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(R,S)-N-[(2-Oxocyclohexyl)methyl]-2-phenyl-1-ethanaminium Chloride

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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 2-phenylethylamine. A mixture of cyclohexanone (2.00 g, 0.02 mol), paraformaldehyde (1.20 g, 0.04 mol) and 2-phenylethylamine hydrochloride (3.18 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: 3.00 g (52 %) of colorless crystals. TLC homogeneous product (TLC: silica gel Merck GF₂₅₄ Al-sheets, eluted by chloroform-ethanol 3:1).

Mp. 155-156 °C (*n*-butanol).

 $^{1}H\ NMR\ (300\ MHz,\ d_{6}\text{-DMSO}):\ 1.23\text{-}1.42\ (m,\ 1H),\ 1.50\text{-}1.84\ (m,\ 3H),\ 1.88\text{-}2.05\ (m,\ 1H),\ 2.09\text{-}2.35\ (m,\ 2H),\ 2.35\text{-}2.47\ (m,\ 1H),\ 2.75\text{-}2.83\ (m,\ 1H),\ 2.92\text{-}3.06\ (m,\ 3H),\ 3.06\text{-}3.18\ (m,\ 2H),\ 3.18\text{-}3.29\ (m,\ 1H),\ 7.20\text{-}7.38\ (m,\ 5H_{arom}),\ 9.15\ (br.\ s,\ N^{^{+}}H_{2}).$

FT IR (fluorolube): 3023, 2936, 2861, 2750, 2705, 1705 (C=O), 1593, 1455, 1424, 1390.

FAB-MS [glycerol; m/z (%)]: 499 (2M + HCl + H+(super)), 463 (2; 2M + H⁺), 324 (6; MH⁺ + glycerol), 232 (100; MH⁺= $C_{15}H_{22}NO^{+}$), 140 (6), 134 (25), 105 (13).

Anal. calcd. for C₁₅H₂₂NOCl (267.80): C 67.28, H 8.28, N 5.23, Cl 13.24; found C 67.24, H 8.27, N 5.23, Cl 13.36.

Reference

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

Sample Availability: Available from the authors and from MDPI.

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