

**Nanometer-Scale Water-Soluble Macrocycles from Nanometer-Sized
Amino Acids**

*Chris M. Gothard and James S. Nowick**

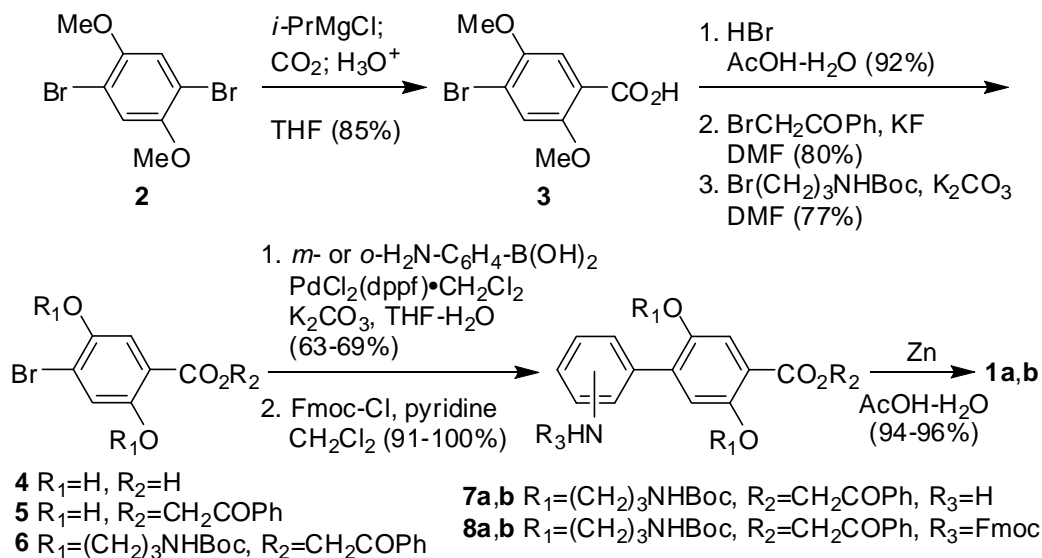
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Irvine, California 92697-2025

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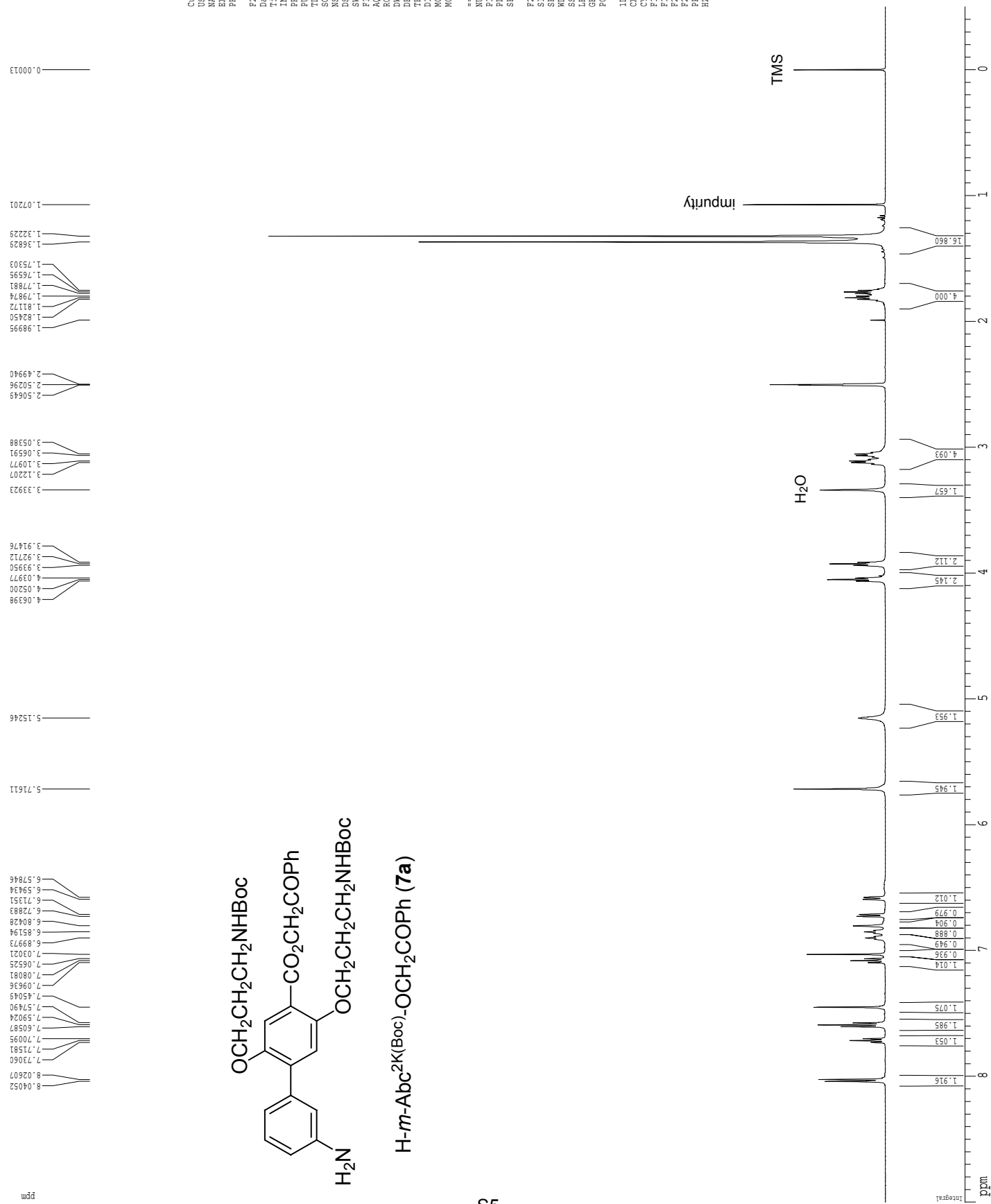
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Scheme S1. Synthesis of Fmoc-*m*-Abc^{2K(Boc)}-OH (**1a**) and Fmoc-*o*-Abc^{2K(Boc)}-OH (**1b**)

* Diether **6** was prepared in a previous publication.¹

¹ Gothard, C. M.; Rao, N. A.; Nowick, J. S. *J. Am. Chem. Soc.* **2007**, *129*, 7272-7273.

^1H NMR (500 MHz, 298 K, CD_3SOCD_3) spectrum of **7a**

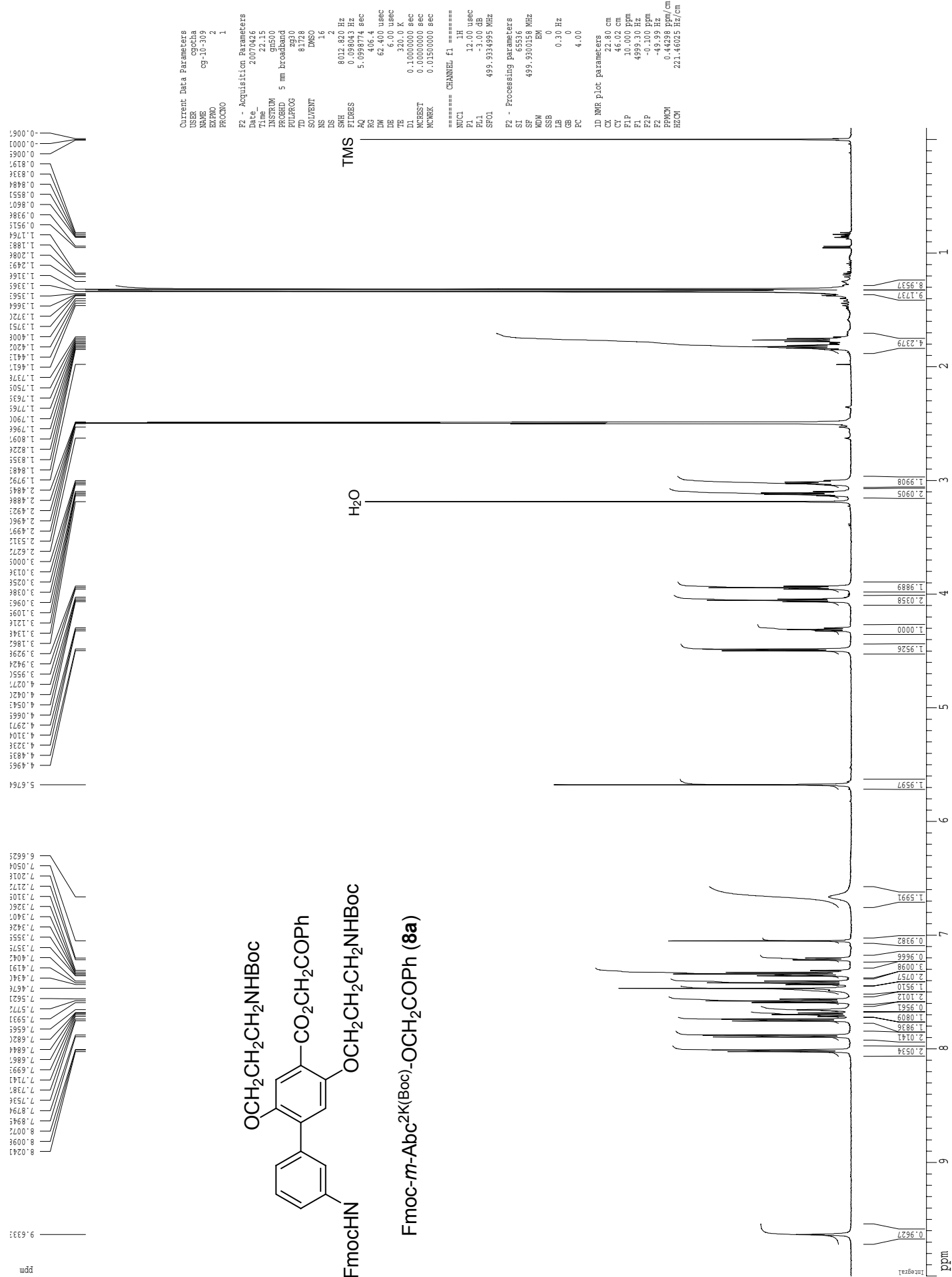
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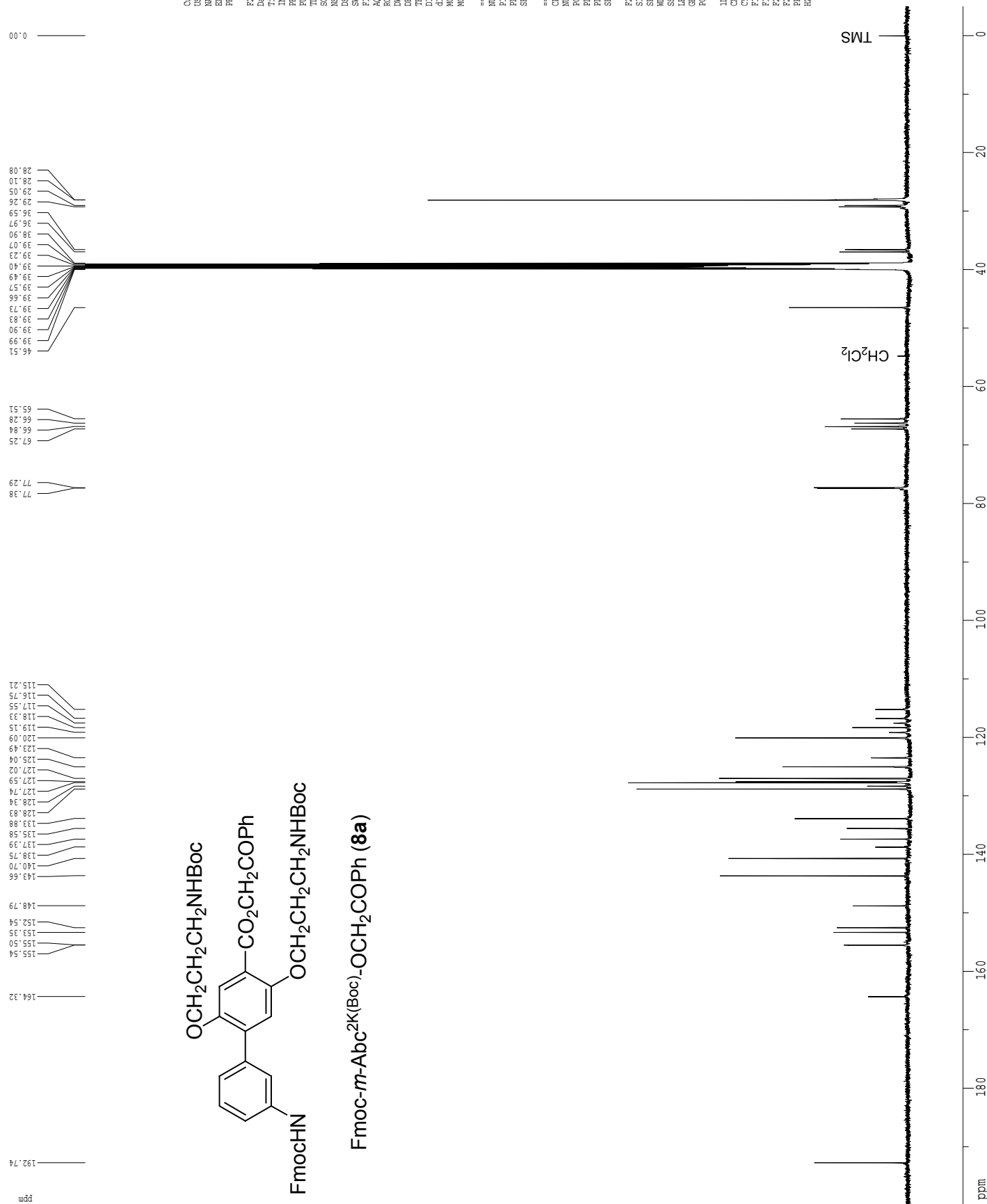
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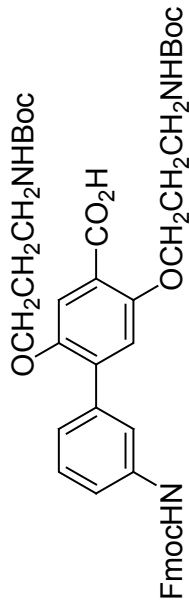
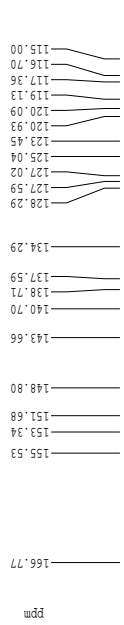
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^1H NMR (500 MHz, 320 K, CD_3SOCD_3) spectrum of **8a**

^{13}C NMR (125 MHz, 298 K, CD_3SOCD_3) spectrum of **8a**

¹³C NMR (125 MHz, 298 K, CD₃SOCD₃) spectrum of **1a**



Fmoc-*m*-Abc²K(Boc)-OH (**1a**)

Supporting Information

C. M. Gothard and J. S. Nowick

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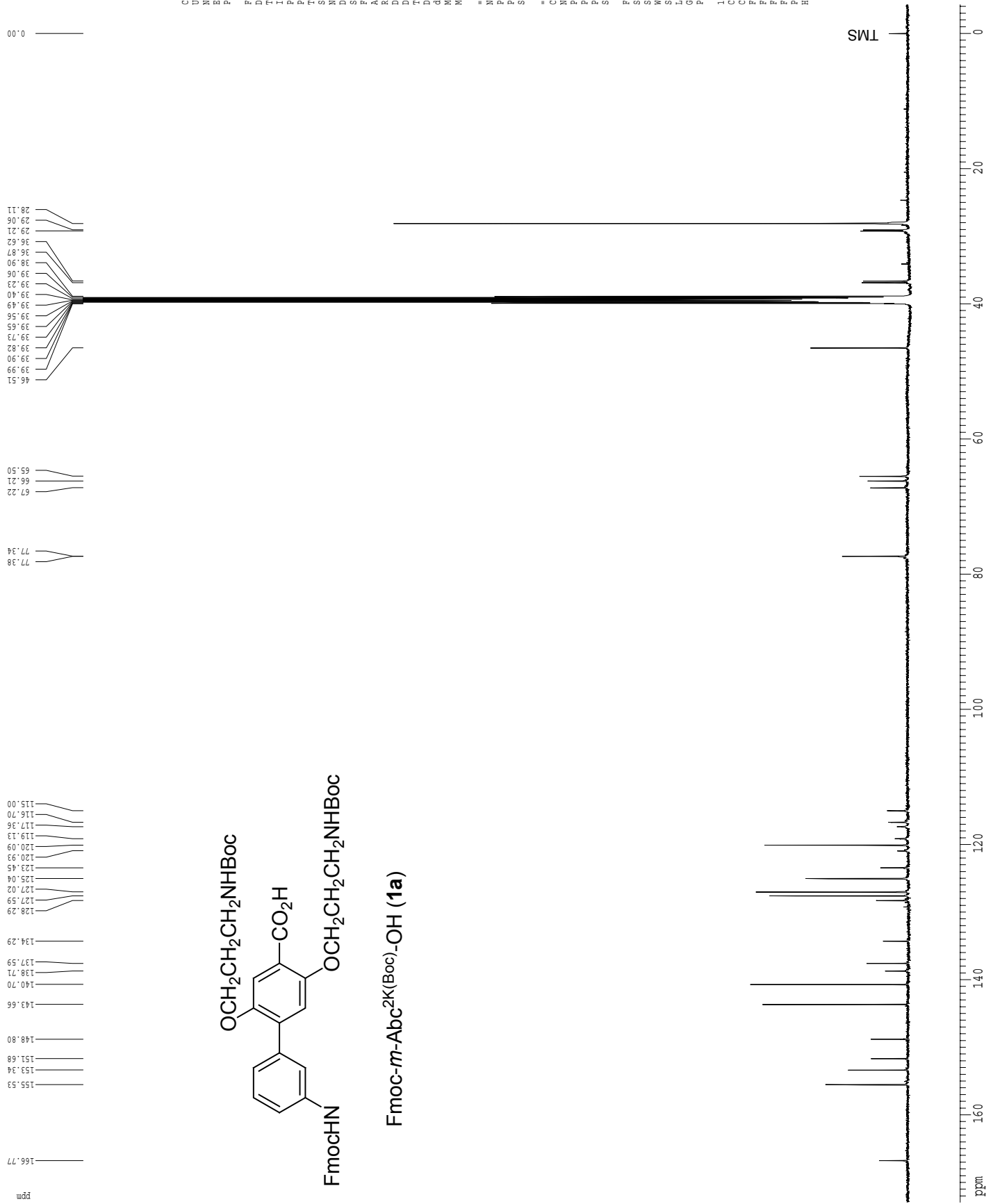
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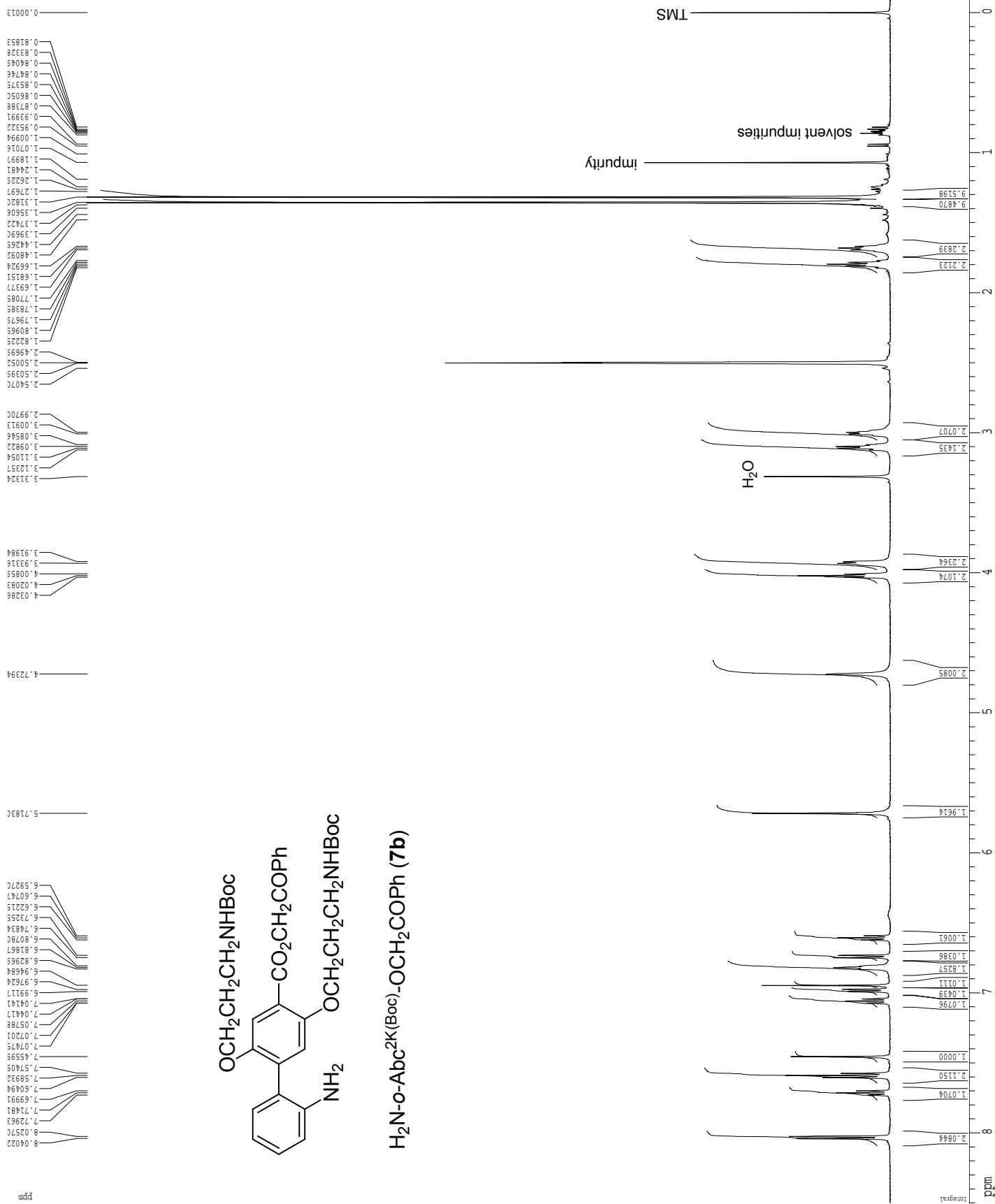
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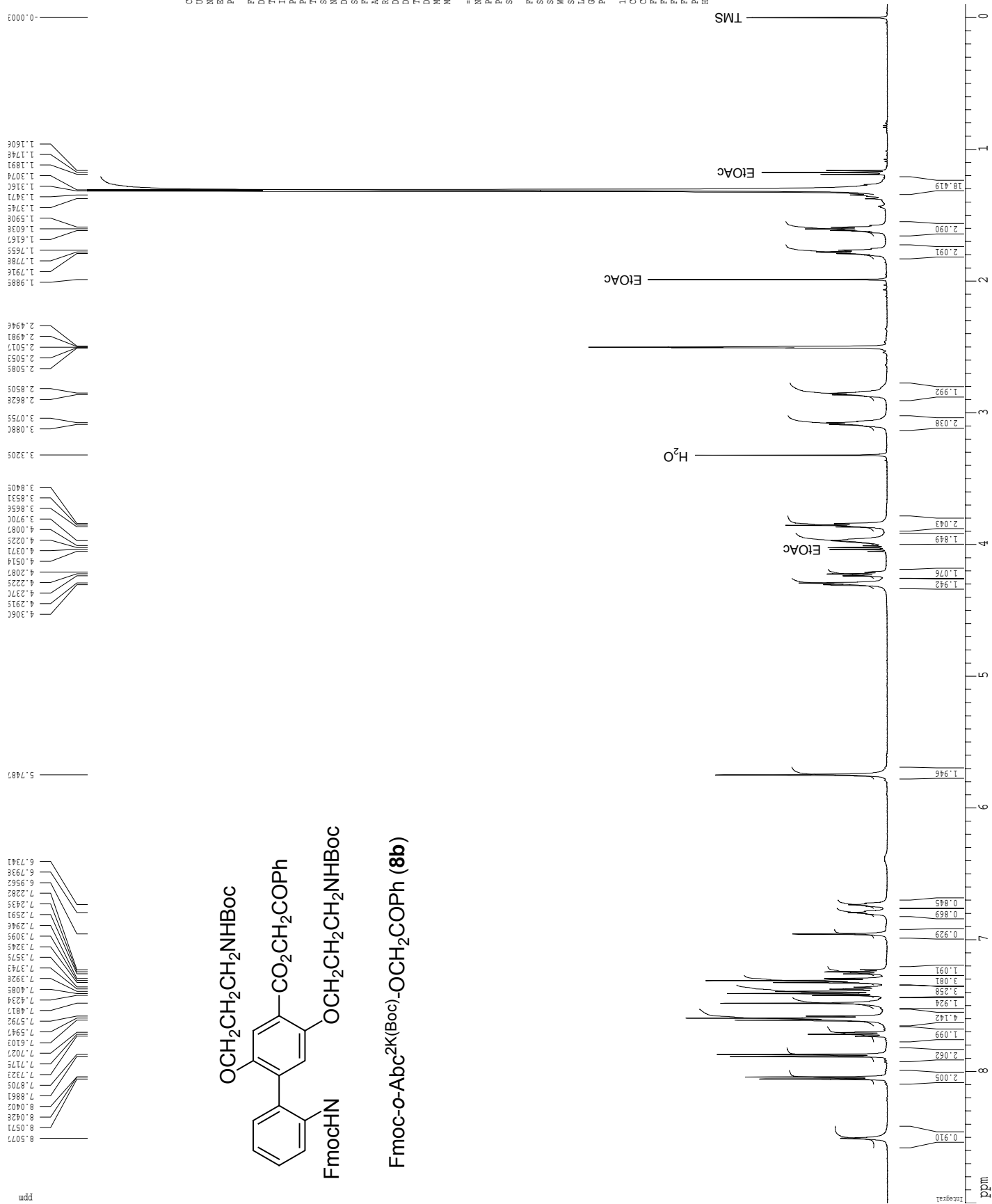
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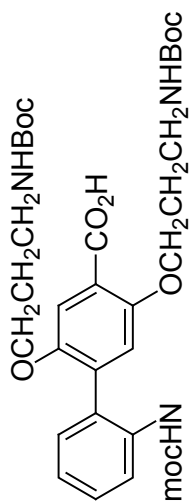
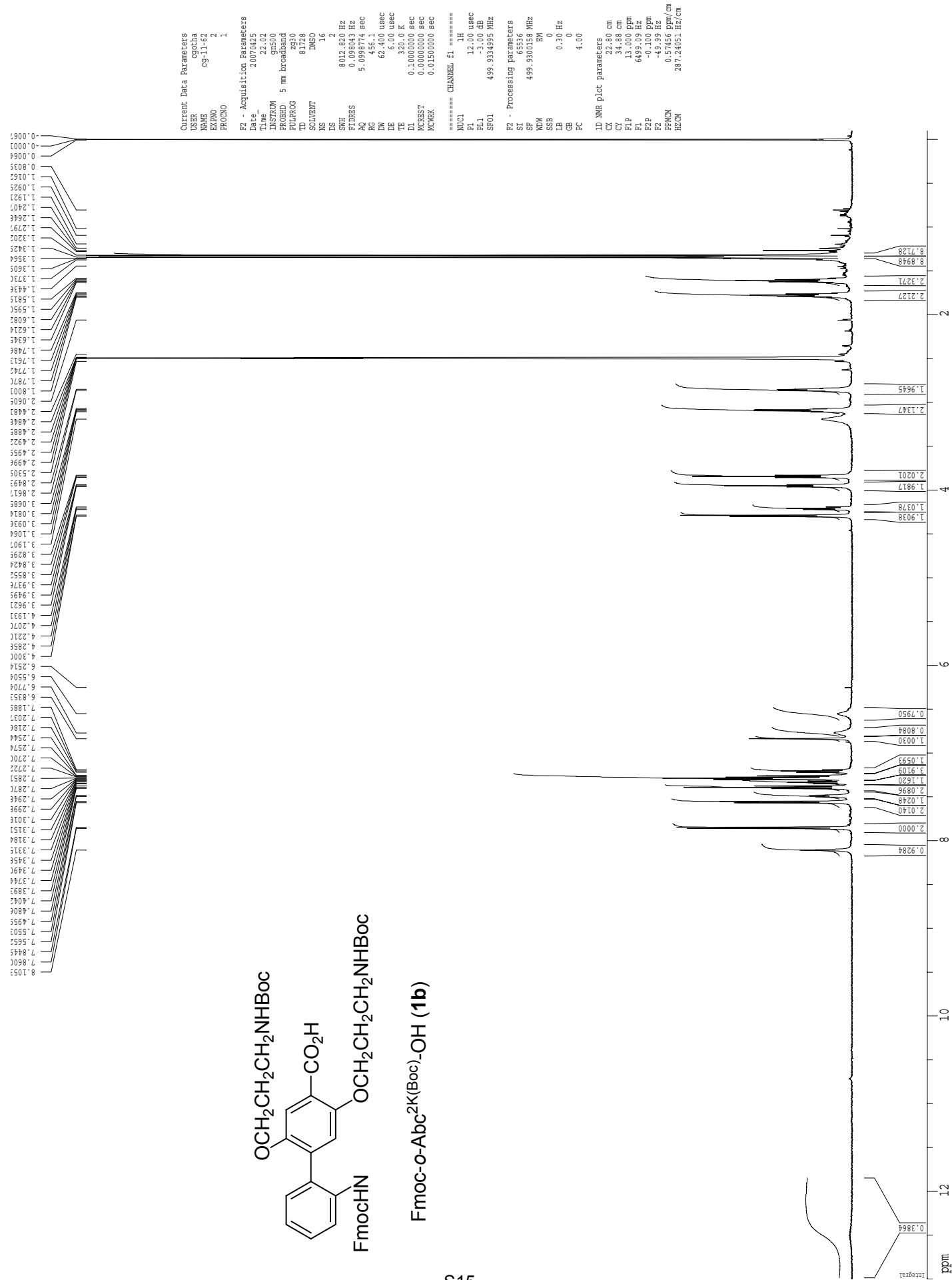
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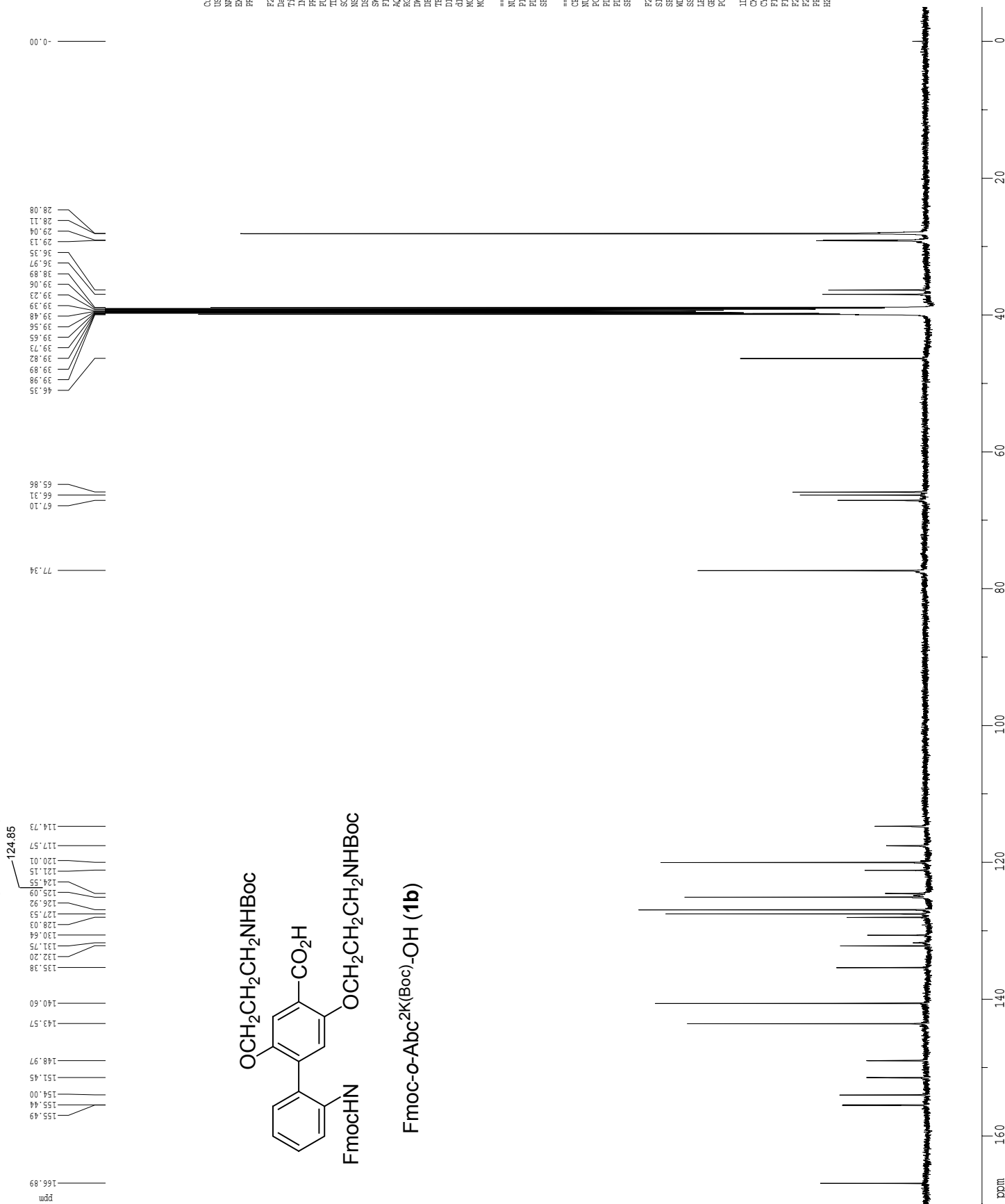
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^1H NMR (500 MHz, 298 K, CD_3SOCD_3) spectrum of **7b**

^1H NMR (500 MHz, 298 K, CD_3SOCD_3) spectrum of **8b**

¹H NMR (500 MHz, 320 K, CD₃SOCD₃) spectrum of **1b****Fmoc-o-Abc²K(Boc)-OH (1b)**

¹³C NMR (125 MHz, 298 K, CD₃SOCD₃) spectrum of **1b**

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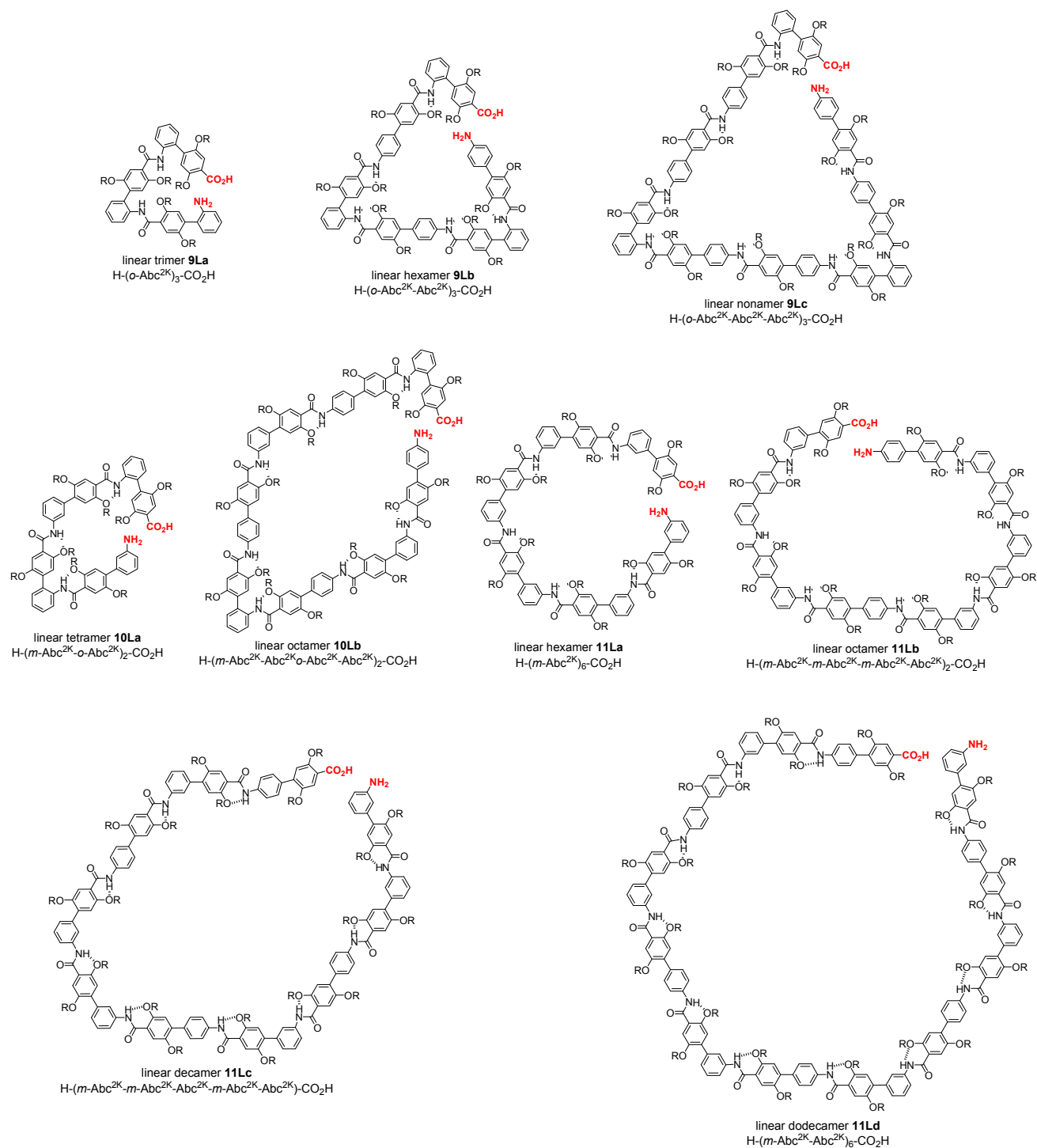
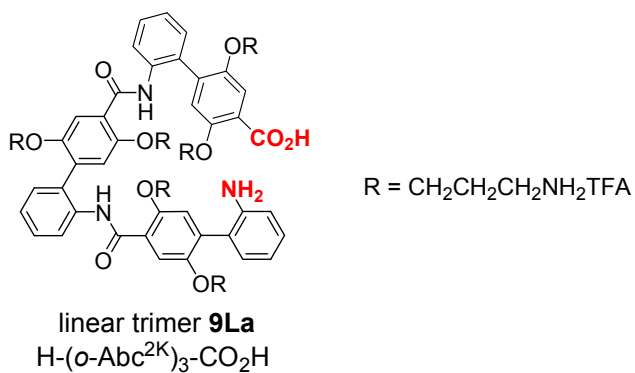
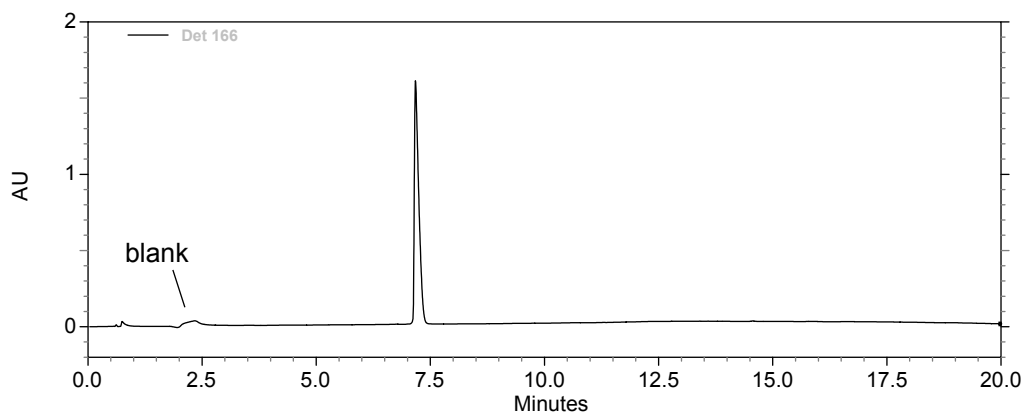


Figure S1. Linear Abc^{2K} oligomers (9La-c, 10La-b, and 11La-d) showing the amino and carboxylic acid groups (red) used in macrocyclization. (R = CH₂CH₂CH₂NHBoc)

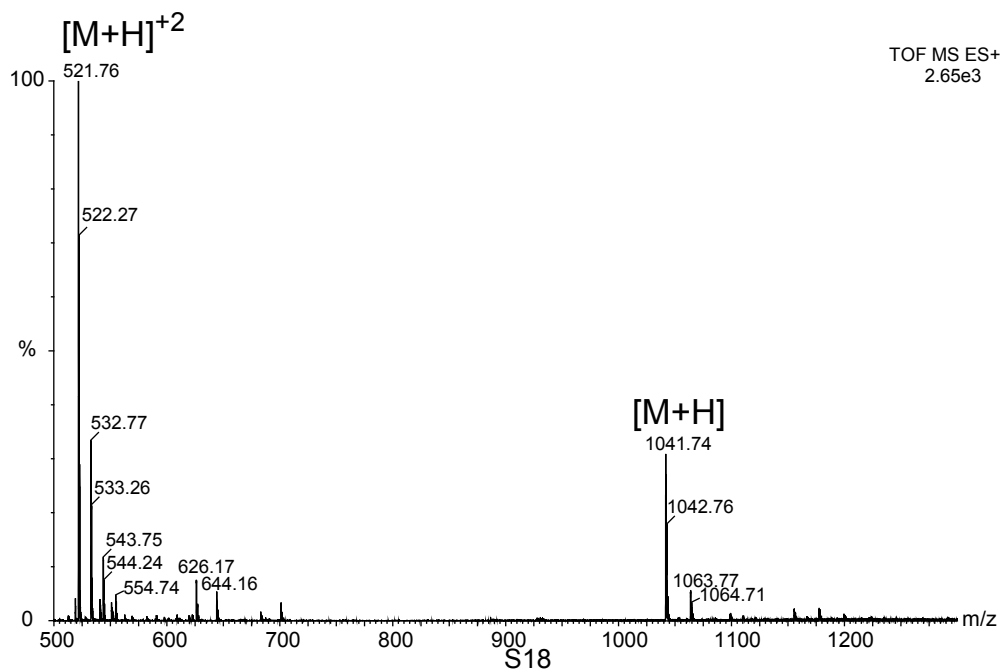
trimer H-(*o*-Abc^{2K})₃-CO₂H (**9La**)
Analytical RP-HPLC chromatograph and mass spectrum (ESI-MS)



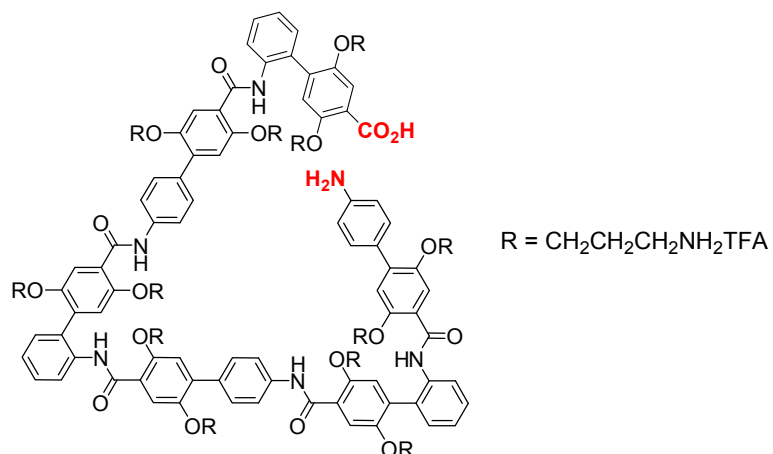
(a) Analytical RP-HPLC (0-30% acetonitrile with 0.1% TFA over 10 min, $\lambda = 214$)



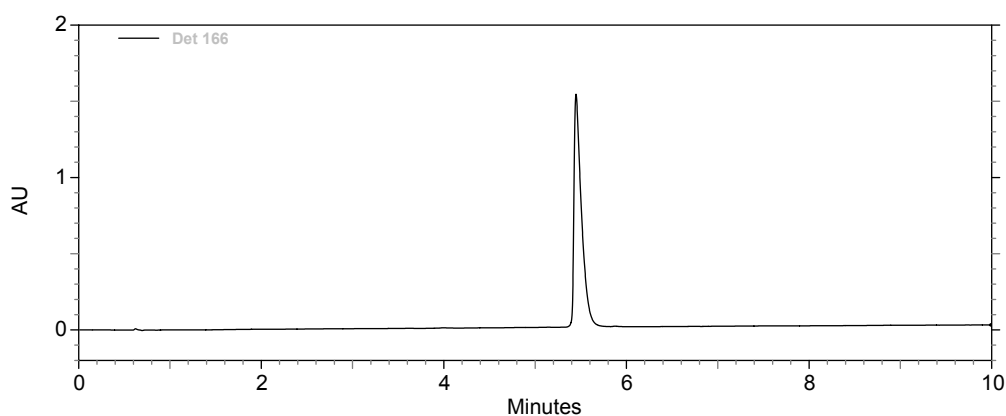
(b) ESI Mass spectrum. (Calcd exact mass for C₅₇H₇₁N₉O₁₀ [M] = 1041.53)



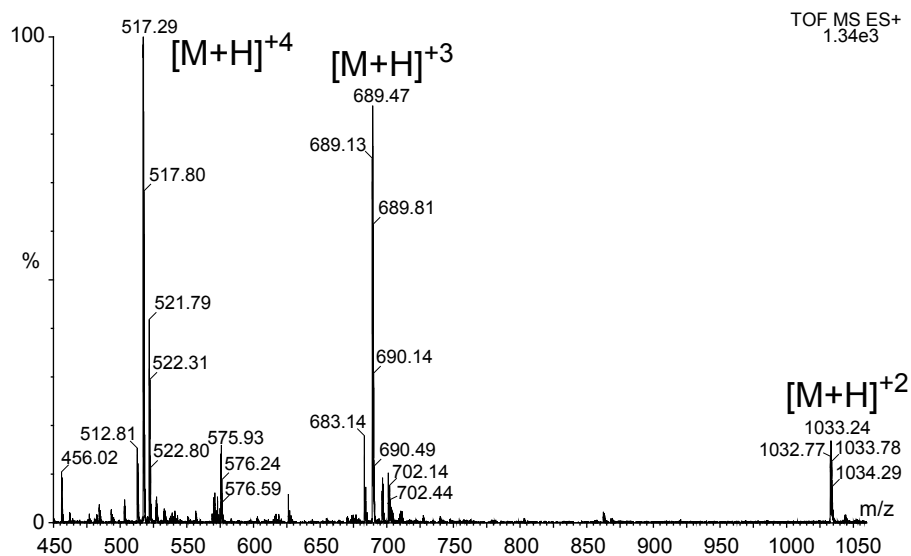
linear hexamer: H-(*p*-Abc^{2K}-*o*-Abc^{2K})₃-CO₂H (**9Lb**)
Analytical RP-HPLC chromatograph and mass spectrum (ESI-MS)



(a) Analytical RP-HPLC (5-50% acetonitrile with 0.1% TFA over 10 min, $\lambda = 214$)



(b) ESI Mass spectrum. (Calcd exact mass for C₁₁₄H₁₄₀N₁₈O₁₉ [M] = 2065.05)



^1H NMR (500 MHz, 298 K, D_2O) spectrum of linear hexamer 9Lb

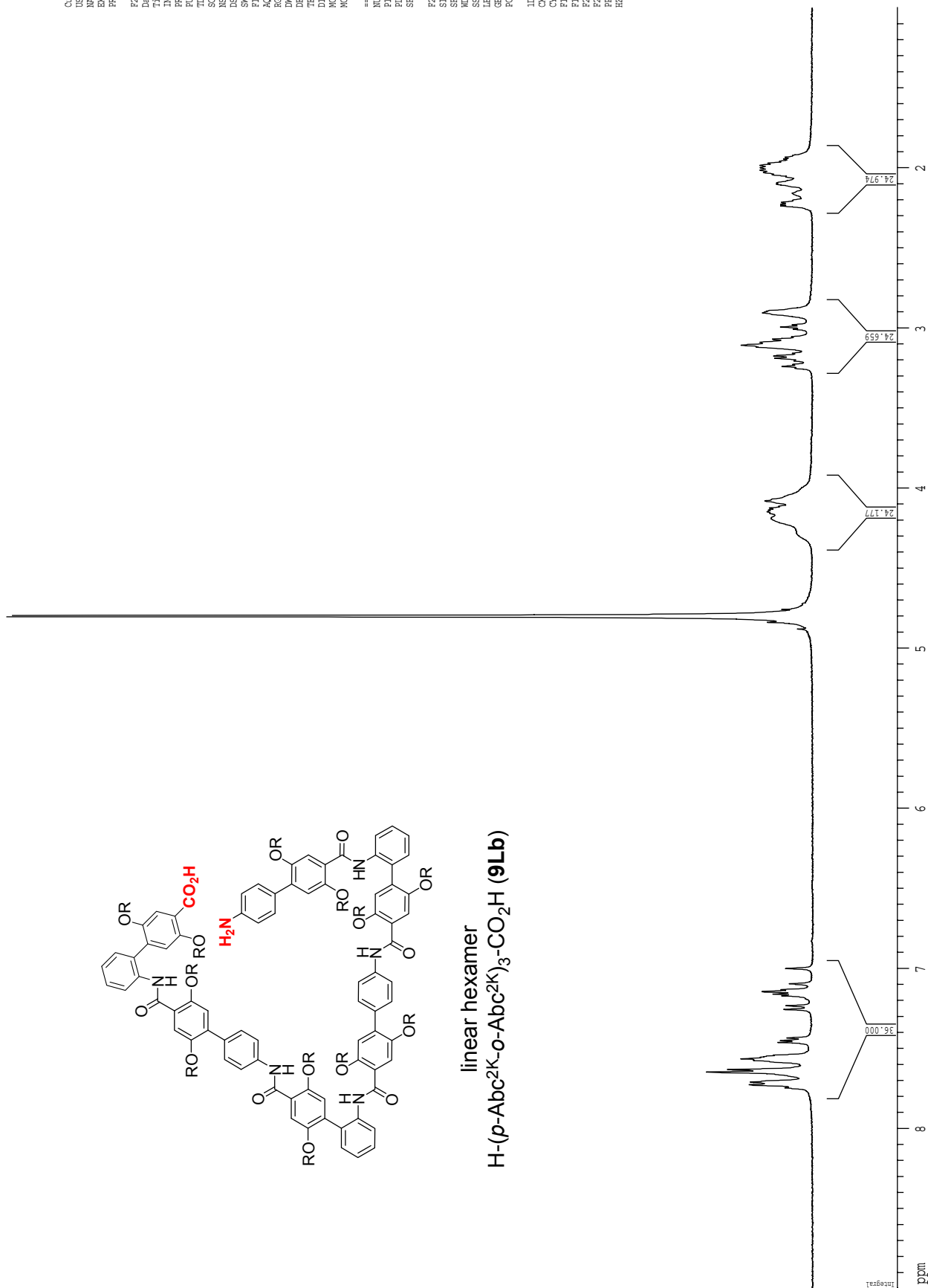
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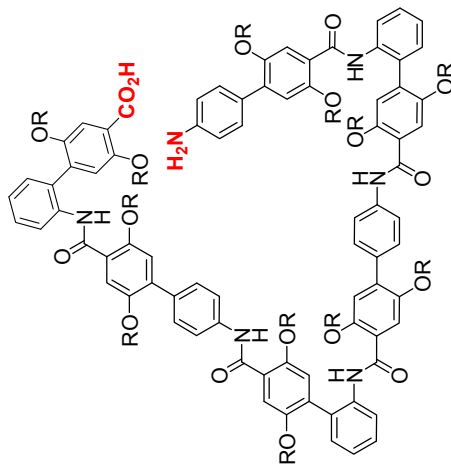
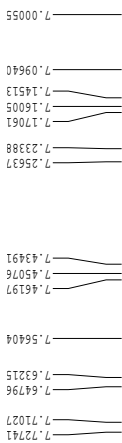
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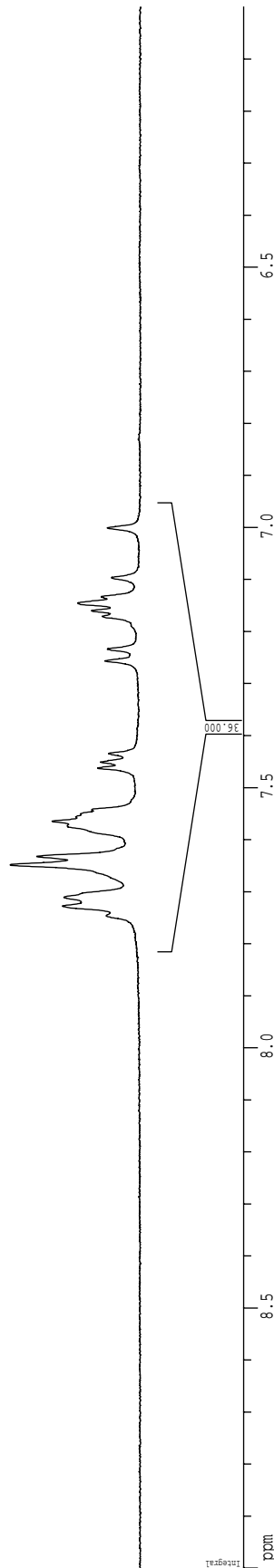


linear hexamer
 $\text{H}-(p\text{-Abc}^2\text{K}-o\text{-Abc}^2\text{K})_3-\text{CO}_2\text{H}$ (9Lb)

¹H NMR (500 MHz, 298 K, D₂O) spectrum of linear hexamer **9Lb** (aromatic region)



linear hexamer
H-(p-Abc²K_o-Abc²K)₃-CO₂H (**9Lb**)



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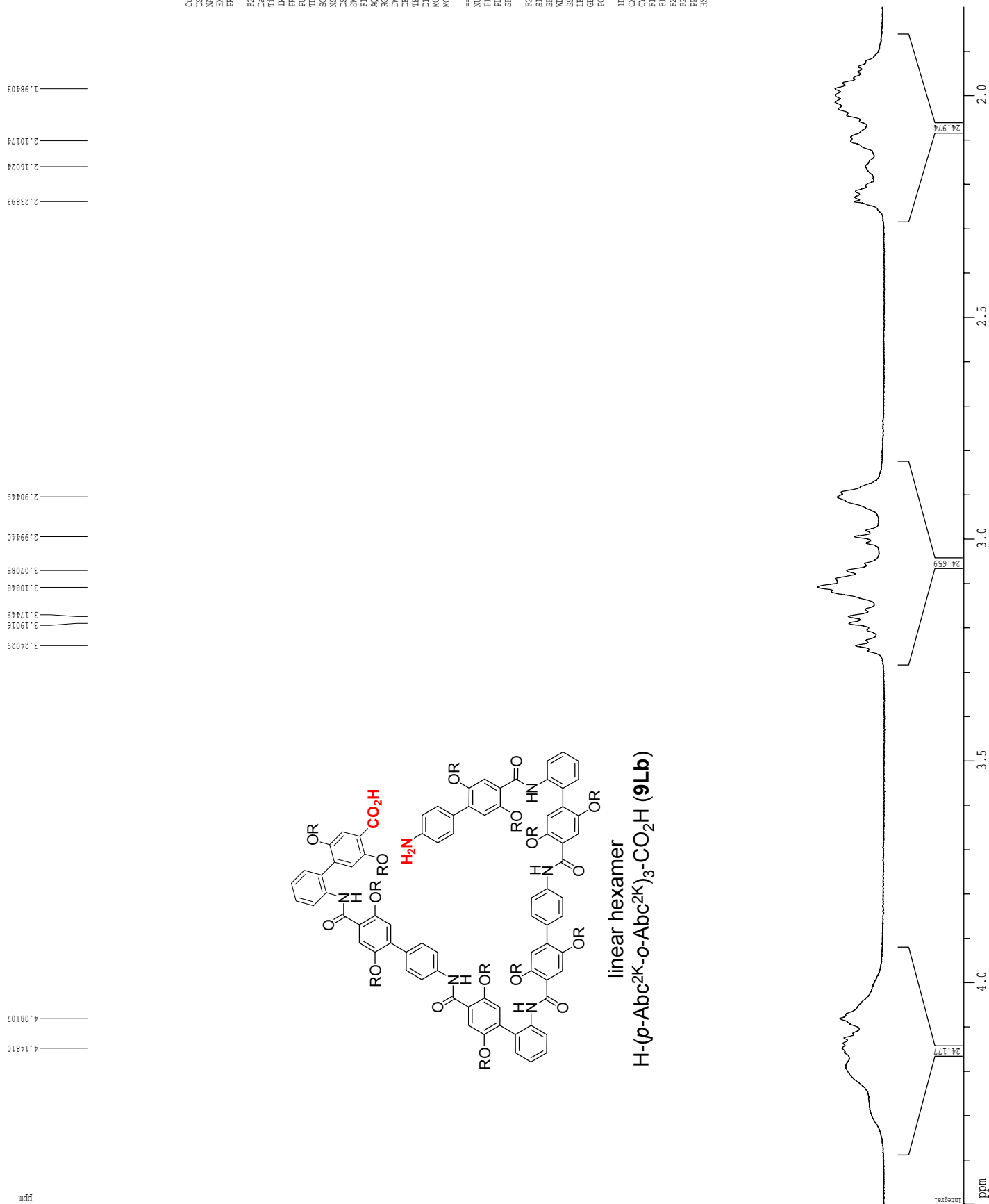
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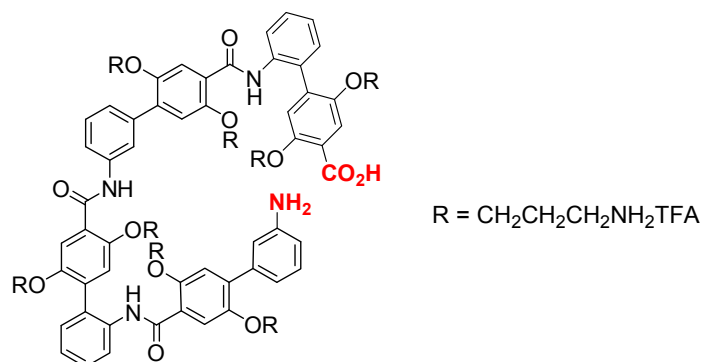
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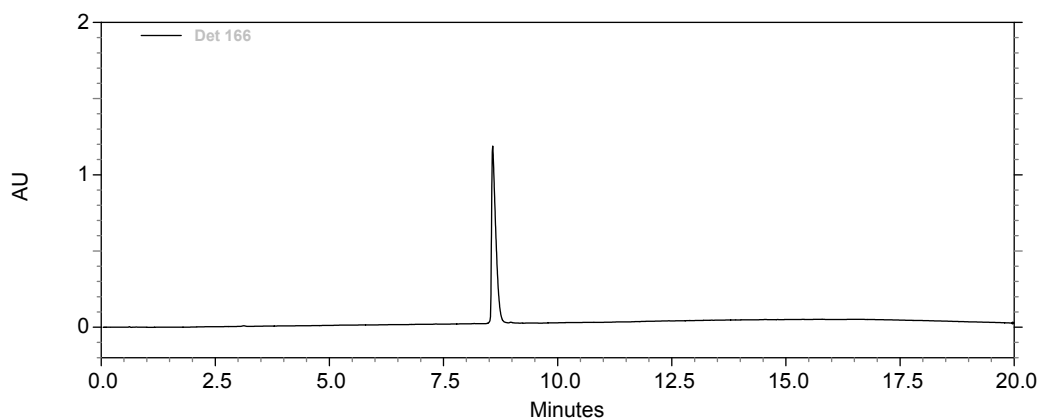
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^1H NMR (500 MHz, 298 K, D_2O) spectrum of linear hexamer **9Lb** (aliphatic region)

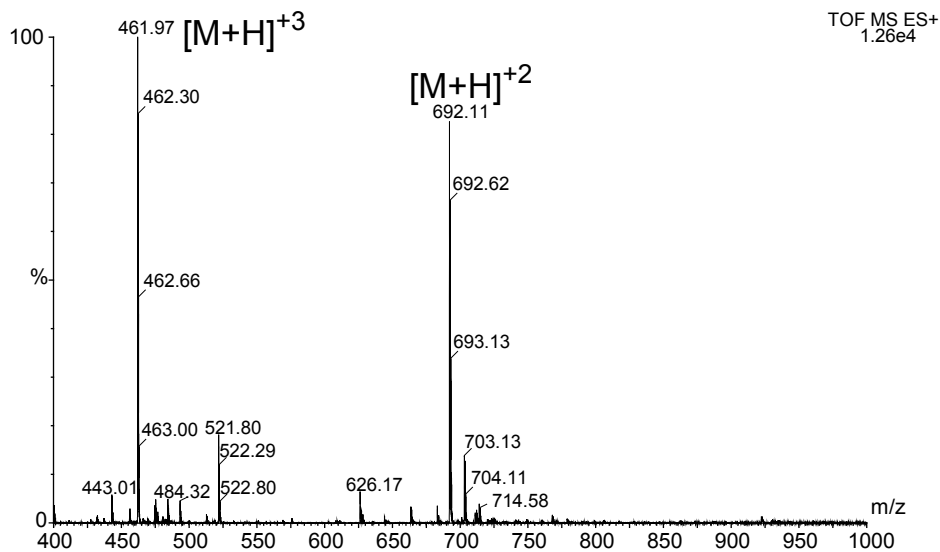
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Analytical RP-HPLC chromatograph and mass spectrum (ESI-MS)

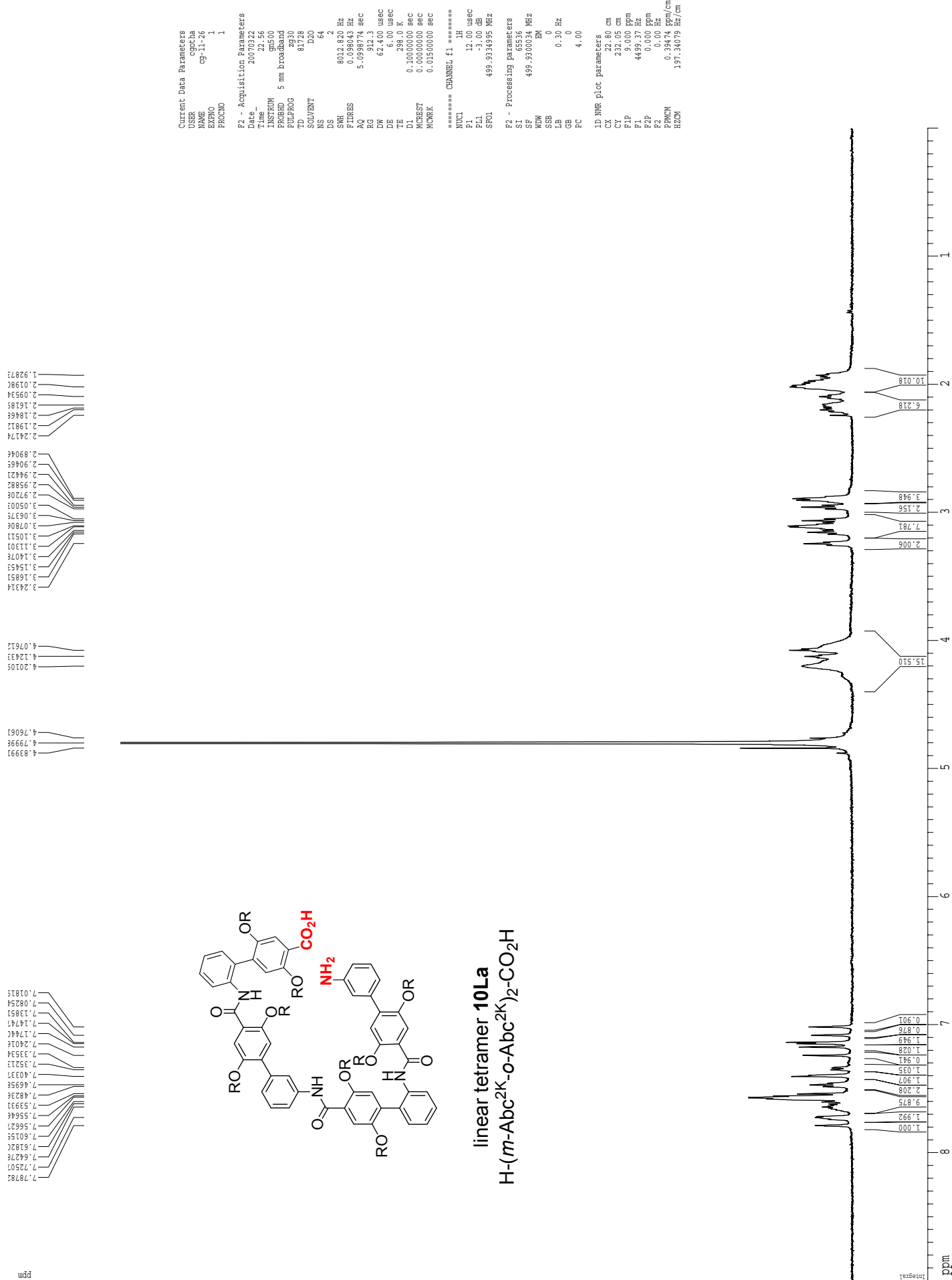


(a) Analytical RP-HPLC (0-30% acetonitrile with 0.1% TFA over 10 min, $\lambda = 214$)

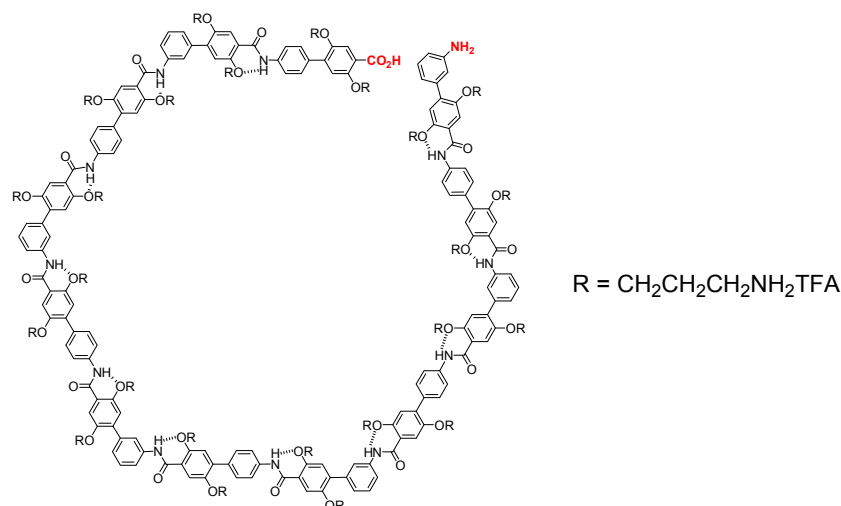


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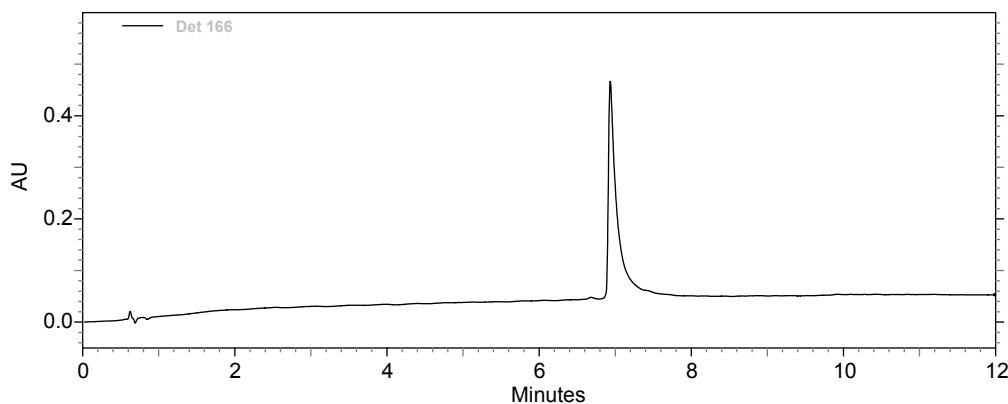


¹H NMR (500 MHz, 298 K, D₂O) spectrum of linear tetramer 10La

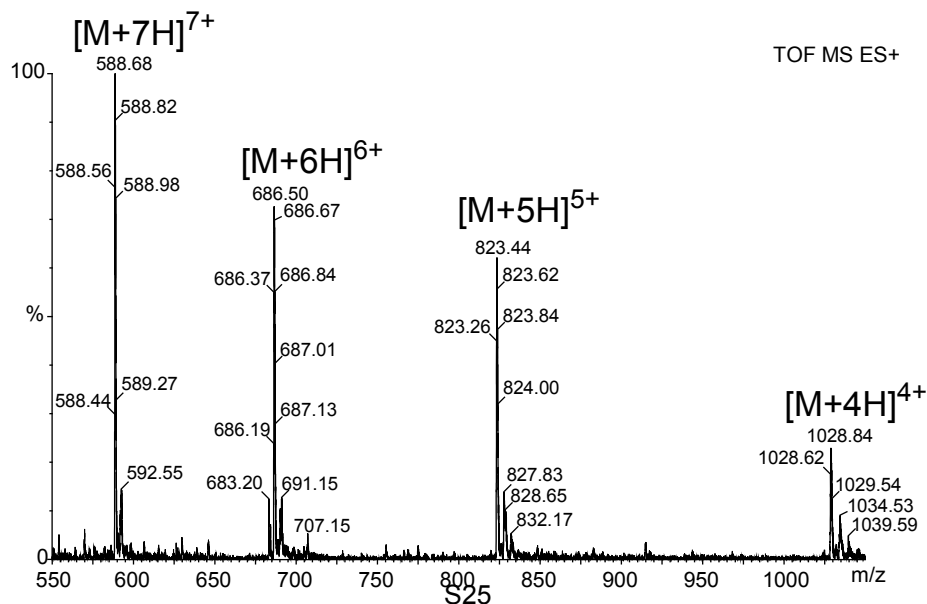
linear dodecamer: H-(*m*-Abc^{2K}-*p*-Abc^{2K})₆-CO₂H (**11Ld**)
 Analytical RP-HPLC chromatograph and mass spectrum (ESI-MS)



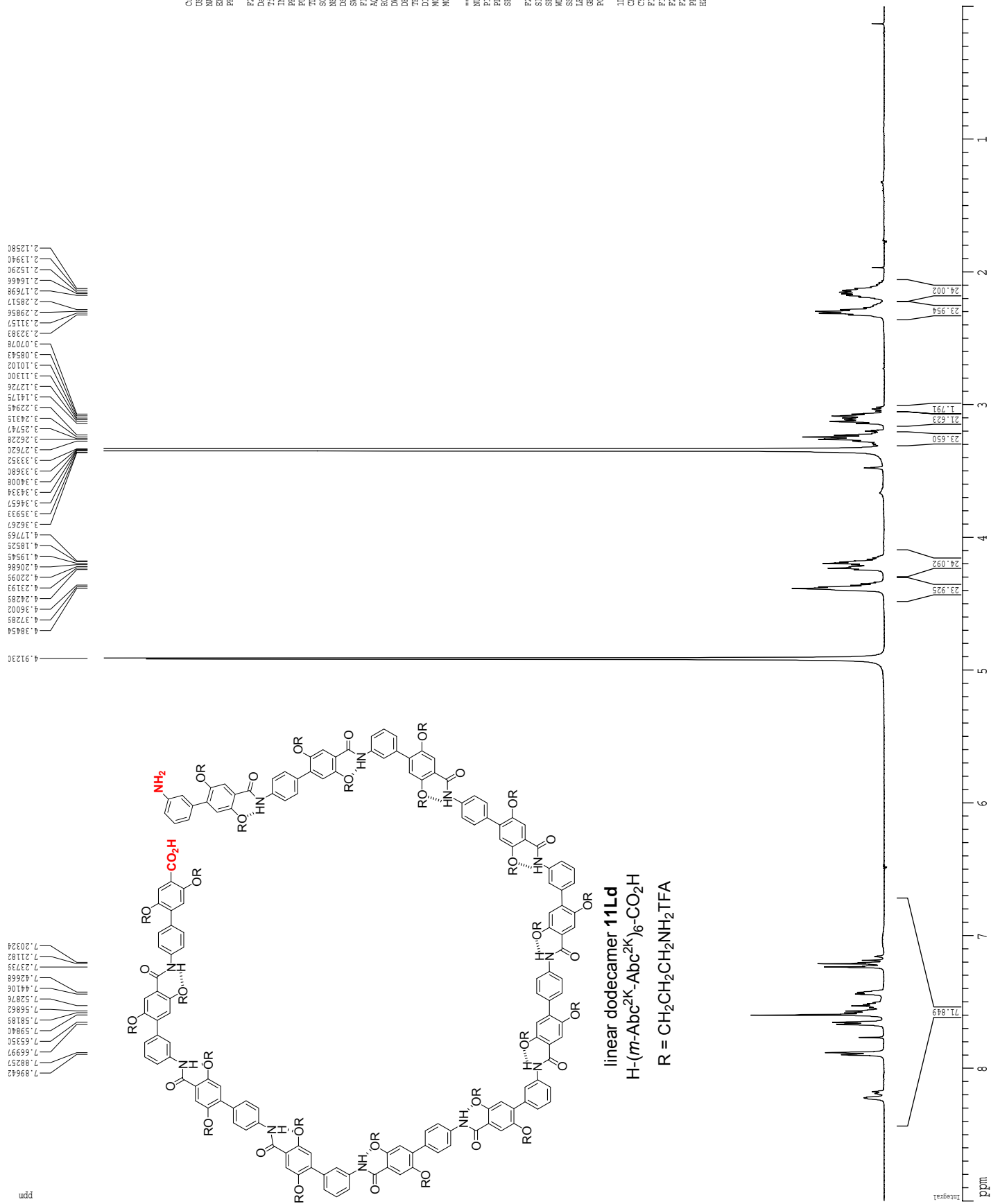
(a) Analytical RP-HPLC (5-50% acetonitrile with 0.1% TFA over 10 min, $\lambda = 214$)



(b) ESI Mass spectrum. (Calcd exact mass for C₂₂₈H₂₇₈N₃₆O₃₇ [M] = 4112.10)



¹H NMR (500 MHz, 298 K, CD₃OD) spectrum of linear dodecamer 11Ld



Supporting Information

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 AQ 5.098774 sec
 RG 9
 DW 62.400 usec
 DE 6.00 usec
 TE 298.0 K
 DL 0.1000000 sec
 ACRESST 0.0000000 sec
 ACUREK 0.0150000 sec

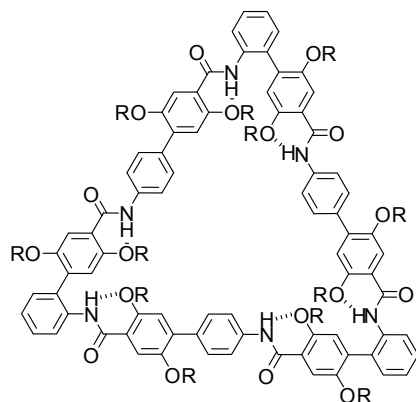
===== CHANNEL f1 =====
 NUC1 1H
 P1 8.00 usec
 PL1 1.60 dB
 SFO1 500.2235015 MHz

F2 - Processing parameters
 SI 65536
 SF 500.2200040 MHz
 WDW EN
 SSB 0
 LB 0.30 Hz
 GB 0
 PC 4.00

ID: NMR plot parameters
 CX 22.80 cm
 CY 177.60 cm
 F1P 9.000 ppm
 F1 4501.98 Hz
 F2P 0.000 Hz
 F2 0.00 Hz
 FREQN0 0.38474 MHz
 HZCNV 197.45528 MHz

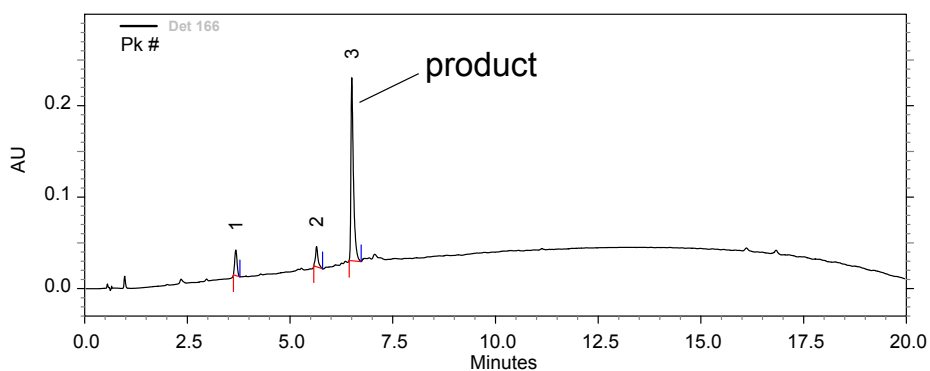
Gothard and J. S. Nowick

Supporting Information for *Journal of Medicinal Chemistry* Nowick
Characterization of crude (*unpurified*) cyclohexamer **9b**
Analytical RP-HPLC chromatograph and mass spectrum (ESI-MS)



$(o\text{-Abc}^{2K}\text{-}p\text{-Abc}^{2K})_3$
 $R = \text{CH}_2\text{CH}_2\text{CH}_2\text{NH}_2\text{TFA}$

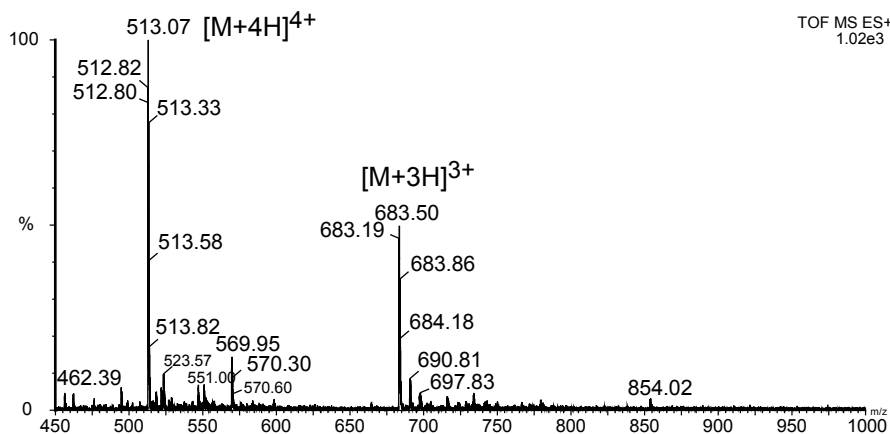
RP-HPLC of crude cyclohexamer triangle **9b**
 (5-90% acetonitrile with 0.1% TFA over 20 min, $\lambda = 214$)



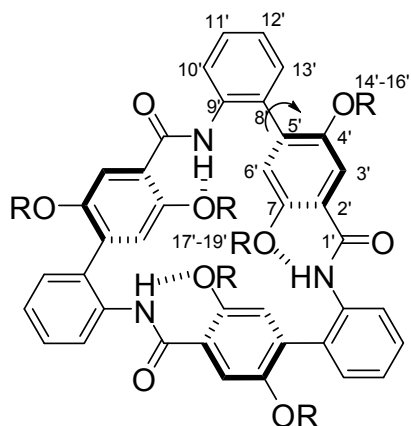
Det 166 Results

Time	Area	Area %	Height	Height %	Pk #
3.680	111117	10.46	27894	11.15	1
5.645	93691	8.82	22120	8.84	2
6.502	857708	80.72	200092	80.00	3
Totals	1062516	100.00	250106	100.00	

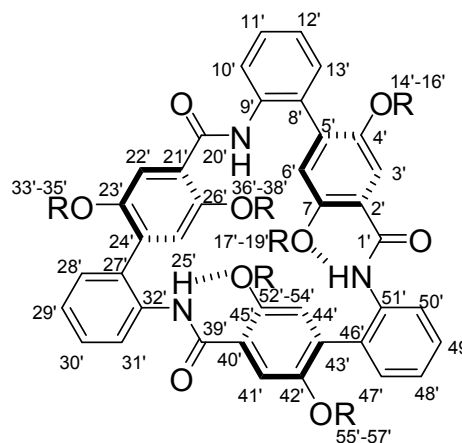
ESI-MS of crude cyclohexamer triangle **9b**
 (Exact MS for $\text{C}_{114}\text{H}_{138}\text{N}_{18}\text{O}_{18} = 2047.04$)



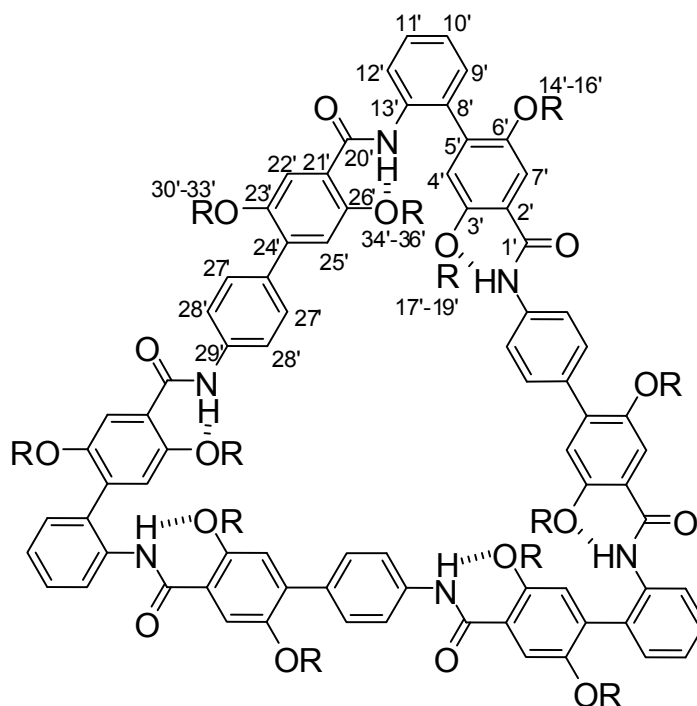
Macrocyclic Symmetry (9a, 9b)



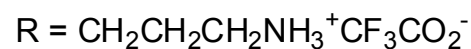
cyclotrimer triangle **9a**
(3 fold symmetric conformer)



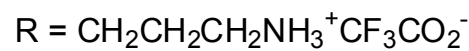
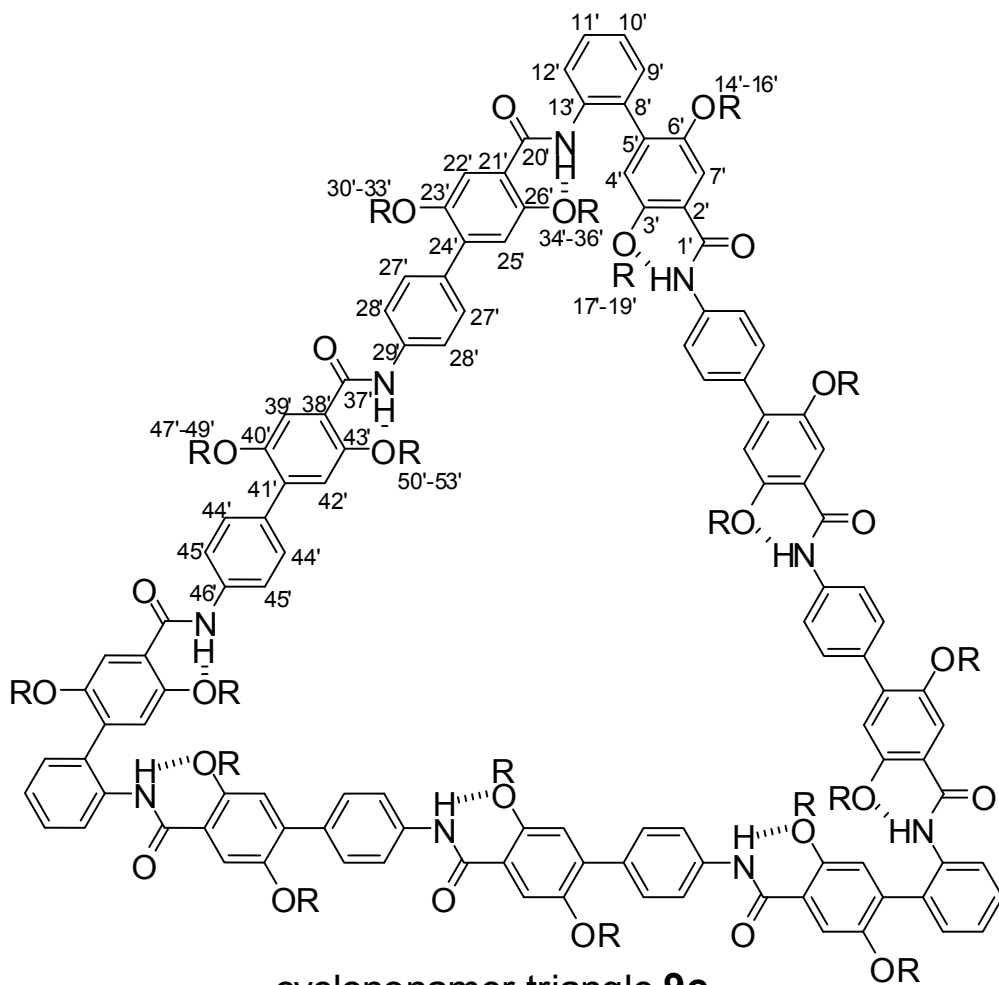
cyclotrimer triangle **9a**
(unsymmetric conformer)



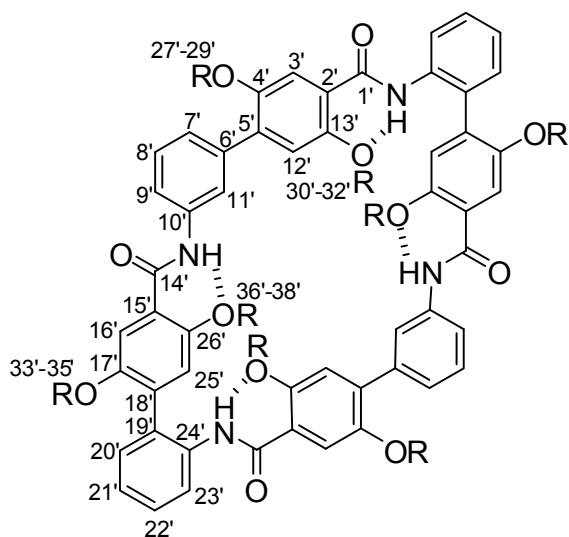
cyclohexamer triangle **9b**
(3-fold symmetry)



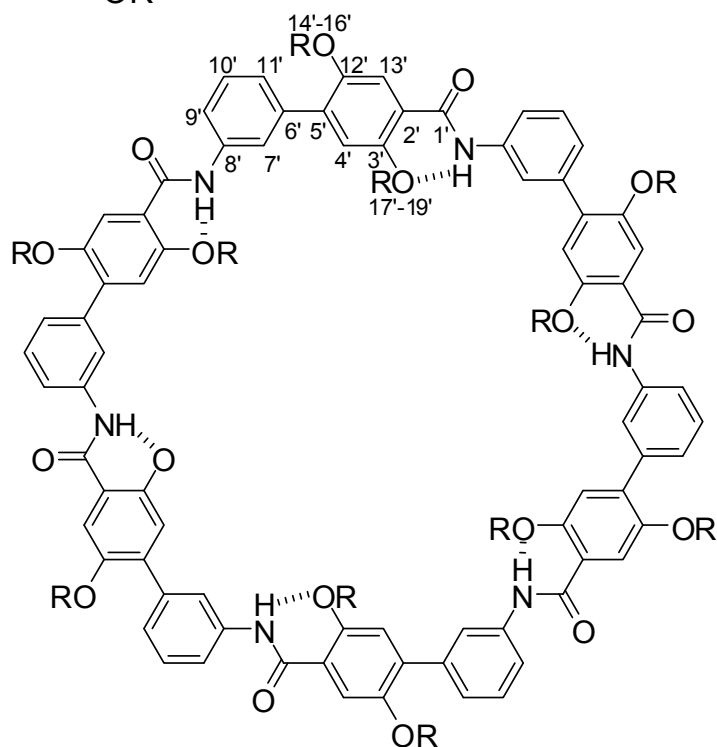
Macrocyclic Symmetry (9c, 10a, 11a)



cyclonamer triangle **9c**
(3-fold symmetry)

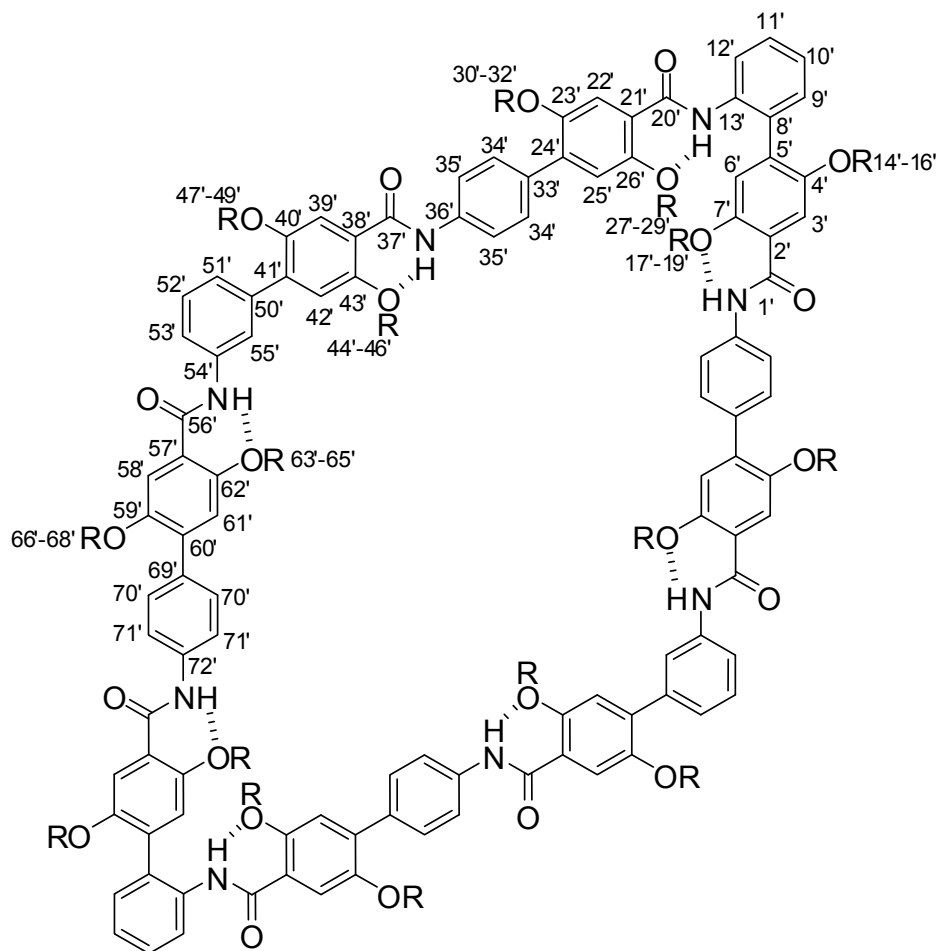


cyclotetramer parallelogram **10a**
(2-fold symmetry)

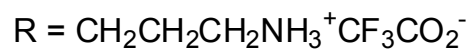


cyclohexamer ring **11a**
(6-fold symmetry)

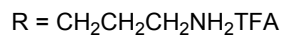
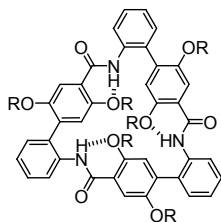
Macrocycle Symmetry (**10b**)



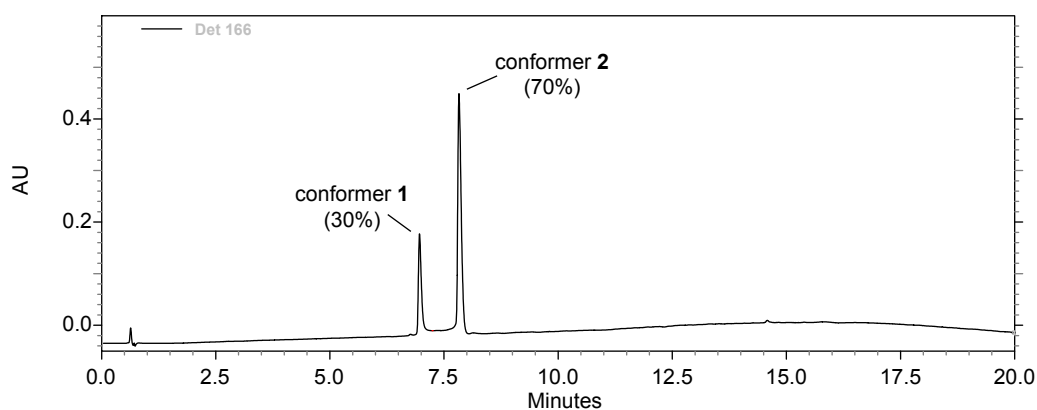
cyclooctamer parallelogram **10b**
(2-fold symmetry)



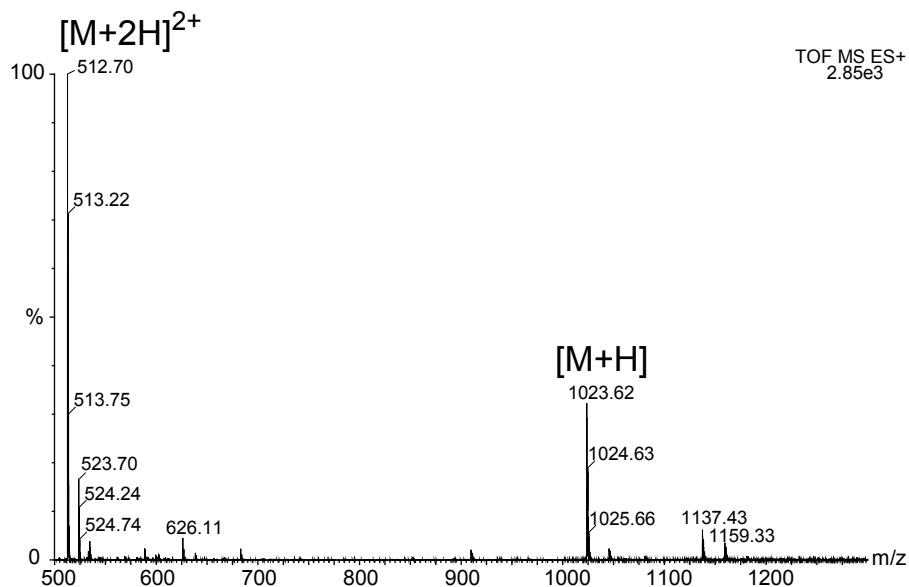
cyclotrimer triangle: cyclo(*o*-Abc^{2K})₃ (**9a**)
Analytical RP-HPLC chromatograph and mass spectrum (ESI-MS)

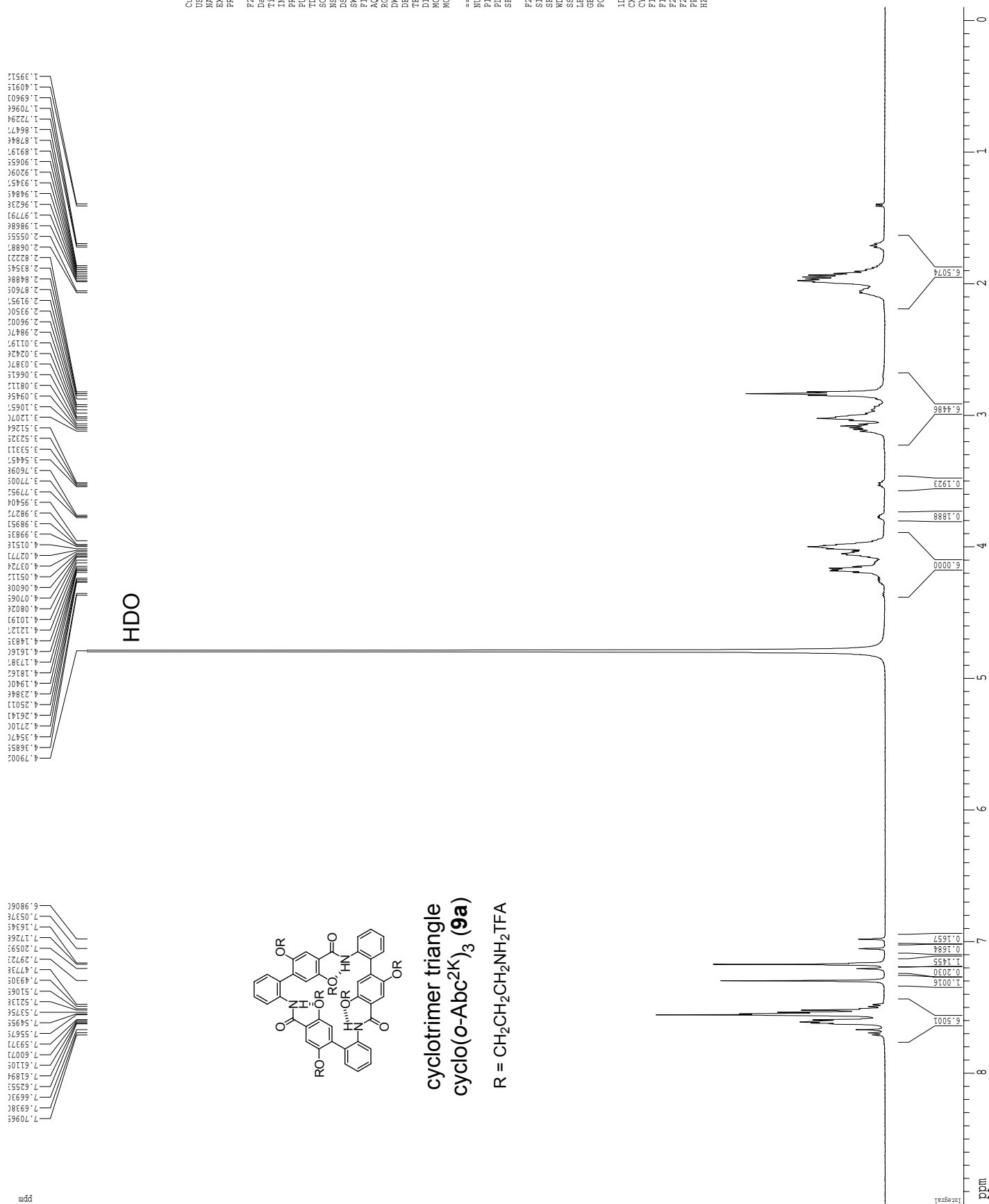


(a) Analytical RP-HPLC (0-30% acetonitrile with 0.1% TFA over 10 min, $\lambda = 214$)



(b) ESI Mass spectrum. (Calcd exact mass for C₅₇H₆₉N₉O₉ [M] = 1023.52)



^1H NMR (500 MHz, 298 K, D_2O) spectrum of cyclotrimer triangle **9a**

```

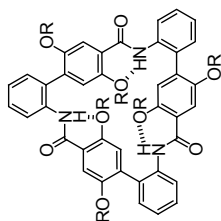
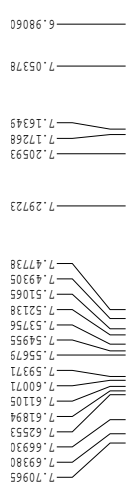
Current Data Parameters
USER          cgotla
NAME          cg-11-49
EXPNO        1
PROCNO       1

F2 - Acquisition Parameters
Date_        20070827
Time         20.17
INSTRUM      cryso00
PROBHD       5 mm CPCL1 1H-
PULPROG      zgpg30
TD           65536
SOLVENT      D2O
NS           16
DS           2
SWH           8012.820 Hz
FIDRES       0.09043 Hz
AQ           5.098774 sec
RG           12.7
DM           62.400 usec
DE           6.00 usec
TE           298.0 K
D1           0.1000000 sec
MORST        0.0000000 sec
MORPK        0.01500000 sec

===== CHANNEL f1 =====
NUC1          1H
P1           8.00 usec
PL1          1.60 dB
SFO1         500.2235015 MHz

F2 - Processing parameters
SI           65536
SF           500.2200083 MHz
WDW          EM
SSB          0
GB           0
PC           4.00

1D NMR plot parameters
CX           22.80 cm
CY           80.96 cm
F1P          9.000 ppm
F1           4501.98 Hz
F2P          -0.100 ppm
F2           -50.02 Hz
P1PCW       0.39512 ppm/cm
H1CN       1.99.64523 Hz/cm
  
```

¹H NMR (500 MHz, 298 K, D₂O) spectrum of cyclotrimer triangle **9a**: Aromatic Region

cyclotrimer triangle
cyclo(*o*-Abc²K)₃ (**9a**)

R = CH₂CH₂CH₂NH₂TFA

```

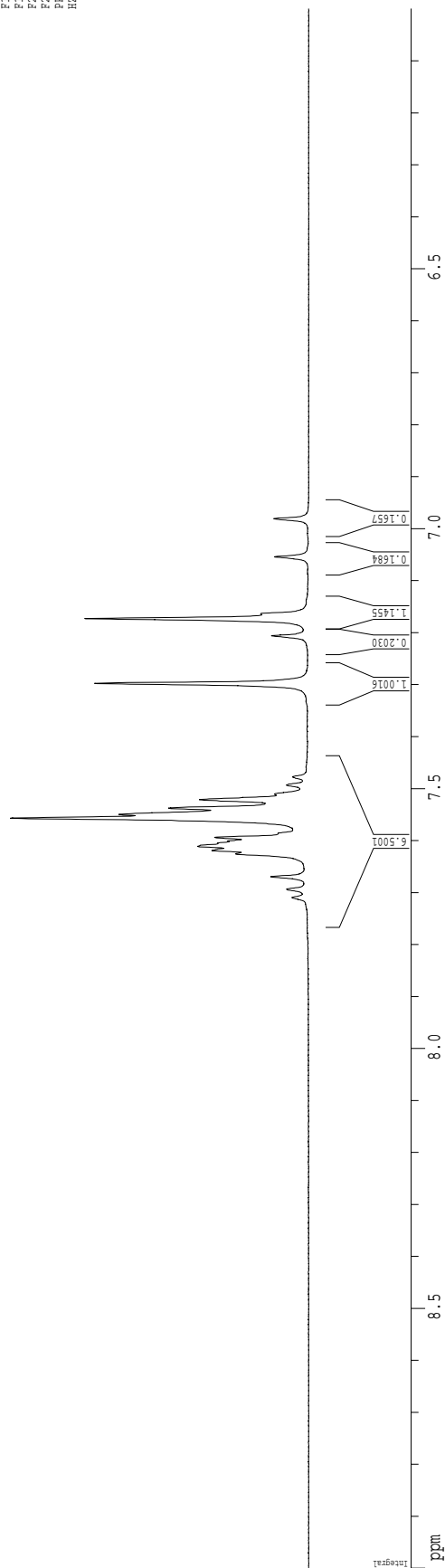
Current Data Parameters
USER      egscha
NAME      C9-11-45
PROCNO    1
PRCNO     1

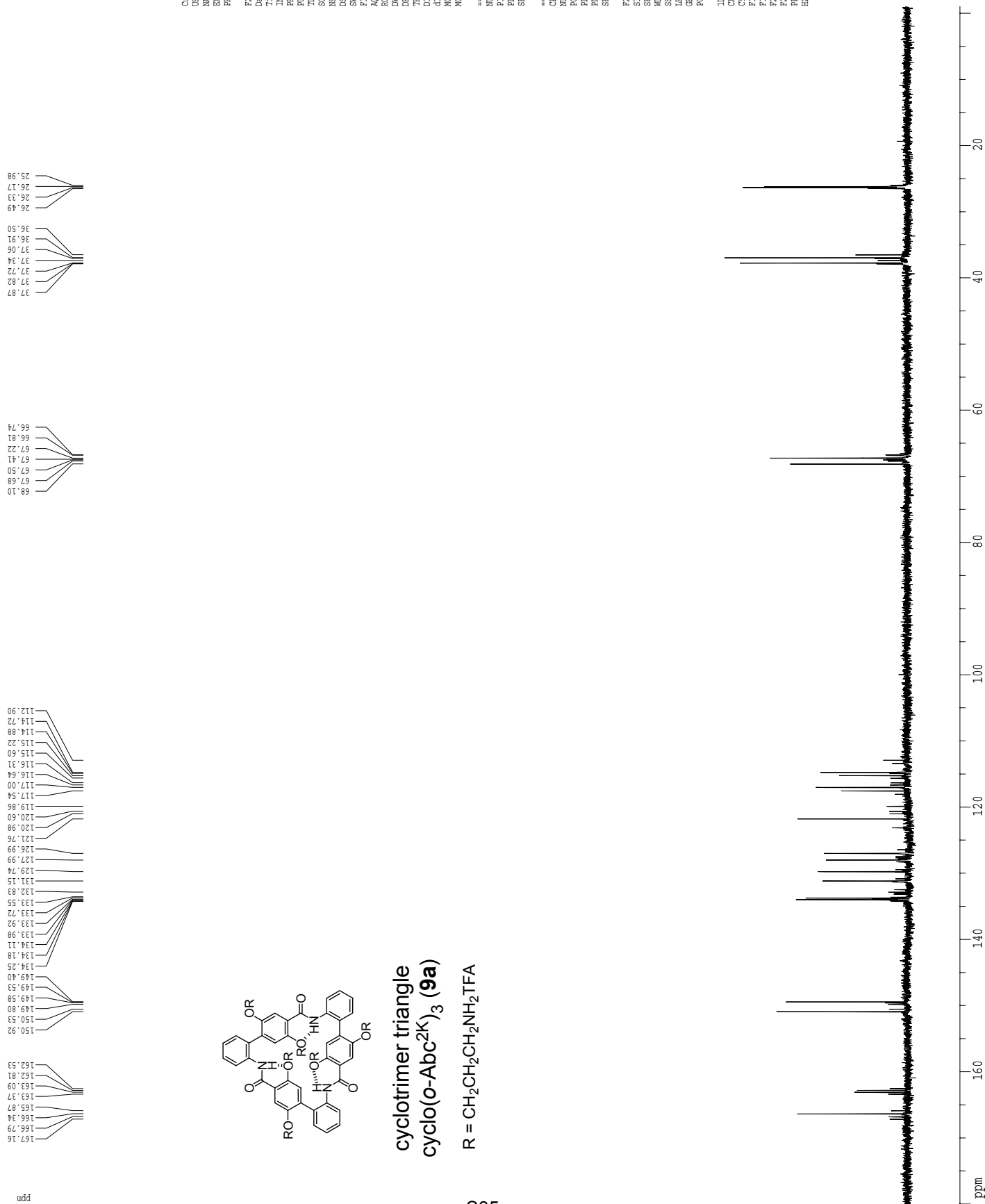
F2 - Acquisition Parameters
Date_     20070827
Time      20.17
INSTRUM   crys500
PROBHD    5 mm CPTCI 1H-
PULPROG   zg30
TD         81728
SOLVENT    D2O
NS         16
DS         2
AQ         0.0312870 Hz
RG         0.636843 Hz
XQ         5.0398774 sec
RG         12.7
DM         62.400 usec
DE         6.00 usec
TE         298.0 K
D1         0.10000000 sec
MCREST    0.00000000 sec
MORRK     0.01500000 sec

===== CHANNEL f1 =====
NUC1       1H
P1         8.00 usec
PL1        0.00 dB
SFO1       500.2235013 MHz

F2 - Processing parameters
SI         65536
SF         500.2200083 MHz
WDW        EM
SSB        0
LB         0.30 Hz
GB         0
PC         4.00

1D NMR plot parameters
CX         22.80 cm
CT         0.16 cm
CI         0.006 cm
F1P        9.000 Hz
F2P        4501.98 Hz
P2P        6.000 ppm
F2         3001.32 Hz
PRCM       0.13158 ppm/cm
HZXCM      65.81842 Hz/cm
  
```

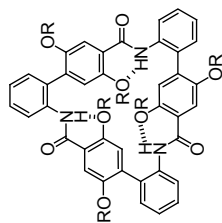


^{13}C NMR (125 MHz, 298 K, D_2O) spectrum of cyclotrimer triangle **9a**

cyclotrimer triangle
 cyclo(o-Abc 2K) $_3$ (**9a**)

R = $\text{CH}_2\text{CH}_2\text{CH}_2\text{NH}_2\cdot\text{TFA}$

¹³C NMR (125 MHz, 298 K, D₂O) spectrum of cyclotrimer triangle **9a**

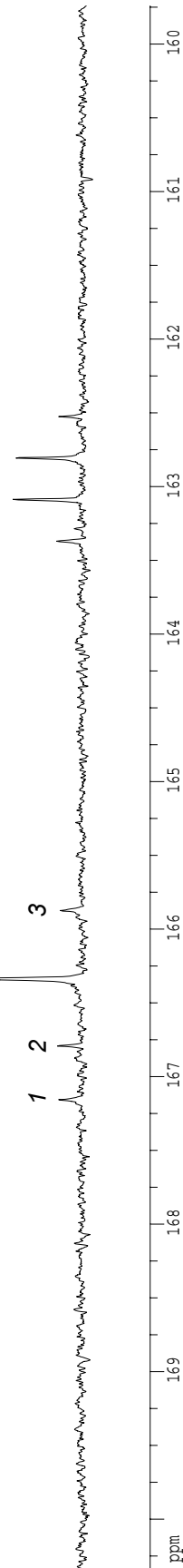


cyclotrimer triangle
cyclo(o-Abc^{2K})₃ (**9a**)

R = CH₂CH₂CH₂NH₂TFA



COCF₃
J = 35.0 Hz



Current Data Parameters
USBR csp01a
NAME 09-11-49
PROCNO 4
PROBNO 1

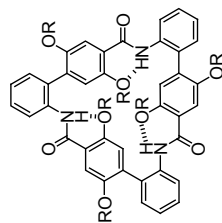
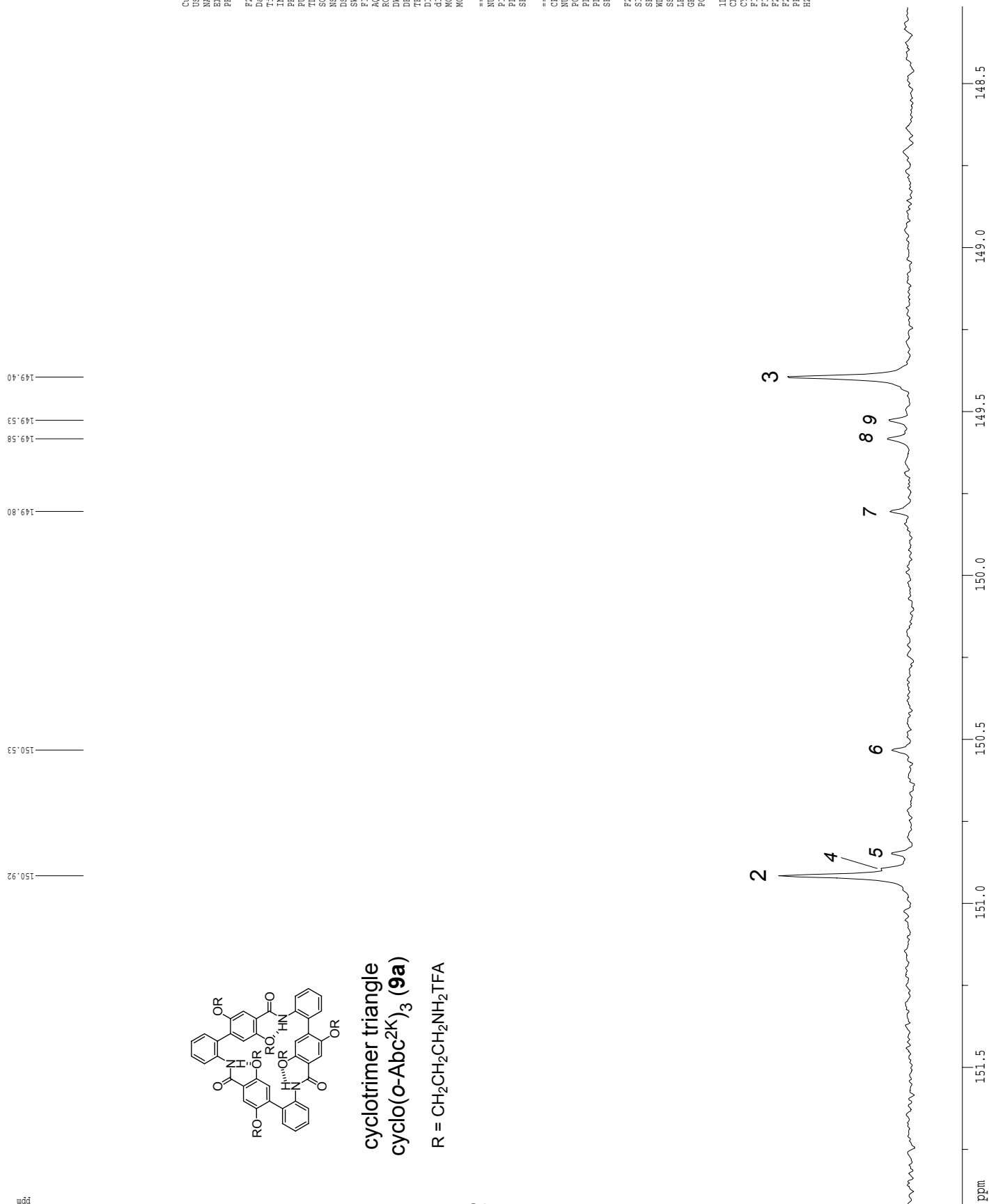
F2 - Acquisition Parameters
Date_ 20070827
Time_ 20.33
INSTRUM cye500
PROBHD 5 mm CPTCI 1H-
PULPROG zgpg30
TD 65418
SOLVENT D2O
NS 2958
DS 4
SWH 30303.031 Hz
FIDRES 0.463222 Hz
AQ 1.0794470 sec
RG 18390.4
DM 16.500 usec
DE 6.00 usec
TE 29.500000 K
D1 0.25000000 sec
d11 0.03000000 sec
MGEST 0.00000000 sec
MORCK 0.01500000 sec

===== CHANNEL F1 =====
NUC1 13C
P1 15.00 usec
PL1 -1.00 dB
SFO1 125.7942548 MHz

===== CHANNEL E2 =====
CPDPRG2 waltz16
NUC2 1H
PCPDZ 100.00 usec
PL2 1.60 dB
PL12 21.54 dB
SFO2 500.2225011 MHz

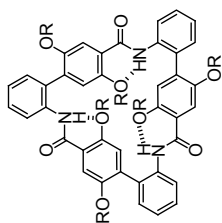
F2 - Processing Parameters
SI 65836
SF 125.7804190 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 2.00

1D NMR plot parameters
CX 22.80 cm
CY 3.49 cm
FIP 170.344 ppm
FL 21425.96 Hz
FZP 159.742 ppm
WDW 0.0923 Hz
SSB 0
FREQ 99.49070 Hz/cm
HZCM

^{13}C NMR (125 MHz, 298 K, D_2O) spectrum of cyclotrimer triangle **9a**

cyclotrimer triangle
 cyclo-(o-Abc^{2K})₃ (**9a**)

R = $\text{CH}_2\text{CH}_2\text{CH}_2\text{NH}_2\text{TFA}$

¹³C NMR (125 MHz, 298 K, D₂O) spectrum of cyclotrimer triangle 9a

cyclotrimer triangle
cyclo(o-Abc^{2K})₃ (9a)

R = CH₂CH₂CH₂NH₂TFA

S38

```

Current Data Parameters
USBR          cgotcia
NAME          c9-11-49
EXPNO        4
PROCNO       1

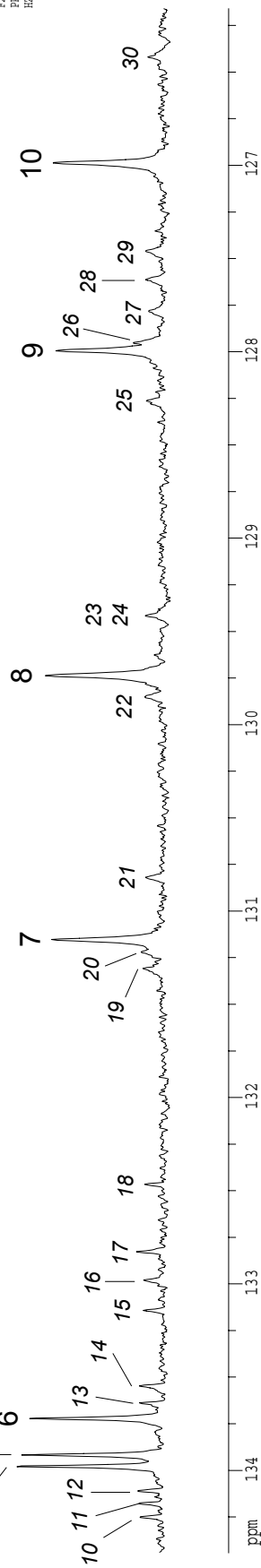
F2 - Acquisition Parameters
Date_         20070827
Time_        20.33
INSTRUM      cryo500
PROBHD       5 mm CPTCI 1H-
PULPROG      zgpg30
TD           65418
SOLVENT      D2O
NS           2958
DS           4
SWH          30303.031 Hz
FIDRES       0.463222 Hz
AQ           1.0794470 sec
RG           183.90.4
DM           16.500 usec
DE           6.00 usec
TE           300.2 K
D1           0.2500000 sec
d11          0.0300000 sec
MORST        0.0000000 sec
MORWK        0.0150000 sec

===== CHANNEL f1 =====
NUC1         13C
P1           15.00 usec
PL1          -1.00 dB
SFO1        125.7942548 MHz

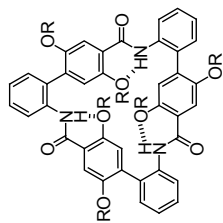
===== CHANNEL f2 =====
CPDPRG2      waltz16
NUC2         1H
PCPD2        100.00 usec
PL2          1.00 dB
PL12         23.54 dB
SFO2        500.2225011 MHz

F2 - Processing parameters
SI           65536
SF          125.7904130 MHz
WDW          EM
SSB          0
LB           1.00 Hz
GB           0
PC           2.00

1D NMR plot parameters
CX          22.80 cm
CY           3.49 cm
FIP         134.443 ppm
FL          16910.26 Hz
FZP         126.160 ppm
FR          15868.42 Hz
GAMMA1      0.38323 ppm/cm
HZCN        45.69448 Hz/cm
  
```



¹³C NMR (125 MHz, 298 K, D₂O) spectrum of cyclotrimer triangle **9a**



cyclotrimer triangle
cyclo(o-Abc^{2k})₃ (**9a**)

R = CH₂CH₂CH₂NH₂TFA

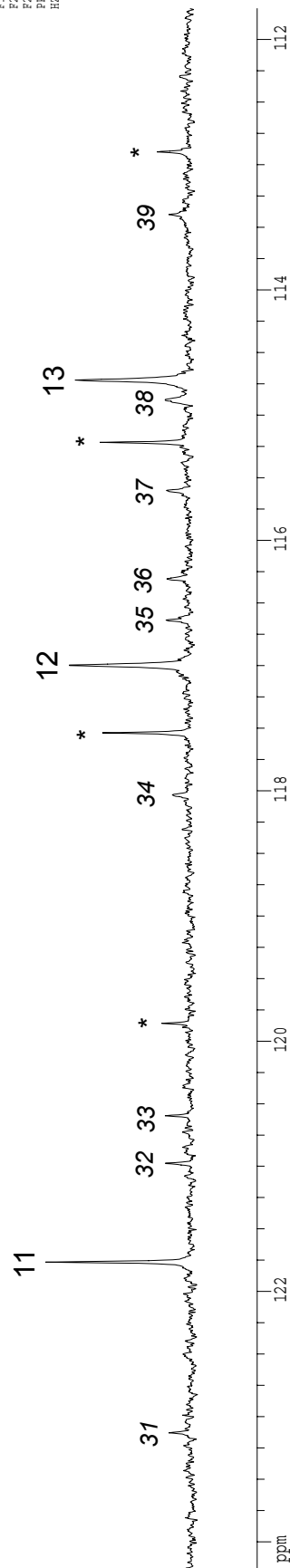
Current Data Parameters
USBR cgottha
NAME c9-11-49
EXPNO 4
PROCNO 1
F2 - Acquisition Parameters
Date_ 20070827
Time_ 20.33
INSTRUM cye500
PROBHD 5 mm CPTCI IH-
PULPROG zgpg30
TD 65418
SOLVENT D2O
NS 2958
DS 4
SWH 30303.031 Hz
FIDRES 0.463222 Hz
AQ 1.0794470 sec
RG 18390.4
DM 16.500 usec
DE 6.00 usec
TE 29.00 K
D1 0.2500000 sec
d11 0.0300000 sec
MGEST 0.0000000 sec
MORCK 0.0150000 sec

S39

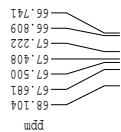
* = CF₃
J = 290.4 Hz

C. M. Gothard and J. S. Nowick

===== CHANNEL F1 =====
NUC1 13C
P1 15.00 usec
PL1 -1.00 dB
SFO1 125.7942548 MHz
===== CHANNEL E2 =====
CPDPRG2 waltz16
NUC2 1H
PCPD2 100.00 usec
PL2 1.60 dB
PL12 23.54 dB
SFO2 500.2225011 MHz
F2 - Processing parameters
SI 65836
SF 125.7804190 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 2.00
1D NMR plot parameters
CX 22.80 cm
CY 3.49 cm
FIP 124.223 ppm
FL 15624.85 Hz
FZP 111.754 ppm
FAP 1.05641 Hz
SFRCH 68.652 ppm/cm
HZCM 68.79326 Hz/cm

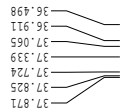
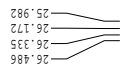


¹³C NMR (125 MHz, 298 K, D₂O) spectrum of cyclotrimer triangle **9a**



cyclotrimer triangle
cyclo(*o*-Abc²K)₃ (**9a**)

R = CH₂CH₂CH₂NH₂TFA



Current Data Parameters
USBR cgottha
NAME 09-11-49
PAPRO 4
PROCNO 1

F2 - Acquisition Parameters
Date_ 20070827
Time_ 20.33
INSTRUM ctye500
PROBHD 5 mm CPTCI 1H-
PULPROG zgpg30
TD 65418
SOLVENT D2O
NS 2958
DS 4
SWH 30303.031 Hz
FIDRES 0.463222 Hz
AQ 1.0794470 sec
RG 18390.4
DM 16.500 usec
DE 8.000 usec
TE 29.500000 K
D1 0.25000000 sec
d11 0.03000000 sec
NOREST 0.00000000 sec
NOVERK 0.01500000 sec

==== CHANNEL F1 =====
NUC1 13C
P1 15.00 usec
PL1 -1.00 dB
SFO1 125.7942548 MHz

==== CHANNEL E2 =====
CPDPRG2 waltz16
NUC2 1H
PCPD2 100.00 usec
PL2 1.60 dB
PL12 23.54 dB
SFO2 500.2225011 MHz

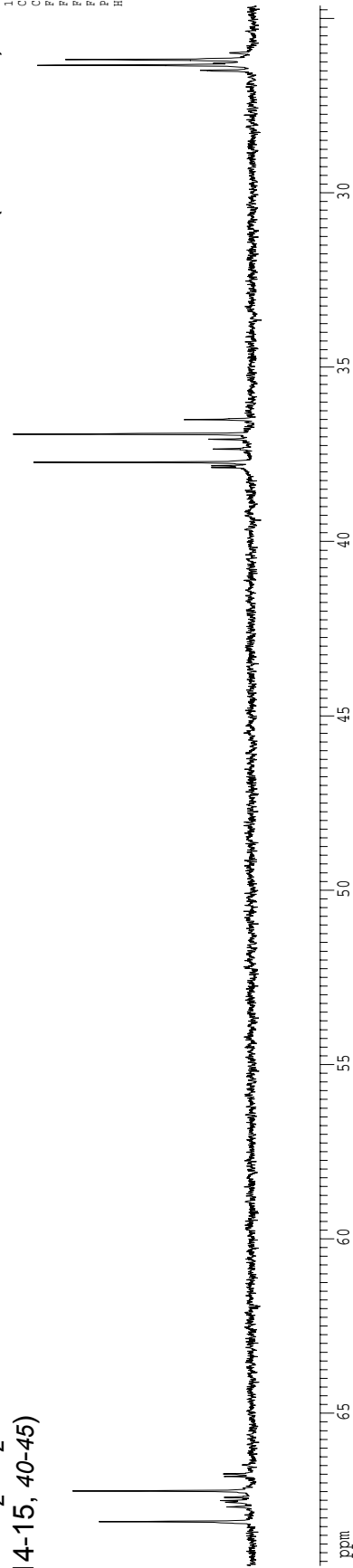
F2 - Processing Parameters
SI 65836
SF 125.7804190 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 2.00

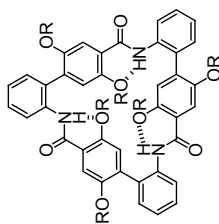
1D NMR plot parameters
CX 22.80 cm
CY 3.49 cm
FIP 69.365 ppm
FL 8724.72 Hz
F2P 24.645 ppm
F2R 3098.90 Hz
SFOCN 1.06132 ppm/cm
HZCN 246.70276 Hz/cm

-CH₂NH₃⁺
(16-17, 46-51)

-CH₂CH₂CH₂-
(18-19, 52-57)

-OCH₂CH₂-
(14-15, 40-45)

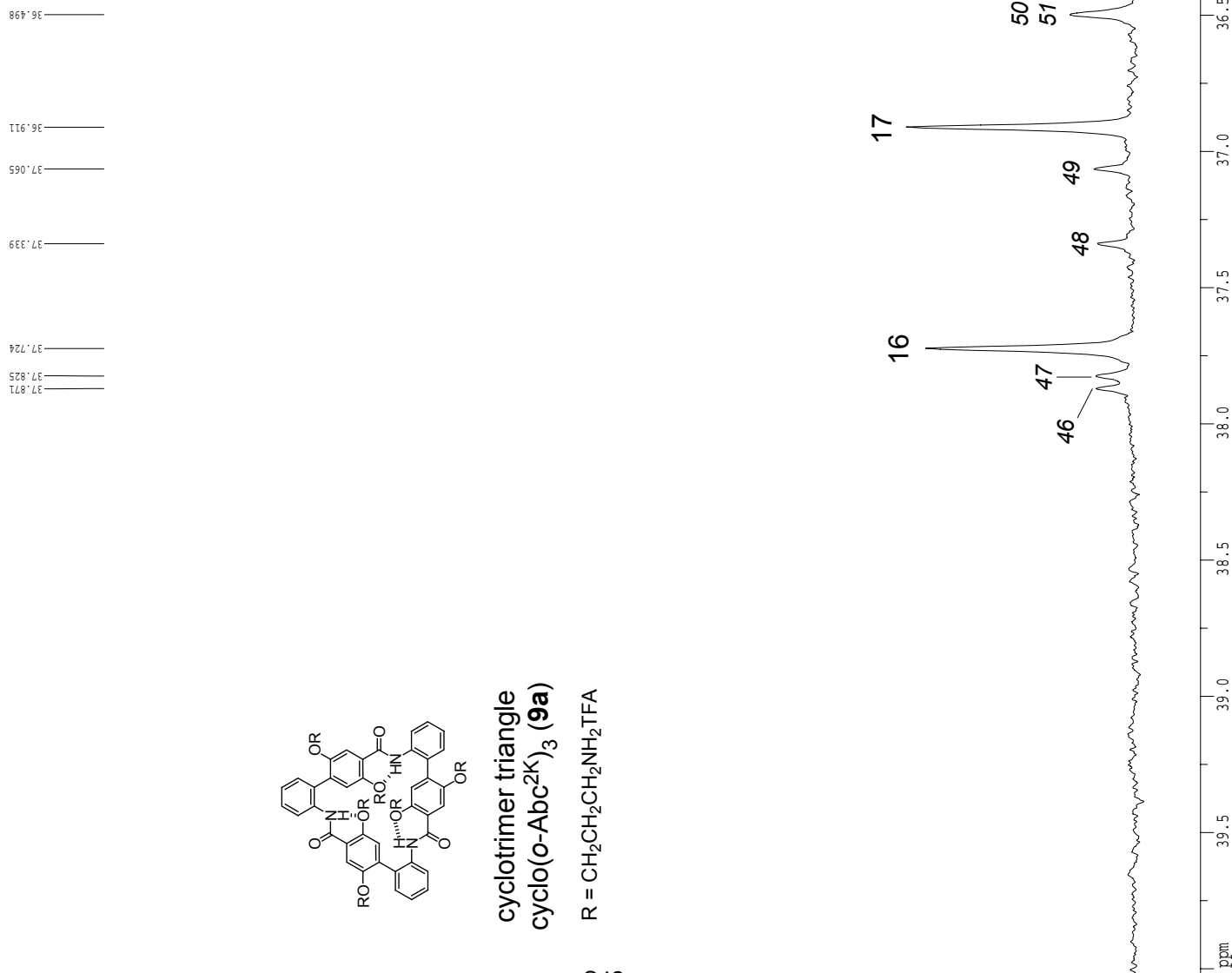


^{13}C NMR (125 MHz, 298 K, D_2O) spectrum of cyclotrimer triangle **9a**

cyclotrimer triangle
cyclo(*o*-Abc^{2K})₃ (**9a**)

R = $\text{CH}_2\text{CH}_2\text{CH}_2\text{NH}_2\cdot\text{TFA}$

S42



```

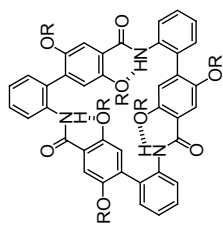
Current Data Parameters
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NAME          09-11-19
PULPROG      zgpg30
PROCNO       4
PROBHD       5 mm QNP1H-
PULPROG      zgpg30
TD           65418
SOLVENT      D2O
NS           2958
DS           4
SWH          30303.031 Hz
FIDRES       0.463222 Hz
AQ           1.0794470 sec
RG           18390.4
DM           16.500 usec
DE           6.00 usec
TE           29.00000000 K
D1           0.25000000 sec
d11          0.03000000 sec
RGRESST      0.00000000 sec
NOVERK       0.01500000 sec

===== CHANNEL F1 =====
NUC1         13C
P1           15.00 usec
PL1          -1.00 dB
SFO1         125.7942548 MHz

===== CHANNEL E2 =====
CPDPRG2      waltz16
NUC2         1H
PCPD2        100.00 usec
PL2          1.60 dB
PL12         23.54 dB
SFO2         500.2225011 MHz

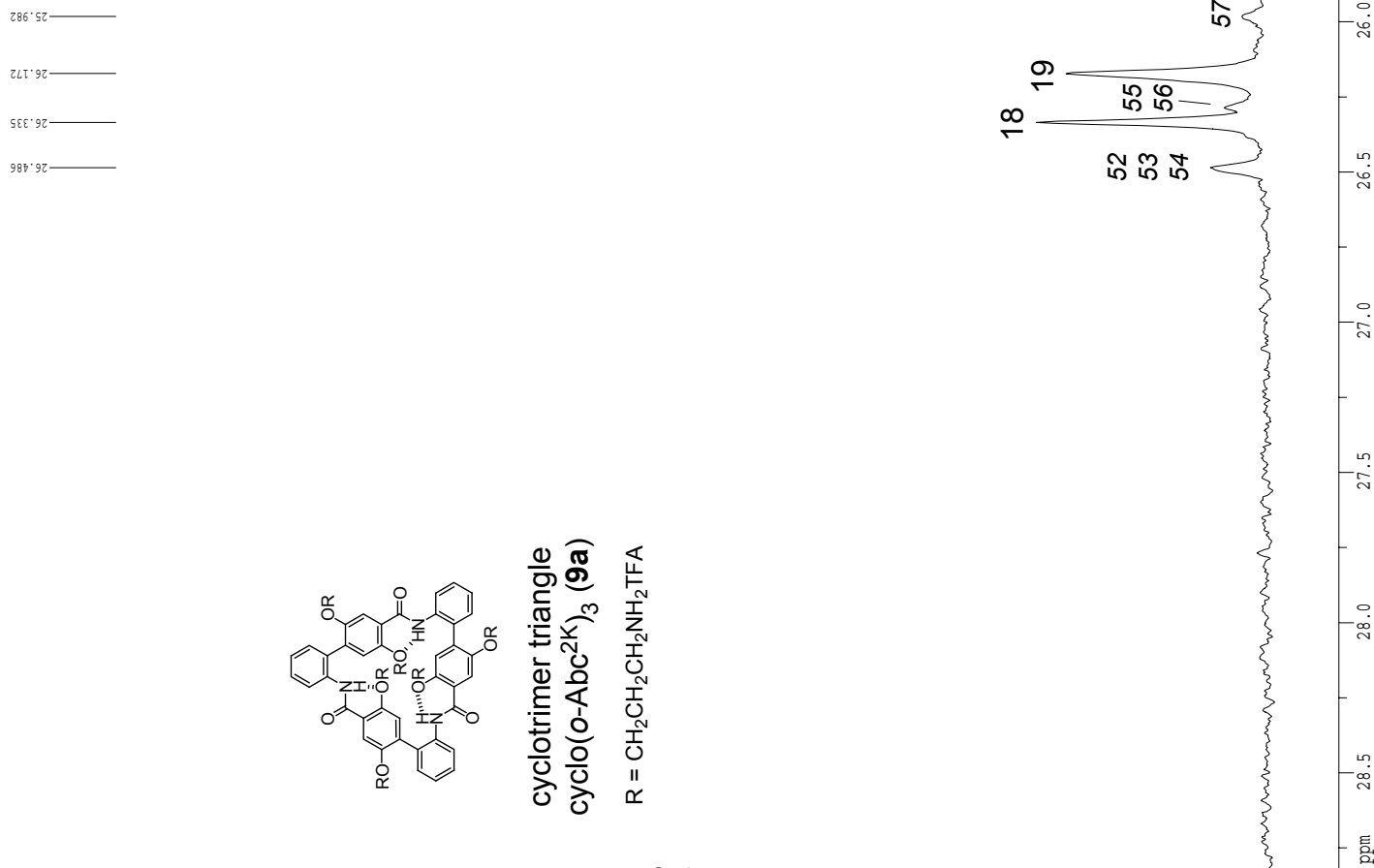
F2 - Processing parameters
SI           65836
SF           125.7804190 MHz
WDW          EM
SSB          0
LB           1.00 Hz
GB           0
PC           2.00

1D NMR plot parameters
CX           22.80 cm
CY           3.49 cm
FIP          40.023 ppm
FL           5034.14 Hz
F2P          34.527 ppm
F3P          0.342176 Hz
SFO(N)       30.32332 ppm/cm
HZCM         30.32332 Hz/cm
  
```


^{13}C NMR (125 MHz, 298 K, D_2O) spectrum of cyclotrimer triangle **9a**

cyclotrimer triangle
 $\text{cyclo}(\text{o-Abc}^{\text{ZK}})_3$ (**9a**)

R = $\text{CH}_2\text{CH}_2\text{CH}_2\text{NH}_2\text{TFA}$



```

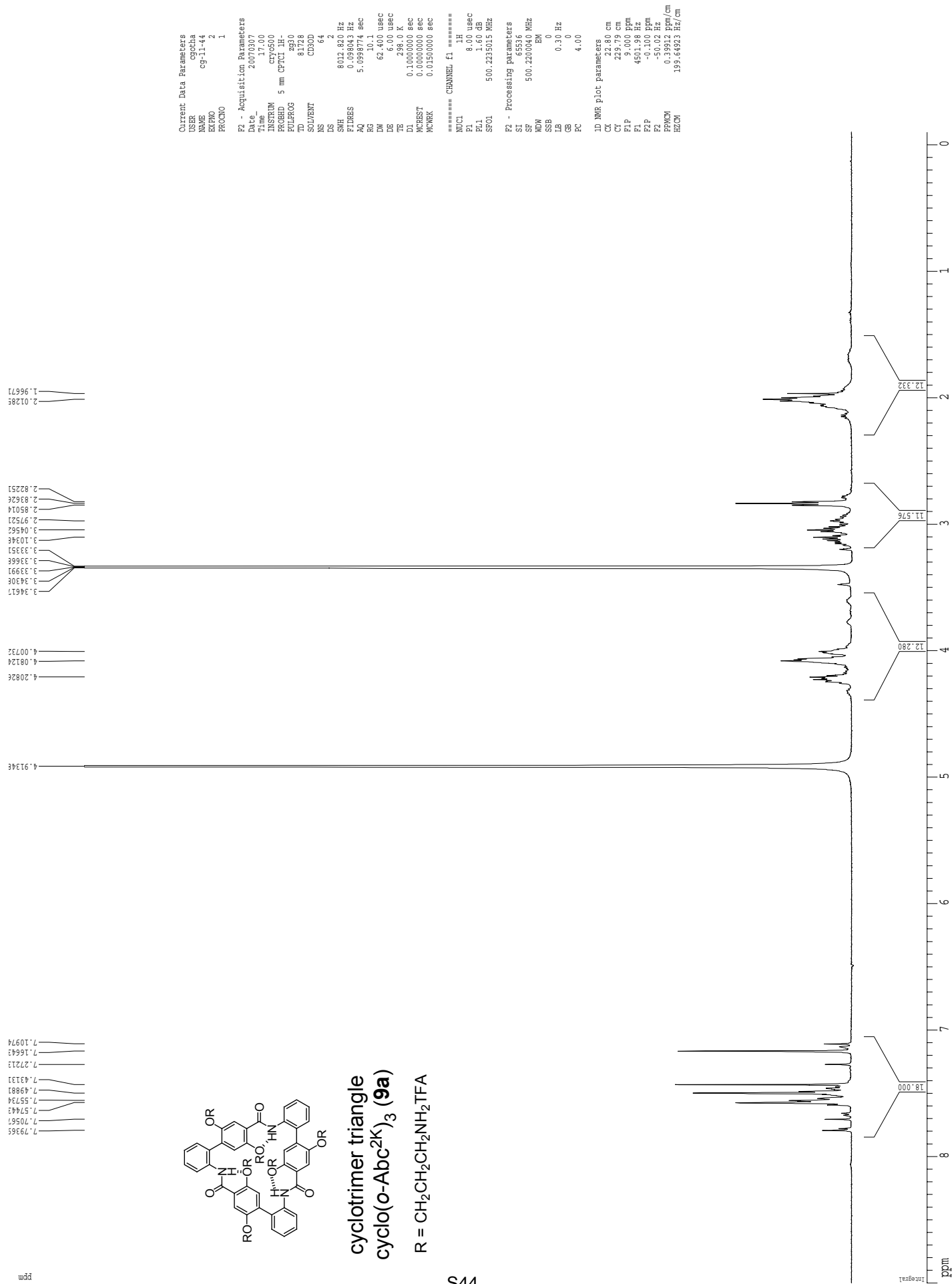
Current Data Parameters
USBR          cgotth
NAME          c9-11-19
PULPROG      zgpg30
PROCNO       4
PROBHD       5 mm QNP1H-
PULPROG      zgpg30
TD           65418
SOLVENT      D2O
NS           2958
DS           4
SWH          30303.031 Hz
FIDRES       0.463222 Hz
AQ           1.0794470 sec
RG           18390.4
DM           16.500 usec
DE           6.00 usec
TE           298.15 K
D1           0.25000000 sec
d11          0.03000000 sec
NOFEST       0.00000000 sec
NOWEAK       0.01500000 sec

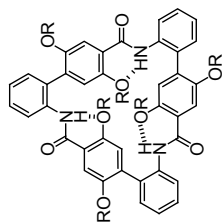
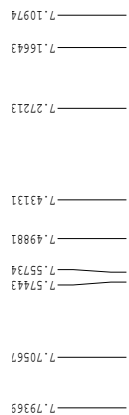
===== CHANNEL F1 =====
NUC1         13C
P1           15.00 usec
PL1          -1.00 dB
SFO1         125.7942548 MHz

===== CHANNEL E2 =====
CPDPRG2      waltz16
NUC2         1H
PCPD2       100.00 usec
PL2          1.60 dB
PL12         23.54 dB
SFO2         500.2225011 MHz

F2 - Processing parameters
SI           65836
SF           125.7804190 MHz
WDW          EM
SSB          0
LB           1.00 Hz
GB           0
PC           2.00

1D NMR plot parameters
CX           22.80 cm
CY           3.49 cm
FIP         28.830 ppm
FL          3626.21 Hz
F2P         23.272 ppm
F3P         0.4118 Hz
SFO(N)      30.65948 Hz/cm
HZCN
  
```

^1H NMR (500 MHz, 298 K, CD_3OD) spectrum of cyclotrimer triangle **9a**

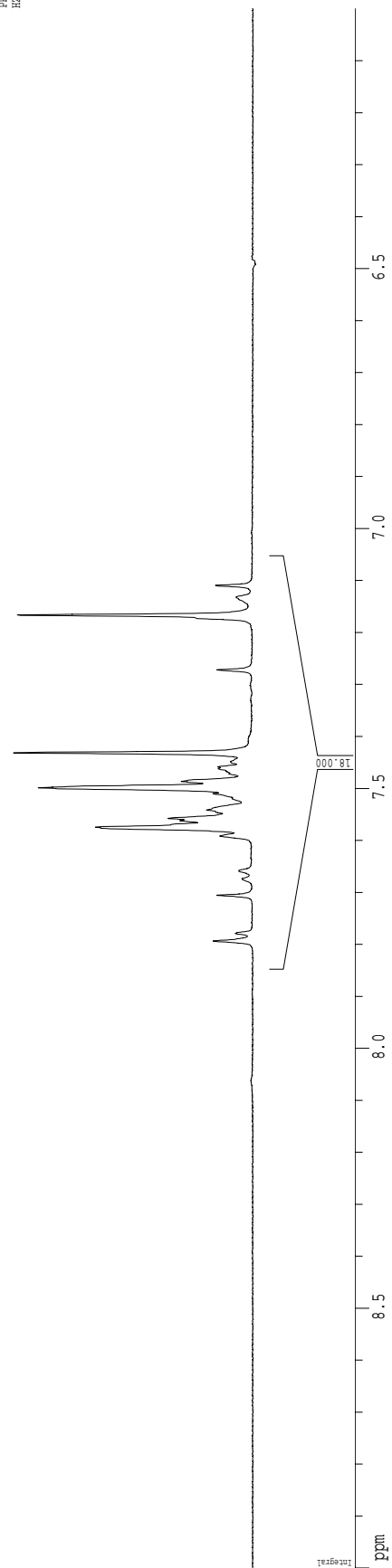
¹H NMR (500 MHz, 298 K, CD₃OD) spectrum of cyclotrimer triangle **9a**

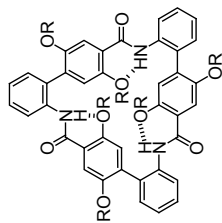
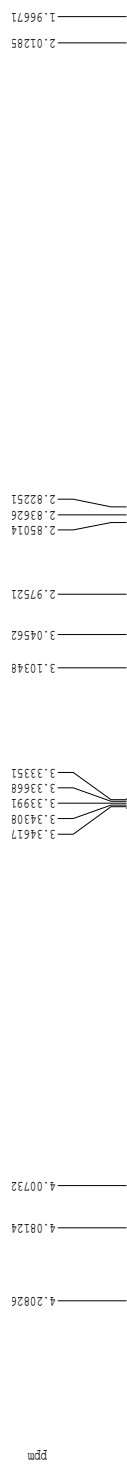
cyclotrimer triangle
cyclo(o-Abc^{2K})₃ (**9a**)

R = CH₂CH₂CH₂NH₂TFA

```

Current Data Parameters
=====
USER          csobla
NAME          CF-11-44
EXPNO        2
PROCNO       1
F2 - Acquisition Parameters
=====
Date_         20070307
Time_        17.00
INSTRUM      cryo500
PROBHD       5 mm CPXI IH-
PULPROG      zg30
TD           81728
SOLVENT      CD3OD
NS           64
DS           2
SWH          8012.820 Hz
FIDRES       0.4943 Hz
AQ           5.0398771 sec
RG           310.1
DM           62.400 usec
DE           6.00 usec
TE           298.0 K
D1           0.10000000 sec
MCREST       0.00000000 sec
MCWARR       0.01500000 sec
===== CHANNEL f1 =====
NUC1          1H
P1           6.00 usec
PL1          -2.00 dB
SFO1         500.2235015 MHz
F2 - Processing parameters
=====
SI           65536
SF           500.2200040 MHz
WDW          EM
SSB          0
LB           0.30 Hz
GB           0
PC           4.00
ID NMR plot parameters
=====
CX           22.50 cm
CY           43.75 cm
EI1          49.00 mm
EI2          49.00 mm
EI3          49.00 mm
FZ1          6.000 ppm
FZ2          3001.32 Hz
PFRMCON      0.13158 ppm/cm
HZCON        65.81842 Hz/cm
  
```



^1H NMR (500 MHz, 298 K, CD_3OD) spectrum of cyclotrimer triangle **9a**

cyclotrimer triangle
cyclo(*o*-Abc^{2K})₃ (**9a**)

R = $\text{CH}_2\text{CH}_2\text{CH}_2\text{NH}_2\text{TFA}$

```

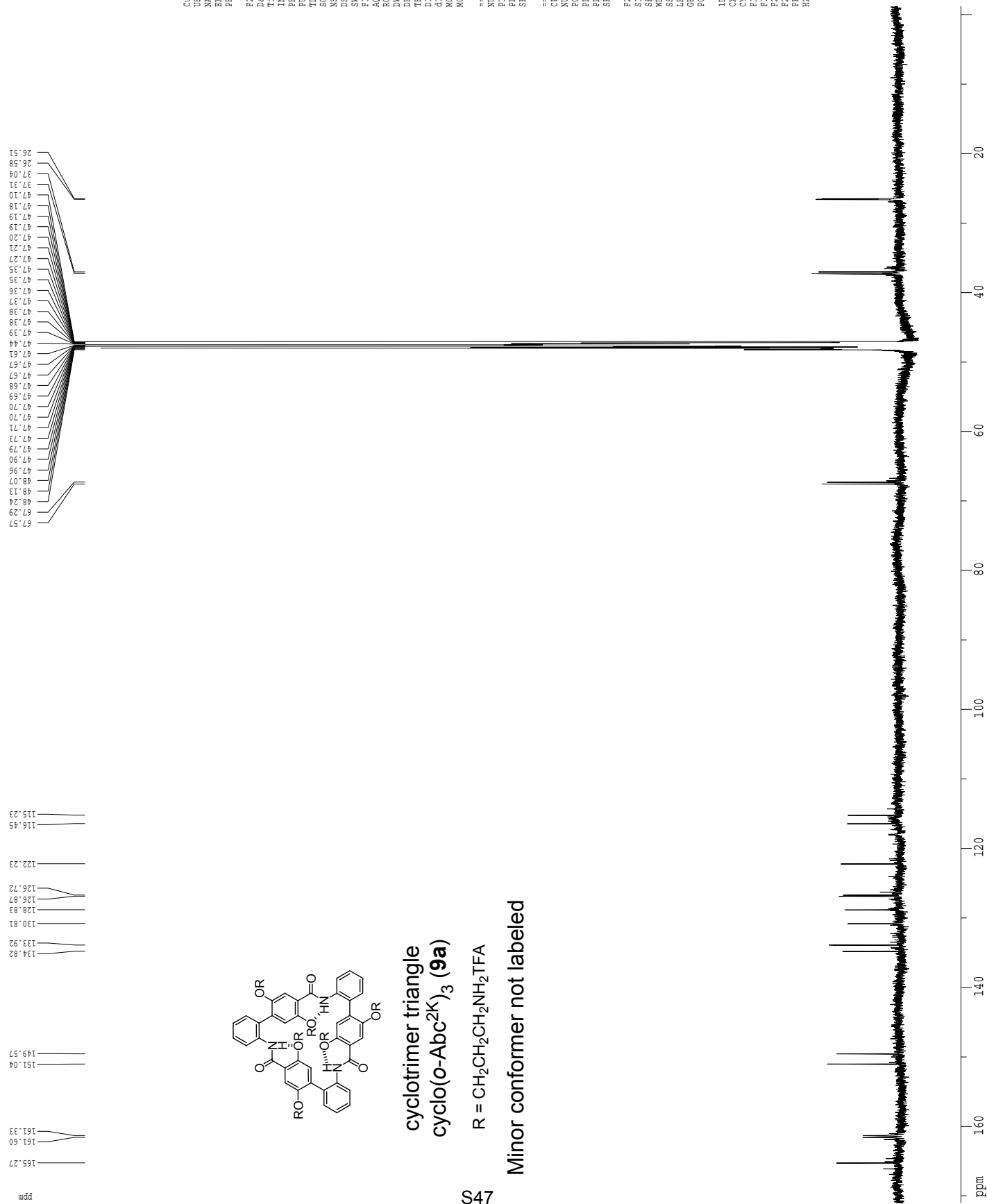
Current Data Parameters
USER          cgccha
NAME         c9-11-44
EXPNO        2
PROCNO       1

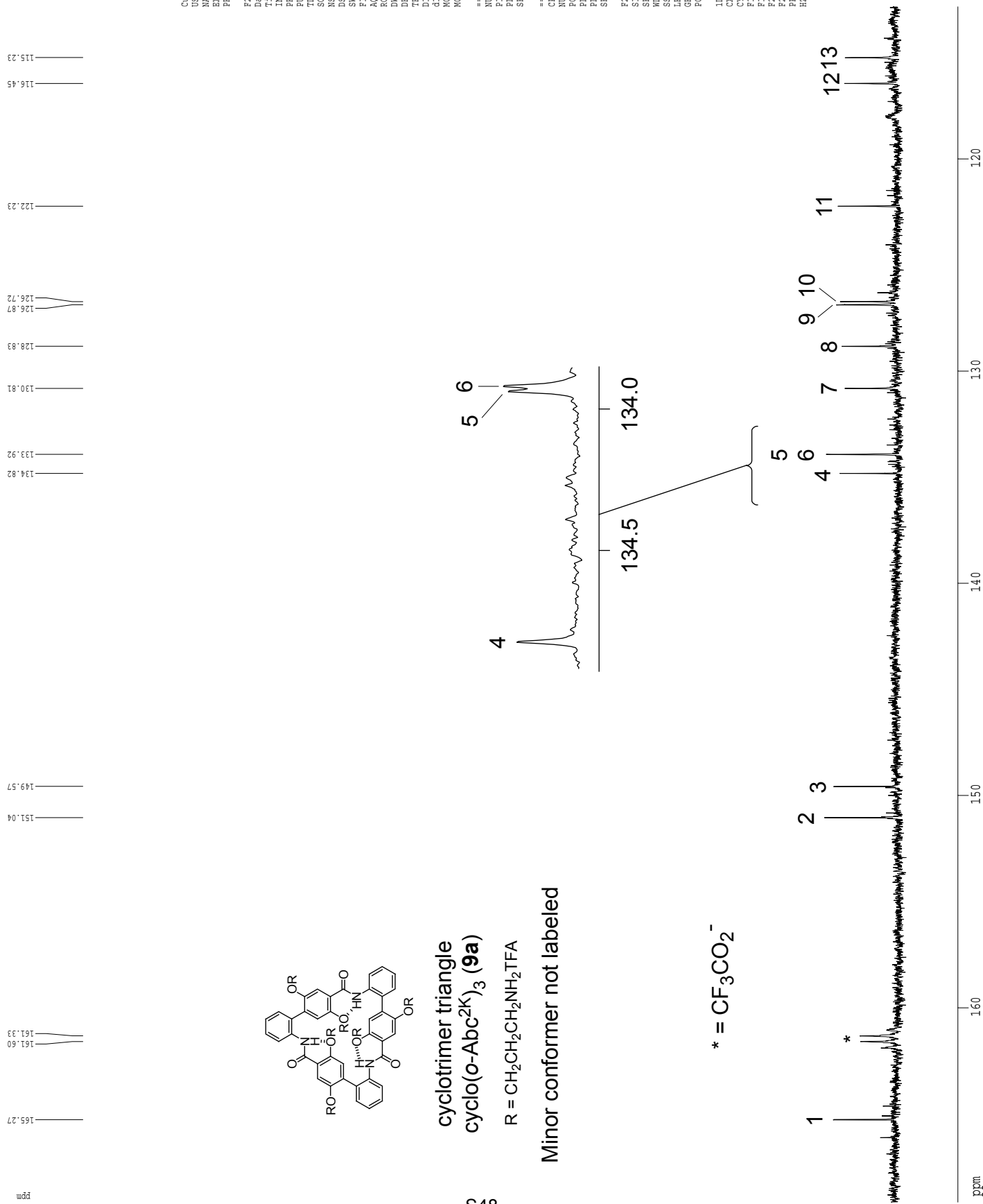
F2 - Acquisition Parameters
Date_        20070306
Time_       17.00
INSTRUM     crys500
PROBHD      5 mm CPYCI 1H-
PULPROG     zg30
TD          81728
SOLVENT     CD3OD
NS          64
DS          2
SWH         8012.820 Hz
FIDRES     0.096043 Hz
AQ         5.0956774 sec
RG          16.00
DB          62.40
DE         6.00 usec
TE         298.0 K
D1         0.10000000 sec
MCREST     0.00000000 sec
MORRK      0.01500000 sec

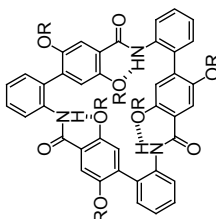
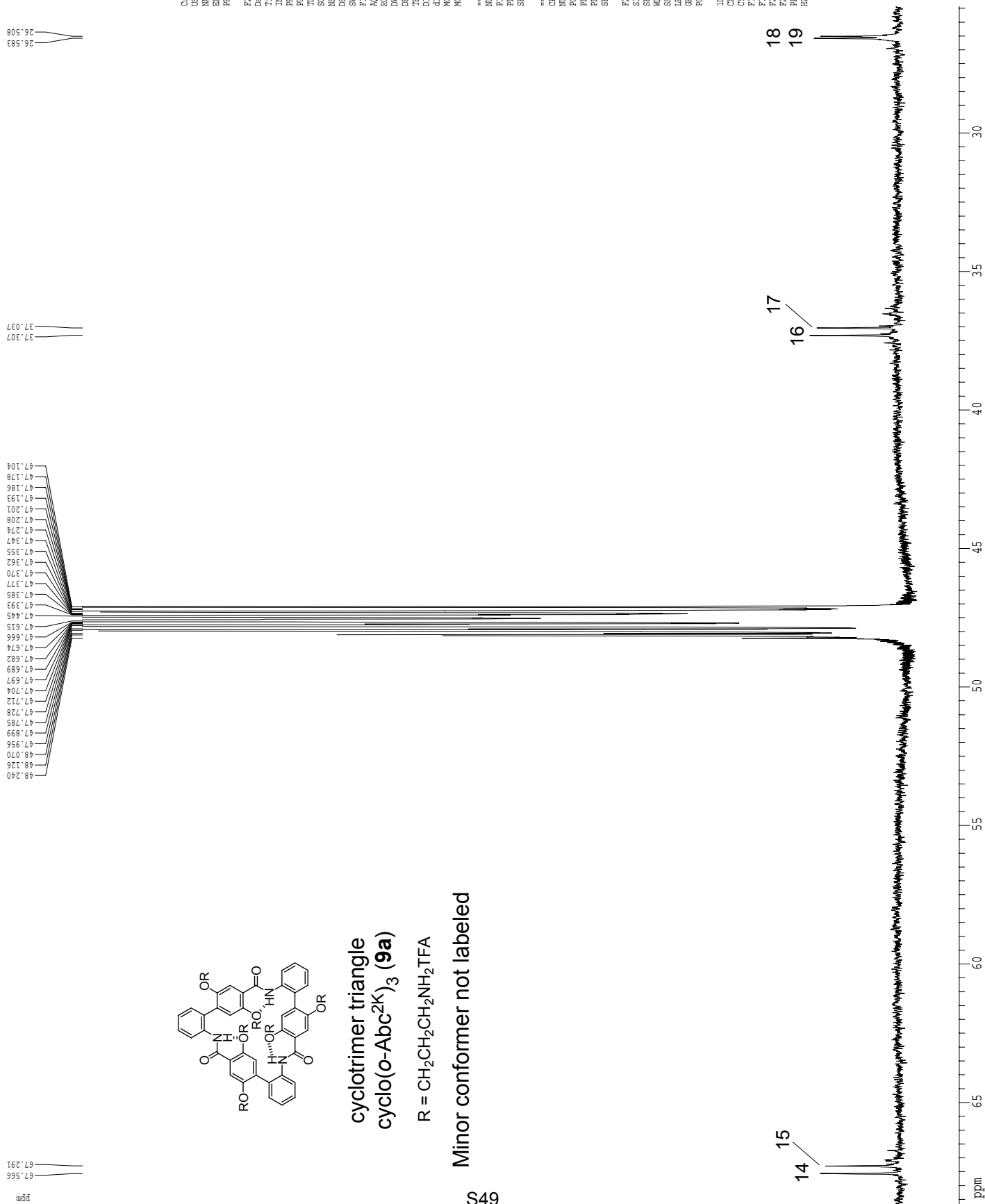
===== CHANNEL f1 =====
NUC1        1H
P1          8.00 usec
PL1         1.60 dB
SFO1        500.2235015 MHz

F2 - Processing parameters
SI          65536
SF          500.2200040 MHz
WDW         EM
SSB         0
LB         0.30 Hz
GB         0
PC         4.00

1D NMR plot parameters
CX         22.80 cm
CY         2257.79 cm
FLP        4.454 ppm
F1P        22.4748 Hz
F2P        743.10 Hz
PRN1CM     0.131195 ppm/cm
HZ1CM      66.000397 Hz/cm
  
```

^{13}C NMR (125 MHz, 298 K, CD_3OD) spectrum of cyclotrimer triangle **9a**

^{13}C NMR (125 MHz, 298 K, CD_3OD) spectrum of cyclotrimer triangle **9a**

¹³C NMR (125 MHz, 298 K, CD₃OD) spectrum of cyclotrimer triangle **9a**

**cyclotrimer triangle
cyclo(o-Abc²K)₃ (**9a**)**

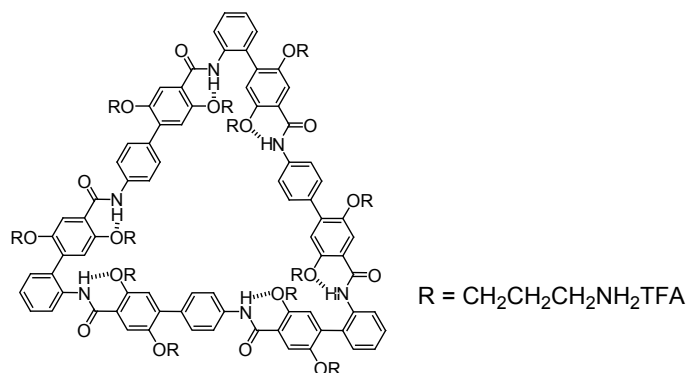
R = CH₂CH₂CH₂NH₂TFA

Minor conformer not labeled

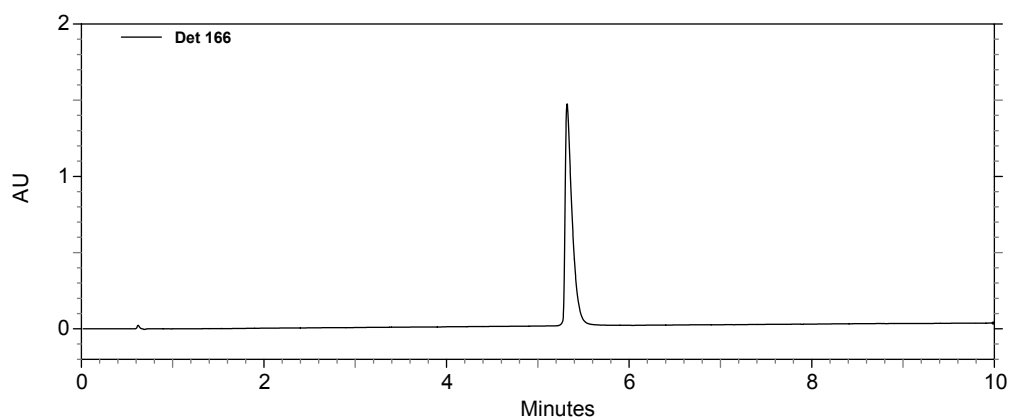
```

Current Data Parameters
USER          csyocia
NAME         c9-11-44_C13
EXPNO        4
PROCNO       1
F2 - Acquisition Parameters
Date_        20070309
Time_        0.36
INSTRUM      cryo500
PROBHD       5 mm CPTCI 1H-
PULPROG      zgpg30
TD           65418
SOLVENT      CD3OD
NS           19000
DS           4
SWH          30203.031 Hz
FIDRES       0.462322 Hz
AQ           1.0794470 sec
RG           6132
RW           16.500 usec
DE           0.00 usec
TE           298.2 K
D1           0.25000000 sec
d11          0.03000000 sec
MGREEST      0.00000000 sec
MCOREK       0.01500000 sec
===== CHANNEL f1 =====
NUC1         13C
P1           15.00 usec
PL1          -1.00 dB
SFO1         125.7942548 MHz
===== CHANNEL f2 =====
CPDPRG2      waltz16
NUC2         1H
PCPD2        100.00 usec
PL2          1.60 dB
PL12         2.54 dB
SFO2         500.2225011 MHz
F2 - Processing parameters
SI           65536
SF           125.7804190 MHz
WDW          EM
SSB          0
LB           1.00 Hz
GB           0
PC           2.00
ID NMR plot parameters
CX           22.80 cm
CY           737.01 cm
FLP          68.664 ppm
F1           8626.53 Hz
F2           325.428 ppm
F3           1.89584 Hz
FREQM        238.46011 Hz/cm
HZCM
  
```

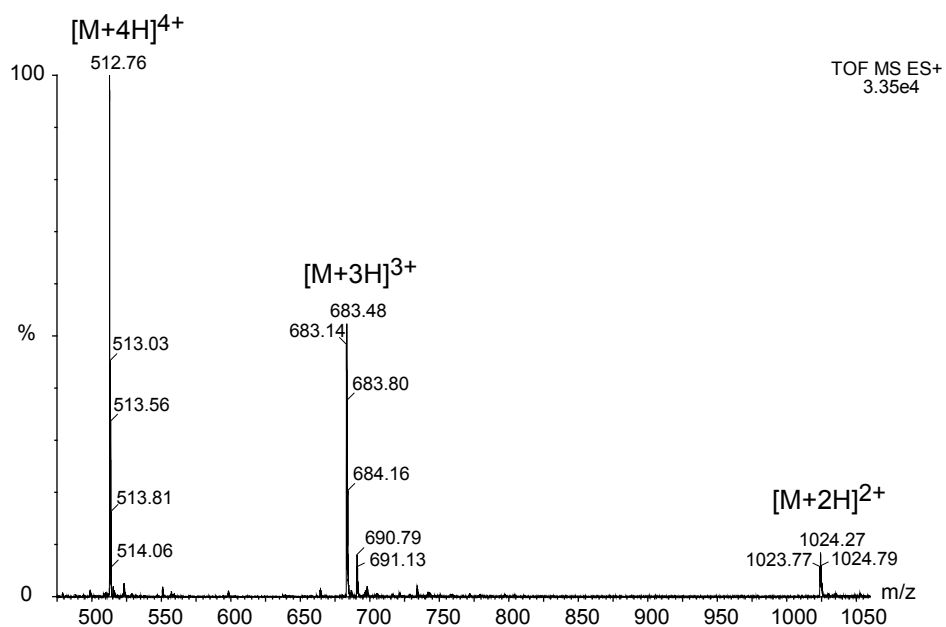
cyclohexamer triangle: (*o*-Abc^{2K}-*p*-Abc^{2K})₃ (**9b**)
Analytical RP-HPLC chromatograph and mass spectrum (ESI-MS)



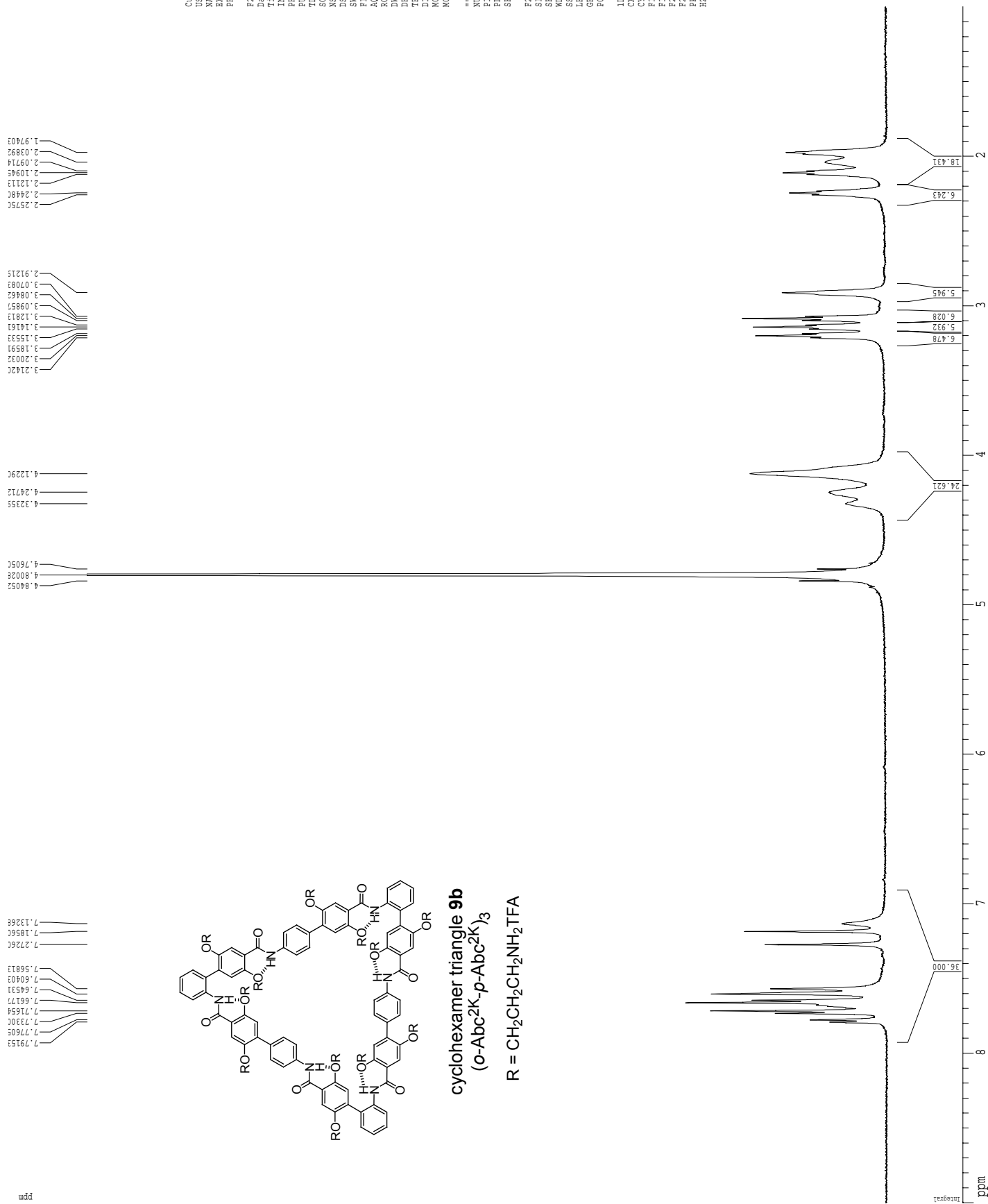
(a) Analytical RP-HPLC (5-50% acetonitrile with 0.1% TFA over 10 min, $\lambda = 214$)

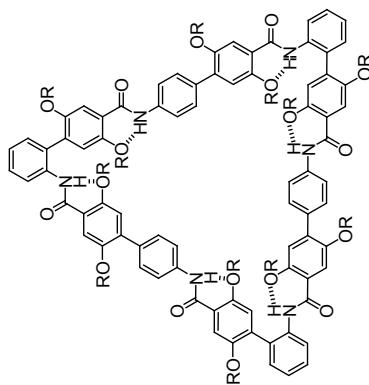


(b) ESI Mass spectrum. (Calcd exact mass for C₁₁₄H₁₃₈N₁₈O₁₈ [M] = 2047.04)



¹H NMR (500 MHz, 298 K, D₂O) spectrum of cyclohexamer triangle **9b**



^1H NMR (500 MHz, 298 K, D_2O) spectrum of cyclohexamer triangle **9b** (aromatic region)cyclohexamer triangle **9b**
(*o*-Abc²K-*p*-Abc²K)₃R = $\text{CH}_2\text{CH}_2\text{CH}_2\text{NH}_2\text{TFA}$

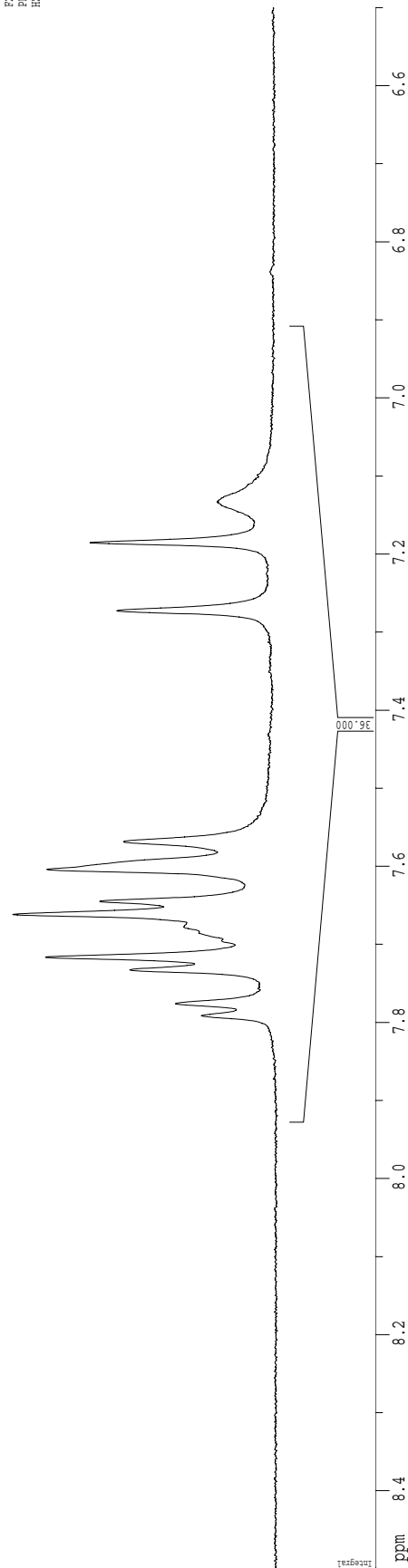
```

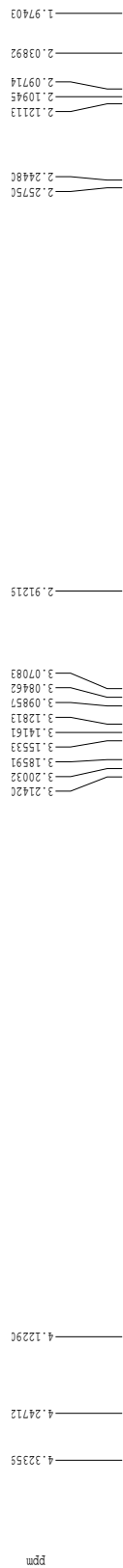
Current Data Parameters
=====
USER      cyotba
NAME      c9-11-31
EXPNO     1
PROCNO    1

F2 - Acquisition Parameters
=====
Date_     20070325
Time      17:14
INSTRUM   spect
PROBHD    5 mm broadband
PULPROG   zgpg30
TD         65536
SOLVENT   D2O
NS         64
DS         2
SWH        8012.820 Hz
FIDRES     0.09043 Hz
AQ         5.0989774 sec
RG         812.7
WB         62.400 usec
DE         5.00 usec
TE         300.2 K
D1         0.10000000 sec
d11        0.00000000 sec
d12        0.00000000 sec
d13        0.00000000 sec
d14        0.00000000 sec
d15        0.01500000 sec
===== CHANNEL f1 =====
NUC1       1H
P1         12.00 usec
PL1        -3.00 dB
SFO1       499.9334995 MHz

F2 - Processing parameters
=====
SI         65536
SF         499.930035 MHz
WDW        EM
SSB        0
LB         0.30 Hz
GB         0
PC         4.00

1D NMR plot parameters
=====
CX         22.80 cm
CY         119.33 cm
F1P        6.500 PPM
F2P        4249.40 Hz
F3P        6.500 PPM
F4P        3289.54 Hz
NUC1       1H
NUC2       13C
HZCN       43.89551 Hz/cm
  
```



^1H NMR (500 MHz, 298 K, D_2O) spectrum of cyclohexamer triangle **9b** (aliphatic region)

```

Current Data Parameters
=====
USER          cyotba
NAME          c9-11-31
EXPNO        1
PROCNO       1

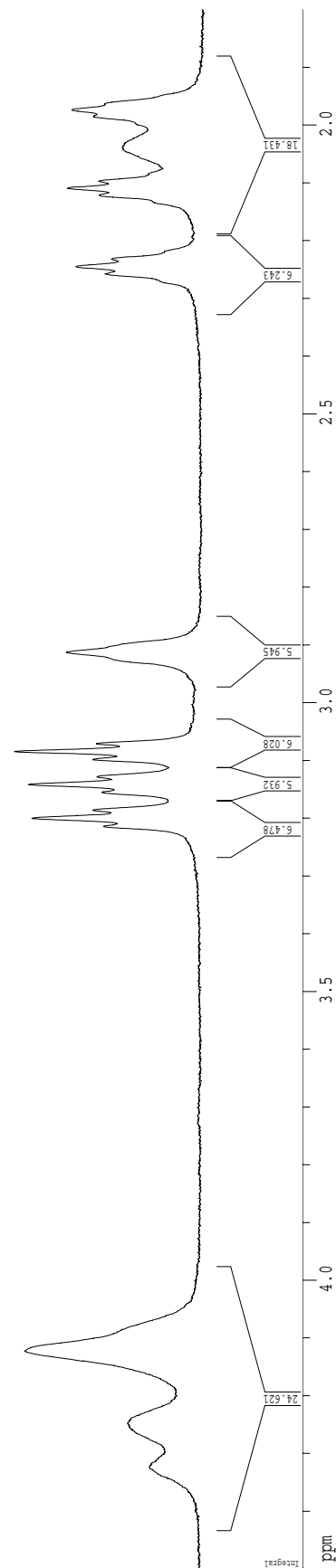
F2 - Acquisition Parameters
=====
Date_        20070325
Time        17:54
INSTRUM     spect
PROBHD      5 mm broadband
PULPROG     zgpg30
TD          65536
SOLVENT     D2O
NS          64
DS          2
SWH         8012.820 Hz
FIDRES     0.09643 Hz
AQ         5.0989774 sec
RG         612.7
GB         6.00 usec
DE         6.00 usec
TE         300.2 K
D1         0.10000000 sec
d11        0.00000000 sec
d12        0.00000000 sec
d13        0.00000000 sec
d14        0.01500000 sec
===== CHANNEL f1 =====
NUC1        1H
P1          12.00 usec
PL1         -3.00 dB
SFO1        499.9334995 MHz

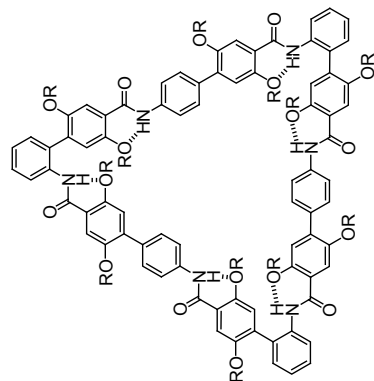
F2 - Processing parameters
=====
SI          65536
SF          499.9300038 MHz
WDW         EM
SSB         0
LB          0.30 Hz
GB          0
PC          4.00

1D NMR plot parameters
=====
CX          22.80 cm
CY          119.33 cm
F1P         4.500 PPM
F2P         2249.69 Hz
F3P         1.800 PPM
F4P         899.87 Hz
F5P         0.11024 PPM/cm
HZCN        59.20224 Hz/cm
  
```

cyclohexamer triangle **9b**
(*o*-Abc²K-*p*-Abc²K)₃

R = $\text{CH}_2\text{CH}_2\text{CH}_2\text{NH}_2\text{TFA}$



¹³C NMR (125 MHz, 320 K, D₂O) spectrum of cyclohexamer triangle **9b**

cyclohexamer triangle **9b**
(*o*-Abc²K-*p*-Abc²K)₃

R = CH₂CH₂CH₂NH₂TFA

```

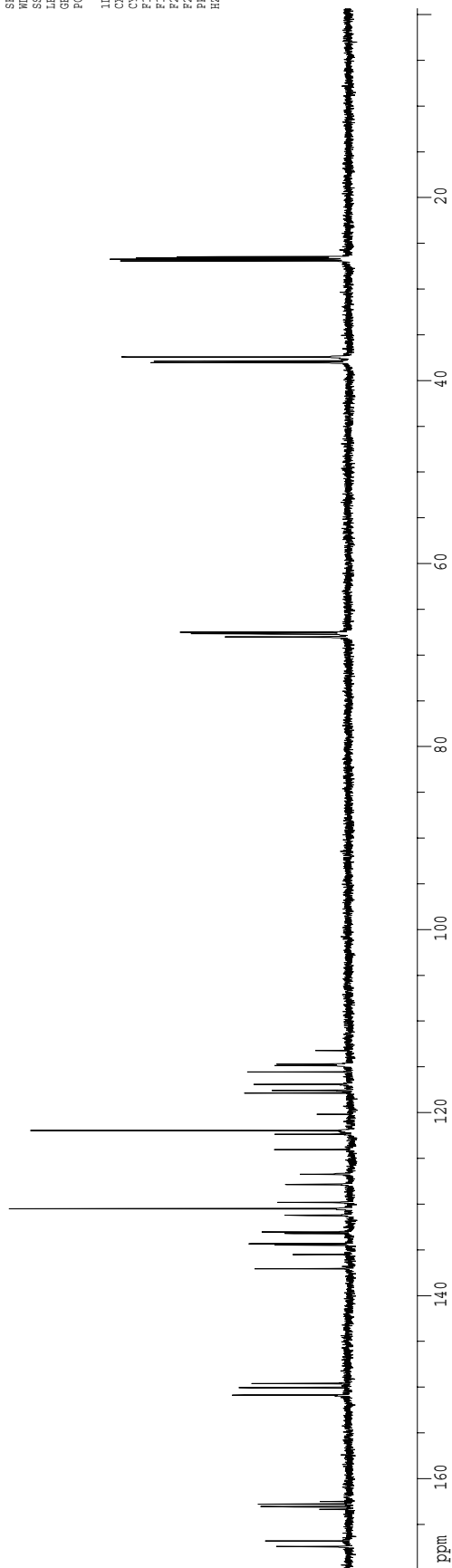
Current Data Parameters
USBR          csodia
NAME          09-11-76
PULPROG      zgpg30
PROCNO       4
PROBHD       5 mm QNP1H-
PULPROG      zgpg30
TD           65418
SOLVENT      D2O
NS           12190
DS           4
SWH          30303.031 Hz
FIDRES       0.463222 Hz
AQ           1.0794470 sec
RG           18390.4
DM           16.500 usec
DE           6.00 usec
TE           300.2 K
D1           0.25000000 sec
d11          0.03000000 sec
NOFEST       0.00000000 sec
NOVERK       0.01500000 sec

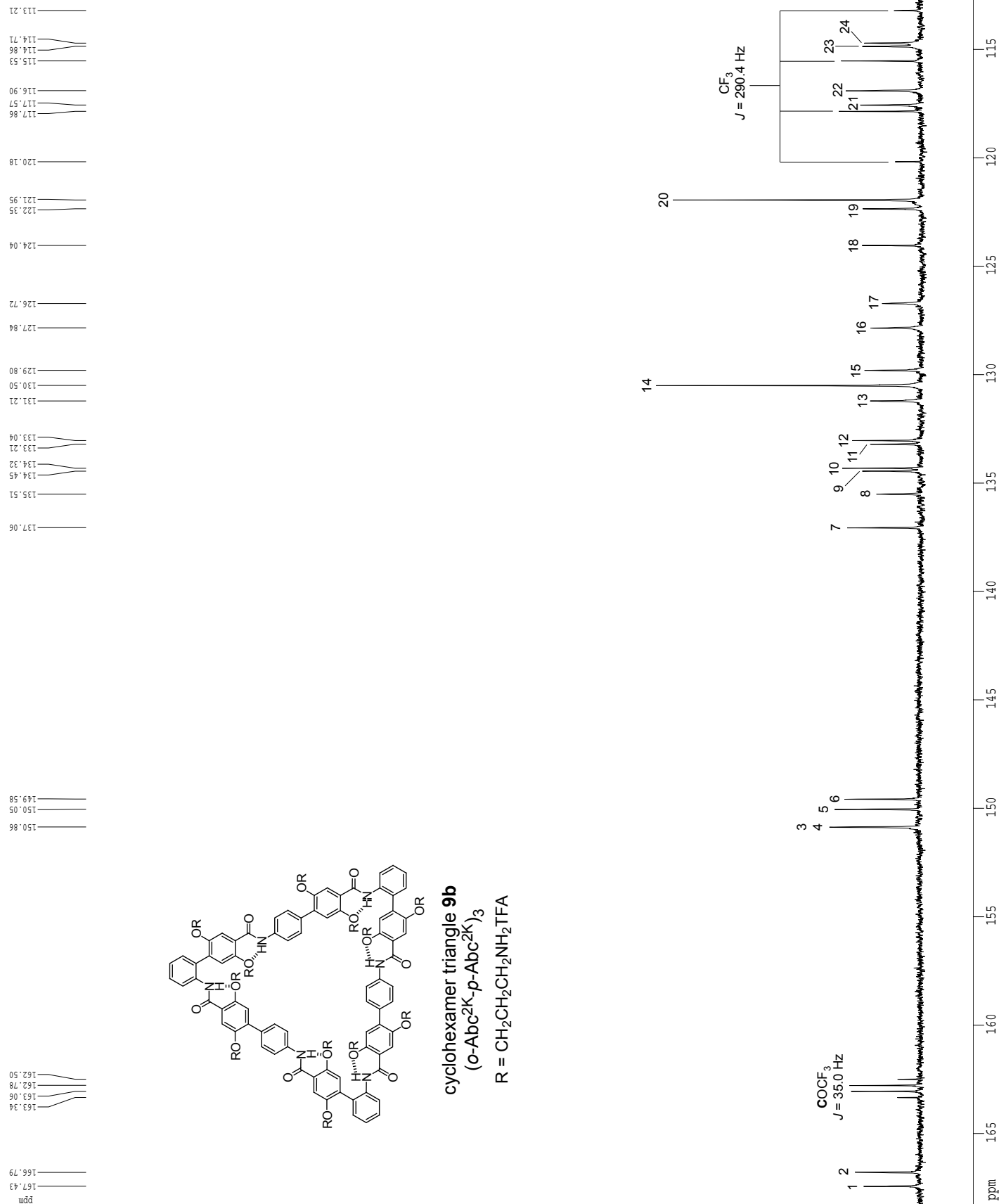
===== CHANNEL F1 =====
NUC1         13C
P1           15.00 usec
PL1          -1.00 dB
SFO1         125.7942548 MHz

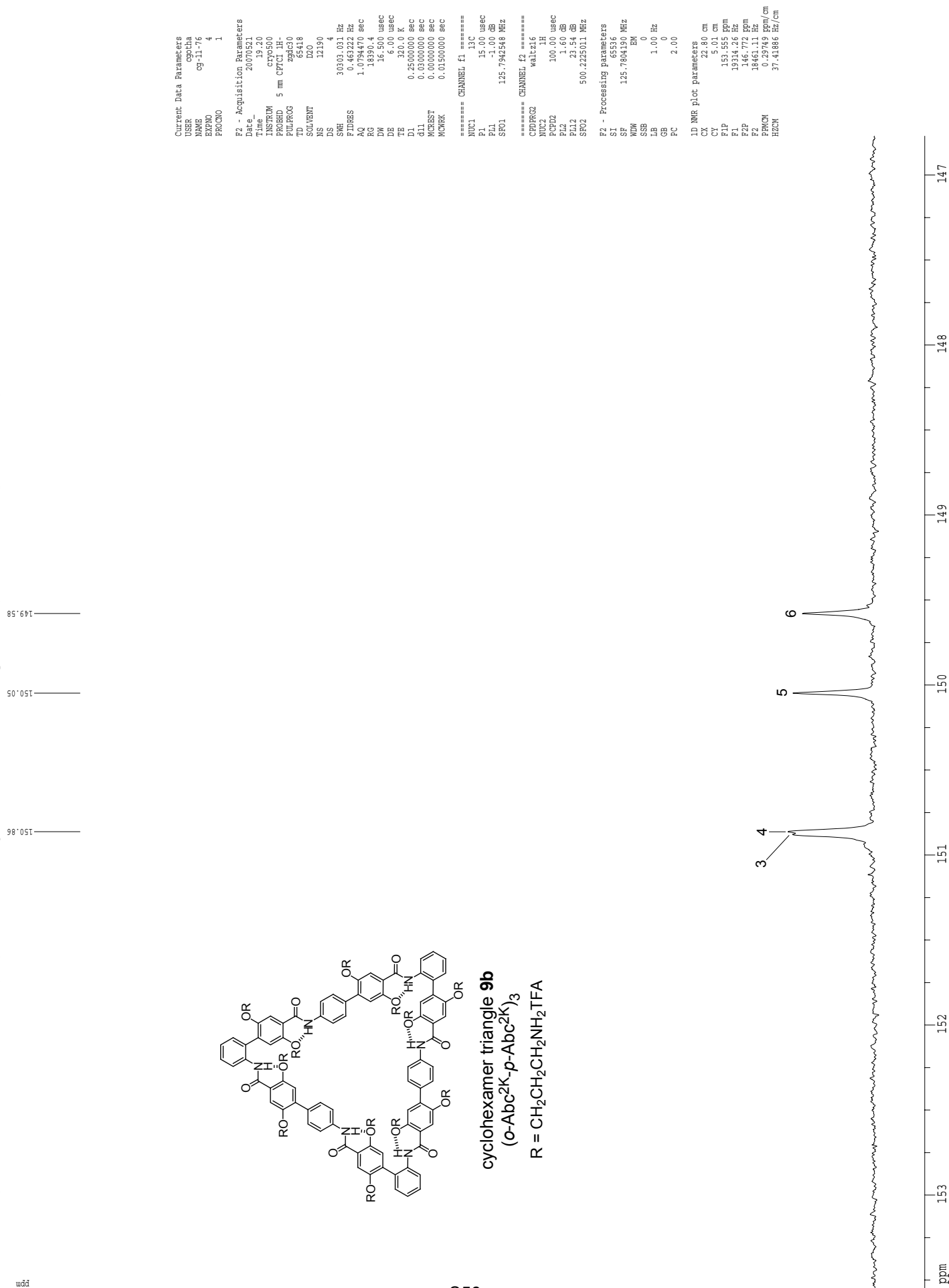
===== CHANNEL E2 =====
CPDPRG2      waltz16
NUC2         1H
PCPD2       100.00 usec
PL2          1.60 dB
PL12         21.54 dB
SFO2         500.2225011 MHz

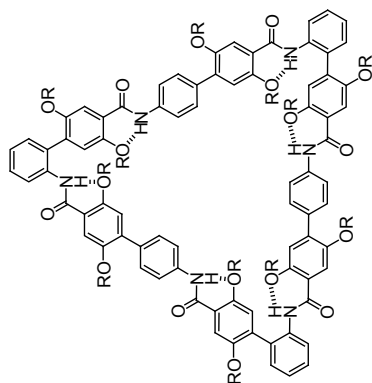
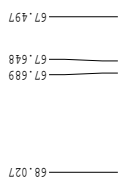
F2 - Processing parameters
SI           65836
SF           125.7804190 MHz
WDW          EM
SSB          0
LB           1.00 Hz
GB           0
PC           2.00

1D NMR plot parameters
CX           22.80 cm
CY           5.01 cm
FIP         169.978 ppm
FL          21379.91 Hz
F2P         -0.652 ppm
F3P         7.948 Hz
F4P(CH)     7.4638 ppm/cm
HZCN        941.20026 Hz/cm
  
```



¹³C NMR (125 MHz, 320 K, D₂O) spectrum of cyclohexamer triangle **9b** (aromatic and carbonyl region)

^{13}C NMR (125 MHz, 320 K, D_2O) spectrum of cyclohexamer triangle **9b** (aromatic expansion 1)

^{13}C NMR (125 MHz, 320 K, D_2O) spectrum of cyclohexamer triangle **9b** (aliphatic region expansion 1)cyclohexamer triangle **9b**
(*o*-Abc²K-*p*-Abc²K)₃R = $\text{CH}_2\text{CH}_2\text{CH}_2\text{NH}_2$ TFA

```

Current Data Parameters
USBR          csobda
NAME          09-11-76
PULPROG      zgpg30
PROCNO       4
PROBHD       5 mm CPTCI 1H-
PULPROG      zgpg30
TD           65418
SOLVENT      D2O
NS           12190
DS           4
SWH          30303.031 Hz
FIDRES       0.463222 Hz
AQ           1.0794470 sec
RG           18390.4
DM           16.500 usec
DE           6.00 usec
TE           300.2 K
D1           0.25000000 sec
d11          0.03000000 sec
d111         0.03000000 sec
d1111        0.00000000 sec
NOVERK       0.01500000 sec

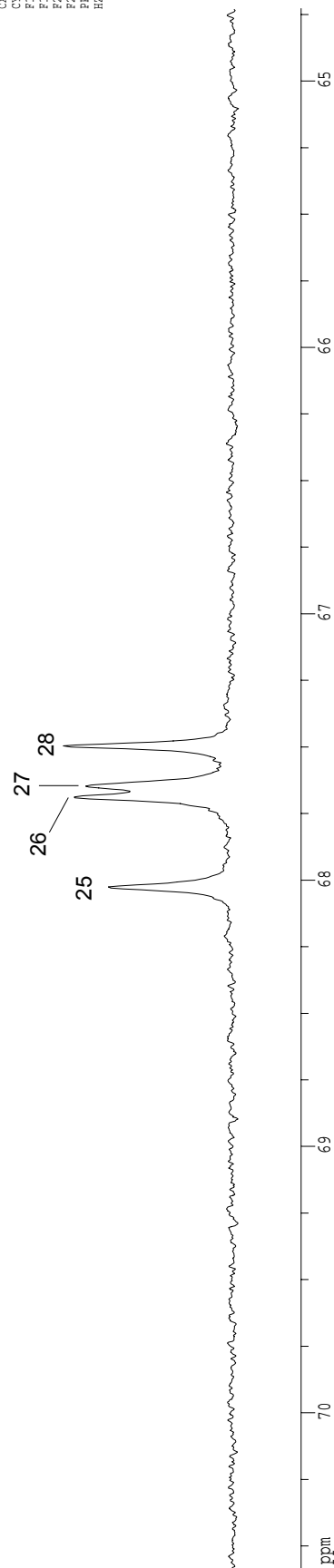
===== CHANNEL F1 =====
NUC1         13C
P1           15.00 usec
PL1          -1.00 dB
SFO1         125.7942548 MHz

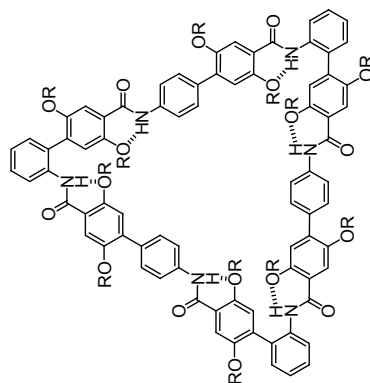
===== CHANNEL E2 =====
CPDPRG2      waltz16
NUC2         1H
PCPD2       100.00 usec
PL2          1.60 dB
PL12         23.54 dB
SFO2         500.2225011 MHz

F2 - Processing parameters
SI           658336
SF           125.7804190 MHz
WDW          EM
SSB          0
LB           1.00 Hz
GB           0
PC           2.00

1D NMR plot parameters
CX           22.80 cm
CY           5.01 cm
FIP          70.597 ppm
FL           8979.66 Hz
F2P          64.728 ppm
F3P          0.3456 Hz
F4P          0.3456 Hz
FREQCN       32.37277 Hz/cm
HZCN         32.37277 Hz/cm

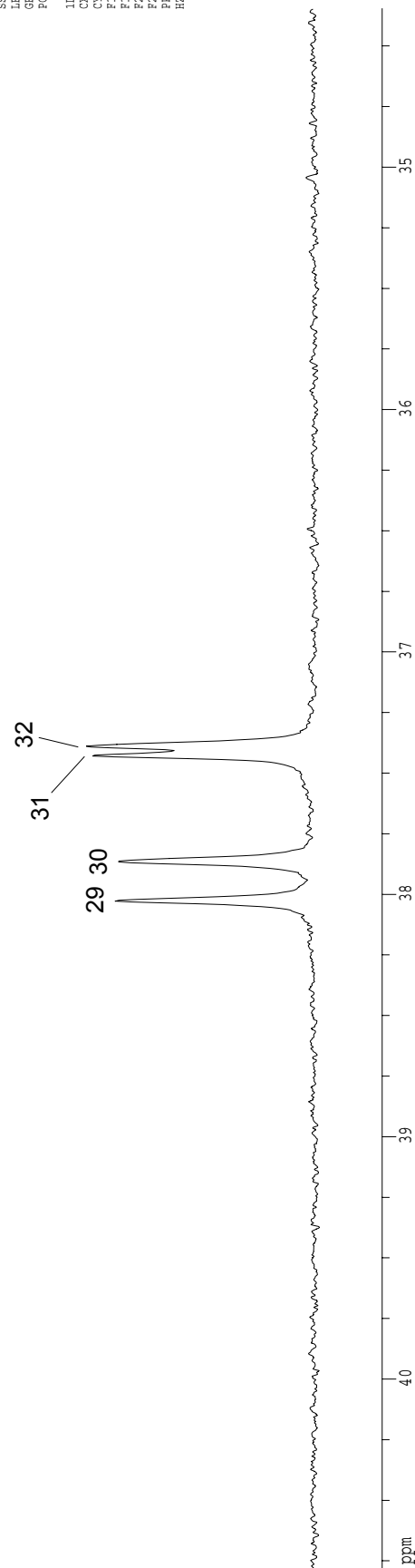
```



¹³C NMR (125 MHz, 320 K, D₂O) spectrum of cyclohexamer triangle **9b** (aliphatic region expansion 2)cyclohexamer triangle **9b**
(*o*-Abc²K-*p*-Abc²K)₃R = CH₂CH₂CH₂NH₂TFA

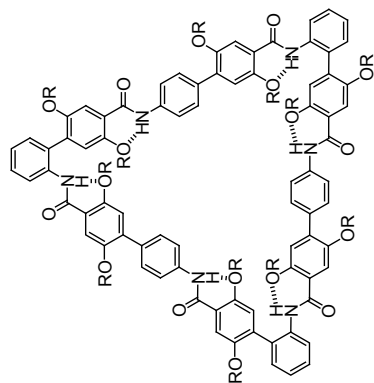
```

Current Data Parameters
USBR          csodia
NAME          09-11-76
PULPROG      4
PROCNO       1
F2 - Acquisition Parameters
Date_         20070521
Time_        19.20
INSTRUM      cryo500
PROBHD       5 mm CPTCI 1H-
PULPROG      zgpg30
TD           65418
SOLVENT      D2O
NS           12190
DS           4
SWH          30303.031 Hz
FIDRES       0.463222 Hz
AQ           1.0794470 sec
RG           18390.4
DM           16.500 usec
DE           6.00 usec
TE           300.2 K
D1           0.25500000 sec
d11          0.03000000 sec
NOEPRST      0.00000000 sec
NOEPRG       0.01500000 sec
===== CHANNEL F1 =====
NUC1         13C
P1           15.00 usec
PL1          -1.00 dB
SFO1         125.7942548 MHz
===== CHANNEL E2 =====
CPDPRG2      waltz16
NUC2         1H
PCPD2       100.00 usec
PL2          1.60 dB
PL12         21.54 dB
SFO2         500.2225011 MHz
F2 - Processing parameters
SI           65836
SF           125.7804190 MHz
WDW          EM
SSB          0
LB           1.00 Hz
GB           0
PC           2.00
1D NMR plot parameters
CX           22.80 cm
CY           5.01 cm
FIP          40.795 ppm
FL           5131.25 Hz
F2P          34.347 ppm
F4P          4320.15 Hz
NUC1CN       0.92323 ppm/cm
NUC2CN       35.15456 Hz/cm
  
```



¹³C NMR (125 MHz, 320 K, D₂O) spectrum of cyclohexamer triangle **9b** (aliphatic region expansion 2)

26.914
26.731
26.591
26.466



cyclohexamer triangle **9b**
(*o*-Abc²K-*p*-Abc²K)₃

R = CH₂CH₂CH₂NH₂TFA

```

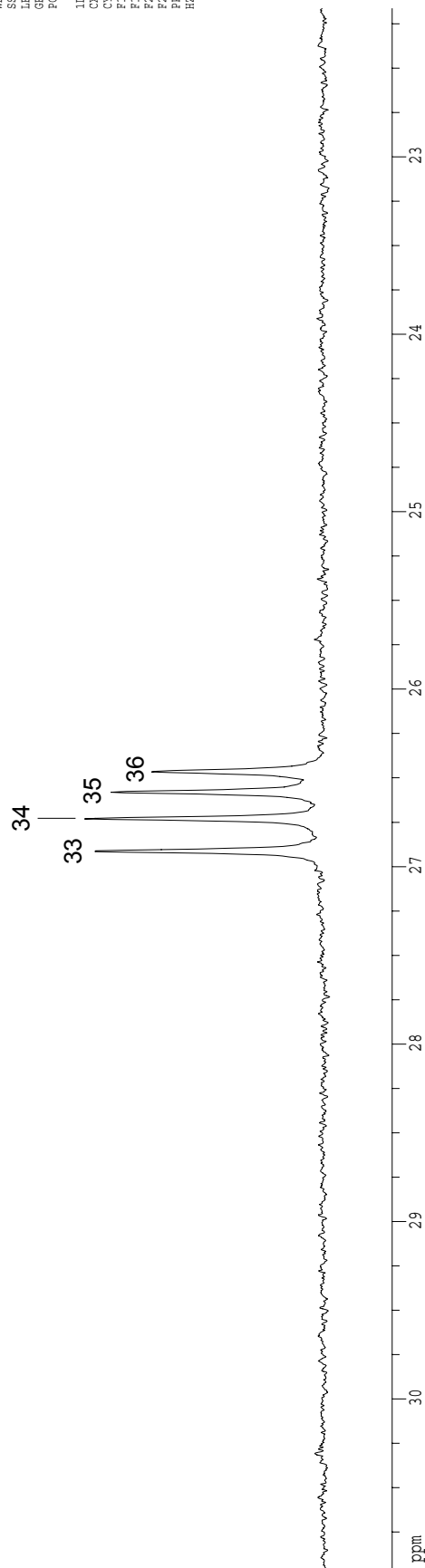
Current Data Parameters
USBR          csodia
NAME          c9-11-76
PULPROG      zgpg30
PROCNO       4
PROBHD       5 mm CPTCI 1H-
PULPROG      zgpg30
TD           65418
SOLVENT      D2O
NS           12190
DS           4
SWH          30303.031 Hz
FIDRES       0.463222 Hz
AQ           1.0794470 sec
RG           18390.4
DM           16.500 usec
DE           6.00 usec
TE           300.2 K
D1           0.25500000 sec
d11          0.03000000 sec
NOFEST       0.00000000 sec
NOWEAK       0.01500000 sec

===== CHANNEL F1 =====
NUC1         13C
P1           15.00 usec
PL1          -1.00 dB
SFO1         125.7942548 MHz

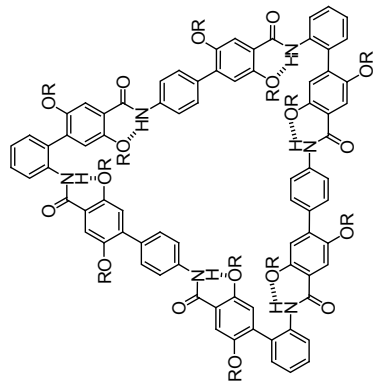
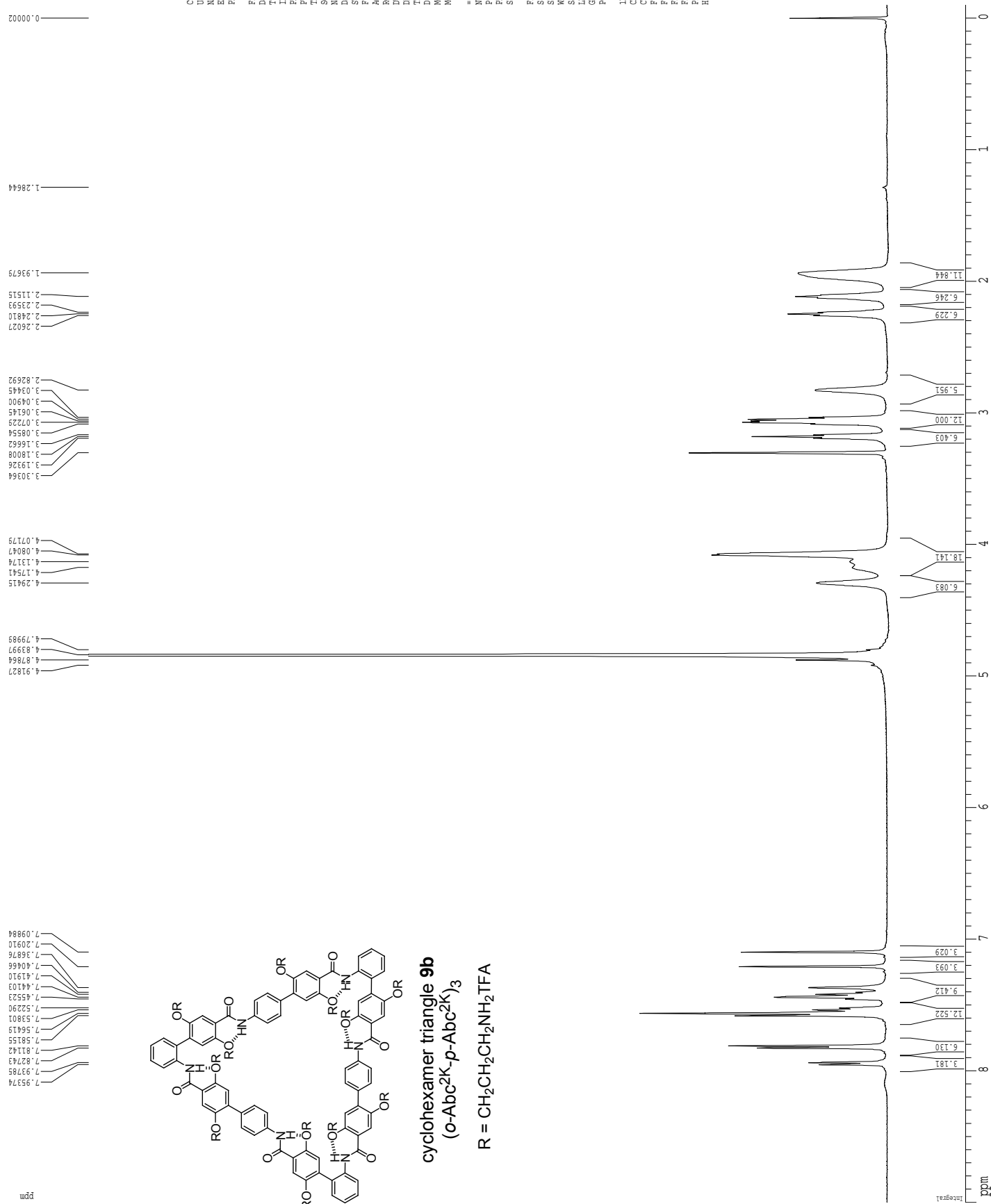
===== CHANNEL E2 =====
CPDPRG2      waltz16
NUC2         1H
PCPD2       100.00 usec
PL2          1.60 dB
PL12        21.54 dB
SFO2         500.2225011 MHz

F2 - Processing parameters
SI           65836
SF           125.7804190 MHz
WDW          EM
SSB          0
LB           1.00 Hz
GB           0
PC           2.00

1D NMR plot parameters
CX           22.80 cm
CY           5.01 cm
FIP          30.964 ppm
FL           3894.62 Hz
F2P          22.165 ppm
F3P          27.878 Hz
SFO(MHz)     0.68652
HZ(CM)       48.54131 Hz/cm
  
```



¹H NMR (500 MHz, 298 K, CD₃OD) spectrum of cyclohexamer triangle **9b**



cyclohexamer triangle **9b**
(*o*-Abc²K-*p*-Abc²K)₃

R = CH₂CH₂CH₂NH₂TFA

Supporting Information

Current Data Parameters
 USR egcda
 EGNO eg-11-3 7
 EXNO 1
 PRGNO 1

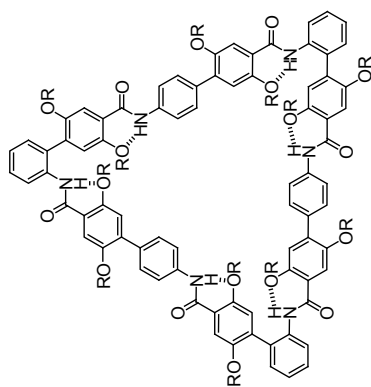
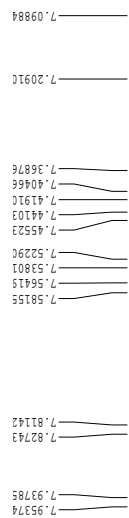
F2 - Acquisition Parameters
 Date 20071031
 Time 18.11
 INSTRUM gms500
 PROBHD 5 mm broadband
 PULPROG zg30
 TD 81728
 SOLVENT CD3OD
 NS 16
 DS 2
 SWH 8012.876 Hz
 FIDRES 0.098843 Hz
 RES 5.0988774 sec
 AQ 161.3
 RM 62.400 usec
 DE 6.00 usec
 TE 298.0 K
 D1 0.10000000 sec
 MCREST 0.00000000 sec
 MCWRR 0.01500000 sec

===== CHANNEL F1 =====
 NUC1 1H
 JH 12.00 usec
 FA1 3.00 dB
 SFO1 499.828488 MHz

F2 - Processing parameters
 SI 65536
 SF 499.8250210 MHz
 MDW EN
 SSB 0
 LB 0.30 Hz
 GB 0
 PC 4.00

1D NMR Plot parameters
 XZ 2.00 cm
 CY 2.00 cm
 CZ 2.00 cm
 F1P 9.000 DDM
 F1 4498.43 Hz
 F2P -0.100 DDM
 F2 -49.98 Hz
 PPMON 0.39912 PPM
 HZ/CN 199.49158 Hz/cm

C. M. Gothard and J. S. Nowick

^1H NMR (500 MHz, 298 K, CD_3OD) spectrum of cyclohexamer triangle **9b** (aromatic region)cyclohexamer triangle **9b**
(*o*-Abc 2 K-*p*-Abc 2 K) $_3$ R = $\text{CH}_2\text{CH}_2\text{CH}_2\text{NH}_2\text{TFA}$

```

Current Data Parameters
USER      cyobha
NAME      c9-11-31
EXPNO     7
PROCNO    1

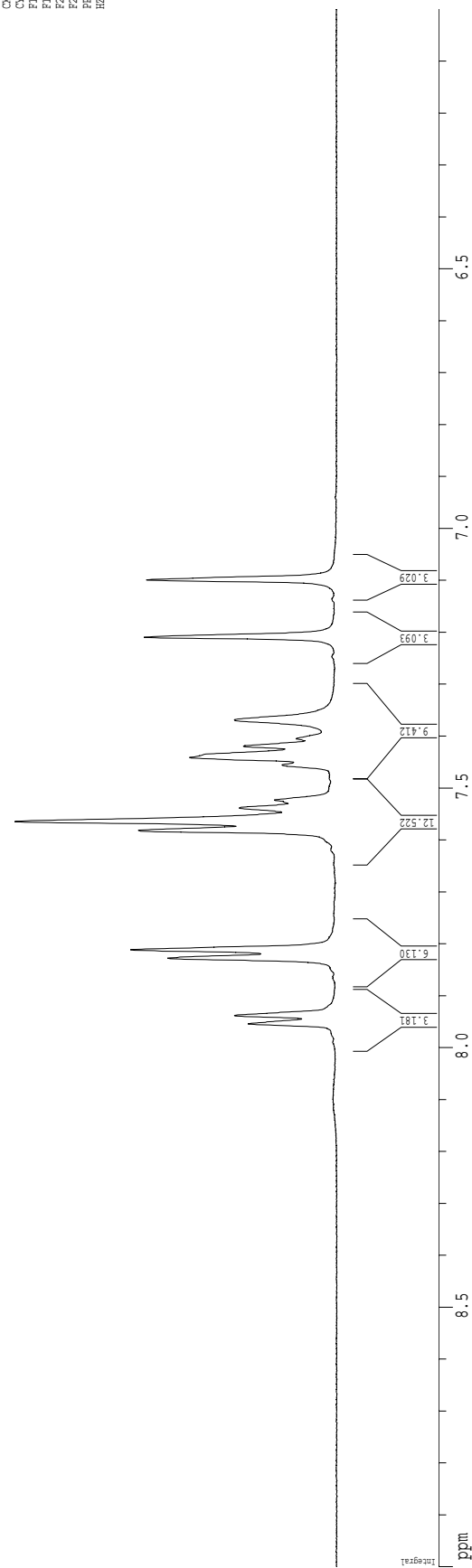
F2 - Acquisition Parameters
Date_     20071031
Time      18.11
INSTRUM   gp50
PROBHD    5 mm broadband
PULPROG   zg30
TD         81728
SOLVENT   CD3OD
NS         16
DS         2
SWH        8012.820 Hz
FIDRES     0.098943 Hz
AQ         5.093674 sec
RG          631
DM          62.400 usec
DE          6.000 usec
TE          298.0 K
D1          0.10000000 sec
MCHREST    0.00000000 sec
MCWREK     0.01500000 sec

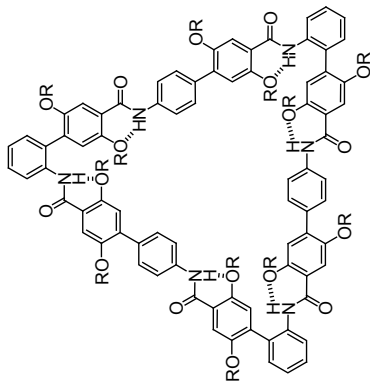
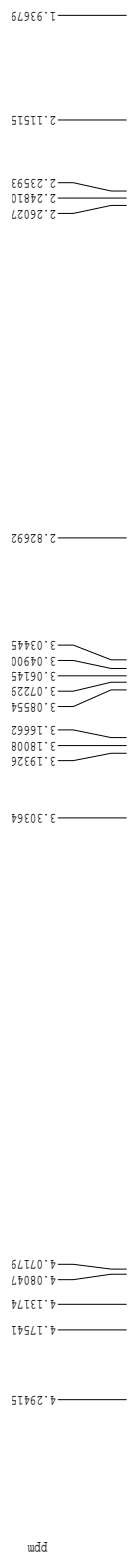
***** CHANNEL f1 *****
NUC1       1H
P1         12.00 usec
PL1        -2.00 dB
SFO1       499.8284968 MHz

F2 - Processing parameters
SI         65536
SF         499.8250210 MHz
WDW        EM
SSB        0
LB         0.30 Hz
GB         0
PC         4.00

ID NMR plot parameters
CX         22.80 cm
CT         51.60 cm
EI         0.00 ppm
F1P        4486.40 ppm
F2P        6.000 ppm
FZ         2998.95 Hz
PPM10M     0.13158 ppm/cm
HZ10M      65.76645 Hz/cm

```



¹H NMR (500 MHz, 298 K, CD₃OD) spectrum of cyclohexamer triangle **9b** (aliphatic region)

S9

cyclohexamer triangle **9b**
(*o*-Abc²K₃-*p*-Abc²K₃)₃

R = CH₂CH₂CH₂NH₂·TFA

```

Current Data Parameters
=====
USER      csocia
NAME      cg-11-31
EXPNO     1
PROCNO    1

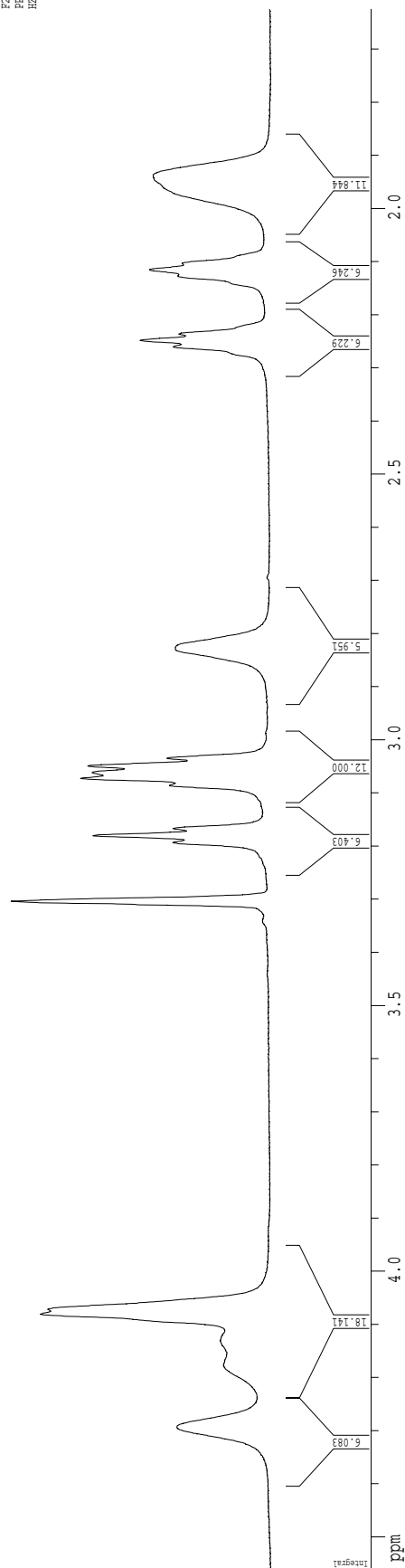
F2 - Acquisition Parameters
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Date_     20071031
Time      18.11
INSTRUM   gm500
PROBHD    5 mm broadband
PULPROG   zg30
TD         81728
SOLVENT   CD3OD
NS         16
DS         2
SFO1      801.282 Hz
SFO2      0.098623 Hz
SFO3      0.098623 Hz
AQ         5.0398774 sec
RG         161.3
DM         62.400 usec
DE         6.00 usec
TE         298.0 K
D1         0.10000000 sec
MCREST    0.00000000 sec
MCWREK    0.01500000 sec

===== CHANNEL f1 =====
NUC1       1H
P1         12.00 usec
PL1        0.00 dB
SFO1      499.824988 MHz

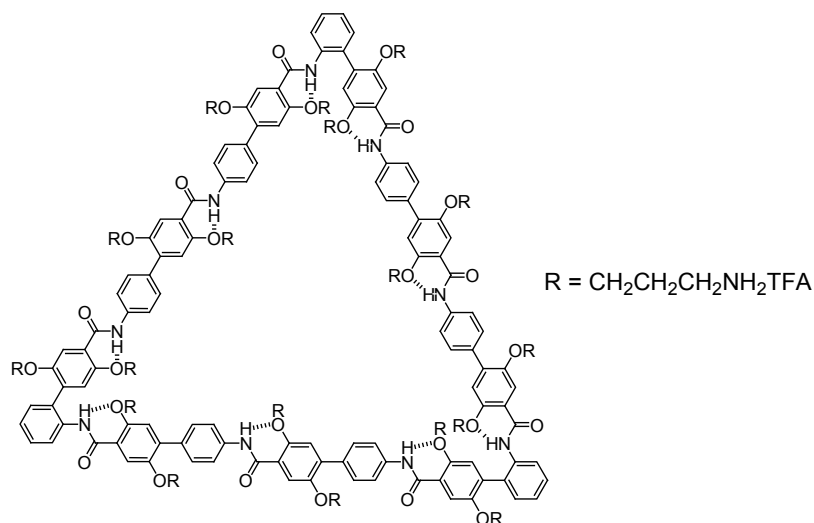
F2 - Processing parameters
=====
SI         65536
SF         499.8250210 MHz
RG         0
EM         0
SSB        0
LB         0.30 Hz
GB         0
CB         0
PC         4.00

ID NRG plot parameters
=====
IX         2.80 cm
CY         2.80 cm
CZ         4.560 cm
FI         2279.36 Hz
F1         1.625 ppm
F2         81.238 Hz
PRGCMN    0.12871 ppm/cm
PRGCMX    64.33254 Hz/cm

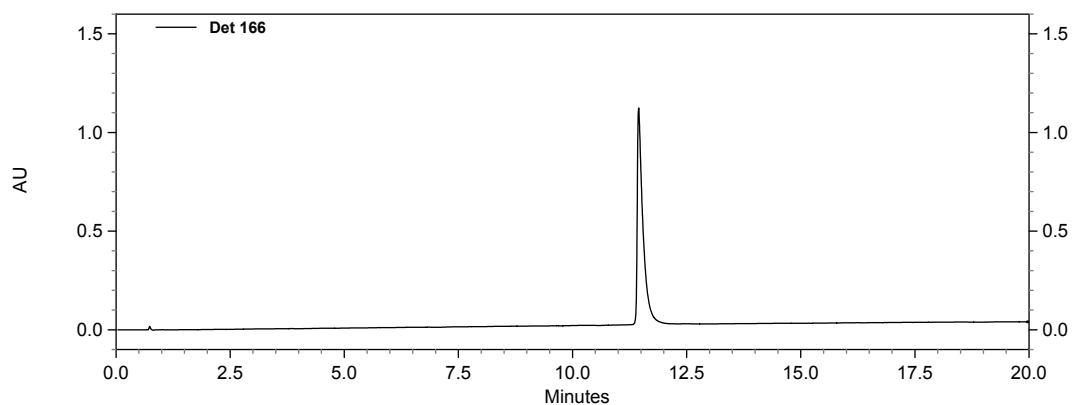
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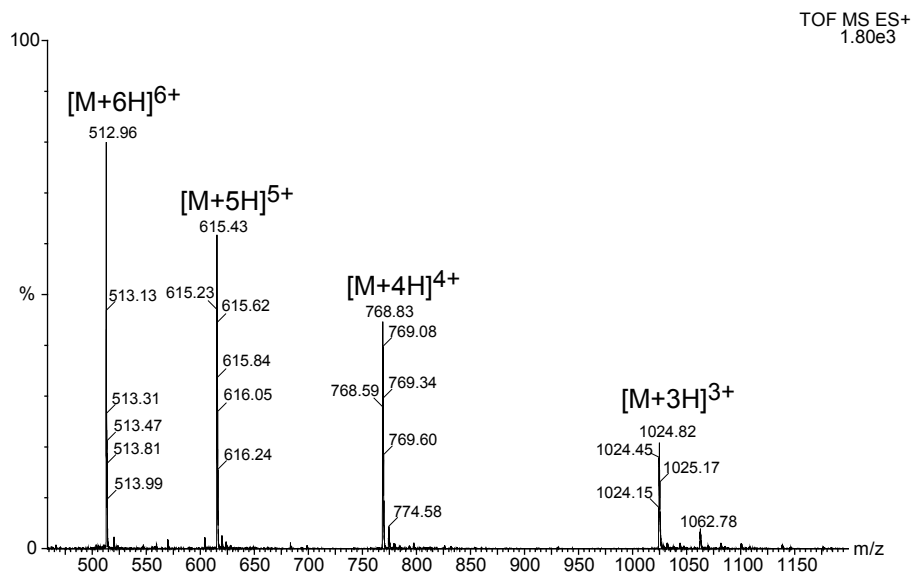
cyclononamer triangle: (*o*-Abc^{2K}-*p*-Abc^{2K}-*p*-Abc^{2K})₃ (**9c**)
 Analytical RP-HPLC chromatograph and mass spectrum (ESI-MS)

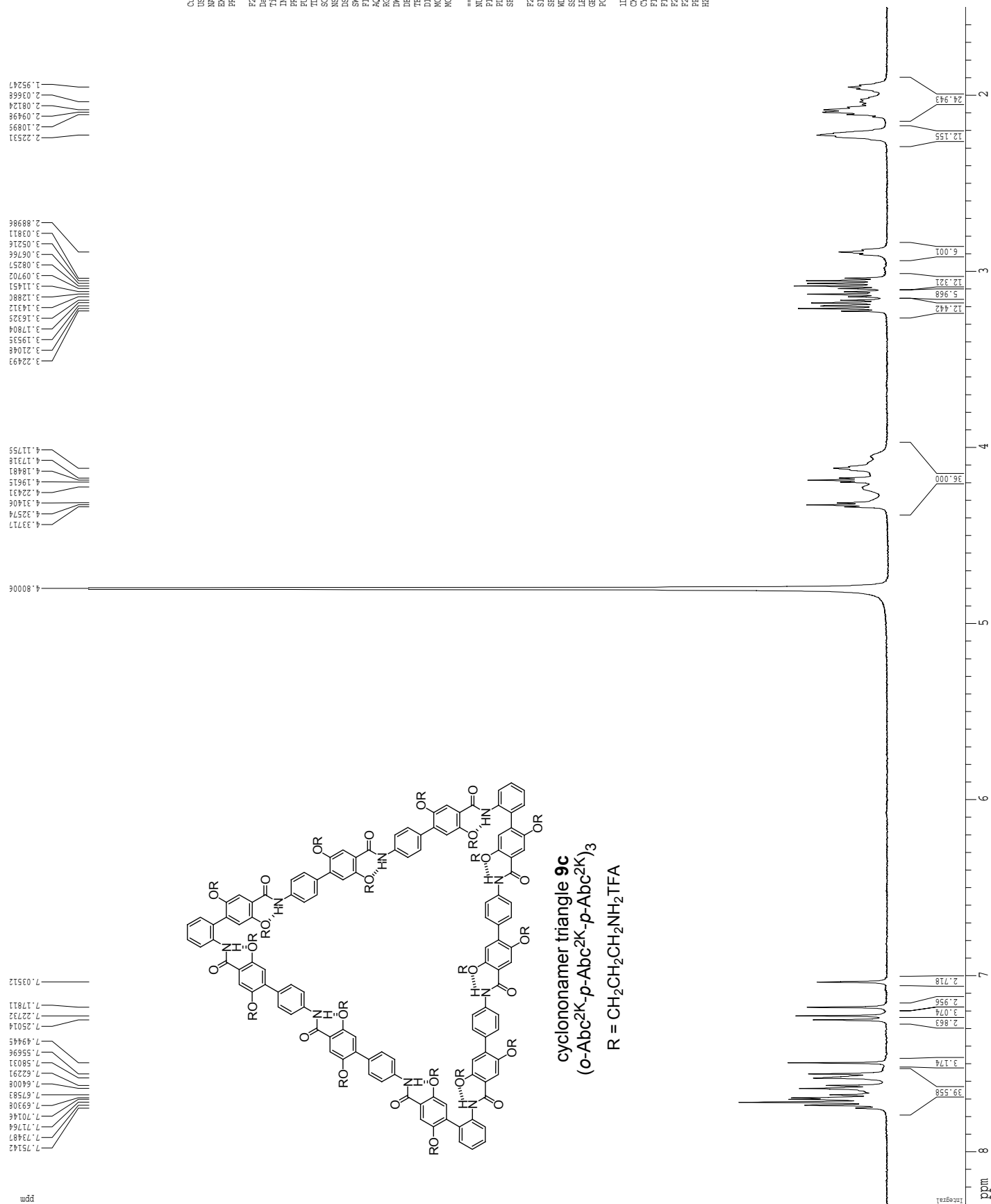


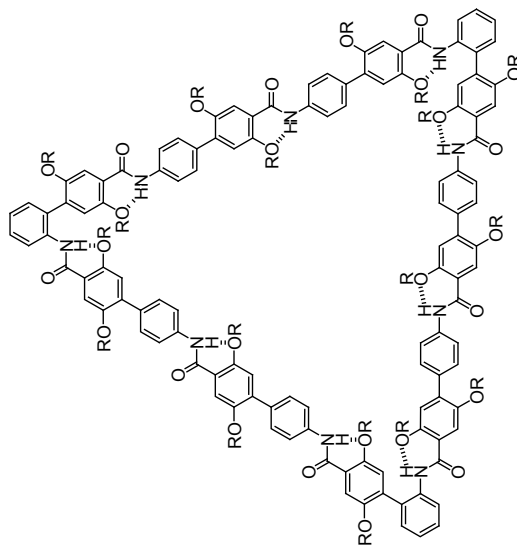
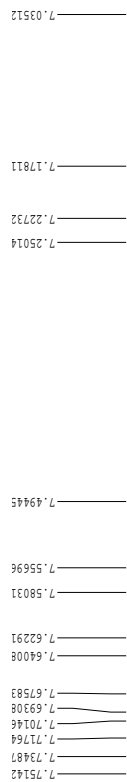
(a) Analytical RP-HPLC (5-50% acetonitrile with 0.1% TFA over 20 min, $\lambda = 214$)



(b) ESI Mass spectrum. (Calcd exact mass for C₁₇₁H₂₀₇N₂₇O₂₇ [M] = 3070.57)



^1H NMR (500 MHz, 298 K, D_2O) spectrum of cyclonamer triangle **9c**

^1H NMR (500 MHz, 298 K, D_2O) spectrum of cyclonamer triangle **9c** (aromatic region)cyclonamer triangle **9c**(o-Abc²K-p-Abc²K)_{2/3}R = $\text{CH}_2\text{CH}_2\text{CH}_2\text{NH}_2\text{TFA}$

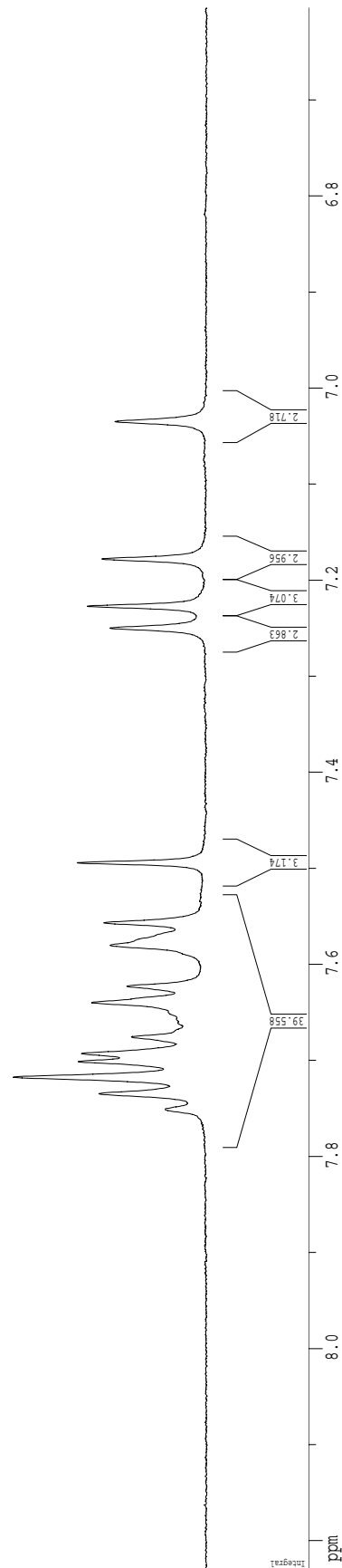
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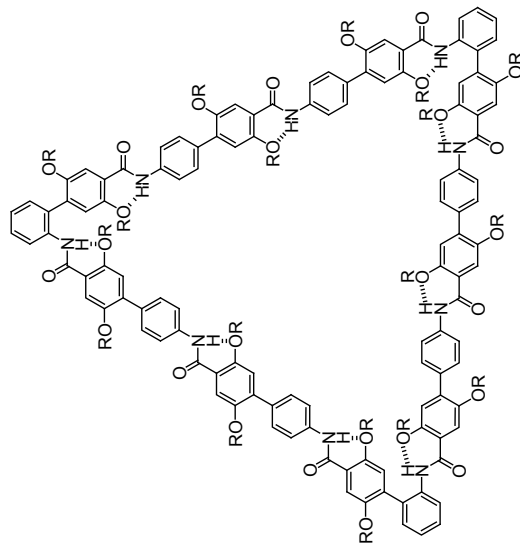
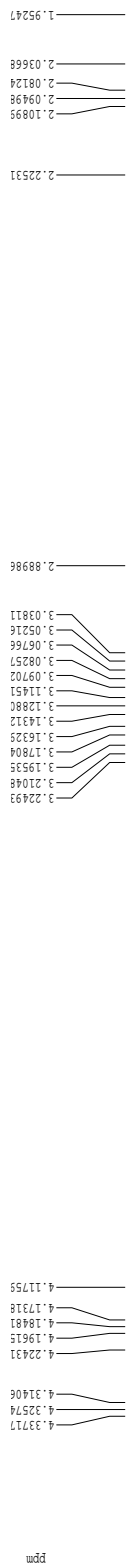
Current Data Parameters
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USER      cgottha
NAME      c9-11-59
EXPNO     1
PROCNO    1

F2 - Acquisition Parameters
=====
Date_     20070509
Time      2.00
INSTRUM   spect
PROBHD    5 mm CPCTC1H-
PULPROG   zgpg30
TD        81728
SOLVENT   D2O
NS        64
DS        2
SWH       8012.820 Hz
FIDRES    0.098043 Hz
AQ        5.0998774 sec
RG        18
DW        62.400 usec
DE        6.00 usec
TE        300.2 K
D1        0.10000000 sec
d11       0.00000000 sec
d12       0.00000000 sec
d13       0.01500000 sec
===== CHANNEL f1 =====
NUC1      1H
P1        8.00 usec
PL1       1.60 dB
SFO1      500.2235015 MHz

F2 - Processing parameters
=====
SI        65536
SF        500.2200051 MHz
RG        64
SSB       0
LB        0.30 Hz
GB        0
PC        4.00

ID NMR plot parameters
CX        22.80 cm
CY        300.57 cm
FAP       8.230 ppm
F1        4116.67 Hz
F2P       6.604 ppm
F2        3303.66 Hz
F3        0.00000000 ppm/cm
F4        35.65662 Hz/cm
  
```



^1H NMR (500 MHz, 298 K, D_2O) spectrum of cyclonamer triangle **9c** (aliphatic region)cyclonamer triangle **9c**
(*o*-Abc²K-*p*-Abc²K-*p*-Abc²K)₃R = $\text{CH}_2\text{CH}_2\text{CH}_2\text{NH}_2\text{TFA}$

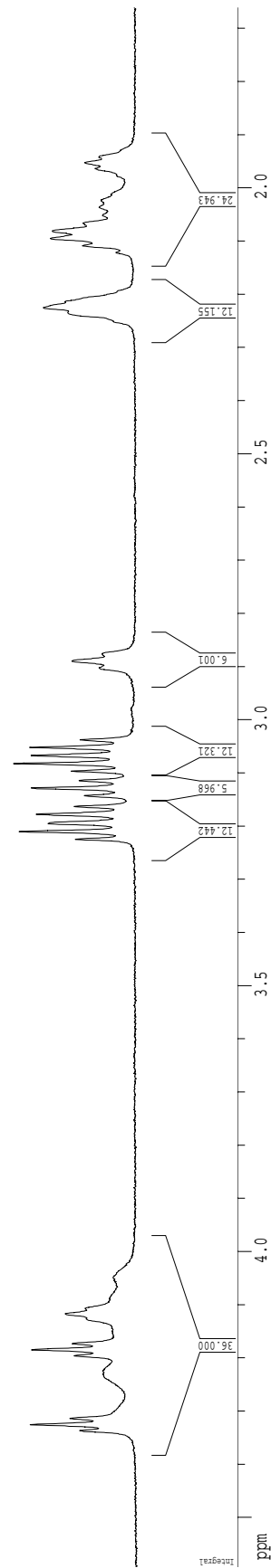
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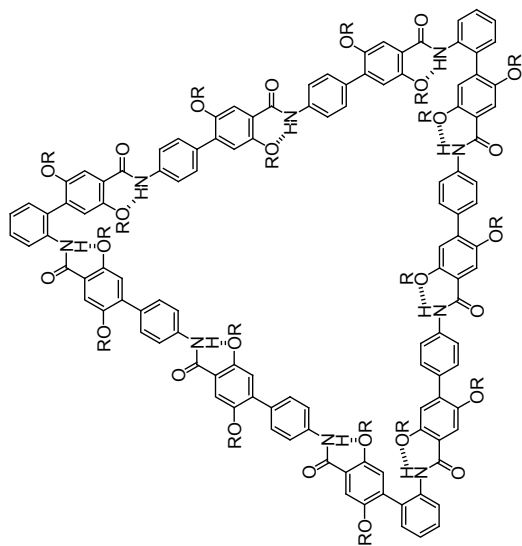
Current Data Parameters
=====
USER          cyotlia
NAME          c9-11-59
EXPNO        1
PROCNO       1

F2 - Acquisition Parameters
=====
Date_         20070309
Time          21:00
INSTRUM      zgpg30
PROBHD       5 mm CPYCI 1H
PULPROG      zgpg30
TD           81728
SOLVENT      D2O
NS           64
DS           2
SWH          8012.820 Hz
FIDRES      0.09643 Hz
AQ          5.0989774 sec
RG          38
DM          62.400 usec
DE          5.00 usec
TE          300.2 K
D1          0.10000000 sec
d11         0.00000000 sec
d12         0.00000000 sec
d13         0.01500000 sec
===== CHANNEL f1 =====
NUC1         1H
P1          8.00 usec
PL1         1.60 dB
SFO1        500.2235015 MHz

F2 - Processing parameters
=====
SI          65536
SF          500.2200031 MHz
WDW         EM
SSB         0
LB          0.30 Hz
GB          0
PC          4.00

1D NMR plot parameters
=====
CX          22.80 cm
CY          300.57 cm
F1P        4.594 PPM
F2P        2297.86 Hz
F3P        1.661 PPM
F4P        831.08 Hz
F5P        0.13256 PPM/cm
HZCM       64.55255 Hz/cm
  
```



¹³C NMR (125 MHz, 320 K, D₂O) spectrum of cyclonamer triangle **9c**

cyclonamer triangle **9c**
(*o*-Abc²K-*p*-Abc²K-*p*-Abc²K)₃

R = CH₂CH₂CH₂NH₂TFA

```

Current Data Parameters
USBR          c9octa
NAME          c9-11-59
PROCNO       6
PROGNO       1

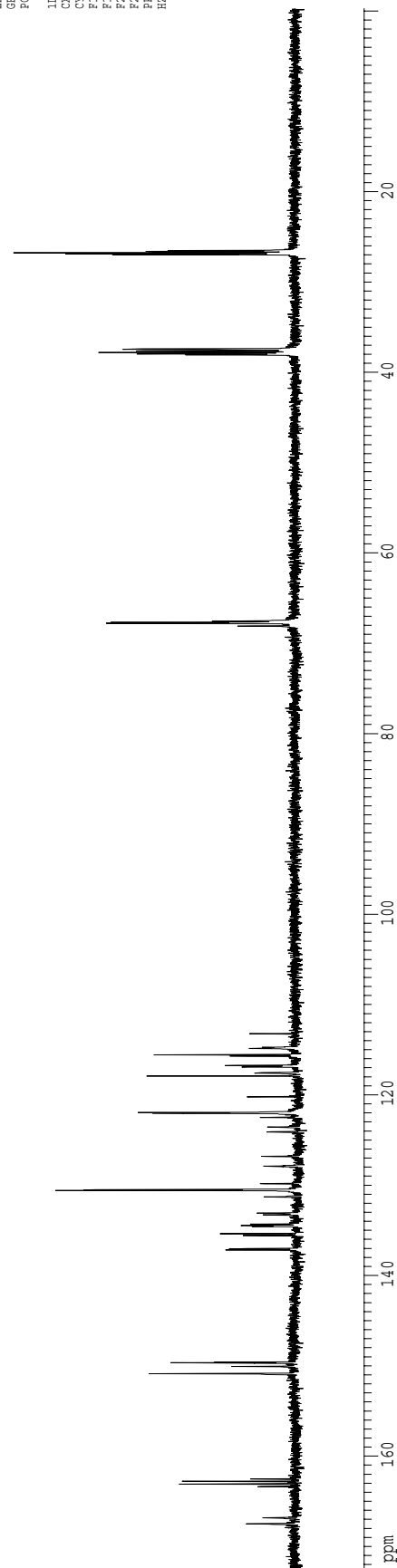
F2 - Acquisition Parameters
Date_         20070509
Time_        21.43
INSTRUM      cryo500
PROBHD       5 mm CPTCI 1H-
PULPROG      zgpg30
TD           65418
SOLVENT      D2O
NS           8500
DS           4
SWH          30303.031 Hz
FIDRES       0.463222 Hz
AQ           1.0794470 sec
RG           18390.4
DM           16.500 usec
DE           6.00 usec
TE           300.2 K
D1           0.25000000 sec
d11          0.03000000 sec
NOEPRST      0.00000000 sec
NOEPRG       0.01500000 sec

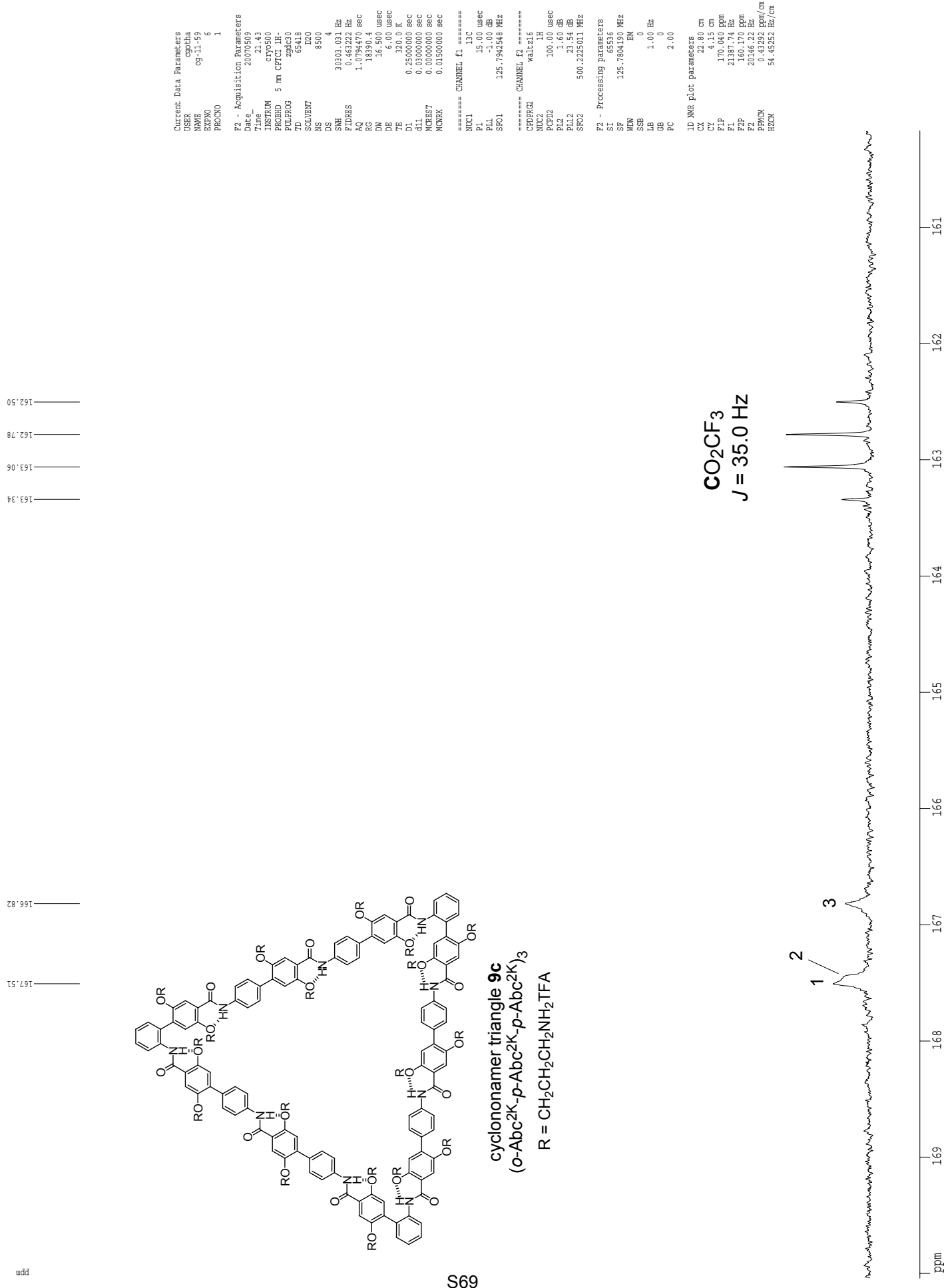
===== CHANNEL F1 =====
NUC1         13C
P1           15.00 usec
PL1          -1.00 dB
SFO1         125.7942548 MHz

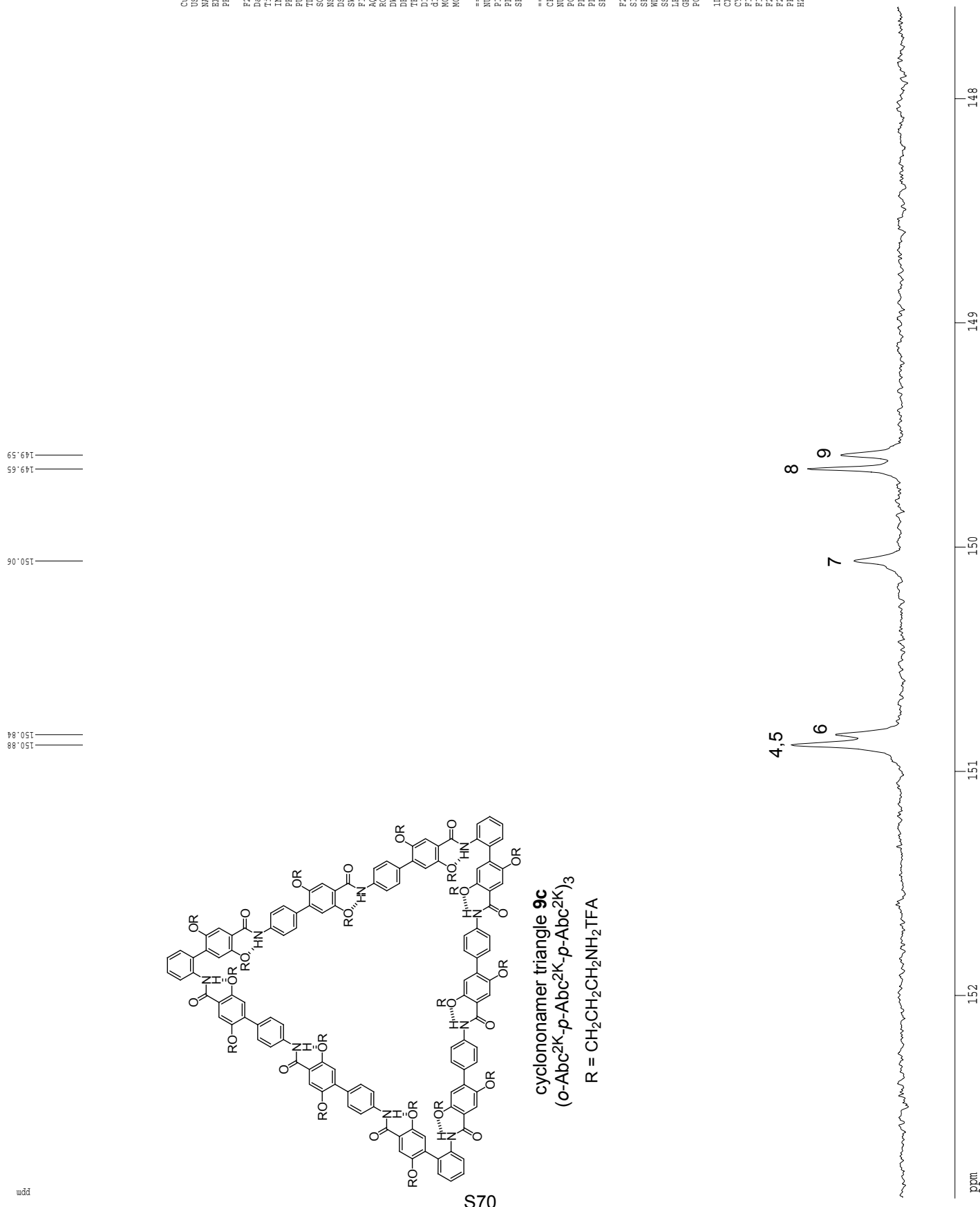
===== CHANNEL E2 =====
CPDPRG2      waltz16
NUC2         1H
PCPD2        100.00 usec
PL2          1.60 dB
PL12         21.54 dB
SFO2         500.2225011 MHz

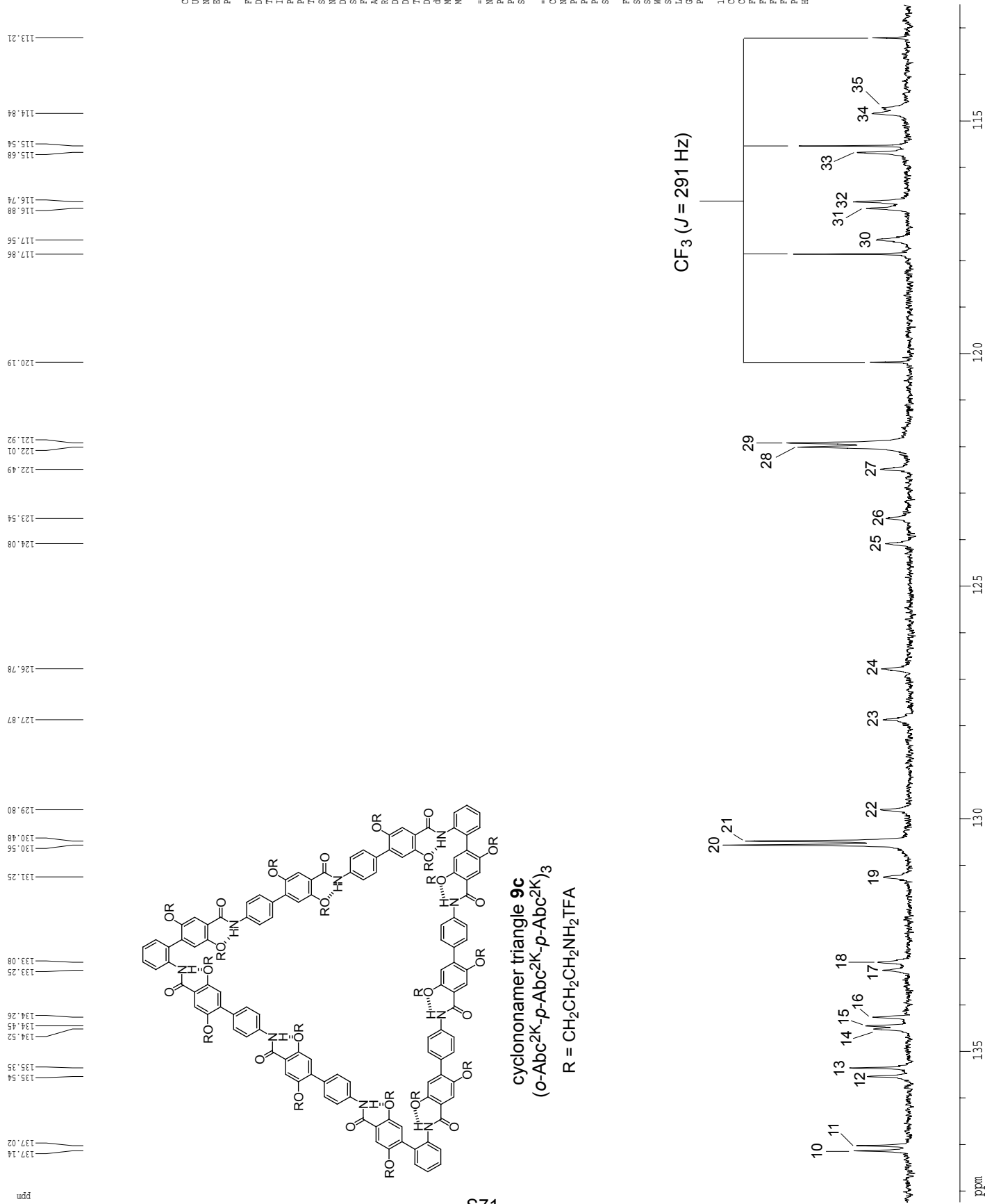
F2 - Processing Parameters
SI           65536
SF           125.7804190 MHz
WDW          EM
SSB          0
LB           1.00 Hz
GB           0
PC           2.00

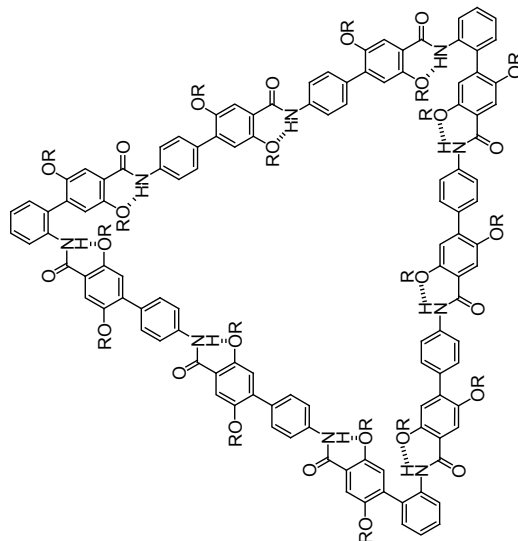
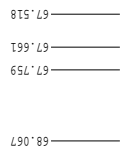
1D NMR plot parameters
CX           22.80 cm
CY           4.15 cm
FIP          172.606 ppm
FL           21710.48 Hz
FZP          -0.248 ppm
FWDW         2.00 Hz
FREQ(CN)     7.56312 ppm/cm
HZ(CN)       953.58447 Hz/cm
  
```



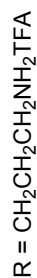
^{13}C NMR (125 MHz, 320 K, D_2O) spectrum of cyclononamer triangle **9c** (carbonyl region expansion)

^{13}C NMR (125 MHz, 320 K, D_2O) spectrum of cyclononamer triangle **9c** (aromatic region expansion 1)

^{13}C NMR (125 MHz, 320 K, D_2O) spectrum of cyclononamer triangle **9c** (aromatic region expansion 2)

¹³C NMR (125 MHz, 320 K, D₂O) spectrum of cyclonamer triangle **9c** (aliphatic region expansion 1)

cyclonamer triangle **9c**
(*o*-Abc2*K*-*p*-Abc2*K*-*p*-Abc2*K*)₃



```

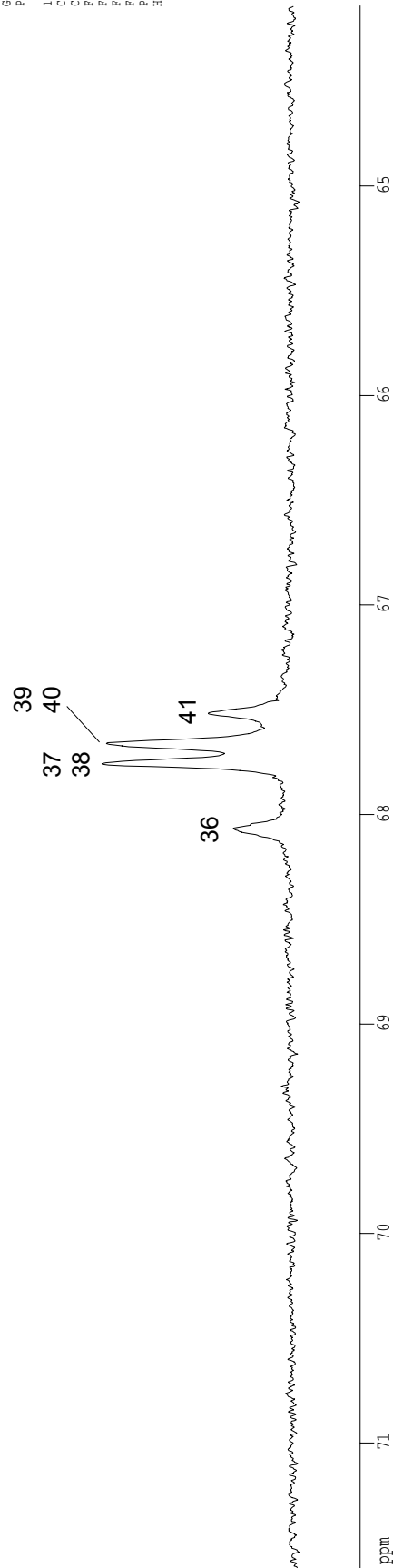
Current Data Parameters
USBR
NAME c9c
PROBHD 5 mm CPTCI 1H-
PULPROG zgpg30
TD 65418
SOLVENT D2O
NS 8500
DS 4
SWH 30303.031 Hz
FIDRES 0.463222 Hz
AQ 1.0794470 sec
RG 18390.4
DM 16.500 usec
DE 6.00 usec
TE 300.2 K
D1 0.25000000 sec
d11 0.03000000 sec
MGEST 0.00000000 sec
MORCK 0.01500000 sec

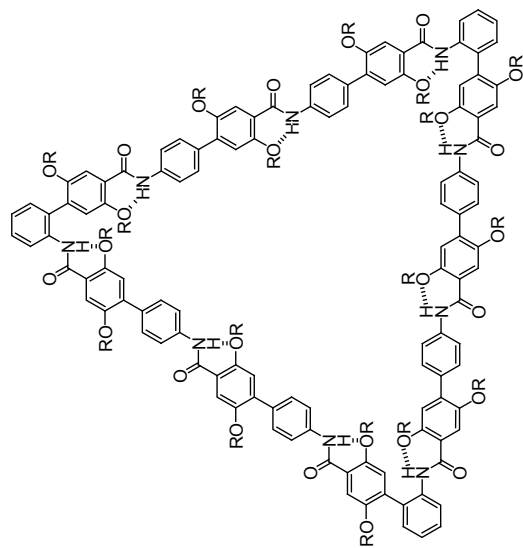
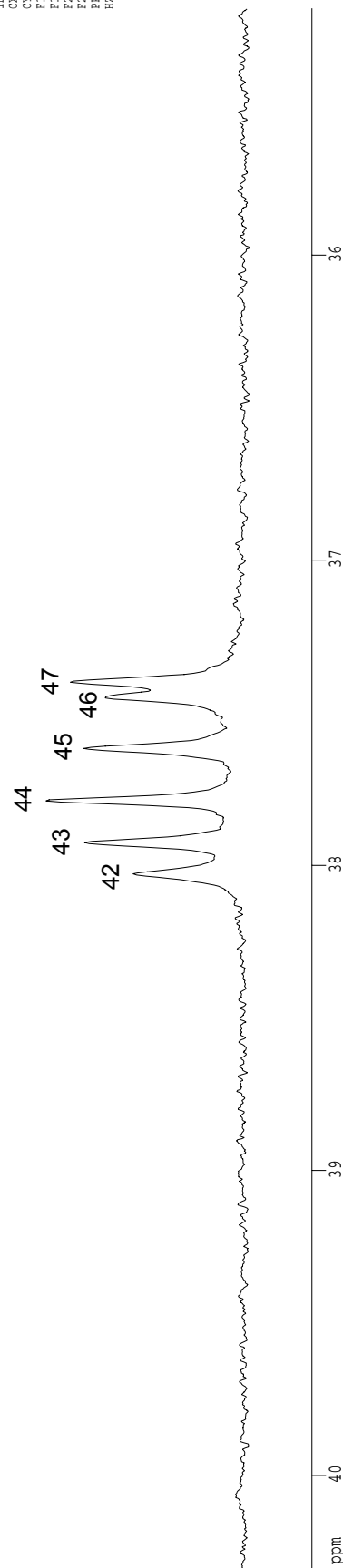
===== CHANNEL F1 =====
NUC1 13C
P1 15.00 usec
PL1 -1.00 dB
SFO1 125.7942548 MHz

===== CHANNEL E2 =====
CPDPRG2 waltz16
NUC2 1H
PCPDZ 100.00 usec
PL2 1.60 dB
PL12 23.54 dB
SFO2 500.2225011 MHz

F2 - Processing parameters
SI 65836
SF 125.7804190 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 2.00

1D NMR plot parameters
CX 22.80 cm
CY 4.15 cm
FIP 71.615 ppm
FL 9007.75 Hz
FZP 64.142 ppm
FWD 819.776 Hz
SFOCN 0.221776 ppm/cm
HZCN 41.22766 Hz/cm
  
```



^{13}C NMR (125 MHz, 320 K, D_2O) spectrum of cyclonamer triangle **9c** (aliphatic region expansion 2)cyclonamer triangle **9c**(o-Abc^{2k}-p-Abc^{2k}-p-Abc^{2k})₃R = $\text{CH}_2\text{CH}_2\text{CH}_2\text{NH}_2$ TFA

```

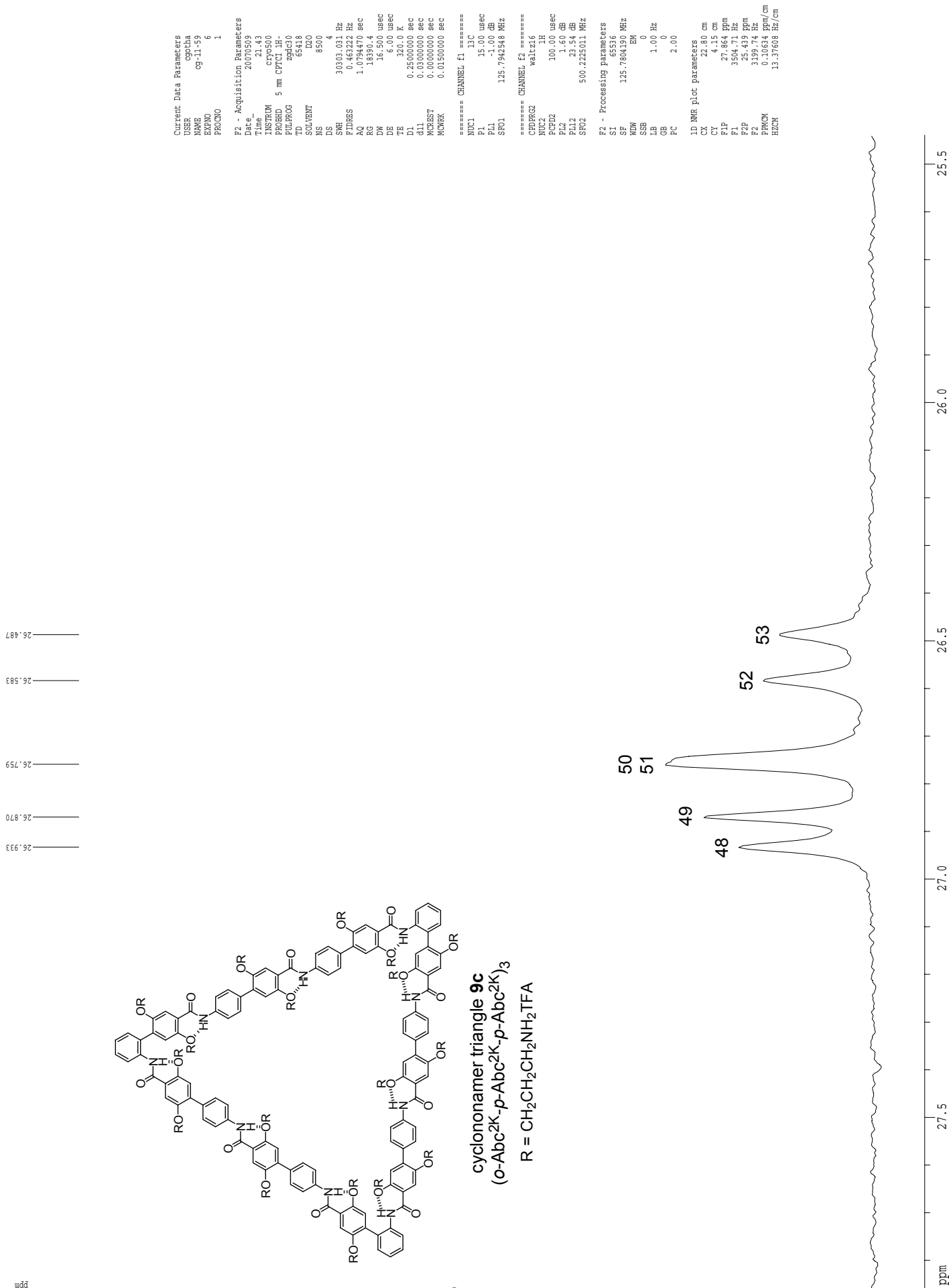
Current Data Parameters
USBR          cgschia
NAME          09-11-59
PULPROG      zgpg30
PROCNO       6
PROBHD       5 mm QNP1H-
PULPROG      zgpg30
TD           65418
SOLVENT      D2O
NS           8500
DS           4
SWH          30303.031 Hz
FIDRES       0.463222 Hz
AQ           1.0794470 sec
RG           18390.4
DM           16.500 usec
DE           6.00 usec
TE           300.2 K
D1           0.25000000 sec
d11          0.03000000 sec
NOFEST       0.00000000 sec
NOVERK       0.01500000 sec

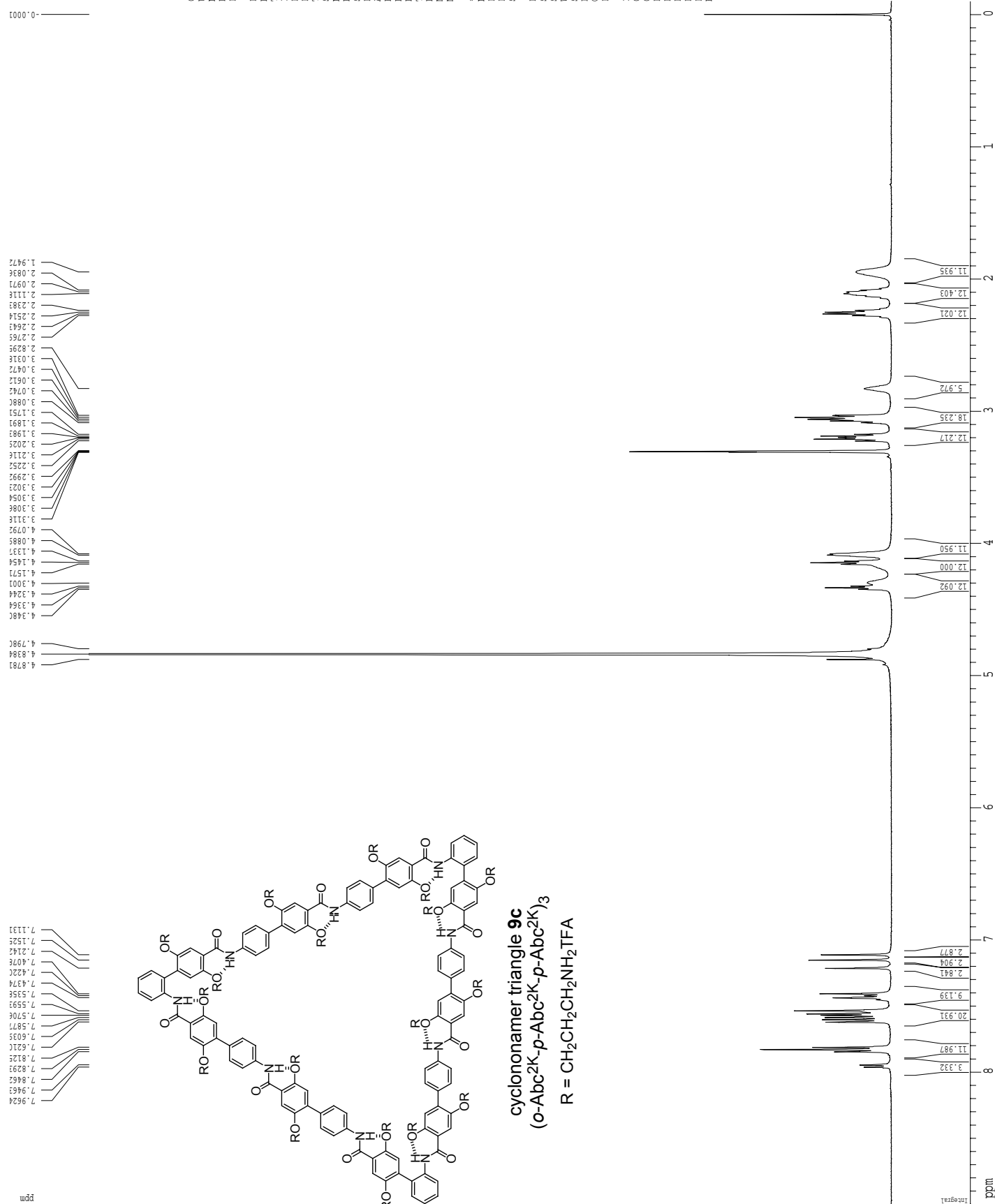
===== CHANNEL F1 =====
NUC1         13C
P1           15.00 usec
PL1         -1.00 dB
SFO1        125.7942548 MHz

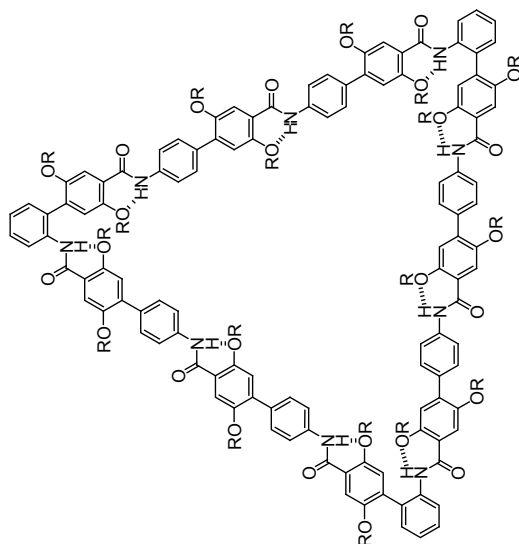
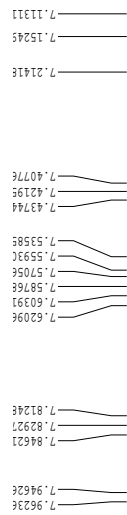
===== CHANNEL E2 =====
CPDPRG2      waltz16
NUC2         1H
PCPD2       100.00 usec
PL2         1.60 dB
PL12        21.54 dB
SFO2        500.2225011 MHz

F2 - Processing parameters
SI           65836
SF          125.7804190 MHz
WDW          EM
SSB          0
LB           1.00 Hz
GB           0
PC           2.00

1D NMR plot parameters
CX          22.80 cm
CY           4.15 cm
FIP         40.315 ppm
FL          5070.88 Hz
F2P         35.152 ppm
F4P         4426.51 Hz
SFO(N)      0.2225011 ppm/cm
HZ(N)       28.20388 Hz/cm
  
```

¹³C NMR (125 MHz, 320 K, D₂O) spectrum of cyclonamer triangle **9c** (aliphatic region expansion 3)

^1H NMR (500 MHz, 298 K, CD_3OD) spectrum of cyclonamer triangle **9c**

^1H NMR (500 MHz, 298 K, CD_3OD) spectrum of cyclononamer triangle **9c** (aromatic region)

cyclononamer triangle **9c**
(*o*-Abc^{2k}-*p*-Abc^{2k}-*p*-Abc^{2k})₃

R = $\text{CH}_2\text{CH}_2\text{CH}_2\text{NH}_2\text{TFA}$

```

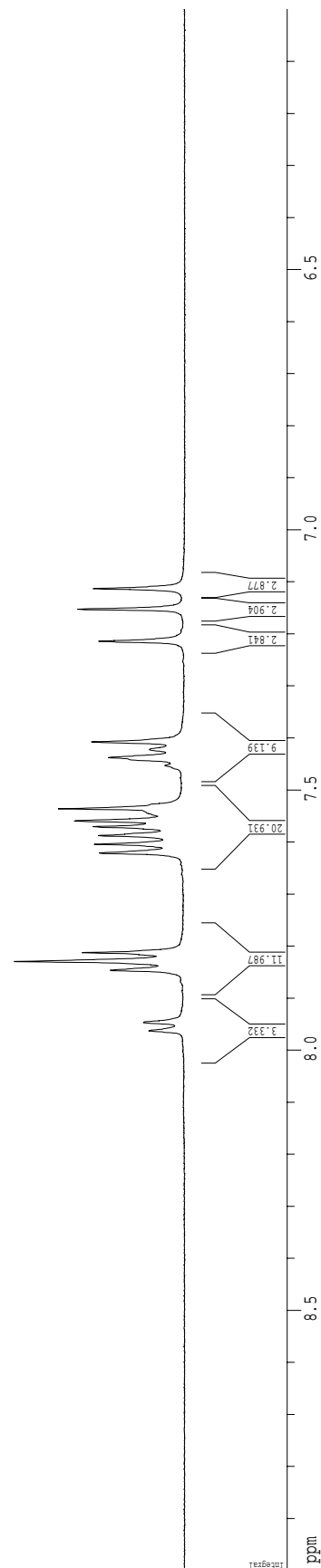
Current Data Parameters
USER          cgothia
NAME          cg-1-58
EXPNO        1
PROCNO       1

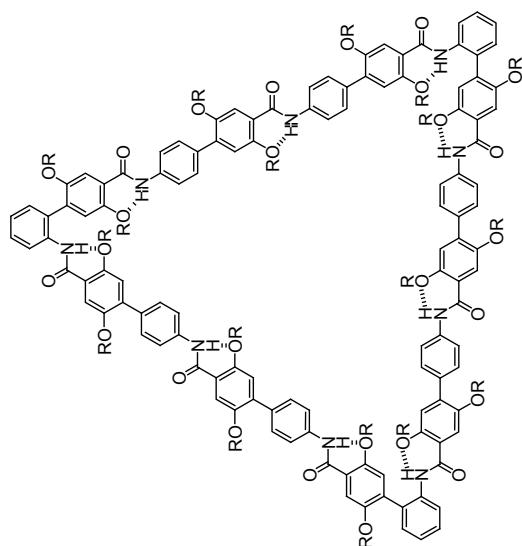
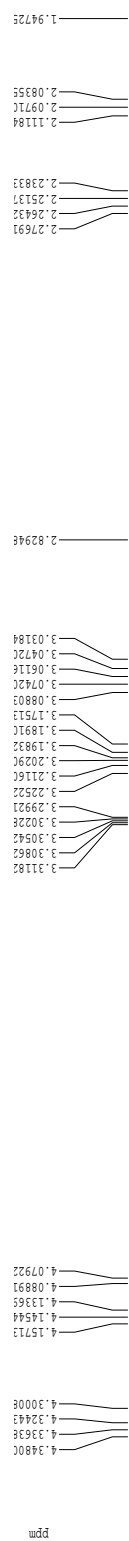
F2 - Acquisition Parameters
Date_        20071021
Time         17.54
INSTRUM      spect
PROBHD       5 mm broadband
PULPROG      zgpg30
TD           81728
SOLVENT      CD3OD
NS           16
DS           2
SWH          8012.820 Hz
FIDRES       0.098043 Hz
AQ           5.0958774 sec
RG           724.1
DW           62.400 usec
DE           6.00 usec
TE           298.0 K
NUC1         13
ACQRESF      0.1100000 sec
SFO1         0.1000000 sec
MCHNK1       0.01500000 sec

***** CHANNEL f1 *****
NUC1         1H
P1           12.00 usec
PL1          -3.00 dB
SFO1         499.8284988 MHz

F2 - Processing parameters
SI           65536
RG           499.8250207 MHz
RO           0.0000000
DE           0.30 Hz
GB           0
PC           4.00

1D NMR plot parameters
CX           22.80 cm
CY           80.36 cm
F1P         9.000 ppm
F1          4498.43 Hz
F2P         6.000 ppm
F2          2998.95 Hz
FREQNCH     0.13158 ppm/cm
HZCM        65.76645 Hz/cm
  
```



¹H NMR (500 MHz, 298 K, CD₃OD) spectrum of cyclonamer triangle **9c** (aliphatic region)

cyclonamer triangle **9c**
(*o*-Abc₂*K*-*p*-Abc₂*K*-*p*-Abc₂*K*)₃

R = CH₂CH₂CH₂NH₂TFA

```

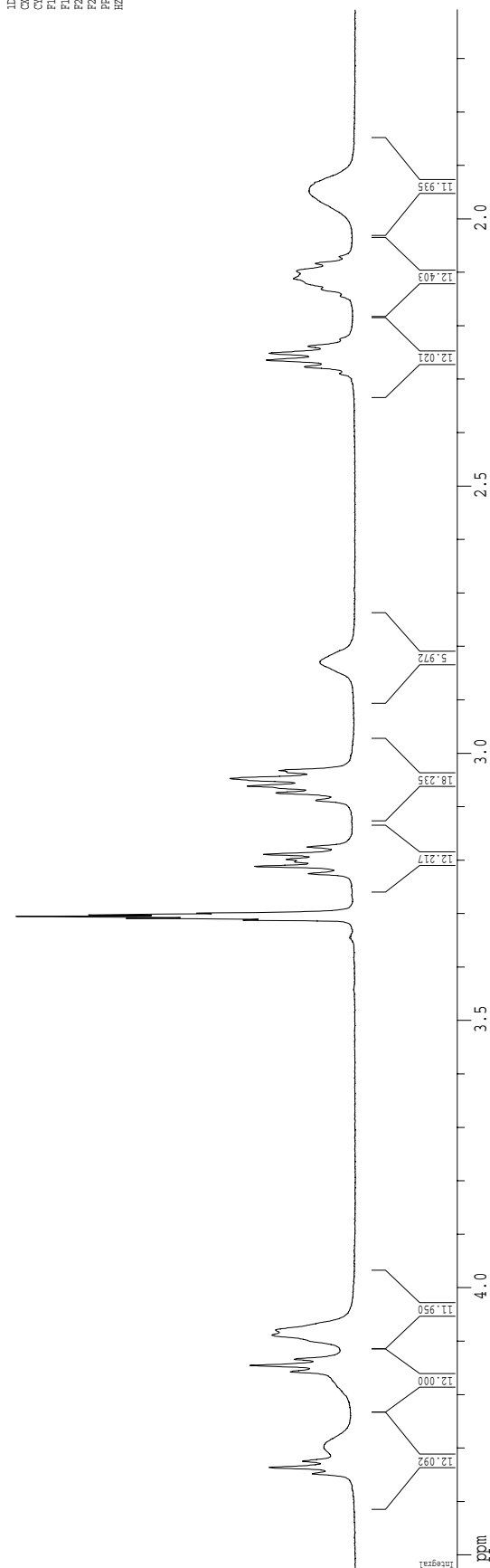
Current Data Parameters
USER          eschla
NAME          C9-11-58
EXPNO        1
PROCNO       1

F2 - Acquisition Parameters
Date_         20071031
Time         17.54
INSTRUM      gm500
PROBHD       5 mm broadband
PULPROG      zg30
TD           81728
AQ           0.1000000 sec
RG           724.1
DM           62.400 usec
DE           6.00 usec
TE           298.0 K
MCREST       0.0000000 sec
MCPRK        0.0150000 sec

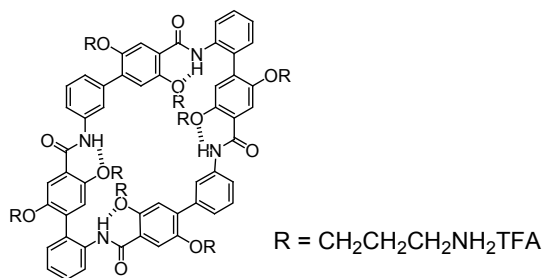
===== CHANNEL f1 =====
NUC1          1H
P1           12.00 usec
PL1          -3.00 dB
SFO1         499.824498 MHz

F2 - Processing parameters
SI           65536
SF           499.8250207 MHz
WDW          EM
SSB          0
LB           0.30 Hz
GB           0
PC           4.00

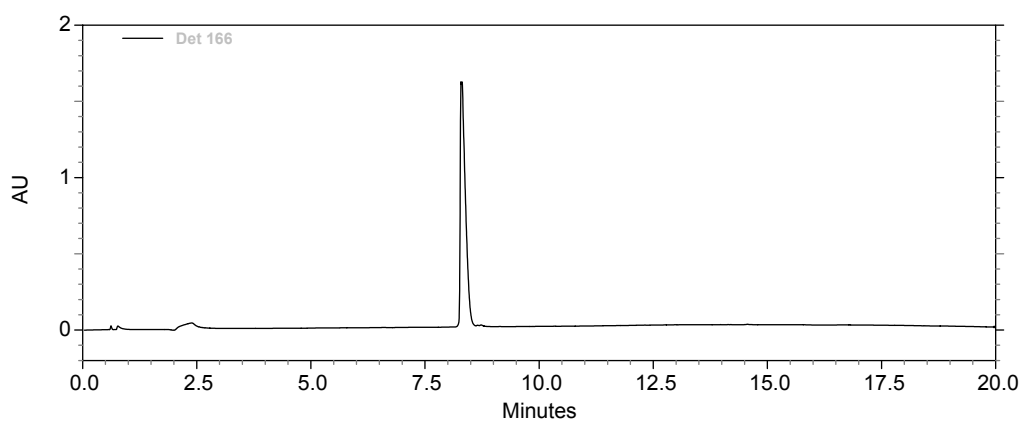
ID NMR plot parameters
CX          22.80 cm
CY          80.36 cm
F1P         4.527 ppm
F2P         1.609 ppm
F2          804.37 Hz
PPMCM       0.12757 ppm/cm
HZCM        63.96493 Hz/cm
  
```



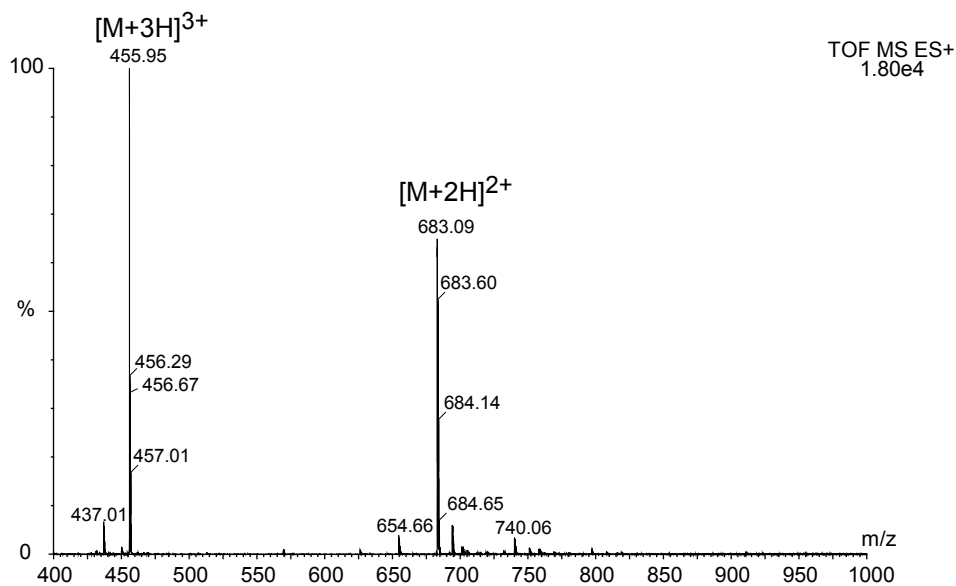
cyclotetramer: (*m*-Abc^{2K}-*o*-Abc^{2K})₂ (**10a**)
Analytical RP-HPLC chromatograph and mass spectrum (ESI-MS)



(a) Analytical RP-HPLC (0-30% acetonitrile with 0.1% TFA over 10 min, $\lambda = 214$)



(b) ESI Mass spectrum. (Calcd exact mass for C₇₆H₉₆N₁₂O₁₂ [M] = 1364.70)



¹H NMR (500 MHz, 298 K, D₂O) spectrum of cyclotetramer parallelogram 10a

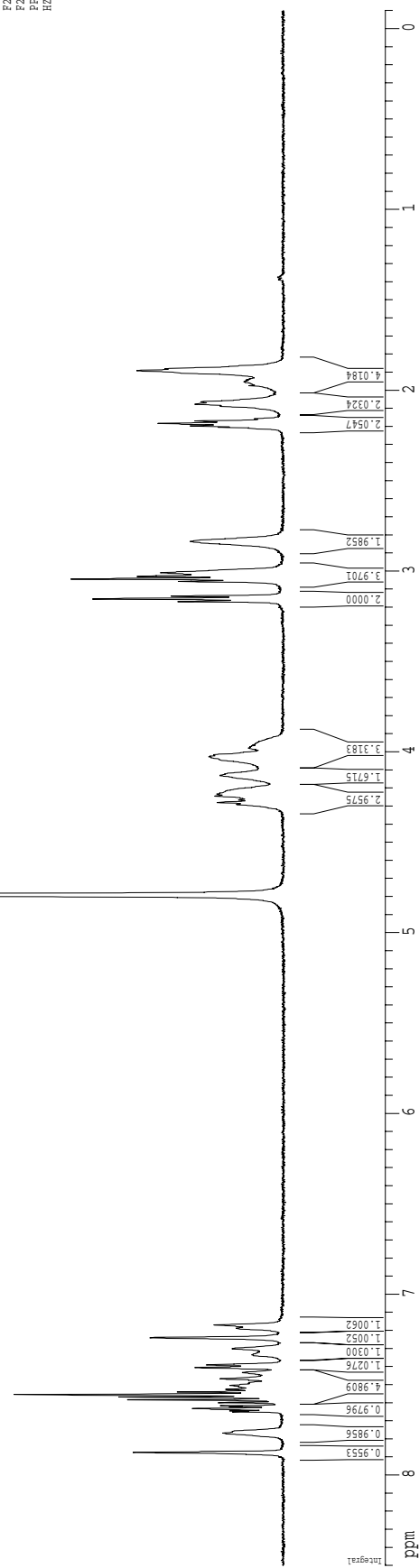
```

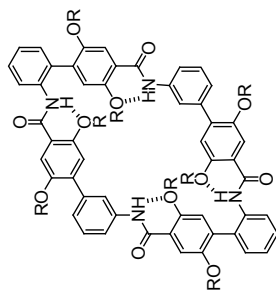
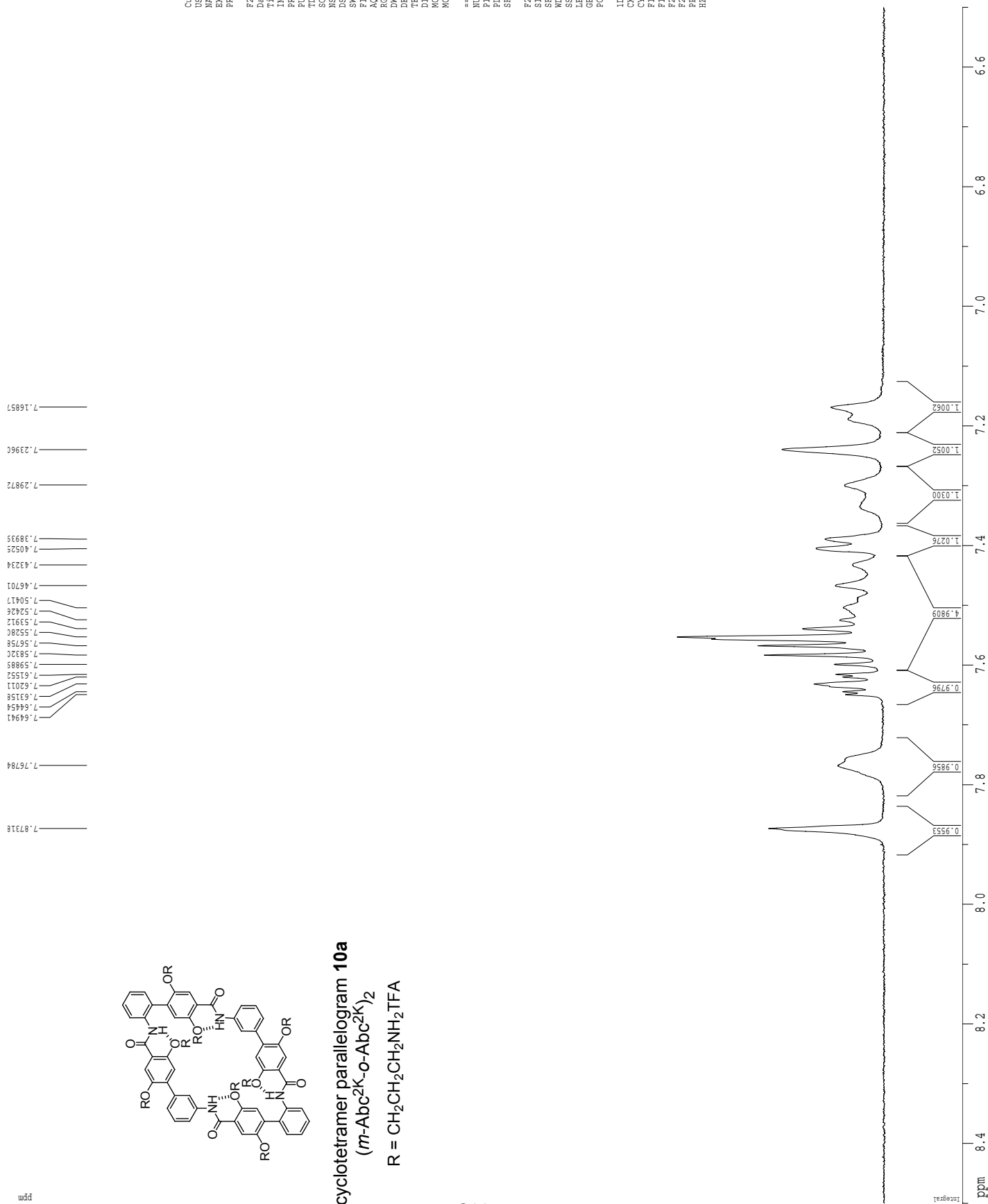
Current Data Parameters
=====
USXR      csotia
NAME      cg-11-25
PROCNO    4
PROBHD    5 mm CPYCI 1H-
PULPROG   zg30
TD         81728
SOLVENT    D2O
NS         64
DS         2
AQ         8013.820 Hz
FIDRES     0.098622 Hz
AQRES      5.0398774 sec
RG         10.1
DM         62.400 usec
DE         6.00 usec
TE         298.0 K
D1         0.10000000 sec
MCREST     0.00000000 sec
MCWREK     0.01500000 sec
===== CHANNEL f1 =====
NUC1       1H
P1         8.00 usec
PL1        0.00 dB
SFO1       500.2335015 MHz
=====
F2 - Processing Parameters
SI         65536
SF         500.2200096 MHz
RG         0
EM         0
SSB        0
LB         0.30 Hz
GB         0
PC         4.00
=====
ID NMR plot parameters
CX         32.80 cm
CY         39.60 cm
CZ         8.500 cm
F1         4251.87 Hz
F2         -0.100 ppm
F3         -50.02 Hz
PRWCMN    0.37719 ppm/cm
HZCMN     188.67949 Hz/cm
  
```

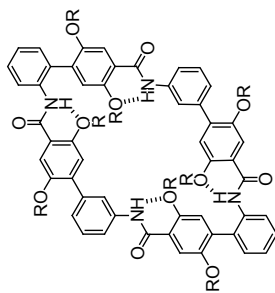
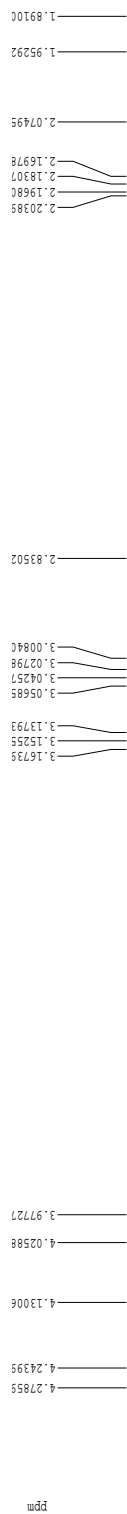
cyclotetramer parallelogram 10a
(*m*-Abc2*k*-*o*-Abc2*k*)₂

R = CH₂CH₂CH₂NH₂TFA

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^1H NMR (500 MHz, 298 K, D_2O) spectrum of cyclotetramer parallelogram **10a** (aromatic region)cyclotetramer parallelogram **10a**
(*m*-Abc²K-o-Abc²K)₂R = $\text{CH}_2\text{CH}_2\text{CH}_2\text{NH}_2\text{TFA}$

¹H NMR (500 MHz, 298 K, D₂O) spectrum of cyclotetramer parallelogram 10a (aliphatic region)

cyclotetramer parallelogram 10a
(m-Abc^{2k}-O-Abc^{2k})₂

R = CH₂CH₂CH₂NH₂TFA

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```

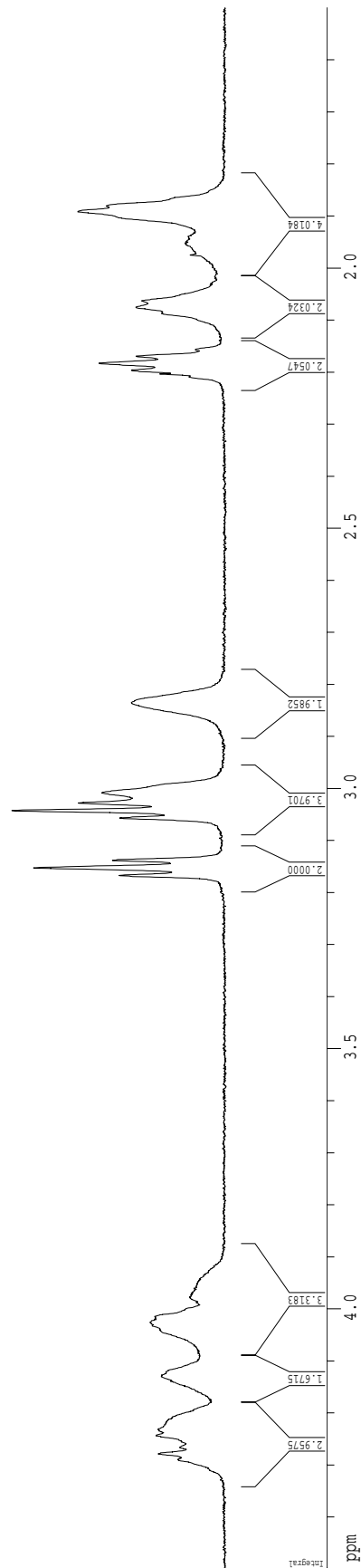
Current Data Parameters
USER          cgolha
NAME          c9-11-25
EXPNO        2
PROCNO       1

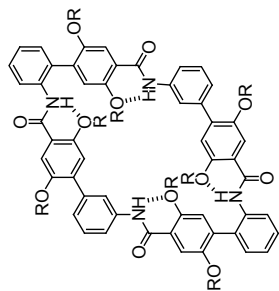
F2 - Acquisition Parameters
Date_         20070313
Time_        23:55
INSTRUM      cryo500
PROBHD       5 mm CPTCI 1H-
PULPROG      zg30
TD           81728
SOLVENT      D2O
NS           64
DS           2
SHE          8012.820 Hz
FIDRES       0.098043 Hz
AQ           5.0999774 sec
RG           0.0000000
DM           62.400 usec
DE           6.00 usec
TE           298.0 K
D1           0.10000000 sec
MCHREST      0.00000000 sec
MCHWK        0.01500000 sec

===== CHANNEL f1 =====
NUC1          1H
P1            8.00 usec
PL1           1.60 dB
SFO1          500.225015 MHz

F2 - Processing parameters
SI            65536
SF            500.220096 MHz
WDW           EM
SSB           0
LB            0.30 Hz
GB            0
PC            4.00

1D NMR plot parameters
CX            22.80 cm
CY            390.87 cm
FLP           4.500 ppm
FIDRES        22.00 Hz
F2P           5.000 ppm
F2            750.33 Hz
F2PWCW        0.13168 ppm/cm
HZCW          65.81842 Hz/cm
  
```



^1H NMR (500 MHz, 330 K, D_2O) spectrum of cyclotetramer parallelogram **10a**cyclotetramer parallelogram **10a**
(*m*-Abc²K₂-o-Abc²K)₂R = $\text{CH}_2\text{CH}_2\text{CH}_2\text{NH}_2\text{TFA}$

```

Current Data Parameters
USER          :
EXPNO        : 3
NAME         : CG-11-25_03
PROCNO      : 1

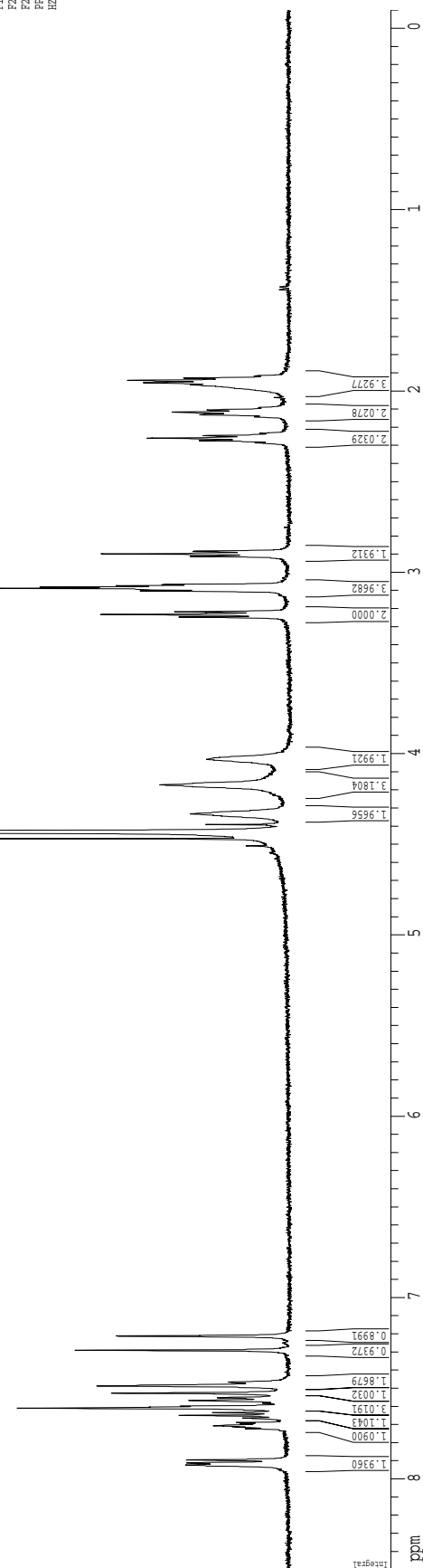
F2 - Acquisition Parameters
Date_       : 20070314
Time       : 22.37
INSTRUM    : gm500
PROBHD     : 5 mm broadband
PULPROG    : zgpg30
TD         : 65536
SOLVENT    : D2O
NS         : 14
DS         : 4
SWH        : 8012.800 Hz
FIDRES     : 0.098043 Hz
AQ         : 5.0598774 sec
RG         : 1149.4
DW        : 62.400 usec
DE        : 6.00 usec
TE        : 330.0 K
D1        : 0.1000000 sec
MCREST     : 0.0000000 sec
MCWEEK     : 0.0150000 sec

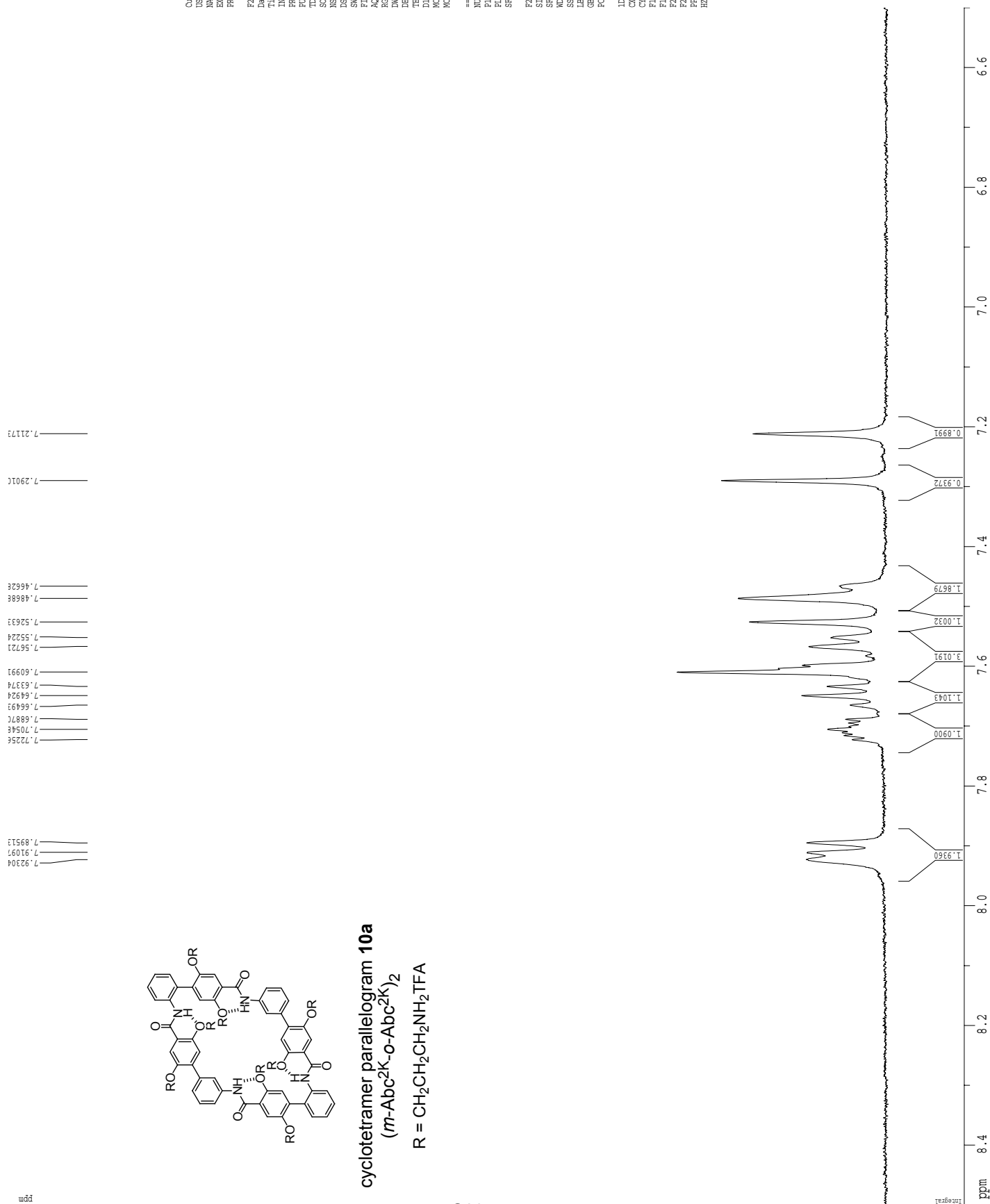
===== CHANNEL f1 =====
NUC1        :  $^1\text{H}$ 
P1         : 12.00 usec
PL1        : -3.00 dB
SFO1       : 499.9334995 MHz

F2 - Processing parameters
SI         : 65536
SF         : 499.9301936 MHz
WDW        : EM
SSB        : 0
LB         : 0.30 Hz
GB         : 0
PC         : 4.00

1D NMR plot parameters
CX         : 22.80 cm
CY         : 394.14 cm
F1P        : 8.500 ppm
F2P        : 4249.41 Hz
F3P        : -0.100 ppm
F4P        : -49.99 Hz
PPMCON    : 0.37719 ppm/cm
HZCON     : 188.57018 Hz/cm

```



^1H NMR (500 MHz, 330 K, D_2O) spectrum of cyclotetramer **10a** (aromatic region)

```

Current Data Parameters
USER          CSOPIA
NAME          C9-11-25_CN
EXPNO        3
PROCNO       1

F2 - Acquisition Parameters
Date_        20070314
Time         22.37
INSTRUM      GHS50
PROBHD       5 mm broadband
PULPROG      zgpg30
SOLVENT      D2O
NS           64
DS           2
SWH           8012.820 Hz
FIDRES       0.098043 Hz
AQ           5.0998774 sec
RG           1149.4
DM           62.400 usec
DE           6.00 usec
TE           330.0 K
D1           0.1000000 sec
ACQRESF      0.0000000 sec
NUC1         1H
===== CHANNEL f1 =====
NUC1         1H
P1           12.00 usec
PL1          -3.00 dB
SFO1         499.9334995 MHz

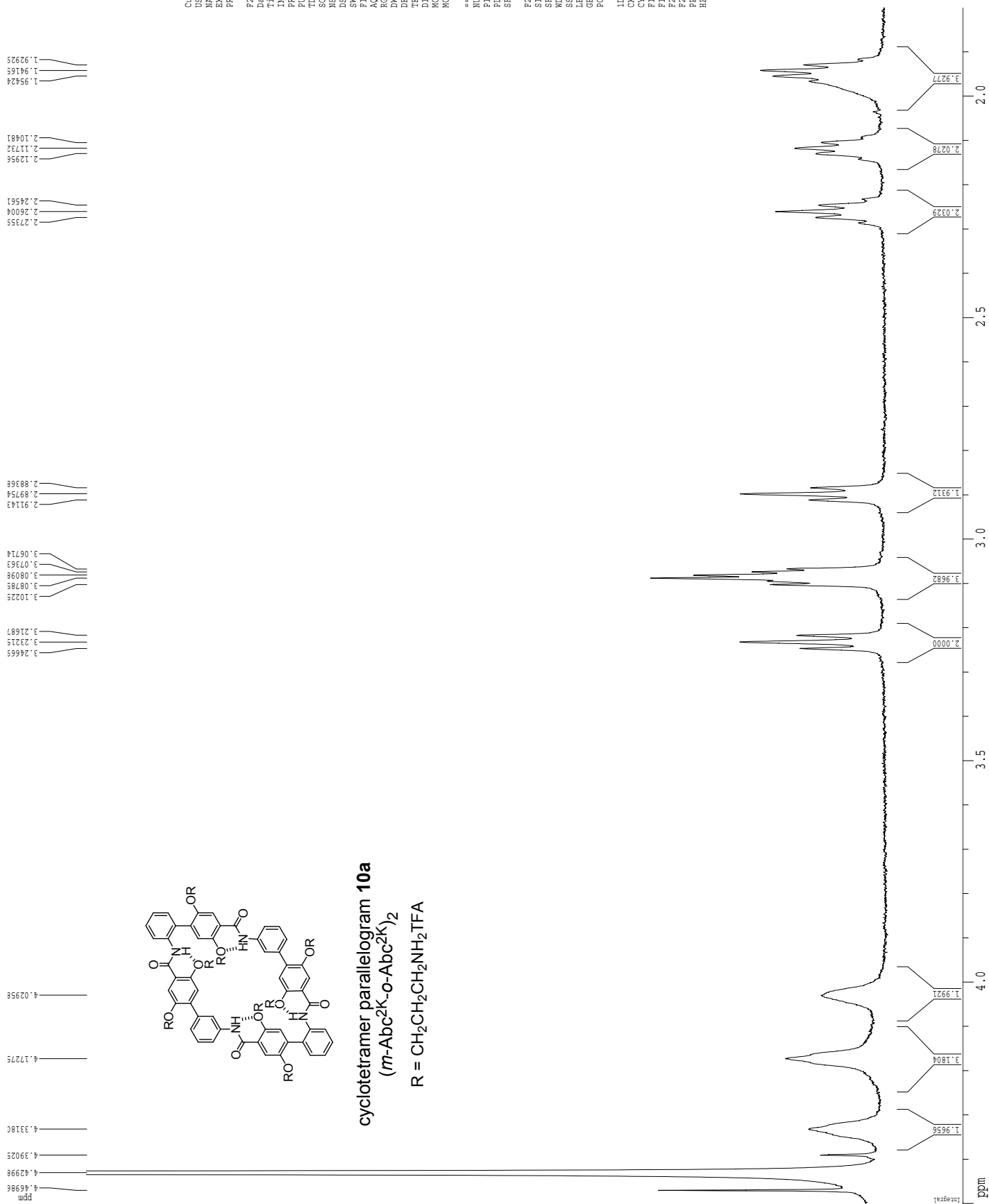
F2 - Processing parameters
SI           65536
SF           499.9301936 MHz
WDW          EM
SSB          0
LB           0.30 Hz
GB           0
PC           4.00

ID: NMR plot parameters
CX           22.80 cm
CY           394.14 cm
F1P          8.500 ppm
F2P          6.500 ppm
F3P          3249.55 Hz
PFMCON       0.08772 ppm/cm
HZCON        43.8553 Hz/cm

```

cyclotetramer parallelogram **10a**
(*m*-Abc²K_o-Abc²K_o)₂R = CH₂CH₂CH₂NH₂TFA

¹H NMR (500 MHz, 330 K, D₂O) spectrum of cyclotetramer parallelogram **10a** (aliphatic region)



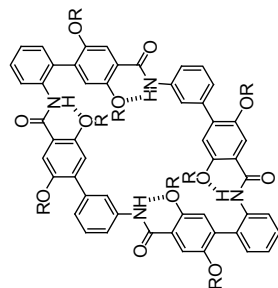
¹³C NMR (125 MHz, 320 K, D₂O) spectrum of cyclotetramer parallelogram **10a**



cyclotetramer parallelogram **10a**
(*m*-*Abc*²*K*-*o*-*Abc*²*K*)₂

R = CH₂CH₂CH₂NH₂TFA

```
Current Data Parameters
USBR          csodia
NAME          09-11-25
PULPROG      zgpg30
PROCNO       1
F2 - Acquisition Parameters
Date_         20070628
Time_         19.03
INSTRUM      cryo500
PROBHD       5 mm CPTCI 1H-
PULPROG      zgpg30
TD           65418
SOLVENT      D2O
NS           8418
DS           4
SWH          30303.031 Hz
FIDRES       0.463222 Hz
AQ           1.0794470 sec
RG           10321.3
DM           16.500 usec
DE           6.00 usec
TE           300.2 K
D1           0.25000000 sec
d11          0.03000000 sec
NOFEST       0.00000000 sec
NOH2K        0.01500000 sec
===== CHANNEL F1 =====
NUC1         13C
P1           15.00 usec
PL1         -1.00 dB
SFO1        125.7942548 MHz
===== CHANNEL E2 =====
CPDPRG2      waltz16
NUC2         1H
PCPD2       100.00 usec
PL2         1.60 dB
PL12        21.54 dB
SFO2        500.2225011 MHz
F2 - Processing parameters
SI           65836
SF          125.7804190 MHz
WDW          EM
SSB          0
LB           1.00 Hz
GB           0
PC           2.00
1D NMR plot parameters
CX          22.80 cm
CY           3.55 cm
FIP         169.978 ppm
FL          21379.91 Hz
F2P         -0.360 ppm
F4P         7.4183 Hz
FREQCN      939.80994 Hz/cm
HZCN
```

^{13}C NMR (125 MHz, 320 K, D_2O) spectrum of cyclotetramer **10a** (aromatic region)cyclotetramer parallelogram **10a**
(*m*- $\text{A}bc^2\text{k}$ -*o*- $\text{A}bc^2\text{k}$)₂R = $\text{CH}_2\text{CH}_2\text{CH}_2\text{NH}_2$ TFA

```

Current Data Parameters
USBR          csodia
NAME          09-11-25
PROCNO       9
PROCNO       1

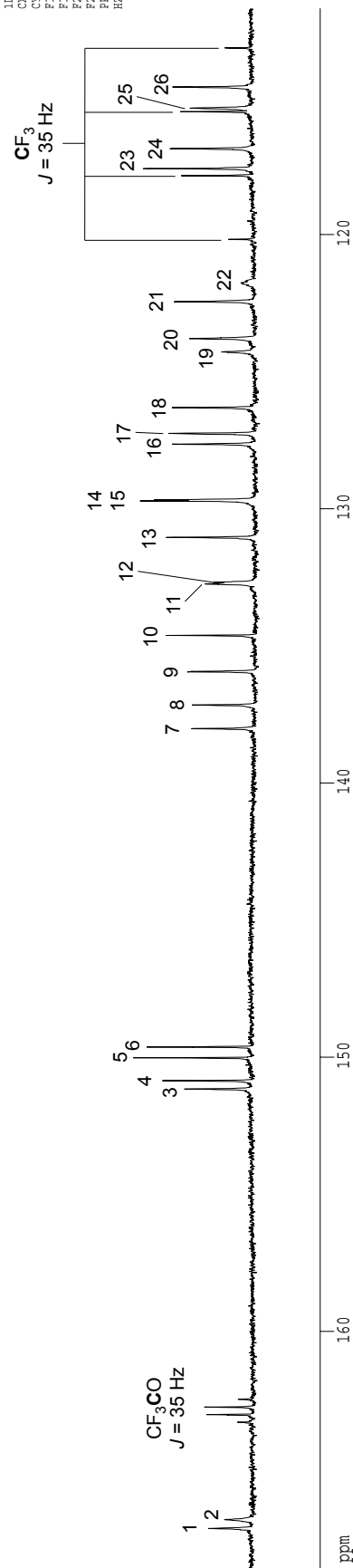
F2 - Acquisition Parameters
Date_         20070628
Time_        19.03
INSTRUM      cryo500
PROBHD       5 mm CPTCI 1H-
PULPROG      zgpg30
TD           65418
SOLVENT      D2O
NS           8418
DS           4
SWH          30303.031 Hz
FIDRES       0.463222 Hz
AQ           1.0794470 sec
RG           10321.3
DM           16.500 usec
DE           6.00 usec
TE           300.2 K
D1           0.25000000 sec
d11          0.03000000 sec
d12          0.00000000 sec
d13          0.00000000 sec
d14          0.01500000 sec
===== CHANNEL F1 =====
NUC1         13C
P1           15.00 usec
PL1          -1.00 dB
SFO1        125.7942548 MHz

===== CHANNEL E2 =====
CPDPRG2      waltz16
NUC2         1H
PCPD2       100.00 usec
PL2          1.60 dB
PL12         23.54 dB
SFO2        500.2225011 MHz

F2 - Processing Parameters
SI           65836
SF          125.7804190 MHz
WDW          EM
SSB          0
LB           1.00 Hz
GB           0
PC           2.00

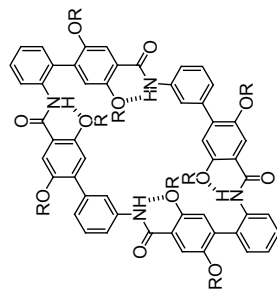
1D NMR plot parameters
CX          22.80 cm
CY           3.55 cm
FIP         168.694 ppm
FL          21216.44 Hz
F2P         111.758 ppm
F3P         14.0570 Hz
SFO(MN)     3.07173 ppm/cm
HZCM        344.05793 Hz/cm

```



^{13}C NMR (125 MHz, 320 K, D_2O) spectrum of cyclotetramer parallelogram **10a** (side chain region)

896.18
67.69
67.87



cyclotetramer parallelogram **10a**
(*m*-Abc²K-*o*-Abc²K)₂

R = $\text{CH}_2\text{CH}_2\text{CH}_2\text{NH}_2$ TFA

```

Current Data Parameters
USBR
CSDIA
NAME 09-11-25
PAPRO 9
PROCNO 1

F2 - Acquisition Parameters
Date_ 20070628
Time_ 19.03
INSTRUM ctye500
PROBHD 5 mm CPTCI 1H-
PULPROG zgpg30
TD 65418
SOLVENT D2O
NS 8418
DS 4
SWH 30303.031 Hz
FIDRES 0.463222 Hz
AQ 1.0794470 sec
RG 10321.3
DM 16.500 usec
DE 6.00 usec
TE 300.2 K
D1 0.25000000 sec
d11 0.03000000 sec
NCESST 0.00000000 sec
NOWEK 0.01500000 sec

===== CHANNEL F1 =====
NUC1 13C
P1 15.00 usec
PL1 -1.00 dB
SFO1 125.7942548 MHz

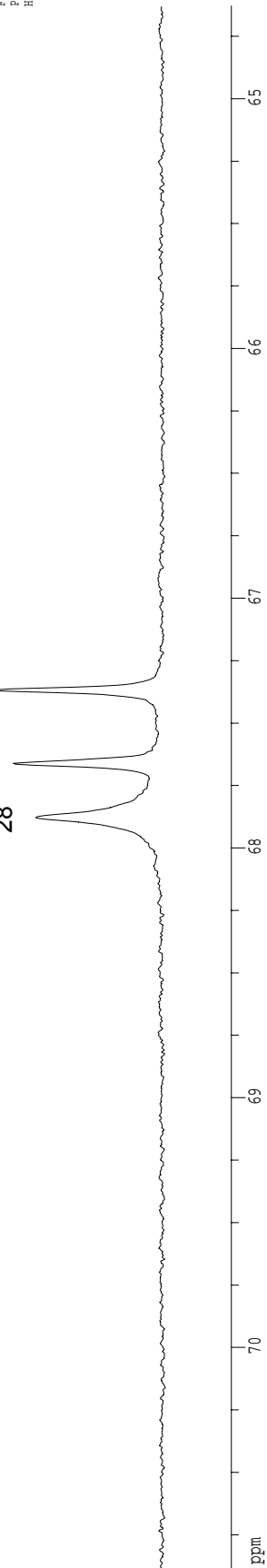
===== CHANNEL E2 =====
CPDPRG2 waltz16
NUC2 1H
PCPDZ 100.00 usec
PL2 1.60 dB
PL12 21.54 dB
SFO2 500.2225011 MHz

F2 - Processing Parameters
SI 65836
SF 125.7804190 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 2.00

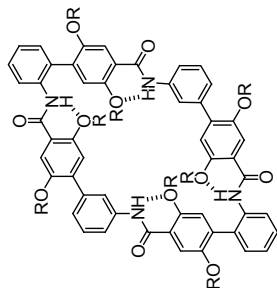
1D NMR plot parameters
CX 22.80 cm
CY 3.55 cm
FIP 70.898 ppm
FL 8917.63 Hz
FZP 64.629 ppm
GZ 0.7210 Hz
SFOCN 0.7210 ppm/cm
HZCN 34.58467 Hz/cm

```

27 28 29 30



¹³C NMR (125 MHz, 320 K, D₂O) spectrum of cyclotetramer parallelogram **10a** (side chain region)



cyclotetramer parallelogram **10a**
(*m*-Abc²K-*o*-Abc²K)₂

R = CH₂CH₂CH₂NH₂TFA

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C. M. Gothard and J. S. Nowick

```

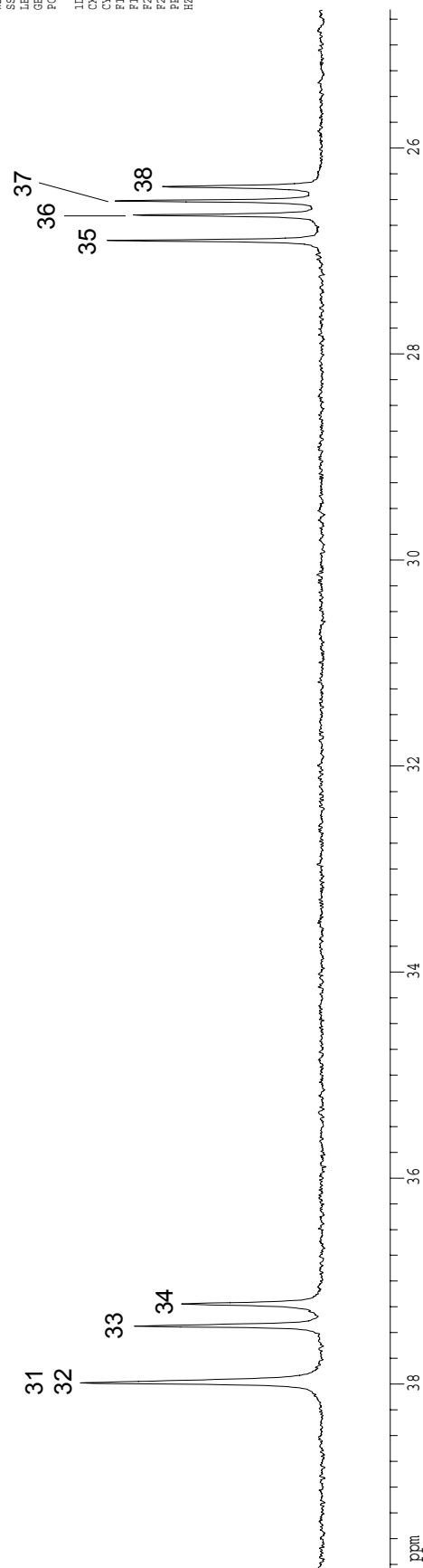
Current Data Parameters
USER          cgothia
NAME          cg-11-25
EXPNO        3
PROCNO       1
P2 - Acquisition Parameters
Date_        20070628
Time_        19.03
INSTRUM      cryo500
PROBHD       5 mm CPYCI 1H-
PULPROG      zgpg30
TD           65418
SOLVENT      D2O
NS           8418
DS           4
SWH          30303.031 Hz
FIDRES       0.463222 Hz
AQ           1.0794470 sec
RG           103211.3
DW           16.500 usec
DE           6.00 usec
TE           300.2 K
D1           0.25000000 sec
d11          0.03000000 sec
MGRESST      0.00000000 sec
MORBK        0.01500000 sec

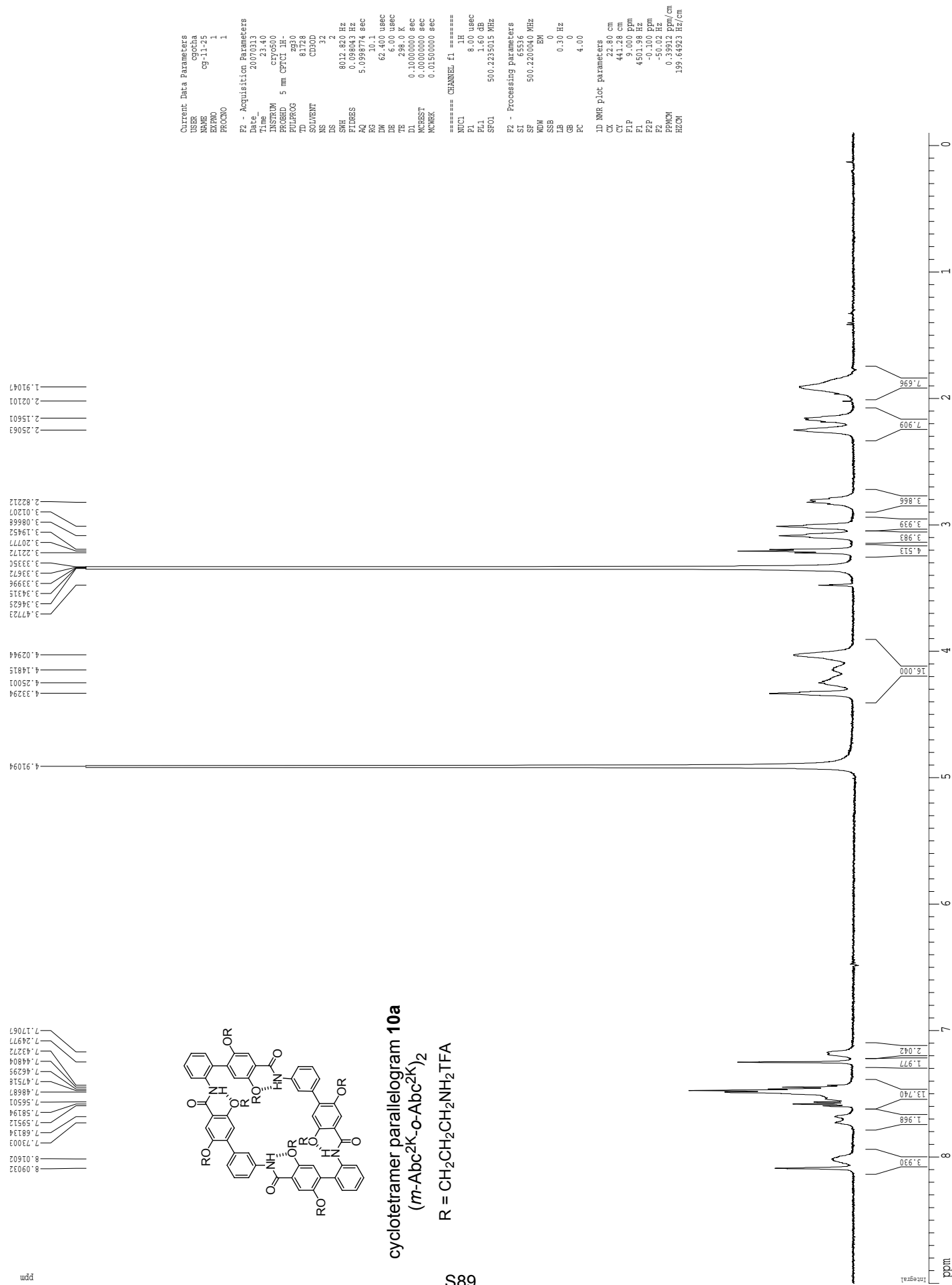
===== CHANNEL f1 =====
NUC1         13C
P1           15.00 usec
PL1          -1.00 dB
SFO1         125.7942548 MHz

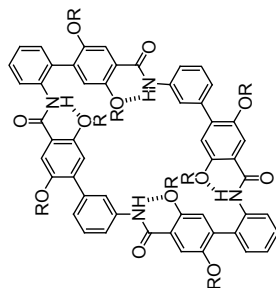
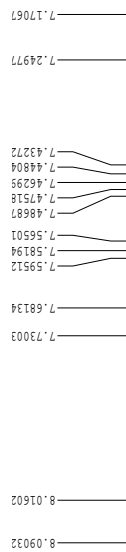
===== CHANNEL f2 =====
CPDPRG2      waltz16
NUC2         1H
PCPD2        100.00 usec
PL2          1.60 dB
PL12         23.54 dB
SFO2         500.2225011 MHz

P2 - Processing parameters
SI           65536
SF           125.794190 MHz
WDW          EM
SSB          0
LB           1.00 Hz
GB           0
PC           2.00

1D NMR plot parameters
CX           22.80 cm
CY           3.55 cm
FIP          39.797 ppm
F1           5005.71 Hz
F2P          241.660 ppm
F2           31072.03 Hz
FREQM        0.06532 ppm/cm
HZCM         83.50816 Hz/cm
    
```



¹H NMR (500 MHz, 298 K, CD₃OD) spectrum of cyclotetramer parallelogram 10a

^1H NMR (500 MHz, 298 K, CD_3OD) spectrum of cyclotetramer parallelogram **10a**cyclotetramer parallelogram **10a**
(*m*- $\text{Abc}^2\text{k-o-Abc}^2\text{k}$)₂R = $\text{CH}_2\text{CH}_2\text{CH}_2\text{NH}_2\text{TFA}$

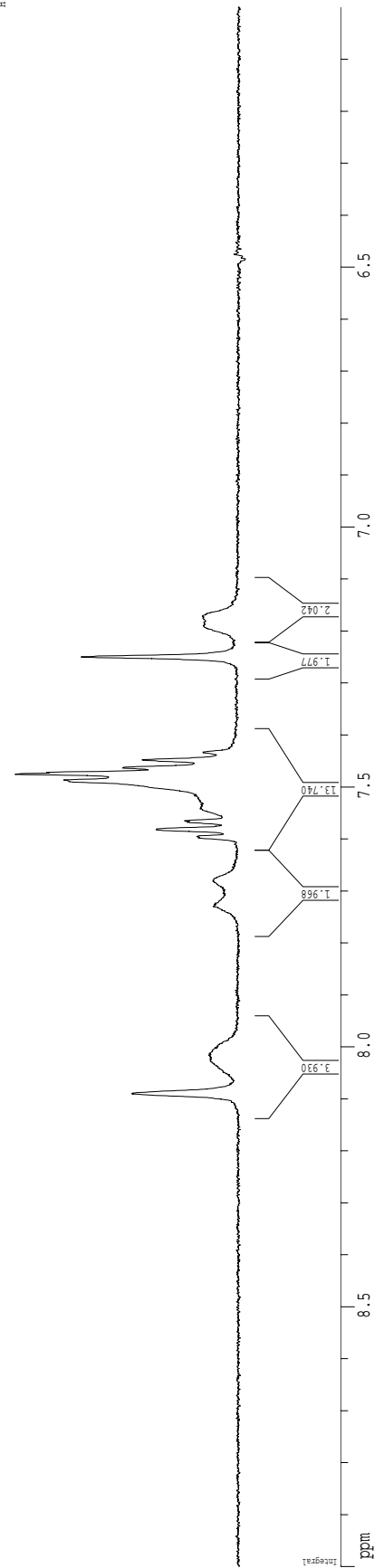
```

Current Data Parameters
=====
USER      cgoctia
NAME      cg-11-25
PROCNO    1
PROBHD    5 mm CPTCI 1H-
PULPROG   zg30
TD         81728
SOLVENT    CD3OD
NS         32
DS         2
SFR        8012.820 Hz
AQ         13.620 Hz
RG         5.0998774 sec
RQ         10.1
DW         62.400 usec
DE         6.00 usec
TE         298.0 K
D1         0.10000000 sec
MCREST     0.00000000 sec
MORF1      0.01500000 sec
===== CHANNEL f1 =====
NUC1       1H
P1         8.00 usec
PL1        -1.00 dB
SFO1       500.235015 MHz

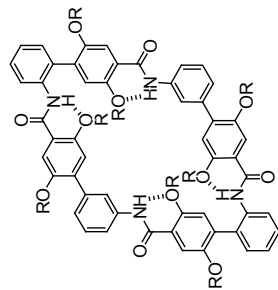
F2 - Processing Parameters
SI         65536
SF         500.230040 MHz
WDW        EM
SSB        0
LB         0.30 Hz
GB         0
PC         4.00

ID NMR plot parameters
CX         22.80 cm
CY         44.70 cm
CZ         8.70 cm
F1P        4601.98 Hz
F2P        6.000 ppm
F3P        3001.32 Hz
PRMCMN     0.13158 ppm/cm
HZCMN      65.81842 Hz/cm

```

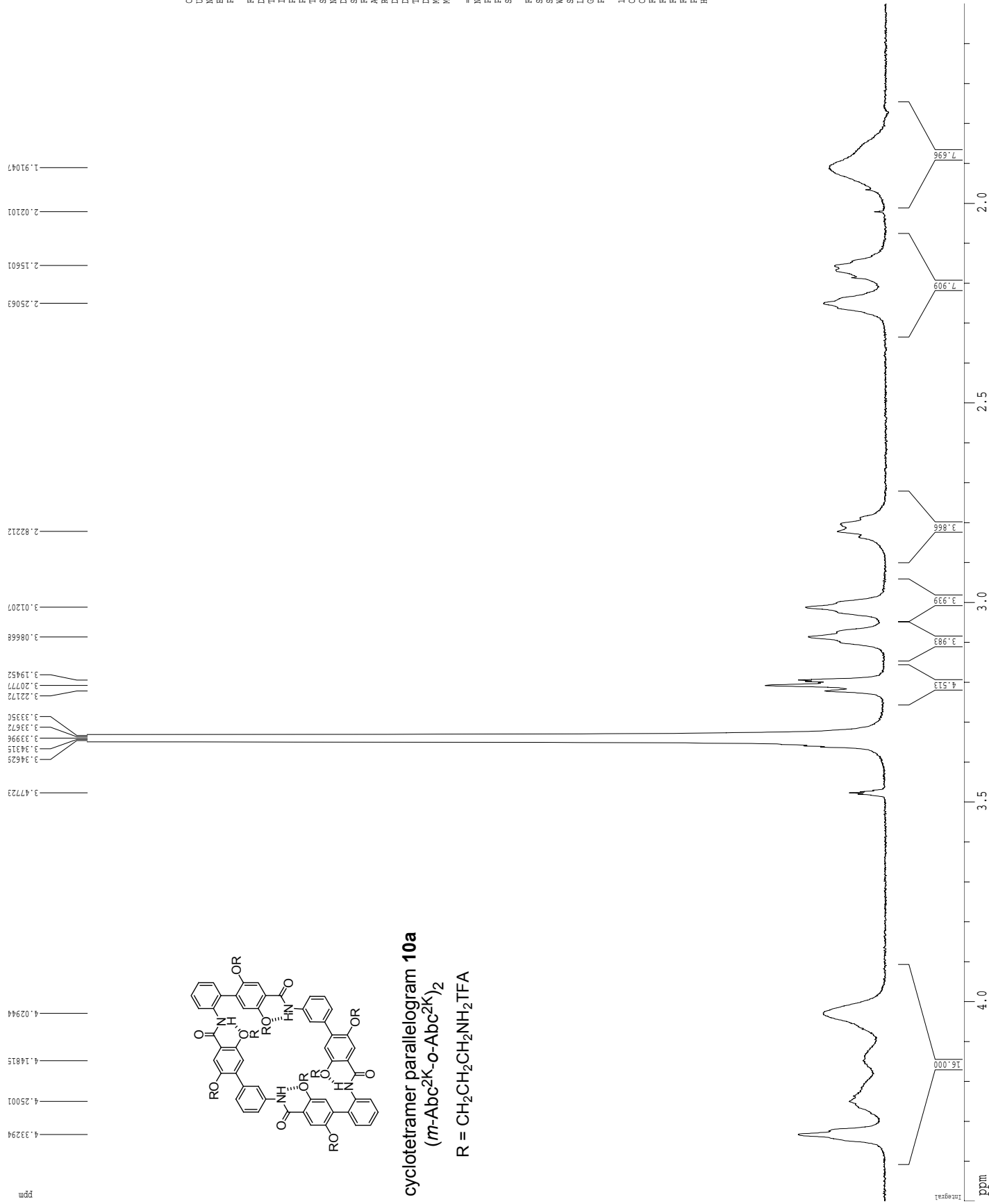


¹H NMR (500 MHz, 298 K, CD₃OD) spectrum of cyclotetramer parallelogram 10a



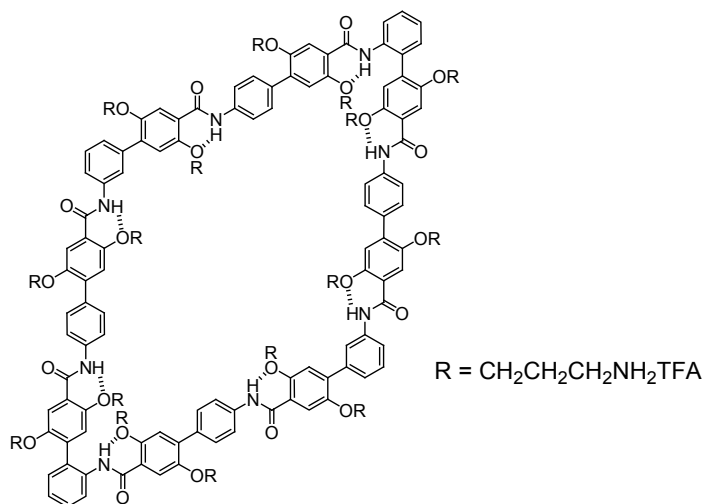
cyclotetramer parallelogram 10a
(*m*-Abc²K-*o*-Abc²K)₂

R = CH₂CH₂CH₂NH₂TFA

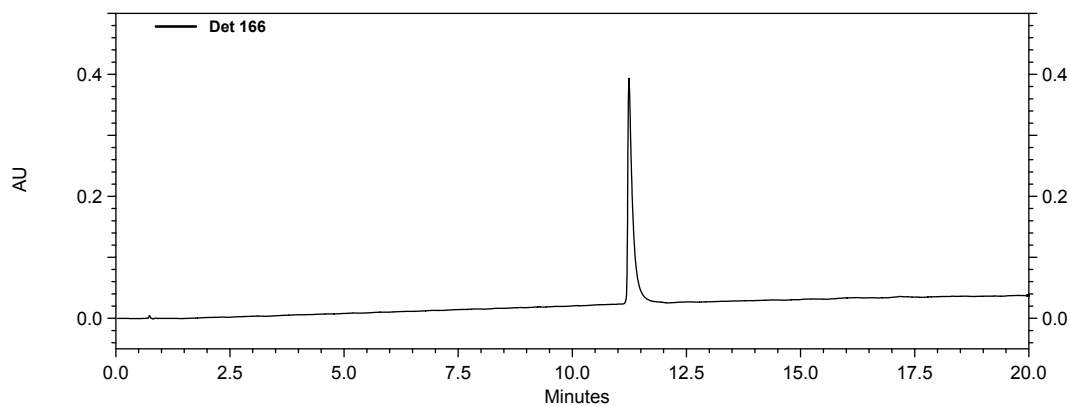


cyclooctamer: (*o*-Abc^{2K}-*p*-Abc^{2K}-*m*-Abc^{2K}-*p*-Abc^{2K})₂
 parallelogram (**10b**)

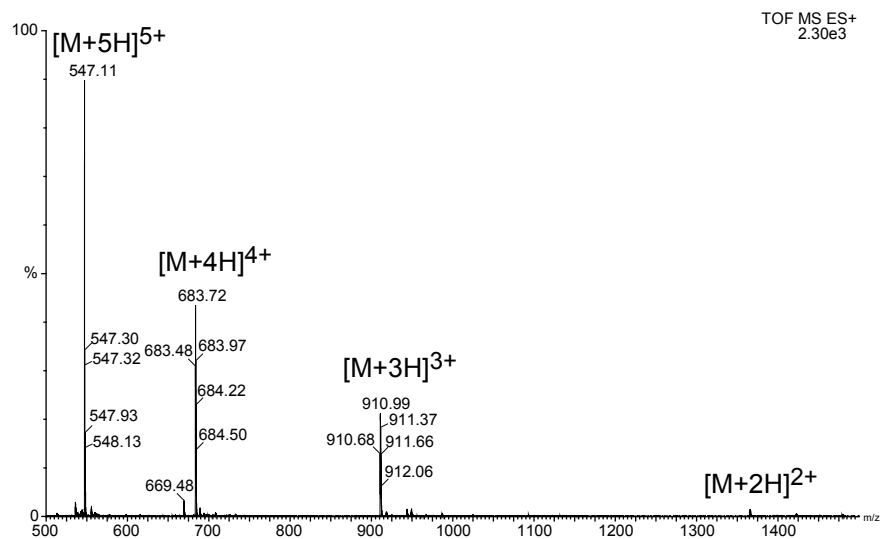
Analytical RP-HPLC chromatograph and mass spectrum (ESI-MS)



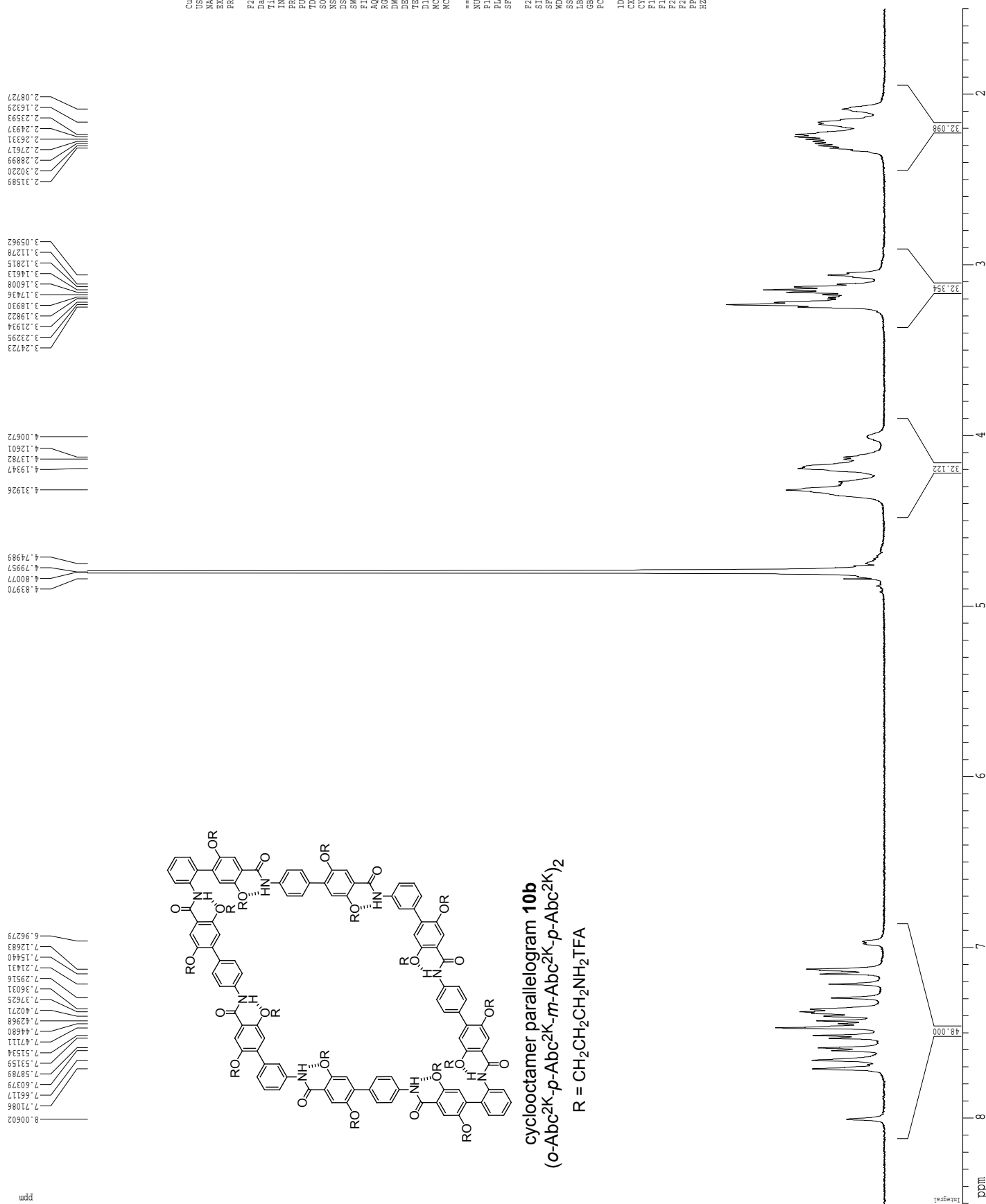
(a) Analytical RP-HPLC (5-50% acetonitrile with 0.1% TFA over 20 min, $\lambda = 214$)



(b) ESI Mass spectrum. (Calcd exact mass for C₁₅₂H₁₈₄N₂₄O₂₄ [M] = 2729.39)



¹H NMR (500 MHz, 298 K, D₂O) spectrum of cyclooctamer parallelogram **10b**



Supporting Information

```

Current Data Parameters
=====
USER      :
NAME      :
EXPNO     : 1
PROCNO    : 1

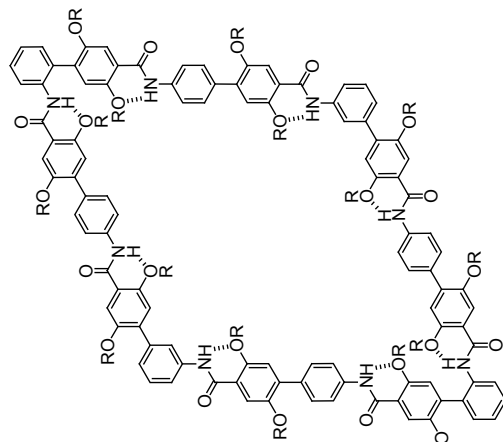
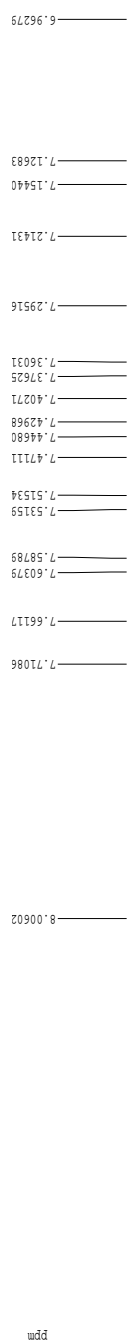
F2 - Acquisition Parameters
=====
Date_     : 20070505
Time      : 23.43
INSTRUM   : gm500
PROBHD    : 5 mm broadband
PULPROG   : zgpg30
TD         : 81728
SOLVENT   : D2O
DS         : 1
AQ         : 0.098043 Hz
RG         : 512
DW         : 62.400 usec
DE         : 6.00 usec
TE         : 298.0 K
MCREST    : 0.1000000 sec
MORPK     : 0.0150000 sec

===== CHANNEL f1 =====
NUC1      : 12.00 usec
P1        : -3.00 dB
SFO1      : 499.9334955 MHz

F2 - Processing parameters
=====
SI         : 65536
SF         : 499.9330032 MHz
WDW        : EM
SSB        : 0
LB         : 0.30 Hz
GB         : 0
PC         : 4.00

ID NMR plot parameters
=====
CY         : 22.80 cm
CX         : 157.94 cm
F1P        : 8.500 ppm
F1         : 4.049.40 Hz
F2P        : 1.500 ppm
F2         : 749.50 Hz
PRGCM      : 0.30702 ppm/cm
HZCM       : 153.48729 Hz/cm
  
```

C. M. Gothard and J. S. Nowick

¹H NMR (500 MHz, 298 K, D₂O) spectrum of cyclooctamer parallelogram **10b** (aromatic region)cyclooctamer parallelogram **10b**
(*o*-Abc²K-*p*-Abc²K-*m*-Abc²K-*p*-Abc²K)₂R = CH₂CH₂CH₂NH₂TFA

```

Current Data Parameters
USER      egecha
NAME      09-11-17
PROBHD    5 mm broadband
PULPROG   zg30
TD         81728
SOLVENT    D2O
NS         16
DS         2
AQ         0.0312872 Hz
RG         0.6398043 Hz
EXRES     5.0398774 sec
RG         512
DM         62.400 usec
DE         6.00 usec
TE         298.0 K
D1         0.10000000 sec
D11        0.00000000 sec
MCREST     0.00000000 sec
MORRK      0.01500000 sec

===== CHANNEL f1 =====
NUC1       1H
P1         12.00 usec
PL1        -1.00 dB
SFO1       499.9334995 MHz

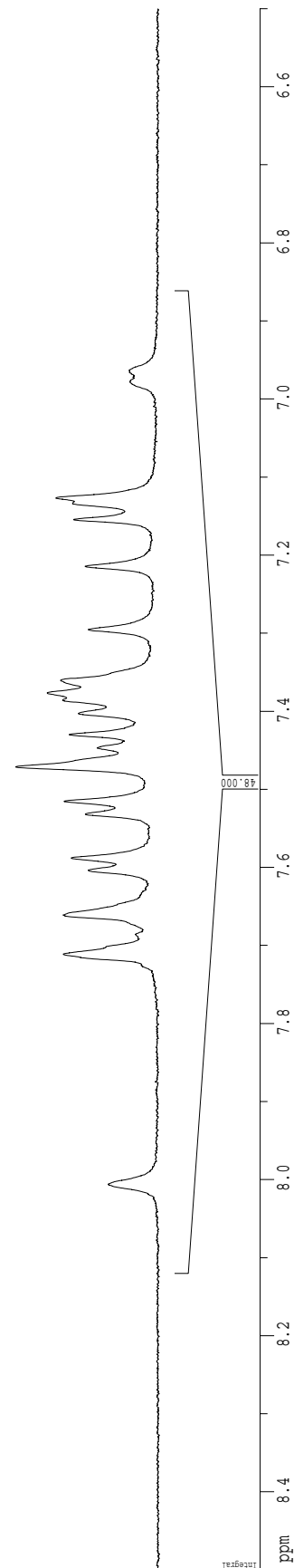
F2 - Acquisition Parameters
Date_      20070505
Time       23.43
INSTRUM    gm500
PROBHD     5 mm broadband
PULPROG    zg30
TD         81728
SOLVENT     D2O
NS         16
DS         2
AQ         0.0312872 Hz
RG         0.6398043 Hz
EXRES     5.0398774 sec
RG         512
DM         62.400 usec
DE         6.00 usec
TE         298.0 K
D1         0.10000000 sec
D11        0.00000000 sec
MCREST     0.00000000 sec
MORRK      0.01500000 sec

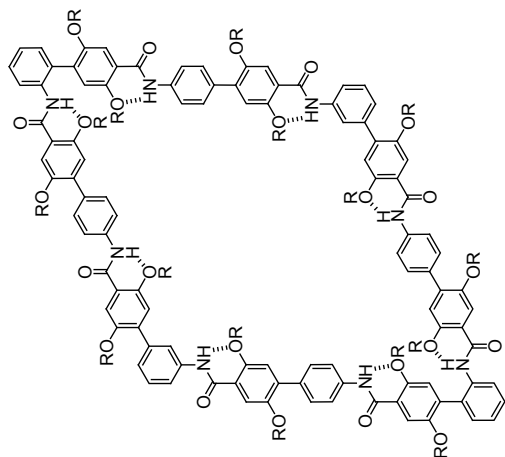
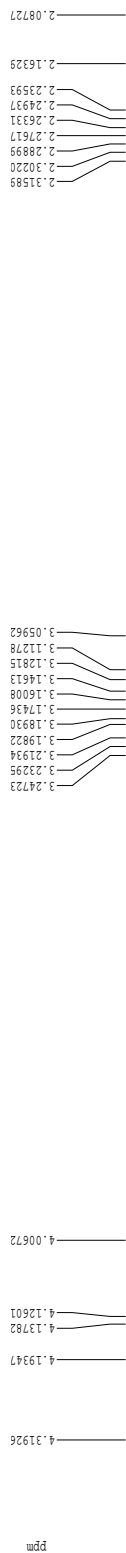
===== CHANNEL f1 =====
NUC1       1H
P1         12.00 usec
PL1        -1.00 dB
SFO1       499.9334995 MHz

F2 - Processing parameters
S1         65536
SF         499.9300032 MHz
WDW        EM
SSB        0
LB         0.30 Hz
GB         0
PC         4.00

1D NMR plot parameters
CX         1272.80 cm
CY         1272.80 cm
CZ         18.5000 cm
F1P        4249.46 Hz
F2P        6.5000 ppm
F3P        3249.54 Hz
PPMCM      0.08772 ppm/cm
HZCM       43.85351 Hz/cm

```



¹H NMR (500 MHz, 298 K, D₂O) spectrum of cyclooctamer parallelogram **10b** (aliphatic region)

cyclooctamer parallelogram **10b**
 (*o*-Abc²K-*p*-Abc²K-*m*-Abc²K-*p*-Abc²K)₂

R = CH₂CH₂CH₂NH₂TFA

```

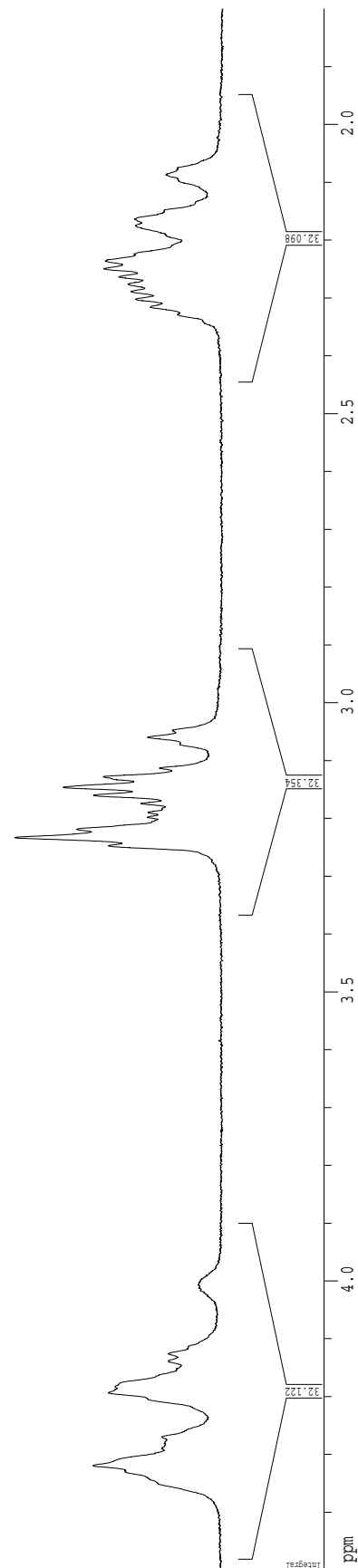
Current Data Parameters
USER          egecha
NAME         09-11-37
PROCNO       1
PRCNO       1

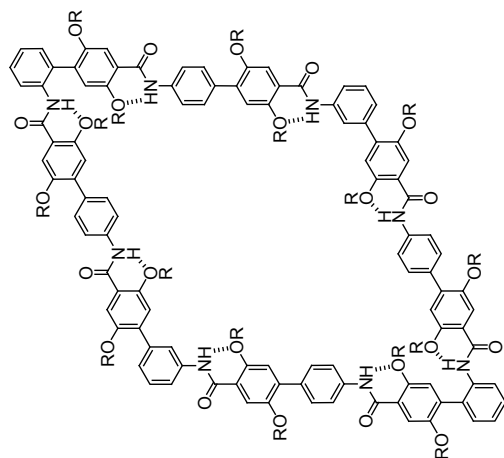
F2 - Acquisition Parameters
Date_        20070505
Time_        23.43
INSTRUM     gm500
PROBHD      5 mm broadband
PULPROG     zg30
TD           81728
SOLVENT     D2O
NS           16
DS           2
AQ           0.0312872 Hz
RG           0.636843 Hz
XRES        5.0398774 sec
AQ           512
DM           62.400 usec
DE           6.00 usec
TE           298.0 K
D1           0.10000000 sec
MCREST      0.00000000 sec
MORRK       0.01500000 sec

===== CHANNEL f1 =====
NUC1         1H
P1           12.00 usec
PL1          -1.00 dB
GB1          0
SFO1         499.9334995 MHz

F2 - Processing parameters
SI           65536
SF           499.9300032 MHz
WDW          EM
SSB          0
LB           0.30 Hz
GB           0
PC           4.00

1D NMR plot parameters
CX           127.80 cm
CY           127.80 cm
F1P          14.506 cm
F2P          2249.69 Hz
P1P          1.800 ppm
P2P          899.87 Hz
PPHMC        0.11842 ppm/cm
HZXCN        59.20224 Hz/cm
  
```



¹H NMR (500 MHz, 330 K, D₂O) spectrum of cyclooctamer parallelogram 10b

cyclooctamer parallelogram 10b
 (o-Abc²K-p-Abc²K-m-Abc²K-p-Abc²K)₂

R = CH₂CH₂CH₂NH₂TFA

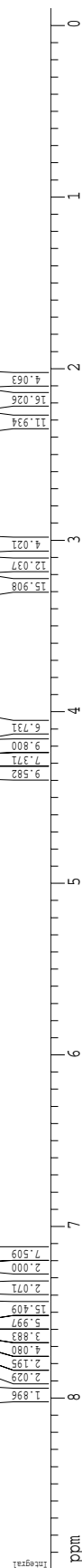
Current Data Parameters
 USER cgccha
 NAME c3f-11-37
 EXPNO 4
 PROCNO 1

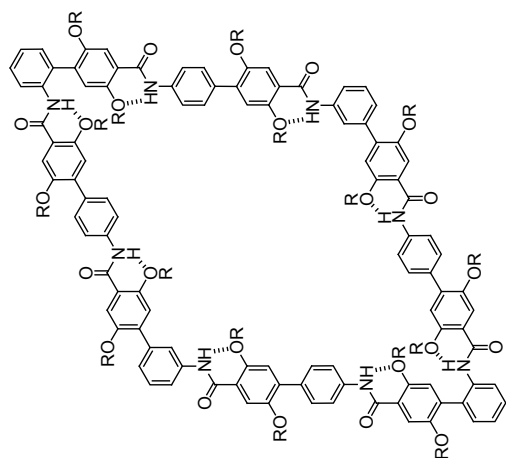
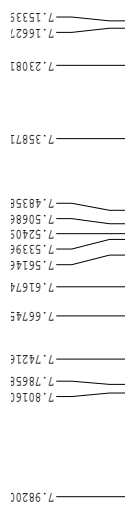
F2 - Acquisition Parameters
 Date_ 20070506
 Time 0.15
 INSTRUM spect
 PROBHD 5 mm hxczcp130
 PULPROG zgpg30
 TD 81728
 SOLVENT D₂O
 NS 32
 DS 2
 SMH 8012.820 Hz
 FIDRES 0.098043 Hz
 AQ 5.0998774 sec
 RG 512
 DW 62.400 usec
 DE 6.00 usec
 TE 330.0 K
 KW 0.1000000 sec
 MWDW 0.0000000 sec
 MORK 0.01500000 sec

==== CHANNEL F1 =====
 NUC1 1H
 P1 12.00 usec
 F1 499.934995 MHz
 SFO1 499.934995 MHz

F2 - Processing parameters
 S1 6556
 SF 499.930194 MHz
 GWDW 0
 ASB 0
 GB 0.30 Hz
 CB 0
 PC 4.00

ID NMR plot parameters
 CX 22.80 cm
 CY 157.95 cm
 FIP 9.000 ppm
 F1 4499.37 Hz
 F2 -0.100 ppm
 FWHM 0.35912 ppm/cm
 HZCN 199.53357 Hz/cm



¹H NMR (500 MHz, 330 K, D₂O) spectrum of cyclooctamer parallelogram **10b** (aromatic region)

cyclooctamer parallelogram **10b**
(*o*-Abc²K-*p*-Abc²K-*m*-Abc²K-*p*-Abc²K)₂

R = CH₂CH₂CH₂NH₂TFA

```

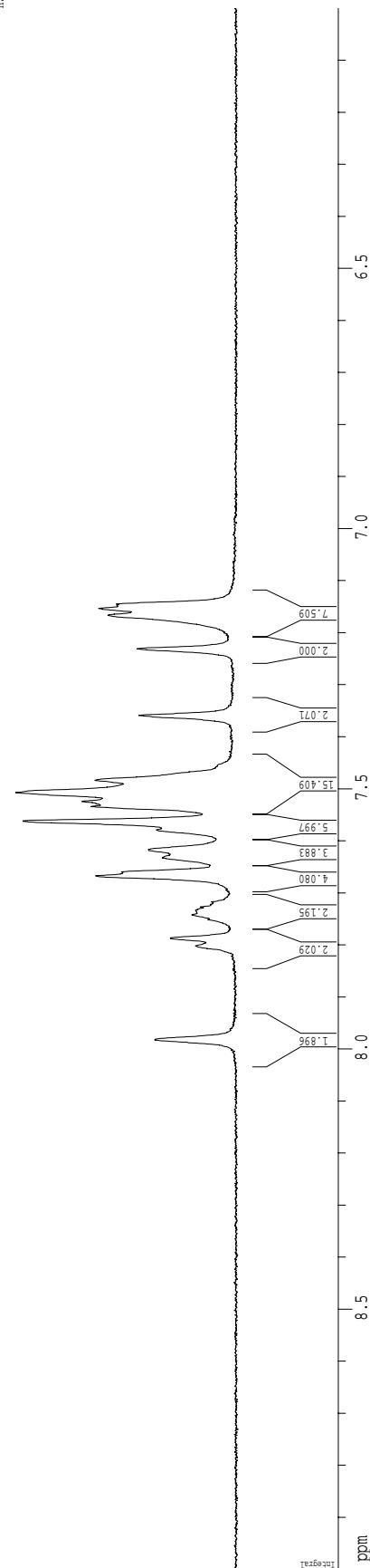
Current Data Parameters
=====
USER          cociba
NAME          cg-11-37
EXNO         4
PROCNO        1

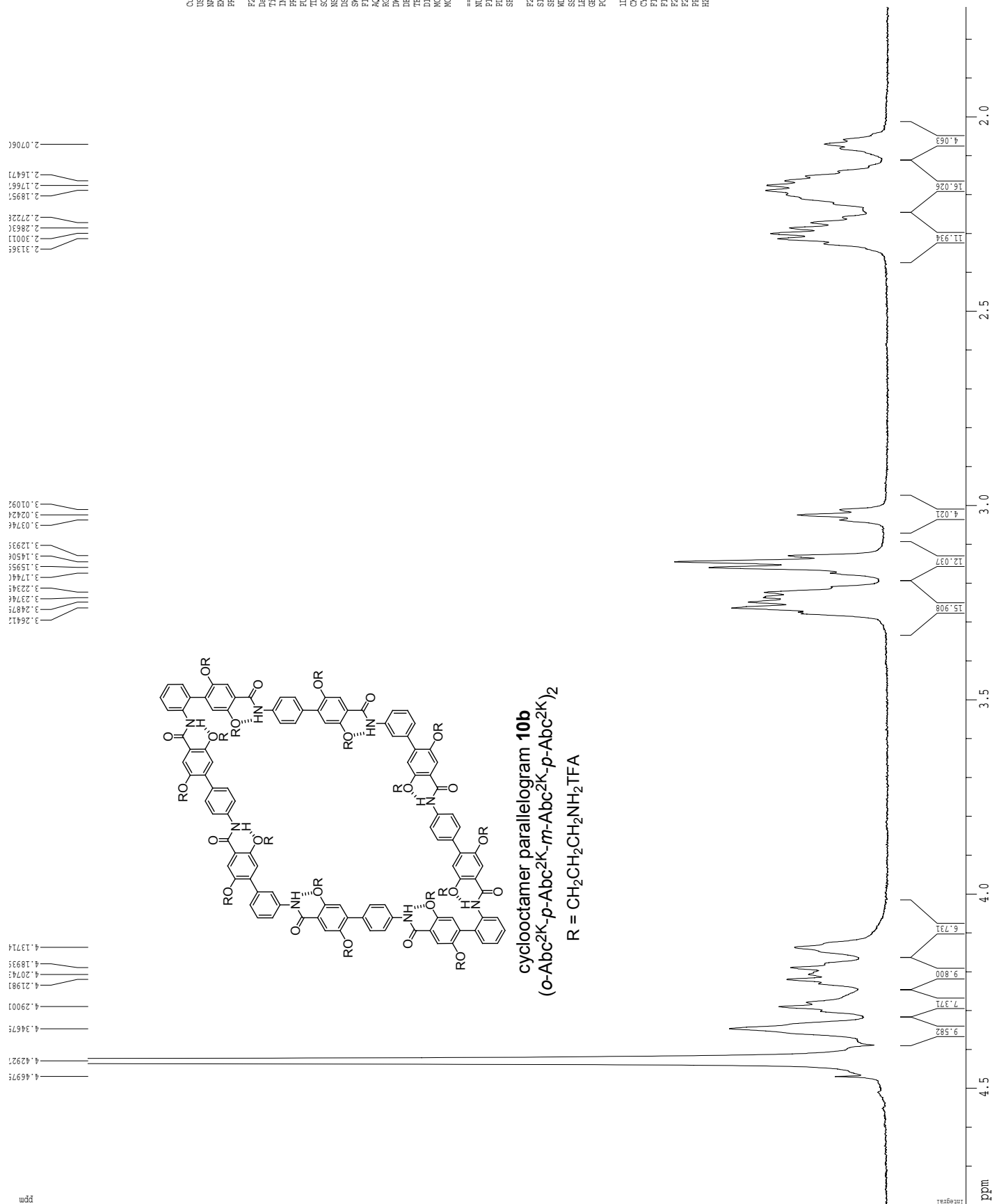
F2 - Acquisition Parameters
=====
Date_         20070506
Time          0.15
INSTRUM      gm500
PROBHD       5 mm broadband
PULPROG      zg30
TD           65536
SOLVENT      D2O
DS           32
NS           2
DS           2
SHE          8012.820 Hz
FIDRES       0.058043 Hz
AQ           5.0398774 sec
RG           512
DW           62.400 usec
DE           6.00 usec
TE           330.0 K
D1           0.1000000 sec
ACQRES1      0.0000000 sec
MCWREK       0.01500000 sec

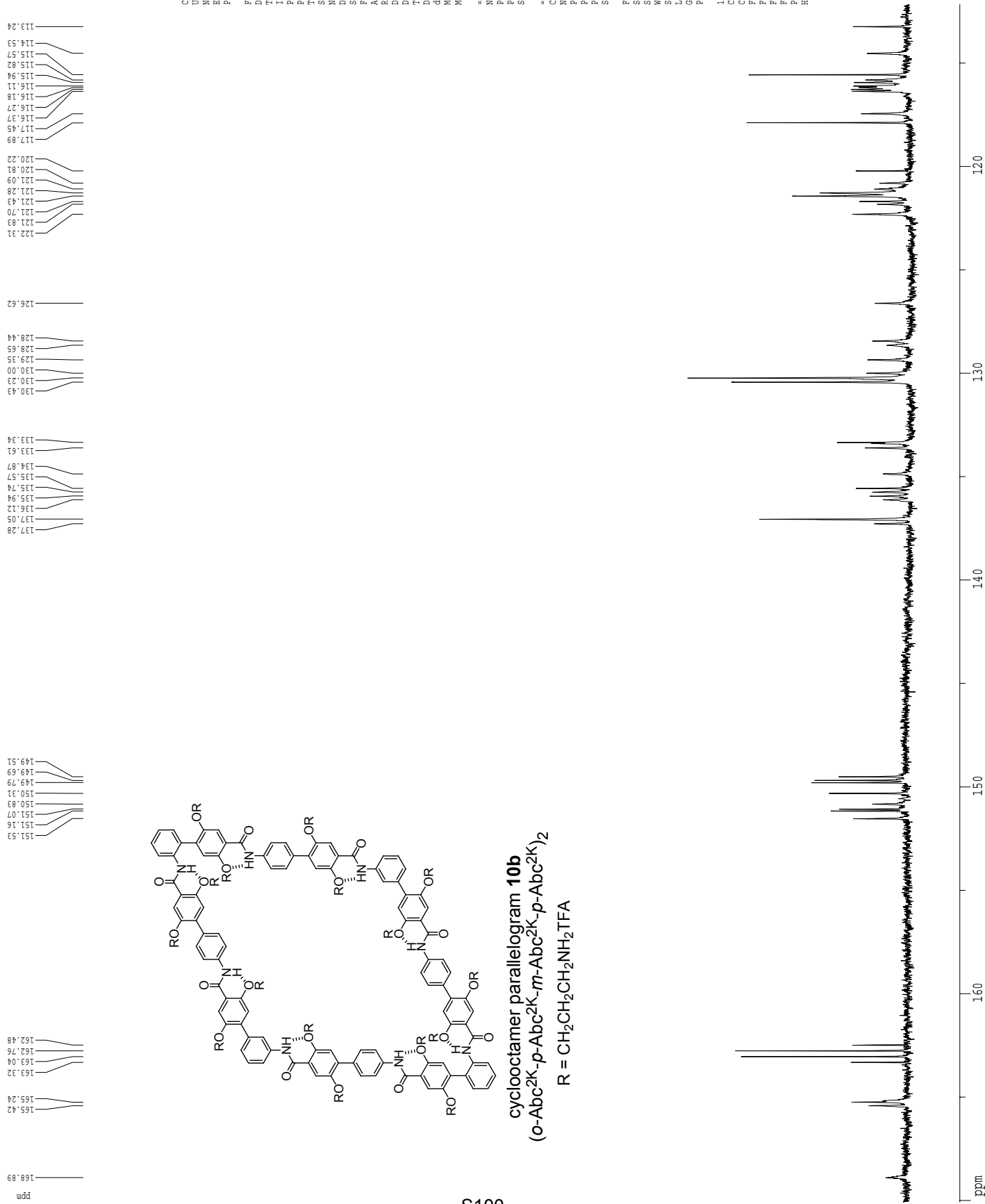
===== CHANNEL f1 =====
NUC1          1H
P1           12.00 usec
PL1          -3.00 dB
SFO1         499.9334995 MHz

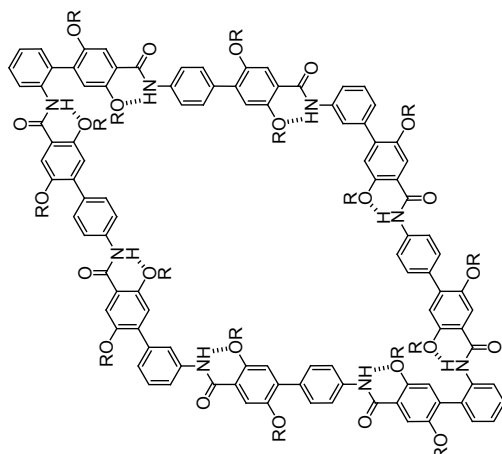
F2 - Processing parameters
=====
SI           65536
SF           499.9301934 MHz
WDW          EM
SSB          0
LB           0.30 Hz
GB           0
PC           4.00

1D NMR plot parameters
=====
CX           22.80 cm
CY           157.95 cm
FIP          9.000 ppm
F1           4459.37 Hz
F2           6.000 ppm
F2           2999.58 Hz
PWCW        0.13158 ppm/cm
HZCW        65.78029 Hz/cm
  
```



¹H NMR (500 MHz, 330 K, D₂O) spectrum of cyclooctamer parallellogram **10b** (aliphatic region)

¹³C NMR (125 MHz, 320 K, D₂O) spectrum of cyclooctamer parallelogram **10b** (aromatic region)

^{13}C NMR (125 MHz, 320 K, D_2O) spectrum of cyclooctamer parallelogram **10b** (carbonyl region)

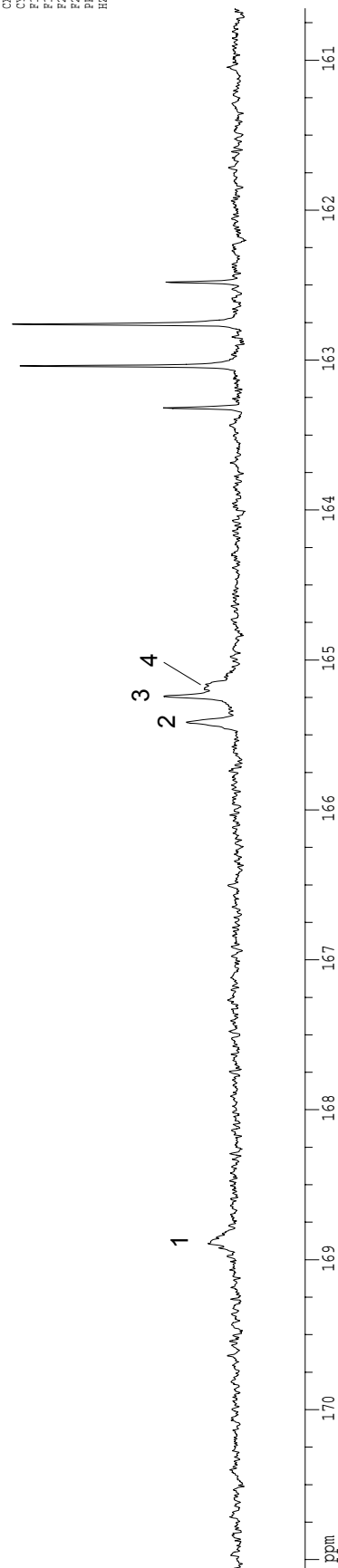
cyclooctamer parallelogram **10b**
 (*o*-Abc²K-*p*-Abc²K-*m*-Abc²K-*p*-Abc²K)₂

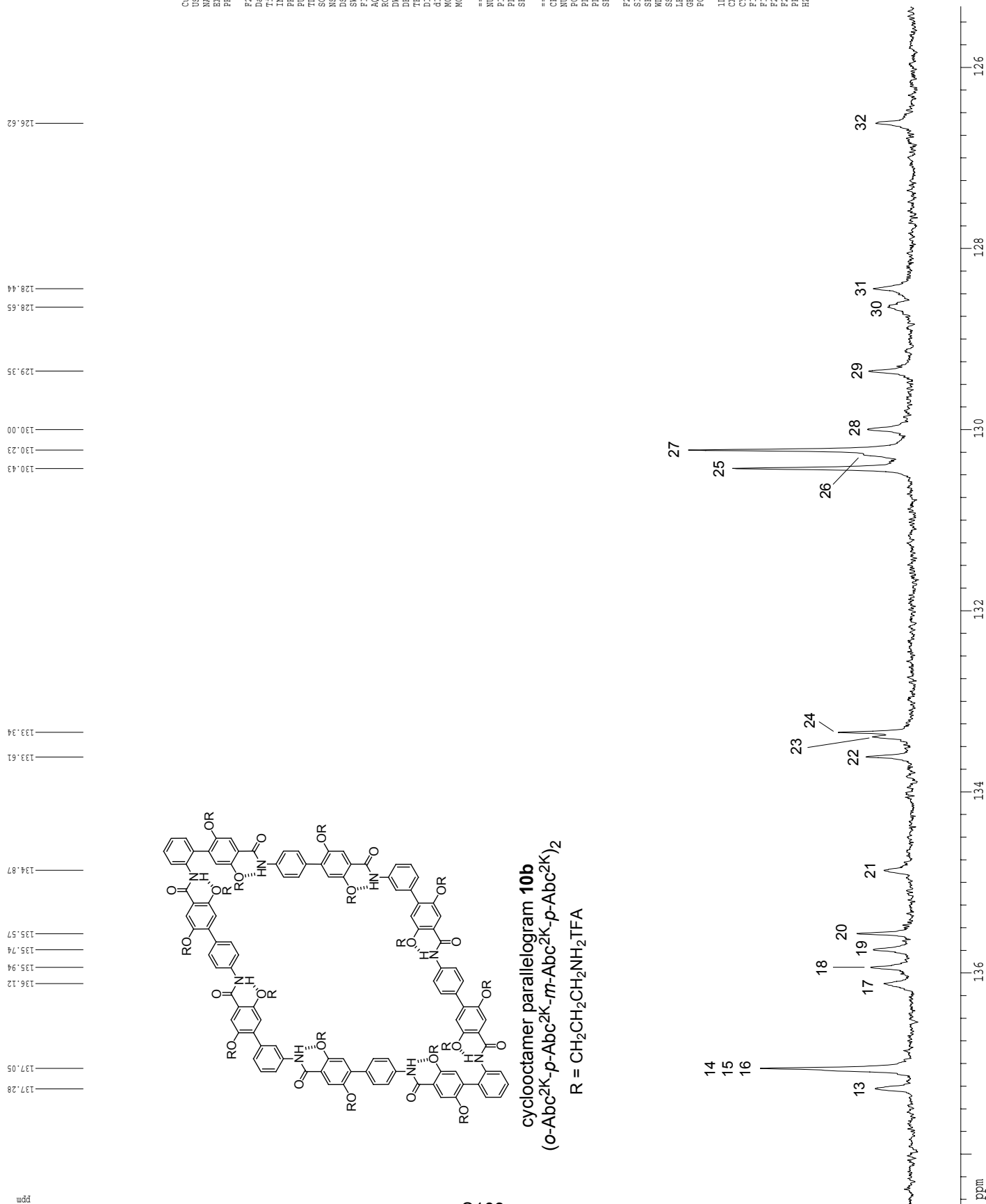
R = $\text{CH}_2\text{CH}_2\text{CH}_2\text{NH}_2\text{TFA}$

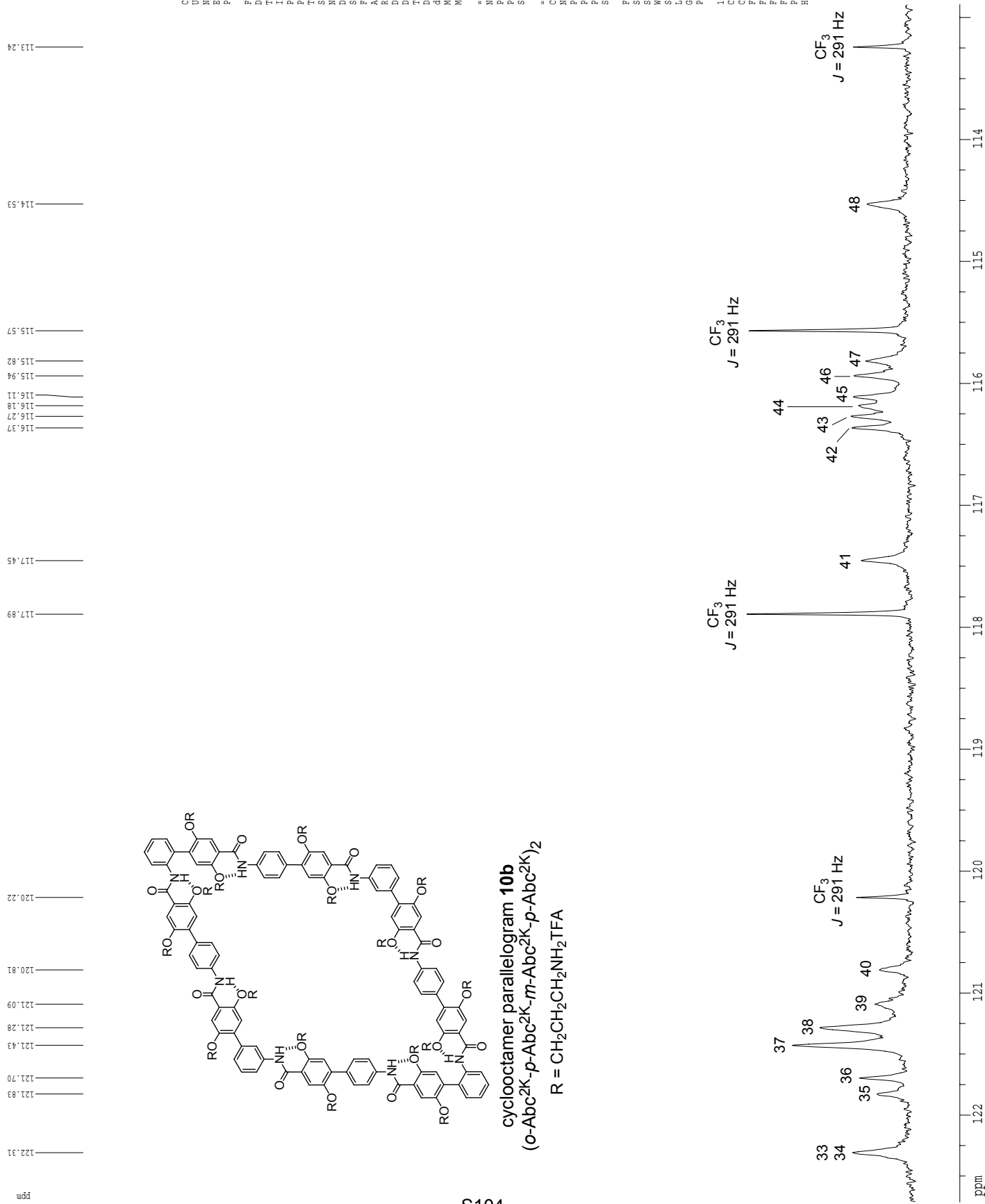
```

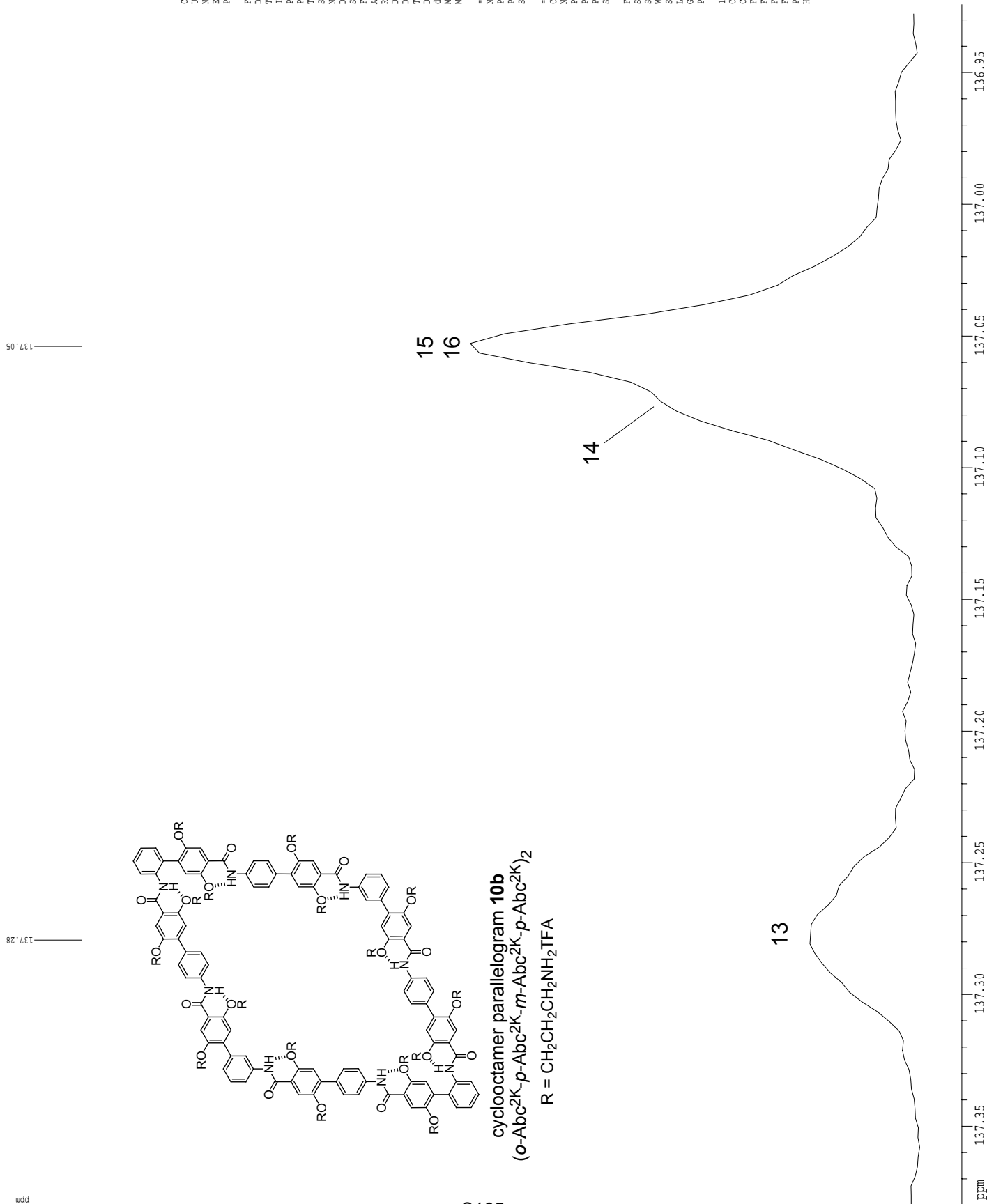
Current Data Parameters
USBR          csodia
NAME          09-11-17
PROCNO       12
PROBNO       1
PROCNO       1
F2 - Acquisition Parameters
Date_         20070517
Time_        1.34
INSTRUM      cryo500
PROBHD       5 mm CPTCI 1H-
PULPROG      zgpg30
TD           65418
SOLVENT      D2O
NS           14000
DS           4
SWH          30303.031 Hz
FIDRES       0.463222 Hz
AQ           1.0794470 sec
RG           14596.5
DM           16.500 usec
DE           6.00 usec
TE           300.2 K
D1           0.25000000 sec
d11          0.03000000 sec
MGSTEST      0.00000000 sec
MORFEC       0.01500000 sec
===== CHANNEL F1 =====
NUC1         13C
P1           15.00 usec
PL1          -1.00 dB
SFO1         125.7942548 MHz
===== CHANNEL E2 =====
CPDPRG2      waltz16
NUC2         1H
PCPD2        100.00 usec
PL2          1.60 dB
PL12         23.54 dB
SFO2         500.2225011 MHz
F2 - Processing parameters
SI           65836
SF           125.7804190 MHz
WDW          EM
SSB          0
LB           1.00 Hz
GB           0
PC           2.00
1D NMR plot parameters
CX           22.80 cm
CY           8.47 cm
FIP          171.069 ppm
FL           21517.09 Hz
FZP          160.657 ppm
WDW          EM
SSB          0
LB           1.00 Hz
GB           0
PC           2.00
HEXCH       57.43966 Hz/cm
  
```

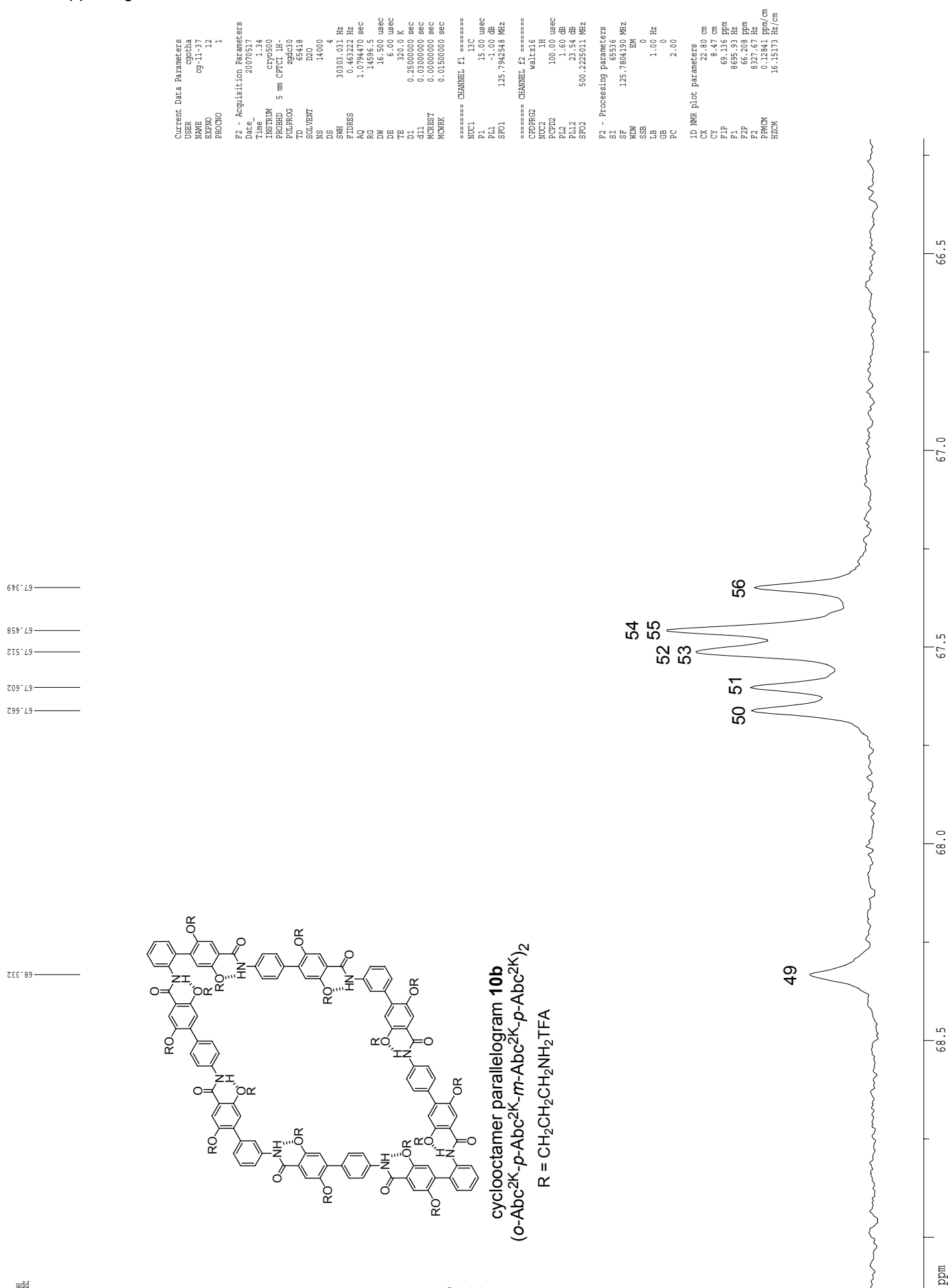
CO_2CF_3
 $J = 35.0 \text{ Hz}$

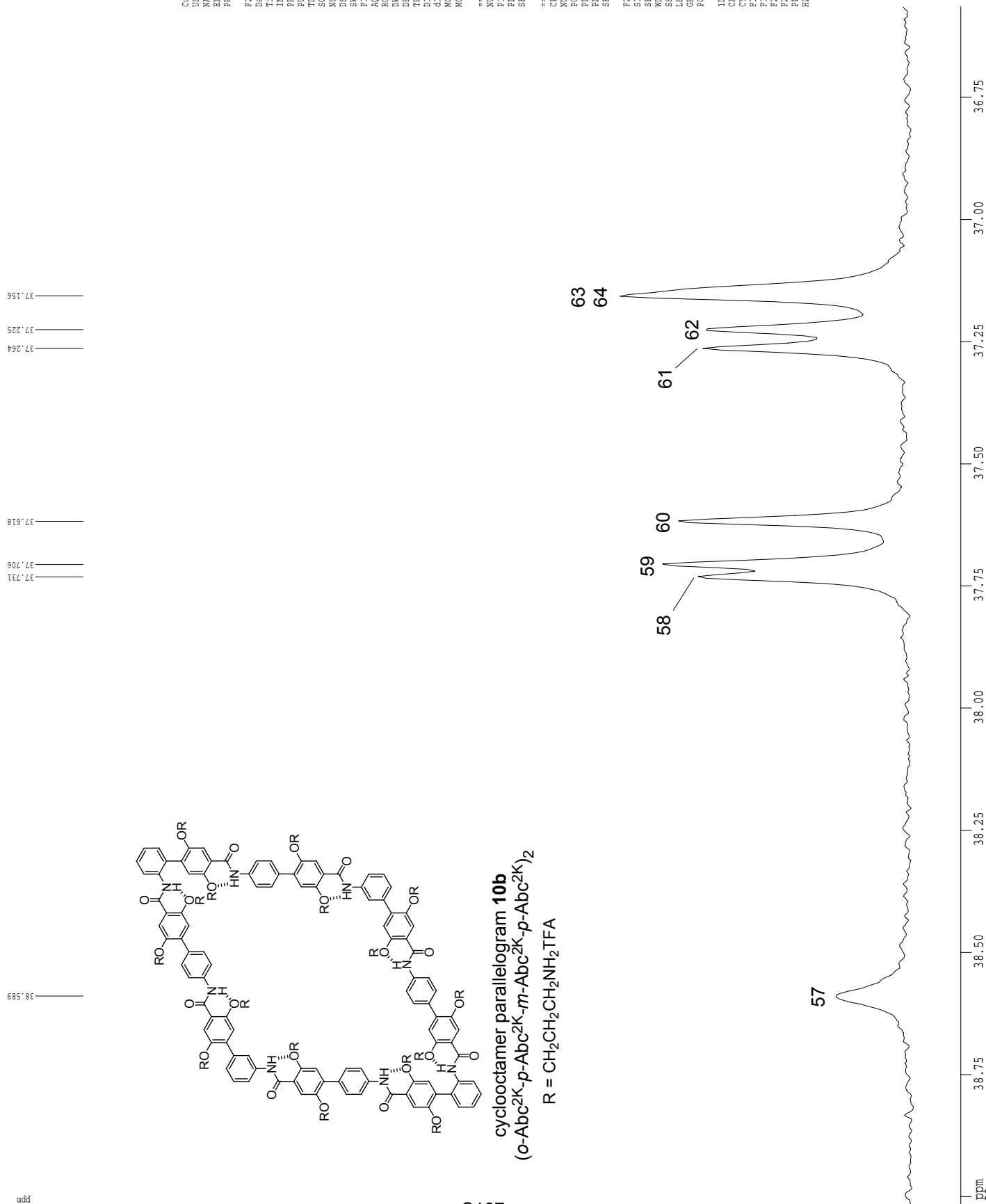


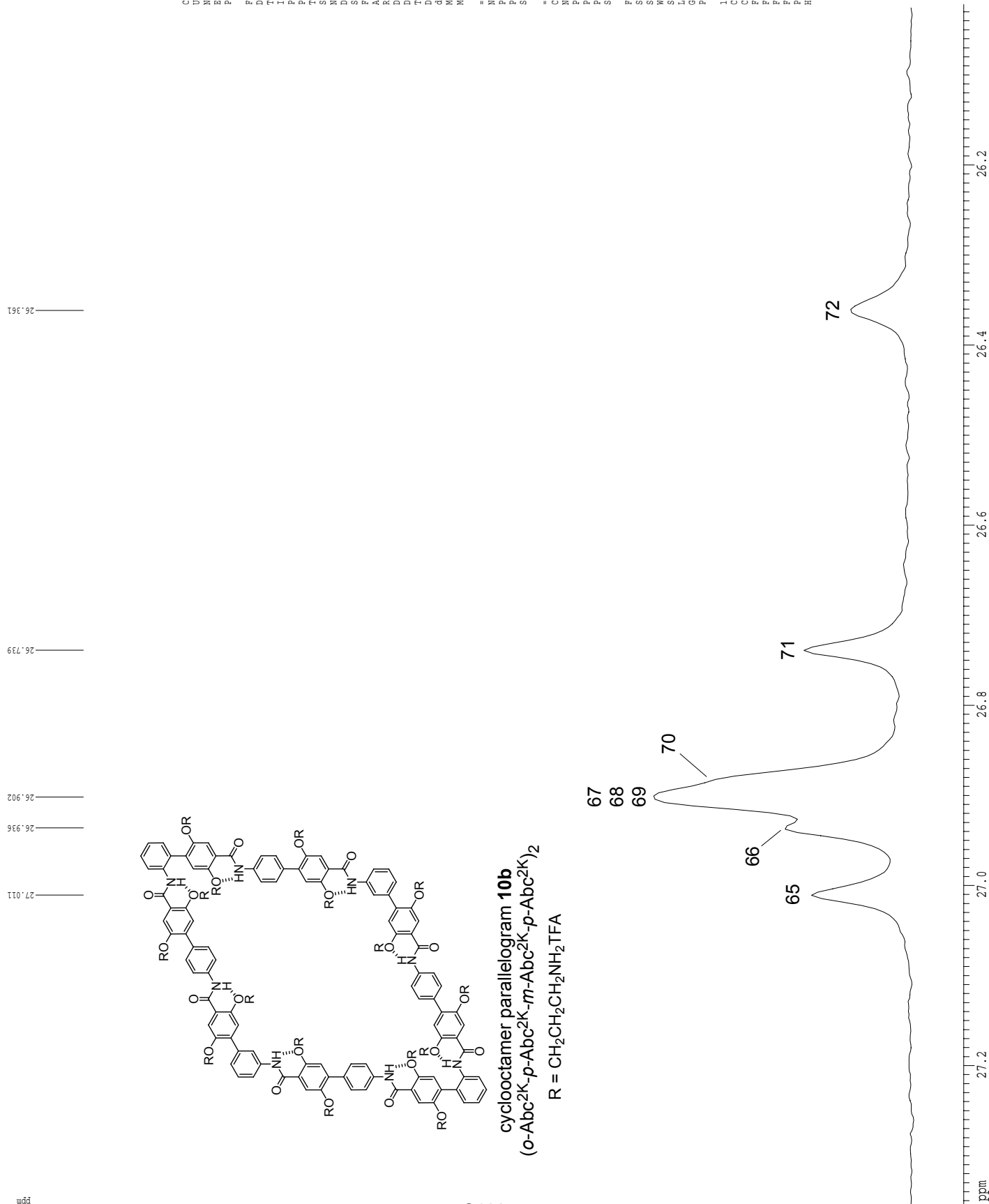
^{13}C NMR (125 MHz, 320 K, D_2O) spectrum of cyclooctamer **10b** (aromatic region expansion 2)

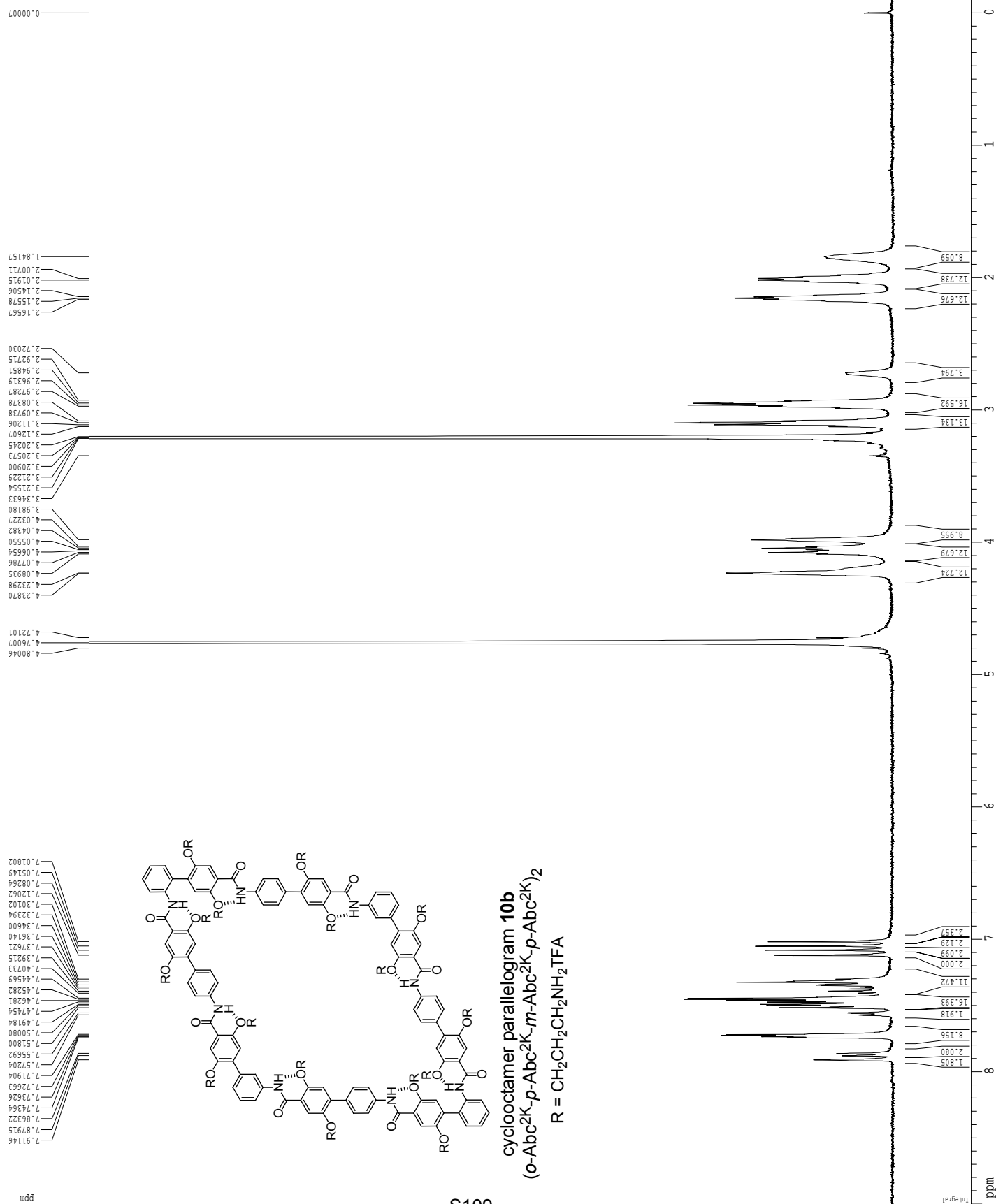
^{13}C NMR (125 MHz, 320 K, D_2O) spectrum of cyclooctamer parallelogram **10b** (aromatic region expansion 3)

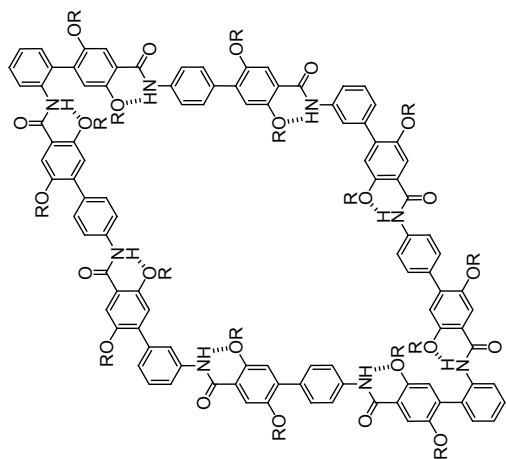
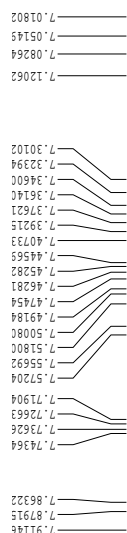
^{13}C NMR (125 MHz, 320 K, D_2O) spectrum of cyclooctamer parallelogram **10b** (aromatic region expansion 3A)

¹³C NMR (125 MHz, 320 K, D₂O) spectrum of cyclooctamer parallelogram **10b** (aliphatic region expansion 1)

¹³C NMR (125 MHz, 320 K, D₂O) spectrum of cyclooctamer parallelogram **10b** (aliphatic region expansion 2)

¹³C NMR (125 MHz, 320 K, D₂O) spectrum of cyclooctamer parallelogram **10b** (aliphatic region expansion 3)

¹H NMR (500 MHz, 298 K, CD₃OD) spectrum of cyclooctamer parallelogram 10b

¹H NMR (500 MHz, 298 K, CD₃OD) spectrum of cyclooctamer parallelogram **10b** (aromatic region)

cyclooctamer parallelogram **10b**
 (o-Abc²K-p-Abc²K-m-Abc²K-p-Abc²K)₂

R = CH₂CH₂CH₂NH₂TFA

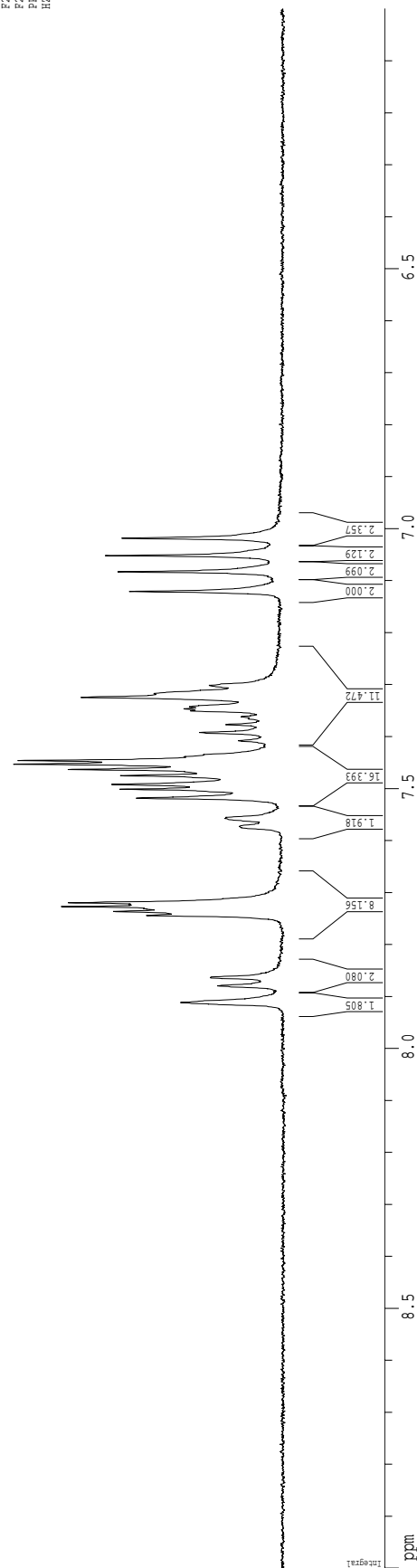
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Current Data Parameters
USER          cpocha
NAME          09-11-37
EXNO         5
PROCNO       1

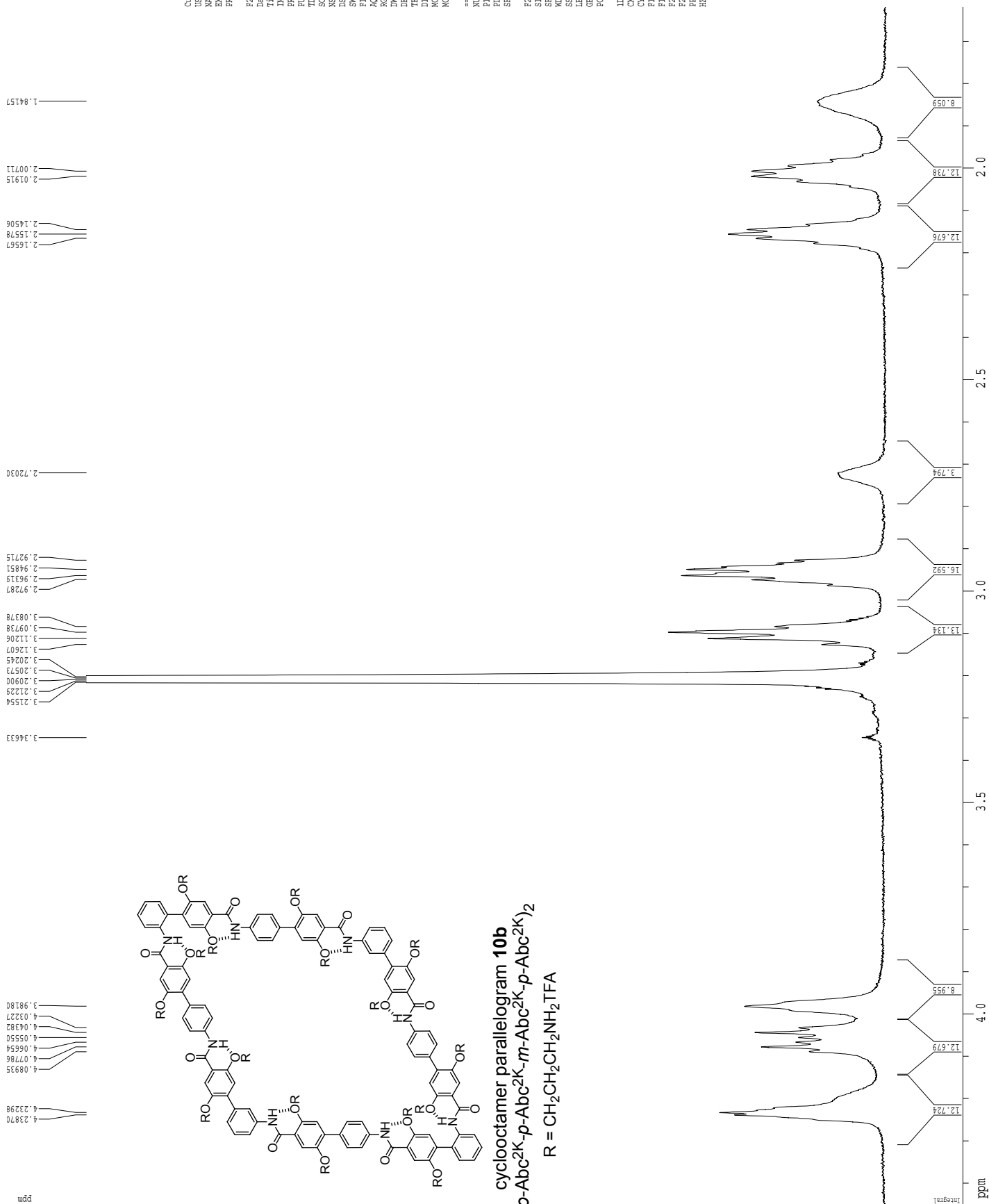
F2 - Acquisition Parameters
Date_        20070506
Time         0.36
INSTRUM      gh500
PROBHD       5 mm broadband
PULPROG      zgpg30
F1          499.913
SOLVENT      CD3OD
NS           16
DS           2
SWH          8012.820 Hz
FIDRES       0.098043 Hz
AQ           5.0598774 sec
RG           512
DM           62.400 usec
DE           6.00 usec
TE           298.0 K
D1           0.10000000 sec
RG2          0.00000000 sec
RG3          0.01500000 sec
===== CHANNEL f1 =====
NUC1         13C
P1           12.00 usec
PL1          -3.00 dB
SFO1         499.9134995 MHz

F2 - Processing parameters
SI           65536
SF           499.91300681 MHz
WDW          EM
SSB          0
GB           0
PC           4.00

ID NMR Plot parameters
CX           22.80 cm
CY           246.39 cm
F1P         9.000 ppm
F1          44499.37 Hz
F2P         6.000 ppm
F2          2999.58 Hz
PPMCM       0.13158 ppm/cm
HZCM        65.78027 Hz/cm
  
```



¹H NMR (500 MHz, 298 K, CD₃OD) spectrum of cyclooctamer **10b** (aliphatic region)



Supporting Information

Current Data Parameters
 USER cyobha
 NAME C9-11-37
 EXPNO 5
 PROCNO 1

F2 - Acquisition Parameters
 Date_ 20070816
 Time 09:32
 INSTRUM g500
 PROBRD 5 mm broadband
 PULPROG zg30
 TD 81728
 SOLVENT CD3OD
 NS 16
 DS 2
 SWH 8012.820 Hz
 FIDRES 0.098043 Hz
 AQ 5.0998774 sec
 RG 512
 RW 62.400 usec
 DE 0.000000 sec
 TE 298.0 K
 D1 0.1000000 sec
 MCREST 0.0000000 sec
 MCWREK 0.0150000 sec

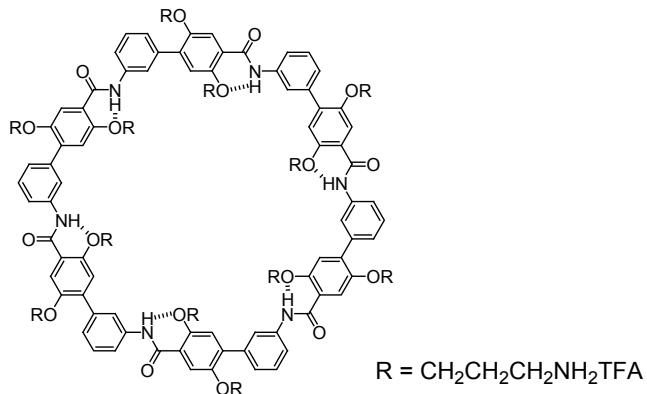
===== CHANNEL F1 =====
 NUC1 1H
 P1 12.00 usec
 PL1 -3.00 dB
 SFO1 499.933495 MHz

F2 - Processing parameters
 SI 32768
 SF 499.930625 MHz
 EQ 0
 EM 0
 SSB 0
 LB 0.30 Hz
 GB 0
 PC 4.00

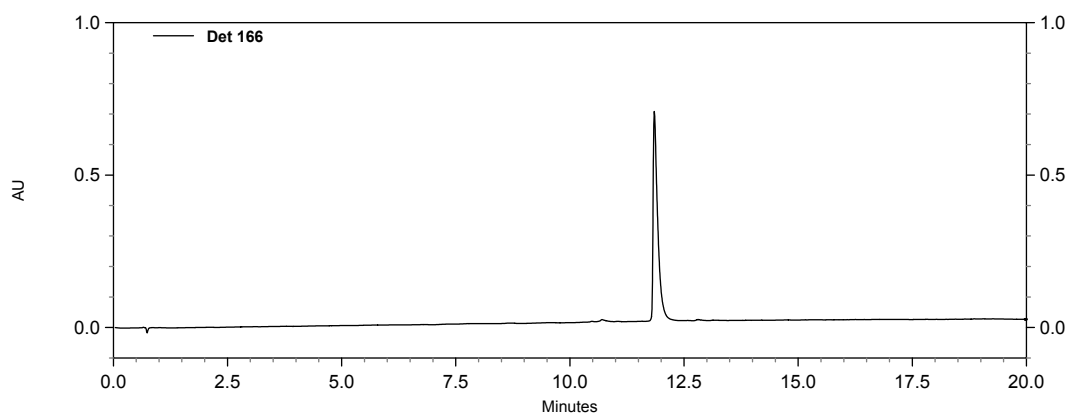
1D NMR plot parameters
 CX 22.80 cm
 CT 246.39 cm
 FIP 4.451 EPM
 F1 2225.02 Hz
 F2 806.619 EPM
 F3 806.619 EPM
 F4 0.72418 EPM
 FREQN 62.08149 Hz
 HZCN 62.08149 Hz

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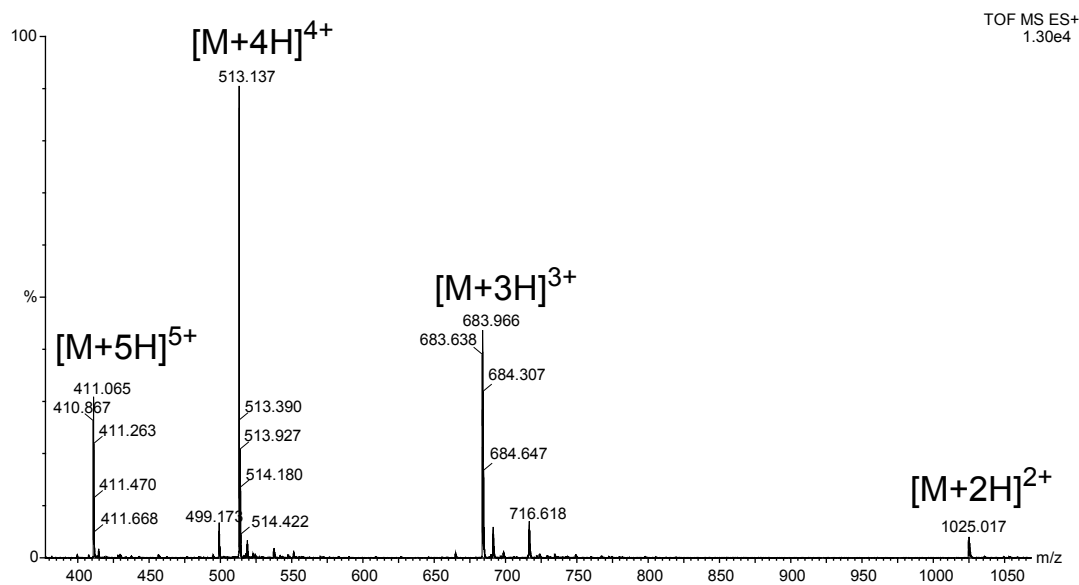
cyclohexamer ring: (*m*-Abc^{2K})₆ (**11a**)
Analytical RP-HPLC chromatograph and mass spectrum (ESI-MS)



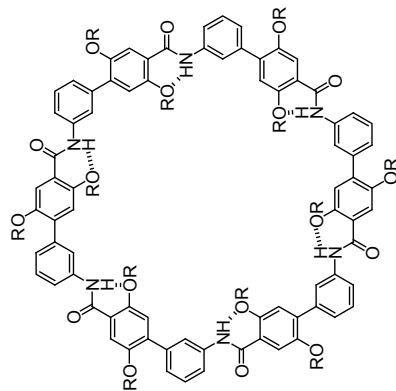
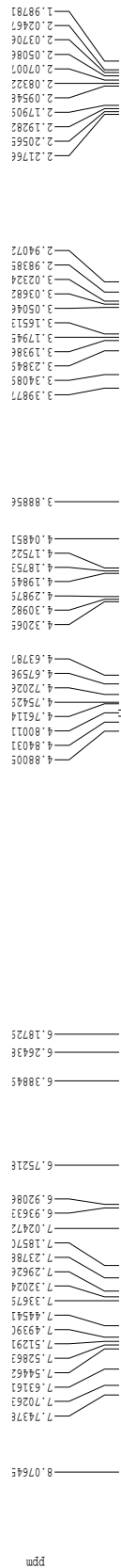
(a) Analytical RP-HPLC (5-50% acetonitrile with 0.1% TFA over 20 min, $\lambda = 214$)



(b) ESI Mass spectrum. (Calcd exact mass for C₁₁₄H₁₃₈N₁₈O₁₈ [M] = 2047.04)



¹H NMR (500 MHz, 298 K, D₂O) spectrum of cyclohexamer ring **11a**



cyclohexamer ring **11a**
(*m*-Abc^{2k})₆
R = CH₂CH₂CH₂NH₂TFA

Current Data Parameters
USER cgocha
NAME cg-7-195
EXPNO 6
PROCNO 1

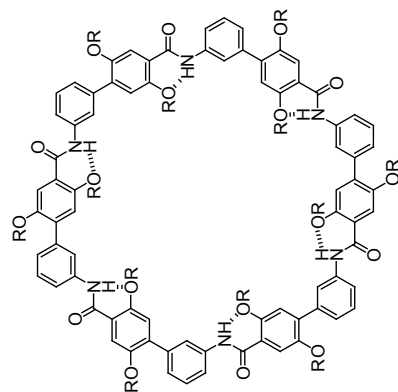
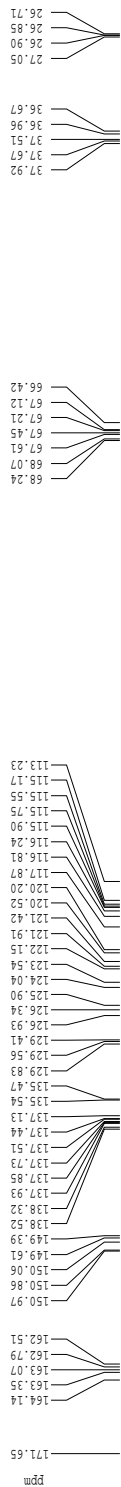
F2 - Acquisition Parameters
Date_ 20060115
Time 21.10
INSTRUM cgy500
PROBHD 5 mm CPCLP
PULPROG zg30
TD 81728
SOLVENT CDCl3
NS 32
DS 2
SWH 8012.820 Hz
FIDRES 0.098043 Hz
AQ 5.0998774 sec
RG 724.1
DM 62.400 usec
DE 6.00 usec
TE 298.0 K
AQC 0.1000000 sec
MORST 0.0000000 sec
MORPK 0.0150000 sec

***** CHANNEL f1 *****
NUC1 1H
P1 8.00 usec
PL1 1.60 dB
SFO1 500.2235015 MHz

F2 - Processing parameters
SI 6536
SF 500.2200045 MHz
WDW EM
SSB 0
GB 0.30 Hz
CB 0
PC 4.00

1D NMR plot parameters
CX 22.80 cm
CY 918.80 cm
F1P 8.500 ppm
F2P 4951.87 Hz
F3P 1.500 ppm
F4P 750.33 Hz
PPMXX 0.30702 ppm/cm
PPMYY 153.157631 Hz/cm

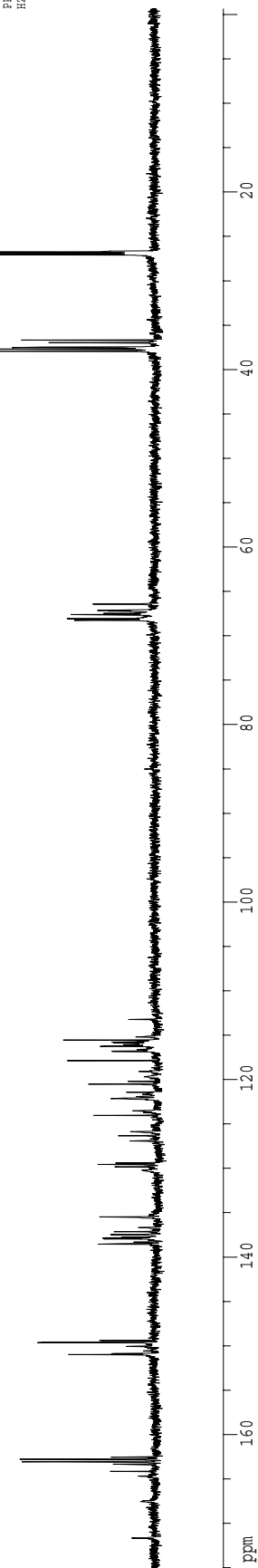


¹³C NMR (125 MHz, 298 K, D₂O) spectrum of cyclohexamer ring **11a**cyclohexamer ring **11a**
(*m*-Abc²K)₆R = CH₂CH₂CH₂NH₂TFA

```

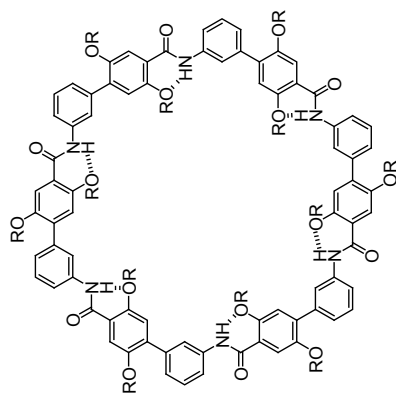
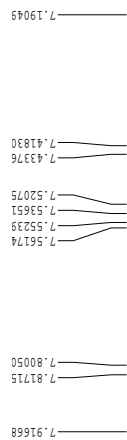
Current Data Parameters
USBR          cgdca
NAME          c9-11-73
PULPROG      4
PROCNO       1
=====
F2 - Acquisition Parameters
Date_        20070524
Time_        1.32
INSTRUM      cryo500
PROBHD       5 mm CPTCI 1H-
PULPROG      zgpg30
TD           65418
SOLVENT      D2O
NS           12000
DS           4
SWH          30303.031 Hz
FIDRES       0.463222 Hz
AQ           1.0794470 sec
RG           18390.4
DM           16.500 usec
DE           6.000 usec
TE           300.2 K
D1           0.25000000 sec
d11          0.03000000 sec
NOEBS1       0.00000000 sec
NOEBS2       0.01500000 sec
===== CHANNEL F1 =====
NUC1         13C
P1           15.00 usec
PL1          -1.00 dB
SFO1         125.7942548 MHz
===== CHANNEL E2 =====
CPDPRG2      waltz16
NUC2         1H
PCPD2        100.00 usec
PL2          1.60 dB
PL12         21.54 dB
SFO2         500.2225011 MHz
=====
F2 - Processing Parameters
SI           65536
SF           125.7804190 MHz
WDW          EM
SSB          0
LB           1.50 Hz
GB           0
PC           2.00
=====
1D NMR plot parameters
CX           22.80 cm
CY           3.41 cm
FIP          175.270 ppm
FL           22045.56 Hz
FZP          -0.652 ppm
FWD          3.945 Hz
SFOCM        7.7150 ppm/cm
HZCM         970.39559 Hz/cm

```



¹H NMR (500 MHz, 298 K, CD₃OD) spectrum of cyclohexamer ring **11a**



^1H NMR (500 MHz, 298 K, CD_3OD) spectrum of cyclohexamer ring **11a** (aromatic region)cyclohexamer ring **11a**
(*m*-Abc²K)₆R = $\text{CH}_2\text{CH}_2\text{CH}_2\text{NH}_2\text{TFA}$

```

Current Data Parameters
USER          cgecha
NAME         CG-1U-263
PROBHD      5 mm CPTCI 1H-
PULPROG     zg30
TD          81728
SOLVENT     CD3OD
AQ          8
AS          2
DS          8032.827 Hz
SWH         0.636843 Hz
FIDRES     5.039974 sec
AQ         10.1
RG         62.400 usec
DE         6.00 usec
TE         298.0 K
D1         0.10000000 sec
D11        0.00000000 sec
MCOREST    0.01500000 sec
MORRK      0.01500000 sec

===== CHANNEL f1 =====
NUC1        1H
P1          8.00 usec
PL1         0.00 dB
SFO1        500.2235013 MHz

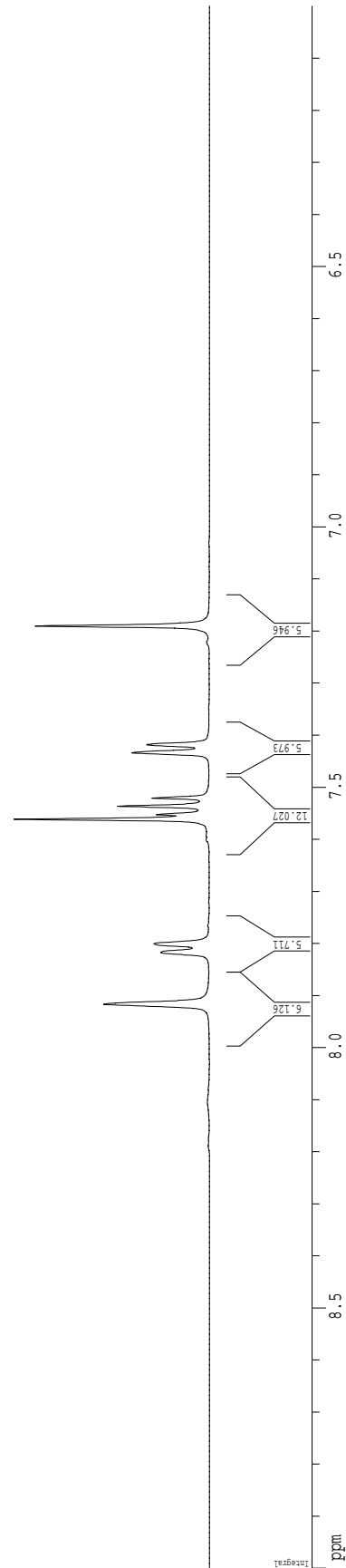
F2 - Acquisition Parameters
Date_       20061126
Time        2.14
INSTRUM     cryo500
PROBHD      5 mm CPTCI 1H-
PULPROG     zg30
TD          81728
SOLVENT     CD3OD
AQ          8
AS          2
DS          8032.827 Hz
SWH         0.636843 Hz
FIDRES     5.039974 sec
AQ         10.1
RG         62.400 usec
DE         6.00 usec
TE         298.0 K
D1         0.10000000 sec
D11        0.00000000 sec
MCOREST    0.01500000 sec
MORRK      0.01500000 sec

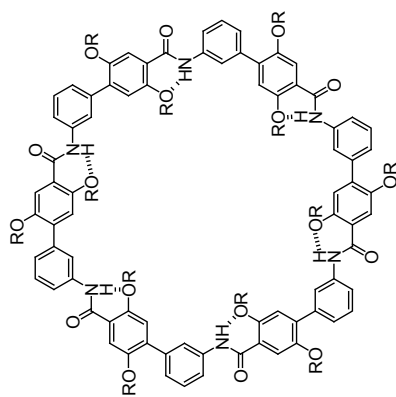
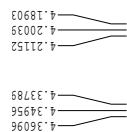
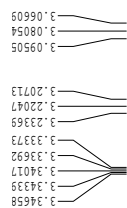
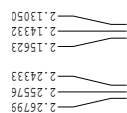
===== CHANNEL f1 =====
NUC1        1H
P1          8.00 usec
PL1         0.00 dB
SFO1        500.2235013 MHz

F2 - Processing parameters
S1          65536
SF          500.2200040 MHz
WDW         EM
SSB         0
LB          0.30 Hz
GB          0
PC          4.00

1D NMR plot parameters
CX          22.90 cm
CY          22.90 cm
CZ          22.90 cm
F1P         9.000 Hz
F2P         4501.98 Hz
P2P         6.000 ppm
F2          3001.32 Hz
PPMCM      0.13158 ppm/cm
HZCM       65.81842 Hz/cm

```



^1H NMR (500 MHz, 298 K, CD_3OD) spectrum of cyclohexamer ring **11a** (aliphatic region)cyclohexamer ring **11a**
(*m*-Abc 2 K) $_6$ R = $\text{CH}_2\text{CH}_2\text{CH}_2\text{NH}_2\cdot\text{TFA}$

```

Current Data Parameters
USER          cgecha
NAME         CG-1U-243
PROCNO       4
PRACNO       1

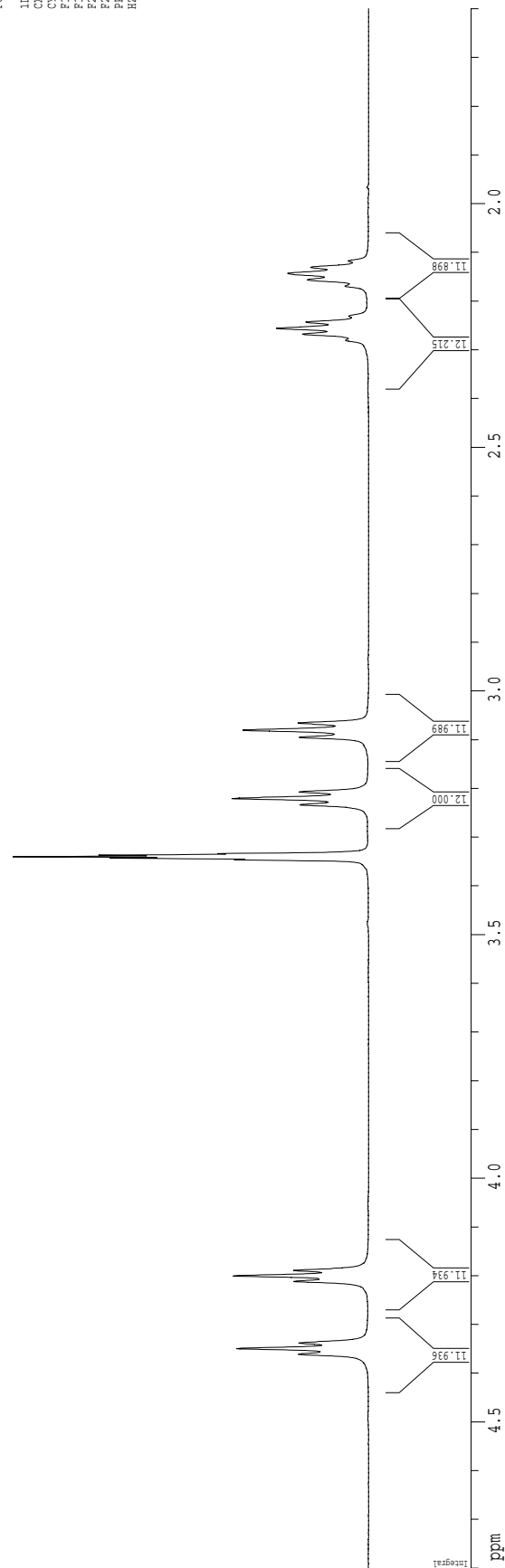
F2 - Acquisition Parameters
Date_        20061126
Time_        2.14
INSTRUM      crys500
PROBHD       5 mm CPTCI 1H-
PULPROG      zg30
TD           81728
SOLVENT      CD3OD
NS           8
DS           2
AQ           0.031870 Hz
RG           0.636040 Hz
FIDRES       5.039974 sec
AQ           10.1
RG           62.400 usec
DE           6.00 usec
TE           298.0 K
D1           0.10000000 sec
D11          0.00000000 sec
MCOREST      0.01500000 sec
MORRK        0.01500000 sec

===== CHANNEL f1 =====
NUC1          1H
P1           8.00 usec
PL1          0.00 dB
SFO1         500.2235015 MHz

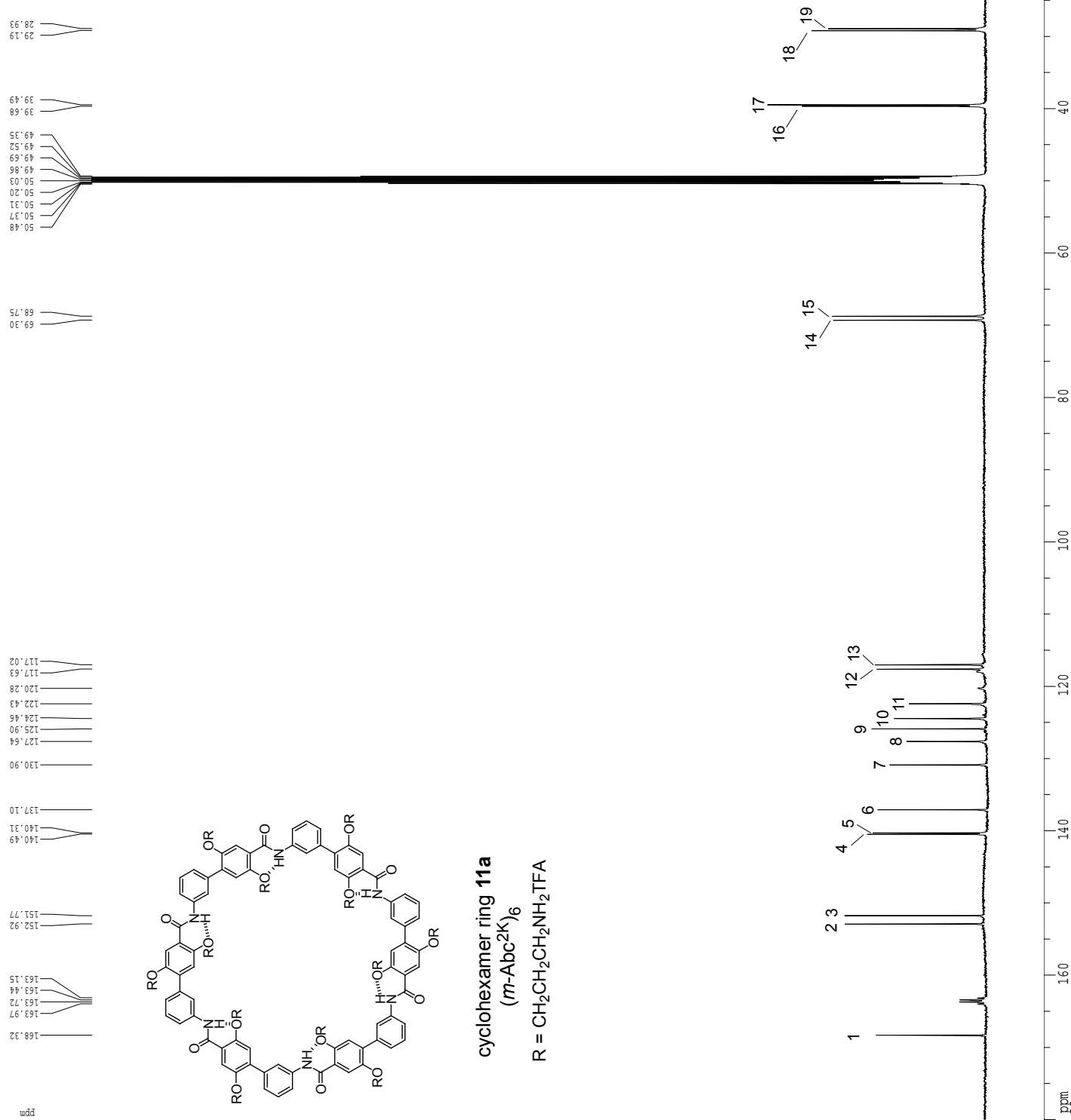
F2 - Processing parameters
SI           65536
SF           500.2200040 MHz
WDW          EM
SSB          0
LB           0.30 Hz
GB           0
PC           4.00

1D NMR plot parameters
CX           22.90 cm
CY           22.90 cm
CZ           4.800 cm
F1F1         2401.06 Hz
F2F2         1.600 ppm
F2          800.35 Hz
PPMCM        0.14035 ppm/cm
HZCM         70.20632 Hz/cm

```



¹³C NMR (125 MHz, 298 K, CD₃OD) spectrum of cyclohexamer ring **11a**



cyclohexamer ring **11a**
(*m*-Abc₂K)₆

R = CH₂CH₂CH₂NH₂TFA

S118

Current Data Parameters
USER cgotha
NAME cg-10-263
EXNO 3
PROCNO 1

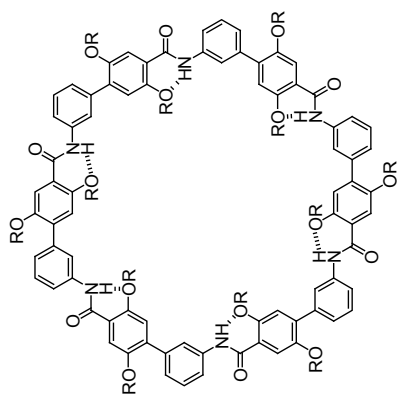
F2 - Acquisition Parameters
Date_ 20091126
Time 22:27
INSTRUM cpcv500
PROBHD 5 mm CPTCI 1H-
PULPROG zgpg30
TD 65418
SOLVENT CD3OD
NS 10000
DS 4
SWH 30303.031 Hz
FIDRES 0.463222 Hz
AQ 1.079470 sec
RG 7298.2
DM 16.500 usec
DE 6.000 usec
TE 298.0 K
DQ 0.25000000 sec
GD 0.03000000 sec
MC1ST 0.00000000 sec
MC1RK 0.01500000 sec

===== CHANNEL f1 =====
NUC1 13C
P1 15.00 usec
PL1 -1.00 dB
SFO1 125.7942548 MHz

===== CHANNEL f2 =====
CPCPRG2 waltz16
NUC2 1H
PCPD2 100.00 usec
PL2 1.60 dB
PL12 23.54 dB
SFO2 500.2225011 MHz

F2 - Processing parameters
SI 6536
SF 125.760131 MHz
WDW EM
SSB 0
LB 2.00 Hz
GB 0
PC 2.00

1D NMR plot parameters
CX 22.80 cm
CY 71.78 cm
F1P 180.000 ppm
F1 22640.42 Hz
F2P 0.000 ppm
F2 0.000 Hz
P1PCW 7.89474 ppm/cm
HZCN 993.00110 Hz/cm

^{13}C NMR (500 MHz, 298 K, CD_3OD) spectrum of cyclohexamer ring **11a** (aromatic and carbonyl region)cyclohexamer ring **11a**
(*m*- Abc^2K)₆R = $\text{CH}_2\text{CH}_2\text{CH}_2\text{NH}_2\text{TFA}$

Current Data Parameters
 USR esocia
 NAME c9-10-263
 PAFNO 3
 PROCNO 1

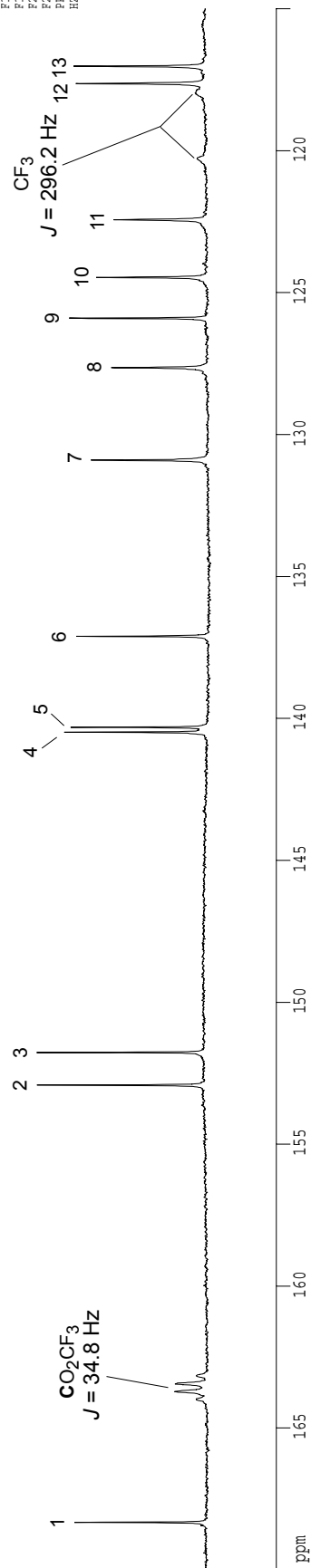
F2 - Acquisition Parameters
 Date_ 20061126
 Time_ 2.21
 INSTRUM cryo500
 PROBHD 5 mm CPTCI 1H-
 PULPROG zgpg30
 TD 65418
 SOLVENT CD3OD
 NS 10000
 DS 4
 SWH 30303.031 Hz
 FIDRES 0.463222 Hz
 AQ 1.0794470 sec
 RG 7298.2
 DW 16.500 usec
 DE 6.00 usec
 TE 298.2 K
 D1 0.2500000 sec
 d11 0.0300000 sec
 NUC1 13C
 NUC2
 P1 15.00 usec
 PL1 -1.00 dB
 SF01 125.7942548 MHz

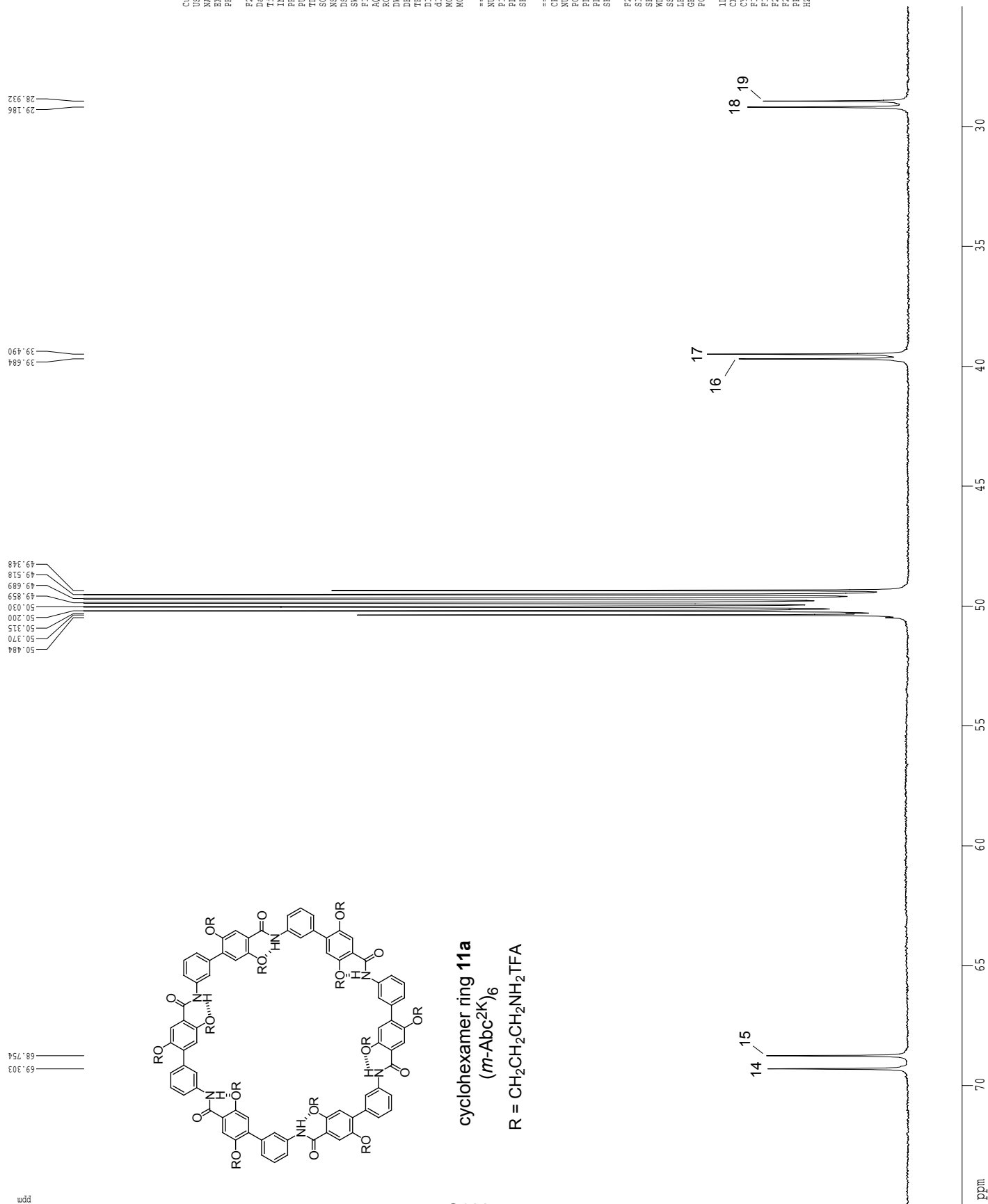
===== CHANNEL F1 =====
 NUC1 13C
 P1 15.00 usec
 PL1 -1.00 dB
 SF01 125.7942548 MHz

===== CHANNEL E2 =====
 CPDPRG2 waltz16
 NUC2 1H
 PCPDZ 100.00 usec
 PL2 1.60 dB
 PL12 21.54 dB
 SF02 500.2225011 MHz

F2 - Processing parameters
 SI 65636
 SF 125.7803381 MHz
 MDW EM
 SSB 0
 LB 2.00 Hz
 GB 0
 PC 2.00

1D NMR plot parameters
 CX 22.80 cm
 CY 71.78 cm
 FIP 170.000 ppm
 FL 21382.62 Hz
 F2P 115.000 ppm
 F4P 14643.72 Hz
 SFO1 125.7803381 MHz
 SFO2 500.2225011 MHz



¹³C NMR (125 MHz, 298 K, CD₃OD) spectrum of cyclohexamer ring **11a** (aliphatic region)

```

Current Data Parameters
USER          cgotha
NAME          CG-10-263
EXPNO        3
PROCNO       1

F2 - Acquisition Parameters
Date_        20061126
Time         2.21
INSTRUM      cryo500
PROBHD       5 mm CPY131
PULPROG      zgpg30
TD           65418
SOLVENT      CD3OD
NS           10000
DS           4
SWH          30303.031 Hz
FIDRES       0.463222 Hz
AQ           1.0794470 sec
RG           7288.2
DW           16.500 usec
DE           6.00 usec
TE           289.0 K
D1           0.25000000 sec
d11          0.03000000 sec
MCRETST      0.00000000 sec
MCHPRK       0.01500000 sec

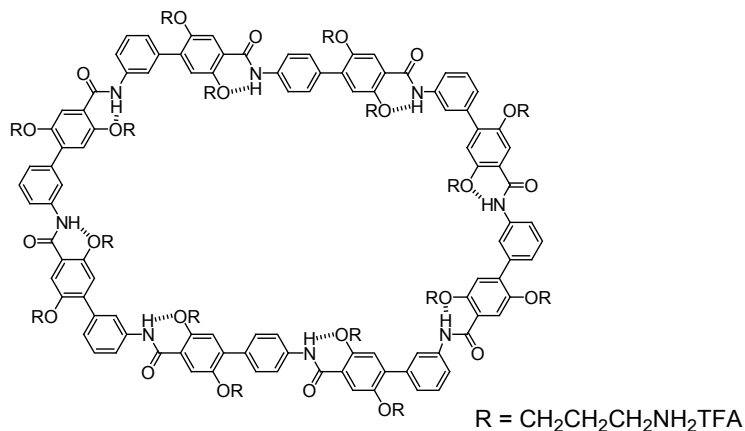
===== CHANNEL f1 =====
NUC1         13C
P1           15.00 usec
PL1          -1.00 dB
SFO1         125.7945548 MHz

===== CHANNEL f2 =====
CPDPRG2      waltz16
NUC2         1H
PCPD2        100.00 usec
PL2          1.60 dB
PL12         23.54 dB
SFO2         500.2225011 MHz

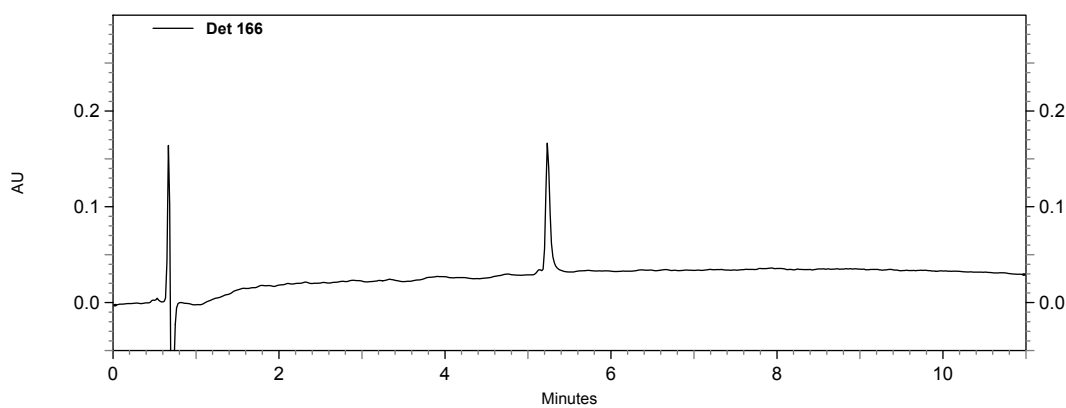
F2 - Processing parameters
SI           65516
SF           125.7901381 MHz
RG          655.16
WDW          EM
SSB          0
GB           0
PC           2.00

ID, NMR plot parameters
CX           22.80 cm
CY           71.78 cm
F1           75.000 ppm
F2           25.000 ppm
F3           3144.50 Hz
PRGCM        2.19298 ppm/cm
HZCM         275.83365 Hz/cm
  
```

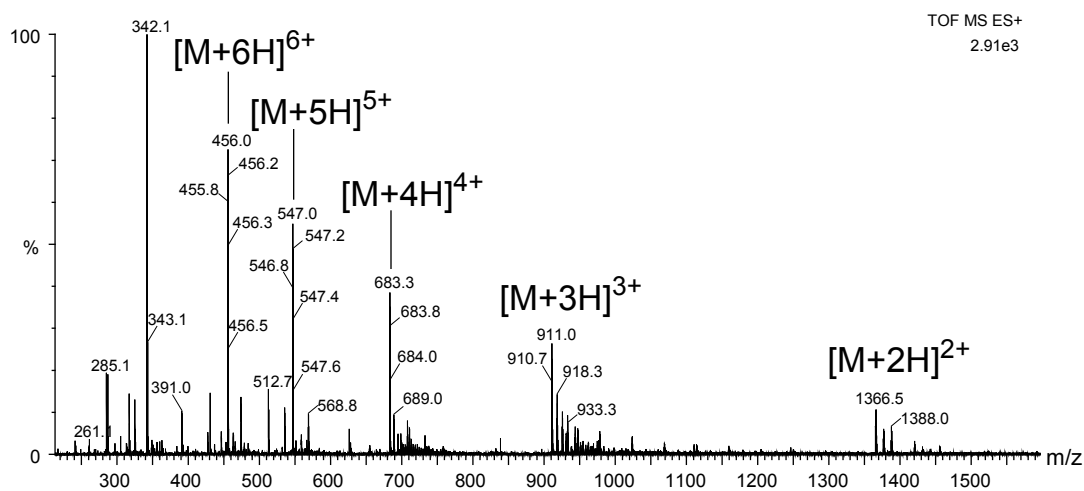
cyclooctamer ring: (*m*-Abc^{2K}-*m*-Abc^{2K}-*m*-Abc^{2K}-*p*-Abc^{2K})₂ (**11b**)
 Analytical RP-HPLC chromatograph and mass spectrum (ESI-MS)

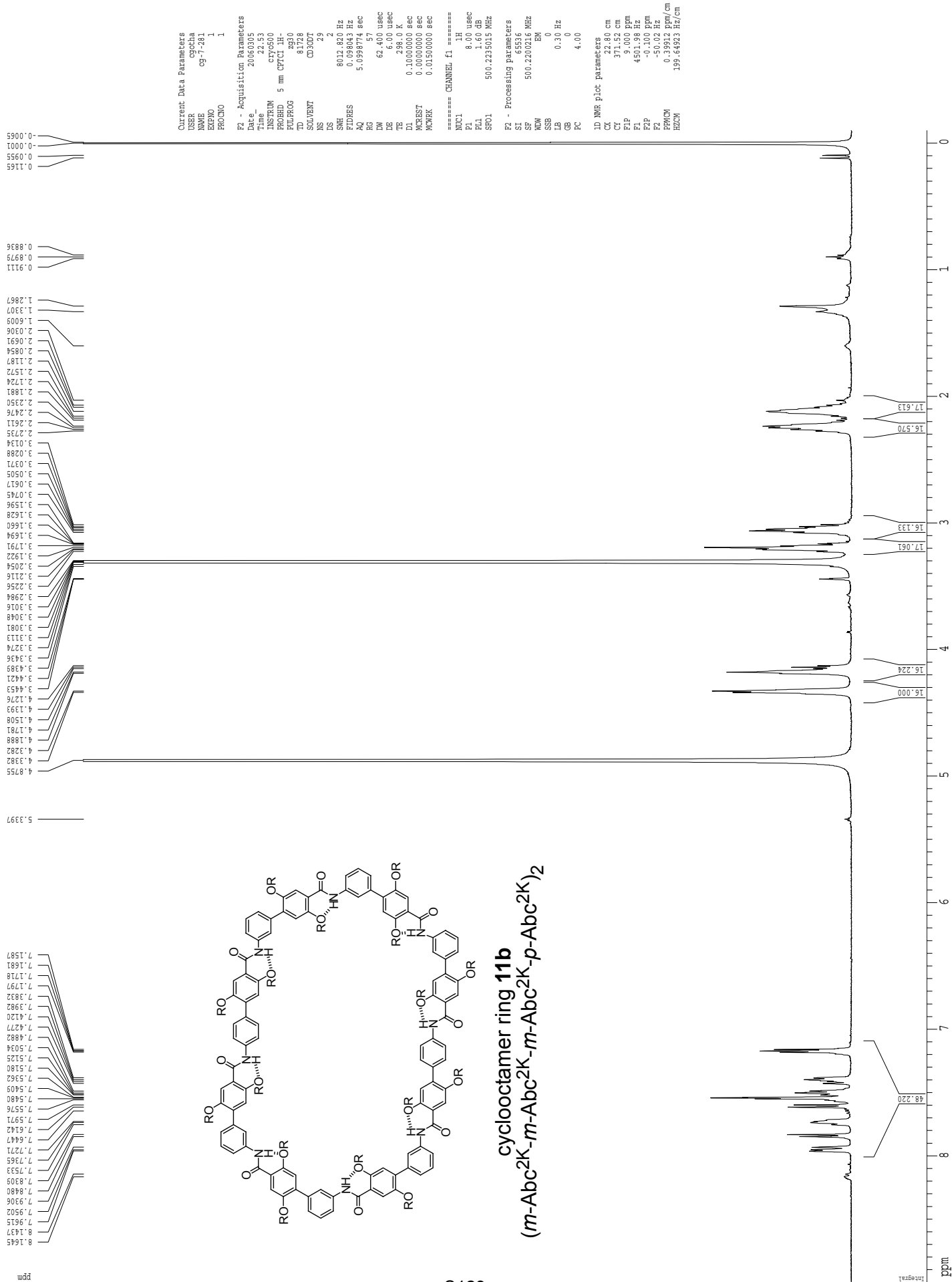


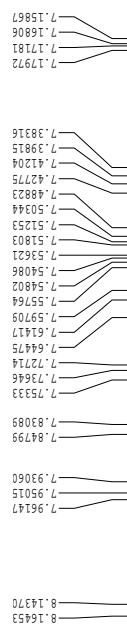
(a) Analytical RP-HPLC (5-50% acetonitrile with 0.1% TFA over 10 min, $\lambda = 214$)



(b) ESI Mass spectrum. (Calcd exact mass for C₁₅₂H₁₈₄N₂₄O₂₄ [M] = 2729.39)



¹H NMR (500 MHz, 298 K, CD₃OD) spectrum of cyclooctamer ring **11b**

¹H NMR (500 MHz, 298 K, CD₃OD) spectrum of cyclooctamer ring 11b

```

Current Data Parameters
Date_      20060305
Time_     22.53
NAME_     cp-7-281
EXPNO_    1
PROCNO_   1

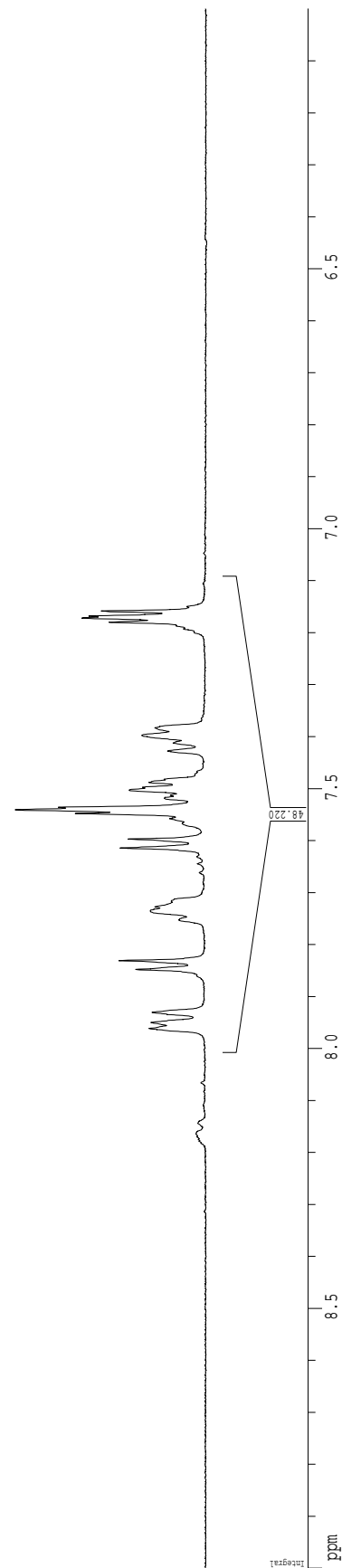
F2 - Acquisition Parameters
Date_     20060305
Time_    22.53
INSTRUM_ crys500
PROBHD_  5 mm CPYCI 1H-
PULPROG_ zg30
TD_      81728
SOLVENT_ CD3OD
AQ_      25
RG_      25
SRH_     8012.820 Hz
FIDRES_  0.698043 Hz
AQ_      5.0398774 sec
RG_      57
DM_      62.400 usec
DE_      6.00 usec
TE_      298.0 K
D1_      0.10000000 sec
MCRETST 0.00000000 sec
MORPK_   0.01500000 sec

===== CHANNEL f1 =====
NUC1_     1H
P1_       8.00 usec
PL1_     -1.60 dB
SFO1_    500.2235015 MHz

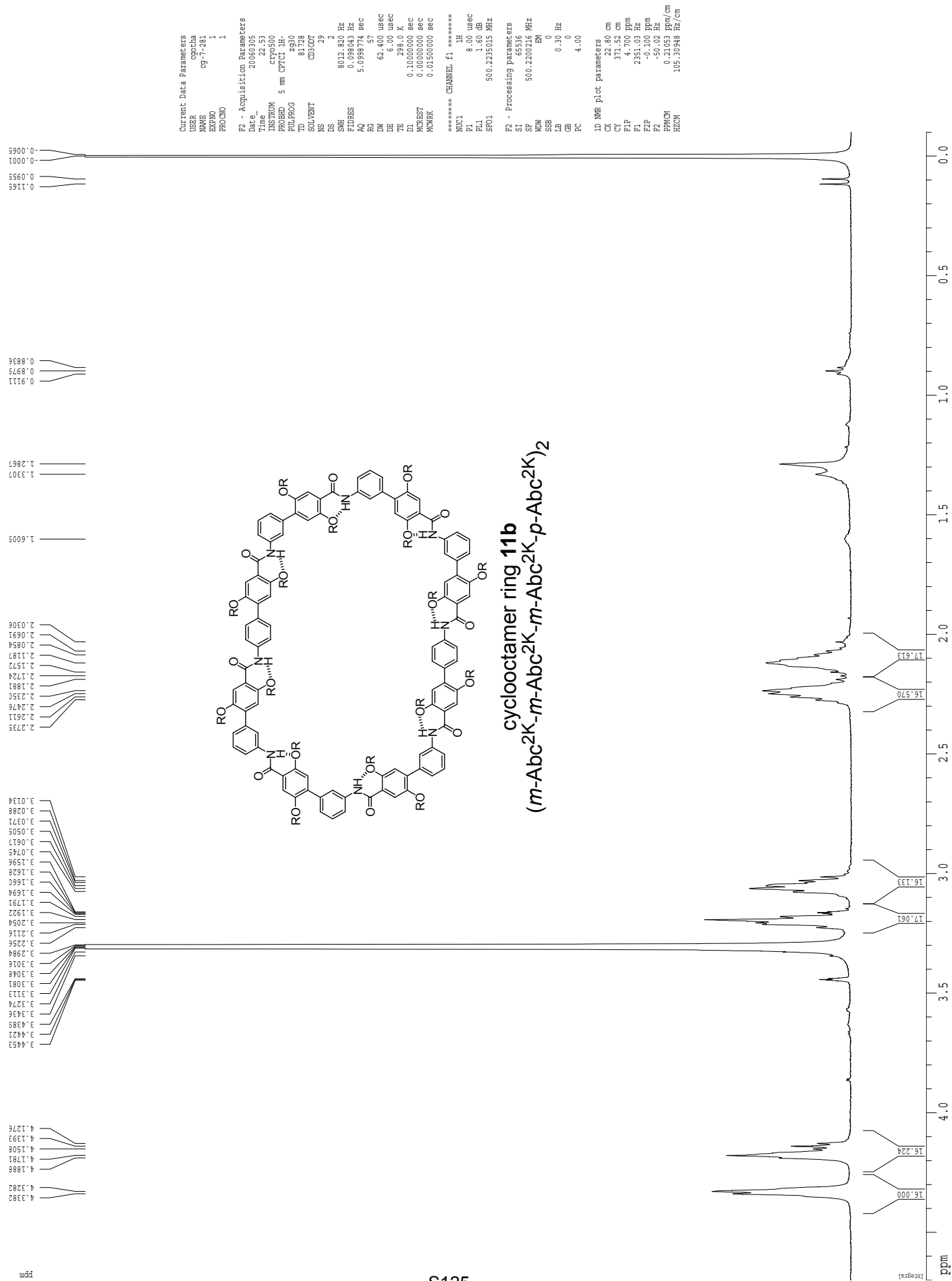
F2 - Processing parameters
SI_       65536
SF_       500.220215 MHz
EM_
SSB_      0
LB_       0.30 Hz
GB_       0
PC_       4.00

ID_NMR plot parameters
CX_       372.80 cm
CY_       371.52 cm
F1P_      9.000 ppm
F1_       4501.98 Hz
F2P_      6.000 ppm
F2_       3001.32 Hz
PPMCM_   0.13158 ppm/cm
HZCM_    65.81843 Hz/cm

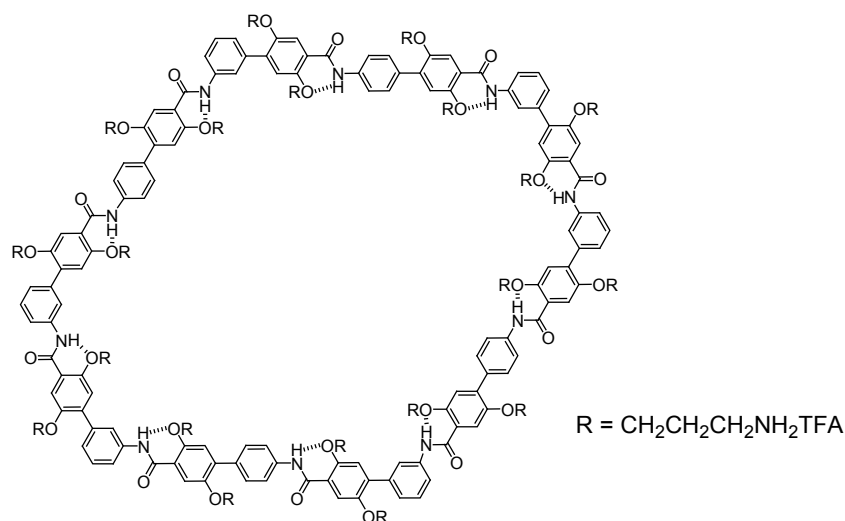
```



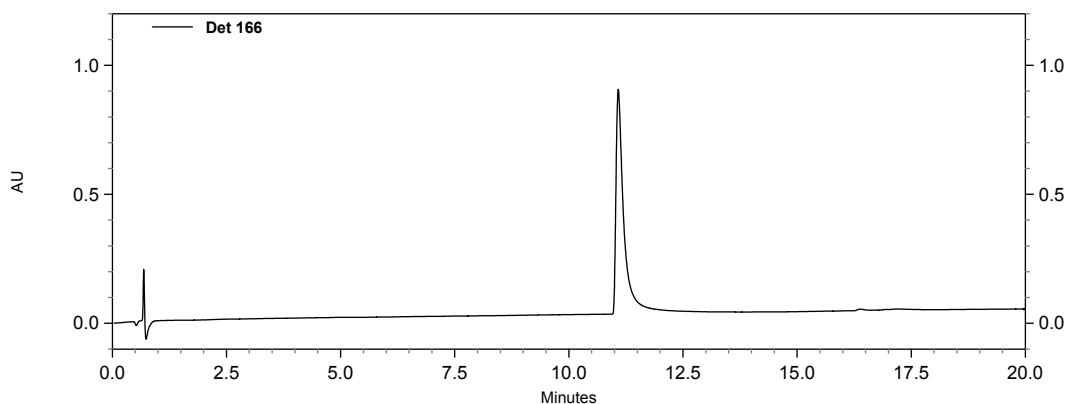
cyclooctamer ring 11b
(m-Abc²K-m-Abc²K-m-Abc²K-p-Abc²K)₂

^1H NMR (500 MHz, 298 K, CD_3OD) spectrum of cyclooctamer ring **11b**

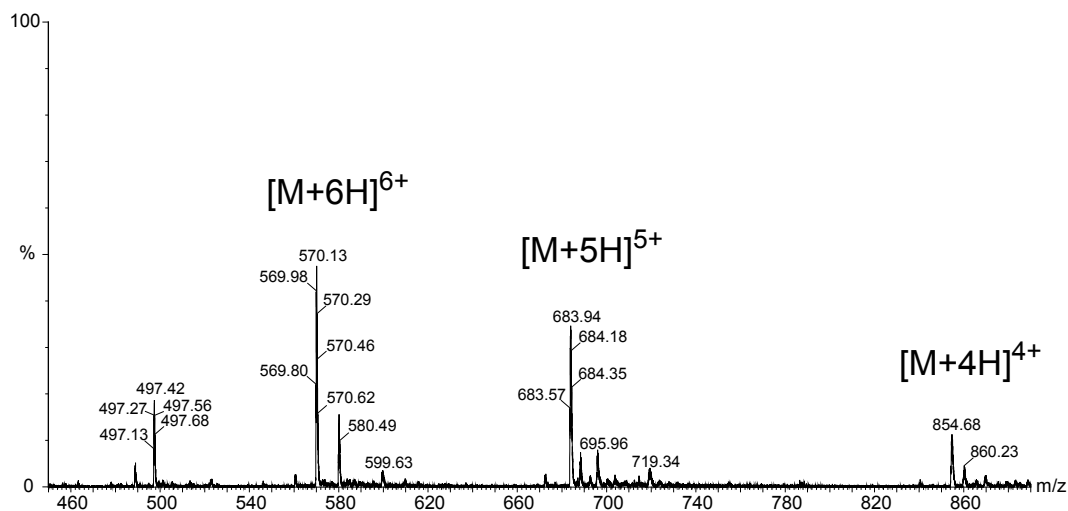
cyclodecamer ring
 $(m\text{-Abc}^{2K}\text{-}m\text{-Abc}^{2K}\text{-}p\text{-Abc}^{2K}\text{-}m\text{-Abc}^{2K}\text{-}p\text{-Abc}^{2K})_2$ (**11c**)
 Analytical RP-HPLC chromatograph and mass spectrum (ESI-MS)

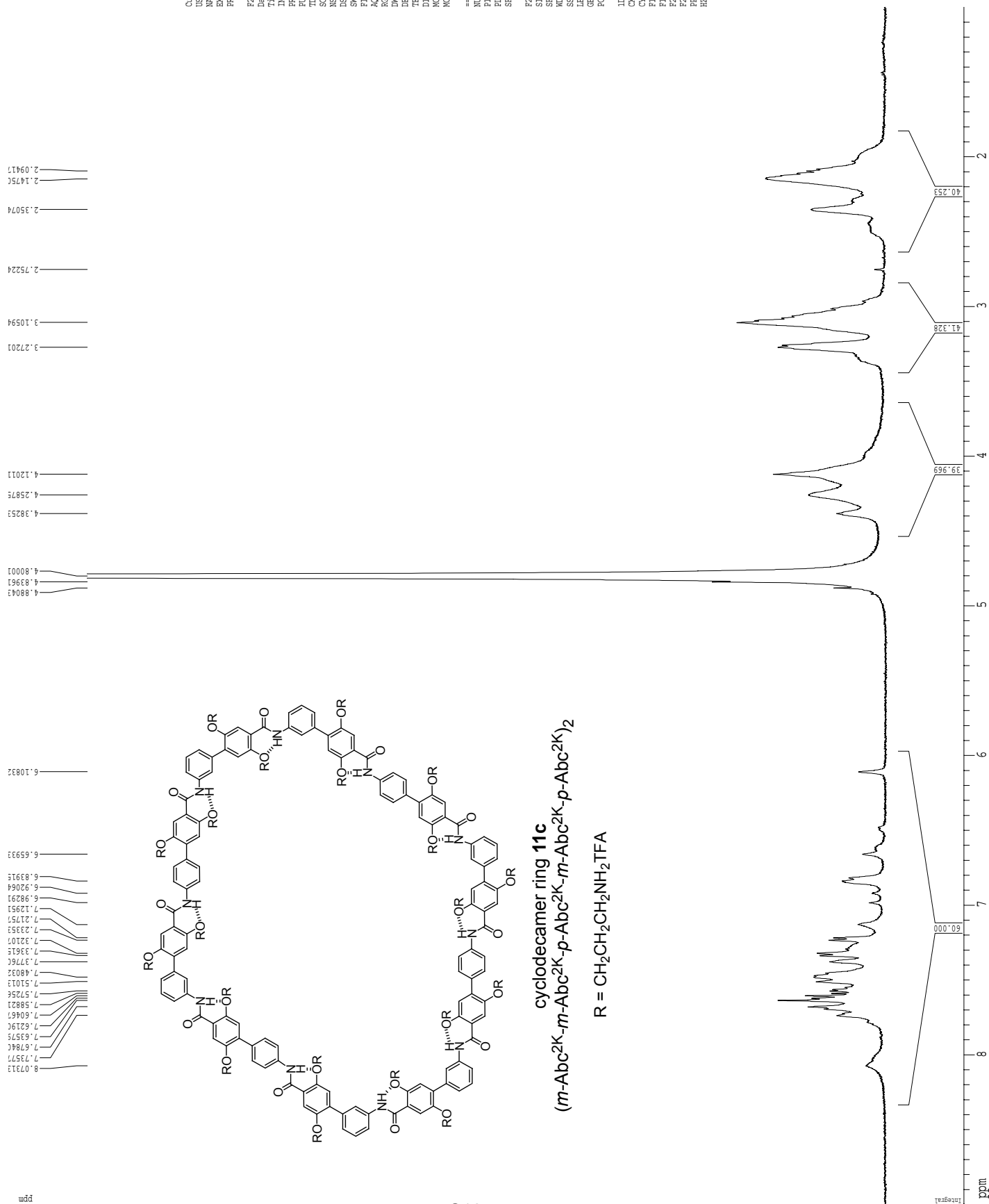


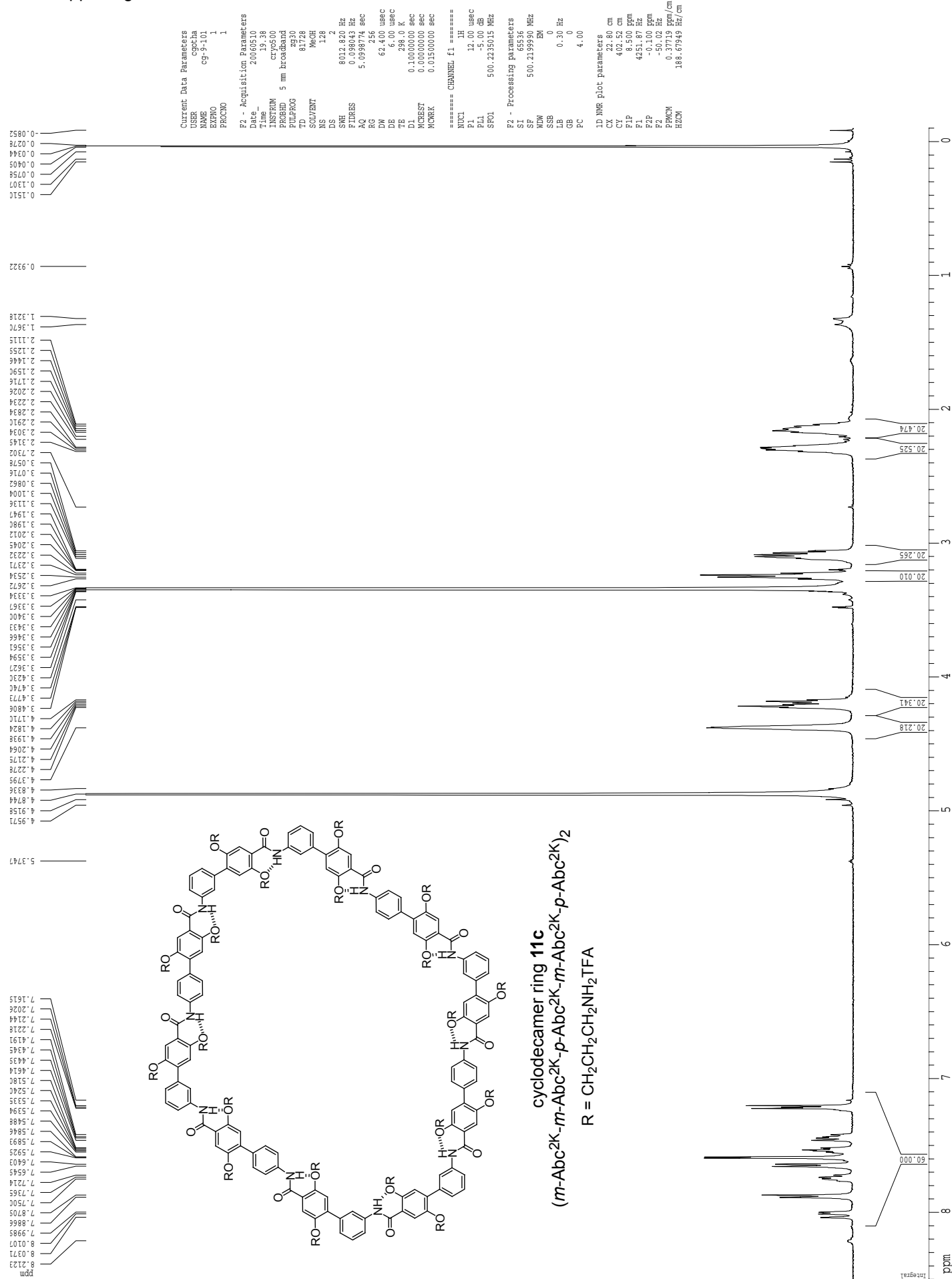
(a) Analytical RP-HPLC (5-50% acetonitrile with 0.1% TFA over 20 min, $\lambda = 214$)

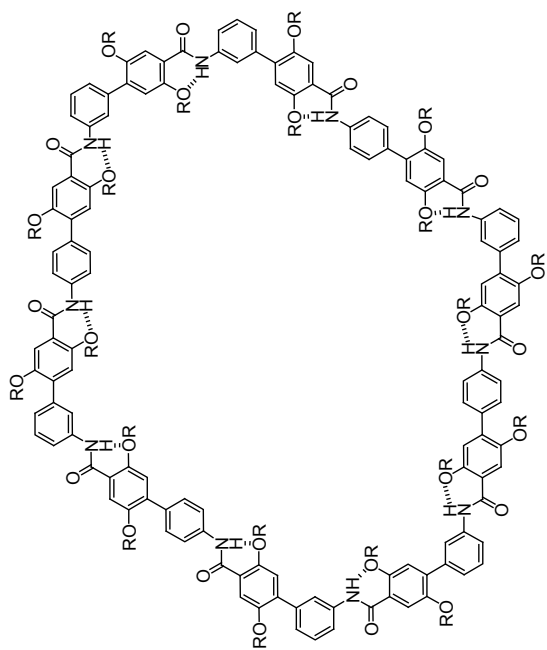
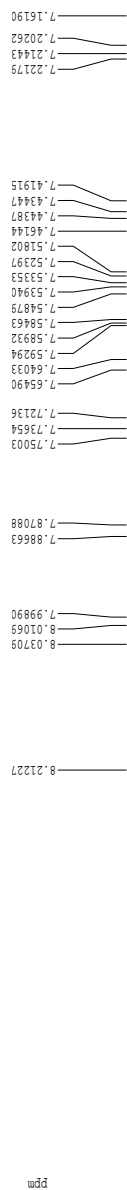


(b) ESI Mass spectrum. (Calcd exact mass for C₁₉₀H₂₃₀N₃₀O₃₀ [M] = 3411.74)



^1H NMR (500 MHz, 298 K, D_2O) spectrum of cyclodecamer ring **11c**

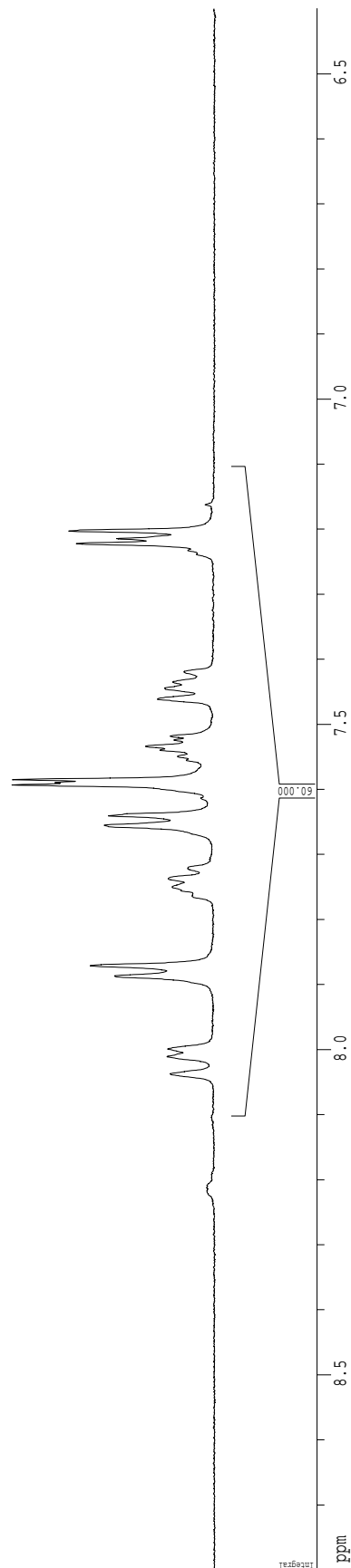
¹H NMR (500 MHz, 298 K, CD₃OD) spectrum of cyclodecamer ring 11c

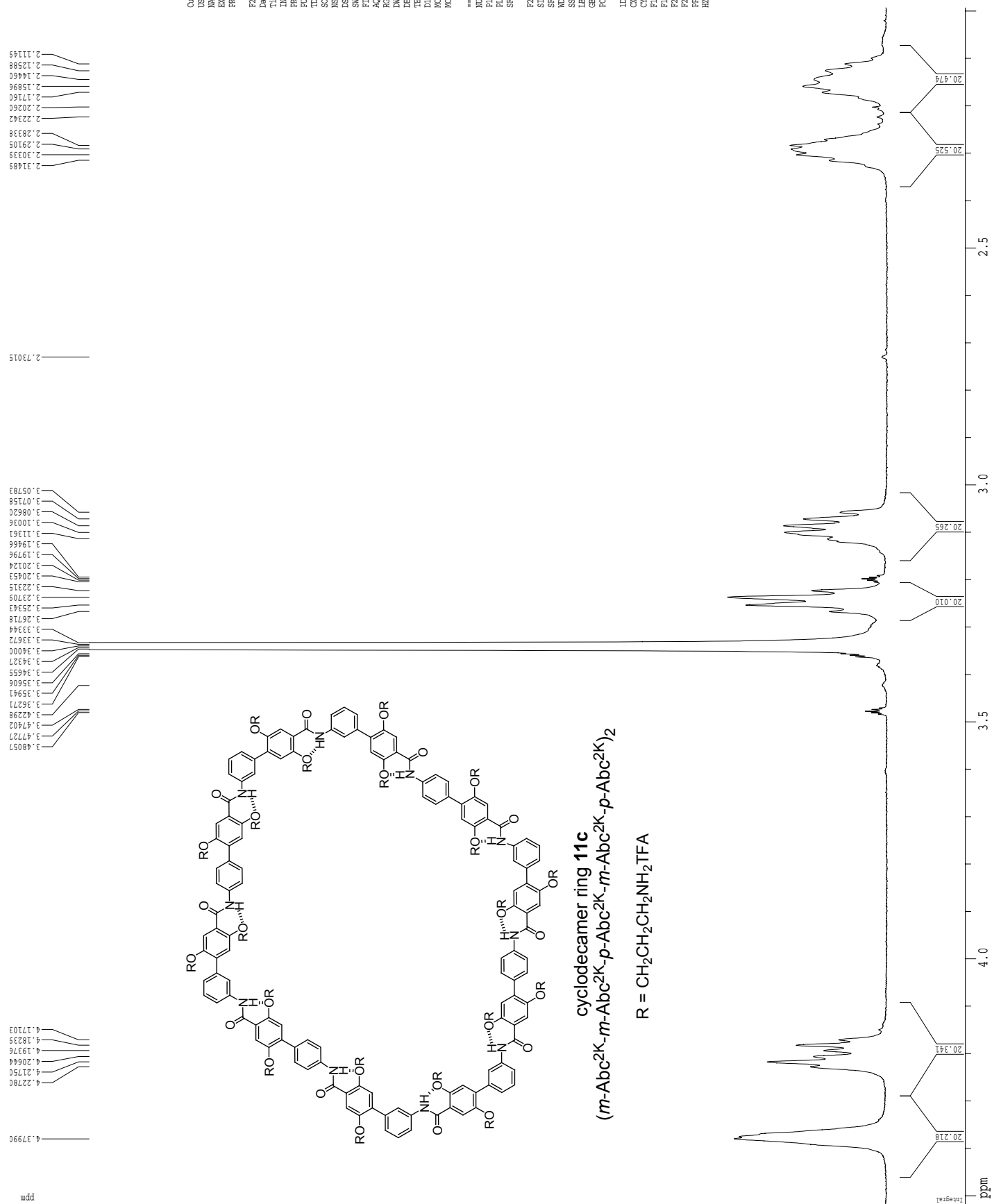
¹H NMR (500 MHz, 298 K, CD₃OD) spectrum of cyclodecamer ring **11c** (aromatic region)

cyclodecamer ring **11c**
 (*m*-Abc²K-*m*-Abc²K-*p*-Abc²K-*m*-Abc²K-*m*-Abc²K-*p*-Abc²K)₂
 R = CH₂CH₂CH₂NH₂TFA

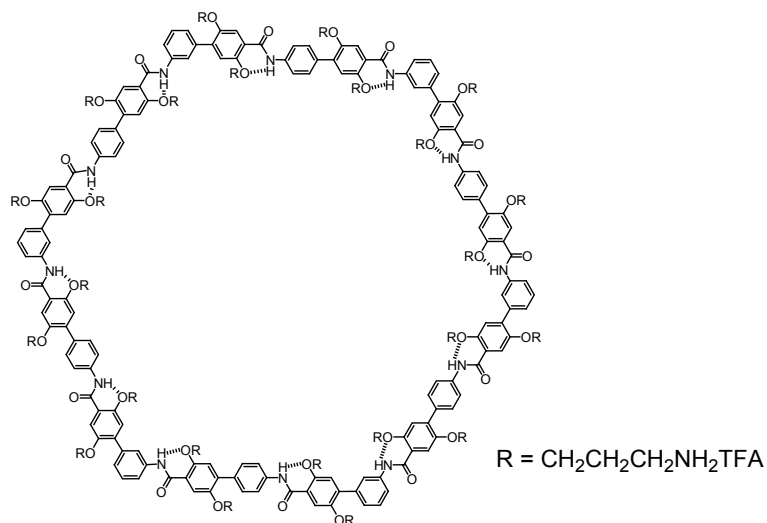
```

Current Data Parameters
USER          egecha
NAME          c9-9-101
PROCNO       1
PRCNO        1
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F2 - Acquisition Parameters
Date_         20060510
Time_        19.38
INSTRUM      crys500
PROBHD       5 mm broadband
PULPROG      zg30
TD           81728
SOLVENT      MeOH
NS           128
DS           2
AQ           0.0312870 Hz
RG           0.6988043 Hz
FIDRES       5.0398774 sec
AQ           255
DM           62.400 usec
DE           6.00 usec
TE           298.0 K
D1           0.10000000 sec
D11          0.00000000 sec
D12          0.00000000 sec
D13          0.01500000 sec
=====
===== CHANNEL f1 =====
NUC1          1H
P1           12.00 usec
PL1          -1.50 dB
SFO1         500.2235013 MHz
=====
F2 - Processing parameters
S1           65536
SF           500.2199990 MHz
WDW          EM
SSB          0
LB           0.30 Hz
GB           0
PC           4.00
=====
1D NMR plot parameters
CX           422.50 cm
CY           422.50 cm
CZ           422.50 cm
F1           8.800 Hz
F2           4401.94 Hz
F3           6.400 ppm
F4           3201.41 Hz
PRCM         0.10526 ppm/cm
HZCM         52.65474 Hz/cm
  
```

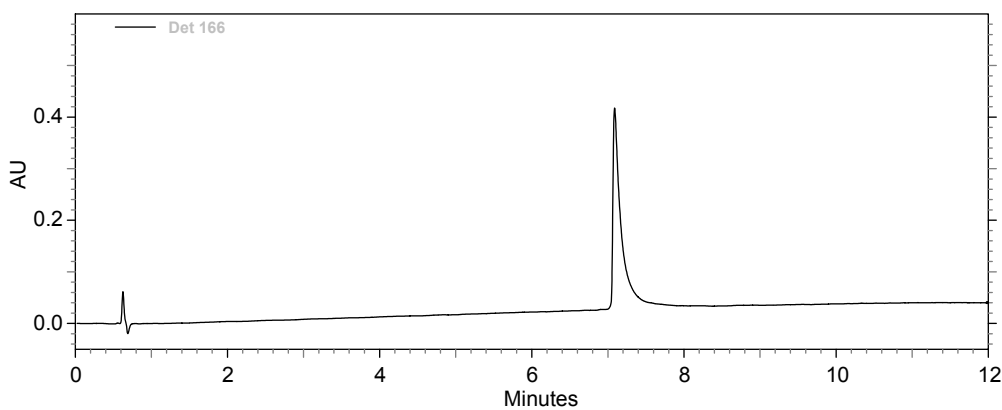


^1H NMR (500 MHz, 298 K, CD_3OD) spectrum of cyclodecamer ring **11c** (aliphatic region)

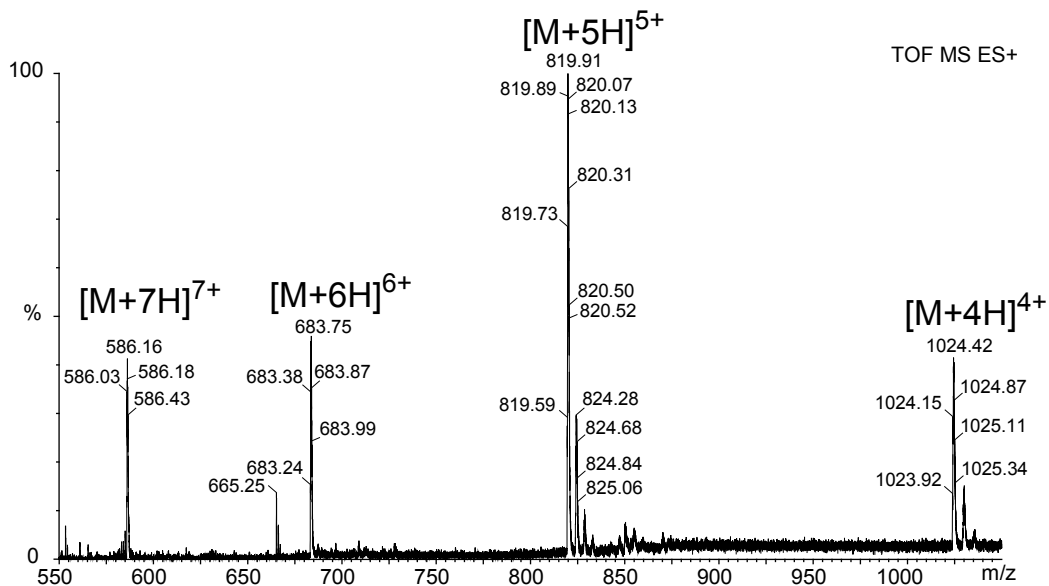
cyclododecamer ring: (*m*-Abc^{2K}-*p*-Abc^{2K})₆ (**11d**)
 Analytical RP-HPLC chromatograph and mass spectrum (ESI-MS)



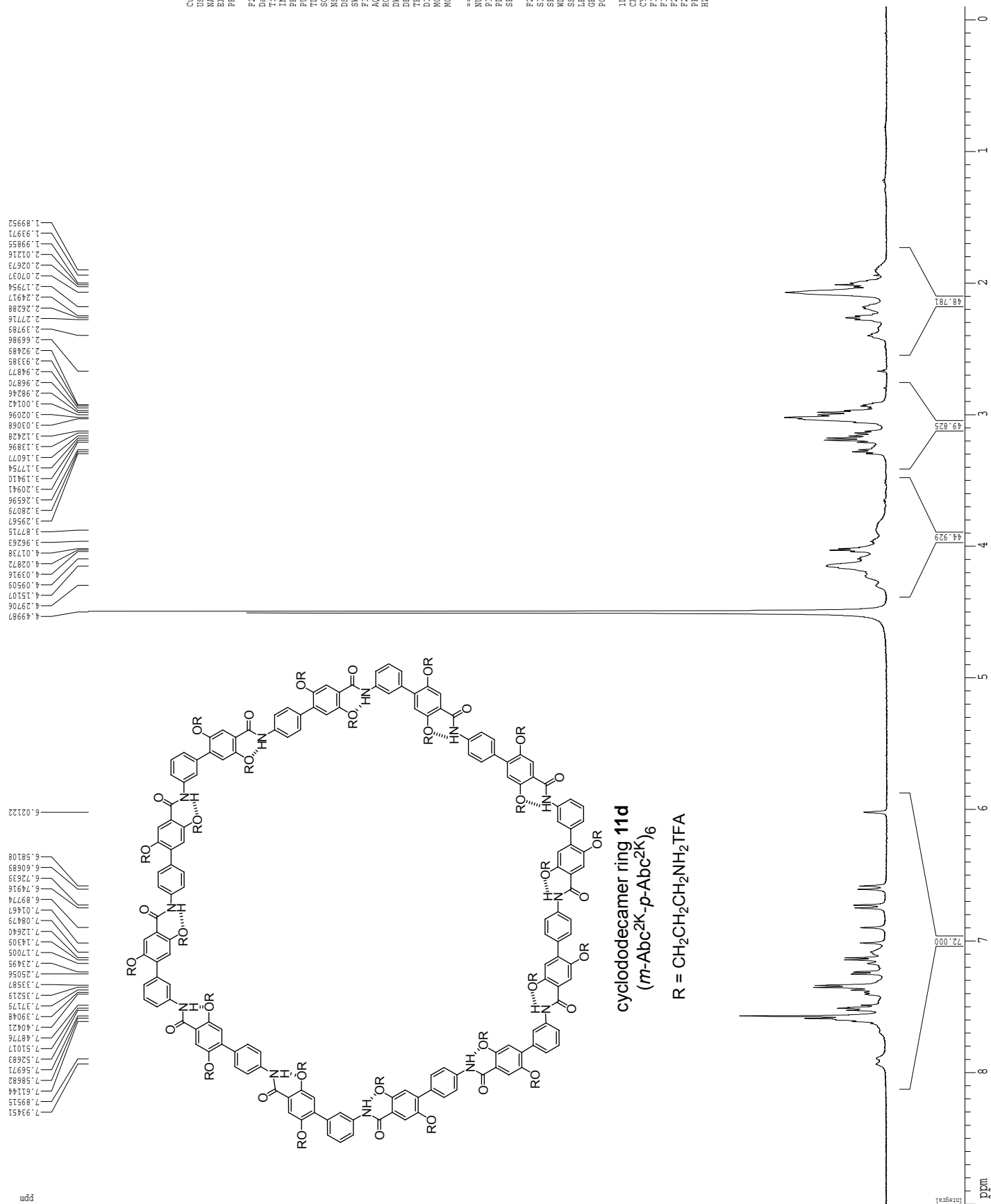
(a) Analytical RP-HPLC (5-50% acetonitrile with 0.1% TFA over 10 min, $\lambda = 214$)



(b) ESI Mass spectrum. (Calcd exact mass for C₂₂₈H₂₇₆N₃₆O₃₆ [M] = 4094.09)



¹H NMR (500 MHz, 323 K, D₂O) spectrum of cyclododecamer ring **11d**



Supporting Information

Current Data Parameters
 USER: C.M.G
 NAME: CP-11-53
 EXPNO: 4
 PROCNO: 1

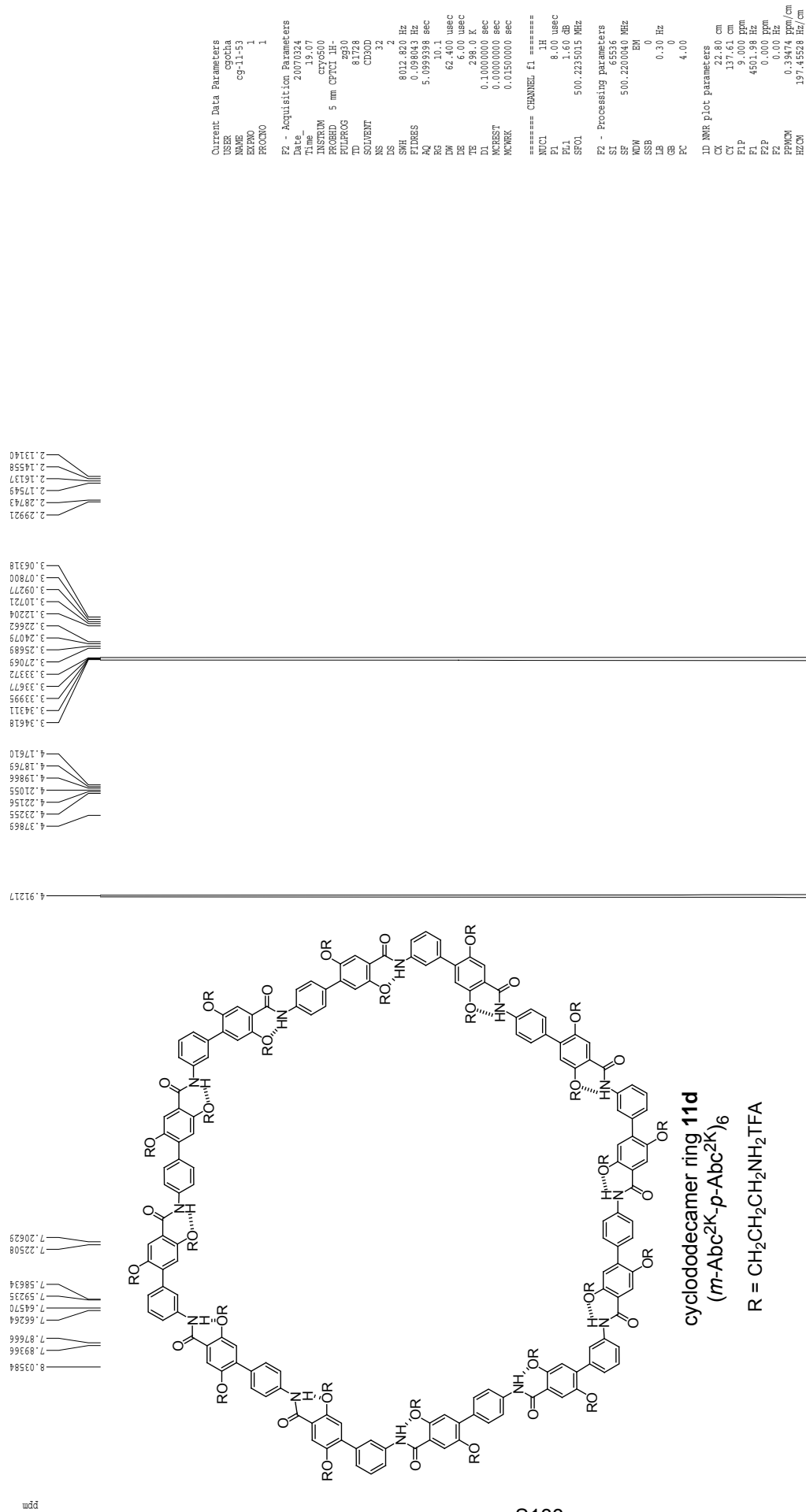
F2 - Acquisition Parameters
 Date_: 20070423
 Time: 22.47
 INSTRUM: cryo500
 PROBHD: 5 mm CPYCI 1H-
 PULPROG: zg30
 TD: 81728
 SOLVENT: D2O
 DS: 2
 SFO1: 801.270 MHz
 SHF: 0.98043 Hz
 FIDRES: 5.0998774 sec
 AQ: 16
 RG: 16
 DW: 62.400 usec
 DE: 6.00 usec
 TE: 323.0 K
 D1: 0.1000000 sec
 MCREST: 0.0000000 sec
 MCPRK: 0.0150000 sec

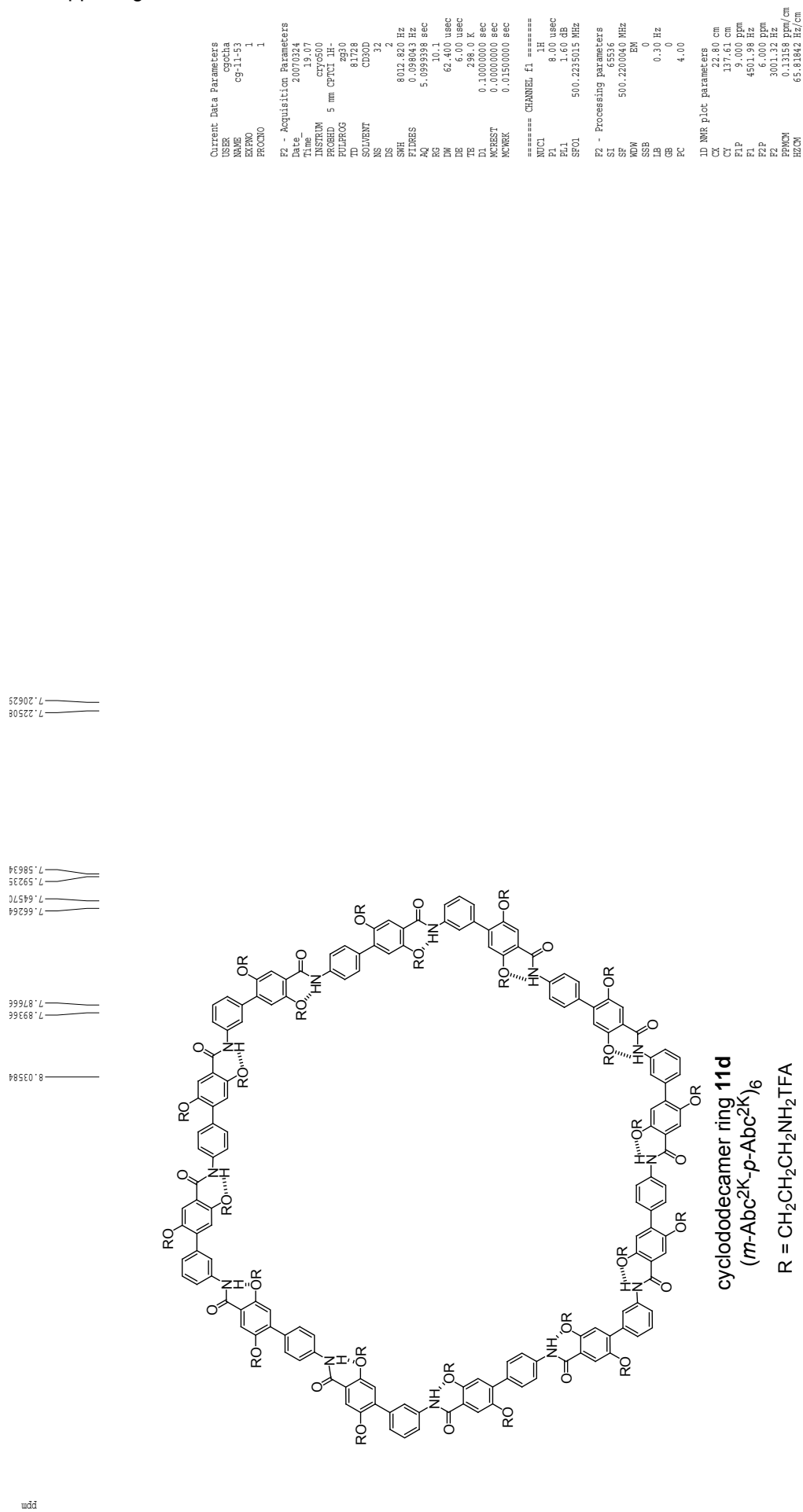
===== CHANNEL f1 =====
 NUC1: 1H
 P1: 8.00 usec
 PL1: 1.60 dB
 SFO1: 500.2635015 MHz

F2 - Processing parameters
 SI: 65536
 SF: 500.2201562 MHz
 MDW: EN
 SSB: 0
 LB: 0.30 Hz
 GB: 0
 PC: 4.00

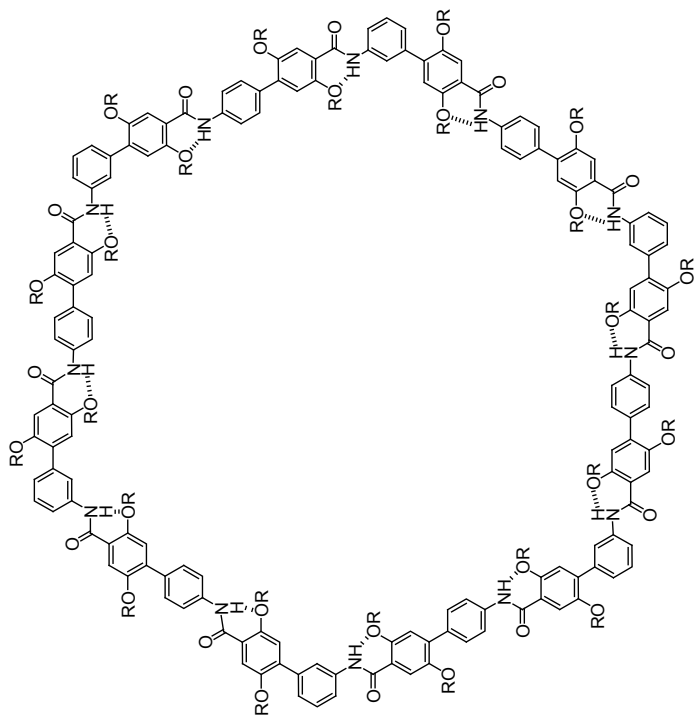
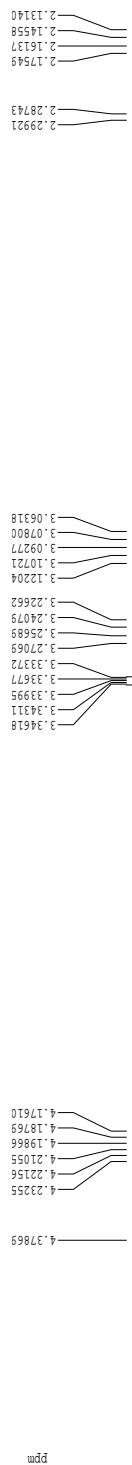
1D NMR plot parameters
 CY: 22.80 sec
 CV: 295.55 sec
 FIP: 9.000 Hz
 F1: 4601.98 Hz
 F2P: -0.100 Hz
 F2: -50.02 Hz
 PRGM: 0.38912 sec
 HZCM: 195.64929 Hz

C. M. G. and J. S. Nowick

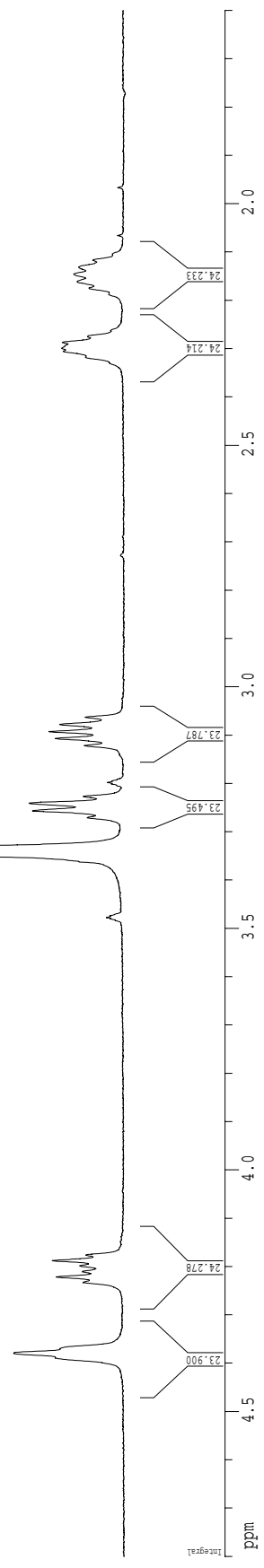
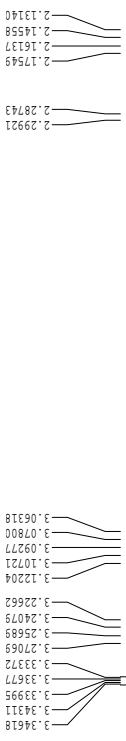
^1H NMR (500 MHz, 298 K, CD_3OD) spectrum of cyclododecamer ring **11d**

¹H NMR (500 MHz, 298 K, CD₃OD) spectrum of cyclododecamer ring **11d** (aromatic region)

¹H NMR (500 MHz, 298 K, CD₃OD) spectrum of cyclododecamer ring **11d** (aliphatic region)



cyclododecamer ring **11d**
(*m*-Abc²K-*p*-Abc²K)₆
R = CH₂CH₂CH₂NH₂TFA



Current Data Parameters
 USSF cysiba
 NAME cysiba
 EXPR0 09-11-13
 PROCNO 1

F2 - Acquisition Parameters
 Date 20070324
 Time 19.07
 INSTRUM cryos00
 PROBHD 5 mm CPXI JH-
 PULPROG zgpg30
 TD 61728
 SFO 500.135000
 SOLVENT CD3OD
 NS 2
 DS 2
 SFR 8012.800 Hz
 FIDRES 0.08043 Hz
 AQ 5.0999398 sec
 RG 10.1
 DM 62.400 usec
 DE 6.00 usec
 TE 298.0 K
 D1 0.1000000 sec
 ACQST 0.0000000 sec
 ACQEK 0.0150000 sec

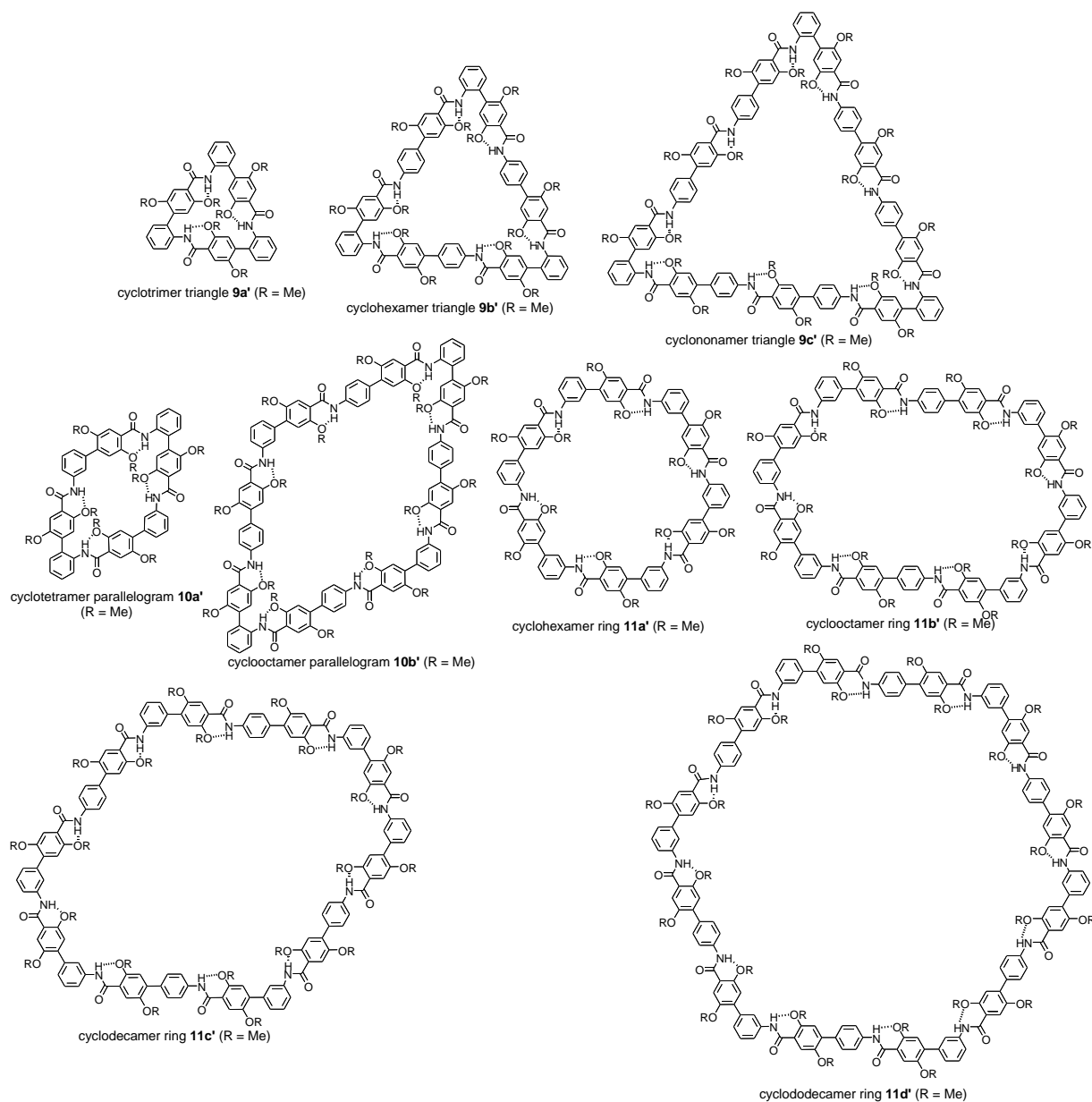
***** CHANNEL f1 *****
 NUCL1 1H
 P1 8.00 usec
 PL1 1.60 dB
 SFO1 500.2235015 MHz

F2 - Processing parameters
 S1 65536
 SF 500.2200040 MHz
 WDW EM
 SSB 0
 LB 0.30 Hz
 GB 0
 PC 4.00

1D NMR plot parameters
 CX 32.80 cm
 CY 137.61 cm
 F1P 4.800 PPT
 F1 2401.06 Hz
 F2P 1.600 PPT
 F2 800.35 Hz
 PPMCH 0.14035 PPT
 HZCN 70.20632 Hz

Molecular Modeling Studies

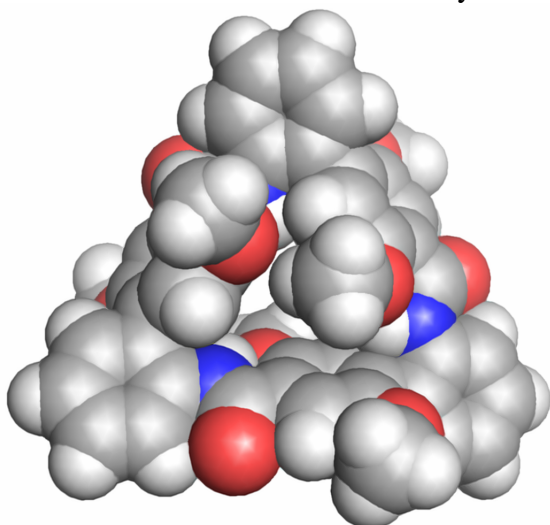
Macrocycles **9a-c**, **10a,b**, and **11a-d** were modeled as simplified homologues in which the propyloxyammonium ($\text{OCH}_2\text{CH}_2\text{CH}_2\text{NH}_3^+$) side chains were replaced with methoxy (OMe) groups (**9a'-c'**, **10a',b'**, and **11a'-d'**). Each molecule was modeled using Maestro/MacroModel v8.5 with the MMFFs implementation of the MMFF force field and MCMM conformational searching. The Ar–Ar and Ar–N bonds were rotated during the search procedure. Rotations about the Ar–CO and Ar–O bonds were not performed. Amide linkages were assumed to adopt *trans* conformations and were not allowed to adopt *cis* conformations (except where noted otherwise). 1000 Monte-Carlo search steps were performed for each structure, and no effort was made to assure that all of the lowest-energy conformers or the global minimum were identified. (Thorough identification of all low-energy conformers is not practical for the larger structures, and is only marginally practical for the smaller structures.) For each molecule, the lowest energy conformer and an overlay of the conformers found within the lowest 5.00 kJ/mol are shown.



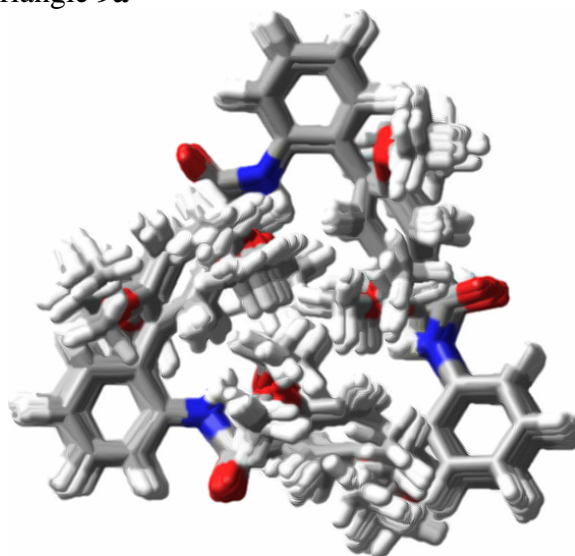
The modeling should be interpreted with several caveats: (1) The MMFF and MMFFs force fields lack good parameters for some of the stretches, bends, and torsions associated with the structures. (2) The MMFFs force field is designed to enforce planarity of the amide nitrogen atoms and may therefore overemphasize the conformational regularity of the structures. (3) The absence of H₂O solvation in the modeling should decrease the effect of hydrophobic interactions in structures. (4) Conformers with *cis*-amide linkages will not be identified; those lacking

intramolecular hydrogen bonds between the *ortho*-methoxy group and the amide NH group, or with alternative rotations about the Ar–OMe bonds may not be identified.

Cyclotrimer Triangle **9a'**

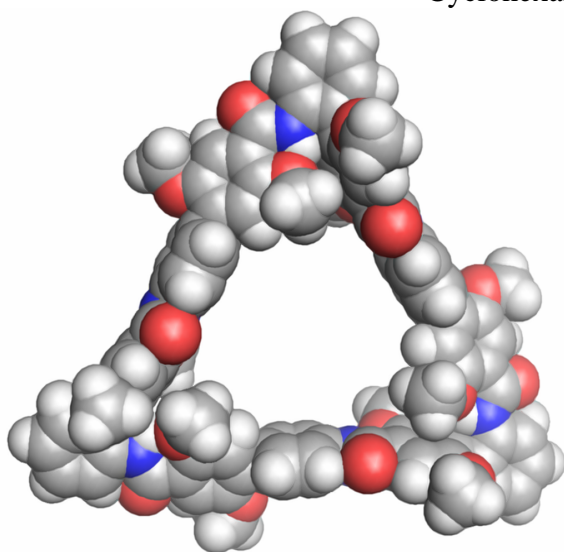


lowest energy conformer found

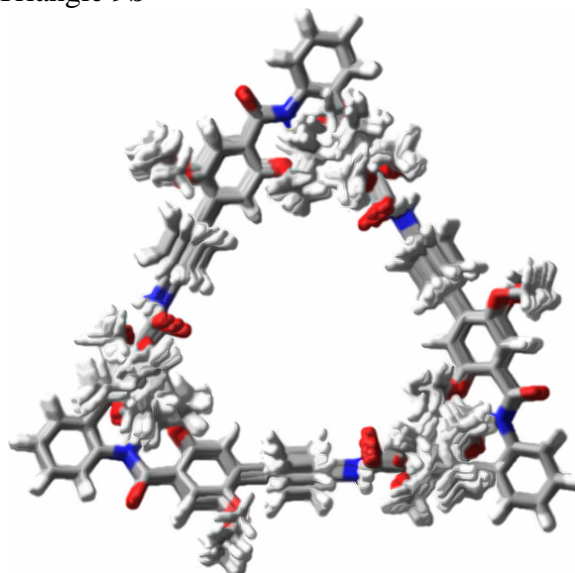


superposition of conformers found within lowest 5.00 kJ/mol (39 conformers).

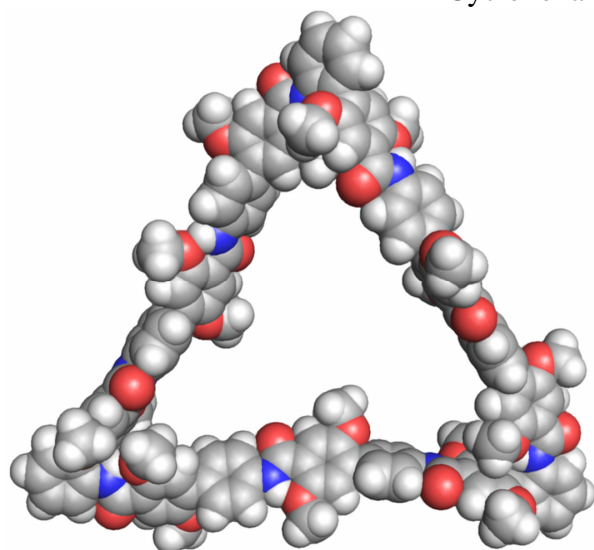
Cyclohexamer Triangle **9b'**



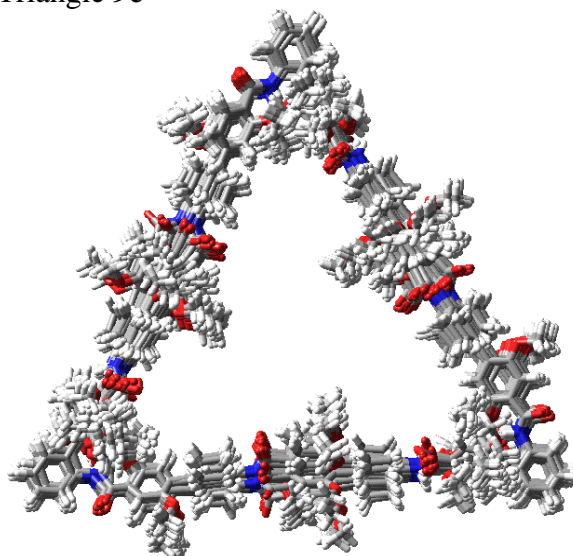
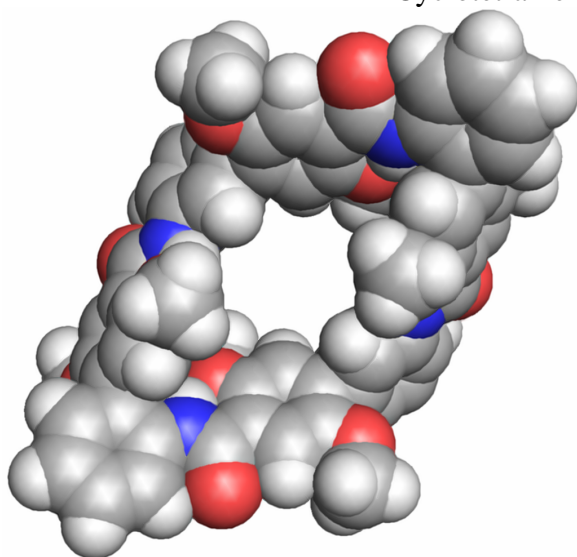
lowest energy conformer found



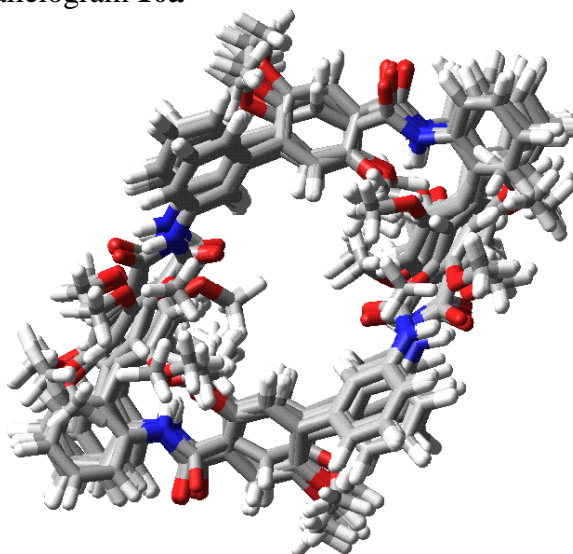
superposition of conformers found within lowest 5.00 kJ/mol (51 conformers).

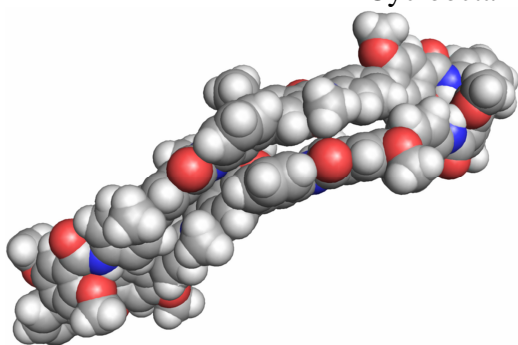
Cyclononamer Triangle **9c'**

lowest energy conformer found

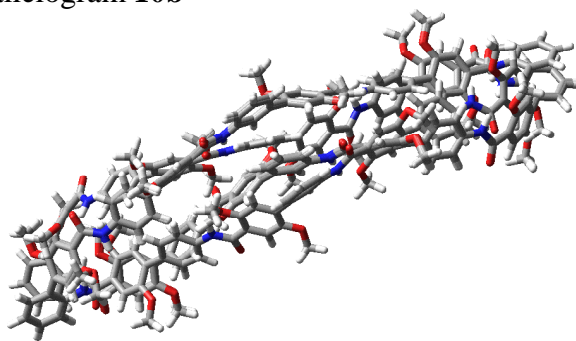
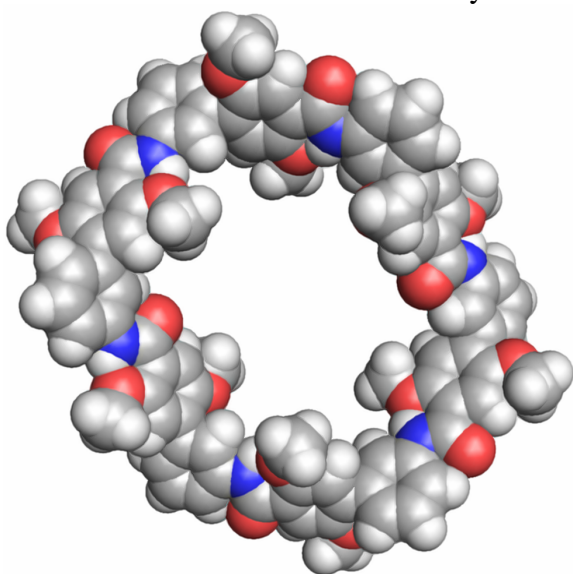
superposition of conformers found
within lowest 5.00 kJ/mol (80 conformers).Cyclotetramer Parallelogram **10a'**

lowest energy conformer found

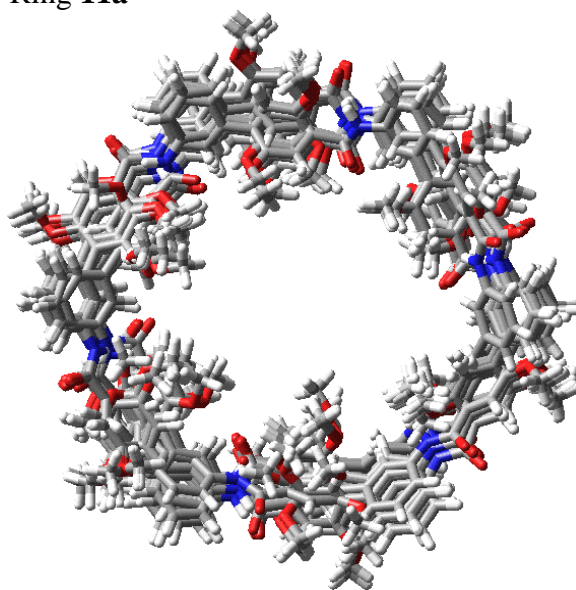
superposition of conformers found
within lowest 5.00 kJ/mol (15 conformers).

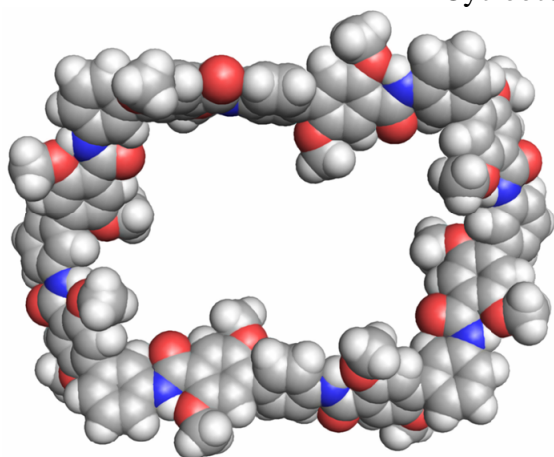
Cyclooctamer Parallelogram **10b'**

lowest energy conformer found

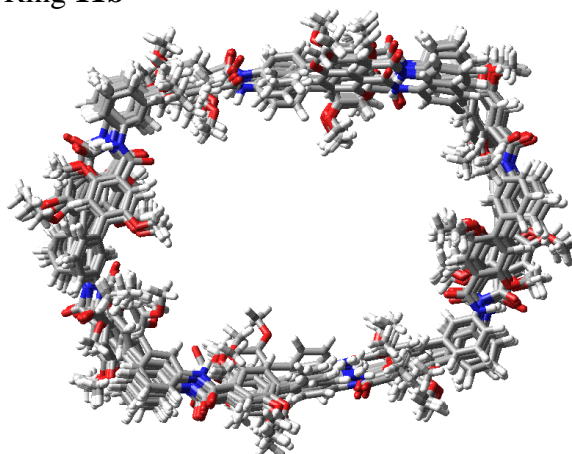
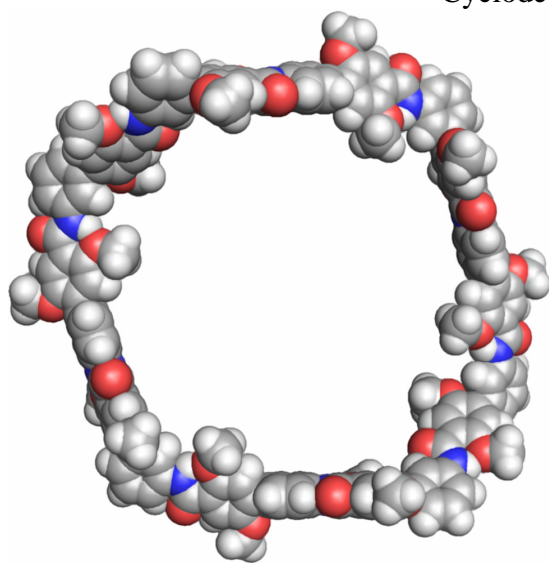
superposition of conformers found
within lowest 5.00 kJ/mol (3 conformers).Cyclohexamer Ring **11a'**

lowest energy conformer found

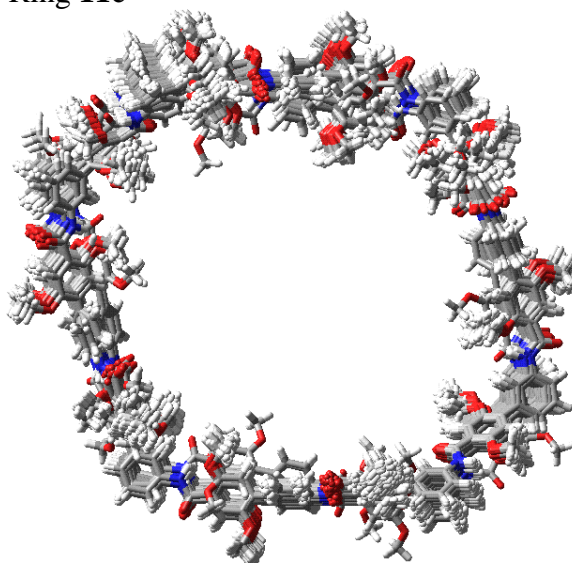
superposition of conformers found
within lowest 5.00 kJ/mol (19 conformers).

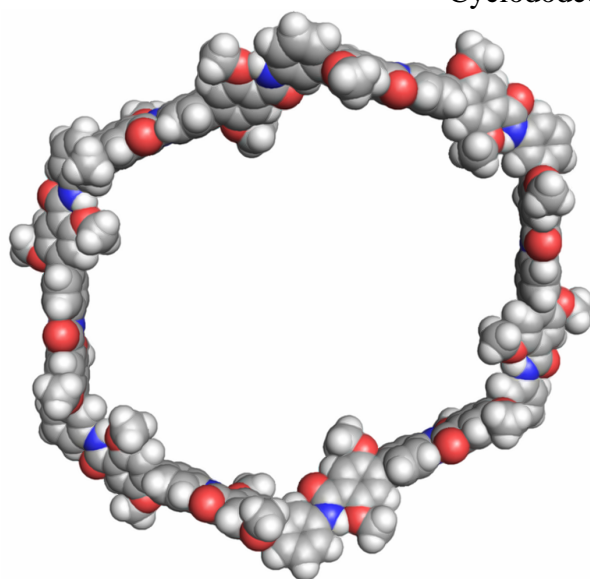
Cyclooctamer Ring **11b'**

lowest energy conformer found

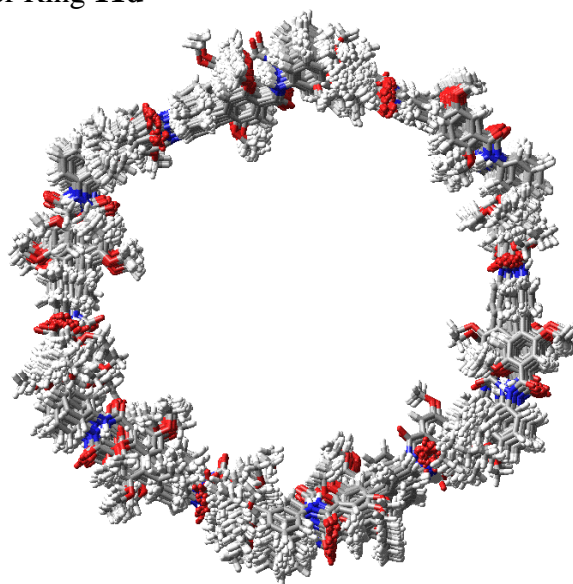
superposition of conformers found
within lowest 5.00 kJ/mol (21 conformers).Cyclodecamer Ring **11c'**

lowest energy conformer found

superposition of conformers found
within lowest 5.00 kJ/mol (173 conformers).

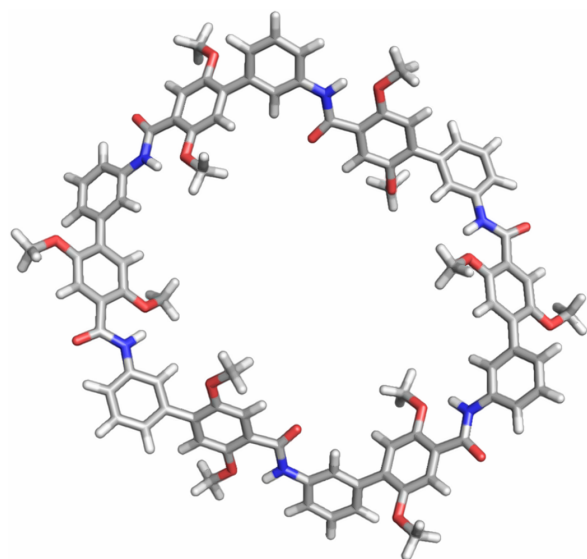
Cyclododecamer Ring **11d'**

lowest energy conformer found

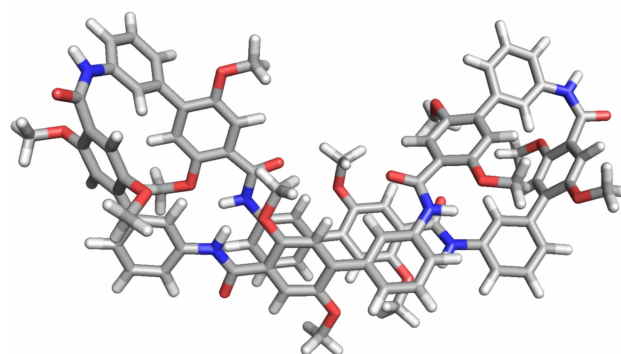
superposition of conformers found
within lowest 5.00 kJ/mol (235 conformers).

Cyclohexamer Ring **11a'** (*cttctt*- and *ttttt*-conformers)

A model of the *cttctt*-conformer of **11a'** was generated using Maestro/MacroModel v8.5 with the MMFFs implementation of the MMFF force field and is shown next to the *ttttt*-conformer.



ttttt-conformer of **11a'**



cttctt-conformer of **11a'**