Phase-Transfer Catalyzed Alkylation of Guanidines by Alkyl Halides Under Biphasic Conditions: A Convenient Protocol for the Synthesis of Highly Functionalized Guanidines

Supporting Information

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***N*¹, *N*²-Bis(*tert*-butoxycarbonyl)pyrrolidine-1-carboxamidine (3a):** Obtained as an off-white solid in 96% yield. ¹H NMR (300 MHz, CDCl₃) δ 10.24 (1H, bs), 3.48 (4H, bs), 1.95-1.79 (4H, m), 1.41 (18H, s); MS (EI) *m/e* 314 (19, MH⁺), 201 (29), 184 (60), 70 (41), 57 (100); HRMS (EI) *m/e* (M⁺) calcd (for C₁₅H₂₇N₃O₄) 313.2002, found 313.1999. This compound has been previously characterized. Feichtinger, K.; Zapf, C.; Sings, H. L.; Goodman, M. J. *Org. Chem.* **1998**, 63, 3804-3805.

***N*¹, *N*²-Bis(*tert*-butoxycarbonyl)guanidine (3c):** Obtained as a white crystalline solid in 99% yield. mp 142-143 °C; *R*_f = 0.25 (80% hexanes/20% EtOAc); ¹H NMR (300 MHz, CDCl₃) δ 8.41 (3H, bs), 1.39 (18H, s). This compound has been previously characterized. Dodd, D. S.; Kozikowski, A. P. *Tetrahedron Lett.* **1994**, 35, 977-980.

***N*¹, *N*²-Bis(*tert*-butoxycarbonyl)-*N*³-diethylguanidine (3d):** Obtained in 88% yield as a white solid. mp 83-84 °C; *R*_f = 0.13 (80% hexanes/20% EtOAc); ¹H NMR (300 MHz, CDCl₃) δ 9.67 (1H, bs), 3.48 (4H, q, *J* = 7.0 Hz), 1.48 (18H, s), 1.20 (6H, t, *J* = 7.0 Hz); ¹³C NMR (75 MHz,

CDCl₃) δ 154.2, 42.4, 28.2, 13.0 (4 quaternary carbons were not observed). This compound has been previously characterized. Ko, S. Y.; Lerpiniere, J.; Christofi, A. M. *Synlett*, **1995**, 815-816.

***N*¹, *N*²-Bis(*tert*-butoxycarbonyl)- *N*³-phenylguanidine (3l)**: Obtained as a white solid in 95% yield. mp 134-135 °C; *R*_f = 0.61 (80% hexanes/20% Et₂O); ¹H NMR (400 MHz, CDCl₃) δ 11.62 (1H, bs), 10.33 (1H, bs), 7.61 (2H, d, *J* = 8.0 Hz), 7.31 (2H, t, *J* = 8.0 Hz), 7.10 (1H, t, *J* = 7.5 Hz), 1.54 (9H, s), 1.51 (9H, s). This compound has been previously characterized. Yong, Y. F.; Fui, Y.; Kowalski, J. A.; Lipton, M. A. *J. Org. Chem.* **1997**, 62, 1540-1542.

***N*¹, *N*², *N*³-Tri(*tert*-butoxycarbonyl)guanidine (8)**: Obtained as a white solid in 72% yield. *R*_f = 0.27 (70% hexanes/30% Et₂O); ¹H NMR (400 MHz, CDCl₃) δ 8.62-8.10 (2H, bs), 1.51 (27H, s); MS (EI) *m/e* 360 (10, MH⁺), 304 (15), 248 (27), 192 (54), 148 (45), 57 (100); HRMS (EI) *m/e* (MH⁺) calcd. (for C₁₆H₃₀N₃O₆) 360.2135, found 360.2139. This compound has been previously characterized. Feichtinger, K.; Sings, H. L.; Baker, T. J.; Matthews, K.; Goodman, M. *J. Org. Chem.* **1998**, 63, 8432-8439.

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