Molbank **2006**, M463 http://www.mdpi.net/molbank/

Synthesis and acidic properties of new 1-phenylacetyl-3-ethyl-4-(4-hydroxybenzylidenamino)-4,5-dihydro-1*H*-1,2,4-triazol-5-one

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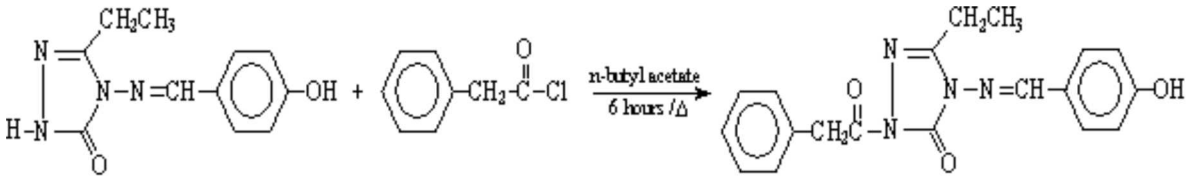
Received: 12 October 2005 / Accepted: 26 November 2005 / Published: 22 January 2006



1 **2**

It is known that 1,2,4-triazole and 4,5-dihydro-1*H*-1,2,4-triazol-5-one rings have weak acidic properties, so some 1,2,4-triazole and 4,5-dihydro-1*H*-1,2,4-triazol-5-one derivatives were titrated potentiometrically with tetrabutylammonium hydroxide in non-aqueous solvents, and the pK_a values of the compounds were determined [1-3]. Determination of the pK_a values of active constituent of certain pharmaceutical preparations is important, because their distribution, transport behavior, bonding to receptors, and contributions to metabolic behavior of the active constituent molecules depend on the ionization constant [4]. 1-Phenylacetyl-3-ethyl-4-(4-hydroxybenzylidenamino)-4,5-dihydro-1*H*-1,2,4-triazol-5-one **2** was synthesized from the reaction of 3-ethyl-4-(4-hydroxybenzylidenamino)-4,5-dihydro-1*H*-1,2,4-triazol-5-one **1** with phenylacetyl chloride. Moreover, the synthesized compound **3** was titrated potentiometrically with tetrabutylammonium hydroxide (TBAH) in four non-aqueous solvents such as isopropyl alcohol, *tert*-butyl alcohol, acetonitrile and *N,N*-dimethylformamide to determine pK_a values. For compound **2**, the half-neutralization potentials (HNP) and the corresponding pK_a values were determined in the four non-aqueous solvents mentioned above. The starting compound **1** was prepared according to literature [5].

3-Ethyl-4-(4-hydroxybenzylidenamino)-4,5-dihydro-1*H*-1,2,4-triazol-5-one **1** (2.32 g, 0.01 mol) was refluxed with a solution of phenylacetyl chloride (0.01 mol) in n-butyl acetate (40 mL) for 6 h and then allowed to cool. The crystals formed were filtered. The product was recrystallized from EtOH-H_2O (1:3) gave pure compound **2** (1.62 g, 46.0 %).

Melting point: 200-201 °C (EtOH-H_2O, 1:3; uncorrected).

UV (λ_{max} nm; EtOH) / ε ($dm^3.mol^{-1}.cm^{-1}$) 310 (13720); 222 (12290); 207 (14100).

IR (KBr, cm^{-1}): 3500 (OH); 1760 (C=O); 1610, 1585 (C=N); 850 (1,4-disubstituted benzenoid ring); 735, 695 (monosubstituted benzenoid ring).

^{1}H-NMR (200 MHz, DMSO-d_6): δ= 1,24 (3H, t, CH_3); 2.72 (2H, q, CH_2); 4.27 (2H, s, CH_2); 6.90 (2H, d, Ar-H, *J*=7.02 Hz); 7.33 (5H, s, Ar-H); 7.70 (2H, d, Ar-H, *J*=7.02 Hz); 9.39 (1H, s, N=CH); 10.32 (1H, s, OH).

^{13}C-NMR (50 MHz, DMSO-d_6): δ= 9.40 (CH_3); 18.80 (CH_2); 41.20 (CH_2Ph); 116.05 (2C); 123.80, 126.90, 128.41 (2C), 129.99 (2C); 130.17 (2C); 134.00, 161.40 (aromatic carbons); 148.20 (triazole C_3); 150.40 (N=CH); 157.00 (triazole C_5); 167.30 (C=O).

Elemental Analysis: Calculated for $C_{19}H_{18}N_4O_3$ (350.38): C, 65.13%; H, 5.18%; N, 15.99%. Found: C, 64.89%; H, 4.47%; N, 15.82%.

The HNP values and the corresponding pK_a values of compound 2 in isopropyl alcohol, *tert*-butyl alcohol, acetonitrile and *N,N*-dimethylformamide were: -275 mV (11.40), -374 mV (13.01), -392 mV (13.23) and -468 mV (-), respectively. The pK_a value has not been obtained in *N,N*-dimethylformamide.

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Sample Availability: Available from MDPI.