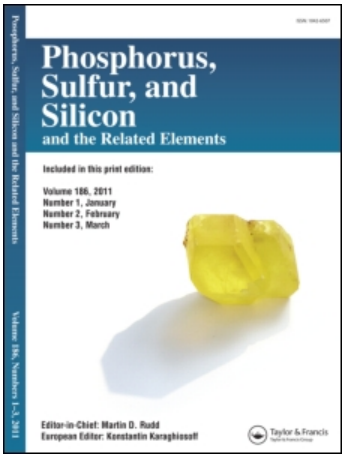

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Utilization of Methyl 3-Aryl-2-thiocyanatopropanoates in the Synthesis of 2-(4-Morpholinyl)- and 2-(Piperazinyl)-5-(benzyl)thiazol-4-ones[1]

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Utilization of Methyl 3-Aryl-2-thiocyanatopropanoates in the Synthesis of 2-(4-Morpholinyl)- and 2-(Piperazinyl)-5-(benzyl)thiazol-4-ones[1]

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*By the reaction of methyl acrylate with arenediazonium salts and KSCN in the presence of copper(II) acetate, the methyl 3-aryl-2-thiocyanatopropanoates **2** have been obtained. These compounds react with morpholine or monosubstituted piperazines in the presence of acetic acid to form 2-(4-morpholinyl)- or 2-(4-R-piperazinyl)-5-(R′-benzyl)thiazol-4-ones **4** and **5**, respectively.*

Keywords Arylation; cyclization; piperazines; thiocyanates; Thiazol-4-ones

INTRODUCTION

Thiazolidin-4-one and thiazol-4-one derivatives have been widely employed in the investigation of biologically active heterocyclic compounds.[2–5] Therefore, the synthesis of combinatorial libraries of these compounds was elaborated.[6–9] In the recent past, 5-R-benzyl-2,4-thiazolidinones have been investigated intensively, since series of these compounds are already used as antidiabetic drugs.[10–15] The 2-amino(imino)-4-thiazolidinones have considerable interest for pharmacological investigations and for chemical modifications.[2]

A general synthesis of such compounds is based on the cyclization of thioureas or other S,N-nucleophiles with α-haloacids or their esters.[2,16] However, the ring closure takes place selectively, only in the

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[1]Synthesis of Heterocycles on the Basis of Arylation Products of Unsaturated Compounds, Communication 15.–Communication 14 see [1].

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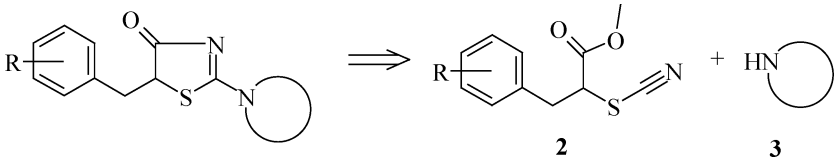
case of considerable difference in the nucleophility of the N-atoms of the reagents.[17] Another strategy for the construction of thiazolidin-4-ones or thiazol-4-ones avoids that complication. The method is based on the reaction of α-thiocyanatocarboxylic acid derivatives with amines.[18–20] Through interaction with thiourea, the esters of α-thiocyanatocarboxylic acid also experience ring closure. When using substituted thioureas, however, there is no ring closure.[2]

In this article, we describe an approach to the synthesis of 2-amino-5-benzylthiazol-4-ones from methyl 3-aryl-2-thiocyanatopropanoates **2** and secondary amines **3** (Scheme 1).

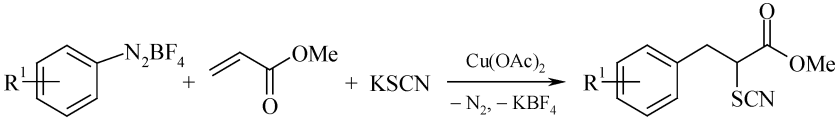


2 **3**

SCHEME 1

RESULTS AND DISCUSSION

Starting methyl 3-aryl-2-thiocyanatopropanoates **2a–d** was obtained from a one-pot procedure by the reaction of methylacrylates with arenediazonium tetrafluoroborates **1a–d** and potassium thiocyanate in the presence of copper(II) acetate as catalyst (Scheme 2).



1 a-d **2 a-d**

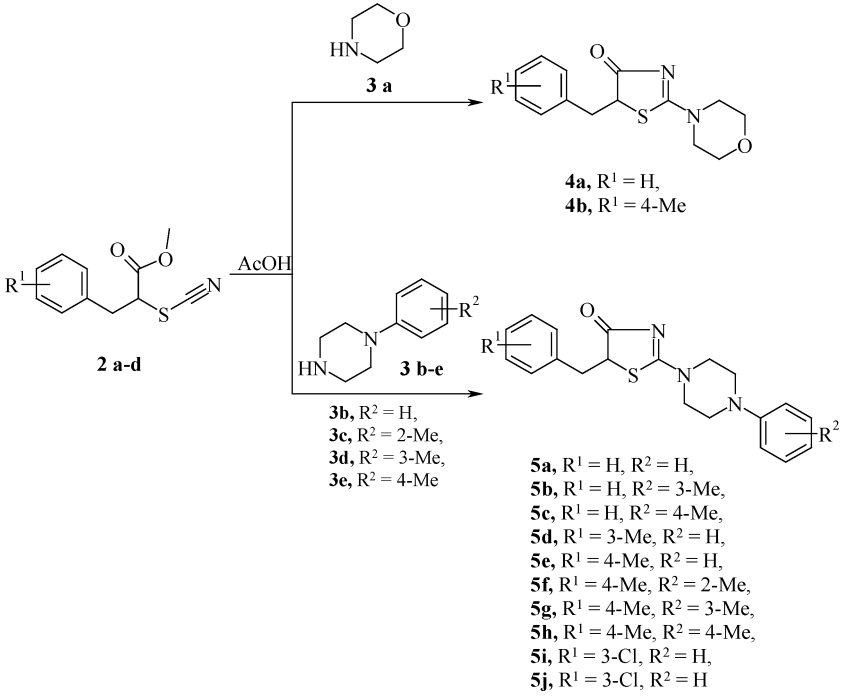
a, R= H; **b**, R= 3-Me; **c**, R= 4-Me; **d**, R= 3-Cl

SCHEME 2

Thiocyanatoarylation of unsaturated compounds is a convenient synthetical modification of the Meerwein arylation reaction.[21–23]

Research showed that compounds **2a–d** possess a low activity in the reaction with secondary amines, such as morpholine **3a** or substituted piperazines **3b–e**. Under conditions cited in the paper,[19] thiazol-4-one ring was not formed. However, we found conditions to obtain 2-substituted 5-R^1-benzylthiazol-4-ones **4a**,**b** and **5a–j** in reasonable yields (Scheme 3). Reaction occurred after long-term heating of reagents

in the presence of equimolar quantity of acetic acid in ethanol or 2-propanol.

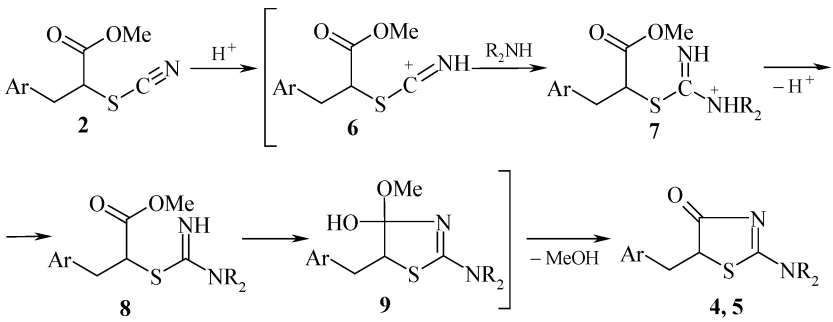


SCHEME 3

In the ^{1}H-NMR spectra, the protons of the $ArCH_2CH$ fragment appear as an AMX spin system, i.e., as three doublets of doublets. In some cases, these signals are broadened, evidently due to the fast modification of the conformation of the morpholine or piperidine ring. In the mass spectra of compounds **4**, **5**, the fragments $[R^2C_6H_4NCH_2CH_2]^+$, $[R^1C_6H_4CH_2]^+$, $[R^2C_6H_4N]^+$, and $[NCH_2CH_2]^+$ became apparent.

Yields of compounds **4**, **5** significantly depend on the form of amine **3**. By using the free amine, or its salt with strong acid, the thiazol-4-one derivatives form with low yields. The best results were obtained using acetates or propionates of corresponding amines. Such results showed that amines **3** enter into the reaction with compounds **2** as the unprotonated base. But for successful reaction the acid was necessary. Evidently, the acid promoted reaction of the nitrile group and thereby catalyzed the reaction (Scheme 4).

Protonation of the N-atom of methyl 3-aryl-2-thiocyanatopropanoates **2** caused the formation of carbocation **6**, which was



SCHEME 4

attacked by R_2NH and generated intermediate cation **7**. On proton elimination, **7** was transformed to acyclic compound **8**, which was stabilized because of cyclization and formed thiazol-4-one **4**, **5**, following the elimination of methanol.

Thus, by using of the methyl-3-aryl-2-thiocyanatopropanoates **2 a–d**, we found a convenient method to synthesize 2-amino-5-R-benzylthiazol-4-one.

EXPERIMENTAL

All melting points are uncorrected. The ^{1}H-NMR spectra were recorded on a Bruker AM300 (300 MHz) or Bruker DRX500 (500 MHz) spectrometers in DMSO-d_6 with Me_4Si as internal standard. The mass spectra were recorded on a Finnigan MAT-INKOS-50 chromatomass spectrometer at 70 eV. The monosubstituted piperazines were obtained by known methods.[24]

General Procedure for the Synthesis of Methyl 3-aryl-2-thiocyanatopropanoates (1a)

Arenediazonium tetrafluoroborate **1a–d** had been prepared in the usual manner from the appropriate aromatic amine (0.11 mol). The solid diazonium salt **1a–d** was gradually added with stirring at 5° to the mixture of acetone (75 ml), methyl acrylate (9.1 ml), and copper(II) sulphate (0.01 mol). When the N_2 evolution ceased, the mixture was diluted with 200 ml of water, extracted with CH_2Cl_2 (3 × 50 ml), the extracts were dried ($MgSO_4$), and concentrated. The residue was vacuum-distilled. Compounds **2a–c** have been described earlier.[23]

Methyl 3-(3-chlorophenyl)-2-thiocyanatopropanoate (2d)

B.p. 154°C/1.5 mm Hg; n_D^{20} 1.5600; Yield 62%. Anal. requires for $C_{11}H_{10}ClNO_2S$ (255.72) calcd./found: C 51.67/51.57; H 3.94/3.80; N 5.48/5.31.

General Procedure for Obtaining 5-R[1]-Benzyl-2-(4-morpholinyl)-1,3-thiazol-4(5H)-ones (4a, b) and 5-R[1]-benzyl-2-(4-phenyl-1-piperazinyl)-1,3-thiazol-4(5H)-ones (5 a–j)

The mixture of appropriate methyl 3-aryl-2-thiocyanatopropanoate **1a–d** (0.01 mol), amine **3a–e** (0.01 mol), and AcOH (0.6 g) in ethanol or 2-propanol (10 ml) was refluxed for 15–30 h. The course of reaction was monitored by TLC. After cooling of reaction mixture, the precipitate was formed, filtered, and crystallized from the appropriate solvent.

5-Benzyl-2-(4-morpholinyl)-1,3-thiazol-4(5H)-one (4a)

m.p. 129–130°C (hexane-benzene, 1:1) in 36% yield. ^{1}H NMR ppm: δ 2.88 (dd, $J = 14.2/10.6$, 1H); 3.43–3.51 (m, 3H); 3.66 (br. s, 4H); 3.78–3.85 (m, 2H); 4.60 (dd, $J = 10.6/4.7$, 1H); 7.19–7.31 (m, 5H). Anal. requires for $C_{14}H_{16}N_2O_2S$ (276.36) calcd./found: C, 60.85/60.77; H, 5.84/5.92; N, 10.14/10.03.

5-(4-Methybenzyl)-2-(4-morpholinyl)-1,3-thiazol-4(5H)-one (4b)

m.p. 117–118°C (cyclohexane-benzene, 2:1) in 34% yield. ^{1}H NMR ppm: δ 2.28 (s, 3H); 2.82 (br. dd, 1H); 3.37–3.48 (m, 3H); 3.64 (br. s, 4H); 3.74–3.83 (m, 2H); 4.57–4.63 (m, 1H); 7.07 (d, 2H); 7.12 (d, 2H). MS (m/z) (%): 290 (57, A^+), 105 (100, [4-$CH_3C_6H_4CH_2$]), 86 (50, [C_4H_8NO]), 42 (67, [NCH_2CH_2]). Anal. requires for $C_{15}H_{18}N_2O_2S$ (290.39) calcd./found: C 62.04/61.85; H 6.25/6.23; N 9.65/9.47.

5-Benzyl-2-(4-phenyl-1-piperazinyl)-1,3-thiazol-4(5H)-one (5a)

m.p. 123–124°C (pentane-benzene, 2:1) in 42% yield. ^{1}H NMR ppm: δ 2.89 (dd, $J = 13.8/10.2$, 1H); 3.18–3.27 (br. s, 4H); 3.49 (dd, $J = 13.8/3.9$, 1H); 3.61 (br. t, 2H); 3.98 (br. t, 2H); 4.58 (dd, $J = 10.2/3.9$, 1H); 6.83 (t, $J = 7.2$, 1H); 6.93 (*d*, $J = 7.8$, 2H), 7.17–7.32 (m, 7H). Anal. requires for $C_{20}H_{21}N_3OS$ (351.47) calcd./found: C 68.35/68.12; H 6.02/5.97; N 11.96/11.85.

5-Benzyl-2-(4-(3-methylphenyl)-1-piperazinyl)-1,3-thiazol-4(5H)-one (5b)

m.p. 128–129°C (ethanol) in 39% yield. ^{1}H NMR ppm: δ 2.29 (s, 3H); 2.89 (br. dd, 1H); 3.22 (br. s, 4H); 3.50 (br. dd, 1H); 3.60 (br. t, 2H); 3.97 (br. t, 2H); 4.58 (br. dd, 1H); 6.60–6.80 (m, 3H); 7.09 (t, 1H); 7.15–7.36 (m, 5H). Anal. requires for $C_{21}H_{23}N_3OS$ (365.50) calcd./found: C 69.01/68.88; H 6.34/6.23; N 11.50/11.42.

5-Benzyl-2-(4-(4-methylphenyl)-1-piperazinyl)-1,3-thiazol-4(5H)-one (5c)

m.p. 115–116°C (pentane-benzene, 2:1) in 35% yield. ^{1}H NMR ppm: δ 2.25 (s, 3H); 2.89 (dd, $J = 14.4/13.8$, 1H); 3.16 (br. *s*, 4H); 3.48 (dd, $J = 13.8/4.0$, 1H); 3.60 (br. t, 2H); 3.97 (br. t, 2H); 4.57 (dd, $J = 10.8/4.5$, 1H); 6.83 (d, $J = 8.7$, 2H); 7.03 (d, $J = 7.8$, 2H); 7.18–7.34 (m, 5H). Anal. requires for $C_{21}H_{23}N_3OS$ (365.50) Calcd./Found: C 69.01/68.82; H 6.34/6.29; N 11.50/11.55.

5-(3-Methylbenzyl)-2-(4-phenyl-1-piperazinyl)-1,3-thiazol-4(5H)-one (5d)

m.p. 173–174°C (cyclohexane-benzene, 1:4) in 42% yield. ^{1}H NMR ppm: δ 2.30 (s, 3H); 2.83 (dd, $J = 13.9/10.4$, 1H); 3.43 (dd, $J = 13.9/3.5$, 1H); 3.18–3.29 (m, 4H); 3.61 (br. s, 2H); 3.96 (br. s, 2H); 4.61 (dd, $J = 10.4/3.5$ 1H); 6.83 (t, $J = 6.9$, 1H); 6.95 (d, $J = 8.1$, 2H); 7.03 (t, $J = 9.2$, 2H); 7.07 (s, 1H); 7.17 (pseudo t, 1H); 7.20–7.26 (m, 2H). MS (m/z) (%): 365 (4, M^+), 119 (25, [$C_6H_5NCH_2CH_2$]), 105 (39, [3-$CH_3C_6H_4CH_2$]), 91 (13, [C_6H_5N]), 77 (24, [C_6H_5]). Anal. requires for $C_{21}H_{23}N_3OS$ (365.50) Calcd./Found: C 69.01/68.90; H 6.34/6.25; N 11.50/11.61.

5-(4-Methylbenzyl)-2-(4-phenyl-1-piperazinyl)-1,3-thiazol-4(5H)-one (5e)

m.p. 182–183°C (cyclohexane-benzene, 1:1) in 58% yield. ^{1}H NMR (DMSO-d_6, 500 MHz) ppm: δ 2.25 (s, 3H); 2.86 (dd, $J = 13.9/10.4$, 1H); 3.40 (dd, $J = 3.5/13.9$, 1H); 3.17–3.29 (m, 4H); 3.59 (br. t, 2H); 3.91–3.98 (m, 2H); 4.60 (dd, $J = 10.4/3.5$, 1H); 6.83 (t, $J = 6.9$, 1H); 6.94 (d, $J = 8.1$, 2H); 7.08 (d, $J = 6.9$, 2H); 7.13 (d, $J = 6.9$, 2H); 7.23 (t, $J = 8.1$, 2H). MS (m/z) (%): 365 (4, M^+), 119 (24, [$C_6H_5NCH_2CH_2$]), 105 (42, [4-$CH_3C_6H_4CH_2$]), 91 (13, [C_6H_5N]), 77 (23, [C_6H_5]). Anal. requires for $C_{21}H_{23}N_3OS$ (365.50) Calcd./Found: C 69.01/69.12; H 6.34/6.40, N 11.50/11.38.

5-(4-Methylbenzyl)-2-(4-(2-methylphenyl)-1-piperazinyl)-1,3-thiazol-4(5H)-one (5f)

M.p. 144–145°C (acetone-hexane, 4:1) in 41% yield. ^{1}H NMR ppm: δ 2.29 (s, 6H); 2.82–2.96 (m, 5H); 3.40 (dd, $J = 14.1/3.8$, 1H); 3.60 (br. t, 2H); 3.90–4.00 (m, 2H); 4.61 (dd, $J = 3.8/10.4$, 1H); 6.95–7.02 (m, 2H); 7.07–7.19 (m, 6H). MS (m/z) (%): 379 (2, M^+), 146 (100, [4-$CH_3C_6H_4CH_2CHCO$]), 133 (33, [2-$CH_3C_6H_4NCH_2CH_2$]), 118 (38, [4-$CH_3C_6H_4CH_2CH$]), 105 (24, [2-$CH_3C_6H_4N$]), 91 (19, [2-$CH_3C_6H_4$]). Anal. requires for $C_{22}H_{25}N_3OS$ (379.53) Calcd./Found: C 69.62/69.49; H 6.64/6.60; N 11.07/11.00.

5-(4-Methylbenzyl)-2-(4-(3-methylphenyl)-1-piperazinyl)-1,3-thiazol-4(5H)-one (5g)

m.p. 160–161°C (cyclohexane-benzene, 2:1) in 57% yield. ^{1}H NMR ppm: δ 2.26 (s, 3H); 2.28 (*s*, 3H); 2.86 (dd, $J = 14.0/10.4$, 1H); 3.16–3.26 (m, 4H); 3.40 (dd, $J = 3.5/14.0$, 1H); 3.58 (br. t, 2H); 3.93 (br. s, 2H); 4.60 (dd, $J = 3.5/10.4$, 1H); 6.65 (d, $J = 8.1$, 1H); 6.73 (d, $J = 8.1$, 1H); 6.76 (s, 1H); 7.06–7.16 (m, 5H). MS (m/z) (%): 379 (4, M^+), 146 (100, [4-$CH_3C_6H_4CH_2CHCO$]), 133 (31, [3-$CH_3C_6H_4NCH_2CH_2$]), 118 (25, [4-$CH_3C_6H_4CH_2CH$]), 105 (29, [3-$CH_3C_6H_4N$]), 91 (26, [3-$CH_3C_6H_4$]). Anal. requires for $C_{22}H_{25}N_3OS$ (379.53) Calcd./Found: C 69.62/69.51; H 6.64/6.52; N 11.07/11.10.

5-(4-Methylbenzyl)-2-(4-(4-methylphenyl)-1-piperazinyl)-1,3-thiazol-4(5H)-one (5h)

m.p. 167–168°C (hexane-benzene, 1:2) in 55% yield. ^{1}H NMR ppm: δ 2.23 (s, 3H); 2.26 (s, 3H); 2.85 (dd, $J = 14.2/9.8$, 1H); 3.40 (dd, $J = 14.2/3.7$, 1H); 3.05–3.15 (m, 4H); 3.57 (br. t, 2H); 3.93 (br. s, 2H); 4.60 (dd, $J = 9.8/3.7$, 1H); 6.84 (d, $J = 8.1$, 1H); 7.04 (d, $J = 8.1$, 1H); 7.08 (d, $J = 8.1$, 2H); 7.13 (d, $J = 8.1$, 2H). MS (m/z) (%): 379 (6, M^+), 146 (100, [4-$CH_3C_6H_4CH_2CHCO$]), 133 (32, [4-$CH_3C_6H_4NCH_2CH_2$]), 118 (27, [4-$CH_3C_6H_4CH_2CH$]), 105 (30, [4-$CH_3C_6H_4N$]), 91 (27, [4-$CH_3C_6H_4$]). Anal. requires for $C_{22}H_{25}N_3OS$ (379.53) Calcd./Found: C 69.62/69.38, H 6.64/6.58, N 11.07/10.96.

5-(3-Chlorobenzyl)-2-(4-phenyl-1-piperazinyl)-1,3-thiazol-4(5H)-one (5i)

m.p. 165–166°C (acetone-hexane, 4:1) in 35% yield. ^{1}H NMR ppm: δ 2.95 (dd, $J = 14.2/9.3$, 1H); 3.44 (dd, $J = 14.2/4.6$, 1H); 3.19–3.29 (m, 4H);

3.61 (br. t, 2H); 3.95 (br. s, 2H); 4.70 (dd, $J = 4.6/9.3$, 1H); 6.83 (pseudo t, 1H); 6.95 (d, $J = 8.1$, 2H); 7.20–7.27 (m, 4H); 7.32–7.34 (m, 2H). MS (m/z) (%): 385 (2, M^+), 119 (30, [$C_6H_5NCH_2CH_2$]), 91 (12, [C_6H_5N]), 77 (24, [C_6H_5]). Anal. requires for $C_{20}H_{20}ClN_3OS$ (385.92) Calcd./Found: C 62.25/62.07; H 5.22/5.18; N 10.89/10.79.

5-(3-Chlorobenzyl)-2-(4-(4-methylphenyl)-1-piperazinyl)-1,3-thiazol-4(5H)-one (5j)

m.p. 121–122°C (propanol-2) in 30% yield. ^{1}H NMR ppm: δ 2.25 (s, 3H); 2.93 (br. dd, 1H); 3.17 (br. s, 4H); 3.45 (br. dd, 1H); 3.61 (br. s, 2H); 3.96 (br. s, 2H); 4.62 (br. dd, 1H); 6.83 (d, $J = 7.2$, 2H); 7.03 (d, $J = 7.2$, 2H); 7.17–7.34 (m, 4H). MS (m/z) (%) : 400 (5, M^+), 133 (27, [4-$CH_3C_6H_4NCH_2CH_2$]), 105 (5, [4-$CH_3C_6H_4N$]), 91 (20, [4-$CH_3C_6H_4$]), 42 (4, [NC(O)]). Anal. requires for $C_{21}H_{22}ClN_3OS$ (399.95) Calcd./Found: C 63.07/63.15; H 5.54/5.47, N 10.51/10.38.

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