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Reaction of 4-Hydroxycoumarin with Arylglyoxals and Ureas

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Abstract—One-pot condensation of 4-hydroxycoumarin with arylglyoxals and ureas gave 4-aryl-5-(4-hydroxy-2-oxo-2*H*-chromen-3-yl)-1*H*-imidazol-2(3*H*)-ones. The same compounds may be obtained from aroylbis(4-hydroxy-2-oxo-2*H*-chromen-3-yl)methanes and urea. The reaction of 4-bromobenzaldehyde with 4-hydroxycoumarin in the presence of urea led to the formation of the corresponding Michael adduct without participation of urea.

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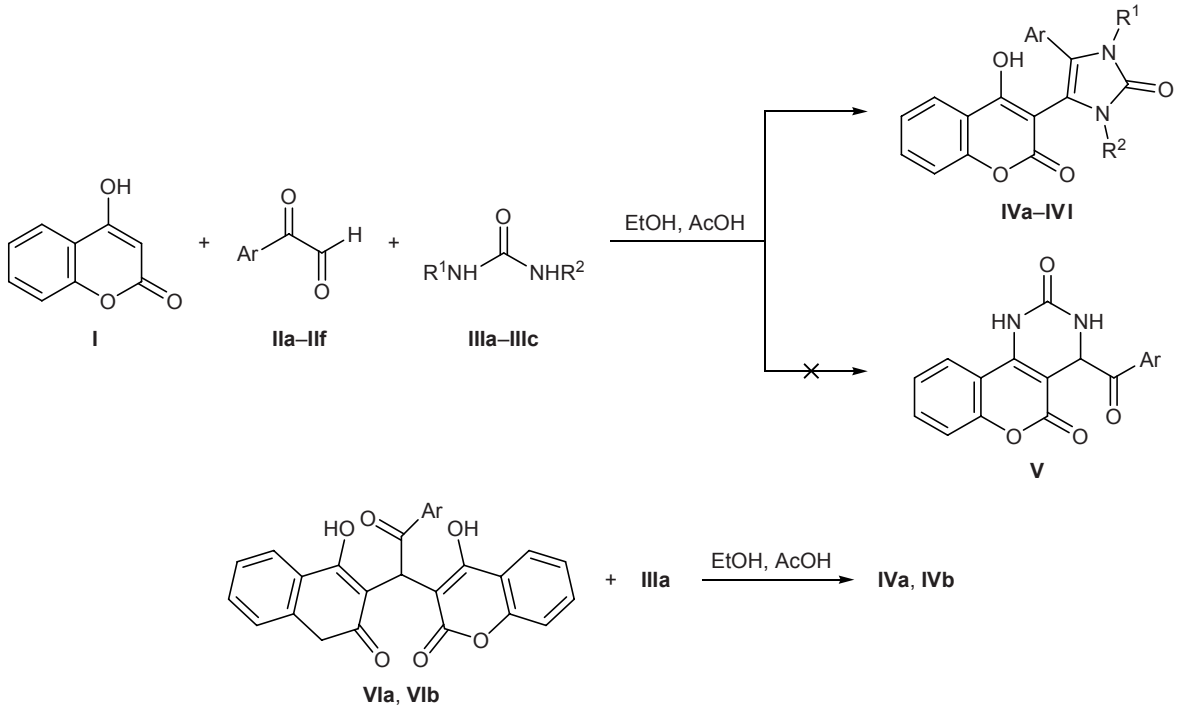
4-Hydroxycoumarin derivatives have found wide application in medical practice as anticoagulants and photosensitizers [1–3], inhibitors of some enzymes [4, 5], and antibacterial and antitumor agents [6, 7]. From the synthetic viewpoint, 4-hydroxycoumarin is a cyclic β-keto ester which can be used as a convenient three-carbon synthon in processes accompanied by recyclization of the chromene ring system [8–12]; it also acts as CH acid and gives rise to bis-adducts with aldehydes and glyoxals [12, 13]. Kidwai et al. [14] reported on Biginelli reaction of 4-hydroxycoumarins with aromatic aldehydes and urea or thiorea. Analogous three-component condensations with participation of arylglyoxals were almost not studied. We can note only one publication [15] concerning one-pot synthesis of dihydropyrimidinones from ethyl acetoacetate, urea, and arylglyoxals.

The goal of the present work was to examine the possibility for synthesizing aroyl derivatives of chromeno[4,3-*d*]pyrimidines with the use of arylglyoxals as carbonyl component. However, heating of a mixture of 4-hydroxycoumarin (**I**), arylglyoxal **IIa**–**IIf**, and urea **IIIa**–**IIIc** in ethanol in the presence of a catalytic amount of acetic acid for a short time (15–50 min) resulted in the formation of colorless crystalline products **IVa**–**IVl** whose physical and chemical properties were not consistent with the structure of expected benzopyrano[4,3-*d*]pyrimidines **V**. A compound like **IV** (namely **IVa**) was also formed in the reaction catalyzed by HCl under the conditions described in [14] for the synthesis of chromenopyrimidines, but the yield and purity were poorer. In addition, compounds **IVa** and **IVb** were also synthesized (in lower yield and with an impurity of 4-hydroxycoumarin) in an alternative way, by heating bis-adducts **VIa** and **VIb** with urea in boiling ethanol in the presence of acetic acid as catalyst (Scheme 1).

The IR spectra of **IVa**–**IVl** contained absorption bands due to stretching vibrations of the lactone (1710–1729 cm^{-1}) and amide carbonyl groups (1669–1695 cm^{-1}). Their ^1H NMR spectra confirmed that the coumarin fragment was conserved during the process: the spectra displayed a dublet from the 5-H proton (J = 7.4–8.0 Hz), a triplet from 7-H (J = 7.0–8.0 Hz), and a multiplet from 6-H and 8-H at δ 7.32–7.45 ppm. In the downfield region of the spectra of **IVa**–**IVf** we observed one-proton singlets at δ ~10.0 and 10.5 ppm (one singlet at δ ~10.5 ppm in the spectra of **IVg**–**IVi**) and a broadened singlet at δ ~12.0 ppm, which disappeared upon addition of D_2O.

Compound **IVe** showed in the mass spectrum the molecular ion peak with m/z 446. Fragmentation paths of the molecular ion (Scheme 2) include opening of the pyran ring with elimination of phenol molecule $[M - 98]^{+\cdot}$ and expulsion of *p*-iodophenyl radical $[M - 203]^{+}$; in addition, 4-hydroxycoumarin ion peak (m/z 162) was present. The base peak was that with m/z 121 due to 2-hydroxybenzoyl ion.

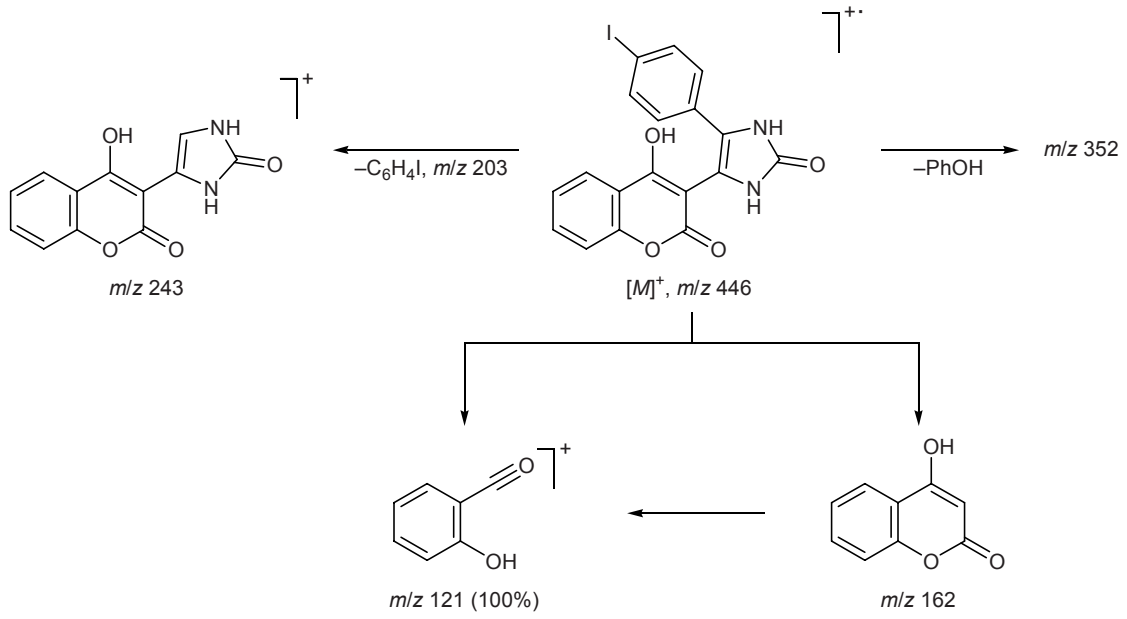
Scheme 1.



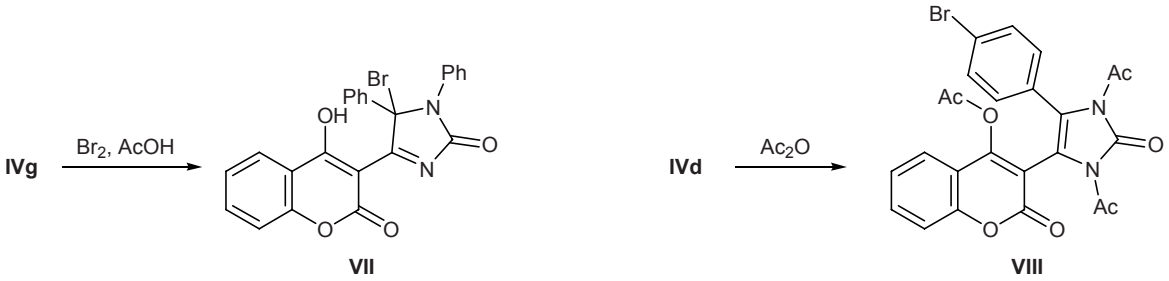
II, Ar = Ph (**a**), 4-MeC_6H_4 (**b**), 4-ClC_6H_4 (**c**), 4-BrC_6H_4 (**d**), 4-IC_6H_4 (**e**), 2-thienyl (**f**); **III**, $R^1 = R^2 = H$ (**a**); R^1 = Ph, R^2 = H (**b**); $R^1 = R^2$ = Me (**c**); **IV**, $R^1 = R^2 = H$; Ar = Ph (**a**), 4-MeC_6H_4 (**b**), 4-ClC_6H_4 (**c**), 4-BrC_6H_4 (**d**), 4-IC_6H_4 (**e**), 2-thienyl (**f**); $R^1 = R^2$ = Me; Ar = Ph (**g**), 4-MeC_6H_4 (**h**), 4-ClC_6H_4 (**i**), R^1 = Ph, R^2 = H; Ar = Ph (**j**), 4-MeC_6H_4 (**k**), 4-ClC_6H_4 (**l**); **VI**, Ar = Ph (**a**), 4-MeC_6H_4 (**b**).

The structure of compounds **IV** was additionally confirmed by their bromination and acylation. Treatment of substituted dihydroimidazol-2-one **IVg** gave bromo derivative **VII**, whereas acylation of **IVd** with acetic anhydride involved three nucleophilic centers (one oxygen and two nitrogen atoms) to afford compound **VIII** (Scheme 3). Thus, the spectral and chemical properties of compounds **IVa**–**IVl** are consistent with the structure of 4-aryl-5-(4-hydroxy-2-oxo-2*H*-chromen-3-yl)-1*H*-imidazol-2(3*H*)-ones.

Scheme 2.

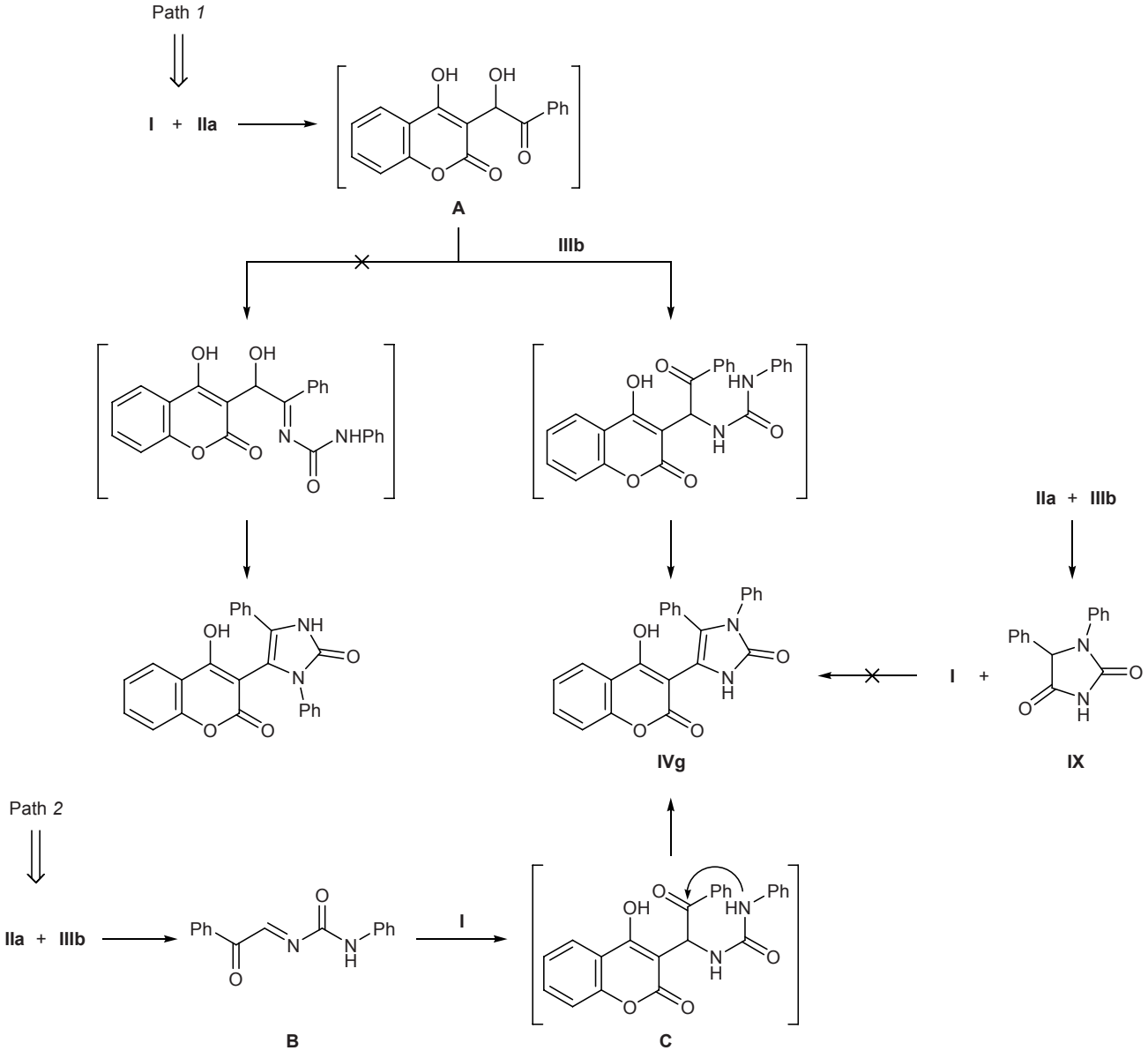


Scheme 3.

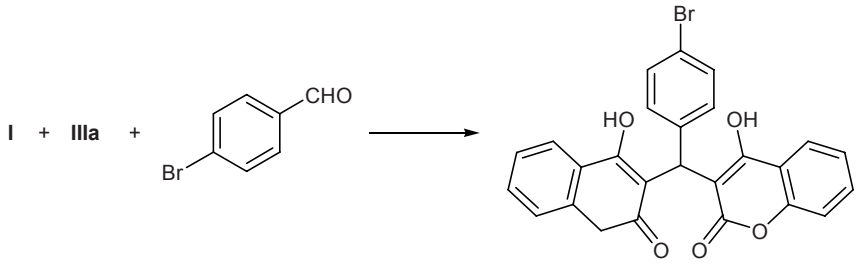


It is known [16–18] that reactions of arylglyoxals with ureas led to the formation of hydantoins. However, as might be expected, the reaction of preliminary prepared hydantoin **IX** with 4-hydroxycoumarin under the above conditions did not produce compound **IVg**. An alternative reaction path includes formation of

Scheme 4.



Scheme 5.



Xb **XI**

intermediate **A** (Scheme 4, path *1*) which then reacts with *N*-phenylurea via addition at the carbonyl group or undergoes replacement of the side-chain hydroxy group (in the phenacyl fragment) (Scheme 4). The first version leads to 1,4-diphenyl-2,3-dihydro-1*H*-imidazol-2-one derivative, while the second, to regioisomeric 3,4-diphenyl-substituted compound. We also cannot rule out formation of Schiff base **B** (path *2*), followed by transformation into intermediate **C** and intramolecular cyclization of the latter. Analogous intermediates were isolated previously [19].

To distinguish between 1,4- and 3,4-diphenyl-substituted 5-(4-hydroxy-2-oxo-2*H*-chromen-3-yl)-2,3-dihydro-1*H*-imidazol-2-ones, we recorded the NOE spectrum of compound **IVg**. Irradiation at a frequency corresponding to resonance of the NH proton gave no response on protons in the *ortho* positions of the phenyl ring attached to carbon atom; therefore, compounds **IVg**–**IVi** were assigned the structure of 3,4-diphenyl derivatives.

Our results indicated that under the above conditions the electrophilic C^4 atom in molecule **I** is not involved in the reaction with such a weak nucleophile as urea. On the other hand, it is known that strong nucleophiles (aromatic *ortho*-diamines [8–11], α-amino acid esters, α-amino ketones [20]) attack just the C^4 atom. Therefore, we tried to perform one-pot reaction of 4-hydroxycoumarin with aromatic aldehydes and urea under the conditions reported in [14]. However, we failed to obtain chromenopyrimidines like **V**. Instead, in the reaction with 4-bromobenzaldehyde (**Xb**), the product was the corresponding bis-adduct **XI** (Scheme 5) whose melting point coincided with that given in [21], while in the reaction with benzaldehyde we isolated only unreacted 4-hydroxycoumarin; i.e., in no case urea was involved in the process. Unfortunately, the ^{1}H NMR spectra of compounds **V** given in [14] were recorded at a frequency of 60 MHz, so that they do not ensure reliable identification of structure **XI** or **V**. Our ^{1}H NMR data for compound **XI** confirm its structure and are consistent with those given in [1].

EXPERIMENTAL

The progress of reactions and the purity of products were monitored by TLC on Silufol UV-254 plates using toluene–ethyl acetate (1 : 1) as eluent (development with iodine vapor). The IR spectra were recorded in KBr on a Specord-75 IR spectrometer. The ^{1}H NMR spectra were measured on a Varian Mercury VX-200 instrument from solutions in DMSO-d_6 using tetramethylsilane as internal reference. The mass spectra (electron impact, 70 eV) were obtained on a Hewlett–Packard LC/MSD 1100 instrument. The elemental compositions were determined on a Leco CHNS-900 analyzer. The melting points were measured on a Kofler hot stage.

Aroylbis(4-hydroxy-2-oxo-2*H*-chromen-3-yl)-methanes VIa and VIb were synthesized according to the procedure described in [12].

4-Aryl-5-(4-hydroxy-2-oxo-2*H*-chromen-3-yl)-2,3-dihydro-1*H*-imidazol-2-ones IVa–IVl (*general procedure***).** *a*. (*One-pot procedure*). 4-Hydroxycoumarin (**I**), 1 mmol, was dissolved in 10 ml of ethanol containing 3–4 drops of acetic acid, 1 mmol of the corresponding aryl(hetaryl)glyoxal **IIa**–**IIf** and 1 mmol of urea **IIIa**–**IIIc** were added, and the mixture was heated for 15–50 min under reflux until a colorless solid separated from the hot solution. The precipitate was filtered off and recrystallized from ethanol.

b. A mixture of equimolar amounts of bisadduct **VI** and urea **III**, 1 mmol each, in 10 ml of ethanol was heated for 1.5 h. The colorless precipitate was filtered off and recrystallized from ethanol.

5-(4-Hydroxy-2-oxo-2*H*-chromen-3-yl)-4-phenyl-2,3-dihydro-1*H*-imidazol-2-one (IVa). Yield 70% (*a*),

41% (*b*), mp 269–270°C. IR spectrum, ν, cm^{-1}: 1719 (C=O, lactone), 1685 (C=O, amide), 1610 (C=C). ^1H NMR spectrum, δ, ppm: 7.12–7.43 m (7H, C_6H_5, 6-H, 8-H), 7.68 t (1H, 7-H, *J* = 7.0 Hz), 7.89 d (1H, 5-H, *J* = 8.0 Hz), 9.97 s (1H, NH), 10.51 s (1H, NH), 12.04 br.s (1H, OH). Found, %: C 67.61; H 3.85; N 8.67. $C_{18}H_{12}N_2O_4$. Calculated, %: C 67.50; H 3.78; N 8.75.

5-(4-Hydroxy-2-oxo-2*H*-chromen-3-yl)-4-(4-methylphenyl)-2,3-dihydro-1*H*-imidazol-2-one (IVb). Yield 68% (*a*), 44% (*b*), mp 263–264°C. IR spectrum, ν, cm^{-1}: 1709 (C=O, lactone), 1689 (C=O, amide), 1608 (C=C). ^1H NMR spectrum, δ, ppm: 2.19 s (3H, CH_3), 7.03 d (2H, *o*-H, *J* = 7.8 Hz), 7.19 d (2H, *m*-H, *J* = 8.2 Hz), 7.32–7.42 m (2H, 6-H, 8-H), 7.67 t (1H, 7-H, *J* = 7.8 Hz), 7.89 d (1H, 5-H, *J* = 7.6 Hz), 9.93 s (1H, NH), 10.48 s (1H, NH), 11.96 br.s (1H, OH). Found, %: C 68.31; H 4.35; N 8.42. $C_{19}H_{14}N_2O_4$. Calculated, %: C 68.26; H 4.22; N 8.38.

4-(4-Chlorophenyl)-5-(4-hydroxy-2-oxo-2*H*-chromen-3-yl)-2,3-dihydro-1*H*-imidazol-2-one (IVc). Yield 69%, mp 278–279°C. IR spectrum, ν, cm^{-1}: 1709 (C=O, lactone), 1688 (C=O, amide), 1609 (C=C). ^1H NMR spectrum, δ, ppm: 7.30 m (4H, C_6H_4), 7.36–7.43 m (2H, 6-H, 8-H), 7.67 t (1H, 7-H, *J* = 7.2 Hz), 7.89 d (1H, 5-H, *J* = 7.6 Hz), 10.03 s (1H, NH), 10.56 s (1H, NH), 12.00 br.s (1H, OH). Found, %: C 61.02; H 3.19; N 7.82. $C_{18}H_{11}ClN_2O_4$. Calculated, %: C 60.94; H 3.13; N 7.90.

4-(4-Bromophenyl)-5-(4-hydroxy-2-oxo-2*H*-chromen-3-yl)-2,3-dihydro-1*H*-imidazol-2-one (IVd). Yield 63%, mp 274–275°C. IR spectrum, ν, cm^{-1}: 1708 (C=O, lactone), 1682 (C=O, amide), 1615 (C=C). ^1H NMR spectrum, δ, ppm: 7.23 m (4H, C_6H_4), 7.36–7.43 m (2H, 6-H, 8-H), 7.69 t (1H, 7-H, *J* = 8.0 Hz), 7.90 d (1H, 5-H, *J* = 7.6 Hz), 10.03 s (1H, NH), 10.56 s (1H, NH), 11.97 br.s (1H, OH). Found, %: C 54.24; H 2.69; N 7.20. $C_{18}H_{11}BrN_2O_4$. Calculated, %: C 54.16; H 2.78; N 7.02.

5-(4-Hydroxy-2-oxo-2*H*-chromen-3-yl)-4-(4-iodophenyl)-2,3-dihydro-1*H*-imidazol-2-one (IVe). Yield 63%, mp 261–262°C. IR spectrum, ν, cm^{-1}: 1710 (C=O, lactone), 1687 (C=O, amide), 1614 (C=C). ^1H NMR spectrum, δ, ppm: 7.09 d (2H, *m*-H, *J* = 8.6 Hz), 7.33–7.43 m (2H, 6-H, 8-H), 7.56–7.82 t (1H, 7-H, *J* = 7.2 Hz), 7.59 d (2H, *o*-H, *J* = 8.6 Hz), 7.89 d (1H, 5-H, *J* = 7.8 Hz), 10.06 s (1H, NH), 10.56 s (1H, NH), 12.01 br.s (1H, OH). Mass spectrum, *m*/*z* (I_{rel}, %): 446 $[M]^+$ (16), 352 (6), 243 (3), 230 (19), 203 (11), 188 (17), 162 (8), 128 (14), 127 (22), 121 (100), 120 (30), 92 (96), 89 (20). Found, %: C 48.25; H 2.60; N 6.29. $C_{18}H_{11}IN_2O_4$. Calculated, %: C 48.45; H 2.48; N 6.48.

5-(4-Hydroxy-2-oxo-2*H*-chromen-3-yl)-4-(2-thienyl)-2,3-dihydro-1*H*-imidazol-2-one (IVf). Yield 57%, mp 277–278°C. IR spectrum, ν, cm^{-1}: 1709 (C=O, lactone), 1670 (C=O, amide), 1609 (C=C). ^1H NMR spectrum, δ, ppm: 6.94 t (1H, β-H, *J* = 4.0 Hz), 7.16–7.22 m (2H, α-H, β-H), 7.35–7.45 m (2H, 6-H, 8-H), 7.70 t (1H, 7-H, *J* = 7.6 Hz), 7.92 d (1H, 5-H, *J* = 7.8 Hz), 10.04 s (1H, NH), 10.56 s (1H, NH), 12.09 br.s (1H, OH). Found, %: C 59.00; H 3.20; N 8.71. $C_{16}H_{10}N_2O_4S$. Calculated, %: C 58.89; H 3.09; N 8.58.

5-(4-Hydroxy-2-oxo-2*H*-chromen-3-yl)-3,4-diphenyl-2,3-dihydro-1*H*-imidazol-2-one (IVg). Yield 69%, mp 254–255°C. IR spectrum, ν, cm^{-1}: 1720 (C=O, lactone), 1693 (C=O, amide), 1615 (C=C). ^1H NMR spectrum, δ, ppm: 6.93 m (2H, *p*-H), 7.09–7.14 m (4H, C_6H_4), 7.19–7.40 m (6H, NC_6H_4, 6-H, 8-H), 7.66 t (1H, 7-H, *J* = 7.4 Hz), 7.86 d (1H, 5-H, *J* = 7.4 Hz), 10.47 s (1H, NH), 12.06 br.s (1H, OH). Found, %: C 72.59; H 4.18; N 7.21. $C_{24}H_{16}N_2O_4$. Calculated, %: C 72.72; H 4.07; N 7.07.

5-(4-Hydroxy-2-oxo-2*H*-chromen-3-yl)-4-(4-methylphenyl)-3-phenyl-2,3-dihydro-1*H*-imidazol-2-one (IVh). Yield 55%, mp 228–230°C. IR spectrum, ν, cm^{-1}: 1729 (C=O, lactone), 1695 (C=O, amide), 1609 (C=C). ^1H NMR spectrum, δ, ppm: 2.12 s (3H, CH_3), 6.81 d (2H, *o*-H, *J* = 8.0 Hz), 6.91 d (2H, *m*-H, *J* = 8.0 Hz), 7.12 d (2H, *m*-H, *J* = 7.4 Hz), 7.22–7.40 m (5H, *o*-H, 6-H, 8-H, *p*-H), 7.66 t (1H, 7-H, *J* = 8.0 Hz), 7.86 d (1H, 5-H, *J* = 8.0 Hz), 10.43 s (1H, NH), 12.08 br.s (1H, OH). Found, %: C 73.24; H 4.28; N 6.95. $C_{25}H_{18}N_2O_4$. Calculated, %: C 73.16; H 4.42; N 6.83.

4-(4-Chlorophenyl)-5-(4-hydroxy-2-oxo-2*H*-chromen-3-yl)-3-phenyl-2,3-dihydro-1*H*-imidazol-2-one (IVi). Yield 58%, mp 223–224°C. IR spectrum, ν, cm^{-1}: 1718 (C=O, lactone), 1690 (C=O, amide), 1614 (C=C). ^1H NMR spectrum, δ, ppm: 6.91 d (2H, *m*-H, *J* = 8.4 Hz), 7.11–7.41 m (9H, *o*-H, C_6H_5, 6-H, 8-H), 7.67 t (1H, 7-H, *J* = 7.6 Hz), 7.86 d (1H, 5-H, *J* = 7.8 Hz), 10.55 s (1H, NH), 12.04 br.s (1H, OH). Found, %: C 67.04; H 3.40; N 6.70. $C_{24}H_{15}ClN_2O_4$. Calculated, %: C 66.91; H 3.51; N 6.50.

5-(4-Hydroxy-2-oxo-2*H*-chromen-3-yl)-1,3-dimethyl-4-phenyl-2,3-dihydro-1*H*-imidazol-2-one (IVj). Yield 47%, mp >300°C. IR spectrum, ν, cm^{-1}:

1702 (C=O, lactone), 1669 (C=O, amide), 1609 (C=C). ^{1}H NMR spectrum, δ, ppm: 2.99 s (3H, CH_3), 3.12 s (3H, CH_3), 7.27–7.39 m (7H, C_6H_5, 6-H, 8-H), 7.64 t (1H, 7-H, *J* = 7.2 Hz), 7.91 d (1H, 5-H, *J* = 7.4 Hz), 12.09 br.s (1H, OH). Found, %: C 69.06; H 4.40; N 8.15. $C_{20}H_{16}N_2O_4$. Calculated, %: C 68.96; H 4.63; N 8.04.

5-(4-Hydroxy-2-oxo-2*H*-chromen-3-yl)-1,3-dimethyl-4-(4-methylphenyl)-2,3-dihydro-1*H*-imidazol-2-one (IVk). Yield 45%, mp 256–257°C. IR spectrum, ν, cm^{-1}: 1702 (C=O, lactone), 1669 (C=O, amide), 1609 (C=C). ^{1}H NMR spectrum, δ, ppm: 2.22 s (3H, CH_3), 2.97 s (3H, CH_3), 3.10 s (3H, CH_3), 7.13 m (4H, C_6H_4), 7.28–7.39 m (2H, 6-H, 8-H), 7.65 t (1H, 7-H, *J* = 8.0 Hz), 7.81 d (1H, 5-H, *J* = 7.4 Hz), 12.02 br.s (1H, OH). Found, %: C 69.72; H 5.11; N 7.90. $C_{21}H_{18}N_2O_4$. Calculated, %: C 69.60; H 5.01; N 7.73.

4-(4-Chlorophenyl)-5-(4-hydroxy-2-oxo-2*H*-chromen-3-yl)-1,3-dimethyl-2,3-dihydro-1*H*-imidazol-2-one (IVl). Yield 45%, mp 259–260°C. IR spectrum, ν, cm^{-1}: 1710 (C=O, lactone), 1675 (C=O, amide), 1608 (C=C). ^{1}H NMR spectrum, δ, ppm: 2.98 s (3H, CH_3), 3.12 s (3H, CH_3), 7.25 d (2H, *m*-H, *J* = 8.6 Hz), 7.33–7.41 m (4H, *o*-H, 6-H, 8-H), 7.66 t (1H, 7-H, *J* = 7.2 Hz), 7.81 d (1H, 5-H, *J* = 7.4 Hz), 12.03 br.s (1H, OH). Found, %: C 62.60; H 3.90; N 7.41. $C_{20}H_{15}ClN_2O_4$. Calculated, %: C 62.75; H 3.95; N 7.32.

1,5-Diphenylimidazolidine-2,4-dione (**IX**) was synthesized according to the procedure described in [17], mp 193–194°C; published data [17]: mp 195°C.

5-Bromo-4-(4-hydroxy-2-oxo-2*H*-chromen-3-yl)-1,5-diphenyl-2,5-dihydro-1*H*-imidazol-2-one (VII). Compound **IVg**, 2 mmol, was dissolved in 15 ml of acetic acid, a solution of 0.1 ml (2 mmol) of bromine in 10 ml of acetic acid was added dropwise under stirring, and the mixture was stirred until it became colorless, stirred for an additional 1 h, and poured onto ice. The precipitate was filtered off and recrystallized from ethanol. Yield 0.53 g (56%), mp 263–264°C. IR spectrum, ν, cm^{-1}: 1720 (C=O, lactone), 1693 (C=O, amide), 1615 (C=C). ^{1}H NMR spectrum, δ, ppm: 6.99–7.39 m (10H, C_6H_5), 7.53–7.62 m (2H, 6-H, 8-H), 7.70 t (1H, 7-H, *J* = 7.0 Hz), 7.88 d (1H, 5-H, *J* = 8.4 Hz), 11.12 s (1H, OH). Found, %: C 60.73; H 3.30; N 5.80. $C_{24}H_{15}BrN_2O_4$. Calculated, %: C 60.65; H 3.18; N 5.89.

3-[1,3-Diacetyl-5-(4-bromophenyl)-2-oxo-2,3-dihydro-1*H*-imidazol-4-yl]-2-oxo-2*H*-chromen-4-yl acetate (VIII). A mixture of 1 mmol of compound **IVd** and 5 ml of acetic anhydride was heated for 15 min under reflux. It was then cooled and poured onto ice, and the light yellow precipitate was filtered off. Yield 0.36 g (72%), mp 188–190°C (from acetone). ^{1}H NMR spectrum, δ, ppm: 2.31 s (3H, CH_3), 2.53 s (3H, NCH_3), 2.69 s (3H, NCH_3), 7.16 d (2H, *m*-H, *J* = 8.2 Hz), 7.34 t (1H, 6-H, *J* = 7.6 Hz), 7.46 d (2H, *o*-H, *J* = 8.2 Hz), 7.55 m (2H, 5-H, 8-H), 7.70 t (1H, 7-H, *J* = 7.6 Hz). Found, %: C 54.95; H 3.35; N 5.42. $C_{24}H_{17}BrN_2O_7$. Calculated, %: C 54.87; H 3.26; N 5.33.

3,3′-(4-Bromophenylmethylene)bis(4-hydroxy-2*H*-chromen-2-one) (XI) was obtained following the procedure described in [14]. Yield 72%, mp 240–242°C; published data [21]: mp 243–244°C. ^{1}H NMR spectrum, δ, ppm: 6.24 s (1H, CH), 7.07 d (2H, *o*-H, *J* = 8.2 Hz), 7.23–7.38 m (6H, *m*-H, 6-H, 8-H), 7.54 t (2H, 7-H, *J* = 7.6 Hz), 7.84 d (2H, 5-H, *J* = 8.2 Hz).

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