Chemistry of Heterocyclic Compounds, Vol. 40, No. 8, 2004

INTERACTION OF CYCLIC SCHIFF'S BASES WITH 2-METHYLTHIOPYRIMIDINE-4,6-DIONE ENOL ACETATE. SYNTHESIS OF 5-(2-ACETYL-6,7-DIMETHOXY-1,2,3,4-TETRAHYDRO-1-ISOQUINOLYL)-6-HYDROXY-2-METHYLTHIO-1,4-DIHYDRO-4-PYRIMIDINONES

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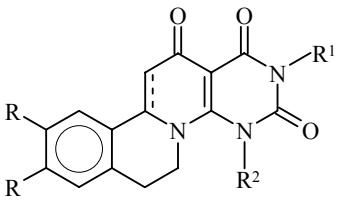
5-(2-Acetyl-1,2,3,4-tetrahydro-1-isoquinolyl)-6-hydroxy-2-methylthio-1,4-dihydro-4-pyrimidinones have been obtained by the interaction of 3,4-dihydroisoquinolines with 2-methylthio-4-oxo-1,4-dihydro-6-pyrimidinyl acetate. It was shown that N,O-methyl derivatives are formed in interaction of the obtained products with diazomethane.

Keywords: 3,4-dihydroisoquinolines, pyrimidine-4,6-dione enol acetates, 5-(1-isoquinolyl)pyrimidines, condensation reaction.

The derivatives of isoquinoline and pyrimidine are two groups of natural and synthetic regulators of many important processes of the active life of simple and higher organisms [1,2]. Isoquinolines are most widely represented in the plant world by the extremely vast group of isoquinoline alkaloids, the biological functions of which still have not been adequately clarified [3]. Pyrimidines, such as barbituric acid and its derivatives, respond to the transmission of signals along nerve channels and participate in the receptor system of γ-aminobutyric acid functioning in nerve cells, they display hypotensive, antiatherosclerotic and other important forms of physiological activity [4]. It therefore seemed of interest to develop approaches to the synthesis and study of the properties of compounds combining isoquinoline and pyrimidine fragments in their structure. Only a few examples of constructing such systems have been described in the literature, in particular derivatives of pyrimido[5',4':5,6]pyrido[2,1-*a*]isoquinoline or 8,15,17-triaza-D-homogonane **1** [5].



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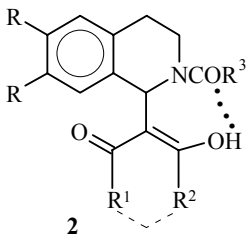

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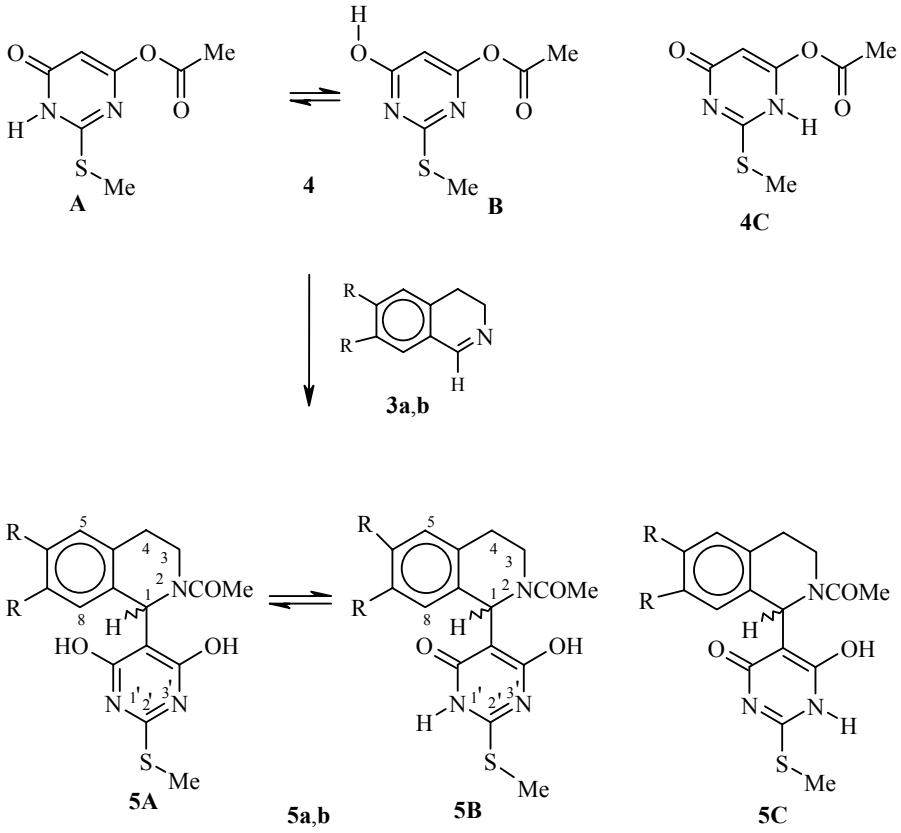
It is known [6,7] that on interacting 3,4-dihydroisoquinolines with enol acylates of aliphatic and alicyclic β-dicarbonyl compounds N-acyl-1-isoquinolyl derivatives of the corresponding β-dicarbonyl compounds (see **2**) are formed. These are of theoretical and practical interest, particularly as potential biologically active compounds. The possibility has been shown in the present work of extending this reaction to heterocyclic β-dicarbonyl compounds, to derivatives of 4,6-dihydroxypyrimidine, with the aim of obtaining products with isoquinoline and pyrimidine fragments in their molecules.

Scheme 1



3, **5 a** R = H, **b** R = OMe

The condensation of 3,4-dihydroisoquinolines **3a,b** with enol acetate **4** was effected by boiling an equimolar mixture of reactants in ethyl alcohol until their disappearance from the reaction mixture (by TLC). Products for which structures **5a** and **5b** are proposed were obtained from the reaction in high yield (72-78%).

It is important to note that the initial compound **4** is in reality a mixture of tautomers, 2-methylthio-6-oxo-1,6-dihydro-4-pyrimidinyl acetate **4A** and 6-hydroxy-2-methylthio-4-pyrimidinyl acetate **4B**. Theoretically it is possible to suggest also the formation of the tautomeric 2-methylthio-4-oxo-1,4-dihydro-6-pyrimidinyl acetate **4C**. However the results of quantum-chemical analysis, particularly ΔH_0 values of -78.718 for tautomer **4A**, -77.047 for tautomer **4B**, and -66.383 kcal/mol for tautomer **4C**, indicate that tautomers **4A,B** are thermodynamically more preferred. According to quantum-chemical analysis the most preferred derivatives of **4** are the conformers represented in Scheme 1 with the acetoxy group unfolded by ~30° relative to the plane of the pyrimidine ring.

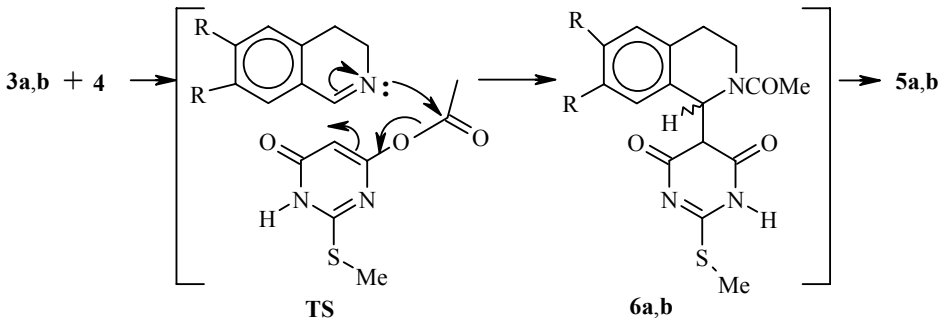
In the IR spectrum of enol acetate **4** (the characteristics of all the compounds synthesized are given in Experimental) a characteristic set of absorption bands (AB) is present in the region of the stretching vibrations of the C–H, N–H, C=O, C=N, and C=C bonds [8,9].

In the UV spectrum of compound **4** there are two AB in the 200-260 nm region, one as a shoulder on the long-wave slope of the very intense AB with a maximum located at 200 nm, and a broad AB at ~285 nm. Differentiation of the spectral contour enabled refinement of the position of the two primary AB located at 223.6 and 243.3 nm and showed that the long-wave AB is the result of the overlap of three AB with maxima at 274.4, 285, and 299.1 nm.

The ^{1}H and ^{13}C NMR spectra of enol acetate **4** (see Experimental) also confirm the structure assigned to it.

It should be noted particularly that the formation of no intermediate product was recorded by TLC in this reaction, either on boiling or at room temperature. This observation indicates that the process occurs by a coordinated mechanism through the six-membered transition state (**TS**) and product **6** and not as sequential Michael C–C addition and O–N isomerization of the acetyl group of adduct **6** into the desired compound **5**. On the other hand, the absence of products of replacement of the acetyl fragment by an appropriate residue of homologous acid on carrying out the reaction in the presence of homologous acids, indicates the intramolecular mechanism for the O–N migration of the acyl residue. This also indicates a preference for the mechanism represented in Scheme 2, since in precisely the six-membered transition state (**TS**) the optimum geometric and steric conditions of forming bonds and redistributing electrons are achieved.

Scheme 2



a R = H, **b** R = OMe

The composition and structure of products **5a,b** are confirmed by data of elemental analysis and physicochemical investigations. Specific features of their IR spectra are the broad AB at 2200-3100 cm^{-1} caused by the vibrations of the NH and OH groups of tautomers **5A** and **5B** and the absorption of the carbonyl groups characteristic of amides at 1640-1670 cm^{-1} [8]. The presence of bands assigned to the vibrations of the NH^{+} group in the spectra at 790-980 cm^{-1} [8, 9], is also notable. This may be caused by the significant contribution of betaine structures to the resulting mesomeric state.

In the electronic absorption spectra of compounds **5a,b** approximately three intense broadened and asymmetric AB are observed, and for the dimethoxy-substituted **5b** these AB are displaced towards the short-wave region of the spectrum. Differential analysis of the spectral contours showed that the AB observed at 220-240 and ~280 nm are composite and are caused by contributions of bands located at 212.3, 230.8, 259.1, 276.8, and 309.8 nm for compound **5a** and 205, 230.4, 267.5, 285, and 311.6 nm for compound **5b**.

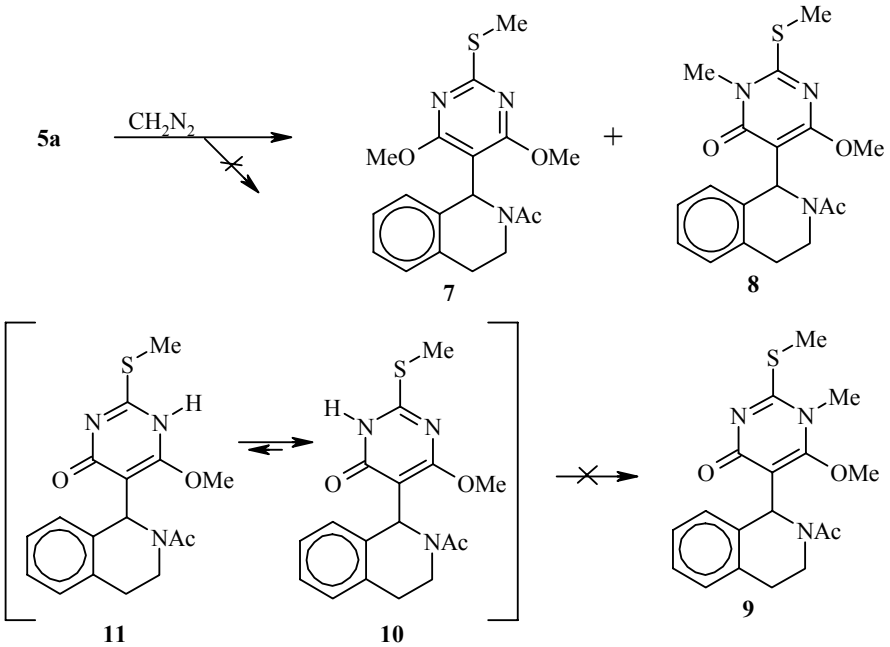
Investigation of compounds **5a,b** by NMR spectroscopy has shown that it is possible to observe the development of their tautomers depending on the solvent and temperature used. In the ^{1}H NMR spectra of compounds **5a,b** in $CDCl_3$ there was only one set of signals of resonance absorption while in the spectra of DMSO-d_6 solutions the signals of separate groups of protons were as a rule duplicated, corresponding to the existence of tautomerism. An analogous picture was also observed for the ^{13}C NMR spectra.

Signals for all the protons of the structures proposed were present in the ^{1}H NMR spectra of compounds **5a,b**. A noteworthy special feature of the spectra of compounds in $CDCl_3$ is the fact that the signals of the NH and OH groups are displayed as a two-proton broadened singlet at 12.50-13.00 ppm. This indicates the existence of rapid proton exchange. On reducing the temperature to -20°C, and then to -50 and -70°C a broadening of this singlet is observed, which is then split, at the minimum, into three broadened signals located at δ ~15, ~12, and ~10 ppm with different integral intensity and variable position, depending on the temperature. These data correlate with the results of theoretical ^{1}H NMR spectra for tautomers **5A** and **5B** of compound **5a**, assuming the disposition of proton signals for the NH and OH groups at 11 (NH), 12 (OH), and 16 ppm (OH), and as a result confirm the tautomeric multicenter character of compounds **5a,b**.

It should be noted that together with tautomers **5A** and **5B**, as in the case of enol acetate **4**, it is theoretically possible to assume the existence of tautomer **5C** as well. However, judging by the heat of formation (ΔH_0) of the indicated tautomers of -41.249 (**5A**), -39.702 (**5B**), and -30.781 kcal/mole (**5C**) it is possible to assert that tautomers **5A** and **5B** are the most populated. Optimization of the geometry of molecules **5a,b** shows that the pyridine ring of the isoquinoline fragment has a boat conformation and the pyrimidine fragment is almost perpendicular (100-105°) to the plane of benzene ring of the isoquinoline fragment, and its plane is unfolded to the latter. These data correlate satisfactorily with the results of X-ray structural analysis of cycloalkanedione analogs of compounds **5a,b** [10].

In the ^{13}C NMR spectrum (DMSO-d_6) of dimethoxy-substituted compound **5b**, associated signals were observed for a part of the signals, which, on the strength of that mentioned above, may be explained by the presence of two tautomers **5A** and **5B** in solution and also by the low (relative to the NMR time scale) rate of their interconversion. Signals which might have been assigned to tautomer **5C** were not observed either in the ^{1}H NMR spectra or in the ^{13}C NMR spectra.

Scheme 3



Further evidence in favor of the structure assigned to compounds **5a,b** was obtained on methylation of the first of them with diazomethane in methanol, as a result of which a mixture of two regiomerically methylated products was obtained. These were isolated in the individual state and characterized. The products were assigned the structures of the O,O- and N,O-dimethyl derivatives **7** and **8** respectively, their ratio in the

mixture was 2:3 (according to ^{1}H NMR spectral data). Strictly speaking, as a result of the interaction with diazomethane, which proceeds analogously to the methylation of barbiturates and pyrimidines studied previously [12], the product of N,O-methylation **9** might theoretically also be formed. The absence of the latter is caused by the thermodynamic unprofitability of N-N proton isomerization of the *ortho*-quinoid derivative **10,** formed as an intermediate, into the *para*-quinoid derivative **11**. Such an explanation is in good agreement with the data of quantum-chemical calculations showing that compounds **7** (ΔH_0 -41.249 kcal/mol) and **8** (ΔH_0 -39 702 kcal/mol) are energetically preferred to the compound **9** (ΔH_0 -30.781 kcal/mol).

The studied interaction of dihydroisoquinolines **3a,b** with the enol acetate of pyrimidine derivative **4** opens approaches to the synthesis of heterocyclic products combining in their structures the pharmacophoric fragments of tetrahydroisoquinoline and heterocyclic β-dicarbonyl compounds.

EXPERIMENTAL

The IR spectra were obtained on a Protege 460 instrument (KBr disks), and the UV spectra on a Specord M 400 spectrophotometer (in ethanol). The ^{1}H and ^{13}C NMR spectra were recorded on Bruker AC 200 (200 MHz for ^{1}H and 50 MHz for ^{13}C nuclei) and Bruker AM 500 (500 MHz for ^{1}H and 200 MHz for ^{13}C nuclei) radiospectrometers, internal standard was TMS. Mass spectra were obtained on a HP 5972 MS mass spectrometer, energy of ionizing electrons was 70 eV. Melting points were determined on a Boetius heating block. A check on the progress of reactions and the purity of products **4, 5a,b, 7**, and **8** was effected by TLC on Silufol UV 254 plates or silica gel 60 F_{254} Merck. Eluents were chloroform–methanol, 9:1 (**5a,b**); chloroform–ethyl acetate, 3:1 (**7, 8**); 2-propanol–water, 4:1 (**4**). The quantum-chemical calculations were carried out by the AM-1 method within the framework of the program set HyperChem 6.01 (Hypercub Inc., info@hyper.com).

3,4-Dihydroisoquinolines **3a,b** were obtained under the conditions of the Bischler–Napieralski reaction for the cyclodehydration of the appropriate formamides by the action of polyphosphoric acid (PFK) for compound **3a** or phosphorus oxychloride for compound **3b** [11].

2-Methylthio-6-oxo-1,6-dihydro-4-pyrimidinyl Acetate (4). 2-Methylthio-4,6-dihydroxypyrimidine [13] (15.8 g, 0.1 mol) was added to solution of Na_2CO_3 (10.6 g, 0.1 mol) in water (150 ml), and the mixture was stirred at 40°C until complete solution. Acetic anhydride (15.5 g, 0.15 mol) was added in small portions to the obtained solution during 5 min with vigorous stirring at 20°C. After the end of CO_2 evolution the reaction mixture was stirred for 30 min and maintained at 15°C for 4 h. The precipitated solid was filtered off, washed with water, and recrystallized from alcohol. Enol acetate **4** (10.6 g, 53%) was obtained as white finely prismatic crystals of mp 223°C. IR spectrum, ν, cm^{-1}: 3140, 3100, 3020, 2945, 2800-2890, 2730-2780, 2690, 1787, 1670, 1583, 1565, 1479, 1390, 1378, 1250, 1221, 1178, 1031, 991, 932, 894, 841, 760. UV spectrum, λ_{max}, nm (ε): 220.3 (14335), 240 (12630), 283.6 (14230); λ_{min}, nm (ε): 213.6 (13505), 235 (12170), 256.2 (6560). ^{1}H NMR spectrum (DMSO-d_6), δ, ppm: 2.26 (3H, s, CH_3C=O); 2.45 (3H, s, CH_3S); 5.92 (1H, s, H-5); 13.04 (1H, br. s, NH). ^{13}C NMR spectrum (DMSO-d_6), δ, ppm: 13.44 (CH_3S); 21.42 ($\underline{CH_3}C$=O); 97.79 ($C_{(5)}$); 164.05 ($C_{(2)}$); 166.24 ($C_{(4)}$ + $C_{(6)}$); 168.85 (OC=O). Found, %: C 41.87; H 3.98; N 13.85; S 15.94; $[M]^+$ 200. $C_7H_8N_2O_3S$. Calculated, %: C 41.99; H 4.03; N 13.99; S 16.01. *M* 200.21.

***rac*-5-(2-Acetyl-1,2,3,4-tetrahydro-1-isoquinolyl)-4-hydroxy-2-methylthio-1,6-dihydro-6-pyrimidinone (5a).** Mixture of compound **3a** (0.66 g, 5 mmol) and enol acetate **4** (1 g, 5 mmol) in ethyl alcohol (20 ml) was boiled for 3 h, then evaporated to 2/3 of initial volume and maintained at +5°C for ~16 h. The precipitated solid was filtered off, washed on the filter with ether, and recrystallized from alcohol–ether, 1:3. Product **5a** (1.13 g, 72.2%) was obtained as light cream prismatic crystals of mp 245-247°C. IR spectrum, ν, cm^{-1}: 2200-3050, 1636, 1600, 1584, 1550, 1480, 1445, 1430, 1388, 1322, 1280, 1221, 970, 925, 813, 796, 763, 750. UV spectrum, λ_{max}, nm (log ε): 208 (4.37), 242.4 (3.83), 284.3 (4.03); λ_{min}, nm (log ε): 236.6 (3.82), 256.4 (3.65). ^{1}H NMR spectrum ($CDCl_3$), δ, ppm (*J*, Hz): 2.18 (3H, s, $COCH_3$); 2.45 (3H, s, SCH_3); 2.92 (1H, dd, $J_{a,e\ gem}$ = 12.0,

$J_{a,a\ vic}$ = 5.0, $J_{a,e\ vic}$ = 5.0, H_a-4); 3.03 (1H, ddd, $J_{e,a\ gem}$ = 12.0, $J_{e,a\ vic}$ = 5.0, $J_{e,e\ vic}$ = 12.0, H_e-4); 3.88 (1H, dd, $J_{a,e\ gem}$ = 14.0, $J_{a,a\ vic}$ = 5.0, $J_{a,e\ vic}$ = 5.0, H_a–3); 4.18 (1H, ddd, $J_{e,a\ gem}$ = 15.0, $J_{e,e\ vic}$ = 12.0, $J_{e,a\ vic}$ = 5.0, H_e-3); 6.18 (1H, s, H-1); 6.90-6.99 (1H, m, H-6); 7.04 (3H, m, H-5, H-7, H-8); 12.50 (2H, br. s, OH, NH). ^{1}H NMR spectrum (DMSO-d_6), δ, ppm: 2.11 and 2.12 (3H, two s, $COCH_3$); 2.48 (3H, s, SCH_3); 2.78 (1H, m, H_a-4); 2.96 (1H, m, H_e-4); 3.60 and 4.14 (1H, two m, H_a-3); 3.96 and 4.61 (1H, two m, H_e-3); 6.09 and 6.17 (1H, two s, H-1); 6.99-7.10 (4H, m, H_{Ar}); 11.90 (2H, br. s, NH, OH). Found, %: C 57.90; H 5.11; N 12.54; S 9.76. $[M]^+$ 331. $C_{16}H_{17}N_3O_3S$. Calculated, %: C 57.99; H 5.17; N 12.68; S 9.67. *M* 331.40.

***rac*-5-(2-Acetyl-6,7-dimethoxy-1,2,3,4-tetrahydro-1-isoquinolyl)-4-hydroxy-2-methylthio-1,6-dihydro-6-pyrimidinone (5b).** Mixture of compound **3b** (0.96 g, 5 mmol) and enol acetate **4** (1 g, 5 mmol) in ethyl alcohol (30 ml) was boiled for 3 h. The crystals precipitated after cooling were filtered off, washed on the filter with ether, and recrystallized from chloroform–ether–hexane, 1:1:2. Product **5b** (1.53 g, 78.1%) was obtained as pale yellow prismatic crystals of mp 251-252°C. IR spectrum, ν, cm^{-1}: 2830-3100, 2500-2800, 1670, 1640, 1607, 1583, 1554, 1525, 1460-1485, 1339, 1264, 1226, 1215, 1200, 1130, 865, 795. UV spectrum, λ_{max}, nm (log ε): 204.6 (4.74), 224 (4.20), 281.2 (4.07); λ_{min}, nm, (log ε): 253.9 (3.62). ^{1}H NMR spectrum ($CDCl_3$), δ, ppm (*J*, Hz): 2.20 (3H, s, $COCH_3$); 2.52 (3H, s, SCH_3); 2.86 (1H, dd, $J_{a,e\ gem}$ = 12.0, $J_{a,a\ vic}$ = 5.0, $J_{a,e\ vic}$ = 5.0, H_a-4); 2.98 (1H, ddd, $J_{e,a\ gem}$ = 12.0, $J_{e,a\ vic}$ = 5.0, $J_{e,e\ vic}$ = 12.0, H_e-4); 3.76 (3H, s, OCH_3); 3.85 (3H, s, OCH_3); 3.88 (1H, dd, $J_{a,e\ gem}$ = 15.0, $J_{a,a\ vic}$ = 5.0, $J_{a,e\ vic}$ = 5.0, H_a-3); 4.17 (1H, ddd, $J_{e,a\ gem}$ = 15.0, $J_{e,e\ vic}$ = 12.0, $J_{e,a\ vic}$ = 5.0, H_e-3); 6.16 (1H, s, H-1); 6.42 (1H, s, H-5); 6.60 (1H, s, H-8); 12.80 (2H, br. s, OH, NH). ^{1}H NMR spectrum (DMSO-d_6), δ, ppm: 2.06 and 2.08 (3H, two s, $COCH_3$); 2.47 (3H, s, SCH_3); 2.60-2.80 (2H, m, 2H-4); 3.54 and 4.02 (1H, m, H_a-3); 3.58 (3H, s, OCH_3); 3.72 (3H, s, OCH_3'); 3.86 and 4.52 (1H, two m, H_e-3); 6.00 and 6.18 (1H, two s, H-1); 6.46 and 6.51 (1H, two s, H-5); 6.70 and 6.71 (1H, two s, H-8); 11.95 (2H, br. s, NH, OH). ^{13}C NMR spectrum (DMSO-d_6), δ, ppm: 13.01 (SCH_3); 22.03 and 22.26 ($\underline{CH_3}C$=O); 28.39 and 29.97 ($C_{(4)}$); 37.95 and 43.02 ($C_{(3)}$); 48.81 and 50.74 ($C_{(1)}$); 55.95 (CH_3O); 56.18 (CH_3O); 101.24 and 101.37 ($C_{(5')}$); 110.39 ($C_{(5)}$); 112.55 ($C_{(8)}$); 127.01 and 128.03 ($C_{(8a)}$); 128.37 ($C_{(4a)}$); 147.91, 148.14 ($C_{(6)}$, $C_{(7)}$); 161.15 ($CH_3\underline{C}$=O); 165.40 ($C_{(4')}$, $C_{(6')}$); 170.02 and 170.15 ($C_{(2')}$). Found, %: C 55.19; H 5.37; N 10.69; S 8.27; $[M]^+$ 391. $C_{18}H_{21}N_3O_5S$. Calculated, %: C 55.23; H 5.41; N 10.73; S 8.29. *M* 391.44.

Methylation of the mixture of tautomers of compound **5a**, **1-[1-(4,6-dihydroxy-2-methylthio-5-pyrimidinyl)-1,2,3,4-tetrahydro-2-isoquinolyl]-1-ethanone (5A) and (2-acetyl-1,2,3,4-tetrahydro-1-isoquinolyl)-4-hydroxy-2-methylthio-1,6-dihydro-6-pyrimidinone (5B).** Gaseous diazomethane, obtained from hydrazine hydrate, chloroform, and potassium hydroxide by the method of [14], was passed into suspension of compound **5a** (0.5 g) in absolute methanol (20 ml) at 10°C. After forming a solution with a stable yellow color, the mixture was left for 1 h, brought to room temperature, and then water (7 ml) was added. The precipitate formed was separated and twice recrystallized from heptane. **1-[1-(4,6-Dimethoxy-2-methylthio-5-pyrimidinyl)-1,2,3,4-tetrahydro-2-isoquinolyl]-1-ethanone (7)** (0.009 g, 1.7%) was obtained as white needle-like crystals of mp 97-98°C. R_f 0.45. ^{1}H NMR spectrum ($CDCl_3$), δ, ppm: 2.12 and 2.13 (3H, two s, $COCH_3$); 2.51 and 2.53 (3H, two s, SCH_3); 2.85-3.05 (2H, m, H-4); 3.61 and 4.00 (1H, two m, H_a-3); 3.90 and 3.92 (6H, two s, OCH_3, OCH_3'); 3.94 and 4.37 (1H, two m, H_e-9); 6.24 and 6.59 (1H two s, H-1); 6.77-7.16 (4H, m, H_{Ar}). The aqueous methanol mother liquor was diluted with water to 60 ml, the precipitated solid was separated and recrystallized from CCl_4. **5-(2-Acetyl-1,2,3,4-tetrahydro-1-isoquinolyl)-1-methyl-4-methoxy-2-methylthio-1,6-dihydro-6-pyrimidinone (8)** (0.007 g, 1.3%) was obtained as white needle-like crystals of mp 143-144°C. R_f 0.25. ^{1}H NMR spectrum (DMSO-d_6), δ, ppm: 2.22 and 2.27 (3H, two s, $COCH_3$); 2.59 (3H, s, SCH_3); 2.82-3.04 (2H, m, 2H-4); 3.51 and 3.98 (1H, two m, H_a-3); 3.84 and 4.09 (3H, two s, OCH_3, OCH_3'); 4.08 and 4.79 (1H, two m, H_e-3); 6.38 and 6.50 (1H, two m, H-1); 6.81-7.16 (4H, m, H_{Ar}).

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