Supplementary Material (ESI) for Chemical Communications

Electronic Supplementary Information

Room-Temperature Aerobic Formation of Stable Aryl-Cu(III) Complex and Its Reactions with Nucleophiles: Highly Efficient and Diverse Arene C-H Functionalizations of Azacalix[1]arene[3]pyridine

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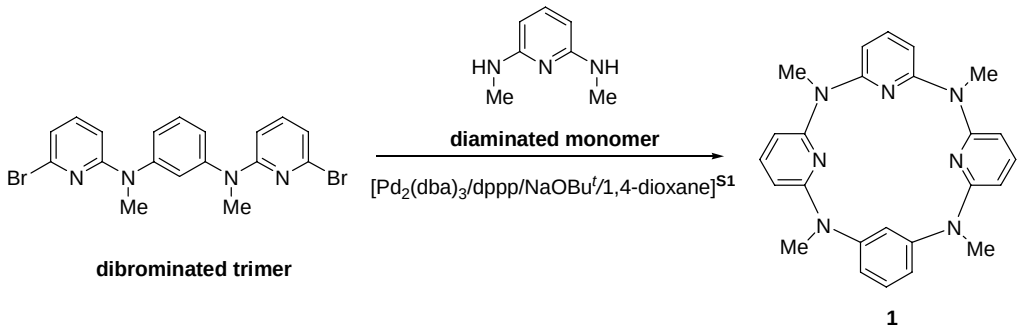
1. General Information

Chemical shifts are reported in ppm with either tetramethylsilane or the residual solvent resonance used as an internal standard. Melting points are uncorrected. Elemental analyses, mass spectrometry, and X-ray photonelectron spectroscopy were performed at the Analytical Laboratory of the Institute. 1, 4-Dioxane was treated with sodium and acetonitrile was treated with CaH_2 before use.

2. Experimental Procedure and Characterization of Products

Synthesis of Azacalix[1]arene[3]pyridine



Scheme S1. Synthesis of azacalix[1]arene[3]pyridine **1**

A mixture of the diamine[S1] (276 mg, 2 mmol), the dibrominated trimer [S1] (896mg, 2 mmol), $Pd_2(dba)_3$ (276 mg, 0.3 mmol), DPPP (246 mg, 0.6 mmol) and sodium *tert*-butoxide (576 mg, 6 mmol) was heated at reflux under argon protection in 400 mL anhydrous 1,4-dioxane for 3 hours. After cooling to room temperature, the mixture was filtered through a celite pad. Solvent was removed and the residue was dissolved in dichloromethane and washed with brine. The organic layer was dried over anhydrous sodium sulfate. After removal of solvent under vacuum, the residue was subjected to a basic aluminium oxide column with a mixture of petroleum ether and acetone (15:1) as eluent to give pure product **1** (270 mg, 31.8 %) as white crystalline solids.

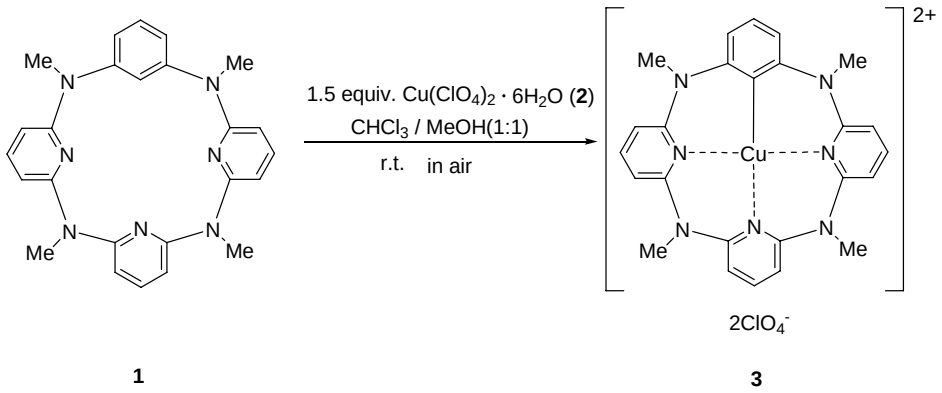
^{1}H NMR (300 MHz, $CDCl_3$): δ 7.39 (t, J = 8.0 Hz, 2H), 7.18 (t, J = 7.7 Hz, 1H), 7.10 (t, J = 7.8 Hz, 1H), 6.77 (dd, J_1 = 1.9 Hz, J_2 = 7.9 Hz, 2H), 6.59 (s, 1H), 6.58 (d, J = 7.5 Hz, 2H), 6.04 (d, J = 8.3 Hz, 2H), 6.01 (d, J = 8.7 Hz, 2H), 3.22 (s, 6H), 3.14 (s, 6H) ppm; ^{13}C NMR (75MHz, $CDCl_3$): δ 158.9, 158.8, 157.9, 149.1, 138.8, 137.5, 129.0, 128.1, 125.9, 120.2, 95.7, 95.3, 38.7, 36.4 ppm; IR(KBr):

1575(s), 1471(s), 1420(s), 1369(m), 1273(w), 1154(m), 909(w), 770(m), 729(m); ESI-MS: *m/z* 424.3 $[M+H]^+$; elemental analysis calcd(%) for $C_{25}H_{25}N_7$: C 70.90, H 5.95, N 23.15; found: C 71.04, H 6.01, N 22.92; mp 243-244℃.

References

S1. For the synthetic procedures of the dibrominated trimer and the diamine and the general procedure of the fragment coupling protocol, see: (a) H.-Y. Gong, X.-H. Zhang, D.-X. Wang, H.-W. Ma, Q.-Y. Zheng, M.-X. Wang*, *Chem. Eur. J.*, **2006**, *12*, 9262-9275; (b) E.-X. Zhang, D.-X. Wang, Q.-Y. Zheng and M.-X. Wang*, *Org. Lett.* **2008**, *10*, 2565-2568.

Synthesis of the Copper (III) Complex 3



1 **3**

Scheme S2. Synthesis of the copper (III) complex **3** from **1**

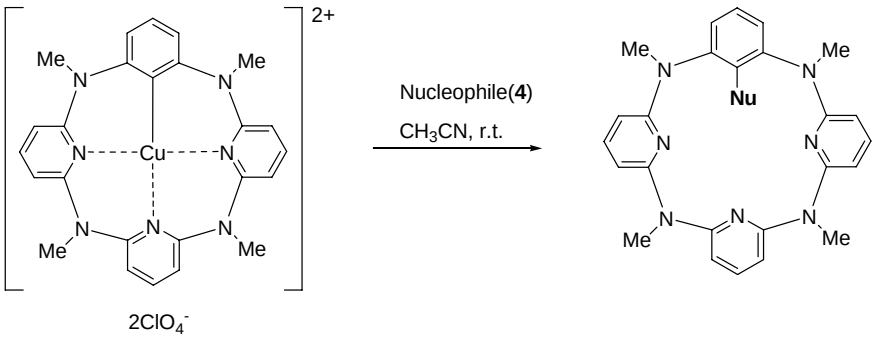
Azacalix[1]arene[3]pyridine **1** (42.3 mg, 0.1 mmol) and $Cu(ClO_4)_2$•$6H_2O$ **2** (55.6 mg, 0.15 mmol) were dissolved in a mixture of chloroform (4 mL) and methanol (4 mL). The solution turned dark blue immediately and then turned light with the formation of dark purple precipitates. After about 60 minutes, the precipitate was filtered and dried to get a purple solid (68.2 mg, Yield 99%). Slow diffusion of ethyl acetate into the acetonitrile solution of the product resulted in the formation of a black single crystal suitable for X-ray diffraction analysis.

^{1}H NMR (300 MHz, $CDCl_3$): δ 8.17 (t, J = 8.1 Hz, 1H), 8.10 (t, J = 8.0 Hz, 2H), 7.51 (t, J = 7.6 Hz, 1H), 7.39 (dd, J_1 = 0.9 Hz, J_2 = 8.3 Hz, 2H), 7.32 (d, J = 8.1 Hz, 2H), 7.19 (dd, J_1 = 0.9 Hz, J_2 = 7.8 Hz, 2H), 7.08 (d, J = 7.6 Hz, 2H), 3.68 (s, 6H), 3.53 (s, 6H) ppm; ^{13}C NMR (75MHz, $CDCl_3$): δ 152.2, 151.2, 150.8, 144.5, 142.9, 138.2, 135.8, 130.2, 114.8, 111.4, 109.7, 108.6, 37.3, 37.0 ppm;

IR(KBr): 1584(s), 1485(s), 1427(s), 1353(w), 1134(s), 1092(s), 943(w), 787(m), 624(m); ESI-MS: *m/z* 584.2 [**3**-ClO_4]$^+$, 242.6 [**3**-2ClO_4]$^{2+}$; elemental analysis calcd(%) for $C_{25}H_{24}N_7CuCl_2O_8$: C 43.84, H 3.53, N 14.31; found: C 43.61, H 3.75, N 14.63;

General Procedure for the Reaction between 3 and Nucleophiles 4

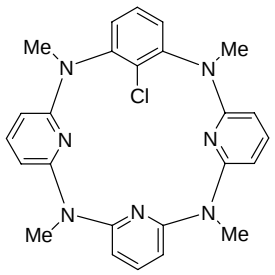
The copper (III) complex **3** (35 mg, 0.05 mmol) was dissolved in acetonitrile (3 mL) to give a dark brown solution. Nucleophile **4** (0.05 mmol or 0.1 mmol, see Table 1) was added into the solution at room temperature. When the color faded away or turned orange, the reaction went completion. Aqueous ammonia (5 mL) was added, and the mixture was extracted by dichloromethane (3×5 mL). The extract was washed with brine and dried over anhydrous sodium sulfate. The solvent was evaporated and the residue was chromatographed on a silica gel column with a mixture of petroleum ether, ethyl acetate, and dichloromethane (12:1:2) to give pure product **5**.



3 **5**

Scheme S3. Reaction of **3** with **4**

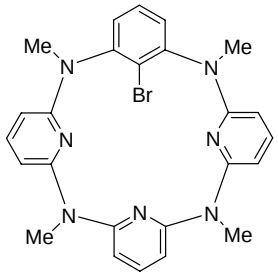
Characterization of products **5**



5a

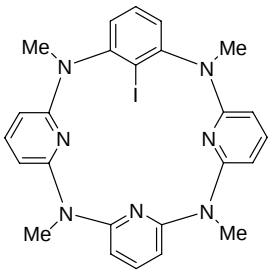
Yield(1.0 eq. NEt_4Cl): 99%. ^{1}H NMR (300 MHz, $CDCl_3$): δ 7.42 (t, *J* = 8.0 Hz, 2H), 7.19 (t, *J* = 7.6 Hz, 1H), 7.07-7.02 (m, 1H), 6.93 (d, *J* = 7.7 Hz, 2H), 6.62 (d, *J* = 7.6 Hz, 2H), 6.06 (d, *J* = 8.0Hz, 2H), 6.02 (d, *J* = 8.1 Hz, 2H), 3.24 (s, 6H), 3.13 (s, 6H) ppm; ^{13}C NMR (75MHz, $CDCl_3$): δ 158.8,

158.7, 156.9, 145.9, 138.9, 137.2, 134.2, 128.8, 126.5, 120.6, 95.3, 94.2, 37.2, 36.2 ppm; IR(KBr): 1574(s), 1476(s), 1423(s), 1370(m), 1277(m), 1158(s), 1135(m), 954(w), 770(s), 732(m), 720(m); ESI-MS: *m/z* 458.2 $[M+H]^+$; elemental analysis calcd(%) for $C_{25}H_{24}N_7Cl$: C 65.57, H 5.28, N 21.41; found: C 65.56, H 5.37, N 21.38; mp 259-260℃.



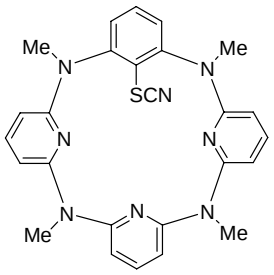
5b

Yield(1.0 eq. NEt_4Br): 97%.^{1}H NMR (300 MHz, $CDCl_3$): δ 7.44 (t, *J* = 8.0 Hz, 2H), 7.19 (t, *J* = 7.7 Hz, 1H), 7.14-7.08 (m, 1H), 6.93 (d, *J* = 7.6 Hz,2H), 6.63 (d, *J* = 7.3 Hz, 2H), 6.06 (d, *J* = 8.0 Hz, 2H), 6.01 (d, *J*=8.0 Hz, 2H), 3.24 (s, 6H), 3.13 (s, 6H) ppm; ^{13}C NMR (75MHz, $CDCl_3$): δ 158.8, 158.6, 156.7, 147.6, 138.9, 137.2, 128.8, 127.4, 127.3, 120.7, 95.2, 94.0, 37.1, 36.1 ppm; IR(KBr): 1573(s), 1475(s), 1420(s), 1370(m), 1275(m), 1157(m), 1133(m), 770(m), 719(w); ESI-MS: *m/z* 502.3 $[M+H]^+$; elemental analysis calcd(%) for $C_{25}H_{24}N_7Br$: C 59.77, H 4.81, N 19.52; found: C 60.03, H 4.80, N 19.21; mp 257-258℃.



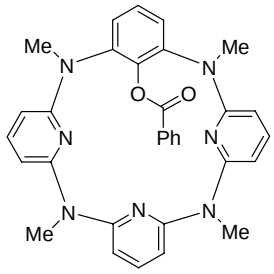
5c

Yield(1.0 eq. NEt_4I): 90%.^{1}H NMR (300 MHz, $CDCl_3$): δ 7.45 (t, *J* = 8.0 Hz, 2H), 7.19-7.12 (m, 2H), 6.87 (d, *J* = 7.1 Hz, 2H), 6.60 (d, *J* = 7.1 Hz, 2H), 6.06 (d, *J* = 8.0 Hz, 2H), 6.00 (d, *J* = 8.0 Hz, 2H), 3.25 (s, 6H), 3.13 (s, 6H) ppm; ^{13}C NMR (75MHz, $CDCl_3$): δ 158.7, 158.5, 156.4, 151.4, 139.0, 137.2, 128.8, 127.7, 120.6, 108.7, 95.2, 94.2, 37.2, 36.1 ppm; IR(KBr): 1585(s), 1474(s), 1419(s), 1369(m), 1342(m), 1275(m), 1153(s), 1130(m), 768(s), 721(m); ESI-MS: *m/z* 550.3 $[M+H]^+$, 572.3 $[M+Na]^+$; elemental analysis calcd(%) for $C_{25}H_{24}N_7I$: C 54.65, H 4.40, N 17.85; found: C 54.76, H 4.45, N 17.59; mp 225-226℃.



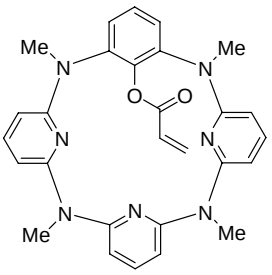
5d

Yield(1.0 eq. NaSCN): 95%.^{1}H NMR (300 MHz, $CDCl_3$): δ 7.48 (t, *J* = 8.0 Hz, 2H), 7.29 (t, *J* = 7.9 Hz, 1H), 7.12 (t, *J* = 7.7 Hz, 1H), 6.97 (d, *J* = 7.9 Hz, 2H), 6.55 (d, *J* = 7.7 Hz, 2H), 6.12 (d, *J* = 8.0 Hz, 4H), 3.26 (s, 6H), 3.24 (s, 6H) ppm; ^{13}C NMR (75MHz, $CDCl_3$): δ 158.7, 158.6, 156.7, 150.3, 139.3, 137.3, 131.6, 127.7, 127.1, 120.3, 112.7, 96.2, 95.1, 38.3, 36.3 ppm; IR(KBr): 2151(m), 1561(s), 1474(s), 1420(s), 1372(m), 1151(m), 1120(m), 767(m); ESI-MS: *m/z* 481.2 $[M+H]^+$, 503.2 $[M+Na]^+$; elemental analysis calcd(%) for $C_{26}H_{24}N_8S$: C 64.98, H 5.03, N 23.32; found: C 64.93, H 5.04, N 23.20; mp 215-216℃.



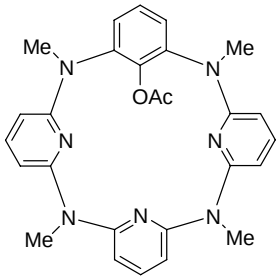
5e

Yield(1.0eq. PhCOONa): 91%.^{1}H NMR (300 MHz, $CDCl_3$): δ 7.65 (d, *J* = 7.6 Hz, 2H), 7.44 (t, *J* = 7.3 Hz, 1H), 7.31-7.20 (m, 5H), 7.07-7.02 (m,1H), 6.92 (d, *J* = 7.7 Hz, 2H), 6.64 (d, *J* = 7.6 Hz, 2H), 6.05 (d, *J* = 7.9 Hz, 2H), 5.82 (d, *J* = 8.0 Hz, 2H), 3.28 (s, 6H), 3.02 (s, 6H) ppm; ^{13}C NMR (75MHz, $CDCl_3$): δ 165.9, 159.0, 158.6, 157.0, 146.3, 140.8, 138.7, 137.5, 133.0, 130.3, 129.2, 128.0, 127.5, 125.8, 120.2, 95.6, 95.0, 37.7, 36.4 ppm; IR(KBr): 1727(s), 1576(s), 1473(s), 1419(s), 1369(m), 1268(m), 1208(w), 1125(w), 772(w), 712(w); ESI-MS: *m/z* 544.3 $[M+H]^+$, 566.3 $[M+Na]^+$; elemental analysis calcd(%) for $C_{32}H_{29}N_7O_2$: C 70.70, H 5.38, N 18.04; found: C 70.72, H 5.42, N 17.94; mp 245-246℃.



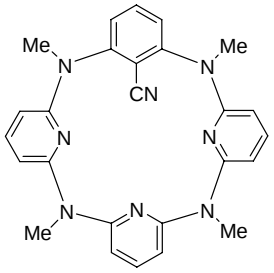
5f

Yield(1.0 eq. CH_2=CHCOONa): 95%.^{1}H NMR (300 MHz, $CDCl_3$): δ 7.37 (t, *J* = 8.0 Hz, 2H), 7.19 (t, *J* = 7.7 Hz, 1H), 7.04-6.99 (t, *J* = 7.8 Hz, 1H), 6.89 (d *J* = 7.7 Hz, 2H), 6.60 (d, *J* = 7.7 Hz, 2H), 6.15 (dd, J_1 = 1.2 Hz, J_2 = 17.2 Hz, 1H), 6.05 (d, *J* = 8.0 Hz, 2H), 6.00-5.91 (m, 1H), 5.94 (d, *J* = 7.7 Hz, 2H), 5.65 (dd, J_1 = 1.2 Hz, J_2 = 10.3 Hz, 1H), 3.25 (s, 6H), 3.02 (s, 6H) ppm; ^{13}C NMR (75MHz, $CDCl_3$): δ 165.3, 158.9, 158.6, 157.0, 146.0, 140.7, 138.8, 137.4, 132.0, 127.8, 127.5, 125.8, 120.3, 95.6, 94.9, 37.6, 36.3 ppm; IR(KBr): 1735(m), 1576(s), 1477(s), 1424(m), 1370(w), 1162(m), 1124(m), 769(w); ESI-MS: *m/z* 494.3 $[M+H]^+$, 516.3 $[M+Na]^+$; elemental analysis calcd(%) for $C_{28}H_{27}N_7O_2$: C 68.14, H 5.51, N 19.87; found: C 67.90, H 5.57, N 19.82; mp 242-243℃.



5g

Yield(2.0 eq. NaOAc): 91%.^{1}H NMR (300 MHz, $CDCl_3$): δ 7.40 (t, *J* = 8.0 Hz, 2H), 7.18 (t, *J* = 7.7 Hz, 1H), 7.04-6.98 (m, 1H), 6.87 (t, *J* = 7.7 Hz, 2H), 6.60 (d, *J* = 7.7 Hz, 2H), 6.06 (d, *J* = 8.0 Hz, 2H), 5.98 (d, *J* = 8.0 Hz, 2H), 3.25 (s, 6H), 3.03 (s, 6H), 1.86 (s, 3H) ppm; ^{13}C NMR (75MHz, $CDCl_3$): δ 170.4, 158.8, 158.6, 157.0, 146.1, 140.7, 138.9, 137.5, 127.7, 125.8, 120.5, 95.5, 94.7, 37.7, 36.2, 20.6 ppm; IR(KBr): 1751(m), 1575(s), 1478(s), 1431(m), 1370(m), 1281(w), 1218(m), 1198(m), 1155(m), 1127(m), 958(w), 768(m), 721(w); ESI-MS: *m/z* 482.4 $[M+H]^+$; elemental analysis calcd(%) for $C_{27}H_{27}N_7O_2$: C 67.34, H 5.65, N 20.36; found: C 67.38, H 5.60, N 20.19; mp 243-244℃.

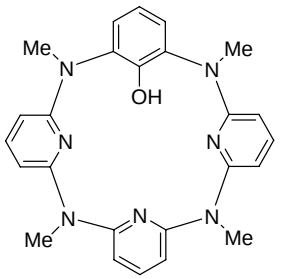


5h

Yield(2.0 eq. KCN): 99%.^{1}H NMR (300 MHz, $CDCl_3$): δ 7.45 (t, *J* = 8.0 Hz, 2H), 7.31 (t, *J* = 8.0 Hz, 1H), 7.19 (t, *J* = 7.7 Hz, 1H), 6.91 (t, *J* = 8.0 Hz, 2H), 6.58 (d, *J* = 7.7 Hz, 2H), 6.13 (d, *J* = 8.0 Hz, 2H), 6.10 (d, *J* = 8.0 Hz, 2H), 3.25 (s, 6H), 3.23 (s, 6H) ppm; ^{13}C NMR (75MHz, $CDCl_3$): δ 159.0, 158.6, 157.1, 152.3, 139.2, 137.3, 132.9, 127.4, 120.0, 115.1, 114.6, 96.7, 95.4, 37.7, 36.4 ppm; IR(KBr): 2224(m), 1577(s), 1475(s), 1422(s), 1366(w), 1275(w), 1156(m), 1119(m), 955(w), 770(m), 719(w); ESI-MS: *m/z* 449.4 $[M+H]^+$, 471.4 $[M+Na]^+$, 487.3 $[M+K]^+$; elemental analysis calcd(%) for $C_{26}H_{24}N_8$: C 69.62, H 5.39, N 24.98; found: C 69.50, H 5.39, N 24.80.

Preparation of hydroxylated tetraazacalix[1]arene[3]pyridine 6

To a solution of **5g** (25.3 mg) in 10 mL of methanol was added 0.5 mL of sodium hydroxide aqueous solution (2.0 M). The mixture was kept stirring at room temperature for several hours till the reactant was consumed. The solvent was removed and the residue was dissolved in dichloromethane. Then the organic phase was dried over with anhydrous sodium sulfate. After filtration and removal of solvent, the residue was chromatographed on a silica gel column with a mixture of petroleum ether, ethyl acetate, and dichloromethane (12:1:2) to give pure product **6** (21.2mg, Yield 93%).



6

Yield: 93%.^{1}H NMR (300 MHz, $CDCl_3$): δ 7.44 (t, *J* = 8.0 Hz, 2H), 7.17 (t, *J* = 7.7 Hz, 1H), 6.79 (d, *J* = 7.4 Hz, 1H), 6.68 (dd, J_1 = 6.9 Hz, J_2 = 8.5 Hz, 1H), 6.57 (d, *J* = 7.6 Hz, 2H), 6.11 (d, *J* = 7.9 Hz, 2H), 6.10 (d, *J* = 8.0 Hz, 2H), 3.25 (s, 6H), 3.14 (s, 6H) ppm; ^{13}C NMR (75MHz, $CDCl_3$): δ 159.0, 158.9, 157.4, 150.0, 139.2, 137.6, 135.6, 126.4, 120.0, 119.5, 96.3, 95.5, 38.4, 36.4 ppm; IR(KBr): 3207(br), 2902(w), 1577(s), 1470(s), 1420(s), 1370(s), 1344(w), 1277(m), 1149(s), 1128(m), 953(w),

770(s), 734(m), 723(m); ESI-MS: *m/z* 440.3 $[M+H]^+$, 462.3 $[M+Na]^+$, 478.3 $[M+K]^+$; elemental analysis calcd(%) for $C_{25}H_{25}N_7O_2$ [**6**•H_2O]: C 65.63, H 5.95, N 21.43; found: C 65.64, H 5.99, N 21.20; mp 207-208℃.

3. X-ray photoelectron Spectroscopy Data of 3

X-ray photoelectron spectroscopy data were obtained with an ESCALab220i-XL electron spectrometer from VG Scientific using 300W MgKα radiation. The base pressure was about 3×10^{-9} mbar. The binding energies were referenced to the C1s line at 284.8 eV from adventitious carbon. And the acquisition parameters were as follows.

Acquisition Parameters :

Total acq. time	5 mins 0.5 secs
No. Scans	6
Lens Mode	LargeArea
Analyser Mode	CAE : Pass Energy 30.0 eV
Energy Step Size	0.1 eV
No. of Energy Steps	501

The binding energy for $Cu2p_{3/2}$ was 936.1ev which is in agreement with copper(III) species.

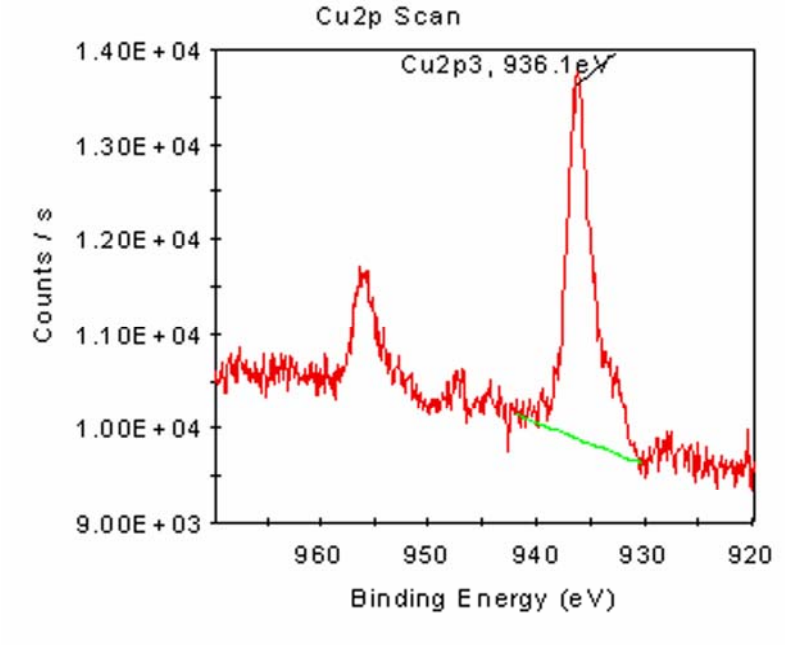
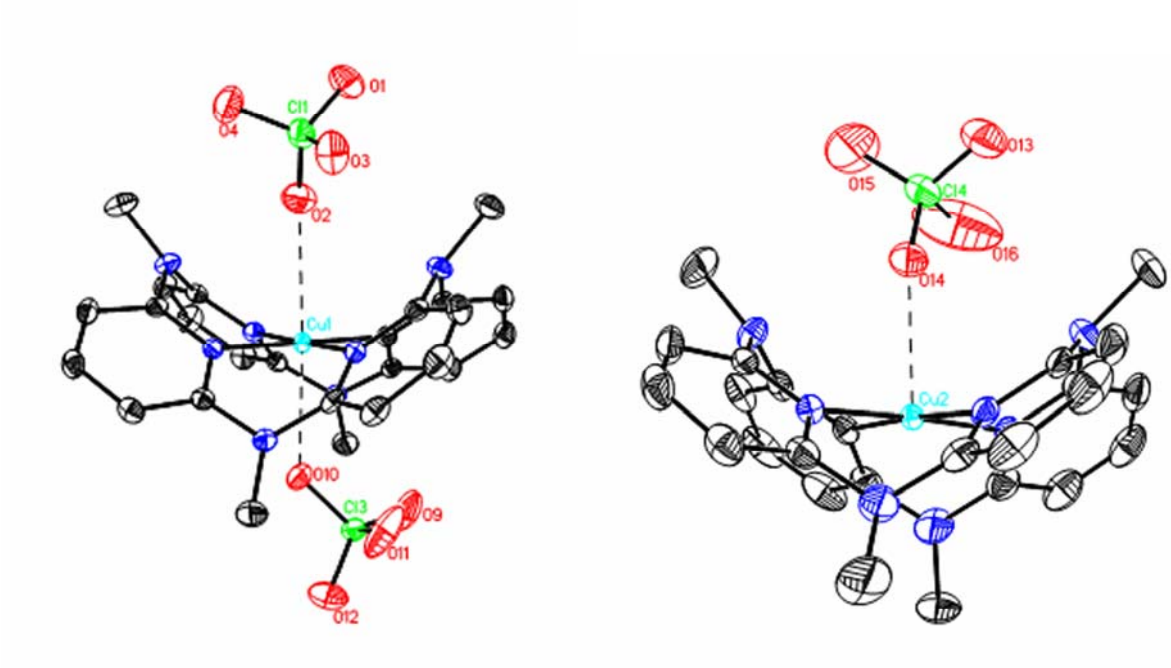


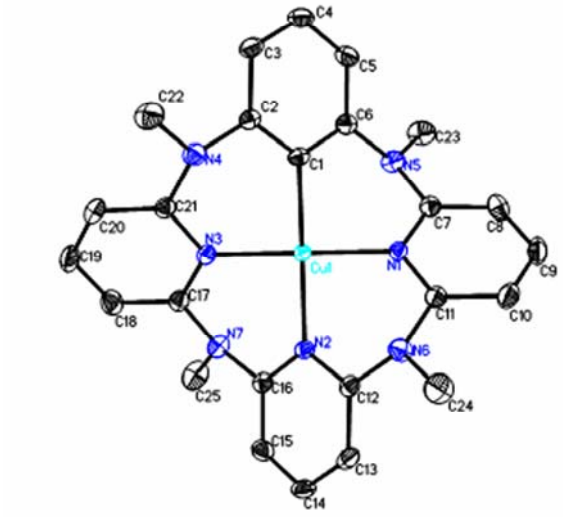
Figure S1. X-ray photoelectron spectroscopy of the complex **3**

4. Crystallographic Data of 3

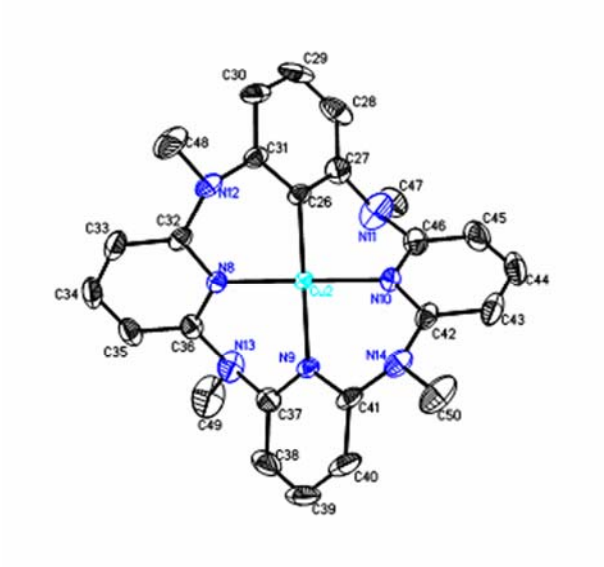
X-ray data of 3:



a **b**



c

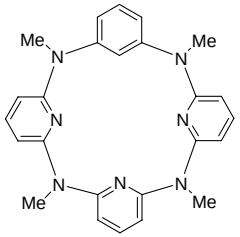


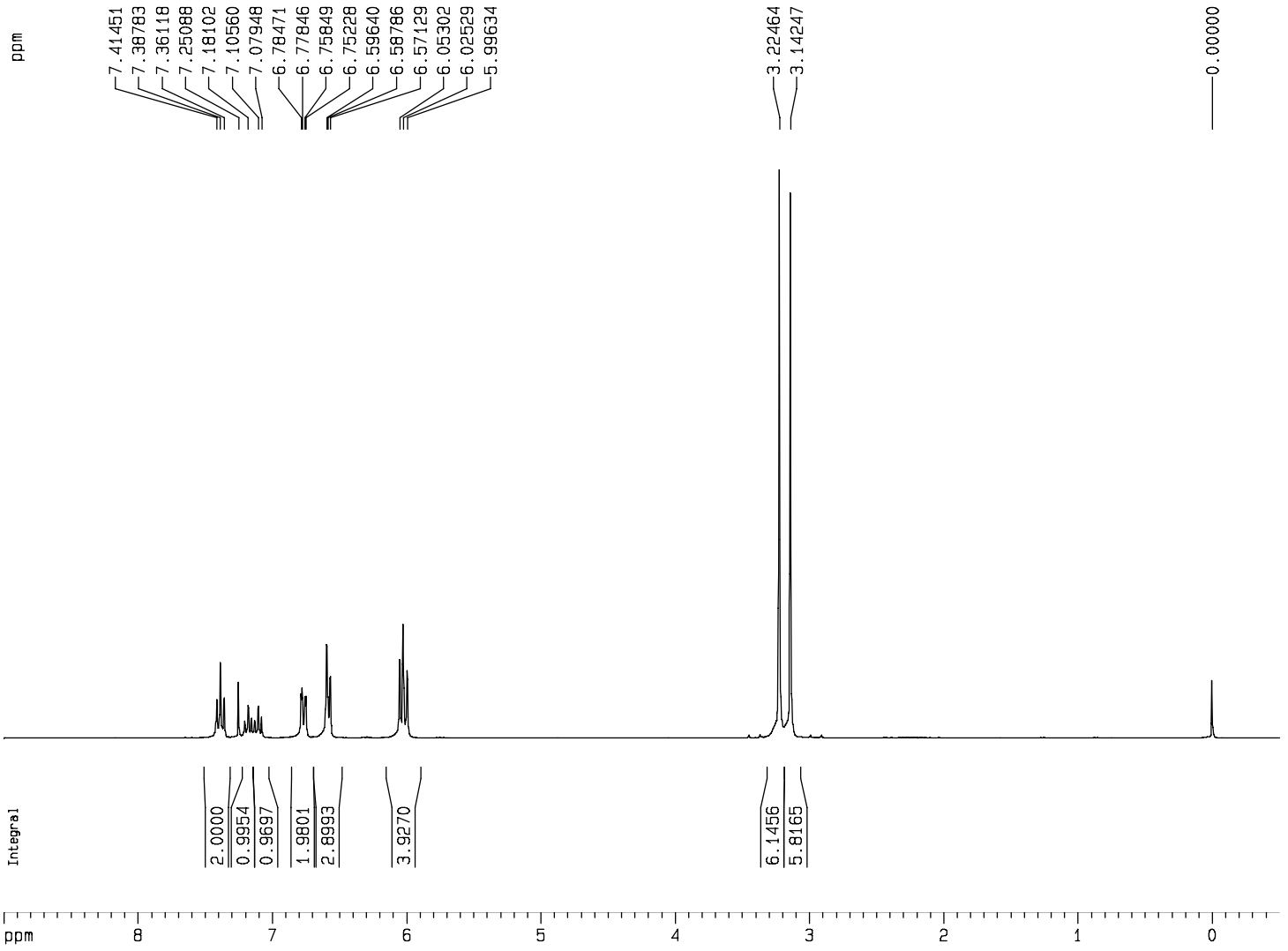
d

Figure S2. X-ray structure of the copper (III) complex **3** with 50% probability ellipsoids. Hydrogen atoms were omitted for clarity. **(a), (c)** is one complex with two perchlorate ions weakly coordinated with copper (III), and **(b), (d)** is another complex with only one perchlorate ion connected with copper(III); **(a), (b):** side view, and **(c), (d):** top view with perchlorate ions omitted. (Hydrogen atoms and solvent molecules were omitted for clarity)

5. Copies of ^{1}H and ^{13}C NMR Spectra of Compounds

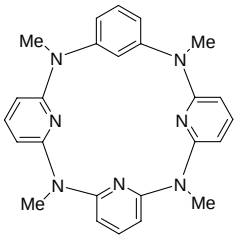
^{1}H NMR of **1**

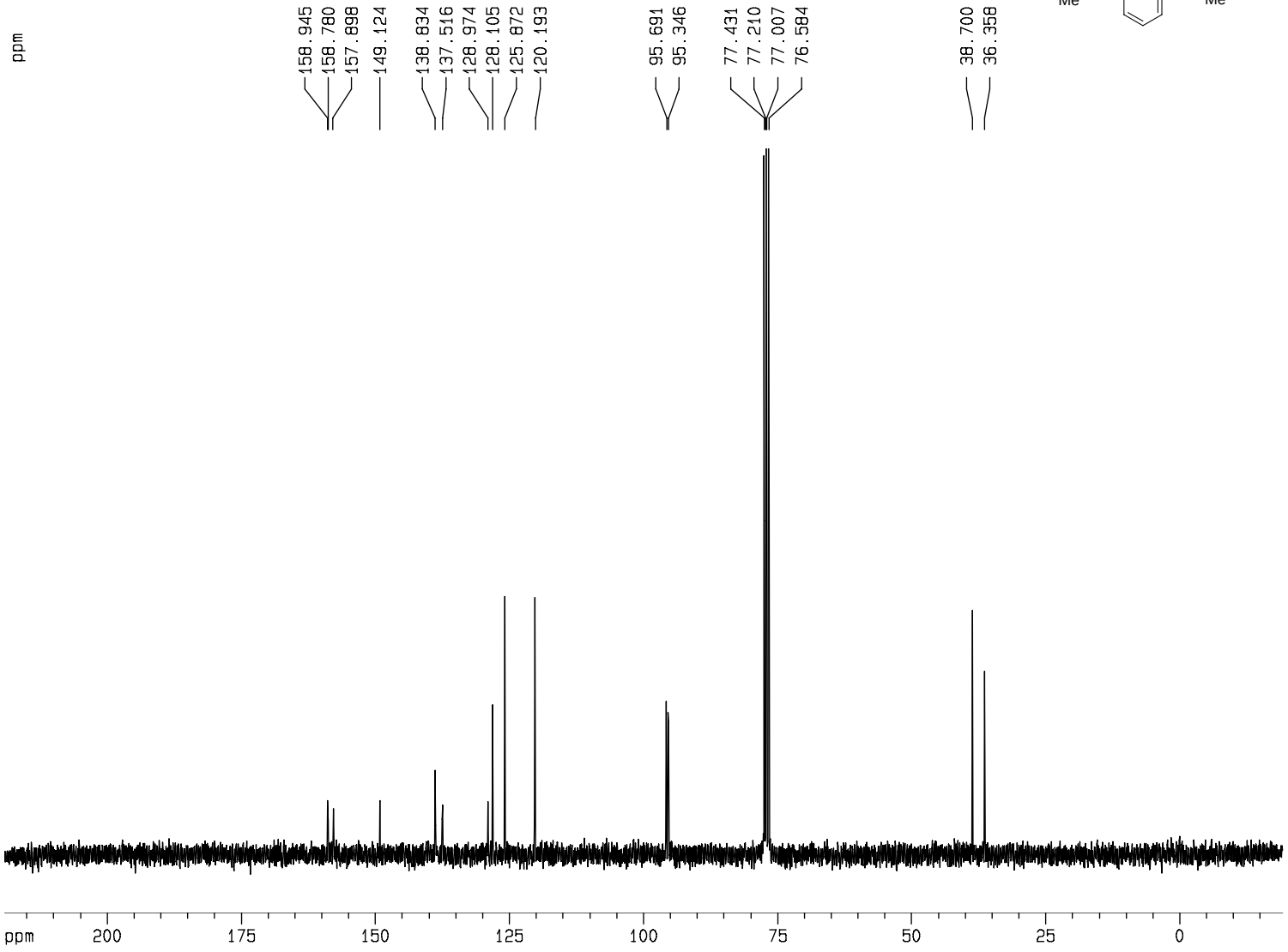





```
Current Data Parameters
NAME          4NMe-3Py1Ph
EXPNO                  20
PROCNO                  1

F2 - Acquisition Parameters
Date_           20081009
Time               12.07
INSTRUM            spect
PROBHD   5 mm DUL 13C-1
PULPROG            zg30
TD                65536
SOLVENT           CDCl3
NS                   16
DS                    0
SWH             8992.806 Hz
FIDRES          0.137219 Hz
AQ              3.6438515 sec
RG                  362
DW                55.600 usec
DE                 6.00 usec
TE                301.7 K
D1             1.00000000 sec
MCREST         0.00000000 sec
MCWRK          0.01500000 sec

======== CHANNEL f1 ========
NUC1                  1H
P1                 7.00 usec
PL1               -1.00 dB
SFO1         300.1324010 MHz

F2 - Processing parameters
SI                 32768
SF           300.1300087 MHz
WDW                   EM
SSB                    0
LB                  0.30 Hz
GB                     0
PC                  1.00

1D NMR plot parameters
CX                 22.00 cm
CY                 10.00 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
```

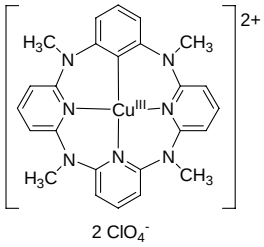
^{13}C NMR of **1**

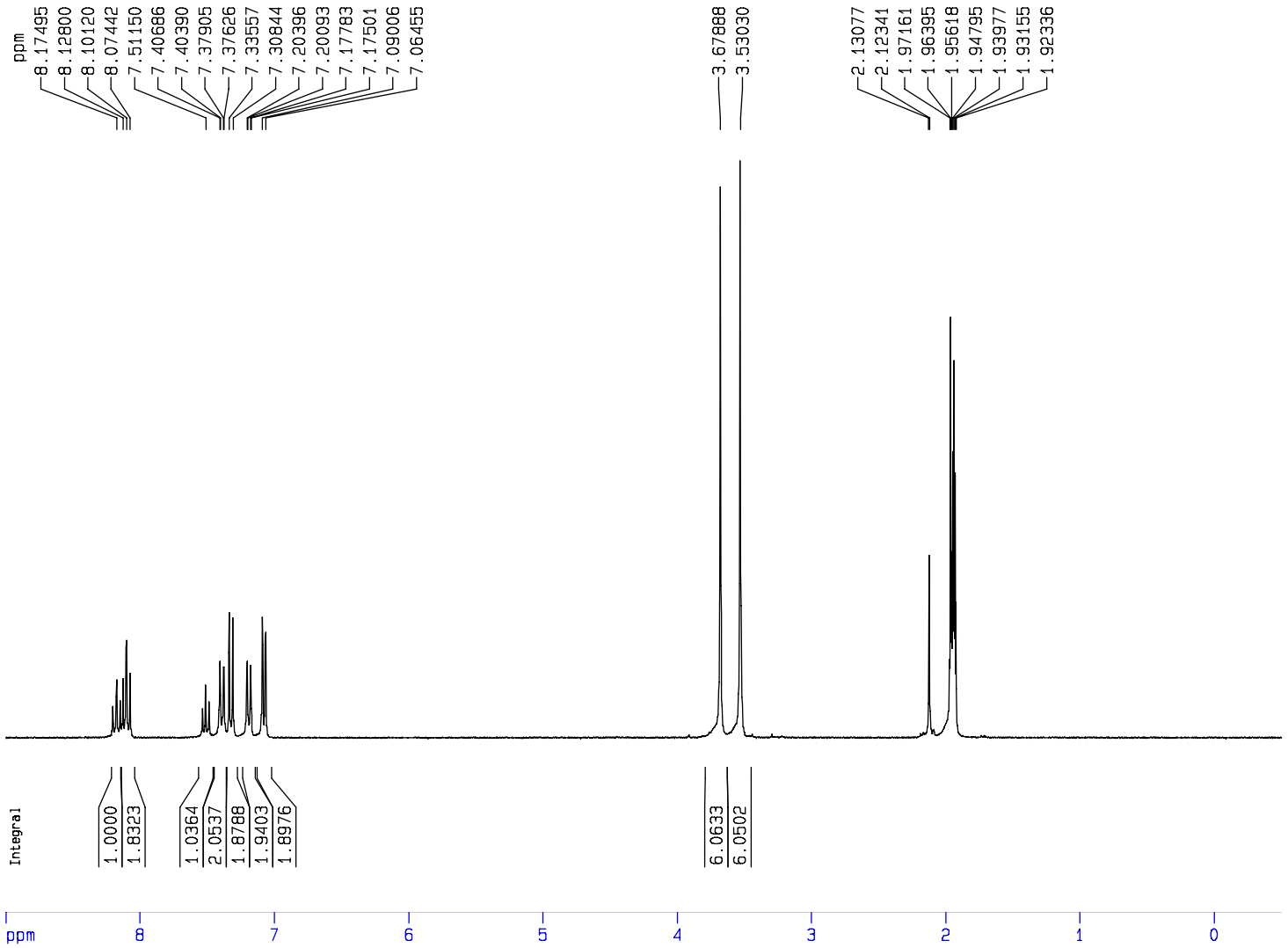





```
Current Data Parameters
NAME         4NMe-3Py1Ph
EXPNO                 21
PROCNO                 1

F2 - Acquisition Parameters
Date_           20081009
Time               12.15
INSTRUM            spect
PROBHD   5 mm DUL 13C-1
PULPROG          zgpg30
TD                65536
SOLVENT           CDCl3
NS                  346
DS                    4
SWH           17985.611 Hz
FIDRES         0.274439 Hz
AQ            1.8219508 sec
RG               1149.4
DW               27.800 usec
DE                6.00 usec
TE               302.1 K
D1           2.00000000 sec
d11          0.03000000 sec
DELTA        1.89999998 sec
MCREST       0.00000000 sec
MCWRK        0.01500000 sec

======== CHANNEL f1 ========
NUC1               13C
P1               12.50 usec
PL1               2.00 dB
SFO1         75.4752953 MHz

======== CHANNEL f2 ========
CPDPRG2         waltz16
NUC2               1H
PCPD2            80.00 usec
PL2              -1.00 dB
PL12             20.16 dB
PL13             16.98 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                12.50 cm
F1P              219.155 ppm
F1             16539.11 Hz
F2P              -19.167 ppm
F2             -1446.51 Hz
PPMCM           10.83281 ppm/cm
HZCM           817.52783 Hz/cm
```

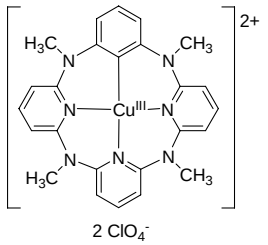
^{1}H NMR of **3**

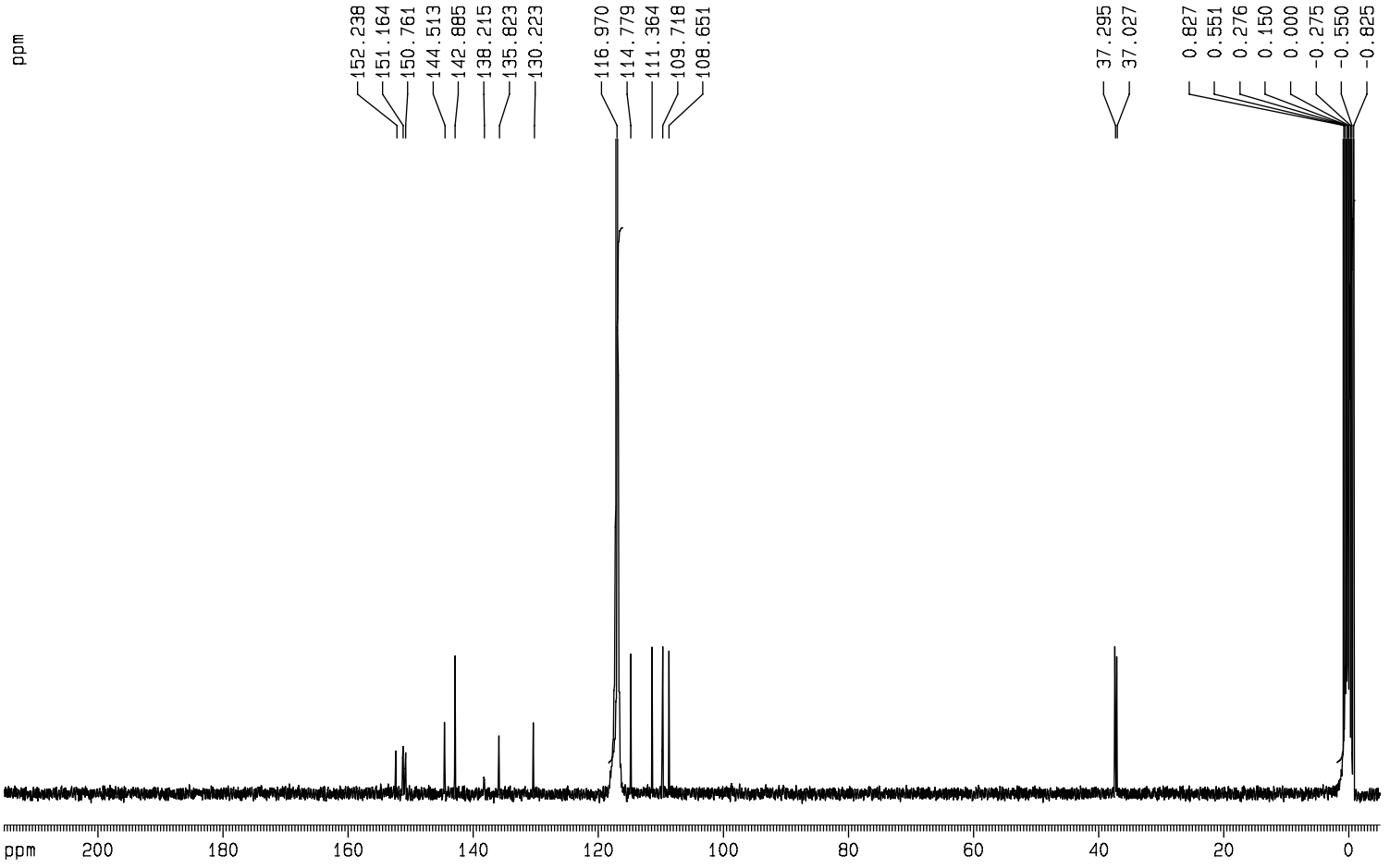





```
Current Data Parameters
NAME                  C-Cu
EXPNO                   20
PROCNO                   1

F2 - Acquisition Parameters
Date_              20081009
Time                  11.47
INSTRUM               spect
PROBHD   5 mm DUL 13C-1
PULPROG                zg30
TD                    65536
SOLVENT               CD3CN
NS                       16
DS                        0
SWH                8992.806 Hz
FIDRES             0.137219 Hz
AQ                 3.6438515 sec
RG                      362
DW                   55.600 usec
DE                    6.00 usec
TE                   300.4 K
D1                 1.00000000 sec
MCREST             0.00000000 sec
MCWRK              0.01500000 sec

======== CHANNEL f1 ========
NUC1                     1H
P1                    7.00 usec
PL1                  -1.00 dB
SFO1            300.1324010 MHz

F2 - Processing parameters
SI                    32768
SF              300.1300075 MHz
WDW                      EM
SSB                       0
LB                    0.30 Hz
GB                        0
PC                    1.00

1D NMR plot parameters
CX                   22.00 cm
CY                   10.00 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
```

^{13}C NMR of **3**



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Current Data Parameters
NAME                  C-Cu
EXPNO                   50
PROCNO                   1

F2 - Acquisition Parameters
Date_              20081012
Time                 22.19
INSTRUM              spect
PROBHD   5 mm DUL 13C-1
PULPROG             zgpg30
TD                   65536
SOLVENT              CD3CN
NS                    9282
DS                       4
SWH              17985.611 Hz
FIDRES           0.274439 Hz
AQ               1.8219508 sec
RG                 18390.4
DW                  27.800 usec
DE                   6.00 usec
TE                  301.2 K
D1               2.00000000 sec
d11              0.03000000 sec
DELTA            1.89999998 sec
MCREST           0.00000000 sec
MCWRK            0.01500000 sec

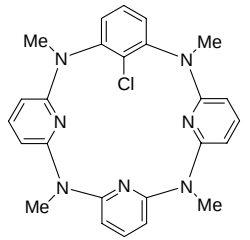
======== CHANNEL f1 ========
NUC1                   13C
P1                   12.50 usec
PL1                   2.00 dB
SFO1            75.4752953 MHz

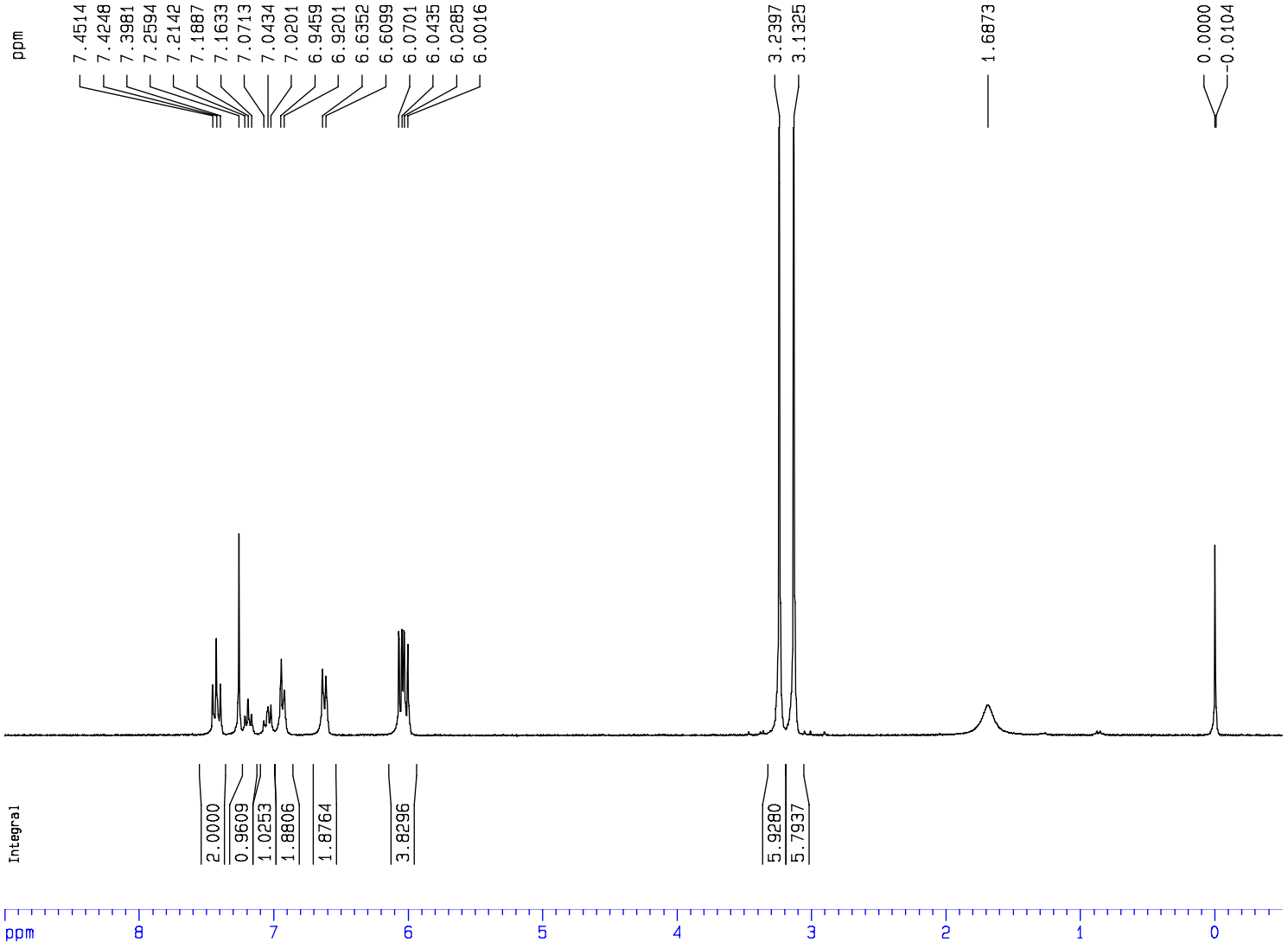
======== CHANNEL f2 ========
CPDPRG2             waltz16
NUC2                   1H
PCPD2                80.00 usec
PL2                  -1.00 dB
PL12                 20.16 dB
PL13                 16.98 dB
SFO2           300.1312005 MHz

F2 - Processing parameters
SI                    32768
SF               75.4677687 MHz
WDW                      EM
SSB                       0
LB                     1.00 Hz
GB                        0
PC                     1.40

1D NMR plot parameters
CX                    22.00 cm
CY                   100.00 cm
F1P                 215.000 ppm
F1                 16225.57 Hz
F2P                  -5.000 ppm
F2                 -377.34 Hz
PPMCM              10.00000 ppm/cm
HZCM              754.67767 Hz/cm
```

^{1}H NMR of **5a**






```
Current Data Parameters
NAME                  C-Cl
EXPNO                   10
PROCNO                   1

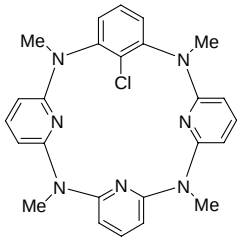
F2 - Acquisition Parameters
Date_              20080830
Time                  20.25
INSTRUM               spect
PROBHD    5 mm DUL 13C-1
PULPROG               zg30
TD                   65536
SOLVENT              CDCl3
NS                      16
DS                       0
SWH                8992.806 Hz
FIDRES             0.137219 Hz
AQ                 3.6438515 sec
RG                     362
DW                  55.600 usec
DE                   6.00 usec
TE                   299.2 K
D1                1.00000000 sec
MCREST            0.00000000 sec
MCWRK             0.01500000 sec

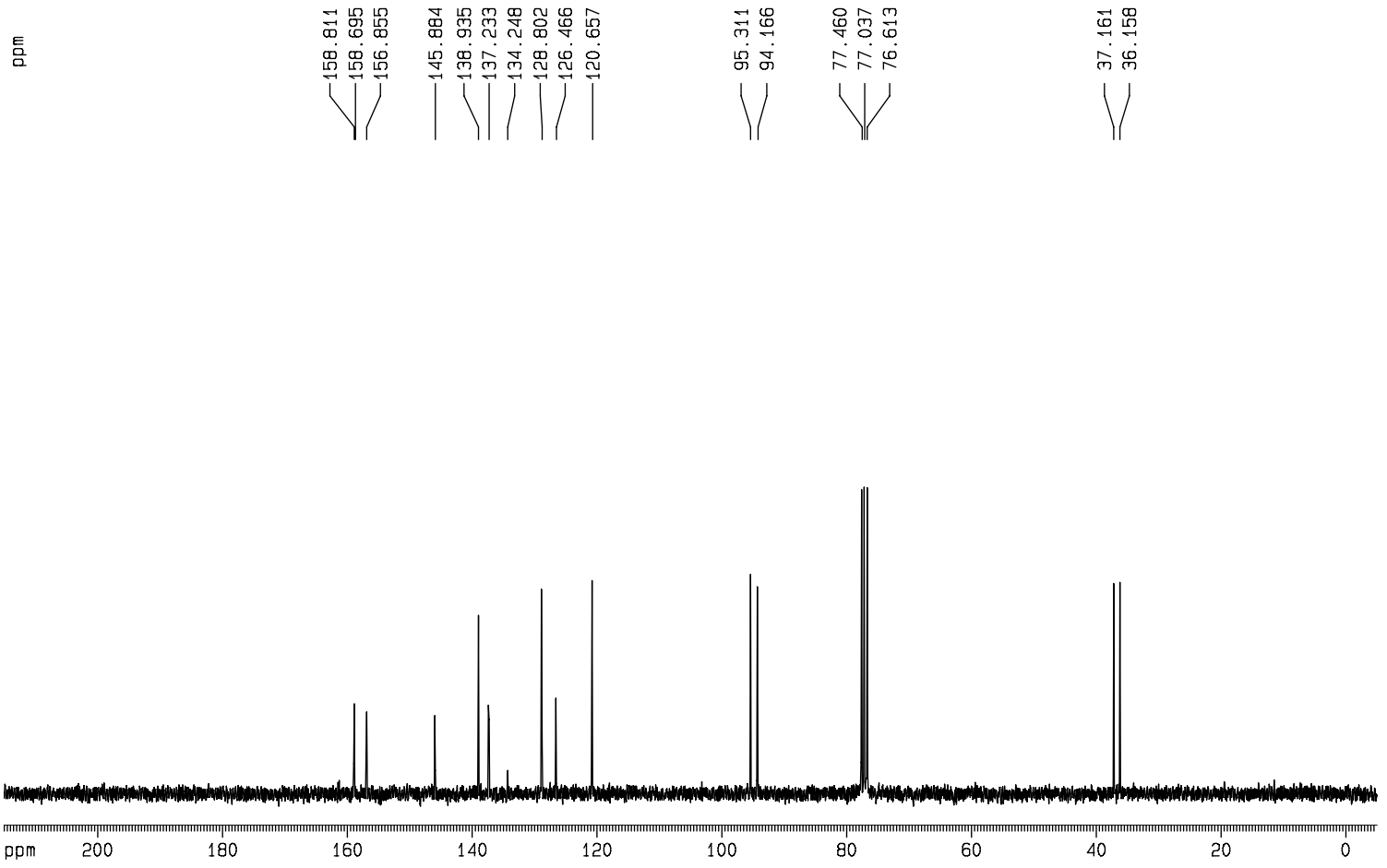
======== CHANNEL f1 ========
NUC1                    1H
P1                   7.00 usec
PL1                 -1.00 dB
SFO1           300.1324010 MHz

F2 - Processing parameters
SI                   32768
SF             300.1300065 MHz
WDW                     EM
SSB                      0
LB                    0.30 Hz
GB                       0
PC                    1.00

1D NMR plot parameters
CX                   22.00 cm
CY                   12.50 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
```

^{13}C NMR of **5a**






```
Current Data Parameters
NAME                  C-Cl
EXPNO                   20
PROCNO                   1

F2 - Acquisition Parameters
Date_              20080830
Time                 20.41
INSTRUM              spect
PROBHD   5 mm DUL 13C-1
PULPROG             zgpg30
TD                   65536
SOLVENT              CDCl3
NS                     236
DS                       4
SWH              17985.611 Hz
FIDRES           0.274439 Hz
AQ               1.8219508 sec
RG                   812.7
DW                  27.800 usec
DE                   6.00 usec
TE                  299.7 K
D1              2.00000000 sec
d11             0.03000000 sec
DELTA           1.89999998 sec
MCREST          0.00000000 sec
MCWRK           0.01500000 sec

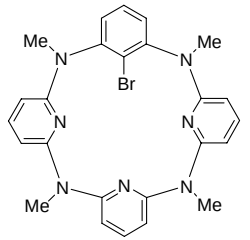
======== CHANNEL f1 ========
NUC1                   13C
P1                   12.50 usec
PL1                   2.00 dB
SFO1           75.4752953 MHz

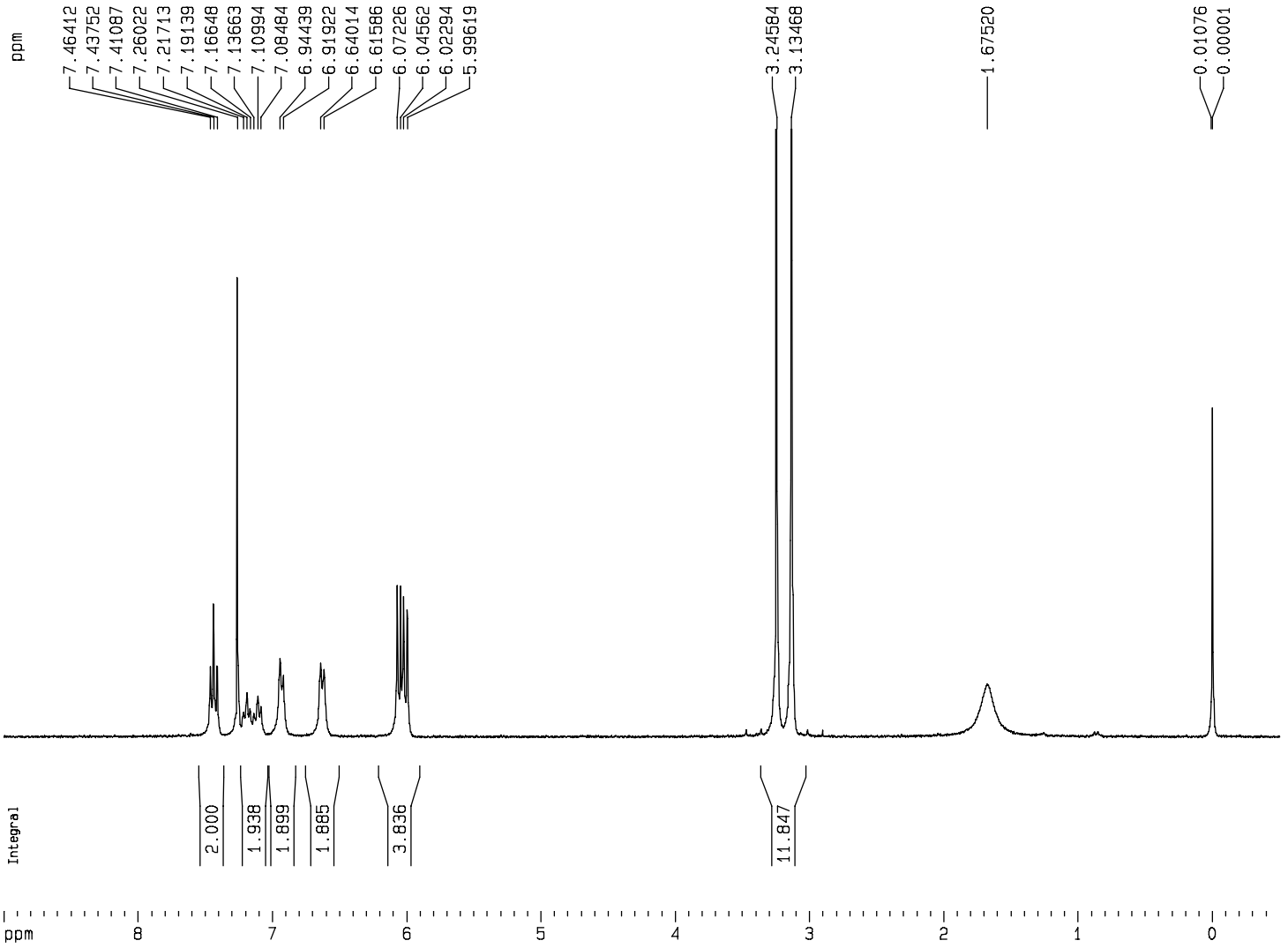
======== CHANNEL f2 ========
CPDPRG2            waltz16
NUC2                   1H
PCPD2               80.00 usec
PL2                 -1.00 dB
PL12                20.16 dB
PL13                16.98 dB
SFO2          300.1312005 MHz

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                  215.000 ppm
F1                 16225.57 Hz
F2P                   -5.000 ppm
F2                  -377.34 Hz
PPMCM               10.00000 ppm/cm
HZCM               754.67749 Hz/cm
```

^{1}H NMR of **5b**






```
Current Data Parameters
NAME                  C-Br
EXPNO                   10
PROCNO                   1

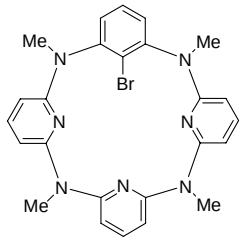
F2 - Acquisition Parameters
Date_              20080830
Time                 19.45
INSTRUM              spect
PROBHD   5 mm DUL 13C-1
PULPROG              zg30
TD                  65536
SOLVENT             CDCl3
NS                     16
DS                      0
SWH               8992.806 Hz
FIDRES            0.137219 Hz
AQ                3.6438515 sec
RG                    362
DW                 55.600 usec
DE                  6.00 usec
TE                 299.1 K
D1              1.00000000 sec
MCREST          0.00000000 sec
MCWRK           0.01500000 sec

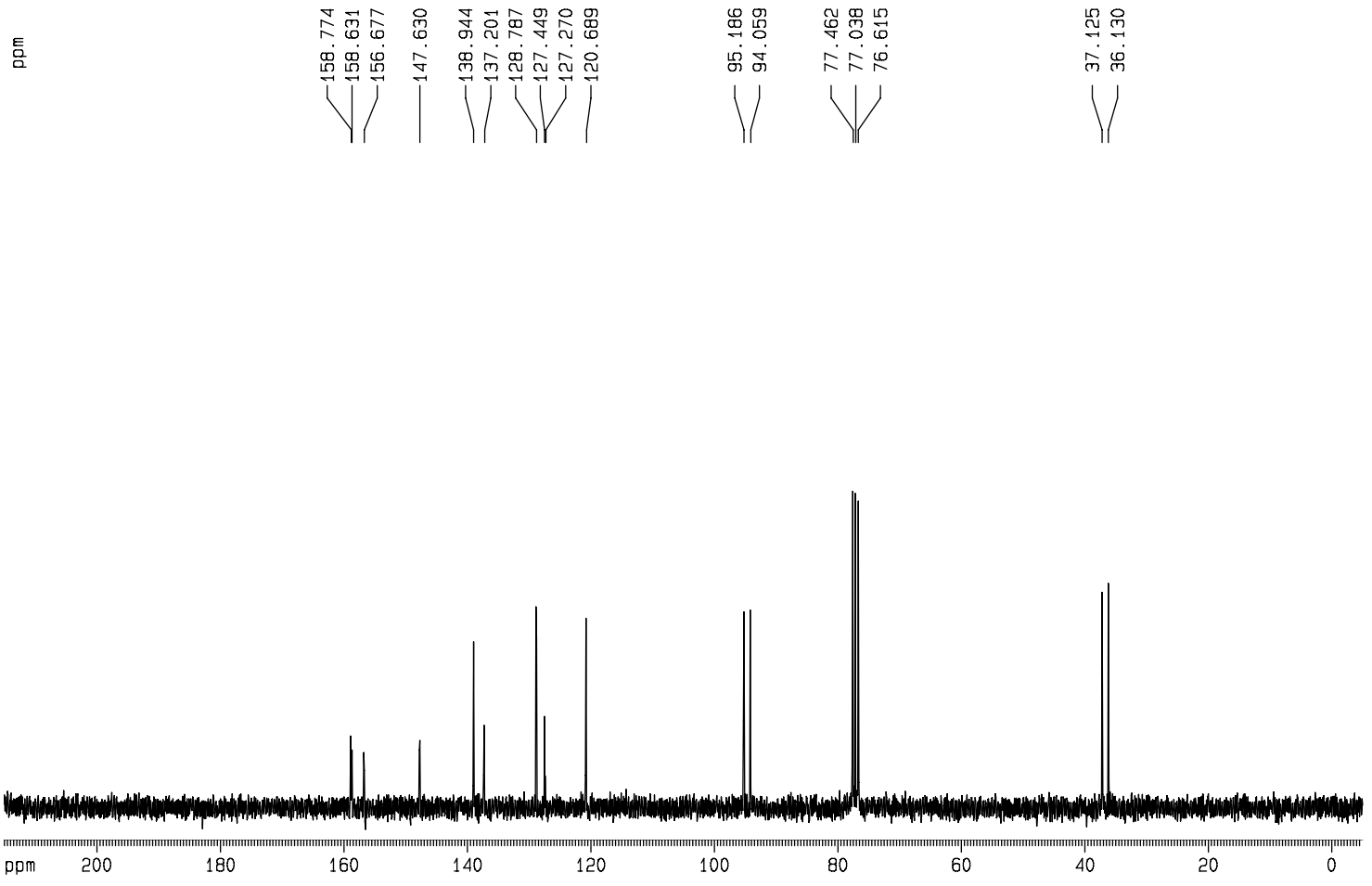
======== CHANNEL f1 ========
NUC1                   1H
P1                  7.00 usec
PL1                -1.00 dB
SFO1          300.1324010 MHz

F2 - Processing parameters
SI                    32768
SF             300.1300063 MHz
WDW                    EM
SSB                     0
LB                   0.30 Hz
GB                      0
PC                   1.00

1D NMR plot parameters
CX                  22.00 cm
CY                  20.00 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
```

^{13}C NMR of **5b**






```
Current Data Parameters
NAME                  C-Br
EXPNO                   20
PROCNO                   1

F2 - Acquisition Parameters
Date_           20080830
Time              20.08
INSTRUM           spect
PROBHD   5 mm DUL 13C-1
PULPROG          zgpg30
TD                65536
SOLVENT           CDCl3
NS                  110
DS                    4
SWH           17985.611 Hz
FIDRES        0.274439 Hz
AQ            1.8219508 sec
RG                 1024
DW               27.800 usec
DE                6.00 usec
TE               300.0 K
D1            2.00000000 sec
d11           0.03000000 sec
DELTA         1.89999998 sec
MCREST        0.00000000 sec
MCWRK         0.01500000 sec

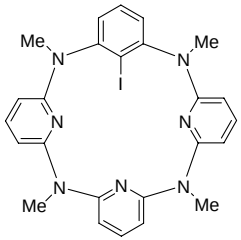
======== CHANNEL f1 ========
NUC1                 13C
P1                12.50 usec
PL1                2.00 dB
SFO1          75.4752953 MHz

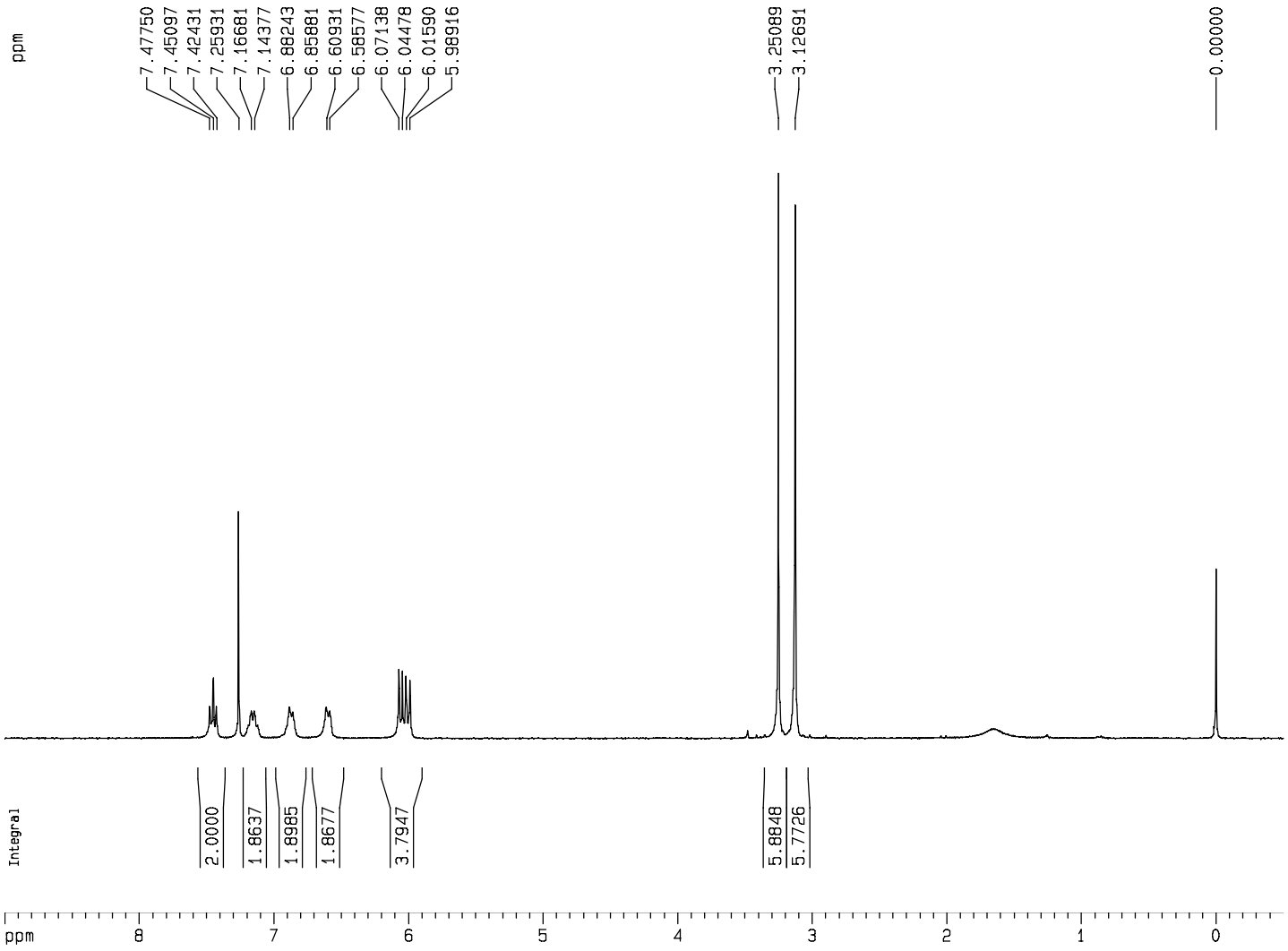
======== CHANNEL f2 ========
CPDPRG2          waltz16
NUC2                 1H
PCPD2             80.00 usec
PL2               -1.00 dB
PL12              20.15 dB
PL13              16.98 dB
SFO2         300.1312005 MHz

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              215.000 ppm
F1             16225.57 Hz
F2P               -5.000 ppm
F2              -377.34 Hz
PPMCM          10.00000 ppm/cm
HZCM          754.67749 Hz/cm
```

^{1}H NMR of **5c**






```
Current Data Parameters
NAME                  C-I
EXPNO                  71
PROCNO                  1

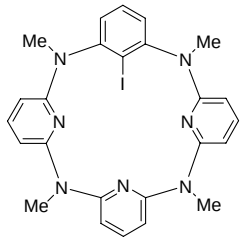
F2 - Acquisition Parameters
Date_           20080902
Time               20.53
INSTRUM            spect
PROBHD   5 mm DUL 13C-1
PULPROG             zg30
TD                 65536
SOLVENT            CDCl3
NS                    16
DS                     0
SWH             8992.806 Hz
FIDRES          0.137219 Hz
AQ              3.6438515 sec
RG                   362
DW                55.600 usec
DE                 6.00 usec
TE                299.6 K
D1             1.00000000 sec
MCREST         0.00000000 sec
MCWRK          0.01500000 sec

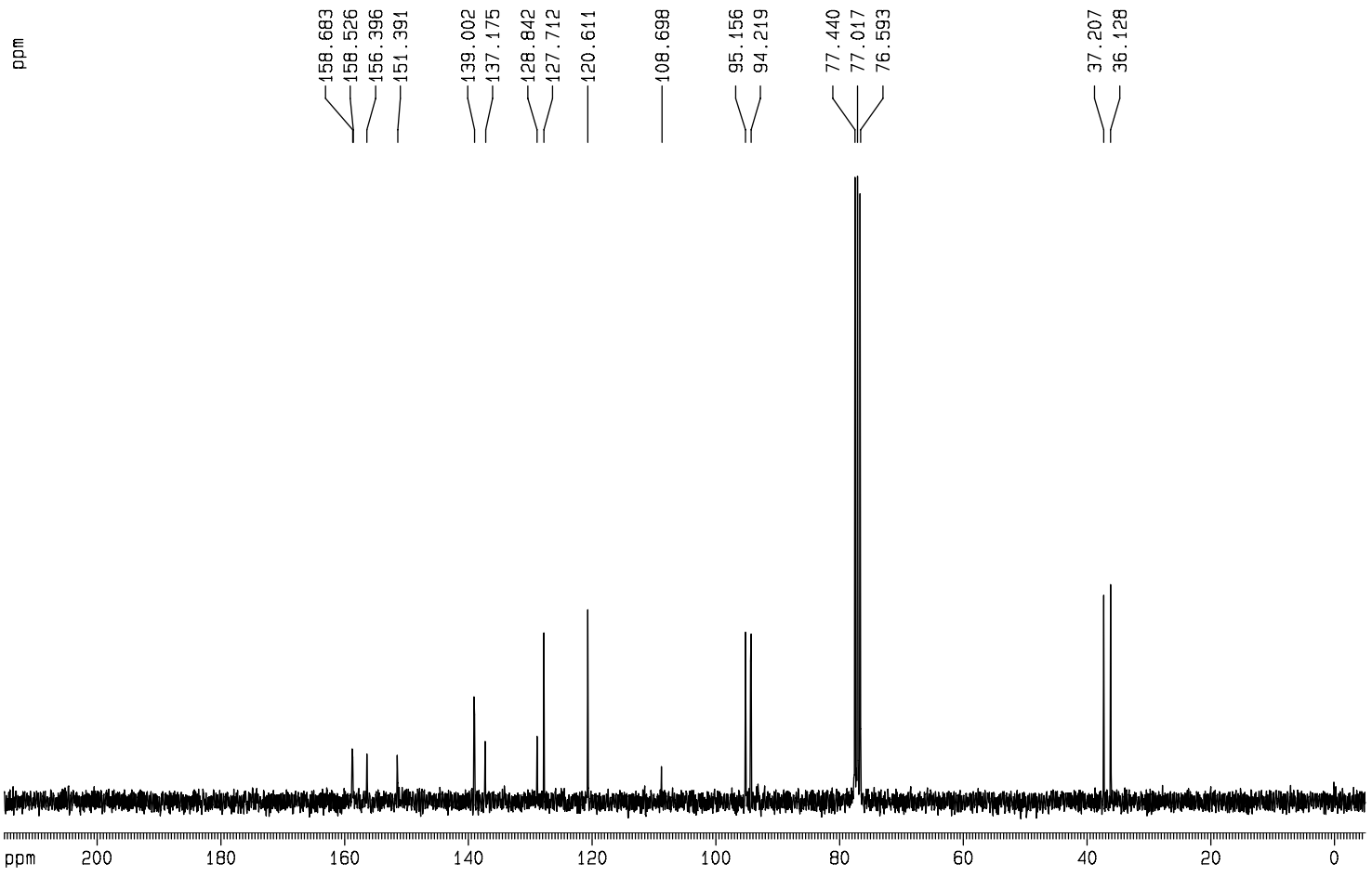
======== CHANNEL f1 ========
NUC1                  1H
P1                 7.00 usec
PL1               -1.00 dB
SFO1         300.1324010 MHz

F2 - Processing parameters
SI                 32768
SF           300.1300064 MHz
WDW                   EM
SSB                    0
LB                 0.30 Hz
GB                     0
PC                  1.00

1D NMR plot parameters
CX                22.00 cm
CY                10.00 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
```

^{13}C NMR of **5c**






```
Current Data Parameters
NAME                  C-I
EXPNO                  21
PROCNO                  1

F2 - Acquisition Parameters
Date_           20080830
Time               21.19
INSTRUM            spect
PROBHD   5 mm DUL 13C-1
PULPROG          zgpg30
TD                65536
SOLVENT             CDCl3
NS                    355
DS                      4
SWH             17985.611 Hz
FIDRES          0.274439 Hz
AQ              1.8219508 sec
RG                   1024
DW                27.800 usec
DE                 6.00 usec
TE                300.4 K
D1            2.00000000 sec
D11           0.03000000 sec
DELTA         1.89999998 sec
MCREST        0.00000000 sec
MCWRK         0.01500000 sec

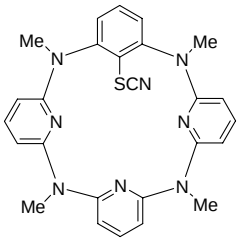
======== CHANNEL f1 ========
NUC1                  13C
P1                 12.50 usec
PL1                 2.00 dB
SFO1          75.4752953 MHz

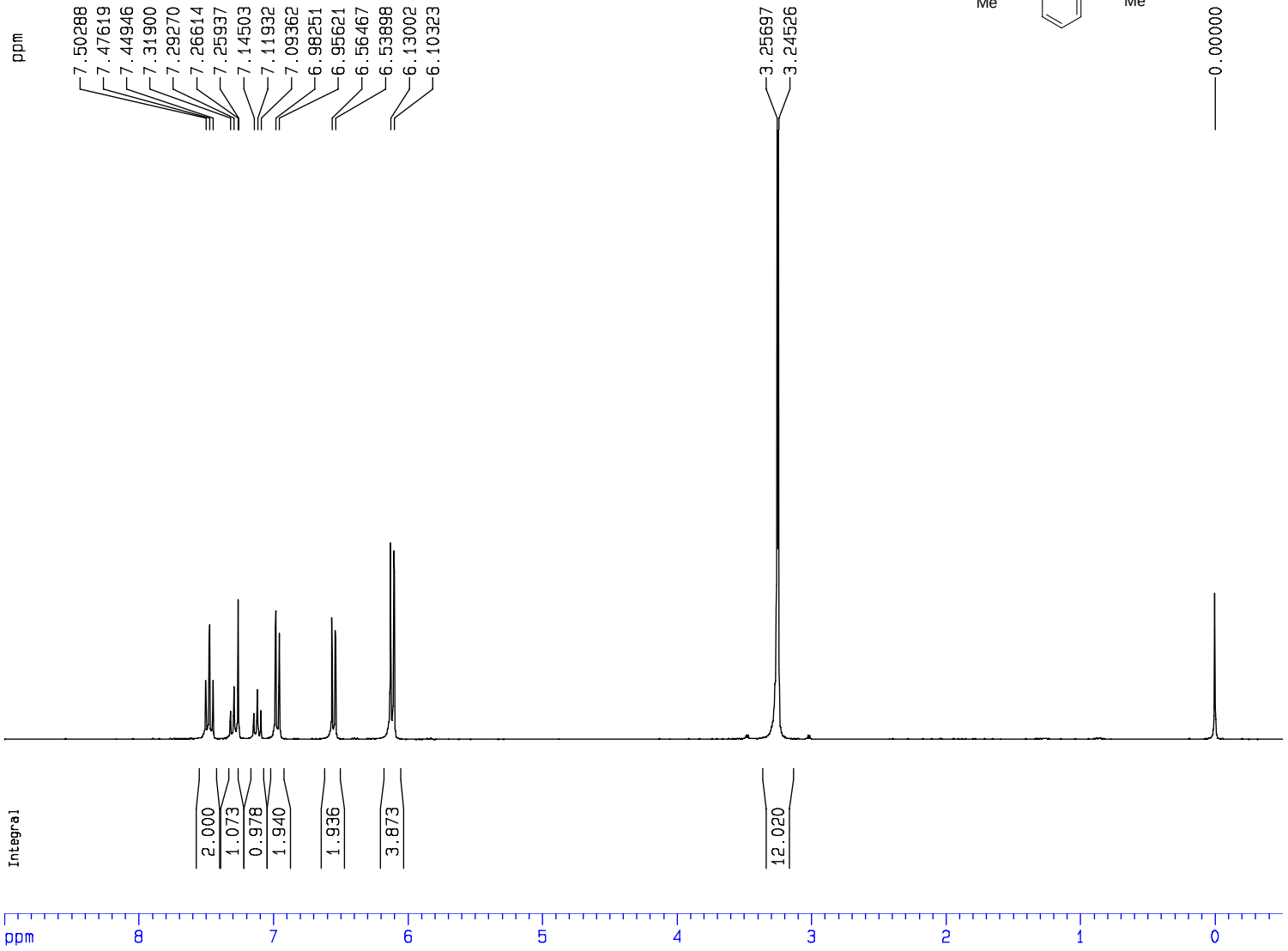
======== CHANNEL f2 ========
CPDPRG2          waltz16
NUC2                  1H
PCPD2              80.00 usec
PL2                -1.00 dB
PL12               20.16 dB
PL13               16.98 dB
SFO2         300.1312005 MHz

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                 10.00 cm
F1P               215.000 ppm
F1               16225.57 Hz
F2P                -5.000 ppm
F2                -377.34 Hz
PPMCM            10.00000 ppm/cm
HZCM            754.67749 Hz/cm
```

^{1}H NMR of **5d**



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Current Data Parameters
NAME                  C-SCN
EXPNO                    10
PROCNO                    1

F2 - Acquisition Parameters
Date_              20081118
Time                  13.13
INSTRUM               spect
PROBHD    5 mm DUL 13C-1
PULPROG                zg30
TD                    65536
SOLVENT               CDCl3
NS                       16
DS                        0
SWH                8992.806 Hz
FIDRES             0.137219 Hz
AQ                 3.6438515 sec
RG                      362
DW                   55.600 usec
DE                    6.00 usec
TE                   297.7 K
D1                1.00000000 sec
MCREST            0.00000000 sec
MCWRK             0.01500000 sec

======== CHANNEL f1 ========
NUC1                     1H
P1                    7.00 usec
PL1                  -1.00 dB
SFO1            300.1324010 MHz

F2 - Processing parameters
SI                    32768
SF              300.1300062 MHz
WDW                      EM
SSB                       0
LB                     0.30 Hz
GB                        0
PC                     1.00

1D NMR plot parameters
CX                    22.00 cm
CY                    12.50 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
```
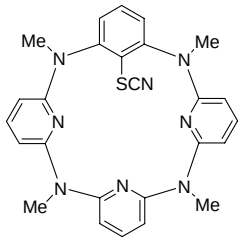
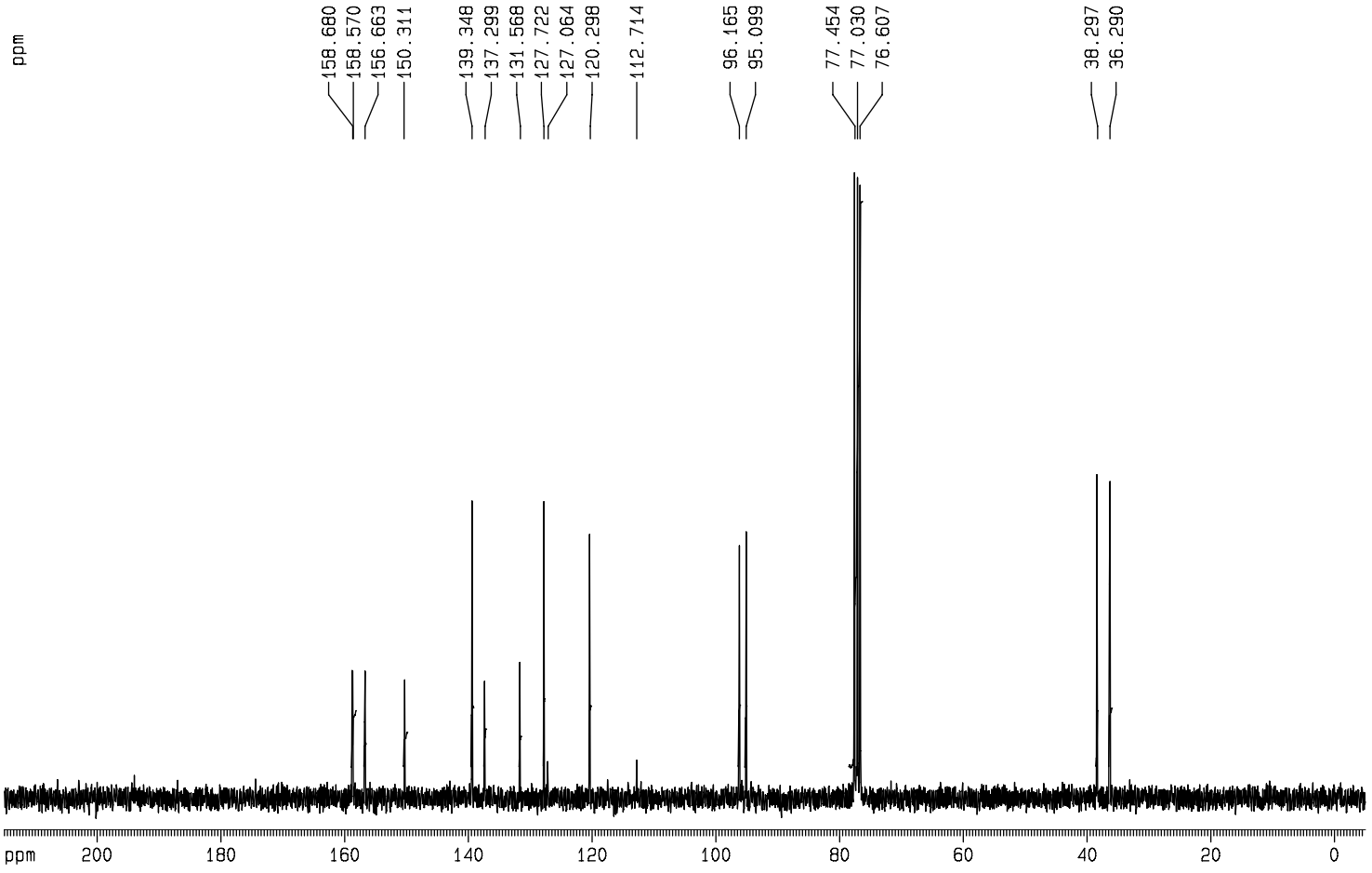

^{13}C NMR of **5d**



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Current Data Parameters
NAME                  C-SCN
EXPNO                    30
PROCNO                    1

F2 - Acquisition Parameters
Date_              20081119
Time                  12.11
INSTRUM               spect
PROBHD   5 mm DUL 13C-1
PULPROG              zgpg30
TD                    65536
SOLVENT               CDCl3
NS                      236
DS                        4
SWH                17985.611 Hz
FIDRES             0.274439 Hz
AQ                 1.8219508 sec
RG                    1149.4
DW                    27.800 usec
DE                     6.00 usec
TE                    298.7 K
D1                2.00000000 sec
D11               0.03000000 sec
DELTA             1.89999998 sec
MCREST            0.00000000 sec
MCWRK             0.01500000 sec

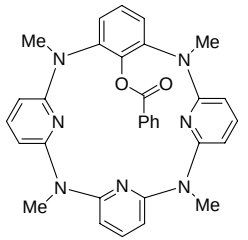
======== CHANNEL f1 ========
NUC1                    13C
P1                    12.50 usec
PL1                    2.00 dB
SFO1              75.4752953 MHz

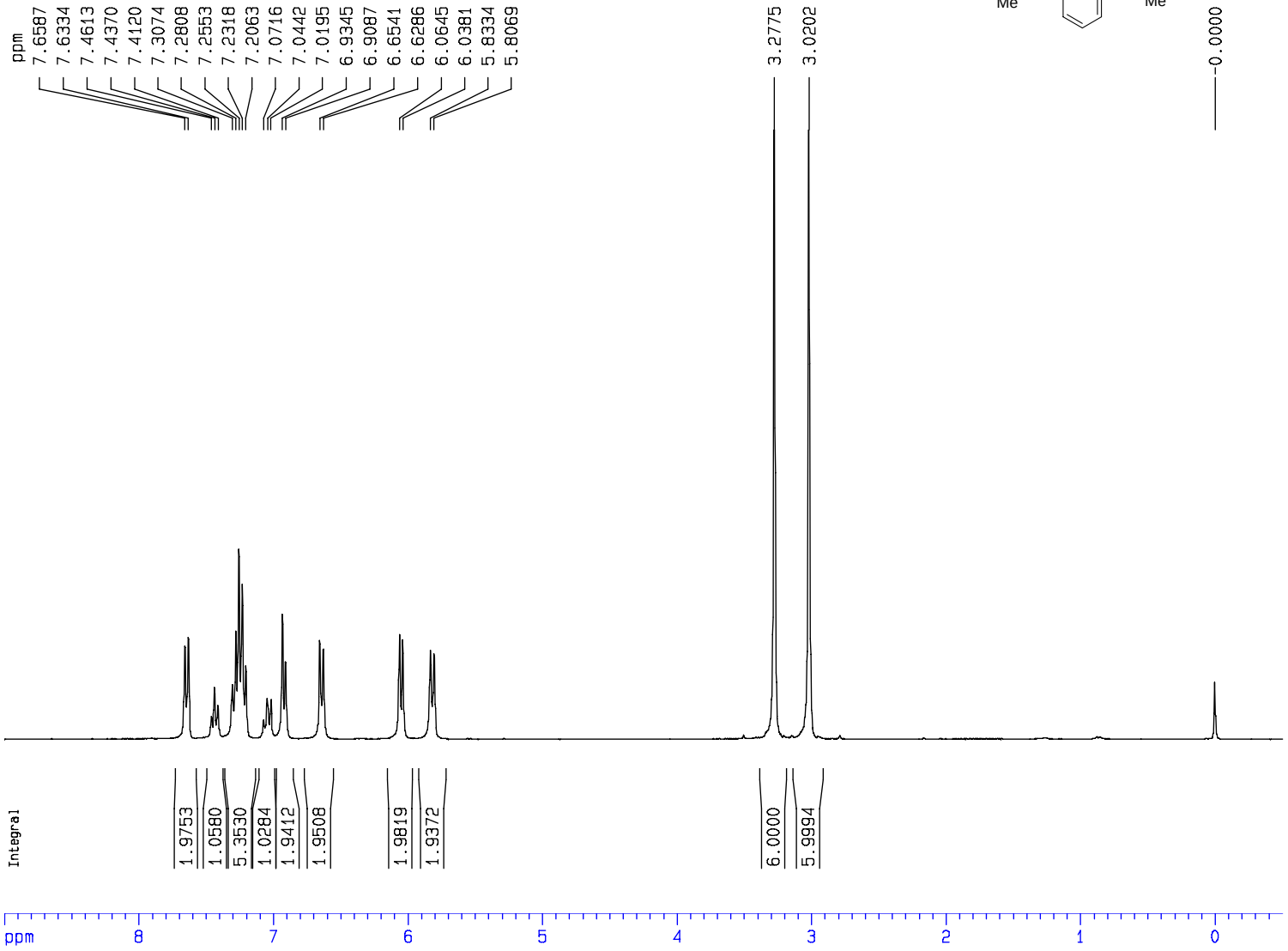
======== CHANNEL f2 ========
CPDPRG2             waltz16
NUC2                    1H
PCPD2                 80.00 usec
PL2                   -1.00 dB
PL12                  20.16 dB
PL13                  16.98 dB
SFO2             300.1312005 MHz

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                    10.00 cm
F1P                  215.000 ppm
F1                 16225.57 Hz
F2P                   -5.000 ppm
F2                  -377.34 Hz
PPMCM              10.00000 ppm/cm
HZCM              754.67749 Hz/cm
```

^{1}H NMR of **5e**



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Current Data Parameters
NAME                  C-OOCPh
EXPNO                      40
PROCNO                      1

F2 - Acquisition Parameters
Date_                20081123
Time                    22.44
INSTRUM                 spect
PROBHD    5 mm DUL 13C-1
PULPROG                  zg30
TD                      65536
SOLVENT                 CDCl3
NS                         16
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                    298.3 K
D1                1.00000000 sec
MCREST            0.00000000 sec
MCWRK             0.01500000 sec

======== CHANNEL f1 ========
NUC1                       1H
P1                    7.00 usec
PL1                  -1.00 dB
SFO1             300.1324010 MHz

F2 - Processing parameters
SI                      32768
SF                300.1300077 MHz
WDW                        EM
SSB                         0
LB                     0.30 Hz
GB                          0
PC                      1.00

1D NMR plot parameters
CX                     22.00 cm
CY                     12.50 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
```
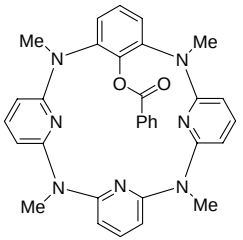
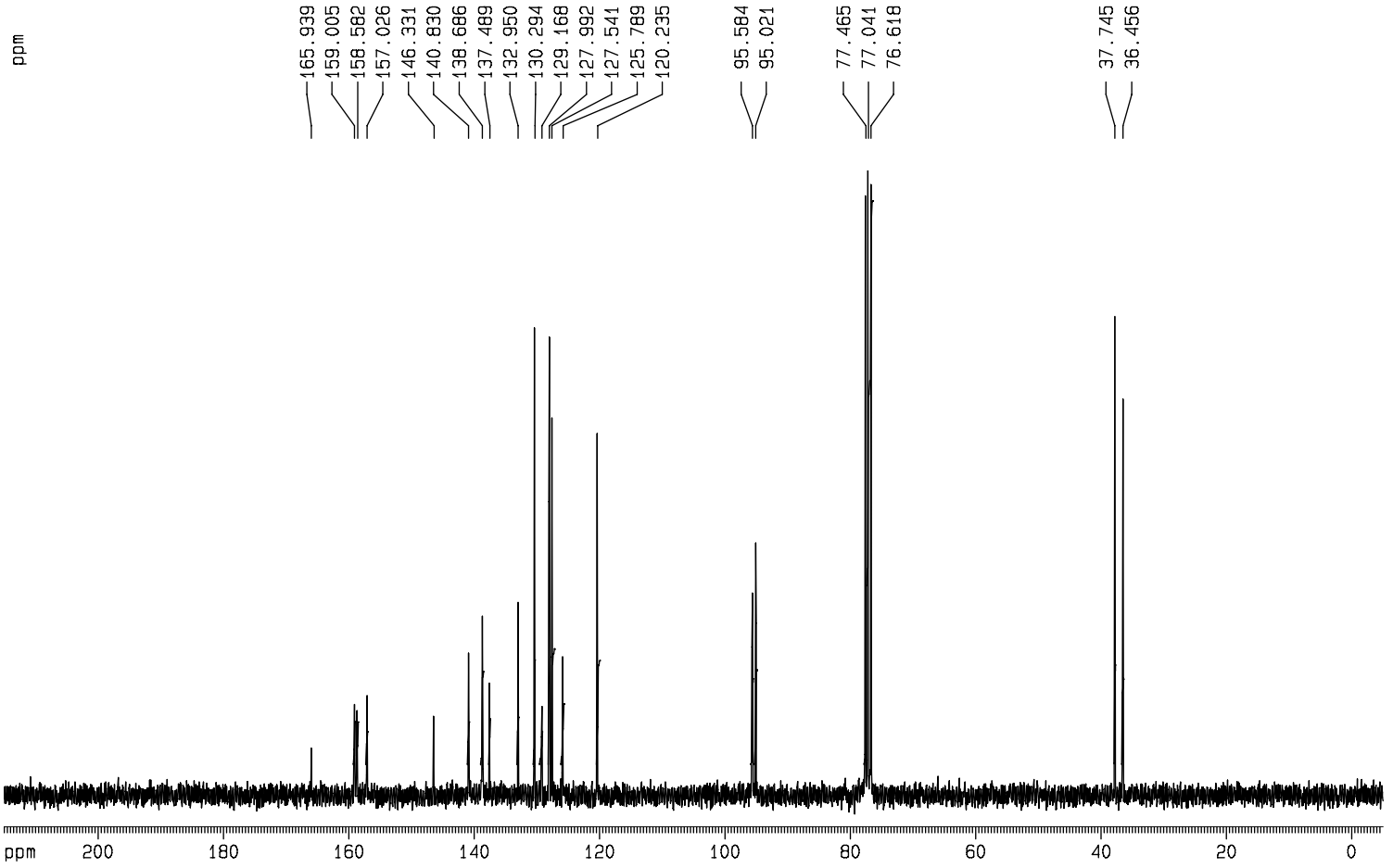
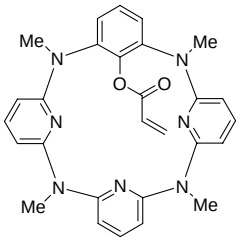

^{13}C NMR of **5e**

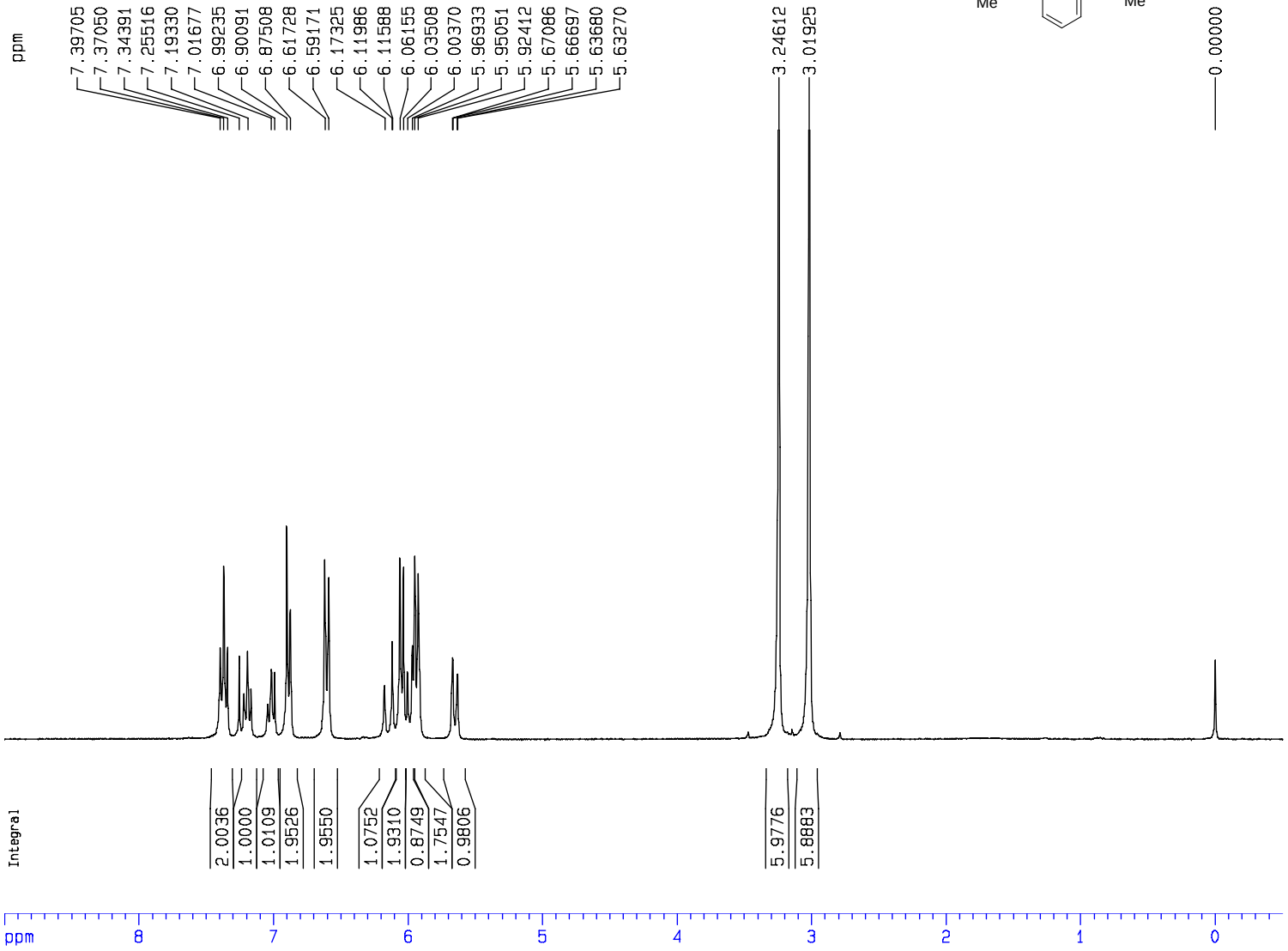





```
Current Data Parameters
NAME                  C-OOCPh
EXPNO                      50
PROCNO                      1

F2 - Acquisition Parameters
Date_               20081124
Time                   12.23
INSTRUM                spect
PROBHD   5 mm DUL 13C-1
PULPROG                zgpg30
TD                      65536
SOLVENT                CDCl3
NS                        231
DS                          4
SWH                17985.611 Hz
FIDRES             0.274439 Hz
AQ                 1.8219508 sec
RG                     812.7
DW                    27.800 usec
DE                     6.00 usec
TE                     299.0 K
D1                 2.00000000 sec
d11                0.03000000 sec
DELTA              1.89999998 sec
MCREST             0.00000000 sec
MCWRK              0.01500000 sec

======== CHANNEL f1 ========
NUC1                      13C
P1                      12.50 usec
PL1                      2.00 dB
SFO1               75.4752953 MHz

======== CHANNEL f2 ========
CPDPRG2                waltz16
NUC2                       1H
PCPD2                  80.00 usec
PL2                    -1.00 dB
PL12                   20.16 dB
PL13                   16.98 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                     10.00 cm
F1P                   215.000 ppm
F1                  16225.57 Hz
F2P                    -5.000 ppm
F2                   -377.34 Hz
PPMCM                10.00000 ppm/cm
HZCM                754.67749 Hz/cm
```

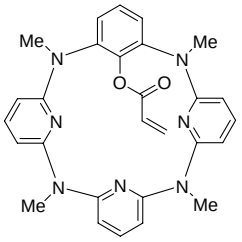
^{1}H NMR of **5f**

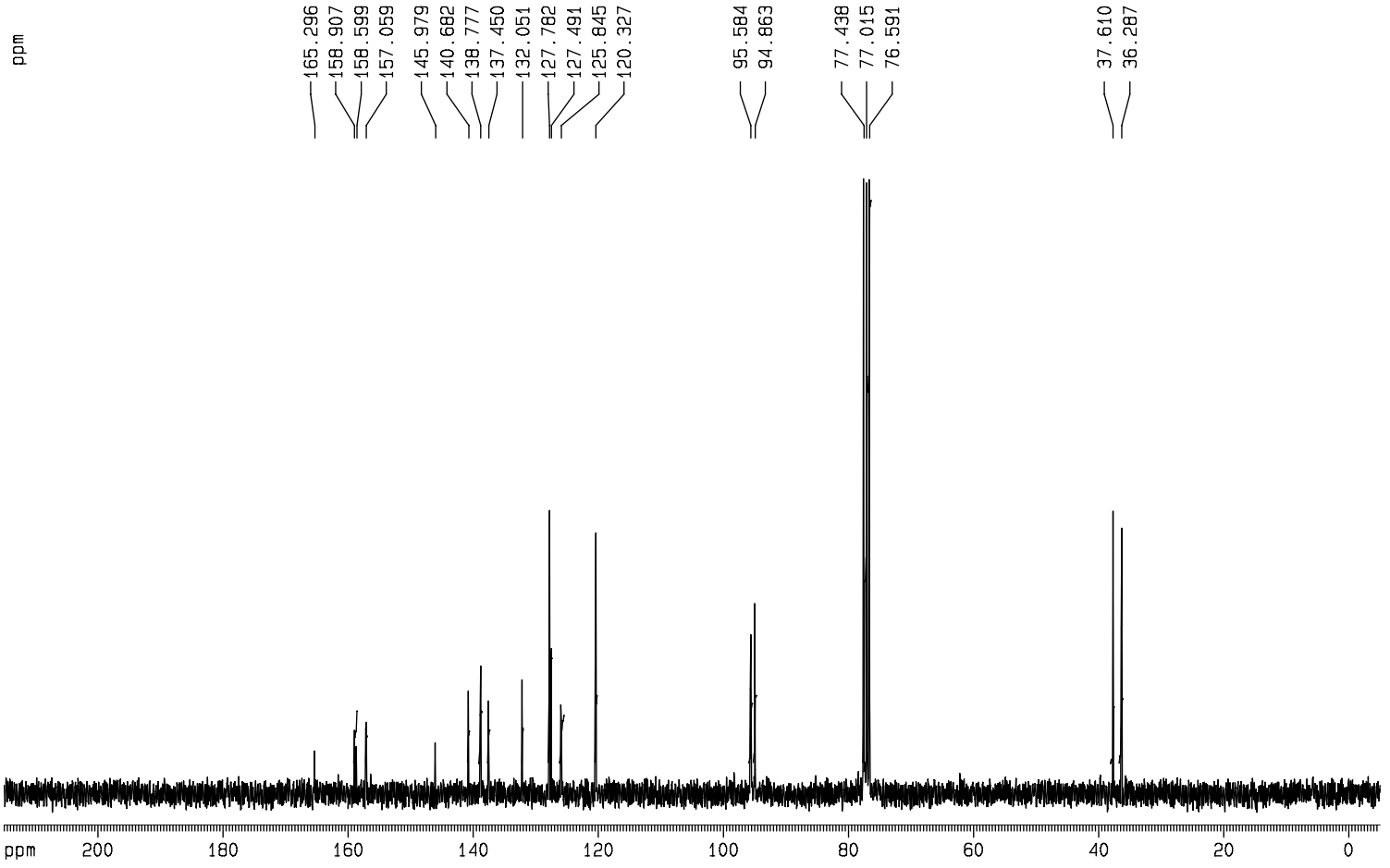





```
Current Data Parameters
NAME                  C-00CC
EXPNO                     21
PROCNO                     1

F2 - Acquisition Parameters
Date_              20081123
Time                  22.00
INSTRUM               spect
PROBHD    5 mm DUL 13C-1
PULPROG                zg30
TD                    65536
SOLVENT               CDCl3
NS                        9
DS                        0
SWH                8992.806 Hz
FIDRES             0.137219 Hz
AQ                 3.6438515 sec
RG                      181
DW                   55.600 usec
DE                    6.00 usec
TE                   298.9 K
D1                 1.00000000 sec
MCREST             0.00000000 sec
MCWRK              0.01500000 sec

======== CHANNEL f1 ========
NUC1                      1H
P1                    7.00 usec
PL1                  -1.00 dB
SFO1            300.1324010 MHz

F2 - Processing parameters
SI                     32768
SF              300.1300074 MHz
WDW                       EM
SSB                       0
LB                    0.30 Hz
GB                        0
PC                    1.00

1D NMR plot parameters
CX                    22.00 cm
CY                    20.00 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
```

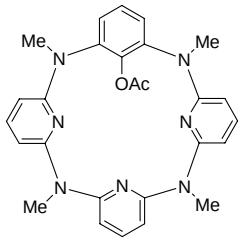
^{13}C NMR of **5f**

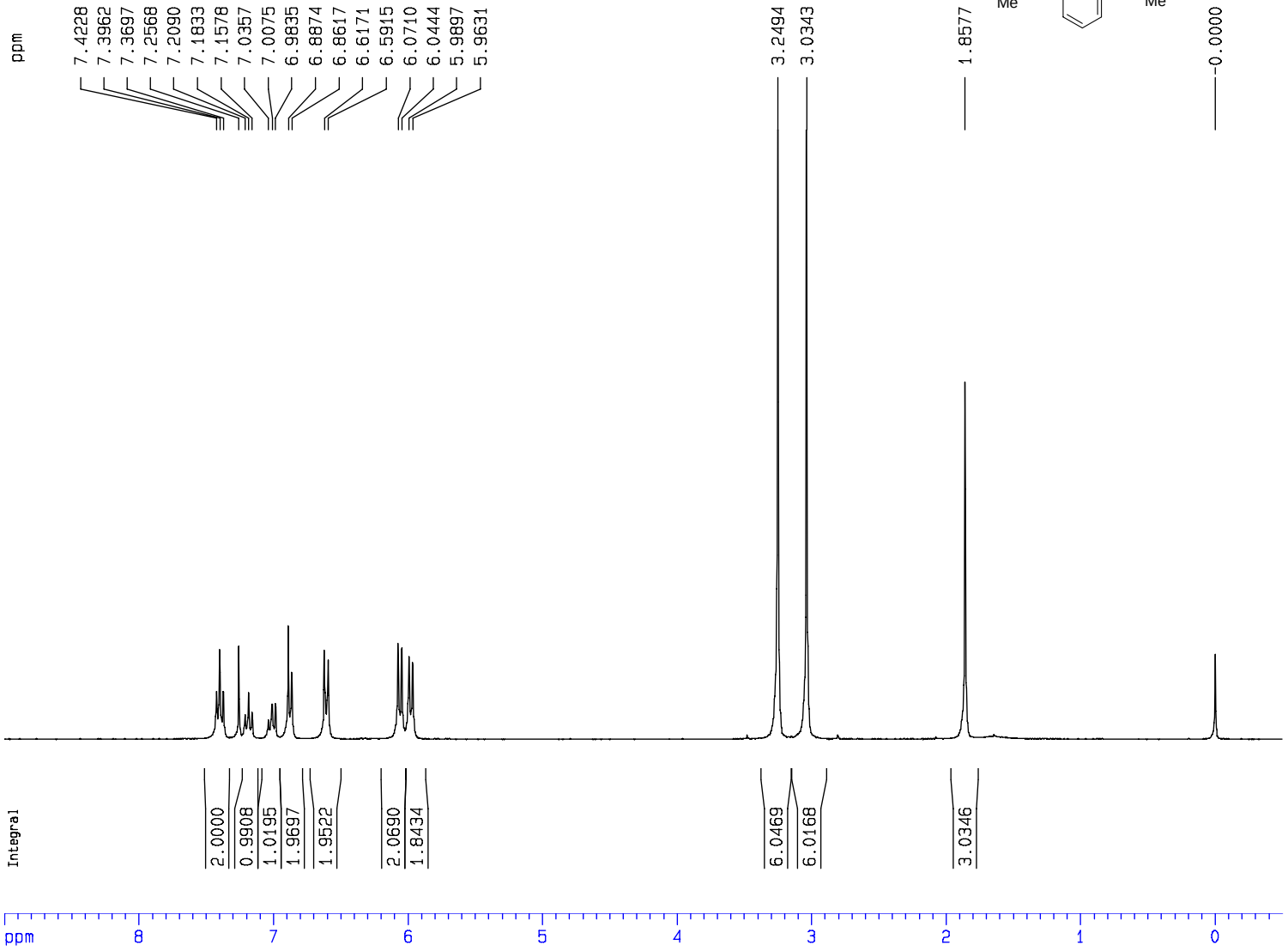





```
Current Data Parameters
NAME                  C-OOCC
EXPNO                     40
PROCNO                     1

F2 - Acquisition Parameters
Date_              20081124
Time                  13.01
INSTRUM               spect
PROBHD   5 mm DUL 13C-1
PULPROG             zgpg30
TD                    65536
SOLVENT               CDCl3
NS                      240
DS                        4
SWH               17985.611 Hz
FIDRES            0.274439 Hz
AQ                1.8219508 sec
RG                    645.1
DW                   27.800 usec
DE                    6.00 usec
TE                   301.1 K
D1              2.00000000 sec
d11             0.03000000 sec
DELTA           1.89999998 sec
MCREST          0.00000000 sec
MCWRK           0.01500000 sec

======== CHANNEL f1 ========
NUC1                    13C
P1                    12.50 usec
PL1                    2.00 dB
SFO1            75.4752953 MHz

======== CHANNEL f2 ========
CPDPRG2             waltz16
NUC2                    1H
PCPD2                80.00 usec
PL2                  -1.00 dB
PL12                 20.16 dB
PL13                 16.98 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                   10.00 cm
F1P                215.000 ppm
F1                 16225.57 Hz
F2P                 -5.000 ppm
F2                 -377.34 Hz
PPMCM             10.00000 ppm/cm
HZCM             754.67749 Hz/cm
```

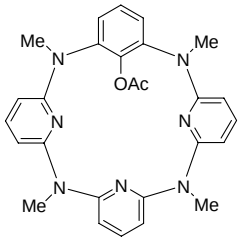
^{1}H NMR of **5g**

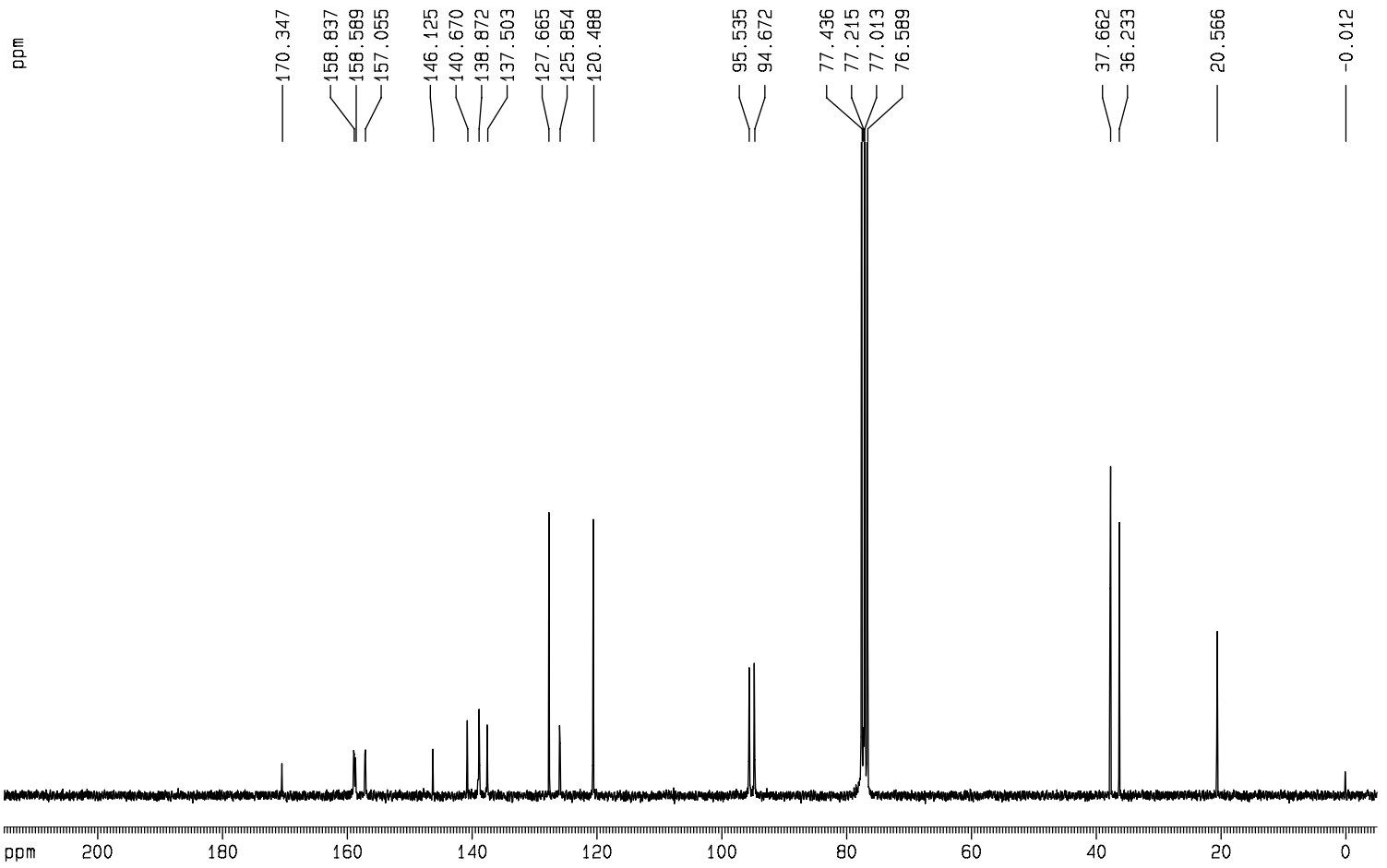





```
Current Data Parameters
NAME                  C-OAc
EXPNO                    21
PROCNO                    1

F2 - Acquisition Parameters
Date_              20080831
Time                  23.14
INSTRUM               spect
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                      362
DW                   55.600 usec
DE                    6.00 usec
TE                   299.9 K
D1                1.00000000 sec
MCREST            0.00000000 sec
MCWRK             0.01500000 sec

======== CHANNEL f1 ========
NUC1                     1H
P1                    7.00 usec
PL1                  -1.00 dB
SFO1           300.1324010 MHz

F2 - Processing parameters
SI                    32768
SF             300.1300071 MHz
WDW                      EM
SSB                       0
LB                    0.30 Hz
GB                        0
PC                    1.00

1D NMR plot parameters
CX                   22.00 cm
CY                   12.50 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
```

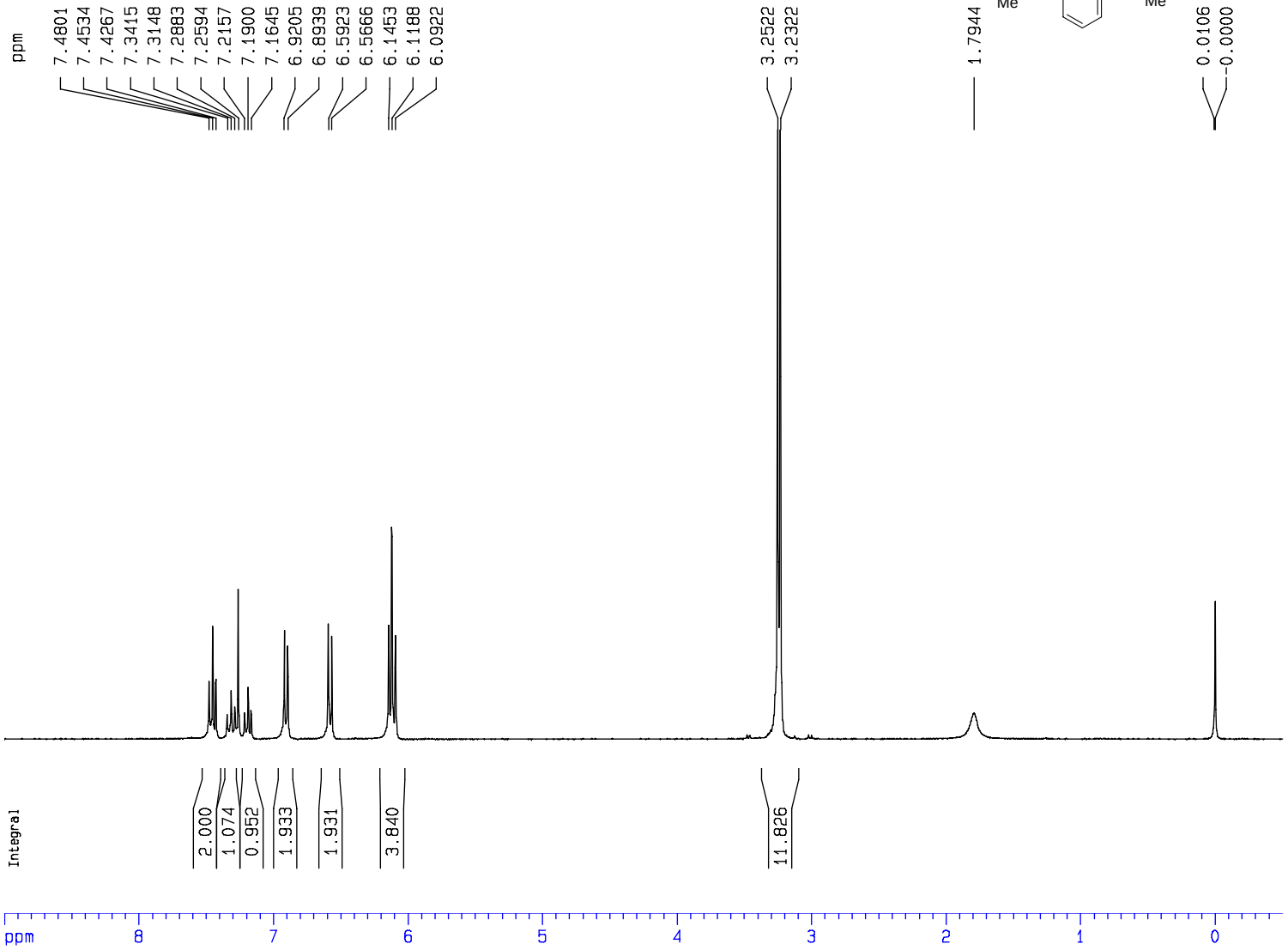
^{13}C NMR of **5g**

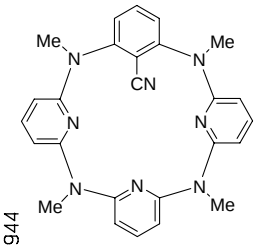





```
Current Data Parameters
NAME                  C-OAc
EXPNO                    23
PROCNO                    1

F2 - Acquisition Parameters
Date_              20080831
Time                  23.37
INSTRUM               spect
PROBHD   5 mm DUL 13C-1
PULPROG             zgpg30
TD                   65536
SOLVENT              CDCl3
NS                    7538
DS                       4
SWH               17985.611 Hz
FIDRES            0.274439 Hz
AQ                1.8219508 sec
RG                    1024
DW                  27.800 usec
DE                   6.00 usec
TE                   300.1 K
D1              2.00000000 sec
d11             0.03000000 sec
DELTA           1.89999998 sec
MCREST          0.00000000 sec
MCWRK           0.01500000 sec

======== CHANNEL f1 ========
NUC1                   13C
P1                   12.50 usec
PL1                   2.00 dB
SFO1            75.4752953 MHz

======== CHANNEL f2 ========
CPDPRG2            waltz16
NUC2                   1H
PCPD2                80.00 usec
PL2                  -1.00 dB
PL12                 20.16 dB
PL13                 16.98 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                   20.00 cm
F1P                215.000 ppm
F1                 16225.57 Hz
F2P                 -5.000 ppm
F2                 -377.34 Hz
PPMCM             10.00000 ppm/cm
HZCM             754.67749 Hz/cm
```

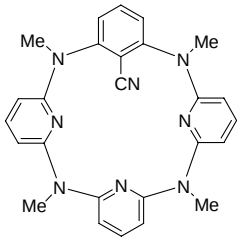
^{1}H NMR of **5h**

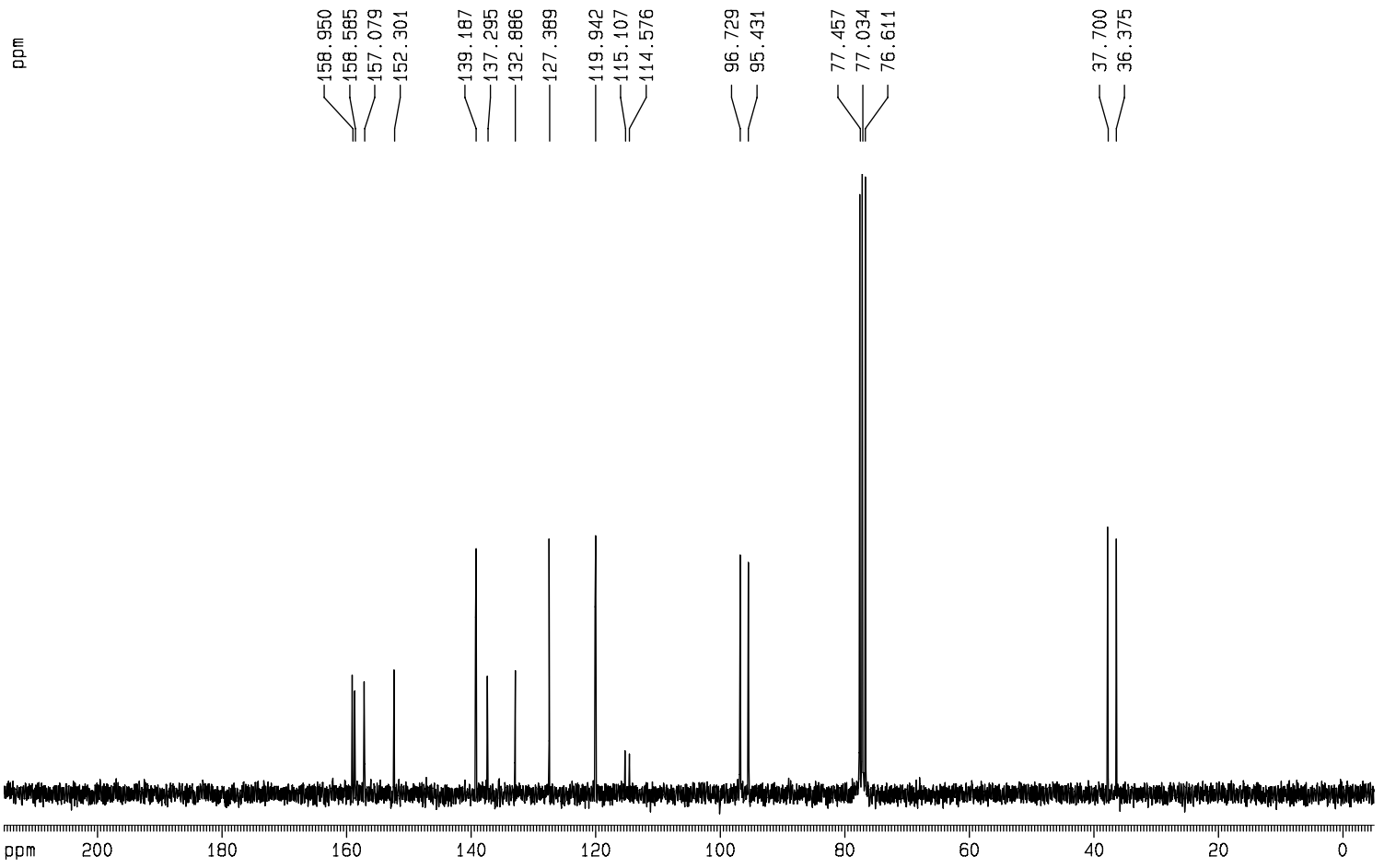





```
Current Data Parameters
NAME                  C-CN
EXPNO                   10
PROCNO                   1

F2 - Acquisition Parameters
Date_              20080830
Time                 20.31
INSTRUM              spect
PROBHD    5 mm DUL 13C-1
PULPROG              zg30
TD                  65536
SOLVENT             CDCl3
NS                     16
DS                      0
SWH              8992.806 Hz
FIDRES           0.137219 Hz
AQ               3.6438515 sec
RG                    362
DW                 55.600 usec
DE                  6.00 usec
TE                 299.4 K
D1              1.00000000 sec
MCREST          0.00000000 sec
MCWRK           0.01500000 sec

======== CHANNEL f1 ========
NUC1                   1H
P1                  7.00 usec
PL1                -1.00 dB
SFO1          300.1324010 MHz

F2 - Processing parameters
SI                  32768
SF            300.1300064 MHz
WDW                    EM
SSB                     0
LB                   0.30 Hz
GB                      0
PC                   1.00

1D NMR plot parameters
CX                  22.00 cm
CY                  12.50 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
```

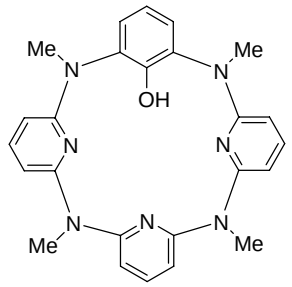
^{13}C NMR of **5h**

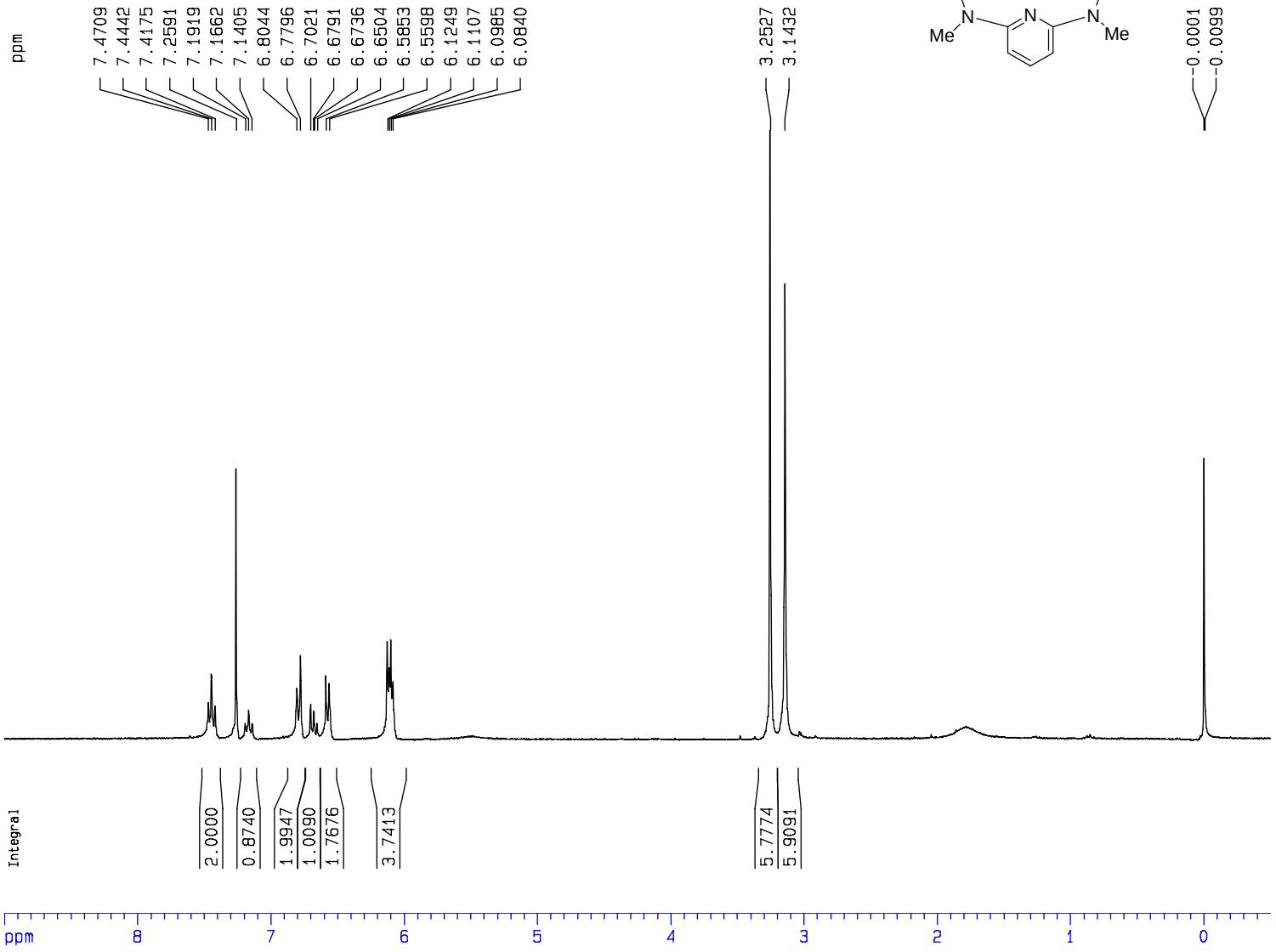





```
Current Data Parameters
NAME                  C-CN
EXPNO                   20
PROCNO                   1

F2 - Acquisition Parameters
Date_              20080830
Time                 21.47
INSTRUM              spect
PROBHD   5 mm DUL 13C-1
PULPROG             zgpg30
TD                   65536
SOLVENT              CDCl3
NS                     214
DS                       4
SWH              17985.611 Hz
FIDRES           0.274439 Hz
AQ               1.8219508 sec
RG                   645.1
DW                  27.800 usec
DE                   6.00 usec
TE                  299.8 K
D1              2.00000000 sec
d11             0.03000000 sec
DELTA           1.89999998 sec
MCREST          0.00000000 sec
MCWRK           0.01500000 sec

======== CHANNEL f1 ========
NUC1                   13C
P1                   12.50 usec
PL1                   2.00 dB
SFO1           75.4752953 MHz

======== CHANNEL f2 ========
CPDPRG2            waltz16
NUC2                   1H
PCPD2                80.00 usec
PL2                  -1.00 dB
PL12                 20.16 dB
PL13                 16.98 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                   10.00 cm
F1P                215.000 ppm
F1                 16225.57 Hz
F2P                 -5.000 ppm
F2                 -377.34 Hz
PPMCM              10.00000 ppm/cm
HZCM              754.67749 Hz/cm
```

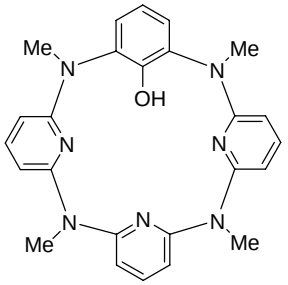
^{1}H NMR of **6**

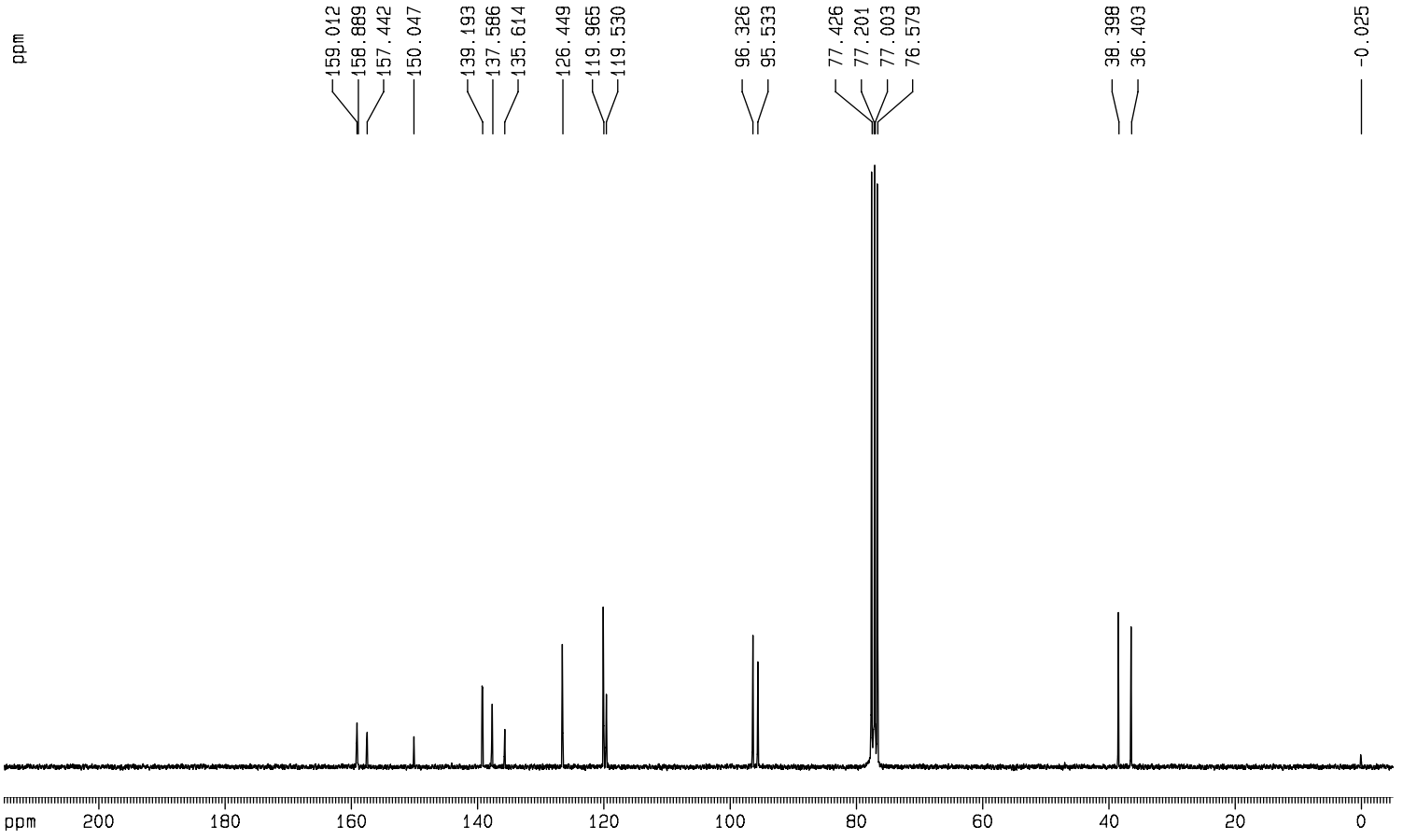





```
Current Data Parameters
NAME                  C-OH
EXPNO                   10
PROCNO                   1

F2 - Acquisition Parameters
Date_              20080921
Time                 15.27
INSTRUM              spect
PROBHD    5 mm DUL 13C-1
PULPROG               zg30
TD                   65536
SOLVENT              CDCl3
NS                      16
DS                       0
SWH               8992.806 Hz
FIDRES            0.137219 Hz
AQ                3.6438515 sec
RG                     362
DW                  55.600 usec
DE                   6.00 usec
TE                  299.5 K
D1              1.00000000 sec
MCREST          0.00000000 sec
MCWRK           0.01500000 sec

======== CHANNEL f1 ========
NUC1                    1H
P1                   7.00 usec
PL1                 -1.00 dB
SFO1           300.1324010 MHz

F2 - Processing parameters
SI                   32768
SF             300.1300065 MHz
WDW                     EM
SSB                      0
LB                    0.30 Hz
GB                       0
PC                    1.00

1D NMR plot parameters
CX                   22.00 cm
CY                   12.50 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
```

^{13}C NMR of **6**






```
Current Data Parameters
NAME                  C-OH
EXPNO                   30
PROCNO                   1

F2 - Acquisition Parameters
Date_              20080921
Time                 21.11
INSTRUM              spect
PROBHD   5 mm DUL 13C-1
PULPROG             zgpg30
TD                   65536
SOLVENT              CDCl3
NS                   10056
DS                       4
SWH              17985.611 Hz
FIDRES           0.274439 Hz
AQ               1.8219508 sec
RG                   812.7
DW                  27.800 usec
DE                   6.00 usec
TE                  301.7 K
D1              2.00000000 sec
d11             0.03000000 sec
DELTA           1.89999998 sec
MCREST          0.00000000 sec
MCWRK           0.01500000 sec

======== CHANNEL f1 ========
NUC1                   13C
P1                   12.50 usec
PL1                   2.00 dB
SFO1            75.4752953 MHz

======== CHANNEL f2 ========
CPDPRG2             waltz16
NUC2                   1H
PCPD2                80.00 usec
PL2                  -1.00 dB
PL12                 20.16 dB
PL13                 16.98 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                   10.00 cm
F1P                215.000 ppm
F1                 16225.57 Hz
F2P                 -5.000 ppm
F2                 -377.34 Hz
PPMCM             10.00000 ppm/cm
HZCM             754.67749 Hz/cm
```