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## Substituted pyridopyrimidinones. Part IV. 2-Chloro-4*H*-pyrido[1,2-*a*]pyrimidin-4-one as a synthone of some new heterotricycles

MOHAMED ABASS\*, MOSTAFA M. ISMAIL, WAFAA R. ABDEL-MONEM  
and AISHA S. MAYAS

*Department of Chemistry, Faculty of Education, Ain Shams University,  
Roxy, Cairo 11757, Egypt*

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**Abstract:** 2-Chloro-4*H*-pyrido[1,2-*a*]pyrimidin-4-one (**1**) was utilized as a synthone precursor to prepare novel heterotricyclic systems. 2-Azido and 2-hydrazino derivatives (**2** and **3**) were obtained by nucleophilic replacement evolving compound **1**. The hydrazine derivative **3** was transformed into the azido derivative **2** by nitrosation. Treatment of compound **3** with [bis(methylthio)methylene]malononitrile afforded 2-pyrazolylpyridopyrimidine **4**. When compound **1** was reacted with 5-amino-3-(methylthio)-1*H*-pyrazole-4-carbonitrile, the same compound **4** was obtained with no evidence for the production of (pyrazolyl-amino)pyridopyrimidine **5** or pyrazolodipyridopyrimidine **6**. Poly-functionalized dipyridopyrimidine **8** was obtained by reaction of compound **1** with 2-[(methylthio)-(phenylamino)methylene]propanedinitrile. Cyanoguanidine was reacted with compound **1** to afford *N*-pyridopyrimidinylguanidine **9**, which was subjected to cyclization reaction, in presence of piperidinium acetate, to give pyridopyrimidopyrimidine **10**.

**Keywords:** pyridopyrimidine; dipyridopyrimidine; pyridopyrimidopyrimidine.

### INTRODUCTION

Recently, a convenient new synthesis of 2-chloro-4*H*-pyrido[1,2-*a*]pyrimidin-4-one (**1**) was described.<sup>1</sup> It is well-known that this type of  $\alpha$ -haloheterocyclic compounds are susceptible to synthetically important nucleophilic substitutions.<sup>2–5</sup> Many hetero-fused pyrimidines exhibit attractive cancer chemotherapy properties as antitumor agents.<sup>6</sup> Risperidone, SSR6907, and ramastine are derivatives of pyrido[1,2-*a*]pyrimidin-4-one, which show antipsychotic activity.<sup>7–9</sup> Dominguez *et al.*<sup>10</sup> reported that some hetero-fused tricyclic systems exhibited significant antimalarial activity. It was cited that the biological reac-

\*Corresponding author. E-mail: m.abass@chemist.com  
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tivity of this category of compounds is essentially due to presence of the pyrido[1,2-*a*]pyrimidinone moiety in their molecular structure.<sup>11</sup> In this study, it was planned to utilize the readily available chloro derivative to obtain novel polycyclic compounds, which could be expected to possess antimalarial activity.

## RESULTS AND DISCUSSION

### *Analytical and spectral characterization of the prepared compounds*

**2-Azido-4H-pyrido[1,2-*a*]pyrimidin-4-one (2).** Anal. Calcd. for C<sub>8</sub>H<sub>5</sub>N<sub>5</sub>O (FW 187.16): C, 51.34; H, 2.69; N, 37.42 %; Found: C, 50.96; H, 2.54; N, 37.40 %. IR (KBr, cm<sup>-1</sup>): 3073, 3042, 2129 (N<sub>3</sub>), 1718 (C=O), 1634 (C=N), 1565, 1517, 1455, 1412, 1110, 930, 839, 782, 760. <sup>1</sup>H-NMR (200 MHz, DMSO-*d*<sub>6</sub>, δ / ppm): 5.85 (1H, *s*, C<sub>3</sub>-H), 7.55 (1H, *dd*, *J* = 7.5, 3.6 Hz, C<sub>7</sub>-H), 7.63 (1H, *d*, *J* = 7.4 Hz, C<sub>9</sub>-H), 8.08 (1H, *dd*, *J* = 7.4, 3.4 Hz, C<sub>8</sub>-H), 8.99 (1H, *d*, *J* = 7.5 Hz, C<sub>6</sub>-H). MS (*m/z* (*I* / %)): M<sup>+</sup> 187 (34).

**2-Hydrazino-4H-pyrido[1,2-*a*]pyrimidin-4-one (3).** Anal. Calcd. for C<sub>8</sub>H<sub>8</sub>N<sub>4</sub>O (FW 176.18): C, 54.54; H, 4.58; N, 31.80 %; Found: C, 54.42; H, 4.42; N, 31.77 %. IR (KBr, cm<sup>-1</sup>): 3428, 3340, 3328, 3270 (NHNH<sub>2</sub>), 3072, 1690 (C=O), 1636 (C=N), 1610, 1570, 1518, 1445, 1352, 1080, 776. <sup>1</sup>H-NMR (200 MHz, DMSO-*d*<sub>6</sub>, δ / ppm): 4.30 (2H, *b*, NH<sub>2</sub>), 5.62 (1H, *s*, C<sub>3</sub>-H), 7.31 (*dd*, 1H, *J* = 7.4, 3.6 Hz, C<sub>7</sub>-H), 7.56 (1H, *d*, *J* = 7.2 Hz, C<sub>9</sub>-H), 7.95 (1H, *dd*, *J* = 7.2, 3.5 Hz, C<sub>8</sub>-H), 8.12 (1H, *b*, NH), 8.93 (1H, *d*, *J* = 7.4 Hz, C<sub>6</sub>-H). MS (*m/z* (*I* / %)): M<sup>+</sup> 176 (80).

**5-Amino-3-(methylthio)-1-(4-oxo-4H-pyrido[1,2-*a*]pyrimidin-2-yl)-1H-pyrazole-4-carbonitrile (4).** Anal. Calcd. for C<sub>13</sub>H<sub>10</sub>N<sub>6</sub>OS (FW 298.33): C, 52.34; H, 3.38; N, 28.17; S, 10.75 %; Found: C, 52.33; H, 3.38; N, 28.10; S, 10.50 %. IR (KBr, cm<sup>-1</sup>): 3360, 3265 (NH), 3115, 3071, 3043, 2924, 2210 (CN), 1676 (C=O), 1614 (C=N), 1549, 1501, 1457, 819, 762. <sup>1</sup>H-NMR (200 MHz, DMSO-*d*<sub>6</sub>, δ / ppm): 2.56 (3H, *s*, SCH<sub>3</sub>), 6.59 (1H, *s*, C<sub>3</sub>-H), 7.43 (1H, *m*, C<sub>7</sub>-H), 8.08 (2H, *m*, C<sub>9</sub>-H + C<sub>8</sub>-H), 8.38 (2H, *s*, NH<sub>2</sub>), 8.99 (1H, *d*, *J* = 7.4 Hz, C<sub>6</sub>-H). <sup>13</sup>C-NMR (70 MHz, DMSO-*d*<sub>6</sub>, δ / ppm): 38.5, 93.7, 100.1, 116.4, 118.0, 121.3, 124.2, 130.6, 133.8, 148.7, 149.7, 154.4, 158.9.

**4-Imino-2-(methylthio)-5-oxo-1-phenyl-1,5-dihydro-4H-dipyrido-[1,2-*a*:2',3'-*d*]-pyrimidine-3-carbonitrile (8).** Anal. Calcd. for C<sub>19</sub>H<sub>13</sub>N<sub>5</sub>OS (FW 359.41): C, 63.50; H, 3.65; N, 19.49; S, 8.92 %; Found: C, 64.0; H, 3.30; N, 19.20; S, 8.60 %. IR (KBr, cm<sup>-1</sup>): 3292 (NH), 3065, 3039, 3001, 2928, 2202 (CN), 1671 (C=O), 1621 (C=N), 1596, 1524, 1492, 1261, 759; <sup>1</sup>H-NMR (200 MHz, DMSO-*d*<sub>6</sub>, δ / ppm): 2.08 (3H, *s*, SCH<sub>3</sub>), 7.05–8.29 (8H, *m*, 5H<sub>arom.</sub> + C<sub>7</sub>-H + C<sub>9</sub>-H + C<sub>8</sub>-H), 8.85 (1H, *d*, *J* = 7.5 Hz, C<sub>6</sub>-H), 10.19 (1H, *s*, NH). <sup>13</sup>C-NMR (70 MHz, DMSO-*d*<sub>6</sub>, δ / ppm): 35.5, 89.3, 96.4, 115.3, 117.7, 122.4, 112.9, 125.2, 129.7, 136.2, 139.0, 140.5, 142.1, 151.2, 156.0, 160.3, 165.9.

**N-Cyano-N'-(4-oxo-4H-pyrido[1,2-*a*]pyrimidin-2-yl)guanidine (9).** Anal. Calcd. for C<sub>10</sub>H<sub>8</sub>N<sub>6</sub>O (FW 228.21): C, 52.63; H, 3.53; N, 36.83 %; Found: C,

52.31; H, 3.40; N, 36.72 %; IR (KBr,  $\text{cm}^{-1}$ ): 3429, 3383 (NH), 3334, 3249, 3194 (NH), 3151, 2920, 2207 (CN), 1669 (C=O), 1639 (C=N), 1571, 1506, 1434, 1357, 777, 721, 669.  $^1\text{H-NMR}$  (200 MHz,  $\text{DMSO-}d_6$ ,  $\delta$  / ppm): 5.40 (1H, *s*, NH, disappeared with  $\text{D}_2\text{O}$ ), 6.0 (1H, *s*, NH, disappeared with  $\text{D}_2\text{O}$ ), 6.55 (1H, *s*,  $\text{C}_3\text{-H}$ ), 7.40 (1H, *dd*,  $J = 7.5, 3.6$  Hz,  $\text{C}_7\text{-H}$ ), 7.49 (1H, *d*,  $J = 7.5$  Hz,  $\text{C}_9\text{-H}$ ), 7.82 (1H, *dd*,  $J = 7.5, 3.6$  Hz,  $\text{C}_8\text{-H}$ ), 7.95–8.08 (1H, *bs*, NH, disappeared with  $\text{D}_2\text{O}$ ), 8.80 (1H, *d*,  $J = 7.5$  Hz,  $\text{C}_6\text{-H}$ ).  $^{13}\text{C-NMR}$  (70 MHz,  $\text{DMSO-}d_6$ ,  $\delta$  / ppm): 94.3, 115.5, 119.2, 122.6, 124.9, 138.0, 151.2, 157.3, 164.8, 165.8; MS ( $m/z$  ( $I$  / %)):  $\text{M}^+$  228 (28).

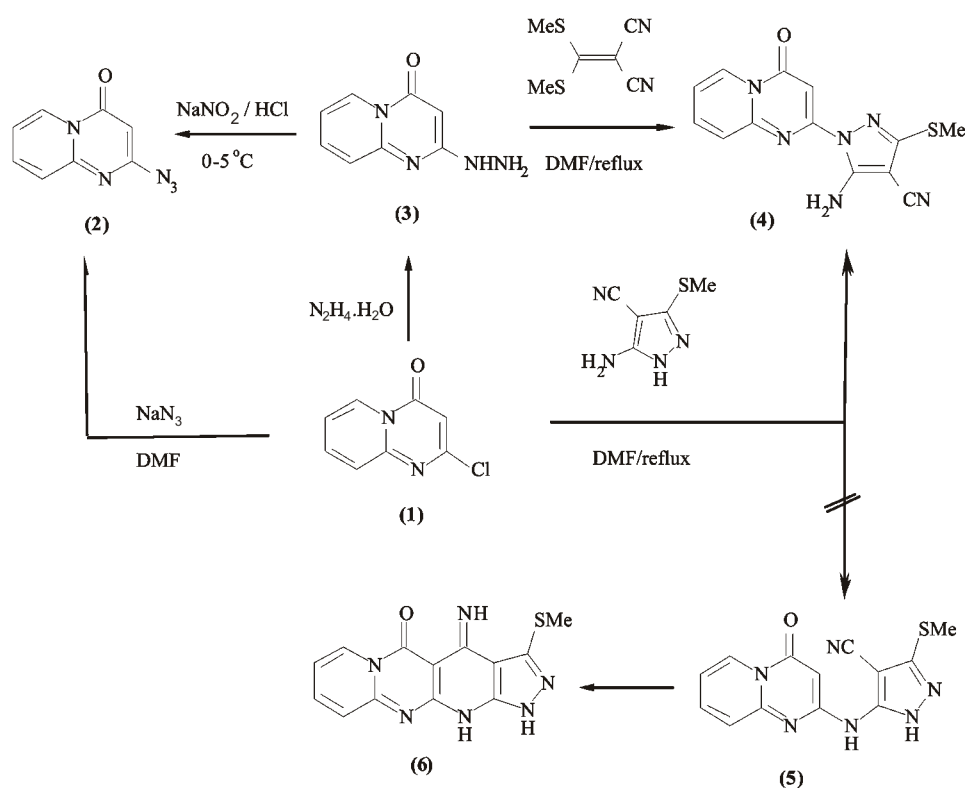
*2,4-Diamino-5H-pyrido[1,2-a]pyrimido[4,5-d]pyrimidin-5-one (10)*. Anal. Calcd. for  $\text{C}_{10}\text{H}_8\text{N}_6\text{O}$  (FW 228.21): C, 52.63; H, 3.53; N, 36.83 %; Found: C, 52.50; H, 3.20; N, 36.50 %. IR (KBr,  $\text{cm}^{-1}$ ): 3435, 3328 ( $\text{NH}_2$ ), 3221, 3172 (NH), 3075, 2936, 1668 (C=O), 1620 (C=N), 1585, 1535, 1440, 1330, 778, 725.  $^1\text{H-NMR}$  (200 MHz,  $\text{DMSO-}d_6$ ,  $\delta$  / ppm): 6.59 (2H, *s*,  $\text{NH}_2$  disappeared with  $\text{D}_2\text{O}$ ), 6.68 (2H, *s*,  $\text{NH}_2$  disappeared with  $\text{D}_2\text{O}$ ), 7.44 (1H, *dd*,  $J = 7.6, 3.7$  Hz,  $\text{C}_7\text{-H}$ ), 7.52 (1H, *d*,  $J = 7.5$  Hz,  $\text{C}_9\text{-H}$ ), 7.89 (1H, *dd*,  $J = 7.5, 3.6$  Hz,  $\text{C}_8\text{-H}$ ), 8.94 (1H, *d*,  $J = 7.6$  Hz,  $\text{C}_6\text{-H}$ ).  $^{13}\text{C-NMR}$  (70 MHz,  $\text{DMSO-}d_6$ ,  $\delta$  / ppm): 98.5, 114.9, 125.4, 128.0, 136.2, 146.2, 154.6, 158.9, 165.5, 169.8; MS ( $m/z$  ( $I$  / %)):  $\text{M}^+$  228 (100).

### Chemistry

Reaction of the chloro-compound **1** with sodium azide was performed in DMF, leading to 2-azidopyridopyrimidinone (**2**).<sup>12,13</sup> The IR spectrum of the product showed a sharp medium peak at  $\nu_{\text{max}}$  2129  $\text{cm}^{-1}$ , which is characteristic for the azide function. The same compound was afforded when 2-hydrazinopyridopyrimidinone (**3**) was treated with *in situ* freshly obtained nitrous acid. This hydrazine–azido conversion was previously described by Kovačič *et al.*,<sup>12</sup> albeit they did not give any characterization for the structure of the azido product but the same melting point was obtained. The hydrazine **3** was preliminary obtained *via* refluxing the chloro compound **1** with hydrazine hydrate, according to the method described by Oakes and Rydon,<sup>14</sup> who did not give spectral characterization of the structure of the product. The IR and  $^1\text{H-NMR}$  spectra of the hydrazine **3** are given herein to fortify the proposed structure (Scheme 1).

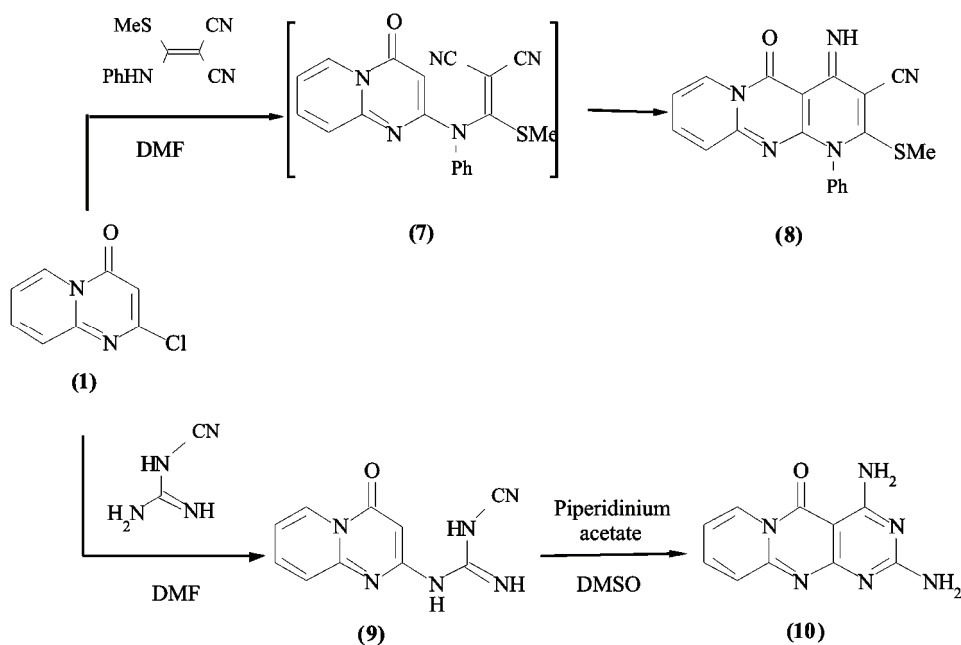
Reaction of the chloro-compound **1** with 5-amino-3-(methylthio)-1*H*-pyrazole-4-carbonitrile,<sup>15</sup> in boiling DMF, did not lead to the expected 5-(pyrido-pyrimidinylamino)-1*H*-pyrazole-4-carbonitrile (**5**) or its targeted cyclized isomer pyrazolo[3',4':1,6]pyrido-[2,3-*d*]pyrido[1,2-*a*]pyrimidinone (**6**). IR spectroscopy revealed the occurrence of the cyano function at  $\nu_{\text{max}}$  2210  $\text{cm}^{-1}$ . The  $^1\text{H-NMR}$  spectrum showed the existence of  $\text{NH}_2$  at  $\delta$  8.38 ppm, which disappeared on addition of  $\text{D}_2\text{O}$ , besides  $\delta$  6.59 ppm, which is specific for position 3 of pyridopyrimidinone and the four proton set of the pyridine ring. These results suggested

that the obtained compound is 1-(pyridopyrimidin-2-yl)pyrazole (**4**). The production of the pyrazole **4** can be attributed to the greater nucleophilicity character of the ring nitrogen compared with the  $\alpha$ -amino group, which backs to the mesomeric effect. However, a clear-cut establishment of the structure was achieved from the cyclization of the hydrazine **3** with [bis(methylthio)methylene]malononitrile.<sup>16,17</sup> This reaction was reported for the formation of 1-substituted 5-amino-3-(methylthio)-1*H*-pyrazole-4-carbonitriles starting from hydrazines<sup>18</sup> (Scheme 1).



Scheme 1.

2-[(Methylthio)(phenylamino)methylene]propanedinitrile was subjected to reaction with the chloro compound **1** in refluxing DMF. Surprisingly, the intended 2-{(methylthio)[phenyl(4-oxo-4*H*-pyrido[1,2-*a*]pyrimidin-2-yl)amino]}propanedinitrile (**7**) was not isolated. The spectra of the product showed the occurrence of an imino function and at the same time the disappearance of the characteristic signal due to C–H at position 3. This suggested that the obtained product was cyclized in a cascade nucleophilic reaction to yield dipyrido[1,2-*a*:2',3'-*d*]pyrimidine **8** (Scheme 2).



Reaction of the chloro compound **1** with cyanoguanidine in boiling DMF gave the claimed *N*-cyano-*N'*-(pyridopyrimidinyl)guanidine **9**. The IR spectrum exhibited a sharp absorbance peak at  $\nu_{\max}$  2207  $\text{cm}^{-1}$  due to the CN function. Trials for cyclization of this guanidine derivative met success by the action of piperidinium acetate in boiling DMSO. It is thought that the relatively high boiling temperature of DMSO is conditional for such a reaction because attempts to perform the same reaction in THF, dioxane, and DMF were not successful. The structure of the product was confirmed from its spectral data which suggested that the product is pyrido[1,2-*a*]pyrimido[4,5-*d*]pyrimidinone **10** (Scheme 2).<sup>10</sup>

#### EXPERIMENTAL

Melting points were determined in an open capillary tube on a digital Gallen-Kamp MFB-595 apparatus. The IR spectra were taken on a Perkin-Elmer FT-IR 1650, using samples in KBr disks. The  $^1\text{H}$ -NMR (200 MHz) and  $^{13}\text{C}$ -NMR (70 MHz) spectra were recorded on Varian Gemini-200 spectrometer, using  $\text{DMSO}-d_6$  as the solvent and TMS as the internal reference. The mass spectrum was determined on a HP-MS 5988 mass spectrometer by direct inlet, operating at 70 eV. Elemental microanalysis was performed on a Perkin Elmer CHN-2400 Analyzer.

##### 2-Azido-4H-pyrido[1,2-*a*]pyrimidin-4-one (**2**)

**Method A.** To a solution of the chloro-derivative **1** (10 mmol) in DMF (20 mL), sodium azide (15 mmol) was added and the reaction mixture was heated on a boiling water bath for 2

h. Then the reaction mixture was diluted with ice-water (20 mL) and left to stand for 1 h. The product was filtered and crystallized from DMF/H<sub>2</sub>O (1:1). Yield 80 %, m.p. 160–161 °C.

*Method B.* To a stirred cold (0–5 °C) solution of the hydrazino derivative **3** (10 mmol), in 2 M hydrochloric acid (10 mL), an aqueous 1 M sodium nitrite solution (10 mL) was added dropwise. Then, stirring was continued at room temperature for 1 h and the obtained precipitate was filtered and crystallized from DMF/H<sub>2</sub>O (1:1) to give the azido derivative **2**. Yield: 72 %, m.p. 160–161 °C (m.p. 160–163 °C<sup>12</sup>).

*2-Hydrazino-4H-pyrido[1,2-a]pyrimidin-4-one (3)*

A mixture of the chloro-compound **1** (10 mmol) and hydrazine hydrate (15 mmol) in DMF (10 mL) was refluxed for 1 h. Then the reaction mixture was poured onto crushed ice and the solid deposits were filtered and crystallized from ethanol to give the hydrazine compound **3**. Yield: 69 %, m.p. 240–242 °C (m.p. 245 °C<sup>14</sup>).

*5-Amino-3-(methylthio)-1-(4-oxo-4H-pyrido[1,2-a]pyrimidin-2-yl)-1H-pyrazole-4-carbonitrile (4)*

*Method A.* To a solution of the hydrazino compound **3** (5 mmol) in DMF (20 mL), [bis-(methylthio)methylene]malononitrile (5 mmol) was added and the reaction mixture was refluxed for 2 h. The solid product, which separated after cooling, was filtered using a suction pump, washed thoroughly with ethanol and crystallized from DMF. Yield: 89 %, m.p. > 300 °C.

*Method B.* A mixture of the chloro-compound **1** (5 mmol) and 5-amino-(3-methylthio)-1H-pyrazole-4-carbonitrile<sup>15</sup> (5 mmol) in DMF (20 mL) was boiled under reflux for 2 h. Afterwards, the reaction mixture was left to cool to room temperature. The so-obtained crystalline deposit was filtered and recrystallized from DMF to give pyridopyrimidylpyrazole **4**. Yield: 77 %.

*4-Imino-2-(methylthio)-5-oxo-1-phenyl-1,5-dihydro-4H-dipyrido-[1,2-a:2',3'-d]pyrimidine-3-carbonitrile (8)*

A mixture of the chloro-compound **1** (10 mmol) and 2-[(methylthio)(phenylamino)-methylene]propanedinitrile (10 mmol) in DMF (25 mL) was heated under reflux for 4 h. Subsequently, the reaction mixture was left to cool to room temperature. The obtained yellow crystalline material was filtered off and recrystallized from DMF to give compound **8**. Yield: 73 %, m.p. 257–258 °C.

*N-Cyano-N'-(4-oxo-4H-pyrido[1,2-a]pyrimidin-2-yl)guanidine (9)*

A mixture of the chloro compound **1** (5 mmol) and cyanoguanidine (5 mmol) in DMF (20 mL) was heated under reflux for 4 h. After cooling to room temperature, the mixture was diluted with cold water and the solid that deposited was collected by filtration and crystallized from acetone to give the guanidine derivative **9**. Yield: 80 %, m.p. 178–180 °C.

*2,4-Diamino-5H-pyrido[1,2-a]pyrimido[4,5-d]pyrimidin-5-one (10)*

A solution of the guanidine derivative **9** (5 mmol) and piperidinium acetate (from 0.15 g piperidine and 0.1 g acetic acid, 1.5 mmol) in DMSO (25 mL) was heated under reflux for 4 h. The reaction mixture was left to cool to room temperature. The so-formed precipitate was filtered off and washed several times with absolute ethanol. The product was crystallized from DMF to give the yellowish orange diamine **10**. Yield 85 %, m.p. > 300 °C (Lit.<sup>10</sup> data is not available).

## ИЗВОД

СУПСТИТУИСАНИ ПИРИДОПИРИМИДИНОНИ. ДЕО IV.  
2-ХЛОРО-4Н-ПИРИДО[1,2-*a*]ПИРИМИДИН-4-ОН КАО СИНТОН  
НЕКИХ НОВИХ ХЕТЕРОЦИКЛИЧНИХ ЈЕДИЊЕЊА

MOHAMED ABASS, MOSTAFA M. ISMAIL, WAFAA R. ABDEL-MONEM и AISHA S. MAYAS

*Department of Chemistry, Faculty of Education, Ain Shams University, Roxy, Cairo 11757, Egypt*

2-Хлоро-4Н-пиридо[1,2-*a*]пириимидин-4-он (**1**) је коришћен као синтон-прекурсор за синтезу нових хетеротрицикличних система. 2-Азидо- и 2-хидразино-деривати (**2** и **3**) добијени су нуклеофилном супституцијом из једињења **1**. Хидразино-дериват **3** трансформисан је нитрозовањем у азидо-дериват **2**. Третирањем једињења **3** са [бис(метилтио)метиле]мало-нонитрилом добијен је 2-пиразолилпиридопириимидин (**4**). Реакцијом једињења **1** са 5-амино-3-(метилтио)-1Н-пиразол-4-карбонитрилом добијено је исто једињење **4** без грађења пиразолиламино)пиридопириимидино **5** или пиразолодипиридопириимидина **6**. Поли-функционализован дипиридопириимидин **8** добивен је реакцијом једињења **1** са 2-[(метилтио)-фениламино)метиле]пропандинитрилом. Цијаногванидин је третиран са једињењем **1** градећи N-пиридопириимидинилгванидин **9**, који реакцијом циклизације у присуству пиперидинијум-ацетата, даје пиридопириимидопириимидин **10**.

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