Chemistry of Heterocyclic Compounds, Vol. 44, No. 8, 2008

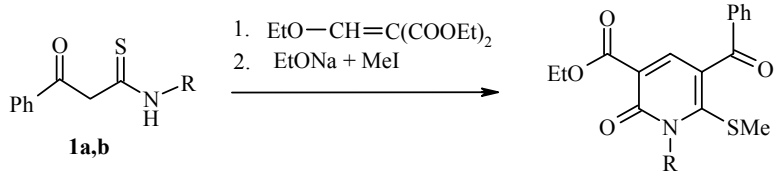
CYCLOCONDENSATION OF 5-BENZOYL-3-ETHOXYCARBONYL-6-METHYLTHIO-1-R-1,2-DIHYDROPYRID-2-ONES WITH HETEROCYCLIC N,N- AND N,C-1,3-DINUCLEOPHILES

V. N. Britsun, A. N. Esipenko, V. V. Piroghenko, and M. O. Lozinskii

[3+3] Cyclocondensation of 5-benzoyl-3-ethoxycarbonyl-6-methylthio-1-R-1,2-dihydropyrid-2-ones with heterocyclic N,N- and N,C-1,3-dinucleophiles proceeds regioselectively to give a series of new tri- and tetracyclic heterosystems, viz. derivatives of 5,6-dihydropyrazolo[1,5-a]pyrido[2,3-d]pyrimidin-6-one, 1,2-dihydropyrido[2,3-d]pyrido[2',3':3,4]pyrazolo[1,5-a]pyrimidin-2-one, 8,9-dihydro-5H-pyrido-[2,3-d]thiazolo[3,2-a]pyrimidin-8-one, 1,2-dihydrobenzo[4,5]imidazo[1,2-a]pyrido[2,3-d]pyrimidin-2-one, and 1,2-dihydrobenzo[4,5]imidazo[1,2-g][1,6]naphthyridin-2-one.

Keywords: 2-aminobenzimidazole, 3-amino-2H-pyrazolo[3,4-*b*]pyridine, 5-amino-3-R^1-4-R^2-pyrazoles, 2-amino-5-R-thiazoles, 5-benzoyl-3-ethoxycarbonyl-6-methylthio-1-R-1,2-dihydropyridin-2-ones, 7-ethoxycarbonyl-5-hydroxy-9-methyl-5-phenyl-2-R-8,9-dihydro-5H-pyrido[2,3-*d*]thiazolo[3,2-*a*]pyrimidin-8-ones, 3-ethoxycarbonyl-5-phenyl-1-R-1,2-dihydrobenzo[4,5]imidazo[1,2-*a*]pyrido[2,3-*d*]pyrimidin-2-ones, 7-ethoxycarbonyl-9-phenyl-5-R1-3-R2-2-R^3-5,6-dihydropyrazolo[1,5-*a*]pyrido[2,3-*d*]pyrimidin-6-ones, 3-ethoxycarbonyl-5-phenyl-1-R-1,2-dihydropyrido[2,3-*d*]pyrido[2',3':3,4]pyrazolo[1,5-*a*]pyrimidin-2-ones, 12-cyano-3-ethoxycarbonyl-5-phenyl-1-R-1,2-dihydrobenzo[4,5]imidazo[1,2-*g*][1,6]naphthyridin-2-ones, 2-(cyanomethyl)benzimidazole, heterocyclization, [3+3] cyclocondensation.

Derivatives of 3-aroyl-2-(methylthio)pyridine are valuable starting materials for obtaining condensed nitrogen-containing heterosystems. In spite of the significant number of studies devoted to the synthesis of these compounds [1-7] their further conversions have not been studied. This is probably explained both by the nonselectivity of the reactions obtaining them [1-3] and the difficulty of availability of the initial reactants [4-7].



1a,b **1**, **2 a** R = Me. **b** R = Et **2a,b**

Institute of Organic Chemistry, National Academy of Sciences of Ukraine, Kiev 2660; e-mail: bvn1967@rambler.ru. Translated from Khimiya Geterotsiklicheskikh Soedinenii, No. 8, pp. 1216-1222, August, 2008. Original article submitted February 5, 2008.

Recently we have developed a preparative method of synthesizing 5-benzoyl-3-ethoxycarbonyl-6-methylthio-1-R-1,2-dihydropyrid-2-ones from available starting materials and the possibility was shown of using them for cyclocondensation with nitrogen-containing 1,3-dinucleophiles, particularly with the aim of obtaining bi- and tricyclic heterosystems [8].

On continuing these investigations we improved the two-step procedure developed by us previously for the synthesis of 5-benzoyl-3-ethoxycarbonyl-6-methylthio-1-R-1,2-dihydropyridin-2-ones **2a,b**, by establishing that these reactions may be carried out as a one-stage (*one-pot*) process accomplished without isolation of the intermediate products.

In comparison with the two-stage method this approach enabled the yields of products **2a,b** to be increased from 45-52 to 65-73%.

Development of a preparative one-step method of synthesizing 1-R-1,2-dihydropyrid-2-ones **2a,b** enabled an investigation [8] of the regioselectivity of their cyclocondensations with heterocyclic N,N- and N,C-1,3-dinucleophiles to be continued. As such were used 5-amino-3-R^1-4-R^2-pyrazoles **3a,b**, 3-amino-2H-pyrazolo[3,4-*b*]pyridine (**5**), 2-amino-5-R-thiazoles **7a,b**, 2-aminobenzimidazole (**9**), and 2-(cyanomethyl)-benzimidazole (**11**).

A special feature of the reaction of 1-R-1,2-dihydropyrid-2-ones **2a,b** with N,C- and unsymmetrical N,N-1,3-dinucleophiles is the possibility of forming two isomeric products, a linear and an angular. It turned out that in all cases the interaction of substrates **2a,b** with reactants **3a,b**, **5**, **7a,b**, **9**, and **11** was effected selectively (Tables 1 and 2).

We showed previously [8] by X-ray structural analysis that the cyclocondensation of 1-R-1,2-dihydropyrid-2-ones **2a,b** with such an aminoazole as 3-amino-1,2,4-triazole (p*K*a 4.17 [9]) proceeds selectively and is effected as a nucleophilic attack of the exocyclic and endocyclic amino groups of the reactant at the methylthio and aroyl groups of the substrate respectively. On the basis of these data it may be concluded that the products of the interaction of 1-R-1,2-dihydropyridin-2-ones **2a,b** with derivatives of 3(5)-aminopyrazole (p*K*a 4.11 [9]) **3a,b**, **5** and 2-aminothiazole (p*K*a 5.39 [10]) **7a,b** have similar structures and are 7-ethoxycarbonyl-9-phenyl-5-R1-3-R2-2-R3-5,6-dihydropyrazolo[1,5-*a*]pyrido[2,3-*d*]pyrimidin-6-ones **4a-c**, 3-ethoxycarbonyl-5-phenyl-1-R-1,2-dihydropyrido[2,3-*d*]pyrido[2',3':3,4]pyrazolo[1,5-*a*]pyrimidin-2-ones **6a,b** and 7-ethoxycarbonyl-5-hydroxy-9-methyl-5-phenyl-2-R-8,9-dihydro-5H-pyrido[2,3-*d*]thiazolo[3,2-*a*]pyrimidin-8-ones **8a,b**.

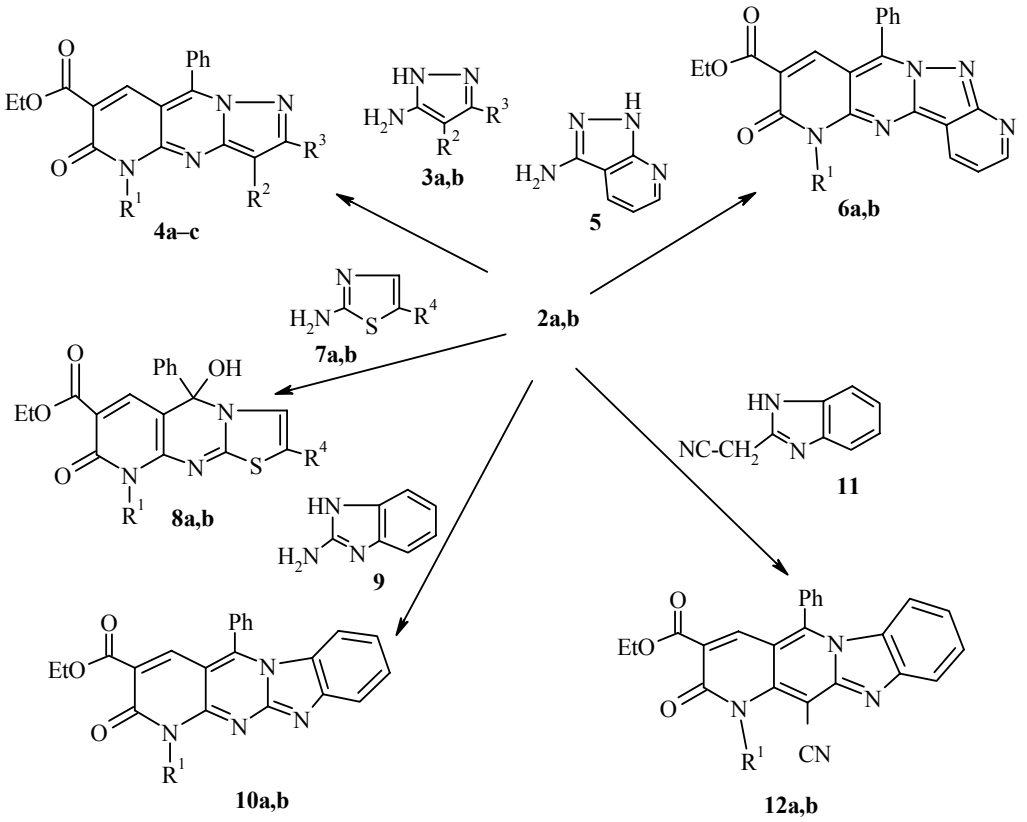
Since the products of cyclocondensation of 1,2-dihydropyridin-2-one **2a** with 2-amino-5-R-thiazoles **7a,b** may have both an acyclic and a cyclic structure, then for the unequivocal determination of their structure we recorded the ^{13}C NMR spectrum of compound **8b**.

The carbon signal of the phenylcarbonyl group (192.6-198.1 ppm [8]) was absent from the ^{13}C NMR spectrum of the reaction product of 1,2-dihydropyrid-2-one **2a** with 2-amino-5-methylthiazole **7b**, and a signal was observed for the sp^3-hybridized C–N carbon at 86.7 ppm [11], which may be assigned only to the C-5 atom of pyrido[2,3-*d*]thiazolo[3,2-*a*]pyrimidin-8-one **8b**.

It should be noted that 5,6-dihydropyrazolo[1,5-*a*]pyrido[2,3-*d*]pyrimidin-6-ones **4a-c**, 1,2-dihydropyrido[2,3-*d*]pyrido[2',3':3,4]pyrazolo[1,5-*a*]pyrimidin-2-ones **6a,b**, and 8,9-dihydro-5H-pyrido[2,3-*d*]thiazolo-[3,2-*a*]pyrimidin-8-ones **8a,b** are new, previously undescribed heterosystems.

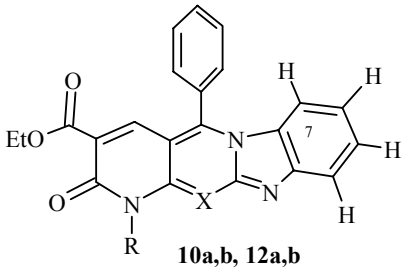
The structures of the products of reaction of 1,2-dihydropyridin-2-ones **2a,b** with such highly basic aminoazines as 3-aminobenzimidazole **9** (p*K*a 7.39, [9]) and 2-(cyanomethyl)benzimidazole **11**, were confirmed by ^{1}H NMR spectroscopy.

The signal of the H-7 proton in the ^{1}H NMR spectra of compounds **10a,b** and **12a,b** was displayed at fairly high fields, at 5.97-6.08 ppm. Such values for proton chemical shifts are not characteristic of benzimidazole and its derivatives, for which, as shown by analysis of literature data, the chemical shifts of protons in positions 4-7 lie in the range 7.20-7.80 ppm [12].



2a, **4a**,**c**, **6a**, **8a**,**b** **10a**, **12a** R^1 = Me, **2b**, **4b**, **6b**, **10b**, **12b** R^1 = Et; **3a**, **4a**,**b** R^2 = H, **3b**, **4c** R^2 = CN; **3a**, **4a**,**b** R^3 = Me, **3b**, **4c** R^3 = H; **7a**, **8a** R^4 = H, **7b**, **8b** R^4 = Me

The anomalous value of the H-7 proton chemical shift in compounds **10a,b** and **12a,b** act, in our opinion, as fair confirmation of the linear structure of these polycyclic compounds. In reality, analysis of spatial models shows that in 1,2-dihydrobenzo[4,5]imidazo[1,2-*a*]pyrido[2,3-*d*]pyrimidin-2-ones **10a,b** and 1,2-dihydrobenzo[4,5]imidazo[1,2-*g*][1,6]naphthyridin-2-ones **12a,b** as a result of the steric difficulties the phenyl ring must be twisted from the planarity of the heterocyclic fragment. An estimate of the size of this dihedral angle for compound **12a** by molecular mechanics methods (MM2) gives a value of ~103°. The position of the phenyl ring causes the H-7 proton to fall in the shielding region of its π-electron ring currents [13], which also leads to a displacement of its signal in the ^{1}H NMR spectra into the stronger field region. Calculation of the contribution of the ring currents of the phenyl ring [14] to the shielding of this proton predicts a displacement of the H-7 proton signal for compound **12a** of 1.9 ppm towards high field, which is in fairly good agreement with the experimental data.



10, **12 a** R = Me, **b** R = Et; **10a**,**b** X = N, **12a**,**b** X = C–CN

TABLE 1. Characteristics of the Synthesized Compounds

Compound	Empirical formula	Found, % / Calculated, %			mp, °C*	Yield, %
		C	H	N		
4a	$C_{20}H_{18}N_4O_3$	66.54 / 66.29	5.18 / 5.01	15.33 / 15.46	223-225	62
4b	$C_{21}H_{20}N_4O_3$	66.89 / 67.01	5.57 / 5.36	15.06 / 14.88	231-233	57
4c	$C_{20}H_{15}N_5O_3$	64.39 / 64.34	4.16 / 4.05	19.02 / 18.76	300-303	53
6a	$C_{22}H_{17}N_5O_3$	66.38 / 66.16	4.37 / 4.29	17.55 / 17.53	368-370	47
6b	$C_{23}H_{19}N_5O_3$	66.84 / 66.82	4.38 / 4.63	17.03 / 16.94	320-324	43
8a	$C_{19}H_{17}N_3O_4S$	59.74 / 59.52	4.40 / 4.47	11.19 / 10.96	350-354	27
8b	$C_{20}H_{19}N_3O_4S$	60.27 / 60.44	5.03 / 4.82	10.63 / 10.57	240-242	24
10a	$C_{23}H_{18}N_4O_3$	69.52 / 69.34	4.67 / 4.55	13.83 / 14.06	325-328	48
10b	$C_{24}H_{20}N_4O_3$	70.02 / 69.89	5.16 / 4.89	13.34 / 13.58	306-309	45
12a	$C_{25}H_{18}N_4O_3$	70.85 / 71.08	4.07 / 4.29	13.35 / 13.26	370-375	56
12b	$C_{26}H_{20}N_4O_3$	71.29 / 71.55	4.68 / 4.62	12.71 / 12.84	355-359	51

*All compounds were recrystallized from DMSO.

TABLE 2. IR Spectra of the Synthesized Compounds

Compound	IR spectrum, ν, cm^{-1}	Compound	IR spectrum, ν, cm^{-1}
4a	3100, 3000, 1720, 1660, 1610, 1530, 1490, 1470, 1440, 1390, 1370	**8b**	3400, 3100, 3000, 1710, 1690, 1620, 1580, 1550, 1530, 1490, 1370
4b	3070, 3000, 1700, 1675, 1600, 1550, 1530, 1490, 1480, 1370	**10a**	3100, 3000, 1730, 1670, 1620, 1580, 1560, 1520, 1490, 1450, 1370
4c	3100, 3000, 2230, 1730, 1660, 1610, 1540, 1500, 1450, 1370	**10b**	3090, 3000, 1725, 1660, 1620, 1580, 1520, 1490, 1455, 1380, 1360
6a	3100, 3000, 1730, 1670, 1620, 1580, 1530, 1500, 1470, 1400, 1375	**12a**	3100, 3000, 2220, 1730, 1680, 1620, 1570, 1530, 1490, 1450, 1370
6b	3060, 3000, 1730, 1660, 1620, 1580 1530, 1490, 1470, 1400, 1380	**12b**	3080, 3000, 2230, 1730, 1670, 1620, 1570, 1530, 1480, 1460, 1380
8a	3400, 3100, 3000, 1700, 1620, 1550, 1470, 1380		

The interaction of 5-benzoyl-3-ethoxycarbonyl-6-methylthio-1-R-1,2-dihydropyrid-2-ones with heterocyclic N,N- and N,C-1,3-dinucleophiles, independent of their basicity, therefore proceeds selectively as a nucleophilic attack of an exocyclic amino (or cyanomethyl) and endocyclic amino group of a dinucleophile at the methylthio and aroyl group of the substrate respectively. This cyclocondensation is a method of synthesis of tri- and tetracyclic heterosystems, including those previously unknown.

EXPERIMENTAL

The ^1H and ^{13}C NMR spectra were recorded on a Varian-300 instrument (300 and 75 MHz respectively), internal standard was TMS. The IR spectra were recorded on a UR-20 instrument in KBr disks.

TABLE 3. ^{1}H and ^{13}C NMR Spectra of the Synthesized Compounds

Com-pound	Chemical shifts, δ, ppm (*J*, Hz)*
4a	1.22 (3H, t, *J* = 6.6, $OCH_2C\underline{H}_3$); 2.35 (1H, s, 2-CH_3); 3.60 (3H, s, 5-CH_3); 4.20 (2H, q, *J* = 6.6, $OC\underline{H}_2CH_3$); 6.43 (1H, H-3); 7.70 (5H, m, C_6H_5); 7.87 (1H, s, H-8)
4b	1.25 (6H, m, 2 $CH_2C\underline{H}_3$); 2.37 (1H, s, 2-CH_3); 4.19 (2H, q, *J* = 6.3, $OC\underline{H}_2CH_3$); 4.38 (2H, q, *J* = 4.8, $NC\underline{H}_2CH_3$); 6.48 (1H, H-3); 7.69 (5H, m, C_6H_5); 7.88 (1H, s, H-8)
4c	1.22 (3H, t, *J* = 6.6, $OCH_2C\underline{H}_3$); 3.65 (3H, s, 5-CH_3); 4.22 (2H, q, *J* = 6.6, $OC\underline{H}_2CH_3$); 7.74 (5H, m, C_6H_5); 7.96 (1H, s, H-8); 8.81 (1H, s, H-2)
6a*[2]	1.44 (3H, t, *J* = 7.2, $OCH_2C\underline{H}_3$); 4.24 (3H, s, 1-CH_3); 4.54 (2H, q, *J* = 7.7, $OC\underline{H}_2CH_3$); 7.77–7.97 (6H, m, H_{Ar}); 8.67 (1H, s, H-4); 9.08 (1H, m, H-10); 9.57 (1H, d, *J* = 6.9, H-9)
6b	1.24 (3H, t, *J* = 6.6, $OCH_2C\underline{H}_3$); 1.39 (3H, t, *J* = 6.3, $NCH_2C\underline{H}_3$); 4.26 (2H, q, *J* = 6.6, $OC\underline{H}_2CH_3$); 4.59 (2H, q, *J* = 6.3, $NC\underline{H}_2CH_3$); 7.23 (1H, m, H_{Ar}); 7.75 (3H, m, H_{Ar}); 7.82 (2H, m, H_{Ar}); 8.03 (1H, s, H-4); 8.67 (1H, m, H-10); 8.84 (1H, d, *J* = 7.0, H-9)
8a	1.50 (3H, t, *J* = 4.2, $OCH_2C\underline{H}_3$); 4.26 (3H, s, 9-CH_3); 4.61 (2H, q, *J* = 4.2, $OC\underline{H}_2CH_3$); 7.59 (1H, m, H_{Ar}), 7.86 (6H, m, H_{Ar}); 9.13 (1H, br. s, H-3)
8b*[3]	1.15 (3H, t, *J* = 6.3, $OCH_2C\underline{H}_3$); 2.17 (3H, s, 2-CH_3); 3.50 (3H, s, 9-CH_3); 4.08 (2H, q, *J* = 6.3, $OC\underline{H}_2CH_3$); 6.77 (1H, s, OH); 7.48 (5H, m, C_6H_5);7.60 (1H, s, H-6); 8.22 (1H, s, H-3)
10a	1.21 (3H, t, *J* = 6.9, $OCH_2C\underline{H}_3$); 3.58 (3H, s, 1-CH_3); 4.20 (2H, q, *J* = 6.9, $OC\underline{H}_2CH_3$); 6.07 (1H, d, *J* = 8.1, H-7); 7.00 (1H, m, H-8); 7.39 (1H, m, H-9); 7.69 (1H, d, *J* = 8.1, H-10); 7.73 (1H, s, H-4); 7.85 (5H, m, C_6H_5)
10b	1.21 (3H, t, *J* = 6.3, $OCH_2C\underline{H}_3$); 1.34 (3H, t, *J* = 6.9, $NCH_2C\underline{H}_3$); 4.20 (2H, q, *J* = 6.3, $OC\underline{H}_2CH_3$); 4.43 (2H, q, *J* = 6.9, $NC\underline{H}_2CH_3$); 6.08 (1H, d, *J* = 7.2, H-7); 7.02 (1H, m, H-8); 7.46 (1H, m, H-9); 7.60 (1H, d, *J* = 6.6, H-10); 7.76 (1H, s, H-4); 7.85 (5H, m, C_6H_5)
12a	1.55 (3H, t, *J* = 3.9, $OCH_2C\underline{H}_3$); 4.58 (3H, s, 1-CH_3); 4.64 (2H, q, *J* = 3.9, $OC\underline{H}_2CH_3$); 6.55 (1H, d, *J* = 5.1, H-7); 7.59 (1H, m, H-8); 7.88 (2H, m, C_6H_5); 8.04 (1H, m, H-9); 8.16 (3H, m, C_6H_5); 8.26 (1H, d, *J* = 4.2, H-10); 8.63 (1H, s, H-4)
12b	1.19 (3H, t, *J* = 6.0, $OCH_2C\underline{H}_3$); 1.47 (3H, t, *J* = 6.3, $NCH_2C\underline{H}_3$); 4.19 (2H, q, *J* = 6.0, $OC\underline{H}_2CH_3$); 4.67 (2H, q, *J* = 6.3, $NC\underline{H}_2CH_3$); 5.97 (1H, d, *J* = 7.0, H-7); 7.05 (1H, m, H-8); 7.49 (1H, m, H-9); 7.60 (1H, d, *J* = 6.8, H-10); 7.72 (1H, s, H-4); 7.88 (5H, m, C_6H_5)

* ^{1}H NMR spectra were recorded in DMSO-d_6 (compounds **4a-c**, **6b**, **8b**, **10a,b,** and **12b**) and CF_3COOD (compounds **6a**, **8a**, and **12a**).

*[2] ^{13}C NMR spectrum of compound **6a** (CF_3COOD), δ, ppm: 12.1 ($OCH_2\underline{C}H_3$); 29.4 (1-CH_3); 64.7 ($O\underline{C}H_2CH_3$); 110.1, 124.7, 125.6, 129.0, 129.6, 130.1, 133.2, 133,7, 140.5, 144.5, 145.2, 145.5, 151.2, 151.4, 152.3 (C_{Ar}); 161.3 (C-2); 165.3 (<u>C</u>OOEt).

*[3] ^{13}C NMR spectrum of compound **8b** (DMSO-d_6), δ, ppm: 12.7 (2-CH_3); 14.8 ($OCH_2\underline{C}H_3$); 29.17 (9-CH_3); 60.0 ($O\underline{C}H_2CH_3$); 86.7 (C-5); 101.3 (C-6), 109.5 (C-5a); 120.3, 123.3, 126.8, 129.0 (C_6H_5); 130.4 (C-2); 132.7 (C-3); 143.4 (C-7); 144.6 (C-9a); 150.1 (C-10a); 159.0 (C-8); 165.4 (<u>C</u>OOEt).

5-Benzoyl-3-ethoxycarbonyl-6-methylthio-1-R-1,2-dihydropyrid-2-ones 2a,b. A solution of sodium ethylate (50 mmol), thioamide **1a,b** (50 mmol), and diethyl ethoxymethylenemalonate (10.80 g, 50 mmol) in anhydrous ethanol (50 ml) was boiled for 1 h, cooled, and methyl iodide (9.23 g, 65 mmol) was added. The mixture was maintained for 2 h at 30°C, cooled, and water (100 ml) was added to the reaction mixture. The solid was filtered off, dried, and recrystallized from 2-propanol. Yield of compound **2a** was 12.08 g (73%), and of **2b** 11,21 g (65%). The melting points and ^{1}H NMR and IR spectra of products **2a,b** were identical to those given in [8].

Compound 2a. ^{13}C NMR spectrum (DMSO-d_6), δ, ppm: 14.6 ($OCH_2\underline{C}H_3$); 19.2 (SCH_3); 34.7 (1-CH_3); 61.1 ($O\underline{C}H_2CH_3$); 119.0 (C-4); 122.3 (C-5); 129.3, 130.0, 134.1, 137.6 (C_6H_5); 141.3 (C-3); 151.9 (C-6); 159.2 (C-2); 164.5 (O=<u>C</u>–OEt); 193.2 (C_6H_5–<u>C</u>=O).

7-Ethoxycarbonyl-9-phenyl-5-R^1-3-R^2-2-R^3-5,6-dihydropyrazolo[1,5-*a*]pyrido[2,3-*d*]pyrimidin-6-ones 4a-c, 3-Ethoxycarbonyl-5-phenyl-1-R^1-1,2-dihydropyrido[2,3-*d*]pyrido[2′,3′:3,4]pyrazolo[1,5-*a*]pyrimidin-2-ones 6a,b, 7-Ethoxycarbonyl-5-hydroxy-9-methyl-5-phenyl-2-R^4-8,9-dihydro-5H-pyrido[2,3-*d*]thiazolo-[3,2-*a*]pyrimidin-8-ones 8a,b, and 12-Cyano-3-ethoxycarbonyl-5-phenyl-1-R^1-1,2-dihydrobenzo[4,5]imidazo[1,2-*g*]naphthyridin-2-ones 12a,b. A solution of 5-benzoyl-3-ethoxycarbonyl-6-methylthio-1-R-1,2-dihydropyridin-2-one **1a,b** (1 mmol) and 5-amino-3-R^1-4-R^2-pyrazole **3a,b,** (or 3-amino-2H-pyrazolo[3,4-*b*]pyridine **5,** 2-amino-5-R-thiazole **7a,b**, 2-(cyanomethyl)benzimidazole **11**) (1 mmol), in 1-hexanol (4 ml) was boiled for 4 h, cooled, and solid compound **4a-c** (or **6a,b**, **8a,b**, **12a,b**) was filtered off. The physicochemical and spectral data are given in Tables 1-3.

3-Ethoxycarbonyl-5-phenyl-1-R^1-1,2-dihydrobenzo[4,5]imidazo[1,2-*a*]pyrido[2,3-*d*]pyrimidin-2-ones 10a,b. A solution of 5-benzoyl-3-ethoxycarbonyl-6-methyl-1-R-1,2-dihydropyridin-2-one **1a,b** (1 mmol) and 2-aminobenzimidazole **9** (1 mmol) in 2-propanol (5 ml) was boiled for 7 h, cooled, and solid compound **10a,b** was filtered off.

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