Supplementary Material (ESI) for Chemical Communications

Supporting Information

Synthesis and structure of nitrogen bridged calix[5]- and -[10]-pyridines and their complexation with fullerenes

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Preparation of 3. To a solution of 2,6-bis(methylamino)pyridine **1** (1.37 g, 10 mmol), 2,6-dibromopyridine **2** (4.47 g, 20 mmol) in dry THF (100 mL) at room temperature was added rapidly KOBu^t (3.36 g, 30 mmol). An exothermal reaction took place immediately and the mixture became a dark red solution. About 10 min later, the solution was heated to reflux. After 2 h, the reaction mixture was cooled down to room temperature and water (10 mL) was added. The precipitate was filtrated off and washed with ethyl acetate. The filtrate was concentrated under vacuum to remove THF. The residue was dissolved in water (30 mL) and extracted with ethyl acetate (3×20 mL). After dry over with anhydrous Mg_2SO_4 and concentration under vacuum, the residue was chromatographed on a silica gel column to give compound **3** (3.91 g, 75%).

Preparation of 5. To a solution of 2,6-bis(methylamino)pyridine **1** (1.37 g, 10 mmol), 2,6-dibromopyridine **2** (2.25 g, 9.5 mmol) in dry THF (30 mL) at 30 ^{o}C was added drop-wise a solution of KOBu^t (2.22 g, 20 mmol) in dry THF (10 mL). The resulting mixture was kept stirring at 50 ^{o}C for 1 h, and then the reaction was quenched by cooling to room temperature and by adding water (10 mL). The mixture was concentrated under vacuum to remove THF and the residue was dissolved in water

(20 mL) and extracted with ethyl acetate (3×20 mL). After dry over with anhydrous Mg_2SO_4 and concentration under vacuum, the residue was chromatographed on a silica gel column using a mixture of ethyl acetate and petroleum ether (1:5) as an eluent to give compound **4** (1.032 g, 71%): mp 91-92 ^oC; IR (KBr) 3384, 3265, 1607, 1573 $cm^{-1;}$ ^1H NMR ($CDCl_3$, 300 MHz) δ 7.39 (t, *J* = 7.9 Hz, 1H), 7.22 (t, *J* = 7.9 Hz, 1H), 7.12 (d, *J* =7.4 Hz, 1H), 6.87 (d, *J* = 8.3 Hz, 1H), 6.43 (d, *J* = 8.3 Hz, 1H), 6.07 (d, *J* = 7.3 Hz, 1H), 4.50 (s, 1H), 3.53 (s,3H), 2.88 (d, *J* = 5.1 Hz, 3H); ^{13}C NMR ($CDCl_3$, 75MHz) δ 158.9, 157.8, 156.2, 139.5, 139.1, 138.2, 118.3, 111.4, 103.9, 100.4, 35.9, 29.1. $C_{12}H_{13}N_4Br$ requires C, 49.16; H, 4.47; N, 19.11. Found: C, 49.21; H, 4.58; N, 18.94. A mixture of **4** (4.5 g, 4.5 g, 15 mmol) and $CuSO_4.5H_2O$ (0.2, 0.8 mmol) in aqueous methylamine solution (25-30%, 40 mL) was heated at 150 ^oC in a sealed tube for 15 h. After colling to room temperature, the mixture was added into a saturated NaOH solution in water (20 mL) and product **5** precipitated. Filtration, wash with water, and dry under vacuum gave pure **5** (3.235 g, 87%): mp 108-110 ^oC; IR (KBr) 3298, 3177, 1602, 1571 cm^{-1}; ^1H NMR ($CDCl_3$, 300 MHz) δ 7.29 (t, *J* = 7.8 Hz, 2H), 6.47 (d, *J* = 7.9 Hz, 2H), 5.93 (d, *J* = 7.9 Hz, 2H), 4.36 (br., s, 2H), 3.51 (s, 3H), 2.89 (d, *J* = 5.1 Hz, 6H); ^{13}C NMR ($CDCl_3$, 75MHz) δ 158.8, 157.1, 138.3, 103.1, 97.6, 35.5, 29.2; MS (ESI) *m/z* 244 ($M+H^+$). $C_{13}H_{17}N_5$ requires C, 64.17; H, 7.04; N, 28.78. Found: C, 64.17; H 7.04; N 28.53.

Synthesis of azacalix[5]pyridine 6 and azacalix[10]pyridine 7. Under argon protection, a mixture of **3** (224 mg, 0.5 mmol), **5** (122 mg, 0.5 mmol), $KOBu^t$ (144 mg, 1.5 mmol), $Pd_2(dba)_3$ (92 mg, 0.1 mmol), dppp (82 mg, 0.2 mmol) in dry

1,4-dioxane (200 mL) was heated rapid to reflux. After refluxing for 4 h, the mixture was cooled to room temperature, and water (10 mL) was added. The solid in the mixture was filtrated off through Celite. The filtrate was concentrated to give a residue (5 mL) which was dissolved in water (50 mL) and extracted with dichloromethane (3×30 mL). The combined organic phase was washed with brine (50 mL). The aqueous phase was extracted again with dichloromethane (30 mL). Combined organic phase was dried over with anhydrous Mg_2SO_4. After the solvent was removed under vacuum, the residue was chromatographed on a silica gel column eluted with a mixture of ethyl acetate and petroleum ether (1:3), pure ethyl acetate and then a mixture of ethyl acetate, methanol and ammonium (2000:100:1) to give products **6** and **7** (Table 1). Azacalix[5]pyridine **6** (62 mg, 23%): mp 225-227 ^{o}C; IR (KBr) 1739 (w) 1587 (sh) 1566 cm^{-1}; ^{1}H NMR ($CDCl_3$, 300 MHz) δ 7.36 (t, 1H J = 8.0 Hz), 6.74 (d, 2H, J = 8.0 Hz), 3.53 (s, 3H); ^{13}C NMR (CDCl3, 75MHz) δ 156.2, 137.8, 106.7, 35.8; MS (MALDI-TOF) *m/z* 531(M+H$^+$), 553 (M+Na$^+$), 569 (M+K$^+$). $C_{30}H_{30}N_{10}$ requires: C,67.90; H, 5.70; N, 26.40. Found: C, 67.85; H, 5.72; N, 26.16. Azacalix[10]pyridine **7** (91 mg, 34.3%): mp 188-190 ^{o}C; IR (KBr) 1591, 1563 cm^{-1}; ^{1}H NMR ($CDCl_3$, 300MHz) δ 7.39 (t, 1H, J = 8.0 Hz), 6.78 (d, 2H, J = 8.0 Hz), 3.59 (s, 3H); ^{13}C NMR ($CDCl_3$, 75MHz) δ 156.1, 137.8, 106.6, 35.8; MS (MALDI-TOF) *m/z* 1060 (M$^+$). $C_{60}H_{60}N_{20}$ requires C, 67.90; H, 5.70; N, 26.40. Found: C, 67.93; H, 5.77; N, 26.22.

Table S1 Macrocyclizative coupling reaction between **3** and **5**[a]

entry	catalyst	ligand	base	**6** (%)[b]	**7** (%)[b]
1	CuI	DMGC[c]	Cs_2CO_3	-	-
2	Cu_2O	oxime[d]	Cs_2CO_3	-	-
3	$Pd(OAc)_2$	dppp	$NaOBu^t$	-	-
4	$Pd_2(dba)_3$	dppf	$NaOBu^t$	7.9	16.2
5	$Pd_2(dba)_3$	dppb	$NaOBu^t$	12.4	18.5
6	$Pd_2(dba)_3$	dppe	$NaOBu^t$	16.2	23.8
7	$Pd_2(dba)_3$	dppp	$NaOBu^t$	23.0	34.3
8[e]	$Pd_2(dba)_3$	dppp	$NaOBu^t$	25.7	18.9
9[e,f]	$Pd_2(dba)_3$	dppp	$NaOBu^t$	30.9	4.5
10[f,g]	$Pd_2(dba)_3$	dppp	$NaOBu^t$	8.5	41.6

[a] A mixture of **3** (1 Mmol), **5** (1 Mmol), catalyst (20%), ligand (40%), base (3 mmol) was refluxed in 1,4-dioxane (mL) for 4 h. [b] Isolated yield. [c] *N*,*N*-dimethylglycine. [d] salicylaldoxime. [e] The concentration of the reactants was halved. [f] The reaction was carried out at 110 °C in *N*,*N*-dimethylacetamide. [g] The concentration of the reactants was increased by 4 folds.

Table S2 Selected bond lengths [Å] and angels [°] of **6**:

N(2)–C(5)	1.391	N(2)–C(6)	1.428
N(2)–C(26)	1.478	N(4)–C(10)	1.400
N(4)–C(11)	1.434	N(4)–C(27)	1.470
N(6)–C(15)	1.423	N(6)–C(16)	1.403
N(6)–C(28)	1.482	N(8)–C(20)	1.399
N(8)–C(21)	1.424	N(8)–C(29)	1.464
N(10)–C(25)	1.404	N(10)–C(30)	1.471
N(10)–C(1)	1.414		
C(5)–N(2)–C(26)	118.1	C(5)–N(2)–C(6)	123.7
C(6)–N(2)–C(26)	116.9	C(10)–N(4)–C(11)	122.0
C(15)–N(6)–C(16)	123.6	C(20)–N(8)–C(21)	118.5
C(25)–N(10)–C(1)	122.4		

Table S3 Selected bond lengths [Å] and angels [°] of **7**:

N(10)–C(29)	1.391	N(10)–C(30)	1.458
N(10)–C(1)	1.408	N(8)–C(25)	1.385
N(8)–C(23)	1.411	N(8)–C(24)	1.454
N(6)–C(19)	1.402	N(6)–C(17)	1.391
N(6)–C(18)	1.451	N(4)–C(13)	1.388
N(4)–C(11)	1.393	N(4)–C(12)	1.437
N(2)–C(7)	1.432	N(2)–C(5)	1.374
N(2)–C(6)	1.422		
C(29)–N(10)–C(30)	117.8	C(1)–N(10)–C(29)	124.2
C(30)–N(10)–C(1)	117.9	C(25)–N(8)–C(23)	123.5
C(19)–N(6)–C(17)	123.6	C(13)–N(4)–C(11)	122.9
C(7)–N(2)–C(5)	120.9		

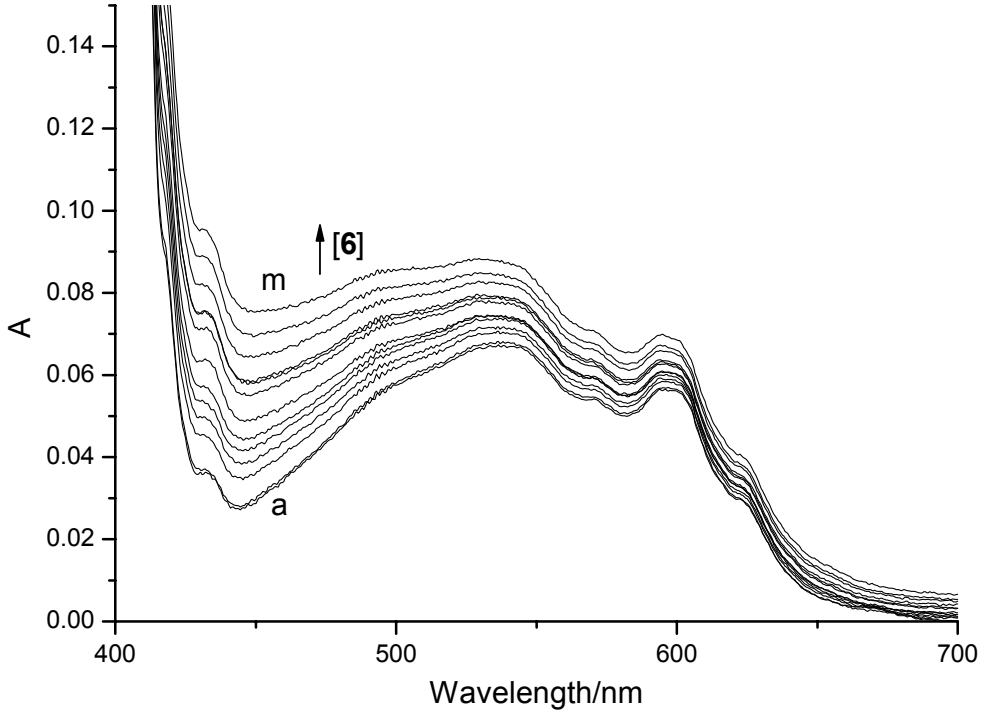


Figure S1. Absorption spectra of C_{60} (7.992×10^{-5} mol dm^{-3}) in the presence of **6** in toluene at 25 ^{o}C. The concentrations of **6** for curves a-m (from bottom to top) are: 0, 0.080, 0.160, 0.240, 0.320, 0.400, 0.480, 0.560, 0.640, 0.720, 0.80, 0.960, 1.12 ($\times10^{-4}$ mol dm^{-3}).

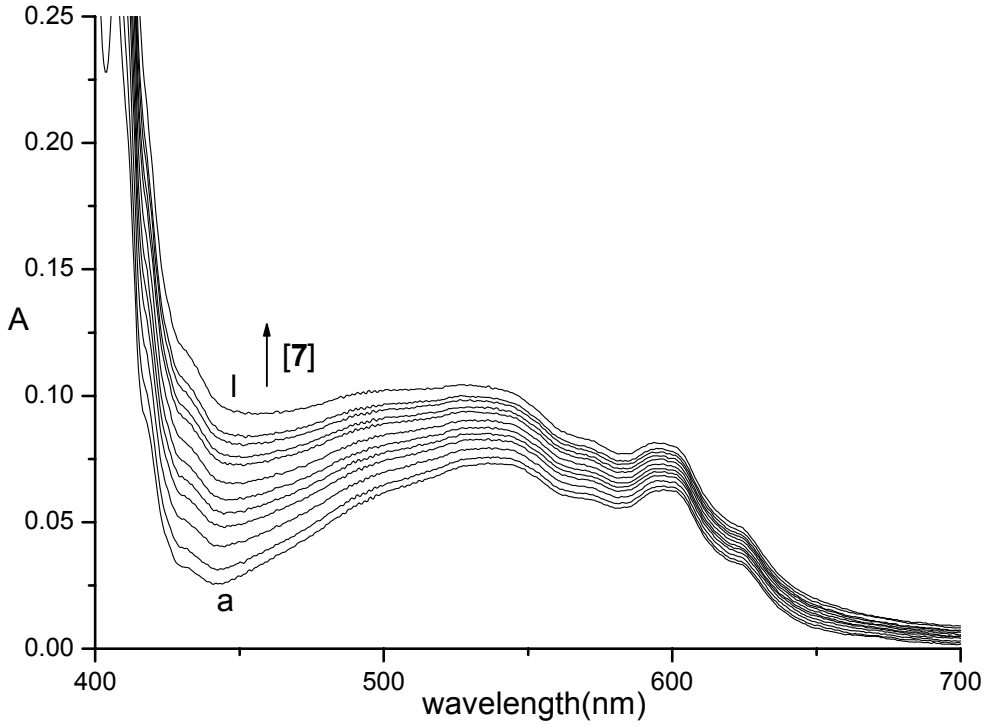


Figure S2. Absorption spectra of C_{60} (7.99×10^{-5} mol dm^{-3}) in the presence of **7** in toluene at 25 ^{o}C. The concentrations of **7** for curves a-l (from bottom to top) are: 0, 0.080, 0.159, 0.240, 0.320, 0.400, 0.480, 0.560, 0.640, 0.720, 0.800, 1.04 ($\times10^{-4}$ mol dm^{-3}).

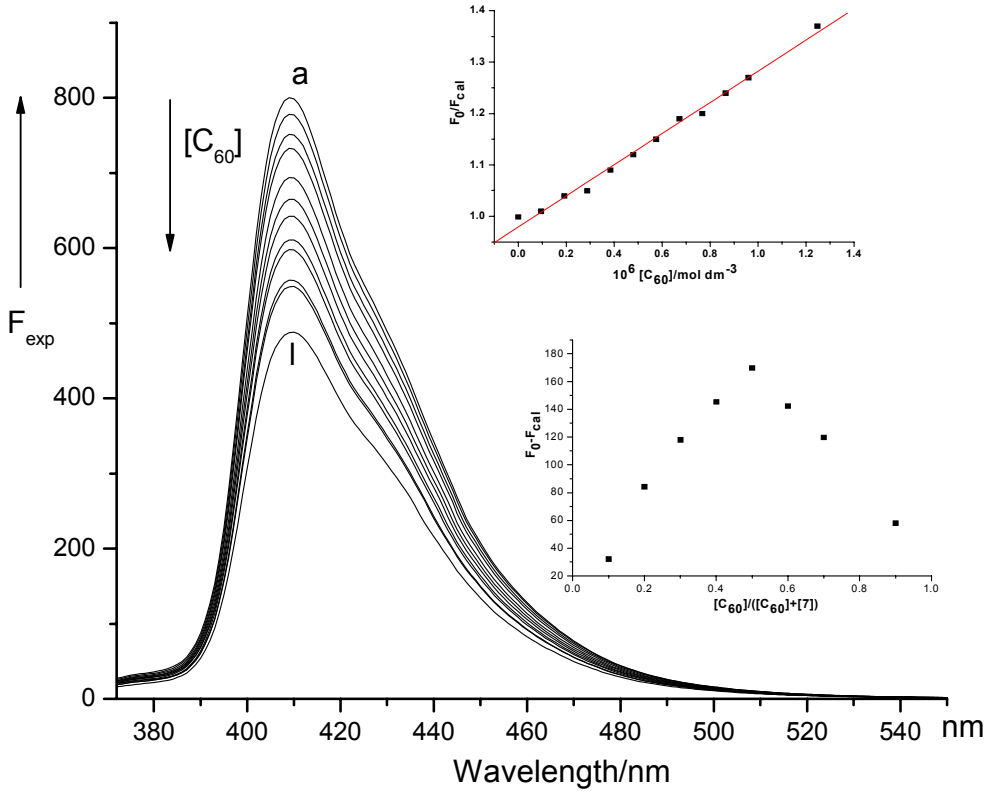


Figure S3. Emission spectra (λ_{ex}=352 nm) of **7** (3.2×10^{-6} mol dm^{-3}) in the presence of C_{60} in toluene at 25°C. The concentrations of C_{60} for curves a-l (from top to bottom) are 0, 0.096, 0.192, 0.288, 0.384, 0.480, 0.576, 0.672, 0.768, 0.864, 0.960, 1.25 ($\times10^{-5}$ mol dm^{-3}). Insets: The up insert is the variation of fluorescence intensity F_0/F_{cal} of **7** with increasing C_{60} concentration. The down inset is the Job plot for **7**-C_{60} complex in toluene solution ([**7**]+[C_{60}]=6.4×10^{-6} mol dm^{-3}).

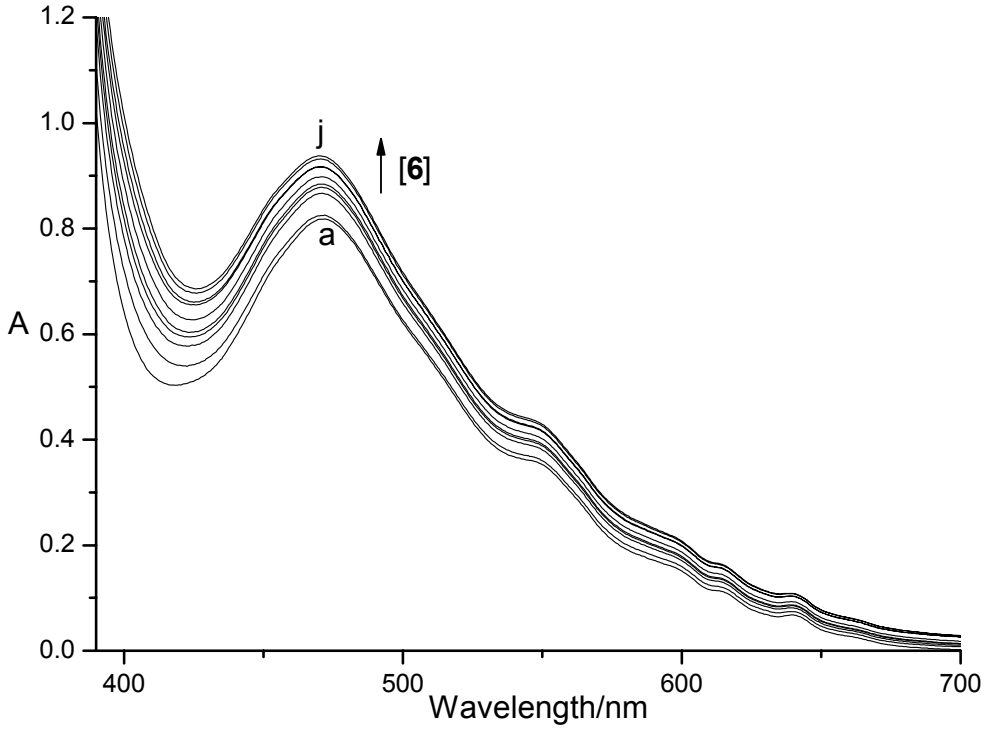


Figure S4. Absorption spectra of C_{70} (4.19×10^{-5} mol dm^{-3}) in the presence of **6** in toluene at 25 ^{o}C. The concentrations of **6** for curves a-j (from bottom to top) are: 0, 0.240, 0.320, 0.400, 0.480, 0.560, 0.640, 0.720, 0.800, 0.880 ($\times10^{-4}$ mol dm^{-3}).

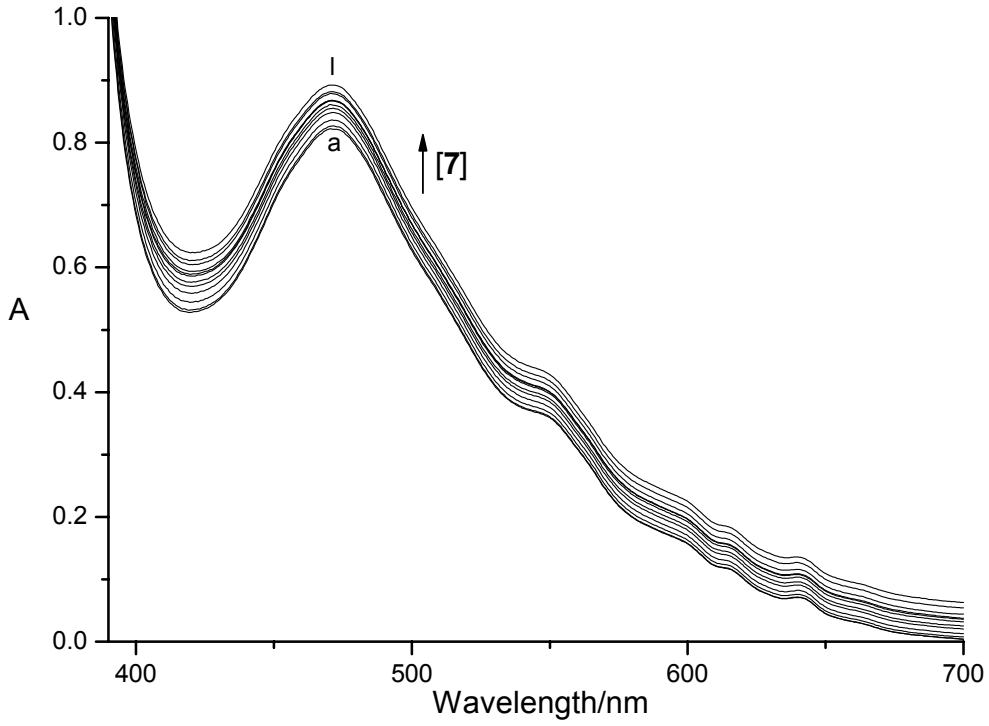


Figure S5. Absorption spectra of C_{70} (4.0×10^{-5} mol dm^{-3}) in the presence of **7** in toluene at 25 ^{o}C. The concentrations of **7** for curves a-l (from bottom to top) are: 0, 0.196, 0.392, 0.588, 0.784, 0.981, 1.18, 1.37, 1.57, 1.76, 1.96, 2.16 ($\times10^{-5}$mol dm^{-3}).

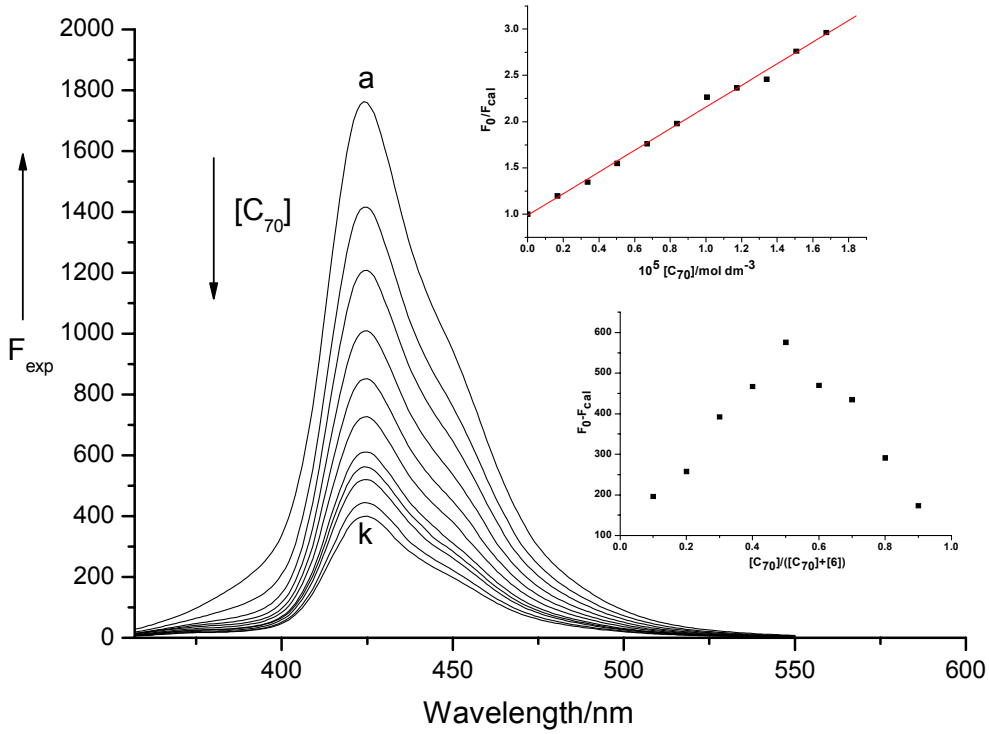


Figure S6. Emission spectra (λ_{ex}= 336 nm) of **6** (3.2×10^{-6} mol dm^{-3}) in the presence of C_{70} in toluene at 25°C. The concentrations of C_{70} for curves a-k (from top to bottom) are 0, 0.167, 0.335, 0.502, 0.670, 0.838, 1.01, 1.17, 1.34, 1.51, 1.68 ($\times10^{-5}$ mol dm^{-3}). Insets: The up inset is the variation of fluorescence intensity F_0/F_{cal} of **6** with increasing C_{70} concentration. The down inset is the Job plot for **6**-C_{70} complex in toluene solution ([**6**]+[C_{70}]=6.4×10^{-6} mol dm^{-3}).

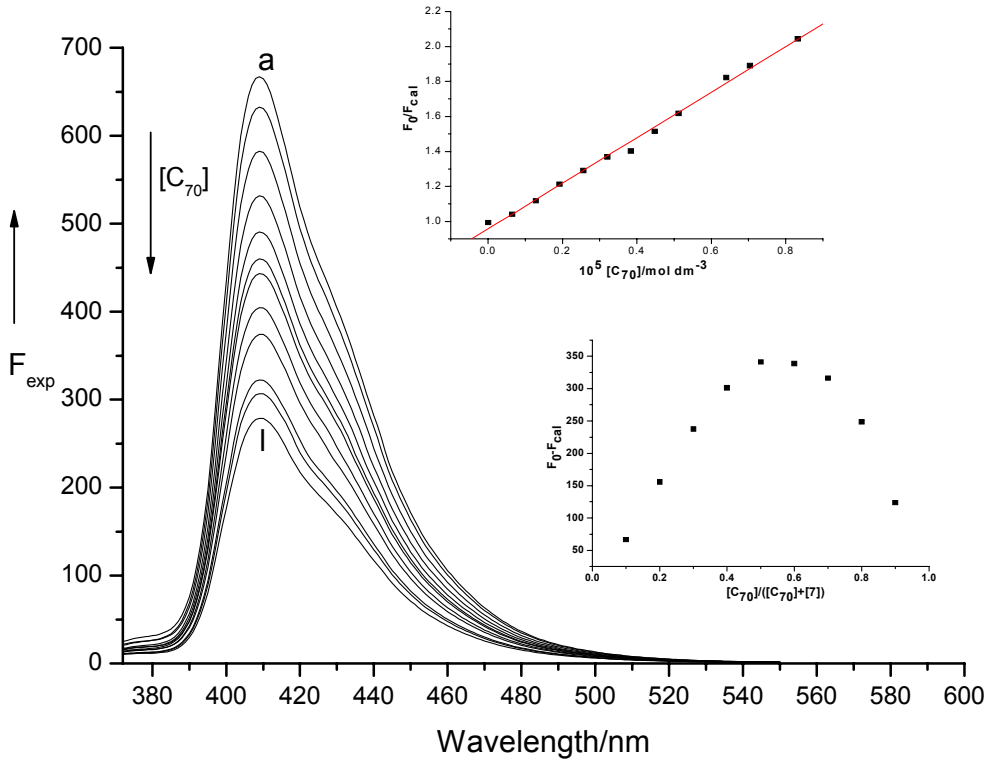
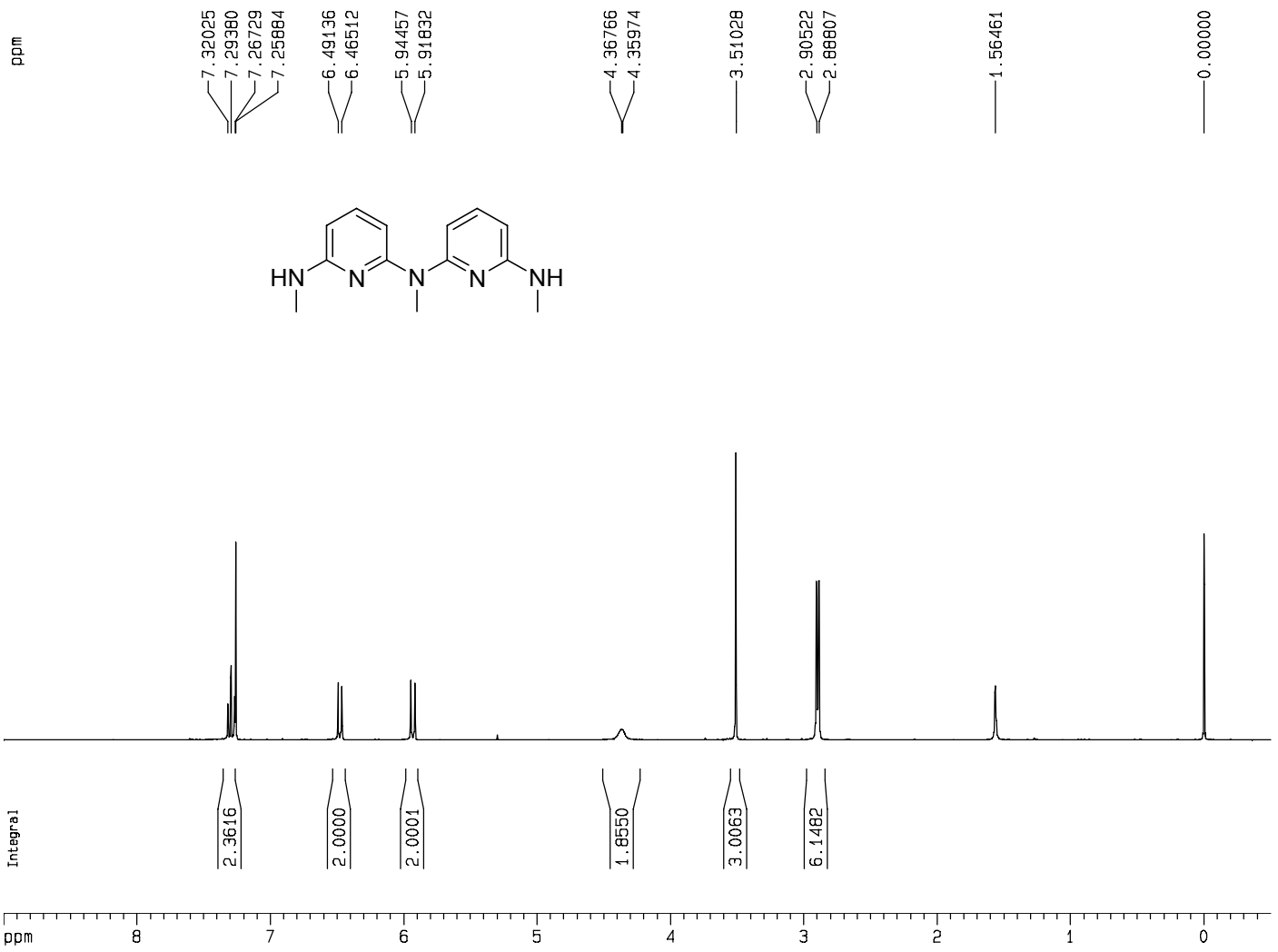


Figure S7. Emission spectra (λ_{ex} = 352nm) of **7** (3.2×10^{-6} mol dm^{-3}) in the presence of C_{70} in toluene at 25°C. The concentrations of C_{70} for curves a-l (from top to bottom) are 0, 0.064, 0.128, 0.192, 0.256, 0.320, 0.384, 0.448, 0.512, 0.640, 0.704, 0.832 ($\times10^{-5}$ mol dm^{-3}). Insets: The up inset is the variation of fluorescence intensity F_0/F_{cal} of **7** with increasing C_{70} concentration. The down inset is the Job plot for **7**-C_{70} complex in toluene solution ([**7**]+[C_{70}]=6.4×10^{-6} mol dm^{-3}).

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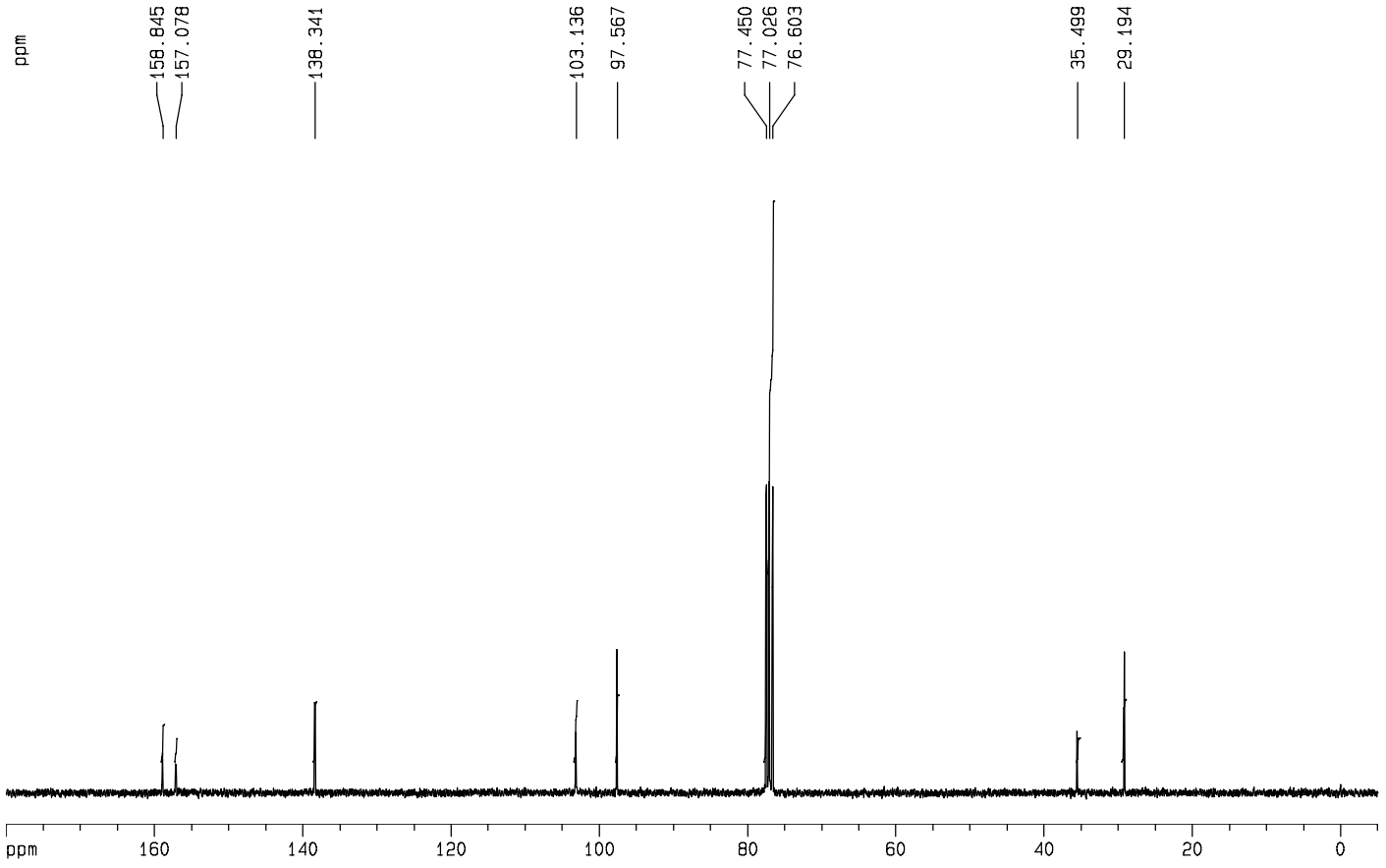


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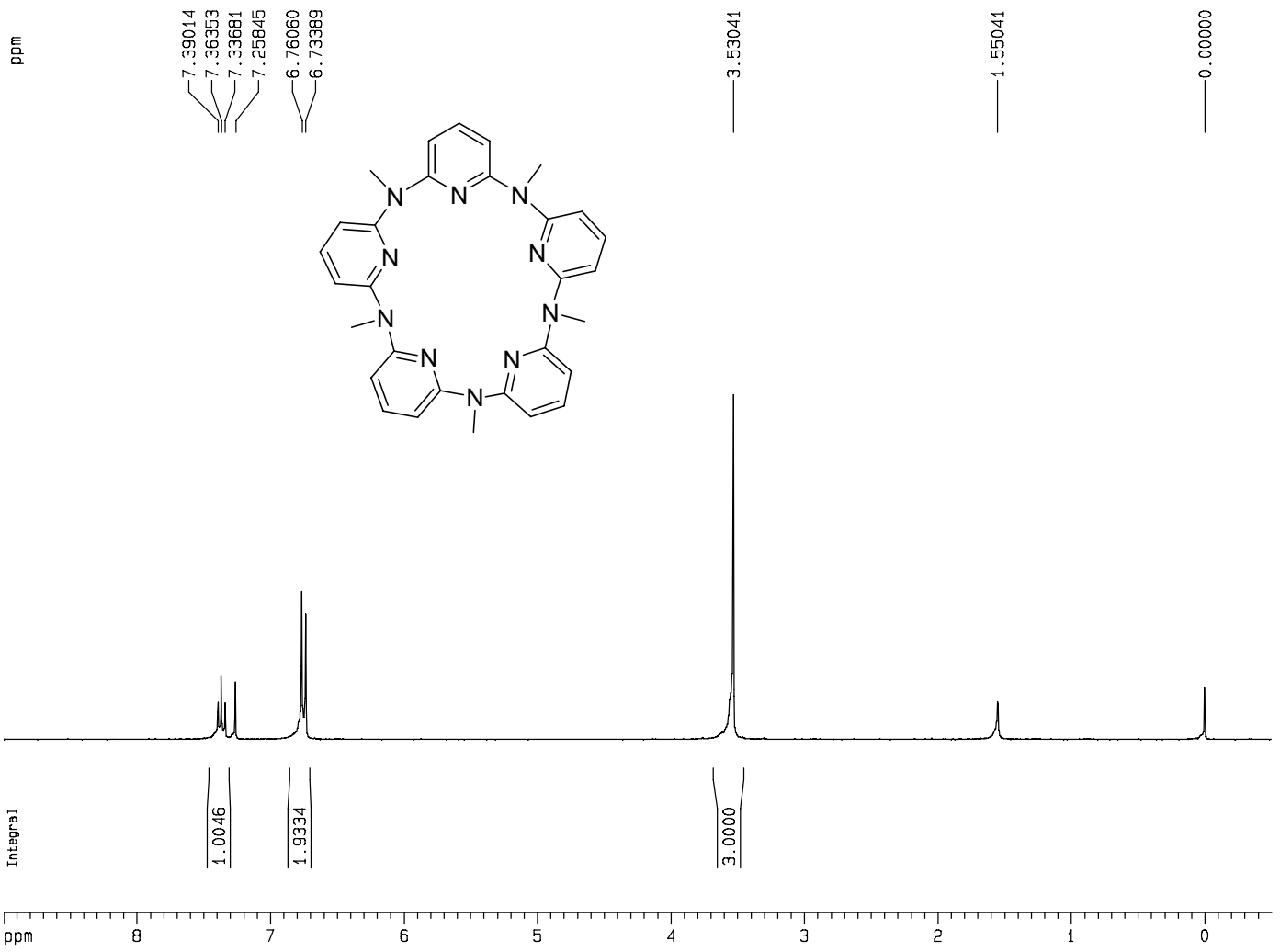


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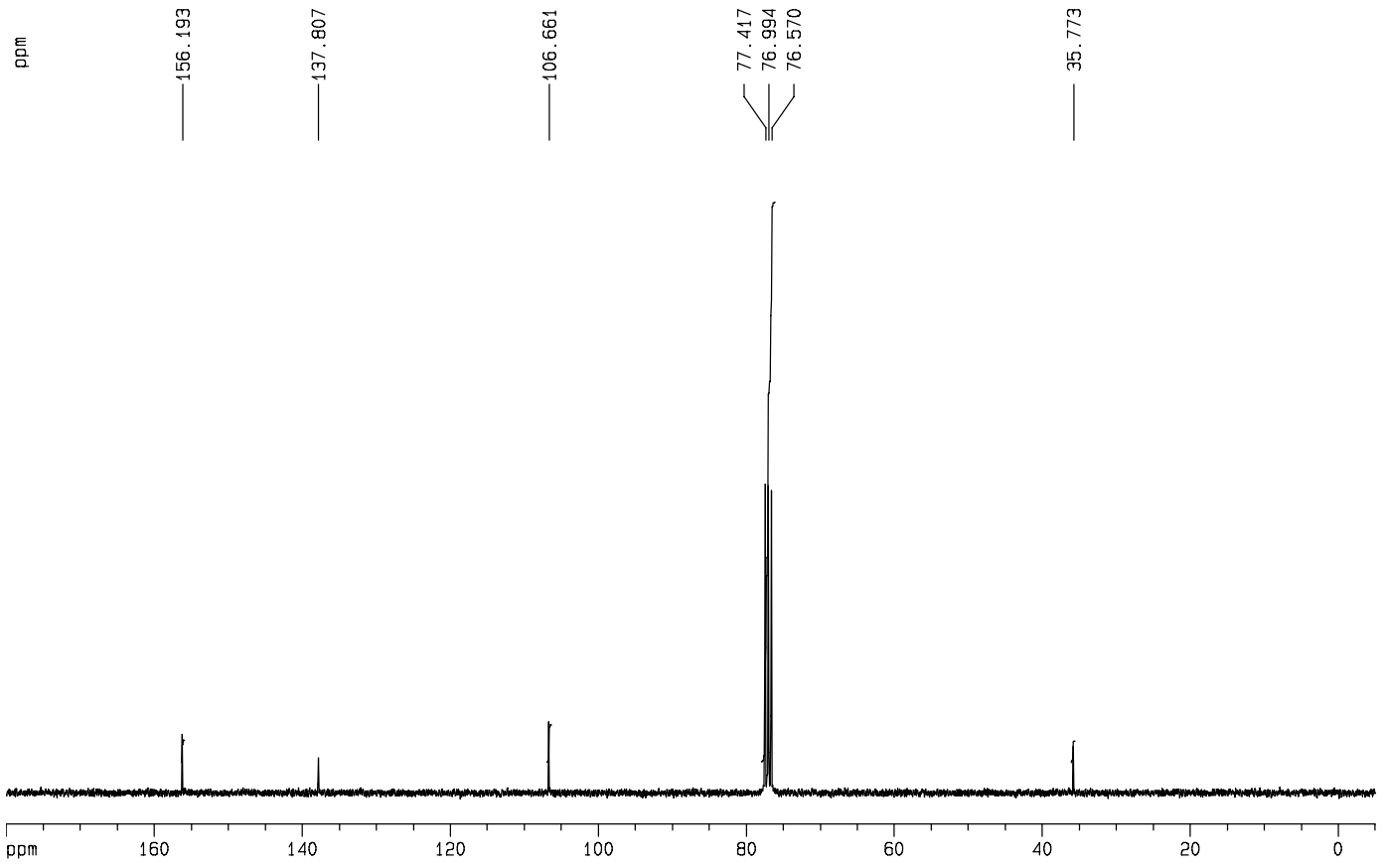


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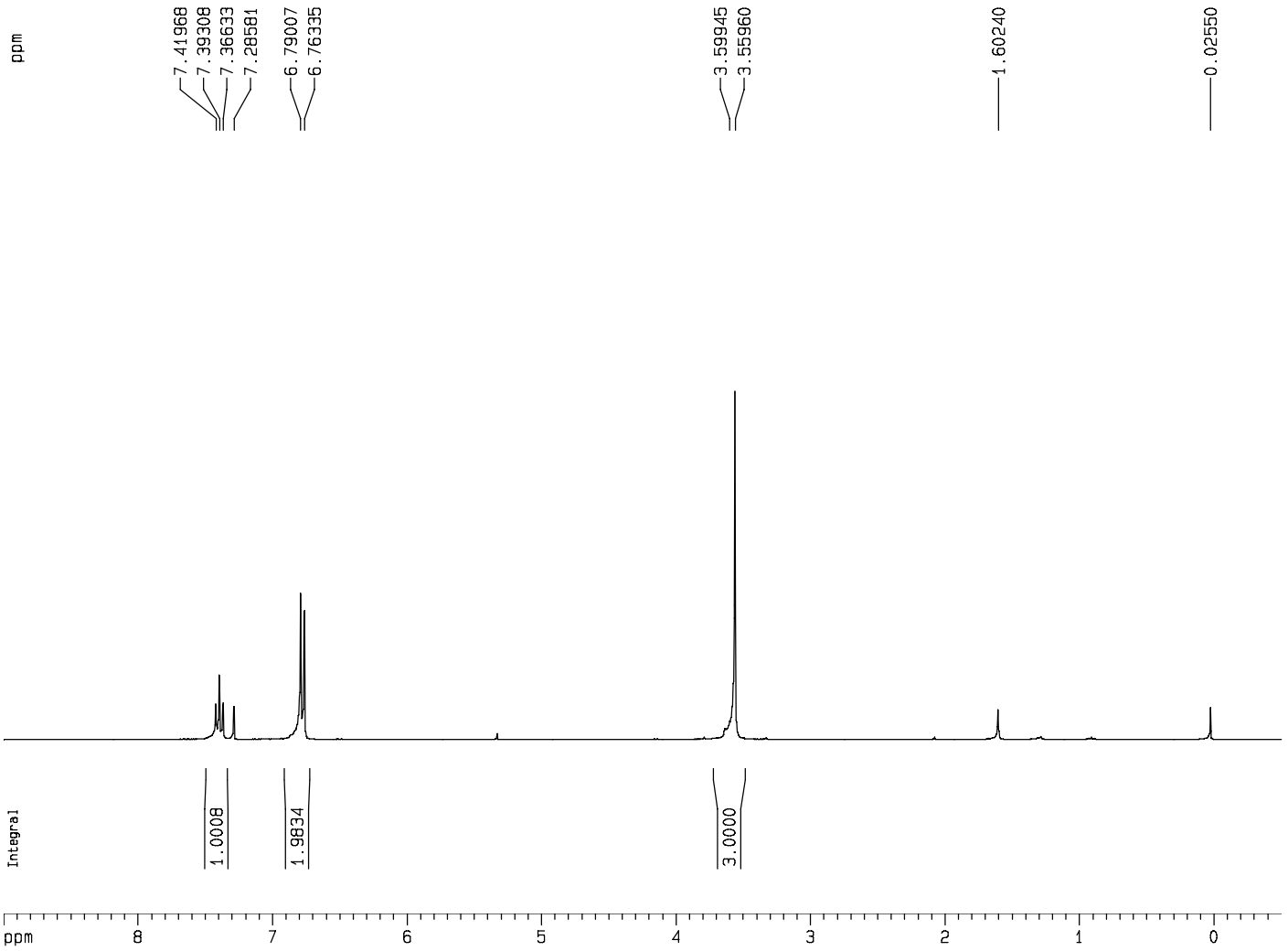


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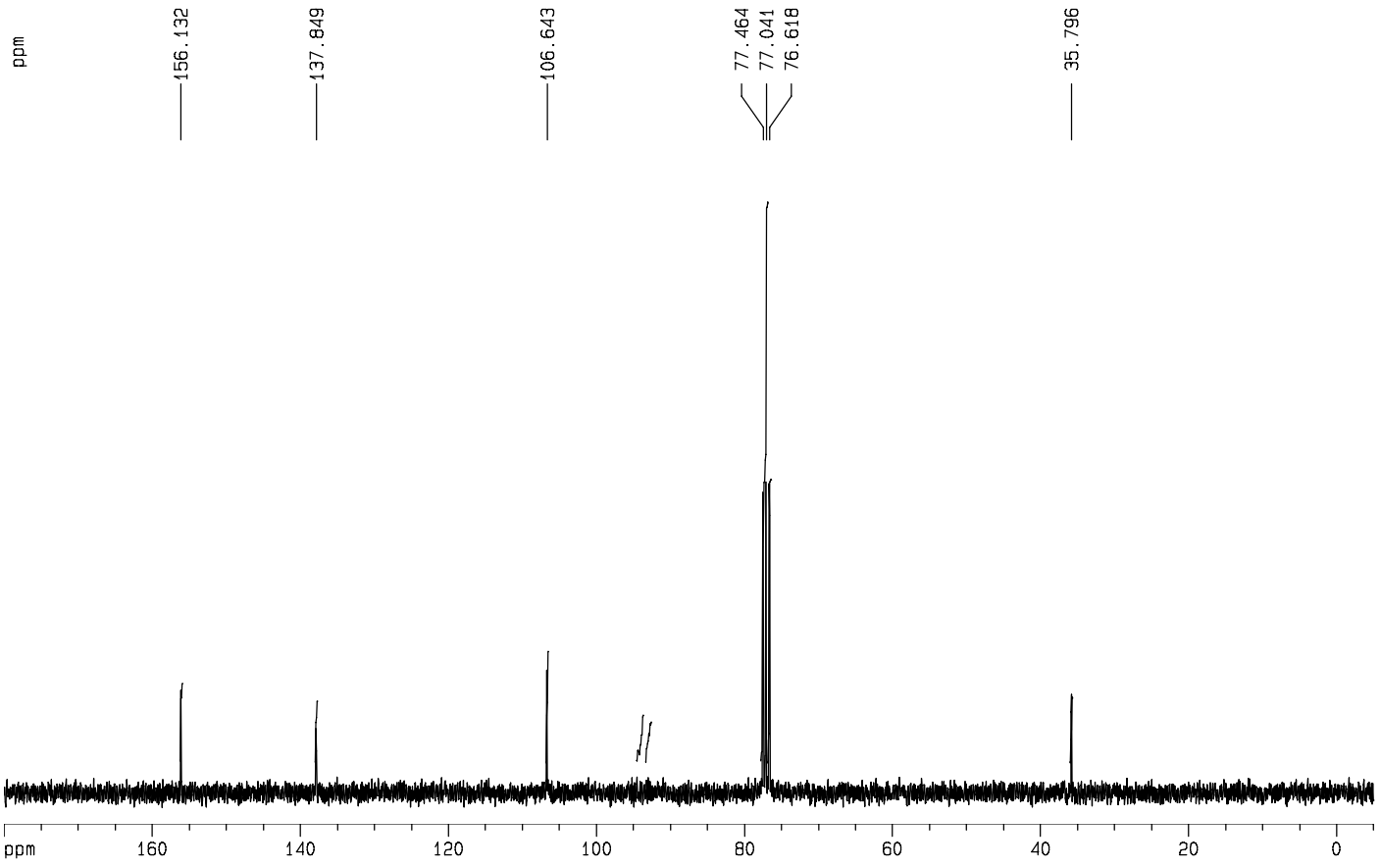


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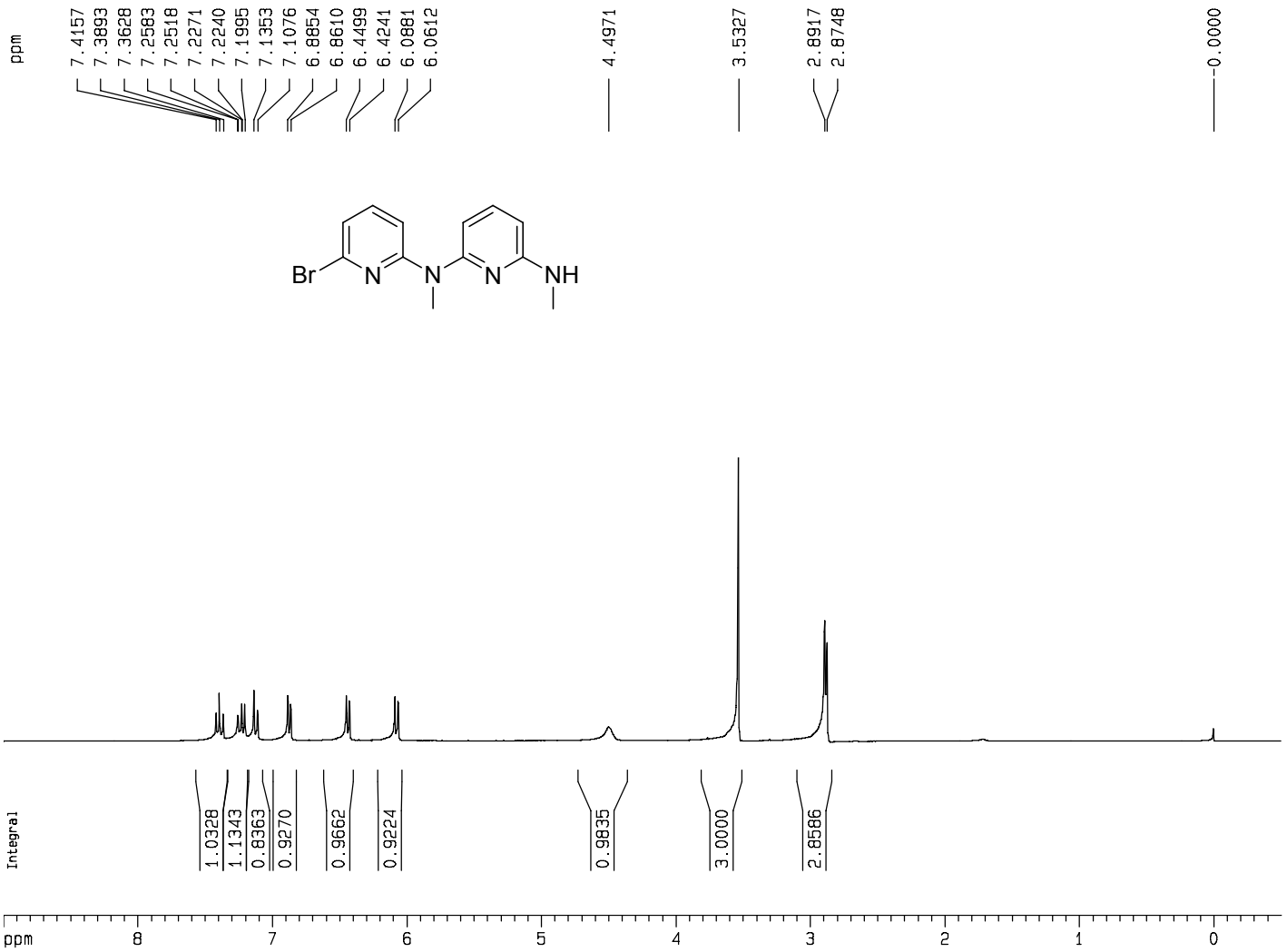
FIDRES           0.274439 Hz
AQ               1.8219508 sec
RG                  1448.2
DW                  27.800 usec
DE                   6.00 usec
TE                  292.8 K
D1              2.00000000 sec
d11             0.03000000 sec
d12             0.00002000 sec

======== CHANNEL f1 ========
NUC1                   13C
P1                    9.40 usec
PL1                  -1.00 dB
SFO1           75.4752953 MHz

======== CHANNEL f2 ========
CPDPRG2            waltz16
NUC2                    1H
PCPD2                80.00 usec
PL2                  -1.00 dB
PL12                 18.00 dB
PL13                 18.00 dB
SFO2          300.1312005 MHz

F2 - Processing parameters
SI                   32768
SF             75.4677490 MHz
WDW                     EM
SSB                      0
LB                    1.00 Hz
GB                       0
PC                    1.40

1D NMR plot parameters
CX                   22.00 cm
CY                    5.00 cm
F1P                 180.000 ppm
F1                13584.20 Hz
F2P                  -5.000 ppm
F2                 -377.34 Hz
PPMCM              8.40909 ppm/cm
HZCM             634.61517 Hz/cm
```



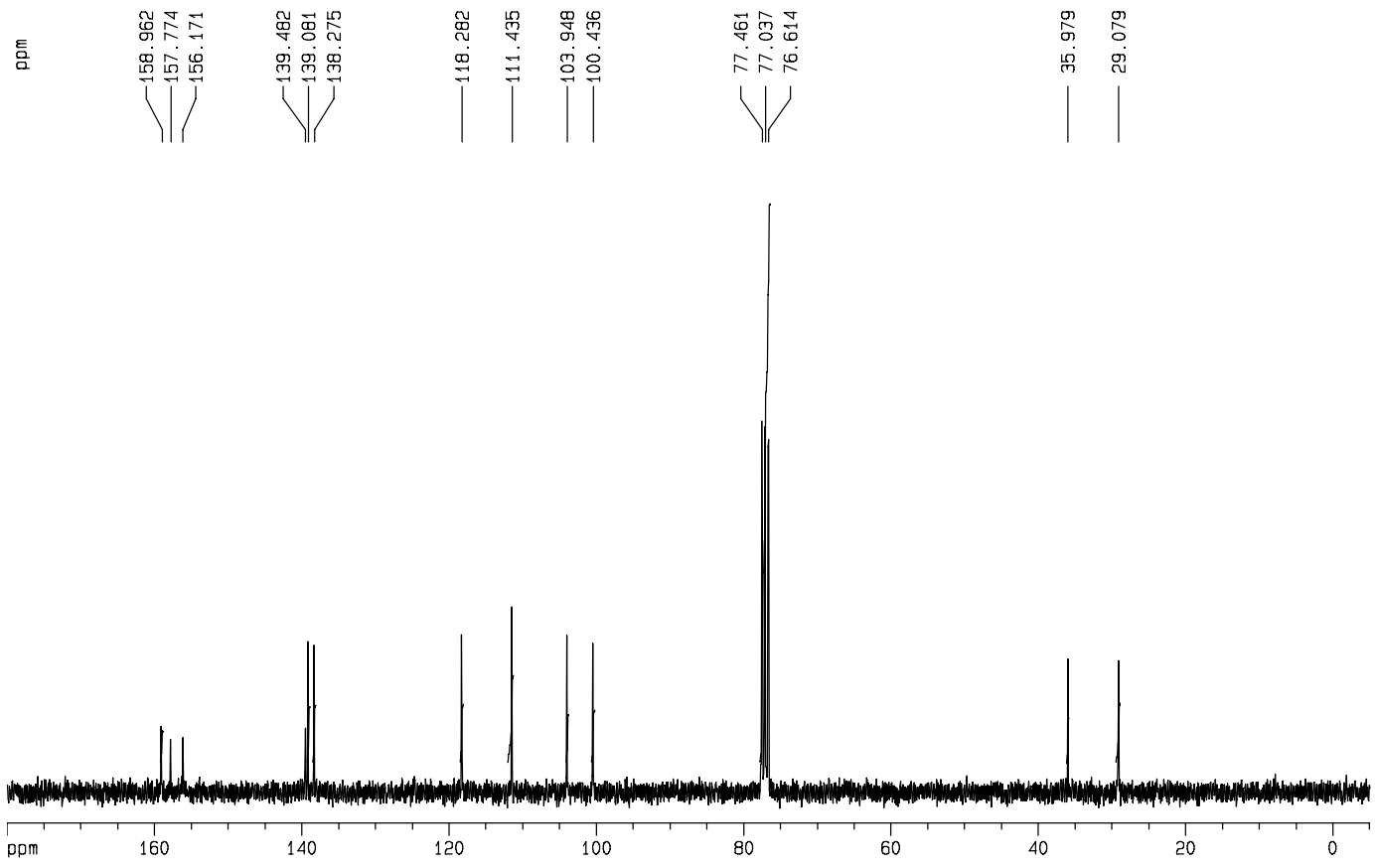
Current Data Parameters
NAME lsq73-26bddi
EXPNO 10
PROCNO 1

F2 - Acquisition Parameters
Date_ 20070327
Time 12.42
INSTRUM av300
PROBHD 5 mm DUL 13C-1
PULPROG zg30
TD 65536
SOLVENT CDCl3
NS 11
DS 0
SWH 8992.806 Hz
FIDRES 0.137219 Hz
AQ 3.6438515 sec
RG 161.3
DW 55.600 usec
DE 6.00 usec
TE 298.8 K
D1 5.00000000 sec

======== CHANNEL f1 ========
NUC1 1H
P1 9.30 usec
PL1 -1.00 dB
SFO1 300.1318534 MHz

F2 - Processing parameters
SI 32768
SF 300.1300062 MHz
WDW EM
SSB 0
LB 0.35 Hz
GB 0
PC 1.00

1D NMR plot parameters
CX 22.00 cm
CY 5.00 cm
F1P 9.000 ppm
F1 2701.17 Hz
F2P -0.500 ppm
F2 -150.07 Hz
PPMCM 0.43182 ppm/cm
HZCM 129.60159 Hz/cm

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Current Data Parameters
NAME       1sq73-26bddi
EXPNO                11
PROCNO                1

F2 - Acquisition Parameters
Date_          20070327
Time              12.45
INSTRUM           av300
PROBHD   5 mm DUL 13C-1
PULPROG          zgpg30
TD                65536
SOLVENT           CDCl3
NS                  140
DS                    4
SWH           17985.611 Hz
FIDRES        0.274439 Hz
AQ            1.8219508 sec
RG                  512
DW               27.800 usec
DE                6.00 usec
TE                298.7 K
D1           2.00000000 sec
d11          0.03000000 sec
d12          0.00002000 sec

======== CHANNEL f1 ========
NUC1                13C
P1                9.40 usec
PL1              -1.00 dB
SFO1         75.4752953 MHz

======== CHANNEL f2 ========
CPDPRG2         waltz16
NUC2                1H
PCPD2            80.00 usec
PL2              -1.00 dB
PL12             18.00 dB
PL13             18.00 dB
SFO2        300.1312005 MHz

F2 - Processing parameters
SI                32768
SF           75.4677490 MHz
WDW                  EM
SSB                   0
LB                 1.00 Hz
GB                    0
PC                 1.40

1D NMR plot parameters
CX                22.00 cm
CY                 6.00 cm
F1P              180.000 ppm
F1             13584.20 Hz
F2P               -5.000 ppm
F2              -377.34 Hz
PPMCM            8.40909 ppm/cm
HZCM           634.61517 Hz/cm
```
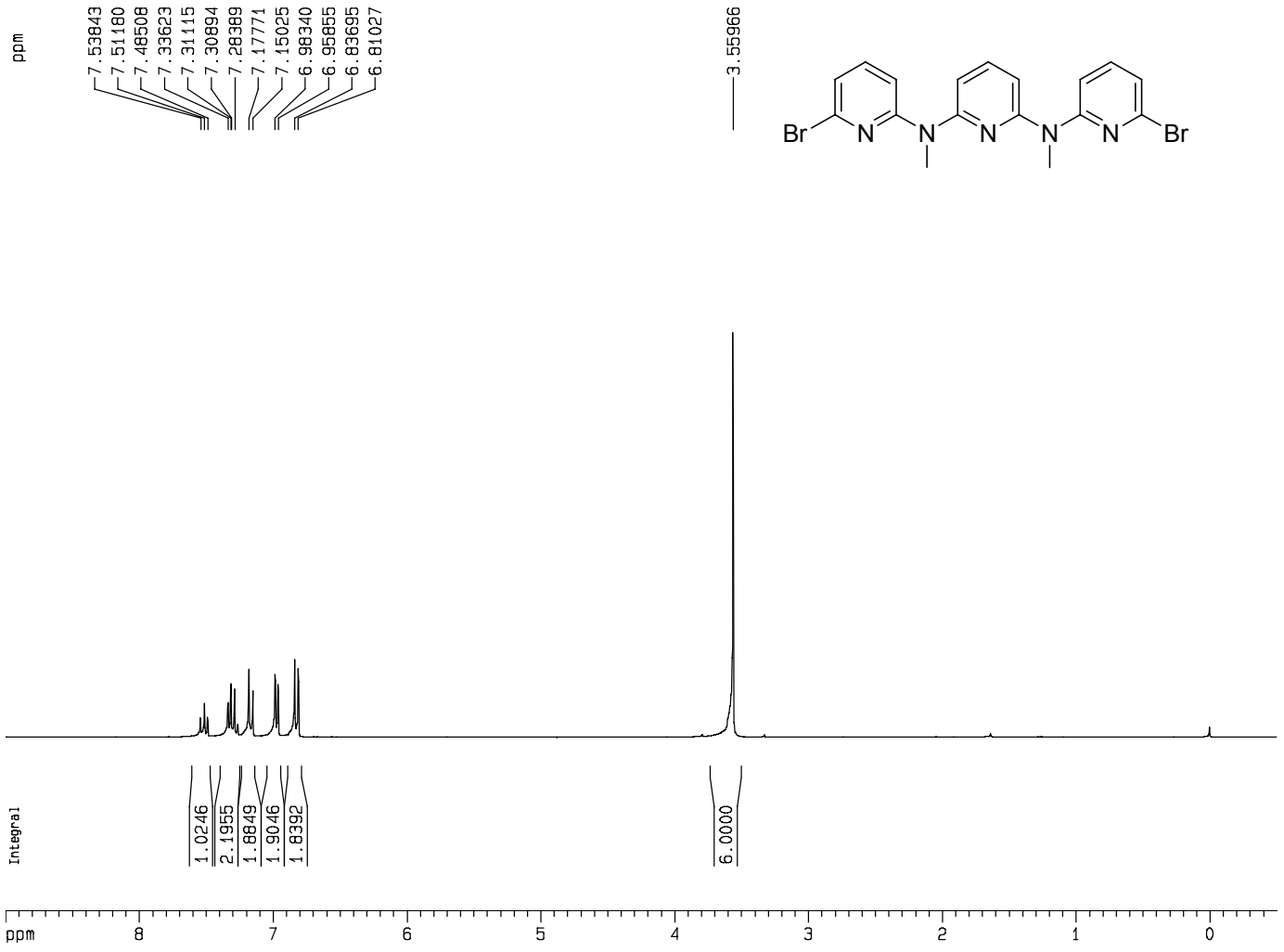
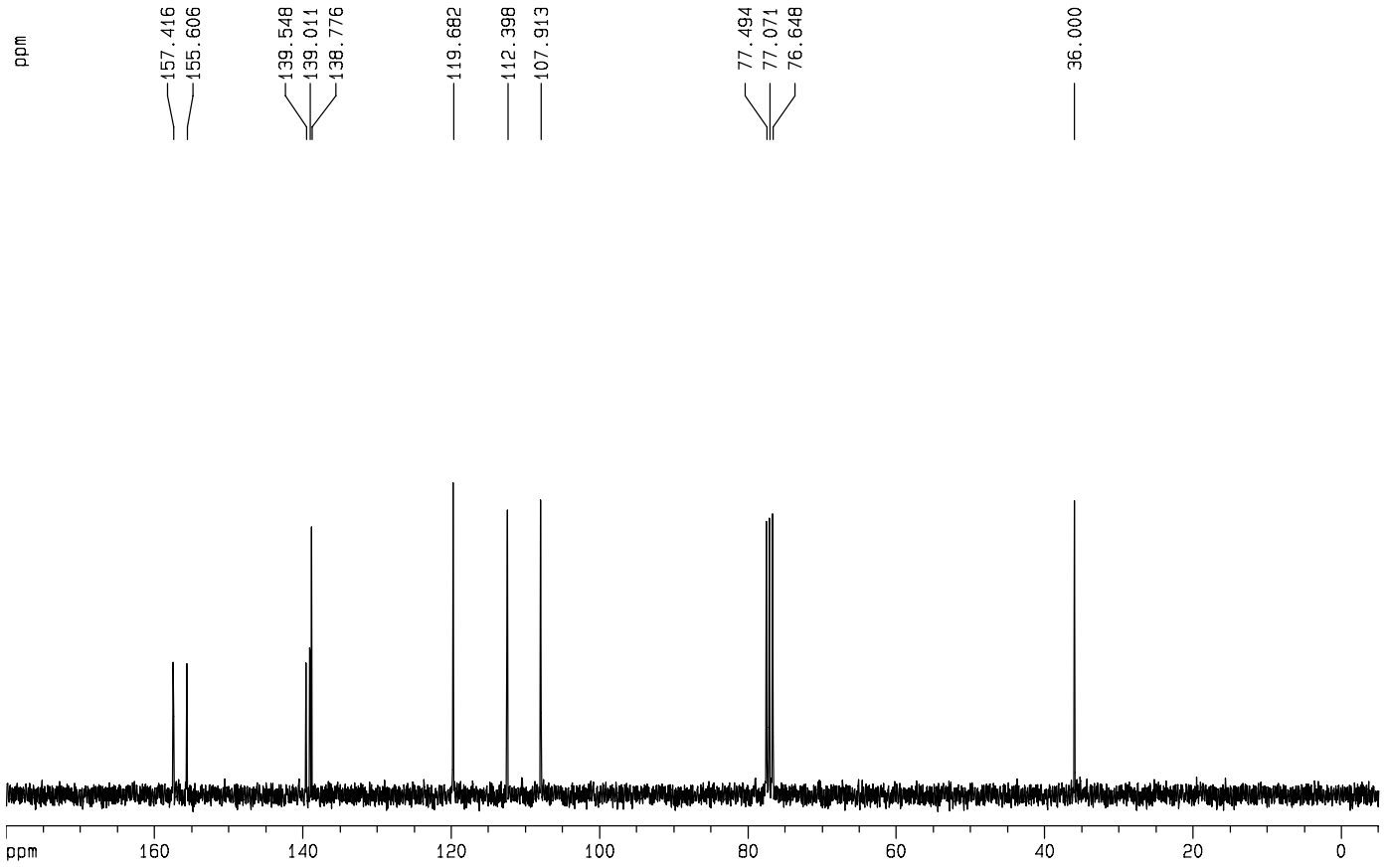




```
Current Data Parameters
NAME       lsq73-26bdtri
EXPNO                 10
PROCNO                 1

F2 - Acquisition Parameters
Date_           20070327
Time               12.32
INSTRUM            av300
PROBHD   5 mm DUL 13C-1
PULPROG             zg30
TD                 65536
SOLVENT            CDCl3
NS                     5
DS                     0
SWH             8992.806 Hz
FIDRES          0.137219 Hz
AQ              3.6438515 sec
RG                 143.7
DW                55.600 usec
DE                 6.00 usec
TE                298.0 K
D1             5.00000000 sec

======== CHANNEL f1 ========
NUC1                  1H
P1                 9.30 usec
PL1                -1.00 dB
SFO1         300.1318534 MHz

F2 - Processing parameters
SI                 32768
SF           300.1300051 MHz
WDW                   EM
SSB                    0
LB                  0.35 Hz
GB                     0
PC                  1.00

1D NMR plot parameters
CX                 22.00 cm
CY                  7.00 cm
F1P                9.000 ppm
F1              2701.17 Hz
F2P               -0.500 ppm
F2              -150.07 Hz
PPMCM           0.43182 ppm/cm
HZCM          129.60159 Hz/cm
```




```
Current Data Parameters
NAME       lsq73-26bdtri
EXPNO                 11
PROCNO                 1

F2 - Acquisition Parameters
Date_           20070327
Time               12.35
INSTRUM            av300
PROBHD   5 mm DUL 13C-1
PULPROG          zgpg30
TD                65536
SOLVENT           CDCl3
NS                   52
DS                    4
SWH           17985.611 Hz
FIDRES         0.274439 Hz
AQ             1.8219508 sec
RG                322.5
DW                27.800 usec
DE                 6.00 usec
TE                298.3 K
D1            2.00000000 sec
d11           0.03000000 sec
d12           0.00002000 sec

======== CHANNEL f1 ========
NUC1                  13C
P1                  9.40 usec
PL1                -1.00 dB
SFO1         75.4752953 MHz

======== CHANNEL f2 ========
CPDPRG2          waltz16
NUC2                  1H
PCPD2              80.00 usec
PL2                -1.00 dB
PL12               18.00 dB
PL13               18.00 dB
SFO2        300.1312005 MHz

F2 - Processing parameters
SI                 32768
SF           75.4677490 MHz
WDW                   EM
SSB                    0
LB                  1.00 Hz
GB                     0
PC                  1.40

1D NMR plot parameters
CX                 22.00 cm
CY                  5.00 cm
F1P               180.000 ppm
F1              13584.20 Hz
F2P                -5.000 ppm
F2               -377.34 Hz
PPMCM              8.40909 ppm/cm
HZCM             634.61517 Hz/cm
```