

Supporting Information

Transfer and amplification of chirality in Phe-based C₃-symmetric tricarboxamides

Julia Buendía,^a Fátima García,^a Belén Yélamos,^b Luis Sánchez*^a

^a*Departamento de Química Orgánica, Facultad de Ciencias Químicas, Universidad Complutense de Madrid, E-28040 Madrid (Spain).*

^b*Departamento de Bioquímica y Biología Molecular I, Facultad de Ciencias Químicas, Universidad Complutense de Madrid, 28040-Madrid (Spain)*

Contents:

<i>1.- Supplementary Figures and Tables</i>	<i>S-2</i>
<i>FTIR spectra</i>	<i>S-2</i>
<i>Concentration and temperature-dependent ¹H NMR spectra</i>	<i>S-3</i>
<i>SEM images</i>	<i>S-4</i>
<i>CD spectra of 1 in different solvents</i>	<i>S-5</i>
<i>Photographs of the organogels formed by 1 and 2</i>	<i>S-5</i>
<i>VT-CD spectra of 1</i>	<i>S-6</i>
<i>Amplification of chirality experiments</i>	<i>S-6</i>
<i>2. Experimental section</i>	<i>S-7</i>
<i>3. Synthetic details and characterization</i>	<i>S-8</i>
<i>4. Collection of spectra</i>	<i>S-16</i>

1. Supplementary Figures and Tables

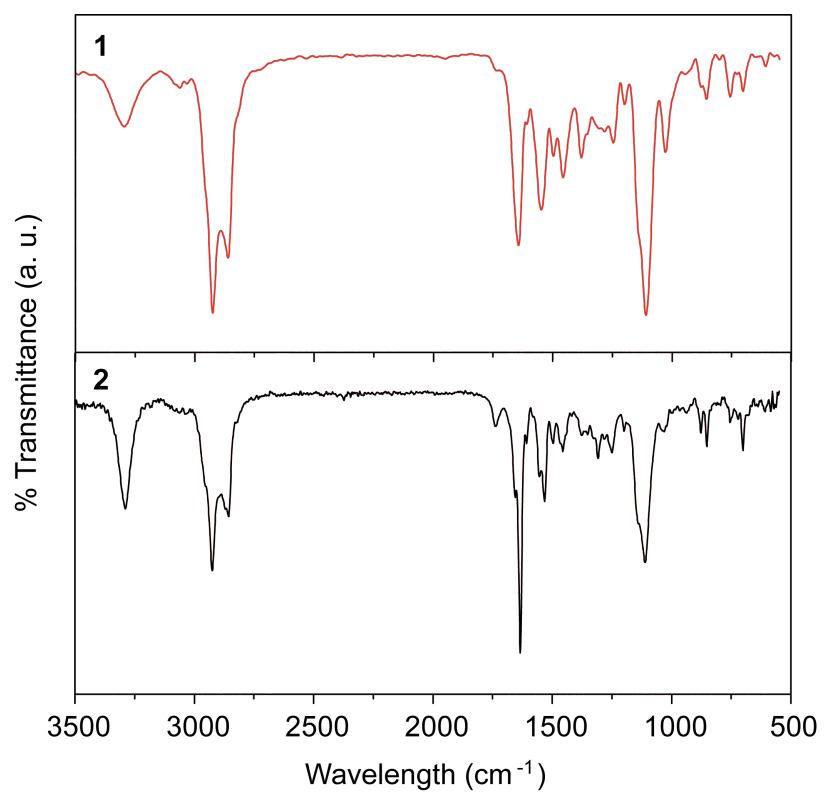


Figure S1. Partial FTIR spectra of **1** and **2**.

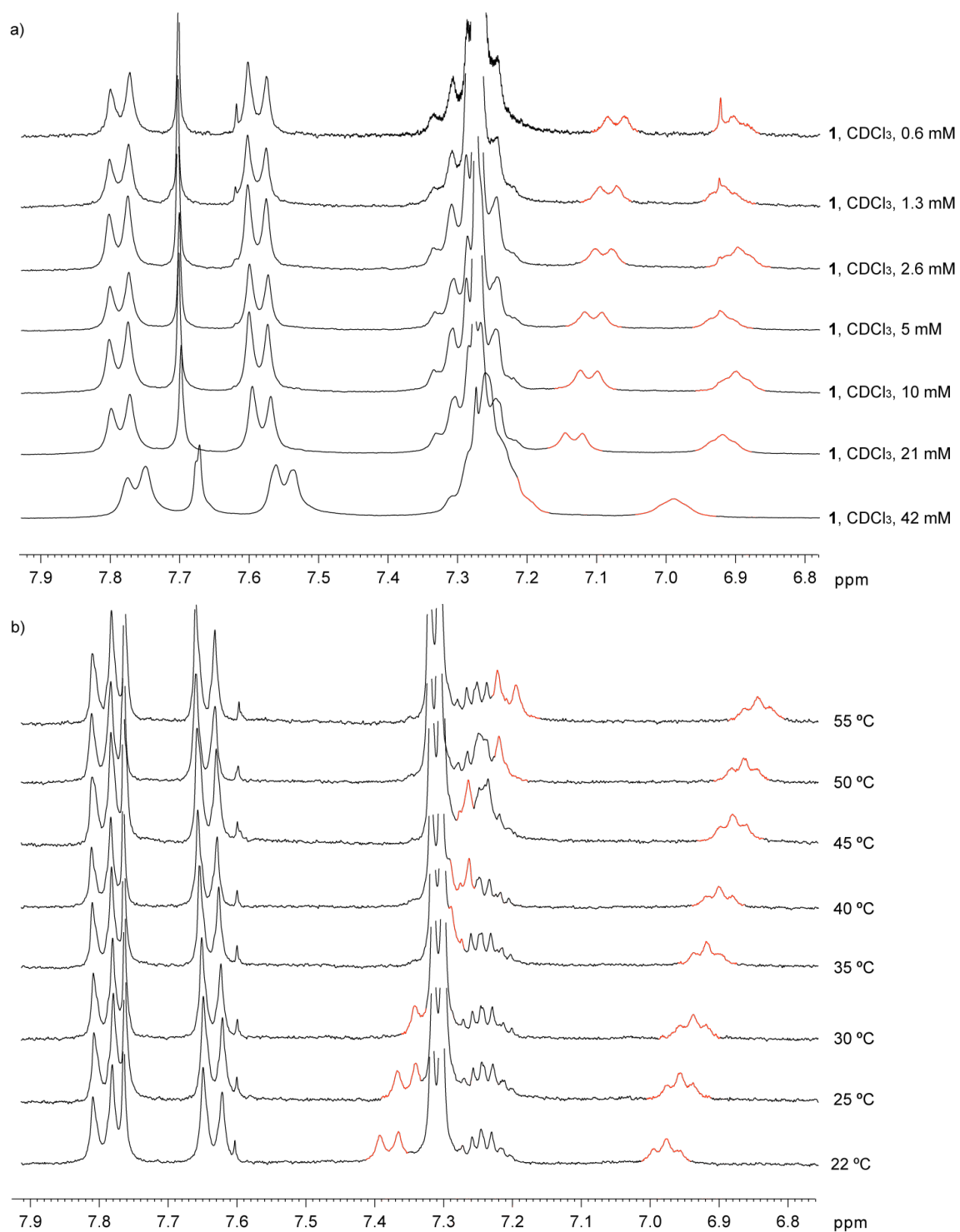


Figure S2. Partial ^1H NMR spectra of **1** in (a) CDCl_3 at different concentrations (300 MHz, 298 K), and in (b) CD_3CN at 1 mM and different temperatures. The resonances in red correspond to the inner and outer amides.

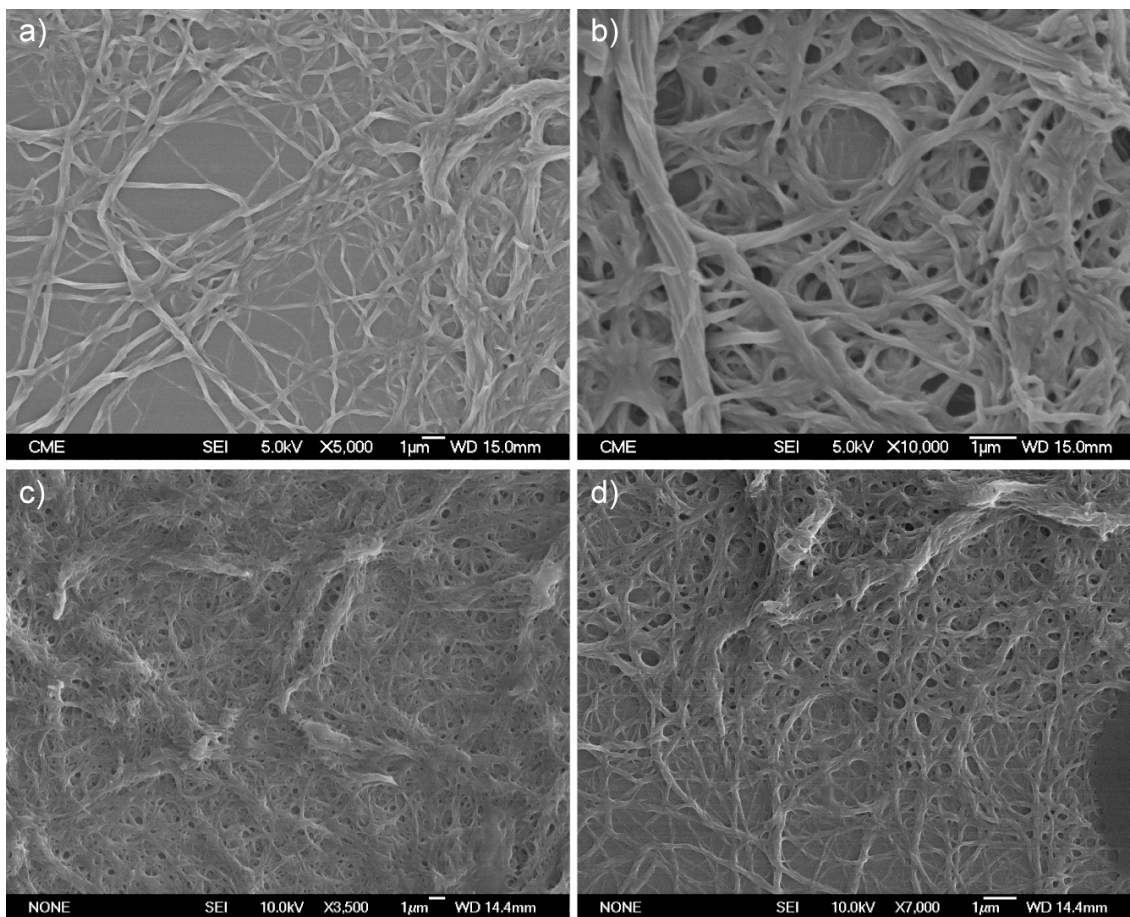


Figure S3. SEM images of the fibrillar structures formed by the self-assembly of **1** (a, b) and **2** (c, d) on to a glass substrate.

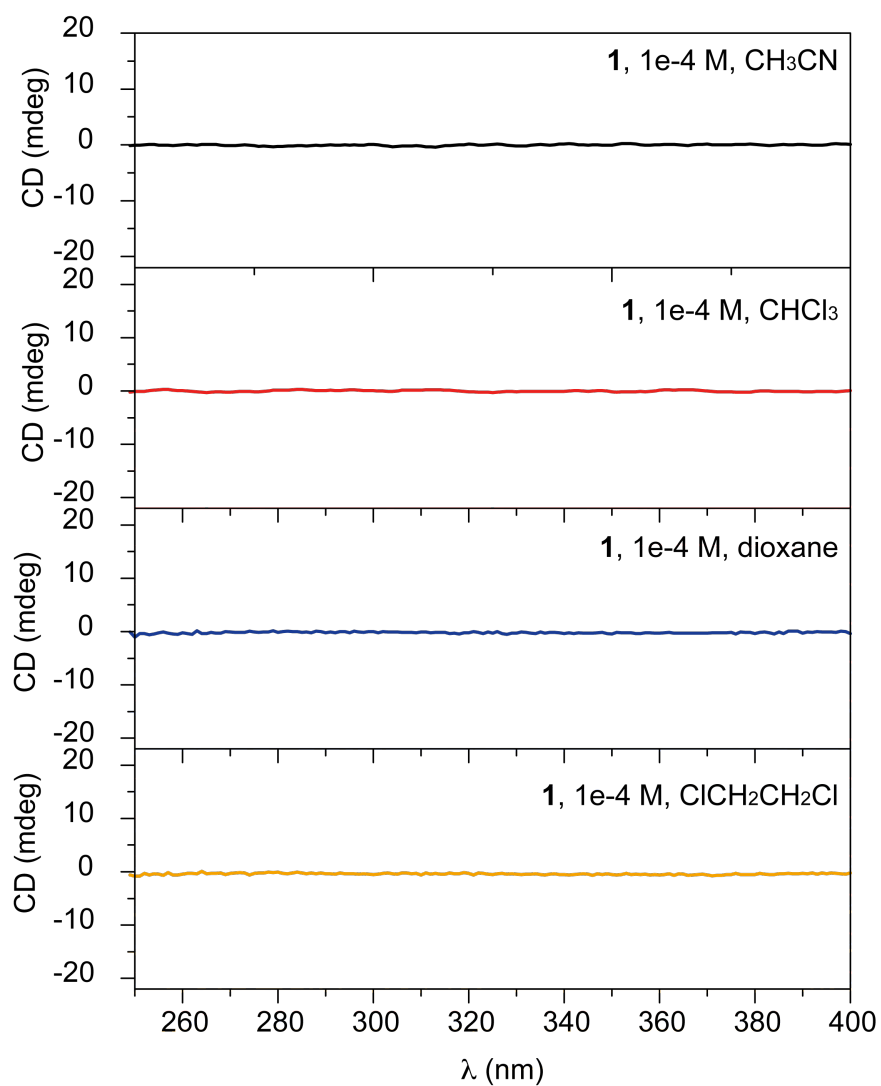


Figure S4. CD spectra of **1** in solvents of different polarity (1×10^{-4} M, 298 K).

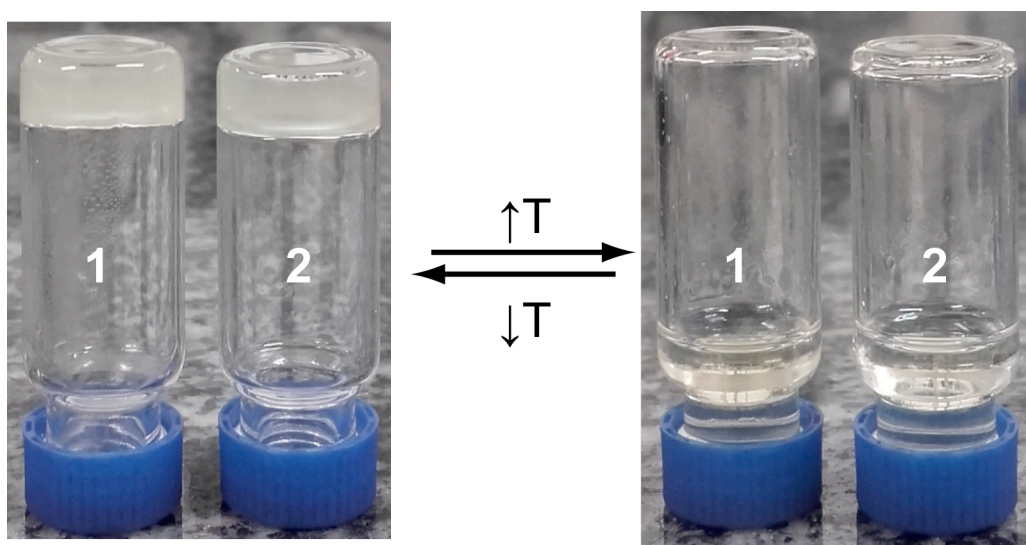


Figure S5. Photograph of the organogels formed from **1** and **2** in CCl_4 at 7 mM at room temperature (left) and at 70 °C (right).

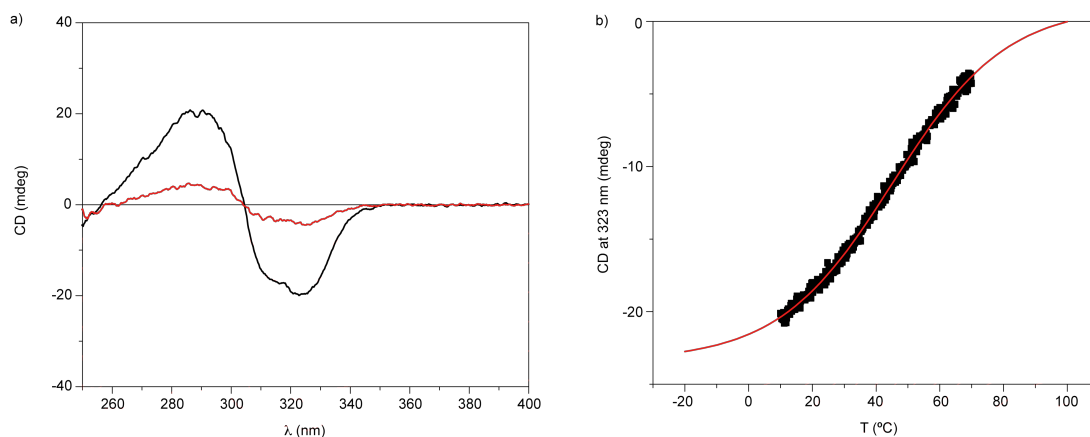


Figure S6. (a) CD spectra of **1** in CCl_4 at 25 °C (black line) and at 70 °C (red line). (b) Cooling curve of **1** in CCl_4 at 2×10^{-4} M. The red line depicts the fitting of the variation of the dichroic signal at 323 nm to a sigmoidal curve ($R^2 = 0.9969$).

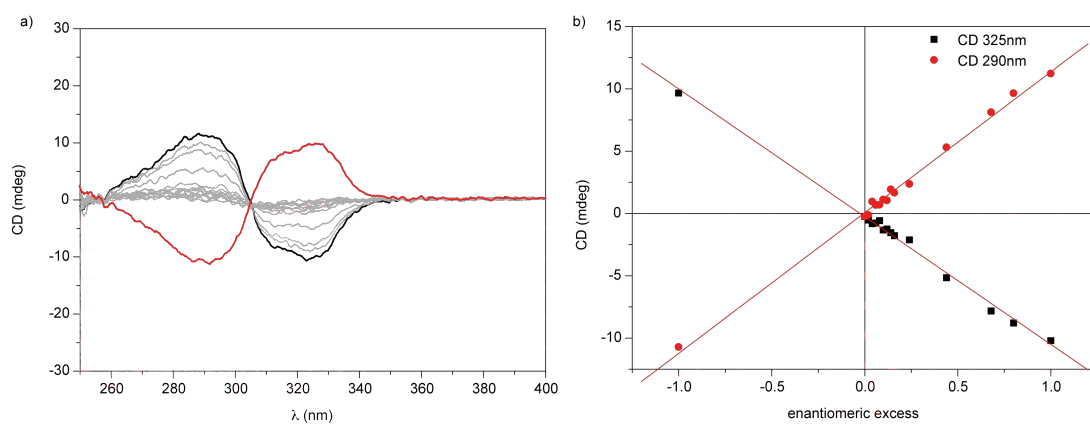
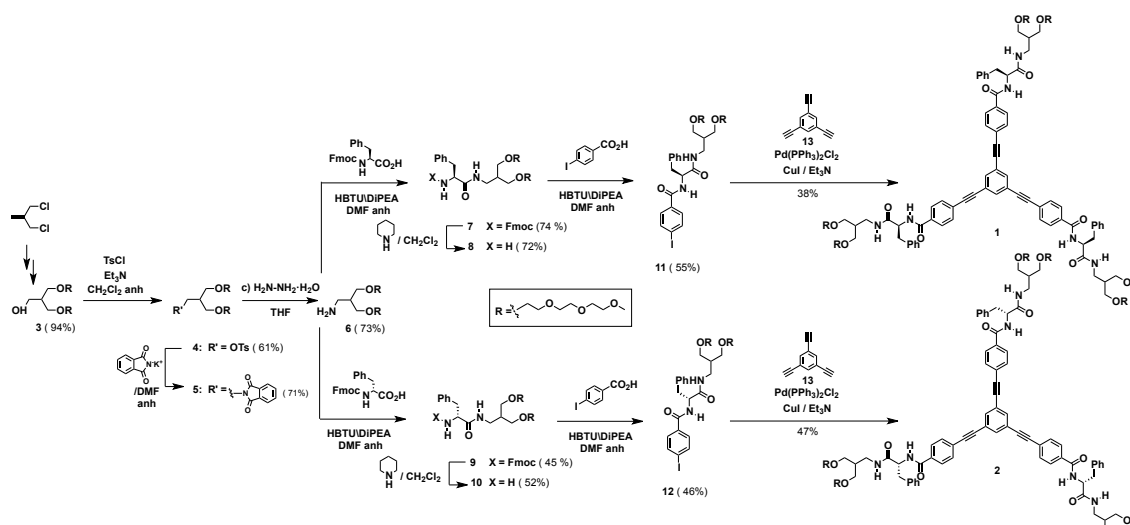


Figure S7. (a) CD spectra of mixtures of **1** and **2** (CCl_4 , 293 K, 1×10^{-4} M). (b) Changes in the CD intensity against the *e.e.* observed upon adding increasing aliquots of **1** to a solution of **2** (CCl_4 , 293 K, 1×10^{-4} M). The red lines represent the fitting to straight line.

2. Experimental section

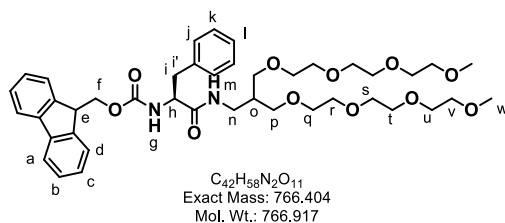
General. All solvents were dried according to standard procedures. Reagents were used as purchased. All air-sensitive reactions were carried out under argon atmosphere. Flash chromatography was performed using silica gel (Merck, Kieselgel 60, 230-240 mesh or Scharlau 60, 230-240 mesh). Analytical thin layer chromatography (TLC) was performed using aluminium-coated Merck Kieselgel 60 F254 plates. NMR spectra were recorded on a Bruker Avance 300 (^1H : 300 MHz; ^{13}C : 75 MHz) spectrometer at 298 K using partially deuterated solvents as internal standards. Coupling constants (J) are denoted in Hz and chemical shifts (δ) in ppm. Multiplicities are denoted as follows: s = singlet, d = doublet, t = triplet, m = multiplet, br = broad. FT-IR spectra were recorded on a Bruker Tensor 27 (ATR device) spectrometer. Circular dichroism (CD) measurements were performed on a Jasco-810 dichrograph equipped with a Peltier thermoelectric temperature controller. The spectra were recorded in the continuous mode between 400 and 200 nm, with a wavelength increment of 1 nm, a response time of 4 s, and a bandwidth of 1 nm. A 1 mm path length quartz cuvette (Hellma) was used. SEM images were obtained from on a JEOL JSM 6335F microscope working at 10kV. Matrix Assisted Laser Desorption Ionization (coupled to a Time-Of-Flight analyzer) experiments (MALDI-TOF) were recorded on a Bruker REFLEX spectrometer.

3. Synthetic details and characterization



Compounds **3**, **4**, **5** and **6** were prepared according to previously reported synthetic procedures (see: Jayaraman, M.; Fréchet, J. M. J. *J. Am. Chem. Soc.* **1998**, *120*, 12996–12997; Park, I.S.; Yoon, Y.R.; Jung, M; Kim, K.; Park, S.; Shin, S.; Lim, Y.; Lee, M. *Chem. Asian. J.* **2011**, *6*, 452-458; Buendía, J.; Sánchez, L.; *Org. Lett.* **2013**, *22*, 5746-5749) and showed identical spectroscopic properties to those reported therein.

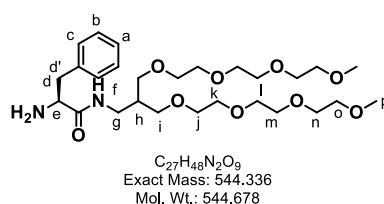
(9H-fluoren-9-yl)methyl (S)-1-(3-(2-(2-(2-methoxyethoxy)ethoxy)ethoxy)-2-((2-(2-(2-methoxyethoxy)ethoxy)ethoxy)ethoxy)methyl)propylcarbamoyl)-2-phenylethylcarbamate (7)



N,N,N',N'-Tetramethyl-*O*-(1*H*-benzotriazol-1-yl)uronium hexafluorophosphate (HBTU) (1.30 g, 3.49 mmol) was dissolved in dry DMF (19 mL) under argon atmosphere and *N*-(9-Fluorenylmethoxycarbonyl)-*L*-phenylalanine (1.35 g, 3.49 mmol) and *N,N*-Diisopropylethylamine (DIPEA) (1.3 mL, 7.76 mmol) were added. The reaction mixture was stirred for 20 minutes. Then, amine **6** (0.77 g, 1.94 mmol) was added. The reaction mixture was stirred at room temperature overnight. The residue was washed with HCl (1M)/water/ice, NaOH (3M) and NaHCO₃, extracted with diethyl ether and dried over MgSO₄. After evaporation of the solvent under reduced pressure, the residue was purified by column chromatography (silica gel, chloroform:methanol 100:1) affording compound **7** as a colorless solid (1.10 g, 74%). ¹H NMR (CDCl₃, 300 MHz) δ 7.70 (2H,

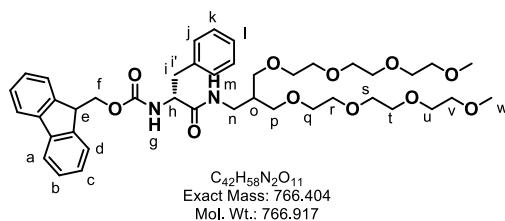
H_a, d, *J* = 7.4); 7.50 (2H, H_b, t, *J* = 7.0); 7.34 (2H, H_c, t, *J* = 7.0); 7.28–7.11 (7H, H_{d+j+k+l}, m); 6.93 (1H, H_m, t, *J* = 5.3); 5.81 (1H, H_g, d, *J* = 8.2); 4.44–4.19 (3H, H_{f+h}, m); 4.12 (1H, H_e, t, *J* = 6.8); 3.60–3.16 (36H, H_{n+p+q+r+s+t+u+v+w}, m); 3.15–2.92 (2H, H_{i+i'}, m); 2.00 (1H, H_o, m). ¹³C NMR (CDCl₃, 75 MHz) δ 170.58, 155.62, 143.68, 141.11, 136.84, 129.21, 128.36, 127.54, 126.91, 126.63, 124.89, 119.79, 71.72, 70.85, 70.41, 70.24, 70.22, 70.16, 70.08, 66.52, 58.80, 56.27, 46.98, 40.32, 38.65, 38.48. FTIR (neat) 701, 741, 760, 942, 1041, 1092, 1244, 1350, 1450, 1530, 1658, 1717, 2871, 3280. HRMS (MALDI-TOF): calc. for C₄₂H₅₈N₂NaO₁₁ [M+Na]⁺ 789.3938; found 789.3972.

(S)-N-(3-(2-(2-(2-methoxyethoxy)ethoxy)ethoxy)-2-((2-(2-(2-methoxyethoxy)ethoxy)ethoxy)methyl)propyl)-2-amino-3-phenylpropanamide (8)



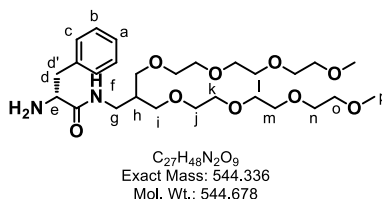
Compound **7** (1.1 g, 1.43 mmol) was dissolved in methylene chloride (35 mL) and piperidine (9 mL) was added. The reaction mixture was stirred for 24 hours. After evaporation of the solvent under reduced pressure, the residue was purified by column chromatography (silica gel, chloroform:methanol 100:5) affording compound **8** as a yellow oil (0.56 g, 72%). ¹H NMR (CDCl₃, 300 MHz) δ 7.64 (1H, H_f, t, *J* = 5.5); 7.30–7.14 (5H, H_{a+b+c}, m); 3.61–3.26 (37H, H_{e+g+i+j+k+l+m+n+o+p}, m); 3.18 (1H, H_{d o d'}, dd, *J* = 13.6, 4.3); 2.64 (1H, H_{d o d'}, dd, *J* = 13.6, 9.1); 2.06 (1H, H_n, m). ¹³C NMR (CDCl₃, 75 MHz) δ 174.36, 138.22, 129.33, 128.59, 126.64, 71.90, 71.12, 70.59, 70.52, 70.48, 70.43, 59.00, 56.74, 41.23, 39.81, 39.13. FTIR (neat) 702, 746, 849, 1033, 1096, 1247, 1292, 1353, 1451, 1525, 1658, 2868, 3357. HRMS (MALDI-TOF): calc. for C₂₇H₄₉N₂O₉ [M+H]⁺ 545.3438; found 545.3423.

(9H-fluoren-9-yl)methyl (R)-1-(3-(2-(2-(2-methoxyethoxy)ethoxy)ethoxy)ethoxy)ethyl-2-((2-(2-(2-methoxyethoxy)ethoxy)ethoxy)methyl)propylcarbamoyl)-2-phenylethylcarbamate (9)



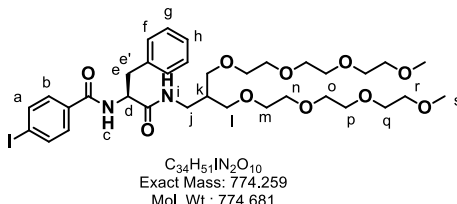
N,N,N',N'-Tetramethyl-*O*-(1*H*-benzotriazol-1-yl)uronium hexafluorophosphate (HBTU) (1.96 g, 5.17 mmol) was dissolved in dry DMF (10 mL) under argon atmosphere and *N*-(9-Fluorenylmethoxycarbonyl)-*D*-phenylalanine (2.0 g, 5.17 mmol) and *N,N*-Diisopropylethylamine (DIPEA) (2.0 mL, 11.48 mmol) were added. The reaction mixture was stirred for 20 minutes. Then, amine **6** (1.14 g, 2.87 mmol) was added. The reaction mixture was stirred at room temperature overnight. The residue was washed with HCl (1M)/water/ice, NaOH (3M) and NaHCO₃, extracted with diethyl ether and dried over MgSO₄. After evaporation of the solvent under reduced pressure, the residue was purified by column chromatography (silica gel, chloroform:methanol 100:1) affording compound **9** as a colorless solid (0.99 g, 45%). ¹H NMR (CDCl₃, 300 MHz) δ 7.75 (2H, H_a, d, *J* = 7.4); 7.54 (2H, H_b, t, *J* = 7.0); 7.39 (2H, H_c, t, *J* = 7.0); 7.33–7.16 (7H, H_{d+j+k+l}, m); 6.95 (1H, H_m, t, *J* = 5.3); 5.72 (1H, H_g, d, *J* = 8.2); 4.46–4.23 (3H, H_{f+h}, m); 4.17 (1H, H_e, t, *J* = 6.8); 3.67–3.21 (36H, H_{n+p+q+r+s+t+u+v+w}, m); 3.16–2.97 (2H, H_{i+i'}, m); 2.04 (1H, H_o, m). ¹³C NMR (CDCl₃, 75 MHz) δ 170.86, 156.86, 143.95, 141.40, 137.01, 129.49, 128.65, 127.81, 127.18, 126.93, 125.18, 120.07, 72.01, 71.18, 70.69, 70.54, 70.52, 70.47, 70.44, 66.89, 59.10, 56.48, 47.27, 40.60, 39.04, 38.75. FTIR (neat) 742, 760, 850, 942, 1041, 1093, 1199, 1244, 1350, 1450, 1532, 1659, 1717, 2872, 3282. HRMS (MALDI-TOF): calc. for C₄₂H₅₈N₂NaO₁₁ [M+Na]⁺ 789.3938; found 789.3967.

(R)-N-(3-(2-(2-(2-methoxyethoxy)ethoxy)ethoxy)ethoxy)-2-((2-(2-(2-methoxyethoxy)ethoxy)ethoxy)methyl)propyl)-2-amino-3-phenylpropanamide (10)



Compound **9** (0.99 g, 1.29 mmol) was dissolved in methylene chloride (32 mL) and piperidine (8 mL) was added. The reaction mixture was stirred for 24 hours. After evaporation of the solvent under reduced pressure, the residue was purified by column chromatography (silica gel, chloroform:methanol 100:5) affording compound **10** as a yellow oil (0.37 g, 52%). ¹H NMR (CDCl₃, 300 MHz) δ 7.67 (1H, H_f, t, J = 5.5); 7.30–7.16 (5H, H_{a+b+c}, m); 3.63–3.30 (37H, H_{e+g+i+j+k+l+m+n+o+p}, m); 3.20 (1H, H_d ó d', dd, J = 13.6, 4.3); 2.67 (1H, H_d ó d', dd, J = 13.6, 9.1); 2.08 (1H, H_n, m). ¹³C NMR (CDCl₃, 75 MHz) δ 174.27, 138.16, 129.35, 128.60, 126.67, 71.90, 71.11, 70.59, 70.52, 70.47, 70.43, 59.01, 56.72, 41.16, 39.82, 39.12. FTIR (neat) 703, 746, 850, 939, 1033, 1097, 1199, 1247, 1292, 1353, 1452, 1525, 1658, 2869, 3357. HRMS (MALDI-TOF): calc. for C₂₇H₄₉N₂O₉ [M+H]⁺ 545.3438; found 545.3425.

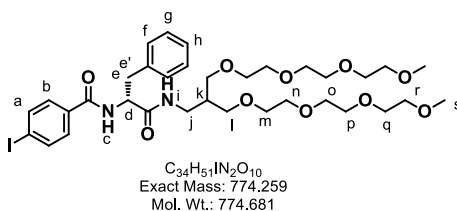
N-((S)-1-(3-(2-(2-(2-methoxyethoxy)ethoxy)ethoxy)ethoxy)-2-((2-(2-(2-methoxyethoxy)ethoxy)ethoxy)methyl)propyl)carbamoyl)-2-phenylethyl)-4-iodobenzamide (11)



N,N,N',N'-Tetramethyl-*O*-(1*H*-benzotriazol-1-yl)uronium hexafluorophosphate (HBTU) (0.70 g, 1.85 mmol) was dissolved in dry DMF (18 mL) under argon atmosphere and 4-iodobenzoic acid (0.46 g, 1.85 mmol) and *N,N*-Diisopropylethylamine (DIPEA) (0.7 mL, 4.12 mmol) were added. The reaction mixture was stirred for 20 minutes. Then, amine **8** (0.56 g, 1.03 mmol) was added. The reaction mixture was stirred at room temperature overnight. The residue was washed with HCl (1M)/water/ice, NaOH (3M) and NaHCO₃, extracted with diethyl ether and dried over MgSO₄. After evaporation of the solvent under reduced pressure, the residue was purified by column chromatography (silica gel, chloroform:methanol 50:1) affording compound **11** as a white solid (0.44 g, 55%). ¹H NMR (CDCl₃, 300 MHz) δ 7.72 (2H, H_a, d, J = 8.5); 7.46 (2H, H_b, d, J = 8.5); 7.29–7.13 (6H, H_{c+f+g+h}, m); 6.99 (1H, H_i, t, J = 5.4); 4.74 (1H, H_d, q,

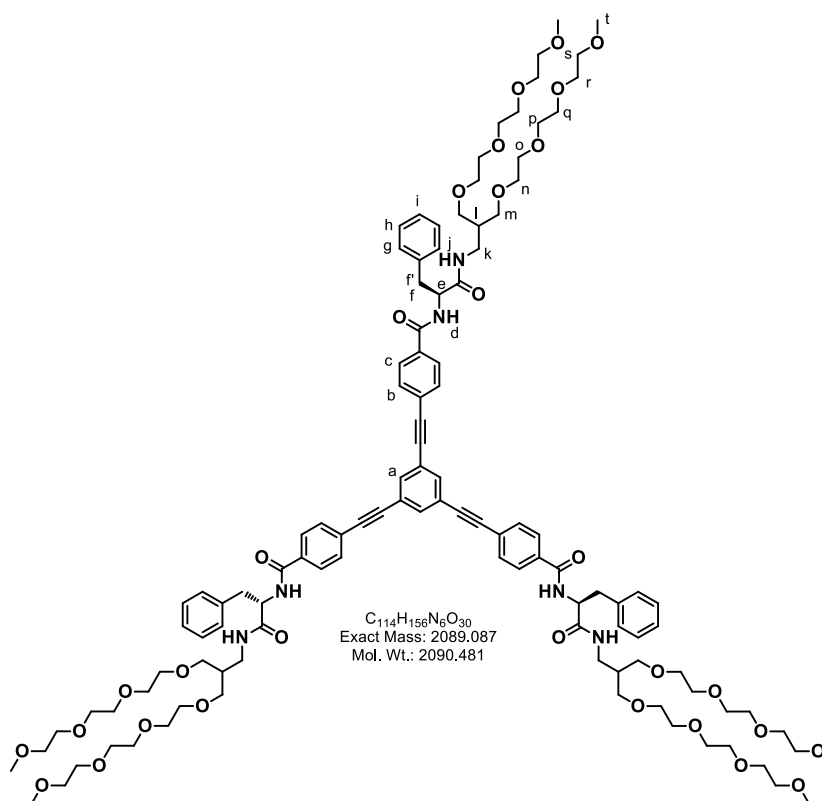
$J = 6.9$); 3.67–3.21 (36H, $H_{j+l+m+n+o+p+q+r+s}$, m); 3.18–3.11 (2H, $H_{e+e'}$, m); 2.03 (1H, H_k , m). ^{13}C NMR (CDCl_3 , 75 MHz) δ 170.66, 166.11, 137.71, 136.91, 133.56, 129.46, 128.86, 128.59, 126.94, 98.66, 71.95, 70.99, 70.95, 70.67, 70.51, 70.43, 70.40, 70.36, 59.05, 55.14, 40.37, 38.76. FTIR (neat) 700, 749, 846, 1005, 1104, 1245, 1353, 1451, 1476, 1534, 1586, 1635, 1722, 2868, 3295. HRMS (MALDI-TOF): calc. for $\text{C}_{34}\text{H}_{51}\text{IN}_2\text{NaO}_{10}$ $[\text{M}+\text{Na}]^+$ 797.2486; found 797.2487.

N-((R)-1-(3-(2-(2-(2-methoxyethoxy)ethoxy)ethoxy)-2-((2-(2-(2-methoxyethoxy)ethoxy)ethoxy)methyl)propylcarbamoyl)-2-phenylethyl)-4-iodobenzamide (12)



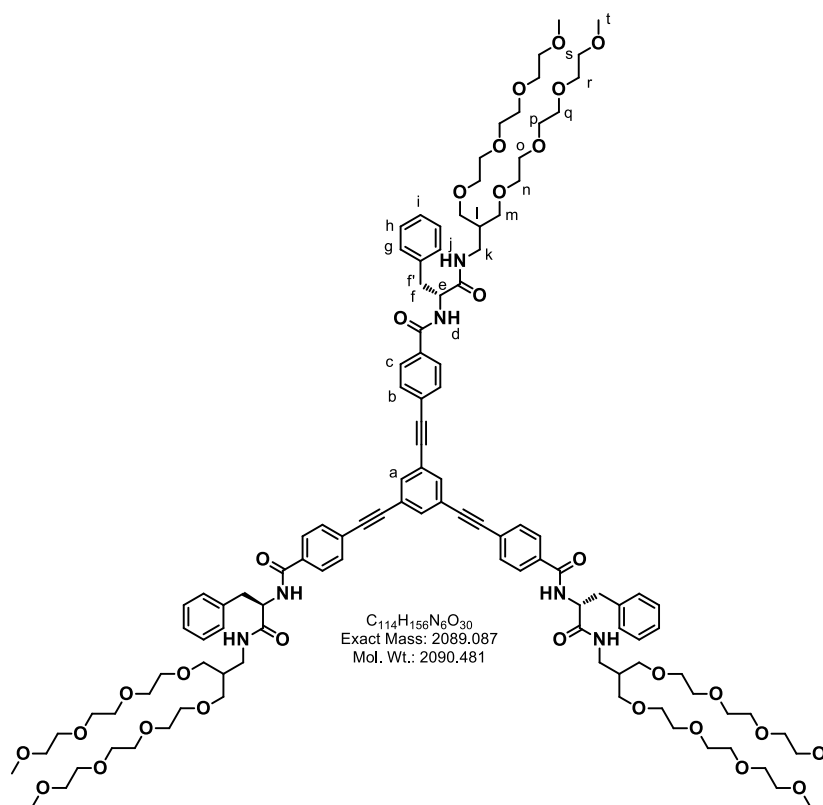
N,N,N',N'-Tetramethyl-O-(1H-benzotriazol-1-yl)uronium hexafluorophosphate (HBTU) (0.46 g, 1.21 mmol) was dissolved in dry DMF (12 mL) under argon atmosphere and 4-iodobenzoic acid (0.30 g, 1.21 mmol) and N,N-Diisopropylethylamine (DIPEA) (0.5 mL, 2.68 mmol) were added. The reaction mixture was stirred for 20 minutes. Then, amine **10** (0.40 g, 0.67 mmol) was added. The reaction mixture was stirred at room temperature overnight. The residue was washed with HCl (1M)/water/ice, NaOH (3M) and NaHCO_3 , extracted with diethyl ether and dried over MgSO_4 . After evaporation of the solvent under reduced pressure, the residue was purified by column chromatography (silica gel, chloroform:methanol 50:1) affording compound **12** as a white solid (0.36 g, 46%). ^1H NMR (CDCl_3 , 300 MHz) δ 7.75 (2H, H_a , d, $J = 8.5$); 7.48 (2H, H_b , d, $J = 8.5$); 7.29–7.16 (5H, H_{f+g+h} , m); 7.10 (1H, H_c , d, $J = 7.4$); 7.01 (1H, H_i , t, $J = 5.4$); 4.75 (1H, H_d , q, $J = 6.9$); 3.67–3.22 (36H, $H_{j+l+m+n+o+p+q+r+s}$, m); 3.18–3.11 (2H, $H_{e+e'}$, m); 2.04 (1H, H_k , m). ^{13}C NMR (CDCl_3 , 75 MHz) δ 170.64, 166.14, 137.82, 136.91, 133.64, 129.53, 128.90, 128.67, 127.03, 98.74, 72.03, 71.12, 71.08, 70.76, 70.59, 70.50, 70.47, 70.42, 59.13, 55.15, 40.57, 38.79. FTIR (neat) 664, 701, 846, 1005, 1105, 1245, 1280, 1353, 1451, 1476, 1535, 1587, 1635, 1722, 2869, 3295. HRMS (MALDI-TOF): calc. for $\text{C}_{34}\text{H}_{51}\text{IN}_2\text{NaO}_{10}$ $[\text{M}+\text{Na}]^+$ 797.2486; found 797.2459.

Compound 1



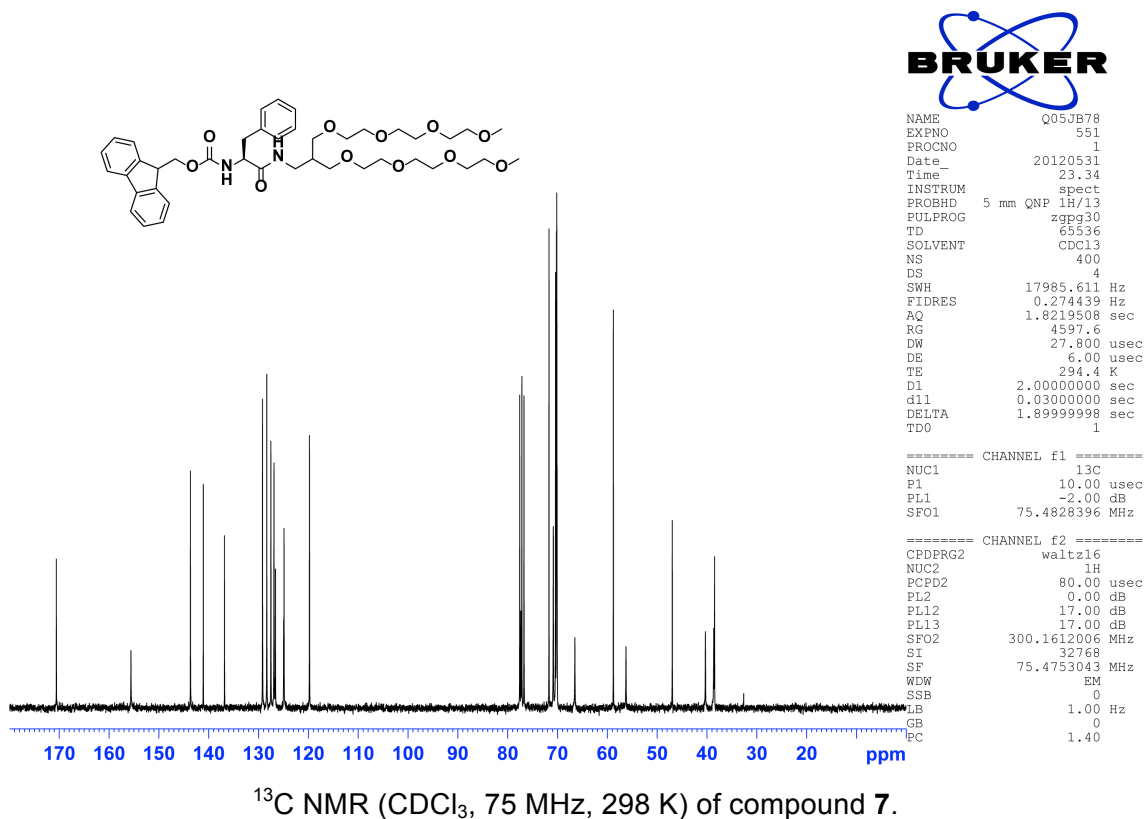
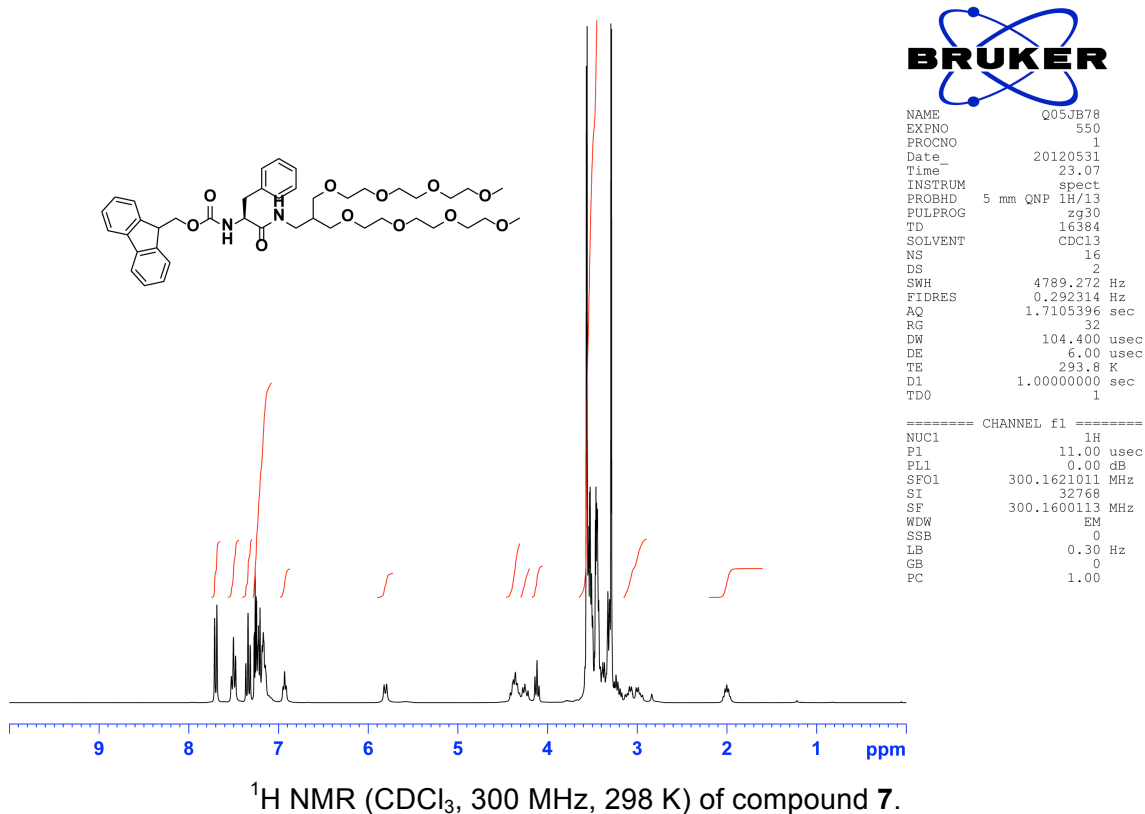
Compound **11** (0.49 g, 0.63 mmol), compound **13** (0.029 g, 0.19 mmol), bis-(triphenylphosphine)-palladium(II) chloride (0.007 g, 0.01 mmol), copper(I) iodide (0.0021 g, 0.011 mmol), were dissolved in dry THF (10 mL) and subjected to several vacuum/argon cycles. After that, triethylamine (2.5 mL) was added and subjected to more vacuum/argon cycles. The reaction mixture was heated at 67 °C and stirred 48 hours. After evaporation of the solvent under reduced pressure, the residue was washed with HCl 1M, extracted with chloroform, washed with NH₄Cl saturated solution and water and dried over MgSO₄. After evaporation of the solvent, the residue was purified by column chromatography (silica gel, chloroform:methanol 50:1) affording compound **1** as a yellow solid (0.15 g, 38%). ¹H NMR (CDCl₃, 300 MHz) δ 7.76 (6H, H_c, d, J = 8.4); 7.68 (3H, H_a, s); 7.56 (6H, H_b, d, J = 8.4); 7.34–7.20 (15H, H_{g+h+i}, m); 7.08 (3H, H_d, d, J = 7.6); 6.90 (3H, H_j, t, J = 5.5); 4.77 (3H, H_e, m); 3.66–3.23 (108H, H_{k+m+n+o+p+q+r+s+t}, m), 3.19 (6H, H_{f+f}, d, J = 6.7); 2.05 (3H, H_l, m). ¹³C NMR (CDCl₃, 75 MHz) δ 170.61, 166.09, 136.97, 134.63, 134.02, 131.87, 129.54, 128.68, 127.38, 127.02, 126.12, 123.91, 90.07, 89.94, 72.03, 71.15, 71.10, 70.77, 70.59, 70.51, 70.47, 70.42, 59.12, 55.21, 40.60, 38.79. FTIR (neat) 699, 751, 851, 877, 1105, 1249, 1306, 1354, 1450, 1497, 1532, 1632, 2868, 2919, 3287. HRMS (MALDI-TOF): calc. for C₁₁₄H₁₅₇N₆NaO₃₀ [M+H+Na]⁺ 2113.0842; found 2113.0869.

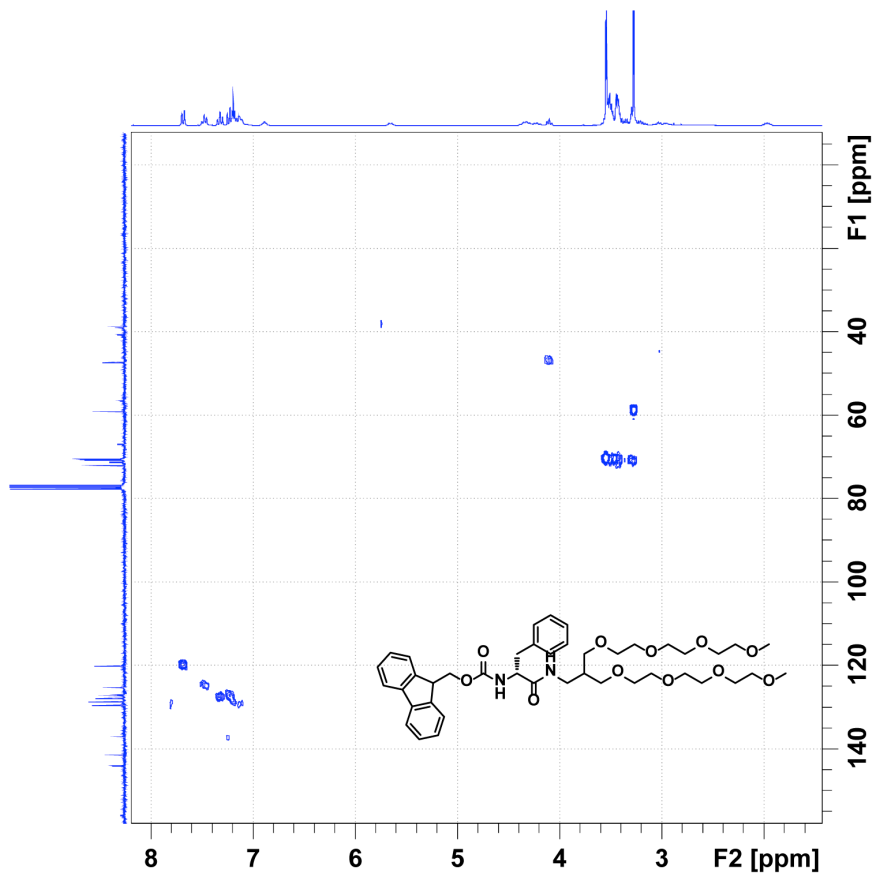
Compound 2



Compound **12** (0.40 g, 0.52 mmol), compound **13** (0.024 g, 0.16 mmol), bis-(triphenylphosphine)-palladium(II) chloride (0.011 g, 0.016 mmol), copper(I) iodide (0.0012 g, 0.006 mmol), were dissolved in dry THF (10 mL) and subjected to several vacuum/argon cycles. After that, triethylamine (2 mL) was added and subjected to more vacuum/argon cycles. The reaction mixture was heated at 67 °C and stirred 20 hours. After evaporation of the solvent under reduced pressure, the residue was washed with HCl 1M, extracted with chloroform, washed with NH₄Cl saturated solution and water and dried over MgSO₄. After evaporation of the solvent, the residue was purified by column chromatography (silica gel, chloroform:methanol 50:1) affording compound **2** as a yellow solid (0.15 g, 47%). ¹H NMR (CDCl₃, 300 MHz) δ 7.77 (6H, H_c, d, J = 8.4); 7.69 (3H, H_a, s); 7.57 (6H, H_b, d, J = 8.4); 7.32–7.20 (15H, H_{g+h+i}, m); 7.16 (3H, H_d, d, J = 7.6); 7.08 (3H, H_j, t, J = 5.5); 4.79 (3H, H_e, m); 3.66–3.27 (108H, H_{k+m+n+o+p+q+r+s+t}, m), 3.19 (6H, H_{f+f}, m); 2.06 (3H, H_l, m). ¹³C NMR (CDCl₃, 75 MHz) δ 170.83, 166.16, 136.90, 134.62, 133.91, 131.87, 129.55, 128.68, 127.42, 127.03, 126.14, 123.89, 90.07, 89.95, 72.01, 71.06, 70.99, 70.74, 70.57, 70.45, 70.47, 70.42, 59.13, 55.21, 40.55, 38.76. FTIR (neat) 700, 754, 853, 1026, 1107, 1245, 1281, 1378, 1454, 1495, 1546, 1641, 2858, 2923, 3294. HRMS (MALDI-TOF): calc. for C₁₁₄H₁₅₆N₆NaO₃₀ [M+Na]⁺ 2112.076; found 2112.084.

4. Collection of spectra



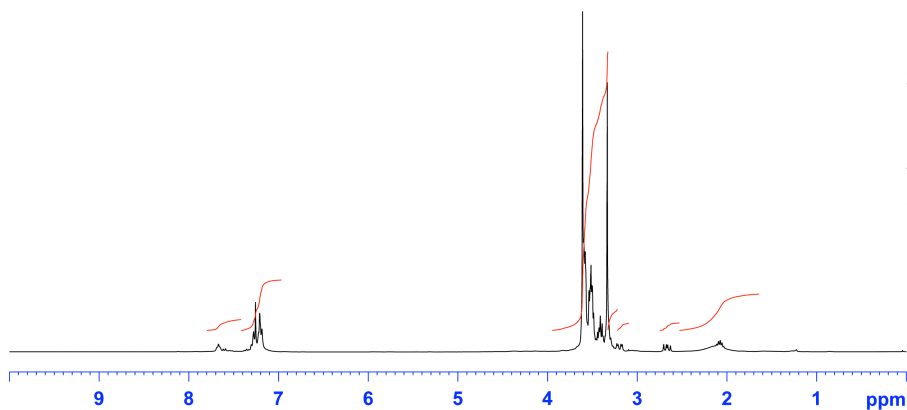
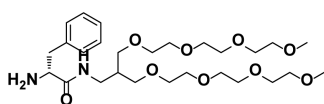


^1H , ^{13}C -HMQC spectrum (CDCl_3 , 298 K) of compound **9**.



```

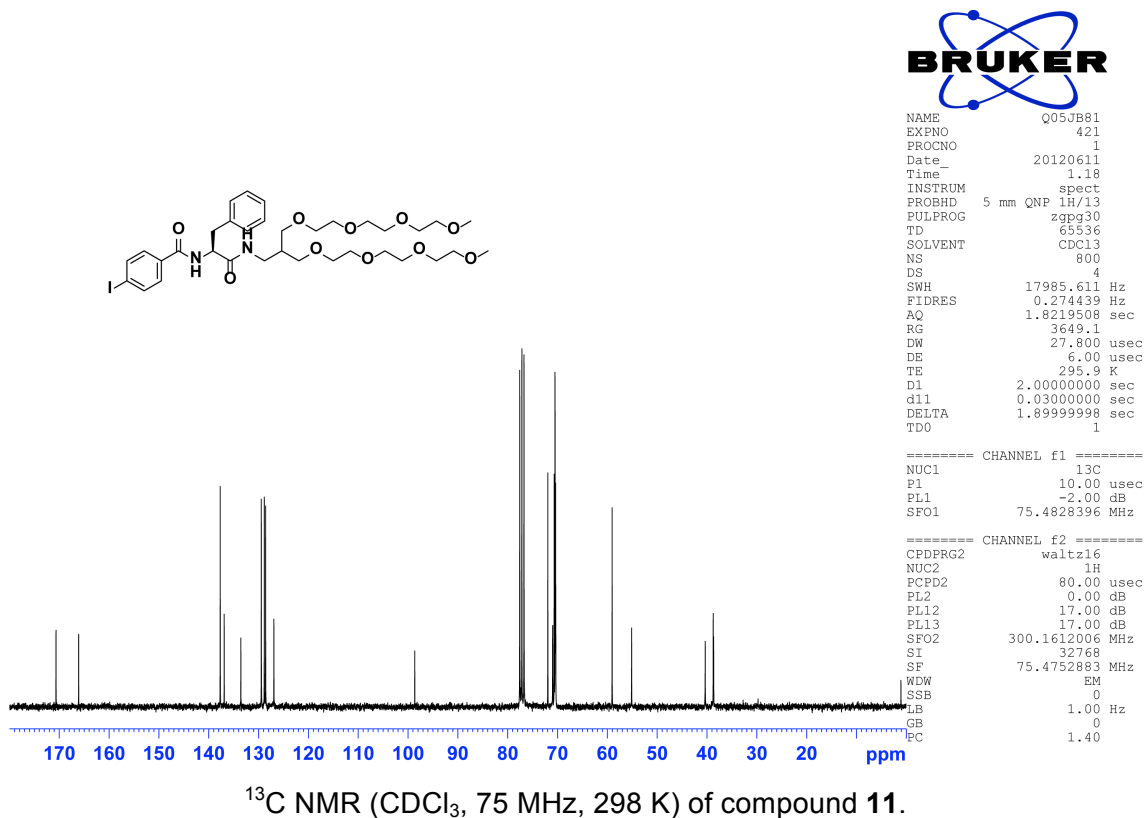
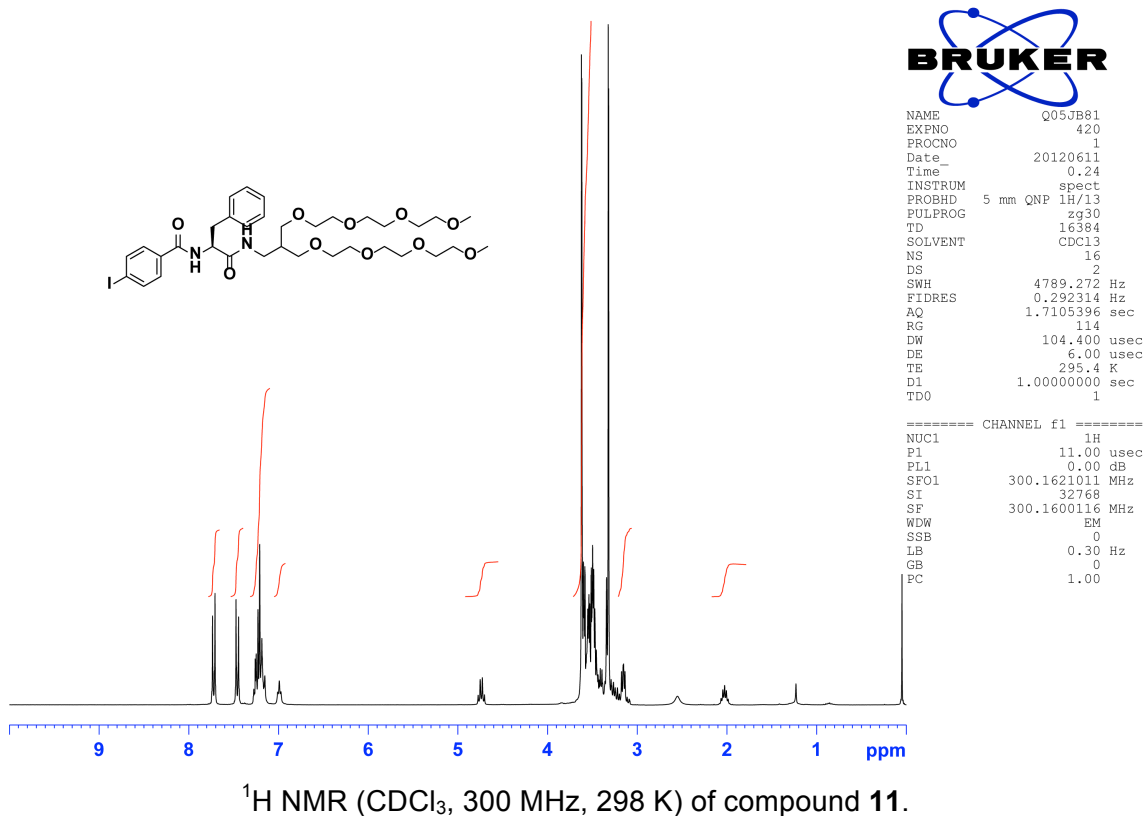
NAME          Q05FG462
EXPNO         520
PROCNO        1
Date_         20120907
Time_         16.36
INSTRUM       spect
PROBHD        5 mm QNP 1H/13
PULPROG       zg30
TD            16384
SOLVENT       CDCl3
NS            16
DS            2
SWH           4789.272 Hz
FIDRES        0.292314 Hz
AQ            1.7105396 sec
RG            114
DW            104.400 usec
DE            6.00 usec
TE            295.4 K
D1            1.0000000 sec
TDO           1
  
```

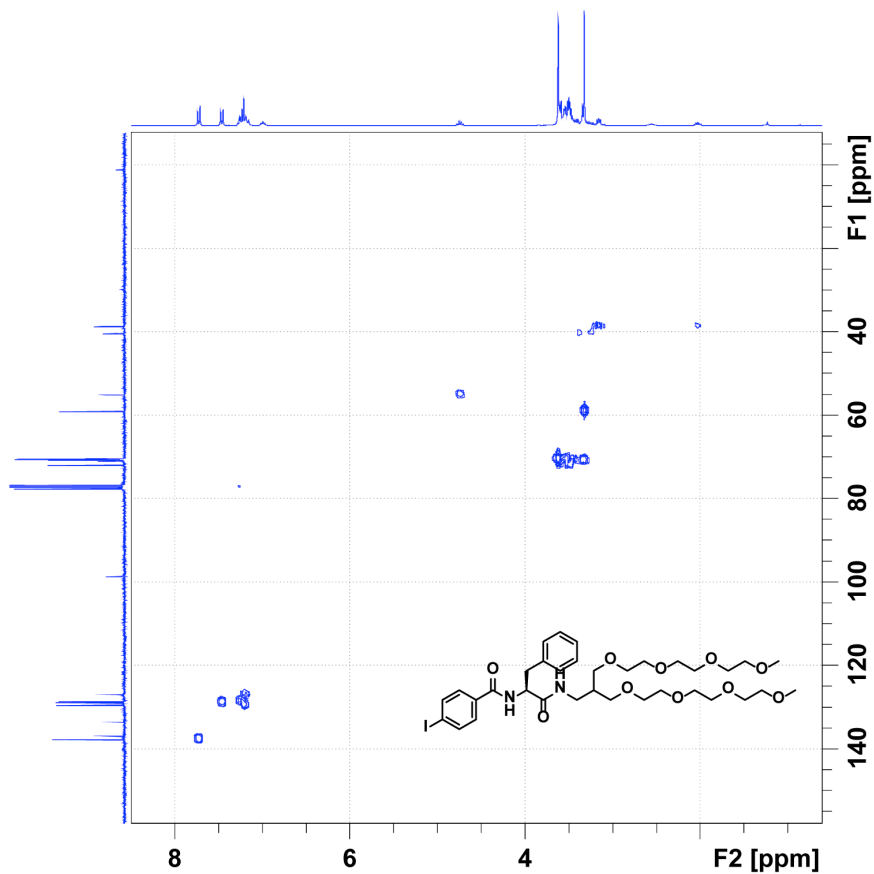


^1H NMR (CDCl_3 , 300 MHz, 298 K) of compound **10**.

```

===== CHANNEL f1 =====
NUC1          1H
P1            12.50 usec
PL1           0.00 dB
SF01         300.1621011 MHz
SI            32768
SF           300.1600122 MHz
WDW           EM
SSB           0
LB            0.30 Hz
GB            0
PC            1.00
  
```



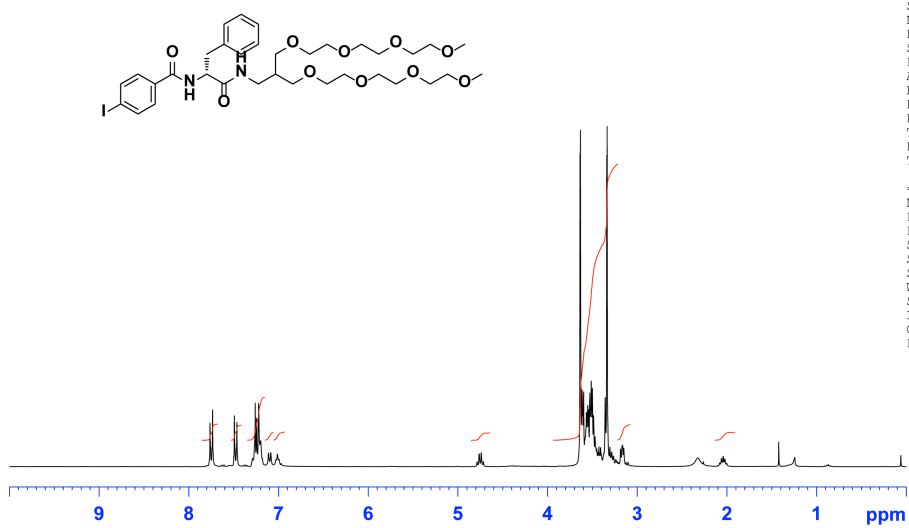
^1H , ^{13}C -HMQC spectrum (CDCl_3 , 298 K) of compound 11.



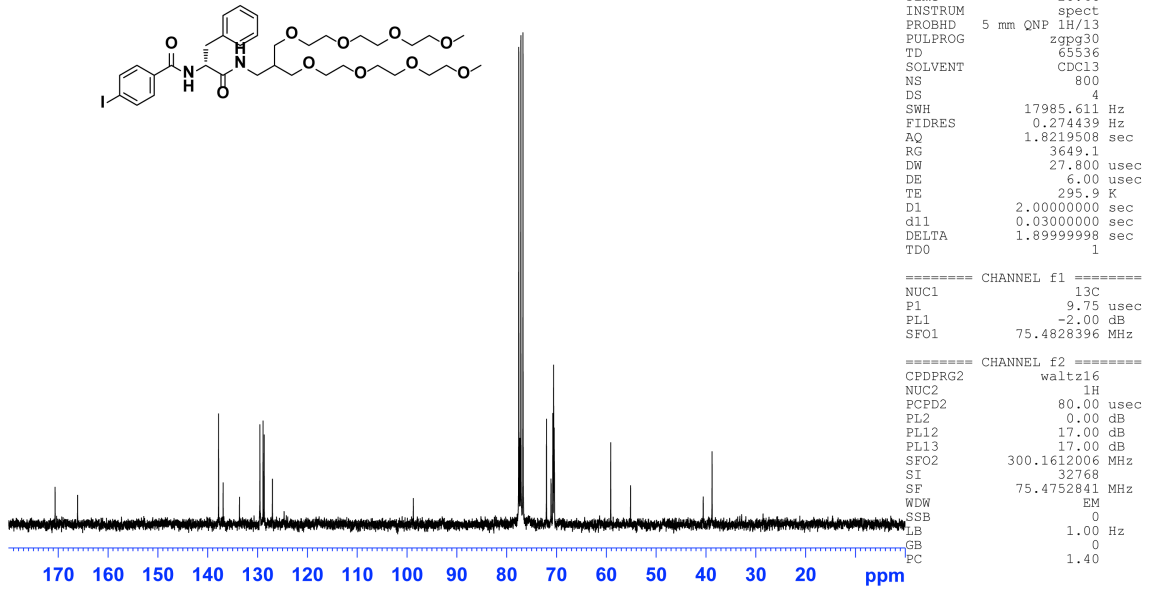
```

NAME          Q05FG463
EXPNO         730
PROCNO        1
Date_         20120924
Time_         19.51
INSTRUM       spect
PROBHD        5 mm QNP 1H/13
PULPROG       zg30
TD            16384
SOLVENT       CDCl3
NS            16
DS            2
SWH           4789.272 Hz
FIDRES        0.292314 Hz
AQ            1.7105396 sec
RG            256
DW            104.400 usec
DE            6.00 usec
TE            295.1 K
D1            1.0000000 sec
TDO           1

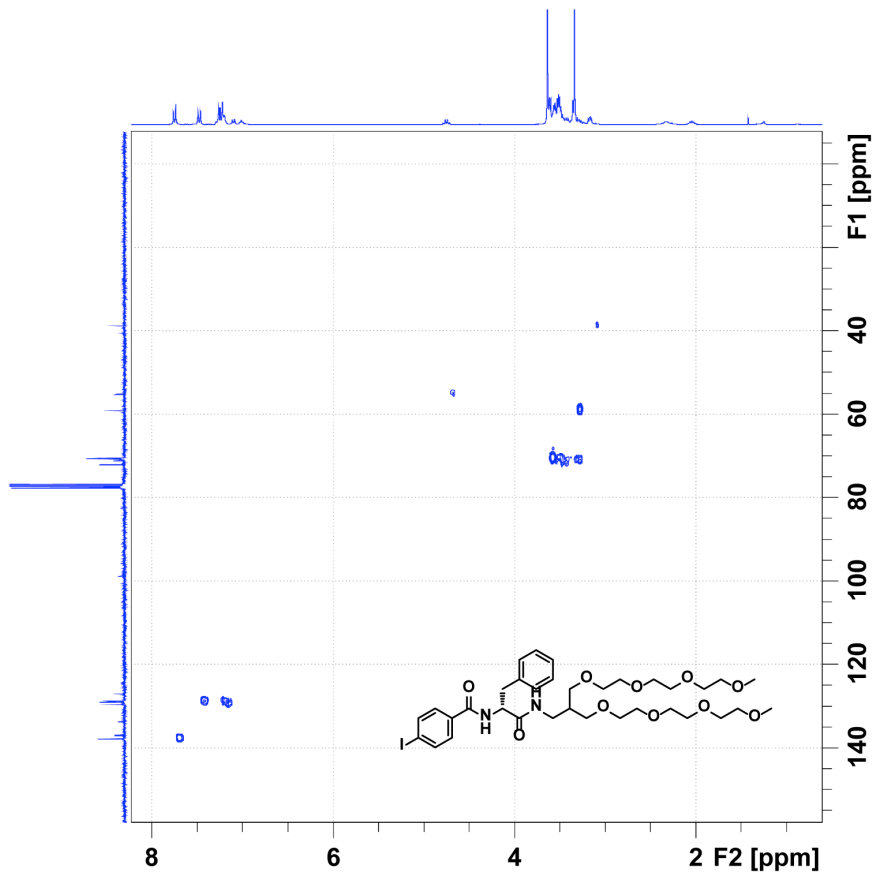
===== CHANNEL f1 =====
NUC1          13C
P1            12.50 usec
PL1           0.00 dB
SF01          300.1621011 MHz
SI            32768
SF            300.1600116 MHz
WDW           EM
SSB           0
LB            0.30 Hz
GB            0
PC            1.00
  
```



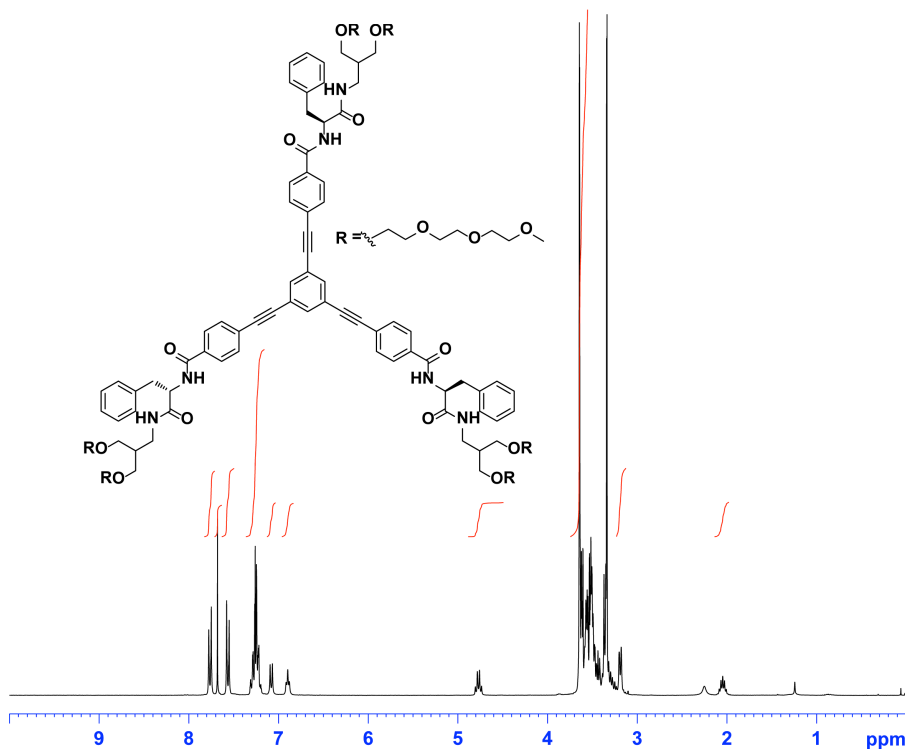
^1H NMR (CDCl_3 , 300 MHz, 298 K) of compound 12.



^{13}C NMR (CDCl_3 , 75 MHz, 298 K) of compound **12**.



^1H , ^{13}C -HMQC spectrum (CDCl_3 , 298 K) of compound **12**.



```

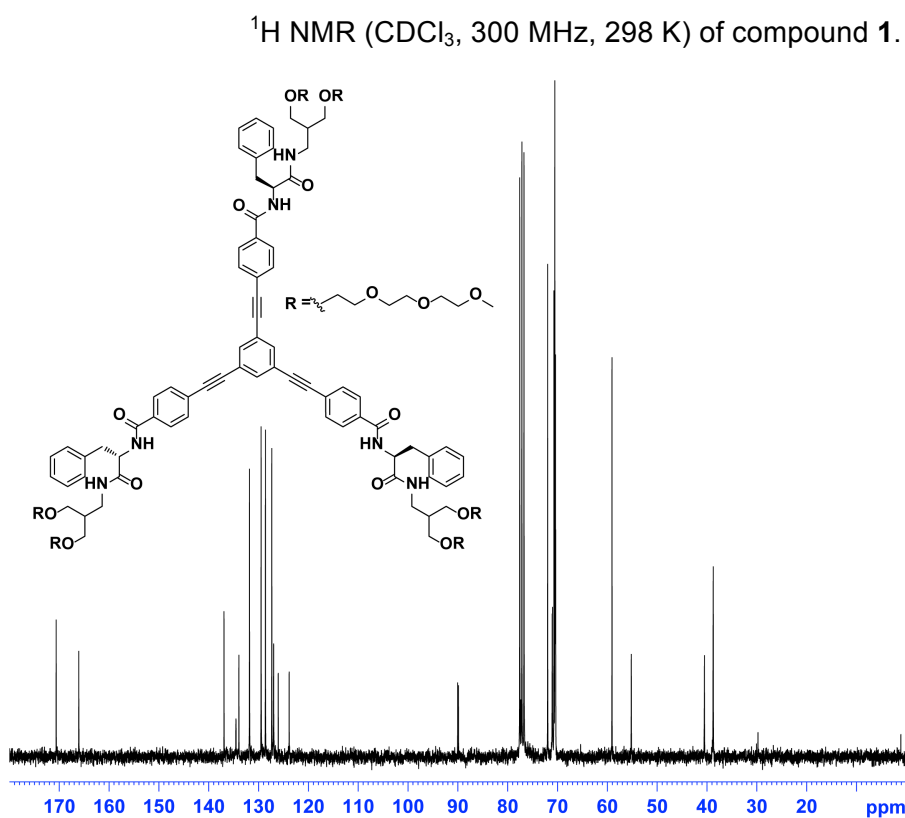
NAME      Q05JB83
EXPNO     590
PROCNO    1
Date_     20120621
Time      17.48
INSTRUM   spect
PROBHD    5 mm QNP 1H/13
PULPROG   zg30
TD         16384
SOLVENT   CDCl3
NS         16
DS         2
SWH        4789.272 Hz
FIDRES     0.292314 Hz
AQ         1.7105396 sec
RG         256
DW         104.400 usec
DE         6.00 usec
TE         295.5 K
D1         1.0000000 sec
D11        1
TD0        1

```

```

===== CHANNEL f1 =====
NUC1      1H
P1        11.00 usec
PL1       0.00 dB
SFO1     300.1621011 MHz
SI        32768
SF        300.1600117 MHz
WDW       EM
SSB       0
LB        0.30 Hz
GB        0
PC        1.00

```



```

NAME      Nueva carpeta
EXPNO     711
PROCNO    1
Date_     20120913
Time      20.35
INSTRUM   spect
PROBHD    5 mm QNP 1H/13
PULPROG   zgpg30
TD         65536
SOLVENT   CDCl3
NS         800
DS         4
SWH        17985.611 Hz
FIDRES     0.274439 Hz
AQ         1.8219508 sec
RG         4096
DW         27.800 usec
DE         6.00 usec
TE         295.7 K
D1         2.0000000 sec
d11        0.0300000 sec
DELTA     1.899999998 sec
TD0        1

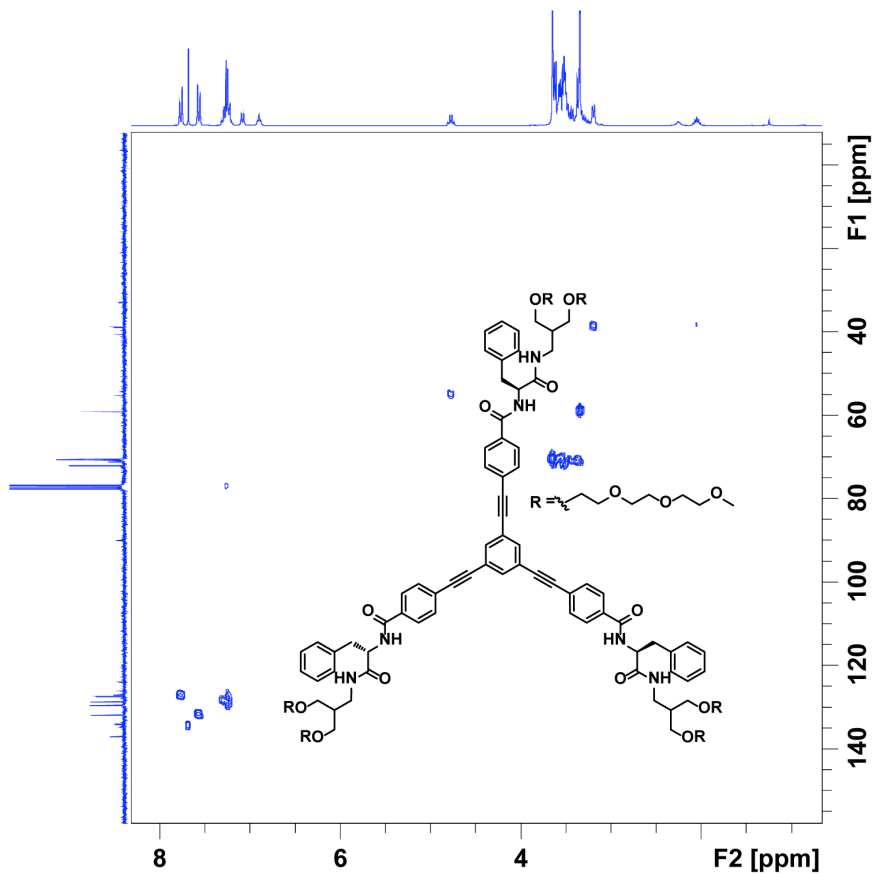
```

```

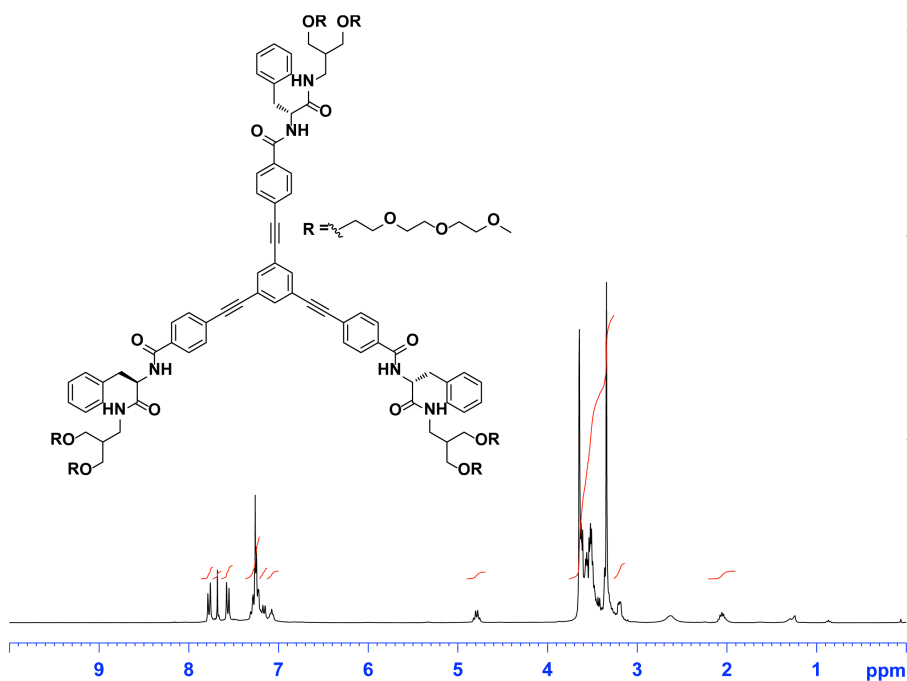
===== CHANNEL f1 =====
NUC1      13C
P1        9.75 usec
PL1       -2.00 dB
SFO1     75.4828396 MHz

===== CHANNEL f2 =====
CPDPRG2   waltz16
NUC2      1H
PCPD2     80.00 usec
PL2       0.00 dB
PL12      17.00 dB
PL13      17.00 dB
SFO2     300.1612006 MHz
SI        32768
SF        75.4752872 MHz
WDW       EM
SSB       0
LB        1.00 Hz
GB        0
PC        1.40

```



^1H , ^{13}C -HMQC spectrum (CDCl_3 , 298 K) of compound 1.



^1H NMR (CDCl_3 , 300 MHz, 298 K) of compound 2.



```

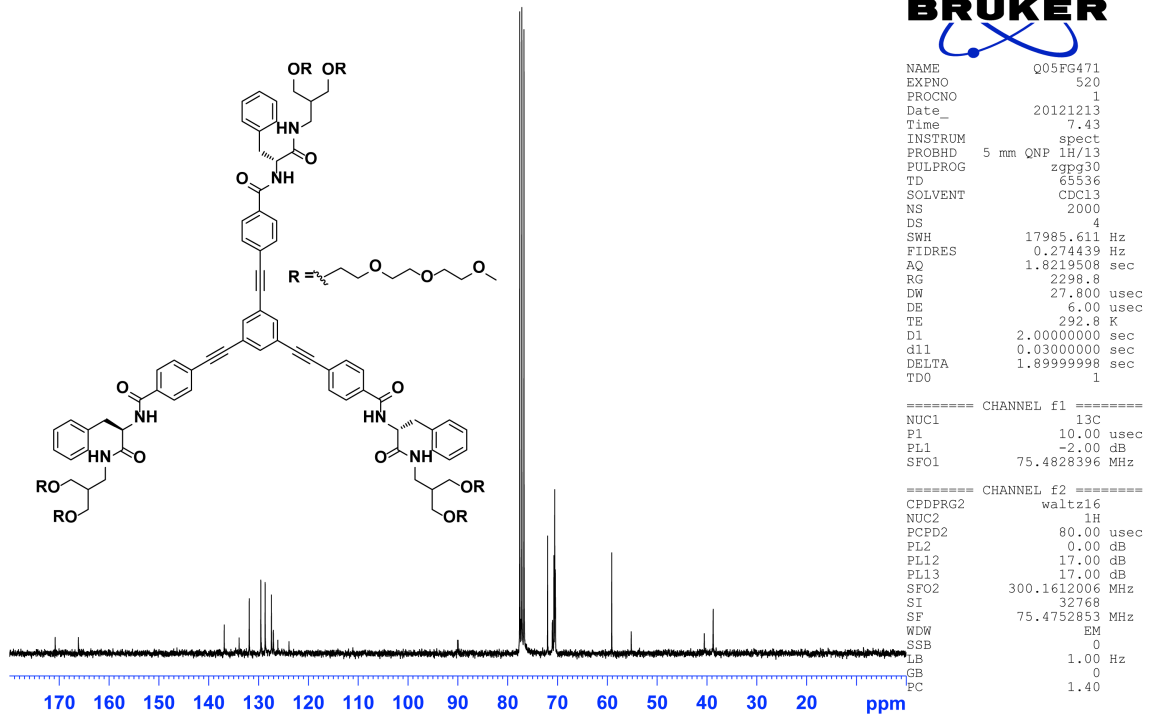
NAME          Q05FG471
EXPNO         470
PROCNO        1
Date_         20121210
Time_         16.23
INSTRUM       spect
PROBHD        5 mm QNP 1H/13
PULPROG       zg30
TD            16384
SOLVENT       CDCl3
NS            16
DS            2
SWH           4789.272 Hz
FIDRES        0.292314 Hz
AQ            1.7105396 sec
RG            287.4
DW            104.400 usec
DE            6.00 usec
TE            292.0 K
D1            1.0000000 sec
TDO           1

```

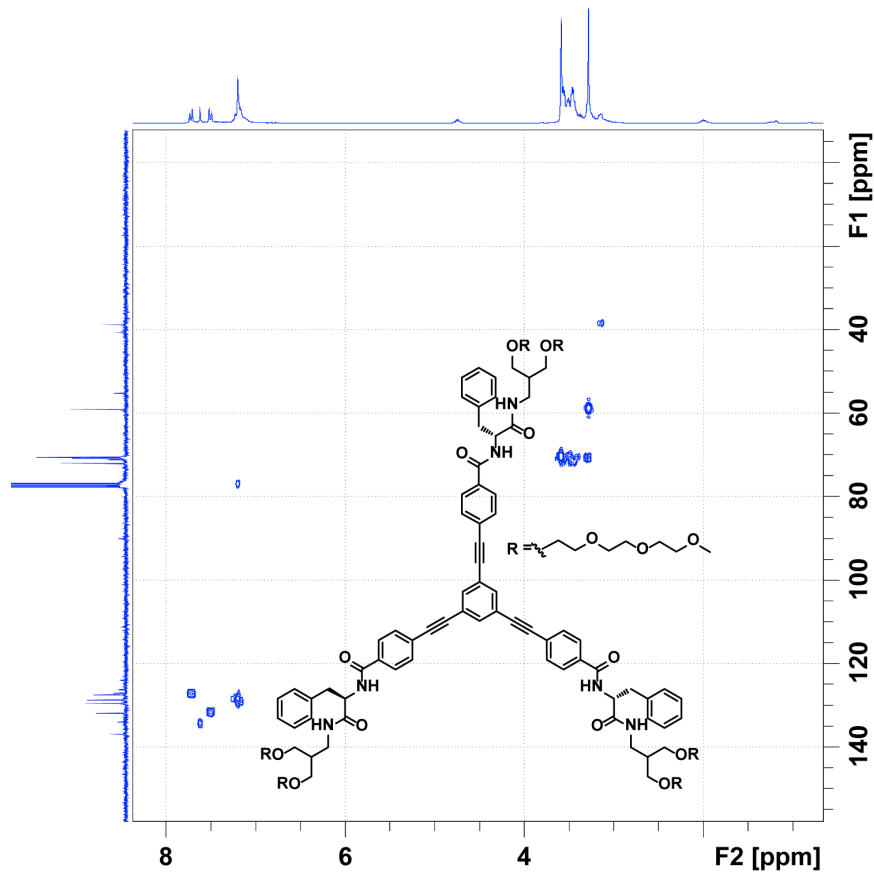
```

===== CHANNEL f1 =====
NUC1          13C
P1            10.50 usec
PL1           0.00 dB
SFO1          300.1621011 MHz
SI            32768
SF            300.1600119 MHz
WDW           EM
SSB           0
LB            0.30 Hz
GB            0
PC            1.00

```



^{13}C NMR (CDCl_3 , 75 MHz, 298 K) of compound 2.



^1H , ^{13}C -HMQC spectrum (CDCl_3 , 298 K) of compound 2.