THREE-COMPONENT SYNTHESIS OF 1-ARYL-1,2,3,4-TETRAHYDROPYRIMIDO[1,2-*a*][1,3,5]TRIAZINE-6-ONES

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Keywords: 2-Arylamino-3H-pyrimidine-4-ones; three-component reaction; aliphatic amines; 1,2,3,4-tetrahydropyrimido[1,2-*a*][1,3,5]triazine-6-ones.

This study is concerned with a three-component method of synthesis of new 1,2,3,4-tetrahydro-6H-pyrimido[1,2-*a*][1,3,5]triazin-6-ones based on 2-amino-3H-pyrimidine-4-ones, aliphatic amines and formaldehyde. Conditions of reaction were optimized.

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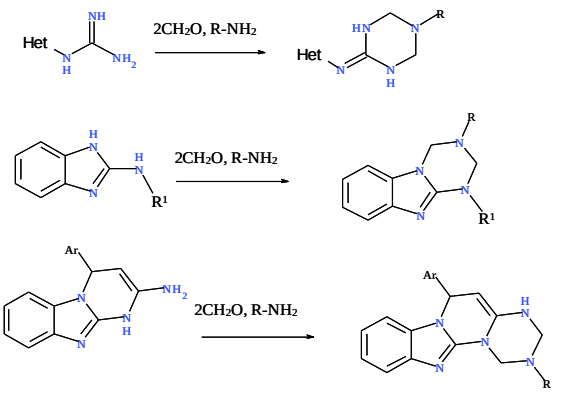
Introduction

Over the last years condensed 1,3,5-triazines are found in the center of medicinal chemistry due to the opportunity of building structurally diverse compounds, exhibited various kinds of biological activity.[1] However, pyrimido[1,2-*a*]triazines were studied insufficiently and in the literature reported only a few number of methods of preparation.[2] For some compounds of this series, antibacterial and antifungal activities were found. Recently, the synthesis of partly hydrogenized pyrimido[1,2-*a*]triaxines[3] capable of inhibit cancer cell growth.[4]

We have previously reported[5,6] about the three-component methods of synthesis of 1,3,5-hexahydrotriazines from hetarylguanidines and its benzimodazo[1,2-*a*]condensed derivatives on the basis of N-alkyl(aryl)amino-benzimidazoles (scheme 1).



Scheme 1. Synthesis of triazines.

Development for annelation methods of triazine cycle to various heterocyclic substrate having 1,3-dinuclepphilic centers in their structure has been undoubted interest. The aim of this work is to develop a one-pot method of synthesis of tetrahydropyrimido[1,2-*a*][1,3,5]triazine system designed in the framework of a program for searching of new antibacterial, anti-inflammatory and antitumor medicines.

Experimental part

General

All commercial reagents were purchased from Bekton, Lancaster, Acros, Aldrich, and Sigma and were used as received without further purification. The course of the reactions and purities of the compounds were monitored by thin layer chromatography (TLC) on SILUFOL UV-254 plates, eluent: chloroform, methanol in different proportions and spots were visualized by exposure to iodine vapours. The ^{1}H-NMR spectra were recorded on a *Bruker AM-300* spectrometer operating at 300.13 MHz, using DMSO-d_6 as solvent and TMS as internal standard. Elemental analyses were determined by using a *Carlo Erba NA 1500* elemental analysis instrument. Melting points were recorded using *Stuart SMP30* melting point instrument.

General procedure for the synthesis of 1,2,3,4-tetrahydro-6H-pyrimido[1,2-*a*][1,3,5]triazin-6-ones (3a-p)

To a suspension of 40 mmol of 2-aminopyrimidinone **1** in 3 ml of ethanol was added 40 mmol of aliphatic amine **2** and 80 mmol 37 % of aqueous formaldehyde solution. The mixture was refluxed until complete dissolving of pyrimidine compound (2-3 hours). When the reaction was completed, the reaction mixture was cooled to room temperature; the obtained precipitate was filtered off and recrystallized from isopropyl alcohol.

^{1}H-NMR spectra of tetrahydropyrimido[1,2-a][1,3,5]triazin-6-ones (**3a-p**), δ in ppm (J in Hz) are the followings:

3-(2-Methoxyethyl)-7,8-dimethyl-1-phenyl-1,2,3,4-tetrahydro-6H-pyrimido[1,2-*a*][1,3,5]triazin-6-one (3a)

Yield = 64 %, M.p.= 95-97 ºC. ^{1}H NMR (DMSO-d_6): δ = 1.87 (s., 3H, CH_3-C7); 2.02 (s., 3H, CH_3-C8); 2.99 (t., J=8.8, 2H, N(3)-CH_2); 3.32 (s, 3H, O-CH_3); 3.59 (t, J=8.8, 2H, O-CH_2);4.72 (s., 2H, CH_2-triazine); 4.98 (s, 2H, CH_2- triazine); 7.22-7.32 (m, 3H, arom.); 7.34-7.40 (m, 2H, arom.). Anal. Calcd. for $C_{17}H_{22}N_4O_2$: C, 64.95; H, 7.05; N, 17.82. Found: C, 65.11; H, 7.03; N, 17.85.

3-Benzyl-7,8-dimethy-1-phenyl-1,2,3,4-tetrahydro-6H-pyrimido[1,2-*a*][1,3,5]triazin-6-one (3b)

Yield = 85 %, M.p. = 142-144 ºC. ^{1}H NMR (DMSO-d_6): δ = 1.88 (s., 3H, CH_3-C7); 2.01 (s., 3H, CH_3-C8); 3.73 (s., 2H, CH_2-C7); 3.82 (s., 3H, O-CH_3); 4.03 (s., 2H, N(3)-CH_2); 4.73 (s., 2H, CH_2-triazine); 5.03 (s., 2H, CH_2-triazine); 6.88-6.97 (m., 2H, arom.); 7.06-7.19 (m., 4H, arom.); 7.20-7.28 (m, 4H, arom.). Anal. Calcd. for $C_{21}H_{22}N_4O$: C, 72.81; H, 6.40; N, 16.17. Found: C, 72.73; H, 6.37; N, 16.14.

3-(2-Furylmethyl)-7,8-dimethyl-1-phenyl-1,2,3,4-tetrahydro-6H-pyrimido[1,2-*a*][1,3,5]triazin-6-one (3c)

Yield = 63 %, M.p. = 141-143 ˚C. ^{1}H NMR (DMSO-d_6): δ = 1.89 (s., 3H, CH_3-C7); 2.01 (s., 3H, CH_3-C8); 4.02 (s., 2H, N(3)-CH_2) 4.68 (s., 2H, CH_2-triazine); 5.02 (s., 2H, CH_2-triazine); 6.24 (d, J=3.2, 1H, H-furane); 6.36 (d.d., J=3.2, J=2.0, 1H, H-furane); 7.25-7.34 (m, 3H, arom.); 7.35-7.41 (m, 2H, arom.); 7.49 (d, J=1.7, 1H, H-furane). Anal. Calcd. for $C_{19}H_{20}N_4O_2$: C, 67.84; H, 5.99; N, 16.65. Found: C, 67.77; H, 6.00; N, 16.61.

7-Benzyl-3-(2-methoxyethyl)-8-methyl-1-phenyl-1,2,3,4-tetrahydro-6H-pyrimido[1,2-*a*][1,3,5]triazin-6-one (3d)

Yield = 64 %, M.p. = 122-124 ºC. ^{1}H NMR (DMSO-d_6): δ = 1.99 (s., 3H, CH_3-C8); 3.00 (t., J=8.8, 2H, N(3)-CH_2); 3.32 (s, 3H, O-CH_3); 3.58 (t, J=8.8, 2H, O-CH_2); 3.71 (s., 2H, CH_2-C7); 4.60 (s., 2H, CH_2-triazine); 5.03 (s, 2H, CH_2-triazine); 7.09-7.16 (m, 2H, arom.); 7.35-7.48 (m, 8H, arom.). Anal. Calcd. for $C_{23}H_{26}N_4O_2$: C, 70.75; H, 6.71; N, 14.35. Found: C, 70.27; H, 6.73; N, 14.31.

1-(4-Fluorophenyl)-3-(2-methoxyethyl)-7,8-dimethyl-1,2,3,4-tetrahydro-6H-pyrimido[1,2-*a*][1,3,5]triazin-6-one (3e)

Yield = 72 %, M.p. = 92-94 ºC. ^{1}H NMR (DMSO-d_6): δ = 1.88 (s., 3H, CH_3-C7); 2.09 (s., 3H, CH_3-C8); 3.02 (t., J=8.8, 2H, N(3)-CH_2); 3.32 (s, 3H, O-CH_3); 3.59 (t, J=8.8, 2H, O-CH_2);4.71 (s., 2H, CH_2-triazine); 4.97 (s, 2H, CH_2-triazine); 7.09-7.16 (m, 2H, arom.); 7.28-7.35 (m, 2H, arom.). Anal. Calcd. for $C_{17}H_{21}FN_4O_2$: C, 61.43; H, 6.37; N, 16.86. Found: C, 61.56; H, 6.39; N, 16.91.

1-(4-Fluorophenyl)-3-(2-furylmethyl)-7,8-dimethyl-1,2,3,4-tetrahydro-6H-pyrimido[1,2-*a*][1,3,5]triazin-6-one (3f)

Yield = 79 %, M.p. = 133-135 ºC. ^{1}H NMR (DMSO-d_6): δ = 1.89 (s., 3H, CH_3-C7); 2.10 (s., 3H, CH_3-C8); 4.04 (s., 2H, N(3)-CH_2); 4.70 (s., 2H, CH_2-triazine); 4.96 (s, 2H, CH_2- triazine); 6.25 (d, J=3.2, 1H, H-furane); 6.35 (d.d., J=3.2, J=2.0, 1H, H-furane); 7.10-7.15 (m, 2H, arom.); 7.27-7.34 (m, 2H, arom.); 7.48 (d, J=1.7, 1H, H-furane). Anal. Calcd. for $C_{19}H_{19}FN_4O_2$: C, 64.40; H, 5.40; N, 15.81. Found: C, 64.54; H, 5.40; N, 15.78.

7-Benzyl-1-(4-fluorophenyl)-8-methyl-3-(3-pyridinylmethyl)-1,2,3,4-tetrahydro-6H-pyrimido[1,2-*a*][1,3,5]triazin-6-one (3g)

Yield = 75 %, M.p. = 143-145 ºC. ^{1}H NMR (DMSO-d_6): δ = 2.02 (s., 3H, CH_3-C8); 3.72 (s., 2H, CH_2-C7); 4.07 (s., 2H, N(3)-CH_2); 4.73 (s., 2H, CH_2-triazine); 4.95 (s, 2H, CH_2- triazine); 6.95-7.07 (m, 2H, arom.); 7.11-7.19 (m., 2H, arom.); 7.20-7.29 (m, 5H, arom.); 7.42 (t., J=7.8, CH-pyridine); 7.75 (d., J=7.8, CH-pyridine); 8.49 (d., J=7.4, CH-pyridine); 8.54 (s., CH-pyridine). Anal. Calcd. for $C_{26}H_{24}FN_5O$: C, 70.73; H, 5.48; N, 15.86. Found: C, 70.67; H, 5.50; N, 15.82.

7-Benzyl-1-(4-fluorophenyl)-3-(2-furylmethyl)-8-methyl-1,2,3,4-tetrahydro-6H-pyrimido[1,2-*a*][1,3,5]triazin-6-one (3h)

Yield = 70 %, M.p. = 140-142 ºC. ^{1}H NMR (DMSO-d_6): δ = 2.03 (s., 3H, CH_3-C8); 3.72 (s., 2H, CH_2-C7); 4.03 (s., 2H, N(3)-CH_2); 4.72 (s., 2H, CH_2-triazine); 4.96 (s, 2H, CH_2- triazine); 6.26 (d, J=3.2, 1H, H-furane); 6.34 (d.d., J=3.2, J=2.0, 1H, H-furane); 6.94-7.06 (m, 2H, arom.); 7.10-7.18 (m., 2H, arom.); 7.21-7.30 (m, 5H, arom.); 7.47 (d, J=1.7, 1H, H-furane). Anal. Calcd. for $C_{25}H_{23}FN_4O_2$: C, 69.75; H, 5.39; N, 13.01. Found: C, 70.02; H, 5.41; N, 12.98.

3-(2-Methoxyethyl)-1-(2-methoxyphenyl)-7,8-dimethyl-1,2,3,4-tetrahydro-6H-pyrimido[1,2-*a*][1,3,5]triazin-6-one (3i)

Yield = 68 %, M.p. = 94-96 ºC. ^{1}H NMR (DMSO-d_6): δ = 1.86 (s., 3H, CH_3-C7); 1.97 (s., 3H, CH_3-C8); 2.98 (t., J=8.8, 2H, N(3)-CH_2); 3.32 (s, 3H, O-CH_3); 3.59 (t, J=8.8, 2H, O-CH_2); 3.80 (s., 3H, O-CH_3); 4.47 (s., 2H, CH_2-triazine); 5.03 (s., 2H, CH_2-triazine); 6.95 (t, J=8.3, 1H, arom.); 7.06 (d., J=7.9 1H, arom.); 7.22-7.31 (m, 2H, arom.). Anal. Calcd. for $C_{18}H_{24}N_4O_3$: C, 62.77; H, 7.02; N, 16.27. Found: C, 62.58; H, 7.04; N, 16.23.

1-(2-Methoxyphenyl)-7,8-dimethyl-3-(3-pyridinylmethyl)-1,2,3,4-tetrahydro-6H-pyrimido[1,2-*a*][1,3,5]triazin-6-one (3j)

Yield = 81 %, M.p. = 154-156 ºC. ^{1}H NMR (DMSO-d_6): δ = 1.87 (s., 3H, CH_3-C7); 1.98 (s., 3H, CH_3-C8); 3.81 (s., 3H, O-CH_3); 4.09 (s., 2H, N(3)-CH_2) 4.48 (s., 2H, CH_2-triazine); 5.04 (s., 2H, CH_2-triazine); 6.97 (t, J=8.3, 1H,

arom.); 7.07 (d., J=7.9 1H, arom.); 7.24-7.37 (m, 3H, arom.+CH-pyridine); 7.76 (d., J=7.8, CH-pyridine); 8.48 (d., J=7.4, CH-pyridine); 8.54 (s., CH-pyridine). Anal. Calcd. for $C_{21}H_{23}N_5O_2$: C, 66.83; H, 6.14; N, 18.55. Found: C, 66.75; H, 6.16; N, 18.58.

7-Benzyl-1-(2-methoxyphenyl)-3-(3-methoxypropyl)-8-methyl-1,2,3,4-tetrahydro-6H-pyrimido[1,2-*a*][1,3,5]triazin-6-one (3k)

Yield = 61 %, M.p. = 93-95 °C. ^{1}H NMR (DMSO-d_6): δ = 1.61-1.68 (m., 2H, CH_2-CH_2-CH_2); 2.02 (s., 3H, CH_3-C8); 2.95 (t., J=8.9, 2H, N(3)-CH_2); 3.30 (s, 3H, O-CH_3); 3.44 (t., J=8.9, 2H, O-CH_2); 3.70 (s., 2H, CH_2-C7); 3.81 (s., 3H, O-CH_3); 4.50 (s., 2H, CH_2-triazine); 5.02 (s., 2H, CH_2-triazine); 6.96 (t, J=8.3, 1H, arom.); 7.08-7.15 (m., 3H, arom.); 7.20-7.35 (m, 5H, arom.). Anal. Calcd. for $C_{25}H_{30}N_4O_3$: C, 69.10; H, 6.96; N, 12.89. Found: C, 68.87; H, 6.98; N, 12.93.

7-Benzyl-3-(2-furylmethyl)-1-(2-methoxyphenyl)-8-methyl-1,2,3,4-tetrahydro-6H-pyrimido[1,2-*a*][1,3,5]triazin-6-one (3l)

Yield = 77 %, M.p. = 144-146 °C. ^{1}H NMR (DMSO-d_6): δ = 2.04 (s., 3H, CH_3-C8); 3.70 (s., 2H, CH_2-C7); 3.81 (s., 3H, O-CH_3); 4.04 (s., 2H, N(3)-CH_2); 4.49 (s., 2H, CH_2-triazine); 5.03 (s., 2H, CH_2-triazine); 6.25 (d, J=3.2, 1H, H-furane); 6.35 (d.d., J=3.2, J=2.0, 1H, H-furane); 6.97 (t, J=8.3, 1H, arom.); 7.07-7.14 (m., 3H, arom.); 7.19-7.33 (m, 5H, arom.); 7.46 (d, J=1.7, 1H, H-furane). Anal. Calcd. for $C_{26}H_{26}N_4O_3$: C, 70.57; H, 5.92; N, 12.66. Found: C, 70.68; H, 5.93; N, 12.63.

3-(2-Hydroxyethyl)-1-(4-methoxyphenyl)-7,8-dimethy-1,2,3,4-tetrahydro-6H-pyrimido[1,2-*a*][1,3,5]triazin-6-one (3m)

Yield = 84 %, M.Pt = 161-163 °C. ^{1}H NMR (DMSO-d_6): δ = 1.89 (s., 3H, CH_3-C7); 1.98 (s., 3H, CH_3-C8); 2.96 (t., J=8.8, 2H, N(3)-CH_2); 3.58 (t, J=8.8, 2H, O-CH_2); 3.81 (s., 3H, O-CH_3); 4.61 (bro. m., 1H, OH); 4.69 (s., 2H, CH_2-triazine); 5.07 (s., 2H, CH_2-triazine); 6.90 (d, J=7.9, 2H, arom.); 7.16 (d., J=7.9, 2H, arom.). Anal. Calcd. for $C_{17}H_{22}N_4O_3$: C, 61.80; H, 6.71; N, 16.96. Found: C, 61.69; H, 6.72; N, 16.91.

1-(4-Methoxyphenyl)-7,8-dimethy-3-[2-(4-morpholinyl)ethyl]-1,2,3,4-tetrahydro-6H-pyrimido[1,2-*a*][1,3,5]triazin-6-one (3n)

Yield = 60 %, M.p. = 112-114 °C. ^{1}H NMR (DMSO-d_6): δ = 1.89 (s., 3H, CH_3-C7); 1.97 (s., 3H, CH_3-C8); 2.48 (t., J=8.9, 4H, $(CH_2)_2$N); 2.67 (t., J=8.8, 2H, CH_2-N); 2.98 (t., J=8.8, 2H, N(3)-CH_2); 3.51 (t., J=8.9, 4H, $(CH_2)_2$O); 3.80 (s., 3H, O-CH_3); 4.69 (s., 2H, CH_2-triazine); 5.08 (s., 2H, CH_2-triazine); 6.91 (d, J=7.9, 2H, arom.); 7.15 (d., J=7.9, 2H, arom.). Anal. Calcd. for $C_{21}H_{29}N_5O_3$: C, 63.14; H, 7.32; N, 17.53. Found: C, 63.38; H, 7.35; N, 17.49.

7-Benzyl-3-(2-furylmethyl)-1-(4-methoxyphenyl)-8-methy-3-(3-pyridinylmethyl)-1,2,3,4-tetrahydro-6H-pyrimido[1,2-*a*][1,3,5]-triazin-6-one (3o)

Yield = 85 %, M.p. = 147-149 °C. ^{1}H NMR (DMSO-d_6): δ = 2.02 (s., 3H, CH_3-C8); 3.72 (s., 2H, CH_2-C7); 3.83 (s., 3H, O-CH_3); 4.02 (s., 2H, N-CH_2); 4.58 (s., 2H, CH_2-triazine); 5.06 (s., 2H, CH_2-triazine); 6.29 (d, J=3.2, 1H, H-furane); 6.38 (d.d., J=3.2, J=2.0, 1H, H-furane);6.90 (d, J=7.9, 2H, arom.); 7.10-7.18 (m., 2H, arom.); 7.17-7.26 (m, 5H, arom.); 7.47 (d, J=1.7, 1H, H-furane). Anal. Calcd. for $C_{26}H_{26}N_4O_3$: C, 70.57; H, 5.92; N, 12.66. Found: C, 70.69; H, 5.94; N, 12.70.

7-Benzyl-1-(4-methoxyphenyl)-8-methy-3-(3-pyridinylmethyl)-1,2,3,4-tetrahydro-6H-pyrimido[1,2-*a*][1,3,5]triazin-6-one (3p)

Yield = 88 %, M.p. = 153-155 °C. ^{1}H NMR (DMSO-d_6): δ = 2.01 (s., 3H, CH_3-C8); 3.73 (s., 2H, CH_2-C7); 3.82 (s., 3H, O-CH_3); 4.09 (s., 2H, N-CH_2); 4.57 (s., 2H, CH_2-triazine); 5.08 (s., 2H, CH_2-triazine); 6.90 (d, J=7.9, 2H, arom.); 7.10-7.18 (m., 2H, arom.); 7.19-7.28 (m, 5H, arom.); 7.43 (t., J=7.8, CH-pyridine); 7.76 (d., J=7.8, CH-pyridine); 8.49 (d., J=7.4, CH-pyridine); 8.54 (s., CH-pyridine). Anal. Calcd. for $C_{27}H_{27}N_5O_2$: C, 71.50; H, 6.00; N, 15.44. Found: C, 71.39; H, 6.02; N, 15.41.

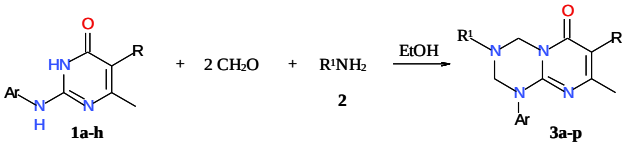
Results and discussions

2-Arylaminopyrimidine-4-ones derivatives **1**, can easily be prepared by the condensation reaction of arylguanidines with 2-alkylacetoacetic esters consists of 1,3-N-C-N binucleophilic fragment in their structure that allows to consider them as promising building blocks for various condensed systems.

Previously we reported a method for annelation of 1,3,5-triazine cycle to 2-aminobenzimidazoles or 2-amino-1,4-dihydro[1,3,5]triazino[1,2-*a*]benzimidazoles with formaldehyde and primary amines.[6] This method is successfully extended to series of 2-arylaminoperimidine-4-ones **1a-h**. It was found that three-component interaction allows to obtain a series of new 7-R-3-R^1-1-aryl-1,2,3,4-tetrahydropyrimido[1,2-*a*][1,3,5]triazine-6-ones **3a-p** in one synthetic step. Although the reaction is more smoothly than in case of the previously studied systems, this reaction can be proceeded under refluxing of equimolar mixture of reagents in ethanol (Scheme 2). Perimido[1,2-*a*][1,3,5]triazine-6-ones **3a-p** obtained were isolated in 60-88 % yield from reaction mass under cooling.

In the NMR ^{1}H spectra of **3a-p** compounds there are no signals of exo- and endocyclic amino groups. Two singlets of methylene group as well as proton signals of corresponded aliphatic amines moieties could unambiguously assigned. Characteristic signals of two methylene groups of tetrahydrotriazines cycle could be observed as two singlets at 4.48-4.73 and 4.91-5.08 ppm.

The compounds **3a-p** are colorless crystalline substances with district melting points.

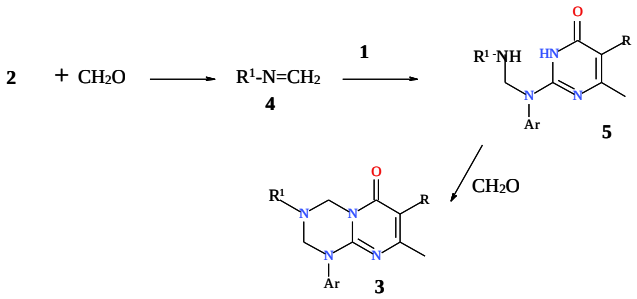


Ar=Ph, R=CH$_3$ (**1a, 3a-c**); Ar=Ph, R=C$_6$H$_5$CH$_2$ (**1b, 3d**); Ar=4-FC$_6$H$_4$, R=CH$_3$ (**1c, 3e,f**); Ar=4-FC$_6$H$_4$, R=C$_6$H$_5$CH$_2$ (**1d, 3g,h**); Ar=2-CH$_3$OC$_6$H$_4$, R=CH$_3$ (**1e, 3i,j**); Ar=2-CH$_3$OC$_6$H$_4$, R=C$_6$H$_6$CH$_2$ (**1f, 3k,l**); Ar=4-CH$_3$OC$_6$H$_4$, R=CH$_3$ (**1g, 3m,n**); Ar=CH$_3$OC$_6$H$_4$, R=C$_6$H$_5$CH$_2$ (**1h,3o,p**).

R^1=CH$_3$OCH$_2$CH$_2$ (**2a, 3a,d,e,i**), R^1=C$_6$H$_5$CH$_2$ (**2b, 3b**), R^1=2-FurCH$_2$ (**2c,3c,f,h,l,o**), R^1=3-PyrCH$_2$ (**2d, 3g,j,p**), R^1=CH$_3$OCH$_2$CH$_2$CH$_2$ (**2e, 3k**), R^1=HOCH$_2$CH$_2$ (**2f, 3m**), R^1=MorphCH$_2$CH$_2$ (**2, 3n**).

Scheme 2. Synthesis of tetrahydropyrimidotriazinones **3a-p**

A possible reaction mechanism is a route with stepwise reactions involving formation of various reactive intermediates. The first step is supposed to be a Schiff base formation (**4**) in the condensation reaction of formaldehyde and the primary amines. Addition of this intermediate to the aminopyrimidone (**1**) may give an intermediate aminomethylene derivative (**5**) which further condensation with a second formaldehyde molecule leads to ring closure into product **3** (Scheme 3).



Scheme 3. Plausible reaction mechanism for the synthesis of **3**.

Conclusions

Three-component annelation method of tetrahydrotriazine cycle to aminopyrmidone derivatives was developed. Series of new 7-R-3-R^1-1-aryl-1,2,3,4-tetrahydropyrimido[1,2-a][1,3,5]triazine-6-ones synthesized will be investigated for their biological activity.

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