

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–IIc** at 20°C in DMF.

The reaction led to formation of the substituted Hantzsch thiazoles **IIIa–IIIc**, 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,

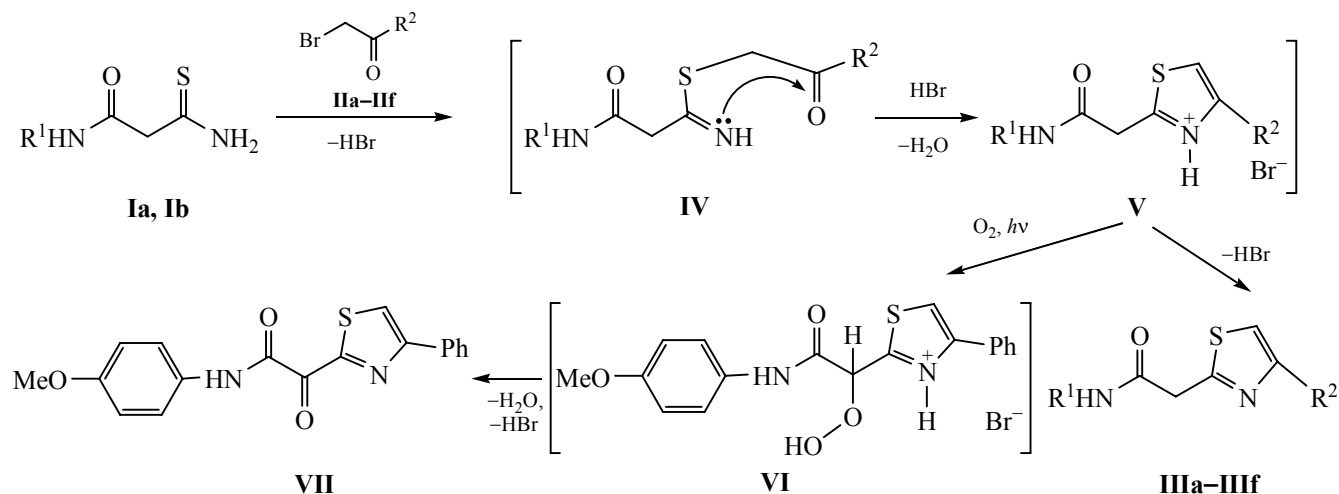
leading to peroxide **VI**. Peroxide **VI** was further transformed into *N*-(4-methoxyphenyl)-2-oxo-2-(4-phenylthiazol-2-yl)acetamide **VII** via elimination of water and hydrogen bromide.

Structures of compounds **IIIa–IIIc** 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 ¹H NMR spectra of compounds **IIIa–IIIc**. Mass spectrum of **IIIc** 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]. ¹³C 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 C⁵N²C⁶C⁷ torsion angles were of −23.4(3)° (**A**)

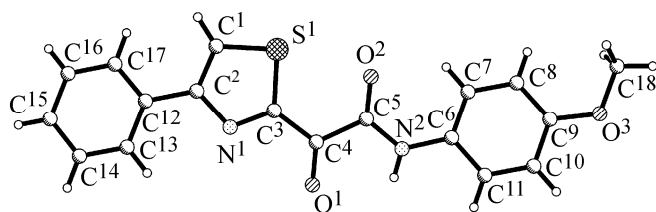
Scheme 1.



I, $R^1 = 4\text{-MeOC}_6\text{H}_4$ (**a**), H (**b**); **II**, $R^2 = \text{Ph}$ (**a**), $4\text{-MeOC}_6\text{H}_4$ (**b**), coumarin-3-yl (**c**), $4\text{-MeC}_6\text{H}_4$ (**d**), 3-oxo-3*H*-benzo[*f*]-chromen-2-yl (**e**), $4\text{-NO}_2\text{C}_6\text{H}_4$ (**f**); **III**, $R^1 = R^2 = 4\text{-MeOC}_6\text{H}_4$ (**a**); $R^1 = 4\text{-MeOC}_6\text{H}_4$, $R^2 = 3\text{-oxo-3H-benzo}[f]\text{chromen-2-yl}$ (**b**); $R^1 = 4\text{-MeOC}_6\text{H}_4$, $R^2 = \text{coumarin-3-yl}$ (**c**); $R^1 = 4\text{-MeOC}_6\text{H}_4$, $R^2 = 4\text{-NO}_2\text{C}_6\text{H}_4$ (**d**); $R^1 = H$, $R^2 = 4\text{-MeC}_6\text{H}_4$ (**e**); $R^1 = H$, $R^2 = \text{coumarin-3-yl}$ (**f**).

and of $-13.8(3)^\circ$ (**B**)], and the $C^7\text{---}H^7\cdots O^2$ intramolecular hydrogen bond [$H\cdots O$ 2.30 Å (**A**) and 2.28 Å (**B**), $C\text{---}H\cdots O$ 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 $7.3(3)^\circ$] resulting in a shortened intermolecular contact $H^{17}\cdots H^{17}$ (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 $19.2(3)^\circ$].

The molecules were linked into the **A**...**B** dimers via intermolecular hydrogen bonding $N^{2A}\cdots H^{2A}\cdots O^{1B}$ ($H\cdots O$ 2.16 Å, $N\text{---}H\cdots O$ 161°) and $N^{2B}\cdots H^{2B}\cdots O^{1A}$ ($H\cdots O$ 2.30 Å, $N\text{---}H\cdots O$ 164°) in the crystal (Fig. 2). Those dimers were linked into chains along the *c* axis of the lattice due to intermolecular contacts $O^{2A}\cdots S^{1B}$ 3.16 Å and $O^{2B}\cdots S^{1A}$ 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 $S\cdots O$ intermolecular interactions could be described as σ -hole bonds, as evidenced by the proximity of the $C^3\text{---}S^1\cdots O^2$ angle ($147^\circ\text{--}162^\circ$) 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 $C^3\text{---}S^1$ 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 $S^1\cdots O^2$ intramolecular σ -hole bonding [2.68 Å (**A**) and 2.72 Å (**B**)] between the region of positive electrostatic potential at continuation of the $C^1\text{---}S^1$ bond and the region of negative potential of the lone-electron pair of the oxygen atom. Existence of such intramolecular $S\cdots O$ 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

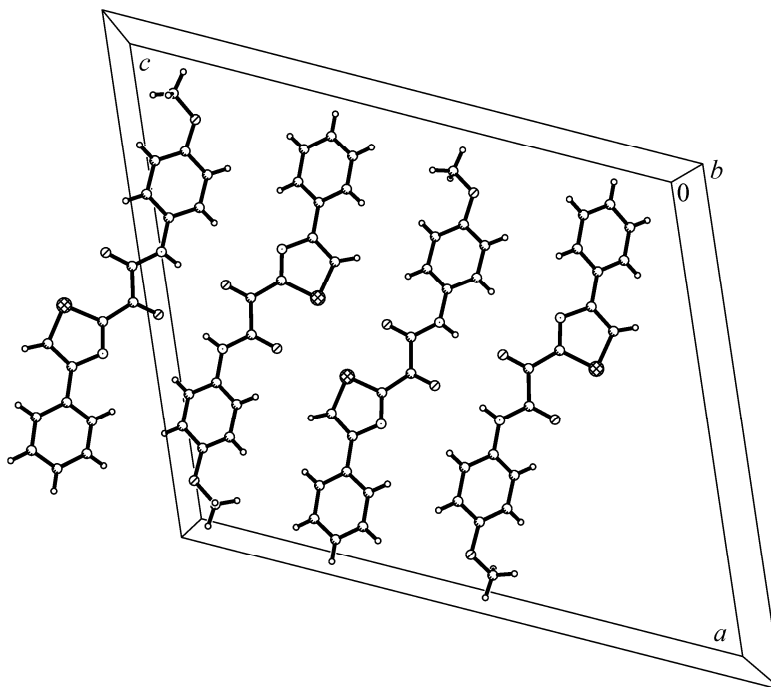


Fig. 2. Crystal packing of the molecule of compound VII.

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, $\beta = 112.893(7)^\circ$, $V = 3094.1(4)$ Å³, $M = 338.37$, $Z = 2$, space group $P2_1/c$, $d_{\text{calc}} = 1.45$ g cm⁻³, $\mu(\text{MoK}\alpha) = 0.229$ mm⁻¹, $F(000) = 1408$. The unit cell parameters and intensities of the 25420 reflections (9784 independent ones, $R_{\text{int}} = 0.044$) were measured with an Xcalibur automatic four-circle diffractometer (MoK α , graphitic monochromator, CCD-detector, ω -scan, $2\theta_{\text{max}} = 64.02^\circ$).

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 $U_{\text{iso}} = nU_{\text{eq}}$ ($n = 1.5$ 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]). ¹H NMR spectrum, δ , ppm: 3.64 s (2H, CH₂), 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, NH₂), 9.64 br.s (1H, NH₂), 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]). ¹H NMR spectrum, δ , ppm: 3.42

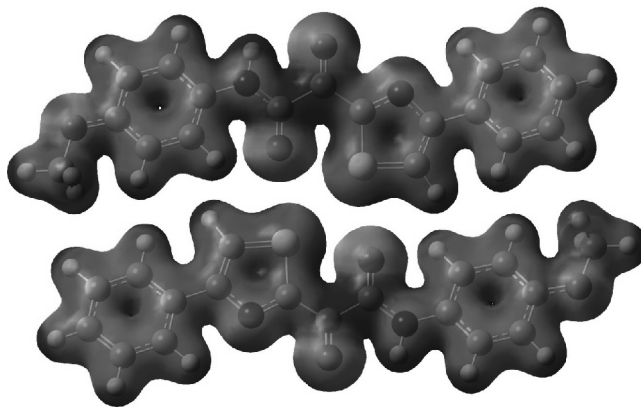


Fig. 3. Simulated electrostatic potential map of molecules VII dimer linked via σ -hole O $\cdots$ S bonds (experimental geometry, B3LYP/TZVP, isosurface of electron density value of 0.025 a. u.).

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

s (2H, CH₂), 6.96 br.s (1H, CONH₂), 7.41 br.s (1H, CONH₂), 9.29 br.s (1H, CSNH₂), 9.43 br.s (1H, CSNH₂).

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, CH₂), 6.77 d (2H, H_{arom}, *J* 8.5 Hz), 6.88 d (2H, H_{arom}, *J* 8.4 Hz), 7.45 s (1H, C⁵H, 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) $[M - \text{NH}_3]^+$, 189 (100) $[M - \text{CH}_3\text{CO}]^+$, 148 (35), 134 (12), 115 (11), 91 (6) $[\text{PhCH}_2]^+$, 77 (5) $[\text{Ph}]^+$, 44 (9) $[\text{CS}]^+$. Found, %: C 61.96; H 5.02; N 11.94. $C_{12}H_{12}N_2\text{OS}$. 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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