Supporting information for

Direct Functionnalization of self-assembled nanotubes overriding unfavorable self-assembling process

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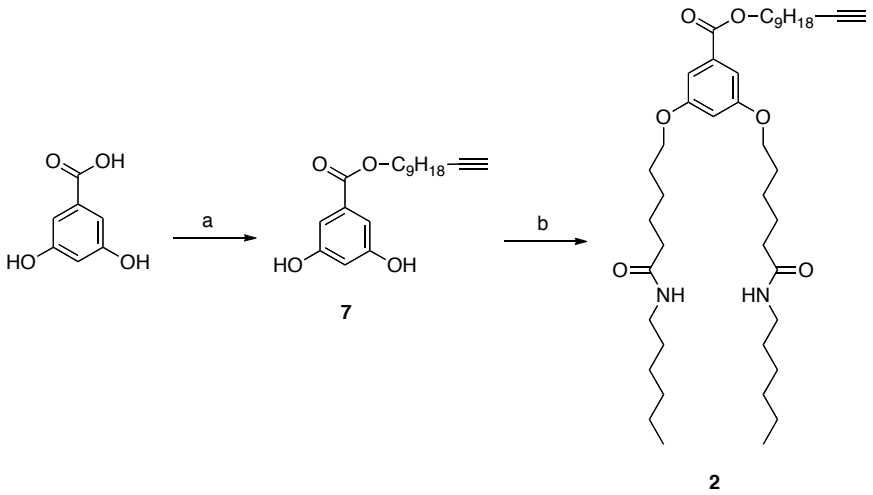
Content :

S1. Synthesis of the starting materials

General : the chemicals were purchased from Aldrich or Acros and used as received. NMR spectra were recorded on a Bruker Avance 400 operating at 400 Mhz for ^{1}H and 100 Mhz for ^{13}C. The FTIR spectra were recorded on a Brucker Vertex 70 spectrometer equipped with a ATR diamond reflection unit (MVPStar). Mass spectra were recorded with a Bruker Daltonique microTOF operating with an electrospray source.



Scheme S1. Synthesis of **2**. a) HO-$C_9H_{18}C{\equiv}CH$, PTSA, Toluene; b) $BrC_5H_{10}CONHC_6H_{13}$, K_2CO_3, Bu_4NBr, DMF, 50 °C.

3,5-Dihydroxy-benzoic acid undec-10-yne ester (**7**) :

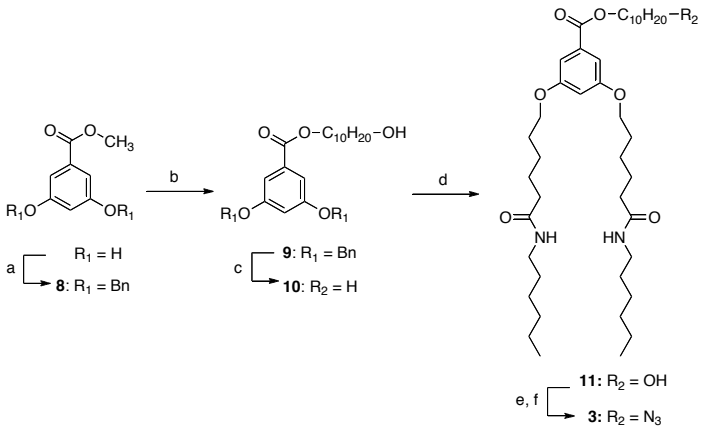
3,5-Dihydroxy-benzoic acid (0.58 g, 3.78 mmol), 10-undecyn-1-ol (0.7 g, 4.16 mmol, 1.1 equiv.) and PTSA (0.36 g, 1.91 mmol, 0.5 equiv.) in solution in toluene (60 mL) were refluxed in a Dean-Stark apparatus. After removal of the water, the mixture was evaporated under vacuum, treated with aqueous $NaHCO_3$ and extracted with CH_2Cl_2 (3 x 20 mL). The combined organic layers were dried (Na_2SO_4) and concentrated under vacuum. The resulting

oil was chromatographed (SiO_2, isopropanol/CH_2Cl_2 : 3/97) to afford pure **7** as a yellow oil (0.48 g, 42 %). ^{1}H NMR (400 MHz, $CDCl_3$) : δ [ppm] 7.08 (d, 2 H, *J* = 6.48 Hz, C2-H, C6-H), 6.60 (s broad, 3 H, C4-H and 2 OH), 4.28 (t, 2 H, *J* = 6.48 Hz, $COOCH_2$), 2.17 (m, 2H, $COO(CH_2)_8C\mathbf{H}_2$), 1.95 (t, 1 H, *J* = 2.56 Hz, $COO(CH_2)_9CC\mathbf{H}$), 1.73 (q, 2 H, *J* = 6.8 Hz, $COOCH_2C\mathbf{H}_2$), 1.51 (q, 2 H, *J* = 7.0 Hz, $COO(CH_2)_7C\mathbf{H}_2$), 1.40-1.30 (m, 10 H, $COO(CH_2)_2(C\mathbf{H}_2)_4$); ^{13}C NMR (100 MHz, $CDCl_3$) : δ [ppm] 167.6 (COO), 157.3 (C3, C5), 132.5 (C1), 109.4 (C2, C6), 108.0 (C4), 85.24 ($\mathbf{C}\blacksquare CH$), 68.5 ($C\blacksquare\mathbf{C}H$), 66.2 ($ArCOO\mathbf{C}H_2$), 29.7 $COO(CH_2)_4\mathbf{C}H_2$), 29.5 ($COO(CH_2)_3\mathbf{C}H_2$), 29.3 ($ArCOOCH_2\mathbf{C}H_2$), 29.0 ($COO(CH_2)_5\mathbf{C}H_2$), 28.9 ($COO(CH_2)_6\mathbf{C}H_2$), 28.8 ($COO(CH_2)_7\mathbf{C}H_2$), 26.3 ($COO(CH_2)_2\mathbf{C}H_2$), 18.70 ($COO(CH_2)_8\mathbf{C}H_2$). ATR-IR (diamond) ν_{max} : 3404 (broad, ν OH), 3257 (ν ≡C-H), 2926 (ν_{as} CH_2), 2852 (ν_s, CH_2), 2108 (w, (ν C≡C), 1695 (s), 1684 (s), 1622 (s), 1599 (s), 1476 (w), 1465 (s), 1433 (w), 1390 (s), 1333 (s), 1305 (s), 1270 (s), 1255 (vs), 1232 (m), 1161 (vs), 1113 (m), 1103 (m), 1069 (w), 1044, 1003 (vs) and 991 (s), 969 (s), 943, 896, 875, 865, 847, 769 (vs), 724 (w), 674 (vs), 656 (s), 626 (s) cm^{-1}; HRMS (ESI+) m/z 327.1553 (MNa^+, calcd for $C_{18}H_{24}O_4$: 327.1598). Anal. Found : C, 70.93; H, 7.99. Calcd for $C_{18}H_{24}O_4$: C, 71.03 ; H, 7.95.

3,5-Bis-(5-hexylcarbamoyl-pentyloxy)-benzoic acid undec-10-yne ester (**2**) :

Compound **7** (0.30 g, 0.98 mmol), 6-bromo-*N*-hexylhexanamide (0.58 g, 2.07 mmol, 2.1 equiv.) and Bu_4NBr (0.13 g, 0.39 mmol, 0.4 equiv.) in DMF (20 ml) were treated with K_2CO_3 (0.68 g, 4.93 mmol, 5 equiv.). The mixture was heated at 50°C during 12 hrs under argon. The reaction mixture was mixed with water (400 mL) and acidified to pH 1-2 with aqueous HCl (10 %). The resulting precipitate was recrystallized from acetonitrile to afford **2** as a white solid (0.57 g, 82 % yield). M. p. 84.5 °C; ^{1}H NMR (400 MHz, $CDCl_3$) : δ [ppm] 7.13 (d, 2 H, *J* = 2.1 Hz, C2-**H**, C6-**H**), 6.61 (t, 1 H, *J* = 2.1 Hz, C4-**H**), 5.43 (s broad, 2 H, N**H**), 4.28 (t, 2 H, *J* = 6.6 Hz, $COOC\mathbf{H}_2$), 3.97 (t, 4 H, *J* = 6.5 Hz, $ArOC\mathbf{H}_2$), 3.23 (q, 4 H, *J* = 7.16 Hz, $C\mathbf{H}_2NHCO$), 2.17 (t, 4 H, *J* = 7.1 Hz, $C\mathbf{H}_2CONH$), 1.80-1.67 (m, 9 H, $COO(CH_2)_8C\mathbf{H}_2$, $COO(CH_2)_9CC\mathbf{H}$, $COOCH_2C\mathbf{H}_2$, $ArOCH_2C\mathbf{H}_2$), 1.54-1.29 (36 H, $NHCOCH_2C\mathbf{H}_2$, $NHCO(CH_2)_2C\mathbf{H}_2$, $CONHCH_2(C\mathbf{H}_2)_4$, $COO(CH_2)_2(C\mathbf{H}_2)_6$), 0.87 (t, 6 H, *J* = 6.84 Hz, $C\mathbf{H}_3$); ^{13}C NMR (100 MHz, $CDCl_3$) : δ [ppm] 173.0 (CONH), 166.8 (COO), 160.4 (C3, C5), 132.6 (C1), 108.1 (C2, C6), 106.6 (C4), 85.1 ($COO(CH_2)_9\mathbf{C}CH$), 68.4 ($C\equiv\mathbf{C}H$), 68.3 ($ArO\mathbf{C}H_2$), 65.6 ($COO\mathbf{C}H_2$), 39.9 ($\mathbf{C}H_2NH$), 37.1 ($\mathbf{C}H_2CONH$), 31.8 ($CH_3CH_2\mathbf{C}H_2$), 30.0 ($NHCH_2\mathbf{C}H_2$), 29.7 ($COO(CH_2)_3\mathbf{C}H_2$), 29.5 ($COO(CH_2)_4\mathbf{C}H_2$), 29.4 ($ArOCH_2\mathbf{C}H_2$), 29.3 ($COO(CH_2)_5\mathbf{C}H_2$), 29.0 ($COO(CH_2)_6\mathbf{C}H_2$), 28.8 ($COO(CH_2)_7\mathbf{C}H_2$), 26.9 ($NH(CH_2)_2\mathbf{C}H_2$), 26.3

($COO(CH_2)_2\mathbf{C}H_2$), 26.1 ($NHCOCH_2\mathbf{C}H_2$), 25.8 ($NHCO(CH_2)_2\mathbf{C}H_2$), 22.9 ($CH_3\mathbf{C}H_2$), 18.7 ($COO(CH_2)_8\mathbf{C}H_2$), 14.3 ($CH_3$); ATR-IR (diamond) ν_{max} : 3287 (ν NH), 3093, 2952 , 2919 (ν_{as} CH_2), 2852 (ν_s CH_2), 2117(w, ν C≡C), 1713 (s, ν CO ester), 1643 (s, amide I), 1600, 1551 (s, amide II), 1466, 1446, 1321, 1301 (amide III), 1230, 1166 cm^{-1}; HRMS (ESI+) m/z 705.5343 (MLi^+, calcd for $C_{42}H_{70}N_2O_6$: 705.5389). Anal. Found : C, 71.91 ; H 10.14 ; N, 3.69. Calcd for $C_{42}H_{70}N_2O_6$: C, 72.17 ; H 10.09 ; N, 4.01.



Scheme S2. Synthesis of **3**. a) BnBr, K_2CO_3, Bu_4NBr, DMF, 50 °C; b) $HO(CH_2)_{10}OH$, NaH, THF, 25 °C; c) H_2, Pd/C; d) $BrC_5H_{10}CONHC_6H_{13}$, K_2CO_3, Bu_4NBr, DMF, 50 °C; e) CH_3SO_2Cl, NEt_3, THF; f) NaN_3, Bu_4NBr, CH_3CN, reflux.

Methyl 3,5-dibenzyloxybenzoate (**8**) :

A solution of methyl 3,5-dihydroxy benzoate (15 g, 89.2 mmol), benzyl bromide (21.75 mL, 182.9 mmol, 2.05 equiv.) and tetrabutylammonium bromide (14.38 g, 44.6 mmol, 0.5 equiv.) in DMF (500 mL) was stirred for 10 min under argon at 50 °C and K_2CO_3 (61.55 g, 0.44 mol, 5 equiv.) was added. The mixture was stirred during 24 hrs at 50 °C and poured into an aqueous solution of HCl (0.1 M, 3 L) and the pH was adjusted to 1 with a 1 M HCl aqueous solution. The heterogeneous mixture was stirred for 3 hrs and filtered. The solid was dissolved in CH_2Cl_2, dried with $MgSO_4$ and concentrated under vacuum. The residue was recrystallized from EtOH to afford pure **8** as a white solid (20 g, 64.5 %). M.p. 69.2 °C. ^{1}H NMR (400 MHz, $CDCl_3$): δ [ppm] 7.44-7.30 (m, 12 H_{ar}), 6.81 (t, 1 H, *J* = 2.24 Hz, C4-**H**), 5.07 (s, 4 H, Ar-O-C$\mathbf{H}_2$-Ar), 3.91 (s, 3 H, COOC$\mathbf{H}_3$). ^{13}C NMR (100 MHz, $CDCl_3$): δ [ppm] 167.1 (COO), 160.1 (C3, C5), 136.8 (C1'), 132.4 (C1), 129.0 (C3'), 128.5 (C4'), 127.9 (C2'), 108.7 (C2), 107.6 (C4), 70.6 (Ar$\mathbf{C}H_2$-O), 52.6 (COO$\mathbf{C}H_3$). IR (ATR-diamond) ν_{max}: 3029 (ν

Ar-H), 2920 (ν_{as}, CH_2), 2862 (ν_s, CH_2), 1711 (νC=O), 1596 (ν C=C), 1499, 1471, 1453, 1438, 1378, 1353, 1297, 1252, 1237, 1211, 1164, 1107, 1084, 1063 (ν C-O-C), 1046, 1031, 1004, 966, 898, 866, 839, 760, 729, 694 cm^{-1}. HRMS (ESI+) m/z 355.1343 (MLi^+, calcd for $C_{22}H_{20}O_4$: 355.1560). Anal. Found : C, 75.87; H, 5.77. Calcd for $C_{22}H_{20}O_4$: C, 75.84; H, 5.79.

3,5-Bis-benzyloxy-benzoic acid 10-hydroxy-decyl ester (**9**) :

A mixture of 1,10-decanediol (7.5 g, 43.05 mmol, 3 equiv.) and NaH (60 % in paraffin, 0.69 g, 17.2 mmol, 1.2 equiv.) in anhydrous THF (200 mL) was stirred for 15 min at 25 °C. Methyl 3,5-dibenzyloxybenzoate (**8)** (5.0 g, 14.35 mmol) was added and the mixture was stirred during 24 hrs. The solvent was evaporated under vacuum and the residue was mixed with AcOEt (50 mL) and aqueous HCl (1M, 200 mL). The organic layer was separated and the aqueous phase was extracted with AcOEt (3x 40 mL); the combined organic phases were washed with brine, dried on $MgSO_4$, evaporated under vacuum to yield a syrup. Chromatography (SiO_2, (AcOEt/cyclohexane : 3/7) afforded pure (**9**) as a white solide (6.86 g, 65 %). M.p. 58.5 °C. ^{1}H NMR (400 MHz, $CDCl_3$): δ [ppm] 7.45-7.34 (m, 12 H_{ar}), 6.86 (t, 1 H, *J* = 2.4 Hz, C4-H), 5.10 (s, 4 H, Ar-O-C**H**$_2$-Ar), 4.3 (t, 2 H, *J* = 6.64 Hz, COOCH2), 3.62 (t, 2 H, *J* = 6.68 Hz, CH_2OH), 1.74 (m, 2 H, $COOCH_2\mathbf{C}H_2$), 1.55 (m, 2H, $\mathbf{C}H_2CH_2OH$), 1.40-1.25 (12 H, 6 CH_2). ^{13}C NMR (100 MHz, $CDCl_3$): δ [ppm] 166.7 (COO), 160.1 (C3, C5), 136.8 (C1'), 132.8 (C1), 129.0 (C3'), 128.5(C4'), 128.0 (C2'), 108.8 (C2), 107.3 (C4), 70.7 ($ArCH_2$-O), 65.7 ($COO\mathbf{C}H_2$), 63.4 (CH_2OH), 33.1 ($\mathbf{C}H_2CH_2OH$), 29.9, 29.8, 29.7, 29.6 ($COO(CH_2)_3(\mathbf{C}H_2)_4(CH_2)_3OH$), 26.3 ($\mathbf{C}H_2(CH_2)_2OH$), 26.0 ($COO(CH_2)_2\mathbf{C}H_2$). IR (ATR-diamond) ν_{max}: 3408 (broad, νO-H), 3035 (ν Ar-H), 2921 (ν_{as}, CH_2), 2852 (ν_s, CH_2), 1712 (ν C=O), 1596 (ν C=C), 1499, 1455, 1444, 1378, 1348, 1297, 1228, 1162 (ν_{as} C-O-C), 1081 ,1057, 1027 (ν_s C-O-C), 993, 974907, 845, 780,765, 735 cm^{-1}. HRMS (ESI+) m/z 513.2584 (MNa^+, calcd for $C_{31}H_{38}O_5$: 513.2617). Anal. Found : C, 75.95; H, 7.85. Calcd for $C_{31}H_{38}O_5$: C, 75.89; H, 7.81.

3,5-Dihydroxy-benzoic acid 10-hydroxy-decyl ester (**10**) :

A mixture of **9** (1 g, 2.04 mmol) and Pd/C 10 % (300 mg, 30 % wt) in ethyl acetate (40 mL) was hydrogenated at 25 °C and room pressure during 5 hrs. The catalyst was removed by filtration with paper filter (5 layers folded). The solvent was evaporated to afford pure 3,5-dihydroxy-benzoic acid **10** (0.62 g, 100 %). M.p. 104 °C. ^{1}H NMR (400 MHz, $(CD_3)_2CO$): δ

[ppm] 8.50 (s, 2 H, OH-phenol), 7.01 (s, 2 H, C2-**H** and C6-**H**), 6.57 (t, 1 H, *J* = 2.4 Hz, C4-H), 5.10 (s, 4 H, Ar-O-C**H**$_2$-Ar), 4.3 (t, 2 H, *J* = 6.6 Hz, COOCH_2), 3.54 (t, 2 H, *J* = 6.7 Hz, CH_2OH), 3.45 (s broad, 1 H, -CH_2-O**H**), 1.74 (m, 2 H, COOCH_2**C**H_2), 1.51-1.20 (m, 14 H, 7 **C**H_2). ^{13}C NMR (100 MHz, $(CD_3)_2CO$): δ [ppm] 166.6 (COO), 159.4 (C3,C5), 133.4 (C1), 108.6 (C2 and C6), 107.8 (C4), 65.3 (COOCH_2), 62.5 (CH_2OH), 33.7 (**C**H_2CH_2OH), 30.1 (COO(CH_2)$_3$(**C**H_2)$_4$(CH_2)$_3$OH) 26.6(**C**H_2CH_2CH_2OH). ATR-IR (solid/diamond) ν_{max}: 3445 (broad, νO-H), 3250 (ν ArC-H), 2929 (ν_{as} CH_2), 2852 (ν_s CH_2), 1694 (ν C=O), 1605 (ν C=C), 1457(ν C=C), 1395, 1343 (δ O-H phenol), 1239, 1155 (ν_{as} C-O-C), 1121, 1042 (ν_S Ar-O), 1003, 967, 865, 769 (s), 726, 679, 620, 586 cm^{-1}. HRMS (ESI+) m/z 317.1917 (MLi^+, calcd for $C_{17}H_{26}O_5$: 317.1940). Anal. Found : C, 65.40; H, 8.62. Calcd for $C_{17}H_{26}O_5$: C, 65.78; H, 8.44.

3,5-Bis-(5-hexylcarbamoyl-pentyloxy)-benzoic acid 10-hydroxy-decyl ester (**11**) :

A solution of **10** (400 mg, 1.29 mmol), 6-bromo-*N*-hexylhexanamide (735 mg, 2.64 mmol, 2.05 equiv.), and tetrabutylammonium bromide (166 mg, 0.52 mmol, 0.4 equiv.) in anhydrous DMF (50 mL) under argon was heated at 50 °C during 10 min. K_2CO_3 (0.89 g, 6.44 mmol, 5 equiv.) was introduced and the mixture was stirred under Ar for 20 hrs. The reaction medium was neutralized with a solution of HCl (0.1 M) to pH 1 and mixed with water (200 mL). The resulting suspension was stirred 2 hrs and filtrated to give a gray solid. This solid was recrystallized twice from cyclohexane and once from cold acetonitrile to afford **11** as a white solid (0.8 g, 90 %). M.p. 79.8 °C. ^{1}H NMR (400 MHz, $CDCl_3$): δ [ppm] 7.14 (s, 2 H, C2-H and C6-H), 6.60 (t, 1 H, *J* = 2.4 Hz, C4-H), 5.54 (s, broad, 2 H, NH), 4.30 (t, 2 H, *J* = 6.6 Hz, COOCH_2), 3.97 (t, 4 H, *J* = 6.3 Hz, ArOCH_2), 3.63 (t, 2 H, *J* = 6.6 Hz, CH_2OH), 3.24 (q, 4 H, *J* = 7.2 Hz, CH_2NHCO), 2.20 (t, 4 H, *J* = 7.5 Hz, CH_2CONH), 1.74 (m, 10 H, COOCH_2C**H$_2$**, ArOCH_2C**H$_2$**, ArOCH_2CH_2C**H$_2$**), 1.57-1.26 (m, 34 H, CH_2), 0.87 (t, 6 H, *J* = 6.8 Hz, CH_3). ^{13}C NMR (100 MHz, $CDCl_3$) : δ [ppm] 172.9 (CONH), 166.7 (COO), 160.1 (C3,C5), 132.4 (C1), 107.8 (C2, C6), 106.3 (C4), 68.1 (ArOCH_2), 65.4 (COO**C**H_2), 63.1 (CH_2OH), 39.7 (CH_2NH), 36.8 (**C**H_2CONH), 32.9 (**C** H_2CH_2OH), 31.6 (CH_3CH_2**C**H_2), 31.1 (CONHCH_2**C**H_2), 29.8 (COO(CH_2)$_3$**C**H_2), 29.6 (COO(CH_2)$_4$**C**H_2), 29.5 (COO(CH_2)$_5$**C**H_2), 29.3 (COO(CH_2)$_6$**C**H_2), 29.1 (ArOCH_2**C**H_2), 28.8 (COOCH_2**C**H_2), 26.7 (CONH(CH_2)$_2$**C**H_2), 26.1 (COO(CH_2)$_2$**C**H_2), 25.9 (**C** H_2CH_2CONH), 25.9 (**C** H_2(CH_2)$_2$OH), 25.6 (**C**H_2(CH_2)$_2$CONH), 22.7 (CH_3**C**H_2), 14.2 (CONH(CH_2)$_5$**C**H_3). ATR-IR (solid/diamond) ν_{max}: 3294 (ν NH), 3100 (ν ArC-H), 2924 (ν_{as} CH_2), 2853 (ν_s, CH_2), 1721 (ν C=O), 1639

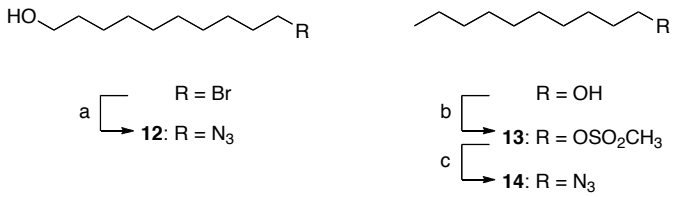
(amide I), 1597 (ν C=C), 1549 (amide II), 1445, 1349, 1299 (amide III), 1231, 1170 (ν_{as} Ar-O-C), 1042 (ν_s Ar-O-C), 763, 722 cm^{-1}. HRMS (ESI+) m/z 711.5399 (MLi^+, calcd for $C_{41}H_{72}N_2O_7$: 711.5500). Anal. Found C, 69.52; H, 10.32 ; N, 3.95. Calcd for $C_{41}H_{72}N_2O_7$: C, 69.85; H, 10.29; N, 3.97.

3,5-Bis-(5-hexylcarbamoyl-pentyloxy)-benzoic acid 10-azido-decyl ester (**3**) :

A solution of **11** (100 mg, 148 µmol), TEA (40 µL, 0.28 mmol, 2 equiv.) in anhydrous THF (30 mL) was stirred at 0°C for 10 min before adding dropwise a freshly prepared solution of mesyl chloride (32 mg, 0.28 mmol, 2 equiv.) in THF (5 mL). After 20 min, the reaction was quenched by water. The THF was evaporated and the residue was extracted with CH_2Cl_2 (3 x 20 mL). The organic layers were dried ($MgSO_4$) and concentrated under vacuum. The residue was chromatographed (SiO_2, (MeOH/CH_2Cl_2 : 4/96) to afford 3,5-bis-(5-hexylcarbamoyl-pentyloxy)-benzoic acid 10-methanesulfonyloxy-decyl ester as a white solid, that was used as is for the following step : ^{1}H NMR (400 MHz, $CDCl_3$): δ [ppm] 7.11 (d, 2 H, *J* = 2.2 Hz,, C2-H and C6-H), 6.57 (t, 1 H, *J* = 2.4 Hz, C4-H), 5.64 (s broad, 2 H, NH), 4.25 (t, 2 H, *J* = 6.6 Hz, $COOCH_2$), 4.20 (t, 2 H, *J* = 6.7 Hz, CH_2O-Ms), 3.94 (t, 4 H, *J* = 6.3 Hz, $ArOCH_2$), 3.21 (q, 4 H, *J* = 6.0 Hz, CH_2NH), 3.0 (s, 3 H, SO_2CH_3) 2.17 (t, 4 H, *J* = 7.4 Hz, C**H**$_2$CONH), 1.72 (m, 12 H, $COOCH_2CH_2$, $ArOCH_2CH_2$, $MsOCH_2CH_2$, $NHCOCH_2CH_2$), 1.53-1.28 (m, 30 H, CH_2), 0.84 (t, 6 H, *J* = 6.5 Hz, CH_3). ^{13}C NMR (100 MHz, $CDCl_3$) : δ [ppm] 173.0 (CONH), 166.8 (COO), 160.2 (C3,C5), 132.4 (C1), 107.9 (C2, C6), 106.3 (C4), 70.4 ($ArOCH_2$), 68.2 ($COOCH_2$), 65.4(**C**H_2-OMs), 39.7 (CH_2NH), 37.5 (**C**H_2CONH), 36.9 (SO_2-**C**H_3), 31.7 ($NH(CH_2)_3$**C**H_2), 29.8-29.0 ($COO(CH_2)_2$(**C**H_2)$_4$, $ArOCH_2$**C**H_2), 26.8-25.7 ($NH(CH_2)_2$**C**H_2, $COO(CH_2)_2$**C**H_2, $CH_2(CH_2)_2OMs$, $NHCOCH_2$**C**H_2, $ArO(CH_2)_2$**C**H_2), 22.7 (CH_3**C**H_2), 14.2 (**C**H_3).

A mixture of this compound (111 mg, 0.16 mmol,), NaN_3 (26 mg, 1.42 mmol, 10 equiv.) and Bu_4NBr (20 mg, 63 µmol, 0.4 equiv.) in acetone (12 mL) was stirred in reflux for 48 hrs. The reaction was let cool to 25 °C and filtrated. The filtrate was evaporated under vacuum and chromatographed (SiO_2, MeOH/ CH_2Cl_2 : 2/98) to afford pure **3** as a white solid (100 mg, yield based on **11** : 97 %). ^{1}H NMR (400 MHz, $CDCl_3$): δ [ppm] 7.12 (d, 2 H, *J* = 2.2 Hz, C2-H and C6-H), 6.60 (t, 1 H, *J* = 2.4 Hz, C4-H), 5.56 (s broad, 2 H, NH), 4.27 (t, 2 H, *J* = 6.80 Hz, $COOCH_2$), 3.95 (t, 4 H, *J* = 6.32 Hz, $ArOCH_2$), 3.23 (m, 6 H, CH_2NH and CH_2-N_3), 2.18 (t, 4 H, *J* = 7.7 Hz, C**H**$_2$CONH), 1.80-1.67 (m, 10 H, $COOCH_2CH_2$, $ArOCH_2$C**H**$_2$, $NHCOCH_2$C**H**$_2$), 1.50-1.24 (34 H, CH_2), 0.86 (t, 6 H, *J* = 6.80 Hz, CH_3). ^{13}C NMR (100

MHz, $CDCl_3$) : δ [ppm] 173.0 (CONH), 166.8 (COO), 160.3 (C3,C5), 132.6 (C1), 108.0 (C2, C6), 106.5 (C4), 68.3 ($ArOCH_2$), 65.6 ($COOCH_2$), 51.8 ($\mathbf{C}H_2N_3$), 39.8 (CH_2NH), 37.0 ($\mathbf{C}H_2CONH$), 31.8 ($NH(CH_2)_3\mathbf{C}H_2$), 29.93-28.97 ($NHCH_2\mathbf{C}H_2$, $COOCH_2\mathbf{C}H_2CH_2(\mathbf{C}H_2)_4CH_2\mathbf{C}H_2$, $ArCH_2\mathbf{C}H_2$), 27.0 ($N_3(CH_2)_2\mathbf{C}H_2$), 26.9 ($NH(CH_2)_2\mathbf{C}H_2$), 26.3 ($COO(CH_2)_2\mathbf{C}H_2$), 26.1 ($NHCOCH_2\mathbf{C}H_2$), 25.8 ($NHCO(CH_2)_2\mathbf{C}H_2$), 22.9 ($CH_3\mathbf{C}H_2$), 14.3 ($CH_3$). IR (ATR-diamond) ν_{max}: 3298 (ν NH), 3100, 2926 (ν_{as}, CH_2), 2854 (ν_s, CH_2), 2098 (s, ν_{op} N_3), 1715 (ν C=O, ester), 1637 (amide I), 1598 (ν C=C), 1544 (amide II), 1464, 1345, 1299 (amide III), 1238, 1173, 1067, 763, 724 cm^{-1}. HRMS (ESI+) m/z 736.5510 (MLi^+, calcd for $C_{41}H_{71}N_5O_6$: 736.5564). Anal. Found : C, 67.75; H, 9.85; N, 9.69. Calcd for $C_{41}H_{71}N_5O_6$: C, 67.45; H, 9.80; N, 9.59.



Scheme S3. Synthesis of the reagents. a) NaN_3, CH_3CN, reflux; b) CH_3SO_2Cl, NEt_3, THF; c) NaN_3, CH_3CN, reflux;

10-Azido-decan-1-ol (**12**) :

Bromodecanol (40 g, 169 mmol) and Bu_4NBr (1.1 g, 4 mmol, 0.02 equiv.) were added to a suspension of NaN_3 (27 g, 415 mmol, 2.5 equiv.) in acetonitrile (300 mL). The mixture was stirred at 25 °C for 5 days, evaporated under vacuum, dissolved in EtOAc (300 mL) and washed with water (3 x 200 mL). The organic layer was dried (Na_2SO_4) concentrated under vacuum. The residue was purified by chromatography (SiO_2, EtOAc/cyclohexane : 10:90 to 25:75 gradient elution) to yield pure **12** as a yellow oil (29.6 g, 88 %). ^{1}H NMR (400 MHz, $CDCl_3$) : δ [ppm], 3.58 (m, 2H, $\mathbf{C}H_2OH$), 3.22 (t, 2H, J = 6.8 Hz, $C\mathbf{H}_2N_3$), 2.00 (s, broad, 1H, OH), 1.54 (m, 4 H, $C\mathbf{H}_2CH_2N_3$ and $C\mathbf{H}_2CH_2OH$), 1.32-1.26 (12 H, CH_2). ^{13}C NMR (100 MHz, $CDCl_3$) : δ [ppm] 63.1 ($\mathbf{C}H_2OH$), 51.7 ($\mathbf{C}H_2N_3$), 33.0 ($\mathbf{C}H_2CH_2OH$), 29.7 ($\mathbf{C}H_2CH_2N_3$), 29.6 ($\mathbf{C}H_2(CH_2)_3OH$ and $\mathbf{C}H_2(CH_2)_4OH$), 29.4 ($\mathbf{C}H_2(CH_2)_5OH$), 29.1 ($\mathbf{C}H_2(CH_2)_6OH$), 26.9 ($\mathbf{C}H_2(CH_2)_7OH$), 26.0 ($\mathbf{C}H_2(CH_2)_2OH$). FTIR (ATR-diamond, neat) ν_{max} : 3600-3200 (broad, ν OH), 2925 (ν_{as} CH_2), 2854 (ν_s, CH_2), 2090 (s, ν_{op} N_3), 1464 (m), 1349 (w), 1258 (br), 922, 1055, 722, 635 cm^{-1}. HRMS (ESI+) m/z 206.1837 (MLi^+, calcd for $C_{10}H_{21}N_3O$: 206.1845). Anal. Found : C, 60.41 ; H 10.82 ; N, 20.93. Calcd for $C_{10}H_{21}N_3O$: C, 60.27 ; H, 10.62 ; N, 21.08.

Methanesulfonic acid decyl ester (**13**) :

A solution of 1-decanol (8.29 g, 10 ml, 52.3 mmol) and TEA (20 ml, 144 mmol, 2.75 equiv.) in anhydrous THF (600 ml) was stirred at 0°C and a freshly prepared solution of mesyl chloride (10 mL, 129 mmol, 2.5 equiv) in THF (100 mL) was added dropwise. After 5 hrs, the mixture was filtered and the filtrate was concentrated under vacuum. The residue was chromatographed (SiO_2, CH_2Cl_2/pentane : 1/1) to afford **13** as a colorless oil (12.0 g, 97 %). ^{1}H NMR (400 MHz, $CDCl_3$) : δ [ppm], 4.19 (t, 2H, J = 6.5 Hz, C**H**$_2$OMs), 2.97 (s, 3 H, OSO_2C**H**$_3$), 1.73 (p, 2 H, J = 6.5 Hz, C**H**$_2$CH$_2$OMs), 1.37-1.24 (14 H, CH_2), 0.85 (t, 3 H, J = 6.8 Hz, C**H**$_3$). ^{13}C NMR (100 MHz, $CDCl_3$) : δ [ppm] 70.5 (**C**H$_2$OMs), 37.5 (OSO_2**C**H$_3$), 32.1 (CH_3CH_2**C**H$_2$), 29.7 (**C**H$_2$(CH$_2$)$_3$OMs), 29.7 (**C**H$_2$(CH$_2$)$_4$OMs), 29.5 (**C**H$_2$(CH$_2$)$_5$OMs), 29.4 (**C**H$_2$(CH$_2$)$_6$OMs), 29.3 (**C**H$_2$CH$_2$OMs**),** 25.7 (**C**H$_2$(CH$_2$)$_2$OMs), 22.9 (CH_3**C**H$_2$), 14.3 (CH_3). FTIR (ATR-diamond, neat) ν_{max} : 2962, 2959, 2926, 2924, 1466 (δ CH_3), 1353 (s), 1330, 1172 (s, ν SO_2), 975, 953, 929 (s), 836, 803, 749, 720. HRMS (ESI+) m/z 243.1590 (MLi$^+$, calcd for $C_{11}H_{24}O_3S$: 243.1606). Anal. Found : C, 56.11 ; H, 10.34. Calcd for $C_{11}H_{24}O_3S$: C, 55.89 ; H, 10.23.

Azidodecane (**14**) :

Methanesulfonic acid decyl ester (**13**) (4 g, 16.92 mmol) and NaN_3 (3.3 g, 50.7 mmol, 3 equiv.) in acetonitrile (100 mL) were refluxed during 40 hrs at 100 °C. After removal of NaN_3 in excess, the solvent was evaporated under vacuum to afford pure azidodecane **14** as a colorless oil (3.1 g, 97 %). ^{1}H NMR (400 MHz, $CDCl_3$) : δ [ppm], 3.26 (t, 2H, J = 6.7 Hz, C**H**$_2$N$_3$), 1.59 (m, 2 H, C**H**$_2$CH$_2$N$_3$), 1.27 (14 H, CH_2), 0.88 (t, 3 H, J = 6.8 Hz, C**H**$_3$). ^{13}C NMR (100 MHz, $CDCl_3$) : δ [ppm] 51.8 (**C**H$_2$N$_3$), 32.2 (**C**H$_2$(CH$_2$)$_7$N$_3$), 29.8 (**C**H$_2$(CH$_2$)$_4$N$_3$), 29.8 (**C**H$_2$(CH$_2$)$_5$N$_3$), 29.6 (**C**H$_2$CH$_2$N$_3$), 29.5 (**C**H$_2$(CH$_2$)$_3$N$_3$), 29.2 (**C**H$_2$(CH$_2$)$_6$N$_3$), 27.1 (**C**H$_2$(CH$_2$)$_2$N$_3$), 23.0 (**C**H$_2$(CH$_2$)$_8$N$_3$), 14.4 (CH_3). IR (ATR-diamond) ν_{max} : 2957 (ν_{as}, CH_3), 2926 (ν_{as}, CH_2), 2872 (ν_s, CH_3), 2860 (ν_s, CH_2), 2094 (s, ν_{op} N_3), 1463, 1375, 1351, 1287, 1261, 1176, 971, 896, 781, 724. MS (+ESI) m/z 183.1 (MLi$^+$, calcd for $C_{10}H_{21}N_3$: 183.2). Anal. Found : C, 65.29; H, 11.55; N, 22.20. Calcd for $C_{10}H_{21}N_3$: C, 65.53; H, 11.55; N, 22.92.

S2. Click reactions with the tubes and studies of the resulting tubes.

Formation and functionnalization of the nanotubes

2 was mixed with cyclohexane (51 mg, 2 % wt. of **2**) degassed with Ar in a screw-cap vial, with a Teflon gasket between the glass and the cap. The mixture was heated until complete dissolution of the solid, and let cool at 25 °C until the formation of the gel. A saturated solution of $Cu(PPh_3)_3Br$ in cyclohexane was prepared by mixing the catalyst (66 mg, 71.5 µmol) with cyclohexane (2.2 g). This solution was filtrated and mixed with a solution of 10-azidodecan-1-ol **12** (150 mg, 750 µmol) in C_6H_{12} (1.3 g). The resulting solution was layered on top of **2**/ cyclohexane gel, under Ar. The gel was let under Ar during 14 days. The tube was opened and the top solution was removed with a Pasteur pipet, and replaced by a solution of acetylacetone in cyclohexane (0.1 M, 7 mL). The solution was left on top of the gel during 24 hrs then removed with a Pasteur pipet and this step was repeated 5 times.

Electron microscopy.

Small pieces of the reacted gels (typically 2 x 2 x 2 mm) were placed between two copper holders and rapidly frozen in liquid N_2. The sample were kept frozen and transferred in a home-made freeze-fracture apparatus (developed by J.-C. Homo). Pt was evaporated (2 nm) under a 45° angle, then a reinforcing carbon layer (20 nm) under a 90° angle respectively to the surface. The sample was warmed up to room temperature and the replica were carefully washed with chloroform and picked up onto 400 mesh grids. The grids were observed with a Philipps CM12 operating at 120 kV and images taken with a SIS Megaview III camera.

Samples of the gel were taken at different heights to verify that the diffusion of the catalyst did not induced heterogeneities in the gel. The micrographs exhibited similar aspect independently of the location of the sample in the gel. The diameters of the tubes were measured with the Analysis software (SIS-Olympus Münster Germany). For each experiment, the diameters of more than 200 tubes were measured and averaged. The uncertainty of the measurement was taken equal to the deviation.

Analysis of the reacted gels.

A gel was prepared, let react and rinse as described above. It was dissolved in CH_2Cl_2 (5 mL) and the solution was dried under vacuum. The crude was weighted and an aliquot of about 5 mg was dissolved in 3 mL THF and volumes of 200 µL of the solution were injected on a chromatography set-up composed of a pump (Shimadzu DGU-20A) operating at a flow of 1

ml/min, a column PL (granulometry 10 µ), a differential refractometer from Shimadzu (RID6A) and a UV detector from Shimadzu (5SPD 10 Avp). The pure starting materials and final product were injected separately in order to identify the elution times and to calculate the molar extinction (ε) and refraction increment (dn/dc). The concentrations in the crude were measured from the areas from both the RI and UV traces. The results were in good agreement for both detections. The ratio of final compound/reactants was also in good agreement with NMR.

Effect of rinsing the gel without acetylacetone.

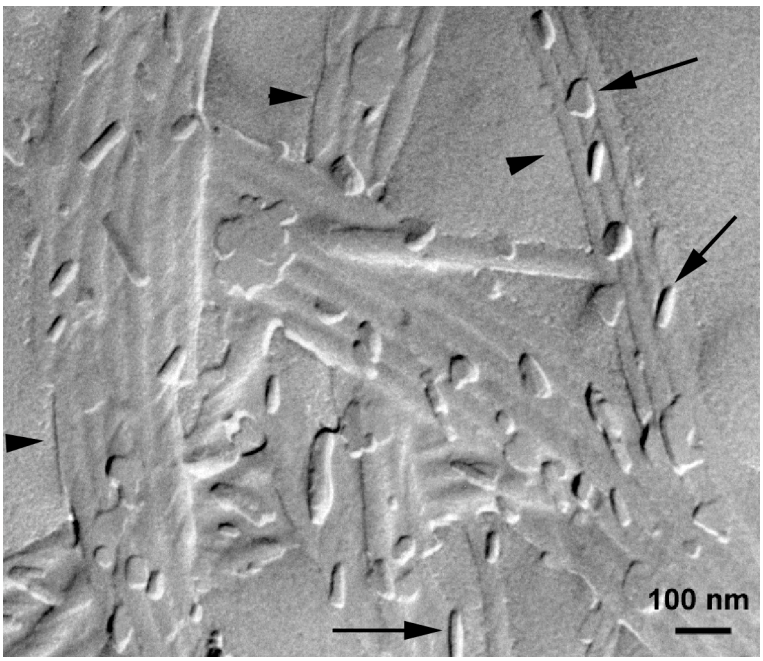


Figure S1. Electron micrograph of a gel of **2** reacted with **12**. In the rinsing step, acetylacetone was omitted. Arrowhead: nanotubes; arrows: nanostructures that are no longer present when acac is used.

S3. Preparation of the pure final compounds

3,5-Bis-(5-hexylcarbamoyl-pentyloxy)-benzoic acid 9-(3-decyl-3*H*-[1,2,3]triazol-4-yl)-nonyl ester (**4**) :

A solution of **2** (200 mg, 0.29 mmol, 1 equiv.), 1-azidodecane **14** (250 mg, 1.36 mmol, 4.8 equiv.), $Cu(PPh_3)_3Br$ (133 mg, 0.14 mmol, 0.5 equiv.) and DIPEA (100 µl, 0.57 mmol, 2

equiv.) in CH_2Cl_2 (8 ml) was stirred 72 hrs at 25°C.The solvent was evaporated under vacuum and the residue was chromatographed (SiO_2, MeOH/ CH_2Cl_2 : 0/100 to 3/97 gradient elution) to afford pure **5** as a white solid (180 mg, 70 %). Recrystallization of the compound from cold acetonitrile afforded **5** as white crystals (needles). M. p. 84.6 °C; ^{1}H NMR (400 MHz, $CDCl_3$) : δ [ppm] 7.29 (s, broad, $H_{triazole}$), 7.14 (d, 2 H, *J* = 2.2 Hz, C2-**H**, C6-**H**), 6.60 (t, 1 H, *J* = 2.2 Hz, C4-**H**), 5.56 (s broad, 2 H, N**H**), 4.28 (m, 4 H, C**H**$_2$N$_{triazole}$ and COOC**H**$_2$), 3.96 (t, 4 H, *J* = 6.3 Hz, ArOC**H**$_2$), 3.24 (q, 4 H, *J* = 7.16 Hz, C**H**$_2$NHCO), 2.71 (s, large, 2 H, C**H**$_2$C$_{triazole}$), 2.18 (t, 4H, C**H** $_2$CONH), 1.88 (s, broad, 2H, C**H** $_2$CH$_2$N$_{triazole}$), 1.79-1.71 (12 H, COOCH$_2$C**H**$_2$, ArOCH$_2$C**H**$_2$, COO(CH$_2$)$_7$C**H**$_2$, ArO(CH$_2$)$_3$C**H**$_2$), 1.49-1.25 (44 H, C**H**$_2$), 0.87 (6 H, C**H**$_3$). ^{13}C NMR (100 MHz, $CDCl_3$) : δ [ppm] 173.06 (**C**ONH), 166.84 (COO), 160.35 (C3, C5), 138.07 (CH$_2$**C**$_{triazole}$), 132.61 (C1 and **C**H$_{triazole}$), 108.03 (C2, C6), 106.57 (C4), 68.31 (ArO**C**H$_2$), 65.61 (ArCOO**C**H$_2$), 39.87 (CH$_2$N$_{triazole}$), 37.05 (CONH**C**H$_2$), 32.18 (NHCO**C**H$_2$), 31.80 (**C**H$_2$(CH$_2$)$_7$N$_{triazole}$, 30.6 (COO(CH$_2$)$_7$**C**H$_2$), 30.0 (CONH(CH$_2$)$_3$**C**H$_2$), 29.8 (CONHCH$_2$**C**H$_2$), 29.7 (ArCOO(CH$_2$)$_4$**C**H$_2$**C**H$_2$), 29.6 (N$_{triazole}$(CH$_2$)$_3$**C**H$_2$ and N$_{triazole}$(CH$_2$)$_6$**C**H$_2$ and ArCOO(CH$_2$)$_5$**C**H$_2$), 29.3 (ArCOOCH$_2$**C**H$_2$), 26.9 (N$_{triazole}$(CH$_2$)$_2$**C**H$_2$), 26.9 (N$_{triazole}$CH$_2$**C**H$_2$), 26.3 (CONH(**C**H $_2$)$_2$CH$_2$), 26.1 (ArCOO(CH$_2$)$_2$**C**H$_2$), 25.9 (NHCOCH$_2$**C**H$_2$), 25.8 (NHCO CH$_2$CH$_2$**C**H$_2$), 223.0 (ArCOO(CH$_2$)$_8$**C**H$_2$), 22.9 (CONH(CH$_2$)$_4$**C**H$_2$ and N$_{triazole}$(CH$_2$)$_8$**C**H$_2$), 14.4 (N$_{triazole}$(CH$_2$)$_8$**C**H$_3$), 14.3 (CONH(CH$_2$)$_5$**C**H$_3$); IR (ATR-diamond) ν_{max} : 3303 (br, ν NH), 3075 (ν CH$_{ar}$), 2955 (ν_{as}, CH$_3$), 2924 (ν_{as}, CH$_2$), 2870 (ν_s, CH$_3$), 2854 (ν_s, CH$_2$), 2361, 2341, 1726 (ArCOO, ν CO), 1716, 1638 (amide I), 1612 (ν -N=N-), 1599 (ν C=C), 1543 (δ NH, amide II band), 1465 (CH$_2$ scissor), 1383, 1345, 1300 (*ν* C-N, amide III and triazole), 1238, 1174 (ν N-N), 1158, 1110, 1068, 1052, 960, 875, 834, 763, 722, 676 cm^{-1}; (HRMS (ESI+) m/z 888.7106 (MLi$^+$, calcd for $C_{52}H_{91}N_5O_6$: 888.7129). Anal. Found C, 70.39; H, 10.38 ; N, 7.66. Calcd for $C_{52}H_{91}N_5O_6$: C, 70.79; H 10.40; N, 7.94.

3,5-Bis-(5-hexylcarbamoyl-pentyloxy)-benzoic acid 9-[3-(10-hydroxy-decyl)-3*H*-[1,2,3]triazol-4-yl]-nonyl ester (**5**) :

A solution of **2** (0.20 g, 0.29 mmol), **12** (0.09 g, 0.45 mmol, 1.58 equiv.), Cu(PPh$_3$)$_3$Br (0,133 g, 1.43 mmol, 0.5 equiv.) and DIPEA (0.1 ml, 0.574 mmol, 2 equiv.) in CH_2Cl_2 (8 ml) was stirred 48 hrs at 25°C. The solvent was evaporated under vacuum and the residue was chromatographed (SiO_2, isopropanol/ CH_2Cl_2 4/96 then MeOH/ CH_2Cl_2 7/93) to afford **4** as a off-white waxy solid (0. 22 g, 85 %). Recrystallization of the compound from cold

acetonitrile afforded pure **4** s a white solid. M. p. 60.5 °C; ^{1}H NMR (400 MHz, $CDCl_3$) : δ [ppm] 7.30 (s, broad, $H_{triazole}$), 7.13 (d, 2 H, *J* = 2.2 Hz, C2-**H**, C6-**H**), 6.60 (t, 1 H, *J* = 2.2 Hz, C4-**H**), 5.56 (s broad, 2 H, N**H**), 4.29 (m, 4 H, C**H**$_2$N$_{triazole}$ and COOC**H**$_2$), 3.96 (t, 4 H, *J* = 6.3 Hz, ArOC**H**$_2$), 3.63 (2 H, *J* = 6.7 Hz, C**H**$_2$OH), 3.23 (q, 4 H, *J* = 7.16 Hz, C**H**$_2$NHCO), 2,70 (s, large, 2 H, C**H**$_2$C$_{triazole}$), 2.19 (t, 4H, C**H**$_2$CONH), 1.87-1.68 (m, 20 H, COOCH$_2$CH$_2$, COO(CH$_2$)$_7$C**H**$_2$, ArOCH$_2$C**H**$_2$, C**H** $_2$CH$_2$N$_{triazole}$, ArO(CH$_2$)$_3$C**H**$_2$, CONHCH$_2$C**H**$_2$, C**H**$_2$CH$_2$OH), 1.55-1.28 (38 H, CH$_2$), 0.87 (t, 6 H, *J* = 6.84 Hz, C**H**$_3$). ^{13}C NMR (100 MHz, $CDCl_3$) : δ [ppm] 173.1 (**C**ONH), 166.9 (COO), 160.4 (C3, C5), 147.2 (CH$_2$**C**$_{triazole}$), 132.6 (C1 or **C**H$_{triazole}$), 108.1 (C2, C6), 106.6 (C4), 68.3 (ArO**C**H$_2$), 65.6 (COO**C**H$_2$), 63.3 (**C**H$_2$OH), 39.9 (CH$_2$N$_{triazole}$), 37.0 (CONH**C**H$_2$), 33.1 (NHCO**C**H$_2$), 31.8 (**C**H$_2$CH$_2$OH), 30.6 (COO(CH$_2$)$_7$**C**H$_2$), 30.0 (CH$_3$CH$_2$**C**H$_2$), 29.7 (CONHCH$_2$**C**H$_2$), 29.62, 29.56, 29.53, 29.24, 29.22, 28.98, 26.93, 26.75, 26.33, 26.10, 26.02, 25.82, 25.80, 22.89 (CH$_3$**C**H$_2$), 14.3 (CH$_3$). ATR-IR (solid) ν_{max} : 3305 (broad, ν NH and ν OH), 3075 (ν CH$_{Ar}$), 2952 (ν_{as}, CH$_3$), 2925 (ν_{as}, CH$_2$), 2870 (ν_s, CH$_3$), 2854 (ν_s, CH$_2$), 2361, 2341, 1726 (ArCOO, ν CO), 1716, 1640 (amide I), 1613 (ν -N=N-), 1599 (ν -C=C-), 1545 (amide II), 1466 (δ CH$_2$), 1384, 1347, 1300, 1228, 1175 (ν N-N), 1160, 1110, 1069, 1055, 961, 876, 835, 725, 676, 618 cm^{-1}; HRMS (ESI$^+$) m/z 904.6994 (MLi$^+$, calcd. for $C_{52}H_{91}N_5O_7$: 904.7079). Anal. Found C, 69.22 ; H 10.33 ; N, 7.68. Calcd for $C_{52}H_{91}N_5O_7$: C, 69.53 ; H 10.21 ; N, 7.80.

3,5-Bis-(5-hexylcarbamoyl-pentyloxy)-benzoic acid 10-[4-(9-hydroxy-nonyl)-[1,2,3]triazol-1-yl]-decyl ester (**6**) :

3 (100 mg, 0.14 mmol), undecyn-1-ol (460 mg, 0.27 mmol, 2 equiv.), and sodium L-ascorbate (50 mg, 0.25mmol, 1.8 equiv.) were dissolved in a water/CH_3CN/THF mixture (1/1/1, 6 mL). Copper (II) sulfate pentahydrate (17 mg, 0.068 mmol, 0.5 equiv.) was added and the resulting solution was stirred at 25 °C under argon during 24 hrs. The mixture was concentrated under vacuum, mixed with H_2O (5 mL) and extracted with CH_2Cl_2 (3 x 10 mL). The organic phases were dried ($MgSO_4$), concentrated and the residue was chromatographed (SiO_2, MeOH/CH_2Cl_2: 1/99 to 5/99 elution gradient) to afford pure **6** (0.11 g, 89.4 %). M.p. 60.8 °C; ^{1}H NMR (400 MHz, $CDCl_3$) : δ [ppm] 7.24 (s, $H_{triazole}$), 7.13 (d, 2 H, *J* = 2.04 Hz, C2-**H**, C6-**H**), 6.60 (t, 1 H, *J* = 2.2 Hz, C4-**H**), 5.54 (s broad, 2 H, N**H**), 4.27 (m, 4 H, C**H**$_2$N$_{triazole}$ and COOC**H**$_2$), 3.96 (t, 4 H, *J* = 6.32 Hz, ArOC**H**$_2$), 3.63 (2 H, *J* = 6.7 Hz, C**H**$_2$OH), 3.23 (q, 4 H, *J* = 6.68 Hz, C**H**$_2$NHCO), 2.69 (t, 2 H, *J* = 7.48 Hz C**H**$_2$C$_{triazole}$), 2.19 (t, 4H, *J* = 7.36 Hz C**H**$_2$CONH), 1.87 (t, 2H, *J* = 6.84 Hz, COOCH$_2$C**H**$_2$), 1.77 (q, 4H, *J* = 7.0 Hz, ArOCH$_2$C**H**$_2$),

1.70 (q, 4H, J = 7.52 Hz, C**H**$_2$CH$_2$N$_{triazole}$), 1.65 (2H, C**H**$_2$CH$_2$C$_{triazole}$), 1.53 (2H, C**H**$_2$CH$_2$OH), 1.49-1.28 (46 H, CH$_2$), 0.87 (t, 6 H, J = 6.68 Hz, C**H**$_3$). ^{13}C NMR (100 MHz, CDCl$_3$) : δ [ppm] 173.1 (**C**ONH), 166.9 (COO), 160.4 (C3, C5), 148.7 (CH$_2$**C**$_{triazole}$), 132.6 (C1), 120.7 (CH$_{triazole}$), 108.1 (C2, C6), 106.6 (C4), 68.3 (ArO**C**H$_2$), 65.6 (COO**C**H$_2$), 63.3 (**C**H$_2$OH), 50.5 (CH$_2$N$_{triazole}$), 39.9 (CONH**C**H$_2$), 37.1 (NHCO**C**H$_2$), 33.1 (**C**H$_2$CH$_2$OH), 31.8(CH$_3$CH$_2$**C**H$_2$), 30.7 (C$_{triazole}$CH$_2$**C**H$_2$), 30.0 (CONHCH$_2$**C**H$_2$, 29.82, 29.79, 29.69, 29.66, 29.6, 29.53, 29.49, 28.29, 29.25, 29.0, 26.9, 26.8, 26.3, 26.10, 26.05, 26.0, 25.8, 22.9 (CH$_3$**C**H$_2$), 14.34 (CH$_3$). ATR-IR (solid) ν$_{max}$: 3309 (broad, ν NH and ν OH), 3073 (ν CH$_{ar}$), 2952 (ν$_{as}$, CH$_3$), 2927 (ν$_{as}$, CH$_2$), 2870 (ν$_s$, CH$_3$), 2856 (ν$_s$, CH$_2$), 1718 (ν CO ester), 1640 (amide I), 1599 (ν C=C) , 1543 (amide II), 1466 (δ CH$_2$), 1449, 1421, 1385, 1345, 1301 (amide III), 1241, 1171 (ν N-N), 1161, 1110, 1068, 1054, 957, 912, 839, 795, 766, 726, 681 cm^{-1}. HRMS (ESI+) m/z 920.6676 (MNa$^+$, calcd for C$_{52}$H$_{91}$N$_5$O$_7$: 920.6816). Anal. Found : C, 69.51; H, 10.42; N, 7.70. Calcd for C$_{52}$H$_{91}$N$_5$O$_7$: C, 69.53; H, 10.21; N, 7.80.