N-Alkylated Oligoamide α-Helical Proteomimetics

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**General Considerations**

All reagents were obtained from Aldrich, Alfa Aesar, Acros or Fluka and used without further purification. All solvents used were HPLC grade. Dry solvents were distilled from sodium/ benzophenone (THF, Et₂O) or calcium hydride (CH₂Cl₂) immediately prior to use. N-Methylimidazole was distilled from calcium hydride. Analytical TLC was performed using 0.2 mm silica gel 60 F₂₅₄ pre-coated aluminium sheets (Merck) and visualised using UV irradiation or, in the case of amine intermediates, by staining with a ninhydrin solution. Flash column chromatography was carried out on silica gel 60 (35 to 70 micron particles, FluoroChem). Solvent ratios are described where appropriate. Solvents were removed under reduced pressure using a Buchi rotary evaporator at diaphragm pump pressure. Samples were freed of remaining traces of solvents under high vacuum. ¹H and ¹³C NMR spectra were measured on a Bruker DPX300 or a Bruker Avance 500 spectrometer using an internal deuterium lock. Chemical shifts are reported in parts per million (ppm) downfield from TMS in δ units and coupling constants are given in hertz (Hz). Coupling constants are reported to the nearest 0.1 Hz. TMS is defined as 0 ppm for ¹H NMR spectra and the centre line of the triplet of CDCl₃ was defined as 77.10 ppm for ¹³C NMR spectra. When describing ¹H NMR data the following abbreviations are be used; s = singlet, d = doublet, t = triplet, q = quartet, m = multiplet app = apparent. Melting points were determined using a Griffin D5 variable temperature apparatus and are uncorrected. Microanalyses were obtained on a Carlo Erba Elemental Analyser MOD 1106 instrument, found composition is reported to the nearest 0.05%. Infrared spectra were recorded on a Perkin-Elmer FTIR spectrometer and samples analysed as solids (unless stated). Mass spectra (HRMS) were recorded in house using a Micromass GCT Premier, using electron impact ionisation (EI) or a Bruker Daltonics micrOTOF, using electron spray ionisation (ES). LC-MS experiments were run on a Waters Micromass ZQ spectrometer, samples ionised by electrospray and analysed by a time-of-flight mass spectrometer, or a Bruker Daltronics HCTUltra™ series spectrometer, samples ionised by electrospray. All experiments were run through a C18 column on an acetonitrile/water gradient (typically 0-95% acetonitrile over 3 minutes). The synthesis of compounds 5 and 6 was described earlier. F. Campbell, *et al.*, *Chem. Commun.* 2007, 2240-2242)

**Butyl-3-oxopropylcarbamate** (Y. Hirai *et al.*, *Tetrahedron Asymmetry*, 2007, 18, 852-856)

To a stirred solution of oxalyl chloride (448 μL, 5.2 mmol, 1.8 eq.) in anhydrous CH₂Cl₂ (25 mL) at -78°C was added dimethylsulfoxide (442 μL, 5.8 mmol, 2.0 eq). The reaction was stirred for 1 hr, after which tert-butyl-3-hydroxypropylcarbamate (500 mg, 2.86 mmol in
35 mL CH₂Cl₂ was added dropwise and the reaction allowed to stir for a further hour at -78°C. Triethylamine (2.00 mL, 5 eq.) was then added and the reaction mixture allowed to warm to room temperature. After 14 h stirring the reaction was shown to be complete by TLC. The reaction mixture was washed with water (2 x 30mL) and the organic fractions combined and concentrated at the pump. Column chromatography (Stationary Phase: Silica, 75 g; Mobile Phase: CH₂Cl₂ to 4% MeOH in CH₂Cl₂) yielded target material (343 mg, 2.0 mmol, 69%) as a yellow viscous oil; Rᵣ: 0.19 (2% MeOH in CH₂Cl₂); δH (500 MHz, CDCl₃) 1.43 (s, 9H, CH₃), 2.71 (t, 2H, J = 6.3 Hz, CH₂), 3.43 (t, 2H, J = 5.9 Hz, CH₂), 4.93 (broad s, 1H, NH), 9.81 (s, 1H, CHO); δC (75 MHz, CDCl₃) 28.4, 34.0, 44.3, 79.5, 155.8, 202.

1-tert-butoxycarbonyl-3-formyl-1H-indole (Bringham et. al. Synthesis 1998, 70, 1501-1505)

Boc₂O (1.5 g, 6.9 mmol) was added to a solution of indole-3-carboxaldehyde (1 g, 6.9 mmol) and Et₃N (0.96 mL, 6.9 mmol) in CH₂Cl₂ (50 mL). The reaction mixture was stirred for 16 hours at room temperature before being quenched with water (20 mL). The mixture was then washed with saturated NaCl (3 x 50 mL) before the organic layer was dried (Na₂SO₄) and evaporated in vacuo. 18 (1.32 g, 78 %) was collected as a cream coloured solid; δH (300 MHz, CDCl₃); 1.68 (9H, s, t-Bu), 7.41 (2H, m, 2 x ArH), 8.13 (1H, d, J = 7 Hz, ArH), 8.24 (1H, s, ArH), 8.33 (1H, d, J = 7 Hz, ArH), 10.11 (1H, s, CHO); νmax/cm⁻¹; 3464, 3335, 3143, 3022, 2815, 1742, 1678

Reductive amination

To a stirred solution of primary aniline (1 eq.) and aldehyde (≥1 eq.) in methanol, under an atmosphere of nitrogen, was added borane-picoline (small excess). The reaction mixture was stirred at 35 °C for 12-36 h, until TLC indicated reaction completion. Concentration and direct purification by column chromatography gave target material which was dried under vacuum and fully characterised.

(Note: borane-picoline – a white solid with greater stability than borane-pyridine can also be used for this reaction.)

4-(Propylamino)benzoic acid 2a

4-Aminobenzoic acid (2.50 g, 18.2 mmol), propionaldehyde (1.70 mL, 23.6 mmmol) and picoline-borane complex (2.00 g, 18.7 mmol) were stirred at room temperature in methanol (25 mL) for 14h. The reaction mixture was then concentrated and triturated with the minimum amount of hot chloroform then allowed to cool. The resultant solid was recrystallised from chloroform to yield target material (2.28 g, 12.7 mmol, 70%) as a pale
cream solid; m.p. 163-164°C; Rf 0.19 (10% MeOH in CH₂Cl₂); δH (300 MHz, MeOD) 0.89 (t, 3H, J = 7.4 Hz, CH₃CH₂CH₂CH₂), 1.53 (m, 2H, CH₂CH₂CH₂CH₂), 2.98 (t, 2H, J = 7.2 Hz, CH₂CH₂CH₂CH₂), 6.46 (d, 2H, J = 8.9 Hz, ArH), 7.66 (d, 2H, J = 8.9 Hz, ArH); δC (75 MHz, MeOD) 12.2, 23.6, 46.1, 112.3, 118.1, 133.1, 155.0, 171.2; νmax/cm⁻¹ (solid state) = ~3000 (COOH), 1657 (CO); ESI-HRMS found m/z 180.1021 [M+H]⁺ C₁₀H₁₄NO₂ requires 180.1019.

4-(Isobutylamino)benzoic acid 2b

4-Aminobenzoic acid (2.00 g, 14.6 mmol), isobutyraldehyde (1.34 mL, 14.6 mmol) and picoline-borane complex (2.38 g, 25.6 mmol) in methanol (25 mL) were stirred at room temperature for 20 h. The reaction mixture was then concentrated and partitioned between EtOAc (40 mL) and aqueous acid (pH 4, 2 x 30 mL). The organic fractions were combined, dried (MgSO₄) and concentrated. Recrystallisation from EtOAc/hexane yielded target material (1.86 g, 9.6 mmol, 66%) as a pale cream solid; m.p. 110-111°C; Rf: 0.33 (10% MeOH in CH₂Cl₂); δH (300 MHz, MeOH) 0.96 (d, 6H, J = 10.1 Hz, CH₃), 1.89 (m, 1H, CH), 2.94 (d, 2H, J = 6.9 Hz, CH₂), 6.55 (d, 2H, J = 8.8 Hz, ArH), 7.74 (d, 2H, J = 8.8 Hz, ArH); δC (75 MHz, CDCl₃) 21.1, 29.4, 52.2, 112.3, 118.0, 133.1, 155.1, 171.2; νmax/cm⁻¹ (solid state) = 3423 (NH), ~3000 (COO⁻H), 1664 (CO); ESI-HRMS found m/z 194.1175 [M+H]⁺ C₁₁H₁₄NO₂ requires 194.1176.

4-(Benzylamino)benzoic acid 2c

4-Aminobenzoic acid (500 mg, 3.7 mmol), benzaldehyde (370 μL, 3.7 mmol) and picoline-borane complex (470 mg, 4.4 mmol) were stirred at room temperature in methanol (15 mL) for 16 h. The resulting precipitate was collected and the filtrate, acidified to prompt further precipitation. The solids were combined and dried under vacuum to yield target material (650 mg, 2.9 mmol, 78%) as a colourless solid; m.p. 164-166°C; Rf: 0.38 (15% EtOAc in CH₂Cl₂); δH (500 MHz, MeOD) 4.39 (s, 2H, CH₂), 6.61 (d, 2H, J = 8.5 Hz, ArH), 7.24 (m, 1H, ArH), 7.33 (m, 4H, ArH), 7.77 (d, 2H, J = 8.5 Hz, ArH); δC (75 MHz, MeOD) 48.3, 112.9, 118.9, 128.4, 128.6, 130.0, 133.1, 141.0, 154.8, 171.2; νmax/cm⁻¹ (solid state) = 3422 (NH), ~3000 (COOH), 1660 (CO); ESI-HRMS found m/z 228.1015 [M+H]⁺ C₁₄H₁₄NO₂ requires 228.1019; found m/z 250.0831 [M+Na]⁺ C₁₄H₁₃NNaO₂ requires 250.0838.

4-(Naphthalen-2-ylmethylamino)benzoic acid 2d

4-Aminobenzoic acid (350 mg, 2.6 mmol), 2-naphthaldehyde (399 mg, 2.6 mmol) and picoline-borane complex (350 mg, 3.3 mmol) in methanol (15 mL) was stirred at room temperature for 16 h. Upon acidification, material precipitated from solution and was collected by filtration. Target material was isolated (527 mg, 1.9 mmol, 75%) as a very pale cream powder; m.p. 197-199°C; Rf: 0.18 (20% Et₂O in CH₂Cl₂); δH (300 MHz, DMSO) 4.51
(d, 2H, J = 6.0 Hz, CH$_2$), 6.64 (m, 1H, ArH), 7.45 (m, 3H, ArH), 7.65 (d, 2H, J = 8.7 Hz, ArH), 7.87 (m, 1H, NH); $\delta_C$ (75 MHz, DMSO) 46.5, 111.6, 117.6, 125.6, 126.0, 126.1, 126.5, 127.9, 128.4, 131.4, 132.6, 133.3, 137.5, 152.8, 167.8; $\nu_{\text{max}}$/cm$^{-1}$ (solid state) = 3423 (NH), ~2900 (COOH), 1656 (CO); ESI-HRMS found m/z 278.1166 [M+H]$^+$ C$_{18}$H$_{16}$NO$_2$ requires 278.1176; found m/z 300.0982 [M+Na]$^+$ C$_{18}$H$_{15}$NNaO$_2$ requires 300.0995.

4-((1-tert-Butoxycarbonyl)-1H-indol-3-yl)methylamino)benzoic acid 2e
4-Aminobenzoic acid (350 mg, 2.6 mmol), tert-butyl-3-formyl-1H-indole-1-carboxylate (650 mg, 2.6 mmol) and picoline-borane complex (380 mg, 3.3 mmol) in methanol (15 mL) was stirred at room temperature for 16h. The reaction mixture was concentrated and target material recrystallised from the crude solid in the minimum amount of EtOAc/hexane. Target material was isolated (800 mg, 2.2 mmol, 84%) as a colourless solid; m.p. 186-187 °C (Found: C, 68.8; H, 6.1; N, 7.65; C$_{21}$H$_{22}$N$_2$O$_4$ requires: C, 68.84; H, 6.05; N, 7.65 %); $R_F$: 0.24 (10% MeOH in CH$_2$Cl$_2$); $\delta_H$ (300 MHz, CDCl$_3$) 1.72 (s, 9H, C(CH$_3$)$_3$), 4.54 (s, 2H, CH$_2$), 6.71 (d, 2H, J = 8.8 Hz, ArH), 7.31 (m, 1H, ArH), 7.40 (m, 1H, ArH), 7.62 (m, 2H, ArH), 8.01 (d, 2H, J = 8.7 Hz, ArH), 8.20 (d, 1H, J = 8.2 Hz, ArH); $\delta_C$ (75 MHz, CDCl$_3$) 28.2, 39.2, 84.0, 111.7, 115.5, 117.3, 117.6, 119.0, 122.8, 124.1, 124.9, 129.2, 132.4, 135.8, 149.6, 152.4, ~172; $\nu_{\text{max}}$/cm$^{-1}$ (solid state) = 3415 (NH), ~2900 (COOH), 1735, 1661 (CO); ESI-MS m/z = 367 [M+H]$^+$.

4-(3-(tert-Butoxycarbonylamino)propylamino)benzoic acid 2f
4-Aminobenzoic acid (271 mg, 2.0 mmol), tert-butyl 3-oxopropylcarbamate (343 mg, 2.0 mmol) and picoline-borane complex (255 mg, 2.4 mmol) in methanol (30 mL) was stirred at room temperature for 16 h. The reaction mixture was then concentrated and partitioned between ether (30 mL) and water (3 x 30 mL). The organic fractions were dried (MgSO$_4$), concentrated and the crude material recrystallised from methanol/water. Target material was isolated (363 mg, 1.2 mmol, 62%) as a colourless powder; m.p. 153 - 155 °C; $R_F$: 0.21 (5% MeOH in CH$_2$Cl$_2$); $\delta_H$ (500 MHz, MeOD) 1.33 (s, 9H, CH$_3$), 1.65 (m, 2H, CH$_2$), 3.05 (m, 4H, 2 x CH$_2$), 6.48 (d, 2H, J = 8.5 Hz, ArH), 7.67 (d, 2H, J = 8.6 Hz, ArH); $\delta_C$ (75 MHz, CDCl$_3$): $\delta$ = 27.7, 29.3, 37.9, 40.2, 78.9, 111.0, 117.1, 131.7, 153.4, 157.6, 169.7; $\nu_{\text{max}}$/cm$^{-1}$ (solid state) = 3318 (NH), ~2900 (COOH), 1666 (CO); ESI-MS m/z = 295 [M+H]$^+$, 239 (-C(CH$_3$)$_3$) [M+H]$^+$; ESI-HRMS found m/z 295.1649 [M+H]$^+$ C$_{19}$H$_{23}$N$_2$O$_4$ requires 295.1652; found m/z 317.1477 [M+Na]$^+$ C$_{19}$H$_{22}$N$_2$NaO$_4$ requires 317.1472.
4-(((9H-Fluoren-9-yl)methoxy)carbonyl)(propyl)amino)benzoic acid 3a
To a refluxing solution of 4-(propylamino)benzoic acid (500 mg, 2.8 mmol) in chloroform (15 mL) was added dropwise a solution of Fmoc chloride (865 mg, 3.3 mmol) in chloroform (10 mL) over the period of 1 h. The reaction mixture was then stirred at reflux for an additional 16 h. Column chromatography (Stationary Phase: Silica, 90 g; Mobile Phase: CH₂Cl₂ to 15% EtOAc in CH₂Cl₂) yielded target material (939 mg, 2.3 mmol, 88%) as an amorphous solid; (Found: C, 74.50; H, 5.85; N, 3.45; requires: C, 74.79; H, 5.77; N, 3.49); Rf 0.36 (15% EtOAc in CH₂Cl₂); δH (300 MHz, CDCl₃) 0.81 (t, 3H, J = 7.4 Hz, CH₃CH₂CH₂), 1.46 (m, 2H, CH₂CH₂CH₂), 3.58 (t, 2H, J = 7.4 Hz, CH₃CH₂CH₂), 4.13 (t, 1H, J = 6.0 Hz, CHCH₂), 4.52 (d, 2H, J = 6.1 Hz, CHCH₂), 7.16-7.39 (m, 8H, ArH), 7.71 (d, 2H, J = 7.5 Hz, ArH), 8.04 (d, 2H, J = 8.6 Hz, ArH); δc (75 MHz, CDCl₃) 11.0, 21.5, 47.2, 51.7, 64.2, 67.3, 119.9, 120.1, 124.8, 126.8, 127.0, 127.7, 131.0, 141.4, 143.7, 146.8, 154.9, 170.9; vmax/cm⁻¹ (solid state) = ~3000 (COOH), 1720, 1673 (CO); ESI-MS m/z 402 [M+H]⁺.

4-(((9H-Fluoren-9-yl)methoxy)carbonyl)(isobutyl)amino)benzoic acid 3b
To a refluxing solution of 4-(isobutylamino)benzoic acid (1.35 g, 7.0 mmol) in chloroform (25 mL) was added dropwise a solution of Fmoc chloride (4.50 g, 17.4 mmol) in chloroform (25 mL) over the period of 1 h. The reaction mixture was then stirred at reflux for a further 16 h. Column chromatography (Stationary Phase: Silica, 150 g; Mobile Phase: CH₂Cl₂ to 20% Et₂O in CH₂Cl₂) yielded target material (2.34 g, 5.6 mmol, 81%) as an amorphous solid; Rf: 0.17 (20% Et₂O in CH₂Cl₂); δH (300 MHz, CDCl₃) 0.83 (d, 6H, J = 6.6 Hz, CH₃), 1.71 (m, 1H, CH), 3.52 (d, 2H, J = 7.5 Hz, CH₂), 4.17 (t, 1H, J = 5.8 Hz, CHCH₂), 4.60 (d, 2H, J = 5.9 Hz, CHCH₂), 7.19 (d, 2H, J = 8.5 Hz, ArH), 7.25-7.44 (m, 6H, ArH), 7.76 (d, 2H, J = 7.5 Hz, ArH), 8.09 (d, 2H, J = 8.5 Hz, ArH); δc (75 MHz, CDCl₃) 20.2, 27.6, 47.7, 57.4, 67.6, 120.3, 125.2, 127.4, 128.1, 131.4, 141.8, 144.1, 147.3, 155.6, 171.7; vmax/cm⁻¹ (solid state) = ~3000 (COOH), 1690 (CO); ESI-MS m/z = 416 [M+H]⁺; ESI-HRMS found m/z 416.1853 [M+H]⁺ requires 416.1856; found m/z 438.1680 [M+Na]⁺ C₂₆H₂₅NNaO₂ requires 438.1676.

4-(((9H-Fluoren-9-yl)methoxy)carbonyl)(benzyl)amino)benzoic acid 3c
To a refluxing solution of 4-(benzylamino)benzoic acid (500 mg, 2.2 mmol) in chloroform (15 mL) was added dropwise a solution of Fmoc chloride (680 mg, 2.6 mmol) in chloroform (10 mL) over the period of 1 h. The reaction mixture was then stirred at reflux for an additional 16 h. Column chromatography (Stationary Phase: Silica, 75 g; Mobile Phase: CH₂Cl₂ to 15% EtOAc in CH₂Cl₂) yielded target material (857 mg, 1.9 mmol, 86%) as an amorphous solid; (Found: C, 77.35; H, 5.15; N, 3.00; requires: C, 77.49; H, 5.16; N, 3.12); Rf
0.3 (15% EtOAc in CH$_2$Cl$_2$); $\delta_H$ (300 MHz, CDCl$_3$) 4.17 (t, 1H, $J = 6.0$ Hz, CHCH$_2$), 4.62 (d, 2H, $J = 6.1$ Hz, CHCH$_2$), 4.90 (s, 2H, CH$_2$), 7.15 (m, 4H, ArH), 7.25 (m, 2H, ArH), 7.32 (m, 5H, ArH), 7.41 (m, 2H, ArH), 7.75 (d, 2H, $J = 7.6$ Hz, ArH), 8.02 (d, 2H, $J = 8.4$ Hz, ArH); $\delta_C$ (75 MHz, CDCl$_3$) 47.6, 45.3, 45.1, 44.7, 44.1, 33.5, 33.3, 31.0, 135.5, 141.3, 143.5, 146.2, 149.5, 150.7, 1688 (CO); ESI-MS m/z 522 [M+Na]$^+$, 472 [M+Na]$^+$.  

4-(((9H-Fluoren-9-yl)methoxy)carbonyl)(naphthalen-2-ylmethyl)amino) benzoic acid 3d

To a refluxing solution of 4-(naphthalen-2-ylmethylamino)benzoic acid (500 mg, 1.8 mmol) in chloroform (25 mL) was added dropwise a solution of Fmoc chloride (840 mg, 3.1 mmol) in chloroform (25 mL) over a period of 1 h. The reaction mixture was then stirred at reflux for a further 16 h. Column chromatography (Stationary Phase: Silica, 100 g; Mobile Phase: CH$_2$Cl$_2$ to 20% Et$_2$O in CH$_2$Cl$_2$) yielded target material (678 mg, 1.36 mmol, 75%) as an amorphous solid; (Found: C, 79.2, H, 5.05; N, 2.7; C$_{33}$H$_{25}$NO$_4$ requires: C, 79.34; H, 5.04; N, 2.80 %); $R_f$: 0.09 (20% Et$_2$O in CH$_2$Cl$_2$); $\delta_H$ (300 MHz, CDCl$_3$) 4.12 (t, 1H, $J = 6.0$ Hz, CHCH$_2$), 4.58 (d, 2H, $J = 6.0$ Hz, CHCH$_2$), 5.00 (s, 2H, CH$_2$), 7.11-7.16 (m, 4H, ArH), 7.25-7.31 (m, 4H, ArH), 7.43-7.47 (m, 2H, ArH), 7.53 (s, 1H, ArH), 7.65-7.77 (m, 5H, ArH), 7.95 (d, 2H, $J = 8.4$ Hz, ArH); $\nu_{max}$/cm$^{-1}$ (solid state) = ~3000 broad (COOH), 1688 (CO); ESI-MS m/z = 522 [M+Na]$^+$, 500 [M+H]$^+$. 

4-(((9H-Fluoren-9-yl)methoxy)carbonyl)((1-tert-butoxycarbonyl)-1H-indol-3-yl)methyl)amino) benzoic acid 3e

To a refluxing solution of 4-((1-tert-butoxycarbonyl)-1H-indol-3-yl)methylamino)benzoic acid (700 mg, 1.9 mmol) in chloroform (15 mL) was added dropwise a solution of Fmoc chloride (680 mg, 2.6 mmol) in chloroform (25 mL) over a period of 1 h. The reaction mixture was then stirred at reflux for a further 14 h. Column chromatography (Stationary Phase: Silica, 80 g; Mobile Phase: CH$_2$Cl$_2$ to 15% EtOAc in CH$_2$Cl$_2$) yielded target material (855 mg, 1.5 mmol, 76%) as an amorphous solid; m.p. 99-101 °C (Found: C, 73.05; H, 5.95; N, 4.5; C$_{36}$H$_{32}$N$_2$O$_6$ requires: C, 73.45; H, 5.95; N, 4.76 %); $R_f$: 0.53 (15% EtOAc in CH$_2$Cl$_2$); $\delta_H$ (500 MHz, CDCl$_3$) 1.62 (s, 9H, C(CH$_3$)$_2$), 4.11 (broad t, 1H, CHCH$_2$), 4.55 (d, 2H, $J = 6.2$ Hz, CHCH$_2$), 4.96 (s, 2H, CH$_2$), 7.10-7.33 (m, 11H, ArH), 7.40 (d, 1H, $J = 7.6$ Hz, ArH), 7.63 (d, 2H, $J = 7.5$ Hz, ArH), 7.97 (d, 2H, $J = 8.4$ Hz, ArH), 8.1 (broad s, 1H, ArH); $\delta_C$ (75 MHz, CDCl$_3$) 28.2, 45.3, 47.1, 67.7, 83.9, 115.3, 116.5, 119.3, 119.9, 122.8, 124.7, 124.7, 126.9, 127.3, 127.6, 128.0, 129.2, 131.0, 135.5, 141.3, 143.5, 146.2, 149.5,
153.4, 155.0, 171.0; \nu_{\text{max}}/\text{cm}^{-1} \text{ (solid state)} = \sim 3000 \text{ (COOH), 1693 (CO)}; \text{ESI-MS } m/z = 611 [\text{M+Na}]^+, 589 [\text{M+H}]^+.

4-(((9H-Fluoren-9-yl)methoxy)carbonyl)(3-(tert-butoxycarbonylamino)propyl)amino)benzoic acid 3f

To a refluxing solution of 4-(3-(tert-butoxycarbonylamino)propylamino)benzoic acid (700 mg, 2.4 mmol) and sodium hydrogencarbonate (1.00 g, excess) in chloroform (40 mL), was added dropwise a solution of Fmoc chloride (925 mg, 3.6 mmol, 1.5 eq.) in chloroform (20 mL) over 2 h. Excess sodium hydrogencarbonate was filtered off before column chromatography (Stationary Phase: Silica, 200 g; Mobile Phase: \text{CH}_2\text{Cl}_2 \text{ to 20\% Et}_2\text{O in CH}_2\text{Cl}_2) yielded pure target material (455 mg, 0.9 mmol, 38\%) as an amorphous solid; \text{RF:} 0.21 (20\% \text{Et}_2\text{O in CH}_2\text{Cl}_2); \delta_H (300 \text{ MHz, CDCl}_3) 1.45 \text{ (s, 9H, CH}_3\text{)}, 1.56 \text{ (m, 2H, CH}_2\text{)}, 3.01 \text{ (m, 2H, CH}_2\text{)}, 3.66 \text{ (t, 2H, } J = 7.1 \text{ Hz, CH}_2\text{)}, 4.10 \text{ (t, 1H, } J = 5.8 \text{ Hz, CH}), 4.56 \text{ (d, 2H, } J = 5.8 \text{ Hz, CH}_2\text{)}, 4.85 \text{ (broad s, 1H, NH)}, 7.08 \text{ (d, 2H, } J = 8.1 \text{ Hz, ArH}), 7.20 - 7.39 \text{ (m, 6H, ArH)}, 7.71 \text{ (d, 2H, } J = 7.6 \text{ Hz, ArH)}, 7.99 \text{ (d, 2H, } J = 8.3 \text{ Hz, ArH}); \delta_C (75 \text{ MHz, CDCl}_3) 28.4, 28.5, 47.3, 47.4, 67.2, 79.3, 119.9, 124.8, 127.0, 127.5, 127.7, 131.1, 141.4, 143.6, 146.1, 155.2, 156.0, 170.1; \nu_{\text{max}}/\text{cm}^{-1} \text{ (solid state)} = \sim 2900 \text{ (broad COOH), 1692 (CO)}; \text{ESI-MS } m/z = 539 \text{ [M+Na]}^+, 417 \text{ (} - \text{CO}_2\text{C(CH}_3\text{))}; \text{ESI-HRMS found } m/z 539.2145 \text{ [M+Na]}^+ \text{C}_{30}\text{H}_{32}\text{N}_2\text{NaO}_6 \text{ requires } 539.2153.

4-(2-(((9H-Fluoren-9-yl)methoxy)carbonylamino)-N-benzylacetamido)benzoic acid 4c

To a refluxing solution of 4-(benzylamino)benzoic acid (800 mg, 3.5 mmol) in chloroform (35 mL) was added dropwise a solution of 2-(((9H-fluoren-9-yl)methoxy)carbonylamino)acetic acid (1.65 g, 5.3 mmol, 1.5 eq.) in chloroform (30 mL) over a period of 1 h. The reaction was then stirred at reflux for 14 h. Column chromatography (Stationary Phase: Silica, 300 g; Mobile Phase: \text{CH}_2\text{Cl}_2 \text{ to 20\% Et}_2\text{O in CH}_2\text{Cl}_2 \text{ to 12\% MeOH in CH}_2\text{Cl}_2) yielded target material (1.43 g, 2.8 mmol, 81\%) as an amorphous solid; \text{RF:} 0.23 (20\% \text{Et}_2\text{O in CH}_2\text{Cl}_2); \delta_H (300 \text{ MHz, CHCl}_3) 3.87 \text{ (broad s, 2H, CH}_2\text{)}, 4.20 \text{ (t, 1H, } J = 7.1 \text{ Hz, CH}), 4.35 \text{ (d, 2H, } J = 7.2 \text{ Hz, CH}_2\text{)}, 4.93 \text{ (s, 2H, CH}_2\text{)}, 6.03 \text{ (broad t, 1H, NH)}, 7.10 \text{ (d, 2H, } J = 8.0 \text{ Hz, ArH}), 7.15-7.42 \text{ (m, 9H, ArH)}, 7.59 \text{ (d, 2H, } J = 7.7 \text{ Hz, ArH}), 7.76 \text{ (d, 2H, } J = 7.8 \text{ Hz, ArH}), 8.01 \text{ (d, 2H, } J = 8.2 \text{ Hz, ArH}); \delta_C (75 \text{ MHz, CHCl}_3) 44.0, 47.4, 53.9, 67.9, 72.0, 125.5, 127.5, 128.2, 128.4, 128.8, 128.9, 129.1, 129.3, 132.2, 136.3, 141.7, 144.1, 157.0, 168.3, 169.0; \nu_{\text{max}}/\text{cm}^{-1} \text{ (solid state)} = 3312 \text{ (broad) (COOH), 1710, 1667, 1602 (CO)}; \text{ESI-HRMS found } m/z 507.1925 \text{ [M+H]}^+ \text{C}_{31}\text{H}_{27}\text{N}_2\text{O}_5 \text{ requires } 507.1914, \text{found } m/z 529.1751 \text{ [M+Na]}^+ \text{C}_{31}\text{H}_{26}\text{N}_2\text{NaO}_5 \text{ requires } 529.1734.
4-(2-((9H-Fluoren-9-yl)methoxy)carbonylamino)-N-(naphthalen-2-ylmethyl)acetamido)benzoic acid 4d

To a refluxing solution of 4-(naphthalen-2-ylmethylamino)benzoic acid (650 mg, 2.3 mmol) in chloroform (25 mL) was added dropwise a solution of 2-(((9H-fluoren-9-yl)methoxy)carbonylamino)acetic acid (1.10 g, 3.5 mmol, 1.5 eq.) in chloroform (20 mL) over a period of 1 h. The reaction was then stirred at reflux for 14 h. Column chromatography (Stationary Phase: Silica, 200 g; Mobile Phase: CH₂Cl₂ to 20% Et₂O in CH₂Cl₂ to 12% MeOH in CH₂Cl₂) yielded target material (835 mg, 1.5 mmol, 65%) as a colourless solid; Rf: 0.26 (20% Et₂O in CH₂Cl₂); δH (500 MHz, DMSO) 3.65 (broad s, 2H, CH₂), 4.24 (t, 1H, J = 6.7 Hz, CH), 4.30 (d, 2H, J = 6.7 Hz, CH₂), 5.11 (s, 2H, CH₂), 7.34 (m, 2H, ArH), 7.39-7.44 (m, 4H, ArH), 7.49 (m, 2H, ArH), 7.60 (m, 1H, ArH), 7.71 (s, 1H, ArH), 7.73 (d, 2H, J = 7.7 Hz, ArH), 7.84-7.93 (m, 7H, ArH); v max/cm⁻¹ (solid state) = 3416 (NH), ~3100 (broad COOH), 1724, 1628, 1600 (CO); ESI-MS: m/z 557.2071; found m/z 579.1912 [M+Na]⁺ C₃₅H₂₈N₂O₅ requires 579.1890.

Solid Phase Synthesis Characterisation

2-(4-(N-Isobutyl-4-(N-isobutyl-4-(isobutyramido)benzamido)benzamido)benzamido)acetamido

Purified by column chromatography (Stationary Phase: Silica, 15 g; Mobile Phase: CH₂Cl₂ to 15% MeOH in CH₂Cl₂). Precipitated from CHCl₃/Hexane. Isolated yield: 4 mg. δH (500 MHz, MeOD) 0.78 (d, 6H, J = 6.7 Hz, (CH₃)₂), 0.86 (d, 12H, J = 6.8 Hz, 2 x (CH₃)₂), 1.76 (m, 3H, 3 x CH), 2.79 (d, 2H, J = 6.8 Hz, CH₂), 3.61 (d, 2H, J = 6.9 Hz, CH₂), 3.74 (d, 2H, J = 7.0 Hz, CH₂), 3.99 (s, 2H, CH₂), 6.21 (d, 2H, J = 9.6 Hz, ArH), 6.83 (d, 2H, J = 8.5 Hz, ArH), 6.88 (d, 2H, J = 8.8 Hz, ArH), 7.12 (m, 4H, ArH), 7.68 (d, 2H, J = 8.7 Hz, ArH); v max/cm⁻¹ (solid state) = 3359 (COOH), 1734, 1635, 1602 (CO); ESI-MS: m/z 601.3380 [M+H]⁺ C₃₅H₄₅N₄O₅ requires 601.3384.

2-(4-(4-(Benzylamino)-N-isobutylbenzamido)-N-isobutylbenzamido)benzamido

Purified by column chromatography (Stationary Phase: Silica, 15 g; Mobile Phase: CH₂Cl₂ to 15% MeOH in CH₂Cl₂). Precipitated from CHCl₃/Hexane. Isolated yield: 5 mg. δH (500 MHz, MeOD) 0.77 (d, 6H, J = 6.7 Hz, (CH₃)₂), 0.87 (d, 6H, J = 6.7 Hz, (CH₃)₂), 1.68 (m, 1H, CH), 1.78 (m, 1H, CH), 3.61 (d, 2H, J = 7.6 Hz, CH₂), 3.75 (d, 2H, J = 6.9 Hz, CH₂), 3.94 (s, 2H, CH₂), 4.23 (s, 2H, CH₂), 6.25 (d, 2H, J = 8.6 Hz, ArH), 6.81 (d, 2H, J = 8.5 Hz, ArH), 6.87 (d, 2H, J = 8.4 Hz, ArH), 7.10 (m, 4H, ArH), 7.20 (m, 4H, ArH), 7.65 (d, 2H, J = 8.4
Hz, ArH); $\nu_{\text{max}}/\text{cm}^{-1}$ (solid state) = 3347 (COOH), 1739, 1635, 1604 (CO); ESI-HRMS found $m/z$ 635.3208 [M+H]$^+$ C$_{38}$H$_{43}$N$_4$O$_5$ requires 635.3228.

2-(4-(N-Benzyl-4-(isobutylamino)benzamido)-N-isobutylbenzamido)benzamido)acetic acid 10

Purified by column chromatography (Stationary Phase: Silica, 15 g; Mobile Phase: CH$_2$Cl$_2$ to 15% MeOH in CH$_2$Cl$_2$). Precipitated from CHCl$_3$/Hexane. Isolated yield: 3 mg. $\delta_H$ (500 MHz, MeOD) 6.84 (d, 6H, $J = 5.7$ Hz, (CH$_3$)$_2$), 0.87 (d, 6H, $J = 6.8$ Hz, (CH$_3$)$_2$), 1.76 (m, 2H, 2 x CH), 2.79 (d, 2H, $J = 6.8$ Hz, CH$_2$), 3.71 (d, 2H, $J = 7.6$ Hz, CH$_2$), 4.00 (s, 2H, CH$_2$), 4.96 (s, 2H, CH$_2$), 6.22 (d, 2H, $J = 8.6$ Hz, ArH), 6.74 (d, 2H, $J = 8.6$ Hz, ArH), 6.91 (d, 2H, $J = 9.3$ Hz, ArH), 7.02 (d, 2H, $J = 7.7$ Hz, ArH), 7.11 (m, 7H, ArH), 7.66 (d, 2H, $J = 8.4$ Hz, ArH); $\nu_{\text{max}}/\text{cm}^{-1}$ (solid state) = 3359 (COOH), 1736, 1635, 1603 (CO); ESI-HRMS found $m/z$ 635.3235 [M+H]$^+$ C$_{38}$H$_{43}$N$_4$O$_5$ requires 635.3228.

2-(4-(N-Benzyl-4-(benzylamino)benzamido)-N-isobutylbenzamido)benzamido)acetic acid 11

Purified by column chromatography (Stationary Phase: Silica, 15 g; Mobile Phase: CH$_2$Cl$_2$ to 15% MeOH in CH$_2$Cl$_2$). Precipitated from CHCl$_3$/Hexane. Isolated yield: 4 mg. $\delta_H$ (500 MHz, MeOD) 0.84 (d, 6H, $J = 7.0$ Hz, (CH$_3$)$_2$), 1.75 (m, 1H, CH), 3.72 (d, 2H, $J = 6.8$ Hz, CH$_2$), 3.96 (s, 2H, CH$_2$), 4.23 (s, 2H, CH$_2$), 4.95 (s, 2H, CH$_2$), 6.26 (d, 2H, $J = 8.6$ Hz, ArH), 6.74 (d, 2H, $J = 8.3$ Hz, ArH), 6.90 (d, 2H, $J = 8.8$ Hz, ArH), 7.10 (m, 10H, ArH), 7.20 (m, 4H, ArH), 7.63 (d, 2H, $J = 8.7$ Hz, ArH); $\nu_{\text{max}}/\text{cm}^{-1}$ (solid state) = 3357 (COOH), 1735, 1632, 1602 (CO); ESI-HRMS found $m/z$ 669.3067 [M+H]$^+$ C$_{41}$H$_{41}$N$_4$O$_5$ requires 669.3071.

2-(4-(N-Benzyl-4-(N-benzyl-4-(isobutylamino)benzamido)benzamido)benzamido)benzamido)acetic acid 12

Purified by column chromatography (Stationary Phase: Silica, 15 g; Mobile Phase: CH$_2$Cl$_2$ to 15% MeOH in CH$_2$Cl$_2$). Precipitated from CHCl$_3$/Hexane. Isolated yield: 6 mg. $\delta_H$ (500 MHz, MeOD) 0.86 (d, 6H, $J = 7.0$ Hz, (CH$_3$)$_2$), 1.76 (m, 1H, CH), 2.77 (d, 2H, $J = 6.8$ Hz, CH$_2$), 3.96 (s, 2H, CH$_2$), 4.96 (s, 2H, CH$_2$), 5.05 (s, 2H, CH$_2$), 6.21 (d, 2H, $J = 9.3$ Hz, ArH), 6.77 (d, 2H, $J = 8.4$ Hz, ArH), 6.92 (m, 4H, ArH), 7.12 (m, 12H, ArH), 7.57 (d, 2H, $J = 8.5$ Hz, ArH); $\nu_{\text{max}}/\text{cm}^{-1}$ (solid state) = 3356 (COOH), 1735, 1640, 1602 (CO); ESI-HRMS found $m/z$ 669.3067 [M+H]$^+$ C$_{41}$H$_{41}$N$_4$O$_5$ requires 669.3054.

2-(4-(N-Benzyl-4-(N-isobutyl-4-(isobutylamino)benzamido)benzamido)benzamido)benzamido)acetic acid 13

Purified by column chromatography (Stationary Phase: Silica, 15 g; Mobile Phase: CH$_2$Cl$_2$ to 15% MeOH in CH$_2$Cl$_2$). Precipitated from CHCl$_3$/Hexane. Isolated yield: 5 mg. $\delta_H$ (500 MHz, MeOD) 0.87 (d, 6H, $J = 6.8$ Hz, (CH$_3$)$_2$), 1.78 (d, 2H, $J = 6.8$ Hz, CH$_2$), 3.97 (s, 2H, CH$_2$), 4.96 (s, 2H, CH$_2$), 6.21 (d, 2H, $J = 9.1$ Hz, ArH), 6.84 (d, 2H, $J = 6.8$ Hz, ArH), 7.02 (d, 2H, $J = 7.6$ Hz, ArH), 7.12 (m, 10H, ArH), 7.54 (d, 2H, $J = 8.5$ Hz, ArH); $\nu_{\text{max}}/\text{cm}^{-1}$ (solid state) = 3358 (COOH), 1735, 1640, 1602 (CO); ESI-HRMS found $m/z$ 669.3067 [M+H]$^+$ C$_{41}$H$_{41}$N$_4$O$_5$ requires 669.3054.
MHZ, MeOD) 0.78 (d, 6H, J = 6.8 Hz, (CH₃)₂), 0.86 (d, 6H, J = 6.8 Hz, (CH₃)₂), 1.74 (m, 2H, 2 x CH) 2.77 (d, 2H, J = 6.8 Hz, CH₂), 3.61 (d, 2H, J = 7.7 Hz, CH₂), 3.95 (s, 2H, CH₂), 5.08 (s, 2H, CH₂), 6.19 (d, 2H, J = 8.5 Hz, ArH), 6.82 (d, 2H, J = 8.5 Hz, ArH), 6.89 (d, 2H, J = 8.4 Hz, ArH), 6.98 (d, 2H, J = 8.5 Hz, ArH), 7.16 (m, 7H, ArH), 7.59 (d, 2H, J = 8.6 Hz, ArH); νmax/cm⁻¹ (solid state) = 3359 (COOH), 1736, 1640, 1602 (CO)

Purified by column chromatography (Stationary Phase: Silica, 15 g; Mobile Phase: CH₂Cl₂ to 15% MeOH in CH₂Cl₂). Precipitated from CHCl₃/Hexane. Isolated yield: 5 mg. δH (500 MHz, MeOD) 0.77 (d, 6H, J = 6.7 Hz, (CH₃)₂), 1.68 (m, 1H, CH), 3.61 (d, 2H, J = 7.4 Hz, CH₂), 3.90 (s, 2H, CH₂), 4.21 (s, 2H, CH₂), 5.08 (s, 2H, CH₂), 6.23 (d, 2H, J = 8.6 Hz, ArH), 6.79 (d, 2H, J = 9.4 Hz, ArH), 6.87 (d, 2H, J = 8.4 Hz, ArH), 6.93 (d, 2H, J = 8.7 Hz, ArH), 7.19 (m, 12H, ArH), 7.55 (d, 2H, J = 8.6 Hz, ArH); νmax/cm⁻¹ (solid state) = 3357 (COOH), 1734, 1635, 1599 (CO); ESI-HRMS found m/z 669.3044 [M+H]+ C₄₁H₄₁N₄O₅ requires 669.3071.

2-(4-(4-(Benzylamino)-N-(naphthalen-2-ylmethyl)benzamido)-N-isobutylbenzamido)benzamido)benzamido)acetic acid 14

Purified by column chromatography (Stationary Phase: Silica, 15 g; Mobile Phase: CH₂Cl₂ to 15% MeOH in CH₂Cl₂). Precipitated from EtOAc/Hexane. Isolated yield: 7 mg. δH (500 MHz, MeOD) 0.80 (d, 6H, J = 6.9 Hz, (CH₃)₂), 1.70 (m, 1H, CH), 3.67 (d, 2H, J = 6.6 Hz, CH₂), 3.95 (s, 2H, CH₂), 4.22 (s, 2H, CH₂), 5.11 (s, 2H, CH₂), 6.23 (d, 2H, J = 8.4 Hz, ArH), 6.76 (d, 2H, J = 7.9 Hz, ArH), 6.91 (d, 2H, J = 8.5 Hz, ArH), 6.99 (d, 2H, J = 8.4 Hz, ArH), 7.01 (d, 2H, J = 8.6 Hz, ArH), 7.09 (m, 1H, ArH), 7.17-7.25 (m, 1H, ArH), 7.32 (m, 2H, ArH), 7.51 (s, 1H, ArH), 7.58-7.65 (m, 5H, ArH); νmax/cm⁻¹ (solid state) = 3348 (COOH), 1736, 1640, 1600 (CO); ESI-HRMS found m/z 719.3199 [M+H]+ C₄₅H₄₃N₄O₅ requires 719.3228.

2-(4-(4-(4-(2-Amino-N-(naphthalen-2-ylmethyl)acetamido)-N-(naphthalen-2-ylmethyl)benzamido)-N-isobutylbenzamido)benzamido)acetic acid 16

Precipitated from MeOH/Et₂O. Isolated yield: 10 mg. δH (500 MHz, MeOD) 0.83 (d, 6H, J = 5.9 Hz, (CH₃)₂), 1.73 (m, 1H, CH), 3.39 (s, 2H, CH₂), 3.63 (d, 2H, J = 6.6 Hz, CH₂), 3.93 (s, 2H, CH₂), 5.02 (s, 2H, CH₂), 5.10 (s, 2H, CH₂), 6.66 (m, 4H, ArH), 6.82 (d, 2H, J = 7.4 Hz, ArH), 6.96 (d, 2H, J = 8.7 Hz, ArH), 7.10 (d, 2H, J = 8.7 Hz, ArH), 7.20 (d, 1H, J = 8.5 Hz, ArH), 7.28 (d, 1H, J = 8.6 Hz, ArH), 7.35 (m, 6H, ArH), 7.44 (s, 1H, ArH), 7.47 (s, 1H,
ArH), 7.63-7.69 (m, 6H, ArH); \( \nu_{\text{max}}/\text{cm}^{-1} \) (solid state) = ~3200 (broad) (COOH), 1637, 1603 (CO); ESI-HRMS found \( m/z \) 824.3544 [M+H]\(^+\) C\(_{51}\)H\(_{46}\)N\(_3\)O\(_6\) requires 824.3443.

2-(4-(4-(2-Amino-N-benzylacetamido)-N-isobutylbenzamido)-N-isobutylbenzamido)benzamido)acetic acid 17

Precipitated from MeOH/Et\(_2\)O. Isolated yield: 8 mg. \( \delta_\text{H} \) (500 MHz, MeOD) 0.77 (d, 6H, \( J = 5.8 \) Hz, (CH\(_3\))\(_2\)), 0.87 (d, 6H, \( J = 7.1 \) Hz, (CH\(_3\))\(_2\)), 1.64 (m, 1H, CH), 1.78 (m, 1H, CH), 3.33 (s, 2H, CH\(_2\)), 3.64 (d, 2H, \( J = 7.4 \) Hz, CH\(_2\)), 3.75 (d, 2H, \( J = 7.2 \) Hz, CH\(_2\)), 3.91 (s, 2H, CH\(_2\)), 4.83 (s, 2H, CH\(_2\)), 6.85 (d, 2H, \( J = 7.5 \) Hz, ArH), 6.90 (d, 2H, \( J = 8.0 \) Hz, ArH), 7.00 (d, 2H, \( J = 8.3 \) Hz, ArH), 7.05 (d, 5H, \( J = 8.0 \) Hz, ArH), 7.15 (m, 4H, ArH), 7.56 (d, 2H, \( J = 8.4 \) Hz, ArH); \( \nu_{\text{max}}/\text{cm}^{-1} \) (solid state) = ~3200 (broad) (COOH), 1645, 1599 (CO); ESI-HRMS found \( m/z \) 692.3413 [M+H]\(^+\) C\(_{40}\)H\(_{44}\)N\(_3\)O\(_6\) requires 692.3443.

2-(4-(4-(2-Amino-N-benzylacetamido)-N-propylbenzamido)-N-isobutylbenzamido)benzamido)acetic acid 18

Precipitated from MeOH/Et\(_2\)O. Isolated yield: 7 mg. \( \delta_\text{H} \) (500 MHz, MeOD) 0.79 (t, 3H, \( J = 7.6 \) Hz, CH\(_2\)CH\(_2\)CH\(_3\)\), 0.88 (d, 6H, \( J = 7.1 \) Hz, (CH\(_3\))\(_2\)), 1.44 (m, 2H, CH\(_2\)CH\(_2\)CH\(_3\)\), 1.79 (m, 1H, CH), 3.35 (s, 2H, CH\(_2\)), 3.73 (t, 2H, \( J = 7.0 \) Hz, CH\(_2\)CH\(_2\)CH\(_3\)\), 3.76 (d, 2H, \( J = 7.8 \) Hz, CH\(_2\)), 3.92 (s, 2H, CH\(_2\)), 4.84 (s, 2H, CH\(_2\)), 6.85 (d, 2H, \( J = 8.3 \) Hz, ArH), 6.92 (d, 2H, \( J = 8.0 \) Hz, ArH), 7.02 (d, 2H, \( J = 8.3 \) Hz, ArH), 7.08 (m, 5H, ArH), 7.17 (m, 4H, ArH), 7.58 (d, 2H, \( J = 7.8 \) Hz, ArH); \( \nu_{\text{max}}/\text{cm}^{-1} \) (solid state) = ~3200 (broad) (COOH), 1645, 1599 (CO); ESI-HRMS found \( m/z \) 678.3286 [M+H]\(^+\) C\(_{39}\)H\(_{44}\)N\(_3\)O\(_6\) requires 678.3286.

2-(4-(4-(2-Amino-N-benzylacetamido)-N-(3-aminopropyl)benzamido)-N-benzylbenzamido)benzamido)acetic acid 19

Precipitated from MeOH/Et\(_2\)O. Isolated yield: 12 mg. \( \delta_\text{H} \) (500 MHz, MeOD) 1.70 (m, 2H, \( J = 7.8 \) Hz, CH\(_2\)), 2.81 (t, 2H, \( J = 6.9 \) Hz, CH\(_2\)), ~3.38 (under solvent peak) (s, 2H, CH\(_2\)), 3.85 (t, 2H, \( J = 7.9 \) Hz, CH\(_2\)), 3.88 (s, 2H, CH\(_2\)), 5.01 (s, 2H, CH\(_2\)), 5.05 (s, 2H, CH\(_2\)), 6.61 (d, 2H, \( J = 7.4 \) Hz, ArH), 6.79 (d, 2H, \( J = 7.6 \) Hz, ArH), 6.94 (d, 4H, \( J = 8.1 \) Hz, ArH), 7.04 (d, 2H, \( J = 8.6 \) Hz, ArH), 7.22-7.26 (m, 6H, ArH), 7.32-7.36 (m, 4H, ArH), 7.40 (s, 1H, ArH), 7.55 (d, 1H, \( J = 7.7 \) Hz, ArH), 7.62 (d, 2H, \( J = 8.8 \) Hz, ArH); \( \nu_{\text{max}}/\text{cm}^{-1} \) (solid state) = ~3200 (broad) (COOH), 1632, 1600 (CO); ESI-HRMS found \( m/z \) 389.1749 [M+2H]\(^{2+}\) C\(_{46}\)H\(_{46}\)N\(_6\)O\(_6\) requires 389.1734.

2-(4-(4-(2-Amino-N-benzylacetamido)-N-benzylbenzamido)benzamido)acetic acid 20

Precipitated from MeOH/Et\(_2\)O. Isolated yield: 16 mg. \( \delta_\text{H} \) (500 MHz, MeOD) 3.35 (s, 2H, CH\(_2\)), 3.90 (s, 2H, CH\(_2\)), 4.83 (s, 2H, CH\(_2\)), 4.98 (s, 2H, CH\(_2\)), 5.06 (s, 2H, CH\(_2\)), 6.71 (d, 2H,
J = 7.8 Hz, ArH), 6.80 (d, 2H, J = 8.8 Hz, ArH), 6.91 (d, 2H, J = 8.8 Hz, ArH), 6.98-7.19 (m, 19H, ArH), 7.45 (d, 2H, J = 7.4 Hz, ArH); ν_{max}/cm⁻¹ (solid state) = ~3200 (broad) (COOH), 1646, 1603 (CO); ESI-HRMS found m/z 760.3122 [M+H]⁺ requires 760.3131.

2-(4-(4-(2-Amino-N-(naphthalen-2-ylmethyl)acetamido)-N-(naphthalen-2-ylmethyl)benzamido)-N-(naphthalen-2-ylmethyl)benzamido)benzamido)benzamido)acetic acid 21

Precipitated from MeOH/Et₂O. Isolated yield: 15 mg. δ_H (500 MHz, MeOD) 3.41 (s, 2H, CH₂), 3.90 (s, 2H, CH₂), 5.00 (s, 2H, CH₂), 5.12 (s, 2H, CH₂), 5.16 (s, 2H, CH₂), 5.64 (d, 2H, J = 7.0 Hz, ArH), 6.68 (d, 2H, J = 7.4 Hz, ArH), 6.88 (d, 2H, J = 8.7 Hz, ArH), 6.95 (d, 2H, J = 8.8 Hz, ArH), 7.10 (d, 2H, J = 8.1 Hz, ArH), 7.21-7.71 (m, 21H, ArH), 7.75 (d, 2H, J = 7.8 Hz, ArH); ν_{max}/cm⁻¹ (solid state) = ~3200 (broad) (COOH), 1645, 1599 (CO); ESI-HRMS found m/z 910.3644 [M+H]⁺ requires 910.3599.

2-(4-(4-(2-Amino-N-benzylacetamido)-N-propylbenzamido)-N-(naphthalen-2-ylmethyl)benzamido)benzamido)acetic acid 22

Precipitated from MeOH/Et₂O. Isolated yield: 9 mg. δ_H (500 MHz, MeOD) 0.78 (t, 3H, J = 7.5 Hz, CH₂CH₂CH₃), 1.43 (m, 2H, CH₂CH₂CH₃), 3.33 (s, 2H, CH₂), 3.71 (t, 2H, J = 7.4 Hz, CH₂CH₂CH₃), 3.84 (s, 2H, CH₂), 4.78 (beneath solvent H₂O) (s, 2H, CH₂), 5.25 (s, 2H, CH₂), 6.76-6.85 (m, 5H, ArH), 6.88 (d, 2H, J = 8.5 Hz, ArH), 7.00 (m, 4H, ArH), 7.06 (d, 2H, J = 7.6 Hz, ArH), 7.14 (d, 2H, J = 8.8 Hz, ArH), 7.35-7.41 (m, 5H, ArH), 7.59 (s, 1H, ArH), 7.65 (m, 1H, ArH), 7.73 (d, 2H, J = 8.8 Hz, ArH); ν_{max}/cm⁻¹ (solid state) = ~3200 (broad) (COOH), 1646, 1602 (CO); ESI-HRMS found m/z 762.3275 [M+H]⁺ requires 762.3286.

2-(4-(4-(2-Amino-N-benzylacetamido)-N-(3-aminopropyl)benzamido)-N-(naphthalen-2-ylmethyl)benzamido)benzamido)acetic acid 23

Precipitated from MeOH/Et₂O. Isolated yield: 11 mg. δ_H (500 MHz, MeOD) 1.75 (m, 2H, J = 6.7 Hz, CH₂), 2.84 (t, 2H, J = 6.9 Hz, CH₂), 3.34 (s, 2H, CH₂), 3.86 (s, 2H, CH₂), 3.90 (t, 2H, J = 7.0 Hz, CH₂), 4.79 (under H₂O solvent peak) (s, 2H, CH₂), 5.27 (s, 2H, CH₂), 6.88 (m, 7H, ArH), 7.00 (m, 4H, ArH), 7.09 (d, 2H, J = 7.7 Hz, ArH), 7.16 (d, 2H, J = 8.8 Hz, ArH), 7.41 (m, 5H, ArH), 7.61 (m, 1H, ArH), 7.67 (m, 1H, ArH), 7.75 (d, 2H, J = 8.4 Hz, ArH); ν_{max}/cm⁻¹ (solid state) = ~3200 (broad) (COOH), 1677, 1652, 1648, 1602 (CO); ESI-HRMS found m/z 389.1741 [M+2H]²⁺ requires 389.1734.

2-(4-(4-(2-Amino-N-benzylacetamido)-N-propylbenzamido)-N-propylbenzamido)benzamido)acetic acid 24

Precipitated from MeOH/Et₂O. Isolated yield: 11 mg. δ_H (500 MHz, CHCl₃) 0.77 (t, 3H, J = 7.4 Hz, CH₂CH₂CH₃), 0.86 (t, 3H, J = 7.8 Hz, CH₂CH₂CH₃), 1.41 (m, 2H, CH₂CH₂CH₃),
1.54 (m, 2H, CH₂CH₂CH₃), 3.72 (t, 2H, J = 7.5 Hz, CH₂CH₂CH₃), 3.83 (t, 2H, J = 7.4 Hz, CH₂CH₂CH₃), 3.91 (s, 2H, CH₂), 4.75 (s, 2H, CH₂), 4.82 (s, 2H, CH₂), 6.84 (d, 2H, J = 7.9 Hz, ArH), 6.89 (d, 2H, J = 8.9 Hz, ArH), 7.00 (d, 2H, J = 8.3 Hz, ArH), 7.06 (m, 5H, ArH), 7.16 (m, 4H, ArH), 7.58 (d, 2H, J = 8.3 Hz, ArH); νmax/cm⁻¹ (solid state) = ~3200 (broad) (COOH), 1646, 1603 (CO); ESI-MS HRMS found m/z 664.3111 [M+H]⁺ C₃₈H₄₂N₅O₆ requires 664.3130.

2-(4-(4-(2-Amino-N-benzylacetamido)-N-(3-aminopropyl)benzamido)-N-propylbenzamido)benzamido)acetic acid 25

Precipitated from MeOH/Et₂O. Isolated yield: 12 mg. δH (500 MHz, MeOD) 0.87 (t, 3H, J = 7.8 Hz, CH₂CH₂CH₃), 1.55 (m, 2H, CH₂CH₂CH₃), 1.72 (m, 2H, CH₂), 2.83 (t, 2H, J = 7.5 Hz, CH₂), 3.32 (s, 2H, CH₂), 3.84 (t, 2H, J = 7.1 Hz, CH₂CH₂CH₃), 3.88 (m, 4H, 2xCH₂), 4.82 (s, 2H, CH₂), 6.86 (d, 2H, J = 8.6 Hz, ArH), 6.90 (d, 2H, J = 8.8 Hz, ArH), 6.99-7.16 (m, 9H, ArH), 7.58 (d, 2H, J = 7.5 Hz, ArH); νmax/cm⁻¹ (solid state) = ~3200 (broad) (COOH), 1664, 1635, 1599 (CO); ESI-MS HRMS found m/z 340.1661 [M+2H]²⁺ C₃₈H₄₄N₄O₆ requires 340.1656, found m/z 679.3213 [M+H]⁺ C₃₈H₄₃N₅O₆ requires 679.3239.

2-(4-(4-(2-Amino-N-(naphthalen-2-ylmethyl)acetamido)-N-(naphthalen-2-ylmethyl)benzamido)-N-propylbenzamido)benzamido)acetic acid 26

Precipitated from MeOH/Et₂O. Isolated yield: 10 mg. δH (500 MHz, MeOD) 0.85 (t, 3H, J = 7.8 Hz, CH₂CH₂CH₃), 1.49 (m, 2H, CH₂CH₂CH₃), 3.41 (s, 2H, CH₂), 3.72 (t, 2H, J = 7.7 Hz, CH₂CH₂CH₃), 3.95 (s, 2H, CH₂), 5.04 (s, 2H, CH₂), 5.12 (s, 2H, CH₂), 6.59 (d, 2H, J = 7.8 Hz, ArH), 6.68 (d, 2H, J = 7.7 Hz, ArH), 6.83 (d, 2H, J = 7.9 Hz, ArH), 6.98 (d, 2H, J = 7.5 Hz, ArH), 7.11 (d, 2H, J = 8.6 Hz, ArH), 7.21 (d, 1H, J = 8.6 Hz, ArH), 7.30-7.35 (m, 7H, ArH), 7.44 (s, 1H, ArH), 7.48 (s, 1H, ArH), 7.65-7.70 (m, 6H, ArH); νmax/cm⁻¹ (solid state) = ~3200 (broad) (COOH), 1630, 1601 (CO); ESI-MS HRMS found m/z 812.3408 [M+H]⁺ C₅₀H₄₆N₅O₆ requires 812.3443.

2-(4-(4-(2-Amino-N-(naphthalen-2-ylmethyl)acetamido)-N-benzylbenzamido)-N-propylbenzamido)benzamido)acetic acid 27

Precipitated from MeOH/Et₂O. Isolated yield: 8 mg. δH (500 MHz, MeOD) 0.87 (t, 3H, J = 6.9 Hz, CH₂CH₂CH₃), 1.53 (m, 2H, CH₂CH₂CH₃), 3.40 (s, 2H, CH₂), 3.75 (t, 2H, J = 6.9 Hz, CH₂CH₂CH₃), 3.94 (s, 2H, CH₂), 4.94 (s, 2H, CH₂), 5.03 (s, 2H, CH₂), 6.64 (m, 4H, ArH), 6.84 (d, 2H, J = 8.4 Hz, ArH), 6.96 (d, 2H, J = 8.5 Hz, ArH), 7.03 (d, 2H, J = 6.8 Hz, ArH), 7.08-7.13 (m, 4H, ArH), 7.30 (d, 1H, J = 8.5 Hz, ArH), 7.36-7.40 (m, 4H, ArH), 7.44 (s, 1H, ArH), 7.65-7.71 (m, 4H, ArH); νmax/cm⁻¹ (solid state) = ~3200 (broad) (COOH), 1646, 1599 (CO); ESI-MS HRMS found m/z 762.3265 [M+H]⁺ C₄₆H₄₄N₅O₆ requires 762.3286.
2-(4-(4-(2-Amino-N-benzylacetamido)-N-(naphthalen-2-ylmethyl)benzamido)-N-isobutylbenzamido)benzamido)acetic acid 28

Precipitated from MeOH/Et₂O. Isolated yield: 15 mg. δ\textsubscript{H} (500 MHz, MeOD) 0.81 (d, 6H, J = 6.1 Hz, (CH\textsubscript{3})\textsubscript{2}), 1.72 (m, 1H, CH), 3.27 (s, 2H, CH\textsubscript{2}), 3.69 (d, 2H, J = 7.9 Hz, CH\textsubscript{2}), 4.01 (s, 2H, CH\textsubscript{2}), 4.80 (s, 2H, CH\textsubscript{2}), 5.57 (s, 2H, CH\textsubscript{2}), 6.72 (d, 2H, J = 7.9 Hz, ArH), 6.89 (d, 2H, J = 8.4 Hz, ArH), 6.96 (m, 4H, ArH), 7.02 (d, 2H, J = 6.0 Hz, ArH), 7.16 (m, 5H, ArH), 7.35 (m, 2H, ArH), 7.50 (s, 1H, ArH), 7.56 (d, 2H, J = 8.0 Hz, ArH), 7.67 (m, 4H, ArH); ν\textsubscript{max}/cm\textsuperscript{-1} (solid state) = ~3000 (broad) (COOH), 1635, 1601 (CO); ESI-HRMS found m/z 776.3430 [M+H]\textsuperscript{+} C\textsubscript{47}H\textsubscript{46}N\textsubscript{5}O\textsubscript{6} requires 776.3443.

2-(4-(4-(2-Amino-N-benzylacetamido)-N-propylbenzamido)benzamido)acetic acid 7

Precipitated from MeOH/Et₂O δ\textsubscript{H} (500 MHz, MeOD) 0.87 (t, 3H, J = 6.7 Hz, CH\textsubscript{2}CH\textsubscript{2}CH\textsubscript{3}), 1.54 (m, 2H, CH\textsubscript{2}CH\textsubscript{2}CH\textsubscript{3}), 3.16 (s, 2H, CH\textsubscript{2}), 3.84 (m, 2H, J = 6.7 Hz, CH\textsubscript{2}CH\textsubscript{2}CH\textsubscript{3}), 4.80 (s, 2H, CH\textsubscript{2}), 6.82 (d, 2H, J = 8.4 Hz, ArH), 6.98 (m, 2H, ArH), 7.07 (d, 2H, J = 8.3 Hz, ArH), 7.13 (m, 3H, ArH), 7.17 (d, 2H, J = 8.5 Hz, ArH), 7.62 (d, 2H, J = 8.3 Hz, ArH); ν\textsubscript{max}/cm\textsuperscript{-1} (solid state) = ~3200 (COOH), 1646, 1602 (CO); ESI-HRMS found m/z 503.2288 [M+H]\textsuperscript{+} C\textsubscript{28}H\textsubscript{31}N\textsubscript{4}O\textsubscript{5} requires 50.2289.

Figure ESI 1. Competition titration curve for the inhibition of p53*-hDM2 interaction (p53\textsubscript{15-29Flu} 54 nM, 41 nM hDM2, 40 mM sodium phosphate, pH 7.4, 200 mM NaCl and 0.02 mg/ml of bovine serum albumin)
Figure ESI 2. $^{15}$N-$^{1}$H HSQC spectra of (600 MHz, 25°C, 60 mM sodium acetate, 60 mM sodium phosphate, pH 7.3, 10% D$_2$O) of (a) red = 125 µM hDM2, black = 125 µM hDM2 and 200 µM p53 peptide (b) red = 125 µM hDM2, green = 125 µM hDM2 and 250 µM 11
**Figure ESI 3.** $^{15}$N-$^1$H HSQC spectra of (600 MHz, 25ºC, 60 mM sodium acetate, 60 mM sodium phosphate, pH 7.3, 10% D$_2$O) of (a) red = 125 µM hDM2, black = 125 µM hDM2 and 200 µM p53 peptide (b) red = 125 µM hDM2, green = 125 µM hDM2 and 250 µM 11

**Figure ESI 4.** Comparisons of backbone distance αC changes between apo hDM2 and (a) p53 peptide (b) nutlin-3 and $^{15}$N-$^1$H HSQC chemical shift mapping between apo hDM2 and (c) p53 (d) 11.
LC-MS and $^1$H NMR spectra of $N$-alkylated proteomimetic s

2-(4-($N$-Isobutyl-4-($N$-isobutyl-4-($isobutylamino$)benzamido)benzamido)benzamido)acetic acid; 8

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**Figure 1:**

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**Figure 2:**

2.1_435_1-C,1,01_292d: UV Chromatogram, 190-650 nm

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2-(4-(4-(Benzylationo)-N-isobutylbenzamido)-N-isobutylbenzamido)benzamido)acetic acid; 9

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![Graph 1](image1)

![Graph 2](image2)

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2-(4-(4-(N-Benzyl-4-(isobutylamino)benzamido)-N-isobutylbenzamido)benzamido)acetic acid; 10

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2-(4-(4-(N-Benzyl-4-(benzylamino)benzamido)-N-isobutylbenzamido)benzamido)acetic acid; 11

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**Benzyl-4-(N-benzyl-4-(isobutylamino)benzamido)benzamido)benzamido)acetic acid; 12**

<table>
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<th>Alternating Ion Polarity</th>
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<td>1300 m/z</td>
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**Evaluation**

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<th>H, Na</th>
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![Graph 1](image1.png)

![Graph 2](image2.png)

<table>
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<th>Area Frac. %</th>
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2-(4-(N-2-(4-(N-Benzyl-4-(N-isobutyl-4-isobutylamino)benzamido)-benzamido)-benzamido) acetic acid; 13
2-(4-(N-Benzyl-4-(benzylamino)-N-isobutylbenzamido)benzamido)benzamido)acetic acid; 14

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**Figure:**

- Top: 2.9_442_1-C,8_01_299.d: BPC 50.0-1300.0 +All MS
- Bottom: 2.9_442_1-C,8_01_299.d: UV Chromatogram, 190-650 nm

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2-(4-(4-(Benzy lamino)-N-(naphthalen-2-ylmethyl)benzamido)-N-isobutylbenzamido)benzamido)acetic acid; 15

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![Graph 1](image1)

![Graph 2](image2)

<table>
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![Graph 3](image3)
2-(4-(4-(2-Amino-N-(naphthalen-2-ylmethyl)acetamido)-N-(naphthalen-2-ylmethyl)benzamido)-N-isobutylbenzamido)benzamido)acetic acid; 16

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![Graph 1](image1)

![Graph 2](image2)

<table>
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2-(4-(4-(2-Amino-N-benzylacetamido)-N-isobutylbenzamido)-N-isobutylbenzamido)benzamido)acetic acid; 17
2-(4-(4-(2-Amino-N-benzylacetamido)-N-propylbenzamido)-N-isobutylbenzamido)benzamido)acetic acid; 18

### Acquisition

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### Evaluation

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Supplementary Material (ESI) for Organic and Biomolecular Chemistry
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2-(4-(4-(2-Amino-N-benzylacetamido)-N-(3-aminopropyl)benzamido)-N-benzylbenzamido)benzamido)acetic acid; 19

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**Retention Time (min):**
- Compound 1: 1.41
- Compound 2: [Additional retention times provided]

**Area %:**
- Compound 1: 100.0
- Compound 2: [Additional area % provided]
2-(4-(4-(2-Amino-N-benzylacetamido)-N-benzylbenzamido)-N-benzylbenzamido)benzamido)acetic acid; 20

Supplementary Material (ESI) for Organic and Biomolecular Chemistry
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### Acquisition

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### Evaluation

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### Chromatogram

#### 3.8_519_1-A,5_01_371.d: BPC 50.0-1300.0 +All MS

| # | Compound | RT [min] | Range [min] | Max. m/z | Area | Area % | Area %
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2-(4-(4-(2-Amino-N-(naphthalen-2-ylmethyl)acetamido)-N-(naphthalen-2-ylmethyl)benzamido)-N-(naphthalen-2-ylmethyl)benzamido)benzamido)acetic acid; 21

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![Graph 1](image1.png)

![Graph 2](image2.png)

<table>
<thead>
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<th>Range [min]</th>
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<th>Area %</th>
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2-(4-(4-(2-Amino-N-benzylacetamido)-N-propylbenzamido)-N-(naphthalen-2-ylmethyl)benzamido)benzamido)acetic acid; 22

### Acquisition
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### Evaluation
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![MS chromatogram](image)

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2-(4-(4-(2-Amino-N-benzylacetamido)-N-(3-aminopropyl)benzamido)-N-(naphthalen-2-ylmethyl)benzamido)benzamido)acetic acid; 23
2-(4-(4-(2-Amino-N-benzylacetamido)-N-propylbenzamido)-N-propylbenzamido)benzamido)acetic acid; 24

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![Chromatogram Image]

![UV Chromatogram Image]

<table>
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<td>Compd 3, 2.18 min</td>
<td>2.18</td>
<td>2.09 - 2.28</td>
<td>719.4</td>
<td>8149957</td>
<td>6.7</td>
<td>6.2</td>
</tr>
</tbody>
</table>
2-(4-(4-(2-Amino-N-benzylacetamido)-N-(3-aminopropyl)benzamido)-N-propylbenzamido)benzamido)acetic acid; 25

**Acquisition**
- Ion Source Type: ESI
- Mass Range Mode: Std/Enhanced
- Scan Begin: 50 m/z
- Scan End: 1300 m/z
- Ion Polarity: Positive
- Alternating Ion Polarity: off

**Evaluation**
- Expected Formula 1
- Expected Mass 1: 678
- Expected Formula 2
- Expected Mass 2
- Adductions: H, Na
- Neutral Losses

### Chromatographic Results

<table>
<thead>
<tr>
<th>#</th>
<th>Compd. Label</th>
<th>RT [min]</th>
<th>Range [min]</th>
<th>Max. m/z</th>
<th>Area</th>
<th>Area %</th>
<th>Area Frac. %</th>
</tr>
</thead>
<tbody>
<tr>
<td>1</td>
<td>Compd 1, 1.17 min</td>
<td>1.17</td>
<td>1.14 - 1.21</td>
<td>319.2</td>
<td>36080462</td>
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<td>2</td>
<td>Compd 2, 1.29 min</td>
<td>1.29</td>
<td>1.21 - 1.38</td>
<td>340.2</td>
<td>1767949305</td>
<td>100.0</td>
<td>95.0</td>
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<tr>
<td>3</td>
<td>Compd 3, 1.76 min</td>
<td>1.76</td>
<td>1.73 - 1.80</td>
<td>279.2</td>
<td>56272421</td>
<td>3.2</td>
<td>3.0</td>
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</table>
2-(4-(4-(2-Amino-N-(naphthalen-2-ylmethyl)acetamido)-N-(naphthalen-2-ylmethyl)benzamido)-N-propylbenzamido)benzamido)acetic acid; 26

**Acquisition**
- Ion Source Type: ESI
- Mass Range Mode: Std/Enhanced
- Ion Polarity: Positive
- Scan Begin: 50 m/z
- Alternating Ion Polarity: off
- Scan End: 1300 m/z

**Evaluation**
- Expected Formula 1
- Expected Formula 2
- Expected Mass 1: 811
- Expected Mass 2

**Graph 1:**
- Intensity vs. Time [min]
- Peak 1
- Peak 2

**Graph 2:**
- Intensity vs. Time [min]
- Peak 1
- Peak 2

<table>
<thead>
<tr>
<th>#</th>
<th>Cmpd. Label</th>
<th>RT [min]</th>
<th>Range [min]</th>
<th>Max. m/z</th>
<th>Area</th>
<th>Area %</th>
<th>Area Frac. %</th>
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</thead>
<tbody>
<tr>
<td>1</td>
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<td>1.60 - 1.66</td>
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<td>1.66 - 1.88</td>
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<td>95.0</td>
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</table>
2-(4-(4-(2-Amino-N-(naphthalen-2-ylmethyl)acetamido)-N-benzylbenzamido)-N-propylbenzamido)benzamido)acetic acid; 27

<table>
<thead>
<tr>
<th>Acquisiton</th>
<th>Ion Source Type</th>
<th>ESI</th>
<th>Mass Range Mode</th>
<th>Std/Enhanced</th>
<th>Ion Polarity</th>
<th>Positive</th>
<th>Alternating Ion Polarity</th>
<th>Off</th>
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<tbody>
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<td></td>
<td></td>
<td></td>
</tr>
</tbody>
</table>

Evaluation
Expected Formula 1
Expected Mass 1 761

Adductions H, Na
Neutral Losses

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Supplementary Material (ESI) for Organic and Biomolecular Chemistry
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2-(4-(4-(2-Amino-N-benzylacetamido)-N-(naphthalen-2-ylmethyl)benzamido)-N-isobutylbenzamido)benzamido)acetic acid; 28

<table>
<thead>
<tr>
<th>Acquisition</th>
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<th>ESI</th>
<th>Ion Polarity</th>
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<th>Alternating Ion Polarity</th>
<th>off</th>
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</thead>
<tbody>
<tr>
<td>Mass Range Mode</td>
<td>Std/Enhanced</td>
<td>Scan Begin</td>
<td>50 m/z</td>
<td>Scan End</td>
<td>1300 m/z</td>
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</table>

<table>
<thead>
<tr>
<th>Evaluation</th>
<th>Expected Formula 1</th>
<th>Expected Formula 2</th>
<th>Adductions</th>
<th>H, Na</th>
</tr>
</thead>
<tbody>
<tr>
<td>Expected Mass 1</td>
<td>Expected Mass 2</td>
<td>Neutral Losses</td>
<td></td>
<td></td>
</tr>
</tbody>
</table>

![MS Spectrum](image1.png)

![Chromatogram](image2.png)

<table>
<thead>
<tr>
<th>#</th>
<th>Cmpd. Label</th>
<th>RT [min]</th>
<th>Range [min]</th>
<th>Max. m/z</th>
<th>Area</th>
<th>Area %</th>
<th>Area Frac. %</th>
</tr>
</thead>
<tbody>
<tr>
<td>1</td>
<td>Cmpd 1</td>
<td>1.68 min</td>
<td>1.64 - 1.89</td>
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