

CYCLOCONDENSATIONS OF β -AROYLACRYLIC ACIDS WITH HETEROCYCLIC O-DIAMINES

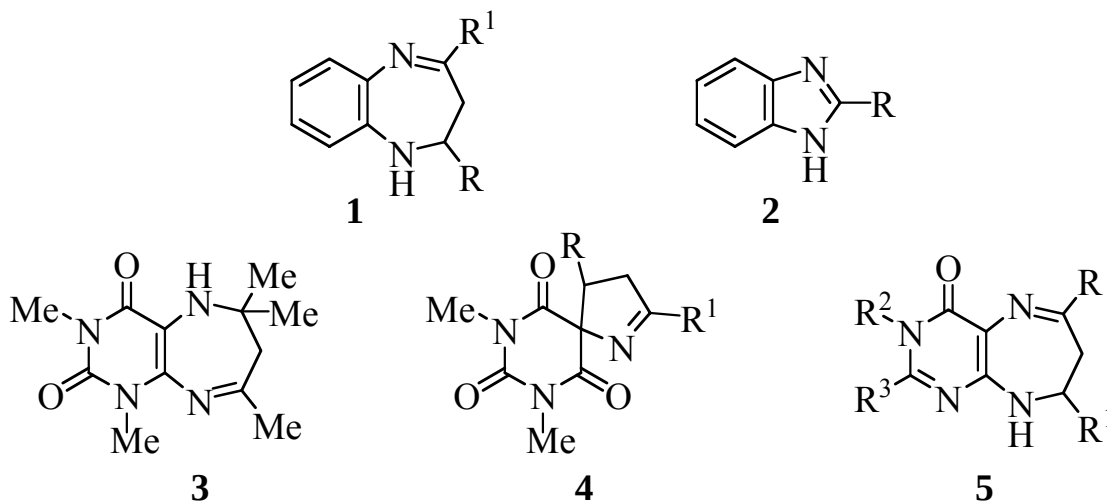
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Abstract – Reaction of β -aroylacrylic acids (**6a–c**) with 2,3-diaminopyridine (**7**), 5,6-diamino-1,3-dimethyluracil (**8**) and 2,5,6-triamino-4-oxypyrimidine (**9**) was studied. 1,3-Dimethyl-5,8-dihydro-1*H*,3*H*,6*H*-pteridine-2,4,7-trione (**11c**) and 2-amino-4-hydroxy-6-(2-oxo-2-phenylethyl)-5,8-dihydro-6*H*-pteridin-7-one (**13a**) were rearranged into pteridin-6-ylideneacetic acids (**12c**) and (**14a**) respectively. Reaction of α,β -dibromo- β -benzoylpropionic acid (**15**) with diamine (**8**) led to 8-benzoylpurine (**17**) *via* the step of the formation of enaminoketone (**16**).

INTRODUCTION

Reactions of α,β -unsaturated ketones with aromatic and heterocyclic *o*-diamines attract attention due to their ability of forming compounds with different structure. Among them 1,5-benzodiazepine (**1**) and benzoimidazole (**2**) (reaction with *o*-phenylenediamine),¹ pyrimidodiazepines (**3**) or spirosystems (**4**) (reaction with 5,6-diamino-1,3-dimethyluracil),^{2,3} pyrimidodiazepines (**5**) (reaction with other derivatives of diaminouracil).^{4,5} The structures of compounds (**1**, **3** and **4**) were confirmed by X-Ray analysis.

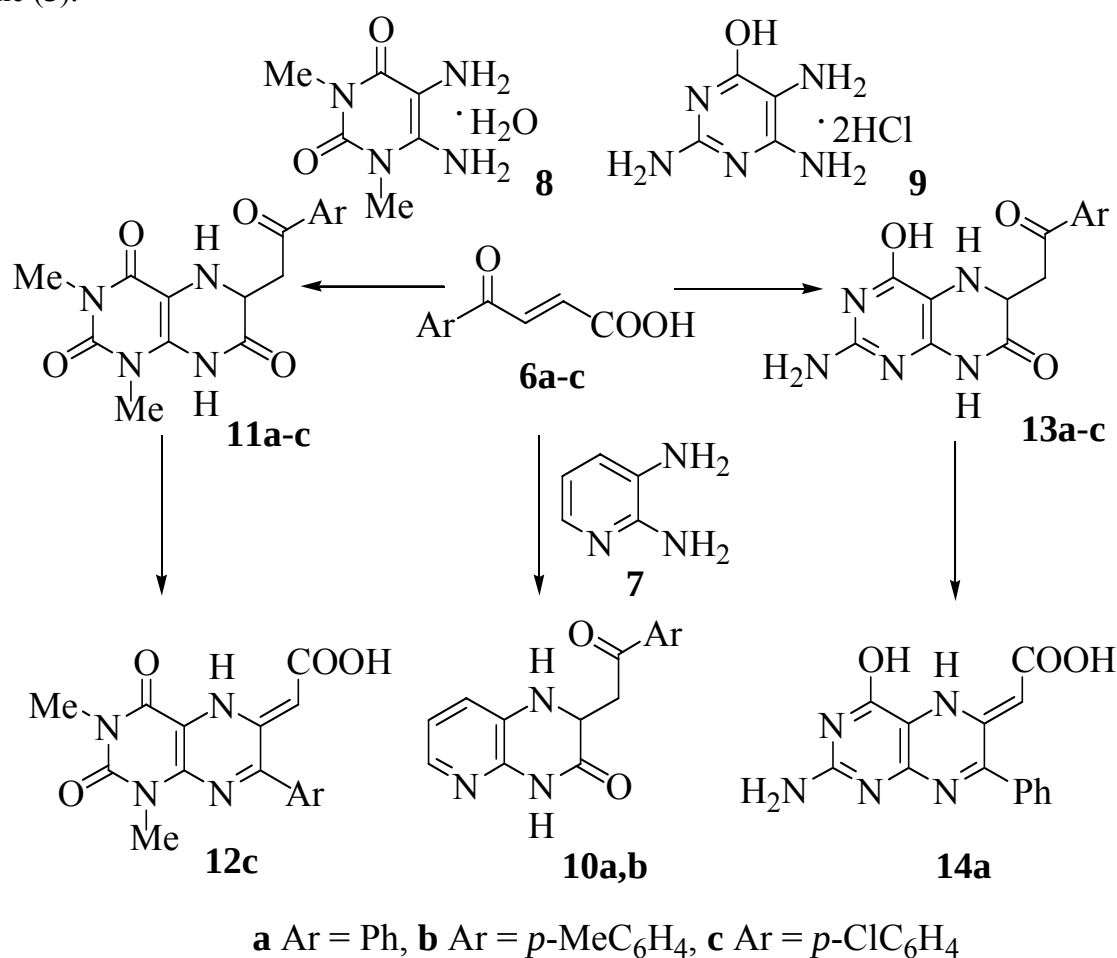


Scheme 1

β -Aroylacrylic acids are simple and polyfunctional building blocks for synthesis of heterocycles. Earlier we reported⁶ that β -aroylacrylic acids form 3-phenacylquinoxalin-2-one derivatives in the reaction with substituted *o*-phenylenediamine. The reaction proceeds *via* the stage of α -addition to enone then subsequent cyclization to the thermodynamically stable quinoxalone ring.

RESULTS AND DISCUSSION

In this study we examined the products of reaction of β -aroylacrylic acids (**6a–c**) with heterocyclic diamines: 2,3-diaminopyridine (**7**), 5,6-diamino-1,3-dimethyluracil (**8**) and 2,5,6-triamino-4-hydroxypyrimidine (**9**).



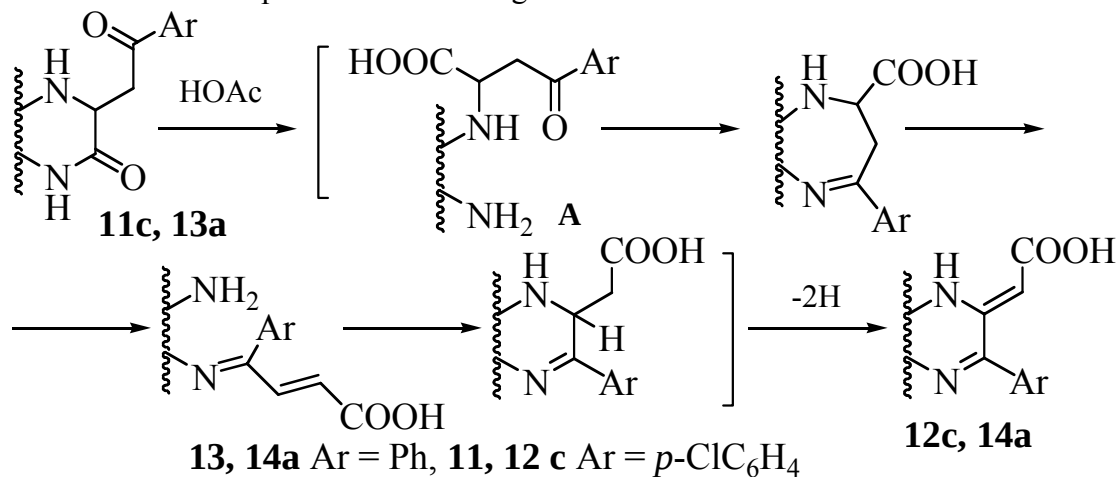
Scheme 2

Diamine (**7**) reacted with acids (**6a,b**) very easy and formed products (**10a,b**) with good yields, but in the case of diamines of pyrimidine series the yield of products (**11a–c**) and (**13a–c**) was lower. Compounds (**13a–c**) were isolated only after boiling of acids (**6a–c**) and HCl salt of diamine (**9**) in methanol with addition of HOAc for 25 h.

The structures of compounds (**10a,b**, **11a–c**, **13a–c**) were confirmed by ¹H NMR, IR spectroscopic data and elemental analyses (EXPERIMENTAL). Thus, in the IR spectra sharp absorptions were observed due

to the stretching vibration of all carbonyl groups and absorptions of unassociated imino groups at 3380–3430 cm^{-1} . Absorption bands of endocyclic NH–CO groups exhibited only in the spectra of compounds (**11a–c**) at 3200 cm^{-1} ; in the case of **10a,b** and **13a–c** they, probably, shifted to low frequency region and were broad because of the considerable molecular association. The ^1H NMR spectra of compounds (**10a,b**, **11a–c** and **13a–c**) exhibited clear signals due to protons of aromatic ring at 8.09–6.78 ppm, singlets due to protons of the NH groups at $\delta = 6.18$ –6.58 and 8.72–10.77 ppm which disappeared after exchange with deuterium, a group of signals due to protons of CH_2 –CH unit with a typical ABX structure (two doublets and doublet of doublets: J_{AB} 11.6 Hz, J_{AX} 2.5 Hz, J_{BX} 4.6–5.0 Hz (**10a,b**); J_{AB} 0 Hz, J_{AX} 2.4 Hz, J_{BX} 5.0–5.4 Hz (**11a–c**); J_{AB} 16.0–18.0 Hz, J_{AX} 4.0–4.8 Hz, J_{BX} 5.8–6.0 Hz (**13a–c**)), and (in **13a–c**) singlets due to protons of the NH and OH groups of pyrimidine ring (6.11–6.16 and 10.35–10.58 ppm respectively). Thus, ^1H NMR spectra showed formation of pyrazine ring fused by either pyridine (**10a,b**) or pyrimidine (**11a–c**, **13a–c**) fragment. Therefore, heterocyclic diamines are similar to *o*-phenylenediamines in the reactions with β -aroylacrylic acids, and react with enone system *via* 1,4-nucleophilic addition. Obviously, it can be concluded that the direction of nucleophilic addition (1,2- or 1,4-) was determined, first of all, by the polarization of ethylenic bond: in the case of strongly polarized olefines 1,4-addition is always realized, irrespectively of the basicity of amino groups of starting diamine. The non-equivalence of amino groups of amines (**7–9**) rises the question about the direction of interaction. It is known^{3,4} that the amino group of the position 5 in diamines (**8**) and (**9**) is more basic, and therefore just this group takes part in the step of α -addition leading to pteridines (**11a–c**) and (**13a–c**). Analogously, more basic 3-amino group of diamine (**7**)⁷ promoted the formation of pyrido[2,3-*b*]pyrazin-3-ones (**10a,b**).

It was proved that compounds (**11c**) and (**13a**) rearranged quite easily into pteridines (**12c**) and (**14a**) respectively while pyrazinones (**10a,b**) remained invariable under the same conditions. It was supported that the formation of **12c** and **14a** proceeded according to Scheme 3.



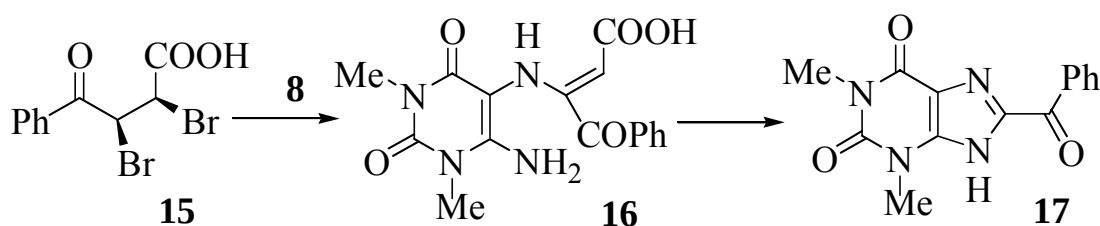
Scheme 3

Such a stability of **10a** is likely to be the result of difficult formation of an intermediate diazepine ring due to low basicity of amino group of the intermediate **A**, and this is the additional argument in favor of the proposed mechanism.

Recently, we have also shown the possibility of rearrangement of 3-phenacylquinoxalin-2-ones into 3-aryl-2-carboxymethylidenequinoxalines.⁸

The structure of compounds (**12c**) and (**14a**) was identified using ¹H NMR spectra and confirmed by IR spectra (EXPERIMENTAL). An additional argument for the structure (**14a**) is the appearance of two broad singlets with close chemical shifts ($\delta = 10.30$ and 10.28 ppm) in the ¹H NMR spectrum. This spectral data were typical for a dihydropteridinols and were observed in the case of spatially close OH and NH groups which take part in rapid exchange.⁹ According to steric consideration acids (**12c**) and (**14a**) exist in the form of *Z*-isomers.

We also examined the interaction of α,β -dibromo- β -benzoylpropionic acid (**15**) with diamine (**8**). Heating (about 3 min) of starting substances resulted in the formation of enaminoketone (**16**) which existed, similarly to **12c** and **14a**, in the form of *Z*-isomer. The structure of **16** was supported by spectral data. The most informative is ¹H NMR spectrum in which signals of all proton containing groups were observed. In the MS spectrum of **16** the molecular ion was absent but the peak of $[M - CO_2]^+$ ion with m/z 300 was quite intensive; the $[CO - Ph]^+$ ion peak was of maximal intensity. The structure of **16** indicated that the reaction proceeds *via* the step of formation of α -bromo- β -benzoylacrylic acid then subsequent nucleophilic addition of C5-NH₂ group at more activated β -position; the process was accompanied by the elimination of HBr.



Scheme 4

Heating of **16** led to the formation of 8-benzoylpurine (**17**) with the loss of HOAc molecule. Compound (**17**) was formed in one stage without the elimination of enaminoketone (**16**) also.

EXPERIMENTAL

General: All melting points were taken on Kofler melting point apparatus and are uncorrected. IR (KBr): Specord 75-IR spectrophotometer. ¹H NMR: Varian Mercury VX-200, Bruker AM-300, ¹³C NMR: Varian Mercury 400, DMSO-d₆ as solvent at 25°C. MS: FINNIGAN MAT. INCOS 50 (70 eV). Microanalyses: LECO CHNS-900 elemental analyzer.

General Procedure for the Preparation of the 2-(2-Oxo-2-arylethyl)-1,4-dihydro-2H-pyrido[2,3-*b*]pyrazin-3-ones (10a,b) A solution of **7** (0.2 g, 1.80 mmol) and the acid (**6a,b**) (1.80 mmol) in ethanol (20 mL) was refluxed for 1 h and the reaction was monitored by TLC. After cooling, the precipitate was filtered off and crystallized from ethanol.

2-(2-Oxo-2-phenylethyl)-1,4-dihydro-2H-pyrido[2,3-*b*]pyrazin-3-one (10a) White needles, yield 67%, mp 206°C. IR (KBr) cm^{-1} : 1662, 1672, 3390. ^1H NMR (300 MHz, DMSO- d_6) δ : 3.40 (dd, 1H, $J = 11.6$ and 2.6 Hz, CH_A), 3.60 (dd, 1H, $J = 11.6$ and 4.6 Hz, CH_B), 4.45 (dd, 1H, $J = 2.6$ and 4.6 Hz, CH_X), 6.21 (s, 1H, NH), 6.78 (q, 1H, $J = 8.1$ and 5.0 Hz, pyrid. H), 6.99 (d, 1H, $J = 7.8$ Hz, pyrid. H), 7.55 (m, 3H, aromat. H), 7.67 (d, $J = 7.5$ Hz, 1H, pyrid. H), 8.01 (d, 2H, $J = 7.5$ Hz, aromat. H), 10.77 (s, 1H, NHCO). *Anal.* Calcd for $\text{C}_{15}\text{H}_{13}\text{N}_3\text{O}_2$: C, 67.41; H, 4.90; N, 15.72. Found: C, 67.45; H, 4.92; N, 15.69.

2-(2-Oxo-2-*p*-tolylethyl)-1,4-dihydro-2H-pyrido[2,3-*b*]pyrazin-3-one (10b) White needles, yield 73%, mp 212°C. IR (KBr) cm^{-1} : 1672, 1682, 3390. ^1H NMR (300 MHz, DMSO- d_6) δ : 2.39 (s, 3H, CH_3), 3.39 (dd, 1H, $J = 11.6$ and 2.5 Hz, CH_A), 3.56 (dd, 1H, $J = 11.6$ and 5.0 Hz, CH_B), 4.43 (dd, 1H, $J = 2.5$ and 5.0 Hz, CH_X), 6.18 (s, 1H, NH), 6.77 (q, 1H, $J = 8.0$ and 4.9 Hz, pyrid. H), 6.99 (d, 1H, $J = 7.5$ Hz, pyrid. H), 7.34 (d, 2H, $J = 7.8$ Hz, aromat. H), 7.56 (d, 1H, $J = 4.5$ Hz, pyrid. H), 7.90 (d, 2H, $J = 8.4$ Hz, aromat. H), 10.74 (s, 1H, NHCO). *Anal.* Calcd for $\text{C}_{16}\text{H}_{15}\text{N}_3\text{O}_2$: C, 68.31; H, 5.37; N, 14.94. Found: C, 68.35; H, 5.33; N, 14.97.

General Procedure for the Preparation of the 1,3-Dimethyl-6-(2-oxo-2-arylethyl)-5,8-dihydro-1H,3H,6H-pteridine-2,4,7-triones (11a–c) A solution of **8** (0.510 g, 3.00 mmol), the acid (3.20 mmol) (**6a–c**) and concentrated HCl (0.06 mL) in ethanol (7 mL) was refluxed for 2–3 h and the reaction was monitored by TLC. After cooling, the product was filtered off and crystallized from ethanol.

1,3-Dimethyl-6-(2-oxo-2-phenylethyl)-5,8-dihydro-1H,3H,6H-pteridine-2,4,7-trione (11a) Yellow crystals, yield 66%, mp 219°C. IR (KBr) cm^{-1} : 1629, 1649, 1675, 3196, 3383. ^1H NMR (200 MHz, DMSO- d_6) δ : 3.16 (s, 3H, CH_3), 3.20 (s, 3H, CH_3), 3.49 (d, 1H, $J = 2.4$ Hz, CH_A), 3.51 (d, 1H, $J = 5.2$ Hz, CH_B), 4.51 (dd, 1H, $J = 2.4$ and 5.2 Hz, CH_X), 7.08 (br s, 1H, NH), 7.46–7.68 (m, 3H, aromat. H), 7.94 (d, 2H, $J = 8.0$ Hz, aromat. H), 8.90 (br s, 1H, NHCO). *Anal.* Calcd for $\text{C}_{16}\text{H}_{16}\text{N}_4\text{O}_4$: C, 58.53; H, 4.91; N, 17.06. Found: C, 58.57; H, 4.89; N, 17.01.

1,3-Dimethyl-6-(2-oxo-2-*p*-tolylethyl)-5,8-dihydro-1H,3H,6H-pteridine-2,4,7-trione (11b) Yellow

crystals, yield 44%, mp 242°C. IR (KBr) cm^{-1} : 1629, 1645, 1675, 3210, 3383. ^1H NMR (200 MHz, DMSO- d_6) δ : 2.36 (s, 3H, CH_3), 3.15 (s, 3H, CH_3), 3.19 (s, 3H, CH_3), 3.45 (d, 1H, $J = 2.4$ Hz, CH_A), 3.47 (d, 1H, $J = 5.4$ Hz, CH_B), 4.45 (dd, 1H, $J = 2.4$ and 5.4 Hz, CH_X), 7.11 (br s, 1H, NH), 7.31 (d, 2H, $J = 8$ Hz, aromat. H), 7.85 (d, 2H, $J = 8$ Hz, aromat. H), 9.00 (br s, 1H, NHCO). *Anal.* Calcd for $\text{C}_{17}\text{H}_{18}\text{N}_4\text{O}_4$: C, 59.64; H, 5.30; N, 16.37. Found: C, 59.65; H, 5.26; N, 16.40.

6-[2-(4-Chlorophenyl)-2-oxoethyl]-1,3-dimethyl-5,8-dihydro-1H,3H,6H-pteridine-2,4,7-trione (11c)

Yellow crystals, yield 53%, mp 198°C. IR (KBr) cm^{-1} : 1629, 1646, 1675, 3196, 3376. ^1H NMR (200 MHz, DMSO- d_6) δ : 3.15 (s, 3H, CH_3), 3.20 (s, 3H, CH_3), 3.47 (d, 1H, $J = 2.3$ Hz, CH_A), 3.50 (d, 1H, $J = 5.0$ Hz, CH_B), 4.45 (dd, 1H, $J = 2.3$ and 5.0 Hz, CH_X), 7.12 (br s, 1H, NH), 7.57 (d, 2H, $J = 8$ Hz, aromat. H), 7.96 (d, 2H, $J = 8$ Hz, aromat. H), 9.05 (br s, 1H, NHCO). *Anal.* Calcd for $\text{C}_{16}\text{H}_{15}\text{N}_4\text{O}_4\text{Cl}$: C, 52.97; H, 4.17; N, 15.44. Found C, 52.94; H, 4.22; N, 15.46.

General Procedure for the Preparation of the 2-Amino-4-hydroxy-6-(2-oxo-2-arylethyl)-5,8-dihydro-6H-pteridin-7-ones (13a–c) A solution of **9** (0.28 g, 1.30 mmol), the acid (1.30 mmol) (**6a–c**) and glacial acetic acid (7 mL) in methanol (50 mL) was refluxed for 25 h, cooled and neutralized with a 25% NH_3 solution. After the methanol was removed, the solid was filtered off and crystallized from ethanol.

2-Amino-4-hydroxy-6-(2-oxo-2-phenylethyl)-5,8-dihydro-6H-pteridin-7-one (13a) Red crystals, yield 59%, mp $>300^\circ\text{C}$. IR (KBr) cm^{-1} : 1635, 1682, 3210, 3123, 3250, 3430. ^1H NMR (200 MHz, DMSO- d_6) δ : 3.34 (dd, 1H, $J = 17.2$ and 4.8 Hz, CH_A), 3.54 (dd, 1H, $J = 17.2$ and 5.8 Hz, CH_B), 4.39 (dd, 1H, $J = 4.8$ and 5.8 Hz, CH_X), 6.12 (br s, 2H, NH_2), 6.55 (br s, 1H, NH), 7.46–7.65 (m, 3H, aromat. H), 7.93 (d, 2H, $J = 7.4$ Hz, aromat. H), 8.75 (br s, 1H, NHCO), 10.35 (br s, 1H, OH). *Anal.* Calcd for $\text{C}_{14}\text{H}_{13}\text{N}_5\text{O}_3$: C, 56.18; H, 4.38; N, 23.40. Found: C, 56.21; H, 4.35; N, 23.46.

2-Amino-4-hydroxy-6-(2-oxo-2-*p*-tolylethyl)-5,8-dihydro-6H-pteridin-7-one (13b) Red crystals, yield 42%, mp $>300^\circ\text{C}$. IR (KBr) cm^{-1} : 1640, 1675, 3430. ^1H NMR (200 MHz, DMSO- d_6) δ : 2.35 (s, 3H, CH_3), 3.30 (dd, 1H, $J = 18.0$ and 4.0 Hz, CH_A), 3.51 (dd, 1H, $J = 18.0$ and 6.0 Hz, CH_B), 4.37 (dd, 1H, $J = 4.0$ and 6.0 Hz, CH_X), 6.16 (br s, 2H, NH_2), 6.57 (br s, 1H, NH), 7.29 (d, 2H, $J = 7.6$ Hz, aromat. H), 7.82 (d, 2H, $J = 7.4$ Hz, aromat. H), 8.87 (br s, 1H, NHCO), 10.58 (br s, 1H, OH). *Anal.* Calcd for $\text{C}_{15}\text{H}_{15}\text{N}_5\text{O}_3$: C, 57.50; H, 4.83; N, 22.35. Found: C, 57.47; H, 4.86; N, 22.30.

2-Amino-6-[2-(4-chlorophenyl)-2-oxoethyl]-4-hydroxy-5,8-dihydro-6H-pteridin-7-one (13c) Red crystals, yield 43%, mp >300°C. IR (KBr) cm^{-1} : 1642, 1655, 3410. ^1H NMR (200 MHz, DMSO- d_6) δ : 3.32 (dd, 1H, $J = 16.0$ and 4.0 Hz, CH_A), 3.52 (dd, 1H, $J = 16.0$ and 6.0 Hz, CH_B), 4.38 (dd, 1H, $J = 4.0$ and 6.0 Hz, CH_X), 6.11 (br s, 2H, NH_2), 6.50 (br s, 1H, NH), 7.56 (d, 2H, $J = 8.2$ Hz, aromat. H), 7.94 (d, 2H, $J = 8.4$ Hz, aromat. H), 8.72 (br s, 1H, NHCO), 10.37 (br s, 1H, OH). *Anal.* Calcd for $\text{C}_{15}\text{H}_{15}\text{N}_5\text{O}_3$: C, 50.39; H, 3.62; N, 20.98. Found: C, 50.44; H, 3.67; N, 20.96.

General Procedure for the Preparation of the Pteridin-6-ylideneacetic acid (12c and 14a) A solution of **11c** (0.22 g, 0.60 mmol) or **13a** (0.18 g, 0.60 mmol) in glacial acetic acid (15 mL) was heated for 3 h to reflux, cooled and poured out on ice. The products was filtered off and crystallized from ethanol.

2-[7-(4-Chlorophenyl)-1,3-dimethyl-2,4-dioxo-1,3,5,6-tetrahydropteridin-6-ylidene]acetic acid (12c) Red crystals, yield 52%, mp >300°C. IR (KBr) cm^{-1} : 1649, 1665, 1695, 3523. ^1H NMR (300 MHz, DMSO- d_6) δ : 3.26 (s, 3H, CH_3), 3.55 (s, 3H, CH_3), 6.83 (s, 1H, CH), 7.59 (d, 2H, $J = 7.5$ Hz, aromat. H), 7.98 (d, 2H, $J = 7.8$ Hz, aromat. H), 12.50 (br s, 1H, NH), 14.70 (br s, 1H, OH). *Anal.* Calcd for $\text{C}_{16}\text{H}_{13}\text{N}_4\text{O}_4\text{Cl}$: C, 53.27; H, 3.63; N, 15.53. Found: C, 53.33; H, 3.66; N, 15.49.

(2-Amino-4-hydroxy-5H-pteridin-6-ylidene)acetic acid (14a) Biscuit crystals, yield 53%, mp >300°C. IR (KBr) cm^{-1} : 1636, 1664, 1720, 3572. ^1H NMR (300 MHz, DMSO- d_6) δ : 6.52 (br s, 2H, NH_2), 6.82 (s, 1H, CH), 7.50 – 7.62 (m, 3H, aromat. H), 7.98 (d, 2H, $J = 7.5$ Hz, aromat. H), 11.28 (br s, 1H, phenol. OH), 11.30 (br s, 1H, NH), 13.05 (s, 1H, COOH). ^{13}C NMR (400 MHz, DMSO- d_6) δ : 91.8, 101.4, 127.9, 129.4, 132.9, 138.9, 141.3, 146.9, 152.7, 154.6, 155.4, 190.5. *Anal.* Calcd for $\text{C}_{14}\text{H}_{11}\text{N}_5\text{O}_3$: C, 56.57; H, 3.73; N, 23.56. Found C, 56.61; H, 3.75; N, 23.51.

3-(6-Amino-1,3-dimethyl-2,4-dioxo-1,2,3,4-tetrahydropyrimidin-5-ylamino)-4-oxo-4-phenyl-2-butenic acid (16) The solution of acid (**15**) (0.40 g, 1.20 mmol), diamine (**8**) (0.17 g, 1.00 mmol) and triethylamine (0.27 mL, 2.00 mmol) in ethanol (7 mL) was heated for 3–5 min. The solid formed during the heating was filtered off and washed by warm water. Yellow crystals, yield 0.112 g, 33%, mp 205°C. IR (KBr) cm^{-1} : 1615, 1690, 3210, 3383. ^1H NMR (200 MHz, DMSO- d_6) δ : 3.19 (s, 3H, CH_3), 3.35 (s, 3H, CH_3), 5.85 (s, 1H, CH), 6.20 (br s, 1H, NH), 6.36 (br s, 2H, NH_2), 7.80 – 7.83 (m, 5H, aromat. H), 10.50 (br s, 1H, COOH). MS (70 eV) m/z (%): 300 (87), 195 (71), 181 (78), 180 (98), 110 (55), 105 (100), 95 (65), 77 (83), 68 (51), 51 (57). *Anal.* Calcd for $\text{C}_{16}\text{H}_{16}\text{N}_4\text{O}_5$: C, 55.81; H, 4.68; N, 16.27. Found C, 55.86; H, 3.63; N, 16.22.

8-Benzoyl-1,3-dimethylpurine-2,6-dione (17). Method A. The solution of **16** (0.10 g, 0.29 mmol) in ethanol/ethyl acetate (5:1) solvent (12 mL) was refluxed for 2 h. After cooling, the precipitate was filtered off and crystallized from ethanol. Yellow crystals, yield 0.037 g, 44%, mp 285°C. **Method B.** The solution of **15** (0.40 g, 1.20 mmol) and triethylamine (0.27 mL, 2.00 mmol) in ethanol (10 mL) was heated for 10 min. Then **3** (0.17 g, 1.00 mmol) was added into the reaction mixture and refluxed for 3 h. The reaction mixture was treated up as mentioned previously. Yellow crystals, yield 0.18 g, 64%, mp 285°C. IR (KBr) cm^{-1} : 1642, 1665, 1699, 3416. ^1H NMR (200 MHz, DMSO- d_6) δ : 3.35 (s, 3H, CH_3), 3.64 (s, 3H, CH_3), 7.58 (m, 3H, aromat. H), 8.30 (m, 2H, aromat. H), 9.20 (br s, 1H, NH). *Anal.* Calcd for $\text{C}_{14}\text{H}_{12}\text{N}_4\text{O}_3$: C, 59.15; H, 4.25; N, 19.71. Found: C, 59.16; H, 4.22; N, 19.75.

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