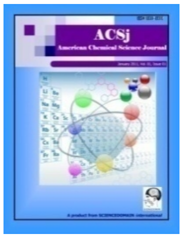

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The Reactions of Hydrochlorides of 2, 3-Tri (Tetra) Methylene-3, 4-Dihydropyrimidine-4-Ones with N-Bromosuccinimide and Bromine

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Authors' contributions

This work was carried out in collaboration between all authors. Authors KZK and AON carried out experiments, author KUK designed the study, wrote the protocol and the first draft of the manuscript, author KMS managed the analyses of the study and author NIM managed the literature searches. All authors read and approved the final manuscript.

Original Research Article

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ABSTRACT

Aims: To carry out reactions between hydrochlorides of 2, 3-tri(tetra)methylene-3, 4-dihydropyrimidine-4-ones and *N*-bromosuccinimide (and/or bromine), to determine way of reactions.
Study Design: Official design.
Place and Duration of Study: Department of Organic Chemistry, Institute of the Chemistry of Plant Substances (ICPS) between January 2012 and September 2013.
Methodology: We have synthesized 6-methyl-2, 3-tri(tetra)methylene-3, 4-dihydropyrimidine-4-ones from ethyl β-aminocrotonate and γ-butyro- or δ-valerolactames in the presence of different agents, such as PCl_5, $POCl_3$ and $SOCl_2$. For convenient operations the oily bases of 6-methyl-2, 3-tri(tetra)methylene-3, 4-dihydropyrimidine-4-ones have been transmuted into the appropriate hydrochlorides. Bromination by equimolar amount of hydrochlorides with *N*-bromosuccinimide at room temperature in water gives 5-bromo-6-methyl-2, 3-tri(tetra)methylene-3, 4-dihydropyrimidine-4-ones. Treatment of them by bromine resulted in the formation of perbromides. The reactions were monitored and

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controlled by TLC. Bromine-products were characterized by IR, ^{1}H NMR, mass-spectra determinations and chemical transformation.

Results: Electrophilic substitution at C5 atom was successfully carried out when we have used *N*-bromosuccinimide. Perbromides of 6-methyl-2, 3-tri(tetra)methylene-3, 4-dihydropyrimidine-4-ones were produced when we have used bromine. Perbromides performed to hydrobromides and bromine complexes when we have treatment of them by acetone and $NaHCO_3$ solution respectively.

Conclusion: Bromination of hydrochlorides of 6-methyl-2, 3-tri(tetra)methylene-3, 4-dihydropyrimidine-4-ones by *N*-bromosuccinimide gives 5-bromine-derivatives. Bromination of them by bromine followed to the formation of perbromides of 6-methyl-2, 3-tri(tetra)methylene-3, 4-dihydropyrimidine-4-ones which are performed to hydrobromides and bromine complexes when we have used corresponding treatments.

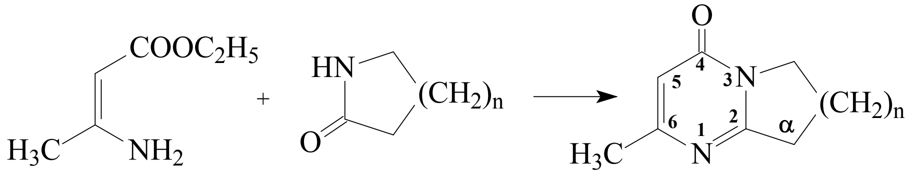
Keywords: Bromination; N-bromosuccinimide; bromine; perbromides; hydrobromide; 6-methyl-2, 3-tri(tetra)methylene-3, 4-dihydropyrimidine-4-ones.

1. INTRODUCTION

Condensed and uncondensed pyrimidines are wide occupied in the nature [1]. Pyrimidines are involved to the content of Ribonucleic acids (RNA) such as uracyl, Deoxyribonucleic acids (DNA) such as thymine or to both types of nucleic acids (cytosine). Pyrimidines are useful for synthesis of drugs for example, barbiturates, chemical agents of plant protection and etc. On the other hand pyrimidines, especially their derivatives containing functional groups at 2 and (or) 4 positions have various reaction capability and reaction direction depends on nature of substituent [1-2]. Thus, alkylation of 2-oxo-, -thioxo-, -selenoxopyrimidine-4-ones moved to O (or S, Se, N1, N3) atoms [3,4] which confirmed by electron spectrums of absorption [5]. It is known that interactions of condensed with benzene ring pyrimidines - 2,3-tri-, -tetra methylene-3,4-dihydroquinazoline-4-ones – quinazolines with *N*-bromosuccinimide (NBS) and bromine moved to N1, α-C, C4 atoms (in case of quinazolines and NBS) and to an aromatic ring [1,6-11]. So it was interesting to study natures of simple pyrimidine-4-ones which contain polymethylene cycle with unknown reaction by electrophilic agents, for example, with brominating agents at conditions of 2,3-polymethylene-3,4-dihydroquinazolines and –thieno (2,3-d) pyrimidine-4-ones [11-13].

2. MATERIALS AND METHODS

We synthesized 6-methyl-2,3-tri(tetra)methylene-3,4-dihydropyrimidine-4-ones (1,2) from ethyl β-aminocrotonate and γ-butyro- or δ-valerolactames in the presence of different agents (PCl_5, $POCl_3$, $SOCl_2$).

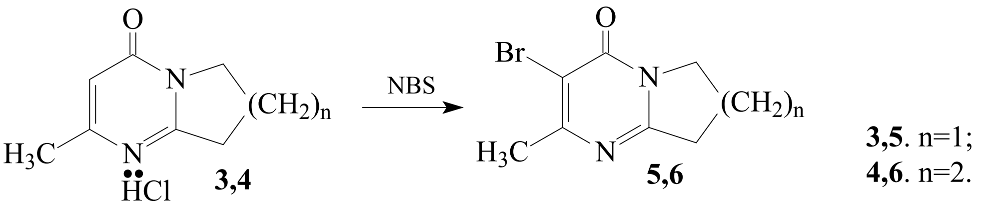


1. n=1; **2**. n=2.

Scheme 1. Synthesis of 6-methyl-2, 3-tri(tetra)methylene-3, 4-dihydropyrimidine-4-ones

As a matter of convenience operations the oily bases of 6-methyl-2, 3-tri(tetra)methylene-3, 4-dihydropyrimidine-4-ones (1,2) have been transmuted into the appropriate hydrochlorides 3, 4.

Bromination by equimolar amount of hydrochlorides 3, 4 with *N*-bromosuccinimide at room temperature in water solution gives 5-bromo-6-methyl-2, 3-tri(tetra)methylene-3, 4-dihydropyrimidine-4-ones (5,6). Duration of reaction we have controlled with High Performance Thin Layer Chromatography (HPTLC), using this method for identification and determination of purity of compounds 5,6.



Scheme 2. Reactions of 3 and 4 with NBS

Structures of products were confirmed by ^{1}H NMR and chromato-mass-spectrums.

2.1. Apparatus Procedure

^{1}H NMR spectra were recorded on an UNITY-400+ spectrometer at 400 MHz by using solvents $CDCl_3$ and D_2O at room temperature and TMS as internal standard. IR spectra were taken on a FT-IR-spectrometer Perkin-Elmer, 2000 in KBr tablets.

HPTLC analysis carried out by the method which was elaborated by us earlier [14] with 1 mg/mL alcohol solution of a substance. Solution (3 μl) sprinkled to plate by Linomat 5. Length between treks is 8 mm, wide of treks is 3mm. For analysis were used AL SIL G/UV (Whatman Paper LTD, Germany) plates. Chromatography plates scanned at 254 nm by Scanner 3 (CAMAG TLC) with WinCATS program. Solvent system of $CHCl_3$-C_6H_6-CH_3OH (10:10:1) were used as eluents.

Chromato-mass-spectral analysis was performed on a chromato-mass-spectrometer with gas chromatograph Trace GC and quadruple mass-spectrometric detector Polaris Q (Thermo Finnigan, EI, 70 eV). Analysis condition: capillary column SiO_2 (7 m x 0.32 mm) with stated phase DB-ms, thickness of NF film is 0.25 μm, temperature programmed from 60ºC (stand of 2 min) to 200ºC with increasing 6^{o}C/min and to 280^{o}C with increasing 8^{o}C/min, injector temperature is 220^{o}C, interface 220^{o}C, scanning of mass-spectrums in mass range 41-450 a.m.u. with scan rate 1 spectrum/s. Regimen of dividing of a flow was 1:25.

2.2 Experimental Details

Synthesis of hydrochloride of 6-methyl-2, 3-trimethylene-3,4-dihydropyrimidine-4-one (3). $POCl_3$ (90ml) was dropped to mixture of ethyl β-aminocrotonate (60 g, 0.47 mol) and γ-butyrolactam (60g, 0.7 mol) by stirring at room temperature. Reaction mixture heated at 80-90ºC during 4 hours, cooled to room temperature and added 100 ml water. Water solution worked up by 5% NaOH solution to pH 7.5-8. Extracted by chloroform (3 x 100 ml) and organic phase dried with Na_2SO_4, filtrated and evaporated solvent. Residue distilled under vacuum, b.p. 130-135°C (9 mm_{Hg}). Obtained product 1 dissolved in acetone and it is dropped HCl through the solution. Yield of compound 3 is 42 g (60.3%), m.p. 167-168°C. R_f 0.48 (chloroform-benzene-methanol, 10:10:1). IR spectrum, ν, cm^{-1}: 1668 ($\nu_{C=O}$). ^1H NMR spectrum (400 MHz, D_2O), δ, ppm (J, Hz): 4.29 (2H, t, α-CH_2, J=7.6), 2.44 (5H, m, β-CH_2, 6-CH_3), 3.52 (2H, t, γ-CH_2, J=7.6), 6.48 (1H, q, H-5, J=7.6). Found, %: C 54.70; H 6.12; N 16.20. $C_8H_{11}N_2OCl$. Calculated, %: C 54.85; H 6.28; N 16.00.

Synthesis of hydrochloride of 6-methyl-2, 3-tetramethylene-3,4-dihydropyrimidine-4-one (4). $POCl_3$ (50ml) was dropped to cooled by ice water mixture of ethyl β-aminocrotonate (32 g, 0.25 mol) and δ-valerolactam (24 g, 0.24 mol). Reaction mixture heated for 4 hours at 90-95°C. Ice (100 g) was added to obtained mixture and added base up to pH 7.5-8. Organic layer was extracted by $CHCl_3$, dried with Na_2SO_4. Obtained an oil-like residue after distillation, chloroform was removed under vacuum. Product 2 has b.p. 150-155°C (14 mm_{Hg}). It was dissolved in acetone and it is dropped HCl through the solution. Residue filtered, washed by acetone then dried. Yield of 4 is 25.6 g (62%), m.p. 192-194°C. R_f 0.56 (benzene-chloroform-methanol, 10:10:1). ^1H NMR spectrum (400 MHz, D_2O), δ, ppm (J, Hz): 3.90 (2H, t, α-CH_2, J=6.0), 1.85-2.05 (4H, m, β-CH_2, γ-CH_2), 3.10 (2H, t, δ-CH_2, J=6.5), 6.40 (1H, q, H-5), 2.33 (3H, d, 6-CH_3, J=0.2). Found, %: C 57.30; H 6.72; N 14.65. $C_9H_{13}N_2OCl$. Calcl, %: C 57.14; H 6.88; N 14.81.

Reaction of 3 with NBS. Synthesis of 5-bromo-6-methyl-2, 3-trimethylene-3, 4-dihydropyrimidine-4-ones (5). Water solution of hydrochloride of 6-methyl-2, 3-trimethylene-3, 4-dihydropyrimidine-4-one (50 mg, 0.268 mmol) treated with 20% HCl to pH=3-4, then added 60 mg NBS and stirred 2 h. It treated by 5% NaOH solution to pH 7-8. Extracted by chloroform, organic phase was dried with Na_2SO_4, filtered and distilled chloroform. Yield of 5 is 35 mg (56%), mp 110-112°C (C_6H_6). IR spectrum, ν, cm^{-1}: 1668 ($\nu_{C=O}$). ^1H NMR spectrum (400 MHz, $CDCl_3$), δ, ppm (J, Hz): 4.29 (2H, t, α-CH_2, J=7.6), 2.44 (5H, m, β-CH_2, 6-CH_3), 3.52 (2H, t, γ-CH_2, J=7.6). Found, %: C 41.81; H 3.78; N 12.12. $C_8H_9N_2OBr$. Calculated, %: C 41.92; H 3.93; N 12.27.

Reaction of 4 with bromine. Synthesis of perbromide of 6-methyl-2,3-trimethylene-3,4-dihydropyrimidine-4-one (7). Hydrochloride of 6-methyl-2, 3-trimethylene-3, 4-dihydropyrimidine-4-one (100 mg, 0.53 mmol) was dissolved in acetic acid (5 ml, 75%). Bromine was added dropwise (5 ml, 1.06 mmol in 2 ml acetic acid). It was obtained a red precipitate. Yield 43 mg (20.5%), m.p. 128-130°C. IR spectrum, ν, cm^{-1}: 2610 ($\nu_{N\cdots HBr3}$), 1668 ($\nu_{C=O}$). Found, %: C 24.41; H 2.78; N 7.01. $C_8H_{11}N_2OBr_3$. Calculated, %: C 24.55; H 2.81; N 7.16.

Hydrobromide of 6-methyl-2, 3-trimethylene-3, 4-dihydropyrimidine-4-one (9) was obtained as white crystals at treatment of 20 mg (0.05 mmol) perbromide of 6-methyl-2,3-trimethylene-3,4-dihydropyrimidine-4-one (8) by 2 ml acetone. Yield 5 mg (45%), m.p. 280-283°C. IR spectrum, ν, cm^{-1}: 2720 ($\nu_{N\cdots HBr}$), 1668 ($\nu_{C=O}$). Found, %: C 43.60; H 5.18; N 12.82. $C_8H_9N_2OBr$. Calculated, %: C 43.63; H 5.00; N 12.72.

Bromine complex of 6-methyl-2, 3-trimethylene-3, 4-dihydropyrimidine-4-one (11) was obtained at treatment of 20 mg (0.050 mmol) of perbromide of 6-methyl-2,3-trimethylene-3,4-dihydropyrimidine-4-one (8) with $NaHCO_3$ (5ml, 10% solution) at room temperature as white crystals. Yield is 6 mg (32%), mp 103-106°C. IR spectrum, ν, cm^{-1}: 2500 ($\nu_{N\cdots Br2}$), 1668

($\nu_{C=O}$). Found, %: C 24.54; H 2.68; N 7.12. $C_8H_{10}N_2OBr_3$. Calculated, %: C 24.61; H 2.56; N 7.18.

Reaction of 6-methyl-2, 3-tetramethylene-3,4-dihydropyrimidine-4-one hydrochloride with bromine. Synthesis of perbromide of 6-methyl-2, 3-tetramethylene-3, 4-dihydropyrimidine-4-one (8). Hydrochloride of 6-methyl-2, 3-tetramethylene-3, 4-dihydropyrimidine-4-one (100 mg, 0.5 mmol) was dissolved in acetic acid (5ml, 70%). Bromine (0.055 ml, 1.065 mmol in 2 ml 70% acetic acid) was dropped to the prepared solution. Precipitate filtered, and treated by ether. Yield of 8 is 50 mg (24.7%), m.p. 120-122ºC. IR spectrum, ν, cm^{-1}: 2620 ($\nu_{N\cdots HBr3}$), 1668 ($\nu_{C=O}$).: Found, %: 26.41; H 3.34; N 6.70. $C_9H_{13}N_2OBr_3$. Calculated, %: C 26.66; H 3.21; N 6.91.

Hydrobromide of 6-methyl-2, 3-tetramethylene-3, 4-dihydropyrimidine-4-one (10) was obtained by treatment 20 mg (0.05 mmol) perbromide of 6-methyl-2,3-tetramethylene-3,4-dihydropyrimidine-4-one (8) with acetone at room temperature as a white precipitate. Yield 7 mg (60%), m.p. 260-262ºC. IR spectrum, ν, cm^{-1}: 2730 ($\nu_{N\cdots HBr}$), 1668 ($\nu_{C=O}$). Found, %: C 46.30; H 5.78; N 11.81. $C_8H_9N_2OBr$. Calculated, %: C 46.15; H 5.55; N 11.96.

Bromine complex of 6-methyl-2, 3-tetramethylene-3,4-dihydropyrimidine-4-one (12). Treatment of perbromide of 6-methyl-2,3-tetramethylene-3,4-dihydropyrimidine-4-one (20 mg, 0.05 mmol) with $NaHCO_3$ (10 %, 5 ml) yielded compound 12 as a white crystal. Yield 7 mg (45 %), m.p. 92-94ºC. IR spectrum, ν, cm^{-1}: 2500 ($\nu_{N\cdots Br2}$), 1670 ($\nu_{C=O}$). Found, %: C 26.65; H 2.66; N 7.12. $C_8H_{10}N_2OBr_3$. Calculated, %: C 26.73; H 2.97; N 6.93.

Synthesis of 5-bromo-6-methyl-2, 3-trimethylene-3,4-dihydropyrimidine-4-one (5) from perbromide of 6-methyl-2, 3-trimethylene-3, 4-dihydropyrimidine-4-one (7). Sample of perbromide of 6-methyl-2,3-trimethylene-3,4-dihydropyrimidine-4-one (50 mg, 0.123 mmol) was dissolved in acetic acid (2 ml, 75%). Mixture heated during 30 min at 80-90ºC, precipitate was separated. Yield 28 mg (56%), m.p. 110-112ºC (acetone).

Formation of 5-bromo-6-methyl-2, 3-tetramethylene-3, 4-dihydropyrimidine-4-one (6) from perbromide of 6-methyl-2, 3-tetramethylene-3, 4-dihydropyrimidine-4-one (8). Perbromide of 6-methyl-2,3-tetramethylene-3,4-dihydropyrimidine-4-one (8) (50 mg, 0.123 mmol) was dissolved in acetic acid (2 ml, 75%), the mixture is heated during 30 min at 80-90ºC. Precipitate filtered, washed by water and dried. Yield 19 mg (64 %), m.p. 112-114ºC (acetone). ^1H NMR spectrum (400 MHz, $CDCl_3$), δ, ppm (J, Hz): 4.14 (2H, t, α-CH_2, J=7.6), 2.90 (2H, t, δ-CH_2, J=7.6), 3.41 (7H, m, β-CH_2, γ-CH_2, 6-CH_3). Found, %: C 44.35; H 4.46; N 11.44. $C_9H_{11}N_2OBr$. Calculated, %: C 44.44; H 4.52; N 11.52.

3. RESULTS AND DISCUSSION

It is found that high yield of hydrochlorides of 6-methyl-2, 3-tri(tetra)methylene-3, 4-dihydropyrimidine-4-ones obtained by heating mixture of ethyl β-aminocrotonate and γ-butyro- or δ-valerolactames in presence $POCl_3$ [12,13].

6-Methyl-2, 3-tri(tetra)methylene-3, 4-dihydropyrimidine-4-ones (1,2) were prepared by hydrochlorides of (3,4) frequently after synthesis of the basis with 60% and 62% yields.

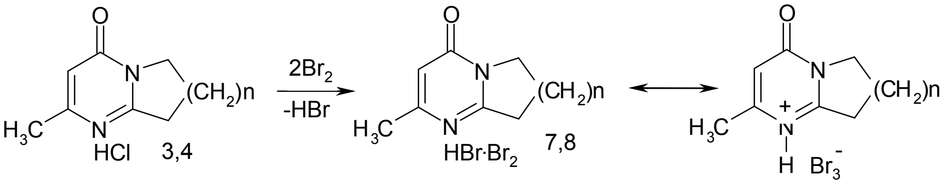
Bromination by equimolar amount of hydrochlorides with *N*-bromosuccinimide at room temperature in water solution gives 5-bromo-6-methyl-2, 3-tri(tetra)methylene-3, 4-

dihydropyrimidine-4-ones, that the reaction goes to C5 atom of pyrimidine ring of the starting compound.

^{1}H NMR spectrum of compound 5 have signals characterized to protons of methylene groups at 4.29 ppm (2H, t., α-CH_2, J=7.6 Hz), 2.44 ppm (5H, m., β-CH_2, 6-CH_3), 3.52 ppm (2H, t., γ-CH_2, J=7.6 Hz). Absence of a proton signal at 6.48 ppm (1H, q, H-5, J=7.6 Hz) and appearance of protons of α- and β-methylene groups as two triplets show that bromination goes at C5 carbon atom. In this case bromination of α-carbon atom did not go, which observed at reactions of analogical tricycle quinazolines [15]. Availability in chromato-mass-spectrum peaks of molecular ions which corresponding to m/z ($I_{relative}$, %): 228/230 $[M]^+$ (100%) and characterized fragment ions of 213/215 $[M-Me]^+$ (23), 200/202 $(M-CO)^+$ (11%), 148 $(M-Br)^+$ (19), 120 $(M-Br-CO)^+$ (12) also confirmed structure of 5-bromo-6-methyl-2,3-trimethylene-3,4-dihydropyrimidine-4-one (5).

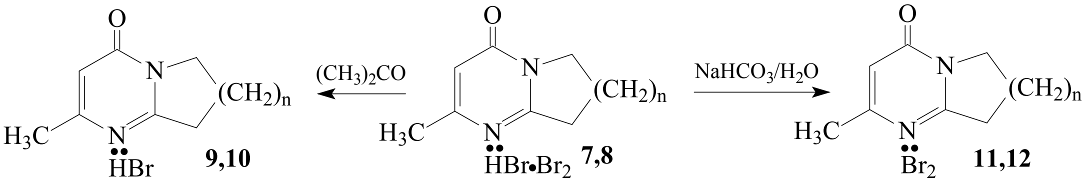
Reaction of hydrochloride of 6-methyl-2,3-tetramethylene-3,4-dihydropyrimidine-4-one (4) with *N*-bromosuccinimide goes analogically to form 5-bromo-6-methyl-2,3-tetramethylene-3,4-dihydropyrimidine-4-one (6).

Earlier we showed possibility formation of perbromide of 2,3-trimethylene-3,4-dihydroquinazolyne-4-one at bromination by molecular bromine in various solvents (chloroform, acetic acid, 80% methanol) [2]. Ractions 3,4 and bromine in 100% acetic acid at room temperature gave perbromides of 7,8, which have two tautomer forms:



Scheme 3. Reaction of 3,4 with bromine

Perbromides 7,8 produced hydrobromides 9,10 when they were worked by acetone. Treatment of compounds 7,8 with water solution of $NaHCO_3$ (5%) afforded bromine complexes 11,12.



Scheme 4. Formation of hydrobromides and bromine complexes

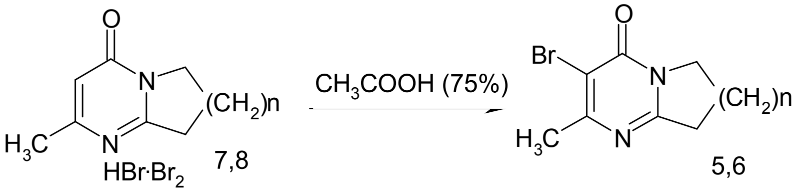
It is known that the characteristic absorption bands of quaternary ammonia salts in IR spectrum appeared at 2200-3000 cm^{-1}. Appearance in IR spectrum an absorption band at 2620 cm^{-1} shows the formation of quaternary salt of compound 7.

The absorption band of hydrobromide 9 appeared at 2730 cm^{-1}. Bromine complex 11 has an absorption band at 2500 cm^{-1}. These displacements and increasing the strength of the absorption band of fragments at N1 confirmed the given structure. Perbromide 7 has H-

Br···Br-Br bonds which decreased the strength of the absorption bands of hydrobromide 9 and bromine complex of 11. It is observed increasing the strength of quaternary bond because it is more stable.

Bromine complex of 6-methyl-2,3-tri(tetra)methylene-3,4-dihydropyrimidine-4-ones (11,12) have absorption bands in IR spectrum. It shows that they have a donor-acceptor bond. The bond formed between electron pair of N and LUMO of bromine molecule [16]. Bromine complex is a πδ(N:Br_2) type. Such as type of a bond was detected in a case of 2,3-trimethylene-3,4-dihydroquinazoline-4-one bromine complex [17].

Bromination of C5 carbon atom take place at heating perbromides of 6-methyl-2,3-tri(tetra)methylene-3,4-dihydropyrimidine-4-ones in AcOH (75%) at 90-95° to form of 5-bromo-6-methyl-2,3-tri(tetra)methylene-3,4-dihydropyrimidine-4-ones (5,6) respectively. Electrophilic substitution reaction of pyrimidine-4-ones by polar bromine molecule (H-Br···$Br^{\delta+}$-$Br^{\delta-}$) takes place in this case.



Scheme 5. Transformation of perbromides to 5-bromine-derivatives

It can be noted, that the bromination of 2,3-trimethylene-3,4-dihydroquinazoline-4-one at the same conditions carried out on other way with substitution of α-hydrogen atoms to form α,α-dibromine-2,3-trimethylene-3,4-dihydroquinazolyne-4-one [18]. It is necessary to note that hydrogen atoms of α-methylene groups are mobile in reactions of tricyclic benz- and thienopyrimidine-4-ones with carbonyl compounds [9,15,19,20].

Chromato-mass spectrum of 5-bromo-6-methyl-2,3-tetramethylene-3,4-dihydropyrimidine-4-one (6) has molecular ion peak with m/z ($I_{relative}$, %): at 242/244 $[M]^+$ (100%), and ion fragments as: 227/229 $[M-Me]^+$ (55), 164 $[M-Br]^+$ (70), 149 $[M-Br-Me]^+$ (13), 134 $(M-Br-CO)^+$ (41). There is not a proton signal at 6.48 ppm (1H, q, H-5, J=7.6Hz) in its ^1H NMR spectrum which was in starting compound and presented signals of methylene group protons at 4.14 ppm (2H, t., α-CH_2, J=7.6 Hz), 2.90 ppm (2H, t., δ-CH_2, J=7.6Hz), 3.41 ppm (7H, m., β-CH_2, γ-CH_2, 6-CH_3).

4. CONCLUSION

Reaction of hydrochlorides of 6-methyl-2,3-tri(tetra)methylene-3,4-dihydropyrimidine-4-ones with NBS yielded 5-bromo-6-methyl-2,3-tri(tetra)methylene-3,4-dihydropyrimidine-4-ones with electrophilic substitution at 5-position of pyrimidine ring.

Hydrochlorides of 6-methyl-2,3-tri(tetra)methylene-3,4-dihydropyrimidine-4-ones produced perbromides by treatment with bromine, which formed corresponding bromine complexes and hydrobromides at treatment by solution of $NaHCO_3$ and acetone, respectively.

Heating perbromides of 6-methyl-2,3-tri(tetra)methylene-3,4-dihydropyrimidine-4-ones in acetic acid (75%) formed 5-bromo-6-methyl-2,3-tri(tetra)methylene-3,4-dihydropyrimidine-4-

ones, respectively. It is the difference between them and their quinazoline analogs, which of reaction go on other way on α-C atom of tetrahydropyrrole (pyridine) ring.

COMPETING INTERESTS

Authors have declared that no competing interests exist.

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