Experimental Procedures

2: A solution of bis(4-methoxycarbonylbenzyl)amine[12] (**1**) (10.6 g, 34 mmol) in dry THF (500 mL) was treated with $LiAlH_4$ (5.6 g, 150 mmol). The reaction mixture was heated under reflux for 20 h and subsequently quenched with acid. The THF solution was decanted and the residue partitioned between 2N NaOH (250 mL) and CH_2Cl_2 (250 mL). The combined organic extracts were washed with 2N NaOH, dried (Na_2SO_4) and the solvents removed in vacuo to afford **2** (7.8 g, 90 %) as a white solid. M.p. 84–85 °C; ^{1}H NMR (200 MHz, $CDCl_3$, 298 K): δ = 3.78 (s, 4H), 4.66 (s, 4H), 7.31 (s, 8H); ^{13}C NMR (75.5 MHz, $CDCl_3$, 293 K): δ = 52.9, 65.2, 127.3, 128.5, 139.7, 139.8; MS (FAB): *m*/*z* = 258 [*M* + H]$^+$; calcd for $C_{16}H_{19}NO_2$ (257.3): C 74.68, H 7.44, N 5.44; found: C 74.71, H 7.49, N 5.40.

3-H·PF_6: A solution of **2** (2.17 g, 8.4 mmol) in dry CH_2Cl_2 (150 mL) was treated with PCC (5.45 g, 25.3 mmol) and stirred under ambient conditions for 1.5 h. The reaction mixture was filtered through Celite, which was subsequently washed with Et_2O (150 mL) and MeOH (100 mL). HCl (100 mL, 12N) was added to the solution and washings prior to the solvents being removed in vacuo. The solid residue was partitioned between 2N NaOH (250 mL) and CH_2Cl_2 (250 mL) and the organic layer was washed exhaustively with more 2N NaOH until it was colorless. The oily residue, obtained upon removal of the solvent, was subjected to column chromatography (SiO_2: CH_2Cl_2/MeOH, 99:1). Treatment of the purified dialdehyde with an excess of methanolic HCl, followed by concentration under reduced pressure, gave a slurry, which was poured into Et_2O (500 mL). The solid was recovered by filtration and dissolved in hot H_2O, to which an excess of aqueous NH_4PF_6 was added, resulting in the precipitation of a white solid, which was washed (Et_2O), and subsequently dried to afford **3**-

H·PF_6 (1.37 g, 41 %). M.p. 158–161 °C with decomp.; ^{1}H NMR (400 MHz, CD_3CN, 300 K): δ = 4.35 (s, 4H), 7.67 (d, *J* = 8 Hz, 4H), 7.97 (d, *J* = 8 Hz, 4H), 10.04 (s, 2H); ^{13}C NMR (100 MHz, CD_3CN, 300 K): δ = 52.3, 131.0, 132.0, 137.4, 138.4, 193.3; MS (FAB): *m*/*z* = 254 [*M* – PF_6]$^+$; calcd for $C_{16}H_{16}NO_2 \cdot H_2O$ (417.28): C 46.05, H 4.35, N 3.36; found: C 46.08, H 4.18, N 3.25.