ISSN 1070-4280, Russian Journal of Organic Chemistry, 2009, Vol. 45, No. 2, pp. 266–269. © Pleiades Publishing, Ltd., 2009.
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Oxidative Coupling of 3,4,4a,5-Tetrahydropyrido-[1,2-*a*]benzimidazole Derivatives with Some Biologically Active Amines

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Received June 21, 2007; final version December 9, 2008

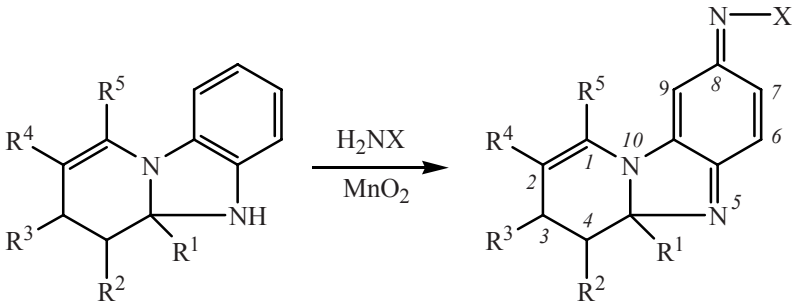
Abstract—The oxidative coupling of primary aliphatic and aromatic amines with 3,4,4a,5-tetrahydropyrido[1,2-*a*]-benzimidazole derivatives resulted in the formation of *p*-quinone diimines of the given series.

DOI: 10.1134/S1070428009020195

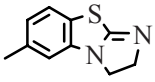
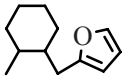
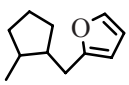
Heterocyclic quinoid compounds attracted recently a special interest. This is due to the fact that many natural and synthetic heterocyclic quinone mono- and diimines are contained in the structure of a number of antibiotics and alkaloids of marine organisms. Among the known procedures of preparation quinone diimines two routes can be separated: the synthesis by oxidation of aromatic diamines, and synthesis by oxidative coupling. The first roupe is well documented, but it is limited to the preparation of symmetric quinone diimines [1]. The oxidative coupling essentially comes to reactions of the type of indamine and indophenol condensations [2] and oxidative auto-condensation of primarily aromatic amines [3].

We showed formerly that the derivatives of 3,4,4a,5-tetrahydropyrido[1,2-*a*]benzimidazole readily enter into the oxidative coupling with versatile aromatic and aliphatic amines in the presence of MnO_2 giving *p*-quinone diimine structures [4]. In extension of this study we investigated the reaction of the mentioned substrates with some amines endowed with indubitable biological activity. We studied also the oxidative coupling with *p*-aminophenol, *p*-aminosalicylic, and anthranilic acids in order to prepare water-soluble quinoid compounds for this was a significant requirement for the biologically active preparations.

The oxidative coupling of substrates **I** with aliphatic [2-(2-furylmethyl)cyclohexyl(cyclopentyl)amines] and aromatic (sulfanylamide, anthranilic acid, *p*-aminophenol, *p*-aminosalicilic acid, 6-amino-2,3-dihydroimidazo[2,1-*b*]-benzothiazole) amines proceeded readily at room temperature in the presence of MnO_2 leading to the formation



Ia, Ib **IIa, IIb, IIIa, IVa, IVb–VIa, VIb, VIIa, VIIIa**

$R^1,R^2 = R^4,R^5 = (CH_2)_4$, R^3 = H (**a**); $R^1,R^2 = (CH_2)_4$, $R^3 = R^5$ = Ph, R^4 = H (**b**); X = 4-$NH_2SO_2C_6H_4$ (**II**), 2-$HOOCC_6H_4$ (**III**), 4-HOC_6H_4 (**IV**), 3-OH-4-$HOOCC_6H_3$ (**V**),  (**VI**),  (**VII**),  (**VIII**).

of the corresponding heterocyclic *p*-quinone diimines **II–VIII** (see the scheme). The reactions occur sufficiently regioselectively, and *p*-quinone monoimines, the products of concurrent oxidation, practically do not form.

All compounds obtained are brightly colored and relatively stable. Compounds **II–V** are notably soluble in water and in water solutions exhibit appreciable properties of acid-base indicators (acid medium: crimson solution; alkaline medium: violet solution). The combination of ionizible groups and strongly conjugated enaminoquinoid structure ensures the protolytic properties of these compounds; these quinoid compounds camn be applied as organic acid-base indicators.

We attempted to carry out cyclization of compound **III** in acetic acid or alcoholic alkali (formerly similar cyclizations had been preformed with binucleophiles like *o*-phenylenediamine, *o*-aminophenol, *o*-aminothiophenol [5]) but the expected lactone did not form presumably because of the difficulty in the closure of the seven-membered ring and of the low nucleophilicity of the carboxy group.

In the IR spectra of the reaction products are observed the absorption bands of the C^{1}=C^{2} bonds of the enamine groups, and also of quinoid bonds C=N and C=C. In the spectra of compounds **IIIa** and **Va, Vb** additionally appeared broad absorption bands of carboxy groups, in the spectra of compounds **IVa, IVb**, of hydroxy groups, in the spectra of compounds **IIa, IIb**, of the sulfamide group. Besided the spectra of compounds **VIa, VIb** contain medium bands at 1708–1712 cm^{-1} corresponding to the stretching vibrations of the imino group of the heterocyclic fragment.

The multiplicity and chemical shifts of signals from protons H^{6}, H^{7}, H^{9} in the ^{1}H NMR spectra of the reaction products testify to their *para*-quinoid structure and are analogous to the spectral findings of the previously prepared quinone diimines [4]. In the spectra of all compounds the signal of proton H^{9} is situated upfield with respect to the signals of protons H^{6} and H^{7}. The similar effect was observed in the spectra of initial compounds [6], apparently owing to the presence in the *ortho*-position to this proton of an electron-donor vinylamine moiety. Besides in the spectra of compounds **IVb–VIb** the signals of H^{9} are observed in a stronger field compared to analogous signals in the spectra of compounds **IVa–VIa**. Quantum-chemical calculations (ab initio, basic 6-31G*, gas phase) with the complete optimization of the geometry of the molecule indicated that proton H^{9} in compounds **IVb–VIb** is located at 3.14 Å from the center of the phenyl ring at the angle 61.8 deg between the plane of the phenyl ring and the straight line connecting the hydrogen atom and the center of the phenyl ring; in this case the shielding of the nucleus is fairly efficient.

In the ^{1}H NMR spectra of compounds **IIIa, IVa, Vb, VIa**, and **VIb** a doubling of signals of quinoid protons is observed [in compounds **VIa** and **VIb** barely visible], analogous to that found for the previously obtained quinone diimines [4, 5]. As before, we assign this phenomenon to the possible π-diastereomerism with respect to the exocyclic C=N bond with the prevalence of the *Z*-form. The lack of doubled signals in the spectra of compounds **IIa, IIb, IVb, Va, VIIa**, **VIIIa** may be due either to low inversion barrier (compounds **IIa** and **Va**), and in this case the chemical shift has the average value, or the compound exists in a single more energetically favorable form (**IIb, IVb, VIIa, VIIIb**). Both statements follow from the assumption that in going from compounds **a** to compounds **b** the degree of the intramolecular charge transfer from atom N^{10} to N^{11} decreases (with decreasing coplanarity of the system N^{10}–C^{9a}=C^{9}–C^{8}=N^{11}) considering the *meso*-ionic charater of the transition state; consequently, the activation energy of *Z*/*E*-inversion is higher.

In the mass spectra of all compounds synthesized the values of pseudomolecular ions $[M + H]^{+}$ obtained by mild chemical ionization were consistent with the calculated values. In the spectra of compounds **VIa** and **VIb** peaks were observed whose intensity grew proportionally to the contact time with the reverse phase of the chromatographic column; the peaks corresponded to the loss of two protons. We believe that this fact is due to the completion of the aromatization of the heterocyclic system.

EXPERIMENTAL

IR spectra were recorded on spectrophotometers Specord 75IR, Spectrum BX-II (Perkin Elmer) in CH_2Cl_2, KBr. ^{1}H NMR spectra were registered on a spectrometer Bruker AC-250 at operating frequency 250 MHz, internal reference TMS, solvent $CDCl_3$. Elemental analysis was carried out on an analyzer Flash EA 1112 CHN/MAS200.

HPLC–MS measurements were performed on a Hewlett-Packard 1100 LC/MSD instrument; (a) column Hypersil ODS (4 × 125 mm), eluent phase 2-propanol–water, 60:40, flow rate 0.3 ml/min, temperature 55°C, diode matrix; (b) direct admission to the ionizing chamber, APCI, positive polarity, fragmentor 70eV, mass range 150–800.

Melting points were measured in capillaries and on a Boetius heating block. The reaction progress was monitored and the homogeneity of compounds was checked by TLC on Silufol UV-254, Sorbfil plates in the systems hexane–ethyl acetate, 1:1, ethyl acetate, ethanol, AcOH–water, 1:10. The products were separated and purified by preparative TLC on plates 25 × 30 cm with Al_2O_3 (II grade by Brockmann), layer 1.5 mm thick, single charge 0.25 g.

All compounds were synthesized by a single procedure, only the ways of isolation and purification varied.

General procedure. To a solution of 1 mmol of compound **Ia** or **Ib** and 1.1 mmol of primary amine in 50 ml of acetone (for compounds **VIIa** and **VIIIa**, ethanol) was added 10–12 mmol of MnO_2, the mixture was stirred at room temperature for 1 h (compounds **IVa–VIa**), 2 h (compounds **IIa, IIb, IVb, Vb**), 4–5 h (compounds **IIIa, VIb, VIIa, VIIIa**) till TLC showed the disappearance of the initial compound, then MnO_2 was filtered off and washed with chloroform (compounds **IIa, IIb**), acetone (compounds **IIIa, IVa, IVb–VIa**, **VIb**), ethanol and dichloromethane (compounds **VIIa** and **VIIIa**). On cooling the filtrates of compounds **IIa**, **IIb, IIIa, VIa, VIb** a pure crystalline product precipitated (in isolation of compound **IIIa** an additional chromatographic purification was required, eluent chloroform–etanol, 5:1]. The filtrates of compounds **IVa**, **IVb, Va, Vb, VIIa, VIIIa** were diluted with water 2–3 times and saturated with NaCl (compounds **IVa, Va**, **Vb**) or Na_2CO_3 (compounds **IVb, VIIa, VIIIa**) till precipitate settled. In the case of compounds **Va, Vb** the precipitate was materially pure product, in the other cases the precipitate was additionally subjected to chromatograpgy using the following eluents: compounds **IVa, IVb**, eluent petroleum ether–ethyl acetate, 1:1; compounds **VIIa** and **VIIIa**, eluent chloroform–etanol, 10:1.

8-(4-Sulfamoylphenylimino)-1,2;4,4a-di(tetramethylene)-3,4,4a,8-tetrahydropyrido[1,2-*a*]-benzimidazole (IIa). Yield 67%, mp 168–170°C. IR spectrum, ν, Cm^{-1}: 3415, 3320 (NH_2), 1638 (C^1=C^2), 1588, 1574 (C=N, C=C_{quin}). ^{1}H NMR spectrum, δ, ppm: 0.80–2.30 m (19H), 5.05 br.s (2H, SO_2NH_2), 5.49 C (1H, H^9), 6.95 d (*AA*', 2H, C_6H_4, *J* 8.0 Hz), 7.02 d (1H, H^7, *J* 9.8 Hz), 7.05 d (1H, H^6, *J* 9.8 Hz), 7.85 d (*BB*', 2H, C_6H_4, *J* 8.0 Hz). Found, %: C 66.38; H 6.02; N 12.57. $[M + H]^+$ 449. $C_{25}H_{28}N_4O_2S$. Calculated, %: C 66.94; H 6.29; N 12.49. *M* 448.58.

8-(4-Sulfamoylphenylimino)-4,4a-tetramethylene-1,3-diphenyl-3,4,4a,8-tetrahydropyrido[1,2-*a*]-benzimidazole (IIb). Yield 90%, mp 265–267°C. IR spectrum, ν, cm^{-1}: 3316, 3260 (NH_2), 1652 (C^1=C^2), 1631, 1597, 1580 (C=N, C=C_{quin}). ^{1}H NMR spectrum, δ, ppm: 1.45–2.25 m (9H), 3.82 d.d (1H, H^3, *J* 9.8, 2.9 Hz), 4.19 d (1H, H^9, *J* 1.1 Hz), 4.80 br.s (2H, SO_2NH_2), 5.33 d (1H, H^2, *J* 2.9 Hz), 6.64 d (*AA*', 2H, C_6H_4, *J* 8.5 Hz), 6.95 d.d (1H, H^7, *J* 9.9, 1.1 Hz), 7.10 d (1H, H^6, *J* 9.9 Hz), 7.13–7.40 m (10H, C_6H_5), 6.54 d (*BB*', 2H, C_6H_4, *J* 8.5 Hz). Found, %: C 72.14; H 5.35; N 10.43. $[M + H]^+$ 547. $C_{33}H_{30}N_4O_2S$. Calculated, %: C 72.50; H 5.53; N 10.25. *M* 546.68.

8-(2-Carboxyphenylimino)-1,2;4,4a-di(tetramethylene)-3,4,4a,8-tetrahydropyrido[1,2-*a*]-benzimidazole (IIIa). Yield 88%, mp > 300°C. IR spectrum, ν, cm^{-1}: 3435 br, 1700 (COOH), 1660 (C^1=C^2), 1630 (C=N), 1574, 1565 (C=C_{quin}). ^{1}H NMR spectrum, δ, ppm: 0.80–2.30 m (19H), 6.12 C (1H, H^9, *Z*-form {5}*), 6.30 s (1H, H^9, *E*-form {1}), 7.07–7.37 m ($2H_{arom}$, H^6, H^7), 7.50 t (1H, C_6H_4, *J* 8.5 Hz), 8.35 d (1H, C_6H_4, *J* 8.5 Hz). Found, %: C 75.92; H 6.31; N 9.87. $[M + H]^+$ 414. $C_{26}H_{27}N_3O_2$. Calculated, %: C 75.52; H 6.58; N 10.16. *M* 413.51.

8-(4-Hydroxyphenylimino)-1,2;4,4a-di(tetramethylene)-3,4,4a,8-tetrahydropyrido[1,2-*a*]benzimidazole (IVa). Yield 73%, mp > 300°C. IR spectrum, ν, cm^{-1}: 3400–3150 (OH), 1666 (C^1=C^2), 1632 (C=N), 1588, 1574 (C=C_{quin}). ^{1}H NMR spectrum, δ, ppm: 0.80–2.30 m (19H), 5.70 d (1H, H^9, *J* 2.0 Hz, *Z*-form {6}), 5.80 d (1H, H^9, *J* 2.0 Hz, *E*-form {1}), 6.65–6.80 m (4H, C_6H_4), 6.83 d.d (1H, H^7, *J* 10.0, 2.0 Hz, *Z*-form {6}), 6.88 d.d (1H, H^7, *J* 10.0, 2.0 Hz, *E*-form {1}), 7.00 d (1H, H^6, *J* 10.0 Hz, *Z*-form {6}), 7.08 d (1H, H^6, *J* 10.0 Hz, *E*-form {1}), 9.25 s (1H, OH). Found, %: C 78.32; H 7.20; N 10.89. $[M + H]^+$ 386. $C_{25}H_{27}N_3O$. Calculated, %: C 77.89; H 7.06; N 10.90. *M* 385.50.

8-(4-Hydroxyphenylimino)-4,4a-tetramethylene-1,3-diphenyl-3,4,4a,8-tetrahydropyrido[1,2-*a*]-benzimidazole (IVb). Yield 74%, mp 130–132°C. IR spectrum, ν, cm^{-1}: 3400–3150 (OH), 1658 (C^1=C^2), 1626 (C=N), 1574, 1506 (C=C_{quin}). ^{1}H NMR spectrum, δ, ppm: 1.40–2.20 m (9H), 3.93 d.d (1H, H^3, *J* 10.0, 3.0 Hz), 4.78 d (1H, H^9, *J* 2.0 Hz), 5.38 d (1H, H^2, *J* 3.0 Hz), 6.38 d (*AA*', 2H, C_6H_4, *J* 8.0 Hz), 6.54 d (*BB*', (2H, C_6H_4, *J* 8.0 Hz), 6.89 d.d (1H, H^7, *J* 10.0, 2.0 Hz), 7.10–7.75 m (8H, C_6H_5), 7.90 d (2H, C_6H_5, *J* 7.0 Hz), 9.25 s (1H, OH). Found, %: C 82.13; H 6.23; N 8.85.

* In braces are given the relative intensities of signals of *E*- and *Z*-forms.

$[M + H]^+$ 484. $C_{33}H_{29}N_3O$. Calculated, %: C 81.96; H 6.04; N 8.69. *M* 483.60.

8-(3-Hydroxy-4-carboxyphenylimino)-1,2;4,4a-di(tetramethylene)-3,4,4a,8-tetrahydropyrido-[1,2-*a*]benzimidazole (Va). Yield 62%, mp 145–146°C. IR spectrum, ν, cm^{-1}: 3500–3300 (OH, COOH), 1700 (C=O), 1630 (C^1=C^2), 1602 (C=N), 1580, 1530 ($C=C_{quin}$). ^{1}H NMR spectrum, δ, ppm: 0.80–2.30 m (19H), 6.25 s (1H, H^9), 6.50–6.80 m (2H, C_6H_3), 7.30 d (1H, H^7, *J* 10.0 Hz), 7.50 d (1H, H^6, *J* 10.0 Hz), 7.76 d (1H, C_6H_3, *J* 8.0 Hz), 8.15 C (1H, OH), 8.88 s (1H, COOH). Found, %: C 72.70; H 6.31; N 9.77. $[M + H]^+$ 430. $C_{26}H_{27}N_3O_3$. Calculated, %: C 72.71; H 6.34; N 9.78. *M* 429.51.

8-(3-Hydroxy-4-carboxyphenylimino)-4,4a-tetramethylene-1,3-diphenyl-3,4,4a,8-tetrahydropyrido[1,2-*a*]benzimidazole (Vb). Yield 60%, mp 187–188°C. IR spectrum, ν, cm^{-1}: 3500–3380 (OH, COOH), 1770 (C=O), 1640 (C^1=C^2), 1620 (C=N), 1576, 1525 ($C=C_{quin}$). ^{1}H NMR spectrum, δ, ppm: 1.40–2.40 m (9H), 3.90 d.d (1H, H^3, *J* 10.0, 3.0 Hz), 4.42 s (1H, H^9, *Z*-form {6}), 4.60 s (1H, H^9, *E*-form {1}), 5.30 d (1H, H^2, *J* 3.0 Hz, *Z*-form {6}), 5.38 d (1H, H^2, *J* 3.0 Hz, *E*-form {1}), 5.62 d (1H, C_6H_3, *J* 8.0 Hz), 5.80 C (1H, C_6H_3), 6.88 d (1H, C_6H_3, *J* 8.0 Hz), 7.15 d (1H, H^7, *J* 10.0 Hz), 7.20–7.65 m (10H, C_6H_5), 7.90 s (1H, OH), 7.95 s (1H, COOH). Found, %: C 77.41; H 5.52; N 7.95. $[M + H]^+$ 528. $C_{34}H_{29}N_3O_3$. Calculated, %: C 77.40; H 5.54; N 7.96. *M* 527.61.

8-{5-(4,5-Dihydroimidazo[1,2-*b*]benzothiazol-5-yl)imino}-1,2;4,4a-di(tetramethylene)-3,4,4a,8-tetrahydropyrido[1,2-*a*]benzimidazole (VIa). Yield 88%, mp 155–157°C. IR spectrum, ν, cm^{-1}: 1708 ($C=N_{imidazole}$), 1668 (C^1=C^2), 1630 (C=N), 1580, 1560 ($C=C_{quin}$). ^{1}H NMR spectrum, δ, ppm: 1.40–2.40 m (19H), 3.81 t (2H, $H_{imidazole}$, *J* 9.1 Hz), 4.34 t (2H, $H_{imidazole}$, *J* 9.1 Hz), 5.66 d (1H, H^9, *J* 1.1 Hz, *Z*-form {10}), 5.97 d (1H, H^9, *J* 1.1 Hz, *E*-form {1}), 6.23 d (1H, $H^4_{benzothiazole}$ *J* 1.7 Hz, *E*-form {1}), 6.33 d (1H, $H^4_{benzothiazole}$ *J* 1.7 Hz, *Z*-form {10}), 6.40 d.d (1H, $H^6_{benzothiazole}$ *J* 8.3, 1.7 Hz, *E*-form {1}), 6.51 d.d (1H, $H^6_{benzothiazole}$ *J* 8.3, 1.7 Hz, *Z*-form {10}), 6.96 d.d (1H, H^7, *J* 9.9, 1.1 Hz), 7.04 d (1H, H^6, *J* 9.9 Hz), 7.18 d (1H, $H^7_{benzothiazole}$ *J* 8.3 Hz). Found, %: C 72.39; H 6.58; N 14.73. $[M + H]^+$ 468. $C_{28}H_{29}N_5S$. Calculated, %: C 71.92; H 6.25; N 14.98. *M* 467.63.

8-{5-(4,5-Dihydroimidazo[1,2-*b*]benzothiazol-5-yl)imino}-4,4a-tetramethylene-1,3-diphenyl-3,4,4a,8-tetrahydropyrido[1,2-*a*]benzimidazole (VIb). Yield 82%, mp 159–161°C. IR spectrum, ν, cm^{-1}: 1712 ($C=N_{imidazole}$), 1648 (C^1=C^2), 1629 (C=N), 1584, 1550 ($C=C_{quin}$). ^{1}H NMR spectrum, δ, ppm: 1.40–2.20 m (9H), 3.79 m (3H, H^3, $H_{imidazole}$), 4.39 t (2H, $H_{imidazole}$, *J* 9.4 Hz), 4.41 d (1H, H^9, *J* 1.4 Hz), 5.31 d (1H, H^2, *J* 2.8 Hz), 5.99 d ((1H, $H^4_{benzothiazole}$, *J* 1.9 Hz), 6.15 d.d (1H, $H^6_{benzothiazole}$, *J* 8.3, 1.9 Hz), 6.95 d.d (1H, $H^7_{benzothiazole}$, *J* 8.3 Hz), 7.07 d (1H, H^6, *J* 9.9 Hz). Found, %: C 76.79; H 5.63; N 12.06. $[M + H]^+$ 566. $C_{36}H_{31}N_5S$. Calculated, %: C 76.43; H 5.52; N 12.38. *M* 565.73.

8-[2-(2-Furylmethyl)cyclohexylimino]-1,2;4,4a-di(tetramethylene)-3,4,4a,8-tetrahydropyrido[1,2-*a*]benzimidazole (VIIa). Yield 63%, mp 176–178°C. IR spectrum, ν, cm^{-1}: 1666 (C^1=C^2), 1625 (C=N), 1590, 1528 ($C=C_{quin}$). ^{1}H NMR spectrum, δ, ppm: 1.20–2.40 m (26H), 2.55 m (4H, H^3, $CH_{2\text{-furyl}}$), 3.67 m (1H, =N–$CH_{cyclohex}$), 5.68 s (1H, H^9), 5.90 d ($1H_{furyl}$, *J* 2.7 Hz), 6.23 d.d ($1H_{furyl}$, *J* 2.7, 1.9 Hz), 6.80 d (1H, H^7, *J* 9.9 Hz), 6.86 d (1H, H^6, *J* 9.9 Hz). Found, %: C 78.87; H 7.98; N 9.46. $[M + H]^+$ 456. $C_{30}H_{37}N_3O$. Calculated, %: C 79.08; H 8.19; N 9.22. *M* 455.63.

8-[2-(2-Furylmethyl)cyclopentylimino]-1,2;4,4a-di(tetramethylene)-1,2-cyclohexene-3,4,4a,8-tetrahydropyrido[1,2-*a*]benzimidazole (VIIIa). Yield 60%, mp 188–190°C. IR spectrum, ν, cm^{-1}: 1668 (C^1=C^2), 1630 (C=N), 1589, 1530 ($C=C_{quin}$). ^{1}H NMR spectrum, δ, ppm: 1.50–2.40 m (25H), 2.60 d.d (2H, $CH_{2\text{-furyl}}$, *J* 8.8, –15.1 Hz), 2.78 d.d (1H, H^3_{eq}, *J* 6.3, –15.1 Hz), 4.00 m (1H, =N–$CH_{cyclopent}$), 5.73 C (1H, H^9), 5.92 d ($1H_{furyl}$, *J* 2.5 Hz), 6.23 d.d ($1H_{furyl}$, *J* 2.7, 1.9 Hz), 6.80 d (1H, H^7, *J* 9.9 Hz), 6.86 d (1H, H^6, *J* 9.9 Hz). Found, %: C 79.21; H 8.09; N 9.76. $[M + H]^+$ 442. $C_{29}H_{35}N_3O$. Calculated, %: C 78.87; H 7.99; N 9.52. *M* 441.61.

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