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UTILITY OF 2-HYDRAZINO-4,6-DIMETHYLPYRIMIDINE IN HETEROCYCLIC SYNTHESIS

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ABSTRACT

2-Hydrazino-4,6-dimethylpyrimidine **(1)** readily underwent ring closure with benzoyl chloride to give 5,7-dimethyl-3-phenyl-1,2,4-triazolo[4,3-a]pyrimidine **(4a)**. Either N-benzoyl derivative **(2)** or the hydrazone **(3a)** can be used for the formation of compound **4a**. Reaction of compound **1** with acetylacetone gave the pyrazole derivative **(6)** rather than 1,2,4-triazepine derivative **(5)**. The pyrrole **(8)** was the sole product from cyclization of **1** with 2,5- hexanedione. Reaction of compound **1** with carbon disulphide or ethyl chloroformate gave 1,2,4-triazolo[4,3-a]pyrimidines **(9** and **11)** respectively. The reaction of thiosemicarbazides **(12 a-c)** with ethyl bromoacetate and DCCD was investigated.

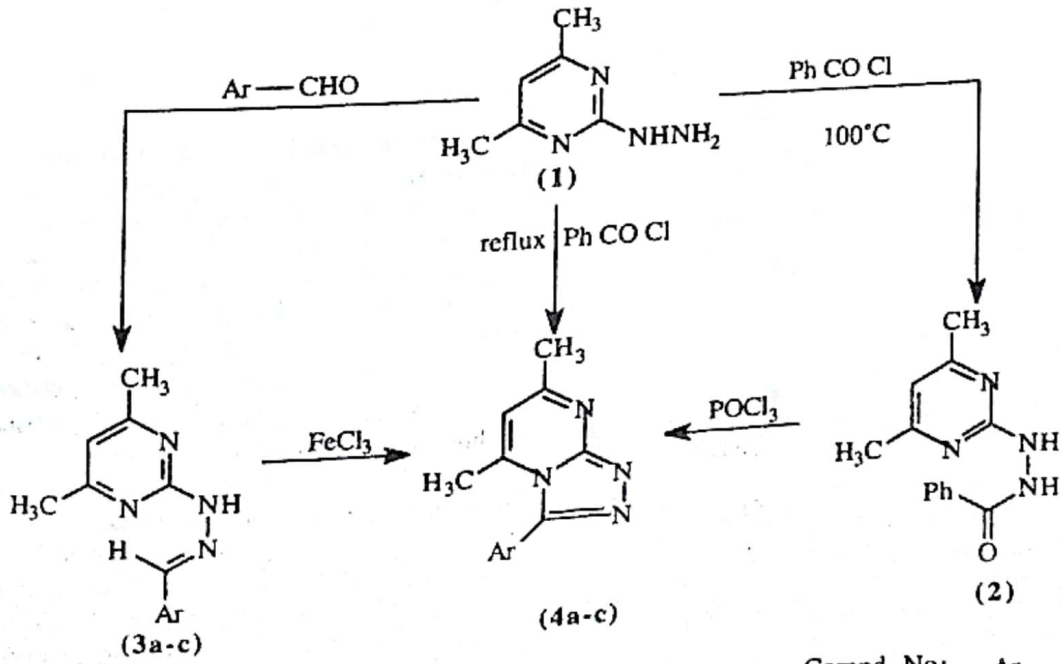
INTRODUCTION

Obviously, hydrazine derivatives are versatile reagents and have been used as synthetic intermediates for heterocyclic compounds[1-5]. Recently[6,7], we have shown the utility of these synthons as precursor for formation of different heterocycles of biological interest. In the present study, we concentrated our investigation on the behaviour of the title reagent in construction of a variety of new heterocyclic derivatives.

RESULTS AND DISCUSSION

2-Hydrazino-4,6-dimethylpyrimidine **1** undergoes ring closure with benzoyl chloride under reflux to give 5,7-dimethyl-3- phenyl-1,2,4-triazolo[4,3-a]pyrimidine **4a**. The reaction proceeds through N^2-benzoylation followed by thermal cyclization. This N^2-benzoyl derivative **2** was obtained from the reaction of **1** with benzoyl chloride at 100°C. Dehydrative cyclization of **2** was achieved either by using phosphoryl chloride or by fusion to form **4a.**

An alternative route for the synthesis of **4a** involved the reaction of benzaldehyde (4,6-dimethylpyrimidin-2-yl)hydrazone **3a** with ferric chloride. The formation of several 1,2,4-triazolo[4,3-a]pyrimidine derivatives (**4 b,c**) from the hydrazones **3b,c** shows the generality of the reaction pathway (Scheme 1).



Compd. No:	Ar
3a, 4a	Ph
3b, 4b	4-$CH_3OC_6H_4$
3c, 4c	4-ClC_6H_4

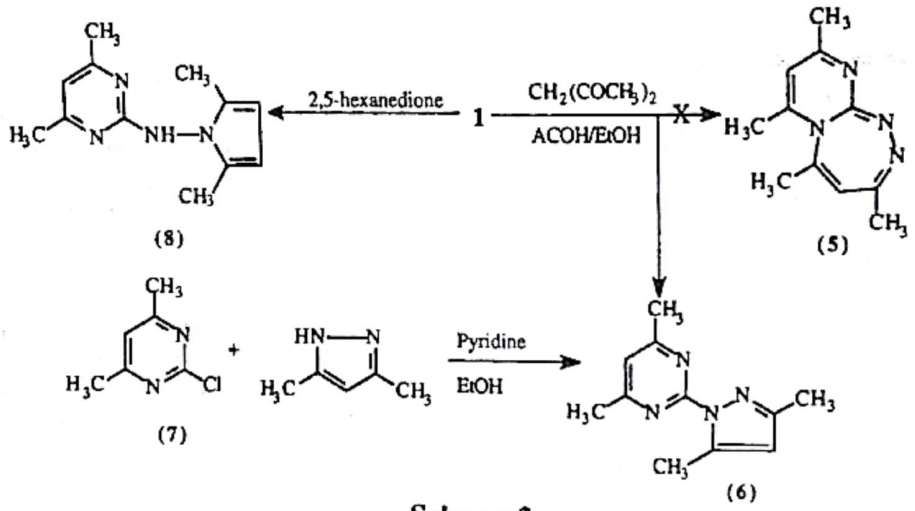
Scheme 1

Reaction of compound **1** with acetylacetone offers the competition between either formation of 7-membered 1,2,4-triazepine derivative **5** or formation of 5-membered pyrazole derivative **6**. The preference for the latter pathway was shown (Scheme 2). In marked contrast, Lancelot et al [8] reported the cyclization of 2-hydrazinopyridine with acetylacetone to afford pyrido-1,2,4-triazepine derivative. Neither elemental analysis nor the spectroscopic data could verify which type of compound (**5** or **6**) the product is. In our investigation, the structure of compound **6** was established by an unambiguous synthesis.

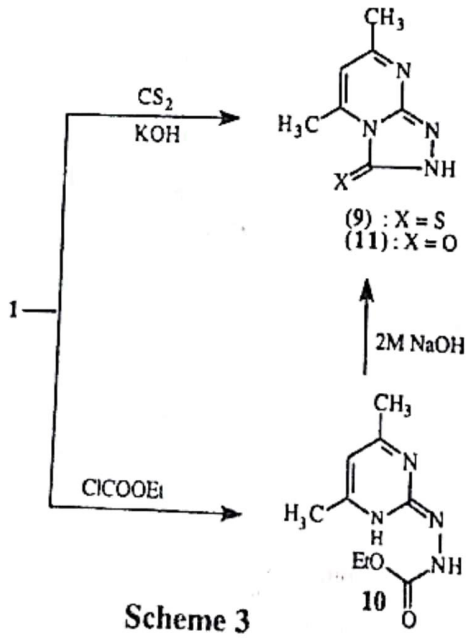
Thus, treating 2-chloro-4,6-dimethylpyrimidine**7** [9] with 1H-3,5-dimethylpyrazole[10] in pyridine gives 2-(3,5-dimethylpyrazolo)-4,6-dimethylpyrimidine **6**. The latter was identical in all respects (mp, ir and ^{1}Hnmr data) with the outcome of the cyclization of compound **1** with acetylacetone.

On the other hand, reaction of **1** with 2,5-hexanedione affords the pyrrole derivative **8**. This reaction pathway has a good literature precedent[11-13].

Furthermore, refluxing **1** with carbon disulphide in alcoholic potassium hydroxide afforded 5,7-dimethyl -1,2,4-triazolo[4,3-a]pyrimidine-3- thione **9**, while with ethyl chloroformate did not permit isolation of 5,7-dimethyl-1,2,4-triazolo[4,3-a] pyrimidin-3-one **11**. Instead, 2-ethoxycarbonyl-(4,6- dimethylpyrimidin-2-yl)hydrazine **10** was isolated. Stirring of compound **10** with 2 M NaOH eliminates a molecule of alcohol giving the cyclized product **11** (Scheme 3).



Scheme 2



Scheme 3

Moreover, reaction of **1** with the appropriate isothiocyanate gave the corresponding thiosemicarbazide (**12a-c**). Cylization of thiosemic-arbazides with α-halocarbonyl compounds was a subject of controversy. It was reported[14] that condensation of a thiosemic- arbazide with α-halocarbonyl compound may result in the formation of one or more five- or six -membered heterocyclic isomers. Herein, the hydrogen ion concentration of the medium favours the formation of specific structural isomers. Reaction of compound **12a** with ethyl bromoacetate either in neutral or basic media gave the five- membered thiazolidone derivative **13**, while in hydrochloric acid, the six- membered thiadiazinone direvative **14** was isolated as the sole product. Finaly, cyclodesulphurization of the thiosemicarbazides (**12a-c**) with dicyclohexylcarbodiimide (DCCD) gave the corresponding triazolo- [4,3-a]pyrimidine derivatives (**15 a-c**) (Scheme 4).

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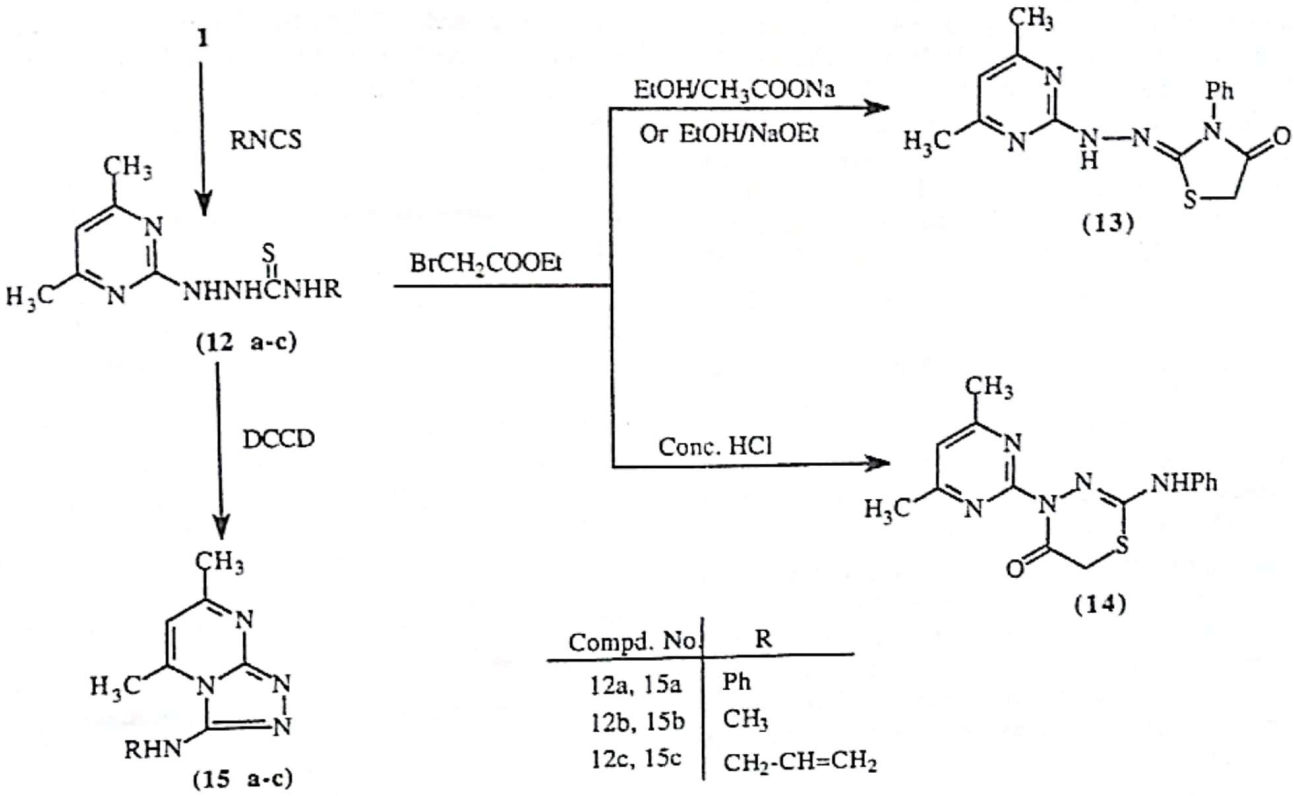

Compd. No.	R
12a, 15a	Ph
12b, 15b	CH_3
12c, 15c	CH_2-CH=CH_2

Scheme 4

EXPERIMENTAL

General : Melting points were uncorrected, SMP_2 melting point apparatus Microanalysis: Microanalytical Center, Cairo University; IR spectra (KBr): Shimadzu IR 435; ^{1}HNMR spectra [DMSO (d_6)] Jeol Fx 90 Q 90 MHz.

Synthesis of the compounds:

The intermediate 2-hydrazino-4,6-dimethylpyrimidine **1** was prepared by a standard procedure[15].

2-Benzoyl-1-(4,6-dimethylpyrimidin-2-yl)hydrazine(2):

A mixture of compound **1** (0.3g) and benzoyl chloride (5ml) was heated on a water bath for 5 h. The solid product thus obtained, was filtered, washed with benzene and crystallized from C_2H_5OH to give compound **2** in 90% yield; m.p 125-127°C; IR (cm^{-1}): 3180 (NH), 1660 (C=O). Analysis: $C_{13}H_{14}N_4O$ (242.27); Calcd.: % C, 64.44; % H, 5.82; % N, 23.12; Found: % C, 64.2; % H, 5.6; % N, 23.0.

Benzaldehyde /or substilutedbenzaldehyde(4,6-dimethylpyrimi-din-2-yl)hydrazones (3a-c):

A solution of the appropriate benzaldehyde (0.004 mol) and compound **1** (0.004 mol) in C_2H_5OH (10 ml) was refluxed for 2 h. The separated solid was filtered and crystallized from C_2H_5OH to give **(3a-c).**

Compoud **(3a)** : yield 81%, m.p. 155-157°C. IR (cm^{-1}): 3220, 3200 (NH); 1620 (C=N). ^{1}HNMR (δppm): 2.1 (s, 6H, $2CH_3$); 6.3 (s, 1H, N=CH); 6.9-7.9 (m, 6H, Ar-H). Analysis: $C_{13}H_{14}N_4$ (226.27); Calcd.: % C, 69.00; % H, 6.23; % N, 24.76; Found: % C, 69.2; % H 6.4; % N, 24.6.

Compound **(3b)** : yield 70%, m.p. 178-180°C; IR (cm^{-1}): 3300, 3180 (NH); 1610 (C=N). ^{1}HNMR (δppm): 2.0 (s, 6H, $2CH_3$); 3.45 (s, 3H, OCH_3); 6.25 (s, 1H, N=CH); 6.6-7.75 (m, 5H, Ar-H). Analysis: $C_{14}H_{16}N_4O$ (256.3); Calcd.: % C, 65.60; % H, 6.29; % N, 21.86. Found: % C, 65.3; % H, 6.3; % N, 21.8.

Compound **(3c)** : yield 65%, m.p. 158-160°C. IR (cm^{-1}): 3210, 3180 (NH); 1620 (C=N). ^{1}HNMR (δppm): 2.1 (s, 6H, $2CH_3$); 6.3 (s, 1H, N=CH); 6.8-7.85 (m, 5H, Ar-H). Analysis: $C_{13}H_{13}ClN_4$ (260.7); Calcd.: % C, 59.88; % H, 5.0; % N, 21.49; Found % C, 59.8; % H, 5.0; % N, 21.4.

5,7-Dimethyl-3-aryl 1,2,4-triazolo[4,3-a]pyrimidines (4a-c) :

Method A: A mixture of compound **2** (0.5 g), dry xylene (5ml) and $POCl_3$ (1ml) was refluxed for 8 h. The

cooled reaction mixture was diluted with petroleum ether (bp. 60-80°C) and the supernatant liquid decanted. The residue was dissolved in H_2O, neutralized with NH_4OH and the precipitated solid was filtered off and crystallized from C_2H_5OH to give (**4a**) in 40% yield, m.p. 170-172°C; IR (cm^{-1}): (No NH band); 2920 (aliphatic CH); 1620 (C=N). ^{1}HNMR (δppm). 2.72 (s, 3H, CH_3), 2.96 (s, 3H, CH_3), 7.44-8.44 (m, 6H, Ar-H). Analysis: $C_{13}H_{12}N_4$ (224.25); Calcd.: % C, 69.62; % H, 5.39; % N, 25.00; Found: % C, 70.0; % H, 5.8; % N, 25.1.

Method B: Compound **2** (1.0 g) was heated at 130°C for 15 min on a sand bath. The solid mass was extracted with benzene - petroleum ether (bp 60 - 80°) mixture. The extract was concentrated, and the separated solid was recrystallized from C_2H_5OH to give **4a** in 30% yield, with m.p. 170-172°C.

Method C: Compound **1** (1.0 g) was refluxed with benzoyl chorine (10 ml) for 5 h. Excess benzoyl chloride was distilled off under reduced pressure and the residue washed with hot petroleum ether (bp 60-80°C). The product was collected and crystallized from C_2H_5OH to give **4a** in 32% yield and m.p. 170-172°C.

Method D: A mixture of the appropriate hydrazone (**3a-c**) (0.001 mol), anhydrous $FeCl_3$ (0.5g) and triethyl orthoacetate (0.5 ml) in C_2H_5OH (10 ml) was heated under reflux for 8 h. The solution was filtered, concentrated and H_2O was added dropwise. The precipitated solid was filtered and crystallized from C_2H_5OH to give (**4a-c**).

Compound (**4a**) : yield 69% and m.p. 170-172°C.

Compound (**4b**) : yiled 65%, m.p. 218-220°C; IR (cm^{-1}): (No NH band); 2930 (C-H aliphatic); 1610 (C=N). ^{1}HNMR (δppm): 2.6 (s, 3H, CH_3), 2.8 (s, 3H, CH_3), 3.8 (s, 3H, OCH_3), 6.7 (s, 1H, Ar-H), 6.96-8.3 (m, 4H, Ar-H). Analysis: $C_{14}H_{14}N_4O$ (254.3); Calcd.: % C, 66.12; % H, 5.54; % N, 22.0; Found: % C, 66.0; % H, 5.3; % N, 22.0.

Compound **4c**: yield 60%, m.p. 242-244°C; IR (cm^{-1}): (no NH band); 2920 (C-H aliphatic); 1610 (C=N). ^{1}HNMR (δppm): 2.5 (s, 3H, CH_3); 2.7 (s, 3H, CH_3); 6.5 (s, 1H, Ar-H); 7.0-8.0 (m, 4H, Ar-H). Analysis: $C_{13}H_{11}ClN_4$ (258.74); Calcd.: % C, 60.34; % H; 4.28; % N, 21.65. Found: %C, 60.1; % H, 4.1; % N, 21.5.

2-(3,5-Dimethylpyrazolo)-4,6-dimethylpyrimidine (6):

Method A: Compound **1** (1.0 g) and acetylacetone (0.7 ml) in C_2H_5OH (50 ml) with a few drops of CH_3COOH were refluxed for 6 h. The reaction mixture was concentrated to obtain compound **6**; yield 80%; m.p. 60-62°C. ^{1}HNMR (δppm): 2.2 (s, 3H, CH_3), 2.4 (s, 6H, $2CH_3$), 2.5 (s, 3H, CH_3); 6.1 (s, 1H, pyrazole); 7.2 (s, 1H, pyrimidine). Analysis: $C_{11}H_{14}N_4$ (202.25); Calcd.: % C, 65.32, % H, 7.0; % N, 27.70; Found % C, 65.0; % H, 7.2; % N, 27.5.

Method B: 2-Chloro-4,6-dimethylpyrimidine **7** [9] (0.001 mol) and 3,5-dimethy- 1H-pyrazole[10] (0.001 mol) in absolute C_2H_5OH (30 ml) with a few drops of pyridine were refluxed for 6 h. The reaction mixture was concentrated to obtain compound **6** in 60% yield and m.p. 60-62°C.

2-[(2,5-Dimethylpyrrolo) amino]-4,6-dimethylpyrimidine (8) :

A mixture of compound **1** (0.004 mol) and 2,5-hexanedione (0.004 mol) in glacial CH_3COOH (5ml) was stirred at room temperature overnight. On dilution with H_2O, the separated solid was filtered off and recrystallized from C_2H_5OH to give compound **8** in 75% yield; m.p 157-158°C; IR(cm^{-1}): 3200 (NH), 2920 (CH_3), 1610 (C=N). ^{1}HNMR (δppm): 2.06 (s, 6H $2CH_3$, of pyrrole), 2.2 (s, 6H, $2CH_3$ of pyrimidine), 5.8 (s, 2H, pyrrole), 6.46 (s, 1H, pyrimidine), 9.3 (s, 1H, NH). Analysis; $C_{12}H_{16}N_4$ (216.27); Calcd: % C, 66.63; %H, 7.45; % N, 25.90; Found: % C, 66.8; % H, 7.1; %N, 25.5.

5,7-Dimethyl-1,2,4-triazolo[4, 3-a]pyrimidine-3-thione (9):

A mixture of compound **1** (0.3 g), C_2H_5OH (20 ml), KOH (0.1 g) and CS_2 (1ml) was refluxed for 5 h. The reaction mixture was filtered, concentrated and neutralized with CH_3COOH. The precipitated product was crystallized from C_2H_5OH to give **9** in yield (84%) and m.p. 300-302°C; IR (cm^{-1}): 3110 (NH), 1280 (C=S), 1610(C=N) Analysis: $C_7H_8N_4S$ (180.22); Calcd.: % C, 46.65; %H, 4.47; %N; 31.08; Found : %C, 46.6; %H, 4.4; %N, 31.0.

2-Ethoxycarbonyl-1- (4,6-dimethylpyrimidin-2-yl) hydrazine (10):

A mixture of **1** (0.3 g) and ethyl chloroformate (3 ml) was heated in pyridine (20 ml) on a water bath for 8 h. The reaction mixture was cooled and acidified with dilute CH_3COOH. The precipitated soild was crystallized from C_2H_5OH to give **10** in 60% yield and m.p. 142-144°C; IR (cm^{-1}): 3320, 3220 (NH), 1745 (C=O). Analysis: $C_9H_{14}N_4O_2$ (210.22); Calcd.: % C, 51.41; % H, 6.71; % N, 26.65; Found :% C, 51.2; % H, 6.5; % N, 26.7.

5,7-Dimethyl-1,2,4-triazolo[4,3-a]pyrimidin-3-one(11):

To a solution of compound **10** (0.3g) in C_2H_5OH (10 ml), was added 2M NaOH (4ml) and the mixture was stirred at room temperature for one h. The solution was diluted with H_2O (30 ml) and neutralized to litmus with dil HCl. The precipitated solid was collected, washed with H_2O and crystallized from C_2H_5OH to give **11** in 50% yield; m.p. 155-157°C; IR (cm^{-1}): 3190 (NH), 1710 (C=O). Analysis: $C_7H_8N_4O$ (164.16); Calcd.: % C, 51.21; % H, 4.91; % N, 34.13, Found: % C, 51.0; % H, 5.0; % N, 34.4.

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1-(4,6-Dimethylpyrimidin-2-yl)-4-substituted thiosemic -arbazides (12 a-c):

A mixture of compound **1** (0.001 mol) and the appropriate iso- thiocyanate (0.001 mol) in C_2H_5OH (10 ml) was refluxed for one h. On cooling, the separated soild was filtered and crystallized from C_2H_5OH to give **12 a-c**.

Compound (**12a**) : Yield 86%, m.p. 195-197°C; IR (cm^{-1}): 3300, 3200 (NH), 1600 (C=N), 1340 (C=S). ^{1}HNMR (δppm): 2.6 (s, 6H, $2CH_3$), 6.96 (s, 1H, pyrimidine), 7.68-8.00 (m, 5H, Ar-H), 8.2-8.4(s, 2H, NH-NH), 9.2 (s, 1H, NH). Analysis: $C_{13}H_{15}N_5S$ (273.34); Calcd.: % C, 57.11; % H, 5.5; % N, 25.61; Found: % C, 57.3; % H, 5.6; %N, 25.7.

Compound (**12b**) : Yield 83%, m.p 229-231°C. IR (cm^{-1}): 3250, 3160 (NH), 1335 (C=S). ^{1}HNMR (δ ppm): 2.52 (s, 6H, $2CH_3$), 3.10 (s, 3H, N-CH_3), 6.96 (s, 1H, pyrimidine), 7.68-8.00 (s, 2H, NH-NH_2), 8.68 (s, 1H, NH). Analysis: $C_8H_{13}N_5S$ (211.27); Calcd.: %, C, 45.50; % H, 6.20; % N, 33.1, Found: % C, 45.3; H, 6.3; % N, 33.0

Compound (**12c**) : Yield 81%, m.p. 231- 233°C. IR (cm^{-1}): 3320, 3290 (NH), 1340 (C=S). ^{1}HNMR (δppm): 2.56 (s, 6H, $2CH_3$), 4.5 (dd, 2H, N-CH_2), 5.36 - 5.56 (m, 2H, =CH_2), 5.92-6.4 (m, 1H, CH=), 6.88 (s, 1H, pyrimidine), 8.3-8.5(s, 2H, NH-NH_2) 8.8 (s, 1H, NH). Analysis: $C_{10}H_{15}N_5S$ (237.3); Calcd.: %C, 50.6; %H, 6.37; %N, 29.5; Found: % C, 50.3; %H, 6.7; % N, 29.4.

N-(4,6-Dimethylpyrimidin-2-yl)-N`[3-phenyl-4-oxo-thiazolidin-2- ylidene) hydrazine (13):

Method A: A mixture of **12a** (0.001 mol), $BrCH_2COOEt$ (0.001 mol) and fused CH_3COONa (0.01 mol) in dry absolute C_2H_5OH (20ml) was heated for 5 h. The excess solvent was evaporated under vaccum. On dilution with H_2O, the precipitated solid was filtered and crystallized from C_2H_5OH to give compound **13** in yield (79%); m.p. 202-204°C; IR (cm^{-1}): 3200 (NH), 1750 (C=O), 1640 (C=N). Analysis. $C_{15}H_{15}N_5OS$ (313.36); Calcd.: % C, 57.49; %H, 4.82; %N, 22.35; Found : % C, 57.2; %H, 5.0; % N, 22.5.

Method B: A mixture of compound **12a** (0.01 mol) and NaOEt (0.23 g Na metal in 10 ml dry absolute C_2H_5OH) was heated with stirring for 2 h; $BrCH_2COOEt$ (0.01 mol) was then added and the mixture was heated for 5 h and the excess solvent was evaporated under vaccum. On dilution with H_2O, the precipitated solid was filtered and crystallized from C_2H_5OH to give **13** in 60% yield and m.p. 202-204°C.

2-Phenylamino-5-oxo-(4,6-dimethylpyrimidin-2-yl)-6-hydro-1,3,4 thiadiazine (14):

A mixture of compound **12a** (0.001 mol) and $BrCH_2COOEt$ (0.001 mol) in conc. HCl (10 ml) was refluxed for one h. The separated solid after cooling was filtered and crystallized from C_2H_5OH to give compound **14** in 40% yield and m.p. 148-150°C (decomp.); IR (cm^{-1}): 3390 (NH); 1745 (C=O); 1670 (C=N). ^{1}HNMR (δ ppm): 2.5 (s, 6H, $2CH_3$); 2.7 (s, 3H, CH_3); 4.3 (s, 2H, CH_2); 6.9-7.9 (m, 6H, Ar-H); 9.75 (s, 1H, NH). Analysis: $C_{15}H_{16}Cl\ N_5OS$ (349.9); Calcd.: % C, 51.48; % H, 4.60; N, 20.00; Found: % C, 51.5; %H, 4.7; %N, 20.2.

3-Substitutedamino-5,7-dimethyl-1,2,4-triazolo[4,3-a]-pyrimidine (15 a-c):

A mixture of compoud **12 a-c** (0.001 mol) and DCCD (0.001 mol) in toluene (10 ml) was refluxed for 5 h. The reaction mixture was cooled and the separated solid was filtered and crystallized from toluene to give **15a-c.**

Compound **15a:** yield, 88%; m.p. 170 - 172°C; IR (cm^{-1}): 3250 (NH). ^{1}HNMR (δ ppm): 2.72 (s, 6H, $2CH_3$), 7.12-7.52 (m, 6H, Ar-H), 8.88 (s, 1H, NH). Analysis: $C_{13}H_{13}N_5$ (239.3); Calcd.: % C, 65.25;% H, 5.47; % N, 29.27; Found: % C, 65.0; % H, 6.0; % N, 29.3.

Compound **15b:** yield 85%; m.p. 272-274°C; IR (cm^{-1}): 3310 (NH). ^{1}HNMR (δppm): 2.6 (s, 6H, $2CH_3$), 3.6 (s, 3H, NCH_3), 7.0 (s, 1H, pyrimidine), 8.2 (s, 1H, NH). Analysis $C_8H_{11}N_5$ (177.2): Calcd.: % C, 54.22; % H, 6.25; % N, 39.5%, Found: % C, 54.2; %H, 6.0; % N, 39.5.

Compound **15c** : yield 80%; m.p. 210-212°C; IR (cm^{-1}): 3300 (NH). Analysis $C_{10}H_{13}N_5$ (203.24) : Calcd.: % C, 59.10; % H, 6.44; % N, 34.46; Found: % C, 59.0; % H, 6.2; % N, 34.2.

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محمد ابراهيم العشماوى - زكريا كامل عبد السميع - سعيد احمد الفقى - نرمين عونى عثمان

قسم الكيميا ء العضوية الصيدلية - كلية الصيدلة - جامعة الزقازيق - مصر

فى هذا البحث تم دراسة تفاعلات المركب الأولى ٢-هيدرازينو-٤ ، ٦- ثنائى ميثيل بيريميدين فى تشييد العديد من الحلقات الملتحمه غير متجانسة الحلقه .

تم حولقة المركب الاولى مع كلوريد البنزويل الى مركب ٥ ، ٧ - ثنائى ميثيل -٣-فينيل-١، ٢، ٤ - ترايازولو [٤ ، ٣ -أ]- بيريميدين.

وكذلك امكن الحصول على المركب الاخير من تفاعلات الهيدرازونات المشتقة من المركب الاولى باستخدام كلوريد الحديدك .

وعند تفاعل المركب الاولى مع أسيتيل اسيتون تم الحصول على مركب البيرازول بدلا من مركب ١ ، ٢ ، ٤-ترايازين كما هو منشور فى التفاعل المشابه سابقاً . وقد تم اثبات التركيب الكيميائى للمركب الناتج بتحضيره بطريقة اخرى .

كذلك تم تحضير مركب البيرول من حولقة المركب الاولى مع ٢، ٥-هكسان ثنائى الكيتون .

وقد تم الحصول على مشتقات ١، ٢، ٤-ترايازولو [٤ ، ٣-أ] بيريميدين من تفاعل المركب الاولى مع كل من ثنائى كبريتيد الكربون وكلوروفورمات الايثيل على التوالى .

ايضا تم تحضير الثيوسيمى كريازيدات المقابله للمركب الاولى ودراسة تفاعلاتها مع بروموخلات الايثيل فى الوسط القاعدى والحامضى على التوالى وكذلك مع ثنائى سيكلوهكسيل كربوثنائى الأيميد .