

3,5-di-*tert*-butylbenzyl alcohol: 3,5-di-*tert*-butylbenzoic acid (20 g, 85 mmol) was dissolved in 30 mL of dry THF. 1M BH₃ (100 mL, 100 mmol) was added via syringe under Ar and the solution was refluxed for 48 h. After the starting material was consumed (as determined by TLC), the solvent was removed *in vacuo*, and the crude material was dissolved in 30 mL of MeOH. 5.0 mL of 5% HCl was added, and the solution was refluxed for 30 min. After removing the solvents *in vacuo*, and the crude material was extracted with Et₂O/H₂O (3 x 100 mL). The organic layers were combined and dried with MgSO₄. The crude material was separated on silica using CH₂Cl₂ as the eluent. 3,5 di-*tert*-butylbenzyl alcohol (18 g, 82 mmol) was recovered in 97% yield as a white solid: mp 49.5-50.2 °C; ¹H NMR (CDCl₃) δ 7.37 (1H, s), 7.22 (2H, s), 4.67 (2H, s), 1.59 (1H, br s), 1.33 (18H, s); ¹³C NMR (CDCl₃) δ 150.7, 139.9, 121.3, 121.1, 65.4, 34.6, 31.3. MS *m/z* [M+Na]⁺; Anal. Calcd for C₁₅H₂₄ONa: 243.1699. Found: 243.1725.

3,5-di-*tert*-butylbenzaldehyde: In a dry 250 mL round bottom flask was dissolved 10 mL of DMSO (15 g, 180 mmol) in 75 mL of dry CH₂Cl₂. The solution was cooled to -78 °C (CO₂, acetone) under Ar, 9.0 mL of oxalyl chloride (12 g, 91 mmol) was added via syringe over 10 min, and the reaction was stirred for 20 min. In a separate flask 3,5 di-*tert*-butylbenzyl alcohol (10 g, 46 mmol) was dissolved in 25 mL of dry CH₂Cl₂, which was added via syringe over 15 min to the activated DMSO. After the reaction was stirred for 45 min, 10 mL of Et₃N was added, and the reaction was allowed to warm to room temperature. The crude material was extracted with H₂O/CH₂Cl₂, organic layers were combined, and then dried with MgSO₄. After filtering off the drying agent and removing the solvent under reduced pressure, 3,5di-*tert*-butylbenzaldehyde was obtained in a quantitative yield as a white solid: mp 77-78.5 °C; ¹H NMR (CDCl₃) δ 7.73 (1H, s), 7.72 (2H, s), 10.0 (1H, s) 1.34 (18H, s); ¹³C NMR (CDCl₃) δ 193.0, 151.8, 136.2, 128.8, 124.1, 34.0, 31.3. MS *m/z* [M+Na]⁺; Anal. Calcd for C₁₅H₂₂ONa: 241.1568. Found: 241.1561.

***N*-(2-amino-ethyl)-3,5-di-*tert*-butylbenzylamine:** In a dry 50 mL round bottom flask was added 3,5-di-*tert*-butyl benzoic acid (2.0 g, 8.5 mmol), carbonyldiimidazole (1.4 g, 10 mmol), and 30 mL of anhydrous CH₃CN. The reaction was stirred for 20 min under Ar at room temperature. In a separate dry 50 mL round bottom flask, fitted with an addition funnel, was placed 6 mL of ethylenediamine. The activated 3,5-di-*tert*-butylbenzoic acid was transferred to the addition funnel and added dropwise to the ethylenediamine over 1 h with rapid stirring. After 3 h, the reaction was extracted with 5% HCl / CHCl₃ (3 X 100 mL), and the organic layers were collected, dried over MgSO₄, and solvent was removed *in vacuo*. The crude material was separated on silica using CHCl₃ containing 5% MeOH. To obtain the free amine, the product was extracted with 2N NaOH / CHCl₃ (3 X 100 mL), and the organic layers were combined and dried with MgSO₄, affording the desired product (2.3 g, 8.2 mmol) in 97% yield as a white solid: mp 191-192 °C; ¹H NMR (CDCl₃) δ 7.60 (2H, s), 7.57 (1H, s), 6.61 (1H, s), 3.52 (2H, **dd**, *J* = 6 Hz) 2.96 (2H, **t**, *J* = 6 Hz), 1.34 (18H, s); ¹³C NMR (CDCl₃) δ 168.8, 151.0, 134.1, 125.4, 121.0, 42.5, 41.3, 34.8, 31.3. FT/IR/MS *m/z* [M+H]⁺; Anal. Calcd for C₁₇H₂₉N₂O: 277.2274. Found: 277.2275.

***N*-3,5-di-*tert*-butylbenzyl-5-amino-valeric acid:** In a dry 50 mL round bottom flask was placed valeric acid (1.6 g, 9.0 mmol), Et₄NOH (1.9 g, 9.0 mmol), and 20 mL of a 50/50 MeOH/CHCl₃ solution. The resulting cloudy mixture was dried with Na₂SO₄ for 20 min. 3,5-di-*tert*-butylbenzaldehyde⁷ (1.0 g, 4.5 mmol) was added to the solution and stirred under Ar for 1 h. After filtering, NaBH₄ (340 mg, 9.0 mmol) was added under rapid stirring. The reaction was left for 1 h, dried under high vacuum exhaustively, and the crude material was extracted with 5% HCl/CHCl₃. The organic layers were combined and dried over MgSO₄. Separation of the crude material was accomplished via HPLC using H₂O / 1 % TFA as the eluent, affording *N*-3,5 di-*tert*-butylbenzyl-5-amino valeric acid TFA salt in a 45% yield as a white solid: mp 159-161 °C; ¹H NMR (CDCl₃) δ 10.9 (1H, s), 8.91 (3H, s), 7.41 (1H, s), 7.33 (2H, s), 4.11 (2H, br s), 2.89 (2H, t, *J* = 6.7 Hz), 2.31 (2H, t, *J* = 6.7 Hz), 1.85 (2H, m), 1.61 (2H, m), 1.29 (18H, s); ¹³C NMR (CDCl₃) δ 177.3, 151.9, 129.1, 124.3, 123.4, 51.7, 46.1, 34.8, 33.1, 31.3, 25.2, 21.6. ESI MS *m/z* [M]⁺; Anal. Calcd for C₂₀H₃₄NO₂: 320.258. Found: 320.2540.