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A New Selective Method for the Synthesis of 1-Alkyl-3-nitro-1,2,4-triazol-5-ones

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Abstract

Pharmaceuticals such as trazodone, nefazodone, aprepitant, itraconasole and others, which comprise the 1,2,4-triazol-5-one moiety, are widely used in modern medicine and pharmacy. The synthesis and characterization of the compounds of this series are of theoretical and practical interest. Here we report two methods that give access to 3-nitro-1,2,4-triazol-5-one (NTO) alkyl derivatives, which is not possible via other common approaches. The success of our methods is due to the use of preparatively obtainable starting substrates and the positional selectivity of all the reactions along a specified nitrogen atom. The developed approach contributes to the progress of the theory of reactivity of ambident heterocycles and allows for selective synthesis of previously unknown 1-substituted derivatives of NTO.

Keywords: 3-nitro-1,2,4-triazole-5-one, selectivity, nucleophilic substitution, nitration

INTRODUCTION

During recent decades, the chemistry of heterocyclic compounds has become one of the upcoming areas of organic chemistry. The role of these compounds in various areas of science and technology (chemistry, medicine, biology, electronics, *etc.*) cannot be overrated, so the development of new methods for the synthesis of their functional derivatives is highly urgent.

In the azole series, the derivatives of 1,2,4-triazole-5-one attract increasing attention. At present, pharmaceuticals containing a triazole-5-one fragment in their structure are widespread: these include antidepressants (trazodone and nefazodone), antiemetics (aprepitant), antifungal (itraconasole) and anticancer drugs [1]. The triazolone fragment in all the listed preparations is functionalized at the N-position.

Investigations in the area of developing the methods of selective synthesis of new promising materials based on 1,2,4-triazole-5-one, as well as the studies of the features of their behaviour in various chemical processes, are of interest both from the theoretical and practical points of view. Studies in this area will make a contribution into the development of the theory of heterocycle reactivity in the reactions of S_N^{ipso}-substitution of the nitro group, into solving the problem of regioselective functionalization of chemical com-

pounds possessing ambident properties, and will allow us to develop the methodology of the directed synthesis of the derivatives of 3-nitro-1,2,4-triazole.

The goal of the work was to develop the methods for the synthesis of N-functionalized derivatives of 1,2,4-triazole-5-one having one of the "Big Four" alkyl substituents in N_1 position: methyl- (Me-), ethyl- (Et-), isopropyl- (*i*-Pr-), *sec*-butyl- (*s*-Bu-).

EXPERIMENTAL

Physicochemical methods of investigation

NMR ^{1}H and ^{13}C spectra were recorded with a Fourier Transform spectrometer AM-400 Avance 200 (Bruker, Germany) with the working frequency 400.13 and 100.61 MHz for ^{1}H and ^{13}C nuclei, respectively, the solvent was DMSO-d_6. IR spectra of the compounds were recorded with a Fourier Transform spectrometer FT-801 (Russia) in KBr tablets. The melting points of the samples were determined with the help of an SMP 30 instrument (Stuart, UK). Elemental analysis of the obtained substances was carried out using the CHNS element analyzer Flash EA 1112 (Thermo Finnigan, Italy).

Procedures of the synthesis and characterization of the compounds under investigation

1-Alkyl-5-nitro-1,2,4-triazoles (1–4) were obtained by alkylation of 3-nitro-1,2,4-triazole by the corresponding dialkyl sulphate in the alkaline medium or by the corresponding alcohol in the acid medium, followed by the isolation of N_2-isomer from the mixture of products using the procedures described in [2, 3].

1-Alkyl-5-methoxy-1,2,4-triazoles (5–8). The general procedure of the synthesis. A solution of 0.01 mol of the corresponding 1-alkyl-5-nitro-1,2,4-triazole (**1–4**) in 6.0 mL of methanol was heated under intense mixing. A solution of 0.01 mol NaOH was added in portions (by 0.001 mol) to the boiling solution. After the reaction, the mass was cooled to the room temperature, and inorganic salts were separated by filtering. The organic solution was evaporated at reduced pressure. The residue was treated with methylene chloride, and the precipitate was separated by filtering. The solution of the product in methylene chloride was washed with the aqueous solution of Na_2CO_3 and water to achieve the neutral reaction of washing water, dried with anhydrous $MgSO_4$, and the solvent was evaporated under reduced pressure.

1-Sec-butyl-5-methoxy-1,2,4-triazole (8). Yield: 89 %. NMR ^{1}H (400 MHz, DMSO-d_6), δ, ppm: 7.56 (s, 1H, C_5–H); 4.10 (m, 1H, J = 6.5, N-CH-); 4.01 (s, 3H, O–CH_3); 1.68 (m, 2H, J = 7.4, –CH–C$\underline{H}_2$–); 1.30 (d, 3H, J = 6.8, –CH–C$\underline{H}_3$); 0.69 (t, 3H, J = 7.4, –CH_2–C$\underline{H}_3$). NMR ^{13}C (100 MHz, DMSO-d_6), δ, ppm: 159.19 (C_5); 147.79 (C_3); 58.34 (–O–CH_3); 53.76 (N–CH–); 28.79 (–CH–C$\underline{H}_2$); 20.06 (–CH–C$\underline{H}_3$); 10.75 (–CH_2–C$\underline{H}_3$). IR, ν , cm^{-1}: 2969, 2940, 2879, 1551, 1514, 1433, 1413, 1381, 1300, 1273, 1207, 1175, 1131, 1117, 1064, 1004, 964, 867, 805, 730, 663, 617. Found, %: C 53.98, H 8.46, N 28.42. Calculated, %: C 54.17, H 8.44, N 27.08.

1-Alkyl-1,2,4-triazole-5-ones (9–12). General procedure of the synthesis. Method A. A 0.02 mol of the corresponding 1-alkyl-1-alkoxy-1,2,4-triazole (5–8) was added to the solution of 33.4 mL of hydrobromic acid and 33.4 mL of glacial acetic acid. The mixture was kept for 3 h at a temperature of 110–115 °C. Then the reaction mixture was cooled to room temperature. The mass was poured during mixing into 200.0 g of ice, and NaOH was added to pH 8.5. Extraction with 200.0 mL of methylene chloride was carried out, then the mixture was acidified with sulphuric acid to pH ~ 3.5. Five extractions, each with 200.0 mL of methylene chloride were carried out. The united organic fraction was dried with anhydrous $MgSO_4$, and the solvent was evaporated at reduced pressure.

1-Methyl-1,2,4-triazole-5-one (9). Synthesis procedure. Method B. To the solution of 0.01 mol KOH in 6.0 mL of distilled water, 0.01 mol of 1-methyl-5-nitro-1,2,4-triazole (**1**) was added. The reaction was carried out at the temperature of active boiling of the solvent for 18 h. After the exposure, the reaction mass was cooled to room temperature, acidified under mixing to pH ~1.5 with hydrochloric acid. Three extractions were carried out, each with 40.0 mL of methylene chloride. The solvent was removed at reduced pressure. Compound **17** was obtained. Then the solvent was evaporated from the aqueous residue under reduced pressure. Ethanol (6.4 mL) was added to the residue, and the mixture was heated to 75 °C. Inorganic salts were separated by filtering. The filtrate was evaporated at reduced pressure. Compound **9** was thus obtained.

1-Methyl-1,2,4-triazole-5-one (9). Yield: 21 % (method A) and 50 % (method B). T_m = 178–181 °C. NMR ^{1}H (400 MHz, DMSO-d_6), d, ppm:

7.76 (s, 1H, C_5–H); 3.25 (s, 3H, N_2–C$\underline{H}_3$); 11.54 (s, 1H, N_4–H). NMR ^{13}C (100 MHz, DMSO-d_6), d, ppm: 150.60 (C_5); 135.21 (C_3); 33.03 (N–CH_3). IR, ν, cm^{-1}: 3433, 3123, 3057, 2955, 2851, 2573, 1728, 1563, 1511, 1456, 1430, 1402, 1327, 1310, 1282, 1210, 1176, 1038, 1005, 985, 961, 911, 880, 836, 810, 735, 693, 665. Found, %: C 37.10, H 5.18, N 43.98. Calculated, %: C 36.36, H 5.09, N 42.41.

1-Ethyl-1,2,4-triazole-5-one (10). Yield: 44 %. T_m = 103–105 °C. NMR ^{1}H (400 MHz, DMSO-d_6), δ, ppm: 11.30 (s, 1H, N_4-H); 7.73 (s, 1H, C_5–H); 3.62 (quart., 2H, J = 7.2, N_2-C$\underline{H}_2$); 1.18 (t, 3H, J = 7.0, N_2-CH_2C$\underline{H}_3$). NMR ^{13}C (100 MHz, DMSO-d_6), δ, ppm: 153.69 (C_5); 134.99 (C_3); 39.25 (N-CH_2); 14.40 (–CH_3). IR, ν, cm^{-1}: 3139, 3073, 2821, 2721, 2575, 1686, 1656, 1553, 1462, 1448, 1403, 1319, 1290, 1152, 1084, 1030, 940, 867, 832, 786, 746, 673, 638. Found, %: C 42.39, H 6.20, N 39.54. Calculated, %: C 42.47, H 6.24, N 37.15.

1-Isopropyl-1,2,4-triazole-5-one (11). Yield: 71 %. T_m = 96–98 °C (hexane). NMR ^{1}H (400 MHz, DMSO-d_6), δ, ppm: 11.43 (s, 1H, N-H); 7.76 (s, 1H, C_5-H); 4.25 (m, 1H, J = 6.2, C$\underline{H}$–(CH_3)$_2$); 1.23 (d, 6H, J = 6.6, –(CH_3)$_2$). NMR ^{13}C (100 MHz, DMSO-d_6), δ, ppm: 153.32 (C_5); 134.93 (C_3); 45.65 (-N-CH-); 21.53 (-$\underline{C}$H-(CH_3)$_2$). IR, ν, cm^{-1}: 3771, 3144, 3071, 2988, 2843, 2572, 1682, 1639, 1563, 1473, 1456, 1427, 1390, 1294, 1215, 1151, 1137, 1111, 1042, 943, 882, 783, 743, 642. Found, %: C 47.36, H 7.15, N 34.61. Calculated, %: C 47.23, H 7.13, N 33.05.

1-*Sec*-butyl-1,2,4-triazole-5-one (12). Yield: 76 %. T_m = 88–90 °C (hexane). NMR ^{1}H (400 MHz, DMSO-d_6), δ, ppm: 11.41 (s, 1H, N-H); 7.79 (s, 1H, C_5-H); 4.00 (m, 1H, J = 6.7, –C$\underline{H}$–CH_3); 1.60 (m, 2H, J = 7.4, –CH–C$\underline{H}_2$–) 1.22 (d, 3H, J = 6.8, –CH–C$\underline{H}_3$); 0.73 (t, 3H, J = 7.3, –CH_2–C$\underline{H}_3$). NMR ^{13}C (100 MHz, DMSO- d_6), δ, ppm: 153.94 (C_5); 135.00 (C_3); 51.15 (-N-CH-); 28.46 (-$\underline{C}H_2$-CH_3); 19.93 (CH–$\underline{C}H_3$); 11.09 (-CH_2-$\underline{C}H_3$). IR, ν, cm^{-1}: 3154, 2973, 2824, 2573, 1665, 1558, 1449, 1401, 1378, 1361, 1300, 1281, 1202, 1152, 1121, 1106, 1049, 1021, 996, 960, 941, 854, 769, 747, 683, 643. Found, %: C 50.97, H 7.89, N 30.62. Calculated, %: C 51.05, H 7.85, N 29.77.

1-Alkyl-3-nitro-1,2,4-triazole-5-one (13–16). General synthesis procedure. To 1.35 mL of 98 % HNO_3, 4.0 mmol of 1-alkyl-1,2,4-triazole-5-one (**9–12**) was added at 5–10 °C during 2 h, then mixing was carried out for 3 h. Then the mixture was poured into 1.2 g of ice-cold water. After 12 h, the precipitate was washed with water and dried.

1-Methyl-3-nitro-1,2,4-triazole-5-one (13). Yield: 84 %. T_m = 226–228 °C. NMR ^{1}H (400 MHz, DMSO-d_6), δ, ppm: 12.82 (s 1H, N–H); 3.48 (s, 3H, N_2–C$\underline{H}_3$). NMR ^{13}C (100 MHz, DMSO-d_6), δ, ppm: 153.30 (C_5); 146.28 (C_3); 32.89 (N–CH_3). IR, ν, cm^{-1}: 3011, 2888, 2816, 2715, 2112, 1870, 1705, 1546, 1487, 1432, 1371, 1253, 1153, 1011, 858, 830, 794, 749, 731, 686, 631, 611. Found, %: C 24.95, H 2.66, N 41.15. Calculated, %: C 25.01, H 2.80, N 38.88.

1-Ethyl-3-nitro-1,2,4-triazole-5-one (14). Yield: 84 %. T_m = 187–189 °C. NMR ^{1}H (400 MHz, DMSO-d_6), δ, ppm: 13.01 (s, 1H, N-H); 3.88 (quart., 2H, J = 7.0, N_2-C$\underline{H}_2$); 1.24 (t, 3H, J = 7.0, N_2–CH_2C$\underline{H}_3$). NMR ^{13}C (100 MHz, DMSO-d_6), δ, ppm: 152.57 (C_5); 146.23 (C_3); 40.69 (N-CH_2); 14.04 (–CH_3). IR, ν, cm^{-1}: 2989, 2927, 1707, 1588, 1556, 1468, 1359, 1301, 1264, 1196, 1146, 1100, 1064, 1018, 849, 796, 749, 727, 662, 615. Found, %: C 30.39, H 3.72, N 36.74. Calculated, %: C 30.38, H 3.82, N 35.43.

1-Isopropyl-3-nitro-1,2,4-triazole-5-one (15). Yield: 87 %. T_m = 162–164 °C (ethanol). NMR ^{1}H (400 MHz, DMSO-d_6), δ, ppm: 13.08 (s, 1H, N-H); 4.44 (m, 1H, J = 6.7, N-CH–); 1.32 (d, 6H, J = 6.7, -CH-(C$\underline{H}_3$)$_2$). NMR ^{13}C (100 MHz, DMSO-d_6), δ, ppm: 152.19 (C_5); 146.35 (C_3); 47.48 (-N-CH-); 21.37 (-CH-($\underline{C}H_3$)$_2$). IR, ν, cm^{-1}: 3878, 3863, 2987, 2942, 2881, 2795, 2707, 1698, 1582, 1536, 1482, 1359, 1260, 1197, 1172, 1127, 1067, 1008, 894, 844, 784, 755, 728, 670, 648, 622. Found, %: C 34.98, H 4.67, N 35.12. Calculated, %: C 34.89, H 4.68, N 32.55.

1-Sec-butyl-3-nitro-1,2,4-triazole-5-one (16). Yield: 86 %. T_m = 152–154 °C (ethanol); T_b = 165–167 °C (1 mm Hg). NMR ^{1}H (400 MHz, DMSO-d_6), δ, ppm: 4.22 (m, 1H, J = 6.7, N-CH–); 1.68 (m, 2H, J = 1.7, –CH–C$\underline{H}_2$–); 1.30 (d, 3H, –CH–C$\underline{H}_3$); 0.80 (t, 3H, J = 0.8, –CH_2–C$\underline{H}_3$). NMR ^{13}C (100 MHz, DMSO-d_6), δ, ppm: 152.84 (C_5); 146.53 (C_3); 53.03 (-N-CH-); 28.42 (-$\underline{C}H_2$-CH_3); 19.65 (CH–$\underline{C}H_3$); 10.84 (-CH_2-$\underline{C}H_3$). IR, ν, cm^{-1}: 2975, 2938, 2882, 2346, 1698, 1581, 1535, 1476, 1414, 1379, 1359, 1296, 1275, 1253, 1187, 1170, 1132, 1113, 1075, 1006, 963, 866, 840, 806, 776,755, 729, 664, 642, 623. Found, %: C 38.64, H 5.37, N 31.45. Calculated, %: C 38.71, H 5.41, N 30.09.

PRESULTS AND DISCUSSION

One of the directions in N-functionalization of heterocycles with pyrrole nitrogen atoms is alkylation. The existing methods of obtaining N-alkyl-5-nitro-1,2,4-triazole-3-ones assume the use of rigid alkylating agents (for example, dialkyl sulphates). These methods are non-selective, depend on the reagent/substrate ratio and lead to a

complicated and difficult-to-separate mixture of the products of N- and O-alkylation. For example, the result of alkylation of 3-nitro-1,2,4-triazole-5-one (NTO) by dimethyl sulphate was a mixture of products: 4-methyl-5-nitro-1,2,4-triazole-5-one, 1-methyl-3-nitro-1,2,4-triazole-5-one and 3-nitro-5-methoxy-1,2,4-triazole [5].

All known methods of the synthesis of N-alkyl-3-nitro-1,2,4-triazole-5-ones are non-selective and are characterized by the rigid conditions, so the development of new methods of selective synthesis of the functional derivatives of NTO is urgent. We developed an efficient methodology of the synthesis of alkyl-substituted NTO comprising two- or three-stage processes.

In the present work, the initial substrates chosen for the synthesis of alkyl-substituted NTO were preparatively available 1-alkyl-5-nitro-1,2,4-triazoles (R = Me (**1**), Et (**2**), *i*-Pr (**3**), *s*-Bu (**4**)), obtained by alkylation of 3-nitro-1,2,4-triazole in the alkaline [2] or acid [3] medium (Scheme 1).

The first method consists of three stages (see Scheme 1):

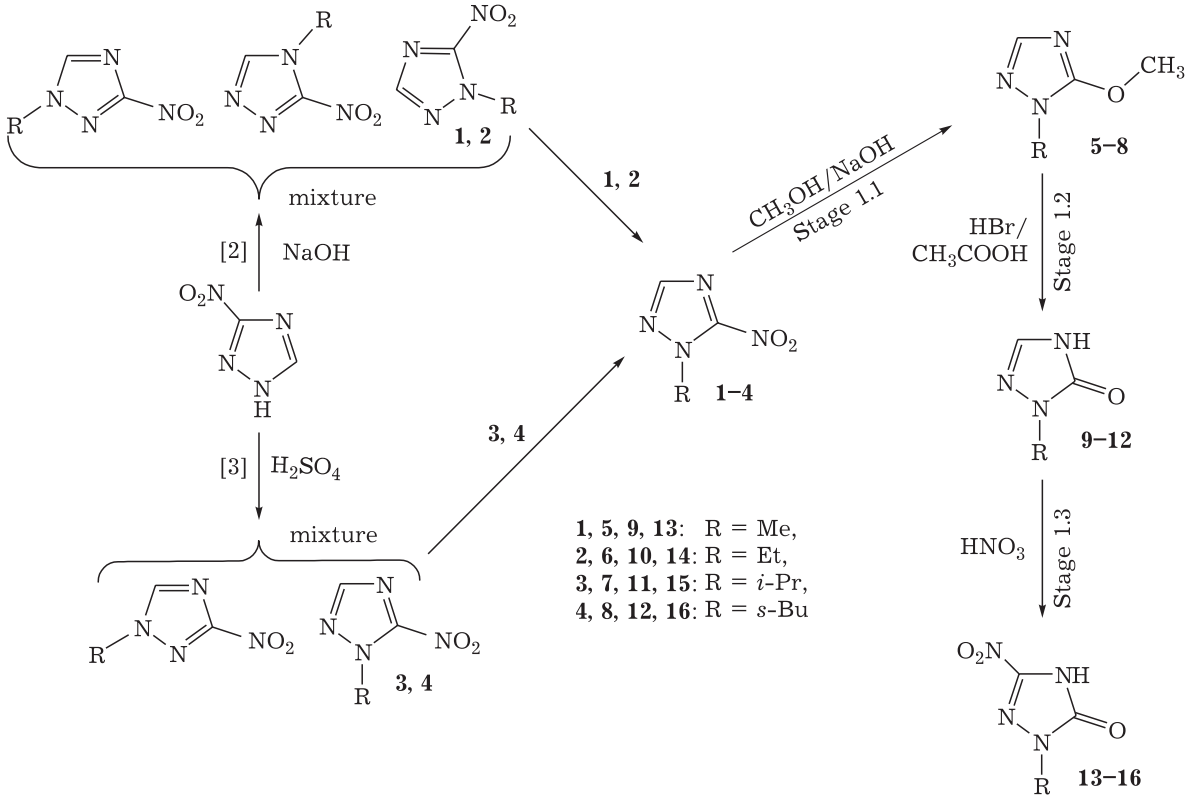
– selective synthesis of 1-alkyl-5-alkoxy-triazoles through the nucleophilic substitution of the nitro group in 1-alkyl-5-nitro-1,2,4-triazoles by O-nucleophiles (stage 1.1);

– selective synthesis of 1-alkyl-1,2,4-triazole-5-ones through saponification of corresponding 1-alkyl-5-alkoxy-1,2,4-triazoles with a mixture of hydrobromic and acetic acids (stage 1.2);

– selective synthesis of 1-alkyl-3-nitro-1,2,4-triazole-5-ones by nitration of 1-alkyl-1,2,4-triazole-5-ones (stage 1.3).

The synthesis of 1-alkyl-5-methoxy-1,2,4-triazoles **5–7** was studied in [4, 6–12]. The process involves the nucleophilic substitution of nitro group in 1-alkyl-5-nitro-1,2,4-triazoles **1–3** by methoxide anion – the most reactive one among the primary O-nucleophiles [4]. As a result, 1-alkyl-5-methoxy-1,2,4-triazoles **5–7** are formed. Within the present investigation, the use of 1-*sec*-butyl-5-nitro-1,2,4-triazole (**4**) as the initial substrate under similar conditions leads to the formation of 1-sec-butyl-5-methoxy-1,2,4-triazole (**8**). It was established that the nature of the alkyl substituent has a substantial effect on the nucleophilic substitution of nitro group in 1-alkyl-5-nitro-1,2,4-triazoles. The reactivity of initial substrates **1–4** in the process of в S_N^{ipso}-substitution of the nitro group depends on the alkyl substituent and decreases as the sequence: Me > Et > *i*-Pr > *s*-Bu.

At stage 1.2, the selective synthesis of 1-alkyl-1,2,4-triazole-5-ones **9–12** was carried out by the



Scheme 1. Three-stage selective synthesis of 1-alkyl-3-nitro-1,2,4-triazole-5-ones (**13–16**).

hydrolysis of alkoxy derivatives **5–8** obtained at stage 1.1 (see Scheme 1) by a mixture of hydrobromic and acetic acids. The activity of initial substrates **5–8** in saponification reaction was evaluated by carrying out the processes under the conditions equal for all reagents (3 h, 110 °C) with the record of the yield of the target product. Unlike for the nucleophilic substitution of the nitro group by methoxide anion (Stage 1.1), a reverse dependence of the effect of alkyl substituent is observed during this process. According to the reactivity, compounds **5–8** in saponification are arranged as a sequence: Me < Et < *i*-Pr < *s*-Bu. The process takes place with the formation of corresponding 1-alkyl-1,2,4- triazole-5-ones **9–12**, which were nitrated as stage 1.3 with concentrated nitric acid. Unlike nitration of NTO, nitration of alkyltriazolones **9–12** is safe, proceeds without the formation of unstable intermediates of N-nitration [13] and is not accompanied by the evolution of brown gases, which are evolved during the decomposition of unstable products of N-nitration. As a result, 1-alkyl-3-nitro-1,2,4-triazole-5-ones **13–16** were obtained with a high yield.

1-Methyl-3-nitro-1,2,4-triazole-5-one **13** may also be obtained using a two-stage method (Scheme 2):

– the synthesis of 1-alkyl-1,2,4-triazole-5-ones by the nucleophilic substitution of the nitro group in 1-alkyl-5-nitro-1,2,4-triazoles by hydroxide anion (Stage 2.1);

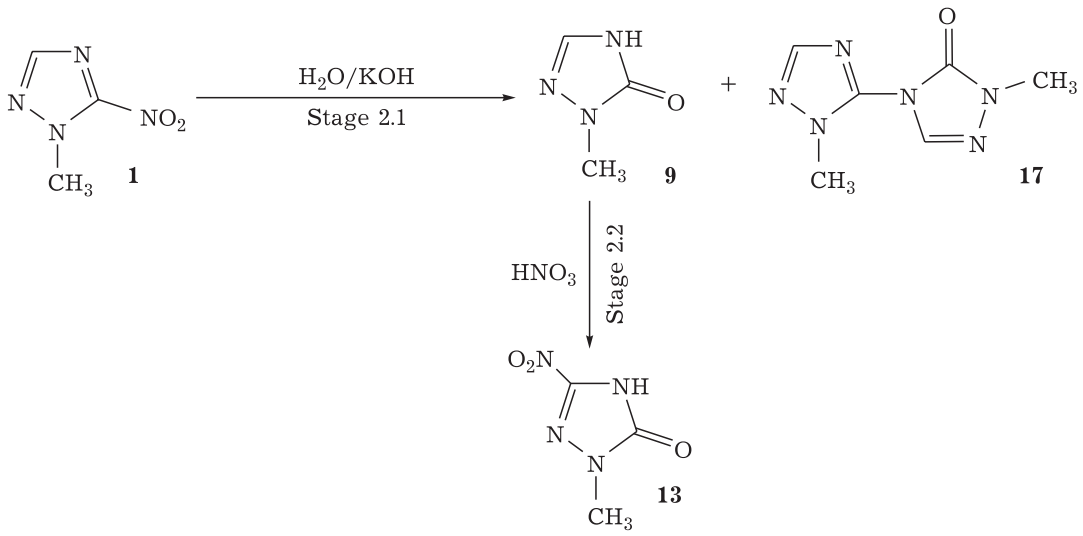
– selective synthesis of 1-alkyl-3-nitro-1,2,4-triazole-5-ones through nitration of 1-alkyl-1,2,4-triazole-5-ones (Stage 2.2).

The use of highly reactive 1-methyl-5-nitro-1,2,4-triazole **1** in the nucleophilic substitution of nitro group by O-nucleophiles [4] allows obtaining 1-methyl-1,2,4-triazole-5-one **9** escaping the stage of alkoxy derivative (Stage 2.1). This process is completed during boiling in the aqueous medium in the presence of an equimolar amount of KOH for 18 h.

However, unlike the nucleophilic substitution of the nitro group by methoxide ion, the nucleophilic substitution of the nitro group in 1-methyl-5-nitro-1,2,4-triazole **1** by hydroxide anion is a typical tandem transformation (see Scheme 2). At first, methyl-1,2,4-triazole-5-one **9** is formed; it is a highly reactive N-nucleophile in heterylation which enters the reaction with initial substrate **1** to form an additional product – 2,2′-dimethyl-2H,2′H-[3,4′] bi([1,2,4]triazolyl)-3′-one **17**, identical to that formed as the side product (not more than 10 %) also in the nucleophilic substitution of nitro group by alcohols in the presence of water [4, 8–10]. The aqueous medium and the high reactivity of N-nucleophile **9** in heterylation lead to the formation of products **9** and **17** at a ratio of 1 : 1.

Nucleophilic substitution of the nitro group in 1-methyl-5-nitro-1,2,4-triazole **1** in the reaction mass was monitored by means of NMR ^{1}H spectroscopy.

The chemical shift of the proton at the carbon atom C_5–H of target product **9** is in the stronger field at 7.74 ppm in comparison with the signal of the ring proton C_5–H of initial nitrotriazole **1**, which appears at 8.15 ppm. This fact allowed us to monitor the completeness of reaction for this



Scheme 2. Two-stage selective synthesis of 1-methyl-3-nitro-1,2,4-triazole-5-one (**13**).

method. The NMR ^{1}H spectrum of the reaction mixture contains two equally intense signals from cyclic protons in the region of 8.05 and 8.22 ppm, which provide evidence of the formation of N–C bicycle **17**.

The second stage of the selective process of obtaining alkyl-substituted NTO (see Scheme 2, stage 2.2) is fully similar to Stage 1.3 and involves nitration of alkyl triazolone **9** by concentrated nitric acid, followed by the isolation of the target product **13**.

CONCLUSION

An efficient method was elaborated for the synthesis of 1-alkyl-3-nitro-1,2,4-triazole-5-ones. The method consists of three selective stages. Unlike other known methods, it allows obtaining a broad range of N-alkyl derivatives of 3-nitro-1,2,4-triazole-5-one.

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