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SYNTHESIS OF 1- AND 2-CARBAMOYLALKYL-2,3-DIHYDRO-1H-INDOLE AND 9-CARBAMOYL-ALKYL-2,3,4,4a,9,9a-HEXAHYDRO-1H-CARBAZOLE DERIVATIVES

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Treatment of 1,2,3,9a-tetrahydro-9H-imidazo- and 1,2,3,4,10,10a-hexahydropyrimido[1,2-a]indol-2-one derivatives with formic acid gave 1-carbamoylalkyl-2,3,3-trimethyl-2,3-dihydro-1H-indoles. 9-Carbamoylmethyl- and 9-(2-carbamoylethyl)-4a-methyl-2,3,4,4a,9,9a-hexahydro-1H-carbazoles were prepared from 5,6,7,7a-tetrahydro-1H,4H-imidazo- and 1,2,6,7,8,8a-hexahydro-5H-pyrimido[2,1-k]-carbazolones in a similar manner. Synthesis of 2-(2-carbamoylpropyl)-2,3,3-trimethyl-2,3-dihydro-1H-indole was carried out by reduction of 1,3-dihydrospiro[2H-indolo-2,2'-piperidine] derivative with Zn in acetic acid solution.

Keywords: 1,2,3,9a-tetrahydro-9H-imidazo[1,2-*a*]indol-2-ones, 1,2,3,4,10,10a-hexahydropyrimido-[1,2-*a*]indol-2-ones, 1-carbamoylalkyl-2,3-dihydroindoles, ring opening.

It has been reported previously [1, 2] that reaction of 2,3,3-trimethyl-3H-indole with α-chloroacetamides and subsequent treatment of 1-carbamoylmethyl-3H-indolium chlorides formed with bases leads to the formation of 1,2,3,9a-tetrahydro-9H-imidazo[1,2-*a*]indol-2-one derivatives or the corresponding 1-carbamoylmethyl-2-methylene-2,3-dihydro-1H-indoles. The latter undergo cyclization to 1,2,3,9a-tetrahydro-9H-imidazo[1,2-*a*]indol-2-ones under the influence of weak carboxylic acids as acetic or propionic acid. When derivatives of 1-carbamoylmethyl-2-methylene-2,3-dihydro-1H-indole are reacted with lithium aluminum hydride, derivatives of 1,2,3,9a-tetrahydro-9H-imidazo[1,2-*a*]indole are formed [3]. The reaction of 2,3,3-trimethyl-3H-indolium salts with acrylamide gave 1,2,3,4,10,10a-hexahydropyrimido[1,2-*a*]indolone derivatives [4, 5]. Under the influence of strong protic acids derivatives of 1,2,3,9a-tetrahydro-9H-imidazo- and 1,2,3,4,10,10a-hexahydropyrimido[1,2-*a*]indole undergo decyclization and are converted to 1-carbamoylalkyl-3H-indolium salts.

In the present paper we wish to report a procedure for the preparation of 1-carbamoylalkyl-2,3-dihydro-1H-indoles by reductive cleavage of 1,2,3,9a-tetrahydro-9H-imidazo[1,2-*a*]indol-2-one and of derivatives of similar heterocyclic systems. It is known that 1,2,3,3-tetramethyl-2,3-dihydro-1H-indole can be synthesized by reduction of 1,3,3-trimethyl-2-methylene-2,3-dihydro-1H-indole with Raney nickel in a methanol solution of hydrochloric acid [6].

We found that the heating of 9,9,9a-trimethyl-1,2,3,9a-tetrahydro-9H-imidazo[1,2-*a*]indol-2-one (**1a**) with formic acid leads to the formation of 1-carbamoylmethyl-2,3,3-trimethyl-2,3-dihydro-1H-indole (**2a**) with good chemoselectivity.

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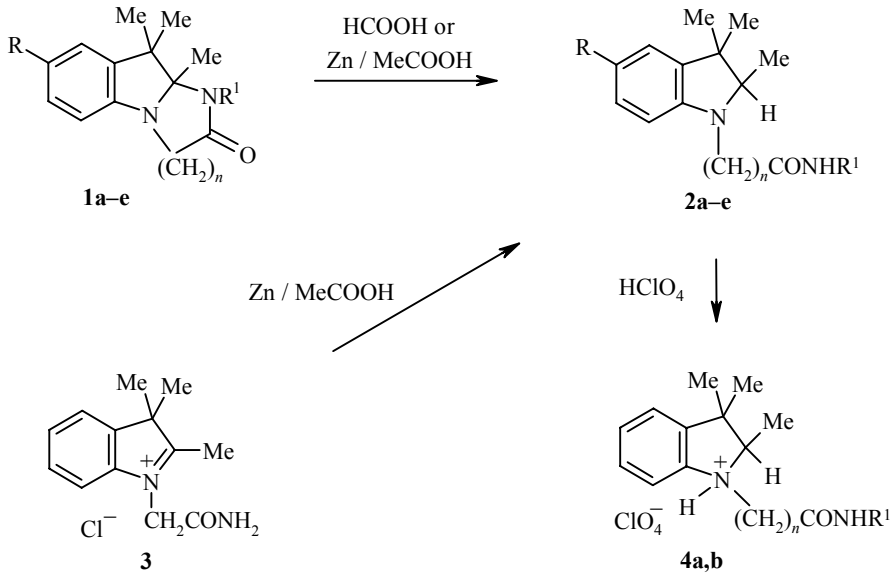
The ^{1}H NMR spectrum of **2a** showed signals of the diastereotopic geminal methyl groups at 1.02 and 1.27, together with a doublet of 2-CH_3 at 1.16, while the quadruplet of the 2-H appeared at 3.12 ppm (J = 6.5 Hz). The IR spectrum of **2a** contains absorption bands at 3440 (N–H), 3223 (N–H) and 1700 cm^{-1} (C=O) characteristic for the primary amides.

1-Carbamoylmethyl-2,3-dihydro-1H-indoles (**2b,c**) were synthesized from **1b,c** in a similar way as **2a**.



1, **2 a** R = R^1 = H, n = 1; **b** R = Me, R^1 = H, n = 1; **c** R = H, R^1 = CH_2Ph, n = 1; **d** R = R^1 = H, n = 2; **e** R = Me, R^1 = H, n = 2; **3 a** R = H, **b** R = Me; **4a** n = 1, **b** n = 2

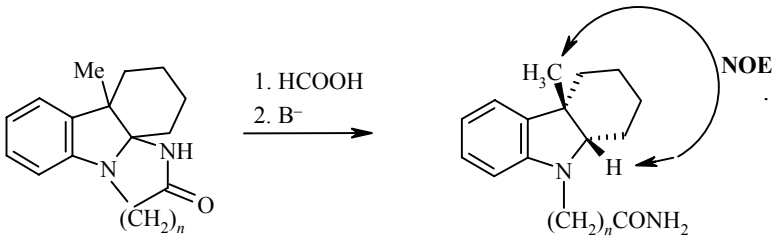
The mechanism of the reaction can be explained by assuming that the 1,2,3,9a-tetrahydro-9H-imidazo[1,2-*a*]indol-2-ones (**1a-c**) exist in a solution of week protonic acid in equilibrium with open-chain 1-carbamoylmethyl-2-methyl-3H-indolium derivatives, and can isomerize further to the corresponding methylene base. It is known that formic acid acting as a hydride ion donor easily reduces enamines [7]. In our case, the lactam ring opening of **1a-e** initiated by a proton is followed by addition of a hydride ion to the α-carbon atom of the indole ring system with the formation of 2,3-dihydro-1H-indole derivatives. It is necessary also to mention that formic acid does not reduce 1-carbamoylmethyl-2,3,3-trimethyl-3H-indolium chloride (**3**). However, 1-carbamoylmethyl-2,3-dihydro-1H-indole (**2a**) was easily obtained from the salt **3** by heating of the latter with Zn in acetic acid.

Reaction of 10,10,10a-trimethyl-1,2,3,4,10,10a-hexahydropyrimido[1,2-*a*]indol-2-ones (**1d,e**) with formic acid afforded 1-(2-carbamoylethyl)-2,3,3-trimethyl-2,3-dihydro-1H-indoles (**2d**,**e**). The reduction of compound **1d** was also carried out using Zn in acetic acid.

Treatment of **2a**,**d** with perchloric acid gave the indolium perchlorates **4a**,**b**.

It is known that the hexahydrocarbazole nucleus is a part of a ring system of such indole-derived alkaloids as strychnine and aspidospermidine [8-10]. Recently it was found that 9-(3-aminopropyl)-4a-methyl-2,3,4,4a,9,9a-hexahydro-1H-carbazoles show affinity for dopamine and serotonin receptors [11].

Heating with formic acid of 5,6,7,7a-tetrahydro-1H,4H-imidazo- and 1,2,6,7,8,8a-hexahydro-5H-pyrimido[2,1-*k*]carbazolones (**5a**,**b**), obtained by the methods of [12], gave, correspondingly, 9-carbamoylmethyl- and 9-(2-carbamoylethyl)-4a-methyl-2,3,4,4a,9,9a-hexahydro-1H-carbazoles (**6a**,**b**).



5a,b **6a,b**

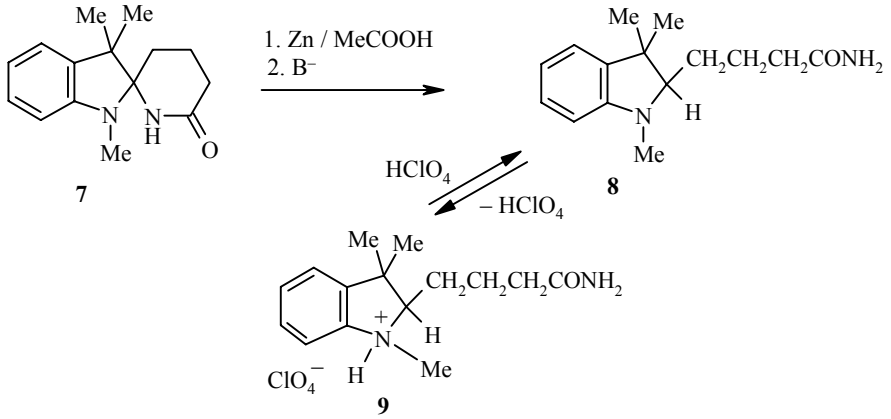
5, 6 a $n = 1$, **b** $n = 2$

The structures of **6a,b** were determined by NMR spectroscopy. The ^{1}H NMR spectrum of **6b** contains the methyl signal at δ 1.29, a multiplet of four methylene groups in the area 1.38-1.80, a triplet at 2.44 and multiplet of the NCH_2CH_2CO moiety at 3.37-3.47, a broad NH_2 singlet at 5.91, and four aromatic methine singlets in the area 6.54-7.07 ppm. The characteristic triplet of 9a-H proton is located at 3.06 ppm ($J = 3.95$ Hz).

The ^{13}C NMR and DEPT spectra of **6b** showed the presence of one methyl carbon, six methylene carbons, five methine carbons, including the α-carbon attached to the indole ring nitrogen (70.52 ppm), one amide carbonyl carbon and three quaternary carbons. For a complete ^{1}H and ^{13}C signal assignment two-dimensional ^{1}H–^{1}H COSY and ^{13}C–^{1}H COSY NMR spectra were used.

The relative stereochemistry of **6b** was determined on the basis of the results of a nuclear Overhauser effect spectroscopy (NOESY) experiment (300 MHz, $CDCl_3$). The 9a-H showed cross peak to 4a-CH_3, and thereby the five-membered ring and the six-membered ring fused together with a *cis* relationship.

It has been reported previously [13] that the reaction of 1,3,3-trimethyl-2-methylene-2,3-dihydro-1H-indole with acrylamide affords 1,3-dihydrospiro[2H-indolo-2,2'-piperidine] (**7**). The piperidine ring of **7** can be easily cleaved by treatment with strong protic acids.



7 **8** **9**

However, compound **7** was not reduced with formic acid under similar conditions as compound **1a**. 2-(3-Carbamoylpropyl)-1,3,3-trimethyl-2,3-dihydro-1H-indole (**8**) was obtained by heating of 1,3-dihydrospiro[2H-indolo-2,2'-piperidine] (**7**) in acetic acid in the presence of Zn. Treatment of **8** with perchloric acid gave perchlorate **9**.

EXPERIMENTAL

^{1}H NMR spectra were obtained on Tesla BS-487C (80 MHz) and Bruker DPX 300 (300 MHz) spectrometers with TMS as internal standard. The ^{13}C NMR spectra were recorded on a Bruker DPX 300 instrument at 75 MHz. The IR spectra were recorded on a Perkin-Elmer 325 instrument for tablets with potassium bromide.

1-Carbamoylmethyl-2,3,3-trimethyl-2,3-dihydro-1H-indole (2a). A. A solution of compound **1a** (0.86 g, 4 mmol) was heated at 90°C for 3 h. The reaction mixture was poured into 20 ml of water, treated with sodium carbonate, and extracted with ether (2 × 15 ml). The extract was dried with sodium sulfate, the solvent removed, and the residue crystallized from acetone. The yield of compound **2a** was 0.53 g (61%); mp 115-116°C. ^{1}H NMR spectrum ($CDCl_3$): 1.02 (3H, s, 3-CH_3); 1.16 (3H, d, J = 7.5 Hz, 2-CH_3); 1.27 (3H, s, 3-CH_3); 3.12 (1H, q, J = 6.5 Hz, 2-H); 3.58 (2H, s, CH_2); 6.36-7.21 ppm (6H, m, ArH, NH_2). Found, %: C 71.47; H 8.42. $C_{13}H_{18}N_2O$. Calculated, %: C 71.53; H 8.31.

B. To a solution of chloride **3** (2.44 g, 10 mmol) in glacial acetic acid (16 ml) was added zinc powder (1.96 g, 30 mmol) and the mixture was heated at 80°C. After 1 h, zinc powder (1.96 g, 30 mmol) was added to the solution again and the reaction mixture was kept at the same temperature for 1 h, poured into water (150 ml), and extracted with ether (2 × 20 ml). The extract was washed with water (20 ml) and dried over calcium chloride. The solvent was removed, and the residue chromatographed on a column (500 × 20 mm) of aluminum oxide, the fraction with R_f 0.47 (acetone–hexane, 1:1) being collected. Removal of the solvent gave 1.40 g (64 %) of compound **2a**.

1-Carbamoylmethyl-2,3,3,5-tetramethyl-2,3-dihydro-1H-indole (2b) was obtained similarly to **2a** (method A) from **1b** (0.42 g, 1.8 mmol) in 0.21 g (50%) yield; mp 114-115°C (acetone). ^{1}H NMR spectrum ($CDCl_3$): 0.84 (3H, s, 3-CH_3); 1.00 (3H, d, J = 7.5 Hz, 2-CH_3); 1.16 (3H, s, 3-CH_3); 2.17 (3H, s, 5-CH_3); 2.97 (1H, q, J = 6.5 Hz, 2-H); 3.42 (2H, s, NCH_2); 6.10-7.12 ppm (5H, m, ArH, NH_2). Found, %: C 72.13; H 8.89. $C_{14}H_{20}N_2O$. Calculated, %: C 72.38; H 8.68.

1-(N-benzylcarbamoylmethyl)-2,3,3-trimethyl-2,3-dihydro-1H-indole (2c) was obtained similarly to **2a** (method A) from **1c** (0.92 g, 3 mmol) in 0.31g (34 %) yield; mp 79-80°C (acetone). ^{1}H NMR spectrum ($CDCl_3$): 0.98 (3H, s, 3-CH_3); 1.14 (3H, d, J = 7.5 Hz, 2-CH_3); 1.26 (3H, s, 3-CH3); 3.02 (1H, q, J = 6.4 Hz, 2-H); 3.53 (2H, s, NCH_2CO); 4.22-4.48 (2H, m, $\underline{CH_2}Ph$); 6.30 (1H, br. s, NH); 6.61-7.22 ppm (4H, m, ArH). Found, %: C 77.45; H 8.04; N 9.27. $C_{20}H_{24}N_2O$. Calculated, %: C 77.88; H 7.84; N 9.08.

1-(2-Carbamoylethyl)-2,3,3-trimethyl-2,3-dihydro-1H-indole (2d) was obtained similarly to **2a** (method A) from **1d** (0.5 g, 2.2 mmol) in 0.18 g (36%) yield; mp 97-98°C (acetone). ^{1}H NMR spectrum (DMSO-d_6): 0.93 (3H, s, 3-CH_3); 1.13 (3H, d, J = 6.5 Hz, 2-CH_3); 1.25 (3H, s, 3-CH_3); 2.10-2.32 (2H, m, CH_2CO); 3.10 (1H, q, J = 6.5 Hz, 2-H); 3.22-3.50 (2H, m, NCH_2); 6.43-6.99 (5H, m, ArNH); 7.22 ppm (1H, br. s, NH). Found, %: C 72.70; H 8.72. $C_{14}H_{20}N_2O$. Calculated, %: C 72.38; H 8.68.

B. To a solution of **1d** (1.15 g, 5 mmol) in glacial acetic acid (8 ml) was added zinc powder (0.98 g, 15 mmol) and the mixture was heated at 80°C. After 1 h, zinc powder (0.98 g, 15 mmol) was added to the solution again and the reaction mixture was kept at the same temperature for 2 h, poured into water (100 ml), and extracted with ether (2 × 15 ml). The extract was washed with water (20 ml) and dried over calcium chloride. The solvent was removed, and the residue chromatographed on a column (300 × 20) of aluminum oxide, the fraction with R_f 0.52 (acetone–hexane, 1:1) being collected. Removal of the solvent gave 0.51 g (43%) of compound **2d**.

1-(2-Carbamoylethyl)-2,3,3,5-tetramethyl-2,3-dihydro-1H-indole (2e) was obtained similarly to **2a** (method A) from **1e** (0.98 g, 4 mmol) in 0.38 g (39%) yield; mp 115-116°C (acetone). ^{1}H NMR spectrum ($CDCl_3$): 0.96 (3H, s, 3-CH_3); 1.14 (3H, d, J = 6.6 Hz, 2-CH_3); 1.28 (3H, s, 3-CH_3); 2.15 (3H, s, 5-CH_3); 2.08-2.30 (2H, m, CH_2CO); 2.98 (1H, q, J = 6.5 Hz, 2-H); 3.18-3.55 (2H, m, NCH_2); 5.80-6.92 (5H, m, ArH, NH_2). Found, %: C 73.20; H 9.25; N 11.31. $C_{15}H_{22}N_2O$. Calculated, %: C 73.13; H 9.0; N 11.37.

1-Carbamoylmethyl-2,3,3-trimethyl-2,3-dihydro-1H-indolium Perchlorate (4a). A solution of **2a** (0.44 g, 2 mmol) in ethanol (3 ml) was treated with perchloric acid (60%) to pH 2. The mixture was held at 0°C for 24 h and the precipitated solid filtered off and recrystallized from ethanol to give 0.37 g (58%) of perchlorate **4a**; mp 251-252°C. ^{1}H NMR spectrum (CF_3COOH): 0.97 (3H, s, 3-CH_3); 1.16 (3H, s, 3-CH_3); 1.33 (3H, d, *J* = 6.5 Hz, 2-CH_3); 3.46-3.93 (1H, m, 2-H); 4.27 (2H, s, NCH_2); 7.03-7.41 ppm (4H, m, ArH). Found, %: Cl 11.40. $C_{13}H_{18}N_2O{\cdot}HClO_4$. Calculated, %: Cl 11.12.

1-(2-Carbamoylethyl)-2,3,3-trimethyl-2,3-dihydro-1H-indolium Perchlorate (4b) was obtained from **2d** (0.46 g, 2 mmol) similarly to perchlorate **4a** in 0.21 g (32%) yield; mp 164-165°C (ethanol). IR spectrum: 3435 (N–H), 3350 (N–H), 1675 (C=O), 1100, 655 cm^{-1} (ClO_4^-). ^{1}H NMR spectrum (CF_3COOH): 0.92 (3H, s, 3-CH_3); 1.16 (3H, s, 3-CH_3); 1.33 (3H, d, *J* = 6.0 Hz, 2-CH_3); 2.93 (2H, q, *J* = 6.5 Hz, CH_2CO); 3.43-4.68 (3H, m, 2-H, NCH_2); 7.06-7.43 ppm (4H, m, ArH). Found, %: Cl 11.00. $C_{14}H_{20}N_2O{\cdot}HClO_4$. Calculated, %: Cl 10.65.

9-Carbamoylmethyl-4a-methyl-2,3,4,4a,9,9a-hexahydro-1H-carbazole (6a) was obtained similarly to **2a** (method **A**) from **5a** (0.2 g, 0.8 mmol) in 0.095 g (47%) yield; mp 161-162°C (acetone). ^{1}H NMR spectrum ($CDCl_3$): 1.37 (CH_3); 1.40-1.61 (6H, m, 3 × CH_2); 1.62-1.92 (2H, m, $C_{(1)}H_2$); 3.08 (1H, t, *J* = 3.6 Hz, 9a-H), 3.51-3.68 (2H, AB-q, *J* = 17.7 Hz, NCH_2); 6.55 (1H, d, *J* = 7.2 Hz, 8-H); 6.64 (2H, br. s, NH_2); 6.82-6.87 (1H, m, 6-H); 7.05-7.07 (1H, dd, *J* = 0.9 and 7.2 Hz, 5-H); 7.09-7.14 ppm (1H, m, 7-H).^{13}C NMR spectrum ($CDCl_3$): 21.05; 21.25; 35.82 (C-2, C-3, C-4); 21.91 (CH_3); 29.37 (C-1); 41.39 (C-4a); 52.39 (NCH_2); 71.89 (C-9a); 108.75 (C-8); 119.09 (C-6); 121.17 (C-5); 127.29 (C-7); 139.95; 150.19 (C-4a, C-8a); 174.22 ppm (C=O). Found, %: C 73.78; H 7.81; N 11.51. $C_{15}H_{20}N_2O$. Calculated, %: C 73.74; H 8.25; N 11.46.

9-(2-Carbamoylethyl)-4a-methyl-2,3,4,4a,9,9a-hexahydro-1H-carbazole (6b) was obtained similarly to **2a** (method A) from **5b** (2.33 g, 9 mmol) in 0.97g (41%) yield; mp 128-129°C (acetone). ^{1}H NMR spectrum ($CDCl_3$): 1.29 (CH_3); 1.38-1.52 (6H, m, 3 × CH_2); 1.56-1.86 (2H, m, $C_{(1)}H_2$); 2.44 (2H, t, *J* = 7.7 Hz, CH_2CO); 3.06 (1H, t, *J* = 4.0 Hz, 9a-H); 3.35-3.50 (2H, m, NCH_2); 5.92 (2H, br. s, NH_2); 6.55 (1H, d, *J* = 7.8 Hz, 8-H); 6.68-6.73 (1H, m, 6-H); 6.96-6.99 (1H, dd, *J* = 0.9 and 7.2 Hz, 5-H); 7.04-7.07 ppm (1H, m, 7-H). ^{13}C NMR spectrum ($CDCl_3$): 21.37; 22.05; 35.93 (C-2, C-3, C-4); 23.33 (CH_3); 24.09 (C-1); 33.78 (CH_2CO); 42.44 (C-4a); 42.39 (NCH_2); 70.52 (C-9a); 108.16 (C-8); 118.66 (C-6); 121.54 (C-5), 127.60 (C-7); 140.06; 150.39 (C-4a, C-8a); 174.99 ppm (C=O). Found, %: C 74.56; H 8.88; N 11.05. $C_{16}H_{22}N_2O$. Calculated, %: C 74.38; H 8.58; N 10.84.

2-(3-Carbamoylpropyl)-1,3,3-trimethyl-2,3-dihydro-1H-indolium Perchlorate (9) and 2-(3-Carbamoylpropyl)-1,3,3-trimethyl-2,3-dihydro-1H-indole (8). To a solution of **7** (0.98 g, 4 mmol) in glacial acetic acid (8 ml) was added zinc powder (0.78 g, 12 mmol) and the mixture was heated at 80°C. After 1 h, zinc powder (0.78 g, 12 mmol) was added to the solution again and the reaction mixture was kept at the same temperature for 3 h, poured into water (100 ml) and extracted with ether (2 × 15 ml). The extract was washed with water (15 ml), dried over calcium chloride, and the solvent was removed. The residue was dissolved in ethanol (2.5 ml) and to the solution was added 40% perchloric acid until pH 2. The solution was kept at 5°C for 24 h, and the precipitated solid filtered off and recrystallized from ethanol to give 0.72 g (52%) of perchlorate **9a**; mp 199-200°C. IR spectrum: 3445 (N–H), 3345 (N–H), 1660 (C=O), 1110, 655 cm^{-1} (ClO_4^-). ^{1}H NMR spectrum (CF_3COOH): 0.93 (3H, s, 3-CH_3); 1.21 (3H, s, 3-CH_3); 1.68-2.63 (6H, m, 3 × CH_2); 3.01-3.51 (1H, m, CH); 3.12 (3H, d, *J* = 5.0 Hz, 1-CH_3); 7.01-7.31 ppm (4H, m, ArH). Found, %: Cl 10.60. $C_{15}H_{22}N_2O{\cdot}HClO_4$. Calculated, %: Cl 10.22.

To a solution of perchlorate **9** (0.69 g, 2 mmol) in ethanol (5 ml) 5% sodium hydroxide (5 ml) was added. The mixture was poured into water (40 ml) and extracted with ether (2 × 15 ml). The extract was washed with water (15 ml), dried with calcium chloride, and the solvent removed to yield 0.38 g (77%) of the oily compound **8**. ^{1}H NMR spectrum ($CDCl_3$): 1.04 (3H, s, 3-CH_3); 1.32 (3H, s, 3-CH_3); 1.51-2.43 (6H, m, 3 × CH_2); 2.61-2.93 (1H, m, CH); 2.71 (3H, s, 1-CH_3); 5.74 (1H, br. s, NH); 6.14 (1H, br. s, NH); 6.41-7.21 ppm (4H, m, ArH). Found, %: C 73.15; H 9.20. $C_{15}H_{22}N_2O$. Calculated, %: C 73.13; H 9.00.

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