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SYNTHESIS OF 5*H*-THIAZOLO[3,2-*a*]PYRIMIDINES

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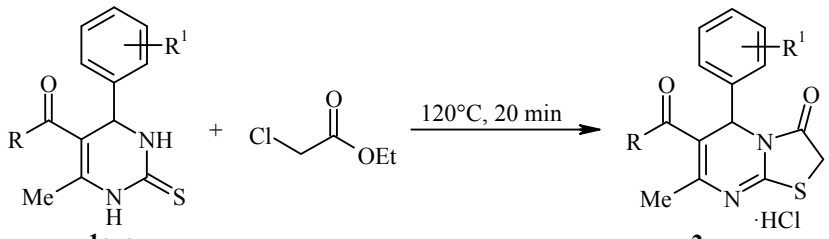
A convenient method has been developed for preparing 5H-thiazolo[3,2-a]pyrimidines by the reaction of tetrahydropyrimidine-2-thiones with halocarboxylic acid esters or with 3-bromopentane-2,4-dione.

Keywords: 3-bromopentane-2,4-dione, halocarboxylic acid esters, tetrahydropyrimidines, thiazolopyrimidines, cyclization.

The search for convenient methods of preparing dihydropyrimidines [1, 2] has been motivated by their ability to regulate the action of calcium channels [3]. Significantly less attention has been devoted to studying the chemical properties of dihydropyrimidines, but work has recently appeared relating to the synthesis of condensed dihydropyrimidine derivatives [3] in connection to their broad spectrum of biological activity. 5*H*-Thiazolo[3,2-*a*]pyrimidines are glutamate receptor antagonists [4] and acetylcholinesterase inhibitors [5], and they show anti-inflammatory, antiparkinsonian, and antiherpes activity [6].

In the synthesis of thiazolo[3,2-*a*]pyrimidines from 1,2,3,4-tetrahydropyrimidine-2-thiones, cyclization can occur at either the N-1 or N-3 atoms of the pyrimidine ring [7]. In the majority of cases, cyclization happens at the N-3 atom to give 5*H*-thiazolo[3,2-*a*]pyrimidines, e.g. when treating tetrahydropyrimidine-2-thiones with α-haloketones [4, 5], 1,2-dibromoethane [7, 8], α-halocarboxylic acids [7, 8] or their acyl halides [9], methyl chloroacetate [10], or bromomalonodinitrile [11]. However, methyl 6-methyl-4-phenyl-2-thioxo-1,2,3,4-tetrahydropyrimidine-5-carboxylate cyclizes with iodoacetamide at the N-1 atom, to give a 7*H*-thiazolo[3,2-*a*]pyrimidine [12].

With the aim of studying the selectivity of this cyclization, we have treated different pyrimidine-2-thiones **1a-g** with α-halocarboxylates and with 3-bromopentane-2,4-dione. We have found that the reaction of pyrimidine-2-thiones **1a-g** with chloroacetate does not require the use of triethylamine (as previously reported [10]) and the 5*H*-thiazolo[3,2-*a*]pyrimidines **2a-g** are formed simply by heating the reagents without solvent.



1a–g **2a–g**

a–**e** R = OEt, **f**, **g** R = Me; **a**, **f** R^1 = H, **b** R^1 = 2-OH, **c** R^1 = 2-Cl, **d** R^1 = 2-NO_2, **e**, **g** R^1 = 4-OMe

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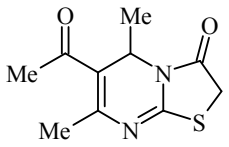
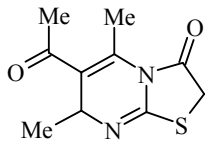
The thiazolopyrimidines **2a-g** were isolated as hydrochlorides with high melting points. They are characterized by a high frequency IR absorption band in the range of 1774-1754 cm^{-1}, which is assigned to the carbonyl group stretching vibration of the thiazolone ring. The methylene groups in this bicyclic structure are diastereotopic, and their ^1H NMR signals appear as doublets with a rather large spin-spin coupling, or in the form of multiplets. The selectivity of the cyclization at the tetrahydropyrimidine ring N-3 atom is confirmed by the appearance of ^{13}C NMR signals for the carbon atoms C-5 (53.9-55.7 ppm) and C-7 (145.0-148.6 ppm) and a ^1H NMR signal at 5.61-6.20 ppm for the proton on the C-5 atom within a narrow range for all of the synthesized thiazolopyrimidines **2a-g** with the exception of the nitro derivative **2d**, which shows a C-5 signal (50.0 ppm) and its bonded proton (6.74 ppm). The signals of the indicated atoms should differ markedly for the two possible 5*H*- and 7*H*-thiazolo[3,2-*a*]pyrimidine isomers. For each of compounds **2a-g** these signals do not deviate far from the mean values and correspond to literature data for ethyl 7-methyl-3-oxo-5-phenyl-2,3-dihydro-5*H*-[1,3]thiazolo[3,2-*a*]-pyrimidine-6-carboxylate in $CDCl_3$: 53.9 (C-5), 150.8 (C-7), and 6.05 ppm (H-5) [6]. This data provides arguments against assigning the thiazolo[3,2-*a*]pyrimidines **2b-d** (having an *ortho* substituent in the benzene ring at C-5 atom) to 7*H*-isomers, in spite of the fact that an *ortho* substituent in the benzene ring at the C-4 atom of a dihydropyrimidine ring can direct an electrophilic attack to the N-1 atom [3].

To examine a possible cyclization at the N-1 atom, we have carried out reactions of *N*-3-substituted tetrahydropyrimidine-2-thiones with ethyl chloroacetate. For the reaction of ethyl 3-acetyl-6-methyl-4-phenyl-2-thioxo-1,2,3,4-tetrahydropyrimidine-5-carboxylate (prepared by method [7]) the reaction does not stop at the sulfur atom alkylation stage, but proceeds further to form the 5*H*-thiazolopyrimidine **2a**. During the reaction, deacetylation of the N-3 atom occurs leading to the more stable 5*H*-isomer **2a** and pointing to a thermodynamic control. Attempts to alkylate the ethyl 3-(2-cyanoethyl)-6-methyl-4-phenyl-2-thioxo-1,2,3,4-tetrahydro-pyrimidine-5-carboxylate (prepared by method [13]) with ethyl chloroacetate proved unsuccessful. The reaction did not occur, probably due to steric hindrance from the cyanoethyl group. Computational analysis of model compounds by the B3LYP/6-31G* method (GAMESS program [14], correspondence of the optimized geometry to energy minimum was checked by calculation of vibrational frequencies) confirmed that the more stable product is that arising from cyclization at the N-3 atom: 6-acetyl-5,7-dimethyl-5*H*-[1,3]thiazolo[3,2-*a*]-pyrimidin-3(2H)-one (**2h**) is by 27.7 kJ/mol more stable than 6-acetyl-5,7-dimethyl-7*H*-[1,3]thiazolo[3,2-*a*]-pyrimidin-3(2H)-one (**2i**), very likely as a result of pyrimidine ring double bond conjugation in the structure of **2h**.

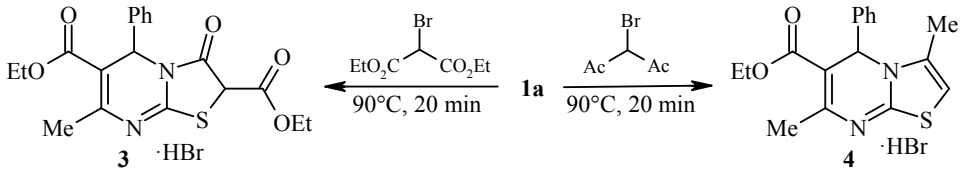


2h



2i

The reaction of the pyrimidinethione **1a** with bromomalonate, 3-bromoacetoacetic ester, and 3-bromopentane-2,4-dione also occurs upon heating. The reaction with the bromomalonate forms a mixture of diastereomeric diethyl 7-methyl-3-oxo-5-phenyl-2,3-dihydro-5*H*-[1,3]thiazolo[3,2-*a*]pyrimidine-2,6-dicarb-oxylates (**3**). The reaction with the 3-bromopentane-2,4-dione gives ethyl 3,7-dimethyl-5-phenyl-5*H*-[1,3]thiazolo[3,2-*a*]pyrimidine-6-carboxylate (**4**), which is formed through loss of the acetyl group in a process that is likely catalyzed by the hydrogen bromide liberated in the course of the reaction. When treating the pyrimidinethione **1a** with 3-bromoacetoacetate, a mixture of difficult to separate products was obtained, apparently because of the primary alkylation product cyclization both at the acetyl and at the ester group and also possibly as a result of loss of the acetyl group.



3 **4**

Hence we have discovered a convenient and simple method for preparing 5-aryl-5*H*-thiazolo[3,2-*a*]-pyrimidines by heating 5-aryl-1,2,3,4-tetrahydropyrimidine-2-thiones with 2-halocarboxylates and have shown that a single isomer is formed in the reaction, most likely due to the thermodynamic factor.

EXPERIMENTAL

IR spectra were recorded on a Shimadzu FTIR8500S instrument for KBr pellets. ^{1}H and ^{13}C NMR spectra were recorded on a JEOL JNM ECX-400 instrument (400 and 100 MHz, respectively) using DMSO-d_6 with TMS as internal standard. Elemental analysis was performed on a EuroVector EA-3000 instrument. Melting points were determined on a PTP-2 apparatus.

Ethyl 5-Aryl-7-methyl-3-oxo-2,3-dihydro-5*H*-[1,3]thiazolo[3,2-*a*]pyrimidine-6-carboxylate Hydrochlorides 2a-e and 6-Acetyl-5-aryl-7-methyl-5*H*-[1,3]thiazolo[3,2-*a*]pyrimidin-3(2*H*)-one Hydrochlorides 2f,g (General Method). A mixture of ethyl chloroacetate (3.0 ml, 35 mmol) and the corresponding pyrimidine-2-thione **1a-g** [15] (3.6 mmol) was heated for 20 min at 120ºC. The precipitate formed was filtered off and washed with EtOAc.

Ethyl 7-Methyl-3-oxo-5-phenyl-2,3-dihydro-5*H*-[1,3]thiazolo[3,2-*a*]pyrimidine-6-carboxylate Hydrochloride (2a). Yield 0.85 g (67%). Yellow crystals; mp 220-222ºC (decomp.). IR spectrum, ν, cm^{-1}: 1762 (N–C=O), 1712 (CO_2Et). ^{1}H NMR spectrum, δ, ppm (*J*, Hz): 1.06 (3H, t, ^3J = 7.3, $CH_2C\underline{H}_3$); 2.35 (3H, s, 7-CH_3); 3.91-4.08 (2H, m, $C\underline{H}_2CH_3$); 4.14 (1H, d, ^2J = 18.1) and 4.19 (1H, d, ^2J = 18.1, 2-CH_2); 5.86 (1H, s, 5-CH); 7.20-7.35 (5H, m, H Ph); 8.01 (1H, br. s, N$^+$H). ^{13}C NMR spectrum, δ, ppm: 14.4 ($CH_2\underline{C}H_3$); 21.2 (7-CH_3); 33.9 (2-CH_2); 55.5 (5-CH); 60.8 ($\underline{C}H_2CH_3$); 108.0 (C-6); 128.2 (C Ph); 129.1 (C Ph); 129.2 (C Ph); 140.4 (C Ph); 148.6 (C-7); 164.8 (C-8a); 164.9 (<u>C</u>OOEt); 171.8 (C-3). Found, %: C 54.23; H 4.59; N 7.62. $C_{16}H_{17}ClN_2O_3S$. Calculated, %: C 54.47; H 4.86; N 7.94.

Ethyl 5-(2-Hydroxyphenyl)-7-methyl-3-oxo-2,3-dihydro-5*H*-[1,3]thiazolo[3,2-*a*]pyrimidine-6-carboxylate Hydrochloride (2b). Yield 0.48 g (36%). Yellow crystals; mp 220-224ºC (decomp.). IR spectrum, ν, cm^{-1}: 1761 (N–C=O), 1705 (CO_2Et). ^{1}H NMR spectrum, δ, ppm (*J*, Hz): 1.00 (3H, t, ^3J = 6.9, $CH_2C\underline{H}_3$); 1.92 (3H, s, 7-CH_3); 3.95-4.05 (2H, m, $C\underline{H}_2CH_3$); 4.19 (1H, ^2J = 17.5) and 4.24 (1H, d, ^2J = 17.5, 2-CH_2); 5.61 (1H, s, 5-CH); 6.50 (1H, br. s, OH); 6.88-7.00 (2H, m, H Ar); 7.20-7.30 (2H, m, H Ar); 10.30 (1H, br. s, N$^+$H). ^{13}C NMR spectrum, δ, ppm: 14.3 ($CH_2\underline{C}H_3$); 21.0 (7-CH_3); 34.1 (2-CH_2); 55.5 (5-CH); 60.8 ($\underline{C}H_2CH_3$); 108.0 (C-6); 128.1 (C Ar); 128.2 (C Ar); 129.1 (C Ar); 129.2 (C Ar); 129.3 (C Ar); 140.4 (C Ar); 148.5 (C-7); 157.5 (C-8a); 164.9 (<u>C</u>OOEt); 171.4 (C-3). Found, %: C 52.32; H 4.96; N 7.28. $C_{16}H_{17}ClN_2O_4S$. Calculated, %: C 52.10; H 4.65; N 7.59.

Ethyl 5-(2-Chlorophenyl)-7-methyl-3-oxo-2,3-dihydro-5*H*-[1,3]thiazolo[3,2-*a*]pyrimidine-6-carboxylate Hydrochloride (2c). Yield 0.85 g (61%). Yellow crystals; mp 220-223ºC (decomp.). IR spectrum, ν, cm^{-1}: 1774 (N–C=O), 1689 (CO_2Et). ^{1}H NMR spectrum, δ, ppm (*J*, Hz): 1.02 (3H, t, ^3J = 6.9, $CH_2C\underline{H}_3$); 2.31 (3H, s, 7-CH_3); 3.90-4.00 (2H, m, $C\underline{H}_2CH_3$); 4.10 (1H, d, ^2J = 17.9) and 4.16 (1H, d, ^2J = 17.9, 2-CH_2); 6.20 (1H, s, 5-CH); 7.22-7.43 (4H, m, H Ar); 7.78 (1H, br. s, N$^+$H). ^{13}C NMR spectrum, δ, ppm: 14.4 ($CH_2\underline{C}H_3$); 21.2 (7-CH_3); 33.4 (2-CH_2); 53.9 (5-CH); 60.8 ($\underline{C}H_2CH_3$); 107.0 (C-6); 128.2 (C Ar); 130.2 (C Ar); 130.7 (C Ar); 131.8 (C Ar); 133.2 (C Ar); 137.7 (C Ar); 148.6 (C-7); 164.8 (<u>C</u>OOEt, C-8a); 171.2 (C-3). Found, %: C 49.40; H 4.49; N 7.05. $C_{16}H_{16}Cl_2N_2O_3S$. Calculated, %: C 49.62; H 4.16; N 7.23.

Ethyl 7-Methyl-5-(2-nitrophenyl)-3-oxo-2,3-dihydro-5*H*-[1,3]thiazolo[3,2-*a*]pyrimidine-6-carboxylate Hydrochloride (2d). Yield 0.69 g (48%). Yellow crystals; mp 240-243ºC (decomp.). IR spectrum, ν, cm^{-1}: 1774 (N–C=O), 1712 (CO_2Et). ^1H NMR spectrum, δ, ppm (*J*, Hz): 0.92 (3H, t, ^3J = 6.3, $CH_2C\underline{H}_3$); 2.31 (3H, s, 7-CH_3); 3.77-3.95 (2H, m, $C\underline{H}_2CH_3$); 4.02 (2H, s, 2-CH_2); 6.74 (1H, s, 5-CH); 7.40-7.60 (2H, m, H Ar); 7.66-7.76 (1H, m, H Ar); 7.90 (1H, d, ^3J = 6.8, H Ar); 7.95 (1H, br. s, N^+H). ^{13}C NMR spectrum, δ, ppm: 14.2 ($CH_2\underline{C}H_3$); 22.9 (7-CH_3); 32.8 (2-CH_2); 50.0 (5-CH); 60.6 ($\underline{C}H_2CH_3$); 106.6 (C-6); 125.0 (C Ar); 130.4 (2C Ar); 133.2 (C Ar); 134.9 (C Ar); 148.6 (C-7); 152.5 (C Ar); 162.1 (C-8a); 165.1 (COOEt); 171.8 (C-3). Found, %: C 48.69; H 4.38; N 10.83. $C_{16}H_{16}ClN_3O_5S$. Calculated, %: C 48.31; H 4.05; N 10.56.

Ethyl 5-(4-Methoxyphenyl)-7-methyl-3-oxo-2,3-dihydro-5*H*-[1,3]thiazolo[3,2-*a*]pyrimidine-6-carboxylate Hydrochloride (2e). Yield 0.90 g (65%). Yellow crystals; mp 214-216ºC (decomp.). IR spectrum, ν, cm^{-1}: 1760 (N–C=O), 1710 (CO_2Et). ^1H NMR spectrum, δ, ppm (*J*, Hz): 1.06 (3H, t, ^3J = 7.3, $CH_2C\underline{H}_3$); 2.35 (3H, s, 7-CH_3); 3.69 (3H, s, OCH_3); 3.95-4.05 (2H, m, $C\underline{H}_2CH_3$); 4.15 (1H, d, ^2J = 18.2) and 4.21 (1H, d, ^2J = 18.2, 2-CH_2); 5.84 (1H, s, 5-CH); 6.87 (2H, d, ^3J = 7.8, H Ar); 7.19 (2H, d, ^3J = 7.8, H Ar); 8.4 (1H, br. s, N^+H). ^{13}C NMR spectrum, δ, ppm: 14.4 ($CH_2\underline{C}H_3$); 20.7 (7-CH_3); 34.2 (2-CH_2); 55.1 (OCH_3); 55.7 (5-CH); 60.9 ($\underline{C}H_2CH_3$); 108.2 (C-6); 114.4 (C Ar); 114.5 (C Ar); 129.7 (C Ar); 132.3 (C Ar); 147.5 (C-7); 159.9 (C-8a); 164.8 (COOEt); 171.4 (C-3). Found, %: C 53.01; H 5.22; N 7.10. $C_{17}H_{19}ClN_2O_4S$. Calculated, %: C 53.33; H 5.00; N 7.32.

6-Acetyl-7-methyl-5-phenyl-5*H*-[1,3]thiazolo[3,2-*a*]pyrimidine-3(2*H*)-one Hydrochloride (2f). Yield 0.60 g (52%). Yellow crystals; mp 226-229ºC (decomp.). IR spectrum, ν, cm^{-1}: 1759 (N–C=O), 1655 (CH_3CO). ^1H NMR spectrum, δ, ppm (*J*, Hz): 2.20 (3H, s, CH_3CO); 2.38 (3H, s, 7-CH_3); 4.18 (1H, d, ^2J = 18.2) and 4.24 (1H, d, ^2J = 18.2, 2-CH_2); 6.03 (1H, s, 5-CH); 7.24-7.35 (5H, m, H Ph); 8.77 (1H, br. s, N^+H). ^{13}C NMR spectrum, δ, ppm: 21.2 (7-CH_3); 31.3 ($C\underline{H}_3CO$); 34.5 (2-CH_2); 55.4 (5-CH); 117.1 (C-6); 128.5 (C Ph); 129.3 (C Ph); 129.4 (C Ph); 139.6 (C Ph); 145.4 (C-7); 165.8 (C-8a); 171.3 (C-3); 196.6 (Me$\underline{C}$O). Found, %: C 55.49; H 4.85; N 8.60. $C_{15}H_{15}ClN_2O_2S$. Calculated, %: C 55.81; H 4.68; N 8.68.

6-Acetyl-5-(4-methoxyphenyl)-7-methyl-5*H*-[1,3]thiazolo[3,2-*a*]pyrimidin-3(2*H*)-one Hydrochloride (2g). Yield 0.66 g (42%). Yellow crystals; mp 223-225ºC (decomp.). IR spectrum, ν, cm^{-1}: 1754 (N–C=O), 1654 (CH_3CO). ^1H NMR spectrum, δ, ppm (*J*, Hz): 2.19 (3H, s, CH_3CO); 2.39 (3H, s, 7-CH_3); 3.70 (3H, s, OCH_3); 4.19 (2H, s, 2-CH_2); 6.00 (1H, s, 5-CH); 6.88 (2H, d, ^3J = 7.8, H Ar); 7.22 (2H, d, ^3J = 7.8, H Ar); 8.55 (1H, br. s, N^+H). ^{13}C NMR spectrum, δ, ppm: 21.2 (7-CH_3); 31.1 ($C\underline{H}_3CO$); 34.4 (2-CH_2); 54.9 (OCH_3); 55.7 (5-CH); 114.5 (C Ph); 114.6 (C Ph); 117.0 (C-6); 129.9 (C Ph); 131.6 (C Ph); 145.0 (C-7); 160.0 (C-8a); 171.4 (C-3); 196.7 (Me$\underline{C}$O). Found, %: C 54.64; H 4.77; N 7.62. $C_{16}H_{17}ClN_2O_3S$. Calculated, %: C 54.47; H 4.86; N 7.94.

5-Phenyl-5*H*-[1,3]thiazolo[3,2-*a*]pyrimidine hydrobromides 3 and 4 were prepared similarly to the thiazolopyrimidines **2a-g** by heating the ethyl 6-methyl-4-phenyl-2-thioxo-1,2,3,4-tetrahydropyrimidine-5-carboxylate (**1a**) (1.0 g, 3.6 mmol) at 90ºC for 20 min with bromomalonic ester or 3-bromopentane-2,4-dione (35.0 mmol), respectively.

Diethyl 7-Methyl-3-oxo-5-phenyl-2,3-dihydro-5*H*-[1,3]thiazolo[3,2-*a*]pyrimidine-2,6-dicarboxylate Hydrobromide (3). Yield 0.91 g (59%). Yellow crystals; mp 150-153ºC (decomp.). IR spectrum, ν, cm^{-1}: 1747 (N–C=O), 1720 (CO_2Et). ^1H NMR spectrum, δ, ppm (J, Hz): 1.06 (6H, t, ^3J = 7.1, $2CH_2C\underline{H}_3$); 2.35 (3H, s, 7-CH_3); 3.95-4.05 (4H, m, $2C\underline{H}_2CH_3$); 5.58 (1H, s, 5-CH); 5.75 (1H, br. s, 2-CH); 7.20-7.40 (5H, m, H Ph); 8.50 (1H, br. s, N^+H). ^{13}C NMR spectrum, δ, ppm: 14.2 ($CH_2\underline{C}H_3$); 14.4 ($CH_2\underline{C}H_3$); 17.7 (7-CH_3); 52.9 (2-CH); 55.9 (5-CH); 60.9 ($\underline{C}H_2CH_3$); 63.4 ($\underline{C}H_2CH_3$); 104.9 (C-6); 127.5 (C Ph); 129.1 (C Ph); 129.3 (C Ph); 141.2 (C Ph); 144.6 (C-7); 156.7 (C-8a); 164.5 (C=O); 164.8 (C=O); 165.3 (C=O). Found, %: C 48.81; H 4.44; N 5.69. $C_{19}H_{21}BrN_2O_5S$. Calculated, %: C 48.62; H 4.51; N 5.97.

Ethyl 3,7-dimethyl-5-phenyl-5*H*-[1,3]thiazolo[3,2-*a*]pyrimidine-6-carboxylate Hydrochloride (4). Yield 0.66 g (52%). Yellow crystals; mp 248-251ºC (decomp.). IR spectrum, ν, cm^{-1}: 1677 (CO_2Et). ^1H NMR spectrum, δ, ppm (*J*, Hz): 1.15 (3H, t, ^3J = 7.1, $CH_2C\underline{H}_3$); 2.17 (3H, s, 3-CH_3); 2.39 (3H, s, 7-CH_3); 4.00-4.12 (2H, m, $C\underline{H}_2CH_3$); 6.40 (1H, s, 5-CH); 7.17 (1H, s, H-2); 7.25-7.40 (5H, m, H Ph); 12.71 (1H, br. s, N^+H). ^{13}C NMR spectrum, δ, ppm: 13.4 (CH_3); 14.5 (CH_3); 18.2 (CH_3); 59.0 (5-CH); 61.1 ($\underline{C}H_2CH_3$); 103.2 (C-2);

109.8 (C-6); 127.8 (C Ph); 129.7 (C Ph); 129.8 (C Ph); 138.1 (C Ph); 140.6 (C-3(7)); 142.6 (C-7(3)); 160.9 (C-8a); 164.5 (C=O). Found, %: C 51.88; H 4.63; N 6.92. $C_{17}H_{19}BrN_2O_2S$. Calculated, %: C 51.65; H 4.84; N 7.09.

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