The Influence of Condensed Cycle on Regiochemistry of Electrophilic Heterocyclization of 3-Alkenyl-2-Thioxopyrimidin-4-One by *p*-Alkoxyphenyltellurium Trichloride

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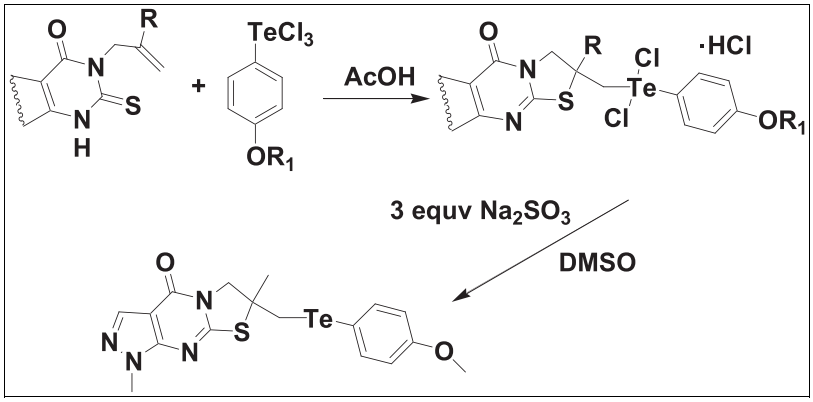

Electrophilic heterocyclization of 5-alkenyl-1-methyl-6-thioxopyrazolo[3,4-*d*]pyrimidin-4-ones and 3-alkenyl-2-thioxoquinazoline-4-ones under the action of *p*-alkoxyphenyltellurium trichloride leads to annulation of thiazoline cycle with formation of 7-[(*p*-alkoxyphenyl)telluromethyl]-1-methyl-6,7-dihydropyrazolo[3,4-*d*][1,3]thiazolo[3,2-*a*]pyrimidin-4(1*H*)-ones hydrochlorides and 2-(*p*-alkoxyphenyl) dichlorotelluromethyl-2,3-dihydro-5*H*-[1,3]thiazolo[2,3-*b*]quinazolin-5-ones hydrochlorides. Reduction of salts by the action of excess of sodium sulfite leads to formation of arylhetaryl telluride.

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INTRODUCTION

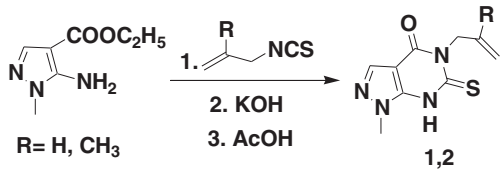
Reactions of electrophilic heterocyclization are widely used for the synthesis of various heterocyclic polycyclic systems [1–7]. Some resources describe the impact of different factors (the nature of the electrophilic reagent, the polarization of the alkenyl substituent, the nucleophilicity of the exo-heteroatom or endocyclic heteroatom, the nature of the condensed ring, and the reaction conditions) on the regiochemistry of the process of electrophilic intramolecular cyclization of alkenyl derivatives of heterocycles [2,5,6,8]. Tellurium-containing electrophiles in such reactions are not studied enough, especially that the aryltellurium trichlorides are the most perspective, because of high stability of corresponding products of electrophilic heterocyclization [9–13]. There are data [9–12] about usage of aryltellurium trihalogenides in the reactions with unsaturated O-nucleophiles, namely, their interaction with unsaturated alcohols, phenols, and acids. Our previous investigation of reactions of aryltellurium trihalogenides with unsaturated mercapto-derivatives of N-alkenyl derivatives 5,6-disubstituted 2-thioxothieno[2,3-*d*]pyrimidin-4-one was described in the paper [13]. Therefore, the aim of our study is the investigation of the effect of nature of condensed cycle on 3-alkenyl-2-thioxopyrimidin-4-one onto the regiochemistry of electrophilic action of the aryltellurium trichlorides.

RESULTS AND DISCUSSION

The 5-alkenyl-6-thioxo-1-methylpyrazolo[3,4-*d*]pyrimidin-4-ones **1** and **2** have been used as model compounds for the study of tellurocyclization; they were synthesized from 5-amino-4-carbetoxy-1-methylpyrazole and the corresponding alkenyl isothiocyanates by the Scheme 1.

Heterocyclization of 5-alkenyl-6-thioxo-1-methylpyrazolo [3,4-*d*]pyrimidin-4-ones with the *p*-methoxyphenyltellurium trichloride was carried out in acetic acid medium, chloroform, acetonitrile at different temperatures. The usage of acetic acid at the room temperature was found as optimal conditions for cyclization. In results, the linear polycyclic systems **3** and **4** with the salt-like structure and exocyclic aryltellurium fragment were obtained (Scheme 2). It is found that cyclization of 5-alkenyl-6-thioxo-1-methylpyrazolo[3,4-*d*]pyrimidin-4-ones with the *p*-methoxyphenyltellurium trichloride proceeds regiospecifically with the formation of hydrochlorides 7-[dichloro(4-methoxyphenyl)-telluromethyl]-1-methyl-6,7-

Scheme 1. Synthesis of 5-alkenyl-6-thioxo-1-methylpyrazolo[3,4-*d*] pyrimidin-4-ones **1** and **2**.

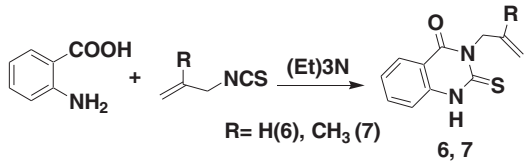


dihydropyrazolo[3,4-*d*][1,3]thiazolo[3,2-*a*]pyrimidin-4(1*H*)-ones **3** and **4** (Scheme 2). Obviously, the addition of aryltellurium trichloride to exocyclic bond C═C is before the heterocyclization process; moreover, the electrophile acts on the terminal carbon of alkenyl fragment. The next intramolecular cyclization is occured on the sulfur atom with the annelation of the thiazolidine cycle. The structure and composition of obtained tellurium-containing polycyclic heterocycles were confirmed by nuclear magnetic resonance (NMR) spectra, chromatography-mass spectrometry, and elemental analysis. Results of spectral investigation well agree with the data of halogenation for similar objects [14]. Thus, type of condensed heterocycle on alkenylthioxopirimidinone does not effect on regiochemistry of process and yields of cyclized products.

The chemical properties of synthesized tellurium-containing polycondensed heterocyclic compounds were studied. The action of triple excess of sodium sulfite on the solution of compound **4** in dimethyl sulfoxide (DMSO) leads to dehydrochlorination of salt **4** and dehalogenation of Tellurium atom, what were confirmed by elemental analysis for compound **5** (Scheme 2). It is important that the reduction by Na_2SO_3 does not split the bond tellurium–carbon as described for the action of sodium borohydride [11].

For investigation of influence of condensed cycle in pyrimidine moiety in reactions of electrophilic cyclization, pirazol cycle was changed on benzene cycle, which electronic density is less. The 3-alkenyl-2-thioxoquinazoline-4-ones **6** and **7** were synthesized from anthranilic acid for these purposes (Scheme 3).

Scheme 3. Synthesis of 3-alkenyl-2-thioxoquinazoline-4-ones **6** and **7**.



Electrophilic cyclization of 3-alkenyl-2-thioxoquinazoline-4-ones **6** and **7** with the *p*-methoxy(ethoxy)phenyltellurium trichloride was carry out under the same conditions. As a result, 2-(*p*-alkoxyphenyl)dichlorotelluromethyl-2,3-dihydro-5*H*-[1,3] thiazolo[2,3-*b*]quinazolin-5-ones **8**–**11** were synthesized. The composition of compounds was proved by NMR spectra and elemental analysis. It is found that the refusing of electronic density of condensed benzene ring does not effect on regiochemistry of heterocyclyzation process; however, the yields of cyclized products are significantly reduced. The influence of alkyl substituent in electrophilic reagent is not significant (Scheme 4).

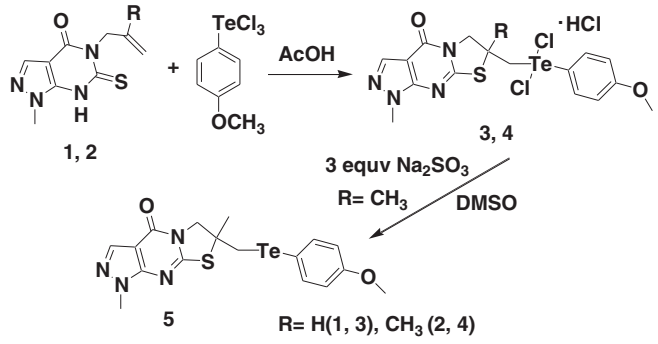
CONCLUSION

Thus, a changing of electronic density of condensed cycle in 3-alkenyl-2-thioxopyrimidin-4-one does not effect on regiochemistry of electrophilic intramolecular cyclization process under the action of aryltellurium trichlorides and leads to the formation of linear annulation of thiazolidine cycle to condensed pyrimidine.

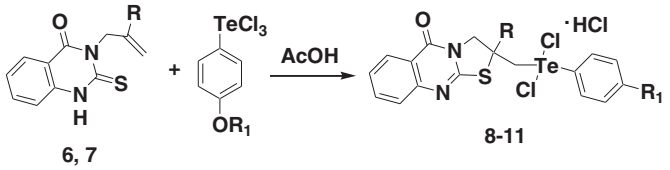
EXPERIMENTAL

The spectra of ^{1}H NMR (400 MHz) and ^{13}C NMR (100 MHz) were recorded on Varian Mercury-400 instruments (Enamine Ltd, Kiyv, Ukraine). The chemical shift values (δ) are given in parts per million (ppm) downfield from TMS as internal standard and are referred

Scheme 2. Electrophilic heterocyclization of 5-alkenyl-6-thioxo-1-methylpyrazolo[3,4-*d*]pyrimidine-4-ones by *p*-methoxyphenyltellurium trichloride.



Scheme 4. Electrophilic heterocyclization of 3-alkenyl-2-thioxoquinazoline-4-ones by *p*-methoxyphenyltellurium trichloride.



6, 7 8-11

R= H (6, 8, 10), CH_3 (7, 9, 11)
R_1= CH_3 (8, 9), C_2H_5 (10, 11)

to the residual peak of the deuterated solvent used $(CD_3)_2SO$. Mass spectra were recorded on an Agilent 1100 LCMSD SL instrument (chemical ionization; atmospheric pressure chemical ionization) and an Agilent 5890 Series II 5972 GCMS instrument (electron impact) (Enamine Ltd, Kiyv, Ukraine). The melting points were determined on Stuart SMP30 instrument (UK). Elemental analyses were performed on Elementar Vario MICRO cube analyzer (Elementar-Straße 1, Langenselbold, Germany). All reagents were obtained from commercial suppliers and used without any further purification. Methallyl isothiocyanate was synthesized from Fizer [15]. Dry solvents were prepared according to the standard methods. The *p*-methoxyphenyltellurium trihalides were received according to described procedure [16].

General procedure for the preparation of 5-alkenyl-6-thioxo-1-methylpyrazolo[3,4-*d*]pyrimidine-4-ones. The 0.05 mol of 5-amino-4-carbethoxy-1-methylpyrazole [17] was dissolved in 30 mL of ethanol. The 0.05 mol of alkenylisothiocianate was added to pre-prepared solution of 5-amino-4-carbethoxy-1-methylpyrazole. The mixture was refluxed for 8 h. Then, 0.1 mol of potassium hydroxide in 5 mL of water was added. Reaction mixture was refluxed for 2 h and cooled, and the yielded solution was precipitated by acetic acid. Product was filtered, washed by water, and recrystallized from ethanol.

1-Methyl-5-(prop-2-en-1-yl)-1,5,6,7-tetrahydro-4H-pyrazolo [3,4-d]pyrimidin-4-one (1). Yield 61%; white powder (ethanol); mp. 214–215°C; ^{1}H NMR (400 MHz, DMSO-d_6): δ (ppm) 13.31 (s, 1H), 8.47 (s, 1H), 5.87 (m, 1H), 5.10 (d, *J* = 8.0 Hz, 1H), 5.07 (d, *J* = 14.0 Hz, 1H), 4.99 (d, *J* = 5.0 Hz, 2H), 3.90 (s, 3H). *Anal*. Calcd for $C_9H_{10}N_4OS$: C, 48.64; H, 4.53; N, 25.21; S, 14.42. Found: C, 48.51; H, 4.45; N, 25.03; S, 14.28.

1-Methyl-5-(2-methylprop-2-en-1-yl)-1,5,6,7-tetrahydro-4H-pyrazolo[3,4-d]pyrimidin-4-one (2). Yield 61%; white powder (ethanol); mp. 207–208°C. ^{1}H NMR (400 MHz, DMSO-d_6): δ (ppm) 13.31 (s, 1H), 8.47 (s, 1H), 4.87 (s, 2H), 4.73 (s, 1H), 4.43 (s, 1H), 3.90 (s, 3H), 1.72 (s, 3H). *Anal*. Calcd for $C_{10}H_{12}N_4OS$: C, 50.83; H, 5.12; N, 23.71; S, 13.57. Found: C, 50.64; H, 4.96; N, 23.52; S, 13.43.

General procedure for the preparation of 7-(*p*-methoxyphenil) dichloromethyl-1,4,6,7-tetrahydropyrazolo[3,4-*d*][1,3]-thiazolo [3,2-*a*]pyrimidine-4-ones hydrochlorides. The 1 mmol of 5-alkenyl-6-thioxo-1-methylpyrazolo[3,4-*d*]pyrimidine-4-one **1** or **2**, dissolved in 20 mL of acetic acid, was added to 1 mmol of *p*-methoxyphenyltellutium trichloride in 20 mL of acetic acid. The reaction mixture was stirring at room temperature for 8 h. Target product was filtered and recrystallized from acetic acid.

7-[Dichloro(4-methoxyphenyl)tellouromethyl]-1-methyl-6,7-dihydropyrazolo[3,4-d][1,3]thiazolo[3,2-a]pyrimidin-4(1H)-one hydrochloride (3). Yield 70%; white powder (acetic acid); mp. 156–158°C. ^{1}H NMR (400 MHz, DMSO-d_6): δ (ppm) 8.46 (s, 1H), 8.05 (d, *J* = 8.5 Hz, 2H), 7.10 (d, *J* = 7.7 Hz, 2H), 4.63 (m, 1H), 4.54 (d, *J* = 12.4 Hz, 1H), 4.35 (dd, *J* = 8.0, 5.8 Hz, 1H), 4.02 (t, *J* = 8 Hz, 1H), 3.95 (s, 3H), 3.89 (dd, *J* = 5.8, 5.2 Hz, 1H), 3.82 (s, 3H). Mass (m/z): 221; 471; 475; 477. *Anal*. Calcd for $C_{16}H_{17}Cl_3N_4O_2STe$: C, 34.11; H, 3.04; N, 9.95; S, 5.69. Found: C, 35.78; H, 3.12; N, 10.36; S, 5.92.

7-[Dichloro(4-methoxyphenyl)tellouromethyl]-1,7-dimethyl-6,7-dihydropyrazolo[3,4-d][1,3]thiazolo[3,2-a]pyrimidin-4(1H)-one hydrochloride (4). Yield 75%; white powder (acetic acid); mp. 187–188°C. ^{1}H NMR (400 MHz, DMSO-d_6): δ (ppm) 8.46 (s, 1H), 8.04 (d, *J* = 8.8 Hz, 2H), 7.11 (d, *J* = 8.8 Hz, 2H), 4.80 (d, *J* = 12.6 Hz, 1H), 4.35 (d, *J* = 12.6 Hz, 1H), 4.29 (d, *J* = 11.8 Hz, 1H), 4.19 (d, *J* = 11.8 Hz, 1H), 3.95 (s, 3H), 3.81 (s, 3H), 1.90 (s, 3H). ^{13}C NMR: δ (ppm) 161.7, 160.6, 159.3, 157.1, 135.8, 130.3, 125.6, 115.4, 104.6, 58.8, 58.1, 56.0, 53.6, 39.6, 28.8. *Anal*. Calcd for $C_{17}H_{19}Cl_3N_4O_2STe$: C, 35.36; H, 3.32; N, 9.70; S, 5.55. Found: C, 37.13; H, 3.37; N, 10.15; S, 5.73.

Preparation of 7-[(4-methoxyphenyl)tellouromethyl]-1,7-dimethyl-6,7-dihydropyrazolo[3,4-d][1,3]thiazolo[3,2-a]pyrimidin-4(1H)-one (5). To 3 mmol of hydrochloride **4**, dissolved in 20 mL of DMSO, 9 mmol of water solution of sodium sulfite was dropped. Mixture was stirring at room temperature for 2 h. Target product was filtered and washed by water. Yield 71%; white powder (acetic acid); mp. 165–167°C. ^{1}H NMR (400 MHz, DMSO-d_6): δ (ppm) 8.44 (s, 1H), 7.69 (d, *J* = 8.2 Hz, 2H), 6.82 (d, *J* = 8.1 Hz, 2H), 4.33 (d, *J* = 12.2 Hz, 1H), 4.20 (d, *J* = 12.6 Hz, 1H), 3.95 (s, 3H), 3.74 (s, 3H), 3.48 (s, 2H), 1.60 (s, 3H). *Anal*. Calcd for $C_{17}H_{18}N_4O_2STe$: C, 43.44; H, 3.86, N. 11.92; S, 6.82. Found: C, 43.34; H, 3.81; N, 11.61; S, 7.03.

General procedure for the preparation of 3-alkenyl-2,3-dihydroquinazoline-4(1*H*)-ones. The 0.05 mol alkenyl isothiocyanate was added to 0.05 mol of anthranilic acid

in 20 mL of triethylamine. The reaction mixture was refluxed for 2 h. Precipitate was filtered and washed by methanol. The product was crystalized from ethanol.

3-Prop-2-en-1-yl-2-thioxo-2,3-dihydroquinazolin-4(1H)-one (6). Yield 61%; white powder (ethanol); mp. 203–205°C; mp. 201°C [18]; IR (KBr): (v/см$^{-1}$) = 1648 (C═O); ^{1}H NMR (400 MHz, DMSO-d_6): δ (ppm) 12.94 (s, 1H), 7.92 (d, J = 8.0 Hz, 1H), 7.72 (t, J = 7.8 Hz, 1H), 7.39 (d, J = 8.4 Hz, 1H), 7.31 (t, J = 7.6 Hz, 1H), 5.90 (m, 1H), 5.15 (dd, J = 5.2, 1.2 Hz, 1H), 5.12 (d, J = 1.6, Hz 1H), 5.03 (d, J = 3.6 Hz, 2H). ^{13}C NMR: δ (ppm) 175.51, 159.43, 139.54, 135.91, 132.24, 127.72, 124.92, 117.58, 116.08, 115.86, 48.07. *Anal*. Calcd for $C_{11}H_{10}N_2OS$: C, 60.53; H, 4.62; N, 12.83; S, 14.69. Found: C, 60.43; H, 4.55; N, 12.70; S, 14.60.

3-(2-Methylprop-2-en-1-yl)-2-thioxo-2,3-dihydroquinazolin-4(1H)-one (7). Yield 61%; white powder (ethanol); mp. 212–214°C. IR (KBr): (v/см$^{-1}$) = 1655 (C═O); ^{1}H NMR (400 MHz, DMSO-d_6) δ (ppm) 12.93 (s, 1H), 7.95 (d, J = 7.2 Hz, 1H), 7.75 (t, J = 7.6 Hz, 1H), 7.37 (d, J = 8.4 Hz, 1H), 7.34 (t, J = 7.6 Hz, 1H), 4.92 (s, 2H), 4.72 (s, 1H), 4.46 (s, 1H), 1.73 (s, 3H). ^{13}C NMR: δ (ppm) 175.82, 159.41, 139.53, 139.26, 135.83, 127.75, 124.81, 116.07, 115.70, 109.40, 50.54, 20.88. *Anal*. Calcd for $C_{12}H_{12}N_2OS$: C, 62.05; H, 5.21; N, 12.06; S, 13.80. Found: C, 61.95; H, 5.12; N, 11.93; S, 13.74.

General procedure for the preparation of 7-(*p*-methoxyphenyl)dichlorotelluromethyl-2,3-dihydro-5*H*-[1,3]thiazolo[2,3-*b*]quinazolin-5-ones hydrochlorides. The 1 mmol of 3-alkenyl-2-thioxo-2,3-dihydroquinazolin-4(1*H*)-one **6** or **7**, dissolved in 20 mL of acetic acid, was added to 1 mmol of *p*-alkoxyphenyltellutium trichloride in 20 mL of acetic acid. The reaction mixture was stirring at room temperature for 8 h. Target product was filtered and recrystallized from acetic acid.

3-[Dichloro(4-methoxyphenyl)telluromethyl]-2,3-dihydro-5H-[1,3]thiazolo[2,3-b]quinazolin-5-one hydrochloride (8). Yield 55%; white powder (acetic acid); mp. 143–145°C; IR (KBr): (v/см$^{-1}$) = 1690 (C═O); ^{1}H NMR (400 MHz, DMSO-d_6) δ (ppm) 8.07 (d, J = 8.0 Hz, 1H), 8.05 (d, J = 7.2 Hz, 2H), 7.78 (t, J = 7.6 Hz, 1H), 7.55 (d, J = 8.4 Hz, 1H), 7.46 (t, J = 8 Hz, 1H), 7.55 (d, J = 8.4 Hz, 1H), 7.08 (d, J = 7.2 Hz, 1H), 4.76 (m, 1H), 4.67 (d, J = 13.2 Hz, 1H), 4.44 (dd, J = 12.8, 7.2 Hz, 1H), 4.10 (t, J = 10.4, Hz, 1H), 3.94 (dd, J = 12.4, 5.6 Hz, 2H), 3.81 (s, 3H). ^{13}C NMR δ (ppm) 161.66, 160.35, 159.69, 147.44, 135.89, 135.25, 126.62, 125.79, 125.03, 123.10, 119.38, 115.36, 55.97, 54.41, 49.83, 41.52. *Anal*. Calcd for $C_{18}H_{17}Cl_3N_2O_2STe$: C, 38.65; H, 3.06; N, 5.01; S, 5.73. Found: C, 40.63; H, 3.15; N, 5.24; S, 5.91.

3-[Dichloro(4-ethoxyphenyl)telluromethyl]-2,3-dihydro-5H-[1,3]thiazolo[2,3-b]quinazolin-5-one hydrochloride (10). Yield 52%; white powder (acetic acid); mp. 148–149°C. IR (KBr): (v/см$^{-1}$) = 1691 (C═O); ^{1}H NMR (400 MHz, DMSO-d_6) δ (ppm) 8.08 (d, J = 8.0 Hz, 1H), 8.04 (d, J = 7.2 Hz, 2H), 7.78 (t, J = 7.6 Hz, 1H), 7.55 (d, J = 8.0 Hz, 1H), 7.46 (t, J = 8 Hz, 1H), 7.55 (d, J = 8.4 Hz, 1H), 7.08 (d, J = 7.2 Hz, 1H), 4.76 (m, 1H), 4.67 (d, J = 12.8 Hz, 1H), 4.44 (dd, J = 12.8, 7.2 Hz, 1H), 4.09 (m, 3H), 3.94 (dd, J = 12.0, 6 Hz, 2H), 1.33 (t, J = 6.4 Hz 3H). ^{13}C NMR δ (ppm) 160.98, 160.10, 159.77, 147.80, 135.89, 135.73, 126.57, 125.24, 123.10, 119.42, 115.75, 63.94, 54.37, 49.86, 41.38, 14.97. *Anal*. Calcd for $C_{19}H_{19}Cl_3N_2O_2STe$: C, 39.80; H, 3.34; N, 4.89; S, 5.59. Found: C, 41.82; H, 3.44; N, 5.07; S, 5.80.

3-[Dichloro(4-methoxyphenyl)telluromethyl]-3-methyl-2,3-dihydro-5H-[1,3]thiazolo[2,3-b]quinazolin-5-one hydrochloride (9). Yield 56%; white powder (acetic acid); mp. 177–178°C. IR (KBr): (v/см$^{-1}$) = 1692 (C═O); ^{1}H NMR (400 MHz, DMSO-d_6) δ (ppm) 8.09 (d, J = 8.0 Hz, 1H), 8.06 (d, J = 7.2 Hz, 2H), 7.77 (t, J = 6.8 Hz, 1H), 7.54 (d, J = 8.4 Hz, 1H), 7.44 (t, J = 7.2 Hz, 1H), 7.10 (d, J = 7.2 Hz, 2H), 4.94 (d, J = 12.4 Hz, 1H), 4.45 (d, J = 12.8, 1H), 4.33 (d, J = 11.8, 1H), 4.24 (d, J = 11.8 Hz, 1H), 3.80 (s, 3H), 1.93 (s, 3H). ^{13}C NMR δ (ppm) 161.69, 160.79, 159.87, 148.58, 136.01, 135.75, 126.80, 126.10, 125.62, 119.65, 115.33, 59.44, 58.02, 56.03, 54.20, 28.89. *Anal*. Calcd for $C_{19}H_{19}Cl_3N_2O_2STe$: C, 39.80; H, 3.34; N, 4.89; S, 5.59. Found: C, 41.83; H, 3.45; N, 5.08; S, 5.78.

3-[Dichloro(4-ethoxyphenyl)telluromethyl]-3-methyl-2,3-dihydro-5H-[1,3]thiazolo[2,3-b]quinazolin-5-one hydrochloride (11). Yield 53%; white powder (acetic acid); mp. 176–178°C. IR (KBr): (v/см$^{-1}$) = 1694 (C═O); ^{1}H NMR (400 MHz, DMSO-d_6) δ (ppm) 8.08 (d, J = 8.0 Hz, 1H), 8.05 (d, J = 7.2 Hz, 2H),7.76 (t, J = 6.8 Hz, 1H), 7.54 (d, J = 8.4 Hz, 1H), 7.45 (t, J = 7.2 Hz, 1H), 7.10 (d, J = 7.2 Hz, 2H), 4.93 (d, J = 13.2 Hz, 1H), 4.45 (d, J = 12.4, 1H), 4.32 (d, J = 11.6, 1H), 4.24 (d, J = 11.2 Hz, 1H), 4.08 (m, 3H), 1.92 (s, 3H), 1.33 (t, J = 6.4 Hz, 3H). ^{13}C NMR δ (ppm) 160.98, 160.07, 159.88, 148.68, 135.78, 135.08, 126.64, 126.17, 125.29, 119.71, 115.72, 63.96, 58.07, 56.31, 53.99, 28.89, 14.97. *Anal*. Calcd for $C_{20}H_{21}Cl_3N_2O_2STe$: C, 40.89; H, 3.60; N, 4.77; S, 5.46. Found: C, 42.85; H, 3.70; N, 4.96; S, 5.68.

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