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CONDENSED ISOQUINOLINES. 37.* HETEROCYCLIZATION USING 3-(ARYLAMINO)- AND 3-(HETARYLAMINO)ISOQUINOLIN-1(2H)-ONES

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The reaction of 3-NHR-isoquinolin-1(2H)-ones (R = Ar) with aromatic aldehydes in the presence of Me_3SiCl or in acetic acid leads to the formation of derivatives of dibenzo[b,f][1,8]naphthyridin-5(6H)-one and benzo[f]isoquino[3,4-b][1,8]naphthyridine-5,9(6H,7H)-dione. The reaction for R = Het in the presence of Me_3SiCl gives derivatives of 5H-pyrido[1',2':1,2]pyrimido[4,5-c]isoquinolin-5-one, benzo[f]isoquinoline[3,4-b][1,8]naphthyridine-5,9[6H,7H]-dione, and derivatives of new heterocyclic systems, 5H-pyrazino[1',2':1,2]pyrimido[4,5-c]isoquinolin-5-one, 5H-[1,3]thiazolo[3',2':1,2]pyrimido-[4,5-c]isoquinolin-5-one, 5-H-benzo[f]pyrazolo[3,4-b][1,8]naphthyridin-5-one, and isoquino[3,4-b]-[1,5]naphthyridin-5(6H)-one. The effect of the structure of substituent R and nature of the substituent in the benzaldehydes on the structure of the reaction products was studied.

Keywords: 3-aminoisocarbostyryl, benzo[*f*]isoquino[3,4-*b*][1,8]naphthyridine, benzo[*f*]pyrazolo[3,4-*b*]-[1,8]naphthyridine, dibenzo[*b,f*][1,8]naphthyridine, isoquino[3,4-*b*][1,5]naphthyridine, pyrazino[1',2':1,2]-pyrimido[4,5-*c*]isoquinoline, pyrido[1',2':1,2]pyrimido[4,5-*c*]isoquinoline, [1,3]thiazolo[3',2':1,2]pyrimido-[4,5-*c*]isoquinoline.

Cyclization starting from enamines is one of the approaches to the synthesis of nitrogen heterocycles [2]. The enamine properties of some derivatives of 3-aminoisoquinolin-1(2H)-one (3-aminoisocarbostyryl) have also been used for heterocyclization [3]. The fusion of a pyrrole or pyridine ring to face *c* in these cases has been carried out predominantly by intramolecular acylation or alkylation. In some studies of the properties of 3-amino-2-methyl- and 3-aminoisoquinolin-1(2H)-ones, Ohta [4, 5] and Deady [6] have described the formation of condensed isoquinolines in reactions with aldehydes or ketones. In the present work, we studied the reaction of 3-(arylamino)- and 3-(hetarylamino)isoquinolin-1(2H)-ones with aromatic aldehydes.

*For Communication 36, see [1].

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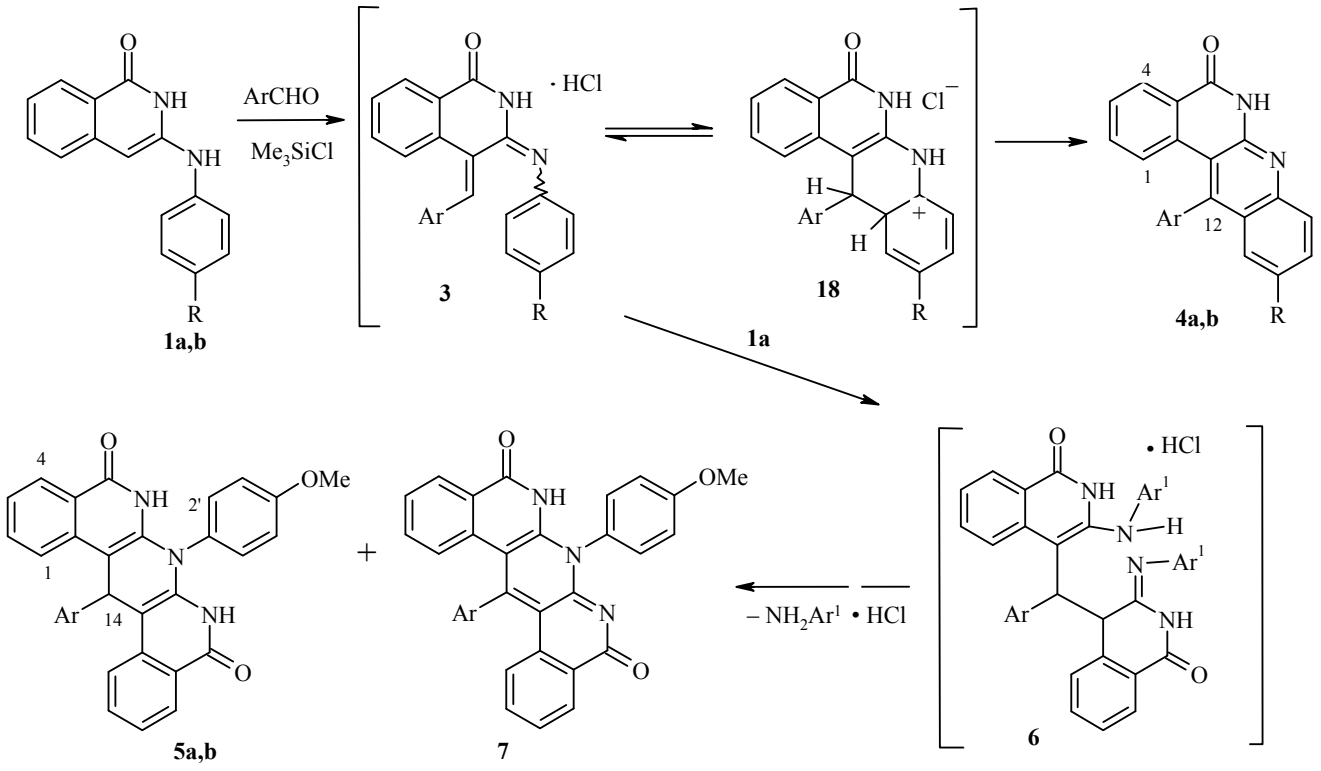
The condensation of 3-aminoisocarbostyryls substituted at the 3-amino group with aldehydes has been studied previously only in the case of a condensed derivative, namely, benzimidazo[1,2-*b*]isoquinolin-11(5H)-one [7, 8]. The corresponding alkylidene and arylidene derivatives were obtained by heating a mixture of the reagents in DMF or in acetic acid in the presence of a base.

We have established that heating 3-ArNH-isoquinolin-1(2H)-ones **1a** and **1b** (Scheme 1) and 3-HetNH-isoquinolin-1(2H)-ones **2a-f** (Schemes 2 and 3) with benzaldehydes in DMF under similar conditions does not yield significant amounts of reaction product. Attempts to obtain condensation products of compounds **1a,b** and **2a-f** in 2-propanol in the presence of piperidine were also unsuccessful, while carrying out this reaction in acetic anhydride led predominantly to acylation [9].

Scheme 1



1a R = OMe, **b** R = NO_2; **4 a**, **b** Ar = 4-$EtOC_6H_4$, **a** R = OMe, **b** R = NO_2;
5 a Ar = 4-$EtOC_6H_4$, **b** Ar = 4-$O_2NC_6H_4$; **7** Ar = 4-$O_2NC_6H_4$

Isoquinolones **2a-f** also proved inert when the reaction was carried out in acetic acid (Schemes 2 and 3). Only heating 3-ArNH-isocarbostyryls **1a** and **1b** with benzaldehyde in acetic acid (Method A) gave reaction products (Scheme 1). Thus, the reaction of compound **1a** with 4-methoxybenzaldehyde gave 12-(4-ethoxyphenyl-10-methoxydibenzo[*b*,*f*][1,8]naphthyridin-5(6H)-one (**4a**) in high (78%) yield (Table 1). The 4-arylidene derivative of the type **3** formed in the initial step of this reaction converts to compound **4a** as the result of intramolecular Friedel-Crafts type alkylation and subsequent oxidation of the electrophilic substitution product. Changes in the spin system of the 4-methoxyaniline fragment in the starting 3-aminoisocarbostyryl [9] in addition to the GC/MS and elemental analysis data indicated formation of products of aromatic electrophilic substitution. The ^{1}H NMR spectrum of dibenzo[*b*,*f*][1,8]naphthyridine **4a** shows three aromatic proton signals from a 1,2,4-trisubstituted benzene ring (Table 2). The condensation of isocarbostyryl **1a** with 4-nitrobenzaldehyde under the same conditions leads to the formation of a mixture of products, of which the major

TABLE 1. Physicochemical Properties of Compounds Synthesized

Compound	Empirical formula	Found, % / Calculated, %			mp, °C (solvent)	Yield, % (method)
		C	H	N		
4a	$C_{25}H_{20}N_2O_3$	74.69 / 75.74	5.10 / 5.08	7.09 / 7.07	289-290 (EtOH)	78 (A) 64 (B)
4b	$C_{24}H_{17}N_3O_4$	69.98 / 70.07	4.12 / 4.16	10.23 / 10.21	>300 (DMF)	60 (B)
5a	$C_{34}H_{27}N_3O_4$	75.43 / 75.40	4.98 / 5.02	7.80 / 7.76	>300 (MeCN-EtOH)	24 (B)
5b	$C_{32}H_{22}N_4O_5$	70.78 / 70.84	4.15 / 4.09	10.31 / 10.33	>300 (DMF)	28 (A) 45 (B)
7	$C_{32}H_{20}N_4O_5$	70.92 / 71.11	3.85 / 3.73	10.32 / 10.37	>300 (EtOH)	14 (B)
8a*	$C_{21}H_{16}ClN_3O$	69.68 / 69.71	4.50 / 4.46	11.60 / 11.61	>300 (DMF)	78 (B)
8b*	$C_{22}H_{18}ClN_3O_2$	67.38 / 67.43	4.59 / 4.63	10.74 / 10.72	>300 (DMF)	80 (B)
8c*	$C_{23}H_{20}ClN_3O_2$	68.02 / 68.06	5.00 / 4.97	10.38 / 10.35	>300 (DMF)	80 (B)
9a	$C_{21}H_{15}N_3O$	77.48 / 77.52	4.60 / 4.65	12.93 / 12.91	296-298 (EtOH)	72 (C)
9b	$C_{22}H_{17}N_3O_2$	74.39 / 74.35	4.80 / 4.82	11.87 / 11.82	247-248 (EtOH)	75 (C)
9c	$C_{23}H_{19}N_3O_2$	74.73 / 74.78	5.15 / 5.18	11.40 / 11.37	212-214 (EtOH)	70 (C)
9d	$C_{21}H_{14}N_4O_3$	68.15 / 68.10	3.78 / 3.81	15.15 / 15.13	183-184 (DMF)	68 (C)
10	$C_{21}H_{16}N_4O_2$	70.71 / 70.77	4.49 / 4.53	15.71 / 15.72	180-181 (*i*-PrOH)	73 (C)
11a*[2]	$C_{21}H_{17}N_3O_2S$	67.14 / 67.18	4.60 / 4.56	11.21 / 11.19	178-180 (EtOH)	83 (C)
11b*[2]	$C_{19}H_{12}N_4O_3S$	60.57 / 60.63	3.25 / 3.21	14.90 / 14.89	206-208 (EtOH)	30 (C)
13*[3]	$C_{19}H_{11}ClN_4O_3S$	55.50 / 55.55	2.73 / 2.70	13.66 / 13.64	>300 (EtOH)	72 (B)
14	$C_{25}H_{17}N_3O_2$	76.67 / 76.71	4.40 / 4.38	10.72 / 10.74	>300 (DMF)	44 (C)
15a	$C_{23}H_{22}N_4O_2$	71.50 / 71.48	5.71 / 5.74	14.52 / 14.50	249-251 (EtOH)	73 (B)
16a	$C_{23}H_{20}N_4O_2$	71.82 / 71.86	5.21 / 5.24	14.58 / 14.57	284-285 (DMF)	71 (B)
16b	$C_{21}H_{15}N_5O_3$	65.39 / 65.45	3.90 / 3.92	18.18 / 18.17	>300 (DMF)	48 (B)
17	$C_{23}H_{19}N_3O_2$	74.72 / 74.78	5.20 / 5.18	11.36 / 11.37	218-220 (dec.) (EtOH)	76 (B)

*Analysis data for Cl, found/calculated, %: **8a** – 9.83/9.80, **8b** –8.57/9.05 (dec.), **8c** – 8.72/8.73.

*[2]Analysis data for S, found/calculated, %: **11a** – 8.58/8.54, **11b** – 8.57/8.52.

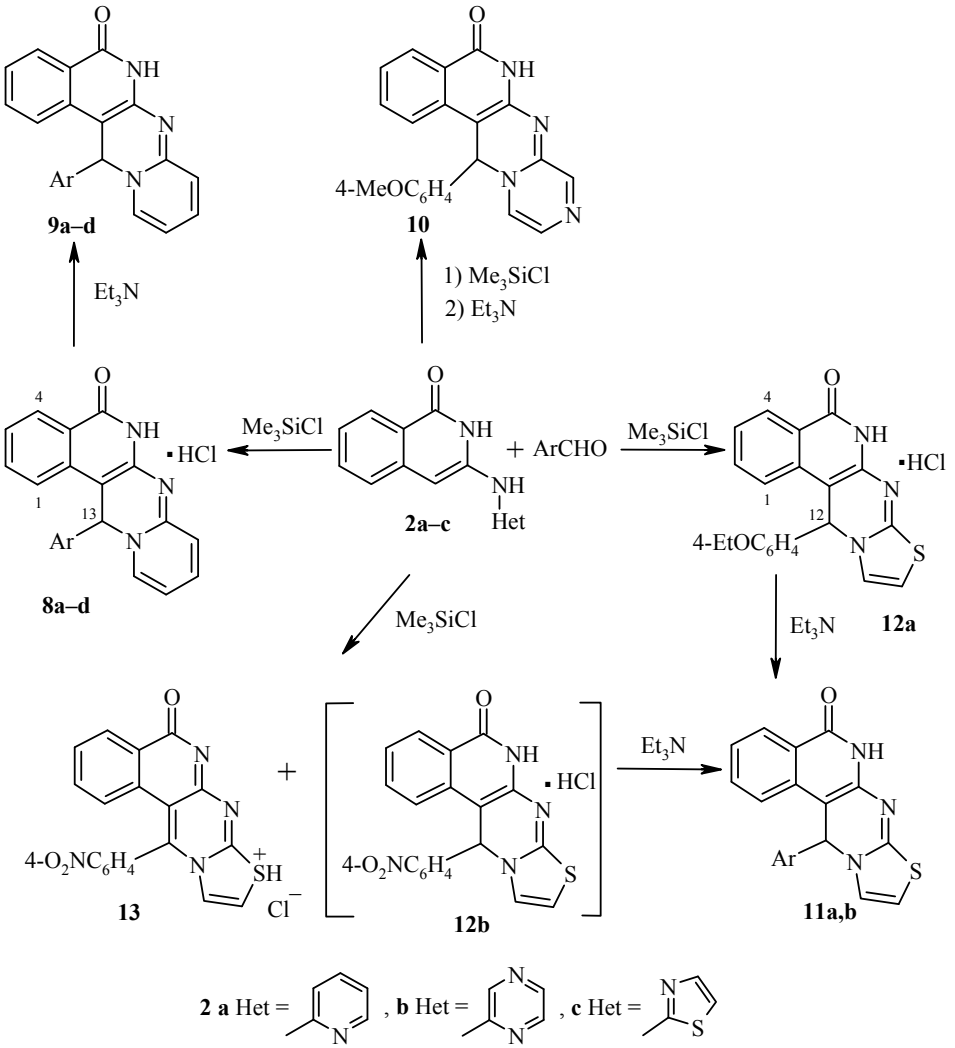
*[3]Analysis data for Cl and S, found/calculated, %: Cl – 8.61/8.63, S – 7.85/7.81.

component 7-(4-methoxyphenyl)-14-(4-nitrophenyl)-8,14-dihydrobenzo[*f*]isoquino[3,4-*b*][1,8]napthyridine-5,9(6H,7H)-dione (**5b**) was isolated. The formation of derivatives of benzo[*f*]isoquino[3,4-*b*][1,8]naphthyridine has been observed early in the condensation of 3-amino-2-methylisoquinolin-1(2H)-one with aromatic aldehydes in acetic acid [4, 5]. This result is apparently the consequence of intramolecular cyclization of Mannich adduct of the type **6** formed in the reaction of intermediate **3** activated by protonation with a molecule of starting isocarbostyryl **1a**. The ^{1}H NMR spectrum of product **5b** displays a sole one-proton singlet for H-14, while all aromatic proton signals have double intensity, which corresponds to a molecule with very high

symmetry (Table 3). The signal of the NH groups at 9.25 ppm is very broad and shifted upfield relative to the amide proton in starting isocarbostyryl **1a** at 10.64 ppm [9] due to rapid exchange with water present in the DMSO-d_6 solvent.

Carrying out this reaction in the presence of Me_3SiCl is a convenient method for the condensation of aldehydes with CH-acids, leading to products in high yield [10, 11]. We have found that heating isocarbostyryl **1a** with 4-methoxybenzaldehyde in anhydrous DMF in the presence of Me_3SiCl (Method B) leads to a mixture of reaction products, the major component of which is dibenzo[*b*,*f*][1,8]naphthyridine derivative **4a** in 64% yield. The second product isolated from the reaction mixture in low (24%) yield, namely, 14-(4-ethoxyphenyl)-7-(4-methoxyphenyl)-8,14-dihydrobenzo[*f*]isoquino[3,4-*b*][1,8]naphthyridine-5,9(6H,7H)-dione (**5a**), has low solubility in polar solvents such as ethanol and acetone. We should note that dione **5a** is also formed when the reaction is carried out in acetic acid but in yield less than 10% as indicated by the ^{1}H NMR spectrum of the reaction mixture. The yield of the product of the condensation of isocarbostyryl **1a** with 4-nitrobenzaldehyde is benzo[*f*]isoquino[3,4-*b*]naphthyridine **5b** is higher (45%) when the reaction is carried out in the presence of Me_3SiCl than when the reaction is carried out in acetic acid (28% yield) (Table 1). Furthermore, the reaction of

Scheme 2



8, **9 a** Ar = Ph, **b** Ar = 4-$MeOC_6H_4$, **c** Ar = 4-$EtOC_6H_4$, **d** Ar = 4-$O_2NC_6H_4$,
11 a Ar = 4-$EtOC_6H_4$, **b** Ar = 4-$O_2NC_6H_4$

1a with 4-nitrobenzaldehyde in the presence of Me_3SiCl gave an additional product, namely, 7-(4-methoxyphenyl)-14-(4-nitrophenyl)benzo[*f*]isoquino[3,4-*b*][1,8]naphthrydine-5,9(6H,7H)-dione (**7**). When the reaction is carried out in acetic acid, dione **7** is also present in the reaction mixture (~20% as indicated by the ^{1}H NMR spectrum) but could not be isolated due to the formation of a larger amount of by-products. In its mass spectrum, the peak with *m/z* 541.0 corresponds to molecular ion $[M+1]^+$, which indicates formation of the product of oxidation of compound **5b** (*m/z* 543.0 $[M+1]^+$). All the aromatic proton signals in the ^{1}H NMR spectrum of dione **7**, similar to the spectrum of dihydro derivative **5b**, have double intensity. One of the two-proton signals is seen upfield at 6.54 ppm and was assigned to resonance of the protons H-1 and H-13, which fall in the benzene ring shielding zone of the substituent at C-14. The signal of the NH group proton is lacking due to exchange with water present in the DMSO-d_6 solvent but the vibrational band for this group is observed in the IR spectrum at 3356 cm^{-1}. Furthermore, the IR spectrum of dione **7** shows vibrational bands for the C=O groups at ν = 1690 and 1684 cm^{-1}.

The condensation of isocarbostyryl **1b** with 4-methoxybenzaldehyde in the presence of Me_3SiCl gave only 12-(4-ethoxyphenyl)-10-nitrodibenzo[*b*,*f*][1,8]naphthyridin-5(6H)-one (**4b**), while formation of intermolecular reaction product **5** could not be detected. The reaction of isocarbostyryl **1b** with 4-nitrobenzaldehyde leads to a complex mixture of unidentified products both in acetic acid and in the presence of Me_3SiCl.

We then studied the reaction of benzaldehydes with 3-HetNH-isoquinolin-1(2H)-ones **2a-f** in the presence of Me_3SiCl (Schemes 2 and 3). The reaction of 3-(2-pyridylamino)isoquinolin-1(2H)-one **2a** with benzaldehydes under these conditions is also terminated by intramolecular cyclization, leading to hydrochloride salts of 13-aryl-6,13-dihydro-5H-pyrido[1',2':1,2]pyrimido[4,5-*c*]isoquinolin-5-ones **8a-d**. In this case, the pyridine ring nitrogen atom acts as the nucleophilic site in the cyclization step. Salts **8** are stable, and, with the exception of compound **8d**, were isolated as pure compounds. Isoquinoline **8d** contained up to 20% impurities, which could not be removed by recrystallization or chromatography, probably due to the instability of this compound relative to oxidation. Salts **8a-d** are readily converted to bases **9a-d** by the action of Et_3N (Method C). The ^{1}H NMR spectra of salts **8a-d** and bases **9a-d** (Table 3) differ by the position of aromatic proton signals, which are observed at lower field in the case of salts **8**. Protonation leads to increased lability of the NH group proton, whose signals in the ^{1}H NMR spectra of salts **8a-d** are very broadened or not observed due to rapid exchange. The greatest difference in the IR spectra of salts **8** and bases **9** is noted for the position of C=O group vibrational band, which is found at 1667-1670 cm^{-1} for salts **8** and at 1631-1645 cm^{-1} for bases **9**.

The reaction of 3-(2-pyrazinylamino)isoquinolin-1(2H)-one **2b** or 3-(1,3-thiazol-2-ylamino)-isoquinolin-1(2H)-one **2c** with 4-alkoxybenzaldehydes proceeds analogously to give 13-(4-methoxyphenyl)-6,13-dihydro-5H-pyrazino[1',2':1,2]pyrimido[4,5-*c*]isoquinolin-5-one (**10**) and 12-(4-ethoxyphenyl)-6,12-dihydro-5H-[1,3]thiazolo[3',2':1,2]pyrimido[4,5-*c*]isoquinolin-5-one (**11a**), respectively (Method C). The protic salt of isoquinolinone **10** is unstable, while in the case of **11a**, the corresponding salt **12a** contained impurities, which were probably products of the oxidation of compound **11a**.

Heating 3-thiazolylaminoisocarbostyryl **2c** with 4-nitrobenzaldehyde in the presence of Me_3SiCl for 5 h leads to a mixture, in which the oxidation product, namely, the hydrochloride salt of 12-(4-nitrophenyl)-5H-[1,3]thiazolo[3',2':1,2]pyrimido[4,5-*c*]isoquinolin-5-one (**13**) is the major product obtained in 72% yield, while 12-(4-nitrophenyl)-6,12-dihydro-5H-[1,3]thiazolo[3',2':1,2]pyrimido[4,5-*c*]isoquinolin-5-one (**11b**) was isolated in low (17%) yield. The yield of dihydro derivative **11b** may be increased to 30% by reducing the reaction time to 1 h (the yield of compound **13** in this case is 51%). The formation of protic salt **12b** in both cases could not be detected by NMR spectroscopy.

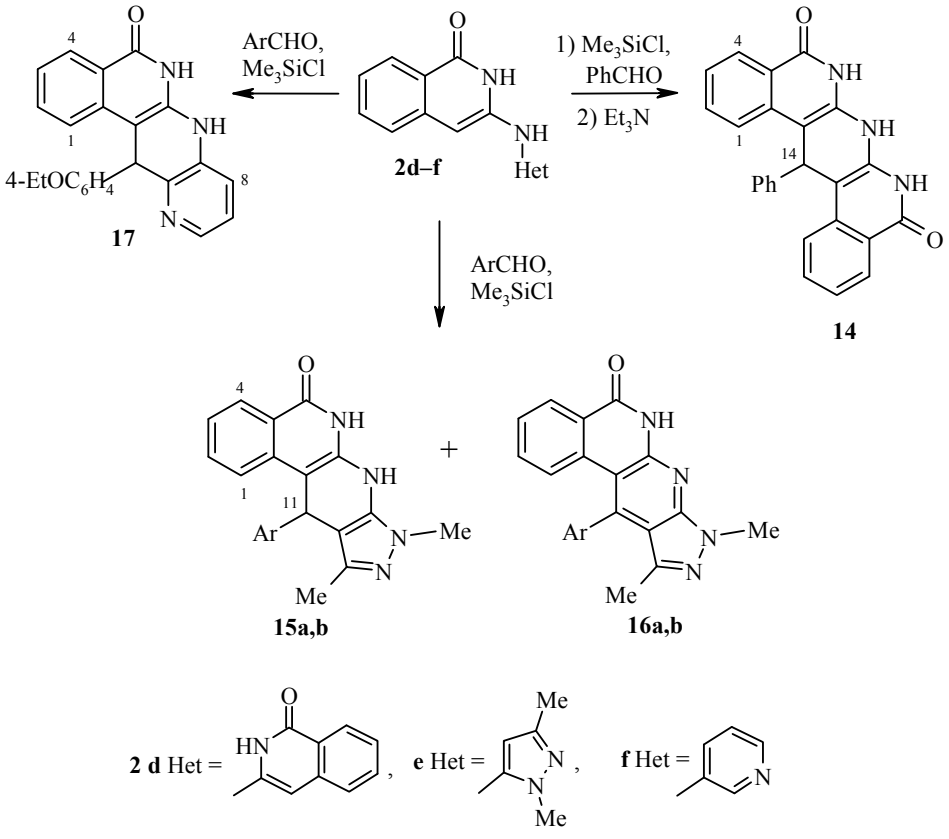
A distinguishing feature of the ^{1}H NMR spectrum of salt **13**, in addition to the lack of a signal for H-12 proton, is the position of the signal for H-1 proton, which is located in the region shielded by the benzene ring of the 12-Ar substituent and observed upfield (at 6.85 ppm) relative to the corresponding proton, H-5, in starting isocarbostyryl **2c** (at 7.49 ppm) [12]. The difference in the chemical shifts of these protons (Δδ ~ 0.64)

is comparable to the difference noted for compound **7** ($\Delta\delta \sim 0.73$, δ H-5 in starting **1a** is 7.27 ppm [9]) and higher than the difference noted for compounds **4a** and **4b** ($\Delta\delta \sim 0.12$ for **4a** and $\Delta\delta \sim 0.42$ for **4b**, δ H-5 in starting compound **1b** is 7.58 ppm [13]). This behavior may be the result of more efficient conjugation between the isoquinoline and pyrimidine rings in **13** or, correspondingly, the pyridine ring in compound **7**. We should note the position of the carbonyl group vibrational band ν_{CO} 1692 cm^{-1} in the IR spectrum of salt **13**, which is in the same region as for compound **7**, in contrast to the appearance of this band in the spectra of the other reaction products **4, 5,** and **8-12** at lower frequencies (1631-1670 cm^{-1}). Furthermore, the IR spectrum of salt **13** has a strong band at 2583 cm^{-1}, in the region characteristic for SH vibrations, while the weak band at 3126 cm^{-1} corresponds to the NH group participating in an intermolecular hydrogen bond. In our opinion, these data indicate the formation of structure **13**, in which the labile proton is localized on the sulfur atom.

The carbon atom is the nucleophilic site in the cyclization step in the condensation of 3-HetNH-isoquinolin-1(2H)-ones **2d** and **2e** with benzaldehydes (Scheme 3) as in the case of 3-ArNH-isoquinolin-1(2H)-ones **1a** and **1b**. In the presence of Me_3SiCl, isocarbostyryl **2d** is readily converted to 14-phenyl-8,14-dihydro-benzo[*f*]isoquino[3,4-*b*][1,8]naphthyridine-5,9(6H,7H)-dione (**14**, Method C).

The reaction of aromatic aldehydes with 1,3-dimethylpyrazolyl derivatives **2e** leads to a mixture of reaction products, whose composition depends on the structure of the aldehyde used and reaction heating time. In the case of heating compound **2e** with 4-ethoxybenzaldehyde for only 1 h, the major reaction product is 8,10-dimethyl-11-(4-ethoxyphenyl)-6,7,8,11-tetrahydro-5H-benzo[*f*]pyrazolo[3,4-*b*][1,8]naphthyridin-5-one (**15a**), while heating for 5 h gives the oxidized product, 11-(4-ethoxyphenyl)-8,10-dimethyl-6,8-dihydro-5H-benzo-[*f*]pyrazolo[3,4-*b*][1,8]naphthyridine-5-one (**16a**). The content of the second component of the mixture (dihydro

Scheme 3



15, 16 a Ar = 4-$EtOC_6H_4$, **b** Ar = 4-$O_2NC_6H_4$

TABLE 2. IR Spectra of Products **4, 5,** and **7-17**

Compound	IR spectrum, ν, cm^{-1}
4a	3148 (NH), 3064, 2980, 1664 (C=O), 1510, 1343, 1245 (C–O), 1049, 820, 761
4b	3137 (NH), 3064, 2986, 1664 (C=O), 1608, 1578 (NO_2), 1555, 1504, 1334 (NO_2), 1290, 1253 (C–O), 833, 747
5a	3384 (NH), 3059, 2980, 2924, 1664 (C=O), 1606, 1589, 1494, 1253 (C–O), 752
5b	3378 (NH), 3076, 1667 (C=O), 1594 (NO_2), 1547, 1497, 1343 (NO_2), 1306, 1253 (C–O), 1007, 755
7	3356 (NH), 3059, 1690 (C=O), 1684 (C=O), 1603, 1592, 1510 (NO_2), 1491, 1438, 1348 (NO_2), 1329, 1273, 1253 (C–O), 1029, 750, 694
8a	3406 (NH), 3064, 2779, 1667 (C=O), 1636, 1550, 1513, 1329, 775
8b	3412 (NH), 3064, 2980, 1670 (C=O), 1636, 1550, 1513, 1262 (C–O), 1175, 769
8c	3384 (NH), 3064, 2980, 1670 (C=O), 1639, 1550, 1513, 1259 (C–O), 1175, 772
9a	3434 (NH), 3028, 1635 (C=O), 1621, 1605, 1515, 1493, 1456, 1333, 1133, 760
9b	3435 (NH), 3070, 2997, 1631 (C=O), 1620, 1603, 1510, 1497, 1253 (C–O), 758
9c	3430 (NH), 3036, 2924, 1636 (C=O), 1617, 1608, 1508, 1500, 1345, 1248 (C–O), 755
9d	3384 (NH), 3070, 1645 (C=O), 1620, 1527 (NO_2), 1516, 1497, 1348 (NO_2), 1141, 822, 758, 736
10	3406 (NH), 3059, 1650 (C=O), 1628, 1606, 1527, 1510, 1250 (C–O), 1175, 758
11a	3440 (NH), 3109, 1636 (C=O), 1608, 1510, 1242 (C–O), 1206, 1172, 758
13	3126 (NH), 3031, 2773, 2583 (SH), 1692 (C=O), 1611, 1583, 1569, 1524 (NO_2), 1348 (NO_2), 1320, 1292, 1147, 836, 764, 738
14	3412 (NH), 3036, 1659 (C=O), 1631, 1608, 1555, 1541, 1429, 1298, 1256, 758, 699
15a	3221 (NH), 3165 (NH), 2980, 1667 (C=O), 1648, 1608, 1583, 1510, 1334, 1245 (C–O), 1046, 766
16a	3132 (NH), 2975, 2930, 1667 (C=O), 1580, 1530, 1342, 1245 (C–O), 1043, 604
16b	3154 (NH), 3064, 2924, 1660 (C=O), 1583, 1513 (NO_2), 1340 (NO_2), 844, 741
17	3330 (NH), 3199 (NH), 3059, 2980, 1662 (C=O), 1608, 1538, 1508, 1253 (C–O), 769

derivative **16a** in the former case and tetrahydro derivative **15a** in the latter) did not exceed 25%, which permitted the efficient separation of this mixture by crystallization. Independently of the heating time, the only product isolated as a pure compound when using 4-nitrobenzaldehyde in this reaction was 6,8-dihydro-5H-benzo[*f*]pyrazolo[3,4-*b*][1,8]naphthyridin-5-one **16b**. The content of 6,7,8,11-tetrahydro derivative **15b** in the reaction mixture (after heating for 1 h) was less than 15% as indicated by ^{1}H NMR spectroscopy.

3-(3-Pyridylamino)isoquinolin-1(2H)-one **2f**, which has an electron-deficient heterocycle at the 3-amino group, is also converted upon the action of 4-ethoxybenzaldehyde in the presence of Me_3SiCl into a cyclic product, namely, 12-(4-ethoxyphenyl)-7,12-dihydroisoquino[3,4-*b*][1,5]naphthyridin-5(6H)-one (**17**) in high (76%) yield. Closure of the dihydropyridine ring at C(2) atom in the 3-aminopyridine fragment of starting isocarbostyryl **2f** is the predominant course of this reaction, which is characteristic for pyridines possessing an electron-donor substituent at the β-position [14]. The structure of naphthyridinone **17** was supported by the two-dimensional NOESY spectrum.

Thus, the reaction of 3-ArNH- and 3-HetNH-isocarbostyryls with aromatic aldehydes gives 4-arylidene derivatives of the type **3**, which then convert under the reaction conditions (in the presence of Me_3SiCl, or in acetic acid in the case of 3-ArNH-isocarbostyryls **1a** and **1b**) in aryl-condensed or hetaryl-condensed derivatives of pyrimido[4,5-*c*]isoquinoline or benzo[*c*][1,8]naphthyridine, including derivatives of previously

unreported heterocyclic systems, namely, pyrazino[1',2':1,2]pyrimido[4,5-*c*]isoquinoline, [1,3]thiazolo-[3',2':1,2]pyrimido[4,5-*c*]isoquinoline, benzo[*f*]pyrazolo[3,4-*b*][1,8]naphthyridine, and isoquino[3,4-*b*][1,5]-naphthyridine.

The mechanism of the intramolecular cyclization depends on the structure of substituent at the 3-amino group of the starting isocarbostyryl and may occur in three directions. For derivatives of 2-aminoazoles or 2-aminoazines (**2a-c**), the mechanism involves alkylation at the endocyclic nitrogen atom of the Het substituent. In the other cases, in which there is no nitrogen atom in the α-position to the amino group in the Het substituent (**2d-f** and 3-ArNH-isocarbostyryls **1a** and **1b**), the mechanism involves Friedel-Crafts type aromatic electrophilic substitution or Michael addition at the β-position of the enamine fragment of a second isocarbostyryl molecule, while nucleophilic substitution of one of the 3-amino groups in the intermediate adduct leads to a cyclic product. The latter cyclization mechanism is more characteristic for 3-ArNH-isocarbostyryls **1a** and **1b** and may be the predominant reaction pathway when using benzaldehydes with an electron-withdrawing ring substituent. This behavior can likely be attributed to lower stability of the intermediate σ-complex (**18**, Scheme 1) and, as a consequence, increased likelihood of intermolecular alkylation.

EXPERIMENTAL

The IR spectra were taken on a Perkin-Elmer Spectrum BX spectrometer for KBr pellets. The ^{1}H and ^{13}C NMR spectra and HMQC and HMBC heteronuclear correlation experiments were carried out on a Varian Mercury-400 spectrometer at 400 and 100 MHz, respectively. In all cases, DMSO-d_6 was the solvent and TMS was the internal standard. The melting points were determined on a Boetius block and were uncorrected. The purity of the products was monitored by HPLC/MS on an Agilent 1100 instrument with an Agilent LC/MSD SL selective detector (the sample was introduced in a TFA matrix) using electron impact. The physicochemical characteristics and the elemental analysis data of the products are given in Tables 1-3.

3-(4-Methoxyanilino)isoquinolin-1(2H)-one (**1a**) was obtained according to Ali et al. [8], while 3-(4-nitroanilino)isoquinolin-1(2H)-one (**1b**) was obtained according to our previous procedure [13] and 3-HetNH-isoquinolin-1(2H)-ones **2a-f** were obtained according to our recent procedure [12].

Condensation in Acetic Acid (General Method). A. 4-Ethoxybenzaldehyde or 4-nitrobenzaldehyde (2.1 mmol) was added to a solution of 3-(arylamino)-1(2H)-isoquinolinone **1a,b** (2 mmol) in acetic acid (10 ml) and heated at reflux for 3-4 h. The solvent was evaporated and 10 ml diethyl ether was added to the residue. The precipitate was filtered off.

Condensation in the Presence of Me_3SiCl (General Method). B. Me_3SiCl (0.87 g, 8 mmol) was added dropwise to a solution of 3-(arylamino)-1(2H)-isoquinolinone **1a,b** or 3-(hetarylamino)isoquinolin-1(2H)-one **2a-f** (2 mmol) and aromatic aldehyde (2.1 mmol) in dry DMF (5 ml) and heated on a water bath at 90-95°C for 4-5 h. The reaction product precipitated upon heating the reaction mixture at reflux or after cooling. The precipitate was filtered off and washed with ethanol. If a precipitate did not form, the solvent was evaporated in vacuum and water (20 ml) was added to the residue. The precipitate formed was filtered off and washed with ethanol.

C. This procedure was carried out according to Method B. Then, the solid was dissolved in 5 ml DMF and 0.5 ml Et_3N was added. The mixture was maintained for 10 min at room temperature and then 20 ml water was added. The precipitate formed was filtered off and washed with ethanol.

12-Aryl-10-methoxybenzo[*b,f*][1,8]naphthyridin-5(6H)-ones 4a,b. Product **4a** was obtained by Method A or B (see procedure for the preparation of compound **5a**). Product **4b** was obtained by Method B.

14-(4-Ethoxyphenyl)-7-(4-methoxyphenyl)-8,14-dihydrobenzo[*f*]isoquino[3,4-][1,8]naphthyridine-5,9(6H,7H)-dione (5a) was obtained by Method B. The solvent was evaporated to give a mixture of **4a** and **5a**,

TABLE 3. ^{1}H NMR Spectra of Products **4, 5,** and **7-17**

Compound	^{1}H NMR spectrum (DMSO-d_6), δ, ppm (*J*, Hz)			
	NH (1H, s)	ArH	Ar–CH(1H, s)	Other signals
1	2	3	4	5
4a	12.02 (H-6)	8.35 (1H, d, ^3J = 7.5, H-4); 7.88 (1H, d, ^3J = 9.0, H-8); 7.55 (1H, t, ^3J = 7.5, H-2); 7.47 (1H, dd, ^4J = 2.5, ^3J = 9.0, H-9); 7.40 (1H, t, ^3J = 7.5, H-3); 7.35 (2H, d, ^3J = 8.5, H-2',6'); 7.25 (2H, d, ^3J = 8.5, H-3',5'); 7.15 (1H, d, ^3J = 8.0, H-1); 6.70 (1H, d, ^4J = 2.5, H-11)	—	4.20 (2H, q, ^3J = 7.0, OCH_2); 3.66 (3H, s, OCH_3); 1.44 (3H, t, ^3J = 7.0, CH_3)
4b	12.48 (H-6)	8.47 (1H, dd, ^3J = 8.0, ^4J =2.0, H-9); 8.36 (1H, d, ^3J = 8.0, H-8); 8.31 (1H, d, ^4J = 2.0, H-11); 8.07 (1H, d, ^3J = 8.5, H-4); 7.61 (1H, t, ^3J = 8.0, H-2); 7.47 (1H, t, ^3J = 8.0, H-3); 7.42 (2H, d, ^3J = 8.5, H-2',6'); 7.30 (2H, d, ^3J = 8.5, H-3',5'); 7.16 (1H, d, ^3J = 8.0, H-1)	—	4.23 (2H, q, ^3J = 7.0, OCH_2); 1.46 (3H, t, ^3J = 7.0, CH_3)
5a	9.31 (2H, br., H-6,8)	8.29 (2H, d, ^3J = 8.0, H-4,10); 8.09 (2H, d, ^3J = 8.0, H-1,13); 7.72 (2H, t, ^3J = 7.5, H-3,11); 7.60 (4H, m, H-2',6',2",6"); 7.34 (2H, t, ^3J = 7.5, H-2,12); 7.21 (2H, d, ^3J = 8.0, H-3',5'); 6.76 (2H, d, ^3J = 8.5, H-3",5")	6.10 (H-14)	3.90 (3H, s, OCH_3); 3.87 (2H, q, ^3J = 7.0, OCH_2); 1.22 (3H, t, ^3J = 7.0, CH_3)
5b	9.25 (2H, br., H-6,8)	8.33 (2H, m, H-4,10); 8.11 (2H, d, ^3J = 8.0, H-3",5"); 8.09 (2H, d, ^3J = 8.0, H-1,13); 8.02 (2H, d, ^3J = 8.0, H-2",6"); 7.74 (2H, t, ^3J = 8.0, H-3,11); 7.64 (2H, d, ^3J = 8.0, H-2',6'); 7.35 (2H, d, ^3J = 8.0, H-2,12); 7.23 (2H, d, ^3J = 8.0, H-3',5')	6.38 (H-14)	3.91 (3H, s, OCH_3)
7	—	8.53 (2H, d, ^3J = 8.0, H-3",5"); 8.18 (2H, d, ^3J = 8.0, H-4,10); 7.84 (2H, d, ^3J = 8.0, H-2",6"); 7.43 (4H, m, H-3,11,2',6'); 7.28 (2H, t, ^3J = 8.0, H-2,12); 7.22 (2H, d, ^3J = 8.0, H-3',5'); 6.54 (2H, d, ^3J = 8.0, H-1,13)	—	3.92 (3H, s, OCH_3)
8a	—	8.41 (1H, d, ^3J = 5.5, H-11); 8.16 (1H, d, ^3J = 7.5, H-4); 7.93 (1H, t, ^3J = 7.0, H-9); 7.65 (1H, t, ^3J = 7.5, H-2); 7.58 (2H, d, ^3J = 7.0, H-2',6'); 7.50 (1H, d, ^3J = 8.0, H-1); 7.35 (4H, m, H-8, H-3'-H-5'); 7.30 (1H, t, ^3J = 7.5, H-3); 7.16 (1H, t, ^3J = 6.0, H-10)	7.42 (H-13)	—
8b	—	8.21 (1H, d, ^3J = 5.5, H-11); 8.15 (1H, d, ^3J = 8.0, H-4); 7.73 (1H, t, ^3J = 6.5, H-9); 7.62 (1H, t, ^3J = 8.0, H-2); 7.49 (2H, d, ^3J = 8.0, H-2',6'); 7.41 (1H, d, ^3J = 8.0, H-1); 7.31 (1H, t, ^3J = 8.0, H-3); 7.16 (1H, d, ^3J = 8.5, H-8); 6.95 (1H, t, ^3J = 5.5, H-10); 6.89 (2H, d, ^3J = H-3',5')	7.19 (H-13)	3.69 (3H, s, OCH_3)
8c	11.60 (br., H-6)	8.58 (1H, d, ^3J = 5.5, H-11); 8.20 (1H, d, ^3J = 7.5, H-4); 8.11 (1H, t, ^3J = 7.0, H-9); 7.71 (1H, t, ^3J = 7.5, H-2); 7.54 (4H, m, H-1,8,2',6'); 7.43 (1H, t, ^3J = 7.5, H-3); 7.37 (1H, t, ^3J = 6.0, H-10); 6.90 (2H, d, ^3J = 8.0, H-3',5')	7.50 (H-13)	3.96 (2H, q, ^3J = 7.0, OCH_2); 1.26 (3H, t, ^3J = 7.0, CH_3)

TABLE 3 (continued)

1	2	3	4	5
8d*	—	8.50 (1H, d, ^3J = 5.5, H-11); 8.22 (3H, m, H-4,3',5'); 8.06 (1H, t, ^3J = 6.5, H-9); 7.91 (2H, d, ^3J = 8.5, H-2',6'); 7.72*[2] (2H, m, H-2,13); 7.57 (1H, d, ^3J = 7.5, H-1); 7.51 (1H, m, H-8); 7.43 (1H, t, ^3J = 7.5, H-3); 7.29 (1H, m, H-10)	7.72*[2] (H-3)	—
9a	11.19 (H-6)	8.09 (1H, d, ^3J = 7.5, H-4); 7.80 (1H, d, ^3J = 6.5, H-11); 7.55-7.48 (3H, m, H-2,2',6'); 7.32 (4H, m, H-1, H-3'-H-5'); 7.25 (1H, t, ^3J = 7.0, H-9); 7.18 (1H, t, ^3J = 7.5, H-3); 6.77 (1H, d, ^3J = 9.0, H-8); 6.50 (1H, t, ^3J = 7.0, H-10)	6.91 (H-13)	—
9b	11.12 (H-6)	8.09 (1H, d, ^3J = 7.5, H-4); 7.80 (1H, d, ^3J = 6.5, H-11); 7.52 (1H, t, ^3J = 7.5, H-2); 7.41 (2H, d, ^3J = 8.5, H-2',6'); 7.32 (1H, t, ^3J = 8.0, H-9); 7.28 (1H, d, ^3J = 8.0, H-); 7.17 (1H, t, ^3J =7.5, H-3); 6.85 (2H, d, ^3J = 8.5, H-3',5'); 6.75 (1H, d, ^3J =9.0, H-8); 6.49 (1H, t, ^3J = 6.5, H-10)	6.88 (H-13)	3.68 (3H, s, OCH_3)
9c	11.23 (br., H-6)	8.10 (1H, d, ^3J = 7.5, H-4); 7.86 (1H, m, H-11); 7.54 (1H, t, ^3J = 7.5, H-2); 7.39 (3H, m, H-9,2',6'); 7.30 (1H, d, ^3J = 8.0, H-1); 7.20 (1H, t, ^3J = 7.5, H-3); 6.83-6.80 (3H, m, H-8,3',5'); 6.57 (1H, m, H-10)	6.90 (H-13)	3.93 (2H, q, ^3J = 7.0, OCH_2); 1.25 (3H, t, ^3J = 7.0, CH_3)
9d	11.30 (br., H-6)	8.20 (2H, d, ^3J = 8.5, H-3',5'); 8.10 (1H, d, ^3J = 8.0, H-4); 7.82 (1H, d, ^3J = 6.5, H-11); 7.77 (2H, d, ^3J = 8.5, H-2',6'); 7.55 (1H, t, ^3J = 8.0, H-2); 7.38 (1H, t, ^3J = 8.0, H-9); 7.34 (1H, d, ^3J = 8.0, H-1); 7.21 (1H, t, ^3J = 8.0, H-3); 6.81 (1H, d, ^3J = 9.0, H-8); 6.55 (1H, t, ^3J = 6.0, H-10)	7.15 (H-13)	—
10	11.45 (H-6)	8.18 (1H, s, H-8); 8.12 (1H, d, ^3J = 8.0, H-4); 7.63 (1H, d, ^3J = 6.0, H-11); 7.55 (1H, t, ^3J = 8.0, H-2); 7.42 (3H, m, H-10,2',6'); 7.26 (2H, m, H-1,3); 6.89 (2H, d, ^3J = 8.5, H-3',5')	6.87 (H-13)	3.67 (3H, s, OCH_3)
11a	11.28 (H-6)	8.09 (1H, d, ^3J = 7.5, H-4); 7.49 (1H, t, ^3J = 7.5, H-2); 7.39 (2H, d, ^3J = 8.5, H-2',6'); 7.19 (2H, m, H-1,2); 7.14 (1H, d, ^3J = 4.0, H-9); 6.88 (2H, d, ^3J = 8.5, H-3',5'); 6.70 (1H, d, ^3J = 4.0, H-10)	6.78 (H-12)	3.95 (2H, q, ^3J = 7.0, OCH_2); 1.26 (3H, t, ^3J = 7.0, CH_3)

TABLE 3 (continued)

1	2	3	4	5
11b	11.41 (H-6)	8.22 (2H, d, ^3J = 8.5, H-3',5'); 8.11 (1H, d, ^3J = 8.0, H-4); 7.80 (2H, d, ^3J = 8.5, H-2',6'); 7.52 (1H, t, ^3J = 8.0, H-2); 8.23 (1H, t, ^3J = 8.0, H-3); 7.20 (2H, m, H-1,9); 6.76 (1H, d, ^3J = 4.0, H-10)	7.07 (H-12)	—
12a*	—	8.12 (1H, d, ^3J = 7.5, H-4); 7.53 (1H, t, ^3J = 7.5, H-2); 7.44 (2H, d, ^3J = 8.0, H-2',6'); 7.29-.20 (3H, m, H-1,2,9); 6.88*[2] (3H, m, H-12,3',5'); 6.84 (1H, d, ^3J = 4.0, H-10)	6.88*[2] (H-12)	3.96 (2H, q, ^3J = 7.0, OCH_2); 1.27 (3H, t, ^3J = 7.0, CH_3)
13	13.81 (SH)	8.76 (2H, d, ^3J = 8.5, H-3',5'); 8.43 (1H, d, ^3J = 8.0, H-4); 8.17 (1H, d, ^3J = 4.0, H-10); 8.03 (2H, d, ^3J = 8.5, H-2',6'); 7.83 (1H, d, ^3J = 4.0, H-9); 7.79 (1H, t, ^3J = 8.0, H-2); 7.62 (1H, t, ^3J = 8.0, H-3); 6.85 (1H, d, ^3J = 8.0, H-1)	—	—
14	11.54 (2H, br., H-6,8); 8.20 (1H, H-7)	8.09 (2H, d, ^3J = 8.0, H-4,10); 8.02 (2H, d, ^3J = 8.0, H-2',6'); 7.63-7.58 (4H, m, H-1,2,12,13); 7.23 (2H, t, ^3J = 8.0, H-3,11); 7.15 (2H, t, ^3J = 8.0, H-3',5'); 6.08 (1H, t, ^3J = H-4')	5.62 (H-14)	—
15a	10.31 (1H, br., H-6); 5.54 (1H, br., H-7)	8.09 (1H, d, ^3J = 7.5, H-4); 7.49 (2H, m, H-1,2); 7.25 (2H, d, ^3J = 8.0, H-2',6'); 7.22 (1H, t, ^3J = 7.0, H-3); 6.78 (2H, d, ^3J = 8.0, H-3',5')	5.48 (H-11)	3.92 (2H, q, ^3J = 7.0, OCH_2); 3.75 (3H, s, 8-CH_3); 2.07 (3H, s, 10-CH_3); 1.26 (3H, t, ^3J = 7.0, CH_3)
16a	12.14 (H-6)	8.31 (1H, d, ^3J = 7.5, H-4); 7.46 (1H, t, ^3J = 7.5, H-2); 7.37 (3H, m, H-3,2',6'); 7.29 (1H, d, ^3J = 8.0, H-1); 7.19 (2H, d, ^3J = 8.5, H-3',5')	—	4.17 (2H, q, ^3J = 7.0, OCH_2); 3.95 (3H, s, 8-CH_3); 1.77 (3H, s, 10-CH_3); 1.42 (3H, t, ^3J = 7.0, CH_3)
16b	12.25 (H-6)	8.52 (2H, d, ^3J = 8.5, H-3',5'); 8.33 (1H, d, ^3J = 8.0, H-4); 7.85 (2H, d, ^3J = 8.5, H-2',6'); 7.50 (1H, t, ^3J = 7.5, H-2); 7.39 (1H, t, ^3J = 7.5, H-3); 7.07 (1H, d, ^3J = 8.0, H-1)	—	3.98 (3H, s, 8-CH_3); 1.72 (3H, s, 10-CH_3)
17	11.28 (1H, br., H-6); 9.78 (1H, br., H-7)	8.29 (1H, d, ^3J = 5.0, H-10); 8.12 (1H, d, ^3J = 8.0, H-4); 7.87 (1H, d, ^3J = 8.0, H-8); 7.59 (2H, m, H-2,9); 7.45 (1H, d, ^3J = 8.0, H-1); 7.37 (2H, d, ^3J = 8.5, H-2',6'); 7.26 (1H, t, ^3J = 8.0, H-3); 6.79 (2H, d, ^3J = 8.5, H-3',5')	5.88 (H-12)	3.90 (2H, q, ^3J = 7.0, OCH_2); 1.24 (3H, t, ^3J = 7.0, CH_3)

*The ^{1}H NMR spectrum of the major component of the reaction mixture is given; the content of compound **8d** was 80%, the content of **12a** was 75%.

*[2]Signals overlap.

which was then heated to reflux in 5 ml 1:1 acetonitrile–ethanol. The precipitate was filtered off and washed with hot ethanol to give dione **5a**. Cooling the filtrate gave a precipitate of compound **4a**, which was filtered off and washed with ethanol.

7-(4-Methoxyphenyl)-14-(4-nitrophenyl)-8,14-dihydrobenzo[f]isoquino[3,4-b][1,8]naphthyri-dine-5,9(6H,7H)-dione (5b) was obtained by Method A or B (see procedure for the preparation of dione **7**).

7-(4-Methoxyphenyl)-14-(4-nitrophenyl)benzo[*f*]isoquino[3,4-*b*][1,8]naphthyridine-5,9(6H,7H)-dione (7) was obtained by Method B. Reaction product **7** was obtained as a precipitate upon heating the reaction mixture at reflux. The precipitate was filtered off and washed with hot ethanol. Cooling the filtrate gave a precipitate of dione **5b**, which was filtered off and washed with ethanol.

13-Aryl-6,13-dihydro-5H-pyrido[1',2':1,2]pyrimido[4,5-*c*]isoquinolin-5-one Hydrochloride (8a-c) and 12-(4-Ethoxyphenyl)-7,12-dihydroisoquino[3,4-*b*][1,5]naphthyridin-5(6H)-one Hydrochloride (17) were obtained by Method B.

13-Aryl-6,13-dihydro-5H-pyrido[1',2':1,2]pyrimido[4,5-*c*]isoquinolin-5-ones (9a-d), 13-(4-methoxy-phenyl)-6,13-dihydro-5H-pyrazino[1',2':1,2]pyrimido[4,5-*c*]isoquinolin-5-one (10), 12-(4-ethoxyphenyl)-6,12-dihydro-5H-[1,3]thiazolo[3',2':1,2']pyrimido[4,5-*c*]isoquinolin-5-one (11a), and 14-phenyl-8,14-dihydrobenzo-[*f*]isoquino[3,4-*b*][1,8]naphthyridine-5,9(6H,7H)-dione (14) were obtained according to Method C.

12-(4-Nitrophenyl)-6,12-dihydro-5H-[1,3]thiazolo[3',2':1,2]pyrimido[4,5-*c*]isoquinolin-5-one (11b) was obtained by Method B. The heating time was 1 h. The precipitate formed during heating of the solution at reflux was filtered off the hot solution and washed with acetone to give compound **13** in 51% yield. Isoquinolinone **11b** was separated from the filtrate by Method C.

12-(4-Nitrophenyl)-5H-[1,3]thiazolo[3',2':1,2]pyrimido[4,5-c]isoquinolin-5-one Hydrochloride (13) was obtained by Method B. The heating time was 5 h. The precipitate formed during heating of the solution at reflux was filtered off the hot solution and washed with acetone to give compound **13**. Isoquinolinone **11b** was separated in 17% yield from the filtrate by Method C.

11-(4-Ethoxyphenyl)-8,10-dimethyl-6,7,8,11-tetrahydro-5H-benzo[*f*]pyrazolo[3,4-*b*][1,8]naphthyridin-5-one (15a) was obtained by Method B. The heating time was 1 h. The precipitate formed during heating of the solution at reflux was filtered off the hot solution and washed with hot ethanol to give **16a** in 20% yield. The filtrate was evaporated in vacuum and 20 ml water was added to the residue. The precipitate of naphthyridinone **15a** was filtered off and washed with ethanol.

11-Aryl-8,10-dimethyl-6,8-dihydro-5H-benzo[*f*]pyrazolo[3,4-*b*][1,8]naphthyridin-5-ones 16a,b were obtained by Method B. The heating time was 5 h. The precipitate of 6,8-dihydrobenzo[*f*]pyrazolo-[3,4-*b*][1,8]naphthyridinone **16a,b** formed upon heating the solution at reflux was filtered off the hot solution and washed with hot ethanol. In the case of the reaction with 4-ethoxybenzaldehyde, naphthyridinone **15a** was separated from the filtrate in 22% yield (see procedure above).

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