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SYNTHESIS AND SOME TRANSFORMATIONS OF HETEROCYCLIC SUBSTITUTED DERIVATIVES OF THIOGLYCOLICACID

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The methods have been developed for the preparation of diheterocyclic systems of a new structure, such as pyrazolo- and 1,3,4-oxadiazolo-1,2,4-triazoles, based on S-substituted thioglycolic acid derivatives.

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***Keywords*:** 1,2,4-triazoles, oxadiazol, pyrazolo-1,2,4-triazoles.

Introduction. We have previously summarized the high biological activities of substituted triazoles and their wide spectrum of action [X]. Individual derivatives of 1,2,4-triazoles exhibit moderate and high antiviral [1], antimicrobial [2, 3], antibacterial [4, 5], anticancer [6, 7], anticonvulsant [8, 9], antifungal [10, 11] activity. Among the numerous developments of recent years, we note the synthesis of condensed systems [12, 13], bis-tetrazolo-1,2,4-triazoles [14], triazino- [15], pyrimidino- [16], pyrido- [17], triazolino- [18], C-glycopyranosyl-1,2,4-triazoles [19], etc.

Obviously, the synthesis and research of new representatives of 1,2,4-triazoles are relevant and appropriate. In order to create new heterocyclic systems and identify the useful properties of the synthesized substances, 3,4-disubstituted-1,2,4-triazole-5-thiols (**1a–e**) were selected as starting compounds, since the synthesis of the latter allows substituents to be introduced into the triazole molecule of various nature, which in turn will allow us to draw conclusions both about the reactivity of the obtained compounds, and about the relationship between structure and biological activity.

It was previously shown that, depending on the structure of the reagent, in the presence of bases, products of either S-substitution or Michael N-addition are formed on the basis of compounds **1**. This is explained by the fact that compounds **1** in the solution are in a thione-thiol equilibrium state. It was also shown that under certain conditions, reactions proceed chemoselectively, with the formation of individual products [20].

Based on the foregoing, this paper presents the results of studies on the synthesis of S-derivatives of 5-mercapto-1,2,4-triazoles (**1**) and their further transformations. To achieve this goal, triazoles **1** were alkylated with chloroacetic

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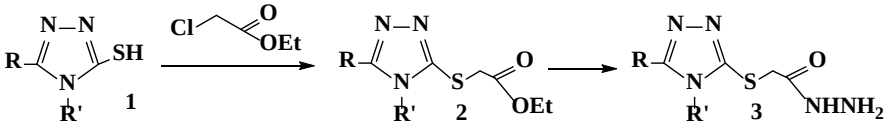
acid ethyl ester. The reaction was found to proceed smoothly with the formation of 2-[(4,5-disubstituted-4*H*-1,2,4-triazole-3-yl)sulfanyl]ethyl acetate (**2a–e**) [20].

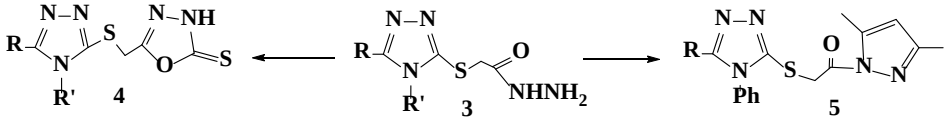


1a. R = 4-Br-C_6H_4-, R' = C_6H_5-;
1b. R = 4-CH_3-C_6H_4-, R' = C_6H_5-;
1c. R = 2-CH_3O-C_6H_4-, R' = allyl;
1d. R = 2-furyl, R' = C_6H_5-;
1e. R = $OHCH_2CH_2CH_2$-, R' = C_6H_5-.

2a. R = 4-Br-C_6H_4-, R' = C_6H_5-;
2b. R = 4-CH_3-C_6H_4-, R' = C_6H_5-;
2c. R = 2-CH_3O-C_6H_4-, R' = allyl;
2d. R = 2-furyl, R' = C_6H_5-;
2e. R = $OHCH_2CH_2CH_2$-, R' = C_6H_5-.

3a. R = 4-Br-C_6H_4-, R' = C_6H_5-;
3b. R = 4-CH_3-C_6H_4-, R' = C_6H_5-;
3c. R = 2-CH_3O-C_6H_4-, R' = allyl;
3d. R = 2-furyl, R' = C_6H_5-;
3e. R = $OHCH_2CH_2CH_2$-, R' = C_6H_5-.

By the known method, the compounds **2** were subjected to hydrazinolysis, resulting in a high yield of acetohydrazides of 2-[(4,5-disubstituted-4*H*-1,2,4-triazole-3-yl)sulfanyls] (**3a–e**), which are good raw materials for various heterocyclic systems. To achieve this goal, some transformations were carried out according to the scheme below.



4a. R = 4-Br-C_6H_4-, R' = C_6H_5-;
4b. R = 4-CH_3-C_6H_4-, R' = C_6H_5-;
4c. R = 2-CH_3O-C_6H_4-, R' = allyl;
4d. R = 2-furyl, R' = C_6H_5-;

5a. R = 2-furyl;
5b. R = $OHCH_2CH_2CH_2$-,

It was shown that the interaction of **3a–d** with a carbon disulfide in the presence of caustic potassium as a result of the further intramolecular cyclization promoted by the latter leads to the formation of triazolo-1,3,4-oxadiazoles 5-[(4,5-disubstituted-4*H*-1,2,4-triazole-3-yl)sulfanyl]methyl-1,3,4-oxadiazol-2(3*H*)-thiones (**4a–d**). Pyrazolo-1,2,4-triazoles (**5a–b**) were also obtained on the basis of **3d–e** by reacting the latter with acetylacetone in the presence of sodium ethylate. The yields of the final products were high. It was also established that the nature of the substituents does not affect the chemoselectivity of the reactions and the yields of the target products.

Experimental Part. ^1H and ^{13}C NMR spectra were recorded on a Varian Mercury-300 MHz spectrometer in DMSO–CCl_4 mixture (1:3) (300 ^1H and 75 ^{13}C). TLC analysis was performed on "Silufol UV-254" plates. Melting points were determined on a "Boetius" micro-heating stage. The starting compounds **1a**–**d** and **2a–d** were synthesized according to known methods [17, 20].

General Method for Preparation of Hydrazides 2-([5-Substituted-4-phenyl-4H-1,2,4-triazole-3-yl]thio)acetates (3a–d). To a mixture of 9.5 *mmol* of the corresponding ester **2a**–**d** in 20 *mL* of ethanol was added 5.2 *mL* of an 85% solution of hydrazine hydrate, left for 2 *h* at room temperature and heated for 4 *h* at 75–80°C. After cooling, the mixture was diluted with water, the precipitate was filtered off, washed with water, dried and re-crystallized.

*2-{[5-(4-Bromphenyl)-4-phenyl-4H-1,2,4-triazole-3-yl]thio}acetohydrazide (**3a**).* Yield 90%, m.p. 230–232°C (ethanol:water=1:4), R_f 0.55 (ethanol:benzene=1:5) [20].

*2-{[5-(4-Tolyl)-4-phenyl-4H-1,2,4-triazole-3-yl]thio}acetohydrazide (**3b**).* Yield 80%, m.p. 136–137°C (ethanol:water=1:1), R_f 0.54 (ethanol:benzene=1:5). ^{1}H NMR, *δ, ppm*: 2.33 s (3H, CH_3); 3.85 s (2H, SCH_2); 4.10 br.s (2H, NH_2); 7.03–7.11 m (2H, $CH_{arom.}$); 7.17–7.26 m (2H, $CH_{arom.}$); 7.27–7.38 m (2H, $CH_{arom.}$); 7.46–7.57 m (2H, $CH_{arom.}$). ^{13}C NMR, *δ, ppm*: 33.6; 60.5; 117.0; 120.9; 126.9; 127.4; 128.5; 129.4; 131.9; 132.7; 148.7; 150.0; 153.8; 167.2. Found, %: C 60.20, H 5.13, N 20.68, S 9.48. $C_{17}H_{17}N_5OS$. Calculated, %: C 60.16, H 5.05, N 20.63, S 9.45.

*2-{[4-Allyl-5-(2-methoxyphenyl)-4H-1,2,4-triazole-3-yl]thio}acetohydrazide (**3c**).* Yield 90%, m.p. 85°C (ethanol:water=2:3), R_f 0.56 (ethanol:benzene=1:6) [20].

*2-{[5-(Furan-2-yl)-4-phenyl-4H-1,2,4-triazole-3-yl]thio}acetohydrazide (**3d**).* Yield 90%, m.p. 111–113°C (ethanol:water=1:1), R_f 0.50 (ethanol:benzene=1:7) [20].

*2-{[5-(3-Hydroxypropyl)-4-phenyl-4H-1,2,4-triazole-3-yl]thio}acetohydrazide (**3e**).* Yield 70%, m.p. 149–150°C (ethanol:water=3:2), R_f 0.50 (ethanol:benzene =1:7). ^{1}H NMR, *δ, ppm*: 2.58 t (2H, *J=7.54 Hz*, CH_2); 3.36–3.49 m (2H, CH_2); 3.93 s (2H, CH_2); 4.14 q (2H, *J*=7.14 *Hz*, CH_2); 4.18 br.s. (1H, OH); 6.10 d (1H, *J*=3.17 *Hz*, NH_2); 6.29 d (1H, *J*=5.55 *Hz*, NH_2); 7.28–7.49 m (2H, $CH_{arom.}$); 7.49–7.73 m (3H, $CH_{arom.}$); 9.28 br.s. (1H, NH). Found, %: C 50.75, H 5.65, N 22.85, S 10.53. $C_{13}H_{17}N_5O_2S$. Calculated, %: C 50.80, H 5.57, N 22.78, S 10.43.

General Method for Preparation of 5-[(4,5-disubstituted-4H-1,2,4-triazole-3-yl)thio]methyl-1,3,4-oxadiazole-2(3H)-thiones(4a–d). To a mixture of 9 *mmol* of the corresponding hydrazide **3a–d** and 6.5 *mL* of ethanol were added 5.4 *mL* of carbon disulfide and, after 15 *min*, 0.0225 *mmol* of potassium. The mixture was stirred at room temperature for 15 *min* and 3 *h* at 75–80°C. After cooling, the mixture was diluted with a 15% solution of hydrogen chlorine to pH 3–4, the precipitate was filtered off, washed with water, dried and re-crystallized.

*5-{[(5-(4-Bromphenyl)-4-phenyl-4H-1,2,4-triazole-3-yl)thio]methyl}-1,3,4-oxadiazole-2(3H)-thione (**4a**).* Yield 94%, m.p. 210–211°C (ethanol:water=10:1), R_f 0.50 (ethanol:benzene=1:2). ^{1}H NMR, *δ, ppm*: 4.01 s (2H, SCH_2); 7.23–7.31 m (3H, $CH_{arom.}$); 7.32–7.48 m (2H, $CH_{arom.}$); 7.49–7.61m (4H, $CH_{arom.}$); 13.02 br.s (1H, NH). ^{13}C NMR, *δ, ppm*: 33.9; 123.3; 124.2; 124.7; 125.4; 127.1; 129.2; 129.6; 131.1; 133.4; 138.8; 151.7; 152.9; 180.4. Found, %: C 44.80, H 2.59, N 17.60, S 14.40. $C_{17}H_{12}BrN_5OS_2$. Calculated, %: C 44.74, H 2.63, N 17.54, S 14.35.

*5-{[(5-(4-Methylphenyl)-4-phenyl-4H-1,2,4-triazole-3-yl)thio]methyl}-1,3,4-oxadiazole-2(3H)-thione (**4b**).* Yield 71%, m.p. 200–201°C (ethanol:water=10:1), R_f 0.58 (ethanol:benzene=1:2). ^{1}H NMR, *δ, ppm*: 2.33 s (3H, CH_3); 4.46 s (2H, SCH_2); 7.00–7.14 m (2H, $CH_{arom.}$); 7.18–7.27 m (2H, $CH_{arom.}$); 7.28–7.37 m (2H, $CH_{arom.}$); 7.45–7.57 m (3H, $CH_{arom.}$); 13.62 br.s (1H, NH). ^{13}C NMR, *δ, ppm*: 13.7; 33.8 (CH2); 123.1; 125.5; 127.1 (2CH); 129.1 (2CH); 129.5 (CH, CH); 131.1 (2CH); 133.5;

151.6; 152.8. Found, %: C 58.80, H 3.29, N 19.10, S 17.50. $C_{18}H_{13}N_5OS_2$. Calculated, %: C 58.85, H 3.54, N 19.07, S 17.44.

5-{[(5-(2-Methoxyphenyl)-4-allyl-4H-1,2,4-triazole-3-yl)thio]methyl}-1,3,4-oxadiazole-2(3H)-thione ***(4c)***. Yield 70%, m.p. 171–172°C (ethanol:water=4:1), R_f 0.53 (ethanol:benzene=1:4). ^{1}H NMR, *δ, ppm*: 3.81–3.87 s (3H, OCH_3); 3.98 s (2H, SCH_2); 4.39–4.49 m (2H, NCH_2); 4.86–4.88 m (1H, $=CH_2$); 5.10–5.12 m (1H, $=CH_2$); 5.70 s (1H, CH=); 7.00–7.14 m (2H, $CH_{arom.}$); 7.33–7.35 m (2H, $CH_{arom.}$); 7.47–7.49 m (1H, $CH_{arom.}$); 13.11 br.s (1H, NH). ^{13}C NMR, *δ, ppm*: 12.9; 13.5; 25.8; 123.1; 124.6; 127.0 (2CH); 129.1 (2CH); 129.5(CH); 131.4; 133.5; 151.6; 152.8. Found, %: C 48.50, H 4.79, N 22.00, S 17.30. $C_{15}H_{15}N_5O_2S_2$. Calculated, %: C 48.52, H 4.84, N 21.56, S 17.25.

5-({[5-(Furan-2-yl)-4-phenyl-4H-1,2,4-triazole-3-yl]thio}methyl)-1,3,4-oxadiazole-2(3H)-thione ***(4d)***. Yield 60%, m.p. 218°C (ethanol:water=5:1), R_f 0.42 (ethanol:benzene=2:5). ^{1}H NMR, *δ, ppm*: 4.45 s (2H, SCH_2); 6.22 d (1H, *J*=4.0*Hz*, CH_{furyl}); 6.40 d (1H, *J*=5.5 *Hz*, CH_{furyl}); 7.36–7.44 m (2H, $CH_{arom.}$); 7.53 s (1H, CH_{furyl}); 7.56–7.63 m (3H, $CH_{arom.}$); 14.20 br.s (1H, NH). ^{13}C NMR, *δ, ppm*: 26.1; 110.9; 110.9; 127.2; 129.4; 129.9; 132.9; 140.9; 143.8; 147.3; 148.7; 159.1; 177.8. Found, %: C 50.40, H 3.06, N 19.66, S 17.98. $C_{15}H_{11}N_5O_2S_2$. Calculated, %: C 50.42, H 3.08, N 19.61, S 17.93.

General Method for Preparation of (3,5-dimethyl-1H-pyrazol-1-yl)-2-(4,5-disubstituted-4H-1,2,4-triazole-3-yl]thio)ethan-1-ones (5a–b). To a mixture of 9 *mmol* of the corresponding hydrazide **3d**–**e** and 6.5 *mL* of ethanol were added 9 *mmol* of acetyl acetone and, after 15 *min*, 0.2 *mL* of concentrated hydrochloric acid. The mixture was stirred at room temperature for 15 *min* and 6 *h* at 80–85°C. After cooling, the mixture was diluted with water, the precipitate was filtered off, washed with water, dried and re-crystallized.

1-(3,5-Dimethyl-1H-pyrazol-1-yl)-2-{[5-(furan-2-yl)-4-phenyl-4H-1,2,4-triazole-3-yl]thio}ethan-1-one ***(5a)***. Yield 60%, m.p. 113–115°C (ethanol:water=1:1); R_f 0.50 (ethanol:benzene=1:6). ^{1}H NMR, *δ, ppm*: 2.10 t (3H, CH_3); 2.46 t (3H, CH_3); 4.45 s (2H, SCH_2); 6.10–6.12 m (1H, CH_{furyl}); 6.22 d (1H, *J*=4.0 *Hz*, CH_{furyl}); 6.40 d (1H, *J*=5.5 *Hz*, CH_{furyl}); 7.36–7.44 m (2H, $CH_{arom.}$); 7.53 s (1H, CH_{furyl}); 7.56–7.63 m (3H, $CH_{arom.}$). ^{13}C NMR, *δ, ppm*: 12.9; 13.5; 38.4; 104.4; 110.1; 110.4; 131.0; 131.2; 131.9; 145.9; 146.9; 151.8; 157.7; 162.6. Found, %:C 60.12, H 4.42, N 18.50, S 8.50. $C_{19}H_{17}N_5O_2S$. Calculated, %: C 60.14, H 4.52, N 18.46, S 8.45.

1-(3,5-Dimethyl-1H-pyrazol-1-yl)-2-{[5-(3-hydroxypropyl)-4-phenyl-4H-1,2,4-triazole-3-yl]thio}ethan-1-one ***(5b)***. Yield 67%, m.p. 70–71°C (ethanol:water=1:1), R_f 0.40 (ethanol:benzene=1:7). ^{1}H NMR, *δ, ppm*: 2.10 t (3H, CH_3); 2.46 t (3H, CH_3); 2,58 t (2H, *J*=7.5 *Hz*, CH_2); 3.36–3.49 m (2H, CH_2); 3.93 s (2H, SCH_2); 4.14 qw (2H, *J*=7.1 *Hz*, CH_2); 4.18 br.s (1H, OH); 6.10–6.12 m (1H, CH); 7.28–7.49 m (2H, $CH_{arom.}$); 7.49–7.73 m (3H, $CH_{arom.}$).^{13}C NMR, *δ, ppm*: 12.9;13.5; 25.3; 29.8; 38.4; 60.4; 110.3; 129.1; 132.0; 142.3; 147.7; 148.2; 149.4; 151.8; 162.3. Found, %: C 37.80, H 4.28, N 18.50, S 8.99. $C_{19}H_{17}N_5O_2S$. Calculated, %: C 37.99, H 4.48, N 18.46, S 8.44.

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ԹԻՈԳԼԻԿՈԼԱԹԹՎԻ ՀԵՏԵՐՈՑԻԿԼԻԿ ՏԵՂԱԿԱԼՎԱԾ ԱԾԱՆՑՅԱԼՆԵՐԻ ՍԻՆԹԵԶ ԵՎ ՈՐՈՇ ՓՈԽԱՐԿՈՒՄՆԵՐ

S-Տեղակալված թիոգլիկոլաթթվի ածանցյալների հենքի վրա մշակվել են նոր կառուցվածքի երկհետերոցիկլիկ համակարգերի ստացման մեթոդներ: Փոխարկումների արդյունքում ստացվել են պիրազոլո- և 1,3,4-օքսադիազոլո-1,2,4-տրիազոլներ:

М. А. САМВЕЛЯН, Т. В. КОЧИКЯН

СИНТЕЗ И НЕКОТОРЫЕ ПРЕВРАЩЕНИЯ ГЕТЕРОЦИКЛОЗАМЕЩЕННЫХ ПРОИЗВОДНЫХ ТИОГЛИКОЛЕВОЙ КИСЛОТЫ

На основе производных S-замещенной тиогликолевой кислоты разработаны способы получения дигетероциклических систем новой структуры, таких как пиразоло- и 1,3,4-оксадиазоло-1,2,4-триазолы.