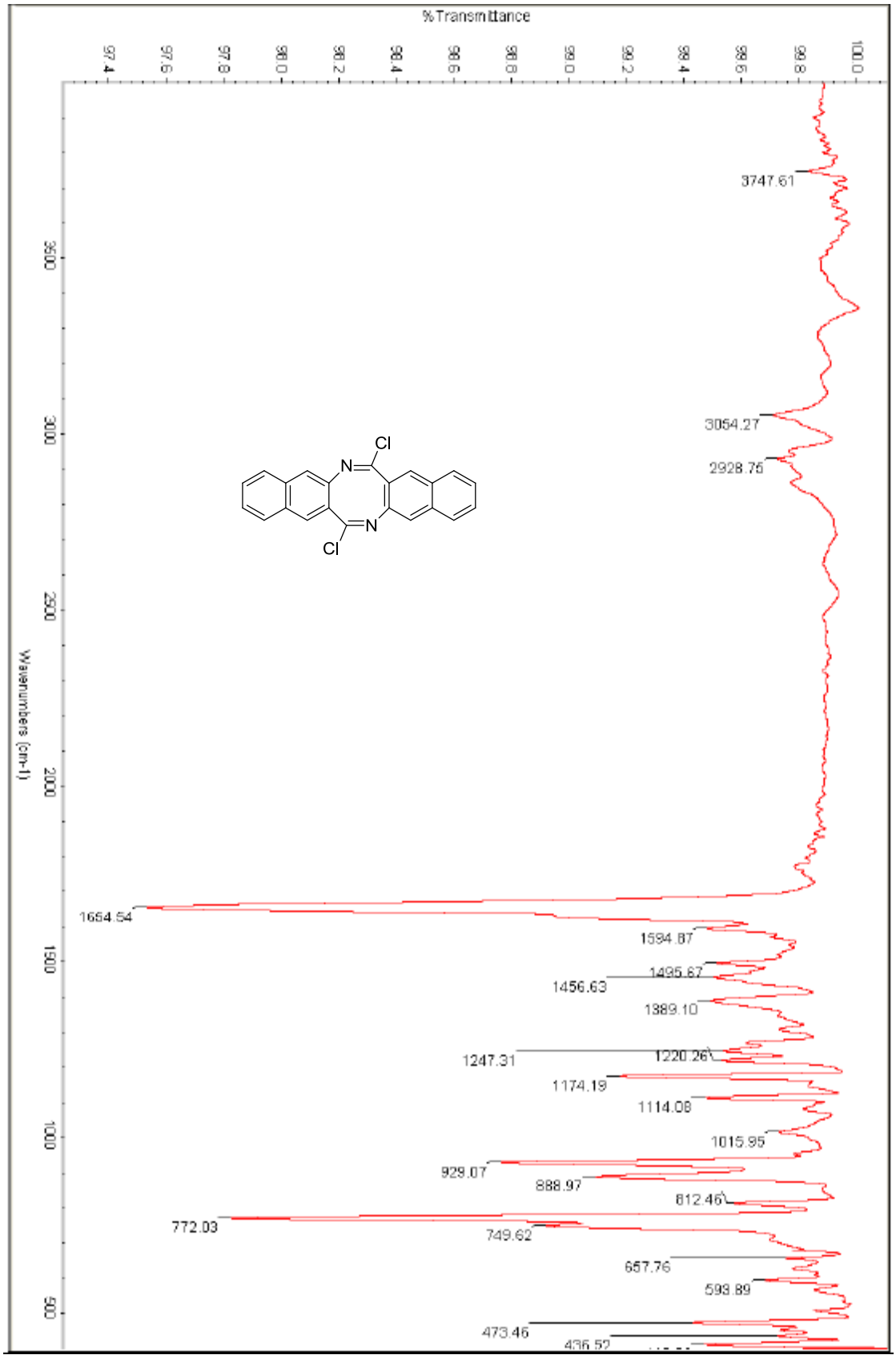
ql01-114;13CNMR;CDCl3



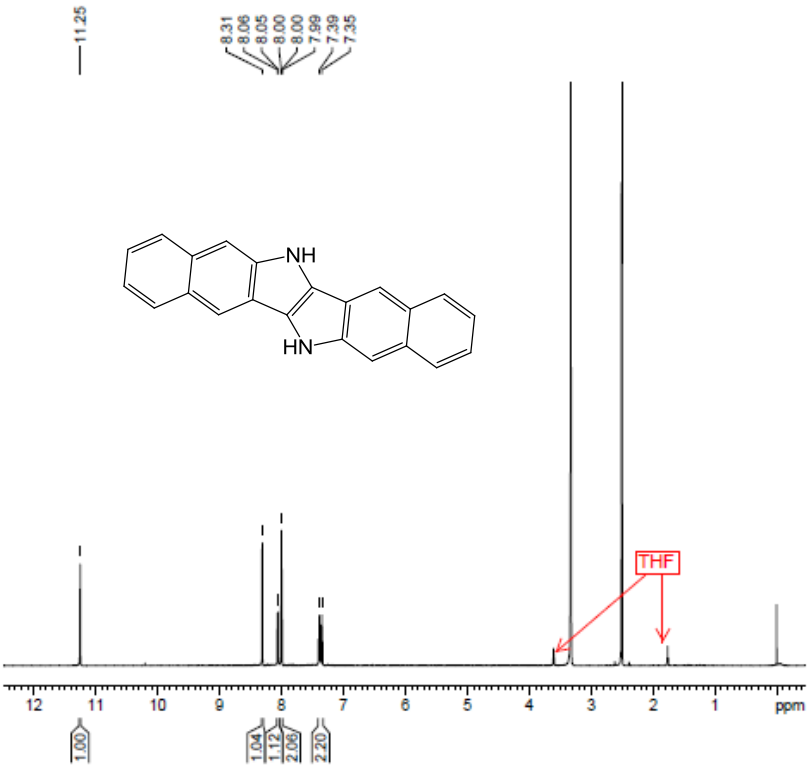

```
NAME          20120320 ql01-11
EXPNO                        2
PROCNO                       1
Date_                 20120320
Time                     10.31
INSTRUM                  spect
PROBHD    5 mm CPTCI 1H-
PULPROG                zgpg30
TD                      65536
SOLVENT                 CDCl3
NS                       3921
DS                          4
SWH                 35714.285 H
FIDRES                0.544957 H
AQ                    0.9175540 s
RG                         101
DW                       14.000 u
DE                       14.00 u
TE                       297.9 K
D1                    2.00000000 s
D11                   0.03000000 s
TD0                          16

======== CHANNEL f1 =====
NUC1                       13C
P1                       11.80 u
PL1                      -0.60 d
PL1W                97.76866913 W
SFO1               150.9191065 M

======== CHANNEL f2 =====
CPDPRG2                waltz16
NUC2                        1H
PCPD2                    80.00 u
PL2                       7.55 d
PL12                     24.00 d
PL13                     24.00 d
PL2W                2.49045730 W
PL12W               0.05640000 W
PL13W               0.05640000 W
SFO2               600.1330006 M
SI                       131072
SF                 150.9028090 M
WDW                          EM
SSB                           0
LB                         1.00 H
```


%Transmittance
Wavenumbers (cm-1)
3747.61
3054.27
2928.75
1654.54
1594.87
1495.67
1456.63
1389.10
1247.31
1220.26
1174.19
1114.08
1015.95
929.07
888.97
812.46
772.03
749.62
657.76
593.89
473.46
436.52
Cl
N
N
Cl

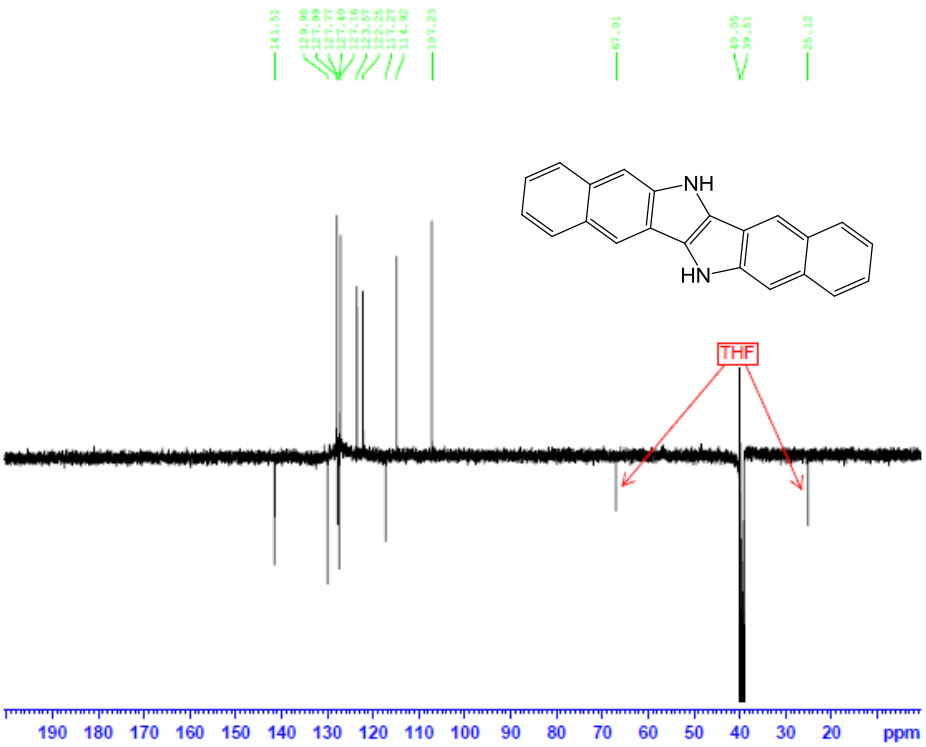
q102-31-2;1HNMR;DMSO




```
NAME        20120619 q102-31-2
EXPNO                        1
PROCNO                       1
Date_               20120619
Time                    17.40
INSTRUM                 spect
PROBHD    5 mm CPTCI 1H-
PULPROG                 zg30
TD                     32768
SOLVENT                 DMSO
NS                         4
DS                         2
SWH                12019.230 Hz
FIDRES              0.366798 Hz
AQ                  1.3631988 sec
RG                        64
DW                     41.600 usec
DE                      6.50 usec
TE                    298.0 K
D1                 1.00000000 sec
TD0                        1

======== CHANNEL f1 ========
NUC1                      1H
P1                     7.80 usec
PL1                    3.85 dB
PL1W               5.83820200 W
SFO1             600.1336008 MHz
SI                       65536
SF               600.1300039 MHz
WDW                       EM
SSB                        0
LB                      0.30 Hz
GB                         0
PC                      1.00
```

q102-31;DEPTQ;DSMO



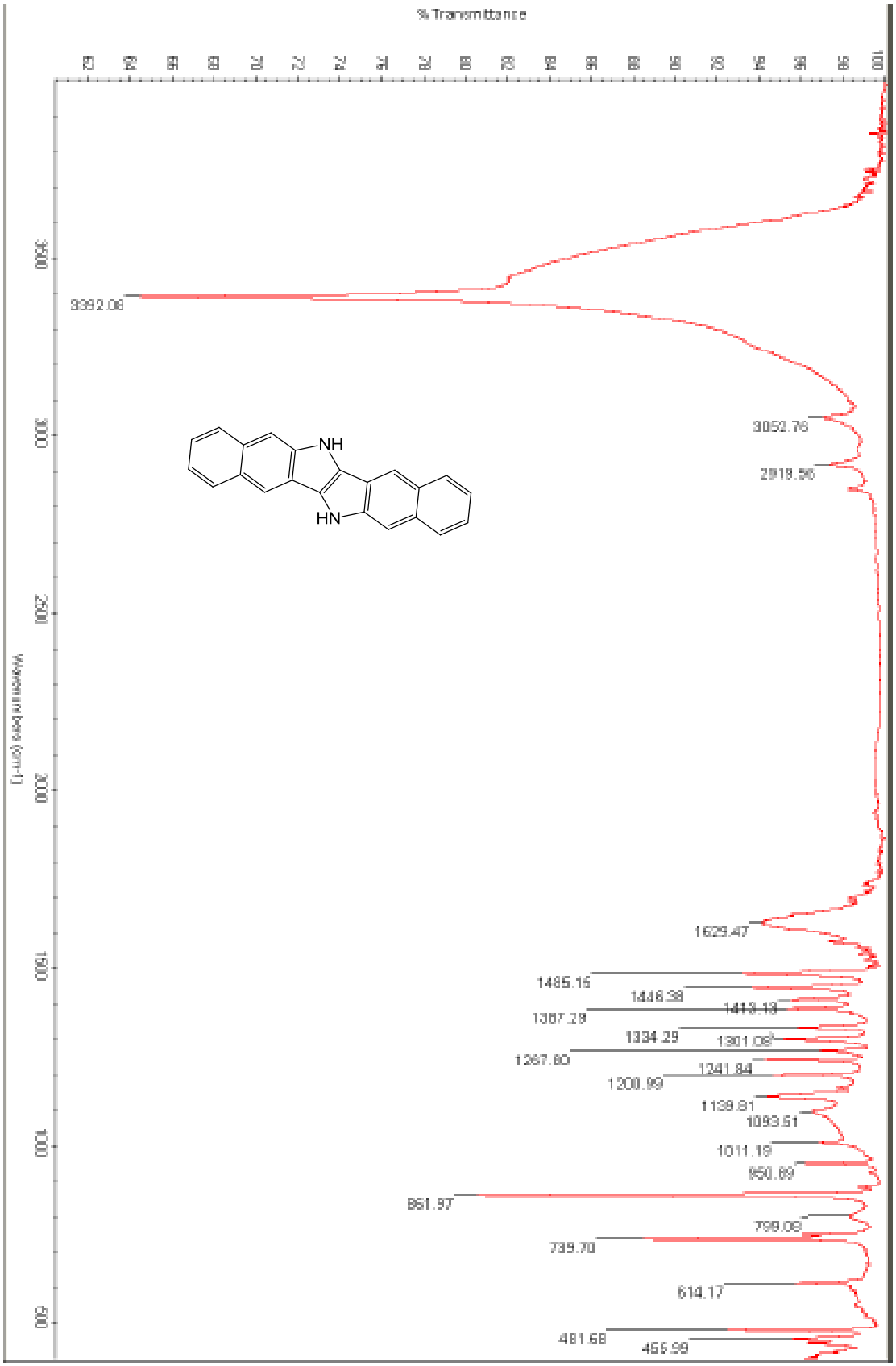

```
Current Data Parameters
NAME      20120508 q102-31
EXPNO                     1
PROCNO                    1

F2 - Acquisition Parameters
Date_            20120508
Time                11.36
INSTRUM             spect
PROBHD   5 mm CPTCI 1H-
PULPROG          deptqsp
TD                 65536
SOLVENT             DMSO
NS                  3525
DS                     8
SWH             37878.788 Hz
FIDRES          0.577984 Hz
AQ              0.8653152 sec
RG                   203
DW                13.200 usec
DE                 6.50 usec
TE                 296.0 K
CNST2         145.0000000
CNST12          1.5000000
D1              2.00000000 sec
D2              0.00344828 sec
D12             0.00002000 sec
TD0                   16
SGOPTNS

======== CHANNEL f1 ========
NUC1                  13C
P1                 13.60 usec
P12              2000.00 usec
PL0               120.00 dB
PL1                -0.60 dB
PLSW          -1.#IND0000 W
PLIW          -1.#IND0000 W
SFO1          150.9178988 MHz
SP2                4.89 dB
SPNAM2      Crp60comp.4
SPOAL2            0.500
SPOFFS2       0 Hz

======== CHANNEL f2 ========
CPDPRG2          waltz16
NUC2                  1H
P0                 18.00 usec
P3                 12.00 usec
P4                 24.00 usec
PCPD2              85.00 usec
PL2                 7.55 dB
PL12               24.00 dB
PL2W          -1.#IND0000 W
PL12W         -1.#IND0000 W
SFO2          600.1330006 MHz

F2 - Processing parameters
SI                   32768
SF            150.9028813 MHz
WDW                     EM
SSB       0
LB                  1.00 Hz
GB        0
PC                  1.40
```


% Transmittance
Wavenumbers (cm-1)
3392.08
3052.76
2918.56
1629.47
1485.16
1448.58
1413.13
1387.29
1334.29
1301.08
1267.80
1241.84
1200.99
1139.81
1093.51
1011.19
950.89
861.97
799.09
739.70
614.17
481.68
455.99
NH
HN