Chemistry of Heterocyclic Compounds, Vol. 39, No. 8, 2003

HETEROCYCLIZATION REACTIONS OF 2-(2-PROPYNYLTHIO)-4(1H)-QUINAZOLINONE DERIVATIVES WHEN TREATED WITH ELECTROPHILIC AND NUCLEOPHILIC REAGENTS

Yu. L. Zborovskii[1], V. V. Orysyk[2], A. A. Dobosh[2], V. I. Staninets[1], V. V. Pirozhenko[1], and A. N. Chernega[1]

Reaction of the potassium salt of 2-thioquinazolin-4-one with propargyl halides leads to formation of 2-propargylthioquinazolin-4-one derivatives, which undergo heterocyclization when they are treated with electrophilic or nucleophilic reagents and, depending on the nature of the cyclization agent, leads to formation of tricyclic systems with angular or linear structure.

Keywords: angular and linear triazoloquinazolinones, electrophilic and nucleophilic reagents, heterocyclization.

Derivatives of thiazolo[2,3-*b*]quinazolinone with linear structure, obtained by condensation of the methyl ester of anthranilic acid with propargyl isothiocyanates, exhibit high physiological activity: they have anti-inflammatory and sedative properties, and are muscle relaxants and tranquilizers [1].

With the aim of developing new methods for obtaining derivatives of thiazoloquinazolinone, we studied heterocyclization of 2-propargylthioquinazolin-4-ones when treated with a number of electrophilic and nucleophilic reagents. The latter, depending on their nature, enable us to obtain condensed heterocyclic systems with both linear and angular structure.

Thus we established that the reaction of quinazolinone **2** with bromine or iodine in acetic acid leads to formation of hydropolyhalides of the angular 2-halomethylidenedihydrothiazoloquinazolin-5-one **3a,b**. Treatment of the latter with an aqueous solution of sodium acetate yields the corresponding bases **4a,b**.

Such cyclization can also be carried out by treatment with sodium ethoxide. In this case, thiazoloquinazolinone **5** is formed, which also has an angular structure.

Using conc. H_2SO_4 as the cyclization agent leads to formation of thiazoloquinazolinone **6**, with linear structure.

The different directions of the considered reactions is probably due to differences in the mechanisms of these conversions.

Sulfuric acid forms a salt with participation of the more basic nitrogen atom N(1) of compound **2**, which blocks this nucleophilic reaction center in the cyclization step and ultimately leads to the linear dihydrothiazoloquinazolinone **7**, which then is isomerized to compound **6**. In contrast, sodium methoxide

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[1] Institute of Organic Chemistry, National Academy of Sciences of Ukraine, Kiev 02094; e-mail: iochkiev@ukrpack.net. [2] Uzhgorod National University, Uzhgorod 88000, Ukraine. Translated from Khimiya Geterotsiklicheskikh Soedinenii, No. 8, pp. 1255-1262, August, 2003. Original article submitted February 27, 2001.

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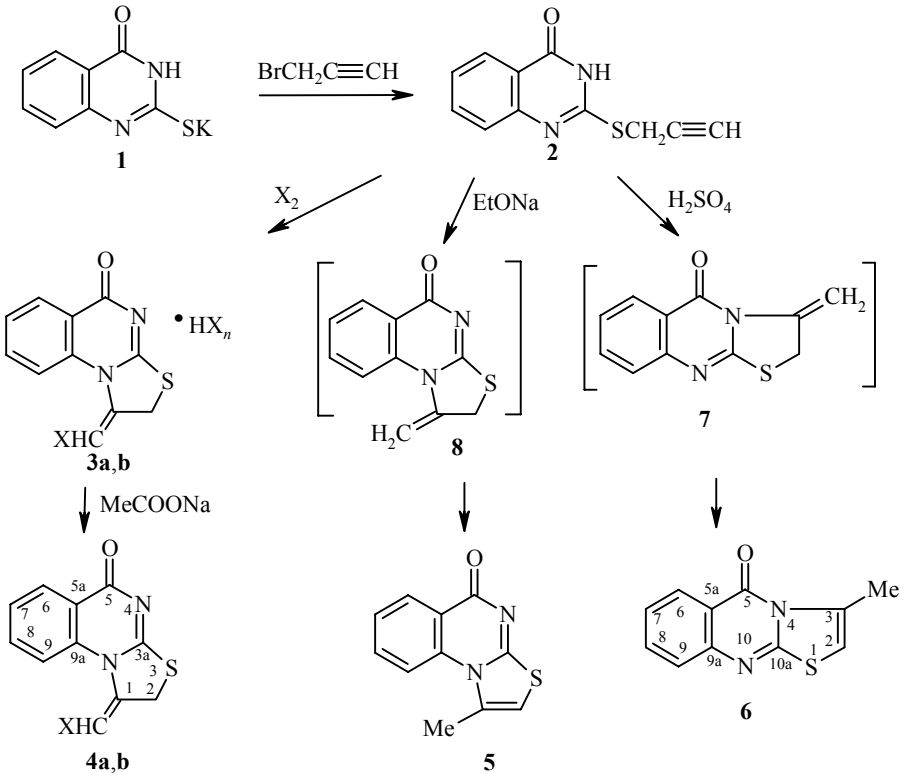
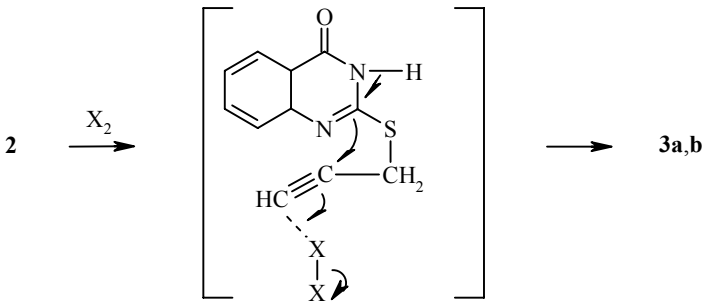


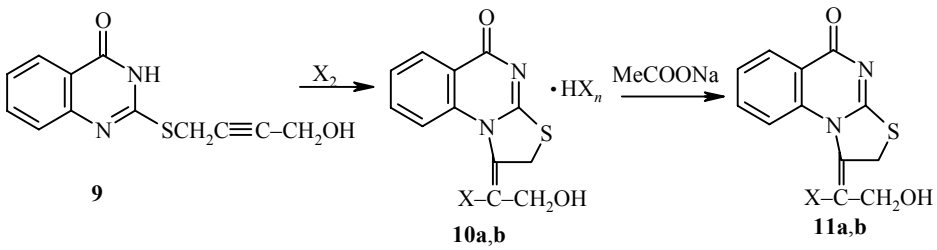
3, **4 a** X = Br, **b** X = I; **3 a** *n* = 3, **b** *n* = 5

deprotonates the N(3) atoms, so the intermediate **8** in this case has an angular structure. For heterocyclization occurring on treatment with halogens, a synchronous donor–acceptor mechanism can be suggested according to which the most nucleophilic atom N(1) participates in the cyclization, while the hydrogen halide molecule is separated only in the last step of the process and so cannot affect the direction of the reaction.

Heterocyclization of quinazolinone **9**, in contrast to compound **2**, could be carried out only by treatment with halogens.



Sodium ethoxide cleaves this substrate to form compound **1**, while conc. H_2SO_4 results in tar formation. An attempt to use a mixture of sulfuric and acetic acids as the cyclization agent likewise did not lead to the target thiazoloquinazolinone. In this case, 2-(4-acetoxy-2-butynylthio)-4(1H)-quinazolinone is formed as the major reaction product.



10, **11 a** X = Br, **b** X = I; **10 a** *n* = 3, **b** *n* = 5

The composition and structure of all the synthesized compounds were confirmed by elemental analysis, ^{1}H NMR and IR spectra, while the structure of compounds **4a,b, 5, 6, 11b** was also confirmed by ^{13}C NMR spectra (Tables 1-3).

We should note that the positions of the absorption bands for the stretching vibrations of the (C=O) bond of the carbonyl group in the IR spectra are quite different for thiazoloquinazolinones with linear and angular structures. Thus in the IR spectra of linear derivatives, this band is located in the interval 1695-1670 cm^{-1}, while for angular derivatives it is located in the 1665-1640 cm^{-1} region [2, 3]. This fact was used to identify the synthesized compounds. Furthermore, the structure of compound **11b** was confirmed by X-ray diffraction (Fig. 1, Table 4).

The tricyclic system S(1)N(1)N(2)C(1-10) is considerably non-planar (deviations of atoms from the mean-square plane are as high as 0.45 Å). The central six-membered heterocycle N(1)N(2)C(1)C(2)C(3)C(8) is planar or approximately planar (the corresponding atoms deviate from the mean-square plane by 0.05-0.11 Å) and is slightly distorted toward a *twist–boat* conformation (modified Cremer–Pople parameters [4] *S*, ψ, and θ

TABLE 1. Characteristics of Synthesized Compounds

Compound	Empirical formula	Found, % / Calculated, %					mp, °C
		C	H	Hal	N	S	
2	$C_{11}H_8N_2OS$	60.52 / 61.09	3.65 / 3.73	—	12.90 / 12.95	14.69 / 14.83	219-220
3a	$C_{11}H_8Br_4N_2OS$	24.51 / 24.66	1.40 / 1.50	58.10 / 59.64	5.17 / 5.23	5.81 / 5.98	189-193
3b	$C_{11}H_8I_6N_2OS$	13.40 / 13.51	0.79 / 0.82	75.90 / 77.88	2.79 / 2.87	3.20 / 3.28	173-176
4a	$C_{11}H_7BrN_2OS$	44.74 / 44.76	2.37 / 2.39	27.12 / 27.07	9.49 / 9.49	10.84 / 10.86	184-186
4b	$C_{11}H_7IN_2OS$	38.41 / 38.61	2.00 / 2.06	37.01 / 37.09	8.09 / 8.19	9.27 / 9.37	230-232
5	$C_{11}H_8N_2OS$	61.09 / 61.09	3.67 / 3.73	—	12.92 / 12.95	14.79 / 14.83	299-300
6	$C_{11}H_8N_2OS$	61.05 / 61.09	3.69 / 3.73	—	12.89 / 12.95	14.75 / 14.83	167-169
9	$C_{12}H_{10}N_2O_2S$	58.41 / 58.52	4.00 / 4.09	—	11.21 / 11.37	12.82 / 13.02	216-217
10a	$C_{12}H_{10}Br_4N_2O_2S$	25.21 / 25.47	1.52 / 1.78	54.22 / 56.48	4.85 / 4.95	5.42 / 5.67	194-197
10b	$C_{12}H_{10}I_6N_2O_2S$	14.10 / 14.30	0.89 / 1.00	73.21 / 75.56	2.65 / 2.78	3.01 / 3.18	136-138
11a	$C_{12}H_9BrN_2O_2S$	44.17 / 44.32	2.54 / 2.79	24.39 / 24.57	8.42 / 8.61	9.59 / 9.86	188-190
11b	$C_{12}H_9IN_2O_2S$	38.59 / 38.73	2.30 / 2.44	34.05 / 34.10	7.46 / 7.53	8.43 / 8.62	213-214

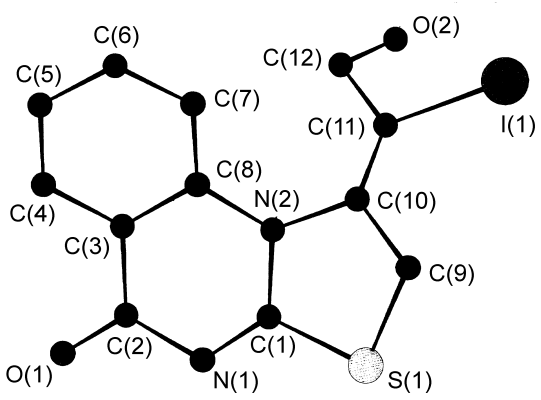


Fig. 1. General form of molecule **11b** with numbering of the atoms. Hydrogen atoms are not shown.

are respectively 0.26, 2.2°, and 79.3°). The benzene ring C(3-8) forms a dihedral angle of 9.4° with this ring. The five-membered heterocycle S(1)N(2)C(1)C(9)C(10) has an *envelope* conformation: the S(1), N(2), C(1), and C(10) atoms are coplanar within 0.05 Å, while the C(9) atom deviates from this plane by 0.52 Å (the dihedral angle between the S(1)N(2)C(1)C(10) and S(1)C(9)C(10) planes is 30.9°). The "base" of this *envelope*

TABLE 2. Spectral Data for Compounds Obtained

Compound	^{1}H NMR spectrum, δ, ppm (*J*, Hz)	IR spectrum, ν(C=O), cm^{-1}
2	3.21 (1H, t, *J* = 3.0, –C≡CH); 4.10 (2H, d, *J* = 3.0, CH_2); 7.45 (1H, m, ArH); 7.56 (1H, m, ArH); 7.78 (1H, m, ArH); 8.05 (1H, m, ArH); 12.65 (1H, br. s, NH)	1680
3a	4.37 (2H, br. s, CH_2); 7.30 (1H, br. s, CHBr); 7.53 (1H, m, ArH); 7.81 (1H, m, ArH); 7.94 (1H, m, ArH); 8.07 (1H, m, ArH)	1715
3b	4.33 (2H, br. s, CH_2); 7.20 (1H, br. s, CHI); 7.52 (1H, m, ArH); 7.80 (1H, m, ArH); 7.92 (1H, m, ArH); 8.06 (1H, m, ArH)	1710
4a	4.35 (2H, br. s, CH_2); 7.28 (1H, br. s, CHBr); 7.51 (1H, m, ArH); 7.78 (1H, m, ArH); 7.92 (1H, m, ArH); 8.05 (1H, m, ArH)	1655
4b	4.32 (2H, br. s, CH_2); 7.17 (1H, br. s, CHI); 7.50 (1H, m, ArH); 7.77 (1H, m, ArH); 7.89 (1H, m, ArH); 8.05 (1H, m, ArH)	1650
5	2.84 (3H, s, CH_3); 7.01 (1H, s, S–CH); 7.61 (1H, m, ArH); 7.79 (1H, m, ArH); 8.25 (2H, m, ArH)	1640
6	2.80 (3H, s, CH_3); 6.85 (1H, s, S–CH); 7.43 (1H, m, ArH); 7.54 (1H, m, ArH); 7.80 (1H, m, ArH); 8.15 (1H, m, ArH)	1695
9	4.07 (2H, br. s, C$\underline{H_2}$OH); 4.16 (2H, s, S–CH_2); 5.17 (1H, t, *J* = 6.0, OH); 7.49 (2H, m, ArH); 7.77 (1H, m, ArH); 8.05 (1H, m, ArH); 12.61 (1H, br. s, NH)	1685
10a	3.89 (d) and 4.05 (d) (2H, *J* = 14, C$\underline{H_2}$–OH); 4.41 (d) and 4.51 (d) (2H, *J* = 13, S–CH_2); 7.44 (2H, m, ArH); 7.76 (1H, m, ArH); 7.99 (1H, m, ArH)	1710
10b	3.70 (d) and 3.93 (d) (2H, *J* = 14, C$\underline{H_2}$–OH); 4.39 (d) and 4.55 (d) (2H, *J* = 12.5, S–CH_2); 7.30 (1H, m, ArH); 7.46 (1H, m, ArH); 7.76 (1H, m, ArH); 7.99 (1H, m, ArH)	1710
11a	3.88 (d) and 4.04 (d) (2H, *J* = 14, C$\underline{H_2}$–OH); 4.39 (d) and 4.50 (d) (2H, *J* = 13, S–CH_2); 5.44 (1H, s, OH); 7.43 (2H, m, ArH); 7.75 (1H, m, ArH); 7.98 (1H, m, ArH)	1655
11b	3.71 (d) and 3.94 (d) (2H, *J* = 14, C$\underline{H_2}$–OH); 4.37 (d) and 4.54 (d) (2H, *J* = 13, S–CH_2); 5.42 (1H, t, *J* = 5, OH); 7.29 (1H, m, ArH); 7.45 (1H, m, ArH); 7.74 (1H, m, ArH); 7.98 (1H, m, ArH)	1660

TABLE 3. ^{13}C NMR Spectra of Thiazoloquinazolinone Derivatives

Compound	Chemical shifts, δ, ppm					
	C(1)	C(2)	C(3)	C(3a)	C(5)	C(5a)
4a	137.28	31.64	—	165.37	168.14	118.06
4b	141.63	30.68	—	161.64	162.17	122.90
5	135.88	105.67	—	163.96	167.85	118.89
6	—	104.81	134.54	—	160.27	117.44
11b	134.40	39.94	—	165.99	168.46	117.86

Compound	Chemical shifts, δ, ppm					
	C(6, 7, 8, 9)	C(9a)	C-10a	–C–X	$–CH_2OH$	$–CH_3$
4a	133.91, 127.92, 126.18, 116.48	138.21	—	97.88	—	—
4b	137.91, 134.82, 127.20, 115.37	148.56	—	91.91	—	—
5	132.98, 128.17, 126.97, 116.84	138.21	—	—	—	19.29
6	134.60, 126.48, 125.26, 124.77	147.47	159.84	—	—	17.99
11b	133.11, 127.22, 125.42, 116.72	138.64	—	100.05	64.77	—

TABLE 4. Major Bond Lengths (*d*) and Bond Angles (ω) in the Molecule of Compound **11b**

Bond	*d*, Å	Angle	ω, deg.
I(1)–C(11)	2.092(7)	C(1)–S(1)–C(9)	91.9(4)
S(1)–C(1)	1.737(7)	C(1)–N(1)–C(2)	117.8(6)
S(1)–C(9)	1.819(9)	C(1)–N(2)–C(8)	119.1(6)
O(1)–C(2)	1.22(1)	C(1)–N(2)–C(10)	113.2(6)
O(2)–C(12)	1.39(1)	C(8)–N(2)–C(10)	126.7(6)
N(1)–C(1)	1.29(1)	S(1)–C(1)–N(1)	122.6(6)
N(1)–C(2)	1.40(1)	S(1)–C(1)–N(2)	111.8(5)
N(2)–C(1)	1.376(9)	N(1)–C(1)–N(2)	125.7(7)
N(2)–C(8)	1.395(9)	N(1)–C(2)–C(3)	117.6(7)
N(2)–C(10)	1.436(9)	C(2)–C(3)–C(8)	119.7(7)
C(2)–C(3)	1.483(12)	N(2)–C(8)–C(3)	116.5(7)
C(3)–C(8)	1.396(11)	S(1)–C(9)–C(10)	103.5(5)

S(1)N(2)C(1)C(10) forms a dihedral angle of 11.3° with the central 6-membered heterocycle. The N(2) atom has a trigonal planar configuration of the bonds: the sum of the bond angles at this atom is 359.0(1.8)°. The conjugation between the lone electron pair of the N(2) atom and the π-systems of the C(1)=N(1) double bond and the benzene ring C(3-8) results in shortening of the N(2)–C(1) and N(2)–C(8) bonds to 1.376(9) and 1.395(8) Å compared with the standard value of 1.45 Å for N(sp^2)–C(sp^2) single bonds [5].

EXPERIMENTAL

The IR spectra were taken on a UR-10 in KBr disks; the ^1H and ^{13}C spectra were obtained on a Varian-VXR (300 MHz) in DMSO-d_6, internal standard TMS.

TABLE 5. Coordinates of Atoms and Equivalent Isotropic Thermal Parameters U_{eq} ($Å^2 \times 10^3$) in Structure **11b**

Atom	*x*	*y*	*z*	U_{eq}
I(1)	1.19963(7)	0.19093(2)	0.92273(7)	0.0551
S(1)	0.5712(3)	0.20760(9)	0.4123(3)	0.0595
O(1)	0.0600(9)	0.0777(3)	0.4760(11)	0.0805
O(2)	0.934(1)	0.1810(2)	1.1761(8)	0.0636
N(1)	0.293(1)	0.1375(3)	0.4446(8)	0.0536
N(2)	0.6170(9)	0.1240(2)	0.6459(8)	0.0469
C(1)	0.4756(11)	0.1512(3)	0.5050(9)	0.0470
C(2)	0.2331(12)	0.0895(3)	0.5232(11)	0.0543
C(3)	0.3863(11)	0.0506(3)	0.6412(11)	0.0528
C(4)	0.3377(13)	-0.0042(3)	0.6914(11)	0.0585
C(5)	0.4801(14)	-0.0408(3)	0.7917(12)	0.0621
C(6)	0.6722(13)	-0.0234(3)	0.8432(12)	0.0588
C(7)	0.7265(12)	0.0300(3)	0.7975(11)	0.0527
C(8)	0.5804(11)	0.0685(3)	0.700(1)	0.0497
C(9)	0.8194(12)	0.1850(3)	0.5371(11)	0.0522
C(10)	0.8013(11)	0.1534(3)	0.699(1)	0.0458
C(11)	0.9206(11)	0.1565(3)	0.870(1)	0.0469
C(12)	0.8780(12)	0.1377(3)	1.043(1)	0.0522

X-ray Diffraction Study of a Single Crystal of Compound 11b with linear dimensions 0.25 × 0.35 × 0.45 mm was done at room temperature on an Enraf-Nonius CAD-4 automatic four-circle diffractometer (CuKα radiation, scanning rate ratio ω/2θ = 1.2, θmax = 60°, *hkl* range $0 \le h \le 9$, $0 \le k \le 26$, $-9 \le l \le 9$). We collected 2043 reflections total, of which 1821 are symmetrically independent (*R* factor used for averaging, 0.044). The crystals of compound **11b** are monoclinic, a = 7.292(2), b = 22.975(3), c = 7.760(1) Å, β = 109.59(2)°, V = 1224.8 Å3, M = 372.18, Z = 4, $d$calc = 2.02 g/cm3, μ = 223.16 cm-1, space group $P21/c$. The structure was deciphered by the direct method and least-squares refined in the full-matrix anisotropic approximation using the CRYSTALS software package [6]. In the refinement, we used 1589 reflections with $I > 3\sigma(I)$ (163 refined parameters, the number of reflections divided by the parameter 9.7). The hydrogen atoms were located geometrically and included in the calculation with fixed positions and thermal parameters. In the refinement, we used the Chebyshev weighting scheme [7] with parameters 6.12, -2.94, 2.74, -2.03, and -1.39. The final values of the *R* factors were R = 0.057 and R_w = 0.063, *GOOF* = 1.095. The residual electron density from a Fourier difference series was 0.65 and -1.29 e/Å3. The atomic coordinates are given in Table 5. The full set of crystallographic data for structure **11b** has been deposited in the Cambridge Structural Database (Reg. No. CCDC 155073).

2-(2-Propynylthio)-4(1H)-quinazoline (2). An 80% solution of propargyl bromide in toluene (6.1 ml) was added to a solution of (10.8 g, 50 mmol) of 2-mercapto-4(1H)-quinazolinone potassium salt **1** in a mixture of ethanol (65 ml) and water (15 ml). The reaction mixture was refluxed on a water bath for 45 min. The crystalline precipitate was filtered out after cooling, washed with water and then alcohol, and dried. It was recrystallized from a 4:1 ethanol–DMF mixture. Yield of quinazolinone **2**: 9.07 g (84%).

2-(4-Hydroxy-2-butynylthio)-4(1H)-quinazolinone (9) was obtained from the potassium salt **1** and 1-chloro-2-butyn-4-ol as for compound **2** and recrystallized from a 1:1 ethanol–dioxane mixture. Yield 79%.

1-Bromomethylidene-1,2-dihydro-5H-thiazolo[3,2-*a*]quinazolin-5-onium Tribromide (3a). Compound **2** (0.432 g, 2 mmol) were dissolved in glacial acetic acid (70 ml) at a temperature of 50-60°C. A solution of bromine (0.21 m, 4 mmol) in glacial acetic acid (15 ml) was added dropwise with stirring to the solution obtained, at a temperature of 18-20°C. The mixture was stirred for another 2 h and allowed to stand for

12 h to crystallize. The fine crystalline precipitate of the orange salt **3a** was washed on the filter with acetic acid and then ether, and then was dried. Yield 0.98 g (92%).

1-Iodomethylidene-1,2-dihydro-5H-thiazolo[3,2-*a*]quinazolin-5-onium Pentaiodide (3b). A solution of iodine (1.52 g, 6 mmol) in CH_3COOH (110 ml) was added with stirring to a solution of compound **2** (0.432 g, 2 mmol) in glacial acetic acid (70 ml) at 18-20°C. After 24 hours, a fine crystalline brown precipitate was formed, which was filtered out, washed with alcohol and then ether, and then dried. Yield of salt **3b**: 1.85 g (95%).

1-Bromomethylidene- and 1-Iodomethylidene-1,2-dihydro-5H-thiazolo[3,2-*a*]quinazolin-5-one (4a) and (4b). An 20% aqueous solution of sodium acetate (20-25 ml) was added to a solution of the corresponding salt **3a** or **3b** (2 mmol) in DMSO (15-20 ml), stirred, and allowed to stand for 2 h. The fine crystalline white precipitate formed was filtered out, washed with water, then alcohol, and then ether, and then dried. Quinazolinones **4a** and **4b** were recrystallized respectively from ethanol and a 1:1 ethanol–dioxane mixture. Yield of compound **4a**: 0.38 g (65%). Yield of compound **4b**: 0.48 g (70%).

1-Methyl-5H-thiazolo[3,2-*a*]quinazolin-5-one (5). Compound **2** (0.432 g, 2 mmol) was added to a 7% solution of sodium ethoxide in ethanol (8 ml) at 10-15°C. The mixture was carefully stirred until the precipitate completely dissolved, and the solution was allowed to stand at 18-20°C. After 24 hours, the crystalline precipitate was filtered out, washed with water, and dried. It was recrystallized from a 1:1 ethanol–DMF mixture. Yield of compound **5**: 0.35 g (80%).

3-Methyl-5H-thiazolo[2,3-*b*]quinazolin-5-one (6). Compound **2** (0.432 g, 2 mmol) were dissolved in conc. H_2SO_4 (5 ml) at 0°C. The solution was allowed to stand at 18-20°C for 12 h, and then was poured into water (50 ml) cooled down to 0°C. After 12 h, the fine crystalline precipitate of quinazolinone **6** formed was filtered out, washed with water, and dried. It was recrystallized from a 1:1 ethanol–dioxane mixture. Yield of compound **6**: 0.32 g (75%).

1-(1-Bromo-2-hydroxyethylidene)-1,2-dihydro-5H-thiazolo[3,2-*a*]quinazolin-5-onium Tribromide (10a). A solution of bromine (0.21 ml, 4 mmol) in CH_3COOH (25 ml) was added dropwise with continuous stirring over a 2 h period to a suspension of compound **9** (0.49 g, 2 mmol) in glacial acetic acid (50 ml), after which stirring was continued for another 2 h. After 15 h, the fine crystalline red-orange material formed was separated by filtration, washed with ether, and dried in air. Yield of salt **10a**: 1.04 g (92%).

1-(2-Hydroxy-1-iodoethylidene)-1,2-dihydro-5H-thiazolo[3,2-*a*]quinazolin-5-onium Pentaiodide (10b). Iodine (1.52 g, 6 mmol) in CH_3COOH (140 ml) was added dropwise with continuous stirring over a 3 h period to a suspension of compound **9** (0.49 g, 2 mmol) in glacial acetic acid (60 ml). Stirring was continued for another 2 h. After 30 h, the fine crystalline black precipitate formed of salt **10b** was separated by filtration, washed with ether, and dried in air. Yield of salt **10b**: 1.80 g (90%).

Bases 11a and 11b. An 20% aqueous solution of sodium acetate (15-20 ml) were added to a solution of salt **10a** or **10b** (2 mmol) in DMSO (20 ml). The corresponding bases were crystallized from a solution over a 12-14 h period. In order to remove tarry impurities from the bases, they were ground with acetone (50-60 ml).

1-(1-Bromo-2-hydroxyethylidene)-1,2-dihydro-5H-thiazolo[3,2-*a*]quinazolin-5-one (11a). Yield 0.42 g (65%).

1-(2-Hydroxy-1-iodoethylidene)-1,2-dihydro-5H-thiazolo[3,2-*a*]quinazolin-5-one (11b). Yield 0.54 g (72%).

REFERENCES

1. F. Gerard, M. Jean Pierre, and P. Jean Varie, French Patent Application 2393001; *Chem. Abstr.* **91**:175390 (1979).
2. L. I. Giannola, S. Polazzo, L. Lamartina, L. R. Sanseverino, and P. Sabatino, *J. Chem. Soc., Perkin Trans. I*, 2095 (1986).

3. M. Gutschow and S. Leistner, *Synthesis*, 1488 (1995).
4. N. S. Zefirov and V. A. Palyulin, *Dokl. Akad. Nauk SSSR*, **252**, 111 (1980).
5. M. Burke-Laing and M. Laing, *Acta Crystallogr.*, **B32**, 3216 (1976).
6. D. J. Watkin, C. K. Prout, J. R. Carruthers, and P. W. Betteridge, *CRYSTALS*, Issue 10, Chemical Crystallography Laboratory, University of Oxford (1996).
7. J. R. Carruthers and D. J. Watkin, *Acta Crystallogr.*, **A35**, 698 (1979).