ISSN 1070-4280, Russian Journal of Organic Chemistry, 2021, Vol. 57, No. 2, pp. 219–225. © Pleiades Publishing, Ltd., 2021.
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Three-Component Condencations of 3-Amino-1,2,4-triazoles, Methyl 3-(2-Cycloamino-4-methylpyrimidin-5-yl)-3-oxopropionoates, and a Series of C_1 Synthons as a Convenient Approach to Pyrimidin-5-yl-1,2,4-triazolo[1,5-*a*]pyrimidines

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Received December 2, 2020; revised December 11, 2020; accepted December 14, 2020

Abstract—A convenient synthetic approach to polysubstituted dihydrogenated or heteroaromatic 1,2,4-triazolo-[1,5-*a*]pyrimidines derivatives containing at position 5 a 4-methylpyrimidine moiety bearing a cycloamino substituent at position 2 and linked to the triazolopyrimidine bicycle through its position 5 was developed. The approach involves unusual three-component condensations of 3-amino-1,2,4-triazoles, methyl 3-(2-R-4-methylpyrimidin-5-yl)-3-oxopropionates, and a series of C_1 synthons whose synthetic equivalents are a series of aromatic aldehydes, triethyl orthoformate, or DMFDMA were used as of C_1 synthons.

Keywords: triazolopyrimidine, 4-methylpyrimidine, 1,3-ketocarboxylic acid ester, C_1 synthon, three-component condensation, molecular hybridization, regioselectivity

DOI: 10.1134/S1070428021020123

INTRODUCTION

In the context of the search for new hybrid multitarget molecules [1] containing fragments, one of which would have an antibacterial and the other an anticoagulant activity, we proposed to combine 1,2,4-triazolo[1,5-*a*]-pyrimidine and 2-cycloalkylamino-substituted pyrimidine scaffolds (Fig. 1).

The first components of the system **I** is a partially hydrogenated or heteroaromatic 1,2,4-triazolo[1,5-*a*]-pyrimidine bicycle **I** is interesting as a scaffold with pronounced antibacterial and anticancer properties [2]. Moreover, over the past years 1,2,4-triazolo[1,5-*a*]-pyrimidine derivatives have been considered as as promising reverse transcriptase inhibitors of a number of viruses [3]. The second component of the system **II** is a 2-cycloamino-4-methylpyrimidine fragment linked through its position 5 to position 5 of the triazolopyrimidine bicycle. Such scaffolds present interest primarily as an inhibitor of the endothelin converting enzyme 1 [4] and vascular adhesion protein [5], and, consequently, can be considered as quite promising scaffolds for the design of new anticoagulants. Furthermore, 2-cycloamino-4-methylpyrimidines can act as an activator of the insulin-like growth factor receptor [6] and an inhibitor of some phosphodiesterases [7].

Three-component reactions of 1,3-dicarbonyl compounds with 3-amino-1,2,4-triazoles and aldehydes [8–12], orthoformates, or dimethylformamide dimethyl acetal (DMPDMA) [9, 13–15] are described in the literature and are convenient methods for the formation of polysubstituted triazolopyrimidine systems.

RESULTS AND DISCUSSION

In the present work we extended this approach to our earlier synthesized methyl 3-(2-R-4-methylpyrimidin-5-yl)-3-oxopropionates **1a** and **1b**, which were reacted with aromatic aldehydes **2a** and **2b** and 5-R-3-amino-1,2,4-triazoles **3a** and **3b** (Scheme 1). After prolonged

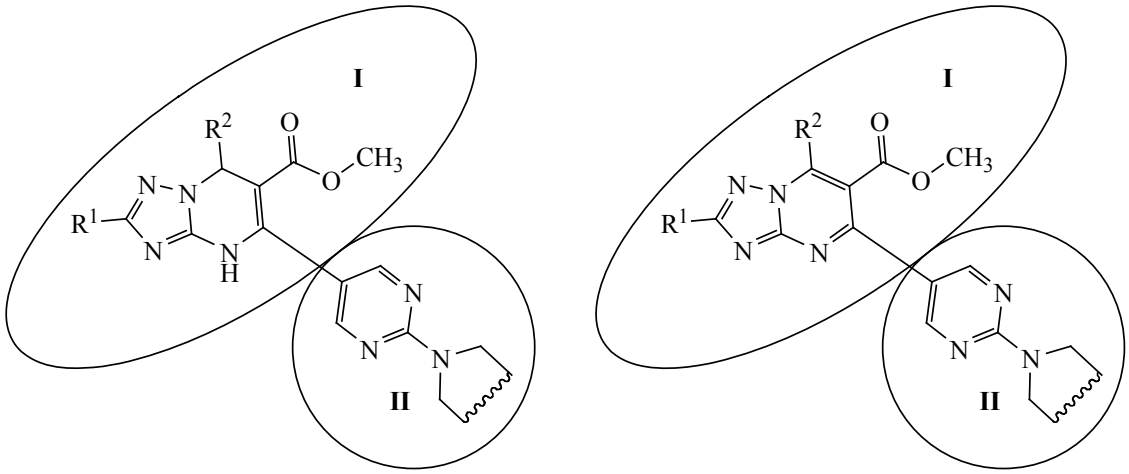
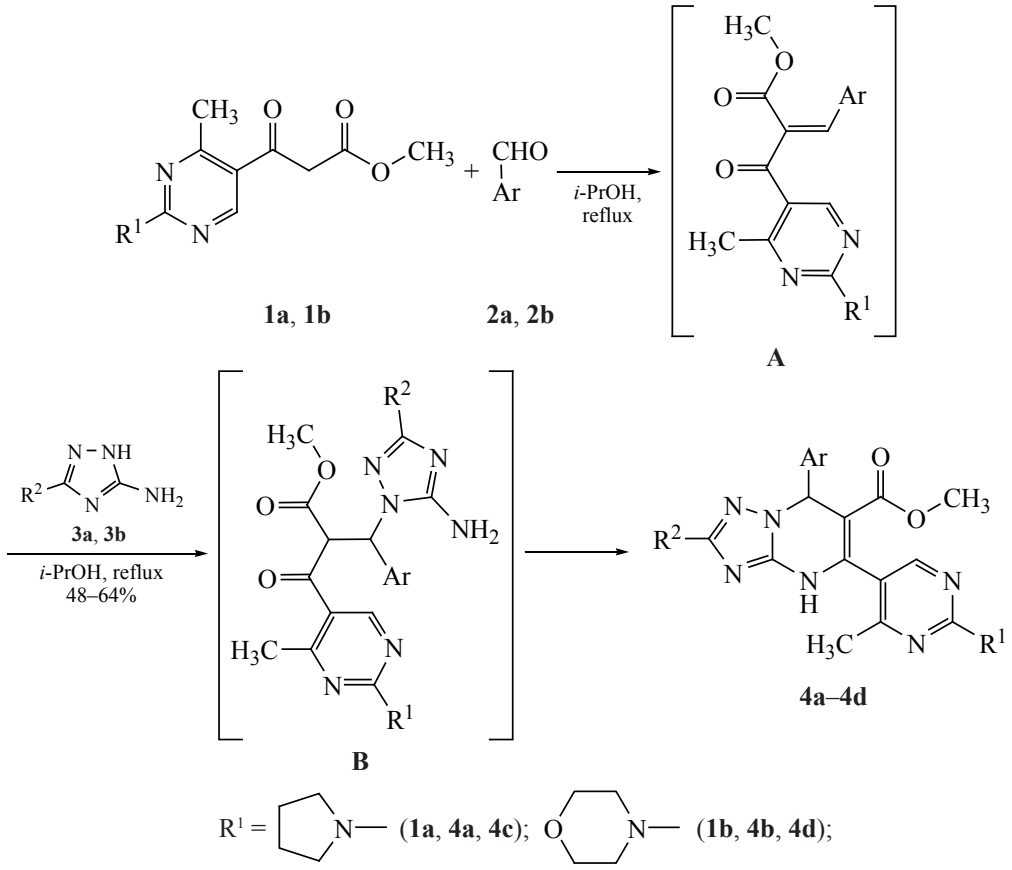


Fig. 1. Partially hydrogenated and heteroaromatic hybride 6-methoxyacrbonyl-7-(2-cycloamino-4-methylpyrimidin-5-yl)-2-R^1-1,2,4-triazolo[1,5-*a*]pyrimidine molecules.

refluxing of an equimolar mixture of the reagents in isopropanol or dioxane gives we isolated products **4a–4d** in yields of 48–64%. It was found that the nature of the solvent only slightly affects the yield and time of the reaction.

The ^{1}H NMR spectra of compounds **4a–4d** contain signals of the aromatic protons of the aryl rings at 6.88–7.60 ppm and characteristic proton signals of the methoxycarbonyl groups at ~3.70 ppm. The NH and CH proton signals of the newly formed pyrimidine ring

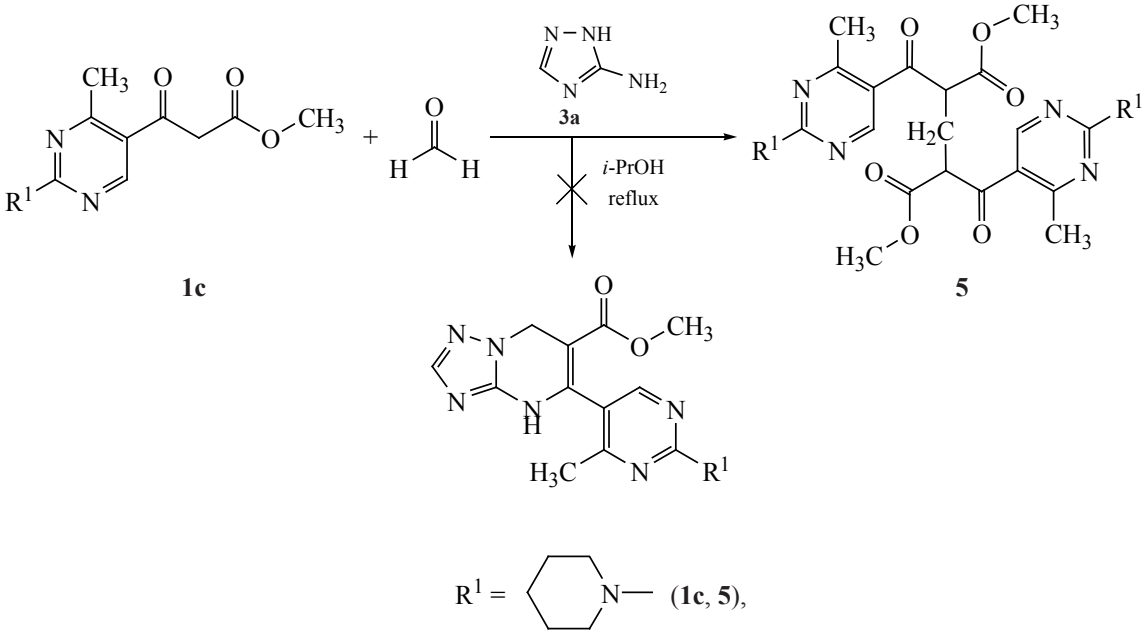
Scheme 1.



R^1 = (**1a**, **4a**, **4c**); (**1b**, **4b**, **4d**);

Ar = C_6H_5 (**2a**, **4a**, **4b**), 4-CH_3O-C_6H_4 (**2b**, **4c**, **4d**), R^2 = H (**3a**, **4a**, **4c**), CH_3 (**3b**, **4b**, **4d**).

Scheme 2.



appear at ~12.00 and 5.45 ppm, respectively. The proton signals of the triazole and pyrimidine rings are observed at 7.82 and 8.79 ppm, respectively. The mass spectra of compounds **4a–4d** contain protonated molecular ion peaks. These data allow the synthesized products to be assigned the structure of methyl 5-(4-methyl-2-R-pyrimidin-5-yl)-7-aryl-4,7-dihidro[1,2,4]triazolo[1,5-*a*]-pyrimidine-6-carboxylates **4a–4d**.

We suggest that the reaction starts with a Knoevenagel-type condensation of esters **1** with aldehydes to form intermediate arylidene derivatives **A**. The subsequent addition of the endocyclic NH group of aminotriazoles **3** at the C=C double bond of intermediate **A** leads to intermediate adduct **B**. The latter undergoes heterocyclization at the aminotriazole exocyclic amino group, yielding the final pyrimidin-5-yltriazolo[1,5-*a*]-pyrimidines **4**.

Attempted synthesis by this three-component condensation of 7-unsubstituted esters of 5-(4-methyl-2-R-pyrimidin-5-yl)-4,7-dihydro[1,2,4]triazolo[1,5-*a*]-pyrimidine-6-carboxylic acids, with formaldehyde as the aldehyde component, failed. It was found that the main product of this reaction was compound **5** formed by the reaction of formaldehyde with two molecules of methyl 3-[4-methyl-2-(piperidin-1-yl)pyrimidin-5-yl]-3-oxopropionate (**1c**) (Scheme 2).

The ^{1}H NMR spectrum of compound **5** contains exclusively characteristic proton signals of methyl 3-[4-methyl-2-(piperidin-1-yl)pyrimidin-5-yl]-3-oxopropionate fragments tethered by a methylene group, the protons of which give two multiplets at 1.95–2.02 and 2.15–2.19 ppm. The mass spectral data, too, provide evidence for the proposed dimethyl 2,4-bis[4-methyl-2-(piperidin-1-yl)pyrimidin-5-ylcarbonyl]glutarate (**5**) structure (Fig. 2).

With DMFDMA as an analog of the aldehyde component, the reactions of 3-(4-methyl-2-R-pyrimidin-5-yl)-3-oxopropionic acid esters **1a–1c** and 5-amino-1,2,4-triazoles **3a–3c** gave hardly separable mixtures of products. However, when the reactions were performed consecutively, we were able to direct the process to the formation of three-component reaction products, specifically methyl 7-(2-R-4-methylpyrimidin-5-yl)-2-R^1-[1,2,4]triazolo[1,5-*a*]pyrimidine-6-carboxylates **6a–6e** (Scheme 3).

The ^{1}H NMR spectra of compounds **6** display singlet signals of the methoxycarbonyl group protons at 3.65–3.78 ppm and proton signals of the newly formed pyrimidine ring at 8.82–9.39 ppm. In addition, the spectra show proton signals of the unsubstituted triazole ring (compounds **6a** and **6c**) at 8.24 ppm, and the spectra of the other derivatives show signals of the corresponding triazole substituents.

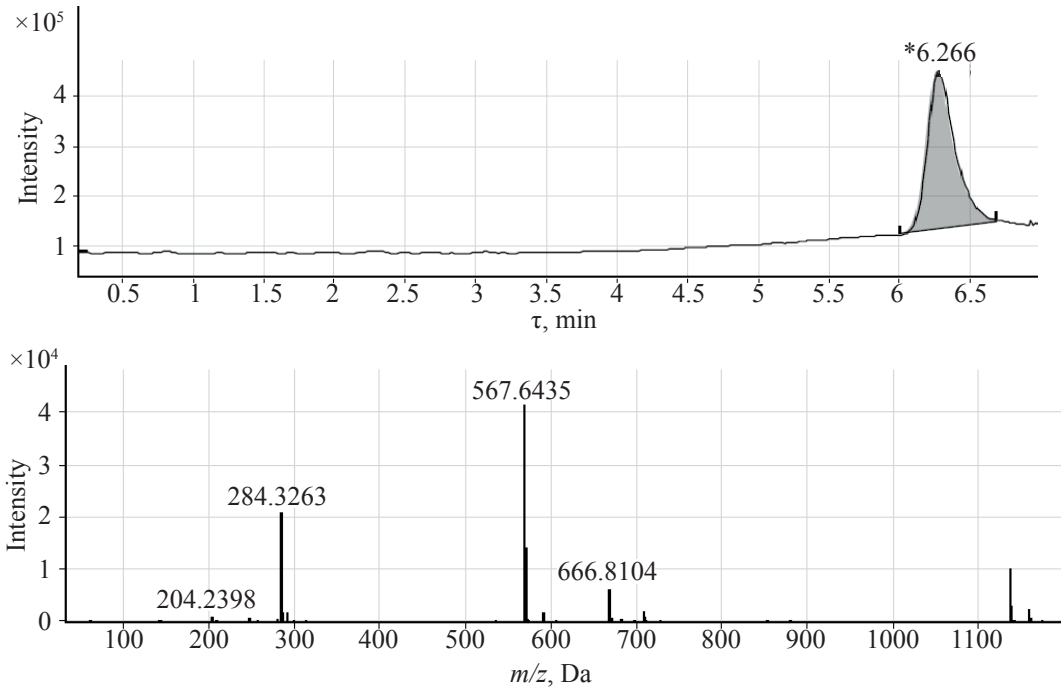


Fig. 2. HPLC–MS analysis of compound **5**.

It can be assumed that enaminone **B** formed at the first stage reacts with 5-aminotriazole with the elimination of dimethylamine, and, therewith, this reaction involves the most reactive exocyclic amino group of aminotriazole. Intermediate **C** formed at this stage undergoes cyclization into the final product **6** via the reaction of the endocyclic amino and carbonyl groups, with the elimination of a water molecule.

EXPERIMENTAL

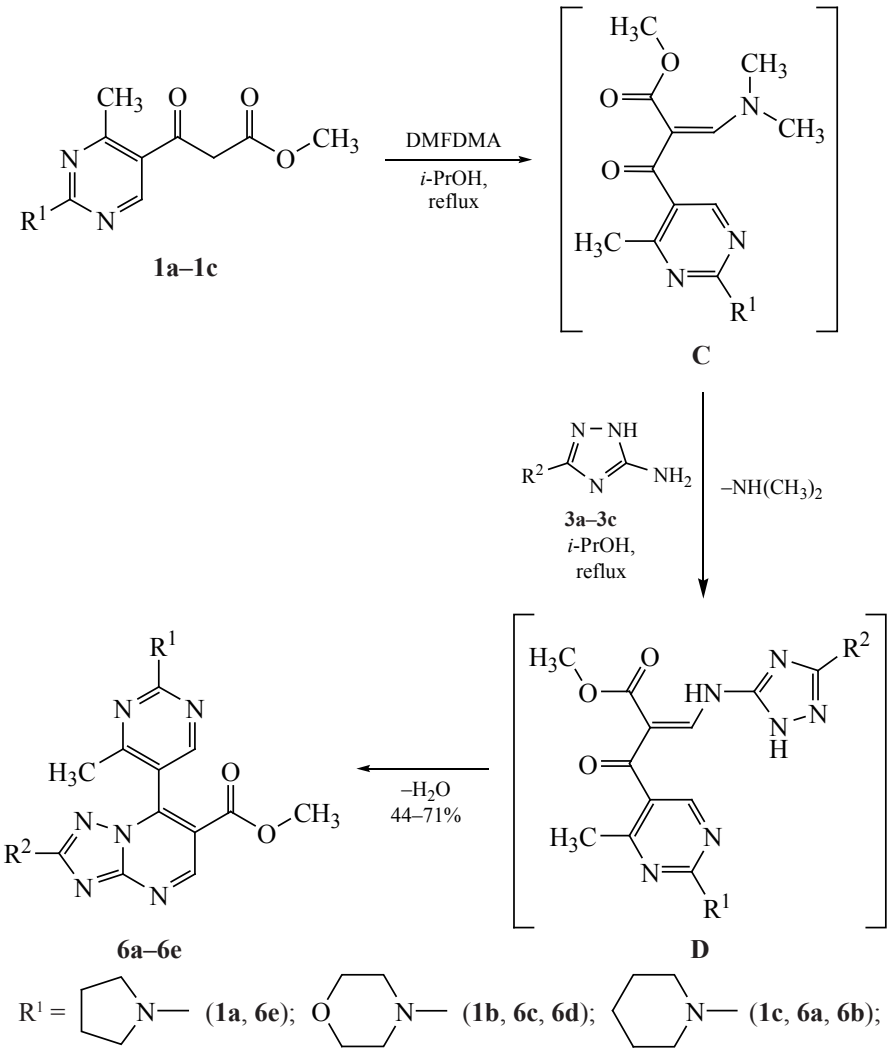
The ^{1}H NMR spectra of the synthesized compounds were registered on a Bruker DRX-500 spectrometer (500 MHz) in DMSO-d_6, internal reference TMS. The LC-MS analysis was performed on an Agilent Agilent 1260 Infinity 6230 TOF LC/MS system with a dual ESI source, operated in the positive ion mode; nebulizer gas (N_2) 20 psig, desiccant gas (N_2) 6 mL/min, 325°C; mass range is 50–2000 Da. Capillary voltage 4.0 kV, fragmentor voltage +191 V, skimmer voltage +66 V, and OctRF voltage 750 V. Chromatography conditions: Poroshell 120 EC-C18 column (4.6×50 mm, 2.7 μm), gradient elution acetonitrile–water (0.1% formic acid), flow rate 0.4 mL/min. An Agilent MassHunter Data Acquisition Software (version B.06.00) was used for data processing. The melting points were measured on a Stuart SMP30 apparatus. The purity of reagents and synthesized compounds, as well as reaction progress were monitored by TLC on Merck Silica gel 60 F_{254} plates, eluent $CHCl_3$–MeOH, 10 : 1 (spot development by exposure to UV light).

The starting 3-(2-R-4-methylpyrimidin-5-yl)-3-oxopropionic acid esters **1a**–**1c** were synthesized by the procedure described in our previous work [16].

Methyl 5-(4-methyl-2-R-pyrimidin-5-yl)-7-aryl-4,7-dihydro[1,2,4]triazolo[1,5-*a*]pyrimidine-6-carboxylates 4a–4d (*general procedure*). A mixture of 4 mmol of ester **1a** or **1b**, 4 mmol of aldehyde **2a** or **2b**, and 4 mmol of 3-amino-1,2,4-triazole **3a** or **3b** was heated under reflux in 5 mL ofisopropanol for 4 h. After cooling, a precipitate formed and was filtered off, washed with isopropanol, and recrystallized from isopropanol–DMF.

Methyl 5-[4-methyl-2-(pyrrolidin-1-yl)pyrimidin-5-yl]-7-phenyl-4,7-dihydro[1,2,4]triazolo[1,5-*a*]-pyrimidine-6-carboxylate (4a). Yield 1.07 g (64%), fine white needles, mp 195–197°C. ^{1}H NMR spectrum (DMSO-d_6), δ, ppm: 1.49–1.55 m (4H, $CH_2CH_{2pyrrolid}$), 2.59 s (3H, CH_3), 3.70 s (3H, CH_3O), 3.81–3.92 m (4H, CH_2NCH_2), 5.45 s (1H, CH), 6.93 t (1H_{arom}, *J* 7.8 Hz), 7.19–7.23 m (2H_{arom}), 7.58–7.61 m (2H_{arom}), 7.82 s (1H, H_{triaz}), 8.79 s (1H, $H_{pyrimid}$), 11.98 s (1H, NH). Mass spectrum, *m/z*: 418.1995 $[M + H]^+$. $C_{22}H_{23}N_7O_2$. $[M + H]^+$ 418.1987.

Scheme 3.



Methyl 2-methyl-5-[4-methyl-2-(morpholin-4-yl)pyrimidin-5-yl]-7-phenyl-4,7-dihydro[1,2,4]-triazolo[1,5-*a*]pyrimidine-6-carboxylate (4b). Yield 1.04 g (60%), fine white needles, mp 184–186°C. ^{1}H NMR spectrum (DMSO-d_6), δ, ppm: 2.06 s (3H, CH_3), 2.59 s (3H, CH_3), 3.70 s (3H, CH_3O), 3.82 t (4H, CH_2NCH_{2morph}, *J* 8.2 Hz), 3.98 t (4H, CH_2OCH_{2morph}, *J* 8.2 Hz), 5.45 s (1H, CH), 6.95 t ($1H_{arom}$, *J* 7.8 Hz), 7.22–7.25 m ($2H_{arom}$), 7.57–7.61 m ($2H_{arom}$), 8.79 s (1H, $H_{pyrimid}$), 11.98 s (1H, NH). Mass spectrum, *m/z*: 448.2098 $[M + H]^+$. $C_{23}H_{25}N_7O_3$. $[M + H]^+$ 448.2093.

Methyl 7-(4-methoxyphenyl)-5-[4-methyl-2-(pyrrolidin-1-yl)pyrimidin-5-yl]-4,7-dihydro[1,2,4]-triazolo[1,5-*a*]pyrimidine-6-carboxylate (4c). Yield 0.86 g (48%), fine white needles, mp 189–191°C. ^{1}H NMR spectrum (DMSO-d_6), δ, ppm: 1.49–1.55 m (4H, $CH_2CH_{2pyrrolid}$), 2.59 s (3H, CH_3), 3.60 s (3H, CH_3O), 3.70 s (3H, CH_3O), 3.81–3.92 m (4H, CH_2NCH_2), 5.45 s (1H, CH), 6.91 d ($2H_{arom}$, *J* 7.8 Hz), 7.21 d ($2H_{arom}$, *J* 7.8 Hz), 7.82 s ($1H_{triaz}$), 8.79 s (1H, $H_{pyrimid}$), 11.98 s (1H, NH) Mass spectrum, *m/z*: 448.2088 $[M + H]^+$. $C_{23}H_{25}N_7O_3$. $[M + H]^+$ 448.2093.

Methyl 7-(4-methoxyphenyl)-2-methyl-5-[4-methyl-2-(morpholin-4-yl)pyrimidin-5-yl]-4,7-dihydro[1,2,4]triazolo[1,5-*a*]pyrimidine-6-carboxylate (4d). Yield 1.03 g (54%), fine white needles, mp 189–200°C. ^{1}H NMR spectrum (DMSO-d_6), δ, ppm: 2.05 s (3H, CH_3), 2.59 s (3H, CH_3), 3.60 s (3H, CH_3O),

3.70 s (3H, CH_3O), 3.81 t (4H, CH_2NCH_{2morph}, *J* 8.2 Hz), 3.97 t (4H, CH_2OCH_{2morph}, *J* 8.2 Hz), 5.45 s (1H, CH), 6.90 d ($2H_{arom}$, *J* 7.8 Hz), 7.20 d ($2H_{arom}$, *J* 7.8 Hz), 8.78 s ($1H_{pyrimid}$), 11.97 s (1H, NH). Mass spectrum, *m/z*: 478.2194 $[M + H]^+$. $C_{24}H_{27}N_7O_4$. $[M + H]^+$ 478.2199.

Dimethyl 2,4-bis{[4-methyl-2-(pyrrolidin-1-yl)-pyrimidin-5-yl]carbonyl}glutarate (5). A mixture of 4 mmol of methyl 3-[4-methyl-2-(pyrrolidin-1-yl)-pyrimidin-5-yl]-3-oxopropanoate **1c**, 4 mmol of 40% formaldehyde, and 4 mmol of aminotriazole **3a** was heated under reflux in 5 mL of isopropanol for 2 h. After cooling, a precipitate formed and was filtered off and washed with isopropanol. Yield 1.03 g (54%), white amorphous material, mp 211–213°C. ^{1}H NMR spectrum (DMSO-d_6), δ, ppm: 1.49–1.71 m (12H, $2CH_2CH_2CH_{2piperid}$), 1.95–2.02 m (1H, CH_2), 2.15–2.19 m (1H, CH_2), 2.56 s (2H, $2CH_3$), 3.69 s (6H, $2CH_3O$), 3.80–3.94 m (8H, $2CH_2NCH_2$), 5.14–5.16 m (2H, 2CH), 8.59 s (2H, $H_{pyrimid}$). Mass spectrum, *m/z*: 567.2936 $[M + H]^+$. $C_{29}H_{38}N_6O_6$. $[M + H]^+$ 567.2928.

Methyl 7-[2-R-4-methylpyrimidin-5-yl]-1,2,4-triazolo[1,5-*a*]pyrimidine-6-carboxylate 6a–6e (*general procedure*). A mixture of 6 mmol of 3-(2-R-4-methylpyrimidin-5-yl)-3-oxopropionic acid ester **1a–1c** and 6 mmol of DMFDMA was heated under reflux in 6 mL of isopropanol for 2 h. After cooling, 5 mmol of the corresponding 5-amino-1,2,4-triazole **3a–3c** was added. The precipitate formed after cooling of the reaction mixture was filtered off, washed with isopropanol, and recrystallized from isopropanol–DMF.

Methyl 7-[4-methyl-2-(piperidin-1yl)pyrimidin-5-yl]-1,2,4-triazolo[1,5-*a*]pyrimidine-6-carboxylate (6a). Yield 1.20 g (59%), white amorphous material, mp 182–185°C. ^{1}H NMR spectrum (DMSO-d_6), δ, ppm: 1.51–1.56 m (6H, $CH_2CH_2CH_2$), 2.16 s (3H, CH_3), 3.65 t (4H, CH_2NCH_2, *J* 5.0 Hz), 3.74 s (3H, CH_3O), 8.24 s (1H, CH_{triaz}), 8.73 s (1H, $CH_{pyrimid}$), 9.31 s (1H, $CH_{pyrimid}$). Mass spectrum, *m/z*: 354.1670 $[M + H]^+$. $C_{17}H_{19}N_7O_2$. $[M + H]^+$ 354.1674.

Methyl 7-[4-methyl-2-(piperidin-1yl)pyrimidin-5-yl]-2-phenyl-1,2,4-triazolo[1,5-*a*]pyrimidine-6-carboxylate (6b). Yield 1.53 g (69%), white amorphous material, mp 268–270°C. ^{1}H NMR spectrum (DMSO-d_6), δ, ppm: 1.51–1.57 m (6H, $CH_2CH_2CH_2$), 2.16 s (3H, CH_3), 3.76 t (4H, CH_2NCH_2, *J* 5.0 Hz), 3.73 s (3H, CH_3O), 7.02 t ($1H_{arom}$, *J* 7.4 Hz), 7.31 t ($2H_{arom}$, *J* 8.1 Hz), 7.73 d ($2H_{arom}$, *J* 8.1 Hz), 8.78 s (1H, $CH_{pyrimid}$), 9.39 s (1H, $CH_{pyrimid}$). Mass spectrum, *m/z*: 430.1995 $[M + H]+$. $C_{23}H_{23}N_7O_2$. $[M + H]^+$ 430.1987.

Methyl 7-[4-methyl-2-(morpholin-4-yl)pyrimidin-5-yl]-1,2,4-triazolo[1,5-*a*]pyrimidine-6-carboxylate (6c). Yield 1.13 g (53%), white amorphous material, mp 268–270°C. ^{1}H NMR spectrum (DMSO-d_6), δ, ppm: 2.14 s (3H, CH_3), 3.72 t (4H, CH_2NCH_2, *J* 5.0 Hz), 3.78 s (3H, CH_3O), 3.84 t (4H, CH_2OCH_2, *J* 5.0 Hz), 8.24 s (1H, CH_{triaz}), 8.75 s (1H, $CH_{pyrimid}$), 8.82 s (1H, $CH_{pyrimid}$). Mass spectrum, *m/z*: 356.1466 $[M + H]+$. $C_{16}H_{17}N_7O_3$. $[M + H]^+$ 356.1466.

Methyl 7-[4-methyl-2-(morpholin-4-yl)pyrimidin-5-yl]-2-phenyl-1,2,4-triazolo[1,5-*a*]pyrimidine-6-carboxylate (6d). Yield 1.53 g (71%), white amorphous material, mp 202–204°C. ^{1}H NMR spectrum (DMSO-d_6), δ, ppm: 2.13 s (3H, CH_3), 3.75 t (4H, CH_2NCH_2, *J* 5.0 Hz), 3.78 s (3H, CH_3O), 3.86 t (4H, CH_2OCH_2, *J* 5.5 Hz), 7.12 t ($1H_{arom}$, *J* 7.4 Hz), 7.40 t ($2H_{arom}$, *J* 8.1 Hz), 7.76 d ($2H_{arom}$, *J* 8.1 Hz), 8.21 s (1H, $CH_{pyrimid}$), 8.84 s (1H, $CH_{pyrimid}$). Mass spectrum, *m/z*: 432.1788 $[M + H]^+$. $C_{22}H_{21}N_7O_3$. $[M + H]^+$ 432.1780.

Methyl 7-[4-methyl-2-(pyrrolidin-1-yl)pyrimidin-5-yl]-2-(methylsulfanyl)-1,2,4-triazolo[1,5-*a*]pyrimidine-6-carboxylate (6e). Yield 1.01 g (44%), white amorphous material, mp 161–163°C. ^{1}H NMR spectrum (DMSO-d_6), δ, ppm: 1.53–1.60 m (4H, CH_2CH_2), 2.15 s (3H, CH_3), 2,38 s (3H, CH_3S), 3.78 t (4H, CH_2NCH_2, *J* 5.8 Hz), 3.77 s (3H, CH_3O), 8.76 s (1H, $CH_{pyrimid}$), 8.87 s (1H, $CH_{pyrimid}$). Mass spectrum, *m/z*: 386.1388 $[M + H]^+$. $C_{17}H_{19}N_7O_2S$. $[M + H]^+$ 386.1395.

CONCLUSIONS

A convenient synthetic approach to polysubstituted dehydrogenated or heteroaromatic 1,2,4-triazolo[1,5-*a*]-pyrimidines containing containing at position 5 a 4-methylpyrimidine moiety bearing a cycloamino substituent at position 2 and linked to the triazolopyrimidine bicycle through its position 5 was developed. This allows creation of a target library of new hybrid molecules containing in their structure two privileged scaffolds, one of which has an antibacterial and the other an anticoagulant activity.

FUNDING

The study was carried out with a grant from the Russian Science Foundation (project no. 18-74-10097).

CONFLICT OF INTEREST

The authors declare no conflict of interest.

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