Supporting Information

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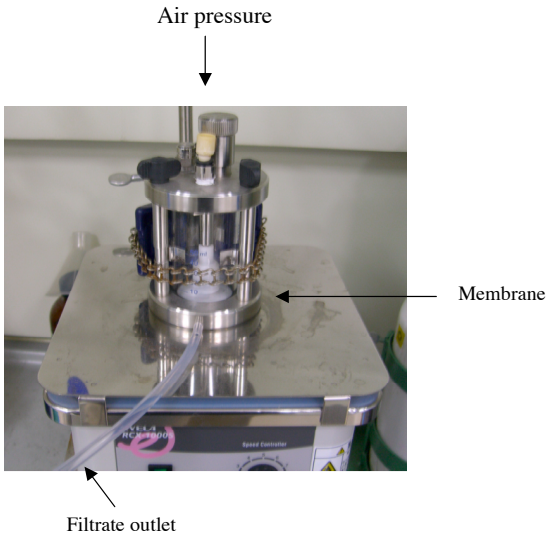
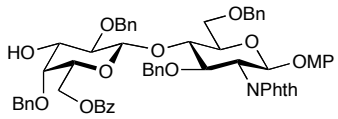
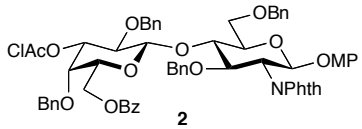
General Procedures. All chemicals were reagent grade and were used as supplied unless otherwise noted. Dry dichloromethane, THF, DMF, and toluene were purchased form Kanto Chemical Co. Technical grade or reagent grade solvents for extraction and chromatography were used without further purification. Molecular sieves AW 300 was purchased from Aldrich and activated by heating at 170 °C *in vacuo* just before use. Silica gel 60 F_{254} plates (E. Merck) were used for analytical and preparative thin-layer chromatography. Silica gel 60N (spherical, neutral, Kanto Chemical Co., Inc, Tokyo) was used for flash column chromatography (40-100 μm) and open column (100-200 μm) chromatography. ^{1}H and ^{13}C NMR spectra were recorded at ambient temperature (23~24 °C) in $CDCl_3$ using JEOL EX 400 MHz spectrometer. ^{13}C-NMR spectra were taken in $CDCl_3$ unless otherwise mentioned, and $CHCl_3$ (δ 77.0 ppm) was used as an internal standard. Chemical shifts are reported in ppm relative to internal tetramethylsilane (δ = 0.00 ppm or $CHCl_3$ as 7.26 ppm) for ^{1}H and internal $CDCl_3$ (δ = 77.00 ppm) for ^{13}C NMR spectra. Optical rotations were measured with a JASCO DIP-310 polarimeter. MALDI-TOF MS spectra were measured by Shimadzu AXIMA-CFR using DHBA and CHCA as matrix. Melting points (not corrected) were measured with a YANACO micro melting point apparatus. Ultrafiltration stirred cell was purchased from Millipore and the picture was shown below. Amicon Ultracell PL Membrane Disk (Molecular cut-off 1000) was purchased from Millipore and was used for ultrafiltration.





***p*-Methoxyphenyl *O*-(6-*O*-benzoyl-2,4-di-*O*-benzyl-β-D-galactopyranosyl)-(1→4)-*O*-3,6-di-*O*-benzyl-2-deoxy-2-phthalimido-β-D-glucopyranoside**

To a solution of the diol **1** (200.0 mg, 0.213 mmol), pyridine (36 μL, 0.426 mmol) in 1,2-dichloroethane (3 mL) was added benzoyl chloride (38 μL, 0.330 mmol) at 0 °C under N_2 atmosphere. The mixture was stirred for 7 h at 0 °C, then the reaction mixture was quenched with 1 M HCl aq. The aqueous layer was extracted with EtOAc. The combined organic layers were washed with sat. $NaHCO_3$, H_2O and brine. After drying the extract over $MgSO_4$, the solvent was evaporated. The residue was purified by silica gel column chromatography (hexane:EtOAc 3:1 to 2:1) to give 197.0 mg (89%) of the product. $[\alpha]_D$ + 62.5 (*c* 1.0, $CHCl_3$); ^{1}H-NMR ($CDCl_3$) δ 8.04-7.98 (2H, m, Ar-H), 7.86-7.56 (4H, m, Ar-H), 7.54-7,48 (2H, m, Ar-H), 7.42-7.15 (16H, m, Ar-H), 7.03-6.96 (2H, m, Ar-H), 6.86-6.77 (5H, m, Ar-H), 6.68-6.63 (2H, m, Ar-H), 5.61 (1H, d, *J* = 8.1 Hz, H-1), 4.92 (1H, d, *J* = 12.2 Hz, benzyl), 4.91 (1H, d, *J* = 11.2 Hz, benzyl), 4.81 (1H, d, *J* = 11.7 Hz, benzyl), 4.70 (1H, d, *J* = 11.2 Hz, benzyl), 4.62 (1H, d, *J* = 11.7 Hz, benzyl), 4.57 (1H, d, *J* = 12.2 Hz, benzyl), 4.53 (1H, d, *J* = 13.2 Hz, benzyl), 4.46 (1H, d, *J* = 7.3 Hz, H-1b), 4.49-4.38 (4H, m), 4.35 (1H, dd, *J* = 11.0, 6.4 Hz, H-6b), 4.25 (1H, dd, *J* = 11.0, 6.8 Hz, H-6b), 4.11 (1H, d, *J* = 9.5, 7.1 Hz, H-4a), 3.89-3.78 (3H, m), 3.74-3.66 (1H, m, H-5a), 3.68 (3H, s, OMe), 3.64-3.53 (3H, m), 2.24 (1H, d, *J* = 4.7 Hz, OH); ^{13}C-NMR ($CDCl_3$) δ 165.9 (C), 155.0 (C), 150.7 (C), 138.4 (C), 138.0 (C), 133.6 (CH), 133.0 (CH), 129.6 (CH), 129.6 (CH), 128.4 (CH), 128.4 (CH), 128.2 (CH), 128.1 (CH), 127.9 (CH), 127.8 (CH), 127.7 (CH), 127.6 (CH), 127.6 (CH), 127.4 (CH), 127.3 (CH), 126.8 (CH), 123.1 (CH), 118.5 (CH), 114.2 (CH), 103.2 (CH), 97.5 (CH), 80.1 (CH), 78.7 (CH), 76.9 (CH), 75.5 (CH), 75.4 (CH), 75.2 (CH_2), 75.0 (CH_2), 74.5 (CH_2), 74.2 (CH), 73.1 (CH_2), 72.4 (CH), 67.9 (CH_2), 62.7 (CH_2), 55.6 (CH_3).

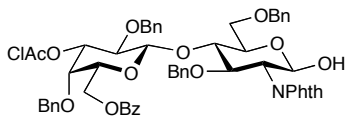


2

***p*-Methoxyphenyl *O*-(6-*O*-benzoyl-2,4-di-*O*-benzyl-3-chloroacetyl-β-D-galactopyranosyl)-(1→4)-*O*-3,6-di-*O*-benzyl-2-deoxy-2-phthalimido-β-D-**

glucopyranoside 2

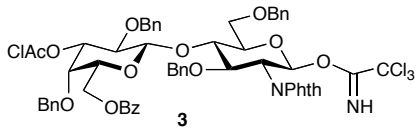
To a solution of the mono-alcohol (1.037 g, 0.995 mmol), pyridine (0.40 mL, 4.98 mmol) in 1,2-dichloroethane (15 mL) was added chloroacetic anhydride (255.2 mg, 1.49 mmol) at 0 °C under N_2 atmosphere. The mixture was stirred for 1.5 h at room temperature. The reaction mixture was quenched with MeOH and stirred for 0.5 h. After dilution of the mixture with EtOAc, the organic layer was washed with aqueous 2 M HCl, H_2O, and brine. After drying the extract over $MgSO_4$, the solvent was evaporated. The residue was purified by silica gel column chromatography (toluene:EtOAc 15:1 to 10:1) to give 1.10 g (99%) of compound **2**. $[\alpha]_D$ + 61.9 (*c* 1.0, $CHCl_3$); ^{1}H-NMR ($CDCl_3$) δ 8.04-7.98 (2H, m, Ar-H), 7.88-7.58 (4H, m, Ar-H), 7.56-7.48 (2H, m, Ar-H), 7.42-7.15 (16H, m, Ar-H), 7.04-6.98 (2H, m, Ar-H), 6.87-6.79 (5H, m, Ar-H), 6.71-6.65 (2H, m, Ar-H), 5.62 (1H, d, *J* = 8.1 Hz, H-1a), 4.91 (1H, d, *J* = 12.2 Hz, benzyl), 4.87 (1H, dd, *J* = 10.2, 3.2 Hz, H-3b), 4.82 (1H, d, *J* = 11.7 Hz, benzyl), 4.65 (1H, d, *J* = 11.7 Hz, benzyl), 4.61 (1H, d, *J* = 12.0 Hz, benzyl), 4.58 (1H, d, *J* = 12.2 Hz, benzyl), 4.54 (1H, d, *J* = 12.0 Hz, benzyl), 4.52 (1H, d, *J* = 12.2 Hz, benzyl), 4.52 (1H, d, *J* = 7.6 Hz, H-1b), 4.45 (1H, dd, *J* = 11.0, 8.1 Hz, H-2a), 4.40 (1H, d, *J* = 12.2 Hz, benzyl), 4.39 (1H, dd, *J* = 11.0, 8.1 Hz, H-3a), 4.32 (1H, dd, *J* = 11.0, 7.1 Hz, H-6b), 4.24 (1H, dd, *J* = 11.0, 6.6 Hz, H-6b), 4.15 (1H, dd, *J* = 9.8, 8.1 Hz, H-4a), 3.89 (1H, d, *J* = 3.2 Hz, H-4b), 3.86 (1H, dd, *J* = 11.0, 4.1 Hz, H-6a), 3.81 (1H, d, *J* = 10.2, 7.6 Hz, H-2b), 3.82-3.75 (1H, m, H-6a), 3.75 (1H, d, *J* = 14.9 Hz, one of $ClCH_2$), 3.70 (3H, s, OMe), 3.70 (1H, d, *J* = 14.9 Hz, one of $ClCH_2$), 3.75-3.64 (1H, m, H-5a), 3.59 (1H, dd, *J* = 7.1, 6.6 Hz, H-5b); ^{13}C-NMR ($CDCl_3$) δ 166.4 (C), 165.7 (C), 155.0 (C), 150.7 (C), 138.4 (C), 137.9 (C), 137.4 (C), 133.6 (CH), 133.1 (CH), 131.4 (C), 129.6 (CH), 128.5 (CH), 128.4 (CH), 128.3 (CH), 128.2 (CH), 128.2 (CH), 127.8 (CH), 127.7 (CH), 127.7 (CH), 127.6 (CH), 127.6 (CH), 127.4 (CH), 126.8 (CH), 123.1 (CH), 118.5 (CH), 114.2 (CH), 103.0 (CH), 97.6 (CH), 78.6 (CH), 76.9 (CH), 76.8 (CH), 75.2 (CH), 75.2 (CH_2), 75.1 (CH_2), 74.5 (CH_2), 74.2 (CH), 73.1 (CH_2), 71.8 (CH), 67.7 (CH_2), 62.2 (CH_2), 55.6 (CH_3), 40.5 (CH_2).



***O*-(6-*O*-Benzoyl-2,4-di-*O*-benzyl-3-chloroacetyl-β-D-galactopyranosyl)-(1→4)-*O*-**

3,6-di-*O*-benzyl-2-deoxy-2-phthalimido-D-glucopyranose

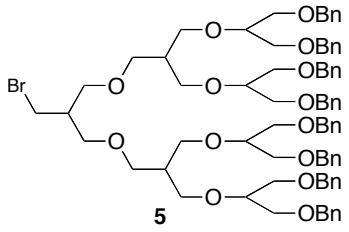
To a solution of the *p*-methoxyphenyl ether (78.4 mg, 0.070 mmol) in toluene:acetonitrile:water (1:1.3:1, 10 mL) was added ceric ammonium nitrate (808.8 mg, 1.40 mmol) at room temperature. After the mixture was stirred vigorously for 15 min, the reaction mixture was diluted with EtOAc. The aqueous layer was extracted with EtOAc. The combined organic layers were washed with H_2O and brine. After drying the extract over $MgSO_4$, the solvent was evaporated. The residue was purified by silica gel column chromatography (toluene:EtOAc 5:1 to 4:1) to give 63.9 mg (90%) of the hemiacetal (α:β = 1:4). $[\alpha]_D$ + 60.5 (*c* 1.1, $CHCl_3$); β-isomer: ^{1}H-NMR ($CDCl_3$) δ 8.00 (2H, d, *J* = 7.3 Hz, Ar-H), 7.83-7.57 (5H, m, Ar-H), 7.53-7.47 (2H, m, Ar-H), 7.42-7.15 (15H, m, Ar-H), 7.04-6.97 (2H, m, Ar-H), 6.85-6.79 (3H, m, Ar-H), 5.31 (1H, d, *J* = 8.5 Hz, H-1a), 4.91 (1H, d, *J* = 12.2 Hz, benzyl), 4.82 (1H, dd, *J* = 10.0, 3.2 Hz, H-3b), 4.78 (1H, d, *J* = 11.7 Hz, benzyl), 4.64 (1H, d, *J* = 12.2 Hz, benzyl), 4.60 (1H, d, *J* = 11.7 Hz, benzyl), 4.57 (2H, d, *J* = 15.6 Hz, benzyl), 4.49 (1H, d, *J* = 12.0 Hz, benzyl), 4.43 (1H, d, *J* = 7.8 Hz, H-1b), 4.41 (1H, dd, *J* = 10.5, 8.5 Hz, H-2a), 4.36 (1H, d, *J* = 12.0 Hz, benzyl), 4.29 (1H, dd, *J* = 11.0, 6.8 Hz, H-6b), 4.23 (1H, dd, *J* = 11.0, 6.3 Hz, H-6b), 4.17-4.07 (3H, m), 3.87 (1H, d, *J* = 2.7 Hz, H-4b), 3.88-3.82 (1H, m), 3.78 (1H, d, *J* = 10.0, 7.8 Hz, H-2b), 3.82-3.75 (1H, m), 3.75 (1H, d, *J* = 14.9 Hz, one of $ClCH_2$), 3.69 (1H, d, *J* = 14.9 Hz, one of $ClCH_2$), 3.74-3.66 (1H, m), 3.62-3.57 (1H, m, H-5a), 3.53 (1H, dd, *J* = 6.8, 6.3 Hz, H-5b); ^{13}C-NMR ($CDCl_3$) δ 168.5 (C), 166.8 (C), 166.4 (C), 138.9 (C), 138.4 (C), 138.0 (C), 137.3 (C), 134.2 (CH), 133.6 (CH), 131.8 (C), 130.0 (CH), 129.9 (CH), 129.4 (C), 128.8 (CH), 128.7 (CH), 128.7 (CH), 128.6 (CH), 128.4 (CH), 128.2 (CH), 128.1 (CH), 128.10 (CH), 128.0 (CH), 127.2 (CH), 123.5 (CH), 103.8 (CH), 93.2 (CH), 82.3 (CH), 78.7 (CH), 77.5 (CH), 77.1 (CH), 75.6 (CH_2), 75.6 (CH_2), 75.4 (CH_2), 74.5 (CH), 73.6 (CH_2), 73.2 (CH), 69.4 (CH), 68.5 (CH_2), 63.1 (CH_2), 58.2 (CH_2), 40.7 (CH_2).



3

Trichloroacetimino *O*-(6-*O*-benzoyl-2,4-di-*O*-benzyl-3-chloroacetyl-β-D-galactopyranosyl)-(1→4)-*O*-3,6-di-*O*-benzyl-2-deoxy-2-phthalimido-D-glucopyranoside

To a solution of the hemiacetal (63.9 mg, 0.063 mmol) in 1,2-dichloroethane (1 mL) were added trichloroacetonitrile (44 μL, 0.442 mmol) and 1,8-diazabicyclo[5.4.0]-undec-7-ene (0.9 μL, 0.006 mmol) at 0 °C under Ar atmosphere. After the mixture was stirred at 0 °C for 1 h, the mixture was purified by silica gel column chromatography (toluene:EtOAc 10:1 to 3:1) to give 63.6 mg (88%) of imidate **3**; ^{1}H-NMR ($CDCl_3$) δ 8.53 (1H, s, NH), 8.04-7.99 (2H, m, Ar-H), 7.76-7.58 (5H, m, Ar-H), 7.52 (2H, t, *J* = 7.8 Hz, Ar-H), 7.41-7.13 (15H, m, Ar-H), 7.02-6.95 (2H, m, Ar-H), .6.87-6.79 (3H, m, Ar-H), 6.40 (1H, d, *J* = 8.5 Hz, H-1a), 4.90 (1H, d, *J* = 12.4 Hz, benzyl), 4.84 (1H, dd, *J* = 11.0, 3.2 Hz, H-3b), 4.81 (1H, d, *J* = 12.0 Hz, benzyl), 4.65 (1H, d, *J* = 12.0 Hz, benzyl), 4.64 (1H, d, *J* = 12.2 Hz, benzyl), 4.60 (1H, d, *J* = 11.7 Hz, benzyl), 4.54 (1H, d, *J* = 12.2 Hz, benzyl), 4.53 (1H, d, *J* = 12.0 Hz, benzyl), 4.51 (1H, d, *J* = 8.1 Hz, H-1b), 4.49-4.42 (2H, m), 4.42 (1H, d, *J* = 12.2 Hz, benzyl), 4.31 (1H, dd, *J* = 11.0, 6.8 Hz, H-6b), 4.24 (1H, dd, *J* = 11.0, 6.1 Hz, H-6b), 4.27-4.20 (1H, m), 3.93 (1H, dd, *J* = 11.0, 2.9 Hz, H-3b), 3.88 (1H, d, *J* = 2.9 Hz, H-4b), 3.82-3.70 (3H, m), 3.75 (1H, d, *J* = 14.9 Hz, one of $ClCH_2$), 3.69 (1H, d, *J* = 14.9 Hz, one of $ClCH_2$), 3.55 (1H, dd, *J* = 6.8, 6.1 Hz, H-5b); ^{13}C-NMR ($CDCl_3$) δ 166.6 (C), 165.8 (C), 160.8 (C), 138.5 (C), 138.0 (C), 137.8 (C), 137.6 (C), 133.7 (CH), 133.2 (CH), 131.4 (C), 129.7 (CH), 129.6 (CH), 128.5 (CH), 128.4 (CH), 128.3 (CH), 127.9 (CH), 127.9 (CH), 127.8 (CH), 127.8 (CH), 127.7 (CH), 127.7 (CH), 126.9 (CH), 123.2 (CH), 102.9 (CH), 94.1 (CH), 90.4 (C), 78.0 (CH), 77.3 (CH), 76.8 (CH), 76.6 (CH), 75.9 (CH), 75.2 (CH_2), 75.1 (CH_2), 74.5 (CH_2), 74.3 (CH), 73.2 (CH_2), 71.9 (CH), 67.2 (CH_2), 62.3 (CH_2), 54.6 (CH), 40.5 (CH_2).

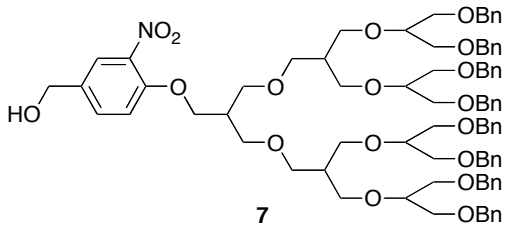


5

3-(3-[1,3-bis(benzyloxy)-2-propoxy]-2-{[1,3-bis(benzyloxy)-2-propoxy]methyl}propoxy)-2-(3-[1,3-bis(benzyloxy)-2-propoxy]-2-{[1,3-bis(benzyloxy)-2-propoxy]methyl}propoxy)propyl bromide 5

To a solution of alcohol **4** (1.00 g, 0.770 mmol) and triphenylphosphine (424.0 mg, 1.62 mmol) in CH_2Cl_2 (10 mL) was added *N*-bromosuccinimide (287.7 mg, 1.62 mmol) at 0 °C under Ar atmosphere. The reaction mixture was stirred at room

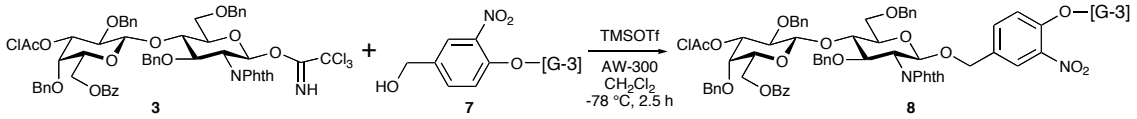
temperature for 2.5 h. The reaction mixture was poured into EtOAc and H_2O. The aqueous layer was extracted with EtOAc. The combined organic layers were washed with H_2O and brine. After drying the extract over $MgSO_4$, the solvent was evaporated. The residue was purified by silica gel column chromatography (toluene:EtOAc 15:1 to 10:1 to 5:1) to give **5** (1.033 g, 98%); ^{1}H-NMR ($CDCl_3$) δ 7.33-7.21 (40H, m, Ar-H), 4.49 (16H, s, benzyl), 3.66-3.39 (34H, m), 3.35 (2H, dd, *J* = 9.5, 5.1 Hz), 3.29 (2H, dd, *J* = 9.5, 6.8 Hz), 2.18-2.09 (3H, m); ^{13}C-NMR ($CDCl_3$) δ 138.1 (C), 128.1 (CH), 127.4 (CH), 127.3 (CH), 78.3 (CH), 73.2 (CH_2), 70.0 (CH_2), 69.7 (CH_2), 69.3 (CH_2), 68.6 (CH_2), 41.3 (CH), 40.9 (CH), 33.7 (CH_2).



7

4-[3-(3-[1,3-bis(benzyloxy)-2-propoxy]-2-{[1,3-bis(benzyloxy)-2-propoxy]methyl}propoxy)-2-(3-[1,3-bis(benzyloxy)-2-propoxy]-2-{[1,3-bis(benzyloxy)-2-propoxy]methyl}propoxy)propoxy]-3-nitrobenzyl alcohol 7

A mixture of bromide **5** (34.1 mg, 0.025 mmol), 4-hydroxymethyl-2-nitrophenol **6** (8.5 mg, 0.050 mmol), and Cs_2CO_3 (16.3 mg, 0.050 mmol) in *N*,*N*-dimethylformamide (0.5 mL) was heated at 80 °C for 10 h under Ar atmosphere. The reaction mixture was poured into EtOAc and H_2O, and the aqueous layer was extracted with EtOAc. The combined organic layers were washed with H_2O and brine. After drying the extract over $MgSO_4$, the solvent was evaporated. The residue was purified by PLC (toluene:EtOAc 3:1) to give 34.1 mg (94%) of aocohol **7**; ^{1}H-NMR ($CDCl_3$) δ 7.68 (1H, d, *J* = 2.2 Hz, Ar-H), 7.34-7.20 (41H, m, Ar-H), 6.93 (1H, d, *J* = 8.8 Hz, Ar-H), 4.46 (16H, s), 4.40 (2H, s), 4.07 (2H, d, *J* = 5.1 Hz), 3.60-3.37 (36H, m), 2.36-2.25 (1H, m), 2.12 (2H, quint, *J* = 5.9 Hz); ^{13}C-NMR ($CDCl_3$) δ 151.7 (C), 139.1 (C), 138.1 (C), 133.1 (C), 132.5 (CH), 128.1 (CH), 127.4 (CH), 127.4 (CH), 123.9 (CH), 114.4 (CH), 78.3 (CH), 73.2 (CH_2), 69.9 (CH_2), 69.4 (CH_2), 68.8 (CH_2), 68.6 (CH_2), 67.3 (CH_2), 63.6 (CH_2), 40.9 (CH), 39.8 (CH); Anal. Calcd for $C_{87}H_{103}NO_{18}$: C 72.03, H 7.16, N 0.97. Found: C 72.00, H 7.22, N 0.94.



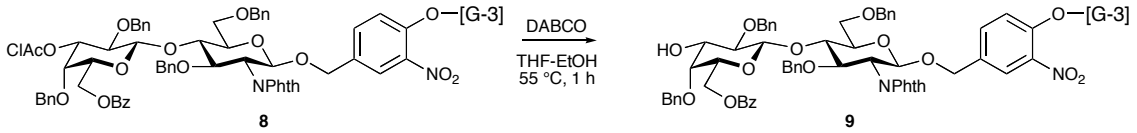
Synthesis of dendrimer-bound poly(lactosamine)

Glycosylation reaction

To a mixture of alcohol **7** (80.1 mg, 0.055 mmol), imidate **3** (76.6 mg, 0.066 mmol) and molecular sieves AW-300 (138 mg) in CH_2Cl_2 (1 mL) was added dropwise a solution of TMSOTf in CH_2Cl_2 (0.10 mL, 0.009 mmol) at -78 °C under Ar atmosphere. The mixture was stirred at -78 °C for 2.5 h. After removing molecular sieves by filtration, the filtrate was diluted with CH_3CN (12.5 ml) and transferred to a Millipore stirred cell (with Amicon® Ultracell PL Membrane Disk, Molecular Weight Cut-Offs = 1,000). The solution was filtered under 0.4 MPa pressure of nitrogen within 30 min. To the residue, CH_3CN was added and filtrated twice under 0.4 MPa pressure of nitrogen to give 107.9 mg (80%) of disaccharide **8**.

Capping reaction

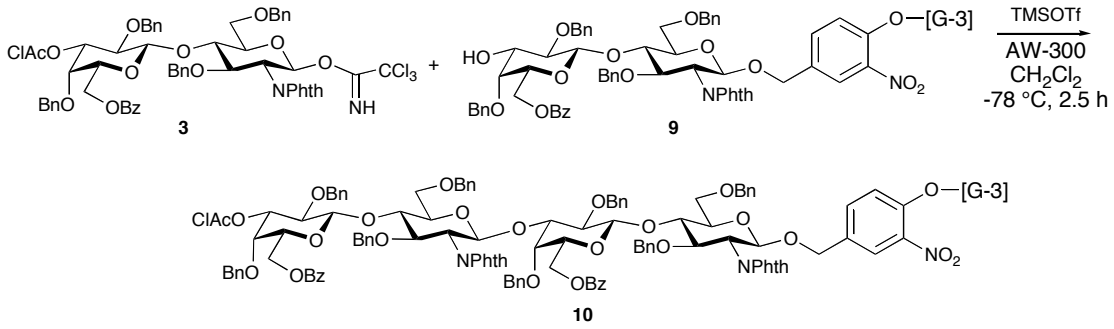
To a solution of the dendrimer-bound lactosamine (107.9 mg, 0.044 mmol) in 1,2-dichloroethane (0.5 mL) was added ethyl isocyanatoformate (5.1 μL, 0.044 mmol) at room temperature under Ar atmosphere. The mixture was stirred at room temperature for 1 h. The mixture was transferred to a Millipore stirred cell (with Amicon® Ultracell PL Membrane Disk, MWCO = 1,000) with CH_3CN (10 mL). The solution was filtered under 0.4 MPa pressure of nitrogen within 15 min to give 104.1 mg (96%) of the product.



Deprotection of chloroacetyl ester

A mixture of the chloroacetyl ester (104.0 mg, 0.043 mmol) and DABCO (71.6 mg, 0.638 mmol) in THF (0.4 mL) and EtOH (1 mL) was stirred at 55 °C for 1 h. The mixture was stirred at room temperature for 1 h. The mixture was transferred to a Millipore stirred cell (with Amicon® Ultracell PL Membrane Disk, MWCO = 1,000) with CH_3CN (10 mL). The solution was filtered under 0.4 MPa pressure of nitrogen

within 60 min to give 104.1 mg (96%) of the product. The residue was diluted with CH_3CN (10 mL) and filtrated within 25 min to give 97.2 mg (97%) of alcohol **9**.

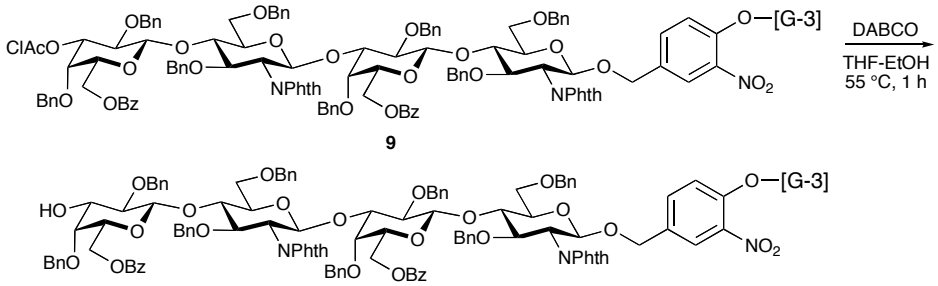


Synthesis of dendrimer-bound tetrasaccharide 10

To a mixture of the alcohol **9** (97.2 mg, 0.041 mmol), imidate **3** (61.7 mg, 0.053 mmol) and molecular sieves AW-300 (138 mg) in CH_2Cl_2 (1 mL) was added dropwise a solution of TMSOTf in CH_2Cl_2 (0.10 mL, 0.006 mmol) at -78 °C under Ar atmosphere. The mixture was stirred at -78 °C for 4 h. After removing molecular sieves by filtration, the filtrate was diluted with CH_3CN (12.5 ml) and transferred to a Millipore stirred cell (with Amicon® Ultracell PL Membrane Disk, Molecular Weight Cut-Offs = 1,000). The solution was filtered under 0.4 MPa pressure of nitrogen within 25 min. To the residue, CH_3CN (12 mL) was added and filtrated twice under 0.4 MPa pressure of nitrogen to give 137.9 mg (quant.) of the tetrasaccharide **10**.

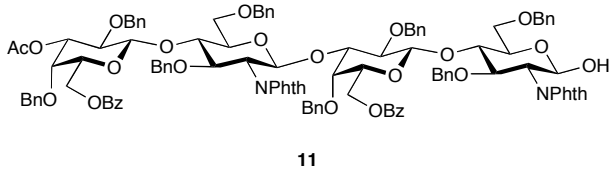
The capping reaction after glycosylation

To a solution of the dendrimer-bound lactosamine (137.9 mg, 0.041 mmol) in 1,2-dichloroethane (0.5 mL) was added ethyl isocyanatoformate (4.2 μL, 0.041 mmol) at room temperature under Ar atmosphere. The mixture was stirred at room temperature for 1 h. The mixture was transfered to a Millipore stirred cell (with Amicon® Ultracell PL Membrane Disk, MWCO = 1,000) with CH_3CN (10 mL). The solution was filtered under 0.4 MPa pressure of nitrogen to within 20 min to give 135.9 mg (99%) of the product.



Deprotection of chloroacetyl ester

A mixture of the chloroacetyl ester **9** (135.9 mg, 0.040 mmol) and DABCO (22.7 mg, 0.202 mmol) in THF (0.3 mL) and EtOH (1 mL) was stirred at 55 °C for 1 h. The mixture was transferred to a Millipore stirred cell (with Amicon® Ultracell PL Membrane Disk, MWCO = 1,000) with CH_3CN (20 mL). The solution was filtered under 0.4 MPa pressure of nitrogen within 65 min. The residue was diluted with CH_3CN (10 mL) and filtrated within 25 min to give 133.2 mg (quant.) of the product.



11

A solution of dendrimer-bound tetrasaccharide **10** (53 mg, 0.0167 mmol) in CH_2Cl_2 (2 mL) and EtOH (2 mL), was added $Mo(CO_2)_6$ (44 mg, 0.167 mmol) and the whole mixture was refluxed for 6 h under Ar atmosphere. After cooling the mixture, the mixture was diluted with $CHCl_3$ and sat. $NaHCO_3$. After separation, the aqueous layer was extracted with $CHCl_3$. The combined layers were washed with brine. After drying the extract over Na_2SO_4, the solvent was evaporated. The crude aniline was dissolved in pyridine (2 mL) then Ac_2O (1 mL) was added. After overnight, the solvent was evaporated, and the residue was dried *in vacuo*. The residue was dissolved in 15% TFA in CH_2Cl_2 (3 mL). After 4h, the mixture was concentrated, co-evaporated with toluene. The residue was purified by silica gel column chromatography (hexane/EtOAc 7:3-1:1) to give 27 mg (84%) of the tetrasaccharide **11**. $[\alpha]_D$ +36 (*c* 1.0, $CHCl_3$); for major peak; ^{1}H-NMR ($CDCl_3$) δ: 7.91-7.88 (m, 3H), 7.6-6.7 (m, 55H), 5.32 (d, *J* = 8.4 Hz, 1H), 5.05 (t, *J* = 8.5 Hz, 1H), 4.94 (d, *J* = 11.7 Hz, 1H), 4.86 (d, *J* = 12.2 Hz, 1H), 4.8-4.7 (m, 15H), 4.64 (d, *J* = 11.5 Hz, 1H), 4.58 (d, *J* = 11.7 Hz, 1H), 4.47-3.30 (m, 22H), 4.32 (t, *J* = 12.0 Hz, 1H), 3.15 (d, *J* = 10.7 Hz, 1H), 3.08 (d, *J* = 10.7 Hz, 1H), 2.76 (d, *J* = 8.8 Hz,

1H), 1.92 (s, 3H); ^{13}C-NMR ($CDCl_3$) 170.4, 168.1, 166.2, 166.0, 138.9, 138.9, 138.8, 138.4, 138.2, 138.0, 133.5, 133.3, 133.2, 131.8, 130.1, 129.9, 129.8, 129.2, 128.6, 128.6, 128.5, 128.4, 128.3, 128.2, 128.0, 128.0, 127.9, 127.8, 127.7, 127.6, 127.5, 127.0, 127.0, 126.6, 123.4, 123.2, 103.4, 103.1, 100.1, 93.2, 82.2, 79.0, 78.1, 77.8, 76.7, 76.5, 75.6, 75.5, 75.3, 75.1, 75.0, 74.6, 74.5, 74.4, 73.4, 72.2, 72.1, 68.5, 67.5, 63.4, 62.5, 67/9, 56.7, 30.1, 21.4.

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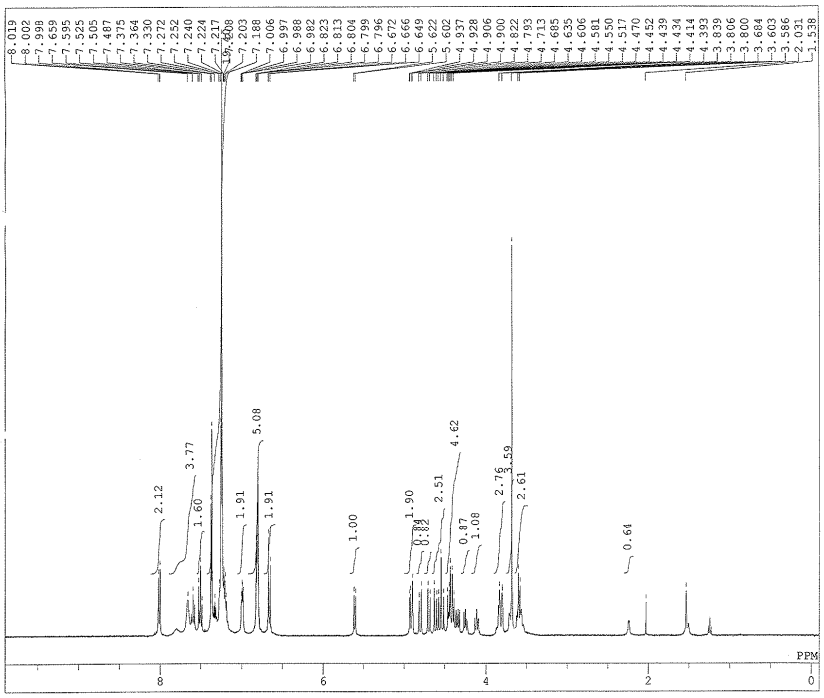


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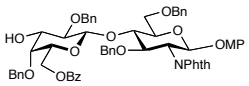

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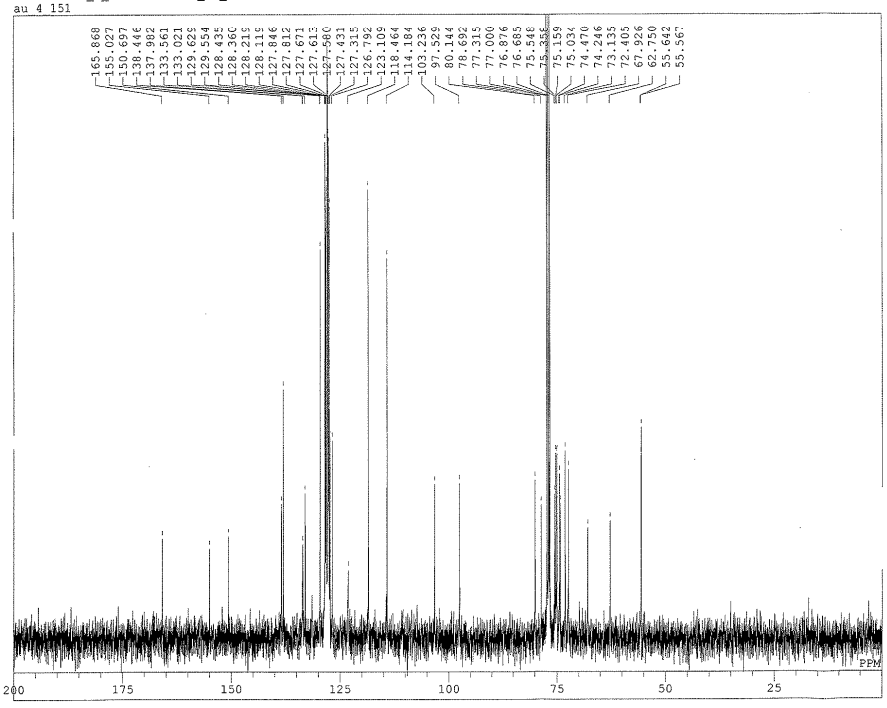


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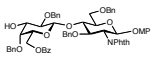

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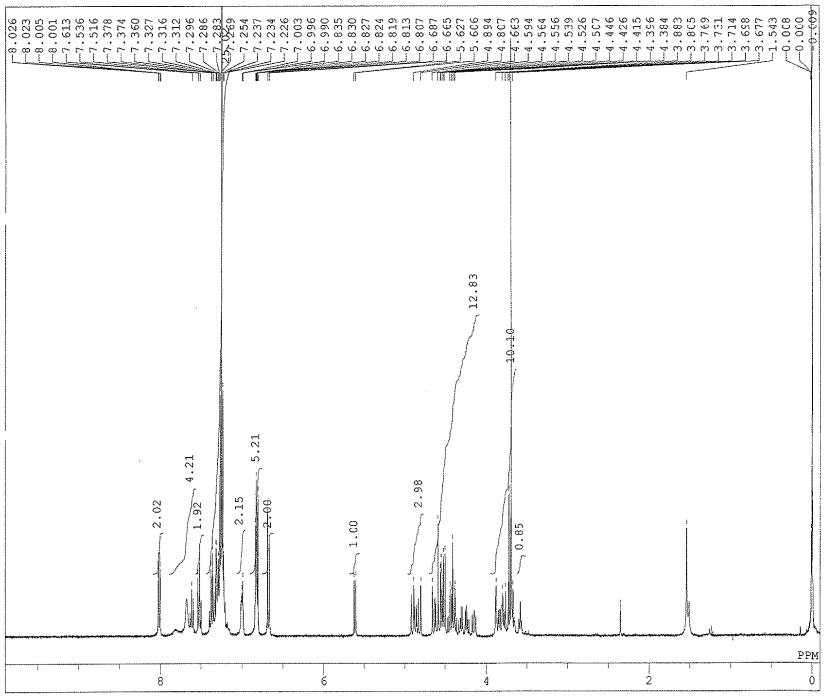


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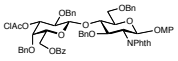

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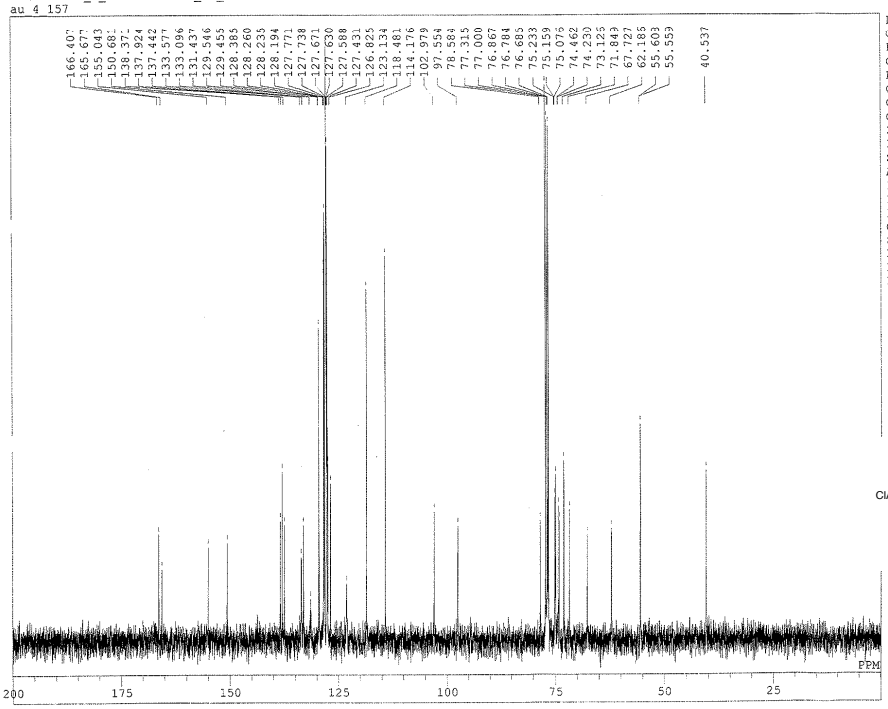


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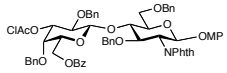

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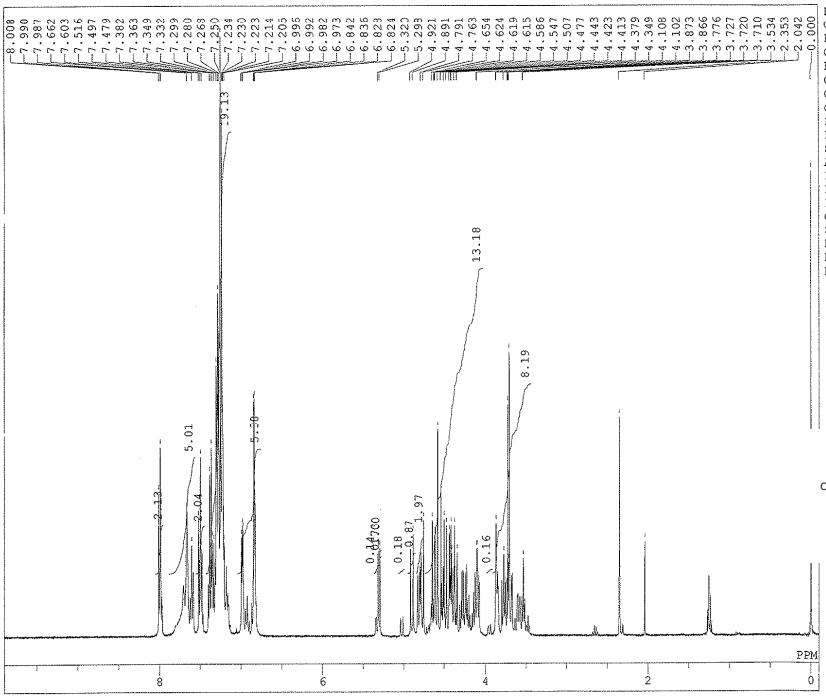


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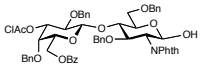

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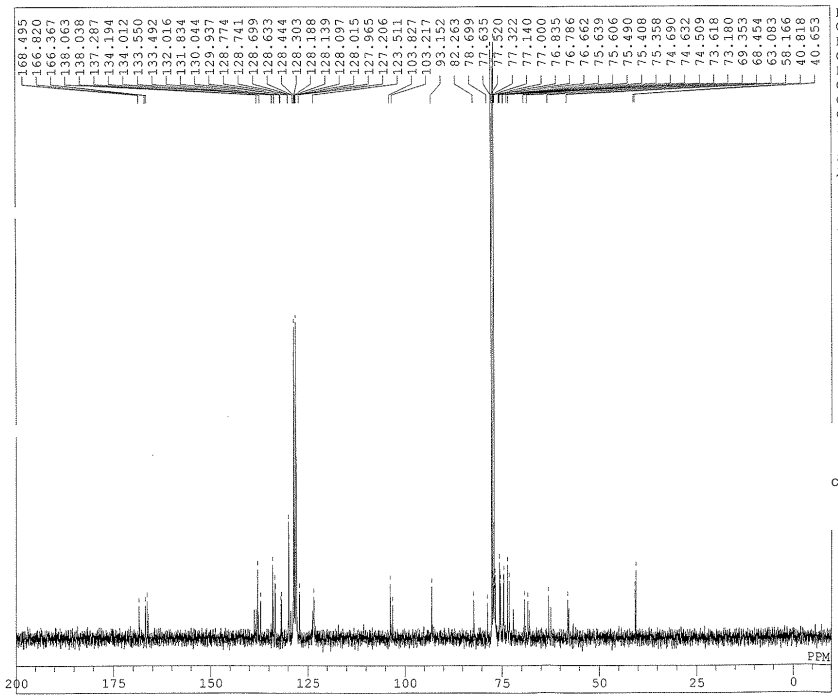


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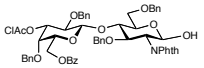

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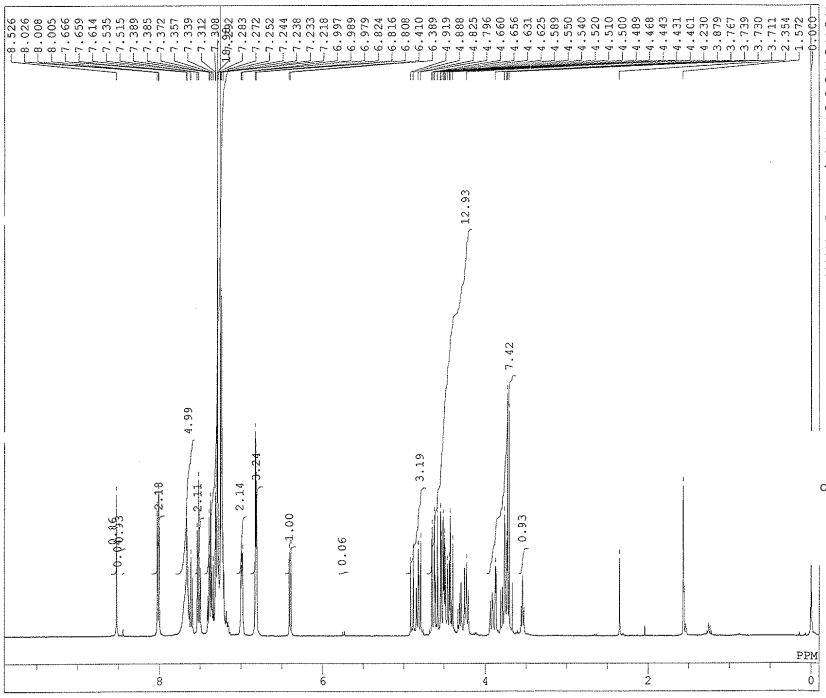

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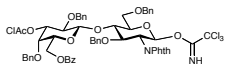

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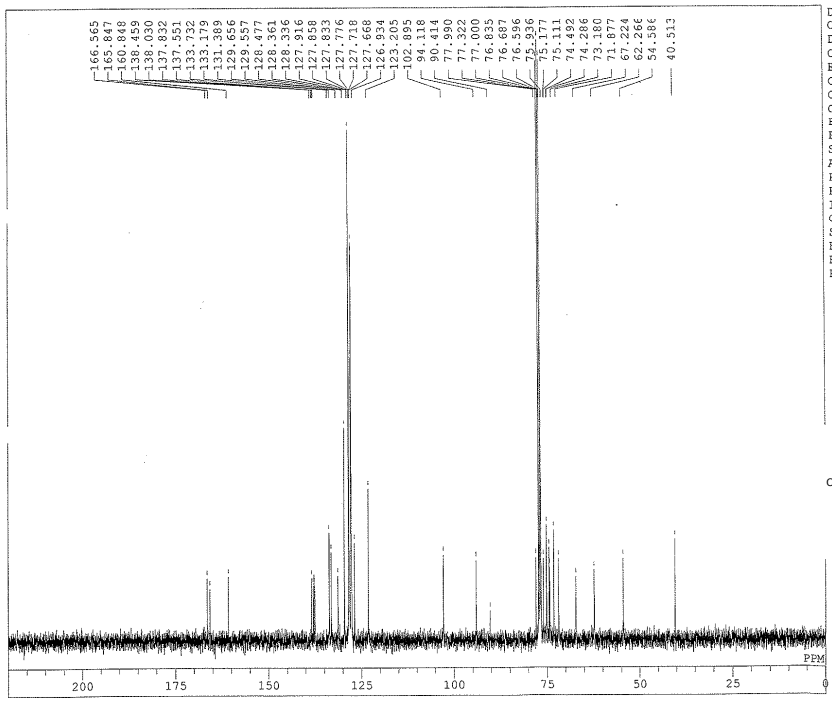

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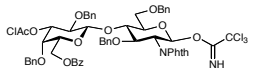

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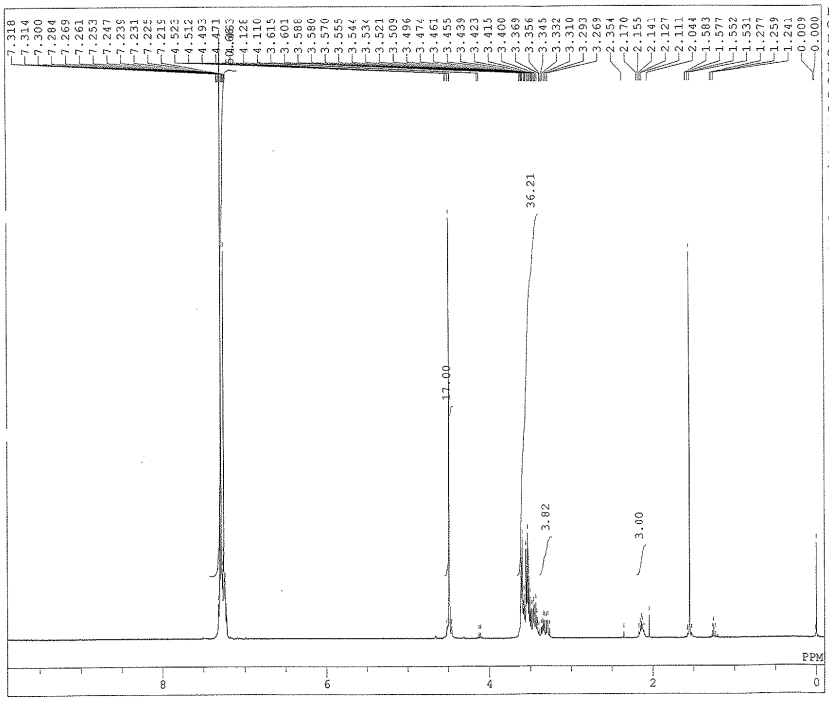


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RGAIN              20
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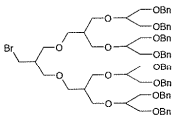

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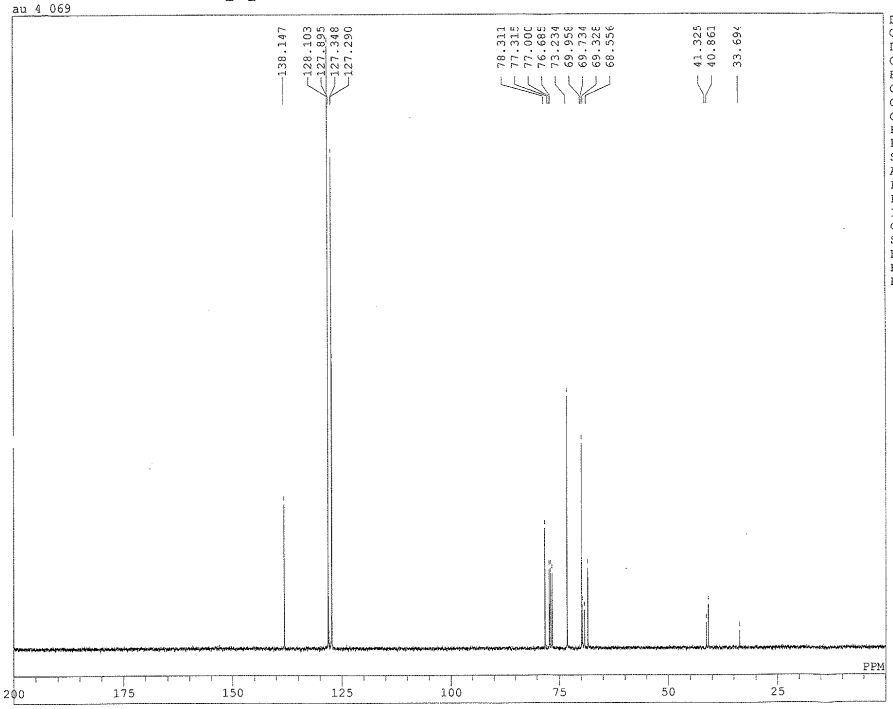


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RGAIN              25
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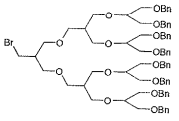

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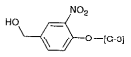

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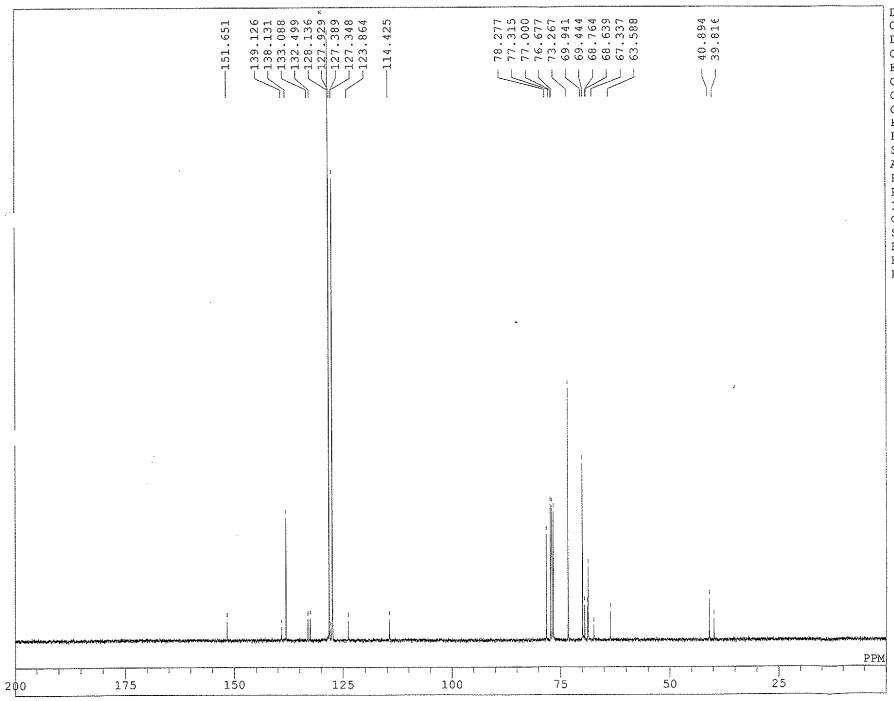


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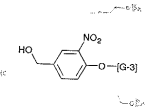

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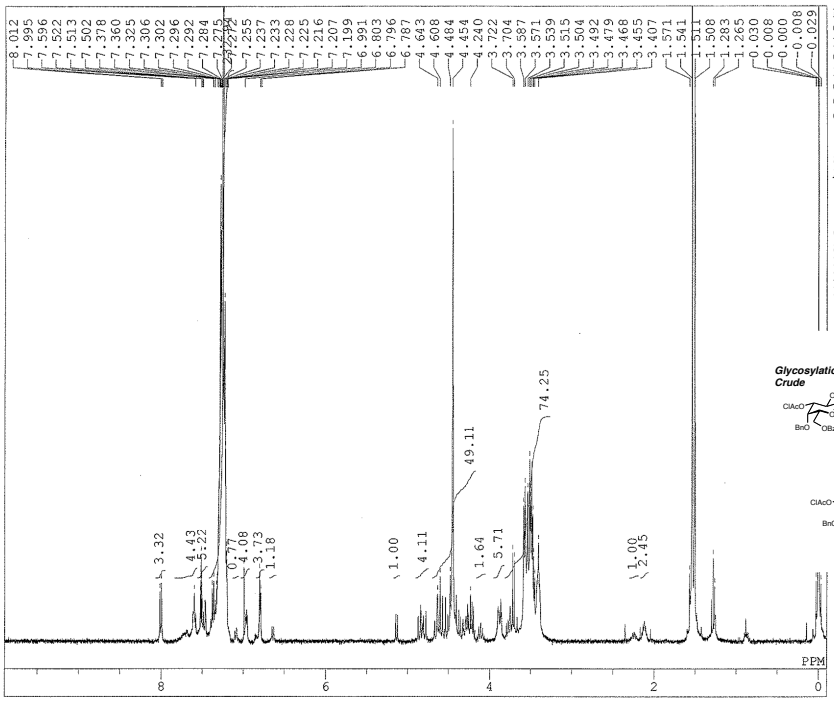

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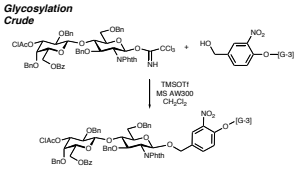
RGAIN	26

Glycosylation Crude



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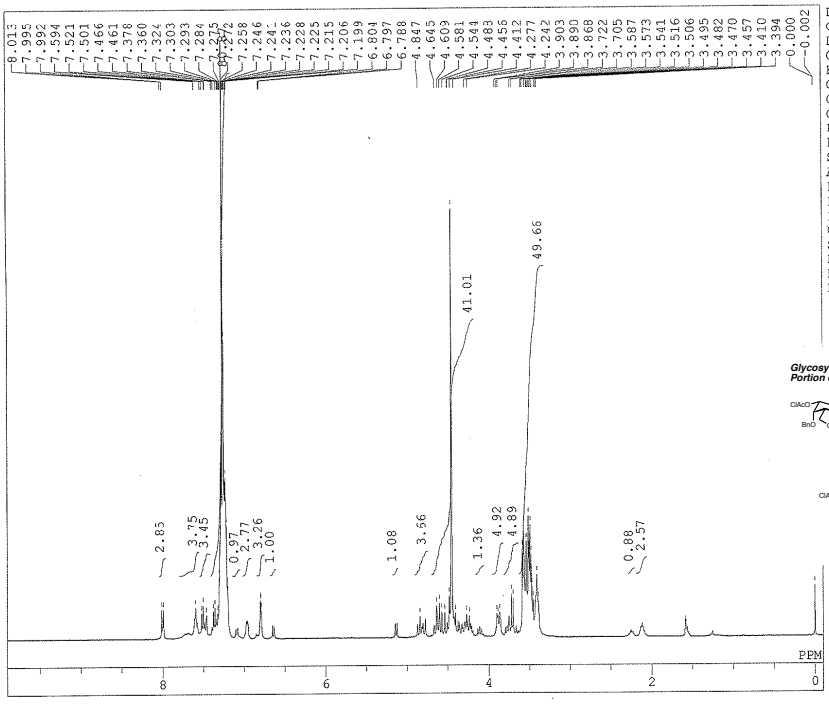

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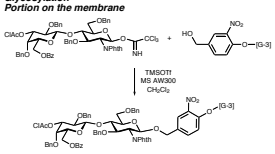
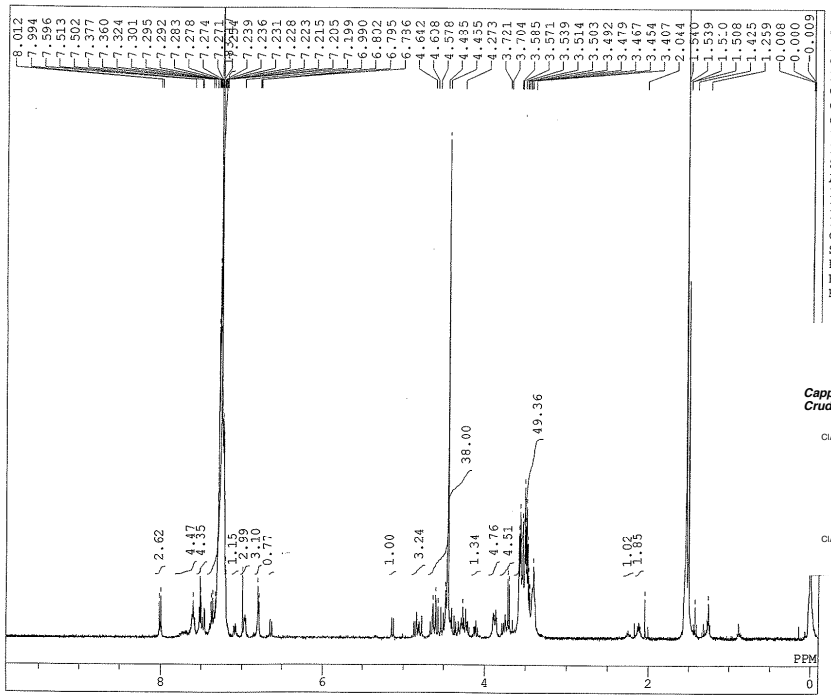
PD	2.901 sec
PW1	5.2 us
IRNUC	1H
CTEMP	24.6 c
SLVNT	CDCL3
EXREF	0.00 ppm
BF	0.12 Hz
RGAIN	14

Glycosylation Portion on the membrane

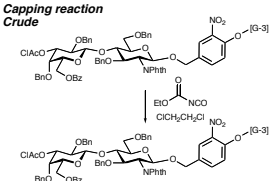


E:\Ueki\au_4_085\au_4_085_crude.ALS

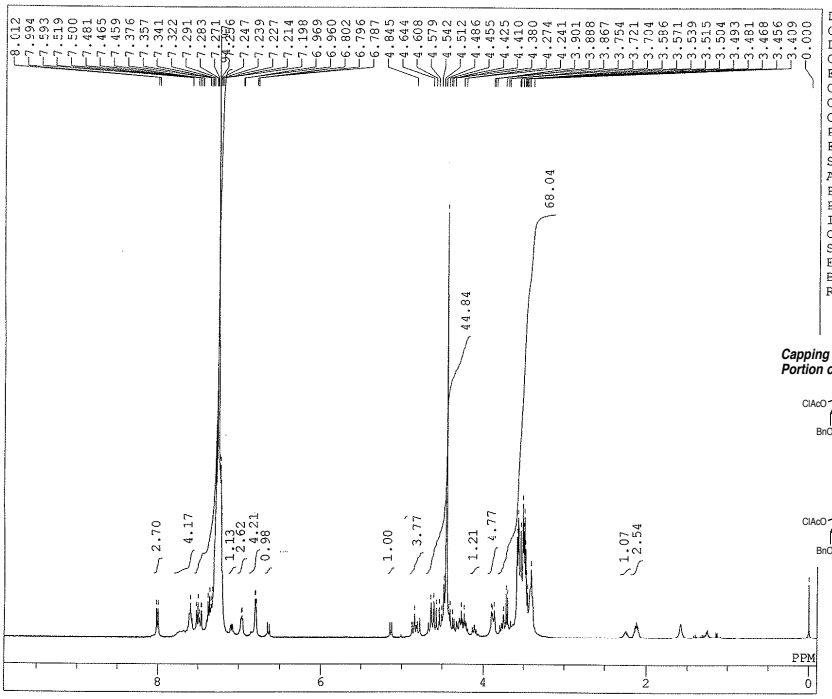


DFILE	E:\Ueki\au_4_085\au_4_085_
COMNT	
DATIM	Fri Dec 24 18:32:18 2004
OBNUC	1H
EXMOD	NON
OBFRQ	399.65 MHz
OBSET	124.00 KHz
OBFIN	10500.0 Hz
POINT	32768
FREQU	7993.6 Hz
SCANS	196
ACQTM	4.099 sec
PD	2.901 sec
PW1	5.2 us
IRNUC	1H
CTEMP	22.6 c
SLVNT	CDCL3
EXREF	0.00 ppm
BF	0.12 Hz
RGAIN	26

Capping reaction
Crude

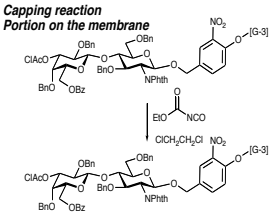


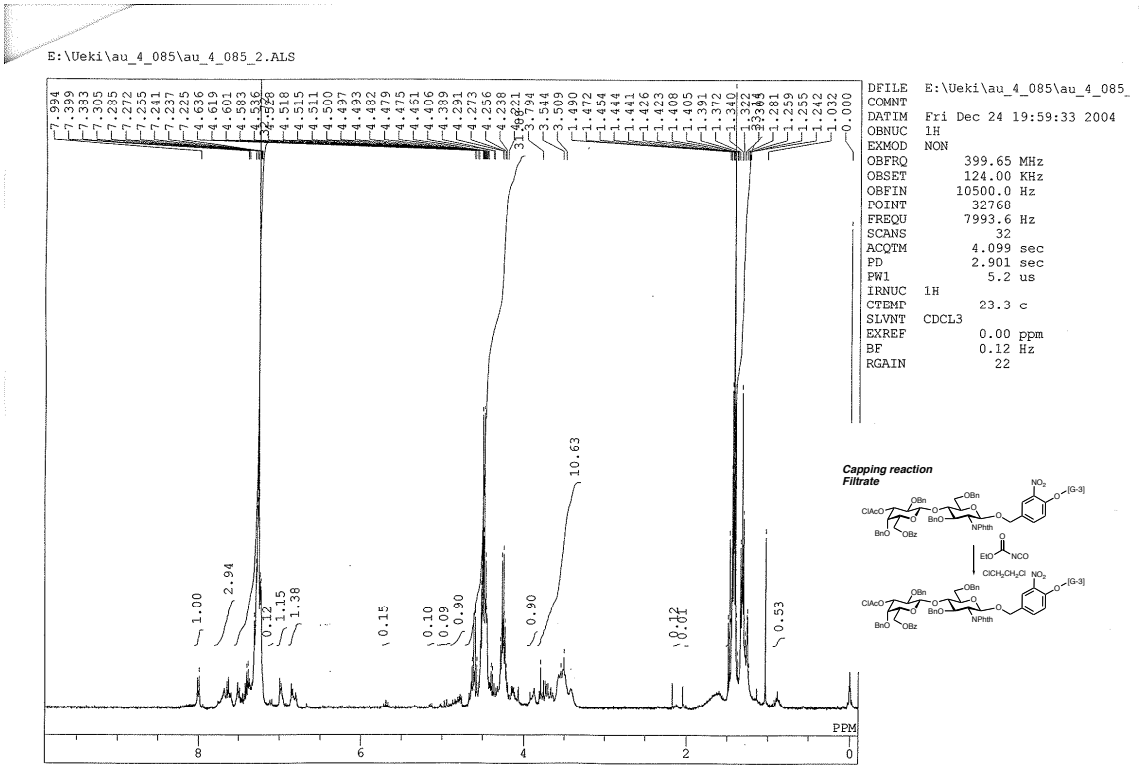
E:\Ueki\au_4_085\au_4_085_1.ALS

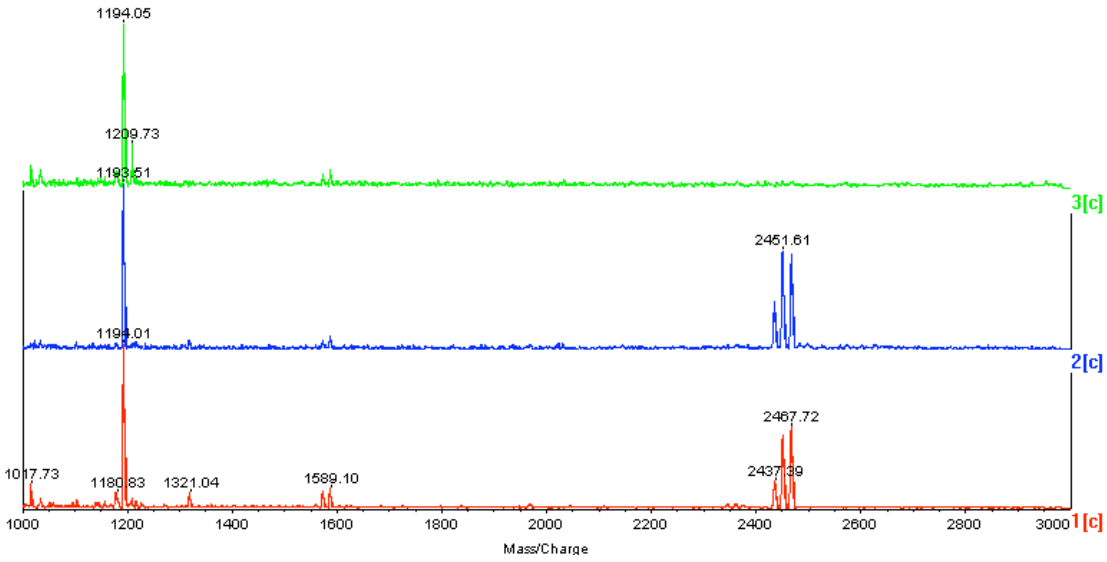


DFILE	E:\Ueki\au_4_085\au_4_085_
COMNT	
DATIM	Fri Dec 24 19:49:54 2004
OBNUC	1H
EXMOD	NON
OBFRQ	399.65 MHz
OBSET	124.00 KHz
OBFIN	10500.0 Hz
POINT	32768
FREQU	7993.6 Hz
SCANS	16
ACQTM	4.099 sec
PD	2.901 sec
PW1	5.2 us
IRNUC	1H
CTEMP	24.2 c
SLVNT	CDCL3
EXREF	0.00 ppm
BF	0.12 Hz
RGAIN	14

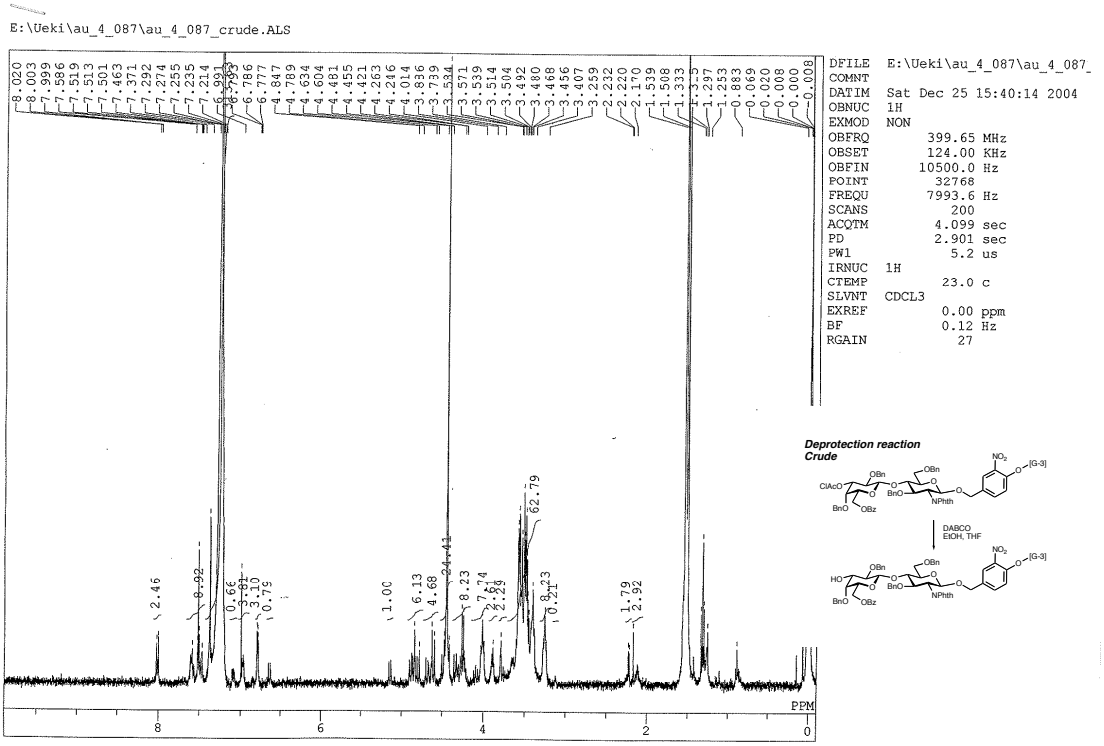
Capping reaction
Portion on the membrane

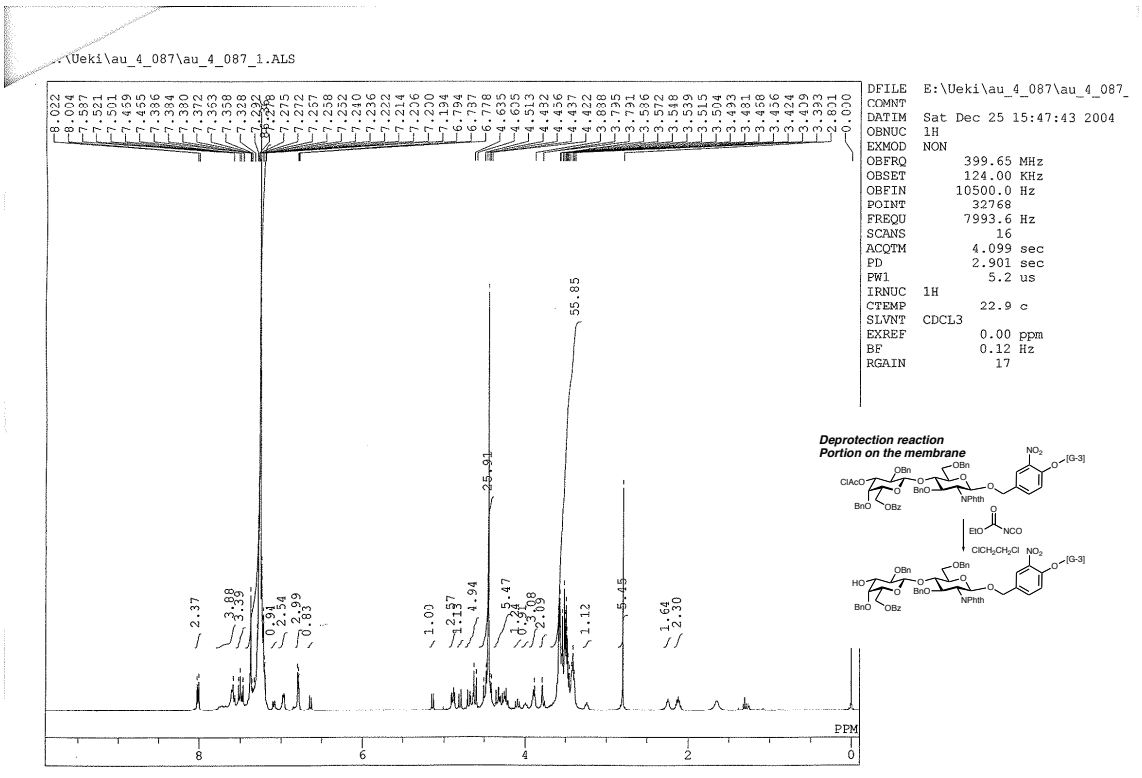


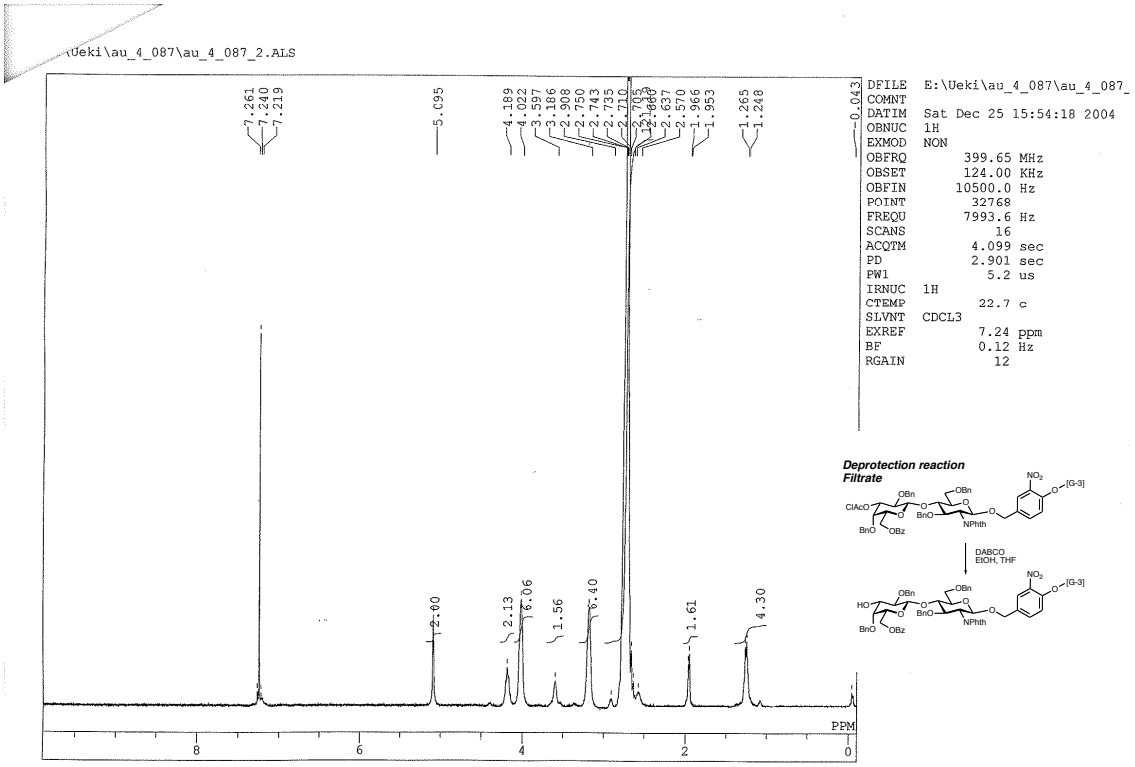


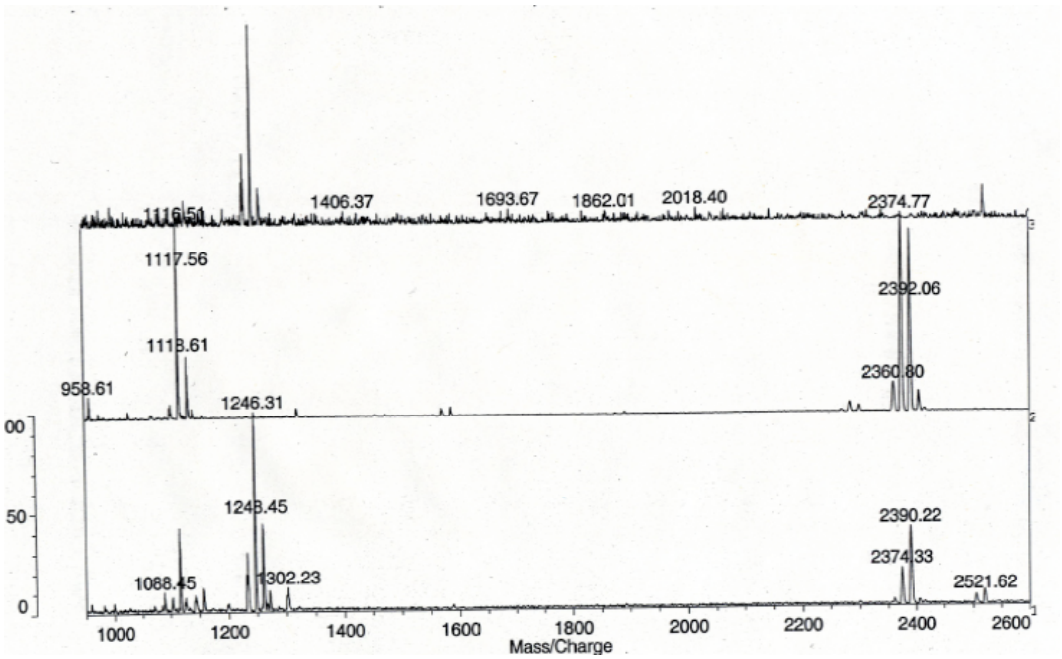


MS spectra for capping reaction; the bottom (red) spectrum is for crude mixture, the middle (blue) spectrum is for portion left on the membrane, and the upper (green) spectrum is for filtrate.

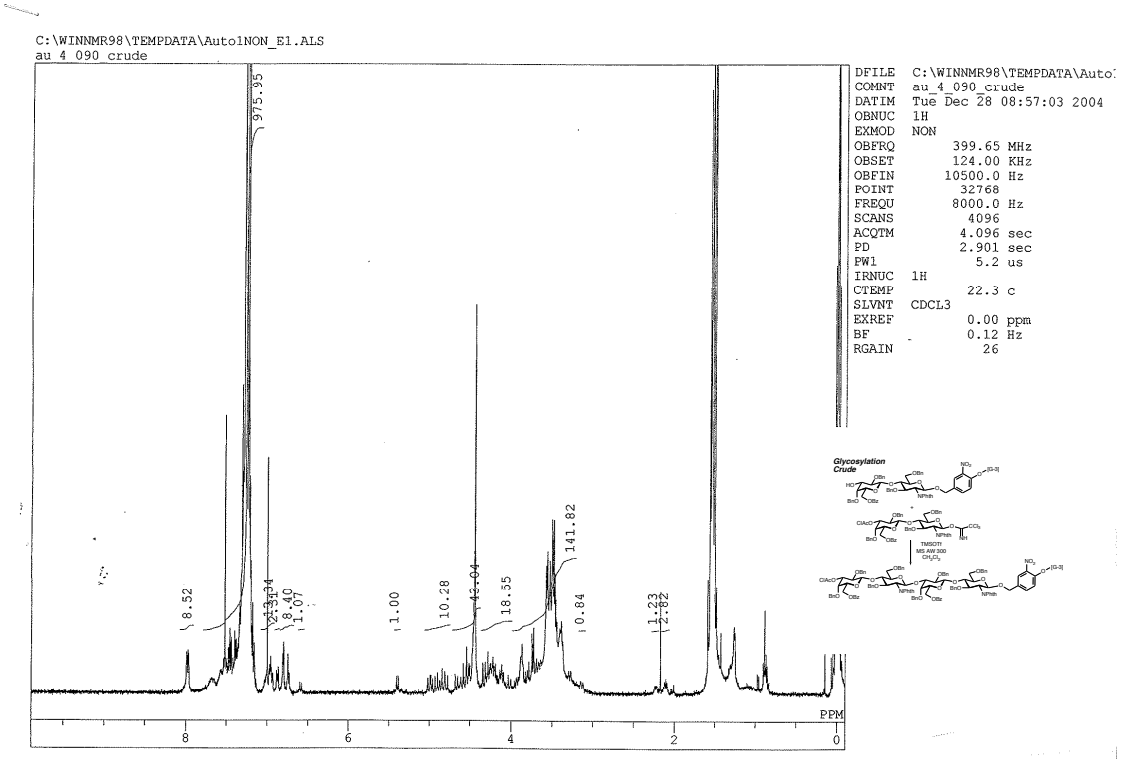

E:\Ueki\au_4_087\au_4_087_crude.ALS
DFILE E:\Ueki\au_4_087\au_4_087_
COMNT
DATIM Sat Dec 25 15:40:14 2004
OBNUC 1H
EXMOD NON
OBFRQ 399.65 MHz
OBSET 124.00 KHz
OBFIN 10500.0 Hz
POINT 32768
FREQU 7993.6 Hz
SCANS 200
ACQTM 4.099 sec
PD 2.901 sec
PW1 5.2 us
IRNUC 1H
CTEMP 23.0 c
SLVNT CDCL3
EXREF 0.00 ppm
BF 0.12 Hz
RGAIN 27
Deprotection reaction
Crude
DABCO
EtOH, THF
PPM

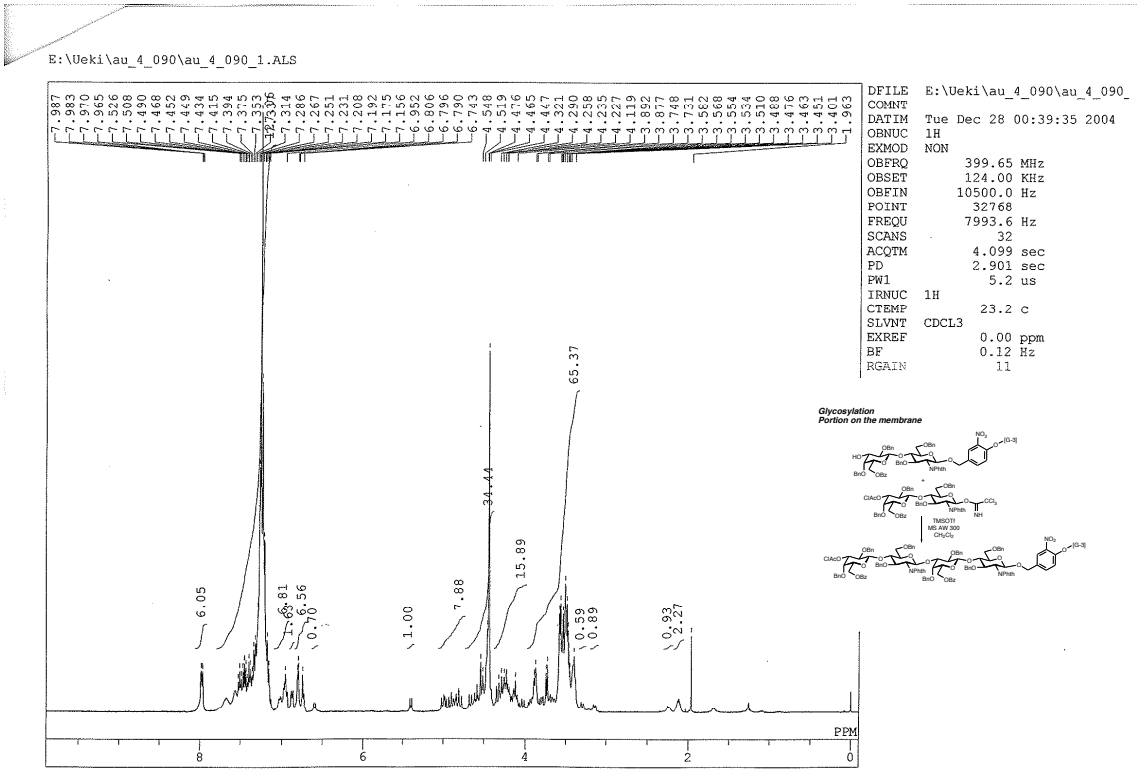

\Ueki\au_4_087\au_4_087_1.ALS
DFILE E:\Ueki\au_4_087\au_4_087_
COMNT
DATIM Sat Dec 25 15:47:43 2004
OBNUC 1H
EXMOD NON
OBFRQ 399.65 MHz
OBSET 124.00 KHz
OBFIN 10500.0 Hz
POINT 32768
FREQU 7993.6 Hz
SCANS 16
ACQTM 4.099 sec
PD 2.901 sec
PW1 5.2 us
IRNUC 1H
CTEMP 22.9 c
SLVNT CDCL3
EXREF 0.00 ppm
BF 0.12 Hz
RGAIN 17
Deprotection reaction
Portion on the membrane
PPM



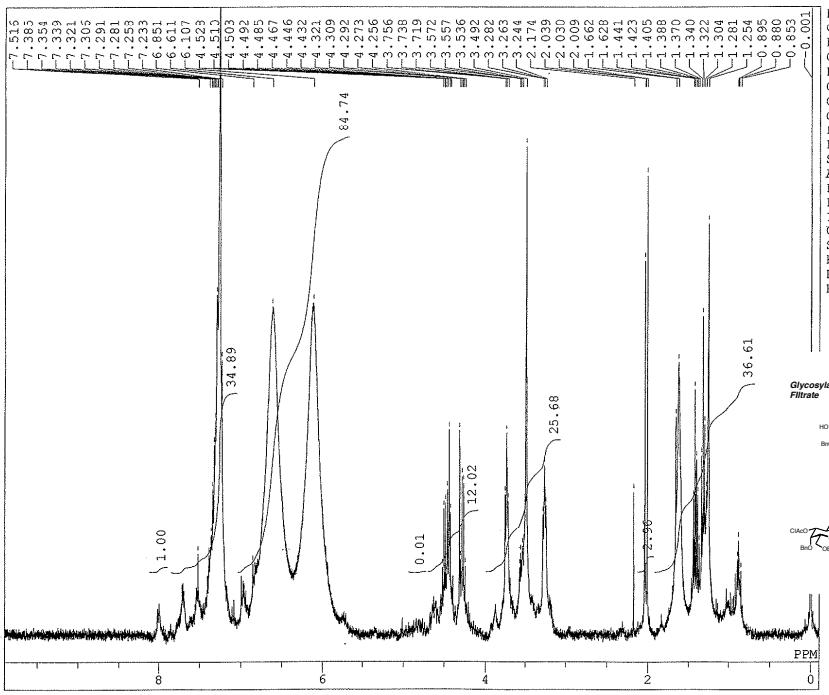


MS spectra for capping reaction; the bottom spectrum is for crude mixture, the middle spectrum is for portion left on the membrane, and the upper spectrum is for filtrate.


C:\WINNMR98\TEMPDATA\Auto1NON_E1.ALS
au_4_090_crude
975.35
8.52
8.34
8.40
1.07
1.00
10.28
45.04
18.35
141.82
0.84
1.23
2.82
PPM
8
6
4
2
0
DFILE C:\WINNMR98\TEMPDATA\Auto1
COMNT au_4_090_crude
DATIM Tue Dec 28 08:57:03 2004
OBNUC 1H
EXMOD NON
OBFRQ 399.65 MHz
OBSET 124.00 KHz
OBFIN 10500.0 Hz
POINT 32768
FREQU 8000.0 Hz
SCANS 4096
ACQTM 4.096 sec
PD 2.901 sec
PW1 5.2 us
IRNUC 1H
CTEMP 22.3 c
SLVNT CDCL3
EXREF 0.00 ppm
BF 0.12 Hz
RGAIN 26
Glycosylation
Crude
TMSOTf
MS AW 300
CH2Cl2


E:\Ueki\au_4_090\au_4_090_1.ALS
7.987
7.983
7.970
7.965
7.526
7.508
7.490
7.468
7.452
7.449
7.434
7.415
7.394
7.315
7.303
7.296
7.286
7.267
7.251
7.231
7.208
7.192
7.175
7.156
6.952
6.806
6.796
6.790
6.743
4.548
4.519
4.476
4.465
4.447
4.321
4.290
4.258
4.235
4.227
4.119
3.882
3.877
3.748
3.731
3.562
3.560
3.554
3.534
3.510
3.488
3.476
3.463
3.451
3.401
1.963
6.05
1.65
6.81
6.56
0.70
1.00
7.88
34.44
15.89
65.37
0.59
0.89
0.93
2.27
PPM
8
6
4
2
0
DFILE E:\Ueki\au_4_090\au_4_090_
COMNT
DATIM Tue Dec 28 00:39:35 2004
OBNUC 1H
EXMOD NON
OBFRQ 399.65 MHz
OBSET 124.00 KHz
OBFIN 10500.0 Hz
POINT 32768
FREQU 7993.6 Hz
SCANS 32
ACQTM 4.099 sec
PD 2.901 sec
PW1 5.2 us
IRNUC 1H
CTEMP 23.2 c
SLVNT CDCL3
EXREF 0.00 ppm
BF 0.12 Hz
RGAIN 11
Glycosylation
Portion on the membrane
TMSOTf
MS AW 300
CH2Cl2

E:\Ueki\au_4_090\au_4_090_2.ALS

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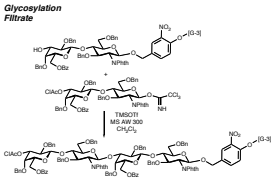
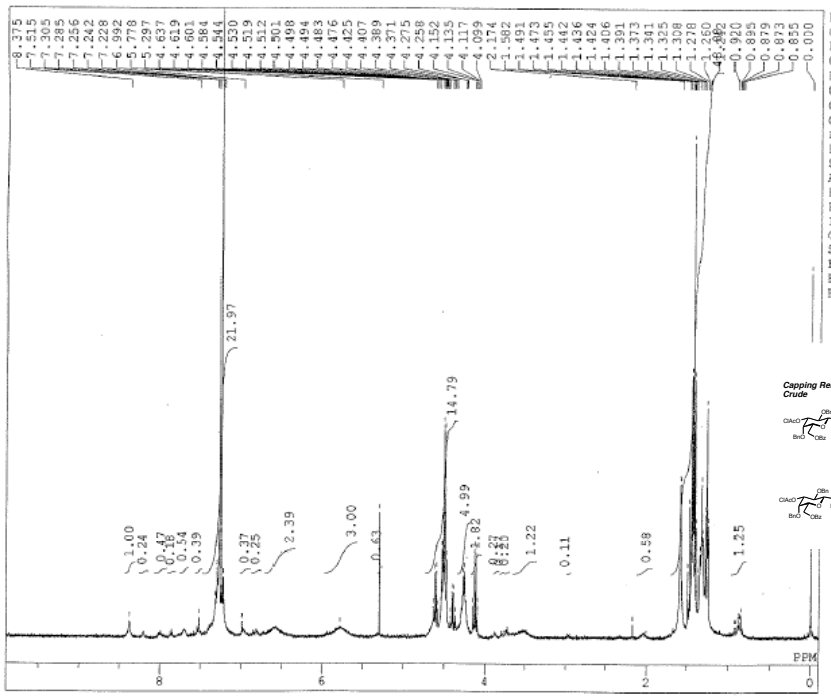
DFILE   E:\Ueki\au_4_090\au_4_090_
COMNT
DATIM   Tue Dec 28 00:49:05 2004
OBNUC   1H
EXMOD   NON
OBFRQ        399.65 MHz
OBSET        124.00 KHz
OBFIN       10500.0 Hz
POINT         32768
FREQU        7993.6 Hz
SCANS            32
ACQTM         4.099 sec
PD            2.901 sec
PW1             5.2 us
IRNUC   1H
CTEMP          22.9 c
SLVNT   CDCL3
EXREF          0.00 ppm
BF             0.12 Hz
RGAIN            22
```



E:\Ueki\au_4_094\au_4_094_2.ALS

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DFILE   E:\Ueki\au_4_094\au_4_094_
COMNT
DATIM   Tue Dec 28 17:27:22 2004
OBNUC   1H
EXMOD   NON
OBFRQ        399.65 MHz
OBSET        124.00 KHz
OBFIN       10500.0 Hz
POINT         32768
FREQU        7993.6 Hz
SCANS            16
ACQTM         4.099 sec
PD            2.901 sec
PW1             5.2 us
IRNUC   1H
CTEMP          22.5 c
SLVNT   CDCL3
EXREF          0.00 ppm
BF             0.12 Hz
RGAIN            23
```
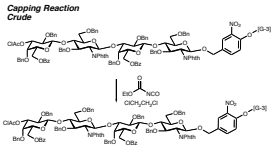
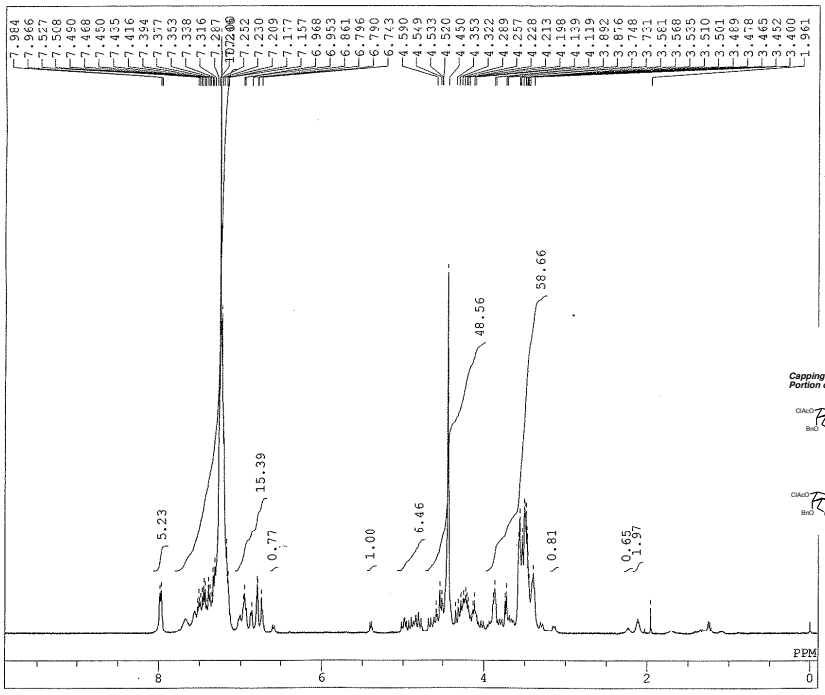
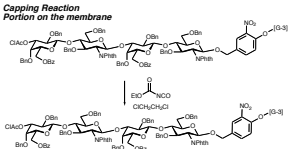



E:\Ueki\au_4_094\au_4_094_1.ALS

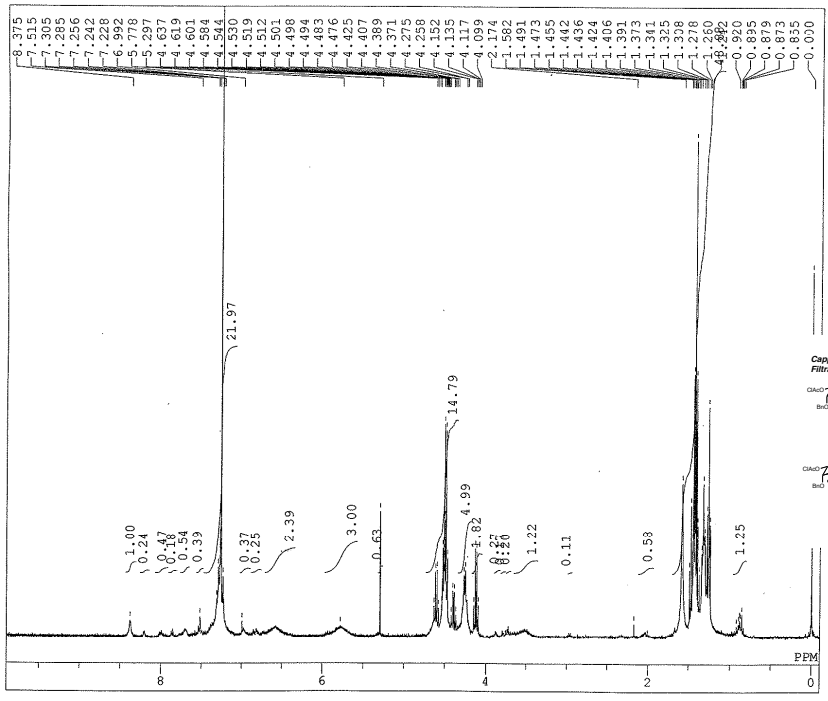


DFILE	E:\Ueki\au_4_094\au_4_094_
COMNT	
DATIM	Tue Dec 28 17:19:34 2004
OBNUC	1H
EXMOD	NON
OBFRQ	399.65 MHz
OBSET	124.00 KHz
OBFIN	10500.0 Hz
POINT	32760
FREQU	7993.6 Hz
SCANS	16
ACQTM	4.099 sec
PD	2.901 sec
PW1	5.2 us
IRNUC	1H
CTEMP	22.5 c
SLVNT	CDCL3
EXREF	0.00 ppm
BF	0.12 Hz
RGAIN	11

Capping Reaction Portion on the membrane

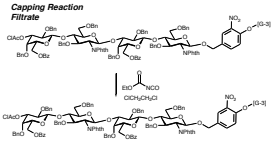


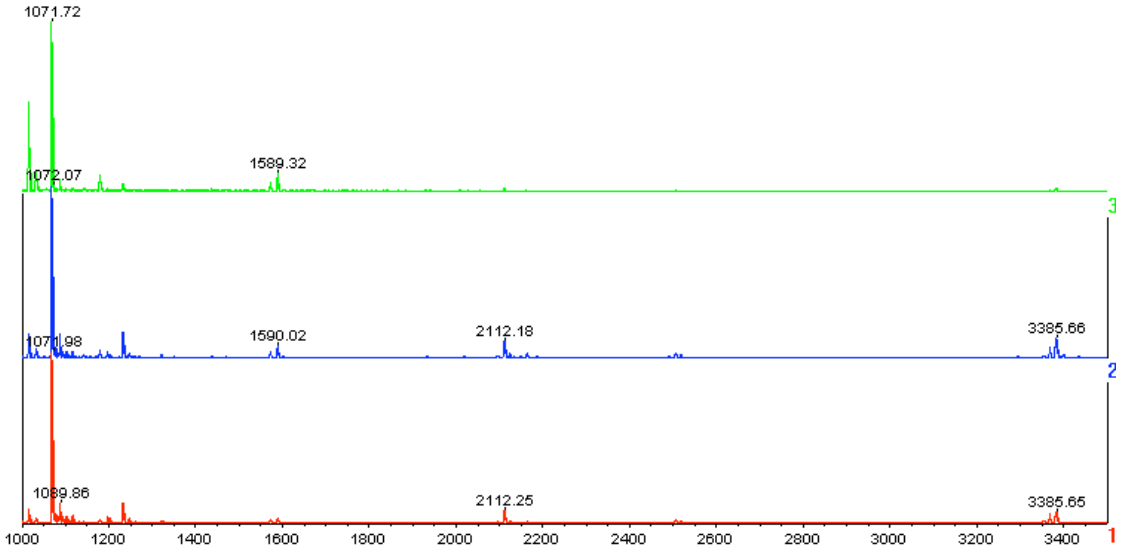
E:\Ueki\au_4_094\au_4_094_2.ALS



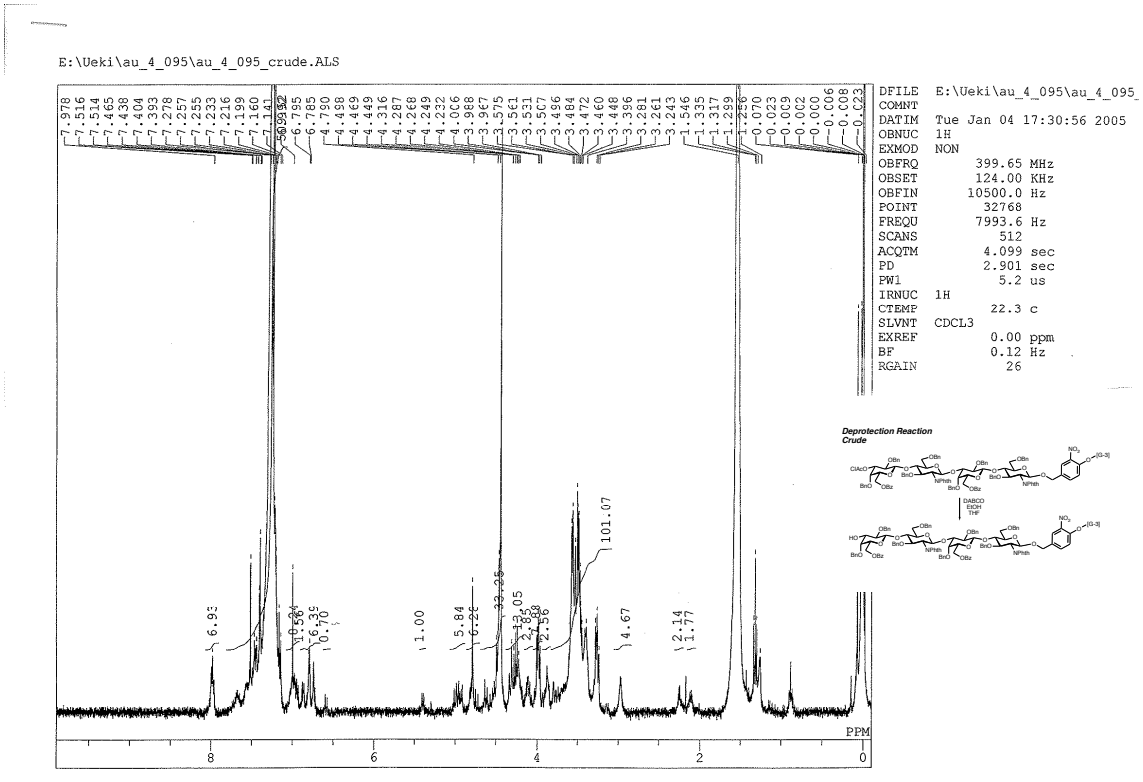
DFILE	E:\Ueki\au_4_094\au_4_094_
COMNT	
DATIM	Tue Dec 28 17:27:22 2004
OBNUC	1H
EXMOD	NON
OBFRQ	399.65 MHz
OBSET	124.00 KHz
OBFIN	10500.0 Hz
POINT	32768
FREQU	7993.6 Hz
SCANS	16
ACQTM	4.099 sec
PD	2.901 sec
PW1	5.2 us
IRNUC	1H
CTEMP	22.5 c
SLVNT	CDCL3
EXREF	0.00 ppm
BF	0.12 Hz
RGAIN	23

Capping Reaction Filtrate

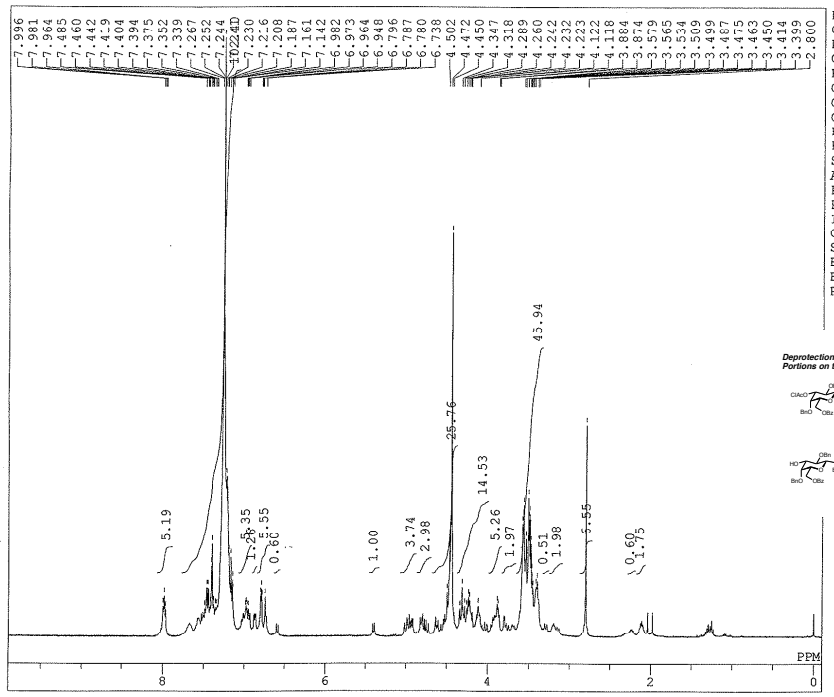




MS spectra for capping reaction; the bottom (red) spectrum is for crude mixture, the middle (blue) spectrum is for portion left on the membrane, and the upper (green) spectrum is for filtrate.

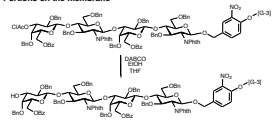


E:\Ueki\au_4_095\au_4_095_1.ALS

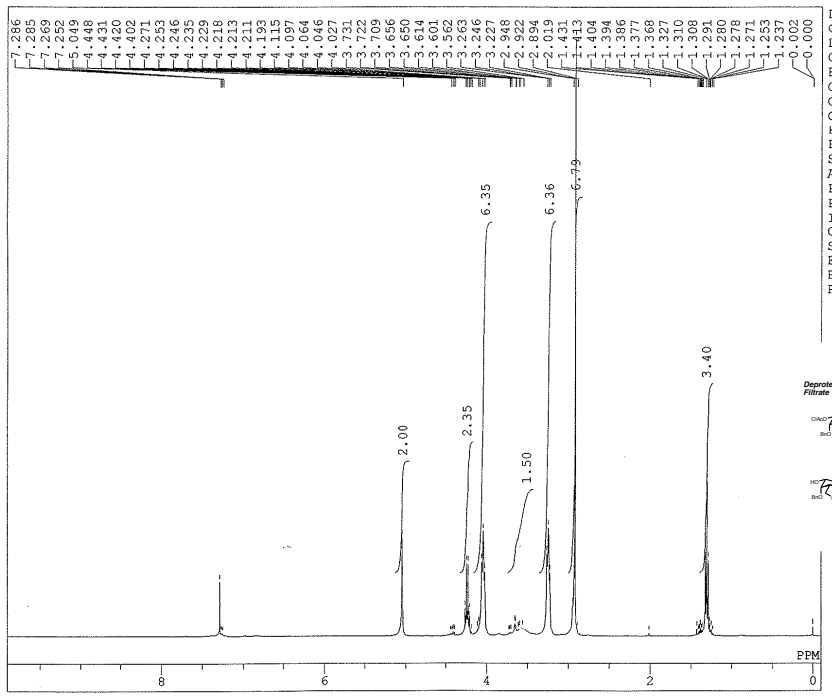


DFILE E:\Ueki\au_4_095\au_4_095_
COMNT
DATIM Tue Jan 04 18:09:41 2005
OBNUC 1H
EXMOD NON
OBFRQ 399.65 MHz
OBSET 124.00 KHz
OBFIN 10500.0 Hz
POINT 32768
FREQU 7993.6 Hz
SCANS 16
ACQTM 4.099 sec
PD 2.901 sec
PW1 5.2 us
IRNUC 1H
CTEMP 22.8 c
SLVNT CDCL3
EXREF 0.00 ppm
BF 0.12 Hz
RGAIN 12

Deprotection Reaction Portions on the membrane

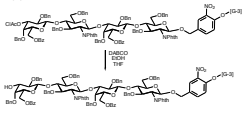


E:\Ueki\au_4_095\au_4_095_2.ALS

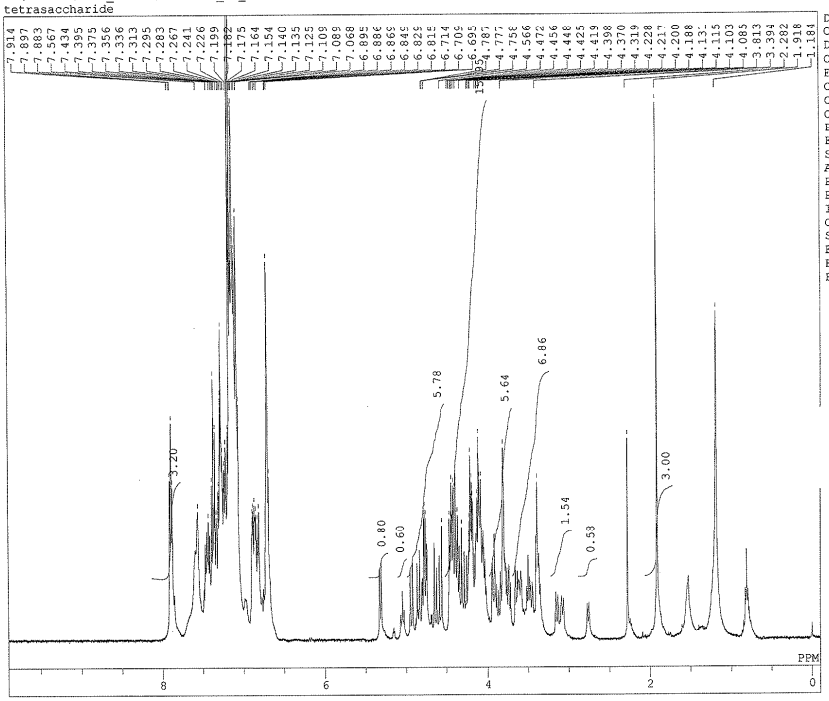


DFILE E:\Ueki\au_4_095\au_4_095_
COMNT
DATIM Tue Jan 04 18:17:30 2005
OBNUC 1H
EXMOD NON
OBFRQ 399.65 MHz
OBSET 124.00 KHz
OBFIN 10500.0 Hz
POINT 32768
FREQU 7993.6 Hz
SCANS 32
ACQTM 4.099 sec
PD 2.901 sec
PW1 5.2 us
IRNUC 1H
CTEMP 22.7 c
SLVNT CDCL3
EXREF 0.00 ppm
BF 0.12 Hz
RGAIN 14

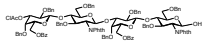
Deprotection Reaction Filtrate



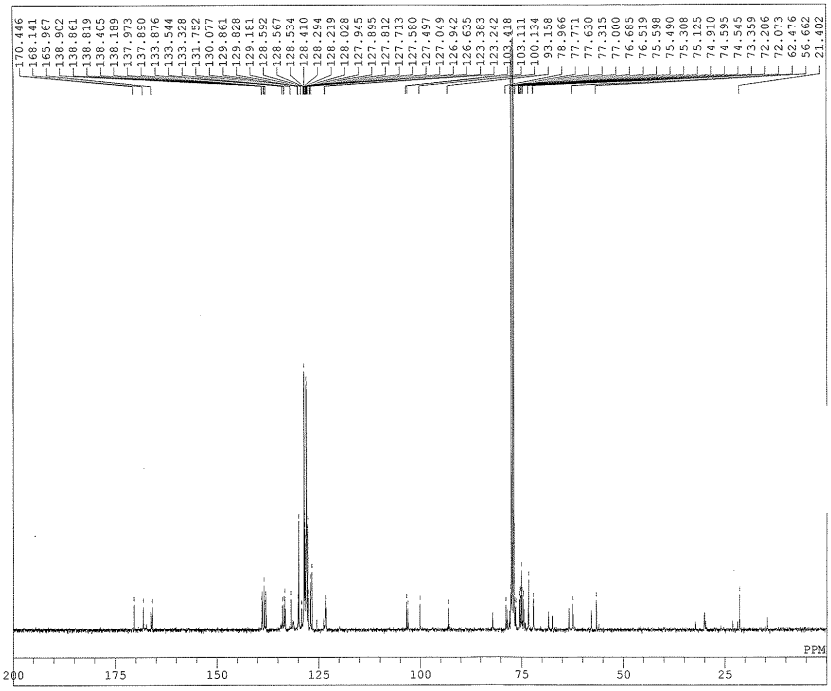
E:\Manabe\Uski_tetra\Auto1NON_E1_FT.als
tetrasaccharide



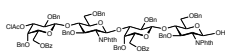

```
DFILE   E:\Manabe\Uski_tetra\Auto1NON
COMNT   tetrasaccharide
DATIM   Sat Jan 06 00:17:50 2007
OBNUC   1H
EXMOD   NON
OBFRQ         399.65 MHz
OBSET         124.00 KHz
OBFIN       10500.0 Hz
POINT         32768
FREQU         8000.0 Hz
SCANS            32
ACQTM          4.096 sec
PD             2.901 sec
PW1              5.5 us
IRNUC   1H
CTEMP           21.3 c
SLVNT   CDCL3
EXREF           0.00 ppm
BF              0.12 Hz
RGAIN             16
```

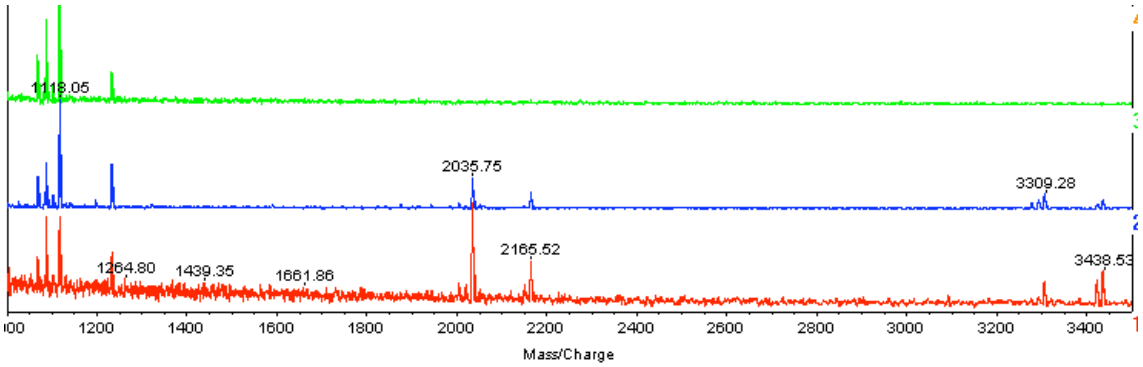


E:\Manabe\Uski_tetra\Auto2BCM_E1_FT.als




```
DFILE   E:\Manabe\Uski_tetra\Auto2BCM
COMNT
DATIM   Sat Jan 06 06:07:32 2007
OBNUC   13C
EXMOD   BCM
OBFRQ         100.40 MHz
OBSET         125.00 KHz
OBFIN       10500.0 Hz
POINT         32768
FREQU       27322.4 Hz
SCANS          7000
ACQTM          1.199 sec
PD             1.794 sec
PW1              5.2 us
IRNUC   1H
CTEMP           20.8 c
SLVNT   CDCL3
EXREF          77.00 ppm
BF              1.20 Hz
RGAIN             25
```





MS spectra for capping reaction; the bottom (red) spectrum is for crude mixture, the middle (blue) spectrum is for portion left on the membrane, and the upper (green) spectrum is for filtrate.