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REACTIONS OF 5-(HET)ARYL-1-ETHYL-2(1*H*)-PYRAZINONES WITH TERMINAL ARYLACETYLENES PROMOTED BY MICROWAVE RADIATION

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The reaction of 5-(het)aryl-1-ethyl-2(1H)-pyrazinones with terminal arylacetylenes, leading to a mixture of two isomeric 4-aryl- and 5-aryl-substituted 2(1H)-pyridones has been investigated. The regioselectivity of this reaction has been shown on the basis of reaction mixtures study by chromato-mass spectrometry. A crystallographic investigation of the synthesized 2(1H)-pyridones and also a forecast of their potential biological activity have been carried out.

Keywords: σ^H-adducts, arylacetylenes, 1,2-dihydropyrazines, ionic liquid, 2(1H)-pyrazinones, 2(1H)-pyridones, microwave radiation.

The readily available and widely functionalized 2-azadiene system of 2(1H)-pyrazinones allow unique possibilities for intra- and intermolecular cycloaddition reactions with both electron-rich and electron-deficient dienophiles [1–3]. However the described reactions of pyrazinones **1** with acetylenes are restricted to several examples of intramolecular cycloaddition reactions when alkyne is one of the substituents in the pyrazinone fragment, and also to the interaction with the dimethyl ester of acetylenedicarboxylic acid **2** [3–5].

These Diels-Alder reactions usually lead to a mixture of pyridone **4** and pyridine **5** adducts due to two competing spontaneous routes of the initially formed bicyclic cycloadducts **3** fragmentation by a retro Diels–Alder reaction. Depending on the substituents R and R^1, the ratio of compounds **4** and **5** vary significantly. When R = Ph, R^1 = Cl the main product of the reaction is pyridine **5**, but at R = Et, R^1 = (Het)Ar pyridone **4** predominates.

The aim of the present study was the investigation of the cycloaddition reaction of 5-(het)aryl-1-ethyl-2(1H)-pyrazinones with terminal acetylenes on microwave irradiation in comparison to the usual thermal con-

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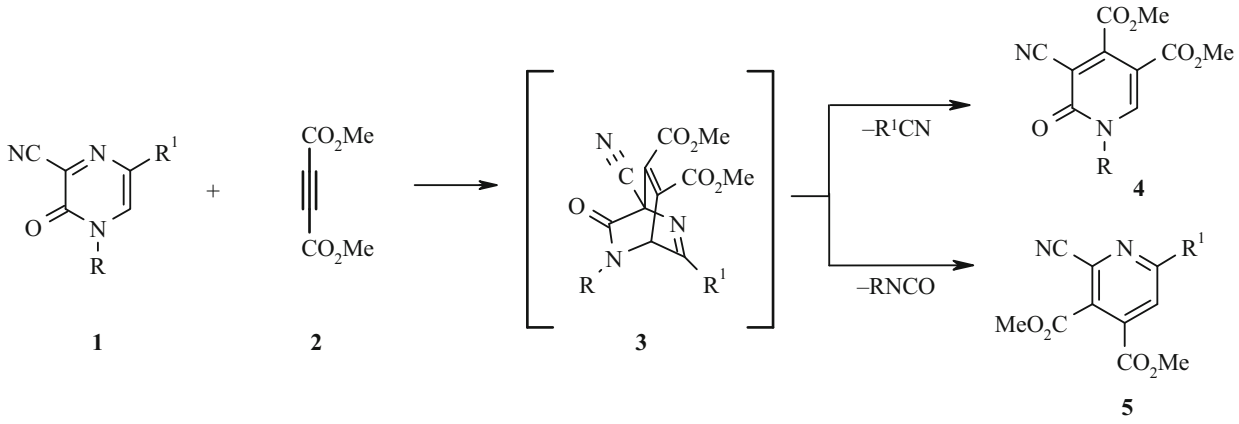


ditions. The commercially available phenylacetylene (**7**), 4-bromophenylacetylene (**8**), and 4-dimethylaminophenylacetylene (**9**) were used as alkynes. Reactions of pyrazinones **6a,b** with terminal arylacetylenes **7–9** proceeded, according to data of both chromato-mass spectrometry and ^{1}H NMR spectrometry, regioselectively and led to a mixture of only two regioisomeric 2(1H)-pyridones, the major were 4-aryl-1-ethyl-2-oxo-1,2-dihydropyridine-3-carbonitriles **12, 14**, and **16**, and the minor were 5-aryl-1-ethyl-2-oxo-1,2-dihydropyridine-3-carbonitriles **13** and **15**.

[4+2] Cycloaddition reactions were carried out under conditions of microwave irradiation in the presence of the ionic liquid bmimPF_6. The experimentally found (by TLC reaction progress control) optimum time for carrying out these reactions was 90 min at 195°C. For comparison the same reactions were carried out under thermal conditions on boiling (178–180°C, 90 min) in 1,2-dichlorobenzene in the absence of any additives (Table 1).

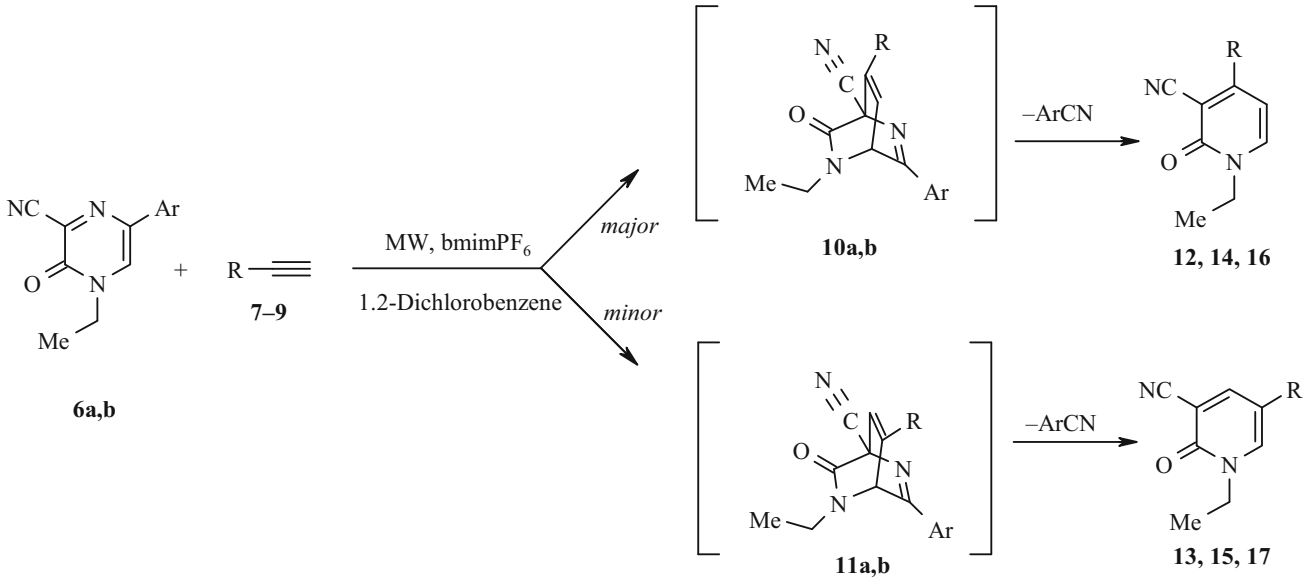
The obtained results indicate the absolute advantage of microwave syntheses. At one and the same time in reactions proceeding under the usual thermal conditions, incomplete conversion of the initial 2(1H)-pyrazinones **6a,b** and the formation of resinous products was observed. When promoted by microwave radiation, reactions proceed with complete conversion of the starting compounds and the formation of a mixture of the regioisomerically substituted 2(1H)-pyridones **12–16**.

TABLE 1. Yields and Ratios of 2(1H)-Pyridines **12–17**

Reaction	Conversion of compound **6a** or **6b**, %*		Product ratio*		Yield, %	
	MW	Boiling	MW	Boiling	Compound	MW
6a+7	94	39	–*[2]	–*[2]	**12** **13**	55 7
6b+7	82	28	–*[2]	–*[2]	**12** **13**	68 11
6a+8	100	55	**14** : **15** 6.1 : 1	**14** : **15** 6.9 : 1	**14** **15**	60 4
6b+8	100	32	**14** : **15** 7.3 : 1	**14** : **15** 5.4 : 1	**14** **15**	–
6a+9	100	100	**16** : **17** 1 : 0	**16** : **17** 1 : 0	**16**	98
6b+9	100	95	**16** : **17** 1 : 0	**16** : **17** 1 : 0	**16**	–

*According to GLC-MS data.

*[2]Ratio not established due to closeness of retention time in GLC-MS data and overlap of signals in ^{1}H NMR spectra of reaction mixtures.



6a Ar = Ph, **b** Ar = 3-Thienyl;

7, 12, 13 R = Ph; **8, 14, 15** R = 4-BrC_6H_4; **9, 16, 17** R = 4-$(Me_2N)C_6H_4$

The structures of the obtained 2(1H)-pyridones **12** and **14–16** were established by X-ray structural analysis (XSA). Data of XSA agree well with the data of ^{1}H NMR spectra in which $J_{5,6}$ = 7.0–7.2 for 4-aryl-1-ethyl-2-oxo-1,2-dihydropyridine-3-carbonitriles **12, 14**, and **16**, while $J_{4,6}$ = 2.6–2.7 Hz for 5-aryl-1-ethyl-2-oxo-1,2-dihydropyridine-3-carbonitriles **13** and **15**.

According to the data of XSA the investigated compounds are crystallized in centrosymmetric space groups (compounds **12**, **14**, and **16** are monoclinic, **15** is orthorhombic). The distribution of bond lengths and valence angles is fairly typical for such a series of compounds. The distribution of bond lengths in the heterocycle indicates clearly the formation of a conjugated dienone system.

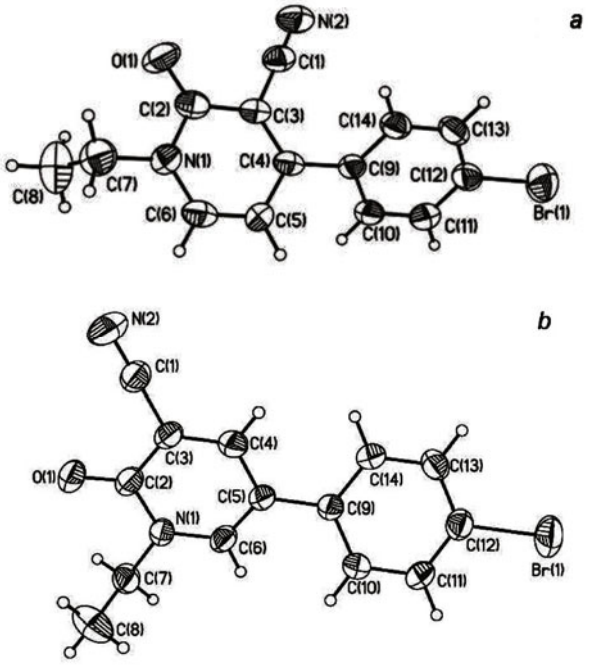


Fig. 1. Geometry of the compounds **14** (*a*) and **15** (*b*) molecules in the crystal.

TABLE 2. Main Crystallographic Parameters and Results of the Refinement of Structural Experiments

Compound	**12**	**14**	**15**	**16**
Empirical formula	$C_{14}H_{12}N_2O$	$C_{14}H_{11}BrN_2O$	$C_{14}H_{11}BrN_2O$	$C_{16}H_{17}N_3O$
M	224.26	303.16	303.16	267.33
T, K	295(2)	295(2)	295(2)	295(2)
System	Monoclinic	Orthorhombic	Monoclinic	Monoclinic
Space group	$P2_1/n$	$Pbca$	$P2_1/n$	$P2_1/c$
Parameters of unit cell				
a, Å	7.8184(8)	13.0840(6)	9.5290(4)	13.9250(8)
d, Å	13.1383(16)	13.9098(13)	11.3804(11)	10.9721(6)
c, Å	11.7049(14)	14.3560(14)	12.1323(14)	9.4267(5)
β, deg	100.244(10)	90	101.047(7)	98.061(5)
V, Å^3 Z	1183.2(2) 4	2612.7(4) 8	1291.3(2) 4	1426.04(14) 4
$D_{calc.}$, g/cm^3	1.259	1.541	1.559	1.245
μ, mm^{-1}	0.081	3.135	3.172	0.080
F(000)	472	1216	608	568
T_{min} T_{max}	– –	0.403 0.546	0.439 0.729	– –
Scanning angle, Θ, deg	3.07 < Θ < 28.28	3.11 < Θ < 28.28	2.82 < Θ < 28.28	2.87 < Θ < 28.28
Reflections total	5509	8893	10151	8779
Independent reflections	2839 (R_{int} = 0.0177)	3070 (R_{int} = 0.0369)	3184 (R_{int} = 0.0332)	3495 (R_{int} = 0.0259)
Reflections with [I>2σ(I)]	1511	1207	1586	1669
Complexity, %, for Θ 26.00°	97.9	97.1	99.8	99.0
S on F^2	1.010	1.010	1.010	1.008
R_1 [I>2σ(I)]	0.0354	0.0389	0.0303	0.0408
wR_2[I>2σ(I)]	0.0733	0.0594	0.0505	0.0843
R_1 (all data)	0.0877	0.1233	0.0843	0.0967
wR_2 (all data)	0.0933	0.0627	0.0531	0.0903
$\Delta\rho_{min}$, e·Å^{-3} $\Delta\rho_{max}$, e·Å^{-3}	0.127 –0.123	0.555 –0.655	0.440 –0.379	0.193 –0.168

The length of the C=O bond in the investigated series lies within 1.222–1.236 Å, the length (provisionally) of the double bonds varies from 1.343 to 1.383 Å. Provisionally the single bonds vary from 1.405 to 1.449 Å. The aromatic substituent is turned relative to the plane of the pyridine ring. The respective dihedral angles were 43.5(2)° (compound **12**), 48.3(2)° (compound **14**), 28.2(2)° (compound **15**), and 36.7(2)° (compound **16**). As we see, for compounds **12, 14**, and **16** the angle of turn is reduced according to the increase in electron-donating properties of the aromatic substituent, which is probably caused by the conjugation increase in the rings π-systems due to the transfer of electrons from the π-donor to the π-accepting pyridine ring. In the case of compound **15** the reduction in the angle of turn of the substituent may be explained by the reduction of CN group steric factor influence.

A special feature of the molecular packing of the investigated compounds is the formation of shortened contacts between the carbonyl oxygen atom and the proton at C(6) of the pyridine ring: d{O(1)···H(6A)[–1/2+x, y, 1.5–z]} = 2.500 (compound **14**), d{O(1)···H(6A)[1.5–x, –1/2+y, 2.5–z]} = 2.431 (compound **15**),

TABLE 3. Calculations Results of the Potential Biological Activity for 2(1*H*)-Pyrazinones **6a,b** and 2(1*H*)-Pyridones **12–16** ($P_a > 70\%$)

Compound							P_a	P_i	Activity
6a	**6b**	**12**	**13**	**14**	**15**	**16**			
1	2	3	4	5	6	7	8	9	10
+	–	+	+	–	–	–	0.871 (**6a**) 0.832 (**12**, **13**)	0.004 0.005	Glutathione thiolesterase inhibitor
+	–	+	+	–	–	–	0.847 (**6a**) 0.790 (**12**, **13**)	0.003 0.004	Ferredoxin-NAD^+ reductase inhibitor
+	–	+	+	–	–	–	0.847 (**6a**) 0.790 (**12**, **13**)	0.003 0.004	Naphthalene 1,2-dioxygenase inhibitor
+	–	–	–	–	–	–	0.836	0.012	Glutaminyl-peptide cyclotransferase inhibitor
+	–	+	+	–	–	–	0.826 (**6a**) 0.761 (**12**, **13**)	0.003 0.004	Methanol dehydrogenase inhibitor
+	–	+	+	–	–	–	0.820 (**6a**) 0.753 (**12**, **13**)	0.004 0.007	Gamma-guanidinobutyraldehyde dehydrogenase inhibitor
+	–	+	+	–	–	–	0.814 (**6a**) 0.752 (**12**, **13**)	0.004 0.005	Tryptophanamidase inhibitor
+	+	+	+	+	+	+	0.805 (**6a**) 0.762 (**6b**) 0.872 (**12**, **13**) 0.782 (**14**, **15**) 0.759 (**16**)	0.006 0.010 0.005 0.008 0.010	Proteasome ATPase inhibitor
+	–	+	+	–	–	–	0.794 (**6a**) 0.807 (**12**, **13**)	0.004 0.004	Chenodeoxycholoyltaurine hydrolase inhibitor
+	–	–	–	–	–	–	0.794	0.004	Arylalkyl acylamidase inhibitor
+	–	+	–	–	–	–	0.785 (**6a**) 0.702 (**12**)	0.010 0.018	2-Chlorobenzoate 1,2-dioxygenase inhibitor
+	–	–	–	–	–	–	0.775	0.004	4-Chlorophenylacetate 3,4-dioxygenase inhibitor
+	–	–	–	–	–	–	0.774	0.005	Amine dehydrogenase inhibitor
+	–	+	+	–	–	–	0.772 (**6a**) 0.793 (**12**, **13**)	0.007 0.005	Electron-transferring-flavoprotein dehydrogenase inhibitor
+	–	–	–	–	–	–	0.771	0.009	2-Hydroxyquinoline 8-monooxygenase inhibitor
+	+	+	+	–	–	–	0.774 (**6a**) 0.775 (**6b**) 0.786 (**12**, **13**)	0.014 0.014 0.013	Muramoyltetrapeptide carboxypeptidase inhibitor
+	–	–	–	–	–	–	0.749	0.010	Mitochondrial processing peptidase inhibitor
+	–	+	–	–	–	–	0.745 (**6a**) 0.756 (**12**)	0.006 0.006	Allyl-alcohol dehydrogenase inhibitor

1	2	3	4	5	6	7	8	9	10
+	–	–	–	–	–	–	0.740	0.011	Formaldehyde transketolase inhibitor
+	–	–	–	–	–	–	0.719	0.007	Aldehyde dehydrogenase (pyrroloquinoline-quinone) inhibitor
+	–	–	–	–	–	–	0.716	0.009	Sulfite reductase inhibitor
+	–	–	–	–	–	–	0.705	0.005	Nicotinate dehydrogenase inhibitor
+	–	–	–	–	–	–	0.708	0.029	Flavonoid 3'-monooxygenase inhibitor
–	–	+	+	–	–	–	0.742 (**12**, **13**)	0.019	Ribulose-phosphate 3-epimerase inhibitor
–	–	+	+	–	–	–	0.716 (**12**, **13**)	0.017	Phosphatidylcholine-retinol O-acyltransferase inhibitor
–	–	+	+	–	–	–	0.714 (**12**, **13**)	0.020	UDP-N-acetylglucosamine 4-epimerase inhibitor
–	–	+	–	–	–	–	0.706	0.013	G-Quadruplex telomerase inhibitor
–	–	+	+	–	–	–	0.741 (**12**, **13**)	0.061	CYP2C12 substrate
–	–	–	–	+	+	–	0.754 (**14**, **15**)	0.012	Prolyl aminopeptidase inhibitor
–	–	–	–	–	–	+	0.796	0.004	Magnesium protoporphyrin IX monomethyl ester (oxidative) cyclase inhibitor
–	–	–	–	–	–	+	0.711	0.004	Camphor 1,2-monooxygenase inhibitor

d{O(1)···H(6)[$-x$, 1/2+y, ½–z]} = 2.25(2) Å (compound **16**). The values of the contacts (particularly for compound **16**) indicate the possibility of generating specific interactions since they being comparable with an intermolecular hydrogen bond. In spite of the simplicity of the geometry and the presence of a powerful π-system, clearly expressed π-contacts were absent.

The forecast of the biological activity for the synthesized 2(1H)-pyridones **12–16,** and their precursors 2(1H)-pyrazinones **6a,b,** has been made in the present work basing on the substance structural formula. It was carried out by the computer program PASS using the available interactive service PASS INet (http://www.ibmc.msk.ru/PASS) [6–10]. Biological activity is described in PASS in a qualitative form (active/inactive) as a list of activities with two probabilities: $\mathbf{P}_a$, to be active and $\mathbf{P}_i$, to be inactive, calculated for each activity kind. This list is drawn up according to the decrease of the $\mathbf{P}_a$–$\mathbf{P}_i$ differences (Table 3). Only activities with $\mathbf{P}_a > \mathbf{P}_i$ are considered as probable for a compound being analyzed.

Data on the forecasting of properties for (het)aryl-substituted pyrazinones and pyridines (Table 3) show that they may display high biological activity over a wide spectrum. This suggests the expediency of experimental confirmation of biological activity for this class compounds with the aim of searching for new prospects of physiologically active substances.

A simple and effective way for obtaining 4-aryl- and 5-aryl-substituted 2(1H)-pyridones from reactions of 5-(het)aryl-1-ethyl-2(1H)-pyrazinones with terminal arylacetylenes promoted by microwave irradiation has been proposed. Crystallographic investigation of the synthesized 2(1H)-pyridones was carried out and a forecast of their potential biological activity was made.

EXPERIMENTAL

The ^{1}H NMR spectra were obtained on a Bruker DRX 400 instrument (at 400 MHz) in $CDCl_3$ solution, internal standard was TMS. Elemental analysis was carried out on an automatic Perkin–Elmer PE 2400 analyzer. Melting points were determined on a combination Boetius stage and are not corrected. Preparative HPLC was carried out using a semi-preparative Agilent 1200 Series liquid chromatograph with a diode matrix detector, analytical wavelength 280 nm. A ZORBAX Eclipse XDB-C18 PrepHT column 21.2 mm × 150 mm, particle size 5 μm was used at room temperature. Mobile phase was a mixture of acetonitrile–water, 60:40 and 50:50, flow rate 20 ml/min, elution was isocratic.

GLC–MS analysis of all samples was carried out using a gas chromatograph-mass spectrometer Agilent GC 7890A MS 5975C Inert XL EI/CI with a quadrupole mass-spectrometric detector and a quartz capillary column HP 5MS, 30 m × 0.25 mm, film thickness 0.25 μm. The mass spectra were recorded by electron ionization (70 eV) scanning along the complete ion current in the range m/z 20–1000. Carrier gas was helium, flow division 1:50, supply through the column 1.0 ml/min. Column temperature was 40°C initially (retention 3 min), programed at 10°C/min to 290°C (retention 20 min), evaporator temperature 250°C, source temperature 230°C, quadrupole 150°C, transfer chamber 280°C. Solutions of samples of concentration 3–4 mg/ml were prepared in acetonitrile.

X-ray structural analysis (XSA) was carried out on an Xcalibur-3 X-ray diffractometer with a CCD detector by the standard procedure (λMoKα, graphite monochromator, ω-scanning). For analysis fragments of yellow crystals were used of size 0.26×0.14×0.11 (compound **12**), 0.32×0.19×0.07 (compound **14**), 0.16×0.09×0.06 (compound **15**), and 0.25×0.20×0.15 mm (compound **16**). Collection and processing of data was carried out using the CrysAlis set of programs [12]. No correction for absorption was introduced for samples of **12** and **16**, for samples of **14** and **15** a correction was introduced analytically according to the model for a polyhedral crystal [12]. The structures of all compounds were solved by the direct method according to program SHELXS-97 and were refined with the aid of the SHELXL-97 program [13] in an anisotropic approximation (isotropic for hydrogen atoms). The hydrogen atoms were partially determined and refined independently, and partially included in the refinement in the rider model with dependent thermal parameters. The XSA data are recorded in the Cambridge Crystallographic Data Center at www.ccdc.cam.ac. uk/data_request/cif (deposition

number CCDC 819354 for compound **12**, CCDC 819355 for compound **14**, CCDC 819356 for compound **15**, and CCDC 819357 for compound **16**).

All microwave experiments were carried out in a single mode CEM Discover system with an operating frequency of 2.45 GHz. The reactions were carried out in a test tube of 10 ml capacity with a hermetic Teflon stopper.

Column chromatography was carried out using Lancaster silica gel 0.040–0.063 mm (230–400 mesh), eluting with ethyl acetate–hexane, 1 : 2.

The progress of reactions and the purity of compounds was checked by TLC on Sorbfil plates, visualizing with UV light.

Solvents were dried and purified according to procedures taken from literature [11]. The starting compounds **6a,b** were obtained according to the known procedure of [5].

4-Aryl-1-ethyl-2-oxo-1,2-dihydropyridine-3-carbonitriles 12, 14, 16, and **5-Aryl-1-ethyl-2-oxo-1,2-dihydropyridine-3-carbonitriles 13, 15 (General Method).** A. Use of microwave radiation. A solution of the corresponding 2(1H)-pyrazinone **6a,b** (0.60 mmol), arylacetylene (0.72 mmol), and bmimPF_6 (1-butyl-3-methylimidazolium hexafluorophosphate) (15 μl, 0.15 mmol) in 1,2-dichlorobenzene (3 ml) was irradiated for 90 min with microwave radiation (250 W) at 195°C, the solvent was distilled at reduced pressure, and the residue was separated chromatographically on silica gel or isolated by preparative HPLC.

B. Reaction under thermal conditions. A solution of the corresponding 2(1H)-pyrazinone **6a,b** (0.60 mol) and arylacetylene (0.72 mmol) in 1,2-dichlorobenzene (3 ml) was boiled for 90 min. The reaction mixture was treated analogously to method A.

When investigating the reaction mixtures, the residue after distillation of the solvent was analyzed by GC–MS without further treatment.

1-Ethyl-2-oxo-4-phenyl-1,2-dihydropyridine-3-carbonitrile (12). Light-yellow crystalline powder; mp 148–150°C. ^{1}H NMR spectrum, δ, ppm (*J*, Hz): 1.43 (3H, t, *J* = 7.2, CH_3); 4.01–4.11 (2H, m, CH_2); 6.35 (1H, d, *J* = 7.0, H-6); 7.39–7.53 (3H, m, H Ph); 7.55 (1H, d, *J* = 7.0, H-5); 7.60–7.63 (2H, m, H Ph). Mass spectrum, *m/z* (I_{rel}, %): 224 $[M]^+$ (100). HPLC: t_R 2.8–3.6 min. GLC: t_R 25.54 min. Found, %: C 74.97; H 5.48; N 12.51. $C_{14}H_{12}N_2O$. Calculated, %: C 74.98; H 5.39; N 12.49.

1-Ethyl-2-oxo-5-phenyl-1,2-dihydropyridine-3-carbonitrile (13). Yellow crystalline powder; mp 135–137°C. ^{1}H NMR spectrum, δ, ppm (*J*, Hz): 1.45 (3H, t, *J* = 7.2, CH_3); 4.13 (2H, q, *J* = 7.2, NCH_2); 7.26–7.41 (3H, m, H Ph); 7.44–7.48 (2H, m, H Ph); 7.74 (1H, d, *J* = 2.7, H-6); 8.07 (1H, d, *J* = 2.7, H-4). Mass spectrum, (EI, 70 eV), *m/z* (I_{rel}, %): 224 $[M]^+$ (100). HPLC: t_R 3.7–4.6 min. GLC: t_R 25.54 min. Found, %: C 75.12; H 5.23; N 12.50. $C_{14}H_{12}N_2O$. Calculated, %: C 74.98; H 5.39; N 12.49.

4-(4-Bromophenyl)-1-ethyl-2-oxo-1,2-dihydropyridine-3-carbonitrile (14). Light-yellow crystalline powder; mp 196–198°C. ^{1}H NMR spectrum, δ, ppm (*J*, Hz): 1.43 (3H, t, *J* = 7.2, CH_3); 4.12 (2H, q, *J* = 7.2, CH_2); 6.31 (1H, d, *J* = 7.0, H-6); 7.49 (2H, d, *J* = 8.4, H-3',5'); 7.55 (1H, d, *J* = 7.0, H-5); 7.65 (2H, d, *J* = 8.4, H-2',6'). Mass spectrum, *m/z* (I_{rel}, %): 302 $[M]^+$ (100) for ^{79}Br, 304 $[M]^+$ (100) for ^{81}Br. HPLC: t_R 2.8–3.6 min. GLC: t_R 28.13 min. Found, %: C 55.69; H 3.75; N 9.11. $C_{14}H_{11}BrN_2O$. Calculated, %: C 55.47; H 3.66; N 9.24.

5-(4-Bromophenyl)-1-ethyl-2-oxo-1,2-dihydropyridine-3-carbonitrile (15). Light-yellow crystalline powder; mp 193–195°C. ^{1}H NMR spectrum, δ, ppm (*J*, Hz): 1.45 (3H, t, *J* = 7.2, CH_3); 4.12 (2H, q, *J* = 7.2, CH_2); 7.25 (2H, d, *J* = 8.5, H-3',5'); 7.59 (2H, d, *J* = 8.5, H-2',6'); 7.72 (1H, d, *J* = 2.6, H-6); 8.02 (1H, d, *J* = 2.6, H-4). Mass spectrum, *m/z* (I_{rel} %): 302 $[M]^+$ (100) for ^{79}Br, 304 $[M]^+$ (100) for ^{81}Br. HPLC: t_R 3.8–4.8 min. GLC: t_R 28.32 min. Found, %: C 55.59; H 3.37; N 9.18. $C_{14}H_{11}BrN_2O$. Calculated, %: C 55.47; H 3.66; N 9.24.

4-(4-Dimethylaminophenyl)-1-ethyl-2-oxo-1,2-dihydropyridine-3-carbonitrile (16). Bright-yellow crystalline powder; mp 187–189°C. ^{1}H NMR spectrum, δ, ppm (*J*, Hz): 1.40 (3H, t, *J* = 7.2, $CH_2C\underline{H}_3$); 3.05 (6H, s, $N(CH_3)_2$); 4.03 (2H, q, *J* = 7.2, $C\underline{H}_2CH_3$); 6.35 (1H, d, *J* = 7.2, H-6); 6.76 (2H, dd, *J* = 6.9, *J* = 2.2, H-3',5'); 7.42 (1H, d, *J* = 7.2, H-5); 7.62 (2H, d, *J* = 6.9, *J* = 2.2, H-2',6'). Mass spectrum, *m/z* (I_{rel}, %): 267 $[M]^+$ (100). HPLC: t_R 2.0–2.5 min. GLC: t_R 30.79 min. Found, %: C 71.81; H 6.48; N 15.53. $C_{16}H_{17}N_3O$. Calculated, %: C 71.89; H 6.41; N 15.72.

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