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Heterocyclization of 5,6-disubstituted 3-alkenyl-2-thioxothieno[2,3-*d*]pyrimidin-4-one with *p*-alkoxyphenyltellurium trichloride

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Abstract: Electrophilic heterocyclization of 5,6-disubstituted 3-alkenyl-2-thioxothieno[2,3-*d*]pyrimidin-4-ones by treatment with *p*-alkoxyphenyltellurium trichlorides leads to annulation of the thiazoline moiety with the formation of 6,7-disubstituted-2-[dichloro-(*p*-alkoxyphenyl)telluromethyl]-2,3-dihydro-5*H*-[1,3]thiazolo[3,2-*a*]thieno[2,3-*d*]pyrimidin-5-one hydrochlorides.

Keywords: 3-alkenyl-2-thioxothieno[2,3-*d*]pyrimidine-4-one; [1,3]thiazolo[3,2-*a*]thieno[2,3-*d*]pyrimidin-5-one; electrophilic cyclization; *p*-alkoxyphenyltellurium trichloride; regioselectivity.

Introduction

Organotellurium compounds show anti-cancer [1–3], antioxidant [3, 4] and anti-bacterial activity [5] and are used in neuropathy [6–11]. In this study we analyzed a new route of introduction of tellurium into a heterocyclic system. Electrophilic heterocyclization of unsaturated substrates under the action of tellurium tetrahalides is described for obtaining tellurium-containing fused heterocyclic systems [12–14]. Using for the same purpose aryltellurium trihalides is barely mentioned in the literature. Indeed, there are only few data about using aryltellurium trihalides in the reaction with unsaturated alcohols, phenols and acids [15–18]. We choose the *N*-alkenyl derivatives of 5,6-disubstituted-2-thioxothieno[2,3-*d*]pyrimidin-4-one as the objects for study electrofilic heterocyclization by *p*-alkoxyphenyltellurium trihalides.

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Results and discussion

5,6-Disubstituted-2-thioxothieno[2,3-*d*]pyrimidin-4-ones **1–6** were synthesized as shown in Scheme 1 and used subsequently as substrates for electrophilic heterocyclization in the presence of *p*-alkoxyphenyltellurium trihalides (Scheme 2). The model compounds **1–6** contain a few nucleophilic centers in the molecule, which provides an opportunity to study regioselectivity of the electrophilic cyclization. Although several cyclization pathways could be suggested (not shown), the reaction takes a single route indicated in Scheme 2.

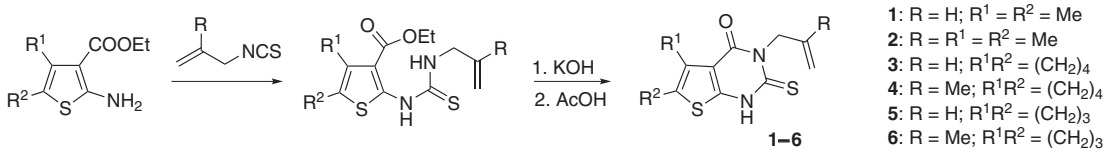
The heterocyclization was successfully conducted in acetic acid, chloroform and acetonitrile at different temperatures. It was found that under the optimal conditions the electrophilic heterocyclization is conducted in acetic acid at room temperature. The tellurium-containing compounds **7–15** were regioselectively obtained as the only products. It can be suggested that the addition of aryltellurium trichloride to a double bond of **1–6** takes place to generate the intermediate product A which is a direct precursor to the final product **7–15**. In one case the hydrochloride **14** was transformed into a free base **16** (Scheme 2) by treatment with sodium sulfite. This is a remarkable result because the expected reduction of tellurium was not observed. All products **1–16** were fully characterized by spectral methods and elemental analysis.

Conclusion

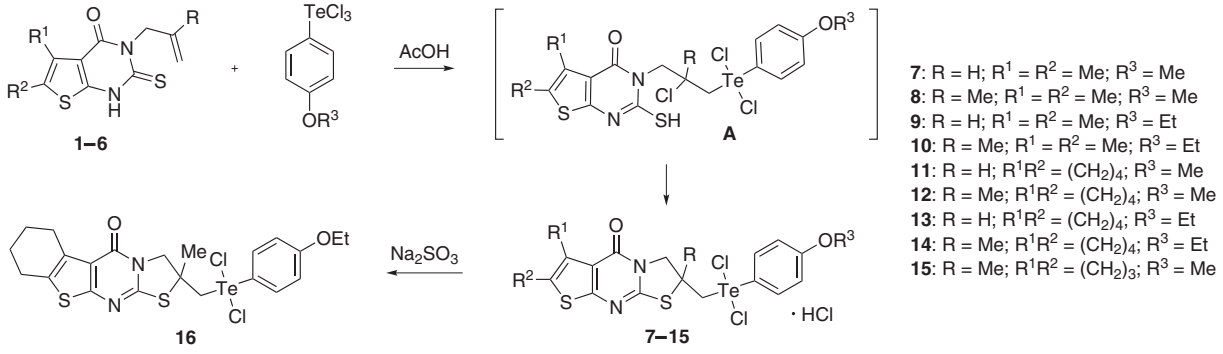
Electrofilic heterocyclization of *N*-alkenyl-substituted 2-thioxotieno[2,3-*d*]pyrimidin-4-ones **1–6** in the presence of an aryltellurium trichloride was investigated. The reaction is highly regioselective.

Experimental

^{1}H NMR (400 MHz) and ^{13}C NMR (100 MHz) spectra were recorded in DMSO-d_6 on a Varian Mercury-400 instrument. Melting points



Scheme 1 Synthesis of 5,6-disubstituted 2-thioxothieno[2,3-*d*]pyrimidin-4-ones **1–6**.



Scheme 2 Synthesis of 6,7-disubstituted-2-(dichloro(*p*-alkoxyphenyl)tellouromethyl)-2,3-dihydro-5*H*-[1,3]thiazolo[3,2-*a*]thieno[2,3-*d*] pyrimidin-5-ones hydrochlorides.

were determined on a Stuart SMP30 instrument. Elemental analyses were performed on an Elementar Vario analyzer. All reagents were obtained from commercial suppliers and used without further purification. Anhydrous solvents were prepared according to the standard methods. The *p*-alkoxyphenyltellurium trihalides were synthesized according to the literature procedure [19].

Synthesis of 5,6-disubstituted 3-alkenyl-2-thioxothieno[2,3-*d*]pyrimidin-4-ones 1–6

A solution of an ethyl 2-aminothiophene-3-carboxylate (0.05 mol) and allyl or methallyl isothiocyanate [20] (0.05 mol) in ethanol (40 mL) was heated under reflux for 8 h and then treated with an aqueous solution (5 mL) of potassium hydroxide (0.1 mol). The mixture was heated for an additional 2 h, cooled and diluted with aqueous acetic acid. The resultant precipitate of **1–6** was crystallized from ethanol.

5,6-Dimethyl-3-(prop-2-en-1-yl)-2-thioxo-2,3-dihydrothieno[2,3-*d*]pyrimidin-4(1*H*)-one (1) Yield 89%.; mp 211–212°C; lit. mp 191–192°C [21] and 209–212°C [22].

5,6-Dimethyl-3-(2-methylprop-2-en-1-yl)-2-thioxo-2,3-dihydrothieno[2,3-*d*]pyrimidin-4(1*H*)-one (2) Yield 89%; mp 189–190°C; ^{1}H NMR: δ 13.53 (s, 1H), 4.85 (s, 2H), 4.71 (s, 1H), 4.42 (s, 1H), 2.25 (s, 6H), 1.72 (s, 3H); ^{13}C NMR: δ 174.1, 156.8, 148.6, 139.3, 129.3, 125.8, 116.6, 109.2, 50.3, 21.0, 13.0, 12.5. Anal. Calcd for $C_{12}H_{14}N_2OS_2$: C, 54.11; H, 5.30; N. 10.52. Found: C, 53.87; H, 5.28; N, 10.44.

3-(Prop-2-en-1-yl)-2-thioxo-2,3,5,6,7,8-hexahydro[1]benzothieno[2,3-*d*]pyrimidin-4(1*H*)-one (3) Yield 85%.; mp 218–219°C; lit. mp 205–208°C [22].

3-(2-Methylprop-2-en-1-yl)-2-thioxo-2,3,5,6,7,8-hexahydro[1]benzothieno[2,3-*d*]pyrimidin-4(1*H*)-one (4) Yield 85%.; mp 202–204°C; ^{1}H NMR: δ 13.56 (s, 1H), 4.85 (s, 2H), 4.72 (s, 1H), 4.42 (s, 1H), 2.75 (m, 2H), 2.64 (m, 2H), 1.77 (m, 4H), 1.72 (s, 3H); ^{13}C NMR: δ 174.3, 156.7, 149.4, 139.4, 131.5, 129.1, 116.0, 109.3, 50.3, 25.4, 24.5, 23.0, 22.1, 21.1. Anal. Calcd for $C_{14}H_{16}N_2OS_2$: C, 57.50; H, 5.52; N. 9.58. Found: C, 57.11; H, 5.39; N, 9.41.

3-(Prop-2-en-1-yl)-2-thioxo-1,2,3,5,6,7-hexahydro-4*H*-cyclopenta[4,5]thieno[2,3-*d*]pyrimidin-4-one (5) Yield 85%; mp 223–224°C; ^{1}H NMR: δ 13.61 (s, 1H), 5.88 (m, 1H), 5.14 (s, 1H), 5.10 (d, J = 6.5 Hz 1H), 4.96 (d, J = 5.1 Hz, 2H), 2.82 (t, J = 7.2 Hz, 4H), 2.41–2.29 (m, 2H). Anal. Calcd for $C_{12}H_{12}N_2OS_2$: C, 54.52; H, 4.85; N. 10.60. Found: C, 53.98; H, 4.69; N, 10.48.

3-(2-Methyl-prop-2-en-1-yl)-2-thioxo-1,2,3,5,6,7-hexahydro-4*H*-cyclopenta[4,5]thieno[2,3-*d*]pyrimidin-4-one (6) Yield 85%; mp 209–210°C; ^{1}H NMR: δ 13.47 (s, 1H), 4.89 (s, 2H), 4.74 (s, 1H), 4.47 (s, 1H), 2.82 (t, J 6.7 Hz, 4H), 2.41–2.27 (m, 2H), 1.73 (s, 3H); ^{13}C NMR: δ 174.3, 156.4, 153.8, 140.5, 139.3, 134.2, 113.2, 109.3, 50.3, 29.0, 28.7, 28.2, 21.0. Anal. Calcd for $C_{13}H_{14}N_2OS_2$: C, 56.09; H, 5.07; N. 10.06. Found: C, 56.11; H, 5.12; N, 10.11.

Synthesis of 6,7-disubstituted-2-(dichloro(*p*-alkoxyphenyl)-tellouromethyl)-2,3-dihydro-5*H*-[1,3]thiazolo[3,2-*a*] thieno[2,3-*d*]pyrimidin-5-ones hydrochlorides 7–15

A mixture of 5,6-disubstituted 2-thioxothieno[2,3-*d*]pyrimidin-4-one **1–6** (1 mmol) and *p*-alkoxyphenyltellurium trichloride (1 mmol) in acetic acid (40 mL) was stirred at room temperature for 8 h. The resultant precipitate was filtered and crystallized from acetic acid.

6,7-Dimethyl-2-[dichloro(4-methoxyphenyl)tellurylmethyl]-2,3-dihydro-5*H*-[1,3]thiazolo[3,2-*a*]thieno[2,3-*d*]pyrimidin-5-one hydrochloride (7) Yield 73%; mp 201–202°C; ^{1}H NMR: δ 8.05 (d, J = 8.9 Hz, 2H), 7.09 (d, J = 8.9 Hz, 2H), 4.69 (m, 1H), 4.60 (dd, J = 13.0 and 2.6 Hz, 1H), 4.38 (dd, J = 12.9 and 7.2 Hz, 1H), 4.04 (dd, J = 11.8 and 8.7 Hz, 1H), 3.91 (dd, J = 13.0 and 2.6 Hz, 1 H), 3.82 (s, 3H), 2.32 (s, 6H); ^{13}C NMR: δ 162.2, 158.8, 157.1, 135.9, 128.8, 128.7, 125.6, 119.7, 115.4, 56.0, 54.1, 49.9, 13.2. Anal. Calcd for $C_{18}H_{19}Cl_3N_2O_2S_2Te$: C, 36.31; H, 3.55; Cl, 17.86; N. 4.70. Found: C, 36.11; H, 3.39; Cl, 17.52; N, 4.58.

2,6,7-Trimethyl-2-(dichloro(4-methoxyphenyl)tellurylmethyl)-2,3-dihydro-5*H*-[1,3]thiazolo[3,2-*a*]thieno[2,3-*d*]pyrimidin-5-one hydrochloride (8) Yield 73%; mp 206–207°C; ^{1}H NMR: δ 8.05 (d, J = 8.9 Hz, 2H), 7.11 (d, J = 9.0 Hz, 2H), 4.85 (d, J = 12.9 Hz, 1H), 4.41 (d, J 12.9 Hz, 1H), 4.31 (d, J = 11.9 Hz, 1H), 4.22(d, J 11.9 Hz, 1H), 3.81 (s, 3H), 2.34 (s, 6H), 1.91 (s, 3H); ^{13}C NMR: δ 162.0, 161.7, 158.6, 157.1, 135.8, 129.0, 128.7, 125.6, 119.9, 115.4, 59.3, 57.8, 56.0, 54.6, 29.0, 13.2. Anal. Calcd for $C_{19}H_{21}Cl_3N_2O_2S_2Te$: C, 37.44; H, 3.80; Cl, 17.45; N. 4.60. Found: C, 37.19; H, 3.69; Cl, 17.28; N, 4.49.

6,7-Dimethyl-2-(dichloro(4-ethoxyphenyl)tellurylmethyl)-2,3-dihydro-5*H*-[1,3]thiazolo[3,2-*a*]thieno[2,3-*d*]pyrimidin-5-one hydrochloride (9) Yield 73%; mp 217–218°C; ^{1}H NMR: δ 8.03 (d, J = 8.8 Hz, 2H), 7.08 (d, J = 8.8 Hz, 2H), 4.68 (m, 1H), 4.60 (d, J = 12.8 Hz, 1H), 4.38 (dd, J = 12.8, 7.2 Hz, 1H), 4.09 (dt, J = 10.5 and 5.2 Hz, 2H), 4.02 (d, J = 8.6 Hz, 1H), 3.90 (dd, J = 11.8 and 5.9 Hz, 1H), 2.33 (s, 6H), 1.34 (t, J = 6.9 Hz, 3H); ^{13}C NMR: δ 162.2, 161.0, 158.9, 157.2, 135.9, 128.8, 128.5, 125.3, 119.7, 115.8, 64.0, 54.1, 49.8, 15.0, 13.2. Anal. Calcd for $C_{19}H_{21}Cl_3N_2O_2S_2Te$: C, 37.44; H, 3.80; Cl, 17.45; N. 4.60. Found: C, 37.26; H, 3.62; Cl, 17.36; N, 4.45.

2,6,7-Trimethyl-2-(dichloro(4-ethoxyphenyl)tellurylmethyl)-2,3-dihydro-5*H*-[1,3]thiazolo[3,2-*a*]thieno[2,3-*d*]pyrimidin-5-one hydrochloride (10) Yield 73%; mp 201–202°C; ^{1}H NMR: δ 8.03 (d, J = 8.8 Hz, 2H), 7.10 (d, J = 8.8 Hz, 2H), 4.85 (d, J = 12.9 Hz, 1H), 4.41 (d, J = 12.9 Hz, 1H), 4.30 (d, J = 11.9 Hz, 1H), 4.22 (d, J = 11.6 Hz, 1H), 4.09 (q, J = 7.0 Hz, 1H), 2.34 (s, 6H), 1.91 (s, 3H), 1.34 (t, J = 7.0 Hz, 3H); ^{13}C NMR: δ 162.0, 161.0, 158.6, 157.1, 135.8, 129.0, 128.7, 125.3, 119.9, 115.7, 64.0, 59.3, 57.8, 54.6, 29.0, 15.0, 13.3, 13.1. Anal. Calcd for $C_{20}H_{23}Cl_3N_2O_2S_2Te$: C, 38.53; H, 4.04; Cl, 17.06; N. 4.49. Found: C, 38.35; H, 3.97; Cl, 16.96; N, 4.40.

2-(Dichloro(4-methoxyphenyl)tellurylmethyl)-2,3,6,7,8,9-hexahydro-5*H*-benzo[4,5]thieno[2,3-*d*][1,3]thiazolo[3,2-*a*]pyrimidin-5-one hydrochloride (11) Yield 74%.; mp 213–214°C; ^{1}H NMR: δ 8.05 (d, J = 8.9 Hz, 2H), 7.10 (d, J = 8.9 Hz, 2H), 4.69 (m, 1H), 4.61 (dd, J = 13.1 and 2.6 Hz, 1H), 4.39 (dd, J = 12.9 and 7.1, Hz, 1H), 4.04 (dd, J = 11.8 and 8.7 Hz, 1H), 3.91 (dd, J = 11.8 and 6.0 Hz, 1H), 2.83 (m, 2H), 2.71 (m, 2H), 1.77 (m, 4H). Anal. Calcd for $C_{20}H_{21}Cl_3N_2O_2S_2Te$: C, 38.65; H, 3.73; Cl, 17.11; N. 4.51. Found: C, 38.11; H, 3.59; Cl, 17.02; N, 4.41.

2-Methyl-2-(dichloro(4-methoxyphenyl)tellurylmethyl)-2,3,6,7,8,9-hexahydro-5*H*-benzo[4,5]thieno[2,3-*d*][1,3]thiazolo[3,2-*a*]pyrimidin-5-one hydrochloride (12) Yield 74%; mp 228–230°C; ^{1}H NMR: δ 8.07 (d, J = 8.8 Hz, 2H), 7.13 (d, J = 8.8 Hz, 2H), 4.86 (d, J = 13.0 Hz, 1H), 4.42 (d, J = 12.9 Hz, 1H), 4.33 (d, J = 11.8 Hz, 1H), 4.24 (d, J = 11.8 Hz, 1H), 3.84 (s, 3H), 2.86 (m, 2H), 2.73 (m, 2H), 1.93 (s, 3H), 1.79 (m, 4H); ^{13}C NMR: δ 162.3, 161.3, 158.3, 156.4, 135.4, 130.6, 114.9, 58.8, 57.3, 55.6, 54.3, 28.5, 25.3, 24.5, 22.5, 21.8. Anal. Calcd for $C_{21}H_{23}Cl_3N_2O_2S_2Te$: C, 39.69; H, 3.96; Cl, 16.74; N. 4.41. Found: C, 39.48; H, 3.79; Cl, 16.66; N, 4.35.

2-(Dichloro(4-ethoxyphenyl)tellurylmethyl)-2,3,6,7,8,9-hexahydro-5*H*-benzo[4,5]thieno[2,3-*d*][1,3]thiazolo[3,2-*a*]pyrimidin-5-one hydrochloride (13) Yield 73%; mp 216–218°C; ^{1}H NMR: δ 8.04 (d, J = 8.3 Hz, 2H), 7.08 (d, J = 8.3 Hz, 2H), 4.71 (m, 1H), 4.58 (d, J = 13.0 Hz, 1H), 4.40 (dd, J = 12.0 and 7.7 Hz, 1H), 4.16–3.98 (m, 3H), 3.92 (dd, J = 10.5 and 5.9 Hz, 1H), 2.83 (m, 2H), 2.70 (m, 2H), 1.77 (m, 4H), 1.35 (t, J = 6.6 Hz, 3H); ^{13}C NMR: δ 163.0, 161.0, 159.0, 156.9, 131.4, 130.9, 125.4, 118.9, 115.8, 64.0, 54.1, 49.8, 25.7, 24.9, 23.0, 22.2, 15.0. Anal. Calcd for $C_{21}H_{23}Cl_3N_2O_2S_2Te$: C, 39.69; H, 3.96; Cl, 16.74; N. 4.41. Found: C, 39.33; H, 3.77; Cl, 16.57; N, 4.29.

2-Methyl-2-(dichloro(4-ethoxyphenyl)tellurylmethyl)-2,3,6,7,8,9-hexahydro-5*H*-benzo[4,5]thieno[2,3-*d*][1,3]thiazolo[3,2-*a*]pyrimidin-5-one hydrochloride (14) Yield 73%; mp 208–209°C; ^{1}H NMR: δ 8.05 (d, J = 7.4 Hz, 2H), 7.10 (d, J = 7.8 Hz, 2H), 4.86 (d, J = 12.9 Hz, 1H), 4.41 (d, J = 13.0 Hz, 1H), 4.32 (d, J = 11.5 Hz, 1H), 4.24 (d, J = 11.6 Hz, 1H), 4.12 (q, J = 7.0 Hz, 2H), 2.85(m, 2H), 2.72 (m, 2H), 1.93 (s, 3H), 1.78 (m, 4H), 1.35 (t, J = 7.0 Hz, 3H); ^{13}C NMR: δ 162.9, 161.1, 158.8, 156.9, 135.9, 115.8, 64.1, 59.3, 57.9, 54.8, 29.0, 25.8, 25.0, 23.1, 22.3, 15.1. Anal. Calcd for $C_{22}H_{25}Cl_3N_2O_2S_2Te$: C, 40.68; H, 4.19; Cl, 16.37; N. 4.31. Found: C, 40.11; H, 4.05; Cl, 16.15; N, 4.26.

2-Methyl-2-(dichloro(4-methoxyphenyl)tellurylmethyl)-2,3,7,8-tetrahydro-5*H*,6*H*-cyclopenta[4,5]thieno[2,3-*d*][1,3]thiazolo[3,2-*a*]pyrimidin-5-one hydrochloride (15) Yield 70%; mp 180–181°C; ^{1}H NMR: δ 8.05 (d, J = 8.9 Hz, 2H), 7.11 (d, J = 8.9 Hz, 2H), 4.87 (d, J = 12.9 Hz, 1H), 4.42 (d, J = 12.9 Hz, 1H), 4.30 (d, J = 11.9, Hz, 1H), 4.22 (d, J = 11.9 Hz, 1H), 3.81(s, 3H), 2.88 (t, J = 6.9 Hz, 4H), 2.41–2.30 (m, 2H), 1.91 (s, 3H); ^{13}C NMR: δ 167.9, 158.4, 156.5, 139.7, 136.8, 135.8, 125.6, 116.7, 115.4, 59.2, 57.8, 56.0, 54.7, 29.5, 29.0, 28.8, 28.6, 27.9. Anal. Calcd for $C_{21}H_{23}Cl_3N_2O_2S_2Te$: C, 39.69; H, 3.96; Cl, 16.74; N. 4.41. Found: C, 39.48; H, 3.88; Cl, 16.64; N, 4.32.

Synthesis of 2-methyl-2-(dichloro(4-ethoxyphenyl)tellurylmethyl)-2,3,6,7,8,9-hexahydro-5*H*-benzo[4,5]thieno[2,3-*d*][1,3]thiazolo[3,2-*a*]pyrimidin-5-one (16) A solution of hydrochloride **14** (0.3 mmol) in DMSO (10 mL) was treated dropwise with a solution of sodium sulfite (1.2 mmol) in water (5 mL). The resultant white precipitate was filtered, washed with water and crystallized from acetic acid**;** yield 70%; mp 95–96°C; ^{1}H NMR: δ 7,67 (d, J = 8.5 Hz, 3H), 6,80 (d, J = 8.5 Hz, 3H), 4.38 (d, J = 12.6 Hz, 2H), 4.00 (q, J = 7 Hz, 3H), 3.49 (s, 3H), 3.32 (s, 9H), 2.80 (s, 4H), 2.69 (s, 4H), 2.54 (s, 2H), 1.80–1.66 (m, 8H), 1.62 (s, 5H), 1.31 (dd, J = 14.8 and 7.8 Hz, 6H); ^{13}C NMR: δ 159.3, 157.0, 140.8, 131.4, 131.1, 116.5, 63.6, 59.1, 57.4, 28.1, 25.8, 25.0, 23.1, 22.3, 22.5, 15.2. Anal. Calcd for $C_{22}H_{24}Cl_2N_2O_2S_2Te$: C, 43.10; H, 4.2;, Cl, 11.57; N. 4.57. Found: C, 43.15; H, 4.39; Cl, 11.71; N, 4.61.

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