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СОЛИ ТРОПИЛИЯ И ТРИТИЛИЯ В РЕАКЦИЯХ С 2-АМИНО-4,6-ДИЗАМЕЩЕННЫМИ ПИРИМИДИНАМИ

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Взаимодействие тетрафторборатов тропилия или тритилия с 2-амино-4,6-диметилпиримидином в среде этанола при комнатной температуре приводит, соответственно, к 4,6-диметил-2-(N-циклогепта-2',4',6'-триен-1'-иламино)пиримидину и 4,6-диметил-2-(N-тритиламино)пиримидину, что связано с подвижностью протонов аминогруппы вследствие амино-иминной таутомерии. Результат взаимодействия тетрафторбората тропилия с 2-амино-4,6-дигидроксипиримидином в аналогичных условиях зависит от соотношения исходных реагентов. Установлено три направления реакции: при использовании соотношения 1 : 1,5 образуется 4,6-дигидрокси-2-(N-циклогепта-2',4',6'-триен-1'-иламино)пиримидин, содержащий только один тропилиевый фрагмент у атома азота аминогруппы; введение в реакцию двойного избытка соли в один прием приводит к 4,6-дигидрокси-2-(N,N-дициклогепта-2',4',6'-триен-1'-ил)аминопиримидину, содержащему два тропилиевых фрагмента у атома азота аминогруппы, образование этого амина можно объяснить высокой подвижностью атомов водорода в аминогруппе вследствие амино-иминной таутомерии и кинетического контроля процесса; последовательное введение соли с интервалом 1,5 ч приводит к 2-(N-циклогепта-2',6',4'-триен-1'-иламино)-3-(циклогепта-2',4',6'-триен-1'-ил)-6-гидроксипиримидин-4(3H)-ону, в котором замещены атомы водорода у экзоциклического и эндоциклического атомов азота гетероцикла. Третье направление реакции, связанное с добавлением второго эквивалента соли с интервалом 1,5 ч, способствует повышению концентрации таутомера, возникающего вследствие лактам-лактимной таутомерии. Структура всех полученных соединений подтверждена данными ^{1}H ЯМР спектроскопии и масс-спектрометрии, а также методом рентгеноструктурного анализа.

Ключевые слова: соли тропилия, тритилия, 2-амино-4,6-диметилпиримидин, 2-амино-4,6-дигидроксипиримидин

TROPYLIUM AND TRITYLIUM SALTS IN REACTIONS WITH 2-AMINO-4,6-DISUBSTITUTED PYRIMIDINES

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Interaction of tropylium or tritylium tetrafluoroborate with 2-amino-4,6-dimethylpyrimidine in an ethanol medium at room temperature was found out to result in 4,6-dimethyl-2-(N-cyclohepta-2',4',6'-triene-1'-ilamino)pyrimidine and 4,6-dimethyl-2-(N-tritylamino)pyrimidine. Interaction between tropylium tetrafluoroborate and 2-amino-4,6-dihydroxypyrimidine under similar conditions is dependent on the ratio of starting reagents. Three reaction routes have been ascertained: with the ratio 1 : 1.5, 4,6-dihydroxy-2-(N-cyclohepta-2',4',6'-triene-1'-ilamino)-pyrimidine containing only one tropylium moiety at the nitrogen atom of amino group is formed; addition of a double excess of the salt at one go leads to 4,6-dihydroxy-2-(N-cyclohepta-2',4',6'-triene-1'-ilamino)aminopyrimidine containing two tropylium moieties at the nitrogen atom of amino group, formation of amine can be explained by a high mobility of hydrogen atoms in amino group because of the amino-imine tautomerism and kinetic control of the process; sequential addition (every 1.5 h) of the salt leads to formation of 2-(N-cyclohepta-2',4',6'-triene-1'-ilamino)-3-(cyclohepta-2',4',6'-triene-1'-il)-6-hydroxypyrimidin-4(3H)one in which the hydrogen atoms at exocyclic and endocyclic nitrogen atoms of the heterocycle are substituted. The reaction route, characterized by sequential addition (every 1.5 h) of the salt, contributes to an increase in concentration of the tautomer because of the lactam-lactim tautomerism resulting in amine. The structure of all the compounds so produced is confirmed by the findings of ^{1}H NMR spectroscopy, mass-spectrometry, and XRD analysis.

Keywords: tropylium/tritylium salts, 2-amino-4,6-dimethylpyrimidine, 2-amino-4,6-dihydroxypyrimidine

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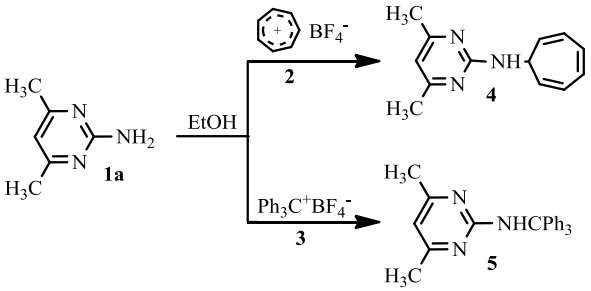
Tropylium/tritylium salts have been used for either C-tropylization or N-tritylization of aromatic [1-5] or heterocyclic [6] amines. Besides, stable and reactive tropylium salts [7] have been used as hydride-ion acceptors. Oxidative functionalization of tetrahydroisoquinolines at C1 position with use of tropylium tetrafluoroborate appears to be an example of interest.

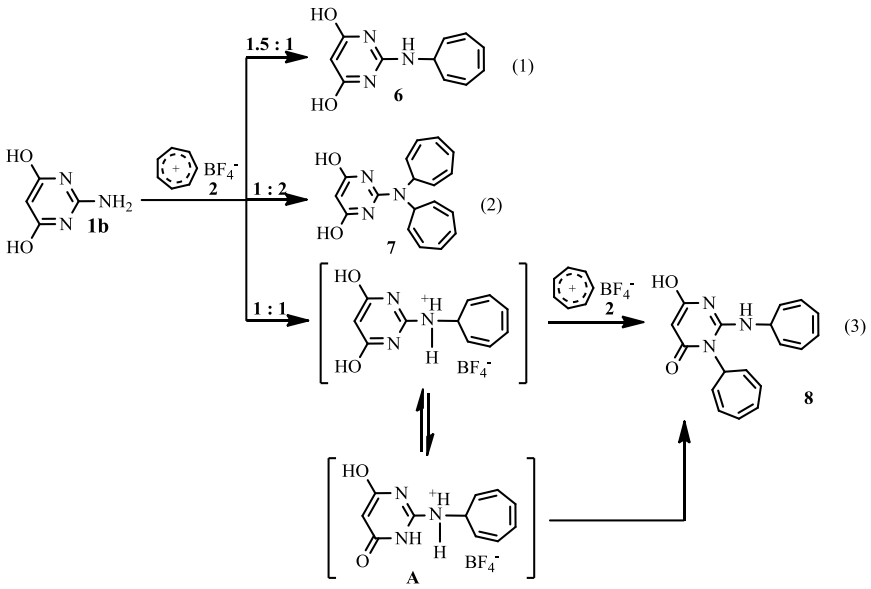
2-Amino-4,6-dimethylpyrimidin and 2-amino-4,6-dihydroxypyrimidine **1a, b** belong to a group of known pharmacophores which are of interest for synthesis of new compounds with potential biological activity [8-14]. Reactivity of these compounds can be attributed to mobility of protons of the NH_2 and OH groups because of appearance of the amino-imine (for **1a**, **1b**) and lactam-lactim tautomerism (for **1b**) [15-19].

This work aims at investigation of a possibility of N-tropylization and N-tritylization for compounds **1a** and **1b**.

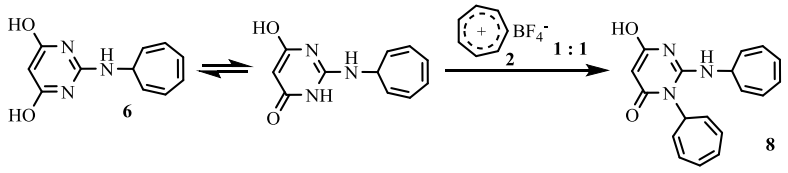
Interaction of tropylium **2** and tritylium **3** tetrafluoroborate with 2-amino-4,6-dimethylpyrimidine **1a** in an ethanol medium at room temperature was found out to result in 4,6-dimethyl-2-(N-cyclohepta-2',4',6'-triene-1'-ilamino)pyrimidine **4** and 4,6-dimethyl-2-(N-tritylamino)pyrimidine **5**, respectively.



The result of interaction between tropylium tetrafluoroborate **2** and 2-amino-4,6-dihydroxypyri-midine **1b** under similar conditions is dependent on the ratio of starting reagents. Three reaction routes have been ascertained: (1) with the ratio 1 : 1.5, 4,6-dihydroxy-2-(N-cyclohepta-2',4',6'-triene-1'-ilamino)-pyrimidine containing only one tropylium moiety at the nitrogen atom of amino group is formed; (2) addition of a double excess of the salt **2** at one go leads to formation of 4,6-dihydroxy-2-(N-cyclohepta-2',4',6'-triene-1'-ilamino)-aminopyrimidine containing two tropylium moieties at the nitrogen atom of amino group; (3) sequential addition (every 1.5 h) of the salt **2** leads to formation of 2-(N-cyclohepta-2',4',6'-triene-1'-ilamino)-3-(cyclohepta-2', 4',6'-triene-1'-il)-6-hydroxy-pyrimidin-4(3H)one **8** in which the hydrogen atoms at exocyclic and endocyclic nitrogen atoms of the heterocycle are substituted.

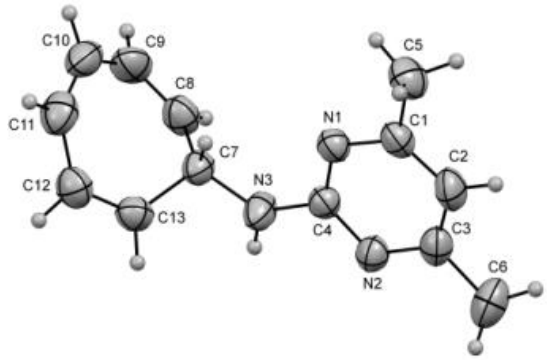


In the reaction route (2), formation of amine **7** can be explained by a high mobility of hydrogen atoms in amino group **1b** because of the amino-imine tautomerism and kinetic control of the process. The reaction route (3), characterized by sequential addition (every 1.5 h) of the salt **2,** contributes to an increase in concentration of the tautomer **A** because of the lactam-lactim tautomerism resulting in amine **8**. The latter result is confirmed by formation of amine **8** obtained by interaction between amine **6** and salt **2**.



The structure of all the compounds so produced is confirmed by the findings of ^{1}H NMR spectroscopy, mass-spectrometry, and XRD analysis for substances **4** and **5** (Figure).

According to the data of X-ray diffraction analysis, the compound **4** crystallizes in a space centrosymmetric group of a monoclinic crystal system. The bonds length and bond angles in a molecule are close to the standard values of corresponding atoms. Pyrimidine cycle is plane within 0.01 Å. The cycloheptatriene cycle is in a boat-like conformation. Atoms C^{11}, C^{10} and C^{7} are removed from a plane $C^{9}C^{8}C^{12}C^{13}$ at 0.51, 0.50 and 0.7Å respectively. The dihedral angle between $C^{9}C^{8}C^{12}C^{13}$ plane and the plane of pyrimidine cycle is 72.8 °. In a crystal, the molecules are connected by intermolecular hydrogen bonding N^{3}–$H^{3A}\cdots N^{2}$, $d(N^{3}\cdots N^{2})$ 3.095(2) Å, $d(N^{3}\cdots H^{3A})$ 0.843(2) Å, $d(H^{3A}\cdots N^{2})$ 2.260(2) Å. The molecules of a compound form dimers by intermolecular hydrogen bonding.



4

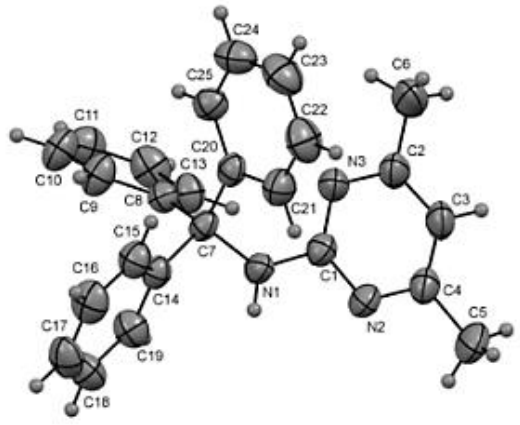


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Fig. The structure of 2-(N-cyclohepta-2',4',6'-triene-1-il)amino-4,6-dimethylpyrimidine **4** and the structure of 4,6-dimethyl-2-(N-tritylamino)pyrimidine **5**.
Рис. Структура 2-(N-циклогепта-2,4,6-триен-1-ил)амино-4,6-диметилпиримидина **4** и структура 4,6-диметил-2-(N-тритиламино)пиримидина **5**.

The compound **5** crystallizes in a space centrosymmetric group of a monoclinic crystal system. Pyrimidine cycle is plane within 0.025 Å. Angle of C-N-C stands at 128.5(2) °.

The CIF files containing complete information on the structures under scrutiny was deposited with the CCDC and registered as Nr. 1850688 for compound **4,** and Nr. 1850689 for compound **5**. The file may freely be retrieved upon request at www.ccdc.cam.ac.uk/data_request/cif.

EXPERIMENTAL PART

The ^{1}H NMR spectra were registered on the Mercury 400 device (400 MHz), with HMDS as internal standard. The mass spectra were registered on the maXis Impact HD mass spectrometer (*Bruker*, Germany). The Xcalibur R diffractometer was used to measure parameters of the elemental cell and of back-scattered intensity. An empirical absorption correction was applied to the findings using the SCALE3 ABSPACK scaling algorithm [20]. The structure was determined by the direct method and corrected by the full matrix OLS method with use of the SHELX-2013 software package [21].

2-(N-cyclohepta-2',4',6'-triene-1-il)amino-4,6-dimethylpyrimidine 4. A 0.36 g portion (2 mmol) of salt **2** was added at one go to 0.49 g (4 mmol) of amine **1a** dissolved in 5 ml of EtOH. The reaction mass was stirred for 3 h at room temperature. After removal of the solvent, 10% solution of NH_3 was added to adjust the pH value to 7-8. Precipitate of white color. Yield: 0.31 g (72.8%); m.p. 115-116 °C (hexane). ^{1}H NMR ($CDCl_3$), δ, ppm: 2.31 s (6H, $2CH_3$), 4.47-4.54 m (1H, C$^{1'}$H), 5.57 ddt (2H, *J* 9.3 Hz, *J* 5.9 Hz, *J* 0.8 Hz, C$^{2'}$H, C$^{7'}$H), 6.07 s (1H, NH), 6.27 dddd (2H, *J* 9.2 Hz, *J* 3.8 Hz, *J* 2.7 Hz, *J* 0.9 Hz, C$^{3'}$H, C$^{6'}$H), 6.33 s (1H, CH), 6.71 ddt (2H, *J* 3.5 Hz, *J* 2.7 Hz, *J* 0.8 Hz, C$^{4'}$H, C$^{5'}$H). Found: 214.1342 [M++H]$^+$ $C_{13}H_{15}N_3$. Calculated for $[C_{13}H_{15}N_3]^+$ 214.1339.

The crystals of compound **4** are monoclinic, steric group *I* 2/a, a = 11.617(3), b = 10.532(3), c = 19.530(5) Å, β = 94.55(3) º, V = 2382.0(11) Å^3, $d_{\text{calculated}}$ = 1.189, μ = 0.073 мм$^{-1}$, $C_{13}H_{15}N_3$, Z = 8. Finite correction parameters: R_1 = 0.0557, wR_2 = 0.1381 [for 1968 reflections with $I > 2\sigma(I)$], R_1 = 0.0825, wR_2 = 0.1544 (for all 2810 independent reflections, R_{int} = 0.0259), S = 1.033.

4,6-Dimethyl-2-(N-tritylamino)pyrimidine 5. A 0.20 g portion (0.60 mmol) of salt **3** was added at one go to 0.09 g (0.73 mmol) of amine **1a** dissolved in 6 ml of CH_2Cl_2. The reaction mass was stirred for 2 h at room temperature. After removal of the solvent, 10% solution of NH_3 was added to adjust the pH value to 7-8. Precipitate of white color. Yield: 0.15 g (68%); m.p. 144-146 °C (hexane). ^{1}H NMR ($CDCl_3$), δ, ppm: 2.05 s (6H, $2CH_3$), 2.80 s (1H, NH), 6.20 s (1H, C^{5}H), 7.17-7.36 m (15H, 3Ph). Found: 366.1959 [M+H]$^+$ $C_{25}H_{23}N_3$. Calculated for $[C_{25}H_{23}N_3]^+$ 366.1965.

The crystals of compound **5** are monoclinic, steric group $P2_1/n$, at 295.0(2) K: a 13.9281(36), b 9.0686(23), c 16.3790(41) Å; β 103.665(26) °; V 2010.24(131) Å^3; Z 4; d calculated 1.21 g/cm^3; F(000) 776; μ 0.072 mm^{-1}. Finite divergence factors: R_1 0.0579, wR_2 0.1373 for reflections with $I > 2\sigma(I)$, R_1 0.1132, wR_2 0.1621 (for all reflections), S 1.002. Completeness of collected findings for θ < 26.00 ° equals 99.8%. Maximal and minimal values of residual electron density peaks equal 0.172 and –0.222 $\bar{e}$/Å^3, respectively.

4,6-Dihydroxy-2-(N-cyclohepta-2',4',6'-triene-1-il)aminopyrimidine 6. A 0.065 g portion (0.37 mmol) of salt **2** was added to 0.07 g (0.55 mmol) of amine **1b** suspended in 10 ml of ethanol. The reaction mass was stirred for 2 h at room temperature; at this point, the initially rosy solution turned light-yellow. After that, 10% solution of NH_3 was added to the reaction mass to adjust the pH value to 7-8. The white crystals were filtered off and rinsed with ethanol and ether. Yield 0.065 g (82%), m.p. 215 °C (with decomposition). ^{1}H NMR (DMSO-d$_6$), δ, ppm: 2.67 t (1H, *J* 5.5 Hz, C$^{1'}$H), 5.26 dd (2H, *J* 9.1 Hz, *J* 5.5 Hz, C$^{2'}$H C$^{7'}$H), 6.02 dd (2H, *J* 9.2 Hz, *J* 2.0 Hz, C$^{3'}$H C$^{6'}$H), 6.42-6.70 m (4H, C$^{4'}$H C$^{5'}$H+NH, C^{5}H), 10.41 s (2H, 2OH). Found: 218.0924 [M+H]$^+$ $C_{11}H_{11}N_3O_2$. Calculated for $[C_{11}H_{12}N_3O_2]^+$ 218.0924.

4,6-Dihydroxy-2-(N,N-dicyclohepta-2',4',6'-triene-1'-il)aminopyrimidine 7. A 0.063 g portion (0.5 mmol) of amine **1b** was added to 0.17 g (1 mmol) of salt **2** suspended in 20 ml of ethanol. The reaction mass was stirred for 4 h at room temperature. After 3 h stirring, the initially bright-yellow solution turned orange. The sediment was filtered and rinsed with water. With additional 5 ml of water added, the precipitate was neutralized dropwise with 10% solution of NH_3 to attain pH 7, light-yellow crystals were filtered off and rinsed with ethanol and ether. Yield 0.08 g (75%), m.p. 180 °C (with decomposition). ^{1}H NMR (DMSO-d$_6$), δ, ppm: 2.37 tt (2H, *J* 5.9 Hz, *J* 1.4 Hz), 5.35–5.12 m (4H), 6.16 dddd (4H , *J* 9.1 Hz, *J* 4.0 Hz, *J* 2.7 Hz, *J* 1.3 Hz) 6.67 dd (4H, *J* 3.6 Hz, *J* 2.5 Hz), 6.78 s (1H), 8.25 s (1H), 11.11 s (1H). Found: 308.1393 [M + H]$^+$ $C_{18}H_{17}N_3O_2$. Calculated for $[C_{18}H_{18}N_3O_2]^+$ 308.1394.

2-(N-cyclohepta-2',4',6'-triene-1'-ilamino)-3-(cyclohepta-2',4',6'-triene-1'-il)-6-hydroxypyrimidin-4(3H)one 8. A 0.180 g portion (1 mmol) of salt **2** was added to 0.127 g (1 mmol) of amine **1b** suspended in 20 ml of ethanol while stirred. 1.5 Hour later, a second 0.180 g portion (1 mmol) of salt **2** was added to the suspension colored bright-yellow, whereupon the reaction mass was stirred for 2 h at

room temperature. The yellow precipitate was treated with 10% solution of NH_3 to attain pH 7, separated and rinsed with hexane and ether. Yield 0.17 g (55%), m.p. 190 °C (with decomposition). ^{1}H NMR (DMSO-d_6), δ, ppm: 2.38 t (1H, *J* 5.9 Hz, $C^{1''}H$), 2.69 tt (1H, *J* 5.4 Hz, *J* 1.5 Hz, $C^{1'}H$), 5.24-5.33 m (4H, $C^{2'}H$, $C^{7'}H$, $C^{2''}H$, $C^{7''}H$), 6.0 dddd (2H, *J* 8.8 Hz, *J* 3.8 Hz, *J* 2.6 Hz, *J* 1.6 Hz, $C^{3''}H$, $C^{6''}H$), 6.15 dddd (2H, *J* 8.8 Hz, *J* 3.8 Hz, *J* 2.6 Hz, *J* 1.6 Hz, $C^{3'}H$, $C^{6'}H$), 6.56 t (2H, *J* 3.1 Hz, $C^{4'}H$, $C^{5'}H$), 6.66 t (2H, *J* 3.0 Hz, $C^{4''}H$, $C^{5''}H$), 8.20 m (3H, NH, C^5H, OH). Found: 308.1395 $[M + H]^+$ $C_{18}H_{17}N_3O_2$. Calculated for $[C_{18}H_{18}N_3O_2]^+$ 308.1394.

2-(N-cyclohepta-2',4',6'-triene-1'-ilamino)-3-(cyclohepta-2',4',6'-triene-1'-il)-6-hydroxypyrimidin-4(3H)one 8. A 0.033 g portion (0.18 mmol) of salt **2** was added at one go to 0.04 g (0.18 mmol) of compound **6** suspended in 5 ml of ethanol while stirred. 10 Min later, the suspension colored white turned yellow. 2 H later, the reaction mass was treated with 10% solution of NH_3 to attain pH 7. The yellow sediment was separated and rinsed with hexane and ether. Yield: 0.05 g (89%), m.p.190 °C (with decomposition).

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