

Synthesis of some new fused coumarin derivatives

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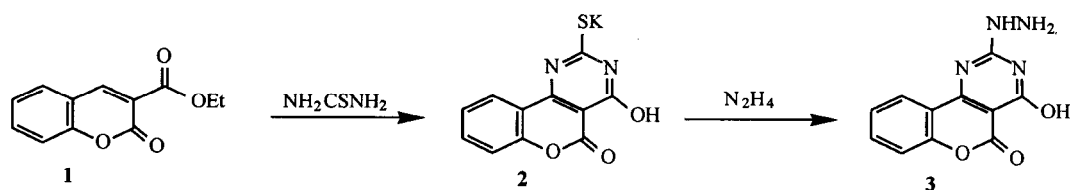
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2-Hydrazino-4-hydroxy-5*H*-[1]-benzopyrano-[4,3-*d*]-pyrimidin-5-one (3) was prepared via condensation of 2 with hydrazine hydrate. Treatment of 3 with methylene chloride, ethyl chloroformate, ethyl chloroacetate and benzaldehyde yielded the corresponding 2-(substituted) hydrazino-4-hydroxy-5*H*-[1]-benzopyrano-[4,3-*d*]-pyrimidin-5-one (4, 5, 6, and 10), followed by cyclization of 4, 5 and 6 with dimethyl formamide and fused sodium acetate under reflux, while compound 10 was cyclized with bromine and sodium acetate in acetic acid. Compound 3 reacted with β -(toloyl) acrylic acid, ethyl α -cyano-*p*-methoxycinnamate, diethyl malonate and acetyl chloride affording the corresponding 2-(substituted) hydrazino-4-hydroxy-5*H*-[1]-benzopyrano-[4,3-*d*]-pyrimidin-5-one (12, 13, 14, 15 and 16).

Keywords Synthesis, fused coumarin derivatives, cyclization

Scheme 1



The reaction of 2-hydrazino-4-hydroxy-5*H*-[1]-benzopyrano-[4,3-*d*]-pyrimidin-5-one (3) with methylene chloride, ethyl chloroformate or ethyl chloroacetate in pyridine under reflux produced 2-(substituted) hydrazino-4-hydroxy-5*H*-[1]-benzopyrano-[4,3-*d*]-pyrimidin-5-one (4, 5 and 6, Scheme 2). 5-Hydroxy-1*H*,6*H*-[1]-benzopyrano-[4,3-*d*]-1,2,4-triazolo-[3,4-*b*]-pyrimidin-6-one (7), 5-hydroxy-1*H*,6*H*-[1]-benzopyrano-[4,3-*d*]-1,2,4-triazolo-[3,4-*b*]-pyrimidin-1,6-dione (8) and 1,2-dihydro-6-hydroxy-7*H*-[1]-benzopy-

As an extension of our previous work,¹⁻⁶ this present work describes the synthesis of some new fused coumarin derivatives starting from salicylaldehyde. Condensation of salicylaldehyde with diethyl malonate in presence of piperidine afforded the corresponding 3-ethoxycarbonylcoumarin (1). Treatment of 1 with thiourea in the presence of anhydrous potassium carbonate in methanol under reflux to form the potassium salt of 2-mercapto-4-hydroxy-5*H*-[1]-benzopyrano-[4,3-*d*]-pyrimidin-5-one (2) in good yield. Heating of the potassium salt of 2 with hydrazine hydrate under fusion gave the corresponding 2-hydrazino-4-hydroxy-5*H*-[1]-benzopyrano-[4,3-*d*]-pyrimidin-5-one (3, Scheme 1).

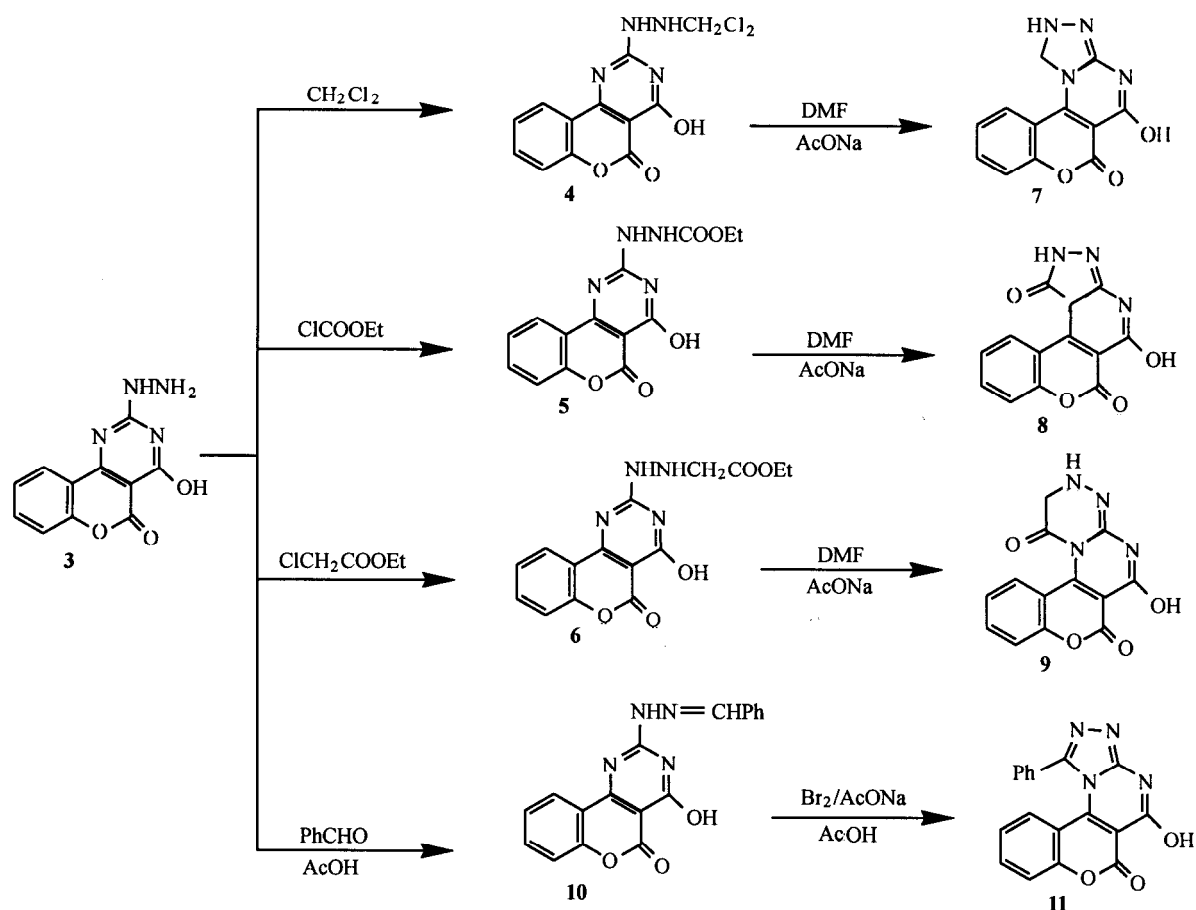
rano-[4',3':4,5]-pyrimido-[2,3-*c*]-1,2,4-triazin-1,7-dione (9) were obtained by boiling of 4, 5 and 6 in dimethyl formamide in presence of fused sodium acetate.

Subsequently, 2-hydrazino-4-hydroxy-5*H*-[1]-benzopyrano-[4,3-*d*]-pyrimidin-5-one (3) was transformed to 5-hydroxy-1-phenyl-6*H*-[1]-benzopyrano-[4,3-*d*]-1,2,4-triazolo-[3,4-*b*]-pyrimidin-6-one (11) via condensation of 3 with benzaldehyde to give 2-benzalhydrazino-4-hydroxy-5*H*-[1]-benzopyrano-[4,3-*d*]-pyrimidin-5-one (10), followed by cyclization of 10 with bromine in

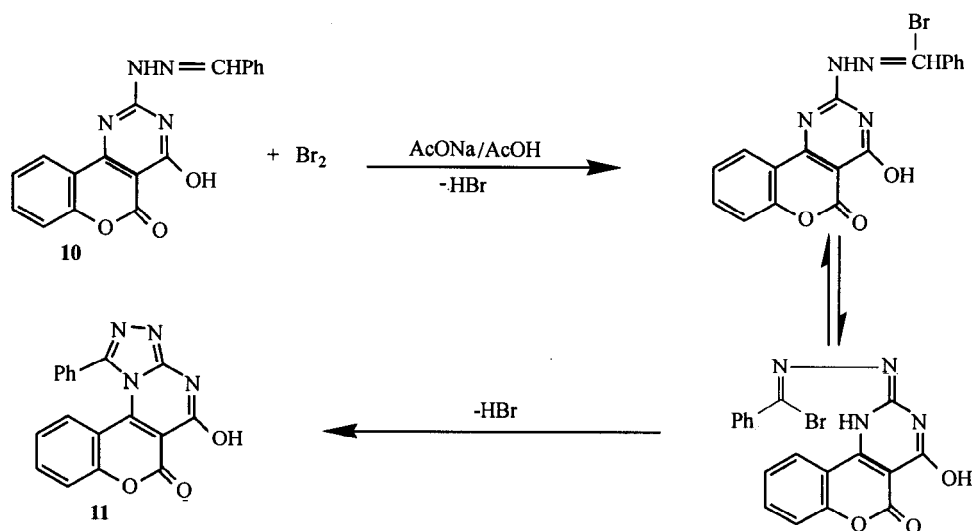
presence of fused sodium acetate in acetic acid. Compound **11** may be formed by bromination of **10** with ring

cyclization via the removal of hydrogen bromide as shown in Scheme 3.

Scheme 2



Scheme 3

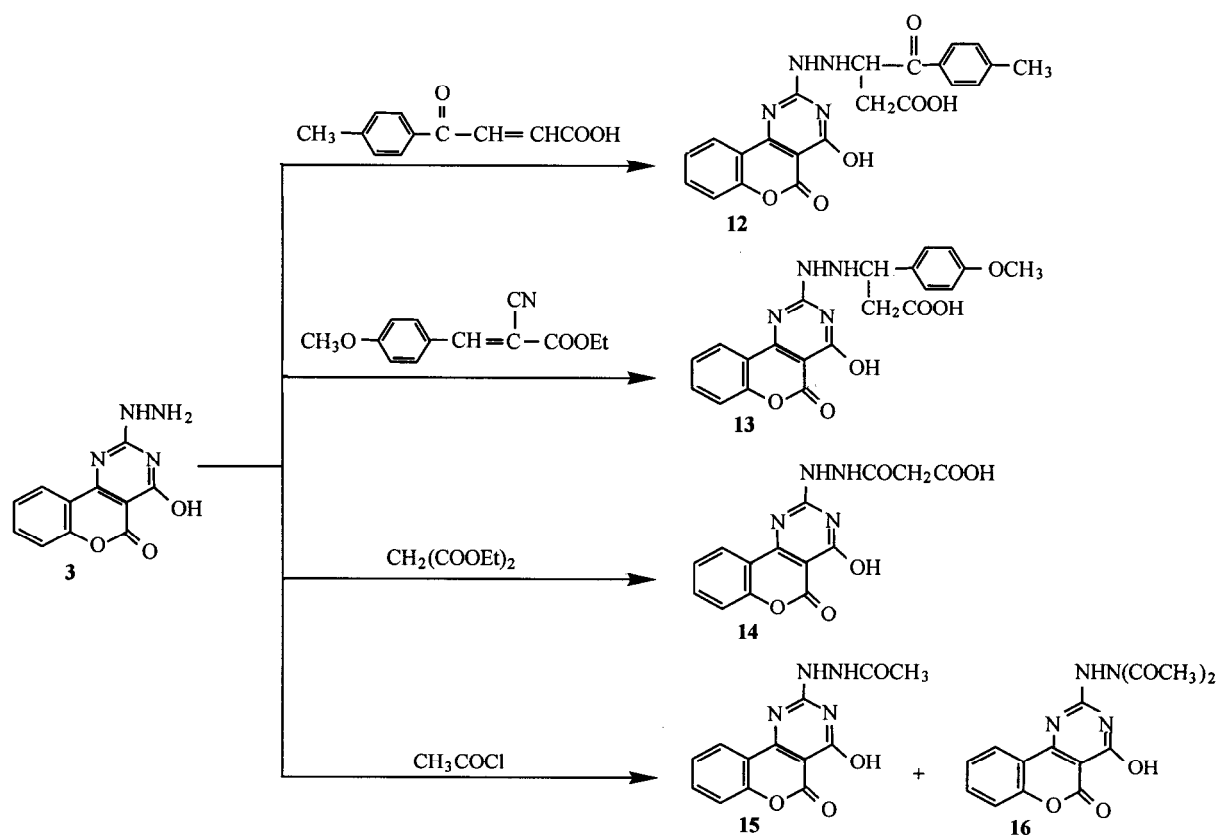


In the detailed work⁷ the reaction of **3** with activated olefinic compounds (namely, β -toloyl-acrylic acid and ethyl α -cyano-*p*-methoxycinnamate) in presence of triethyl amine led to the formation of 2-(substituted)hydrazino-4-hydroxy-5*H*-[1]-benzopyrano-[4,3-d]-pyrimidin-5-one (**12** and **13**, Scheme 4). Condensation of **3** with diethyl malonate in presence of triethyl amine afforded the corresponding 2-malonyl-hydrazino-4-hydroxy-

5*H*-[1]-benzopyrano-[4,3-d]-pyrimidin-5-one (**14**).

On the other hand, the acylation of **3** with acetyl chloride in acetic acid led to the formation of 2-acetyl-hydrazino-4-hydroxy-5*H*-[1]-benzopyrano-[4,3-d]-pyrimidin-5-one (**15**) and 2-diacetyl-hydrazino-4-hydroxy-5*H*-[1]-benzopyrano-[4,3-d]-pyrimidin-5-one (**16**).

Scheme 4



Experimental

NMR spectra were recorded on a General Electric QE 300 instrument and chemical shifts were given with respect to TMS. IR spectra were recorded on a Perkin-Elmer 1420 spectrometer and a Biorad FTS7 (KBr). Mass spectra were obtained on a VG Autospec (EI and FAB +) and a Hewlett packard MS-Engine Thermo-spray. Microanalyses were conducted using an elemental analyzer 1106. Melting points were determined on a Reichert Hot stage and uncorrected.

Potassium salt of 2-mercapto-4-hydroxy-5*H*-[1]-benzopyrano-[4,3-d]-pyrimidin-5-one (**2**)

A mixture of **1** (0.01 mol), thiourea (0.01 mol) and anhydrous potassium carbonate (0.03 mol) in methanol (60 mL) was heated under reflux for 2 h. The solid formed was filtered off, washed with methanol and dried to give **2** as pale yellow powder, yield 54%, mp: > 500°C.

2-Hydrazino-4-hydroxy-5H-[1]-benzopyrano-[4,3-d]-pyrimidin-5-one (3)

A mixture of **2** (0.01 mol) and hydrazine hydrate (0.02 mol) was fused on a hot plate for 10–15 min. The reaction mixture was added to boiling methanol (70 mL) and heated under reflux for 2 h, then cooled. The solid formed was filtered off, washed with methanol, dried and purified by recrystallization with dimethyl formamide to give **3** as orange crystals, yield 73%, mp: > 470°C. ν_{\max} (KBr): 3430–2560 (br. OH), 3336, 3175(NH₂), 3251(NH), 1712(C=O), 1616(C=N), 1125, 1045(C-O) cm⁻¹. δ_{H} (DMSO-*d*₆): 4.36 (s, 2H, NH₂), 7.29–7.78 (m, 4H, ArH), 8.83 (br. s, 1H, OH), 9.35 (s, 1H, NH). *m/z*(%): 245(M⁺ + 1, 67.31), 244(M⁺, 100), 229(25.80), 213 (17.10), 188(26.41), 170(2.61), 145(4.8), 102(24.0), 91(26.60), 88(20.91), 76(24.30). Anal. C₁₁H₈N₄O₃. Calcd: C, 54.10; H, 3.30; N, 22.93. Found: C, 53.97; H, 3.19; N, 22.71.

2-(Substituted) hydrazino-4-hydroxy-5H-[1]-benzopyrano-[4,3-d]-pyrimidin-5-one (4, 5 and 6)

A mixture of **3** (0.01 mol) and methylene chloride, ethyl chloroformate or ethyl chloroacetate (0.01 mol) in pyridine (50 mL) was heated under reflux for 2 h, then cooled and acidified with hydrochloric acid (6 mol/L). The deposited solid was filtered off, washed with water, dried and purified by recrystallization with dimethyl formamide to give **4**, **5** and **6**.

Compound **4** as pale yellow crystals, yield 57%, mp: 342°C. ν_{\max} (KBr): 3390–2596 (br. OH), 3175 (NH), 1710 (C=O), 1615 (C=N), 1095, 1035 (C—O) cm⁻¹. δ_{H} (DMSO-*d*₆): 3.81 (s, 2H, CH₂), 4.35 (s, 1H, NH), 7.30–7.77 (m, 4H, ArH), 8.84 (br. s, 1H, OH), 9.87 (s, 1H, NH). *m/z*(%): 294(M⁺ + 2, 6), 292(M⁺, 17), 256 (100), 242 (13), 229 (7.2), 187 (22.1), 144 (15.20), 102 (23), 91 (23), 87 (53), 77 (13.1), 63 (23). Anal. C₁₂H₉ClN₄O₃. Calcd: C, 49.25; H, 3.10; N, 19.14; Cl, 12.11. Found: C, 49.01; H, 2.97; N, 19.02; Cl, 11.98.

Compound **5** as yellow crystals, yield 62%, mp: 275°C. ν_{\max} (KBr): 3350–2540 (br. OH), 3253 (NH), 1740–1712 (C=O of ester and pyrane), 1617 (C=N), 1226, 1062, 1039 (C—O) cm⁻¹. δ_{H} (DM-

SO-*d*₆): 1.24 (t, *J* = 1.2 Hz, 3H, CH₃), 4.14 (q, *J* = 1.0 Hz, 2H, OCH₂), 7.31–7.75 (m, 4H, ArH), 8.83 (br. s, 1H, OH), 9.50 (br. s, 1H, NH), 10.30 (br. s, 1H, NHCO). *m/z*(%): 317 (M⁺ + 1, 15.9), 316 (M⁺, 16.8), 270 (33.8), 244 (14.1), 229 (17.4), 214 (100), 197 (10.9), 186 (25.6), 145 (10.2), 125 (23.9), 114 (10.0), 102 (13.9), 91 (11.4), 76 (9.4), 69 (15.1), 63 (16.2). Anal. C₁₄H₁₂N₄O₅. Calcd: C, 53.17; H, 3.82; N, 17.71. Found: C, 53.02; H, 3.46; N, 17.52.

Compound **6** as pale yellow crystals, yield 63%, mp: 285°C. ν_{\max} (KBr): 3390–2570 (br. OH), 3251 (NH), 1745 (C=O of ester), 1713 (C=O), 1625 (C=N), 1178, 1037, 1010 (C—O) cm⁻¹. δ_{H} (DMSO-*d*₆): 1.22 (t, *J* = 1.2 Hz, 3H, CH₃), 2.51 (s, 2H, CH₂), 4.21 (q, *J* = 1.0, 2H, OCH₂), 7.31–7.77 (m, 4H, ArH), 8.83 (br. s, 1H, OH), 4.31 (s, 1H, NH), 10.09 (s, 1H, NH). *m/z*(%): 331 (M⁺ + 1, 12), 330 (M⁺, 21), 302 (57.12), 284 (23), 242 (100), 229 (13.1), 215 (57.3), 188 (15.0), 144 (2.9), 102 (10.1), 92 (4.1), 76 (7.5), 63 (10.2). Anal. C₁₅H₁₄N₄O₅. Calcd: C, 54.55; H, 4.27; N, 16.96. Found: C, 54.31; H, 4.09; N, 16.71.

5-Hydro-1H, 6H-[1]-benzopyrano-[4,3-d]-1,2,4-triazolo-[3,4-b]-pyrimidin-6-one (7) 5-hydroxy-1H, 6H-[1]-benzopyrano-[4,3-d]-1,2,4-triazolo-[3,4-b]-pyrimidin-1,6-dione (8) and 1,2-dihydro-6-hydroxy-7H-[1]-benzopyrano-[4',3':4,5]-pyrimido-[2,3-c]-1,2,4-triazin-1,7-dione (9)

To a solution of **4**, **5** or **6** (0.01 mol) in dimethyl formamide (60 mL) was added fused sodium acetate (0.03 mol). The mixture was heated under reflux for 15 h, then cooled and poured into water. The deposited solid was filtered off, washed with water, dried and purified by recrystallization with dimethyl formamide to give **7**, **8** or **9**.

Compound **7** as yellow crystals, yield 78%, mp: 376°C. ν_{\max} (KBr): 3381–2530 (br. OH), 3210 (NH), 1710 (C=O), 1630 (C=N), 1075, 1031 (C—O) cm⁻¹. δ_{H} (DMSO-*d*₆): 4.01 (s, 2H, CH₂), 7.30–7.76 (m, 4H, ArH), 8.84 (br. s, 1H, OH), 9.86 (s, 1H, NH). *m/z*(%): 257 (M⁺ + 1, 7.13), 256 (43.3), 228 (100), 186 (13.5), 145 (9.7), 117 (5.5), 92 (14.5), 63 (20.6). Anal. C₁₂H₈N₄O₃. Calcd: C, 56.26; H, 3.15; N, 21.86. Found: C,

56.00; H, 2.96; N, 21.48.

Compound **8** as yellow crystals, yield 79%, mp: 256°C. ν_{\max} (KBr): 3375—2592 (br. OH), 3179 (NH), 1715 (C=O), 1685 (C=O), 1627 (C=N), 1059, 1030, 1010 (C—O) cm^{-1} . δ_{H} (DMSO- d_6): 7.31—7.76 (m, 4H, ArH), 8.85 (br. s, 1H, OH), 10.4 (s, 1H, NH). m/z (%): 271 ($M^+ + 1$, 31), 270 (M^+ , 34.8), 242 (14.1), 228 (6.8), 214 (100), 186 (27.6), 145 (13.2), 117 (6.51), 91 (14.10), 63 (17.2). Anal. $\text{C}_{12}\text{H}_6\text{N}_4\text{O}_4$. Calcd: C, 53.34; H, 2.24; N, 20.73. Found: C, 53.14; H, 2.03; N, 20.46.

Compound **9** as yellow crystals, yield 73%, mp: > 390°C. ν_{\max} (KBr): 3380—2560 (br. OH), 3224 (NH), 1710 (C=O), 1687 (C=O), 1627 (C=N), 1120, 1096, 1041 (C—O) cm^{-1} . δ_{H} (DMSO- d_6): 3.79 (s, 2H, CH_2), 7.31—7.78 (m, 4H, ArH), 8.83 (br. s, 1H, OH), 10.41 (s, 1H, NH). m/z (%): 285 ($M^+ + 1$, 13.1), 284 (M^+ , 27.3), 242 (37.6), 214 (100), 188 (11.2), 145 (13.0), 91 (21.0), 63 (9.51). Anal. $\text{C}_{13}\text{H}_8\text{N}_4\text{O}_4$. Calcd: C, 54.94; H, 2.84; N, 19.70. Found: C, 54.63; H, 2.58; N, 19.47.

2-Benzalhydrazino-4-hydroxy-5H-[1]-benzopyrano-[4,3-d]-pyrimidin-5-one (**10**)

A mixture of **3** (0.01 mol) and benzaldehyde (0.01 mol) in acetic acid (70 mL) was heated under reflux for 6 h, then cooled. The solid obtained was filtered off, washed with water, dried and purified by recrystallization with dimethyl formamide to give **10** as colourless crystals, yield 72%, mp: 360°C. ν_{\max} (KBr): 3420—2779 (br. OH), 3203 (NH), 1720 (C=O), 1630 (C=N), 1124, 1051, 1031 (C—O) cm^{-1} . δ_{H} (DMSO- d_6): 6.81—7.79 (m, 10H, ArH and olefinic proton), 8.84 (br. s, 1H, OH), 10.51 (s, 1H, NH). m/z (%): 333 ($M^+ + 1$, 4.4), 332 (M^+ , 7.1), 331 ($M^+ - 1$, 2.6), 230 (79.6), 229 (100), 188 (30.4), 173 (1.6), 144 (7.7), 120 (5.2), 119 (15.5), 103 (15.4), 90 (11.6), 77 (22.1), 63 (9.8). Anal. $\text{C}_{18}\text{H}_{12}\text{N}_4\text{O}_3$. Calcd: C, 65.06; H, 3.64; N, 16.85. Found: C, 64.83; H, 3.52; N, 16.66.

5-Hydroxy-1-phenyl-6H-[1]-benzopyrano-[4,3-d]-1,

2,4-triazolo-[3,4-b]-pyrimidin-6-one (**11**)

A mixture of **10** (0.01 mol), bromine (0.01 mol) and fused sodium acetate (0.03 mol) in acetic acid (70 mL) was heated under reflux for 3—4 h, then cooled and poured onto water. The resulting product was filtered, washed with water, dried and purified by recrystallization with acetic acid to give **11** as yellow crystals, yield 74%, mp: 395°C. ν_{\max} (KBr): 3345—2651 (br. OH), 1719 (C=O), 1632 (C=N), 1095, 1057, 1029 (C—O) cm^{-1} . δ_{H} (DMSO- d_6): 7.10—7.81 (m, 9H, ArH), 8.83 (br. s, 1H, OH). m/z (%): 331 ($M^+ + 1$, 1.2), 330 (M^+ , 58.2), 253 (34.1), 227 (100), 185 (13.7), 145 (16.2), 144 (6.3), 120 (3.2), 119 (11.2), 103 (221), 91 (10.2), 77 (18.1). Anal. $\text{C}_{18}\text{H}_{10}\text{N}_4\text{O}_3$. Calcd: C, 65.46; H, 3.05; N, 16.96. Found: C, 65.33; H, 2.88; N, 16.58.

2-(Substituted) hydrazino-4-hydroxy-5H-[1]-benzopyrano-[4,3-d]-pyrimidin-5-one (**12** and **13**)

A mixture of **3** (0.01 mol), activated olefinic compound (such as β -toloyl-acrylic acid or ethyl α -cyano-*p*-methoxycinnamate) (0.01 mol) and triethyl amine (0.03 mol) was fused on a hot plate for 5—10 min. The mixture was added to boiling acetic acid (60 mL) and heated under reflux for 2—3 h, then cooled. The solid formed was filtered off, washed with water, dried and purified by crystallization with dimethyl formamide to give **12** or **13**.

Compound **12** as pale yellow crystals, yield 67%, mp: > 450°C. ν_{\max} (KBr): 3420—2268 (br. OH), 3251 (NH), 1724 (C=O), 1702 (C=O of carboxylic acid), 1687 (C=O), 1621 (C=N), 1062, 1010 (C—O) cm^{-1} . δ_{H} (DMSO- d_6): 2.41 (s, 3H, CH_3), 2.67 (d, $J = 1.4$ Hz, 2H, CH_2), 3.69 (t, $J = 1.2$ Hz, 1H, CH), 4.38 (s, 1H, NHCH), 7.11—7.78 (m, 8H, ArH), 8.83 (br. s, 1H, OH), 10.30 (s, 1H, NH), 12.01 (br. s, 1H, OH). m/z (%): 434 (M^+ , 105), 314 (17.4), 268 (100), 241 (1.1), 213 (6.5), 172 (5.6), 144 (3.5), 120 (0.6), 104 (18.8), 103 (10.3), 92 (2.8), 88 (8.7), 89 (2.9), 77 (8.9), 76 (15.5), 63 (10.9). Anal. $\text{C}_{22}\text{H}_{18}\text{N}_4\text{O}_6$. Calcd: C, 60.83; H, 4.18; N, 12.89. Found: C, 60.65; H, 3.88; N, 12.58.

Compound **13** as yellow crystals, yield 66%, mp: 374°C. ν_{\max} (KBr): 3410—2225 (br. OH), 1720 (C=

O), 1695 (C = O of acid), 1625 (C = N), 1095, 1057, 1025 (C—O) cm^{-1} . δ_{H} (DMSO- d_6): 2.81(d, $J = 1.4$ Hz, 2H, CH_2), 3.75(t, $J = 1.2$ Hz, 1H, CH), 3.86(s, 3H, OCH_3), 4.32(s, 1H, NHCH), 7.02—8.1(m, 8H, ArH), 8.84(br. s, 1H, OH), 10.21(s, 1H, NH), 11.83(br. s, 1H, OH). m/z (%): 423($\text{M}^+ + 1$, 3.6), 422(M^+ , 13.4), 362(4.7), 229(100), 188(30.1), 173(1.2), 145(4.1), 144(6.0), 133(8.2), 134(6.1), 116(3.4), 117(2.1), 91(13.4), 92(7.0), 89(5.3), 88(2.9), 63(10.0), 60(1.5). Anal. $\text{C}_{21}\text{H}_{18}\text{N}_4\text{O}_6$. Calcd: C, 59.72; H, 4.30; N, 13.26. Found: C, 59.49; H, 4.03; N, 13.06.

2-Malonyl-hydrazino-4-hydroxy-5H-[1]-benzopyrano-[4,3-d]-pyrimidin-5-one (14)

A mixture of **3** (0.01 mol), diethyl malonate (0.01 mol) and triethyl amine (0.03 mol) was fused on a hot plate for 5—10 min. The mixture was added to boiling acetic acid (50 mL) and heated under reflux for 2—3 h, then cooled. The resulting solid was filtered off, washed with water, dried and purified by crystallization with dimethyl formamide to give **14** as pale yellow crystals, yield 64%, mp: > 450°C. ν_{max} (KBr): 3430—2231(br. OH), 3225(NH), 1730—1702(C = O of pyrane and acid), 1685(C = O), 1623(C = N), 1147, 1105, 1035, 1004(C—O) cm^{-1} . δ_{H} (DMSO- d_6): 3.53(s, 2H, CH_2), 7.31—7.78(m, 4H, ArH), 8.85(br. s, 1H, OH), 10.08(s, 1H, NH), 10.25(s, 1H, NH) 12.01(br. s, 1H, OH). m/z (%): 331($\text{M}^+ + 1$; 1.10), 330(M^+ , 7.0), 286(19.6), 244(100), 224(8.4), 214(7.0), 213(11.0), 188(15.8), 187(2.7), 186(6.7), 145(7.0), 144(2.6), 116(9.8), 115(5.4), 102(15.7), 91(13.4), 89(7.3), 76(12.7), 75(10.3), 63(15.8). Anal. $\text{C}_{14}\text{H}_{10}\text{N}_4\text{O}_6$. Calcd: C, 50.92; H, 3.05; N, 16.96. Found: C, 50.62; H, 3.12; N, 16.59.

2-(Monoacetyl or diacetyl) hydrazino-4-hydroxy-5H-[1]-benzopyrano-[4,3-d]-pyrimidin-5-one (15 or 16)

A mixture of **3** (0.01 mol) and acetyl chloride (0.02 mol) in acetic acid (60 mL) was heated under reflux for 2—3 h. The solid formed on hot was filtered

off, dried and purified by crystallization with dimethyl formamide to give **15** as colourless crystals, yield 32%, mp: 301°C. ν_{max} (KBr): 3380—2513(br. OH), 3225(NH), 1720, 1687(C = O), 1621(C = N), 1034, 1012(C—O) cm^{-1} . δ_{H} (DMSO- d_6): 2.10(s, 3H, CH_3), 7.30—7.78(m, 4H, ArH), 8.84(br. s, 1H, OH), 10.02(s, 1H, NH), 10.24(s, 1H, NH). m/z (%): 287($\text{M}^+ + 1$, 12.6), 286(M^+ , 14.4), 271(1.6), 244(100), 229(12.7), 215(60.0), 214(11.6), 188(16.3), 187(3.2), 186(7.3), 158(5.7), 145(7.4), 144(3.6), 120(18.6), 119(11.4), 103(11.7), 102(12.5), 91(11.3), 77(9.9), 76(9.6), 63(14.0). Anal. $\text{C}_{13}\text{H}_{10}\text{N}_4\text{O}_4$. Calcd: C, 54.55; H, 3.52; N, 19.57. Found: C, 54.31; H, 3.27; N, 19.41.

Pouring the filtrate into water yielded the crude product, which was filtered and washed with water, dried and purified by recrystallization with ethanol to give **16** as colourless crystals, yield 51%, mp: 232°C. ν_{max} (KBr): 3370—2515(br. OH), 3174(NH), 1732, 1718, 1683(C = O), 1624(C = N), 1132, 1116, 1020(C—O) cm^{-1} . δ_{H} (DMSO- d_6): 2.13(s, 6H, 2 \times COCH_3), 7.31—7.79(m, 4H, ArH), 8.83(br. s, 1H, OH), 10.12(s, 1H, NH). m/z (%): 329($\text{M}^+ + 1$, 1.1), 328(M^+ , 1.2), 313(1.9), 286(27.0), 229(100), 215(27.3), 213(4.0), 188(6.5), 187(1.1), 186(4.9), 145(3.8), 144(1.1), 120(6.9), 119(4.7), 114(3.5), 103(5.3), 102(4.7), 91(5.0), 63(4.9). Anal. $\text{C}_{15}\text{H}_{12}\text{N}_4\text{O}_5$. Calcd: C, 54.88; H, 3.68; N, 17.06. Found: C, 54.61; H, 3.37; N, 16.79.

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