Synthesis of indolines and tetrahydroquinolines from *ortho*-(alk-2-enyl)anilines

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Intramolecular cyclization of *o*-alkenylanilines was studied. Heating of *o*-(cyclopent-2-en-1-yl)arylammonium chlorides at 200—220 °C yields cyclopenta[*b*]indolines as the main reaction products. Cyclization of 4-methyl-2-(pent-3-en-2-yl)aniline under the same conditions gave a mixture of indolines and tetrahydroquinolines. An alk-1-enylarylamine containing a vinylic double bond does not form cyclization products on the nitrogen atom.

Key words: 2-methyl-6-(cyclopent-2-en-1-yl)aniline, 2,4-dimethyl-6-(cyclopent-2-en-1-yl)aniline, intramolecular cyclization, cyclopenta[*b*]indolines, 2,4,6-trimethyl-1,2,3,4-tetrahydroquinolines, 3,5-dimethyl-2-ethylindoline, 1,2,3,3a,4,8b-hexahydrocyclopenta[*b*]indoles.

Depending on catalysts, intramolecular cyclization of 2-(alk-2-enyl)anilines can occur on either the aromatic ring or the nitrogen atom.[1,2] The character of substituents in the allylic fragment significantly influences the structure of the final products.[3] Earlier, it was shown that cyclopenta[*b*]indolines result only from a photochemical cyclization of 2-(cyclopent-2-en-1-yl)-*N*-ethylaniline or *N*-methyl-*N*-(cyclopent-1-en-1-yl)aniline,[4] whereas *N*-unsubstituted 2-(cyclopent-2-en-1-yl)aniline does not undergo photochemical transformation into indoline. Scarce literature data on acid-catalyzed heterocyclization of alkenylarylamines mainly deals with compounds containing acyclic allylic fragments. It is of theoretical and practical interest to study the catalyzed cyclization of *o*-(cycloalk-2-enyl)anilines because compounds having the indoline or tetrahydroquinoline structure are precursors of stable nitroxyl radicals[5] and can be used in the synthesis of valuable alkaloids.[6,7]

In the present work, we studied intramolecular cyclization of *o*-(alk-2-en-1-yl)anilines in the presence of HCl at 200—220 °C. It was shown that this reaction is a convenient method for the synthesis of cyclopenta[*b*]indolines (Scheme 1). Thus, when heated under these conditions, *o*-cyclopentenylarylammonium (**2a—e**) chlorides, obtained according to the known procedure[8] by condensation of anilines **1a—e** with chlorocyclopentene, are isomerized into indolines **3a—e**. Heating of *N*-cyclopentenylarylammonium[9] (**4a—e**), chlorides in an excess of the corresponding amine **1a—e** at 200—220 °C for 9—10 h also yields heterocycles **3a—e**. An analysis of this reaction mixture in the course of the heating shows the presence of the corresponding *o*-cyclopentenylanilines **2a—e**, which are gradually transformed into indolines **3a—e**. Thus, the condensation of arylamines **1a—e** with chlorocyclopentene[8] followed by heating to 200—220 °C allows the one-pot synthesis of indolines **3a—e** in high yields.

The structures of heterocycles **3a—e** were unambiguously proven by ^{1}H and ^{13}C NMR and IR spectroscopy and elemental analysis. Thus, the IR spectra[9] of compounds **3a—e** exhibit a band at 3390 cm^{-1} characteristic of the NH group.[10] The formation of tricyclic compounds **3a—e** is evidenced by signals for the angular methine protons observed in ^{1}H NMR spectra at δ 3.80 and 4.40. These signals have a large vicinal coupling constant (J_{HH} = 8.8 Hz), which suggests their *cis*-arrangement.[11]

Signals for the angular methine carbon atoms located in α- and β-positions with respect to the nitrogen atom (δ 63.0 and 47.0, respectively) are also observed in the ^{13}C NMR spectra of compounds **3a—e** recorded with the use of a pulse sequence of *J*-modulated spin echo.[12]

Cyclization of alkenylarylamines **2a—e** into indolines **3a—e** under severe conditions allows interaction between the positively charged nitrogen atom and the π orbital of the cyclopentenyl ring. There are two possible pathways for the formation of the C—N bond (Scheme 2, intermediate **A** or **B**), which give the quinoline (**6a—e**) or indoline (**3a—e**) structures. Our experimental data support the latter pathway.

We attempted to perform cyclization of 2,4-dimethyl-6-(cyclopent-1-en-1-yl)aniline (**5**) in the presence of HCl. Compound **5** was obtained by heating arylamine **2c** in the presence of KOH at 300 °C for 45 min (yield 93%). Its structure was confirmed spectroscopically; the

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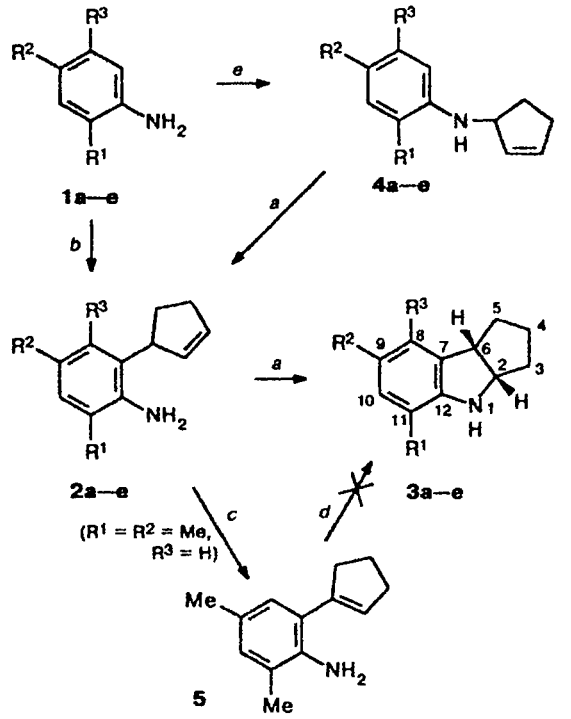
Scheme 1



a: $R^1 = R^2 = R^3 = H$
b: $R^1 = Me$, $R^2 = R^3 = H$
c: $R^1 = R^2 = Me$, $R^3 = H$
d: $R^1 = OMe$, $R^2 = R^3 = H$
e: $R^1 = R^3 = Me$, $R^2 = H$

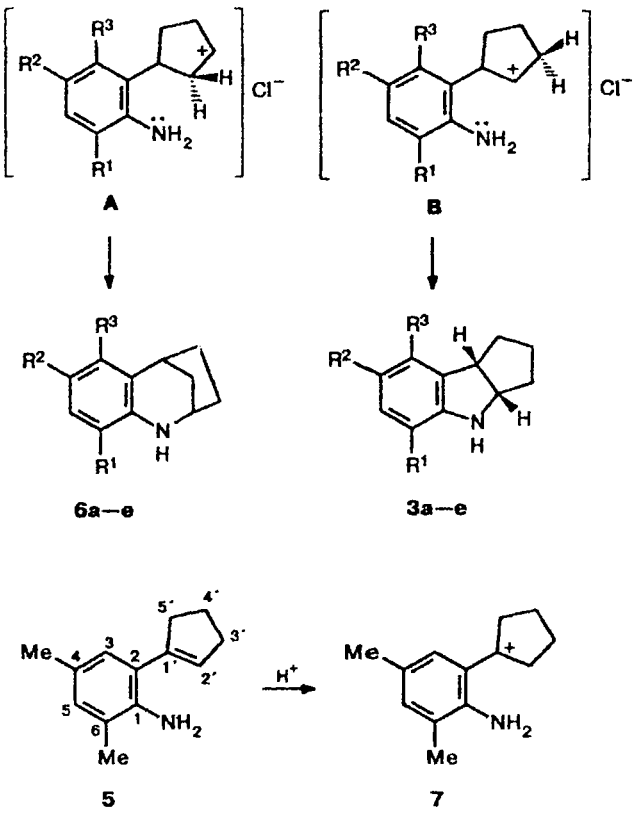
Reagents and conditions: *a*. (1) HCl(gas)/hexane, 20 °C; (2) **1a—e** (base), 200—220 °C; *b*. 1-Chlorocyclopent-2-ene (see Ref. 8); *c*. KOH, 300—310 °C; *d*. (1) HCl (gas)/hexane, 20 °C; (2) **1c** (base), 200—220 °C; *e*. 1-Chlorocyclopent-2-ene, Et_3N (see Ref. 9).

positions of ^1H and ^{13}C signals correspond to the values calculated from the increments of substituents.[12]

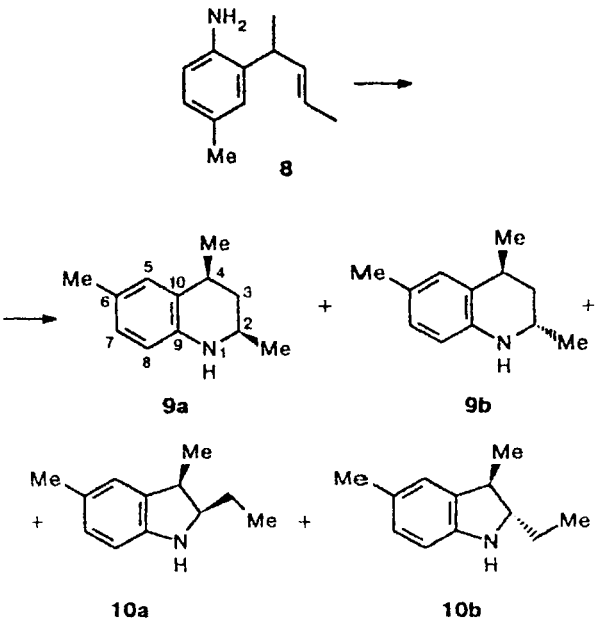
Heating of arylammonium **5** chloride at 200—220 °C leads to complete disappearance of the initial compound **5**, but the formation of indoline **3c**, though possible, is not observed. Apparently, protonation of the double bond that is shifted toward the aromatic ring results in the resonance-stabilized carbocation **7** only, which further reacts with another molecule of arylamine to give side products.

Unlike *o*-cyclopentenylarylamines **2a—e**, 4-methyl-2-(pent-3-en-2-yl)aniline (**8**) under the experimental conditions undergoes cyclization into *cis*- and *trans*-2,4,6-trimethyl-1,2,3,4-tetrahydroquinolines (**9a,b**) and, probably, *cis*- and *trans*-3,5-dimethyl-2-ethylindolines (**10a,b**) (Scheme 3). This fact is surprising because it does not fall into the general pattern of cyclization of alicyclic alkenylarylamines under these conditions.[13—15] Only isomers of quinoline **9a,b**, crystallized from the distillate, were isolated in the pure state (see Experimental).

Scheme 2



Scheme 3



The IR spectra of compounds **9a,b** exhibit a characteristic band of the NH group at 3395 cm^{-1}. In the

^{1}H NMR spectrum of quinoline **9a**, one can observe multiplets of the H(2) and H(4) junction methine protons (δ 3.47 and 3.00, respectively) and doublets for the methyl protons at the C(2) and C(4) atoms (δ 1.21 and 1.33, respectively). Signals of the $H(3)_{a,b}$ methylene protons are observed at δ 1.40 and 2.00. A downfield shift of the resonance signal of one of these protons by 0.6 ppm is caused by superposition of the positive β-effects of deshielding of the *cis*-methyl substituents.[16] The aromatic region of the ^{1}H NMR spectrum of quinolines **9a,b** shows two one-proton doublets and a one-proton singlet. In the ^{13}C NMR spectrum of quinoline **9a** recorded with the use of a pulse sequence of *J*-modulated spin echo, signals for the C(2) and C(4) atoms are observed at δ 47.6 and 30.9, respectively. The positions of the signals for the aromatic carbon atoms correspond to the values calculated from the additive parameters.

In the ^{1}H NMR spectrum of *trans*-quinoline **9b**, two doublet signals of the methyl groups are observed at δ 1.22 and 1.29. Signals of the H(2) and H(4) methine protons appear at δ 3.45 and 2.86, respectively, and signals of the $H(3)_{e,b}$ methylene protons are observed at δ 1.67. In the ^{13}C NMR spectrum of *trans*-quinoline **9b**, signals of the C(2) and C(4) junction atoms are shifted upfield (δ 42.4 and 30.2, respectively) because of stronger shielding compared to the *cis*-isomer.[17]

Experimental

IR spectra were recorded on a UR-20 instrument. ^{1}H and ^{13}C spectra were recorded on a Bruker AM 300 instrument (300 and 75 MHz, respectively) in $CDCl_3$ with Me_4Si as the internal standard. The purity of reaction products was checked on a Chrom-5 chromatograph (flame-ionization detector, l = 1.2 m, Chromaton N-AW with 5% SE-30, helium as the carrier gas, 12 deg min^{-1}). Column chromatography was performed on silica gel L 5/40 μm with hexane as the eluent.

Cyclization of *o*-alkenylamines (general procedure). ***A.*** Gaseous HCl was passed through a solution of compound **2a—e**, **4a—e**, **5**, or **8** in 70 mL of hexane until a precipitate ceased to form. The chloride obtained was filtered off, dried, and mixed with amine **1a—e** (15 mL) (in the case of amine **8**, with *para*-toluidine) in a round-bottomed flask equipped with a reflux condenser. The reaction mixture was refluxed on an oil bath at 200—220 °C until the initial alkenylaniline disappeared (GLC). Then it was cooled to 20—22 °C, washed with a 20% aqueous solution of NaOH (2×50 mL), dried over KOH, and fractionated by distillation *in vacuo*.

B. Gaseous HCl was passed through 25 mL of cyclopentadiene at −40 °C to a volume of 28 mL (15 min). The resulting chlorocyclopentene was mixed with the corresponding arylamine **1a—e** (100 mL) and kept at 140 °C for 4 h. Then, the reaction mixture was heated on an oil bath to 200—220 °C, refluxed until cyclopentenylaniline **2a—e** disappeared (GLC), and treated as described in method *A*.

1,2,3,3a,4,8b-Hexahydrocyclopenta[*b*]indole (3a). Yield 70% (*B*); b.p. 111 °C (3 Torr). Found (%): C, 82.90; H, 7.92; N, 8.63. $C_{11}H_{13}N$. Calculated (%): C, 83.02; H, 8.18; N, 8.81. IR, ν/cm^{-1}: 3390 (NH). ^{1}H NMR, δ: 1.50—2.00 (m, 6 H, 3 CH_2); 3.80 (br.s, 1 H, NH); 3.85 (m, 1 H, H(6)); 4.40 (m, 1 H, H(2)); 6.55—7.10 (m, 4 H, ArH). ^{13}C NMR, δ: 24.5 (C(4)); 35.0 (C(3)); 37.0 (C(5)); 47.3 (C(6)); 63.4 (C(2)); 108.7 (C(11)); 118.5 (C(8)); 124.6 (C(9)); 124.6 (C(10)); 133.5 (C(7)); 151.3 (C(12)).

5-Methyl-1,2,3,3a,4,8b-hexahydrocyclopenta[*b*]indole (3b). Yield 80% (*A*) and 70% (*B*); b.p. 110—113 °C (3 Torr). Found (%): C, 83.04; H, 8.29; N, 7.89. $C_{12}H_{15}N$. Calculated (%): C, 83.24; H, 8.67; N, 8.09. IR, ν/cm^{-1}: 3390 (NH). ^{1}H NMR, δ: 1.55—2.05 (m, 6 H, 3 CH_2); 2.12 (s, 3 H, CH_3); 3.55 (br.s, 1 H, NH); 3.81 (ddd, 1 H, H(2), $J_{H(2),H(6)}$ = 8.8 Hz, $J_{H(2),H(3)_a}$ = 2.6 Hz, $J_{H(2),H(3)_b}$ = 11.3 Hz); 4.40 (m, 1 H, H(6)); 6.66 (t, 1 H, H(9), J = 7.4 Hz); 6.86 (d, 1 H, H(10)); 6.93 (d, 1 H, H(8)). ^{13}C NMR, δ: 24.7 (C(4)); 36.2 (C(3)); 37.3 (C(5)); 47.7 (C(6)); 63.5 (C(2)); 118.1 (C(11)); 118.6 (C(9)); 122.2 (C(8)); 128.4 (C(10)); 132.9 (C(7)); 149.8 (C(12)).

5,7-Dimethyl-1,2,3,3a,4,8b-hexahydrocyclopenta[*b*]indole (3c). Yield 80% (*A*); b.p. 116—119 °C (3 Torr). Found (%): C, 83.25; H, 8.97; N, 7.23. $C_{13}H_{17}N$. Calculated (%): C, 83.42; H, 9.09; N, 7.49. IR, ν/cm^{-1}: 3390 (NH). ^{1}H NMR, δ: 1.60—2.05 (m, 6 H, 3 CH_2); 2.18 (s, 3 H, CH_3); 2.32 (s, 3 H, CH_3); 3.50 (br.s, 1 H, NH); 3.83 (ddd, 1 H, H(6), $J_{H(6),H(2)}$ = 8.8 Hz, $J_{H(6),H(5)_b}$ = 2.5 Hz); 4.42 (ddd, 1 H, H(2), $J_{H(2),H(3)_a}$ = 6.1 Hz, $J_{H(2),H(3)_b}$ = 2.6 Hz); 6.76 (s, 1 H, ArH); 6.83 (s, 1 H, ArH). ^{13}C NMR, δ: 16.9 (CH_3); 20.7 (CH_3); 24.5 (C(4)); 34.8 (C(3)); 37.1 (C(5)); 47.6 (C(6)); 63.5 (C(2)); 118.1 (C(11)); 122.6 (C(8)); 127.9 (C(9)); 128.8 (C(10)); 133.1 (C(7)); 147.6 (C(12)).

5-Methoxy-1,2,3,3a,4,8b-hexahydrocyclopenta[*b*]indole (3d). Yield 70% (*B*); b.p. 134 °C (3 Torr). Found (%): C, 76.02; H, 7.76; N, 7.15. $C_{12}H_{15}NO$. Calculated (%): C, 76.19; H, 7.94; N, 7.41. IR, ν/cm^{-1}: 3400 (NH). ^{1}H NMR, δ: 1.67—2.15 (m, 6 H, 3 CH_2); 3.91 (s, 3 H, CH_3); 3.89 (m, 2 H, H(6), NH); 4.50 (m, 1 H, H(2)); 6.70—6.90 (m, 3 H, ArH). ^{13}C NMR, δ: 24.4 (C(4)); 34.5 (C(3)); 36.7 (C(5)); 47.8 (C(6)); 55.3 (CH_3); 63.7 (C(2)); 108.7 (C(10)); 118.7 (C(8)); 119.6 (C(9)); 134.0 (C(12)); 140.0 (C(7)); 144.5 (C(11)).

5,8-Dimethyl-1,2,3,3a,4,8b-hexahydrocyclopenta[*b*]indole (3e). Yield 70% (*A*); b.p. 115 °C (3 Torr). Found (%): C, 83.24; H, 9.15; N, 7.35. $C_{13}H_{17}N$. Calculated (%): C, 83.42; H, 9.09; N, 7.49. IR, ν/cm^{-1}: 3390 (NH). ^{1}H NMR, δ: 1.72—2.10 (m, 6 H, 3 CH_2); 2.22 (s, 3 H, CH_3); 2.41 (s, 3 H, CH_3); 3.70 (br.s, 1 H, NH); 3.88 (m, 1 H, H(6)); 4.52 (m, 1 H, H(2)); 6.61 (d, 1 H, H(10), J = 7.4 Hz); 6.93 (d, 1 H, H(9)). ^{13}C NMR, δ: 16.8 (CH_3); 18.4 (CH_3); 24.8 (C(4)); 33.7 (C(3)); 36.8 (C(5)); 46.8 (C(6)); 64.0 (C(2)); 115.5 (C(11)); 119.8 (C(9)); 128.5 (C(10)); 131.5 (C(8)); 134.8 (C(7)); 150.0 (C(12)).

2,4-Dimethyl-6-(cyclopent-1-en-1-yl)aniline (5). Arylamine **2c** (5 g) and crystalline KOH (10 g) were refluxed at 300—310 °C for 45 min. The reaction mixture was cooled to 20—22 °C, and the product was decanted and distilled *in vacuo*. Yield 4.65 g (93%), b.p. 114 °C (3 Torr). Found (%): C, 82.77; H, 9.39; N, 7.26. $C_{13}H_{18}N$. Calculated (%): C, 82.98; H, 9.57; N, 7.45. IR, ν/cm^{-1}: 3380, 3460 (NH_2). ^{1}H NMR, δ: 2.05 (m, 2 H, CH_2); 2.20 (s, 3 H, CH_3); 2.30 (s, 3 H, CH_3); 2.60 (m, 2 H, CH_2); 2.73 (m, 2 H, CH_2); 3.60 (br.s, 2 H, NH_2); 6.02 (s, 1 H, =CH); 6.80 (s, 1 H, ArH); 6.90 (s, 1 H, ArH). ^{13}C NMR, δ: 18.0 (CH_3); 20.8 (CH_3); 23.7 (C(4′)); 34.2 (C(5′)); 37.0 (C(3′)); 125.6 (C(2)); 126.5 (C(5)); 127.1 (C(6)); 128.4 (C(2′)); 129.4 (C(4)); 130.1 (C(3)); 140.2 (C(1′)); 142.2 (C(1)).

***cis*-, *trans*-2,4,6-Trimethyl-1,2,3,4-tetrahydroquinolines (9a,b).** The mixture of products of cyclization of amine **8** obtained by method *A* was distilled *in vacuo*. Crystalline

cis-quinoline **9a** (admixed with *trans*-compound **9b**) that formed in the distilled solution was washed with a minimum amount of hexane and recrystallized from hot hexane. *cis*-Isomer **9a**: yield 30%, m.p. 59 °C. Found (%): C, 82.11; H, 9.63; N, 7.82. $C_{12}H_{17}N$. Calculated (%): C, 82.29; H, 9.71; N, 8.00. IR, ν/cm^{-1}: 3395 (NH). ^1H NMR, δ: 1.21 (d, 3 H, C(2)—CH_3, J = 5.7 Hz); 1.33 (d, 3 H, C(4)—CH_3, J = 6.8 Hz); 1.40 (ddd, 1 H, $H(3)_a$, $J_{H(3)_a,H(3)_b}$ = −12.8 Hz); 2.00 (ddd, 1 H, $H(3)_b$); 2.26 (s, 3 H, C(6)—CH_3); 3.00 (ddq, 1 H, H(4)); 3.47 (ddq, 1 H, H(2), $J_{H(2),H(3)_a}$ = 2.3 Hz, $J_{H(2),H(3)_b}$ = 10.0 Hz); 3.65 (br.s, 1 H, NH); 6.48 (d, 1 H, H(8)); 6.85 (d, 1 H, H(7), J = 8.0 Hz); 7.03 (s, 1 H, H(5)). ^{13}C NMR, δ: 20.5 (CH_3); 20.8 (CH_3); 20.9 (CH_3); 30.9 (C(4)); 40.9 (C(3)); 47.5 (C(2)); 114.2 (C(8)); 126.4 (C(6)); 127.3 (C(7)); 127.4 (C(5)); 134.1 (C(10)); 142.5 (C(9)).

After the mother liquor was allowed to stand for ~72 h, *trans*-isomer **9b** precipitated with a minor admixture of *cis*-product **9a**, yield 11%. Found (%): C, 82.11; H, 9.63; N, 7.82. $C_{12}H_{17}N$. Calculated (%): C, 82.29; H, 9.71; N, 8.00. IR, ν/cm^{-1}: 3395 (NH). ^1H NMR, δ: 1.22 (d, 3 H, C(2)—CH_3, J = 5.9 Hz); 1.29 (d, 3 H, C(4)—CH_3, J = 6.8 Hz); 1.67 (m, 2 H, $C(3)H_2$); 2.24 (s, 3 H, C(6)—CH_3); 2.86 (m, 1 H, H(4)); 3.42 (br.s, 2 H, NH); 3.46 (m, 1 H, H(2)); 6.46 (d, 1 H, H(8), J = 8.0 Hz); 6.82 (d, 1 H, H(7)); 6.90 (s, 1 H, H(5)). ^{13}C NMR, δ: 18.3 (CH_3); 20.9 (CH_3); 24.9 (CH_3); 30.2 (C(4)); 37.4 (C(3)); 42.4 (C(2)); 114.3 (C(8)); 126.6 (C(6)); 127.3 (C(5)); 127.5 (C(7)); 134.8 (C(10)); 141.8 (C(9)).

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