Electronic Supplementary Information

Highly Efficient Construction of Large Molecular Cavity Using 1,3-Alternate Tetraoxacalix[2]arene[2]triazine as a Platform

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1. General information

^{1}H and ^{13}C NMR spectra were recorded on a Bruker Avance 300 and 600 spectrometers. 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 were performed at the Analytical Laboratory of the Institute. All chemicals were dried or purified according to standard procedures prior to use.

2. General Procedure for the Synthesis of Large Cavity bis-Tetraoxacalix[2]arene[2]triazines 3, 4, 5, 6 and 7. Both solutions of bis-nucleophilic reagent **2a**, **2b**, **2c**, **2d** or **2e** (1.0 mmol) in dry THF (200 ml) and dichlorinated tetraoxacalix[2]arene[2]triazine **1** (1.0 mmol) in dry THF (200 ml) were added dropwise at the same rate to a refluxing suspension of K_2CO_3 (2.76 g, 20.0 mmol) in dry THF (480 ml). After addition of the reagents, which took about 10 h, the resulting mixture was refluxed for another 14 h. Filtration removed the solids, and the filtrate was concentrated under vacuum. The residue was then chromatographed on a silica gel column (200-300 mesh) with a mixture of dichloromethane and methanol (200:1 – 100:1) as the mobile phase to give pure product **3, 4, 5, 6** and **7** as white solid.

3: mp >300 °C; IR (KBr) ν 3281, 1727, 1585 cm^{-1}; ^1H NMR (300 MHz, $CDCl_3$) δ 9.42-9.45 (4H, m, NH), 7.48-7.33 (30H, m, CH), 7.12 (2H, t, *J* = 7.6 Hz, CH), 7.04 (2H, s, CH), 6.91 (4H, d, *J* = 7.2 Hz, CH), 6.33-6.32 (2H, m, CH), 5.46-5.24 (12H, m, CH_2), 4.40-4.34 (4H, m, CH_2); ^{13}C NMR (75 MHz, $CDCl_3$) δ 172.2, 171.1, 169.3, 164.4, 164.3, 151.8, 151.2, 140.1, 135.4, 132.5, 132.2, 128.7, 128.6, 128.5, 128.4, 128.2, 127.9, 125.1, 121.1, 120.1, 120.0, 67.3, 67.2, 43.2; MS (MALDI-TOF) *m*/*z* 1549.1 ($M+H^+$), 1571.0 ($M+Na^+$), 1587.0 ($M+K^+$). Anal. Calcd for $C_{84}H_{60}N_{16}O_{16}$: C, 65.11; H, 3.90; N, 14.46. Found: C, 64.83; H, 3.90; N, 14.19.

4: mp >300 °C; IR (KBr) ν 1729, 1571 cm^{-1}; ^1H NMR (300 MHz, $CDCl_3$) δ 7.89 (4H, d, *J* = 2.2 Hz, CH), 7.31-7.45 (38H, m, CH), 7.19 (2H, t, *J* = 2.2 Hz, CH), 6.79 (4H, t, *J* = 2.1 Hz, CH), 5.42 (4H, s, CH_2), 5.20 (8H, s, CH_2); ^{13}C NMR (75 MHz, $CDCl_3$) δ 174.4, 173.2, 164.2, 163.7, 151.3, 151.2, 135.5, 135.1, 133.6, 133.3, 128.7, 128.6, 128.5, 128.3, 122.1, 120.6, 120.5, 120.0; MS (MALDI-TOF) *m*/*z* 1766.3 ($M+H^+$), 1788.3 ($M+Na^+$), 1804.3 ($M+K^+$). Anal. Calcd for $C_{96}H_{60}N_{12}O_{24}$: C, 65.31; H, 3.43; N, 9.52. Found: C, 65.51; H, 3.58; N, 9.46.

5: mp >300 °C; IR (KBr) ν 1726, 1590 cm^{-1}; ^1H NMR (300 MHz, $CDCl_3$) δ 7.48-7.47 (4H, m, CH), 7.38-7.30 (24H, m, CH), 6.53 (4H, s, br, CH), 5.26 (8H, s, CH_2),

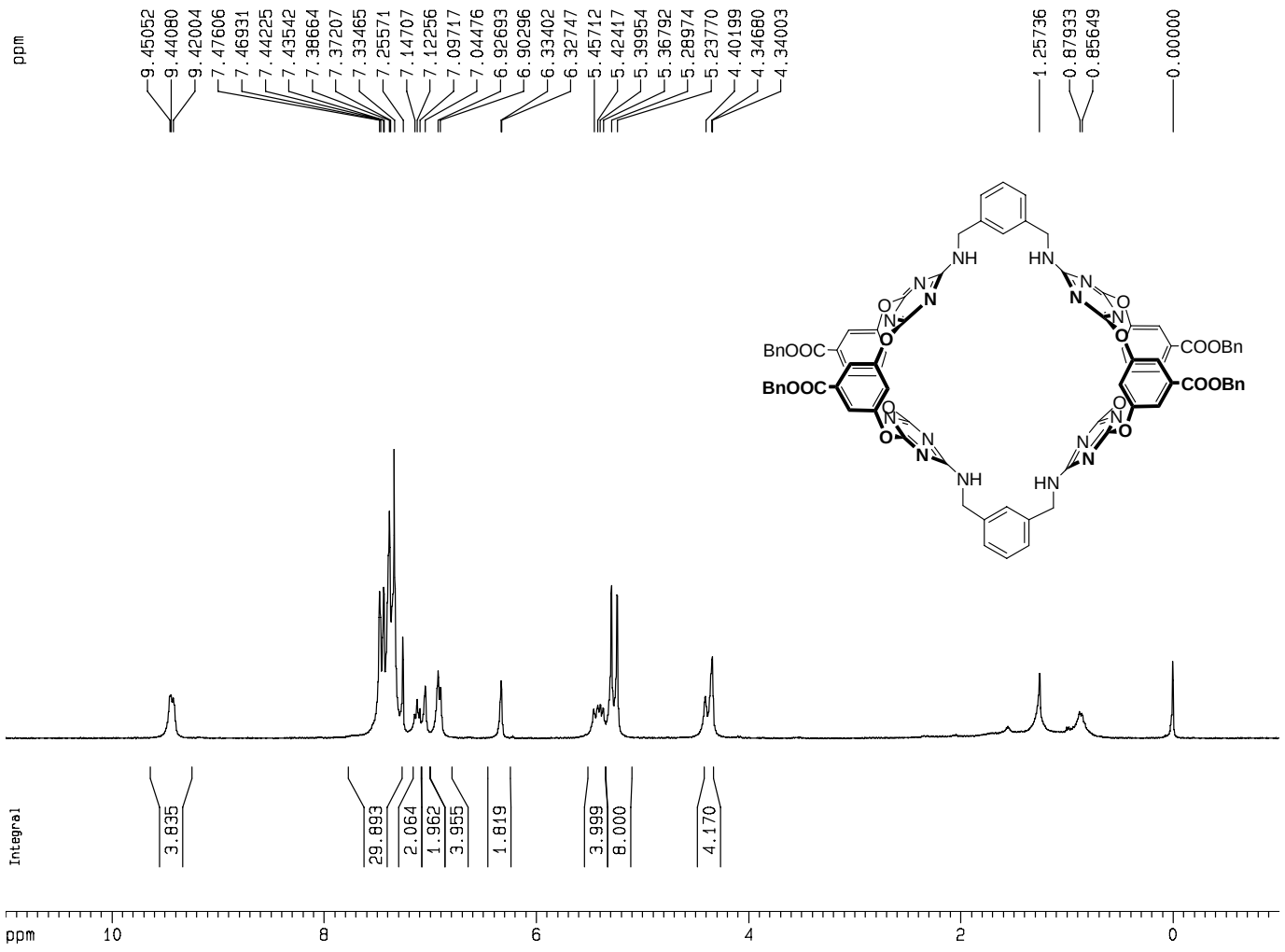
5.05-5.01 (4H, d, J = 12.3 Hz, CH_2), 3.40 (12H, s, CH_3), 3.07-3.03 (4H, d, J = 12.4 Hz, CH_2); ^{13}C NMR (75 MHz, $CDCl_3$) δ 171.7, 171.0, 168.8, 164.4, 151.9, 151.6, 135.7, 132.1, 128.7, 128.4, 128.2, 121.2, 120.1, 119.6, 67.0, 46.6, 35.2; MS (MALDI-TOF) *m*/*z* 1453.8 ($M+H^+$), 1475.7 ($M+Na^+$), 1491.7 ($M+K^+$). Anal. Calcd for $C_{76}H_{60}N_{16}O_{16}$: C, 62.81; H, 4.16; N, 15.42. Found: C, 62.86; H, 4.24; N, 15.44.

6: mp 293-294 °C; $[\alpha]_D^{25}$ -12° (*c* 1.0, $CHCl_3$); IR (KBr) ν 3274, 1727, 1586 cm^{-1}; ^{1}H NMR (300 MHz, $CDCl_3$) δ 7.58-7.57 (4H, m, CH), 7.40-7.30 (30H, m, CH), 7.19 (18H, s, CH), 6.83-6.81 (4H, m, NH), 5.49-5.48 (4H, m, CH), 5.26-5.25 (8H, m, CH_2); ^{13}C NMR (75 MHz, $CDCl_3$) δ 172.3, 171.9, 168.7, 164.5, 152.9, 151.6, 137.8, 135.5, 132.4, 128.7, 128.6, 128.3, 128.2, 127.5, 120.7, 120.5, 119.7, 67.2, 62.1; MS (MALDI-TOF) *m*/*z* 1701.7 ($M+H^+$), 1723.7 ($M+Na^+$), 1739.6 ($M+K^+$). Anal. Calcd for $C_{96}H_{68}N_{16}O_{16}$: C, 67.76; H, 4.03; N, 13.17. Found: C, 67.79; H, 4.26; N, 13.04.

7: mp 295-296 °C; $[\alpha]_D^{25}$ 98.6° (*c* 1.0, $CHCl_3$); IR (KBr) ν 3265, 1727, 1590 cm^{-1}; ^{1}H NMR (300 MHz, $CDCl_3$) δ 7.59-7.58 (4H, m, CH), 7.43-7.42 (4H, m, CH), 7.37-7.31 (20H, m, CH), 6.78 (4H, t, J = 2.2 Hz, CH), 6.75 (4H, s, br, NH), 5.32-5.23 (8H, m, CH_2), 3.97 (4H, s, br, CH), 2.23-2.22 (4H, m, CH_2), 1.83 (4H, s, br, CH_2), 1.42-1.40 (8H, m, CH_2); ^{13}C NMR (75 MHz, d_6-DMSO) δ 171.0, 170.5, 168.3, 163.8, 163.7, 152.1, 151.2, 135.6, 135.5, 131.5, 131.3, 128.3, 128.1, 128.0, 120.2, 119.1, 118.0, 66.5, 54.1, 31.9, 24.2; MS (MALDI-TOF) *m*/*z* 1505.1 ($M+H^+$), 1527.0 ($M+Na^+$), 1543.0 ($M+K^+$). Anal. Calcd for $C_{80}H_{64}N_{16}O_{16}$: C, 63.82; H, 4.28; N, 14.89. Found: C, 63.71; H, 4.47; N, 14.56.

4. ^{1}H NMR and ^{13}C NMR Spectra of Compounds

3-^{1}H NMR

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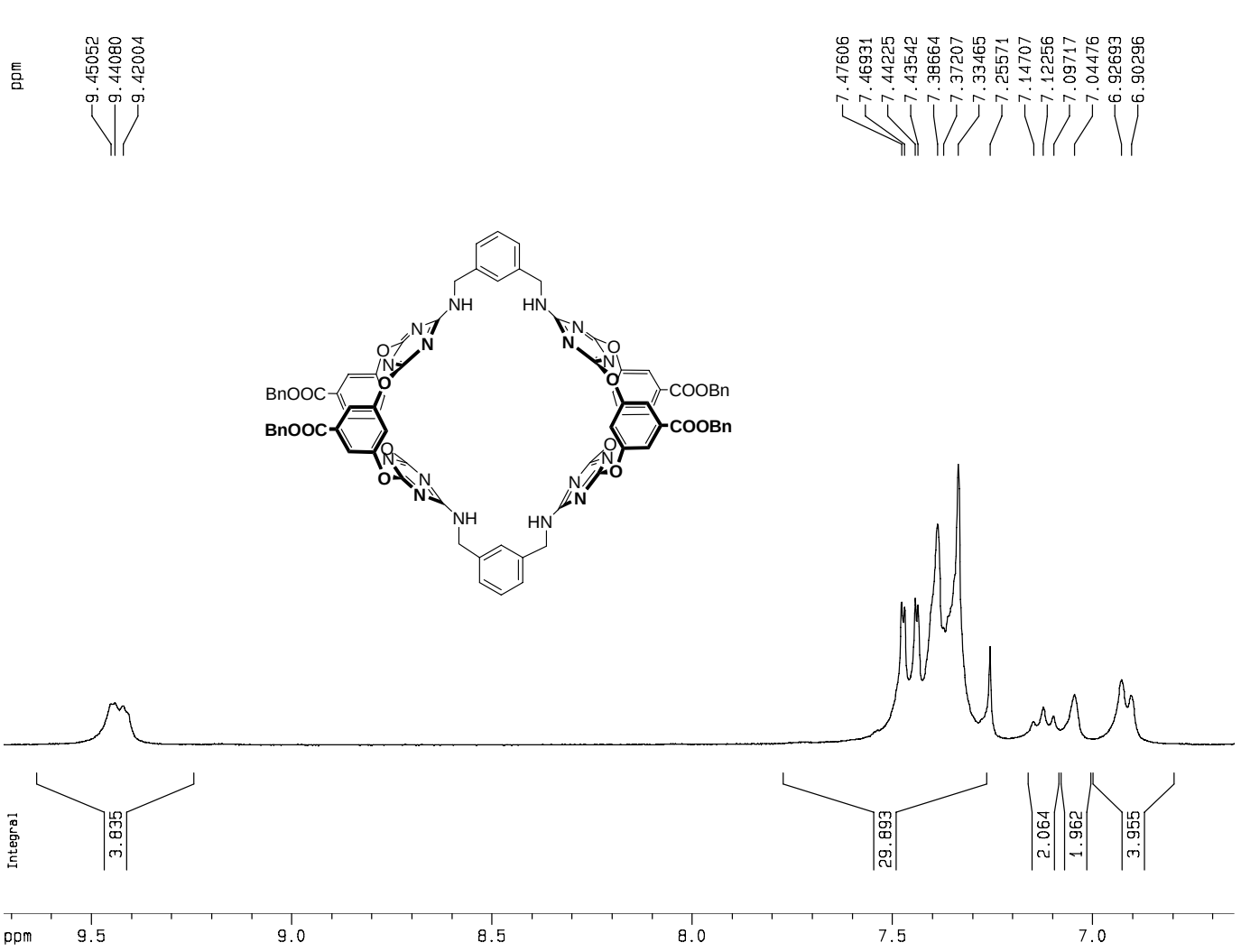
3-^{1}H NMR (expanded)

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PL1              -1.00 dB
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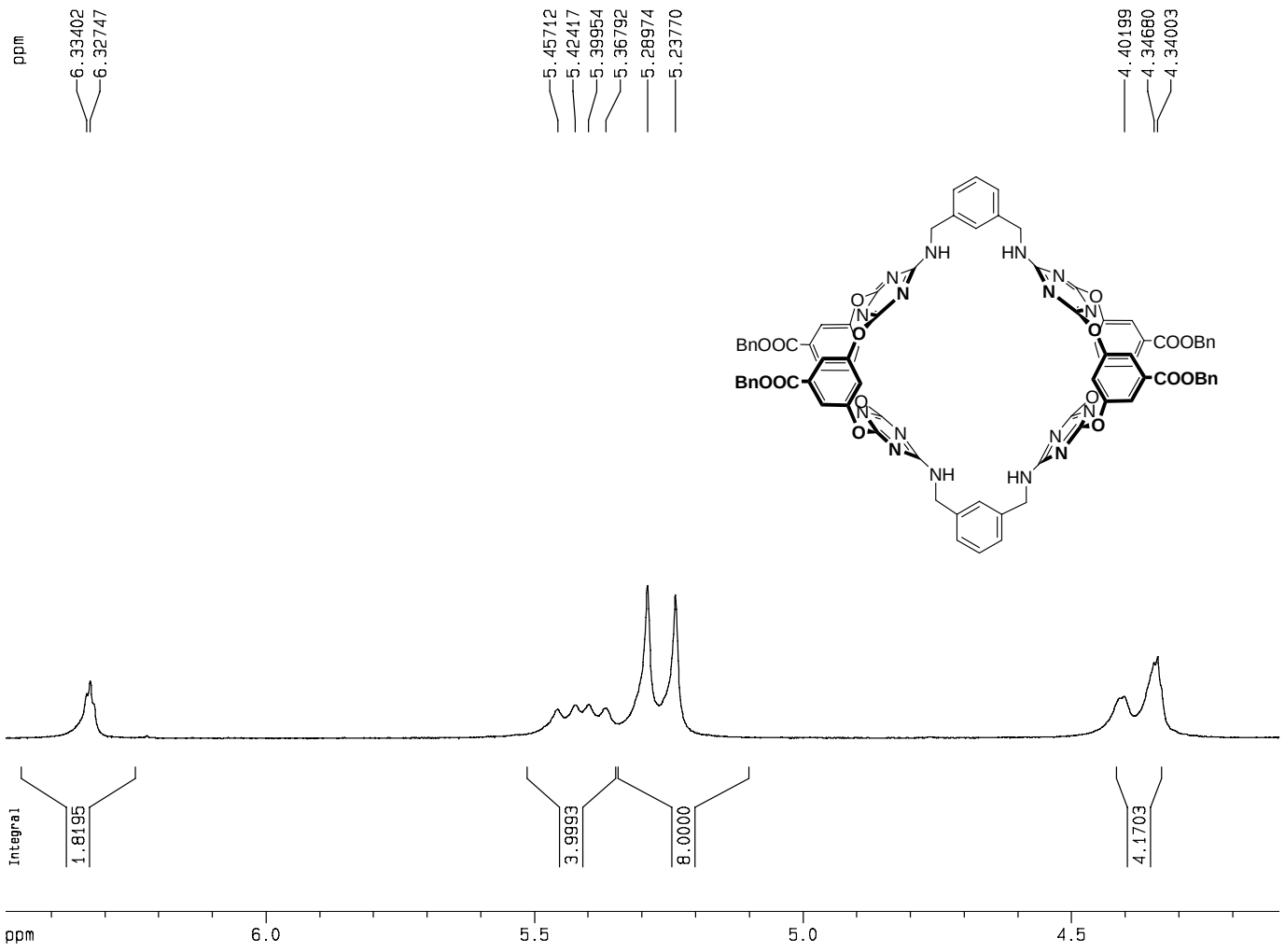
3-^{1}H NMR (expanded)

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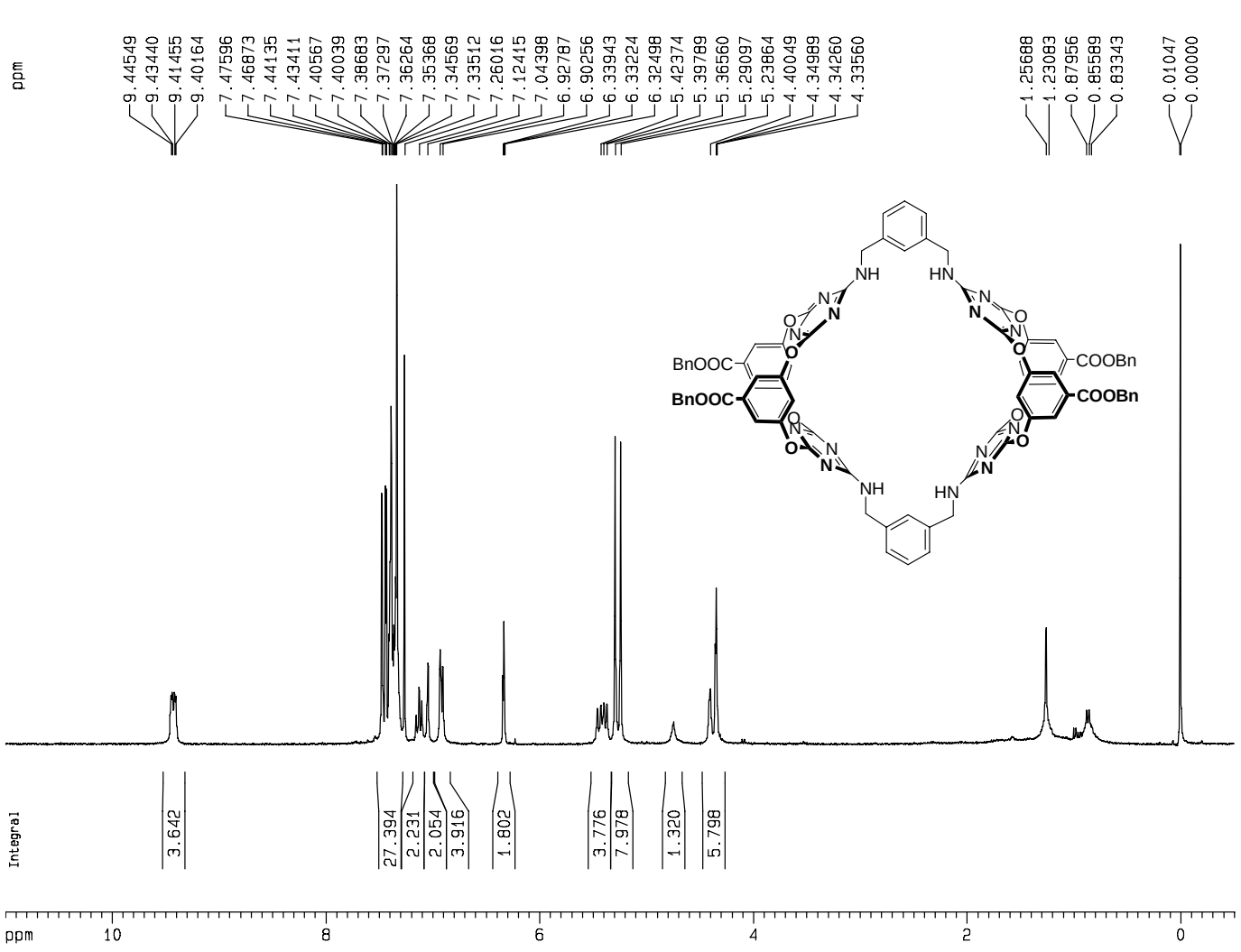


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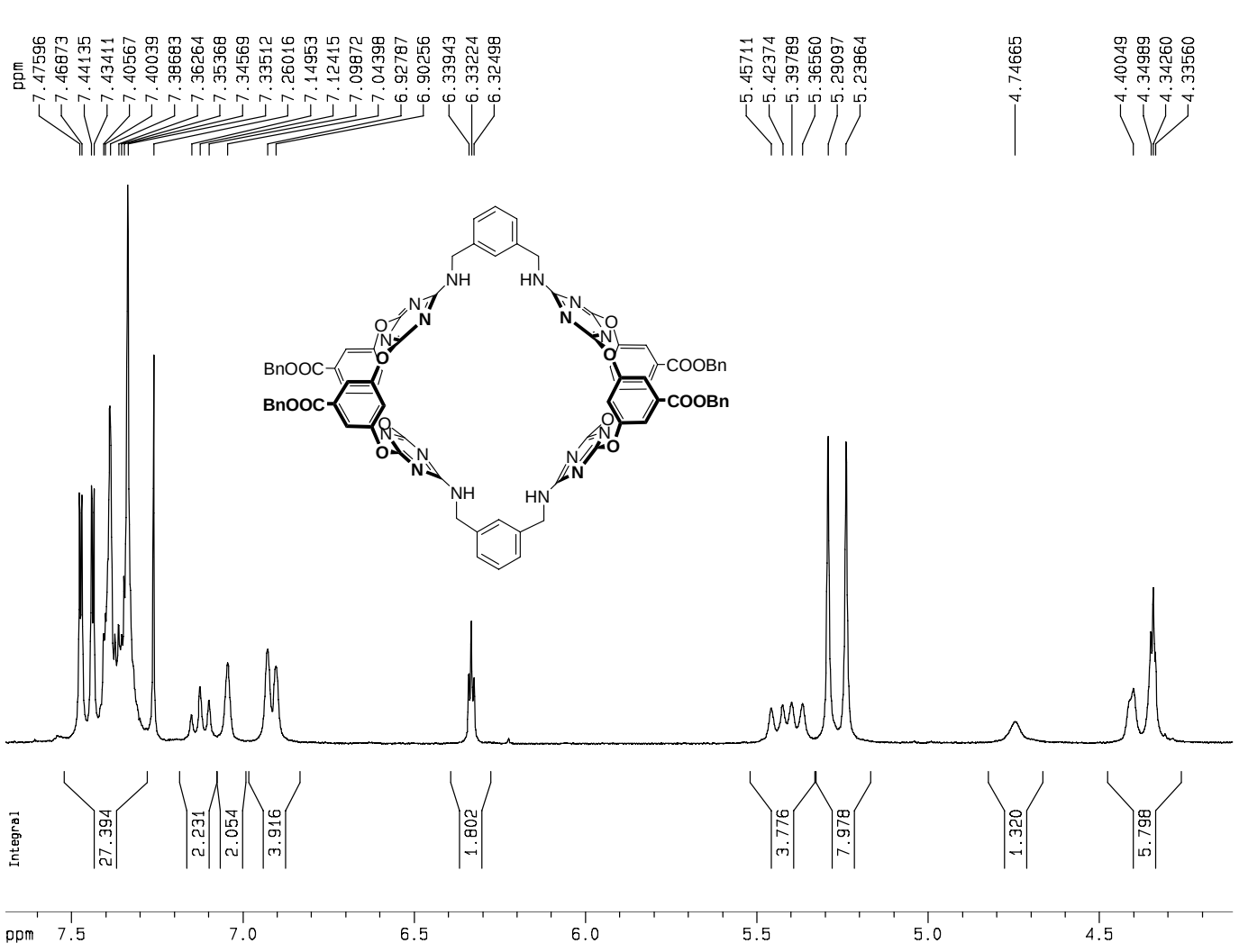
3-^{1}H NMR-D_2O (expanded)

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PROCNO                 1

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FIDRES          0.094190 Hz
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RG                   362
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======== CHANNEL f1 ========
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PL1               -1.00 dB
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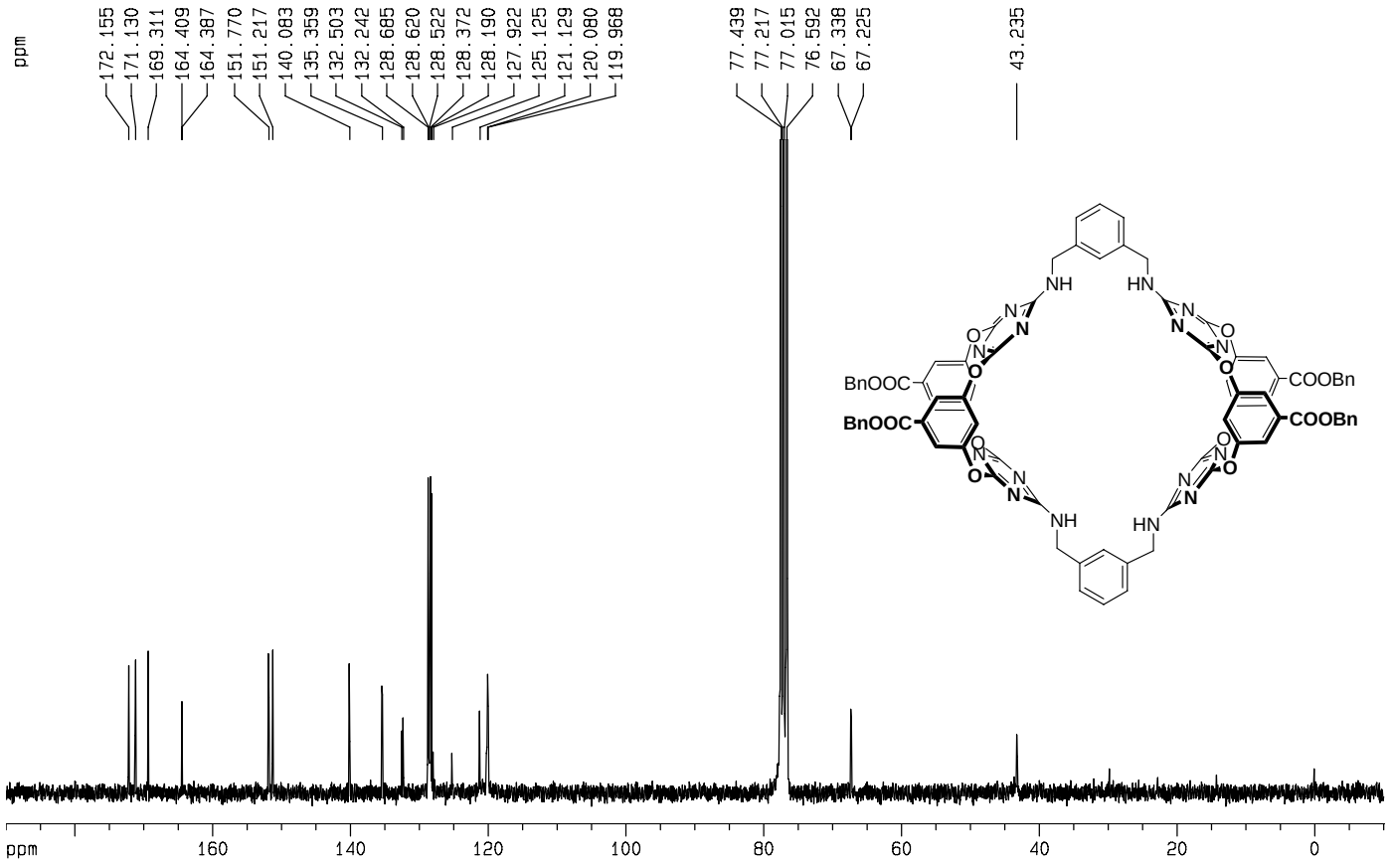
3-^{13}C NMR

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======== CHANNEL f2 ========
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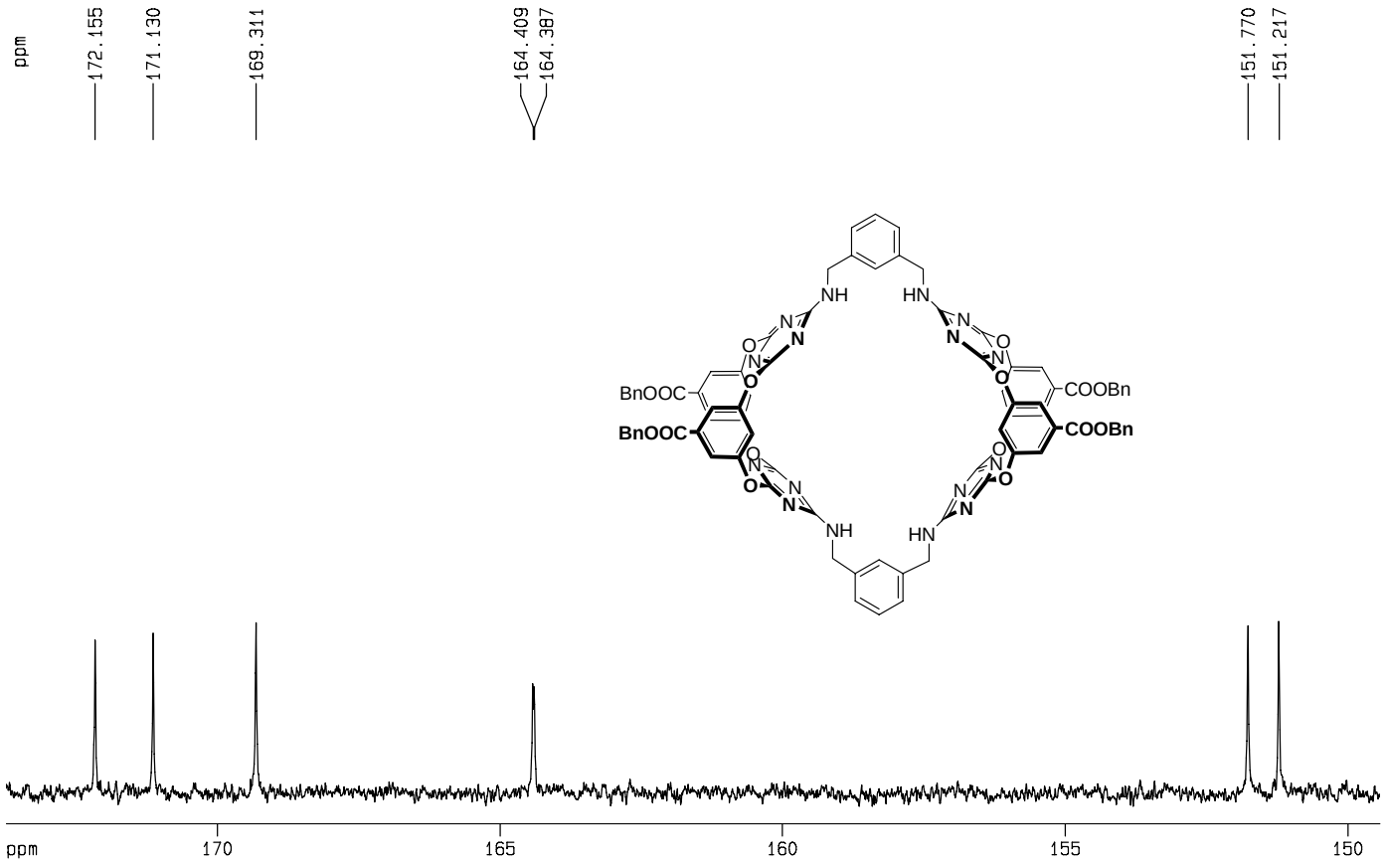
3-^{13}C NMR (expanded)

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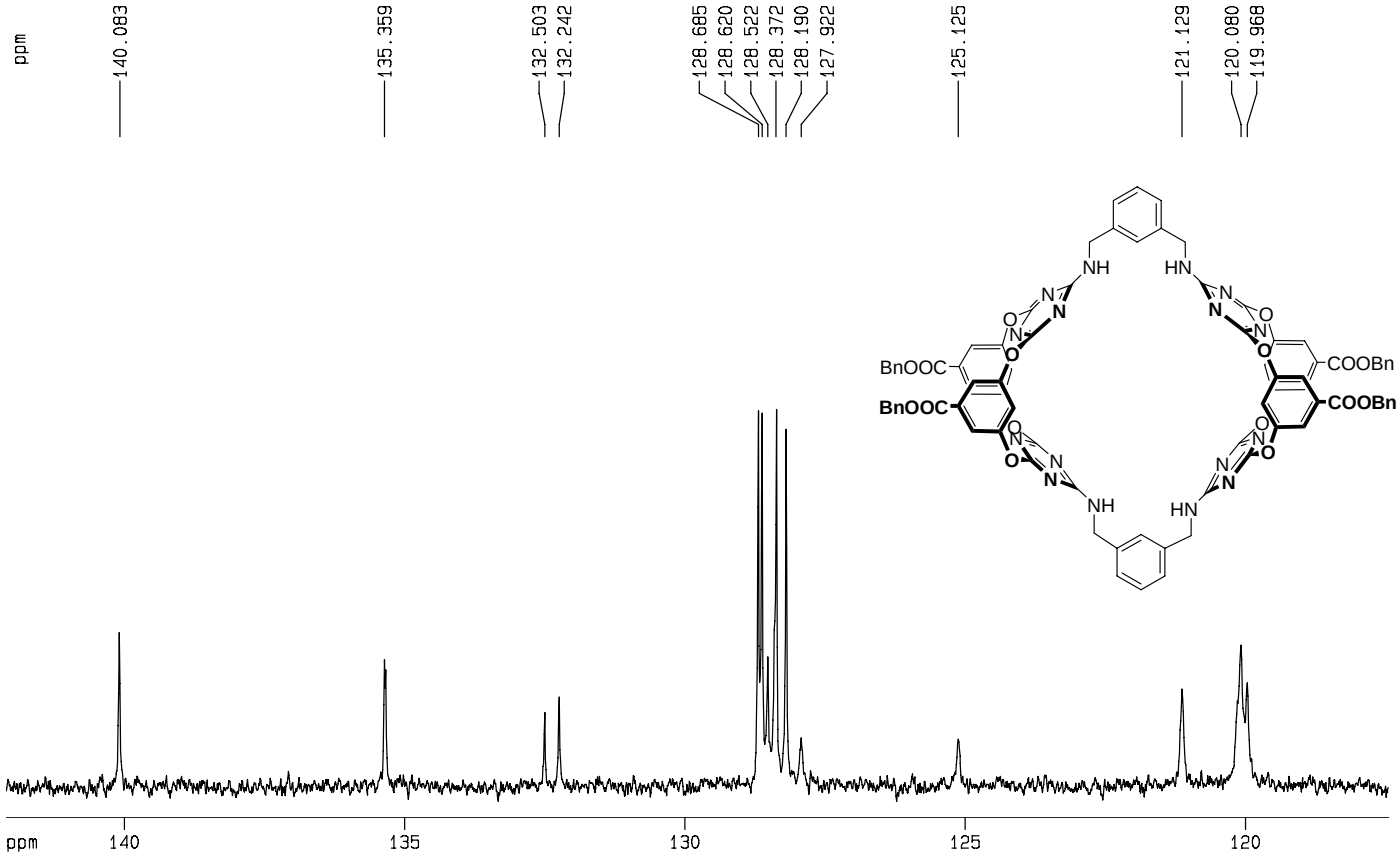
3-^{13}C NMR (expanded)

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======== CHANNEL f2 ========
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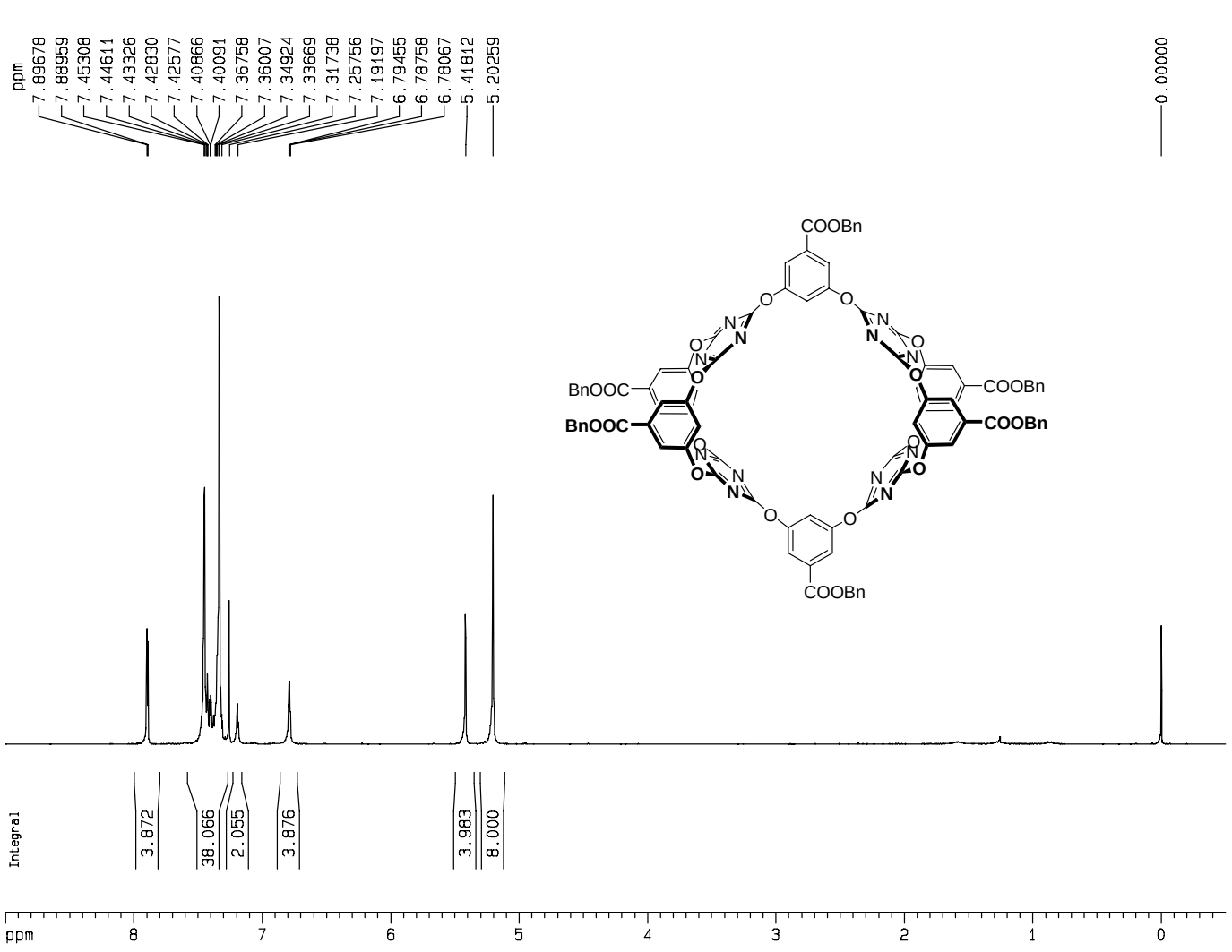
4-^{1}H NMR

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D1           2.00000000 sec

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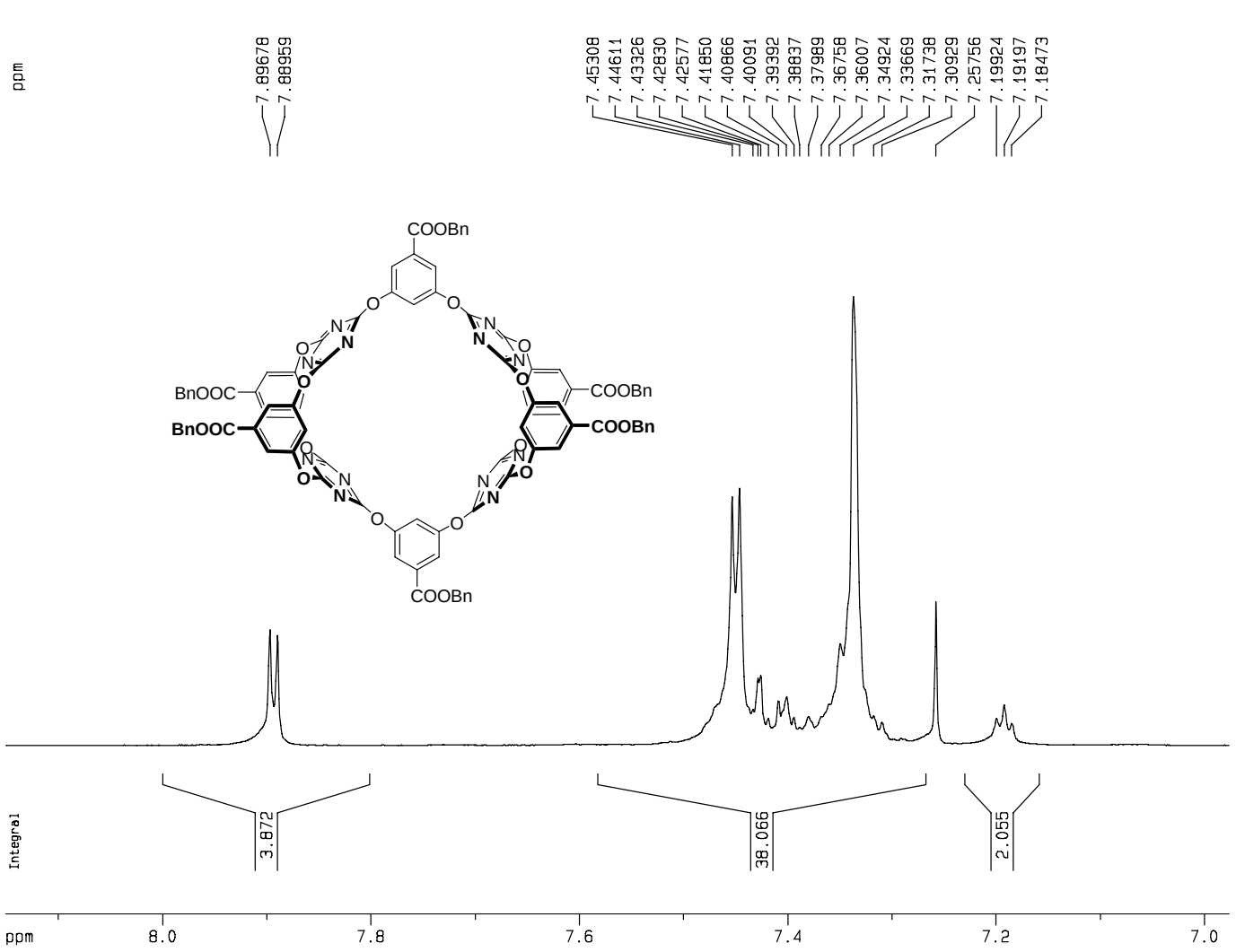
4-^{1}H NMR (expanded)

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DS                     2
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FIDRES             0.094190 Hz
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TE                298.0 K
D1             2.00000000 sec

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PL1                -1.00 dB
SFO1         300.1318534 MHz

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WDW                   EM
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1D NMR plot parameters
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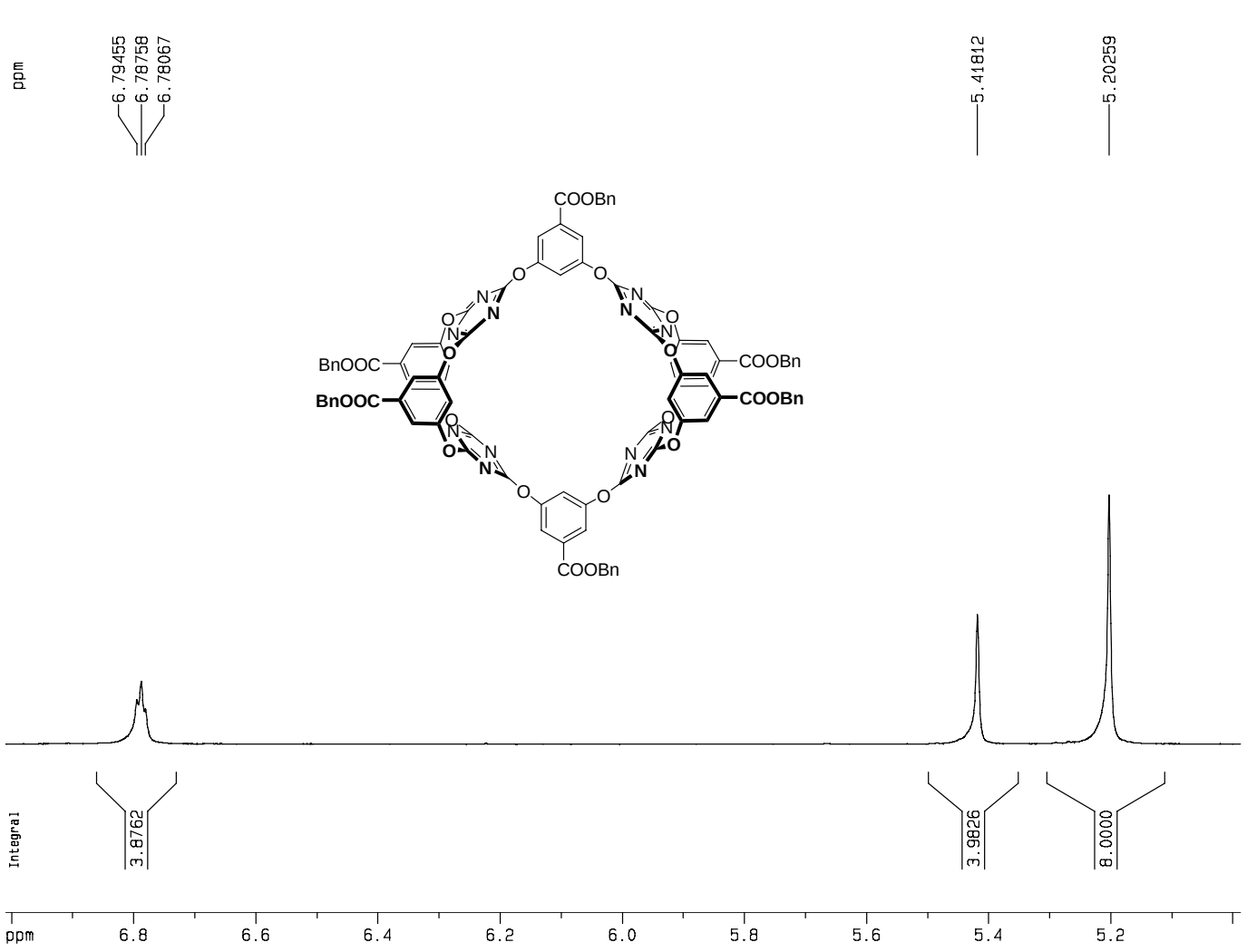
4-^{1}H NMR(expanded)

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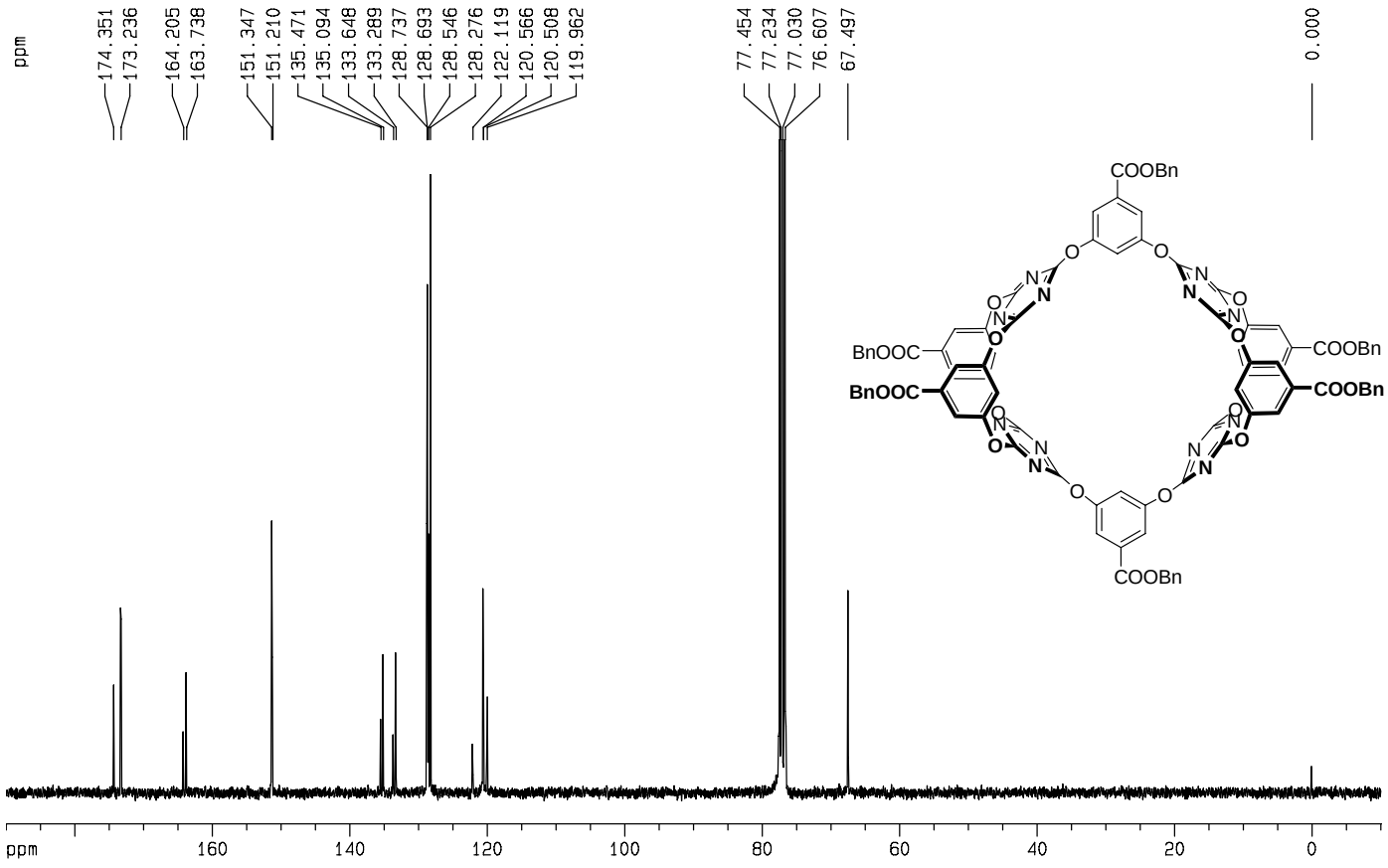
TD                65536
SOLVENT           CDCl3
NS                    7
DS                    2
SWH           6172.839 Hz
FIDRES        0.094190 Hz
AQ            5.3084660 sec
RG                  362
DW               81.000 usec
DE                6.00 usec
TE               298.0 K
D1           2.00000000 sec

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

F2 - Processing parameters
SI                32768
SF          300.1300069 MHz
WDW                  EM
SSB                   0
LB                0.30 Hz
GB                    0
PC                 1.00

1D NMR plot parameters
CX               22.00 cm
CY                8.00 cm
F1P               7.010 ppm
F1             2103.79 Hz
F2P               4.987 ppm
F2             1496.86 Hz
PPMCM         0.09192 ppm/cm
HZCM         27.58796 Hz/cm
```

4-^{13}C NMR

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Current Data Parameters
NAME         hou6-36-1-H
EXPNO                 10
PROCNO                 1

F2 - Acquisition Parameters
Date_           20060314
Time                0.06
INSTRUM           av300
PROBHD   5 mm DUL 13C-1
PULPROG          zgpg30
TD                65536
SOLVENT           CDCl3
NS                 7440
DS                    4
SWH           17985.611 Hz
FIDRES        0.274439 Hz
AQ            1.8219508 sec
RG               5792.6
DW               27.800 usec
DE                6.00 usec
TE               298.3 K
D1           2.00000000 sec
d11          0.03000000 sec
d12          0.00002000 sec

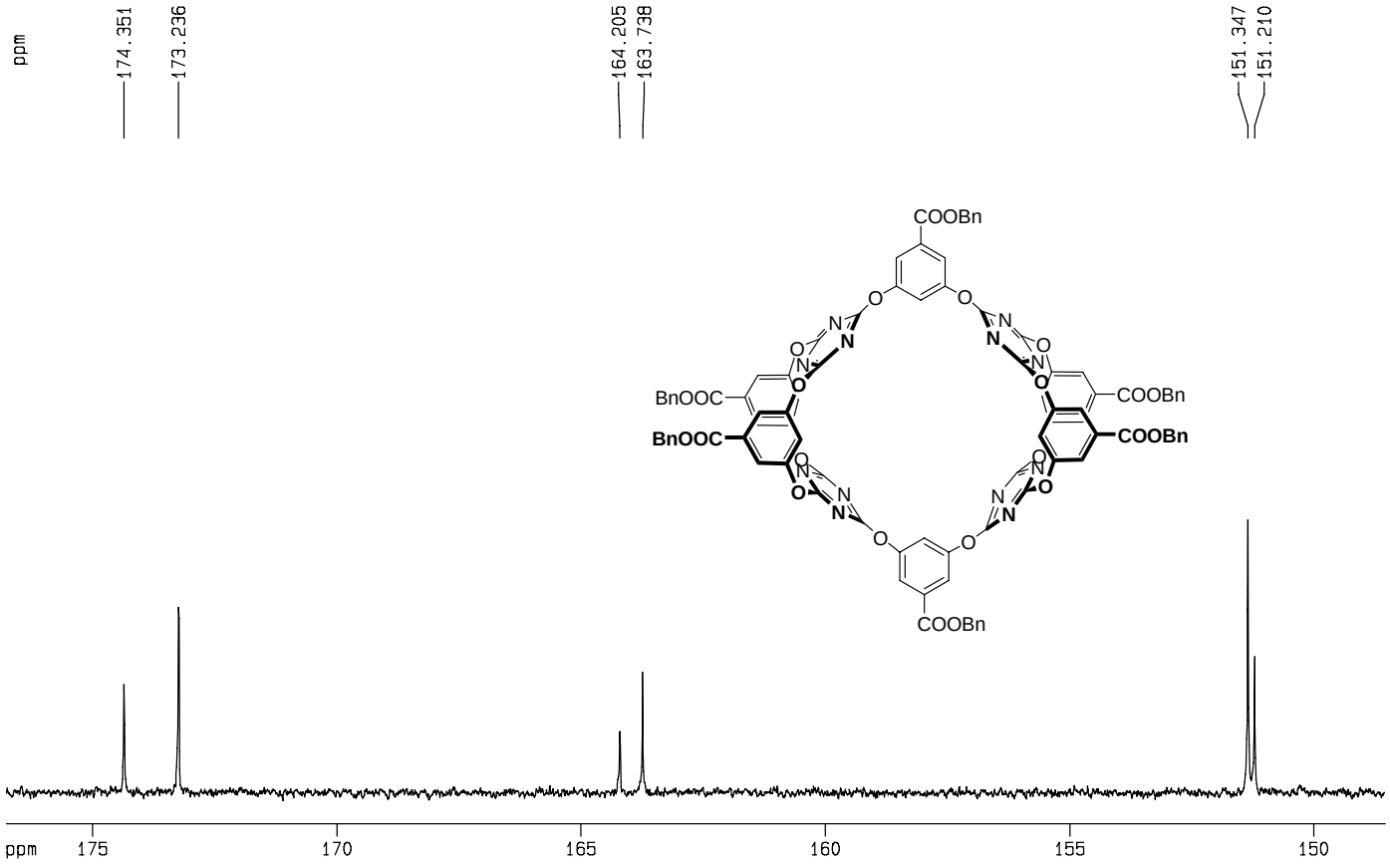
======== CHANNEL f1 ========
NUC1                 13C
P1                9.40 usec
PL1               -1.00 dB
SFO1         75.4752953 MHz

======== 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           75.4677475 MHz
WDW                    EM
SSB                    0
LB                  1.00 Hz
GB                     0
PC                  1.40

1D NMR plot parameters
CX                 22.00 cm
CY                 30.00 cm
F1P              190.000 ppm
F1             14338.87 Hz
F2P              -10.000 ppm
F2              -754.68 Hz
PPMCM            9.09091 ppm/cm
HZCM           686.07043 Hz/cm
```

4-^{13}C NMR (expanded)

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Current Data Parameters
NAME         hou6-36-1-H
EXPNO                 10
PROCNO                 1

F2 - Acquisition Parameters
Date_           20060314
Time                0.06
INSTRUM            av300
PROBHD   5 mm DUL 13C-1
PULPROG           zgpg30
TD                 65536
SOLVENT            CDCl3
NS                  7440
DS                     4
SWH           17985.611 Hz
FIDRES         0.274439 Hz
AQ             1.8219508 sec
RG                5792.6
DW                27.800 usec
DE                 6.00 usec
TE                298.3 K
D1            2.00000000 sec
d11           0.03000000 sec
d12           0.00002000 sec

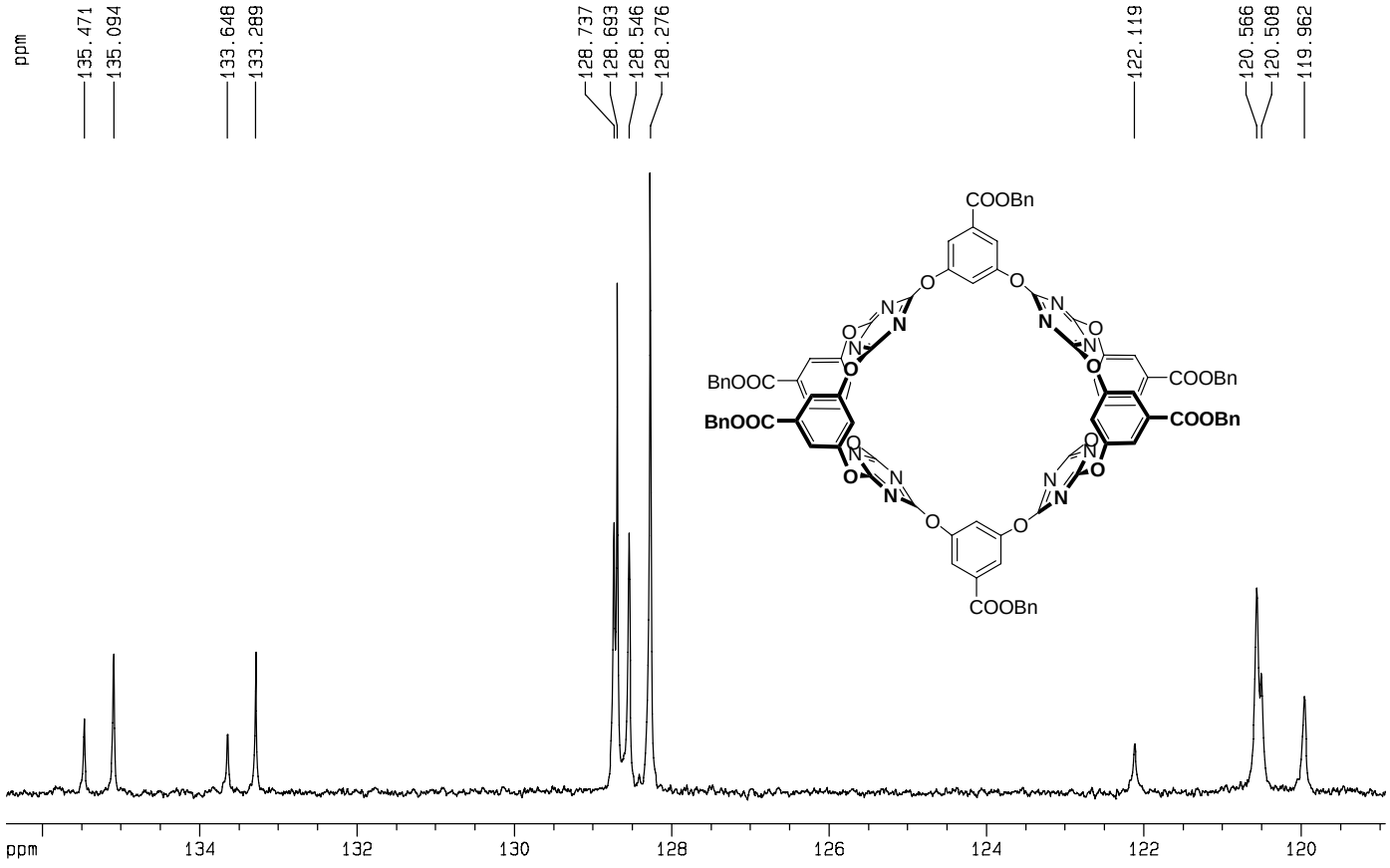
======== CHANNEL f1 ========
NUC1                 13C
P1                  9.40 usec
PL1                -1.00 dB
SFO1          75.4752953 MHz

======== 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            75.4677475 MHz
WDW                   EM
SSB                    0
LB                  1.00 Hz
GB                     0
PC                  1.40

1D NMR plot parameters
CX                 22.00 cm
CY                 30.00 cm
F1P               176.776 ppm
F1              13340.86 Hz
F2P                148.548 ppm
F2              11210.57 Hz
PPMCM           1.28308 ppm/cm
HZCM           96.83119 Hz/cm
```

4-^{13}C NMR (expanded)




```
Current Data Parameters
NAME         hou6-36-1-H
EXPNO                 10
PROCNO                 1

F2 - Acquisition Parameters
Date_           20060314
Time                0.06
INSTRUM            av300
PROBHD   5 mm DUL 13C-1
PULPROG           zgpg30
TD                 65536
SOLVENT            CDCl3
NS                  7440
DS                     4
SWH           17985.611 Hz
FIDRES         0.274439 Hz
AQ             1.8219508 sec
RG                5792.6
DW                27.800 usec
DE                 6.00 usec
TE                298.3 K
D1            2.00000000 sec
d11           0.03000000 sec
d12           0.00002000 sec

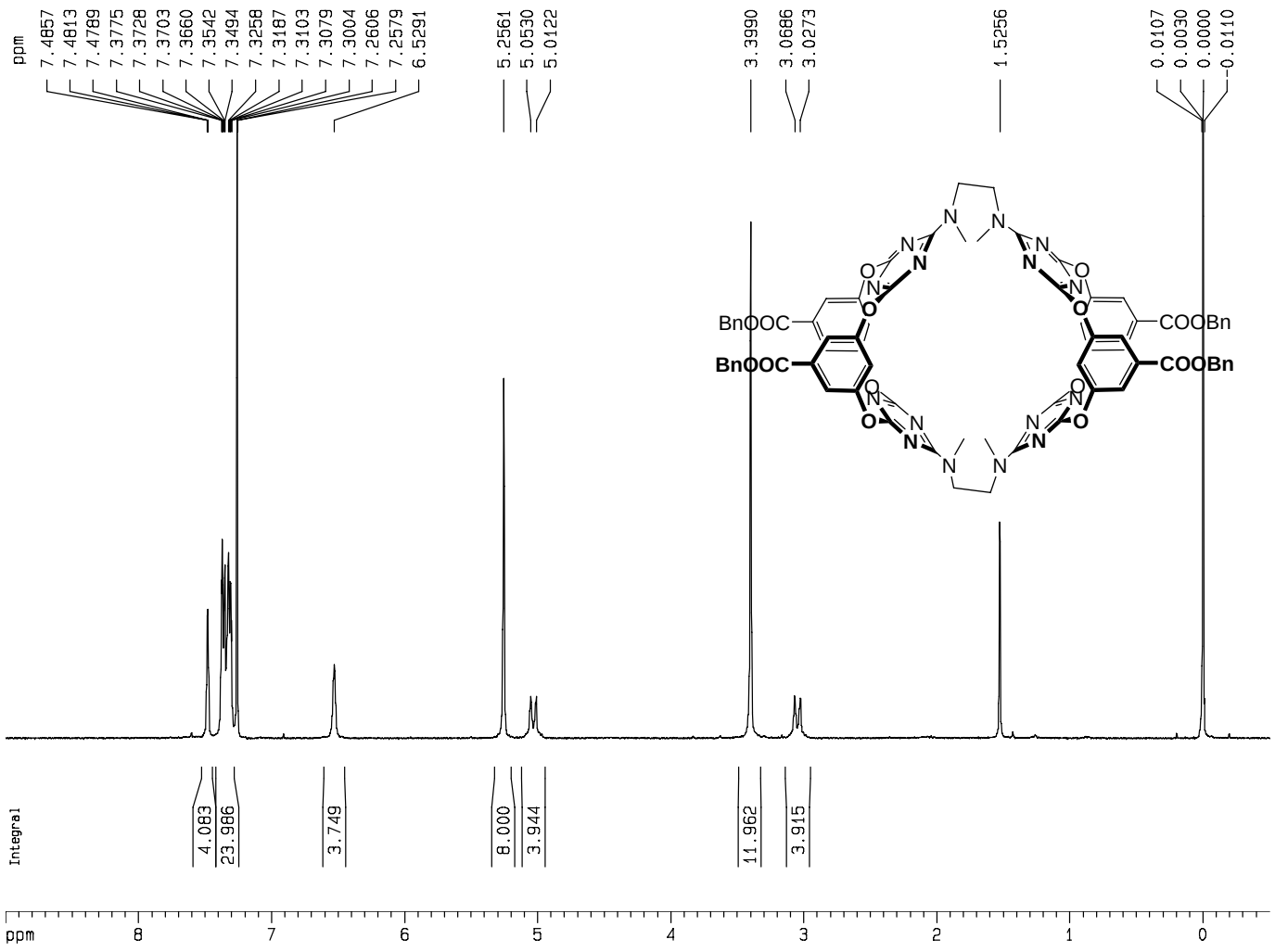
======== CHANNEL f1 ========
NUC1                 13C
P1                  9.40 usec
PL1                -1.00 dB
SFO1          75.4752953 MHz

======== 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            75.4677475 MHz
WDW                    EM
SSB                    0
LB                  1.00 Hz
GB                     0
PC                  1.40

1D NMR plot parameters
CX                 22.00 cm
CY                 30.00 cm
F1P               136.458 ppm
F1              10298.17 Hz
F2P               118.929 ppm
F2               8975.32 Hz
PPMCM             0.79676 ppm/cm
HZCM             60.12967 Hz/cm
```

5-^{1}H NMR




```
Current Data Parameters
NAME          hou6-42-1-H
EXPNO                  10
PROCNO                  1

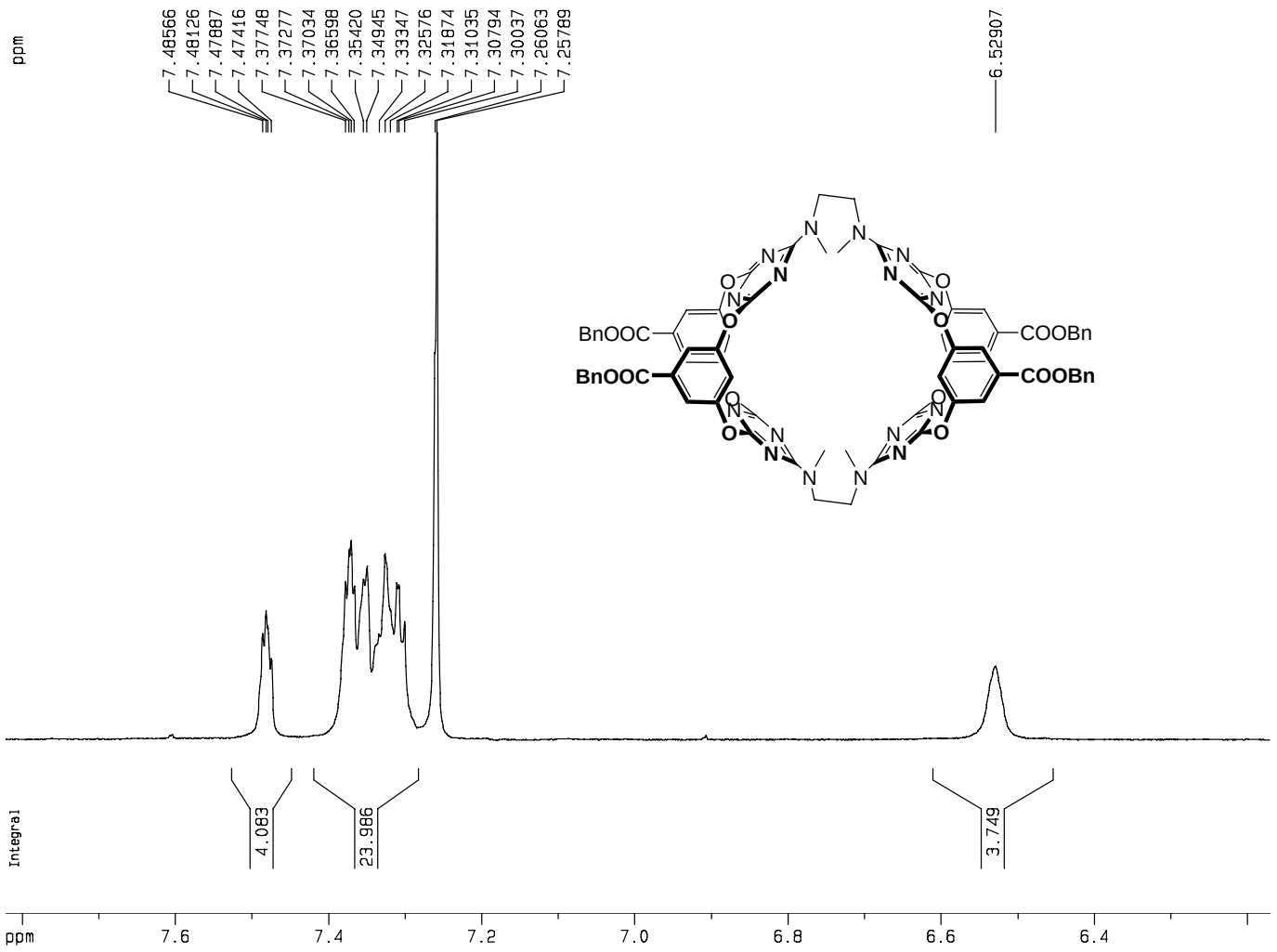
F2 - Acquisition Parameters
Date_            20060905
Time                16.10
INSTRUM             av300
PROBHD   5 mm DUL 13C-1
PULPROG              zg30
TD                  65536
SOLVENT             CDCl3
NS                     60
DS                      2
SWH             6172.839 Hz
FIDRES          0.094190 Hz
AQ              5.3084660 sec
RG                    512
DW                 81.000 usec
DE                  6.00 usec
TE                 302.4 K
D1              2.00000000 sec

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

F2 - Processing parameters
SI                  32768
SF            300.1300067 MHz
WDW                    EM
SSB                     0
LB                 0.30 Hz
GB                      0
PC                   1.00

1D NMR plot parameters
CX                 22.00 cm
CY                 18.00 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
```

5-^{1}H NMR (expanded)




```
Current Data Parameters
NAME         hou6-42-1-H
EXPNO                 10
PROCNO                 1

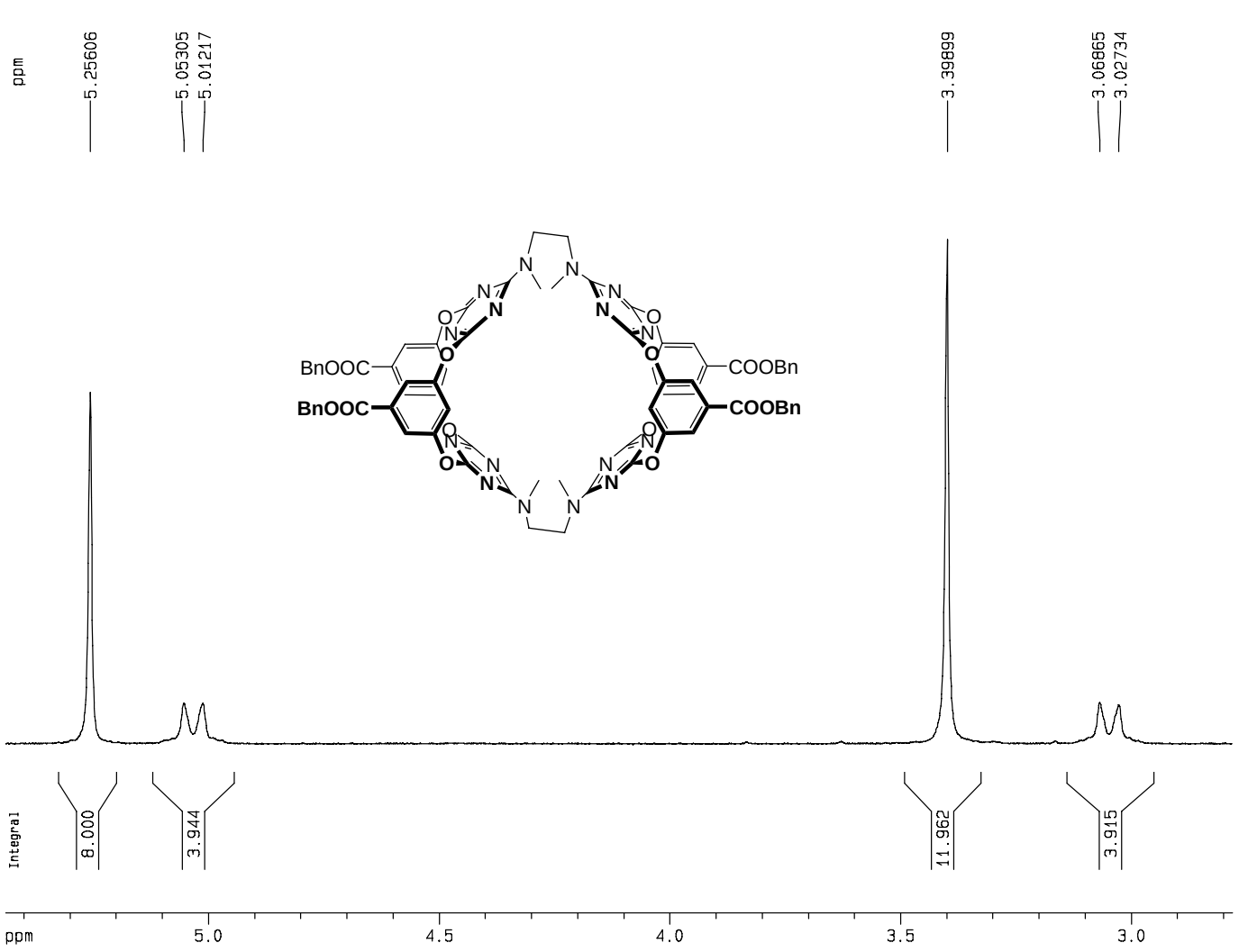
F2 - Acquisition Parameters
Date_           20060905
Time               16.10
INSTRUM            av300
PROBHD   5 mm DUL 13C-1
PULPROG             zg30
TD                 65536
SOLVENT            CDCl3
NS                    60
DS                     2
SWH            6172.839 Hz
FIDRES         0.094190 Hz
AQ             5.3084660 sec
RG                   512
DW                81.000 usec
DE                 6.00 usec
TE                302.4 K
D1            2.00000000 sec

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

F2 - Processing parameters
SI                 32768
SF           300.1300067 MHz
WDW                   EM
SSB                    0
LB                 0.30 Hz
GB                     0
PC                  1.00

1D NMR plot parameters
CX                22.00 cm
CY                18.00 cm
F1P               7.822 ppm
F1                2347.56 Hz
F2P               6.171 ppm
F2                1851.99 Hz
PPMCM          0.07505 ppm/cm
HZCM          22.52622 Hz/cm
```

5-^{1}H NMR (expanded)




```
Current Data Parameters
NAME         hou6-42-1-H
EXPNO                 10
PROCNO                 1

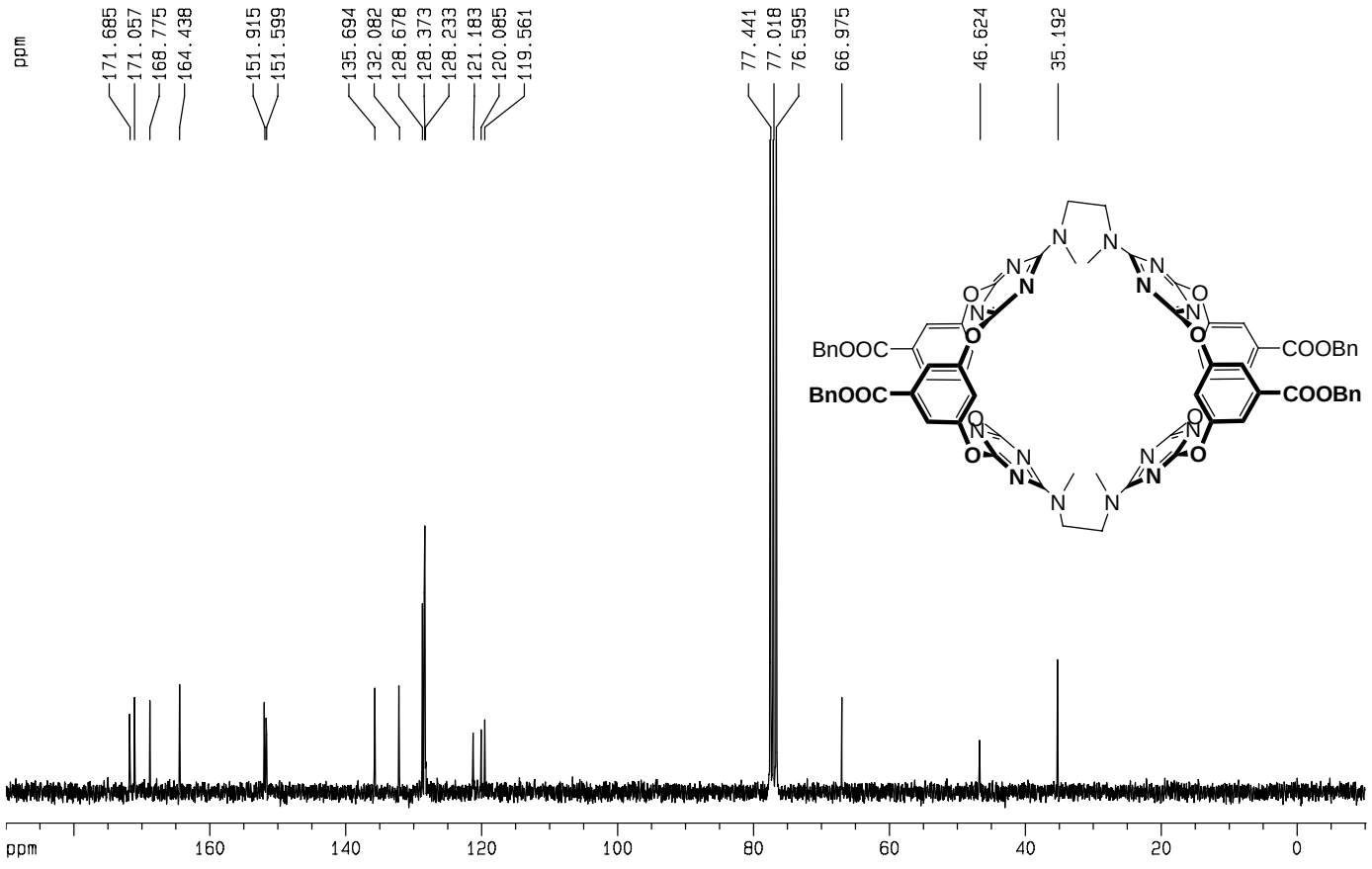
F2 - Acquisition Parameters
Date_           20060905
Time               16.10
INSTRUM            av300
PROBHD   5 mm DUL 13C-1
PULPROG             zg30
TD                 65536
SOLVENT            CDCl3
NS                    60
DS                     2
SWH             6172.839 Hz
FIDRES          0.094190 Hz
AQ              5.3084660 sec
RG                   512
DW                81.000 usec
DE                 6.00 usec
TE                302.4 K
D1             2.00000000 sec

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

F2 - Processing parameters
SI                 32768
SF           300.1300067 MHz
WDW                   EM
SSB                    0
LB                 0.30 Hz
GB                     0
PC                  1.00

1D NMR plot parameters
CX                22.00 cm
CY                18.00 cm
F1P                5.440 ppm
F1              1632.80 Hz
F2P                2.781 ppm
F2               834.80 Hz
PPMCM           0.12086 ppm/cm
HZCM           36.27276 Hz/cm
```

5-^{13}C NMR




```
Current Data Parameters
NAME         hou6-42-1-C
EXPNO                 10
PROCNO                 1

F2 - Acquisition Parameters
Date_           20061018
Time                8.31
INSTRUM            av300
PROBHD   5 mm DUL 13C-1
PULPROG          zgpg30
TD                65536
SOLVENT           CDCl3
NS                  362
DS                    4
SWH           17985.611 Hz
FIDRES        0.274439 Hz
AQ            1.8219508 sec
RG                 2048
DW               27.800 usec
DE                6.00 usec
TE                299.6 K
D1           2.00000000 sec
d11          0.03000000 sec
d12          0.00002000 sec

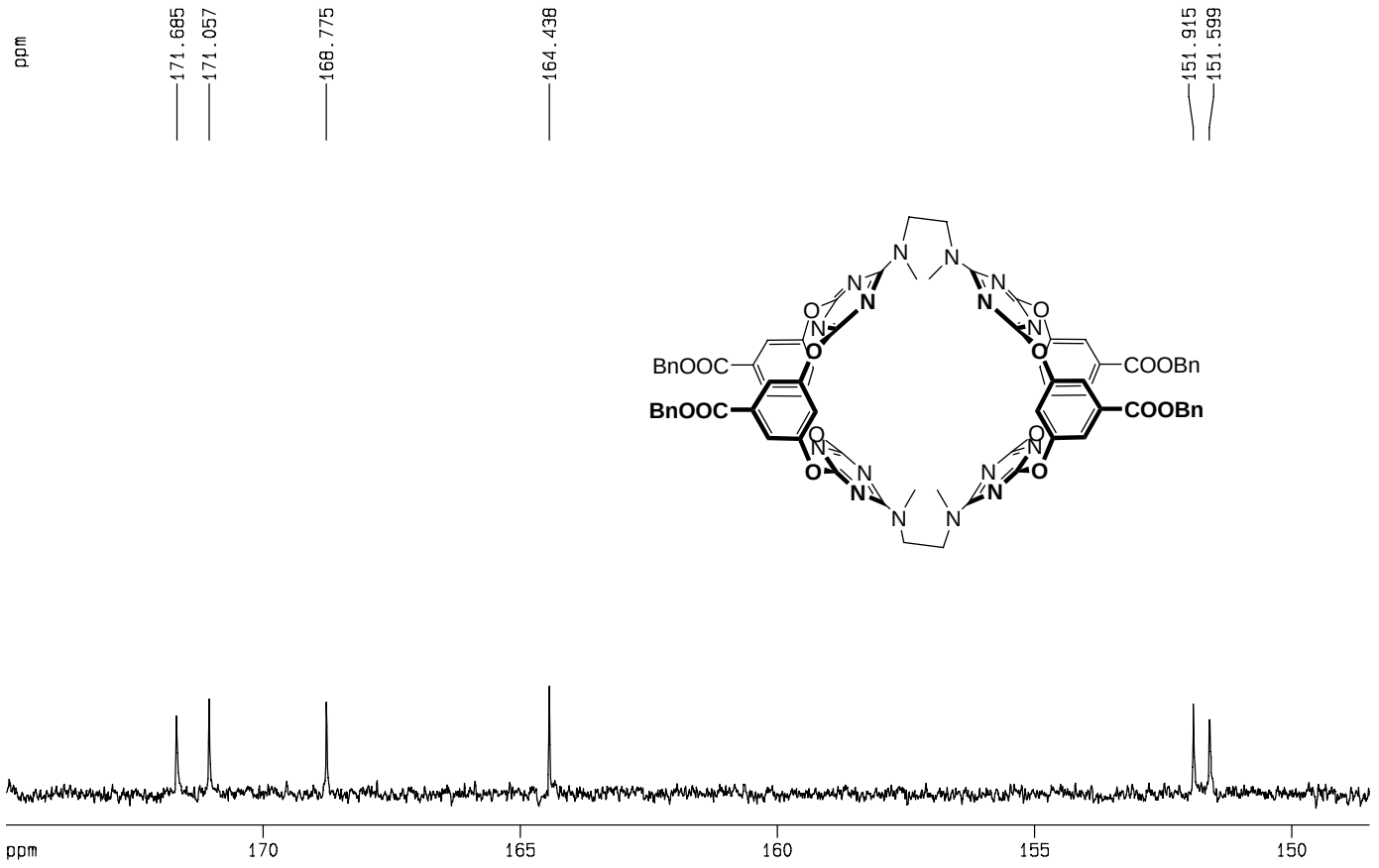
======== CHANNEL f1 ========
NUC1                13C
P1                9.40 usec
PL1               -1.00 dB
SFO1         75.4752953 MHz

======== 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                12.00 cm
F1P             190.000 ppm
F1             14338.87 Hz
F2P             -10.000 ppm
F2              -754.68 Hz
PPMCM           9.09091 ppm/cm
HZCM          686.07043 Hz/cm
```

5-^{13}C NMR (expanded)




```
Current Data Parameters
NAME         hou6-42-1-C
EXPNO                 10
PROCNO                 1

F2 - Acquisition Parameters
Date_           20061018
Time                8.31
INSTRUM            av300
PROBHD   5 mm DUL 13C-1
PULPROG           zgpg30
TD                 65536
SOLVENT            CDCl3
NS                   362
DS                     4
SWH           17985.611 Hz
FIDRES         0.274439 Hz
AQ             1.8219508 sec
RG                  2048
DW                27.800 usec
DE                 6.00 usec
TE                299.6 K
D1           2.00000000 sec
d11          0.03000000 sec
d12          0.00002000 sec

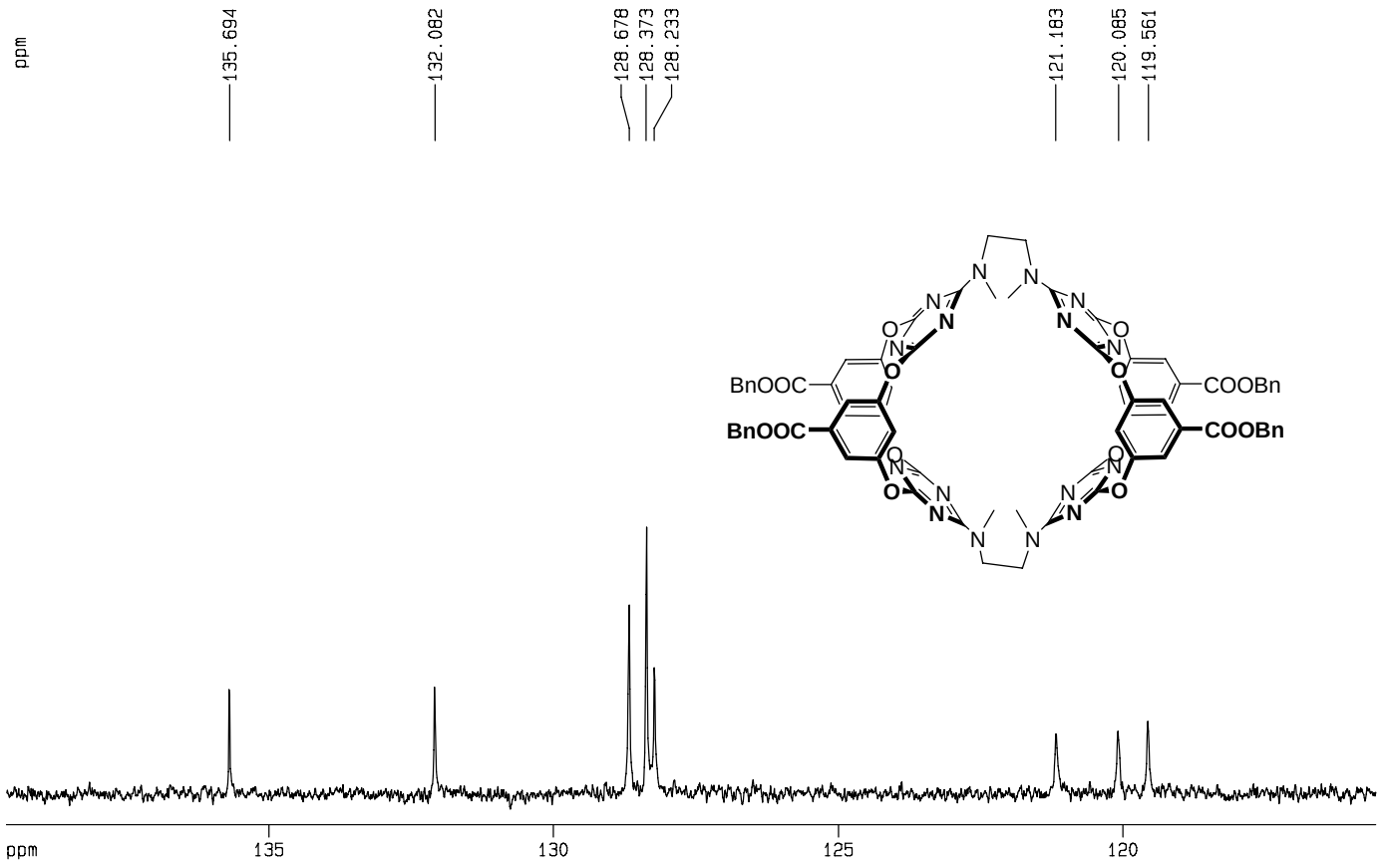
======== CHANNEL f1 ========
NUC1                13C
P1                 9.40 usec
PL1               -1.00 dB
SFO1         75.4752953 MHz

======== 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                 12.00 cm
F1P              174.998 ppm
F1              13206.73 Hz
F2P              148.484 ppm
F2              11205.78 Hz
PPMCM            1.20518 ppm/cm
HZCM            90.95229 Hz/cm
```

5-^{13}C NMR (expanded)




```
Current Data Parameters
NAME         hou6-42-1-C
EXPNO                 10
PROCNO                 1

F2 - Acquisition Parameters
Date_           20061018
Time                8.31
INSTRUM            av300
PROBHD   5 mm DUL 13C-1
PULPROG           zgpg30
TD                 65536
SOLVENT            CDCl3
NS                   362
DS                     4
SWH            17985.611 Hz
FIDRES         0.274439 Hz
AQ             1.8219508 sec
RG                  2048
DW                27.800 usec
DE                 6.00 usec
TE                299.6 K
D1           2.00000000 sec
d11          0.03000000 sec
d12          0.00002000 sec

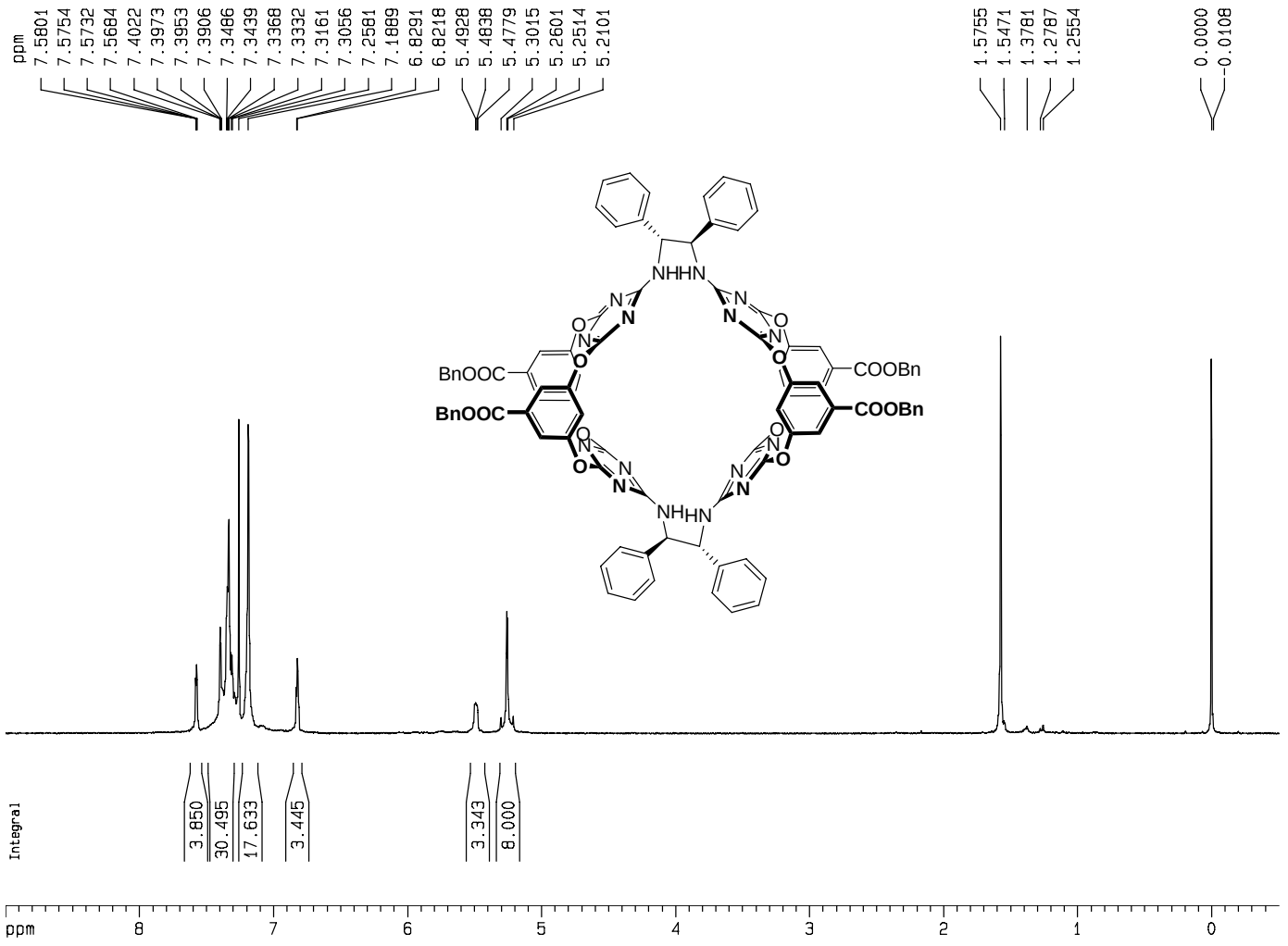
======== CHANNEL f1 ========
NUC1                13C
P1                 9.40 usec
PL1               -1.00 dB
SFO1          75.4752953 MHz

======== 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                 12.00 cm
F1P              139.607 ppm
F1             10535.86 Hz
F2P              115.564 ppm
F2              8721.32 Hz
PPMCM          1.09291 ppm/cm
HZCM          82.47910 Hz/cm
```

6-^{1}H NMR




```
Current Data Parameters
NAME                  hou6-55
EXPNO                      20
PROCNO                      1

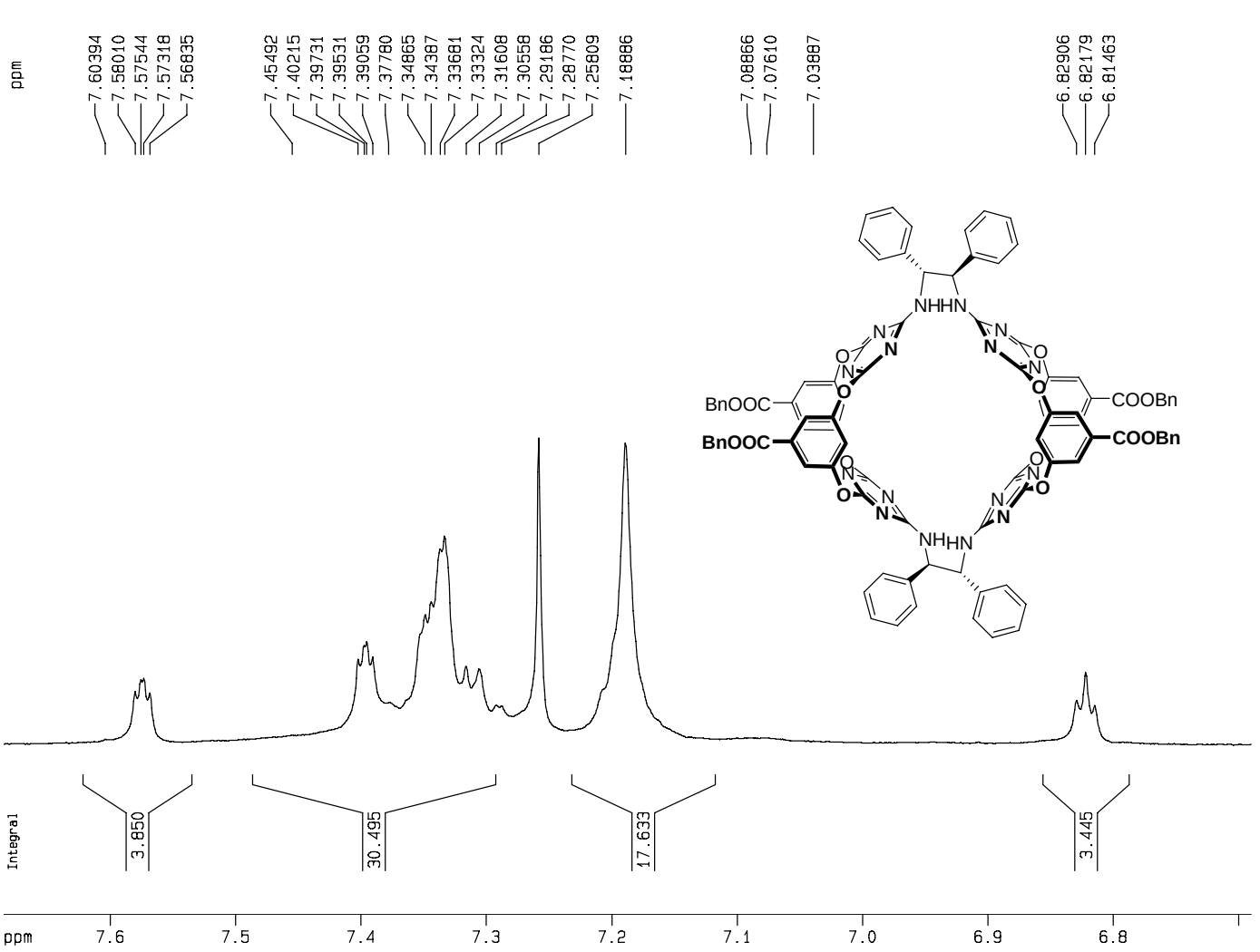
F2 - Acquisition Parameters
Date_               20060628
Time                   16.39
INSTRUM                av300
PROBHD   5 mm DUL 13C-1
PULPROG                 zg30
TD                     65536
SOLVENT                CDCl3
NS                        16
DS                         2
SWH                6172.839 Hz
FIDRES             0.094190 Hz
AQ                 5.3084660 sec
RG                       362
DW                    81.000 usec
DE                     6.00 usec
TE                    301.1 K
D1                 2.00000000 sec

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

F2 - Processing parameters
SI                      32768
SF                300.1300070 MHz
WDW                        EM
SSB                         0
LB                      0.30 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/c
HZCM              129.60159 Hz/cm
```

6-^{1}H NMR (expanded)




```
Current Data Parameters
NAME                  hou6-55
EXPNO                      20
PROCNO                      1

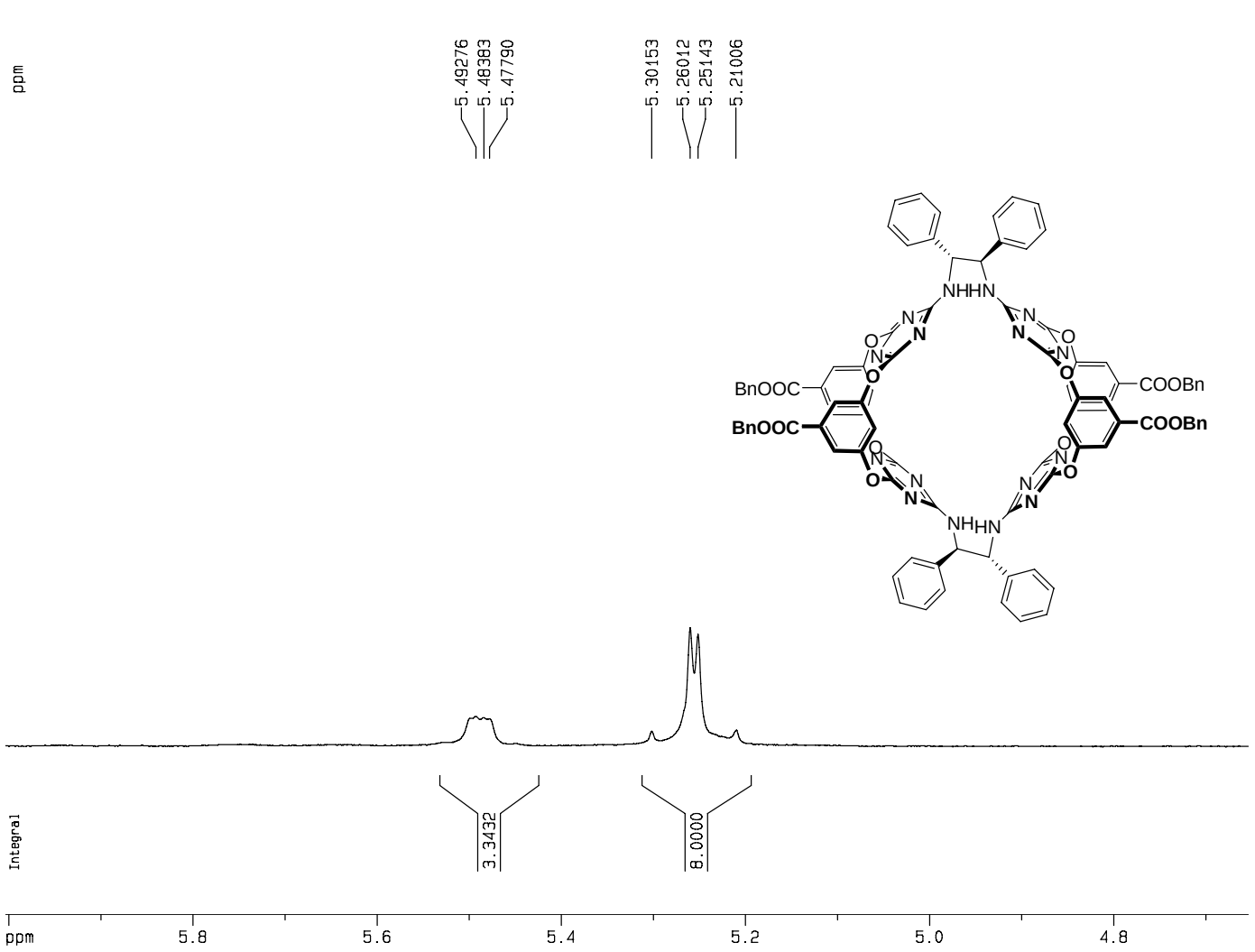
F2 - Acquisition Parameters
Date_               20060628
Time                   16.39
INSTRUM                av300
PROBHD   5 mm DUL 13C-1
PULPROG                zg30
TD                     65536
SOLVENT                CDCl3
NS                        16
DS                         2
SWH                6172.839 Hz
FIDRES             0.094190 Hz
AQ                 5.3084660 sec
RG                       362
DW                  81.000 usec
DE                   6.00 usec
TE                   301.1 K
D1                 2.00000000 sec

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

F2 - Processing parameters
SI                       32768
SF               300.1300070 MHz
WDW                        EM
SSB                         0
LB                     0.30 Hz
GB                          0
PC                       1.00

1D NMR plot parameters
CX                     22.00 cm
CY                      7.00 cm
F1P                    7.685 ppm
F1                   2306.48 Hz
F2P                    6.690 ppm
F2                   2007.83 Hz
PPMCM                0.04523 ppm/c
HZCM                13.57506 Hz/cm
```

6-^{1}H NMR (expanded)




```
Current Data Parameters
NAME                  hou6-55
EXPNO                      20
PROCNO                      1

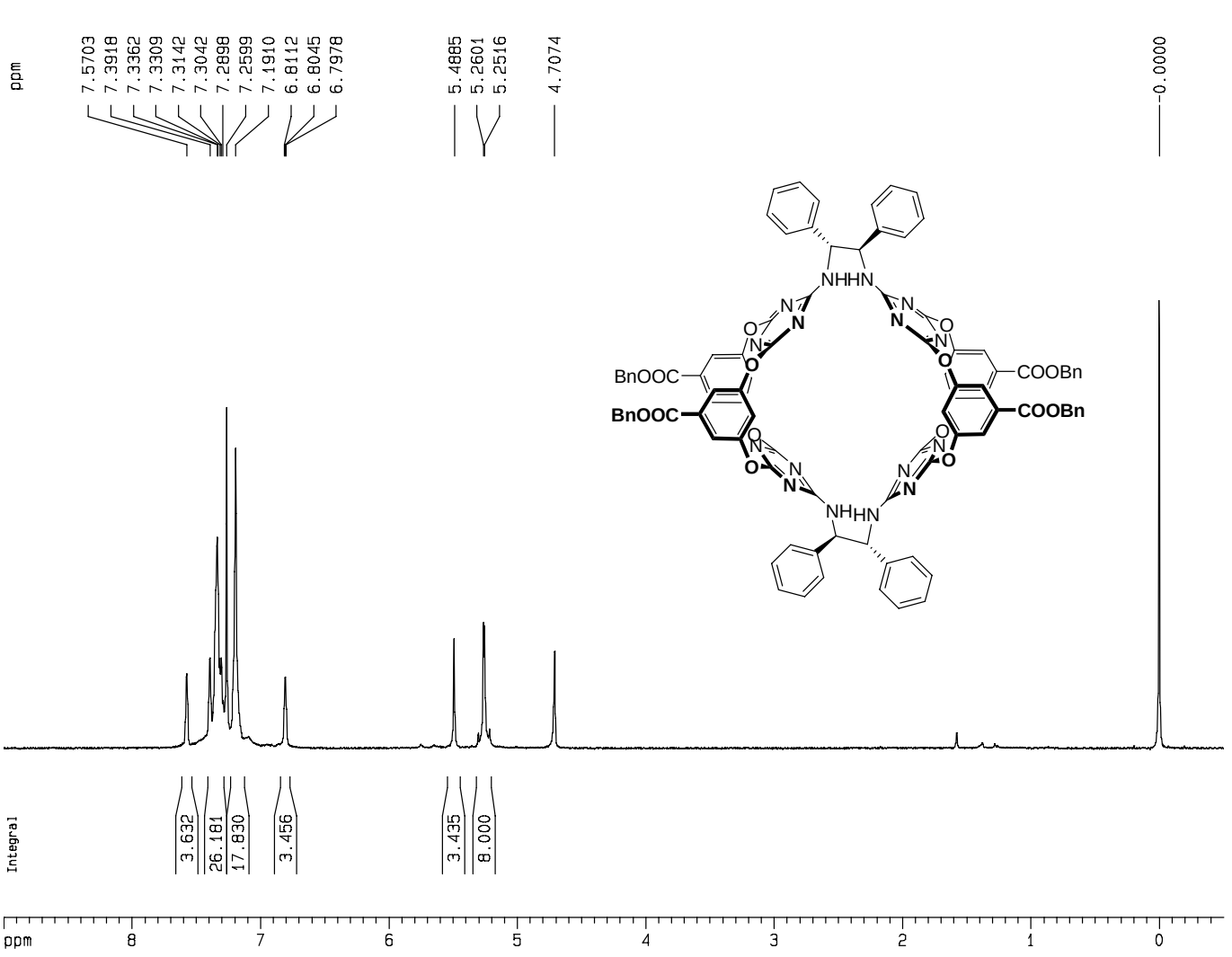
F2 - Acquisition Parameters
Date_               20060628
Time                   16.39
INSTRUM                av300
PROBHD   5 mm DUL 13C-1
PULPROG                zg30
TD                     65536
SOLVENT                CDCl3
NS                        16
DS                         2
SWH                 6172.839 Hz
FIDRES              0.094190 Hz
AQ                  5.3084660 sec
RG                       362
DW                     81.000 usec
DE                      6.00 usec
TE                     301.1 K
D1                 2.00000000 sec

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

F2 - Processing parameters
SI                       32768
SF                300.1300070 MHz
WDW                        EM
SSB                         0
LB                      0.30 Hz
GB                          0
PC                       1.00

1D NMR plot parameters
CX                      22.00 cm
CY                       7.00 cm
F1P                     6.003 ppm
F1                    1801.80 Hz
F2P                     4.654 ppm
F2                    1396.68 Hz
PPMCM                0.06135 ppm/c
HZCM                18.41428 Hz/cm
```

6-^{1}H NMR-D_2O




```
Current Data Parameters
NAME          hou6-55-DH
EXPNO                 10
PROCNO                 1

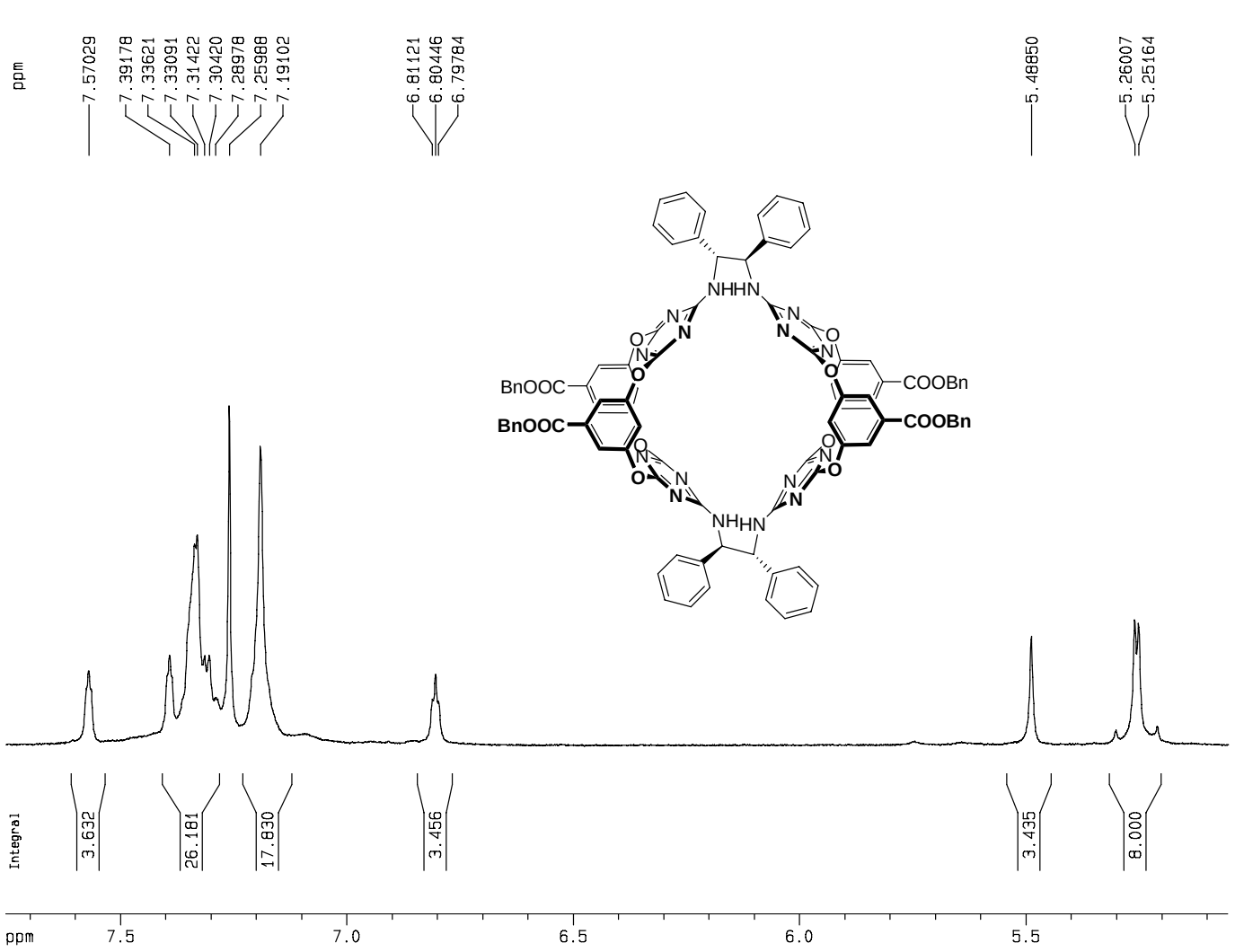
F2 - Acquisition Parameters
Date_           20060906
Time                8.43
INSTRUM            av300
PROBHD   5 mm DUL 13C-1
PULPROG             zg30
TD                 65536
SOLVENT            CDCl3
NS                    16
DS                     2
SWH             6172.839 Hz
FIDRES          0.094190 Hz
AQ              5.3084660 sec
RG                574.7
DW                81.000 usec
DE                 6.00 usec
TE                302.7 K
D1             2.00000000 sec

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

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

1D NMR plot parameters
CX                 22.00 cm
CY                  8.00 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
```

6-^{1}H NMR-D_2O (expanded)




```
Current Data Parameters
NAME          hou6-55-DH
EXPNO                 10
PROCNO                 1

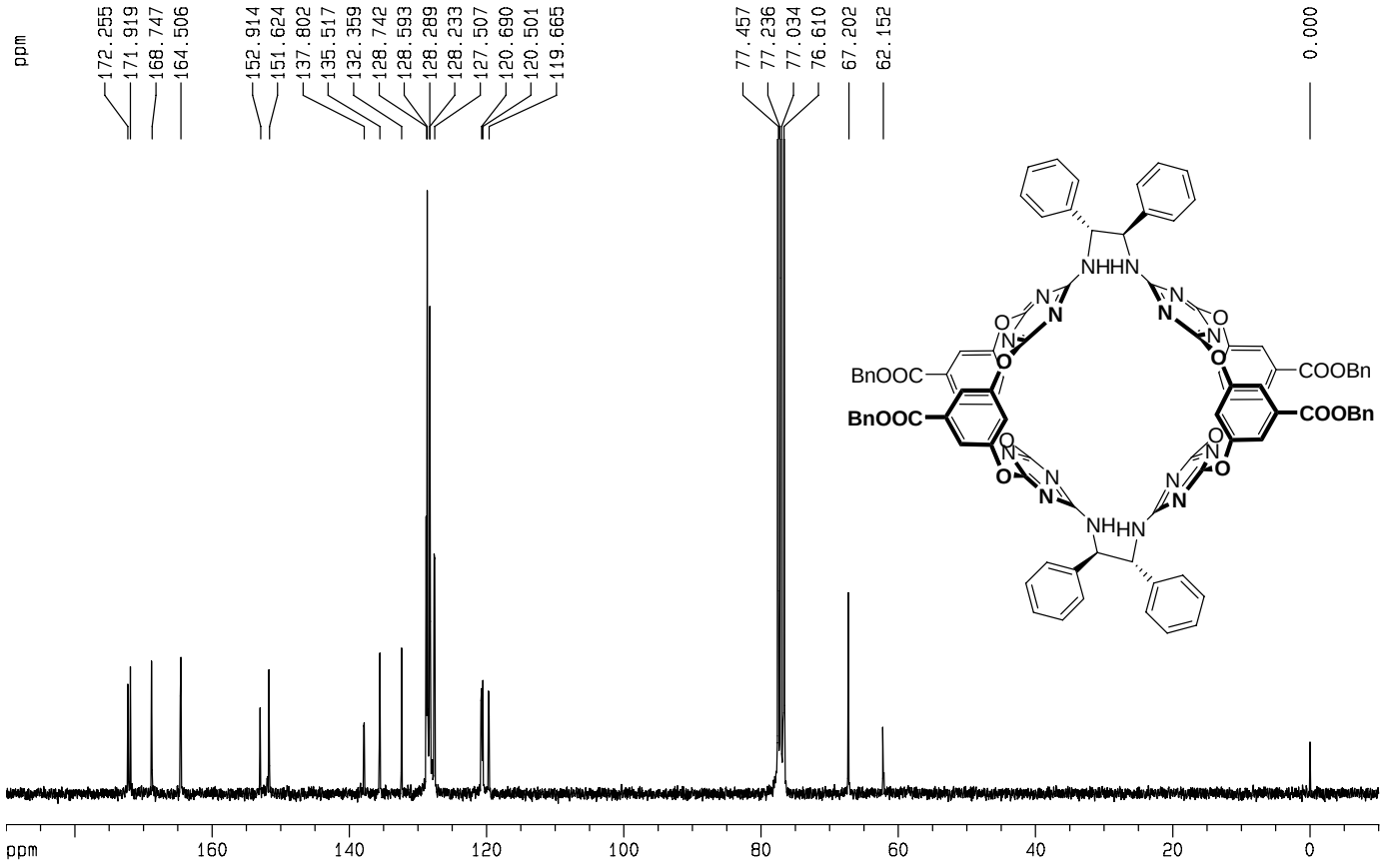
F2 - Acquisition Parameters
Date_           20060906
Time                8.43
INSTRUM          av300
PROBHD   5 mm DUL 13C-1
PULPROG            zg30
TD                65536
SOLVENT           CDCl3
NS                   16
DS                    2
SWH            6172.839 Hz
FIDRES         0.094190 Hz
AQ             5.3084660 sec
RG                574.7
DW               81.000 usec
DE                6.00 usec
TE               302.7 K
D1            2.00000000 sec

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

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

1D NMR plot parameters
CX                22.00 cm
CY                 8.00 cm
F1P                7.753 ppm
F1              2327.02 Hz
F2P                5.054 ppm
F2              1516.95 Hz
PPMCM          0.12269 ppm/cm
HZCM           36.82149 Hz/cm
```

6-^{13}C NMR

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Current Data Parameters
NAME                 hou6-55-C
EXPNO                       10
PROCNO                       1

F2 - Acquisition Parameters
Date_               20061016
Time                    23.28
INSTRUM                 av300
PROBHD   5 mm DUL 13C-1
PULPROG                 zgpg30
TD                       65536
SOLVENT                 CDCl3
NS                       8331
DS                          4
SWH                 17985.611 Hz
FIDRES                0.274439 Hz
AQ                    1.8219508 sec
RG                       1448.2
DW                      27.800 usec
DE                       6.00 usec
TE                       299.5 K
D1                 2.00000000 sec
d11                0.03000000 sec
d12                0.00002000 sec

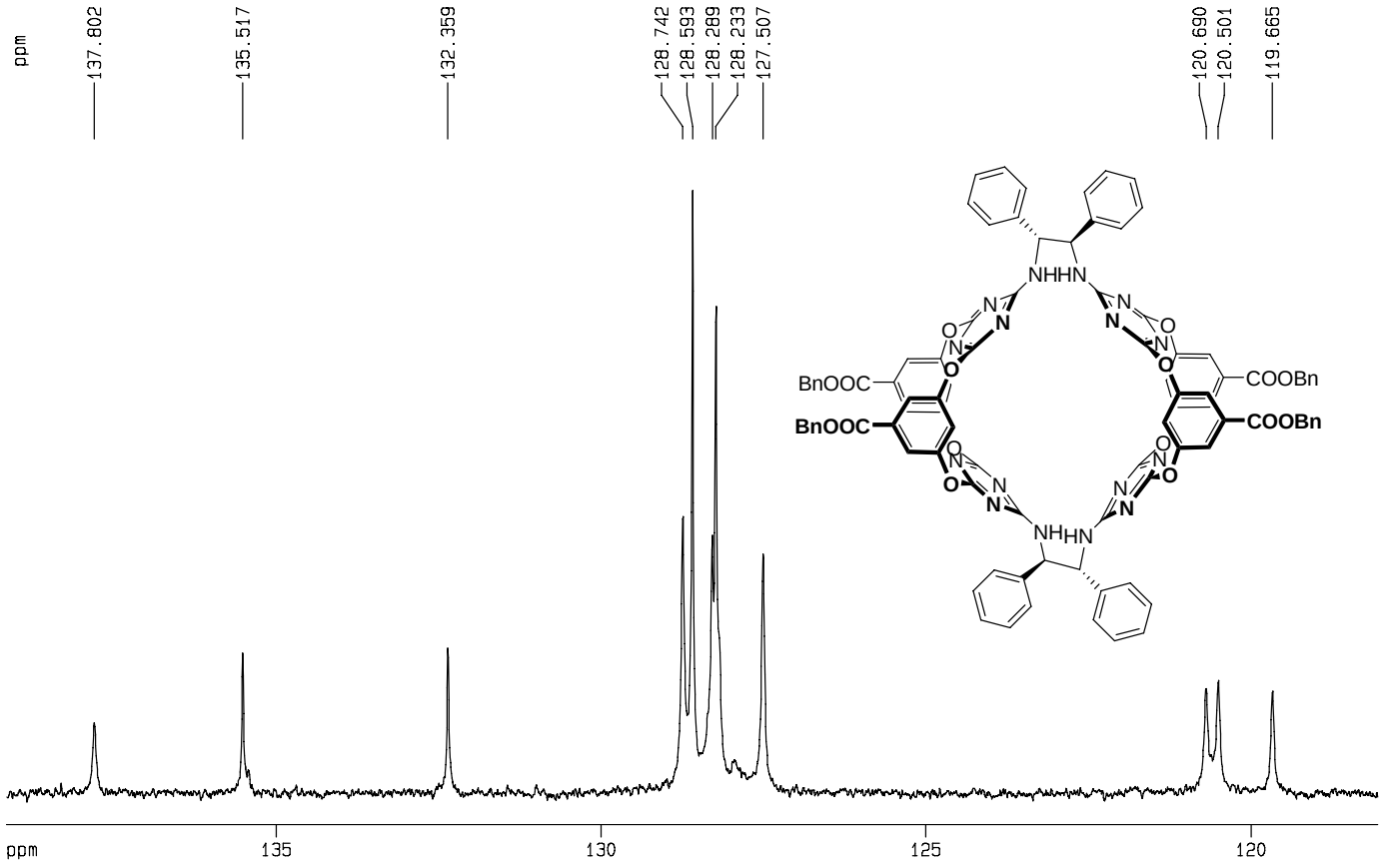
======== CHANNEL f1 ========
NUC1                        13C
P1                        9.40 usec
PL1                      -1.00 dB
SFO1                75.4752953 MHz

======== 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                  75.4677477 MHz
WDW                         EM
SSB                          0
LB                        1.00 Hz
GB                           0
PC                        1.40

1D NMR plot parameters
CX                       22.00 cm
CY                       35.00 cm
F1P                     190.000 ppm
F1                    14338.87 Hz
F2P                     -10.000 ppm
F2                     -754.68 Hz
PPMCM                  9.09091 ppm/cm
HZCM                686.07043 Hz/cm
```

6-^{13}C NMR (expanded)

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Current Data Parameters
NAME                 hou6-55-C
EXPNO                       10
PROCNO                       1

F2 - Acquisition Parameters
Date_               20061016
Time                    23.28
INSTRUM                 av300
PROBHD   5 mm DUL 13C-1
PULPROG                 zgpg30
TD                       65536
SOLVENT                  CDCl3
NS                        8331
DS                           4
SWH                 17985.611 Hz
FIDRES                0.274439 Hz
AQ                    1.8219508 sec
RG                       1448.2
DW                      27.800 usec
DE                       6.00 usec
TE                      299.5 K
D1                  2.00000000 sec
d11                 0.03000000 sec
d12                 0.00002000 sec

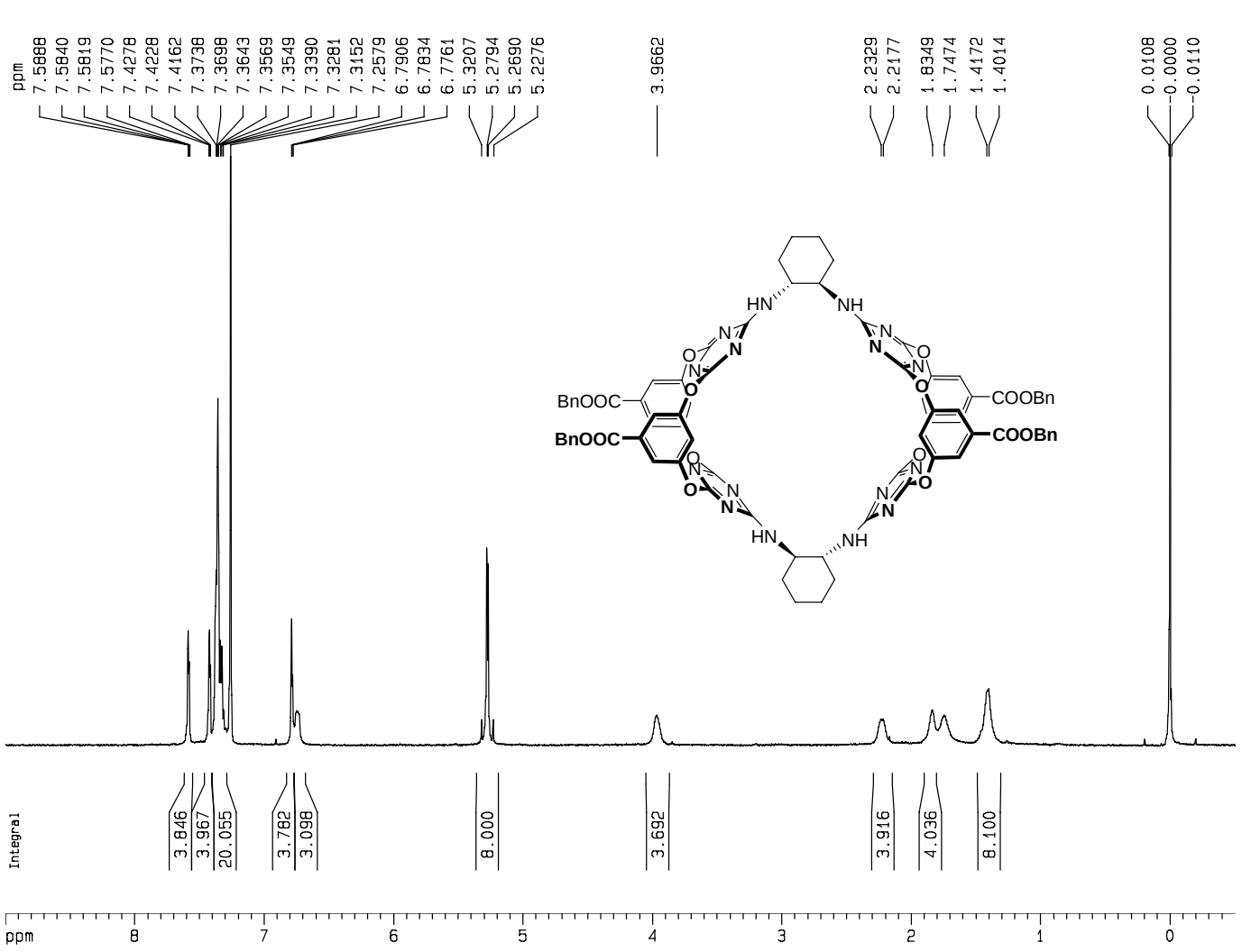
======== CHANNEL f1 ========
NUC1                       13C
P1                        9.40 usec
PL1                      -1.00 dB
SFO1                75.4752953 MHz

======== 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                  75.4677477 MHz
WDW                         EM
SSB                          0
LB                        1.00 Hz
GB                           0
PC                        1.40

1D NMR plot parameters
CX                       22.00 cm
CY                       35.00 cm
F1P                     139.153 ppm
F1                    10501.53 Hz
F2P                     118.045 ppm
F2                     8908.60 Hz
PPMCM                 0.95943 ppm/cm
HZCM                 72.40611 Hz/cm
```

7-^{1}H NMR

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Current Data Parameters
NAME            hou7-4-H
EXPNO                 10
PROCNO                 1

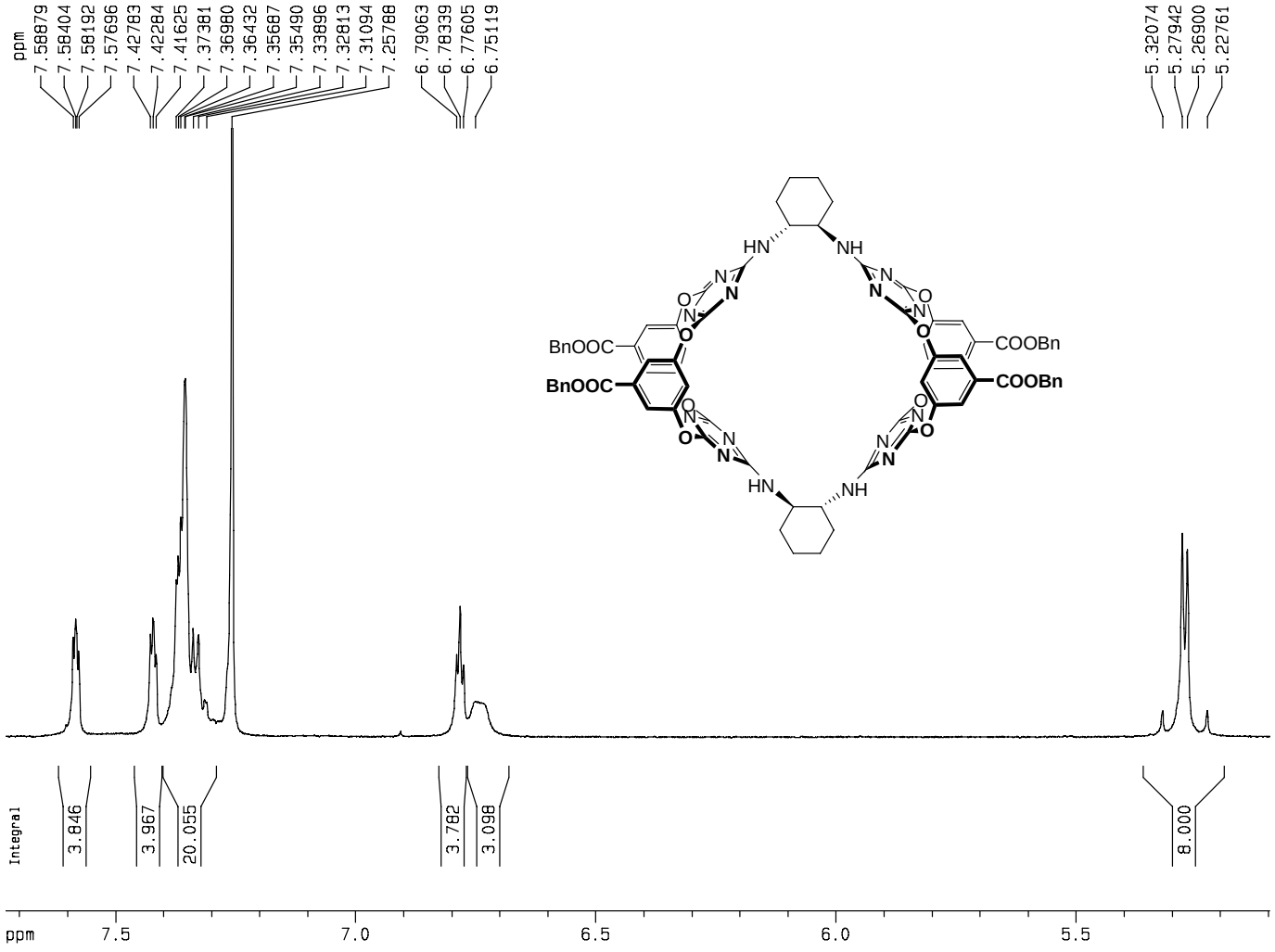
F2 - Acquisition Parameters
Date_           20060904
Time               23.14
INSTRUM            av300
PROBHD   5 mm DUL 13C-1
PULPROG             zg30
TD                 65536
SOLVENT            CDCl3
NS                    92
DS                     2
SWH             6172.839 Hz
FIDRES          0.094190 Hz
AQ              5.3084660 sec
RG                 574.7
DW                81.000 usec
DE                 6.00 usec
TE                302.6 K
D1             2.00000000 sec

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

F2 - Processing parameters
SI                 32768
SF          300.1300069 MHz
WDW                   EM
SSB                    0
LB                0.30 Hz
GB                     0
PC                1.00

1D NMR plot parameters
CX                22.00 cm
CY                25.00 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
```

7-^{1}H NMR (expanded)




```
Current Data Parameters
NAME                  hou7-4-H
EXPNO                       10
PROCNO                       1

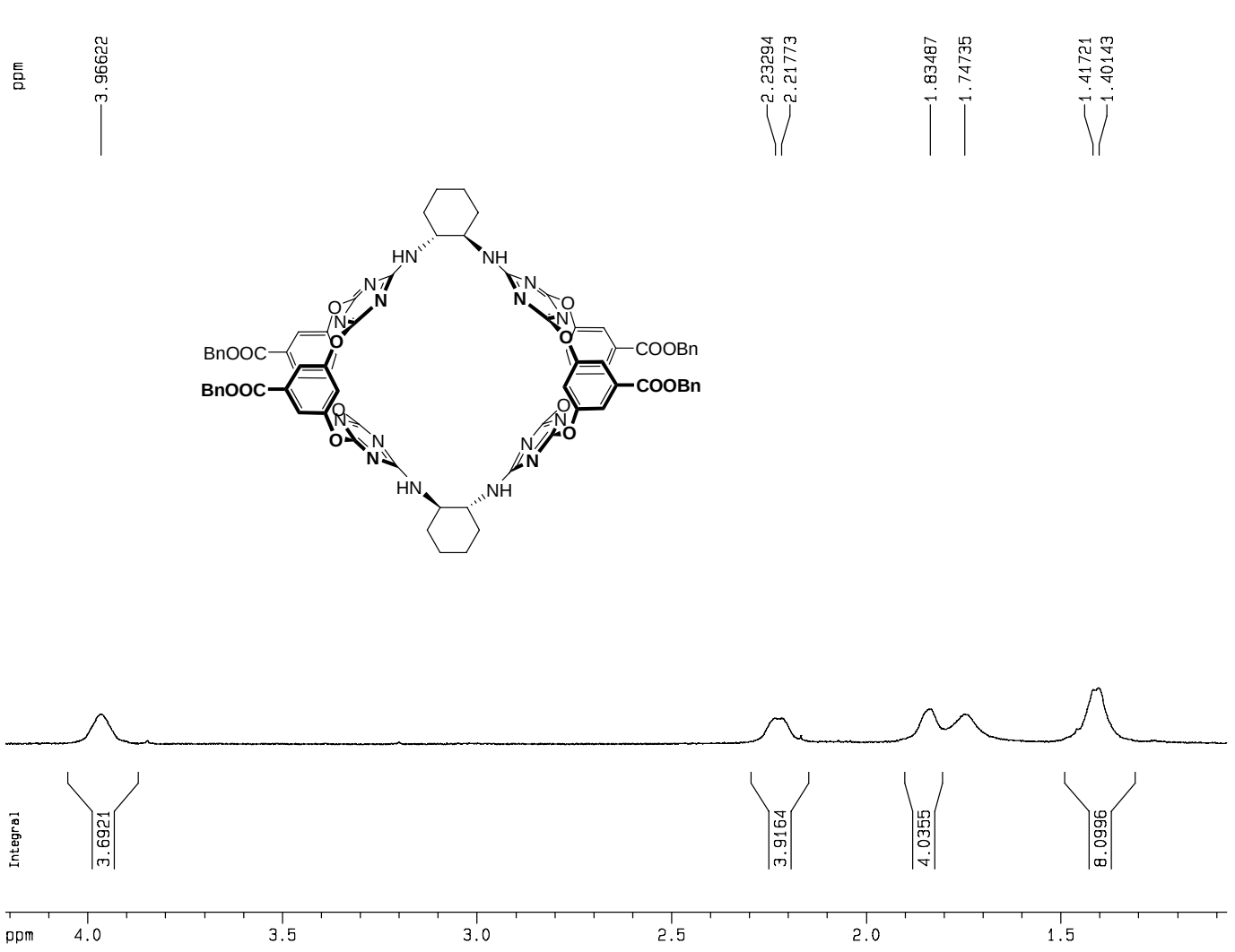
F2 - Acquisition Parameters
Date_                 20060904
Time                     23.14
INSTRUM                  av300
PROBHD   5 mm DUL 13C-1
PULPROG                  zg30
TD                       65536
SOLVENT                  CDCl3
NS                          92
DS                           2
SWH                 6172.839 Hz
FIDRES              0.094190 Hz
AQ                  5.3084660 sec
RG                       574.7
DW                    81.000 usec
DE                     6.00 usec
TE                     302.6 K
D1                 2.00000000 sec

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

F2 - Processing parameters
SI                       32768
SF                300.1300069 MHz
WDW                         EM
SSB                          0
LB                     0.30 Hz
GB                           0
PC                        1.00

1D NMR plot parameters
CX                     22.00 cm
CY                     25.00 cm
F1P                    7.729 ppm
F1                  2319.67 Hz
F2P                    5.097 ppm
F2                  1529.79 Hz
PPMCM               0.11963 ppm/cm
HZCM               35.90372 Hz/cm
```

7-^{1}H NMR (expanded)




```
Current Data Parameters
NAME            hou7-4-H
EXPNO                 10
PROCNO                 1

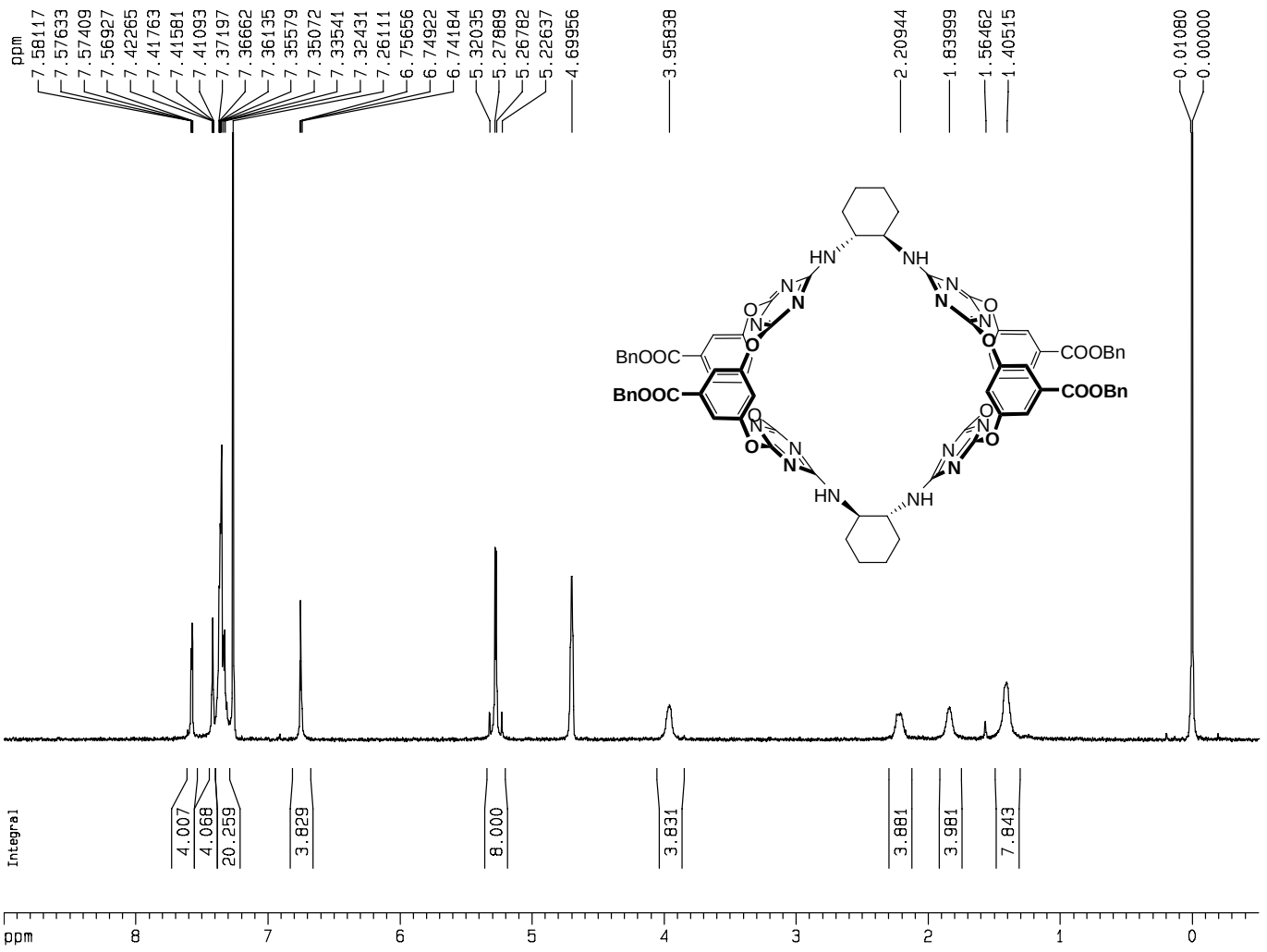
F2 - Acquisition Parameters
Date_           20060904
Time               23.14
INSTRUM            av300
PROBHD   5 mm DUL 13C-1
PULPROG             zg30
TD                 65536
SOLVENT            CDCl3
NS                    92
DS                     2
SWH             6172.839 Hz
FIDRES          0.094190 Hz
AQ              5.3084660 sec
RG                 574.7
DW                81.000 usec
DE                 6.00 usec
TE                302.6 K
D1             2.00000000 sec

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

F2 - Processing parameters
SI                 32768
SF           300.1300069 MHz
WDW                   EM
SSB                    0
LB                  0.30 Hz
GB                     0
PC                  1.00

1D NMR plot parameters
CX                 22.00 cm
CY                 25.00 cm
F1P                4.211 ppm
F1               1263.98 Hz
F2P                1.074 ppm
F2                322.31 Hz
PPMCM           0.14261 ppm/cm
HZCM            42.80286 Hz/cm
```

7-^{1}H NMR-D_2O

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Current Data Parameters
NAME        hou7-4-h-D2O
EXPNO                 10
PROCNO                 1

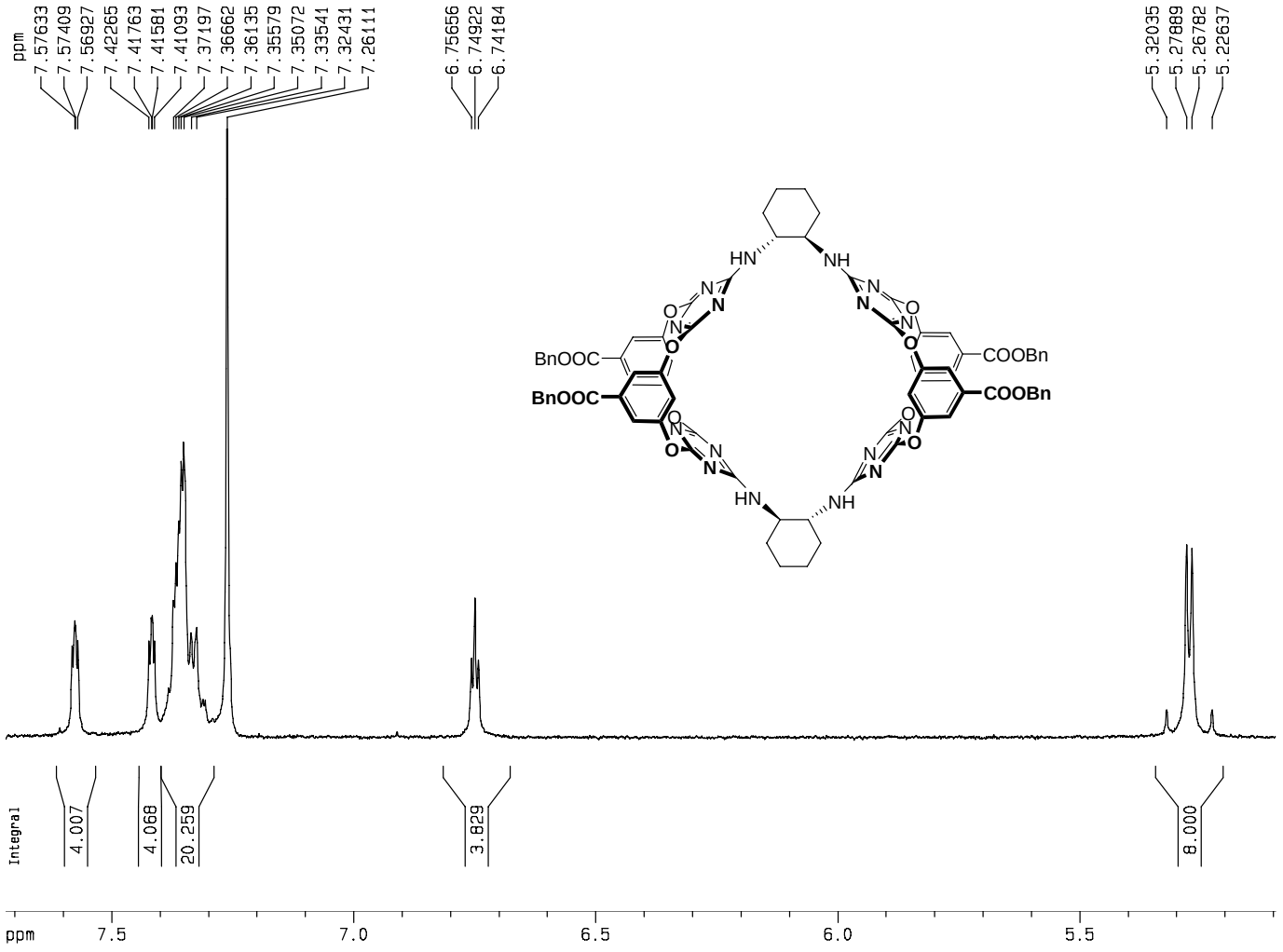
F2 - Acquisition Parameters
Date_           20060906
Time                8.49
INSTRUM            av300
PROBHD   5 mm DUL 13C-1
PULPROG             zg30
TD                 65536
SOLVENT            CDCl3
NS                    12
DS                     2
SWH             6172.839 Hz
FIDRES          0.094190 Hz
AQ              5.3084660 sec
RG                 645.1
DW                81.000 usec
DE                 6.00 usec
TE                302.5 K
D1             2.00000000 sec

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

F2 - Processing parameters
SI                 32768
SF           300.1300060 MHz
WDW                   EM
SSB                    0
LB                  0.30 Hz
GB                     0
PC                  1.00

1D NMR plot parameters
CX                 22.00 cm
CY                 25.00 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
```

7-^{1}H NMR-D_2O (expanded)




```
Current Data Parameters
NAME        hou7-4-h-D20
EXPNO                 10
PROCNO                 1

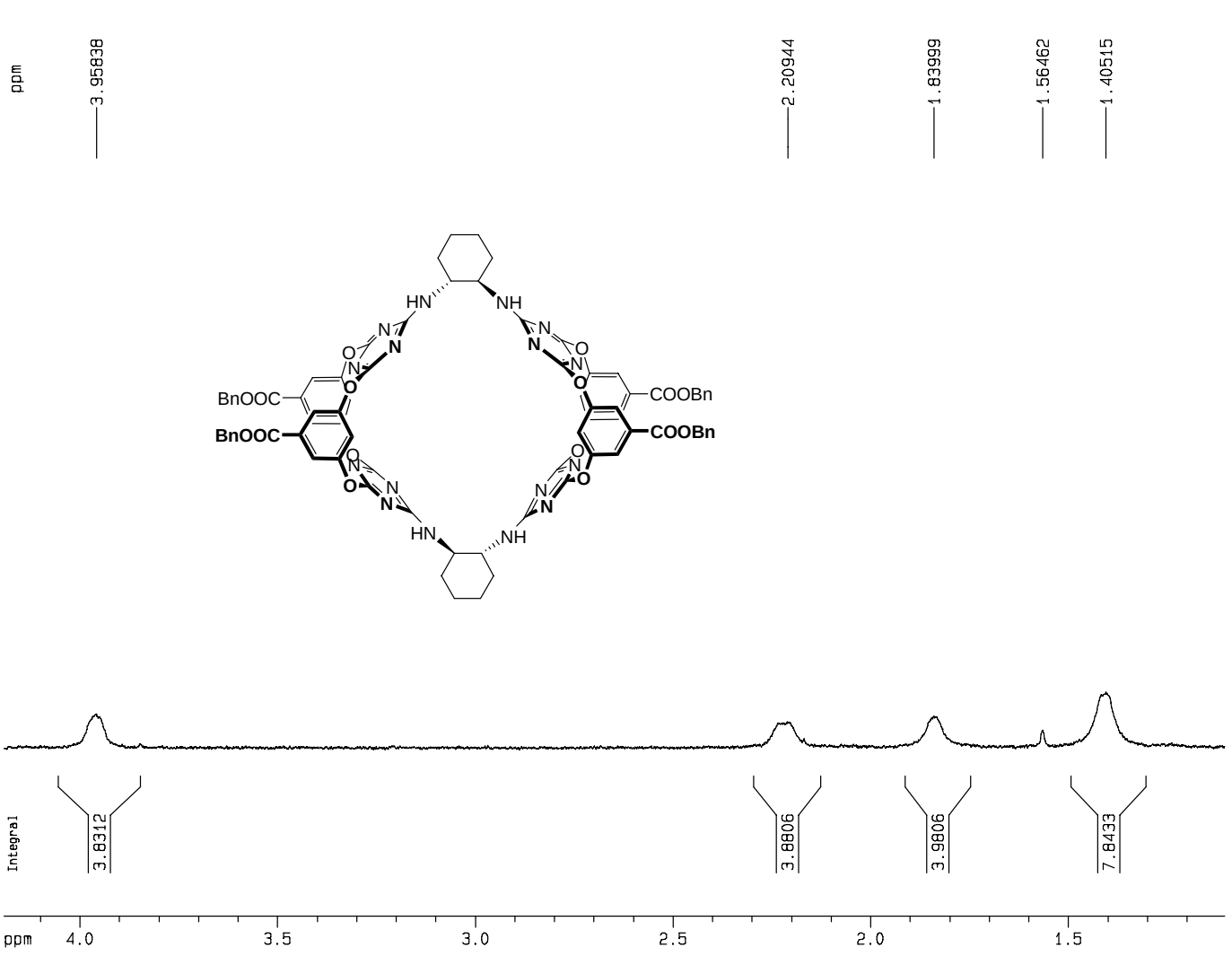
F2 - Acquisition Parameters
Date_           20060906
Time                8.49
INSTRUM           av300
PROBHD   5 mm DUL 13C-1
PULPROG             zg30
TD                 65536
SOLVENT            CDCl3
NS                    12
DS                     2
SWH            6172.839 Hz
FIDRES         0.094190 Hz
AQ             5.3084660 sec
RG                645.1
DW               81.000 usec
DE                6.00 usec
TE                302.5 K
D1            2.00000000 sec

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

F2 - Processing parameters
SI                 32768
SF           300.1300060 MHz
WDW                   EM
SSB                    0
LB                  0.30 Hz
GB                     0
PC                  1.00

1D NMR plot parameters
CX                 22.00 cm
CY                 25.00 cm
F1P                7.719 ppm
F1                2316.79 Hz
F2P                5.095 ppm
F2                1529.25 Hz
PPMCM          0.11927 ppm/cm
HZCM           35.79734 Hz/cm
```

7-^{1}H NMR-D_2O (expanded)




```
Current Data Parameters
NAME        hou7-4-h-D2O
EXPNO                 10
PROCNO                 1

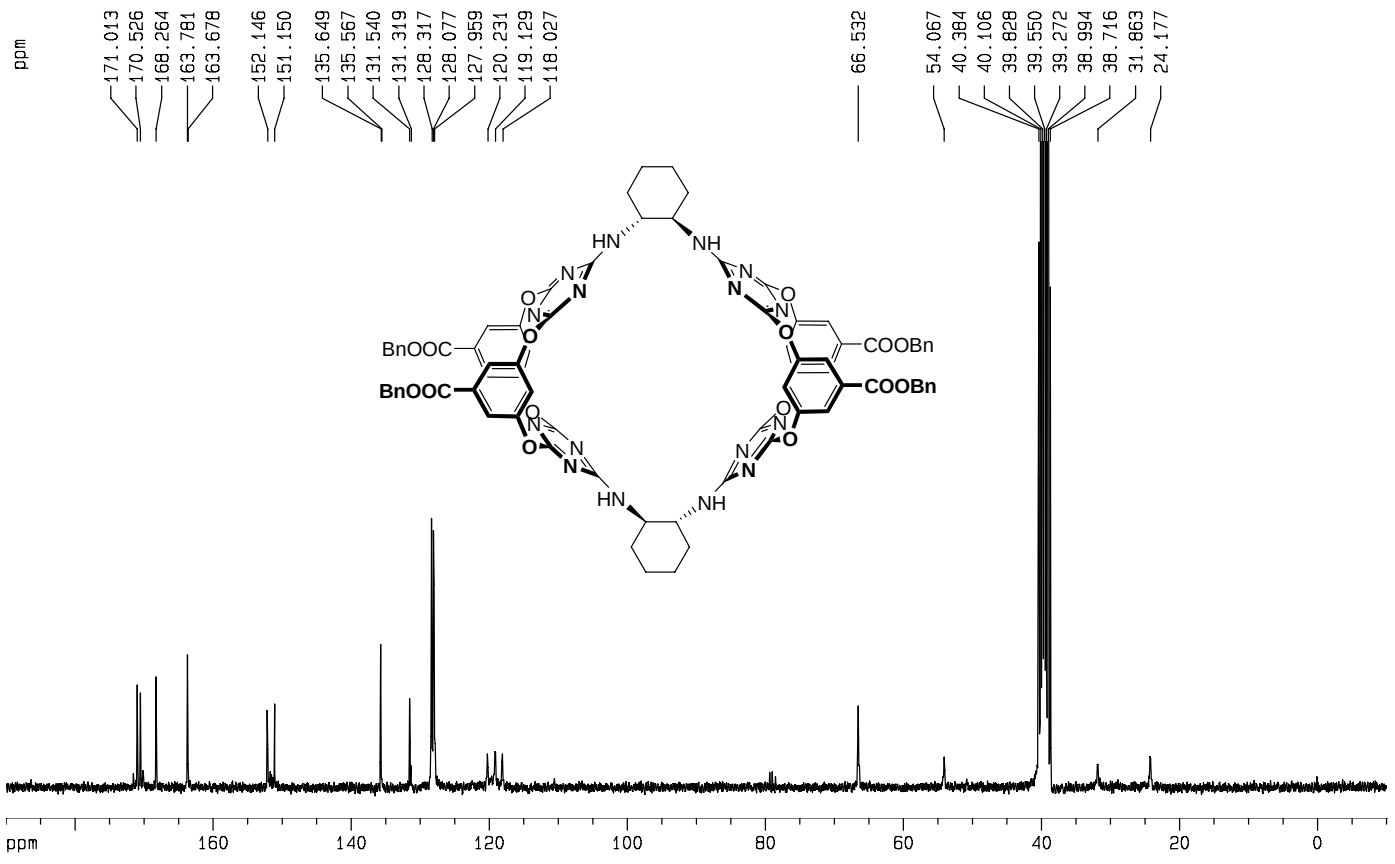
F2 - Acquisition Parameters
Date_           20060906
Time                8.49
INSTRUM            av300
PROBHD   5 mm DUL 13C-1
PULPROG             zg30
TD                 65536
SOLVENT            CDCl3
NS                    12
DS                     2
SWH             6172.839 Hz
FIDRES          0.094190 Hz
AQ              5.3084660 sec
RG                 645.1
DW                81.000 usec
DE                 6.00 usec
TE                302.5 K
D1             2.00000000 sec

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

F2 - Processing parameters
SI                 32768
SF           300.1300060 MHz
WDW                   EM
SSB                    0
LB                  0.30 Hz
GB                     0
PC                  1.00

1D NMR plot parameters
CX                 22.00 cm
CY                 25.00 cm
F1P                4.192 ppm
F1               1258.14 Hz
F2P                1.104 ppm
F2                331.35 Hz
PPMCM           0.14036 ppm/cm
HZCM            42.12704 Hz/cm
```

7-^{13}C NMR

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Current Data Parameters
NAME            hou7-4-C
EXPNO                 10
PROCNO                 1

F2 - Acquisition Parameters
Date_           20061120
Time               22.30
INSTRUM            av300
PROBHD   5 mm DUL 13C-1
PULPROG          zgpg30
TD                65536
SOLVENT             DMSO
NS                  9270
DS                     4
SWH           17985.611 Hz
FIDRES         0.274439 Hz
AQ             1.8219508 sec
RG                2896.3
DW                27.800 usec
DE                 6.00 usec
TE                299.8 K
D1            2.00000000 sec
d11           0.03000000 sec
d12           0.00002000 sec

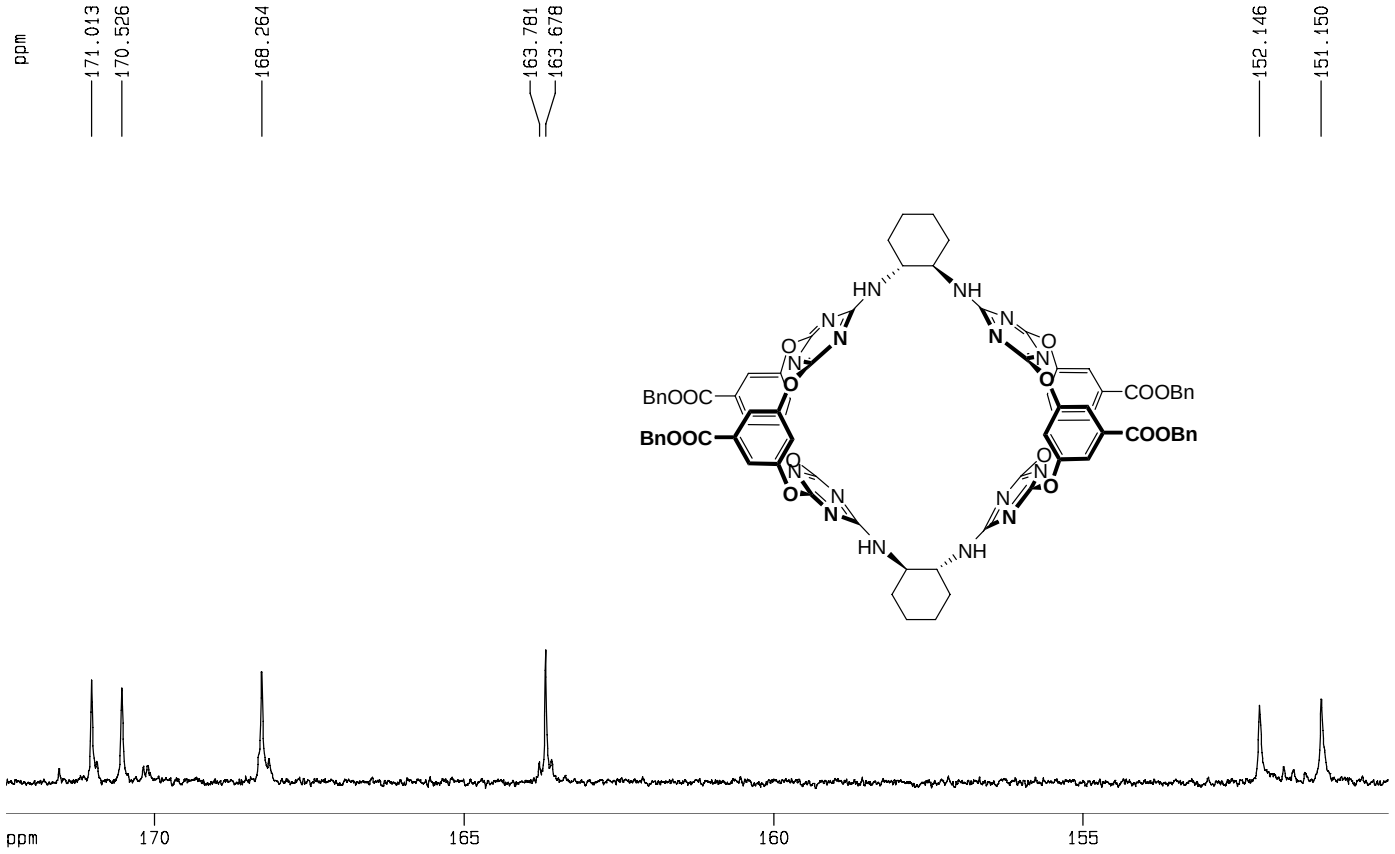
======== CHANNEL f1 ========
NUC1                13C
P1                 9.40 usec
PL1               -1.00 dB
SFO1          75.4752953 MHz

======== 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            75.4677867 MHz
WDW                   EM
SSB                    0
LB                  1.00 Hz
GB                     0
PC                  1.40

1D NMR plot parameters
CX                 22.00 cm
CY                 60.00 cm
F1P              190.000 ppm
F1              14338.88 Hz
F2P              -10.000 ppm
F2               -754.68 Hz
PPMCM            9.09091 ppm/cm
HZCM           686.07080 Hz/cm
```

7-^{13}C NMR (expanded)

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Current Data Parameters
NAME            hou7-4-C
EXPNO                 10
PROCNO                 1

F2 - Acquisition Parameters
Date_           20061120
Time               22.30
INSTRUM            av300
PROBHD   5 mm DUL 13C-1
PULPROG          zgpg30
TD                65536
SOLVENT            DMSO
NS                 9270
DS                    4
SWH           17985.611 Hz
FIDRES         0.274439 Hz
AQ             1.8219508 sec
RG                2896.3
DW                27.800 usec
DE                 6.00 usec
TE                299.8 K
D1            2.00000000 sec
d11           0.03000000 sec
d12           0.00002000 sec

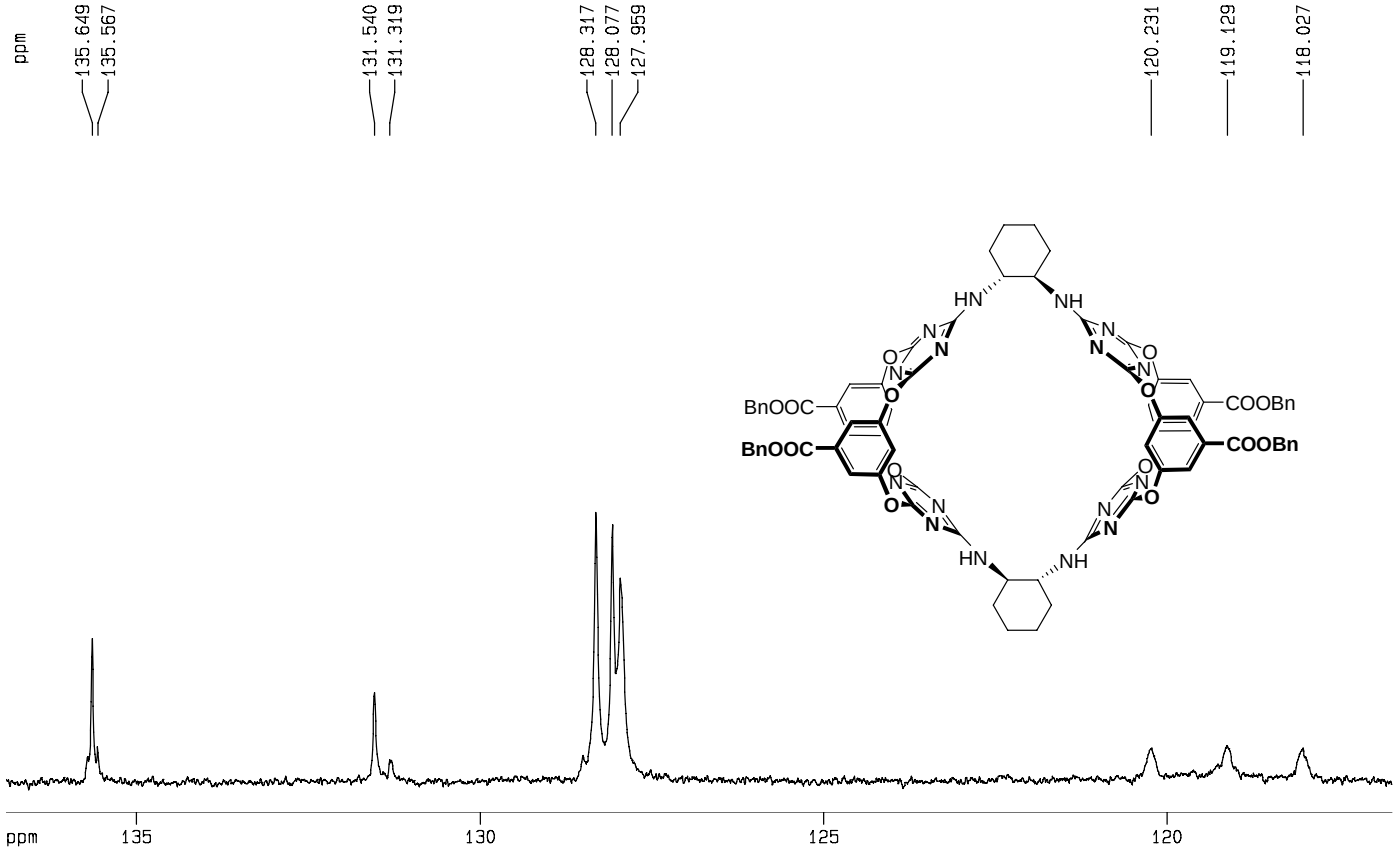
======== CHANNEL f1 ========
NUC1                13C
P1                9.40 usec
PL1               -1.00 dB
SFO1         75.4752953 MHz

======== 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            75.4677867 MHz
WDW                   EM
SSB                    0
LB                 1.00 Hz
GB                     0
PC                 1.40

1D NMR plot parameters
CX                22.00 cm
CY                60.00 cm
F1P              172.392 ppm
F1              13010.07 Hz
F2P              150.068 ppm
F2              11325.29 Hz
PPMCM            1.01475 ppm/cm
HZCM            76.58092 Hz/cm
```

7-^{13}C NMR (expanded)

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Current Data Parameters
NAME                  hou7-4-C
EXPNO                       10
PROCNO                       1

F2 - Acquisition Parameters
Date_               20061120
Time                    22.30
INSTRUM                 av300
PROBHD   5 mm DUL 13C-1
PULPROG                 zgpg30
TD                       65536
SOLVENT                   DMSO
NS                        9270
DS                           4
SWH                 17985.611 Hz
FIDRES                0.274439 Hz
AQ                    1.8219508 sec
RG                       2896.3
DW                      27.800 usec
DE                       6.00 usec
TE                      299.8 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                  75.4677867 MHz
WDW                         EM
SSB                          0
LB                       1.00 Hz
GB                           0
PC                       1.40

1D NMR plot parameters
CX                      22.00 cm
CY                      60.00 cm
F1P                    136.899 ppm
F1                   10331.46 Hz
F2P                    116.729 ppm
F2                    8809.28 Hz
PPMCM                 0.91682 ppm/cm
HZCM                 69.19041 Hz/cm
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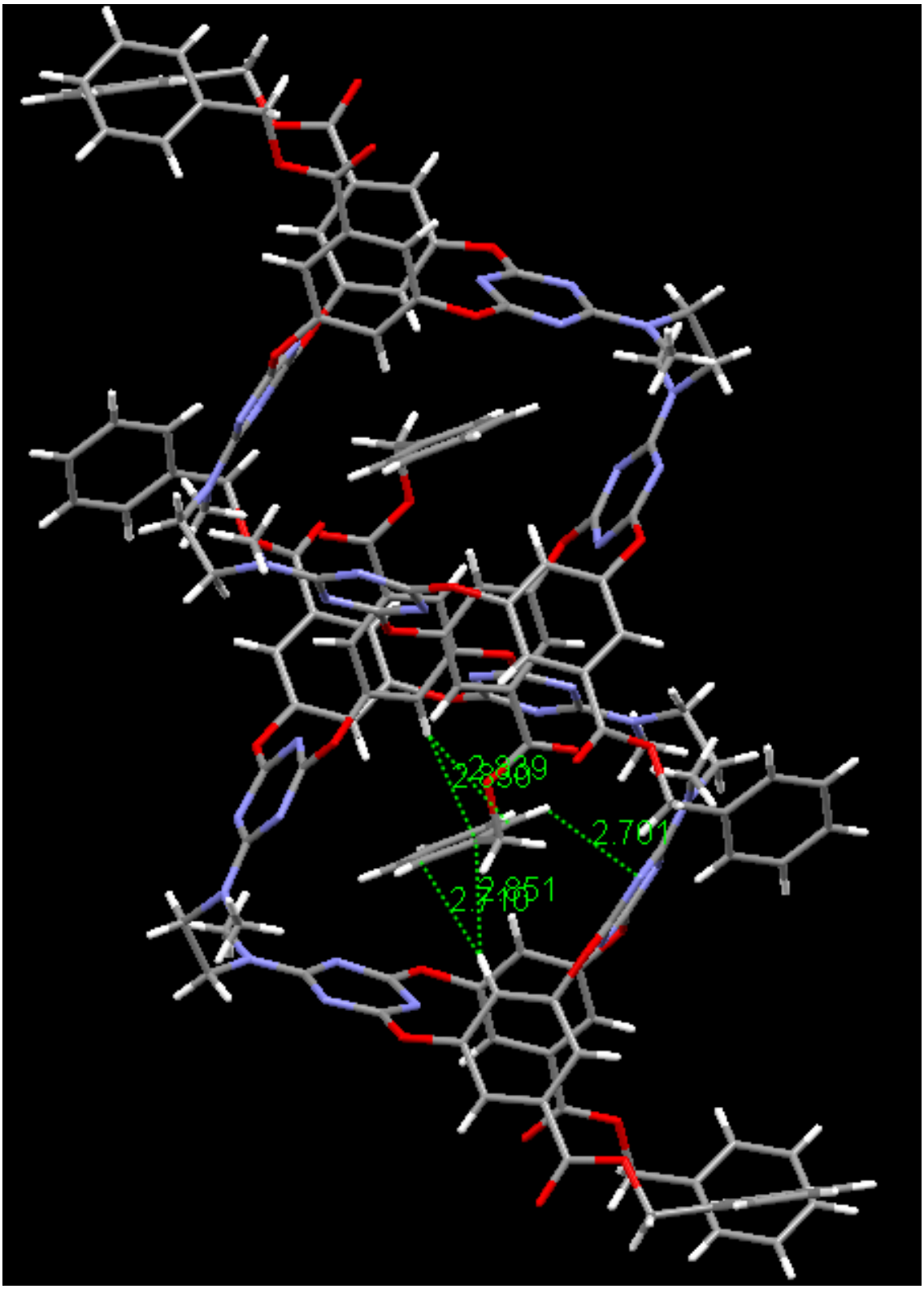
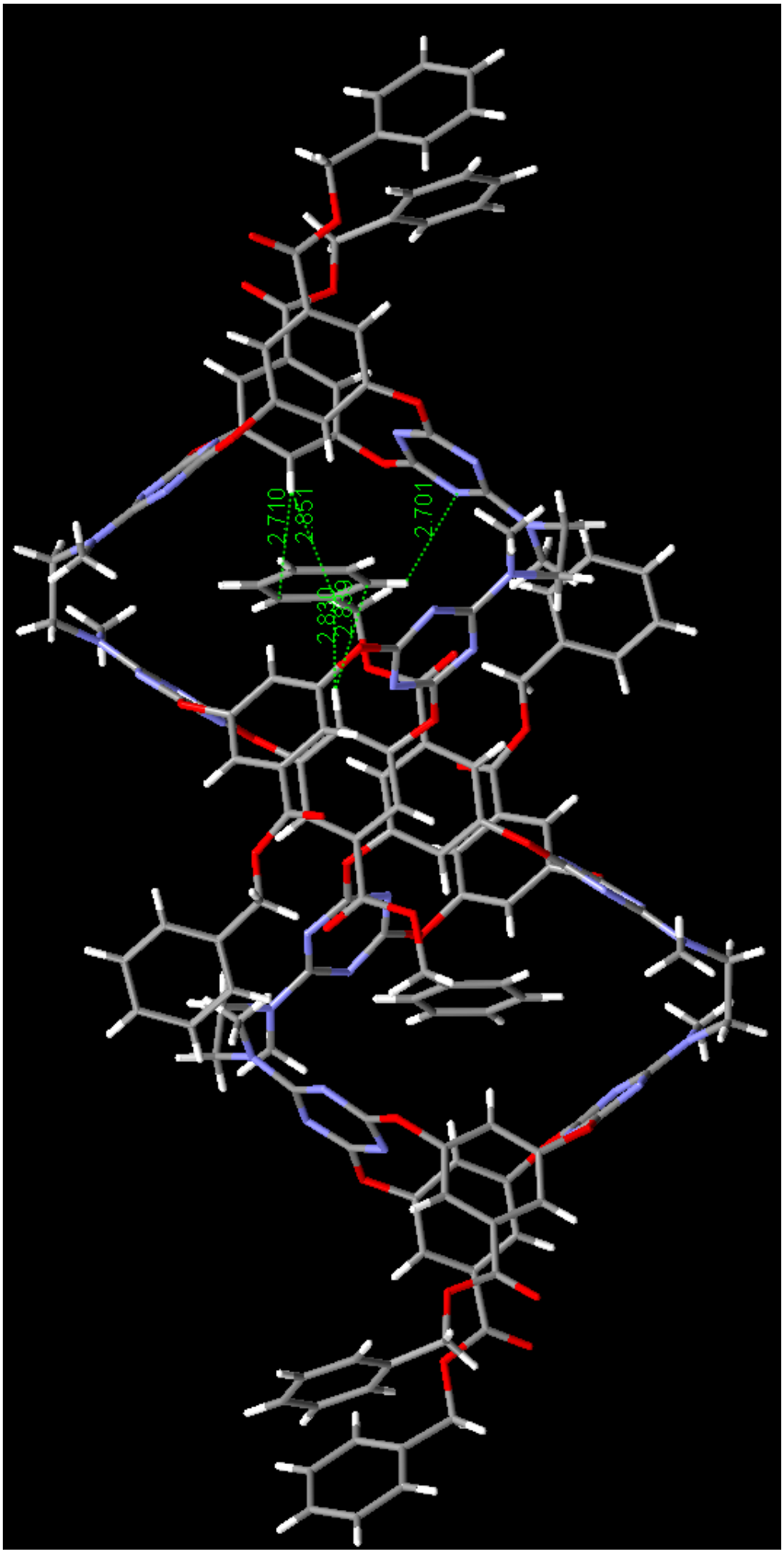

2.701


2.710
2.851
2.701