Supporting Information for:

Nucleophilic activity of a linked bis{guanidine} leading to formation of a dicationic C_4N_4-heterocycle

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Contents:

*$[H_2C\{hpp\}_2CH_2][Cl]_2$ (**2a-H_2**)*

$H_2C\{hpp\}_2$ (**1**, 1.00g, 3.44 mmol) was dissolved in CH_2Cl_2 (~ 2 mL) and left at room temperature for 3 days, during which time colourless crystals deposited from solution. Yield 0.77 g, (41 %, calculated for the bis-dichloromethane solvate). Elemental analysis calcd (%) for $C_{16}H_{28}Cl_2N_6 \cdot 2(CH_2Cl_2)$ (*542.08*): C 39.85, H 5.95, N 15.50; found: C 39.73, H 6.18, N 15.41. **2a-H_2**: ^{1}H NMR (500 MHz, D_2O, 298 K): Major Isomer: δ 5.15 and 4.22 (d, $^2J_{HH}$ = 15.6, *exo*cyclic CH_2). Minor Isomer: δ 5.31 and 4.31 (d, $^2J_{HH}$ = 15.8, *exo*cyclic CH_2). The remaining methylene groups of the bicyclic framework overlap in the following regions: δ 3.43 (m, 4H, hpp-CH_2), 3.29 (m, 8H, hpp-CH_2), 3.20 (m, 4H, hpp-CH_2), 1.88 (m, 8H, hpp-CH_2). ^{13}C NMR (125 MHz, D_2O, 298 K): δ 160.8 (CN$_3$), 152.0 (CN$_3$), 74.8 (*exo*cyclic CH$_2$), 68.1 (*exo*cyclic CH$_2$), 47.5 (hpp-CH$_2$), 47.4 (hpp-CH$_2$), 47.2 (hpp-CH$_2$), 21.4 (hpp-CH$_2$), 21.1 (hpp-CH$_2$). **2a-D_2**: [^{2}H]- NMR (76.8 MHz, D_2O, 298 K): 5.05 (br, CD_2), 4.13 (br, CD_2).

*$[H_2C\{hpp\}_2CH_2][BPh_4]_2$ (**2b**)*

A solution of $NaBPh_4$ (0.25 g, 0.73 mmol) in 2 mL H_2O was added *via* syringe to **2a-H_2** (0.20 g, 0.37 mmol) in 1 mL H_2O, causing the immediate precipitation of a white solid. The product was isolated by filtration and purified by slow cooling of a saturated MeCN solution, affording pure **2b** as colourless prisms. Yield 0.25 g, (72 %). Elemental analysis calcd (%) for $C_{64}H_{68}B_2N_6$ (*931.56*): C 81.53, H 7.21, N 8.92; found: C 81.46, H 7.25, N 8.89. ^{1}H NMR (300 MHz, CD_3CN, 298 K): Anion resonances: δ 7.25 (br m, 16H, *o*-C_6H_5), 6.99 (t, $^3J_{HH}$ = 7.40, 16H, *m*-C_6H_5), 6.83 (t, $^3J_{HH}$ = 7.22, 8H, *p*-C_6H_5). Major Isomer: δ 5.06 and 4.15 (d, $^2J_{HH}$ = 15.7, *exo*cyclic CH_2). Minor Isomer: 5.17 and 4.20 (d, $^2J_{HH}$ = 15.9, *exo*cyclic CH_2). The remaining methylene groups of the bicyclic framework overlap in the following regions: δ 3.42 (m, 4H, hpp-CH_2), 3.34 (m, 8H, hpp-CH_2), 3.26 (m, 4H, hpp-CH_2), 1.93 (m, 8H, hpp-CH_2). ^{13}C NMR (75 MHz, CD_3CN, 353 K): δ 164.2 (q, $^1J_{CB}$ = 49.1, *i*-C_6H_5), 161.4 (CN$_3$), 136.1 (C_6H_5), 125.8 (q, J_{CB} = 2.7, C_6H_5), 122.0 (C_6H_5), 75.4 (*exo*cyclic CH$_2$), 68.7 (*exo*cyclic CH$_2$), 48.1 (hpp-CH$_2$), 48.0 (hpp-CH$_2$), 47.9 (hpp-CH$_2$), 47.8 (hpp-CH$_2$), 21.9 (hpp-CH$_2$), 21.6 (hpp-CH$_2$).

General procedure for the preparation of $[H_2C\{hppR\}_2][X]_2$

A sample of **1** was dissolved in neat R–X reagent and stirred for 1-3 days. Addition of Et_2O resulted in formation of a white solid that was separated from excess RX by filtration.

General procedure for the preparation of $\{H_2C\{hppR\}_2][BPh_4]_2$

A solution of two equivalents of $NaBPh_4$ in H_2O was added to an aqueous stirred solution of $[\{H_2C\{hppR\}_2][X]_2$. The product generally precipitated from the reaction mixture and was isolated by filtration. Purification was achieved by crystallization, as detailed below:

*$[H_2C\{hppMe\}_2][I]_2$ (**3**).*

Compound **3a** was purified by crystallization from MeOH at -30 °C. Yield 98 %. Elemental analysis calcd (%) for $C_{17}H_{32}N_6I_2$ (*574.08*): C 35.54, H 5.62, N 14.64; found: C 35.65, H 5.72, N 14.53. ^{1}H NMR (300 MHz, D_2O, 298 K): δ 4.72 (s, 2H, $H_2C\{hppMe\}_2$), 3.38 (m, 8H, hpp-CH_2), 3.20 (m, 8H, hpp-CH_2), 2.91 (s, 6H, hpp*Me*), 1.96 (m, 8H, hpp-CH_2). ^{13}C NMR (75 MHz, D_2O, 298 K): δ 158.2 (CN_3), 66.1 ($H_2C\{hppMe\}_2$), 48.9 (hpp-CH_2), 48.3 (hpp-CH_2), 48.1 (hpp-CH_2), 44.1 (hpp-CH_2), 41.6 (hpp*Me*), 21.4 (hpp-CH_2), 20.6 (hpp-CH_2).

*$[H_2C\{hppCH_2Ph\}_2][BPh_4]_2$ (**4b**).*

Compound **4b** was purified by crystallization from MeOH / acetone at room temperature. Yield 65 %. Elemental analysis calcd (%) for $C_{77}H_{80}B_2N_6$ (*1111.12*): C 83.19, H 7.26, N 7.56; found: C 83.23, H 7.30, N 7.61. ^{1}H NMR (300 MHz, D_6-acetone, 298 K): Anion resonances: δ 7.36 (br m, 16H, *o*-C_6H_5), 6.95 (t, $^3J_{HH}$ = 7.38, 16H, *m*-C_6H_5), 6.81 (t, $^3J_{HH}$ = 7.19, 8H, *p*-C_6H_5). Cation resonances: δ 7.41 (m, 6H, *m*- and *p*-C_6H_5), 7.27 (d, $^3J_{HH}$ = 7.50, 4H, *o*-C_6H_5), 5.01 (s, 2H, $H_2C\{hppCH_2Ph\}_2$), 4.44 (s, 4H, hppCH_2Ph), 3.49 (m, 12H, hpp-CH_2), 3.27 (m, 4H, hpp-CH_2), 2.14 (m, 4H, hpp-CH_2), 1.88 (m, 4H, hpp-CH_2). ^{13}C NMR (75 MHz, D_6-acetone, 273K): δ 164.6 (q, $^1J_{CB}$ = 49.1, *i*-C_6H_5-BPh_4), 158.9 (CN_3), 136.6 (C_6H_5-BPh_4), 135.1 (CH_2Ph), 129.8 (CH_2Ph), 128.8 (CH_2Ph), 127.6 (CH_2Ph), 125.7 (q, J_{CB} = 2.8, C_6H_5-BPh_4), 122.0 (C_6H_5), 66.2 ($H_2C\{hppCH_2Ph\}_2$), 57.0 ($H_2C\{hppCH_2Ph\}_2$), 49.5 (hpp-CH_2), 48.8 (hpp-CH_2), 46.1 (hpp-CH_2), 44.1 (hpp-CH_2), 22.0 (hpp-CH_2), 20.9 (hpp-CH_2).

Reaction of $H_2C\{hpp\}_2$ with Me_2CHI

Excess Me_2CHI was added to an NMR tube containing a solution of $H_2C\{hpp\}_2$ in CD_3CN and the sample was sealed. After 15h the ^1H NMR spectrum showed 100% conversion to a new guanidine containing species, with resonances at δ 6.07 (m, 1H, =C*H*Me), 5.29-5.13 (m, 2H, =C*H*$_2$) and 1.37 (d, *J* = 6.6 Hz, =CH*Me*) corresponding to propene.

^1H NMR (300 MHz, CD_3CN, 298 K): δ 5.19 (s, 2H, *H*$_2$C{hpp}{hppH}), 3.63 (m, 4H, hpp-C*H*$_2$), 3.51 (m, 4H, hpp-C*H*$_2$), 3.41 (m, 8H, hpp-C*H*$_2$), 2.17 (m, 8H, hpp-C*H*$_2$).‡ ^{13}C NMR (75 MHz, CD_3CN, 298 K): δ 152.2 (*C*N_3), 66.6 (H_2*C*{hpp}{hppH}), 48.2 (hpp-*C*H_2), 48.0 (hpp-*C*H_2), 47.7 (hpp-*C*H_2), 41.0 (hpp-*C*H_2), 22.5 (hpp-*C*H_2), 22.2 (hpp-*C*H_2).

(‡ resonance corresponding to the N*H* proton(s) not observed).

Optimized coordinates for 'chair'-conformation (red)

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Optimized coordinates for 'twist-boat'-conformation (blue)

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Optimized coordinates for transition-state (green)

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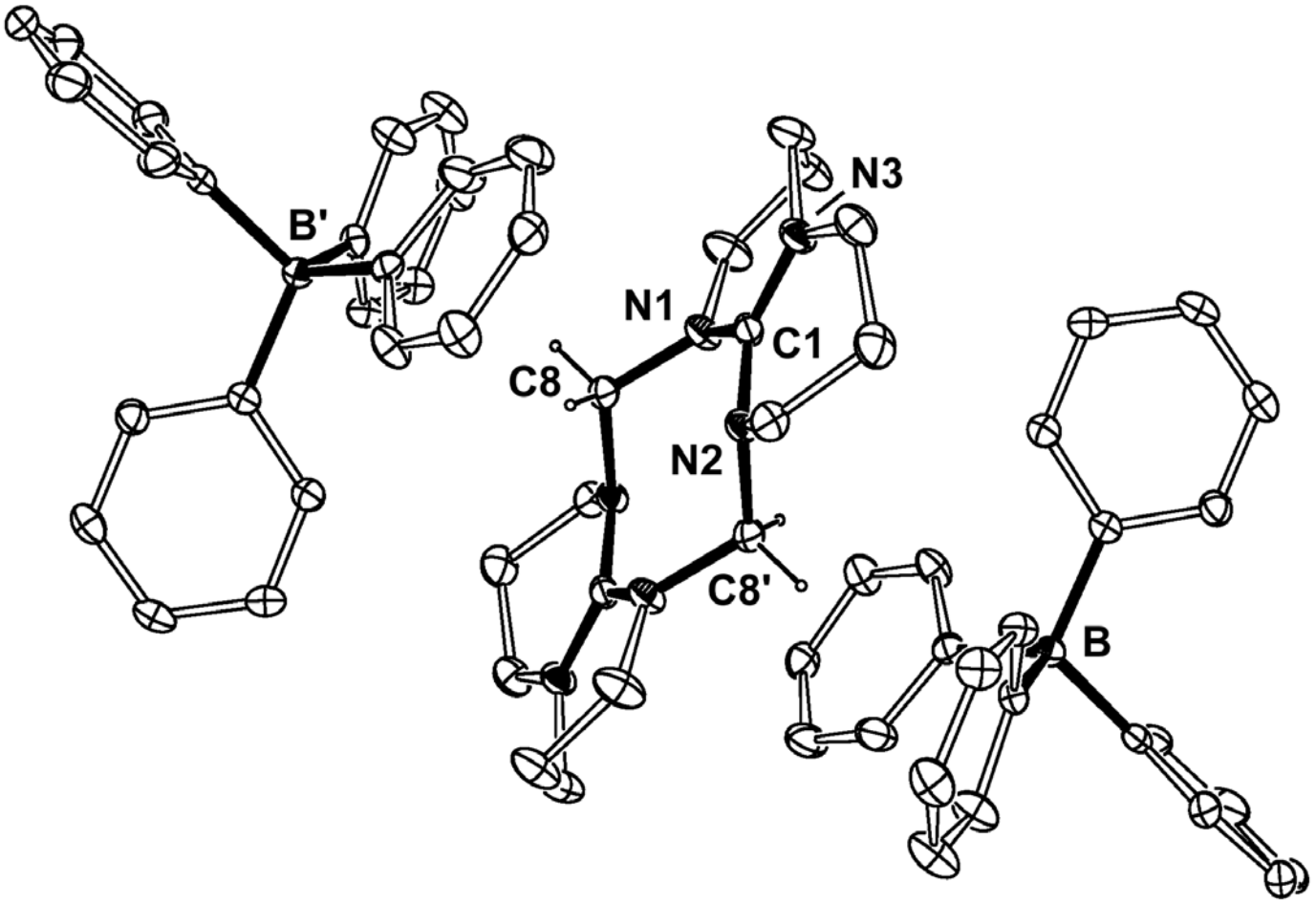
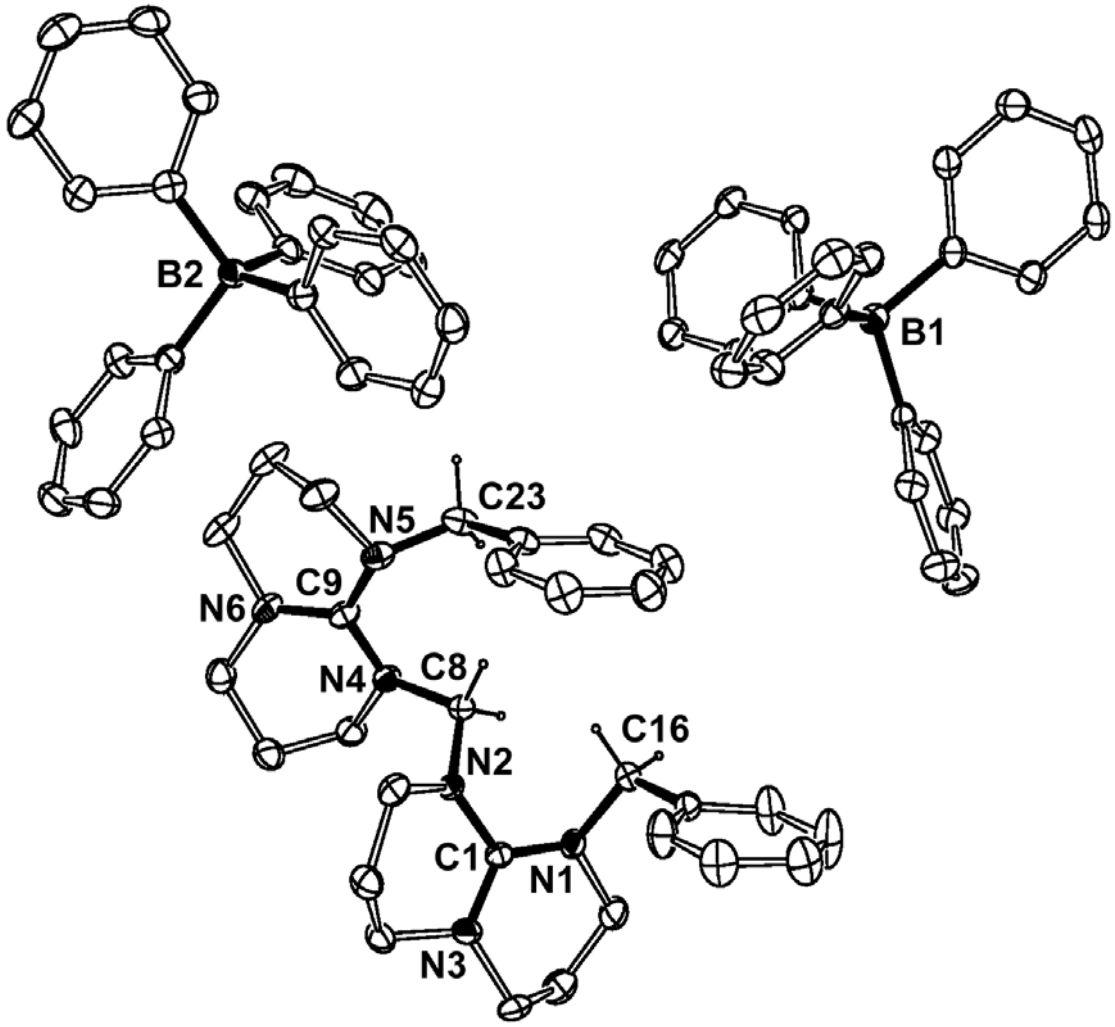
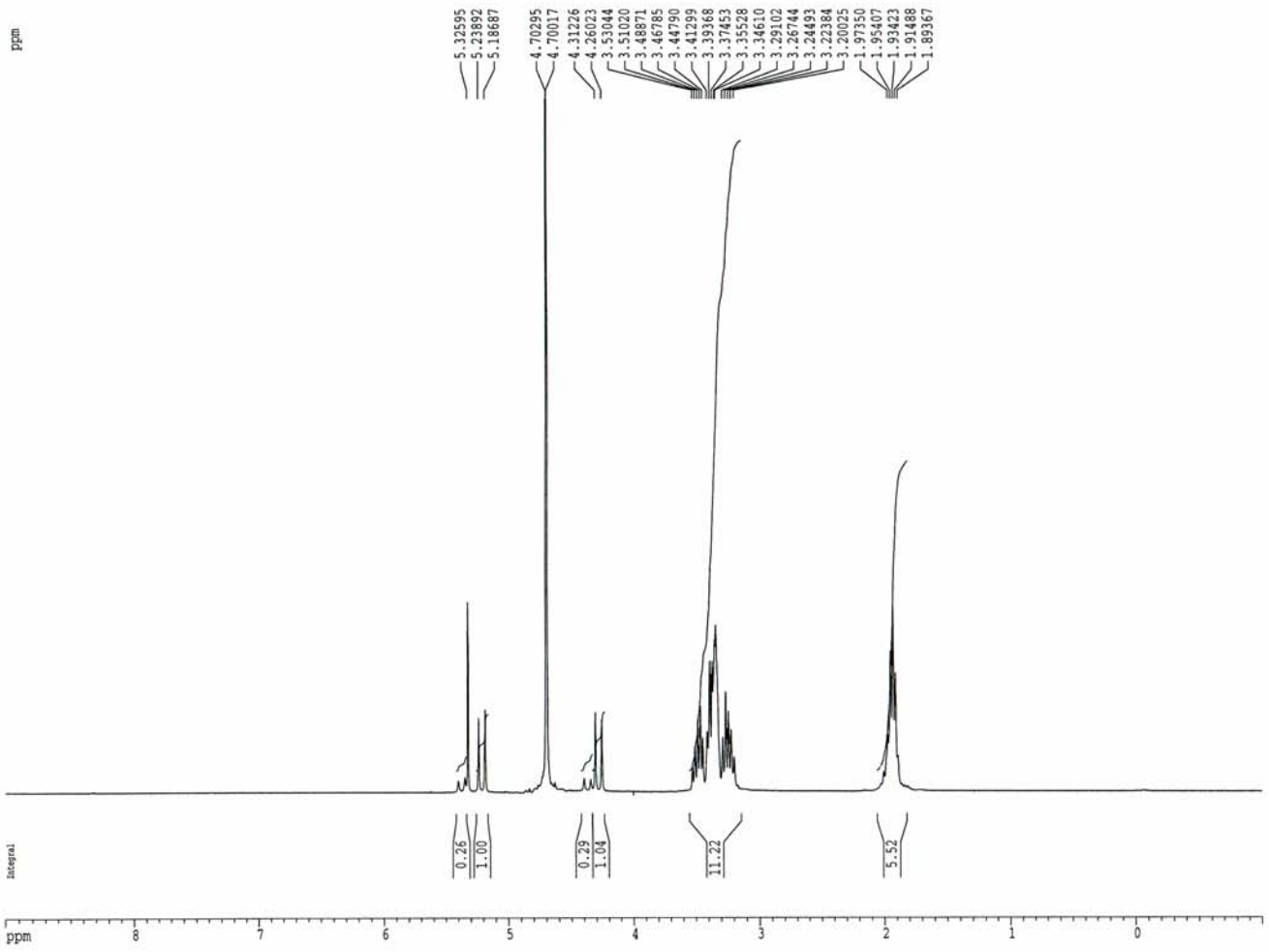
ORTEP for $[H_2C\{hpp\}_2CH_2][BPh_4]_2$ (2b)



ORTEP for $[H_2C\{hppCH_2Ph\}_2][BPh_4]_2$ (4b)



^{1}H NMR [$H_2C\{hpp\}_2CH_2$] [Cl]$_2$ (2a-H_2): D_2O, 298K, 300 MHz

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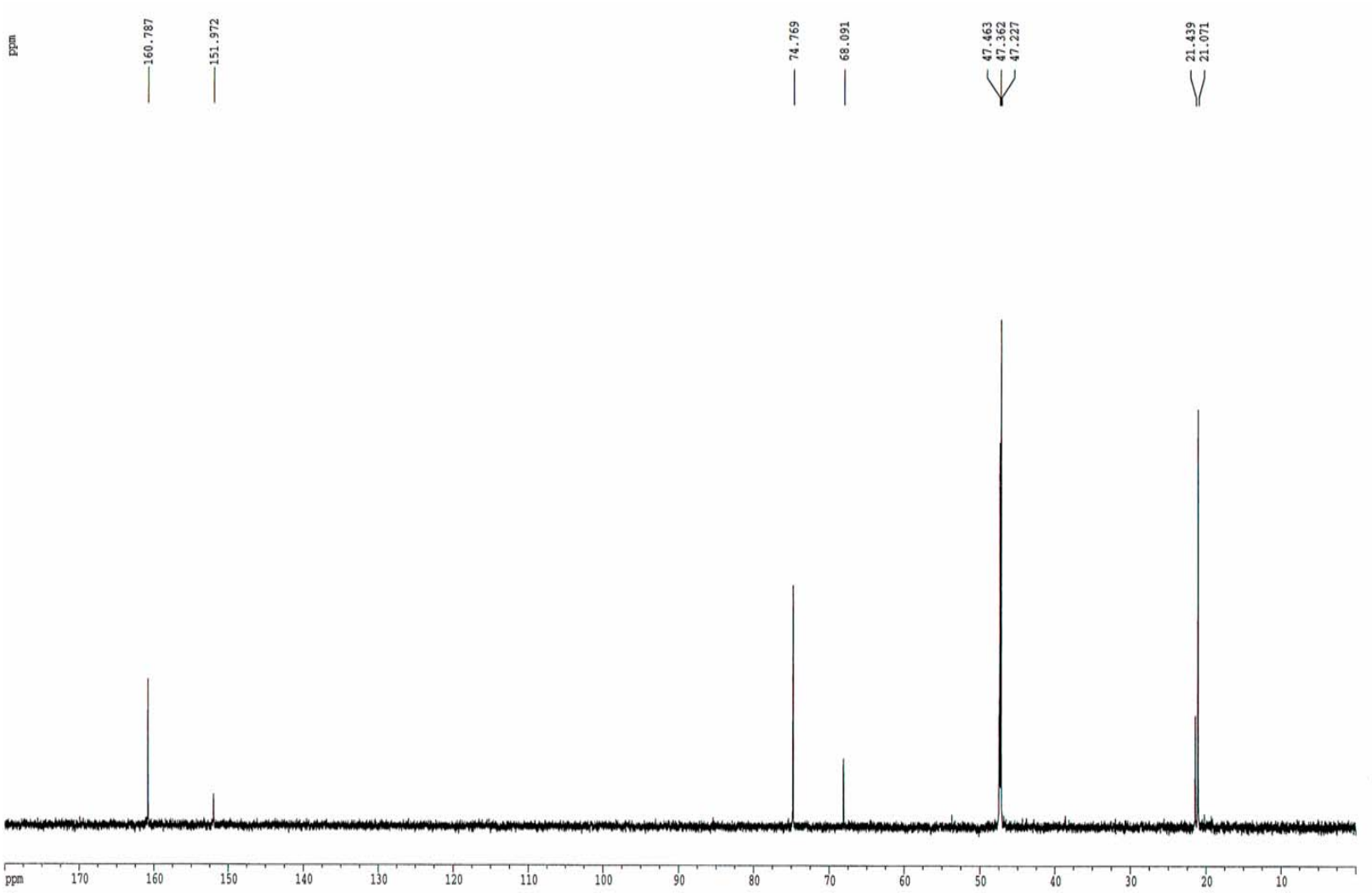

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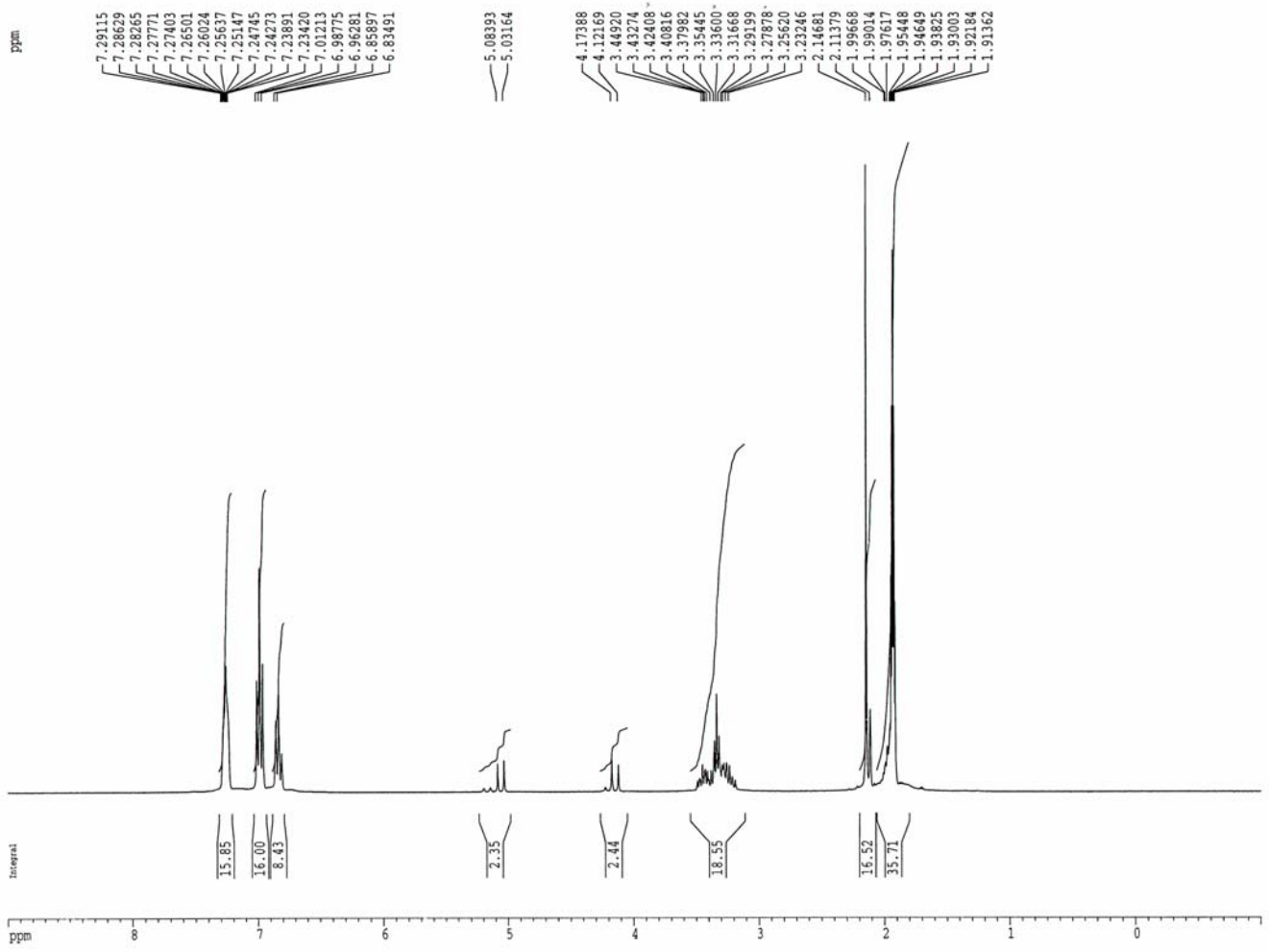


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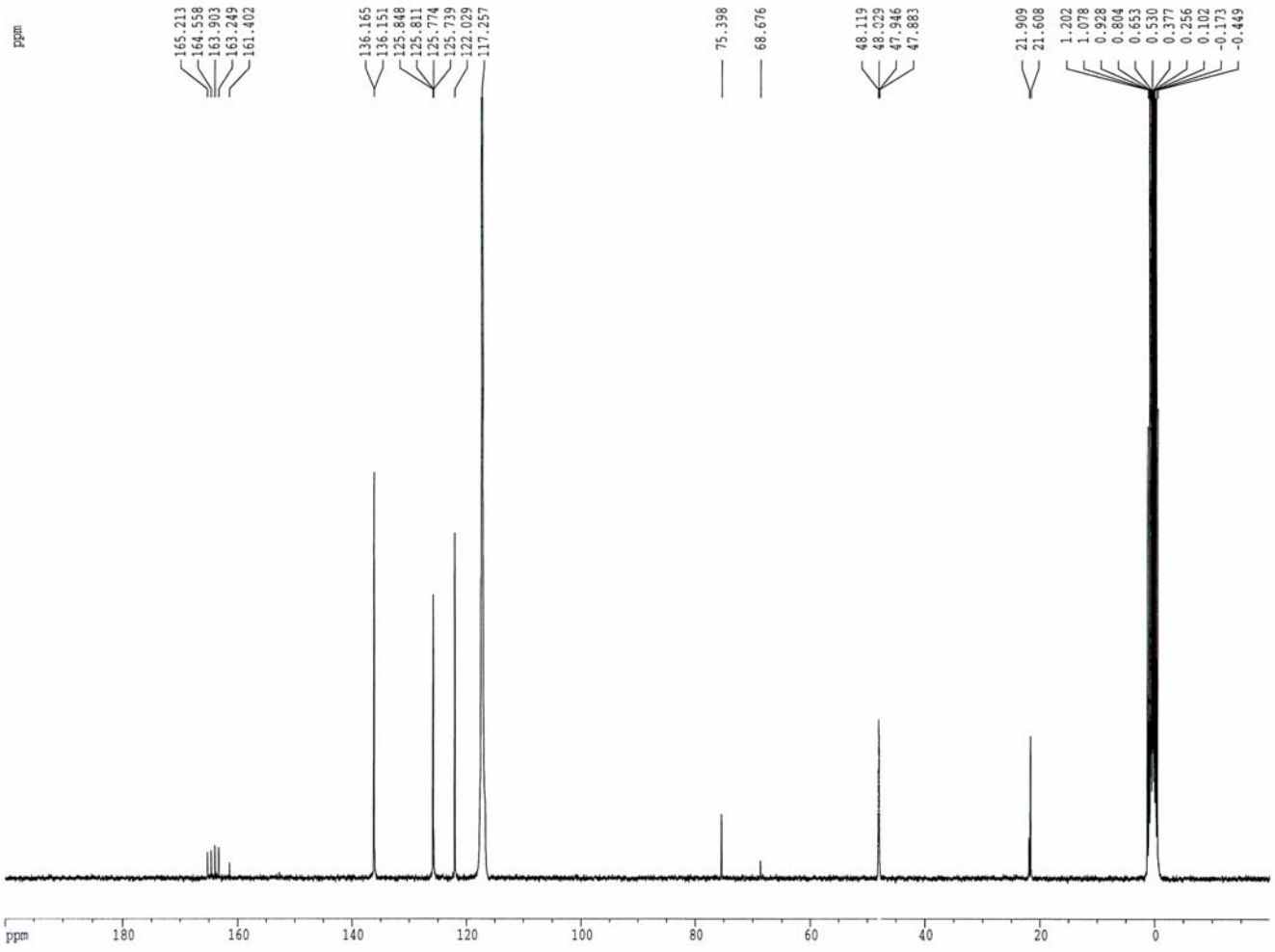
^{13}C NMR $[H_2C\{hpp\}_2CH_2]$ $[BPh_4]_2$ (2b): CD_3CN, 333K, 75 MHz

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PL12                  17.00 dB
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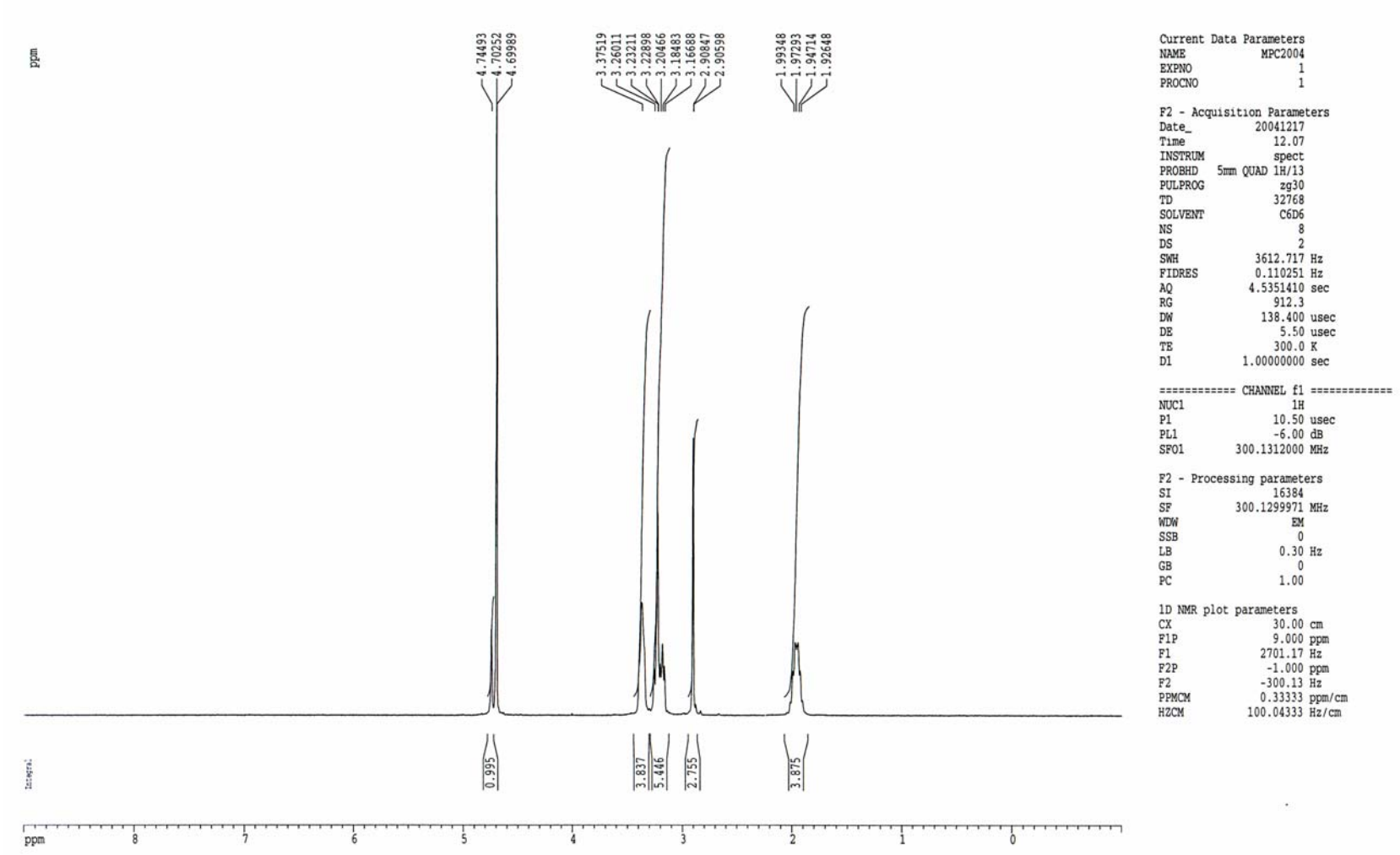
WDW                          EM
SSB                           0
LB                     1.00 Hz
GB                            0
PC                     1.40

1D NMR plot parameters
CX                    30.00 cm
F1P                  200.000 ppm
F1                 15093.55 Hz
F2P                  -20.000 ppm
F2                 -1509.35 Hz
PPMCM                 7.33333 ppm/cm
HZCM               553.42999 Hz/cm
```

^{1}H NMR [$H_2C\{hppMe\}_2$] [I]$_2$ (3): D_2O, 298K, 300 MHz




```
Current Data Parameters
NAME              MPC2004
EXPNO                   1
PROCNO                  1

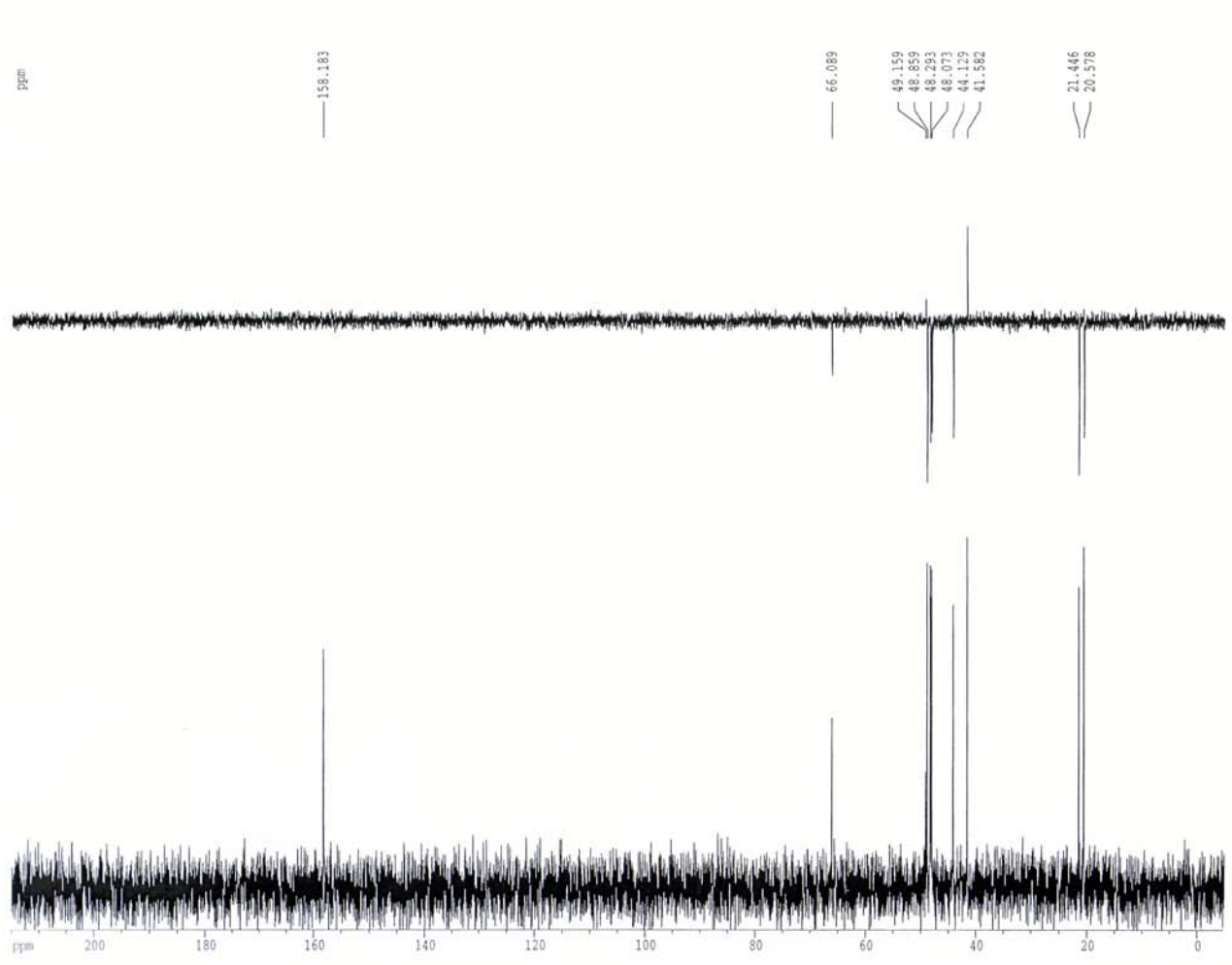
F2 - Acquisition Parameters
Date_            20041217
Time                12.07
INSTRUM             spect
PROBHD   5mm QUAD 1H/13
PULPROG             zg30
TD                 32768
SOLVENT             C6D6
NS                     8
DS                     2
SWH             3612.717 Hz
FIDRES             0.110251 Hz
AQ              4.5351410 sec
RG                 912.3
DW                 138.400 usec
DE                   5.50 usec
TE                 300.0 K
D1              1.00000000 sec

============ CHANNEL f1 ============
NUC1                   1H
P1                  10.50 usec
PL1                 -6.00 dB
SFO1          300.1312000 MHz

F2 - Processing parameters
SI                  16384
SF            300.1299971 MHz
WDW                    EM
SSB                     0
LB                   0.30 Hz
GB                      0
PC                   1.00

1D NMR plot parameters
CX                  30.00 cm
F1P                 9.000 ppm
F1                2701.17 Hz
F2P                -1.000 ppm
F2                -300.13 Hz
PPMCM             0.33333 ppm/cm
HZCM            100.04333 Hz/cm
```

^{13}C NMR [H_2C{hppMe}$_2$] [I]$_2$ (3): D_2O, 298K, 75 MHz (+ DEPT 135)




```
Current Data Parameters
NAME                  Martyn
EXPNO                     11
PROCNO                     1

F2 - Acquisition Parameters
Date_               20041217
Time                   17.06
INSTRUM                spect
PROBHD                 dual

PULPROG              zgpg30
TD                    65536
SOLVENT                D2O
NS                      256
DS                        4
SWH                18832.393 Hz
FIDRES             0.287360 Hz
AQ                 1.7400308 sec
RG                     8192
DW                   26.550 usec
DE                    6.00 usec
TE                   300.0 K
D1                2.00000000 sec
D11               0.03000000 sec
D12               0.00002000 sec

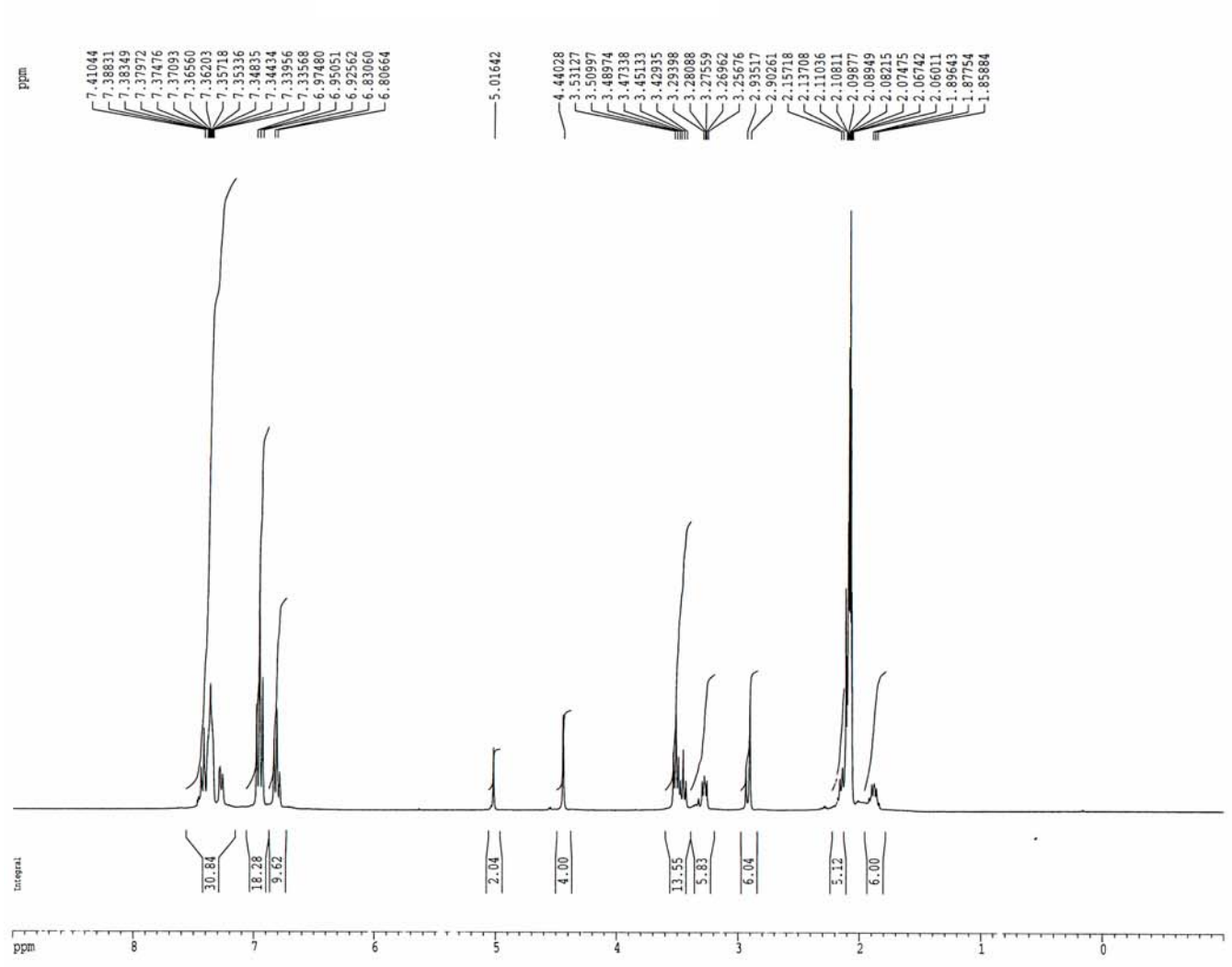
============ CHANNEL f1 =============
NUC1                    13C
P1                     5.50 usec
PL1                   -3.00 dB
SFO1              75.4760200 MHz

============ CHANNEL f2 =============
CPDPRG2              waltz16
NUC2                    1H
PCPD2                100.00 usec
PL2                   -6.00 dB
PL12                  16.00 dB
PL13                  18.00 dB
SFO2             300.1312005 MHz

F2 - Processing parameters
SI                      32768
SF                75.4677190 MHz
WDW                        EM
SSB                         0
LB                     1.00 Hz
GB                          0
PC                     1.40

1D NMR plot parameters
CX                    30.00 cm
F1P                  215.000 ppm
F1                 16225.56 Hz
F2P                   -5.000 ppm
F2                  -377.34 Hz
PPMCM                7.33333 ppm/cm
HZCM               553.42993 Hz/cm
```

^{1}H NMR [$H_2C\{hppCH_2Ph\}_2$] [BPh_4]$_2$ (4b): D_6-acetone, 298K, 300 MHz




```
Current Data Parameters
NAME                  MPC523e
EXPNO                       1
PROCNO                      1

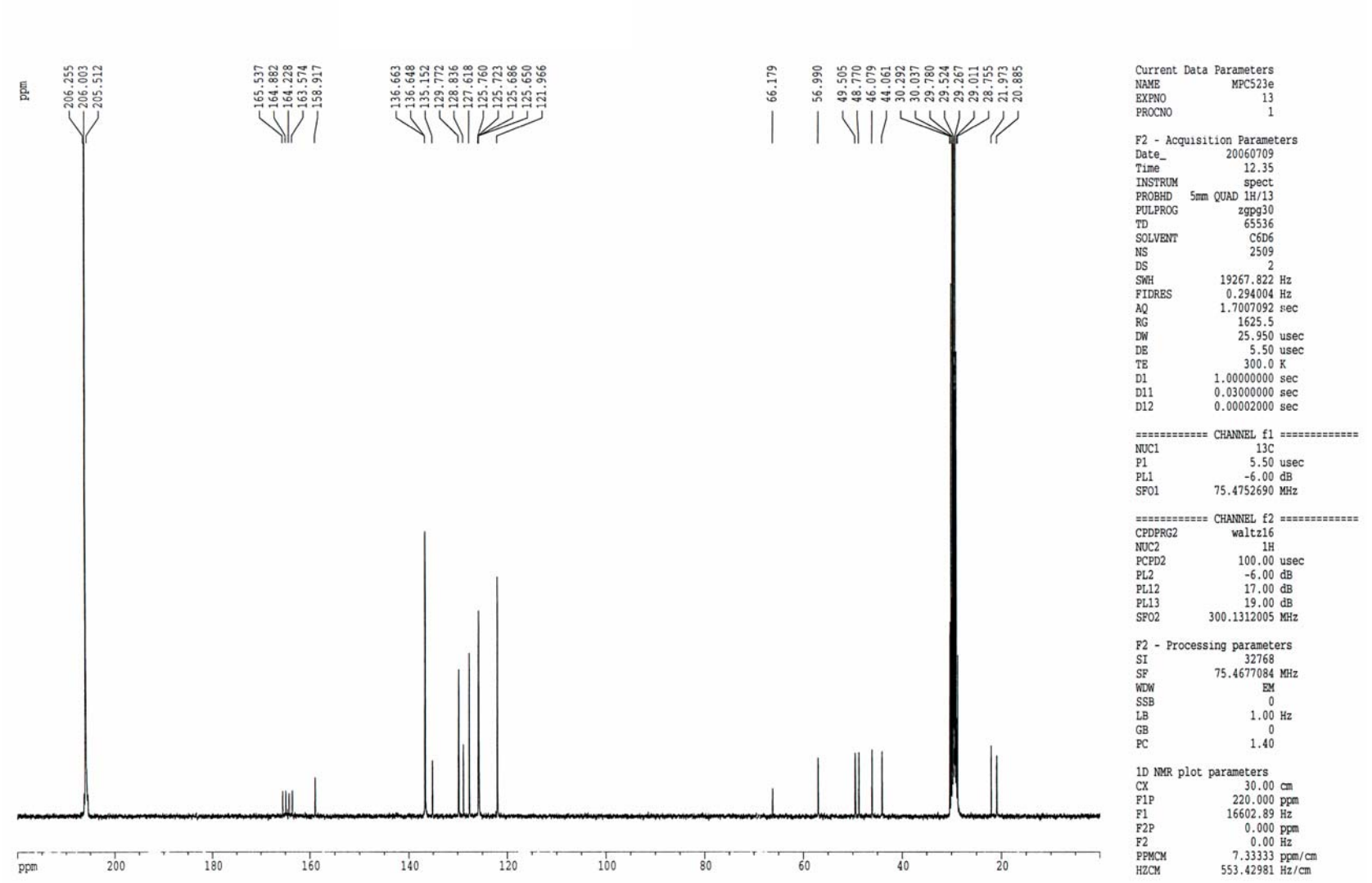
F2 - Acquisition Parameters
Date_                20060709
Time                    12.29
INSTRUM                 spect
PROBHD   5mm QUAD 1H/13
PULPROG                 zg30
TD                     32768
SOLVENT                 C6D6
NS                         8
DS                         2
SWH                 3612.717 Hz
FIDRES              0.110251 Hz
AQ                4.5351410 sec
RG                     143.7
DW                   138.400 usec
DE                     5.50 usec
TE                     300.0 K
D1                1.00000000 sec

============ CHANNEL f1 =============
NUC1                       1H
P1                      10.50 usec
PL1                     -6.00 dB
SFO1               300.1312000 MHz

F2 - Processing parameters
SI                       16384
SF                 300.1299966 MHz
WDW                         EM
SSB                          0
LB                        0.30 Hz
GB                           0
PC                        1.00

1D NMR plot parameters
CX                       30.00 cm
F1P                       9.000 ppm
F1                     2701.17 Hz
F2P                      -1.000 ppm
F2                     -300.13 Hz
PPMCM                 0.33333 ppm/cm
HZCM                100.04333 Hz/cm
```

^{13}C NMR [$H_2C\{hppCH_2Ph\}_2$] [BPh_4]$_2$ (4b): D_6-acetone, 298K, 75 MHz




```
Current Data Parameters
NAME                  MPC523e
EXPNO                      13
PROCNO                      1

F2 - Acquisition Parameters
Date_               20060709
Time                   12.35
INSTRUM                spect
PROBHD   5mm QUAD 1H/13
PULPROG                zgpg30
TD                      65536
SOLVENT                  C6D6
NS                       2509
DS                          2
SWH                19267.822 Hz
FIDRES              0.294004 Hz
AQ                  1.7007092 sec
RG                     1625.5
DW                    25.950 usec
DE                     5.50 usec
TE                     300.0 K
D1                  1.00000000 sec
D11                 0.03000000 sec
D12                 0.00002000 sec

============ CHANNEL f1 =============
NUC1                      13C
P1                     5.50 usec
PL1                   -6.00 dB
SFO1               75.4752690 MHz

============ CHANNEL f2 =============
CPDPRG2                waltz16
NUC2                       1H
PCPD2                 100.00 usec
PL2                   -6.00 dB
PL12                  17.00 dB
PL13                  19.00 dB
SFO2              300.1312005 MHz

F2 - Processing parameters
SI                       32768
SF                 75.4677084 MHz
WDW                         EM
SSB                          0
LB                        1.00 Hz
GB                           0
PC                        1.40

1D NMR plot parameters
CX                       30.00 cm
F1P                     220.000 ppm
F1                    16602.89 Hz
F2P                       0.000 ppm
F2                        0.00 Hz
PPMCM                  7.33333 ppm/cm
HZCM                 553.42981 Hz/cm
```