

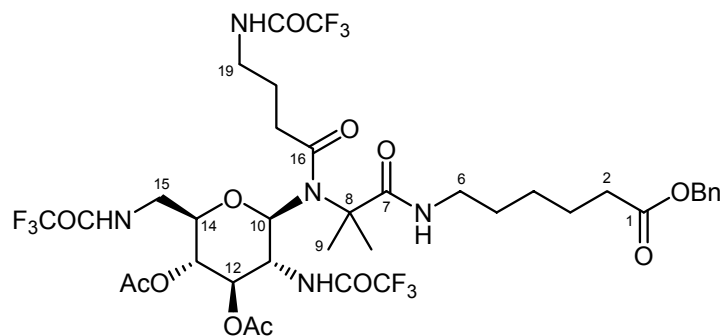
Supporting Information

Synthesis of Multivalent Aminoglycoside Mimics via the Ugi Multicomponent Reaction

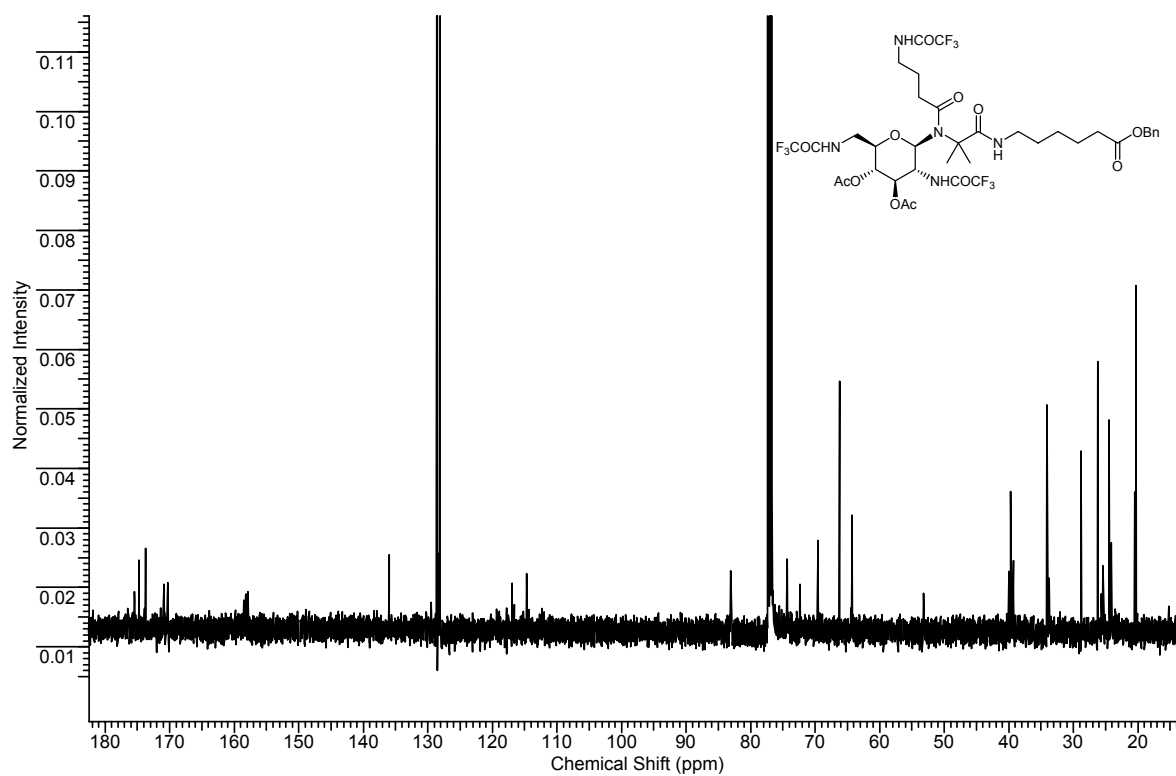
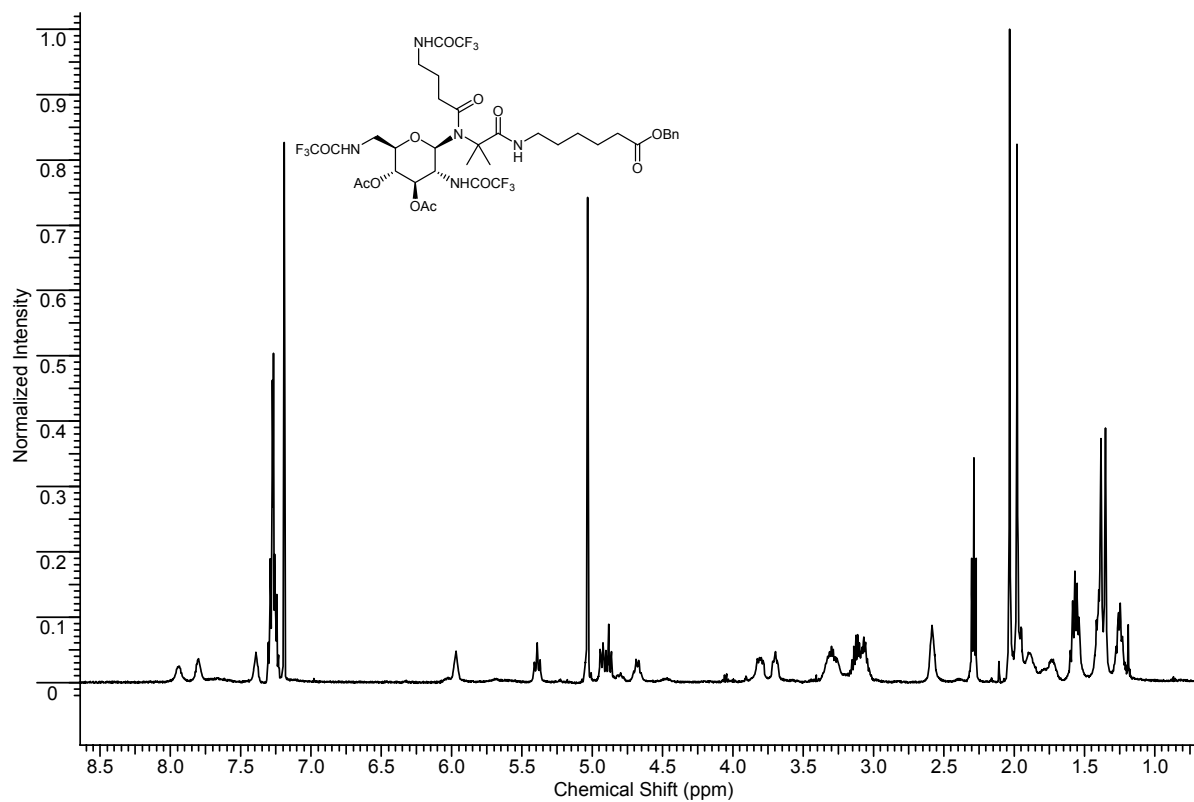
Bernhard Westermann,* and Simon Dörner

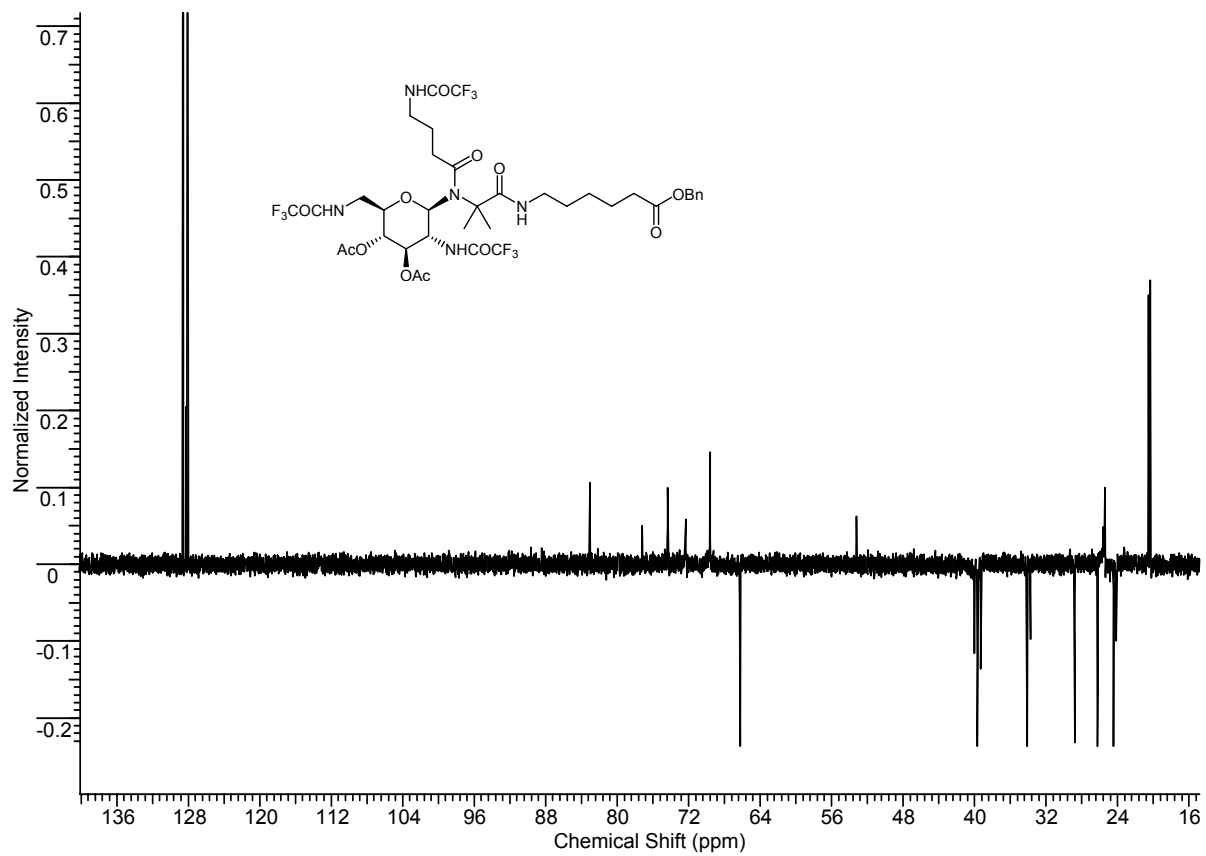
Leibniz Institute of Plant Biochemistry, Department of Bioorganic Chemistry, Weinberg 3, 06120 Halle (Saale), Germany. Fax: 49 345 5582 1309; Tel: 49 345 5582 1340; E-mail: bwesterm@ipb-halle.de

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)

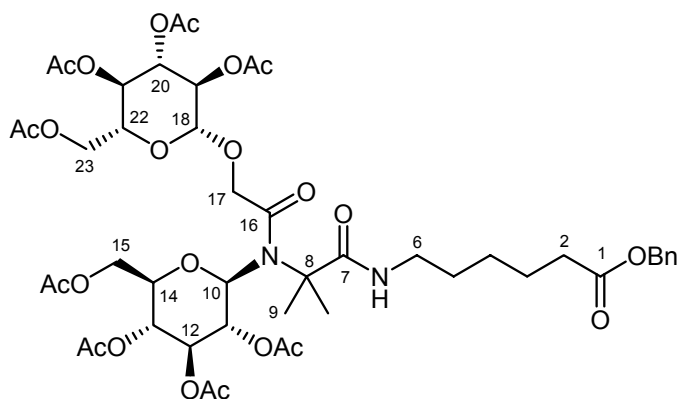


¹H-NMR (500 MHz, CDCl₃): δ [ppm] = 1.21–1.29 (m, 2H, 4-H), 1.33–1.36 (m, 8H, 5-H, 9-H), 1.51–1.60 (m, 2H, 3-H), 1.68–1.77 (m, 2H, 18-H), 1.88 (s, 3H, CH_{3(Ac)}), 2.03 (s, 3H, CH_{3(Ac)}), 2.26–2.32 (t, J = 7.4 Hz, 2H, 2-H), 2.54–2.62 (m, 2H, 17-H), 3.00–3.17 (m, 3H, 15-H^a, 6-H), 3.22–3.38 (m, 2H, 19-H), 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, CH₂ benzyl.), 5.36–5.43 (pt, J = 9.6 Hz, 1H, 12-H), 5.94–6.00 (bs, 1H, 1'-NH), 7.22–7.31 (m, 5H, CH arom.), 7.36–7.42 (bs, 1H, 19-NH), 7.76–7.84 (bs, 1H, 15-NH), 7.90–7.99 (bs, 1H, 11-NH); **¹³C-NMR** (125 MHz, CDCl₃): 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, CH₂ benzyl.), 69.58 (d, C-13), 72.32 (d, C-12), 74.32 (d, C-14), 83.05 (d, C-10), 115.50 (m, CF₃), 128.07, 128.22, 128.56 (d, CH arom.), 136.00 (s, C arom.), 158.25 (m, C(O)CF₃), 170.28, 170.85, 173.72, 174.74, 175.43 (s, CO); **HRMS (ESI)** calcd for C₃₇H₄₆F₉N₅O₁₂Na [M+Na⁺]: 946.2891, found: 946.2875

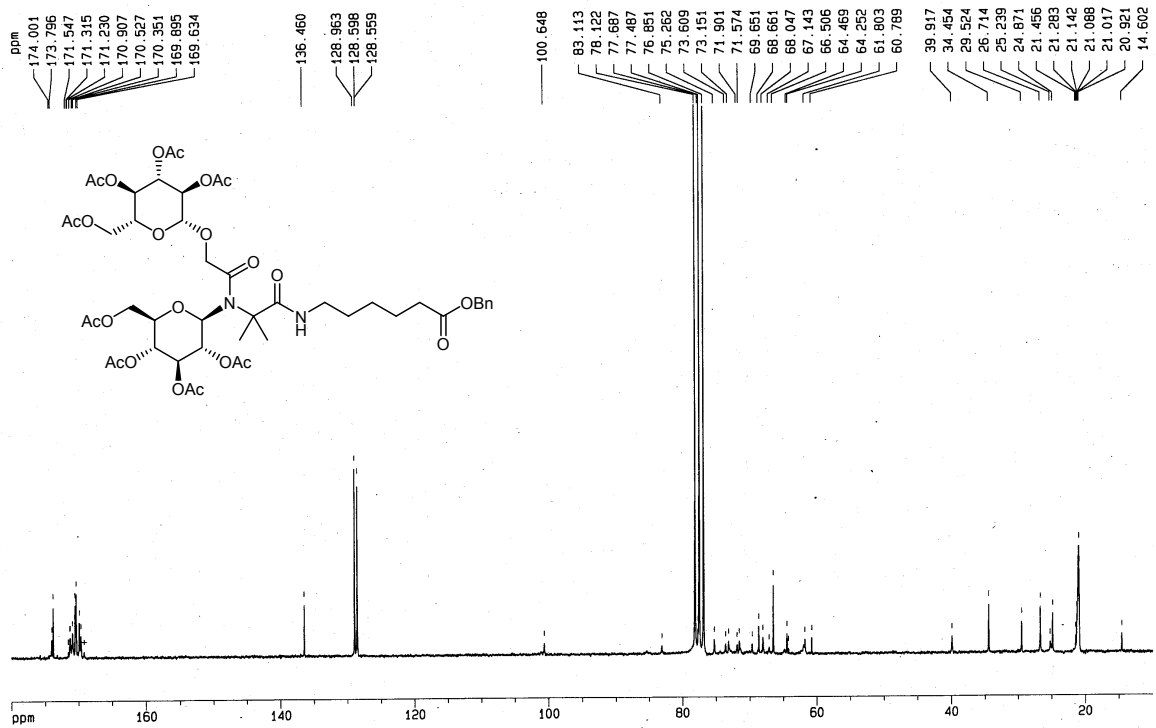
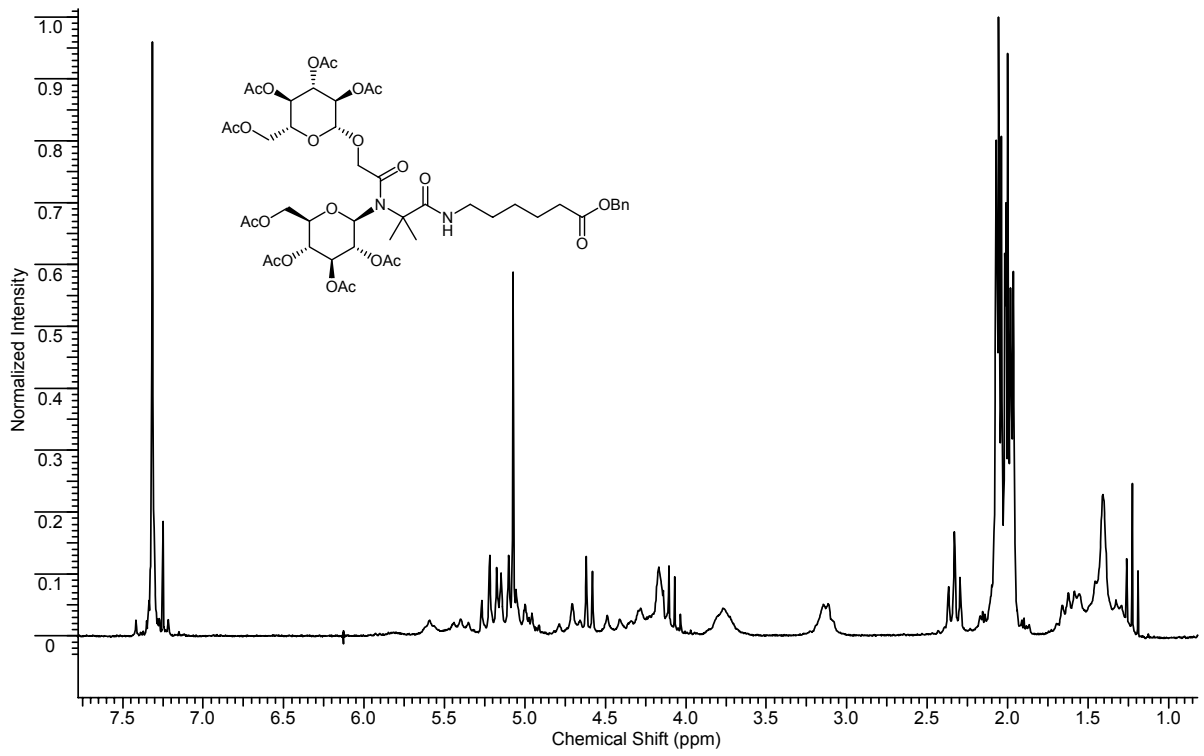


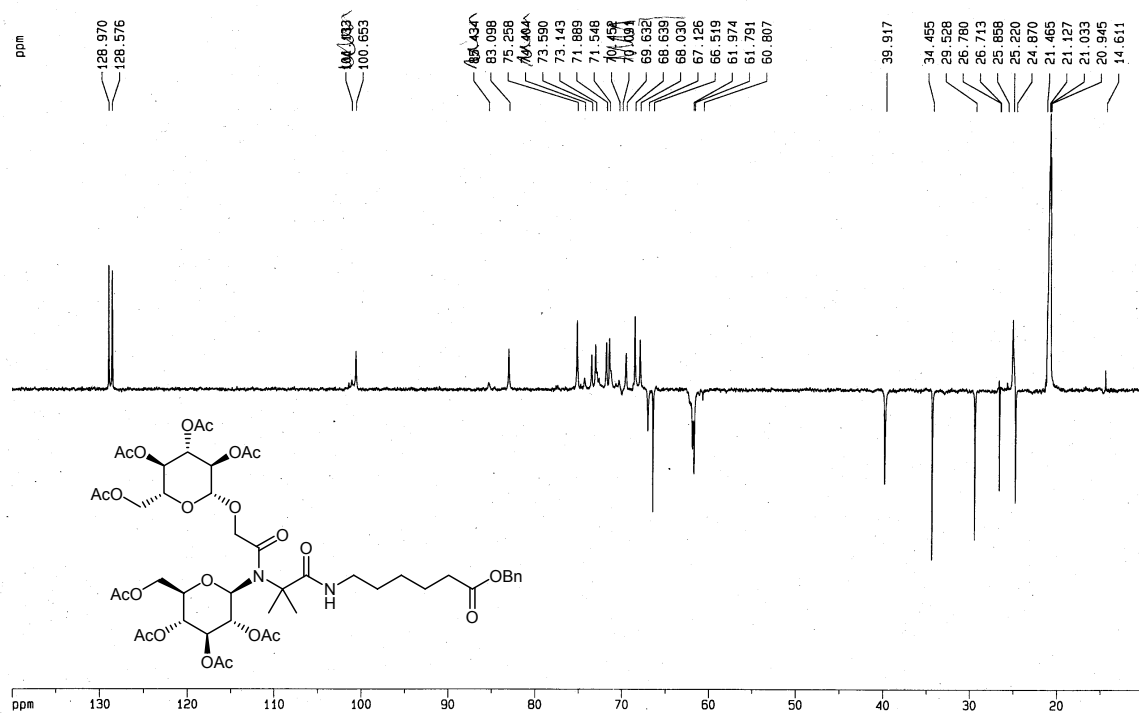


2,3,4,6-Tetra-*O*-acetyl-*N*-(2-{{6-(benzyloxy)-6-oxohexyl}amino}-1,1-dimethyl-2-oxoethyl)-*N*-{[(2,3,4,6-tetra-*O*-acetyl- β -D-glucopyranosyl)oxy]acetyl}- β -D-glucopyranosylamine (17)

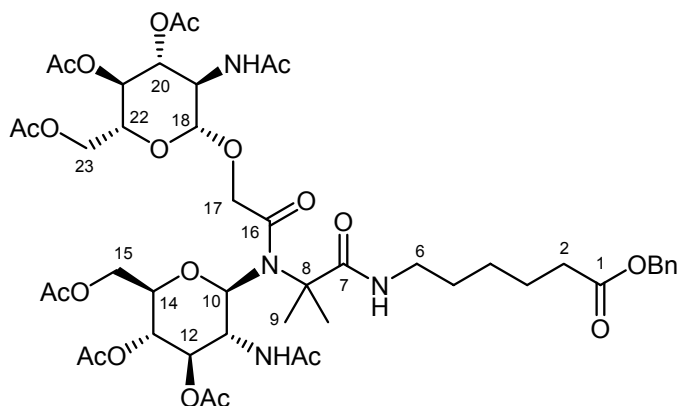


¹H-NMR (200 MHz, CDCl₃): δ [ppm] = 1.25–1.72 (m, 12H), 1.96, 1.98, 2.00, 2.01, 2.02, 2.04, 2.06, 2.07 (s, 3H), 2.33 (t, J = 7.3 Hz, 2H), 3.04–3.22 (m, 2H), 3.66–3.88 (m, 2H), 4.11–5.91 (m, 17H), 7.31 (s, 5H); ¹³C-NMR (50 MHz, CDCl₃): δ [ppm] = 20.92, 21.02, 21.09, 21.14, 21.28, 21.46 (q, CH_{3(Ac)}), 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, OCH₂), 64.47 (s, C-8), 66.52, 67.13 (t, OCH₂), 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 C₄₇H₆₄N₂O₂₃Na [M+Na⁺]: 1047.3792, found: 1047.3805; Anal. calcd for C₄₇H₆₄N₂O₂₃: C, 55.07; H, 6.29; N, 2.73; found: C, 54.64; H, 6.12; N, 2.76

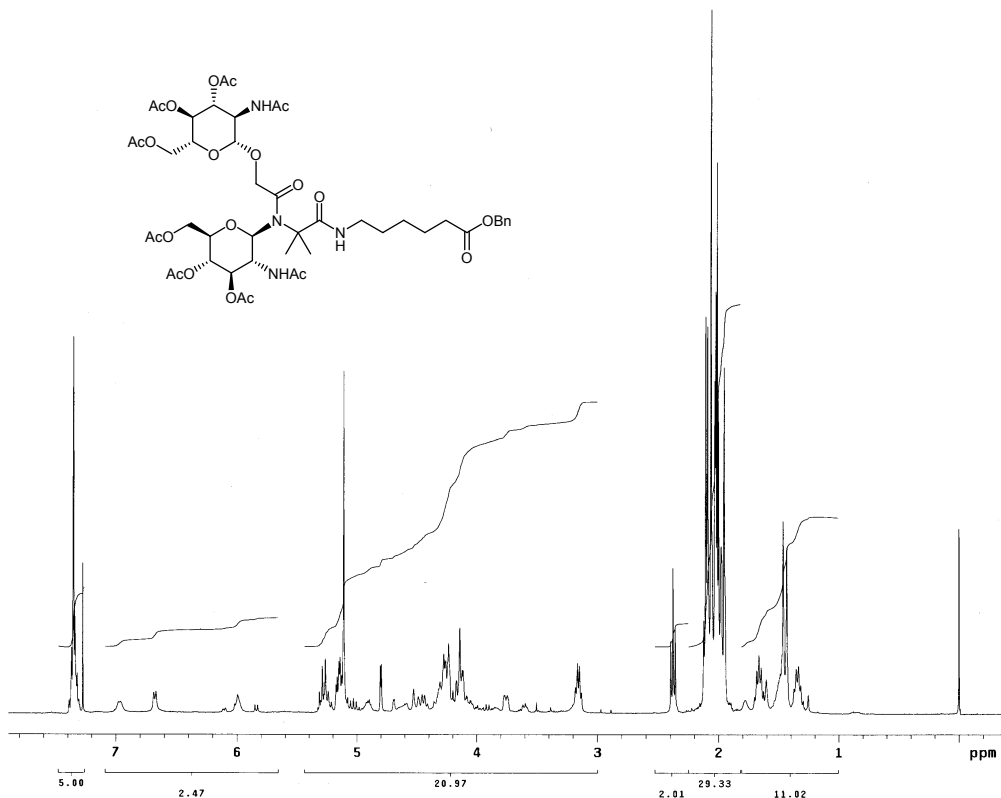




3,4,6-Tri-*O*-acetyl-2-(acetylamino)-*N*-(2-{[6-(benzyloxy)-6-oxohexyl]amino}-1,1-dimethyl-2-oxoethyl)-2-deoxy-*N*-({[3,4,6-tri-*O*-acetyl-2-(acetylamino)-2-deoxy- β -D-glucopyranosyl]oxy}acetyl)- β -D-glucopyranosylamine (18)



¹H-NMR (500 MHz, CDCl₃): 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); **¹³C-NMR** (125 MHz, CDCl₃): δ [ppm] = 20.50, 20.54, 20.60, 20.65, 20.69, 20.73 (q, CH₃(OAc)), 23.15, 23.20 (q, CH₃(NHAc)), 24.43, 26.25 (t, CH₂), 26.82 (q, C-9), 28.95, 34.04, 39.61 (t, CH₂), 51.74, 53.58 (d, C-11, C-19), 61.49, 61.71, 62.00, 66.11 (t, OCH₂), 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 C₄₇H₆₆N₄O₂₁Na [M+Na⁺]: 1045.4112, found: 1045.4114



1H Standard-Parameter
 Wed Jul 21 19:06 2004
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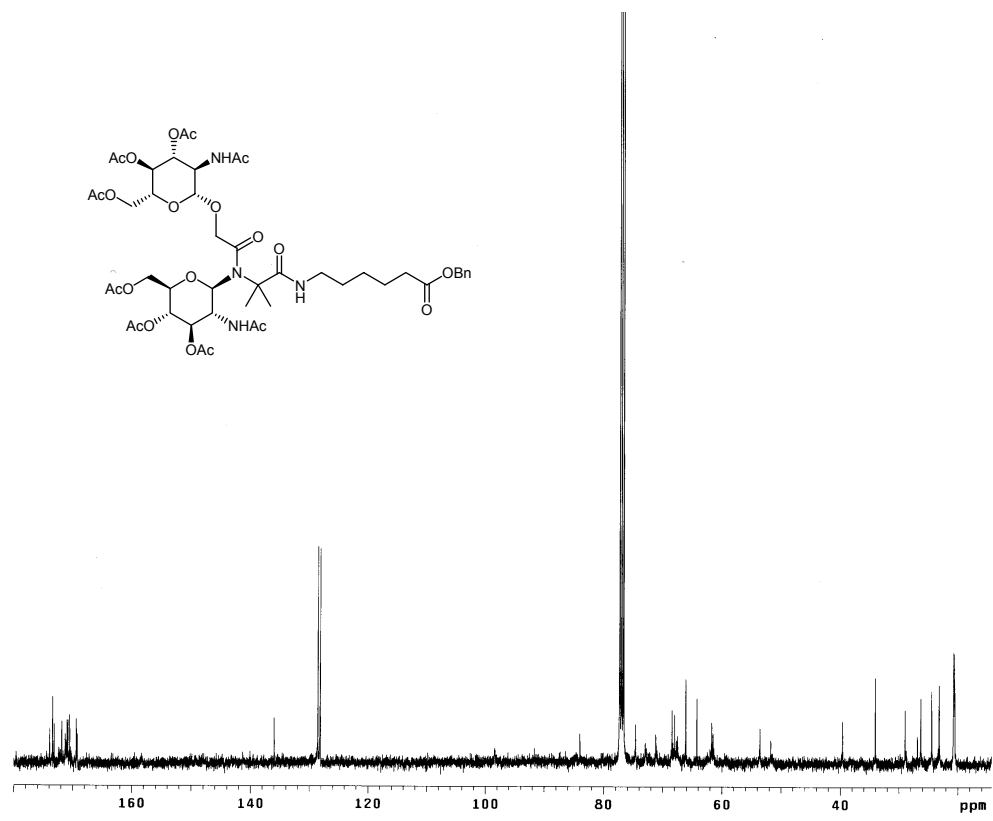
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 gain not used

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 in nw
 dp y

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 fn

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dos247.13C
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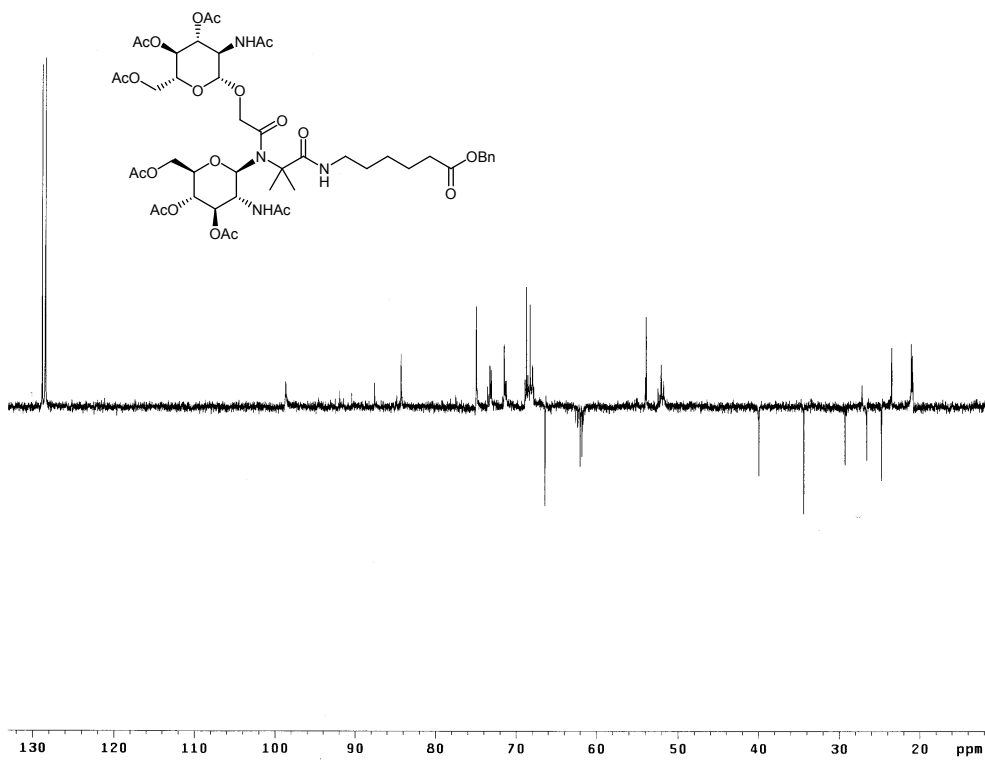
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 in n
 dp y

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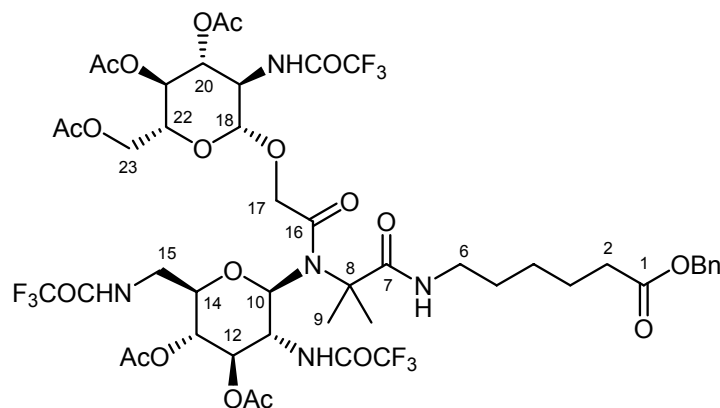


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probe agv5
dept
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sw 25641.9
bs 16
ss 8
tpwr 61
pw 14.7
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d2 0
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ct 8000
alock n
gain not used
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in n
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dn H1
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dpwr 40
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dmm ccw
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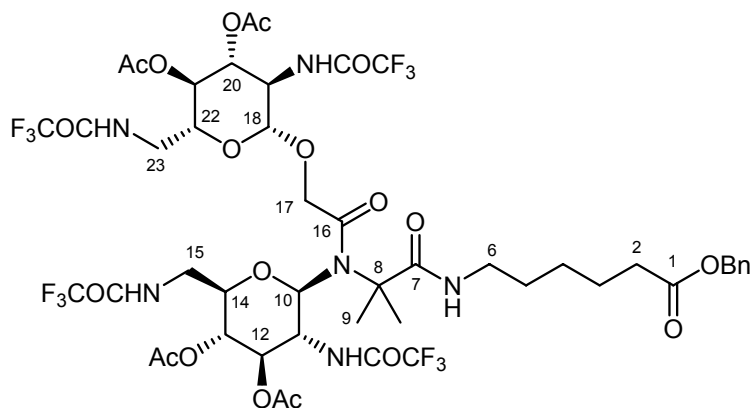
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3,4-Di-*O*-acetyl-*N*-(2-{[6-(benzyloxy)-6-oxohexyl]amino}-1,1-dimethyl-2-oxoethyl)-2,6-dideoxy-*N*-[(3,4,6-tri-*O*-acetyl-2-deoxy-2-[(trifluoroacetyl)amino]- β -D-glucopyranosyl]-oxy)acetyl]-2,6-bis[(trifluoroacetyl)amino]- β -D-glucopyranosylamine (19)

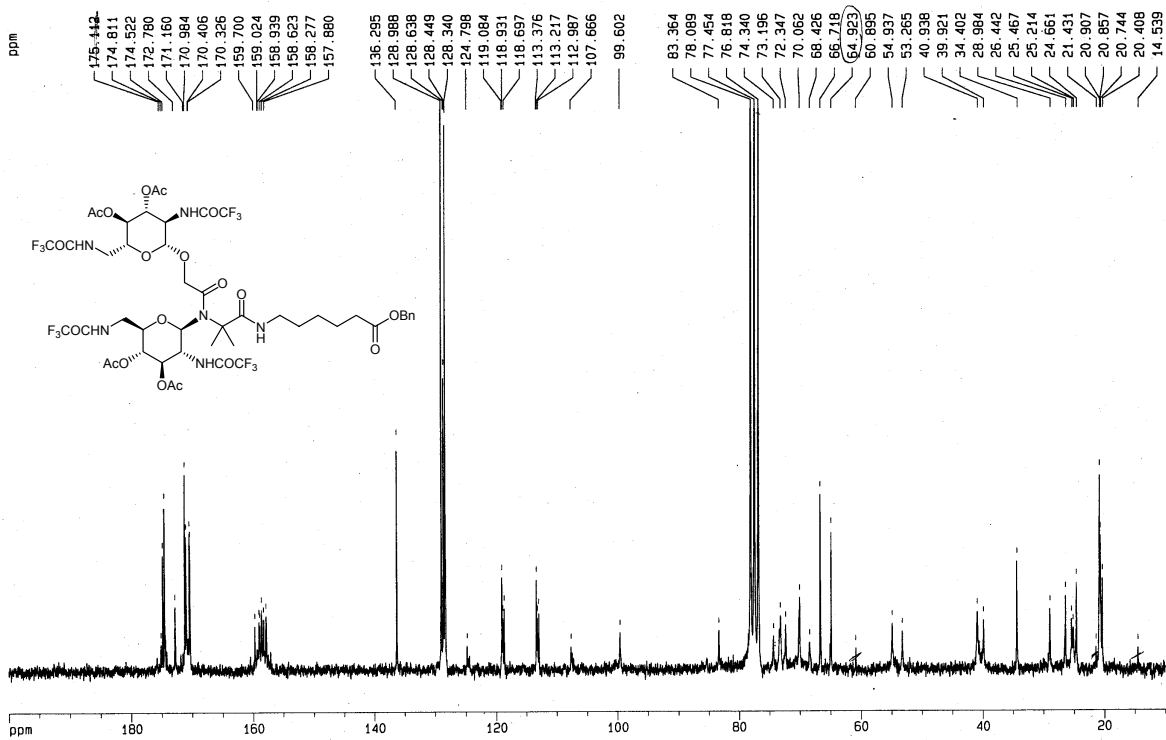
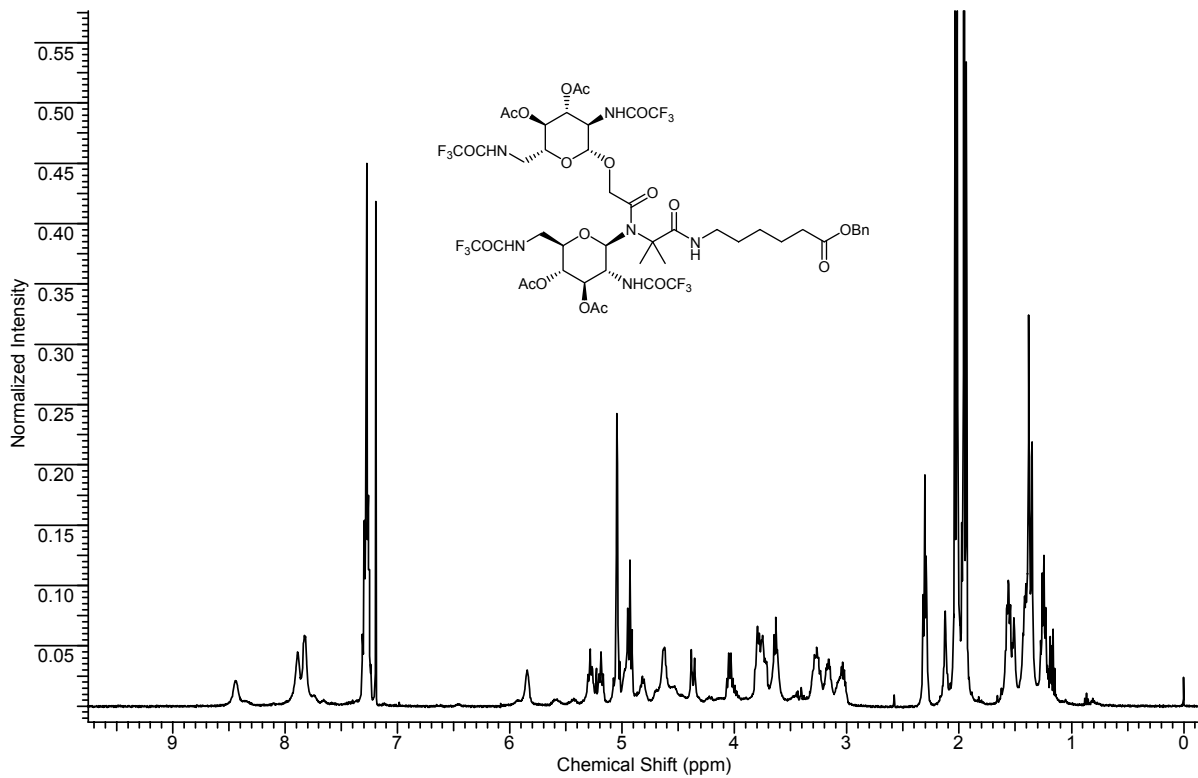


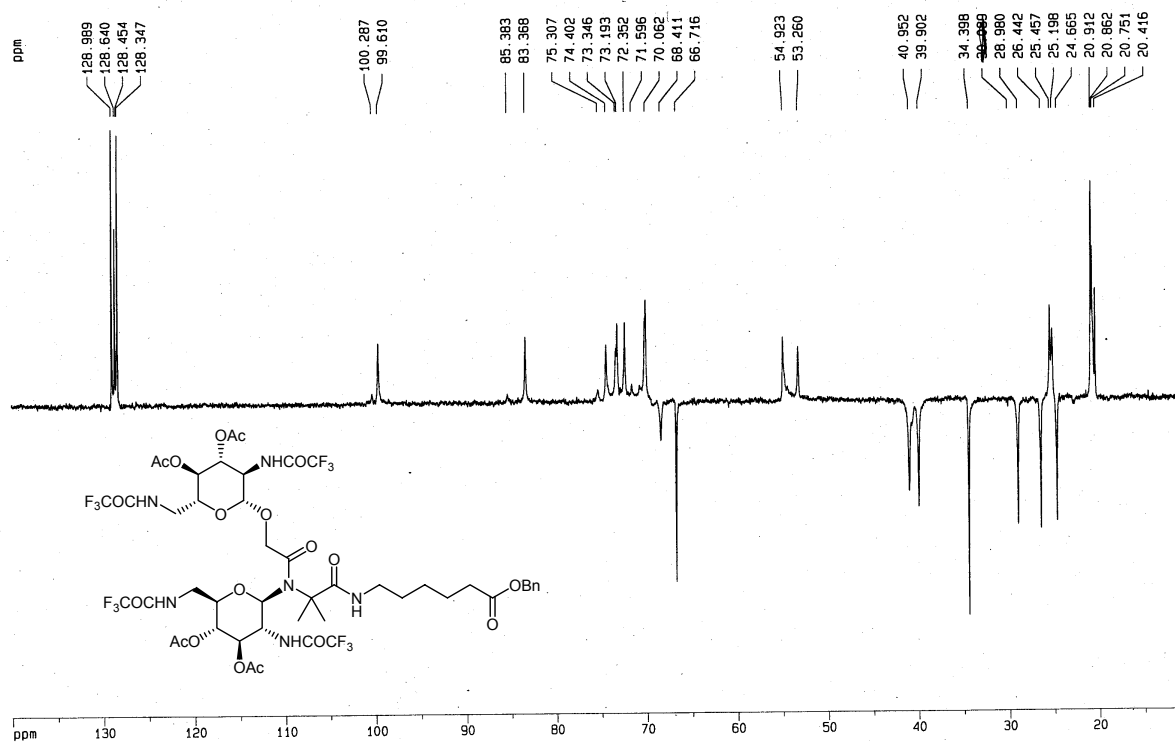
¹H-NMR (500 MHz, CDCl₃): δ [ppm] = 1.30–1.39 (m, 2H), 1.40–1.74 (m, 8H), 1.77–1.89 (m, 2H), 2.03, 2.03, 2.06, 2.10, 2.11 (s, 3H), 2.38 (t, J = 7.2 Hz, 2H), 3.12–3.36 (m, 2H), 3.72–3.86 (m, 4H), 4.05–4.31 (m, 4H) 4.44–4.66 (m, 2H), 4.76 (d, J = 8.2 Hz, 1H), 4.92–5.05 (m, 2H), 5.10–5.22 (m, 4H), 5.30 (bt, J = 9.1 Hz, 1H), 5.82 (bs, 1H), 7.22–7.32 (m, 5H), 7.70–8.18 (m, 3H); **¹³C-NMR** (125 MHz, CDCl₃): 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, OCH₂), 64.40 (s, C-8), 66.29, 67.88 (t, OCH₂), 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, CF₃), 128.05, 128.22, 128.58 (d, CH arom.), 135.98 (s, C arom.), 158.06 (m, C(O)CF₃), 169.32, 170.78, 170.80, 170.81, 170.86, 171.00, 173.97, 174.00 (s, CO); **HRMS (ESI)** calcd for C₄₇H₅₈F₉N₅O₂₀Na [M+Na⁺]: 1206.3424, found: 1206.3421

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-glucopyranosyl}oxy)acetyl]-2,6-bis[(trifluoroacetyl)amino]- β -D-glucopyranosylamine (20)

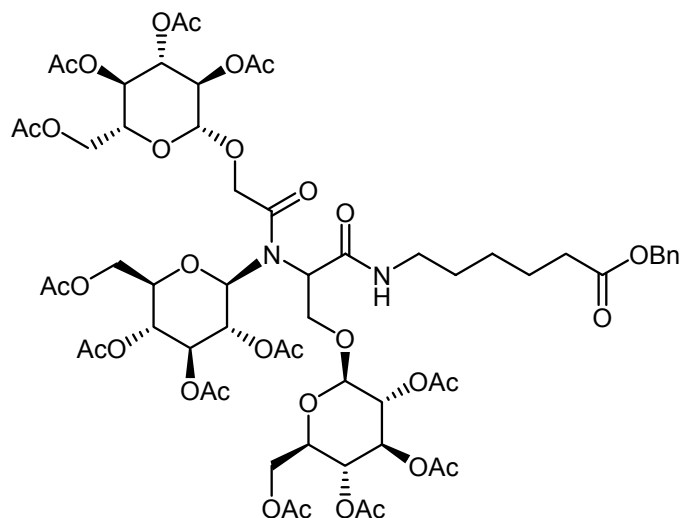


¹H-NMR (500 MHz, CDCl₃): δ [ppm] = 1.20–1.38 (m, 3H), 1.38–1.55 (m, 6H), 1.55–1.70 (m, 3H), 1.81 (s, 2H), 2.02, 2.04, 2.10, 2.12 (s, 3H), 2.38 (t, J = 7.2 Hz, 2H), 3.04–3.39 (m, 4H), 3.60–3.92 (m, 4H), 4.02–4.16 (m, 1H), 4.37–4.48 (d, J = 15.6 Hz, 1H), 4.61–4.71 (m, 1H), 4.80–4.93 (m, 1H), 4.93–5.05 (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); **¹³C-NMR** (125 MHz, CDCl₃): 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, OCH₂), 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, CF₃), 128.35, 128.64, 128.99 (d, CH arom.), 136.30 (s, C arom.), 159.02 (m, C(O)CF₃), 170.33, 170.41, 170.98, 171.16, 172.78, 174.52, 174.81 (s, CO); **HRMS (ESI)** calcd for C₄₇H₅₆F₁₂N₆O₁₉Na [M+Na⁺]: 1259.3301, found: 1259.3264

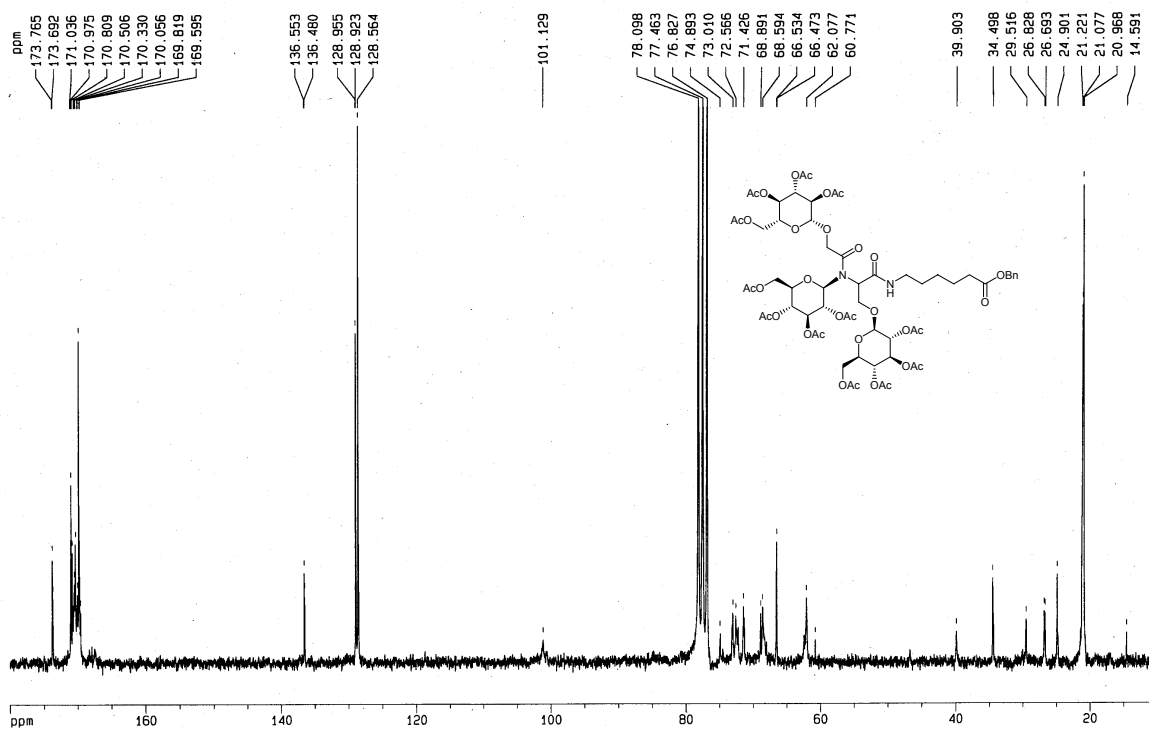
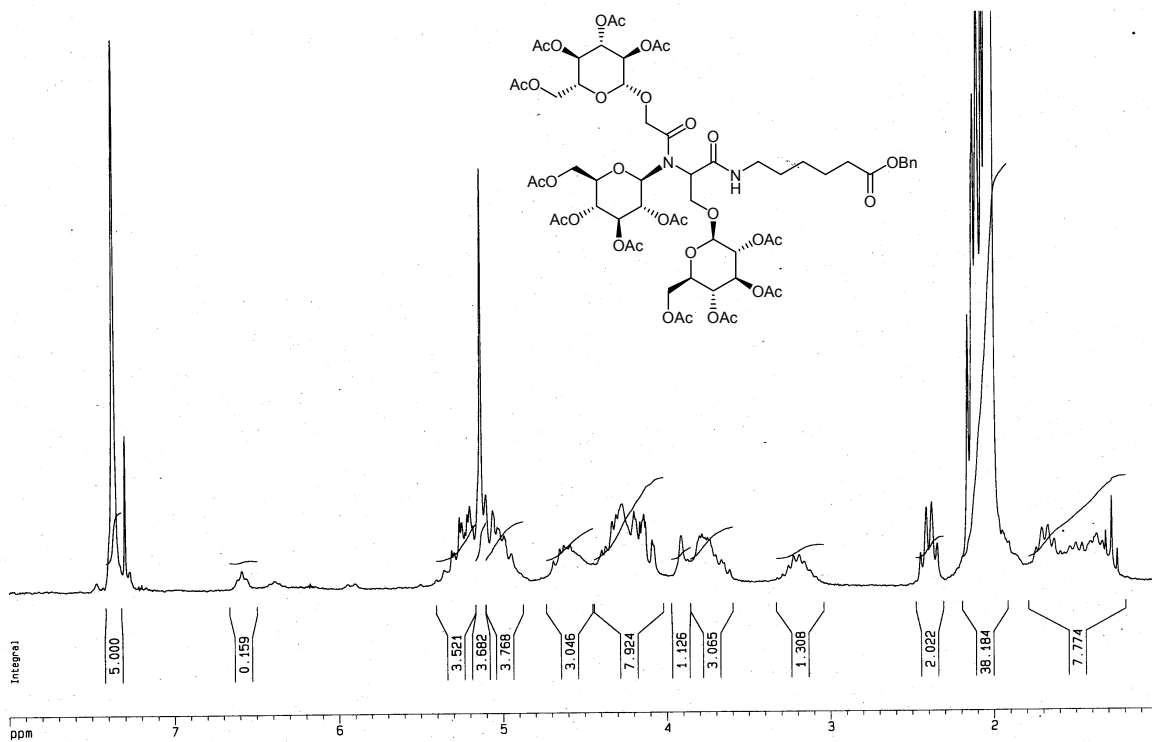


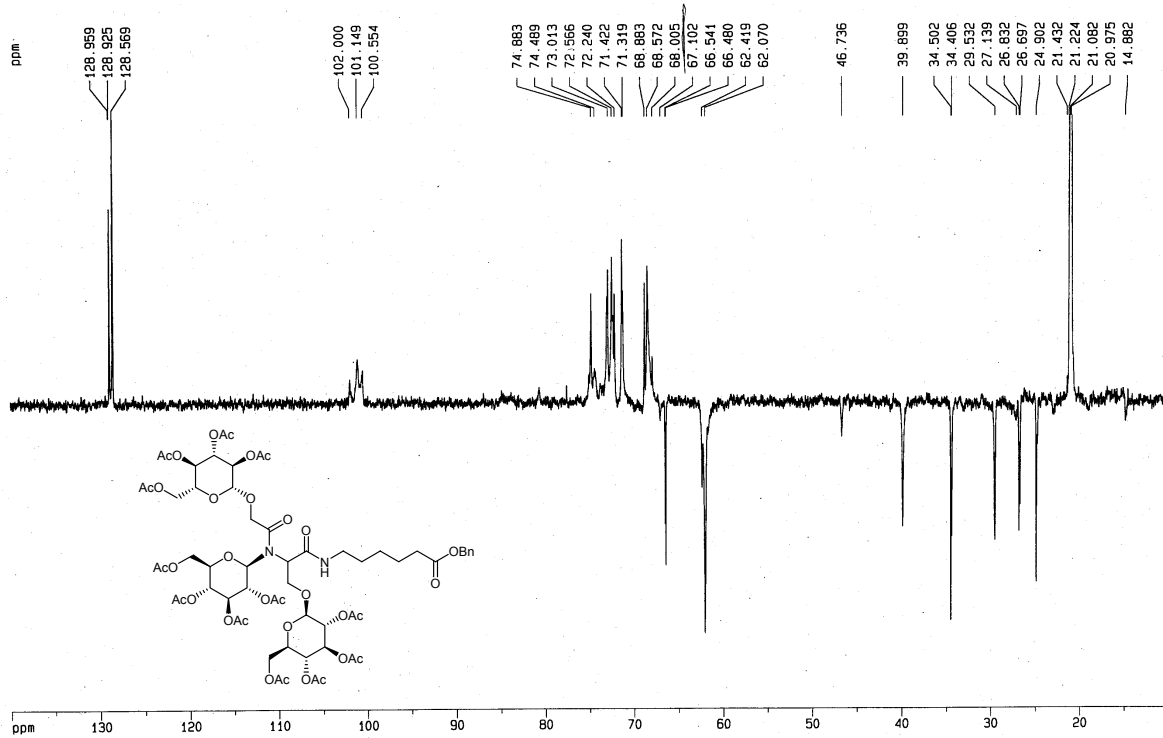


2,3,4,6-Tetra-*O*-acetyl-*N*-((1*R*,5*S*)-2-{{6-(benzyloxy)-6-oxohexyl}amino}-2-oxo-1-{{(2,3,4,6-tetra-*O*-acetyl- β -D-glucopyranosyl)oxy}methyl}ethyl)-*N*-{{(2,3,4,6-tetra-*O*-acetyl- β -D-glucopyranosyl)oxy}acetyl}- β -D-glucopyranosylamine (21)

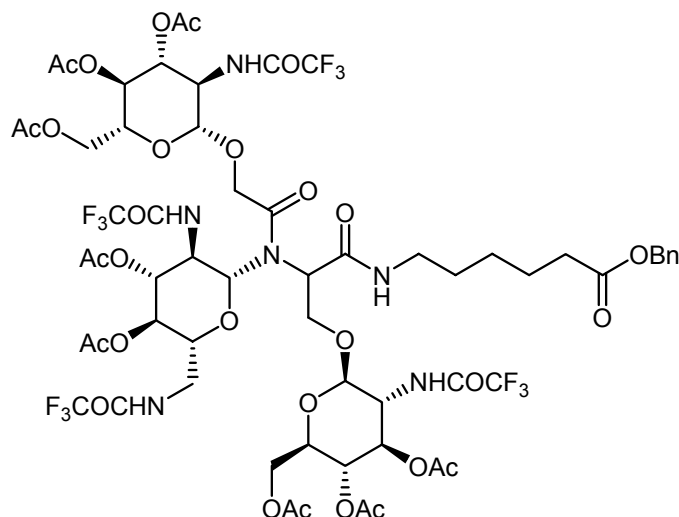


¹H-NMR (200 MHz, CDCl₃): isomeric mixture: δ [ppm] = 1.19–1.79 (m, 6H), 1.92–2.19 (m, 36H), 2.48–2.31 (m, 2H), 3.04–3.33 (m, 2H), 3.60–5.41 (m, 28H), 6.49–6.66 (m, 1H), 7.37 (s, 5H); **¹³C-NMR** (50 MHz, CDCl₃): signal ranges: δ [ppm] = 20.98–21.43 (q, CH_{3(Ac)}), 24.90, 26.83, 29.53, 34.50, 39.90, 46.74, 62.07, 62.42, 66.48 (t, CH₂, OCH₂), 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 C₆₀H₈₀N₂O₃₃Na [M+Na⁺]: 1379.4536, found: 1379.4491

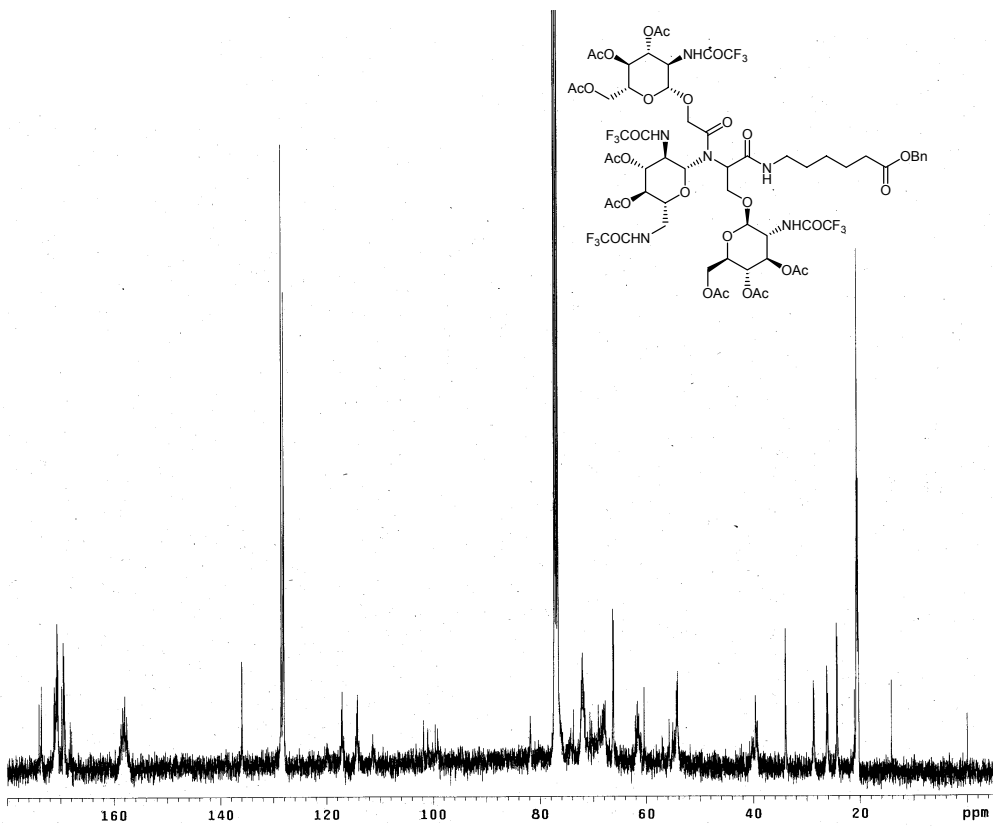
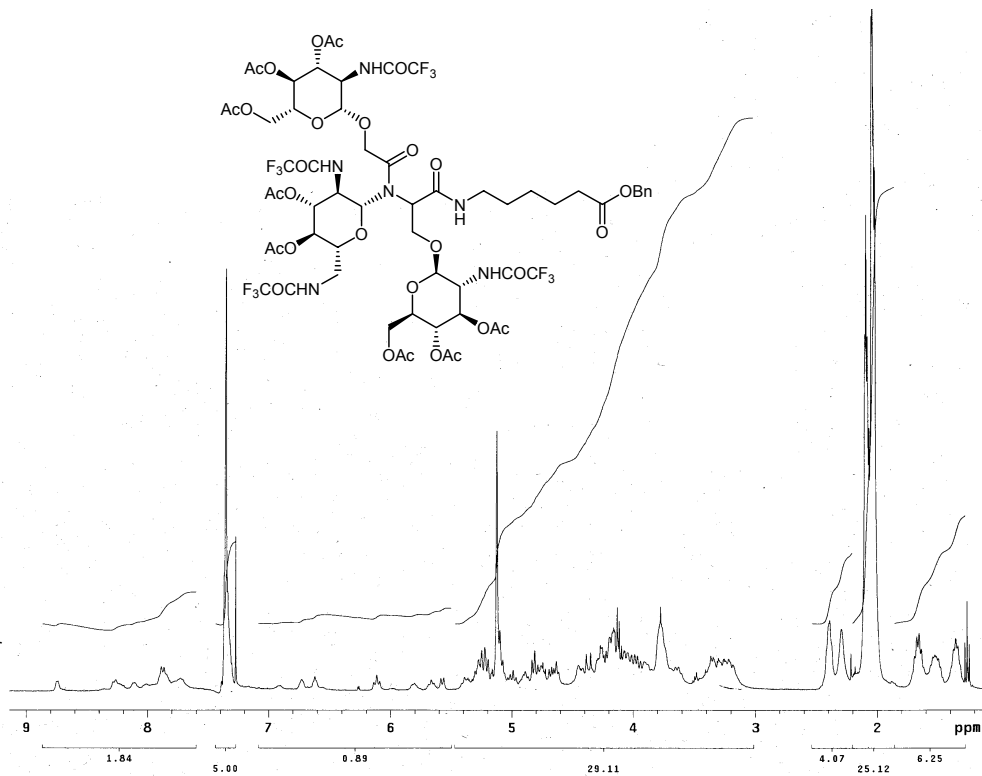


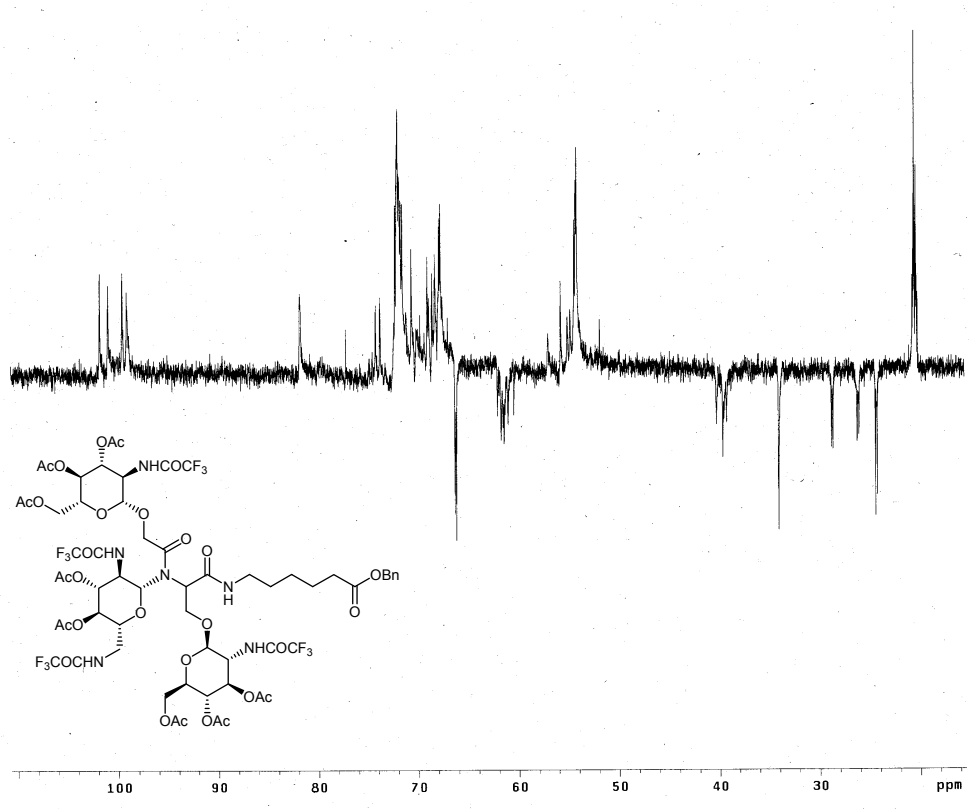


3,4-Di-*O*-acetyl-*N*-{(1*R*,5)-2-{{6-(benzyloxy)-6-oxohexyl}amino}-2-oxo-1-[(3,4,6-tri-*O*-acetyl-2-deoxy-2-[(trifluoroacetyl)amino]-β-D-glucopyranosyl}oxy)methyl]ethyl}-2,6-dideoxy-*N*-[(3,4,6-tri-*O*-acetyl-2-deoxy-2-[(trifluoroacetyl)amino]-β-D-glucopyranosyl]-oxy)acetyl]-2,6-bis[(trifluoroacetyl)amino]-β-D-glucopyranosylamine (22)



¹H-NMR (400 MHz, CDCl₃): 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); **¹³C-NMR** (100 MHz, CDCl₃): 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, CH₂, NCH₂), 54.18–54.60 (d, NCH), 61.20–62.03, 66.29 (t, OCH₂), 67.75–101.81 (d, CH sugar), 116.08 (m, CF₃), 127.99, 128.10, 128.54 (d, CH arom.), 135.95 (s, C arom.), 158.03 (m, C(O)CF₃), 167.92–171.30 (s, CO); **HRMS (ESI)** calcd for C₆₀H₇₂F₁₂N₆O₂₉Na [M+Na⁺]: 1591.4044, found: 1591.4009





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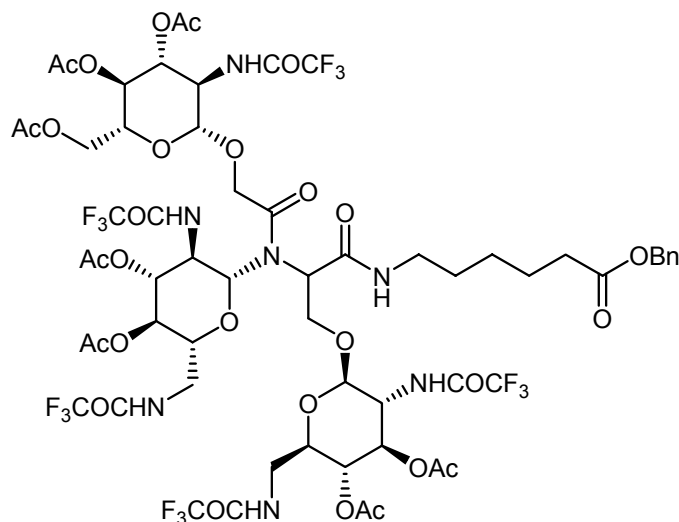
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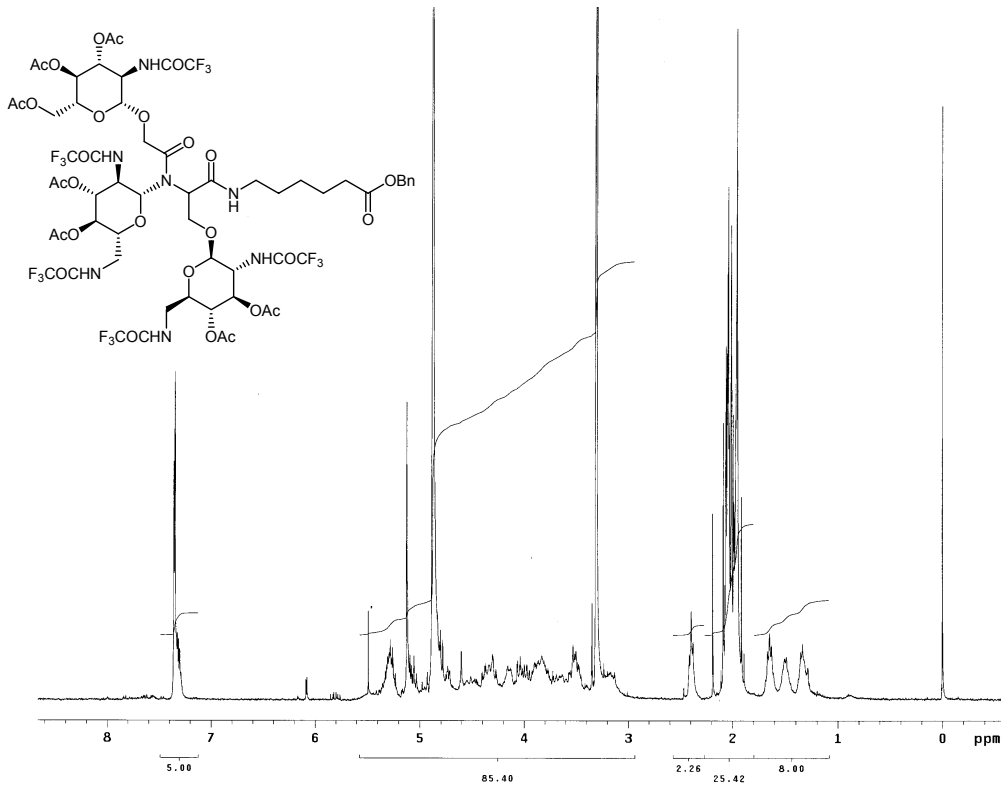
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dmm ccw
dwt 9350
pp1v1 60
pp 11.5
j 140.0
PROCESSING
lb 1.00
wtfile ft
proc fn
fn 65536
wexp procp1ot
DISPLAY
sp 1555.9
wp 9578.9
vs 232
sc 65
wc 225
hzmm 42.57
rfl 1998.3
rfp 0
th 3.8
rp -89.7
lp -247.2
nm no ph

```

3,4-Di-*O*-acetyl-*N*-{(1*R*,5)-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)



¹H-NMR (400 MHz, CDCl₃): 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.); **¹³C-NMR** (100 MHz, CDCl₃): 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, CH₂, NCH₂), 51.68–55.45 (d, NCH), 62.63–66.91 (t, OCH₂), 69.46–103.58 (d, CH sugar), 116.08 (m, CF₃), 127.97, 128.00, 128.34 (d, CH arom.), 136.55 (s, C arom.), 158.03 (m, C(O)CF₃), 169.54–174.03 (s, CO); **HRMS (ESI)** calcd for C₆₀H₇₀F₁₅N₇O₂₈Na [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

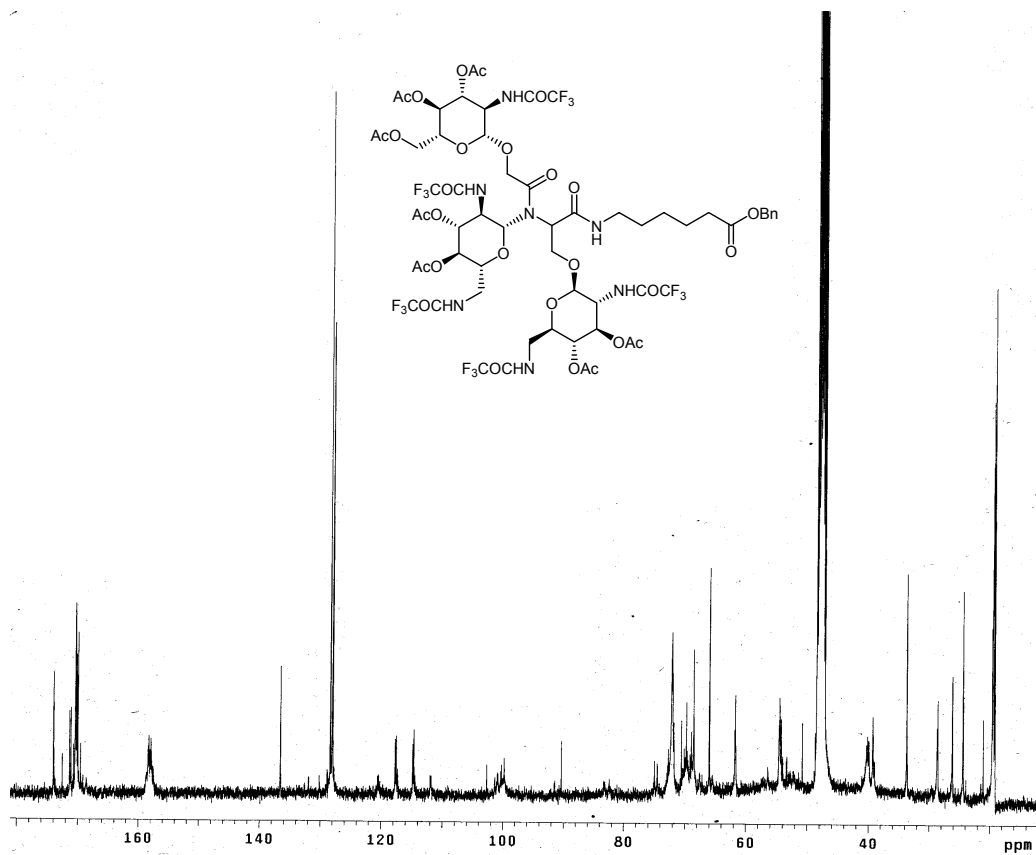
ACQUISITION
instrum m400
probe asw5
seqf1 82pu1
sfrq 399.939
tn H1
at 2.562
np 65536
sw 12787.7
bs 9
tpwr 58
pw 2.8
d1 1.000
d2 0
tof 828.3
nt 40
ct 40
alock n
gain not used

FLAGS
il n
in nw
dp DEC. & VT y

dn H1
dfrq 399.938
homo n
dpwr 30
dof 0
dm nnn
dmm C
dmf 200

PROCESSING
lb 0.40
wtfile ft
proc 65536

DISPLAY
sp -238.1
wp 3702.4
vs 2585
sc 65
wc 225
hzmm 18.46
is 323.73
rf1 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

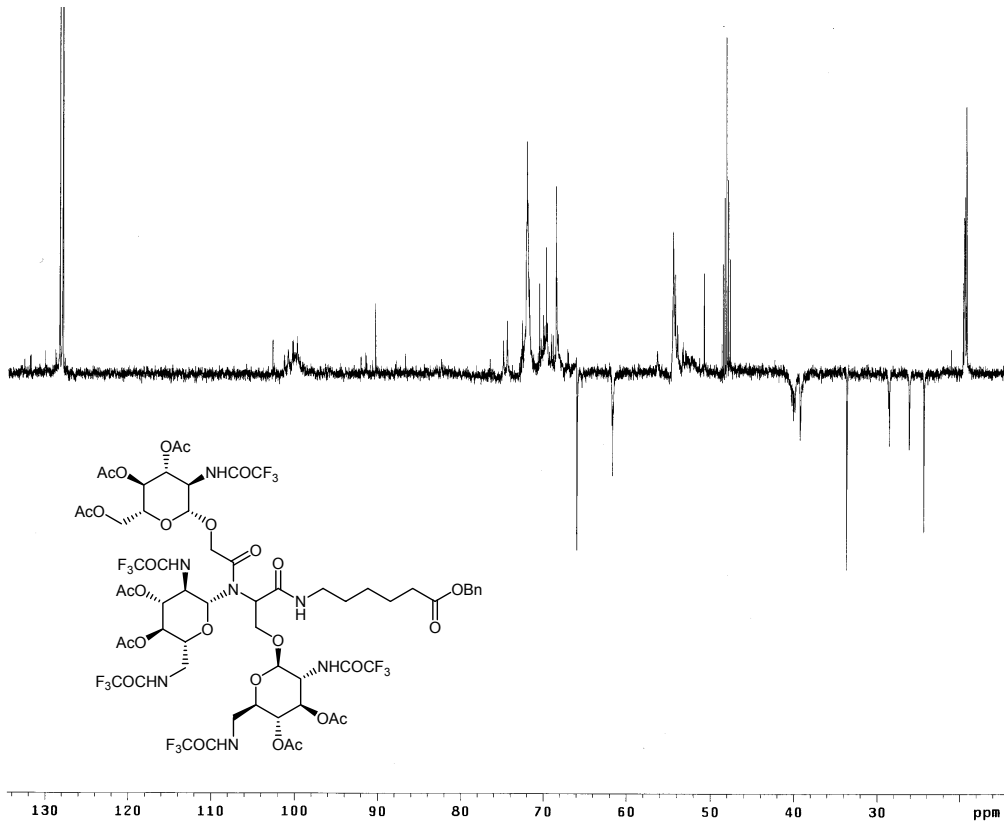
ACQUISITION
instrum m400
probe asw5
seqf1 82pu1
sfrq 100.575
tn C13
at 1.311
np 87216
sw 25641.8
bs 16
tpwr 61
pw 4.0
d1 1.689
d2 0
tof 1327.2
nt 45000
ct 45000
alock n
gain not used

FLAGS
il n
in nw
dp DEC. & VT y

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 ft
proc 65536

DISPLAY
sp 1219.0
wp 17053.6
vs 4503
sc 65
wc 225
hzmm 75.79
rf1 1970.6
rfp 0
th 2.1
ins -164.1
rp -348.4
nm ph
  
```



```

dos250.dept135.MeOD
Sun Aug  8 07:35 2004
exp7  dept
SAMPLE
date Aug  6 2004 1p
solvent CD3OD nm
file /export/home/~
nwc/dos/dos250.dept
135.MeOD.fid
ACQUISITION
instrum m400
probe asw5
seqfil dept
sfrq 100.575
tn 013
at 1.311
np 67216
sw 25641.0
bs 16
ss 8
tpwr 51
pw 14.7
d1 2.000
d2 0
tof 1327.2
nt 28000
ct 0
alock n
gain not used
SPECIAL
j mult 140.0
mult 1.5
FLAGS
i1 n
in n
dp y
DEC. & VT
dn H1
dfrq 399.938
homo n
dpwr 48
dcf 0
dm nny
dmm ccm
dmf 9350
pp1v1 60
pp 11.5
j 140.0
PROCESSING
lb 1.00
wtfile
proc ft
fn 65536
wexp procp1ot
DISPLAY
sp 1457.6
wp 12074.4
vs 145
sc 65
wc 225
hzmm 53.66
rf1 1970.6
rfp 0
tn 20.0
rp 25.4

```