SUPPORTING INFORMATION for

N-Oxyamide-linked glycolipid coated AuNP for receptor-targeting imaging and drug delivery

Na Chen, a Zhi-Hao Yu, b Dan Zhou, b Xi-Le Hu, b Yi Zang, c Xiao-Peng He, a,b Jia Li, a,c and Juan Xie a

a PPSM, ENS de Cachan, CNRS, Université Paris-Saclay, Cachan, 94235 France
b Key Laboratory for Advanced Materials & Institute of Fine Chemicals, East China University of Science and Technology, 130 Meilong Rd., Shanghai 200237, PR China
c National Center for Drug Screening, State Key Laboratory of Drug Research, Shanghai Institute of Materia Medica (SIMM), Chinese Academy of Sciences (CAS), 189 Guo Shoujing Rd., Shanghai 201203, PR China

Table of contents
1. Fig. S1-S7
2. Experimental section
3. Original spectral copies of new compounds
1. Fig. S1-S7

**Figure S1.** Plotting the absorbance of CNAu at 520 nm in a 60% fetal bovine serum/40% Tris-HCl solution as a function of time.

**Figure S2.** Dynamic light scattering of AuNP, CNAu and CNAu with PNA (0.3 μM).
**Figure S3.** Zeta potential of AuNP and CNAu.

**Figure S4.** Calibration curve obtained by plotting the absorption (at 620 nm) of anthrone/sulfuric acid as a function of increasing D-galactose. The galactose (galactoglycerolipid) loading concentration was determined to be 21.6 μg / mg AuNP.
Figure S5. UV-vis spectra of (a) 28@AuNP, (b) 30@AuNP and (c) 17@AuNP in the absence and presence of increasing selective lectin (PNA = peanut agglutinin; Con A = concanavalin A). (d) Plotting UV-vis absorption as a function of lectin concentration; the limit of detection of 30@AuNP, 17@AuNP, 28@AuNP and 21@AuNP are 7.1, 8.0, 10.7 and 10.2 nM, respectively (3σ/k).

Figure S6. Fluorescence imaging (a) and quantification (b) of Hep-G2 (human liver cancer), HeLa (human cervix cancer) and A549 (human lung cancer) cells with DCM@AuNP and DCM@CNAu (Scale bar: 100 μM; cell nucleus were stained by Hoechst 33342).
**Figure S7.** Cell viability of different cells treated with (a) increasing HCPT concentration for 15 min, and with (b) HCPT (1 μM) with increasing incubation time. The concentration of CNAu was kept at 0.1 μM.
2. Experimental section

**General.** All commercial available reagents purchased from Sigma-Aldrich, TCI Chemical and Carlo-Erba were used without further purification. Column chromatography was performed on Silica gel 60 (40-60 μm). The solvents for column chromatography were used without purification. The reactions carried out under anhydrous conditions are performed under argon in glassware previously dried in an oven. Methanol is dried over molecular sieve 3Å. THF, DMF, dichloromethane, acetonitrile and toluene were previously dried through alumina cartridge using a solvent purificator MBRAUN SPS-800. Reactions were monitored by TLC on Silica Gel 60F-254 plates with detection by UV (254 nm or 365 nm) or by spraying with 10% H₂SO₄ in EtOH and heating about 30 s at 400-600 °C. Melting points were determined with a Kofler melting point apparatus. Optical rotations were measured using a Jasco P-2000 polarimeter at room temperature in a 10 cm, 1 mL cell. NMR spectra were recorded on a JOEL ESC-400 spectrometer in CDCl₃ or CD₃OD solution. Chemical shift was given in units of parts per million related to TMS or solvent protons as internal reference. High-resolution mass Spectra (HRMS) were recorded on a Q-TOF MaXis using standard conditions or Bruker Microflex™ MALDI-TOF mass spectrometry. Zeta potential was measured on a zetasizer nanosystem (Malvern Instruments). Dynamic light scattering (DLS) was carried out on a Horiba LB-550 Dynamic Light Scattering Nano-Analyzer.

**General procedure for desilylation:** To a solution of silylated compound (1 eq.) in anhydrous THF (15 mL/mmol) under argon, was added TBAF (3 eq.). After stirring at room temperature overnight, the solution was removed under vacuum. The residue was diluted with EtOAc (25 mL/mmol), washed with saturated aq NH₄Cl (10 mL/mmol), H₂O (10 mL/mmol) and brine (10 mL/mmol), dried over MgSO₄, filtered, evaporated, and purified by column chromatography over silica gel to afford the silylated compound.

**General procedure for the Mitsunobu reaction:** To a solution of alcohol (1 eq.), Ph₃P (3 eq.) and PhthNOH (3 eq.) in toluene (20 mL/mmol) at 0 °C under argon, was added DIAD (3 eq.) dropwise. The resulting mixture was stirred at room temperature for 1 h and then extracted with EtOAc (3×30 mL/mmol). The combined organic layers were washed with saturated aq NaHCO₃ (3×30 mL/mmol), H₂O (30 mL/mmol) and brine (30 mL/mmol), dried over MgSO₄, filtered, evaporated and purified by column chromatography over silica gel to afford the O-phthalimido compound.

**General procedure for hydrazinolysis:** To a solution of O-phthalimido compound (1 eq.) in MeOH (20 mL/mmol), was added N₂H₄-H₂O (2 eq.). The mixture was stirred at room temperature for 2 h and then extracted with CH₂Cl₂ (3×50 mL/mmol). The combined organic layers were washed with saturated aq NaHCO₃ (50 mL/mmol), H₂O (50 mL/mmol) and brine (50 mL/mmol), dried over MgSO₄, filtered, evaporated, and purified by column chromatography over silica gel to afford the O-amino compound.

**General procedure for N-oxyamide formation:** To a solution of carboxylic acid (1 eq.) in anhydrous CH₂Cl₂ (15 mL/mmol), was added HOBT (2 eq.), EDC·HCl (2 eq.) and Et₃N (2 eq.) under argon at 0 °C. After stirring for 20 min, the oxyamine derivative (1 eq.) was added. The resulting mixture was stirred at room temperature overnight. The solution was diluted with EtOAc (100
mL/mmol), washed with aq HCl (1N, 2×40 mL/mmol), saturated aq NaHCO₃ (2×40 mL/mmol) and brine (40 mL/mmol), dried over MgSO₄, filtered, evaporated, and purified by column chromatography over silica gel to give the N-oxyamide.

**General procedure for esterification:** To a solution of carboxylic acid (1 eq.) in anhydrous CH₂Cl₂ (15 mL/mmol), were added EDC-HCl (2 eq.), DMAP (2 eq.) and Et₃N (2 eq.) under argon at 0 °C. After the mixture being stirred for 20 min, the alcohol (1 eq.) was added. The resulting mixture was stirred until room temperature overnight. The solution was diluted with CH₂Cl₂ (50 mL/mmol), washed with aq HCl (1N, 2×40 mL/mmol), saturated aq NaHCO₃ (2×40 mL/mmol) and brine (40 mL/mmol), dried over MgSO₄, filtered, evaporated, and purified by column chromatography over silica gel to give the ester.

**General procedure A for the deacetylation:** To a solution of acetylated compound (1 eq.) in 85% EtOH (50 mL/mmol) was added NaOH (0.3 eq.). The mixture was stirred overnight. The solution was diluted with CH₂Cl₂ (15 mL/mmol) was added EDC·HCl (2 eq.), DMAP (2 eq.) and Et₃N (2 eq.) under argon at 50 °C overnight. The solution was poured into ice-cold brine and extracted with CHCl₃ (3×100 mL/mmol). The combined CHCl₃ layers were dried over MgSO₄, filtered and evaporated to give the deacetylated compound.

**General procedure B for the deacetylation:** To a solution of acetylated compound (1eq.) in anhydrous MeOH (20 mL/mmol), was added sodium methoxide (0.3 eq.). After completion of the reaction, cation exchange resin was added to neutralize the solution. The resin was filtered and the filtrate concentrated to afford the deacetylated compound.

**General procedure B for the deacetylation:** To a solution of acetylated compound (1 eq.) in anhydrous MeOH (20 mL/mmol), was added sodium methoxide (0.3 eq.). After completion of the reaction, cation exchange resin was added to neutralize the solution. The resin was filtered and the filtrate concentrated to afford the deacetylated compound.

**(2R)-1,2-Di-O-benzyl-3-O-(2′,3′,4′,6′-tetra-O-acetyl-β-D-galactopyranosyl)glycerol (2)** To a solution of β-D-galactose pentaacetate (2 g, 5.12 mmol) in CH₂Cl₂ (15 mL) was added HBr (33% in AcOH, 3.52 mL, 20.48 mmol) at 0 °C. The mixture was stirred at room temperature under argon for 6 h, and then the solution neutralized with aq NaHCO₃ in an ice bath. The resulting 2,3,4,6-tetra-O-acetyl-α-D-galactopyranosyl bromide was extracted with CH₂Cl₂ (100 mL), dried over MgSO₄, filtered and evaporated to give a syrup. To a solution of (2S)-1,2-di-O-benzyl-glycerol 1 (1.39 g, 5.12 mmol), HgBr₂ (0.92 g, 2.56 mmol) and Hg(CN)₂ (0.65 g, 2.56 mmol) in dry acetonitrile (10 mL) was added dropwise over a period of 1 h, a solution of glycosyl bromide in acetonitrile (5 mL). The reaction solution was stirred at room temperature for 15 h, and then concentrated to an oil which was dissolved in CH₂Cl₂ (100 mL) and washed with saturated aq NaHCO₃ (2×30 mL) and water (30 mL). The dried (MgSO₄) organic layer was concentrated. Purification by column chromatography (petroleum ether/EtOAc: 3/1) afforded compound 2 (2.30 g, 75%) as a colorless paste: Rₚ = 0.20 (petroleum ether/EtOAc: 3/1); [α]D -15.0 (c 0.1, CHCl₃); ¹H NMR (400 MHz, CDCl₃): δ 7.38-7.27 (m, 10H, H-Ph), 5.40-5.34 (m, H-4'), 5.20 (dd, J = 10.5, 7.9 Hz, 1H, H-2'), 4.99 (t, J = 10.5, 3.4 Hz, 1H, H-3'), 4.67-4.62 (m, 2H, OCH₂), 4.56-4.46 (m, 3H, H-1', OCH₂), 4.19-4.08 (m, 2H, H-6'), 3.98 (dd, J = 9.8, 4.2 Hz, 1H, H-3a), 3.86 (td, J = 6.5, 0.8 Hz, 1H, H-5'), 3.78-3.68 (m, 2H, H-2,3b), 3.63-3.55 (m, 2H, H-1), 2.15, 2.04, 1.98, 1.95 (4xs, 12H, 4xOAc); ¹³C NMR (100 MHz, CDCl₃): δ 170.5, 170.4, 170.3, 169.5 (C=O); 138.6, 138.3 (C₀); 128.5, 127.8 (CH-Ph); 101.2 (C-1'), 77.0 (CH), 73.6, 72.3 (CH₂); 71.0 (C-3'), 70.8 (C-5'), 69.9, 69.3 (CH₂); 69.0 (C-2'), 67.2 (C-4'), 61.4 (C-6'), 20.8, 20.7 (OAc); HRMS (ESI) m/z: [M+Na]⁺ Calcd for C₃₁H₃₈NaO₁₂ 625.2261; Found 625.2261.
(2R)-1-O-(2',3',4',6'-Tetra-O-acetyl-β-D-galactopyranosyl)glycerol (3). A mixture of compound 2 (7 g, 11.63 mmol) and 10% Pd/C (1 g) in EtOAc (100 mL) containing 10 mL each of EtOH and AcOH was vigorously shaken under H₂ at room temperature overnight. The catalyst was filtered off, the filtrate evaporated. Purification by column chromatography (CH₂Cl₂/MeOH: 100/3) afforded compound 3 (3.45 g, 70%) as a colourless paste: Rᶠ = 0.22 (CH₂Cl₂/MeOH: 100/3); [α]D + 3.0 (c 0.1, CHCl₃); ¹H NMR (400 MHz, CDCl₃): δ 5.40 (dd, J = 3.7, 0.9 Hz, 1H, H-4'), 5.20 (dd, J = 10.5, 7.8 Hz, 1H, H-2'), 5.03 (dd, J = 10.5, 3.2 Hz, 1H, H-3'), 4.51 (d, J = 8.2 Hz, 1H, H-1'), 4.21-4.10 (m, 2H, H-6'), 3.99-3.92 (m, 1H, H-5'), 3.90-3.75 (m, 3H, H-2,3), 3.70 (dd, J = 11.4, 3.7 Hz, 1H, H-1a), 3.61 (dd, J = 11.4, 4.6 Hz, 1H, H-1b), 2.99 (s, 2H, 2×OH), 2.17, 2.08, 2.07, 2.00 (4×s, 12H, 4×OAc); ¹³C NMR (100 MHz, CDCl₃): δ 170.6, 170.4, 170.3, 170.0 (C=O); 102.0 (C-1'), 72.8 (CH₂), 71.0 (C-5'), 70.7 (C-3'), 70.5 (CH), 68.9 (C-2'), 67.1 (C-4'), 63.5, 61.5 (CH₂), 20.9, 20.8, 20.7 (OAc); HRMS (ESI) m/z: [M+Na]⁺ Calcd for C₁₉H₂₆NaO₁₂ 445.1322; Found 445.1321.

(2S)-1-O-tert-Butyldimethylsilyl-3-O-(2',3',4',6'-tetra-O-acetyl-β-D-galactopyranosyl)glycerol (4). To a solution of compound 3 (2.8 g, 6.64 mmol), N-methylimidazole (1.59 mL, 19.92 mmol) and iodine (5.05 g, 19.92 mmol) in anhydrous THF (30 mL) under argon at 0 °C, was added TBSCI (1.1 g, 7.30 mmol). The reaction mixture was stirred for 20 min at 0 °C, then quenched with saturated aq Na₂S₂O₃ and the solution was evaporated. The residue was diluted with EtOAc (200 mL), washed with saturated aq Na₂S₂O₃ (2×50 mL), H₂O (50 mL) and brine (50 mL), dried over MgSO₄, filtered, concentrated, and purified by column chromatography over silica gel (CH₂Cl₂/MeOH: 100/1) to afford compound 4 (3.20 g, 90%) as a colourless paste: Rᶠ = 0.73 (CH₂Cl₂/MeOH: 100/3); [α]D -7.3 (c 0.1, CHCl₃); ¹H NMR (400 MHz, CDCl₃): δ 5.34 (d, J = 3.5 Hz, 1H, H-4'), 5.16 (dd, J = 10.5, 8.0 Hz, 1H, H-2'), 4.97 (dd, J = 10.5, 3.3 Hz, 1H, H-3'), 4.47 (d, J = 7.8 Hz, 1H, H-1'), 4.13-4.05 (m, 2H, H-6'), 3.93-3.85 (m, 1H, H-5'), 3.80-3.65 (m, 3H, H-2,3), 3.61-3.53 (m, 2H, H-1), 2.54 (s, 1H, OH), 2.11, 2.01, 2.01, 1.93 (4×s, 12H, 4×OAc), 0.85 (s, 9H, tBu), 0.02 (s, 6H, Si(CH₃)₂); ¹³C NMR (100 MHz, CDCl₃): δ 170.4, 170.2, 170.1, 169.5 (C=O); 101.8 (C-1'), 71.6 (CH₂), 70.9, 70.8, 70.5, 68.9, 67.1 (CH); 63.7, 61.4 (CH₂), 25.9, 20.8, 20.7, 20.6 (CH₃); 18.3 (C₀, tBu), -5.4 (CH₃, Si(CH₃)₂); HRMS (ESI) m/z: [M+Na]⁺ Calcd for C₂₃H₄₀NaO₁₂Si 559.2187; Found 559.2188.

(2R)-2-O-Phthalimido-1-O-tert-butyldimethylsilyl-3-O-(2',3',4',6'-tetra-O-acetyl-β-D-galactopyranosyl)glycerol (5). The phthalimidoxyl group was introduced into compound 4 (1.68 g, 3.13 mmol) via Mitsunobu reaction according to the general procedure. Purification by column chromatography over silica gel (CH₂Cl₂/MeOH: 100/0.5) afforded compound 5 (2.06 g, 97%) as a yellowish paste: Rᶠ = 0.60 (CH₂Cl₂/MeOH: 100/1); [α]D + 4.0 (c 0.1, CHCl₃); ¹H NMR (400 MHz, CDCl₃): δ 7.88-7.71 (m, 4H, Phth), 5.36 (dd, J = 3.4, 1.0 Hz, 1H, H-4'), 5.16 (dd, J = 10.6, 7.8 Hz, 1H, H-2'), 5.00 (dd, J = 10.6, 3.6 Hz, 1H, H-3'), 4.65 (d, J = 8.2 Hz, 1H, H-1'), 4.46-4.39 (m, 1H, H-2), 4.18-4.08 (m, 3H, H-3a,6'), 3.98-3.88 (m, 4H, H-1,3b,5'), 2.12, 2.05, 2.04, 1.96 (4×s, 12H, 4×OAc), 0.79 (s, 9H, tBu), 0.00, -0.01 (2×s, 6H, Si(CH₃)₂); ¹³C NMR (100 MHz, CDCl₃): δ 170.5, 170.4, 170.3, 169.7, 163.8 (C=O); 134.5 (CH), 129.1 (C₀), 123.6 (CH), 101.3 (C-1'), 87.0 (CH), 71.1 (C-3'), 70.7 (C-5'), 68.8 (C-2'), 67.9 (CH₂), 67.1 (C-4'), 62.0, 61.4 (CH₂), 25.8, 20.9, 20.8 (CH₃);
18.3(C₆, tBu), -5.6 (CH₃, Si(CH₃)₂); HRMS (ESI) m/z: [M+Na]⁺ Calcd for C₃₁H₄₃NNaO₁₄Si 704.2351; Found 704.2346.

(2S)-2-O-Phthalimido-1-O-(2',3',4',6'-tetra-O-acetyl-β-D-galactopyranosyl)glycerol (6). Desilylation of compound 5 (1.45 g, 2.13 mmol) using AcCl according to the general procedure A and purification by column chromatography over silica gel (petroleum ether/EtOAc: 2/3) gave compound 6 (0.93 g, 77%) as a white solid: Rᶠ = 0.39 (petroleum ether/EtOAc: 1/2); [α]D -17.7 (c 0.1, CHCl₃); mp 66 °C; ¹H NMR (400 MHz, CDCl₃): δ 7.97-7.78 (m, 4H, Phth), 5.40 (d, J = 3.2 Hz, 1H, H-4'), 5.23 (dd, J = 10.6, 8.2 Hz, 1H, H-2'), 5.03 (dd, J = 10.6, 3.2 Hz, 1H, H-3'), 4.65 (d, J = 7.8 Hz, 1H, H-1'), 4.44-4.30 (m, 1H, H-2), 4.28-4.11 (m, 3H, H-3a,6'), 4.05 (dd, J = 11.6, 6.3 Hz, 1H, H-3b), 4.01-3.92 (m, 1H, H-5'), 3.84 (dd, J = 13.8, 2.8 Hz, 1H, H-1a), 3.73 (dd, J = 13.8, 4.1 Hz, 1H, H-1b), 2.16, 2.10, 2.07, 1.99 (4×s, 12H, 4×OAc); ¹³C NMR (100 MHz, CDCl₃): δ 170.6, 170.4, 170.3, 169.8, 164.7 (C=O), 135.1 (CH), 128.6 (C₆), 124.1 (CH), 101.4 (C-1'), 88.2 (CH), 71.0, 70.9, 68.7 (CH); 67.7 (CH₂), 67.2 (CH), 61.4, 60.5 (CH₂); 20.9, 20.8, 20.7 (OAc); HRMS (ESI) m/z: [M+Na]⁺ Calcd for C₃₅H₄₇NNaO₁₆ 590.1486; Found 590.1483.

(2R)-1-O-Palmitoyl-2-O-phthalimido-3-O-(2',3',4',6'-tetra-O-acetyl-β-D-galactopyranosyl)glycerol (7). Esterification of compound 6 (0.93 g, 1.64 mmol) with palmitic acid according to the general procedure and purification by column chromatography over silica gel (petroleum ether/EtOAc: 3/2) afforded compound 7 (1.23 g, 93%) as a colourless paste: Rᶠ = 0.60 (petroleum ether/EtOAc: 1/1); [α]D -18.7 (c 0.1, CHCl₃); ¹H NMR (400 MHz, CDCl₃): δ 7.93-7.75 (m, 4H, Phth), 5.38 (d, J = 3.6 Hz, 1H, H-4'), 5.19 (dd, J = 10.5, 8.2 Hz, 1H, H-2'), 5.02 (dd, J = 10.5, 3.2 Hz, 1H, H-3'), 4.64 (d, J = 8.2 Hz, 1H, H-1'), 4.62-4.55 (m, 1H, H-2), 4.42 (dd, J = 12.8, 3.7 Hz, 1H, H-1a), 4.34 (dd, J = 12.8, 5.9 Hz, 1H, H-1b), 4.23-4.09 (m, 3H, H-3a,6'), 4.01 (dd, J = 11.7, 5.8 Hz, 1H, H-3b), 3.98-3.91 (m, 1H, H-5'), 2.31 (t, J = 7.6 Hz, 1H, CH₂), 2.15, 2.08, 2.06, 1.98 (4×s, 12H, 4×OAc), 1.67-1.54 (m, 2H, CH₂), 1.45-1.21 (m, 24H, 12×CH₂), 0.88 (t, J = 6.9 Hz, 3H, CH₃); ¹³C NMR (100 MHz, CDCl₃): δ 173.5, 170.6, 170.4, 170.3, 169.7, 163.6 (C=O); 134.7 (CH), 128.9 (C₀), 123.8 (CH), 101.3 (C-1'), 84.5 (CH), 71.0 (C-3'), 70.8 (C-5'), 68.6 (C-2'), 67.6 (CH₂), 67.1 (C-4'), 62.4, 61.3, 34.1, 32.0, 29.8, 29.8, 29.7, 29.6, 29.5, 29.4, 29.2, 24.8, 22.8 (CH₂); 20.9, 20.8, 20.7, 14.2 (CH₃); HRMS (ESI) m/z: [M+Na]⁺ Calcd for C₄₉H₇₃NNaO₁₅ 828.3782; Found 828.3788.

(2R)-2-O-Amino-1-O-palmitoyl-3-O-(2',3',4',6'-tetra-O-acetyl-β-D-galactopyranosyl)glycerol (8). Hydrazinolysis of compound 7 (50 mg, 0.062 mmol) under mild conditions according to the general procedure and purification by column chromatography over silica gel (petroleum ether/EtOAc: 2/1) afforded compound 8 (39 mg, 93%) as a white paste: Rᶠ = 0.44 (petroleum ether/EtOAc: 1/1); [α]D -53.0 (c 0.1, CHCl₃); ¹H NMR (400 MHz, CDCl₃): 5.39 (d, J = 3.2 Hz, 1H, H-4'), 5.21 (dd, J = 10.5, 7.8 Hz, 1H, H-2'), 5.00 (dd, J = 10.5, 3.2 Hz, 1H, H-3'), 4.53 (d, J = 8.2 Hz, 1H, H-1'), 4.41-4.33 (m, 1H, H-2), 4.28-4.09 (m, 4H, H-1,6'), 3.97 (dd, J = 11.4, 5.0 Hz, 1H, H-3a), 3.93-3.87 (m, 1H, H-5'), 3.77 (dd, J = 11.2, 6.2 Hz, 1H, H-3b), 2.31 (t, J = 7.6 Hz, 1H, CH₂), 2.16, 2.06, 2.04, 1.99 (4×s, 12H, 4×OAc), 1.71-1.57 (m, 2H, CH₂), 1.35-1.22 (m, 24H, 12×CH₂), 0.88 (t, J = 6.9 Hz, 3H, CH₃); ¹³C NMR (100 MHz, CDCl₃): δ 173.7, 170.6, 170.4, 170.4, 169.6 (C=O); 101.8 (C-1'), 79.1 (CH), 71.0 (C-3'), 70.7 (C-5'), 68.9 (C-2'), 68.4 (CH₂), 67.1 (C-4'), 62.8,
Coupling of compound 8 (115 mg, 0.17 mmol) with hexanoic acid (21 μL, 0.17 mmol) according to the general procedure for N-oxyamide formation, followed by purification by column chromatography over silica gel (petroleum ether/ETHOAc: 2/1) afforded compound 9 (115 mg, 88%) as a colourless paste: Rf = 0.55 (petroleum ether/ETHOAc: 1/1); [α]D -62.0 (c 0.1, CHCl3); 1H NMR (400 MHz, CDCl3): δ 8.86 (s, 1H, NH), 5.37 (t, J = 3.2 Hz, 1H, H-4'), 5.16 (dd, J = 10.3, 8.6 Hz, 1H, H-2'), 5.01 (dd, J = 10.5, 3.2 Hz, 1H, H-3'), 4.50 (d, J = 7.8 Hz, 1H, H-1'), 4.36 (dd, J = 12.2, 3.5 Hz, 1H, H-1a), 4.24-4.04 (m, 4H, H-2,1b,6'), 4.02-3.95 (m, 1H, H-3a), 3.93-3.87 (m, 1H, H-5'), 3.67 (dd, J = 10.1, 7.2 Hz, 1H, H-3b), 2.31 (t, J = 7.6 Hz, 2H, CH2), 2.15-1.93 (m, 14H, 4×OAc, CH2), 1.65-1.53 (m, 4H, 2×CH2), 1.35-1.11 (m, 28H, 14×CH2), 0.91-0.79 (m, 6H, 2×CH3); 13C NMR (100 MHz, CDCl3): δ 174.2, 170.8, 170.4, 170.2, 170.1 (C=O); 101.2 (C-1'), 82.3 (CH), 70.8 (C-5'), 70.6 (C-3'), 69.1 (C-2'), 68.2 (CH2), 66.9 (C-4'), 61.4, 61.2, 34.1, 33.1, 31.9, 31.4, 29.7, 29.6, 29.5, 29.4, 29.3, 29.1, 25.1, 24.9, 23.9, 22.7, 22.4 (CH2); 21.0, 20.9, 20.7, 20.6, 14.2, 14.0 (CH3); HRMS (ESI) m/z: [M+Na]+ Calcd for C39H71NNaO14 796.4459; Found 796.4456.

Coupling of compound 8 (135 mg, 0.20 mmol) with octanoic acid (32 μL, 0.20 mmol) according to the general procedure for N-oxyamide formation, followed by purification by column chromatography over silica gel (petroleum ether/ETHOAc: 2/1) afforded compound 10 (127 mg, 81%) as a yellowish paste: Rf = 0.56 (petroleum ether/ETHOAc: 1/1); [α]D -52.0 (c 0.1, CHCl3); 1H NMR (400 MHz, CDCl3): δ 8.89 (s, 1H, NH), 5.41 (t, J = 3.2 Hz, 1H, H-4'), 5.21 (dd, J = 10.1, 8.3 Hz, 1H, H-2'), 5.05 (dd, J = 10.5, 3.2 Hz, 1H, H-3'), 4.54 (d, J = 7.8 Hz, 1H, H-1'), 4.42 (dd, J = 12.4, 3.2 Hz, 1H, H-1a), 4.26-4.09 (m, 4H, H-2,1b,6'), 4.06-3.98 (m, 1H, H-3a), 3.97-3.90 (m, 1H, H-5'), 3.71 (dd, J = 9.8, 7.4 Hz, 1H, H-3b), 2.36 (t, J = 7.3 Hz, 2H, CH2), 2.19-1.97 (m, 14H, 4×OAc, CH2), 1.69-1.57 (m, 4H, 2×CH2), 1.38-1.20 (m, 32H, 16×CH2), 0.91-0.84 (m, 6H, 2×CH3); 13C NMR (100 MHz, CDCl3): δ 174.3, 170.8, 170.5, 170.3, 170.1 (C=O); 101.2 (C-1'), 82.4 (CH), 70.9 (C-5'), 70.6 (C-3'), 69.1 (C-2'), 68.2 (CH2), 67.0 (C-4'), 61.4, 61.3, 34.2, 33.2, 32.0, 31.8, 29.8, 29.7, 29.6, 29.5, 29.4, 29.3, 29.2, 29.0, 25.5, 24.9, 22.8, 22.7 (CH2); 21.0, 20.8, 20.7, 14.2 (CH3); HRMS (ESI) m/z: [M+Na]+ Calcd for C49H71NNaO14 824.4772; Found 824.4769.

Coupling of compound 8 (184 mg, 0.23 mmol) with 2-ethylhexanoic acid (36 μL, 0.23 mmol) according to the procedure for N-oxyamide formation, followed by purification by column chromatography over silica gel (petroleum ether/ETHOAc: 2/1) afforded compound 11 (143 mg, 78%) as a colourless paste: Rf = 0.45 (petroleum ether/ETHOAc: 1/1); [α]D -54.3 (c 0.1, CHCl3); 1H NMR (400 MHz, CDCl3): δ 8.89 (s, 1H, NH), 5.39 (t, J = 2.8 Hz, 1H, H-4'), 5.18 (dd, J = 10.2, 8.0 Hz, 1H, H-2'), 5.03 (dd, J = 10.5, 3.2 Hz, 1H, H-3'), 4.51 (d, J = 7.8 Hz, 1H, H-1'), 4.46-4.37 (m, 1H, H-1a), 4.26-4.08 (m, 4H, H-2,1b,6'), 3.99 (dd, J = 10.0, 4.4 Hz, 1H, H-3a), 3.95-3.89 (m, 1H, H-5'), 3.70 (dd, J = 10.0, 7.3 Hz, 1H, H-3b), 2.34 (t, J = 7.5 Hz, 2H, CH2), 2.13, 2.08, 2.03, 1.97 (4xs, 12H, 4×OAc), 1.94-1.84 (m, 1H, CH), 1.69-1.55 (m, 4H, 2×CH2),
1.52-1.36 (m, 2H, CH2), 1.36-1.18 (m, 28H, 14×CH2), 0.95-0.82 (m, 9H, 3×CH3); 13C NMR (100 MHz, CDCl3): δ 174.2, 173.4, 170.7, 170.4, 170.2, 170.0 (C=O); 101.1 (C-1’), 82.4 (CH), 70.8 (C-5’), 70.5 (C-3’), 69.1 (C-2’), 68.0 (CH2), 66.9 (C-4’), 61.4, 61.2 (CH2); 45.8 (CH), 34.1, 32.1, 31.9, 29.7, 29.5, 29.4, 29.1, 25.8, 24.8, 22.7 (CH2); 20.9, 20.6, 14.1, 14.0, 12.0 (CH3); HRMS (ESI) m/z: [M+Na]+ Calcd for C43H71NNaO14 824.4772; Found 824.4767.

(2R)-1-O-Palmitoyl-2-O-palmitoylaminol-3-O-(2’,3’,4’,6’-tetra-O-acetyl-β-D-galactopyranosyl)glycerol (12). Coupling of compound 8 (170 mg, 0.25 mmol) with palmitic acid (64 mg, 0.25 mmol) according to the general procedure for N-oxamidate formation, followed by purification by column chromatography over silica gel (petroleum ether/EtOAc: 2/1) afforded compound 12 (204 mg, 90%) as a white solid; Rf = 0.29 (petroleum ether/EtOAc: 2/1); [α]D -38.0 (c 0.1, CHCl3); mp 45 °C; 1H NMR (400 MHz, CDCl3): δ 8.86 (s, 1H, NH), 5.41 (t, J = 2.8 Hz, 1H, H-4’), 5.21 (dd, J = 10.4, 8.3 Hz, 1H, H-2’), 5.05 (dd, J = 10.5, 3.2 Hz, 1H, H-3’), 4.53 (d, J = 7.8 Hz, 1H, H-1’), 4.42 (dd, J = 12.4, 3.2 Hz, 1H, H-1a), 4.26-4.08 (m, 4H, H-2,1b,6’), 4.05-3.98 (m, 1H, H-3a), 3.96-3.89 (m, 1H, H-5’), 3.71 (dd, J = 10.1, 7.3 Hz, 1H, H-3b), 2.36 (t, J = 7.6 Hz, 2H, CH2), 2.18-1.98 (m, 14H, 4×OAc, CH2), 1.68-1.57 (m, 4H, 2×CH2), 1.36-1.17 (m, 48H, 24×CH2), 0.91-0.83 (m, 6H, 2×CH3); 13C NMR (100 MHz, CDCl3): δ 174.3, 170.8, 170.5, 170.3, 170.2 (C=O); 101.3 (C-1’), 82.4 (CH), 70.9 (C-5’), 70.6 (C-3’), 69.1 (C-2’), 68.2 (CH2), 67.0 (C-4’), 61.4, 61.3, 34.2, 33.3, 32.0, 29.8, 29.6, 29.5, 29.4, 29.2, 25.5, 25.0, 22.8 (CH2); 21.1, 20.8, 20.7, 14.3 (CH3); HRMS (ESI) m/z: [M+Na]+ Calcd for C49H87NNaO14 936.6024; Found 936.6024.

(2R)-2-O-Oleoylamino-1-O-palmitoyl-3-O-(2’,3’,4’,6’-tetra-O-acetyl-β-D-galactopyranosyl)glycerol (13). Coupling of compound 8 (115 mg, 0.17 mmol) with oleic acid (48 mg, 0.217 mmol) according to the general procedure for N-oxamidate formation, followed by purification by column chromatography over silica gel (petroleum ether/EtOAc: 2/1) afforded compound 13 (147 mg, 92%) as a colourless paste: Rf = 0.56 (petroleum ether/EtOAc: 1/1); [α]D -42.0 (c 0.1, CHCl3); 1H NMR (400 MHz, CDCl3): δ 8.84 (s, 1H, NH), 5.41 (t, J = 3.2 Hz, 1H, H-4’), 5.39-5.29 (m, 2H, CH=CH), 5.21 (dd, J = 10.6, 7.8 Hz, 1H, H-2’), 5.05 (dd, J = 10.6, 3.6 Hz, 1H, H-3’), 4.53 (d, J = 7.8 Hz, 1H, H-1’), 4.42 (dd, J = 12.4, 3.2 Hz, 1H, H-1a), 4.26-4.09 (m, 4H, H-2,1b,6’), 4.05-3.98 (m, 1H, H-3a), 3.96-3.89 (m, 1H, H-5’), 3.71 (dd, J = 10.5, 7.3 Hz, 1H, H-3b), 2.36 (t, J = 7.6 Hz, 2H, CH2), 2.18-1.96 (m, 16H, 4×OAc, 2×CH2), 1.68-1.57 (m, 4H, 2×CH2), 1.38-1.17 (m, 46H, 23×CH2), 0.91-0.84 (m, 6H, 2×CH3); 13C NMR (100 MHz, CDCl3): δ 174.3, 170.8, 170.5, 170.3, 170.1 (C=O); 130.1, 129.8 (CH=CH); 101.2 (C-1’), 82.4 (CH), 70.9 (C-5’), 70.6 (C-3’), 69.1 (C-2’), 68.2 (CH2), 67.0 (C-4’), 61.4, 61.2, 34.2, 33.2, 32.0, 29.8, 29.6, 29.4, 29.2, 27.3, 25.5, 24.9, 22.8 (CH2); 21.0, 20.8, 20.6, 14.2 (CH3); HRMS (ESI) m/z: [M+Na]+ Calcd for C51H99NNaO14 962.6181; Found 962.6176.

(2R)-3-O-β-D-Galactopyranosyl-2-O-hexanoylamino-1-O-palmitoylgllycerol (14). Deacetylation of 9 (115 mg, 0.15 mmol) according to the general procedure A and purification by column chromatography over silica gel (CH2Cl2/MeOH: 20/1) led to compound 14 (42 mg, 47%) as a yellowish solid: Rf = 0.17 (CH2Cl2/MeOH: 100/8); [α]D -17.3 (c 0.1, MeOH); mp 65 °C; 1H NMR (400 MHz, CD3OD): δ 4.33 (dd, J = 12.1, 4.0 Hz, 1H, H-1a), 4.30-4.22 (m, 2H, H-1’), 4.21-4.15 (m, 1H, H-2), 4.07 (dd, J = 11.4, 4.1 Hz, H-3a), 3.83 (dd, J = 3.2, 0.6 Hz, 1H, H-4’), 3.81-3.69 (m,
3H, H-3b,6''), 3.58-3.46 (m, 3H, H-2',3',5''), 2.36 (t, J = 7.4 Hz, 2H, CH₂), 2.10 (t, J = 7.6 Hz, 2H, CH₂), 1.67-1.56 (m, 4H, 2xCH₂), 1.39-1.24 (m, 28H, 14xCH₂), 0.95-0.87 (m, 6H, 2xCH₃); ¹³C NMR (100 MHz, CD₃OD): δ 175.2, 173.2 (C=O); 105.5 (C-1'), 83.7, 76.8, 74.8, 72.4, 70.2 (CH); 68.9, 63.3, 62.5, 34.9, 33.7, 33.1, 32.4, 30.8, 30.6, 30.5, 30.2, 26.3, 25.9, 23.7, 23.4 (CH₂); 14.5, 14.3 (CH₃); HRMS (ESI) m/z: [M+K⁺] Calcd for C₃₁H₅₉KNO₁₀ 644.3776; Found 644.3778.

(2R)-3-O-β-D-Galactopyranosyl-2-O-octanoylaminol-1-O-palmitoylglycerol (15). Deacetylation of 10 (81 mg, 0.10 mmol) according to the general procedure A and purification by column chromatography over silica gel (CH₂Cl₂/MEOH: 20/1) led to compound 15 (32 mg, 50%) as a yellowish paste: Rf = 0.18 (CH₂Cl₂/MEOH: 100/8); [α]D -17.3 (c 0.1, MeOH); ¹H NMR (400 MHz, CD₃OD): δ 4.33 (dd, J = 12.1, 4.0 Hz, 1H, H-1a), 4.30-4.22 (m, 2H, H-1',1b), 4.21-4.14 (m, 1H, H-2), 4.06 (dd, J = 11.4, 4.1 Hz, H-3a), 3.82 (d, J = 2.8 Hz, 1H, H-4'), 3.81-3.69 (m, 3H, H-3b,6'), 3.59-3.44 (m, 3H, H-2',3',5'), 2.36 (t, J = 7.5 Hz, 2H, CH₂), 2.10 (t, J = 7.4 Hz, 2H, CH₂), 1.67-1.55 (m, 4H, 2xCH₂), 1.39-1.23 (m, 32H, 16xCH₂), 0.95-0.84 (m, 6H, 2xCH₃); ¹³C NMR (100 MHz, CD₃OD): δ 175.3, 173.2 (C=O); 105.5 (C-1'), 83.7, 76.8, 74.8, 72.5, 70.3 (CH); 68.9, 63.3, 62.5, 34.9, 33.8, 33.1, 32.9, 30.8, 30.6, 30.5, 30.2, 30.1, 26.6, 25.9, 23.8, 23.7 (CH₂); 14.5 (CH₃); HRMS (ESI) m/z: [M+Na⁺] Calcd for C₃₃H₆₉NaNO₁₀ 656.4350; Found 656.4346.

(2R)-2-O-(2-Ethylhexanoyl)amino-3-O-β-D-galactopyranosyl-1-O-palmitoylglycerol (16). Deacetylation of 11 (64 mg, 0.08 mmol) according to the general procedure A and purification by column chromatography over silica gel (CH₂Cl₂/MEOH: 20/1) led to compound 16 (26 mg, 52%) as a yellowish paste: Rf = 0.25 (CH₂Cl₂/MEOH: 100/8); [α]D -17.5 (c 0.1, MeOH); ¹H NMR (400 MHz, CD₃OD): δ 4.34 (dd, J = 12.2, 4.2 Hz, 1H, H-1a), 4.30-4.23 (m, 2H, H-1',1b), 4.22-4.14 (m, 1H, H-2), 4.12-4.05 (m, H-3a), 3.83 (d, J = 3.2 Hz, 1H, H-4'), 3.81-3.70 (m, 3H, H-3b,6'), 3.60-3.45 (m, 3H, H-2',3',5'), 2.37 (t, J = 7.5 Hz, 2H, CH₂), 2.02-1.92 (m, 1H, CH), 1.69-1.54 (m, 4H, 2xCH₂), 1.52-1.22 (m, 30H, 15xCH₂), 0.97-0.87 (m, 9H, 3xCH₃); ¹³C NMR (100 MHz, CD₃OD): δ 175.7, 175.2 (C=O); 105.6 (C-1'), 83.9, 76.8, 74.9, 72.5, 70.2 (CH); 68.9, 63.2, 62.5 (CH₂); 46.6 (CH), 34.8, 33.3, 33.1, 30.8, 30.6, 30.5, 30.2, 26.9, 25.9, 23.7, 23.7 (CH₂); 14.5, 14.3, 12.4 (CH₃); HRMS (ESI) m/z: [M+Na⁺] Calcd for C₃₃H₆₉NaNO₁₀ 656.4350; Found 656.4353.

(2R)-3-O-β-D-Galactopyranosyl-1-O-palmitoyl-2-O-palmitoylaminoglycerol (17). Deacetylation of 12 (55 mg, 0.06 mmol) according to the general procedure A and purification by column chromatography over silica gel (CH₂Cl₂/MEOH: 20/1) led to compound 17 (26 mg, 58%) as a white powder: Rf = 0.38 (CH₂Cl₂/MEOH: 10/1); [α]D -13.3 (c 0.1, MeOH); mp 117 °C; ¹H NMR (400 MHz, CD₃OD): δ 4.34 (dd, J = 12.1, 4.0 Hz, 1H, H-3a), 4.31-4.23 (m, 2H, H-1',3b), 4.22-4.15 (m, 1H, H-2), 4.06 (dd, J = 11.4, 4.1 Hz, H-1a), 3.87-3.82 (m, 1H, H-4'), 3.82-3.70 (m, 3H, H-1b,6'), 3.61-3.45 (m, 3H, H-2',3',5'), 2.36 (t, J = 7.4 Hz, 2H, CH₂), 2.10 (t, J = 7.3 Hz, 2H, CH₂), 1.71-1.57 (m, 4H, 2xCH₂), 1.42-1.24 (m, 48H, 24xCH₂), 0.97-0.87 (m, 6H, 2xCH₃); ¹³C NMR (100 MHz, CD₃OD): δ 175.2, 173.2 (C=O); 105.6 (C-1'), 83.8, 76.8, 75.0, 72.5, 70.3 (CH); 68.9, 63.4, 62.6, 34.9, 33.8, 33.1, 33.0, 30.8, 30.6, 30.5, 30.4, 30.2, 26.6, 25.9, 23.8, 23.7 (CH₂); 14.5, 14.4 (CH₃); HRMS (ESI) m/z: [M+Na⁺] Calcd for C₄₁H₇₉NaNO₁₀ 768.5602; Found 768.5606.

(2R)-3-O-β-D-Galactopyranosyl-2-O-oleoylaminol-1-O-palmitoylaminoglycerol (18). Deacetylation of 13 (80 mg, 0.085 mmol) according to the general procedure A and purification by
column chromatography over silica gel (CH$_2$Cl$_2$/MeOH: 20/1) led to compound 18 (36 mg, 55%) as a white solid: $R_f = 0.16$ (CH$_2$Cl$_2$/MeOH: 100/8); $[\alpha]_D -14.0$ (c 0.1, MeOH); mp 114 °C; $^1$H NMR (400 MHz, CD$_3$OD): $\delta$ 4.59-4.72 (m, 2H, CH=CH), 4.34 (dd, $J = 11.9$, 3.7 Hz, 1H, H-3a), 4.31-4.23 (m, 2H, H-1',3b), 4.20-4.15 (m, 1H, H-2), 4.07 (dd, $J = 11.4$, 4.1 Hz, H-1a), 3.85 (d, $J = 2.3$ Hz, 1H, H-4'), 3.83-3.71 (m, 3H, H-1b,6'), 3.60-3.45 (m, 3H, H-2',3',5'), 2.36 (t, $J = 7.6$ Hz, 2H, CH$_2$), 2.10 (t, $J = 6.9$ Hz, 2H, CH$_2$), 1.68-1.57 (m, 4H, 2xCH$_2$), 1.39-1.21 (m, 48H, 24xCH$_2$), 0.93-0.84 (m, 6H, 2xCH$_3$); $^{13}$C NMR (100 MHz, CD$_3$OD): $\delta$ 171.5, 173.0 (C=O); 132.2, 129.7 (CH=CH), 105.3 (C-1'), 83.6, 76.5, 74.6, 72.2, 70.0 (CH); 68.8, 63.2, 62.3, 34.8, 33.7, 32.9, 30.6, 30.4, 30.3, 30.1, 26.4, 25.7, 23.6 (CH$_2$); 14.4, 14.0 (CH$_3$); HRMS (ESI) $m/z$: [M+Na]$^+$ Calced for C$_{43}$H$_{81}$NNaO$_{10}$ 794.5758; Found 794.5759.

(2S)-1-O-$\beta$-D-Galactopyranosyl-2-O-(N-hexanoyl-N'$'$-palmitoyl)aminoglycerol  (19).

Deacetylation of compound 9 (94 mg, 0.12 mmol) with Na/MeOH according to the general procedure B led to compound 19 (70 mg, 96%) as a yellowish paste: $R_f = 0.27$ (CH$_2$Cl$_2$/MeOH: 5/1); $[\alpha]_D -23.3$ (c 0.1, MeOH); $^1$H NMR (400 MHz, CD$_3$OD): $\delta$ 4.29 (d, 1H, d, $J = 7.8$ Hz, H-1'), 4.03 (dd, $J = 11.0$, 4.0 Hz, 1H, H-1a), 4.01-3.95 (m, 1H, H-2), 3.85-3.81 (m, 1H, H-4'), 3.80-3.71 (m, 3H, H-1b,6'), 3.71-3.61 (m, 2H, H-3), 3.58-3.46 (m, 3H, H-2',3',5'), 2.31 (t, $J = 7.5$ Hz, 2H, CH$_2$), 2.12 (t, $J = 7.4$ Hz, 2H, CH$_2$), 1.68-1.55 (m, 4H, 2xCH$_2$), 1.43-1.23 (m, 28H, 14xCH$_2$), 0.97-0.86 (m, 6H, 2xCH$_3$); $^{13}$C NMR (100 MHz, CD$_3$OD): $\delta$ 175.9, 173.8 (C=O); 105.4 (C-1'), 87.5, 76.8, 74.9, 72.5, 70.3 (CH); 69.0, 62.5, 61.0 34.8, 33.6, 33.1, 32.4, 30.8, 30.7, 30.6, 30.5, 30.4, 30.2, 26.3, 26.0, 23.7, 23.4 (CH$_2$); 14.5, 14.3 (CH$_3$); HRMS (ESI) $m/z$: [M+Na]$^+$ Calced for C$_{31}$H$_{59}$NaNO$_{10}$ 628.4037; Found 628.4034.

(2S)-1-O-$\beta$-D-Galactopyranosyl-2-O-(N-(2-ethylhexanoyl)-N'$'$-palmitoyl)aminoglycerol  (20).

Deacetylation of compound 10 (92 mg, 0.115 mmol) with Na/MeOH according to the general procedure B led to compound 20 (72 mg, 99%) as a yellowish paste: $R_f = 0.33$ (CH$_2$Cl$_2$/MeOH: 5/1); $[\alpha]_D -17.3$ (c 0.1, MeOH); $^1$H NMR (400 MHz, CD$_3$OD): $\delta$ 4.29 (d, 1H, d, $J = 7.8$ Hz, H-1'), 4.05 (dd, $J = 11.0$, 3.7 Hz, 1H, H-1a), 4.02-3.95 (m, 1H, H-2), 3.83 (d, $J = 3.2$ Hz, 1H, H-4'), 3.81-3.71 (m, 3H, H-1b,6'), 3.71-3.62 (m, 2H, H-3), 3.60-3.47 (m, 3H, H-2',3',5'), 2.31 (t, $J = 7.4$ Hz, 2H, CH$_2$), 2.04-1.94 (m, 1H, CH), 1.67-1.54 (m, 4H, 2xCH$_2$), 1.52-1.39 (m, 2H, CH$_2$), 1.39-1.21 (m, 28H, 14xCH$_2$), 0.98-0.86 (m, 9H, 3xCH$_3$); $^{13}$C NMR (100 MHz, CD$_3$OD): $\delta$ 176.3, 175.8 (C=O); 105.4 (C-1'), 87.6, 76.7, 74.9, 72.5, 70.2 (CH); 68.9, 62.5, 60.9 (CH$_2$); 46.6 (CH), 34.8, 33.2, 33.1, 30.8, 30.7, 30.6, 30.5, 30.4, 30.2, 26.9, 26.0, 23.7, 23.6 (CH$_2$); 14.5, 14.3, 12.3 (CH$_3$); HRMS (ESI) $m/z$: [M+K]$^+$ Calced for C$_{33}$H$_{63}$KNO$_{10}$ 672.4089; Found 672.4088.

(2S)-1-O-$\beta$-D-Galactopyranosyl-2-O-palmitoylaminoglycerol  (21).

Deacetylation of compound 12 (50 mg, 0.055 mmol) with Na/MeOH according to the general procedure B led to compound 21 (25 mg, 68%) as a yellowish solid: $R_f = 0.44$ (CH$_2$Cl$_2$/MeOH: 5/1); $[\alpha]_D -25.7$ (c 0.1, MeOH); mp 91 °C; $^1$H NMR (400 MHz, CD$_3$OD): $\delta$ 4.29 (d, 1H, d, $J = 7.4$ Hz, H-1'), 4.04 (dd, $J = 11.0$, 4.0 Hz, 1H, H-1a), 4.01-3.96 (m, 1H, H-2), 3.83 (d, $J = 2.8$ Hz, 1H, H-4'), 3.81-3.71 (m, 3H, H-1b,6'), 3.71-3.62 (m, 2H, H-3), 3.59-3.46 (m, 3H, H-2',3',5'), 2.13 (t, $J = 7.4$ Hz, 2H, CH$_2$), 1.68-1.56 (m, 2H, CH$_2$), 1.42-1.22 (m, 24H, 12xCH$_2$), 0.95-0.85 (m, 3H, CH$_3$); $^{13}$C NMR (100 MHz, CD$_3$OD): $\delta$ 173.9 (C=O), 105.4 (C-1'), 87.4, 76.8, 74.9, 72.5, 70.3 (CH); 69.0, 62.5, 60.9, 33.7, 33.1, 30.8, 30.6, 30.5,
(2R)-2-O-Amino-1-O-tert-butyldimethylsilyl-3-O-(2',3',4',6'-tetra-O-acetyl-β-D-glucopyranosyl) glycerol (23). Hydrazinolysis of compound 22 (123 mg, 0.18 mmol) by hydrazine under mild conditions according to the general procedure and purification by column chromatography over silica gel (petroleum ether/EtOAc: 2/1) afforded compound 23 (91 mg, 92%) as a colourless paste: Rf = 0.39 (petroleum ether/EtOAc: 1/1); [α]D -52.3 (c 0.1, CHCl3); 1H NMR (400 MHz, CDCl3): δ 5.20 (t, J = 9.4 Hz, 1H, H-3'), 5.07 (t, J = 9.9 Hz, 1H, H-4'), 5.00 (dd, J = 9.4, 8.0 Hz, 1H, H-2'), 4.58 (d, J = 7.8 Hz, 1H, H-1'), 4.29-4.08 (m, 2H, H-6'), 3.98 (dd, J = 10.6, 3.3 Hz, 1H, H-3a), 3.78-3.62 (m, 5H, H-1,2,3b,5'), 2.07, 2.05, 2.02, 2.00 (4×s, 12H, 4×OAc), 0.88 (s, 9H, tBu), 0.05 (s, 6H, Si(CH3)3); 13C NMR (100 MHz, CDCl3): δ 170.8, 170.4, 169.5 (C=O); 101.2 (C-1'), 83.8 (CH), 72.9 (C-3'), 71.9 (C-5'), 71.4 (C-2'), 68.5 (C-4'), 68.3, 62.0, 61.5 (CH2); 25.9, 20.8, 20.7 (CH3); 18.4 (Cq, tBu), -5.3 (CH3, Si(CH3)3). HRMS (ESI) m/z: [M+K]+ Calcd for C23H49KNO13Si 590.2035; Found 590.2037.

(2S)-2-O-Octanoylamino-1-O-(2’,3’,4’,6’-tetra-O-acetyl-β-D-glucopyranosyl)glycerol (24). Coupling of oxyamine compound 23 (102 mg, 0.186 mmol) with octanoic acid according to the general procedure and purification by column chromatography over silica gel (petroleum ether/EtOAc: 1/2) afforded compound 25 (58 mg, 56%, two steps) as a white solid: Rf = 0.12 (petroleum ether/EtOAc: 1/1); [α]D -103.7 (c 0.1, CHCl3); mp 108 °C; 1H NMR (400 MHz, CDCl3): δ 9.27 (s, 1H, NH), 5.23 (t, J = 9.6 Hz, 1H, H-3'), 5.05 (t, J = 9.9 Hz, 1H, H-4'), 4.99 (dd, J = 9.7, 7.9 Hz, 1H, H-2'), 4.48 (d, J = 8.2 Hz, 1H, H-1'), 4.24 (dd, J = 12.4, 4.6 Hz, 1H, H-6'a), 4.14 (dd, J = 12.4, 2.3 Hz, 1H, H-6'b), 4.04-3.90 (m, 1H, H-1a,2), 3.77-3.67 (m, 1H, H-5'), 3.63-3.47 (m, 3H, H-1b,3), 2.24-1.96 (m, 14H, 4×OAc, CH2), 1.69-1.57 (m, 2H, CH2), 1.41-1.19 (m, 8H, 4×CH2), 0.85 (t, J = 6.5 Hz, 3H, CH3); 13C NMR (100 MHz, CDCl3): δ 172.6, 172.4, 170.6, 170.5, 169.6 (C=O); 101.1 (C-1'), 88.8 (CH), 72.1 (C-3'), 72.0 (C-5'), 71.8 (C-2'), 69.4 (CH2), 68.3 (C-4'), 61.8, 60.4, 33.0, 31.7, 29.1, 29.0, 25.3, 22.7 (CH2); 21.0, 20.8, 20.7, 14.1 (CH3); HRMS (ESI) m/z: [M+Na]+ Calcd for C25H51N4NaO13 586.2476; Found 586.2474.

(2S)-2-O-Palmitoylamino-1-O-(2’,3’,4’,6’-tetra-O-acetyl-β-D-glucopyranosyl)glycerol (25). Coupling of oxyamine compound 23 (135 mg, 0.245 mmol) with palmitic acid according to the general procedure and purification by column chromatography over silica gel (petroleum ether/EtOAc: 1/1) afforded compound 25 (78 mg, 47%, two steps) as a white solid: Rf = 0.14 (petroleum ether/EtOAc: 1/1); [α]D -66.7 (c 0.1, CHCl3); mp 117 °C; 1H NMR (400 MHz, CDCl3): δ 9.19 (s, 1H, NH), 5.24 (t, J = 9.6 Hz, 1H, H-3'), 5.06 (t, J = 9.8 Hz, 1H, H-4'), 5.00 (dd, J = 9.6, 7.8 Hz, 1H, H-2'), 4.48 (d, J = 7.8 Hz, 1H, H-1'), 4.25 (dd, J = 12.8, 4.6 Hz, 1H, H-6'a), 4.14 (dd, J = 12.4, 1.8 Hz, 1H, H-6'b), 4.01-3.91 (m, 2H, H-1a, 2), 3.74-3.67 (m, 1H, H-5'), 3.61-3.48 (m, 3H, H-1b,3), 2.16-1.96 (m, 14H, 4×OAc, CH2), 1.68-1.56 (m, 2H, CH2), 1.34-1.17 (m, 24H, 12×CH2), 0.85 (t, J = 6.9 Hz, 3H, CH3);
13C NMR (100 MHz, CDCl3): δ 172.6, 171.1, 170.8, 170.2, 169.6 (C=O); 101.2 (C-1’), 89.0 (CH), 72.2 (C-3’), 72.0 (C-5’), 71.8 (C-2’), 69.4 (CH2), 68.3 (C-4’), 61.8, 60.4, 33.0, 32.0, 29.7, 29.6, 29.4, 29.3, 25.4, 22.8 (CH2); 21.1, 20.9, 20.7, 14.2 (CH3); HRMS (ESI) m/z: [M+Na]+ Calcd for C33H57NNaO13 698.3728; Found 698.3734.

(2S)-2-O-Oleoylamo-1-O-(2’’3’,4’,6’’-tetra-o-acetyl-β-D-glucopyranosyl)glycerol (26). Coupling of oxamine compound 23 (110 mg, 0.20 mmol) with oleic acid according to the general procedure led to the corresponding N-oxamid which was used directly for the next step without purification. Desilylation of this crude compound using TBAF according to the general procedure and purification by column chromatography over silica gel (petroleum ether/EtOAc: 1/1) afforded compound 26 (69 mg, 49%, two steps) as a white solid: Rf = 0.16 (petroleum ether/EtOAc: 1/1); [α]D

72.7 (c 0.1, CHCl3); mp 85 °C; 1H NMR (400 MHz, CDCl3): δ 9.21 (s, 1H, NH), 5.42-5.28 (m, 2H, CH=CH), 5.24 (t, J = 9.6 Hz, 1H, H-3’), 5.10-4.96 (m, 2H, H-2’,4’), 4.48 (d, J = 7.8 Hz, 1H, H-1’), 4.30-4.11 (m, 2H, H-6’), 4.03-3.91 (m, 2H, H-1a,2), 3.76-3.67 (m, 1H, H-5’), 3.63-3.48 (m, 3H, H-1b,3), 2.24-1.93 (m, 16H, 4xOAc, 2xCH2), 1.69-1.57 (m, 2H, CH2), 1.46-1.12 (m, 22H, 11xCH2), 0.85 (t, J = 6.6 Hz, 3H, CH3); 13C NMR (100 MHz, CDCl3): δ 172.6, 171.1, 170.8, 170.1, 169.5 (C=O); 130.1, 129.8 (CH=CH); 101.1 (C-1’), 88.9 (CH), 72.1 (C-3’), 72.0 (C-5’), 71.8 (C-2’), 69.4 (CH2), 68.3 (C-4’), 61.8, 60.4, 33.0, 32.0, 29.8, 29.6, 29.4, 29.2, 29.0, 27.3, 25.3, 22.7 (CH2); 21.0, 20.8, 20.7, 14.2 (CH3); HRMS (ESI) m/z: [M+Na]+ Calcd for C33H57NNaO13 724.3884; Found 724.3878.

(2S)-1-O-β-D-Glucopyranosyl-2-O-octanoylamino-glycerol (27). Deacetylation of compound 24 (28 mg, 0.05 mmol) with Na/MeOH according to the general deacetylation procedure B led to compound 27 (17 mg, 89%) as a yellowish paste: Rf = 0.44 (CH2Cl2/MeOH: 5/1); [α]D

-63.3 (c 0.1, CHCl3); 1H NMR (400 MHz, CD3OD): δ 4.32 (d, J = 7.8 Hz, 1H, H-1’), 4.03 (dd, J = 11.0, 3.8 Hz, 1H, H-1a), 4.00-3.93 (m, 1H, H-2), 3.87 (d, J = 11.6 Hz, 1H, H-6’a), 3.79-3.60 (m, 4H, H-1b,3,6’b), 3.41-3.17 (m, 4H, H-2’,3’,4’,5’), 2.11 (t, J = 7.3 Hz, 2H, CH2), 1.68-1.54 (m, 2H, CH2), 1.41-1.20 (m, 8H, 4xCH2), 0.90 (t, J = 6.4 Hz, 3H, CH3); 13C NMR (100 MHz, CD3OD): δ 174.0 (C=O), 104.7 (C-1’), 87.1, 78.0, 75.0, 71.6 (CH); 69.0, 62.7, 61.0, 33.7, 32.9, 30.8, 30.2, 26.1, 23.7 (CH2); 14.4 (CH3); HRMS (ESI) m/z: [M+Na]+ Calcd for C31H49NNaO9 418.2053; Found 418.2054.

(2S)-3-O-β-D-Glucopyranosyl-2-O-palmitoylamino-glycerol (28). Deacetylation of compound 25 (48 mg, 0.071 mmol) with Na/MeOH according to the general deacetylation procedure B led to compound 28 (36 mg, 100%) as a yellowish paste: Rf = 0.62 (CH2Cl2/MeOH: 5/1); [α]D

+45.0 (c 0.1, CHCl3); 1H NMR (400 MHz, CD3OD): δ 4.33 (d, J = 7.8 Hz, 1H, H-1’), 4.04 (dd, J = 11.0, 4.1 Hz, 1H, H-1a), 4.01-3.94 (m, 1H, H-2), 3.91-3.84 (m, 1H, H-6’a), 3.78-3.60 (m, 4H, H-1b,3,6’b), 3.40-3.24 (m, 3H, H-3’,4’,5’), 3.21 (dd, J = 9.1, 7.9 Hz, 1H, H-2’), 2.12 (t, J = 7.3 Hz, 2H, CH2), 1.66-1.55 (m, 2H, CH2), 1.38-1.21 (m, 24H, 12xCH2), 0.90 (t, J = 6.9 Hz, 3H, CH3); 13C NMR (100 MHz, CD3OD): δ 173.9 (C=O), 104.7 (C-1’), 87.4, 78.0, 75.0, 71.6 (CH); 69.0, 62.7, 60.9, 33.7, 33.1, 30.8, 30.6, 30.5, 30.4, 30.2, 26.6, 23.7 (CH2); 14.5 (CH3); HRMS (ESI) m/z: [M+Na]+ Calcd for C43H69NNaO9 530.3305; Found 530.3306.

(2S)-3-O-β-D-Glucopyranosyl-2-O-oleoylamino-glycerol (29). Deacetylation of compound 26 (49 mg, 0.07 mmol) with Na/MeOH according to the general deacetylation procedure B led to
compound 29 (37 mg, 99%) as a yellowish paste. Rf = 0.51 (CH$_2$Cl$_2$/MeOH: 5/1); [α]$_D$ -22.3 (c 0.1, CHCl$_3$); $^1$H NMR (400 MHz, CD$_3$OD): δ 5.40-5.29 (m, 2H, CH=CH), 4.01-3.94 (m, 1H, H-1'), 4.04 (dd, J = 11.1, 4.1 Hz, 1H, H-1a), 4.33 (d, J = 7.8 Hz, 1H, H-6'a), 3.78-3.60 (m, 4H, H-1b,3,6'b), 3.39-3.24 (m, 3H, H-3',4',5'); 2.12 (t, J = 7.4 Hz, 2H, CH$_2$), 2.07-1.97 (m, 4H, 2×CH$_2$), 1.66-1.55 (m, 2H, CH$_2$), 1.41-1.19 (m, 20H, 10×CH$_2$), 0.90 (t, J = 6.6 Hz, 3H, CH$_3$); $^{13}$C NMR (100 MHz, CD$_3$OD): δ 173.9 (C=O), 130.9, 130.8 (CH=CH); 104.7 (C-1'), 87.3, 78.0, 75.0, 71.6 (CH); 69.0, 62.7, 60.9, 33.7, 33.1, 30.8, 30.6, 30.5, 30.3, 30.2, 28.1, 26.6, 23.7 (CH$_2$); 14.5 (CH$_3$); HRMS (ESI) m/z: [M+Na]$^+$ Calcd for C$_{27}$H$_{51}$NNaO$_9$ 556.3462; Found 556.3463.

**Preparation of glyco-AuNPs.** A solution of SH-PEG-functionalized AuNP in Tris-HCl (20 mL) (Anal. Chem. 2015, 87, 9078) was mixed with an aqueous solution of 17, 18, 28 or 21 (10 μM, 600 μL). The mixture was stirred overnight and then centrifuged at 8000 rpm for 20 min to remove uncoated glycolipid. The resulting glyco-AuNPs were dispersed in Tris-HCl buffer for detection of proteins. Absorption spectra were measured on a Varian Cary 500 UV-Vis spectrophotometer.

**Cell imaging.** Cells at a seeding density of 15,000 cells/well in μ-Clear 96-well plate were incubated with medium containing DCM (1 μM) or DCM@CNAu (1/0.1 μM) for 15 min at 37 °C with 5% CO$_2$, and then incubated with Hoechst 33342 for another 10 min. Cell images were obtained by Operetta (Perkin Elmer, U.S.A.) after washing with PBS (excitation at 360-400 nm for Hoechst and 460-490 nm for DCM). Fluorescence quantification was carried out by Columbus (Perkin Elmer).

**Cell viability.** Human hepatocellular carcinoma cell line Hep-G2 and human cervical carcinoma cell line HeLa were cultured in DMEM-HG medium with 10% fetal bovine serum (FBS) at 37 °C with 5% CO$_2$. Human lung carcinoma cell line A549 was cultured in Ham's F-12 medium with 10% fetal bovine serum (FBS) at 37 °C with 5% CO$_2$. Cells at a seeding density of 8,000 cells/well in 96-well plate were incubated with medium containing HCPT (1 μM) or HCPT@CNAu (1/0.1 μM) for 15 min, and then cultured in growth medium for 72 h. Then, cell viability was determined by MTS assay (Cell Titer 96® AQueous Assay).
3. Original spectral copies of new compounds

\(^1\)H NMR, \(^{13}\)C NMR, Dept-135, COSY, HMQC of 2 in CDCl\(_3\)................................. S18-21
\(^1\)H NMR, \(^{13}\)C NMR, Dept-135, COSY, HETCOR of 3 in CDCl\(_3\)................................. S21-23
\(^1\)H NMR, \(^{13}\)C NMR, Dept-135, COSY, HMQC of 4 in CDCl\(_3\)................................. S23-26
\(^1\)H NMR, \(^{13}\)C NMR, Dept-135, COSY, HMQC of 5 in CDCl\(_3\)................................. S26-28
\(^1\)H NMR, \(^{13}\)C NMR, Dept-135, COSY, HETCOR of 6 in CDCl\(_3\)................................. S28-30
\(^1\)H NMR, \(^{13}\)C NMR, Dept-135, COSY, HETCOR of 7 in CDCl\(_3\)................................. S30-32
\(^1\)H NMR, \(^{13}\)C NMR, Dept-135, COSY, HETCOR of 8 in CDCl\(_3\)................................. S33-35
\(^1\)H NMR, \(^{13}\)C NMR, Dept-135, COSY, HETCOR of 9 in CDCl\(_3\)................................. S35-37
\(^1\)H NMR, \(^{13}\)C NMR, Dept-135, COSY, HETCOR of 10 in CDCl\(_3\)................................. S38-40
\(^1\)H NMR, \(^{13}\)C NMR, Dept-135, COSY, HETCOR of 11 in CDCl\(_3\)................................. S40-42
\(^1\)H NMR, \(^{13}\)C NMR, Dept-135, COSY, HETCOR of 12 in CDCl\(_3\)................................. S43-45
\(^1\)H NMR, \(^{13}\)C NMR, Dept-135, COSY, HETCOR of 13 in CDCl\(_3\)................................. S45-47
\(^1\)H NMR, \(^{13}\)C NMR, Dept-135, COSY, HETCOR of 14 in CD\(_2\)OD................................. S48-50
\(^1\)H NMR, \(^{13}\)C NMR, Dept-135, COSY, HETCOR of 15 in CD\(_2\)OD................................. S50-52
\(^1\)H NMR, \(^{13}\)C NMR, Dept-135, COSY, HETCOR of 16 in CD\(_2\)OD................................. S53-55
\(^1\)H NMR, \(^{13}\)C NMR, Dept-135, COSY, HETCOR of 17 in CD\(_2\)OD................................. S55-57
\(^1\)H NMR, \(^{13}\)C NMR, Dept-135, COSY, HETCOR of 18 in CD\(_2\)OD................................. S58-60
\(^1\)H NMR, \(^{13}\)C NMR, Dept-135, COSY, HETCOR of 19 in CD\(_2\)OD................................. S60-62
\(^1\)H NMR, \(^{13}\)C NMR, Dept-135, COSY, HETCOR of 20 in CD\(_2\)OD................................. S63-65
\(^1\)H NMR, \(^{13}\)C NMR, Dept-135, COSY, HETCOR of 21 in CD\(_2\)OD................................. S65-67
\(^1\)H NMR, \(^{13}\)C NMR, Dept-135, COSY, HETCOR of 22 in CDCl\(_3\)................................. S68-70
\(^1\)H NMR, \(^{13}\)C NMR, Dept-135, COSY, HETCOR of 23 in CDCl\(_3\)................................. S70-72
\(^1\)H NMR, \(^{13}\)C NMR, Dept-135, COSY, HETCOR of 24 in CDCl\(_3\)................................. S73-75
\(^1\)H NMR, \(^{13}\)C NMR, Dept-135, COSY, HETCOR of 25 in CDCl\(_3\)................................. S75-77
\(^1\)H NMR, \(^{13}\)C NMR, Dept-135, COSY, HETCOR of 26 in CDCl\(_3\)................................. S78-80
\(^1\)H NMR, \(^{13}\)C NMR, Dept-135, COSY, HETCOR of 27 in CD\(_2\)OD................................. S80-82
\(^1\)H NMR, \(^{13}\)C NMR, Dept-135, COSY, HETCOR of 28 in CD\(_2\)OD................................. S83-85
$^1$H NMR of 2 in CDCl$_3$ at 400 MHz

$^{13}$C NMR of 2 in CDCl$_3$ at 100 MHz
Dept-135 of 2 in CDCl$_3$

COSY of 2 in CDCl$_3$
HMOC of 2 in CDCl$_3$

$^1$H NMR of 3 in CDCl$_3$ at 400 MHz
$^{13}$C NMR of 3 in CDCl$_3$ at 100 MHz

Dept-135 of 3 in CDCl$_3$
COSY of 3 in CDCl$_3$

HETCOR of 3 in CDCl$_3$
$^1$H NMR of 4 in CDCl$_3$ at 400 MHz

$^{13}$C NMR of 4 in CDCl$_3$ at 100 MHz
Dept-135 of 4 in CDCl₃

COSY of 4 in CDCl₃
HMOC of 4 in CDCl₃

¹H NMR of 5 in CDCl₃ at 400 MHz
$^{13}$C NMR of 5 in CDCl$_3$ at 100 MHz

Dept-135 of 5 in CDCl$_3$
COSY of 5 in CDCl$_3$

HMQC of 5 in CDCl$_3$
$^1$H NMR of 6 in CDCl$_3$ at 400 MHz

$^{13}$C NMR of 6 in CDCl$_3$ at 100 MHz
Dept-135 of 6 in CDCl$_3$

COSY of 6 in CDCl$_3$
HMOC of 6 in CDCl$_3$

$^1$H NMR of 7 in CDCl$_3$ at 400 MHz
$^{13}$C NMR of 7 in CDCl$_3$ at 100 MHz

Dept-135 of 7 in CDCl$_3$
COSY of 7 in CDCl$_3$

HETCOR of 7 in CDCl$_3$
$^1$H NMR of 8 in CDCl$_3$ at 400 MHz

$^{13}$C NMR of 8 in CDCl$_3$ at 100 MHz
Dept-135 of 8 in CDCl$_3$

COSY of 8 in CDCl$_3$
HETCOR of 8 in CDCl$_3$

$^1$H NMR of 9 in CDCl$_3$ at 400 MHz
$^{13}$C NMR of 9 in CDCl$_3$ at 100 MHz

Dept-135 of 9 in CDCl$_3$
COSY of 9 in CDCl$_3$

HETCOR of 9 in CDCl$_3$
$^1$H NMR of 10 in CDCl$_3$ at 400 MHz

$^{13}$C NMR of 10 in CDCl$_3$ at 100 MHz
Dept-135 of 10 in CDCl₃

COSY of 10 in CDCl₃
HETCOR of 10 in CDCl₃

¹H NMR of 11 in CDCl₃ at 400 MHz
$^{13}$C NMR of 11 in CDCl$_3$ at 100 MHz

Dept-135 of 11 in CDCl$_3$
COSY of 11 in CDCl₃

HETCOR of 11 in CDCl₃
$^1$H NMR of 12 in CDCl$_3$ at 400 MHz

$^{13}$C NMR of 12 in CDCl$_3$ at 100 MHz
Dept-135 of 12 in CDCl₃

COSY of 12 in CDCl₃
HETCOR of 12 in CDCl$_3$

$^1$H NMR of 13 in CDCl$_3$ at 400 MHz
$^{13}$C NMR of 13 in CDCl$_3$ at 100 MHz

Dept-135 of 13 in CDCl$_3$
COSY of 13 in CDCl₃

HETCOR of 13 in CDCl₃
$^1$H NMR of 14 in CD$_3$OD at 400 MHz

$^{13}$C NMR of 14 in CD$_3$OD at 100 MHz
Dept-135 of 14 in CD$_3$OD

COSY of 14 in CD$_3$OD
HETCOR of 14 in CD$_3$OD

$^1$H NMR of 15 in CD$_3$OD at 400 MHz
$^{13}$C NMR of 15 in CD$_3$OD at 100 MHz

Dept-135 of 15 in CD$_3$OD
COSY of 15 in CD$_3$OD

HETCOR of 15 in CD$_3$OD
$^1$H NMR of 16 in CD$_3$OD at 400 MHz

$^{13}$C NMR of 16 in CD$_3$OD at 100 MHz
Dept-135 of 16 in CD$_3$OD

COSY of 16 in CD$_3$OD
HETCOR of 16 in CD$_3$OD

$^1$H NMR of 17 in CD$_3$OD at 400 MHz
$^{13}$C NMR of 17 in CD$_3$OD at 100 MHz

Dept-135 of 17 in CD$_3$OD
COSY of 17 in CD$_3$OD

HETCOR of 17 in CD$_3$OD
$^1$H NMR of 18 in CD$_3$OD at 400 MHz

$^{13}$C NMR of 18 in CD$_3$OD at 100 MHz
Dept-135 of 18 in CD$_3$OD

COSY of 18 in CD$_3$OD
HETCOR of 18 in CD$_3$OD

$^1$H NMR of 19 in CD$_3$OD at 400 MHz
$^{13}$C NMR of 19 in CD$_3$OD at 100 MHz

Dept-135 of 19 in CD$_3$OD
COSY of 19 in CD$_3$OD

HETCOR of 19 in CD$_3$OD
$^1$H NMR of 20 in CD$_3$OD at 400 MHz

$^{13}$C NMR of 20 in CD$_3$OD at 100 MHz
Dept-135 of 20 in CD$_3$OD

COSY of 20 in CD$_3$OD
HETCOR of 20 in CD$_3$OD

$^1$H NMR of 21 in CD$_3$OD at 400 MHz
$^{13}$C NMR of 21 in CD$_3$OD at 100 MHz

Dept-135 of 21 in CD$_3$OD
COSY of 21 in CD$_3$OD

HETCOR of 21 in CD$_3$OD
$^1$H NMR of 23 in CDCl$_3$ at 400 MHz

$^{13}$C NMR of 23 in CDCl$_3$ at 100 MHz
Dept-135 of 23 in CDCl₃

COSY of 23 in CDCl₃
HETCOR of 23 in CDCl$_3$

$^1$H NMR of 24 in CDCl$_3$ at 400 MHz
$^{13}$C NMR of 24 in CDCl$_3$ at 100 MHz

Dept-135 of 24 in CDCl$_3$
COSY of 24 in CDCl$_3$

HETCOR of 24 in CDCl$_3$
$^1$H NMR of 25 in CDCl$_3$ at 400 MHz

$^{13}$C NMR of 25 in CDCl$_3$ at 100 MHz
Dept-135 of 25 in CDCl₃

COSY of 25 in CDCl₃
HETCOR of 25 in CDCl$_3$

$^1$H NMR of 26 in CDCl$_3$ at 400 MHz
$^{13}$C NMR of 26 in CDCl$_3$ at 100 MHz

Dept-135 of 26 in CDCl$_3$
COSY of 26 in CDCl$_3$

HETCOR of 26 in CDCl$_3$
$^1$H NMR of 27 in CD$_3$OD at 400 MHz

$^{13}$C NMR of 27 in CD$_3$OD at 100 MHz
Dept-135 of 27 in CD$_3$OD

COSY of 27 in CD$_3$OD
HETCOR of 27 in CD$_3$OD

$^1$H NMR of 28 in CD$_3$OD at 400 MHz
$^{13}$C NMR of 28 in CD$_3$OD at 100 MHz

Dept-135 of 28 in CD$_3$OD
COSY of $28$ in CD$_3$OD

HETCOR of $28$ in CD$_3$OD
$^1$H NMR of 29 in CD$_3$OD at 400 MHz

$^{13}$C NMR of 29 in CD$_3$OD at 100 MHz
Dept-135 of 29 in CD$_3$OD

COSY of 29 in CD$_3$OD
HETCOR of 29 in CD$_3$OD