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Synthesis and acidic properties of novel 3-methyl-4-[(2-amino-1,3,4-thiadiazol-5-il)-thioacetylamino]-4,5-dihydro-1*H*-1,2,4-triazol-5-one

Haydar Yüksek[a]*, Muzaffer Alkan[a] and Şule Bahçeci[b]

[a]Education Faculty, Kafkas University, 36100-Kars, Turkey
tel: (+90)-474-2126608, fax: (+90)-474-2121185, e-mail: hyuksek98@yahoo.com
[b]Fatih Education Faculty, Karadeniz Technical University, 61335-Trabzon, Turkey
*Author to whom correspondence should be addressed

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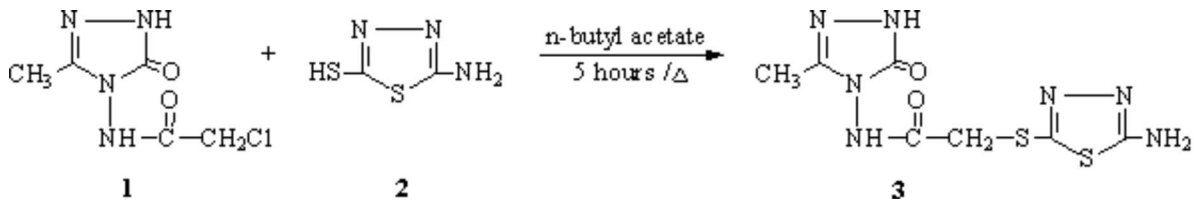

A number of studies involving the determination of pK_a values of some 4,5-dihydro-1*H*-1,2,4-triazol-5-one derivatives in non-aqueous solvents has been revealed [1-3]. 3-Methyl-4-[(2-amino-1,3,4-thiadiazol-5-il)-thioacetylamino]-4,5-dihydro-1*H*-1,2,4-triazol-5-one **3** was synthesized from the reaction of 3-methyl-4-chloroacetylamino-4,5-dihydro-1*H*-1,2,4-triazol-5-one **1** with 2-amino-5-mercapto-1,3,4-thiadiazole **2**. Moreover, the synthesized compound **3** was titrated potentiometrically with tetrabutylammonium hydroxide (TBAH) in three non-aqueous solvents such as isopropyl alcohol, *tert*-butyl alcohol and *N,N*-dimethylformamide to determine pK_a values. For compound **3**, the half-neutralization potentials (HNP) and the corresponding pK_a values were determined in the three non-aqueous solvents mentioned above. The starting compound **1** was prepared according to literature [2,4].

3-Methyl-4-chloroacetylamino-4,5-dihydro-1*H*-1,2,4-triazol-5-one **1** (1.91 g, 0.01 mol) and 2-amino-5-mercapto-1,3,4-thiadiazole **2** (1.33 g, 0.01 mol) in n-butyl acetate (30 mL) was refluxed for five hours and then evaporated at 50-55 °C in vacuo. Several recrystallizations of the residue from ethanol gave pure compound **3** (1.32 g, 45.99 %).

Melting point: 151-152 °C (EtOH, uncorrected).

UV (λ_{max} nm; EtOH) / ε ($dm^3.mol^{-1}.cm^{-1}$): 282 (5980), 203 (9150).

IR (KBr, cm^{-1}): 3400, 3250, 3100 (NH_2, NH); 1750 (C=O); 1605 (C=N).

^{1}H-NMR (200 MHz, DMSO-d_6): δ= 2.09 (3H, s, CH_3); 4.14 (2H, s, CH_2); 6.40 (2H, br, NH_2); 11.40 (1H, s, NH); 11.64 (1H, s, NH).

^{13}C-NMR (50 MHz, DMSO-d_6): δ= 10.45 (CH_3); 34.75 (CH_2); 144.70, 151.76, 152.21, 166.86 (heterocyclic carbons); 169.89 (C=O).

The potentiometric titration curves of 0.001 M compound **3** *solutions* titrated with 0.05 N TBAH in isopropyl alcohol, *tert*-butyl alcohol and *N,N*-dimethylformamide are presented in Figure **1**.

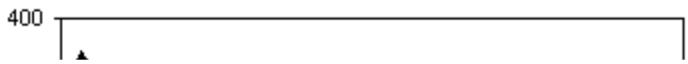

Figure 1

The HNP values and the corresponding pK_a values of compound **3** are presented Table **1**.

Solvent	**HNP_1 (mV)**	**pKa_1**	**HNP_2 (mV)**	**pKa_2**
Isopropyl alcohol	291	2.06	-221	10.64
tert-butyl alcohol	287	2.16	-269	11.61
N,N-dimethylformamide	218	3.36	-342	12.68

Table1: HNP values and the corresponding pK_a values of compound **3**

As seen Figure **1** and Table **1**, compound **3** give two end points as well as two half-neutralization potential values. The potentiometric titration curves of compound **3** resemble the titration curves of diprotic acids.

References:

1. H. Yüksek, Z. Ocak, M. Alkan, Ş. Bahçeci and M. Ozdemir, *Molecules.,* **2004**, *9*, 232-240.
2. H. Yüksek, M. Alkan, Z. Ocak, Ş. Bahçeci, M. Ocak and M. Ozdemir, *Indian J. Chem.,* **2004**, *43B*, 1527-1531.
3. Ş. Bahçeci, H. Yüksek, Z. Ocak, C. Köksal and M. Ozdemir, *Acta Chim. Slov.,* **2002**, *49*, 783-794.
4. A.A. Ikizler and R. Un, *Chim. Acta Turc*., **1979,** *7,* 269-290; *Chem. Abstr*. **1981,** *94,* 15645d.

Sample Availability: Available from the Authors.