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Synthesis of Nitrogen Heterocycles Underlain by Application of 3-(4-Acetyl[phenyl)-2*H*-coumarin

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Abstract—3-(4-Acetylphenyl)-*2H*-chromen-2-one was obtained from 4-acetylphenyldiazonium chloride in the conditions of Meerwein reaction. Reactions of 3-[4-(2-bromoacetyl)phenyl]-*2H*-chromen-2-one with pyridine, 4-methylpyridine, quinoline, benzo[*f*]quinoline, and triphenylphosphine afforded quaternary salts, and with thioacetamide, thiourea, 2-aminopyridine, 2-aminopyrimidine, and 6-aminopurine provided the corresponding derivatives of thiazole, imidazo[1,2-*a*]pyridine, imidazo[1,2-*a*]pyrimidine, imidazo[2,1-*i*]purine. In the reaction of the same bromo derivative with thiosemicarbazide and aromatic aldehydes a thiazole ring is built and the corresponding hydrazones are formed.

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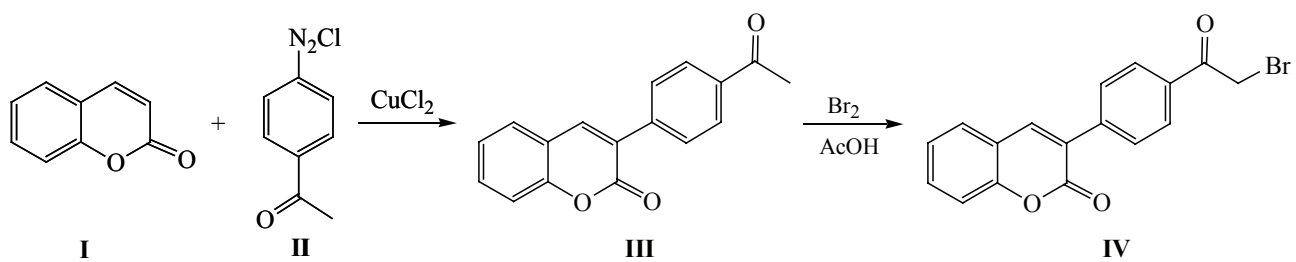
The interest in the chemistry of coumarins (2*H*-benzopyran-2-ones) is to a large extent due to the wide range of biological actions exhibited by this class compounds [1, 2], and also by their applications as laser dyes and luminescent indicators [3]. In many of these compounds the coumarin scaffold is combined with a heterocyclic or onium fragment. The synthesis of such compounds often utilizes the reactions of 3-(ω-bromo-acetyl) coumarins with nucleophilic reagents [4–7], substituted 2-hydroxybenzaldehydes with 2-hetaryl-acetonitriles [8], and also the recyclization of 2-iminocoumarins under the treatment with binucleophilic reagents [9].

In this research we used for the molecular design of coumarin derivatives the arylation of unsaturated compounds with arenediazonium salts (Meerwein reaction) [10]. Coumarin (**I**) reacts with 4-acetylphenyldiazonium chloride (**II**) in the presence of copper(II) chloride giving 3-(4-acetylphenyl)-2*H*-chromen-2-one (**III**) in 40% yield. The bromination of the latter at heating in acetic acid furnished compound **IV** (Scheme 1).

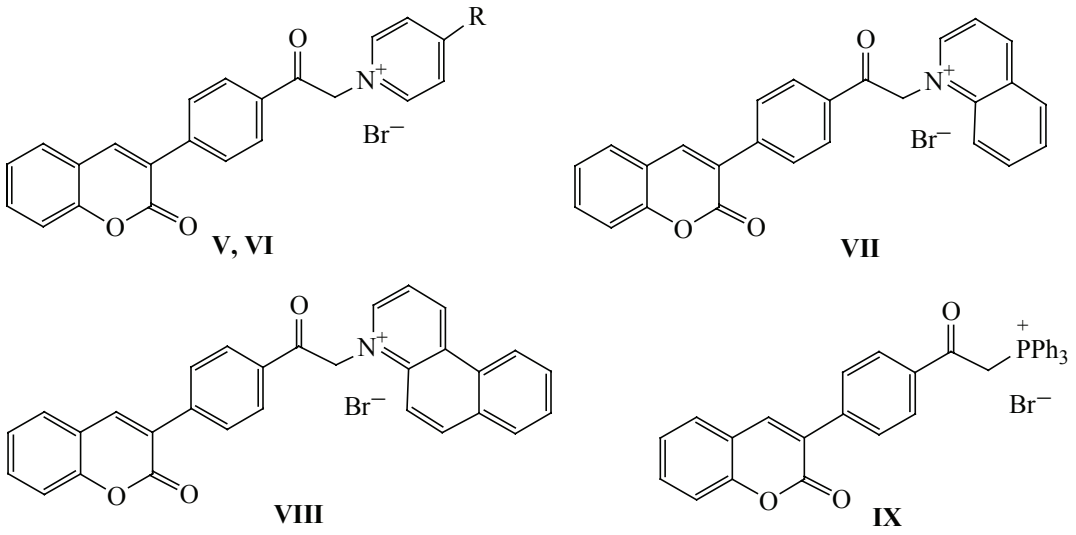
3-[4-(2-Bromoacetyl)phenyl]-2*H*-chromen-2-one (**IV**) at heating in toluene with heterocyclic bases (pyridine, 4-methylpyridine, quinoline, benzo[*f*]quinoline), and also with triphenylphosphine readily forms quaternary salts **V**–**IX** (Scheme 2).

At treating the solution of phosphonium salt **IX** in DMF with water solution of potassium carbonate triphenylphosphorylide **X** was formed which at boiling in

Scheme 1.

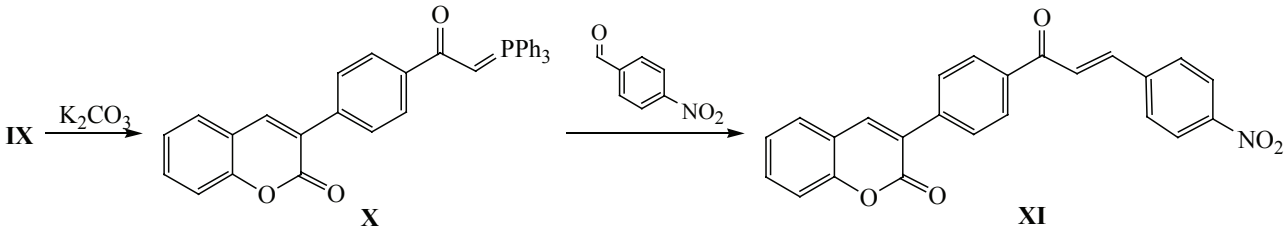


Scheme 2.



R = H (**V**), Me (**VI**).

Scheme 3.



toluene with 4-nitrobenzaldehyde afforded ketone **XI** (Scheme 3).

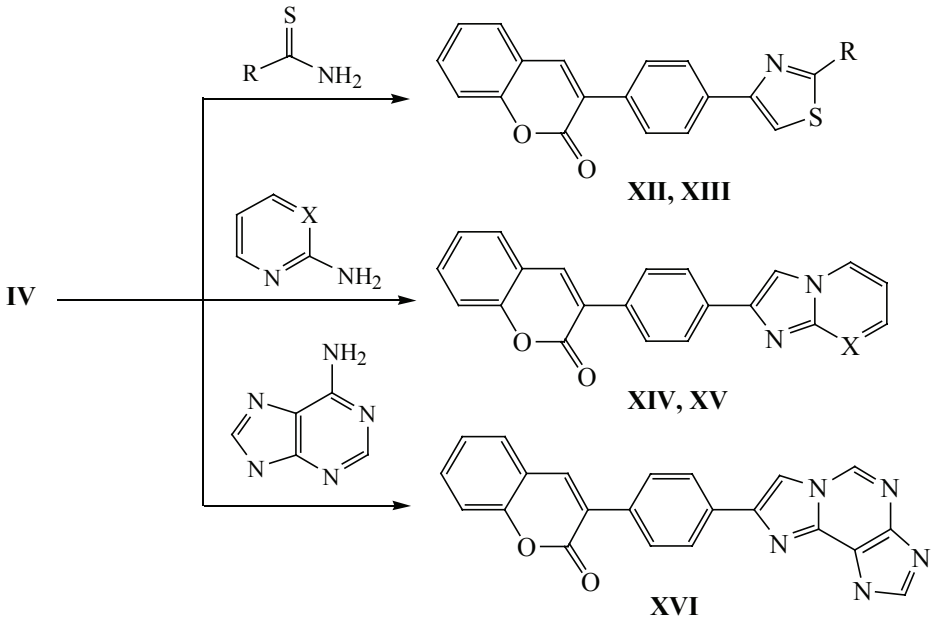
In the IR spectra of salts **V–VIII** the absorption band of the carbonyl group of the fragment $COCH_2$ appears in the region 1660–1680 cm^{-1}, the vibrations of the C–N bond give rise to the band at 3400 cm^{-1} [11], the absorption band of the carbonyl group of the chromenone ring is observed in the region 1715–1725 cm^{-1} [12]. The IR spectrum of phosphonium salt **IX** contains the absorption band of the C=O bond of the oxo group at 1670 cm^{-1}. In going to phosphorylide **X** a strong shift to low frequencies occurs in the absorption of this bond, and it appears as two bands at 1565 and 1605 cm^{-1}. The P=C bond of compound **X** is characterized by two absorption bands at 1385 and 880 cm^{-1}.

The absorption bands in the IR spectrum of compound **XI** at 1610 and 1665 cm^{-1} belong to the stretching vibrations of the aliphatic double bond and of carbonyl group, respectively. The intensity of the absorption band of the carbonyl group is considerably less than that of the C=C bond indicating the mutual *s*-*cis*-position of the carbonyl and the vinyl groups [13]. In the region of 980–990 cm^{-1} an absorption band is observed originating from the bending =C–H vibrations of the *trans*-substituted vinyl group [14].

We used the bromoacetyl group of compound **IV** for designing nitrogen heterocycles. The attention directed to the synthesis of compounds containing a coumarin fragment and nitrogen heterocycles originates from the presence among them of substances possessing high biological activity [15, 16]. We investigated cyclocondensations of 3-[4-(2-bromoacetyl)phenyl]-2*H*-chromen-2-one (**IV**) with thioacetamide, thiourea, 2-aminopyridine, 2-aminopyrimidine, and 6-aminopurine and obtained derivatives of thiazole **XII, XIII**, imidazo[1,2-*a*]pyridine **XIV**, imidazo[1,2-*a*]pyrimidine **XV**, imidazo[2,1-*i*]purine **XVI** (Scheme 4).

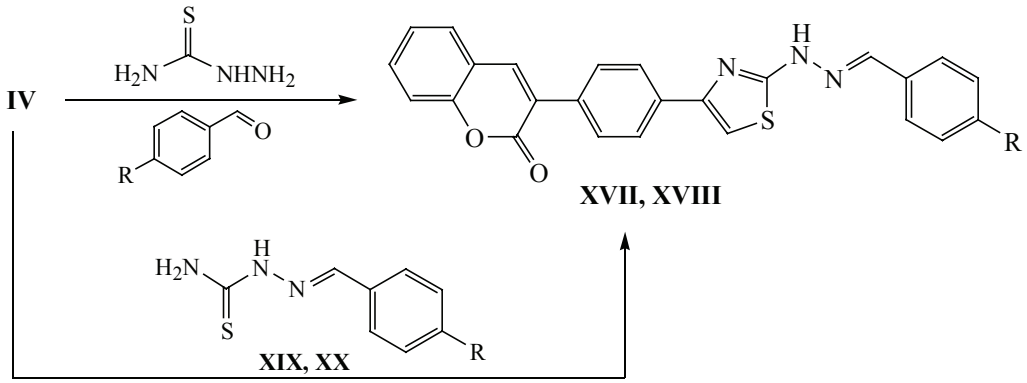
Bromo derivative **IV** reacted with thiosemicarbazide in ethanol at heating with the formation of a thiazole ring. The subsequent addition to the reaction mixture of 4-nitro- or 4-dimethylaminobenzaldehyde led to the formation of compounds **XVII, XVIII**. The same

Scheme 4.



R = Me (**XII**), NH_2 (**XIII**); X = CH (**XIV**), N (**XV**).

Scheme 5.



R = NO_2 (**XVII, XIX**), NMe_2 (**XVIII, XX**).

compounds were obtained at heating reagent **IV** with the thiosemicarbazones of the corresponding aldehydes **XIX, XX** (Scheme 5).

The thiazole ring in the IR spectra of compounds **XII, XIII, XVII, XVIII** gives rise to the absorption bands at 1535–1560 cm^{-1}.

EXPERIMENTAL

IR spectra were recorded on a spectrophotometer Specord 75IR from pellets with KBr. ^1H NMR spectra were registered on a spectrometer Varian Mercury 400 (400 MHz) in DMSO-d_6, internal reference TMS. Melting points were measured on Boëtius heating block.

4-Acetylphenyldiazonium chloride (II). A mixture of 3.8 g (0.028 mol) of 4-aminoacetophenone, 18 mL of conc. HCl, and 10 mL of water was heated to boiling. The obtained solution was cooled to 0–5°C and kept for 5 min. To the formed slurry of aminoacetophenone hydrochloride at efficient cooling was added dropwise while stirring 2.4 g (0.034 mol) of $NaNO_2$ in 10 mL of water. On completing the addition the reaction mixture was left standing for 15 min in an ice bath. The obtained solution of diazonium salt was filtered, the filtrate was

neutralized by portions of 20–30 mL with saturated sodium acetate solution to pH 6.

3-(4-Acetylphenyl)-2*H*-chromen-2-one (III). To a mixture of 4.1 g (0.028 mol) of coumarin **I**, 0.36 g (2.1 mol) of $CuCl_2 \cdot 2H_2O$, and 35 mL of acetone was added at stirring dropwise a cooled solution of 4-acetylphenyldiazonium chloride at a rate 1–2 drop per second. After the end of nitrogen liberation (~2 h) the formed precipitate was filtered off, washed with water, dried, and recrystallized from a mixture ethanol–DMF. Yield 2.96 g (40%), mp 225–227°C. ^{1}H NMR spectrum, δ, ppm: 2.62 s (3H, CH_3), 7.36–7.47 m ($2H_{Ht}$), 7.66 t ($1H_{Ht}$, *J* 8.2 Hz), 7.81 d ($1H_{Ht}$, *J* 7.6 Hz), 7.89 d ($2H_{arom}$, *J* 8.2 Hz), 8.04 d ($2H_{arom}$, *J* 8.2 Hz), 8.38 s ($1H_{Ht}$). Found, %: C 77.08; H 4.31. $C_{17}H_{12}O_3$. Calculated, %: C 77.26; H 4.58.

3-[4-(2-Bromoacetyl)phenyl]-2*H*-chromen-2-one (IV). To a solution of 2.64 g (0.01 mol) of reagent **III** in 120 mL of acetic acid at 85–90°C was added dropwise 0.52 mL (0.01 mol) of bromine. Then the solution was cooled, the crystalline precipitate was filtered off, washed with water, dried, and recrystallized from aqueous DMF. Yield 2.61 g (76%), mp 175–177°C. ^{1}H NMR spectrum, δ, ppm: 5.64 s (2H, CH_2), 7.40 t ($1H_{Ht}$, *J* 7.4 Hz), 7.45 d ($1H_{Ht}$, *J* 8.2 Hz), 7.65 t ($1H_{Ht}$, *J* 7.4 Hz), 7.81 d ($1H_{Ht}$, *J* 7.4 Hz), 7.96 d ($2H_{arom}$, *J* 8.6 Hz), 8.05 d ($2H_{arom}$, *J* 8.6 Hz), 8.40 s ($1H_{Ht}$). Found, %: C 59.63; H 3.38; Br 23.05. $C_{17}H_{11}BrO_3$. Calculated, %: C 59.50; H 3.23; Br 23.28.

Quaternary salts V–IX. A mixture of equimolar amounts (1 mmol) of compound **IV** and an appropriate heterocyclic base (or triphenylphosphine) was boiled in 15–20 mL of anhydrous toluene for 1–1.5 h. The formed precipitates of salts **V–IX** were filtered off and dried.

1-{2-Oxo-2-[4-(2-oxo-2*H*-chromen-3-yl)phenyl] ethyl}pyridinium bromide (**V**). Yield 0.37 g (88%), mp >262°C (CH_3COOH). ^{1}H NMR spectrum, δ, ppm: 6.59 s (2H, CH_2), 7.44 t (1H, coumarin, *J* 7.4 Hz), 7.49 d (1H, coumarin, *J* 8.2 Hz), 7.69 t (1H, coumarin, *J* 7.4 Hz), 7.87 d (1H, coumarin, *J* 7.0 Hz), 8.05 d ($2H_{arom}$, *J* 8.2 Hz), 8.17 d ($2H_{arom}$, *J* 8.2 Hz), 8.32 t ($2H_{Py}$, *J* 6.7 Hz), 8.51 s (1H, coumarin), 8.77 t ($1H_{Py}$, *J* 7.4 Hz), 9.08 d ($2H_{Py}$, *J* 5.9 Hz). Found, %: C 62.69; H 4.03; N 3.41. $C_{22}H_{16}BrNO_3$. Calculated, %: C 62.58; H 3.82; N 3.32.

4-Methyl-1-{2-oxo-2-[4-(2-oxo-2*H*-chromen-3-yl) phenyl]ethyl}pyridinium bromide (**VI**). Yield 0.40 g (92%), mp 260–262ºC (toluene–C_2H_5OH). ^{1}H NMR spectrum, δ, ppm: 2.68 s (3H, CH_3), 6.57 s (2H, CH_2), 7.41 t (1H, coumarin, *J* 7.4 Hz), 7.46 d (1H, coumarin, *J* 8.2 Hz), 7.67 t (1H, coumarin, *J* 7.4 Hz), 7.86 d (1H, coumarin, *J* 7.8 Hz), 8.03 d ($2H_{arom}$, *J* 8.2 Hz), 8.10–8.21 m (4H), 8.51 s (1H, coumarin), 8.94 d ($2H_{Py}$, *J* 5.9 Hz). Found, %: C 63.46; H 4.28; N 3.47. $C_{23}H_{18}BrNO_3$. Calculated, %: C 63.32; H 4.16; N 3.21.

1-{2-Oxo-2-[4-(2-oxo-2*H*-chromen-3-yl)-phenyl] ethyl}quinolinium bromide (**VII**). Yield 0.34 g (71%), mp 248–250°C (aqueous ethanol). ^{1}H NMR spectrum, δ, ppm: 7.09 c (2H, CH_2), 7.44 t (1H, coumarin, *J* 7.4 Hz), 7.50 d (1H, coumarin, *J* 8.2 Hz), 7.70 t (1H, coumarin, *J* 7.4 Hz), 7.89 d (1H, coumarin, *J* 7.8 Hz), 8.05–8.09 m ($3H_{arom}$), 8.23–8.28 m ($3H_{arom}$), 8.36 d.d ($1H_{quin.}$, *J* 8.2, 5.9 Hz), 8.49–8.52 m ($2H_{arom}$), 8.58 d ($1H_{quin}$, *J* 8.2 Hz), 9.48 d ($1H_{quin}$, *J* 8.2 Hz), 9.58 d ($1H_{quin}$, *J* 5.9 Hz). Found, %: C 66.41; H 3.72; N 3.11. $C_{26}H_{18}BrNO_3$. Calculated, %: C 66.12; H 3.84; N 2.97.

4-{2-Oxo-2-[4-(2-oxo-2*H*-chromen-3-yl)-phenyl] ethyl}benzo[*f*]quinolinium bromide (VIII). Yield 0.45 g (86%), mp 204–206°C (CH_3COOH). ^{1}H NMR spectrum, δ, ppm: 7.18 s (2H, CH_2), 7.42 t (1H, coumarin, *J* 7.4 Hz), 7.47 d (1H, coumarin, *J* 8.2 Hz), 7.67 t (1H, coumarin, *J* 7.8 Hz), 7.86 d (1H, coumarin, *J* 7.0 Hz), 7.97 t (1H, $C_{13}H_9N$, *J* 7.4 Hz), 8.01–8.08 m ($3H_{arom}$), 8.24–8.29 m ($3H_{arom}$), 8.35 d (1H, $C_{13}H_9N$, *J* 8.6 Hz), 8.47–8.51 m ($2H_{arom}$), 8.63 d (1H, $C_{13}H_9N$, *J* 8.6 Hz), 9.15 d (1H, $C_{13}H_9N$, *J* 7.4 Hz), 9.57 br.s (1H, $C_{13}H_9N$), 10.26 d (1H, $C_{13}H_9N$, *J* 6.7 Hz). Found, %: C 69.20; H 4.10; N 2.81. $C_{30}H_{20}BrNO_3$. Calculated, %: C 68.98; H 3.86; N 2.68.

{2-Oxo-2-[4-(2-oxo-2*H*-chromen-3-yl)phenyl]-ethyl}triphenylphosphonium bromide (IX). Yield 0.58 g (95%), mp 163–165°C (ethanol). ^{1}H NMR spectrum, δ, ppm: 6.39 d (2H, J_{PH} 12.9 Hz), 7.40 t (1H, coumarin, *J* 7.4 Hz), 7.44 d (1H, coumarin, *J* 8.2 Hz), 7.64 t (1H, coumarin, *J* 7.0 Hz), 7.78–8.03 m ($17H_{arom}$), 8.20 d (2H, C_6H_4, *J* 8.2 Hz), 8.47 s (1H, coumarin). Found, %: C 69.25; H 4.12. $C_{35}H_{26}BrO_3P$. Calculated, %: C 69.43; H 4.33.

1-[4-(2-Oxo-2*H*-chromen-3-yl)phenyl]-2-triphenyl-λ^5-phosphanylidene-1-ethanone (X). To a solution of 0.6 g (1 mmol) of phosphonium salt **IX** in 15 mL of DMF was added at stirring 25 mL of 10% water solution of K_2CO_3. The formed precipitate was filtered off, washed with water, and dried. Yield 0.42 g (81%), mp 192–194°C (toluene). ^{1}H NMR spectrum, δ, ppm: 7.37–7.47 m (3H), 7.78–8.03 m (15H), 7.81 d (2H, *J* 7.8 Hz), 7.89 d (1H,

J 7.8 Hz), 7.96 d (2H, *J* 7.4 Hz), 8.03 d (1H, *J* 7.4 Hz), 8.30 s (1H, coumarin). Found, %: C 80.31; H 4.67. $C_{35}H_{25}O_3P$. Calculated, %: C 80.14; H 4.80.

3-(4-Nitrophenyl)-1-[4-(2-oxo-2*H*-chromen-3-yl) phenyl]prop-2-en-1-one (XI). To a solution of 0.26 g (0.5 mmol) of compound **X** in 20 mL of anhydrous toluene was added 0.08 g (0.5 mmol) of 4-nitrobenzaldehyde, and the mixture was boiled for 2 h. The formed precipitate was filtered off, washed with ether. Yield 0.15 g (75%), mp >260°C (DMF). ^{1}H NMR spectrum, δ, ppm: 7.43 t (1H, coumarin, *J* 7.8 Hz), 7.49 d (1H, coumarin, *J* 8.2 Hz), 7.68 t (1H, coumarin, *J* 7.4 Hz), 7.85 d (1H, coumarin, *J* 7.8 Hz), 7.88 d (1H, CH, *J* 15.7 Hz), 7.98 d (2H, C_6H_4, *J* 8.2 Hz), 8.19–8.33 m ($6H_{arom}$ + $1H_{CH}$), 8.46 s (1H, coumarin). Found, %: C 72.38; H 3.59; N 3.76. $C_{24}H_{15}NO_5$. Calculated, %: C 72.54; H 3.80; N 3.52.

Heterocyclic derivatives XII–XVI. General procedure. To 0.34 g (1 mmol) of bromoketone **IV** in 15 mL of ethanol was added an equimolar quantity of an appropriate reagent (thioacetamide, thiourea, 2-aminopyridine, 2-aminopyrimidine, 6-aminopurine). The reaction mixture was boiled for 1 h. The precipitate was filtered off, washed with ether.

3-[4-(2-Methyl-1,3-thiazol-4-yl)phenyl]-2*H*-chromen-2-one (XII). Yield 0.29 g (91%), mp 228–230°C (aqueous DMF). ^{1}H NMR spectrum, δ, ppm: 2.74 s (3H, CH_3), 7.39 t (1H, coumarin, *J* 7.4 Hz), 7.45 d (1H, coumarin, *J* 8.2 Hz), 7.63 t (1H, coumarin, *J* 7.8 Hz), 7.79–7.84 m (3H), 8.01–8.05 m ($3H_{arom}$), 8.34 s (1H, coumarin). Found, %: C 71.29; H 4.18; N 4.14. $C_{19}H_{13}NO_2S$. Calculated, %: C 71.45; H 4.10; N 4.39.

3-[4-(2-Amino-1,3-thiazol-4-yl)phenyl]-2*H*-chromen-2-one (XIII). Yield 0.26 g (81%), mp 256–258°C (aqueous DMF). ^{1}H NMR spectrum, δ, ppm: 7.12 s (1H, thiazole), 7.16 br.s (2H, NH_2), 7.37 t (1H, coumarin, *J* 7.4 Hz), 7.43 d (1H, coumarin, *J* 8.2 Hz), 7.61 t (1H, coumarin, *J* 7.4 Hz), 7.74–7.80 m (3H, Ar), 7.88 d (2H, C_6H_4, *J* 8.2 Hz), 8.29 s (1H, coumarin). Found, %: C 67.37; H 3.61; N 8.42. $C_{18}H_{12}N_2O_2S$. Calculated, %: C 67.48; H 3.78; N 8.74.

3-(4-Imidazo[1,2-*a*]pyridin-2-ylphenyl)-2*H*-chromen-2-one (XIV). Yield 0.22 g (65%), mp 229–231°C (aqueous DMF). ^{1}H NMR spectrum, δ, ppm: 7.05 t (1H, imidazopyridine, *J* 6.7 Hz), 7.38–7.47 m (3H), 7.64 t (1H, *J* 7.0 Hz), 7.69 d (1H, *J* 9.0 Hz), 7.81 d (1H, *J* 7.8 Hz), 7.88 d (2H, C_6H_4, *J* 8.2 Hz), 8.07 d (2H, C_6H_4, *J* 8.2 Hz), 8.36 s (1H, coumarin), 8.58 s (1H, imidazopyridine), 8.63 d (1H, imidazopyridine, *J* 6.7 Hz). Found, %: C 77.84; H 3.92; N 8.25. $C_{22}H_{14}N_2O_2$. Calculated, %: C 78.09; H 4.17; N 8.28.

3-(4-Imidazo[1,2-*a*]pyrimidin-2-ylphenyl)-2*H*-chromen-2-one (XV). Yield 0.24 g (71%), mp 166–168°C (aqueous DMF). ^{1}H NMR spectrum, δ, ppm: 7.40–7.47 m (2H), 7.66 t (1H, coumarin, *J* 7.4 Hz), 7.81 d (1H, coumarin, *J* 6.7 Hz), 7.88 t (1H, coumarin, *J* 7.8 Hz), 7.93–8.17 m (6H), 8.41–8.46 m (2H). Found, %: C 74.15; H 3.81; N 12.56. $C_{21}H_{13}N_3O_2$. Calculated, %: C 74.33; H 3.86; N 12.38.

3-{4-(1*H*-Imidazo[2,1-*i*]purin-8-yl)phenyl}-2*H*-chromen-2-one (XVI). Yield 0.33 g (87%), mp 218–221°C (precipitated by water from DMF). ^{1}H NMR spectrum, δ, ppm: 7.42–7.49 m (2H), 7.68 t (1H, coumarin, *J* 7.4 Hz), 7.81–8.22 m (8H), 8.37 s (1H), 8.46 s (1H, coumarin). Found, %: C 69.56; H 3.27; N 18.22. $C_{22}H_{13}N_5O_2$. Calculated, %: C 69.65; H 3.45; N 18.46.

4-Nitrobenzaldehyde {4-[4-(2-oxo-2*H*-chromen-3-yl)phenyl]thiazol-2-yl}hydrazone (XVII). A mixture of 0.68 g (2 mmol) of compound **IV**, 0.18 g (2 mmol) of thiosemicarbazide in 50 mL of anhydrous ethanol was boiled for 10 min. To the reaction mixture 0.3 g (2 mmol) of 4-nitrobenzaldehyde was added, and the mixture was boiled for another 15 min. The formed precipitate was filtered off, washed with ether. Yield 0.87 g (93%), mp >260°C (precipitation by water from DMF). ^{1}H NMR spectrum, δ, ppm: 7.37 t (1H, coumarin, *J* 7.4 Hz), 7.44 d (1H, coumarin, *J* 8.2 Hz), 7.63 t (1H, coumarin, *J* 7.8 Hz), 7.79–7.84 m (2H), 7.90 d (2H, C_6H_4, *J* 8.2 Hz), 8.04 d (2H, C_6H_4, *J* 8.2 Hz), 8.11 d (2H, $C_6H_4NO_2$, *J* 8.8 Hz), 8.21 d (2H, $C_6H_4NO_2$, *J* 8.8 Hz), 8.28 s (1H, CH), 8.38 s (1H, coumarin), 11.72 s (1H, NH). Found, %: C 64.24; H 3.61; N 11.78. $C_{25}H_{16}N_4O_4S$. Calculated, %: C 64.09; H 3.44; N 11.96.

4-Dimethylaminobenzaldehyde {4-[4-(2-oxo-2*H*-chromen-3-yl)phenyl]thiazole-2-yl}hydrazone (XVIII) was obtained similarly. Yield 0.6 g (64%), mp >260°C (precipitation by water from DMF). ^{1}H NMR spectrum, δ, ppm: 2.95 s (6H, CH_3), 6.73 d (2H, C_6H_4, *J* 8.6 Hz), 7.36–7.99 m (12H), 8.31–8.35 m (1H), 11.63 s (1H, NH). Found, %: C 69.27; H 4.60; N 11.87. $C_{27}H_{22}N_4O_2S$. Calculated, %: C 69.51; H 4.75; N 12.01.

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