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TAUTOMERISM IN THE SERIES OF PYRROLO[2,3-*h*]-, -[3,2-*f*]-, -[2,3-*f*]-, -[3,2-*g*]-, AND -[3,2-*h*]QUINOLINES

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Molecules of pyrroloquinolines obtained from substituted 4-, 5-, 6-, and 7-aminoindoles and β-keto esters (acetoacetic, trifluoroacetoxy, and oxaloacetic esters) in quinoline and hydroxyquinoline forms have been investigated by semi-empirical calculations. On the basis of the experimental data and the calculated ^{1}H NMR spectra spectral criteria have been found for the assignment to one or the other tautomeric form.

Keywords: pyrroloquinolines, calculated and experimental ^{1}H NMR spectra of pyrroloquinolines, tautomerism in a series of pyrroloquinolines, quinoline and hydroxyquinoline forms, chemical shifts of protons of β-H hydroxyquinoline and β-H quinoline.

In the process of investigating new methods for the synthesis of condensed tricyclic nitrogen-containing heterosystems from substituted 4-, 5-, 6-, and 7-aminoindoles and various keto esters (acetoacetic, trifluoroacetoacetic, and oxaloacetic esters) we obtained a representative series (80 compounds) of hydroxy(oxo)-substituted pyrroloquinolines with different ring connection [1-16] (compounds **1-80** in Table 1). As an index of the structures of the compounds synthesized by ^{1}H NMR spectroscopy it was observed that they exist in quinolone (**a**) and hydroxyquinoline (**b**) forms depending on the ring connection and the nature of the substituents.

The differences in the chemical shifts for protons of the same type, especially β-H (form **b**) and β-H (form **a**) are characteristic for the tautomers **a** and **b** within limits of 0.5-1.0 ppm. Since these protons in forms such as **a** and **b** do not undergo exchange in practice, it is legitimate to assign signals for the different tautomers using calculated ^{1}H NMR spectra. According to the theoretical spectra, the signals of the β-H protons of the tautomers **a** fall within the range 5.58-7.61 ppm depending on the substituents, while the signal for the same protons in form **b** lie within the range 6.72-8.25 ppm. The experimental ^{1}H NMR spectra of pyrrolo-[3,2-*f*]quinolones with R = H and OMe confirm that they exist in the γ-quinolone form **a**. The formation of quinolone forms for 6-methyl-substituted (R = Me) pyrrolo[3,2-*f*]quinolines **6, 7, 15, 16, 19, 20, 24-29**, and also for pyrrolo[2,3-*h*]quinoline **3** depends on the nature of the substituent in the α-position of the pyridine ring. For example, α-methyl-substituted compounds **6, 7, 15,** and **16** exist in the γ-quinolone structure **a**, whereas the

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Table 1. Experimental (E) and Calculated (C) Chemical Shifts of the β-H Protons (Form **a**) and β-H Protons (Form **b**) in the ^{1}H NMR Spectra of the Tautomeric Structures of **1-80**

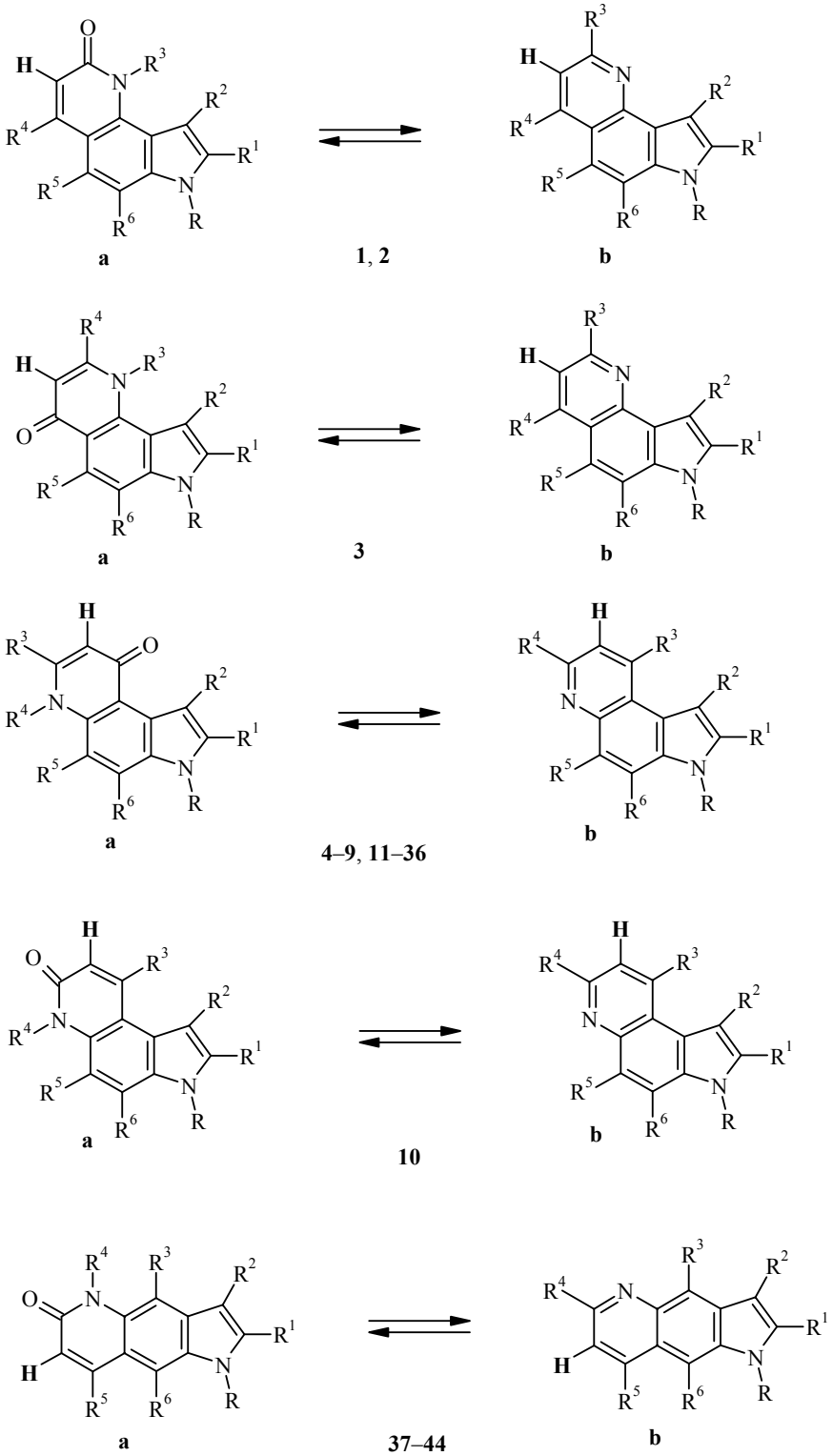


Table 1 (continued)

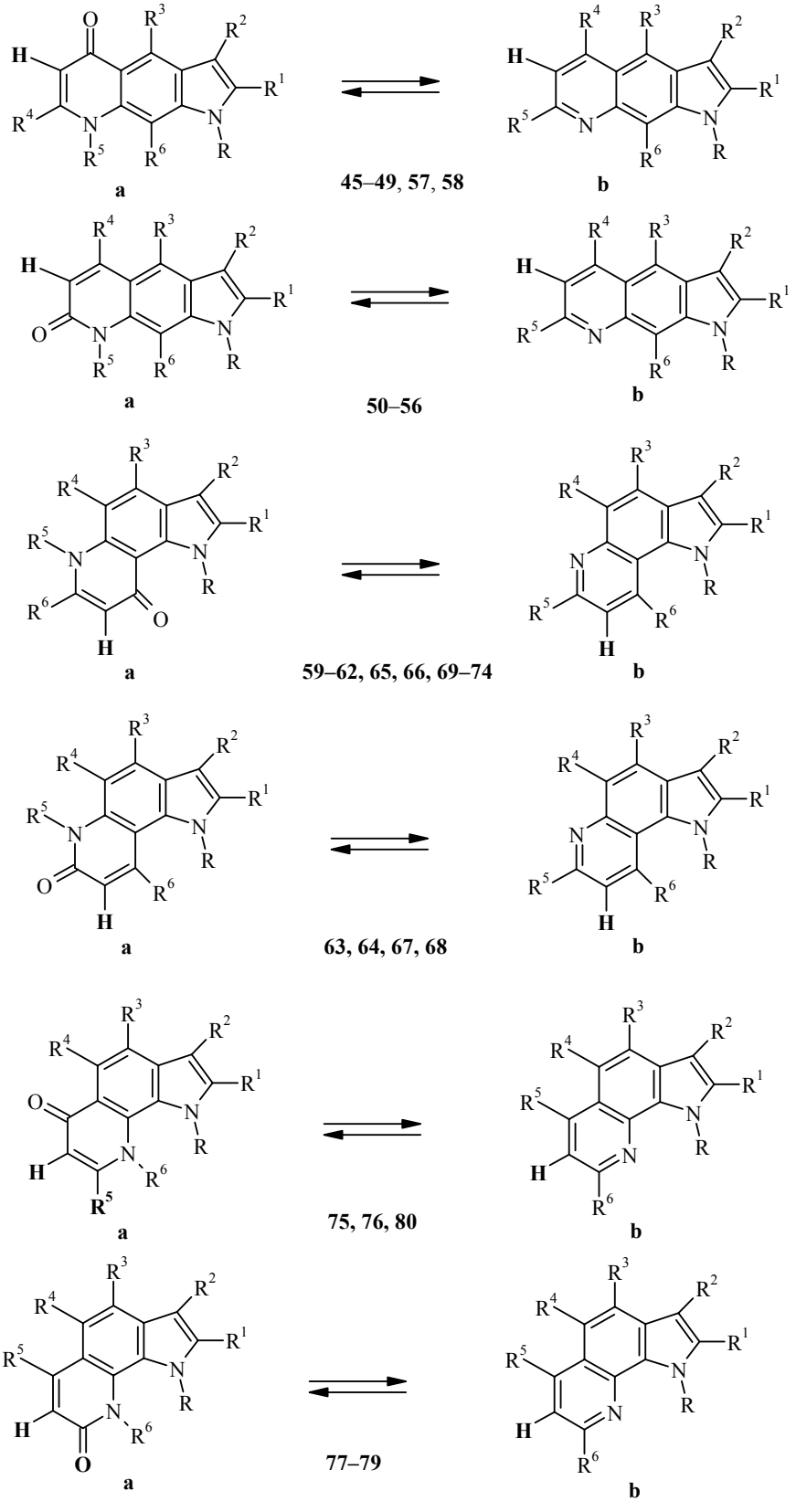


Table 1 (continued)

Tautomeric structures (α-, γ-quinoline/ α-, γ-hydroxyquinoline)								δ, ppm, β-H (**a**)/β-H (**b**)	
No.	R	R^1	R^2	R^3	R^4	R^5	R^6	E	C
1	2	3	4	5	6	7	8	9	10
				Pyrrolo[2,3-*h*]quinolines					
1a	H	Me	Me	H	CF_3	H	H	6.80	6.85
1b	H	Me	Me	OH	CF_3	H	H	—	7. 35
2a	Me	Me	Me	H	CF_3	H	H	6.80	6.84
2b	Me	Me	Me	OH	CF_3	H	H	—	7.35
3a	H	Me	Me	H	CO_2Et	H	H	6.64	6.75
3b	H	Me	Me	CO_2Et	OH	H	H	7.44	7.97
				Pyrrolo[3,2-*f*]quinolines					
4a	H	Me	Me	Me	H	H	H	5.90	5.82
4b	H	Me	Me	OH	Me	H	H	—	7.11
5a	Me	Me	Me	Me	H	H	H	5.82	5.82
5b	Me	Me	Me	OH	Me	H	H	—	7.09
6a	H	Me	Me	Me	H	Me	H	5.84	5.85
6b	H	Me	Me	OH	Me	Me	H	—	7.08
7a	Me	Me	Me	Me	H	Me	H	5.82	5.85
7b	Me	Me	Me	OH	Me	Me	H	—	7.06
8a	Me	Me	Me	Me	Me	Me	H	—	5.56
8b	Me	Me	Me	OMe	Me	Me	H	6.72	6.85
9a	H	Me	Me	Me	H	H	Me	5.77	5.82
9b	H	Me	Me	OH	Me	H	Me	—	7.09
10a	H	Me	Me	Me	H	H	H	6.29	6.52
10b	H	Me	Me	Me	OH	H	H	—	6.83
11a	H	Me	H	Me	H	H	H	5.82	5.90
11b	H	Me	H	OH	Me	H	H	—	7.15
12a	H	Me	Me	Me	H	OMe	H	5.84	5.85
12b	H	Me	Me	OH	Me	OMe	H	—	7.16
13a	H	Ph	H	Me	H	H	H	5.93	5.90
13b	H	Ph	H	OH	Me	H	H	—	7.12
14a	Me	Ph	H	Me	H	H	H	5.93	5.90
14b	Me	Ph	H	OH	Me	H	H	—	7.10
15a	H	Ph	H	Me	H	Me	H	6.00	5.85
15b	H	Ph	H	OH	Me	Me	H	—	7.09
16a	Me	Ph	H	Me	H	Me	H	6.00	5.86
16b	Me	Ph	H	OH	Me	Me	H	—	7.07
17a	H	Me	Me	CF_3	H	H	H	7.18	7.14
17b	H	Me	Me	OH	CF_3	H	H	—	7.73
18a	Me	Me	Me	CF_3	H	H	H	7.19	7.14
18b	Me	Me	Me	OH	CF_3	H	H	—	7.71
19a	H	Me	Me	CF_3	H	Me	H	—	7.14
19b	H	Me	Me	OH	CF_3	Me	H	7.64	7.69
20a	Me	Me	Me	CF_3	H	Me	H	—	7.14
20b	Me	Me	Me	OH	CF_3	Me	H	7.84	7.67
21a	Me	Me	Me	CF_3	H	OMe	H	7.21	7.14
21b	Me	Me	Me	OH	CF_3	OMe	H	—	7.77
22a	H	Ph	H	CF_3	H	H	H	7.19	7.14
22b	H	Ph	H	OH	CF_3	H	H	—	7.74
23a	Me	Ph	H	CF_3	H	H	H	7.22	7.14
23b	Me	Ph	H	OH	CF_3	H	H	—	7.72
24a	H	Ph	H	CF_3	H	Me	H	—	7.14
24b	H	Ph	H	OH	CF_3	Me	H	7.68	7.70

Table 1 (continued)

1	2	3	4	5	6	7	8	9	10
25a	Me	Ph	H	CF_3	H	Me	H	—	7.14
25b	Me	Ph	H	OH	CF_3	Me	H	7.40	7.68
26a	H	Me	Me	CO_2Et	H	Me	H	6.60	6.73
26b	H	Me	Me	OH	CO_2Et	Me	H	7.60	8.05
27a	Me	Me	Me	CO_2Et	H	Me	H	6.60	6.73
27b	Me	Me	Me	OH	CO_2Et	Me	H	7.80	8.03
28a	H	Ph	H	CO_2Et	H	Me	H	6.74	6.73
28b	H	Ph	H	OH	CO_2Et	Me	H	7.75	8.06
29a	Me	Ph	H	CO_2Et	H	Me	H	6.73	6.73
29b	Me	Ph	H	OH	CO_2Et	Me	H	7.94	8.04
30a	H	Me	Me	CO_2Et	H	OMe	H	7.03	6.73
30b	H	Me	Me	OH	CO_2Et	OMe	H	—	8.15
31a	H	Me	H	CO_2Et	H	H	H	6.66	6.73
31b	H	Me	H	OH	CO_2Et	H	H	—	8.12
32a	Me	Me	H	CO_2Et	H	H	H	6.67	6.73
32b	Me	Me	H	OH	CO_2Et	H	H	—	8.10
33a	H	Ph	H	CO_2Et	H	H	H	6.72	6.73
33b	H	Ph	H	OH	CO_2Et	H	H	—	8.10
34a	Me	Ph	H	CO_2Et	H	H	H	6.72	6.73
34b	Me	Ph	H	OH	CO_2Et	H	H	—	8.08
35a	H	Me	Me	CO_2Et	H	H	H	6.60	6.73
35b	H	Me	Me	OH	CO_2Et	H	H	—	8.09
36a	Me	Me	Me	CO_2Et	H	H	H	6.60	6.73
36b	Me	Me	Me	OH	CO_2Et	H	H	—	8.07
				Pyrrolo[2,3-*g*]quinolines					
37a	H	Me	Me	H	H	CF_3	H	6.70	6.85
37b	H	Me	Me	H	OH	CF_3	H	—	7.41
38a	Me	Me	Me	H	H	CF_3	H	6.73	6.85
38b	Me	Me	Me	H	OH	CF_3	H	—	7.42
39a	Me	Me	Me	H	Me	CF_3	H	6.86	6.82
39b	Me	Me	Me	H	OMe	CF_3	H	—	7.42
40a	Me	Ph	H	H	Me	CF_3	H	6.89	6.82
40b	Me	Ph	H	H	OMe	CF_3	H	—	7.42
41a	H	Ph	H	H	H	CF_3	H	6.84	6.85
41b	H	Ph	H	H	OH	CF_3	H	—	7.41
42a	Me	Ph	H	H	H	CF_3	H	6.77	6.85
42b	Me	Ph	H	H	OH	CF_3	H	—	7.39
43a	Me	Ph	$COCF_3$	H	H	CF_3	H	6.86	6.85
43b	Me	Ph	$COCF_3$	H	OH	CF_3	H	—	7.39
44a	H	Me	Me	H	H	CF_3	Me	6.86	6.84
44b	H	Me	Me	H	OH	CF_3	Me	—	7.33
				Pyrrolo[3,2-*g*]quinolines					
45a	Me	Me	Me	H	Me	H	H	5.81	5.73
45b	Me	Me	Me	H	OH	Me	H	—	6.99
46a	H	Me	Me	H	Me	H	OMe	5.79	5.73
46b	H	Me	Me	H	OH	Me	OMe	—	7.06
47a	Me	Me	Me	H	Me	H	OMe	5.90	5.73
47b	Me	Me	Me	H	OH	Me	OMe	6.60	7.06
48a	Me	Me	Me	H	Me	Me	OMe	—	5.58
48b	Me	Me	Me	H	OMe	Me	OMe	6.77	6.85
49a	Me	Me	Me	H	CF_3	H	H	6.80	7.16

Table 1 (continued)

1	2	3	4	5	6	7	8	9	10
49b	Me	Me	Me	H	OH	CF_3	H	—	7.61
50a	H	Me	Me	H	Me	H	H	6.22	6.41
50b	H	Me	Me	H	Me	OH	H	—	6.71
51a	Me	Me	Me	H	CF_3	H	H	6.74	6.85
51b	Me	Me	Me	H	CF_3	OH	H	—	7.35
52a	H	Me	Me	H	CF_3	H	OMe	6.79	6.85
52b	H	Me	Me	H	CF_3	OH	OMe	—	7.41
53a	Me	Me	Me	H	CF_3	H	OMe	6.80	6.85
53b	Me	Me	Me	H	CF_3	OH	OMe	—	7.41
54a	Me	Me	Me	H	CF_3	Me	OMe	6.85	6.82
54b	Me	Me	Me	H	CF_3	OMe	OMe	—	7.44
55a	H	Me	Me	H	CF_3	H	Me	6.78	6.85
55b	H	Me	Me	H	CF_3	OH	Me	—	7.31
56a	Me	Me	Me	H	CF_3	H	Me	6.79	6.85
56b	Me	Me	Me	H	CF_3	OH	Me	—	7.31
57a	H	Me	Me	H	CO_2Et	H	OMe	6.64	6.75
57b	H	Me	Me	H	OH	CO_2Et	OMe	7.46	8.03
58a	Me	Me	Me	H	CO_2Et	H	OMe	—	6.75
58b	Me	Me	Me	H	OH	CO_2Et	OMe	7.45	8.03
					Pyrrolo[2,3-*f*]quinolines				
59a	H	Me	Me	H	H	H	Me	6.02	6.17
59b	H	Me	Me	H	H	Me	OH	—	7.13
60a	Me	Me	Me	H	H	H	Me	5.94	6.17
60b	Me	Me	Me	H	H	Me	OH	—	7.09
61a	H	Me	Me	H	H	H	CF_3	7.04	7.61
61b	H	Me	Me	H	H	CF_3	OH	—	7.73
62a	Me	Me	Me	H	H	H	CF_3	6.80	7.61
62b	Me	Me	Me	H	H	CF_3	OH	—	7.69
63a	H	Me	Me	H	H	H	Me	6.33	6.66
63b	H	Me	Me	H	H	OH	Me	—	6.85
64a	H	Me	Me	H	Me	H	Me	6.32	6.66
64b	H	Me	Me	H	Me	OH	Me	—	6.81
65a	H	Me	Me	H	Me	H	Me	6.02	6.18
65b	H	Me	Me	H	Me	Me	OH	—	7.09
66a	Me	Me	Me	H	Me	H	Me	5.94	6.18
66b	Me	Me	Me	H	Me	Me	OH	—	7.06
67a	H	Me	Me	H	Me	H	CF_3	6.99	7.10
67b	H	Me	Me	H	Me	OH	CF_3	—	7.43
68a	H	Me	Me	H	OMe	H	CF_3	7.00	7.10
68b	H	Me	Me	H	OMe	OH	CF_3	—	7.53
69a	H	Me	Me	H	H	H	CO_2Et	6.75	7.20
69b	H	Me	Me	H	H	CO_2Et	OH	—	8.09
70a	Me	Me	Me	H	H	H	CO_2Et	6.67	7.20
70b	Me	Me	Me	H	H	CO_2Et	OH	—	8.05
71a	Me	Me	Me	H	Me	H	CO_2Et	6.68	7.20
71b	Me	Me	Me	H	Me	CO_2Et	OH	7.71	8.02
72a	H	Me	Me	H	Me	H	CO_2Et	6.75	7.20
72b	H	Me	Me	H	Me	CO_2Et	OH	7.72	8.05
73a	H	Me	Me	H	OMe	H	CO_2Et	6.73	7.20
73b	H	Me	Me	H	OMe	CO_2Et	OH	—	8.15
74a	Me	Me	Me	H	OMe	H	CO_2Et	6.65	7.20
74b	Me	Me	Me	H	OMe	CO_2Et	OH	—	8.11

Table 1 (continued)

1	2	3	4	5	6	7	8	9	10
					Pyrrolo[3,2-*h*]quinolines				
75a	H	Me	Me	H	H	Me	H	5.85	5.73
75b	H	Me	Me	H	H	OH	Me	—	7.06
76a	Me	Me	Me	H	H	Me	H	—	5.73
76b	Me	Me	Me	H	H	OH	Me	6.65	7.04
77a	H	Me	Me	H	H	CF_3	H	6.74	6.85
77b	H	Me	Me	H	H	CF_3	OH	—	7.41
78a	Me	Me	Me	H	H	CF_3	H	—	6.84
78b	Me	Me	Me	H	H	CF_3	OH	7.08	7.39
79a	Me	Me	Me	H	H	CF_3	Me	—	6.82
79b	Me	Me	Me	H	H	CF_3	OMe	7.21	7.42
80a	Me	Me	Me	H	H	CO_2Et	H	—	6.75
80b	Me	Me	Me	H	H	OH	CO_2Et	7.48	8.01

α-trifluoromethyl-, ethoxycarbonyl-substituted pyrroloquinolines **3**, **19, 20**, **24-29** are observed predominantly or exclusively in the γ-hydroxyquinoline form **b**. The authors of the analyzed work also noted that the formation of form **a** for these compounds is sterically hindered by the *ortho*-methyl (*ortho*-C-methyl for pyrrolo-[2,3-*h*]quinoline) relative the N–H substituent.

According to the ^{1}H NMR spectra in DMSO-d_6 solution, pyrrolo[2,3-*f*]quinolines, independent of the substituent at position **5**, exist as tautomers of structure **a**, apart from compounds **71** and **72** (R = Me) which consist of mixtures of tautomers **a** and **b** in equal proportions. This fact, and the existence of pyrrolo-[3,2-*h*]quinolines **76**, **78-80** (R' = Me) exclusively in form **b**, is explained as for the pyrrolo[3,2-*f*]quinolines above by the relative steric requirements of the *ortho*- and *peri*-methyl groups for the pyrrolo[3,2-*h*]quinoline group relative to the N–H group for formation of the γ-quinoline structures **a**. For the pyrrolo[3,2-*g*]quinolines **47**, **48**, **57**, and **58** the given tendency is also continued for the models with R = OMe, i.e., these compounds exist predominantly in the tautomeric form **b**.

It seemed of interest to analyze the correspondences of regularities found for pyrroloquinolines on related structures. To this end we compared the experimental ^{1}H NMR spectra of compounds **81-90** cited in papers [17-22] with those calculated by us (Table 2). It is seen from the Table that the chemical shifts of the β-H protons for structures **82-86** and **88-90** agree well with the correct conclusions of tautomeric form **a** drawn by the authors.

The authors assigned form **a** for compound **87** with the signal of the β-H proton in the ^{1}H NMR spectrum 5.88 ppm. However, according to the calculated spectra, this chemical shift corresponds better to the β-H of the hydroxyquinoline form **b**: δ 5.13 β-H (**a**) and δ 6.01 ppm β-H (**b**).

The spectrum of compound **87** has the signal of the characteristic proton is observed at 6.60 ppm, which according to the calculations (6.75 – β-H (**a**) and 7.89 ppm for β-H (**b**)) corresponds to form **a,** which contradicts the structure **81b** suggested by the authors.

The rules revealed in the analysis of the structures **1-7**, **9-38**, **41-47**, **49-53**, **55-78**, and **80** can be used to indicate the structures of compounds obtained by methylation of a series of pyrroloquinolines, in which alternative formation of O- and N-methyl derivatives is possible. The authors of papers [12-14] studied the behavior of some compounds of γ- and α-quinoline nature in methylation reactions. According to their results, methylation of α-pyrrolo[2,3-*g*]- and -[3,2-*g*]quinolones **37**, **38**, **41**, **42**, **52**, and **53** gave compounds **39**, **40**, and **54** with signals of the β-H protons in the regions 6.86, 6.89, and 6.85 ppm, respectively, which corresponds to methylation, according to the calculated ^{1}H NMR spectra (β-H (**a**) δ 6.82 and β-H (**b**) δ 7.42 ppm), leading to the

Table 2. Experimental (E) and Calculated (C) Chemical Shifts of β-H (form **a**) and β-H (form **b**) Protons in the ^{1}H NMR Spectra of Tautomeric Structures of Compounds **81-90** Obtained by the Authors [17-22].

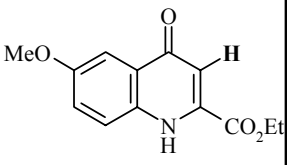
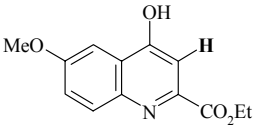
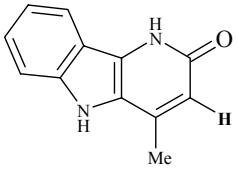
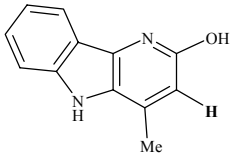
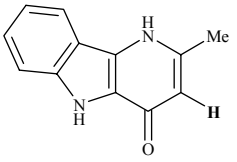
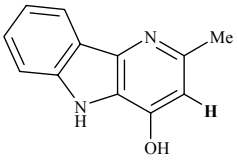
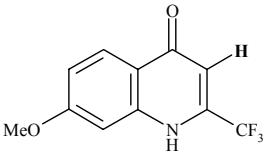
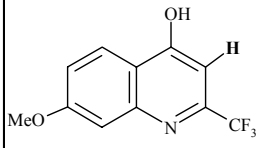
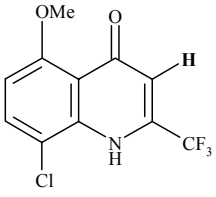
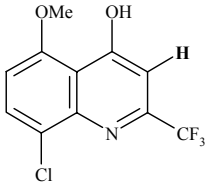
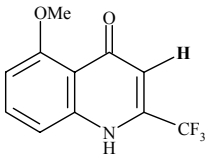
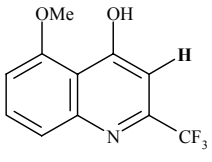
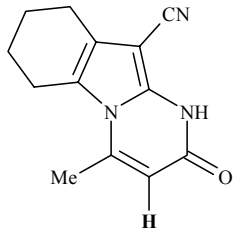
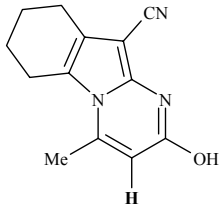
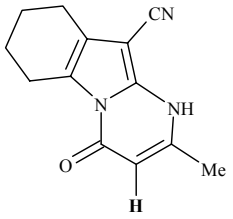
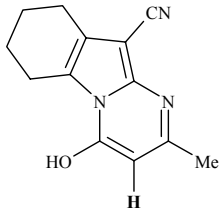
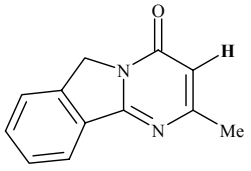
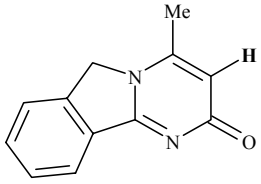
Tautomeric structure (α-, γ-quinolines)	δ, ppm, β-H (**a**)		Tautomeric structure (α-, γ-quinolines)	δ, ppm, β-H (**b**)	
	E	C		E	C
1	2	3	4	5	6
 **81a**	6.60	6.75	 **81b**	—	7.89
 **82a**	6.29	6.29	 **82b**	—	6.71
 **83a**	6.02	6.02	 **83b**	—	6.98
 **84a**	6.93	7.16	 **84b**	—	7.53
 **85a**	7.23	7.12	 **85b**	—	7.45
 **86a**	7.04	7.12	 **86b**	—	7.24

Table 2 (continued)

1	2	3	4	5	6
 **87a**	—	5.13	 **87b**	5.88	6.01
 **88a**	5.40	5.29	 **88b**	—	5.82
 **89**	6.07	6.07			
 **90**	5.89	5.70			

N-methylated tautomers **a**. In the same papers it was proposed that **8**, **48**, and **79** pyrroloquinolones with *peri*-methyl groups were formed. However the chemical shifts of the β-H protons (for compounds **8**, **48**, and **79** experimental values: 6.72, 6.77, and 7.21, calculated values 6.85, 6.85, and 7.42 ppm respectively) in the ^{1}H NMR spectra indicated that they were in tautomeric form **b**. Evidently substituents *peri* to the NH group in γ-quinolones **6**, **7**, **46**, and **47** and the α-quinolones **77** and **78** under the conditions of the methylation reaction facilitate the formation of the sterically more suitable O-methyl-substituted isomers **8b**, **48b**, and **79b** in place of the generally expected structures **8a**, **48a**, and **79a**.

Thus, on the basis of a comparative analysis of the experimental and calculated ^{1}H NMR spectra we propose the possibility of determining the tautomeric forms for pyrroloquinolines with α- and γ-hydroxy(oxo)-substituents in the pyridine ring and the structures of related analogs. This method can be used to indicate the direction of methylation (O or N substitution) for α- and γ-hydroxy(oxo)quinolines.

EXPERIMENTAL

Experimental ^{1}H NMR spectra of DMSO-d_6 solutions with TMS as internal standard were recorded with a Bruker DRX-500 spectrometer (pyrroloquinolines **1-3**, **5-9**, **12-45**, **47-49**, **51-58**, **60-62**, **64**, **67-80**), Varian-100XL (pyrroloquinolines **4**, **10**, **11**, **50**, **59**), Bruker AC-200P (pyrroloquinolines **46**, **65**, **66**), Bruker AMX-500 (compound **81**), JNM-4H-100 (compounds **82**, **83**), Varian HA-100 (compounds **84-86**), Jeol-C-60HL (compounds **87**, **88**), ZKR-60 (compound **89**), and Bruker CPX-500 (compound **90** in $CDCl_3$) [1-22]. Calculated ^{1}H NMR spectra were obtained using the program *ACD/LABS HNMR Spectrum Generator: Chemsketch Window.*

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