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SYNTHESIS OF DERIVATIVES OF THE NEW CONDENSED SYSTEM 4H,7H-FURO[3′,4′:6,7]CYCLOHEPTA-[1,2-*b*]PYRAN

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8-Hydroxy-1,3-dimethyl-4H-cyclohepta[c]furan-4-one reacts with arylidenemalononitriles and, like 1,3-oxo enols, forms the corresponding condensed 2-amino-4H-pyrans. The analogous reaction with 3-(dicyanomethylene)indolin-2-ones give spirocyclic 2-amino-4H-pyrans. The 4-aminopyrimidine ring is formed on the basis of the enamino nitrile fragment of the new pyrans by successive reaction with triethoxymethane and then with aqueous ammonia.

Keywords: 2-amino-4H-pyran, 4-aminopyrimidine, benzylidene malononitrile, hydroxytropone, enol, pyrano[2,3-*d*]pyrimidine, tropolone, tropone, Michael addition.

2-Amino-4H-pyrans have attracted the attention of researchers for more than a decade [1-4]. This interest is explained by the prospects of using them as drugs for the treatment of diseases of the central nervous system (dementia, Parkinson's disease) [5], arthritis, sclerosis, tumors [6], and arterial hypertension [7]. They are also attracted by the comparative simplicity of their synthesis and the possibility of using them as synthons for the production of more complicated condensed systems [8-10].

The aim of the present investigation was to synthesize condensed 2-amino-3-cyano-4H-pyrans from 8-hydroxy-1,3-dimethylcyclohepta[*c*]furan-4-one (**1**) and certain ylidene derivatives of malononitrile.

It had been shown by numerous investigations that ylidene malononitriles form 2-amino-3-cyano-4H-pyrans in reaction with active enols having various structures – electron-rich phenols, naphthols, carbocyclic and heterocyclic 1,3-oxo enols [1-4].

Compound **1**, which became available comparatively recently, is also a typical enol (or, more accurately, a vinylog of a cyclic 1,3-oxo enol – 1,5-oxo dienol). This is demonstrated by its IR and ^{1}H NMR spectra and also by its physical characteristics – it is a yellow substance with a comparatively high melting point and low solubility in nonpolar or low-polarity solvents [11].

Earlier neither the hydroxytropone **1** itself nor its benzannulated analogs had been brought into similar reactions with unsaturated nitriles. These reactions had also not been investigated for monocyclic hydroxytropones. Their study is therefore of interest, while the expected products containing the 4H-pyran fragment, which has already exhibited its medicinal activity, and the heteroannulated tropone cyclic system, which also exhibits various types of activity in the composition of natural and synthetic compounds [12], may have useful physiological activity.

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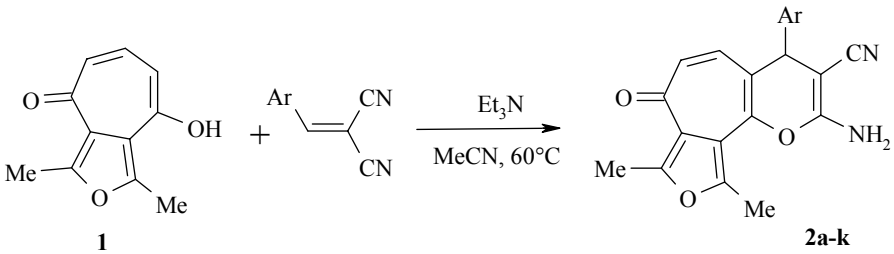


1 **2a-k**

2 a Ar = 4-$MeOC_6H_4$, **b** Ar = 4-$EtOC_6H_4$, **c** Ar = 4-$MeSC_6H_4$, **d** Ar = 4-FC_6H_4, **e** Ar = 4-BrC_6H_4, **f** Ar = 2-FC_6H_4, **g** Ar = 2-ClC_6H_4, **h** Ar = 3,4-$OCH_2OC_6H_3$, **i** Ar = 2,4-$Cl_2C_6H_3$, **j** Ar = Ph, **k** Ar = 2-thienyl

It was found that the hydroxytropone **1** reacts readily with arylidene derivatives of malononitrile with the formation of the expected 2-amino-4H-pyrans **2a-k**. The reaction takes place under mild conditions in acetonitrile at 50-60°C after the addition of catalytic amounts of piperidine or triethylamine and gives yields of 30-80%.

Since the result of the reaction corresponded to the expected result, the mechanism proposed earlier for similar reactions can be used for its description. Electrophilic attack is probably directed at position 7 of the initial hydroxytropone, after which the Michael-type adduct undergoes intramolecular cyclization, forming compound **2**.

TABLE 1. The Characteristics of the Synthesized Compounds

Compound	Empirical formula	Found, % / Calculated, %			mp, °C	Yield, %
		C	H	N		
2a	$C_{22}H_{18}N_2O_4$	70.81 / 70.58	4.67 / 4.85	7.55 / 7.48	246-248	82
2b	$C_{23}H_{20}N_2O_4$	71.41 / 71.12	4.97 / 5.19	7.30 / 7.21	274-276	44
2c	$C_{22}H_{18}N_2O_3S$	67.51 / 67.67	4.67 / 4.65	7.01 / 7.17	259-261	57
2d	$C_{21}H_{15}FN_2O_3$	69.56 / 69.61	4.27 / 4.17	7.72 / 7.73	230-232	42
2e	$C_{21}H_{15}BrN_2O_3$	59.12 / 59.59	3.73 / 3.57	6.42 / 6.62	280-282	45
2f	$C_{21}H_{15}FN_2O_3$	69.82 / 69.61	4.11 / 4.17	7.67 / 7.73	256-258	53
2g	$C_{21}H_{15}ClN_2O_3$	66.59 / 66.58	3.91 / 3.99	7.31 / 7.40	277-279	52
2h	$C_{22}H_{16}N_2O_5$	67.75 / 68.04	4.27 / 4.15	7.21 / 7.21	277-279 (dec.)	63
2i	$C_{21}H_{14}Cl_2N_2O_3$	61.10 / 61.03	3.35 / 3.41	6.73 / 6.78	252-254	59
2j	$C_{21}H_{16}N_2O_3$	73.22 / 73.24	4.69 / 4.68	8.43 / 8.13	260-262	32
2k	$C_{19}H_{14}N_2O_3S$	65.19 / 65.13	3.77 / 4.03	8.29 / 7.99	272-273	54
4a	$C_{22}H_{15}N_3O_4$	68.61 / 68.57	3.99 / 3.92	10.77 / 10.90	>350 (dec.)	75
4b	$C_{23}H_{17}N_3O_4$	69.03 / 69.17	4.39 / 4.29	10.71 / 10.52	336-337 (dec.)	28
4c	$C_{24}H_{19}N_3O_4$	69.70 / 69.72	4.69 / 4.63	10.46 / 10.16	265-267	56
5	$C_{25}H_{22}N_2O_5$	69.38 / 69.76	5.09 / 5.15	6.65 / 6.51	160-162	53
7	$C_{23}H_{19}N_3O_4$	69.02 / 68.82	4.75 / 4.77	10.68 / 10.47	244-246	46

The pyran **2a** was synthesized successfully by a three-component condensation, when the hydroxytropone **1**, 4-methoxybenzaldehyde, and malononitrile were brought into reaction in equimolar amounts. In this case, however, a less pure product was obtained, and after repeated recrystallization the total yield was lower than with the use of the previously prepared ylidene derivative.

Compounds **2a-k** are yellow crystalline substances poorly soluble in chloroform and alcohol and readily soluble in pyridine and DMF.

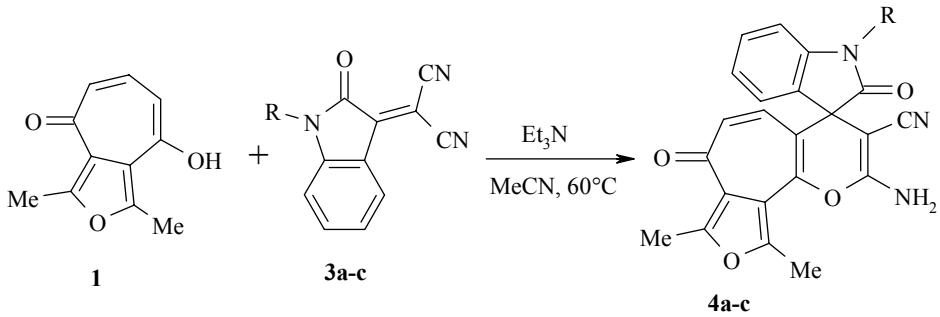
The ^{1}H NMR spectra of the products **2a-k** contain signals for the methyl groups of the furan ring in the form of three-protons singlets at 2.50-2.75 and 2.68-2.75 ppm, two doublets for the methine protons of the seven-membered ring at 6.05-6.20 and 6.20-6.65 ppm, and a signal for the pyran proton in the region of 4.20-4.90 ppm. The broad singlet for the protons of the amino group in the ^{1}H NMR spectra of the compounds, recorded in DMSO-d_6, appears in the region of 7.00-7.15 ppm, and those in the spectra obtained in $CDCl_3$ appear in the region of 4.50-4.60 ppm.

The IR spectra of compounds **2a-k** contain absorption bands corresponding to the vibrations of the N–H bonds of the amino groups at 3153 cm^{-1}, the nitrile group at 2180-2193 cm^{-1}, and the conjugated carbonyl at 1666-1693 cm^{-1}.

In order to synthesize spiroannulated 4H-pyrans the initial hydroxytropone **1** was brought into reaction with a series of ylidene derivatives of malononitrile obtained from such cyclic ketones as isatin, N-alkylisatins, ninhydrin, and cyclohexanone.

The reactions with isatin derivatives **3a-c** were successful. The reaction takes place under the same conditions as the reaction of hydroxytropone **1** with arylidene malononitriles. The spiro compounds **4a-c** are formed in good yields.

The ^{1}H NMR spectra of the products **4a-c** contain signals for the methyl groups of the furan ring in the form of three-proton singlets at 2.60 and 2.73 ppm, two doublets for the methine protons of the seven-membered ring at 5.70 and 5.95 ppm, and a broad singlet for the protons of the amino group at 6.82 or 7.35 ppm.



3, **4 a** R = H, **b** R = Me, **c** R = Et

Since the carbon atom at position 4 of the pyran ring in compounds **2** and **4** is asymmetric, the introduction of a diastereotopic group into the molecule would make it possible to detect its presence in the ^{1}H NMR spectrum. In fact the signal for the diastereotopic methylene of the N-ethyl fragment in the spectrum of compound **4c** is complicated and represents a doublet of quartets and not the usual quartet.

The IR spectra of compounds **4a-c** contain absorption bands corresponding to the vibrations of the N–H bonds of the amino group at 3273 and 3153 cm^{-1}, the nitrile group at 2180 cm^{-1}, the carbonyl of the isatin fragment at 1693 cm^{-1}, and the conjugated carbonyl at 1653 cm^{-1}.

With the ninhydrin derivative the reaction probably takes place in a more complicated way in so far as under standard conditions the reaction mixture becomes very dark, and a poorly soluble dark tarry precipitate that could not be purified separates. Cyclohexylidene malononitrile exhibited low reactivity; even when the reaction mixture was kept for several days, according to TLC, it contained significant amounts of unreacted hydroxytropone **1** and a complex mixture of several products, which could not be separated or identified.

As already mentioned, on the basis of the enamino nitrile fragment of 2-amino-3-cyano-4H-pyrans it is possible to form more complex pyrimidine-, pyrido-, or pyrazolo-condensed systems.

TABLE 2. The Spectral Characteristics of the Synthesized Compounds

Com-pound	IR spectrum, ν, cm^{-1}	^{1}H NMR spectrum, δ ppm (*J*, Hz)*
1	2	3
2a	3286, 3153, 2180, 1666, 1593, 1540, 1500, 1240, 1206	2.60 (3H, s, 10-CH_3); 2.72 (3H, s, 8-CH_3); 3.72 (3H, s, OCH_3); 4.25 (1H, s, H-4); 6.05 (1H, d, *J* = 13, H-6); 6.43 (1H, d, *J* = 13, H-5); 6.90 (2H, d, *J* = 9, H-3'+H-5'); 7.00 (2H, br. s, NH_2); 7.15 (2H, d, *J* = 9, H-2'+H-6')
2b	3286, 3140, 2180, 1673, 1633, 1600, 1533, 1246, 1200	1.38 (3H, t, *J* = 8, $OCH_2C\underline{H}_3$); 2.70 (6H, s, 8-CH_3+10-CH_3); 4.00 (2H, q, *J* = 8, $OC\underline{H}_2CH_3$); 4.20 (1H, s, H-4); 4.48 (2H, br. s, NH_2); 6.12 (1H, d, *J* = 13, H-6); 6.30 (1H, d, *J* = 13, H-5); 6.82 (2H, d, *J* = 9, H-3'+H-5'); 7.15 (2H, d, *J* = 9, H-2+H-6')
2c	3286, 3140, 2180, 1673, 1600, 1553, 1200	2.50 (3H, s, SCH_3); 2.75 (6H, s, 8-CH_3+10-CH_3); 4.28 (1H, s, H-4); 4.57 (2H, br. s, NH_2); 6.20 (1H, d, *J* = 13, H-6); 6.35 (1H, d, *J* = 13, H-5); 7.22 (2H, d, *J* = 9, H-3'+H-5'); 7.28 (2H, d, *J* = 9, H-2'+H-6')
2d	3300, 3166, 2186, 1673, 1600, 1553, 1500, 1206	2.68 (6H, s, 8-CH_3+10-CH_3); 4.23 (1H, s, H-4); 4.50 (2H, br. s, NH_2); 6.13 (1H, d, *J* = 13, H-6); 6.26 (1H, d, *J* = 13, H-5); 7.02 (2H, dd, *J* = 9, *J* = 9, H-3'+H-5'); 7.19 (2H, dd, *J* = 9, H-2'+H-6')
2e	3326, 3193, 2193, 1673, 1633, 1606, 1540, 1213	2.60 (3H, s, 10-CH_3); 2.72 (3H, s, 8-CH_3); 4.39 (1H, s, H-4); 6.06 (1H, d, *J* = 13, H-6); 6.47 (1H, d, *J* = 13, H-5); 7.10 (2H, br. s, NH_2); 7.22 (2H, d, *J* = 9, H-3'+H-5'); 7.55 (2H, d, *J* = 9, H-2'+H-6')
2f	3313, 3160, 2186, 1673, 1600, 1553, 1500, 1206	2.60 (3H, s, 10-CH_3); 2.72 (3H, s, 8-CH_3); 4.60 (1H, s, H-4); 6.07 (1H, d, *J* = 13, H-6); 6.43 (1H, d, *J* = 13, H-5); 7.10 (2H, br. s, NH_2); 7.15-7.35 (4H, m, H-3'+H-4'+H-5'+ H-6')
2g	3313, 3180, 2186, 1673, 1600, 1553, 1206	2.60 (3H, s, 10-CH_3); 2.72 (3H, s, 8-CH_3); 4.83 (1H, s, H-4); 6.05 (1H, d, *J* = 13, H-6); 6.28 (1H, d, *J* = 13, H-5); 7.05 (2H, br. s, NH_2); 7.18-7.48 (4H, m, H-3'+H-4'+H-5'+ H-6')
2h	3286, 3140, 2180, 1673, 1633, 1600, 1533, 1246, 1200	2.61 (3H, s, 10-CH_3); 2.71 (3H, s, 8-CH_3); 4.27 (1H, s, H-4); 5.99 (2H, s, OCH_2O); 6.07 (1H, d, *J* = 13, H-6); 6.50 (1H, d, *J* = 13, H-5); 6.70-6.78 (2H, m, H-4'+H-7'); 6.87 (1H, d, *J* = 8, H-6'); 7.00 (2H, br. s, NH_2)
2i	3313, 3180, 2186, 1673, 1600, 1553, 1206	2.68 (3H, s, 10-CH_3); 2.70 (3H, s, 8-CH_3); 4.60 (2H, br. s, NH_2); 4.90 (1H, s, H-4); 6.12 (1H, d, *J* = 13, H-6); 6.20 (1H, d, *J* = 13, H-5); 7.14 (1H, d, *J* = 8, H-6'); 7.22 (1H, dd, *J* = 8, *J* = 2, H-5'); 7.40 (1H, d, *J* = 2, H-3')
2j	3300, 3153, 2186, 1673, 1593, 1553, 1206	2.62 (3H, s, 10-CH_3); 2.72 (3H, s, 8-CH_3); 4.34 (1H, s, H-4); 6.05 (1H, d, *J* = 13, H-6); 6.50 (1H, d, *J* = 13, H-5); 7.03 (2H, br. s, NH_2); 7.20-7.38 (5H, m, C_6H_5)
2k	3433, 3140, 2180, 1673, 1633, 1600, 1553, 1193	2.61 (3H, s, 10-CH_3); 2.68 (3H, s, 8-CH_3); 4.72 (1H, s, H-4); 6.12 (1H, d, *J* = 13, H-6); 6.67 (1H, d, *J* = 13, H-5); 6.95 (1H, dd, *J* = 5, *J* = 3, H-4'); 7.03 (1H, d, *J* = 3, H-3'); 7.13 (2H, br. s, NH_2); 7.43 (1H, d, *J* = 5, H-5')
4a	3273, 3153, 2180, 1693, 1653, 1600, 1553, 1206	2.60 (3H, s, 10-CH_3); 2.73 (3H, s, 8-CH_3); 5.75 (1H, d, *J* = 13, H-6); 6.05 (1H, d, *J* = 13, H-5); 6.92 (1H, d, *J* = 8, H-7'); 7.05 (1H, t, *J* = 8, H-5'); 7.18-7.38 (4H, m, NH_2+H-4'+H-6'); 10.75 (1H, s, CO–NH)
4b	3273, 3153, 2180, 1693, 1653, 1600, 1553, 1206	2.62 (3H, s, 10-CH_3); 2.73 (3H, s, 8-CH_3); 3.20 (3H, s, N–CH_3); 5.70 (1H, d, *J* = 13, H-6); 5.95 (1H, d, *J* = 13, H-5); 7.08-7.19 (2H, m, H-7'+H-5'); 7.23–7.45 (4H, m, NH_2+H-4'+H-6')
4c	3270, 3153, 2186, 1693, 1653, 1600, 1206	1.20 (3H, t, *J* = 7, $CH_2C\underline{H}_3$); 2.63 (3H, s, 10-CH_3); 2.75 (3H, s, 8-CH_3); 3.80 (2H, dq, *J* = 7, *J* = 10, $C\underline{H}_2CH_3$); 5.67 (1H, d, *J* = 13, H-6); 5.96 (1H, d, *J* = 13, H-5); 6.82 (2H, br. s, NH_2); 7.07–7.17 (2H, m, H-7'+H-5'); 7.25 (1H, d, *J* = 8, H-4'); 7.40 (1H, t, *J* = 8, H-6')

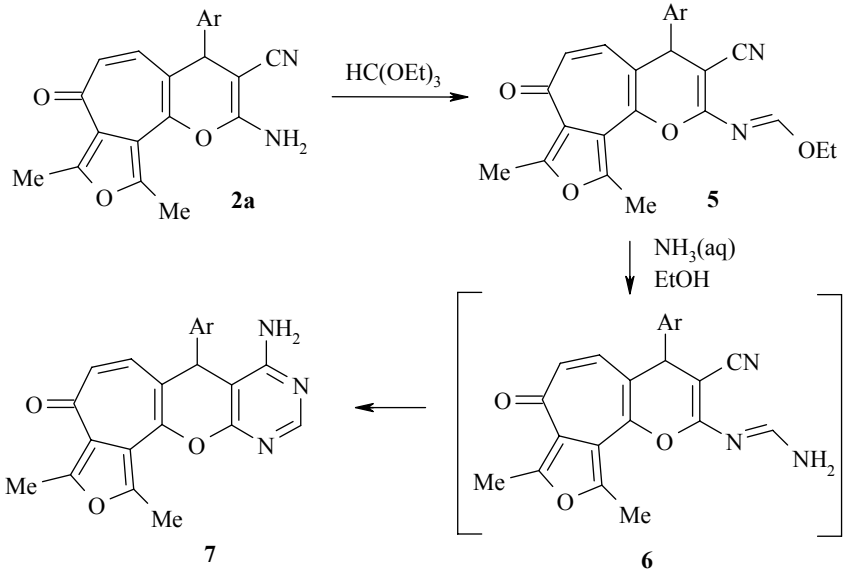
TABLE 2. (continued)

1	2	3
5	2193, 1660, 1626, 1606, 1560, 1233, 1180	1.37 (3H, t, J = 7, $CH_2C\underline{H}_3$); 2.68 (6H, s, 10-CH_3+8-CH_3); 3.77 (3H, s, OCH_3); 4.28 (1H, s, H-4); 4.38 (2H, q, J = 7, $C\underline{H}_2CH_3$); 6.13 (1H, d, J = 13, H-6); 6.28 (1H, d, J = 13, H-5); 6.87 (2H, d, J = 8, H-3'+H-5'); 7.18 (2H, d, J = 8, H-2'+H-6'); 8.24 (1H, s, CH=N)
7	3393, 3153, 1653, 1606, 1540, 1260, 1193	2.61 (3H, s, 1-CH_3); 2.83 (3H, s, 3-CH_3); 3.68 (3H, s, OCH_3); 4.75 (1H, s, H-7); 6.12 (1H, d, J = 13, H-5); 6.57 (1H, d, J = 13, H-6); 6.82-6.90 (4H, m, NH_2+H-3'+H-5'); 7.30 (2H, d, J = 9, H-2'+H-6'), 8.12 (1H, s, H-10)

* The ^{1}H NMR spectra were recorded in DMSO-d_6 (compounds **2a**, **e-h**, **j**,**k**, **4a-c**, **7**) and $CDCl_3$ (compounds **2b-d**, **i**, **5**).

In the case of compound **2a** we were able to realize the synthesis of the corresponding annulated 4-aminopyrimidine in two stages. First, the corresponding ethoxymethylene derivative **5** was obtained by boiling the initial pyran **2a** in triethyl orthoformate for an hour. Its solution in alcohol was then treated with aqueous ammonia. After standing at room temperature for two days the aminopyrimidine **7** was isolated.

In the ^{1}H NMR spectrum of compound **7** there is a signal for the methine proton of the pyrimidine ring at 8.1 ppm and also a broad signal for the amino group at 6.9 ppm, partly overlapping with the signal of the protons of the aryl group. Further evidence for the cyclization that occurs is the absence of the absorption band of the C≡N bond in the region of ~2180-2193 cm^{-1}, which is present in the spectrum of the initial pyran **5**.



5-7 Ar = 4-$MeOC_6H_4$

Thus, 8-hydroxy-1,3-dimethyl-4H-cyclohepta[*c*]furan-4-one (**1**) exhibits high nucleophilic reactivity in reaction with ylidene malononitriles. These reactions lead to the formation of the expected respective condensed 2-amino-3-cyano-4H-pyrans, including spirocyclic compounds. The obtained pyrans can be converted into pyranopyrimidines in two stages.

EXPERIMENTAL

The IR spectra were recorded in vaseline oil on a Specord IR-71 spectrometer. The ^{1}H NMR spectra were recorded on Bruker DPX-250 (250 MHz) and Varian VXR-300 Unity (300 MHz) spectrometers in $CDCl_3$ and DMSO-d_6. The ^{13}C NMR spectra were recorded on a Varian VXR-300 Unity spectrometer (75.5 MHz) in DMSO-d_6 with TMS as internal standard.

The required 8-hydroxy-1,3-dimethyl-4H-cyclopenta[*c*]furan-4-one (**1**) was obtained by the previously developed method [11]. The ylidene malonitriles were prepared by the Knoevenagel reaction of malononitrile and the corresponding aldehyde or ketone according to the described procedures.

2-Amino-4-((het)aryl)-8,10-dimethyl-7-oxo-4H,7H-furo[3′,4′:6,7]cyclohepta[1,2-*b*]pyran-3-carbonitriles 2a-k and 2-Amino-8,10-dimethyl-2′,7-dioxo-1′,2′-dihydro-7H-spiro[furo[3′,4′:6,7]cyclohepta-[1,2-*b*]pyran-4,3′-indole]-3-carbonitriles 4a-c (General Method). To a suspension of the hydroxytropone **1** (1.9 g, 10 mmol) in acetonitrile with stirring we added the ylidene malononitrile (10 mmol) and triethylamine (0.1 ml), and we heated the mixture to 50-60°C. In most cases a precipitate of the product began to separate after 3-5 min. The reaction mixture was left at room temperature overnight. The product was filtered off, washed with acetonitrile, and recrystallized from a mixture of acetonitrile and DMF or from dichloroethane. The yields of the products amounted to 32-82%. The characteristics of the obtained compounds are given in Table 1. ^{13}C NMR spectrum for **2b**: 14.3, 14.7, 16.3, 42.6, 57.4, 63.0, 111.1, 111.8, 114.7 (2C); 119.5, 119.8, 128.8 (2C); 129.4, 136.7, 137.6, 146.3, 151.1, 156.5, 157.7, 158.1, 183.6. ^{13}C NMR spectrum for **4b**: 14.1, 16.5, 26.6, 51.7, 54.9, 107.4, 109.3, 111.5, 117.6, 119.5, 123.7, 124.8, 129.7, 130.2, 133.2, 133.9, 143.3, 148.1, 152.2, 156.7, 159.0, 176.7, 183.4.

2-Amino-4-(4-methoxyphenyl)-8,10-dimethyl-7-oxo-4H,7H-furo[3′,4′:6,7]cyclohepta[1,2-*b*]pyran-3-carbonitrile 2a (from hydroxytropone, aldehyde, and malononitrile). To a mixture of compound **1** (1.9 g, 10 mmol), 4-methoxybenzaldehyde (12 ml, 10 mmol), and malononitrile (0.66 g, 10 mmol) in acetonitrile with stirring was added triethylamine (0.1 ml), and we heated the mixture to 50-60°C. A precipitate of the product began to separate after 1-2 min. The reaction mixture was left at room temperature overnight. The product was filtered off, washed with acetonitrile, and crystallized twice from a mixture of acetonitrile and DMF. The yield was 62%.

2-(1-Ethoxymethyleneamino)-4-(4-methoxyphenyl)-8,10-dimethyl-7-oxo-4H,7H-furo[3′,4′:6,7]cyclohepta[1,2-*b*]pyran-3-carbonitrile (5). A suspension of the pyran **2a** (0.5 g, 1.3 mmol) was boiled in triethyl orthoformate (10 ml) for 1.5 h. The solution was evaporated to dryness. The residue was dissolved in chloroform and passed through a small column of aluminum oxide, eluted with chloroform, and evaporated. The residue was crystallized from a 1:1 mixture of toluene and petroleum ether.

8-Amino-7-(4-methoxyphenyl)-1,3-dimethyl-4H,7H-furo[3″,4″:3′,4′]cyclohepta[1′,2′:5,6]pyrano-[2,3-*d*]pyrimidin-4-one (7). A suspension of the pyran **2a** (0.5 g, 1.3 mmol) in triethyl orthoformate (10 ml) was boiled for 1.5 h and evaporated to dryness. The residue was dissolved in chloroform and passed through a column of aluminum oxide and eluted with chloroform. The eluate was evaporated. The residue was dissolved with gentle heat (40-50°C) in ethanol (15 ml), and aqueous ammonia (0.5 ml) was added. The flask was covered and kept at room temperature for two days. The precipitate was filtered off and purified by recrystallization from ethanol.

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