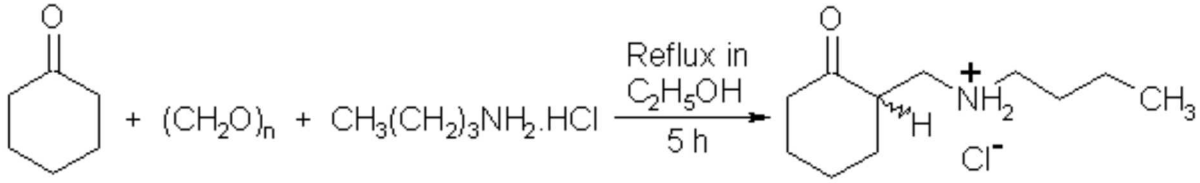
Molecules **2001**, *6*, M215

(*R,S*)-*N*-[(2-Oxocyclohexyl)methyl]-1-butanaminium Chloride

Edmont V. Stoyanov* and Ivo C. Ivanov

Faculty of Pharmacy, Medical University of Sofia, Dunav 2, BG-1000 Sofia, Bulgaria
Tel.: (+359 2) 988 3142; Fax: (+359 2) 987 9874; E-mail: estoyanov@mbox.pharmfac.acad.bg

Received: 20 January 2000 / Accepted: 15 May 2001 / Published: 25 May 2001



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 GF_{254} Al-sheets, eluted by chloroform-ethanol 3:1).

Mp. 139-140 ºC (*n*-butanol).

^{1}H NMR (300 MHz, d_6-DMSO): 0.87 (t, J = 7.4 Hz, CH_3), 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, J_1 »J_2» 6 Hz, 2H, COCHC*H*$_2$N), 2.97 [sextet, J = 6.2 Hz, 1H, COC*H*$(CH_2)_2$], 3.10-3.20 (m, 1H), 9.0 (br. s, N^+H_2).

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; $M^{\cdot}+$), 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; $MH^+ = C_{11}H_{22}NO^+$), 140 (2), 86 (27), 44 (3).

Anal. calcd. for $C_{11}H_{22}NOCl$ (219.76): C 60.12, H 10.09, N 6.37, Cl 16.13; found C 60.04, H 9.99, N 6.33, Cl 16.43.

Reference

1. Mannich, C.; Hieronimus, O. *Ber. Dtsch. Chem. Ges.* **1942**, *75*, 49-55.

Sample Availability: Available from the authors and from MDPI.