Supporting Information

Synthesis of 1,2,3-Triazole linked Galactopyranosides and Evaluation of Cholera Toxin Inhibition

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2-[2-(2-Prop-2-ynoxyethoxy)ethoxy]ethanol (3). Compound **3** was prepared as described in the literature,[1] but at two-thirds the scale and in a yield of 58% (lit.[1] 75%). Data for compound **3** were identical to literature values.[1]

2-[2-[2-(2-prop-2-ynoxyethoxy)ethoxy]ethoxy]ethanol (4). Compound **4** was prepared as described for compound **3**, but using tetraethylene glycol (20 g) and it was obtained in a yield of 41% (lit.[2] 65%). Data for compound **4** were identical to literature values.[2]

2-[2-[2-(2-prop-2-ynoxyethoxy)ethoxy]ethoxy]ethyl 4-methylbenzenesulfonate (6). Compound **6** was prepared as described for compound **5**, using compound **4** (2.80 g) giving **6** {51% (lit.[2] 81%)}. Data for compound **6** were identical to literature values.[2]

1,4-Bis[2-[2-[2-(2-prop-2-ynoxyethoxy)ethoxy]ethoxy]ethyl]piperazine (8). Compound **8** was obtained by the same procedure used for compound **7** with the following modifications: to a solution of piperazine (248 mg, 2.9 mmol) in dry THF (40 mL) was added sodium hydride (60% dispersion in oil, 483 mg) and the reaction mixture was stirred at room temperature for 1 h before a solution of 2-[2-[2-(2-prop-2-ynoxyethoxy)ethoxy]ethoxy]ethyl 4-methylbenzenesulfonate **6** (2.33 g, 6.0 mmol) in dry THF (10 mL + 10 mL rinse) was added. After 40 min, 4-dimethylaminopyridine (DMAP, 17 mg) was added to the reaction mixture, which was then refluxed at 77 °C for 25 h. The crude oil was purified by column chromatography on silica gel eluting with 5-50% MeOH:EtOAc containing 1% Et_3N to yield compound **8** as a yellow oil (690 mg, 47%). IR (ATR, cm^{-1}) 3248, 2866, 2809, 2113, 1456, 1349, 1296, 1249, 1090, 1011, 930, 845, ^1H NMR δ ($CDCl_3$, 300 MHz), 4.16 (4H, t, J = 2.4 Hz), 3.66-3.55 (28H, m), 2.57-2.51 (12H, m), 2.40 (2H, d, J = 2.3 Hz); ^{13}C NMR δ ($CDCl_3$, 75 MHz), 79.6, 74.5, 70.5, 70.34, 70.30, 69.0, 68.8, 58.3, 57.6 and 53.4. MS-ESI: *m/z* $[M + H]^+$ for $C_{26}H_{47}N_2O_8^+$ calculated 515.3; observed 515.3. HRMS-ESI: *m/z* $[M + H]^+$ for $C_{26}H_{47}N_2O_8^+$, calculated 515.3332; observed 515.3350.

[(2*R*,3*S*,4*S*,5*R*,6*R*)-4,5-diacetoxy-2-(acetoxymethyl)-6-[(1*R*)-4-[2-[2-[2-[2-[4-[2-[2-[2-[2-[[(1*R*)-1-[(2*R*,3*R*,4*S*,5*S*,6*R*)-3,4,5-triacetoxy-6-(acetoxymethyl)tetrahydropyran-2-yl]triazol-4-yl]methoxy]ethoxy]ethoxy]ethoxy]ethyl]piperazin-1-yl]ethoxy]ethoxy]ethoxy]ethoxymethyl]triazol-1-yl]tetrahydropyran-3-yl] acetate (10). Compound **10** was obtained by the same procedure used for compound **9** with the following modifications. Compound **8** (298 mg, 0.58 mmol) was suspended in a 2:1 *t*-BuOH:H_2O mixture (6 mL) to which was added 2,3,4,6-tetraacetate-β-D-galactopyranosyl azide (476 mg) and copper(0) (368 mg). The reaction mixture was allowed to stir vigorously for 16 h (not under inert atmosphere). The crude material was then purified by column chromatography on silica gel eluting with 17-20% MeOH:EtOAc containing 1% Et_3N to yield compound **10** as a thick brown/yellow oil (431 mg, 59%). $[\alpha]_D^{20}$-4 (*c* 4.04, MeOH); IR (ATR, cm^{-1}) 2869, 2859, 1746, 1454,

1432, 1368, 1210, 1089, 1042, 947, 922, ^{1}H NMR δ ($CDCl_3$, 300 MHz), 7.82 (2H, s), 5.82 (2H, d, J = 9.3 Hz), 5.55-5.49 (4H, m), 5.27-5.19 (2H, m), 4.66 (4H, s), 4.22-4.09 (6H, m), 3.64-3.58 (28H, m), 2.61 (12H, br s), 2.19 (6H, s), 2.01 (6H, s), 1.98 (6H, s), 1.86 (6H, s); ^{13}C NMR δ ($CDCl_3$, 75 MHz), 170.3, 169.9, 169.8, 169.0, 145.8, 121.1, 86.2, 74.0, 70.8, 70.5, 70.3, 69.7, 67.9, 66.9, 64.5, 61.2, 57.5, 53.1, 20.6, 20.5 and 20.2. MS-ESI: *m/z* $[M + H]^+$ for $C_{54}H_{85}N_8O_{26}^+$ calculated 1261.6; observed 1261.5. HRMS-ESI: *m/z* $[M + H]^+$ for $C_{54}H_{85}N_8O_{26}^+$, calculated 1261.5575; observed 1261.5526.

(2*R*,3*R*,4*S*,5*R*,6*R*)-2-(Hydroxymethyl)-6-[(1*R*)-4-[2-[2-[2-[2-[4-[2-[2-[2-[2-[[(1*R*)-1-[(2*R*,3*R*,4*S*,5*R*,6*R*)-3,4,5-trihydroxy-6-(hydroxymethyl)tetrahydropyran-2-yl]triazol-4-yl]methoxy]ethoxy]ethoxy]ethoxy]ethyl]piperazin-1-yl]ethoxy]ethoxy]ethoxy]ethoxymethyl]triazol-1-yl]tetrahydropyran-3,4,5-triol (12). Compound **12** was obtained by the same procedure as used for compound **11** with the following modifications. Compound **10** (386 mg, 0.31 mmol) was dissolved in distilled MeOH (10 mL) to which was added potassium carbonate (11.6 mg). The reaction mixture was stirred at room temperature for 22 h and the crude product was purified by column chromatography on silica gel eluting with 25% EtOAc:MeOH containing 1% Et_3N. The foam/solid that was obtained was redissolved in MeOH and filtered through a 0.45 µM filter to remove silica particles to yield compound **12** as a colourless foam (224 mg, 79%). $[\alpha]_D^{20}$ +5 (*c* 1.53, MeOH); IR (ATR, cm^{-1}) 3403, 3373, 2917, 2875, 2850, 1648, 1567, 1537, 1454, 1351, 1307, 1243, 1096, 892, ^{1}H NMR δ (D_2O, 300 MHz) 8.34 (2H, s), 5.75 (2H, d, J = 9.2 Hz), 4.77 (4H, s), 4.26 (2H, t, J = 9.3 Hz), 4.12 (2H, d, J = 3 Hz), 4.04 (2H, t, J = 6 Hz), 3.93 (2H, dd, J = 2.9 Hz, 9.7 Hz), 3.83-3.70 (32H, m), 2.65 (12H, br t, J = 5.1 Hz); ^{13}C NMR δ (D_2O, 75 MHz) 143.9, 123.8, 87.7, 77.9, 72.6, 69.3, 69.24, 69.19, 69.0, 68.7, 68.2, 67.0, 62.7, 60.5, 56.0 and 51.5. MS-ESI: *m/z* $[M + H]^+$ for $C_{38}H_{69}N_8O_{18}^+$ calculated 925.5; observed 925.4. HRMS-ESI: *m/z* $[M + H]^+$ for $C_{38}H_{69}N_8O_{18}^+$, calculated 925.4730; observed 925.4720.

2-[2-(2-Hydroxyethoxy)ethoxy]ethyl 4-methylbenzene-sulfonate (13). Compound **13** was prepared as described,[3] using triethylene glycol (157.5 g) in a yield of 73%. Data for compound **13** were identical to literature values.[4]

2-[2-[2-(2-Hydroxyethoxy)ethoxy]ethoxy]ethyl 4-methylbenzenesulfonate (14). Compound **14** was prepared as described in the literature using tetraethylene glycol (68.87 g) in a yield of 83% (lit.[3] 85%).[3] Data for compound **14** were identical to literature values.[3]

2-[2-(2-Azidoethoxy)ethoxy]ethanol (15). Compound **15** was prepared as described,[3] using **13** (23.14 g) in a yield of 69%. Data for compound **15** were identical to literature values.[5]

2-[2-[2-(2-Azidoethoxy)ethoxy]ethoxy]ethanol (16). Compound **16** was prepared as described in the literature, using **14** (10.08 g) and sodium azide (5 equivalents) in a yield of 81% (lit.[3] 76%). Data for compound **16** were identical to literature values.[3]

2-[2-(2-Azidoethoxy)ethoxy]ethyl 4-methylbenzenesulfonate (17). Compound **17** was prepared as described for compound **14**, using compound **15** (2.06 g), TsCl (2 equiv.) and Et_3N (2.7 equiv.). The reaction was allowed to stir at room temperature for 3 d to give **17** (90%), after work-up. Data for compound **17** were identical to literature values.[5]

2-[2-[2-(2-Azidoethoxy)ethoxy]ethoxy]ethyl 4-methylbenzenesulfonate (18). Compound **18** was prepared as described for compound **17**, using **16** (6.22 g) and the reaction was allowed to stir at room temperature for 23 h to give **18** (96%) after work-up. Data for compound **18** were identical to literature values.[6]

1,4-Bis[2-[2-[2-(2-azidoethoxy)ethoxy]ethoxy]ethyl]piperazine (20). Compound **20** was obtained by the same procedure as used for compound **19** with the following modifications: 2-[2-[2-(2-azidoethoxy)ethoxy]ethoxy]ethyl 4-methylbenzenesulfonate **17** (5.12 g, 13.7 mmol), piperazine (576 mg, 6.7 mmol) and sodium hydride 60% oil dispersion (1.10 g, 4.1 eq) in dry THF (30 mL). The crude product was purified via column chromatography on silica gel eluting with 0-9% MeOH:EtOAc containing 1% Et_3N to yield compound **20** as a pale yellow oil (2.06 g, 63%). ^{1}H NMR δ ($CDCl_3$, 500 MHz), 3.61-3.54 (24H, m), 3.32 (4H, t, J = 5.0 Hz), 2.55-2.51 (12H, m); ^{13}C NMR δ ($CDCl_3$, 125 MHz), 70.6, 70.51, 70.47, 70.2, 69.9, 68.6, 57.6, 53.2 and 50.5. MS-ESI: *m/z* $[M + H]^+$ for $C_{20}H_{41}N_8O_6^+$ calculated 489.3; observed 489.3. HRMS-ESI: *m/z* $[M + H]^+$ for $C_{20}H_{41}N_8O_6^+$, calculated 489.3149; observed 489.3161.

3,7-Anhydro-4,5,6,8-tetra-*O*-benzyl-1,1,2,2-tetradehydro-1,2-*D*-glycero-*L*-mannooctitol (21). Compound **21** was prepared as described in the literature.[7] Data for compound **21** were identical to literature values.[8]

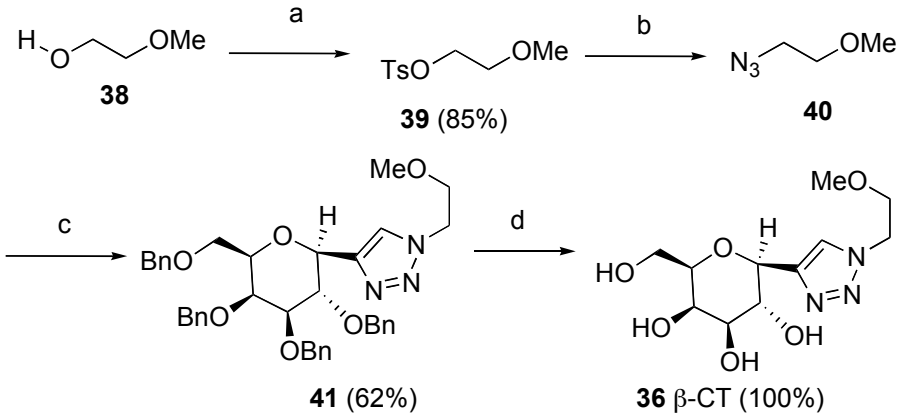
[(2*R*,3*S*,4*R*,5*S*,6*S*)-3,4,5-Triacetoxy-6-ethynyl-tetrahydropyran-2-yl]methyl acetate (24). Compound **24** was prepared as described in literature, using compound **21** (191 mg) and the reaction was stirred at room temperature for 7 d and 15 h to give **24** (54% (lit.[8] 81%)). Data for compound **24** were identical to literature values.[8]

Trimethyl(2-tributylstannylethynyl)silane (27). Compound **27** was prepared as described in the literature.[9] Data for compound **27** were identical to literature values.[9]

Acetyl 2,3,4,6-tetra-*O*-benzyl-*D*-galactopyranoside (28). To a solution of 2,3,4,6-tetra-*O*-benzyl-D-galactopyranoside[7] (3.13 g, 5.8 mmol) in dry pyridine (35 mL) at 0 °C was slowly added acetic anhydride (32 mL). The reaction mixture was stirred at 0 °C for 1 h, after which DMAP (22 mg, 0.2 mmol) was added. The reaction mixture was then allowed to stir at room temperature for another 1 h, after which the reaction mixture was slowly poured into ice/water. After ethyl acetate extraction, evaporation of the solvent, and co-evaporations with distilled toluene the resulting crude oil was dry loaded onto silica and purified by column chromatography eluting with 50-83% EtOAc:Hexane to yield compound **28** as a colourless oil (3.37 g, 100%). Data for compound **28** were identical to literature values.[10,11] Compound **28** was obtained as 1:1 α:β anomeric mixture as evident by the equal intensity of the anomeric signals in the ^{1}H NMR spectrum [δ 6.50 ppm, J = 3.6 Hz (α-anomeric H) and 5.68 ppm, J = 8.1 Hz (β-anomeric H)].

Trimethyl-[2-[(2*R*,3*S*,4*R*,5*S*,6*R*)-3,4,5-tribenzyloxy-6-(benzyloxymethyl)tetrahydropyran-2-yl]ethynyl]silane (29). Compound **29** was prepared as described in the literature, using compound **28** (3.37 g) to give **29** (65% (lit.[12] 87%)). Data for compound **29** were identical to literature values.[12]

(2*R*,3*S*,4*R*,5*S*,6*R*)-3,4,5-Tribenzyloxy-2-(benzyloxymethyl)-6-ethynyl-tetrahydropyran (30). Compound **30** was prepared as described in the literature, using **29** (1.41 g) to give **30** (68% (lit.[12] 100%)). Data for compound **30** were identical to literature values.[12]



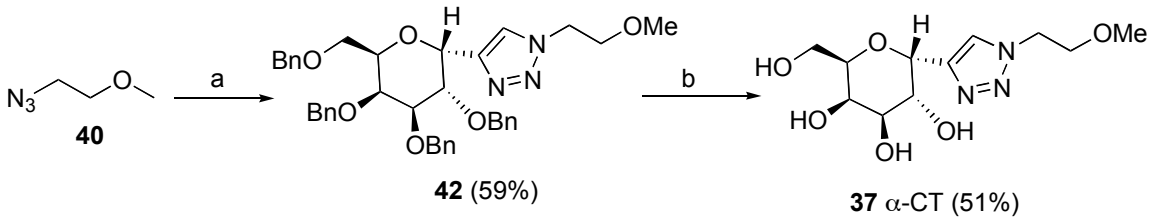
Scheme S1. *Reagents and conditions* **a** Et_3N, TsCl, CH_2Cl_2; **b** EtOH, NaN_3, 85 °C; **c** **21**, Cu(0), TBTA, 2:1:1 *t*BuOH:H_2O:CH_2Cl_2; **d** H_2, 5% Pd/C, EtOH/AcOH.

2-Methoxyethyl 4-methylbenzenesulfonate (39). To a solution of 2-methoxyethanol **38** (3.06 g, 40.2 mmol) in dry CH_2Cl_2 (50 mL) was added Et_3N (15.10 mL, 109 mmol) and the solution was cooled to 0 °C. TsCl was added to this solution and the reaction mixture was allowed to stir at 0°C for 40 min and then 25 h at room temperature. The resulting orange mixture was diluted with CH_2Cl_2 and washed with water. The aqueous fractions were backwashed with CH_2Cl_2 and the combined organic layers were washed with 2% AcOH and dried over $MgSO_4$. The crude oil was dry loaded onto silica and purified by column chromatography on silica gel eluting with 10-67% EtOAc:Hexane to yield compound **39** as a pale yellow oil (7.90 g, 85%). Data for compound **39** were identical to literature values.[13]

1-Azido-2-methoxy-ethane (40). Compound **40** was prepared as described for compound **16**, using **39** (1.03 g). (Note: compound **40** is extremely volatile and most of the CH_2Cl_2 was evaporated at 0 °C with a little bit of CH_2Cl_2 remaining, which prevented a yield being obtained).**WARNING:** No further purification of **40** was attempted due to the hazards involved in distilling low molecular weight azides. Data for compound **40** were identical to literature values.[14]

1-(2-Methoxyethyl)-4-[(2*S*,3*S*,4*R*,5*S*,6*R*)-3,4,5-tribenzyloxy-6-(benzyloxymethyl)tetrahydropyran-2-yl]triazole (41). Compound **41** was obtained by the same procedure used for compound **22**, compound **21** (94 mg, 0.17 mmol), compound **40** (102 mg, excess, not accurate due to volatility of the azide), Cu(0) (54 mg, 5 eq.) and TBTA (1.8 mg, 2mol%). The crude material was dry loaded onto silica and purified by column chromatography on silica gel eluting with 10-67% EtOAc:Hexane to yield compound **41** as a colourless oil (69 mg, 62%). $[\alpha]_D^{20}$ +7 (*c* 1.26, DCM); IR (ATR, cm^{-1}) 3028, 2868, 1722, 1497, 1452, 1362, 1103, ^{1}H NMR δ ($CDCl_3$, 300 MHz), 7.61 (1H, s), 7.41-7.18 (18H, m), 7.06-7.03 (2H, m), 5.03 (1H, d, J = 11.7 Hz), 4.77-4.65 (4H, m), 4.56-4.35 (6H, m), 4.27 (1H, t, J = 9.6 Hz), 4.08 (1H, d, J = 2.1 Hz), 3.76-3.60 (6H, m), 3.24 (3H, s); ^{13}C NMR δ ($CDCl_3$, 75 MHz), 145.9, 138.7, 138.3, 138.2, 137.8, 128.3, 128.1, 128.0, 127.9, 127.7, 127.5, 127.4, 127.3, 123.5, 84.3, 78.4, 77.4, 74.9, 74.6, 74.5, 73.9, 73.4, 72.4, 70.6, 68.6, 58.8 and 50.1. MS-ESI: *m/z* $[M + H]^+$ for $C_{39}H_{44}N_3O_6^+$ calculated 650.3; observed 650.2. HRMS-ESI: *m/z* $[M + Na]^+$ for $C_{39}H_{43}N_3NaO_6^+$, calculated 672.3050; observed 672.3046.

(2*R*,3*R*,4*R*,5*R*,6*S*)-2-(Hydroxymethyl)-6-[1-(2-methoxyethyl)triazol-4-yl]tetrahydropyran-3,4,5-triol (36). Compound **36** was obtained by the same procedure used for compound **23** using compound **41** (60 mg, 0.1 mmol) and 5% Pd/C catalyst (341 mg) in 90% EtOH/AcOH (5.5 mL). Compound **36** was obtained as a pale yellow oil (27 mg, 100%). ^{1}H NMR δ (D_2O, 300 MHz), 8.36 (1H, s), 5.42 (1H, br s), 4.53-4.45 (1H, m), 4.20-3.88 (9H, m), 3.39 (3H, s); ^{13}C NMR δ (D_2O, 75 MHz), 146.4, 126.4, 79.0, 73.6, 73.3, 70.1, 69.2, 68.7, 60.7, 57.8 and 51.3. MS-ESI: *m/z* $[M + H]^+$ for $C_{11}H_{20}N_3O_6^+$ calculated 290.1; observed 290.1. HRMS-ESI: *m/z* $[M + Na]^+$ for $C_{11}H_{19}N_3NaO_6^+$, calculated 312.1172; observed 312.1146.



Scheme S2 *Reagents and conditions:* **a 30**, Cu(0), TBTA, 2:1:1 *t*BuOH:H_2O:CH_2Cl_2; **b** H_2, 5% Pd/C, 90% EtOH:AcOH.

1-(2-Methoxyethyl)-4-[(2*R*,3*S*,4*R*,5*S*,6*R*)-3,4,5-tribenzyloxy-6-(benzyloxymethyl)tetrahydropyran-2-yl]triazole (42). Compound **42** was obtained by the same procedure used for compound **22** using compound **30** (101 mg, 0.18 mmol), compound **40** (165 mg, excess, not accurate due to volatility of the azide), Cu(0) (58 mg, 5 eq.), TBTA (2 mg, 2% mol). The crude material was dry loaded onto silica and purified by column chromatography on silica gel eluting with 10-67% EtOAc:Hexane to yield compound **42** as a colourless oil (71 mg, 59%). $[\alpha]_D^{20}$ +43 (*c* 0.93, DCM); IR (ATR, cm^{-1}) 3028, 2870, 1722, 1495, 1452, 1096, ^{1}H NMR δ ($CDCl_3$, 300 MHz), 7.68 (1H, s), 7.30-7.13 (20H, m), 5.29 (1H, d, J = 4.2 Hz), 4.75-4.43 (9H, m), 4.28-3.87 (5H, m), 3.73-3.67 (3H, m), 3.29 (3H, s); ^{13}C NMR δ ($CDCl_3$, 75 MHz), 144.7, 138.1, 138.0, 137.8, 127.9, 127.6, 127.5, 127.4, 127.3, 127.2, 127.1, 124.3, 76.3, 73.7, 73.4, 73.2, 72.84, 72.78, 72.5, 70.3, 67.0, 66.8, 58.6 and 49.9. MS-ESI: *m/z* $[M + H]^+$ for $C_{39}H_{44}N_3O_6^+$ calculated 650.3; observed 650.3. HRMS-ESI: *m/z* $[M + Na]^+$ for $C_{39}H_{43}N_3NaO_6^+$, calculated 672.3050; observed 672.3036.

(2*R*,3*R*,4*R*,5*R*,6*R*)-2-(Hydroxymethyl)-6-[1-(2-methoxyethyl)triazol-4-yl]tetrahydropyran-3,4,5-triol (37). Compound **37** was obtained by the same procedure used for compound **23** using compound **42** (145 mg, 0.22 mmol) and 5% Pd/C catalyst (888 mg). The crude material was purified via column chromatography on silica gel eluting with 1% AcOH:MeOH. Compound **37** was obtained as a thick yellow oil (33 mg, 51%). ^{1}H NMR δ (D_2O, 300 MHz), 8.39 (1H, s), 6.05 (1H, br s), 4.75-4.67 (2H, m), 4.51 (1H, br s), 4.31 (1H, br s), 4.17 (1H, br s), 4.05-3.85 (4H, m), 3.63 (1H, t, J = 6.1 Hz), 3.40 (3H, s); ^{13}C NMR δ (D_2O, 75 MHz), 143.5, 127.8, 73.7, 69.7, 69.3, 67.6, 67.2, 59.4, 57.9 and 51.2. MS-ESI: *m/z* $[M + H]^+$ for $C_{11}H_{20}N_3O_6^+$ calculated 290.1; observed 290.1. HRMS-ESI: *m/z* $[M + Na]^+$ for $C_{11}H_{19}N_3NaO_6^+$, calculated 312.1172; observed 312.1136.

Biological studies

Control plots were obtained by plotting [(CT concentration) divided by (10^3 × rate of absorbance change)] *vs* (CT concentration). A typical example is shown in Fig. S1, which illustrates the linear nature of the plot. Knowing the intercept and gradient of this plot, the concentration of free CT (i.e., CT not bound to immobilised G_{M1}) could be determined from the rate of

absorbance change at any given concentration of inhibitor of CT binding to immobilised G_{M1}. An example of a polynomial fit to a plot of free [CT] *vs* concentration of inhibitor (in this case compound **37**) is shown in Fig. S2.

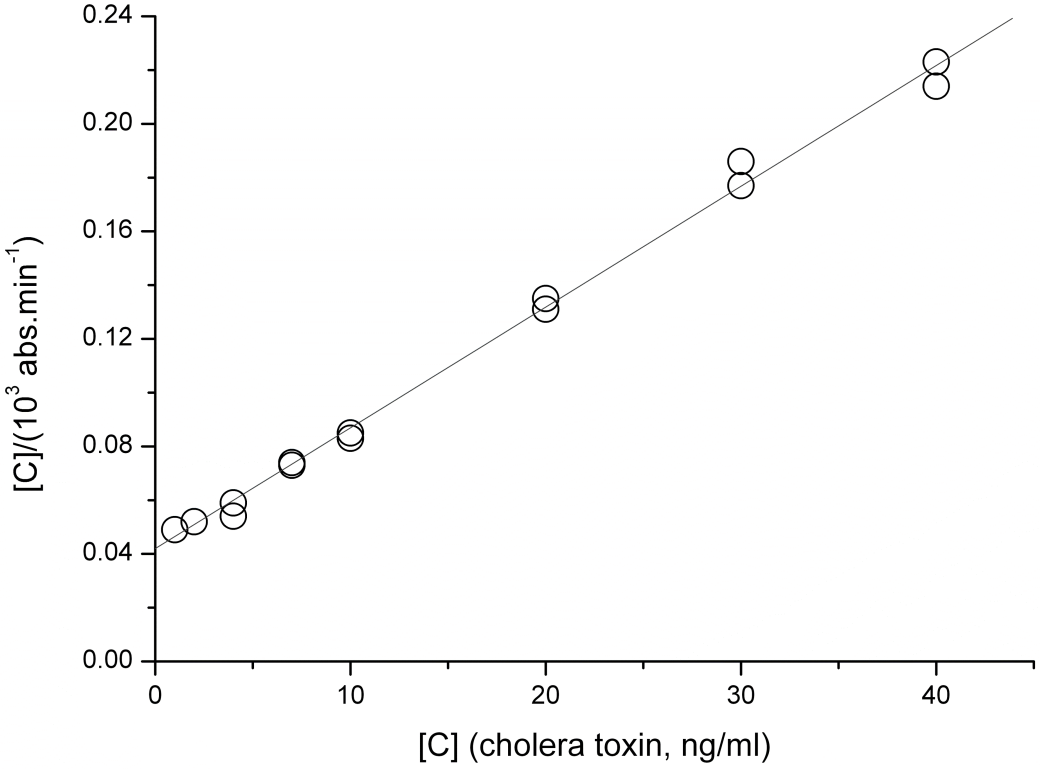


Fig. S1. Linear form of the relationship of rate of absorbance change with concentration of CT in control wells.

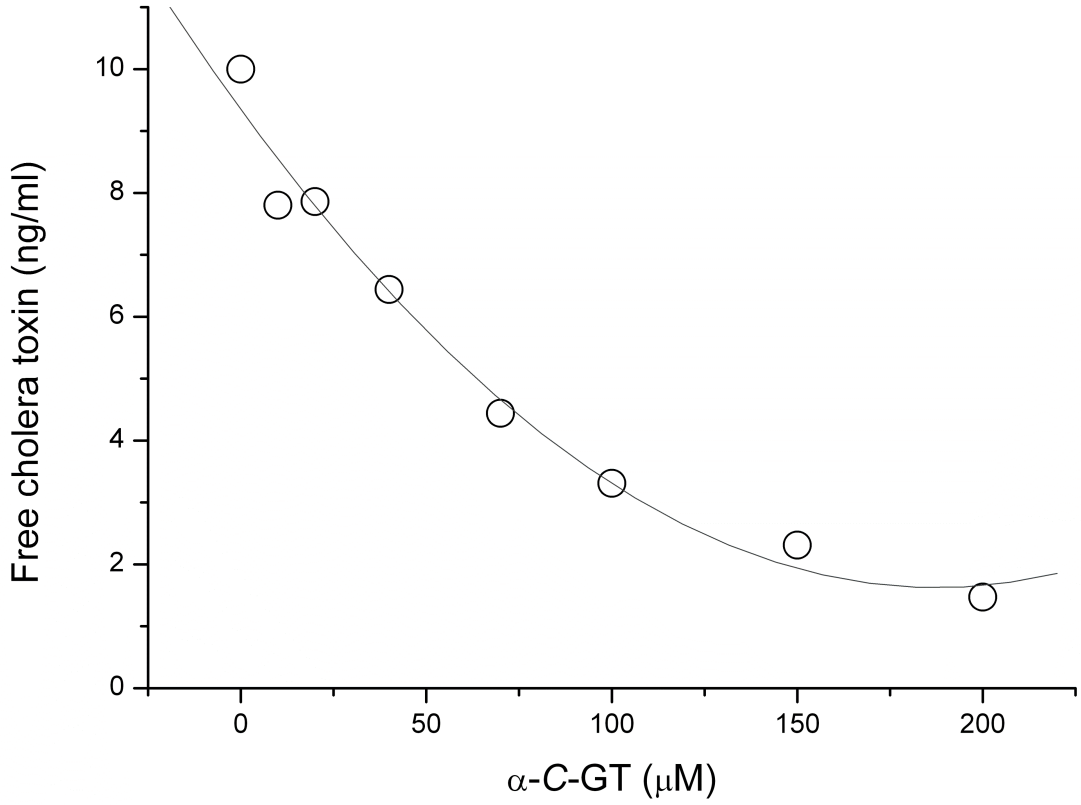


Fig. S2. Inhibition of the binding of 10 ng/ml CT to immobilised GM1 by compound **37** (α-C-GT).

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