Regioselective reaction of *ortho*-piperidinobenzaldehydes with pyrazolone*

*A. Yu. Platonova,[a] E. V. Deeva,[a] O. A. Zimovets,[a] D. V. Shatunova,[a] O. S. El´tsov,[a] P. A. Slepukhin,[b] T. V. Glukhareva,[a] and Yu. Yu. Morzherin[a]**

[a]*Ural Federal University named after the First President of Russia B. N. El´tsin, 19 ul. Mira, 620002 Ekaterinburg, Russian Federation. E-mail: morzherin@mail.ustu.ru*

[b]*I. Ya. Postovsky Institute of Organic Synthesis, Ural Branch of the Russian Academy of Sciences, 22/20 ul. S. Kovalevskoy, 620219 Ekaterinburg, Russian Federation. E-mail: slepukhin@ios.uran.ru*

Cyclization of 2-(4-*R*-piperidino)benzaldehydes with 5-methyl-2-phenyl-2,4-dihydro-3*H*-pyrazol-3-one proceeding *via tert*-amino effect mechanism is stereoselective. Relative configuration of (3*R**,4a*S**,5*R**)-2,3,4,4a,5,6-hexahydro-1*H*-benzo[*c*]quinolizine was established on the base of NMR spectroscopy data and X-ray analysis.

Key words: *tert*-amino effect, *ortho*-(4-*R*-piperidino)benzaldehyde, pyrazolone, Knoevenagel reaction/condensation, diastereomers, benzo[*c*]quinolizine, NMR spectroscopy.

Quinoline systems attract the chemists´ attention owing to their biological activity.[1,2] One of the most efficient pathways towards these systems is three step synthesis from *ortho*-fluorobenzaldehydes. The key step in this synthesis is the cyclization involving *tert*-amino effect.[3—15]

The term *tert*-amino effect was proposed for some reactions of tertiary anilines bearing the double C=C bond in the *ortho*-position to the amino group, cyclization of which with active methylene compounds afforded novel C—C bond and serves for the synthesis of polyfused heterocyclic systems.[12—15] The possibility to synthesize a wide variety of heterocycles is of interest for the study of their biological activity.

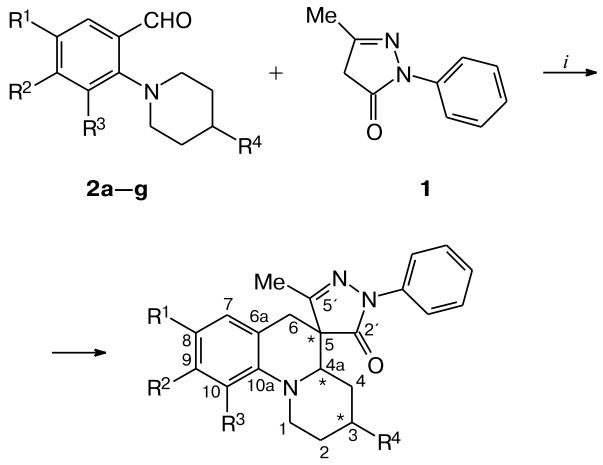
The present work is a continuation of our research in the reactions of *ortho*-(4-*R*-dialkylamino)benzaldehydes with unsymmetrical active methylene compounds.

Results and Discussion

It has previously been shown[16—17] that in the presence of the additional substituent in the dialkylamino group the reaction of 2-dialkylaminobenzaldehyde with the cyclic methylene active compounds is diastereoselective, the reaction of unsubstituted at the cyclic dialkylamino group 2-dialkylaminobenzaldehydes with pyrazolone **1** is also diastereoselective. In both cases, the reactions led to formation of two stereogenic centers. Based on the data obtained, it was suggested that the reaction of *o*-(4-*R*-piperidino)benzaldehydes **2a—g** with pyrazolone **1** can proceed diastereoselectively despite of formation of three stereogenic centers.

Thus, this reaction carried out in 1-butanol afforded the products **3a—g** of the tandem of the Knoevenagel condensation and *tert*-amino effect cyclization in good yields (38—97%). The reaction gave also the second isomer (to

Scheme 1



2a—g **1**

3a—g

	R^1	R^2	R^3	R^4		R^1	R^2	R^3	R^4
a	H	H	H	H	**e**	Br	H	H	NC_5H_{10}
b	H	H	H	Me	**f**	Br	H	H	CH_2Ph
c	H	Cl	H	CH_2Ph	**g**	CF_3	H	H	CH_2Ph
d	H	H	F	Ph					

i. Bu^nOH, pyrrolidine (cat.), reflux, 6 h.

* Dedicated to academician V. N. Charushin on the occasion of his 60 birthday.

Published in Russian in *Izvestiya Akademii Nauk. Seriya Khimicheskaya,* No. 5, pp. 937—940, May, 2011.

5% according to the NMR data), which isolation failed due to low content.

Structures of the main products were established by X-ray diffraction (on the example of compound **3d**), ^{1}H and ^{13}C NMR spectra, as well as by comparison of the latter with the spectra of the previously synthesized compounds[17] bearing two stereogenic centers. All signals for the aliphatic protons are assigned by 2D ^{1}H—^{1}H COSY, ^{1}H—^{1}H NOESY and ^{1}H—^{13}C HSQC (for compound **3d**) and ^{1}H—^{13}C HMBC experiments (for compounds **3a**,**d**). The most informative cross-peaks in the 2D ^{1}H—^{13}C HMBC spectra in DMCO-d_6 are H(4a)/C(5′), H(4a)/C(2′), H(4a)/C(5), H(4a)/C(1), H(4a)/C(3), H(4a)/C(6), H(6_{eq}) and H(6_{ax})/C(5′), H(6_{eq}) and H(6_{ax})/C(2′), H(6_{eq}) and H(6_{ax})/C(5), H(6_{eq}) and H(6_{ax})/C(4a), H(6_{eq}) and H(6_{ax})/C(7), H(6_{eq}) and H(6_{ax})/C(6a), H(3)/C(4), H(3)/C(2), H(3)/C(Ar), H(1_{ax}) and H(1_{eq})/C(2), H(1_{ax}) and H(1_{eq})/C(3), H(1_{ax}) and H(1_{eq})/C(4a), H(4_{ax}) and H(4_{eq})/C(2), H(4_{ax}) and H(4_{eq})/C(3), H(4_{ax}) and H(4_{eq})/C(4a), H(4_{ax})/C(5). It should be noted that in the spectrum, no cross-peak H(4_{eq})/C(5) was observed, due probably to the fact that according to X-ray diffraction data the dihedral angle C(5)C(4a)C(4)H(4_{ax}) is 65°, while the angle C(5)C(4a)C(4)H(4_{eq}) is 52°. In ^{1}H—^{1}H NOESY spectrum of compound **3d**, the most interesting cross-peaks are H(3_{ax})/H_{ortho}(Ar), H(2)/H_{ortho}(Ar), H(6_{ax})/H(7). Analysis of ^{1}H NMR spectra reveals that the protons H(4a) and H(3) are axial. Thus, the signal for the proton H(4a) is observed at δ 3.44 as broadened doublet with coupling constant of 11.6 Hz characteristic of axial protons. The signal for the axial proton H(4) is doublet of doublet of doublets with large coupling constants of 12.0, 11.6 and 12.0 Hz. The spin-spin coupling constant values for the proton H(4) indicate that the axial orientation has not only the proton H(4a), but also the proton H(3). Note that in the ^{1}H NMR spectrum of the minor product, the signal for the proton H(4a) exhibits downfield shift (~0.1 ppm) to δ 3.56, this proton resonates as broadened doublet with coupling constant of ~9.2 Hz; the signal for the proton H(4_{ax}) exhibits upfield shift (~0.3 ppm) and this proton resonates as doublet of doublets with coupling constants of ~6.8 and 7.2 Hz. These facts indicate axial orientation of the protons H(3) and H(4a). Consequently, the structure of the minor isomer differs from that of the main product only in configuration of the carbon atom C(5). Similar pattern is observed for all series of compounds **3a**—**g**. Based on the chemical shifts for the protons H(6_{eq}), H(6_{ax}), and H(4a) for both isomers, the configuration for the carbon atom C(5) was suggested. In the main product, the carbonyl group and the protons H(6_{ax}) and H(4a) locate on the same side relative to the ring plane, *i.e.*, the configuration of the stereogenic carbon atoms is 3*R**,4a*S**,5*R**. In the minor product, the carbonyl group locates on the other side of the ring plane to the axial proton H(4a), *i.e.*, the relative configuration of the minor isomer is 3*R**,4a*S**,5*S**. The axial proton H(4a) is in the cone of anisotropy formed by the C=O bond in the case of the main reaction product **3d** and is not in the cone in the case of the minor product (Fig. 1).

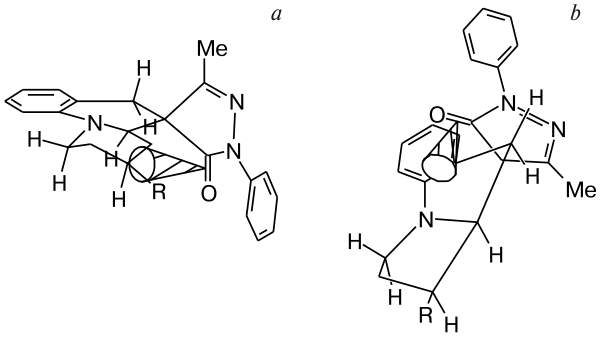


Fig. 1. Relative configuration of the main (*a*) and the minor (*b*) reaction products.

According to the X-ray diffraction (Fig. 2 and Table 1), the protons H(4a) and H(3) have axial orientation, the phenyl substituent in the dialkylamino group has the equatorial orientation, and the carbonyl group and the protons H(6_{ax}) and H(4a) locate on the same side of the ring plane. The X-ray analysis reveals that the distance between the oxygen atom at the pyrazole ring and the proton H(4a) is 2.612 Å, the sum of the van der Waals radii of the oxygen atom and proton is equal to 2.60 Å (see Ref. 18), and the radius of the interaction of the unbound atoms O...H is 2.46—2.48 Å (see Ref. 19).

In summary, in the present work, it was shown that despite of formation of three stereogenic centers, the cyclization reaction of *ortho*-(4-*R*-piperidino)benzaldehydes with pyrazolone **1** proceeds diastereoselectively to give mainly (3*R**,4a*S**,5*R**)-3-*R*-3′-methyl-1′-phenyl-2,3,4,4a,5,6-hexahydro-1*H*-spiro[benzo[*c*]quinolizine-5,4′-pyrazole]-5′-ones.

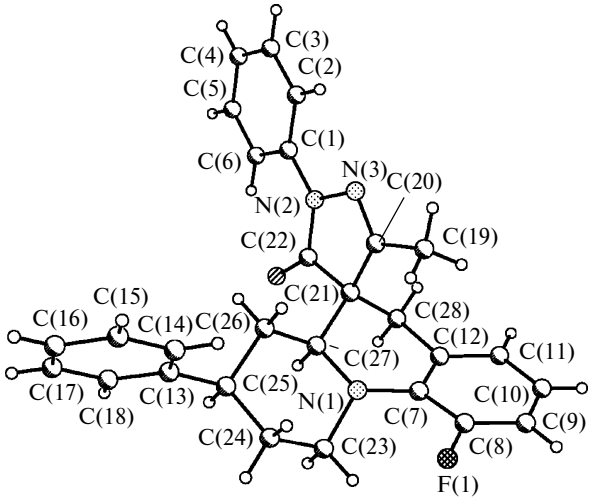


Fig. 2. General view of the molecule of **3d**.

Experimental

The course of the reactions and purity of the compounds were monitored by TLC on Silufol UV 254 plates, elution with ethyl acetate—hexane 1 : 1, 1 : 2, and 1 : 5. ^{1}H and ^{13}C NMR were recorded on Bruker WM-250 (^{1}H, 250 MHz) and Bruker Avance II (^{1}H, 400 MHz; ^{13}C, 100 MHz) instruments in DMSO-d_6 relative to $SiMe_4$ as the internal standard.

X-ray diffraction analysis was carried out on Xcalibur-3 diffractometer equipped with CCD area detector (λ(Mo-Kα) = = 0.71073, graphite monochromator, ω-scanning mode, scanning step is 1°, T = 295(2) K). The structure was solved by direct method and refined using SHELXTL-97 software.[20] The positions and thermal parameters of the non-hydrogen atoms were refined in an isotropic and then in an anisotropic approximation by the full matrix least squares method. The hydrogen atoms were revealed on maxima of electron density and were included in the refinement by the riding model. The main crystallographic parameters are given in Table 1.

The starting *ortho*-fluorobenzaldehydes and cyclic amines (Aldrich), 1-butanol (Reakhim, per analysis), and ethanol (Ormet Ltd) were used as purchased.

The starting *ortho*-(4-*R*-piperidino)benzaldehydes **2a—g** were synthesized in 60—80% yields by nucleophilic substitution of the corresponding 4-*R*-piperidines for fluorine atom in 2-fluorobenzaldehydes according to the known procedures.[16—17]

Synthesis of spiro derivatives of fused quinolizinepyrazolones 3a—g (general procedure). To a solution of the corresponding 2-aminobenzaldehyde **2** (1.0 mmol) in 1-butanol (8.0 mL), 5-methyl-2-phenyl-2,4-dihydro-3*H*-pyrazol-3-one (**1**) (185 mg, 1.06 mmol) and pyrrolidine (8 μL, 0.1 mmol) were added. The reaction mixture was refluxed for 6 h, after completion of the reaction (TLC monitring), the solvent was removed *in vacuo*, the residue was recrystallized from ethanol.

3´-Methyl-1´,3-diphenyl-2,3,4,4a,5,6-hexahydro-1*H*-spiro[benzo[*c*]quinolizine-5,4´-pyrazol]-5´-one (3a). Yield 375 mg (89%), m.p. 155 °C. ^{1}H NMR (DMSO-d_6), δ: 7.83 (dd, 2 H, Ar, J = 8.8 Hz, J = 1.2 Hz); 6.91 (dd, 2 H, Ar, J = 8.8 Hz, J = 7.2 Hz); 7.09—7.27 (m, 8 H, Ar); 7.04 (d, 1 H, Ar, J = 7.6 Hz); 6.74 (dd, 1 H, Ar, J = 7.6 Hz, J = 6.8 Hz); 4.24 (br.d, 1 H, C(1_{eq})H, J = 12.1 Hz); 3.25 (d, 1 H, C(6_{eq})H, J = 16.5 Hz); 2.85 (dd, 1 H, C(4_a)H, J = 11.1 Hz, J = 2.5 Hz); 2.84 (d, 1 H, C(6_{ax})H, J = 16.6 Hz); 2.70 (br.d, 1 H, C(1_{ax})H, J = 10.1 Hz); 1.92 (s, 3 H, Me); 1.54 (ddd, 1 H, C(2_{ax})H, J = 8.3 Hz, J = 3.0 Hz); 1.48—1.20 (m, 1 H, C(3_{ax})H); 1.25 (dd, 1 H, C(4_{eq})H, J = 7.9 Hz, J = 1.5 Hz); 1.05 (d, 1 H, C(2_{eq})H, J = 8.3 Hz); 0.89 (ddd, 1 H, C(4_{ax})H, J = 11.1 Hz, J = 7.9 Hz, J = 12.1 Hz). ^{13}C NMR (DMSO-d_6), δ: 173.30, 162.78, 145.25, 144.93, 137.39, 129.15, 128.98, 128.42, 127.70, 126.55, 126.33, 124.93, 119.28, 118.26, 117.99, 113.74, 58.32, 55.53, 47.63, 40.47, 34.86, 33.42, 32.32, 16.12. Found (%): N, 9.99. $C_{28}H_{27}N_3O$. Calculated (%): N, 9.97.

3,3´-Dimethyl-1´-phenyl-2,3,4,4a,5,6-hexahydro-1*H*-spiro[benzo[*c*]quinolizine-5,4´-pyrazol]-5´-one (3b). Yield 262 mg (73%), m.p. 105 °C. ^{1}H NMR (DMSO-d_6), δ: 7.91 (dd, 2 H, *o*-H, Ar, J = 7.7 Hz); 7.38 (ddd, 2 H, *m*-H, Ar, J = 8.5 Hz, J = 5.5 Hz, J = 1.8 Hz); 7.15 (t, 1 H, *p*-H, Ar, J = 7.7 Hz); 6.95 (dd, 1 H, Ar, J = 7.7 Hz, J = 7.5 Hz); 7.08 (d, 1 H, Ar, J = 7.6 Hz); 7.02 (d, 1 H, Ar, J = 7.0 Hz); 6.66 (dd, 1 H, Ar, J = 7.3 Hz, J = 7.1 Hz); 4.09 (br.dd, 1 H, C(4a)H, J = 7.9 Hz, J = 3.0 Hz); 3.23 (d, 1 H, C(6_{eq})H, J = 16.2 Hz); 3.15 (br.dd, 1 H, C(1_{eq})H, J = 9.2 Hz, J = 2.1 Hz); 2.68 (d, 1 H, C(6_{ax})H, J = 16.2 Hz); 2.67 (dd, 1 H, C(1_{ax})H, J = 12.8 Hz, J = 9.2 Hz); 1.89 (s, 3 H, Me); 1.57—1.48 (m, 1 H, C(3_{ax})H); 1.54 (br.d, 1 H, C(2_{ax})H, J = 12.0 Hz); 1.25 (ddd, 1 H, C(4_{eq})H, J = 7.9 Hz, J = 8.2 Hz, J = 1.5 Hz); 1.05 (br.d, 1 H, C(2_{eq})H, J = 7.7 Hz); 0.92 (d, 3 H, Me, J = 6.4 Hz); 0.72 (dddd, 1 H, C(4_{ax})H, J = 7.9 Hz, J = 7.9 Hz, J = 8.5 Hz). Found (%): N, 11.71. $C_{23}H_{25}N_3O$. Calculated (%): N, 11.69.

3-Benzyl-9-chloro-3´-methyl-1´-phenyl-2,3,4,4a,5,6-hexahydro-1*H*-spiro[benzo[*c*]quinolizine-5,4´-pyrazol]-5´-one (3c). Yield 329 mg (70%), m.p. 98 °C. ^{1}H NMR (DMSO-d_6), δ: 7.82 (d, 2 H, *o*-H, Ar, J = 7.5 Hz); 7.38 (t, 2 H, *m*-H, Ar, J = 7.8 Hz); 7.20—6.83 (m, 8 H, Ar); 6.65 (d, 1 H, Ar, J = 7.5 Hz); 3.99 (br.d, 1 H, C(1_{eq})H, J = 11.0 Hz); 3.17 (d, 1 H, C(6_{eq})H, J = 16.5 Hz); 3.15 (dd, 1 H, C(4_a)H, J = 11.0 Hz, J = 2.0 Hz); 2.70 (d, 1 H, C(6_{ax})H, J = 16.5 Hz); 2.62—2.34 (m, 3 H, C(1_{ax})H, C(2)H_2); 1.90 (s, 3 H, Me); 1.83—1.52 (m, 3 H, C(4_{eq})H and CH_2Ph);

Table 1. Crystallographic data and parameters of X-ray diffraction experiment for compound **3d**

Parameter	Value
Solvent for recrystallization	EtOH
Molecular formula	$C_{28}H_{26}FN_3O$
Molecular weight	439.54
T/K	295(2)
λ/Å	0.71073
Crystal system	Orthorhombic
Space group	$P2_12_12_1$
a/Å	6.6769(4)
b/Å	18.2353(8)
c/Å	18.7690(16)
α/deg	90.00
β/deg	90.00
γ/deg	90.00
V/Å^3	2285.2(3)
Z	4
d_{calc}/g cm^{-3}	1.277
μ/mm^{-1}	0.084
F(000)	928
Crystal size/mm	0.25×0.20×0.15
Scanning range, θ/deg	3.12—28.28
Completeness to θ (%)	99.5 (28.28°)
Range of reflection indices h, k, l	$-6 < h < 8$
	$-24 < k < 20$
	$-24 < l < 25$
Number of measured reflections	3206
Number of independent reflections	1764
R_{int}	0.0380
Number of reflections with $I > 2\sigma(I)$	1764
Number of refinement parameters	307
GOOF (on F^2)	1.008
R Factors (on $I > 2\sigma(I)$)	
R_1	0.0289
wR_2	0.0451
R Factors (on all reflections)	
R_1	0.0701
wR_2	0.0482
Residual electron density (min/max)/e·Å^{-3}	−0.102/0.108

1.28—1.19 (m, 1 H, C(3_{ax})H); 0.74 (ddd, 1 H, C(4_{ax})H, J = 11.0 Hz, J = 11.0 Hz, J = 11.8 Hz). Found (%): N, 8.95. $C_{29}H_{28}ClN_3O$. Calculated (%): N, 8.94.

3´-Methyl-9-fluoro-1´,3-diphenyl-2,3,4,4a,5,6-hexahydro-1*H*-spiro[benzo[*c*]quinolizine-5,4´-pyrazol]-5´-one (3d). Yield 274 mg (67%), m.p. 186 °C. ^{1}H NMR (DMSO-d_6), δ: 7.81 (d, 2 H, *o*-H, Ar, J = 7.6 Hz); 7.41 (dd, 2 H, *m*-H, Ar, J = 7.6 Hz, J = 8.0 Hz); 7.14—7.27 (m, 6 H, *p*-H, Ar); 7.05 (dd, 1 H, Ar, J = 14.0 Hz, J = 12.8 Hz); 6.9 (d, 1 H, Ar, J = 7.2 Hz); 6.80 (ddd, 1 H, Ar, J = 8.0 Hz, J = 3.2 Hz, J = 2.4 Hz); 4.26 (dd, 1 H, C(1_{eq})H, J = 12.0 Hz, J = 2.4 Hz); 3.44 (br.d, 1 H, C(4a)H, J = 11.6 Hz); 3.29 (d, 1 H, C(6_{eq})H, J = 16.0 Hz); 3.01 (ddd, 1 H, C(1_{ax})H, J = 12.0 Hz, J = 7.4 Hz, J = 4.8 Hz); 2.90 (d, 1 H, C(6_{ax})H, J = 16.0 Hz); 2.78—2.82 (m, 1 H, C(3_{ax})H); 1.18—1.90 (m, 2 H, C(2_{eq})H, C(2_{ax})H); 1.74 (s, 3 H, Me); 1.51 (br.d, 1 H, C(4_{eq})H, J = 12.0 Hz); 1.32 (ddd, 1 H, C(4_{ax})H, J = 12.0 Hz, J = 12.0 Hz, J = 11.6 Hz). ^{13}C NMR (DMSO-d_6), δ: 172.69, 161.41, 144.65, 137.21, 128.77, 128.22, 126.47, 126.13, 124.88, 124.80, 119.52, 118.06, 115.41, 61.52, 55.86, 51.31, 51.17, 41.83, 34.63, 33.93, 32.94, 16.01. Found (%): N, 9.61. $C_{28}H_{26}FN_3O$. Calculated (%): N, 9.56.

8-Bromo-3´-methyl-1´-phenyl-3-(piperidin-1-yl)-2,3,4,4a,5,6-hexahydro-1*H*-spiro[benzo[*c*]quinolizine-5,4´-pyrazol]-5´-one (3e). Yield 192 mg (38%), m.p. 170 °C. ^{1}H NMR (DMSO-d_6), δ: 7.88 (d, 2 H, *o*-H, Ar, J = 8.2 Hz); 7.38 (t, 2 H, *m*-H, Ar, J = 7.7 Hz); 7.21—7.12 (m, 3 H, Ar); 6.89 (d, 1 H, Ar, J = 8.8 Hz); 4.10 (br.d, 1 H, C(1_{eq})H, J = 11.4 Hz); 3.24 (dd, 1 H, C(4_{ax})H, J = 11.9 Hz); 3.24 (d, 1 H, C(6_{eq})H, J = 17.0 Hz); 2.70 (d, 1 H, C(6_{ax})H, J = 17.0 Hz); 2.28—2.49 (m, 7 H, CH); 1.94 (s, 3 H, Me); 1.29—1.59 (m, 8 H, CH); 1.04 (ddd, C(4_{ax})H, 1 H, J = 12.1 Hz, J = 9.9 Hz, J = 11.9 Hz). Found (%): N, 11.05. $C_{27}H_{31}BrN_4O$. Calculated (%): N, 11.04.

3-Benzyl-8-bromo-3´-methyl-1´-phenyl-2,3,4,4a,5,6-hexahydro-1*H*-spiro[benzo[*c*]quinolizine-5,4´-pyrazol]-5´-one (3f). Yield 452 mg (88%), m.p. 145 °C. ^{1}H NMR (DMSO-d_6), δ: 7.82 (d, 2 H, *o*-H, Ar, J = 7.5 Hz); 7.40 (t, 2 H, *m*-H, Ar, J = 7.8 Hz); 7.20—7.09 (m, 7 H, Ar); 7.03 (d, 1 H, Ar, J = 7.5 Hz); 6.86 (d, 1 H, Ar, J = 8.8 Hz); 4.01 (d, 1 H, C(4a)H, J = 12.3 Hz); 3.21 (d, 1 H, H(6_{eq}), J = 17.0 Hz); 2.73 (d, 1 H, H(6_{ax}), J = 17.0 Hz); 3.12 (d, 1 H, C(1_{eq})H, J = 10.5 Hz); 2.73—2.34 (m, 3 H, C(1_{ax})H and CH_2Ph); 1.91 (s, 3 H, Me); 1.79—1.53 (m, 3 H, C(4_{eq})H, C(2)H_2); 1.25—1.21 (m, 1 H, C(3_{ax})H); 0.80—0.75 (m, 1 H, C(4_{ax})H). Found (%): N, 9.00. $C_{29}H_{28}BrN_3O$. Calculated (%): N, 8.97.

3-Benzyl-3´-methyl-1´-phenyl-8-trifluoromethyl-2,3,4,4a,5,6-hexahydro-1*H*-spiro[benzo[*c*]quinolizine-5,4´-pyrazol]-5´-one (3g). Yield 247 mg (97%), m.p. 156 °C. ^{1}H NMR (DMSO-d_6), δ: 7.82 (d, 2 H, *o*-H, Ar, J = 7.5 Hz); 7.40 (t, 2 H, *m*-H, Ar, J = 7.8 Hz); 7.20—7.09 (m, 7 H, Ar); 7.03 (d, 1 H, Ar, J = 7.5 Hz); 6.86 (d, 1 H, Ar, J = 8.8 Hz); 4.01 (d, 1 H, C(4a)H, J = 12.3 Hz); 3.21 (d, 1 H, H(6_{eq}), J = 17.0 Hz); 2.73 (d, 1 H, H(6_{ax}), J = 17.0 Hz); 3.12 (d, 1 H, C(1_{eq})H, J = 10.5 Hz); 2.73—2.34 (m, 3 H, C(1_{ax})H and CH_2Ph); 1.91 (s, 3 H, Me); 1.79—1.53 (m, 3 H, C(4_{eq})H, C(2)H_2); 1.25—1.21 (m, 1 H, C(3_{ax})H); 0.80—0.75 (m, 1 H, C(4_{ax})H). Found (%): N, 8.39. $C_{30}H_{28}F_3N_3O$. Calculated (%): N, 8.34.

References

1. M. Balasubramanian, J. G. Keay, in *Comprehensive Heterocyclic Chemistry II*, Eds A. R. Katritzky, C. W. Rees, E. F. V. Scriven, Pergamon, Oxford, 1996, **5**, 245.
2. J. C. Ruble, A. R. Hurd, T. A. Johnson, D. A. Sherry, M. R. Barbachin, P. L. Toogood, G. L. Bundy, D. R. Graber, G. M. Kalimar, *J. Am. Chem. Soc.*, 2009, **131**, 3991.
3. O. Meth-Cohn, H. Suschitzky, *Adv. Heterocycl. Chem.*, 1972, **14**. 211.
4. W. H. N. Nijhuis, W. Verboom, D. N. Reinhoudt, *J. Am. Chem. Soc.*, 1987, **109**, 3136.
5. P. Mátyus, O. Éliás, P. Tapolcsányi, A. Polonka-Bálint, B. Halász-Dajka, *Synthesis*, 2006, 2625.
6. J. M. Quintela, *Recent Res. Dev. Org. Chem.*, 2003, **7**, 259.
7. N. Krogsgaard-Larsen, M. Begtrup, M. M. Herth, J. Kehler, *Synthesis*, 2010, 4287.
8. Y. K. Kang, S. M. Kim, D. Y. Kim, *J. Am. Chem. Soc.*, 2010, **132**, 11847.
9. K. Mori, Y. Ohshima, K. Ehara, T. Akiyama, *Chem. Lett.*, 2010, **38**, 524.
10. A. P. Gorulya, A. V. Tverdokhlebov, A. A. Tolmachev, O. V. Shishkin, S. V. Shishkina, *Tetrahedron*, 2011, **67**, 1030.
11. S. Majumder, P. J. Bhuyan, *Synlett*, 2011, 173.
12. W. Verboom, D. N. Reinhoudt, R. Visser, S. Harkema, *J. Org. Chem.*, 1984, **49**, 269.
13. W. Verboom, D. N. Reinhoudt, *Recl. Trav. Chim. Pays-Bas*, 1990, **109**, 311.
14. W. H. N. Nijhuis, W. Verboom, A. Abu El-Fadl, G. J. Van Hummel, D. N. Reinhoudt, *J. Org. Chem.*, 1989, **54**, 209.
15. E. Kelderman, H. G. Noorlander-Bunt, J. van Eeerden, W. Verboom, D. N. Reinhoudt, *Recl. Trav. Chim. Pays-Bas*, 1991, **110**, 115.
16. E. V. D´yachenko, T. V. Glukhareva, E. F. Nikolaenko, A. V. Tkachev, Yu. Yu. Morzherin, *Izv. Akad. Nauk, Ser. Khim.*, 2004, 1191 [*Russ. Chem. Bull., Int. Ed.*, 2004, **53**, 1240].
17. T. V. Glukhareva, E. V. Deeva, A. Yu. Platonova, I. V. Geide, Yu. Yu. Morzherin, M. I. Kodess, *Zh. Org. Khim.*, 2009, **45**, 757 [*Russ. J. Org. Chem.* (*Engl. Transl.*), 2009, **45**, 743].
18. L. Pauling, *The Nature of the Chemical Bond and the Structure of Molecules and Crystals*, Cornell Univ. Press, London, 1960, 644 p.
19. S. R. Rowland, R. Taylor, *J. Phys. Chem.*, 1996, **100**, 7384.
20. G. M. Sheldrick, *Acta Crystallogr. Sec. A: Found. Crystallogr.*, 2008, **64**, 112.

Received February 11, 2011;
in revised form April 12, 2011