

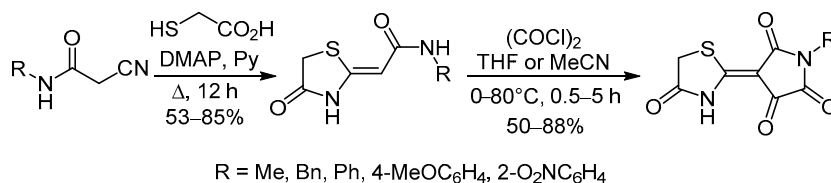
Synthesis of 4-(4-oxo-1,3-thiazolidin-2-ylidene)-pyrrolidine-2,3,5-triones

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It was shown that 2-(1,3-thiazolidin-2-ylidene)acetamides can be used as 2-enamides in cyclocondensation reactions with oxalyl chloride, leading to the formation of new heterocyclic assemblies – 4-(4-oxo-1,3-thiazolidin-2-ylidene)pyrrolidine-2,3,5-triones in 50–88% yields.

Keywords: oxalyl chloride, pyrrolidine-2,3,5-trione, thiazolidin-4-one, cyclocondensation, exocyclic double bond, heterocyclic assemblies.

Molecular assemblies derived from 1,3-thiazolidin-4-one ring connected by exocyclic C=C double bond to other five-membered heterocycles attract the attention of researchers due to their biological effects (such as antimalarial¹ and anticancer² activity), as well as by their electrochemical^{3,4} and photophysical properties.^{5,6} Among compounds of this type the most often reported in the literature are (1,3-thiazolidinylidene)-1,3-thiazolidines⁷ and (2-oxoindolin-3-ylidene)-1,3-thiazolidin-4-ones.⁸ Some examples of molecular assemblies have been described where the 1,3-thiazolidine ring is linked through a C=C bond with pyrazole,⁹ oxazole,¹⁰ and other heterocycles. At the same time, molecular assemblies of 1,3-thiazolidine and pyrrolidine-2,3,5-one have not been described in the literature at all.

In this work, we propose a convenient method for the synthesis of new heterocyclic assemblies – 4-(4-oxo-1,3-thiazolidin-2-ylidene)pyrrolidine-2,3,5-triones by cyclocondensation of 2-(1,3-thiazolidin-2-ylidene)acetamides with oxalyl chloride.

The starting 2-(1,3-thiazolidin-2-ylidene)acetamides **1a–e** were synthesized in 53–85% yields by reacting the respective nitriles **2a–e** with mercaptoacetic acid (Scheme 1, Table 1) upon heating for 12 h in pyridine in the presence of *p*-dimethylaminopyridine (DMAP). Despite the fact that this method for the synthesis of 1,3-thiazolidin-

4-one derivatives is widely reported in the literature,^{11–15} 1,3-thiazolidines **1a,b,d,e** have not been described previously. Synthetic procedure optimization for the preparation of previously described¹⁵ compound **1c** allowed to increase its yield from 60%¹⁵ to 85%.

Based on the chemical shift of the 2'-CH methine proton signal (5.55–5.82 ppm) in ¹H NMR spectra of compounds **1a–e** and taking into account the literature data,¹⁶ we assigned (*Z*)-configuration to the exocyclic C=C double bond.

2-(1,3-Thiazolidin-2-ylidene)acetamides **1** have been previously studied in alkylation,¹⁷ bromination,^{18,19} thionation,²⁰ cyclocondensation,²¹ and Claisen condensation²² reactions. However, their interaction with such bis-electrophile as oxalyl chloride has not been described in the literature. At the same time, it is known that the reactions of oxalyl chloride with 2-enamides as bisnucleophiles led to the formation of pyrrolidine-2,3,5-triones.^{23–25}

As a result of reaction between 1,3-thiazolidines **1a–d** and oxalyl chloride at 0–5°C in anhydrous THF, we synthesized 4-(4-oxo-1,3-thiazolidin-2-ylidene)pyrrolidine-2,3,5-triones **3a–d** in 82–88% yields (Scheme 1, Table 1). It should be noted that, due to the low reactivity of 1,3-thiazolidine **1e** containing a *p*-nitrophenyl moiety, the reaction with oxalyl chloride proceeded in MeCN upon heating to 80°C for 0.5 h. However, product **3e** could not be isolated as individual compound and purified, as it was unstable in solution.

[†] Deceased

Scheme 1

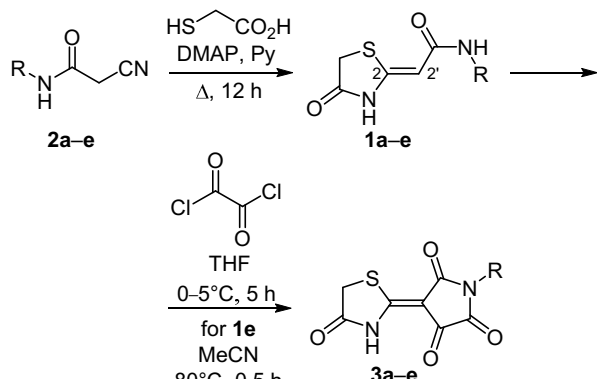


Table 1. Yields of thiazolidines 1a–e and 3a–e

R	Compound	Yield, %	Compound	Yield, %
Me	1a	53	3a	86
Bn	1b	61	3b	88
Ph	1c	85	3c	82
4-MeOC ₆ H ₄	1d	72	3d	85
4-O ₂ NC ₆ H ₄	1e	71	3e	50*

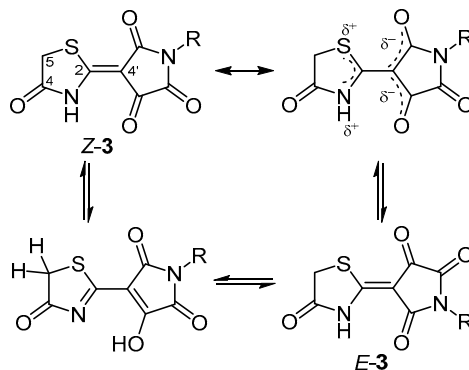
* According to ¹H NMR spectroscopy data, the content of the target compound was 85%.

All of the molecular assemblies **3a–d** showed [M]⁺ peaks of the molecular ion in their mass spectra. The characteristic fragment ion peaks were [M–CO]⁺, [M–C₂O₂H]⁺, and [M–CO–C₂O₂H]⁺. ¹H NMR spectra of compounds **3a–d** in DMSO-*d*₆ solution showed a single set of signals for all protons, except for the methylene group in the thiazolidine ring, which gave two broadened singlets in the range of 3.99–4.02 ppm.²⁶ Additional studies showed doubling of methylene, methyl, and NH group signals in ¹H NMR spectra of compound **3a** that were recorded in DMF-*d*₆ solution at –18°C. This phenomenon can be explained by hindered rotation around the exocyclic C=C bond that had a partial double bond nature, with the rotation slowing down at lower temperature. A decrease in the order of the exocyclic double bond in molecular assemblies **3a–d** was possible due to its push-pull character and ketone-enol tautomerism (Scheme 2).²⁷ The analysis of ¹H NMR spectra obtained for the molecular assembly **3a** in DMF-*d*₆ solution at the temperature range from –18 to 28°C enabled an approximate estimate of the coalescence temperature as 0°C (see the Supplementary information file).

It was also shown that ¹H NMR spectrum of compound **3a** in DMSO solution, which was acquired at 30°C, showed coalescence of methylene proton signals that could be explained by the faster rotation of rings around the exocyclic bond.

IR spectra of compounds **1a** and **3a** (R = Me) featured absorption bands at 1556 and 1574 cm^{–1}, respectively. This observation pointed to the presence of C=C double bond in their structure.²⁸ Absorption bands in the range of 1540–

Scheme 2



1585 cm^{–1} were also observed for compounds **1b–e** and **3b–d** (R = Bn, Ar), but reliable assignment of these absorptions was impossible due to the presence of benzene rings in the structure, which also produced absorption in this range.

Thus, in the current work we propose a convenient method for the synthesis of new molecular assemblies containing 1,3-thiazolidin-4-one and pyrrolidine-2,4,5-trione rings, and note that these compounds in solution phase exist in equilibrium with their isomeric forms that arise due to rotation around the exocyclic C=C bond with a partial double bond character.

Experimental

IR spectra were recorded on a Bruker ALPHA spectrometer that was equipped with a ZnSe ATR accessory. ¹H and ¹³C NMR spectra were acquired on a Bruker Avance II spectrometer (400 and 100 MHz, respectively) in DMSO-*d*₆ (an additional ¹H NMR spectrum for compound **3a** was acquired in DMF-*d*₆) at the Laboratory of Complex Investigation and Expert Evaluation of Organic Materials at the Collective Use Center of the Ural Federal University. TMS was used as internal standard. ¹H and ¹³C NMR spectra of compounds **3a–d** were acquired immediately after dissolution of samples because of their instability in solution. Mass spectra were recorded on a Shimadzu GCMS-QP2010 Plus gas chromatography-mass spectrometer (EI ionization, 70 eV). Elemental analysis was performed on a PE 2400 Series II CHNS-analyzer. Melting points were determined on a Stuart SMP3 apparatus.

Preparation of 2-(4-oxo-1,3-thiazolidin-2-ylidene)acetamides 1a–e (General method). Pyridine was added dropwise with stirring to cyanoacetamide **2a–e** (15 mmol) in a round-bottom flask until complete dissolution of the cyanoacetamide. DMAP (18 mg, 0.15 mmol) and mercaptoacetic acid (3.2 ml, 46 mmol) were added to the obtained solution. The obtained mixture was refluxed for 12 h in a flask equipped with a reflux condenser, then diluted with 0.5 M HCl solution (5 ml). The precipitate of 1,3-thiazolidinone **1** was filtered off and washed with hot MeCN (15 ml). When necessary, the product was additionally purified by refluxing a suspension of thiazolidine in MeCN, followed by hot filtration.

(2Z)-N-Methyl-2-(4-oxo-1,3-thiazolidin-2-ylidene)acetamide (1a). Yield 1.37 g (53%), white powder, mp 238–243°C (decomp.). IR spectrum, ν, cm^{–1}: 1184, 1299, 1398,

1415, 1470, 1556 (C=C), 1624 (C=O), 1699 (C=O), 3312 (NH). ^1H NMR spectrum, δ , ppm (J , Hz): 2.58 (3H, d, $J = 4.8$, CH_3); 3.61 (2H, s, CH_2); 5.55 (1H, s, CH); 7.61 (1H, br. s, NH); 11.20 (1H, s, NH). ^{13}C NMR spectrum, δ , ppm: 25.3; 31.9; 92.4; 151.4; 167.0; 174.0. Mass spectrum, m/z (I_{rel} , %): 172 $[\text{M}]^+$ (94), 142 $[\text{M}-\text{NHCH}_3]^+$ (100), 114 $[\text{M}-\text{NHCH}_3-\text{CO}]^+$ (55), 98 (30), 86 (37), 68 (78), 42 (35), 40 (22). Found, %: C 41.70; H 4.61; N 16.51. $\text{C}_6\text{H}_8\text{N}_2\text{O}_2\text{S}$. Calculated, %: C 41.85; H 4.68; N 16.27.

(2Z)-N-Benzyl-2-(4-oxo-1,3-thiazolidin-2-ylidene)acetamide (1b). Yield 2.27 g (61%), white powder, mp 208–209°C. IR spectrum, ν , cm^{-1} : 1301, 1539 (C=C), 1629 (C=O), 1701 (C=O), 2782, 2882, 3059, 3304 (NH). ^1H NMR spectrum, δ , ppm (J , Hz): 3.54 (2H, s, CH_2); 4.28 (2H, d, $J = 6.0$, NCH_2); 5.63 (1H, s, CH); 7.16–7.31 (5H, m, H Ph); 8.05 (1H, t, $J = 6.0$, NH); 11.19 (1H, s, NH). ^{13}C NMR spectrum, δ , ppm: 31.8; 41.8; 92.2; 126.5; 127.1; 128.1; 139.9; 152.0; 166.4; 173.8. Mass spectrum, m/z (I_{rel} , %): 248 $[\text{M}]^+$ (59), 142 $[\text{M}-\text{NHBn}]^+$ (20), 115 $[\text{M}-\text{NHBn}-\text{CO}+\text{H}]^+$ (15), 106 (100), 91 (79), 77 (10), 68 (24). Found, %: C 58.27; H 4.82; N 11.33. $\text{C}_{12}\text{H}_{12}\text{N}_2\text{O}_2\text{S}$. Calculated, %: C 58.05; H 4.87; N 11.28.

(2Z)-2-(4-Oxo-1,3-thiazolidin-2-ylidene)-N-phenylacetamide (1c).²⁹ Yield 2.99 g (85%), white powder, mp 282–285°C (mp 287–289°C²⁹).

(2Z)-N-(4-Methoxyphenyl)-2-(4-oxo-1,3-thiazolidin-2-ylidene)acetamide (1d). Yield 2.85 g (72%), white powder, mp 261–263°C. IR spectrum, ν , cm^{-1} : 1156, 1316, 1413, 1455, 1508, 1547, 1574, 1604, 1647 (C=O), 1696 (C=O), 2834, 2903, 2962, 2994, 3068, 3131, 3172, 3237, 3274 (NH), 3295. ^1H NMR spectrum, δ , ppm (J , Hz): 3.60 (2H, s, CH_2); 3.74 (3H, s, CH_3); 5.76 (1H, s, CH); 6.78 (2H, d, $J = 9.0$, H Ar); 7.50 (2H, d, $J = 9.0$, H Ar); 9.51 (1H, s, NH); 11.35 (1H, s, NH). ^{13}C NMR spectrum, δ , ppm: 32.0; 55.1; 92.6; 113.8; 120.0; 133.0; 153.5; 154.7; 164.9; 174.1. Mass spectrum, m/z (I_{rel} , %): 264 $[\text{M}]^+$ (9), 142 $[\text{M}-\text{NHC}_6\text{H}_4\text{OCH}_3]^+$ (7), 123 (100), 108 (48), 68 (15). Found, %: C 54.30; H 4.69; N 10.71. $\text{C}_{12}\text{H}_{12}\text{N}_2\text{O}_3\text{S}$. Calculated, %: C 54.53; H 4.58; N 10.60.

(2Z)-N-(4-Nitrophenyl)-2-(4-oxo-1,3-thiazolidin-2-ylidene)acetamide (1e). Yield 2.97 g (71%), yellow powder, mp 252–260°C (decomp.). IR spectrum, ν , cm^{-1} : 1106, 1136, 1256, 1306, 1323 (NO_2), 1406, 1498, 1529, 1543, 1581 (NO_2), 1679 (C=O), 1707 (C=O), 2818, 2898, 3009, 3066, 3109, 3366 (NH). ^1H NMR spectrum, δ , ppm (J , Hz): 3.64 (2H, s, CH_2); 5.82 (1H, s, CH); 7.83 (2H, d, $J = 9.2$, H Ar); 8.10 (2H, d, $J = 9.2$, H Ar); 10.28 (1H, s, NH); 11.57 (1H, s, NH). ^{13}C NMR spectrum, δ , ppm (J , Hz): 32.2; 91.9; 118.0; 125.0; 141.4; 146.2; 157.0; 165.8; 174.3. Mass spectrum, m/z (I_{rel} , %): 280 $[\text{M}+\text{H}]^+$ (2), 279 $[\text{M}]^+$ (15), 142 $[\text{M}-\text{NHC}_6\text{H}_4\text{NO}_2]^+$ (100), 138 (44), 114 $[\text{M}-\text{NHC}_6\text{H}_4\text{NO}_2-\text{CO}]^+$ (36), 108 (13), 86 (25), 68 (68). Found, %: C 47.39; H 3.46; N 15.21. $\text{C}_{11}\text{H}_9\text{N}_3\text{O}_4\text{S}$. Calculated, %: C 47.31; H 3.25; N 15.05.

Preparation of 4-(4-oxo-1,3-thiazolidin-2-ylidene)pyrrolidine-2,3,5-triones 3a–e (General method). Anhydrous THF (2 ml) was cooled in ice bath and oxalyl chloride (41 μl , 0.48 mmol) was dissolved in it with stirring, followed by the addition of thiazolidine **1a–d** (0.4 mmol)

after 2 min. The obtained suspension was stirred at the same temperature for 5 h, then a 1:4 mixture of H_2O –THF (1 ml) was gradually added to the reaction mixture until the evolution of bubbles ceased. Cold water (2 ml) was then added, the precipitate was filtered off and washed with Et_2O .

1-Methyl-4-(4-oxo-1,3-thiazolidin-2-ylidene)pyrrolidine-2,3,5-trione (3a). Yield 78 mg (86%), white powder, mp 246–250°C (decomp.). IR spectrum, ν , cm^{-1} : 1026, 1150, 1235, 1327, 1559, 1574 (C=C), 1670 (C=O), 1713 (C=O), 1739 (C=O), 1763 (C=O), 3219 (NH). ^1H NMR spectrum ($\text{DMSO}-d_6$), δ , ppm: 3.03 (3H, s, CH_3); 4.00 (1H, s) and 4.02 (1H, s, CH_2); 11.69 (1H, br. s, NH). ^1H NMR spectrum ($\text{DMF}-d_6$), δ , ppm: 3.05 (3H, s, CH_3); 4.19 (2H, s, CH_2); 11.87 (1H, br. s, NH). ^{13}C NMR spectrum, δ , ppm: 23.4; 32.1; 96.5; 162.3 (2C); 167.2; 175.0 (2C). Mass spectrum, m/z (I_{rel} , %): 226 $[\text{M}]^+$ (55), 198 $[\text{M}-\text{CO}]^+$ (1), 169 $[\text{M}-\text{C}_2\text{O}_2\text{H}]^+$ (32), 141 $[\text{M}-\text{C}_2\text{O}_2\text{H}-\text{CO}]^+$ (100), 99 (14), 71 (12), 68 (47), 46 (35). Found, %: C 42.32; H 2.68; N 12.22. $\text{C}_8\text{H}_6\text{N}_2\text{O}_4\text{S}$. Calculated, %: C 42.48; H 2.67; N 12.38.

1-Benzyl-4-(4-oxo-1,3-thiazolidin-2-ylidene)pyrrolidine-2,3,5-trione (3b). Yield 106 mg (88%), light-green powder, mp 232–236°C (decomp.). IR spectrum, ν , cm^{-1} : 1238, 1328, 1389, 1568 (C=C), 1674 (C=O), 1712 (C=O), 1761 (C=O), 3219 (NH). ^1H NMR spectrum, δ , ppm: 4.02 (1H, s) and 4.04 (1H, s, CH_2); 4.72 (2H, s, NCH_2); 7.23–7.38 (5H, m, H Ph); 11.82 (1H, br. s, NH). ^{13}C NMR spectrum, δ , ppm: 32.1; 40.7; 96.3; 127.3; 127.4; 128.4; 136.1; 161.9 (2C); 168.0; 174.8 (2C). Mass spectrum, m/z (I_{rel} , %): 302 $[\text{M}]^+$ (100), 274 $[\text{M}-\text{CO}]^+$ (11), 245 $[\text{M}-\text{C}_2\text{O}_2\text{H}]^+$ (12), 211 (19), 186 (10), 171 (15), 141 $[\text{M}-\text{C}_2\text{O}_2\text{NHCH}_2\text{C}_6\text{H}_5+\text{H}]^+$ (88), 106 (42), 91 (96), 70 (27), 68 (43), 46 (32), 39 (17). Found, %: C 55.85; H 3.15; N 9.32. $\text{C}_{14}\text{H}_{10}\text{N}_2\text{O}_4\text{S}$. Calculated, %: C 55.62; H 3.33; N 9.27.

4-(4-Oxo-1,3-thiazolidin-2-ylidene)-1-phenylpyrrolidine-2,3,5-trione (3c). Yield 95 mg (82%), white powder, mp 267–272°C (decomp.). IR spectrum, ν , cm^{-1} : 1138, 1216, 1399, 1496, 1573 (C=C), 1678 (C=O), 1719 (C=O), 1746 (C=O), 1787 (C=O), 2932, 2982, 3190 (NH). ^1H NMR spectrum, δ , ppm (J , Hz): 4.04 (1H, s) and 4.06 (1H, s, CH_2); 7.34–7.43 (3H, m, H Ph); 7.49 (2H, dd, $J = 7.5$, $J = 7.5$, H Ph); 11.84 (1H, br. s, NH). ^{13}C NMR spectrum, δ , ppm: 32.3; 96.4; 126.9; 128.2; 128.8; 131.5; 161.2 (2C); 168.7; 175.3 (2C). Mass spectrum, m/z (I_{rel} , %): 288 $[\text{M}]^+$ (31), 260 $[\text{M}-\text{CO}]^+$ (27), 141 $[\text{M}-\text{C}_2\text{O}_2\text{NHC}_6\text{H}_5+\text{H}]^+$ (100), 119 (54), 99 (11), 91 (24), 77 (10), 68 (35), 46 (24). Found, %: C 54.11; H 2.74; N 9.83. $\text{C}_{13}\text{H}_8\text{N}_2\text{O}_4\text{S}$. Calculated, %: C 54.16; H 2.80; N 9.72.

1-(4-Methoxyphenyl)-4-(4-oxo-1,3-thiazolidin-2-ylidene)pyrrolidine-2,3,5-trione (3d). Yield 108 mg (85%), light-green powder, mp 235–240°C (decomp.). IR spectrum, ν , cm^{-1} : 1145, 1163, 1211, 1242, 1253, 1299, 1330, 1392, 1405, 1441, 1468, 1512, 1568 (C=C), 1679 (C=O), 1715 (C=O), 1766 (C=O), 2834, 2955, 3012, 3212 (NH). ^1H NMR spectrum, δ , ppm (J , Hz): 3.82 (3H, s, CH_3); 4.05 (2H, s, CH_2); 6.99 (2H, d, $J = 8.0$, H Ar); 7.25 (2H, d, $J = 8.0$, H Ar); 11.78 (1H, br. s, NH). ^{13}C NMR spectrum, δ , ppm: 32.3; 55.4; 96.4; 114.0; 123.9; 128.2; 158.9; 161.4

(2C); 168.3; 175.2 (2C). Mass spectrum, m/z (I_{rel} , %): 318 $[M]^+$ (19), 290 $[M-CO]^+$ (9), 149 (100), 134 (29), 106 (13). Found, %: C 52.68; H 2.96; N 8.69. $C_{14}H_{10}N_2O_5S$. Calculated, %: C 52.83; H 3.17; N 8.80.

1-(4-Nitrophenyl)-4-(4-oxo-1,3-thiazolidin-2-ylidene)pyrrolidine-2,3,5-trione (3e) was obtained according to the general procedure from thiazolidine **1e** (0.12 g, 0.4 mmol) in anhydrous MeCN, by heating the suspension for 30 min at 80°C. The mixture was treated with 1:4 mixture of H₂O–MeCN (1 ml). Yield 67 mg (50%), light-yellow powder, mp 260–285°C (decomp.). ¹H NMR spectrum, δ , ppm (J , Hz): 4.07 (2H, s, CH₂); 7.73 (2H, d, J = 8.8, H Ar); 8.35 (2H, d, J = 8.8, H Ar). The substance is not obtained in analytically pure form.

Supplementary information file containing IR, ¹H and ¹³C NMR, and mass spectra of compounds **1a–e**, **3a–e**, is available from the journal website at <http://link.springer.com/journal/10593>.

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