Supporting Information

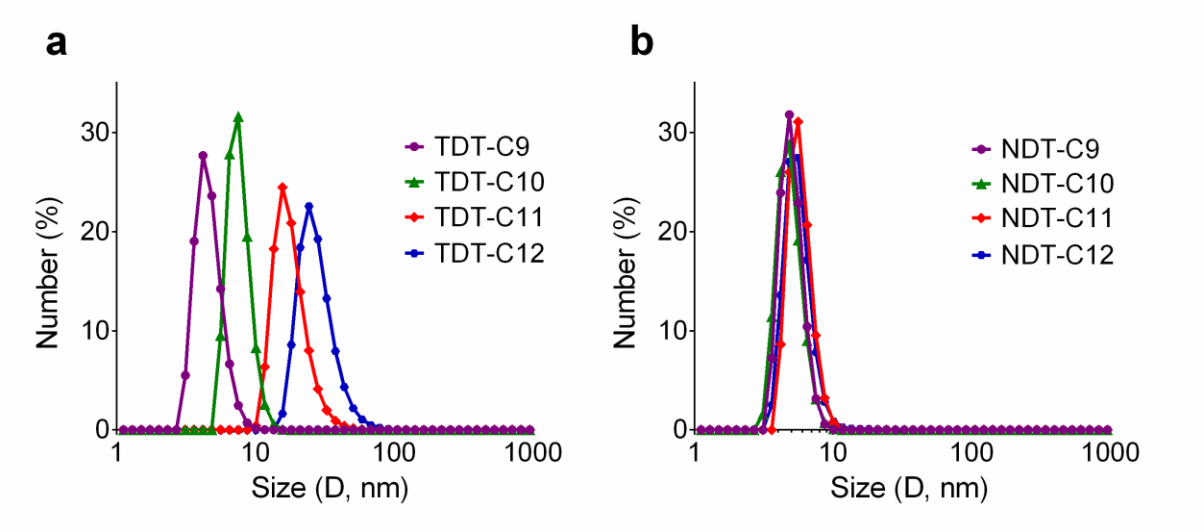


Figure S1. Dynamic light scattering profile of micelles formed by TDTs and NDTs. Each TDT (TDT-C9, TDT-C10, TDT-C11, or TDT-C12) or NDT (NDT-C9, NDT-C10, NDT-C11, or NDT-C12) was used at 0.5 wt% Autocorrelation analysis of scattered light intensity as a function of time produces the translational diffusion coefficient and hydrodynamic radius (R_h) of detergent micelles.

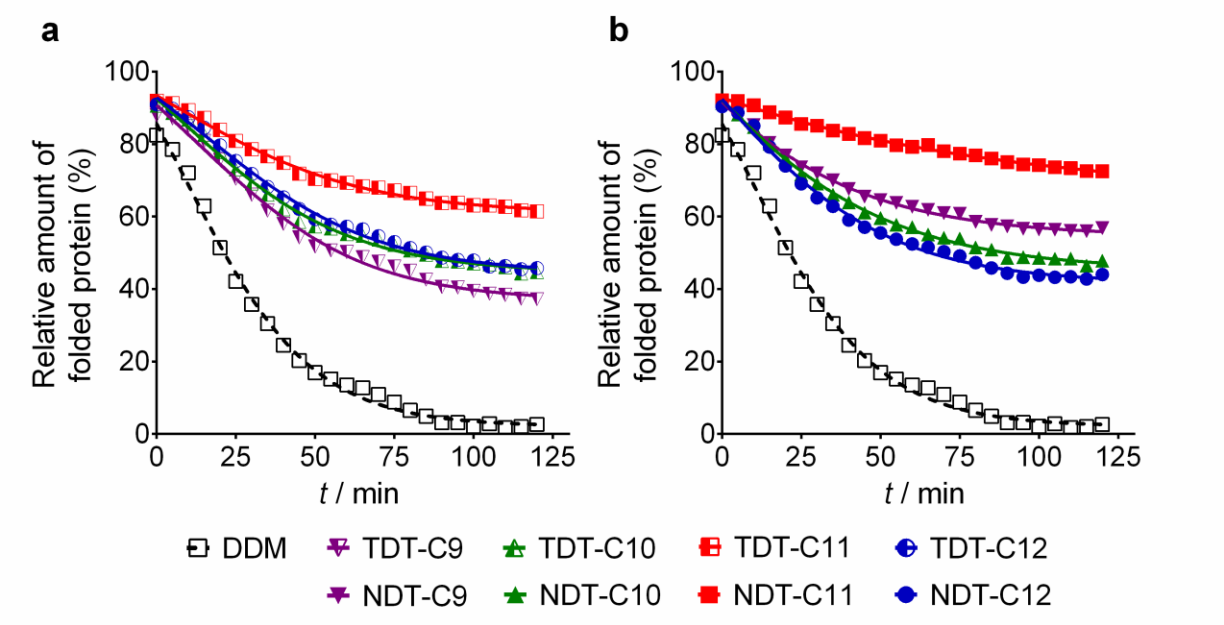


Figure S2. Thermal denaturation profile of UapA in DDM and novel detergents (TDTs and NDTs). The protein was purified in DDM and then exchanged into a novel detergent at CMC+0.2 wt%. DDM was used as a positive control. Thermal stability of UapA protein was monitored by CPM assay performed at 40℃ for 120 min. The relative amounts of folded protein were normalized relative to the most destabilising condition in this experiment, that is, DDM after 2 h incubation. Mean standard deviations (n = 2) for DDM, TDT-C9, TDT-C10, TDT-C11, TDT-C12, NDT-C9, NDT-C10, NDT-C11 and NDT-C12 are 3.8, 5.0, 10.5, 14.6, 3.6, 7.1, 5.1, 3.0, 8.9, respectively.

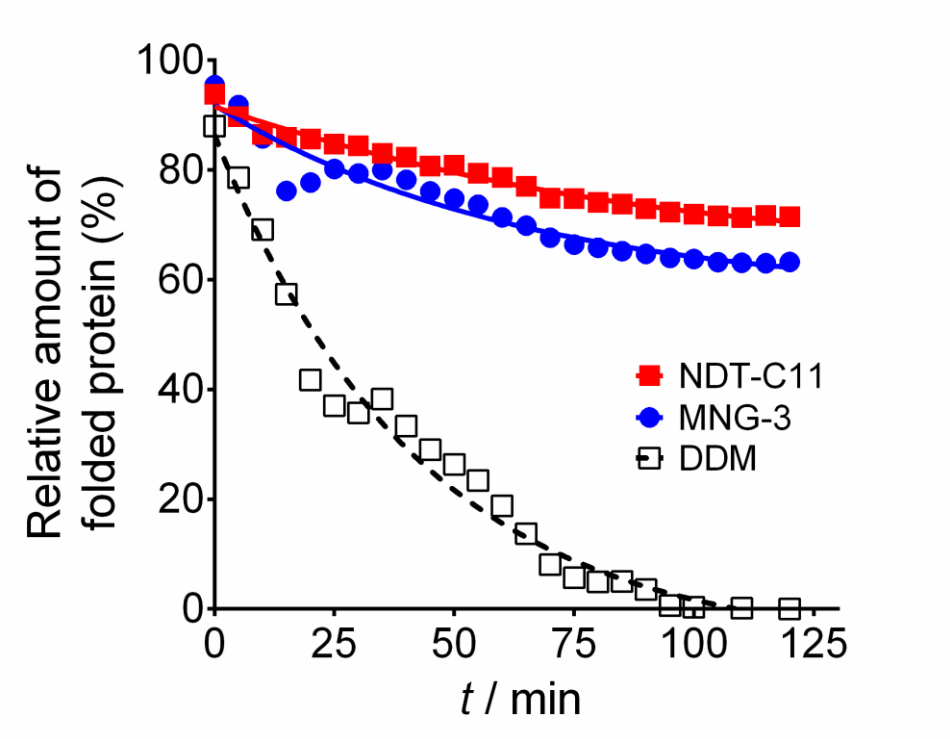


Figure S3. Thermal denaturation profile of UapA in DDM, NDT-C11 and MNG-3. The protein was purified in DDM and then exchanged into a novel detergent (NDT-C11 or MNG-3) at CMC+0.2 wt%. DDM was used as a positive control. Thermal stability of UapA protein was monitored by CPM assay performed at 40℃ for 120 min. The relative amounts of folded protein were normalized relative to the most destabilising condition in this experiment, that is, DDM after 2 h incubation.

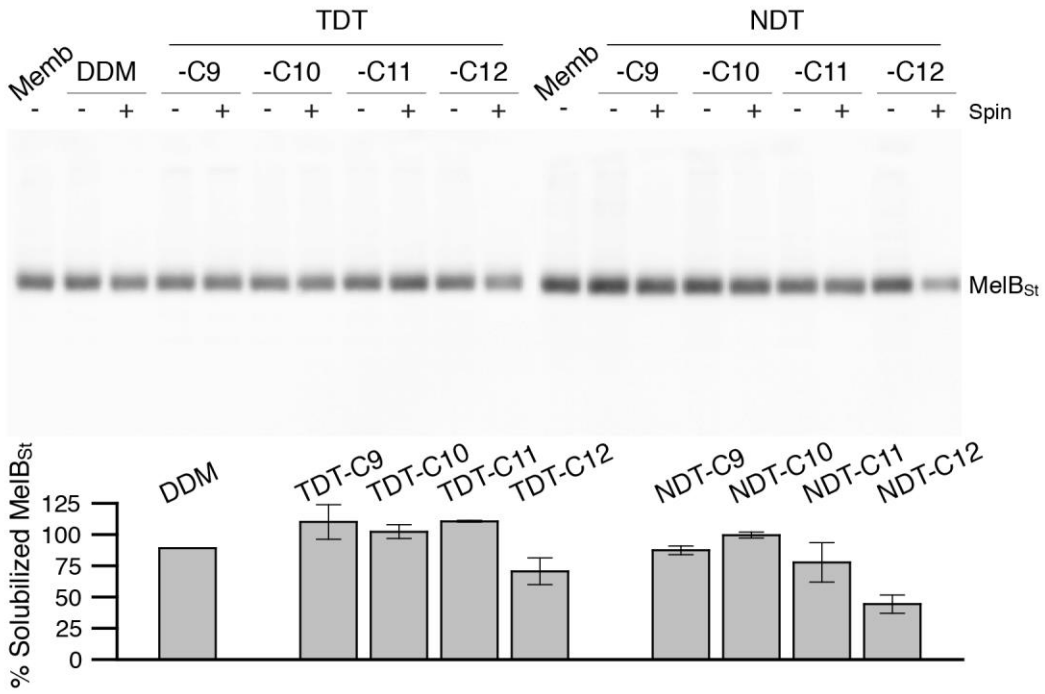


Figure S4. Detergent efficiency for $MelB_{St}$ solubilisation from the *E. coli* membranes. The solubility test was carried out at 0℃ for 90 min. Upper panel, solubilised material after ultracentrifugation of detergent-treated membranes was separated SDS-15%PAGE followed by Western blotting. Equal volumes of each sample before and after centrifugation were analysed. An untreated membrane sample ("Memb") was included as a control. The lower panel shows a histogram of average band densities. Detergent efficiency for $MelB_{St}$ solubilisation is expressed as the percentage of $MelB_{St}$ band from samples with ultracentrifugation (+) relative to the band without such a treatment (-). The density was measured by ImageQuant software. Error bars, SEM, $n = 2$.

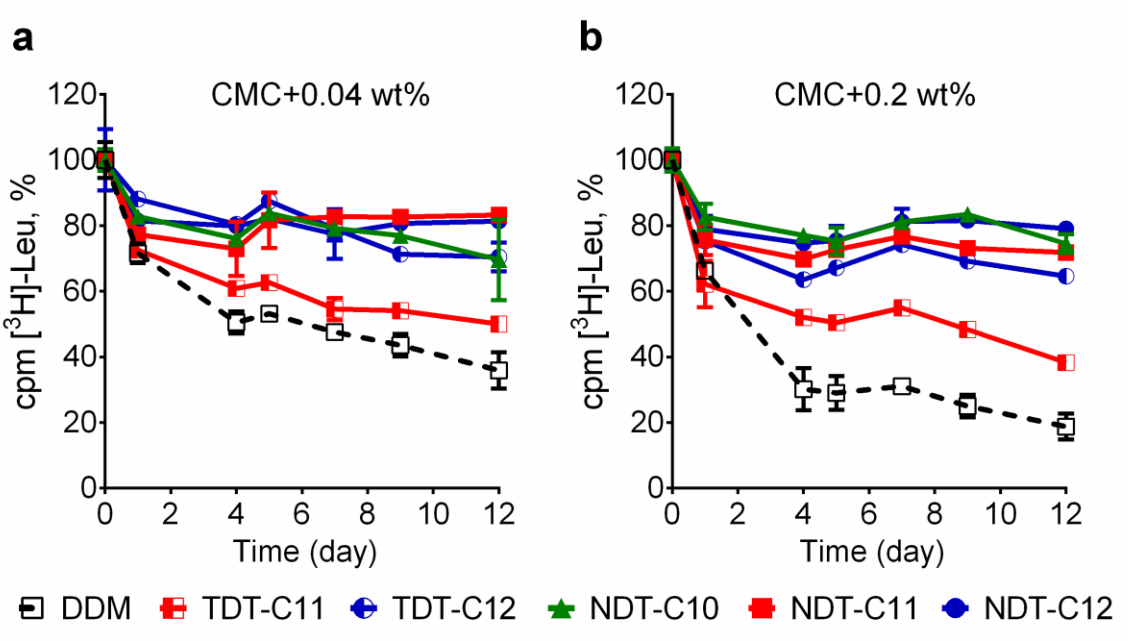


Figure S5. Long-term stability of wild type of leucine transporter (LeuT) solubilized in newly prepared amphiphiles (TDT-C11, TDT-C12, NDT-C10, NDT-C11 and NDT-C12) and a conventional detergent (DDM). Individual detergents were used at CMC+0.04 wt% (a) or CMC+0.2 wt% (b). Protein activity for LeuT was measured by scintillation proximity assay (SPA) at regular intervals during 12-days of incubation at room temperature. Results are expressed as % activity relative to activity at day 0 (mean ± s.e.m., n = 2).

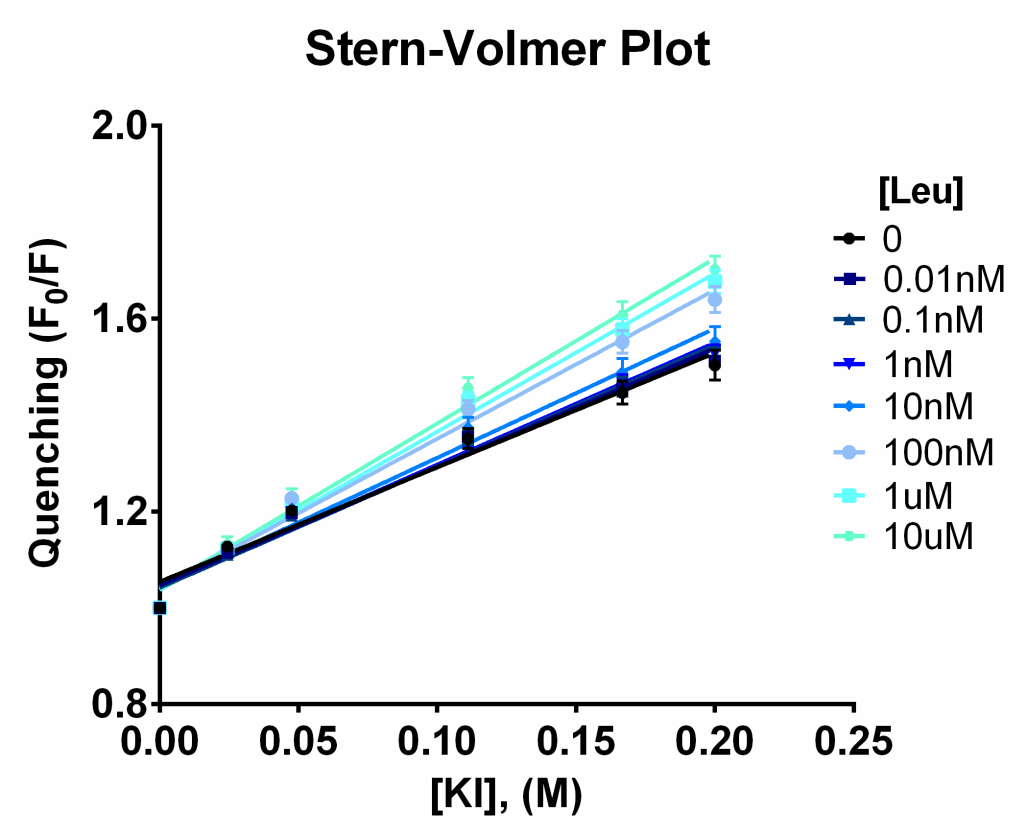


Figure S6. Leucine induced increase in the aqueous accessibility of the LeuT coupled TMR fluorophore**.** Iodide quenching of LeuT E192C^{TMR} in CMC + 0.04 wt% NDT-C11 in absence of substrate (black circles) and increasing concentrations of leucine ranging from dark blue to turquoise. Fluorescence intensities (F) were corrected for sample dilution, normalized to the initial intensity of the sample (F_0). Data are mean ± s.e.m. of three independent experiments.

Table S1 Radioligand binding constants and iodide quenching-response constants. Values of radioligand binding constants were calculated from SPA saturation binding experiments. Values of iodide quenching-response constants and ΔK_{SV} were calculated from site-directed fluorescence quenching spectroscopy experiments. The affinity (K_d) are shown as means ± s.e.m. The EC_{50} and ΔK_{SV} are shown as means [s.e.m. interval]. All data are from three to four independent experiments.

	K_d (nM)	EC_{50} (nM)	ΔK_{SV} (M^{-1})
DDM	141.8 ± 18	163 [118;227]	1.1 [1.04;1.17]
NDT (C11)	64 ± 6.5	41 [19;87]	0.9 [0.78;1.03]
MNG-3	N.D.[a]	94 [42;209]	0.37 [0.32;0.43]

[a] N.D. = not determined

Detergent CMC determination by diphenylhexatriene (DPH) encapsulation

5.0 mM stock solutions of TDTs or NDTs were prepared in distilled, deionized water. A series of detergent solutions with a range of concentrations were prepared from the stock solution. 200 µL each detergent sample was placed into 96-well plate in duplicates. A DPH stock solution was made by dissolving 3.0 mg DPH in 5.0 mL THF. A DPH working solution was prepared by adding 50 µL of the stock solution into 950 µL of distilled water. For dye encapsulation, 2.0 µL DPH work solution was added into each well containing a detergent solution. Following 15 ~ 20 min incubation at room temperature, fluorescence intensities were measured at 430 nm upon excitation at 358 nm by using Synergy Mx Monochromator-Based Multi-Mode Microplate reader. Detergent CMC values were determined by plotting florescence intensities as a function of detergent concentrations.

Detergent micelle size measurement by dynamic light scattering (DLS) experiment

The TDTs or NDTs were dissolved in distilled, deionized water to give detergent concentration of 1.0 wt%. These TDT or NDT solutions were filtered using a syringe filter with a pore size of 0.22 µm. Hydrodynamic radii of the micelles produced by the TDTs or NDTs were measured by using Malvern Zeta Sizer Nano ZS90 particle analyzer. A He-Ne laser at 633 nm with a maximum power of 5mW was used as a light source and scattered light was collected at the angle of 90°. Temperature was kept at 25 ℃ throughout all measurements. The translational diffusion coefficient and hydrodynamic radius (R_h) of detergent micelles was calculated by autocorrelation analysis of scattered light intensity as a function of time. R_h values were expressed as mean ± SD (n = 4).

Protein stability evaluation

Thermal denaturation assay

UapAG411V$_{\Delta 1\text{-}11}$ was expressed as a GFP fusion in *Saccharomyces cerevisiae* strain FGY217 and isolated as described previously in sample buffer (20 mM Tris (pH 7.5), 150 mM NaCl, 0.03% DDM, 1 mM xanthine).[1] The protein was concentrated to approximately 10 mg/ml using a 100 kDa molecular weight cut off filter (Millipore). The protein was diluted 1:150 into buffer containing either DDM or TDTs/NDTs at concentrations of CMC+ 0.04 wt% or CMC + 0.2 wt% in Greiner 96-well plates. The CPM dye (Invitrogen), stored in DMSO (Sigma), was diluted in dye buffer (20 mM Tris (pH 7.5), 150 mM NaCl, 0.03% DDM, 5 mM EDTA) and 3 µl of the diluted dye was added to each protein test condition. The reaction was monitored for 120 min at 40 ℃ using a microplate spectrofluorometer set at excitation and emission wavelengths of 387 nm and 463 nm, respectively. Relative maximum fluorescence was used to calculate the percentage of relative folded protein remaining after 130 min at 40 ℃. Relative unfolding profiles of proteins were plotted against time using GraphPad Prism.

MelB solubilization, thermal stability and ligand binding assays

E. coli DW2 strain (Δ*melB* and Δ*lacZY*) harboring pK95ΔAHB/WT $MelB_{St}$/CH10 plasmid were used to produce the protein.[2] The plasmid encodes the wild-type melibiose permease of *Salmonella typhimurium* ($MelB_{St}$) with a 10-His tag at the C-terminus. Cell growth and membrane preparation were carried out as described.[3] Protein assay was carried out with a Micro BCA kit (Thermo Scientific, Rockford, IL). The membrane samples containing $MelB_{St}$ (final total membrane protein concentration was 10 mg/mL) in a solubilization buffer (20 mM sodium phosphate, pH 7.5, 200 mM NaCl, 10% glycerol and 20 mM melibiose) were mixed with individual detergents (DDM, TDTs or NDTs) at 1.5 % (w/v). The extractions were incubated at four different temperatures (0, 45, 55, and 65 ℃) for 90 min. Insoluble fractions were removed by ultracentrifugation at 355,590 *g* in a Beckman OptimaTM MAX Ultracentrifuge using a TLA-100 rotor for 45 min at 4 ℃. Twenty μg membrane proteins were applied for the untreated membrane (Memb) or detergent extracts prior to ultracentrifugation, and equal volume of solutions were loaded for samples that the ultracentrifugation were applied. All samples were analyzed by SDS-15% PAGE, and $MelB_{St}$ was visualized by immunoblotting with a Penta-His-HRP antibody (Qiagen, Germantown, MD) as described.[3]

Preparation of RSO vesicles RSO membrane vesicles were prepared from *E. coli* DW2 cells containing $MelB_{St}$ or $MelB_{Ec}$ by osmotic lysis, [4-6] resuspended with 100 mM KP_i (pH 7.5) , and stored at -80 ℃.

Trp→D^2G FRET D^2G was kindly provided by H. Ronald Kaback and Gérard Leblanc. RSO membrane vesicles in 100 mM KP_i, pH 7.5, and 50 mM NaCl at a protein concentration of 1 mg/ml were solubilized with an indicated detergent (1.0%) at 23 ℃ for 30 min and ultracentrifuged using TLA 120.2 rotor at >300,000 g for 45 min at 4 ℃. The soluble fractions (supernatant) were used for Trp→D^2G FRET measurements with an Amico-Bowman Series 2 (AB2) spectrofluorometer. Trp residues were excited at 290 nm, emission was recorded at 465 nm for $MelB_{Ec}$ or 490 nm for $MelB_{St}$. D^2G at 10 μM and excess melibiose or equal volume of water were added at 1-min- and 2-min time points, respectively.

LeuT stability assay and site-directed fluorescence quenching spectroscopy experiment

LeuT stability assay Wild type of the leucine transporter (LeuT) from *Aquifex aeolicus* was purified according to the protocol described previously.[8] At first, LeuT was expressed in *E. coli* C41(DE3) transformed with pET16b encoding C-terminally 8xHis-tagged transporter (expression plasmid was kindly provided by Dr E. Gouaux, Vollum Institute, Portland, Oregon, USA). Briefly, after isolation of bacterial membranes and solubilisation in 1 % DDM, protein was bound to Ni^{2+}-NTA resin (Life Technologies, Denmark) and eluted in 20 mM Tris-HCl (pH 8.0), 1 mM NaCl, 199 mM KCl, 0.05 %

DDM and 300 mM imidazole. Subsequently, approx. 1.5 mg/ml protein stock was diluted 10 times into an identical buffer without DDM and imidazole, but supplemented with TDTs or NDTs and DDM (a positive control) at the final concentrations of CMC + 0.04 wt% or CMC + 0.2 wt%, respectively. Protein samples were stored at room temperature and after regular intervals, samples were centrifuged prior to protein activity measurement. Protein activity was determined by measuring [^{3}H]-Leu binding using scintillation proximity assay (SPA).[9] Assay was performed with 5 µL of the respective protein samples in the buffer containing 200 mM NaCl and the respective test compounds at the concentrations indicated above. SPA reaction was carried out in the presence of 20 nM [^{3}H]-Leu and copper chelate (His-Tag) YSi beads (both from PerkinElmer, Denmark). Total [^{3}H]-Leu binding fort the respective samples was measured using MicroBeta liquid scintillation counter (PerkinElmer).

Construction, expression, purification and fluorescent labelling The leucine transporter mutant (LeuT E192C^{TMR}) was generated, purified and labelled as previously described.[10] Briefly the residue E192 was mutated to a cysteine using QuikChange (Agilent Technologies) to allow fluorescent coupling via maleimide labeling. The LeuT variant was expressed in *Escherichia coli* C41 (DE3). The membranes was isolated by disruption, and LeuT was solubilized in 1 % (w/v) DDM, whereafter the detergent solubilized LeuT was immobilized on Chelating Sepharose Fast Flow resin (GE Healthcare, Little Chalfont, UK) and washed with tetramethylrhodamine-5-maleimide (TMR, Life Technologies, Carlsbad, California, USA) for 16 hours at 4°C. Subsequently LeuT was eluted in buffer containing 300 mM imidazole, 20 mM Tris-HCl (pH 8.0), 1 mM NaCl, 199 mM KCl and 0.05 % DDM.

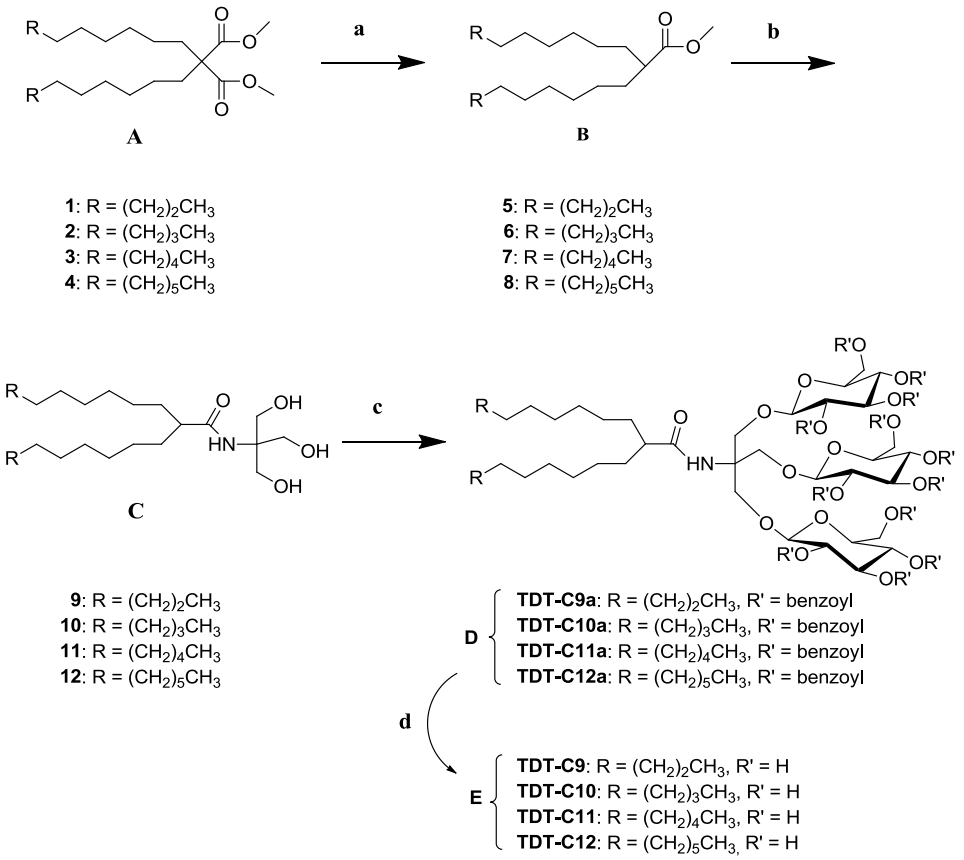
Radioligand binding Binding assay was performed using the Scintillation Proximity Assay (SPA).[9] Purified LeuT E192C^{TMR} was diluted in buffer A (20 mM Tris-HCl (pH 8.0), 200 mM NaCl, 100 µM TCEP) and CMC + 0.04 % DDM or NDT-C11, mixed with [^{3}H]leucine (25.1 Ci mmol^{-1}, PerkinElmer, Waltham, Massachusetts, USA) at the indicated concentrations and 0.125 mg ml^{-1} YSi-Cu Histag SPA beads (PerkinElmer). Data was analysed by non-linear regression analysis and fitted to a single-site hyperbolic function.

Fluorescence Spectroscopy Fluorescence-based experiments were performed with 0.5 µg ml^{-1} fluorescently labelled LeuT diluted buffer A supplemented with CMC + 0.04 wt% DDM, MNG-3 or NDT-C11. Aliquots of LeuT with increasing concentration of leucine were incubated on shaker at room temperature for 1 hour. Steady-state fluorescence intensities were recorded on a FluoroMax4 (Horiba Scientific, Edison, New Jersey, USA) at λ_{em} = 572 nm and excitation source at λ_{ex} = 552 nm at 25°C. Quencher-titration was carried out by successive addition of small aliquots containing 1 M KI in buffer A supplemented with 10 mM $Na_2S_2O_3$. Fluorescence intensities (F) were corrected for sample dilution, normalized to the initial intensity of the sample (F_0). All data were analysed by linear- (Stern-Volmer plots) or nonlinear regression in GraphPad Prism 6.0 (GraphPad Software). The

degree of accessibility was obtained from the Stern-Volmer equation: $F_0/F = 1 + K_{SV} \times [Q]$, where F_0/F is the normalized fluorescence quenching, K_{SV} is the Stern-Volmer constant and [Q] is the quencher concentration. When means are calculated from a logarithmic scale, means are shown as pIC_{50} and the [s.e.m. interval] from $pIC_{50} \pm$ s.e.m.

Preparation of TRIS-derived triglucosides (TDTs)

Supplementary scheme 1



a) LiCl, H_2O, DMSO, 160˚C; (b) Tris(hydroxymethyl)aminomethane, NaOMe, DMSO, 50˚C; (c) Perbenzoylated glucosylbromide, AgOTf, CH_2Cl_2 , -45℃ → room temperature; (d) NaOMe, MeOH, room temperature.

General procedure for the synthesis of dialkylated dimethylmalonate (**A**)

Dimethylmalonate (1.0 equiv.) was added dropwise to a cold solution of NaH (3.0 equiv.) in DMSO. The mixture was allowed to stir until the evolution of gas ceases. To this solution alkyl iodide (2.5 equiv.) was added slowly. The resulting mixture was allowed to stir for 3 hours at room temperature. Reaction was quenched by the addition of ice cold 10% NH_4Cl solution. The resulting solution was washed twice with ethylacetate. Combined ethylacetate fraction was washed with brine and dried over anhydrous Na_2SO_4. Concentration of the organic solvent resulted in oily residue. After column chromatographic purification of the residue (EtOAc/hexane), the desired product was obtained as colorless oil.

General procedure for demethoxy carbonylation under Krapcho's decarboxylation condition (step a; **A→B**)

To a stirred solution of **compound A** in DMSO was added LiCl (2.2 equiv.) and water (1.1 equiv.). The mixture

was stirred for a few minutes and then heated to 160 ˚C for 12 hours. After addition of brine, the reaction mixture was extracted with EtOAc twice. Combined organic layers were washed with brine and dried over anhydrous Na_2SO_4. Concentrating the reaction mixture by rotary evaporation gave dark oily residue. After column chromatographic purification desired dialkylated monoester was obtained as colorless oil.

General procedure for the tris coupling with dialkylated methyl ester (step b; **B→C**)

To a mixture of dialkylated methyl ester (**compound B)** (1.0 equiv.) in DMSO was added Tris(hydroxymethyl)aminomethane (1.5 equiv.) and NaOMe (2.0 equiv.) in one portion under nitrogen. The solution was stirred at 50˚C for 6 hours. The reaction mixture was diluted with water and then extracted with ethylacetate. The organic layer was washed with brine and dried over anhydrous Na_2SO_4. After concentration of the ethylacetate solution, the residue was purified by flash column chromatography (EtOAc/hexane) providing desired product as a white solid.

General procedure for glycosylation reaction[10] (step c; **C→D**)

Under nitrogen atmosphere, a mixture of **compound C** (1.0 equiv.), AgOTf (3.6 equiv.), 2,4,6-collidine (1.0 equiv.) in anhydrous CH_2Cl_2 was stirred at -45 ℃. A solution of perbenzoylated glucosylbromide (3.6 equiv.) in CH_2Cl_2 was added dropwise to this suspension. Stirring was continued for 30 min at -45 ℃, and then the reaction mixture was allowed to warm to 0 ℃ and left stirring for 1.5 hours. After completion of reaction (as detected by TLC), pyridine was added to the reaction mixture, and it was diluted with CH_2Cl_2 before being filtered over celite. The filtrate was washed successively with 1 M aqueous $Na_2S_2O_3$ solution, 0.1 M aqueous HCl solution, and brine. The organic layer was dried with anhydrous Na_2SO_4 and the solvents were removed by rotary evaporation. The residue was purified by silica gel column chromatography (EtOAc/hexane) providing desired product as a glassy solid.

General Procedure for the de-O-benzoylations under Zemplén's condition[10] (step d; **D→E**)

The O-benzoylated compounds **(compound D)** were dissolved in MeOH and then treated with the required amount of a methanolic solution of 0.5 M NaOMe such that the final concentration of NaOMe was 0.05 M. The reaction mixture was left stirring for 6 hours at room temperature, and then neutralized with Amberlite IR-120 (H+ form) resin. The resin was removed by filtration and washed with MeOH and solvent was removed from the combined filtrate in vacuo. The residue was purified by silica gel column chromatography ($MeOH/CH_2Cl_2$) to afford the desired product as white solid.

Dimethyl 2-nonylmalonate (1) was prepared in 91% yield according to the general procedure for preparation of dialkylated dimethylmalonate. ^{1}H NMR (400 MHz, $CDCl_3$): δ 3.70 (s, 6H), 1.88-1.83 (m, 4H), 1.34-1.05 (m, 28H), 0.87 (t, *J* = 7.2 Hz, 6H); ^{13}C NMR (100 MHz, $CDCl_3$): δ 172.3, 57.5, 52.0, 31.8, 29.7, 29.4, 29.2, 29.2, 23.9, 22.6, 14.0.

Dimethyl 2-decylmalonate (2) was prepared in 91% yield according to the general procedure for preparation of dialkylated dimethylmalonate. ^{1}H NMR (400 MHz, $CDCl_3$): δ 3.70 (s, 6H), 1.88-1.83 (m, 4H), 1.34-1.25 (m, 32H), 0.87 (t, *J* = 7.2 Hz, 6H); ^{13}C NMR (100 MHz, $CDCl_3$): δ 172.5, 57.7, 52.2, 32.50, 32.0, 29.9, 29.7, 29.6,

29.4, 24.1, 23.6, 22.8, 14.2.

Dimethyl 2-undecylmalonate (3) was prepared in 86% yield according to the general procedure for preparation of dialkylated dimethylmalonate. ^{1}H NMR (400 MHz, $CDCl_3$): δ 3.70 (s, 6H), 1.88-1.83 (m, 4H), 1.30-1.24 (m, 36H), 0.87 (t, *J* = 7.2 Hz, 6H); ^{13}C NMR (100 MHz, $CDCl_3$): δ 172.4, 57.7, 52.1, 51.2, 45.7, 32.6, 32.4, 32.0, 29.9, 29.7, 29.6, 29.4, 29.4, 27.5, 24.0, 22.7, 14.1.

Dimethyl 2-dodecylmalonate (4) was prepared in 89% yield according to the general procedure for preparation of dialkylated dimethylmalonate. ^{1}H NMR (400MHz, $CDCl_3$): δ 3.66 (s, 6H), 1.90-1.87 (m, 4H), 1.30-1.25 (m, 40H), 0.87 (t, *J* = 7.2 Hz, 6H); ^{13}C NMR (100MHz, $CDCl_3$): δ 169.9, 61.5, 52.3, 32.1, 29.9, 29.7, 29.6, 29.5, 29.4, 28.9, 27.5, 22.9, 14.4.

Methyl 2-nonylundecanoate (5) was prepared in 80% yield according to the general procedure for Krapcho's demethoxy carbonylation. ^{1}H NMR (400 MHz, $CDCl_3$): δ 3.66 (s, 3H), 2.32-2.23 (m, 1H), 1.67-1.40 (m, 4H) 1.34-1.25 (m, 28H), 0.87 (t, *J* = 7.2 Hz, 6H); ^{13}C NMR (100 MHz, $CDCl_3$): δ 169.8, 61.5, 52.3, 33.1, 29.8, 29.7, 29.5, 29.4, 28.9, 27.5, 22.9, 14.3.

Methyl 2-decyldodecanoate (6) was prepared in 82% yield according to the general procedure for Krapcho's demethoxy carbonylation. ^{1}H NMR (400 MHz, $CDCl_3$): δ 3.66 (s, 3H), 2.32-2.23 (m, 1H), 1.67-1.40 (m, 4H), 1.34-1.25 (m, 32H), 0.87 (t, *J* = 7.2 Hz, 6H); ^{13}C NMR (100 MHz, $CDCl_3$): δ 169.8, 61.5, 52.3, 32.1, 29.8, 29.7, 29.5, 29.4, 28.9, 27.5, 22.9, 14.3.

Methyl 2-undecyltridecanoate (7) was prepared in 78% yield according to the general procedure for Krapcho's demethoxy carbonylation. ^{1}H NMR (400 MHz, $CDCl_3$): δ 3.66 (s, 3H), 2.32-2.23 (m, 1H), 1.67-1.40 (m, 4H), 1.30-1.24 (m, 40H), 0.87 (t, *J* = 7.2 Hz, 6H); ^{13}C NMR (100 MHz, $CDCl_3$): δ 169.8, 61.5, 52.3, 33.1, 29.8, 29.7, 29.6, 29.4, 28.9, 27.5, 22.9, 14.3.

Methyl 2-dodecyltetradecanoate (8) was prepared in 81% yield according to the general procedure for demethoxycarbonylation. ^{1}H NMR (400 MHz, $CDCl_3$): δ 3.65 (s, 3H), 2.32-2.30 (m, 1H), 1.67-1.40 (m, 4H), 1.30-1.25 (m, 44H), 0.87 (t, *J* = 7.2 Hz, 6H); ^{13}C NMR (100 MHz, $CDCl_3$): δ 169.9, 61.5, 52.3, 32.1, 29.9, 29.9, 29.7, 29.6, 29.5, 29.4, 28.9, 27.5, 22.9, 14.4.

***N*-(1,3-dihydroxy-2-(hydroxymethyl)propan-2-yl)-2-nonylundecanamide (9)** was prepared in 74% yield according to the general procedure for tris coupling reaction. ^{1}H NMR (400 MHz, $CDCl_3$): δ 6.48 (s, 1H), 4.64 (br s, 3H), 3.60 (s, 6H), 2.10-2.03 (m, 1H), 1.65-1.33 (m, 4H), 1.38-1.20 (m, 28H), 0.88 (t, *J* = 6.8 Hz, 6H); ^{13}C NMR (100 MHz, $CDCl_3$): δ 178.4, 131.0, 128.9, 62.5, 60.2, 48.1, 33.0, 32.0, 29.7, 29.7, 29.6, 29.4, 27.6, 22.8, 14.2.

***N*-(1,3-dihydroxy-2-(hydroxymethyl)propan-2-yl)-2-decyldodecanamide (10)** was prepared in 72% yield according to the general procedure for tris coupling reaction. ^{1}H NMR (400 MHz, $CDCl_3$): δ 6.49 (s, 1H), 5.20-5.1 (br s, 3H), 3.57 (s, 6H), 2.10-2.01 (m, 1H), 1.65- 1.39 (m, 4H), 1.38-1.20 (m, 32H), 0.88 (t, *J* = 6.8 Hz, 6H); ^{13}C NMR (100 MHz, $CDCl_3$): δ 178.3, 131.0, 62.3, 60.1, 48.0, 33.0, 31.9, 29.6, 29.5, 29.4, 29.3, 27.5, 22.6, 14.1.

***N*-(1,3-dihydroxy-2-(hydroxymethyl)propan-2-yl)-2-undecyltridecanamide (11)** was prepared in 70% yield according to the general procedure for tris coupling reaction. ^{1}H NMR (400 MHz, $CDCl_3$): δ 6.51 (s, 1H), 5.63 (br s, 3H), 3.54 (s, 6H), 2.12-2.05 (m, 1H), 1.55-1.31 (m, 4H), 1.38-1.20 (m, 36H), 0.88 (t, *J* = 6.8 Hz, 6H); ^{13}C NMR (100 MHz, $CDCl_3$): δ 178.3, 131.0, 62.4, 60.1, 48.3, 33.0, 31.9, 29.6, 29.5, 29.4, 29.3, 28.4, 27.5, 22.6, 14.0.

***N*-(1,3-dihydroxy-2-(hydroxymethyl)propan-2-yl)-2-dodecyltetradecanamide (12)** was prepared in 72% yield according to the general procedure for tris coupling reaction. ^{1}H NMR (400 MHz, $CDCl_3$): δ 6.45 (s, 1H), 3.98 (br s, 3H), 3.64 (s, 6H), 2.11-2.05 (m, 1H), 1.63-1.39 (m, 4H), 1.38-1.20 (m, 40H), 0.88 (t, *J* = 6.8 Hz, 6H)); ^{13}C NMR (100 MHz, $CDCl_3$): δ 178.4, 131.1, 128.9, 62.5, 61.2, 48.1, 33.4, 32.0, 29.7, 29.6, 29.4, 28.3, 28.1, 27.6, 22.8, 14.1.

TDT-C9a was prepared in 75% yield according to the general procedure for glycosylation reactions. ^{1}H NMR (400 MHz, $CDCl_3$): δ 8.10-7.80 (m, 18H), 7.60-7.10 (m, 42H), 5.75 (s, 1H), 5.63 (t, *J* = 9.6 Hz, 3H), 5.51 (t, *J* = 9.6 Hz, 3H), 5.38 (t, *J* = 8.0 Hz, 3H), 4.45-4.32 (m, 6H), 4.02 (d, *J* = 8.0 Hz, 3H), 3.78 (d, *J* = 9.6 Hz, 3H), 3.4-3.3 (m, 3H), 3.21-3.05 (m, 6H), 1.80-1.65 (m, 1H), 1.45-1.10 (m, 32H), 0.86 (t, *J* = 6.8 Hz, 6H); ^{13}C NMR (100 MHz, $CDCl_3$): δ 176.4, 166.0, 165.7, 165.0, 164.6,133.7, 133.4, 133.3, 129.9, 129.8, 129.7, 129.6, 129.5, 129.4, 129.0, 128.8, 128.3, 101.4, 72.6, 71.9, 69.5, 68.1, 62.9, 59.3, 32.8, 32.0, 31.9, 29.9, 29.7, 29.4, 27.5, 27.3, 22.7, 14.2.

TDT-C10a was prepared in 80% yield according to the general procedure for glycosylation reactions. ^{1}H NMR (400 MHz, $CDCl_3$): δ 8.10-7.80 (m, 18H), 7.60-7.10 (m, 42H), 5.75 (s, 1H), 5.63 (t, *J* = 9.6 Hz, 3H), 5.51 (t, *J* = 9.6 Hz, 3H), 5.38 (t, *J* = 8.0 Hz, 3H), 4.45-4.32 (m, 6H), 4.02 (d, *J* = 8.0 Hz, 3H), 3.78 (d, *J* = 9.6 Hz, 3H), 3.4-3.3 (m, 3H), 3.21-3.05 (m, 6H), 1.80-1.65 (m, 1H), 1.45-1.10 (m, 36H), 0.86 (t, *J* = 6.8 Hz, 6H); ^{13}C NMR (100 MHz, $CDCl_3$): δ 176.5, 166.1, 165.8, 165.1, 164.7, 133.8, 133.5, 133.3, 133.2, 130.0, 129.9, 129.8, 129.7, 129.6, 129.5, 129.1, 128.9, 128.9, 128.5, 128.4, 101.5, 72.7, 72.0, 69.6, 68.2, 63.0, 59.4, 32.8, 32.0, 30.0, 29.8, 29.6, 29.5, 27.6, 27.4, 22.8, 14.2.

TDT-C11a was prepared in 65% yield according to the general procedure for glycosylation reactions. ^{1}H NMR (400 MHz, $CDCl_3$): δ 8.10-7.80 (m, 18H), 7.60-7.10 (m, 42H), 5.75 (s, 1H), 5.63 (t, *J* = 9.6 Hz, 3H), 5.51 (t, *J* = 9.6 Hz, 3H), 5.38 (t, *J* = 8.0 Hz, 3H), 4.45-4.32 (m, 6H), 4.02 (d, *J* = 8.0 Hz, 3H), 3.78 (d, *J* = 9.6 Hz, 3H), 3.4-3.3 (m, 3H), 3.21-3.05 (m, 6H), 1.80-1.65 (m, 1H), 1.45-1.10 (m, 40H), 0.86 (t, *J* = 6.8 Hz, 6H); ^{13}C NMR (100 MHz, $CDCl_3$): δ 176.4, 166.0, 165.7, 165.0, 164.6, 133.7, 133.4, 133.2, 133.1, 129.9, 129.8, 129.7, 129.6, 129.4, 129.0, 128.9, 128.8, 128.4, 128.3, 101.4, 72.6, 71.9, 69.5, 68.1, 62.9, 59.3, 53.4, 48.2, 32.8, 32.7, 31.9, 31.6, 29.9, 29.8, 29.4, 27.5, 27.3, 22.7, 22.6, 14.1.

TDT-C12a was prepared in 70% yield according to the general procedure for glycosylation reactions. ^{1}H NMR (400 MHz, $CDCl_3$): δ 8.10-7.80 (m, 18H), 7.60-7.10 (m, 42H), 5.75 (s, 1H), 5.63 (t, *J* = 9.6 Hz, 3H), 5.51 (t, *J* = 9.6 Hz, 3H), 5.38 (t, *J* = 8.0 Hz, 3H), 4.45-4.32 (m, 6H), 4.02 (d, *J* = 8.0 Hz, 3H), 3.78 (d, *J* = 9.6 Hz, 3H), 3.4-3.3 (m, 3H), 3.21-3.05 (m, 6H), 3.02-2.90 (m, 2H), 1.80-1.65 (m, 1H), 1.45-1.05 (m, 36H), 0.86 (t, *J* = 6.8 Hz, 6H) ;^{13}C NMR (100 MHz, $CDCl_3$): δ 176.5, 166.2, 165.8, 165.1, 164.7, 133.8, 133.5, 133.4, 133.2, 130.0, 129.9, 129.8, 129.7, 129.5, 129.1, 129.0, 128.9, 128.5, 128.4, 101.5, 72.7, 69.6, 68.2, 63.0, 59.4, 53.6, 48.3, 32.9, 32.8,

32.0, 30.0, 29.9, 29.8, 29.5, 27.6, 27.4, 22.8, 14.2.

TDT-C9 was prepared in 90% yield according to the general procedure for deprotection reactions. ^{1}H NMR (400 MHz, CD_3OD): δ 4.50-4.31 (m, 6H), 3.89-3.85 (m, 6H), 3.67-3.63 (m, 3H), 3.37-3.17 (m, 12H), 2.2-2.1 (m, 1H), 1.38-1.21 (m, 32H), 0.80 (t, *J* = 6.8 Hz, 6H); ^{13}C NMR (100 MHz, CD_3OD): δ 179.5, 104.8, 78.1, 75.2, 71.8, 69.4, 62.9, 61.2, 34.4, 34.3, 33.2, 31.2, 31.0, 30.9, 30.8, 30.6, 28.7, 28.5, 23.8, 14.5. HRMS (EI): calcd. for $C_{42}H_{79}NO_{19}[M+Na]^+$ 924.5144, found 924.5143.

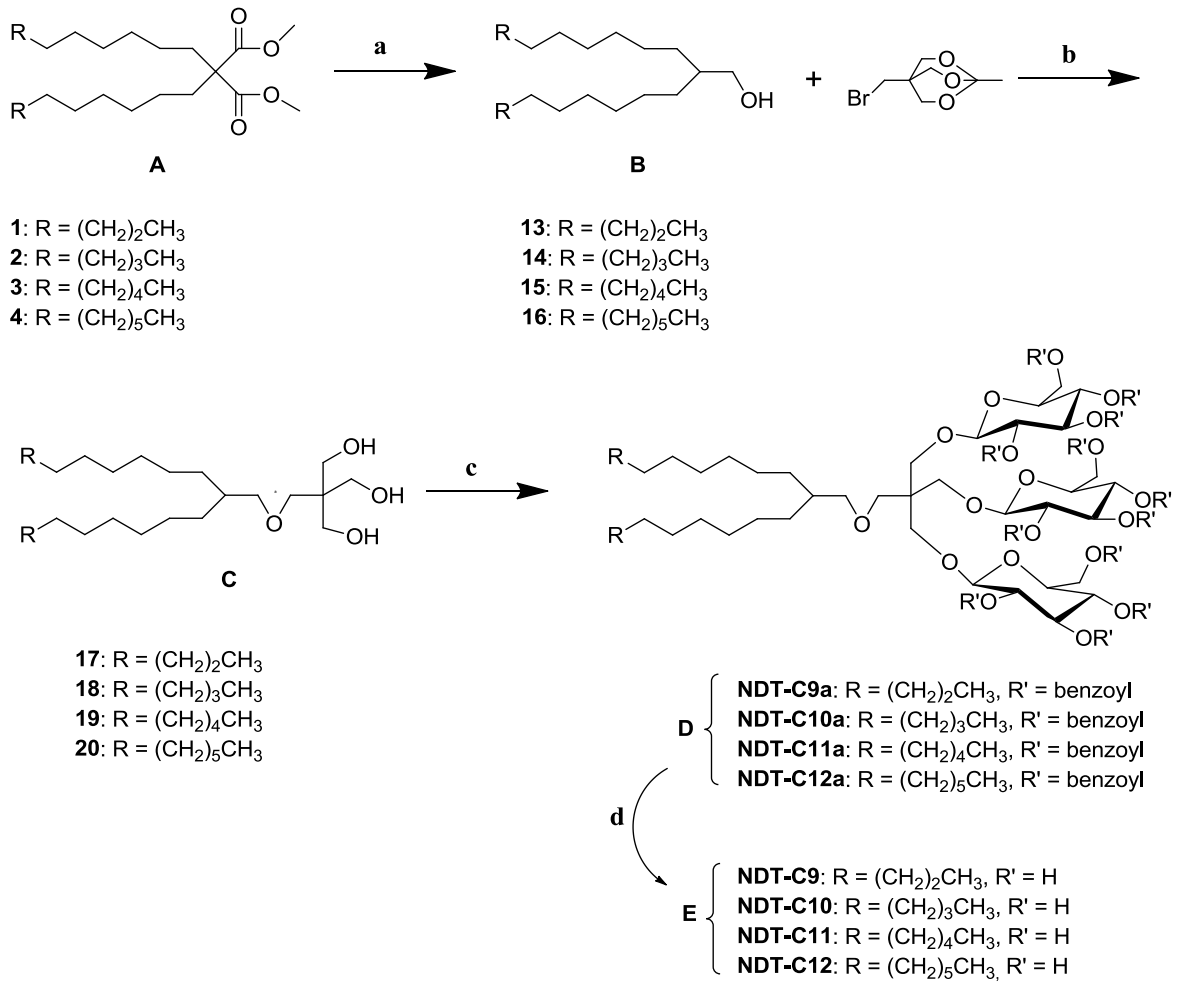
TDT-C10 was prepared in 95% yield according to the general procedure for deprotection reactions. ^{1}H NMR (400 MHz, CD_3OD): δ 4.50-4.31 (m, 6H), 3.89-3.85 (m, 6H), 3.67-3.63 (m, 3H), 3.37-3.17 (m, 12H), 2.2-2.1 (m, 1H), 1.38-1.21 (m, 36H), 0.80 (t, *J* = 6.8 Hz, 6H); ^{13}C NMR (100 MHz, CD_3OD): δ 179.6, 104.8, 78.1, 75.2, 71.8, 69.5, 62.9, 61.3, 34.3, 34.3, 33.2, 31.2, 31.0, 30.9, 30.8, 30.6, 28.7, 28.6, 23.8, 14.6. HRMS (EI): calcd. for $C_{44}H_{83}NO_{19}[M+Na]^+$ 952.5457, found 952.5454.

TDT-C11 was prepared in 90% yield according to the general procedure for deprotection reactions. ^{1}H NMR (400 MHz, CD_3OD): δ 4.65-4.37 (m, 6H), 3.92-3.89 (m, 6H), 3.72-3.67 (m, 3H), 3.45-3.22 (m, 12H), 2.31-2.20 (m, 1H), 1.41-1.21 (m, 40H), 0.92 (t, *J* = 6.8 Hz, 6H); ^{13}C NMR (100 MHz, CD_3OD): δ 179.5, 104.8, 78.0, 75.1, 71.8, 69.4, 62.9, 61.1, 39.6, 34.3, 34.2, 33.2, 31.2, 31.0, 30.9, 30.8, 30.6, 28.6, 28.5, 23.8, 14.6. HRMS (EI): calcd. for $C_{46}H_{87}NO_{19}[M+Na]^+$ 980.5770, found 980.5766.

TDT-C12 was prepared in 94% yield according to the general procedure for deprotection reactions. ^{1}H NMR (400 MHz, CD_3OD): δ 4.50-4.31 (m, 6H), 3.89-3.85 (m, 6H), 3.67-3.63 (m, 3H), 3.36-3.17 (m, 12H), 2.25-2.15 (m, 1H), 1.40-1.22 (m, 42H), 0.89 (t, *J* = 6.8 Hz, 6H); ^{13}C NMR (100 MHz, CD_3OD): δ 179.6, 104.8, 78.1, 75.2, 71.8, 69.4, 62.9, 61.2, 33.2, 31.2, 31.0, 30.9, 30.8, 30.6, 28.7, 28.5, 23.8, 14.6. HRMS (EI): calcd. for $C_{48}H_{91}NO_{19}[M+Na]^+$ 1008.6083, found 1008.6086.

Preparation of neopentyl glycol-derived Triglucosides (NDTs)

Supplementary scheme 2



a) (i) LiCl, H_2O, DMSO, 160˚C; (ii) $LiAlH_4$, THF, room temperature; (b) (i) NaH, DMF:THF (1:1), 100˚C; (ii) CH_2Cl_2: MeOH (1:1), HCl, NaOH; (c) Perbenzeoylated glucosylbromide, AgOTf, CH_2Cl_2 , -45˚C→ room temperature; (d) NaOMe, MeOH, room temperature.

General procedure for the reduction of dialkylated ester by using LAH (step a; **A→B**)

To dialkylated dimethyl malonate in DMSO was added LiCl and H_2O. The mixture was heated to reflux for 12 hrs. After cooling the reaction mixture to room temperature, water was added followed by washing with ethylacetate. Combined organic layers were washed with brine and then dried over anhydrous Na_2SO_4. After concentration, an oily residue was obtained which was used as such in the subsequent reduction step. $LiAlH_4$ (2.0 equiv.) was added to ice cold solution of dialkylated mono methyl ester in THF. The mixture was stirred at room temperature for 6 hours. Reaction was quenched by the addition of MeOH, water, 1M aqueous HCl

solution successively at 0 ℃ and then extracted with diethyl ether twice. The organic layers were combined and washed with brine and dried over anhydrous Na_2SO_4. The residue obtained after rotary evaporation was purified by silica gel column chromatography (EtOAc/hexane), affording the desired product.

General procedure for the synthesis of dialkylated tri-ol[11] (step b; **B→C**)

To a solution of dialkylated mono-ol (**compound B;** 1.0 equiv.) in DMF was added NaH (3.0 equiv.). The mixture was heated to 50˚C for 30 min. After cooling the mixture to room temperature, 4-(bromomethyl)-l-methyl-2,6,7-trioxabicyclo[2.2.2]-octane (3.0 equiv.) dissolved in THF was added dropwise. The resulting mixture was heated at 100˚C for 24 hours. After quenching with methanol, organic solvents were removed under reduced pressure. The solid residue was dissolved in diethyl ether, and the organic solution was washed with brine and dried over anhydrous Na_2SO_4. After concentration of the organic solvent, the residue was dissolved in DCM/MeOH mixture. To this solution was added a few drops of conc. HCl and the resulting mixture was heated at 50˚C for 4 hours. After neutralization with NaOH and concentration of the reaction mixture, the residue was purified by column chromatography (EtOAc/hexane) to afford the desired product.

2-nonylundecan-1-ol (13) was prepared in 89% yield according to the general procedure for Krapcho's decarboxylation and reduction of dialkylated monoesters. ^{1}H NMR (400 MHz, $CDCl_3$): δ 3.53 (d, *J* = 5.6 Hz, 2H), 1.45 (t, *J* = 5.6 Hz, 1H), 1.36-1.18 (m, 36H), 0.88 (t, *J* = 6.4 Hz, 6H); ^{13}C NMR (100 MHz, $CDCl_3$): δ 65.7, 40.5, 31.9, 30.9, 30.1, 29.6, 29.3, 26.9, 22.7, 14.1.

2-decyldodecan-1-ol (14) was prepared in 88% yield according to the general procedure for Krapcho's decarboxylation and reduction of dialkylated monoesters. ^{1}H NMR (400 MHz, $CDCl_3$): δ 3.53 (d, *J* = 5.6 Hz, 2H), 1.45 (t, *J* = 5.6 Hz, 1H), 1.36-1.18 (m, 36H), 0.88 (t, *J* = 6.4 Hz, 6H); ^{13}C NMR (100 MHz, $CDCl_3$): δ 65.6, 40.5, 31.9, 30.9, 30.1, 29.7, 29.3, 26.9, 22.7, 14.0.

2-undecyltridecan-1-ol (15) was prepared in 76% yield according to the general procedure for Krapcho's decarboxylation and reduction of dialkylated monoesters. ^{1}H NMR (400MHz, $CDCl_3$): δ 3.53 (d, *J* = 5.6 Hz, 2H), 1.45 (t, *J* = 5.6 Hz, 1H), 1.36-1.18 (m, 40H), 0.88 (t, *J* = 6.4 Hz, 6H); ^{13}C NMR (100 MHz, $CDCl_3$): δ 65.7, 40.6, 32.1, 31.1, 30.3, 29.9, 29.8, 29.5, 27.0, 22.8, 14.2.

2-dodecyltetardecan-1-ol (16) was prepared in 83% yield according to the general procedure for Krapcho's decarboxylation and reduction of dialkylated monoesters. ^{1}H NMR (400 MHz, $CDCl_3$): δ 3.53 (d, *J* = 5.6 Hz, 2H), 1.45 (t, *J* = 5.6 Hz, 1H), 1.36-1.18 (m, 44H), 0.88 (t, *J* = 6.4 Hz, 6H); ^{13}C NMR (100 MHz, $CDCl_3$): δ 65.7, 40.5, 31.9, 30.9, 30.1, 29.6, 29.3, 26.9, 22.7, 14.1.

2-(((2-nonylundecyl)oxy)methyl)-2-(hydroxymethyl)propane-1,3-diol (17) was prepared in 44% yield according to the general procedure for the synthesis of dialkylated triol. ^{1}H NMR (400 MHz, $CDCl_3$): δ 3.72 (d, *J* = 6.0 Hz, 6H), 3.43 (s, 2H), 3.3 (d, *J* = 5.6 Hz, 2H), 2.72 (t, *J* = 5.6 Hz, 3H), 1.61-1.51 (m, 1H), 1.36-1.18 (m, 32H), 0.88 (t, *J* = 6.4 Hz, 6H); ^{13}C NMR (100 MHz, $CDCl_3$): δ 72.0, 70.5, 68.9, 68.5, 67.9, 66.8, 63.5, 44.4, 43.0, 30.8, 28.6, 28.6, 28.4, 28.3, 25.1, 21.6, 13.1.

2-(((2-decyldodecyl)oxy)methyl)-2-(hydroxymethyl)propane-1,3-diol (18) was prepared in 42% yield

according to the general procedure for the synthesis of dialkylated triol. ^{1}H NMR (400 MHz, $CDCl_3$): δ 3.72 (d, *J* = 6.0 Hz, 6H), 3.43 (s, 2H), 3.30 (d, *J* = 5.6 Hz, 2H), 2.72 (t, *J* = 5.6 Hz, 3H), 1.61-1.50 (m, 1H), 1.36-1.18 (m, 32H), 0.88 (t, *J* = 6.4 Hz, 6H); δ ^{13}C NMR (100 MHz, $CDCl_3$): δ 72.0, 70.5, 68.9, 68.5, 67.9, 66.8, 63.6, 44.4, 43.1, 30.8, 28.6, 28.6, 28.4, 28.3, 25.1, 21.6, 13.1.

2-(((2-undecyltridecyl)oxy)methyl)-2-(hydroxymethyl)propane-1,3-diol (19) was prepared in 40% yield according to the general procedure for the synthesis of dialkylated triol. ^{1}H NMR (400 MHz, $CDCl_3$): δ 3.72 (s, 6H), 3.44 (s, 2H), 3.31 (d, *J* = 5.6 Hz, 2H), 2.52 (s, 3H), 1.59-1.41 (m, 1H), 1.36-1.18 (m, 40H), 0.88 (t, *J* = 6.8 Hz, 6H); ^{13}C NMR (100 MHz, $CDCl_3$): δ 73.2, 71.7, 70.1, 69.7, 69.1, 68.0, 64.7, 45.6, 45.1, 32.0, 29.8, 29.8, 29.6, 29.5, 26.3, 22.8, 14.3.

2-(((2-dodecyltetradecyl)oxy)methyl)-2-(hydroxymethyl)propane-1,3-diol (20) was prepared in 44% yield according to the general procedure for the synthesis of dialkylated triol. ^{1}H NMR (400 MHz, $CDCl_3$): δ 3.70 (s, 6H), 3.42 (s, 2H), 3.30 (d, *J* = 5.6 Hz, 2H), 2.96 (s, 3H), 1.60-1.51 (m, 1H), 1.36-1.18 (m, 44H), 0.88 (t, *J* = 6.8 Hz, 6H); ^{13}C NMR (100 MHz, $CDCl_3$): δ 73.0, 71.7, 71.1, 69.7, 69.1, 68.0, 66.3, 64.7, 45.6, 45.1, 32.0, 29.8, 29.8, 29.6, 29.5, 28.6, 26.3, 21.8, 14.3.

For the glycosylation and deprotection reaction, the same procedure was used as for the synthesis of TDTs.

NDT-C9a was prepared in 50% yield according to the general procedure for glycosylation reactions. ^{1}H NMR (400 MHz, $CDCl_3$): δ 8.10-7.80 (m, 18H), 7.60-7.10 (m, 42H), 5.63 (t, *J* = 9.6 Hz, 3H), 5.51 (t, *J* = 9.6 Hz, 3H), 5.38 (t, *J* = 8.0 Hz, 3H), 4.45-4.32 (m, 6H), 4.02 (d, *J* = 8.0 Hz, 3H), 3.78 (d, *J* = 9.6 Hz, 3H), 3.4-3.3 (m, 3H), 3.21-3.05 (m, 6H), 3.02-2.90 (m, 2H), 1.42-1.36 (m, 1H), 1.35-1.01 (m, 32H), 0.86 (t, *J* = 6.8 Hz, 6H); ^{13}C NMR (100 MHz, CD_3OD): δ 166.1, 165.9, 165.2, 164.8, 133.6, 133.5, 133.3, 133.2, 130.1, 129.9, 129.8, 129.7, 129.1, 129.0, 128.5, 128.4, 101.6, 72.8, 72.1, 71.8, 69.8, 68.0, 63.2, 53.6, 45.2, 38.0, 32.1, 31.4, 31.3, 30.3, 29.9, 29.6, 27.0, 26.9, 22.8, 14.3.

NDT-C10a was prepared in 50% yield according to the general procedure for glycosylation reactions. ^{1}H NMR (400 MHz, $CDCl_3$): δ 8.10-7.80 (m, 18H), 7.60-7.10 (m, 42H), 5.62 (t, *J* = 9.6 Hz, 3H), 5.50 (t, *J* = 9.6 Hz, 3H), 5.38 (t, *J* = 8.0 Hz, 3H), 4.43-4.32 (m, 6H), 4.02 (d, *J* = 8.0 Hz, 3H), 3.79 (d, *J* = 9.6 Hz, 3H), 3.35 (t, *J* = 4.8 Hz, 3H), 3.18-3.07 (m, 6H), 3.21-3.05 (m, 6H), 2.97-2.95 (m, 2H), 1.41-1.36 (m, 1H), 1.35-1.01 (m, 36H), 0.86 (t, *J* = 6.8 Hz, 6H); ^{13}C NMR (100 MHz, CD_3OD): δ 166.2, 165.9, 165.2, 164.9, 134.0, 133.8, 133.6, 133.5, 133.4, 133.2, 130.2, 130.1, 130.0, 129.9, 129.8, 129.1, 128.7, 128.6, 128.5, 101.6, 72.9, 72.8, 72.1, 71.8, 69.9, 63.3, 62.1, 32.1, 31.5, 30.4, 30.0, 29.9, 29.6, 27.0, 22.9, 14.3.

NDT-C11a was prepared in 47% yield according to the general procedure for glycosylation reactions. ^{1}H NMR (400 MHz, $CDCl_3$): δ 8.04-7.80 (m, 24H), 7.55-7.37 (m, 36H), 5.62 (t, *J* = 9.6 Hz, 3H), 5.50 (t, *J* = 9.6 Hz, 3H), 5.37 (t, *J* = 8.0 Hz, 3H), 4.39-4.34 (m, 6H), 4.01 (d, *J* = 8.0 Hz, 3H), 3.78 (d, *J* = 9.6 Hz, 3H), 3.37-3.33 (m, 3H), 3.13-3.10 (m, 6H), 2.96-2.94 (m, 2H), 1.43-1.37 (m, 1H), 1.36-1.02 (m, 40H), 0.86 (t, *J* = 6.8 Hz, 6H); ^{13}C NMR (100 MHz, CD_3OD): δ 166.2, 165.9, 165.4, 164.8, 133.6, 133.5, 133.3, 130.2, 130.1, 130.0, 129.9, 129.8, 129.1, 129.0, 128.6, 128.5, 101.5, 90.6, 72.8, 72.3, 72.0, 71.8, 71.7, 70.2, 69.9, 69.8, 69.6, 68.2, 68.0, 63.2, 63.0, 45.5, 45.0, 32.0, 29.8, 29.7, 29.5, 26.4, 22.8, 14.3.

NDT-C12a was prepared in 65% yield according to the general procedure for glycosylation reactions. ^{1}H NMR (400 MHz, CDCl3): δ 8.04-7.80 (m, 24H), 7.55-7.37 (m, 36H), 5.62 (t, *J* = 9.6 Hz, 3H), 5.50 (t, *J* = 9.6 Hz, 3H), 5.37 (t, *J* = 8.0 Hz, 3H), 4.39-4.34 (m, 6H), 4.01 (d, *J* = 8.0 Hz, 3H), 3.78 (d, *J* = 9.6 Hz, 3H), 3.37-3.33 (m, 3H), 3.13-3.10 (m, 6H), 2.96-2.94 (m, 2H), 1.42-1.37 (m, 1H), 1.36-1.02 (m, 40H), 0.86 (t, *J* = 6.8 Hz, 6H)); ^{13}C NMR (100 MHz, $CDCl_3$): δ 166.3, 166.0, 165.9, 165.3, 165.1, 164.7, 133.5, 133.4, 133.2, 133.1, 130.1, 129.9, 129.8, 129.7, 129.6, 129.2, 129.0, 128.9, 128.8, 128.4, 128.3, 101.5, 92.3, 90.5, 74.6, 74.1, 72.7, 72.3, 72.0, 71.7, 70.2, 69.5, 68.4, 67.7, 63.1, 62.9, 45.1, 37.9, 31.9, 30.2, 29.8, 29.7, 29.4, 26.9, 26.8, 22.7, 14.1.

NDT-C9 was prepared in 90% yield according to the general procedure for deprotection reactions. ^{1}HNMR (400 MHz, CD_3OD): δ 4.50 (d, *J* = 8.0 Hz, 3H), 4.21 (d, *J* = 8.0 Hz, 3H), 3.88 (d, *J* = 6.0 Hz, 3H), 3.77-3.74 (m, 6H), 3.58-3.52 (m, 2H), 3.40-3.25 (m, 10H), 3.10 (t, *J* = 8.0 Hz, 3H), 1.48-1.40 (m, 1H), 1.30-1.10 (m, 32H), 0.80 (t, *J* = 6.8 Hz, 6H); ^{13}C NMR (100 MHz, CD_3OD): δ 133.6, 132.5, 130.0, 129.0, 105.2, 78.1, 77.9, 76.0, 75.2, 71.7, 70.7, 70.2, 62.8, 46.7, 39.4, 33.2, 32.6, 31.3, 30.9, 30.6, 28.0, 23.8, 14.6. HRMS (EI): calcd. for $C_{43}H_{82}O_{19}[M+Na]^+$ 925.5348, found 925.5346.

NDT-C10 was prepared in 93% yield according to the general procedure for deprotection reactions. ^{1}HNMR (400 MHz, CD_3OD): δ 4.30 (d, *J* = 8.0 Hz, 3H), 3.98 (d, *J* = 8.0 Hz, 3H), 3.84 (d, *J* = 2 Hz, 3H), 3.70-3.60 (m, 6H), 3.48 (s, 2H), 3.34-3.21 (m, 10H), 3.19 (t, *J* = 8.0 Hz, 3H), 1.48-1.40 (m, 1H), 1.30-1.10 (m, 36H), 0.80 (t, *J* = 6.8 Hz, 6H); ^{13}C NMR (100 MHz, CD_3OD): δ 105.2, 78.1, 77.9, 76.0, 75.2, 71.7, 70.2, 62.8, 46.7, 39.5, 33.2, 32.6, 31.3, 30.9, 30.6, 28.0, 23.8, 14.6. HRMS (EI): calcd. for $C_{45}H_{86}O_{19}[M+Na]^+$ 953.5661, found 953.5657.

NDT-C11 was prepared in 90% yield according to the general procedure for deprotection reactions. ^{1}HNMR (400 MHz, CD_3OD): δ 4.30 (d, *J* = 8.0 Hz, 3H), 3.98 (d, *J* = 10.0 Hz, 3H), 3.84 (d, *J* = 12 Hz, 3H), 3.69-3.62 (m, 6H), 3.48 (s, 2H), 3.34-3.21 (m, 10H), 3.18 (t, *J* = 8.0 Hz, 3H), 1.56-1.50 (m, 1H), 1.36-1.20 (m, 40H), 0.89 (t, *J* = 6.8 Hz, 6H); ^{13}C NMR (100 MHz, CD_3OD): δ 105.0, 78.0, 77.8, 75.2, 72.6, 71.1, 70.8, 70.7, 70.1, 69.9, 69.7, 62.7, 46.7, 46.5, 39.5, 33.1, 31.0, 30.8, 30.6, 27.5, 23.8, 14.6. HRMS (EI): calcd. for $C_{47}H_{90}O_{19}[M+Na]^+$ 981.5974, found 981.5977.

NDT-C12 was prepared in 90% yield according to the general procedure for deprotection reactions. ^{1}HNMR (400 MHz, CD_3OD): δ 4.30 (d, *J* = 8.0 Hz, 3H), 3.98 (d, *J* = 10.0 Hz, 3H), 3.84 (d, *J* = 12 Hz, 3H), 3.69-3.62 (m, 6H), 3.48 (s, 2H), 3.34-3.21 (m, 10H), 3.18 (t, *J* = 8.0 Hz, 3H), 1.56-1.50 (m, 1H), 1.36-1.20 (m, 44H), 0.89 (t, *J* = 6.8 Hz, 6H); ^{13}C NMR (100 MHz, CD_3OD): δ 105.1, 78.0, 77.7, 76.0, 75.1, 71.5, 71.3, 70.6, 70.1, 62.7, 46.6, 39.3, 33.1, 32.5, 31.2, 30.8, 30.5, 27.9, 23.8, 14.6. HRMS (EI): calcd. for $C_{49}H_{94}O_{19}[M+Na]^+$ 1009.6287, found 1009.6290.

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