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SYNTHESIS OF PYRROLO[2,3-*g*]- AND PYRROLO[3,2-*f*]QUINOLINES FROM 5-AMINO-2,3-DIMETHYL- AND 1,2,3-TRIMETHYLINDOLES WITH 4,4,4-TRIFLUOROACETOACETIC ESTER

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It has been shown that the condensation reaction of 5-amino-2,3-dimethyl- and 1,2,3-trimethylindoles with 4,4,4-trifluoroacetoacetic ester occurs either via the trifluoromethylcarbonyl or the ester group depending upon the conditions under which it is carried out. Under thermal conditions (refluxing in diphenyl) the enaminocrotonates formed are readily converted to pyrrolo[3,2-f]quinolines and the amides cyclize in trifluoroacetic acid at 78°C to give pyrrolo[2,3-g]quinolines.

Keywords: 5-amino-2,3-dimethylindole, 5-amino-1,2,3-trimethylindole, pyrrolo[2,3-*g*]quinoline, pyrrolo[3,2-*f*]quinoline, ethyl 4,4,4-trifluoroacetoacetate.

Continuing our development of methods for the synthesis of trifluoromethyl-substituted pyrroloquinolines [1-4] as potentially biologically active compounds we have studied the reactions of 5-amino-2,3-dimethyl- and -1,2,3-trimethylindoles with 4,4,4-trifluoroacetoacetate ester.

In contrast to acetoacetate ester [5] we have found that its analog fully fluorinated at the acetyl group reacts with 5-amino-2,3-dimethyl- and -1,2,3-trimethylindoles **1, 2** under the same conditions (refluxing in absolute benzene with traces of glacial acetic acid) to give reaction products involving not only the carbonyl but also the carbethoxy group (principally).

We have also found that, when the reaction is carried out in absolute benzene at 10-15°C in the presence of a dehydrating agent (ignited potassium sulfate), both amine **1** and **2** give the enaminocrotonates **3** (40%) and **4** (50%) as the sole products. It should be noted that the formation of both the amides and the enaminocrotonates involving the aminoindole **2** occur for a shorter time and in higher yields and this apparently points to some increase in the basicity of the amine due to the effect of the N-methyl group.

The ^{1}H NMR spectrum of compound **3** shows a triplet and quadruplet signal for the protons of the ethoxy group, singlets for the 2-, 3-CH_3, H_{vinyl}, N–H_{amine}, H-4, and H-1 protons and two doublets for the H-6 and H-7 protons with J = 7 Hz (the spectroscopic characteristics of the compounds prepared are given in Table 1).

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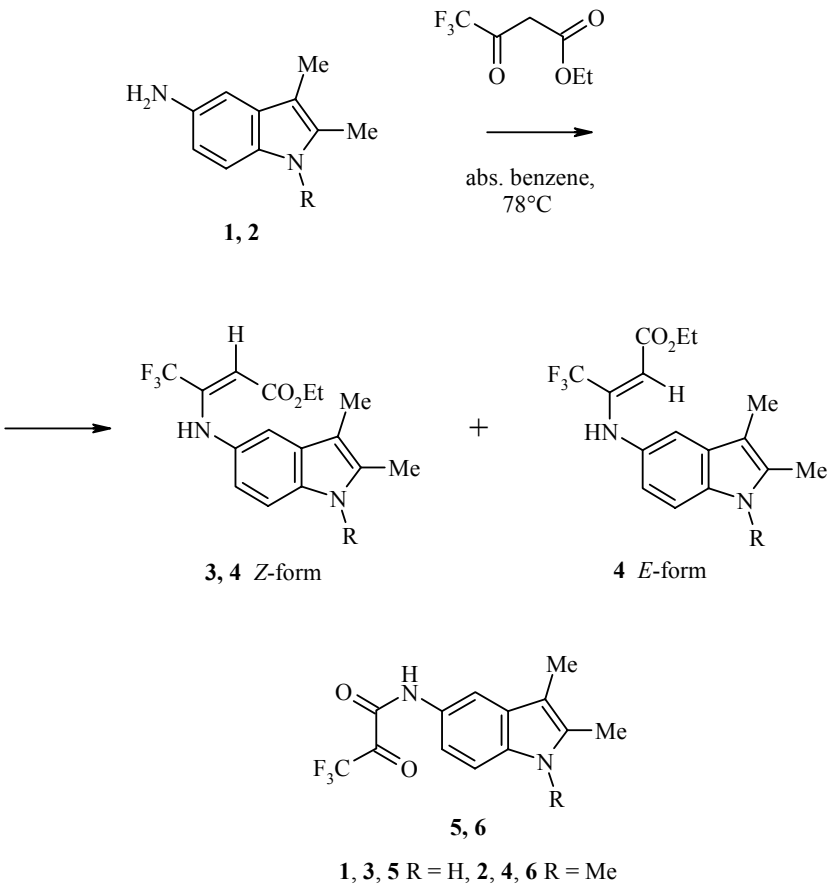
TABLE 1. Spectroscopic Characteristics of Compounds **3, 4, 7-10, 14**

Compound	UV spectrum		^{1}H NMR spectrum, δ, ppm (*J*, Hz)	Mass spectrum, *m/z* (I_{rel}, %)
	λ_{max}, nm	log ε		
***Z*-3**	208 sh 233 294	4.31 4.44 4.39	1.24 (3H, t, *J* = 7, $OCH_2\underline{CH_3}$); 2.12 (3H, s, 3-CH_3); 2.31 (3H, s, 2-CH_3); 4.14 (2H, q, *J* = 7, $O\underline{CH_2}CH_3$); 5.25 (1H, s, H vinyl); 6.85 (1H, d, *J* = 7, H-6); 7.18 (1H, s, H-4); 7.19 (1H, d, *J* = 7, H-7); 9.70 (1H, s, 5-NH); 10.71 (1H, s, H-1)	326 $[M]^+$ (100), 280 (70), 279 (42), 265 (18), 257 (32), 253 (43), 252 (84), 251 (73), 238 (31), 237 (45), 231 (26), 211 (23), 183 (36), 169 (15), 144 (47), 143 (44), 128 (11), 115 (31), 91 (15), 77 (18), 69 (21)
***E*-4**	208 235 294	4.29 4.41 4.35	1.08 (3H, t, *J* = 7, $OCH_2\underline{CH_3}$); 2.16 (3H, s, 3-CH_3); 2.33 (3H, s, 2-CH_3); 3.67 (3H, s, 1-CH_3); 3.93 (2H, q, *J* = 7, $O\underline{CH_2}CH_3$); 4.78 (1H, s, H vinyl); 6.90 (1H, d, *J* = 7, H-6); 7.22 (1H, s, H-4); 7.30 (1H, d, *J* = 7, H-7); 8.75 (1H, s, 5-NH)	340 $[M]^+$ (100), 295 (26), 294 (67), 293 (35), 279 (18), 271 (31), 267 (18), 266 (28), 265 (40), 252 (22), 251 (29), 231 (11), 225 (10), 198 (13), 197 (21), 183 (11), 158 (47), 157 (19), 156 (12), 143 (17), 115 (16), 113 (10), 91 (8), 69 (10)
***Z*-4**			1.23 (3H, t, *J* = 7, $OCH_2\underline{CH_3}$); 2.16 (3H, s, 3-CH_3); 2.33 (3H, s, 2-CH_3); 3.67 (3H, s, 1-CH_3); 4.15 (2H, q, *J* = 7, $O\underline{CH_2}CH_3$); 5.25 (1H, s, H vinyl); 6.90 (1H, d, *J* = 7, H-6); 7.22 (1H, s, H-4); 7.30 (1H, d, *J* = 7, H-7); 9.70 (1H, s, 5-NH)	
7	220 241 286 345	4.47 4.43 4.27 3.98	2.41 (3H, s, 2-CH_3); 2.56 (3H, s, 1-CH_3); 7.18 (1H, s, H-8); 7.55 (1H, d, *J* = 8, H-5); 7.75 (1H, d, *J* = 8, H-4); 11.42 (1H, s, H-6); 11.63 (1H, s, H-3)	280 $[M]^+$ (100), 279 (85), 265 (20), 140 (11), 115 (10), 91 (8), 78 (11), 77 (15), 75 (10), 69 (30), 63 (13), 51 (11), 41 (14), 40 (12), 39 (16)
8	222 244 290 351	4.52 4.44 4.31 4.03	2.42 (3H, s, 2-CH_3); 2.59 (3H, s, 1-CH_3); 3.80 (3H, s, 3-CH_3); 7.19 (1H, s, H-8); 7.62 (1H, d, *J* = 8, H-5); 7.95 (1H, d, *J* = 8, H-4); 11.62 (1H, s, H-6)	294 $[M]^+$ (100), 293 (71), 280 (15), 279 (56), 207 (8), 140 (8), 135 (11), 69 (23), 56 (9)
9	235 273 357	4.63 4.05 4.30	2.18 (3H, s, 3-CH_3); 2.39 (3H, s, 2-CH_3); 6.70 (1H, s, H-7); 7.30 (1H, s, H-9); 7.52 (1H, s, H-4); 10.83 (1H, s, H-1); 11.85 (1H, s, H-5)	280 $[M]^+$ (100), 279 (73), 265 (40), 251 (9), 69 (12)
10	238 273 357	4.45 3.89 4.06	2.22 (3H, s, 3-CH_3); 2.40 (3H, s, 2-CH_3); 3.70 (3H, s, 1-CH_3); 6.73 (1H, s, H-7); 7.93 (1H, s, H-9); 7.51 (1H, s, H-4); 11.77 (1H, s, H-5)	294 $[M]^+$ (100), 293 (76), 279 (38), 265 (9), 250 (5), 69 (5)
14	238 273 357	4.70 4.15 4.41	2.29 (3H, s, 3-CH_3); 2.43 (3H, s, 2-CH_3); 3.73 (3H, s, 1-CH_3); 3.74 (3H, s, 5-CH_3); 6.86 (1H, s, H-7); 7.51 (1H, s, H-9); 7.58 (1H, s, H-4)	308 $[M]^+$ (100), 293 (27), 265 (8), 69 (6)



1, **3**, **5** R = H, **2**, **4**, **6** R = Me

The nature of the chemical shifts of the vinyl and amino protons (5.25 and 9.70 ppm) confirms the *Z*-structure of the enamine. The spectrum of the enaminocrotonate **4** is similar except in the absence of the H-1 proton signal and the presence of a singlet for the 1-CH_3 protons. It was also found that compound **4** exists both in the *Z*- and *E*-form (10%) as judged by the chemical shift of the N–H (8.75) and H_{vinyl} (4.78 ppm) group protons.

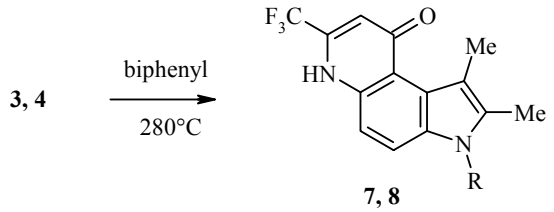
The mass spectra of compounds **3** and **4** show a molecular ion peak together with a strong peak for the $[M-46]^+$ ion which corresponds to loss of C_2H_5OH (characteristic of all indolylaminocrotonates) which gives the molecular ion corresponding to the pyrroloquinolones **7, 8** (structures identified on page 80) since the mass spectroscopic decomposition of the latter and the further pattern of decomposition of the enamines **3, 4** are identical.

The UV spectra of the fluorinated enaminocrotonates show two absorption bands which agree with the pattern of the spectra of the nonfluorinated analogs reported in the literature [5].

The predominant formation of the amides **5, 6** upon heating confirms their formation under conditions of thermodynamic control, evidently associated with the acceptor effect of the trifluoromethyl group which increases the activity of the ester group. Proof of the formation of amides **5, 6**, as before with other aminoindoles [3, 4] arises from the absence in their ^1H NMR spectra of the ethoxycarbonyl group protons. The overall integrated intensities of the protons in the spectra of the amides obtained correspond to the number of hydrogen atoms in their molecules. A clear assignment of the signals shown is hindered by the existence in solution (DMSO-d_6) of the amides in at least three forms: carbonyl, enol, and cyclic. This is confirmed by chromatography. In this connection the amides could not be characterized as individual compounds but their molecular weights measured mass spectrometrically corresponded to those calculated.

The aminocrotonates **3, 4** and amides **5, 6** were then introduced by us into cyclization reactions with the aim of studying the route of formation of the pyridine ring at the two alternative free *ortho* positions in the benzene ring as well as the effect of an N-methyl group on this process.

As in the case of the trifluoromethyl-substituted indolylaminocrotonates with a free β-position in the pyridine ring studied before [1, 3], the enamines **3, 4** are converted to the corresponding pyrroloquinolones **7, 8** when refluxed in diphenyl. Compound **8** is formed in high yield (85%) and needs a shorter reaction time than does the formation of the pyrroloquinolone **7** (73%).

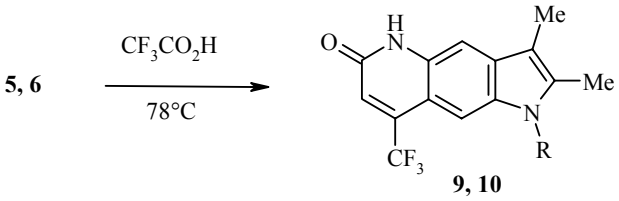


7 R = H, **8** R = Me

The presence in the ^{1}H NMR spectra of compounds **7, 8** of two aromatic proton doublets for H-4 and H-5 with J = 8 Hz confirms the angular structure of the pyrroloquinolones. This is also indicated by the low field shift (~0.5 ppm) of the 1-CH_3 signal, falling under the *peri*-effect of the carbonyl oxygen atom. The spectra also show singlets for the protons of the 2-, 3-CH_3 groups (for compound **8**), 2-CH_3 and H-3 (for compound **7**) and H-6 in both structures. The pyrroloquinolones **7, 8** are stable towards electronic impact. In their mass spectra the strongest peaks are the molecular ion peaks, $[M-1]^+$, and $[M-15]^+$. The fragment ion with *m/z* 69 points to the presence of the trifluoromethyl group in the molecules. The UV spectroscopic picture for compounds **7, 8** agrees well with literature data for the nonfluorinated analogs [5].

Hence, in enamines **3, 4** under thermal conditions, the closing of the pyridine ring does not depend on the presence of a free 6 position and the steric demand of the β-CH_3 group in the pyrrole fragment and it occurs at position 4 as the most reactive. Hence the target synthesis of the trifluoromethyl-containing pyrrolo[3,2-*f*]-quinolones can be achieved.

In contrast to the enaminocrotonates, the amides **5, 6** in trifluoroacetic acid at 78°C give a good yield of exclusively linear pyrroloquinolones **9, 10**. In this case the N-methyl group slightly activates the ring formation process. The yields of the pyrroloquinolones **9** and **10** are 61 and 83% respectively.



9 R = H, **10** R = Me

The linear conjugation of the rings in the pyrroloquinolones **9, 10** are shown by the ^{1}H NMR spectra which reveal two isolated proton signals for the benzene ring H-4 and H-9. The spectra also contain singlet signals for the protons of the 2-, 3-CH_3 groups and the H-5, H-7, and H-1 (for compound **9**) and 1-CH_3 (for **10**). The strongest peak in the mass spectra of both compounds **9** and **10** is the molecular ion peak which demonstrates their stability towards electron impact. The rather strong signal of ion $[M-H]^+$ and subsequent fission of a CO molecule to give the $[M-H-CO]^+$ ion is supporting evidence of the quinolone form of compounds **9, 10** in the gaseous phase. The *m/z* 69 ions indicate the presence of a trifluoromethyl group in the molecules.

The electronic spectra of the pyrroloquinolones **9, 10** show three absorption bands in the regions 235, 273, and 357 nm. According to quantum-chemical calculations [6] the short wavelength band can be assigned to an electronic transition in the pyrrole ring, that at 273 nm to the pyridone part of the molecule, and the long wavelength maximum to a π-π* transition in the benzene ring.

It should be noted that, in contrast to the linear pyrroloquinolines prepared from the enamino ketones [6] the UV spectra of the pyrroloquinolones with the same ring conjugation have an absorption band intensity at 273 nm which is lower than the short wavelength maximum and this is apparently related to some weakening of the conjugation in the quinolone ring.

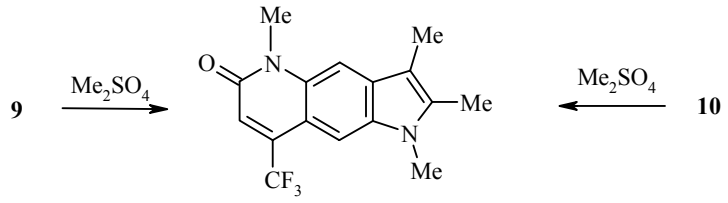
Thus the acid cyclization of the amides **5, 6** takes place at position 6 of the indole to give exclusively the pyrrolo[2,3-*g*]quinolones and this differs from the nonfluorinated analogs (prepared from diketene and aminoindoles) which give a mixture of linear and angular products [7] under similar conditions. By contrast to these the pyrrolo[3,2-*f*]quinolones with a methyl and trifluoromethyl substituent in a *peri*-position are problematic, probably because of the large steric demand of the trifluoromethyl group when compared with the methyl. These problems are not overcome by carrying out the cyclization in refluxing diphenyl. The single product (along with tarring) in this case is the linear structured pyrroloquinolone.

Our proposal is indirectly confirmed by semiempirical quantum-chemical calculations for molecules of the model pyrroloquinoline isomers **9, 11-13** carried out using the PM3 method and the Hyper Chem 5.0 program package.

The results obtained show (see Table 2) that the calculated heats of formation (ΔH_f) for the angular pyrroloquinolines **11, 13** are greater than the corresponding isomers **9, 12** with a linear conjugation of the rings while the difference in ΔH_f for the isomeric molecules **12, 13** with *peri*-methyl substituents is two times less that for the **9, 10** system where one of the *peri*-substituents is a CF_3 group (7.59 kcal/mol compared with 16.56 kcal/mol).

In addition, the hypothetical angular system **11** with a trifluoromethyl radical shows a change in the pyridine and pyrrole ring carbon atom valence angles linked to the *peri*-substituents. The results obtained fit in well in terms of the previously formulated concept of the route of formation of a pyrroloquinoline system based on substituted 5-aminoindoles [8] and add somewhat to its conclusions.

By studying the behavior of the pyrroloquinolines **9, 10** in a methylation reaction using dimethyl sulfate in aqueous acetone in the presence of KOH we have found that, under the same conditions, they undergo methylation and the pyrroloquinolone **10** already having one methyl group on the pyrrole nitrogen atom and compound **9** unsubstituted on the nitrogen atom to form exactly the same double methylated compound **14**. The formation of monomethylation products is excluded by the reaction conditions which use an excess of dimethyl sulfate and, evidently, involve identical acidities for the quinolone and pyrrole N–H groups. The latter proposal is confirmed by the rather similar ^{1}H NMR chemical shifts of these protons.



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The ^{1}H NMR spectrum of compound **14** shows signal for the four methyl groups, two of which (N–CH_3) have very similar chemical shifts. The benzene ring protons and 7-H also show three singlets. The mass spectrum of the pyrroloquinolone **14** has a molecular ion peak which is a maximum, as do the starting pyrroloquinolones **9, 10**. The UV spectrum of compound shows three characteristic absorption peaks and is virtually identical with that of the starting pyrroloquinolones **9, 10** indicating their identical structure.

TABLE 2. Bond Lengths and Angle Values in Compounds **9, 11-13**

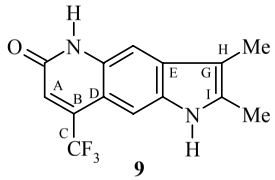
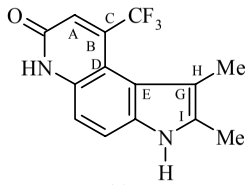
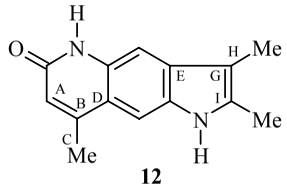
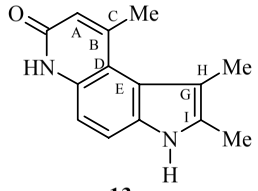
Compound*	ΔH_f, kcal/mol	Distance B–G, Å	Angles, deg					
			ABC	CBD	ABD	EGH	HGI	EGI
1	2	3	4	5	6	7	8	9
 **9**	-157.62		118.81	119.53	121.66	125.10	127.19	107.71
 **11**	-141.06	3.45	112.32	125.55	122.12	131.58	119.32	109.12

TABLE 2 (continued)

1	2	3	4	5	6	7	8	9
 **12**	–12.49		119.71	119.62	120.67	125.04	127.19	107.77
 **13**	-4.90	3.39	114.73	123.69	121.57	131.30	119.93	108.77

* Compounds **12** and **13** have been reported in [7], compound **11** is a hypothetical structure.

TABLE 3. Physicochemical Characteristics of the Compounds Prepared

Compound	Empirical formula	Found, % / Calculated, %			mp, °C	R_f*	Yield, %
		C	H	N			
3	$C_{16}H_{17}F_3N_2O_2$	58.73 / 58.89	5.20 / 5.25	32.6 / 32.6	76-77 (petroleum ether)	0.16; 0.47 (A)	40
4	$C_{17}H_{19}F_3N_2O_2$	59.91 / 59.99	5.58 / 5.63	34.0 / 34.0	67-68 (petroleum ether)	0.38; 0.75 (A)	50
7	$C_{14}H_{11}F_3N_2O$	59.87 / 60.00	3.92 / 3.96	28.0 / 28.0	>300 (toluene)	0.40 (B)	73
8	$C_{15}H_{13}F_3N_2O$	61.14 / 61.22	4.56 / 4.45	29.4 / 29.4	125-126 (toluene)	0.58 (B)	85
9	$C_{14}H_{11}F_3N_2O$	59.81 / 60.00	3.92 / 3.96	28.0 / 28.0	295-297 (ethanol)	0.44 (C)	61
10	$C_{15}H_{13}F_3N_2O$	61.09 / 61.22	4.41 / 4.45	29.4 / 29.4	>300 (ethanol+DMF)	0.46 (D)	83
14	$C_{16}H_{15}F_3N_2O$	62.23 / 62.33	4.86 / 4.90	30.8 / 30.8	239-240 (aq. ethanol)	0.67 (C)	78

* Solvent systems: benzene (A); benzene–ethyl acetate, 3:1 (B); 3:2 (C), 1:1 (D).

EXPERIMENTAL

^{1}H NMR spectra were recorded on a Bruker DRX 500 (500 MHz) instrument with DMSO-d_6 and TMS as internal standard. Mass spectra were obtained on a Finnigan MAT INCOS-50 mass spectrometer with direct introduction of the sample into the ion source and ionization energy of 70 eV. Electronic spectra were recorded on a Specord spectrophotometer using ethanol. Purification of the reaction products was carried out using column chromatography on Al_2O_3 (neutral I and II Brockmann activation grade). Monitoring of the course of the reaction and the purity of the products obtained was carried out using TLC on Silufol UV-254 plates.

The physicochemical characteristics of the compounds prepared are given in Table 3. Preparation of the starting aminoindoles **1, 2** has been reported in [9, 10].

Ethyl (*Z*)-3-[(2,3-Dimethyl-1H-indol-5-yl)amino]-4,4,4-trifluoro-2-butenoate (3) and 4,4,4-Trifluoro-3-oxobutanoic Acid N-(2,3-Dimethyl-1H-indol-5-yl)amide (5). A mixture of the aminoindole **1** (1.2 g, 0.01 mol) and ethyl 4,4,4-trifluoroacetoacetate (1.4 g, 0.01 mol) in absolute benzene (300 ml) was refluxed in the presence of a catalytic amount of glacial acetic acid for 30 h with a Dean–Stark apparatus. After all of the aminoindole had taken part in the reaction (chromatographic monitoring) the volume of the reaction mixture was reduced by distillation of benzene to 50 ml. Traces of enamine **3** were removed by passage of a solution heated to reflux in a mixture of hexane and benzene through a layer (2.5-3 cm) of aluminium oxide. The yield of compound **5** was 1.4 g. The product obtained was not a single compound according to the chromatographic data. The overall integrated areas of the protons in the ^{1}H NMR spectrum agreed with the amide structure. Mass spectrum, calculated: M = 298; found: M = 298.

Ethyl (*E*,*Z*)-4,4,4-Trifluoro-3-[(1,2,3-trimethyl-1H-indol-5-yl)amino]-2-butenoate (4) and 4,4,4-Trifluoro-3-oxobutanoic Acid N-(1,2,3-Trimethyl-1H-indol-5-yl)amide (6) were prepared and separated similarly from the aminoindole **2** (1.14 g, 6.55 mmol) and trifluoroacetoacetic ester (1.2 g, 6.55 mmol) (25 h). Traces of enamine **4** were removed similarly. The yield of compound **6** was 1.6 g. The overall integrated areas of the protons in the ^{1}H NMR spectrum agreed with the amide structure. Mass spectrum, calculated: M = 312; found: M = 312.

Ethyl (*Z*)-3-[(2,3-Dimethyl-1H-indol-5-yl)amino]-4,4,4-trifluoro-2-butenoate (3). A mixture of the aminoindole **1** (0.211 g, 1.32 mmol) and trifluoroacetoacetic ester (0.328 g, 1.72 mmol) in absolute benzene in

the presence of a catalytic amount of acetic acid and dehydrating agent (ignited $CaSO_4$) was stirred for 90 days at ~20°C (chromatographic control). At the end of the reaction the solution was filtered from calcium sulfate and the benzene was evaporated. It was purified by passing a solution in petroleum ether heated to reflux through a layer (1 cm) of aluminium oxide. Yield 0.42 g.

Ethyl (*E*,*Z*)-4,4,4-Trifluoro-3-[(1,2,3-trimethyl-1H-indol-5-yl)amino]-2-butenoate (4) was prepared and purified similarly from the aminoindole **2** (0.515 g, 2.96 mmol) and trifluoroacetoacetic ester (0.7 g, 3.8 mmol) for 80 days. Yield 0.508 g.

1,2-Dimethyl-7-trifluoromethyl-6,9-dihydro-3H-pyrrolo[3,2-*f*]quinolin-9-one (7) was prepared from the enamine **3** (0.296 g, 0.91 mmol) by heating in refluxing diphenyl for 25 min. At the end of the reaction (chromatographic monitoring) the still warm reaction mixture was poured into petroleum ether. The precipitated product was filtered off and repeatedly washed with hot petroleum ether to remove diphenyl. It was recrystallized from toluene. Yield 0.186 g.

1,2,3-Trimethyl-7-trifluoromethyl-6,9-dihydro-3H-pyrrolo[3,2-*f*]quinolin-9-one (8) was prepared similarly from the enamine **4** (0.48 g, 1.4 mmol) for 20 min and recrystallized from toluene. Yield 0.355 g.

2,3-Dimethyl-8-trifluoromethyl-5,6-dihydro-1H-pyrrolo[2,3-*g*]quinolin-6-one (9) was prepared from the amide **5** (1.4 g, 4.7 mmol) by heating in a ten-fold excess of trifluoroacetic acid for 1 h. At the end of the reaction (chromatographic monitoring) the reaction mass was poured into 12% aqueous ammonia with ice. The precipitate was filtered off, repeatedly washed with water, and dried in air. It was recrystallized from alcohol with activated carbon. Yield 0.81 g.

1,2,3-Trimethyl-8-trifluoromethyl-5,6-dihydro-1H-pyrrolo[2,3-*g*]quinolin-6-one (10) was prepared similarly from the amide **6** (1.6 g, 5.13 mmol) for 1 h. It was recrystallized from alcohol with DMF. Yield 0.97 g.

1,2,3,5-Tetramethyl-8-trifluoromethyl-5,6-dihydro-1H-pyrrolo[2,3-*g*]quinolin-6-one (14). The starting pyrroloquinolone was dissolved in aqueous 2-butanone and a ten-fold excess of dimethyl sulfate and KOH were added. The reaction mixture was refluxed to completion (chromatographic monitoring) and poured into water. The precipitate was filtered off, washed repeatedly with water, and dried in air.

A. From the pyrroloquinolone **9** (0.46 g, 6.3 mmol) (1.5 h) the yield was 0.392 g.

B. From the pyrroloquinolone **10** (0.5 g, 1.7 mmol) (3 h) the yield was 0.1 g.

REFERENCES

1. S. Ya. Yamashkin, G. A. Romanova, I. S. Romanova, and M. A. Yurovskaya, *Khim. Geterotsikl. Soedin.*, 1354 (2003).
2. S. Ya. Yamashkin, G. A. Romanova, I. S. Romanova, and M. A. Yurovskaya, *Khim. Geterotsikl. Soedin.*, 1202 (2003).
3. S. Ya. Yamashkin, G. A. Romanova, and M. A. Yurovskaya, *Reports of the Moscow University, Ser. 2, Chemistry* [in Russian], **45**, 6 (2004).
4. S. Ya. Yamashkin, G. A. Romanova, and M. A. Yurovskaya, *Reports of the Moscow University, Ser. 2, Chemistry* [in Russian], **45**, 12 (2004).
5. S. A. Yamashkin, L. G. Yudin, and A. N. Kost, *Khim. Geterotsikl. Soedin.*, 1570 (1981).
6. S. A. Yamashkin, O. B. Tomilin, and O. V. Boyarkina, *Khim. Geterotsikl. Soedin.*, 1525 (2003).
7. S. A. Yamashkin, L. G. Yudin, and A. N. Kost, *Khim. Geterotsikl. Soedin.*, 493 (1983).
8. S. A. Yamashkin, I. V. Trushkov, O. B. Tomilin, I. I. Terekhin, and M. A. Yurovskaya, *Khim. Geterotsikl. Soedin.*, 1223 (1998).
9. Z. J. Veidelek, *Collect. Czech. Chem. Commun.*, **22**, 1852 (1957).
10. B. E. Shaw and D. W. Woolley, *J. Am. Chem. Soc.*, **75**, 1877 (1953).