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Novel synthesis of 1,2,4-triazolo[4,3-*a*]pyrimidin-5-one derivatives

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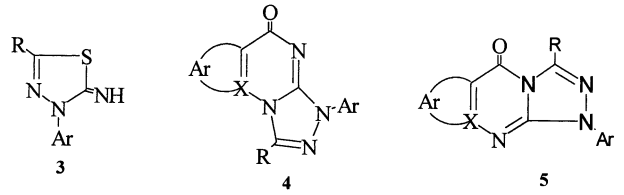
Abstract—1,2-Dihydro-2-thioxopyrimidin-4(3*H*)ones **6** react with C-ethoxycarbonyl-*N*-arylhydrazonoyl chlorides **1** to give 1,2,4-triazolo[4,3-*a*]pyrimidines **11** in good yield. The latter products **11** react with benzenediazonium chloride, nitrous acid, acetyl chloride and chloroacetyl chloride to give the corresponding substitution products **16**–**18**. The structures of the newly synthesized compounds are established on the basis of chemical and spectroscopic evidence. © 2001 Published by Elsevier Science Ltd.

1. Introduction

Previously, we reported that hydrazonoyl halides **1** react with thiourea and with heterocyclic compounds containing a thiourea moiety to give the corresponding thiadiazoline **3** and triazoloazines **4** or **5**, respectively.[1–3] The angular structure **4** was tentatively assumed for these products on the basis of N1 is more basic due to the effect of carbonyl group,[4,5] however on solid structure proof has been given.



3 **4** **5**

In the present investigation, we report on the reaction of 1,2-dihydro-2-thioxopyrimidin-4(3*H*)one derivatives **6** with hydrazonoyl chlorides **1** and determine the structure of the products in addition to their reactivity towards some electrophiles. The newly synthesized compounds appear to be promising for further chemical transformations as well as biological activity evaluations.

2. Results and discussion

The reaction of **6** with hydrazonoyl chlorides **1** was carried out in chloroform in the presence of triethylamine under reflux. After work up of the reaction mixture, only a single product was isolated, in each case. As expected from all our previous results with related compounds[2,3] these products did not contain sulfur. On the basis of this finding the structures **7**–**10** (Scheme 1) were discarded for the products isolated. The spectral and elemental analytical data are compatible, however, with one of the two isomeric structures **11** or **12** (Scheme 1).

The proposed mechanism leading to the formation of the latter products **11** or **12** suggested that the studied reactions involve an initial formation of thiohydrazonoate esters **7** or amidrazones of type **13**. The latter amidrazones **13** was excluded on the basis of (1) although amidrazones of type **13** are known to be stable,[1,6,7] all attempts to separate them from the reaction mixture failed in all cases; (2) reaction of 2-thiouracile derivatives with various halogen compounds yielded in all cases *S*-substituted products.[8–11] The thiohydrazonoate esters **7** undergo an S→N migration via the spiro intermediates **8**, to give the thiohydrazides **9**. The spiro intermediates **8** may also be formed via 1,3-dipolar cycloaddition of nitrilimines **2**, generated in situ from reaction of hydrazonoyl chlorides **1** with triethylamine, to the thione group C=S of **6**. The latter intermediates **9** undergo cyclization followed by elimination of hydrogen sulfide to give the final products **11** or **12** (Scheme 1).

All these products gave a singlet signal at δ 5.5 in their ^{1}H NMR spectra assignable to pyrimidine CH and showed three stretching frequencies near 3281, 1750 and 1687 cm^{-1} assignable to NH, ester CO and amide CO groups, respectively. These data cannot determine the exact structure for the resulting products and conclusive evidence for the structure **11** was obtained by X-ray crystallographic analysis of a crystal of the product **16a** resulting from reaction of **11a** with benzenediazonium chloride.

Compounds **11a** and **11d** are readily coupled with equimolar amount of benzenediazonium chloride to yield exclusively the corresponding 6-phenyl-azo derivatives **16a** and

Keywords: 1,2-dihydro-2-thioxopyrimidin-6-phenylamino-4(3*H*)ones; C-ethoxycarbonyl-*N*-arylhydrazonoyl chlorides; 1,2,4-triazolo[4,3-*a*]-pyrimidine-5-ones.

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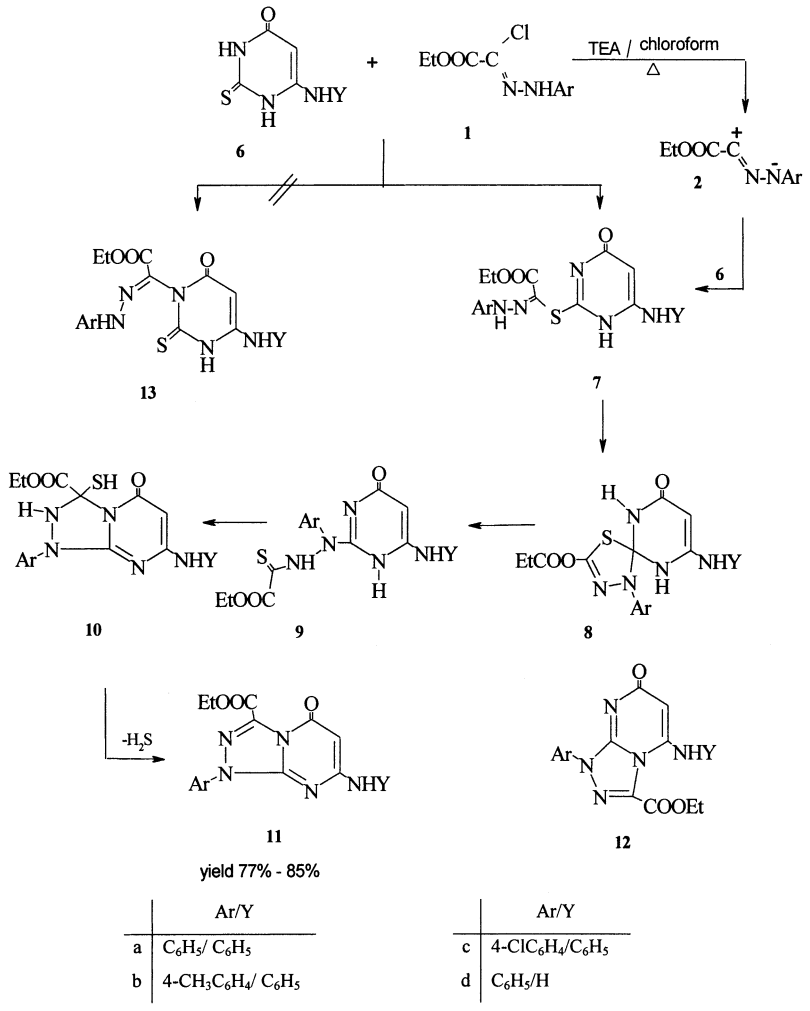


Scheme 1.

16b, respectively (Scheme 2). The structures of the latter products were established basis on elemental and spectral analyses (IR, ^{1}H NMR, MS). For example, ^{1}H NMR spectrum of **16a** revealed the absence of singlet signal at δ 5.5 (pyrimidine CH). Also, the structures of **16a** and **16b** were confirmed by their alternative synthesis from reaction of 5-phenylazo-1,2-dihydro-2-thioxopyrimidin-4(3*H*)ones **14a** and **14b** with compound **1a**, respectively (Scheme 2). Furthermore, the structure of the product **16a** was confirmed by X-ray crystallography (Fig. 1).

Nitrozation of compounds **11** with sodium nitrite in acetic acid yielded the corresponding 6-nitrosotriazolo[4,3-*a*]pyrimidine derivatives **17** in excellent yield. These compounds are also prepared by reaction of 5-nitroso-1,2-dihydro-2-thioxopyrimidin-4(3*H*)one **15** with hydrazonoyl chlorides **1** (Scheme 2). The structures of the products **17** were also confirmed by elemental and spectral data.

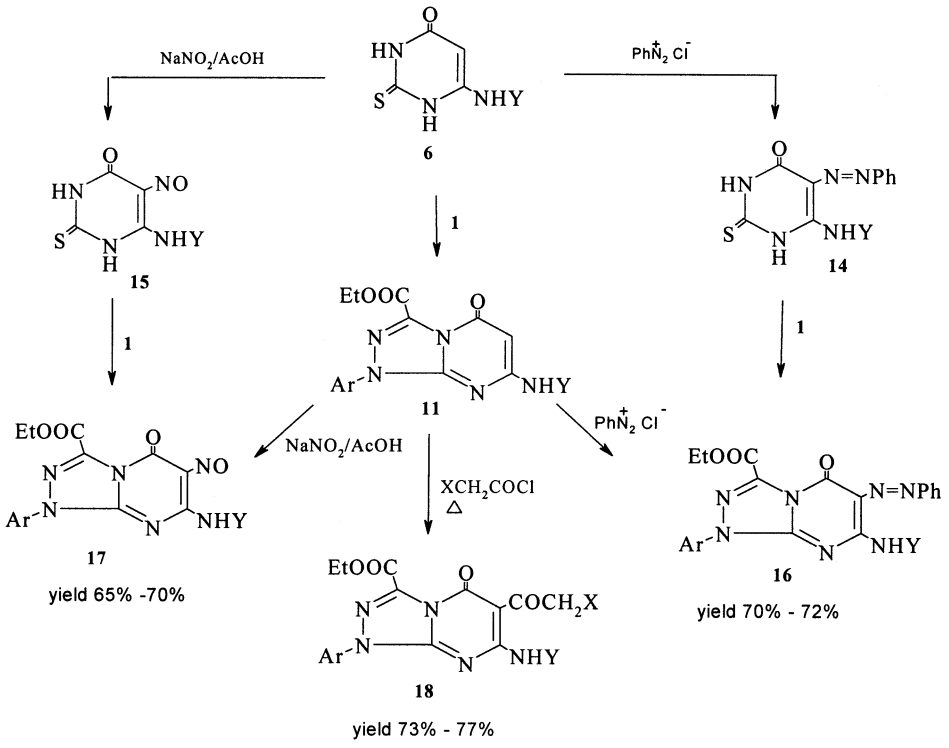
Acylation of compounds **11** with acetyl chloride and chloroacetyl chloride furnished the corresponding substitution product **18a**–**18c**. Elemental analysis and spectroscopic data confirmed the structure **18a**–**18c**. For example, the ^{1}H NMR spectra of the latter products revealed the absence of singlet signal at δ 5.5 of compound **11**, instead it showed a two signals at δ 2.2 and 2.8 corresponding to acetyl groups in compounds **18a** and **18b**, respectively, and a singlet signal at δ 5.1 assignable to the chloroacetyl group in compound **18c**.

2.1. X-Ray crystallographic study of compound 16a

The molecular structure and numbering scheme is depicted in Fig. 1. The crystals were quite small and of low quality with some reflection profiles showing shoulders, but the overall structure is clearly defined. The amine group forms an intramolecular hydrogen bond with the outer N-atom of the N=N group. The two fused rings are planar. Both the phenyl substituent of N(6) and the –N=N–Ph group are virtually coplanar with the fused rings, although the phenyl substituent at N(6) is titled slightly out of the plane due to the slight pyramidalization at N(6). The other substituents do not lie close to the fused ring plane.

3. Experimental

Melting points were taken on a Gallenkamp apparatus and are uncorrected. Infrared spectra (KBr) were determined on a Pye Unicam SP-3000 infrared spectrophotometer. ^{1}H



17	Ar/Y
a	C_6H_5/ C_6H_5
b	4-$CH_3C_6H_4$/ C_6H_5
c	4-ClC_6H_4/C_6H_5

18	X/Y
a	H/H
b	H/C_6H_5
c	Cl/C_6H_5

16	Y
a	C_6H_5
b	H

Scheme 2.

NMR spectra were determined on a Varian Gemini 200 spectrometer (200 MHz) in $CDCl_3$ with TMS as an internal standard. Mass spectra were recorded on a GCMS-QP 1000EX Shimadzu, Japan. Elemental analyses were carried out at the microanalytical center, University of Cairo, Giza, Egypt. The C-ethoxycarbonylhydrazonoyl chlorides **1a–1d**[12] and **6**,[13] **14**[13] and **15**[13] were prepared as previously described.

3.1. Synthesis of 1,2,4-triazolo[4,3-*a*]pyrimidin-5-ones 11a–11d: general method

To a stirred solution of the appropriate hydrazonoyl chlorides **1** (5 mmol) and 2-thioxopyrimidin-4(3*H*)one **6** (5 mmol) in chloroform (40 ml) was added triethylamine (0.7 ml, 5 mmol) at room temperature. The reaction mixture was refluxed until the hydrazonoyl halide disappeared (4–6 h) as indicated by TLC analysis. The solvent was evaporated under reduced pressure and the residue was treated with methanol (10 ml). The solid formed was collected and crystallized from a suitable solvent to give the corresponding 1,2,4-triazolo[4,3-*a*]pyrimidin-5-ones **11a–11d**.

3.1.1. Ethyl (1-phenyl-7-phenylamino-5-oxo-1,2,4-triazolo[4,3-*a*]pyrimidin-3-yl)carboxylate 11a. The compound was obtained in 85% yield, pale yellow crystal mp 173°C (ethanol); IR (KBr) ν 3272 (NH), 1746 (C=O), 1687 (C=O) cm^{-1}; ^1H NMR ($CDCl_3$) δ 1.42 (t, J=7 Hz, 3H), 4.55 (q, J=7 Hz, 2H), 5.49 (s, 1H, pyrimidine CH), 7.0 (s, 1H, NH), 7.13–8.21 (m, 10H); MS, *m/z*: 375, 303, 275, 220, 187, 144, 93, 77. Anal. calcd for $C_{20}H_{17}N_5O_3$: C, 64.0; H, 4.5; N, 18.7. Found: C, 64.1; H, 4.7; N, 18.4%.

3.1.2. Ethyl (1-(*p*-tolyl)7-phenylamino-5-oxo-1,2,4-triazolo[4,3-*a*]pyrimidin-3-yl)carboxylate 11b. The compound was obtained in 80% yield, yellow crystal mp 206°C (acetic acid); IR (KBr) ν 3281 (NH), 1750 (C=O), 1689 (C=O) cm^{-1}; ^1H NMR ($CDCl_3$) δ 1.49 (t, J=7 Hz, 3H); 2.42 (s, 3H); 4.60 (q, J=7 Hz, 2H); 5.49 (s, 1H, pyrimidine CH), 6.93 (s, 1H, NH), 7.26–8.0 (m, 9H); MS, *m/z*: 389, 227, 207, 144, 105, 60. Anal. calcd for $C_{21}H_{19}N_5O_3$: C, 64.8; H, 4.9; N, 18.0. Found: C, 64.6; H, 4.7; N, 17.8%.

3.1.3. Ethyl (1-(*p*-chlorophenyl)7-phenylamino-5-oxo-1,2,4-triazolo[4,3-*a*]pyrimidin-3-yl)carboxylate 11c. The compound was obtained in 78% yield, white crystal mp 231°C (acetic acid); IR (KBr) ν 3287 (NH), 1741 (C=O), 1692 (C=O) cm^{-1}; ^1H NMR ($CDCl_3$) δ 1.42 (t, J=7 Hz, 3H), 4.52 (q, J=7 Hz, 2H), 5.45 (s, 1H, pyrimidine CH), 6.97 (s, 1H, NH), 7.26–8.23 (m, 9H); MS, *m/z*: 409, 353, 277, 229, 183, 133, 77. Anal. calcd for $C_{20}H_{16}ClN_5O_3$: C, 58.7; H, 3.9; N, 17.1. Found: C, 58.8; H, 4.0; N, 17.0%.

3.1.4. Ethyl (1-phenyl-7-amino-5-oxo-1,2,4-triazolo[4,3-*a*]-pyrimidin-3-yl)carboxylate 11d. The compound was obtained in 77% yield; pale yellow crystal mp 224°C (acetic acid); IR (KBr) ν 3471, 3284 (NH_2), 1741 (C=O), 1668

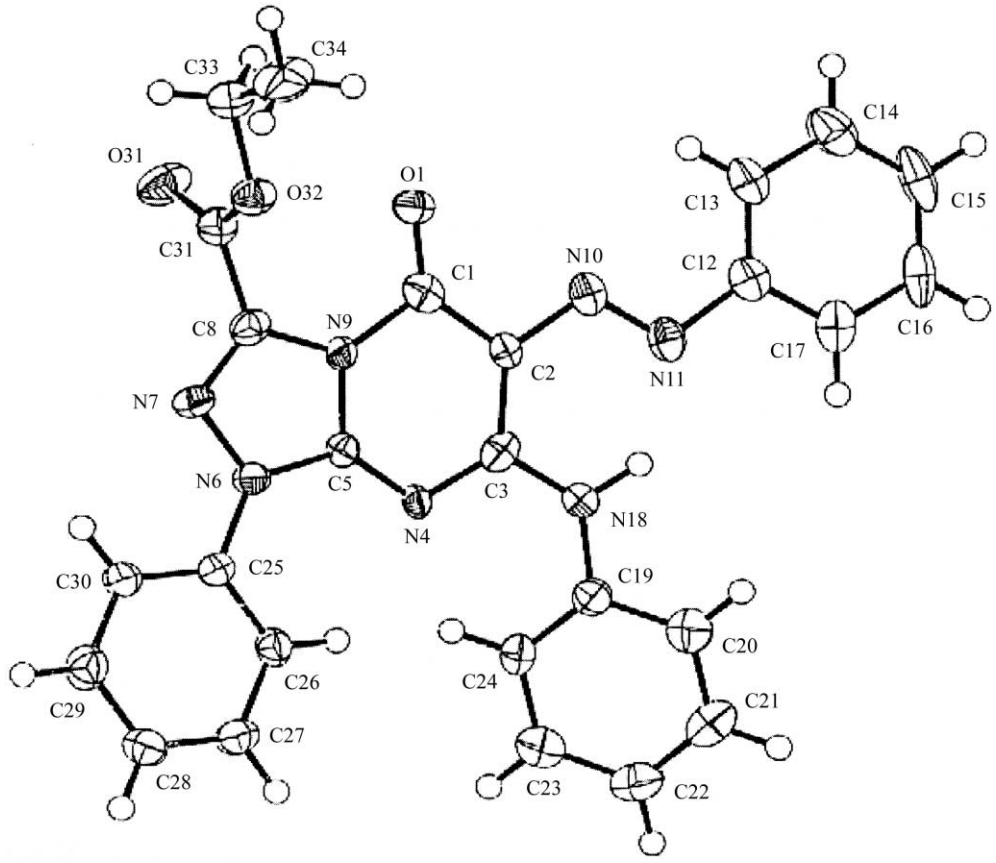


Figure 1. Diagram of compound **16a** with crystallographic numbering system.

(C═O) cm^{-1}; ^1H NMR ($CDCl_3$) δ 1.45 (t, J=7 Hz, 3H), 4.57 (q, J=7 Hz, 2H), 4.92 (s, 2H, NH_2), 5.16 (s, 1H, pyrimidine CH), 7.33–8.09 (m, 5H); MS, *m/z*: 299, 271, 226, 197, 173, 159, 105, 77, 68, 51. Anal. calcd for $C_{14}H_{13}N_5O_3$: C, 56.2; H, 4.4; N, 23.4. Found: C, 56.0; H, 4.5; N, 23.3%.

3.2. Synthesis of ethyl (1-phenyl-6-phenylazo-5-oxo-1,2,4-triazolo[4,3-*a*]pyrimidin-3-yl)carboxylate derivatives 16a and 16b

Method A. To a solution of **11a**,**d** (5 mmol) in ethanol (100 ml), sodium hydroxide (0.2 g, 5 mmol) was added. The mixture was then treated gradually with stirring at room temperature with a solution of benzenediazonium chloride (prepared from aniline (0.5 ml, 5 mmol) and the appropriate quantities of hydrochloric acid and sodium nitrite). The product was separated on standing, collected by filtration and crystallized from diemthylformamide to give **16a** and **16b**.

Method B. Compounds **16a** and **16b** were prepared by the same method described for the synthesis of **11** using 5-phenylazo-2-thioxopyrimidin-4(3*H*)-one derivatives **14a** and **14b** in place of **6**. The products were identical in all respects (mp, mmp, IR) to those resulting from method A.

3.2.1. Ethyl (1-phenyl-6-phenylazo-7-phenylamino-5-oxo-1,2,4-triazolo[4,3-*a*]pyrimidin-3-yl)carboxylate 16a. The compound was obtained in 70% yield; orange crystal mp 253°C; IR (KBr) ν 3210 (NH), 1753 (C═O), 1701 (C═O) cm^{-1}; ^1H NMR ($CDCl_3$) δ 1.50 (t, J=7 Hz, 3H), 4.64 (q, J=7 Hz, 2H), 7.19–8.14 (m, 15H), 13.90 (s, 1H, NH); MS, *m/z* 479, 450, 402, 328, 275, 220, 129, 91, 77. Anal. calcd for $C_{26}H_{21}N_7O_3$: C, 65.1; H, 4.4; N, 20.5. Found: C, 65.3; H, 4.2; N, 20.2%.

3.2.2. Ethyl (1-phenyl-6-phenylazo-7-amino-5-oxo-1,2,4-triazolo[4,3,-*a*]pyrimidin-3-yl)carboxylate 16b. The compound was obtained in 72% yield; orange crystal mp 230°C; IR (KBr) ν 3446, 3280 (NH_2), 1747 (C═O), 1712 (C═O) cm^{-1}; ^1H NMR ($CDCl_3$) δ 1.52 (t, J=7 Hz, 3H), 4.66 (q, J=7 Hz, 2H), 6.06 (s, 2H, NH_2), 7.32–8.10 (m, 10H); MS, *m/z* 403, 375, 326, 270, 232, 131, 91, 77, 51; Anal. calcd for $C_{20}H_{17}N_7O_3$: C, 59.6; H, 4.2; N, 24.3. Found: C, 59.9; H, 4.1; N, 24.4%.

3.3. Synthesis of ethyl (1-aryl-6-nitroso-7-phenylamino-5-oxo-1,2,4-triazolo[4,3-*a*]pyrimidin-3-yl)carboxylate 17a–17c

Method A. To a solution of **11** (5 mmol) in acetic acid (10 ml), a concentrated solution of sodium nitrite (2 g in 5 ml water) was added with stirring. The solid that formed was collected and crystallized from ethanol to give **17a**–**17c**.

Method B. Compounds **17** were prepared by the same method described for the synthesis of **11** using 6-phenylamino-5-nitroso-1,2-dihydro-2-thioxopyrimidine-4(3*H*)one

15a–15c in place of **6**. The products were identical in all respects to those obtained by method A.

3.3.1. Ethyl (1-phenyl-6-nitroso-7-phenylamino-5-oxo-1,2,4-triazolo[4,3-*a*]pyrimidin-3-yl)carboxylate 17a. The compound was obtained in 70% yield, pale yellow crystal mp 192°C; IR (KBr) ν 3220 (NH), 1750 (C=O), 1700 (C=O) cm^{-1}; ^1H NMR ($CDCl_3$) δ 1.43 (t, J=7 Hz, 3H), 4.45 (q, J=7 Hz, 2H), 7.23–7.92 (m, 10H), 11.78 (s, 1H, NH). Anal. calcd for $C_{20}H_{16}N_6O_4$: C, 59.4; H, 4.0; N, 20.8. Found: C, 59.5; H, 4.1; N, 20.7%.

3.3.2. Ethyl (1-(*p*-tolyl)-6-nitroso-7-phenylamino-5-oxo-1,2,4-triazolo[4,3-*a*]pyrimidin-3-yl)carboxylate 17b. The compound was obtained in 69% yield; white crystal mp 185°C; IR (KBr) ν 3218 (NH), 1748 (C=O), 1700 (C=O) cm^{-1}; ^1H NMR ($CDCl_3$) δ 1.44 (t, J=7 Hz, 3H), 2.42 (s, 3H), 4.47 (1, J=7 Hz, 2H), 7.26–7.80 (m, 9H), 11.87 (s, 1H, NH). Anal. calcd for $C_{21}H_{18}N_6O_4$: C, 60.3; H, 4.3; N, 20.1. Found: C, 60.1; H, 4.1; N, 20.0%.

3.3.3. Ethyl (1-(*p*-chlorophenyl)-6-nitroso-7-phenylamino-5-oxo-1,2,4-triazolo[4,3-*a*]pyrimidin-3-yl)carboxylate 17c. The compound was obtained in 65% yield; pale yellow crystal mp 175°C; IR (KBr) ν 3200 (NH), 1748 (C=O), 1701 (C=O) cm^{-1}; ^1H NMR ($CDCl_3$) δ 1.43 (t, J=7 Hz, 3H), 4.45 (q, J=7 Hz, 2H), 7.23–8.97 (m, 9H), 11.87 (s, 1H, NH). Anal. calcd for $C_{20}H_{15}ClH_6O_4$: C, 54.7; H, 3.4; N, 19.2. Found: C, 55.0; H, 3.5; N, 19.1%.

3.4. Synthesis of ethyl (1-phenyl-6,7-disubstituted-5-oxo-1,2,4-triazolo-[4,3-*a*]pyrimidin-3-yl)carboxylate 18a–18c: general procedure

Compounds **11a**,**d** (5 mmol) was refluxed in acetyl chloride (10 ml) or chloroacetyl chloride (10 ml) for 3 h. The reaction mixture was cooled and the solid formed was collected and crystallized from suitable solvent to give products **18a–18c**.

3.4.1. Ethyl (1-phenyl-6-acetyl-7-amino-5-oxo-1,2,4-triazolo[4,3-*a*]pyrimidin-3-yl)carboxylate 18a. The compound was obtained in 75% yield; white crystal mp 245°C (DMF–ethanol); IR (KBr) ν 3340, 3132 (NH_2), 1720 (C=O), 1712 (C=O), 1689 (C=O) cm^{-1}; ^1H NMR ($CDCl_3$) δ 1.53 (t, J=7 Hz, 3H), 2.21 (s, 3H), 4.60 (q, J=7 Hz, 2H), 7.13–8.02 (m, 7H, ArH+NH_2); MS, *m*/*z* 341, 326, 303, 254, 212, 187, 144, 91, 77. Anal. calcd for $C_{16}H_{15}N_5O_4$: C, 56.3; H, 4.4; N, 20.5. Found: C, 56.4; H, 4.5; N, 20.4%.

3.4.2. Ethyl (1-phenyl-6-acetyl-7-phenylamino-5-oxo-1,2,4-triazolo-[4,3-*a*]pyrimidin-3-yl)carboxylate 18b. The compound was obtained in 73% yield; white crystal mp 175°C (acetic acid); IR (KBr) ν 3370 (NH), 1743 (C=O), 1705 (C=O), 1678 (C=O) cm^{-1}; ^1H NMR ($CDCl_3$) δ 1.51 (t, J=7 Hz, 3H), 2.80 (s, 3H), 4.61 (q, J=7 Hz, 2H), 7.26–8.12 (m, 10H), 13.10 (s, 1H, NH); MS, *m*/*z* 417, 327, 326, 237, 213, 144, 117, 77. Anal. calcd for $C_{22}H_{19}N_5O_4$: C, 63.3; H, 4.6; N, 16.8. Found: C, 63.5; H, 4.4; N, 16.7%.

3.4.3. Ethyl (1-phenyl-6-chloroacetyl-7-phenylamino-5-oxo-1,2,4-triazolo[4,3-*a*]pyrimidin-3-yl)carboxylate 18c. The compound was obtained in 77% yield; white crystal mp 174°C (ethanol); IR (KBr) ν 3250 (NH), 1735 (C=O), 1700 (C=O), 1667 (C=O) cm^{-1}; ^1H NMR ($CDCl_3$) δ 1.50 (t, J=7 Hz, 3H), 4.62 (q, J=7 Hz, 2H), 5.09 (s, 2H), 7.35–8.06 (m, 10H), 12.77 (s, 1H, NH); MS, *m*/*z*: 451, 416, 402, 330, 288, 213, 144, 77. Anal. calcd for $C_{22}H_{18}ClN_5O_4$: C, 58.5; H, 4.0; N, 15.5. Found: C, 58.6; H, 4.1; N, 15.6%.

3.5. X-Ray analysis

The structure was solved by direct methods using SIR927, which revealed (CCDC Ref. no. 155856) the positions of all non-hydrogen atoms. The non-hydrogen atoms were refined anisotropically. All of the H-atoms were fixed in geometrically calculated positions [d(C–H)=0.95 Å] and each was assigned a fixed isotropic displacement parameter with a value equal to 1.2_{Ueq} of its parent C-atom. Refinement of the structure was carried out on F using full-matrix least-squares procedures, which minimized the function $\sum(|F_o| - |F_c|)$.[14] The weighting scheme was based on counting statistics and included a factor to downweight the intense reflections. Plots of $\sum(|F_o| - |F_c|)^{14}$ verse $|F_o|$, reflection order in data collection, $\sin\theta/\lambda$, and various classes of indices showed no unusual trends. A correction for secondary extinction was applied.

Neutral atom scattering factors for non-hydrogen atoms were taken from Maslen, Fox and O'Keefe,[15] and the scattering factors for H-atoms were taken from Stewart et al.[16] Anomalous dispersion effects were included in F_c;[17] the values for f' and f'' were those of Creagh and McAuley.[15] The values of the mass attenuation coefficients are those of Creagh and Hubbell.[15] All calculations were performed using the teXsan crystallographic software package.[18]

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