Simple synthesis of alkyl 5-oxo-5,8-dihydropyrido[2,3-*d*]pyrimidine-7-carboxylates from 5-acetyl-4-aminopyrimidines

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A method for the synthesis of methyl and ethyl 2-R^1-4-R^2-5-oxo-5,8-dihydropyrido[2,3-*d*]pyrimidine-7-carboxylates was proposed. The method is based on condensation of 2-R^1-6-R^2-5-acetyl-4-aminopyrimidines with ethyl oxalate in the presence of MeONa or EtONa. Products of the Friedländer self-condensation of the starting pyrimidines were also obtained.

Key words: 5-acetyl-4-aminopyrimidines, ethyl oxalate, condensation, the Friedländer self-condensation, alkyl 5-oxopyrido[2,3-*d*]pyrimidine-7-carboxylates, 7-pyrimidinylpyrido[2,3-*d*]pyrimidines.

Pyrido[2,3-*d*]pyrimidines exhibit a variety of biological activity, which makes them subjects of intensive investigation. The methods for the synthesis of these compounds are widely covered in the literature (see the reviews[1,2] and some of the recent communications[3—7]). Derivatives of pyrido[2,3-*d*]pyrimidine-6-carboxylic acid are still attracting particular attention;[8—10] earlier,[2] such well known antibacterial preparations as piromidic and pipemidic acids were discovered among them.

At the same time, derivatives of isomeric pyrido[2,3-*d*]pyrimidine-7-carboxylic acid remain almost uninvestigated. Thus, methyl esters of substituted 2,4,5-trioxopyrido[2,3-*d*]pyrimidine-7-carboxylic acids have been isolated only as by-products in the reaction of 6-R-amino-1,3-dimethyluracils with dimethyl acetylenedicarboxylate.[11] Ethyl 2-amino-4-oxo- and 4-oxo-5-phenyl-3,4-dihydropyrido[2,3-*d*]pyrimidine-7-carboxylates have been synthesized very recently[12] by the reactions of 2,6-diamino-6-hydroxypyrimidine with the corresponding ethyl 4-R-2-oxobut-3-ynoates (R = Me_3Si and Ph).

Earlier,[13—15] we demonstrated that substituted 5-acetyl-4-aminopyrimidines, which are conveniently synthesized from acetylacetone or benzoylacetone, can be used as efficient block reagents for construction of a pyrido[2,3-*d*]pyrimidine system. For instance, their condensation with amide acetals easily affords the corresponding pyrimidinylamidines, which then undergo cyclization under the action of MeONa into substituted 8*H*-pyrido[2,3-*d*]pyrimidin-5-ones.[14,15] Under analogous conditions, the latter can be obtained by intramolecular cyclization of 4-benzoylamino-5-acetylpyrimidines.[14]

In continuation of these studies, 2-R^1-6-R^2-5-acetyl-4-aminopyrimidines (**1a—g**) were used as starting reagents for the synthesis of alkyl esters of 2,4-disubstituted 5-oxo-5,8-dihydropyrido[2,3-*d*]pyrimidine-7-carboxylic acids (**2a,b**; **3a—g**) (Scheme 1). Recently,[16] ethyl 7-aryl-(1*H*,5*H*)-4,5-dioxopyrano[4,3-*b*]pyridine-2-carboxylates were obtained by heating 3-acetyl-4-amino-6-arylpyran-2-ones (like pyrimidines **1**, they contain vicinal Ac and NH_2 groups) with ethyl oxalate in EtOH in the presence of EtONa. It turned out that a similar approach makes it possible to obtain ethyl esters **2a,b** from pyrimidines **1a,b** even at room temperature. The reactions of compounds **1a—g** with ethyl oxalate in MeOH in the presence of MeONa afforded the corresponding methyl esters **3a—g***. However, refluxing was required for pyrimidines **1e—g** because of their poor solubility in MeOH.

Esters **2a,b** and **3a—g** are white crystalline substances. They are well soluble in $CHCl_3$ and DMSO, moderately soluble in acetone, and insoluble in water. The exceptions are compounds **3e—g**, which are poorly soluble in all solvents, including DMSO. The structures of compounds **2** and **3** were confirmed by spectroscopic data. Their mass spectra contain intense molecular ion peaks (except for ester **3c** with the most intense $[M - H]^+$ ion peak).

The IR spectra of esters **2** and **3** show a narrow ν(NH) band at 3376—3384 (in $CHCl_3$) or 3296—3344 cm^{-1} (KBr) and ν(CO) bands at 1720—1736 and 1640—1656 cm^{-1} (Tables 1, 2). The ^1H NMR spectra exhibit singlets at δ 6.70—7.07 for the H(6) atom and at δ 2.95—3.13 for the methyl group that is *peri* to the CO group, as well as signals for the COOMe or COOEt protons.

* No traces of ethyl esters were detected (^1H NMR data). Both ethyl oxalate and ethyl esters **2** were experimentally found to undergo transesterification under the action of MeONa in MeOH.

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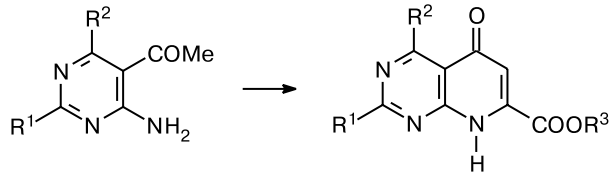
Scheme 1



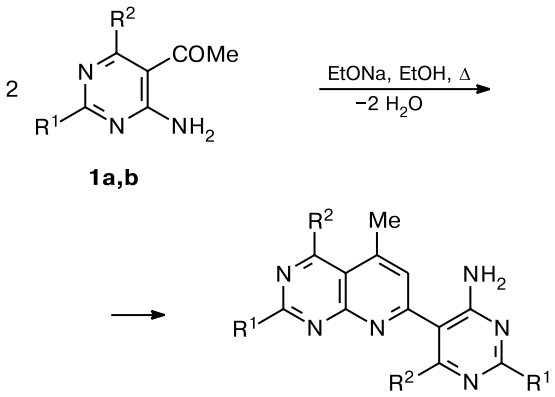
1a—g **2a,b, 3a—g**

	R^1	R^2		R^1	R^2
a	Me	Me	**e**	Ph	SMe
b	Ph	Me	**f**	4-ClC_6H_4	Me
c	Me	Ph	**g**	4-$NO_2C_6H_4$	Me
d	CCl_3	Me			

R^3 = Et (**2**), Me (**3**)

Reagents and conditions: 1) $(EtOOC)_2$, R^3ONa, R^3OH, 20—65 °C; 2) AcOH, 20 °C.

Scheme 2



1a,b **4a,b**

$R^1 = R^2$ = Me (**a**); R^1 = Ph, R^2 = Me (**b**)

It is essential that a competitive Friedländer self-condensation of pyrimidines **1** is prevented under the chosen temperature conditions. Thus the corresponding 7-(4-aminopyrimidin-5-yl)pyrido[2,3-*d*]pyrimidines **4a,b** were obtained in 76 and 85% yields, respectively, by refluxing compounds **1a,b** with EtONa in EtOH in the absence of ethyl oxalate (Scheme 2). In a MeONa—MeOH system, the starting reagents were recovered unchanged.

Compounds **4a,b** are insoluble in organic solvents, though pyridopyrimidine **4b** is rather well soluble in DMSO. Their mass spectra show intense $[M]^{+\bullet}$, $[M - H]^+$, and $[M - Me]^+$ ion peaks. The ^1H NMR spectra of these compounds in DMSO-d_6 contain singlets at δ 7.5—7.6 for the H(6) atoms and at δ 6.8—7.0 for the NH_2 group. Biheterocycles **4a,b** are of interest by themselves as potential chelating ligands. In addition, the NH_2 group allows them to be easily modified.

The methods for the synthesis of pyrido[2,3-*d*]pyrimidines **2**—**4** are simple and involve accessible starting

Table 1. Yields, melting points, elemental analysis data, and mass spectra of compounds **2a,b** and **3a—g**

Compound	Yield (%)	M.p. /°C	Found / Calculated (%) C	H	N	Molecular formula	MS, *m/z* (I_{rel} (%))
2a	57	184—185	58.36 / 58.29	5.39 / 5.30	17.08 / 17.00	$C_{12}H_{13}N_3O_3$	247 $[M]^+$ (100), 219 $[M - CO]^+$ (40), 175 $[M - C_2H_4 - CO_2]^+$ (30), 173 $[M - EtOH - CO]^+$ (75), 147 $[M - C_2H_4 - CO_2 - CO]$ (28), 132 $[M - EtOH - CO - MeCN]^+$ (55), 68 (55)
2b	56	186—187	65.84 / 66.01	4.83 / 4.89	13.64 / 13.59	$C_{17}H_{15}N_3O_3$	309 $[M]^+$ (100), 281 $[M - CO]^+$ (13), 237 $[M - C_2H_4 - CO_2]^+$ (9), 209 $[M - C_2H_4 - CO_2 - CO]^+$ (8), 104 $[PhC{=}NH]^+$ (26)
3a	63	244—245	56.44 / 56.65	4.63 / 4.75	18.12 / 18.02	$C_{11}H_{11}N_3O_3$	233 $[M]^+$ (100), 175 $[M - CH_2 - CO_2]^+$ (15), 173 $[M - MeOH - CO]^+$ (48), 132 $[M - MeOH - CO - MeCN]^+$ (43), 42 (61)
3b	74	249—250	65.11 / 65.08	4.55 / 4.44	14.05 / 14.23	$C_{16}H_{13}N_3O_3$	295 $[M]^+$ (100), 237 $[M - CH_2 - CO_2]^+$ (20), 235 $[M - MeOH - CO]^+$ (26), 104 $[PhC{=}NH]^+$ (100)
3c	50	254—255	64.84 / 65.08	4.51 / 4.44	14.16 / 14.23	$C_{16}H_{13}N_3O_3$	295 $[M]^+$ (17), 294 $[M - H]^+$ (59), 235 $[M - MeOH - CO]^+$ (14), 234 $[M - H - MeOH - CO]^+$ (27), 43 (100)
3d	74	203—204	39.51 / 39.25	2.52 / 2.40	12.66 / 12.49	$C_{11}H_8Cl_3N_3O_3$	335 $[M]^+$ (100), 300 $[M - Cl]^+$ (37), 275 $[M - MeOH - CO]^+$ (8), 240 $[M - Cl - MeOH - CO]^+$ (34), 68 (79)
3e	77	304—305	59.26 / 58.70	4.41 / 4.00	12.79 / 12.84	$C_{16}H_{13}N_3O_3S$	327 $[M]^+$ (96), 294 $[M - SH]^+$ (100), 267 $[M - MeOH - CO]^+$ (39), 234 $[M - SH - MeOH - CO]^+$ (49), 104 $[PhC{=}NH]^+$ (59)
3f	85	279—280	58.10 / 58.28	3.93 / 3.67	13.16 / 12.47	$C_{16}H_{12}ClN_3O_3$	329 $[M]^+$ (100), 269 $[M - MeOH - CO]^+$ (20), 138 $[ClC_6H_4{=}NH]^+$ (75)
3g	79	267—268	55.79 / 56.47	3.47 / 3.55	16.45 / 16.47	$C_{16}H_{12}N_4O_5$	340 $[M]^+$ (100), 310 $[M - NO]^+$ (9), 280 $[M - MeOH - CO]^+$ (21), 234 $[M - C_6H_4NO]^+$ (12), 149 $[NO_2C_6H_4{=}NH]^+$ (35)

Table 2. IR and ^{1}H NMR spectra of compounds **2a,b** and **3a—g**

Compound	IR		^{1}H NMR	
	Conditions	ν/cm^{-1}	Solvent	δ, *J*/Hz
2a	$CHCl_3$	3384 (NH), 1728 (COOEt), 1644 (CO), 1584, 1552, 1520	$CDCl_3$	1.43 (t, 3 H, $\underline{CH_3}CH_2$, *J* = 6.8); 2.70, 3.02 (both s, 3 H each, Me); 4.48 (q, 2 H, CH_2, *J* = 6.8); 6.97 (s, 1 H, H(6)); 9.25 (br.s, 1 H, NH)
2b	$CHCl_3$	3384 (NH), 1728 (COOEt), 1644 (CO), 1584, 1528	$CDCl_3$	1.45 (t, 3 H, $\underline{CH_3}CH_2$, *J* = 6.8); 3.12 (s, 3 H, Me); 4.50 (q, 2 H, CH_2, *J* = 6.8); 6.99 (s, 1 H, H(6)); 7.45—7.60 (m, 3 H, Ph); 8.50—8.60 (m, 2 H, Ph); 9.23 (br.s, 1 H, NH)
3a	$CHCl_3$	3384 (NH), 1736 (COOMe), 1644 (CO), 1584, 1552, 1520	$CDCl_3$	2.69, 3.02 (both s, 3 H each, Me); 4.02 (s, 3 H, COOMe); 6.95 (s, 1 H, H(6)); 9.19 (br.s, 1 H, NH)
3b	$CHCl_3$	3384 (NH), 1736 (COOMe), 1644 (CO), 1576, 1536, 1520	$CDCl_3$	3.13 (s, 3 H, Me); 4.05 (s, 3 H, COOMe); 6.98 (s, 1 H, H(6)); 7.45—7.60 (m, 3 H, Ph); 8.48—8.60 (m, 2 H, Ph); 9.21 (br.s, 1 H, NH)
3c	$CHCl_3$	3384 (NH), 1736 (COOMe), 1644 (CO), 1576, 1540, 1512	$CDCl_3$	2.80 (s, 3 H, Me); 4.05 (s, 3 H, COOMe); 6.93 (s, 1 H, H(6)); 7.40—7.58 (m, 3 H, Ph); 7.58—7.68 (m, 2 H, Ph); 9.30 (br.s, 1 H, NH)
3d	$CHCl_3$	3376 (NH), 1736 (COOMe), 1648 (CO), 1580, 1548, 1520	$CDCl_3$	3.13 (s, 3 H, Me); 4.09 (s, 3 H, COOMe); 7.07 (s, 1 H, H(6)); 9.42 (br.s, 1 H, NH)
3e	KBr	3296 (NH), 1728 (COOMe), 1640 (CO), 1596, 1572, 1520	DMSO-d_6	2.61 (s, 3 H, SMe); 3.92 (s, 3 H, COOMe); 6.71 (s, 1 H, H(6)); 7.48—7.68 (m, 3 H, Ph); 8.45—8.60 (m, 2 H, Ph); 12.32 (br.s, 1 H, NH)
3f	KBr	3296 (NH), 1720 (COOMe), 1648 (CO), 1576, 1536, 1512	DMSO-d_6	2.95 (s, 3 H, Me); 3.94 (s, 3 H, COOMe); 6.70 (s, 1 H, H(6)); 7.61 and 8.46 (both d, 2 H each, Ph, *J* = 7.5); 12.30 (br.s, 1 H, NH)
3g	KBr	3344 (NH), 1728 (COOMe), 1656 (CO), 1608, 1576, 1544, 1512	DMSO-d_6	2.98 (s, 3 H, Me); 3.96 (s, 3 H, COOMe); 6.71 (s, 1 H, H(6)); 8.38 and 8.68 (both d, 2 H each, Ph, *J* = 8.0)

reagents. The obtained results confirmed that 5-acetyl-4-aminopyrimidines are effective for use in construction of fused heterocyclic systems.

Experimental

^{1}H NMR spectra were recorded on a Brucker WM-250 instrument. IR spectra were recorded on a Specord M-80 instrument. Mass spectra were obtained with a Kratos MS-30 instrument (EI, 70 eV, ionization chamber temperature 250 °C, direct inlet of samples).

Substituted 5-acetyl-4-aminopyrimidines **1a—c,e** were prepared according to the known procedures.[13—15]

Synthesis of 5-acetyl-4-amino-6-methylpyrimidines (1d,f,g) (general procedure). A mixture of a corresponding *N*-cyanoamidine (5.0 mmol) and $Ni(OAc)_2$ (5.0 mmol) in 12 mL of acetylacetone was stirred at 130—140 °C for 5 h. The solvent was evaporated *in vacuo*, and the residue was chromatographed on SiO_2 in C_6H_6 (for **1d**) or $CHCl_3$ (for **1f,g**). The solvent was removed *in vacuo*, and the residue was recrystallized from an appropriate solvent.

5-Acetyl-4-amino-6-methyl-2-trichloromethylpyrimidine (1d). Yield 78%, m.p. 133—134 °C (from hexane). Found (%): C, 36.29; H, 3.10; Cl, 39.88; N, 15.87. $C_8H_8Cl_3N_3O$. Calculated (%): C, 35.75; H, 2.98; Cl, 39.66; N, 15.64. IR ($CHCl_3$), ν/cm^{-1}: 3495 and 3360 (NH_2), 1660 (CO), 1598, 1535. ^{1}H NMR ($CDCl_3$), δ: 2.63, 2.73 (both s, 3 H each, Me), 6.91 (br.s, 2 H, NH_2).

5-Acetyl-4-amino-2-(4-chlorophenyl)-6-methylpyrimidine (1f). Yield 51%, m.p. 180—181 °C (from benzene). Found (%): C, 59.64; H, 4.73; N, 16.17. $C_{13}H_{12}ClN_3O$. Calculated (%): C, 59.66; H, 4.62; N, 16.06. IR ($CHCl_3$), ν/cm^{-1}: 3504 and 3360 (NH_2), 1648 (CO), 1584, 1544. ^{1}H NMR (DMSO-d_6), δ: 2.52, 2.58 (both s, 3 H each, Me); 7.54 (br.s, 2 H, NH_2); 7.61 and 8.42 (both d, 2 H each, Ph, *J* = 7.5 Hz).

5-Acetyl-4-amino-6-methyl-2-(4-nitrophenyl)pyrimidine (1g). Yield 66%, m.p. 175—176 °C (from benzene). Found (%): C, 57.50; H, 4.61; N, 21.12. $C_{13}H_{12}N_4O_3$. Calculated (%): C, 57.35; H, 4.44; N, 20.58. IR ($CHCl_3$), ν/cm^{-1}: 3504 and 3352 (NH_2), 1652 (CO), 1588, 1540. ^{1}H NMR ($CDCl_3$), δ: 2.64, 2.78 (both s, 3 H each, Me); 6.85 (br.s, 2 H, NH_2); 8.30 and 8.60 (both d, 2 H each, Ph, *J* = 8.0 Hz).

Ethyl 5-oxo-5,8-dihydropyrido[2,3-*d*]pyrimidine-7-carboxylates (2a,b). Ethyl oxalate (6.0 mmol) and EtONa (7.2 mmol) in 5 mL of EtOH were added to pyrimidine **1a** or **1b** (2.0 mmol) in 6 mL of anhydrous EtOH. The homogeneous reaction mixture was stirred at 20 °C for 2 h (in the case of compound **2b**, a precipitate of its sodium salt was formed), acidified with AcOH to pH ~6.0, and stirred at ~20 °C for an additional 15 min. The precipitate that formed was filtered off, washed with water, and dried to give compounds **2a,b** (their yields, melting points, and spectroscopic data are given in Tables 1 and 2).

Methyl 5-oxo-5,8-dihydropyrido[2,3-*d*]pyrimidine-7-carboxylates (3a—d). Esters **3a—d** were obtained analogously from pyrimidines **1a—d**, ethyl oxalate, and MeONa in MeOH. In the case of pyrimidines **1c,d**, the filtrate was concentrated *in vacuo*, and water (10 mL) was added. The product was extracted with chloroform (2×20 mL), and the organic layer was concentrated. The residue was recrystallized from MeCN to give additional amounts of esters **3c,d**. Their yields, melting points, and spectroscopic data are given in Tables 1 and 2.

Methyl 5-oxo-5,8-dihydropyrido[2,3-*d*]pyrimidine-7-carboxylates (3e—g). Ethyl oxalate (12.0 mmol) and MeONa (14.4 mmol) in 10 mL of MeOH were added to a pyrimidine (**1e—g**) (2.0 mmol) in 8 mL of anhydrous MeOH. The reaction mixture was refluxed for 30 min and then stirred at ~20 °C for 12 h. The resulting heterogeneous mixture was acidified with AcOH and stirred at ~20 °C for 30 min. The precipitate that formed was filtered off, washed with water, dried, and refluxed with MeCN (10 mL) to eliminate the unreacted pyrimidines **1e—g**. After the mixture was cooled to ~20 °C, the precipitate was filtered off to give esters **3e—g** (their yields, melting points, and spectroscopic data are given in Tables 1 and 2).

Conversion of ethyl ester 2b into methyl ester 3b. A mixture of ethyl ester **2b** (0.155 g, 0.5 mmol) and MeONa (1.5 mmol) in 6 mL of MeOH was stirred at ~20 °C for 2 h and acidified with AcOH. The precipitate that formed was filtered off and washed with MeOH (3 mL) to give ester **3b** (0.11 g, 77%). The product has the same melting point and ^{1}H NMR spectrum as compound **3b** synthesized from pyrimidine **1b** and ethyl oxalate.

7-(4-Amino-2,6-dimethylpyrimidin-5-yl)-2,4,5-trimethylpyrido[2,3-*d*]pyrimidine (4a). A mixture of pyrimidine **1a** (0.413 g, 2.5 mmol) and EtONa (2.5 mmol) in 8 mL of EtOH was refluxed 6 h and then cooled to ~20 °C. The precipitate that formed was filtered off and washed with EtOH (5 mL) to give compound **4a** (0.279 g, 76%), m.p. 327—328 °C (decomp.). Found (%): C, 64.92; H, 6.47; N, 29.08. $C_{16}H_{18}N_6$. Calculated (%): C, 65.28; H, 6.16; N, 28.55. MS, *m/z* (I_{rel} (%)): 294 $[M]^+$ (63), 293 $[M - H]^+$ (100), 279 $[M - Me]^+$ (98). IR (KBr), ν/cm^{-1}: 3352 and 3304 (NH_2), 1592, 1556. ^{1}H NMR (DMSO-d_6), δ: 2.15, 2.37, 2.70, 2.91, 3.04 (all s, 3 H each, Me); 6.76 (br.s, 2 H, NH_2); 7.52 (s, 1 H, H(6)).

7-(4-Amino-6-methyl-2-phenylpyrimidin-5-yl)-4,5-dimethyl-2-phenylpyrido[2,3-*d*]pyrimidine (4b) was synthesized analogously from pyrimidine **1b**. The yield of compound **4b** was 85%, m.p. 247—248 °C. Found (%): C, 74.38; H, 5.22; N, 20.12. $C_{26}H_{22}N_6$. Calculated (%): C, 74.62; H, 5.30; N, 20.08. MS, *m/z* (I_{rel} (%)): 418 $[M]^+$ (68), 417 $[M - H]^+$ (83), 403 $[M - Me]^+$ (98), 104 $[PhC{=}NH]^+$ (100). IR (KBr), ν/cm^{-1}: 3384 and 3300 (NH_2), 1624, 1592, 1540. ^{1}H NMR (DMSO-d_6), δ: 2.32, 2.92, 3.17 (all s, 3 H each, Me); 6.99 (br.s, 2 H, NH_2); 7.45—7.55, 7.55—7.65 (both m, 3 H each, Ph); 7.61 (s, 1 H, H(6)); 8.32—8.42, 8.52—8.65 (both m, 2 H each, Ph).

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