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CONDENSED ISOQUINOLINES 26*. OXIDATION REACTIONS OF 6,11-DIHYDRO-13H-ISOQUINO-[3,2- *b*]QUINAZOLIN-13-ONE DERIVATIVES

L. M. Potikha, V. A. Kovtunenko, and A. V. Tarasevich

Oxidation of the hydrobromide of 6,11-dihydro-13H-isoquino[3,2-b]quinazolin-13-one with dimethyl sulfoxide leads to 11-hydroxy-11H-isoquino[3,2-b]quinazoline-6,13-dione, and with hydrogen peroxide to the hydrobromide of 2-[(4-oxo-3,4-dihydro-2-quinazolinyl)carbonyl]benzoic acid. Salts of 5- and 6-alkyl-substituted isoquino[3,2-b]quinazolines are readily oxidized on boiling in nitrobenzene, which leads to aromatization of the isoquino[3,2-b]quinazoline system to 5-methyl-13-oxo-5H,13H-isoquino[3,2-b]quinazolinium perchlorate and 6-benzyl-13H-isoquino[3,2-b]quinazolin-13-one. The structures of the dehydrogenation products were established from ^{1}H NMR and UV spectra. The interaction of the obtained compounds with $NaBH_4$ has been studied.

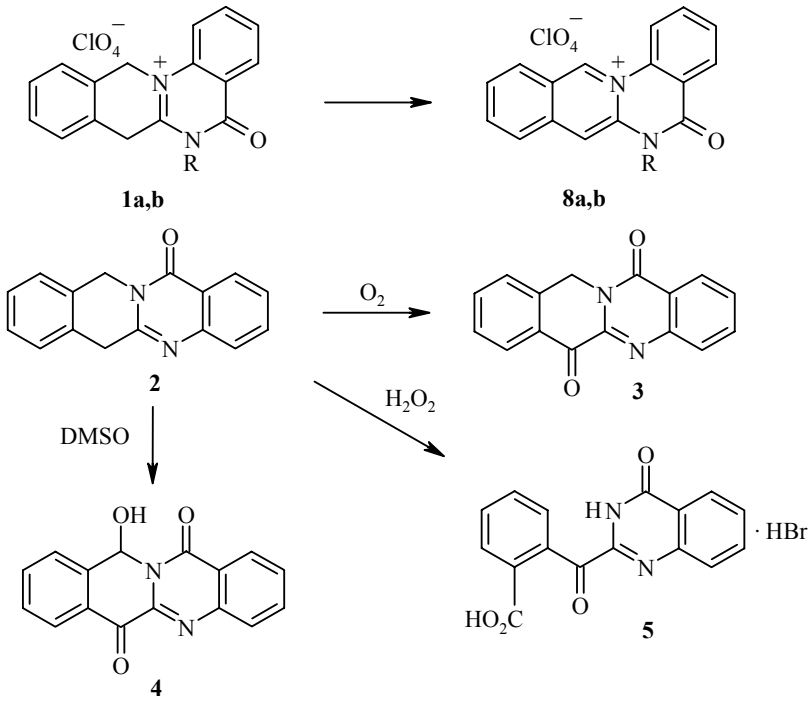
Keywords: 6-benzyl-13H-isoquino[3,2-*b*]quinazolin-13-one, isoquino[3,2-*b*]quinazoline, 2-[(4-oxo-3,4-dihydro-2-quinazolinyl)carbonyl]benzoic acid, 6-methyl-13-oxo-5H,13H-isoquino[3,2-*b*]quinazolinium perchlorate, oxidation.

One of the characteristic properties of secondary enamines, and imines close to them in behavior, with an activated methylene group is the ease of carrying out oxidation reactions [2, 3]. In the case of condensed heterocyclic systems this is aided by definite structural factors such as the presence of benzylic positions and the possibility of forming conjugated systems in the reaction products, which also determines the main directions of the conversions. Previously [4-8] we showed that such reactions in the condensed isoquinoline series take place fairly readily even in the presence of relatively weak oxidizing agents like the oxygen of the air, aromatic aldehydes, or sulfo derivatives. But their main directions are aromatization of heterosystems, as in the case of derivatives of 7,12-dihydro-5H-isoquino[2,3-*a*]quinazolin-5-one **1a,b** [4, 5], or the formation of products of dimeric structure, on oxidation of benzimidazo[1,2-*b*]isoquinolin-11(5H)-one [6] or 6,11-dihydro-13H-isoquino[3,2-*b*]quinazolin-13-one (**2**) [5, 7, 8]. Oxidation is the main side reaction on interacting isoquino-[3,2-*b*]quinazolin-13-one **2** with electrophilic reagents, but in certain cases this is the main direction of the conversions, consequently it seemed to us useful to study this problem in a more detailed manner.

*For Communication 25 see [1].

Kiev Taras Shevchenko National University, Kiev 01033, Ukraine; e-mail: potikha_l@mail.ru. Translated from Khimiya Geterotsiklicheskikh Soedinenii, No. 12, pp. 1833-1840, December, 2007. Original article submitted January 22, 2007.

It was shown previously [5], that the proton salts of isoquino[3,2-*b*]quinazolin-13-one **2** are oxidized at positions 6 and 11 by air oxygen at temperatures >180°C (heating in benzonitrile, N-methylpyrrolid-2-one), which leads to a mixture of products of dimeric structure and 11H-isoquino[3,2-*b*]quinazoline-6,13-dione (**3**).

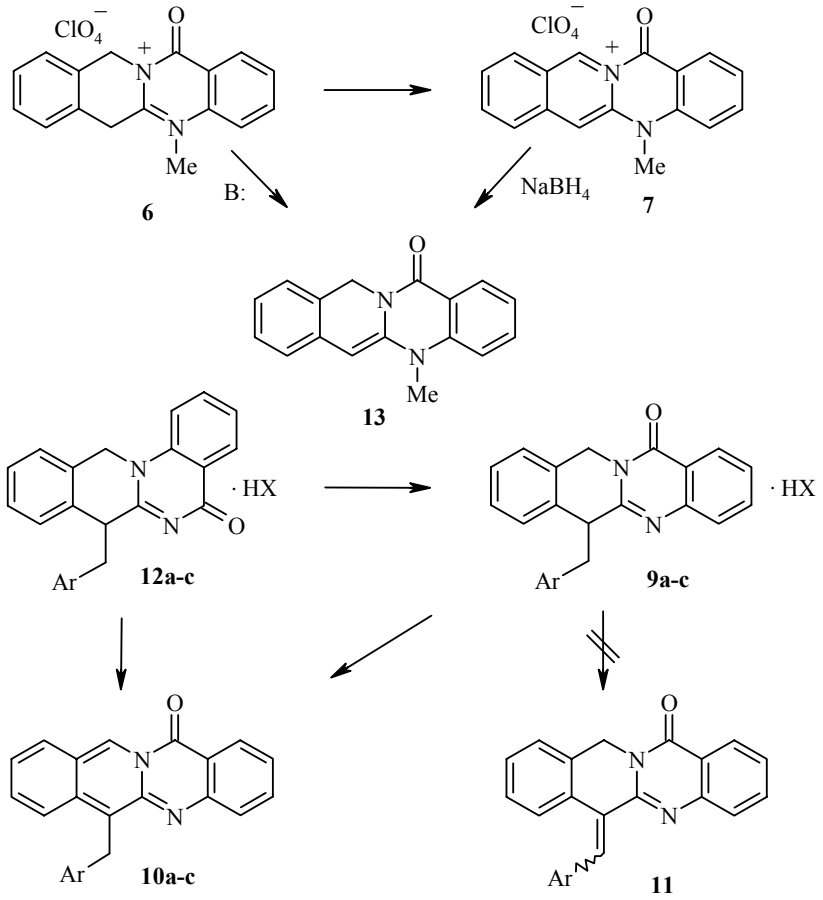


1,8 **a** R = H, **b** R = Me

It turned out that the oxidation of salt of isoquino[3,2-*b*]quinazoline (**2**·HBr) also takes place readily and in good yield on heating in DMSO. Reaction takes place at both methylene groups and leads to the formation of 11-hydroxy-11H-isoquino[3,2-*b*]quinazoline-6,13-dione (**4**). The presence of a hydroxyl group in the structure of the reaction product was established by spectral data, a broad band for the stretching vibrations ν_{OH} (3200 cm^{-1}) and ν_{CO} (1300 cm^{-1}) in the IR spectrum, and also a broadened one-proton signal (exchanging with D_2O) at 7.37 ppm in the ^1H NMR spectrum. The ^1H NMR spectrum of compound **4** did not contain signals in the region below 7.0 ppm, but the resonance of the H-11 proton is observed at 7.15 ppm (s). Assignment of the signals was made by studying proton-proton correlations (NOE, COSY). Confirmation for the structure of **4** was also found in the ^{13}C NMR spectrum: 178.25 (C-6), 160.53 (C-13), 73.26 ppm (d, J_{CH} = 162 Hz, C-11). Additional evidence in favor of the structure of compound **4** might also be the overall similarity of the electronic spectra of 11H-isoquino[3,2-*b*]quinazoline-6,13-dione (**3**) [9] and of **4** (see EXPERIMENTAL).

Still deeper oxidation of salt 2·HBr takes place with hydrogen peroxide. The reaction is accompanied by fission of the isoquinoline ring and leads to 2-[(4-oxo-3,4-dihydro-2-quinazolinyl)carbonyl]benzoic acid hydrobromide (**5**) . It must be said that reaction with H_2O_2 takes place only on heating. Extended (~10 h) storage of a solution of **2**·HBr and H_2O_2 in acetonitrile at room temperature did not lead to an apparent change in the reaction mixture. The ^1H NMR spectrum of compound **5** also contains no signals in the region below 7.0 ppm, but at low field a broadening was observed in the signals for OH (13.15) and $N_{(3')}H$ (12.54 ppm). On attempting to carry out the oxidation of free base **2** with DMSO and H_2O_2 a more complex mixture of products was obtained, in which the content of compounds **4** and **5** (according to data of TLC and ^1H NMR spectra) was small.

The preparation of aromatic derivatives of isoquino[3,2-*b*]quinazolin-13-one **2** is extremely interesting both from a theoretical and a practical point of view. It should be noted that an aromatic compound with an isoquino[3,2-*b*]quinazoline nucleus is mentioned previously only in one example *viz*. 11H-isoquino-[3,2-*b*]quinazolin-11-one [10], which was obtained on high temperature oxidation of the corresponding 5,13-dihydro derivative with iodine. However the structure of this compound was not demonstrated conclusively by the author. We expected that in the case of compound **2** and its proton salts aromatization might be carried out by interaction with various dehydrogenating agents. However under all the conditions tried by us, particularly heating compound **2** and its salts (**2**·HBr, **2**·$HClO_4$) with sulfur or iodine in high-boiling solvents and boiling in nitrobenzene, resinification occurs or a complex mixture of products is formed, containing chiefly products of oxidative dimerization.



9,10,12 a Ar = Ph, X = Cl; **b** Ar = 3-$O_2NC_6H_4$, X = Br; **c** Ar = 2-MeC_6H_4, X = Br

The desired result was obtained on heating salts of 5- and 6-alkyl-substituted isoquino[3,2-*b*]quinazolin-13-ones in nitrobenzene. Such a change in the direction of oxidation in comparison with isoquinoquinazoline **2** is evidently explained by steric hindrance to the dimerization reaction from the side of the 5- and especially the 6-alkyl substituents. So the brief (~10 min) heating of 5-methyl-13-oxo-6,13-dihydro-11H-isoquino-[3,2-*b*]quinazolinium perchlorate (**6**) in nitrobenzene leads to 5-methyl-13-oxo-5H,13H-isoquino[3,2-*b*]quinazo-linium perchlorate (**7**) (Table 1). In the ^1H NMR spectrum of dehydrogenation product **7** the signals for the aliphatic protons of the methylene groups were absent (see Table 2), but the low field disposition in the spectrum of a series of signals indicates the presence in the heterocyclic fragment of a delocalized

TABLE 1. Physicochemical Characteristics of Compounds **7, 10a-c**

Compound	Empirical formula	Found, % / Calculated, %				mp,°C*	Yield, %
		C	H	Hal	N		
7	$C_{17}H_{13}ClN_2O_5$	56.53 / 56.60	3.58 / 3.63	9.84 / 9.83	7.80 / 7.77	275-276	65
10a	$C_{23}H_{16}N_2O$	82.05 / 82.12	4.70 / 4.79	—	8.35 / 8.33	218-220	73
10b	$C_{23}H_{15}N_3O_3$	72.30 / 72.43	3.88 / 3.96	—	11.01 / 11.02	240-242	62
10c ·HBr	$C_{24}H_{19}BrN_2O$	66.70 / 66.83	4.36 / 4.44	18.55 / 18.53	6.52 / 6.49	201-203	59

*Solvent: AcOH (compounds **7, 10c**·HBr) and DMF (compounds **10a,b**).

TABLE 2. Spectral Characteristics of Compounds **7, 10a-c**

Compound	IR spectrum, ν, cm^{-1}	UV spectrum, λ_{max}, nm ($\varepsilon\times10^3$)	^{1}H NMR spectrum, δ, ppm (*J*, Hz)		
			H-11, s	Ar–H	other signals
7	3100, 1740 (br., C=O), 1595, 1470, 1305, 1074 (ClO_4^-), 755	208* (59.3), 235 (72.2), 327 (63.8), 390* (7.7)	10.60	8.89 (1H, s, H-6); 8.62 (1H, d, *J* = 8.0, H-10); 8.55 (1H, d, ^oJ = 8.0, H-1); 8.26 (1H, d, ^oJ = 8.0, H-7); 8.18 (1H, t, ^oJ = 8.0, H-3); 8.10 (2H, m, H-4,8); 7.78 (1H, t, ^oJ = 8.0, H-2); 7.67 (1H, t, ^oJ = 8.0, H-9)	4.23 (3H, s, CH_3)
10a	1670 (C=O, C=N), 1495, 1440, 770, 740, 693	—	9.81	8.38 (1H, d, ^oJ = 8.0, H-10); 8.01 (1H, d, ^oJ = 8.2, H-1); 7.92 (1H, d, ^oJ = 8.2, H-7); 7.86 (1H, t, ^oJ = 8.0, H-3); 7.79 (1H, d, ^oJ = 8.0, H-4); 7.43 (2H, m, H-2,8); 7.32 (2H, d, ^oJ = 8.0, H-2',6'); 7.24 (1H, t, ^oJ = 7.8, H-9); 7.17 (2H, t, ^oJ = 8.0, H-3',5'); 7.09 (1H, t, ^oJ = 8.0, H-4')	4.96 (2H, s, CH_2)
10b	1670 (C=O, C=N), 1495, 1445, 1337 (NO_2)	237 (89.4), 266 (83.4), 310* (53.0), 368 (12.5), 390 (23.8), 407 (38.1), 450* (6.0), 503 (11.3), 520 (9.5), 575* (7.1)	9.84	4.38 (1H, d, ^oJ = 8.0, H-10); 4.32 (1H, s, H-2'); 8.07 (2H, m, H-1,4'); 8.00 (1H, d, ^oJ = 8.0, H-7); 7.90 (1H, t, ^oJ = 8.0, H-3); 7.82 (2H, m, H-4,6'); 7.46 (3H, m, H-2,8,5'); 7.28 (1H, t, ^oJ = 8.0, H-9)	5.07 (2H, s, CH_2)
10c·HBr	3410 (NH), 1725 C=O), 1625 (C=N), 750	238 (160.0), 267 (110.5), 284* (91.7), 311* (62.0), 370 (23.3), 390 (37.7), 410 (59.1), 445* (13.5), 500 18.0), 517 (17.0), 580* (10.8)	10.44	8.62 (1H, d, ^oJ = 8.0, H-1); 8.46 (1H, d, ^oJ = 8.0, H-10); 8.15 (2H, m, H-3,7); 8.07 (1H, t, ^oJ = 8.0, H-8); 7.93 (1H, m, H-9); 7.70 (1H, t, ^oJ = 8.0, H-2); 7.56 (1H, d, ^oJ = 8.0, H-4); 7.45 (1H, d, ^oJ = 7.2, H-6'); 7.31 (1H, t, ^oJ = 7.2, H-4'); 7.06 (1H, t, ^oJ = 7.2, H-5'); 6.62 (1H, d, ^oJ = 8.0, H-3')	4.91 (2H, s, CH_2); 2.61 (3H, s, CH_3)

* Indicated by point of inflection.

positive charge, Thus the low field singlet at 10.60 ppm was assigned by us to the resonance of H-11 on the basis of data obtained when studying the spectral behavior of the isomeric 5-oxo-5H,6H-isoquino-[2,3-*a*]quinazolinium salts **8a,b** [4, 5]. The characteristic region for absorption of aromatic protons of the type –C<u>H</u>=N^+ for compounds **8a,b** was 10.9-12.00 ppm. The signals of the remaining aromatic protons of compound **7** were displaced towards low field by 0.3 ppm on average in comparison with the corresponding signals for the initial salt **6** [1]. The greatest difference in chemical shifts is found for the H-10 proton (d, 8.62 ppm, $\Delta\delta = 1.07$), which is in full agreement with the structure assigned to dehydrogenation product **7**.

The dehydrogenation of proton salts of 6-benzyl-6,11-dihydro-13H-isoquino[3,2-*b*]quinazolin-13-ones **9a-c** also takes place readily in nitrobenzene, and leads to 6-benzyl-13H-isoquino[3,2-*b*]quinazolin-13-ones **10a-c**. This conversion is probably catalyzed by acid, since on using free bases of compounds **9a-c** in the reaction heavy resinification occurs and the yield of products of structure **10** is small. 6-Benzyl- and 6-(3-nitro-benzyl)isoquinoquinazolines **10a,b** were obtained as the free bases, but 6-(2-methylbenzyl)isoquinoquinazoline **10c** as the hydrobromide. Evidently the higher stability of the proton salt of compound **10c** in comparison with the proton salts of compounds **10a,b** may be explained by the donor effect of the 2-methylbenzyl substituent. Nonetheless, the low stability of the proton salts of the dehydrogenation products **10** was somewhat unexpected, since in the case of isoquino[2,3-*a*]quinazolines **8a** such salts were completely stable [5].

Definite doubts as to the correctness of the structure assigned to the oxidation product of salt **9** were also provoked by the picture of the absorption in the ^{1}H NMR spectra. The singlet of the aliphatic protons of the 6-C<u>H</u>$_2$Ar methylene group at 4.90-5.07 and the H-11 methine proton at 9.81-10.44 ppm may also be attributed to the resonance of the corresponding groups in 6-(arylidene)-6,11-dihydro-13H-isoquino[3,2-*b*]quinazolin-13-ones (**11**). The difference consisted of the position of the signal of the methine proton of the arylidene residue, the resonance of which for compound **11** was observed at higher field (7.0-9.0 ppm) [8]. But since the configuration of compound **11** has not been established we have not excluded the possibility of forming, on oxidation of salts **9**, the isomeric 6-arylidene derivatives with a configuration alternative to the condensation product of isoquinoquinazoline **2** with benzaldehydes.

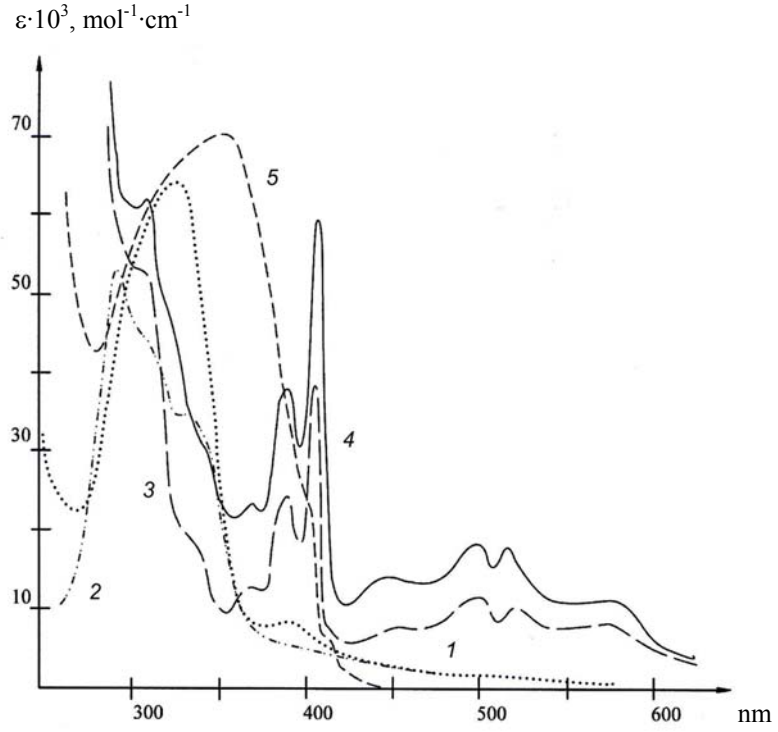


Fig. 1. UV spectra of compounds **7** *(1)*, **8b** *(2)*, **10b** (3), **10c** *(4)*, and **11**·HBr *(5)* (Ar = 4-$O_2NC_6H_4$) in methanol.

A final conclusion on the structures of compounds **10a-c** as the products of dehydrogenation at positions 6 and 11 was made on the basis of an analysis of their electronic spectra. The absorption curves of solutions of compounds **10b,c** in methanol differed significantly in their shape from the absorption curves of 6-arylidene derivatives **11** (see Fig. 1). In the UV spectra of compounds **10b,c** absorption bands were observed with maxima in the longer wave region with differences ($\Delta\lambda$ >40 nm) exceeding in size those characteristic for *cis* and *trans* isomers of olefins [11]. These bands have a clearly expressed fine structure which indicates the presence of a more extended conjugation chain than for compounds **11**. Such a phenomenon is characteristic of a conjugated system formed from 7-12 double bonds [11]. An interesting result was also obtained on studying the electronic spectra of N-methylisoquinoquinazoline perchlorates **8b** and **7**. The absorption curve of compound **7** differed significantly in shape from the absorption curves of the dehydrogenation products **10** (see Fig. 1), which indicates a different distribution of electron density in the molecule. Evidently in the case of the N-substituted isoquinoquinazoline **7**, as in the isomeric compound **8b**, a structure is formed with a high degree of aromaticity for the isoquinoquinazoline fragment, i.e. with retention of the aromatic sextet of the benzene ring of the isoquinoline fragment, in difference to compound **10**, which has an *o*-quinonoid structure.

In connection with the abovementioned it was also interesting to carry out the dehydrogenation of the hydrohalides of 7-benzyl-7,12-dihydro-5H-isoquino[2,3-*a*]quinazolin-5-ones **12a-c** which are isomeric to salts **9**. However on heating in nitrobenzene salts **12a-c** readily rearranged into **9a-c** hydrohalides, and the products of the dehydrogenation reaction proved to be 6-benzyl-13H-isoquino[3,2-*b*]quinazolin-13-ones **10**, obtained previously also by thermal rearrangement in the absence of solvent [12].

It was noted previously that 6-methyl-5-oxo-5H,6H-isoquino[2,3-*a*]quinazolinium perchlorate **8b** reacts readily with nucleophilic reagents at position 12 with the formation of addition products or fission of the isoquinoline ring [4]. Logically it was expected to display similar properties to the products of dehydrogenation of an isoquinoquinazoline of linear structure, compounds **7** and **10**. We studied the interaction of these compounds with $NaBH_4$. N-Methylisoquinoquinazoline perchlorate **7** reacts with $NaBH_4$ in ethanol at room temperature. The reaction occurs several minutes after adding the equivalent amount of reducing agent. The mixture of products obtained by us contained only traces of the hydride anion addition product, 6-methyl-5,11-dihydro-13H-isoquino[3,2-*b*]quinazolin-13-one (**13**, according to a ^{1}H NMR spectrum of the mixture), obtained previously [1] on interacting salt **6** with bases. 6-Benzylisoquinoquinazolines **10a,b** proved to be inert to the action of $NaBH_4$ in alcohols (ethanol, methanol) both at room temperature and on heating. Use of acetic acid or DMF as solvent led to a mixture of unidentified products. The interaction of salt **10c** with $NaBH_4$ in alcohols is also accompanied by the formation of a complex mixture of products.

EXPERIMENTAL

Melting points were determined on a Boetius type heating instrument and are not corrected. The IR spectra of compounds (KBr disks) were recorded on a Pye Unicam SP3-300 instrument. The ^{1}H and ^{13}C NMR spectra were recorded on a Varian Mercury 400 instrument (400 and 100 MHz respectively), internal standard was TMS. The UV spectra were recorded on a Specord M400 spectrophotometer in methanol. Mass spectra were obtained by HPLC on an AGILENT/100 Series instrument (CI, acetonitrile, 0.05% formic acid). A check on the progress of reactions and the purity of the compounds obtained was carried out with TLC on Silufol UV-254 plates.

6,11-Dihydro-13H-isoquino[3,2-*b*]quinazolin-13-one 2 was obtained by the method of [9], **5-methyl-13-oxo-6,13-dihydro-11H-isoquino[3,2-*b*]quinazolin-5-ium perchlorate 6** as in [1], **6-benzyl-6,11-dihydro-13H-isoquino[3,2-*b*]quinazolin-13-one hydrohalides 9a-c**, and **7-benzyl-7,12-dihydro-5H-isoquino[2,3-*a*]-quinazolin-5-one hydrohalides** as in [12].

11-Hydroxy-11H-isoquino[3,2-*b*]quinazoline-6,13-dione (4). Isoquino[3,2-*b*]quinazoline salt **2**·HBr (1-g, 3.04 mmol) was dissolved with heating in DMSO (5 ml). A 48% HBr solution (1 ml) was added and the mixture boiled for 10 min. A precipitate formed during boiling. The mixture was cooled, the solid substance was filtered off, and washed with a small quantity of DMSO and 2-propanol. Yield was 0.69 g (73%); mp 210-212°C (DMF). IR spectrum (thin film), ν, cm^{-1}: 3200 (OH), 1680 (br., C=O), 1595 (C=N), 1300 (C–O), 1050, 960, 767. UV spectrum, λ_{max}, nm ($\varepsilon\times10^{-3}$): 271 (61.62), 319 (51.68), 340 (52.67). Mass spectrum, *m/z* (I_{rel}, %): 279 $[M+1]^+$ (100), 261 $[M+1–H_2O]^+$ (10). ^{1}H NMR spectrum, δ, ppm (*J*, Hz): 8.29 (1H, d, ^oJ = 8.0, H-7); 8.05 (1H, d, ^oJ = 7.5, H-10); 7.92 (2H, m, H-2,4); 7.83 (1H, t, ^oJ = 7.5, H-3); 7.77 (1H, d, ^oJ = 7.5, H-1); 7.65 (2H, m, H-8,9); 7.37 (1H, br. s, OH); 7.15 (1H, s, H-11). ^{13}C NMR spectrum, δ, ppm: 178.25 (C-6); 160.53 (C-13); 146.71 (C-5a); 144.89 (C-4a); 140.13 (C-10a); 135.89, 135.84, 130.30, 129.97 (C-6a); 129.83, 129.70, 129.18, 127.39, 127.18, 122.49 (C-13a); 73.26 (C-11). Found, %: C 68.95; H 3.78; N 10.09. $C_{16}H_{10}N_2O_3$. Calculated, %: C 69.06; H 3.62; N 10.07.

2-[(4-Oxo-3,4-dihydro-2-quinazolinyl)carbonyl]benzoic Acid Hydrobromide (5). A mixture of salt **2**·HBr (1 g, 3.04 mmol) and 30% H_2O_2 (20 ml) was boiled for 2.5 h. In the process of boiling the solid substance almost completely dissolved and a new solid precipitated. The mixture was cooled, the solid was filtered off, washed with water and a small quantity of 2-propanol. Yield was 0.68 g (60%); mp 264-266°C (DMF). IR spectrum (thin film), ν, cm^{-1}: 3100 (br., OH, NH), 2960, 2500, 1700 (C=O), 1680 (C=O), 1650 (C=O), 1605 (C=N), 1440, 1190 (C–O), 765. ^{1}H NMR spectrum, δ, ppm (*J*, Hz): 13.15 (1H, br., OH); 12.54 (1H, s, NH); 8.18 (1H, d, ^oJ = 8.0, H-3); 7.98 (1H, d, ^oJ = 8.0, H-6), 7.66-7.78 (4H, m, Ar-H); 7.56 (2H, m, Ar-H). Mass spectrum, *m/z* (I_{rel}, %): 295 $[M+1]^+$ (100), 277 $[M+1-H_2O]^+$ (30). Found, %: Br 21.31; N 7.46. $C_{16}H_{10}BrN_2O_4$. Calculated, %: Br 21.30; N 7.47.

5-Methyl-13-oxo-5H,13H-isoquino[3,2-*b*]quinazolinium Perchlorate (7). A suspension of 5-methyl-isoquinoquinazoline perchlorate **6** (1 g, 2.76 mmol) in nitrobenzene (10 ml) was heated until all the solid had dissolved. The mixture was boiled for a further10 min. After cooling, the precipitated solid was filtered off, and washed with acetone.

6-Benzyl-13H-isoquino[3,2-*b*]quinazolin-13-one (10a). A. A suspension of 6-benzyl-6,11-dihydro-13H-isoquino[3,2-*b*]quinazolin-13-one hydrochloride (**9a)** (1 g, 2.67 mmol) in nitrobenzene (10 ml) was heated until all the solid substance had dissolved. The mixture was boiled for a further 5 min, and the solvent evaporated in vacuum. 2-Propanol (15 ml) was added to the dark-red residual oil. The resulting solid was filtered off and washed with 2-propanol.

B. The reaction was carried out analogously to method A using 7-benzyl-7,12-dihydro-5H-isoquino-[2,3-*a*]quinazolin-5-one hydrobromide **12a** (1 g, 2.67 mmol).

6-(3-Nitrobenzyl)-13H-isoquino[3,2-*b*]quinazolin-13-one (10b) was obtained by method A using the appropriate 3-nitrobenzylisoquinoquinazoline hydrochloride **9b.**

6-(2-Methylbenzyl)-13H-isoquino[3,2-*b*]quinazolin-13-one (10c) hydrobromide was obtained by method A using the appropriate 2-methylbenzylisoquinoquinazoline hydrochloride **9c**. After cooling the reaction mixture, a solid was precipitated, which was filtered off, and washed with acetone.

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