

Supporting Information of:

***Synthesis of 1D-Glyconanomaterials by a Hybrid Noncovalent-Covalent
Functionalization of Single Wall Carbon Nanotubes: Study of their
Selective Interactions with Lectins and with Live Cells.***

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General methods.

Chemicals employed all over this work were purchased from Aldrich Chemical Co. Dry solvents were purchased from SDS in *anhydrous grade* and in addition dried in a solvent purification system (Pure Solv MD5, Innovative Technology). The monitoring of the reactions was carried out by TLC, employing aluminum sheets coated with silica gel 60 F₂₅₄ (normal phase) purchased from Merk., with detection by charring with phosphomolybdic acid/EtOH and sulphuric acid/EtOH. For flash chromatography, silica Gel (Merck 230-400 mesh) was used. The organic extracts were dried over anhydrous sodium sulfate and concentrated under vacuum. Columns were eluted with positive air pressure. Chromatographic eluents are given as volume to volume ratios (v/v). NMR spectra were recorded with a BRUKER AC-500 apparatus. Deuterated solvents are indicated in brackets. Chemical shift values (δ) are referred to tetramethylsilane (TMS), utilized as internal reference; then, the spectral signals were calibrated according to the non-deuterated residual peak of the solvent. Optical rotations $[\alpha]_D^{20}$ were determined with a Perkin-Elmer 341 polarimeter using a sodium lamp ($\lambda = 589$ nm) with a 10 cm cell length. UV/Vis spectra were recorded on a UV/vis Perkin Elmer Lambda 12, using quartz cuvettes. HR-MS were recorded on a Kratos MS-80RFA 241-MC apparatus. Transmission Electron Microscopy (TEM) images were taken by Philips CM 10 or CM 200 apparatuses with an accelerating voltage of 80 kV or 200 kV, respectively. Typically, a very small volume of the aqueous solutions (20 μ L) was deposited over carbon-coated copper grids and uranyl acetate 2% as the negative stain. High resolution transmission electron microscopy (HRTEM) images were taken by a JEOL JEM-2200FS microscope, equipped with a field emission gun working at an accelerating voltage of 200 kV, a CEOS spherical aberration corrector and an Omega filter. Scanning electron microscopy (SEM) images were obtained on a JEOL JSM-5400 apparatus. Samples were prepared by depositing 15 μ l of the suspension onto grids, allowing the grids to absorb for 2 minutes. Atomic force microscopy (AFM) images were taken by working on a tapping mode by a Pico Plus Molecular Imaging followed by a treatment with the WSxM 5.0 Develop 2.0 software. First, AFM samples were prepared by evaporation of the aqueous solutions previously deposited on a

just exfoliated mica substrate ($5 \times 5 \text{ mm}^2$). Small angle X-ray scattering (SAXS) was performed on a *PANalytical X'Pert PRO*

NMR spectra were recorded with a Bruker AC-500 (^1H , 500 MHz) spectrometer. Chemical shifts are reported in ppm, and coupling constants are reported in Hz. Routine spectra were referenced to the residual proton or carbon signals of the solvent.

High-resolution mass spectra were recorded on a Kratos MS-80RFA 241-MC apparatus.

Optical rotations were determined with a Perkin-Elmer 341 polarimeter.

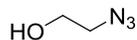
TEM analyses were performed on a Philips CM 10 or CM 200 apparatuses with an accelerating voltage of 80 kV or 200 kV, respectively. CM-120 operating at electron energy of 100 keV. Samples were prepared by depositing 15 μl of the suspension onto grids, allowing the grids to absorb for 2 minutes. Typically, a very small volume of the aqueous solutions (20 μL) was deposited over carbon-coated copper grids and uranyl acetate 2% as the negative stain. High resolution transmission electron microscopy (HRTEM) images were taken by a JEOL JEM-2200FS microscope, equipped with a field emission gun working at an accelerating voltage of 200 kV, a CEOS spherical aberration corrector and an Omega filter.

AFM images were taken by working on a tapping mode by Pico Plus Molecular Imaging followed by a treatment with the WSxM 5.0 Develop 2.0 software. AFM samples were prepared by evaporation of the aqueous solutions previously deposited on a just exfoliated mica substrate ($5 \times 5 \text{ mm}^2$).

UV-Vis spectra were obtained by Perkin-Elmer Lambda 12 Spectrometer.

Synthetic procedures and chemical characterizations

Compound 8

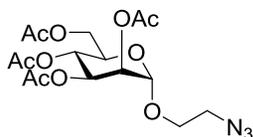


A solution of 2-bromoethanol (3.0 mL, 42 mmol) and sodium azide (3.3 g, 50.7 mmol) in water (15 mL) was heated for 3 h at 60°C. The residue was extracted with CH₂Cl₂ (2 x 20 mL) and the organic phase was washed with brine (15 mL). After drying over Na₂SO₄ and removal of solvent, the compound **8** was obtained with yield of 90% (3.3 g).

¹H NMR (300 MHz, CDCl₃): δ 3.75-3.65 (m, 2H), 3.37 (t, ³J(H,H) = 5.1 Hz, 2H).

¹³C NMR (100 MHz, CDCl₃): 61.2, 53.4.

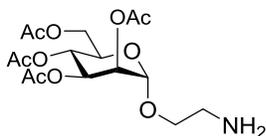
Compound 9



To a solution of the per-*O*-acetylated mannoside (2.3 mmol, 914 mg) and 2-azidoethanol (342.5 mg, 3.46 mmol) in anhydrous CH₂Cl₂ (10 mL) was added BF₃Et₂O (1.4 mL, 11.5 mmol) under an argon atmosphere at 0°C. The reaction mixture was heated at rt and stirred for 15 h. Then, the mixture was neutralized with aqueous NaHCO₃ (10 mL), the aqueous layer was extracted twice with dichloromethane (20 mL) and the organics were washed with brine (15 mL), dried with sodium sulfate, filtered, and evaporated. The crude product was purified by silica gel chromatography eluting with Et₂O/Hex (4:1) to give 578 mg (59% yield) of a white amorphous solid compound **9**. R_f = 0.40 (Et₂O/Hex (4:1)).

[α]_D²⁰ = +39.0 (c 0.9, CHCl₃). ¹H NMR (500 MHz, CDCl₃): δ 5.39 (dd, ³J(H,H) = 10.0 Hz, ³J(H,H) = 3.4 Hz, 1H), 5.32-5.28 (m, 2H), 4.88 (d, ³J(H,H) = 1.6 Hz, 1H), 4.31 (dd, ²J(H,H) = 12.4 Hz, ³J(H,H) = 5.4 Hz, 1H), 4.15 (dd, ²J(H,H) = 12.4 Hz, ³J(H,H) = 2.4 Hz, 1H), 4.08-4.04 (m, 1H), 3.92-3.88 (m, 1H), 3.72-3.68 (m, 1H), 3.54-3.45 (m, 2H), 2.19 (s, 3H), 2.13 (s, 3H), 2.08 (s, 3H), 2.02 (s, 3H). ¹³C NMR (125.7 MHz, CDCl₃): δ 170.6, 170.1, 170.0, 169.8, 97.7, 69.4, 68.9, 67.0, 66.0, 62.5, 50.4, 20.8, 20.7, 20.6. HRMS (ESI, m/z): [M + Na]⁺: calcd 440.1281, found 440.1291.

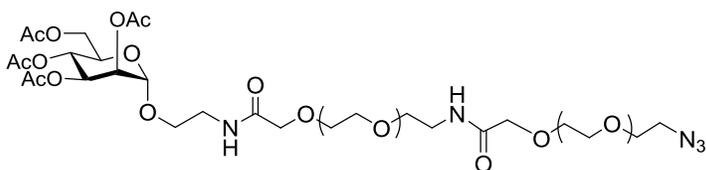
Compound 10



Azide derivative **9** (200 mg, 0.48 mmol) was placed in a Fisher-Porter, dissolved in CH_2Cl_2 (5 mL) and mixed with Pd/C (0.1 eq). The recipient was tightly sealed and a positive H_2 pressure (4 bars) insufflated, allowing the reaction to stir overnight. Subsequently, the mixture was filtered through a plug of celite® and the filtrate evaporated in vacuum. The compound was isolated from the crude by flash column chromatography, using $\text{CH}_2\text{Cl}_2/\text{MeOH}$ (9:1) as eluent, to give compound **10** (190 mg, 99%) as a white amorphous solid: $R_f = 0.19$ ($\text{CH}_2\text{Cl}_2/\text{MeOH}$ 9:1).

$[\alpha]_D^{20} = +36.8$ (c 0.7, CHCl_3). $^1\text{H NMR}$ (500 MHz, CDCl_3): δ 5.39 (dd, $^3J(\text{H,H}) = 10.0$ Hz, $^3J(\text{H,H}) = 3.4$ Hz, 1H), 5.40-5.28 (m, 2H), 4.97 (d, $^3J(\text{H,H}) = 1.2$ Hz, 1H), 4.31 (dd, $^2J(\text{H,H}) = 12.4$ Hz, $^3J(\text{H,H}) = 5.4$ Hz, 1H), 4.15 (dd, $^2J(\text{H,H}) = 12.4$ Hz, $^3J(\text{H,H}) = 2.4$ Hz, 1H), 4.12-4.05 (m, 2H), 3.93-3.87 (m, 1H), 3.42-3.32 (m, 4H), 2.15 (s, 3H), 2.12 (s, 3H), 2.06 (s, 3H), 1.98 (s, 3H). $^{13}\text{C NMR}$ (125.7 MHz, CDCl_3): δ 170.6, 170.1, 170.0, 169.6, 97.7, 69.2, 69.1, 68.7, 65.8, 64.0, 62.2, 39.1, 20.7, 20.6, 20.5. HRMS (ESI, m/z): $[\text{M} + \text{Na}]^+$ $\text{C}_{16}\text{H}_{25}\text{NO}_{10}$: calcd 414.1376, found 414.1400.

Compound 12



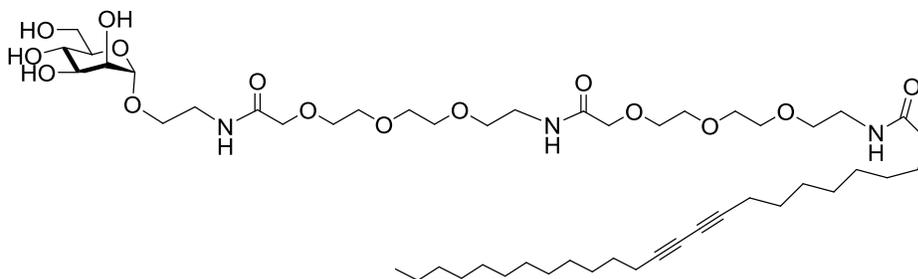
To a solution of compound **11** (105 mg, 0.25 mmol) in DMF (1 mL) was added, sequentially at room temperature, TBTU (80 mg, 0.25 mmol) and DIPEA (61 μL , 0.38 mmol). The solution was stirred for 5 min before a solution of compound **10** (100 mg, 0.25 mmol) and DIPEA (61 μL , 0.38 mmol) in DMF (1 mL) was added slowly. The solution was stirred for 2 h before the solvent was removed under vacuum. The residue was dissolved in CH_2Cl_2 (100 mL) and washed with 1M HCl (20 mL), saturated aqueous NaHCO_3 (40 mL) and brine (20 mL). After drying over Na_2SO_4 and removal of solvent, the crude product was purified by silica gel chromatography eluting with $\text{CH}_2\text{Cl}_2/\text{MeOH}$ 20:1 to give 119 mg of a yellow oil compound **12** (60% yield): $R_f = 0.47$ ($\text{CH}_2\text{Cl}_2/\text{MeOH}$ 9:1).

$[\alpha]_D^{20} = +14.4$ (c 0.7, CHCl_3). $^1\text{H NMR}$ (500 MHz, CDCl_3): δ 7.33-7.32 (m, 1H), 7.13-7.11 (m, 1H), 5.28-5.23 (m, 3H), 4.81 (s, 1H), 4.22 (dd, $^2J(\text{H,H}) = 12.4$ Hz, $^3J(\text{H,H}) =$

and DIPEA (18 μL , 0.10 mmol). The solution was stirred for 5 min before a solution of compound **13** (33 mg, 0.06 mmol) and DIPEA (18 μL , 0.10 mmol) in DMF (1 mL) was added slowly. The solution was stirred for 16 h before the solvent was removed under vacuum. The residue was dissolved in CH_2Cl_2 (50 mL) and washed with 1M HCl (20 mL), saturated aqueous NaHCO_3 (40 mL) and brine (20 mL). After drying over Na_2SO_4 and removal of solvent, the crude product was purified by silica gel chromatography eluting with $\text{CH}_2\text{Cl}_2/\text{MeOH}$ 9:1 to give 70 mg of a white amorphous solid compound **14** (73% yield). $R_f = 0.33$ ($\text{CH}_2\text{Cl}_2/\text{MeOH}$ 9:1).

$[\alpha]_D^{20} = +16.5$ (c 1.1, CHCl_3). $^1\text{H NMR}$ (500 MHz, CDCl_3): δ 7.37-7.35 (m, 1H), 7.19-7.18 (m, 1H), 6.40-6.39 (m, 1H), 5.30-5.25 (m, 3H), 4.84 (d, $^3J(\text{H,H}) = 1.5$ Hz, 1H), 4.27 (dd, $^2J(\text{H,H}) = 12.4$ Hz, $^3J(\text{H,H}) = 5.4$ Hz, 1H), 4.12 (dd, $^2J(\text{H,H}) = 12.4$ Hz, $^3J(\text{H,H}) = 2.5$ Hz, 1H), 4.06 (s, 2H), 4.04 (s, 2H), 4.02-3.97 (m, 1H), 3.84-3.80 (m, 1H), 3.72-3.47 (m, 27H), 2.25 (t, $^3J(\text{H,H}) = 6.9$ Hz, 4H), 2.18 (m, 5H), 2.12 (s, 3H), 2.06 (s, 3H), 2.00 (s, 3H), 1.65-1.60 (m, 2H), 1.55-1.48 (m, 4H), 1.34-1.25 (m, 26H), 0.89 (t, $^3J(\text{H,H}) = 6.5$ Hz, 3H). $^{13}\text{C NMR}$ (125.7 MHz, CDCl_3): δ 173.2, 170.5, 170.2, 170.0, 169.9, 169.7, 169.5, 97.4, 77.5, 77.1, 70.9, 70.8, 70.7, 70.6, 70.5, 70.4, 70.2, 70.1, 70.0, 69.9, 69.2, 68.9, 68.6, 66.7, 66.0, 65.2, 65.1, 62.3, 39.0, 38.6, 38.2, 36.4, 31.8, 29.5, 29.3, 29.2, 29.1, 28.9, 28.8, 28.7, 28.2, 25.5, 22.5, 20.7, 20.6, 20.5, 19.0, 14.0. HRMS (ESI, m/z): $[\text{M} + \text{Na}]^+$: calcd 1148.6457, found 1148.6498.

Compound 15



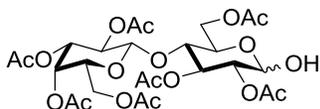
To a solution of compound **14** (23 mg, 0.024 mmol) in dry methanol (2 mL) was added NaOMe solution 1M (100

μL , 0.1 mmol). The reaction was allowed to proceed at rt in the dark for 1 h at which time the reaction was judged complete by TLC analysis. The solution was neutralized with Amberlyst Ir-120 (plus) resin. The resin was removed by filtration and the solvent removed

under vacuum. The crude product was purified by size-exclusion chromatography (sephadex G20) eluting with methanol. Lyophilization of the solvent gave the desired compound **15** with a 90% of yield (20 mg). $R_f = 0.16$ ($\text{CH}_2\text{Cl}_2/\text{MeOH}$ 9:1).

$[\alpha]_D^{20} = +9.8$ (c 1.0, MeOH). ^1H NMR (500 MHz, MeOD): δ 8.5 (s, 1H), 8.0 (s, 2H), 4.78 (s, 1H), 4.01 (s, 2H), 4.00 (s, 2H), 3.85-3.77 (m, 3H), 3.72-3.35 (m, 31H), 2.25 (t, $^3J(\text{H,H}) = 6.9$ Hz, 4H), 2.20 (t, $^3J(\text{H,H}) = 7.0$ Hz, 2H), 1.62-1.60 (m, 2H), 1.52-1.47 (m, 4H), 1.41-30 (m, 26H), 0.89 (t, $^3J(\text{H,H}) = 6.9$ Hz, 3H). ^{13}C NMR (125.7 MHz, MeOD): δ 176.4, 172.9, 172.8, 101.6, 77.9, 77.8, 74.8, 72.6, 72.1, 72.0, 71.9, 71.5, 71.4, 70.6, 70.5, 68.6, 67.0, 66.4, 62.9, 40.3, 39.8, 39.7, 37.0, 33.1, 30.7, 30.6, 30.5, 30.3, 30.2, 30.1, 30.0, 29.9, 29.5, 27.0, 23.7, 19.7, 14.4. HRMS (ESI, m/z): $[\text{M} + \text{Na}]^+$: calcd 980.6035, found 980.5988.

Compound 17,¹⁰⁰

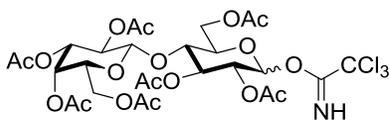


To a solution of per-*O*-acetylated lactoside (1.0 g, 1.47 mmol) in DMF (20 mL) was added $\text{H}_2\text{NNH}_3\text{OAc}$ (189 mg, 2.06 mmol).

The reaction was stirred at rt for 1 h. The mixture was neutralized with water (20mL) and the residue was extracted with CH_2Cl_2 (2 x10 mL) and the organic phase was washed with brine (15 mL). After drying over Na_2SO_4 and removal of solvent, the crude product was purified by silica gel chromatography eluting with EtOAc/Hex (2:1) to give a white amorphous solid (800 mg, 86% yield). $R_f = 0.28$ (EtOAc/Hex 2:1), ratio $\alpha:\beta$ (1:0.5).

^1H NMR (500 MHz, CDCl_3): δ 5.55 (t, $^3J(\text{H,H}) = 9.2$ Hz, 1H), 5.40-5.37 (m, 2H), 5.3 (t, $^3J(\text{H,H}) = 9.2$ Hz, 1H), 5.16-5.14 (m, 1H), 5.0-4.97 (m, 1H), 4.86 (dd, $^3J(\text{H,H}) = 9.2$ Hz, $^3J(\text{H,H}) = 3.4$ Hz, 1H), 4.80 (d, $^3J(\text{H,H}) = 9.2$ Hz, 1H), 4.75 (t, $^3J(\text{H,H}) = 9.2$ Hz, 1H), 4.54-4.49 (m, 2H), 4.20-4.08 (m, 3H), 3.92-3.88 (m, 1H), 3.82-3.77 (m, 2H), 3.70-3.68 (m, 1H), 3.50 (d, $^3J(\text{H,H}) = 7.6$ Hz, 1H), 3.07 (d, $^3J(\text{H,H}) = 3.6$ Hz, 1H), 2.18 (s, 3H), 2.15 (s, 3H), 2.10 (s, 3H), 2.08 (s, 3H), 2.07 (s, 3H), 2.06 (s, 3H), 1.99 (s, 3H). ^{13}C NMR (125.7 MHz, CDCl_3): δ 170.2, 170.1, 170.0, 169.4, 168.9, 101.0, 95.2, 90.0, 76.2, 76.1, 73.4, 72.8, 71.9, 71.1, 70.9, 70.8, 70.6, 70.5, 69.4, 69.0, 68.2, 66.5, 61.9, 61.7, 60.7, 20.7, 20.6, 20.4. HRMS (ESI, m/z): $[\text{M} + \text{Na}]^+$: calcd 659.1799, found 659.1830.

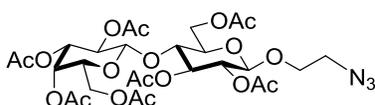
Compound 18,¹⁰⁰



To a solution of **17** (780 mg, 1.2 mmol) in CH₂Cl₂ (15 mL) was added potassium carbonate (170 mg, 1.2 mmol) and trichloroacetonitrile (1.3 mL, 12.3 mmol). The reaction mixture was stirred at rt for 4 h, and then it was filtered through a plug of celite® and the filtrate evaporated in vacuum. The crude product was purified by silica gel chromatography eluting with EtOAc/Hex 2:1 to give 874 mg of a white amorphous solid compound **18** (91% yield). R_f = 0.51 (EtOAc/Hex 2:1), ratio α:β (1:1).

¹H NMR (500 MHz, CDCl₃): δ 8.69 (s, 1H), 8.65 (s, 1H), 6.40 (d, ³J(H,H) = 3.5 Hz, 1H), 5.79 (d, ³J(H,H) = 9.5 Hz, 1H), 5.47 (t, ³J(H,H) = 9.5 Hz, 1H), 5.27-5.26 (m, 1H), 5.20 (t, ³J(H,H) = 8.4 Hz, 1H), 5.11 (t, ³J(H,H) = 8.0 Hz, 1H), 5.06-4.96 (m, 2H), 4.91-4.88 (m, 2H), 4.49-4.39 (m, 2H), 4.09-4.01 (m, 4H), 3.91-3.79 (m, 4H), 2.07 (s, 6H), 2.03 (s, 6H), 1.99 (s, 9H), 1.96 (s, 6H), 1.95 (s, 3H), 1.94 (s, 3H), 1.93 (s, 3H), 1.88 (s, 6H). ¹³C NMR (125.7 MHz, CDCl₃): δ 170.1, 170.0, 169.4, 169.1, 168.9, 160.6, 160.4, 100.9, 100.7, 95.0, 92.7, 90.5, 75.6, 75.4, 73.0, 72.2, 70.9, 70.7, 70.5, 70.2, 69.7, 69.4, 69.0, 68.8, 66.5, 61.6, 61.4, 60.8, 60.7, 60.1, 20.2, 14.0.

Compound 19

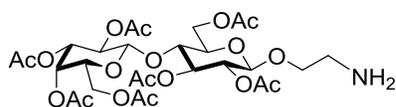


To a solution of **18** (450 mg., 0.58 mmol) and 2-azidoethanol (285 mg, 2.9 mmol) in anhydrous CH₂Cl₂ (20 mL) was added BF₃Et₂O (8 μL, 0.06 mmol) under an argon atmosphere and at 0°C. The reaction mixture was heated at rt and stirred for 36 h. Then, the mixture was neutralized with aqueous NaHCO₃ (10 mL), the aqueous layer was extracted twice with dichloromethane (20 mL) and the organics were washed with brine (15mL), dried with sodium sulfate, filtered, and evaporated. The crude product was purified by silica gel chromatography eluting with EtAcO/Hex (2:1) to give 190 mg (45% yield) of a white amorphous solid. R_f = 0.26 EtAcO/Hex (2:1).

[α]_D²⁰ = +15.2 (c 1.0, CHCl₃). ¹H NMR (500 MHz, CDCl₃): δ 5.34 (d, ³J(H,H) = 3.0 Hz, 1H), 5.20 (t, ³J(H,H) = 9.5 Hz, 1H), 5.10 (t, ³J(H,H) = 10.0 Hz, 1H), 4.96 (dd, ³J(H,H) = 10.0 Hz, ³J(H,H) = 3.5 Hz, 1H), 4.92 (t, ³J(H,H) = 9.5 Hz, 1H), 4.56-4.49 (m, 3H), 4.14-4.06 (m, 3H), 3.99-3.96 (m, 1H), 3.89-3.86 (m, 1H), 3.82 (t, ³J(H,H) = 9.5 Hz,

1H), 3.69-3.61 (m, 1H), 3.48-3.45 (m, 1H), 3.29-3.25 (m, 1H), 2.14 (s, 3H), 2.12 (s, 3H), 2.06 (s, 3H), 2.04 (s, 9H), 1.96 (s, 3H). ¹³C NMR (125.7 MHz, CDCl₃): δ 170.1, 170.0, 169.7, 169.1, 101.1, 100.4, 76.1, 72.8, 72.7, 71.5, 70.9, 70.7, 69.1, 68.6, 66.6, 61.8, 60.8, 50.5, 20.8, 20.7, 20.5. HRMS (ESI, m/z): [M + Na]⁺: calcd 728.2126, found 728.2131.

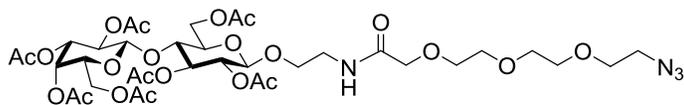
Compound 20



Compound **19** (88 mg, 0.12 mmol) was placed in a Fisher-Porter, dissolved in CH₂Cl₂ (5 mL) and mixed with Pd/C (0.1 eq). The recipient was tightly sealed and a positive H₂ pressure (4 bars) insufflated, allowing the reaction to stir overnight. Subsequently, the mixture was filtered through a plug of celite® and the filtrate evaporated in vacuum. The compound was isolated from the crude by flash column chromatography, using CH₂Cl₂/MeOH (9:1) as eluent, to give compound **20** (84 mg, 99%) as a white amorphous solid. R_f = 0.23 (CH₂Cl₂/MeOH 9:1).

[α]_D²⁰ = - 10.1 (c 0.3, CHCl₃). ¹H NMR (500 MHz, CDCl₃): δ 5.37 (d, ³J(H,H) = 3.0 Hz, 1H), 5.20 (t, ³J(H,H) = 9.5 Hz, 1H), 5.11 (dd, ³J(H,H) = 10.0 Hz, ³J(H,H) = 7.8 Hz, 1H), 5.02 (dd, ³J(H,H) = 10.0 Hz, ³J(H,H) = 3.0 Hz, 1H), 4.91 (dd, ³J(H,H) = 9.5 Hz, ³J(H,H) = 7.9 Hz, 1H), 4.82-4.80 (m, 1H), 4.63 (d, ³J(H,H) = 7.8 Hz, 1H), 4.60 (d, ³J(H,H) = 7.9 Hz, 1H), 4.19-4.04 (m, 5H), 3.96-3.95 (m, 1H), 3.88 (t, ³J(H,H) = 9.5 Hz, 1H), 3.78-3.75 (m, 1H), 3.36-3.34 (m, 1H), 3.24-3.20 (m, 1 H), 2.57 (s, 2H), 2.20 (s, 3H), 2.18 (s, 3H), 2.09 (s, 6H), 2.08 (s, 3H), 2.07 (s, 3H), 1.99 (s, 3H). ¹³C NMR (125.7 MHz, CDCl₃): δ 171.3, 170.4, 170.2, 170.1, 170.0, 169.7, 169.2, 100.9, 100.8, 77.2, 75.5, 73.2, 72.5, 71.3, 70.9, 70.6, 69.1, 67.1, 66.7, 60.7, 40.4, 21.1, 20.8, 20.7, 20.5. HRMS (ESI, m/z): [M + Na]⁺: calcd 702.2221, found 702.2216.

Compound 22

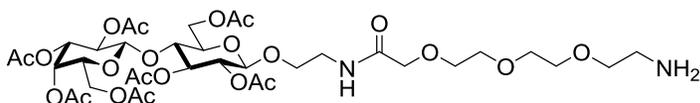


To a solution of compound **21** (30 mg, 0.13 mmol) in DMF (1 mL) was added, sequentially at room temperature, TBTU (42 mg, 0.13 mmol) and DIPEA (29 μL, 0.18 mmol). The solution was stirred for 5 min before a solution of compound **20** (80 mg, 0.12 mmol) and

DIPEA (29 μ L, 0.18 mmol) in DMF (1 mL) was added slowly. The solution was stirred for 2 h before the solvent was removed under vacuum. The residue was dissolved in CH_2Cl_2 (100 mL) and washed with 1M HCl (20 mL), saturated aqueous NaHCO_3 (40 mL) and brine (20 mL). After drying over Na_2SO_4 and removal of solvent, the crude product was purified by silica gel chromatography eluting with $\text{CH}_2\text{Cl}_2/\text{MeOH}$ 20:1 to give 93 mg of a yellow oil compound **22** (86% yield): $R_f = 0.36$ ($\text{CH}_2\text{Cl}_2/\text{MeOH}$ 9:1).

$[\alpha]_D^{20} = -5.0$ (c 0.6, CHCl_3). ^1H NMR (500 MHz, CDCl_3): δ 7.14 (t, $^3J(\text{H,H}) = 7.0$ Hz, 1H), 5.33 (d, $^3J(\text{H,H}) = 3.0$ Hz, 1H), 5.16 (t, $^3J(\text{H,H}) = 9.5$ Hz, 1H), 5.11 (dd, $^3J(\text{H,H}) = 10.0$ Hz, $^3J(\text{H,H}) = 7.8$ Hz, 1H), 4.93 (dd, $^3J(\text{H,H}) = 10.0$ Hz, $^3J(\text{H,H}) = 3.0$ Hz, 1H), 4.86 (dd, $^3J(\text{H,H}) = 9.5$ Hz, $^3J(\text{H,H}) = 7.9$ Hz, 1H), 4.48-4.40 (m, 3H), 4.14-3.98 (m, 6H), 3.88-3.36 (m, 18H), 2.13 (s, 3H), 2.10 (s, 3H), 2.04 (s, 3H), 2.02 (s, 9H), 1.94 (s, 3H). ^{13}C NMR (125.7 MHz, CDCl_3): δ 170.2, 170.1, 170.0, 169.9, 169.8, 169.5, 168.9, 100.9, 100.4, 76.1, 72.6, 72.5, 71.4, 70.7, 70.5, 70.4, 70.3, 70.2, 69.8, 68.9, 68.5, 66.5, 61.8, 60.6, 50.5, 38.4, 20.6, 20.3. HRMS (ESI, m/z): $[\text{M} + \text{Na}]^+$: calcd 917.3127, found 917.3153.

Compound 23



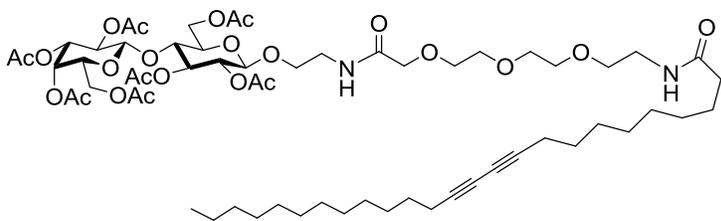
Compound **22** (88 mg, 0.1 mmol) was placed in a Fisher-Porter, dissolved in CH_2Cl_2 (5 mL) and

mixed with Pd/C (0.1 eq). The recipient was tightly sealed and a positive H_2 pressure (4 bars) insufflated, allowing the reaction to stir overnight. Subsequently, the mixture was filtered through a plug of celite® and the filtrate evaporated in vacuum. The compound was isolated from the crude by flash column chromatography, using $\text{CH}_2\text{Cl}_2/\text{MeOH}$ (9:1) as eluent, to give compound **13** (85 mg, 99%) as a white amorphous solid. $R_f = 0.14$ ($\text{CH}_2\text{Cl}_2/\text{MeOH}$ 9:1).

$[\alpha]_D^{20} = -6.4$ (c 0.8, CHCl_3). ^1H NMR (500 MHz, CDCl_3): δ 7.18-7.15 (m, 1H), 5.36 (d, $^3J(\text{H,H}) = 3.2$ Hz, 1H), 5.19 (t, $^3J(\text{H,H}) = 9.5$ Hz, 1H), 5.11 (dd, $^3J(\text{H,H}) = 10.0$ Hz, $^3J(\text{H,H}) = 7.8$ Hz, 1H), 4.98 (dd, $^3J(\text{H,H}) = 10.0$ Hz, $^3J(\text{H,H}) = 3.2$ Hz, 1H), 4.92 (dd, $^3J(\text{H,H}) = 9.5$ Hz, $^3J(\text{H,H}) = 7.9$ Hz, 1H), 4.52-4.49 (m, 3H), 4.17-4.07 (m, 4H), 3.98 (dd, $^2J(\text{H,H}) = 12.4$ Hz, $^3J(\text{H,H}) = 4.0$ Hz, 1H), 3.91-3.85 (m, 2H), 3.80 (t, $^3J(\text{H,H}) = 9.5$ Hz, 1H), 3.73-3.60 (m, 11H), 3.54 (t, $^3J(\text{H,H}) = 5.0$ Hz, 2H), 3.47-3.37 (m, 2H), 2.16 (s, 3H),

2.13 (s, 3H), 2.07 (s, 3H), 2.06 (s, 9H), 1.98 (s, 3H), 1.90-1.88 (m, 2H). ^{13}C NMR (125.7 MHz, CDCl_3): δ 170.2, 170.0, 169.6, 168.9, 100.9, 100.4, 76.1, 72.6, 71.4, 70.8, 70.7, 70.5, 70.2, 69.9, 69.0, 68.5, 66.5, 61.8, 60.6, 39.8, 38.5, 20.7, 20.6, 20.4. HRMS (ESI, m/z): $[\text{M} + \text{Na}]^+$: calcd 891.2993, found 891.3015.

Compound 24

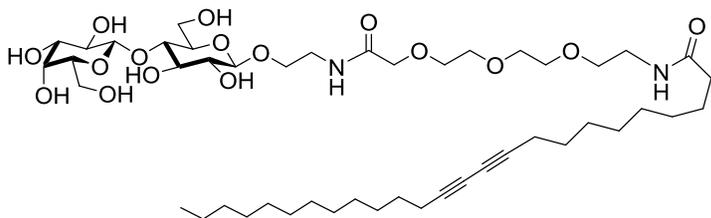


To a solution of 10, 12-pentacosadienoic acid (42 mg, 0.11 mmol) in DMF (1 mL) were added, sequentially at room temperature in the dark, TBTU

(32 mg, 0.1 mmol) and DIPEA (27 μL , 0.15 mmol). The solution was stirred for 5 min before a solution of compound **23** (80 mg., 0.09 mmol) and DIPEA (27 μL , 0.15 mmol) in DMF (1 mL) was added slowly. The solution was stirred for 16 h before the solvent was removed under vacuum. The residue was dissolved in CH_2Cl_2 (50 mL) and washed with 1M HCl (20 mL), saturated aqueous NaHCO_3 (40 mL) and brine (20 mL). After drying over Na_2SO_4 and removal of solvent, the crude product was purified by silica gel chromatography eluting with $\text{CH}_2\text{Cl}_2/\text{MeOH}$ 9:1 to give 94 mg of a white amorphous solid compound **24** (84% yield). $R_f = 0.70$ ($\text{CH}_2\text{Cl}_2/\text{MeOH}$ 9:1).

$[\alpha]_D^{20} = -2.9$ (c 0.9, CHCl_3). ^1H NMR (500 MHz, CDCl_3): δ 7.13 (t, $^3J(\text{H,H}) = 6.0$ Hz, 1H), 6.34 (t, $^3J(\text{H,H}) = 6.0$ Hz, 1H), 5.33 (d, $^3J(\text{H,H}) = 3.0$ Hz, 1H), 5.16 (t, $^3J(\text{H,H}) = 9.5$ Hz, 1H), 5.09 (dd, $^3J(\text{H,H}) = 10.3$ Hz, $^3J(\text{H,H}) = 7.8$ Hz, 1H), 4.94 (dd, $^3J(\text{H,H}) = 10.3$ Hz, $^3J(\text{H,H}) = 3.0$ Hz, 1H), 4.87 (dd, $^3J(\text{H,H}) = 9.5$ Hz, $^3J(\text{H,H}) = 7.9$ Hz, 1H), 4.49-4.46 (m, 3H), 4.12-3.84 (m, 8H), 3.77 (t, $^3J(\text{H,H}) = 9.5$ Hz, 1H), 3.68-3.60 (m, 11H), 3.54 (t, $^3J(\text{H,H}) = 5.2$ Hz, 2H), 3.43 (t, $^3J(\text{H,H}) = 6.4$ Hz, 2H), 2.23 (t, $^3J(\text{H,H}) = 6.9$ Hz, 4H), 2.18-2.15 (m, 5H), 2.10 (s, 3H), 2.04 (s, 3H), 2.03 (s, 9H), 1.95 (s, 3H), 1.63-1.58 (m, 2H), 1.52-1.47 (m, 4H), 1.38-1.23 (m, 26H), 0.86 (t, $^3J(\text{H,H}) = 6.5$ Hz, 3H). ^{13}C NMR (125.7 MHz, CDCl_3): δ 176.9, 173.4, 170.3, 170.1, 170.0, 169.7, 169.0, 101.0, 100.6, 77.6, 77.4, 76.2, 72.8, 71.6, 70.8, 70.6, 70.5, 70.4, 70.1, 70.0, 69.1, 68.7, 66.6, 65.3, 65.2, 62.0, 60.7, 39.2, 38.6, 36.5, 33.9, 31.9, 29.4, 29.3, 29.2, 29.1, 28.9, 28.8, 28.7, 28.3, 25.7, 24.8, 20.8, 20.6, 20.5, 19.2, 14.1. HRMS (ESI, m/z): $[\text{M} + \text{Na}]^+$: calcd 1247.6302, found 1247.6299.

Compound 25.

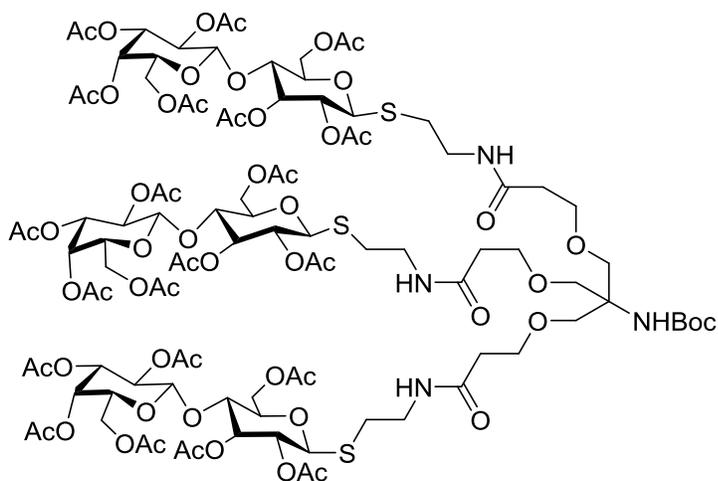


To a solution of compound **24** (88 mg, 0.07 mmol) in dry methanol (2 mL) was added NaOMe solution 1M (100 μ L, 0.1 mmol). The reaction was

allowed to proceed at rt in the dark for 1 h at which time the reaction was judged complete by TLC analysis. The solution was neutralized with Amberlyst Ir-120 (plus) resin. The resin was removed by filtration and the solvent removed under vacuum. The crude product was purified by size-exclusion chromatography (sephadex G20) eluting with methanol. Lyophilization of the solvent gave the desired compound **25** with a 92% of yield (66 mg). $R_f = 0.4$ ($\text{CH}_3\text{CN}/\text{H}_2\text{O}/\text{NH}_4\text{OH}$ 6:1:1).

$[\alpha]_D^{20} = +1.2$ (c 0.5, MeOH). ^1H NMR (500 MHz, MeOD): δ 8.36 (s, 1H), 4.39 (d, $^3J(\text{H,H}) = 7.5$ Hz, 1H), 4.35 (d, $^3J(\text{H,H}) = 8.0$ Hz, 1H), 4.04 (s, 2H), 4.00-3.78 (m, 6H), 3.75-3.38 (m, 21H), 3.31-3.27 (m, 1H), 2.25 (t, $^3J(\text{H,H}) = 6.9$ Hz, 4H), 2.15 (t, $^3J(\text{H,H}) = 7.6$ Hz, 2H), 1.60 (t, $^3J(\text{H,H}) = 7.0$ Hz, 2H), 1.56-1.50 (m, 4H), 1.45-1.30 (m, 26H), 0.93 (t, $^3J(\text{H,H}) = 6.5$ Hz, 3H). ^{13}C NMR (125.7 MHz, MeOD): δ 176.4, 172.9, 105.1, 104.4, 80.7, 77.8, 77.0, 76.5, 76.3, 74.8, 74.7, 71.9, 71.5, 71.4, 71.3, 71.2, 70.6, 70.3, 69.5, 66.4, 62.5, 61.9, 40.3, 40.0, 37.0, 35.1, 33.1, 30.7, 30.6, 30.5, 30.3, 30.2, 30.1, 30.0, 29.8, 29.5, 27.0, 26.1, 23.7, 19.7, 14.5. HRMS (ESI, m/z): $[\text{M} + \text{Na}]^+$: calcd 953.5562, found 953.55601.

Compound 27

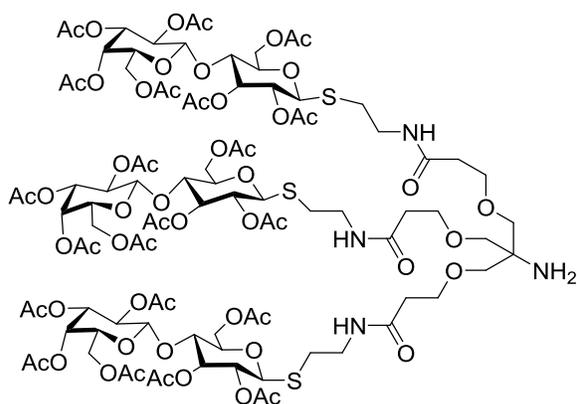


TBTU (80.2 mg, 0.25 mmol) and DIPEA (125 μ L, 0.36 mmol) were added sequentially at room temperature and under an argon atmosphere to a solution of tri-acid **26** (32 mg, 0.08 mmol) in dry DMF (1 mL). The resulting solution was stirred for 5 min. and then, a

solution of compound **2** (179 mg, 0.25 mmol) and DIPEA (125 μ L, 0.36 mmol) in dry DMF (2 mL) was added slowly. The afforded mixture was stirred for 14 h under an argon atmosphere before the solvent was removed under vacuum. The obtained residue was dissolved in CH_2Cl_2 (100 mL), washed with a 1M HCl solution (20 mL) and neutralized with a saturated aqueous NaHCO_3 (30 mL), and finally with brine (20 mL). After drying over Na_2SO_4 and removal of solvent, the crude product was subjected to a chromatographic column with $\text{CH}_2\text{Cl}_2/\text{MeOH}$ (10:1) to give 189 mg (87%) as a yellowish solid. $R_f = 0.20$ ($\text{CH}_2\text{Cl}_2/\text{MeOH}$ 10:1).

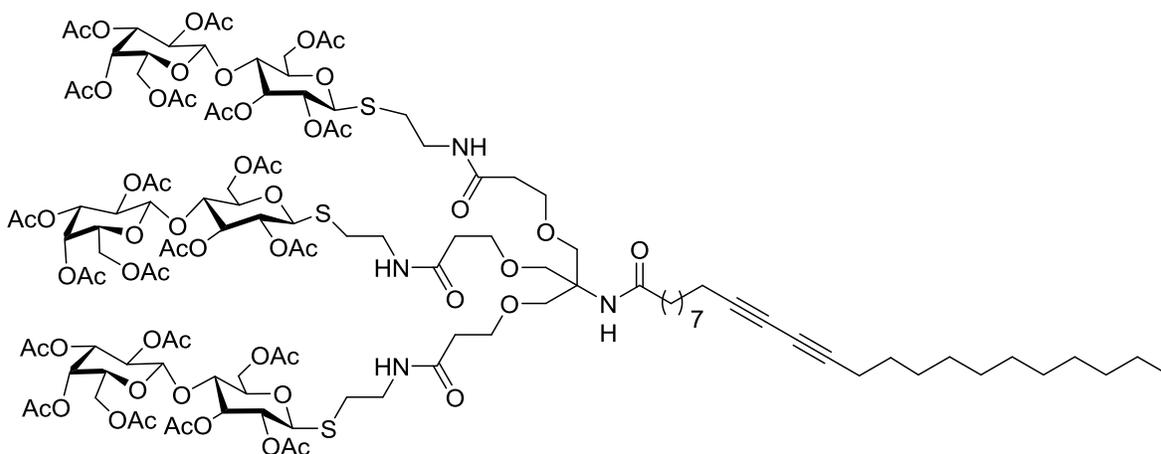
$[\alpha]_D^{20} = -9.5$ (c 1.0, CHCl_3). ^1H NMR (500 MHz, CDCl_3): δ 6.79-6.78 (m, 1H), 5.37 (d, $^3J(\text{H,H}) = 2.9$ Hz, 3H), 5.22 (t, $^3J(\text{H,H}) = 9.1$ Hz, 3H), 5.12 (dd, $^3J(\text{H,H}) = 10.3$ Hz, $^3J(\text{H,H}) = 7.9$ Hz, 3H), 5.00 (dd, $^3J(\text{H,H}) = 10.3$ Hz, $^3J(\text{H,H}) = 2.9$ Hz, 3H), 4.93 (t, $^3J(\text{H,H}) = 9.1$ Hz, 3H), 4.60-4.53 (m, 9H), 4.18-4.08 (m, 9H), 3.93-3.90 (m, 3H), 3.82 (t, $^3J(\text{H,H}) = 9.1$ Hz, 3H), 3.71-3.65 (m, 25H), 3.56-3.39 (m, 3H), 2.92-2.73 (m, 6H), 2.46 (t, $^3J(\text{H,H}) = 5.6$ Hz, 6H), 2.17 (s, 9H), 2.14 (s, 9H), 2.08 (s, 9H), 2.07 (s, 27H), 1.99 (s, 9H), 1.27 (s, 9H). ^{13}C NMR (125.7 MHz, CDCl_3): δ 171.4, 170.3, 170.2, 170.0, 169.9, 169.6, 169.5, 168.9, 100.9, 83.5, 77.1, 75.9, 73.5, 70.8, 70.6, 70.0, 69.6, 69.0, 67.2, 66.5, 61.7, 60.6, 58.5, 39.3, 36.5, 30.4, 28.3, 20.8, 20.7, 20.6, 20.4. EM: $m/z = 2493.4$ $[\text{M}+\text{Na}]^+$.

Compound 28



A 1:1 solution of TFA/ CH_2Cl_2 (4 mL) was added over the protected amine **27** (170 mg, 0.07 mmol) and stirred for 5 h. After removal of the solvent under vacuum, the crude product was purified by silica gel chromatography, eluting with a mixture of $\text{CH}_2\text{Cl}_2/\text{MeOH}$ (9:1), affording the free amine **28** (165 mg, 99%) as yellow syrup.

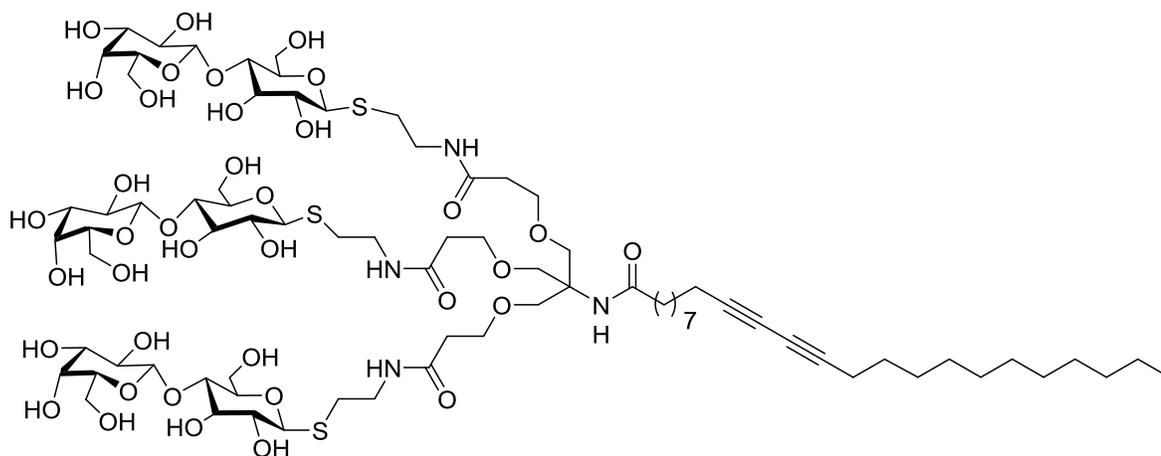
Compound 29



To a solution of 10, 12-pentacosadienoic acid (28 mg, 0.07 mmol) in DMF (1 mL) were added, sequentially at room temperature in the dark, TBTU (22.5 mg, 0.07 mmol) and DIPEA (18 μ L, 0.10 mmol). The solution was stirred for 5 min before a solution of compound **28** (165 mg, 0.07 mmol) and DIPEA (18 μ L, 0.10 mmol) in DMF (1 mL) was added slowly. The solution was stirred for 16 h before the solvent was removed under vacuum. The residue was dissolved in CH_2Cl_2 (50 mL) and washed with 1M HCl (20 mL), saturated aqueous NaHCO_3 (40 mL) and brine (20 mL). After drying over Na_2SO_4 and removal of solvent, the crude product was purified by silica gel chromatography eluting with $\text{CH}_2\text{Cl}_2/\text{MeOH}$ 9:1 to give 162 mg of a yellow amorphous solid compound **29** (82% yield). $R_f = 0.38$ ($\text{CH}_2\text{Cl}_2/\text{MeOH}$ 10:1).

$[\alpha]_D^{20} = -3.1$ (c 0.4, CHCl_3). ^1H NMR (500 MHz, CDCl_3): δ 6.79-6.77 (m, 1H), 5.37 (d, $^3J(\text{H,H}) = 2.9$ Hz, 3H), 5.22 (t, $^3J(\text{H,H}) = 9.1$ Hz, 3H), 5.12 (dd, $^3J(\text{H,H}) = 10.3$ Hz, $^3J(\text{H,H}) = 7.9$ Hz, 3H), 5.00 (dd, $^3J(\text{H,H}) = 10.3$ Hz, $^3J(\text{H,H}) = 2.9$ Hz, 3H), 4.93 (t, $^3J(\text{H,H}) = 9.1$ Hz, 3H), 4.61-4.53 (m, 9H), 4.19-4.08 (m, 9H), 3.94-3.90 (m, 3H), 3.82 (t, $^3J(\text{H,H}) = 9.1$ Hz, 3H), 3.72-3.65 (m, 15H), 3.56-3.38 (m, 6H), 2.92-2.72 (m, 6H), 2.47 (t, $^3J(\text{H,H}) = 5.6$ Hz, 3H), 2.28-2.22 (m, 6H), 2.17 (s, 9H), 2.14 (s, 9H), 2.08 (s, 9H), 2.07 (s, 27H), 1.99 (s, 9H), 1.62-1.58 (m, 2H), 1.55-1.50 (m, 4H), 1.40-1.28 (m, 26H), 0.90 (t, $^3J(\text{H,H}) = 6.9$ Hz, 3H). ^{13}C NMR (125.7 MHz, CDCl_3): δ 171.4, 170.3, 170.2, 170.0, 169.9, 169.6, 169.5, 168.9, 100.9, 83.5, 77.7, 77.5, 75.8, 73.5, 70.8, 70.6, 70.0, 69.5, 69.0, 67.2, 66.5, 61.7, 60.6, 39.3, 36.2, 31.8, 30.4, 29.5, 29.3, 29.2, 29.1, 29.0, 28.9, 28.7, 28.2, 25.7, 22.5, 20.8, 20.7, 20.6, 20.4, 19.1, 14.0. EM: $m/z = 2749.9$ $[\text{M}+\text{Na}]^+$.

Compound 30



To a solution of compound **29** (25 mg, 0.0092 mmol) in dry methanol (2 mL) was added NaOMe solution 1M (50 μ L, 0.05 mmol). The reaction was allowed to proceed at rt in the dark for 1 h at which time the reaction was judged complete by TLC analysis. The solution was neutralized with Amberlyst Ir-120 (plus) resin. The resin was removed by filtration and the solvent removed under vacuum. The crude product was purified by size-exclusion chromatography (sephadex G20) eluting with methanol. Lyophilization of the solvent gave the desired glycodendron **30** with 51% of yield (23 mg).

$[\alpha]_D^{20} = -16.3$ (c 0.6, MeOH). $^1\text{H NMR}$ (500 MHz, MeOD): δ 4.45 (d, $^3J(\text{H,H}) = 9.8$ Hz, 3H), 4.36 (d, $^3J(\text{H,H}) = 8.5$ Hz, 3H), 3.94-3.76 (m, 12H), 3.72-3.68 (m, 18H), 3.61-3.43 (m, 30H), 3.32-3.28 (m, 3H), 2.92-2.74 (m, 6H), 2.45 (t, $^3J(\text{H,H}) = 5.6$ Hz, 3H), 2.25 (t, $^3J(\text{H,H}) = 6.9$ Hz, 4H), 2.19 (t, $^3J(\text{H,H}) = 7.0$ Hz, 2H), 1.60-1.58 (m, 2H), 1.53-1.47 (m, 4H), 1.40-1.28 (m, 26H), 0.90 (t, $^3J(\text{H,H}) = 6.5$ Hz, 3H). $^{13}\text{C NMR}$ (125.7 MHz, MeOD): δ 174.2, 105.1, 87.1, 80.6, 80.5, 77.9, 77.1, 74.8, 74.1, 72.5, 70.3, 70.2, 70.0, 68.7, 66.4, 62.5, 62.1, 41.2, 37.7, 33.1, 30.7, 30.6, 30.5, 30.4, 30.2, 30.1, 29.9, 29.6, 29.5, 27.1, 23.7, 19.7, 14.4. EM: $m/z = 1867.2$ $[\text{M}+\text{Na}]^+$.

Determination of carbohydrate quantity by anthrone method.

A freshly prepared solution of anthrone (0.5 % w/v in concentrated H₂SO₄, 1 mL) was added to various concentrations of D-mannose and D-lactose of known concentration (0.5 mL) under stirring in a water-bath. After that, the solution was then heated to 90°C for 12 min. Then, the resulting green bluish solutions were rapidly cooled down in an ice bath during further 10 min. Next, the absorbance of the solution was measured at 620 nm and the data was plotted against D-mannose and D-lactose concentrations, obtaining the calibration curves for each sugar. To calculate the carbohydrate quantity on SWCNTs, 0.5 mg of SWCNTs-Lac and 1.15 mg of SWCNTs-Man were dissolved in 0.5 mL Milli-Q water, and then a freshly prepared solution of anthrone was added, following the same procedure described above.

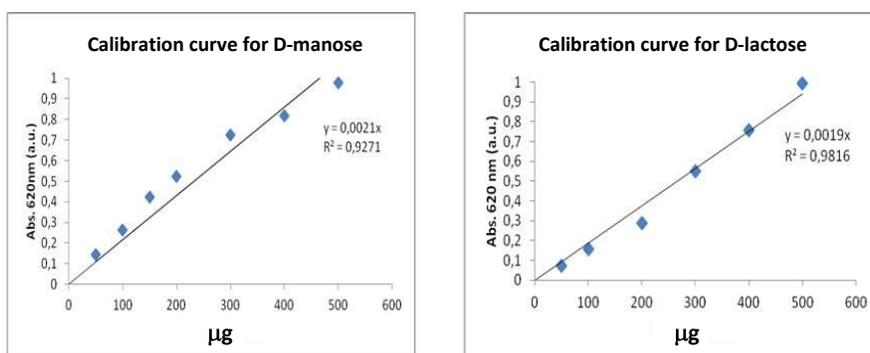
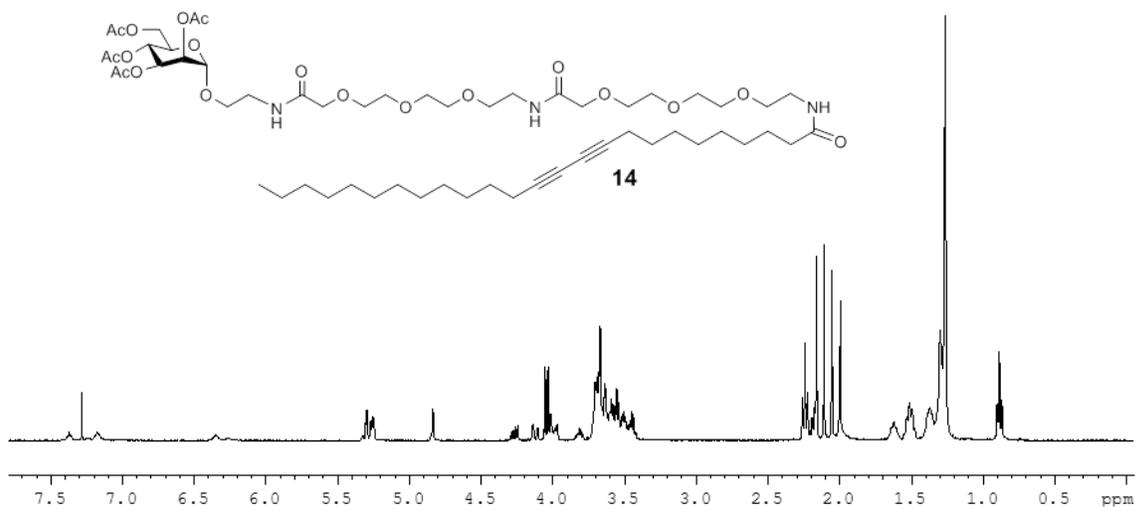
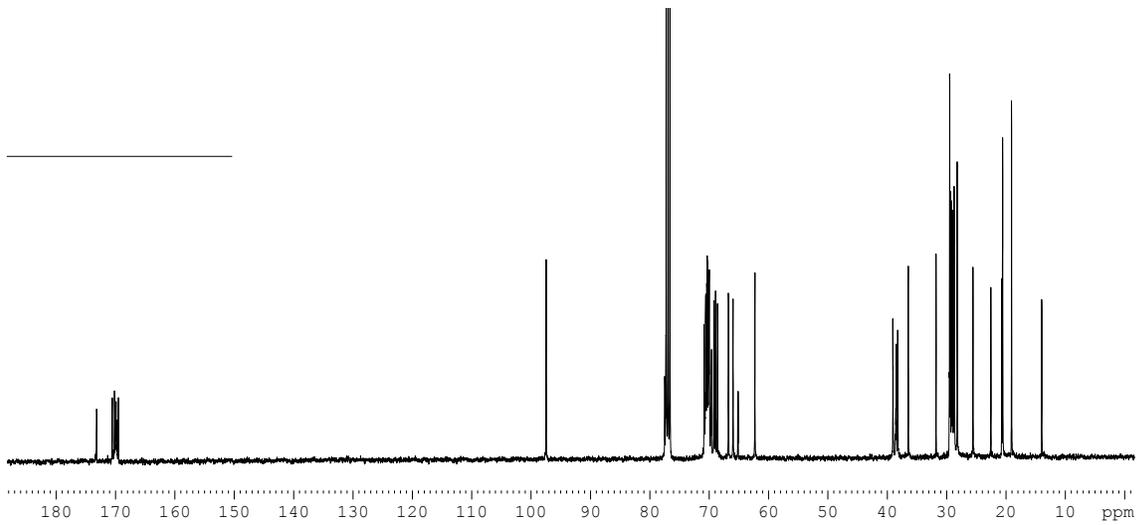


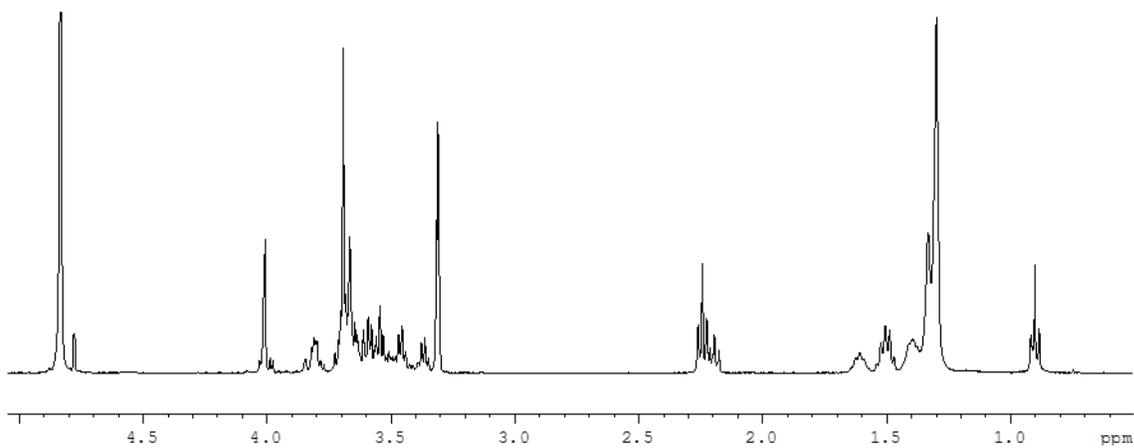
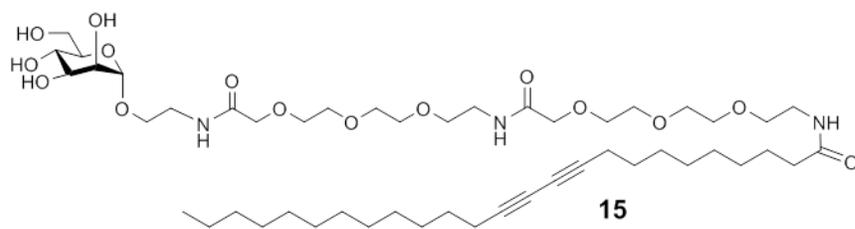
Figure 1. Calibration curves for D-mannose and D-lactose.



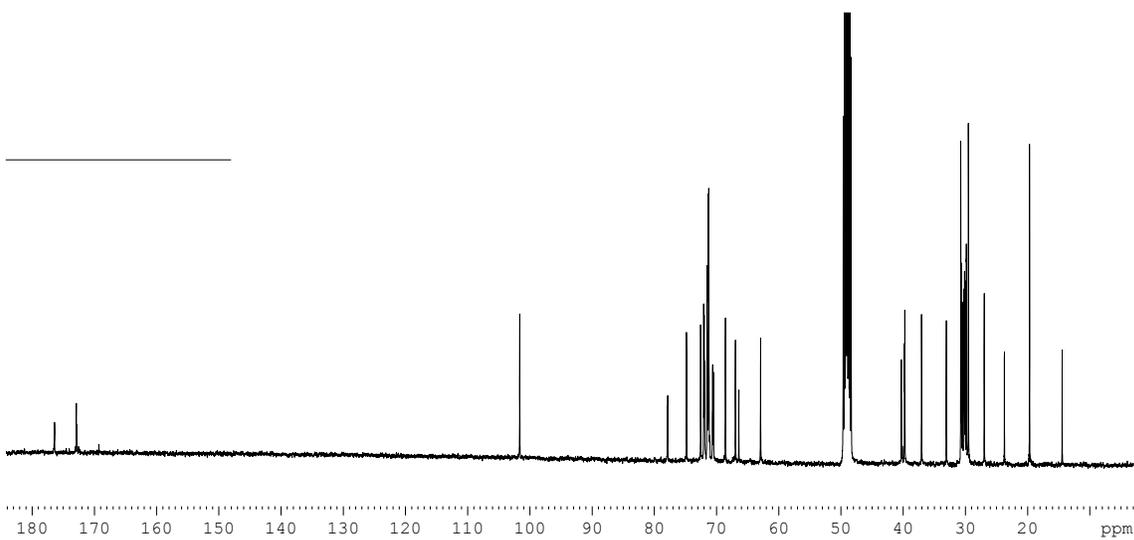
¹H NMR spectrum (500 MHz, CDCl₃) of compound **14**.



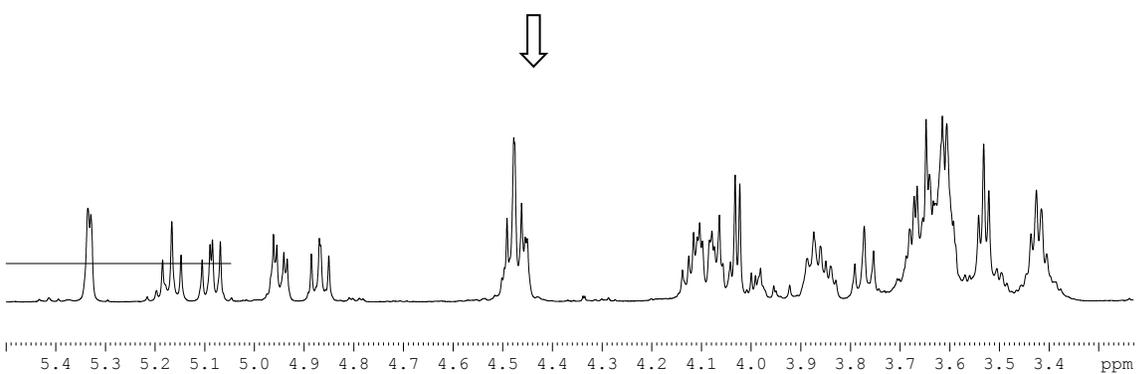
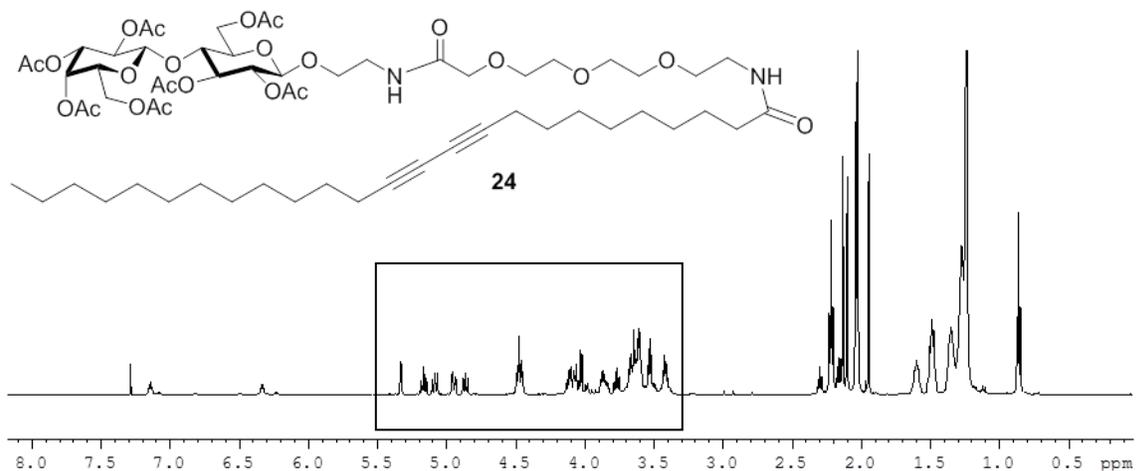
¹³C NMR spectrum (125.7 MHz, CDCl₃) of compound **14**.



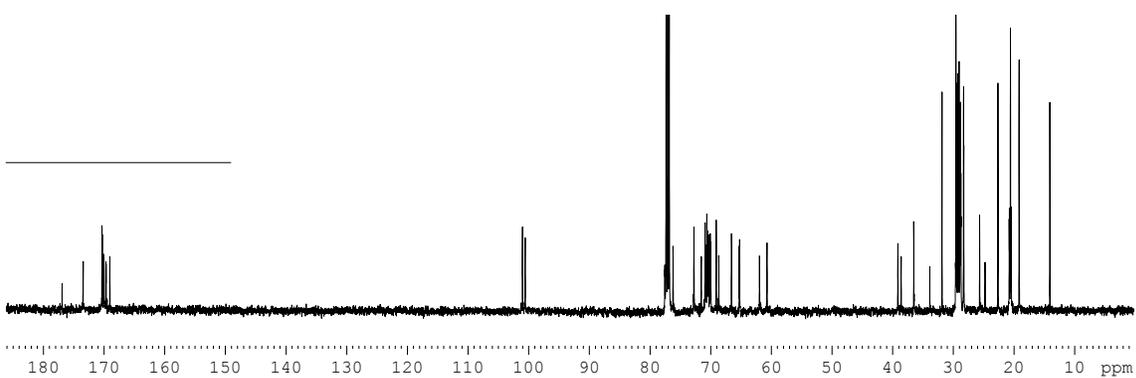
¹H NMR spectrum (500 MHz, D₂O) of compound **15**.



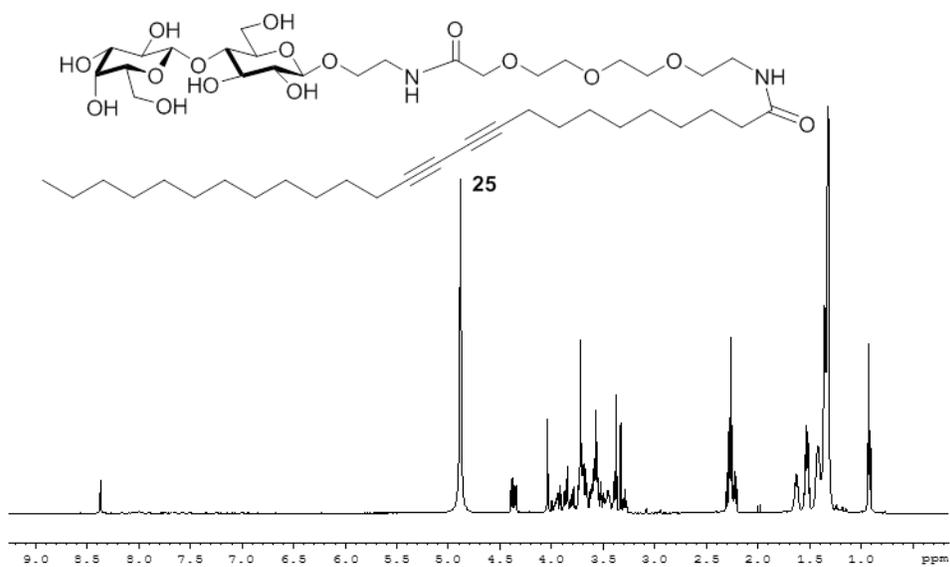
¹³C NMR spectrum (125.7 MHz, D₂O) of compound **15**.



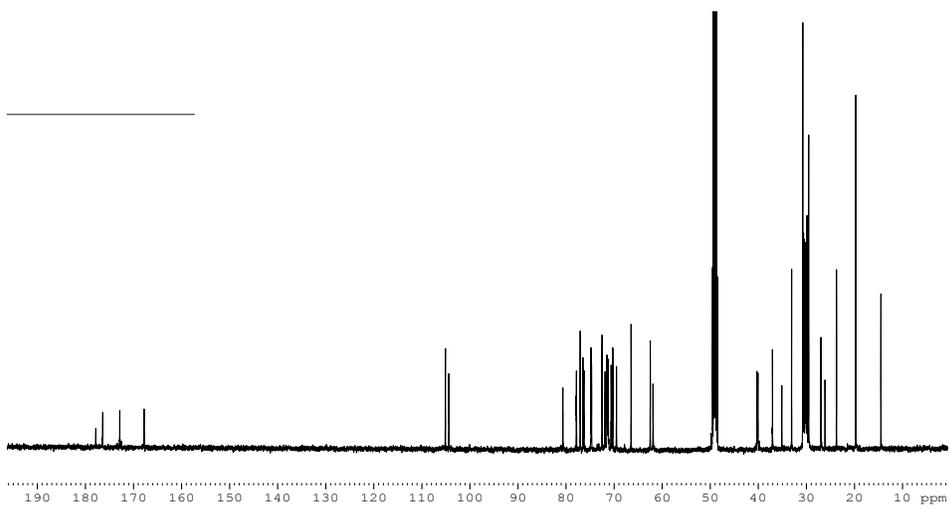
^1H NMR spectrum (500 MHz, CDCl_3) of compound **24**.



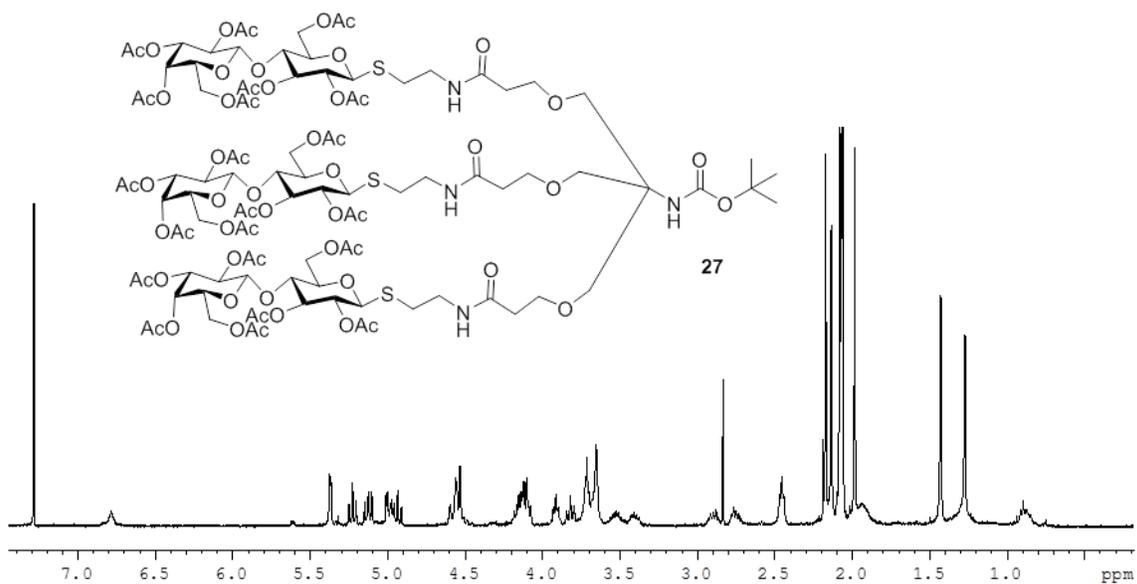
^{13}C NMR spectrum (125.7 MHz, CDCl_3) of compound **24**.



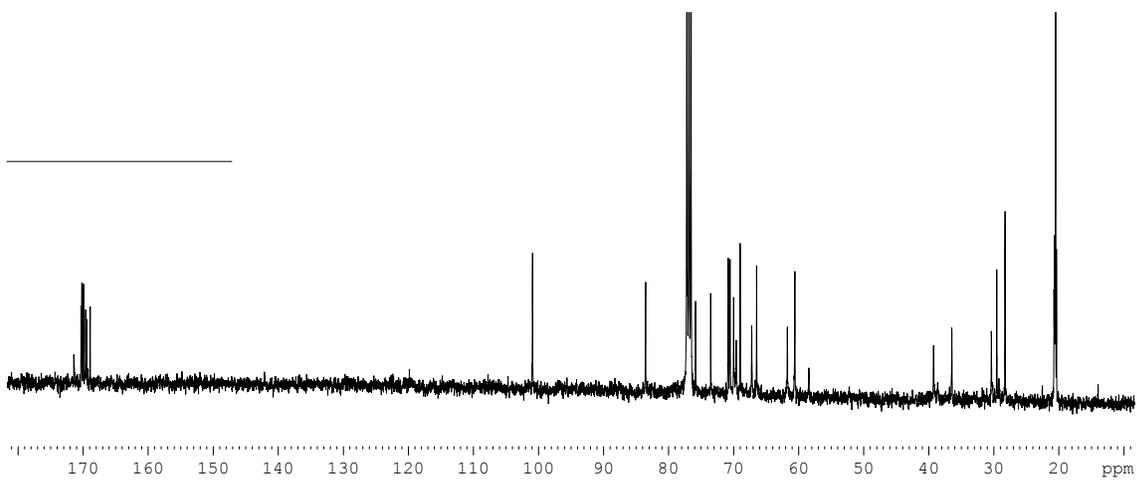
¹H NMR spectrum (500 MHz, D₂O) of compound **25**.



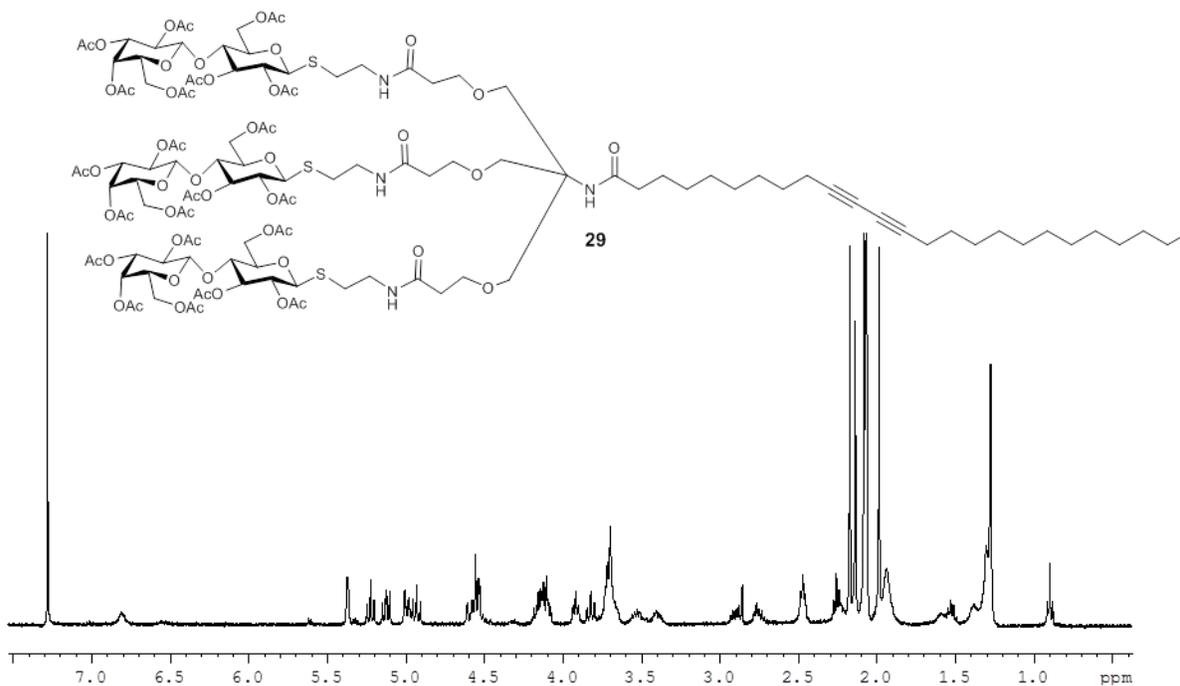
¹³C NMR spectrum (125.7 MHz, D₂O) of compound **25**.



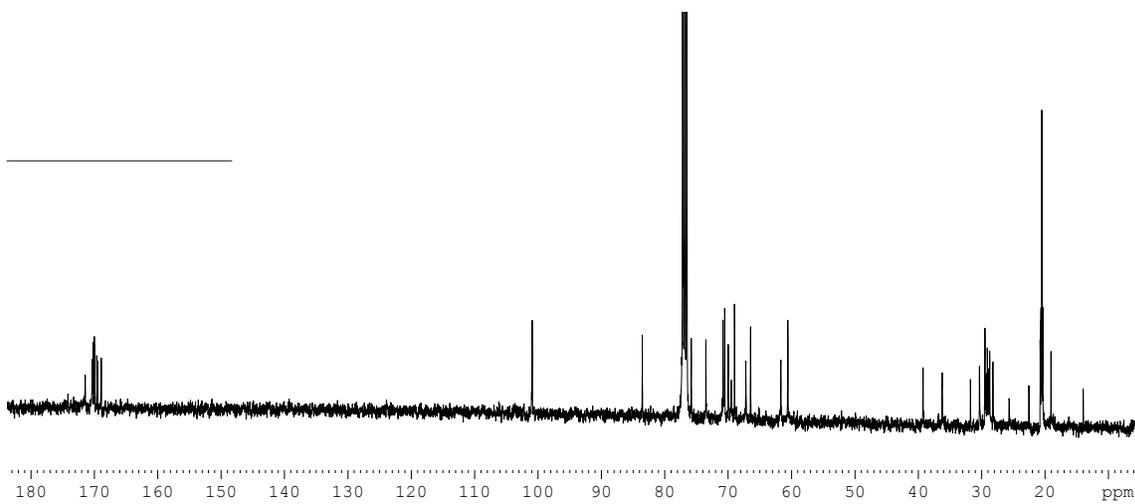
¹H NMR spectrum (500 MHz, CDCl₃) of compound 27.



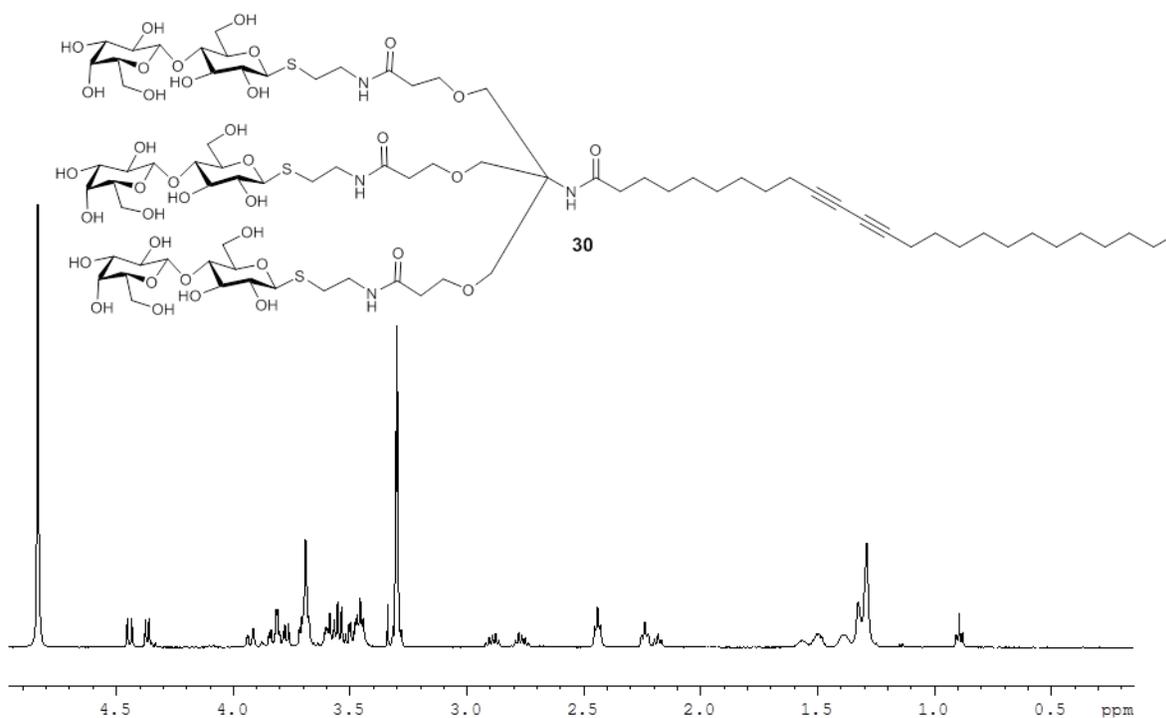
¹³C NMR spectrum (125.7 MHz, CDCl₃) of compound 27.



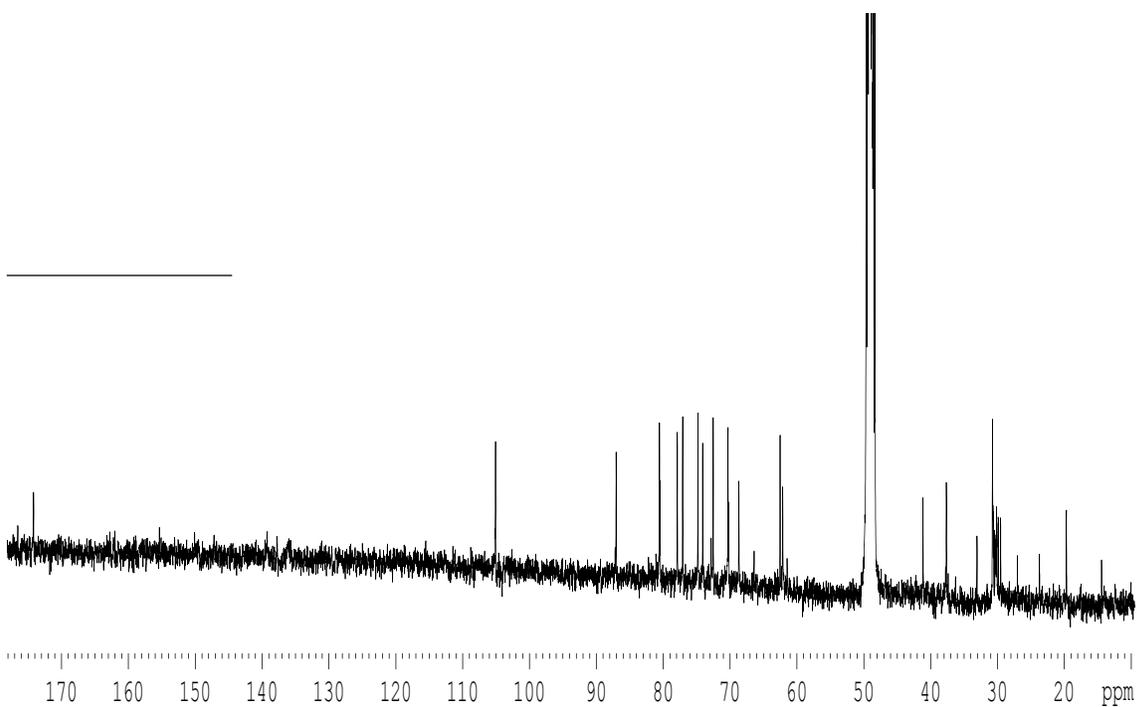
^1H NMR spectrum (500 MHz, CDCl_3) of compound **29**.



^{13}C NMR spectrum (125.7 MHz, CDCl_3) of compound **29**.

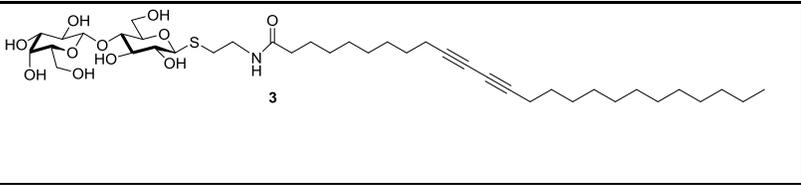
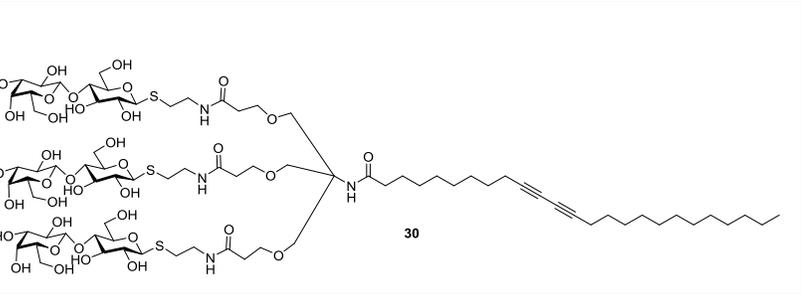
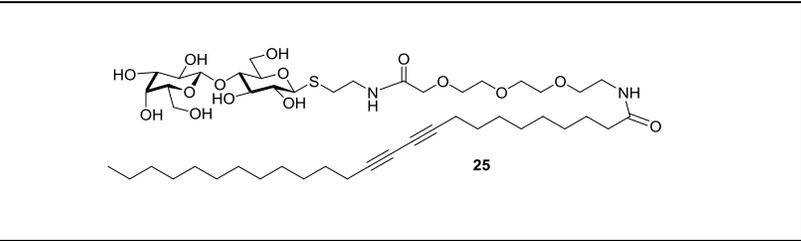
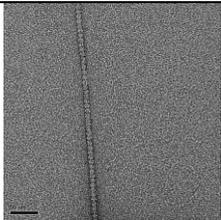
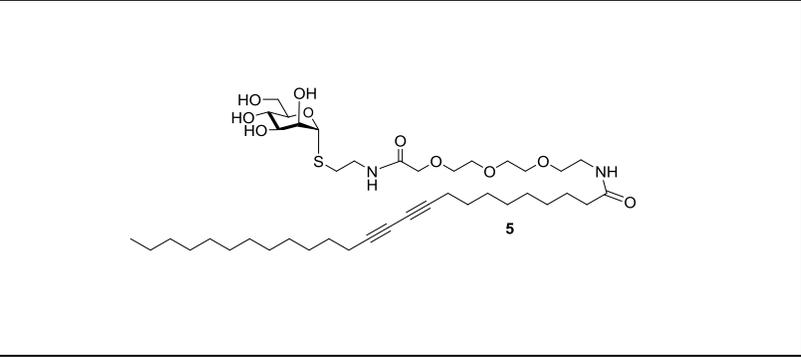
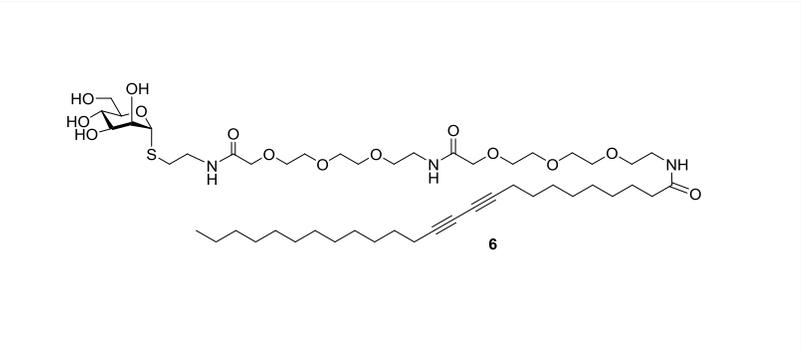
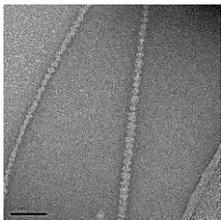


^1H NMR spectrum (500 MHz, D_2O) of compound **30**.



^{13}C NMR spectrum (125.7 MHz, D_2O) of compound **30**.

Table 1 Results of the interaction of glycolipids with CNTs.

Studied Glycolipids	Adsorption on CNTs
 <p>3</p>	No
 <p>30</p>	No
 <p>25</p>	
 <p>5</p>	No
 <p>6</p>	

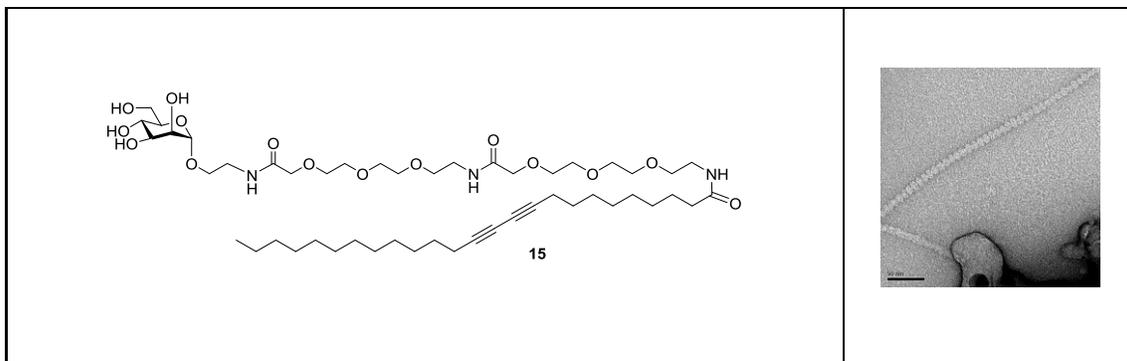
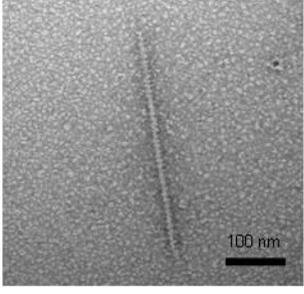
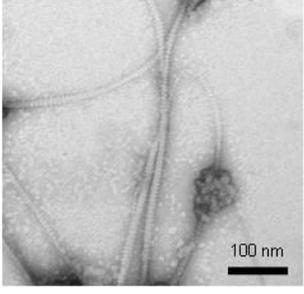
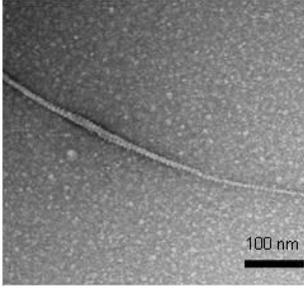
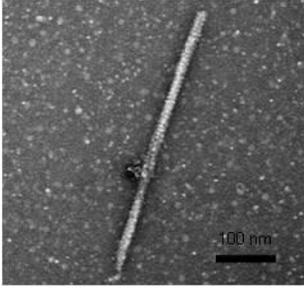


Tabla 2. Degree of functionalization of different SWCNTs with neoglycolipids **4** and **6**.

Glycolípido \ SWNT	4		6	
	Funcionado	TEM	Funcionado	TEM
<i>Aldrich</i>	+		NO	-
<i>Nanocyl</i>	+		NO	-
<i>Carbon solution</i>	++++		+++	

<i>Elicarb</i>	++		++	
<i>Nanolab</i>	++		++	
<i>Mkano</i>	NO	-	NO	-

¹ +++++ > 90%, ++++ > 75%, +++ > 50%, ++ > 25% functionalized.

Figure 2. A) TEM images of: **I)** SWNTs-4-lectin PNA; **II)** Lectin PNA. **B)** TEM images of: **I)** SWNTs-6-lectin Con A; **II)** Lectin Con A.

