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

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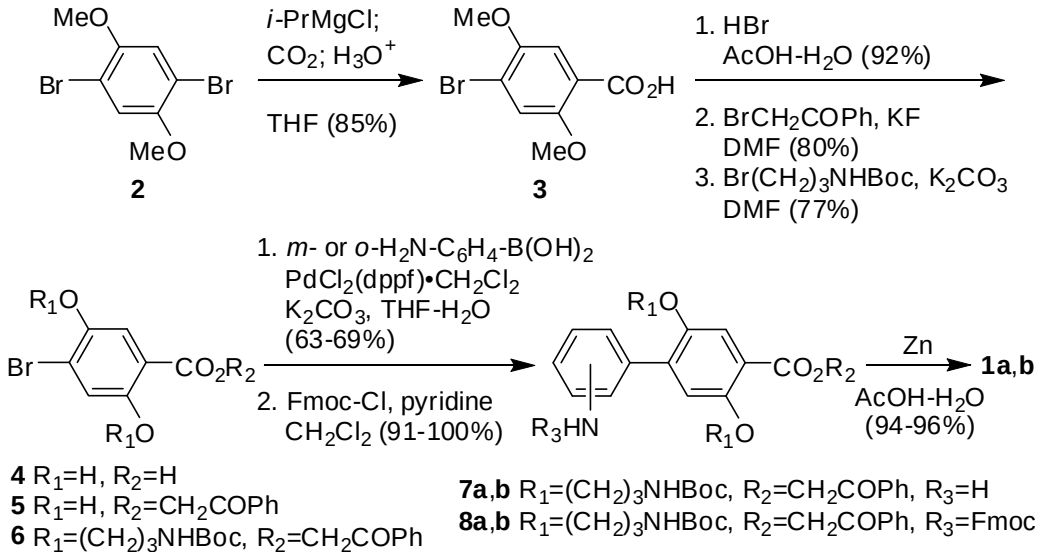
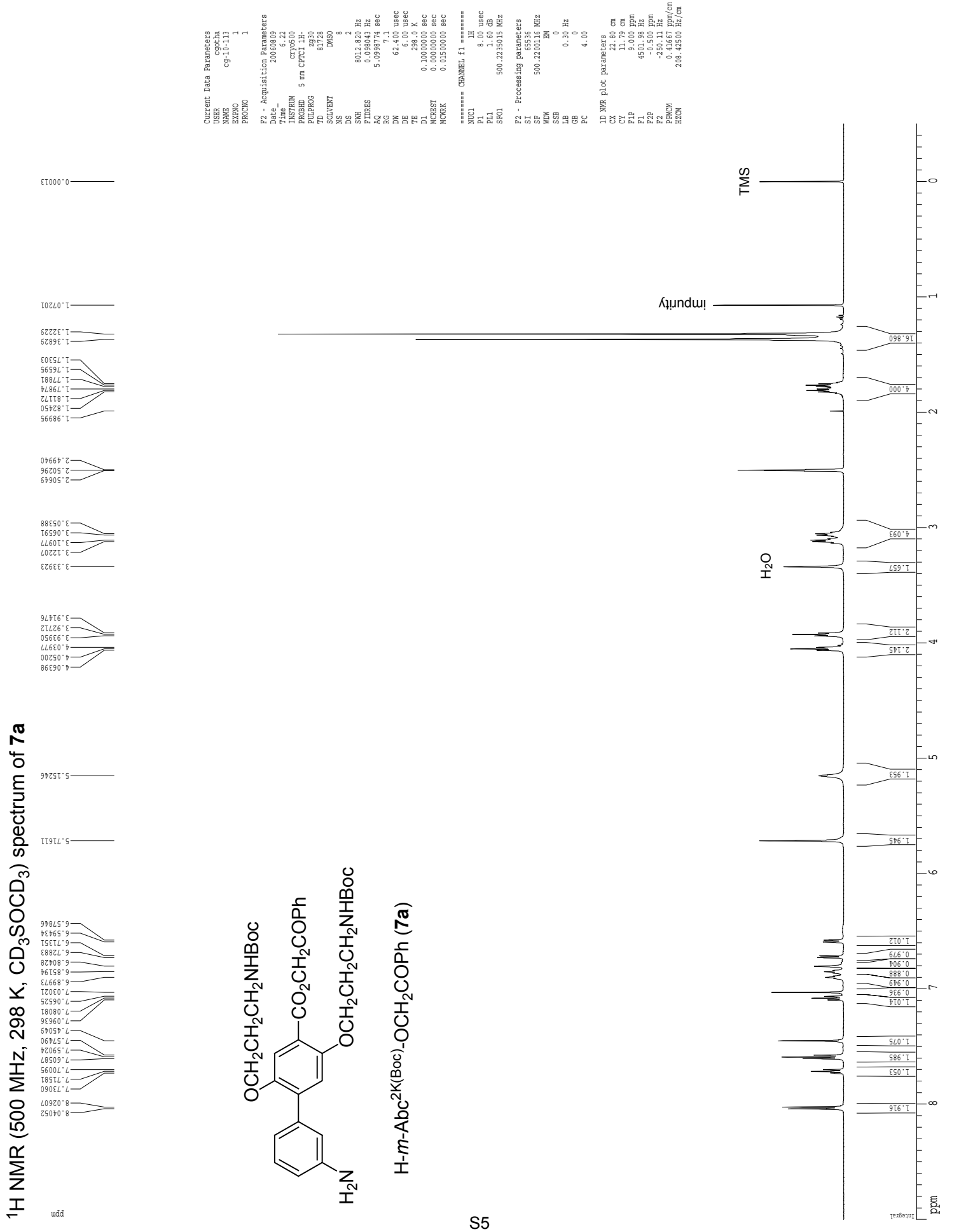
Supporting Information Table of Contents

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

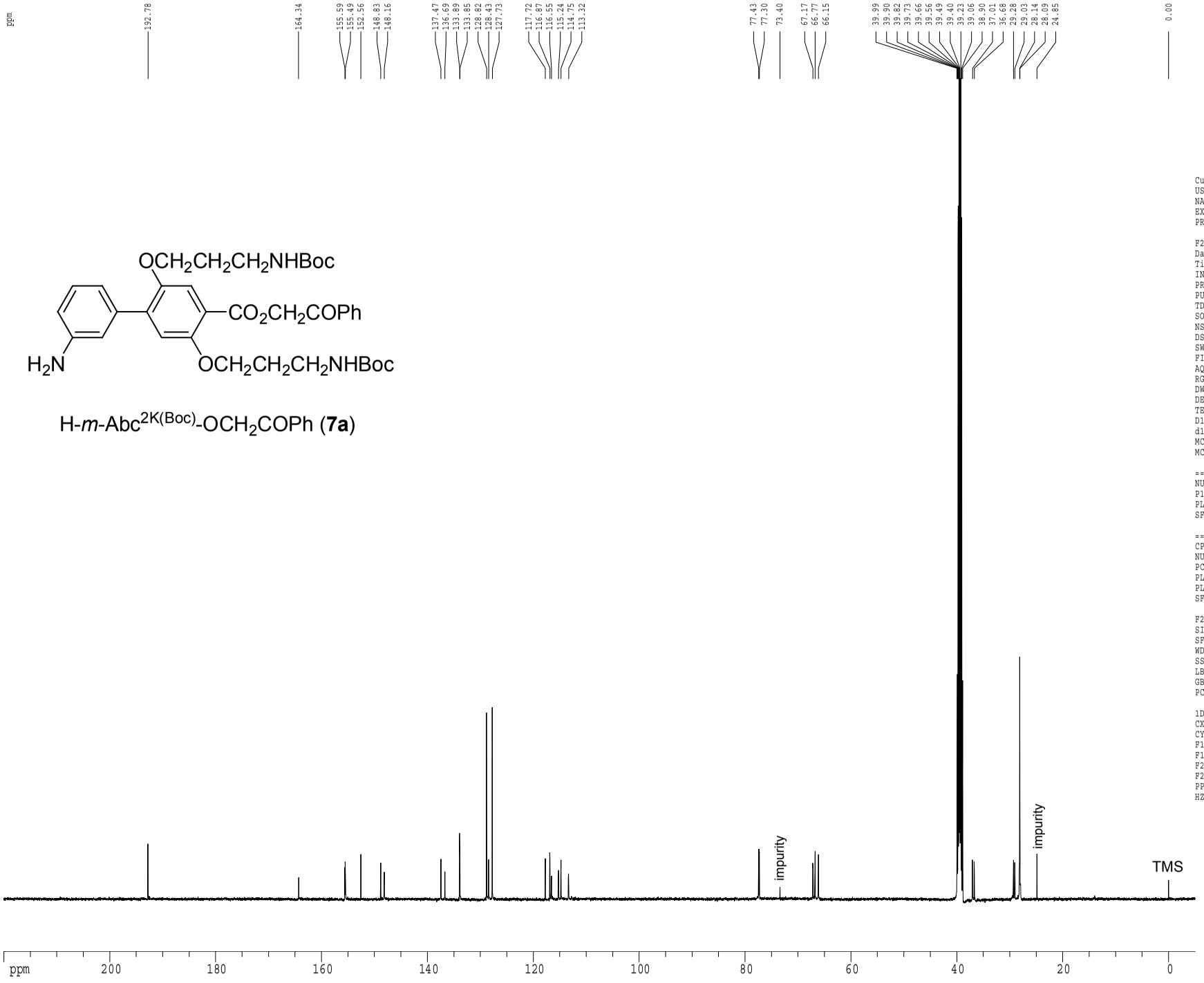


* Diether **6** was prepared in a previous publication.[1]

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


^{1}H NMR (500 MHz, 298 K, CD_3SOCD_3) spectrum of **7a**
$OCH_2CH_2CH_2NHBoc$
CO_2CH_2COPh
$OCH_2CH_2CH_2NHBoc$
H_2N
H-*m*-Abc$^{2K(Boc)}$-OCH_2COPh (**7a**)
TMS
impurity
H_2O
ppm

^{13}C NMR (125 MHz, 298 K, CD_3SOCD_3) spectrum of **7a**



H-*m*-Abc$^{2K(Boc)}$-OCH_2COPh (**7a**)

S6

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```

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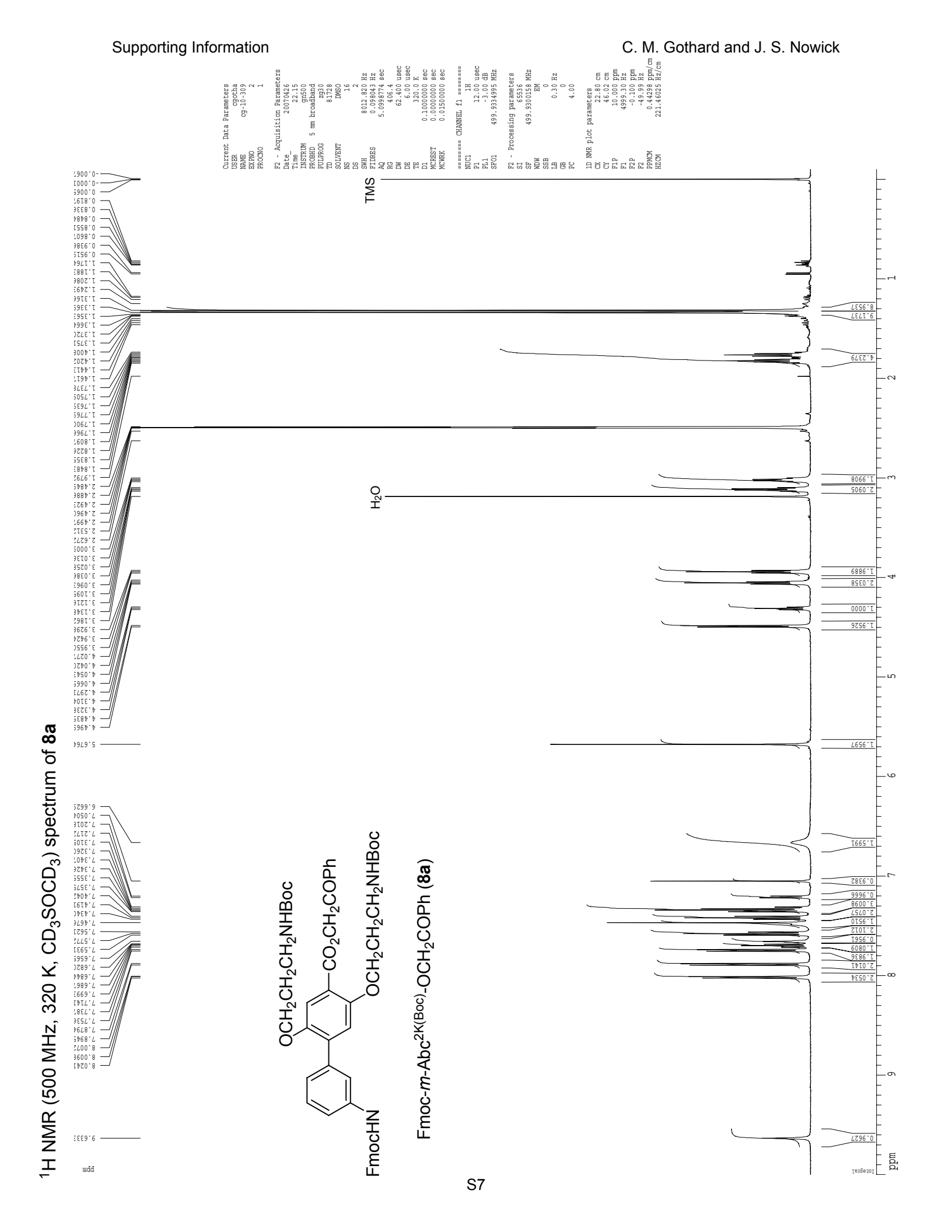
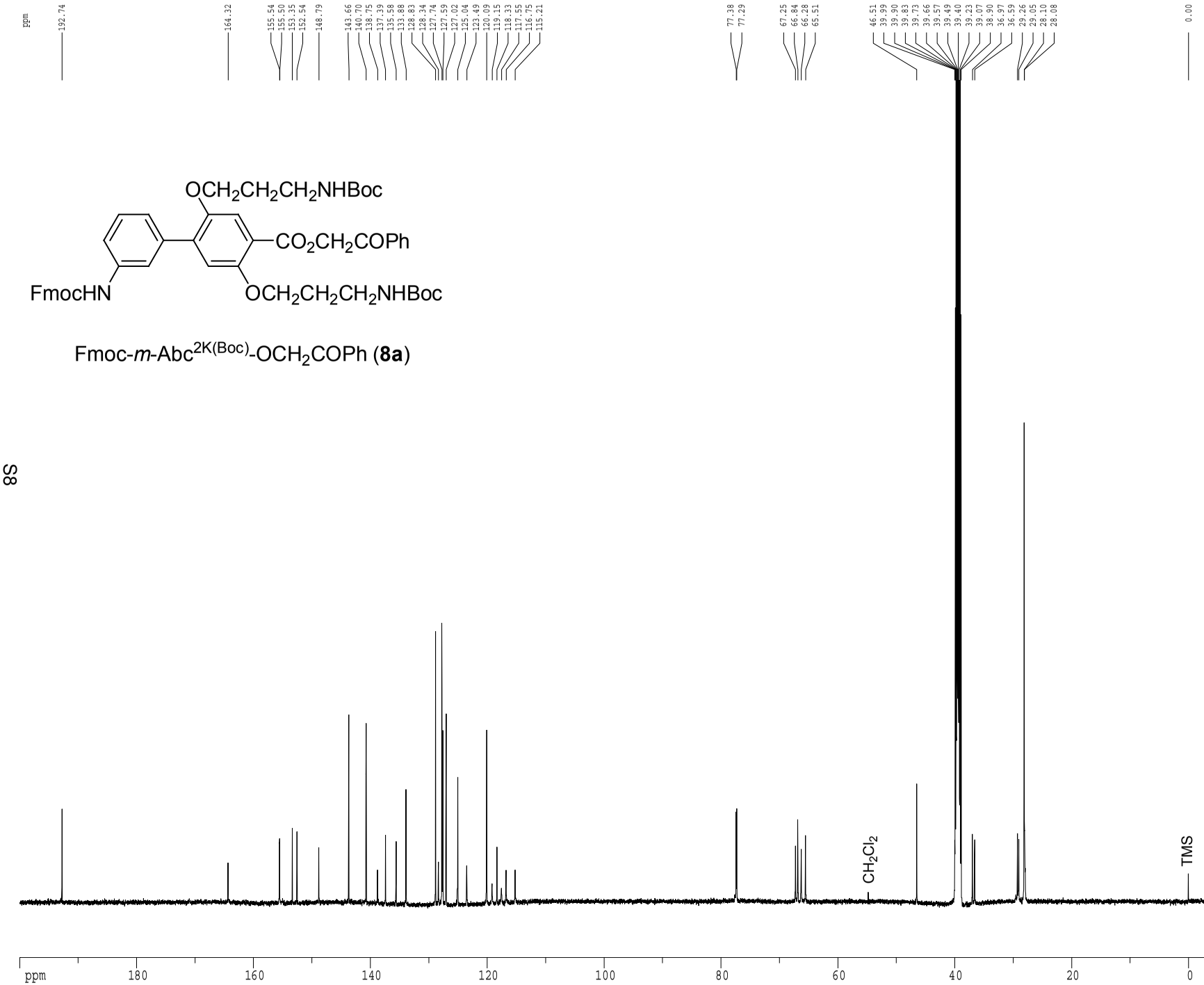
^{1}H NMR (500 MHz, 320 K, CD_3SOCD_3) spectrum of **8a**



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

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D1              0.25000000 sec
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======== 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

F2 - Processing parameters
SI                   65536
SF            125.7804942 MHz
WDW                     EM
SSB                      0
LB                  1.00 Hz
GB                       0
PC                    2.00

1D NMR plot parameters
CX                  22.80 cm
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F1              25156.10 Hz
F2P                -5.000 ppm
F2               -628.90 Hz
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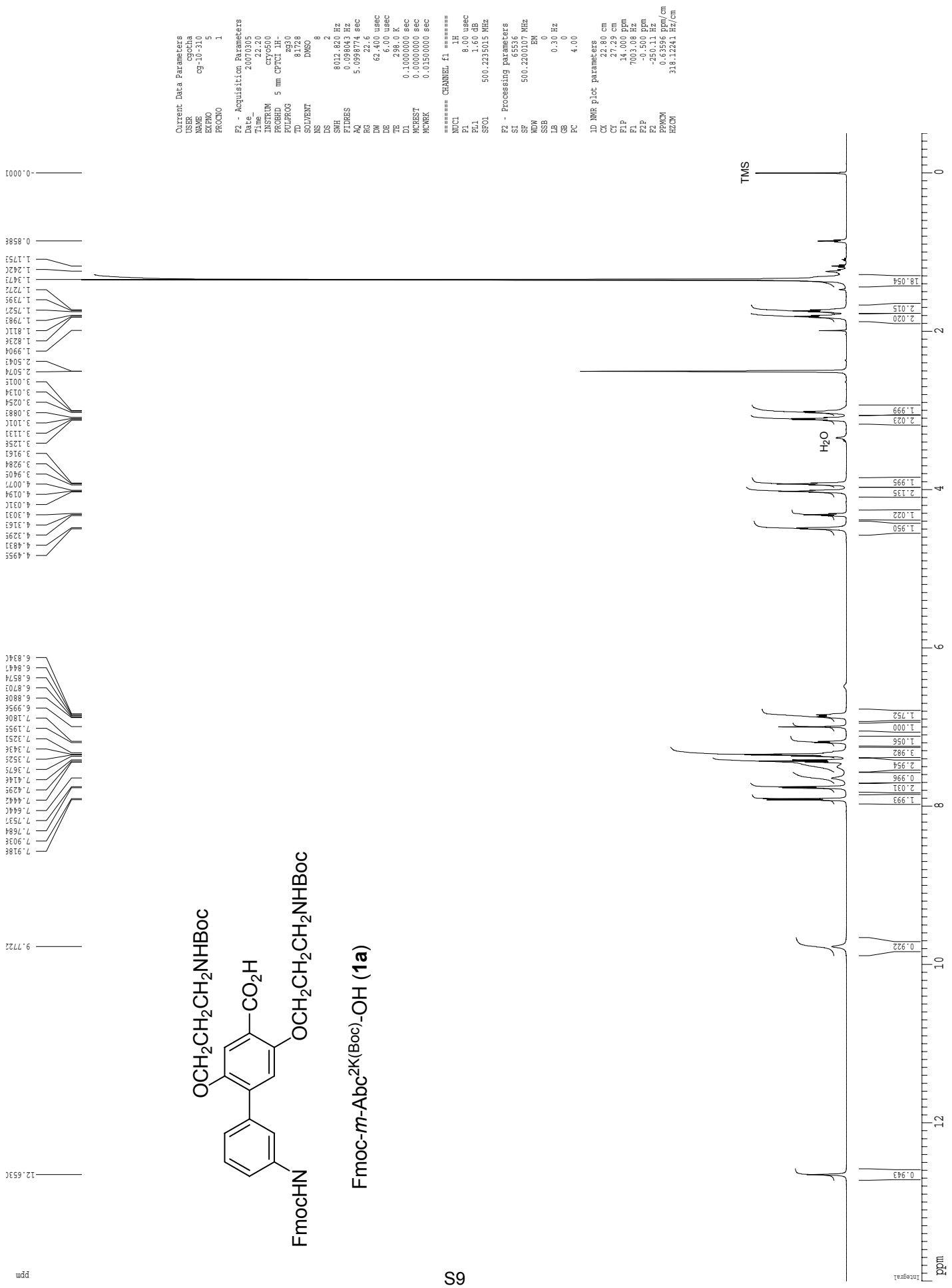
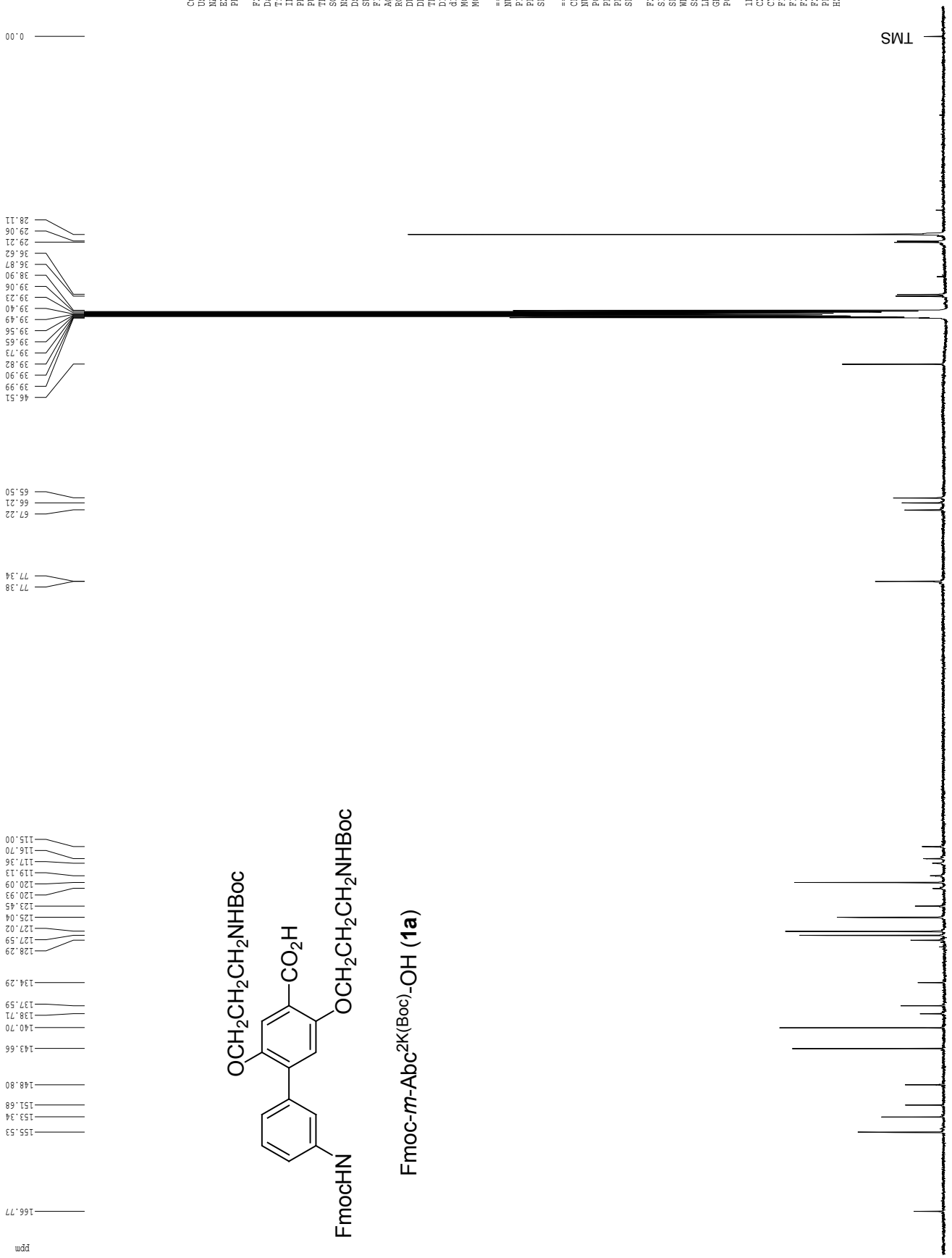
^{1}H NMR (500 MHz, 298 K, CD_3SOCD_3) spectrum of **1a**



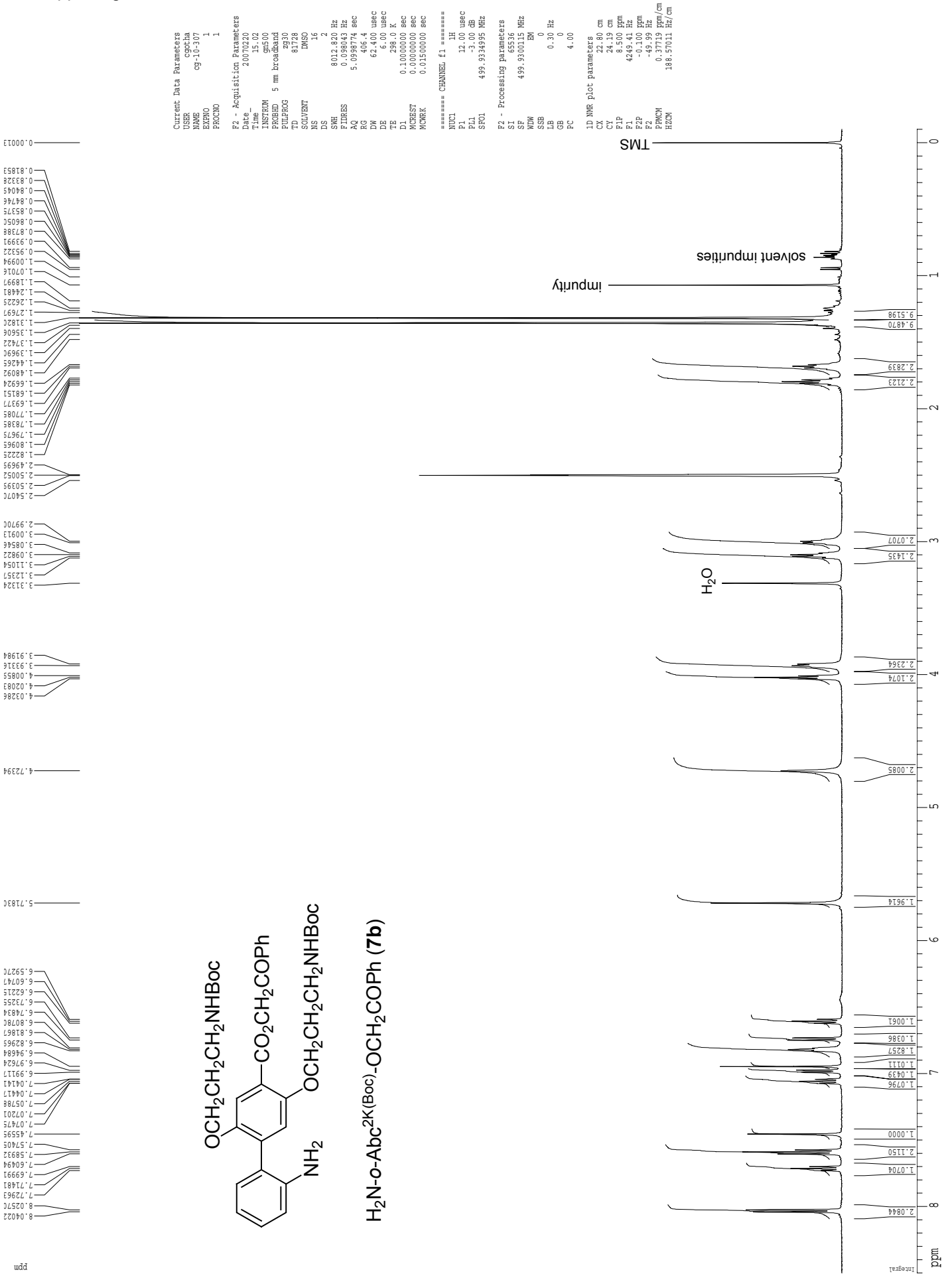
Fmoc-*m*-Abc$^{2K(Boc)}$-OH (**1a**)

^{13}C NMR (125 MHz, 298 K, CD_3SOCD_3) spectrum of **1a**



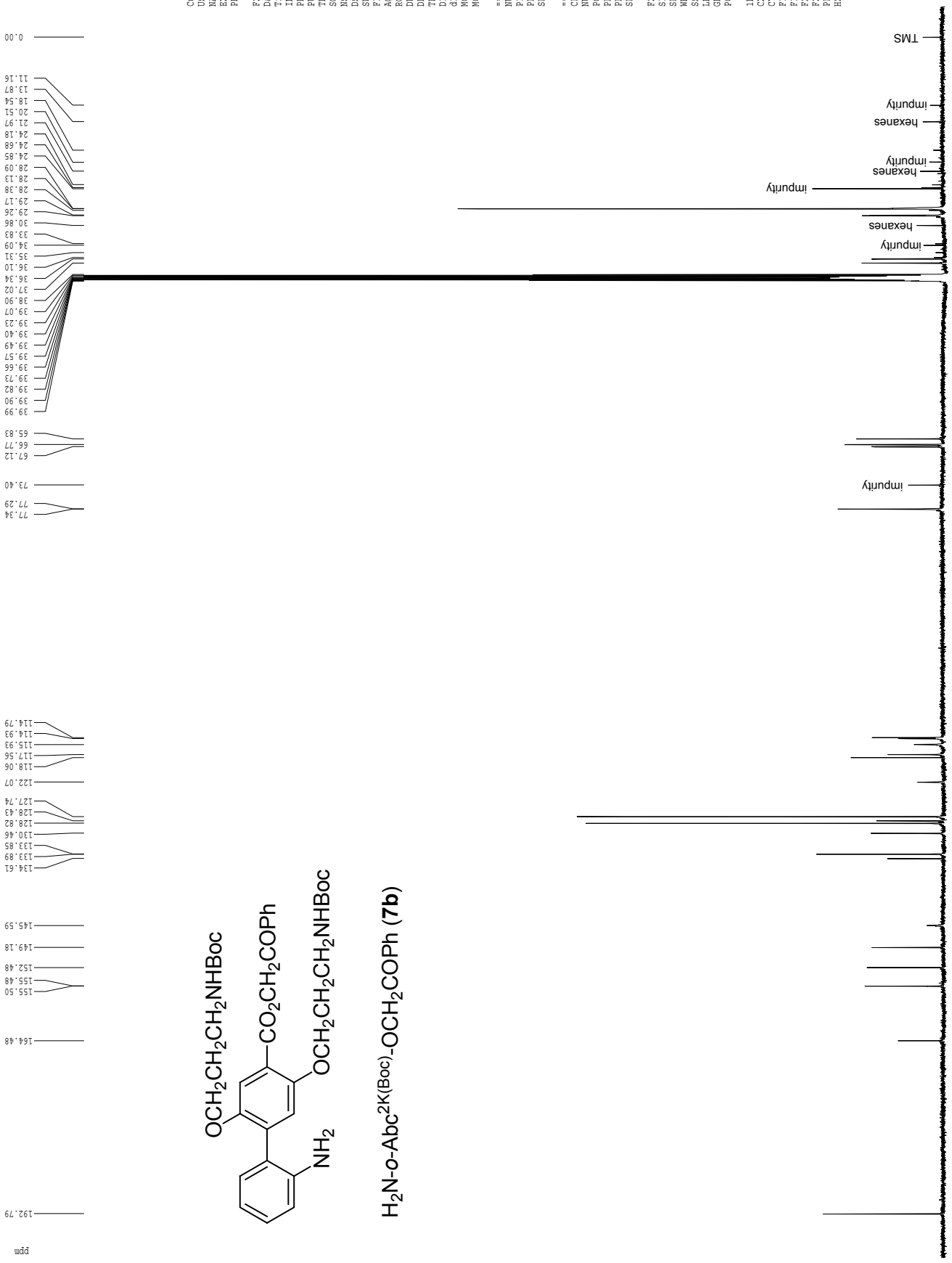
Fmoc-*m*-Abc$^{2K(Boc)}$-OH (**1a**)

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



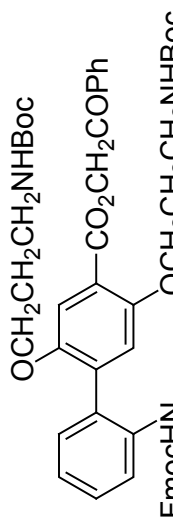
H_2N-*o*-$Abc^{2K(Boc)}$-OCH_2COPh (**7b**)

^{13}C NMR (125 MHz, 298 K, CD_3SOCD_3) spectrum of **7b**



H_2N-*o*-Abc$^{2K(Boc)}$-OCH_2COPh (**7b**)

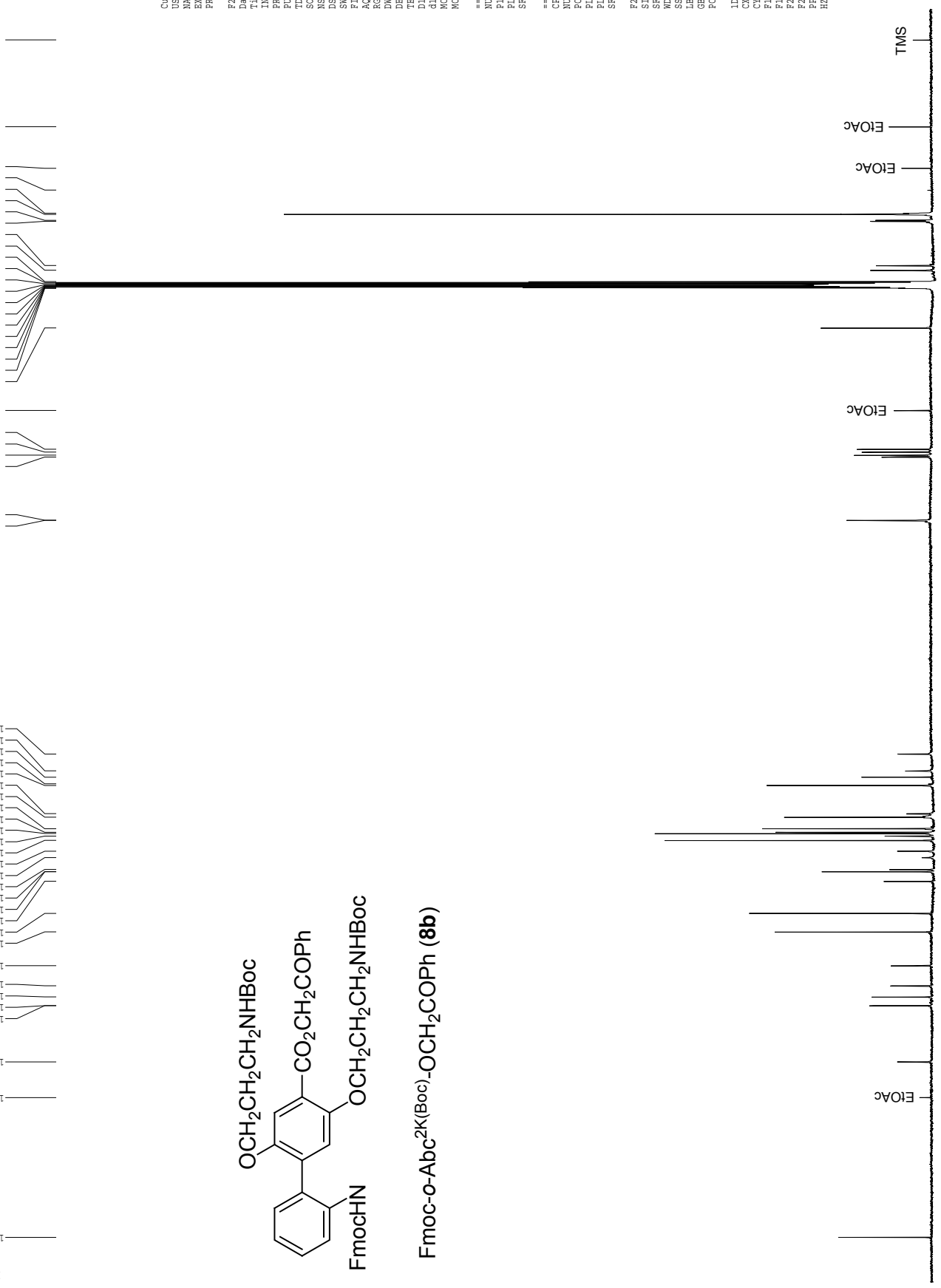
^{1}H NMR (500 MHz, 298 K, CD_3SOCD_3) spectrum of **8b**



Fmoc-*o*-Abc$^{2K(Boc)}$-OCH_2COPh (**8b**)

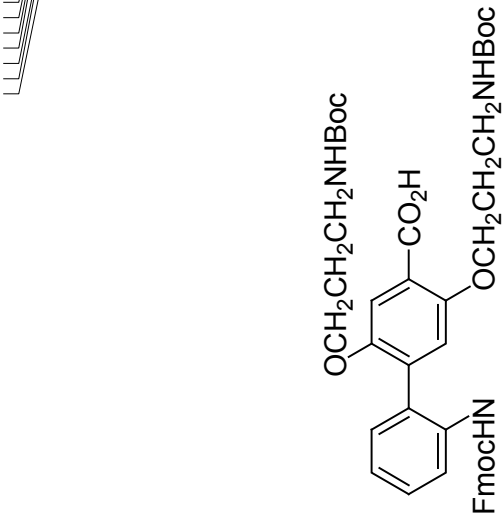
S13

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

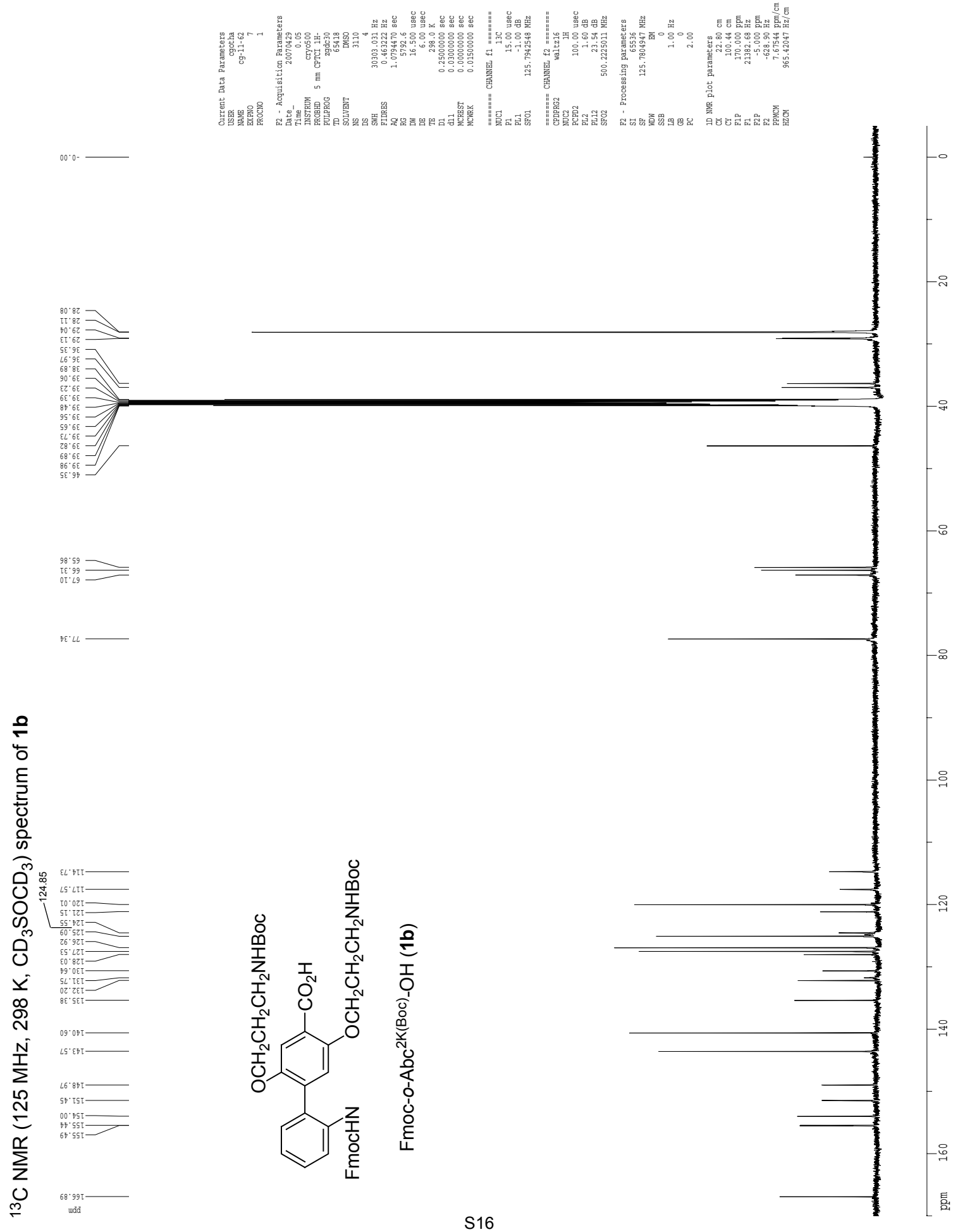


Fmoc-*o*-Abc$^{2K(Boc)}$-OCH_2COPh (**8b**)

^{1}H NMR (500 MHz, 320 K, CD_3SOCD_3) spectrum of **1b**



Fmoc-o-Abc$^{2K(Boc)}$-OH (**1b**)


^{13}C NMR (125 MHz, 298 K, CD_3SOCD_3) spectrum of **1b**
Fmoc-o-Abc$^{2K(Boc)}$-OH (**1b**)
FmocHN
CO_2H
$OCH_2CH_2CH_2NHBoc$
$OCH_2CH_2CH_2NHBoc$
124.85
ppm

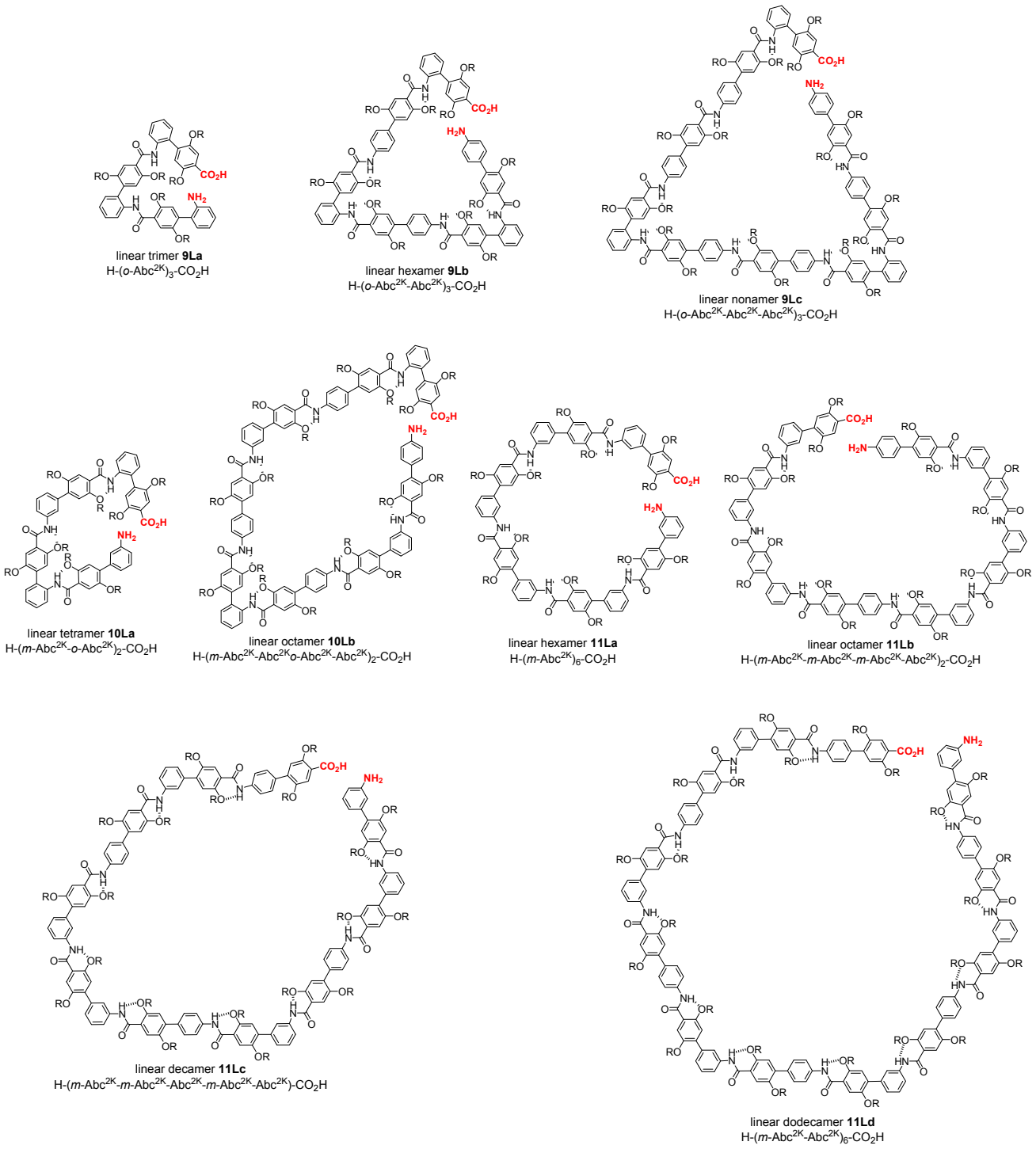
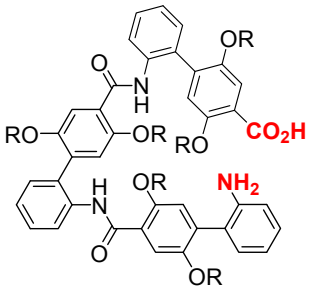


Figure S1. Linear Abc^{2K} oligomers (**9La**-**c**, **10La**-**b**, and **11La**-**d**) showing the amino and carboxylic acid groups (red) used in macrocyclization. (R = $CH_2CH_2CH_2NHBoc$)

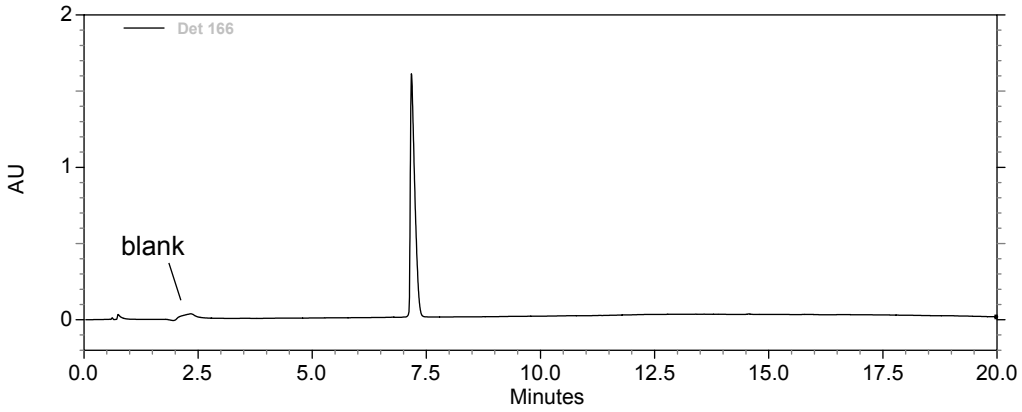
trimer H-(*o*-Abc^{2K})$_3$-CO$_2$H (**9La**)
Analytical RP-HPLC chromatograph and mass spectrum (ESI-MS)



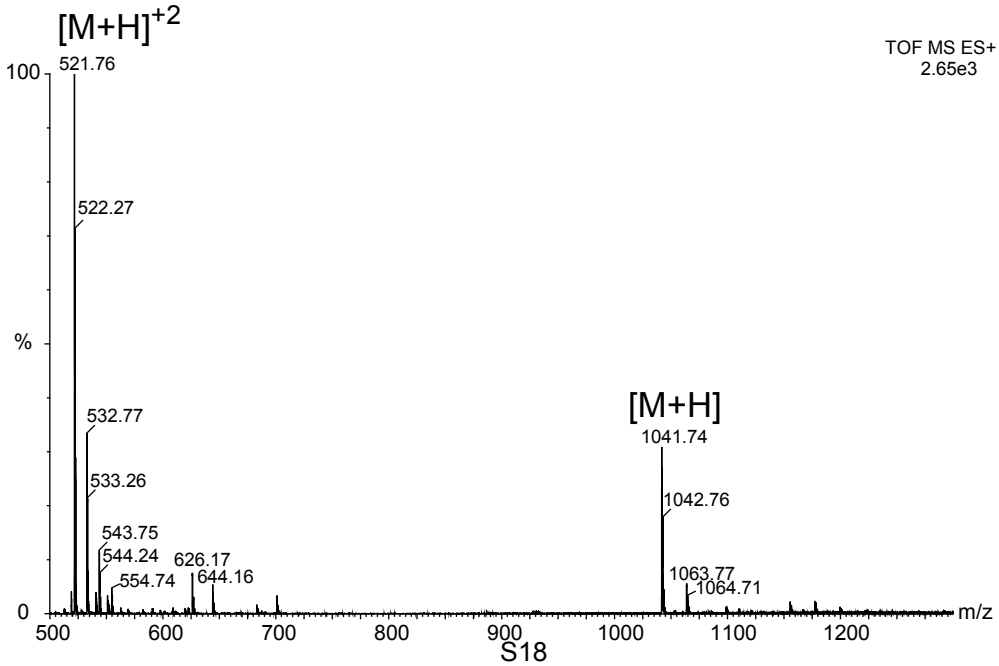
R = $CH_2CH_2CH_2NH_2$TFA

linear trimer **9La**
H-(*o*-Abc^{2K})$_3$-CO$_2$H

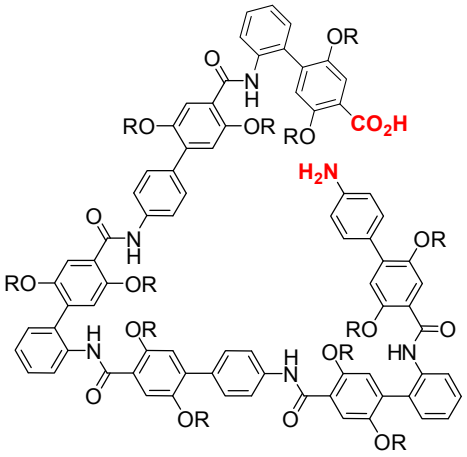
(a) Analytical RP-HPLC (0-30% acetonitrile with 0.1% TFA over 10 min, λ = 214)



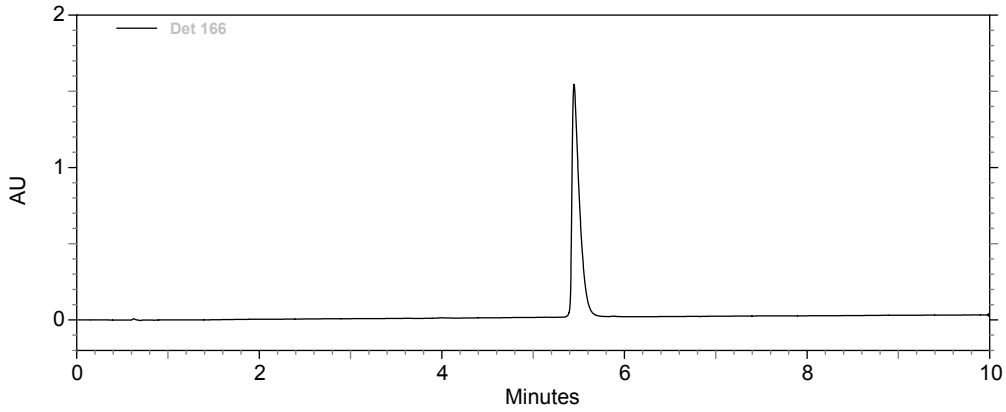
(b) ESI Mass spectrum. (Calcd exact mass for $C_{57}H_{71}N_9O_{10}$ [M] = 1041.53)



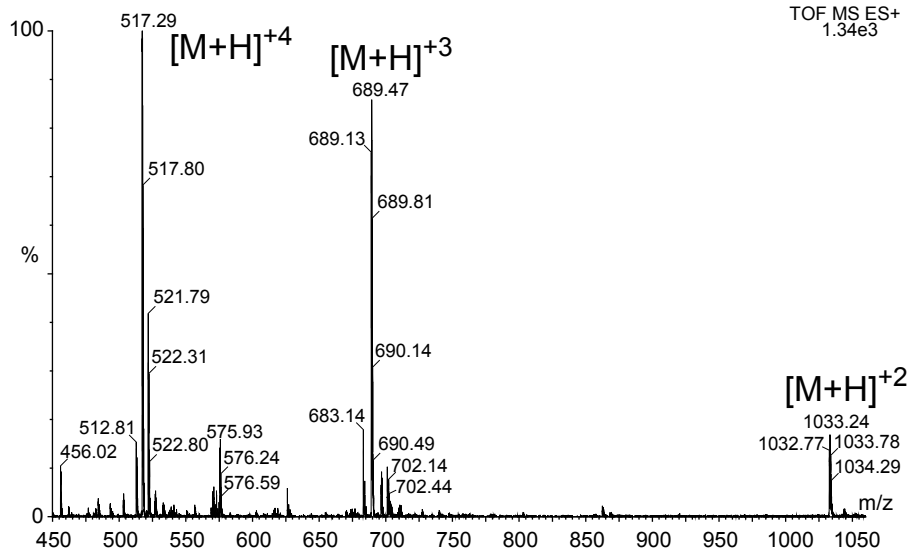
linear hexamer: H-(*p*-Abc^{2K}-*o*-Abc^{2K})$_3$-CO_2H (**9Lb**)
Analytical RP-HPLC chromatograph and mass spectrum (ESI-MS)



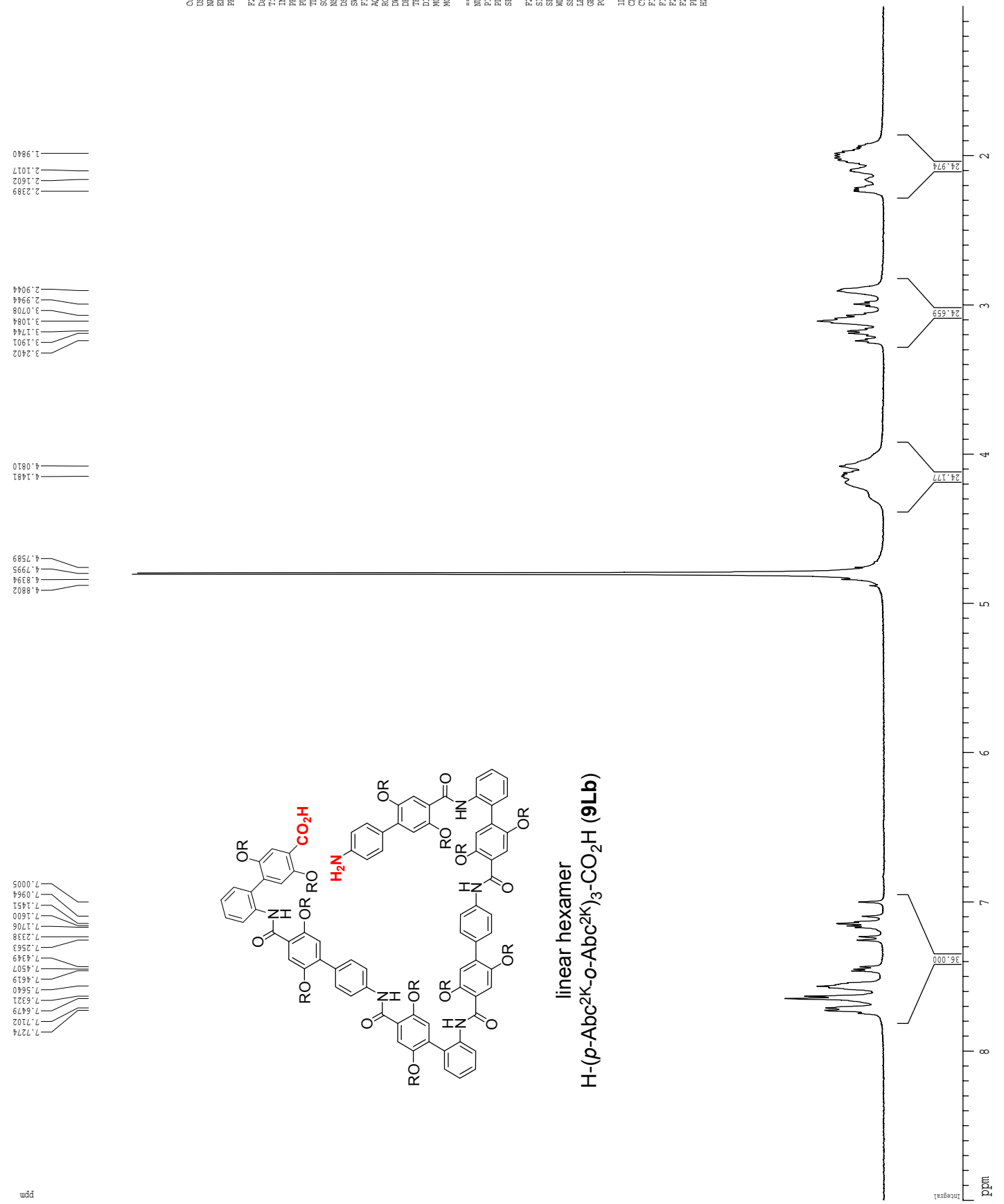
(a) Analytical RP-HPLC (5-50% acetonitrile with 0.1% TFA over 10 min, λ = 214)



(b) ESI Mass spectrum. (Calcd exact mass for $C_{114}H_{140}N_{18}O_{19}$ [M] = 2065.05)

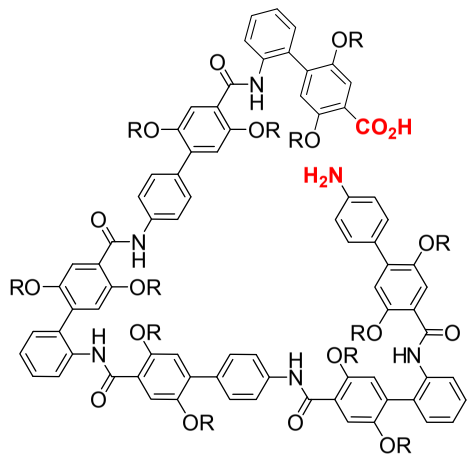


^{1}H NMR (500 MHz, 298 K, D_2O) spectrum of linear hexamer **9Lb**



linear hexamer
H-(*p*-Abc^{2K}-*o*-Abc^{2K})$_3$-CO_2H (**9Lb**)

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



linear hexamer
H-(*p*-Abc^{2K}-*o*-Abc^{2K})$_3$-CO_2H (**9Lb**)

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PROCNO                  1

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RG                 812.7
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DE                 6.00 usec
TE                298.0 K
D1            0.10000000 sec
MCREST        0.00000000 sec
MCWRK         0.01500000 sec

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

F2 - Processing parameters
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1D NMR plot parameters
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CY                82.40 cm
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F2P               6.000 ppm
F2              2999.58 Hz
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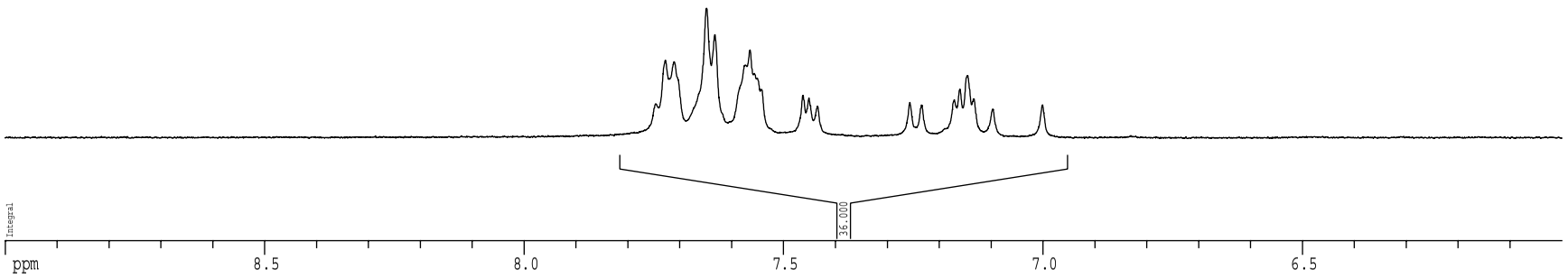

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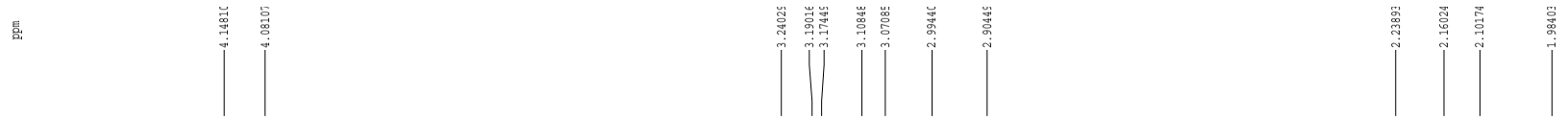
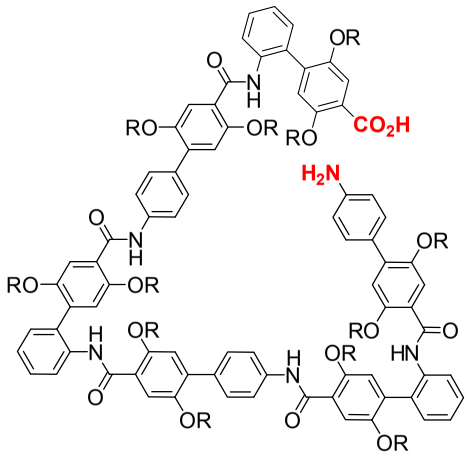
^{1}H NMR (500 MHz, 298 K, D_2O) spectrum of linear hexamer **9Lb** (aliphatic region)





linear hexamer
H-(*p*-Abc^{2K}-*o*-Abc^{2K})$_3$-CO_2H (**9Lb**)

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NAME           cg-11-32
EXPNO                 1
PROCNO                1

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TD                81728
SOLVENT             D2O
NS                   32
DS                    2
SWH           8012.820 Hz
FIDRES        0.098043 Hz
AQ            5.0999398 sec
RG                812.7
DW               62.400 usec
DE                6.00 usec
TE                298.0 K
D1           0.10000000 sec
MCREST       0.00000000 sec
MCWRK        0.01500000 sec

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

F2 - Processing parameters
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SF          499.9300035 MHz
WDW                  EM
SSB                   0
LB                 0.30 Hz
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PC                 4.00

1D NMR plot parameters
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HZCM          59.20224 Hz/cm
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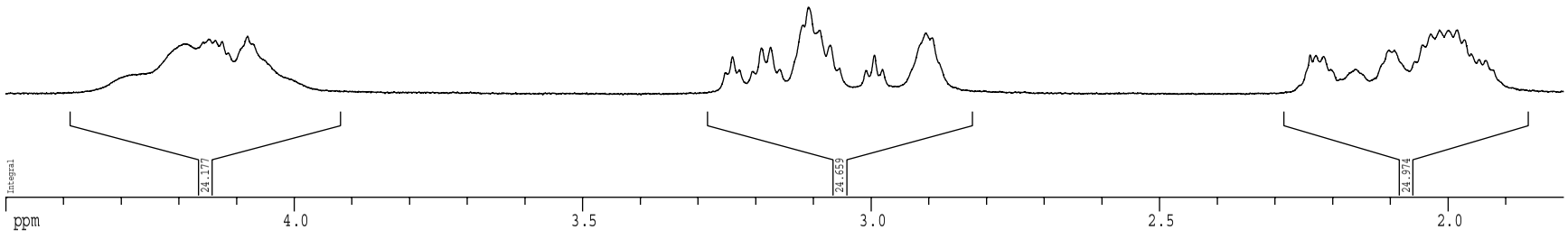

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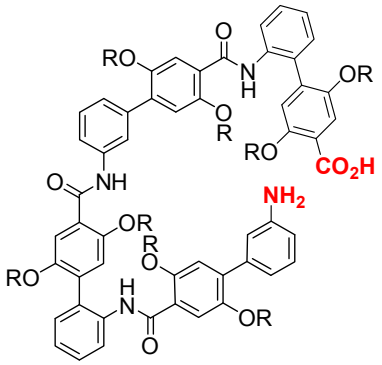
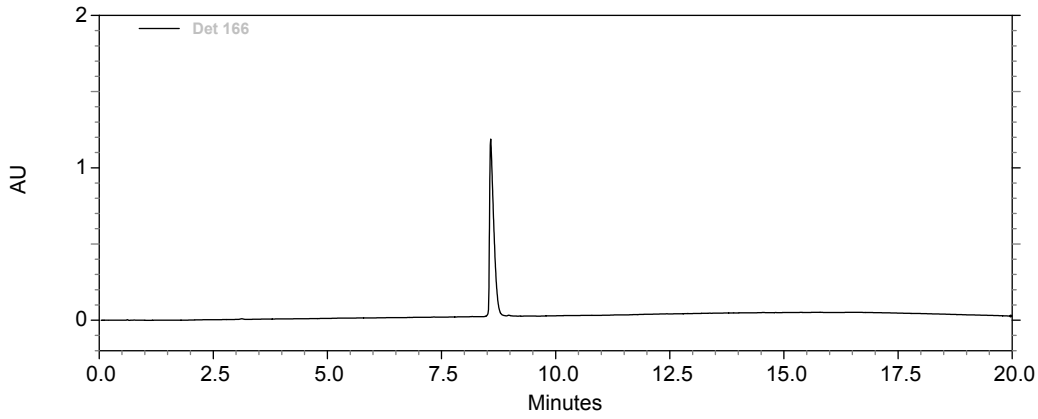
linear tetramer: H-(*m*-Abc^{2K}-*o*-Abc^{2K})$_2$-CO$_2$H (**10La**)
Analytical RP-HPLC chromatograph and mass spectrum (ESI-MS)

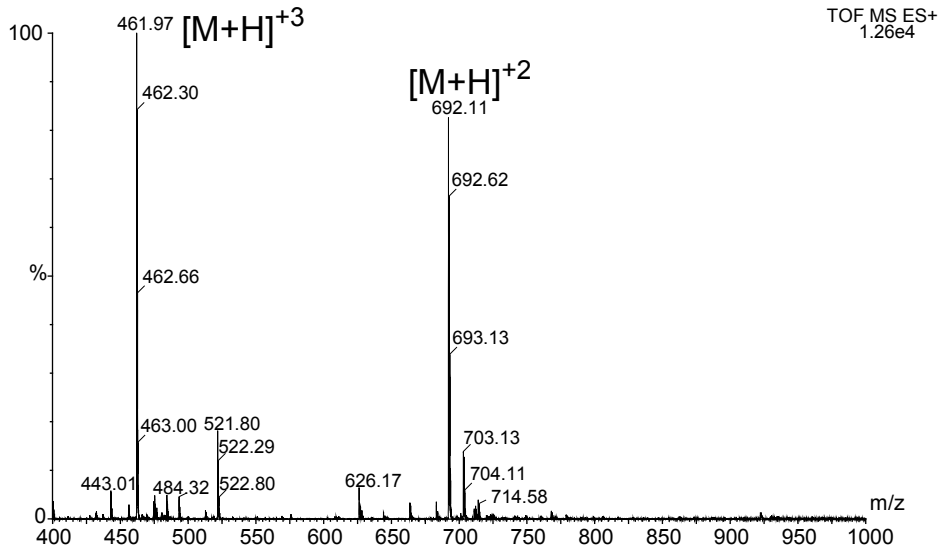


R = $CH_2CH_2CH_2NH_2$TFA

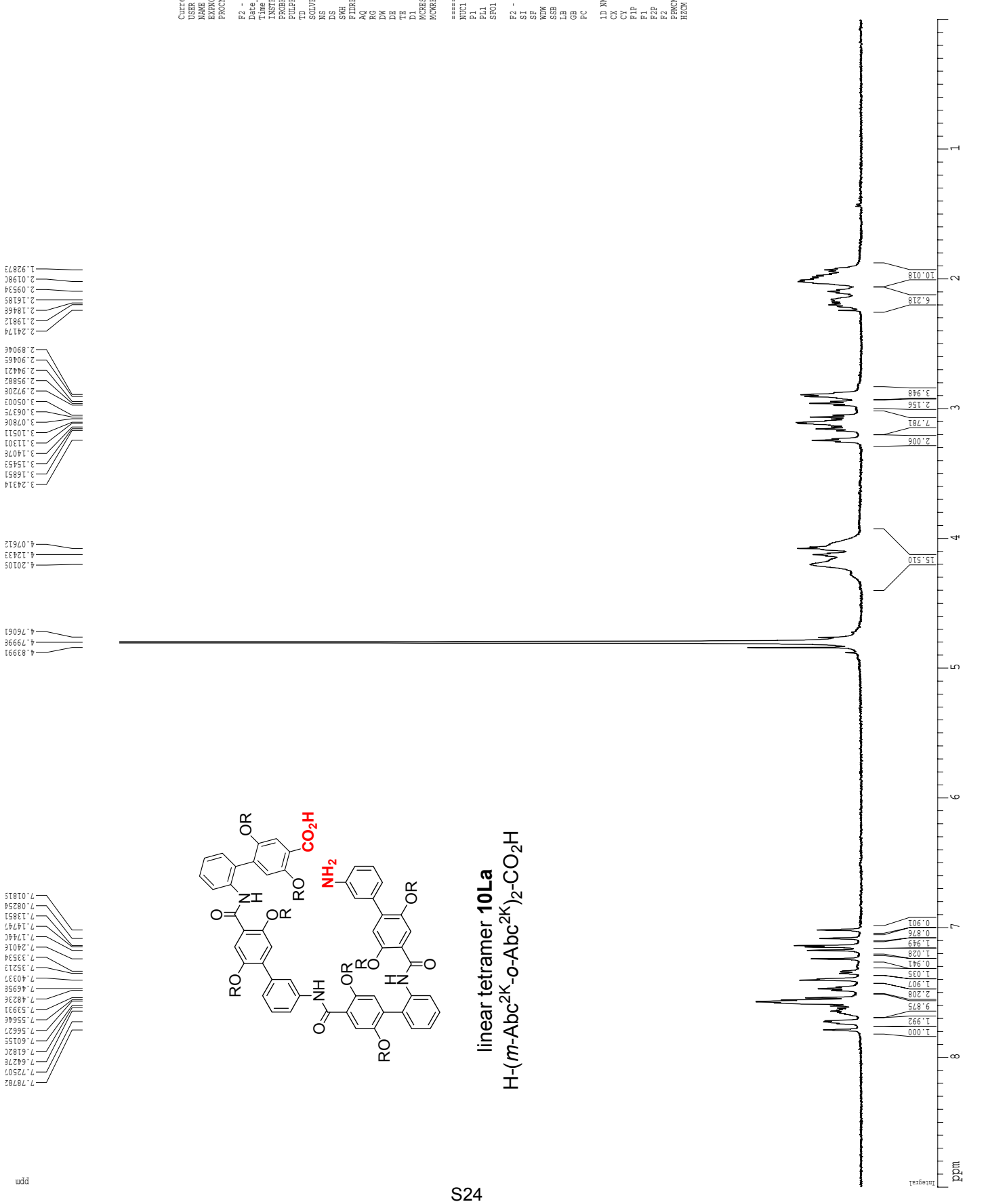
(a) Analytical RP-HPLC (0-30% acetonitrile with 0.1% TFA over 10 min, λ = 214)



(b) ESI Mass spectrum. (Calcd exact mass for $C_{76}H_{94}N_{12}O_{13}$ [M] = 1382.71)

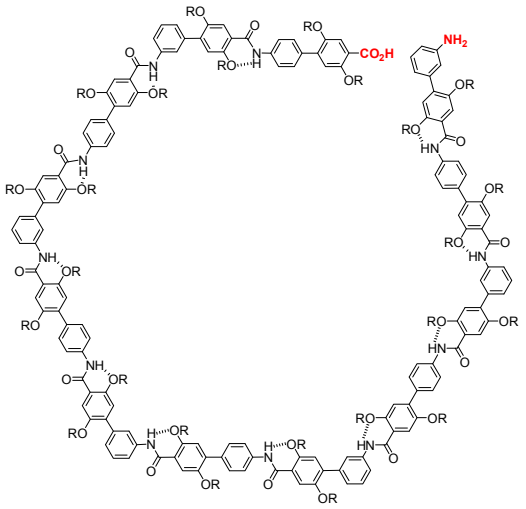


^{1}H NMR (500 MHz, 298 K, D_2O) spectrum of linear tetramer **10La**



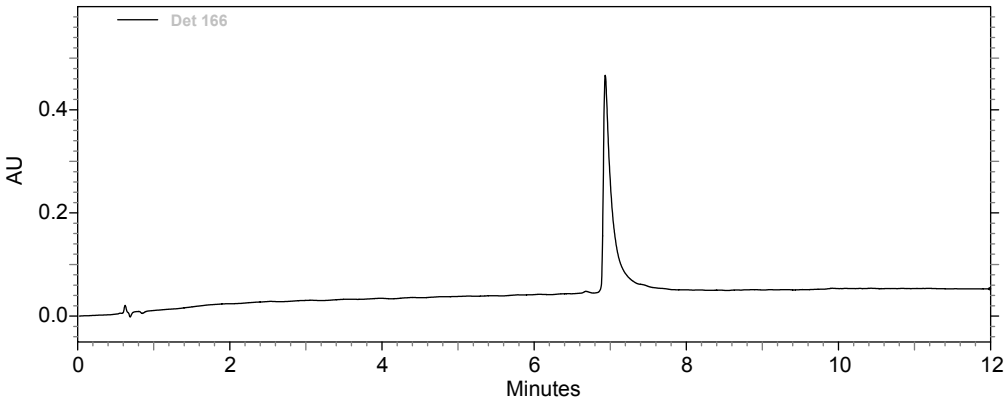
linear tetramer **10La**
H-(*m*-Abc^{2K}-*o*-Abc^{2K})$_2$-CO_2H

linear dodecamer: H-(*m*-Abc^{2K}-*p*-Abc^{2K})$_6$-CO$_2$H (**11Ld**)
Analytical RP-HPLC chromatograph and mass spectrum (ESI-MS)

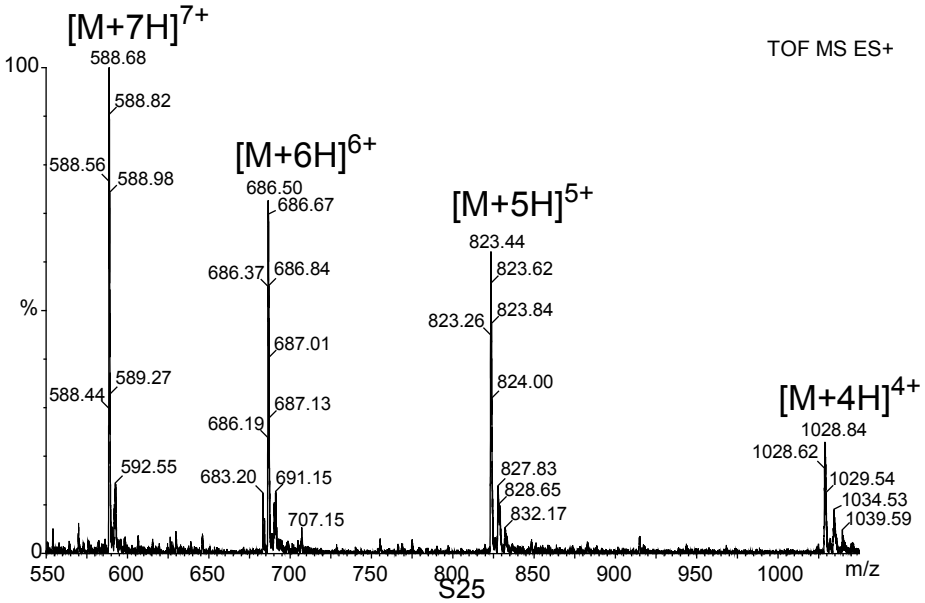


R = $CH_2CH_2CH_2NH_2TFA$

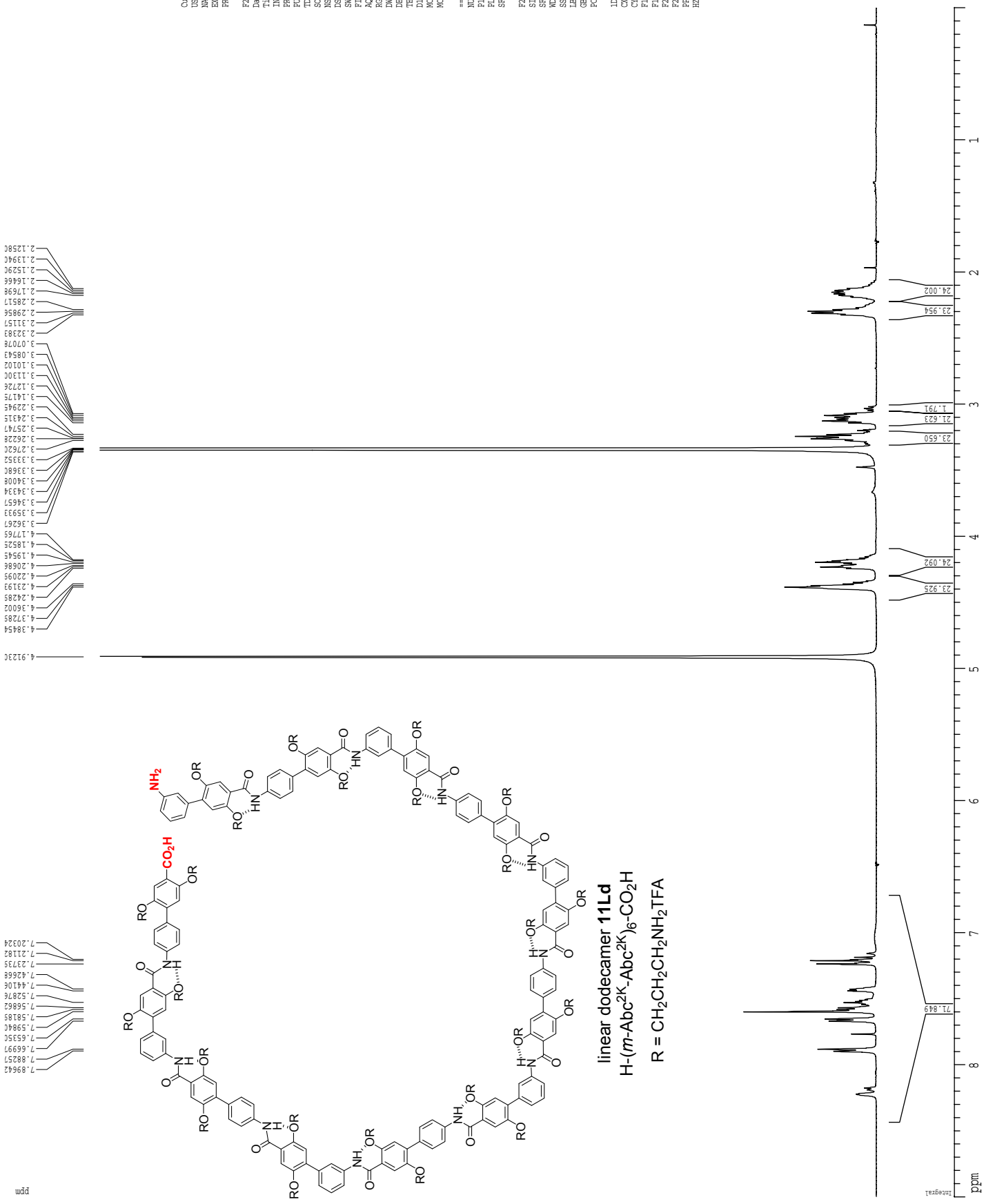
(a) Analytical RP-HPLC (5-50% acetonitrile with 0.1% TFA over 10 min, λ = 214)



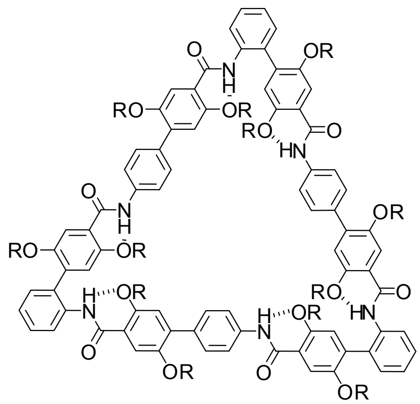
(b) ESI Mass spectrum. (Calcd exact mass for $C_{228}H_{278}N_{36}O_{37}$ [M] = 4112.10)



^{1}H NMR (500 MHz, 298 K, CD_3OD) spectrum of linear dodecamer **11Ld**



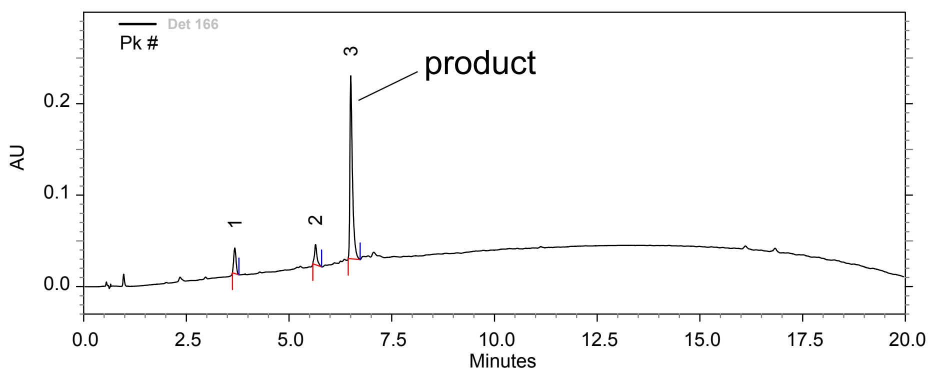
Characterization of crude (*unpurifed*) cyclohexamer **9b**
Analytical RP-HPLC chromatograph and mass spectrum (ESI-MS)



(o-Abc^{2K}-p-Abc^{2K})$_3$

R = $CH_2CH_2CH_2NH_2TFA$

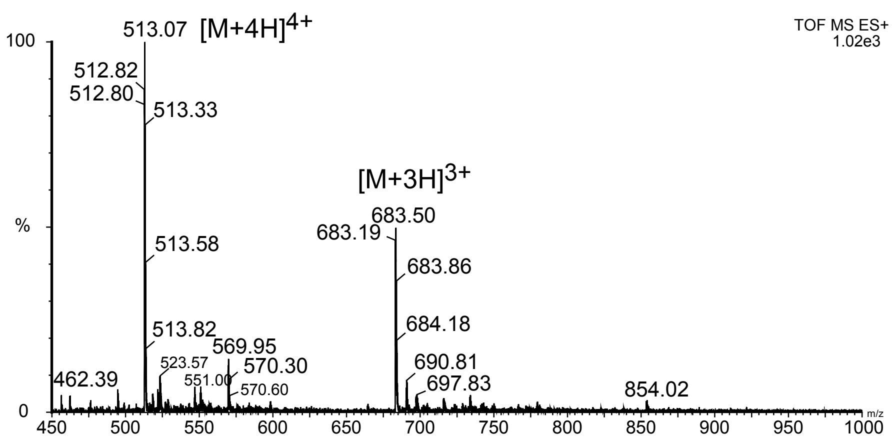
RP-HPLC of crude cyclohexamer triangle **9b**
(5-90% acetonitrile with 0.1% TFA over 20 min, λ = 214)



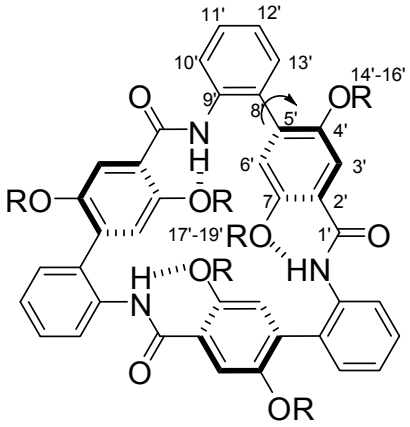
Det 166 Results

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	

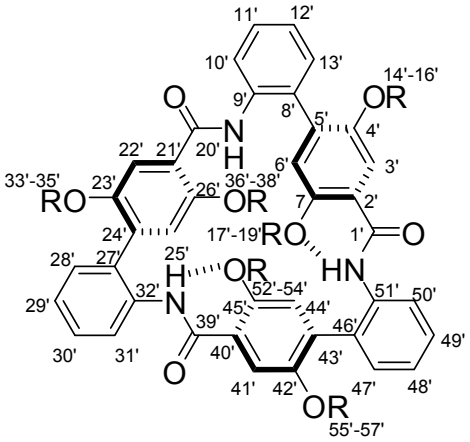
ESI-MS of crude cyclohexamer triangle **9b**
(Exact MS for $C_{114}H_{138}N_{18}O_{18}$ = 2047.04)



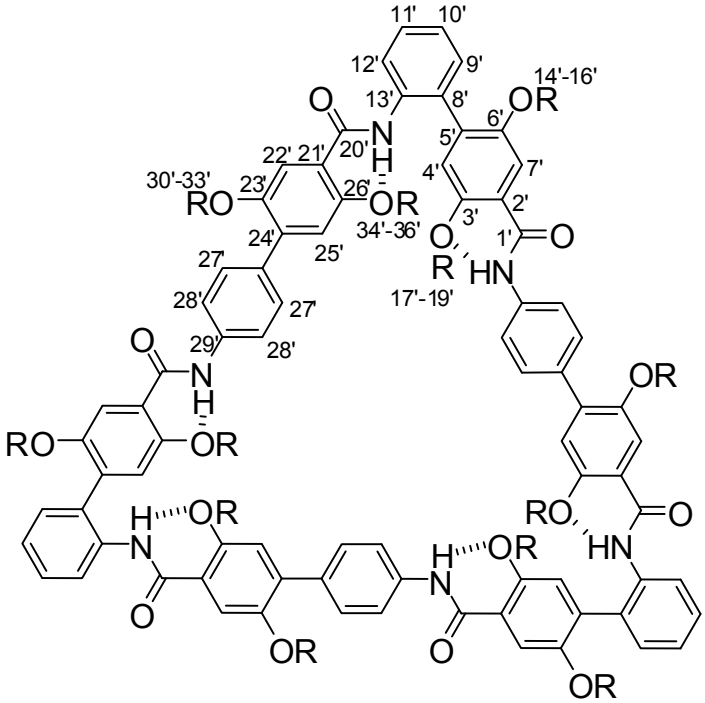
Macrocycle Symmetry (**9a**, **9b**)



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



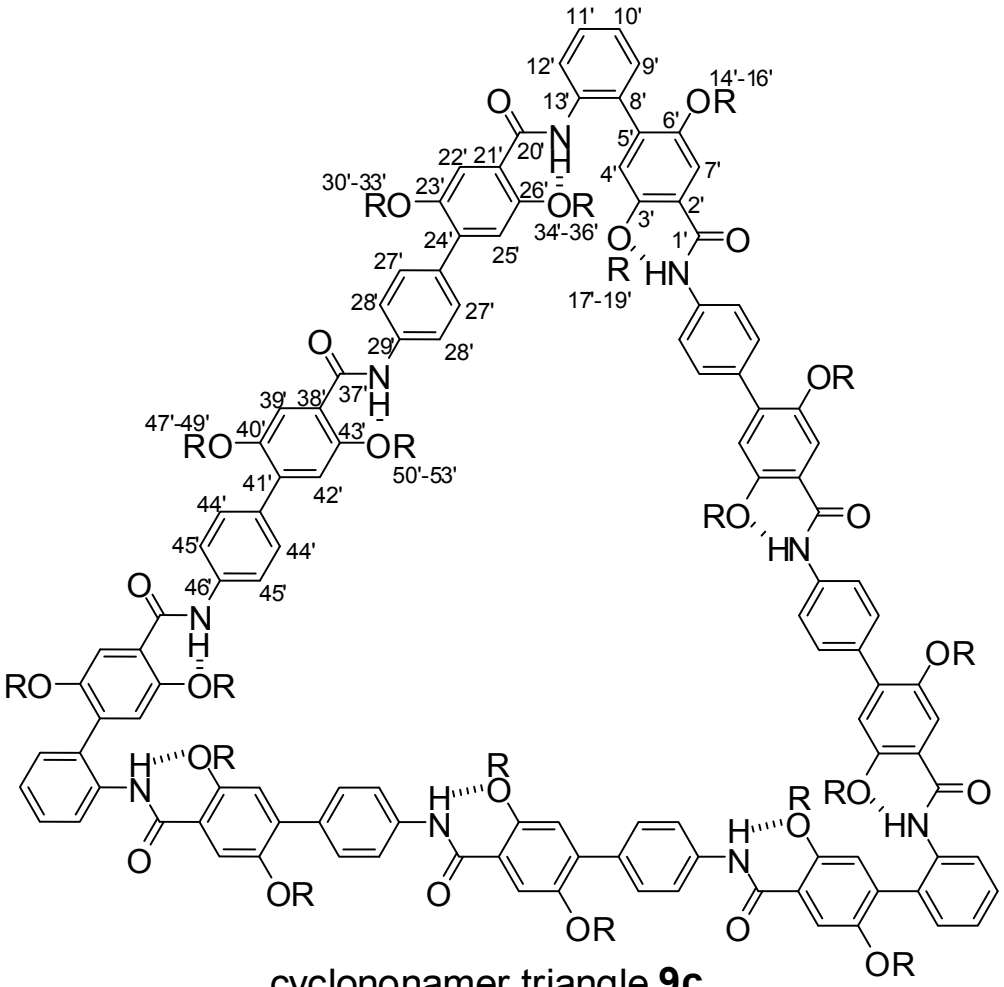
cyclotrimer triangle **9a**
(unsymmetric conformer)



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

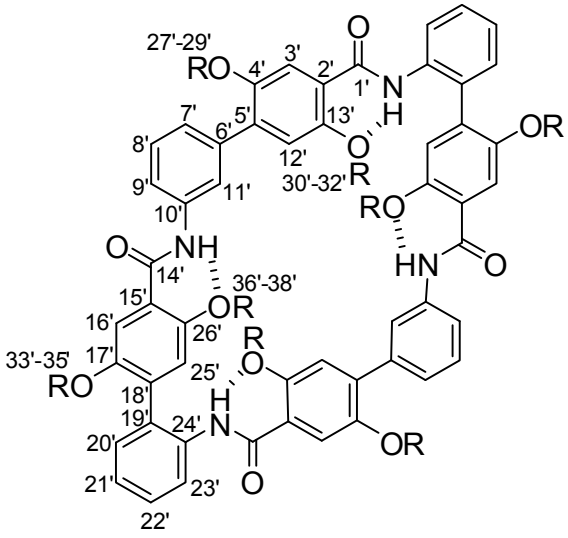
R = $CH_2CH_2CH_2NH_3^+CF_3CO_2^-$

Macrocycle Symmetry (**9c**, **10a, 11a**)

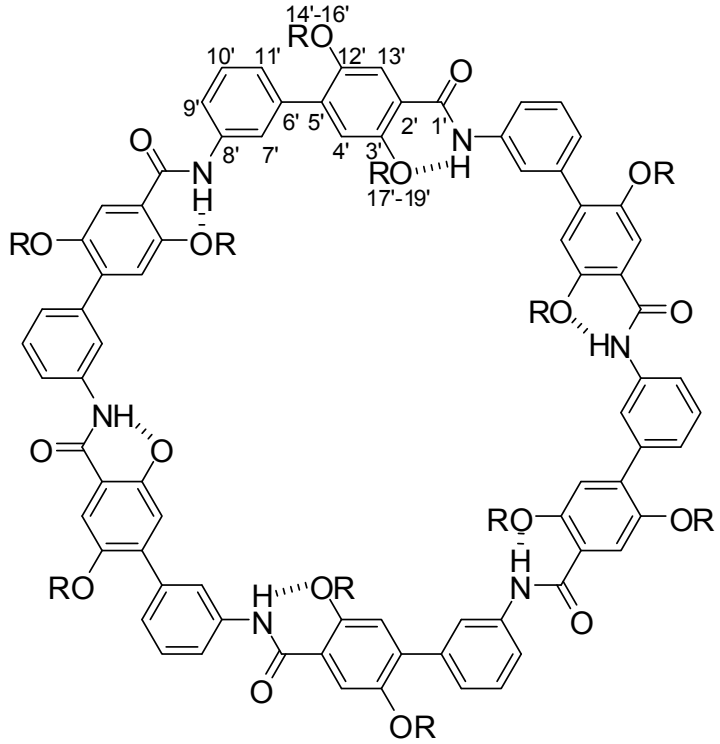


R = $CH_2CH_2CH_2NH_3^+CF_3CO_2^-$

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

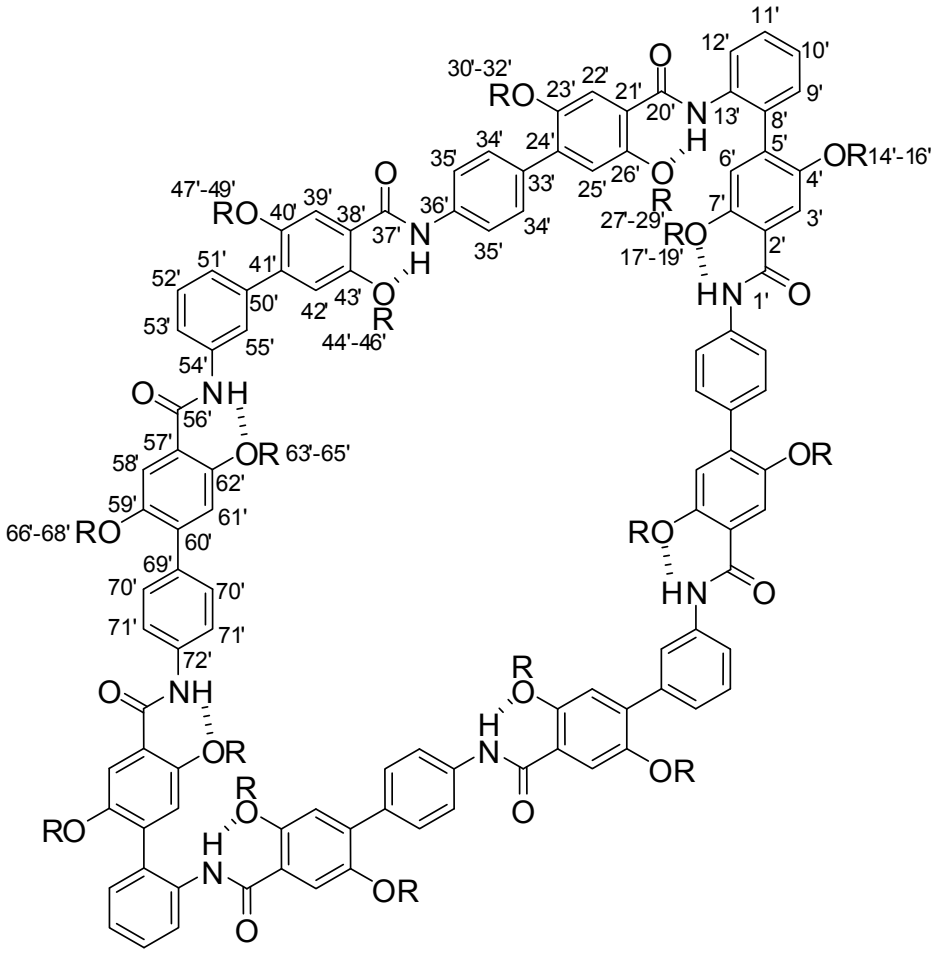


cyclotetramer parallelogram **10a**
(2-fold symmetry)



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

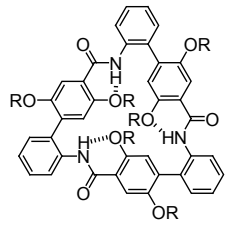
Macrocycle Symmetry (**10b**)



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

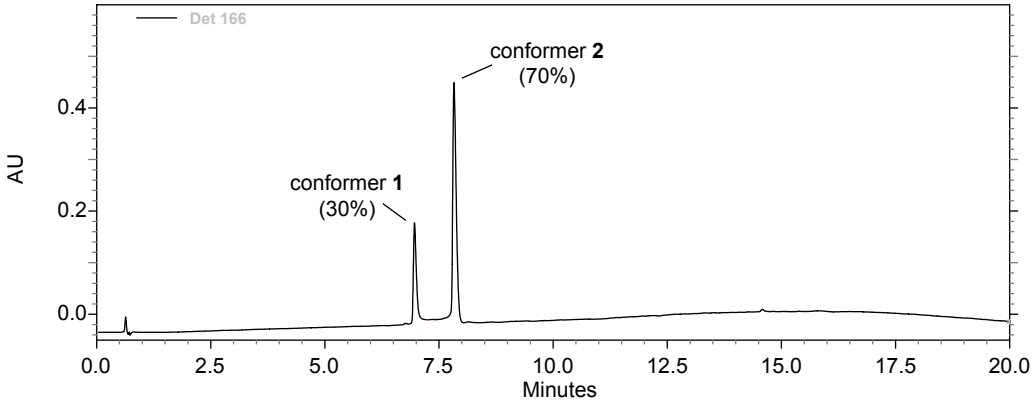
R = $CH_2CH_2CH_2NH_3^+CF_3CO_2^-$

cyclotrimer triangle: cyclo(*o*-Abc^{2K})$_3$ (**9a**)
Analytical RP-HPLC chromatograph and mass spectrum (ESI-MS)

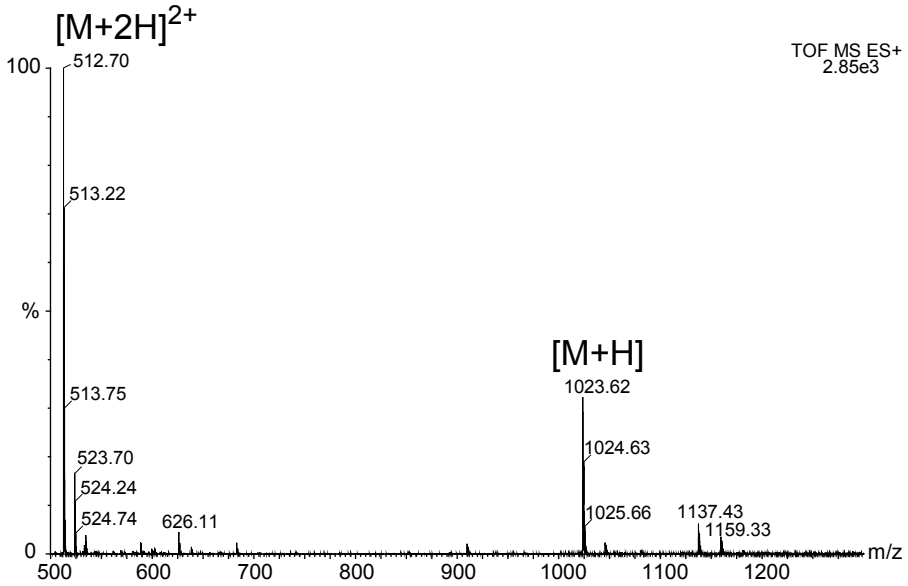


R = $CH_2CH_2CH_2NH_2TFA$

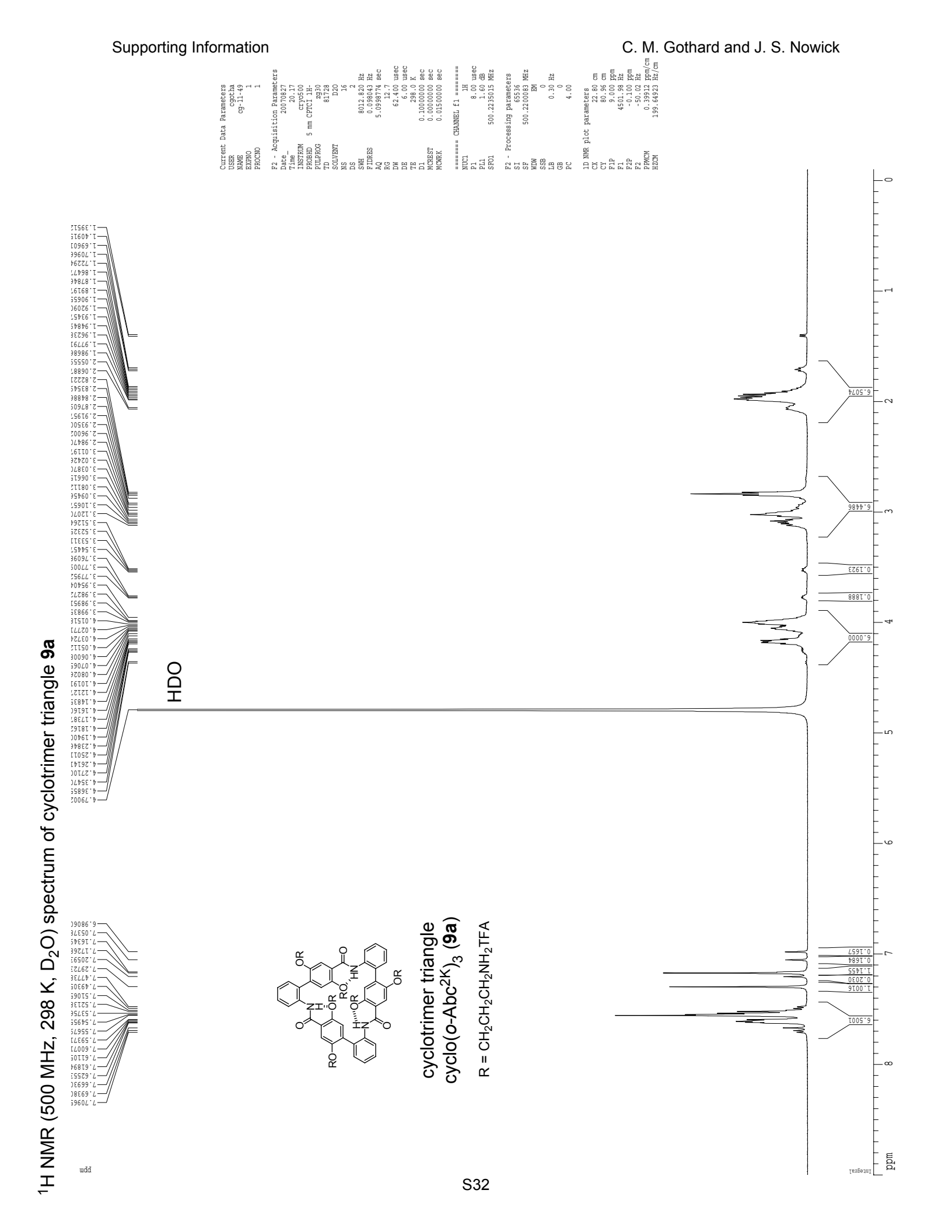
(a) Analytical RP-HPLC (0-30% acetonitrile with 0.1% TFA over 10 min, λ = 214)



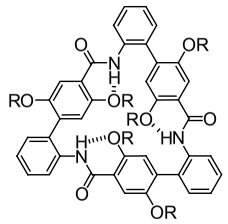
(b) ESI Mass spectrum. (Calcd exact mass for $C_{57}H_{69}N_9O_9$ [M] = 1023.52)



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



^{1}H NMR (500 MHz, 298 K, D_2O) spectrum of cyclotrimer triangle **9a**: Aromatic Region



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

R = $CH_2CH_2CH_2NH_2TFA$

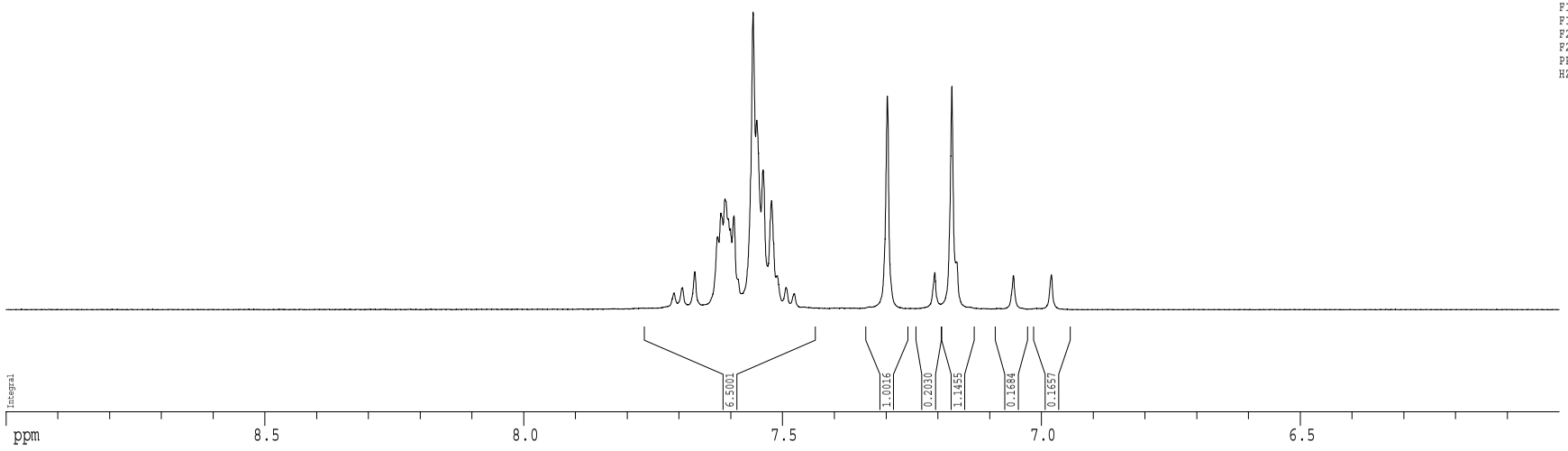

```
Current Data Parameters
USER                cgotha
NAME              cg-11-49
EXPNO                    1
PROCNO                   1

F2 - Acquisition Parameters
Date_            20070827
Time                20.17
INSTRUM          cryo500
PROBHD    5 mm CPTCI 1H-
PULPROG              zg30
TD                  81728
SOLVENT               D2O
NS                     16
DS                      2
SWH              8012.820 Hz
FIDRES           0.098043 Hz
AQ               5.0998774 sec
RG                   12.7
DW                 62.400 usec
DE                  6.00 usec
TE                298.0 K
D1             0.10000000 sec
MCREST         0.00000000 sec
MCWRK          0.01500000 sec

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

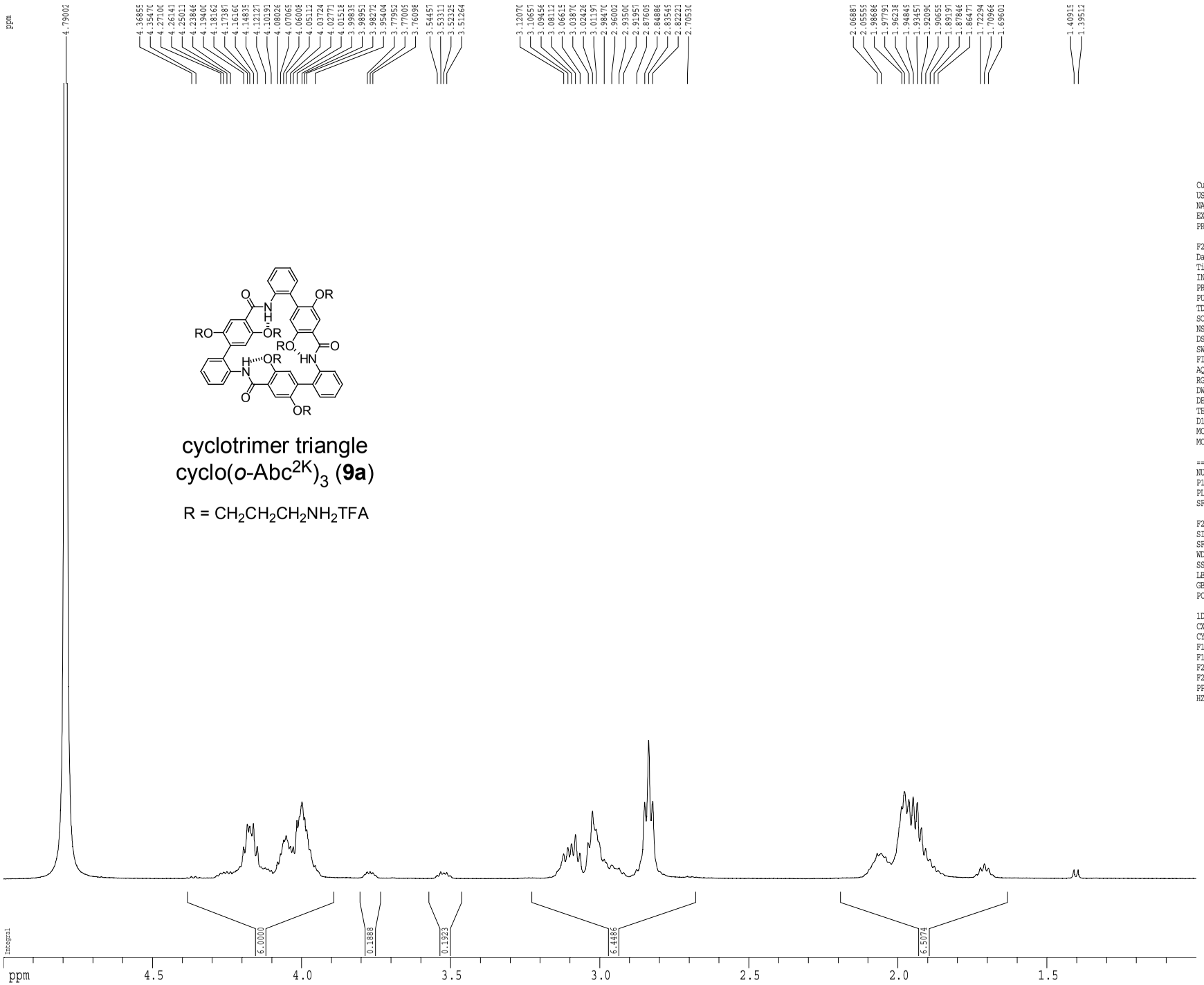
F2 - Processing parameters
SI                    65536
SF           500.2200083 MHz
WDW                     EM
SSB                      0
LB                  0.30 Hz
GB                       0
PC                    4.00

1D NMR plot parameters
CX                  22.80 cm
CY                  80.96 cm
F1P                 9.000 ppm
F1                4501.98 Hz
F2P                 6.000 ppm
F2                3001.32 Hz
PPMCM             0.13158 ppm/cm
HZCM             65.81842 Hz/cm
```



C. M. Gothard and J. S. Nowick

^{1}H NMR (500 MHz, 298 K, D_2O) spectrum of cyclotrimer triangle **9a**: Aliphatic Region



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

R = $CH_2CH_2CH_2NH_2TFA$

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

F2 - Acquisition Parameters
Date_ 20070827
Time 20.17
INSTRUM cryo500
PROBHD 5 mm CPTCI 1H-
PULPROG zg30
TD 81728
SOLVENT D2O
NS 16
DS 2
SWH 8012.820 Hz
FIDRES 0.098043 Hz
AQ 5.0998774 sec
RG 12.7
DW 62.400 usec
DE 6.00 usec
TE 298.0 K
D1 0.10000000 sec
MCREST 0.00000000 sec
MCWRK 0.01500000 sec

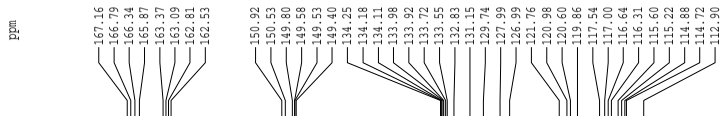
======== CHANNEL f1 ========
NUC1 1H
P1 8.00 usec
PL1 1.60 dB
SFO1 500.2235015 MHz

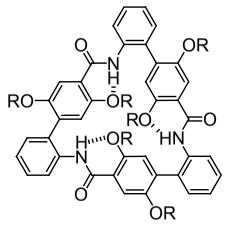
F2 - Processing parameters
SI 65536
SF 500.2200083 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 4.00

1D NMR plot parameters
CX 22.80 cm
CY 80.96 cm
F1P 5.000 ppm
F1 2501.10 Hz
F2P 1.000 ppm
F2 500.22 Hz
PPMCM 0.17544 ppm/cm
HZCM 87.75790 Hz/cm

C. M. Gothard and J. S. Nowick

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





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

R = $CH_2CH_2CH_2NH_2TFA$


```
Current Data Parameters
USER                cgotha
NAME             cg-11-49
EXPNO                   4
PROCNO                  1

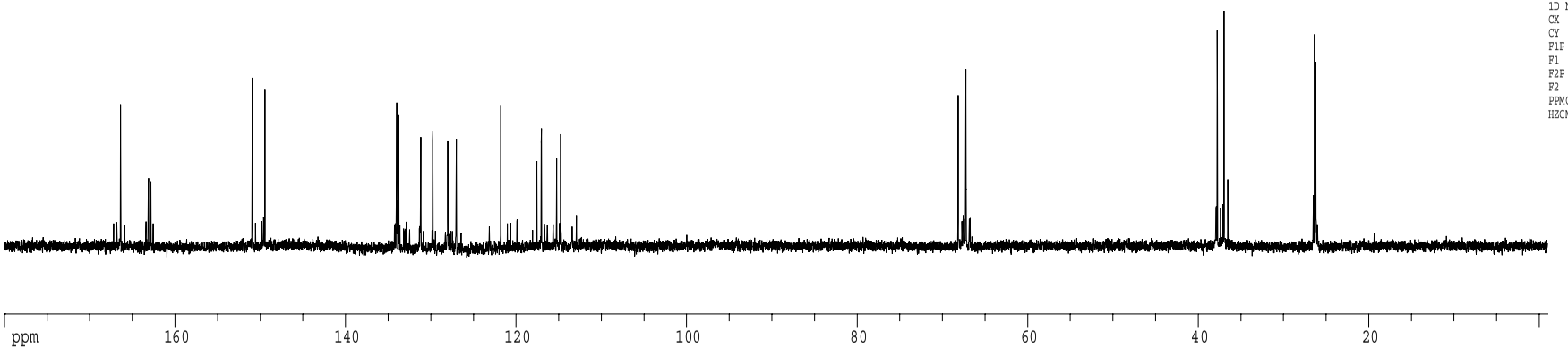
F2 - Acquisition Parameters
Date_            20070827
Time                20.33
INSTRUM           cryo500
PROBHD    5 mm CPTCI 1H-
PULPROG           zgdc30
TD                 65418
SOLVENT              D2O
NS                  2958
DS                     4
SWH            30303.031 Hz
FIDRES          0.463222 Hz
AQ              1.0794470 sec
RG                18390.4
DW                 16.500 usec
DE                  6.00 usec
TE                298.0 K
D1             0.25000000 sec
d11            0.03000000 sec
MCREST         0.00000000 sec
MCWRK          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

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                   3.49 cm
F1P                180.000 ppm
F1               22640.47 Hz
F2P                -1.000 ppm
F2               -125.78 Hz
PPMCM             7.93860 ppm/cm
HZCM            998.52002 Hz/cm
```

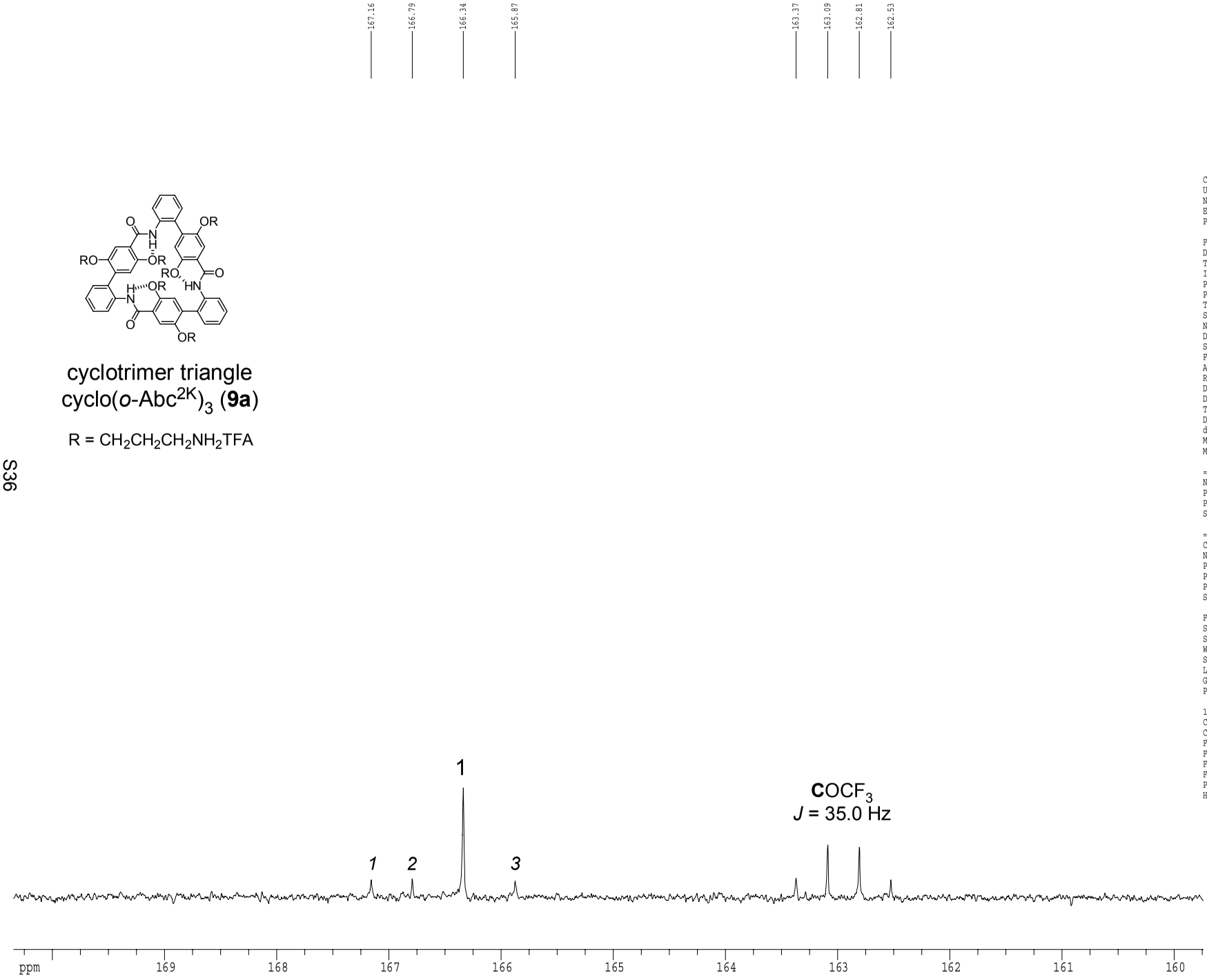


C. M. Gothard and J. S. Nowick

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

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

R = $CH_2CH_2CH_2NH_2TFA$




```
Current Data Parameters
USER                cgotha
NAME             cg-11-49
EXPNO                   4
PROCNO                  1

F2 - Acquisition Parameters
Date_            20070827
Time                20.33
INSTRUM           cryo500
PROBHD   5 mm CPTCI 1H-
PULPROG             zgdc30
TD                  65418
SOLVENT               D2O
NS                   2958
DS                      4
SWH             30303.031 Hz
FIDRES           0.463222 Hz
AQ               1.0794470 sec
RG                 18390.4
DW                 16.500 usec
DE                  6.00 usec
TE                 298.0 K
D1              0.25000000 sec
d11             0.03000000 sec
MCREST          0.00000000 sec
MCWRK           0.01500000 sec

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

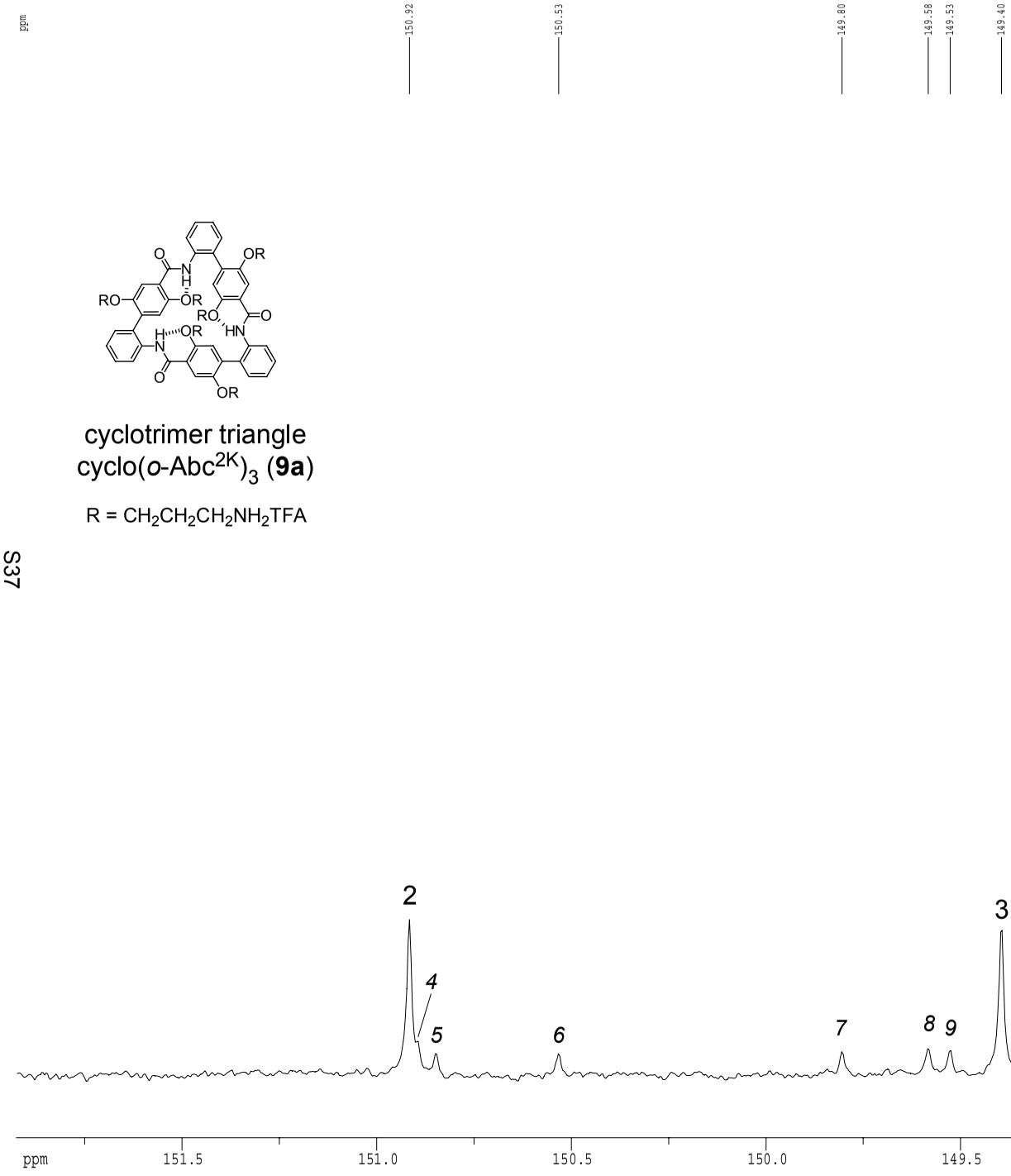
======== CHANNEL f2 ========
CPDPRG2           waltz16
NUC2                   1H
PCPD2              100.00 usec
PL2                 1.60 dB
PL12                23.54 dB
SFO2          500.2225011 MHz

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                   3.49 cm
F1P               170.344 ppm
F1               21425.96 Hz
F2P               159.742 ppm
F2               20092.37 Hz
PPMCM             0.46502 ppm/cm
HZCM             58.49070 Hz/cm
```


C. M. Gothard and J. S. Nowick

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



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

R = $CH_2CH_2CH_2NH_2TFA$

Current Data Parameters
USER cgotha
NAME cg-11-49
EXPNO 4
PROCNO 1

F2 - Acquisition Parameters
Date_ 20070827
Time 20.33
INSTRUM cryo500
PROBHD 5 mm CPTCI 1H-
PULPROG zgdc30
TD 65418
SOLVENT D2O
NS 2958
DS 4
SWH 30303.031 Hz
FIDRES 0.463222 Hz
AQ 1.0794470 sec
RG 18390.4
DW 16.500 usec
DE 6.00 usec
TE 298.0 K
D1 0.25000000 sec
d11 0.03000000 sec
MCREST 0.00000000 sec
MCWRK 0.01500000 sec

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

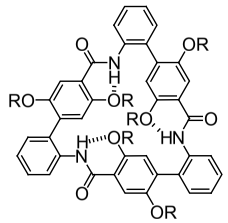
======== CHANNEL f2 ========
CPDPRG2 waltz16
NUC2 1H
PCPD2 100.00 usec
PL2 1.60 dB
PL12 23.54 dB
SFO2 500.2225011 MHz

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 3.49 cm
F1P 151.925 ppm
F1 19109.23 Hz
F2P 148.266 ppm
F2 18648.94 Hz
PPMCM 0.16051 ppm/cm
HZCM 20.18848 Hz/cm

C. M. Gothard and J. S. Nowick

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



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

R = $CH_2CH_2CH_2NH_2TFA$


```
Current Data Parameters
USER                cgotha
NAME             cg-11-49
EXPNO                   4
PROCNO                  1

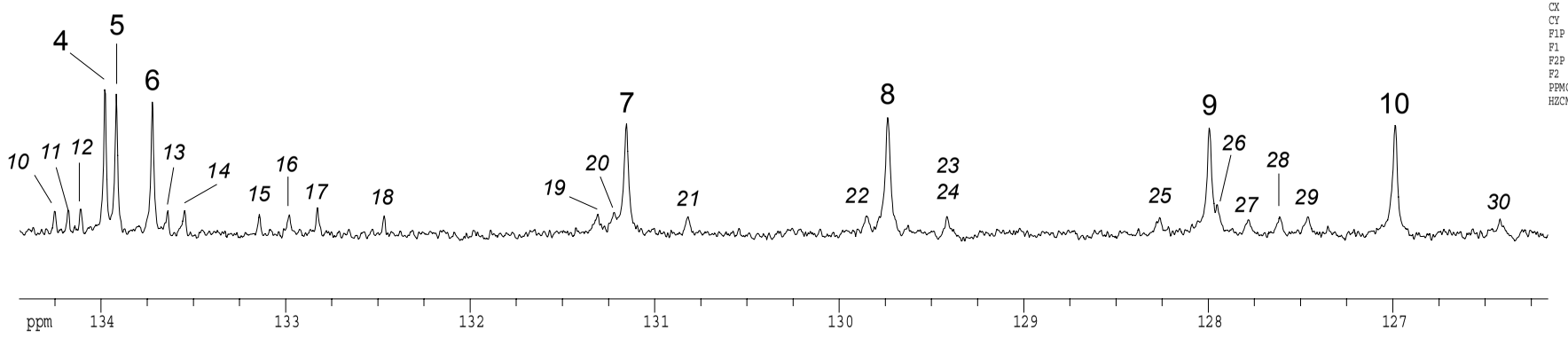
F2 - Acquisition Parameters
Date_            20070827
Time                20.33
INSTRUM           cryo500
PROBHD    5 mm CPTCI 1H-
PULPROG           zgdc30
TD                 65418
SOLVENT              D2O
NS                  2958
DS                     4
SWH            30303.031 Hz
FIDRES          0.463222 Hz
AQ              1.0794470 sec
RG                18390.4
DW                 16.500 usec
DE                  6.00 usec
TE                298.0 K
D1             0.25000000 sec
d11            0.03000000 sec
MCREST         0.00000000 sec
MCWRK          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

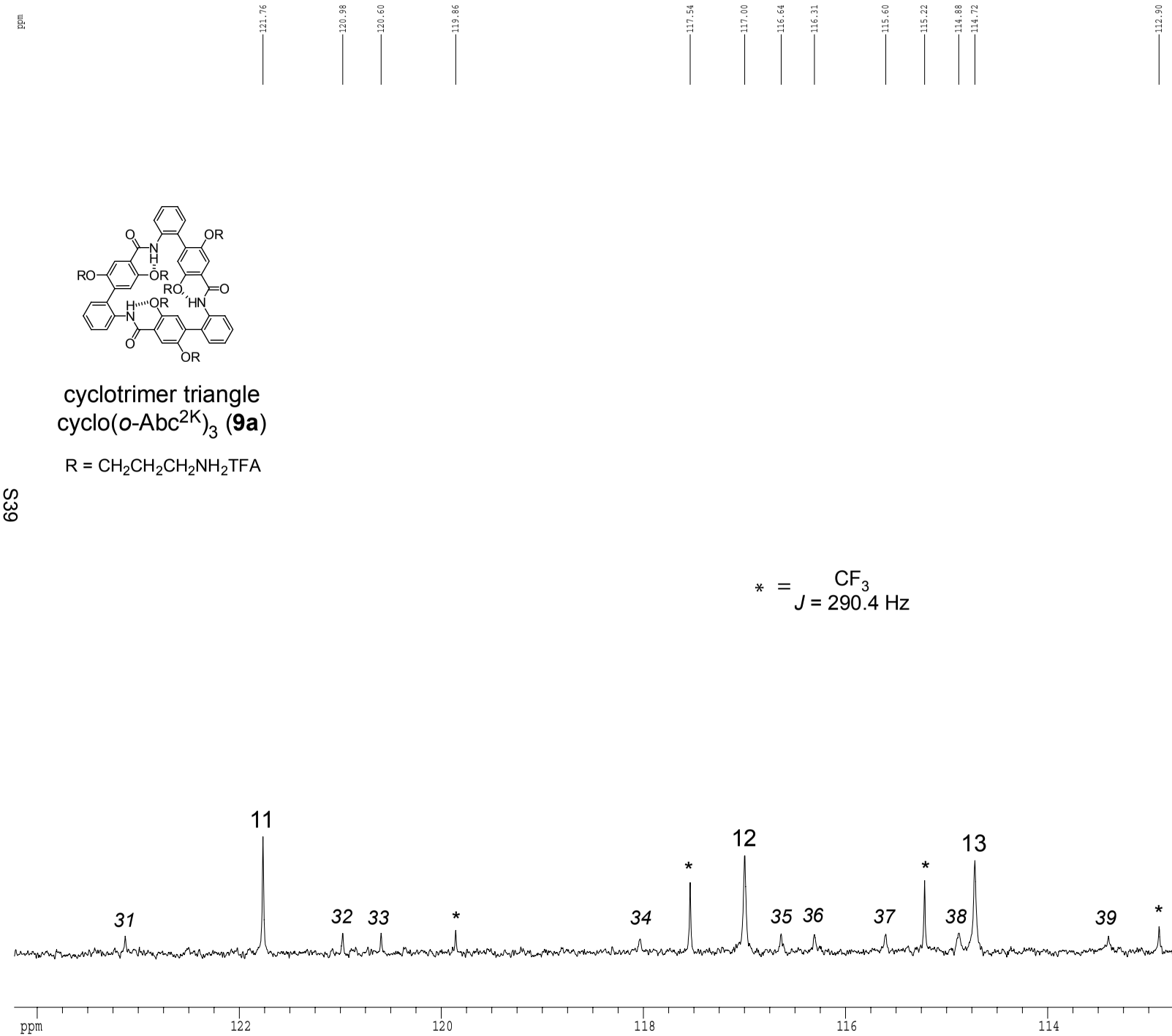
F2 - Processing parameters
SI                 65536
SF           125.7804190 MHz
WDW                   EM
SSB                    0
LB                  1.00 Hz
GB                     0
PC                  2.00

1D NMR plot parameters
CX                 22.80 cm
CY                  3.49 cm
F1P               134.443 ppm
F1              16910.26 Hz
F2P               126.160 ppm
F2              15868.42 Hz
PPMCM           0.36329 ppm/cm
HZCM            45.69448 Hz/cm
```



C. M. Gothard and J. S. Nowick

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



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

R = $CH_2CH_2CH_2NH_2TFA$

* = CF_3
J = 290.4 Hz

Current Data Parameters
USER cgotha
NAME cg-11-49
EXPNO 4
PROCNO 1

F2 - Acquisition Parameters
Date_ 20070827
Time 20.33
INSTRUM cryo500
PROBHD 5 mm CPTCI 1H-
PULPROG zgdc30
TD 65418
SOLVENT D2O
NS 2958
DS 4
SWH 30303.031 Hz
FIDRES 0.463222 Hz
AQ 1.0794470 sec
RG 18390.4
DW 16.500 usec
DE 6.00 usec
TE 298.0 K
D1 0.25000000 sec
d11 0.03000000 sec
MCREST 0.00000000 sec
MCWRK 0.01500000 sec

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

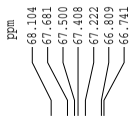
======== CHANNEL f2 ========
CPDPRG2 waltz16
NUC2 1H
PCPD2 100.00 usec
PL2 1.60 dB
PL12 23.54 dB
SFO2 500.2225011 MHz

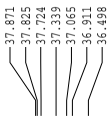
F2 - Processing parameters
SI 65536
SF 125.7804190 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 2.00

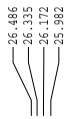
1D NMR plot parameters
CX 22.80 cm
CY 3.49 cm
F1P 124.223 ppm
F1 15624.85 Hz
F2P 111.754 ppm
F2 14056.41 Hz
PPMCM 0.54692 ppm/cm
HZCM 68.79126 Hz/cm

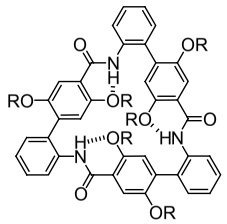
C. M. Gothard and J. S. Nowick

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









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

R = $CH_2CH_2CH_2NH_2TFA$

C. M. Gothard and J. S. Nowick

Current Data Parameters
USER cgotha
NAME cg-11-49
EXPNO 4
PROCNO 1

F2 - Acquisition Parameters
Date_ 20070827
Time 20.33
INSTRUM cryo500
PROBHD 5 mm CPTCI 1H-
PULPROG zgdc30
TD 65418
SOLVENT D2O
NS 2958
DS 4
SWH 30303.031 Hz
FIDRES 0.463222 Hz
AQ 1.0794470 sec
RG 18390.4
DW 16.500 usec
DE 6.00 usec
TE 298.0 K
D1 0.25000000 sec
d11 0.03000000 sec
MCREST 0.00000000 sec
MCWRK 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

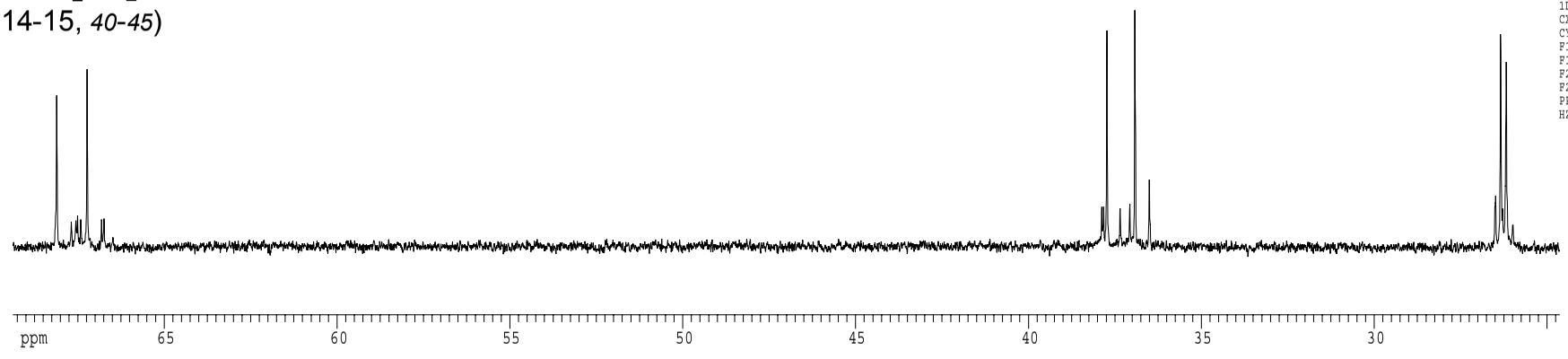
F2 - Processing parameters
SI 65536
SF 125.7804190 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 2.00

1D NMR plot parameters
CX 22.80 cm
CY 3.49 cm
F1P 69.365 ppm
F1 8724.72 Hz
F2P 24.645 ppm
F2 3099.90 Hz
PPMCM 1.96138 ppm/cm
HZCM 246.70276 Hz/cm

$-O\mathbf{C}H_2CH_2-$
(14-15, *40-45*)

$-\mathbf{C}H_2NH_3^+$
(16-17, *46-51*)

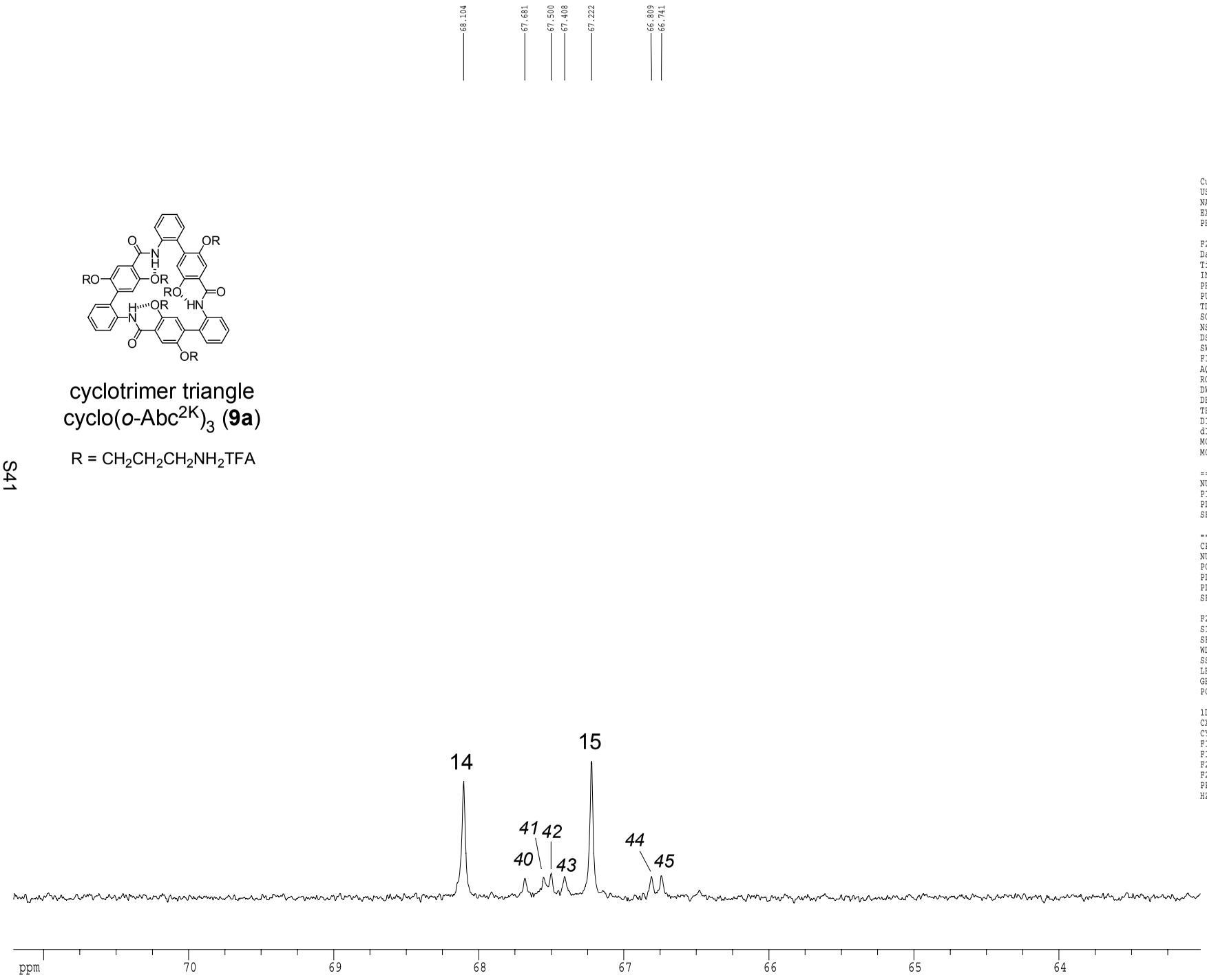
$-CH_2\mathbf{C}H_2CH_2-$
(18-19, *52-57*)



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

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

R = $CH_2CH_2CH_2NH_2TFA$



Current Data Parameters
USER cgotha
NAME cg-11-49
EXPNO 4
PROCNO 1

F2 - Acquisition Parameters
Date_ 20070827
Time 20.33
INSTRUM cryo500
PROBHD 5 mm CPTCI 1H-
PULPROG zgdc30
TD 65418
SOLVENT D2O
NS 2958
DS 4
SWH 30303.031 Hz
FIDRES 0.463222 Hz
AQ 1.0794470 sec
RG 18390.4
DW 16.500 usec
DE 6.00 usec
TE 298.0 K
D1 0.25000000 sec
d11 0.03000000 sec
MCREST 0.00000000 sec
MCWRK 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

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 3.49 cm
F1P 71.206 ppm
F1 8956.36 Hz
F2P 63.026 ppm
F2 7927.41 Hz
PPMCM 0.35880 ppm/cm
HZCM 45.12946 Hz/cm

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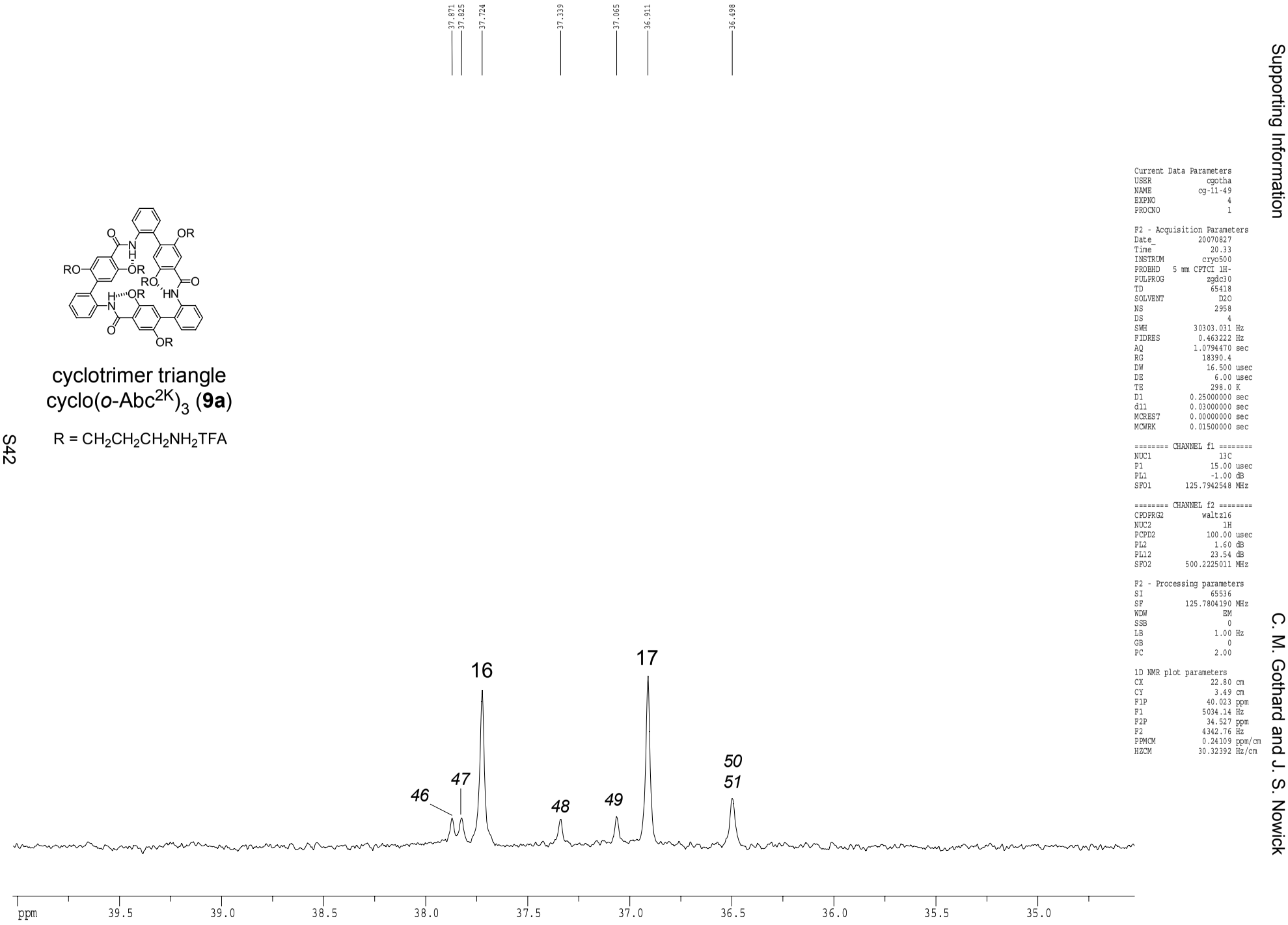
^{13}C NMR (125 MHz, 298 K, D_2O) spectrum of cyclotrimer triangle **9a**

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

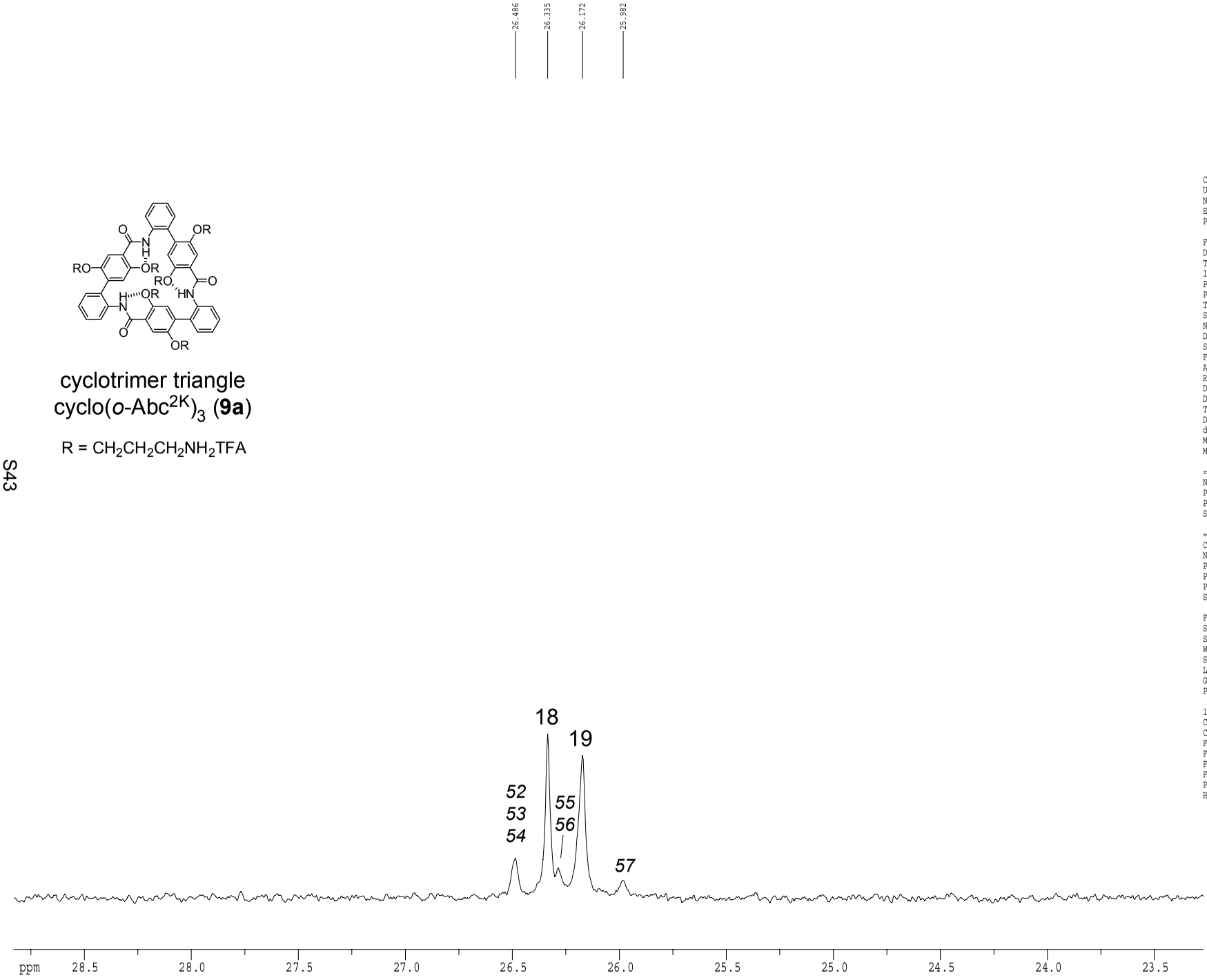
R = $CH_2CH_2CH_2NH_2TFA$



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

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

R = $CH_2CH_2CH_2NH_2TFA$

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Current Data Parameters
USER                cgotha
NAME             cg-11-49
EXPNO                   4
PROCNO                  1

F2 - Acquisition Parameters
Date_            20070827
Time                20.33
INSTRUM           cryo500
PROBHD   5 mm CPTCI 1H-
PULPROG           zgdc30
TD                  65418
SOLVENT               D2O
NS                   2958
DS                      4
SWH              30303.031 Hz
FIDRES           0.463222 Hz
AQ               1.0794470 sec
RG                 18390.4
DW                 16.500 usec
DE                  6.00 usec
TE                 298.0 K
D1              0.25000000 sec
d11             0.03000000 sec
MCREST          0.00000000 sec
MCWRK           0.01500000 sec

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

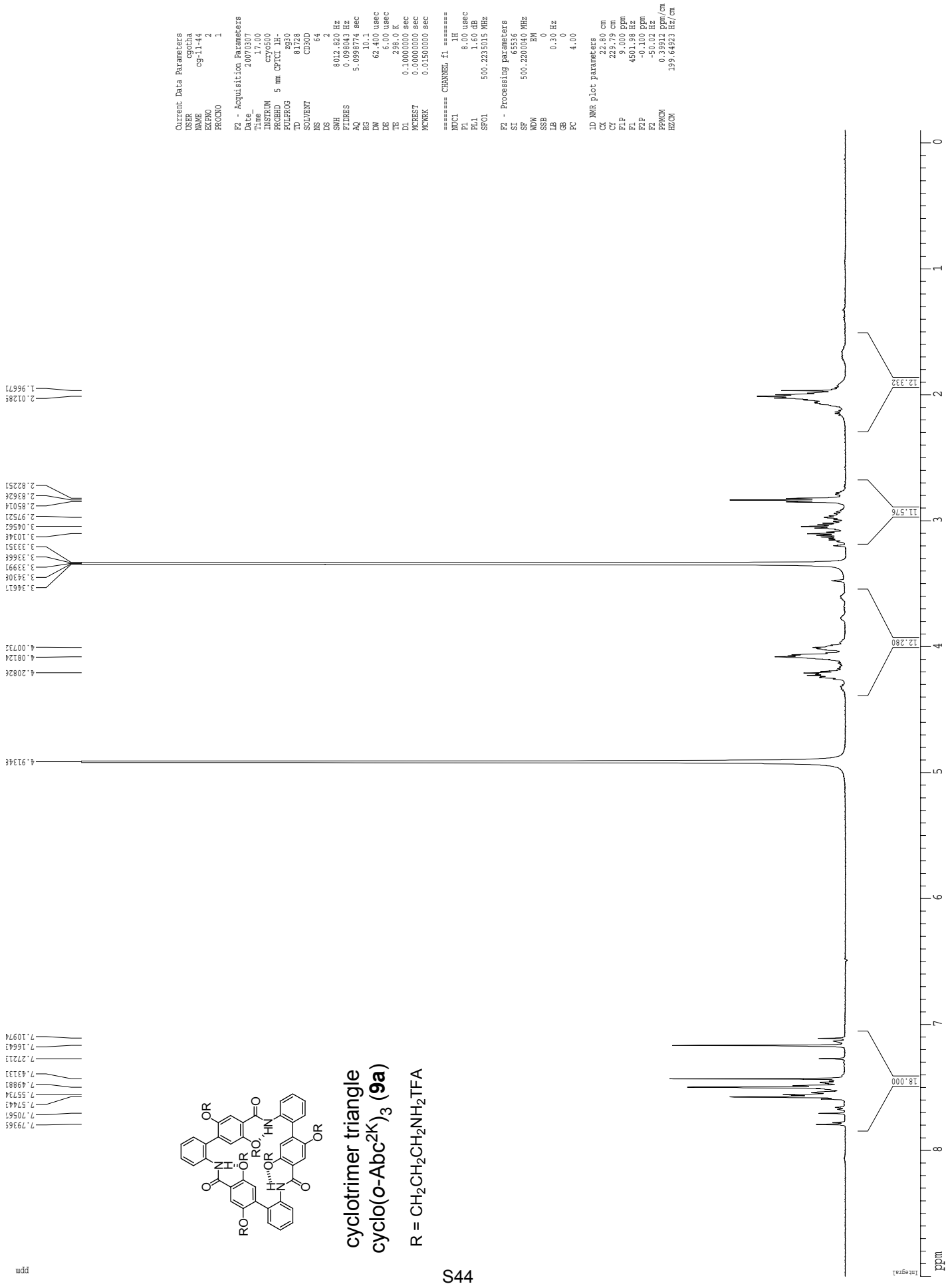
======== CHANNEL f2 ========
CPDPRG2           waltz16
NUC2                  1H
PCPD2             100.00 usec
PL2                 1.60 dB
PL12                23.54 dB
SFO2          500.2225011 MHz

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                  3.49 cm
F1P               28.830 ppm
F1               3626.21 Hz
F2P               23.272 ppm
F2               2927.18 Hz
PPMCM            0.24375 ppm/cm
HZCM            30.65948 Hz/cm
```


C. M. Gothard and J. S. Nowick

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



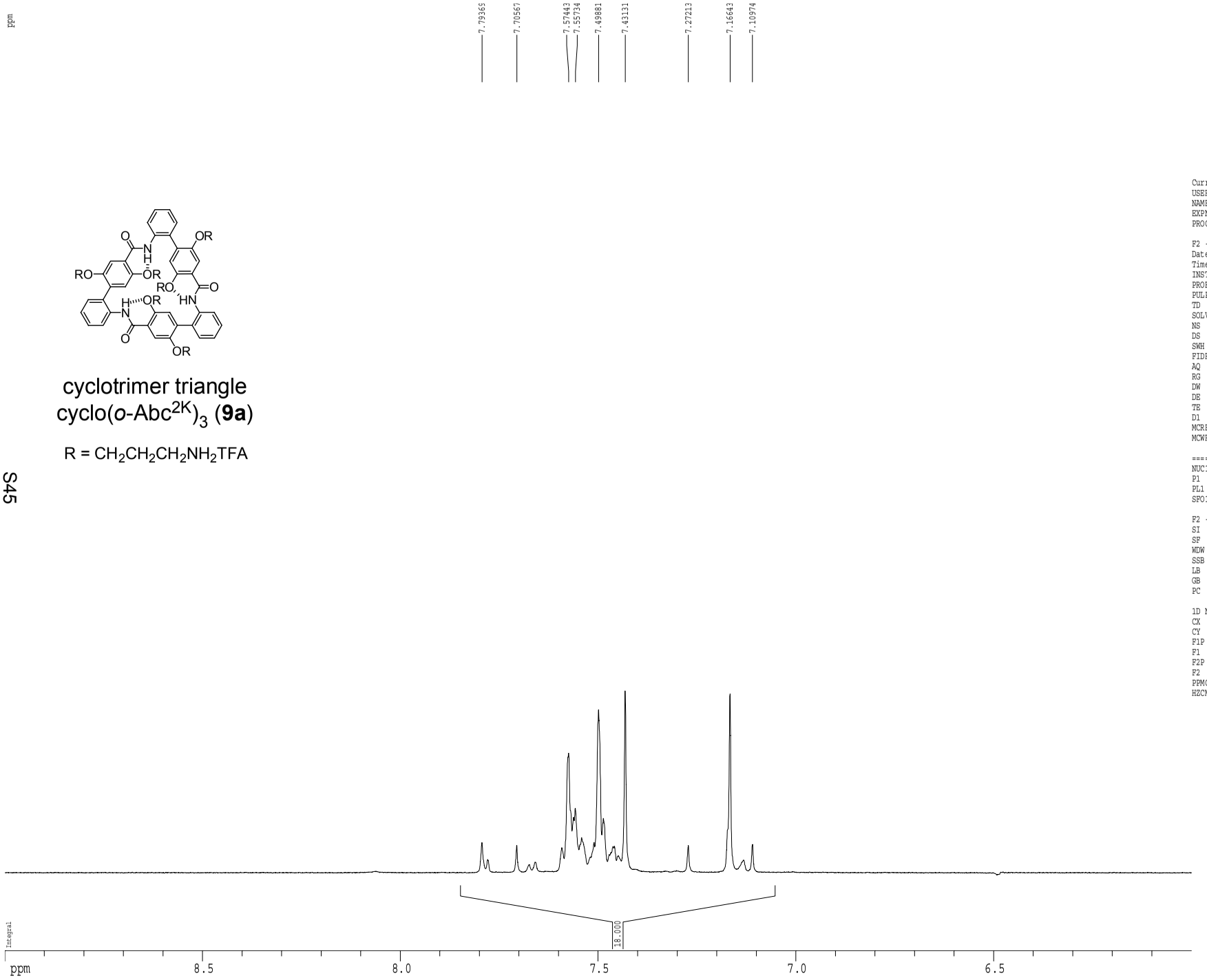
cyclotrimer triangle
cyclo(*o*-Abc^{2K})$_3$ (**9a**)

R = $CH_2CH_2CH_2NH_2$TFA

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

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

R = $CH_2CH_2CH_2NH_2TFA$

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Current Data Parameters
USER                cgotha
NAME              cg-11-44
EXPNO                    2
PROCNO                   1

F2 - Acquisition Parameters
Date_            20070307
Time                17.00
INSTRUM          cryo500
PROBHD   5 mm CPTCI 1H-
PULPROG              zg30
TD                  81728
SOLVENT             CD3OD
NS                     64
DS                      2
SWH              8012.820 Hz
FIDRES           0.098043 Hz
AQ               5.0998774 sec
RG                   10.1
DW                 62.400 usec
DE                  6.00 usec
TE                 298.0 K
D1              0.10000000 sec
MCREST          0.00000000 sec
MCWRK           0.01500000 sec

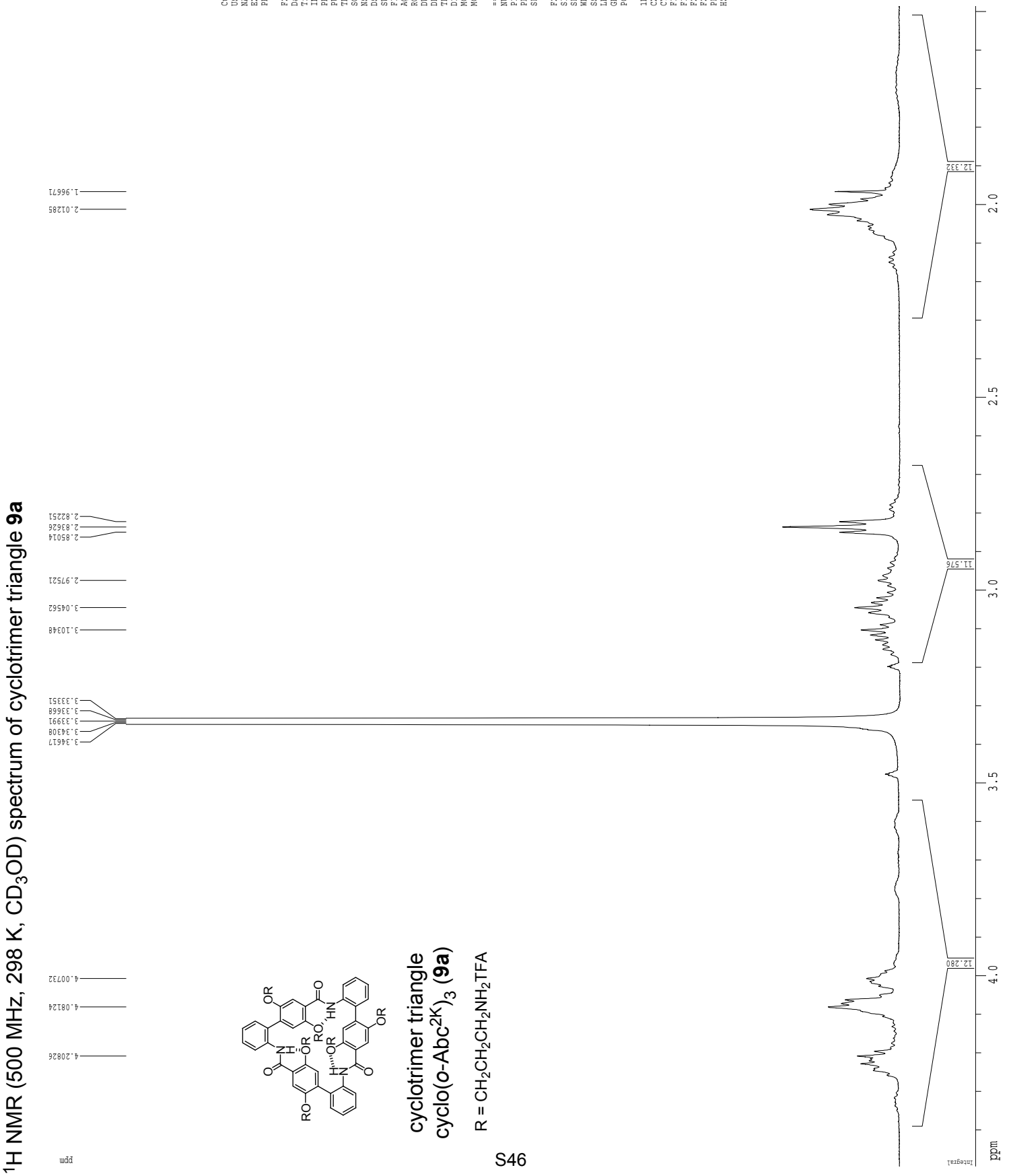
======== CHANNEL f1 ========
NUC1                   1H
P1                  8.00 usec
PL1                 1.60 dB
SFO1          500.2235015 MHz

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                 229.79 cm
F1P                 9.000 ppm
F1                4501.98 Hz
F2P                 6.000 ppm
F2                3001.32 Hz
PPMCM             0.13158 ppm/cm
HZCM             65.81842 Hz/cm
```


C. M. Gothard and J. S. Nowick

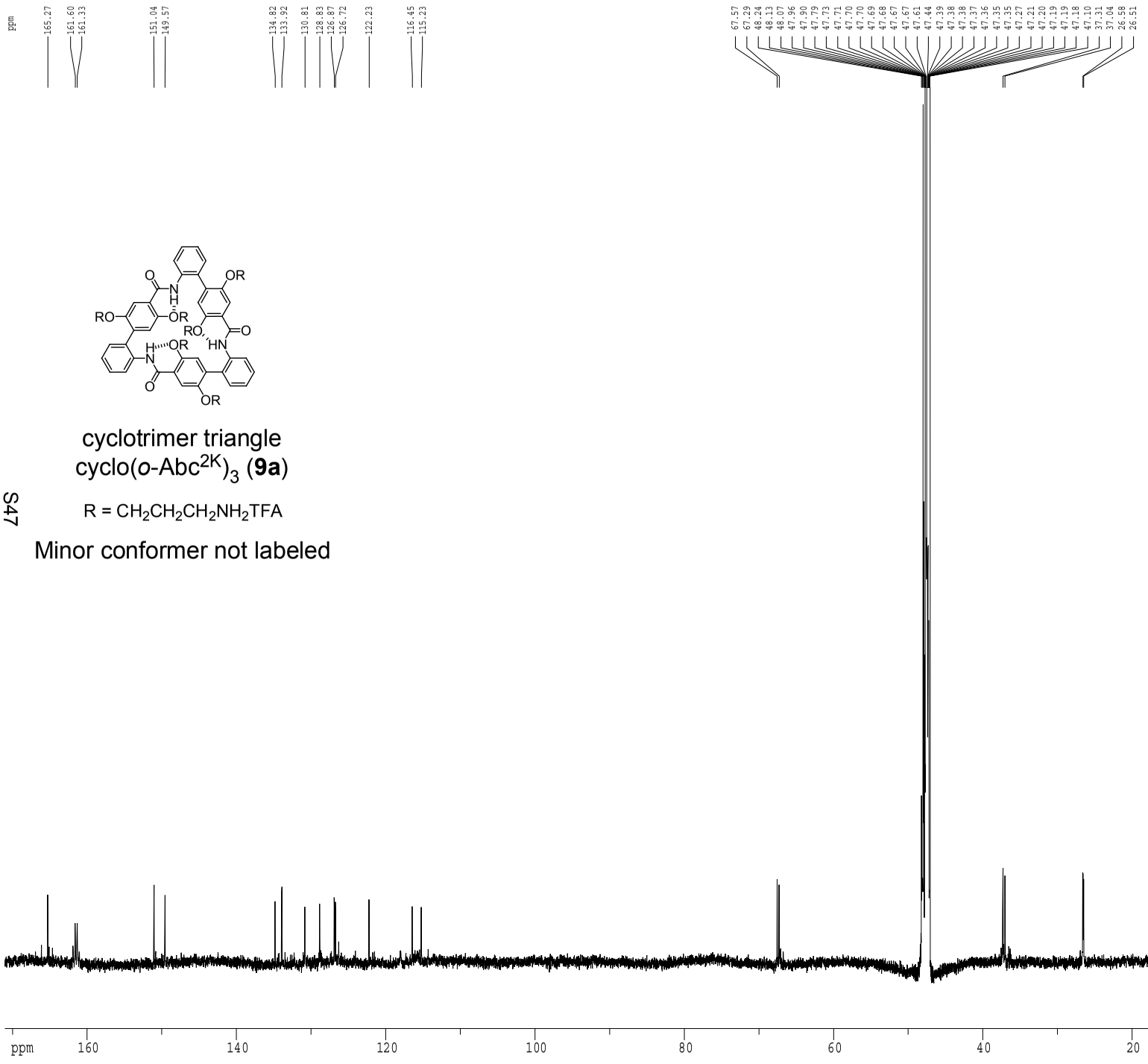
^{1}H NMR (500 MHz, 298 K, CD_3OD) spectrum of cyclotrimer triangle **9a**



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

R = $CH_2CH_2CH_2NH_2$TFA

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



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

R = $CH_2CH_2CH_2NH_2TFA$

Minor conformer not labeled

Current Data Parameters
USER cgotha
NAME cg-11-44_C13
EXPNO 2
PROCNO 1

F2 - Acquisition Parameters
Date_ 20070309
Time 0.36
INSTRUM cryo500
PROBHD 5 mm CPTCI 1H-
PULPROG zgdc30
TD 65418
SOLVENT CD3OD
NS 19000
DS 4
SWH 30303.031 Hz
FIDRES 0.463222 Hz
AQ 1.0794470 sec
RG 8192
DW 16.500 usec
DE 6.00 usec
TE 298.0 K
D1 0.25000000 sec
d11 0.03000000 sec
MCREST 0.00000000 sec
MCWRK 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

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 737.01 cm
F1P 170.986 ppm
F1 21506.70 Hz
F2P -1.136 ppm
F2 -142.85 Hz
PPMCM 7.54920 ppm/cm
HZCM 949.54175 Hz/cm

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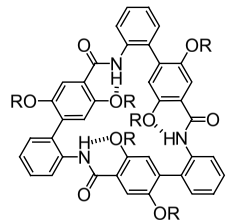
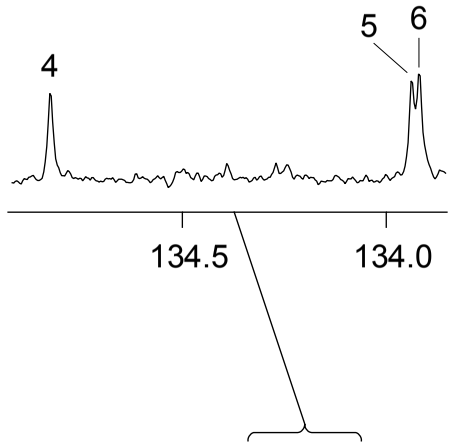
^{13}C NMR (125 MHz, 298 K, CD_3OD) spectrum of cyclotrimer triangle **9a**



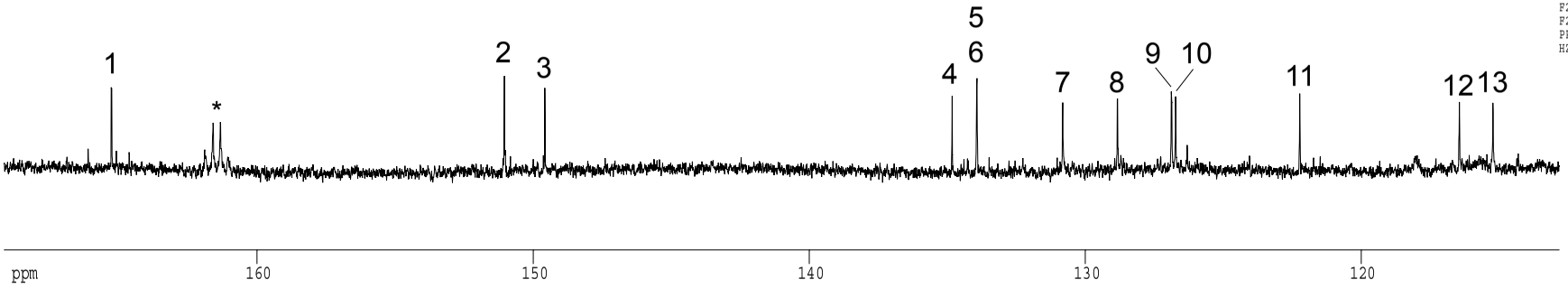
cyclotrimer triangle
cyclo(*o*-Abc^{2K})$_3$ (**9a**)

R = $CH_2CH_2CH_2NH_2TFA$

Minor conformer not labeled



* = $CF_3CO_2^-$

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Current Data Parameters
USER                cgotha
NAME          cg-11-44_C13
EXPNO                    2
PROCNO                   1

F2 - Acquisition Parameters
Date_             20070309
Time                 0.36
INSTRUM           cryo500
PROBHD    5 mm CPTCI 1H-
PULPROG           zgdc30
TD                  65418
SOLVENT             CD3OD
NS                  19000
DS                      4
SWH            30303.031 Hz
FIDRES          0.463222 Hz
AQ              1.0794470 sec
RG                   8192
DW                16.500 usec
DE                 6.00 usec
TE                298.0 K
D1             0.25000000 sec
d11            0.03000000 sec
MCREST         0.00000000 sec
MCWRK          0.01500000 sec

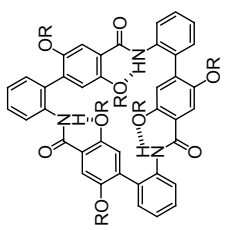
======== CHANNEL f1 ========
NUC1                  13C
P1                 15.00 usec
PL1                -1.00 dB
SFO1          125.7942548 MHz

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

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                  737.01 cm
F1P                169.159 ppm
F1                21276.89 Hz
F2P                112.835 ppm
F2                14192.45 Hz
PPMCM              2.47035 ppm/cm
HZCM             310.72122 Hz/cm
```

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

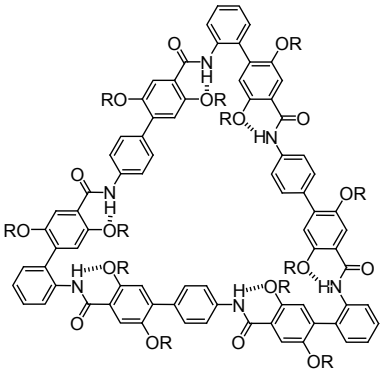


cyclotrimer triangle
cyclo(*o*-Abc^{2K})$_3$ (**9a**)

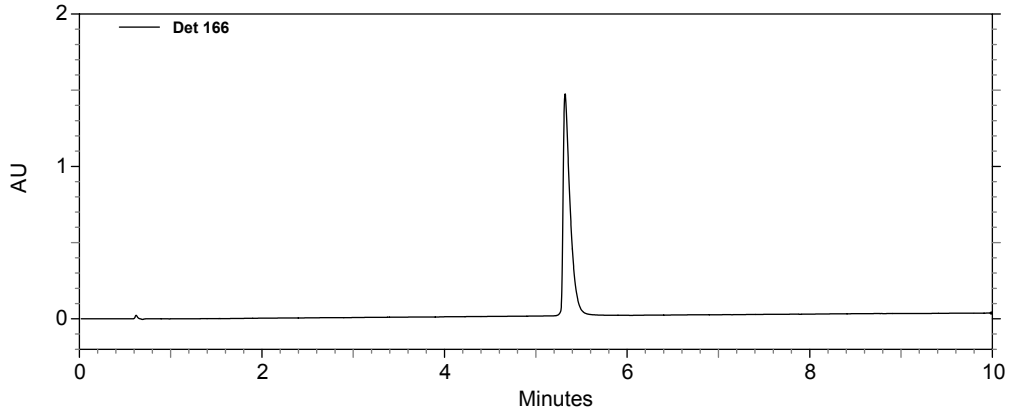
R = $CH_2CH_2CH_2NH_2$TFA

Minor conformer not labeled

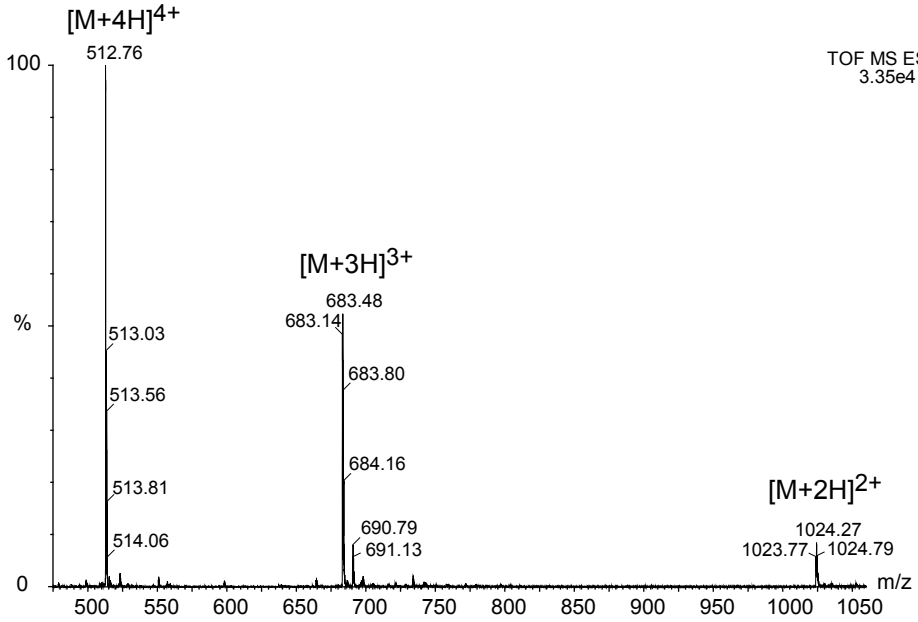
cyclohexamer triangle: (*o*-Abc^{2K}-*p*-Abc^{2K})$_3$ (**9b**)
Analytical RP-HPLC chromatograph and mass spectrum (ESI-MS)



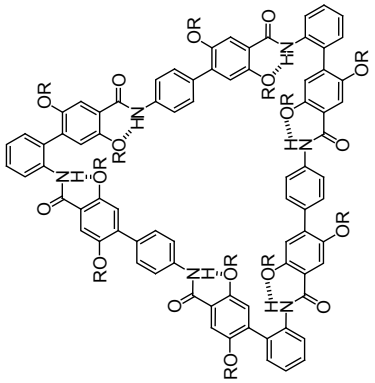
(a) Analytical RP-HPLC (5-50% acetonitrile with 0.1% TFA over 10 min, λ = 214)



(b) ESI Mass spectrum. (Calcd exact mass for $C_{114}H_{138}N_{18}O_{18}$ [M] = 2047.04)



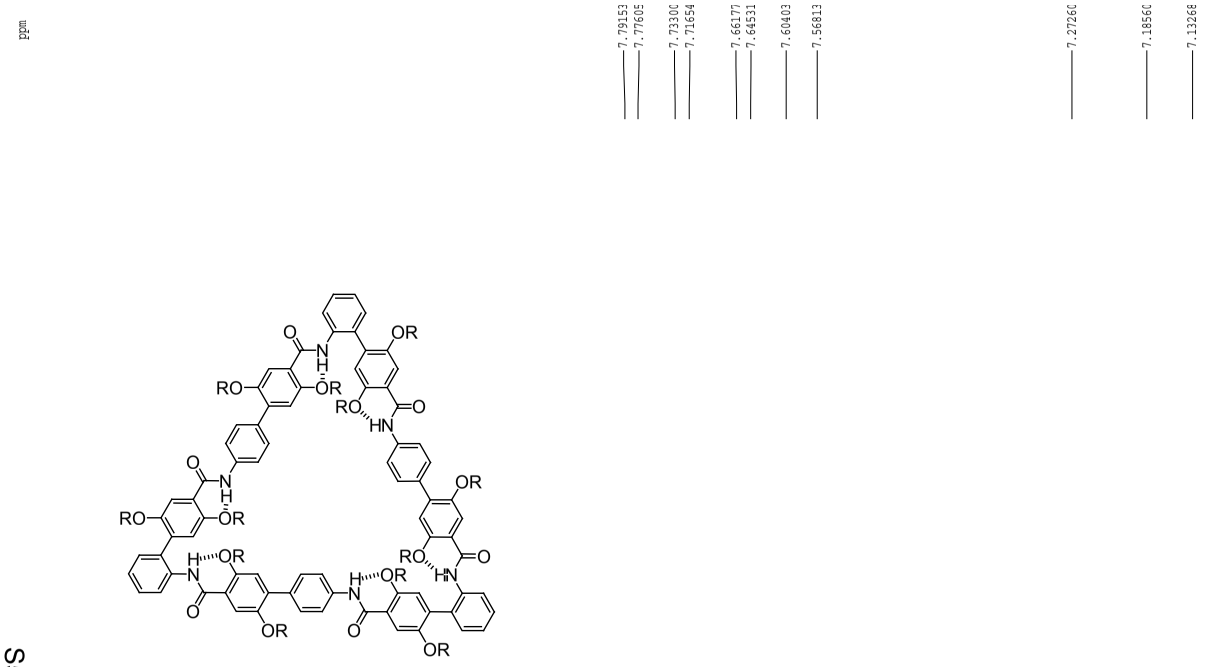
^{1}H NMR (500 MHz, 298 K, D_2O) spectrum of cyclohexamer triangle **9b**



cyclohexamer triangle **9b**
(*o*-Abc^{2K}-*p*-Abc^{2K})$_3$

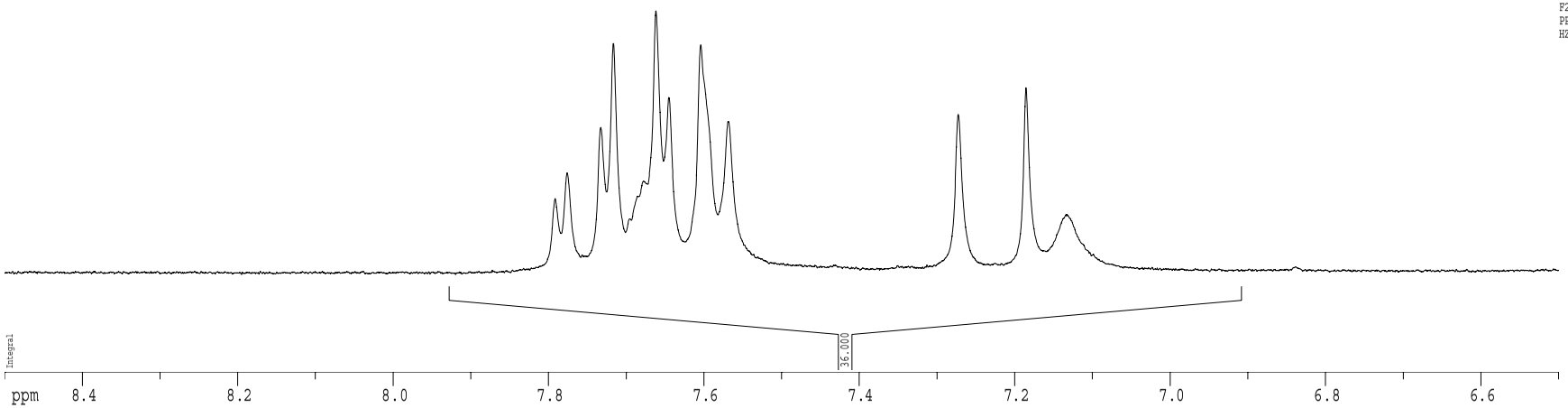
R = $CH_2CH_2CH_2NH_2$TFA

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



cyclohexamer triangle **9b**
(*o*-Abc^{2K}-*p*-Abc^{2K})$_3$

R = $CH_2CH_2CH_2NH_2$TFA

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

F2 - Acquisition Parameters
Date_              20070325
Time                 17.14
INSTRUM              qn500
PROBHD   5 mm broadband
PULPROG               zg30
TD                   81728
SOLVENT                D2O
NS                        64
DS                         2
SWH                8012.820 Hz
FIDRES             0.098043 Hz
AQ                 5.0998774 sec
RG                     812.7
DW                    62.400 usec
DE                     6.00 usec
TE                    298.0 K
D1              0.10000000 sec
MCREST          0.00000000 sec
MCWRK           0.01500000 sec

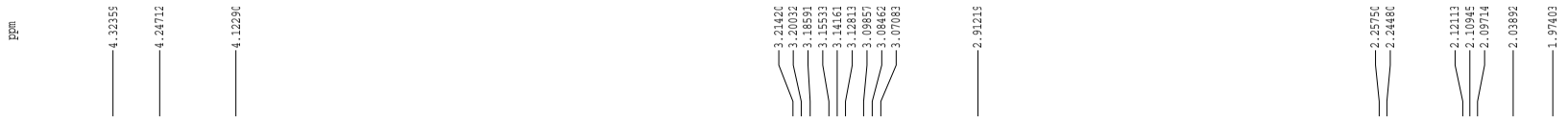
======== CHANNEL f1 ========
NUC1                     1H
P1                    12.00 usec
PL1                   -3.00 dB
SFO1           499.9334995 MHz

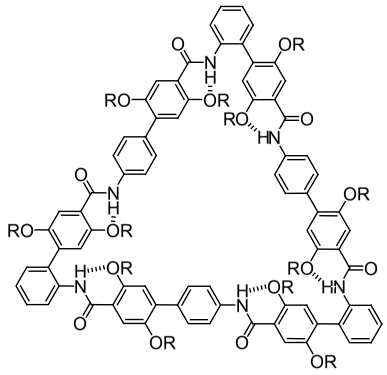
F2 - Processing parameters
SI                       65536
SF             499.9300035 MHz
WDW                      EM
SSB                       0
LB                     0.30 Hz
GB                        0
PC                     4.00

1D NMR plot parameters
CX                    22.80 cm
CY                   119.33 cm
F1P                   8.500 ppm
F1                  4249.40 Hz
F2P                   6.500 ppm
F2                  3249.54 Hz
PPMCM              0.08772 ppm/cm
HZCM              43.85351 Hz/cm
```

C. M. Gothard and J. S. Nowick

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





cyclohexamer triangle **9b**
(*o*-Abc^{2K}-*p*-Abc^{2K})$_3$

R = $CH_2CH_2CH_2NH_2TFA$

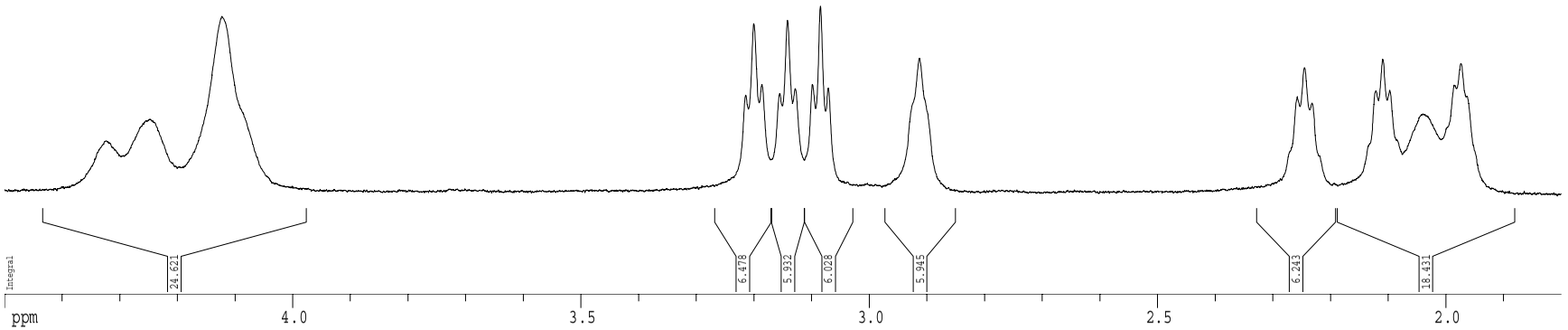
```
Current Data Parameters
USER                cgotha
NAME             cg-11-31
EXPNO                   1
PROCNO                  1

F2 - Acquisition Parameters
Date_            20070325
Time                17.14
INSTRUM             qn500
PROBHD   5 mm broadband
PULPROG              zg30
TD                  81728
SOLVENT               D2O
NS                     64
DS                      2
SWH              8012.820 Hz
FIDRES           0.098043 Hz
AQ               5.0998774 sec
RG                  812.7
DW                62.400 usec
DE                 6.00 usec
TE                298.0 K
D1            0.10000000 sec
MCREST        0.00000000 sec
MCWRK         0.01500000 sec

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

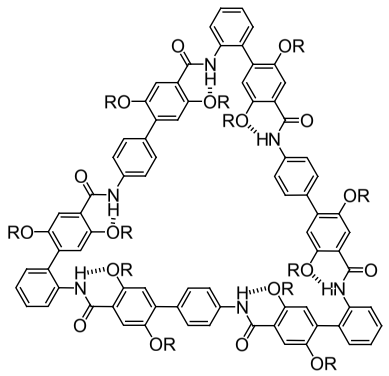
F2 - Processing parameters
SI                  65536
SF           499.9300035 MHz
WDW                    EM
SSB                     0
LB                   0.30 Hz
GB                      0
PC                   4.00

1D NMR plot parameters
CX                  22.80 cm
CY                 119.33 cm
F1P                4.500 ppm
F1                2249.69 Hz
F2P                1.800 ppm
F2                 899.87 Hz
PPMCM             0.11842 ppm/cm
HZCM             59.20224 Hz/cm
```

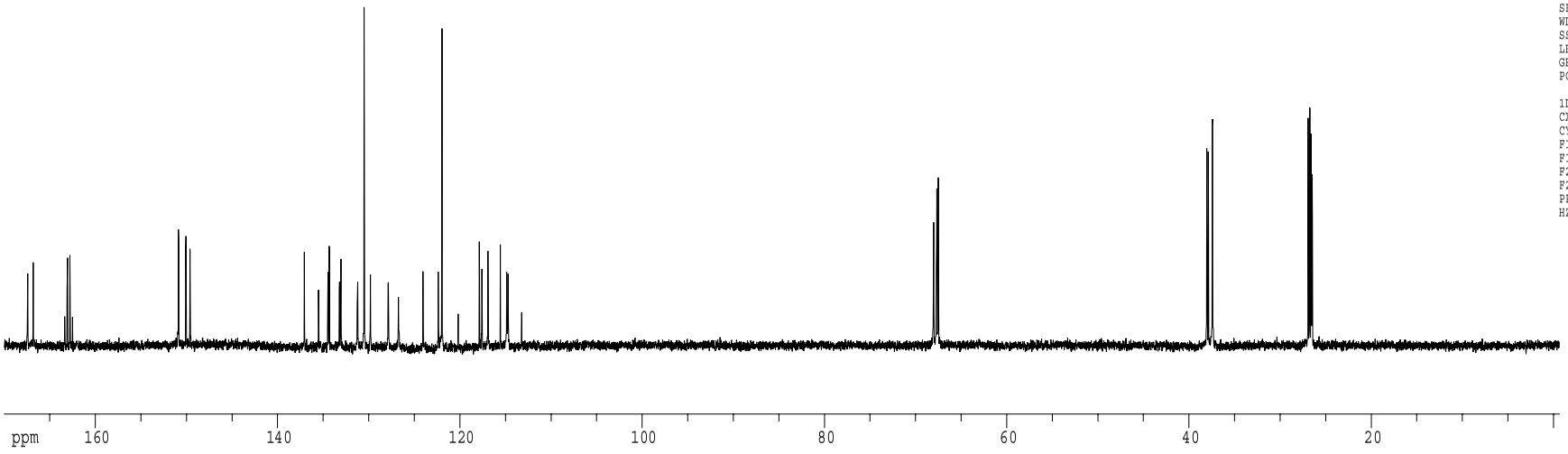


C. M. Gothard and J. S. Nowick

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



cyclohexamer triangle **9b**
(*o*-Abc^{2K}-*p*-Abc^{2K})$_3$
R = $CH_2CH_2CH_2NH_2TFA$

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Current Data Parameters
USER                cgotha
NAME             cg-11-76
EXPNO                   4
PROCNO                  1

F2 - Acquisition Parameters
Date_            20070521
Time                19.20
INSTRUM          cryo500
PROBHD   5 mm CPTCI 1H-
PULPROG          zgdc30
TD                  65418
SOLVENT             D2O
NS                  12190
DS                      4
SWH           30303.031 Hz
FIDRES         0.463222 Hz
AQ             1.0794470 sec
RG               18390.4
DW                16.500 usec
DE                 6.00 usec
TE                320.0 K
D1            0.25000000 sec
d11           0.03000000 sec
MCREST        0.00000000 sec
MCWRK         0.01500000 sec

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

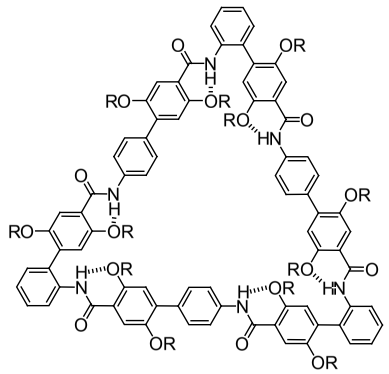
======== CHANNEL f2 ========
CPDPRG2          waltz16
NUC2                1H
PCPD2             100.00 usec
PL2                1.60 dB
PL12              23.54 dB
SFO2        500.2225011 MHz

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                 5.01 cm
F1P             169.978 ppm
F1             21379.91 Hz
F2P              -0.632 ppm
F2               -79.45 Hz
PPMCM           7.48288 ppm/cm
HZCM          941.20026 Hz/cm
```

C. M. Gothard and J. S. Nowick

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



cyclohexamer triangle **9b**
$(o\text{-Abc}^{2K}\text{-}p\text{-Abc}^{2K})_3$
R = $CH_2CH_2CH_2NH_2TFA$


```
Current Data Parameters
USER              cgotha
NAME             cg-11-76
EXPNO                   4
PROCNO                  1

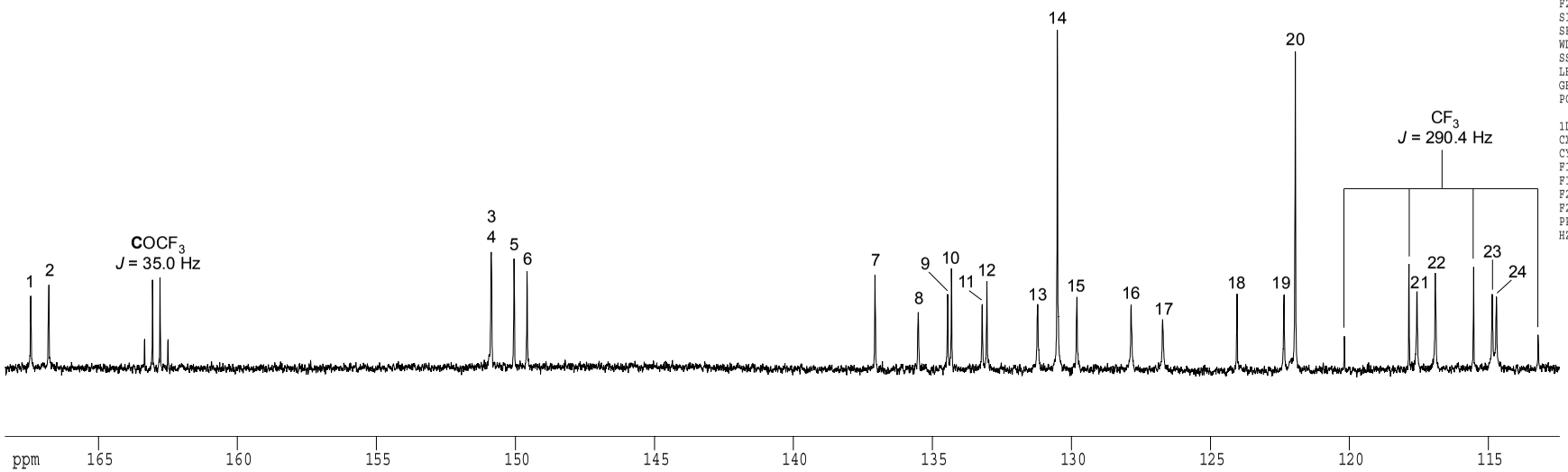
F2 - Acquisition Parameters
Date_            20070521
Time                19.20
INSTRUM          cryo500
PROBHD   5 mm CPTCI 1H-
PULPROG           zgdc30
TD                 65418
SOLVENT              D2O
NS                 12190
DS                     4
SWH           30303.031 Hz
FIDRES         0.463222 Hz
AQ            1.0794470 sec
RG              18390.4
DW               16.500 usec
DE                6.00 usec
TE                320.0 K
D1            0.25000000 sec
d11           0.03000000 sec
MCREST        0.00000000 sec
MCWRK         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

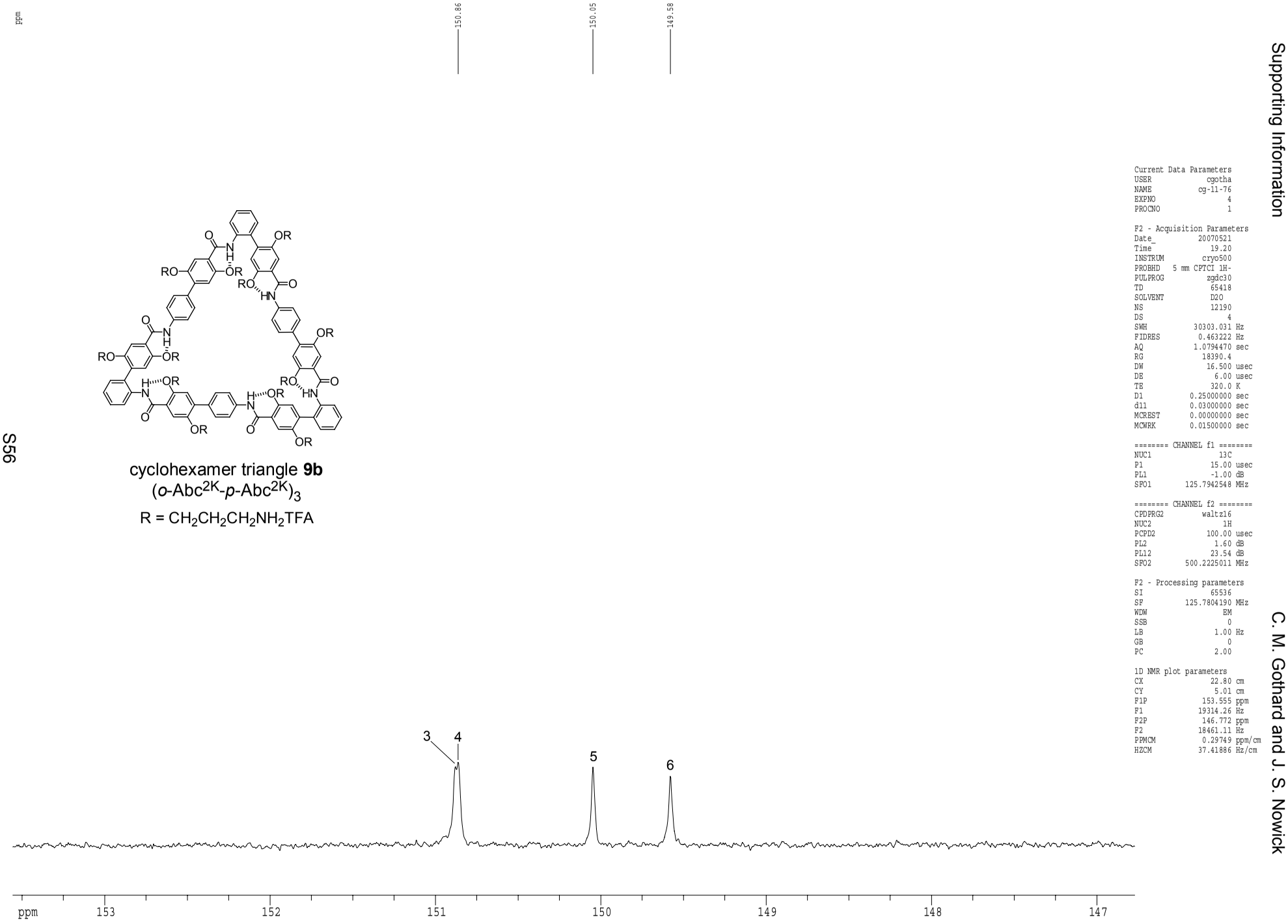
F2 - Processing parameters
SI                 65536
SF          125.7804190 MHz
WDW                   EM
SSB                    0
LB                  1.00 Hz
GB                     0
PC                  2.00

1D NMR plot parameters
CX                 22.80 cm
CY                  5.01 cm
F1P              168.349 ppm
F1             21175.00 Hz
F2P              112.449 ppm
F2             14143.87 Hz
PPMCM            2.45176 ppm/cm
HZCM           308.38278 Hz/cm
```



C. M. Gothard and J. S. Nowick

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



cyclohexamer triangle **9b**
(*o*-Abc^{2K}-*p*-Abc^{2K})$_3$

R = $CH_2CH_2CH_2NH_2TFA$

```
Current Data Parameters
USER                cgotha
NAME             cg-11-76
EXPNO                   4
PROCNO                  1

F2 - Acquisition Parameters
Date_            20070521
Time                19.20
INSTRUM          cryo500
PROBHD   5 mm CPTCI 1H-
PULPROG          zgdc30
TD                  65418
SOLVENT             D2O
NS                  12190
DS                      4
SWH            30303.031 Hz
FIDRES          0.463222 Hz
AQ              1.0794470 sec
RG                18390.4
DW                 16.500 usec
DE                  6.00 usec
TE                 320.0 K
D1             0.25000000 sec
d11            0.03000000 sec
MCREST         0.00000000 sec
MCWRK          0.01500000 sec

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

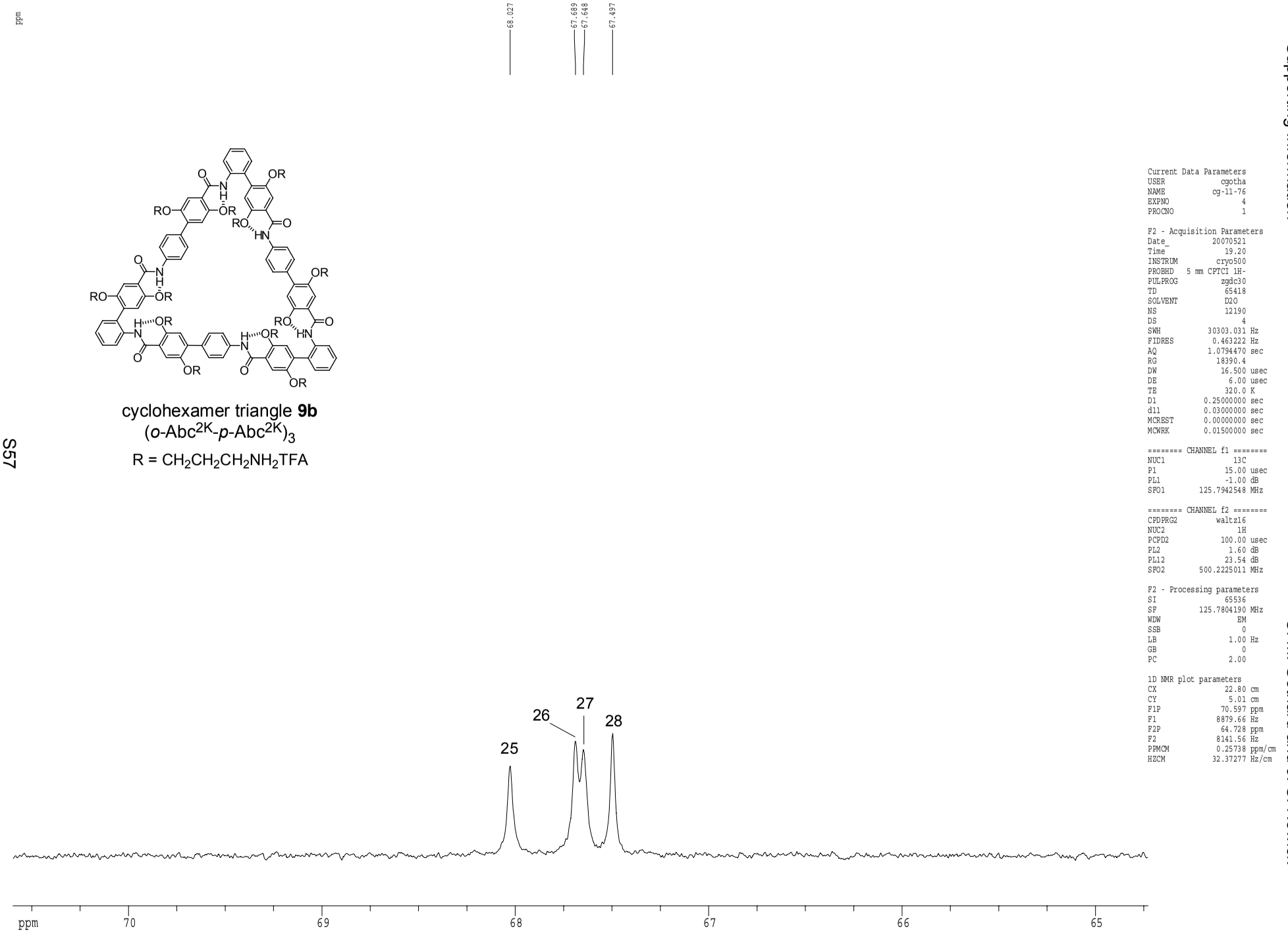
======== CHANNEL f2 ========
CPDPRG2           waltz16
NUC2                  1H
PCPD2             100.00 usec
PL2                 1.60 dB
PL12               23.54 dB
SFO2         500.2225011 MHz

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                   5.01 cm
F1P               153.555 ppm
F1              19314.26 Hz
F2P               146.772 ppm
F2              18461.11 Hz
PPMCM            0.29749 ppm/cm
HZCM            37.41886 Hz/cm
```


C. M. Gothard and J. S. Nowick

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



cyclohexamer triangle **9b**
(*o*-Abc^{2K}-*p*-Abc^{2K})$_3$
R = $CH_2CH_2CH_2NH_2TFA$

```
Current Data Parameters
USER                cgotha
NAME             cg-11-76
EXPNO                   4
PROCNO                  1

F2 - Acquisition Parameters
Date_            20070521
Time                19.20
INSTRUM           cryo500
PROBHD    5 mm CPTCI 1H-
PULPROG             zgdc30
TD                  65418
SOLVENT               D2O
NS                  12190
DS                      4
SWH             30303.031 Hz
FIDRES           0.463222 Hz
AQ               1.0794470 sec
RG                18390.4
DW                  16.500 usec
DE                   6.00 usec
TE                  320.0 K
D1              0.25000000 sec
d11             0.03000000 sec
MCREST          0.00000000 sec
MCWRK           0.01500000 sec

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

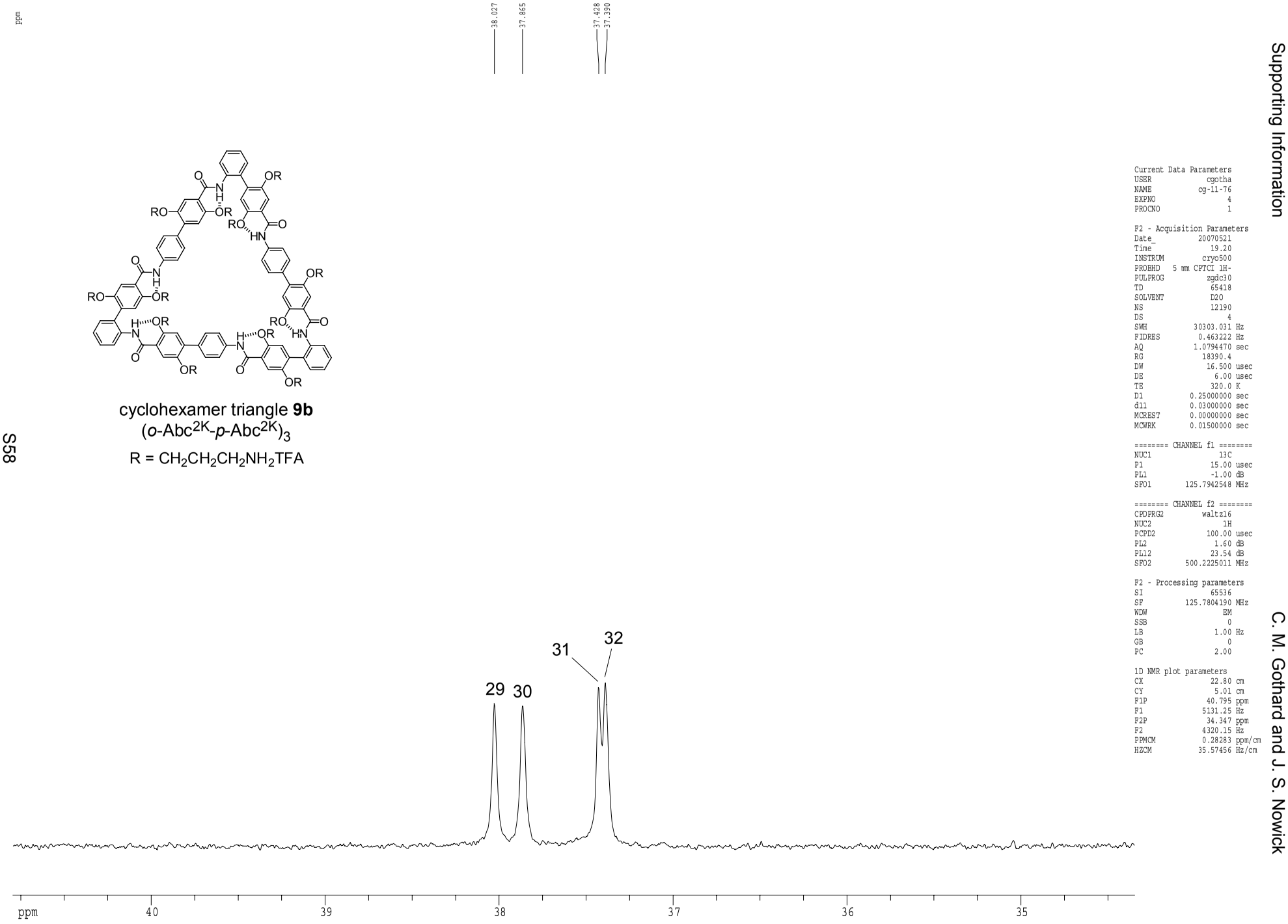
======== CHANNEL f2 ========
CPDPRG2           waltz16
NUC2                  1H
PCPD2              100.00 usec
PL2                  1.60 dB
PL12                23.54 dB
SFO2          500.2225011 MHz

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                   5.01 cm
F1P                70.597 ppm
F1                8879.66 Hz
F2P                64.728 ppm
F2                8141.56 Hz
PPMCM             0.25738 ppm/cm
HZCM             32.37277 Hz/cm
```


C. M. Gothard and J. S. Nowick

^{13}C NMR (125 MHz, 320 K, D_2O) spectrum of cyclohexamer triangle **9b** (aliphatic region expansion 2)



Current Data Parameters
USER cgotha
NAME cg-11-76
EXPNO 4
PROCNO 1

F2 - Acquisition Parameters
Date_ 20070521
Time 19.20
INSTRUM cryo500
PROBHD 5 mm CPTCI 1H-
PULPROG zgdc30
TD 65418
SOLVENT D2O
NS 12190
DS 4
SWH 30303.031 Hz
FIDRES 0.463222 Hz
AQ 1.0794470 sec
RG 18390.4
DW 16.500 usec
DE 6.00 usec
TE 320.0 K
D1 0.25000000 sec
d11 0.03000000 sec
MCREST 0.00000000 sec
MCWRK 0.01500000 sec

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

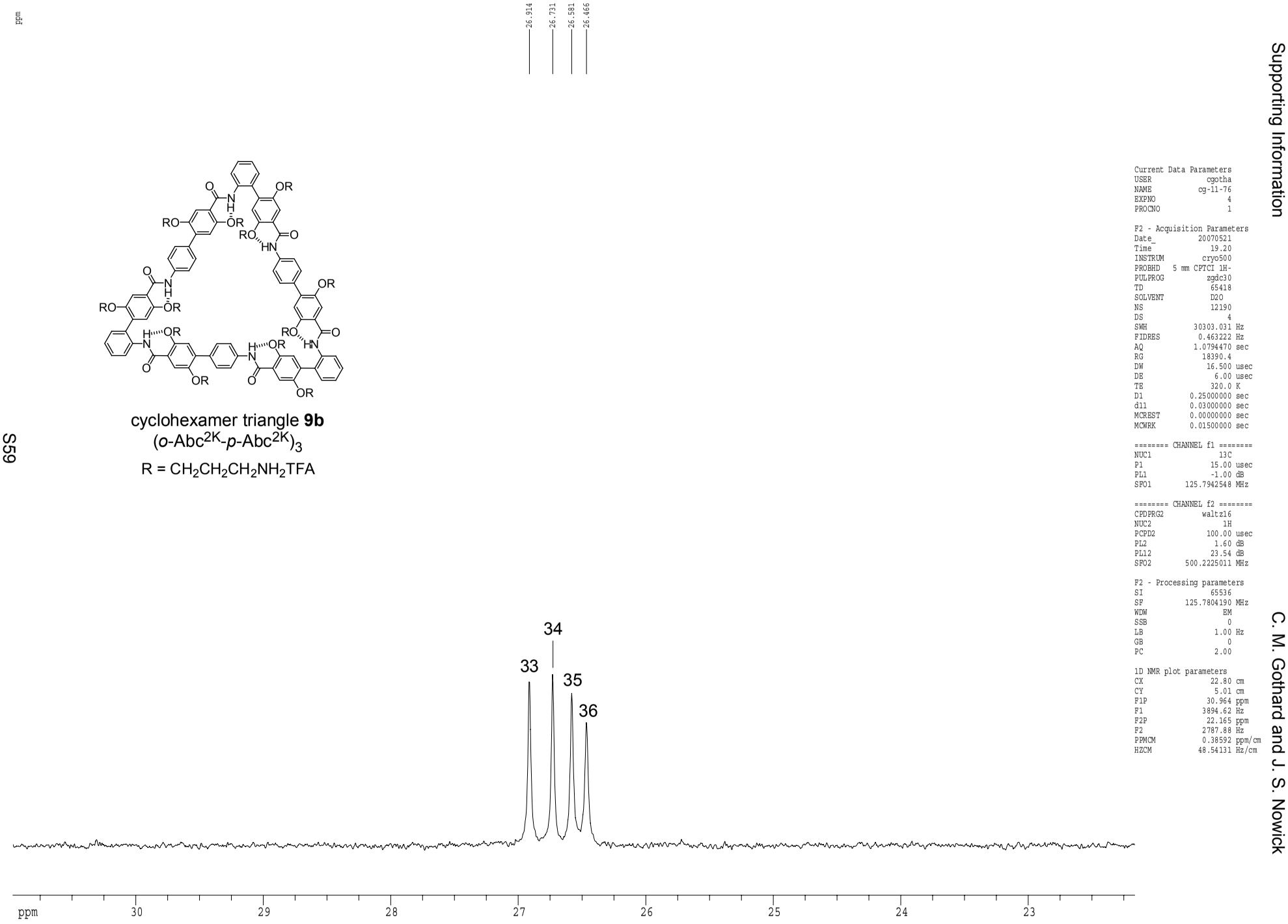
======== CHANNEL f2 ========
CPDPRG2 waltz16
NUC2 1H
PCPD2 100.00 usec
PL2 1.60 dB
PL12 23.54 dB
SFO2 500.2225011 MHz

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 5.01 cm
F1P 40.795 ppm
F1 5131.25 Hz
F2P 34.347 ppm
F2 4320.15 Hz
PPMCM 0.28283 ppm/cm
HZCM 35.57456 Hz/cm

C. M. Gothard and J. S. Nowick

^{13}C NMR (125 MHz, 320 K, D_2O) spectrum of cyclohexamer triangle **9b** (aliphatic region expansion 2)



cyclohexamer triangle **9b**
(*o*-Abc^{2K}-*p*-Abc^{2K})$_3$
R = $CH_2CH_2CH_2NH_2TFA$

```
Current Data Parameters
USER                cgotha
NAME             cg-11-76
EXPNO                   4
PROCNO                  1

F2 - Acquisition Parameters
Date_            20070521
Time                19.20
INSTRUM           cryo500
PROBHD    5 mm CPTCI 1H-
PULPROG             zgdc30
TD                  65418
SOLVENT               D2O
NS                    12190
DS                        4
SWH              30303.031 Hz
FIDRES            0.463222 Hz
AQ                1.0794470 sec
RG                  18390.4
DW                   16.500 usec
DE                    6.00 usec
TE                   320.0 K
D1              0.25000000 sec
d11             0.03000000 sec
MCREST          0.00000000 sec
MCWRK           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

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                    5.01 cm
F1P                 30.964 ppm
F1                 3894.62 Hz
F2P                 22.165 ppm
F2                 2787.88 Hz
PPMCM              0.38592 ppm/cm
HZCM              48.54131 Hz/cm
```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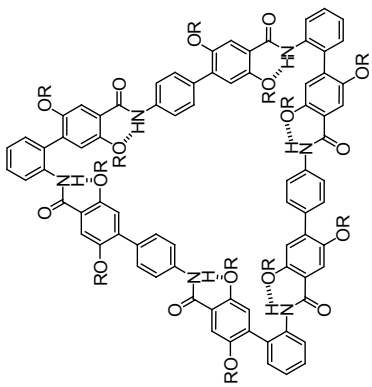
^{1}H NMR (500 MHz, 298 K, CD_3OD) spectrum of cyclohexamer triangle **9b**



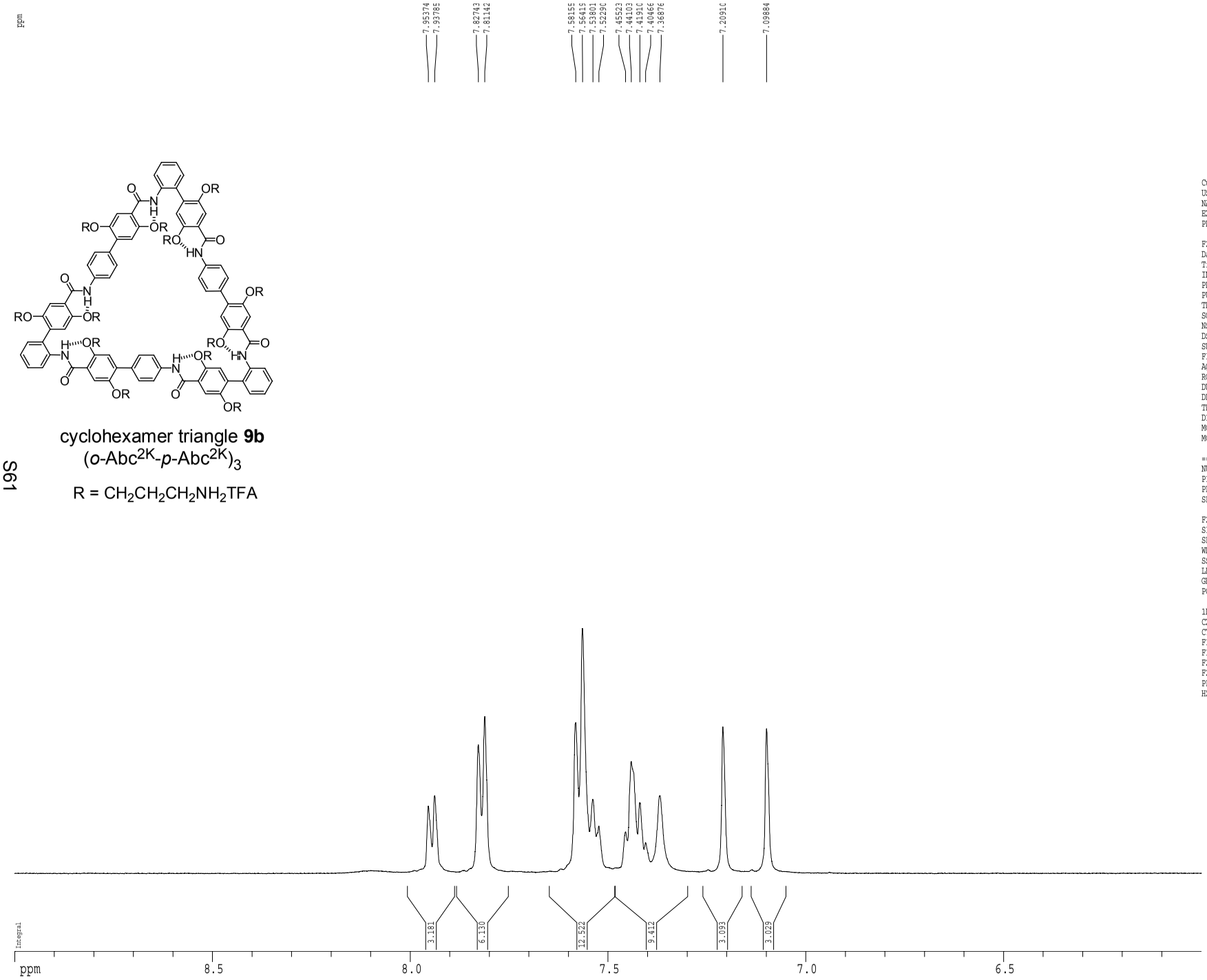
cyclohexamer triangle **9b**
(*o*-Abc^{2K}-*p*-Abc^{2K})$_3$

R = $CH_2CH_2CH_2NH_2TFA$

^{1}H NMR (500 MHz, 298 K, CD_3OD) spectrum of cyclohexamer triangle **9b** (aromatic region)

cyclohexamer triangle **9b**
(*o*-Abc^{2K}-*p*-Abc^{2K})$_3$

R = $CH_2CH_2CH_2NH_2TFA$




```
Current Data Parameters
USER                cgotha
NAME              cg-11-31
EXPNO                    7
PROCNO                   1

F2 - Acquisition Parameters
Date_            20071031
Time                18.11
INSTRUM             gn500
PROBHD   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                  161.3
DW                62.400 usec
DE                 6.00 usec
TE                298.0 K
D1             0.10000000 sec
MCREST         0.00000000 sec
MCWRK          0.01500000 sec

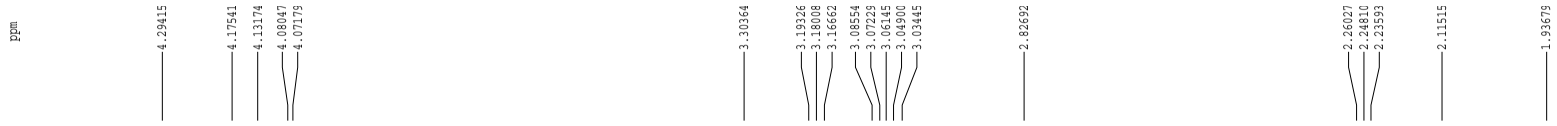
======== CHANNEL f1 ========
NUC1                   1H
P1                 12.00 usec
PL1                -3.00 dB
SFO1         499.8284988 MHz

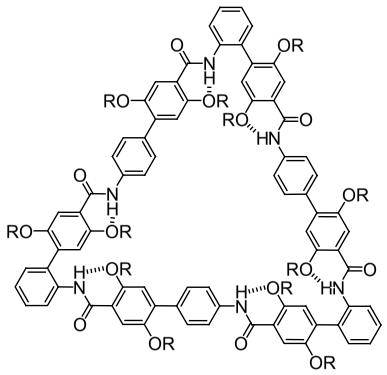
F2 - Processing parameters
SI                  65536
SF           499.8250210 MHz
WDW                    EM
SSB                     0
LB                   0.30 Hz
GB                      0
PC                   4.00

1D NMR plot parameters
CX                  22.80 cm
CY                  91.60 cm
F1P                 9.000 ppm
F1                4498.43 Hz
F2P                 6.000 ppm
F2                2998.95 Hz
PPMCM             0.13158 ppm/cm
HZCM             65.76645 Hz/cm
```


C. M. Gothard and J. S. Nowick

^{1}H NMR (500 MHz, 298 K, CD_3OD) spectrum of cyclohexamer triangle **9b** (aliphatic region)





cyclohexamer triangle **9b**
(*o*-Abc^{2K}-*p*-Abc^{2K})$_3$

R = $CH_2CH_2CH_2NH_2TFA$


```
Current Data Parameters
USER                cgotha
NAME              cg-11-31
EXPNO                    7
PROCNO                   1

F2 - Acquisition Parameters
Date_            20071031
Time                18.11
INSTRUM             gn500
PROBHD    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                  161.3
DW                62.400 usec
DE                 6.00 usec
TE                298.0 K
D1             0.10000000 sec
MCREST         0.00000000 sec
MCWRK          0.01500000 sec

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

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

1D NMR plot parameters
CX                  22.80 cm
CY                  91.60 cm
F1P                 4.560 ppm
F1                2279.16 Hz
F2P                 1.625 ppm
F2                 812.38 Hz
PPMCM            0.12871 ppm/cm
HZCM            64.33254 Hz/cm
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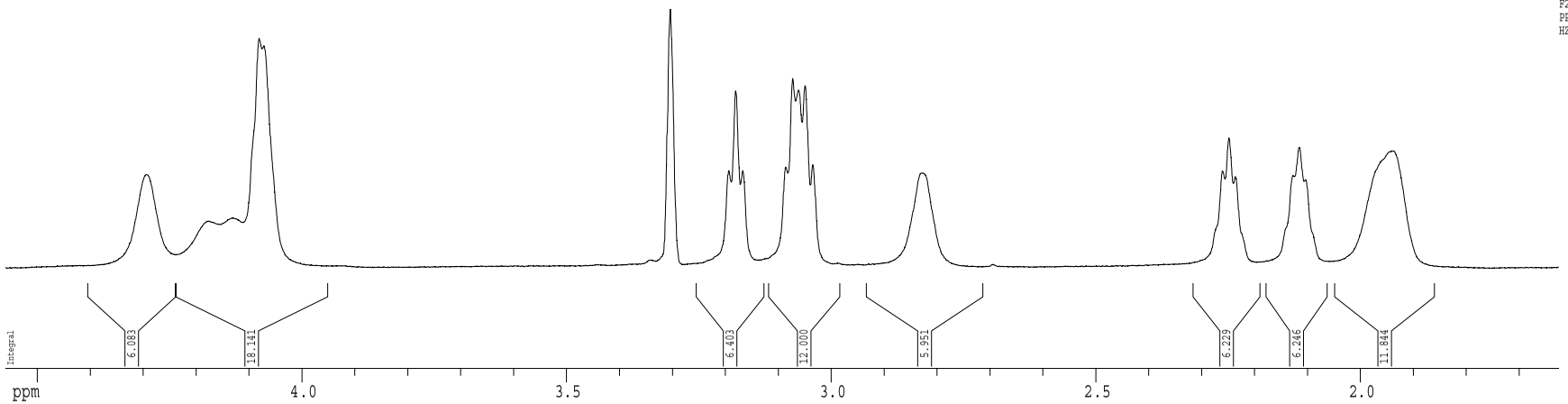

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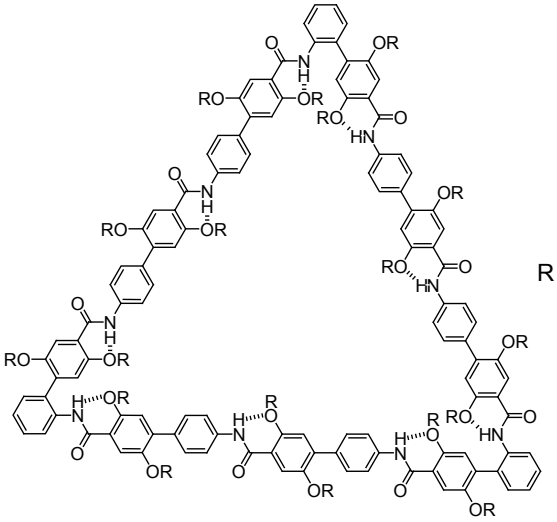
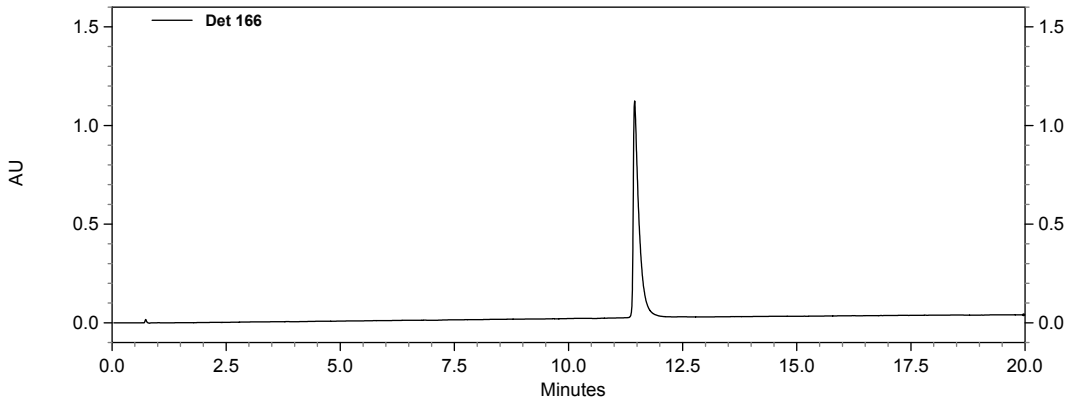
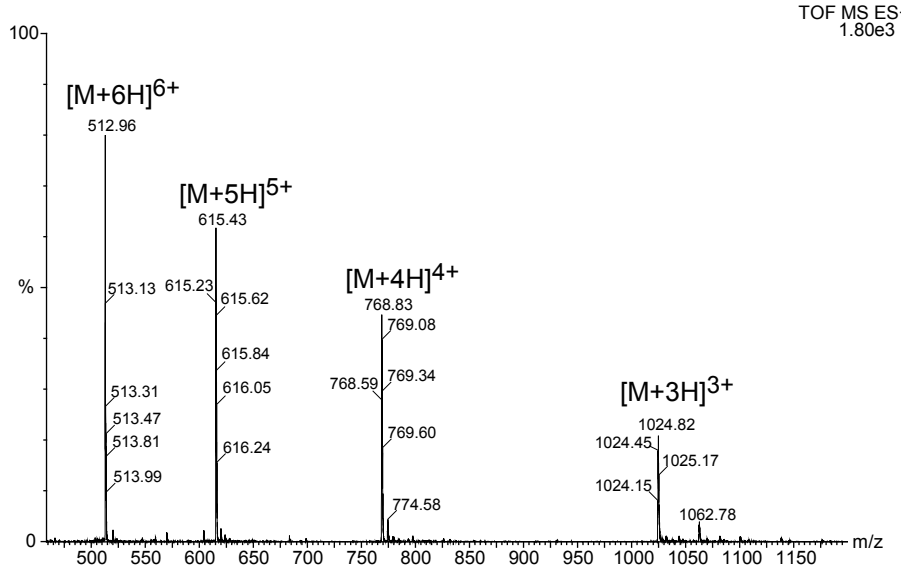
cyclononamer triangle: (*o*-Abc^{2K}-*p*-Abc^{2K}-*p*-Abc^{2K})$_3$ (**9c**)
Analytical RP-HPLC chromatograph and mass spectrum (ESI-MS)



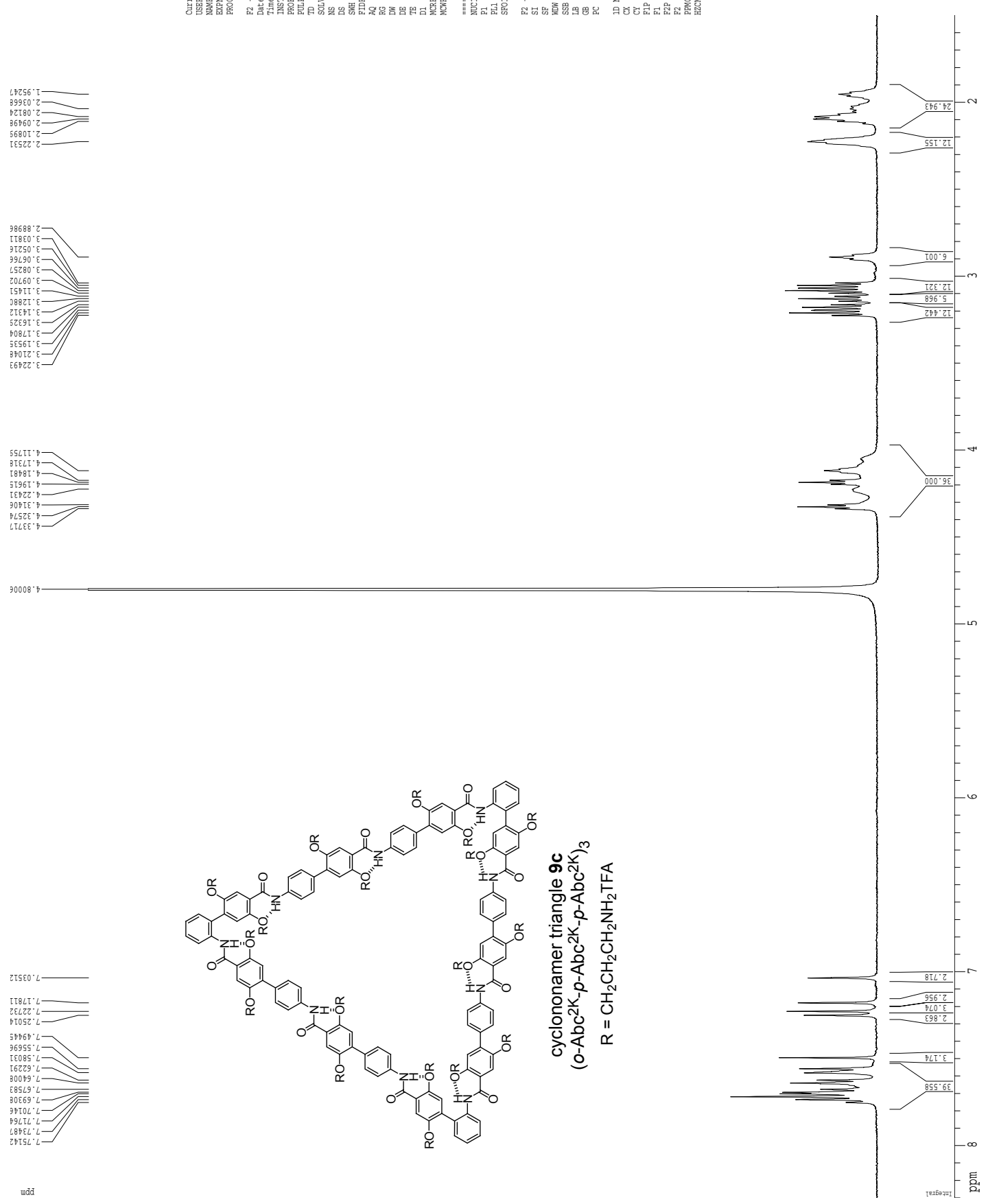
(a) Analytical RP-HPLC (5-50% acetonitrile with 0.1% TFA over 20 min, λ = 214)



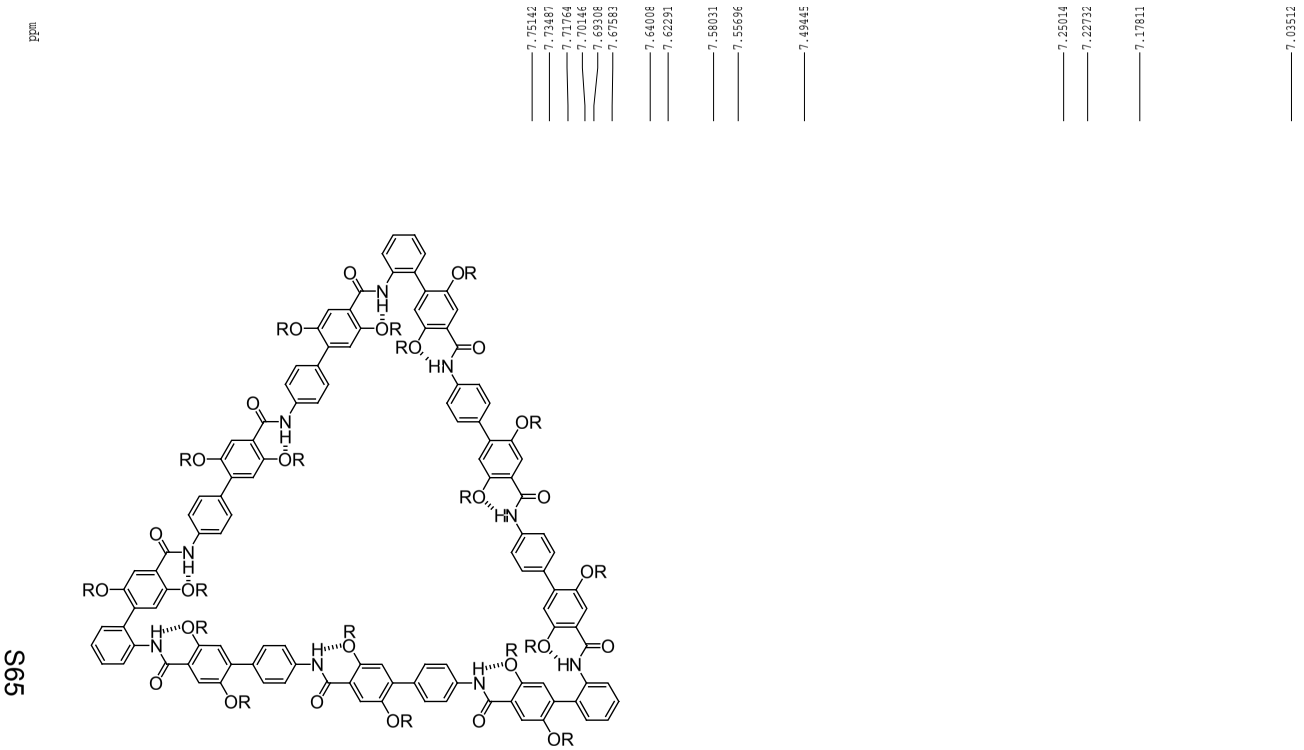
(b) ESI Mass spectrum. (Calcd exact mass for $C_{171}H_{207}N_{27}O_{27}$ [M] = 3070.57)



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



^{1}H NMR (500 MHz, 298 K, D_2O) spectrum of cyclononamer triangle **9c** (aromatic region)



cyclononamer triangle **9c**
(*o*-Abc^{2K}-*p*-Abc^{2K}$_2$)$_3$
R = $CH_2CH_2CH_2NH_2$TFA

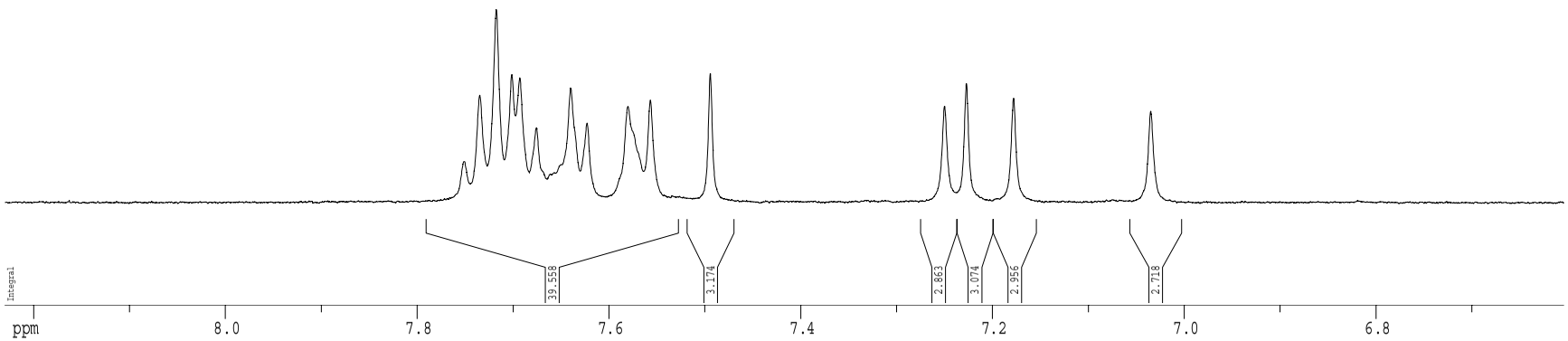
Current Data Parameters
USER cgotha
NAME cg-11-59
EXPNO 1
PROCNO 1

F2 - Acquisition Parameters
Date_ 20070509
Time 21.10
INSTRUM cryo500
PROBHD 5 mm CPTCI 1H-
PULPROG zg30
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 298.0 K
D1 0.10000000 sec
MCREST 0.00000000 sec
MCWRK 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
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 8.230 ppm
F1 4116.67 Hz
F2P 6.604 ppm
F2 3303.66 Hz
PPMCM 0.07129 ppm/cm
HZCM 35.65862 Hz/cm



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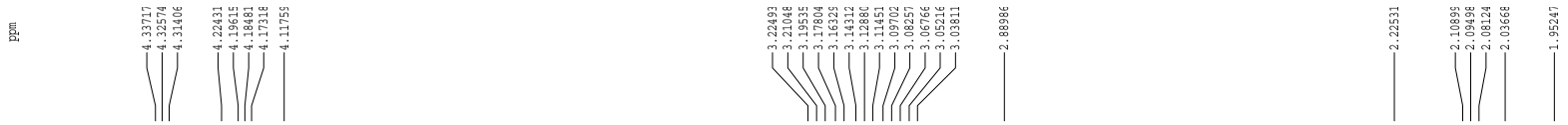
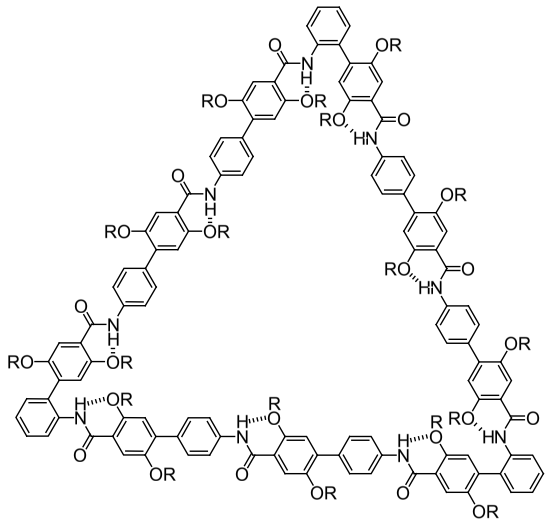
^{1}H NMR (500 MHz, 298 K, D_2O) spectrum of cyclononamer triangle **9c** (aliphatic region)





cyclononamer triangle **9c**
(*o*-Abc^{2K}-*p*-Abc^{2K}-*p*-Abc^{2K})$_3$

R = $CH_2CH_2CH_2NH_2TFA$

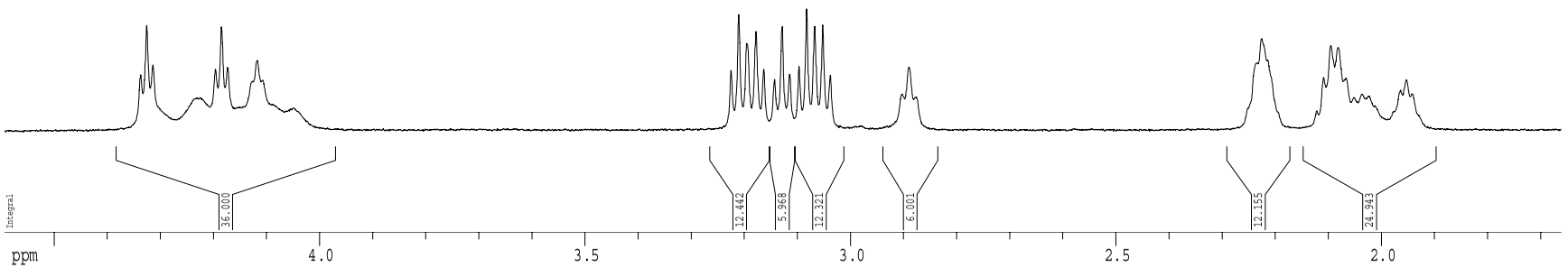

```
Current Data Parameters
USER              cgotha
NAME             cg-11-59
EXPNO                   1
PROCNO                  1

F2 - Acquisition Parameters
Date_           20070509
Time              21.10
INSTRUM          cryo500
PROBHD   5 mm CPTCI 1H-
PULPROG             zg30
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                298.0 K
D1           0.10000000 sec
MCREST       0.00000000 sec
MCWRK        0.01500000 sec

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

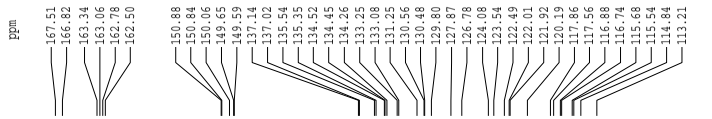
F2 - Processing parameters
SI                 65536
SF           500.2200051 MHz
WDW                   EM
SSB                    0
LB                  0.30 Hz
GB                     0
PC                  4.00

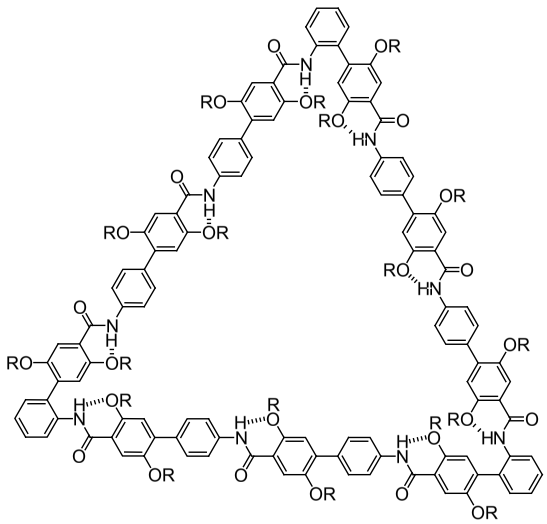
1D NMR plot parameters
CX                 22.80 cm
CY                300.57 cm
F1P                4.594 ppm
F1                2297.86 Hz
F2P                1.661 ppm
F2                 831.08 Hz
PPMCM           0.12861 ppm/cm
HZCM           64.33255 Hz/cm
```



C. M. Gothard and J. S. Nowick

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





cyclononamer triangle **9c**
$(o\text{-Abc}^{2K}\text{-}p\text{-Abc}^{2K}\text{-}p\text{-Abc}^{2K})_3$

R = $CH_2CH_2CH_2NH_2TFA$


```
Current Data Parameters
USER                cgotha
NAME             cg-11-59
EXPNO                   6
PROCNO                  1

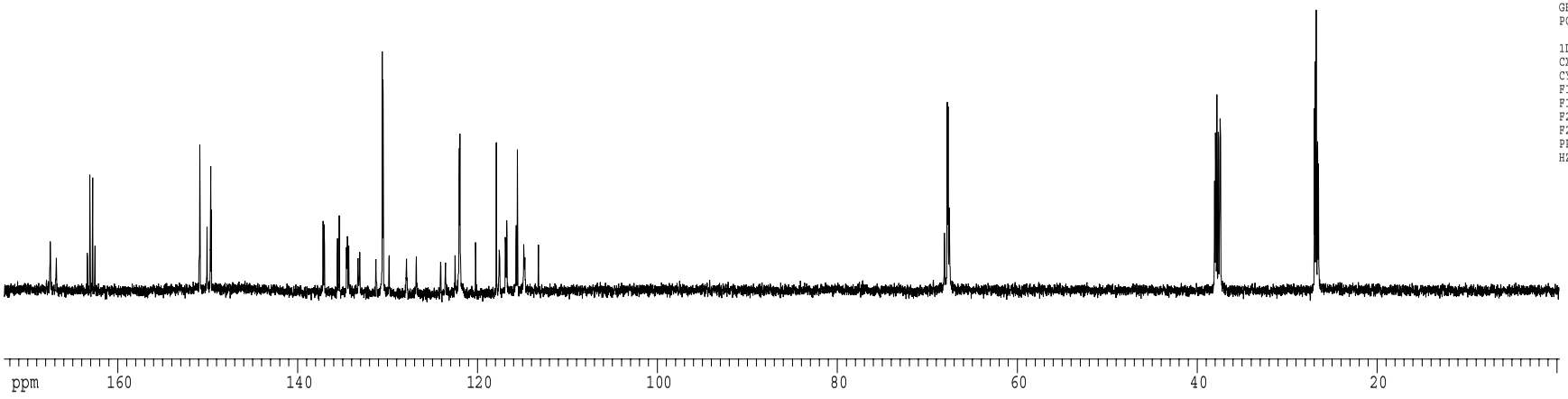
F2 - Acquisition Parameters
Date_            20070509
Time                21.43
INSTRUM           cryo500
PROBHD    5 mm CPTCI 1H-
PULPROG           zgdc30
TD                 65418
SOLVENT              D2O
NS                  8500
DS                     4
SWH            30303.031 Hz
FIDRES          0.463222 Hz
AQ              1.0794470 sec
RG                18390.4
DW                 16.500 usec
DE                  6.00 usec
TE                 320.0 K
D1              0.25000000 sec
d11             0.03000000 sec
MCREST          0.00000000 sec
MCWRK           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

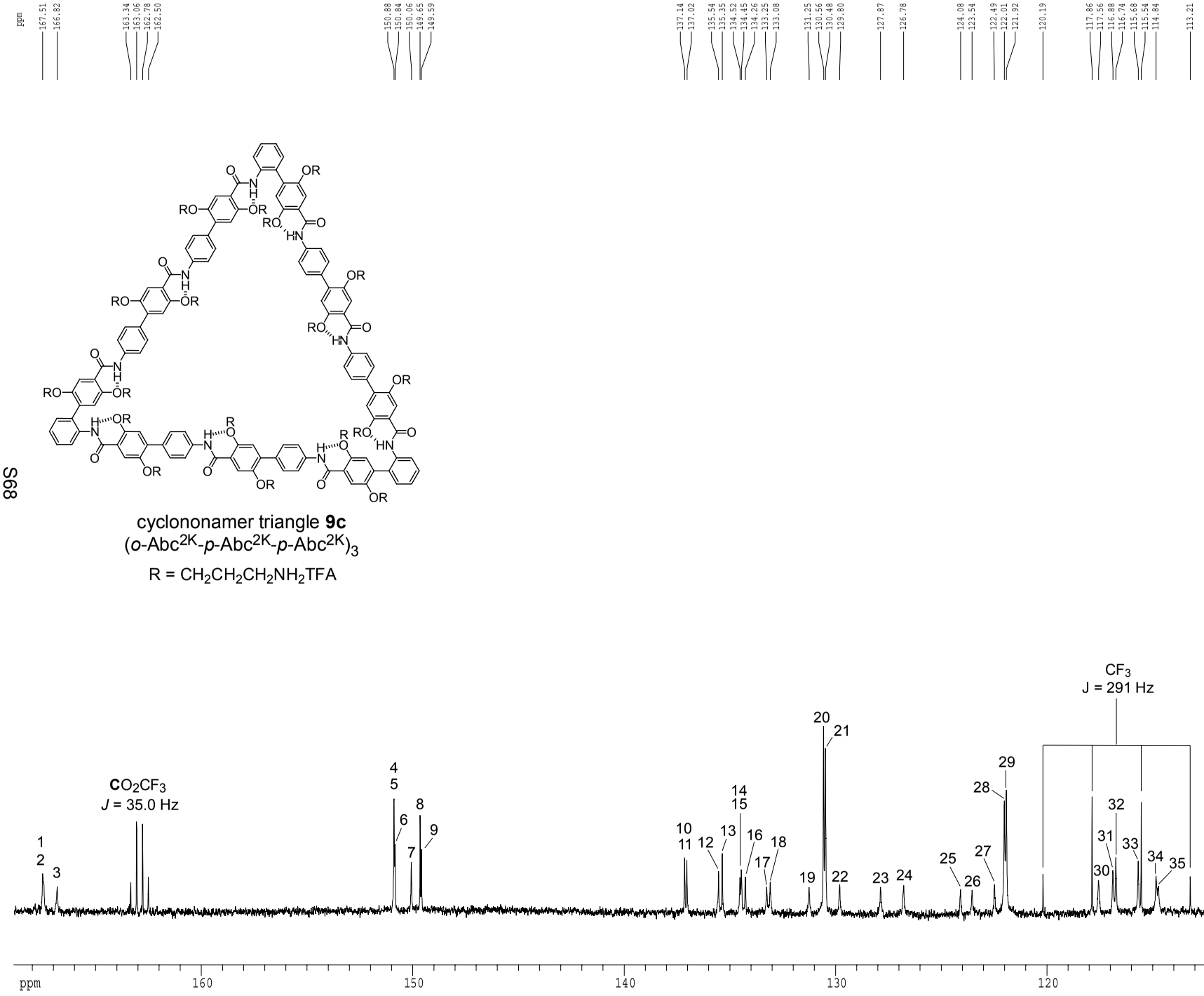
F2 - Processing parameters
SI                 65536
SF           125.7804190 MHz
WDW                   EM
SSB                    0
LB                  1.00 Hz
GB                     0
PC                  2.00

1D NMR plot parameters
CX                 22.80 cm
CY                  4.15 cm
F1P               172.606 ppm
F1              21710.48 Hz
F2P                -0.248 ppm
F2                -31.24 Hz
PPMCM           7.58134 ppm/cm
HZCM          953.58447 Hz/cm
```



C. M. Gothard and J. S. Nowick

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

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Current Data Parameters
USER                cgotha
NAME             cg-11-59
EXPNO                   6
PROCNO                  1

F2 - Acquisition Parameters
Date_            20070509
Time                21.43
INSTRUM           cryo500
PROBHD    5 mm CPTCI 1H-
PULPROG           zgdc30
TD                 65418
SOLVENT              D2O
NS                  8500
DS                     4
SWH            30303.031 Hz
FIDRES          0.463222 Hz
AQ              1.0794470 sec
RG                18390.4
DW                 16.500 usec
DE                  6.00 usec
TE                 320.0 K
D1              0.25000000 sec
d11             0.03000000 sec
MCREST          0.00000000 sec
MCWRK           0.01500000 sec

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

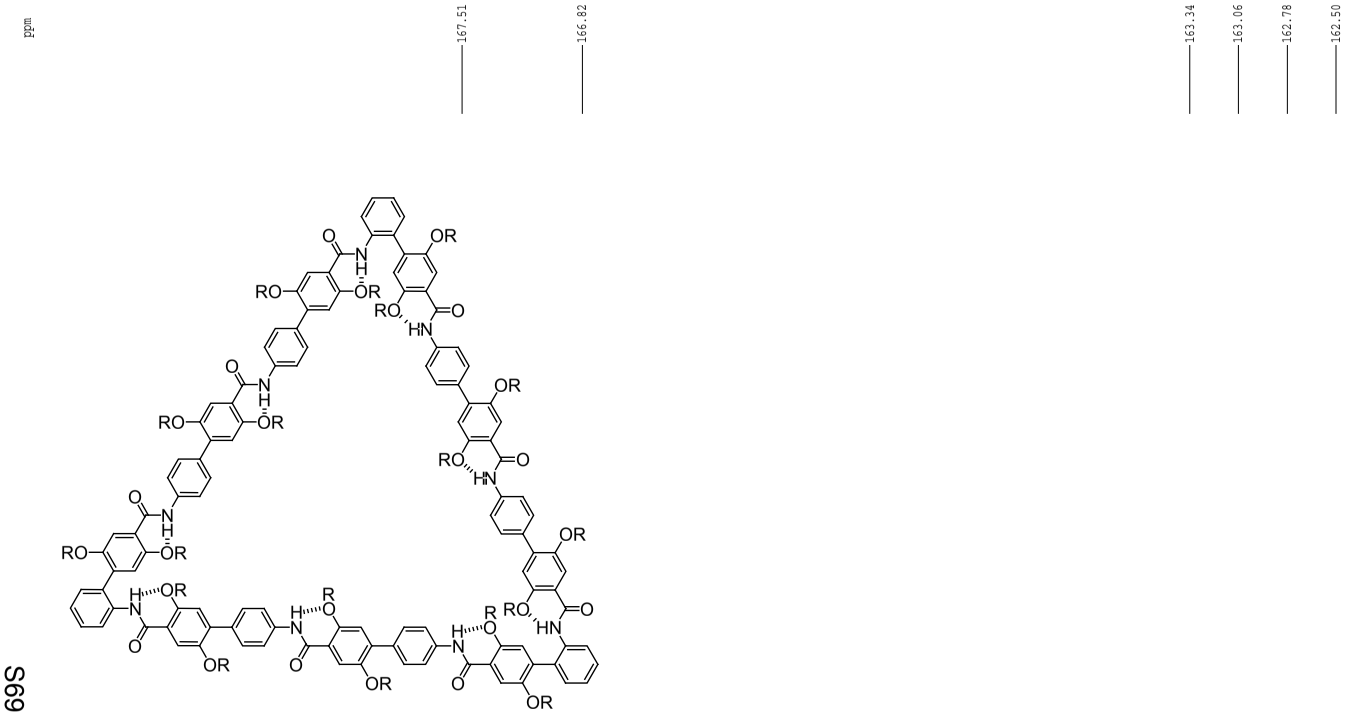
======== CHANNEL f2 ========
CPDPRG2           waltz16
NUC2                  1H
PCPD2              100.00 usec
PL2                 1.60 dB
PL12               23.54 dB
SFO2          500.2225011 MHz

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
F1P               168.865 ppm
F1              21239.86 Hz
F2P               111.692 ppm
F2              14048.72 Hz
PPMCM             2.50755 ppm/cm
HZCM            315.40091 Hz/cm
```


C. M. Gothard and J. S. Nowick

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



cyclononamer triangle **9c**
(*o*-Abc^{2K}-*p*-Abc^{2K}-*p*-Abc^{2K})$_3$

R = $CH_2CH_2CH_2NH_2TFA$


```
Current Data Parameters
USER                cgotha
NAME             cg-11-59
EXPNO                   6
PROCNO                  1

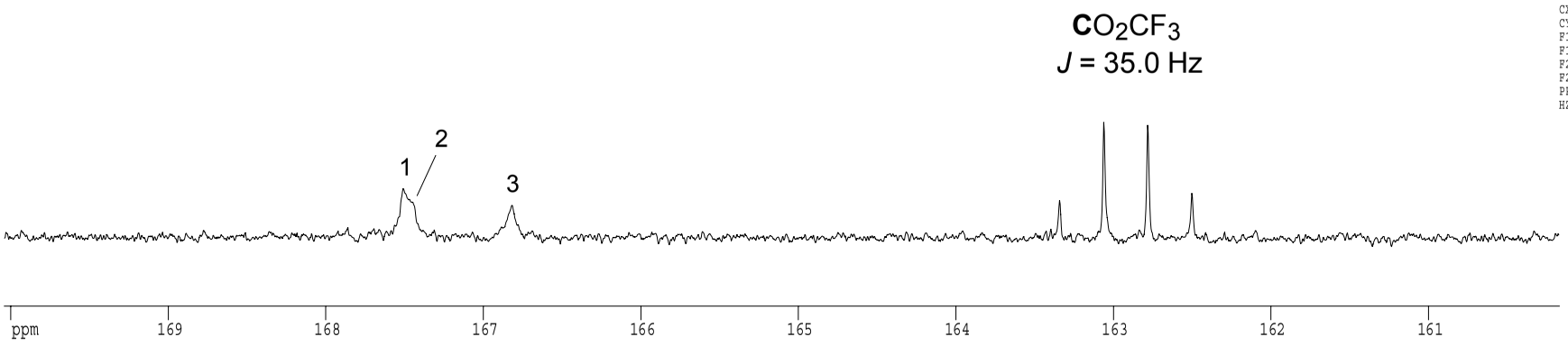
F2 - Acquisition Parameters
Date_            20070509
Time                21.43
INSTRUM           cryo500
PROBHD    5 mm CPTCI 1H-
PULPROG           zgdc30
TD                  65418
SOLVENT               D2O
NS                     8500
DS                        4
SWH              30303.031 Hz
FIDRES            0.463222 Hz
AQ                1.0794470 sec
RG                  18390.4
DW                   16.500 usec
DE                    6.00 usec
TE                   320.0 K
D1                0.25000000 sec
d11               0.03000000 sec
MCREST            0.00000000 sec
MCWRK             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

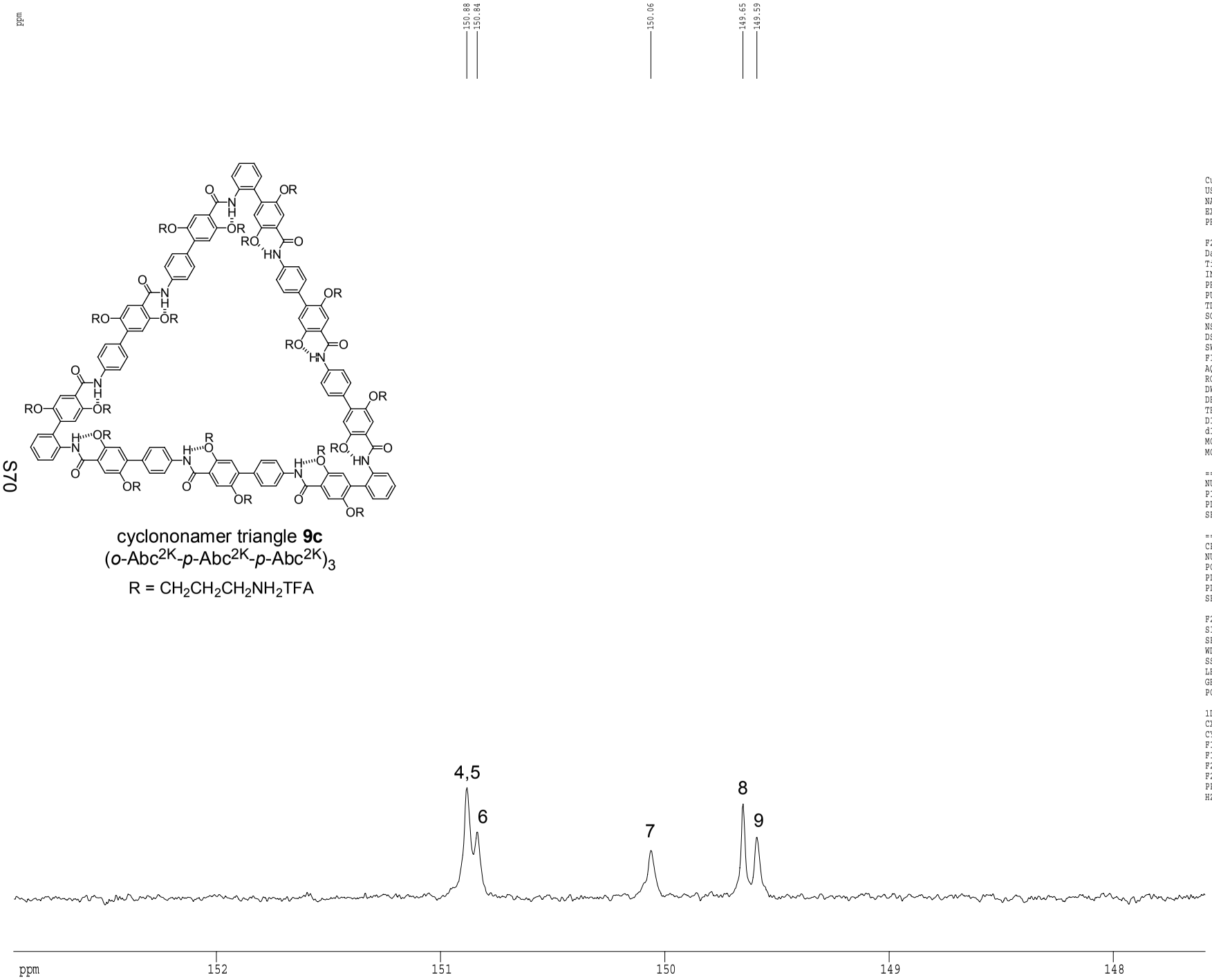
F2 - Processing parameters
SI                     65536
SF             125.7804190 MHz
WDW                       EM
SSB                        0
LB                      1.00 Hz
GB                         0
PC                      2.00

1D NMR plot parameters
CX                     22.80 cm
CY                      4.15 cm
F1P                  170.040 ppm
F1                 21387.74 Hz
F2P                  160.170 ppm
F2                 20146.22 Hz
PPMCM                0.43292 ppm/cm
HZCM                54.45252 Hz/cm
```



C. M. Gothard and J. S. Nowick

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



cyclononamer triangle **9c**
(*o*-Abc^{2K}-*p*-Abc^{2K}-*p*-Abc^{2K})$_3$

R = $CH_2CH_2CH_2NH_2TFA$

```
Current Data Parameters
USER                cgotha
NAME             cg-11-59
EXPNO                   6
PROCNO                  1

F2 - Acquisition Parameters
Date_            20070509
Time                21.43
INSTRUM           cryo500
PROBHD    5 mm CPTCI 1H-
PULPROG           zgdc30
TD                  65418
SOLVENT               D2O
NS                   8500
DS                      4
SWH             30303.031 Hz
FIDRES           0.463222 Hz
AQ               1.0794470 sec
RG                 18390.4
DW                  16.500 usec
DE                   6.00 usec
TE                  320.0 K
D1              0.25000000 sec
d11             0.03000000 sec
MCREST          0.00000000 sec
MCWRK           0.01500000 sec

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

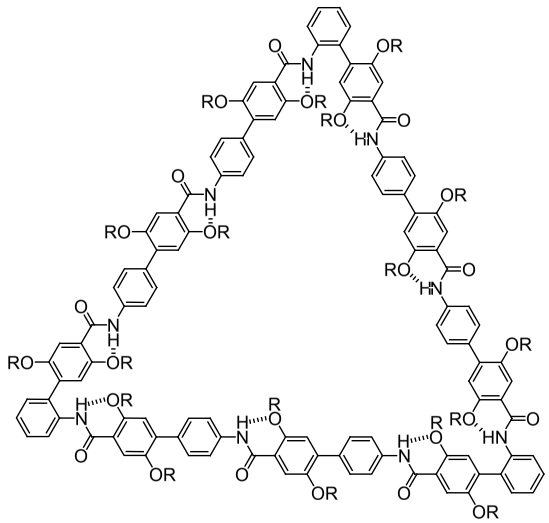
======== CHANNEL f2 ========
CPDPRG2           waltz16
NUC2                  1H
PCPD2              100.00 usec
PL2                 1.60 dB
PL12                23.54 dB
SFO2          500.2225011 MHz

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
F1P                152.904 ppm
F1               19232.28 Hz
F2P                147.591 ppm
F2               18564.00 Hz
PPMCM             0.23303 ppm/cm
HZCM             29.31067 Hz/cm
```

C. M. Gothard and J. S. Nowick

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



cyclononamer triangle **9c**
$(o\text{-Abc}^{2K}\text{-}p\text{-Abc}^{2K}\text{-}p\text{-Abc}^{2K})_3$

R = $CH_2CH_2CH_2NH_2TFA$


```
Current Data Parameters
USER                cgotha
NAME              cg-11-59
EXPNO                    6
PROCNO                   1

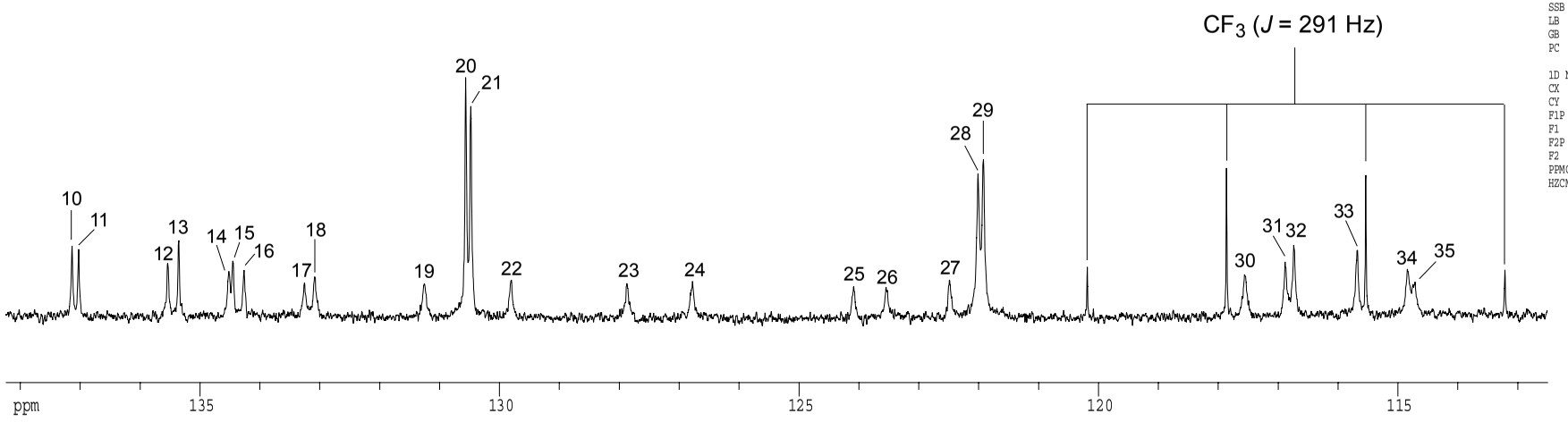
F2 - Acquisition Parameters
Date_            20070509
Time                21.43
INSTRUM           cryo500
PROBHD    5 mm CPTCI 1H-
PULPROG             zgdc30
TD                   65418
SOLVENT                D2O
NS                    8500
DS                       4
SWH             30303.031 Hz
FIDRES              0.463222 Hz
AQ                1.0794470 sec
RG                  18390.4
DW                    16.500 usec
DE                     6.00 usec
TE                    320.0 K
D1                0.25000000 sec
d11               0.03000000 sec
MCREST            0.00000000 sec
MCWRK             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

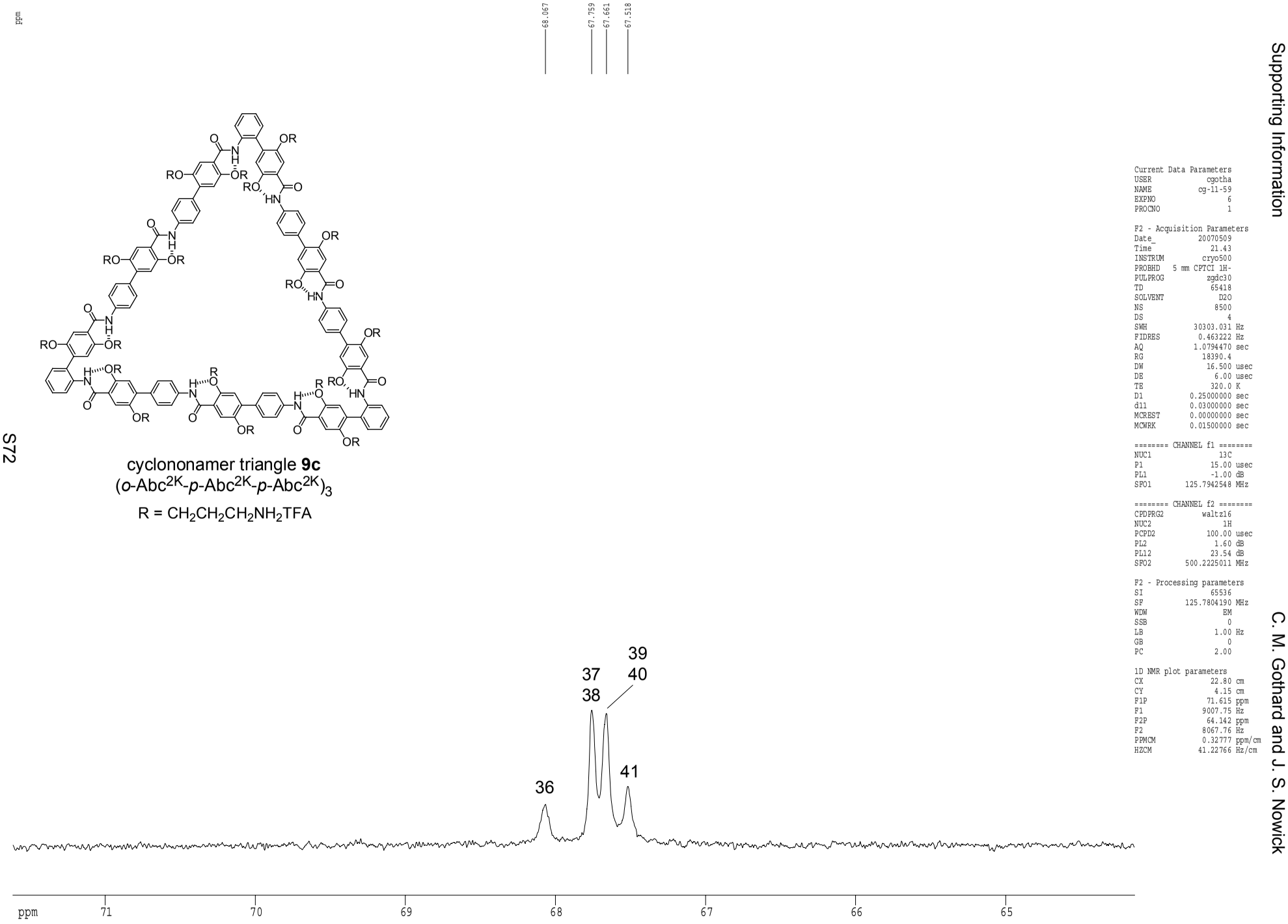
F2 - Processing parameters
SI                      65536
SF             125.7804190 MHz
WDW                        EM
SSB                         0
LB                     1.00 Hz
GB                          0
PC                     2.00

1D NMR plot parameters
CX                     22.80 cm
CY                      4.15 cm
F1P                  138.237 ppm
F1                 17387.46 Hz
F2P                  112.500 ppm
F2                 14150.31 Hz
PPMCM                1.12879 ppm/cm
HZCM               141.98015 Hz/cm
```



C. M. Gothard and J. S. Nowick

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



cyclononamer triangle **9c**
(*o*-Abc^{2K}-*p*-Abc^{2K}-*p*-Abc^{2K})$_3$

R = $CH_2CH_2CH_2NH_2TFA$

```
Current Data Parameters
USER                cgotha
NAME              cg-11-59
EXPNO                    6
PROCNO                   1

F2 - Acquisition Parameters
Date_             20070509
Time                 21.43
INSTRUM            cryo500
PROBHD    5 mm CPTCI 1H-
PULPROG             zgdc30
TD                    65418
SOLVENT                 D2O
NS                       8500
DS                          4
SWH               30303.031 Hz
FIDRES             0.463222 Hz
AQ                 1.0794470 sec
RG                   18390.4
DW                    16.500 usec
DE                     6.00 usec
TE                    320.0 K
D1                 0.25000000 sec
d11                0.03000000 sec
MCREST             0.00000000 sec
MCWRK              0.01500000 sec

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

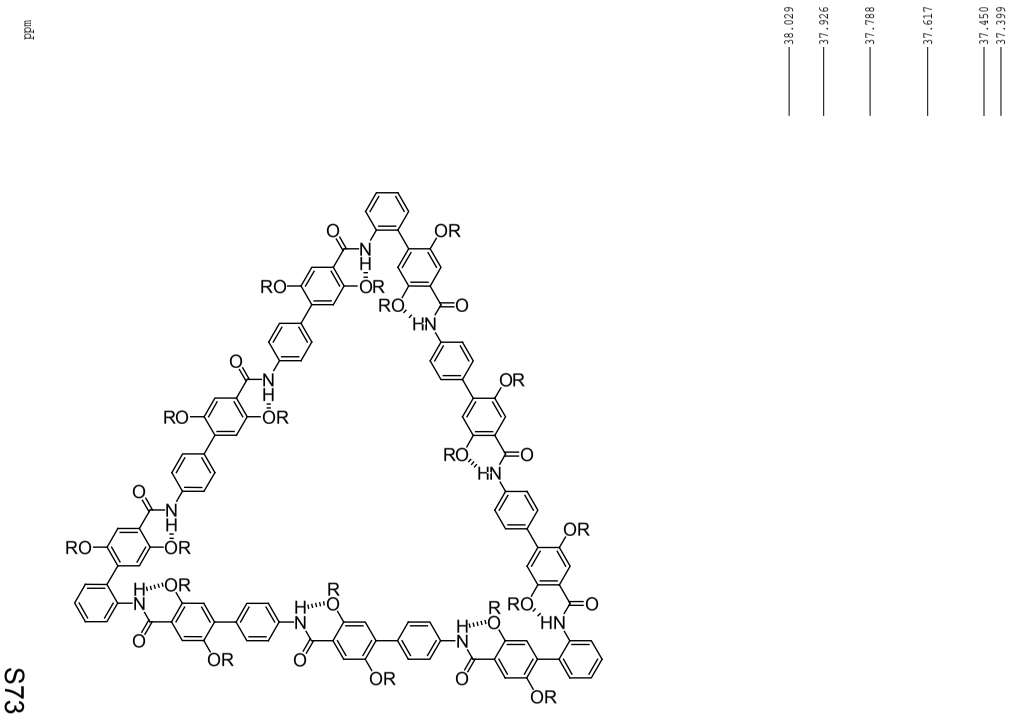
======== CHANNEL f2 ========
CPDPRG2              waltz16
NUC2                     1H
PCPD2                100.00 usec
PL2                    1.60 dB
PL12                  23.54 dB
SFO2             500.2225011 MHz

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
F1P                     71.615 ppm
F1                     9007.75 Hz
F2P                     64.142 ppm
F2                     8067.76 Hz
PPMCM                 0.32777 ppm/cm
HZCM                 41.22766 Hz/cm
```


C. M. Gothard and J. S. Nowick

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



cyclononamer triangle **9c**
(*o*-Abc^{2K}-*p*-Abc^{2K}-*p*-Abc^{2K})$_3$

R = $CH_2CH_2CH_2NH_2TFA$

```
Current Data Parameters
USER                cgotha
NAME              cg-11-59
EXPNO                    6
PROCNO                   1

F2 - Acquisition Parameters
Date_             20070509
Time                 21.43
INSTRUM            cryo500
PROBHD    5 mm CPTCI 1H-
PULPROG             zgdc30
TD                    65418
SOLVENT                 D2O
NS                       8500
DS                          4
SWH                30303.031 Hz
FIDRES              0.463222 Hz
AQ                  1.0794470 sec
RG                    18390.4
DW                     16.500 usec
DE                      6.00 usec
TE                     320.0 K
D1                  0.25000000 sec
d11                 0.03000000 sec
MCREST              0.00000000 sec
MCWRK               0.01500000 sec

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

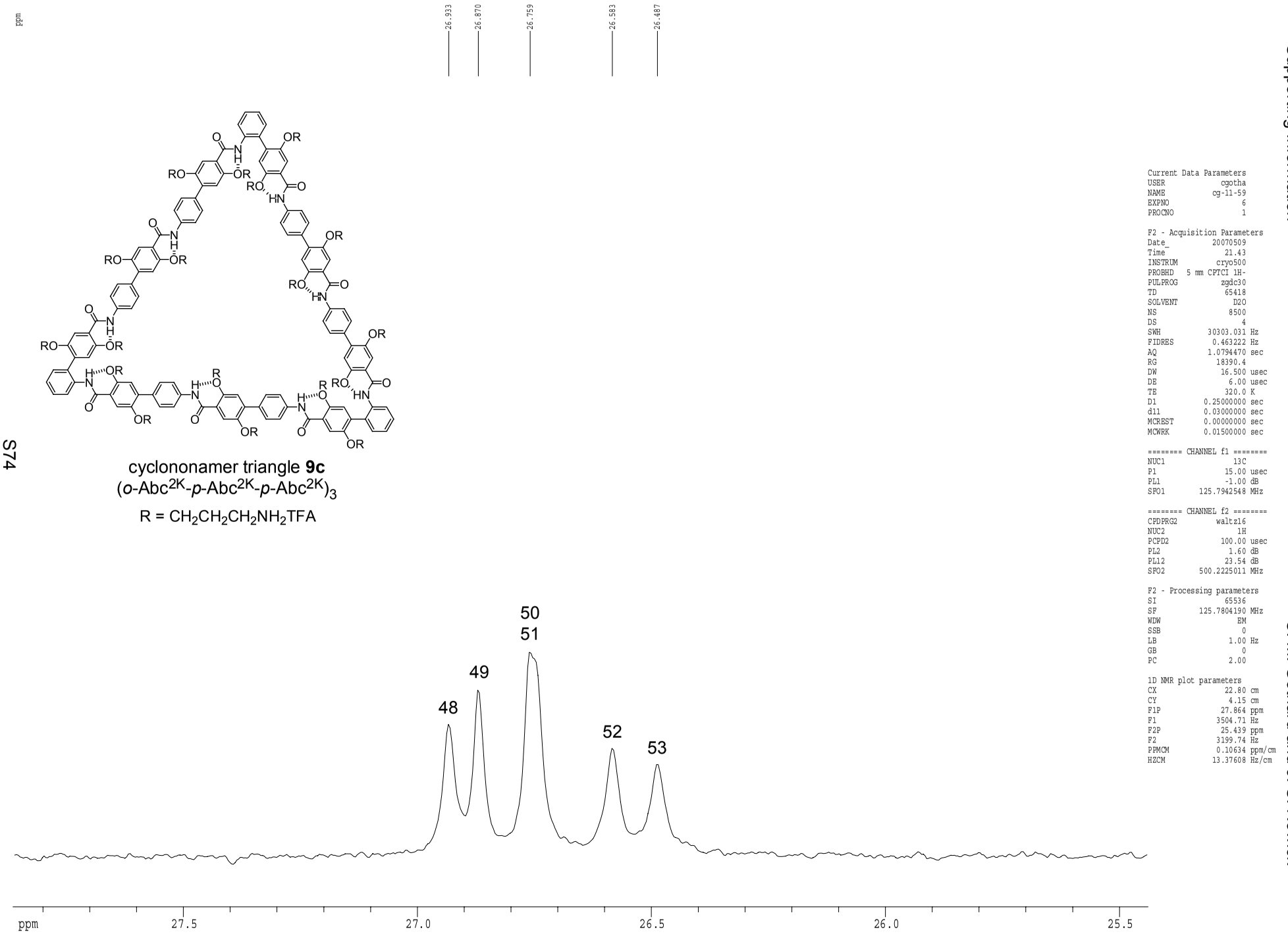
======== CHANNEL f2 ========
CPDPRG2               waltz16
NUC2                      1H
PCPD2                  100.00 usec
PL2                     1.60 dB
PL12                   23.54 dB
SFO2             500.2225011 MHz

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
F1P                   40.315 ppm
F1                   5070.88 Hz
F2P                    35.192 ppm
F2                   4426.51 Hz
PPMCM                 0.22469 ppm/cm
HZCM                 28.26186 Hz/cm
```


C. M. Gothard and J. S. Nowick

^{13}C NMR (125 MHz, 320 K, D_2O) spectrum of cyclononamer triangle **9c** (aliphatic region expansion 3)



cyclononamer triangle **9c**
(*o*-Abc^{2K}-*p*-Abc^{2K}-*p*-Abc^{2K})$_3$

R = $CH_2CH_2CH_2NH_2TFA$

```
Current Data Parameters
USER                cgotha
NAME             cg-11-59
EXPNO                   6
PROCNO                  1

F2 - Acquisition Parameters
Date_            20070509
Time                21.43
INSTRUM           cryo500
PROBHD    5 mm CPTCI 1H-
PULPROG           zgdc30
TD                 65418
SOLVENT              D2O
NS                    8500
DS                       4
SWH             30303.031 Hz
FIDRES           0.463222 Hz
AQ               1.0794470 sec
RG                 18390.4
DW                  16.500 usec
DE                   6.00 usec
TE                  320.0 K
D1              0.25000000 sec
d11             0.03000000 sec
MCREST          0.00000000 sec
MCWRK           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

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

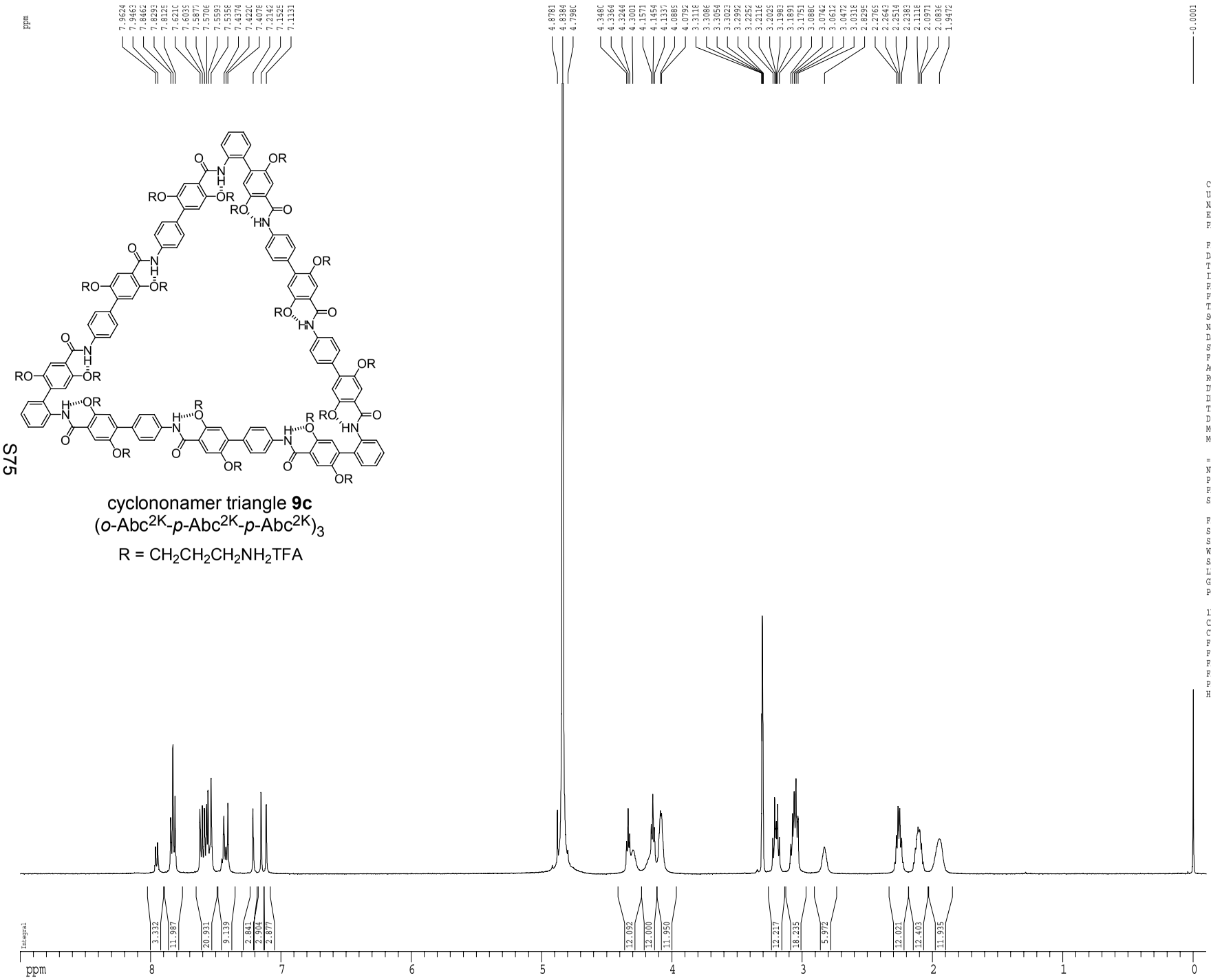
1D NMR plot parameters
CX                   22.80 cm
CY                    4.15 cm
F1P                27.864 ppm
F1                3504.71 Hz
F2P                25.439 ppm
F2                3199.74 Hz
PPMCM             0.10634 ppm/cm
HZCM             13.37608 Hz/cm
```


C. M. Gothard and J. S. Nowick

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

cyclononamer triangle **9c**
(*o*-Abc^{2K}-*p*-Abc^{2K}-*p*-Abc^{2K})$_3$

R = $CH_2CH_2CH_2NH_2TFA$




```
Current Data Parameters
USER                cgotha
NAME             cg-11-58
EXPNO                   1
PROCNO                  1

F2 - Acquisition Parameters
Date_            20071031
Time                17.54
INSTRUM             gn500
PROBHD   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                  724.1
DW                62.400 usec
DE                 6.00 usec
TE                298.0 K
D1              0.10000000 sec
MCREST          0.00000000 sec
MCWRK           0.01500000 sec

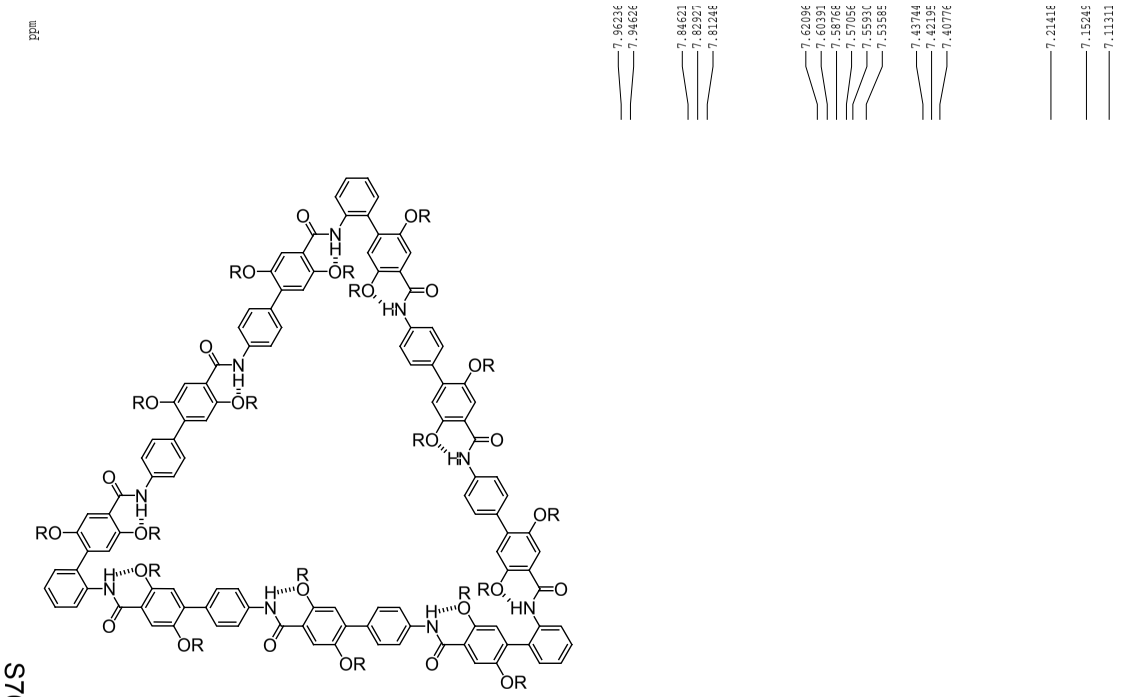
======== CHANNEL f1 ========
NUC1                   1H
P1                  12.00 usec
PL1                 -3.00 dB
SFO1          499.8284988 MHz

F2 - Processing parameters
SI                  65536
SF            499.8250207 MHz
WDW                    EM
SSB                     0
LB                   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                -0.100 ppm
F2                 -49.98 Hz
PPMCM            0.39912 ppm/cm
HZCM           199.49158 Hz/cm
```

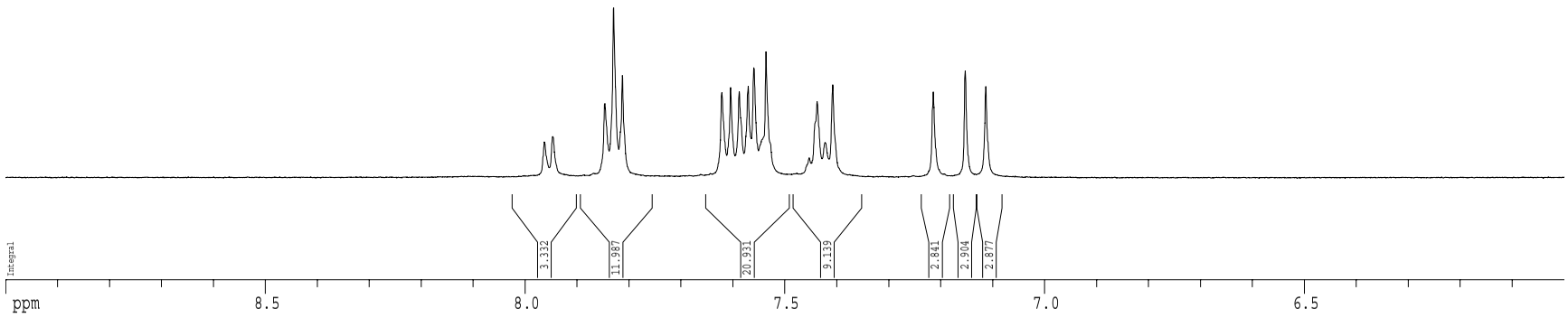

C. M. Gothard and J. S. Nowick

^{1}H 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 = $CH_2CH_2CH_2NH_2TFA$




```
Current Data Parameters
USER              cgotha
NAME            cg-11-58
EXPNO                  1
PROCNO                 1

F2 - Acquisition Parameters
Date_           20071031
Time              17.54
INSTRUM           gn500
PROBHD   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                724.1
DW               62.400 usec
DE                6.00 usec
TE                298.0 K
D1           0.10000000 sec
MCREST       0.00000000 sec
MCWRK        0.01500000 sec

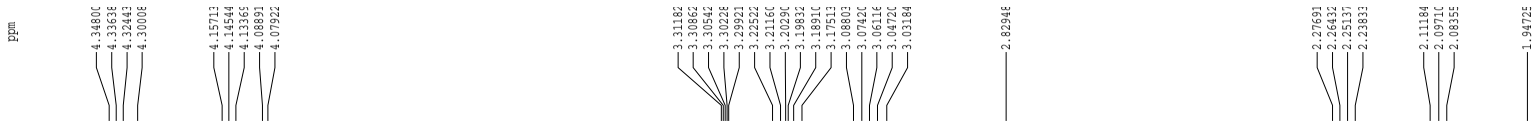
======== CHANNEL f1 ========
NUC1                1H
P1                12.00 usec
PL1               -3.00 dB
SFO1         499.8284988 MHz

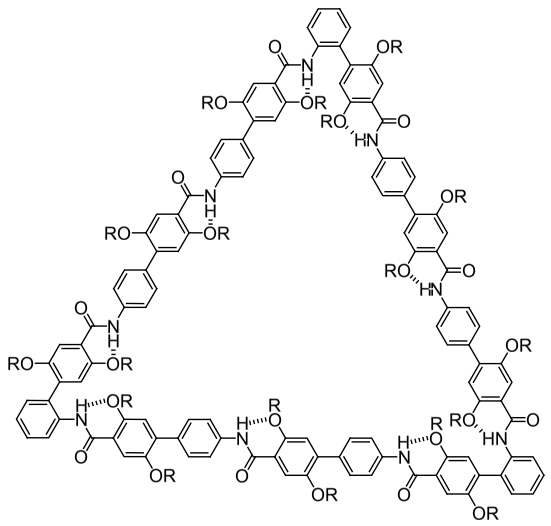
F2 - Processing parameters
SI                65536
SF           499.8250207 MHz
WDW                  EM
SSB                   0
LB                 0.30 Hz
GB                    0
PC                 4.00

1D NMR plot parameters
CX                22.80 cm
CY                20.36 cm
F1P               9.000 ppm
F1              4498.43 Hz
F2P               6.000 ppm
F2              2998.95 Hz
PPMCM           0.13158 ppm/cm
HZCM           65.76645 Hz/cm
```


C. M. Gothard and J. S. Nowick

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





cyclononamer triangle **9c**
(*o*-Abc^{2K}-*p*-Abc^{2K}-*p*-Abc^{2K})$_3$

R = $CH_2CH_2CH_2NH_2TFA$


```
Current Data Parameters
USER                cgotha
NAME             cg-11-58
EXPNO                   1
PROCNO                  1

F2 - Acquisition Parameters
Date_            20071031
Time                17.54
INSTRUM             gn500
PROBHD   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                  724.1
DW                 62.400 usec
DE                  6.00 usec
TE                298.0 K
D1             0.10000000 sec
MCREST         0.00000000 sec
MCWRK          0.01500000 sec

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

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

1D NMR plot parameters
CX                  22.80 cm
CY                  80.36 cm
F1P                 4.527 ppm
F1                2262.77 Hz
F2P                 1.609 ppm
F2                 804.37 Hz
PPMCM            0.12797 ppm/cm
HZCM            63.96493 Hz/cm
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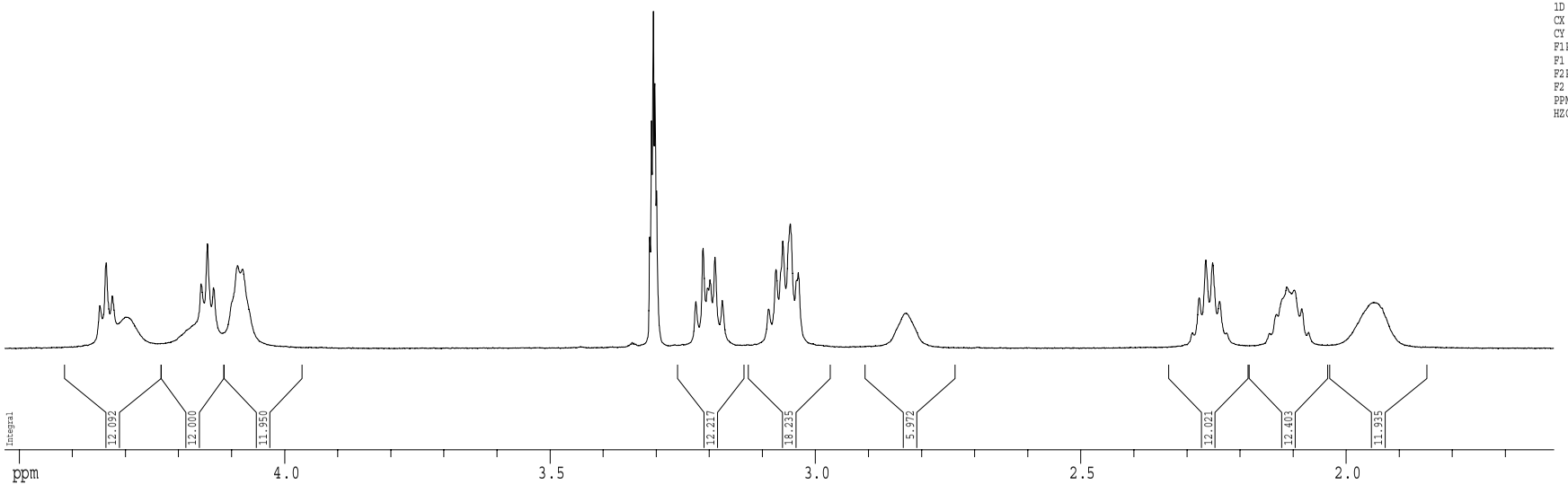
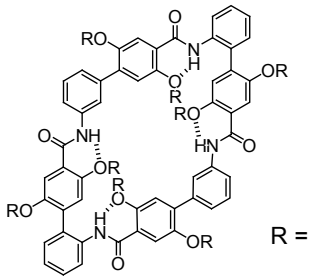


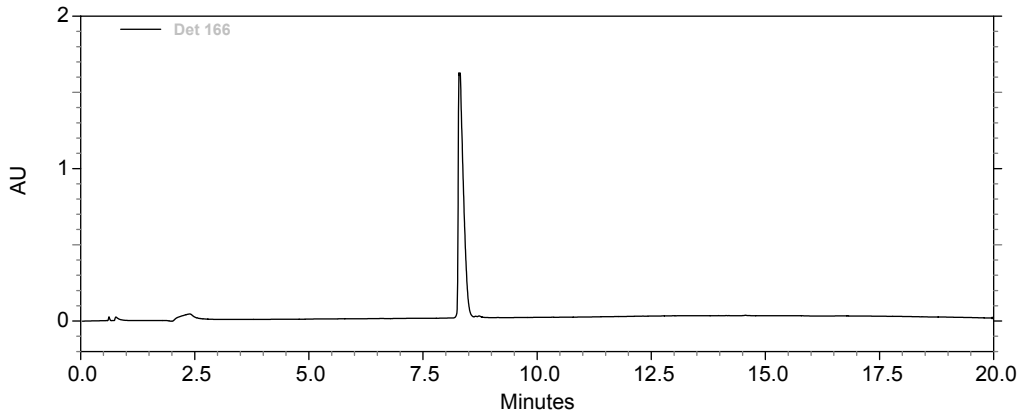
C. M. Gothard and J. S. Nowick

cyclotetramer: (*m*-Abc^{2K}-*o*-Abc^{2K})$_2$ (**10a**)
Analytical RP-HPLC chromatograph and mass spectrum (ESI-MS)

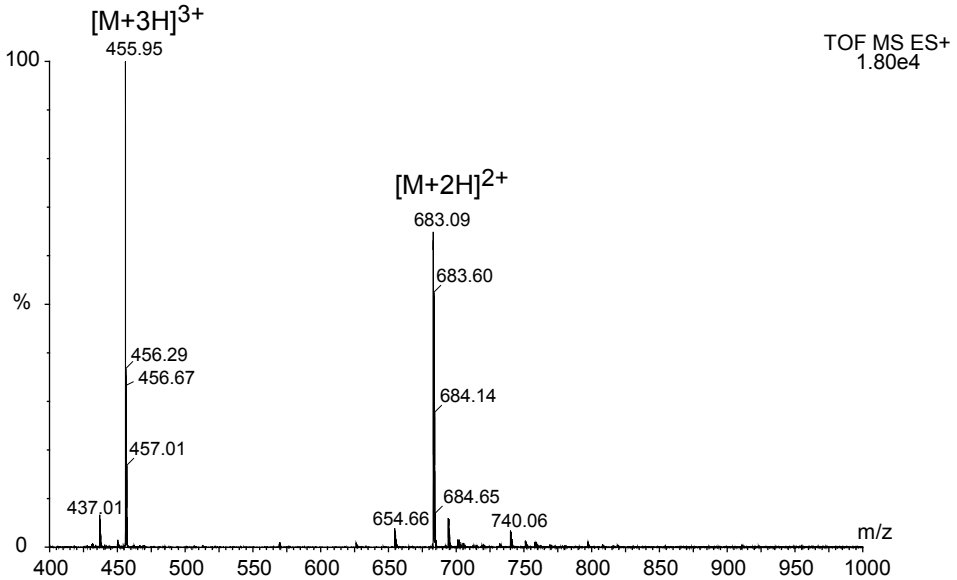


R = $CH_2CH_2CH_2NH_2TFA$

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



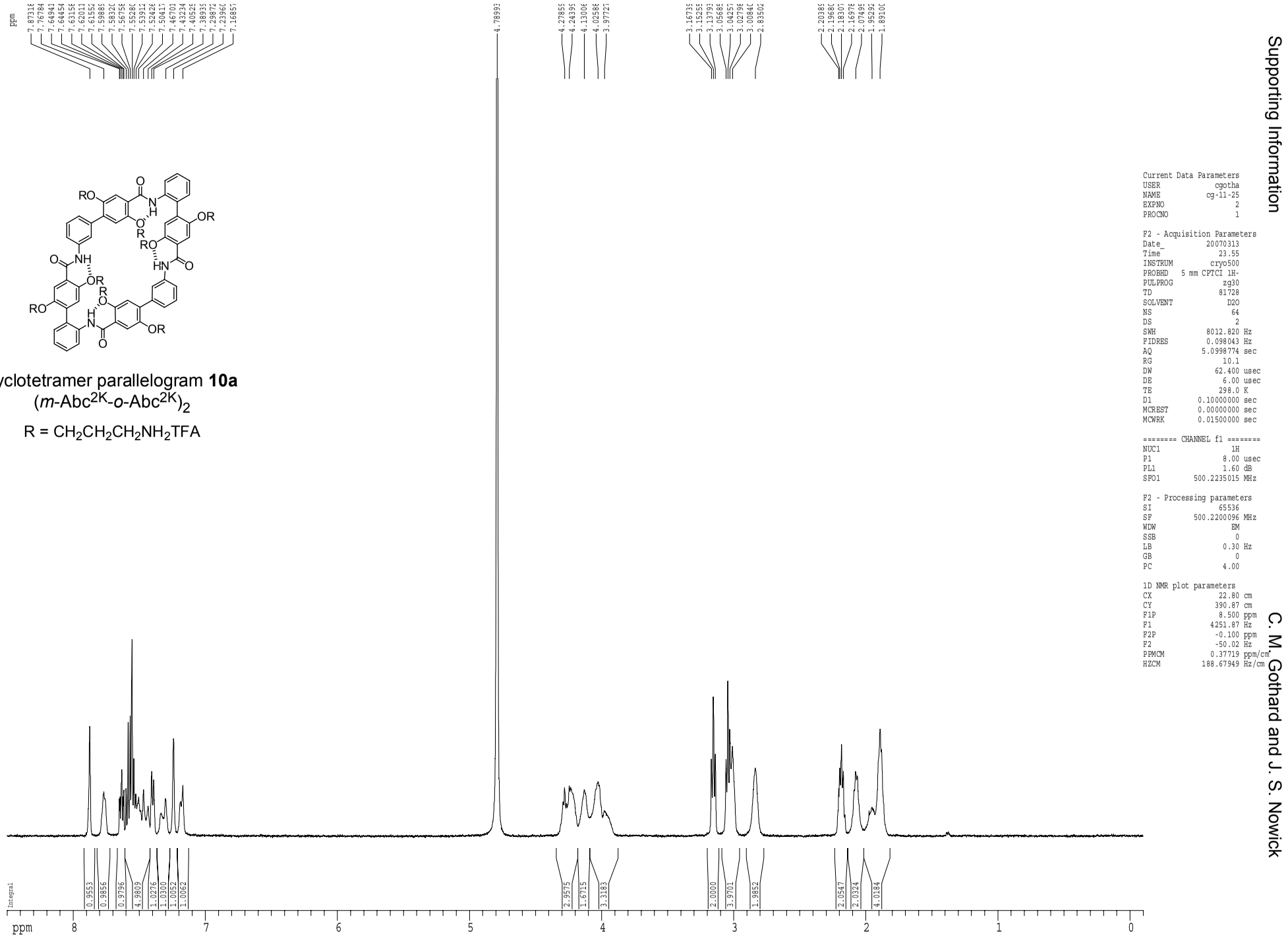
(b) ESI Mass spectrum. (Calcd exact mass for $C_{76}H_{96}N_{12}O_{12}$ [M] = 1364.70)



^{1}H NMR (500 MHz, 298 K, D_2O) spectrum of cyclotetramer parallelogram **10a**

cyclotetramer parallelogram **10a**
(*m*-Abc^{2K}-*o*-Abc^{2K})$_2$

R = $CH_2CH_2CH_2NH_2TFA$

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Current Data Parameters
USER                cgotha
NAME             cg-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
SWH              8012.820 Hz
FIDRES           0.098043 Hz
AQ               5.0998774 sec
RG                    10.1
DW                 62.400 usec
DE                  6.00 usec
TE                 298.0 K
D1             0.10000000 sec
MCREST         0.00000000 sec
MCWRK          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            500.2200096 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
F1P                 8.500 ppm
F1                4251.87 Hz
F2P                -0.100 ppm
F2                 -50.02 Hz
PPMCM            0.37719 ppm/cm
HZCM           188.67949 Hz/cm
```

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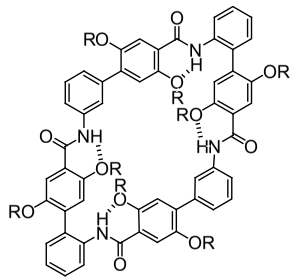
^{1}H NMR (500 MHz, 298 K, D_2O) spectrum of cyclotetramer parallelogram **10a** (aromatic region)



cyclotetramer parallelogram **10a**
$(m\text{-Abc}^{2K}\text{-}o\text{-Abc}^{2K})_2$

R = $CH_2CH_2CH_2NH_2TFA$

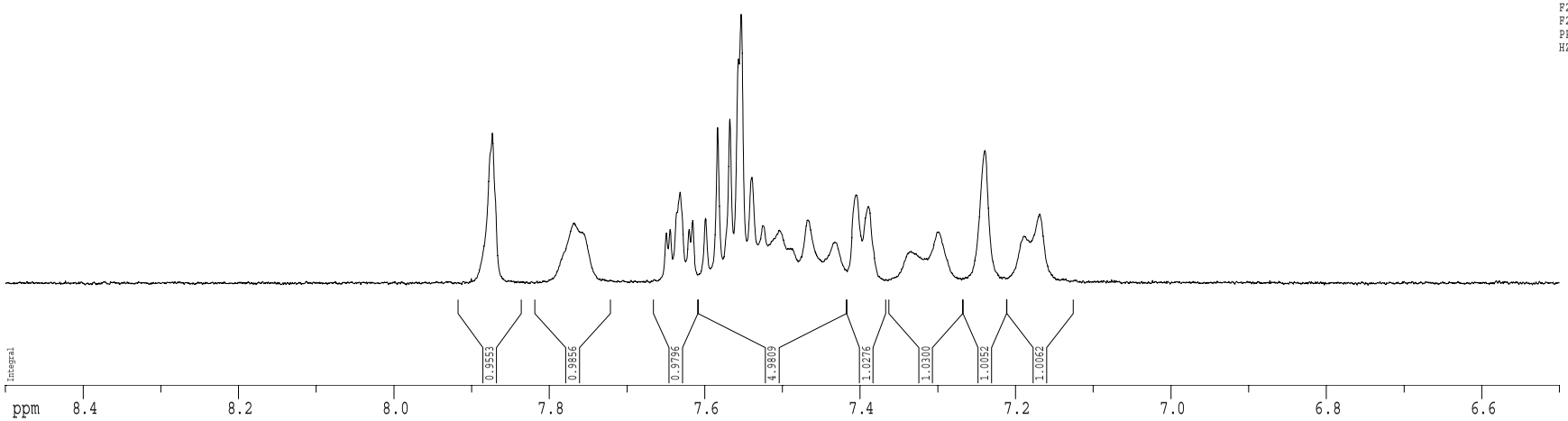

```
Current Data Parameters
USER                cgotha
NAME              cg-11-25
EXPNO                    2
PROCNO                   1

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
SWH              8012.820 Hz
FIDRES           0.098043 Hz
AQ               5.0998774 sec
RG                    10.1
DW                 62.400 usec
DE                  6.00 usec
TE                 298.0 K
D1              0.10000000 sec
MCREST          0.00000000 sec
MCWRK           0.01500000 sec

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

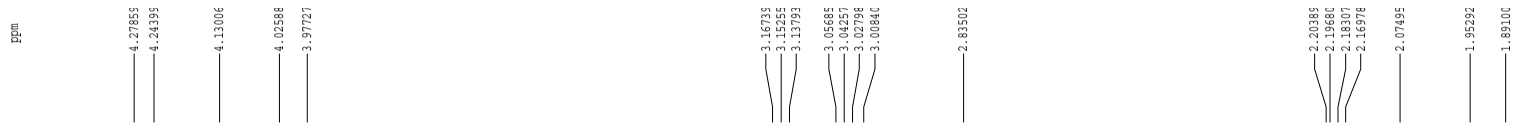
F2 - Processing parameters
SI                    65536
SF            500.2200096 MHz
WDW                      EM
SSB                       0
LB                   0.30 Hz
GB                        0
PC                    4.00

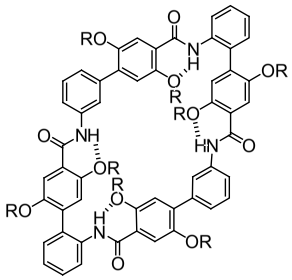
1D NMR plot parameters
CX                   22.80 cm
CY                  390.87 cm
F1P                  8.500 ppm
F1                4251.87 Hz
F2P                  6.500 ppm
F2                3251.43 Hz
PPMCM             0.08772 ppm/cm
HZCM             43.87895 Hz/cm
```



C. M. Gothard and J. S. Nowick

^{1}H NMR (500 MHz, 298 K, D_2O) spectrum of cyclotetramer parallelogram **10a** (aliphatic region)





cyclotetramer parallelogram **10a**
(*m*-Abc^{2K}-*o*-Abc^{2K})$_2$

R = $CH_2CH_2CH_2NH_2$TFA

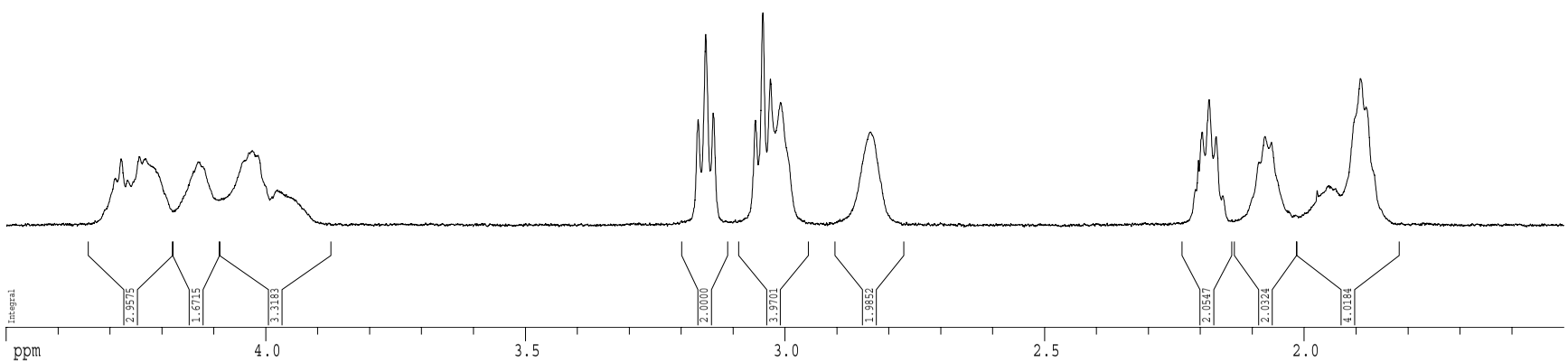

```
Current Data Parameters
USER              cgotha
NAME             cg-11-25
EXPNO                   2
PROCNO                  1

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
SWH             8012.820 Hz
FIDRES          0.098043 Hz
AQ              5.0998774 sec
RG                  10.1
DW                62.400 usec
DE                 6.00 usec
TE                298.0 K
D1           0.10000000 sec
MCREST       0.00000000 sec
MCWRK        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           500.2200096 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
F1P                4.500 ppm
F1              2250.99 Hz
F2P                1.500 ppm
F2               750.33 Hz
PPMCM           0.13158 ppm/cm
HZCM           65.81842 Hz/cm
```



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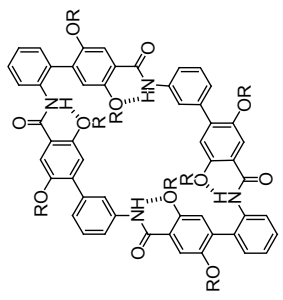
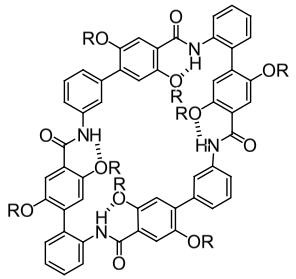
^{1}H NMR (500 MHz, 330 K, D_2O) spectrum of cyclotetramer parallelogram **10a**



cyclotetramer parallelogram **10a**
(*m*-Abc^{2K}-*o*-Abc^{2K})$_2$

R = $CH_2CH_2CH_2NH_2$TFA

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



cyclotetramer parallelogram **10a**
(*m*-Abc^{2K}-*o*-Abc^{2K})$_2$

R = $CH_2CH_2CH_2NH_2TFA$

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Current Data Parameters
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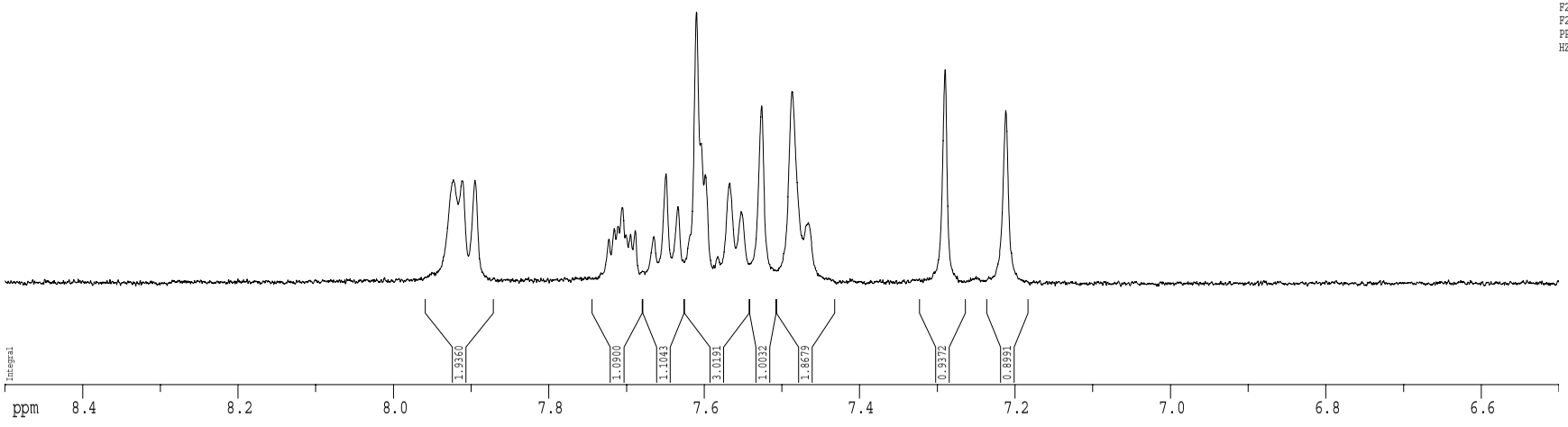
NAME         cg-11-25_GN
EXPNO                   3
PROCNO                  1

F2 - Acquisition Parameters
Date_          20070314
Time              22.37
INSTRUM           gn500
PROBHD   5 mm broadband
PULPROG            zg30
TD                81728
SOLVENT             D2O
NS                   64
DS                    2
SWH            8012.820 Hz
FIDRES         0.098043 Hz
AQ             5.0998774 sec
RG                1149.4
DW               62.400 usec
DE                6.00 usec
TE                330.0 K
D1            0.10000000 sec
MCREST        0.00000000 sec
MCWRK         0.01500000 sec

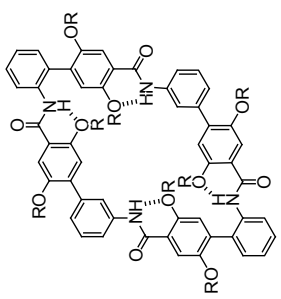
======== CHANNEL f1 ========
NUC1                 1H
P1                12.00 usec
PL1               -3.00 dB
SFO1         499.9334995 MHz

F2 - Processing parameters
SI                65536
SF          499.9301936 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
F1               4249.41 Hz
F2P               6.500 ppm
F2               3249.55 Hz
PPMCM          0.08772 ppm/cm
HZCM          43.85353 Hz/cm
```



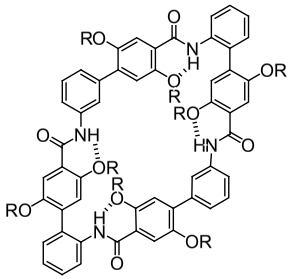
^{1}H NMR (500 MHz, 330 K, D_2O) spectrum of cyclotetramer parallelogram **10a** (aliphatic region)



cyclotetramer parallelogram **10a**
(*m*-Abc^{2K}-*o*-Abc^{2K})$_2$

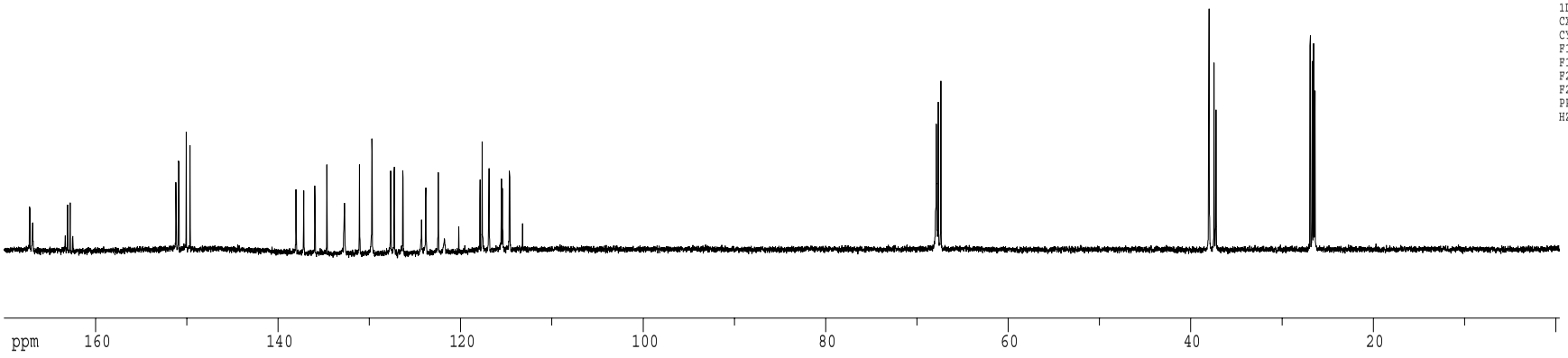
R = $CH_2CH_2CH_2NH_2TFA$

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



cyclotetramer parallelogram **10a**
(*m*-Abc^{2K}-*o*-Abc^{2K})$_2$

R = $CH_2CH_2CH_2NH_2TFA$

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Current Data Parameters
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NAME             cg-11-25
EXPNO                   9
PROCNO                  1

F2 - Acquisition Parameters
Date_            20070628
Time                19.03
INSTRUM          cryo500
PROBHD    5 mm CPTCI 1H-
PULPROG          zgdc30
TD                  65418
SOLVENT             D2O
NS                   8418
DS                      4
SWH             30303.031 Hz
FIDRES          0.463222 Hz
AQ              1.0794470 sec
RG                10321.3
DW                16.500 usec
DE                 6.00 usec
TE                320.0 K
D1             0.25000000 sec
d11            0.03000000 sec
MCREST         0.00000000 sec
MCWRK          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

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                   3.55 cm
F1P               169.978 ppm
F1              21379.91 Hz
F2P                -0.380 ppm
F2                 -47.75 Hz
PPMCM            7.47183 ppm/cm
HZCM           939.80994 Hz/cm
```

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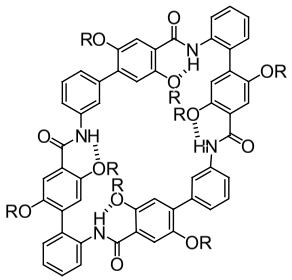
^{13}C NMR (125 MHz, 320 K, D_2O) spectrum of cyclotetramer parallelogram **10a** (aromatic region)



cyclotetramer parallelogram **10a**
(*m*-Abc^{2K}-*o*-Abc^{2K})$_2$

R = $CH_2CH_2CH_2NH_2TFA$


```
Current Data Parameters
USER                cgotha
NAME              cg-11-25
EXPNO                    9
PROCNO                   1

F2 - Acquisition Parameters
Date_            20070628
Time                19.03
INSTRUM           cryo500
PROBHD    5 mm CPTCI 1H-
PULPROG             zgdc30
TD                   65418
SOLVENT                D2O
NS                    8418
DS                       4
SWH              30303.031 Hz
FIDRES           0.463222 Hz
AQ               1.0794470 sec
RG                 10321.3
DW                   16.500 usec
DE                    6.00 usec
TE                   320.0 K
D1               0.25000000 sec
d11              0.03000000 sec
MCREST           0.00000000 sec
MCWRK            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

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                     3.55 cm
F1P                 168.694 ppm
F1                21218.44 Hz
F2P                 111.758 ppm
F2                14057.00 Hz
PPMCM              2.49719 ppm/cm
HZCM             314.09793 Hz/cm
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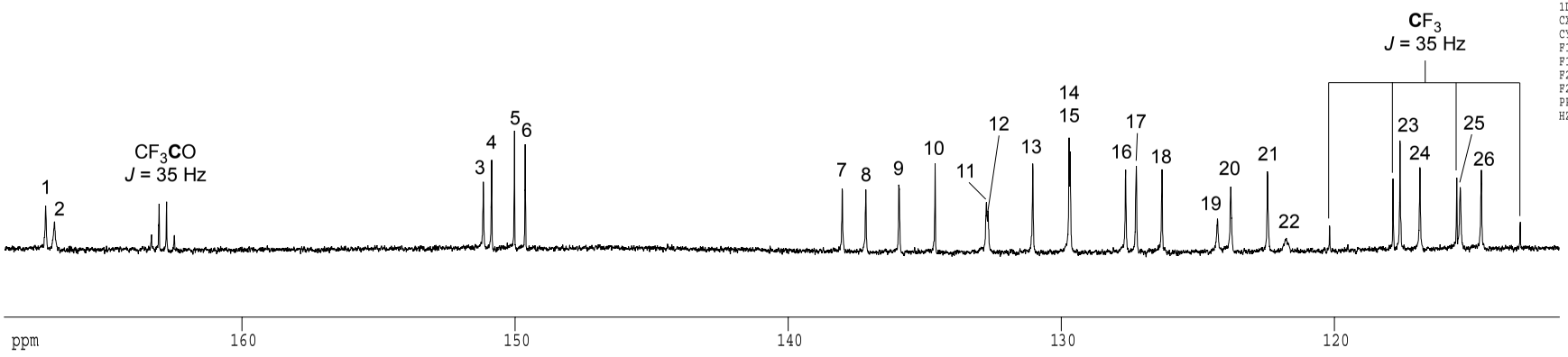
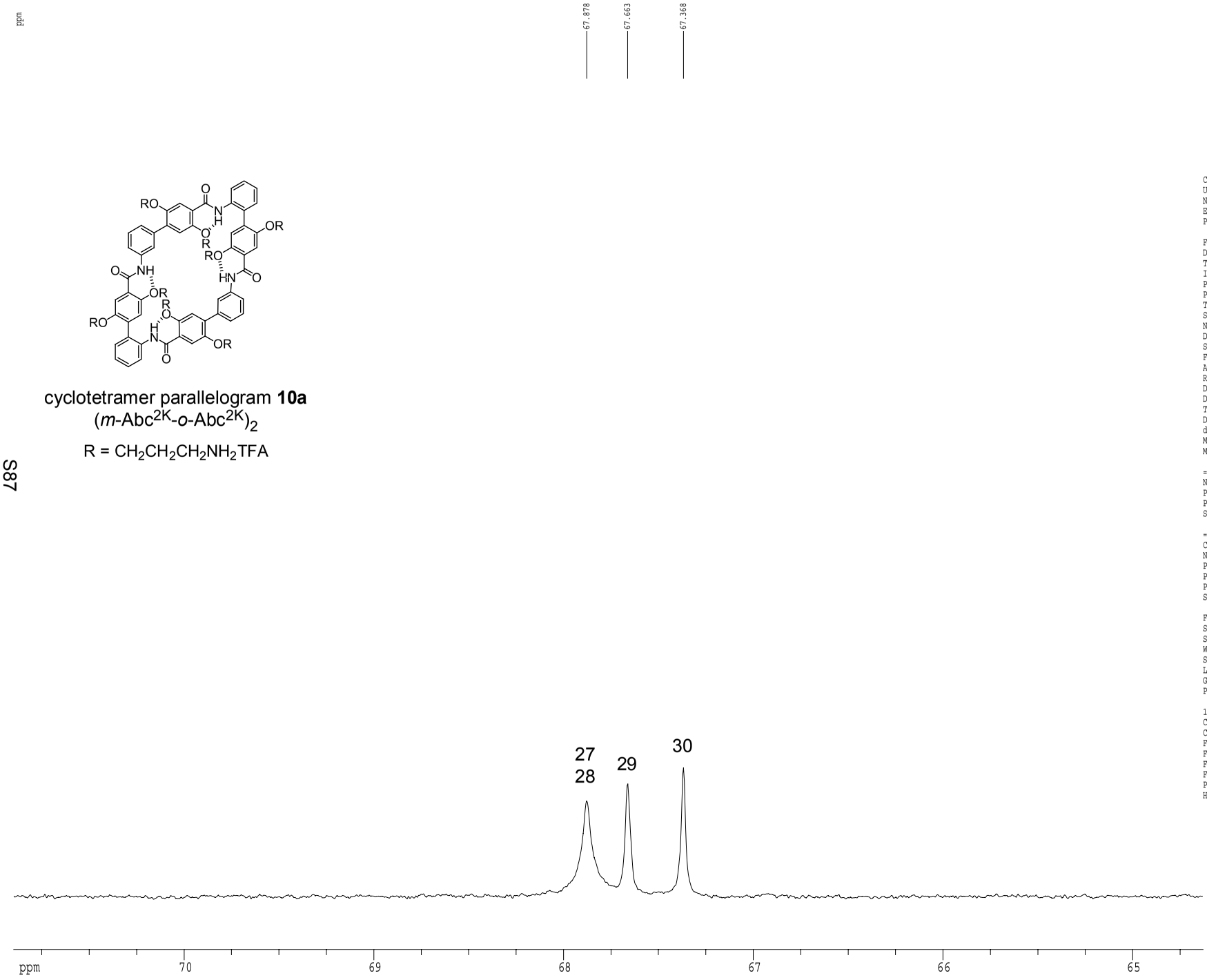


C. M. Gothard and J. S. Nowick

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

cyclotetramer parallelogram **10a**
(*m*-Abc^{2K}-*o*-Abc^{2K})$_2$

R = $CH_2CH_2CH_2NH_2$TFA




```
Current Data Parameters
USER                cgotha
NAME             cg-11-25
EXPNO                   9
PROCNO                  1

F2 - Acquisition Parameters
Date_            20070628
Time                19.03
INSTRUM          cryo500
PROBHD   5 mm CPTCI 1H-
PULPROG           zgdc30
TD                 65418
SOLVENT              D2O
NS                    8418
DS                       4
SWH             30303.031 Hz
FIDRES          0.463222 Hz
AQ              1.0794470 sec
RG                10321.3
DW                  16.500 usec
DE                   6.00 usec
TE                 320.0 K
D1              0.25000000 sec
d11             0.03000000 sec
MCREST          0.00000000 sec
MCWRK           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

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                   3.55 cm
F1P                70.898 ppm
F1                8917.63 Hz
F2P                64.629 ppm
F2                8129.10 Hz
PPMCM             0.27496 ppm/cm
HZCM             34.58467 Hz/cm
```

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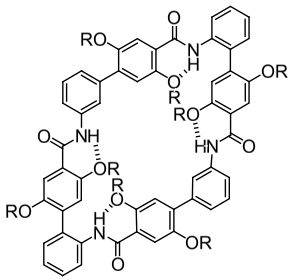
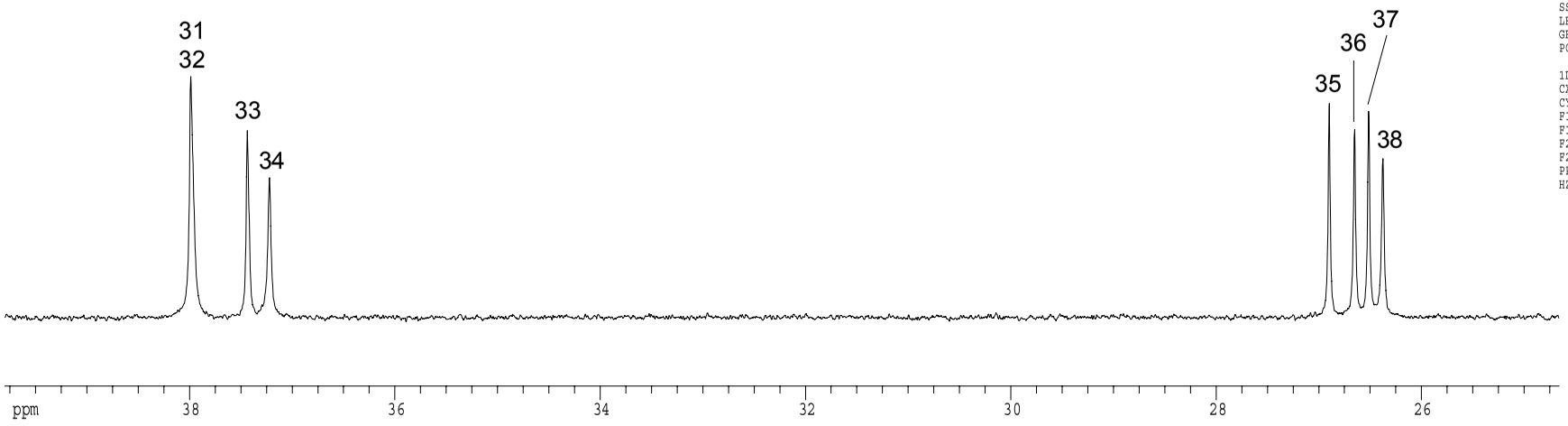
^{13}C NMR (125 MHz, 320 K, D_2O) spectrum of cyclotetramer parallelogram **10a** (side chain region)



cyclotetramer parallelogram **10a**
(*m*-Abc^{2K}-*o*-Abc^{2K})$_2$

R = $CH_2CH_2CH_2NH_2$TFA




```
Current Data Parameters
USER              cgotha
NAME             cg-11-25
EXPNO                   9
PROCNO                  1

F2 - Acquisition Parameters
Date_           20070628
Time              19.03
INSTRUM         cryo500
PROBHD   5 mm CPTCI 1H-
PULPROG          zgdc30
TD                65418
SOLVENT             D2O
NS                 8418
DS                    4
SWH           30303.031 Hz
FIDRES         0.463222 Hz
AQ             1.0794470 sec
RG              10321.3
DW                16.500 usec
DE                 6.00 usec
TE                320.0 K
D1             0.25000000 sec
d11            0.03000000 sec
MCREST         0.00000000 sec
MCWRK          0.01500000 sec

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

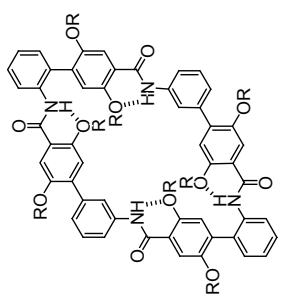
======== CHANNEL f2 ========
CPDPRG2          waltz16
NUC2                 1H
PCPD2             100.00 usec
PL2                 1.60 dB
PL12               23.54 dB
SFO2         500.2225011 MHz

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                   3.55 cm
F1P                39.797 ppm
F1                5005.71 Hz
F2P                24.660 ppm
F2                3101.73 Hz
PPMCM             0.66392 ppm/cm
HZCM             83.50816 Hz/cm
```

C. M. Gothard and J. S. Nowick

^{1}H NMR (500 MHz, 298 K, CD_3OD) spectrum of cyclotetramer parallelogram **10a**

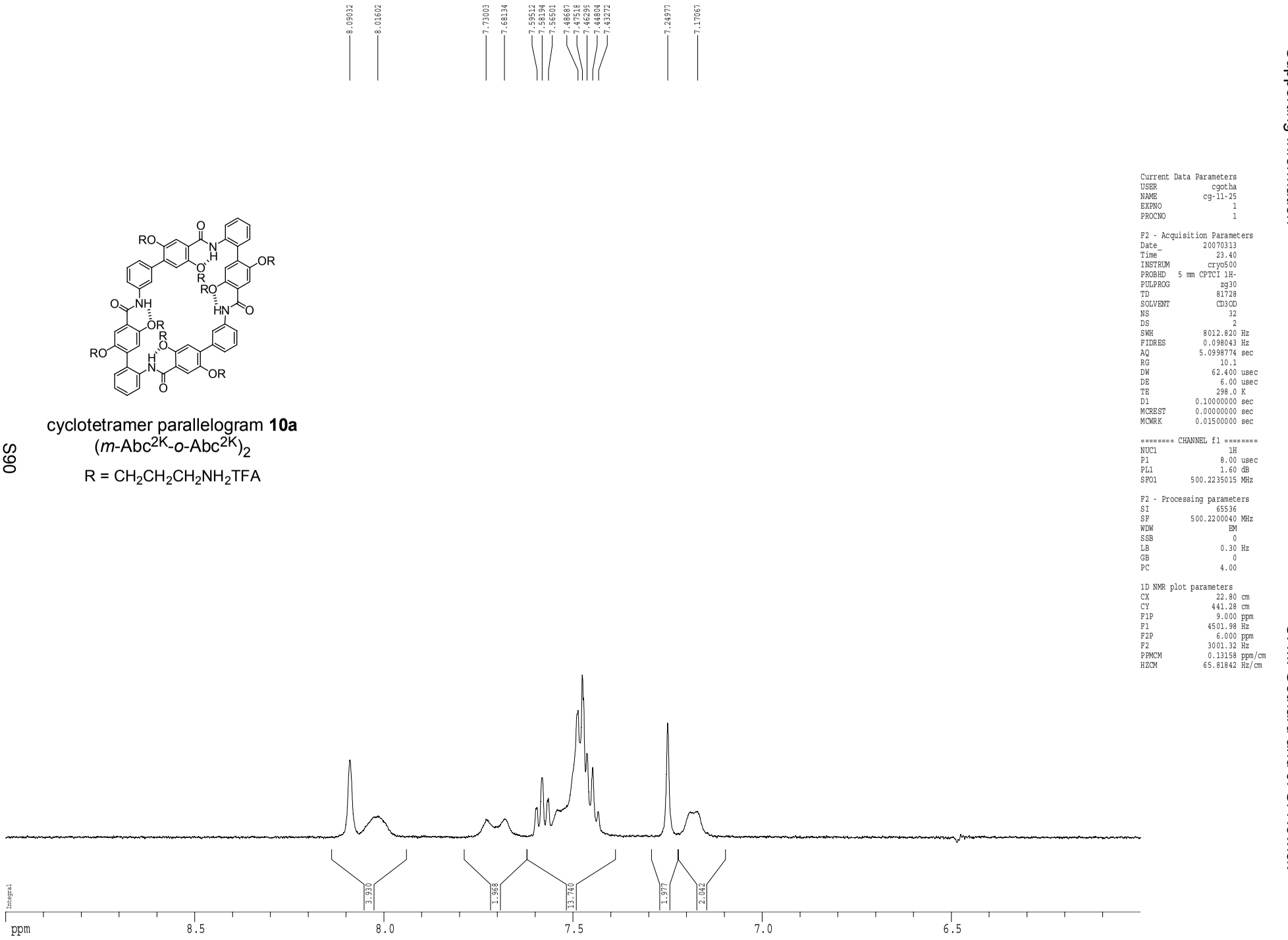


cyclotetramer parallelogram **10a**
(*m*-Abc^{2K}-*o*-Abc^{2K})$_2$
R = $CH_2CH_2CH_2NH_2$TFA

^{1}H NMR (500 MHz, 298 K, CD_3OD) spectrum of cyclotetramer parallelogram **10a**

cyclotetramer parallelogram **10a**
(*m*-Abc^{2K}-*o*-Abc^{2K})$_2$

R = $CH_2CH_2CH_2NH_2TFA$

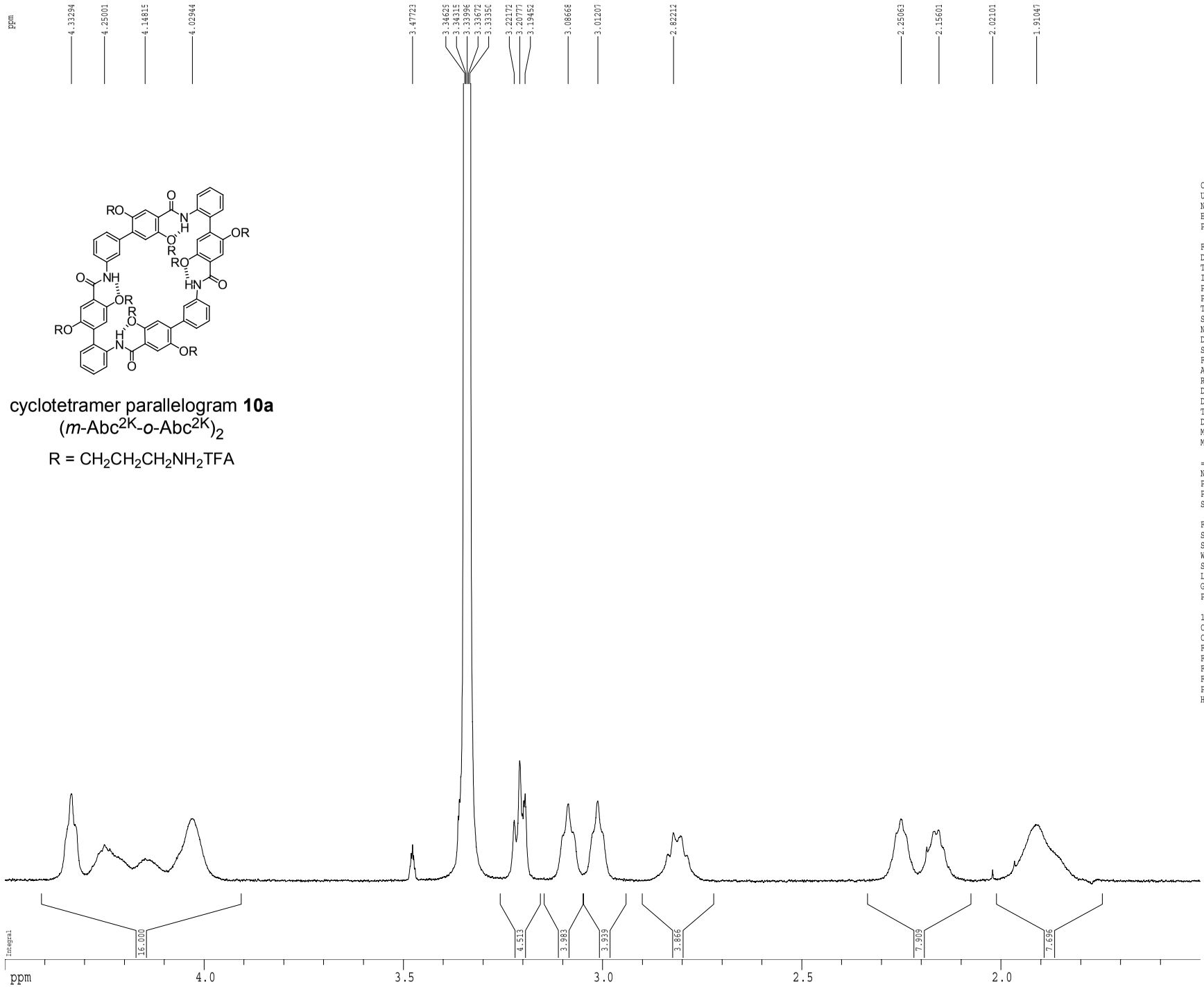


C. M. Gothard and J. S. Nowick

^{1}H NMR (500 MHz, 298 K, CD_3OD) spectrum of cyclotetramer parallelogram **10a**

cyclotetramer parallelogram **10a**
(*m*-Abc^{2K}-*o*-Abc^{2K})$_2$

R = $CH_2CH_2CH_2NH_2TFA$

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NAME              cg-11-25
EXPNO                    1
PROCNO                   1

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Time                 23.40
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PULPROG                zg30
TD                    81728
SOLVENT               CD3OD
NS                       32
DS                        2
SWH               8012.820 Hz
FIDRES               0.098043 Hz
AQ                5.0998774 sec
RG                       10.1
DW                   62.400 usec
DE                    6.00 usec
TE                   298.0 K
D1              0.10000000 sec
MCREST          0.00000000 sec
MCWRK           0.01500000 sec

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

F2 - Processing parameters
SI                       65536
SP             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                  441.28 cm
F1P                   4.500 ppm
F1                 2250.99 Hz
F2P                   1.500 ppm
F2                  750.33 Hz
PPMCM              0.13158 ppm/cm
HZCM              65.81842 Hz/cm
```


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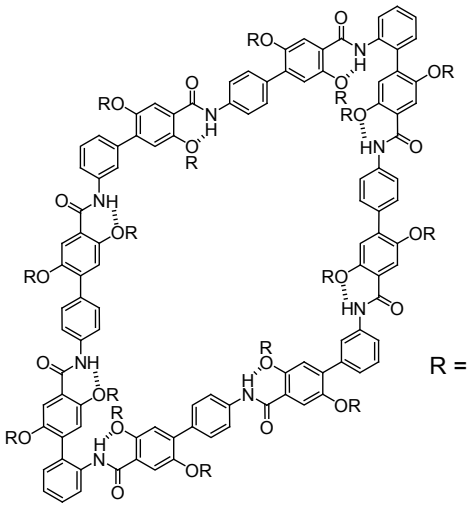
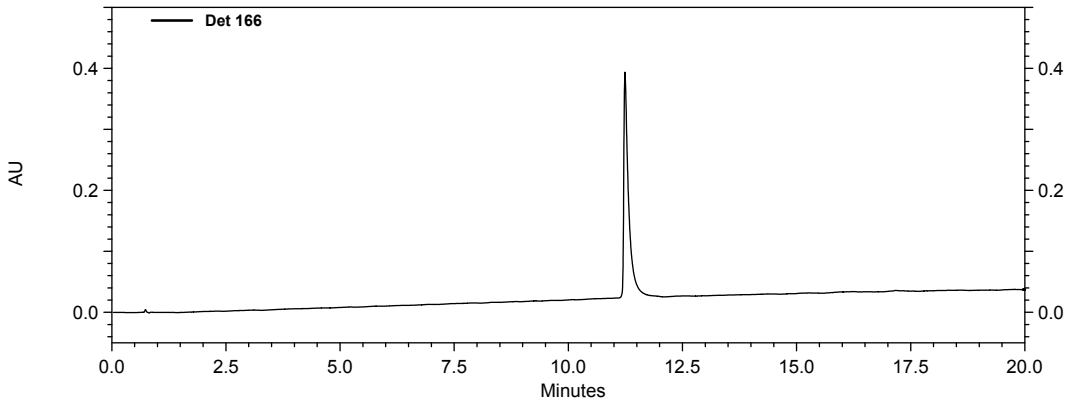
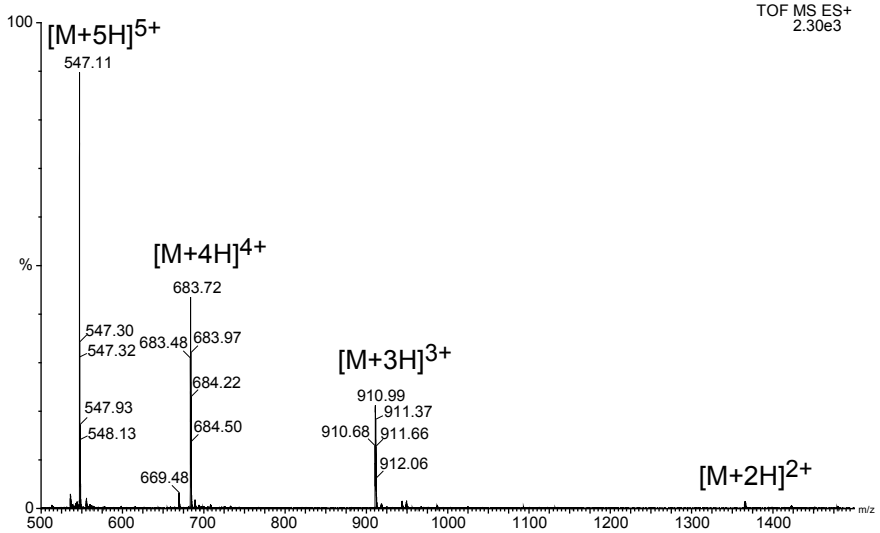
cyclooctamer: (*o*-Abc^{2K}-*p*-Abc^{2K}-*m*-Abc^{2K}-*p*-Abc^{2K})$_2$ parallelogram (**10b**) Analytical RP-HPLC chromatograph and mass spectrum (ESI-MS)



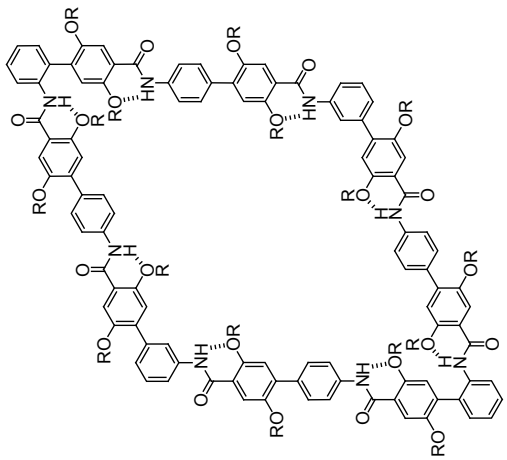
(a) Analytical RP-HPLC (5-50% acetonitrile with 0.1% TFA over 20 min, λ = 214)



(b) ESI Mass spectrum. (Calcd exact mass for $C_{152}H_{184}N_{24}O_{24}$ [M] = 2729.39)

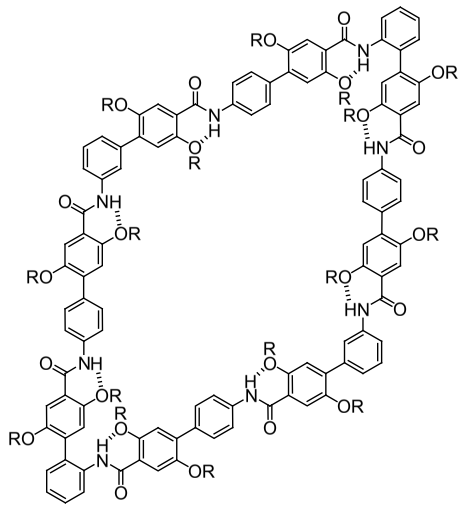


^{1}H NMR (500 MHz, 298 K, D_2O) spectrum of cyclooctamer parallelogram **10b**



cyclooctamer parallelogram **10b**
(*o*-Abc^{2K}-*p*-Abc^{2K}-*m*-Abc^{2K}-*p*-Abc^{2K})$_2$
R = $CH_2CH_2CH_2NH_2$TFA

^{1}H NMR (500 MHz, 298 K, D_2O) spectrum of cyclooctamer parallelogram **10b** (aromatic region)



cyclooctamer parallelogram **10b**
(*o*-Abc^{2K}-*p*-Abc^{2K}-*m*-Abc^{2K}-*p*-Abc^{2K})$_2$

R = $CH_2CH_2CH_2NH_2TFA$

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Current Data Parameters
USER                cgotha
NAME             cg-11-37
EXPNO                   1
PROCNO                  1

F2 - Acquisition Parameters
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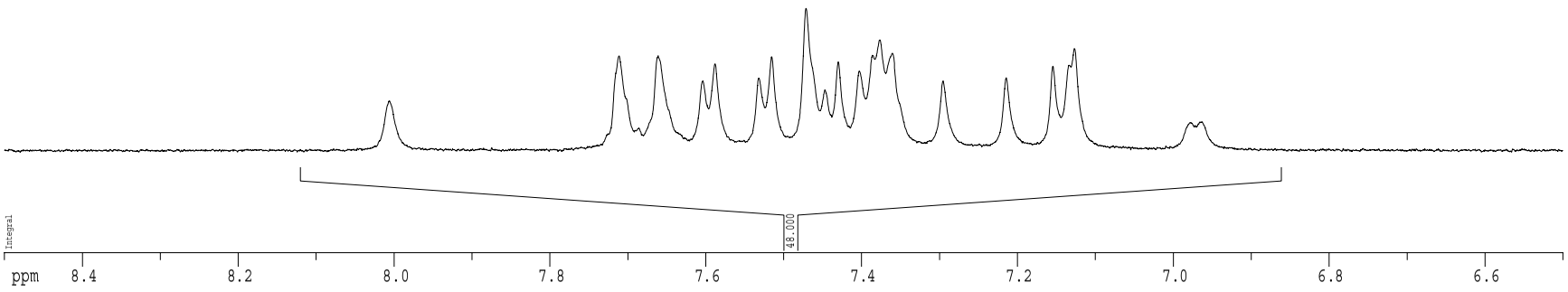
Time                23.43
INSTRUM             gn500
PROBHD   5 mm broadband
PULPROG              zg30
TD                  81728
SOLVENT               D2O
NS                     16
DS                      2
SWH              8012.820 Hz
FIDRES           0.098043 Hz
AQ               5.0998774 sec
RG                    512
DW                 62.400 usec
DE                  6.00 usec
TE                 298.0 K
D1              0.10000000 sec
MCREST          0.00000000 sec
MCWRK           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            499.9300032 MHz
WDW                     EM
SSB                      0
LB                    0.30 Hz
GB                       0
PC                    4.00

1D NMR plot parameters
CX                   22.80 cm
CY                  157.94 cm
F1P                  8.500 ppm
F1                4249.40 Hz
F2P                  6.500 ppm
F2                3249.54 Hz
PPMCM             0.08772 ppm/cm
HZCM             43.85351 Hz/cm
```



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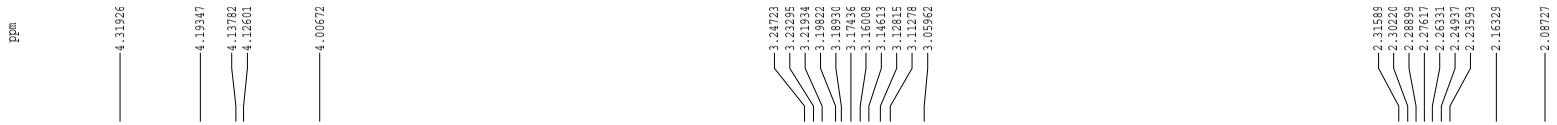
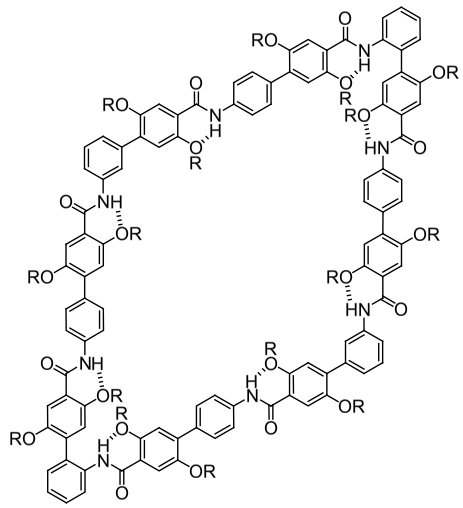
^{1}H NMR (500 MHz, 298 K, D_2O) spectrum of cyclooctamer parallelogram **10b** (aliphatic region)





cyclooctamer parallelogram **10b**
(*o*-Abc^{2K}-*p*-Abc^{2K}-*m*-Abc^{2K}-*p*-Abc^{2K})$_2$

R = $CH_2CH_2CH_2NH_2TFA$

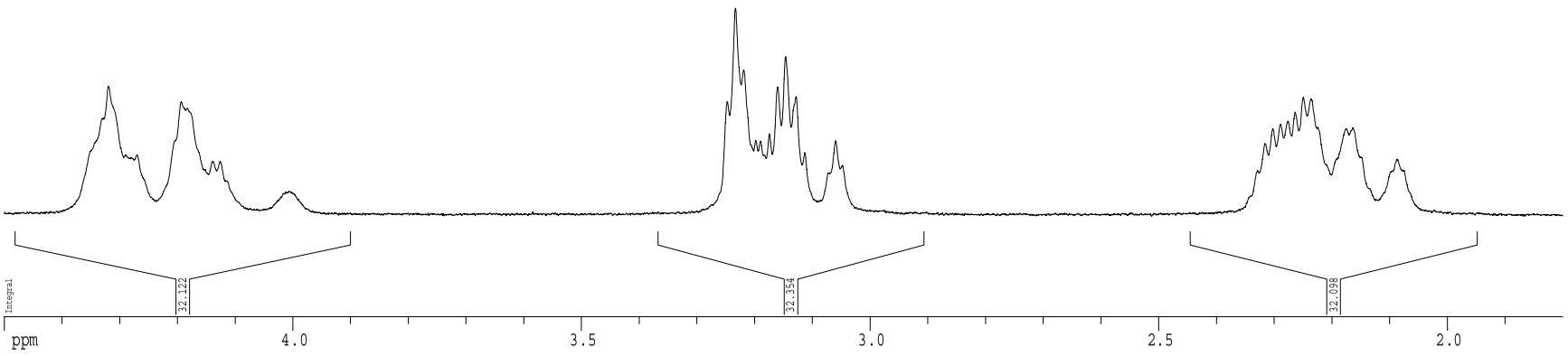

```
Current Data Parameters
USER                cgotha
NAME             cg-11-37
EXPNO                   1
PROCNO                  1

F2 - Acquisition Parameters
Date_            20070505
Time                23.43
INSTRUM             gn500
PROBHD   5 mm broadband
PULPROG              zg30
TD                  81728
SOLVENT               D2O
NS                     16
DS                      2
SWH              8012.820 Hz
FIDRES           0.098043 Hz
AQ               5.0998774 sec
RG                    512
DW                62.400 usec
DE                 6.00 usec
TE                298.0 K
D1             0.10000000 sec
MCREST         0.00000000 sec
MCWRK          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           499.9300032 MHz
WDW                     EM
SSB                      0
LB                  0.30 Hz
GB                       0
PC                   4.00

1D NMR plot parameters
CX                  22.80 cm
CY                 157.94 cm
F1P                 4.500 ppm
F1                2249.69 Hz
F2P                 1.800 ppm
F2                 899.87 Hz
PPMCM            0.11842 ppm/cm
HZCM            59.20224 Hz/cm
```



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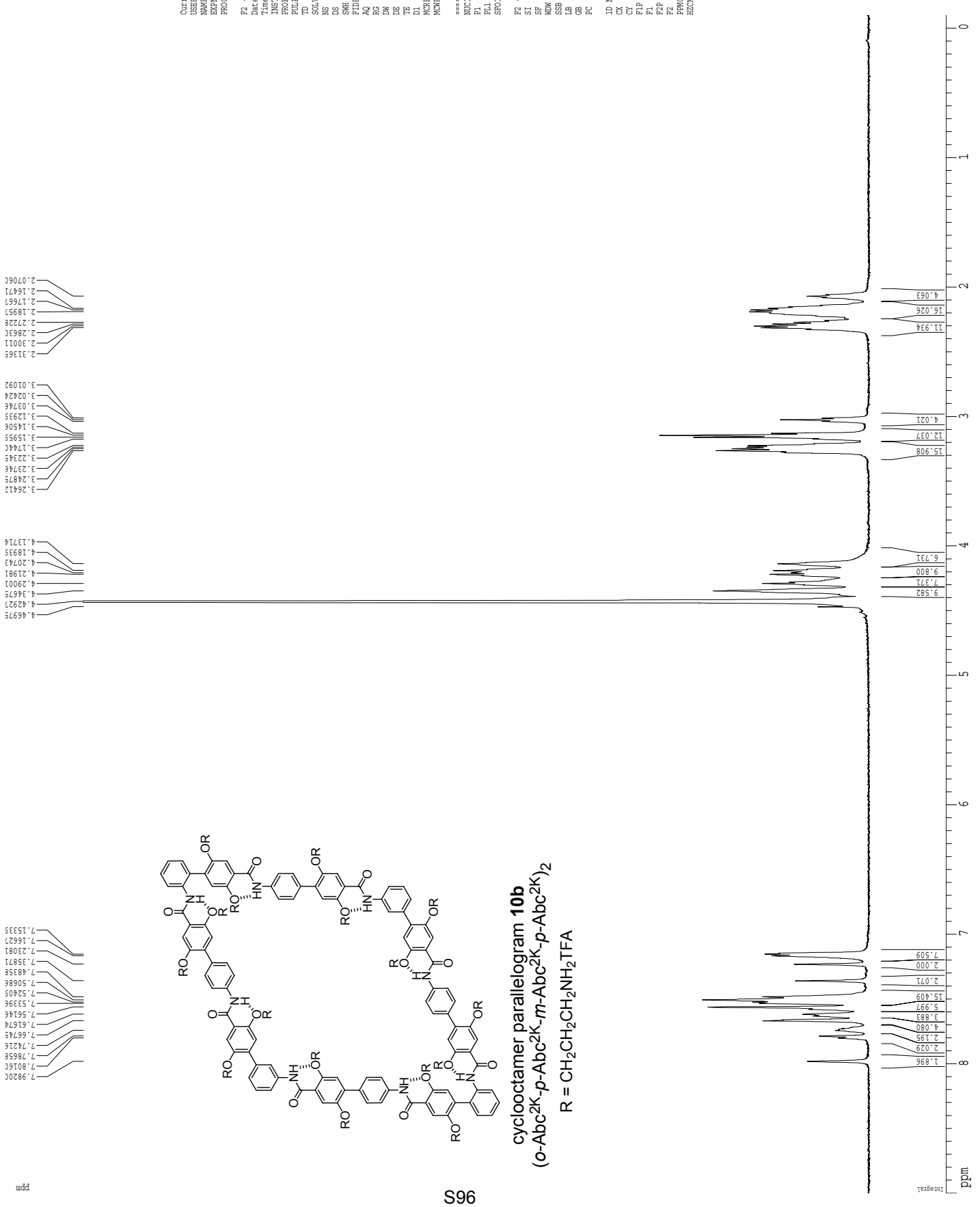
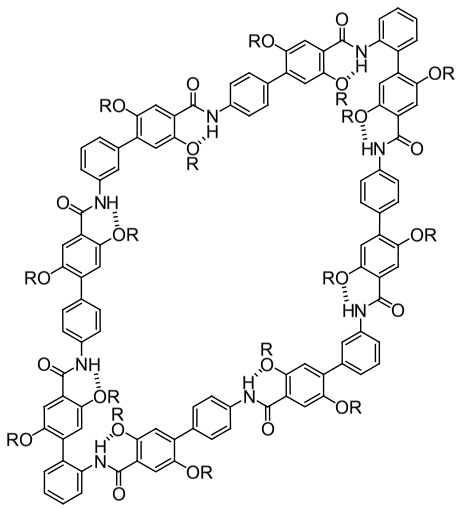
^{1}H NMR (500 MHz, 330 K, D_2O) spectrum of cyclooctamer parallelogram **10b**



^{1}H NMR (500 MHz, 330 K, D_2O) spectrum of cyclooctamer parallelogram **10b** (aromatic region)



cyclooctamer parallelogram **10b**
$(o\text{-Abc}^{2K}\text{-}p\text{-Abc}^{2K}\text{-}m\text{-Abc}^{2K}\text{-}p\text{-Abc}^{2K})_2$
R = $CH_2CH_2CH_2NH_2TFA$

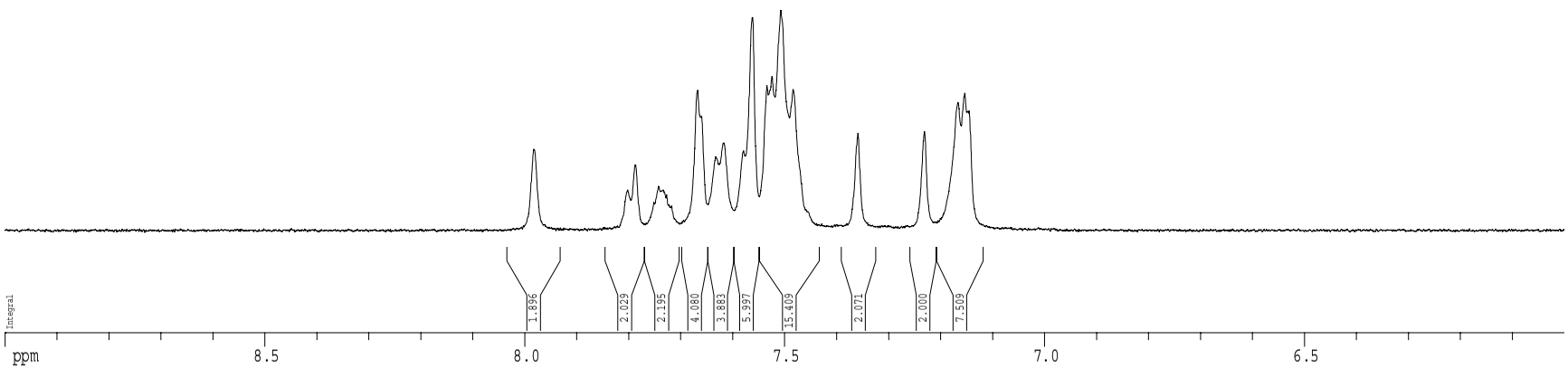

```
Current Data Parameters
USER              cgotha
NAME             cg-11-37
EXPNO                   4
PROCNO                  1

F2 - Acquisition Parameters
Date_           20070506
Time                0.15
INSTRUM            gn500
PROBHD   5 mm broadband
PULPROG             zg30
TD                 81728
SOLVENT              D2O
NS                    32
DS                     2
SWH            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
D1           0.10000000 sec
MCREST       0.00000000 sec
MCWRK        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
F1P               9.000 ppm
F1             4499.37 Hz
F2P               6.000 ppm
F2             2999.58 Hz
PPMCM          0.13158 ppm/cm
HZCM          65.78029 Hz/cm
```



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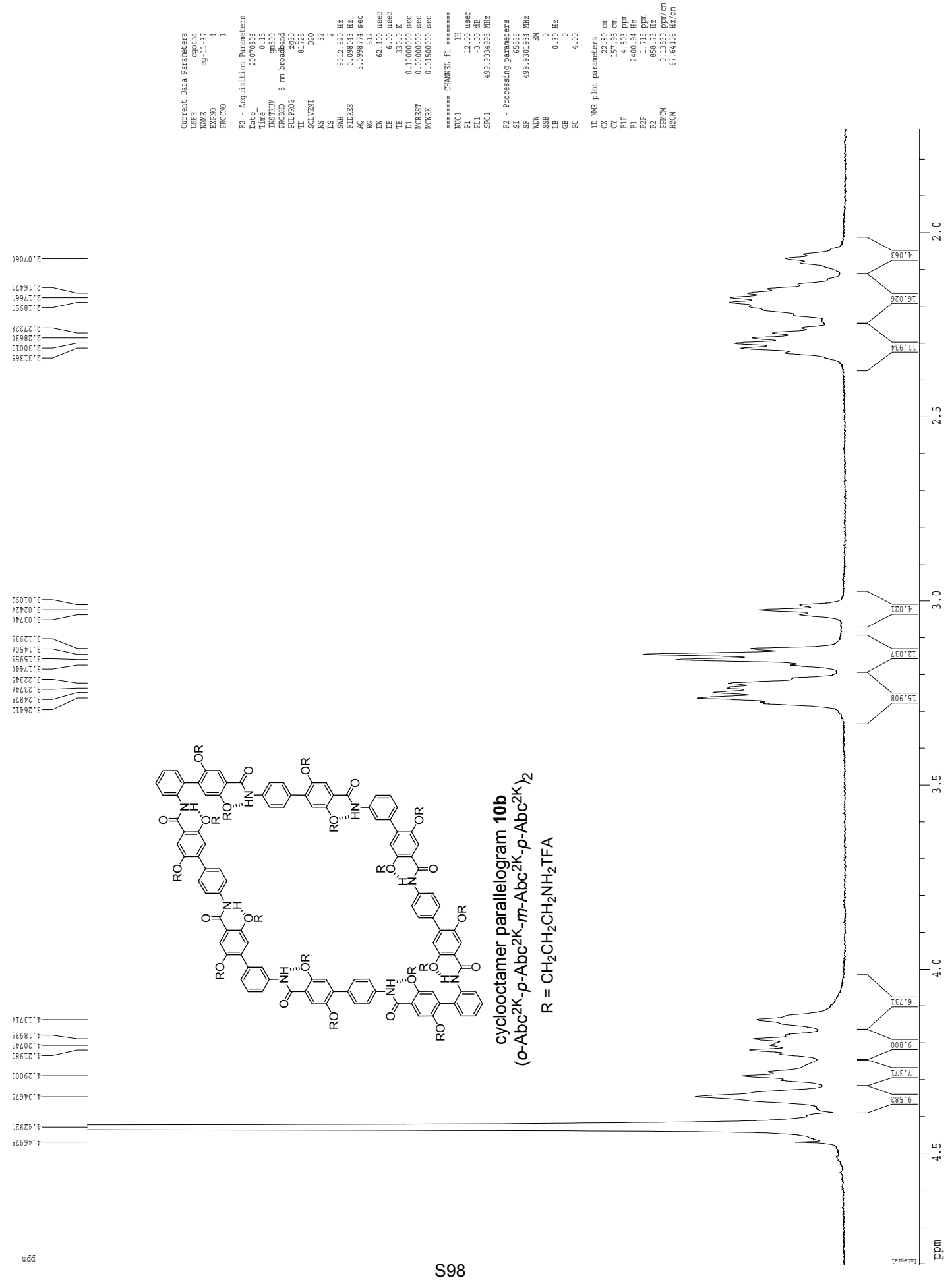
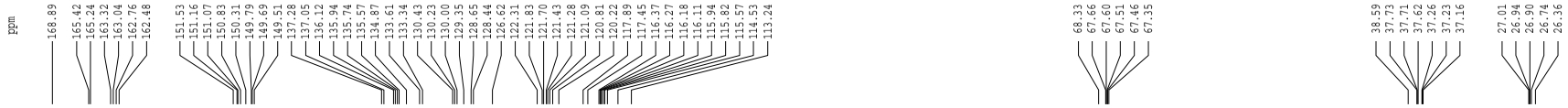
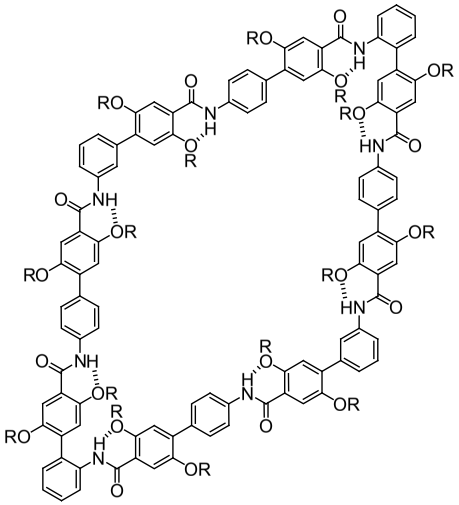
^{1}H NMR (500 MHz, 330 K, D_2O) spectrum of cyclooctamer parallelogram **10b** (aliphatic region)



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





cyclooctamer parallelogram **10b**
$(o\text{-Abc}^{2K}\text{-}p\text{-Abc}^{2K}\text{-}m\text{-Abc}^{2K}\text{-}p\text{-Abc}^{2K})_2$
R = $CH_2CH_2CH_2NH_2TFA$


```
Current Data Parameters
USER                cgotha
NAME             cg-11-37
EXPNO                  12
PROCNO                  1

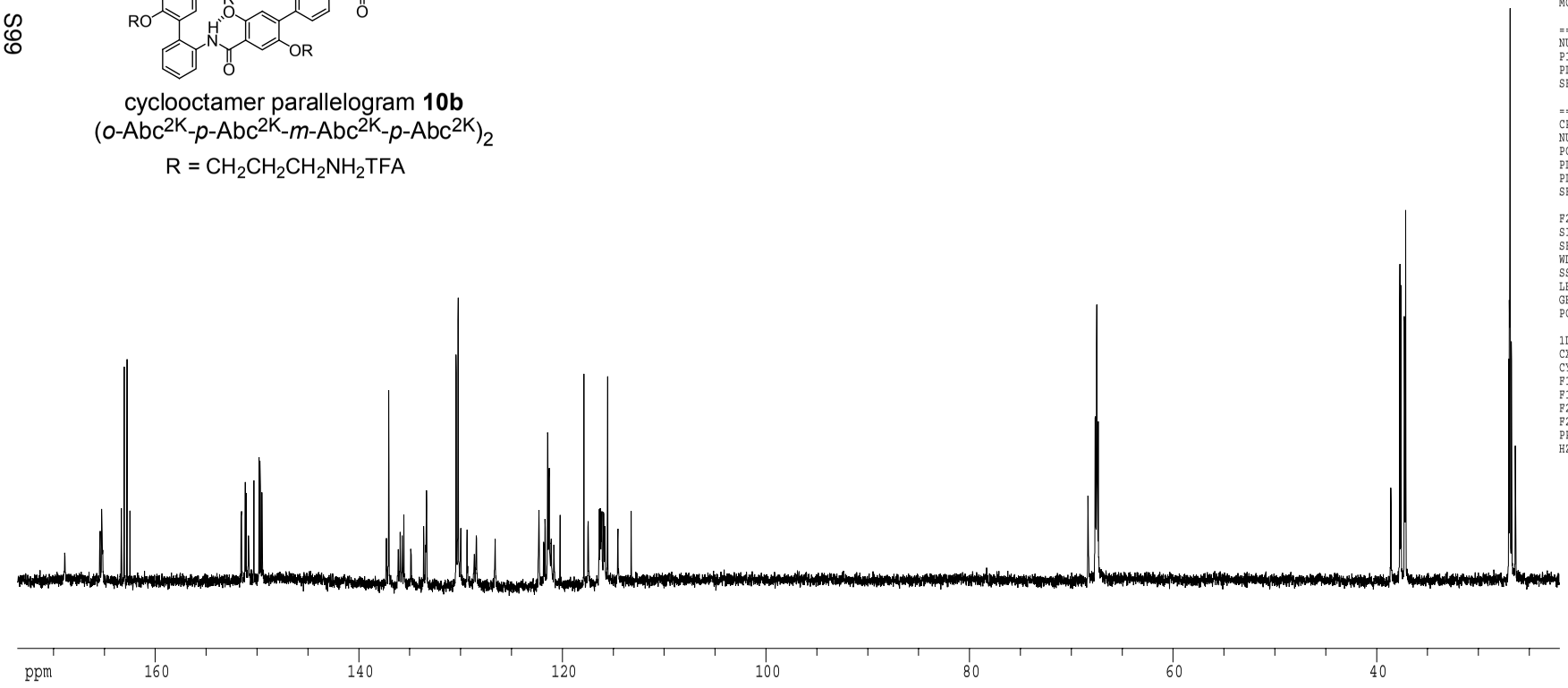
F2 - Acquisition Parameters
Date_            20070517
Time                1.34
INSTRUM          cryo500
PROBHD    5 mm CPTCI 1H-
PULPROG          zgdc30
TD                 65418
SOLVENT              D2O
NS                   14000
DS                       4
SWH            30303.031 Hz
FIDRES          0.463222 Hz
AQ              1.0794470 sec
RG                14596.5
DW                16.500 usec
DE                 6.00 usec
TE                320.0 K
D1             0.25000000 sec
d11            0.03000000 sec
MCREST         0.00000000 sec
MCWRK          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

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                   8.47 cm
F1P                173.506 ppm
F1                21823.68 Hz
F2P                 22.049 ppm
F2                 2773.34 Hz
PPMCM             6.64286 ppm/cm
HZCM            835.54108 Hz/cm
```



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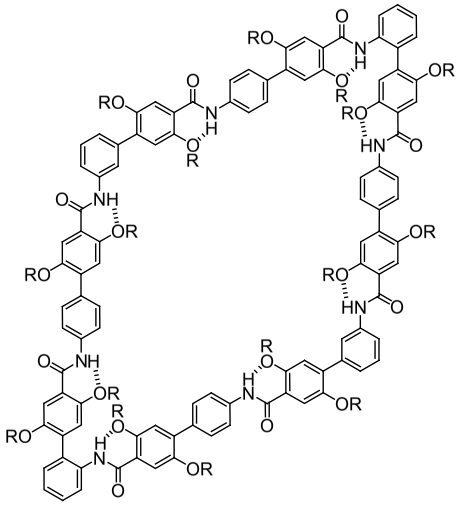
^{13}C NMR (125 MHz, 320 K, D_2O) spectrum of cyclooctamer parallelogram **10b** (aromatic region)



cyclooctamer parallelogram **10b**
(*o*-Abc^{2K}-*p*-Abc^{2K}-*m*-Abc^{2K}-*p*-Abc^{2K})$_2$

R = $CH_2CH_2CH_2NH_2TFA$

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```
Current Data Parameters
USER                cgotha
NAME              cg-11-37
EXPNO                   12
PROCNO                   1

F2 - Acquisition Parameters
Date_            20070517
Time                 1.34
INSTRUM           cryo500
PROBHD    5 mm CPTCI 1H-
PULPROG           zgdc30
TD                  65418
SOLVENT               D2O
NS                    14000
DS                        4
SWH             30303.031 Hz
FIDRES           0.463222 Hz
AQ               1.0794470 sec
RG                 14596.5
DW                   16.500 usec
DE                    6.00 usec
TE                   320.0 K
D1              0.25000000 sec
d11             0.03000000 sec
MCREST          0.00000000 sec
MCWRK           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

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                     8.47 cm
F1P                  170.070 ppm
F1                 21391.42 Hz
F2P                  112.177 ppm
F2                 14109.63 Hz
PPMCM                2.53916 ppm/cm
HZCM               319.37680 Hz/cm
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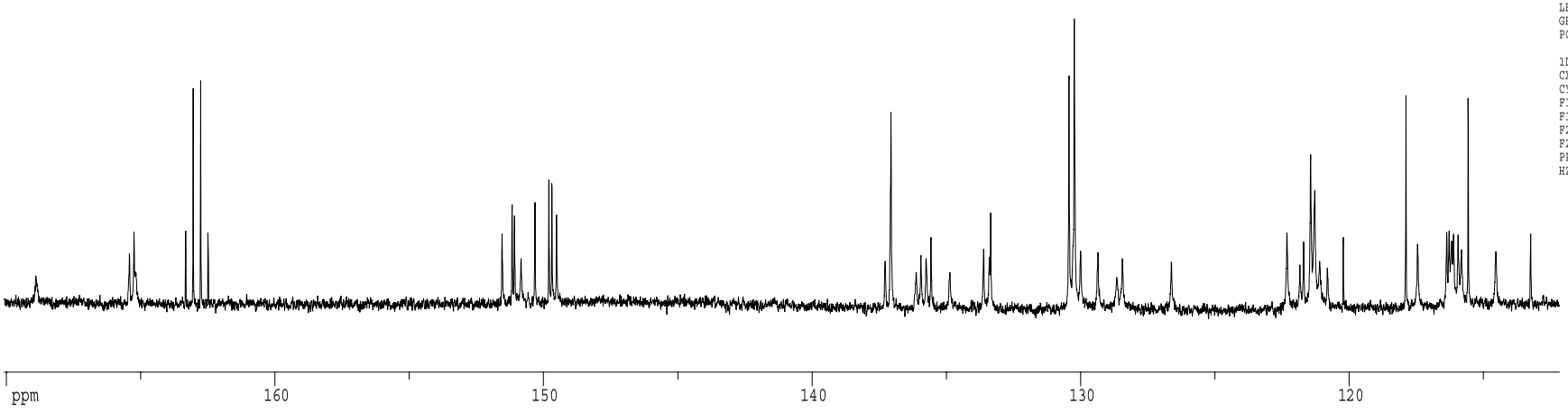
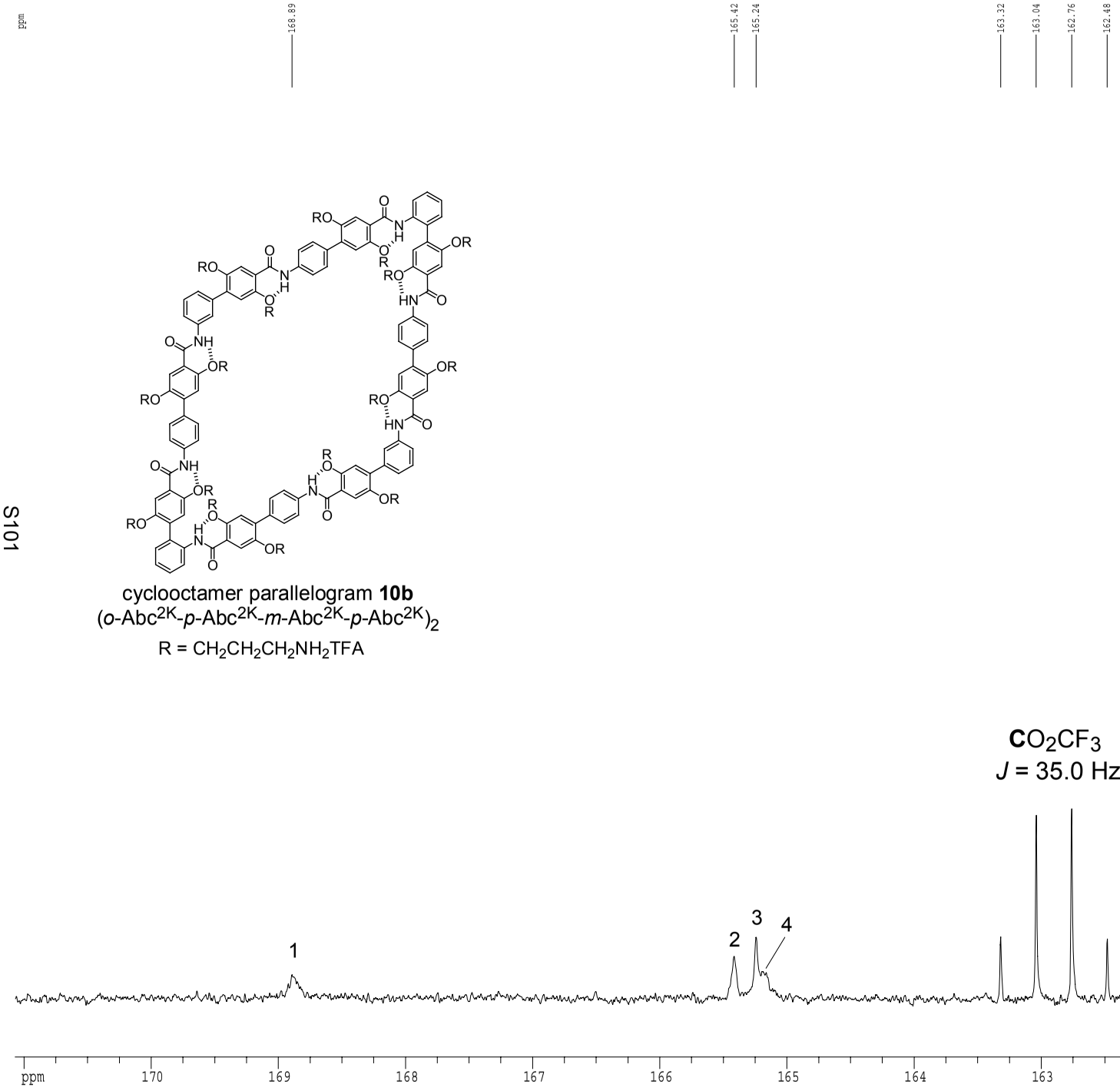


^{13}C NMR (125 MHz, 320 K, D_2O) spectrum of cyclooctamer parallelogram **10b** (carbonyl region)



cyclooctamer parallelogram **10b**
(*o*-Abc^{2K}-*p*-Abc^{2K}-*m*-Abc^{2K}-*p*-Abc^{2K})$_2$

R = $CH_2CH_2CH_2NH_2TFA$

Current Data Parameters
USER cgotha
NAME cg-11-37
EXPNO 12
PROCNO 1

F2 - Acquisition Parameters
Date_ 20070517
Time 1.34
INSTRUM cryo500
PROBHD 5 mm CPTCI 1H-
PULPROG zgdc30
TD 65418
SOLVENT D2O
NS 14000
DS 4
SWH 30303.031 Hz
FIDRES 0.463222 Hz
AQ 1.0794470 sec
RG 14596.5
DW 16.500 usec
DE 6.00 usec
TE 320.0 K
D1 0.25000000 sec
d11 0.03000000 sec
MCREST 0.00000000 sec
MCWRK 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

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 8.47 cm
F1P 171.069 ppm
F1 21517.09 Hz
F2P 160.657 ppm
F2 20207.47 Hz
PPMCM 0.45667 ppm/cm
HZCM 57.43966 Hz/cm

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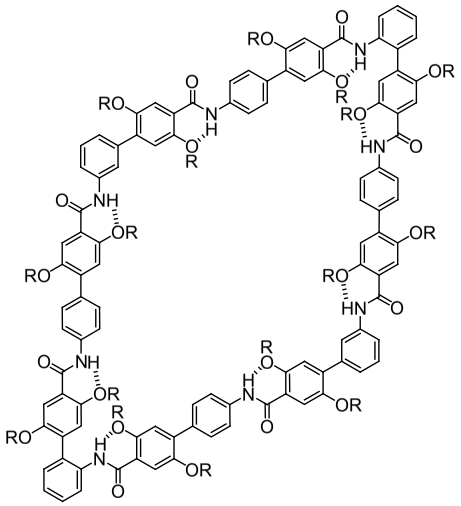
^{13}C NMR (125 MHz, 320 K, D_2O) spectrum of cyclooctamer parallelogram **10b** (aromatic region expansion 1)



cyclooctamer parallelogram **10b**
(*o*-Abc^{2K}-*p*-Abc^{2K}-*m*-Abc^{2K}-*p*-Abc^{2K})$_2$

R = $CH_2CH_2CH_2NH_2TFA$

```
Current Data Parameters
USER                cgotha
NAME             cg-11-37
EXPNO                  12
PROCNO                  1

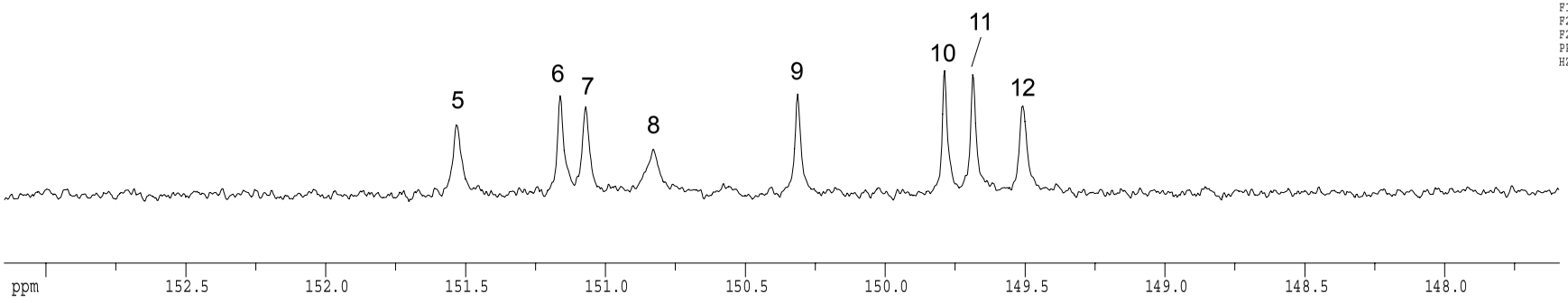
F2 - Acquisition Parameters
Date_            20070517
Time                1.34
INSTRUM          cryo500
PROBHD    5 mm CPTCI 1H-
PULPROG           zgdc30
TD                 65418
SOLVENT              D2O
NS                   14000
DS                       4
SWH             30303.031 Hz
FIDRES          0.463222 Hz
AQ              1.0794470 sec
RG                14596.5
DW                 16.500 usec
DE                  6.00 usec
TE                 320.0 K
D1              0.25000000 sec
d11             0.03000000 sec
MCREST          0.00000000 sec
MCWRK           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

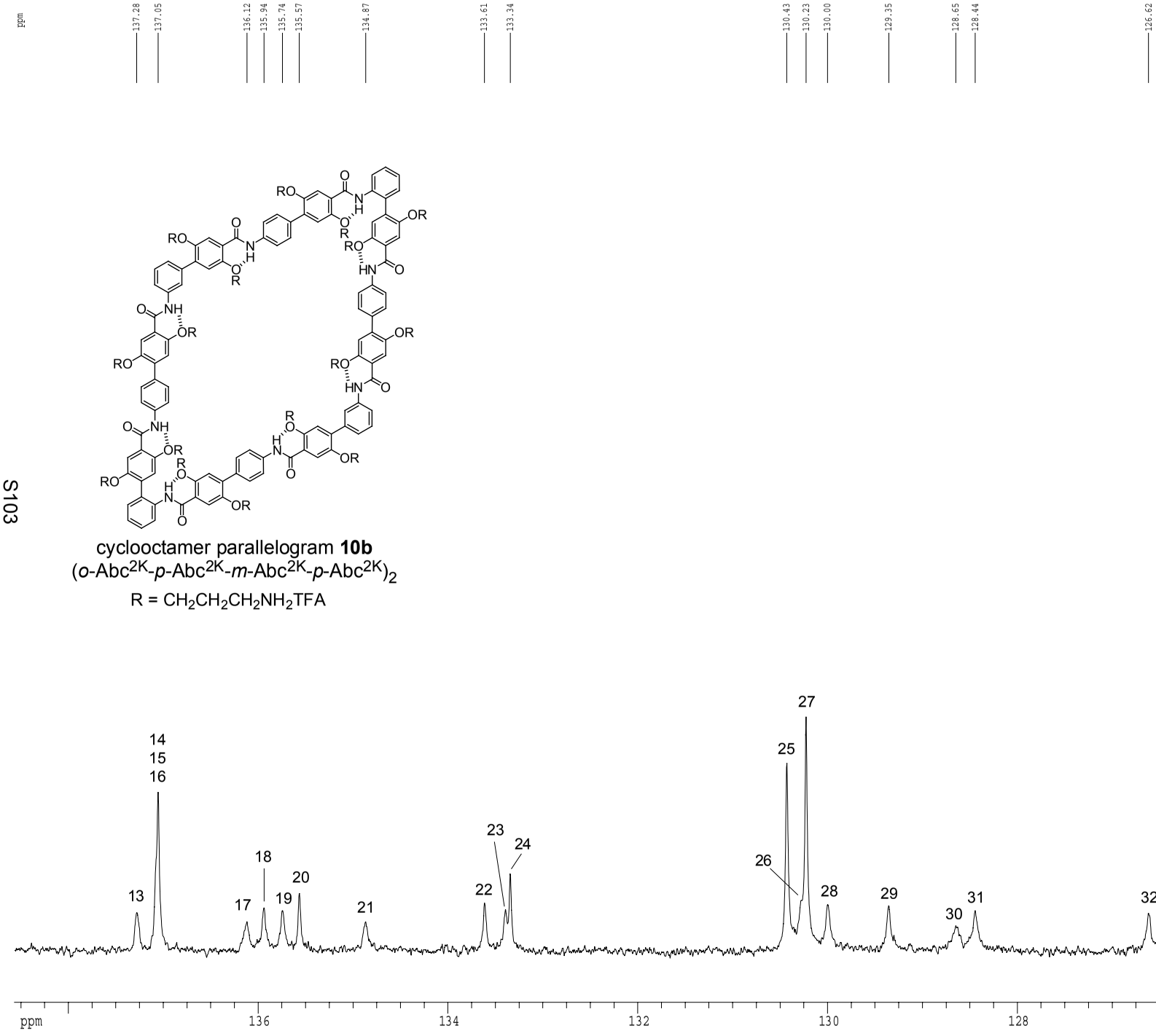
F2 - Processing parameters
SI                   65536
SF            125.7804190 MHz
WDW                    EM
SSB                     0
LB                   1.00 Hz
GB                      0
PC                   2.00

1D NMR plot parameters
CX                  22.80 cm
CY                   8.47 cm
F1P                153.148 ppm
F1                19263.04 Hz
F2P                147.590 ppm
F2                18563.96 Hz
PPMCM             0.24377 ppm/cm
HZCM              30.66156 Hz/cm
```



C. M. Gothard and J. S. Nowick

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



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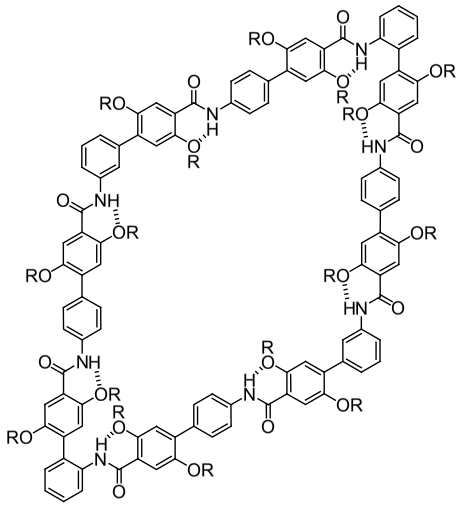
^{13}C NMR (125 MHz, 320 K, D_2O) spectrum of cyclooctamer parallelogram **10b** (aromatic region expansion 3)



cyclooctamer parallelogram **10b**
(*o*-Abc^{2K}-*p*-Abc^{2K}-*m*-Abc^{2K}-*p*-Abc^{2K})$_2$
R = $CH_2CH_2CH_2NH_2TFA$


```
Current Data Parameters
USER                cgotha
NAME              cg-11-37
EXPNO                   12
PROCNO                   1

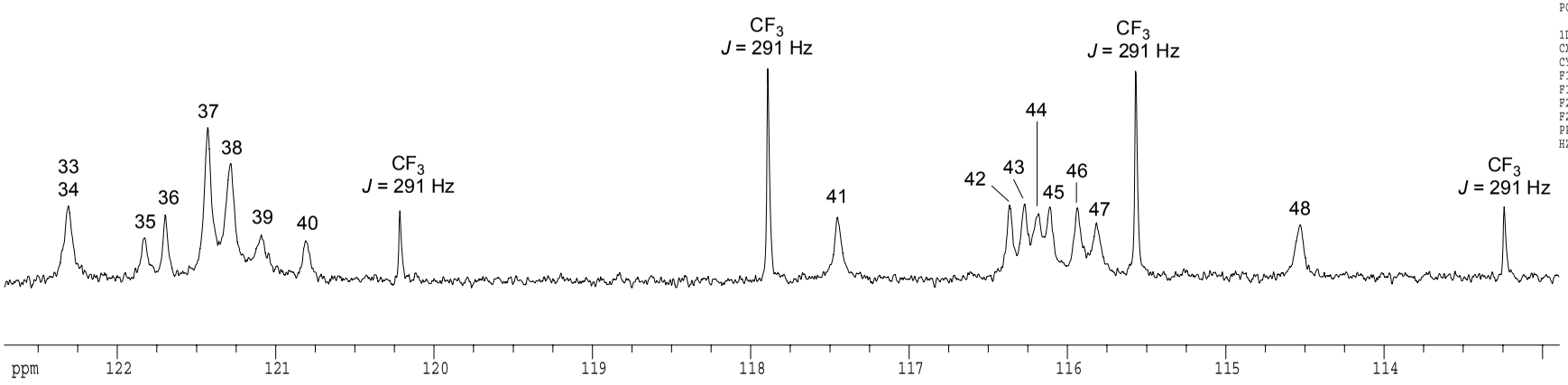
F2 - Acquisition Parameters
Date_            20070517
Time                 1.34
INSTRUM           cryo500
PROBHD    5 mm CPTCI 1H-
PULPROG           zgdc30
TD                  65418
SOLVENT               D2O
NS                    14000
DS                        4
SWH             30303.031 Hz
FIDRES          0.463222 Hz
AQ              1.0794470 sec
RG                14596.5
DW                16.500 usec
DE                 6.00 usec
TE                 320.0 K
D1             0.25000000 sec
d11            0.03000000 sec
MCREST         0.00000000 sec
MCWRK          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

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

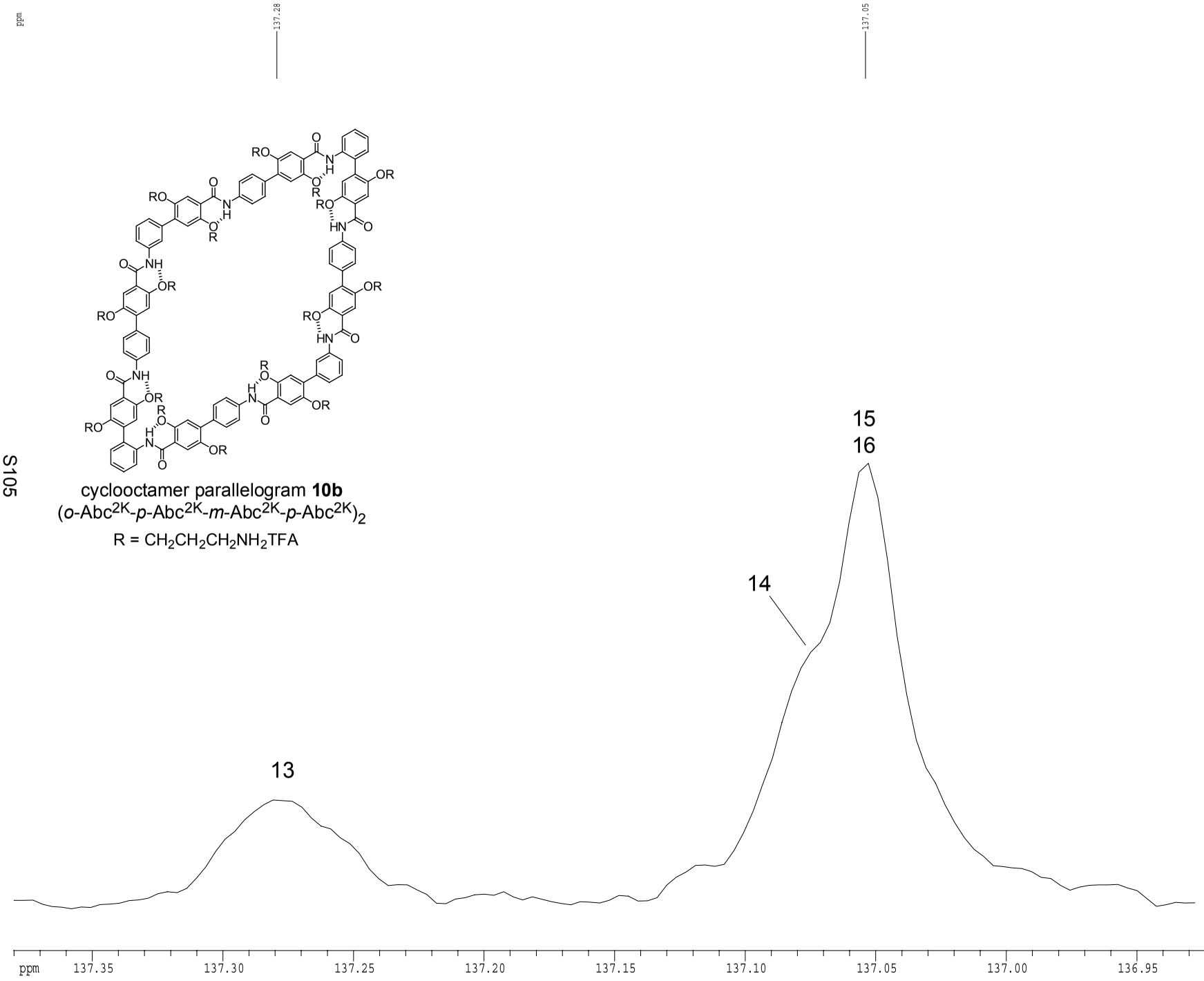
1D NMR plot parameters
CX                   22.80 cm
CY                    8.47 cm
F1P                122.713 ppm
F1                 15434.95 Hz
F2P                112.894 ppm
F2                 14199.86 Hz
PPMCM           0.43067 ppm/cm
HZCM            54.17035 Hz/cm
```



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

cyclooctamer parallelogram **10b**
(*o*-Abc^{2K}-*p*-Abc^{2K}-*m*-Abc^{2K}-*p*-Abc^{2K})$_2$

R = $CH_2CH_2CH_2NH_2TFA$

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Current Data Parameters
USER                cgotha
NAME              cg-11-37
EXPNO                   12
PROCNO                   1

F2 - Acquisition Parameters
Date_             20070517
Time                  1.34
INSTRUM           cryo500
PROBHD    5 mm CPTCI 1H-
PULPROG           zgdc30
TD                 65418
SOLVENT              D2O
NS                   14000
DS                       4
SWH             30303.031 Hz
FIDRES          0.463222 Hz
AQ              1.0794470 sec
RG                14596.5
DW                16.500 usec
DE                 6.00 usec
TE                 320.0 K
D1              0.25000000 sec
d11             0.03000000 sec
MCREST          0.00000000 sec
MCWRK           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

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                   24.95 cm
F1P                137.380 ppm
F1                 17279.73 Hz
F2P                136.924 ppm
F2                 17222.39 Hz
PPMCM             0.01999 ppm/cm
HZCM              2.51490 Hz/cm
```


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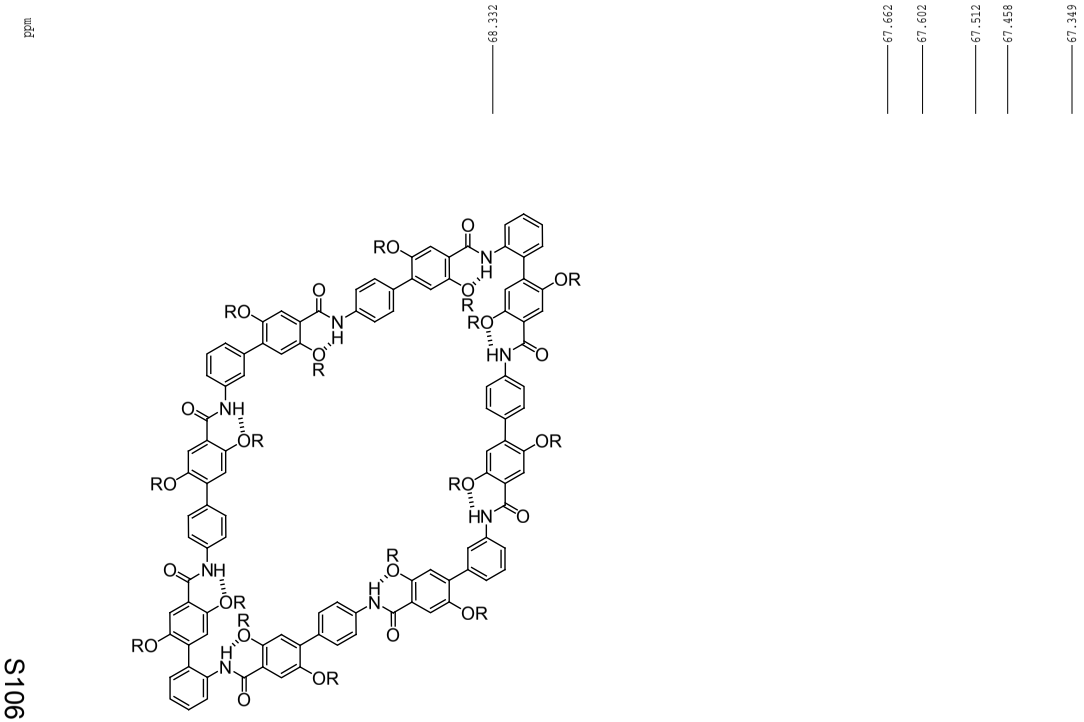
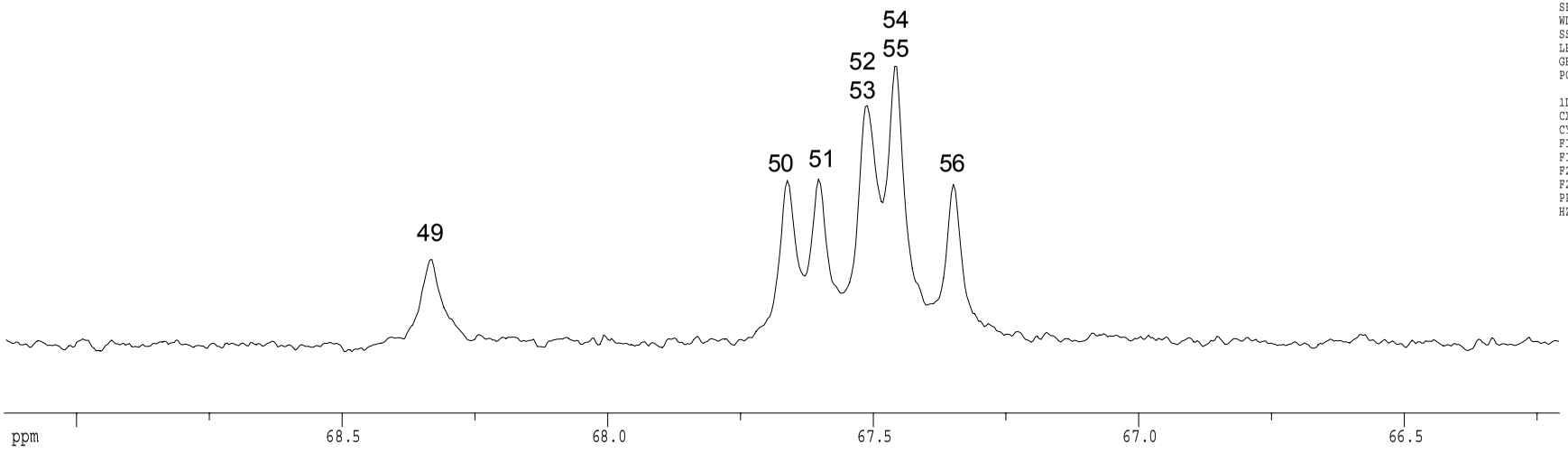
^{13}C NMR (125 MHz, 320 K, D_2O) spectrum of cyclooctamer parallelogram **10b** (aliphatic region expansion 1)



cyclooctamer parallelogram **10b**
(*o*-Abc^{2K}-*p*-Abc^{2K}-*m*-Abc^{2K}-*p*-Abc^{2K})$_2$
R = $CH_2CH_2CH_2NH_2TFA$

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Current Data Parameters
USER                cgotha
NAME             cg-11-37
EXPNO                  12
PROCNO                  1

F2 - Acquisition Parameters
Date_            20070517
Time                1.34
INSTRUM          cryo500
PROBHD   5 mm CPTCI 1H-
PULPROG            zgdc30
TD                  65418
SOLVENT               D2O
NS                    14000
DS                        4
SWH              30303.031 Hz
FIDRES            0.463222 Hz
AQ                1.0794470 sec
RG                  14596.5
DW                    16.500 usec
DE                     6.00 usec
TE                    320.0 K
D1                0.25000000 sec
d11               0.03000000 sec
MCREST            0.00000000 sec
MCWRK             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

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                      8.47 cm
F1P                   69.136 ppm
F1                  8695.93 Hz
F2P                   66.208 ppm
F2                  8327.67 Hz
PPMCM                 0.12841 ppm/cm
HZCM                 16.15173 Hz/cm
```

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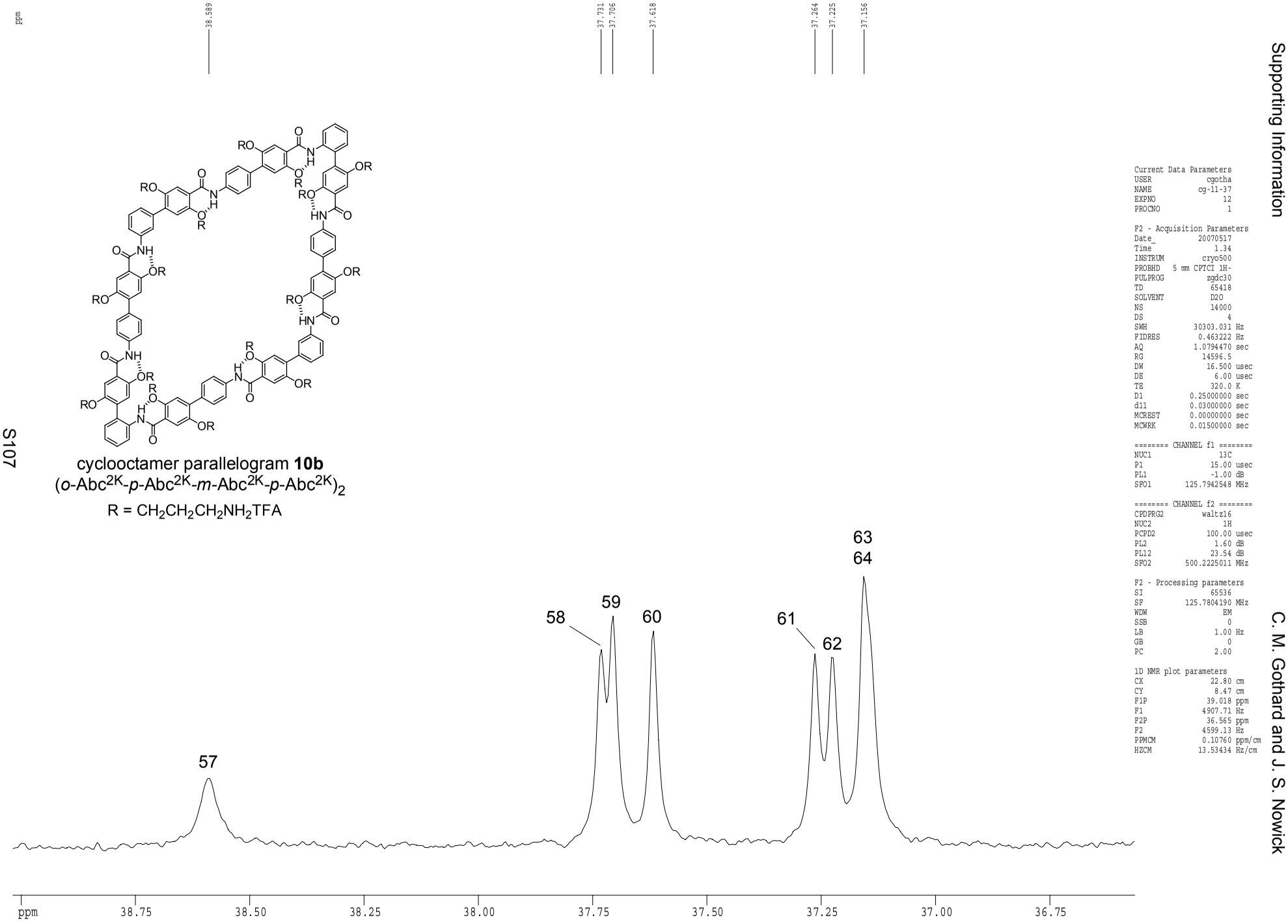
^{13}C NMR (125 MHz, 320 K, D_2O) spectrum of cyclooctamer parallelogram **10b** (aliphatic region expansion 2)



Current Data Parameters
USER cgotha
NAME cg-11-37
EXPNO 12
PROCNO 1

F2 - Acquisition Parameters
Date_ 20070517
Time 1.34
INSTRUM cryo500
PROBHD 5 mm CPTCI 1H-
PULPROG zgdc30
TD 65418
SOLVENT D2O
NS 14000
DS 4
SWH 30303.031 Hz
FIDRES 0.463222 Hz
AQ 1.0794470 sec
RG 14596.5
DW 16.500 usec
DE 6.00 usec
TE 320.0 K
D1 0.25000000 sec
d11 0.03000000 sec
MCREST 0.00000000 sec
MCWRK 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

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 8.47 cm
F1P 39.018 ppm
F1 4907.71 Hz
F2P 36.565 ppm
F2 4599.13 Hz
PPMCM 0.10760 ppm/cm
HZCM 13.53434 Hz/cm

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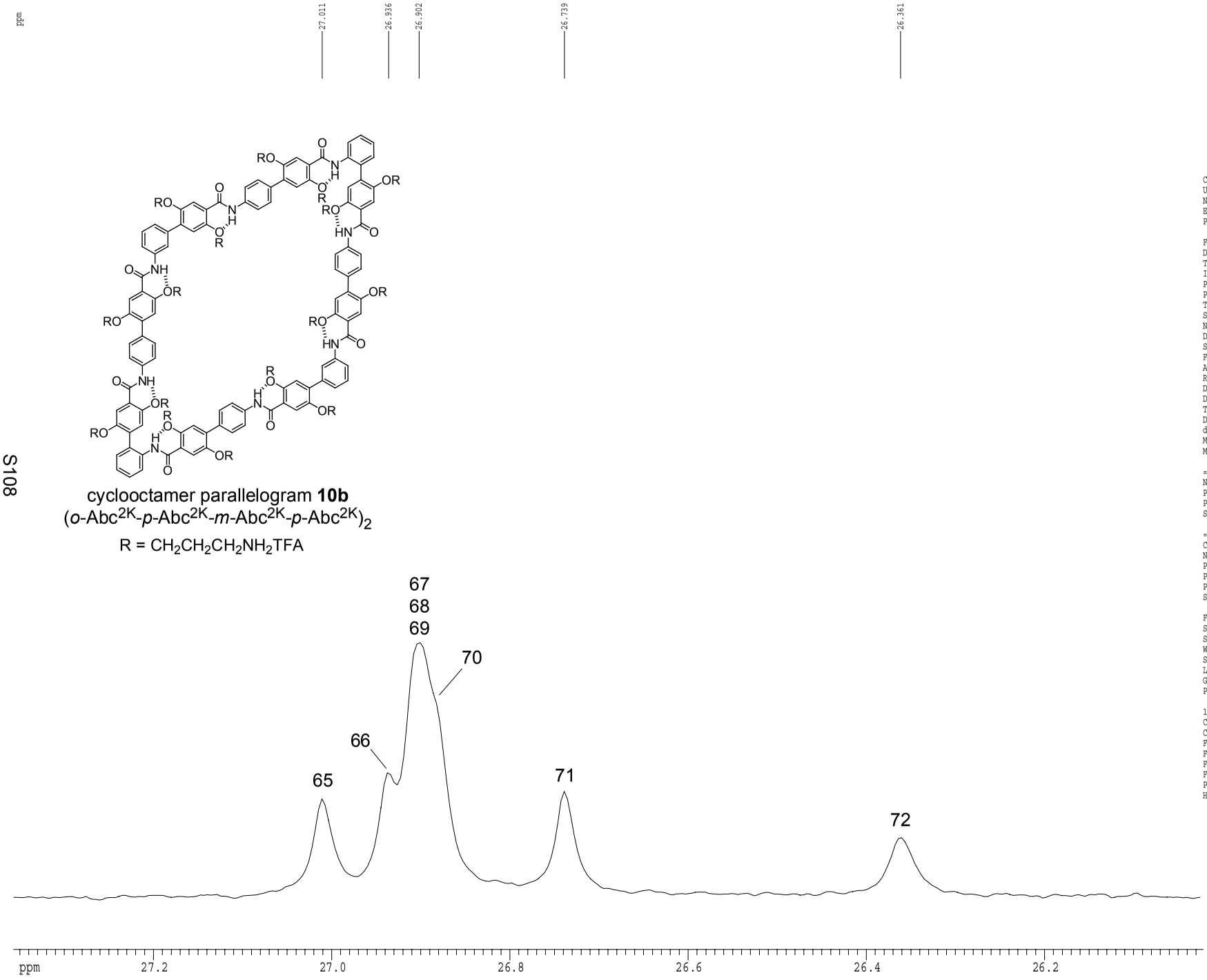
^{13}C NMR (125 MHz, 320 K, D_2O) spectrum of cyclooctamer parallelogram **10b** (aliphatic region expansion 3)



cyclooctamer parallelogram **10b**
(*o*-Abc^{2K}-*p*-Abc^{2K}-*m*-Abc^{2K}-*p*-Abc^{2K})$_2$

R = $CH_2CH_2CH_2NH_2TFA$

```
Current Data Parameters
USER                cgotha
NAME              cg-11-37
EXPNO                   12
PROCNO                   1

F2 - Acquisition Parameters
Date_            20070517
Time                 1.34
INSTRUM           cryo500
PROBHD    5 mm CPTCI 1H-
PULPROG           zgdc30
TD                  65418
SOLVENT               D2O
NS                    14000
DS                        4
SWH              30303.031 Hz
FIDRES           0.463222 Hz
AQ               1.0794470 sec
RG                 14596.5
DW                   16.500 usec
DE                    6.00 usec
TE                   320.0 K
D1               0.25000000 sec
d11              0.03000000 sec
MCREST           0.00000000 sec
MCWRK            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

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                    4.89 cm
F1P                 27.357 ppm
F1                 3440.96 Hz
F2P                 26.022 ppm
F2                 3273.07 Hz
PPMCM              0.05854 ppm/cm
HZCM               7.36353 Hz/cm
```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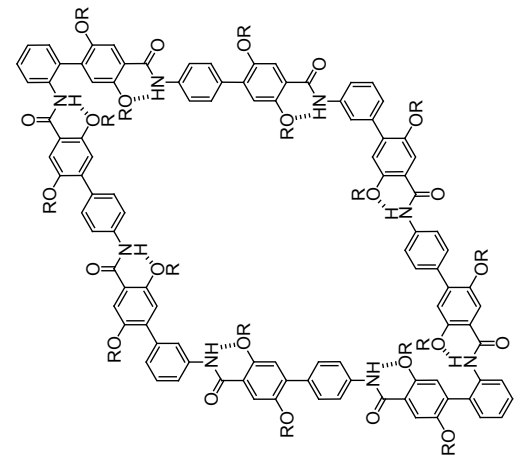
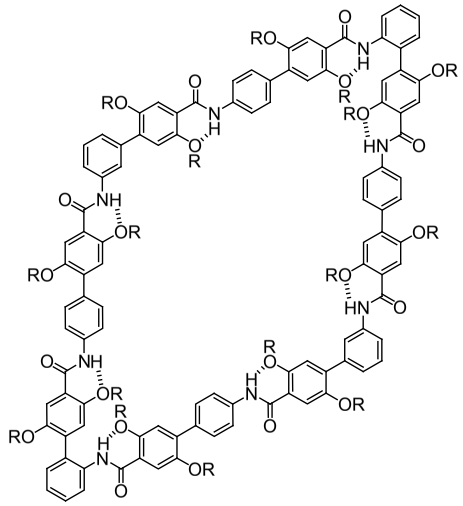
^{1}H NMR (500 MHz, 298 K, CD_3OD) spectrum of cyclooctamer parallelogram **10b**



cyclooctamer parallelogram **10b**
(*o*-Abc^{2K}-*p*-Abc^{2K}-*m*-Abc^{2K}-*p*-Abc^{2K})$_2$
R = $CH_2CH_2CH_2NH_2$TFA

^{1}H NMR (500 MHz, 298 K, CD_3OD) spectrum of cyclooctamer parallelogram **10b** (aromatic region)



cyclooctamer parallelogram **10b**
(*o*-Abc^{2K}-*p*-Abc^{2K}-*m*-Abc^{2K}-*p*-Abc^{2K})$_2$
R = $CH_2CH_2CH_2NH_2TFA$

Current Data Parameters

USER	cgotha
NAME	cg-11-37
EXPNO	5
PROCNO	1

F2 - Acquisition Parameters

Date_	20070506
Time_	0.36
INSTRUM	qn500
PROBHD	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
DW	62.400 usec
DE	6.00 usec
TE	298.0 K
D1	0.10000000 sec
MCREST	0.00000000 sec
MCWRK	0.01500000 sec

======== CHANNEL f1 ========

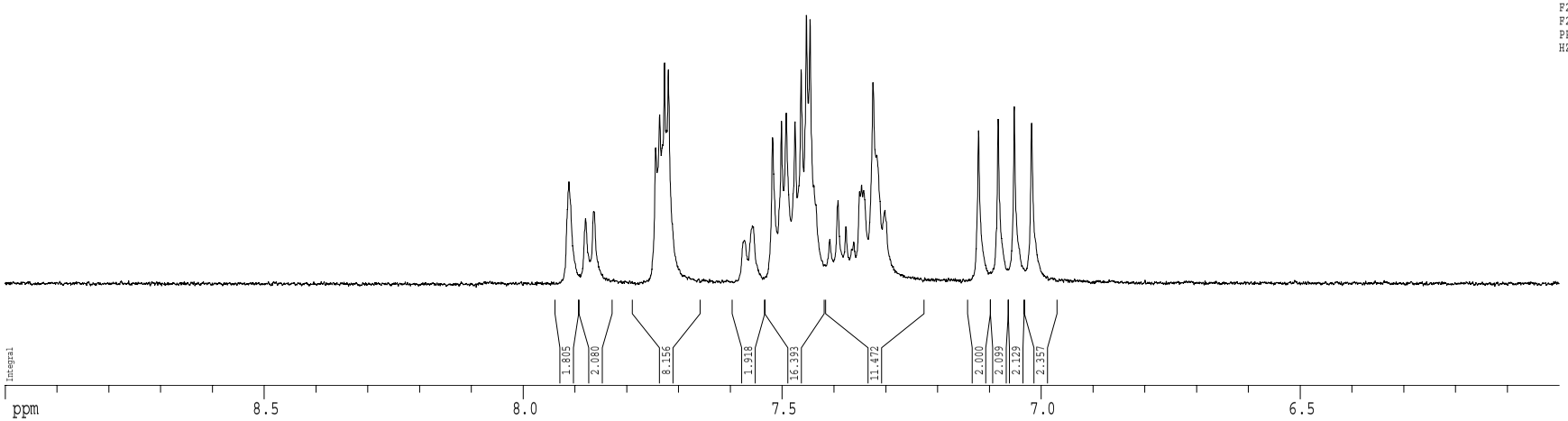
NUC1	1H
P1	12.00 usec
PL1	-3.00 dB
SFO1	499.9334995 MHz

F2 - Processing parameters

SI	65536
SF	499.9300681 MHz
WDW	EM
SSB	0
LB	0.30 Hz
GB	0
PC	4.00

1D NMR plot parameters

CX	22.80 cm
CY	246.39 cm
F1P	9.000 ppm
F1	4499.37 Hz
F2P	6.000 ppm
F2	2999.58 Hz
PPMCM	0.13158 ppm/cm
HZCM	65.78027 Hz/cm



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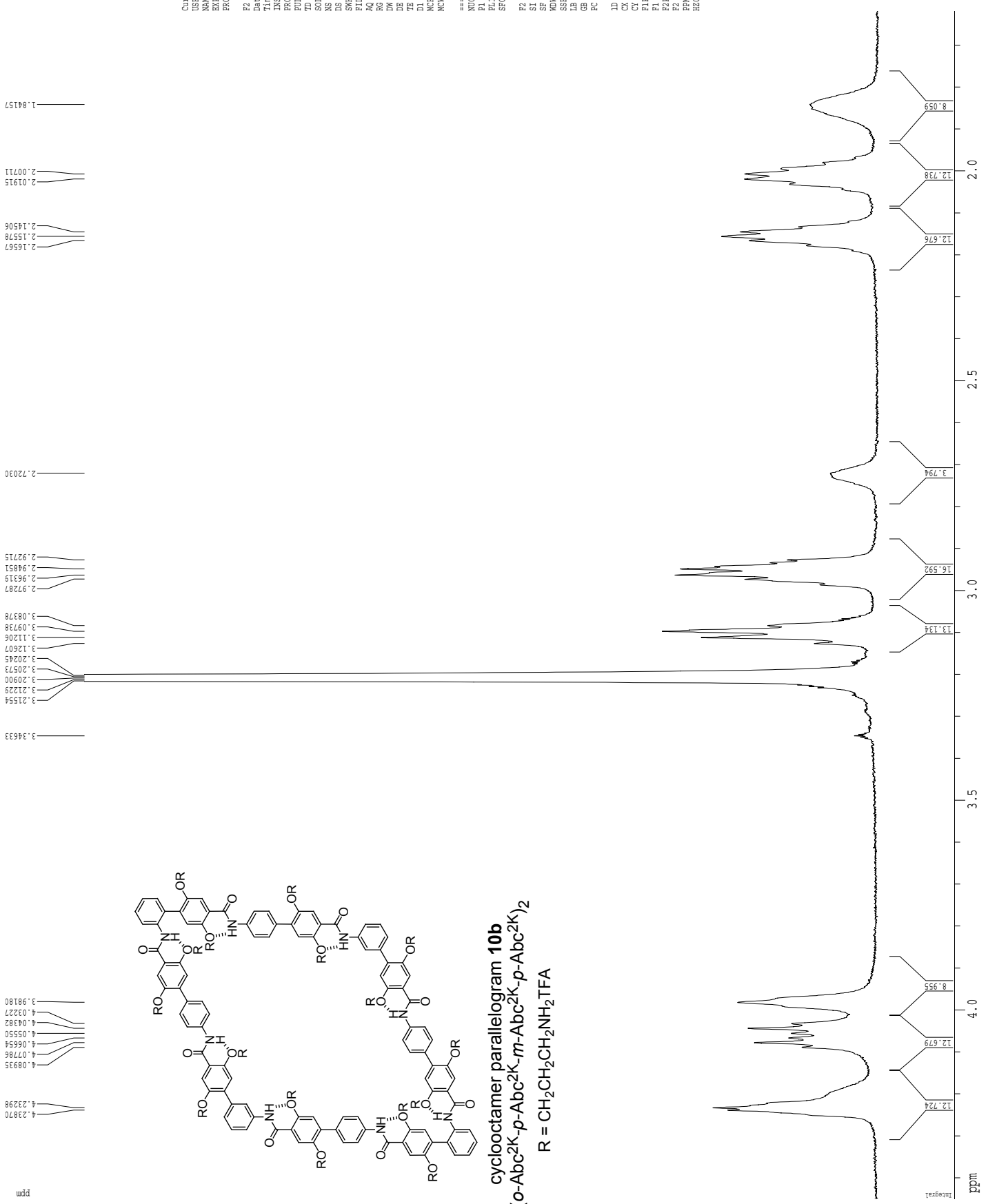
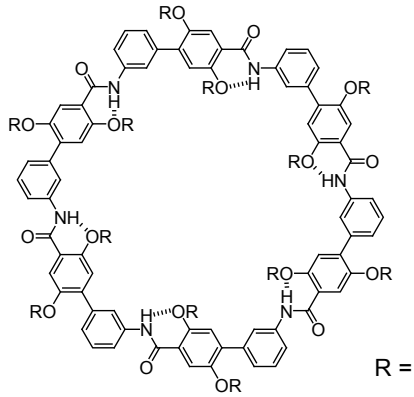
^{1}H NMR (500 MHz, 298 K, CD_3OD) spectrum of cyclooctamer parallelogram **10b** (aliphatic region)

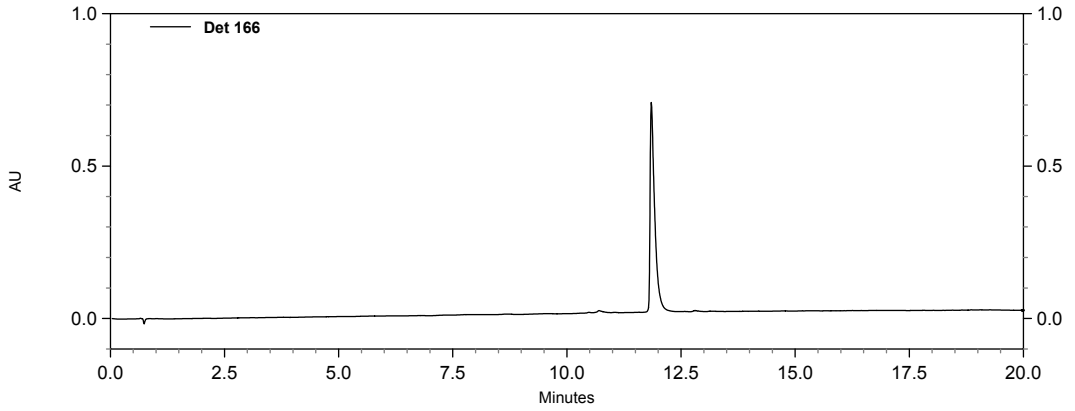


cyclooctamer parallelogram **10b**
(*o*-Abc^{2K}-*p*-Abc^{2K}-*m*-Abc^{2K}-*p*-Abc^{2K})$_2$
R = $CH_2CH_2CH_2NH_2$TFA

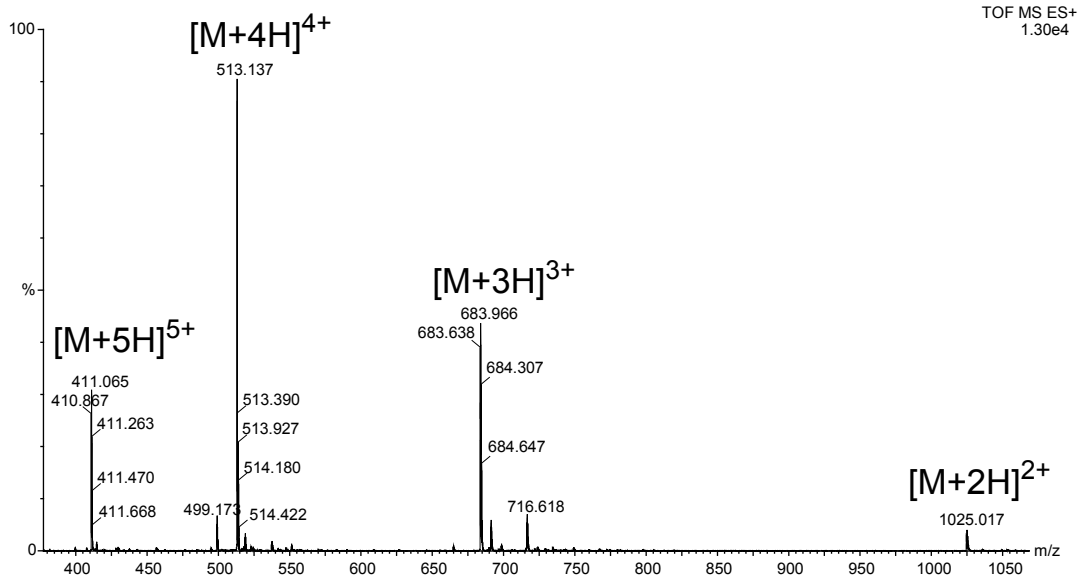
cyclohexamer ring: (*m*-Abc^{2K})$_6$ (**11a**)
Analytical RP-HPLC chromatograph and mass spectrum (ESI-MS)



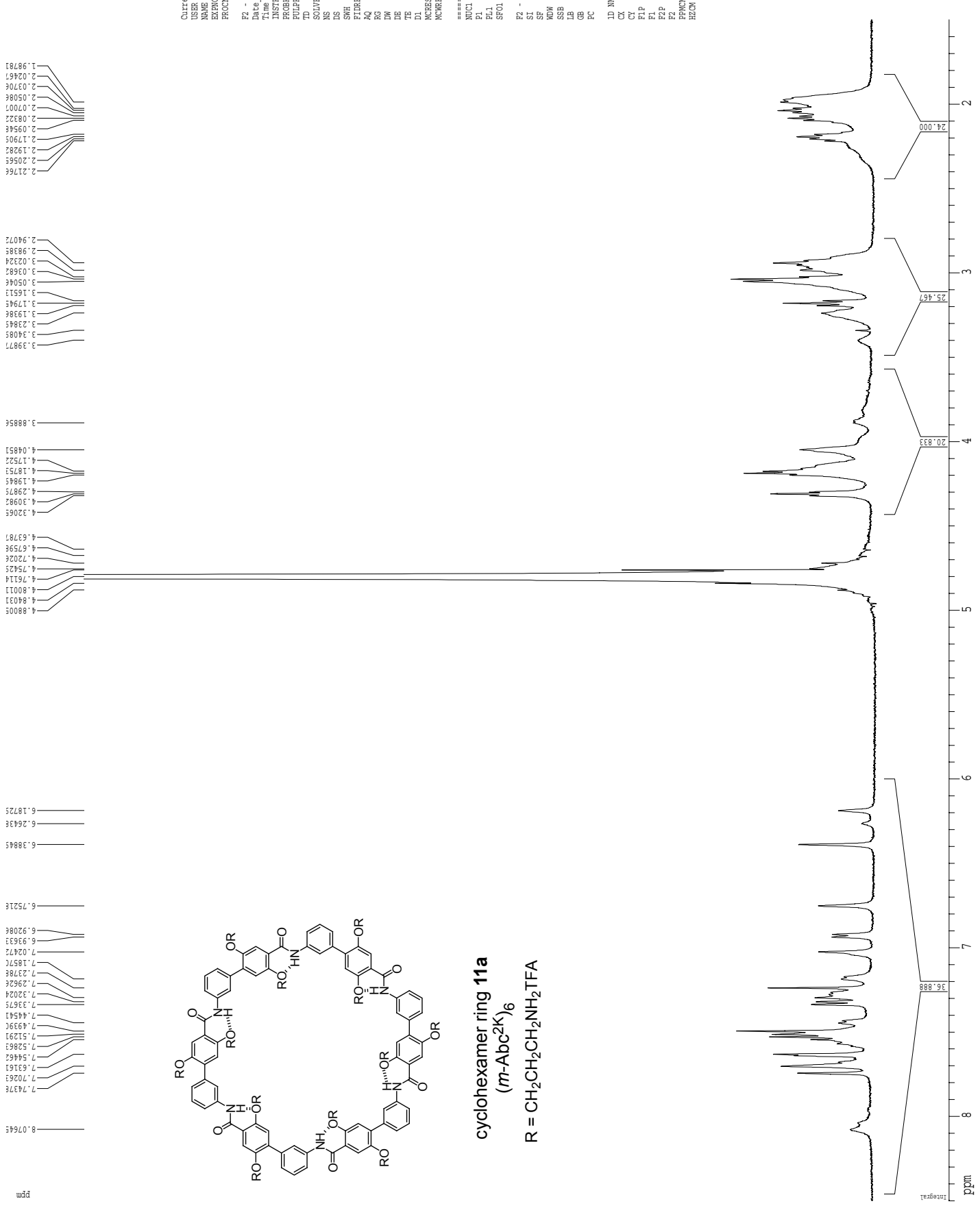
(a) Analytical RP-HPLC (5-50% acetonitrile with 0.1% TFA over 20 min, λ = 214)



(b) ESI Mass spectrum. (Calcd exact mass for $C_{114}H_{138}N_{18}O_{18}$ [M] = 2047.04)

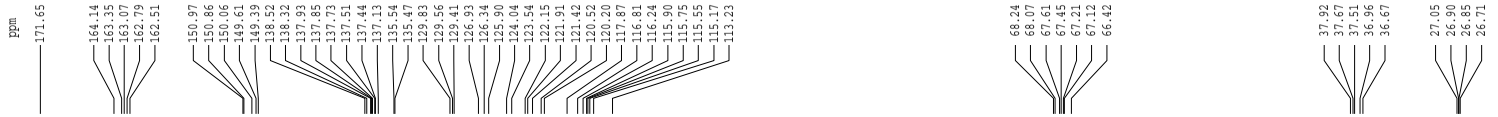


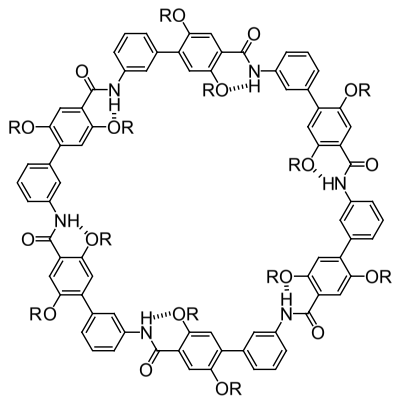
^{1}H NMR (500 MHz, 298 K, D_2O) spectrum of cyclohexamer ring **11a**



cyclohexamer ring **11a**
(*m*-Abc^{2K})$_6$
R = $CH_2CH_2CH_2NH_2$TFA

^{13}C NMR (125 MHz, 298 K, D_2O) spectrum of cyclohexamer ring **11a**





cyclohexamer ring **11a**
(*m*-Abc^{2K})$_6$
R = $CH_2CH_2CH_2NH_2TFA$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Current Data Parameters
USER                cgotha
NAME             cg-11-73
EXPNO                   4
PROCNO                  1

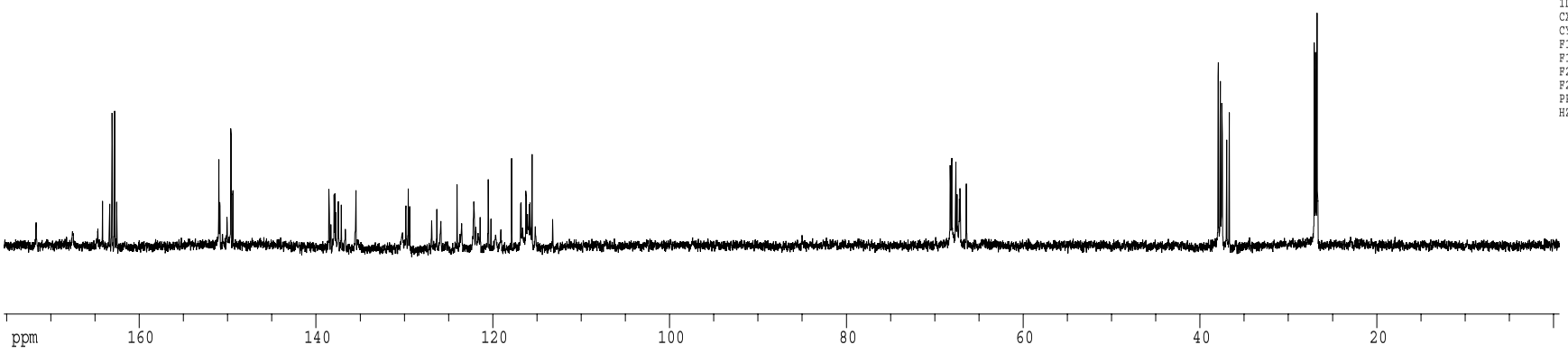
F2 - Acquisition Parameters
Date_            20070524
Time                1.32
INSTRUM          cryo500
PROBHD   5 mm CPTCI 1H-
PULPROG          zgdc30
TD                 65418
SOLVENT              D2O
NS                 12000
DS                     4
SWH            30303.031 Hz
FIDRES         0.463222 Hz
AQ             1.0794470 sec
RG               18390.4
DW                16.500 usec
DE                 6.00 usec
TE                320.0 K
D1             0.25000000 sec
d11            0.03000000 sec
MCREST         0.00000000 sec
MCWRK          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

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
F1P              175.270 ppm
F1             22045.56 Hz
F2P               -0.632 ppm
F2               -79.45 Hz
PPMCM           7.71500 ppm/cm
HZCM          970.39539 Hz/cm
```



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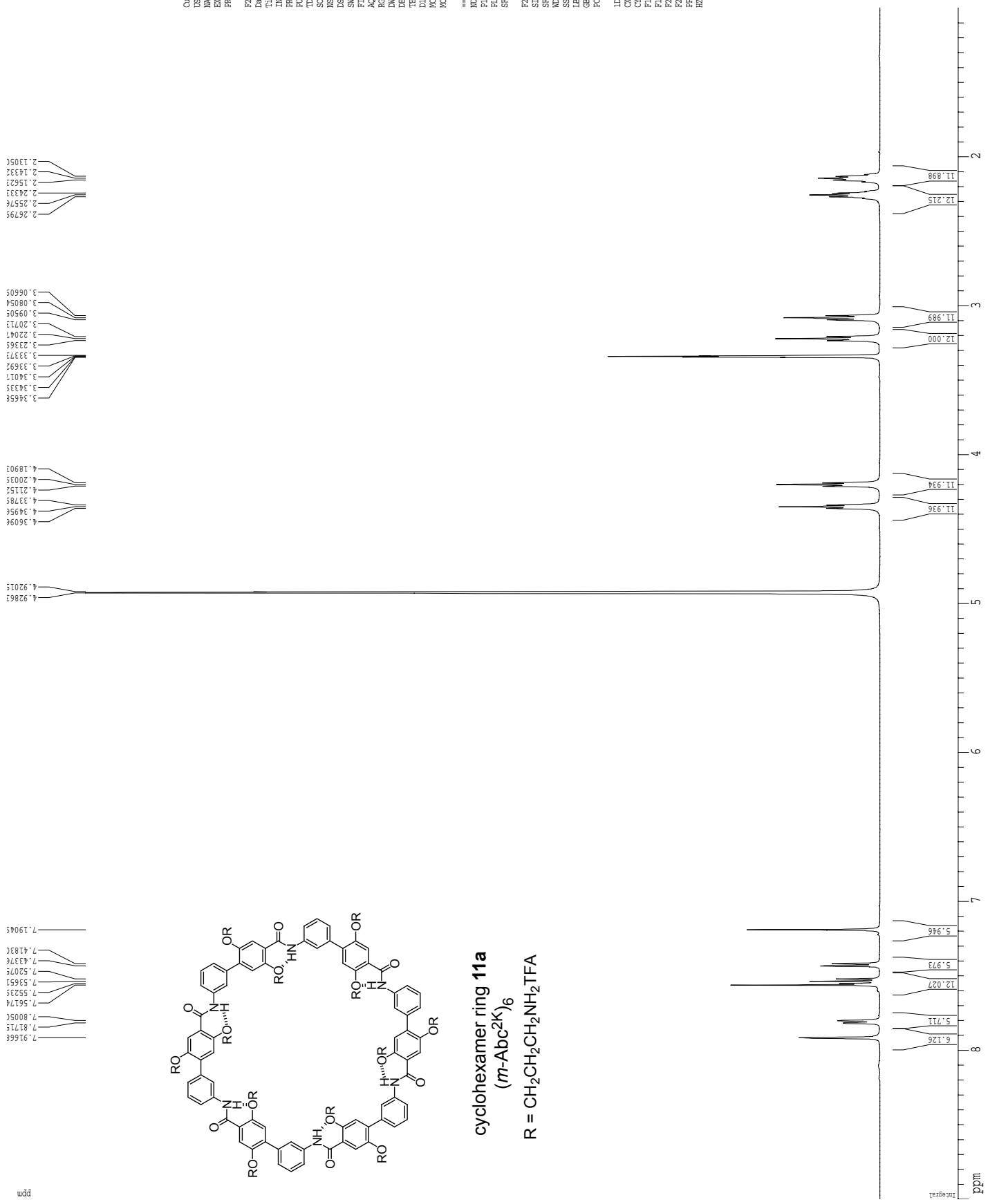
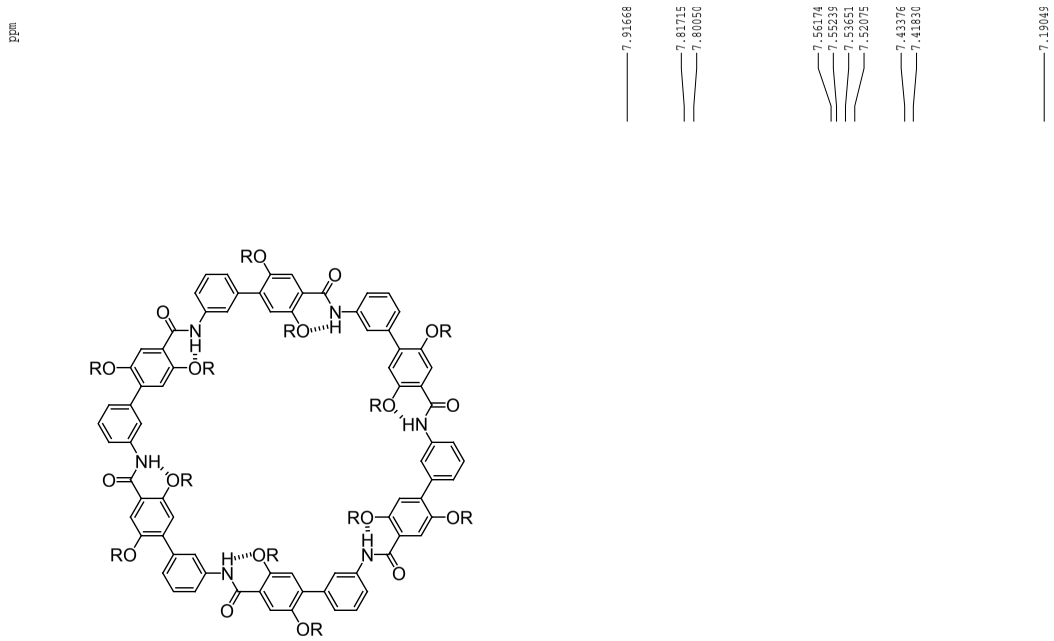
^{1}H NMR (500 MHz, 298 K, CD_3OD) spectrum of cyclohexamer ring **11a**



^{1}H NMR (500 MHz, 298 K, CD_3OD) spectrum of cyclohexamer ring **11a** (aromatic region)



cyclohexamer ring **11a**
(*m*-Abc^{2K})$_6$
R = $CH_2CH_2CH_2NH_2$TFA


```
Current Data Parameters
USER                cgotha
NAME             cg-10-263
EXPNO                    4
PROCNO                   1

F2 - Acquisition Parameters
Date_             20061126
Time                 2.14
INSTRUM           cryo500
PROBHD    5 mm CPTCI 1H-
PULPROG               zg30
TD                   81728
SOLVENT              CD3OD
NS                       8
DS                       2
SWH               8012.820 Hz
FIDRES               0.098043 Hz
AQ                5.0998774 sec
RG                      10.1
DW                   62.400 usec
DE                    6.00 usec
TE                   298.0 K
D1              0.10000000 sec
MCREST          0.00000000 sec
MCWRK           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                   29.29 cm
F1P                  9.000 ppm
F1                 4501.98 Hz
F2P                  6.000 ppm
F2                 3001.32 Hz
PPMCM              0.13158 ppm/cm
HZCM              65.81842 Hz/cm
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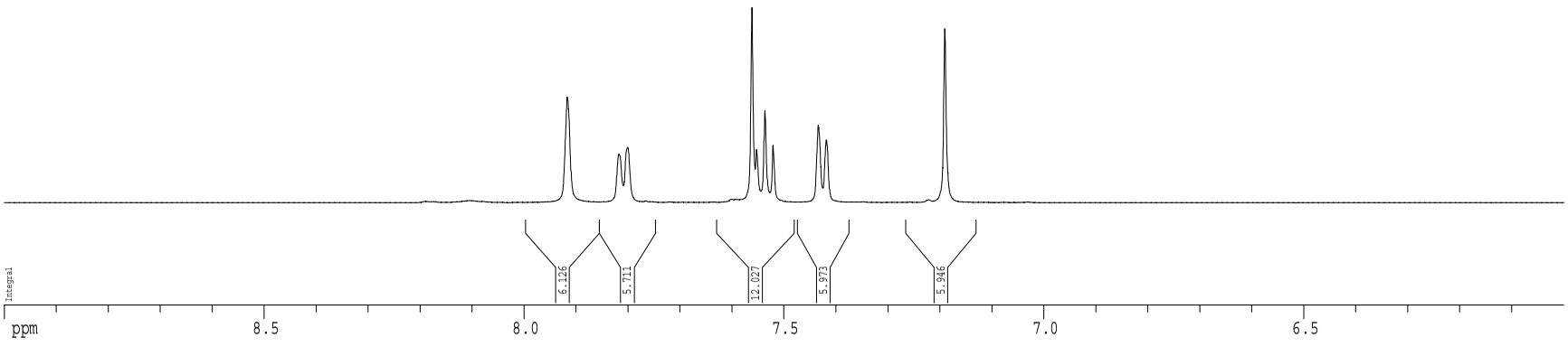
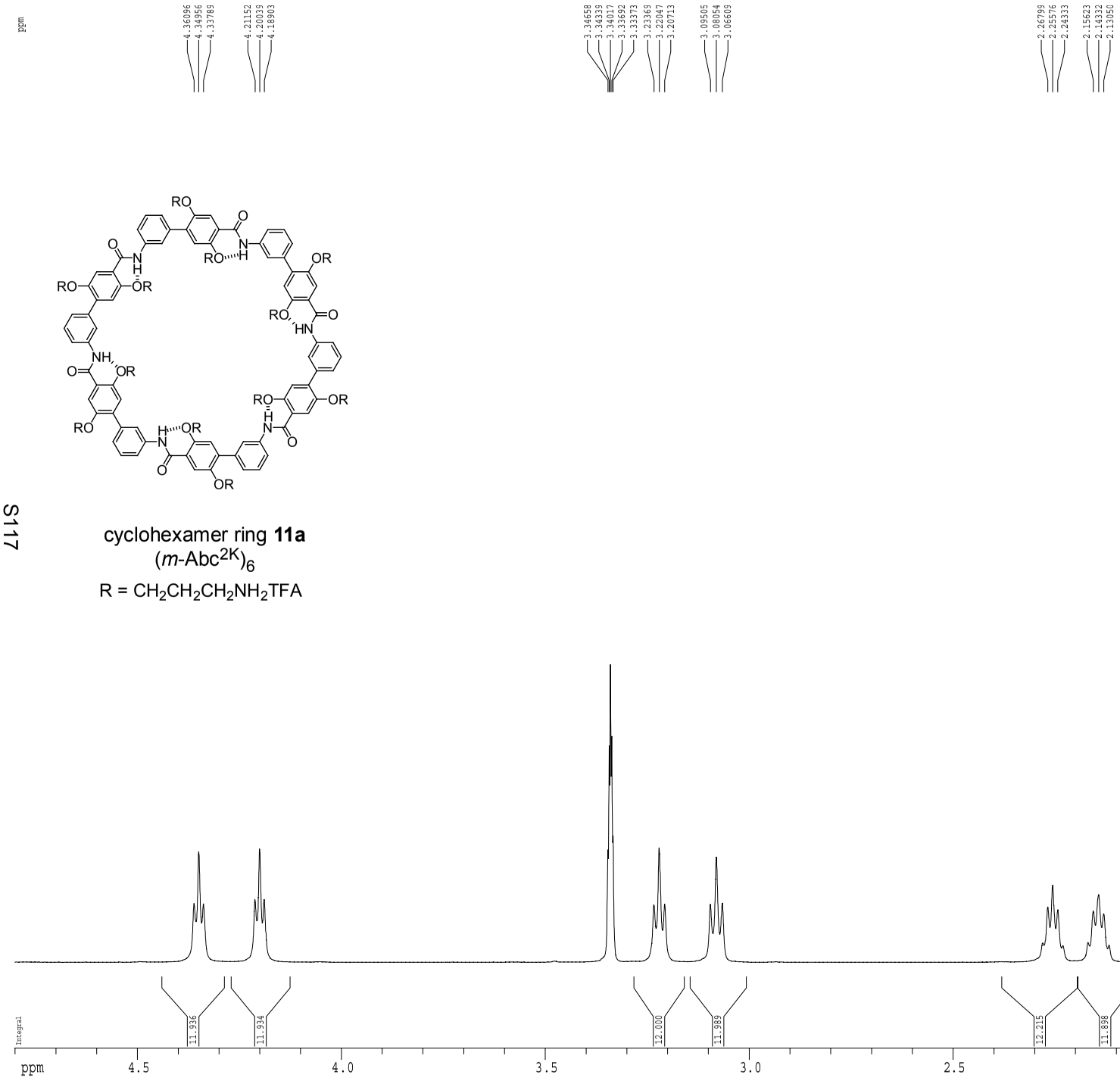


C. M. Gothard and J. S. Nowick

^{1}H NMR (500 MHz, 298 K, CD_3OD) spectrum of cyclohexamer ring **11a** (aliphatic region)



Current Data Parameters
USER cgotha
NAME cg-10-263
EXPNO 4
PROCNO 1

F2 - Acquisition Parameters
Date_ 20061126
Time 2.14
INSTRUM cryo500
PROBHD 5 mm CPTCI 1H-
PULPROG zg30
TD 81728
SOLVENT CD3OD
NS 8
DS 2
SWH 8012.820 Hz
FIDRES 0.098043 Hz
AQ 5.0998774 sec
RG 10.1
DW 62.400 usec
DE 6.00 usec
TE 298.0 K
D1 0.10000000 sec
MCREST 0.00000000 sec
MCWRK 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 29.29 cm
F1P 4.800 ppm
F1 2401.06 Hz
F2P 1.600 ppm
F2 800.35 Hz
PPMCM 0.14035 ppm/cm
HZCM 70.20632 Hz/cm

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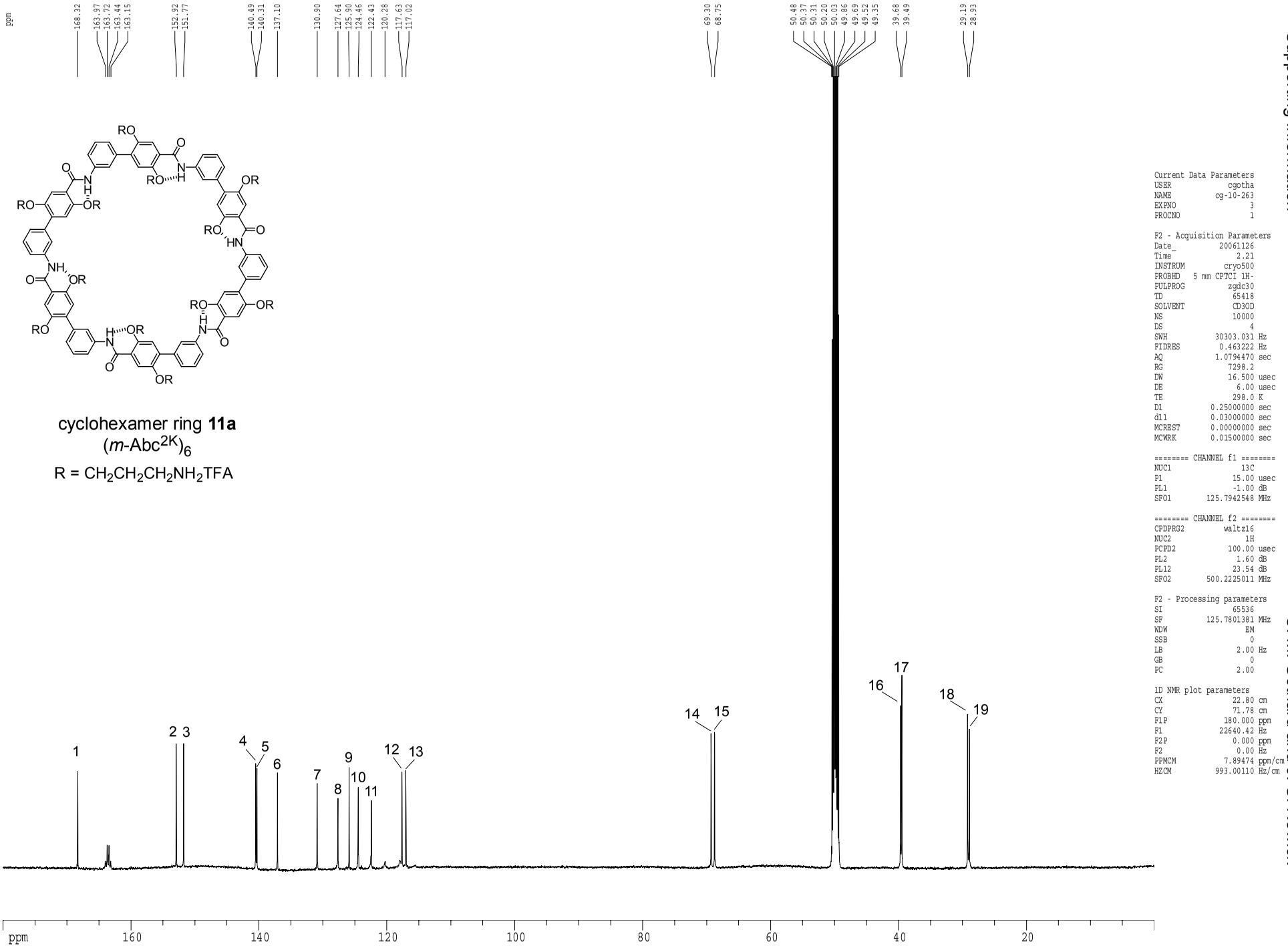
^{13}C NMR (125 MHz, 298 K, CD_3OD) spectrum of cyclohexamer ring **11a**



cyclohexamer ring **11a**
$(m\text{-Abc}^{2K})_6$
R = $CH_2CH_2CH_2NH_2TFA$

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 CPTCI 1H-
PULPROG zgdc30
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 298.0 K
D1 0.25000000 sec
d11 0.03000000 sec
MCREST 0.00000000 sec
MCWRK 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

F2 - Processing parameters
SI 65536
SF 125.7801381 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 0.00 Hz
PPMCM 7.89474 ppm/cm
HZCM 993.00110 Hz/cm

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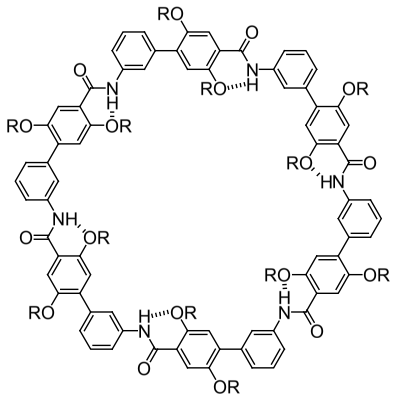
^{13}C NMR (500 MHz, 298 K, CD_3OD) spectrum of cyclohexamer ring **11a** (aromatic and carbonyl region)



cyclohexamer ring **11a**
$(m\text{-Abc}^{2K})_6$
R = $CH_2CH_2CH_2NH_2TFA$


```
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 CPTCI 1H-
PULPROG             zgdc30
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                 298.0 K
D1              0.25000000 sec
d11             0.03000000 sec
MCREST          0.00000000 sec
MCWRK           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

F2 - Processing parameters
SI                   65536
SF            125.7801381 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               170.000 ppm
F1               21382.62 Hz
F2P               115.000 ppm
F2               14464.72 Hz
PPMCM             2.41228 ppm/cm
HZCM            303.41699 Hz/cm
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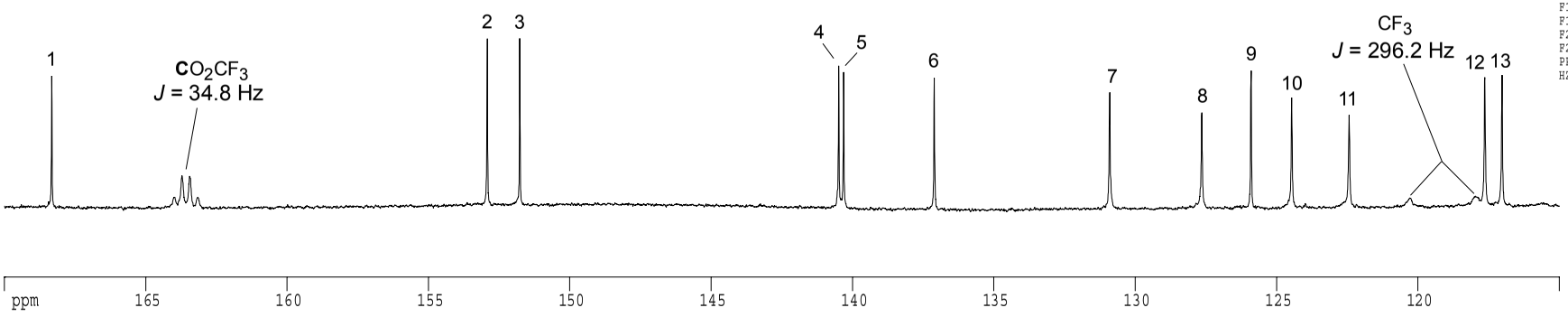
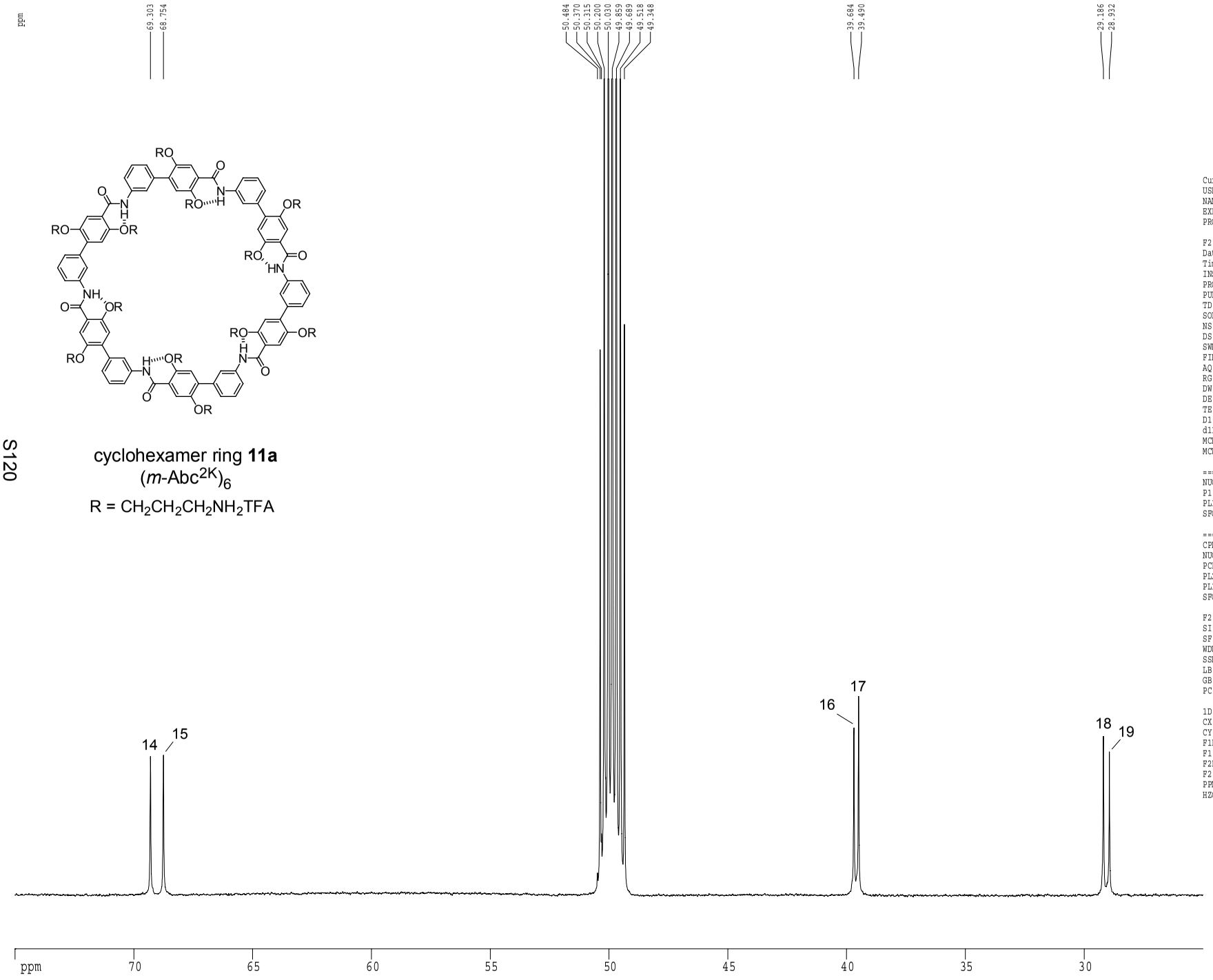


^{13}C NMR (125 MHz, 298 K, CD_3OD) spectrum of cyclohexamer ring **11a** (aliphatic region)



cyclohexamer ring **11a**
$(m\text{-Abc}^{2K})_6$
R = $CH_2CH_2CH_2NH_2TFA$


```
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 CPTCI 1H-
PULPROG           zgdc30
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                298.0 K
D1            0.25000000 sec
d11           0.03000000 sec
MCREST        0.00000000 sec
MCWRK         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

F2 - Processing parameters
SI                 65536
SF          125.7801381 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                75.000 ppm
F1               9433.51 Hz
F2P                25.000 ppm
F2               3144.50 Hz
PPMCM           2.19298 ppm/cm
HZCM          275.83365 Hz/cm
```

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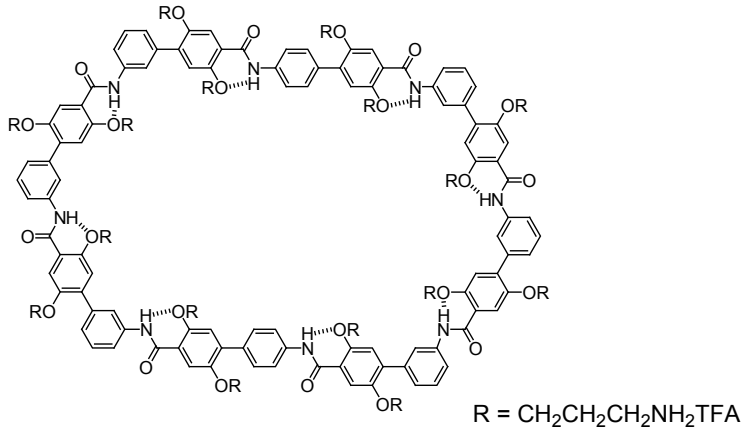
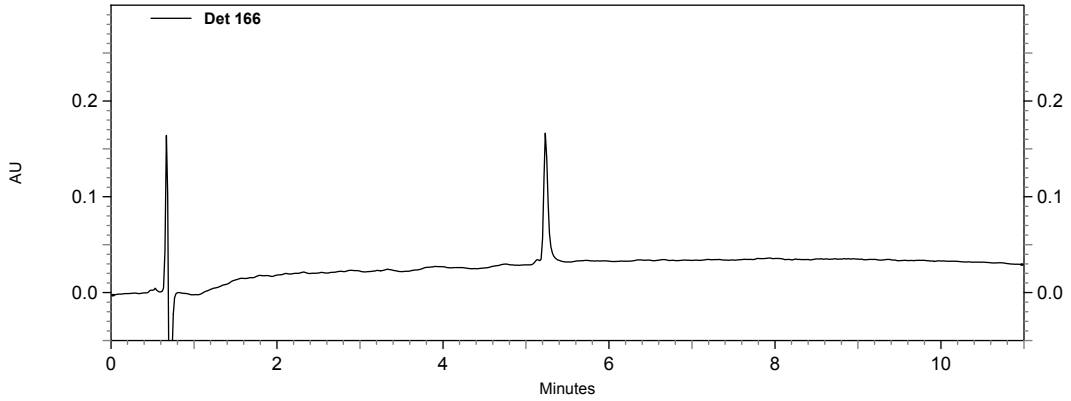
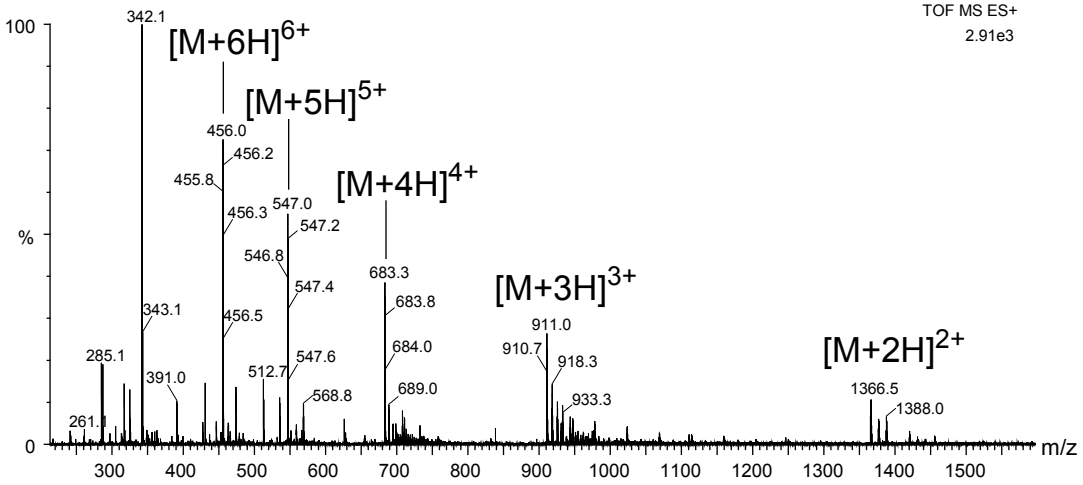
cyclooctamer ring: (*m*-Abc^{2K}-*m*-Abc^{2K}-*m*-Abc^{2K}-*p*-Abc^{2K})$_2$ (**11b**) Analytical RP-HPLC chromatograph and mass spectrum (ESI-MS)



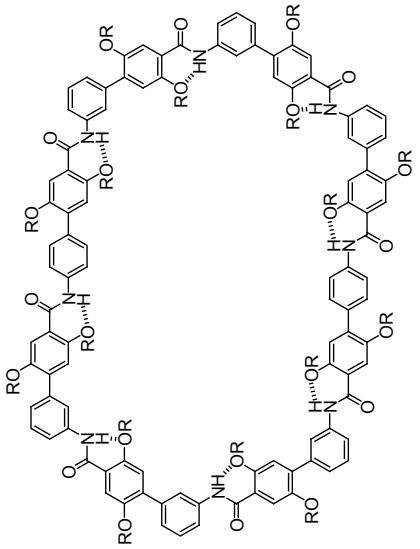
(a) Analytical RP-HPLC (5-50% acetonitrile with 0.1% TFA over 10 min, λ = 214)



(b) ESI Mass spectrum. (Calcd exact mass for $C_{152}H_{184}N_{24}O_{24}$ [M] = 2729.39)

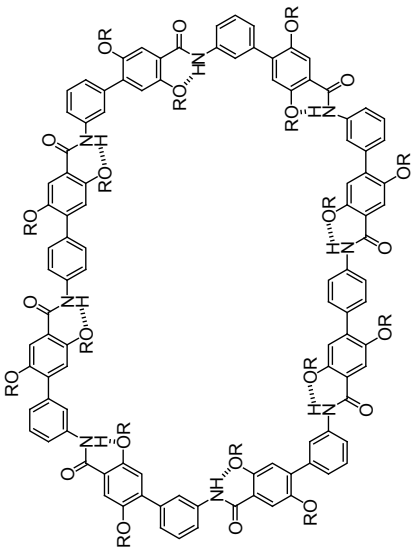


^{1}H NMR (500 MHz, 298 K, D_2O) spectrum of cyclooctamer ring **11b**



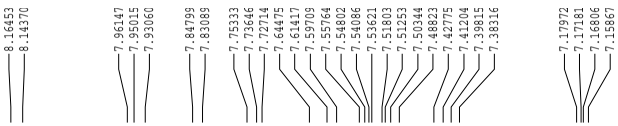
cyclooctamer ring **11b**
(*m*-Abc^{2K}-*m*-Abc^{2K}-*p*-Abc^{2K})$_2$

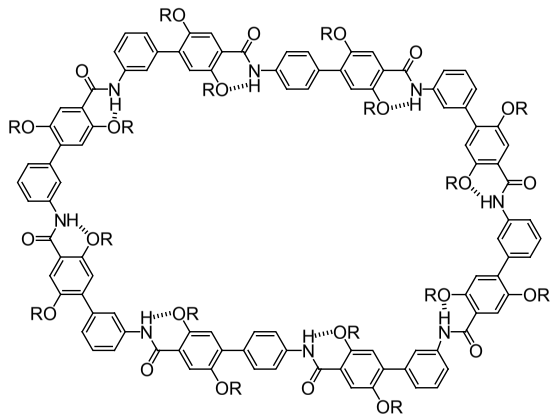
^{1}H NMR (500 MHz, 298 K, CD_3OD) spectrum of cyclooctamer ring **11b**



cyclooctamer ring **11b**
(*m*-Abc^{2K}-*m*-Abc^{2K}-*m*-Abc^{2K}-*p*-Abc^{2K})$_2$

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





cyclooctamer ring **11b**
(*m*-Abc^{2K}-*m*-Abc^{2K}-*m*-Abc^{2K}-*p*-Abc^{2K})$_2$

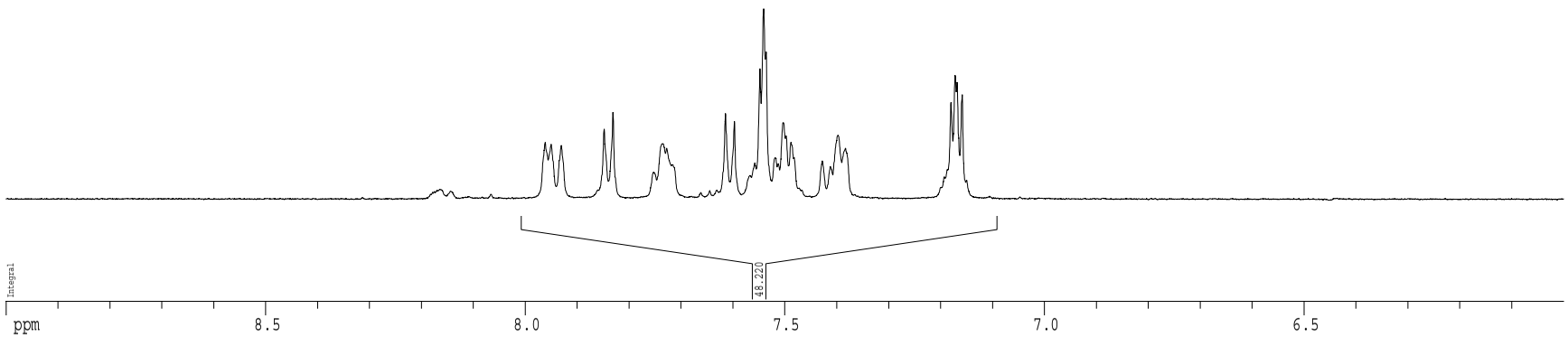
Current Data Parameters
USER cgotha
NAME cg-7-281
EXPNO 1
PROCNO 1

F2 - Acquisition Parameters
Date_ 20060305
Time 22.53
INSTRUM cryo500
PROBHD 5 mm CPTCI 1H-
PULPROG zg30
TD 81728
SOLVENT CD3OD
NS 29
DS 2
SWH 8012.820 Hz
FIDRES 0.098043 Hz
AQ 5.0998774 sec
RG 57
DW 62.400 usec
DE 6.00 usec
TE 298.0 K
D1 0.10000000 sec
MCREST 0.00000000 sec
MCWRK 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 500.2200216 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 4.00

1D NMR plot parameters
CX 22.80 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



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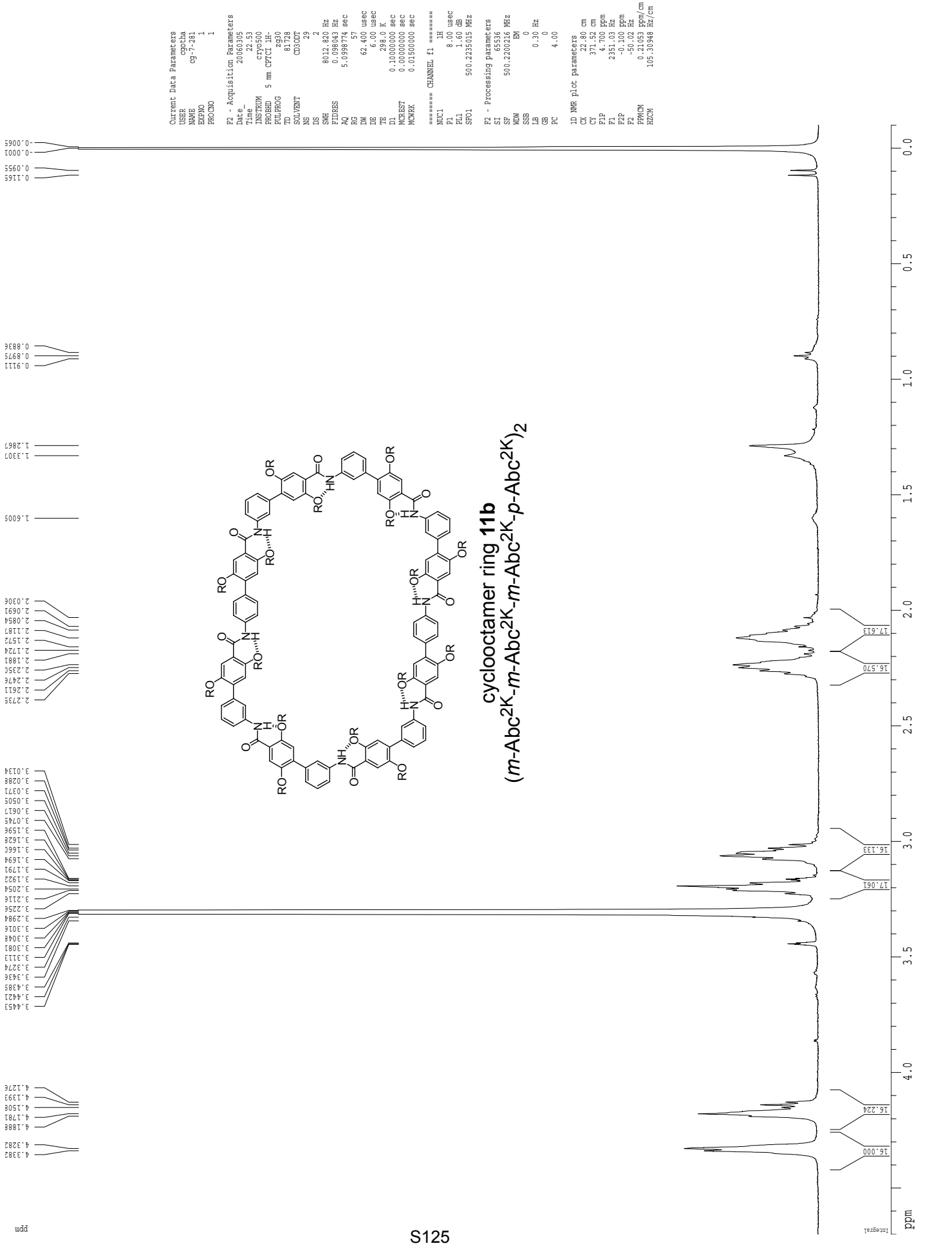
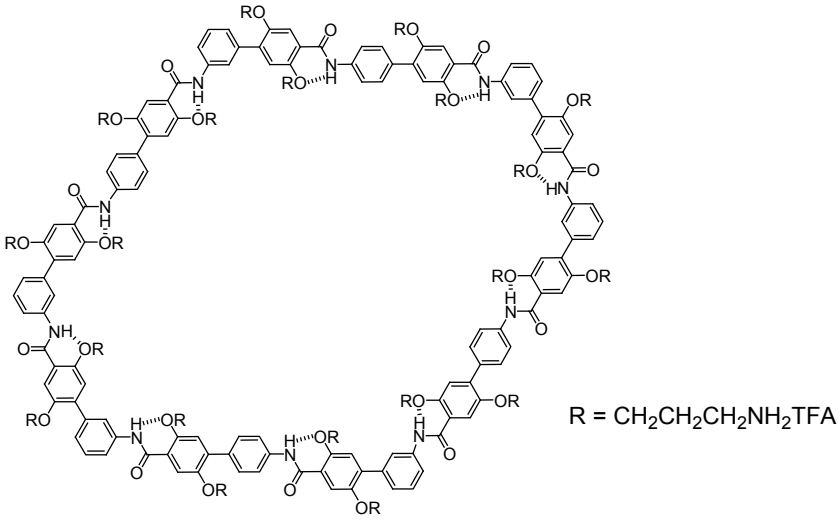
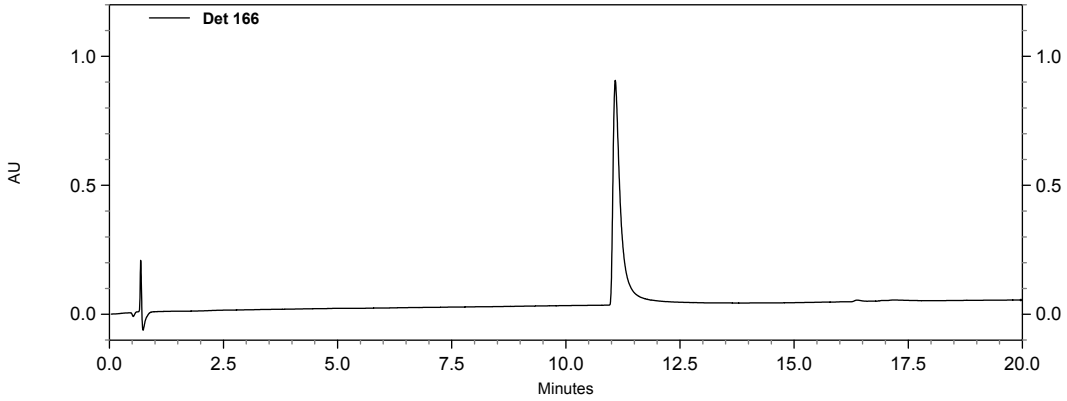
^{1}H NMR (500 MHz, 298 K, CD_3OD) spectrum of cyclooctamer ring **11b**



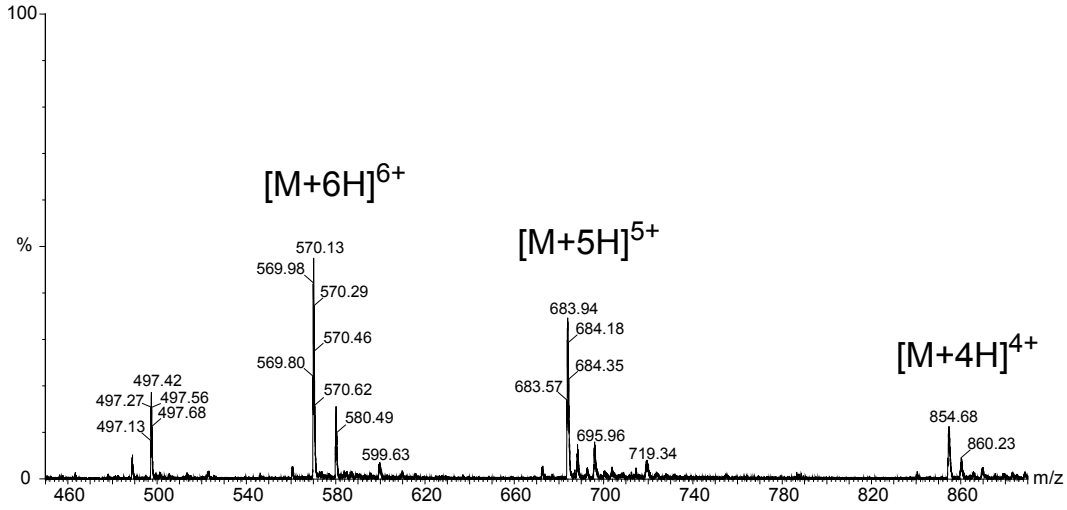
cyclodecamer ring (m-Abc^{2K}-m-Abc^{2K}-p-Abc^{2K}-m-Abc^{2K}-p-Abc^{2K})$_2$ (**11c**) Analytical RP-HPLC chromatograph and mass spectrum (ESI-MS)



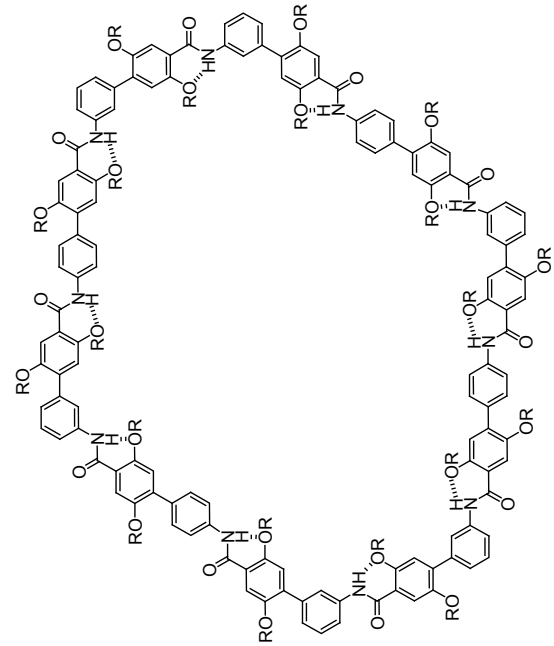
(a) Analytical RP-HPLC (5-50% acetonitrile with 0.1% TFA over 20 min, λ = 214)



(b) ESI Mass spectrum. (Calcd exact mass for $C_{190}H_{230}N_{30}O_{30}$ [M] = 3411.74)



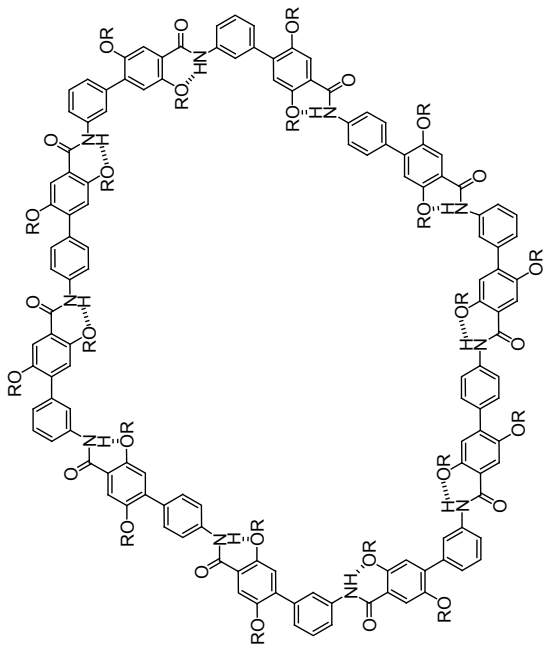
^{1}H NMR (500 MHz, 298 K, D_2O) spectrum of cyclodecamer ring **11c**



cyclodecamer ring **11c**
(*m*-Abc^{2K}-*m*-Abc^{2K}-*p*-Abc^{2K}-*m*-Abc^{2K}-*p*-Abc^{2K})$_2$

R = $CH_2CH_2CH_2NH_2$TFA

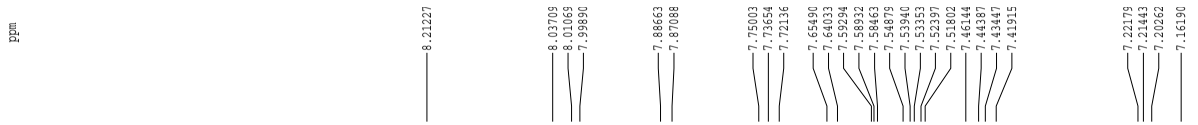
^{1}H NMR (500 MHz, 298 K, CD_3OD) spectrum of cyclodecamer ring **11c**

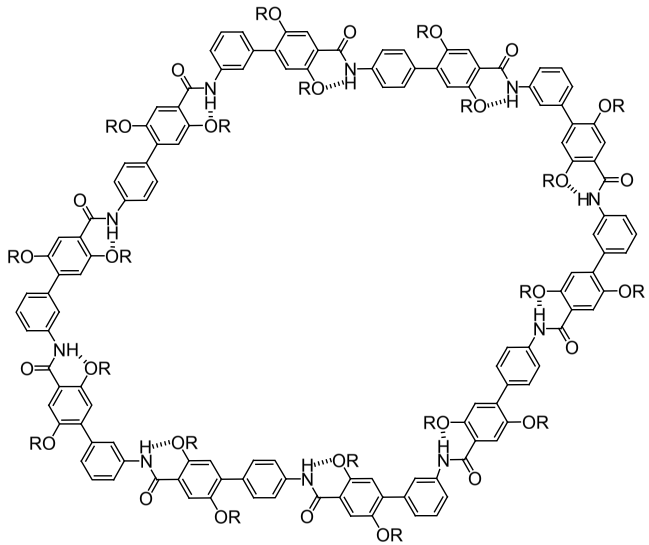


cyclodecamer ring **11c**
(*m*-Abc^{2K}-*m*-Abc^{2K}-*p*-Abc^{2K}-*m*-Abc^{2K}-*p*-Abc^{2K})$_2$

R = $CH_2CH_2CH_2NH_2TFA$

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





cyclodecamer ring **11c**
(*m*-Abc^{2K}-*m*-Abc^{2K}-*p*-Abc^{2K}-*m*-Abc^{2K}-*p*-Abc^{2K})$_2$

R = $CH_2CH_2CH_2NH_2TFA$

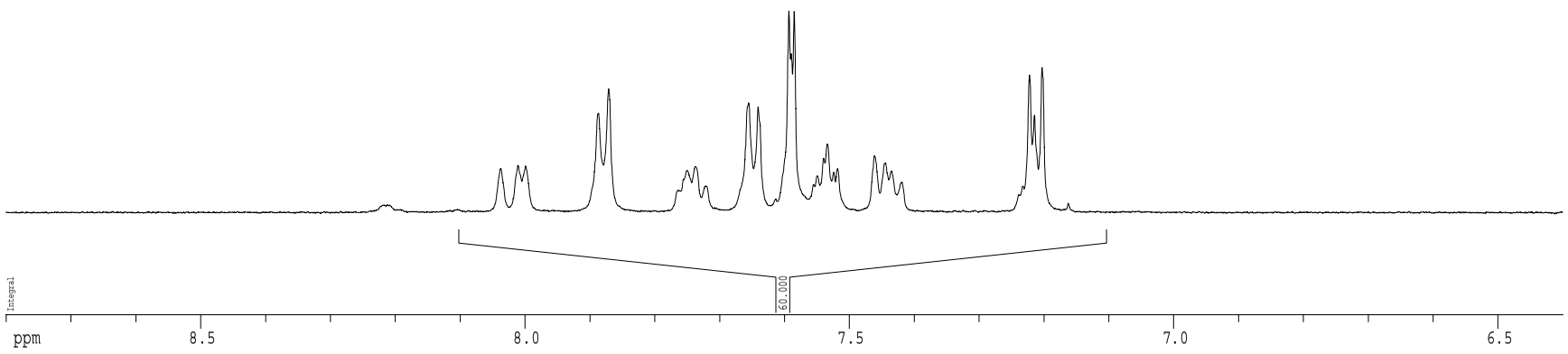
Current Data Parameters
USER cgotha
NAME cg-9-101
EXPNO 1
PROCNO 1

F2 - Acquisition Parameters
Date_ 20060510
Time 19.38
INSTRUM cryo500
PROBHD 5 mm broadband
PULPROG zg30
TD 81728
SOLVENT MeOH
NS 128
DS 2
SWH 8012.820 Hz
FIDRES 0.098043 Hz
AQ 5.0998774 sec
RG 256
DW 62.400 usec
DE 6.00 usec
TE 298.0 K
D1 0.10000000 sec
MCREST 0.00000000 sec
MCWRK 0.01500000 sec

======== CHANNEL f1 ========
NUC1 1H
P1 12.00 usec
PL1 -5.00 dB
SFO1 500.2235015 MHz

F2 - Processing parameters
SI 65536
SF 500.2199990 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 4.00

1D NMR plot parameters
CX 22.80 cm
CY 402.52 cm
F1P 8.800 ppm
F1 4401.94 Hz
F2P 6.400 ppm
F2 3201.41 Hz
PPMCM 0.10526 ppm/cm
HZCM 52.65474 Hz/cm



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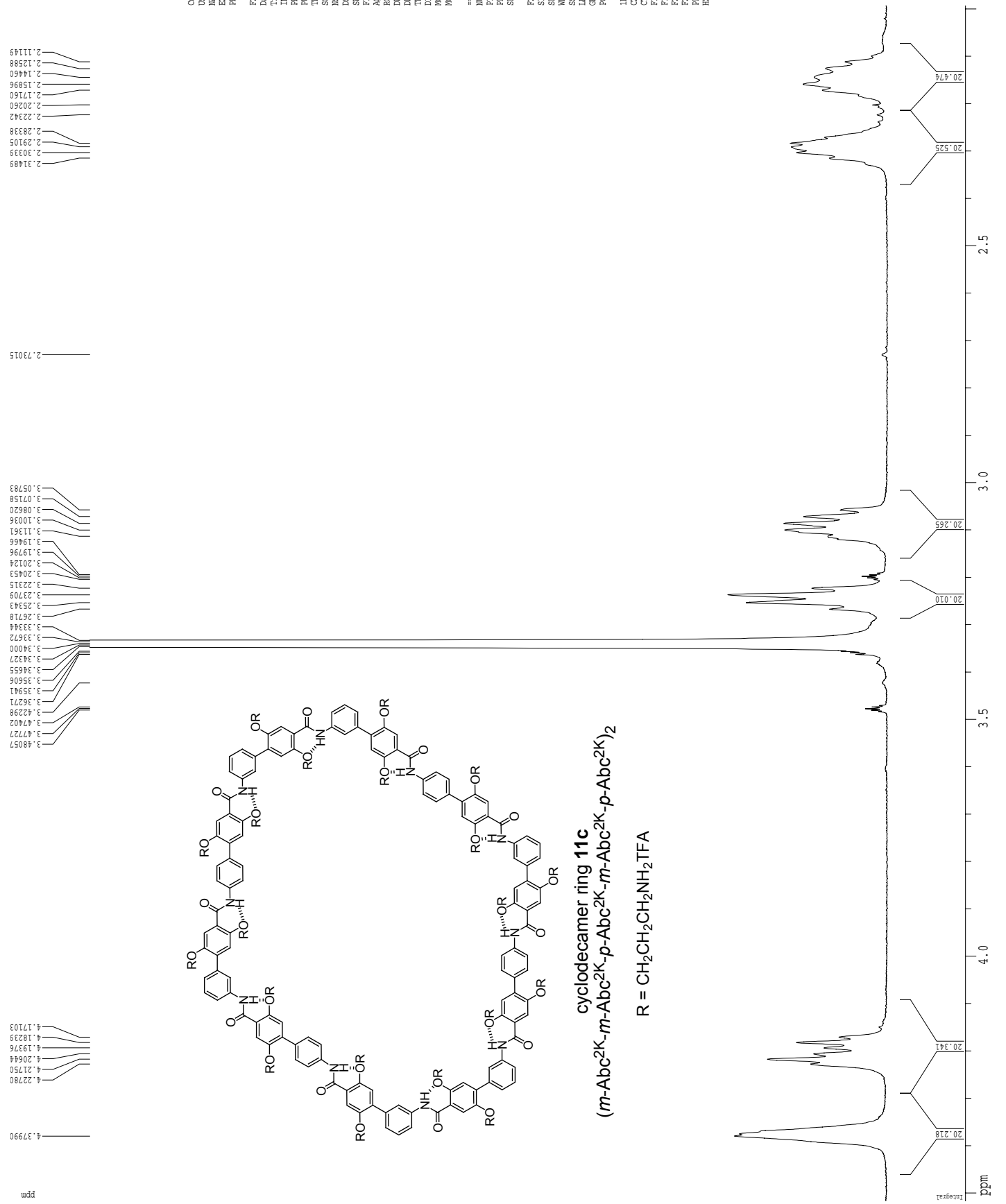
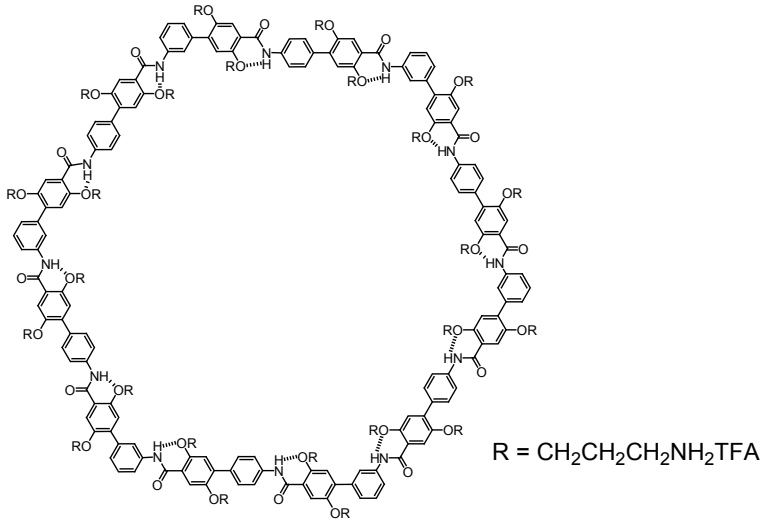
^{1}H NMR (500 MHz, 298 K, CD_3OD) spectrum of cyclodecamer ring **11c** (aliphatic region)



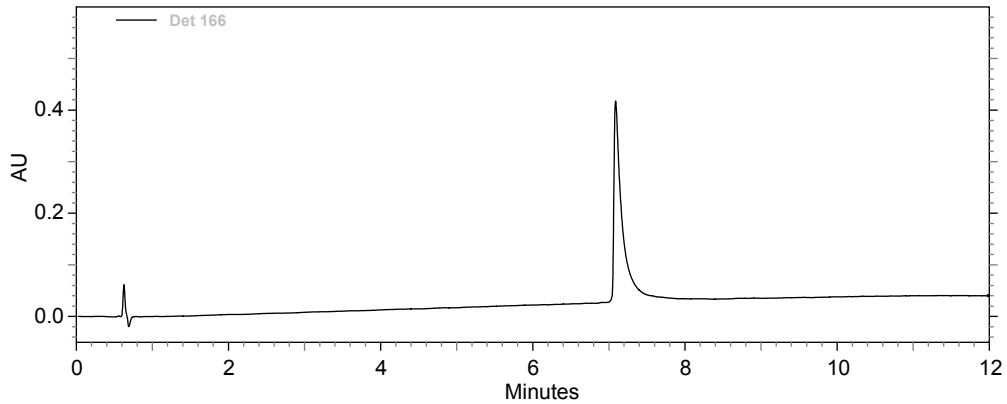
cyclodecamer ring **11c**
(*m*-Abc^{2K}-*m*-Abc^{2K}-*p*-Abc^{2K}-*m*-Abc^{2K}-*p*-Abc^{2K})$_2$

R = $CH_2CH_2CH_2NH_2$TFA

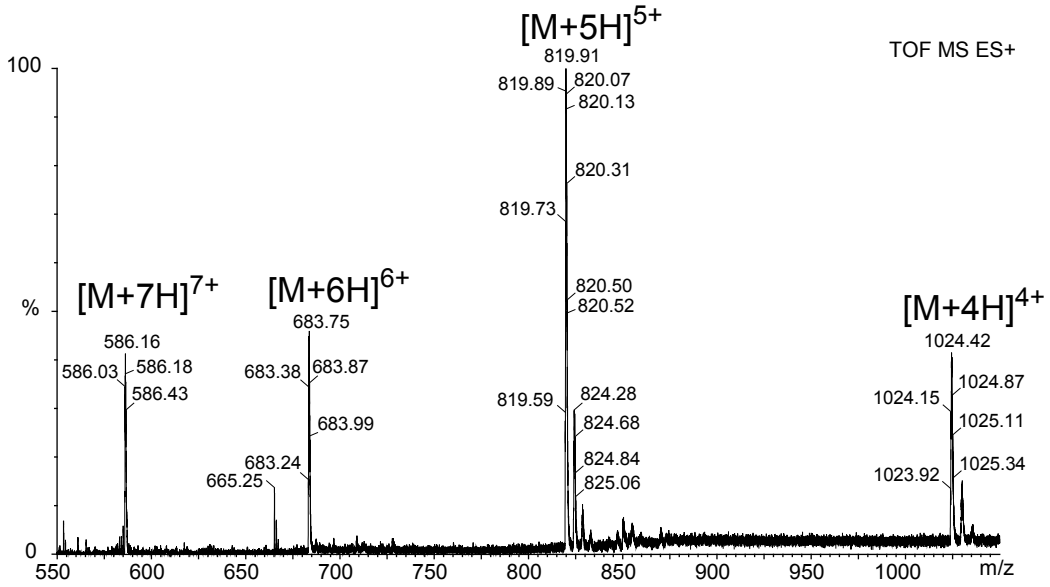
cyclododecamer ring: (*m*-Abc^{2K}-*p*-Abc^{2K})$_6$ (**11d**)
Analytical RP-HPLC chromatograph and mass spectrum (ESI-MS)



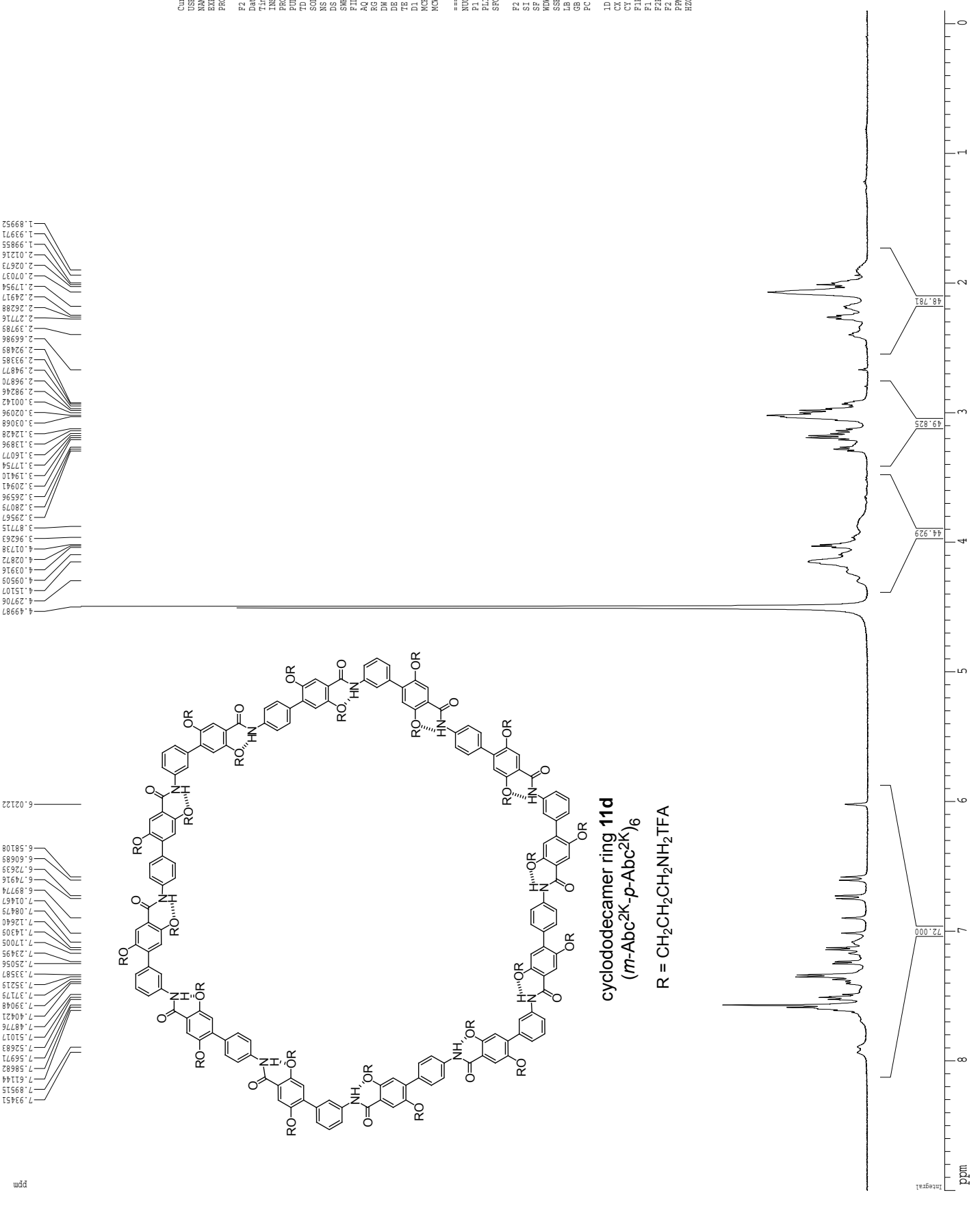
(a) Analytical RP-HPLC (5-50% acetonitrile with 0.1% TFA over 10 min, λ = 214)



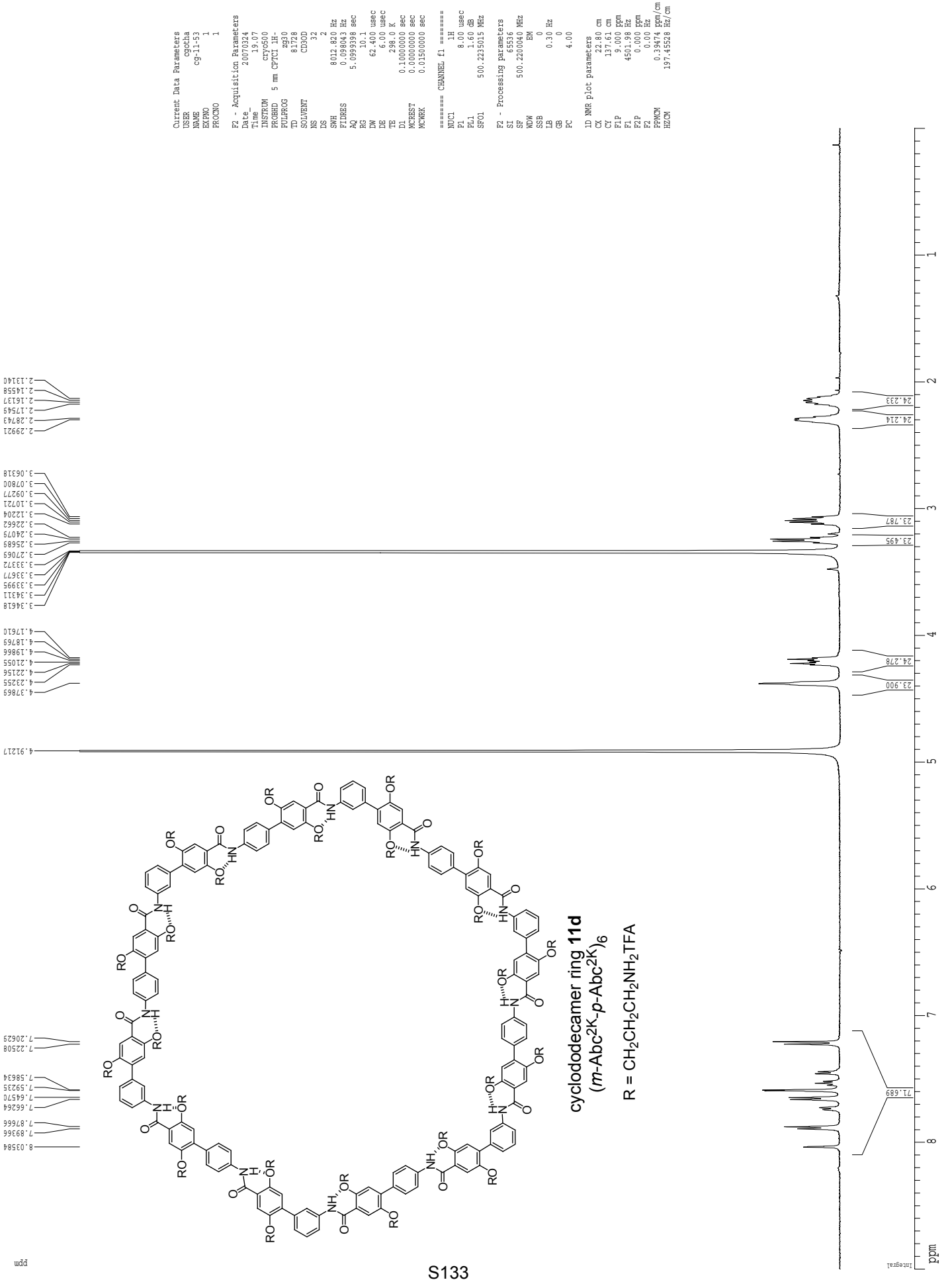
(b) ESI Mass spectrum. (Calcd exact mass for $C_{228}H_{276}N_{36}O_{36}$ [M] = 4094.09)



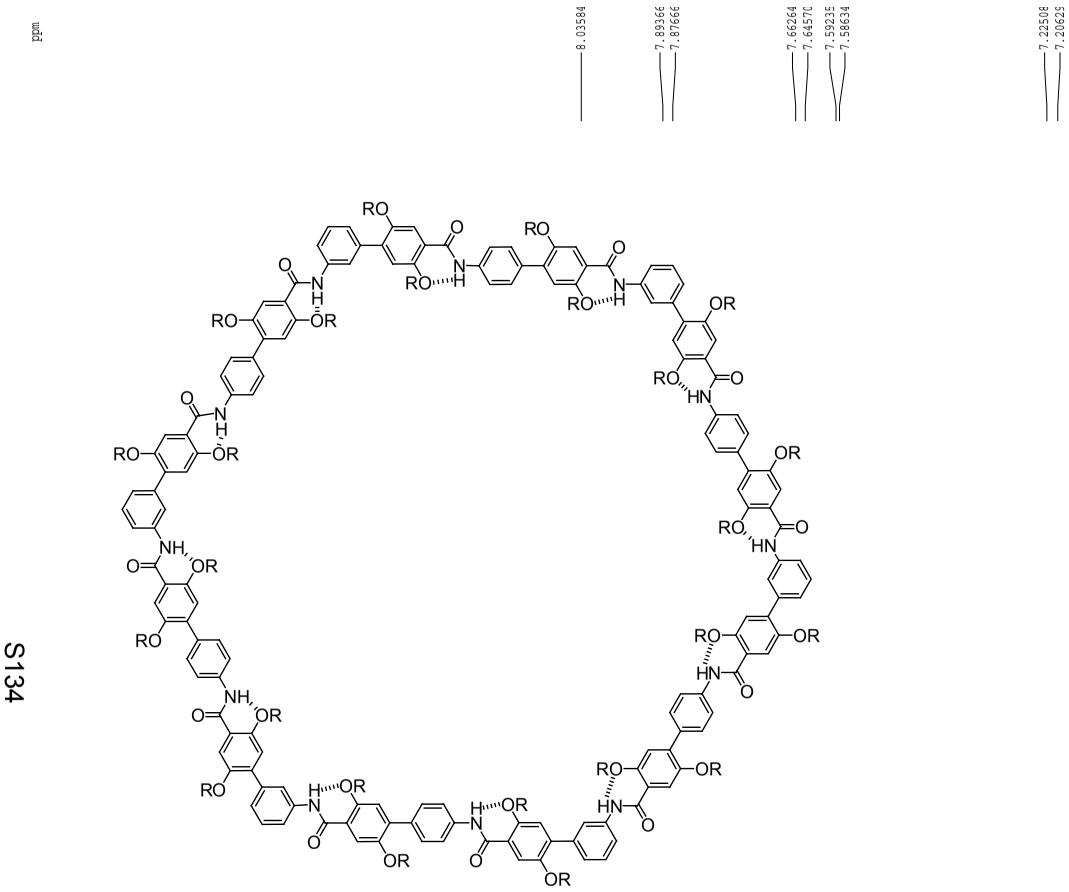
^{1}H NMR (500 MHz, 323 K, D_2O) spectrum of cyclododecamer ring **11d**



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



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



cyclododecamer ring **11d**
$(m\text{-Abc}^{2K}\text{-}p\text{-Abc}^{2K})_6$

R = $CH_2CH_2CH_2NH_2TFA$

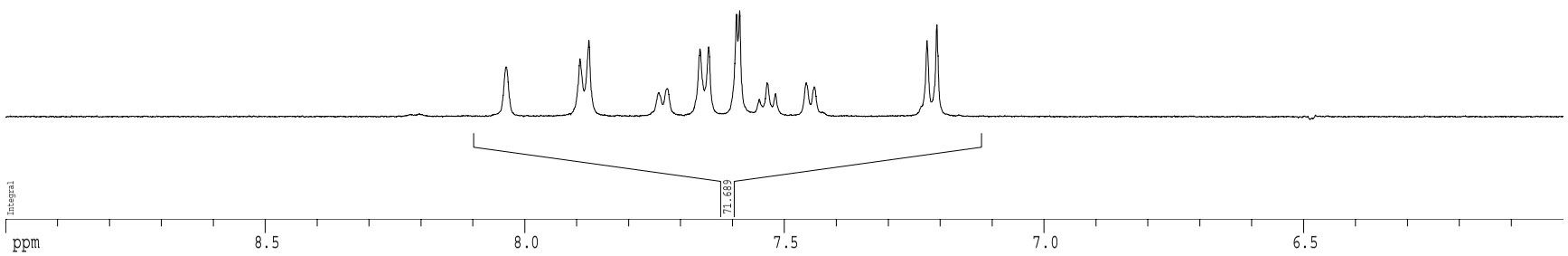
Current Data Parameters
USER cgotha
NAME cg-11-53
EXPNO 1
PROCNO 1

F2 - Acquisition Parameters
Date_ 20070324
Time 19.07
INSTRUM cryo500
PROBHD 5 mm CPTCI 1H-
PULPROG zg30
TD 81728
SOLVENT CD3OD
NS 32
DS 2
SWH 8012.820 Hz
FIDRES 0.098043 Hz
AQ 5.0999398 sec
RG 10.1
DW 62.400 usec
DE 6.00 usec
TE 298.0 K
D1 0.10000000 sec
MCREST 0.00000000 sec
MCWRK 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 137.61 cm
F1P 9.000 ppm
F1 4501.98 Hz
F2P 6.000 ppm
F2 3001.32 Hz
PPMCM 0.13158 ppm/cm
HZCM 65.81842 Hz/cm



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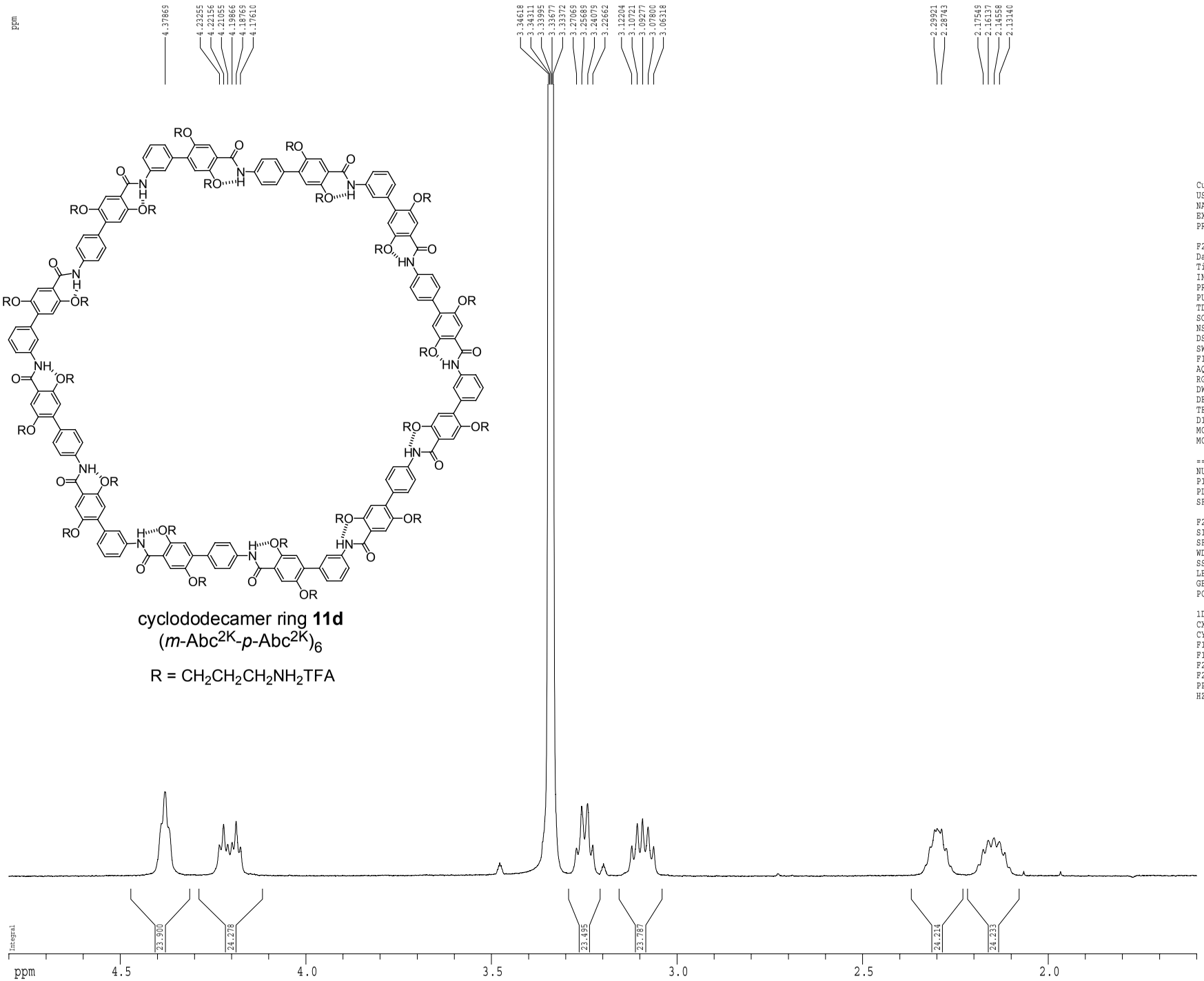
^{1}H NMR (500 MHz, 298 K, CD_3OD) spectrum of cyclododecamer ring **11d** (aliphatic region)

cyclododecamer ring **11d**
(*m*-Abc^{2K}-*p*-Abc^{2K})$_6$

R = $CH_2CH_2CH_2NH_2$TFA



Current Data Parameters
USER cgotha
NAME cg-11-53
EXPNO 1
PROCNO 1

F2 - Acquisition Parameters
Date_ 20070324
Time 19.07
INSTRUM cryo500
PROBHD 5 mm CPTCI 1H-
PULPROG zg30
TD 81728
SOLVENT CD3OD
NS 32
DS 2
SWH 8012.820 Hz
FIDRES 0.098043 Hz
AQ 5.0999398 sec
RG 10.1
DW 62.400 usec
DE 6.00 usec
TE 298.0 K
D1 0.10000000 sec
MCREST 0.00000000 sec
MCWRK 0.01500000 sec

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

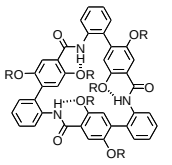
F2 - Processing parameters
SI 65536
SF 500.2200040 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 4.00

1D NMR plot parameters
CX 22.80 cm
CY 137.61 cm
F1P 4.800 ppm
F1 2401.06 Hz
F2P 1.600 ppm
F2 800.35 Hz
PPMCM 0.14035 ppm/cm
HZCM 70.20632 Hz/cm

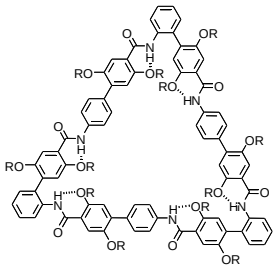
C. M. Gothard and J. S. Nowick

Molecular Modeling Studies

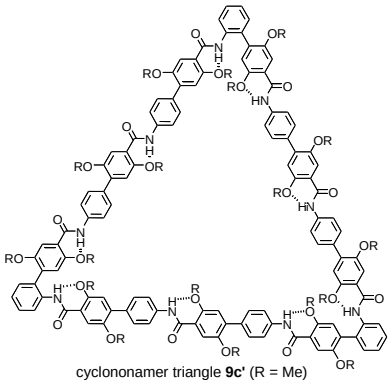
Macrocycles **9a-c**, **10a**,**b**, and **11a-d** were modeled as simplified homologues in which the propyloxyammonium ($OCH_2CH_2CH_2NH_3^+$) side chains were replaced with methoxy (OMe) groups (**9a'-c'**, **10a'**,**b'**, and **11a'-d'**). Each molecule was modeled using Maestro/MacroModel v8.5 with 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.



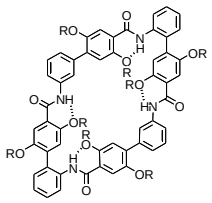
cyclotrimer triangle **9a'** (R = Me)



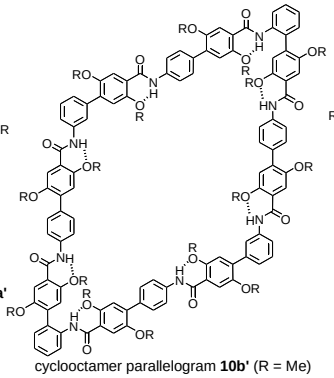
cyclohexamer triangle **9b'** (R = Me)



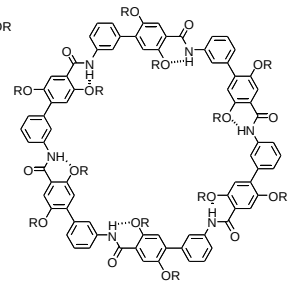
cyclononamer triangle **9c'** (R = Me)



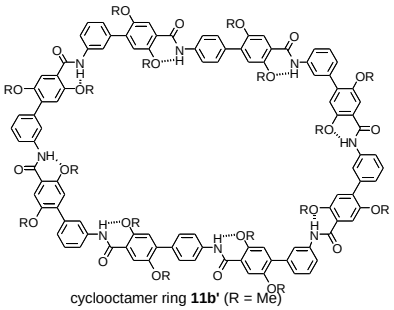
cyclotetramer parallelogram **10a'** (R = Me)



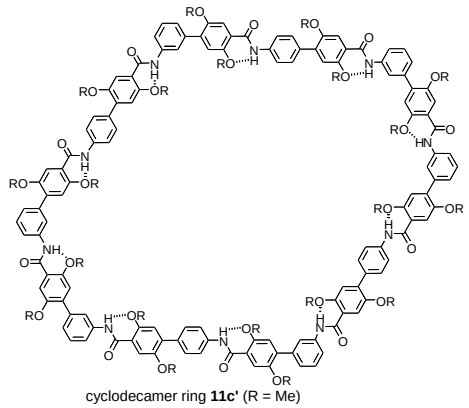
cyclooctamer parallelogram **10b'** (R = Me)



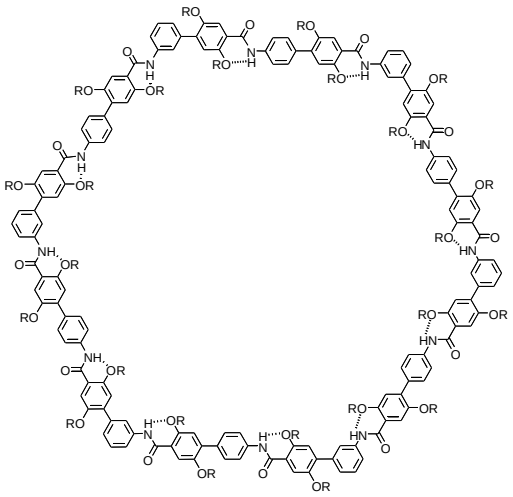
cyclohexamer ring **11a'** (R = Me)



cyclooctamer ring **11b'** (R = Me)



cyclodecamer ring **11c'** (R = Me)

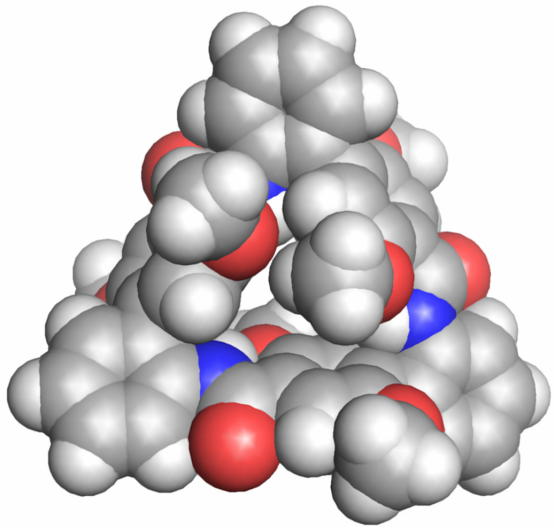


cyclododecamer ring **11d'** (R = Me)

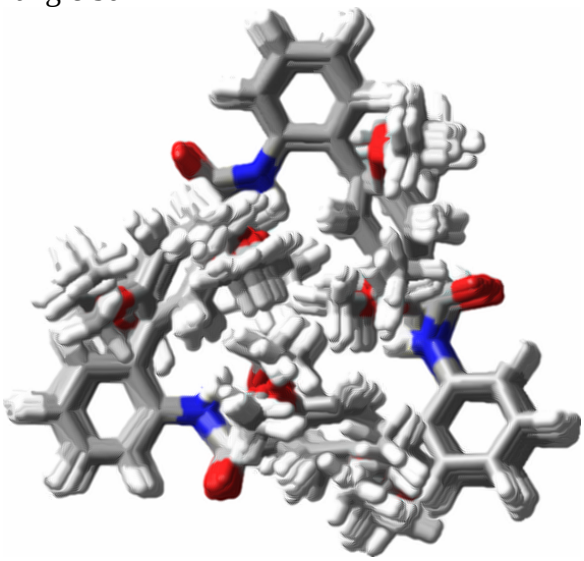
The modeling should be interpreted with several caveats: (1) The MMFF and MMFFs force fields lack good parameters for some of the stretches, bends, and torsions associated with the structures. (2) The MMFFs force field is designed to enforce planarity of the amide nitrogen atoms and may therefore overemphasize the conformational regularity of the structures. (3) The absence of H_2O 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'**

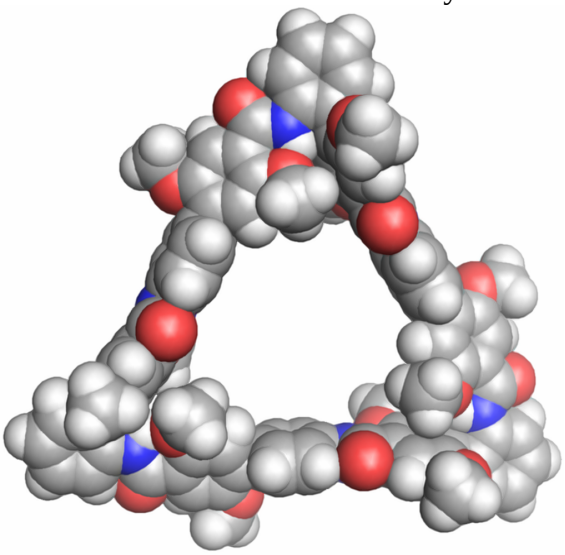


lowest energy conformer found

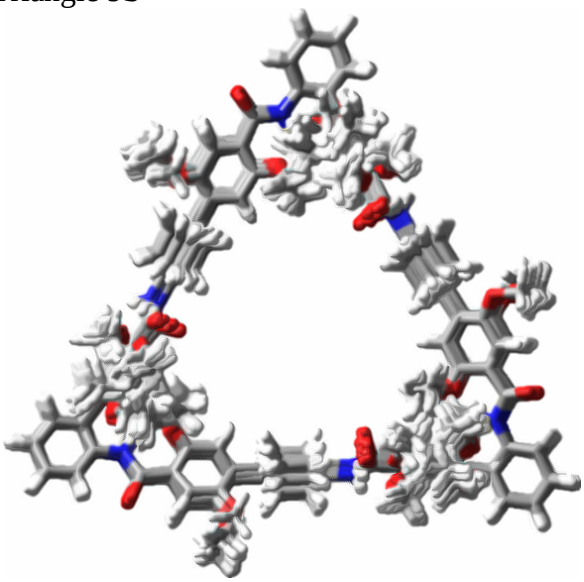


superposition of conformers found within lowest 5.00 kJ/mol (39 conformers).

Cyclohexamer Triangle **9b'**

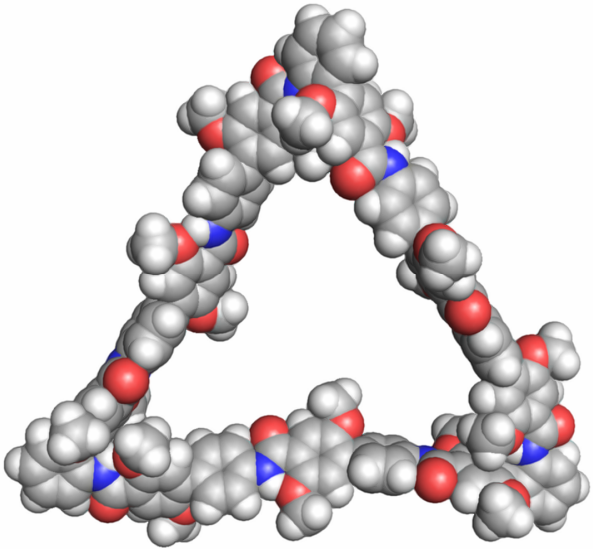


lowest energy conformer found

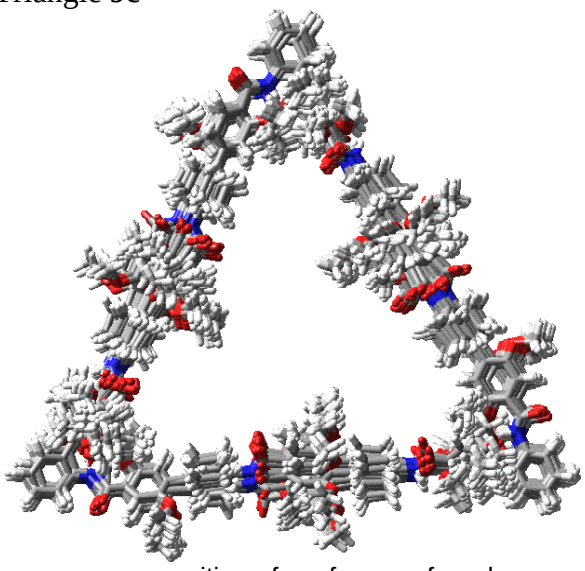


superposition of conformers found within lowest 5.00 kJ/mol (51 conformers).

Cyclononamer Triangle **9c'**

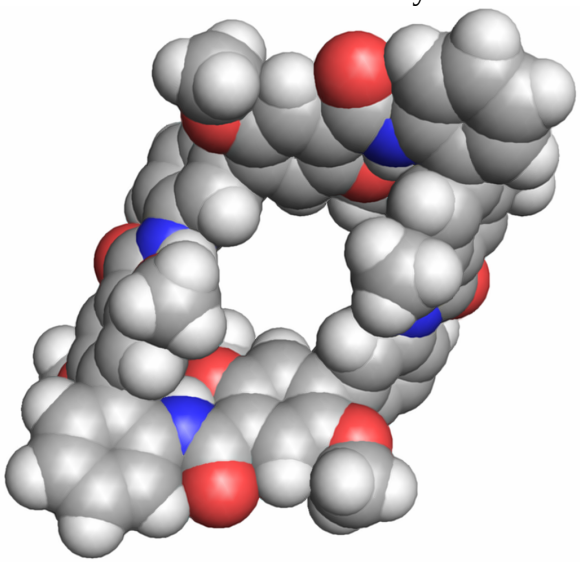


lowest energy conformer found

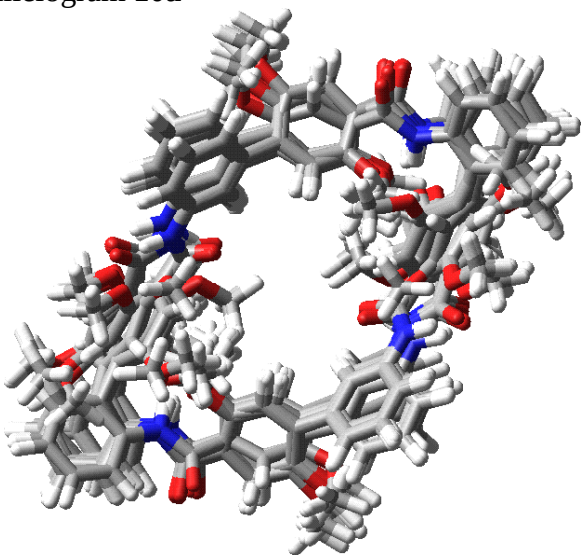


superposition of conformers found
within lowest 5.00 kJ/mol (80 conformers).

Cyclotetramer Parallelogram **10a'**

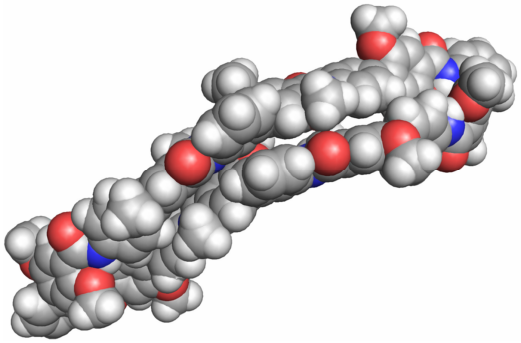


lowest energy conformer found

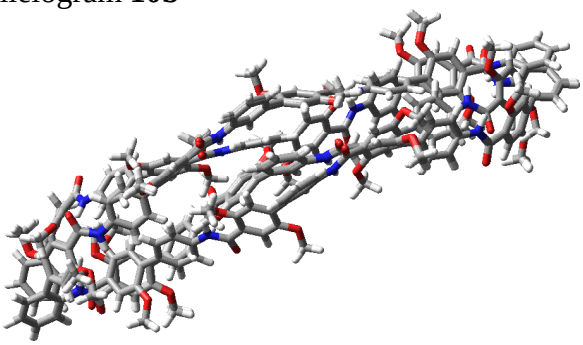


superposition of conformers found
within lowest 5.00 kJ/mol (15 conformers).

Cyclooctamer Parallelogram **10b'**

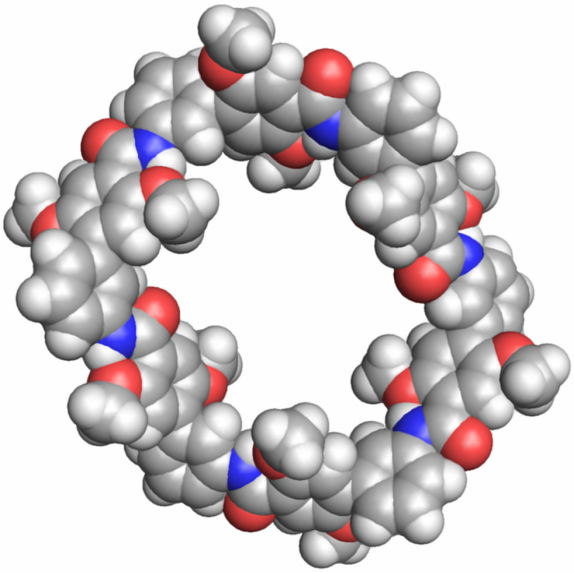


lowest energy conformer found

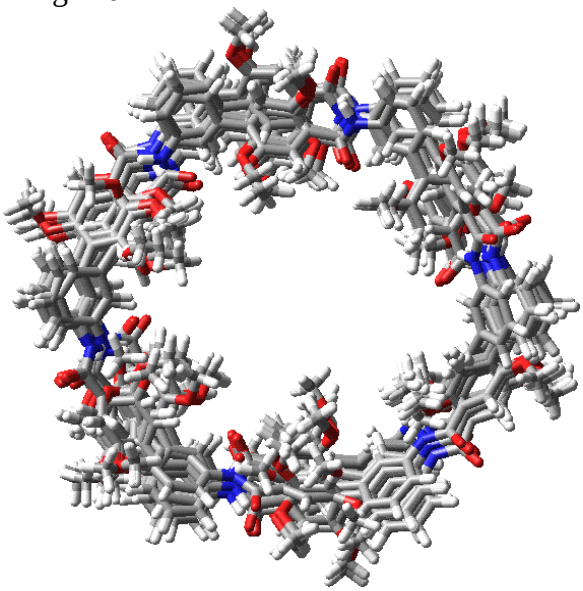


superposition of conformers found
within lowest 5.00 kJ/mol (3 conformers).

Cyclohexamer Ring **11a'**

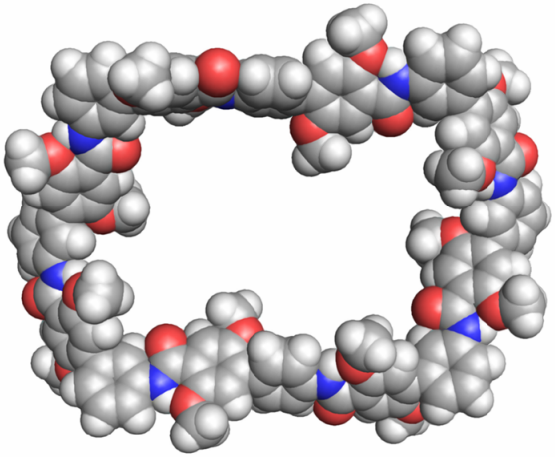


lowest energy conformer found

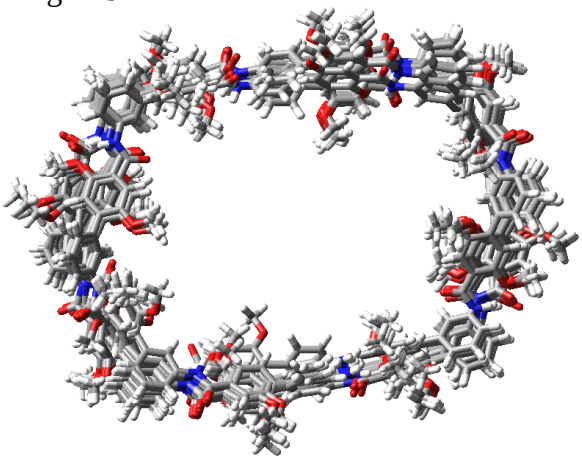


superposition of conformers found
within lowest 5.00 kJ/mol (19 conformers).

Cyclooctamer Ring **11b'**

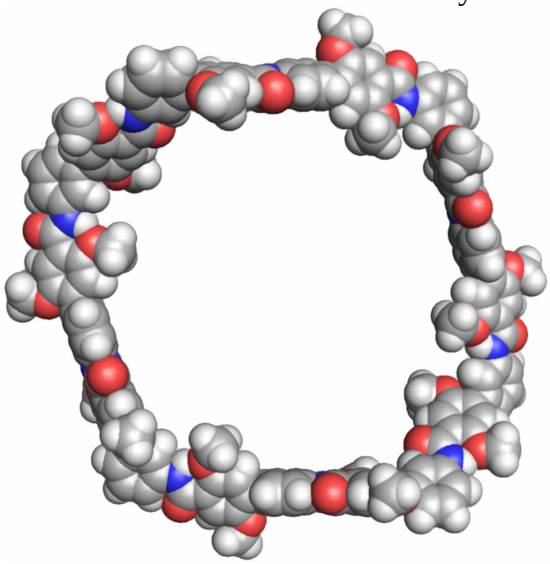


lowest energy conformer found

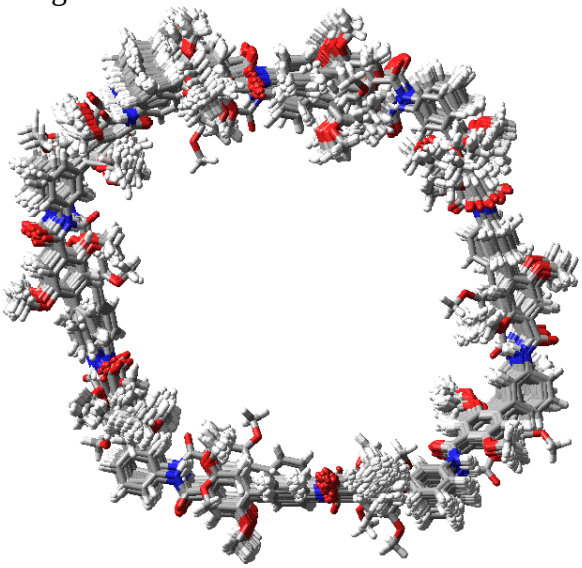


superposition of conformers found
within lowest 5.00 kJ/mol (21 conformers).

Cyclodecamer Ring **11c'**

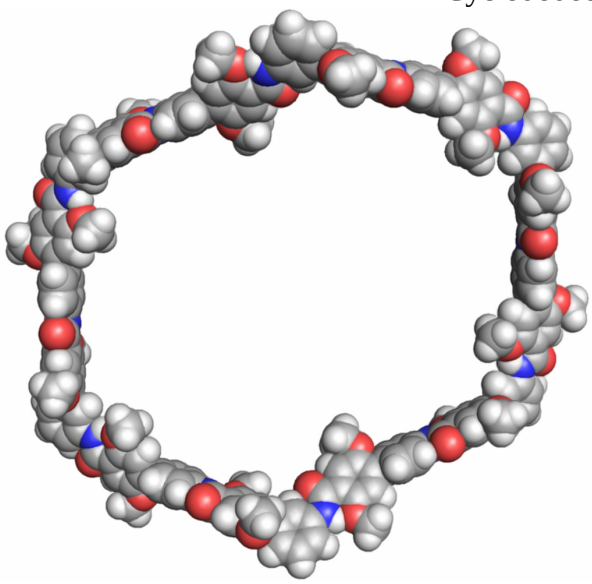


lowest energy conformer found

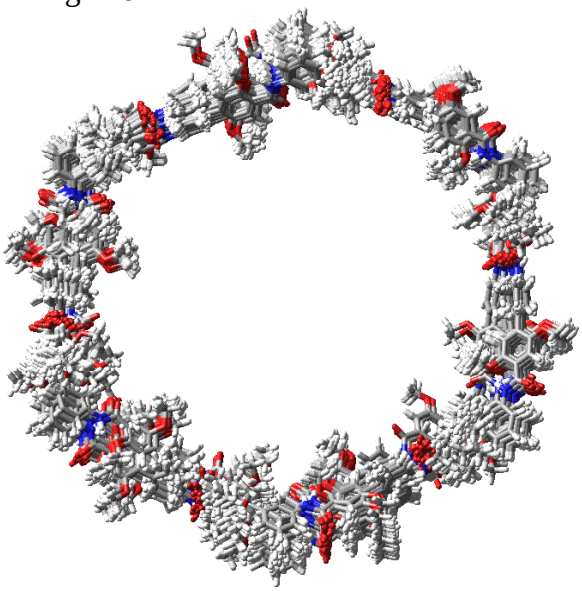


superposition of conformers found
within lowest 5.00 kJ/mol (173 conformers).

Cyclododecamer Ring **11d'**



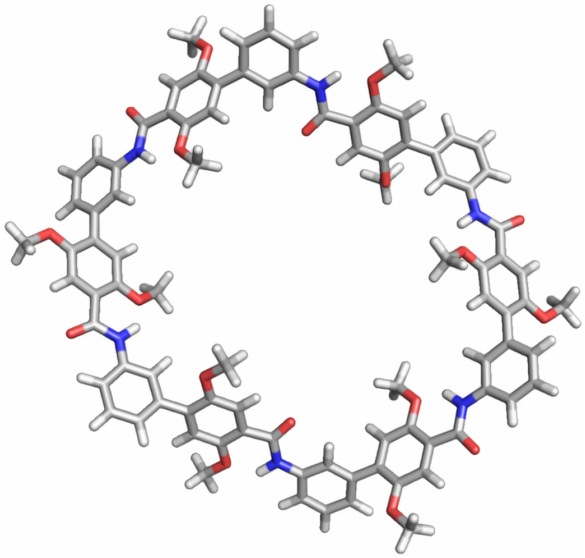
lowest energy conformer found



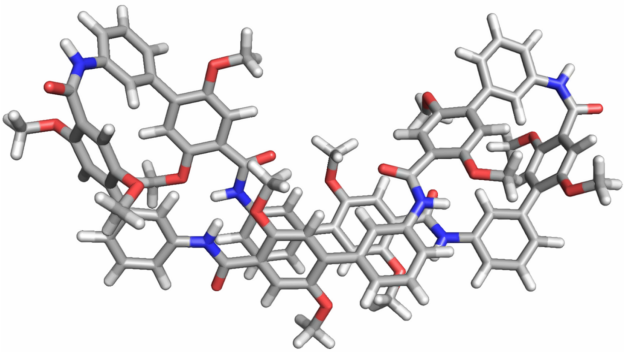
superposition of conformers found
within lowest 5.00 kJ/mol (235 conformers).

Cyclohexamer Ring **11a'** (*cttctt*- and *tttttt*-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 *tttttt*-conformer.



tttttt-conformer of **11a'**



cttctt-conformer of **11a'**