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Synthesis of Substituted Thiazol-2-yl-acetamides. Molecular and Crystal Structure of *N*-(4-Methoxyphenyl)-2-oxo-2-(4-phenylthiazol-2-yl)acetamide

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Abstract—Reaction of 3-amino-3-thioxopropanamides with α-bromoketones has afforded substituted thiazol-2-yl-acetamides. Structure of *N*-(4-methoxyphenyl)-2-oxo-2-(4-phenylthiazol-2-yl)acetamide has been studied by X-ray diffraction.

Keywords: 3-amino-3-thioxopropanamide, α-bromoketones, thiazol-2-yl-acetamides, XRD

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3-Amino-3-thioxopropanamides are used in organic synthesis as CH-acids in preparation of functionally substituted pyridines via the Michael reaction [1–4] as well as *C*-nucleophiles in nucleophilic vinyl substitution [5, 6] and condensation with carbonyl compounds [7, 8]. Alkylation of 3-amino-3-thioxopropanamides results in *S*-methyl derivatives [9].

Extending our earlier studies on transformation of 3-amino-3-thioxopropanamides [10–13], herein we report on reactions of amides **Ia** and **Ib** with α-bromoketones **IIa**–**IIf** at 20°C in DMF.

The reaction led to formation of the substituted Hantzsch thiazoles **IIIa**–**IIIf**, promising precursors for preparation of antimicrobial [14, 15] and antitumor [16, 17] drugs. Some of the Hantzsch thiazoles have demonstrated substantial efficiency in treatment of Alzheimer's disease [18] and as a fluorescent probe [19] (Scheme 1).

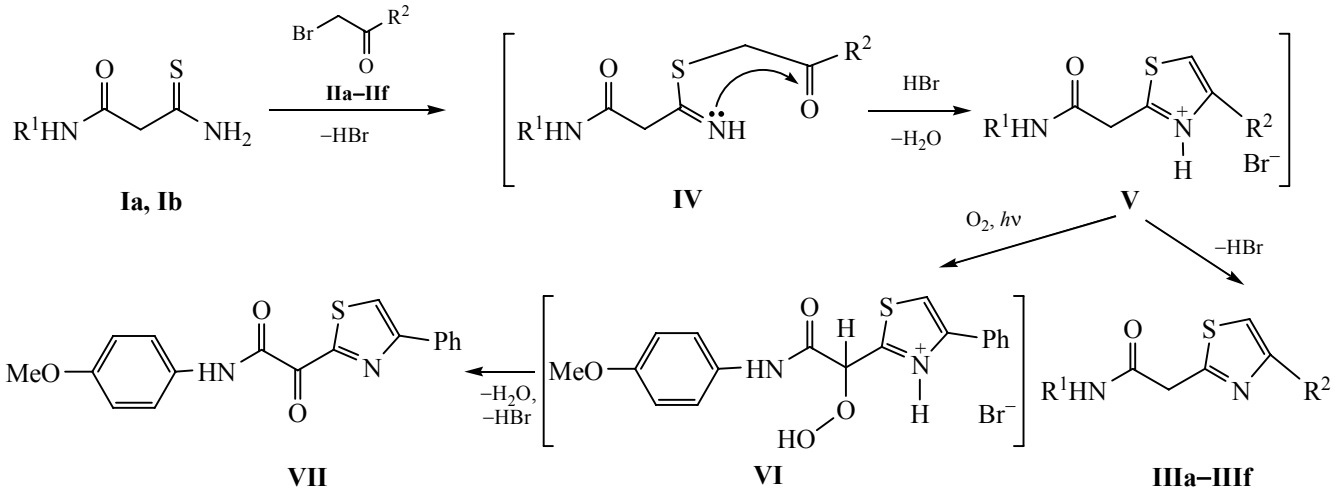
Apparently, the discussed reaction proceeded via formation of intermediate **IV** followed by intramolecular cyclization into the substituted thiazolium bromide **V**. The latter was hydrolyzed to yield thiazole **III**. In the case of thioamide **Ia** and phenacyl bromide **IIa**, the reaction did not stop at the stage of formation of thiazole **III**. The presence of π-deficient thiazolium cation near the methylene group in the molecule of intermediate **V** promoted its oxidation [20] with air oxygen upon exposition of the solution to light, leading to peroxide **VI**. Peroxide **VI** was further transformed into *N*-(4-methoxyphenyl)-2-oxo-2-(4-phenylthiazol-2-yl)acetamide **VII** via elimination of water and hydrogen bromide.

Structures of compounds **IIIa**–**IIIf** and **IV** were confirmed by the spectral data. In particular, the IR spectra contained characteristic absorption bands of carbonyl group stretching at 1658–1714 cm^{-1}. A singlet signal of the methylene protons at 3.91–4.41 ppm was observed in ^{1}H NMR spectra of compounds **IIIa**–**IIIf**. Mass spectrum of **IIIf** contained a weak peak of $[M + 2]^+$ confirming the presence of sulfur atoms in the molecule [21]. The molecular ion peak with even mass number pointed at an even number of nitrogen atoms in the molecule of compound **IV** [22]. ^{13}C NMR spectrum of **VII** contained the signals of all the carbon atoms of the suggested structure with the expected chemical shifts.

Structure of compound **VII** was studied by single crystal X-ray diffraction (Figs. 1–3, Tables 1 and 2).

Independent part of the unit cell contained two molecules of compound **VII** with similar geometry parameters. Oxoacetamide fragment was located in the plane of the thiazole ring (the angle between the average planes was of 7.1° in the molecule **A** and of 6.6° in the molecule **B**). The methoxyphenyl moiety was almost coplanar with the oxoacetamide fragment [the $C^5N^2C^6C^7$ torsion angles were of –23.4(3)° (**A**)

Scheme 1.



I, R^1 = 4-$MeOC_6H_4$ (**a**), H (**b**); **II**, R^2 = Ph (**a**), 4-$MeOC_6H_4$ (**b**), coumarin-3-yl (**c**), 4-MeC_6H_4 (**d**), 3-oxo-3*H*-benzo[*f*]-chromen-2-yl (**e**), 4-$NO_2C_6H_4$ (**f**); **III**, $R^1 = R^2$ = 4-$MeOC_6H_4$ (**a**); R^1 = 4-$MeOC_6H_4$, R^2 = 3-oxo-3*H*-benzo[*f*]chromen-2-yl (**b**); R^1 = 4-$MeOC_6H_4$, R^2 = coumarin-3-yl (**c**); R^1 = 4-$MeOC_6H_4$, R^2 = 4-$NO_2C_6H_4$ (**d**); R^1 = H, R^2 = 4-MeC_6H_4 (**e**); R^1 = H, R^2 = coumarin-3-yl (**f**).

and of –13.8(3)° (**B**)], and the C^7–H^7···O^2 intramolecular hydrogen bond [H···O 2.30Å (**A**) and 2.28 Å (**B**), C–H···O 117° (**A**) and 121° (**B**)] was formed. The phenyl substituent of molecule **B** was located almost in the thiazole ring plane [the $C^1C^2C^{12}C^{17}$ torsion angle of 7.3(3)°] resulting in a shortened intermolecular contact H^1···H^{17} (2.27 Å with the sum of the van der Waals radii being of 2.32 Å [23]). The similar shortened contact was not formed in the molecule **A** due to a small rotation of the phenyl substituent with respect to the thiazole ring [the $C^1C^2C^{12}C^{17}$ torsion angle of 19.2(3)°].

The molecules were linked into the **A**···**B** dimers via intermolecular hydrogen bonding N^{2A}–H^{2A}···O^{1B} (H···O 2.16 Å, N–H···O 161°) and N^{2B}–H^{2B}···O^{1A} (H···O 2.30 Å, N–H···O 164°) in the crystal (Fig. 2). Those dimers were linked into chains along the *c* axis of the lattice due to intermolecular contacts O^{2A}···S^{1B} 3.16 Å and O^{2B}···S^{1A} 3.08 Å between the molecules **A** and **B** (the sum of the van der Waals radii being of 3.11 Å) .

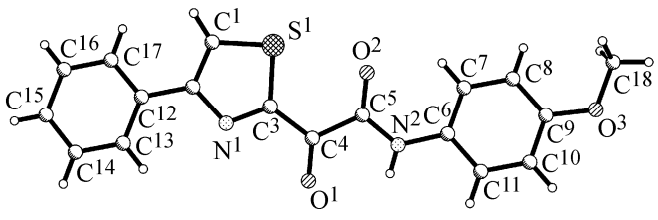


Fig. 1. General view of the molecule of compound **VII**.

Apparently, the S···O intermolecular interactions could be described as σ-hole bonds, as evidenced by the proximity of the C^3–S^1···O^2 angle (147°–162°) to 180°. To confirm the assumption, we performed quantum-chemical simulation of electrostatic potential of the molecules **VII** dimer (Fig. 3). The region of the positive electrostatic potential at continuation of the C^3–S^1 bond was oriented towards the area of the negative electrostatic potential of the O^2 atom of the adjacent molecule. On top of that, the molecule contained the S^1···O^2 intramolecular σ-hole bonding [2.68Å (**A**) and 2.72 Å (**B**)] between the region of positive electrostatic potential at continuation of the C^1–S^1 bond and the region of negative potential of the lone-electron pair of the oxygen atom. Existence of such intramolecular S···O bonds has been shown earlier [24].

EXPERIMENTAL

IR spectra of KBr pellets were recorded with a UR-20 spectrophotometer. ^1H and ^{13}C NMR spectra of solutions in DMSO-d_6 were recorded with a Varian-Gemini spectrometer (400.13 and 100 MHz, respectively) relative to TMS as internal reference. Elemental analysis was performed using an EuroVector EA–3000 microanalyzer. Chromato-mass spectroscopy studies were performed using an Agilent 1100/DAD/HSD/VLG 119562 and MX-1321 (70 eV) devices. Melting points were determined using a

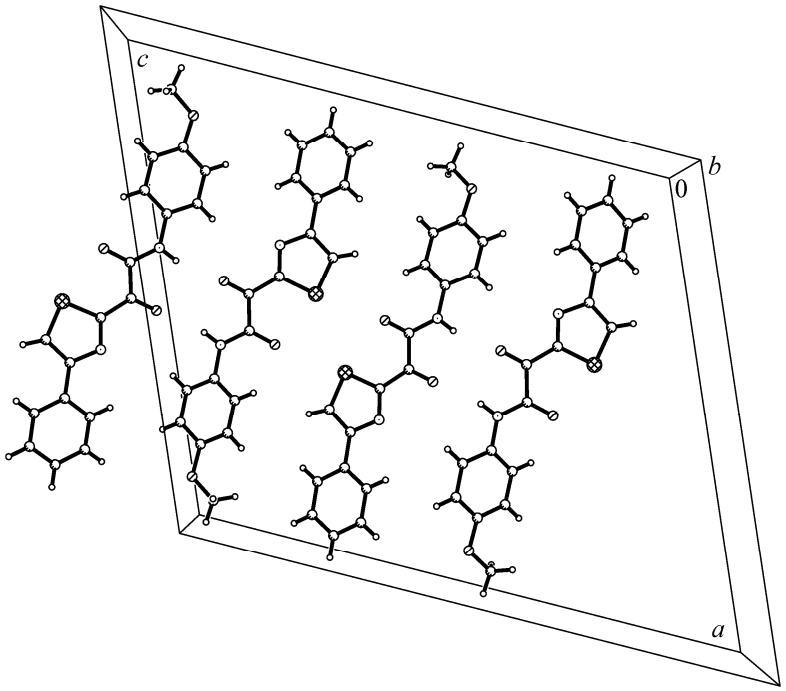


Fig. 2. Crystal packing of the molecule of compound **VII**.

Kofler bench. The reaction progress was monitored by TLC on Silufol UV-254 plates, eluting with acetone–hexane mixture (3 : 5) and detecting with iodine vapors or UV irradiation

Single crystal X-ray diffraction. Crystals of compound **VII** were monoclinic, $C_{18}H_{14}N_2O_3S$; the unit cell parameters at 298 K: a = 20.1357(11), b = 7.1124(6), c = 23.4523(14) Å, β = 112.893(7)°, V = 3094.1(4) Å^3, M = 338.37, Z = 2, space group $P2_1/c$, d_{calc} = 1.45 g cm^{-3}, μ(MoK_α) = 0.229 mm^{-1}, F(000) = 1408. The unit cell parameters and intensities of the 25420 reflections (9784 independent ones, R_{int} = 0.044) were measured with an Xcalibur automatic four-circle diffractometer (MoK_α, graphitic monochromator, CCD-detector, ω-scan, $2\theta_{max}$ = 64.02°).

The structure was solved via the direct method using SHELX-97 software [25]. The hydrogen atoms were geometrically positioned and refined using a *rider* model with $U_{iso} = nU_{eq}$ (n = 1.5 for the methyl group and n = 1.2 for other of hydrogen atoms). The structure was refined via F^2 full-matrix least-squares method using anisotropic approximation for non-hydrogen atoms to wR_2 0.143 over 9784 reflections [R_1 = 0.053 over 5512 reflections with $F > 4\sigma(F)$, S = 1.01]. The bond lengths and angles are listed in Tables 1 and 2, respectively.

3-Amino-*N*-(4-methoxyphenyl)-3-thioxopropanamide (Ia) was prepared as described in Ref. [8]. Lilac plate crystals, sublimation point 138–139°C (mp 143–144°C [8]). ^{1}H NMR spectrum, δ, ppm: 3.64 s (2H, CH_2), 3.72 s (3H, Me), 6.84 d (2H, H_{arom}, J 8.0 Hz), 7.51 d (2H, H_{arom}, J 8.0 Hz), 9.36 br.s (1H, NH_2), 9.64 br.s (1H, NH_2), 9.97 br.s (1H, NH).

3-Amino-3-thioxopropanamide (Ib) was prepared as described in Ref. [8]. Yellow crystals, mp 105–107°C (mp 103–105°C [8]). ^{1}H NMR spectrum, δ, ppm: 3.42

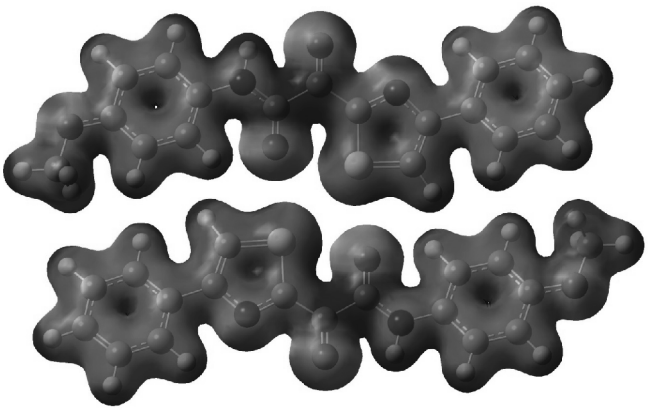


Fig. 3. Simulated electrostatic potential map of molecules **VII** dimer linked via σ-hole O···S bonds (experimental geometry, B3LYP/TZVP, isosurface of electron density value of 0.025 a. u.).

Table 1. Bond lengths (Å) in the molecule of compound **VII**

Bond	**A**	**B**	Bond	**A**	**B**
S^1–C^1	1.6778(19)	1.6788(19)	C^4–C^5	1.535(2)	1.534(2)
S^1–C^3	1.7120(16)	1.7162(16)	C^6–C^7	1.384(2)	1.388(2)
O^1–C^4	1.2124(19)	1.2074(19)	C^6–C^{11}	1.388(2)	1.388(2)
O^2–C^5	1.2147(19)	1.220(2)	C^7–C^8	1.386(2)	1.384(2)
O^3–C^9	1.3755(19)	1.370(2)	C^8–C^9	1.375(2)	1.377(3)
O^3–C^{18}	1.417(2)	1.413(2)	C^9–C^{10}	1.384(2)	1.384(2)
N^1–C^2	1.368(2)	1.358(2)	C^{10}–C^{11}	1.376(2)	1.375(2)
N^1–C^3	1.316(2)	1.315(2)	C^{12}–C^{13}	1.383(2)	1.381(2)
N^2–C^5	1.334(2)	1.334(2)	C^{12}–C^{17}	1.389(2)	1.391(3)
N^2–C^6	1.416(2)	1.418(2)	C^{13}–C^{14}	1.383(2)	1.381(3)
C^1–C^2	1.371(2)	1.370(2)	C^{14}–C^{15}	1.379(3)	1.368(3)
C^2–C^{12}	1.474(2)	1.475(2)	C^{15}–C^{16}	1.374(3)	1.376(3)
C^3–C^4	1.471(2)	1.468(2)	C^{16}–C^{17}	1.375(2)	1.377(3)

Table 2. Bond angles (deg) in the molecule of compound **VII**

Angle	**A**	**B**	Angle	**A**	**B**
$C^1S^1C^3$	89.27(8)	89.23(9)	$C^7C^6C^{11}$	119.37(15)	119.21(16)
$C^9O^3C^{18}$	116.91(15)	117.49(16)	$C^{11}C^6N^2$	118.26(15)	117.91(15)
$C^3N^1C^2$	110.33(13)	110.80(14)	$C^6C^7C^8$	120.11(16)	120.14(17)
$C^5N^2C^6$	126.04(14)	127.81(14)	$C^9C^8C^7$	120.28(16)	120.33(17)
$C^2C^1S^1$	111.53(13)	111.41(14)	$O^3C^9C^{10}$	115.86(15)	115.20(16)
$N^1C^2C^1$	113.95(15)	114.09(15)	$C^8C^9O^3$	124.45(16)	125.33(16)
$N^1C^2C^{12}$	120.00(14)	119.60(15)	$C^8C^9C^{10}$	119.69(16)	119.47(16)
$C^1C^2C^{12}$	126.04(16)	126.31(16)	$C^{11}C^{10}C^9$	120.34(16)	120.56(17)
$N^1C^3S^1$	114.91(13)	114.46(13)	$C^{10}C^{11}C^6$	120.17(16)	120.19(16)
$N^1C^3C^4$	120.64(14)	121.07(14)	$C^{13}C^{12}C^2$	120.75(16)	120.25(16)
$C^4C^3S^1$	124.42(13)	124.47(13)	$C^{13}C^{12}C^{17}$	118.70(16)	118.32(17)
$O^1C^4C^3$	122.63(15)	122.01(15)	$C^{17}C^{12}C^2$	120.55(16)	121.42(17)
$O^1C^4C^5$	120.08(15)	119.92(15)	$C^{12}C^{13}C^{14}$	120.44(18)	120.58(19)
$C^3C^4C^5$	117.29(14)	118.07(15)	$C^{15}C^{14}C^{13}$	120.27(19)	120.8(2)
$O^2C^5N^2$	125.74(16)	125.84(16)	$C^{16}C^{15}C^{14}$	119.52(19)	119.23(19)
$O^2C^5C^4$	120.17(15)	120.87(15)	$C^{15}C^{16}C^{17}$	120.45(19)	120.5(2)
$N^2C^5C^4$	114.09(14)	113.29(15)	$C^{16}C^{17}C^{12}$	120.63(18)	120.62(19)
$C^7C^6N^2$	122.37(15)	122.87(15)			

s (2H, CH_2), 6.96 br.s (1H, $CONH_2$), 7.41 br.s (1H, $CONH_2$), 9.29 br.s (1H, $CSNH_2$), 9.43 br.s (1H, $CSNH_2$).

Substituted thiazol-2-ylacetamides (IIIa–IIIe) and *N*-(4-methoxyphenyl)-2-oxo-2-(4-phenylthiazol-2-yl)acetamide (VII) (*general procedure*). 10 mmol of the corresponding α-bromoketone **III** was added to a stirred solution of 10 mmol of thioxopropanamide **Ia** or **Ib** in 20 mL of DMF at 20°C. The mixture was stirred for 2 h and then incubated during 48 h. The reaction mixture was diluted with equal volume of water and incubated during 4 days. The resulting precipitate was filtered off and sequentially washed with water, ethanol, and hexane.

***N*-(4-Methoxyphenyl)-2-[4-(4-methoxyphenyl)-thiazol-2-yl]acetamide (IIIa).** Yield 2.9 g (82%), yellow powder, mp 151–152°C (PrOH). IR spectrum, ν, cm^{-1}: 3300 (NH), 1672 (CONH). ^1H NMR spectrum, δ, ppm: 3.74 s (3H, MeO), 3.84 s (3H, MeO), 4.08 s (2H, CH_2), 6.77 d (2H, H_{arom}, *J* 8.5 Hz), 6.88 d (2H, H_{arom}, *J* 8.4 Hz),7.45 s (1H, C^5H, thiazole), 7.51 d

(2H, H_{arom}, *J* 8.5 Hz), 7.79 d (2H, H_{arom}, *J* 8.4 Hz), 10.01 br.s (1H, NH). Mass spectrum, *m/z* (I_{rel},%): 355 (100) $[M + 1]^+$. Found, %: C 64.25; H 5.01; N 7.84. $C_{19}H_{18}N_2O_3S$. Calculated, %: C 64.39; H 5.12; N 7.90. *M* 354.425.

***N*-(4-Methoxyphenyl)-2-[4-(3-oxo-3*H*-benzo[*f*]-chromen-2-yl)thiazol-2-yl]acetamide (IIIb).** Yield 2.7 g (62%), yellow powder, mp 216–217°C (DMF), sublimation point 170°C. IR spectrum, ν, cm^{-1}: 3314 (NH), 1711 (C=O), 1666 (CONH). ^1H NMR spectrum, δ, ppm: 3.76 s (3H, Me), 4.18 s (2H, CH_2), 7.82 d (2H, H_{arom}, *J* 8.6 Hz), 7.45–7.61 m (3H, H_{arom}), 7.73 t (1H, H_{arom}, *J* 8.1 Hz), 7.96 d (2H, H_{arom}, *J* 8.6 Hz), 8.07 d (1H, H_{arom}, *J* 8.3 Hz), 8.42 s (1H, C^5H, thiazole), 8.46 d (1H, H_{arom}, *J* 8.4 Hz), 9.49 s (1H, C^1H, chromen), 10.06 br.s (1H, NH). Mass spectrum, *m/z* (I_{rel},%): 443 (100) $[M + 1]^+$. Found, %: C 67.74; H 3.98; N 6.28. $C_{25}H_{18}N_2O_4S$. Calculated, %: C 67.86; H 4.10; N 6.33. *M* 442.492.

***N*-(4-Methoxyphenyl)-2-[4-(2-oxo-2*H*-chromen-3-yl)thiazol-2-yl]acetamide (IIIc).** Yield 3.0 g (76%) colorless powder, mp 201–202°C (BuOH). IR spectrum, ν, cm^{-1}: 3296 (NH), 1702 (C=O), 1670 (CONH). ^1H NMR spectrum, δ, ppm: 3.72 s (3H, Me), 4.21 s (2H, CH_2), 6.91 d (2H, H_{arom}, *J* 8.7 Hz), 7.39 t (1H, C^7H, coumarin, *J* 8.1 Hz), 7.47 d (1H, C^5H, coumarin, *J* 8.1 Hz), 7.54 d (2H, H_{arom}, *J* 8.7 Hz), 7.65 t (1H, C^6H, coumarin, *J* 8.1 Hz), 7.94 d (1H, C^8H, coumarin, *J* 8.1 Hz), 8.36 s (1H, C^5H, thiazole), 8.77 s (1H, C^4H, coumarin), 10.26 br.s (1H, NH). Mass spectrum, *m/z* (I_{rel},%): 393 (100) $[M + 1]^+$. Found, %: C 64.18; H 3.95; N 7.02. $C_{21}H_{16}N_2O_4S$. Calculated, %: C 64.27; H 4.11; N 7.14. *M* 392.432.

***N*-(4-Methoxyphenyl)-2-[4-(4-nitrophenyl)thiazol-2-yl]acetamide (IIId).** Yield 2.8 g (76%), yellow powder, mp 189–190°C (AcOH). IR spectrum, ν, cm^{-1}: 3315 (NH), 1662 (CONH). ^1H NMR spectrum, δ, ppm: 4.02 s (3H, Me), 4.41 s (2H, CH_2), 7.06 d (2H, H_{arom}, *J* 8.6 Hz), 7.77 d (2H, H_{arom}, *J* 8.6 Hz), 8.38 s (1H, C^5H, thiazole), 8.44 d (2H, H_{arom}, *J* 8.3 Hz), 8.53 d (2H, H_{arom}, *J* 8.3 Hz), 10.33 br.s (1H, NH). Mass spectrum, *m/z* (I_{rel},%): 370 (100) $[M + 1]^+$. Found, %: C 58.45; H 3.92; N 11.24. $C_{18}H_{15}N_3O_4S$. Calculated, %: C 58.53; H 4.09; N 11.36. *M* 369.397.

2-[5-(*p*-Tolyl)thiazol-2-yl]acetamide (IIIe). Yield 1.7 g (75%), colorless needles, mp 131°C (EtOH). IR spectrum, ν, cm^{-1}: 3390, 3300, 3280 (NH_2), 1680 (CONH). ^1H NMR spectrum, δ, ppm: 2.35 s (3H, Me), 3.91 s (2H, CH_2), 7.11 br.s (1H, NH_2), 7.22 d (2H, H_{arom}, *J* 7.7 Hz), 7.59 br.s (1H, NH_2), 7.82 d (2H, H_{arom}, *J* 7.7 Hz), 7.85 s (1H, C^5H, thiazole). Mass spectrum, *m/z* (I_{rel}, %): 234 (4) $[M + 2]^+$, 233 (9) $[M + 1]^+$, 232 (59) $[M]^+$, 215 (2) $[M - NH_3]^+$, 189 (100) $[M - CH_3CO]^+$, 148 (35), 134 (12), 115 (11), 91 (6) $[PhCH_2]^+$, 77 (5) $[Ph]^+$, 44 (9) $[CS]^+$. Found, %: C 61.96; H 5.02; N 11.94. $C_{12}H_{12}N_2OS$. Calculated, %: C 62.05; H 5.21; N 12.06. *M* 232.303.

2-[5-(2-Oxo-2*H*-chromen-3-yl)thiazol-2-yl]acetamide (IIIf). Yield 2.3 g (82%) colorless powder, mp 211–213°C (BuOH). IR spectrum, ν, cm^{-1}: 3402, 3385, 3287 (NH_2), 1714 (C=O), 1671 (CONH). ^1H NMR spectrum, δ, ppm: 3.91 s (2H, CH_2), 7.05 br.s (1H, NH_2), 7.21–7.42 m (2H, H_{arom}, NH_2), 7.44–7.65 m (2H, H_{arom}), 7.78 d (1H, H_{arom}, *J* 7.6 Hz), 8.28 s (1H, C^5H, thiazole), 8.73 s (1H, C^4H, coumarin). Mass spectrum, *m/z* (I_{rel},%): 287 (100) $[M + 1]^+$. Found, %: C 58.64; H 3.43; N 9.66. $C_{14}H_{10}N_2O_3S$. Calculated, %: C 58.73; H 3.52; N 9.78. *M* 286.308.

***N*-(4-Methoxyphenyl)-2-oxo-2-(4-phenylthiazol-2-yl)acetamide (VII).** Yield 2.2 g (66%), yellow needles, mp 198–200°C (BuOH). IR spectrum, ν, cm^{-1}: 3322 (NH), 1658 (C=O). ^1H NMR spectrum, δ, ppm: 3.78 s (3H, Me), 6.87 d (2H, H_{arom}, *J* 8.5 Hz), 7.35 t (1H, H_{arom}, *J* 7.0 Hz), 7.44 t (2H, H_{arom}, *J* 7.5 Hz), 7.75 d (2H, H_{arom}, *J* 8.5 Hz), 8.03 d (2H, H_{arom}, *J* 7.0 Hz), 10.81 br.s (1H, NH). ^{13}C NMR spectrum, δ_C, ppm: 55.73, 114.57, 122.34, 124.98, 126.87, 129.42, 129.51, 130.97, 133.54, 156.79, 157.12, 159.38, 160.48, 178.84. Mass spectrum, *m/z* (I_{rel}, %): 339 (100) $[M + 1]^+$. Found, %: C 63.78; H 4.00; N 8.11. $C_{18}H_{14}N_2O_3S$. Calculated, %: C 63.89; H 4.17; N 8.28. *M* 338.384.

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