SYNTHESIS AND ANTIVIRAL ACTIVITY OF ACYCLIC NUCLEOSIDE ANALOGUES OF 6-METHYLURACIL AND 4-ALKYLAMINO-6-METHYL-2(1*H*)-PYRIMIDINONES

Sunita BHAT*

Department of Medicinal Chemistry,
Central Drug Research Institute, Lucknow-226001, India

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Reaction of 2,4-Dimethoxy-6-methylpyrimidine (*I*) with allyl bromide or benzyl bromide afforded 1-substituted 4-methoxy-6-methyl-2(1*H*)-pyrimidinone intermediates *III*, *X*. Oxidation of the compound *III* afforded racemic *cis* diol *VI*. O-Demethylation and nucleophilic displacement of the intermediates *III*, *VI* and *X* gave 1-substituted 6-methyluracils *IV*, *VII*, *IX* and 1-substituted 4-alkylamino-6-methyl-2(1*H*)-pyrimidinones *V*, *VIII*, *XII* in good yields. The compounds *II* – *XII* were evaluated against Ranikhet disease virus (RDV); compounds *Vb*, *VII*, *X*, *XIIb* – *XIId* showed 100, 43, 44, 75, 72 and 100% inhibition, respectively.

The chemistry and biochemistry of pyrimidine nucleosides are of considerable importance not only because of their relevance to nucleic acid chemistry and molecular biology, but also because of the fact that several of the pyrimidine nucleosides have been found to exhibit anticancer and antiviral activities. Considerable attention has been paid to the synthesis of 5-substituted uracil analogue as inhibitors of thymidylate synthetase and uridine phosphorylase[1,2]. 6-Methyluridine and 6-methylcytidine when tested in vitro against[3] Herpes simplex virus in African green monkey kidney cell[4] (BSC_1) were found to exhibit modest activity. As part of our programme of evaluating acyclic nucleosides of pyrimidine[5], in this communication we report the synthesis and antiviral activity of 1-(2,3-dihydroxypropyl)-6-methyluracil (*VII*), 1-benzyl-6-methyluracil[6] (*XI*), 4-alkylamino-1-(2,3-dihydroxypropyl)-6-methyl-2(1*H*)-pyrimidinone (*VIIIb* – *VIIId*) and 4-alkylamino-1-benzyl-6-methyl-2(1*H*)-pyrimidinone (*XIIa* – *XIId*) and their intermediates.

2,4-Dimethoxy-6-methylpyrimidine[7] (*I*), the key intermediate was prepared in two steps[8] from 6-methyluracil[9]. Condensation of the 2,4-dimethoxy-6-methylpyrimidine (*I*)

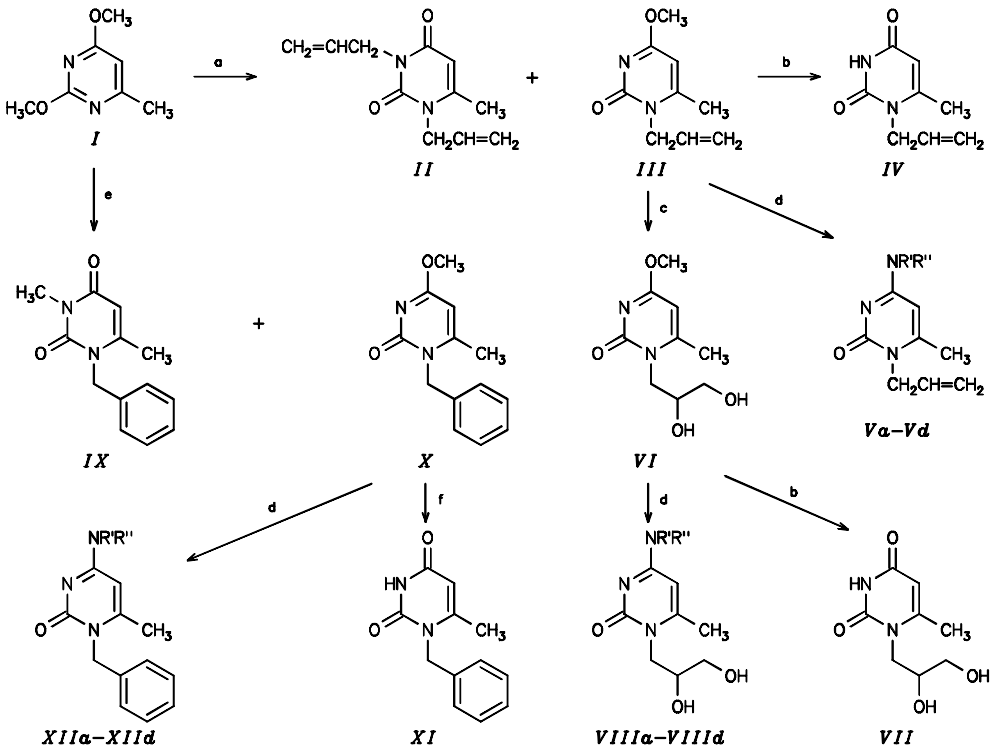
* Present address: Department of Medicinal Chemistry, College of Pharmacy, University of Michigan, Ann Arbor, Michigan 48109-1065, U.S.A.

with allyl bromide by modified Hilbert–Johnson procedure[10] gave mixture of products as monitored by TLC. The two major product obtained by chromatography on silica gel were 1,3-bisallyl-6-methyluracil (*II*) and 1-allyl-4-methoxy-5-methoxymethyl-6-methyl-2(1*H*)-pyrimidinone (*III*). In the NMR spectrum of *II*, H-1′ and H-1″ protons appeared as multiplets from δ 4.30 to 4.80, whereas a singlet at δ 3.80 in *III* was due to 4-OCH_3 protons.

Similar reaction of compound *I* with benzyl bromide gave mixture of two products which were resolved by column chromatography and identified as 1-benzyl-3,6-dimethyl-2(1*H*)-pyrimidinone (*IX*) and 1-benzyl-4-methoxy-6-methyl-2(1*H*)-pyrimidinone (*X*), respectively (see Scheme 1). In the NMR spectrum of *IX*, a singlet at δ 3.25 was due to 3-CH_3, whereas in *X* the singlet at δ 3.86 was due to the 4-OCH_3 protons. Since the reaction was carried out at high temperature, it is likely that the molecule of *X* underwent Dimroth rearrangement with the methyl group migration from 4-OCH_3 position to the N-3 position to give *IX*.

Hydroxylation of the compound *III* with sodium chlorate and osmium tetroxide[11] in aqueous methanol afforded racemic diol, 1-(2,3-dihydroxypropyl)-4-methoxy-6-methyl-2(1*H*)-pyrimidinone (*VI*). Demethylation of *III*, *VI* and *X* with aqueous sodium hydroxide or HCl gas gave uracil derivatives[6] *IV*, *VII* and *XI*. The site of glycosylation was confirmed to be N-1 by direct comparison of the UV spectrum of *VI* (λ_{max} (MeOH) 264, 210 nm; (MeOH + NaOH) 264 nm) with the UV spectrum of the 6-methyluridine (λ_{max} (pH 1 – 4) 268 nm; λ_{max} (pH 14) 271 nm)[12] which is turn resembles that of the N-1 methyluracil. The N-1 and N-3 methyluracils are reported in the methylation of the 2,4-dimethoxypyrimidine[13]. The N-3 methyluracil showed bathochromic shift from λ_{max} 261 nm at pH 2 to 280 nm at pH 14, whereas N-1 isomer showed no shift up to pH 14 (λ_{max} (pH 5.4 – 7.2) 267, 207; λ_{max} (pH 14) 265 nm)[13]. However, N-1,3-disubstituted uracil can be identified by its NMR spectra which showed double integral signals for glycone protons. Pressure amination of *III* and *X* with methanolic ammonia afforded corresponding cytosine analogues *Va* and *XIIa*, respectively. Amination of *VI* with methanolic ammonia under various conditions did not give the desired cytosine. Compounds were also aminated with aqueous methylamine, ethylamine and dimethylamine solution. Similarly 1-allyl-4-alkylamino-6-methyl-2(1*H*)-pyrimidinones *Vb – Vd*, 4-alkylamino-1-(2,3-dihydroxypropyl)-6-methyl-2(1*H*)-pyrimidinones *VIIIb – VIIId* and 4-alkylamino-1-benzyl-6-methyl-2(1*H*)-pyrimidinones *XIIb – XIId* were synthesized in good yields from *III*, *VI* and *X* using methylamine, ethylamine and dimethylamine to determine if alkyl substitution on the 4-amino group would have any effect on the antiviral activity.

The compounds *II – XII* were evaluated for antiviral activity against Ranikhet disease virus (RDV) (0.064 HA/ml) in chorio-allantoic membrane (CAM) cultures at the dose of 0.1 μg/culture by the method described earlier[14]. The compounds *Vb*, *VII*, *X*, *XIIb*, *XIIc* and *XIId* showed 100, 43, 44, 75, 72 and 100% inhibition, respectively. The



a) CH_2Cl_2, Na_2CO_3, $CH_2{=}CHCH_2Br$; b) aq. NaOH; c) aq. MeOH, $NaClO_3$, OsO_4;
d) NHR'R''; e) CH_2Cl_2, Na_2CO_3, $C_6H_5CH_2Br$; f) HCl, $CHCl_3$

In formulae *V*, *VIII*, *XII* : ***a***, R' = R'' = H; ***b***, R' = H, R'' = CH_3;
c, R' = H, R'' = C_2H_5; ***d***, R' = R'' = CH_3

SCHEME 1

remaining compounds were found to exhibit either low degree of activity or were inactive.

EXPERIMENTAL

Melting points were taken in IEC-45 instrument and are uncorrected. Compounds were checked for their homogeneity by TLC on silica gel or GF-254 plates and their spots were located under UV lamp, by iodine vapours or by spraying with Dragendorff's reagent. UV absorption spectra (λ_{max}, nm) were recorded on Perkin–Elmer Lambda-15 and Hitachi-320 model. IR spectra ($\tilde{\nu}_{max}$, cm^{-1}) were recorded on Perkin–Elmer 157 or Acculab 1 model. ^1H NMR spectra (δ, ppm) were recorded on Perkin–Elmer R-32 or EM-360L instruments using TMS as internal reference. Mass spectra were run on Jeol JMS-D 300 using direct inlet system.

1,3-Bisallyl-6-methyluracil (*II*) and 1-Allyl-4-methoxy-6-methyl-2(1*H*)-pyrimidone (*III*)

A mixture of *I* (4.0 g, 26.0 mmol) in CH_3CN (100 ml), anhydrous Na_2CO_3 (3.3 g, 31.2 mmol) and allyl bromide (2.7 ml, 31.2 mmol) were refluxed at 140 °C for 36 h. The resulting mixture was cooled, filtered and the solid was washed with CH_3CN (100 ml). The filtrate and washings were combined and evaporated under reduced pressure to give a syrup which was chromatographed on SiO_2 column. Elution with benzene–ethyl acetate (8 : 2) gave 1.9 g (35%) of compound *II*, m.p. 57 – 58 °C (C_6H_6). IR spectrum (KBr): 1 700, 1 660 (C=O). Mass spectrum (*m*/*z*): 206 (M^+). ^1H NMR spectrum ($CDCl_3$): 2.14 s, 3 H (6-CH_3); 4.30 – 4.80 m, 4 H (H-1′ and H-1″); 5.00 – 5.20 m, 4 H (H-3′ and H-3″); 5.40 s, 1 H (H-5); 5.54 – 6.00 m, 2 H (H-2′ and H-2″). For $C_{11}H_{14}N_2O_2$ (206.2) calculated: 64.05% C, 6.84% H, 13.58% N; found: 64.28% C, 6.96% H, 18.41% N.

1-Allyl-4-methoxy-6-methyl-2(1H)-pyrimidone (III) was obtained by continued elution of the column with benzene–ethyl acetate (7 : 3) as an oil (1.9 g, 41%). Mass spectrum (*m*/*z*): 180 (M^+). ^1H NMR spectrum ($CDCl_3$): 2.23 s, 3 H (6-CH_3); 3.80 s, 3 H (4-OCH_3); 4.42 – 4.57 m, 2 H (H-1′); 5.00 – 5.70 m, 2 H (H-3′); 5.65 s, 1 H (H-5); 5.70 – 6.00 m, 1 H (H-2′). For $C_9H_{12}N_2O_2$ (180.2) calculated: 59.98% C, 6.71% H, 15.54% N; found: 60.25% C, 6.83% H, 15.70% N.

1-Allyl-6-methyluracil (*IV*)

A mixture of *III* (0.5 g, 2.8 mmol) and 2 M aqueous NaOH (20 ml) was stirred at 50 °C for 15 h. The resulting mixture was cooled, neutralized with AcOH and concentrated under reduced pressure. The product was dissolved in water (100 ml), extracted with EtOAc (250 ml), the organic layer was dried over Na_2SO_4 and concentrated in vaccuo. The residue was chromatographed on SiO_2 column, elution of the column with chloroform–methanol (9 : 1) gave 0.24 g (52%) of methyl uracil *IV* as amorphous solid, m.p. 168 °C (EtOAc). IR spectrum (KBr): 1 720, 1 660 (C=O). Mass spectrum (*m*/*z*): 166 (M^+). ^1H NMR spectrum ($CDCl_3$ + $(CD_3)_2SO$): 2.16 s, 3 H (6-CH_3); 4.35 – 4.54 m, 2 H (H-1′); 5.05 – 5.27 m, 2 H (H-3′); 5.55 s, 1 H (H-5); 5.62 – 6.04 m, 1 H (H-2′). For $C_8H_{10}N_2O_2$ (166.2) calculated: 57.81% C, 6.06% H, 16.86% N; found: 57.60% C, 6.28% H, 16.75% N.

1-Allyl-6-methylcytosine (*Va*)

A mixture of *III* (0.5 g, 2.8 mmol) and a solution of NH_3 in methanol (20 ml) was heated in a steel bomb at 120 °C for 12 h. The excess of MeOH and NH_3 was removed under reduced pressure. The product was chromatographed over SiO_2 column. Elution of the column with chloroform–methanol (8 : 2) gave 0.41 g (90%) of cytosine *Va* as amorphous solid, m.p. 220 – 222 °C (dec. MeOH–H_2O). IR spectrum (KBr): 3 300, 3 100 (NH_2), 1 660 (C=O). Mass spectrum (*m*/*z*): 165 (M^+). ^1H NMR

spectrum (TFA): 2.32 s, 3 H (6-CH_3); 4.40 – 4.54 m, 2 H (H-1′); 5.07 – 5.30 m, 2 H (H-3′); 5.45 – 5.90 m, 1 H (H-2′); 6.00 s, 1 H (H-5); 7.50 bs, 2 H (D_2O exchangeable, NH_2). For $C_8H_{11}N_3O$ (165.2) calculated: 58.16% C, 6.71% H, 25.43% N; found: 58.36% C, 6.80% H, 25.75% N.

1-Allyl-4-methylamino-6-methyl-2(1*H*)-pyrimidinone (*Vb*)

A mixture of *III* (0.3 g, 1.7 mmol) and aqueous CH_3NH_2 (20 ml) was heated at 80 °C for 6 h. The reaction mixture was cooled and excess of reagent was removed under reduced pressure and coevaporated with 50 ml of EtOH. The residue was crystallized from diethyl ether–hexane, yield 0.24 g (79%), m.p. 142 °C. IR spectrum (KBr): 3 300 (NH), 1 620 (C=O). Mass spectrum (*m*/*z*): 179 (M^+). ^{1}H NMR spectrum ($CDCl_3$): 2.10 s, 3 H (6-CH_3); 2.81 bs, 3 H ($NHCH_3$); 4.30 – 4.48 m, 2 H (H-1′); 4.92 – 5.14 m, 2 H (H-3′); 5.54 s, 1 H (H-5); 5.60 – 6.00 m, 2 H (H-2′). For $C_9H_{13}N_3O$ (179.2) calculated: 60.31% C, 7.30% H, 23.47% N; found: 60.42% C, 7.30% H, 23.86% N.

1-Allyl-4-ethylamino-6-methyl-2(1H)-pyrimidinone (Vc) *and 1-allyl-4-dimethylamino-6-methyl-2(1H)-pyrimidinone* (Vd) were synthesized from compound *III*, ethylamine and dimethylamine solutions by the same procedure. The characteristic data of compounds are presented in Table I.

1-(2,3-Dihydroxypropyl)-4-methoxy-6-methyl-2(1*H*)-pyrimidinone (*VI*)

To a stirred solution of *III* (4.5 g, 25.0 mmol) and $NaClO_3$ (3.24 g, 30.6 mmol) in 50% aqueous MeOH (200 ml) was added OsO_4 (0.072 g, 0.28 mmol) with stirring. The stirring was continued for 24 h, the resulting mixture was filtered through a Celite pad and the solid was washed with H_2O (100 ml). The filtrate and washings were combined and solvent removed under reduced pressure. The crude product was chromatographed on SiO_2 column. Elution of the column with chloroform–methanol (85 : 15) and evaporation of the appropriate fraction furnished 3.9 g (72%) of racemic diol *V*, m.p. 120 °C. IR spectrum (KBr): 3 400 (OH), 1 640 (C=O). Mass spectrum (*m*/*z*): 214 (M^+). ^{1}H NMR spectrum ($CDCl_3$ + $(CD_3)_2SO$): 2.36 s, 3 H (6-CH_3); 3.45 d, J = 3 Hz, 2 H (H-1′); 3.80 s, 3 H (4-OCH_3); 3.84 – 4.00 m, 3 H (H-2′ and H-3′); 5.71 s, 1 H (H-5). For $C_9H_{14}N_2O_4$ (214.2) calculated: 50.45% C, 6.58% H, 13.08% N; found: 50.63% C, 6.50% H, 13.29% N.

1-(2,3-Dihydroxypropyl)-6-methyluracil (*VII*)

A mixture of *VI* (0.4 g, 1.9 mmol) and 2 M aqueous NaOH (30 ml) was stirred at room temperature for 24 h. The resulting mixture was worked up as described for *IV*. The crude product was chromatographed on SiO_2 column. Elution of the column with chloroform–methanol (8 : 2) gave 0.08 g (21%) of methyluracil *VII*, m.p. 148 °C (MeOH–H_2O). IR spectrum (KBr): 3 300 (OH), 1 620 (C=O). UV spectrum (MeOH, MeOH–NaOH): 264, 212; (MeOH + HCl): 266, 215. Mass spectrum (*m*/*z*): 200 (M^+). ^{1}H NMR spectrum ($CDCl_3$ + $(CD_3)_2SO$): 2.61 s, 3 H (6-CH_3); 3.82 – 4.00 m, 2 H (H-1′); 4.06 – 4.30 m, 3 H (H-2′ and H-3′); 5.97 s, 1 H (H-5). For $C_8H_{12}N_2O_4$ (200.2) calculated: 47.99% C, 6.04% H, 13.99% N; found: 48.14% C, 6.36% H, 14.26% N.

1-(2,3-Dihydroxypropyl)-4-methylamino-6-methyl-2(1*H*)-pyrimidinone (*VIIIb*)

From *VI* (0.3 g, 1.4 mmol) and aqueous CH_3NH_2 (10 ml) by the method described for *Vb* was prepared *VIIIb* in 65% yield, m.p. 177 – 179 °C. Mass spectrum (*m*/*z*): 213 (M^+). ^{1}H NMR spectrum ($CDCl_3$ + $(CD_3)_2SO$): 2.22 s, 3 H (6-CH_3); 2.75 d, 3 H (4-$NHCH_3$); 3.22 – 3.45 m, 2 H (H-1′); 3.64 – 3.92 m, 3 H (H-2′ and H-3′); 5.52 s, 1 H (H-5). For $C_9H_{15}N_3O_3$ (213.2) calculated: 50.69% C, 7.09% H, 19.70% N; found: 50.84% C, 7.15% H, 19.59% N.

1-(2,3-Dihydroxypropyl)-4-ethylamino-6-methyl-2(1H)-pyrimidinone (VIIIc) *and 1-(2,3-dihydroxypropyl)-4-dimethylamino-6-methyl-2(1H)-pyrimidinone* (VIIId) were synthesized from *VI* and ethylamine or dimethylamine solutions by the method described for *Vb*. The characteristic data are presented in Table I.

1-Benzyl-3,6-dimethyl-2(1*H*)-pyrimidinone (*IX*)

A mixture of *I* (4.0 g, 26.0 mmol) in CH_2Cl_2 (100 ml), Na_2CO_3 (3.3 g, 31.2 mmol) and benzyl bromide (5.33 g, 31.2 mmol) was heated in a steel bomb at 150 °C for 48 h. The resulting mixture was cooled, filtered and the solid was washed with CH_2Cl_2 (100 ml). The filtrate and washings were mixed and evaporated under reduced pressure to give syrup which was chromatographed on SiO_2 column. Elution with benzene–ethyl acetate (8 : 2) and evaporation of the appropriate fractions gave 0.9 g (15 %) of compound *IX*, m.p. 150 °C (C_6H_6). Mas spectrum (*m*/*z*): 230 (M^+). ^{1}H NMR spectrum ($CDCl_3$): 2.08 s, 3 H (6-CH_3); 3.25 s, 3 H (3-CH_3); 5.00 s, 2 H (CH_2); 5.54 s, 1 H (H-5); 7.10 – 7.30 m, 5 H (C_6H_5). For $C_{13}H_{14}N_2O_2$ (230.3) calculated: 67.80% C, 6.12% H, 12.16% N; found: 67.95% C, 6.30% H, 12.45% N.

1-Benzyl-4-methoxy-6-methyl-2(1H)-pyrimidinone (X) was obtained by continued elution of the column with benzene–ethyl acetate (7.5 : 2.5) as a solid (1.8 g, 30%), m.p. 72 °C (C_6H_6). Mass spectrum (*m*/*z*): 230 (M^+). ^{1}H NMR spectrum ($CDCl_3$): 2.13 s, 3 H (6-CH_3); 3.86 s, 3 H (4-OCH_3); 5.15 s, 2 H (CH_2); 5.65 s, 1 H (H-5); 7.17 m, 5 H (C_6H_5). For $C_{13}H_{14}N_2O_2$ (230.3) calculated: 67.80% C, 6.12% H, 12.16% N; found: 67.62% C, 6.28% H, 12.30% N.

1-Benzyl-6-methyluracil (*XI*)

A mixture of *IX* (1.0 g, 4.3 mmol) in $CHCl_3$ (176 ml) was saturated with HCl gas at 10 °C and the mixture was allowed to stand at room temperature for 10 h. The hydrogen chloride was removed from the resulting reaction mixture at room temperature and concentrated under reduced pressure. The product was crystallized from MeOH–Et_2O, yield 0.71 g (75%), m.p. 233 – 234 °C (ref.[34] reported m.p. 230 – 232 °C). IR spectrum (KBr): 1 720, 1 650 (C=O). Mass spectrum (*m*/*z*): 216 (M^+). ^{1}H NMR spectrum (TFA): 2.15 s, 3 H (6-CH_3); 5.02 s, 2 H (CH_2); 5.83 s, 1 H (H-5); 7.16 – 7.50 m, 5 H (C_6H_5). For $C_{12}H_{12}N_2O_2$ (216.2) calculated: 66.65% C, 5.59% H, 12.95% N; found: 66.84% C, 5.67% H, 13.16% N.

1-Benzyl-6-methylcytosine (*XIIa*)

From *X* (0.3 g, 1.3 mmol) and a solution of NH_3 in methanol (10 ml) by the method described for *Va* was prepared *XIIa* (0.15 g, 54%), m.p. >200 °C (MeOH). IR spectrum (KBr): 3 400, 3 100 (NH_2), 1 680 (C=O). Mass spectrum (*m*/*z*): 215 (M^+). ^{1}H NMR spectrum (TFA): 2.22 s, 3 H (6-CH_3); 5.05 s, 2 H (CH_2); 5.96 s, 1 H (H-5); 6.85 – 7.20 m, 5 H (C_6H_5); 7.50 bs, 2 H (D_2O exchangeable, NH_2). For $C_{12}H_{13}N_3O$ (215.2) calculated: 66.95% C, 6.08% H, 19.52% N; found: 66.23% C, 6.29% H, 19.63% N.

1-Benzyl-4-methylamino-6-methyl-2(1H)-pyrimidinone (XIIb) was prepared in 80% yield from *X* (0.2 g, 0.9 mmol) and aqueous CH_3NH_2 (5 ml) by the method described for *Vb*, m.p. 149 – 150 °C. Mass spectrum (*m*/*z*): 229 (M^+). ^{1}H NMR spectrum ($CDCl_3$ + $(CD_3)_2SO$): 2.04 s, 3 H (6-CH_3); 2.81 d, 3 H (4-$NHCH_3$); 5.08 s, 2 H (CH_2); 5.60 s, 1 H (H-5); 7.02 – 7.26 m, 5 H (C_6H_5). For $C_{13}H_{15}N_3O$ (229.3) calculated: 68.09% C, 6.59% H, 18.32% N; found: 68.23% C, 6.76% H, 18.54% N.

1-Benzyl-4-ethylamino-6-methyl-2(1H)-pyrimidinone (XIIc) *and 1-benzyl-4-dimethylamino-6-methyl-2(1H)-pyrimidinone* (XIId) were synthesized from *X* and ethylamine or dimethylamine solutions by the method described for *Vb*. The characteristic data are presented in Table I.

TABLE I
Physical and spectral data of 4-alkylaminopyrimidinones *Vc*, *Vd*, *VIIIc*, *VIIId*, *XIIc*, *XIId*

Compound	M.p., °C Yield, %	Formula (M.w.)	Calculated/Found			Spectral data
			% C	% H	% N	
Vc	71 – 73	$C_{10}H_{15}N_3O$	62.15	7.82	21.74	MS (*m/z*): 193 (M^+). ^{1}H NMR ($CDCl_3$): 1.07 t, 3 H (4-$NHCH_2CH_3$);
	56	(193.2)	62.48	7.65	22.82	3.10 – 3.35 m, 3 H ($NHCH_2$); 4.36 – 4.50 m, 2 H (H-1′)
Vd[a]	79 – 80[b]	$C_{10}H_{15}N_3O$	62.15	7.82	21.74	MS (*m/z*): 193 (M^+). ^{1}H NMR ($CDCl_3$): 3.05 s, 6 H (4-$N(CH_3)_2$);
	58	(193.2)	62.67	7.53	21.60	4.43 – 4.55 m, 2 H (H-1′)
VIIIc	129 – 130[c]	$C_{10}H_{17}N_3O_3$	52.84	7.54	18.48	MS (*m/z*): 227 (M^+). ^{1}H NMR (D_2O): 1.45 t, 3 H (4-$NHCH_2CH_3$);
	71	(227.3)	52.61	7.80	18.29	3.55 q, 3 H (4-$NHCH_2$); 3.80 – 3.96 m, 2 H (H-1′)
VIIId[a]	oil[b]	$C_{10}H_{17}N_3O_3$	52.84	7.54	18.48	MS (*m/z*): 227 (M^+). ^{1}H NMR (D_2O): 3.22 s, 6 H (4-$N(CH_3)_2$);
	89	(227.3)	53.12	7.28	18.31	3.76 – 3.90 m, 2 H (H-1′)
XIIc[a]	148 – 150[d]	$C_{14}H_{17}N_3O$	69.10	7.04	17.27	MS (*m/z*): 243 (M^+). ^{1}H NMR ($CDCl_3$ + $(CD_3)_2SO$): 1.10 t, 3 H
	68	(243.3)	68.94	7.26	17.09	(4-$NHCH_2CH_3$); 3.00 – 3.20 m, 3 H (4-$NHCH_2$); 5.08 s, 2 H (CH_2)
XIId[a]	137 – 138[b]	$C_{14}H_{17}N_3O$	69.10	7.04	17.27	MS (*m/z*): 243 (M^+). ^{1}H NMR ($CDCl_3$): 3.06 s, 6 H (4-$N(CH_3)_2$);
	25	(243.3)	69.36	7.28	17.50	5.15 s, 2 H (CH_2)

[a] Purified by column chromatography, gradient elution with $CHCl_3$–MeOH. Crystallized from: [b] ethyl acetate–hexane; [c] methanol–water; [d] methanol.

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