The Mannich bases of cyclohexanone with benzylamine and 3,4-methylenedioxybenzylamine hydrochlorides have been prepared in moderate yields using aqueous formaldehyde solution [1]. We report now the synthesis of an analogous product from n-butylamine. A mixture of cyclohexanone (2.00 g, 0.02 mol), paraformaldehyde (1.20 g, 0.04 mol) and n-butylamine hydrochloride (2.22 g, 0.02 mol) was refluxed under stirring in anhydrous ethanol (15 mL) for 5 h (TLC monitoring). The reaction mixture gradually turned into a solution. The solvent was then removed under reduced pressure and the residue was triturated with ice-cooled acetone (20 mL). The separated crystals were filtered, washed with cold acetone, recrystallized from n-butanol and air-dried. Yield: 2.85 g (61 %) of the title compound as colorless crystals.

TLC homogeneous product (TLC: silica gel Merck GF254 Al-sheets, eluted by chloroform-ethanol 3:1).

Mp. 139-140 ºC (n-butanol).

1H NMR (300 MHz, d6-DMSO): 0.87 (t, J = 7.4 Hz, CH3), 1.22-1.41 (m, 3H), 1.47-1.70 (m, 4H), 1.70-1.83 (m, 1H), 1.95-2.05 (m, 1H), 2.17-2.32 (m, 2H), 2.36-2.49 (m, 1H), 2.67-2.78 (m, 1H), 2.83 (dd, J1 » J2 » 6 Hz, 2H, COCH2N), 2.97 [sextet, J = 6.2 Hz, 1H, COCH(CH2)2], 3.10-3.20 (m, 1H), 9.0 (br. s, N+H2).

FT IR (fluorolube): 3050-3400, 2942, 2937, 2747, 2566, 2458, 1713 (C=O), 1622, 1588, 1478, 1460, 1445, 1390, 1345.

EI MS [70 eV; m/z (%)]: 184 (2; MH+), 183 (9; M+), 140 (53), 86 (100), 44 (64), 30 (45);
FAB MS [glycerol; m/z (%)]: 403 (17; M+2 glycerol+HCl), 367 (1; 2M+H+), 276 (4; 2M+H+), 184 (100; MH+ = C11H22NO3), 140 (2), 86 (27), 44 (3).


Reference

Sample Availability: Available from the authors and from MDPI.

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