PURINES, PYRIMIDINES, AND CONDENSED SYSTEMS BASED ON THEM. 9.* CONCERNING THE SYNTHESIS OF 1-R-PYRAZOLO[3,4-d]PYRIMIDINE-4,6-DIONES

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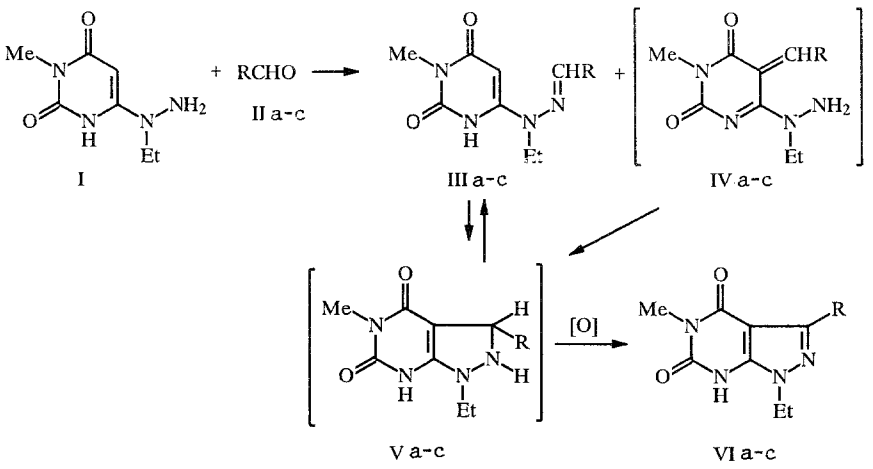
1-Ethyl-3-aryl-5-methylpyrazolo[3,4-d]pyrimidine-4,6-diones are formed in small amounts, together with the expected hydrazones, in the reaction of 3-methyl-6-(1-ethylhydrazino)uracil with aromatic aldehydes in ethanol. The yield of these diones increases substantially when the reaction is carried out in acetic acid and sodium nitrite is subsequently added to the mixture. A change in the temperature conditions makes it possible to direct this reaction toward the formation of 1-ethyl-3-aryl-6-methylpyrimido[5,7-e]as-triazine-5,7-diones.

It was shown in 1973 that arylhydrazones derived from 5-hydrazinouracils cyclize on heating above their melting points into 3-arylpyrazolo[3,4-d]-pyrimidine-4,6-diones in a yield of 20-60% [2]. The reaction requires the elimination of two hydrogen atoms, and this fact (taking into account the absence of oxidizing agents) explains the necessity for such rigorous conditions. In the course of the study of the reactivity of 3-methyl-6-(1-ethylhydrazino)uracil (I), we found that pyrazolo[3,4-d]pyrimidine-4,6-dione derivatives can be also obtained under milder conditions.

When hydrazine I is heated with anisaldehyde, veratraldehyde and p-dimethylaminobenzaldehyde in alcohol, colorless hydrazones III are formed in a 76-80% yield (R_f ~0.3, Silufol, eluent — ethyl acetate). However, according to thin layer chromatography data, in each of these three reactions, together with the corresponding hydrazones, small amounts (yields up to 8%) of a compound with R_f ~0.7 are formed, appearing in UV light in the form of bright blue or blue spots. We have ascribed to these products the structure of 1-ethyl-3-aryl-5-methylpyrazolo[3,4-d]pyrimidine-4,6-diones (VI). The characteristic feature of the PMR spectra of these compounds is the absence of signals in the 5 and 7.5 ppm region, which in the spectra of hydrazones III belong to an H-5 proton of the uracil ring and to the azomethine proton, respectively. The reaction of hydrazine I with aldehydes IIa-c in acetic acid also leads to the formation of a mixture of compounds IIIa-c and IVa-c with the hydrazones predominating.

Theoretically, pyrazoles VIa-c can be obtained as a result of the cyclization of hydrazones IIIa-c themselves as well as of their isomeric 5-arylidene derivatives of the initial hydrazine IVa-c. In both cases, the intermediate should be pyrazoline V,



II-VI a R=4-$MeOC_6H_4$; b R=3,4-$(MeO)_2C_6H_3$; c R=4-$NMe_2C_6H_4$

*For Communication 8, see [1].

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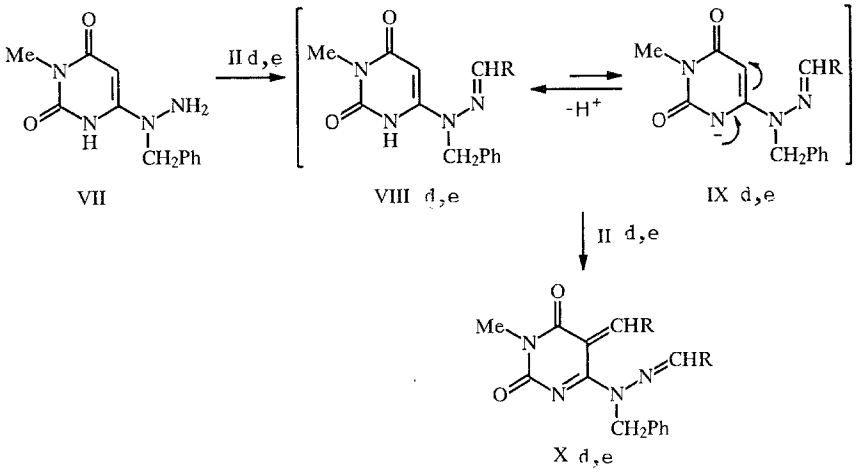
which undergoes an oxidative dehydrogenation, most probably by the action of atmospheric oxygen, but this reaction requires a high temperature. Under the conditions we have used, i.e., on boiling in ethanol for several hours, compounds IIIa-c remain unchanged; however, on heating them in acitic acid they gradually decompose, but the formation of pyrazoles VIa-c is not recorded even chromatographically. From these data, we assume that in the reaction of hydrazine I with aldehydes IIa-c, two reactions are competing: the formation of he corresponding hydrazone IIIa-c and the 5-arylidene derivative IVa-c, whereby the yield of the latter is lower because of the nucleophilicity of the 5-position in the uracil ring compared with the terminal nitrogen atom of the hydrazino group.

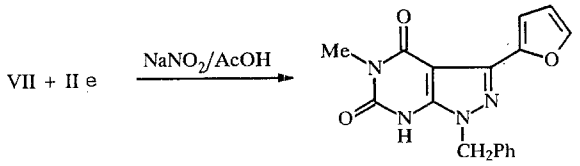
Compounds IVa-c probably cyclize very rapidly with the formation of pyrazolines Va-c, and therefore they cannot be isolated. Their formation is indirectly indicated by a bright-red color of the reaction mixture, although both the starting compounds and the end products are colorless. We confirmed the presence of a red color in 5-arylideneuracils in experiments with 3-methyl-6-(1-benzylhydrazino)uracil (VII). The reaction of this compound with aromatic aldehydes proceeds in a very complex way with the formation of a difficultly separable mixture of many compounds. However, in the case of benzaldehyde and furfural we succeeded in isolating red-orange compounds in a 15% yield, which are, judging from their physicochemical properties, bisarylidene derivatives X. The formation of these compounds is probably the result of a slightly increased acidity of the hydrazones VIIId-e and the initial hydrazine VII, by the action of an I-effect of the benzyl group, resulting in an increase in the concentration of the N-anion in the reaction mixture, for example in the case of IX, in which the attack of the 5-position of an electrophile is facilitated.



VII VIII d,e IX d,e X d,e

II, X d R + Ph, e R = 2-furyl

Since hydrazones VIIId-e could not be isolated, we tried to identify them in the solution in another way. To do this, the abundant orange precipitate formed in the reaction of hydrazine VII with furfural in acetic acid was subjected without isolation to nitrosation. As known, hydrazones of 3-methyl-6-(1-methylhydrazino)uracil readily give 5-nitroso derivatives by the action of nitrous acid, which rapidly cyclize into the corresponding 1-methyl-3-arylpyrimido[5,4-e]-as-triazine-5,7-diones (the 3-aryl-substituted derivatives of the antibiotic toxoflavine) [3, 4]. However, in the case of compound VII, we have isolated in 36% yield only a colorless compound, which on further investigation turned out to be pyrazole XI.



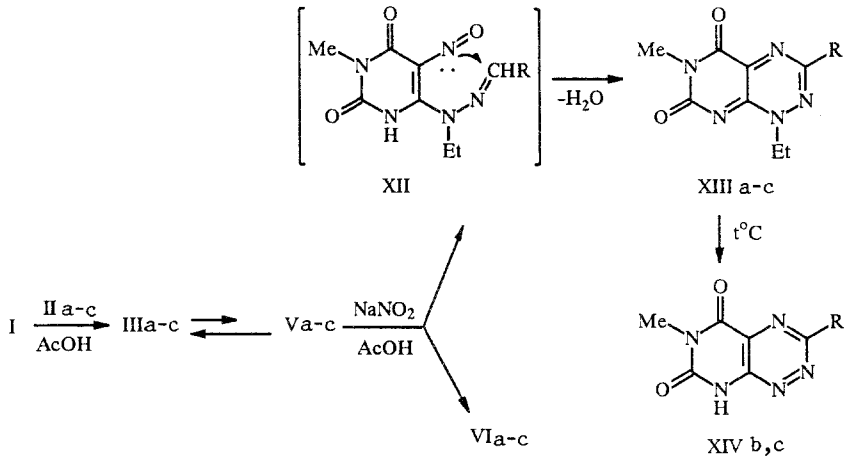
XI

Since the yield of compound XI was substantially higher than the yield of pyrazoles VI in the absence of nitrous acid, we assumed that the latter promotes the cyclization of hydrazones into pyrazoles by dehydrogenation of pyrazolines of type V (see, for example, the dehydrogenation of 1,4-dihydropyridines by means of nitrous acid [5]). It therefore appeared possible that the yield of pyrazoles VI could also be thus increased. This supposition was confirmed.

The results of the reaction of hydrazine I with aldehydes IIa-c in acetic acid with the subsequent addition of sodium nitrite to the reaction mixture are very strongly dependent on the temperature conditions produced after the addition of $NaNO_2$. Thus in the case of anisaldehyde, holding the mixture at 20°C for 30 min leads to the formation of the red-colored 1-ethyl-3-p-

methoxyphenyl-6-methylpyrimido[5,4-e]-as-triazine-5,7-dione (XIIIa) in a 57% yield. If this mixture is boiled for 10 min, then pyrazole VIa is formed in a 52% yield. It was found empirically that the formation of pyrazoles VIb, c, on the contrary, is facilitated by holding the mixture at room temperature; in this case their yields were 44 and 36%, respectively. The formation of pyrimidotriazine XIIIb (yield 60%) is facilitated by heating to 100°C. In an attempt to obtain 1-ethyl-3-p-dimethylamino-6-methylpyrimido[5,4-e]-as-triazine-5,7-dione (XIIIc) under these conditions, its deethylation was unexpectedly observed with the formation of a 3-p-dimethylamino derivative of the rheomycin antibiotic XIVc.

The results obtained indirectly indicate the presence of an equilibrium between hydrazones III and pyrazolines V, which under normal conditions is undoubtedly strongly shifted in the direction of hydrazones. Addition of nitrous acid, promoting the dehydrogenation of pyrazolines, shifts this equilibrium to the right side, which raises the possibility of obtaining pyrazoles VI in a fairly good yield. Unfortunately, from the available data it is difficult to formulate the patterns of conditions for the formation of pyrimidotriazines XIII and pyrazoles VI depending on the nature of the substituent in the 3-aryl ring. It is clear that they have a complex character, which is determined by both the position of the III ⇄ V equilibrium and by the relative reaction rates, leading to the formation of compounds VI and XIII.



XII—XIVa R=4-MeOC$_6$H$_4$,b R=3,4-(MeO)$_2$C$_6$H$_3$, c R=4-NMe$_2$C$_6$H$_4$

EXPERIMENTAL

The PMR spectra were run on a Tesla BS-80 (for compounds IIIa-c, VII, Xb, XIIIa) and Bruker WH-90 (for compounds VIa-c, Xa, XIV) spectrometers with a working frequency of 80 and 90 MHz respectively, using TMS as internal standard. The IR spectra were recorded on a UR-20 spectrophotometer. The mass spectrum was obtained on a Jeol JMS O1 SC-2 mass spectrometer with direct introduction of the sample into the ionic source. Ionizing voltage 75 eV, cathode emission current 300 μA, accelerating voltage 8 kV, temperature of the ionization chamber 125°C (Xb), 110°C (XI). The course of the reaction and the purity of the compounds obtained were monitored by TLC on Silufol UV-254 plates using ethyl acetate as eluent, and the development in an iodine chamber. The melting points were determined in sealed capillaries and are uncorrected.

The elemental analysis data for C, H, N correspond to the calculated values.

Reaction of 6-(1-Ethyl)hydrazinouracil (I)* with Aldehydes IIa-c. A mixture of 1.84 g (0.01 mole) of I and 0.01 mole of II in 20 ml of ethanol was boiled for 2 h. The red-colored solutions were cooled, the precipitate that separated out, which is a mixture of compounds III and VI, was filtered off and washed with a small amount of cold ethanol. Products III and VI were separated by column chromatography (150 g of silica gel, eluent — ethyl acetate). The yield of compounds VI did not exceed 8%. Double recrystallization from ethanol purifies compound III from VI without column chromatography. (On the synthesis of pyrazolopyrimidines VI in higher yields, see below.)

3-Methyl-6-(1-ethyl-2-p-methoxybenzylidene)hydrazinouracil (IIIa, $C_{15}H_{18}N_4O_3$). Pale-yellow crystals, mp 204°C (from ethanol). R_f 0.27. PMR spectrum ($CDCl_3$): 1.25 (3H, t, J = 7.2 Hz, $-CH_2\underline{CH_3}$), 3.27 (3H, s, N-CH_3), 3.82 (3H, s, O-CH_3), 3.82 (2H, q, J = 7.2 Hz, $\underline{CH_2}CH_3$), 5.07 (1H, d, J = 4 Hz, H-5), 6.93 (2H, d, J = 10 Hz, H-2′,6′), 7.60 (2H, d, J = 10 Hz, H-3′,5′), 7.65 (1H, s, =CH—), 9.20 ppm (br.s, NH). IR spectrum (mineral oil): 1600 (ring), 1630, 1710 (C=O), 3130 (=CH—), 3200-3400 cm^{-1} (ass. NH). Yield 2.3 g (76%).

*The synthesis of compound I will be reported separately.

3-Methyl-6-[1-ethyl-2-(3′,4′-dimethoxy)benzylidene)]hydrazinouracil (IIIb, $C_{16}H_{20}N_4O_4$). White crystals, mp 192°C (from ethanol). R_f 0.25. PMR spectrum ($CDCl_3$): 1.25 (3H, t, J = 7.2 Hz, $-CH_2\underline{CH_3}$), 3.27 (3H, s, N-CH_3), 3.90 (3H, s, O-CH_3), 3.92 (3H, s, O-CH_3), 3.91 (2H, q, J = 7.2 Hz, $\underline{CH_2}CH_3$), 5.07 (1H, d, J = 4 Hz, H-5), 6.95-7.25 (3H, m, H_{arom}), 7.55 (1H, s, =CH—), 9.20 ppm (br.s, NH). IR spectrum (mineral oil): 1600 (ring), 1630, 1700 (C=O), 3100 (=CH—), 3120-3300 cm^{-1} (ass. NH). Yield 2.6 g (80%).

3-Methyl-6-(1-ethyl-2-p-dimethylaminobenzylidene)hydrazinouracil (IIIc, $C_{16}H_{21}N_5O_2$). Cream-colored crystals, mp232°C(fromethanol). R_f 0.3, PMR spectrum ($CDCl_3$): 1.22 (3H, t, J = 7.2 Hz, $-CH_2\underline{CH_3}$), 2.96 (6H, s, $-N(CH_3)_2$, 3.27 (3H, s, N-CH_3), 3.82 (2H, q, J = 7.2 Hz, $-\underline{CH_2}CH_3$), 5.05 (1H, d, J = 4.2 Hz, H-5), 6.68 (2H, d, J = 10 Hz, H-2′,6′), 7.50 (2H, d, J = 10 Hz, H-3′,5′), 7.65 (1H, s, =CH—), 9.20 ppm (br.s, NH). IR spectrum (mineral oil): 1610 (ring), 1660, 1710 (C=O), 3100 (=CH—), 3380-3420 cm^{-1} (NH). Yield 2.5 g (79%).

3-Methyl-6-(1-benzylhydrazino)uracil (VII, $C_{12}H_{14}O_2$). A mixture of 4 g (0.025 mole) of 3-methyl-6-chlorouracil and 16.6 g (0.14 mole) of benzylhydrazine in 25 ml of ethanol was boiled for 6 h. The ethanol was evaporated on a rotary evaporator, and from the remaining yellow oil, crystals of the reaction product were precipitated with water. They were filtered, washed successively with cold water and alcohol, and were dried, first in air, and then in a vacuum-desiccator over $CaCl_2$. The yield of the crude product was 3.9 g (64%). It was purified by recrystallization from ethanol or dioxane. White crystals, which gradually turned yellow in darkness (in light they become white again). Mp 211-212°C. PMR spectrum (DMSO-D_6): 3.23 (3H, s, N—CH_3), 4.04 (2H, s, —CH_2—), 5.08 (1H, s, H-5), 7.55 ppm (5H, pseudo-singlet, C_6H_5). IR spectrum (mineral oil): 1700 (C=O), 3185 (w. br., NH), 3255 cm^{-1} (s, narrow, NH_2).

2,3,4,5-Tetrahydro-3-methyl-5-benzylidene-6-(1-benzyl-2-benzylidene)hydrazinopyrimidine-2,4-dione(Xd, $C_{26}H_{22}N_4O_2$). A 0.53 g portion (5 mmoles) of benzaldehyde was added to a suspension of 1.2 g (5 mmoles) of compound VII in 20 ml of ethanol and the mixture was boiled for 3 h. Alcohol was evaporated on a rotary evaporator from the red solution formed and the residue was pulverized. The crystals obtained were dissolved in 7 ml of chloroform and the solution was chromatographed on a column (130 g of silica gel, eluent — chloroform). The first fraction was collected. The yield of the crude product was 0.3 g (14%). Red-orange crystals, mp 175-177°C (from ethanol), R_f 0.8 (eluent — chloroform). PMR spectrum (DMSO-D_6): 3.11 (3H, s, N—CH_3), 4.22 (2H, br.s, —CH_2—), 6.07 (1H, s, C=C—), 7.20-7.65 (m, H_{arom}), 8.03 ppm (1H, s, N=CH—).

2,3,4,5-Tetrahydro-3-methyl-5-α-furfurylidene-6-(1-benzyl-2-α-furfurylidene)hydrazinopyrimidine-2,5-dione(Xe, $C_{22}H_{16}N_4O_4$). A mixture of 0.62 g (2.5 mmoles) of hydrazine VII and 0.28 ml (3.4 mmoles) of furfural was boiled in 4 ml of ethanol for 15 min. The crystals (0.54 g) that precipitated after cooling were separated and washed with a small amount of cold alcohol. The crude product consists of a complex mixture. By multiply repeated recrystallization from methanol or by means of a preparative TLC on silica gel, 0.15 g (15%) of orange crystals of compound Xe were isolated with R_f 0.54 (chloroform), mp 181-183°C. PMR spectrum ($CDCl_3$): 3.28 (3H, s, CH_3), 4.11 (1H, d, J = 13.6 Hz, —CH_2—), 4.48 (1H, d, J = 13.6 Hz, —CH_2—), 6.18 (1H, s, C=C—), 6.39 (1H, q, J = 1.7, 3.3 Hz, H-4′), 6.52 (2H, m, H-3′,4′), 7.33 (5H, pseudosinglet, C_6H_5), 7.46 (1H, d, J = 1.3 Hz, H-5′), 7.68 (1H, d, J = 1.7 Hz, H-5′), 8.08 (1H, s, N=CH—), 8.36 ppm (1H, d, J = 3.8 Hz, H-3′). IR spectrum (chloroform): 1600 (ring), 1665, 1713 cm^{-1} (C=O). Mass spectrum, m/z (% I): 402 (28) M^+, 311 (16) [M—$C_6H_5CH_2$], 254 (60) [311—CH_3NCO], 226 (18) [254-CO], 91 (100).

1-Benzyl-3-(2-furyl-5-methylpyrazolo[3,4-d]pyrimidine-4,6-dione (XI, $C_{17}H_{14}N_4O_3$). A 0.7 g portion (3 mmoles) of hydrazine VII was dissolved with heating in 5 ml of glacial acetic acid. To the solution obtained 0.32 ml (4 mmoles) of furfural was added and the mixture was boiled for 20 min. On cooling a thick red precipitate precipitated. A solution of 0.21 g (3 mmoles) of sodium nitrite in 0.5 ml of water was added to the suspension obtained, and the mixture was stirred for 2 h at 20°C and was then allowed to stand for 12 h in a refrigerator. The crystals that separated out were filtered off, washed with ice water, and dried. Almost greyish crystals, mp 259-261°C (from ethanol or ethyl acetate). PMR spectrum (CF_3COOH): 3.04 (3H, s, CH_3), 5.28 (2H, s, —CH_2—), 6.25 (1H, br.s, H-4′), 6.80 (5H, m, C_6H_5), 7.28 ppm (2H, m, H-3′,5′). IR spectrum (mineral oil): 1630 (ring), 1680, 1740 cm^{-1} (C=O). Mass spectrum, m/z (% I): 322 (29) M^+, 265 (3) [M—CH_3NCO], 237 (2) [265-CO], 229 (5) [M^+—C_4H_3O—CN], 91 (100) $C_6H_5CH_2$. Yield 0.35 g (36%).

1-Ethyl-3-p-methoxyphenyl-6-methylpyrimido[5,4-e]as-triazine-5,7-dione (XIIIa, $C_{15}H_{15}N_5O_3$). A mixture of 1.84 g (0.01 mole) of ethylhydrazinouracil I and 1.36 g (0.01 mole) of anisaldehyde in 10 ml of glacial acetic acid was boiled for 3 h. The mixture was cooled to 20°C and a solution of 0.7 g (0.01 mole) of sodium nitrite in 5 ml of water was added. The dark-pink colored reaction mixture was stirred for 30 min at room temperature. The precipitate that separated out was filtered off, washed with a small amount of water, and then with ethanol. Red crystals, mp 190°C (from butanol, dec.), R_f 0.5. PMR spectrum ($CDCl_3$): 1.60 (3H, t, J = 7.2 Hz, $-CH_2CH_3$), 3.45 (3H, s, N-CH_3), 3.85 (3H, s, O—CH_3), 4.65 (2H, q, J = 7.2 Hz, $-CH_2CH_3$), 6.92 (2H, d, H-2′,6′), 8.15 ppm (2H, d, J = 10 Hz, H-3′,5′). IR spectrum (mineral oil): 1600 (ring), 1655, 1700 cm^{-1} (C=O). Yield 1.8 g (57%).

1-Ethyl-3-(3′,4′-dimethoxyphenyl-6-methylpyrimido[5,4-e]-as-triazine-5,7-dione (XIIIb, $C_{16}H_{17}N_5O_4$). A mixture of 1.84 g (0.01 mole) of ethylhydrazinouracil I and 1.7 g (0.01 mole) of veratraldehyde in 10 ml of glacial acetic acid was boiled for 3 h. The mixture was cooled to 20°C, and a solution of 0.7 g (0.01 mole) of sodium nitrite in 5 ml of water was added. The reaction mixture was stirred on a boiling water bath for 1 h, and then for 4 h at 20°C. The thick red paste was diluted threefold with diethyl ether, and the fine precipitate was filtered off. Red crystals, R_f 0.47. The product was not analyzed, since on recrystallization, it becomes deethylated and converts into XIVb. Yield 2 g (60%).

3-(p-Diethylaminophenyl)-6-methylpyrimido[5,4-e]-as-triazine-5,7(6H, 8H)-dione (XIVc, $C_{14}H_{14}N_6O_2$). A 1.6 g portion (5 mmoles) of hydrazone III was suspended in 10 ml of glacial acetic acid. To the thick orange paste that has formed, a solution of 0.35 g (5 mmoles) of sodium nitrite in 3 ml of water was added. The color deepened to brown. The reaction mixture was boiled for 4 h, then was cooled, the precipitate was filtered off, and washed with cold water and ether. Red-violet crystals, mp 330°C (from DMFA). PMR spectrum (DMSO-D_6): 3.03 (6H, s, N—$(CH_3)_2$), 3.29 (3H, s, N—CH_3), 6.85 (2H, d, J = 9 Hz, H-2′,6′), 8.23 (2H, d, J = 9 Hz, H-3′,5′), 12.6 ppm (1H, s, NH). a 42% portion of the starting hydrazone IIIc was recovered from the reaction mixture. Yield 0.47 g (31%).

1-Ethyl-3-(p-methoxyphenyl-5-methylpyrazolo[3,4-d]-pyrimidine 4,6-dione (VIa, $C_{15}H_{16}N_4O_3$). A mixture of 1.84 g (0.01 mole) of ethylhydrazinouracil I and 1.36 g (0.01 mole) of anisaldehyde in 10 ml of glacial acetic acid was boiled for 3 h. The mixture was cooled to 20°C and a solution of 0.7 g (0.01 mole) of sodium nitrite in 5 ml of water was added. The reaction mixture with the red precipitate that separated out was heated to boiling. The precipitate dissolved. After 10 min a light-colored precipitate separated out, which was filtered after cooling. White crystals, mp 236°C (from ethanol), R_f 0.7. PMR spectrum ($CDCl_3$): 1.48 (3H, t, J = 7.2 Hz, —CH_2CH_3), 3.35 (3H, s, N—CH_3), 3.88 (3H, s, O—CH_3), 4.22 (2H, q, J = 7.2 Hz, —CH_2CH_3), 7.05 (2H, d, J = 10 Hz, d, H-2′,6′), 7.45 (2H, d, J = 10 Hz, H-3′,5′), 10.24 ppm (1H, s, NH). IR spectrum (mineral oil): 1620 (ring), 1690, 1730 (C=O), 3100-3300 cm^{-1} (ass. NH). Yield 1.6 g (52%).

1-Ethyl-3-(3′,4′-dimethoxyphenyl)-5-methylpyrazolo[3,4-d]-pyrimidine-4,6-dione (VIb, $C_{16}H_{18}N_4O_4$). A mixture of 1.84 g (0.01 mole) of ethylhydrazinouracil I and 1.7 g (0.01 mole) of veratraldehyde in 10 ml of glacial acetic acid was boiled for 3 h. The mixture was cooled to 20°C a solution of 0.7 g (0.01 mole) of sodium nitrite in 5 ml of water was added. The mixture was stirred at 20°C for 3 h, allowed to stand for 12 h in a refrigerator, and was diluted by a tenfold volume of water. The cream-colored crystals that separated out were filtered off, mp 221°C (from ethanol). R_f 0.7. PMR spectrum ($CDCl_3$): 1.47 (3H, t, J = 7.2 Hz, —$CH_2\underline{CH_3}$), 3.34 (3H, s, N—CH_3), 3.95 (6H, s, 2—O—CH_3), 4.18 (2H, q, J = 7.2 Hz, —$\underline{CH_2}CH_3$), 7.05 (2H, s, H_{arom}), 9.23 ppm (1H, s, NH). IR spectrum (mineral oil): 1625 (ring), 1690, 1725 (C=O), 3100-3250 cm^{-1} (ass. NH). Yield 1.5 g (44%).

1-Ethyl-3-(p-dimethylaminophenyl)-5-methylpyrazolo[3,4,-d]-pyrimidine-4,6-dione (VIc, $C_{16}H_{19}N_5O_2$). A 1.84 g portion (0.01 mole) of ethylhydrazinouracil I and 1.49 g (0.01 mole) of p-dimethylaminebenzaldehyde were mixed together in a dry state, 10 ml of glacial acetic acid was added, and the mixture was boiled for 3 h. The red-colored reaction mixture was cooled to room temperature and a solutionn of 0.7 g (0.01 mole) of sodium nitrite in 3 ml of water was added. The mixture was stirred at room temperature for 2 h and was allowed to stand in a refrigerator for 12 h. The precipitate was filtered off, washed with a small amount of ice water, and then with cold ethanol. White crystals, mp 305°C (from ethanol). R_f 0.7. PMR spectrum ($CDCl_3$): 1.47 (3H, t, J = 7.2 Hz, $-CH_2\underline{CH_3}$), 3.04 (6H, s, N$-(CH_3)_2$, 3.34 (3H, s, N$-CH_3$), 4.18 (2H, q, J = 7.2 Hz, $-\underline{CH_2}CH_3$), 6.80 (2H, s, J = 9 Hz, H-2′,6′), 7.39 (2H, d, J = 9 Hz, H-3′,5′), 9.05 ppm (1H, s, NH). IR spectrum (mineral oil): 1620 (ring), 1680, 1720 (C=O), 3160-3220 cm^{-1} (ass. NH). Yield 1.1 g (36%).

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