Cyclization of *ortho*-(alk-2-enyl)anilines under the action of iodine

R. R. Gataullin,[a] F. F. Minnigulov,[b] T. V. Khakimova,[a] T. V. Kazhanova,[b] A. A. Fatykhov,[a] L. V. Spirikhin,[a] and I. B. Abdrakhmanov[a]*

[a]Institute of Organic Chemistry, Ufa Research Center of the Russian Academy of Sciences, 71 prosp. Oktyabrya, 450054 Ufa, Russian Federation. Fax: +7 (347 2) 35 6066. E-mail: chemorg@anrb.ru
[b]Bashkir State Agricultural University, 34 ul. 50-letiya Oktyabrya, 450003 Ufa, Russian Federation.

The reaction of *ortho*-(cyclohex-2-enyl)aniline with I_2 in nonpolar and polar solvents affords predominantly 1-iodohexahydrocarbazole and azatricyclotridecatriene, respectively. Under analogous conditions, 4-methyl-2-(1-methylbut-2-en-1-yl)aniline undergoes cyclization to form exclusively products with quinoline structures regardless of the solvent used.

Key words: *ortho*-(alk-2-enyl)anilines, iodocyclization, 1-iodo-1,2,3,4,4a,9a-hexahydrocarbazole, 13-iodo-2-azatricyclo[7.3.1.$0^{2,7}$]trideca-3,5,7-triene, 3-iodo-1,2,3,4-tetrahydroquinolines.

Nitrogen-containing heterocyclic compounds are often synthesized by cyclization of alkenylamides or alkenylsulfamides under the action of halogens, in particular, of iodine.[1] Examples of cyclization of aminoalkenes under these conditions are few in number.[2—4] Generally, it is assumed that an onium complex of alkene with halogen is initially formed in these reactions. The direction of subsequent transformations of this complex into a heterocycle depends on the reaction conditions, the nature of the solvent, and the structure of the alkenyl radical.[5] Recently, we have reported[6] cyclization of 2-(cyclopent-2-enyl)anilines under the action of I_2 giving rise exclusively to 3-iodine-substituted indolines. As part of our continuing studies, we examined the analogous reactions of 2-(cyclohex-2-enyl)- and 2-(1-methylbut-2-en-1-yl)anilines prepared from 3-bromocyclohexene[7] and 3-chloropentene,[8] respectively.

Thus, the reaction of *ortho*-(cyclohex-2-enyl)aniline (**1**) with I_2 in the presence of $NaHCO_3$ affords heterocycles **2—4** (Scheme 1) whose ratio depends on the properties of the solvent (Table 1). In particular, 8-azatricyclotridecatriene **3** and hexahydrocarbazole **2** were obtained as the major products in MeCN and CCl_4, respectively. In the reactions performed in MeCN, the time it took for amine **1** to be converted into reaction products **2—4** was substantially larger. In addition, the latter reactions required one more equivalent of I_2, the yield of diiodo derivative **4** reaching 9%.

Scheme 1

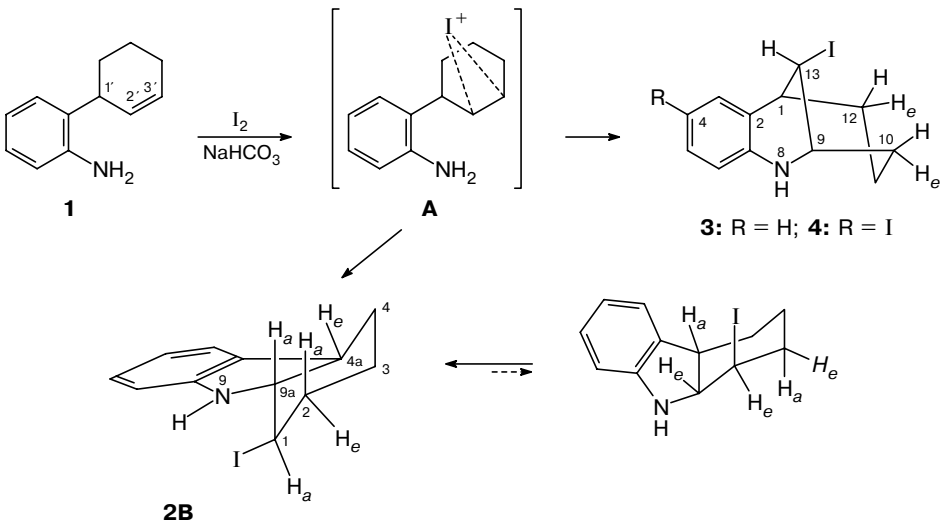

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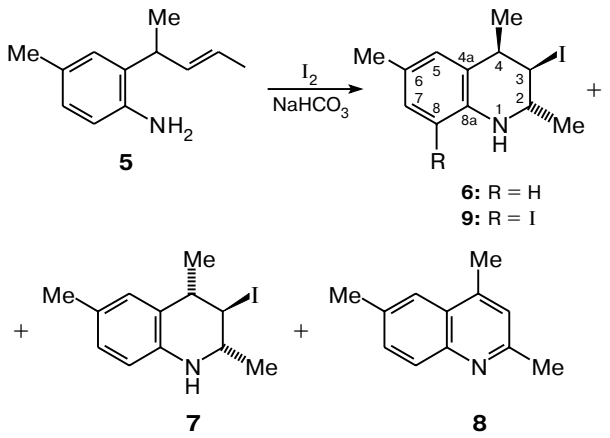
Table 1. Dependence of the isomer ratio on the solvent in the reaction of amine **1** with I_2

Solvent	Reaction time /h	Ratio between the reaction products, **3** : **2**
MeCN	72	3 : 1
CCl_4	48	1 : 3
cyclo-C_6H_{12}	72	1 : 6
HCOOH	130	6 : 1

Therefore, the reaction of amine **1** with I_2 afforded initially complex **A** whose 5-*exo*- and 6-*endo*-cyclization[9] gave rise to heterocycles **2** and **3**, respectively. The intramolecular nucleophilic attack in nonpolar solvents (see Table 1) occurs preferentially at the C(2′) atom of the cyclohexenyl substituent, whereas polar solvents favor the attack at the C(3′) atom.

Unlike cyclization of amine **1**, the formation of regioisomers in the reaction of amine **5** with I_2 is virtually independent of the properties of the solvent. This reaction afforded exclusively 3-iodo-2,4,6-trimethyl-1,2,3,4-tetrahydroquinolines **6** and **7** and quinoline **8** (the yield of the latter varied from 8 to 20%) (Scheme 2). The reaction in MeCN also gave rise to diiodo derivative **9** (4%).

Scheme 2



The structures of compounds **2**—**9** were established by NMR spectroscopy. In the spectrum of carbazole **2**, the signal for the H(1) proton has two large spin-spin coupling constants (12.0 and 9.1 Hz) and one smaller constant (4.0 Hz). Studies by the double resonance method demonstrated that the constants equal to 12.0 and 4.0 Hz are caused by vicinal coupling with the geminal protons at the C(2) atom, whereas the constant of 9.1 Hz appears due to coupling with the protons at the C(9a) atom. The large spin-spin coupling constants are indicative of the *trans*-diaxial interaction, *i.e.*, the equilibrium is shifted toward conformer **B** (see Scheme 1) with the equatorial arrangement of the iodine atom. The vicinal spin-spin coupling constant between the H(9a) and H(4a) protons (7.2 Hz) confirms the *cis* arrangement of these protons and, consequently, *cis*-fusion of the rings.[10—12]

The ^{13}C NMR spectra measured in the pulsed J-modulated spin-echo mode have doublet signals for the methine carbon atoms at δ 69.3, 42.6, and 39.0 (C(9a), C(4a), and C(1), respectively) along with the signals for the carbon atoms of the CH groups of the benzene fragment. The triplet signal for the C(2) atom is shifted downfield by 36.3 ppm due to the β-effect of the heavy iodine atom.[5]

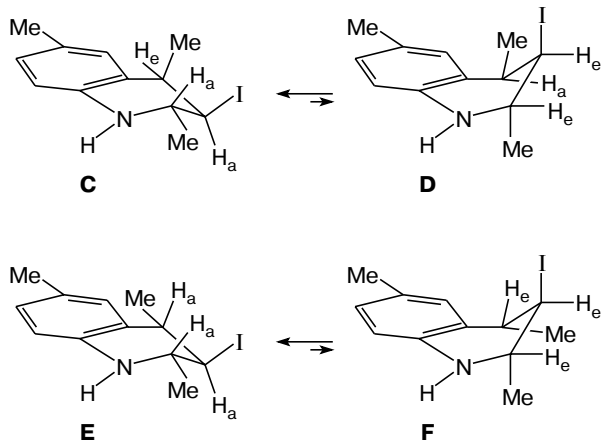
The assignment of the signals in the ^{13}C NMR spectra of azatricyclotridecatrienes **3** and **4** was made based on the multiplicities of the signals, the results of calculations from the increments of the chemical shifts,[10] and comparison with the published data for substituted bicyclo[3.1.1]heptanes[13] and analogous bridging systems.[14] The *anti* orientation of the I atom with respect to the phenyl ring was assigned based on the ^1H NMR spectral data. The bridging H(13) proton gives a signal as a doublet of doublets of doublets of doublets with the spin-spin coupling constant of no higher than 2 Hz. Studies by the double resonance method demonstrated that two spin-spin coupling constants for the H(13) proton are caused by coupling with the nodal H(1) and H(9) protons, whereas two other constants (W constants) appear due to coupling with the equatorial $H_e(12)$ and $H_e(10)$ protons.[15]

The structures of isomers **6** and **7** were established by ^1H NMR spectroscopy. The signals for the H(4) and H(2) protons are observed as doublets of quartets, whereas the signal for the H(3) proton is manifested as a doublet of doublets. The unambiguous assignment of the signals for the H(2) and H(4) protons was made based on the two-dimensional heteronuclear ^{13}C-^1H COSY spectrum in which the signals for the C(2) and C(4) atoms are rather characteristic. The methyl groups at the C(2) and C(4) atoms in compounds **6** and **7** differ in the spin-spin coupling constants with the vicinal protons. The constants for the C(2) and C(4) atoms are 6.3—6.4 and 7.3 Hz, respectively. For compound **7**, the spin-spin coupling constants of the vicinal H(3) proton with H(4) and H(2) are 10.4 and 10.3 Hz, respectively. These values are characteristic of the *trans*-diaxial arrangement of the protons.[15] Consequently, compound **7** has a structure with the mutual *trans* configurations of the substituents at the C(2) and C(3) atoms and at the C(3) and C(4) atoms. Conformer **E** with the equatorial arrangement of the substituents prevails in solution (Scheme 3). The observed shift of the conformational equilibrium was confirmed by the results of experiments performed at different temperatures. Thus, the vicinal spin-spin coupling constants between the H(3) and H(2) protons and between the H(3) and H(4) protons were decreased by 0.2—0.25 Hz as the temperature of the specimens was increased from 25 to 55 °C due to an

increase in the fraction of the conformers with the axial arrangement of the substituents.

In the ^{13}C NMR spectrum of compound **6**, the signals for the C(4) and C(3) atoms are observed at higher field (at δ 36.1 and 42.4, respectively) compared to those in the spectrum of compound **7** (at δ 42.8 and 46.2, respectively).

Scheme 3



Based on these data, it was concluded that the substituents at the C(3) and C(4) atoms in compound **6** are in the *cis* arrangement. It is known[10] that interactions between 1,2-*cis* substituents lead to upfield shifts of the signals for the carbon atoms to which these substituents are bound compared to the corresponding signals observed in the spectra of 1,2-*trans* compounds. In the ^1H NMR spectra of these compounds, the signals for the methyl groups at the C(4) atom differ most substantially. The chemical shifts of these signals in isomers **6** and **7** are 1.7 and 1.4 ppm, respectively. The spin-spin coupling constants between the H(3) and H(4) protons are 10.4 and 4.0 Hz for compounds **7** and **6**, respectively, which is indicative of the different mutual arrangement of these protons (*trans* in compound **7** and *cis* in compound **6**). These data suggest that stereoisomer **6** and diastereomer **7** differ in the orientation of the methyl group at the C(4) atom. Therefore, compound **6** is a diastereomer with the *trans,cis* arrangement of the substituents at the C(2), C(3), and C(4) atoms and exists in solutions predominantly as conformer **C** (see Scheme 3).

In the ^1H NMR spectrum of 3,8-diiodoquinoline **9**, the chemical shifts of the signals for the aliphatic protons and their spin-spin coupling constants are analogous to those in the spectrum of compound **6**. The ^{13}C NMR spectrum of heterocycle **9** has six signals for the atoms of the aromatic moiety of the molecule. The chemical shift for the C(8) atom (83.6 ppm) indicates that the iodine atom is bound to the C(8) atom.[16]

To summarize, cyclization of 2-(cyclohex-2-enyl)aniline under the action of iodine afforded predominantly an isomer with either a carbazole or azatricyclotridecatriene structure depending on the solvent used, whereas the reaction of 2-(1-methylbut-2-enyl)-4-methylalanine performed under these conditions gave rise exclusively to 3-iodine-substituted tetrahydroquinolines regardless of the solvent.

Experimental

The ^1H (300.13 MHz) and ^{13}C (75.47 MHz) NMR spectra were recorded on a Bruker AM-300 instrument in $CDCl_3$ with Me_4Si as the internal standard. The IR spectra were measured on a UR-20 instrument. The mass spectra were obtained on an MX 1320 instrument (70 eV). The course of the reaction was monitored using Silufol UV 254 plates.

Procedure for cyclization of alkenylaniline 1. A mixture of arylamine **1** (0.17 g, 1 mmol), $NaHCO_3$ (1.5 g), and I_2 (0.51 g, 2 mmol) in a solvent (10 mL) (see Table 1) was shaken at 20 °C for 24—130 h. The course of the reaction was monitored by TLC (a 98 : 2 hexane—MeOH mixture as the eluent). After completion of the reaction, CH_2Cl_2 (50 mL) was added. The precipitate that formed was filtered off and washed with CH_2Cl_2 (10 mL). The combined filtrates were washed with a 5% aqueous solution of $Na_2S_2O_3$ (3×10 mL) and water (1×20 mL) and dried over Na_2SO_4. The solvent was evaporated *in vacuo*. The residue was chromatographed (hexane as the eluent) on a column with SiO_2 (3 g) to isolate the reaction products.

(1*R,4a*R**,9a*R**)-1-Iodo-1,2,3,4,4a,9a-hexahydro-9*H*-carbazole (2).** Chromatography of the reaction mixture obtained in the reaction in CCl_4 afforded compound **2** as a brown viscous oil in a yield of 0.17 g (57%), R_f 0.60 (CH_2Cl_2). Found (%): C, 47.67; H, 4.86; I, 41.87; N, 3.92. $C_{12}H_{14}IN$. Calculated (%): C, 48.18; H, 4.72; I, 42.42; N, 4.68. IR, ν/cm^{-1}: 3472 (NH). ^1H NMR, δ: 1.10—2.30 (m, 6 H, 3 CH_2), 3.20 (m, 1 H, H(4a)); 3.88 (dd, H(9a), $J_{H(9a),H(1)}$ = 9.1 Hz, $J_{H(9a),H(4a)}$ = 7.1 Hz); 3.97 (ddd, 1 H, H(1), J_1 = 4.0 Hz, J_2 = 9.1 Hz, J_3 = 12.0 Hz); 4.10 (br.s, 1 H, NH); 6.80 (d, 1 H, H(8), J = 7.6 Hz); 6.70 (t, 1 H, H(6)); 7.10 (d, 1 H, H(5), J = 7.5 Hz); 7.20 (t, 1 H, H(7)). ^{13}C NMR, δ: 23.2 (C(3)); 23.5 (C(4)); 36.3 (C(2)); 39.0 (C(1)); 42.6 (C(4a)); 69.5 (C(9a)); 110.5 (C(8)); 119.2 (C(6)); 122.7 (C(5)); 127.4 (C(7)); 129.5 (C(4b)); 149.7 (C(8a)).

(1*S,9*S**,13*S**)-13-Iodo-2-azatricyclo[7.3.1.0^{2,7}]trideca-3,5,7-triene (3).** Chromatography of the reaction mixture obtained in the reaction in MeCN afforded compound **3** as a dark viscous oil in a yield of 0.15 g (49%), R_f 0.50 (CH_2Cl_2). Found (%): C, 48.42; H, 4.23; I, 41.94; N, 4.00. $C_{12}H_{14}IN$. Calculated (%): C, 48.18; H, 4.72; I, 42.42; N, 4.68. IR, ν/cm^{-1}: 3475 (NH). ^1H NMR, δ: 1.50—1.90 (m, 4 H, H_a(10), H_a(12), C(11)H_2); 2.30—2.60 (m, 2 H, H_e(10), H_e(12)); 3.20 (m, 1 H, H(1)); 3.70 (dddd, 1 H, H(13), J = 2.1, 2.0, 1.6, and 1.5 Hz); 4.20 (br.s, 1 H, NH); 4.90 (ddd, 1 H, H(9), J = 2.0, 1.6, and 4.2 Hz); 6.60 (d, 1 H, H(6), J = 7.3 Hz); 6.80 (t, 1 H, H(4), J = 7.3 Hz); 7.00 (d, 1 H, H(3), J = 7.3 Hz); 7.10 (t, 1 H, H(5)). ^{13}C NMR, δ: 16.6 (C(11)); 29.9 (C(12)); 30.1 (C(10)); 33.2 (C(13)); 41.3 (C(1)); 51.7 (C(9)); 112.4 (C(6)); 118.1 (C(4)); 123.4 (C(2)); 127.2 (C(3)); 127.8 (C(5)); 144.3 (C(7)).

(1*S,9*S**,13*S**)-6,13-Diiodo-2-azatricyclo[7.3.1.0^{2,7}]trideca-3,5,7-triene (4)**. Chromatography of the products, which were obtained in the reaction performed in MeCN, afforded compound **4** as a viscous oil in 9% yield, R_f 0.70 (CH_2Cl_2). Found (%): C, 33.91; H, 3.08; N, 3.30; I, 59.71. $C_{12}H_{13}I_2N$. Calculated (%): C, 33.91; H, 3.08; I, 59.71; N, 3.30. IR, ν/cm^{-1}: 3473 (NH). ^1H NMR, δ: 1.80—2.00 (m, 4 H, H_a(10), H_a(12), C(11)H_2); 2.10—2.30 (m, 2 H, H_e(10), H_e(12)); 3.10

(m, 1 H, H(1)); 3.70 (dddd, 1 H, H(13), J = 2.1, 2.0, 1.6, and 1.2 Hz); 4.10 (br.s, 1 H, NH); 4.70 (ddd, 1 H, H(9), J = 2.0, 1.7, and 4.1 Hz); 6.58 (d, 1 H, H(6), J = 8.0 Hz); 6.90 (s, 1 H, H(3)); 7.00 (d, 1 H, H(5)). ^{13}C NMR, δ: 16.5 (C(11)); 30.1 (C(12)); 30.2 (C(10)); 31.4 (C(13)); 40.9 (C(1)); 51.5 (C(9)); 80.3 (C(4)); 112.5 (C(6)); 126.3 (C(3)); 123.7 (C(2)); 131.5 (C(5)); 144.3 (C(7)).

Procedure for iodocyclization of alkenylaniline 5. The reaction with I_2 was performed and the reaction mixture was worked up according to the above-described procedure. A mixture of compounds **6—9** was obtained from compound **5** (1.75 g, 10 mmol) and I_2 (5.1 g, 20 mmol) in a solvent (benzene, MeCN, CH_2Cl_2, CCl_4, or 1,2-dichloroethane; 30 mL) in a yield of ~4 g. The mixture of the products was kept at ~20 °C for two days and treated with CCl_4 (5 mL). The crystals of quinoline **8** that formed were filtered off, washed with CCl_4 (1—3 mL), and dried *in vacuo*. The filtrate was concentrated to the minimum volume and chromatographed on a column with SiO_2 (50 g) using hexane as the eluent to isolate products **6**, **7**, and **9**.

(2*R,3*R**,4*R**)-3-Iodo-2,4,6-trimethyl-1,2,3,4-tetrahydroquinoline (6)** was obtained as an oil in a yield of 1.11 g (37%), R_f 0.40 (CH_2Cl_2). Found (%): C, 47.67; H, 4.86; I, 41.87; N, 4.92. $C_{12}H_{16}IN$. Calculated (%): C, 47.86; H, 5.36; I, 42.14; N, 4.65. IR, ν/cm^{-1}: 3474 (NH). ^{1}H NMR, δ: 1.39 (d, 3 H, C(2)CH_3, J = 6.4 Hz); 1.42 (d, 3 H, C(4)CH_3, J = 7.4 Hz); 2.25 (s, 3 H, CH_3); 2.91 (dq, 1 H, H(4), $J_{H(4),H(3)}$ = 4.0 Hz, $J_{H(4),CH_3}$ = 7.4 Hz); 3.84 (dq, 1 H, H(2), $J_{H(2),CH_3}$ = 6.4 Hz, $J_{H(2),H(3)}$ = 8.4 Hz); 4.35 (dd, 1 H, H(3), $J_{H(3),H(4)}$ = 4.0 Hz, $J_{H(2),H(3)}$ = 8.4 Hz); 6.40 (d, 1 H, H(8), $J_{H(8),H(7)}$ = 8.0 Hz); 6.86 (d, 1 H, H(7)); 6.94 (s, 1 H, H(5)). ^{13}C NMR, δ: 20.6, 22.1, 24.8 (3 CH_3); 36.1 (C(4)); 42.4 (C(3)); 51.3 (C(2)); 113.8 (C(8)); 123.2 (C(4a)); 128.1 (C(7)); 128.3 (C(5)); 128.9 (C(6)); 139.4 (C(8a)).

(2*R,3*R**,4*S**)-3-Iodo-2,4,6-trimethyl-1,2,3,4-tetrahydroquinoline (7)** was obtained as an oil in a yield of 0.9 g (30%), R_f 0.50 (CH_2Cl_2). Found (%): C, 47.82; H, 5.40; I, 41.92; N, 4.00. $C_{12}H_{16}IN$. Calculated (%): C, 47.86; H, 5.36; I, 42.14; N, 4.65. IR, ν/cm^{-1}: 3476 (NH). ^{1}H NMR, δ: 1.37 (d, 3 H, C(2)CH_3, J = 6.3 Hz); 1.70 (d, 3 H, C(4)CH_3, J = 7.3 Hz); 2.20 (s, 3 H, CH_3); 3.20 (dq, 1 H, H(4), $J_{H(4),CH_3}$ = 7.3 Hz, $J_{H(4),H(3)}$ = 10.4 Hz); 3.60 (dq, 1 H, H(2), $J_{H(2),CH_3}$ = 6.3 Hz, $J_{H(2),H(3)}$ = 10.3 Hz); 4.00 (dd, 1 H, H(3), $J_{H(3),H(2)}$ = 10.3 Hz, $J_{H(3),H(4)}$ = 10.4 Hz); 6.50 (d, 1 H, H(8), J = 7.9 Hz); 6.90 (d, 1 H, H(7)); 7.10 (s, 1 H, H(5)). ^{13}C NMR, δ: 20.6, 21.6, and 24.9 (CH_3); 42.8 (C(4)); 46.2 (C(3)); 54.2 (C(2)); 114.4 (C(8)); 124.1 (C(4a)); 128.4 (C(7)); 128.3 (C(5)); 128.9 (C(6)); 141.2 (C(8a)).

2,4,6-Trimethylquinoline (8) was obtained in a yield of 0.34 g (20%), m.p. 125 °C. Found (%): C, 83.85; H, 7.32; N, 7.78. $C_{12}H_{13}N$. Calculated (%): C, 84.17; H, 7.65; N, 8.18. ^{1}H NMR, δ: 2.60 (s, 3 H, CH_3); 2.90 (s, 3 H, CH_3); 3.20 (s, 3 H, CH_3); 7.50 (s, 1 H, H(3)); 7.80 (d, 1 H, H(8), J = 8.7 Hz); 7.90 (s, 1 H, H(5)); 8.90 (d, 1 H, H(7), J = 8.7 Hz). ^{13}C NMR, δ: 20.0, 20.2 и 22.0 (3 CH_3); 120.7 (C(3)); 123.6 (C(5)); 123.9 (C(8)); 127.0 (C(4a)); 135.9 (C(6)); 136.3 (C(7)); 140.2 (C(4)); 154.5 (C(8a)); 155.9 (C(2)).

(2*R,3*R**,4*R**)-3,8-Diiodo-2,4,6-trimethyl-1,2,3,4-tetrahydroquinoline (9)** was obtained as an oil in a yield of 0.17 g (4%), R_f 0.60 (CH_2Cl_2). Found (%): C, 32.83; H, 4.03; I, 58.84; N, 2.64. $C_{12}H_{15}I_2N$. Calculated (%): C, 33.75; H, 3.54; I, 59.43; N, 3.28. IR, ν/cm^{-1}: 3479 (NH). ^{1}H NMR, δ: 1.45 (d, 3 H, C(2)CH_3, J = 6.4 Hz); 1.40 (d, 3 H, C(4)CH_3, J = 6.8 Hz); 2.20 (s, 3 H, CH_3); 2.90 (dq, 1 H, H(4), $J_{H(4),H(3)}$ = 4.0 Hz, $J_{H(4),HCH_3}$ = 6.8 Hz); 3.90 (dq, 1 H, H(2), $J_{H(2),H(3)}$ = 8.2 Hz, $J_{H(2),CH_3}$ = 6.4 Hz); 4.20 (dd, 1 H, H(3), $J_{H(3),H(4)}$ = 4.0 Hz, $J_{H(2),H(3)}$ = 8.2 Hz); 4.30 (br.s, 1 H, NH); 6.80 (s, 1 H, H(7)); 7.40 (s, 1 H, H(5)). ^{13}C NMR, δ: 19.9, 21.9, and 24.8 (CH_3); 38.1 (C(4)); 40.6 (C(3)); 51.6 (C(2)); 83.6 (C(8)); 123.7 (C(4a)); 127.7 (C(6)); 128.4 (C(5)); 137.5 (C(7)); 139.2 (C(8a)).

References

1. C. Cardillo and M. Orena, *Tetrahedron*, 1990, **46**, 3321.
2. A. Bongini, C. Cardillo, M. Orena, G. Porzi, and S. Sandri, *Chem. Lett.*, 1988, 87.
3. S. R. Wilson and R. A. Sawicki, *J. Org. Chem.*, 1979, **44**, 287.
4. S. R. Wilson, R. A. Sawicki, and J. C. Huffman, *J. Org. Chem.*, 1981, **46**, 3887.
5. M. Watanabe, H. Okada, T. Teshima, M. Nogucli, and A. Kakehi, *Tetrahedron*, 1996, **52**, 2827.
6. R. R. Gataullin, T. V. Kazhanova, F. F. Minnigulov, A. A. Fatykhov, L. V. Spirikhin, and I. B. Abdrakhmanov, *Izv. Akad. Nauk, Ser. Khim.*, 2000, 1789 [*Russ. Chem. Bull., Int. Ed.*, 2000, **49**, 1767].
7. I. B. Abdrakhmanov, V. M. Sharafutdinov, and G. A. Tolstikov, *Izv. Akad. Nauk SSSR, Ser. Khim.*, 1982, 2160 [*Bull. Acad. Sci. USSR, Div. Chem. Sci.*, 1982, **31** (Engl. Transl.)].
8. I. B. Abdrakhmanov, V. M. Sharafutdinov, N. G. Nigmatullin, I. A. Sagitdinov, and G. A. Tolstikov, *Izv. Akad. Nauk SSSR, Ser. Khim.*, 1982, 1466 [*Bull. Acad. Sci. USSR, Div. Chem. Sci.*, 1982 (Engl. Transl.)].
9. J. E. Baldwin, *J. Chem. Soc., Chem. Comm.*, 1976, p. 734.
10. E. Pretsch, T. Clerk, J. Seible, and W. Simon, *Tables of Spectral Data for Structure Determination of Organic Compounds*, Springer Verlag, Berlin—Heidelberg—New York—Tokyo, 1983.
11. L. M. Jackman and S. Sternhell, *Application of Nuclear Magnetic Resonance in Organic Chemistry*, Pergamon Press, Oxford, 1969, p. 236.
12. R. A. Abramovitch and S. S. Singer, *J. Org. Chem.*, 1976, **41**, 1712.
13. J. K. Whitersell and M. A. Minton, *Stereochemical Analysis of Alicyclic Compounds by ^{13}C NMR Spectroscopy*, Chapman and Hale, London—New York, 1987, p. 114.
14. M. Sindler-Kulyk and W. H. Laarhoven, *J. Am. Chem. Soc.*, 1978, **100**, 3819.
15. H. Gunter, *NMR Spectroscopy. An Introduction*, Wiley, New York—London, 1980.
16. B. I. Ionin, B. A. Ershov, and A. I. Kol´tsov, *YaMR-spektroskopiya v organicheskoi khimii* [*NMR Spectroscopy in Organic Chemistry*], Khimiya, Leningrad, 1983, 142 (in Russian).

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