Synthesis A. Ziyaei Halimehjani, S. Hosseinkhany Paper

One-Pot Three-Component Route for the Synthesis of Rhodanine Derivatives in Water

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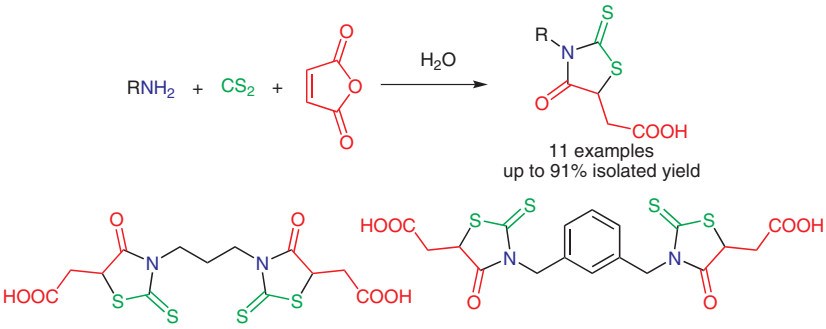

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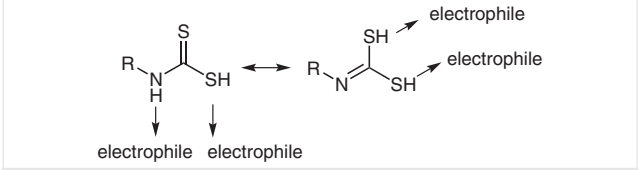
Abstract A one-pot three-component route for the synthesis of rhodanine derivatives by the reaction of primary amines, carbon disulfide, and maleic anhydride in water is described. The reactions proceed via a sequential Michael addition–intramolecular amide bond formation. Bis(rhodanine) derivatives were also synthesized by using a diamine in this protocol.

Key words rhodanine derivatives, dithiocarbamates, water, primary amines, carbon disulfide

Multicomponent reactions (MCRs), which can produce a diversity of complex compounds in a limited number of reaction steps, provide one of the most efficient and effective methods for the sustainable and diversity-oriented synthesis of biologically active compounds.[1] Therefore, besides the famous multicomponent reactions reported to date, the design of novel MCRs has attracted great attention from research groups working in various areas such as drug discovery, organic synthesis, and materials science. In addition, performing the MCRs under green reaction conditions improves their efficiency in the modern organic synthesis. Among the different branches of green chemistry, conducting an organic reaction in water, under solvent- or catalyst-free conditions, is ideal for the environmentally friendly synthesis of organic compounds.[2] Although the use of catalysts for organic transformations has several advantages, usually such processes require extra steps for producing the catalyst or for removing the catalyst from the reaction medium.

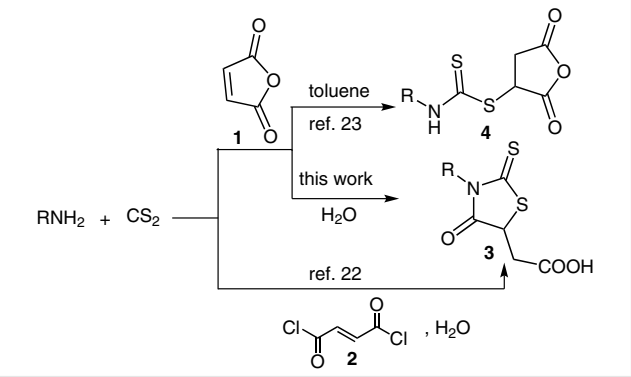
Among the five-membered heterocyclic compounds containing N/S heteroatoms, rhodanine (2-thioxo-1,3-thiazolidin-4-one) and its derivatives are well-known molecules due to their diverse biological activities including antimicrobial,[3] antiviral,[4] anticonvulsant,[5] antidiabetic,[6] antimalarial,[7] antifungal,[8] antitumor,[9] anti-inflammatory,[10] and herbicidal properties.[11] These compounds have also found wide applications as inhibitors of numerous targets for pharmaceutical interventions in a number of diseases.[12] Usually, these compounds have been prepared via the addition of isothiocyanate to mercaptoacetic acid followed by acid cyclization,[13] or the reaction of in situ prepared dithiocarbamic acids from primary amines or ammonia with a diversity of electrophiles such as chloroacetic acid,[13] chloroacetyl chloride,[14] ethyl chloroacetate,[15] diethyl 2-chloromalonate,[16] dialkyl acetylenedicarboxylates,[17] α-chloro-β,γ-alkenoate esters,[18] and 1,2-diaza-1,3-dienes.[19]

Dithiocarbamates are the analogues of carbamates in which both oxygens are replaced by sulfur. These compounds can be simply accessed via the reaction of an amine, carbon disulfide, and an electrophile such as an alkyl halide, epoxide, carbonyl compound, or Michael acceptor.[20] Dithiocarbamic acids derived from primary amines are potential bis-nucleophiles due to the sulfur and nitrogen groups (Scheme 1). At first, they usually attack an electrophile through a sulfur atom, and then through the nitrogen atom or thiocarbonyl group. The second attack via the nitrogen or thiocarbonyl group is dependent on the structure of the electrophile.[13–19,21]



Scheme 1 Sulfur and nitrogen nucleophilicity of dithiocarbamates

In continuation of our research toward the development of green procedures for the synthesis of dithiocarbamates and their applications in organic transformations, herein we report a green one-pot three-component route for the synthesis of rhodanine derivatives **3** in water at room temperature without using any catalyst, as outlined in Scheme 2. Recently, these compounds have been synthesized by Alizadeh and Zohreh via a one-pot three-component reaction of primary amines, carbon disulfide, and fumaryl chloride (**2**) in water.[22] We have found that by replacing the sensitive, expensive, and toxic fumaryl chloride (**2**) with maleic anhydride (**1**), the same products can be obtained in higher yield and in shorter reaction time. Another report in this context, by Shahvelayati and co-workers, revealed that the reaction of primary amines, carbon disulfide, and maleic anhydride (**1**) in toluene gave only the Michael adducts **4**. Furthermore, they also claimed that the Michael adducts are stable in refluxing toluene for 12 hours.[23]



Scheme 2 One-pot three-component reaction of primary amines, carbon disulfide, and maleic anhydride or fumaryl chloride

Reaction of dithiocarbamic acids with maleic anhydride was optimized with the reaction of benzylamine, carbon disulfide, and maleic anhydride in water by varying the equivalents of the starting materials and the reaction temperature. Contrary to the report by Shahvelayati and co-workers,[23] we found that simple mixing of benzylamine (3 mmol) and carbon disulfide (5 mmol) in water (10 mL) for 20 minutes, followed by addition of maleic anhydride (3 mmol) and further stirring at room temperature for 5 hours, gave only (3-benzyl-4-oxo-2-thioxothiazolidin-5-yl)acetic acid (**3a**) in 73% yield (Table 1, entry 1); no Michael adduct was observed. Continuing on, the generality of the reaction was confirmed by using various primary amines such as butylamine, propylamine, ethylamine, methylamine, isopropylamine, allylamine, hexylamine, cyclohexylamine, 1-phenylethylamine, and 3-phenylpropylamine; in each case, a high to excellent yield was obtained (Table 1, entries 2–11). Hindered primary amines such as *tert*-butylamine and 1-aminoadamantane were also examined, without success.

Then, the generality of the reaction was expanded to diamines, namely 1,3-diaminopropane and 3-(aminomethyl)benzylamine. We found that by using 3 equivalents of carbon disulfide and 2 equivalents of maleic anhydride with 1 equivalent of a diamine under similar conditions as described above, the corresponding bis(rhodanine) derivatives **5a**,**b** were obtained in high to excellent yield (Scheme 3). The structures of all products were elucidated from their elemental analyses, and IR, ^{1}H NMR, and ^{13}C NMR spectra. It is notable that in large-scale syntheses, the product can be simply collected via decanting in the case of an oily product or by filtration of the precipitate from the reaction mixture.

Table 1 Rhodanine Derivatives from the Reaction of Primary Amines, Carbon Disulfide, and Maleic Anhydride in Water[a]

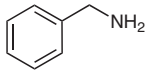
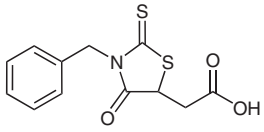
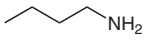
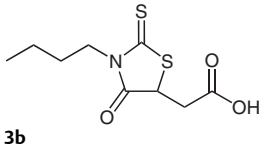
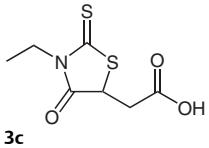
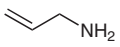
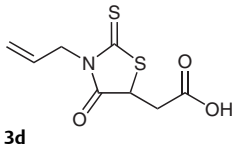
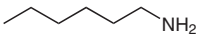
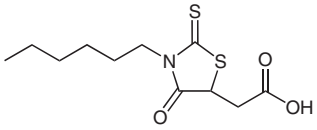
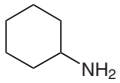
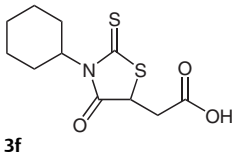
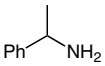
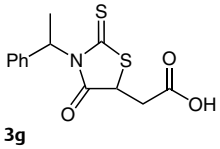
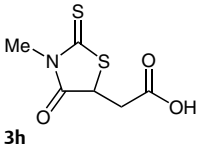
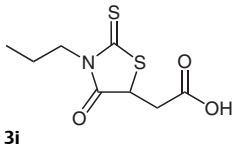
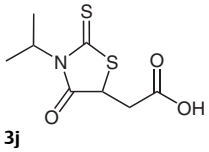
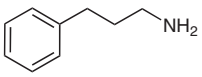
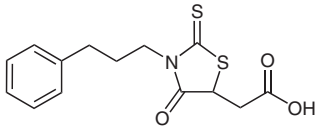
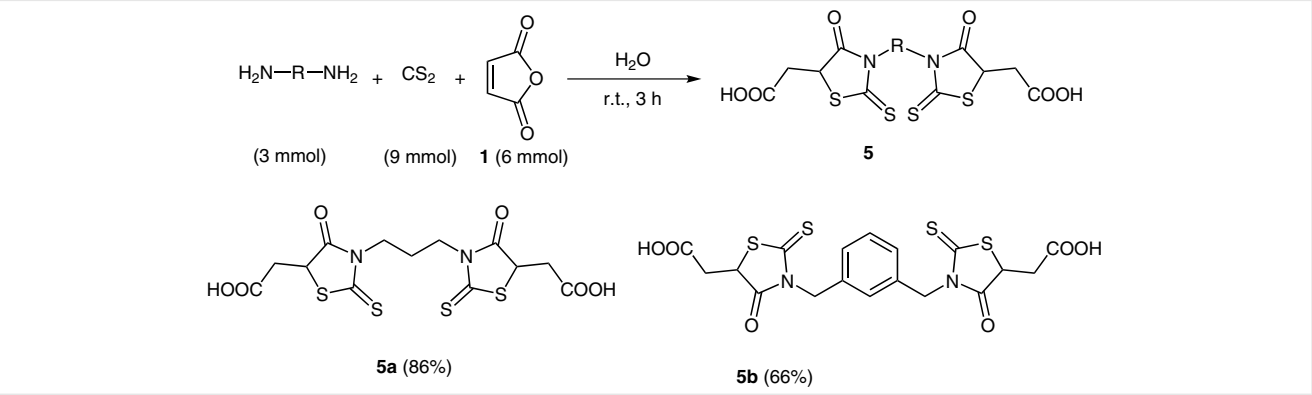
Entry	Amine	Product	Yield[b] (%)
1		 **3a**	73
2		 **3b**	91
3		 **3c**	66

Table 1 (continued)

Entry	Amine	Product	Yield[b] (%)
4		 **3d**	76
5		 **3e**	73
6		 **3f**	71
7		 **3g**	78
8		 **3h**	69
9		 **3i**	80
10		 **3j**	63
11		 **3k**	56

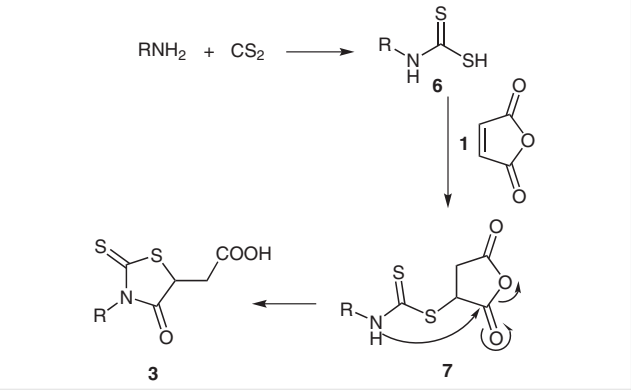
[a] Reaction conditions: amine (3 mmol), CS_2 (5 mmol), maleic anhydride (**1**; 3 mmol), H_2O (10 mL), r.t., 5 h.
[b] Isolated yield.

A proposed mechanism for this transformation is given in Scheme 4. It is conceivable that the initial event is the formation of a dithiocarbamic acid **6** from a primary amine and carbon disulfide. Further, Michael addition of the in situ prepared **6** to maleic anhydride (**1**) affords the intermediate **7**, which then undergoes intramolecular amide bond formation via a 5-*exo*-*trig* nucleophilic attack by nitrogen at the anhydride group to give the corresponding product **3**.



Scheme 3 Synthesis of bis(rhodanine) derivatives using diamines

In conclusion, we have demonstrated a facile, highly efficient, and environmentally benign procedure for the one-pot synthesis of (3-alkyl-4-oxo-2-thioxothiazolidin-5-yl)acetic acids (rhodanine derivatives) in water in high to excellent yields using simple and inexpensive starting materials. This procedure was also applied for the synthesis of bis(rhodanine) derivatives from diamines. The presence of a carboxylic acid group in the structure of the products allows further constructions in synthetic organic chemistry for the preparation of novel, biologically active compounds.



Scheme 4 Proposed mechanism for the formation of rhodanines **3**

All chemicals and solvents were obtained from commercial sources and used without further purification. All ^{1}H and ^{13}C NMR spectra were obtained on a Bruker AMX 300 MHz spectrometer and referenced to internal tetramethylsilane at 0.0 ppm. Reactions were monitored by thin-layer chromatography using Merck TLC silica gel 60 F254 plates. IR spectra were recorded on a Perkin-Elmer Spectrum RXI FT-IR spectrometer. Elemental analyses were conducted with a Perkin-Elmer 2004 Series II CHN analyzer.

Rhodanine Derivatives 3; General Procedure

To a magnetically stirred mixture of an amine (3 mmol) and H_2O (10 mL), was added CS_2 (5 mmol). The mixture was stirred for 20 min, which was followed by the addition of maleic anhydride (**1**; 3 mmol) and further stirring at r.t. for 5 h. On reaction completion, the product was extracted with EtOAc (2 × 20 mL), and the combined organic layers were washed with water and dried over anhydrous Na_2SO_4. The solvent was evaporated under reduced pressure to give the product in pure form, without need for additional purification. The products were characterized by their IR, ^{1}H NMR, and ^{13}C NMR spectra, and CHN analyses. For the synthesis of bis(rhodanine) derivatives **5**, 2 equivalents of maleic anhydride and 3 equivalents of CS_2 were applied. In large-scale syntheses, the product can be simply collected via decanting in the case of an oily product or by filtration of the precipitate from the reaction mixture.

(3-Benzyl-4-oxo-2-thioxothiazolidin-5-yl)acetic Acid (3a)

Yield: 0.615 g (73%); yellow solid; mp 92–95 °C.

IR (KBr): 3550–2400 (OH), 1704, 1350, 1242, 1194 cm^{-1}.

^{1}H NMR (300 MHz, $CDCl_3$): δ = 3.06 (dd, *J* = 18.0, 8.9 Hz, 1 H), 3.27 (dd, *J* = 18.1, 3.8 Hz, 1 H), 4.43 (dd, *J* = 8.9, 3.8 Hz, 1 H), 5.21 ($AB_{quartet}$, *J* = 14.1 Hz, 2 H), 7.26–7.42 (m, 5 H), 8.9 (br s, 1 H).

^{13}C NMR (75 MHz, $CDCl_3$): δ = 36.3, 45.4, 47.8, 128.1, 128.5, 129.1, 134.5, 174.5, 175.4, 200.1.

Anal. Calcd for $C_{12}H_{11}NO_3S_2$: C, 51.23; H, 3.94; N, 4.98. Found: C, 51.47; H, 3.72; N, 5.21.

(3-Butyl-4-oxo-2-thioxothiazolidin-5-yl)acetic Acid (3b)

Yield: 0.674 g (91%); colorless solid; mp 79–81 °C.

IR (KBr): 3350–2400 (OH), 1733, 1707, 1360, 1336, 1282, 1194, 1131 cm^{-1}.

^{1}H NMR (300 MHz, $CDCl_3$): δ = 0.95 (t, *J* = 7.2 Hz, 3 H), 1.35 (m, 2 H), 1.62 (m, 2 H), 3.00 (dd, *J* = 17.8, 9.8 Hz, 1 H), 3.25 (dd, *J* = 17.9, 3.3 Hz, 1 H), 3.99 (t, *J* = 7.4 Hz, 2 H), 4.41 (dd, *J* = 8.2, 3.4 Hz, 1 H), 8.36 (br s, 1 H).

^{13}C NMR (75 MHz, $CDCl_3$): δ = 13.6, 19.9, 28.6, 36.7, 44.6, 45.6, 174.2, 175.6, 200.6.

Anal. Calcd for $C_9H_{13}NO_3S_2$: C, 43.70; H, 5.30; N, 5.66. Found: C, 44.01; H, 5.48; N, 6.05.

(3-Ethyl-4-oxo-2-thioxothiazolidin-5-yl)acetic Acid (3c)

Yield: 0.433 g (66%); brownish solid; mp 87–90 °C.

IR (KBr): 3450–2400 (OH), 1722, 1709, 1436, 1344, 1252, 1125 cm^{-1}.

^1H NMR (300 MHz, $CDCl_3$): δ = 1.22 (t, *J* = 7.0 Hz, 3 H), 3.08 (dd, *J* = 18.1, 8.6 Hz, 1 H), 3.28 (dd, *J* = 18.0, 3.6 Hz, 1 H), 4.06 (t, *J* = 6.9 Hz, 2 H), 4.41 (dd, *J* = 8.5, 3.7 Hz, 1 H), 8.61 (br s, 1 H).

^{13}C NMR (75 MHz, $CDCl_3$): δ = 11.8, 36.3, 40.0, 45.3, 174.9, 175.2, 200.0.

Anal. Calcd for $C_7H_9NO_3S_2$: C, 38.34; H, 4.14; N, 6.39. Found: C, 37.99; H, 4.09; N, 6.07.

(3-Allyl-4-oxo-2-thioxothiazolidin-5-yl)acetic Acid (3d)

Yield: 0.527 g (76%); yellowish solid; mp 89–92 °C.

IR (KBr): 3280–2350 (OH), 1728, 1703, 1646, 1333, 1240, 1139 cm^{-1}.

^1H NMR (300 MHz, $CDCl_3$): δ = 3.06 (dd, *J* = 18.1, 8.8 Hz, 1 H), 3.29 (dd, *J* = 18.0, 3.8 Hz, 1 H), 4.44 (dd, *J* = 8.8, 3.6 Hz, 1 H), 4.60 (d, *J* = 5.9 Hz, 2 H), 5.23–5.32 (m, 2 H), 5.76 (m, 1 H), 9.41 (br s, 1 H).

^{13}C NMR (75 MHz, $CDCl_3$): δ = 36.4, 45.4, 46.6, 119.6, 129.2, 174.9, 175.0, 199.7.

Anal. Calcd for $C_8H_9NO_3S_2$: C, 41.54; H, 3.92; N, 6.06. Found: C, 41.66; H, 3.72; N, 6.17.

(3-Hexyl-4-oxo-2-thioxothiazolidin-5-yl)acetic Acid (3e)

Yield: 0.602 g (73%); yellow syrup.

IR (KBr): 3500–2400 (OH), 1730, 1352, 1183, 1137 cm^{-1}.

^1H NMR (300 MHz, $CDCl_3$): δ = 0.87 (t, *J* = 6.1 Hz, 3 H), 1.28–1.30 (m, 6 H), 1.58 (m, 2 H), 2.95 (dd, *J* = 17.8, 9.1 Hz, 1 H), 3.22 (dd, *J* = 17.8, 3.7 Hz, 1 H), 3.94 (m, 2 H), 4.39 (dd, *J* = 9.1, 3.7 Hz, 1 H), 9.8 (br s, 1 H).

^{13}C NMR (75 MHz, $CDCl_3$): δ = 13.9, 22.4, 26.3, 26.5, 31.2, 36.7, 44.8, 45.6, 174.1, 175.6, 200.8.

Anal. Calcd for $C_{11}H_{17}NO_3S_2$: C, 47.98; H, 6.22; N, 5.09. Found: C, 48.20; H, 6.47; N, 5.36.

(3-Cyclohexyl-4-oxo-2-thioxothiazolidin-5-yl)acetic Acid (3f)

Yield: 0.581 g (71%); cream solid; mp 135–139 °C.

IR (KBr): 3300–2400 (OH), 1743, 1703, 1200, 1137 cm^{-1}.

^1H NMR (300 MHz, $CDCl_3$): δ = 1.20–1.43 (m, 5 H), 1.84–1.88 (m, 3 H), 2.26–2.34 (m, 2 H), 2.95 (dd, *J* = 17.9, 8.6 Hz, 1 H), 3.16 (dd, *J* = 17.8, 3.3 Hz, 1 H), 4.24 (m, 1 H), 4.87 (t, *J* = 12.2 Hz, 1 H), 9.8 (br s, 1 H).

^{13}C NMR (75 MHz, $CDCl_3$): δ = 25.2, 25.9, 27.4, 27.6, 31.0, 36.7, 43.7, 58.3, 174.6, 176.0, 201.7.

Anal. Calcd for $C_{11}H_{15}NO_3S_2$: C, 48.33; H, 5.53; N, 5.12. Found: C, 48.06; H, 5.14; N, 5.59.

[4-Oxo-3-(1-phenylethyl)-2-thioxothiazolidin-5-yl]acetic Acid (3g)

Yield: 0.690 g (78%); yellow syrup.

IR (KBr): 3400–2400 (OH), 1730, 1243, 1095 cm^{-1}.

^1H NMR (300 MHz, $CDCl_3$): δ = 1.82–1.88 (m, 3 H), 3.04–3.12 (m, 2 H), 4.12–4.16 (m, 1 H), 6.40 (m, 1 H), 7.28–7.45 (m, 5 H), 8.06 (br s, 1 H).

^{13}C NMR (75 MHz, $CDCl_3$): δ = 14.5, 36.1, 43.9, 55.0, 127.1, 127.7, 128.2, 137.9, 174.1, 175.1, 201.3.

Anal. Calcd for $C_{13}H_{13}NO_3S_2$: C, 52.86; H, 4.44; N, 4.74. Found: C, 52.68; H, 4.28; N, 4.89.

(3-Methyl-4-oxo-2-thioxothiazolidin-5-yl)acetic Acid (3h)

Yield: 0.424 g (69%); greenish solid; mp 96–100 °C.

IR (KBr): 3400–2400 (OH), 1700, 1309, 1132 cm^{-1}.

^1H NMR (300 MHz, $CDCl_3$): δ = 3.03 (dd, *J* = 18.1, 9.3 Hz, 1 H), 3.32–3.40 (m, 4 H), 4.46 (dd, *J* = 9.3, 3.7 Hz, 1 H).

^{13}C NMR (75 MHz, $CDCl_3$): δ = 31.5, 36.4, 45.7, 174.2, 175.3, 201.3.

Anal. Calcd for $C_6H_7NO_3S_2$: C, 35.11; H, 3.44; N, 6.82. Found: C, 35.24; H, 3.11; N, 6.62.

(4-Oxo-3-propyl-2-thioxothiazolidin-5-yl)acetic Acid (3i)

Yield: 0.559 g (80%); yellow solid; mp 89–92 °C.

IR (KBr): 3300–2400 (OH), 1729, 1707, 1357, 1285, 1242, 1213, 1132 cm^{-1}.

^1H NMR (300 MHz, $CDCl_3$): δ = 0.93 (t, *J* = 7.4 Hz, 3 H), 1.60–1.73 (m, 2 H), 3.05 (dd, *J* = 18.1, 8.7 Hz, 1 H), 3.26 (dd, *J* = 18.1, 3.8 Hz, 1 H), 3.92–3.97 (m, 2 H), 4.04 (m, 1 H), 9.88 (br s, 1 H).

^{13}C NMR (75 MHz, $CDCl_3$): δ = 11.1, 20.0, 36.4, 45.2, 46.3, 175.2, 175.5, 200.3.

Anal. Calcd for $C_8H_{11}NO_3S_2$: C, 41.18; H, 4.75; N, 6.00. Found: C, 41.47; H, 4.76; N, 5.99.

(3-Isopropyl-4-oxo-2-thioxothiazolidin-5-yl)acetic Acid (3j)

Yield: 0.44 g (63%); yellow solid; mp 152–155 °C.

IR (KBr): 3250–2400 (OH), 1723, 1701, 1320, 1263, 1115 cm^{-1}.

^1H NMR (300 MHz, $CDCl_3$): δ = 1.40–1.49 (m, 6 H), 3.03–3.24 (m, 2 H), 4.24 (m, 1 H), 5.22 (m, 1 H), 10.60 (br s, 1 H).

^{13}C NMR (75 MHz, $CDCl_3$): δ = 17.9, 18.2, 36.2, 43.4, 50.4, 175.3, 175.7, 201.0.

Anal. Calcd for $C_8H_{11}NO_3S_2$: C, 41.18; H, 4.75; N, 6.00. Found: C, 41.33; H, 4.31; N, 6.07.

[4-Oxo-3-(3-phenylpropyl)-2-thioxothiazolidin-5-yl]acetic Acid (3k)

Yield: 0.519 g (56%); colorless solid; mp 102–107 °C.

IR (KBr): 3200–2400 (OH), 1738, 1707, 1262, 1157 cm^{-1}.

^1H NMR (300 MHz, $CDCl_3$): δ = 1.96–2.07 (m, 2 H), 2.69 (t, *J* = 7.8 Hz, 2 H), 2.97 (dd, *J* = 18.1, 8.8 Hz, 1 H), 3.22 (dd, *J* = 18.1, 3.8 Hz, 1 H), 4.05 (t, *J* = 7.5 Hz, 2 H), 4.29 (dd, *J* = 8.7, 3.8 Hz, 1 H), 7.17–7.32 (m, 5 H), 8.70 (br s, 1 H).

^{13}C NMR (75 MHz, $CDCl_3$): δ = 27.5, 32.9, 36.8, 44.7, 45.1, 126.1, 128.2, 128.4, 140.5, 174.6, 175.4, 200.2.

Anal. Calcd for $C_{14}H_{15}NO_3S_2$: C, 54.35; H, 4.89; N, 4.53. Found: C, 54.63; H, 4.61; N, 4.70.

{3-[3-(5-Carboxymethyl-4-oxo-2-thioxothiazolidin-3-yl)propyl]-4-oxo-2-thioxothiazolidin-5-yl}acetic Acid (5a)

Yield: 1.089 g (86%); cream solid; mp 170–172 °C.

IR (KBr): 3400–2400 (OH), 1743, 1709, 1358, 1229, 1102 cm^{-1}.

^1H NMR (300 MHz, DMSO-d_6): δ = 1.86 (t, *J* = 7.4 Hz, 2 H), 3.00–3.15 (m, 4 H), 3.88 (m, 4 H), 4.69 (t, *J* = 6.3 Hz, 2 H), 12.80 (br s, 2 H).

^{13}C NMR (75 MHz, DMSO-d_6): δ = 23.8, 35.5, 41.6, 46.0, 171.5, 176.0, 202.7.

Anal. Calcd for $C_{13}H_{14}N_2O_6S_4$: C, 36.95; H, 3.34; N, 6.63. Found: C, 37.06; H, 3.56; N, 6.48.

(3-{3-[(5-Carboxymethyl-4-oxo-2-thioxothiazolidin-3-yl)methyl]benzyl}-4-oxo-2-thioxothiazolidin-5-yl)acetic Acid (5b)

Yield: 0.96 g (66%); yellow solid; mp 93–97 °C.

IR (KBr): 3450–2400 (OH), 1731, 1341, 1187 cm^{-1}.

^1H NMR (300 MHz, DMSO-d_6): δ = 3.09 (d, J = 4.5 Hz, 4 H), 4.80 (d, J = 4.5 Hz, 2 H), 5.01 ($AB_{quartet}$, J = 15.3 Hz, 4 H), 7.15–7.42 (m, 4 H).

^{13}C NMR (75 MHz, DMSO-d_6): δ = 35.6, 46.4, 46.9, 126.4, 126.6, 128.5, 135.1, 171.6, 176.2, 202.8.

Anal. Calcd for $C_{18}H_{16}N_2O_6S_4$: C, 44.61; H, 3.33; N, 5.78. Found: C, 44.47; H, 3.53; N, 5.68.

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Supporting Information

Supporting information for this article is available online at http://dx.doi.org/10.1055/s-0034-1380454.

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