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A Convenient Synthesis of Novel Derivatives of Pyrido[2,3-*d*][1,2,4] triazolo[4,3-*a*]pyrimidine-5,6-dione

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Summary. Reaction of 6-Amino-2-thiouracil with hydrazonoyl halides yielded regioselectively 7-amino-1,3-disubstituted-1,2,4-triazolo[4,3-*a*]pyrimidine derivatives. Upon treatment with methyl (*Z*)-2-benzoylamino-3-dimethylaminopropenoate, the corresponding methyl (*Z*)-2-benzoylamino-3-([1,2,4]triazolo[4,3-*a*]pyrimidin-7-yl)-amino propenoates were obtained which cyclized in the presence of sodium ethoxide to afford novel derivatives of pyrido[2,3-*d*][1,2,4]triazolo[4,3-*a*]pyrimidine-5,6-(1*H*,8*H*)-diones.

Keywords. Pyrimidines; Synthetic methods; 1,2,4-Triazolopyrimidinone; Propenoate ester; Pyrido-1,2,4-triazolo-pyrimidinedione.

Introduction

Triazolopyrimidines have been reported to exhibit *in vivo* leishmanicidal activity against the amastigate stage of *Leishmania donovani* [1, 2] and cardiovascular activity [3, 4]. They are cardiotonics, coronary vasadilators, and they have antihypertensive properties [5]. They act against *Aspergillus* and *Pencicillium* species [6] and have been tested as microbicidal and bioregulator agents [7]. Literature protocols, applicable to the synthesis of 1,2,4-triazolo[4,3-*a*]pyrimidines have so far been confined in general to the condensation of 2-hydrazinopyrimidin-4(3*H*)-ones with carboxylic acids or their derivatives [1] or the oxidative cyclization of 2-arylmethylene hydrazinopyrimidin-4(3*H*)-ones with ferric chloride [8]. The 2-hydrazinopyrimidin-4(3*H*)-ones are usually prepared *via* two steps from 2-thiouracil [8].

In this paper a facile one-pot synthesis for some new derivatives of [1,2,4]triazolo[4,3-*a*]pyrimidines is disclosed. Furthermore, we report the use of some of the latter for the synthesis of novel derivatives of pyrido[2,3-*d*]-1,2,4-triazolo[4,3-*a*]pyrimidine-5,6-(1*H*,8*H*)-diones. So far, only one example of pyrido[1,2,4]triazolopyrimidines has been reported in the literature [9].

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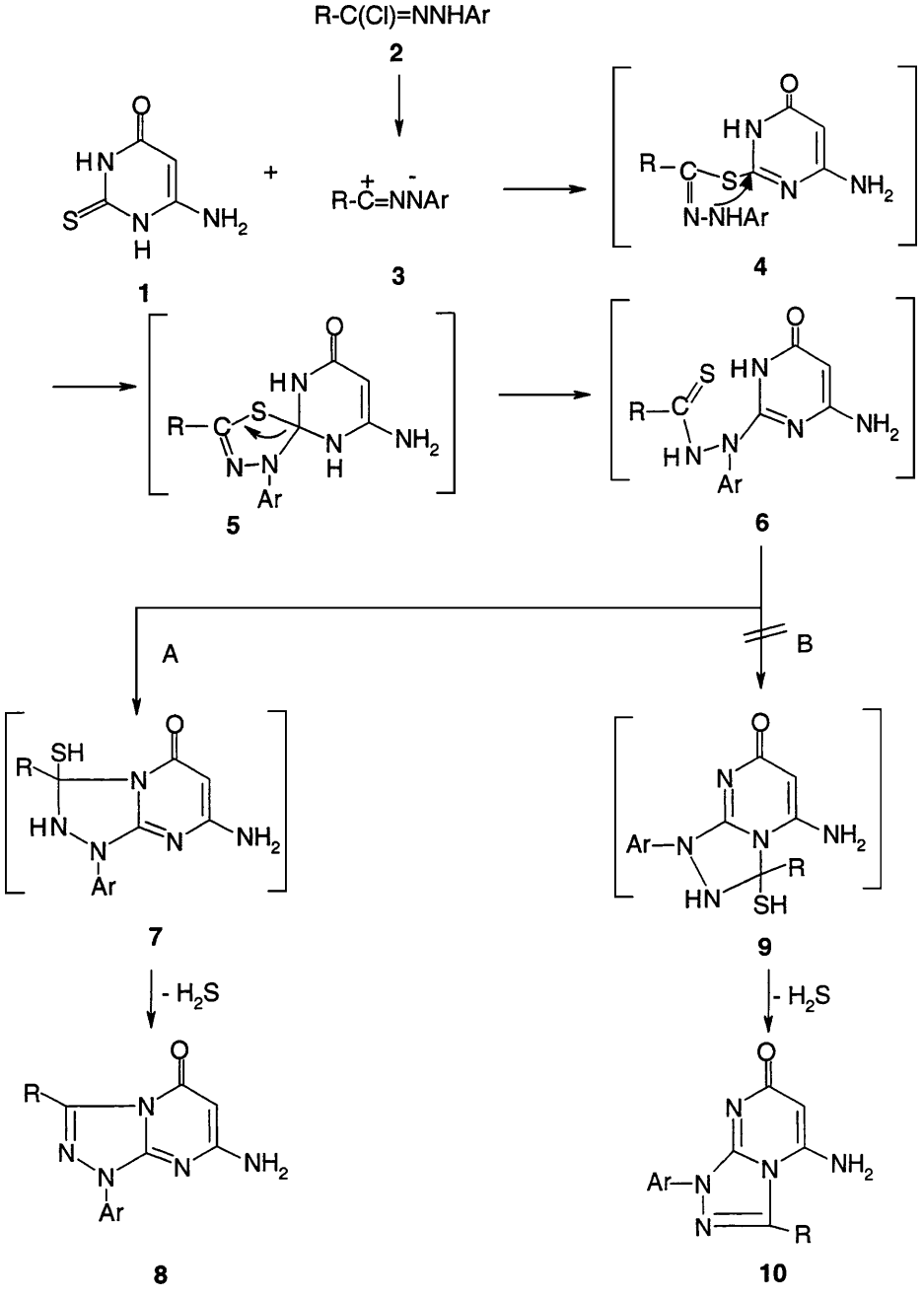
Results and Discussion

L. Grubert [10] has reported that heating of nitrilimines with 1-methyl-4-phenyl-pyrimidine-2-thione in benzene and triethylamine afforded spiro[pyrimidine-2(1*H*),2′(3*H*′)[1,3,4]thiadiazole]. When investigating the reaction of nitrilimines **3**, which were generated *in situ* from hydrazonoyl halides **2** and triethylamine with



Scheme 1. *R*/*Ar*: **a**, C_6H_5/C_6H_5; **b**, CH_3CO/C_6H_5; **c**, $EtOCO/C_6H_5$; **d**, C_6H_5NHCO/C_6H_5; **e**, C_6H_5CO/C_6H_5; **f**, CH_3CO/C_6H_4-4-CH_3; **g**, C_6H_5NHCO/C_6H_4-4-CH_3

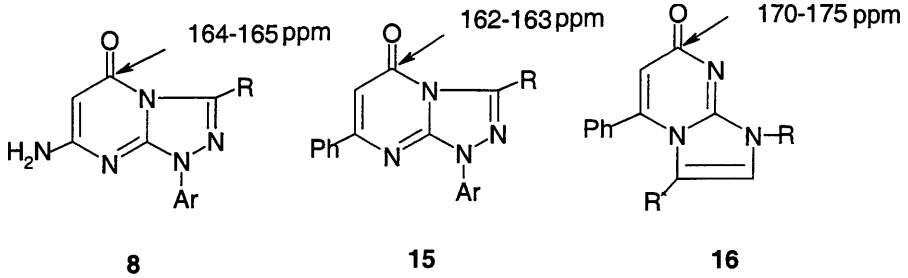
6-amino-2-thiouracil (**1**) in refluxing dioxane or sodium ethoxide in ethanol at room temperature, a single product of a regioselective reaction was isolated. The compound was characterized as 7-amino-1,3-disubstituted-[1,2,4]triazolo- [4,3-*a*]pyrimidin-5(1*H*)-one (**8**) (Scheme 1).

The reaction pathway accounting for the formation of **8** from **1** and **3** is outlined in Scheme 1. It is proposed that the reaction involves an initial 1,3-addition to give thiohydrazonate esters **4** which undergo a *Smiles* rearrangement to the thiohydrazides **6** *via* intermediates **5**. The latter then cyclize with concurrent elimination of hydrogen sulfide to give the final product. As outlined in Scheme 1, there are two possible routes (A and B) for the cyclization of **6** that will lead to **8** and **10**.

The actual structure of the products was found to resemble that of **8** by their analytical and spectroscopic data (IR, ^{1}H, ^{13}C NMR and MS). For example, the carbonyl stretching frequencies of **8** (1680–1700 cm^{-1}) were found to be similar to those of pyrimidines of structure **15** (1680–1690 cm^{-1}) and not **16** (1640–1660 cm^{-1}) [11, 12]. Furthermore, the carbonyl resonance of the products was found at 164–165 ppm similar to that of **15** [12] (Fig. 1). On the basis of these evidences, the products were assigned structure **8**, and structure **10** was discarded.

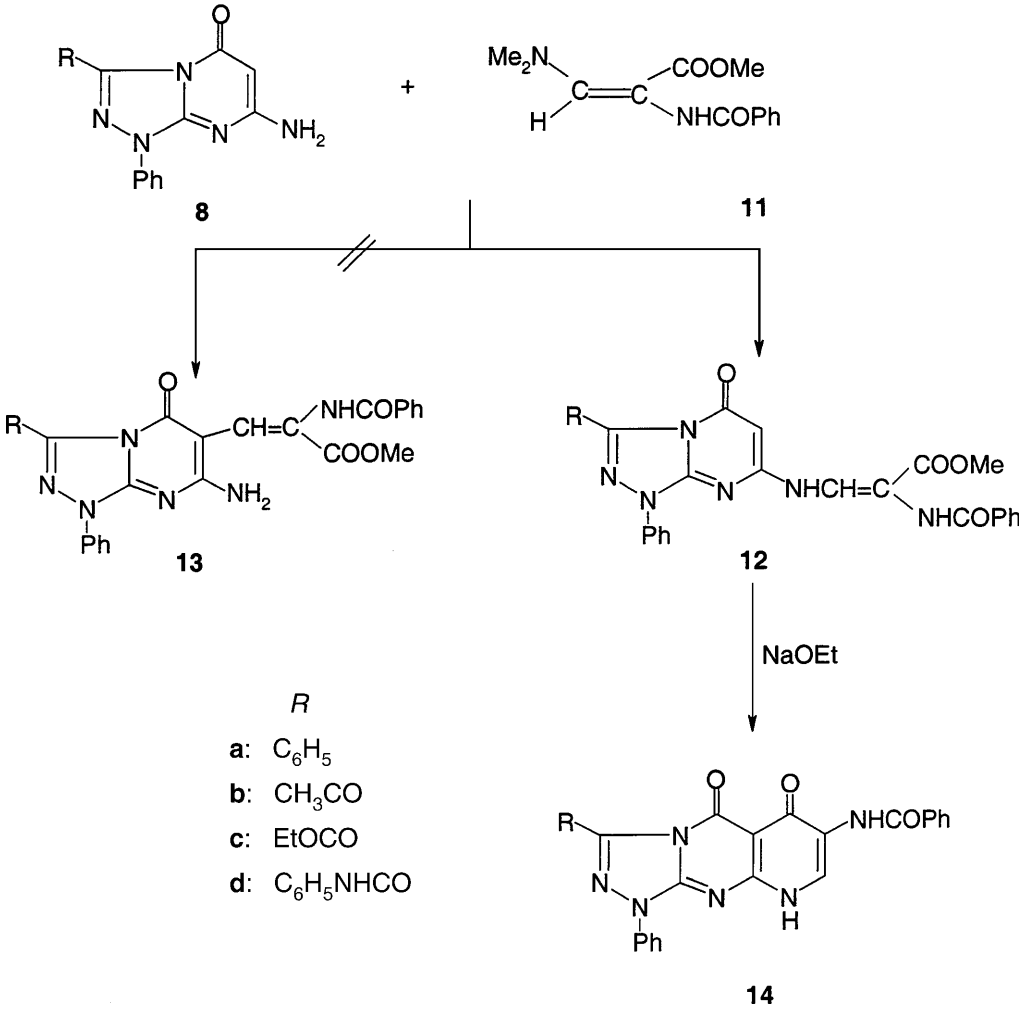
Next, the reaction of the 7-amino-[1,2,4]triazolo[4,3-*a*]pyrimidin-5(1*H*)-ones **8** with methyl (*Z*)-2-benzoylamino-3-dimethylamino propenoate (**11**) in refluxing acetic acid was examined. It was found to afford the hitherto unreported methyl (*Z*)-2-benzoylamino-3-([1,2,4]triazolo[4,3-*a*]-pyrimidin-7-yl)-aminopropenoate **12** and not isomer **13** (Scheme 2). The assignment of structures **12** were based on their elemental analyses and spectral data. Their ^{1}H NMR spectra showed a signal at 3.7–3.8 ppm for the $COOCH_3$ group and a resonance at 5.68–6.10 ppm for CH-6 of the pyrimidine ring, but gave no indication for an amino group.

Cyclization of **12** to the respective pyrido[2,3-*d*][1,2,4]triazolo-[4,3-*a*]pyrimidine-5,6-(1*H*,8*H*)-diones **14** was carried out by sodium ethoxide in ethanol at room temperature (Scheme 2). The structures of **14** were confirmed by their elemental analyses and spectroscopic data; their ^{1}H NMR spectra revealed the absence of a signal for the $COOCH_3$ group. Furthermore, the assignment of the structure **14** is also substantiated by the chemical shift values of the carbonyl carbon of the pyridinone ring (C6, α,β-unsaturated ketone) in **14a** and **14b** (169 and 170 ppm) which are similar to that of **16** (170–175 ppm, Fig. 1).



8 **15** **16**

Fig. 1. ^{13}C NMR shifts of strategic carbon atoms



Scheme 2

Experimental

Melting points were measured on a Gallenkamp apparatus and are uncorrected. Infrared spectra were recorded in KBr using a Pye Unicam SP-300 instrument. ^{1}H and ^{13}C NMR spectra were measured in *DMSO*-d_6 on a Bruker AC 250 spectrometer at the Fachbereich Chemie, Universität Rostock, Germany, mass spectra on a GCMS-QP 100 EX facility. Elemental analyses were carried out at the Microanalytical Laboratory at Cairo University, Giza, Egypt, and the at Fachbereich Chemie, Universität Rostock, Germany; the results agreed favourably (± 0.3) with the calculated values. The starting 6-aminouracil-2-thione (**1**) [13], the hydrazonoyl halides **2** [14–17], and methyl (*Z*)-2-benzoylamino-3-dimethylamino propenoate (**11**) [18] were prepared as described in the literature.

Reaction of **1** *with hydrazonoyl halides* **2***; general procedure*

Method A. To a mixture of 1.43 g of thione **1** (0.01 mol) and the appropiate hydrazonoyl halide **2** (0.01 mol) in 40 cm^3 dioxane (and 10 cm^3 *DMF* if necessary for solubility reasons), 1.4 cm^3 (0.01 mol) of triethylamine were added, and the mixture was heated under reflux until H_2S evolution ceased

(10–15 h). Then the solvent was distilled off, and the residue was cooled. The solid formed was filtered, washed with MeOH, and crystallized from ethanol:dioxane = 3:1 to give **8**.

Method B. To a stirred ethanolic solution of NaOEt prepared from 0.023 g (0.01 mol) Na and 20 cm^3 absolute EtOH, 1.43 g (0.01 mol) of **1** were added. After 10 min, 0.01 mol of the appropriate hydrazonoyl halides **2** were added to the resulting solution, and the mixture was stirred at room temperature for 24 h. During this period the educts dissolved, and a new product precipitated. The latter was filtered, washed with H_2O, dried, and finally crystallized from ethanol:dioxane = 3:1 to give **8**. The products proved to be identical in all respects with those obtained by method A.

7-Amino-1,3-diphenyl[1,2,4]triazolo[4,3-a]pyrimidin-5(1H)-one (**8a**; $C_{17}H_{13}N_5O$)

Yield: 70%; m.p.: 250°C; IR: $\nu = 3485, 3300, 1683\ cm^{-1}$; ^1H NMR (*DMSO*-d_6, δ, 250 MHz): 4.9 (s, CH), 6.9 (s, NH_2), 7.39–8.20 (m, 10 Ar–H) ppm; ^{13}C NMR (*DMSO*-d_6, δ, 62.9 MHz): 76, 121, 122, 126, 127, 128, 130, 131, 137, 144, 149, 157, 164 ppm; MS: m/z (%) = 303 (M^+, 80), 193 (5), 160 (17), 91 (100), 77 (24).

7-Amino-3-acetyl-1-phenyl[1,2,4]triazolo[4,3-a]pyrimidin-5(1H)-one (**8b**; $C_{13}H_{11}N_5O_2$)

Yield: 65%; m.p.: 185°C; IR: $\nu = 3480, 3310, 1700, 1690\ cm^{-1}$; ^1H NMR (*DMSO*-d_6, δ, 250 MHz): 2.7 (s, CH_3CO), 5.00 (s, CH), 6.80 (s, NH_2), 7.20–8.20 (m, 5 Ar–H) ppm; ^{13}C NMR (*DMSO*-d_6, δ, 62.9 MHz): 21, 77, 120, 121, 123, 128, 129, 130, 142, 164, 192 ppm; MS: m/z (%) = 269 (M^+, 100), 194 (7), 159 (9), 91 (61), 77 (79).

7-Amino-3-ethoxycarbonyl-1-phenyl[1,2,4]triazolo[4,3-a]pyrimidin-5(1H)-one (**8c**; $C_{14}H_{13}N_5O_3$)

Yield: 75%; m.p.: 215°C; IR: $\nu = 3480, 33150, 1750, 1695\ cm^{-1}$; ^1H NMR (*DMSO*-d_6, δ, 250 MHz): 1.39 (t, $J = 7.2$ Hz, CH_3), 4.5 (q, $J = 7.2$ Hz, CH_2), 4.92 (s, CH), 7.00 (s, NH_2), 7.40–8.00 (m, 5 Ar–H) ppm; ^{13}C NMR (*DMSO*-d_6, δ, 62.9 MHz): 13, 63, 77, 121, 127, 129, 135, 136, 147, 154, 160, 165 ppm; MS: m/z (%) = 299 (M^+, 90), 194 (10), 159 (9), 91 (61), 77 (100).

7-Amino-3-phenylcarbamoyl-1-phenyl[1,2,4]triazolo[4,3-a]pyrimidin-5(1H)-one (**8d**; $C_{18}H_{14}N_6O_2$)

Yield: 80%; m.p.: 235°C; IR: $\nu = 3485, 3326, 1694, 1650\ cm^{-1}$; ^1H NMR (*DMSO*-d_6, δ, 250 MHz): 5.00 (s, CH), 7.10 (s, NH_2), 7.50–8.10 (m, 10 Ar–H), 13.00 (s, NH) ppm; ^{13}C NMR (*DMSO*-d_6, δ, 62.9 MHz): 65, 120, 122, 127, 128, 129, 130, 137, 139, 142, 152, 156, 162, 165 ppm; MS: m/z (%) = 346 (M^+, 80), 226 (33), 199 (13), 160 (12), 91 (77), 77 (89).

7-Amino-3-benzoyl-1-phenyl[1,2,4]triazolo[4,3-a]pyrimidin-5(1H)-one (**8e**; $C_{18}H_{13}N_5O_2$)

Yield: 80%; m.p.: 242°C; IR: $\nu = 3485, 3326, 1690, 1660\ cm^{-1}$; ^1H NMR (*DMSO*-d_6, δ, 250 MHz): 5.00 (s, CH), 6.88 (s, NH_2), 7.30–8.10 (m, 10 Ar–H) ppm; MS: m/z (%) = 331 (M^+, 80), 222 (30), 105 (100), 77 (90).

7-Amino-3-acetyl-1-(p-methylphenyl)-[1,2,4]triazolo[4,3-a]pyrimidin-5(1H)-one (**8f**; $C_{14}H_{13}N_5O_2$)

Yield: 73%; m.p.: 165°C; IR: $\nu = 3485, 3300, 1700, 1685\ cm^{-1}$; ^1H NMR (*DMSO*-d_6, δ, 250 MHz): 2.3 (s, CH_3), 2.7 (s, CH_3CO), 4.98 (s, CH), 6.90 (s, NH_2), 7.30–8.10 (m, 4 Ar–H) ppm; MS: m/z (%) = 283 (M^+, 100), 192 (7), 160 (10), 91 (60), 77 (89).

7-Amino-3-phenylcarbamoyl-1-(p-methylphenyl)-[1,2,4]triazolo[4,3-a]pyrimidin-5(1H)-one (**8g**; $C_{19}H_{16}N_6O_2$)

Yield: 82%; m.p.: 225°C; IR: $\nu = 3485$, 3320, 1690, 1650 cm^{-1}; ^1H NMR (*DMSO*-d_6, δ, 250 MHz): 2.30 (s, CH_3), 5.00 (s, CH), 7.10 (s, NH_2), 7.3–8.0 (m, 9 Ar–H), 11.80 (s, NH) ppm; MS: m/z (%) = 360 (M^+, 100), 220 (50), 180 (40), 149 (11), 91 (100), 77 (63).

Reaction of **8** *with methyl (Z)-2-benzoylamino-3-dimethylamino propenoate* **11**; *general procedure*

To a boiling solution of 0.01 mol of compound **8** in 50 cm^3 glacial acetic acid, 0.01 mol of methyl (*Z*)-2-benzoylamino-3-dimethylamino propenoate (**11**) were added, and the mixture was refluxed for 6–10 h (TLC monitoring). The solvent was evaporated *in vacuo*, and the residue was recrystallized from dioxane:ethanol = 1:1 to give **12**.

Methyl (Z)-2-benzoylamino-3-(1,3-diphenyl[1,2,4]triazolo[4,3-a]pyrimidin-7-yl)-amino propenoate (**12a**; $C_{28}H_{22}N_6O_4$)

Yield: 86%; m.p.: 180–182°C; IR: $\nu = 3435$, 1710, 1680, 1660 cm^{-1}; ^1H NMR (*DMSO*-d_6, δ, 250 MHz): 3.71 (s, $COOCH_3$), 6.10 (s, 6′-CH) 7.3–8.20 (m, 15 Ar–H), 8.85 (d, $J = 12$ Hz, 3-CH), 9.60 (s, NH), 10.20 (d, $J = 12$ Hz, 3-NH) ppm; ^{13}C NMR (*DMSO*-d_6, δ, 62.9 MHz): 51, 68, 83, 107, 121, 121, 124, 127, 128, 129, 130, 132, 133, 134, 136, 138, 139, 147, 152, 157, 162, 165 ppm; MS: $m/z = 506$ (M^+, 80), 475 (25), 385 (25), 265 (10), 105 (100), 91 (50), 77 (75).

Methyl (Z)-2-benzoylamino-3-(3-acetyl-1-phenyl[1,2,4]triazolo[4,3-a]pyrimidin-7-yl)-amino propenoate (**12b**; $C_{28}H_{22}N_6O_4$)

Yield: 75%; m.p.: 232–234°C; IR: $\nu = 3455$, 1725, 1700, 1675, 1660 cm^{-1}; ^1H NMR (*DMSO*-d_6, δ, 250 MHz): 2.70 (s, $COCH_3$), 3.80 (s, $COOCH_3$), 6.00 (s, 6′-CH), 7.2–8.10 (m, 10 Ar–H), 8.50 (d, $J = 12$, 3-CH), 9.10 (s, 1H, NH), 10.30 (d, $J = 12$ Hz, 3-NH) ppm; MS: $m/z = 472$ (M^+, 65), 440 (20), 384 (20), 265 (5), 105 (100), 91 (40), 77 (70).

Methyl (Z)-2-benzoylamino-3-(3-ethoxycarbonyl-1-phenyl[1,2,4]triazolo[4,3-a]pyrimidin-7-yl)-amino propenoate (**12c**; $C_{25}H_{22}N_6O_6$)

Yield: 68%; m.p.: 191–192°C; IR: $\nu = 3430$, 1750, 1710, 1675, 1655 cm^{-1}; ^1H NMR (*DMSO*-d_6, δ, 250 MHz): 1.39 (t, $J = 7.1$ Hz, CH_3), 3.72 (s, 3H, H), $COOCH_3$), 4.5 (q, $J = 7.1$ Hz, CH_2), 5.99 (s, 6′-CH) 7.30–8.10 (m, 5 Ar–H), 8.50 (d, $J = 12$ Hz, 3-CH), 8.80 (s, NH), 9.25 (d, $J = 12$ Hz, 3-NH), 10.50 (s, NH) ppm; MS: $m/z = 502$ (M^+, 80), 470 (25), 385 (20), 265 (10), 105 (90), 91 (50), 77 (100).

Methyl (Z)-2-benzoylamino-3-(3-phenylcarbamoyl-1-phenyl[1,2,4] triazolo[4,3-a]pyrimidin-7-yl)-amino propenoate (**12d**; $C_{29}H_{23}N_7O_5$)

Yield: 76%; m.p.: 250–251°C; IR: $\nu = 3448$, 1708, 1660, 1638 cm^{-1}; ^1H NMR (*DMSO*-d_6, δ, 250 MHz): 3.72 (s, $COOCH_3$), 5.68 (s, 6′-CH) 7.15–8.18 (m, 15 Ar–H), 8.40 (d, $J = 12$ Hz, 3-CH), 8.77 (s, NH), 9.30 (d, 12 Hz, 3-NH), 12.10 (s, 1H, NH) ppm; ^{13}C NMR (*DMSO*-d_6, δ, 62.9 MHz): 52, 67, 83, 107, 120, 122, 125, 128.2, 128.5, 129, 130, 132, 133, 134, 136, 138, 139, 148, 153, 157, 159, 165, 166 ppm; MS: $m/z = 549$ (M^+, 50), 517 (20), 384 (20), 265 (15), 237 (15), 119 (100), 91 (80), 77 (20).

Cyclization of **12***; general procedure*

To a stirred ethanolic NaOEt solution prepared from 0.46 g (20 mmol) Na and 40 cm^3 of absolute EtOH, 0.01 mol of **12** were added, and the mixture was stirred at room temperature overnight. The excess slovent was evaporated *in vacuo*, and 20 cm^3 H_2O were added. The solid formed was filtered, dried, and crystallized from dioxane to afford **14**.

7-Benzoylamino-1,3-diphenyl-pyrido[2,3-d][1,2,4]triazolo [4,3-a]pyrimidin-5,6-(1H,8H)-dione (**14a**; $C_{27}H_{18}N_6O_3$)

Yield: 78%; m.p.: 272–273°C; IR: $\nu = 3350$, 1680, 1658, 1645 cm^{-1}; ^{1}H NMR (*DMSO*-d_6, δ, 250 MHz): 7.15–8.18 (m, 15 Ar–H), 8.30 (d, $J = 2$ Hz, 3-CH), 8.90 (s, NH), 9.35 (d, $J = 2$ Hz, 3-NH) ppm; ^{13}C NMR (*DMSO*-d_6, δ, 62.9 MHz): 70, 85, 110, 121, 122, 125, 127, 128, 129, 130, 131, 132, 134, 136, 138, 139, 147, 152, 157, 164, 169 ppm; MS: m/z (%) = 474 (M^+, 50), 397 (20), 369 (30), 265 (15), 105 (100), 77 (80).

7-Benzoylamino-3-acetyl-1-phenyl-pyrido[2,3-d][1,2,4]triazolo [4,3-a]pyrimidin-5,6-(1H,8H)-dione (**14b**; $C_{23}H_{16}N_6O_4$)

Yield: 62%; m.p.: 275–276°C; IR: $\nu = 3345$, (NH), 1720, 1680, 1660, 1650 cm^{-1}; ^{1}H NMR (*DMSO*-d_6, δ, 250 MHz): 2.6 (s, $COCH_3$), 7.30–8.00 (m, 10 Ar–H), 8.50 (d, $J = 2$ Hz, 3-CH), 9.10 (s, NH), 9.43 (d, $J = 2$ Hz, 3-NH) ppm; MS: m/z (%) = 440 (M^+, 40), 363 (10), 320 (30), 265 (15), 105 (50), 77 (90).

7-Benzoylamino-3-ethoxycarbonyl-1-phenyl-pyrido[2,3-d][1,2,4] triazolo[4,3-a]pyrimidin-5,6-(1H,8H)-dione (**14c**; $C_{24}H_{18}N_6O_5$)

Yield: 75%; m.p.: 264–265°C; IR: $\nu = 3350$, 1740, 1675, 1660, 1645 cm^{-1}; ^{1}H NMR (*DMSO*-d_6, δ, 250 MHz): 1.38 (t, $J = 7.1$ Hz, CH_3), 4.4 (q, $J = 7.1$ Hz, CH_2), 7.15–8.20 (m, 10 Ar–H)), 8.40 (d, $J = 2$ Hz, 3-CH), 8.95 (s, 1H, NH), 10.10 (d, $J = 2$ Hz, 3-NH) ppm; MS: m/z (%) = 470 (M^+, 20), 393 (50), 365 (30), 350 (15), 105 (40), 77 (100).

7-Benzoylamino-3-phenylcarbamoyl-1-phenyl-pyrido[2,3-d][1,2,4] triazolo[4,3-a]pyrimidin-5,6-(1H,8H)-dione (**14d**; $C_{28}H_{19}N_7O_4$)

Yield: 78%; m.p.: 287–289°C; IR: $\nu = 3300$, 1680, 1660, 1630 cm^{-1}; ^{1}H NMR (*DMSO*-d_6, δ, 250 MHz): 7.20–8.20 (m, 15 Ar–H), 8.50 (d, $J = 2$ Hz, 3-CH), 8.90 (s, NH), 9.40 (d, $J = 2$ Hz, 3-NH) ppm; ^{13}C NMR (*DMSO*-d_6, δ, 62.9 MHz): 69, 84, 110, 121, 123, 125, 127, 128, 129, 130, 131, 132, 133, 135, 138, 139, 146, 152, 157, 159, 165, 170 ppm; MS: $m/z = 417$ (M^+, 65), 440 (25), 412 (30), 397 (15), 105 (30), 77 (100).

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