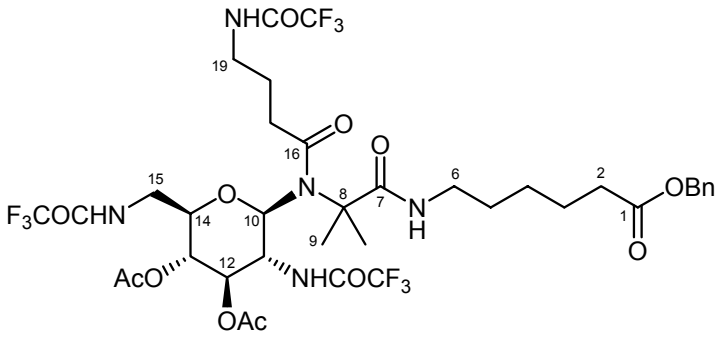
Supporting Information

Synthesis of Multivalent Aminoglycoside Mimics via the Ugi Multicomponent Reaction

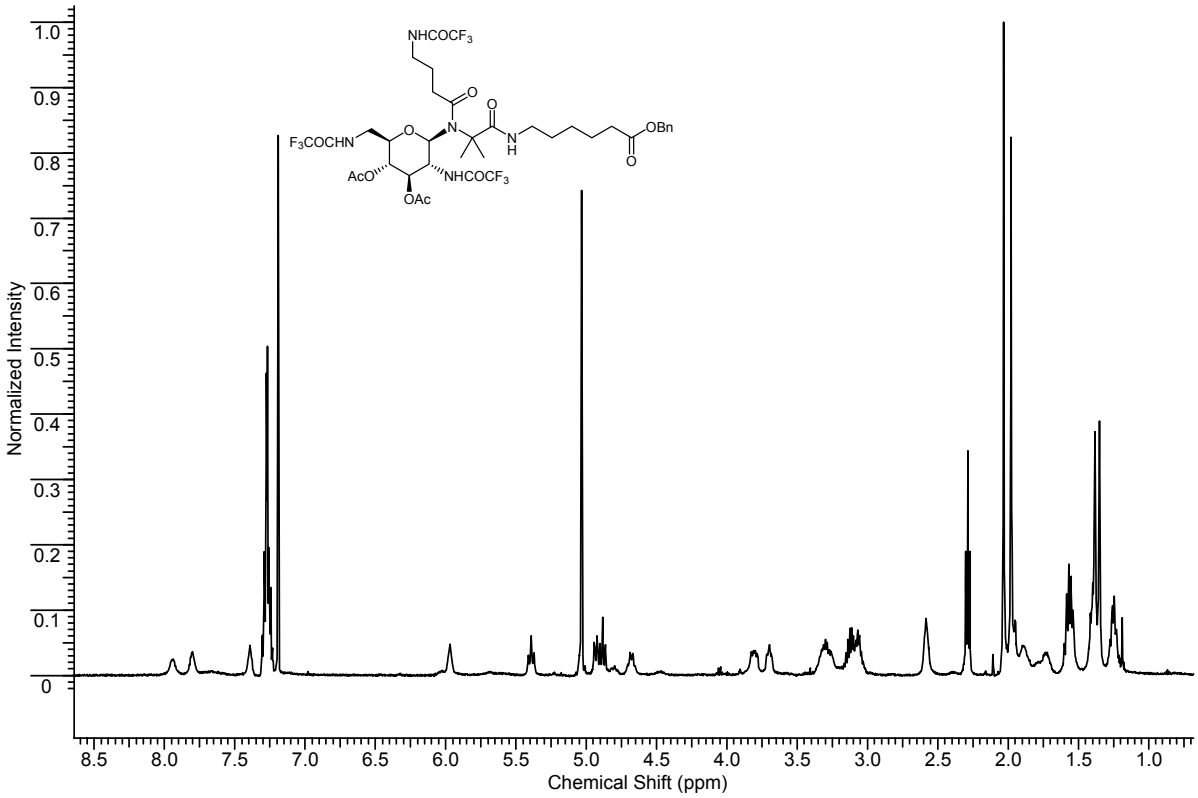
Bernhard Westermann,* and Simon Dörner

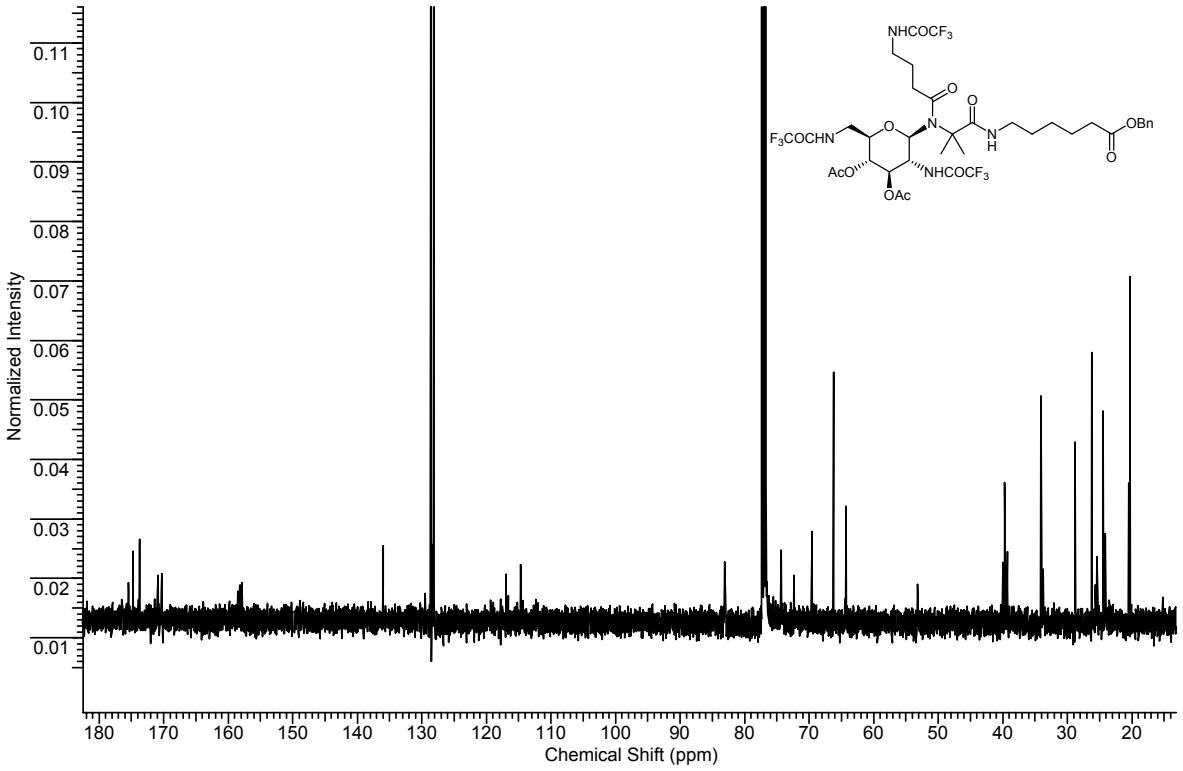
Leibniz Institute of Plant Biochemistry, Department of Bioorganic Chemistry, Weinberg 3, 06120 Halle (Saale), Germany. Fax: 49 345 5582 1309; Tel: 49 345 5582 1340; E-mail: bwesterm@ipb-halle.de

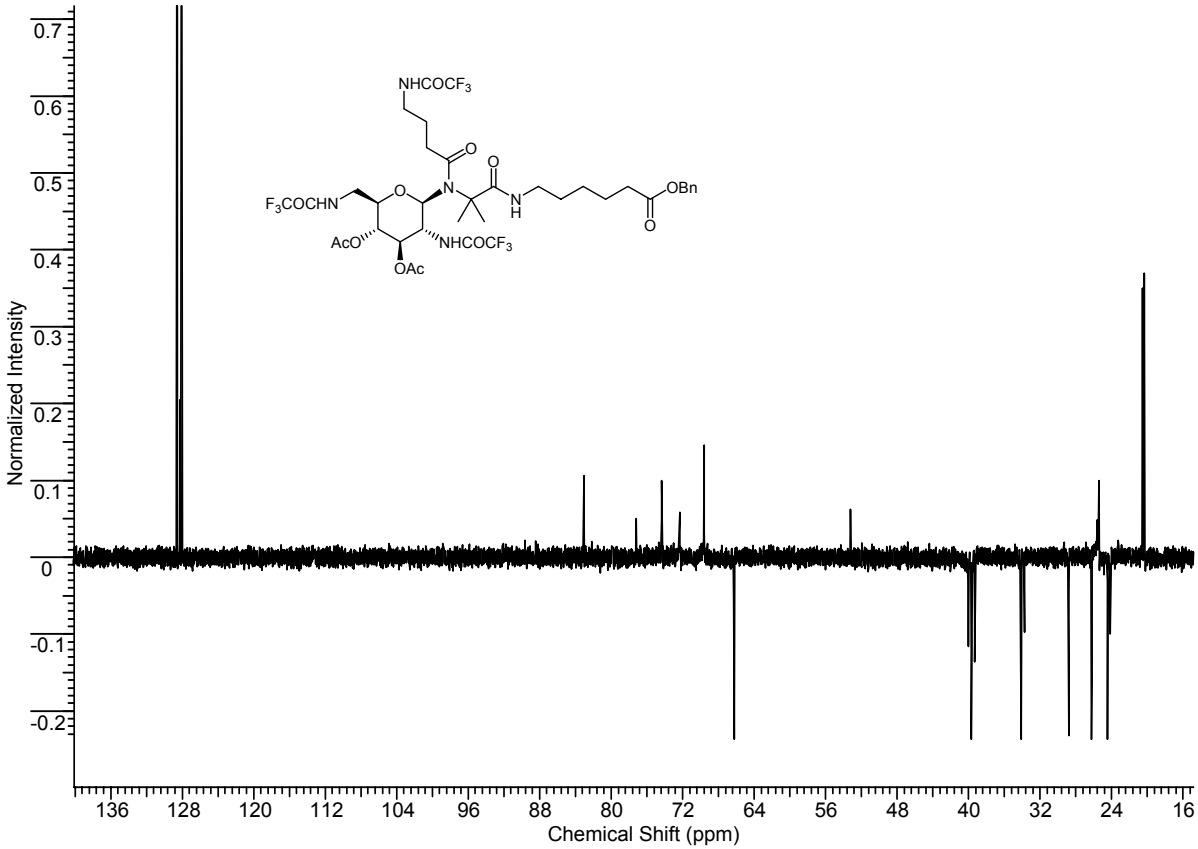
12,13-Di-*O*-acetyl-*N*-(7-{[1-(benzyloxy)-1-oxohexyl]amino}-8,8-dimethyl-7-oxoethyl)-11,15-dideoxy-11,15-bis[(trifluoroacetyl)amino]-*N*-{19-[(trifluoroacetyl)amino]butanoyl}-β-D-glucopyranosylamin (16)



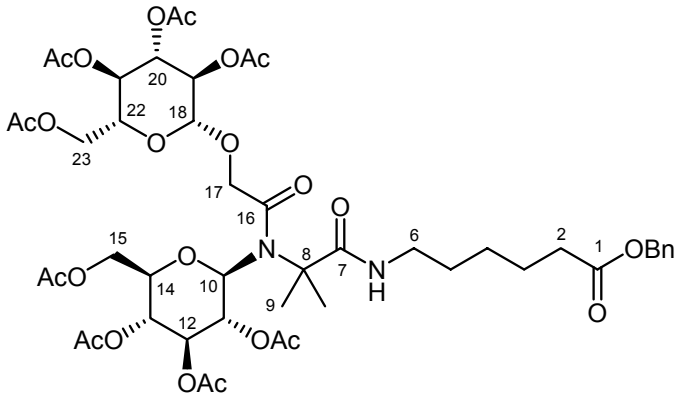
^{1}H-NMR (500 MHz, $CDCl_3$): δ [ppm] = 1.21–1.29 (m, 2H, 4-H), 1.33–1.36 (m, 8H, 5-H, 9-H), 1.51–1.60 (m, 2H, 3-H), 1.68–1.77 (m, 2H, 18-H), 1.88 (s, 3H, $CH_{3(Ac)}$), 2.03 (s, 3H, $CH_{3(Ac)}$), 2.26–2.32 (t, *J* = 7.4 Hz, 2H, 2-H), 2.54–2.62 (m, 2H, 17-H), 3.00–3.17 (m, 3H, 15-H^{a}, 6-H), 3.22–3.38 (m, 2H, 19-H), 3.66–3.73 (m, 1H, 14-H), 3.76–3.86 (m, 1H, 15-H^{b}), 4.64–4.72 (m, 1H, 11-H), 4.85–4.91 (pt, *J* = 9.8 Hz, 1H, 13-H), 4.91–4.96 (d, *J* = 10.0 Hz, 1H, 10-H), 5.02–5.05 (s, 2H, CH_2 benzyl.), 5.36–5.43 (pt, *J* = 9.6 Hz, 1H, 12-H), 5.94–6.00 (bs, 1H, 1'-N*H*), 7.22–7.31 (m, 5H, CH arom.), 7.36–7.42 (bs, 1H, 19-N*H*), 7.76–7.84 (bs, 1H, 15-N*H*), 7.90–7.99 (bs, 1H, 11-N*H*); **^{13}C-NMR** (125 MHz, $CDCl_3$): δ [ppm] = 20.31, 20.50 (q, $CH_{3(Ac)}$), 24.09 (t, C-18), 24.41 (t, C-3), 25.41, 25.60 (q, C-9), 26.18 (t, C-4), 28.76 (t, C-5), 33.73 (t, C-17), 34.07 (t, C-2), 39.28 (t, C-19), 39.65 (t, C-6), 39.97 (t, C-15), 53.17 (d, C-11), 66.21 (t, CH_2 benzyl.), 69.58 (d, C-13), 72.32 (d, C-12), 74.32 (d, C-14), 83.05 (d, C-10), 115.50 (m, CF_3), 128.07, 128.22, 128.56 (d, CH arom.), 136.00 (s, C arom.), 158.25 (m, *C*(O)CF_3), 170.28, 170.85, 173.72, 174.74, 175.43 (s, CO); **HRMS (ESI)** calcd for $C_{37}H_{46}F_9N_5O_{12}Na$ [M+Na^{+}]: 946.2891, found: 946.2875


NHCOCF3
F3COCHN
AcO
NHCOCF3
OAc
OBn
Normalized Intensity
Chemical Shift (ppm)

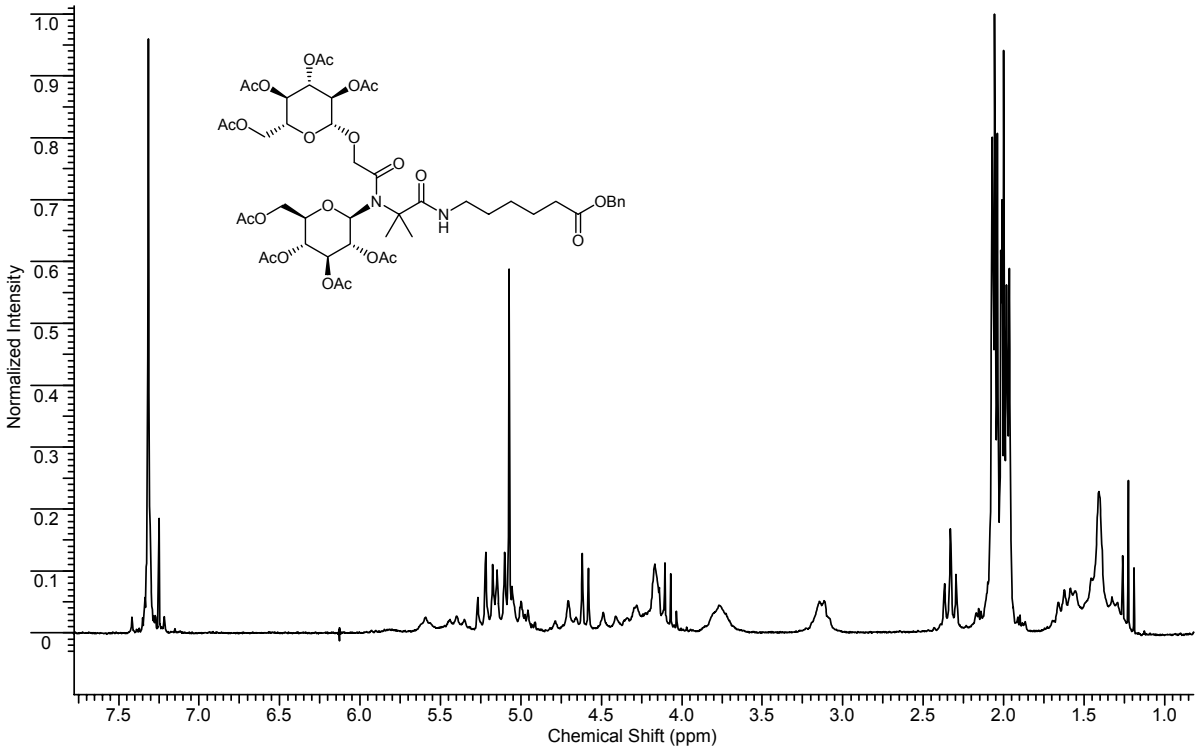

NHCOCF3
F3COCHN
AcO
NHCOCF3
OAc
OBn
Normalized Intensity
Chemical Shift (ppm)

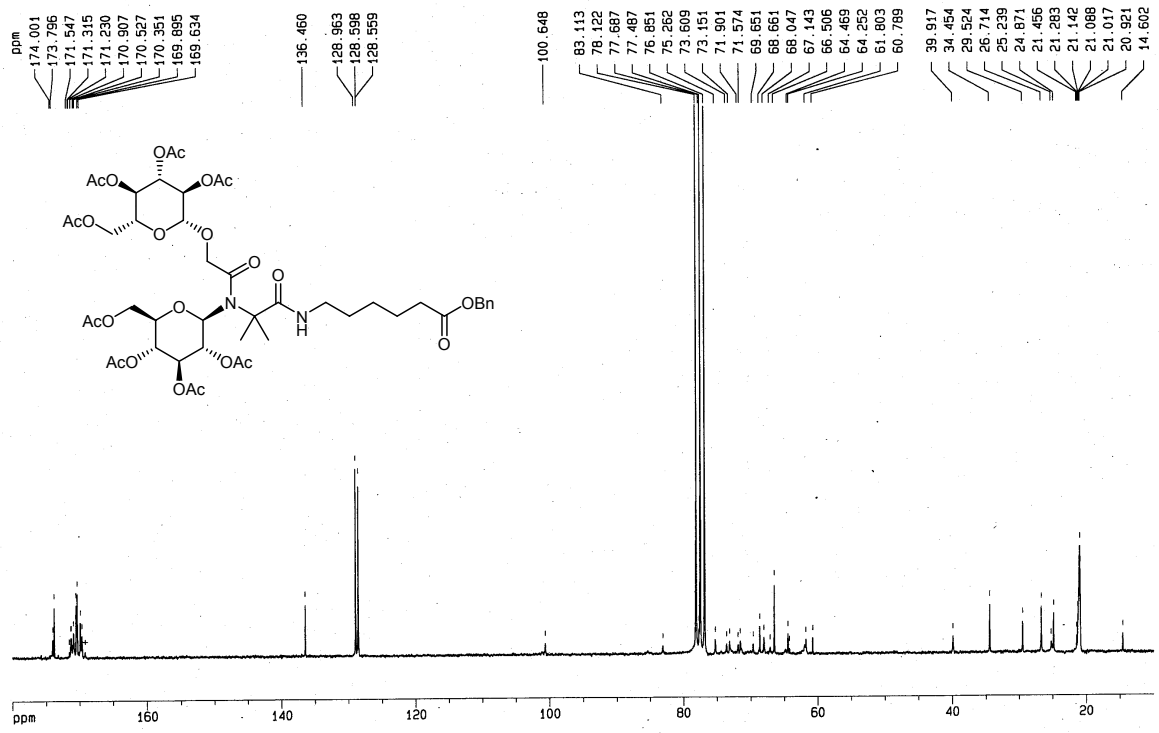

NHCOCF3
O
O
N
F3COCHN
O
AcO
NHCOCF3
OAc
N
H
OBn
O
Normalized Intensity
0.7
0.6
0.5
0.4
0.3
0.2
0.1
0
-0.1
-0.2
136
128
120
112
104
96
88
80
72
64
56
48
40
32
24
16
Chemical Shift (ppm)

2,3,4,6-Tetra-*O*-acetyl-*N*-(2-{[6-(benzyloxy)-6-oxohexyl]amino}-1,1-dimethyl-2-oxoethyl)-*N*-{[(2,3,4,6-tetra-*O*-acetyl-β-D-glucopyranosyl)oxy]acetyl}-β-D-glucopyranosylamine (17)



^{1}H-NMR (200 MHz, $CDCl_3$): δ [ppm] = 1.25–1.72 (m, 12H), 1.96, 1.98, 2.00, 2.01, 2.02, 2.04, 2.06, 2.07 (s, 3H), 2.33 (t, *J* = 7.3 Hz, 2H), 3.04–3.22 (m, 2H), 3.66–3.88 (m, 2H), 4.11–5.91 (m, 17H), 7.31 (s, 5H); **^{13}C-NMR** (50 MHz, $CDCl_3$): δ [ppm] = 20.92, 21.02, 21.09, 21.14, 21.28, 21.46 (q, $CH_{3(Ac)}$), 24.87 (t, C-3), 25.22 (q, C-9), 26.71, 29.53, 34.46, 39.92 (t, C-2, C-4–C-6), 61.79, 61.97 (t, OCH_2), 64.47 (s, C-8), 66.52, 67.13 (t, OCH_2), 68.03, 68.64, 69.63, 71.55, 71.89, 73.14, 73.59, 75.26 (d, C-11–C-14, C-19–C-22), 83.10 (d, C-10), 100.65 (d, C-18), 128.56, 128.60, 128.96 (d, CH arom.), 136.46 (s, C arom.), 167.63, 169.90, 170.35, 170.53, 170.91, 171.23, 171.32, 171.55, 173.80, 174.00 (s, CO); **HRMS (ESI)** calcd for $C_{47}H_{64}N_2O_{23}Na$ [M+Na^+]: 1047.3792, found: 1047.3805; Anal. calcd for $C_{47}H_{64}N_2O_{23}$: C, 55.07; H, 6.29; N, 2.73; found: C, 54.64; H, 6.12; N, 2.76


Normalized Intensity
Chemical Shift (ppm)
OAc
AcO
OBn
O
N
H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171.315
171.230
170.907
170.527
170.351
169.895
169.634
136.460
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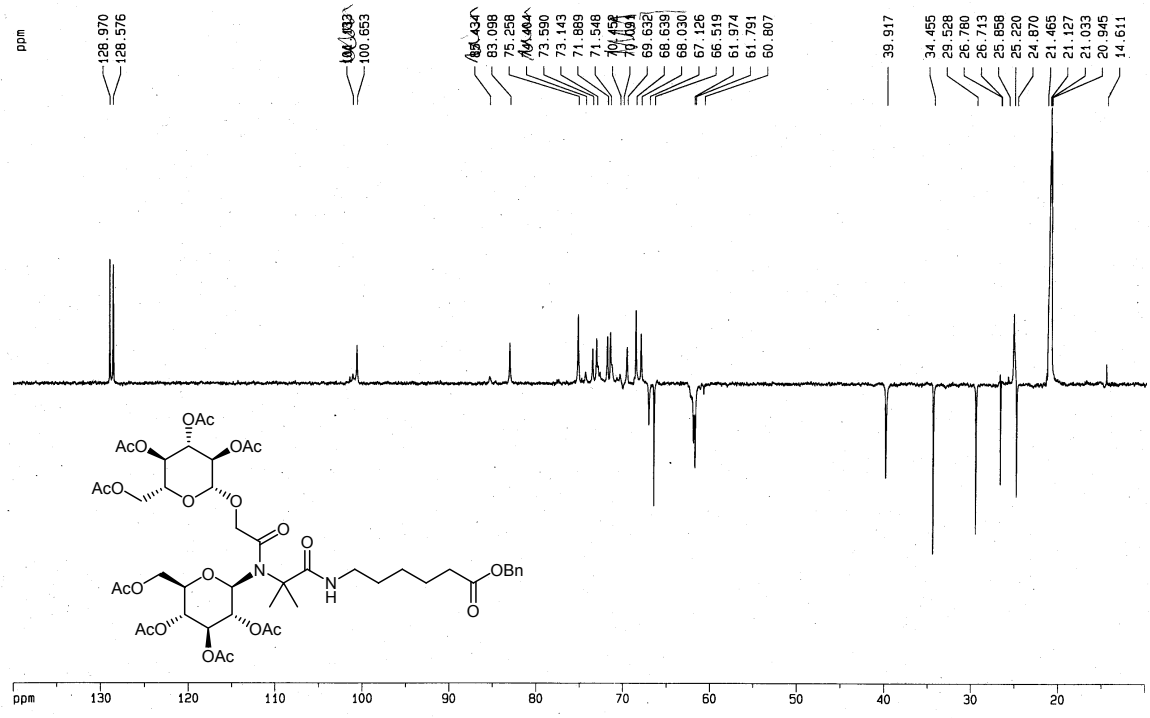
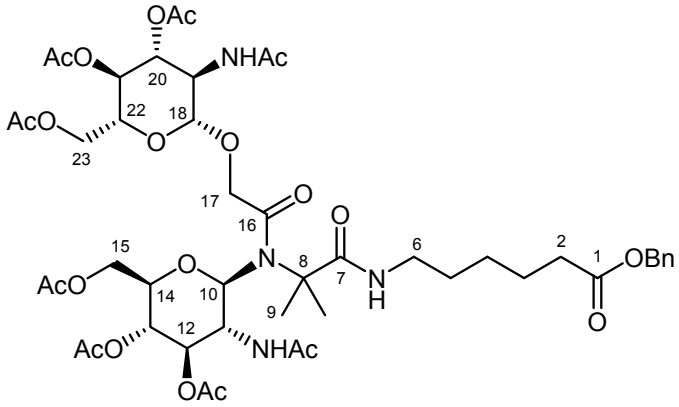
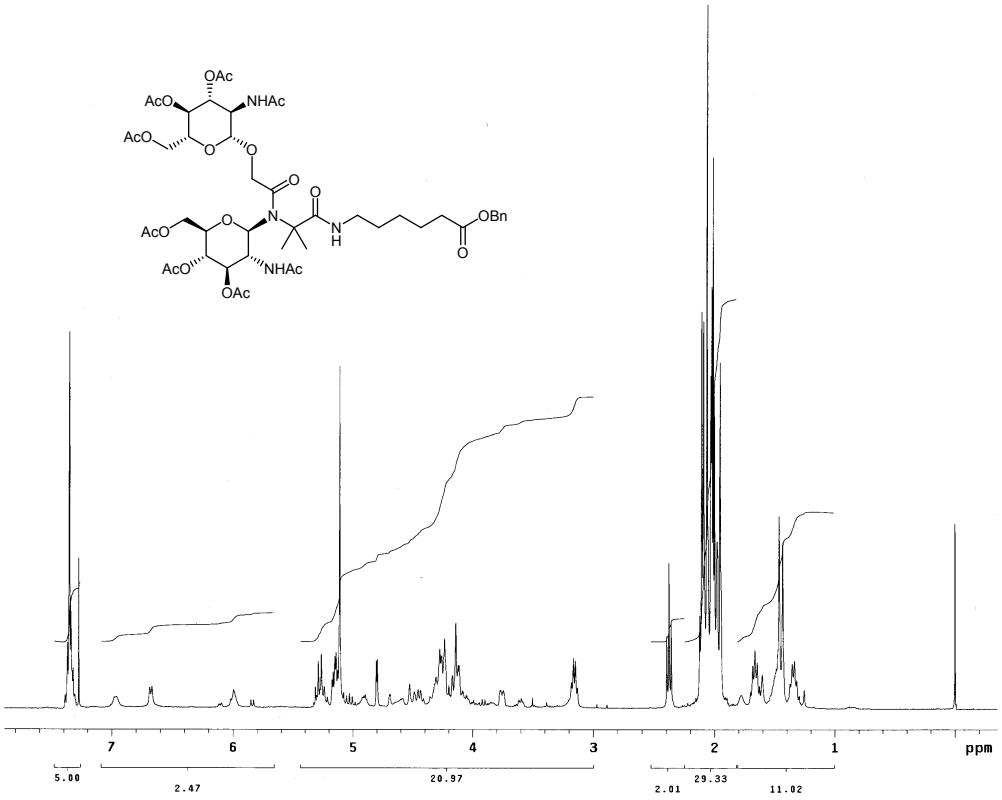
128.598
128.559
100.648
83.113
78.122
77.687
77.487
76.851
75.262
73.609
73.151
71.901
71.574
69.651
68.661
68.047
67.143
66.506
64.469
64.252
61.803
60.789
39.917
34.454
29.524
26.714
25.239
24.871
21.456
21.283
21.142
21.088
21.017
20.921
14.602
OAc
AcO
OBn
O
N
H


ppm
128.970
128.576
100.653
83.098
75.258
73.590
73.143
71.889
71.546
69.632
68.639
68.030
67.126
66.519
61.974
61.791
60.807
39.917
34.455
29.528
26.780
26.713
25.858
25.220
24.870
21.465
21.127
21.033
20.945
14.611
AcO
OAc
AcO
O
O
O
N
O
N
H
OBn
O
AcO
O
AcO
OAc
OAc
ppm
130
120
110
100
90
80
70
60
50
40
30
20

3,4,6-Tri-*O*-acetyl-2-(acetylamino)-*N*-(2-{[6-(benzyloxy)-6-oxohexyl]amino}-1,1-dimethyl-2-oxoethyl)-2-deoxy-*N*-({[3,4,6-tri-*O*-acetyl-2-(acetylamino)-2-deoxy-β-D-glucopyranosyl]oxy}acetyl)-β-D-glucopyranosylamine (18)



^{1}H-NMR (500 MHz, $CDCl_3$): δ [ppm] = 1.20–1.84 (m, 12H), 1.95, 2.01, 2.02, 2.03, 2.06, 2.09, 2.10 (s, 3H), 2.38 (t, *J* = 7.4 Hz, 2H), 3.06–3.24 (m, 2H), 3.55–5.37 (m, 18H), 5.93–6.07 (m, 1H), 6.60–6.74 (m, 1H), 6.90–7.05 (m, 1H), 7.36 (s, 5H); **^{13}C-NMR** (125 MHz, $CDCl_3$): δ [ppm] = 20.50, 20.54, 20.60, 20.65, 20.69, 20.73 (q, $CH_{3(OAc)}$), 23.15, 23.20 (q, $CH_{3(NHAc)}$), 24.43, 26.25 (t, CH_2), 26.82 (q, C-9), 28.95, 34.04, 39.61 (t, CH_2), 51.74, 53.58 (d, C-11, C-19), 61.49, 61.71, 62.00, 66.11 (t, OCH_2), 64.24 (s, C-8), 67.50, 67.96, 68.43, 71.19, 72.96, 74.64 (d, C-11–C-14, C-19–C-22), 83.98 (d, C-10), 98.38 (d, C-18), 128.11, 128.52 (d, CH arom.), 135.99 (s, C arom.), 169.19, 169.37, 170.49, 170.79, 170.92, 171.23, 171.27, 171.86, 173.16, 173.44, 173.94 (s, CO); **HRMS (ESI)** calcd for $C_{47}H_{66}N_4O_{21}Na$ [M+Na^+]: 1045.4112, found: 1045.4114

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1H Standard-Parameter

Wed Jul 21 19:06 2004

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nwc/dos/dos247.1H.~
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probe           asw5
seqfil          s2pul
sfrq         399.937
tn                H1
at             2.562
np             65536
sw          12787.7
bs                 8
tpwr              58
pw               2.8
d1             1.000
d2                 0
tof            828.3
nt                40
ct                40
alock              n
gain        not used
      FLAGS
il                 n
in                nw
dp                 y
      DEC. & VT
dn                H1
dfrq         399.936
homo               n
dpwr              30
dof              0.0
dm                nnn
dmm                c
dmf              200
   PROCESSING
lb              0.40
wtfile
proc               ft
fn              65536

     DISPLAY
sp            -139.3
wp            3295.4
vs               160
sc                65
wc               225
hzmm          14.65
is            662.01
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rfp                0
th               4.1
ins            5.000
rp               5.4
lp            -199.0
nm    ph
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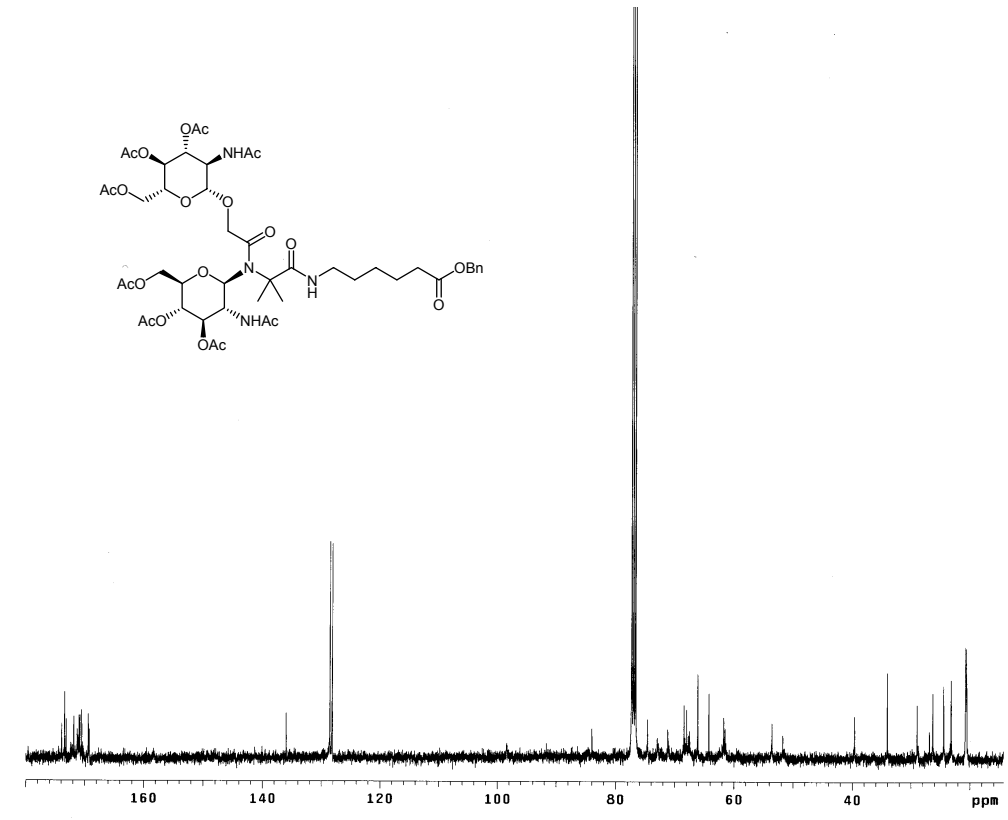


```
dos247.13C

Wed Jul 21 19:11 2004

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                .fid
   ACQUISITION
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probe           asw5
seqfil          s2pul
sfrq         100.574
tn                C13
at             1.311
np             67216
sw          25641.0
bs                16
tpwr              61
pw               4.0
d1             1.689
d2                 0
tof           1327.2
nt                6000
ct                6000
alock              n
gain        not used
      FLAGS
il                 n
in                 n
dp                 y
      DEC. & VT
dn                H1
dfrq         399.936
homo               n
dpwr              40
dof                0
dm               yyy
dmm                w
dmf             9350
pp              17.5
   PROCESSING
lb             1.00
wtfile
proc               ft
fn              65536

     DISPLAY
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wp           16658.4
vs               409
sc                65
wc               225
hzmm          74.04
rfl           9742.0
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th                3.4
rp               33.7
lp            -300.8
nm    ph
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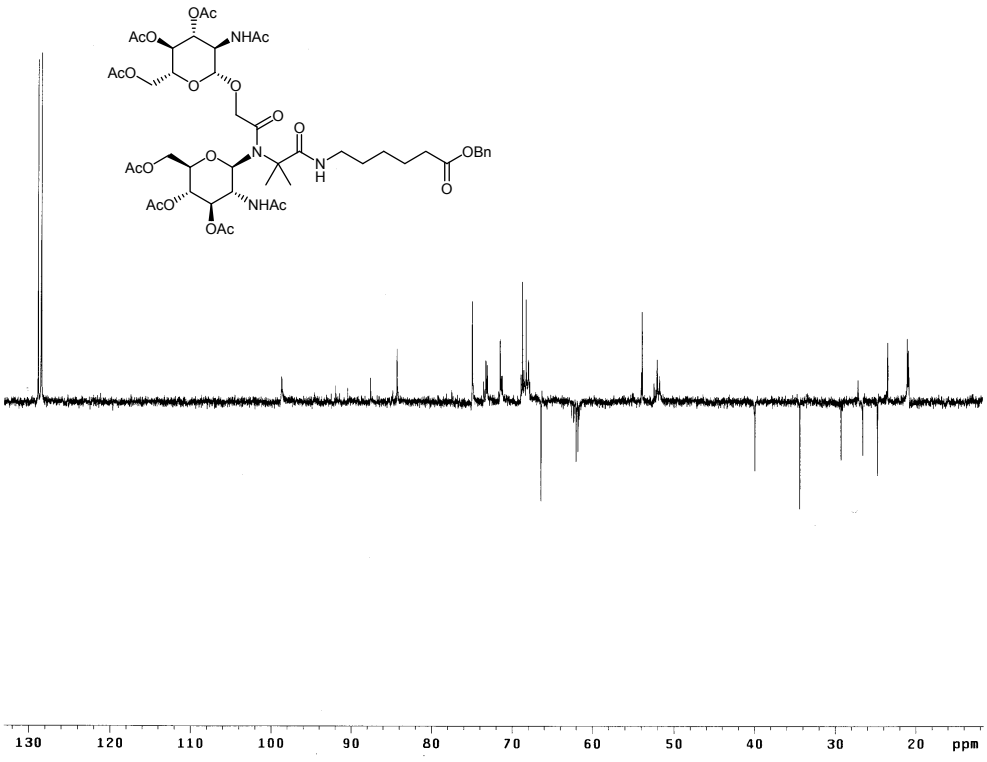


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13C Standard-Parameter

Thu Jul 22 00:11 2004

exp7  dept

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solvent       CDCl3
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                t.fid
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probe            asw5
seqfil           dept
sfrq          100.574
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at              1.311
np              67216
sw            25641.0
bs                 16
ss                  8
tpwr               61
pw               14.7
d1              2.000
d2                  0
tof            1327.2
nt                8000
ct                8000
alock                n
gain         not used
      SPECIAL
j              140.0
mult             1.5
       FLAGS
il                   n
in                   n
dp                   y
     DEC. & VT
dn                  H1
dfrq           399.936
homo                 n
dpwr                40
dof                  0
dm                 nny
dmm                ccw
dmf             9350
pplvl                60
pp                 11.5
j                140.0
     PROCESSING
lb                1.00
wtfile
proc                ft
fn               65536

wexp        procplot
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vs                 80
sc                 65
wc                225
hzmm            54.27
rfl           1970.6
rfp                  0
th                 5.2
rp               -58.8
```
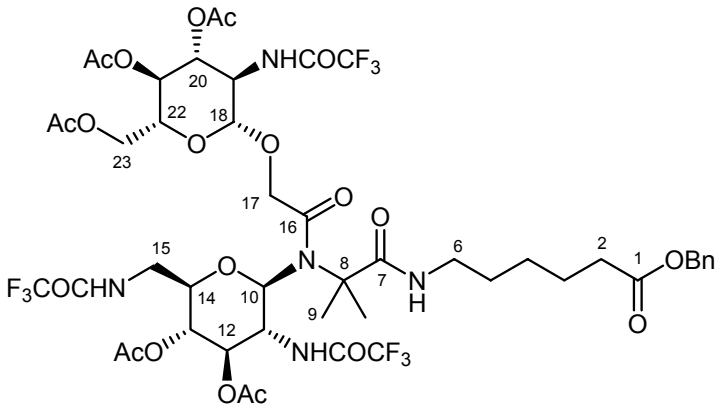
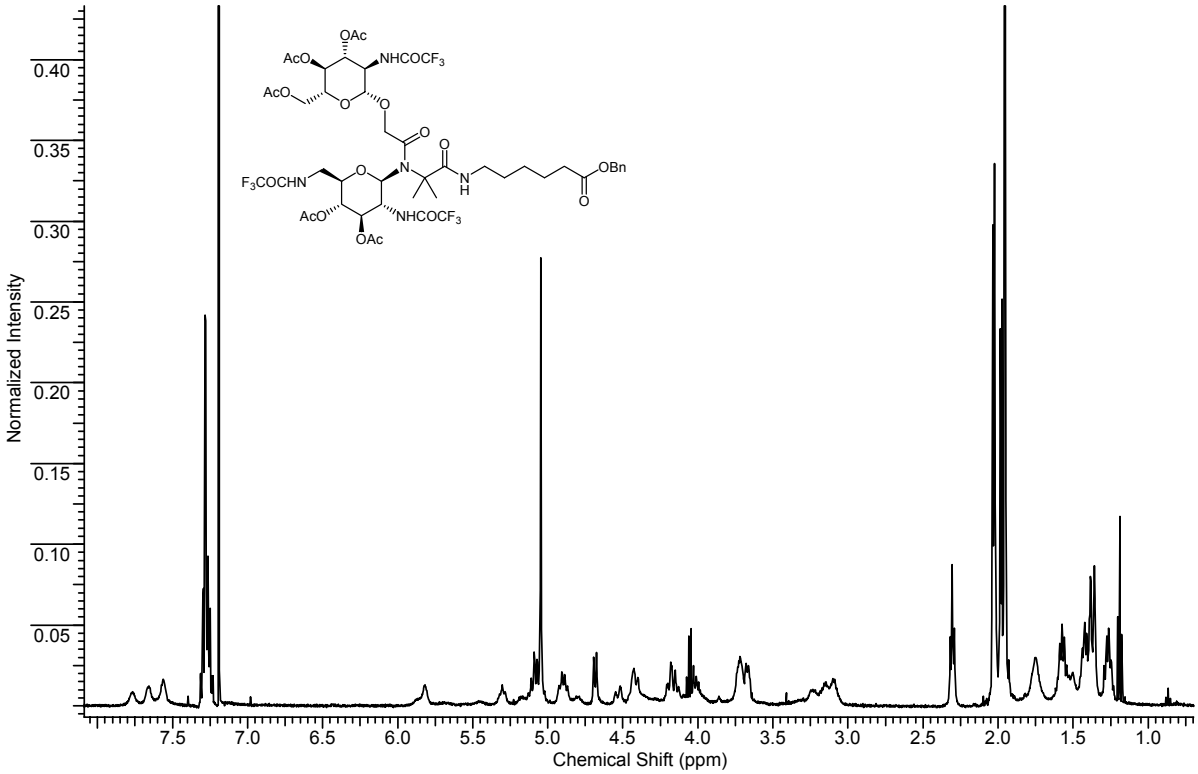

3,4-Di-*O*-acetyl-*N*-(2-{[6-(benzyloxy)-6-oxohexyl]amino}-1,1-dimethyl-2-oxoethyl)-2,6-dideoxy-*N*-[({3,4,6-tri-*O*-acetyl-2-deoxy-2-[(trifluoroacetyl)amino]-β-D-glucopyranosyl}-oxy)acetyl]-2,6-bis[(trifluoroacetyl)amino]-β-D-glucopyranosylamine (19)



^{1}H-NMR (500 MHz, $CDCl_3$): δ [ppm] = 1.30–1.39 (m, 2H), 1.40–1.74 (m, 8H), 1.77–1.89 (m, 2H), 2.03, 2.03, 2.06, 2.10, 2.11 (s, 3H), 2.38 (t, *J* = 7.2 Hz, 2H), 3.12–3.36 (m, 2H), 3.72–3.86 (m, 4H), 4.05–4.31 (m, 4H) 4.44–4.66 (m, 2H), 4.76 (d, *J* = 8.2 Hz, 1H), 4.92–5.05 (m, 2H), 5.10–5.22 (m, 4H), 5.30 (bt, *J* = 9.1 Hz, 1H), 5.82 (bs, 1H), 7.22–7.32 (m, 5H), 7.70–8.18 (m, 3H); **^{13}C-NMR** (125 MHz, $CDCl_3$): δ [ppm] = 20.30, 20.40, 20.49, 20.53, 20.61 (q, $CH_{3(Ac)}$), 24.34 (t, C-3), 25.20, 25.44 (q, C-9), 26.09, 28.67, 34.04, 39.57, 39.97 (t, C-2, C-4–C-6, C-15), 53.43, 54.47 (d, C-11, C-19), 61.70 (t, OCH_2), 64.40 (s, C-8), 66.29, 67.88 (t, OCH_2), 68.03, 69.03, 72.40, 72.69, 74.69, 77.20 (d, C-12–C-14, C-20–C-22), 83.14 (d, C-10), 99.84 (d, C-18), 115.42 (m, CF_3), 128.05, 128.22, 128.58 (d, CH arom.), 135.98 (s, C arom.), 158.06 (m, *C*(O)CF_3), 169.32, 170.78, 170.80, 170.81, 170.86, 171.00, 173.97, 174.00 (s, CO); **HRMS (ESI)** calcd for $C_{47}H_{58}F_9N_5O_{20}Na$ [M+Na^+]: 1206.3424, found: 1206.3421


Normalized Intensity
0.40
0.35
0.30
0.25
0.20
0.15
0.10
0.05
7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0
Chemical Shift (ppm)
OAc
AcO
NHCOCF3
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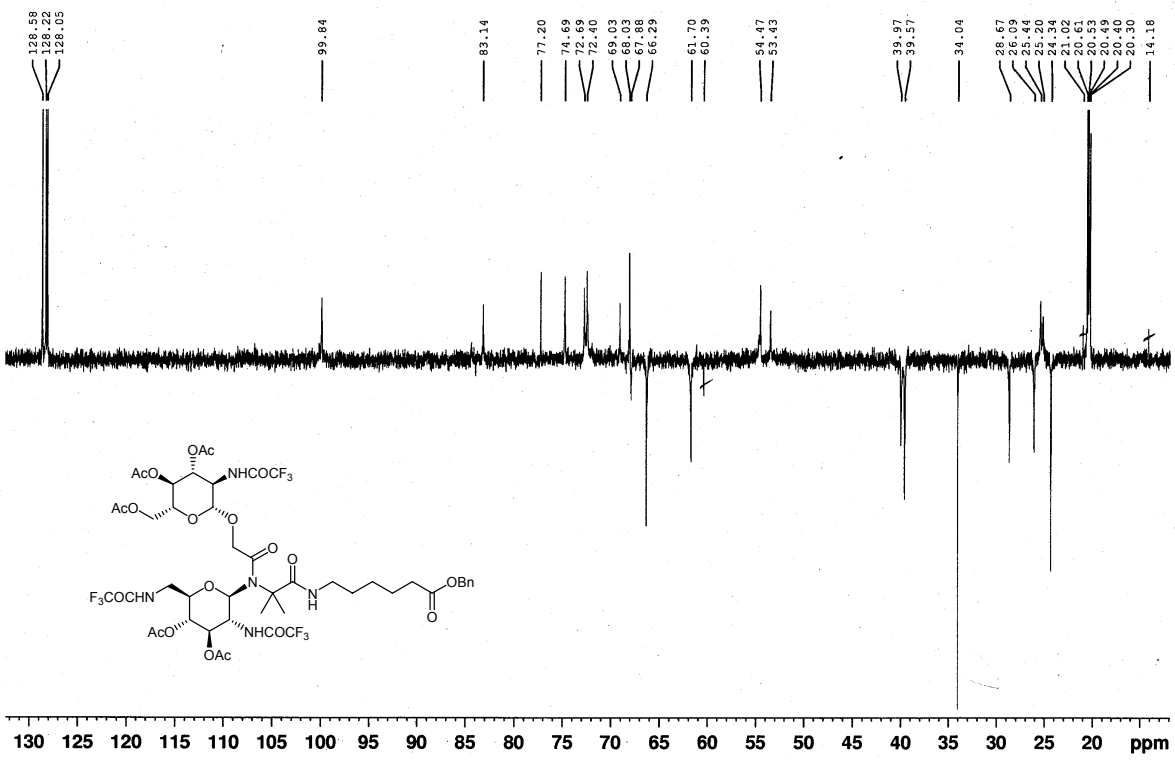
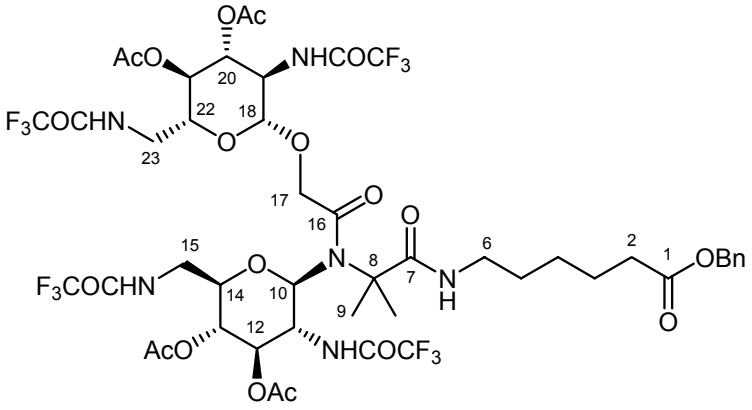
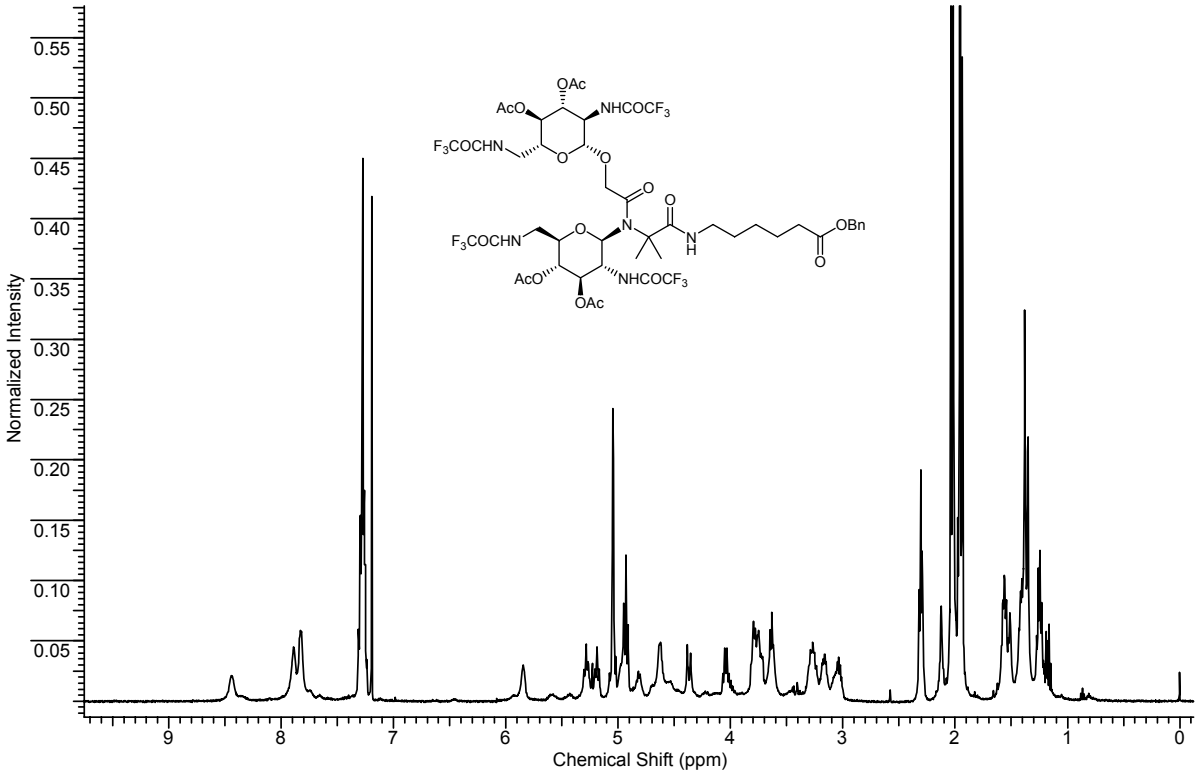
O
F3COCHN
O
AcO
NHCOCF3
OAc

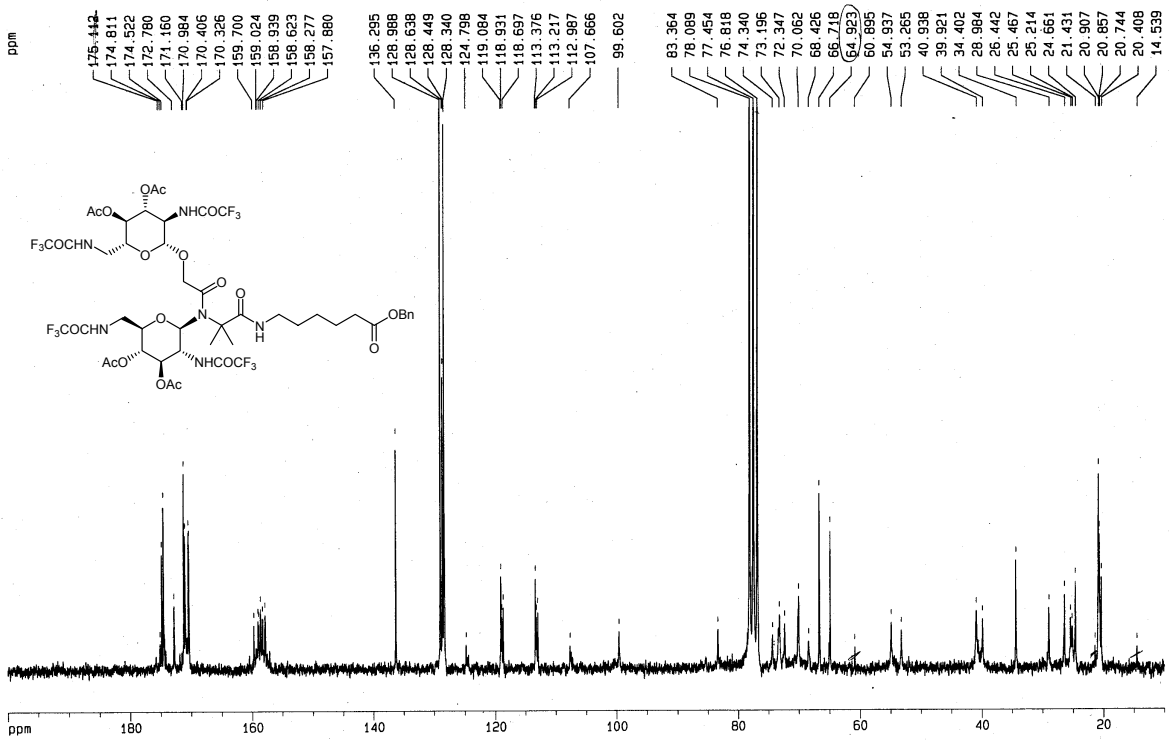

128.58
128.22
128.05
99.84
83.14
77.20
74.69
72.69
72.40
69.03
68.03
67.88
66.29
61.70
60.39
54.47
53.43
39.97
39.57
34.04
28.67
26.09
25.44
25.20
24.34
21.02
20.61
20.53
20.49
20.40
20.30
14.18
OAc
AcO
NHCOCF3
AcO
O
O
O
N
O
N
H
OBn
O
F3COCHN
O
AcO
NHCOCF3
OAc
130 125 120 115 110 105 100 95 90 85 80 75 70 65 60 55 50 45 40 35 30 25 20 ppm

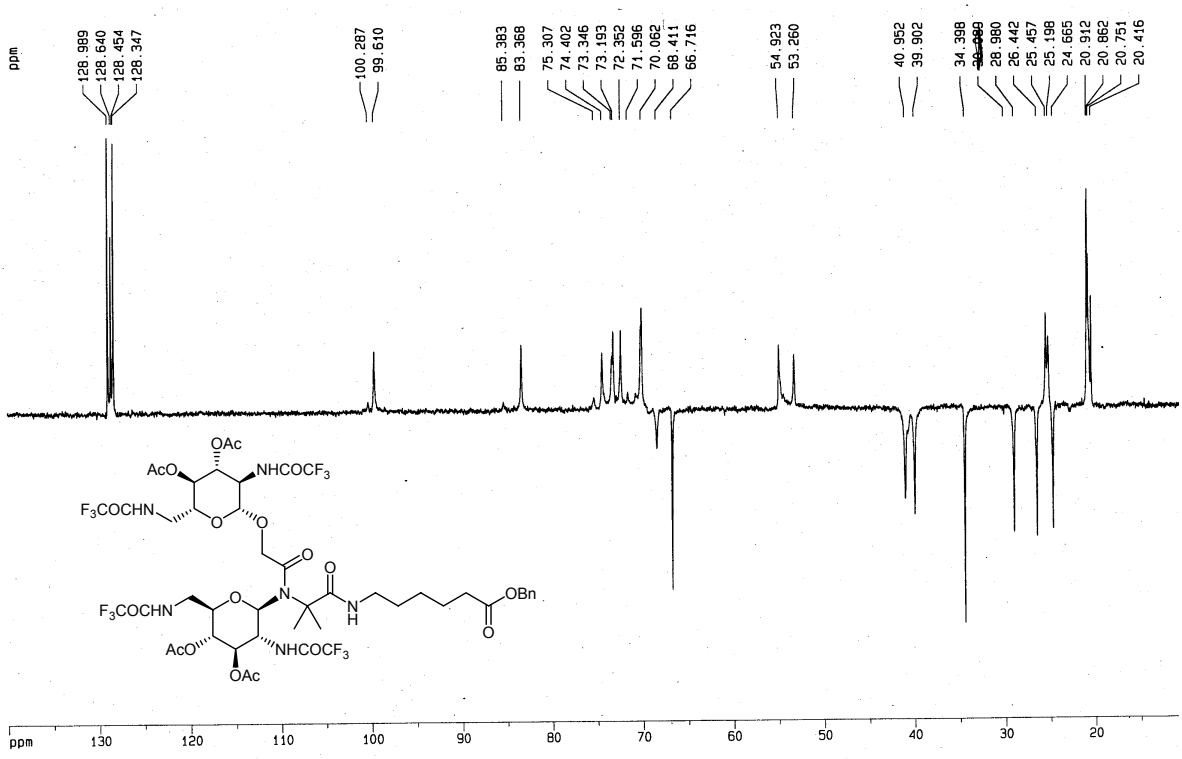
3,4-Di-*O*-acetyl-*N*-(2-{[6-(benzyloxy)-6-oxohexyl]amino}-1,1-dimethyl-2-oxoethyl)-2,6-dideoxy-*N*-[({3,4-di-*O*-acetyl-2,6-dideoxy-2,6-bis[(trifluoroacetyl)amino]-β-D-gluco-pyranosyl}oxy)acetyl]-2,6-bis[(trifluoroacetyl)amino]-β-D-glucopyranosylamine (20)



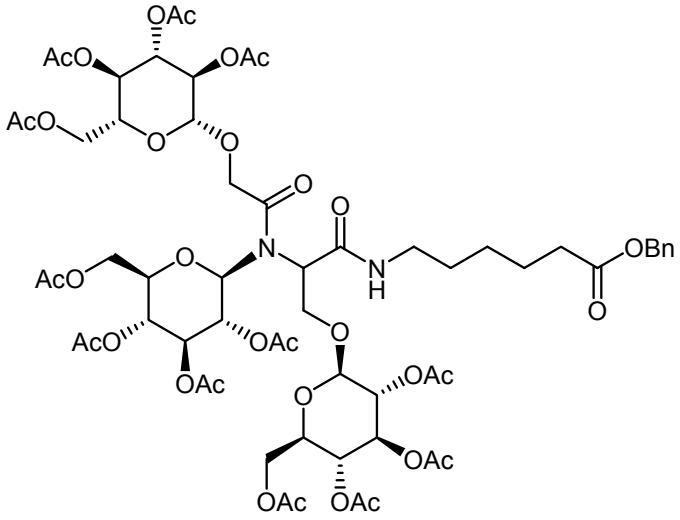
^{1}H-NMR (500 MHz, $CDCl_3$): δ [ppm] = 1.20–1.38 (m, 3H), 1.38–1.55 (m, 6H), 1.55–1.70 (m, 3H), 1.81 (s, 2H), 2.02, 2.04, 2.10, 2.12 (s, 3H), 2.38 (t, J = 7.2 Hz, 2H), 3.04–3.39 (m, 4H), 3.60–3.92 (m, 4H), 4.02–4.16 (m, 1H), 4.37–4.48 (d, J = 15.6 Hz, 1H), 4.61–4.71 (m, 1H), 4.80–4.93 (m, 1H), 4.93–5.05 (m, 2H), 5.10 (s, 2H), 5.23 (t, J = 9.8 Hz, 1H), 5.32 (t, J = 9.4 Hz, 1H), 5.90 (bs, 1H), 7.35 (s, 5H), 7.72–7.96 (m, 3H), 8.44 (bs, 1H); **^{13}C-NMR** (125 MHz, $CDCl_3$): δ [ppm] = 20.42, 20.75, 20.86, 20.91 (q, $CH_{3(Ac)}$), 24.67 (t, C-3), 25.20, 25.46 (q, C-9), 26.44, 28.98, 34.40, 39.90, 40.21, 40.95 (t, C-2, C-4–C-6, C-15, C-23), 53.26, 54.92 (d, C-11, C-19), 64.92 (s, C-8), 66.72, 68.41 (t, OCH_2), 70.06, 70.07, 72.35, 73.19, 73.35, 74.40 (d, C-12–C-14, C-20–C-22), 83.37 (d, C-10), 99.61 (d, C-18), 116.23 (m, CF_3), 128.35, 128.64, 128.99 (d, CH arom.), 136.30 (s, C arom.), 159.02 (m, C(O)CF_3), 170.33, 170.41, 170.98, 171.16, 172.78, 174.52, 174.81 (s, CO); **HRMS (ESI)** calcd for $C_{47}H_{56}F_{12}N_6O_{19}Na$ [M+Na^+]: 1259.3301, found: 1259.3264


Normalized Intensity
Chemical Shift (ppm)
OAc
AcO
NHCOCF3
F3COCHN
O
N
H
OBn
NHCOCF3
OAc

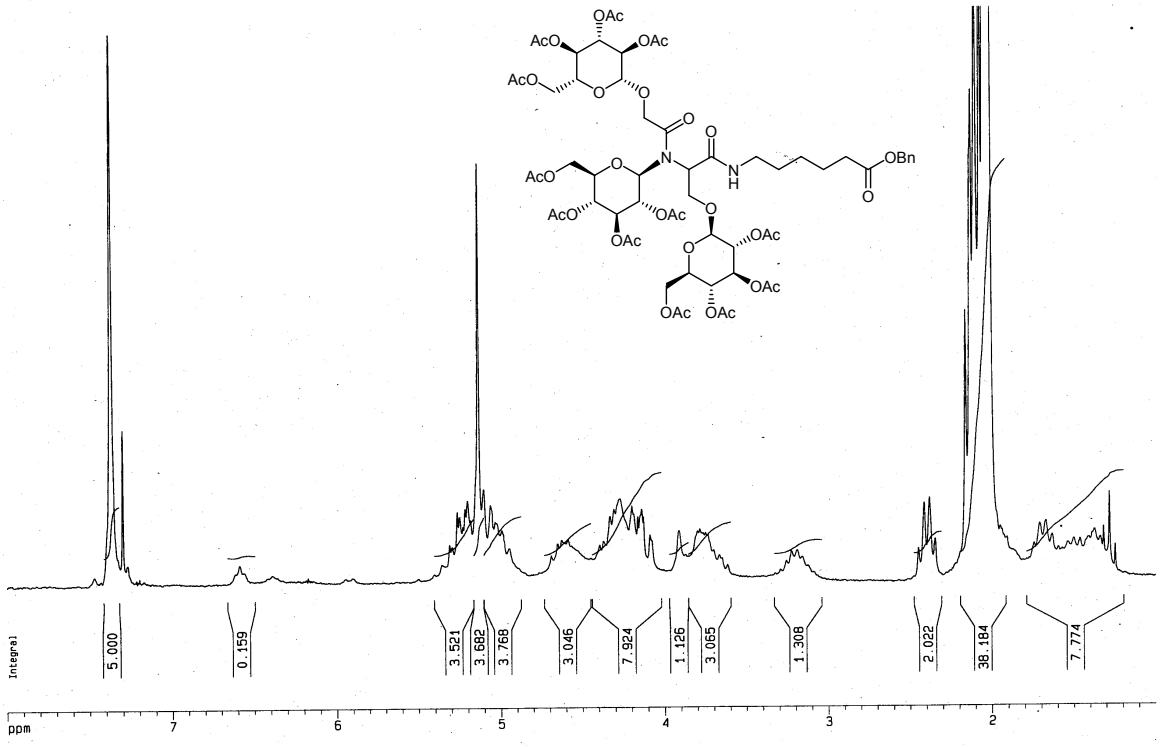

ppm
175.442
174.811
174.522
172.780
171.160
170.984
170.406
170.326
159.700
159.024
158.939
158.623
158.277
157.880
136.295
128.988
128.638
128.449
128.340
124.798
119.084
118.931
118.697
113.376
113.217
112.987
107.666
99.602
83.364
78.089
77.454
76.818
74.340
73.196
72.347
70.062
68.426
66.718
64.923
60.895
54.937
53.265
40.938
39.921
34.402
28.984
26.442
25.467
25.214
24.661
21.431
20.907
20.857
20.744
20.408
14.539
OAc
AcO
NHCOCF3
F3COCHN
O
N
H
OBn
NHCOCF3
OAc

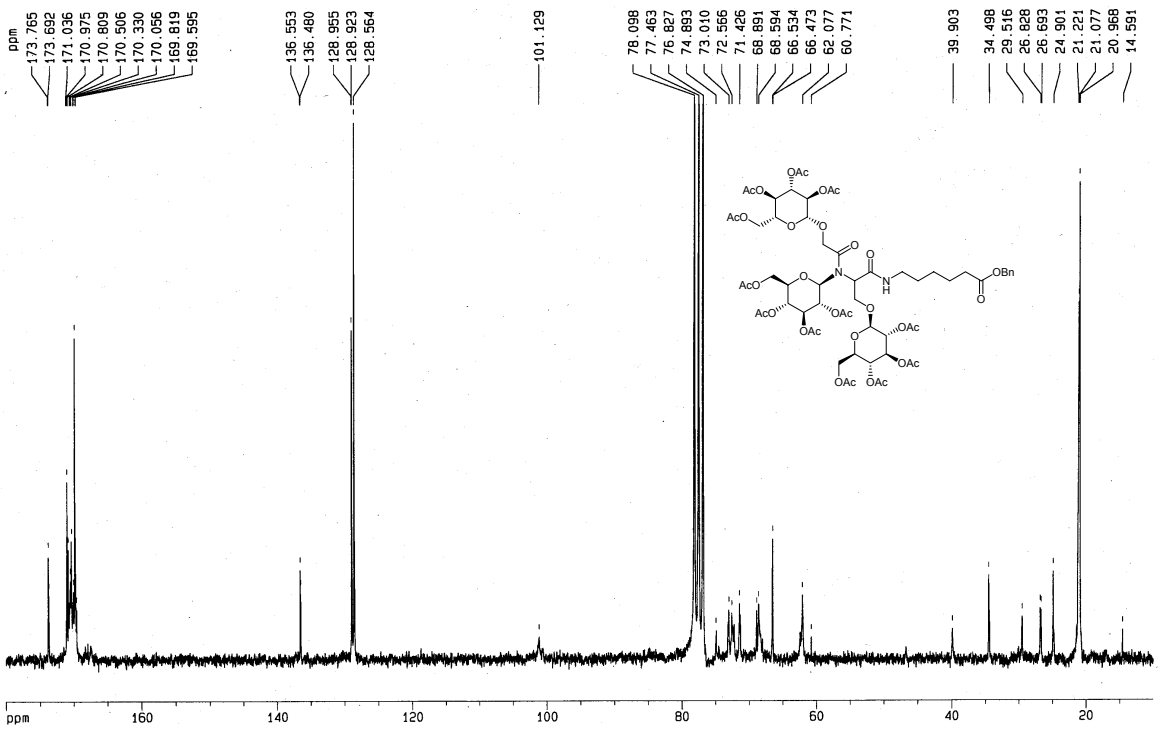

ppm
128.989
128.640
128.454
128.347
100.287
99.610
85.383
83.368
75.307
74.402
73.346
73.193
72.352
71.596
70.062
68.411
66.716
54.923
53.260
40.952
39.902
34.398
28.980
26.442
25.457
25.198
24.665
20.912
20.862
20.751
20.416
OAc
AcO
NHCOCF3
F3COCHN
O
N
H
OBn
ppm
130
120
110
100
90
80
70
60
50
40
30
20

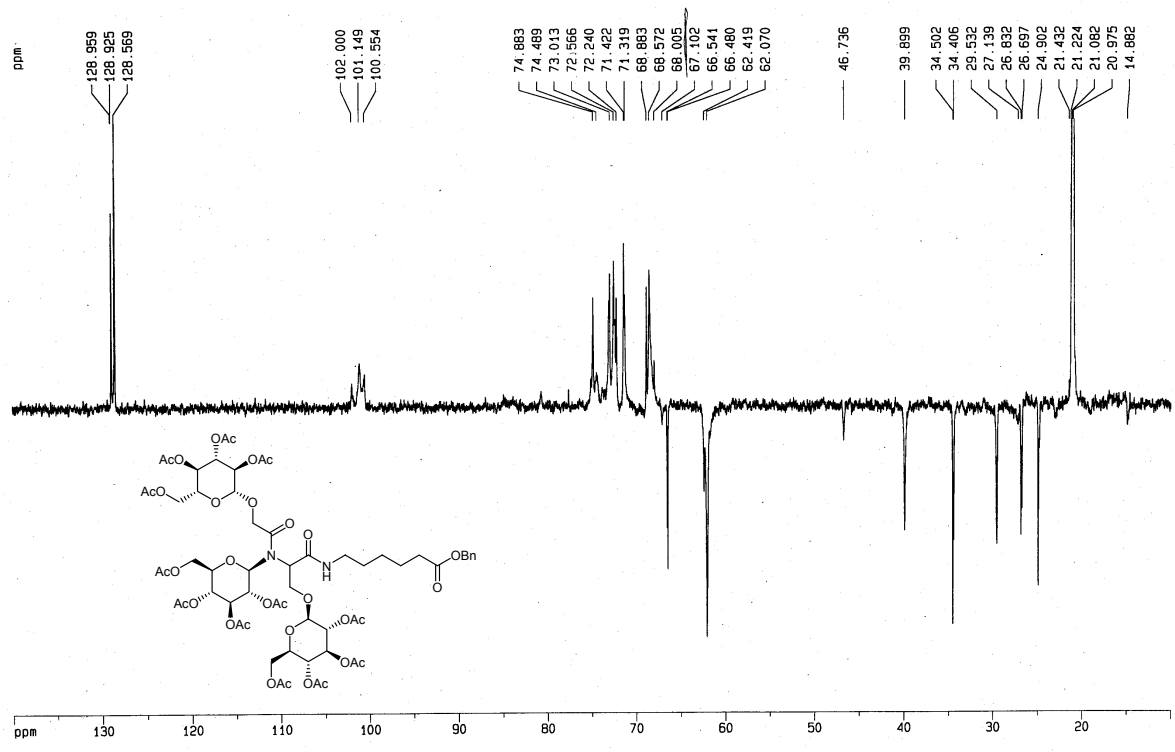
2,3,4,6-Tetra-*O*-acetyl-*N*-((1*R,S*)-2-{[6-(benzyloxy)-6-oxohexyl]amino}-2-oxo-1-{[(2,3,4,6-tetra-*O*-acetyl-β-D-glucopyranosyl)oxy]methyl}ethyl)-*N*-{[(2,3,4,6-tetra-*O*-acetyl-β-D-glucopyranosyl)oxy]acetyl}-β-D-glucopyranosylamine (21)



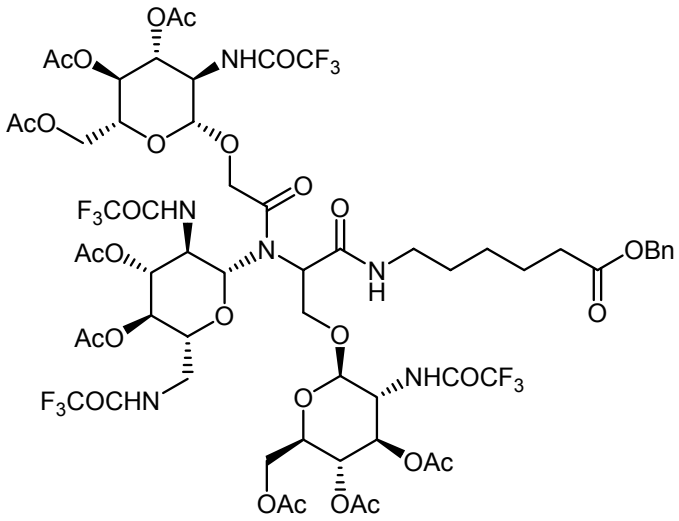
^{1}H-NMR (200 MHz, $CDCl_3$): isomeric mixture: δ [ppm] = 1.19–1.79 (m, 6H), 1.92–2.19 (m, 36H), 2.48–2.31 (m, 2H), 3.04–3.33 (m, 2H), 3.60–5.41 (m, 28H), 6.49–6.66 (m, 1H), 7.37 (s, 5H); **^{13}C-NMR** (50 MHz, $CDCl_3$): signal ranges: δ [ppm] = 20.98–21.43 (q, $CH_{3(Ac)}$), 24.90, 26.83, 29.53, 34.50, 39.90, 46.74, 62.07, 62.42, 66.48 (t, CH_2, OCH_2), 68.01–101.15 (d, CH sugar), 128.57, 128.93, 128.96 (d, CH arom.), 136.55 (s, C arom.), 169.60–173.77 (s, CO); **HRMS (ESI)** calcd for $C_{60}H_{80}N_2O_{33}Na$ [M+Na$^+$]: 1379.4536, found: 1379.4491


Integral
5.000
0.159
3.521
3.682
3.768
3.046
7.924
1.126
3.065
1.308
2.022
38.184
7.774
ppm
7
6
5
4
3
2

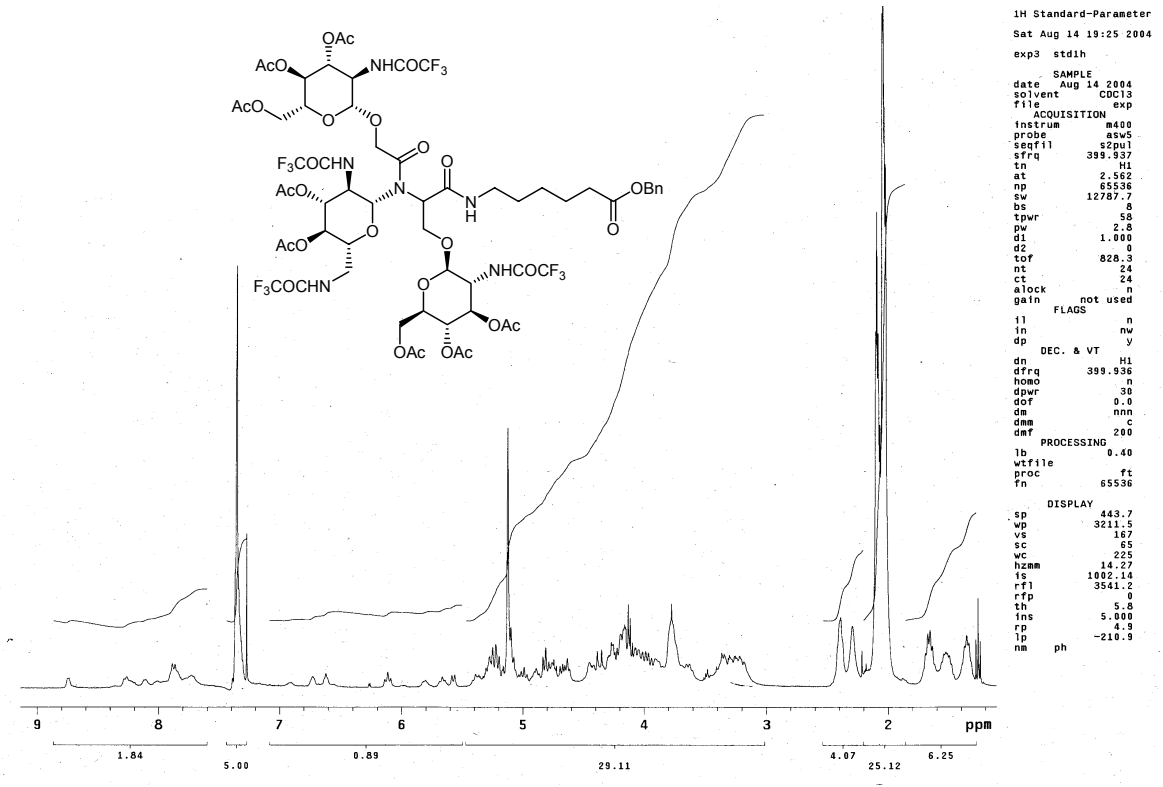

ppm
173.765
173.692
171.036
170.975
170.809
170.506
170.330
170.056
169.819
169.595
136.553
136.480
128.955
128.923
128.564
101.129
78.098
77.463
76.827
74.893
73.010
72.566
71.426
68.891
68.594
66.534
66.473
62.077
60.771
39.903
34.498
29.516
26.828
26.693
24.901
21.221
21.077
20.968
14.591
ppm
160
140
120
100
80
60
40
20


ppm
128.959
128.925
128.569
102.000
101.149
100.554
74.883
74.489
73.013
72.566
72.240
71.422
71.319
68.883
68.572
68.005
67.102
66.541
66.480
62.419
62.070
46.736
39.899
34.502
34.406
29.532
27.139
26.832
26.697
24.902
21.432
21.224
21.082
20.975
14.882
OAc
AcO
OBn
ppm
130
120
110
100
90
80
70
60
50
40
30
20

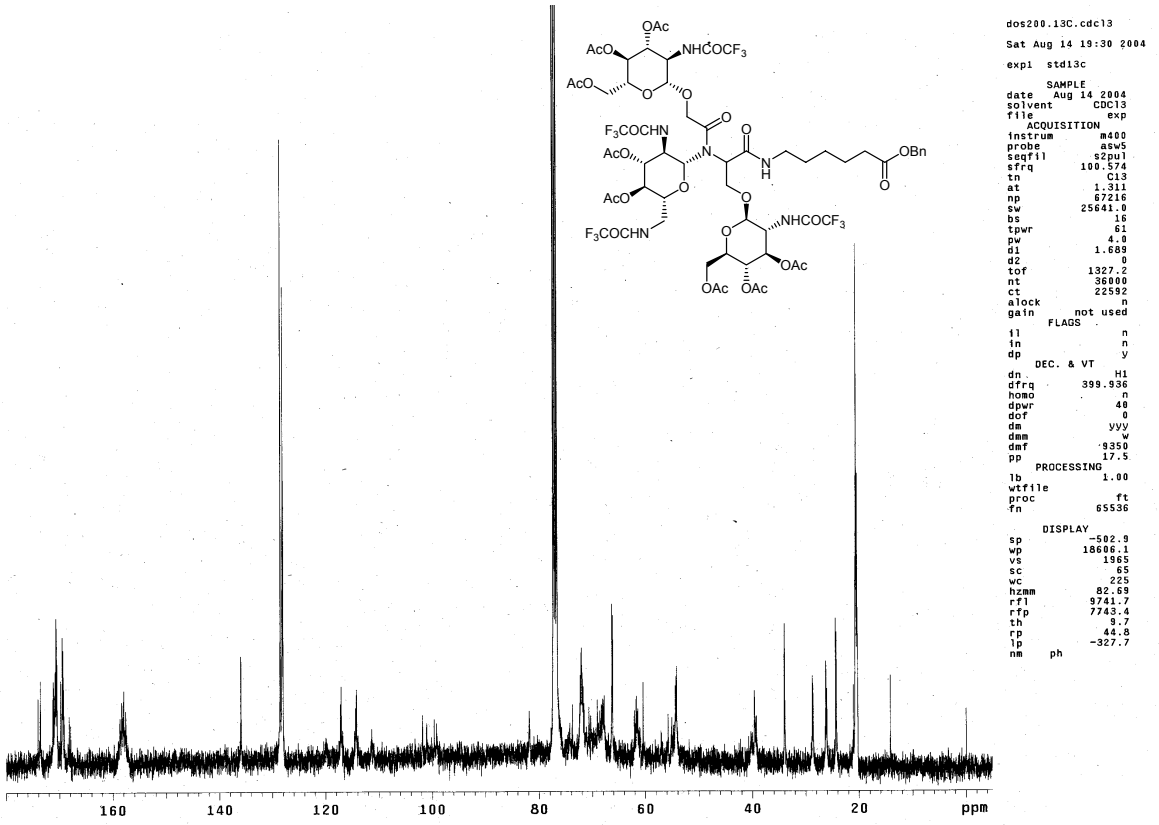
3,4-Di-*O*-acetyl-*N*-{(1*R*,*S*)-2-{[6-(benzyloxy)-6-oxohexyl]amino}-2-oxo-1-[({3,4,6-tri-*O*-acetyl-2-deoxy-2-[(trifluoroacetyl)amino]-β-D-glucopyranosyl}oxy)methyl]ethyl}-2,6-dideoxy-*N*-[({3,4,6-tri-*O*-acetyl-2-deoxy-2-[(trifluoroacetyl)amino]-β-D-glucopyranosyl}-oxy)acetyl]-2,6-bis[(trifluoroacetyl)amino]-β-D-glucopyranosylamine (22)



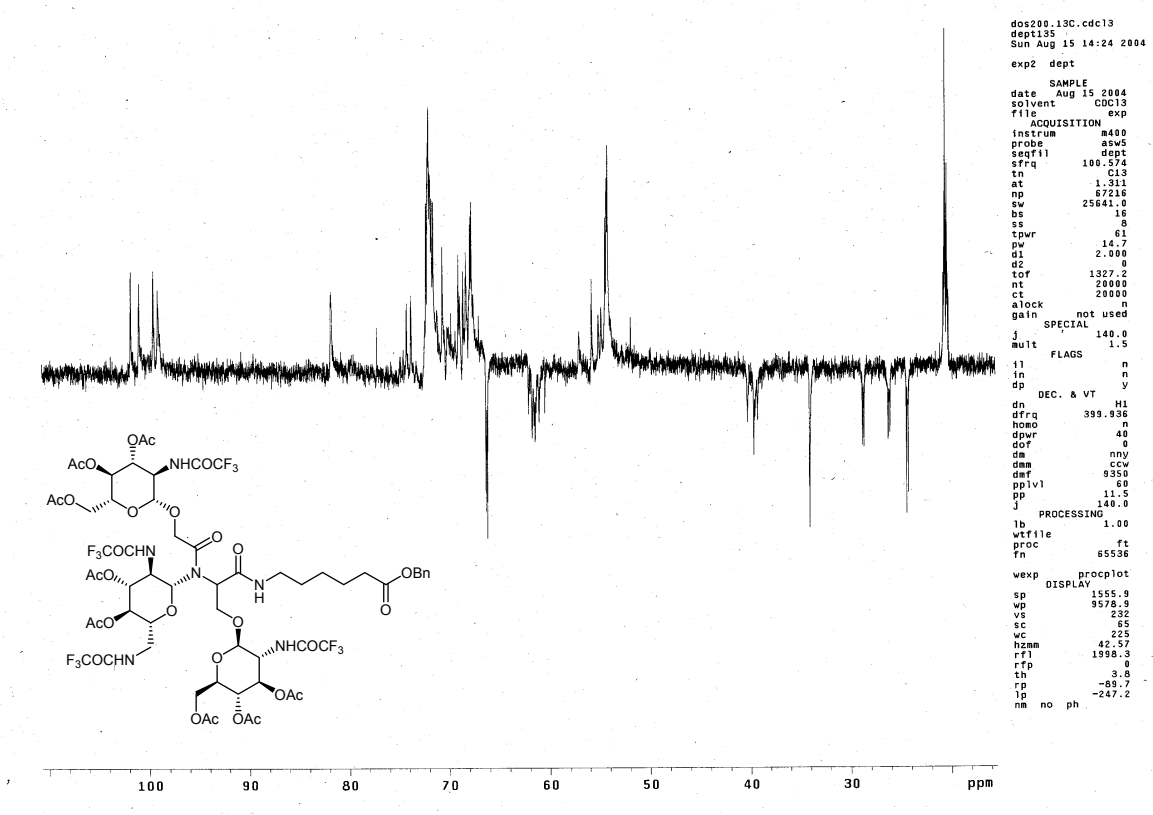
^{1}H-NMR (400 MHz, $CDCl_3$): isomeric mixture: δ [ppm] = 1.27–1.77 (m, 6H), 1.93–2.16 (m, 24H), 2.22–2.49 (m, 4H), 3.12–5.47 (m, 30H), 5.50–7.00 (m, 1H), 7.30–7.43 (m, 5H), 7.60–8.80 (m, 2H); **^{13}C-NMR** (100 MHz, $CDCl_3$): signal ranges: δ [ppm] = 20.25–20.74 (q, $CH_{3(Ac)}$), 24.37, 26.20, 28.60, 34.03, 39.31–40.04 (t, CH_2, NCH_2), 54.18–54.60 (d, NCH), 61.20–62.03, 66.29 (t, OCH_2), 67.75–101.81 (d, CH sugar), 116.08 (m, CF_3), 127.99, 128.10, 128.54 (d, CH arom.), 135.95 (s, C arom.), 158.03 (m, *C*(O)CF_3), 167.92–171.30 (s, CO); **HRMS (ESI)** calcd for $C_{60}H_{72}F_{12}N_6O_{29}Na$ [M+Na^+]: 1591.4044, found: 1591.4009




```
1H Standard-Parameter

Sat Aug 14 19:25 2004

exp3  std1h

      SAMPLE
date   Aug 14 2004
solvent       CDCl3
file            exp
   ACQUISITION
instrum         m400
probe           asw5
seqfil        s2pul
sfrq        399.937
tn              H1
at           2.562
np           65536
sw         12787.7
bs               8
tpwr            58
pw             2.8
d1           1.000
d2               0
tof          828.3
nt              24
ct              24
alock            n
gain       not used
      FLAGS
il               n
in              nw
dp               y
    DEC. & VT
dn              H1
dfrq        399.936
homo             n
dpwr            30
dof            0.0
dm             nnn
dmm              c
dmf            200
    PROCESSING
lb             0.40
wtfile
proc             ft
fn            65536

     DISPLAY
sp           443.7
wp          3211.5
vs             167
sc              65
wc             225
hzmm         14.27
is         1002.14
rfl          3541.2
rfp               0
th              5.8
ins           5.000
rp              4.9
lp           -210.9
nm     ph
```

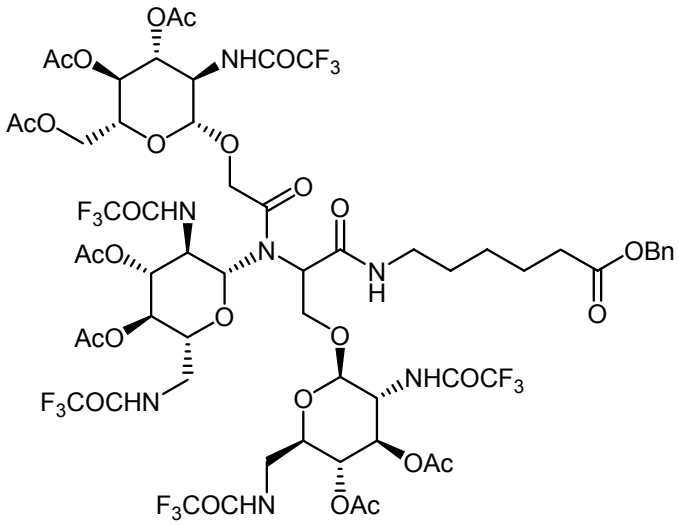



```
dos200.13C.cdcl3

Sat Aug 14 19:30 2004

exp1  std13c

      SAMPLE
date   Aug 14 2004
solvent       CDCl3
file            exp
   ACQUISITION
instrum         m400
probe           asw5
seqfil        s2pul
sfrq        100.574
tn              C13
at           1.311
np           67216
sw         25641.0
bs              16
tpwr            61
pw             4.0
d1           1.689
d2               0
tof          1327.2
nt            36000
ct            22592
alock            n
gain       not used
      FLAGS
il               n
in               n
dp               y
    DEC. & VT
dn              H1
dfrq        399.936
homo             n
dpwr            40
dof              0
dm             yyy
dmm              w
dmf           9350
pp            17.5
    PROCESSING
lb             1.00
wtfile
proc             ft
fn            65536

     DISPLAY
sp          -502.9
wp         18606.1
vs            1965
sc              65
wc             225
hzmm         82.69
rfl          9741.7
rfp          7743.4
th               9.7
rp              44.8
lp           -327.7
nm     ph
```

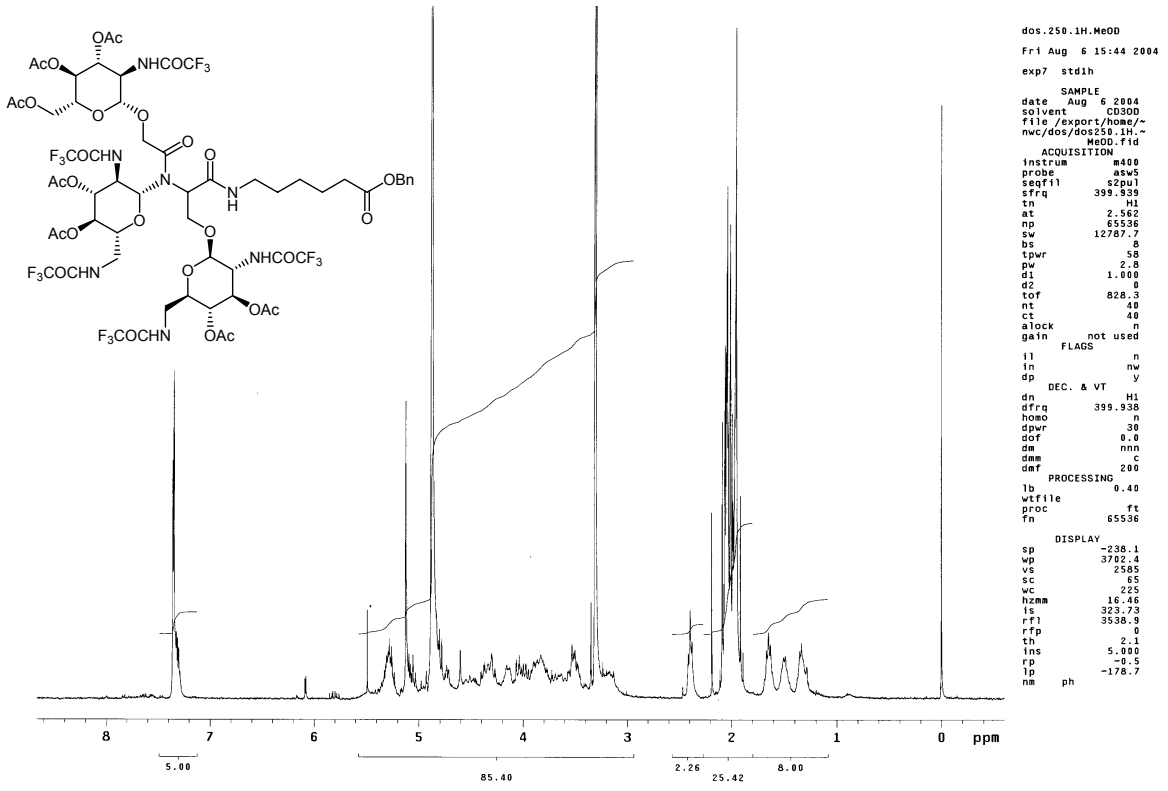



```
dos200.13C.cdcl3
dept135
Sun Aug 15 14:24 2004

exp2  dept

      SAMPLE
date    Aug 15 2004
solvent          CDCl3
file              exp
    ACQUISITION
instrum          m400
probe            asw5
seqfil           dept
sfrq          100.574
tn                C13
at              1.311
np              67216
sw            25641.0
bs                 16
ss                  8
tpwr               61
pw               14.7
d1              2.000
d2                  0
tof            1327.2
nt              20000
ct              20000
alock               n
gain       not used
      SPECIAL
j                140.0
mult               1.5
        FLAGS
il                  n
in                  n
dp                  y
    DEC. & VT
dn                 H1
dfrq          399.936
homo                n
dpwr               40
dof                 0
dm                nny
dmm               ccw
dmf              9350
pplvl              60
pp               11.5
j                140.0
    PROCESSING
lb                1.00
wtfile
proc                ft
fn              65536

wexp          procplot
     DISPLAY
sp              1555.9
wp              9578.9
vs                 232
sc                  65
wc                 225
hzmm             42.57
rfl              1998.3
rfp                 0
th                  3.8
rp               -89.7
lp              -247.2
nm  no  ph
```

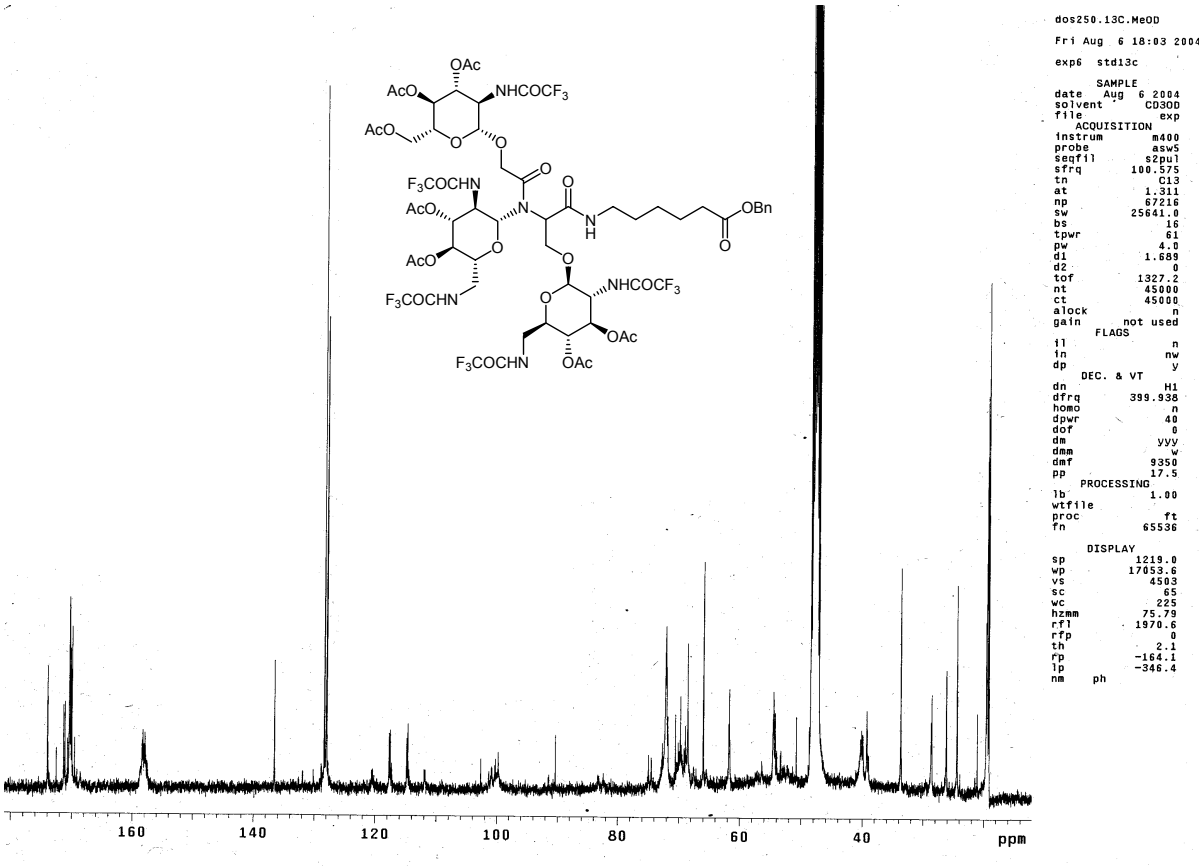
3,4-Di-*O*-acetyl-*N*-{(1*R,S*)-2-{[6-(benzyloxy)-6-oxohexyl]amino}-1-[({3,4-di-*O*-acetyl-2,6-dideoxy-2,6-bis[(trifluoroacetyl)amino]-β-D-glucopyranosyl}oxy)methyl]-2-oxoethyl}-2,6-dideoxy-*N*-[({3,4,6-tri-*O*-acetyl-2-deoxy-2-[(trifluoroacetyl)amino]-β-D-glucopyranosyl}oxy)acetyl]-2,6-bis[(trifluoroacetyl)amino]-β-D-glucopyranosylamine (23)



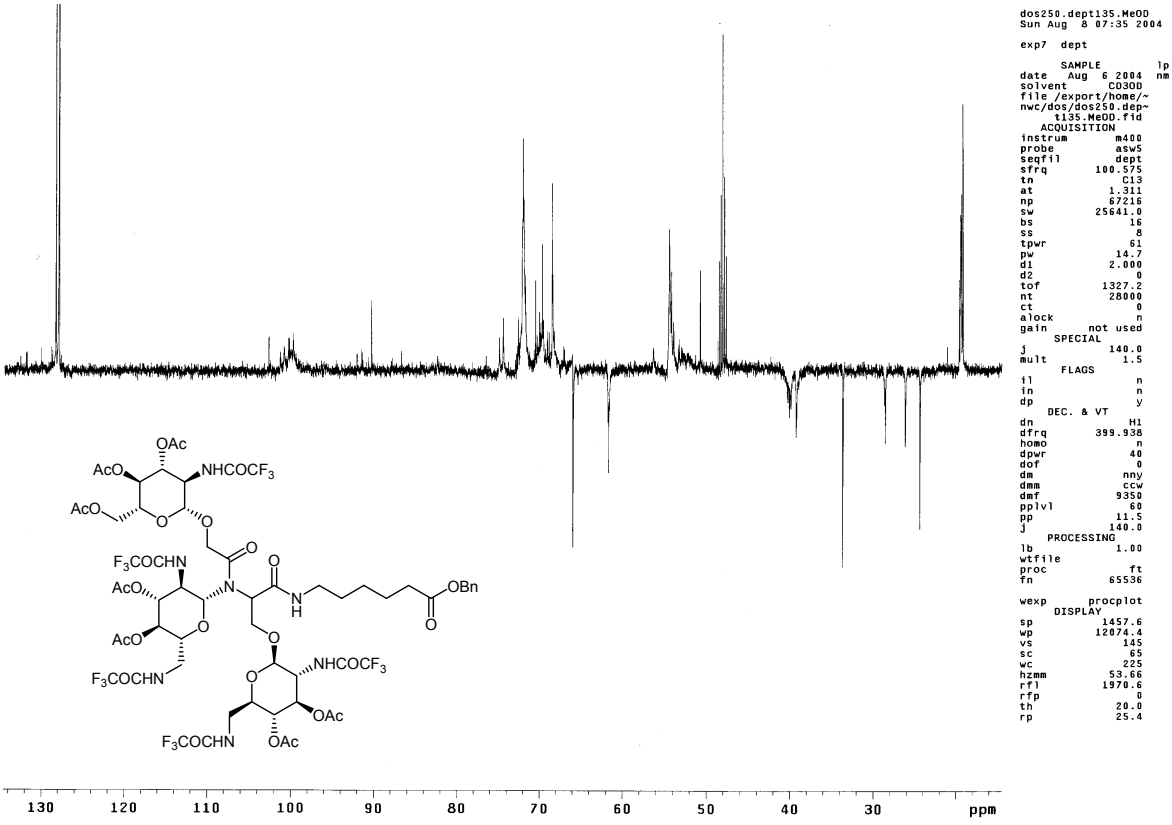
^{1}H-NMR (400 MHz, $CDCl_3$): isomeric mixture: δ [ppm] = 1.26–1.40 (m, 2H), 1.44–1.57 (m, 2H), 1.59–1.72 (m, 2H), 1.90–2.10 (m, 21H, $CH_{3(Ac)}$), 2.34–2.43 (m, 2H), 3.02–5.43 (m, 31H), 7.28–7.37 (m, 5H, CH arom.); **^{13}C-NMR** (100 MHz, $CDCl_3$): signal ranges: δ [ppm] = 20.03–20.53 (q, $CH_{3(Ac)}$), 25.32, 27.09, 29.48, 34.58, 40.76, 41.01, 41.19 (t, CH_2, NCH_2), 51.68–55.45 (d, NCH), 62.63–66.91 (t, OCH_2), 69.46–103.58 (d, CH sugar), 116.08 (m, CF_3), 127.97, 128.00, 128.34 (d, CH arom.), 136.55 (s, C arom.), 158.03 (m, *C*(O)CF_3), 169.54–174.03 (s, CO); **HRMS (ESI)** calcd for $C_{60}H_{70}F_{15}N_7O_{28}Na$ [M+Na^+]: 1644.3922, found: 1644.3949




```
dos.250.1H.MeOD

Fri Aug  6 15:44 2004

exp7  std1h

        SAMPLE
date   Aug  6 2004
solvent       CD3OD
file /export/home/~
nwc/dos/dos250.1H.~
              MeOD.fid
   ACQUISITION
instrum         m400
probe           asw5
seqfil          s2pul
sfrq         399.939
tn               H1
at            2.562
np            65536
sw           12787.7
bs                8
tpwr             58
pw              2.8
d1            1.000
d2                0
tof           828.3
nt               40
ct               40
alock             n
gain       not used
      FLAGS
il                n
in               nw
dp                y
    DEC. & VT
dn               H1
dfrq         399.938
homo              n
dpwr             30
dof             0.0
dm              nnn
dmm               c
dmf             200
    PROCESSING
lb              0.40
wtfile
proc              ft
fn             65536

    DISPLAY
sp            -238.1
wp            3702.4
vs              2585
sc                65
wc               225
hzmm          16.46
is            323.73
rfl           3538.9
rfp               0
th              2.1
ins           5.000
rp             -0.5
lp            -178.7
nm    ph
```




```
dos250.13C.MeOD

Fri Aug  6 18:03 2004

exp6  std13c

        SAMPLE
date   Aug  6 2004
solvent       CD3OD
file              exp
   ACQUISITION
instrum         m400
probe           asw5
seqfil          s2pul
sfrq         100.575
tn               C13
at             1.311
np             67216
sw           25641.0
bs                16
tpwr              61
pw               4.0
d1             1.689
d2                0
tof           1327.2
nt             45000
ct             45000
alock             n
gain       not used
      FLAGS
il                n
in               nw
dp                y
    DEC. & VT
dn               H1
dfrq         399.938
homo              n
dpwr             40
dof               0
dm              yyy
dmm               w
dmf            9350
pp             17.5
    PROCESSING
lb               1.00
wtfile
proc              ft
fn             65536

    DISPLAY
sp            1219.0
wp           17053.6
vs              4503
sc                65
wc               225
hzmm          75.79
rfl           1970.6
rfp               0
th              2.1
rp             -164.1
lp             -346.4
nm    ph
```




```
dos250.dept135.MeOD
Sun Aug  8 07:35 2004

exp7  dept

      SAMPLE                lp
date    Aug  6 2004        nm
solvent       CD3OD
file /export/home/~
nwc/dos/dos250.dep~
    t135.MeOD.fid
   ACQUISITION
instrum          m400
probe            asw5
seqfil           dept
sfrq          100.575
tn                C13
at              1.311
np              67216
sw            25641.0
bs                 16
ss                  8
tpwr               61
pw               14.7
d1              2.000
d2                  0
tof            1327.2
nt              28000
ct                  0
alock               n
gain         not used
     SPECIAL
j             140.0
mult            1.5
      FLAGS
il                  n
in                  n
dp                  y
    DEC. & VT
dn                 H1
dfrq          399.938
homo                n
dpwr               40
dof                 0
dm                nny
dmm               ccw
dmf              9350
pplvl              60
pp               11.5
J             140.0
   PROCESSING
lb                1.00
wtfile
proc                ft
fn              65536

wexp       procplot
    DISPLAY
sp            1457.6
wp           12074.4
vs               145
sc                65
wc               225
hzmm          53.66
rfl           1970.6
rfp                 0
th               20.0
rp               25.4
```