Supporting information for

Diastereoselective addition of sugar radicals to camphorsultam glyoxilic oxime ether: A route toward *C*-glycosylthreonine and allothreonine

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Table of contents **page**

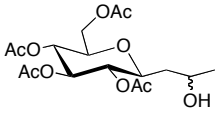
General Methods

All moisture-sensitive reactions were performed under argon using oven-dried glassware. Whenever necessary, solvents were distilled prior to use. Reactions were monitored on t.l.c. (silica gel 60 F_{254}). Detection was performed using UV light and/or 5% sulfuric or phosphomolybdic acid in ethanol, followed by heating. Flash chromatography was performed on silica gel 6-35 μm. ^{1}H- and ^{13}C-NMR spectra were recorded at room temperature with Bruker spectrometers (250, 300, 360 or 400 MHz). Chemical shifts are reported in δ vs Me_4Si for ^{1}H-NMR spectra in $CDCl_3$, and relative to acetone for ^{13}C-NMR spectra in D_2O. Optical rotations were measured on an Electronic Digital Jasco DIP-370 Polarimeter. These optical rotations were given for pure compounds (as judged by NMR, chromatography and centesimal analysis). Mass spectra were recorded in positive mode on a Finnigan MAT 95 S spectrometer using electrospray ionisation. Elemental analysis were performed at the Service Central de Microanalyses du CNRS (Gif-sur-Yvette, France). X-ray structures were determined with Bruker KAPPA APEX II.

Experimental procedure and characterisation of products 10a,b; 11a,b; 12a,b

To a 0.1 M solution of the glycosidic ketone **7**, **8** or **9** in dry MeOH was added $NaBH_4$ (2 equiv) at -10 °C under argon. The reaction mixture was stirred at-10 °C for 20 min, then warmed to 0 °C, diluted with CH_2Cl_2 and neutralized by KH_2PO_4 (10% aq). The aqueous phase was extracted with CH_2Cl_2 and the combined organic phases were filtered through a phase separator filter, then concentrated to give the alcohol in quantitative yield. Alcohols were iodinated without any further purification. Analytical samples were obtained as described below.

1-*C*-(2',3',4',6'-Tetra-*O*-acetyl-β-D-glucopyranosyl)-2(*S*)- and 2(*R*)-propan-2-ol (10a, 10b)



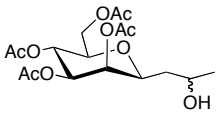
From 1-*C*-(2',3',4',6'-tetra-*O*-acetyl-β-D-glucopyranosyl)-propan-2-one **4** (100 mg, 0.26 mmol). **10a,b** were obtained as a 2:1 mixture (101 mg, 100%). The major isomer **10a** was isolated by HPLC (Column dimension: 250 x 20 mm; Material: SiO_2, 5μm; Mobile phase:

85:15 Et_2O – Cyclohexane; Flow rate: 20 mL / min). Eluted first was **10a**: Mp. 93-94 °C (cyclohexane-EtOAc); $[\alpha]_D^{28}$ = + 8.9 (*c* = 1, $CHCl_3$); ^{1}H NMR (300 MHz, $CDCl_3$) δ 5.19 (dd, 1 H, $J_{2',3'} = J_{3',4'}$ = 9.5 Hz, H-3'), 5.02 (dd, 1 H, $J_{1',2'} = J_{2',3'}$ = 9.5 Hz, H-2'), 4.90 (dd, 1 H, $J_{4',5'}$ = 9.5 Hz, H-4'), 4.23 (dd, 1 H, $J_{6'a,6'b}$ = 12.5 Hz, $J_{5',6'a}$ = 2.5 Hz, H-6'a), 4.09 (dd, 1 H, $J_{5',6'b}$ = 6.5 Hz, H-6'b), 4.02 (ddd, 1 H, $J_{1a,2}$ = 11.5 Hz, $J_{2,3}$ = 6.5 Hz, H-2), 3.72 (ddd, 1 H, H-5'), 3.67 (ddd, 1 H, $J_{1',2'}$ = 9.5 Hz, H-1'), 3.02 (s, 1 H, O*H*), 2.10, 2.06, 2.05, 2.01 (4 s, 12 H, 4 Ac), 1.65 (2 ddd, 2 H, H-1a, H-1b), 1.20 (d, 3 H, H-3); ^{13}C NMR (90 MHz, $CDCl_3$) δ 20.4, 20.5 (CO*C*H_3), 23.1 (C3), 39.5 (C1), 62.2 (C2), 67.0 (C6'), 68.5 (C4'), 71.7 (C2'), 73.8 (C3'), 75.6 (C1'), 78.1 (C5'), 169.3, 169.5, 170.2, 170.5 (*C*OCH_3); HRMS (ESI) Calcd for $C_{17}H_{26}O_{10}Na$: 413.1418. Found 413.1423; Anal. Calcd for $C_{17}H_{26}O_{10}$ (%): C, 52.30; H, 6.71; O, 40.98. Found: C, 52.15; H, 6.76; O, 40.75.

Eluted second was **10b**, contaminated by 25% of **10a.**

10b: ^{1}H NMR (300 MHz, $CDCl_3$) δ = 5.21 (dd, 1 H, $J_{2',3'} = J_{3',4'}$ = 9.5 Hz, H-3'), 5.07 (dd, 1 H, $J_{1',2'} = J_{2',3'}$ = 9.5 Hz, H-2'), 4.95 (dd, 1 H, $J_{4',5'}$ = 9.5 Hz, H-4'), 4.25 (dd, 1 H, $J_{6'a,6'b}$ = 12.5 Hz, $J_{5',6'a}$ = 5.0 Hz, H-6'a), 4.13 (dd, 1 H, $J_{5',6'b}$ = 2.5 Hz, H-6'b), 4.11 (ddd, 1 H, H-2), 3.76 (ddd, 1 H, H-1'), 3.70 (ddd, 1 H, H-5'), 2.72 (s, 1 H,O*H*), 2.10, 2.06, 2.05, 2.02 (4 s, 12 H, 4 Ac), 1.60 (2 ddd, 2 H, H-1a, H-1b), 1.23 (d, 3 H, $J_{2,3}$ = 6.5 Hz, H-3); ^{13}C NMR (90 MHz, $CDCl_3$) δ 20.4, 20.5 (CO*C*H_3), 23.7 (C3), 39.7 (C1), 62.1 (C2), 63.6 (C6'), 65.6 (C4'), 71.4 (C2'), 74.1 (C3'), 74.9 (C1'), 75.5 (C5'), 169.3, 169.5, 170.2, 170.5 (*C*OCH_3).

1-*C*-(2',3',4',6'-Tetra-*O*-acetyl-β-D-mannopyranosyl)-2(*S*)- and 2(*R*)-propan-2-ol (11a, 11b)

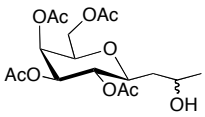


From 1-*C*-(2',3',4',6'-tetra-*O*-acetyl-β-D-mannopyranosyl)-propan-2-one **8** (100 mg, 0.26 mmol). **11a,b** were obtained as a 1:1 mixture (101 mg, 100%). The major isomer **11a** was isolated by HPLC (Column dimension: 250 x 20 mm; Material: SiO_2, 5μm; Mobile phase: 90:10 Et_2O – Cyclohexane; Flow rate: 20 mL / min). Eluted first was **11a**: Mp. 101-102 °C (cyclohexane-EtOAc); $[\alpha]_D^{28}$ = -11.9 (*c* = 1, $CHCl_3$); ^{1}H NMR (300 MHz, $CDCl_3$): δ 5.34 (dd, 1 H, $J_{1',2'}$ = 0.5 Hz, $J_{2',3'}$ = 3.5 Hz, H-2'), 5.20 (dd, 1 H, $J_{3',4'} = J_{4',5'}$ = 9.5 Hz, H-4'), 5.06 (dd, 1

H, H-3'), 4.10 (dd, 1 H, $J_{6'a,6'b}$ = 12.5 Hz, $J_{5',6'a}$ = 2.5 Hz, H-6'a), 4.11 (dd, 1 H, $J_{5',6'b}$ = 6.5 Hz, H-6'b), 3.99 (ddd, 1 H, $J_{1a,2}$ = 9.0 Hz, $J_{1b,2}$ = 2.5 Hz, $J_{2,3}$ = 6.5 Hz, H-2), 3.89 (ddd, 1 H, $J_{1a,1'}$ = 9.5 Hz, $J_{1b,1'}$ = 3.5 Hz, H-1'), 3.72 (ddd, 1 H, H-5'), 2.89 (s, 1 H,O*H*), 2.19, 2.10, 2.06, 1.99 (4 s, 12 H, 4 Ac), 1.76 (ddd, 1 H, $J_{1a,1b}$ = 14.5 Hz, H-1a), 1.52 (ddd, 1 H, H-1b), 1.19 (d, 3 H, $J_{2,3}$ = 6.5 Hz, H-3); ^{13}C NMR (90 MHz, $CDCl_3$): δ 20.6, 20.7 (CO*C*H$_3$), 23.9 (C3), 39.5 (C1), 62.9 (C2), 64.4 (C6'), 66.3 (C4'), 70.7 (C2'), 72.4 (C3'), 74.0 (C1'), 76.2 (C5'), 169.7, 170.1, 170.6, 170.7 (*C*OCH$_3$); HRMS (ESI) Calcd for $C_{17}H_{26}O_{10}Na$: 413.1418. Found 413.1414; Anal. Calcd for $C_{17}H_{26}O_{10}$ (%): C, 52.30; H, 6.71; O, 40.98. Found: C, 52.43; H, 6.76; O, 40.85. Eluted second was **11b** contaminated by **11a**.

11b*:* ^{1}H NMR (300 MHz, $CDCl_3$): δ 5.32 (dd, 1 H, $J_{1',2'}$ = 0.5 Hz, $J_{2',3'}$ = 3.5 Hz, H-2'), 5.24 (dd, 1 H, $J_{3',4'}$ = $J_{4',5'}$ = 9.5 Hz, H-4'), 5.08 (dd, 1 H, H-3'), 4.27 (dd, 1 H, $J_{6'a,6'b}$ = 12.5 Hz, $J_{5',6'a}$ = 6.0 Hz, H-6'a), 4.12 (dd, 1 H, $J_{5',6'b}$ = 2.5 Hz, H-6'b), 4.02 (ddd, 1 H, $J_{1a,2}$ = 2.5 Hz, $J_{1b,2}$ = 9.5 Hz, $J_{2,3}$ = 6.5 Hz, H-2), 3.96 (ddd, 1 H, $J_{1a,1'}$ = 9.5 Hz, $J_{1b,1'}$ = 3.0 Hz, H-1'), 3.68 (ddd, 1 H, H-5'), 2.19, 2.10, 2.06, 1.99 (4 s, 12 H, 4 Ac), 1.80 (ddd, 1 H, $J_{1a,1b}$ = 14.5 Hz, H-1a), 1.41 (ddd, 1 H, H-1b); ^{13}C NMR (90 MHz, $CDCl_3$): δ 20.4, 20.5 (CO*C*H$_3$), 23.5 (C3), 38.9 (C1), 62.9 (C2), 66.2 (C6'), 66.7 (C4'), 69.8 (C2'), 72.1 (C3'), 74.0 (C1'), 76.3 (C5'), 169.7, 170.1, 170.6, 170.7 (*C*OCH$_3$).

1-*C*-(2',3',4',6'-Tetra-*O*-acetyl-β-D-galactopyranosyl)-2(*S*)- and 2(*R*)-propan-2-ol (12a, 12b)



From 1-*C*-(2',3',4',6'-tetra-*O*-acetyl-β-D-galactopyranosyl)-propan-2-one **9** (100 mg, 0.26 mmol). **12a,b** were obtained as a 2:1 mixture (101 mg, 100%). The major isomer **12a** was isolated by HPLC (Column dimension: 250 x 20 mm; Material: SiO_2, 5μm; Mobile phase: 85:15 Et_2O – Cyclohexane; Flow rate: 20 mL / min). Eluted first was **12a**: $[\alpha]_D^{28}$ = + 17.9 (*c* = 1, $CHCl_3$); ^{1}H NMR (300 MHz, $CDCl_3$): δ 5.43 (dd, 1 H, $J_{3',4'}$ = 3.5 Hz, $J_{4',5'}$ = 0.5 Hz, H-4'), 5.11 (dd, 1 H, $J_{1',2'}$ = $J_{2',3'}$ = 9.5 Hz, H-2'), 5.01 (dd, 1 H, H-3'), 4.10 (dd, 1 H, $J_{6'a,6'b}$ = 12.5 Hz, $J_{5',6'a}$ = 5.5 Hz, H-6'a), 4.09 (dd, 1 H, $J_{5',6'b}$ = 6.0 Hz, H-6'b), 4.02 (ddd, 1 H, $J_{1a,2}$ = 3.5 Hz, $J_{1b,2}$ = 11.5 Hz, $J_{2,3}$ = 6.5 Hz, H-2), 3.94 (ddd, 1 H, H-5'), 3.64 (ddd, 1 H, $J_{1a,1'}$ = 2.5 Hz, $J_{1b,1'}$

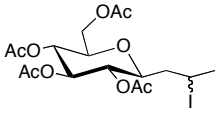
= 9.5 Hz, H-1'), 3.07 (s, 1 H, O*H*), 2.16, 2.06, 1.98 (4 s, 12 H, 4 Ac), 1.72 (ddd, 1 H, $J_{1a,1b}$ = 14.5 Hz, H-1a), 1.63 (ddd, 1 H, H-1b), 1.19 (d, 3 H, H-3); ^{13}C NMR (90 MHz, $CDCl_3$): δ 20.7 (CO*C*H$_3$), 23.3 (C3), 39.6 (C1), 62.1 (C2), 67.6 (C6', C4'), 69.1 (C2'), 71.8 (C3'), 74.5 (C1'), 79.1 (C5'), 169.8, 170.2, 170.5 (*C*OCH$_3$); HRMS (ESI) Calcd for $C_{17}H_{26}O_{10}Na$: 413.1418. Found 413.1421; Anal. Calcd for $C_{17}H_{26}O_{10}$ (%): C, 52.30; H, 6.71; O, 40.98. Found: C, 52.05; H, 6.71; O, 40.91. Eluted second was **12b** contaminated by **12a**.

12b: ^{1}H NMR (300 MHz, $CDCl_3$): δ 5.45 (dd, 1 H, $J_{3',4'}$ = 3.5 Hz, $J_{4',5'}$ = 0.5 Hz, H-4'), 5.16 (dd, 1 H, $J_{1',2'} = J_{2',3'}$ = 9.5 Hz, H-2'), 5.04 (dd, 1 H, H-3'), 4.16 (dd, 1 H, $J_{6'a,6'b}$ = 12.5 Hz, $J_{5',6'a}$ = 7.5 Hz, H-6'a), 4.07 (dd, 1 H, $J_{5',6'b}$ = 7.0 Hz, H-6'b), 4.05 (ddd, 1 H, $J_{2,3}$ = 6.5 Hz, H-2), 3.91 (ddd, 1 H, H-5'), 3.75 (ddd, 1 H, $J_{1a,1'}$ = 3.5 Hz, $J_{1b,1'}$ = 9.5 Hz, H-1'), 2.17, 2.07, 2.06, 2.00 (4 s, 12 H, 4 Ac), 1.63 (2 ddd, 2 H, $J_{1a,1b}$ = 14.5 Hz, H-1a, H-1b), 1.23 (d, 3 H, H-3); ^{13}C NMR (90 MHz, $CDCl_3$): δ 20.7 (CO*C*H$_3$), 23.8 (C3), 39.6 (C1), 61.7 (C2), 63.9 (C6'), 67.7 (C4'), 68.8 (C2'), 72.1 (C3'), 74.3 (C1'), 75.6 (C5'), 169.8, 170.2, 170.5 (*C*OCH$_3$).

Experimental procedure and characterisation of products 13a,b; 14a,b; 15a,b

To a solution of the alcohol **10a,b** or **11a,b** or **12a,b** (3.8 g; 9.74 mmol) in pyridine (80 mL) was added TsCl (2,8 g; 14.6 mmol) at r.t. under argon. The reaction mixture was kept at r.t for 24 h, then concentrated. The residue was suspended in CH_2Cl_2 and washed with brine. The organic layer was dried ($MgSO_4$) and concentrated. The residue was dissolved in acetone (80 mL) and NaI (7.3 g; 48 mmol) was added. The reaction mixture was refluxed for 24 h, then cooled to r.t., filtrated and concentrated. The residue was suspended in CH_2Cl_2 and washed with $Na_2S_2O_3$ (5% aq.). The organic layer was dried ($MgSO_4$) and concentrated. The residue was purified by flash chromatography.

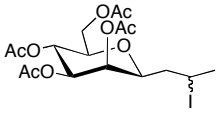
1-*C*-(2',3',4',6'-Tetra-*O*-acetyl-β-D-glucopyranosyl)-2(*S*)- and 2(*R*)-iodopropane (13a, 13b)



From **12a,b**. Flash chromatography (7:3 Petroleum ether / EtOAc) afforded **13a,b** as a 2:1 mixture. (4,8 g, 100%). The two isomers were separated by HPLC (Column dimension: 250 x 20 mm; Material: SiO_2, 5μm; Mobile phase: 40:60 Et_2O – Cyclohexane; Flow rate: 20 mL /

min). Eluted first was **13a**: Mp. 83-84 °C (cyclohexane); $[\alpha]_D^{28}$ = - 26.5 (*c* = 1, $CHCl_3$); ^1H NMR (360 MHz, $CDCl_3$): δ 5.23 (dd, 1 H, $J_{2',3'} = J_{3',4'}$ = 9.5 Hz, H-3'), 5.04 (dd, 1 H, $J_{1',2'}$ = 9.5 Hz, H-2'), 4.95 (dd, 1 H, $J_{4',5'}$ = 9.5 Hz, H-4'), 4.34 (ddd, 1 H, $J_{1a,2}$ = 11.5 Hz, $J_{1b,2}$ = 2.5 Hz, $J_{2,3}$ = 7.0 Hz, H-2), 4.27 (dd, 1 H, $J_{6'a,6'b}$ = 12.5 Hz, $J_{5',6'a}$ = 5.5 Hz, H-6'a), 4.10 (dd, 1 H, $J_{5',6'b}$ = 2.5 Hz, H-6'b), 3.70 (ddd, 1 H, H-5'), 3.67 (ddd, 1 H, $J_{1a,1'}$ = 2.5 Hz, $J_{1b,1'}$ = 9.5 Hz, H-1'), 2.10, 2.07, 2.03, 2.01 (4 s, 12 H, 4 Ac), 1.96 (d, 3 H, H-3), 1.85 (ddd, 1 H, $J_{1a,1b}$ = 14.5 Hz, H-1a), 1.56 (ddd, 1 H, H-1b); ^{13}C NMR (90 MHz, $CDCl_3$): δ 20.5, 20.6 ($COCH_3$), 21.9 (C2), 27.4 (C3), 44.2 (C1), 62.1 (C6'), 68.5 (C4'), 71.6 (C2'), 74.1 (C3'), 75.7 (C1'), 76.4 (C5'), 169.3, 169.4, 170.2, 170.5 ($COCH_3$); HRMS (ESI) Calcd for $C_{17}H_{25}O_9INa$: 523.0435. Found 523.0444; Anal. Calcd for $C_{17}H_{25}O_9I$ (%): C, 40.81; H, 5.04; O, 28.78. Found: C, 40.85; H, 5.17; O, 28.74. Eluted second was **13b**: Mp. 120-121 °C (cyclohexane); $[\alpha]_D^{28}$ = + 0.16 (*c* = 1, $CHCl_3$); ^1H NMR (360 MHz, $CDCl_3$): δ 5.20 (dd, 1 H, $J_{2',3'} = J_{3',4'}$ = 9.5 Hz, H-3'), 5.04 (dd, 1 H, $J_{1',2'}$ = 9.5 Hz, H-2'), 4.90 (dd, 1 H, $J_{4',5'}$ = 9.5 Hz, H-4'), 4.38 (ddd, 1 H, $J_{1a,2}$ = 4.5 Hz, $J_{1b,2}$ = 11.5 Hz, $J_{2,3}$ = 7.0 Hz, H-2), 4.23 (dd, 1 H, $J_{6'a,6'b}$ = 12.5 Hz, $J_{5',6'a}$ = 5.5 Hz, H-6'a), 4.10 (dd, 1 H, $J_{5',6'b}$ = 2.5 Hz, H-6'b), 3.65 (ddd, 1 H, H-5'), 3.60 (ddd, 1 H, $J_{1a,1'}$ = 9.5 Hz, $J_{1b,1'}$ = 2.5 Hz, H-1'), 2.21 (ddd, 1 H, $J_{1a,1b}$ = 14.5 Hz, H-1a), 2.10, 2.07, 2.03, 2.01 (4 s, 12 H, 4 Ac), 2.05 (ddd, 1 H, H-1b), 1.91 (d, 3 H, H-3); ^{13}C NMR (90 MHz, $CDCl_3$): δ 20.4, 20.5 ($COCH_3$), 25.1 (C2), 29.2 (C3), 43.7 (C1), 62.1 (C6'), 68.6 (C4'), 71.5 (C2'), 75.9 (C3'), 77.2 (C1'), 78.1 (C5'), 169.4, 169.8, 170.2, 170.5 ($COCH_3$); HRMS (ESI) Calcd for $C_{17}H_{25}O_9INa$: 523.0435. Found 523.0449.

1-*C*-(2',3',4',6'-Tetra-*O*-acetyl-β-D-mannopyranosyl)-2(*S*)- and 2(*R*)-iodopropane (14a, 14b)

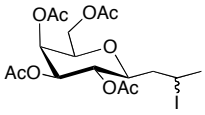


From **11a,b**. Flash chromatography (7:3 Petroleum ether / EtOAc) afforded **14a,b** as a 55:45 mixture. (4,8 g, 100%) The two isomers were separated by HPLC (Column dimension: 250 x 20 mm; Material: SiO_2, 5μm; Mobile phase: 47:53 Ether – Cyclohexane; Flow rate: 20 mL / min). Eluted first was **14a**: Mp. 78-79 °C (cyclohexane); $[\alpha]_D^{28}$ = - 46.2 (*c* = 1, $CHCl_3$); ^1H NMR (360 MHz, $CDCl_3$): δ 5.34 (dd, 1 H, $J_{1',2'}$ = 0.5 Hz, $J_{2',3'}$ = 3.5 Hz, H-2'), 5.22 (dd, 1 H, $J_{3',4'} = J_{4',5'}$ = 10.0 Hz, H-4'), 5.11 (dd, 1 H, H-3'), 4.34 (ddd, 1 H, $J_{2,3}$ = 7.0 Hz, H-2), 4.30 (dd,

1 H, $J_{6'a,6'b}$ = 12.5 Hz, $J_{5',6'a}$ = 5.5 Hz, H-6'a), 4.11 (dd, 1 H, $J_{5',6'b}$ = 2.5 Hz, H-6'b), 3.86 (ddd, 1 H, H-1'), 3.68 (ddd, 1 H, H-5'), 2.18, 2.11, 2.05, 1.99 (4 s, 12 H, 4 Ac), 1.96 (d, 3 H, H-3), 1.74-1.65 (2 ddd, 2 H, H-1a, H-1b); ^{13}C NMR (90 MHz, $CDCl_3$): δ 20.5, 20.6, 20.7 (CO*C*H$_3$), 25.9 (C2), 29.2 (C3), 42.7 (C1), 62.5 (C6'), 66.1 (C4'), 69.9 (C2'), 72.1 (C3'), 76.4 (C1'), 77.3 (C5'), 169.5, 169.7, 170.1, 170.3 (*C*OCH$_3$); HRMS (ESI) Calcd for $C_{17}H_{25}O_9INa$: 523.0435. Found 523.0445; Anal. Calcd for $C_{17}H_{25}O_9I$ (%): C, 40.81; H, 5.04; O, 28.78. Found: C, 40.89; H, 5.11; O, 28.95.

Eluted second was **14b**: Mp. 100-101 °C (cyclohexane); $[\alpha]_D^{28}$ = + 0.2 (*c* = 1, $CHCl_3$); ^{1}H NMR (360 MHz, $CDCl_3$): δ 5.33 (dd, 1 H, $J_{1',2'}$ = 0.5 Hz, $J_{2',3'}$ = 3.0 Hz, H-2'), 5.22 (dd, 1 H, $J_{3',4'} = J_{4',5'}$ = 10.0 Hz, H-4'), 5.11 (dd, 1 H, H-3'), 4.26 (dd, 1 H, $J_{6'a,6'b}$ = 12.5 Hz, $J_{5',6'a}$ = 5.5 Hz, H-6'a), 4.18 (ddd, 1 H, $J_{2,3}$ = 7.0 Hz, H-2), 4.11 (dd, 1 H, $J_{5',6'b}$ = 2.5 Hz, H-6'b), 3.86 (ddd, 1 H, H-1'), 3.68 (ddd, 1 H, H-5'), 2.18, 2.11, 2.05, 1.99 (4 s, 12 H, 4 Ac), 1.96 (d, 3 H, H-3), 1.74-1.65 (2 ddd, 2 H, H-1a, H-1b); ^{13}C NMR (90 MHz, $CDCl_3$): δ 20.6, 20.7, 20.8 (CO*C*H$_3$), 22.1 (C2), 28.3 (C3), 42.8 (C1), 62.8 (C6'), 66.2 (C4'), 69.1 (C2'), 72.2 (C3'), 76.3 (C1'), 77.2 (C5'), 169.7, 170.1, 170.5, 170.6 (*C*OCH$_3$); HRMS (ESI) Calcd for $C_{17}H_{25}O_9INa$: 523.0435. Found 523.0447.

1-*C*-(2',3',4',6'-*O*-Acetyl-β-D-galactopyranosyl)-2(*S*)- and 2(*R*)-iodopropane (15a, 15b)



From **12a,b**. Flash chromatography (7:3 Petroleum ether / EtOAc) afforded **15a,b** as a 3:1 mixture. (4.8 g, 100%) The two isomers were separated by HPLC (Column dimension: 250 x 20 mm; Material: SiO_2, 5μm; Mobile phase: 45:55 Et_2O – Cyclohexane; Flow rate: 20 mL / min). Eluted first was **15a**: Mp. 147-148 °C (cyclohexane); $[\alpha]_D^{28}$ = - 19.6 (*c* = 1, $CHCl_3$); ^{1}H NMR (250 MHz, $CDCl_3$): δ 5.45 (dd, 1 H, $J_{3',4'}$ = 3.5 Hz, $J_{4',5'}$ = 0.5 Hz, H-4'), 5.16 (dd, 1 H, $J_{1',2'} = J_{2',3'}$ = 9.5 Hz, H-2'), 5.07 (dd, 1 H, H-3'), 4.35 (ddd, 1 H, $J_{1a,2}$ = 11.5 Hz, $J_{1b,2}$ = 2.5 Hz, $J_{2,3}$ = 7.0 Hz, H-2), 4.16 (dd, 1 H, $J_{6'a,6'b}$ = 11.5 Hz, $J_{5',6'a}$ = 7.0 Hz, H-6'a), 4.07 (dd, 1 H, $J_{5',6'b}$ = 6.0 Hz, H-6'b), 3.91 (ddd, 1 H, H-5'), 3.64 (ddd, 1 H, $J_{1a,1'}$ = 2.0 Hz, $J_{1b,1'}$ = 9.5 Hz, H-1'), 2.15, 2.09, 2.08, 1.99 (4 s, 12 H, 4 Ac), 1.97 (d, 3 H, H-3), 1.87 (ddd, 1 H, $J_{1a,1b}$ = 14.5 Hz, H-1a), 1.64 (ddd, 1 H, H-1b); ^{13}C NMR (90 MHz, $CDCl_3$): δ 20.5, 20.6 (CO*C*H$_3$), 25.0 (C2), 29.2 (C3), 43.7 (C1), 61.4 (C6'), 67.6 (C4'), 68.8 (C2'), 71.8 (C3'), 74.4 (C1'), 78.3 (C5'),

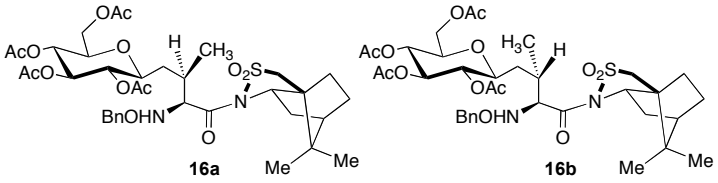
169.9, 170.0, 170.2 (*C*OCH$_3$); HRMS (ESI) Calcd for $C_{17}H_{25}O_9INa$: 523.0435. Found 523.0433; Anal. Calcd for $C_{17}H_{25}O_9I$ (%): C, 40.81; H, 5.04; O, 28.78. Found: C, 41.09; H, 5.08; O, 28.97.

Eluted second was **15b**: White crystals from cyclohexane (mp. 73-74 °C); $[\alpha]_D^{28}$ = + 10.8 (*c* = 1, $CHCl_3$); ^{1}H NMR (250 MHz, $CDCl_3$): δ 5.43 (dd, 1 H, $J_{3',4'}$ = 9.5, $J_{4',5'}$ = 0.5 Hz, H-4'), 5.11 (dd, 1 H, $J_{1',2'} = J_{2',3'}$ = 9.5 Hz, H-2'), 5.04 (dd, 1 H, H-3'), 4.40 (ddd, 1 H, $J_{1a,2}$ = 4.5 Hz, $J_{1b,2}$ = 11.5 Hz, $J_{2,3}$ = 7.0 Hz, H-2), 4.15 (dd, 1 H, $J_{6'a,6'b}$ = 11.5 Hz, $J_{5',6'a}$ = 7.0 Hz, H-6'a), 4.06 (dd, 1 H, $J_{5',6'b}$ = 6.5 Hz, H-6'b), 3.87 (ddd, 1 H, H-5'), 3.57 (ddd, 1 H, $J_{1a,1'}$ = 9.5 Hz, $J_{1b,1'}$ = 2.5 Hz, H-1'), 2.28 (ddd, 1 H, $J_{1a,1b}$ = 14.5 Hz, H-1a), 2.17, 2.09, 2.06, 2.00 (4 s, 12 H, 4 Ac), 2.12 (ddd, 1 H, H-1b), 1.91 (d, 3 H, H-3); ^{13}C NMR (90 MHz, $CDCl_3$): δ 20.4, 20.5 (CO*C*H$_3$), 22.0 (C2), 27.2 (C3), 44.2 (C1), 61.5 (C6'), 67.5 (C4'), 68.9 (C2'), 71.9 (C3'), 74.3 (C1'), 78.2 (C5'), 169.9, 170.0, 170.2 (*C*OCH$_3$); HRMS (ESI) Calcd for $C_{17}H_{25}O_9INa$: 523.0435. Found 523.0441.

Experimental procedure and characterisation of products 16a,b; 17a,b; 18a,b

To a degassed solution of camphorsultam derivative **(-)-1** or **(+)-1** (75 mg, 0.20 mmol) in CH_2Cl_2 (2 mL) were added $Zn(OTf)_2$ (145 mg, 0.40 mmol), iodinated compound **13a,b** or **14a,b** or **15a,b** (300 mg, 0.60 mmol), Bu_3SnH (140 μL, 0.50 mmol) and Et_3B was added (2 mL of a 1 M solution in hexane, 2 mmol) by portion of 400 μL each 30 min. After the last addition, the reaction mixture was stirred 2 h at -78°C then diluted with saturated aqueous $NaHCO_3$. The aqueous layer was extracted with CH_2Cl_2. The organic layer was dried ($MgSO_4$) and concentrated. The residue was purified by flash chromatography (3:97 acetone-CH_2Cl_2) to give the product as a diastereoisomeric mixture (90 mg, 60 %).

***C*-glucosides 16a and 16b**

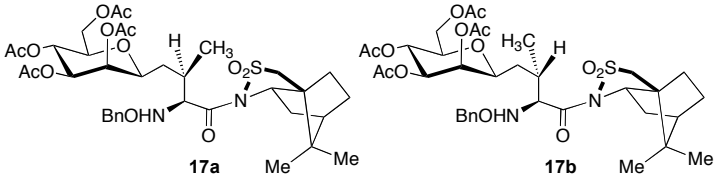


From camphorsultam derivative **(-)-1** and **13a,b**, compounds **16a** and **16b** were obtained as a 80:20 diastereoisomeric mixture. The major isomer **16a** was isolated by HPLC (Column

dimension: 250 x 21.2 mm; Material: Hypersil APS 2, 5µm; Mobile phase: 25:75 EtOAc – Cyclohexane; Flow rate: 20 mL / min) and crystallization from cyclohexane - EtOAc.

16a: Mp. 154-155 °C (*i*Pr_2O); $[\alpha]_D^{28}$ = - 28.2 (*c* = 1, $CHCl_3$); ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.39-7.29 (m, 5 H, HAr), 6.38 (d, 1 H, $J_{2,NH}$ = 12.5 Hz, N*H*), 5.17 (dd, 1 H, $J_{3',4'} = J_{2',3'}$ = 9.5 Hz, H-3'), 5.13 (dd, 1 H, $J_{4',5'}$ = 9.5 Hz, H-4'), 4.81 (dd, 1 H, $J_{1',2'}$ = 9.5 Hz, H-2'), 4.66 (d, 1 H, *J* = 12.0 Hz, NHOCH_2), 4.62 (d, 1 H, *J* = 12.0 Hz, NHOCH_2), 4.38 (dd, 1 H, $J_{5',6'a}$ = 3.0 Hz, $J_{6'a,6'b}$ = 12.5 Hz, H-6'a), 4.32 (dd, 1 H, $J_{2,3}$ = 3.0 Hz, H-2), 4.07 (dd, 1 H, $J_{5',6'b}$ = 2.0 Hz, H-6'b), 3.98 (dd, 1H, $J_{2'',3''a}$ = 5.0 Hz, $J_{2'',3''b}$ = 7.5 Hz, H-2''), 3.58 (ddd, 1 H, H-5'), 3.46 (s, 2 H, H-10''a, H-10''b), 3.38 (ddd, 1 H, $J_{1',1a}$ = 9.5 Hz, $J_{1',1b}$ = 1.0 Hz, H-1'), 2.29 (dddd, 1 H, H-3), 2.10 (m, 2 H, Hsult), 2.11, 2.08, 2.05, 2.02 (4 s, 12 H, 4 Ac), 1.95-1.80 (m, 4 H, 3 Hsult, H-4a), 1.60 (s, 1 H, Hsult), 1.42 (s, 1 H, Hsult), 1.32 (ddd, 1 H, H-4b), 1.10 (s, 3 H, H-8''), 0.97 (s, 3 H, H-9''), 0.73 (s, 3 H, $J_{CH3,3}$ = 7.0 Hz, CH_3); ^{13}C NMR (90 MHz, $CDCl_3$): δ 15.0 (CH_3), 19.9 (C8''), 20.6, 20.7, 20.8, 20.9 (CO*C*H_3, C9''), 26.5 (C5''), 30.0 (C3), 32.8 (C6''), 35.5 (C4), 38.5 (C3''), 44.6 (C4''), 47.8 (C7''), 48.6 (C1''), 53.0 (C10''), 61.8 (C6'), 64.3 (C2), 65.0 (C2''), 68.4 (C4'), 72.1 (C2'), 74.7 (C3'), 75.0 (C1'), 75.2 (C5'), 75.9 (*C*H_2OPh), 127.8, 128.4, 128.5, 138.0 (CAr), 169.5, 169.8, 170.4, 170.9 (*C*OCH_3), 173.3 (C1); HRMS (ESI) Calcd for $C_{36}H_{50}O_{13}N_2SNa$: 773.2926. Found 773.2933; Anal. Calcd for $C_{22}H_{33}O_{12}N$ (%): C, 57.59; H, 6.71; N, 3.73; O, 27.70; S, 4.27. Found: C, 57.52; H, 6.71; N, 3.68; O, 27.85; S, 4.04.

C-mannosides 17a and 17b

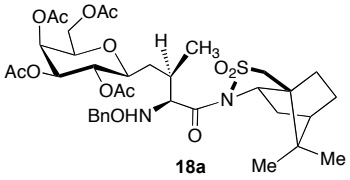


From camphorsultam derivative **(-)-1** and **14a,b**, compounds **17a** and **17b** were obtained as a 85:15 diastereoisomeric mixture. The major isomer **17a** was isolated by HPLC (Column dimension: 250 x 21.2 mm; Material: Hypersil APS 2, 5µm; Mobile phase: 25:75 EtOAc – Cyclohexane; Flow rate: 20 mL / min) and crystallization from *i*Pr_2O.

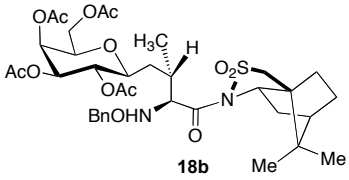
17a: Mp. 133-134 °C (*i*Pr_2O); $[\alpha]_D^{28}$ = - 48.4 (*c* = 1, $CHCl_3$); ^{1}H NMR (250 MHz, $CDCl_3$) δ (ppm): δ 7.49-7.34 (m, 5 H, HAr), 6.37 (d, 1 H, $J_{2,NH}$ = 12.0 Hz, N*H*), 5.23 (dd, 1 H, $J_{3',4'}$ =

$J_{4',5'}$ =10.0 Hz, H-4'), 4.98 (dd, 1 H, $J_{2',3'}$ = 3.5 Hz, H-3'), 4.94 (dd, 1 H, $J_{1',2'}$ = 1.0 Hz, H-2'), 4.69 (d, 1 H, J = 12.5 Hz, NHOCH_2), 4.54 (d, 1 H, J = 12.5 Hz, NHOCH_2), 4.42 (dd, 1 H, $J_{5',6'a}$ = 3.5 Hz, $J_{6'a,6'b}$ = 12.5 Hz, H-6'a), 4.07 (dd, 1 H, $J_{5',6'b}$ = 3.0 Hz, H-6'b), 4.20-3.95 (m, 2 H, H-2'', H-2), 3.52-3.38 (m, 3 H, H-5', H-10''), 3.10 (ddd, 1 H, H-1'), 2.20-1.95 (m, 2 H, H-3, Hsult), 2.14, 2.08, 2.03, 2.00 (4 s, 12 H, Ac) 1.95-1.80 (m, 4 H, Hsult), 1.45-1.31 (m, 2 H, H-4a, H-4b), 1.06 (s, 3 H, H-8''), 0.96 (s, 3 H, H-9''), 0.68 (d, 3 H, J = 7.0 Hz, CH_3); ^{13}C NMR (90 MHz, $CDCl_3$): δ 14.7 (CH_3), 19.9 (C8''), 20.6 (C9''), 20.7, 20.8, 20.8, 21.0 (CO*C*H$_3$), 26.5 (C5''), 30.1 (C3), 32.7 (C6''), 34.6 (C4), 38.4 (C3''), 44.5 (C4''), 47.8 (C7''), 48.7 (C1''), 53.0 (C10''), 62.4 (C6'), 64.9 (C2), 65.1 (C2''), 66.4 (C4'), 70.6 (C2'), 72.4 (C3'), 73.8 (C1'), 75.6 (C5'), 76.1 (*C*H$_2$OPh), 128.0, 128.4, 129.0, 138.4 (CAr), 169.7, 170.1, 170.5, 170.9 (*C*OCH$_3$), 173.4 (C1); HRMS (ESI) Calcd for $C_{36}H_{50}O_{13}N_2SNa$: 773.2926. Found 773.2916; Anal. Calcd for $C_{22}H_{33}O_{12}N$ (%): C, 57.59; H, 6.71; N, 3.73; O, 27.70; S, 4.27. Found: C, 57.70; H, 6.37; N, 3.73; O, 27.85; S, 4.06.

C-galactosides 18a and 18b



18a



18b

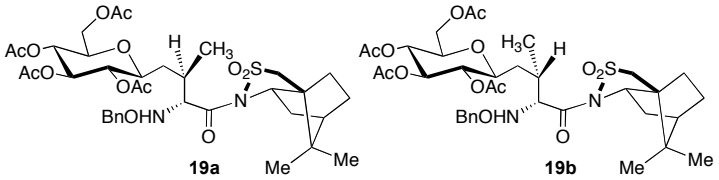
From camphorsultam derivative **(-)-1** and **15a,b**, compounds **18a** and **18b** were obtained as a 80:20 diastereoisomeric mixture. The major isomer **18a** was isolated by HPLC (Column dimension: 250 x 21.2 mm; Material: Hypersil APS 2, 5μm; Mobile phase: 25:75 EtOAc – Cyclohexane; Flow rate: 20 mL / min). Eluted first was **18a**: ^{1}H NMR (250 MHz, $CDCl_3$) δ: 7.41-7.28 (m, 5 H, HAr), 6.40 (d, 1 H, $J_{2,NH}$ = 12.0 Hz, N*H*), 5.41 (dd, 1 H, $J_{3',4'}$ = 3.0 Hz, $J_{4',5'}$ = 0.2 Hz, H-4'), 5.02 (dd, 1 H, $J_{2',3'}$ = 9.5 Hz, H-3'), 4.98 (dd, 1 H, $J_{1',2'}$ = 9.5 Hz, H-2'), 4.65 (s, 2 H, J = 12.5 Hz, NHOCH_2), 4.32 (dd, 1 H, H-2), 4.21 (dd, 1 H, $J_{5',6'a}$ = 5.5 Hz, $J_{6'a,6'b}$ = 10.5 Hz, H-6'a), 4.05 (dd, 1 H, $J_{5',6'b}$ = 9.0 Hz, H-6'b), 4.01 (dd, 1H, $J_{2'',3''a}$ = 5.0 Hz, $J_{2'',3''b}$ = 7.5 Hz, H-2''), 3.80 (ddd, 1 H, H-5'), 3.50 (dd, 2 H, $J_{10''a,1''} = J_{10''b,1''}$ = 14.0 Hz, H-10''a, H-10''b), 3.36 (ddd, 1 H, $J_{1',4a}$ = 1.0 Hz, $J_{1',4b}$ = 9.5 Hz, H-1'), 2.27 (dddd, 1 H, H-3), 2.13, 2.05, 2.04, 1.98 (4 s, 12 H, 4 Ac), 1.95-1.70 (m, 6 H, Hsult), 1.45-1.30 (m, 3 H, H-4a, H-4b, Hsult), 1.09 (s, 3 H, H-8''), 0.97 (s, 3 H, H-9''), 0.72 (d, 3 H, $J_{CH3,3}$ = 7.0 Hz, CH_3); ^{13}C NMR (90 MHz, $CDCl_3$): δ 14.9 (CH_3), 19.8 (C8''), 20.6, 20.7, 20.8, (C9'', CO*C*H$_3$), 26.4 (C5''),

30.0 (C3), 32.6 (C6''), 35.7 (C4), 38.3 (C3''), 44.5 (C4''), 47.8 (C7''), 48.6 (C1''), 52.9 (C10''), 60.2 (C6'), 64.3 (C2), 64.8 (C2''), 67.5 (C4'), 68.2 (C2'), 69.6 (C3'), 72.1 (C1'), 75.2 (C5'), 75.8 (*C*H_2OPh), 127.7, 128.3, 128.4, 138.0 (CAr), 169.9, 170.1, 170.2, 170.3 (*C*OCH_3), 173.3 (C1); HRMS (ESI) Calcd for $C_{36}H_{50}O_{13}N_2SNa$: 773.2926. Found 773.2908; Anal. Calcd for $C_{22}H_{33}O_{12}N$ (%): C, 57.59; H, 6.71; N, 3.73; O, 27.70; S, 4.27. Found: 57.42, 6.55, 3.71, 27.58, 4.72.

Eluted second was **18b** contaminated by **18a**.

18b: ^{1}H NMR (250 MHz, $CDCl_3$) δ 7.41-7.28 (m, 5 H, HAr), 6.18 (d, 1 H, $J_{2,NH}$ = 11.0 Hz, N*H*), 5.39 (dd, 1 H, $J_{3',4'}$ = 2.0 Hz, $J_{4',5'}$ = 0.2 Hz, H-4'), 5.08-4.95 (m, 2 H, H-2', H-3'), 4.71 (d, 1 H, *J* = 12.5 Hz, NHOCH_2), 4.62 (d, 1 H, *J* = 12.5 Hz, NHOCH_2), 4.20 (dd, 1 H, H-2), 4.10-3.92 (m, 3 H, H-6'a, H-6'b, H-2''), 3.80 (ddd, 1 H, $J_{5',6'a}$ = 1.5 Hz, $J_{5',6'b}$ = 7.0, $J_{6'a,6'b}$ = 9.0 Hz H-5'), 3.50 (s, 2 H, H-10''a, H-10''b), 3.46-3.32 (m, 1 H, H-1'), 2.20-1.62 (m, 7 H, H-3, Hsult), 2.15, 2.04, 2.03, 1.98 (4 s, 12 H, 4 Ac), 1.50-1.20 (m, 3 H, H-4a, H-4b, Hsult), 1.14 (s, 3 H, H-8''), 1.01 (d, 3 H, $J_{CH3,3}$ = 7.0 Hz, CH_3), 0.99 (s, 3 H, H-9'').

C-glucosides 19a and 19b



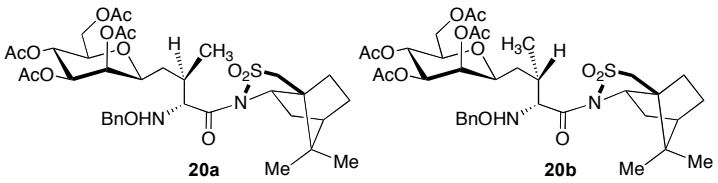
From camphorsultam derivative **(+)-1** and **13a,b**, compounds **19a** and **19b** were obtained as a 75:25 diastereoisomeric mixture. The major isomer **19a** was isolated by HPLC (Column dimension: 250 x 21.2 mm; Material: Hypersil APS 2, 5μm; Mobile phase: 25:75 EtOAc – Cyclohexane; Flow rate: 20 mL / min). Eluted first was **19a**: Mp. 133-134 °C (*i*Pr_2O); $[\alpha]_D^{28}$ = + 19 (*c* = 1, $CHCl_3$); ^{1}H NMR (400 MHz, $CDCl_3$) δ (ppm): δ 7.40-7.28 (m, 5 H, HAr), 5.96 (d, 1 H, $J_{2,NH}$ = 12.0 Hz, N*H*), 5.02 (dd, 1 H, $J_{3',4'} = J_{2',3'}$ = 9.5 Hz, H-3'), 4.90 (dd, 1 H, $J_{4',5'}$ = 9.5 Hz, H-4'), 4.70 (dd, 1 H, $J_{1',2'}$ = 9.5 Hz, H-2'), 4.68, 4.62 (2 d, 2 H, *J* = 12.0 Hz, NHOCH_2), 4.32 (dd, 1 H, $J_{2,3}$ = 8.0 Hz, H-2), 4.05 (dd, 1 H, $J_{5',6'a}$ = 5.5 Hz, $J_{6'a,6'b}$ = 12.5 Hz, H-6'a), 4.02 (dd, 1H, $J_{2'',3''a}$ = 2.0 Hz, $J_{2'',3''b}$ = 12.0 Hz, H-2''), 3.96 (dd, 1 H, $J_{5',6'b}$ = 2.0 Hz, H-6'b), 3.57 (ddd, 1 H, $J_{1',4a}$ = 9.5 Hz, $J_{1',4b}$ = 3.0 Hz, H-1'), 3.52 (2 d, 2 H, $J_{10''a,1''} = J_{10''b,1''}$ = 14.0 Hz, H-10''a, H-10''b), 2.97 (ddd, 1 H, H-5'), 2.12 (m, 2 H, Hsult), 2.07, 2.05, 2.01 (4 s, 12 H, 4 Ac), 1.95-1.85 (m, 3 H, Hsult), 1.80 (dddd, 1 H, H-3), 1.56 (s, 1 H, Hsult), 1.55-

1.46 (m, 2 H, H-4a, H-4b), 1.44 (s, 1 H, Hsult), 1.14 (s, 3 H, H-8''), 0.99 (s, 3 H, H-9''), 0.98 (d, 3 H, CH_3); ^{13}C NMR (90 MHz, $CDCl_3$): δ 18.0 (CH_3), 20.0 (C8''), 20.6, 20.7, 20.8 (CO*C*H_3, C9''), 26.4 (C5''), 31.8 (C3), 33.0 (C6''), 36.7 (C4), 38.4 (C3''), 44.6 (C4''), 47.8 (C7''), 48.3 (C1''), 53.3 (C10''), 62.3 (C6'), 65.4 (C2), 67.2 (C2''), 68.6 (C4'), 72.6 (C2'), 74.4 (C3'), 74.9 (C1'), 75.0 (C5'), 75.8 (*C*H_2OPh), 127.7, 128.4, 128.5, 138.9 (Ar), 169.5, 169.9, 170.4, 170.7 (*C*OCH_3), 174.3 (C1); HRMS (ESI) Calcd for $C_{36}H_{50}O_{13}N_2SNa$: 773.2926. Found 773.2923; Anal. Calcd for $C_{22}H_{33}O_{12}N$ (%): C, 57.59; H, 6.71; N, 3.73; O, 27.70; S, 4.27. Found: C, 57.01; H, 6.54; N, 3.71; O, 27.15; S, 4.25.

Eluted second was **19b** contaminated by **19a.**

19b ^1H NMR (250 MHz, $CDCl_3$) δ (ppm): δ 7.40-7.24 (m, 5 H, HAr), 6.24 (d, 1 H, $J_{2,NH}$ = 11.0 Hz, N*H*), 5.21-5.05 (m, 2 H, H-3', H-4'), 4.70 (dd, 1 H, $J_{1',2'}$ = 9.5 Hz, H-2'), 4.85-4.78 (m, 1H, H-2), 4.68, (d, 1 H, *J* = 12.0 Hz, NHOCH_2), 4.62 (d, 1 H, *J* = 12.0 Hz, NHOCH_2), 4.38 (dd, 1 H, $J_{5',6'b}$ = 3.0 Hz, $J_{6'a,6'b}$ = 12.5 Hz, H-6'a), 4.21 (dd, 1 H, $J_{5',6'a}$ = 1.5 Hz, Hz, H-6'a), 4.02-3.95 (m, 1H, H-2''), 3.78-3.65 (m, 2 H, H-1', H-5'), 3.52 (s, 2 H, H-10''a, H-10''b), 2.20-1.82 (m, 6 H, Hsult, H-3), 2.07, 2.02, 1.84, 1.60 (4 s, 12 H, 4 Ac), 1.50-1.30 (m, 3 H, H-4a, H-4b, Hsult), 1.15 (s, 3 H, H-8''), 1.02 (s, 3 H, H-9''), 0.98 (d, *J* = 7.0 Hz, 3 H, CH_3).

C-mannosides 20a and 20b

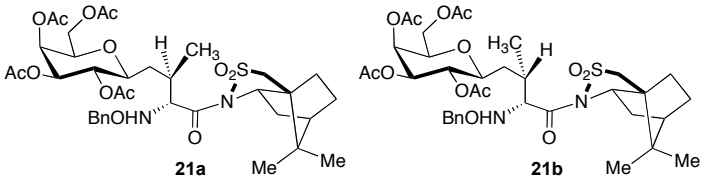


From camphorsultam derivative **(+)-1** and **14a,b**, compounds **20a** and **20b** were obtained as a 75:25 diastereoisomeric mixture. The major isomer **20a** was isolated by HPLC (Column dimension: 250 x 21.2 mm; Material: Hypersil APS 2, 5μm; Mobile phase: 25:75 EtOAc – Cyclohexane; Flow rate: 20 mL / min).

20a: $[\alpha]_D^{28}$ = + 43 (*c* = 1, $CHCl_3$); ^1H NMR (360 MHz, $CDCl_3$) δ (ppm): δ 7.41-7.30 (m, 5 H, HAr), 5.98 (d, 1 H, $J_{2,NH}$ = 12.0 Hz, N*H*), 5.18 (dd, 1 H, $J_{1',2'}$ = 1.0 Hz, $J_{2',3'}$ = 3.5 Hz, H-2'), 5.11 (dd, 1 H, $J_{3',4'} = J_{4',5'}$ =10.0 Hz, H-4'), 4.90 (dd, 1 H, H-3'), 4.70, 4.65 (2 d, 2 H, *J* = 12.5 Hz, NHOCH_2), 4.31 (dd, 1 H, $J_{2,3}$ = 9.5 Hz, H-2), 4.14 (dd, 1 H, $J_{5',6'a}$ = 5.5 Hz, $J_{6'a,6'b}$ = 12.5

Hz, H-6'a), 4.07 (dd, 1 H, $J_{5',6'b}$ = 2.5 Hz, H-6'b), 4.04 (dd, 1H, H-2''), 3.76 (ddd, 1 H, $J_{1',4a}$ = 9.5 Hz, $J_{1',4b}$ = 3.0 Hz, H-1'), 3.54 (2 d, 2 H, $J_{10''a,1''} = J_{10''b,1''}$ = 14.0 Hz, H-10''a, H-10''b), 3.08 (ddd, 1 H, H-5'), 2.13, 2.09, 2.07, 2.00 (4 s, 12 H, 4 Ac), 1.96-1.80 (m, 3 H, Hsult, H-3), 1.76-1.70 (m, 4 H, Hsult), 1.52-1.38 (m, 3 H, H-4a, H-4b, Hsult), 1.15 (s, 3 H, H-8''), 1.00 (s, 3 H, H-9''), 0.99 (d, 3 H, CH_3); ^{13}C NMR (90 MHz, $CDCl_3$): δ 17.7 (CH_3), 19.9 (C8''), 20.6, 20.7 (C9'', CO*C*H_3), 26.4 (C5''), 31.5 (C3), 32.9 (C6''), 35.8 (C4), 38.4 (C3''), 44.5 (C4''), 47.7 (C7''), 48.2 (C1''), 53.2 (C10''), 62.7 (C6'), 65.3 (C2), 66.2 (C2''), 68.3 (C4'), 70.2 (C2'), 72.4 (C3'), 74.7 (C1'), 74.9 (C5'), 75.5 (*C*H_2OPh), 127.5, 128.0, 128.3, 128.7, 138.9 (CAr), 169.7, 170.1, 170.6, 170.7 (*C*OCH_3), 174.2 (C1); HRMS (ESI) Calcd for $C_{36}H_{50}O_{13}N_2SNa$: 773.2926. Found 773.2928; Anal. Calcd for $C_{22}H_{33}O_{12}N$: C, 57.59; H, 6.71; N, 3.73; O, 27.70; S, 4.27. Found: C, 57.63; H, 6.31; N, 3.66; O, 27.55; S, 4.03.

C-galactosides 21a and 21b

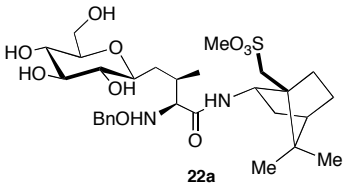


From camphorsultam derivative **(+)-1** and **15a,b**, compounds **21a** and 2**1b** were obtained as a 67:33 diastereoisomeric mixture. The major isomer **21a** was isolated by HPLC (Column dimension: 250 x 21.2 mm; Material: Hypersil APS 2, 5μm; Mobile phase: 25:75 EtOAc – Cyclohexane; Flow rate: 20 mL / min).

21a: ^{1}H NMR (360 MHz, $CDCl_3$) δ (ppm): δ 7.41-7.29 (m, 5 H, HAr), 5.95 (d, 1 H, $J_{2,NH}$ = 12.0 Hz, N*H*), 5.12 (dd, 1 H, $J_{3',4'}$ = 3.5 Hz, $J_{4',5'}$ = 0.5 Hz, H-4'), 4.89 (dd, 1 H, $J_{1',2'} = J_{2',3'}$ = 9.5 Hz, H-2'), 4.80 (dd, 1 H, H-3'), 4.70, 4.62 (2 d, 2 H, *J* = 12.5 Hz, NHOCH_2), 4.25 (dd, 1 H, H-2), 4.04 (dd, 1 H, $J_{2'',3''a}$ = 5.5 Hz, $J_{2'',3''b}$ = 7.5 Hz, H-2''), 3.92 (dd, 1 H, $J_{H5',H6'a}$ = 7.5 Hz, $J_{H6'a,H6'b}$ = 11.5 Hz, H-6'a), 3.85 (dd, 1 H, $J_{5',6'b}$ = 5.5 Hz, H-6'b), 3.54 (2 d, 2 H, $J_{10''a,1''} = J_{10''b,1''}$ = 14.0 Hz, H-10''a, H-10''b), 3.53 (ddd, 1 H, H-1'), 2.84 (ddd, 1 H, H-5') 2.12 (dddd, 1 H, H-3), 2.12, 2.10, 2.07, 1.97 (4 s, 12 H, 4 Ac), 2.00-1.80 (m, 4 H, Hsult), 1.60-1.30 (m, 5 H, H-4a, H-4b, Hsult), 1.14 (s, 3 H, H-8''), 0.98 (s, 3 H, H-9''), 0.97 (d, 3 H, CH_3); ^{13}C NMR (90 MHz, $CDCl_3$): δ 18.3 (CH_3), 20.0, 20.7, 20.8, 20.9 (C8'', C9'', CO*C*H_3), 26.4 (C5''), 31.5 (C3), 33.0 (C6''), 37.2 (C4), 38.4 (C3''), 44.6 (C4''), 47.8 (C7''), 48.3 (C1''), 53.3 (C10''), 62.1 (C2), 65.4 (C2''), 67.5 (C6'), 68.1 (C4'), 70.1 (C2'), 72.1 (C3'), 73.4

(C1'), 75.0 (C5'), 76.3 (*C*H_2OPh), 127.7, 128.4, 139.2 (CAr), 170.1, 170.2, 170.4, 170.5 (*C*OCH_3), 174.3 (C1); HRMS (ESI) Calcd for $C_{36}H_{50}O_{13}N_2SNa$: 773.2926. Found 773.2929; Anal. Calcd for $C_{22}H_{33}O_{12}N$: C, 57.59; H, 6.71; N, 3.73; O, 27.70; S, 4.27. Found: C, 57.37; H, 6.76; N, 3.57; O, 27.65; S, 4.33.

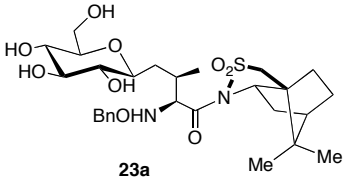
Compound 22a



22a

To a solution of **15a** (100mg, 0.13 mmol) in dry MeOH (10 mL) was added a solution of MeONa in MeOH (1mL, 1 M). The reaction mixture was stirred for 1 hour, then treated with Dowex 50 X-8 200 H+ to reach pH 6. The resin was filtered and the solution was concentrated to give **22a** (79 mg, 97 %), as a white solid. (mp. 109-110 °C); ^{1}H NMR (360 MHz, CD_3OD) δ (ppm): δ 7.40-7.28 (m, 5 H, HAr), 7.07 (d, 1 H, N*H*), 4.69 (2 d, 2 H, NHOCH_2), 4.26 (d, 1 H, N*H*), 3.90-3.00 (m, 10 H, H-1', H-2', H-3', H-4', H-5', H-6', H-2'', H-10''), 2.30-1.50 (m, 10 H, H-3, H-4a, H-4b, Hsult), 1.28 (s, 3 H, H-8''), 0.94 (m, 6 H, H-9'', CH_3); ^{13}C NMR (90 MHz, $CDCl_3$): δ 16.3 (CH_3), 20.2 (C8''), 20.6 (C9''), 26.9 (C5''), 30.7 (C3), 31.8 (C6''), 35.2 (C2), 39.5 (C3''), 44.6 (C4''), 48.9 (C7''), 49.2 (C1''), 54.5 (C10''), 61.8 (C6'), 67.6 (C2), 70.1, 73.6, 76.2, 78.5, 79.6 (C2'', C1', C2', C3', C4', C5', *C*H_2OPh), 128.2, 128.6, 129.6, 136.7 (CAr), 172.9 (C1); HRMS (ESI) Calcd for $C_{29}H_{46}O_{10}N_2SNa$: 637.2765. Found 637.2776.

Compound 23a

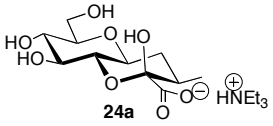


23a

To a solution of **15a** (170 mg, 0.23 mmol) in MeOH (5 mL) was added concentrated HCl (250 μL). The reaction mixture was refluxed for 5 h, then cooled to r.t. and treated with Dowex 1X8 (HCO_3^- form) to reach pH 7. The resin was filtered and the solution was

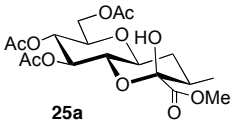
concentrated. Flash chromatography of the residue afforded **23a** (130 mg, 98 %) as a white solid (mp 88-89 °C); $[\alpha]_D^{28}$ = - 45 (*c* = 1, $CHCl_3$); ^{1}H NMR (360 MHz, CD_3OD) δ (ppm): δ 7.40-7.24 (m, 5 H, HAr), 4.69, 4.61 (2 d, 2 H, J = 12.0 Hz, NHOCH_2), 4.45 (d, 1 H, N*H*), 3.98 (dd, 1H, $J_{2'',3''a}$ = 5.0 Hz, $J_{2'',3''b}$ = 6.5 Hz, H-2''), 3.84 (dd, 1 H, $J_{5',6'a}$ = 2.5 Hz, $J_{6'a,6'b}$ = 12.0 Hz, H-6'a), 3.70 (dd, 1 H, $J_{5',6'b}$ = 5.0 Hz, H-6'b), 3.70, 3.60 (2 d, 2 H, $J_{10''a,1''}$ = $J_{10''b,1''}$ = 14.0 Hz, H-10''a, H-10''b), 3.37 (dd, 1 H, $J_{2,3}$ = 2.5 Hz, H-2) 3.29 (ddd, 1 H, H-5'), 2.42-2.30 (m, 2 H, H-3, Hsult), 2.10-1.83 (m, 5 H, Hsult), 1.49 (ddd, 1 H, H-4a), 1.40-1.25 (m, 2 H, H-4b, Hsult), 1.15 (s, 3 H, H-8''), 1.02 (s, 3 H, H-9''), 0.80 (s, 3 H, $J_{CH3,3}$ = 6.5 Hz, CH_3); ^{13}C NMR (90 MHz, CD_3OD): δ 15.5 (CH_3), 19.9 (C8''), 20.8 (C9''), 26.4 (C5''), 30.6 (C3), 32.7 (C6''), 35.5 (C2), 38.5 (C3''), 44.6 (C4''), 47.8 (C7''), 48.6 (C1''), 53.0 (C10''), 62.1 (C6'), 62.3 (C2), 65.1 (C2''), 70.3, 74.2, 75.2, 75.6, 75.9, 79.0 (C1', C2', C3', C4', C5', *C*H_2OPh), 127.8, 128.2, 128.3, 128.7, 138.0 (CAr), 173.5 (C1); HRMS (ESI) Calcd for $C_{28}H_{42}O_9N_2SNa$: 605.2503. Found 605.2503; Anal. Calcd for $C_{22}H_{33}O_{12}N$ (%): C, 57.71; H, 7.26; N, 4.81; S, 5.50. Found: C, 57.71; H, 7.26; N, 4.81; S, 5.50.

Compound 24a



To a warmed (60 °C) solution of **23a** (50 mg, 0.09 mmol) in phosphate buffer (0.1 M, 1 mL, pH 7) was added 40% aqueous formaldehyde (150 μL). The reaction mixture was stirred overnight at 60 °C, then cooled to room temperature, extracted with CH_2Cl_2 and concentrated. The residue was eluted on a Sephadex-DEAE column with 0.2 M triethylammonium bicarbonate eluent. The collected fractions were lyophilized three times to afford **24a** as a white solid (26 mg, 75%). ^{1}H NMR (360 MHz, CD_3OD) δ (ppm): δ 3.87 (dd, 1 H, $J_{5',6'a}$ = 1.5 Hz, $J_{6'a,6'b}$ = 12.0 Hz, H-6'a), 3.65 (ddd, 1 H, $J_{5',6'b}$ = 4.0 Hz, $J_{4',5'}$ = 11.5 Hz, H-5'), 3.55-3.29 (m, 5 H, H-1', H-2', H-3', H-4', H-6'b), 3.22 (dd, 6 H, N(C$H_2$$CH_3$)$_3$), 2.28 (ddd, 1 H, $J_{3,4a}$ = 4.5 Hz, $J_{3,4b}$ = 12.0 Hz, $J_{3,CH3}$ = 6.5 Hz, H-3), 1.88 (ddd, 1 H, $J_{4a,1'}$ = 4.5 Hz, $J_{4a,4b}$ = 12.0 Hz, H-4a), 1.55 (ddd, 1 H, $J_{4b,1'}$ = 14.5 Hz, H-4b), 1.33 (dd, 9 H, N(CH_2CH_3)$_3$, 0.87 (d, 3 H, CH_3); ^{13}C NMR (90 MHz, $CDCl_3$): δ 7.8 (N(CH_2*C*H_3)$_3$), 15.3 (CH_3), 32.9 (C4), 34.8 (C3), 46.2 (N(*C*$H_2$$CH_3$)$_3$), 61.8 (C-6'), 71.1 (C4'), 73.9 (C3'), 75.0 (C5'), 75.4 (C2'), 80.8 (C1'), 97.1 (C2), 175.4 (C1); HRMS (ESI) Calcd for $C_{11}H_{17}O_8^-$: 277.0918. Found 277.0865.

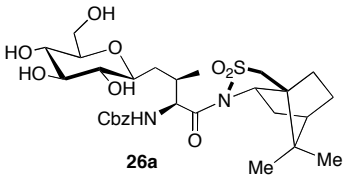
Compound 25a



25a

24a (26 mg, 0.07 mmol) was treated with CH_2N_2 in MeOH (500 μL). The solution was concentrated, then suspended in 2:1 pyridine-acetic anhydride (1.5 mL) at 0 °C. The reaction mixture was stirred overnight, then concentrated. The residue was suspended in CH_2Cl_2 and washed with water and brine. The organic layer was dried ($MgSO_4$) and concentrated. Flash chromatography (95:5 CH_2Cl_2-acetone) afforded **25a** (27 mg, 94 %). Mp 134-135 °C (*i*Pr_2O); $[\alpha]_D^{28}$ = 8 (*c* = 1, $CHCl_3$); ^{1}H NMR (360 MHz, CD_3OD) δ (ppm): δ 5.18 (dd, 1 H, $J_{2',3'}$ = $J_{3',4'}$ = 10.0 Hz, H-3'), 5.02 (dd, 1 H, $J_{4',5'}$ = 10.0 Hz, H-4'), 4.22 (dd, 1 H, $J_{5',6'a}$ = 5.0 Hz, $J_{6'a,6'b}$ = 12.5 Hz, H-6'a), 4.11 (dd, 1 H, $J_{5',6'b}$ = 2.5 Hz, H-6'b), 3.93 (d, 1 H, $J_{OH,3}$ = 1.5 Hz, O*H*), 3.85 (s, 3 H, CO_2CH_3), 3.84 (dd, 1 H, $J_{1',2'}$ = 10.0 Hz, H-2'), 3.70 (ddd, 1 H, H-5'), 3.37 (ddd, 1 H, $J_{1',4a}$ = 4.5 Hz, $J_{1',4b}$ = 14.5 Hz, H-1'), 2.28 (ddd, 1 H, $J_{3,4a}$ = 4.5 Hz, $J_{3,4b}$ = 12.5 Hz, $J_{3,CH3}$ = 6.5 Hz, H-3), 2.10, 2.04 (3 s, 9 H, 3 Ac), 1.98 (ddd, 1 H, $J_{4a,4b}$ = 12.5 Hz, H-4a), 1.72 (ddd, 1 H, H-4b), 0.88 (d, 3 H, CH_3); HRMS (ESI) Calcd for $C_{18}H_{26}O_{11}Na$: 441.1367. Found 441.1368.

Compound 26a

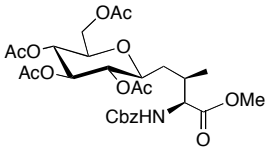


26a

To a solution of **23a** (60 mg, 0.1 mmol) in CH_3CN (1.5 mL) and H_2O (100 μL) was added $Mo(CO)_6$ (80 mg, 3 equiv) under argon at room temperature. The reaction mixture was refluxed for 6 h, then coevaporated with toluene. The residue was suspended in acetone, and treated at 0 °C with Na_2CO_3 (32 mg, 0.3 mmol) in H_2O (200 μL) then benzyl chloroformate (21 μL, 0.15 mmol). The reaction mixture was stirred for 2 h at room temperature, then concentrated. The residue was suspended in CH_2Cl_2 and washed with water and brine. The organic layer was dried ($MgSO_4$) and concentrated. Flash chromatography of the residue (85:15 CH_2Cl_2-MeOH) afforded **26a** (30 mg, 50 %) as a white solid. ^{1}H NMR (360 MHz,

CD_3OD) selected data δ: 7.40-7.30 (m, 5 H, HAr), 5.60 (d, 1 H, $J_{2,NH}$ = 9.0 Hz, N*H*), 4.70, 4.60 (2 d, 2 H, J = 12.0 Hz, NHOCH_2), 3.90-3.00 (m, 9 H, H-1', H-2', H-3', H-4', H-5', H-6'a, H-6'b, H-2'', H-10''), 2.20-1.80 (m, 5 H, Hsult), 1.40-1.20 (m, 3 H, H-4a, H-4b, Hsult), 1.11 (s, 3 H, H-8''), 0.96 (s, 3 H, H-9''), 0.78 (s, 3 H, $J_{CH3,3}$ = 6.5 Hz, CH_3); ^{13}C NMR (90 MHz, $CDCl_3$) selected data δ 14.9 (CH_3), 19.8 (C8''), 20.7 (C9''), 26.5 (C5''), 32.0 (C3), 32.7 (C6''), 35.4 (C2), 38.4 (C3''), 44.6 (C4''), 47.8 (C7''), 48.7 (C1''), 52.9 (C10''), 62.6 (C6'), 65.0, 65.1 (C2, C2''), 67.6, 70.8, 74.0, 79.2 (C1', C2', C3', C4', C5', *C*H_2OPh), 128.1, 128.3, 128.6, 135.9 (CAr), 155.9 (NH(*C*O)OCH_2), 171.7 (C1); HRMS (ESI) Calcd for $C_{29}H_{42}O_{10}N_2SNa$: 633.2452. Found 633.2461.

Methyl-4-*C*-(2',3',4',6'-*O*-acetyl-β-D-glucopyranosyl)-2-*S*-benzyloxycarbonylamino-3-*R*-methyl-butanate (27a)

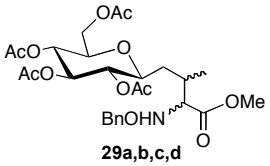


27a

To a solution of **26a** (70 mg, 0.11 mmol) in THF (1.1 mL) was added 37 % aqueous H_2O_2 (30 μL, 0.22 mmol) and 35 % aqueous tetrabutyl ammonium hydroxyde (80 μL, 0.22 mmol) at -10 °C. The reaction mixture was stirred for 4 h, then warmed to r.t. and treated with Dowex 50X8 (H^+ form) to reach pH 3. The resin was filtered and the solution was concentrated. The residue was eluted on a Sephadex-DEAE column with 0.2 M triethylammonium bicarbonate eluent. The collected fractions were lyophilized three times and the residue was treated with CH_2N_2 in MeOH (500 μL). The solution was concentrated, then suspended in 2:1 pyridine / acetic anhydride (1.5 mL) at 0 °C. The reaction mixture was stirred overnight, then concentrated. The residue was suspended in CH_2Cl_2 and washed with water and brine. The organic layer was dried ($MgSO_4$) and concentrated. Flash chromatography of the residue (95:5 CH_2Cl_2-acetone) afforded **27a** (27 mg, 40 % for 3 steps) as an oil; $[\alpha]_D^{28}$ = -3 (*c* = 1, $CHCl_3$); ^{1}H NMR (250 MHz, $CDCl_3$) δ (ppm): δ 7.43-7.32 (m, 5 H, HAr), 5.27 (d, 1 H, $J_{2,NH}$ = 9.5 Hz, N*H*), 5.24 (dd, 1 H, $J_{3',4'} = J_{2',3'}$ = 9.5 Hz, H-3'), 5.13 (2 d, 2 H, *J* = 14.0 Hz, NHOCH_2), 5.05 (dd, 1 H, $J_{4',5'}$ = 9.5 Hz, H-4'), 4.85 (dd, 1 H, $J_{1',2'}$ = 9.5 Hz, H-2'), 4.51 (dd, 1 H, $J_{2,3}$ = 3.5 Hz, H-2), 4.26 (dd, 1 H, $J_{5',6'a}$ = 5.5 Hz, $J_{6'a,6'b}$ = 12.5 Hz, H-6'a), 4.11 (dd, 1 H, $J_{5',6'b}$ = 2.5 Hz, H-6'b), 3.76 (s, 3 H, CO_2Me), 3.78-3.60 (2 ddd, 2 H, H-1', H-5'), 2.43

(dddd, 1 H, H-3), 2.08, 2.04, 2.01, 1.97 (4 s, 12 H, 4 Ac), 1.55 (ddd, 1 H, $J_{1',4a}$ = 2.5 Hz, $J_{4a,4b}$ = 14.5 Hz, $J_{4a,3}$ = 9.5 Hz, H-4a) , 1.39 (ddd, 1 H, $J_{1',4b}$ = 9.5 Hz, $J_{4b,3}$ = 4.5 Hz, H-4b), 0.85 (s, 3 H, $J_{CH3,3}$ = 7.0 Hz, CH_3); ^{13}C NMR (90 MHz, $CDCl_3$): δ 15.7 (CH_3), 20.7 ($CO\mathit{C}H_3$), 29.7 (C4), 34.9 (C4), 52.5 ($CO_2\mathit{Me}$), 55.8 (C2), 62.5 (C6'), 67.2 (NH(CO)O$\mathit{C}H_2$), 68.7 (C4'), 72.4 (C2'), 74.3 (C3'), 75.3 (C1'), 75.7 (C5'), 128.1, 128.3, 128.6, 136.1 (CAr), 156.5 (NH(CO)OCH_2), 169.6, 169.9, 170.3, 170.7 ($\mathit{C}OCH_3$), 172.5 ($\mathit{C}O_2$Me); HRMS (ESI) Calcd for $C_{28}H_{37}O_{13}NNa$: 618.2157. Found 618.2168; Anal. Calcd for $C_{28}H_{37}O_{13}N$ (%): C, 56.46; H, 6.26; N, 2.35; O, 34.92. Found: C, 56.22; H, 6.27; N, 2.28; O, 35.16.

Methyl-4-*C*-(2',3',4',6'-*O*-acetyl-β-D-glucopyranosyl)-2-(benzyloxyamino)-3-methyl-butanate 29a,b,c,d

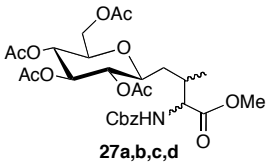


29a,b,c,d

To a degassed solution of **13a** (500 mg, 1 mmol) and methyl *E*-2-(benzyloxyimino)ethanate **28** (39 mg, 0.2 mmol) in CH_2Cl_2 (2 mL) were added Bu_3SnH (120 μL, 2.5 éq) and Et_3B (0.5 mL, 2.5 eq) under argon. The reaction mixture was stirred overnight, then diluted with saturated aqueous $NaHCO_3$. The aqueous layer was extracted with CH_2Cl_2. The organic layer was dried ($MgSO_4$) and concentrated. Flash chromatography of the residue (5:95 acetone-CH_2Cl_2) afforded the coupling product as an 1:1:1:1 mixture (82 mg, 74 %).

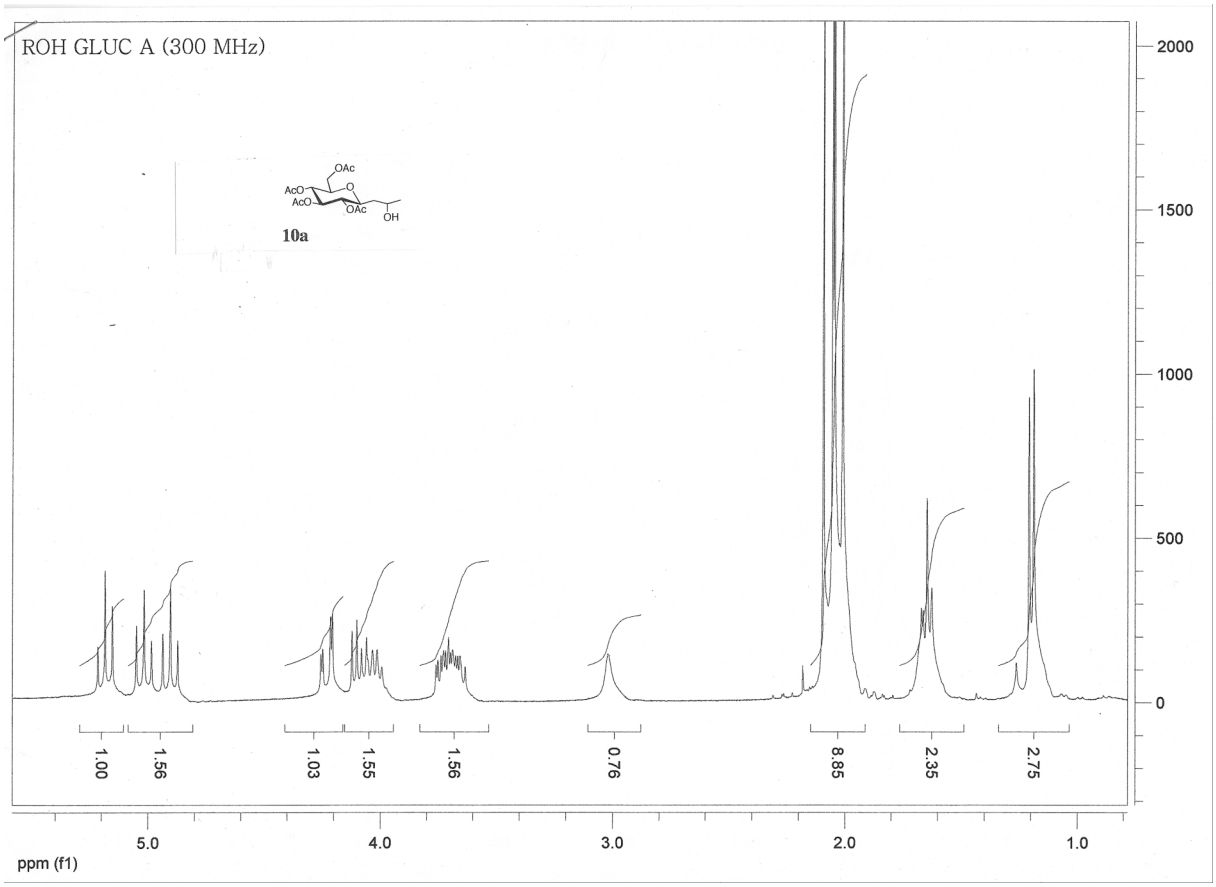
^{1}H NMR (250 MHz, $CDCl_3$) selected data: δ (ppm): 7.40-7.30 (m, 5 H, HAr); 6.12-5.95 (m, 1 H, N*H*), 5.20-4.58 (m, 4 H, H-2', H-3', H'4, H-2).4.69-4.60 (m, 2 H, OC$\mathit{H}_2C_6H_5$), 4.27-3.90 (2 dd, 2 H, H-6'a, H-6'b), 3.75 (s, 3 H, CO_2Me), 3.63-3.05 (2 ddd, 2 H, H-1', H-5'), 2.09-1.98 (4 s, 12 H, 4 Ac), 1.90-1.24 (m, 3 H, H-3, H-4a, H-4b), 0.90-0.77 (d, 3 H, CH_3); ^{13}C NMR (90 MHz, $CDCl_3$) selected data: δ 14.5, 16.0, 17.6 (CH_3), 20.5 (OAc), 29.8, 30.1, 30.2, 30.6 (C3), 34.4, 35.0, 35.1, 36.5 (C4), 51.5, 51.8, 53.3 (CO_2Me, C2), 62.2, 62.2, 65.5 (C6'), 67.6, 68.5, 68.6, 68.7 (C4'), 71.9, 72.1, 72.3, 72.5 (C2'), 74.0, 74.1, 74.2 (C3'), 75.0-76.0 (C1', C5'), 127.7-128.5, 137.7, 138.8 (CAr), 169.3-170.4 (OAc), 173.8 (CO_2Me); HRMS (ESI) Calcd for $C_{27}H_{37}O_{12}NNa$: 590.2208. Found 590.2218; Anal. Calcd for $C_{27}H_{37}O_{12}N$ (%): C, 57.14; H, 6.57; N, 2.47. Found: C, 57.08; H, 6.62; N, 2.41.

Methyl-4-*C*-(2',3',4',6'-tetra-*O*-acetyl-β-D-glucopyranosyl)-2-benzyloxycarbonylamino-3-methyl-butanate (27a,b,c,d)



27a,b,c,d

To a solution of **29a,b,c,d** (100 mg, 0.18 mmol) in CH_3CN (2.3 mL) and H_2O (170 μL) was added $Mo(CO)_6$ (142 mg, 3 equiv) under argon at room temperature. The reaction mixture was refluxed for 6 h, then coevaporated with toluene. The residue was suspended in acetone, and treated at 0°C with Na_2CO_3 (52 mg, 0.5 mmol) in H_2O (330 μL) then benzyl chloroformate (35 μL, 0.25 mmol). The reaction mixture was stirred for 2 h at room temperature, then concentrated. The residue was suspended in CH_2Cl_2 and washed with water and brine. The organic layer was dried ($MgSO_4$) and concentrated. Flash chromatography of the residue (5:95 acetone-CH_2Cl_2) afforded **27a,b,c,d** (55 mg, 40 %) as an oil. ^{1}H NMR (250 MHz, $CDCl_3$) selected data: δ = 7.40-7.30 (m, 5 H, HAr), 5.59, 5.48, 5.34, 5.28 (d, 1 H, $J_{2,NH}$ = 9.5 Hz, N*H*), 5.24-5.14 (dd, 1 H, H-3'), 5.12, 5.11 (2 d, 2 H, *J* = 14.0 Hz, NHOCH_2), 5.09-4.98 (dd, 1 H, $J_{4',5'}$ = 9.5 Hz, H-4'), 4.89-4.80 (dd, 1 H, H-2'), 4.51-4.06 (m, 3 H, H-2, H-6'a, H-6'b), 3.77-3.45 (m, 5 H, CO_2Me, H-1', H-5'), 2.45-2.20 (dddd, 1 H, H-3), 2.08, 2.06, 2.04, 2.03, 2.02, 2.01, 1.97 (4 s, 12 H, 4 Ac), 1.68-1.28 (2 ddd, 2 H, H-4a, H-4b), 0.97, 0.96, 0.89, 0.85 (d, 3 H, $J_{CH3,3}$ = 7.0 Hz, CH_3); ^{13}C NMR (90 MHz, $CDCl_3$) selected data: δ 14.8, 15.6, 17.0 (CH_3), 20.4-20.8 (CO*C*H_3), 29.6 (C4), 52.1, 52.2, 52.3, 52.4 (CO_2*Me*), 55.8, 57.6, 58.1, 58.4 (C2), 62.1, 62.3, 62.4 (C6'), 66.9, 67.0, 67.1 (NH(CO)O*C*H_2), 68.5, 68.6, 68.7, (C4'), 72.0, 72.1, 72.2, 72.3 (C2'), 74.3-74.4 (C3'), 75.3, 75.6, 75.7, 75.8, 75.9 (C1',C5'), 126.9, 127.6, 128.0-128.3, 128.6, 136.1-136.2 (CAr), 155.8, 156.0, 156.1, 156.2, 156.3, 156.5 (NH(*C*O)OCH_2), 169.4-170.7 (*C*OCH_3), 171.7, 172.1, 172.2, 172.4 (*C*O_2Me).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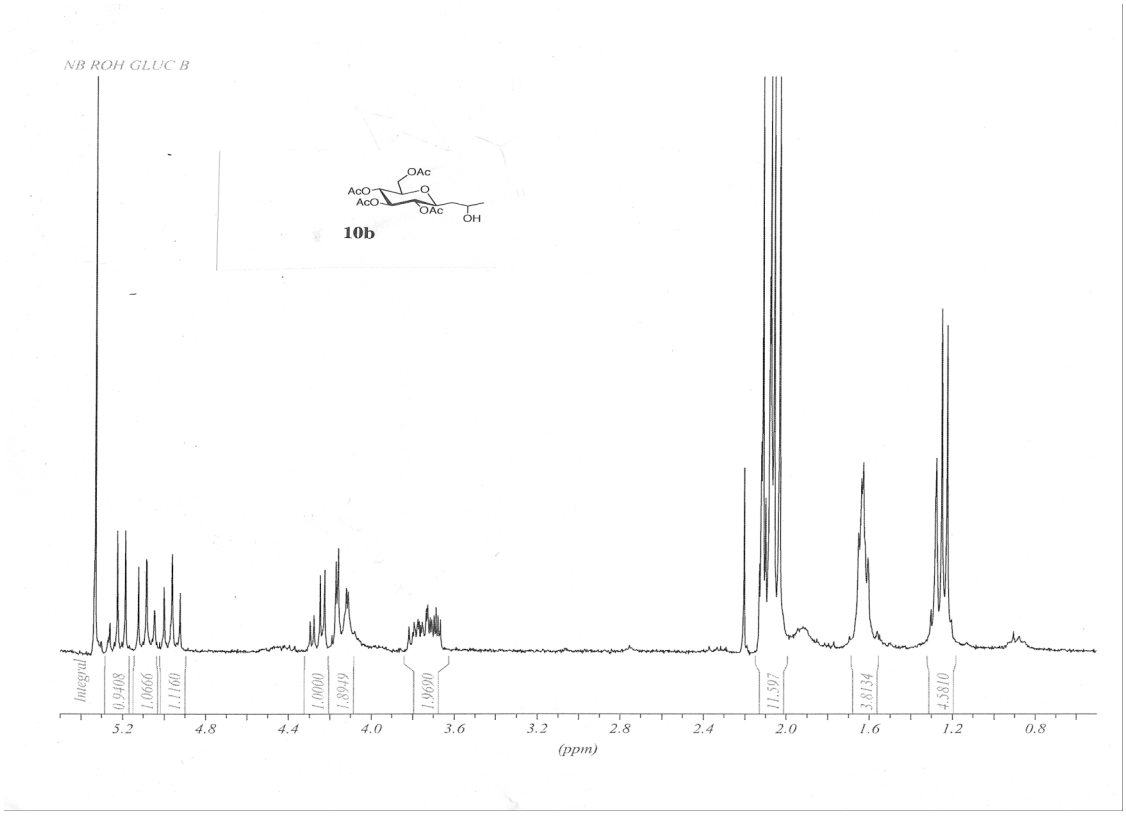
OAc
AcO
AcO
O
OAc
OH
10a
2000
1500
1000
500
0
1.00
1.56
1.03
1.55
1.56
0.76
8.85
2.35
2.75
5.0
4.0
3.0
2.0
1.0
ppm (f1)


NB ROH GLUC B
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AcO
AcO
O
OAc
OH
10b
Integral
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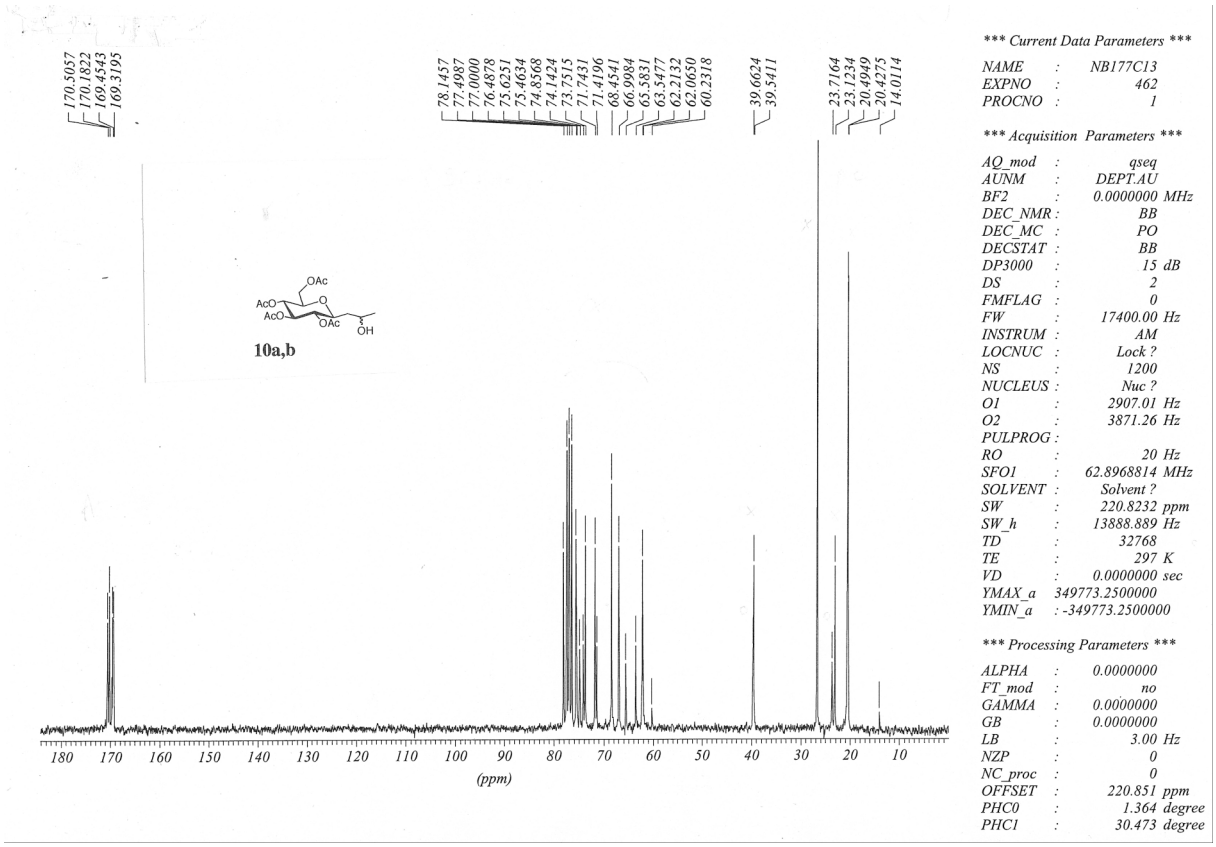
1.0666
1.1160
1.9000
1.8949
1.9690
11.597
3.8134
4.5810
5.2
4.8
4.4
4.0
3.6
3.2
2.8
2.4
2.0
1.6
1.2
0.8
(ppm)


170.5057
170.1822
169.4543
169.3195
78.1457
77.4987
77.0000
76.4878
73.6251
73.4634
74.8568
74.1424
73.7515
71.7431
71.4196
68.4541
66.9984
63.5831
63.5477
62.2132
62.0650
60.2318
39.6624
39.5411
23.7164
23.1234
20.4949
20.4275
14.0114
OAc
AcO
AcO
OAc
OH
10a,b
180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10
(ppm)
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EXPNO : 462
PROCNO : 1
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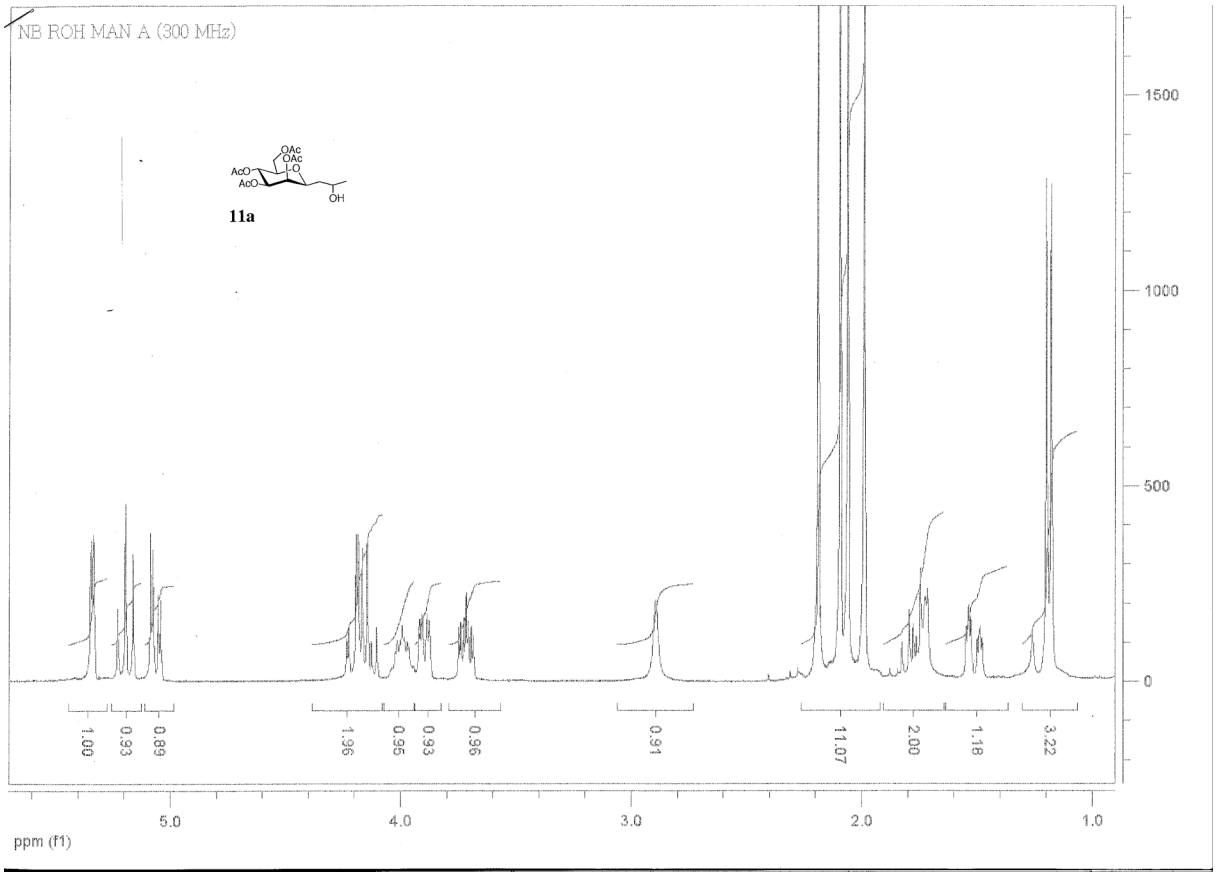
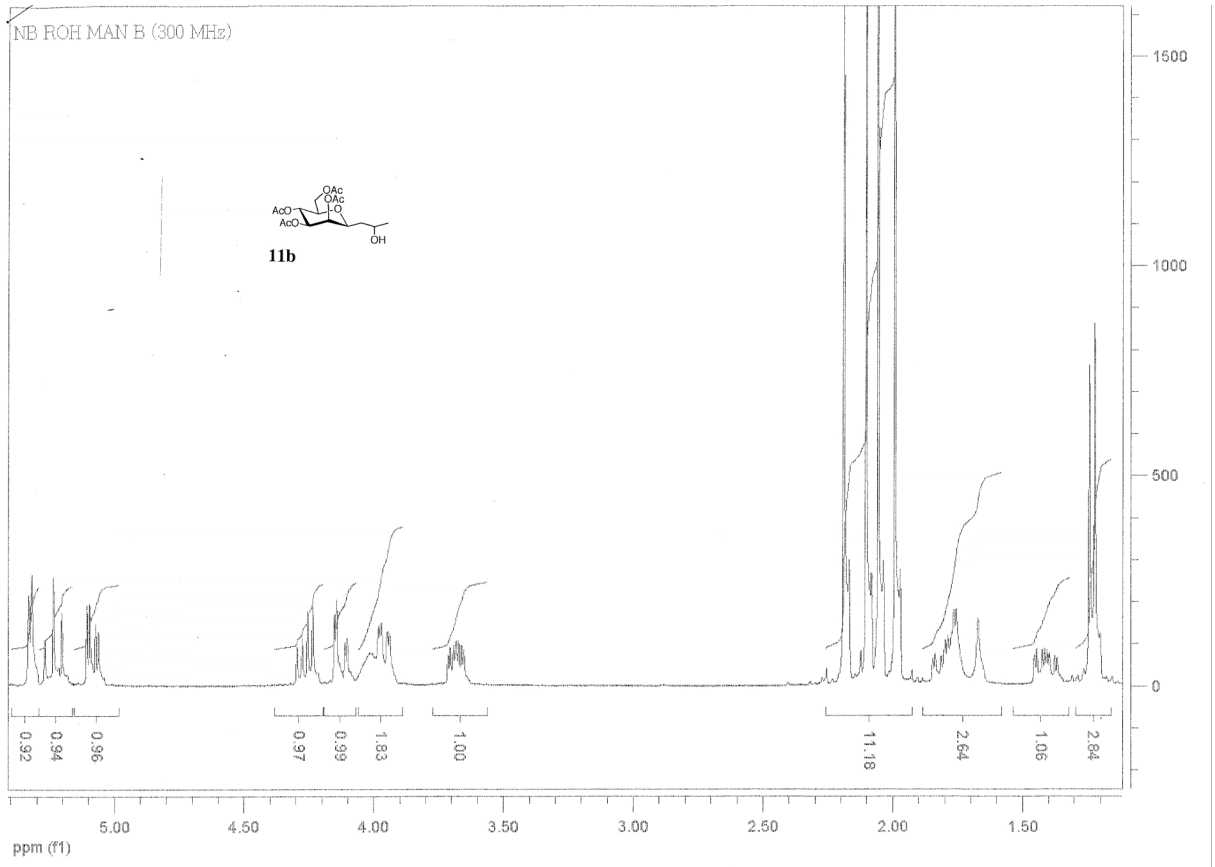
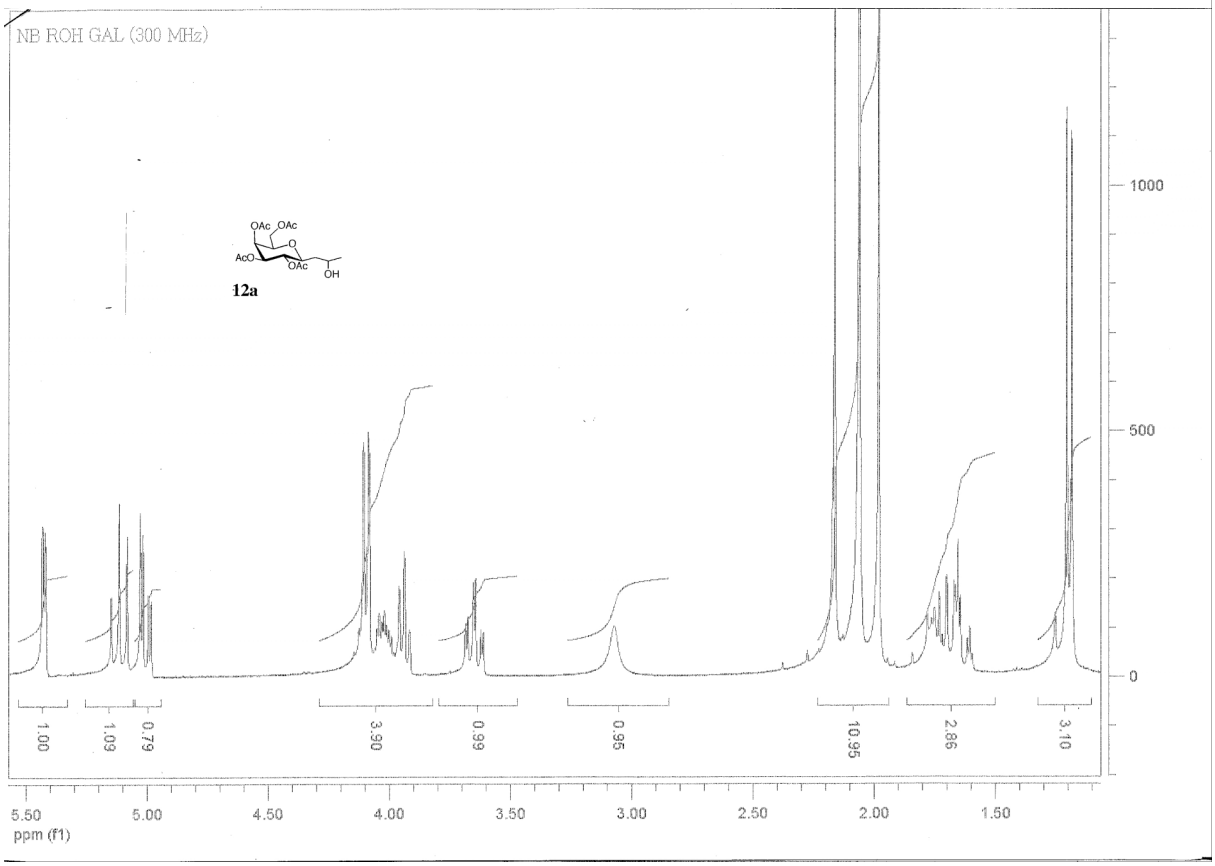
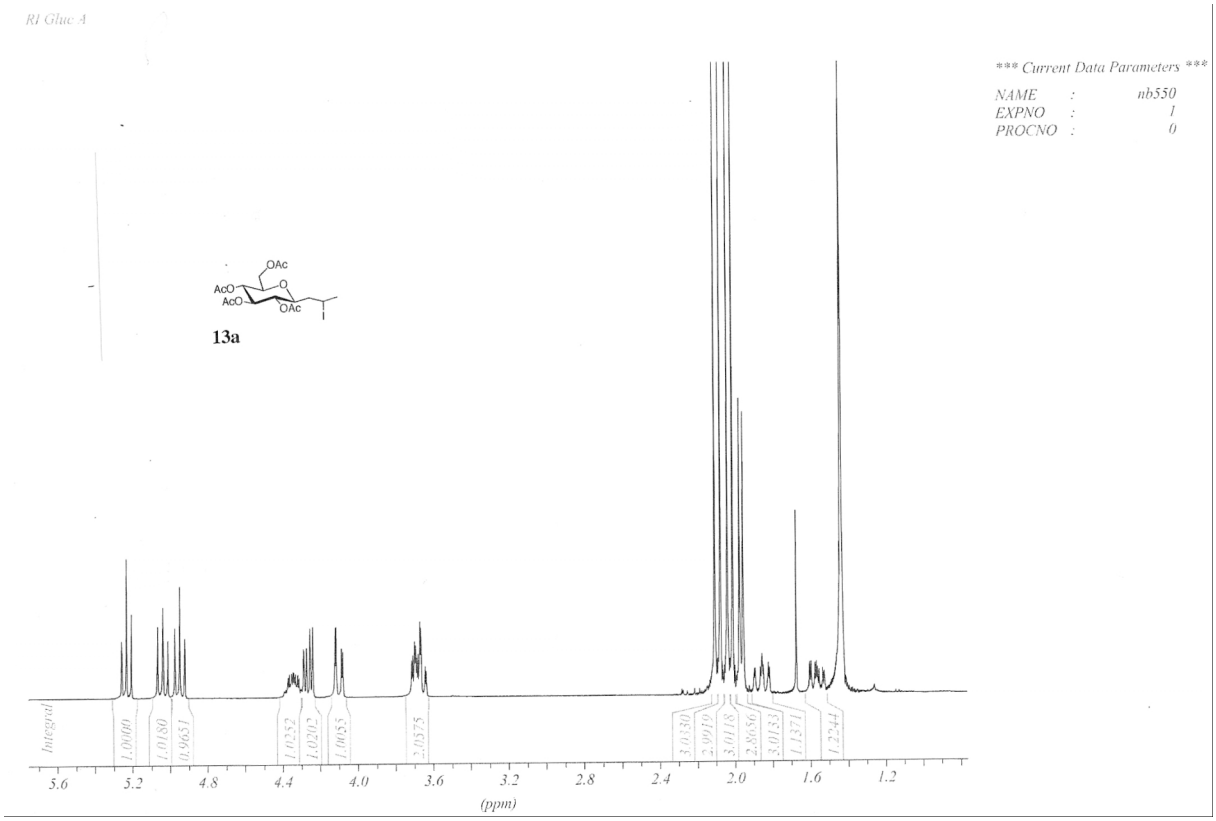
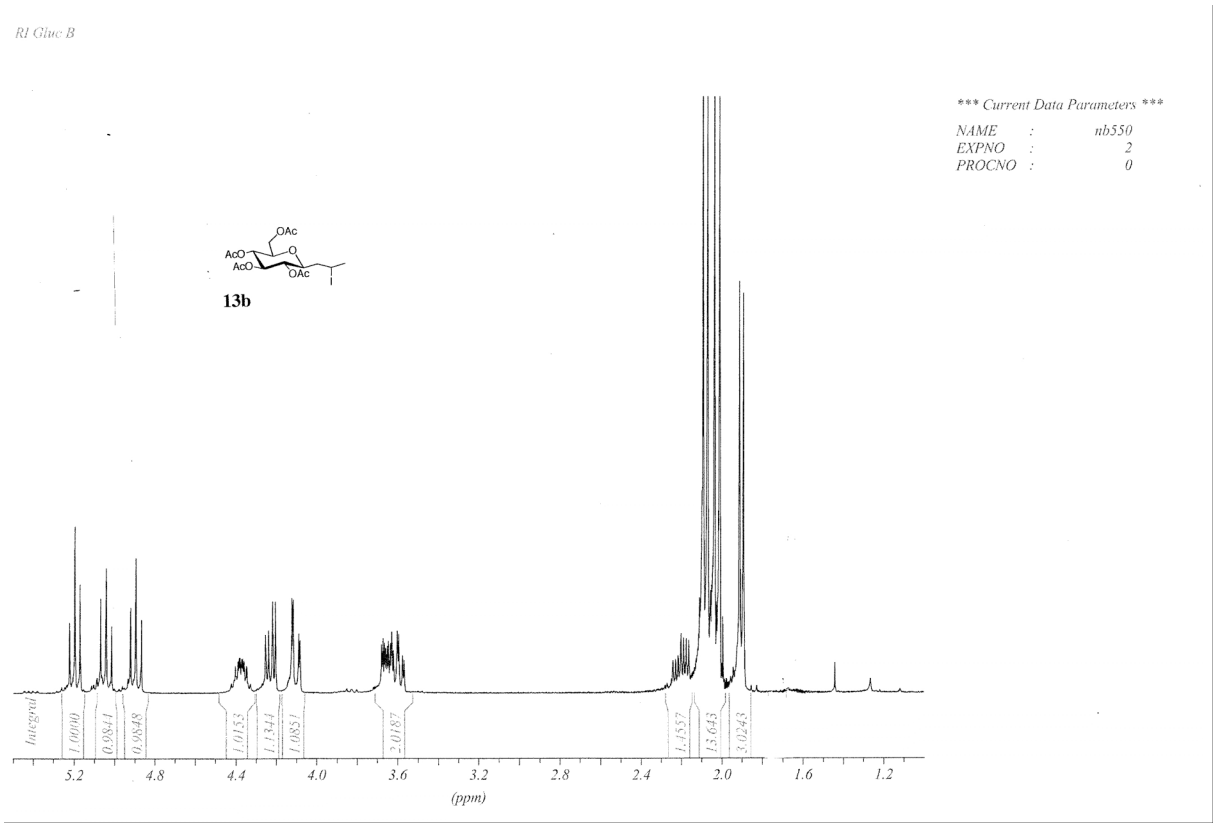
DEC_MC : PO
DECSTAT : BB
DP3000 : 15 dB
DS : 2
FMFLAG : 0
FW : 17400.00 Hz
INSTRUM : AM
LOCNUC : Lock ?
NS : 1200
NUCLEUS : Nuc ?
O1 : 2907.01 Hz
O2 : 3871.26 Hz
PULPROG :
RO : 20 Hz
SFO1 : 62.8968814 MHz
SOLVENT : Solvent ?
SW : 220.8232 ppm
SW_h : 13888.889 Hz
TD : 32768
TE : 297 K
VD : 0.0000000 sec
YMAX_a 349773.2500000
YMIN_a : -349773.2500000
*** Processing Parameters ***
ALPHA : 0.0000000
FT_mod : no
GAMMA : 0.0000000
GB : 0.0000000
LB : 3.00 Hz
NZP : 0
NC_proc : 0
OFFSET : 220.851 ppm
PHC0 : 1.364 degree
PHC1 : 30.473 degree


NB ROH MAN A (300 MHz)
OAc
OAc
AcO
AcO
OH
11a
1500
1000
500
0
1.00
0.93
0.89
1.96
0.96
0.93
0.96
0.91
11.07
2.00
1.16
3.22
5.0
4.0
3.0
2.0
1.0
ppm (f1)


NB ROH MAN B (300 MHz)
OAc
OAc
AcO
AcO
O
OH
11b
1500
1000
500
0
0.92
0.94
0.96
0.97
0.99
1.83
1.00
11.18
2.64
1.06
2.84
5.00
4.50
4.00
3.50
3.00
2.50
2.00
1.50
ppm (f1)


NB ROH GAL (300 MHz)
OAc
OAc
AcO
O
OAc
OH
12a
1000
500
0
1.00
1.09
0.79
3.90
0.99
0.95
10.95
2.86
3.10
5.50
5.00
4.50
4.00
3.50
3.00
2.50
2.00
1.50
ppm (f1)


RI Gluc A
*** Current Data Parameters ***
NAME : nb550
EXPNO : 1
PROCNO : 0
13a
Integral
1.0000
1.0180
0.9651
1.0352
1.0202
1.0055
3.0575
3.0330
2.9919
3.0018
2.8656
3.0133
1.1371
1.2244
5.6
5.2
4.8
4.4
4.0
3.6
3.2
2.8
2.4
2.0
1.6
1.2
(ppm)


RI Gluc B
*** Current Data Parameters ***
NAME : nb550
EXPNO : 2
PROCNO : 0
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Integral
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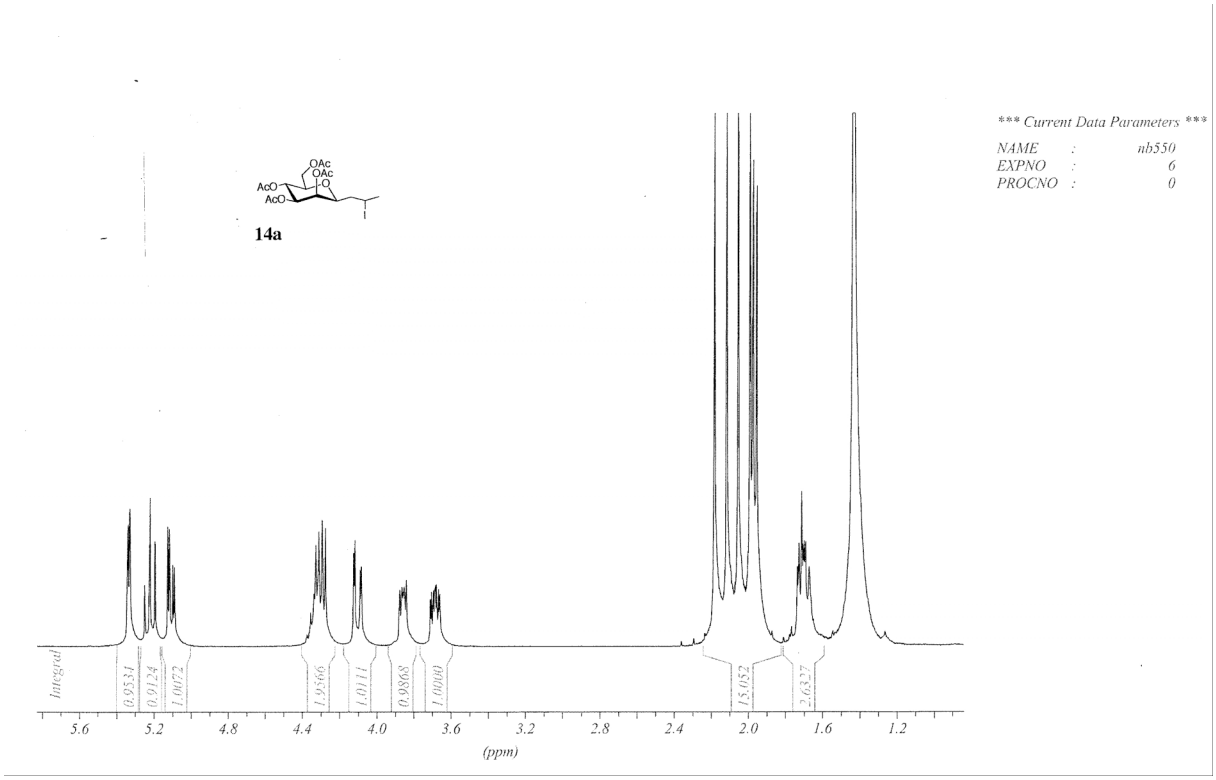
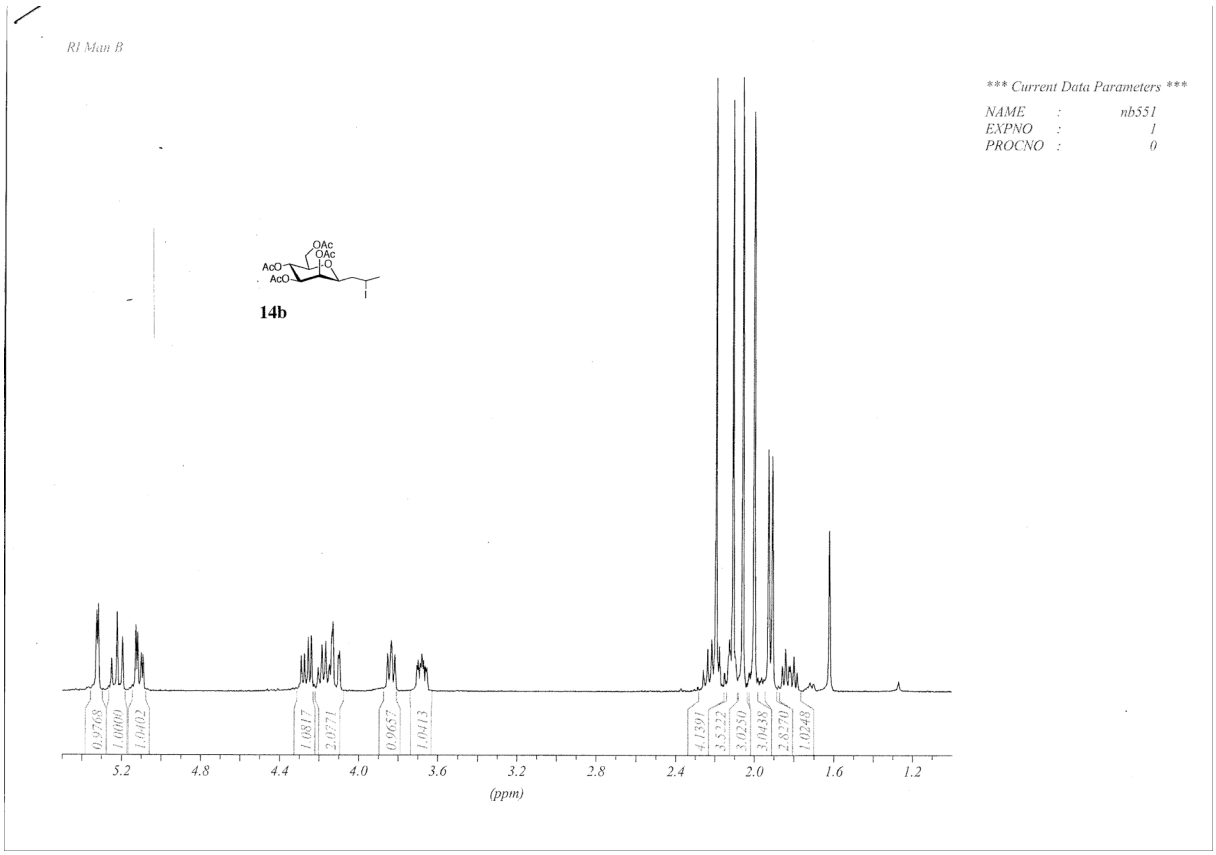
0.9841
0.9848
1.0155
1.1344
1.0851
3.0187
1.1557
15.643
3.0243
5.2
4.8
4.4
4.0
3.6
3.2
2.8
2.4
2.0
1.6
1.2
(ppm)


14a
*** Current Data Parameters ***
NAME : nb550
EXPNO : 6
PROCNO : 0
Integral
0.9531
0.9124
1.0072
1.9566
1.0114
0.9868
1.0000
15.052
2.6327
5.6
5.2
4.8
4.4
4.0
3.6
3.2
2.8
2.4
2.0
1.6
1.2
(ppm)


RI Man B
14b
*** Current Data Parameters ***
NAME : nb551
EXPNO : 1
PROCNO : 0
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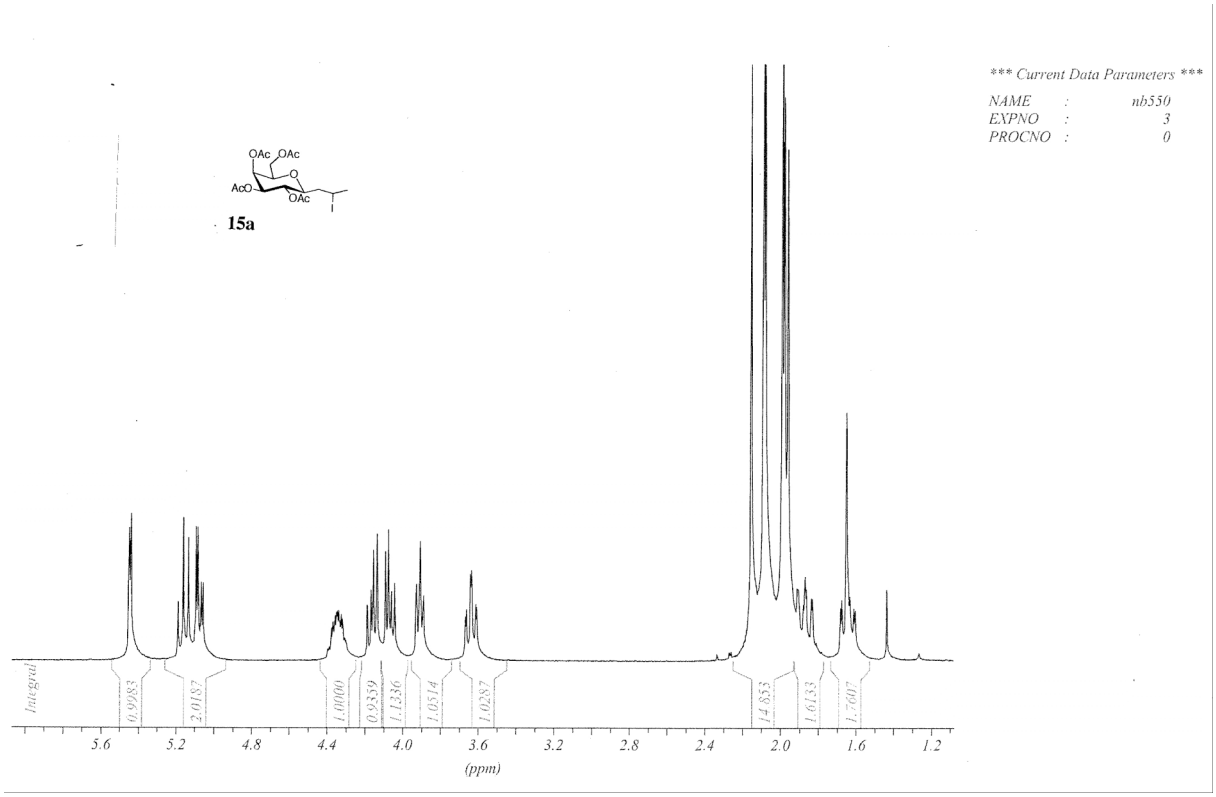
1.0000
1.0402
1.0817
2.0771
0.9657
1.0413
4.1391
3.5222
3.0250
3.0438
2.8220
1.0248
5.2
4.8
4.4
4.0
3.6
3.2
2.8
2.4
2.0
1.6
1.2
(ppm)


*** Current Data Parameters ***
NAME : nb550
EXPNO : 3
PROCNO : 0
OAc OAc
O
AcO
OAc
I
15a
Integral
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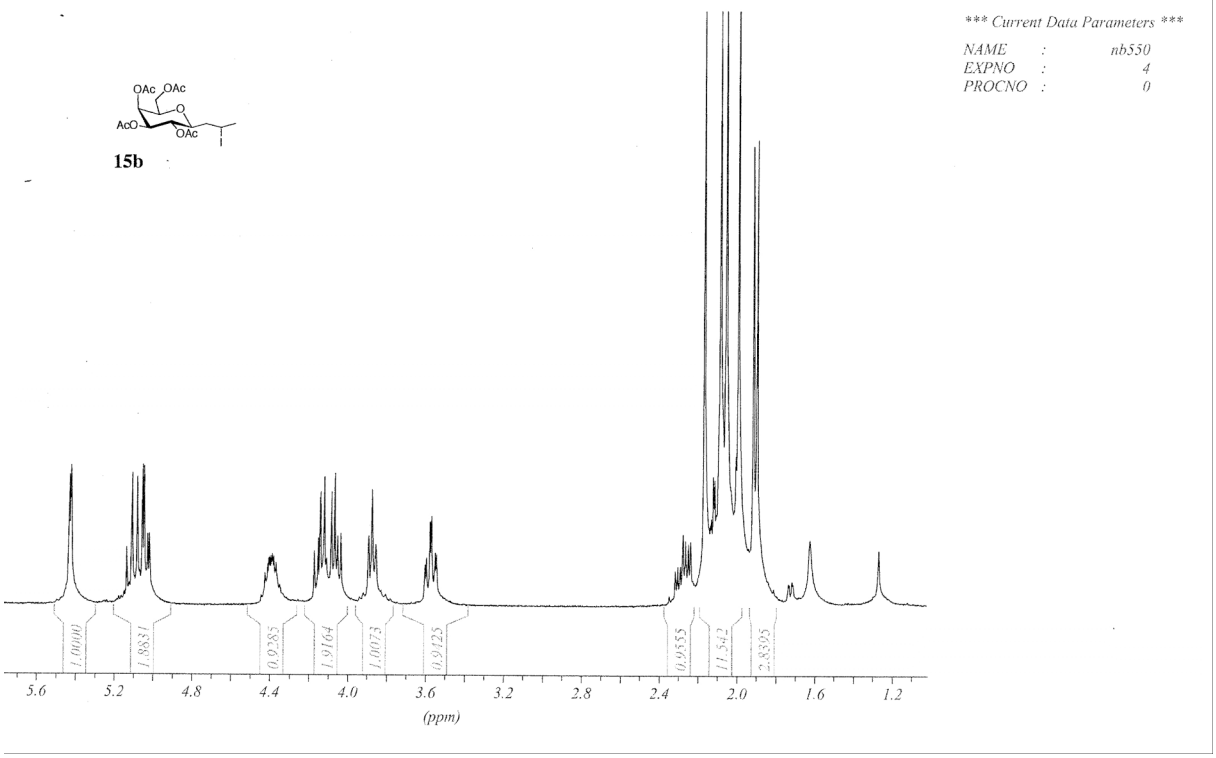
2.0187
1.0000
0.9359
1.1336
1.0514
1.0287
14.853
1.6133
1.7607
5.6
5.2
4.8
4.4
4.0
3.6
3.2
2.8
2.4
2.0
1.6
1.2
(ppm)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PROCNO : 0
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O
AcO
OAc
I
15b
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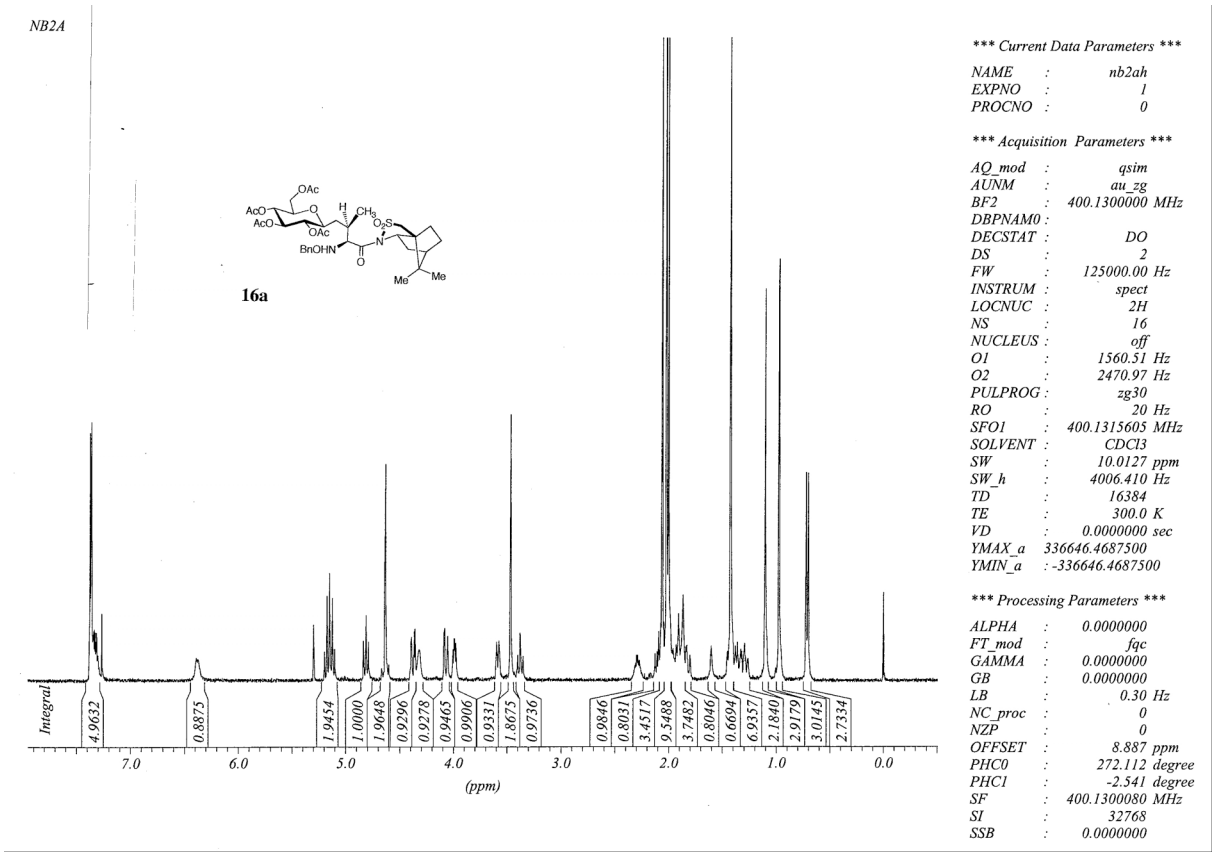
1.8831
0.9285
1.9164
1.0073
0.9125
0.9555
11.542
2.8395
5.6
5.2
4.8
4.4
4.0
3.6
3.2
2.8
2.4
2.0
1.6
1.2
(ppm)


NB2A
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0.8875
1.9454
1.0000
1.9648
0.9296
0.9278
0.9465
0.9906
0.9331
1.8675
0.9736
0.9846
0.8031
3.4517
9.5488
3.7482
0.8046
0.6694
6.9357
2.1840
2.9179
3.0145
2.7334
7.0
6.0
5.0
4.0
3.0
2.0
1.0
0.0
(ppm)
*** Current Data Parameters ***
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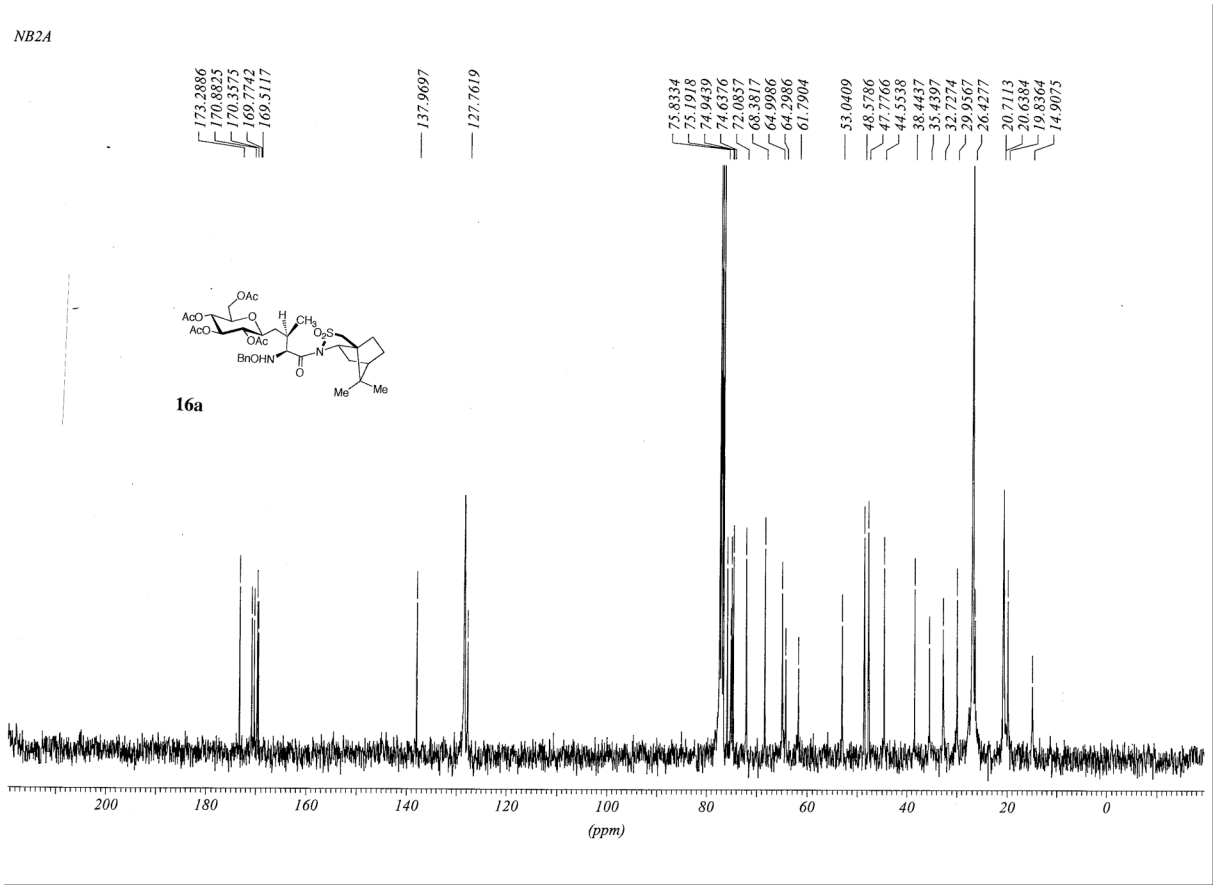
EXPNO : 1
PROCNO : 0
*** Acquisition Parameters ***
AQ_mod : qsim
AUNM : au_zg
BF2 : 400.1300000 MHz
DBPNAM0 :
DECSTAT : DO
DS : 2
FW : 125000.00 Hz
INSTRUM : spect
LOCNUC : 2H
NS : 16
NUCLEUS : off
O1 : 1560.51 Hz
O2 : 2470.97 Hz
PULPROG : zg30
RO : 20 Hz
SFO1 : 400.1315605 MHz
SOLVENT : CDCl3
SW : 10.0127 ppm
SW_h : 4006.410 Hz
TD : 16384
TE : 300.0 K
VD : 0.0000000 sec
YMAX_a 336646.4687500
YMIN_a : -336646.4687500
*** Processing Parameters ***
ALPHA : 0.0000000
FT_mod : fqc
GAMMA : 0.0000000
GB : 0.0000000
LB : 0.30 Hz
NC_proc : 0
NZP : 0
OFFSET : 8.887 ppm
PHC0 : 272.112 degree
PHC1 : -2.541 degree
SF : 400.1300080 MHz
SI : 32768
SSB : 0.0000000


NB2A
16a
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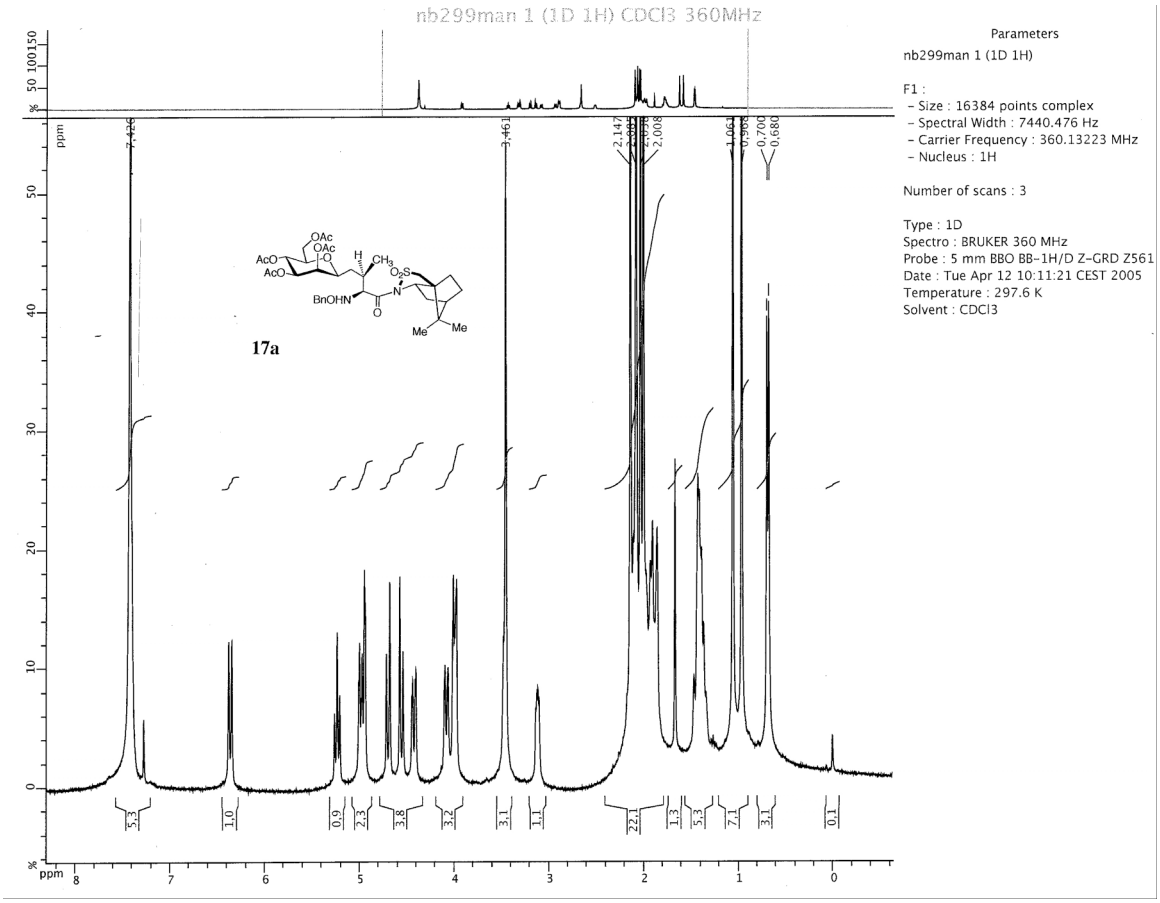
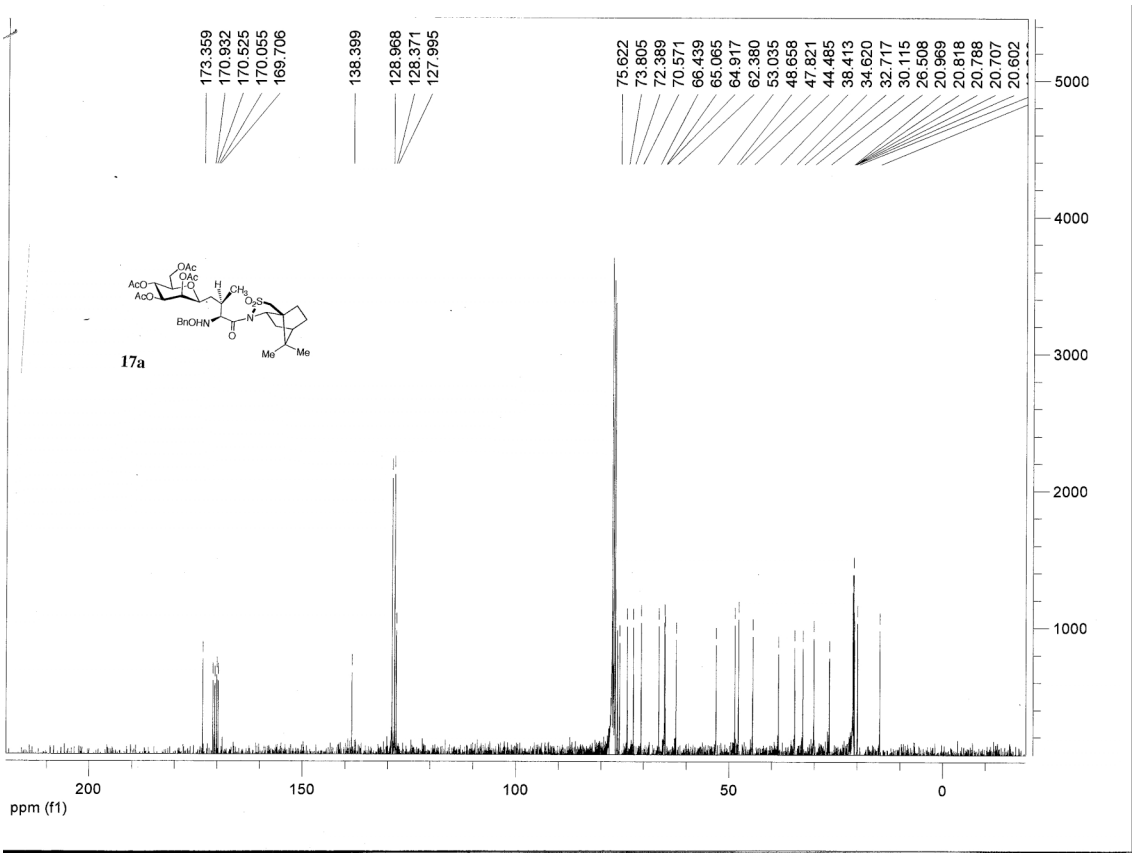
170.8825
170.3575
169.7742
169.5117
137.9697
127.7619
75.8334
75.1918
74.9439
74.6376
72.0857
68.3817
64.9986
64.2986
61.7904
53.0409
48.5786
47.7766
44.5538
38.4437
35.4397
32.7274
29.9567
26.4277
20.7113
20.6384
19.8364
14.9075
200
180
160
140
120
100
80
60
40
20
0
(ppm)


nb299man 1 (1D 1H) CDCl3 360MHz
Parameters
nb299man 1 (1D 1H)
F1 :
- Size : 16384 points complex
- Spectral Width : 7440.476 Hz
- Carrier Frequency : 360.13223 MHz
- Nucleus : 1H
Number of scans : 3
Type : 1D
Spectro : BRUKER 360 MHz
Probe : 5 mm BBO BB-1H/D Z-GRD Z561
Date : Tue Apr 12 10:11:21 CEST 2005
Temperature : 297.6 K
Solvent : CDCl3
17a
ppm


173.359
170.932
170.525
170.055
169.706
138.399
128.968
128.371
127.995
75.622
73.805
72.389
70.571
66.439
65.065
64.917
62.380
53.035
48.658
47.821
44.485
38.413
34.620
32.717
30.115
26.508
20.969
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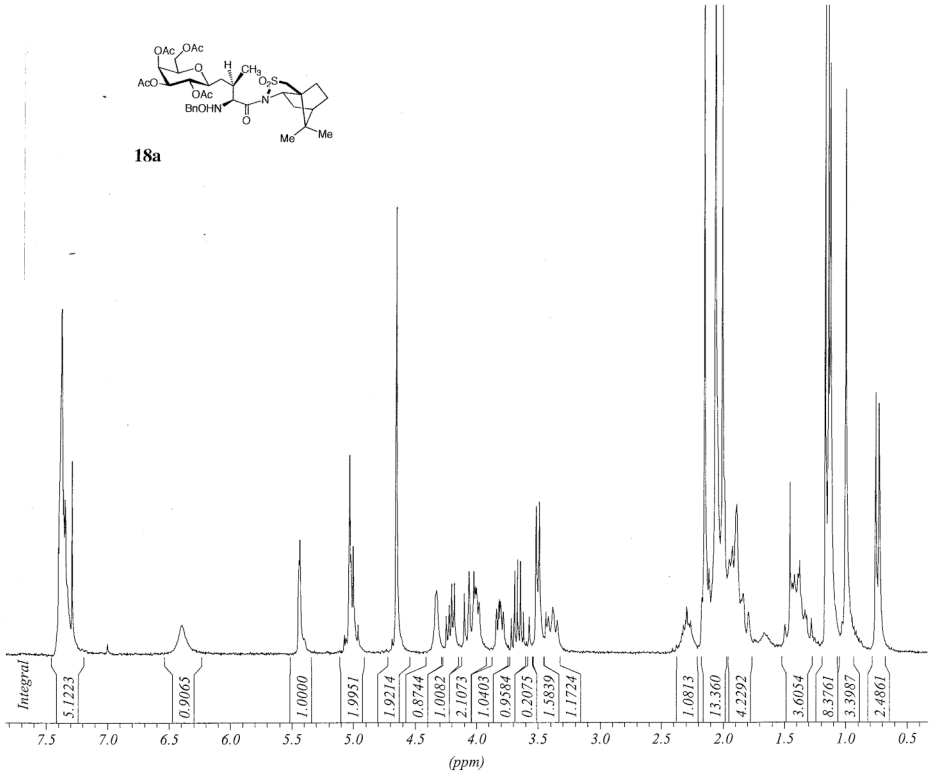
20.707
20.602
17a
ppm (f1)

NB 353B



*** Current Data Parameters ***

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PROCNO	:	0

*** Acquisition Parameters ***

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AUNM	:	au_zg
BF2	:	250.1300000 MHz
DBPNAM0	:	
DECSTAT	:	DO
DS	:	2
FW	:	93750.00 Hz
INSTRUM	:	spect
LOCNUC	:	2H
NS	:	16
NUCLEUS	:	off
O1	:	1544.66 Hz
O2	:	1544.66 Hz
PULPROG	:	zg30
RO	:	20 Hz
SFO1	:	250.1315447 MHz
SOLVENT	:	CDCl3
SW	:	20.6930 ppm
SW_h	:	5175.983 Hz
TD	:	16384
TE	:	0.0 K
VD	:	0.0000000 sec
YMAX_a		414874.0000000
YMIN_a		-414874.0000000

*** Processing Parameters ***

ALPHA	:	0.0000000
FT_mod	:	fqc
GAMMA	:	0.0000000
GB	:	0.0000000
LB	:	0.00 Hz
NC_proc	:	0
NZP	:	0
OFFSET	:	16.522 ppm
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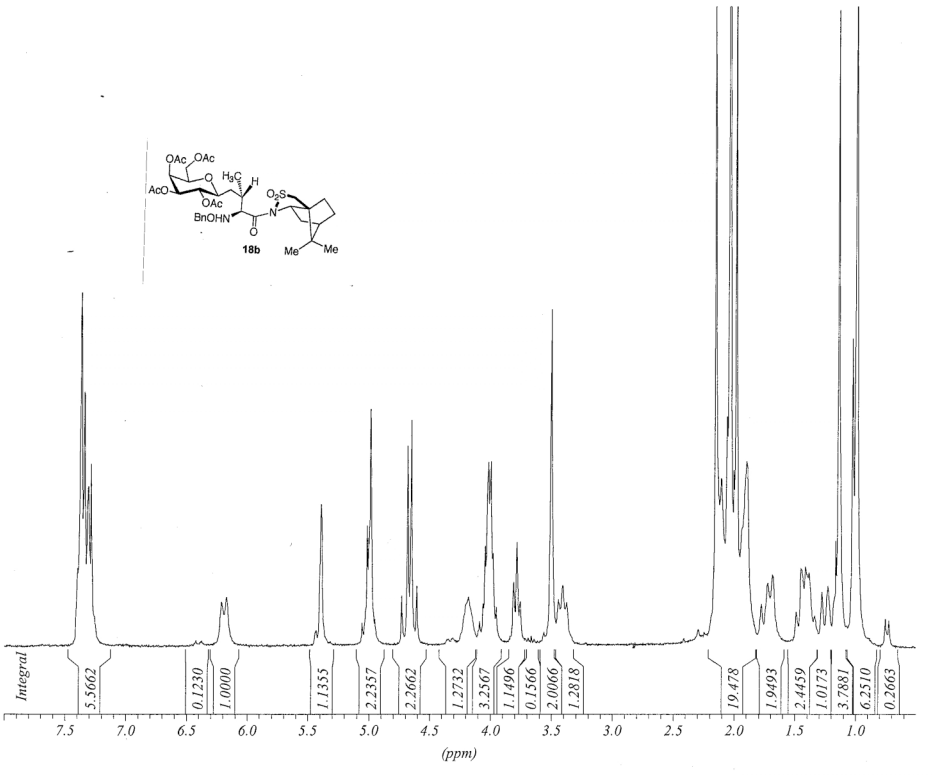
PHC1	:	4.403 degree
SF	:	250.1300000 MHz
SI	:	8192
SSB	:	0.0000000

NB 353A



*** Current Data Parameters ***

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EXPNO	:	1
PROCNO	:	0

*** Acquisition Parameters ***

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AUNM	:	au_zg
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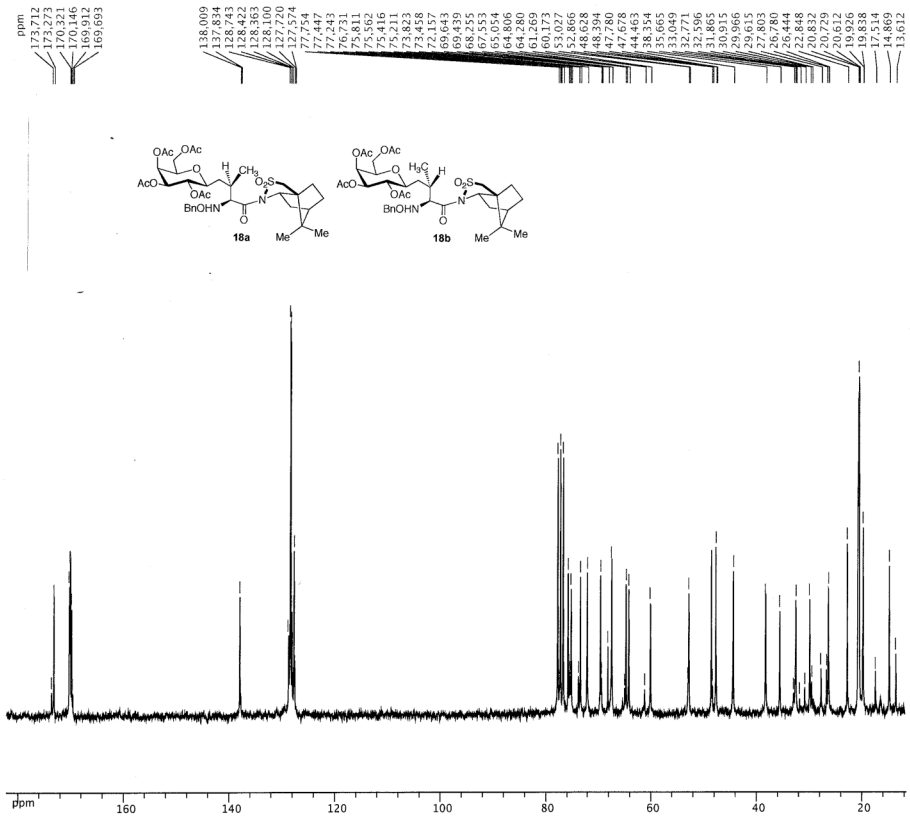
DECSTAT	:	DO
DS	:	2
FW	:	93750.00 Hz
INSTRUM	:	spect
LOCNUC	:	2H
NS	:	16
NUCLEUS	:	off
O1	:	1544.66 Hz
O2	:	1544.66 Hz
PULPROG	:	zg30
RO	:	20 Hz
SFO1	:	250.1315447 MHz
SOLVENT	:	CDCl3
SW	:	20.6930 ppm
SW_h	:	5175.983 Hz
TD	:	16384
TE	:	0.0 K
VD	:	0.0000000 sec
YMAX_a		535147.0000000
YMIN_a		-535147.0000000

*** Processing Parameters ***

ALPHA	:	0.0000000
FT_mod	:	fqc
GAMMA	:	0.0000000
GB	:	0.0000000
LB	:	0.00 Hz
NC_proc	:	0
NZP	:	0
OFFSET	:	16.522 ppm
PHC0	:	0.000 degree
PHC1	:	0.000 degree
SF	:	250.1300000 MHz
SI	:	8192
SSB	:	0.0000000

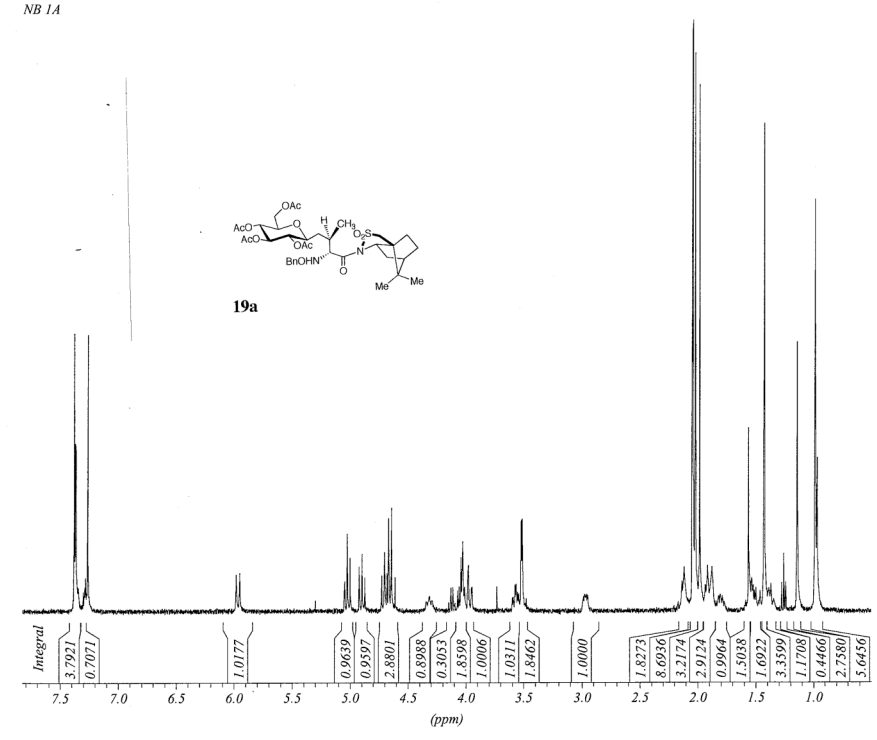


Parameters
nb591 2 (1D 13C)

F1 :
- Size : 16384 points complex
- Spectral Width : 15060.241 Hz
- Carrier Frequency : 62.901527 MHz
- Nucleus : 13C

Number of scans : 672

Type : 1D
Spectro : BRUKER 250 MHz
Probe : 5 mm QNP 1H/13C/31P/19F Z3(
Date : Fri Oct 06 10:03:12 CEST 2006
Temperature : 0.0 K
Solvent : CDCl3



*** Current Data Parameters ***

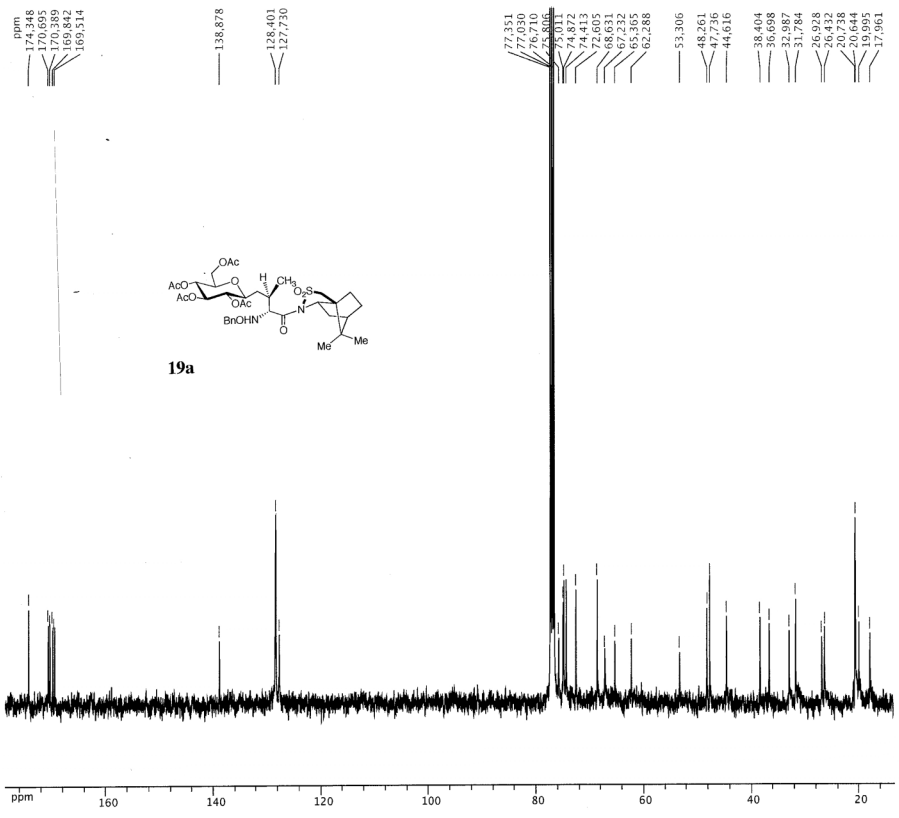
NAME	:	nb1ah
EXPNO	:	1
PROCNO	:	0

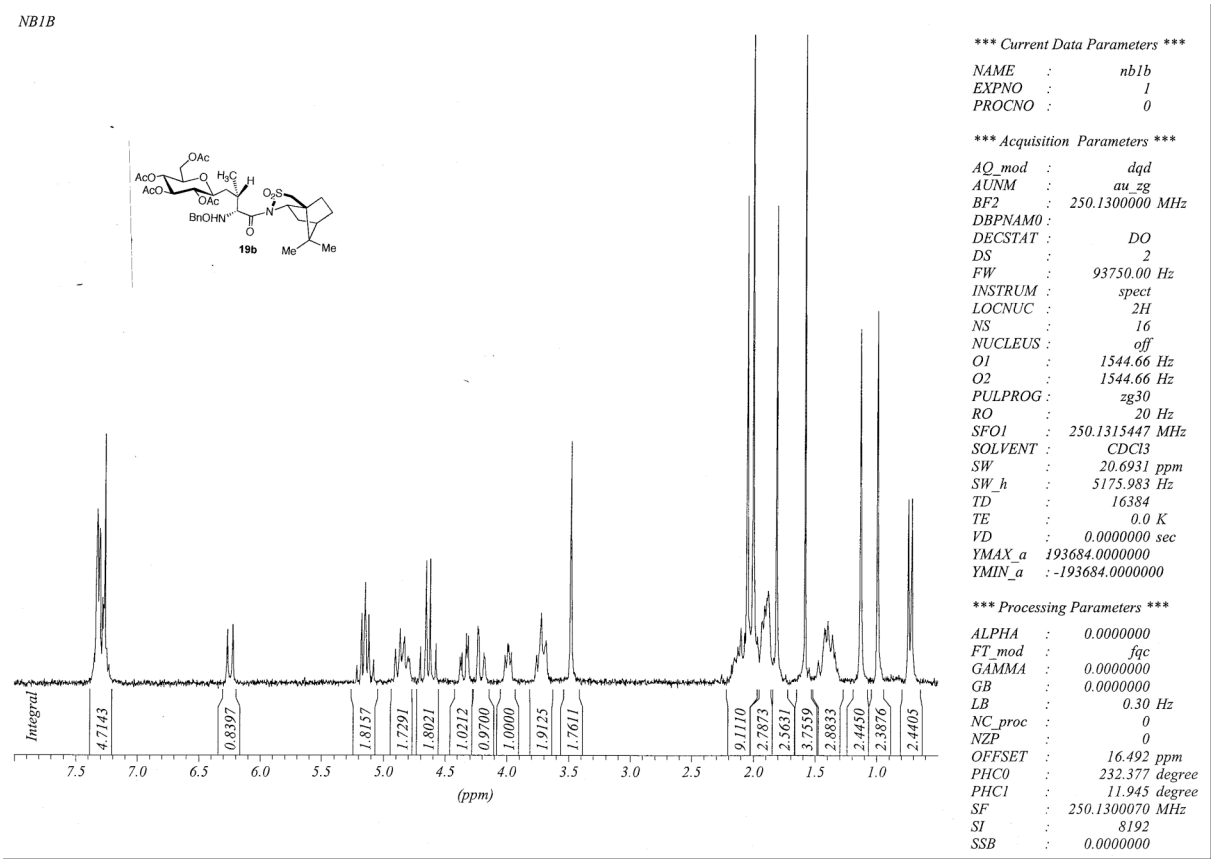
*** Acquisition Parameters ***

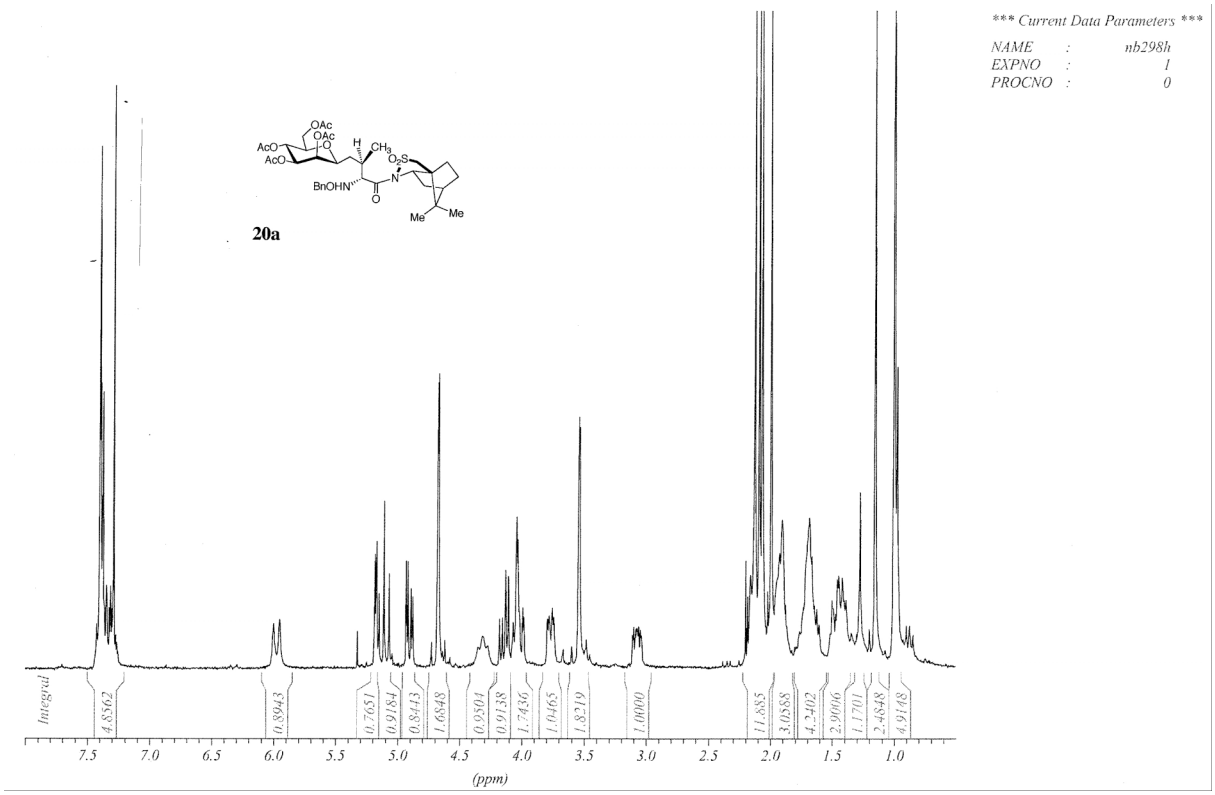
AQ_mod	:	qsim
AUNM	:	au_zg
BF2	:	400.1300000 MHz
DBPNAM0	:	
DECSTAT	:	DO
DS	:	2
FW	:	125000.00 Hz
INSTRUM	:	spect
LOCNUC	:	2H
NS	:	16
NUCLEUS	:	off
O1	:	1560.51 Hz
O2	:	2470.97 Hz
PULPROG	:	zg30
RO	:	20 Hz
SFO1	:	400.1315605 MHz
SOLVENT	:	CDCl3
SW	:	10.0127 ppm
SW_h	:	4006.410 Hz
TD	:	16384
TE	:	300.0 K
VD	:	0.0000000 sec
YMAX_a		259751.5781250
YMIN_a		-259751.5781250

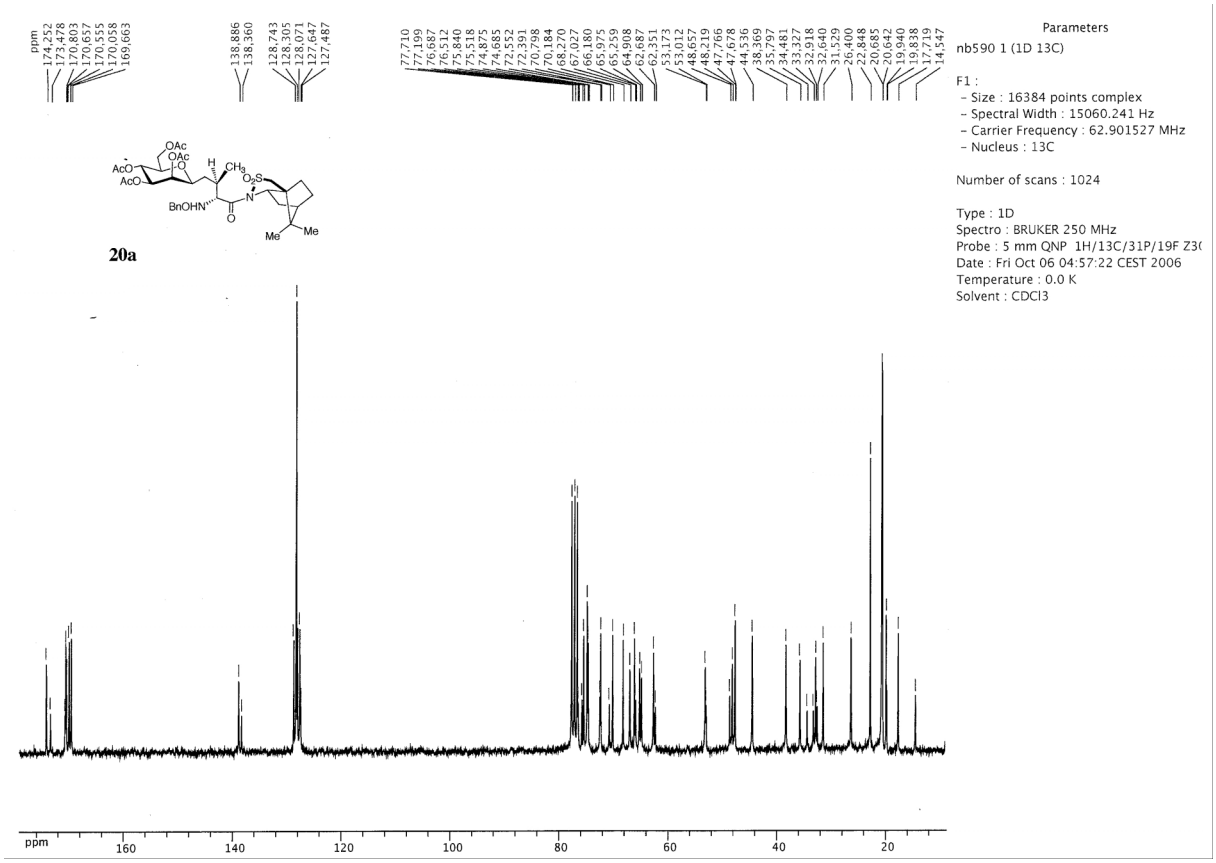
*** Processing Parameters ***

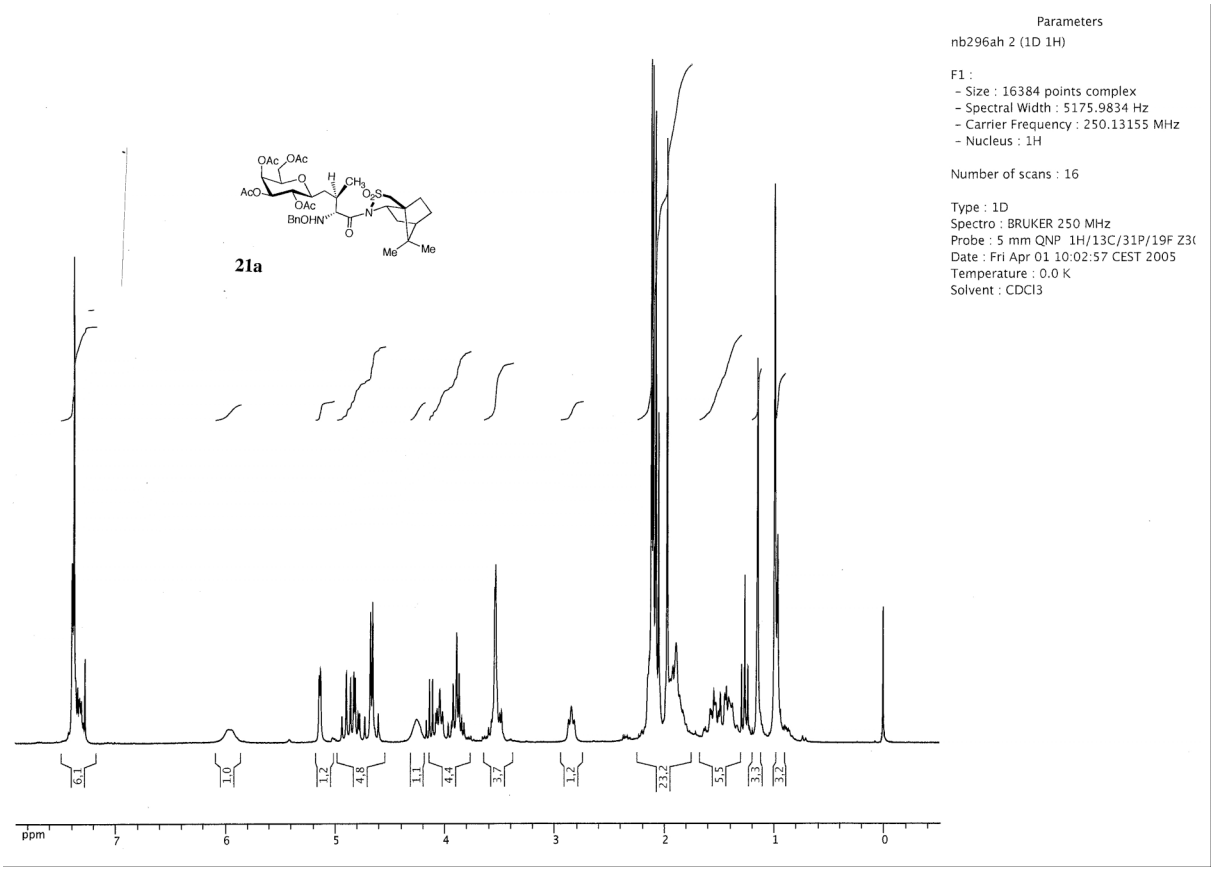
ALPHA	:	0.0000000
FT_mod	:	fqc
GAMMA	:	0.0000000
GB	:	0.0000000
LB	:	0.30 Hz
NC_proc	:	0
NZP	:	0
OFFSET	:	8.884 ppm
PHC0	:	272.742 degree
PHC1	:	2.528 degree
SF	:	400.1300090 MHz
SI	:	32768
SSB	:	0.0000000

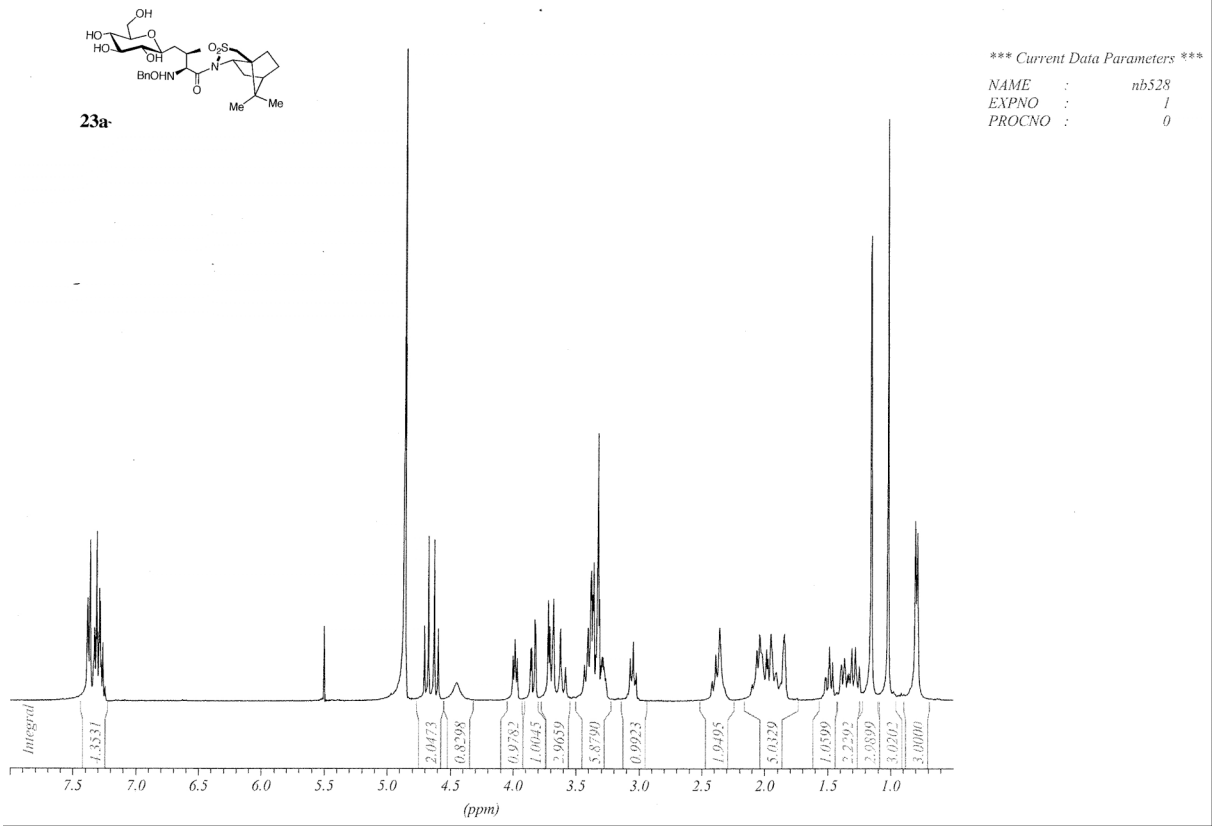

ppm
174.348
170.695
170.389
169.842
169.514
138.878
128.491
127.730
77.351
77.030
76.710
75.806
75.011
74.872
74.413
72.605
68.631
67.232
65.565
62.288
53.306
48.261
47.736
44.616
38.404
36.698
32.987
31.784
26.928
26.432
20.738
20.644
19.995
17.961
Parameters
nb1ah 4 (1D 13C)
F1 :
- Size : 32768 points complex
- Spectral Width : 24038.46 Hz
- Carrier Frequency : 100.62283 MHz
- Nucleus : 13C
Number of scans : 1024
Type : 1D
Spectro : BRUKER 400 MHz
Probe : 5 mm BBO 13C-1H Z-GRD Z8248
Date : Wed Mar 16 02:33:10 CET 2005
Temperature : 300.0 K
Solvent : CDCl3
OAc
AcO
AcO
OAc
BnOHN
Me
Me
19a
ppm
160
140
120
100
80
60
40
20

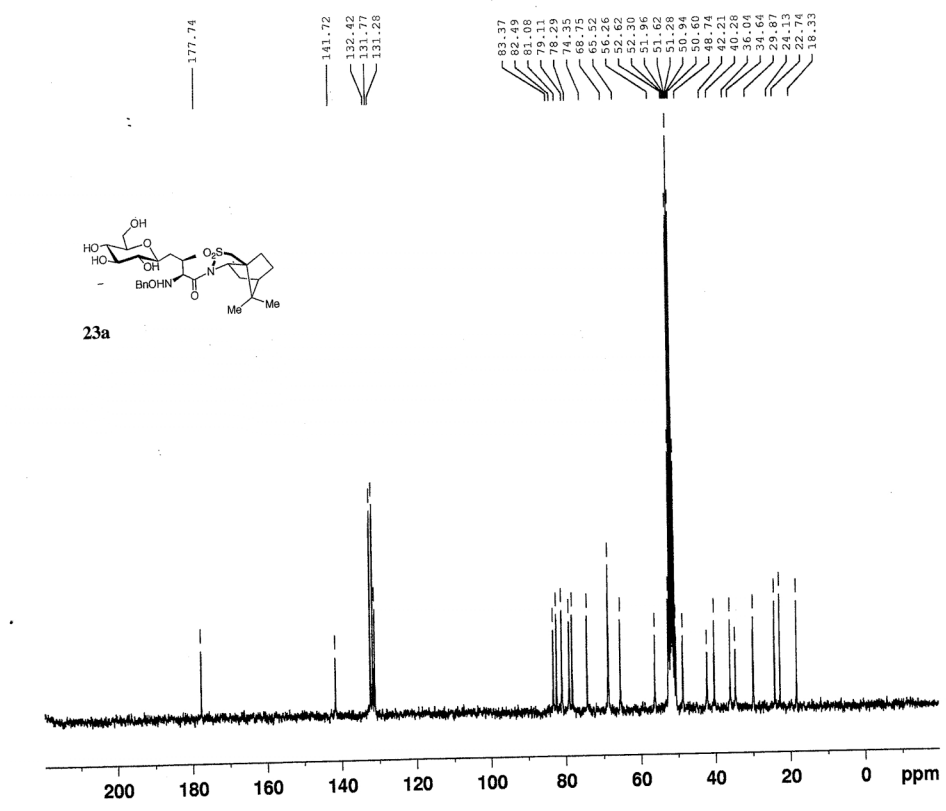

NB1B
OAc
AcO
AcO
OAc
BnOHN
19b
Me
Me
Integral
4.7143
0.8397
1.8157
1.7291
1.8021
1.0212
0.9700
1.0000
1.9125
1.7611
9.1110
2.7873
2.5631
3.7559
2.8833
2.4450
2.3876
2.4405
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
(ppm)
*** Current Data Parameters ***
NAME : nb1b
EXPNO : 1
PROCNO : 0
*** Acquisition Parameters ***
AQ_mod : dqd
AUNM : au_zg
BF2 : 250.1300000 MHz
DBPNAM0 :
DECSTAT : DO
DS : 2
FW : 93750.00 Hz
INSTRUM : spect
LOCNUC : 2H
NS : 16
NUCLEUS : off
O1 : 1544.66 Hz
O2 : 1544.66 Hz
PULPROG : zg30
RO : 20 Hz
SFO1 : 250.1315447 MHz
SOLVENT : CDCl3
SW : 20.6931 ppm
SW_h : 5175.983 Hz
TD : 16384
TE : 0.0 K
VD : 0.0000000 sec
YMAX_a 193684.0000000
YMIN_a : -193684.0000000
*** Processing Parameters ***
ALPHA : 0.0000000
FT_mod : fqc
GAMMA : 0.0000000
GB : 0.0000000
LB : 0.30 Hz
NC_proc : 0
NZP : 0
OFFSET : 16.492 ppm
PHC0 : 232.377 degree
PHC1 : 11.945 degree
SF : 250.1300070 MHz
SI : 8192
SSB : 0.0000000


*** Current Data Parameters ***
NAME : nb298h
EXPNO : 1
PROCNO : 0
20a
Integral
4.8562
0.8943
0.7651
0.9184
0.8443
1.6848
0.9504
0.9138
1.7436
1.0465
1.8219
1.0000
11.885
3.0388
4.2402
2.9006
1.1701
2.4648
4.9148
(ppm)

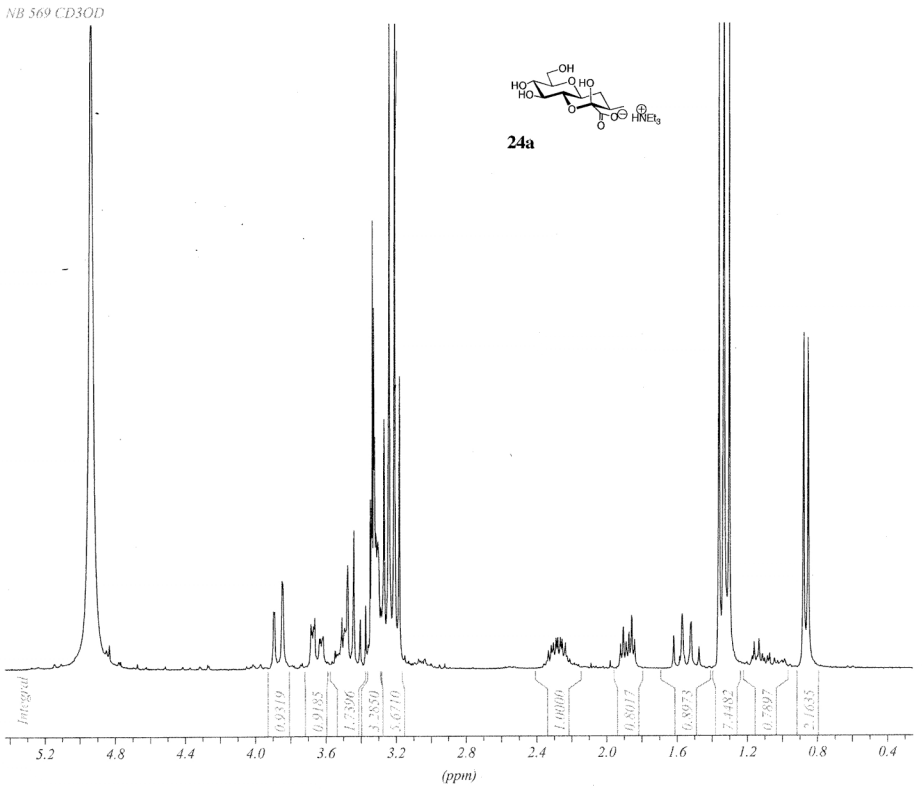

ppm
174,252
173,478
170,803
170,657
170,555
170,058
169,663
138,886
138,360
128,743
128,305
128,071
127,647
127,487
77,710
77,199
76,687
76,175
75,840
75,518
74,875
74,685
72,552
72,391
70,798
70,184
68,270
67,027
66,180
65,975
65,559
64,908
64,687
62,351
53,173
53,012
48,657
48,219
47,766
47,678
44,536
38,369
35,797
34,481
33,327
32,918
32,646
31,529
26,400
22,848
20,685
20,642
19,940
19,838
17,719
14,547
20a
Parameters
nb590 1 (1D 13C)
F1 :
- Size : 16384 points complex
- Spectral Width : 15060.241 Hz
- Carrier Frequency : 62.901527 MHz
- Nucleus : 13C
Number of scans : 1024
Type : 1D
Spectro : BRUKER 250 MHz
Probe : 5 mm QNP 1H/13C/31P/19F Z3(
Date : Fri Oct 06 04:57:22 CEST 2006
Temperature : 0.0 K
Solvent : CDCl3
ppm
160
140
120
100
80
60
40
20


Parameters
nb296ah 2 (1D 1H)
F1 :
- Size : 16384 points complex
- Spectral Width : 5175.9834 Hz
- Carrier Frequency : 250.13155 MHz
- Nucleus : 1H
Number of scans : 16
Type : 1D
Spectro : BRUKER 250 MHz
Probe : 5 mm QNP 1H/13C/31P/19F Z3(
Date : Fri Apr 01 10:02:57 CEST 2005
Temperature : 0.0 K
Solvent : CDCl3
21a
ppm

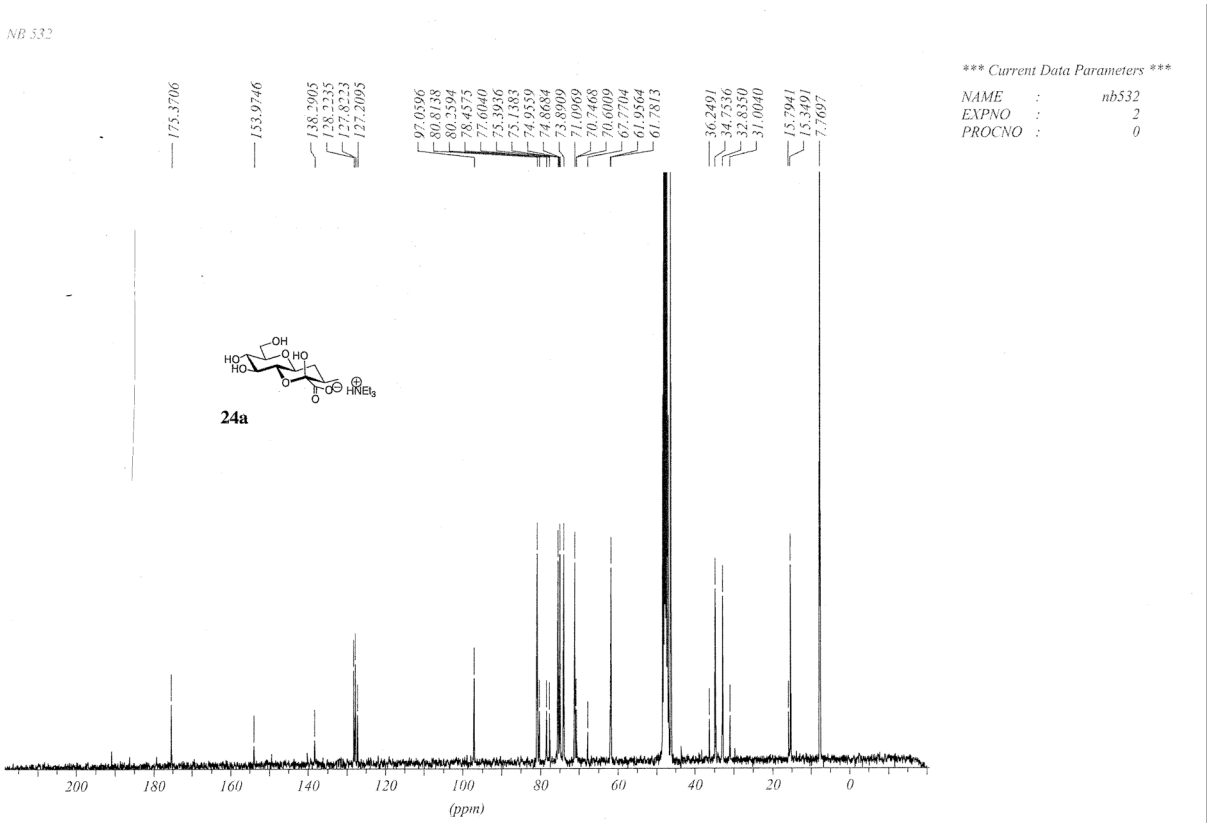

*** Current Data Parameters ***
NAME : nb528
EXPNO : 1
PROCNO : 0
23a
Integral
(ppm)

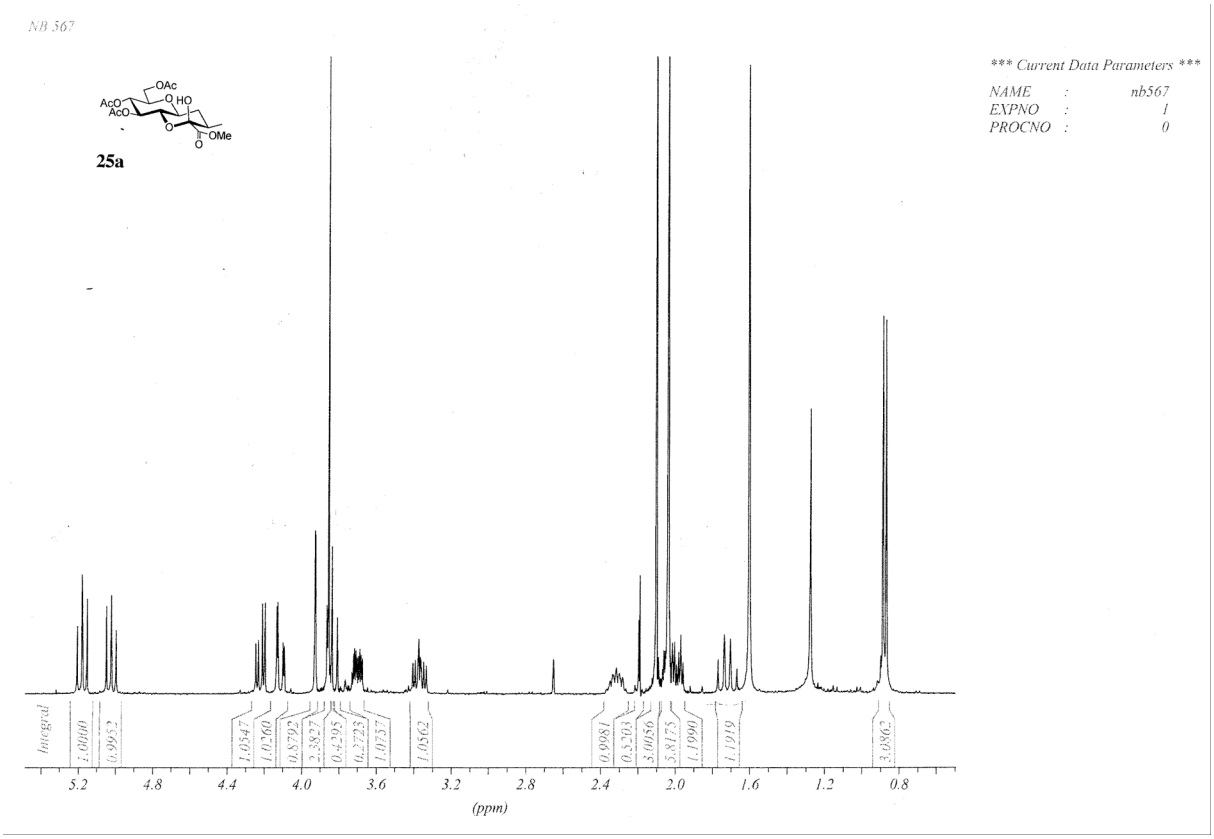


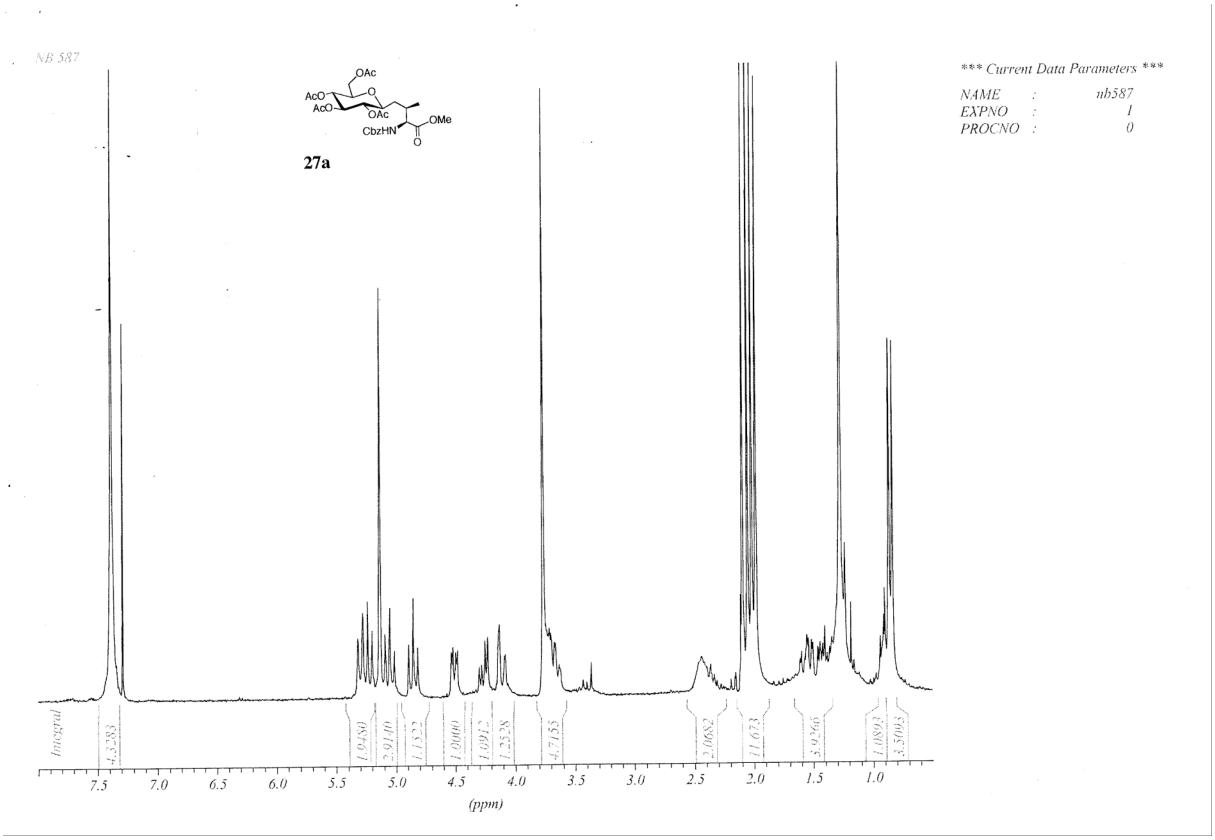

```
Current Data Parameters
NAME                  nb424
EXPNO                     1
PROCNO                    1

F2 - Acquisition Parameters
Date_              20051124
Time                 23.33
INSTRUM              spect
PROBHD    5 mm QNP  1H/1
PULPROG            zgpg30
TD                  16384
SOLVENT             CDCl3
NS                   1024
DS                      4
SWH             15060.241 Hz
FIDRES          0.919204 Hz
AQ              0.5439988 sec
RG                   6502
DW                33.200 usec
DE                 6.00 usec
TE                  0.0 K
D1             2.00000000 sec
d11            0.03000000 sec
DELTA          1.89999998 sec
MCREST         0.00000000 sec
MCWRK          0.01500000 sec

======== CHANNEL f1 ========
NUC1                 13C
P1                 6.90 usec
PL1                -6.00 dB
SFO1          62.9015280 MHz

======== CHANNEL f2 ========
CPDPRG2          waltz16
NUC2                  1H
PCPD2            100.00 usec
PL2                -6.00 dB
PL12               19.00 dB
PL13               19.00 dB
SFO2         250.1310005 MHz

F2 - Processing parameters
SI                   32768
SF            62.8952390 MHz
WDW                    EM
SSB                     0
LB                 3.00 Hz
GB                      0
PC                   1.40
```

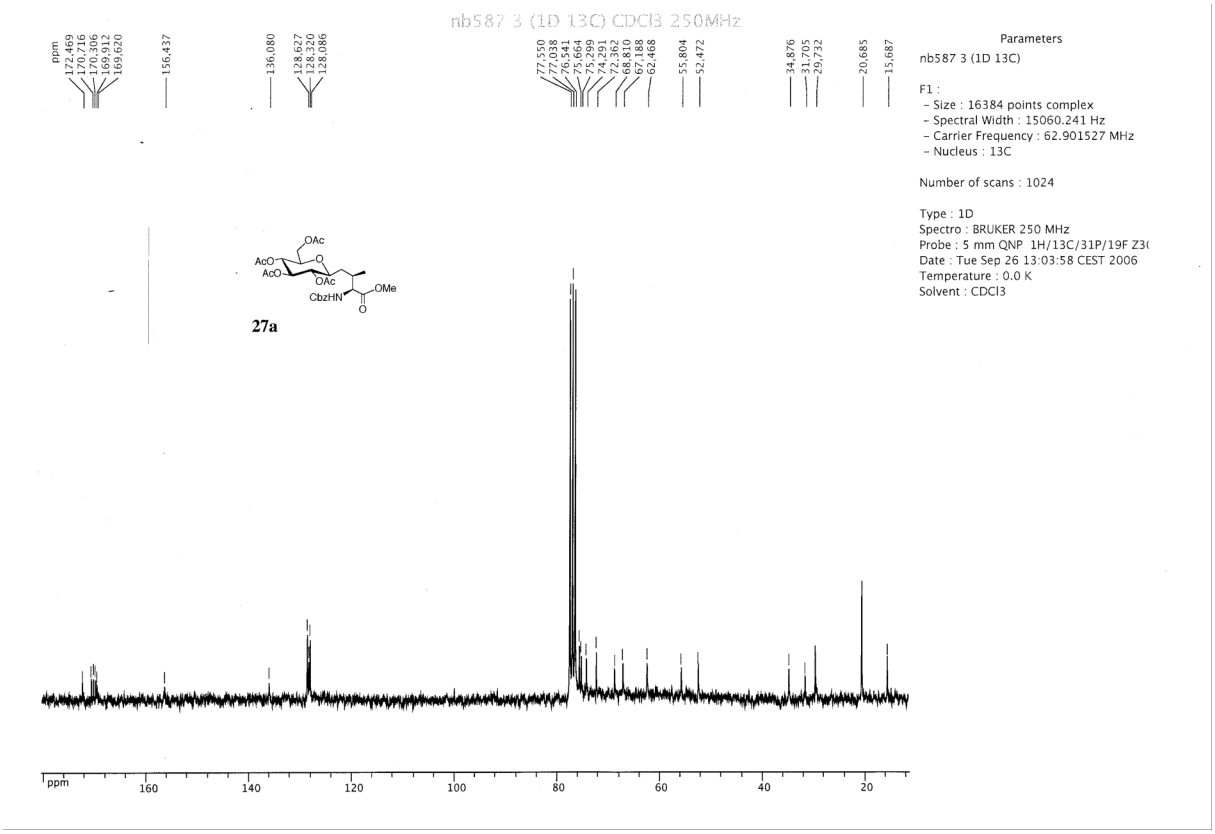


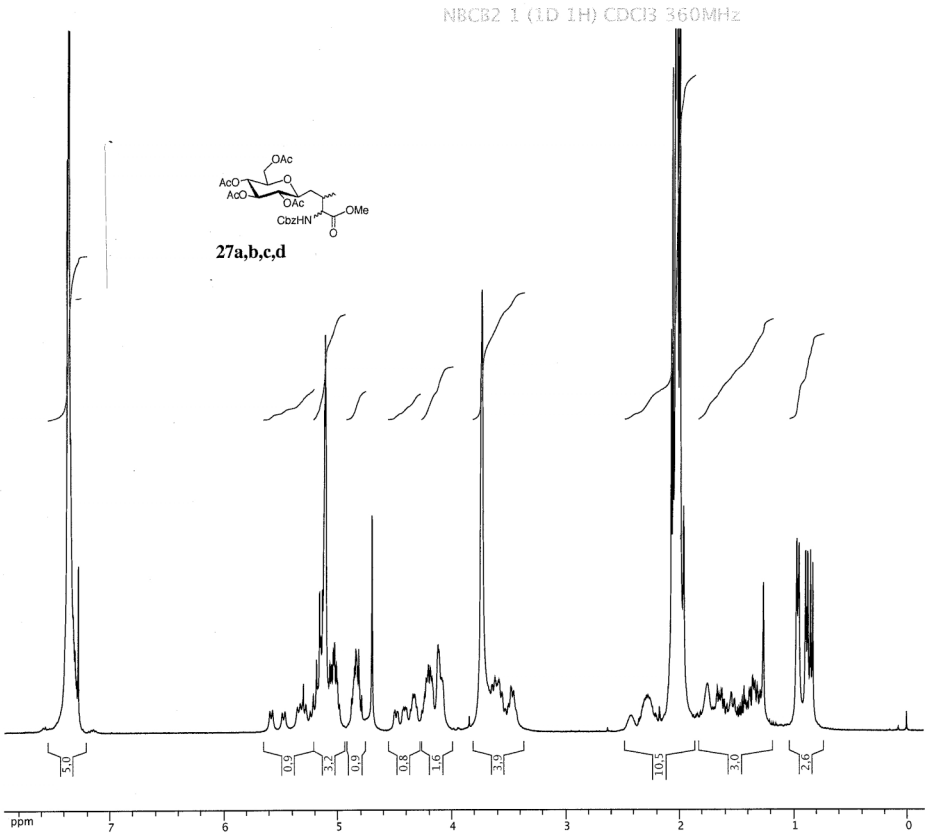

```
*** Current Data Parameters ***
NAME      :      nb569
EXPNO     :          1
PROCNO    :          0
```

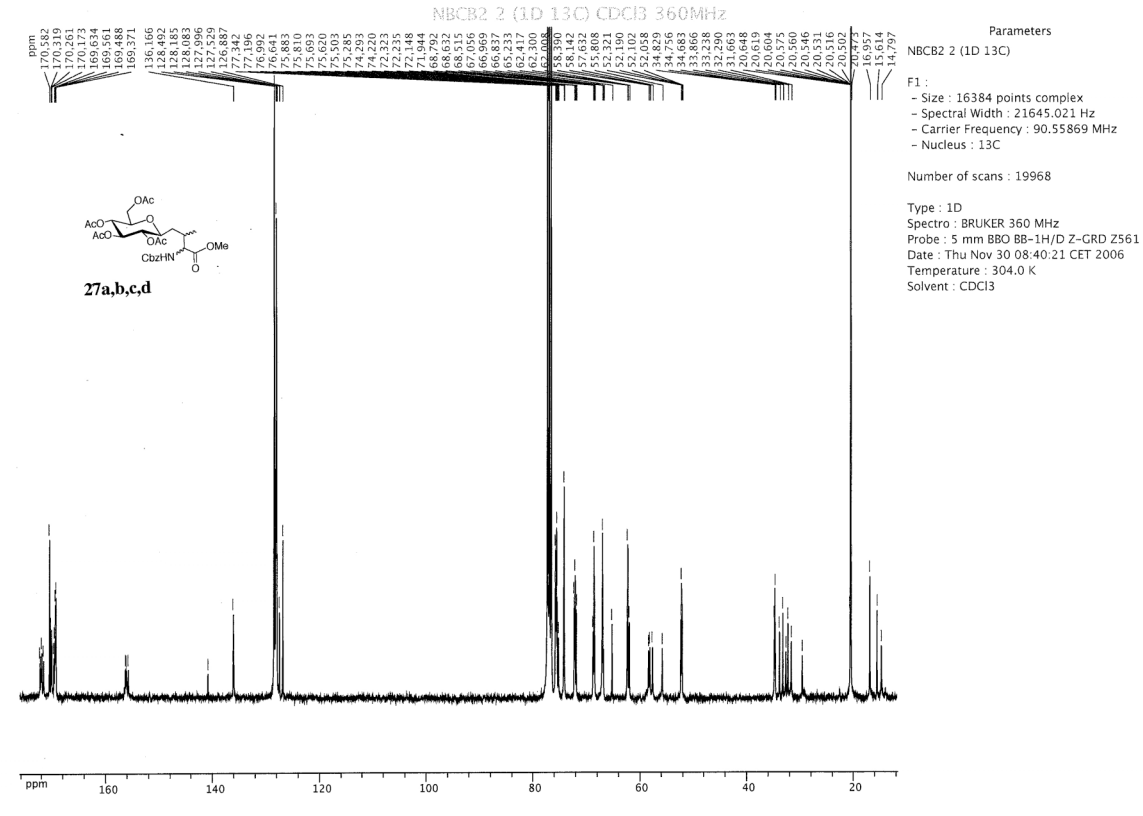

NB 532
*** Current Data Parameters ***
NAME : nb532
EXPNO : 2
PROCNO : 0
175.3706
153.9746
138.2905
128.2235
127.8223
127.2095
97.0596
80.8138
80.2594
78.4575
77.6040
75.3936
75.1383
74.9559
74.8684
73.8909
71.0969
70.7468
70.6009
67.7704
61.9564
61.7813
36.2491
34.7536
32.8350
31.0040
15.7941
15.3491
7.7697
24a
200
180
160
140
120
100
80
60
40
20
0
(ppm)


NB 567
*** Current Data Parameters ***
NAME : nb567
EXPNO : 1
PROCNO : 0
25a
Integral
1.0000
0.9952
1.0547
1.0260
0.8792
2.3827
0.4295
0.2723
1.0737
1.0562
0.9981
0.5203
5.0056
3.8175
1.1990
1.1919
3.0862
5.2
4.8
4.4
4.0
3.6
3.2
2.8
2.4
2.0
1.6
1.2
0.8
(ppm)


NB 587
27a
*** Current Data Parameters ***
NAME : nb587
EXPNO : 1
PROCNO : 0
Integral
(ppm)


nb587 3 (1D 13C) CDCl3 250MHz
ppm
27a
Parameters
nb587 3 (1D 13C)
F1 :
- Size : 16384 points complex
- Spectral Width : 15060.241 Hz
- Carrier Frequency : 62.901527 MHz
- Nucleus : 13C
Number of scans : 1024
Type : 1D
Spectro : BRUKER 250 MHz
Probe : 5 mm QNP 1H/13C/31P/19F Z3(
Date : Tue Sep 26 13:03:58 CEST 2006
Temperature : 0.0 K
Solvent : CDCl3


NBCB2 1 (1D 1H) CDCl3 360MHz
27a,b,c,d
Parameters
NBCB2 1 (1D 1H)
F1 :
- Size : 16384 points complex
- Spectral Width : 7183.908 Hz
- Carrier Frequency : 360.11252 MHz
- Nucleus : 1H
Number of scans : 16
Type : 1D
Spectro : BRUKER 360 MHz
Probe : 5 mm BBO BB-1H/D Z-GRD Z561
Date : Wed Nov 29 19:04:40 CET 2006
Temperature : 302.4 K
Solvent : CDCl3
5.0
0.9
3.2
0.9
0.8
1.6
3.9
10.5
3.0
2.6
ppm


NBCB2 2 (1D 13C) CDCl3 360MHz
27a,b,c,d
Parameters
NBCB2 2 (1D 13C)
F1 :
- Size : 16384 points complex
- Spectral Width : 21645.021 Hz
- Carrier Frequency : 90.55869 MHz
- Nucleus : 13C
Number of scans : 19968
Type : 1D
Spectro : BRUKER 360 MHz
Probe : 5 mm BBO BB-1H/D Z-GRD Z561
Date : Thu Nov 30 08:40:21 CET 2006
Temperature : 304.0 K
Solvent : CDCl3
ppm