

Supporting Information

Polymer-supported (2,6-Dichloro-4-alkoxyphenyl)(2,4-dichlorophenyl)methanol: A New Linker for Solid-Phase Organic Synthesis

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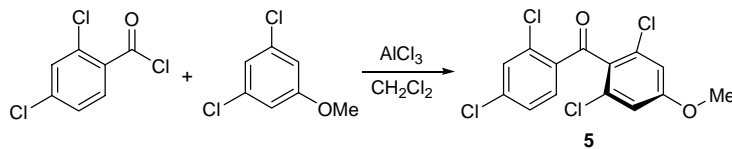
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General Procedures and Methods: All glassware was oven dried, assembled hot and cooled under a stream of nitrogen before use. Reactions with air sensitive materials were carried out by standard syringe techniques. Commercially available reagents were used as received without further purification. Thin layer chromatography was performed using 0.25 mm silica gel 60 (F254, Merck) plates visualizing at 254 nm, or developed with potassium permanganate solutions by heating with a hot-air gun. Specified products were purified by flash column chromatography using silica gel 60 (230-400 mesh, Merck). IR absorptions on NaCl plates were run on a Perkin Elmer FT-IR 1600. ¹H NMR spectral data were obtained using Varian 300, 400 MHz instruments. The residual solvent signal was utilized as an internal reference. ¹³C NMR spectral data were obtained using a Varian 100 MHz spectrometer. Chemical shifts were reported in parts per million (ppm) downfield from TMS, using the middle resonance of CDCl₃ (77.0 ppm) as an internal standard. For all NMR spectra, δ values are given in ppm and *J* values in Hz. Mass spectra were obtained at Colorado State University's Central Instrument Facility.

Reagents and solvents are commercial grade and were used as supplied. Reaction vessels were flame-dried or oven-dried and cooled under an inert atmosphere when necessary.

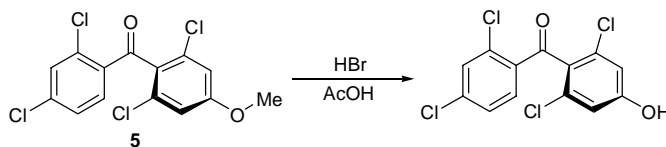
Experimental Procedures and Compound data:

(2,6-dichloro-4-methoxyphenyl)(2,4-dichlorophenyl)methanone (5)



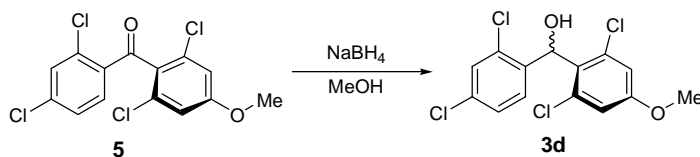
Anhydrous AlCl₃ (4.5 g, 33.9 mmol) was taken in a round bottom flask and dry CH₂Cl₂ (150 mL) was added. At -78°, 2,4-dichlorobenzoyl chloride (4.7 mL, 33.9 mmol) and 3,5-dichloro anisole (5.0 g, 28.2 mmol) were added. The reaction was kept at the same temperature for 1 h and warmed up to r.t over 5h. The reaction mixture was quenched by 2N HCl at 0°C and extracted with CH₂Cl₂. The organic phase was washed with 2N NaOH, brine, dried over Na₂SO₄, filtered, and concentrated *in vacuo*. Purification by silica gel chromatography (25:1, hexanes:EtOAc) to provide (2,6-dichloro-4-methoxyphenyl)(2,4-dichlorophenyl)methanone (5) (7.0 g, 71 %) as a white powder. Data for 5: ¹H NMR (300 MHz, CDCl₃): δ 7.68 (d, *J* = 8.2 Hz, 1H), 7.54 (d, *J* = 2.1 Hz, 1H), 7.37 (dd, *J* = 8.1, 2.1 Hz, 1H), 6.95 (s, 2H), 3.87 (s, 3H); ¹³C NMR (75 MHz, CDCl₃): δ 197.6, 163.8, 142.0, 138.3, 137.7, 136.4, 130.3, 129.9, 127.8, 122.9, 118.4, 117.9, 114.6, 56.1; IR (film): 1619 cm⁻¹, 1584, 1553, 1400, 1309; LRMS (FAB) C₁₄H₈Cl₄O₂ found = 353, 351, and 349.

2,6-dichloro-4-hydroxyphenyl(2,4-dichlorophenyl)methanone



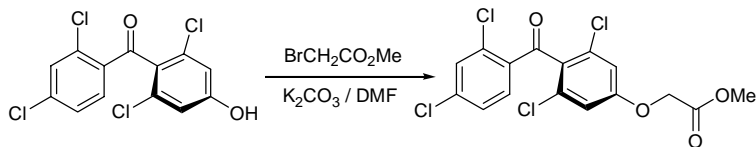
(2,6-Dichloro-4-methoxyphenyl)(2,4-dichlorophenyl)methanone (**5**) (5.0 g, 14.3 mmol) was dissolved in AcOH (50 mL) at 60°C and then HBr (48%, 50 mL) was added. After 6h at 110 °C, all volatiles were evaporated *in vacuo*. Then water was added to it and extracted with ethyl acetate. The organic extract was finally washed with brine, dried over Na₂SO₄, and concentrated *in vacuo*. Purification by silica gel chromatography (9:1 hexanes:EtOAc) to 2,6-dichloro-4-hydroxyphenyl(2,4-dichlorophenyl)methanone (4.6 g, 96%) as a white powder. ¹H NMR (300 MHz, CD₃OD): δ 7.71 (d, *J* = 8.2 Hz, 1H), 7.65 (d, *J* = 1.2 Hz, 1H), 7.56 (dd, *J* = 8.1, 1.3 Hz, 1H), 6.91 (s, 2H); ¹³C NMR (100 MHz, CD₃OD): δ 190.4, 160.1, 139.2, 134.7, 134.6, 133.2, 133.2, 132.7, 131.1, 128.9, 127.5, 115.5, 115.5; IR (film): 3583 cm⁻¹, 1450, 1400, 1305. LRMS (FAB) C₁₃H₆C₁₄O₂ found = 37, 36, and 334.

(2,6-dichloro-4-methoxyphenyl)(2,4-dichlorophenyl)methanol (**3d**)



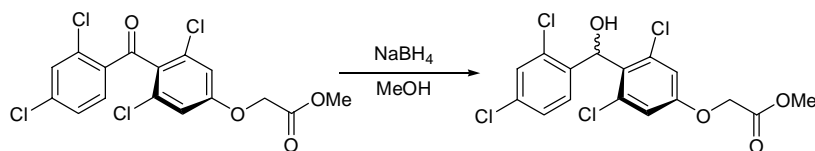
(2,6-Dichloro-4-methoxyphenyl)(2,4-dichlorophenyl)methanone (**5**) (1.0 g, 2.8 mmol) was dissolved in methanol and cooled to 0°C. Into the reaction mixture NaBH₄ (318 mg, 8.4 mmol) was added. The reaction mixture was quenched by aq.NH₄Cl and extracted with EtOAc. The combined extracts were washed with brine, dried over Na₂SO₄, and concentrated *in vacuo*. Purification by silica gel chromatography (5:1, hexanes:EtOAc) to provide **3d** (980 mg, 97%) as a white powder. ¹H NMR (300 MHz, CDCl₃): δ 7.69 (d, *J* = 8.4 Hz, 1H), 7.38 (d, *J* = 3.0 Hz, 1H), 7.31 (dd, *J* = 8.2, 3.0 Hz, 1H), 6.91 (s, 2H), 6.61 (d, *J* = 2.1 Hz, 1H), 3.85 (s, 3H); ¹³C NMR (75 MHz, CDCl₃): δ 159.6, 137.8, 136.3, 133.9, 133.0, 130.5, 129.6, 129.6, 128.0, 126.6, 115.4, 115.4, 70.2, 55.9; IR (film): 3482 cm⁻¹, 1438, 1410, 1325; LRMS (FAB) C₁₄H₁₀C₁₄O₂ found = 352 and 335.

Methyl 2-(3,5-dichloro-4-(2,4-dichlorobenzoyl)phenoxy)acetate



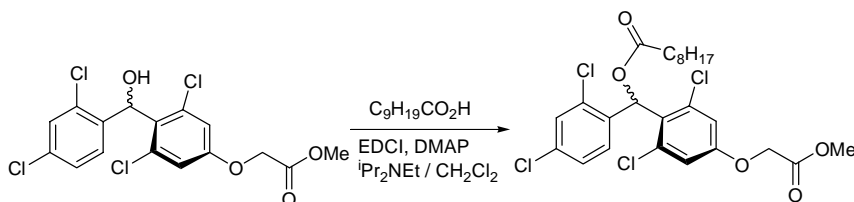
2,6-Dichloro-4-hydroxyphenyl(2,4-dichlorophenyl)methanone (500 mg, 1.5 mmol) was dissolved in DMF (10 mL) and K₂CO₃ (622mg, 4.5 mmol) and methyl bromoacetate (0.4 mL, 4.5 mmol) were added. After 6h at rt, the reaction mixture was quenched with water and extracted with EtOAc. The organic extracts were with brine, dried over Na₂SO₄, and concentrated *in vacuo*. Purification by silica gel chromatography (15:1, hexanes:EtOAc) to provide methyl 2-(3,5-dichloro-4-(2,4-dichlorobenzoyl)phenoxy)acetate (560 mg, 92%) as a white powder. ¹H NMR (300 MHz, CDCl₃): δ 7.67 (d, *J* = 8.4 Hz, 1H), 7.55 (d, *J* = 2.1 Hz, 1H), 7.39 (dd, *J* = 8.1, 2.1 Hz, 1H), 6.95 (s, 2H), 4.72 (s, 2H), 3.89 (s, 3H); ¹³C NMR (75 MHz, CDCl₃): δ 190.0, 168.3, 159.1, 139.7, 135.2, 134.0, 133.4, 133.3, 131.8, 131.6, 127.6, 127.6, 115.2, 115.2, 65.5, 52.8; IR (film): 1759 cm⁻¹, 1688, 1583, 1556, 1437; LRMS (FAB) C₁₆H₁₀Cl₄O₄ found = 410, 409, 408, and 407.

Methyl 2-(3,5-dichloro-4-((2,4-dichlorophenyl)(hydroxy)methyl)phenoxy)acetate



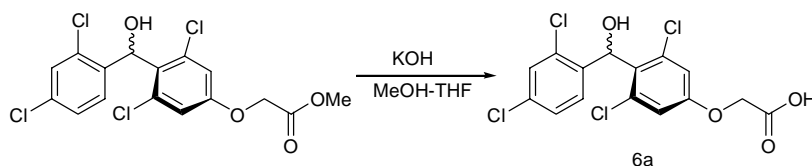
2-(3,5-Dichloro-4-(2,4-dichlorobenzoyl)phenoxy)acetate (408 mg, 1.0 mmol) was dissolved in methanol and cooled to 0°C. Into the reaction mixture NaBH₄ (113 mg, 3.0 mmol) was added. The reaction mixture was quenched by aq. NH₄Cl and extracted with EtOAc. The combined extracts were washed with brine, dried over Na₂SO₄, and concentrated *in vacuo*. Purification by silica gel chromatography (5:1, hexanes:EtOAc) to provide methyl 2-(3,5-dichloro-4-((2,4-dichlorophenyl)(hydroxy)methyl)phenoxy)acetate (395 mg, 96%) as a white powder. ¹H NMR (300 MHz, CDCl₃): δ 7.70 (d, *J* = 8.4 Hz, 1H), 7.38 (d, *J* = 2.1 Hz, 1H), 7.31 (dd, *J* = 8.4, 2.1 Hz, 1H), 6.94 (s, 2H), 6.6 (d, *J* = 1.8 Hz, 1H), 4.71 (s, 2H), 3.87 (s, 3H); ¹³C NMR (75 MHz, CDCl₃): δ 169.3, 158.6, 137.7, 136.4, 133.9, 132.9, 130.5, 129.6, 128.5, 126.6, 126.6, 115.9, 115.9, 69.9, 61.3, 52.8; IR (film): 3335 cm⁻¹, 1598, 1556, 1468; LRMS (FAB) C₁₆H₁₂Cl₄O₄ found 410, 367, 365, and 363.

(2,6-Dichloro-4-(2-methoxy-2-oxoethoxy)phenyl)(2,4-dichlorophenyl)methyl nonanoate



Methyl 2-(3,5-dichloro-4-((2,4-dichlorophenyl)(hydroxy)methyl)phenoxy)acetate (150 mg, 0.35 mmol) was dissolved in CH₂Cl₂ (3.0 mL) and nonanoic acid (0.09 mL, 0.53 mmol), EDCI (102 mg, 0.53 mmol), DMAP (43 mg, 0.53 mmol) and *N,N*-diisopropylethyl amine (0.09 mL, 0.53 mmol) were added. After 3 h at rt, the reaction mixture was quenched by 1N HCl and extracted with EtOAc. The combined extracts were washed with aq. NaHCO₃, brine, dried over Na₂SO₄, and concentrated *in vacuo*. Purification by silica gel chromatography (20:1, hexanes:EtOAc) to provide (2,6-dichloro-4-(2-methoxy-2-oxoethoxy)phenyl)(2,4-dichlorophenyl)methyl nonanoate (190 mg, 94%) as a liquid. ¹H NMR (300 MHz, CDCl₃): δ 7.57 (s, 1H), 7.44 (d, *J* = 1.8 Hz, 1H), 7.30 (s, 1H), 7.26 (dd, *J* = 8.7, 2.1 Hz, 1H), 6.95 (s, 2H), 4.72 (s, 2H), 3.88 (s, 3H), 2.49-2.37 (m, 2H), 1.70-1.68 (m, 2H), 1.32 (m, 10H), 0.94-0.90 (m, 3H); ¹³C NMR (75 MHz, CDCl₃): δ 179.5, 172.5, 158.8, 136.9, 136.9, 134.8, 134.4, 133.6, 130.8, 130.0, 126.7, 125.3, 116.0, 116.0, 66.7, 62.2, 51.9, 35.5, 34.1, 32.0, 29.2, 25.0, 24.4, 24.1, 14.3; IR (film): 2955 cm⁻¹, 2927, 2855, 1818, 1743, 1598, 1557; LRMS (FAB) C₂₅H₂₈Cl₄O₅ found 550, 478, and 477.

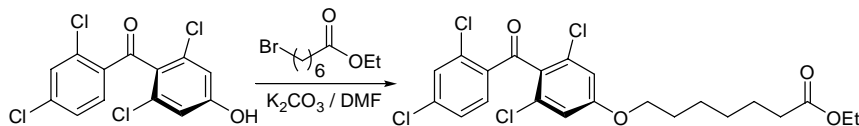
2-(3,5-Dichloro-4-((2,4-dichlorophenyl)(hydroxy)methyl)phenoxy)acetic acid (6a)



Methyl 2-(3,5-dichloro-4-((2,4-dichlorophenyl)(hydroxy)methyl)phenoxy)acetate (205 mg, 0.5 mmol) was dissolved in MeOH : THF (9:1) solution and 2N KOH was added. After 3h at rt, the reaction was poured into 2N HCl and extracted with EtOAc. The combined extracts were washed with brine, dried over Na₂SO₄, and concentrated *in vacuo* to provide **6a** (192 mg, 97%) as a white solid. ¹H NMR (300 MHz, CDCl₃): δ 7.71 (d, *J* = 8.2 Hz, 1H), 7.36 (d, *J* = 2.1 Hz, 1H), 7.32 (dd, *J* = 8.2, 2.1 Hz, 1H), 6.92 (s, 2H), 5.97 (d, *J* = 1.8 Hz, 1H), 4.66 (s, 2H); ¹³C NMR (100 MHz, CDCl₃): δ 169.3, 158.6, 137.6, 136.3, 133.8, 132.7, 130.5,

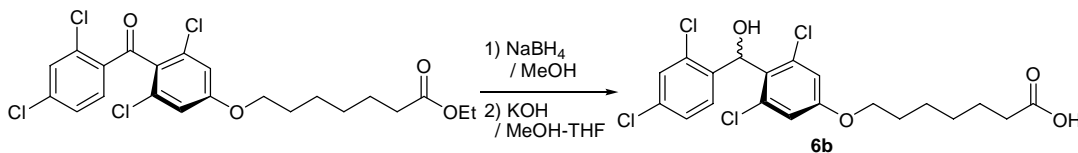
129.5, 128.4, 126.4, 126.4, 115.4, 115.4, 70.2, 61.3; IR (film): 3330 cm^{-1} , 2935, 1702, 1587; LRMS (FAB) $\text{C}_{15}\text{H}_{10}\text{Cl}_4\text{O}_4$ found 397, 395, and 393.

Ethyl 7-(3,5-dichloro-4-(2,4-dichlorobenzoyl)phenoxy)heptanoate



2,6-Dichloro-4-hydroxyphenyl(2,4-dichlorophenyl)methanone (5.0 g, 14.9 mmol) was dissolved in DMF (40 mL) and K_2CO_3 (6.17g, 44.7 mmol) and 7-bromo heptanoate (8.7 mL, 44.7 mmol) were added. After 12h at rt, the reaction mixture was quenched with water and extracted with EtOAc. The organic extracts were with brine, dried over Na_2SO_4 , and concentrated *in vacuo*. Purification by silica gel chromatography (15:1, hexanes:EtOAc) to provide Ethyl 7-(3,5-dichloro-4-(2,4-dichlorobenzoyl)phenoxy)heptanoate (7.0 g, 95%) as a white solid. ^1H NMR (400 MHz, CDCl_3): δ 7.60 (d, $J = 8.4$ Hz, 1H), 7.46 (d, $J = 2.1$ Hz, 1H), 7.29 (dd, $J = 9.2, 2.1$ Hz, 1H), 6.85 (s, 2H), 4.11 (q, $J = 7.2$ Hz, 2H), 3.94 (t, $J = 6.4$ Hz, 2H), 2.29 (t, $J = 7.6$ Hz, 2H), 1.81-1.75 (m, 2H), 1.68-1.60 (m, 2H), 1.50-1.32 (m, 4H), 1.23 (t, $J = 7.2$ Hz, 3H); ^{13}C NMR (100 MHz, CDCl_3): δ 190.3, 173.9, 160.6, 139.5, 135.1, 134.4, 133.3, 133.2, 131.5, 130.5, 127.6, 127.6, 115.0, 115.0, 68.9, 60.5, 34.4, 28.9, 28.9, 25.8, 25.0, 14.5; IR (film): 2920 cm^{-1} , 1731, 1690, 1592, 1551; LRMS (FAB) $\text{C}_{22}\text{H}_{22}\text{Cl}_4\text{O}_4$ found 495, 494, 493, 492, and 491.

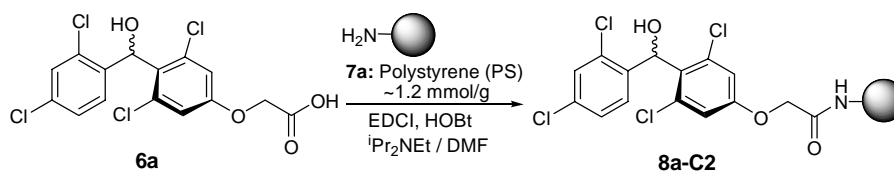
7-(3,5-Dichloro-4-((2,4-dichlorophenyl)(hydroxy)methyl)phenoxy)heptanoic acid (6b)



Ethyl 7-(3,5-dichloro-4-(2,4-dichlorobenzoyl)phenoxy)heptanoate (5.5g, 11.2 mmol) was dissolved in methanol and cooled to 0°C . Into the reaction mixture NaBH_4 (1.27g, 33.6 mmol) was added. The reaction mixture was quenched by aq. NH_4Cl and extracted with EtOAc. The combined extracts were washed with brine, dried over Na_2SO_4 , and concentrated *in vacuo*. Purification by silica gel chromatography (5:1, hexanes:EtOAc) to provide ethyl 7-(3,5-dichloro-4-((2,4-dichlorophenyl)(hydroxy)methyl)phenoxy)heptanoate (5.4 g, 98%) as a colorless oil. ^1H NMR (400 MHz, CDCl_3): δ 7.62 (d, $J = 8.2$ Hz, 1H), 7.30 (d, $J = 2.0$ Hz, 1H), 7.20 (dd, $J = 8.1, 2.0$ Hz, 1H), 6.82 (s, 2H), 6.5 (d, $J = 1.6$ Hz, 1H), 4.10 (q, $J = 7.2$ Hz, 2H), 3.90 (t, $J = 6.4$ Hz, 2H), 2.28 (t, $J = 7.2$ Hz, 2H), 1.79-1.70 (m, 2H), 1.67-1.58 (m, 2H), 1.48-1.29 (m, 4H), 1.22 (t, $J = 7.2$ Hz, 3H); ^{13}C NMR (100 MHz, CDCl_3): δ 174.0, 159.1, 137.8, 136.2, 133.8, 133.0, 130.5, 129.5, 129.5, 127.8, 126.5, 115.9, 70.2, 68.6, 60.5, 51.7, 34.4, 34.1, 28.9, 25.8, 25.0, 14.4; IR (film): 3512 cm^{-1} , 2938, 1732, 1596, 1554; LRMS (FAB) $\text{C}_{22}\text{H}_{24}\text{Cl}_4\text{O}_4$ found 494, 481, 480, 479, 478, and 477.

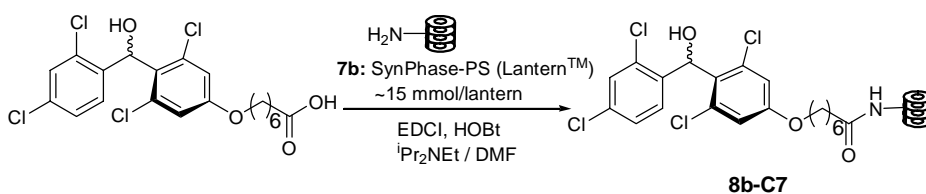
Ethyl 7-(3,5-dichloro-4-((2,4-dichlorophenyl)(hydroxy)methyl)phenoxy)heptanoate (5.0 g, 10.1 mmol) was dissolved in MeOH : THF (9:1) solution and 2N KOH was added. After 12h at rt, the reaction was poured into 2N HCl and extracted with EtOAc. The combined extracts were washed with brine, dried over Na_2SO_4 , and concentrated *in vacuo* to provide **6b** (4.6 g, 97%) as a white solid. ^1H NMR (400 MHz, CDCl_3): δ 7.62 (d, $J = 8.2$ Hz, 1H), 7.34 (d, $J = 1.6$ Hz, 1H), 7.30 (dd, $J = 8.4, 2.0$ Hz, 1H), 6.82 (s, 1H), 6.80 (d, $J = 2.0$ Hz, 1H), 6.51 (s, 1H), 6.41 (s, 1H) 3.92-3.88 (m, 2H), 2.35-2.30 (m, 2H), 1.80-1.71 (m, 2H), 1.68-1.54 (m, 2H), 1.50-1.35 (m, 2H), 1.34-1.20 (m, 2H); ^{13}C NMR (100 MHz, CDCl_3): δ 179.9, 159.1, 137.8, 136.2, 133.9, 130.5, 129.6, 129.3, 127.5, 127.4, 126.5, 115.9, 115.9, 68.6, 62.3, 34.1, 28.9, 28.9, 25.8, 24.7; IR (film): 3334 cm^{-1} , 2940, 1708, 1597, 1554, 1468; LRMS $\text{C}_{20}\text{H}_{20}\text{Cl}_4\text{O}_4$ found (ESI) 465.

Synthesis of hydroxytetrachlorodiphenylmethyl (HTPM) linkers (**8a-C2**)



(Aminomethyl)polystyrene resins (500 mg, ~1.2 mmol/g, purchased from Aldrich) were placed in a flask. DMF (3 mL), **6a** (0.72 mmol), EDCI (0.72 mmol), HOBT (0.72 mmol), and $i\text{Pr}_2\text{NEt}$ (2.4 mmol) were added. The reaction mixture was gently stirred for 12h at rt. The resins were washed with THF-water (3:1), THF, and EtOAc and dried under high vacuum for 24h. Loading determined to be 1.1 mmol/g by coupling of Fmoc- β -Ala-OH (DICI, DMAP) and subsequent release of Fmoc chromophore.

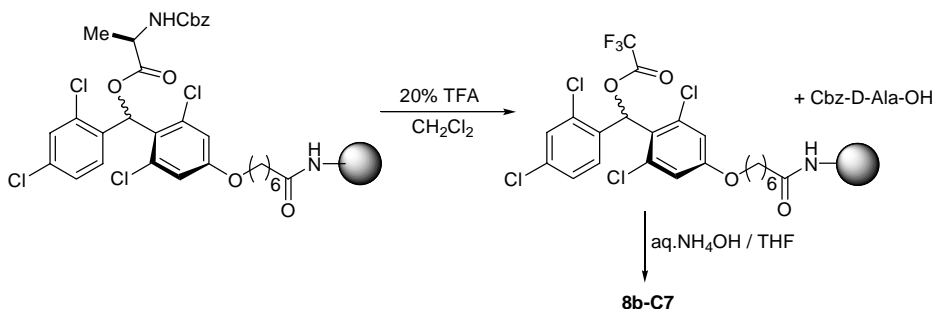
Similarly, Synphase-PS lanterns **7b** were coupled with **6a** to provide **8b-C2**. Loading determined to be 13 μmol /lantern by coupling of Fmoc- β -Ala-OH (DICI, DMAP) and subsequent release of Fmoc chromophore.



Aminomethylated Lanterns (100, ~15 μmol /lantern) were placed in a flask. DMF (mL), **6a** (0.72 mmol), EDCI (0.72 mmol), HOBT (0.72 mmol), and $i\text{Pr}_2\text{NEt}$ (2.4 mmol) were added. The reaction mixture was gently stirred for 12h at rt. The lanterns were washed with THF-water (3:1), THF, and EtOAc and dried under high vacuum for 24h. Loading determined to be 12 μmol /lantern by coupling of Fmoc- β -Ala-OH (DICI, DMAP) and subsequent release of Fmoc chromophore.

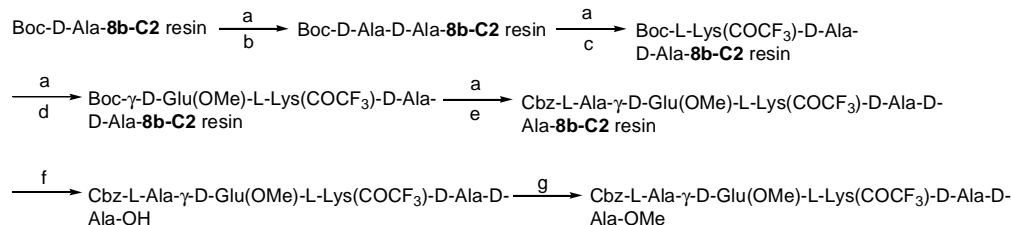
Similarly, Synphase-PS lanterns **7b** were coupled with **6a** to provide **8b-C2**. Loading determined to be 13 μmol /lantern by coupling of Fmoc- β -Ala-OH (DICI, DMAP) and subsequent release of Fmoc chromophore. Similarly, (Aminomethyl)polystyrene resins (1.0 g, ~1.2 mmol/g, purchased from Aldrich) **7b** were coupled with **6b** to provide **8b-C7**. Loading determined to be 1.0 mmol/g by loading Cbz-D-Ala-OH (DICI, DMAP) followed by cleavage (20% TFA).

Regeneration of the HTPM resin.



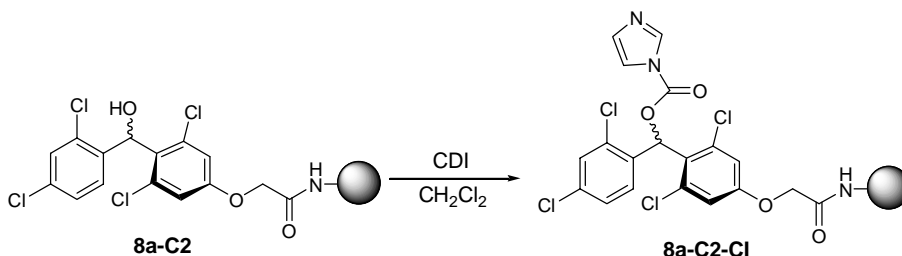
Cbz-D-Ala-HTPM resins (100 mg, 1.1 mmol/g) in THF (2 mL) was added TFA (~0.2 mL). The reaction mixture was gently stirred for 1h. The resins were washed with CH_2Cl_2 and the recovered resins in THF were subjected to hydrolysis with aq NH_4OH for 6h. The resins were washed with THF-water (3/1), THF, and EtOAc to provide the regenerated **8b-C7**. These resins exhibited the equal surface activity as the freshly prepared resins.

Synthesis of Cbz-L-Ala- γ -D-Glu(OMe)-L-Lys(COCF₃)-D-Ala-D-Ala-OMe



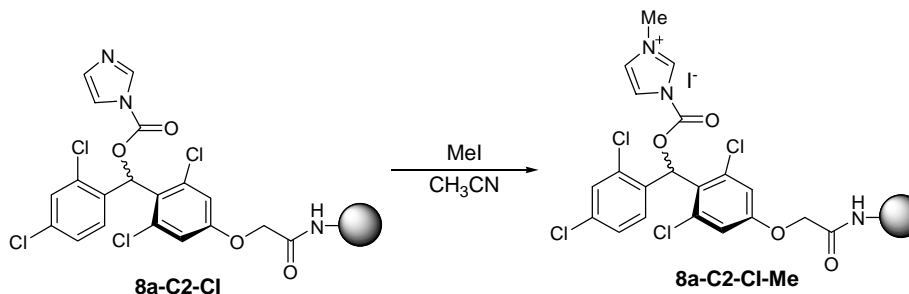
a) 20% TsOH/THF-CH₂Cl₂ (1/1), 6h.; b) Boc-D-Ala-OH, BOP, HOBt, NMM / DMF, 6h, c) Boc-L-Lys(COCF₃)-OH, BOP, HOBt, NMM / DMF, 6h.; d) Boc- γ -D-Glu(OMe)-OH, BOP, HOBt, NMM / DMF, 6h.; e) Cbz-L-Ala-OH, BOP, HOBt, NMM / DMF, 6h.; f) 20% TFA in CH₂Cl₂, 1h.; g) TMSCH₂N₂ / MeOH-CHCl₃ (1/1). Overall yield 90%. Data for Cbz-L-Ala- γ -D-Glu(OMe)-L-Lys(COCF₃)-D-Ala-D-Ala-OMe. ¹H NMR (400 MHz, DMSO): δ 9.38 (d, *J* = 2.1 Hz, 1H), 8.26 (d, *J* = 2.1 Hz, 1H), 8.17 (d, *J* = 2.1 Hz, 1H), 8.00 (d, *J* = 2.1 Hz, 1H), 7.81 (d, *J* = 2.0 Hz, 1H), 7.35 (d, *J* = 8.4 Hz, 1H), 7.19-7.31 (m, 2H), 7.17 (t, *J* = 8, 8.4 Hz, 1H), 7.00 (d, *J* = 8.4 Hz, 1H), 5.38 (s, 2H), 4.97 (t, *J* = 18, 12 Hz, 2H), 4.25-4.11 (m, 2H), 4.11-4.06 (m, 2H), 3.55 (s, 3H), 3.30 (s, 3H), 3.11-3.07 (m, 1H), 2.11-2.05 (m, 2H), 1.90-1.70 (m, 2H), 1.55-1.38 (m, 2H), 1.24 (d, *J* = 12 Hz, 2H), 1.23-1.11 (m, 4H); LRMS (FAB) C₃₂H₄₅F₃N₆O₁₁Na found 769.7.

Synthesis of 1H-imidazole-1-carbonyl-HTPM resins.



The resins **8a-C2** (100 mg, 1.1 mmol/g) in CH₂Cl₂ (2 mL) was added 1,1'-carbonyldiimidazole (0.55 mmol). After 6h at rt, the resins were washed with CH₂Cl₂ and kept under vacuum. Loading determined to be 1.1 mmol mmol/g by coupling of *N*-methylbenzylamine and subsequent release. This linker is stable over 15 days without noticeable decrease of the loading efficiency.

Synthesis of carbonylimidazolium salt-HTPM resins.



The resins **8a-C2-Cl** (100 mg, 1.1 mmol/g) in CH₃CN (2 mL) was added MeI (1.1 mmol). After 24h at rt, the resins were washed with CH₃CN and THF, and dried under high vacuum for 6h. Loading was established to be 0.72~0.77 mmol/g by the reaction of estrone (5eq) in the presence of DMAP (5eq) in CH₂Cl₂ followed by cleavage.

