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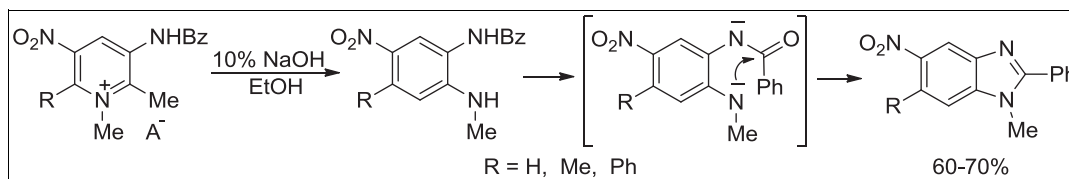
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The synthesis of substituted 5-nitro-2-phenyl-1*H*-benzimidazoles has been described via domino anionic process rearrangement of 3-benzoylamino-1,2-dimethyl-5-nitropyridinium salts in the presence of NaOH water-alcohol solution. Substituted *N*-benzoyl-*o*-phenylenediamines was obtained via recyclization of 3-benzoylamino-1,2-dimethyl-5-nitropyridinium salts in the presence of aqueous methylamine solution.

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INTRODUCTION

Benzimidazole is one of biologically significant heterocycles. Benzimidazole nucleus is included in the structure of vitamin B₁₂. Several benzimidazoles belong to a new class of universal non-natural nucleobases that are capable to complementary binding with any natural nucleobase [1].

The first benzimidazole alkaloids (Kealiquinone, Kealiinine A, B, and C) were isolated from sponge *Leucetta chagosensis* in the last two decades [2].

Benzimidazole derivatives have a broad spectrum of biological activity [3]. Many examples of widely used classes of drugs containing the benzimidazole ring system are known [4].

The classical method of synthesis of substituted benzimidazoles includes a condensation of *o*-phenylenediamines with acids and their derivatives, cyan bromide, and also with aldehydes and ketones in the presence of oxidant (Phillips reaction) [5]. Modern methods of synthesis of benzimidazoles are presented by Pd-catalyzed carbonylation of aryl halides with *o*-phenylenediamines and intramolecular Pd-catalyzed *N*-arylation of *o*-bromophenylamidines [6]. Also, the synthesis of benzimidazoles by cyclization of Passerini reaction products in the presence of trifluoroacetic anhydride is known [7]. Recently, a new method for the synthesis of 2-hetaryl benzimidazoles by acid-catalyzed quinoxaline-benzimidazole rearrangement was proposed [8].

RESULTS AND DISCUSSION

In the present work, we report a one-pot synthesis of substituted benzimidazoles (**6a-c**) from 3-benzoylamino-1,2-dimethyl-5-nitropyridinium salt (**3a-c**) in the presence of NaOH water-alcohol solution. Pyridines (**2a-c**) were synthesized via benzoylation of 3-aminopyridines (**1a-c**) (Schotten-Baumann reaction).

3-Aminopyridines (**1a-c**) were obtained earlier by different sextet rearrangements (Curtius, Hofmann, and Schmidt rearrangements) and described in our previous work [9]. Pyridinium salts (**3a-c**) were synthesized via alkylation of 3-benzoylamino-5-nitropyridines (**2a-c**) by dimethyl sulfate and fluorosulfuric acid methyl ester (Scheme 1).

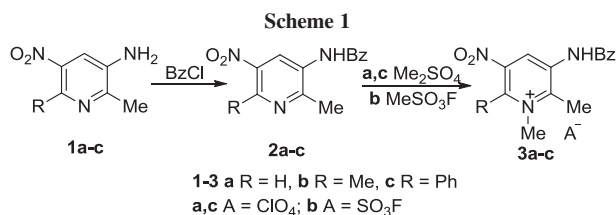
Recyclization of nitropyridinium salts (**3a-c**) in the presence of aqueous methylamine solution at RT leads to *N*-benzoyl-*o*-phenylenediamines (**4a-c**).

Apparently, transformation of pyridinium salts (**3a-c**) into *ortho*-phenylenediamine (**4a-c**) proceeds through the nucleophilic attack by the hydroxide ion at the most electron-deficient carbon atom in the pyridine ring at 6-position to form pseudo-base **A** (a neutral analog of a σ -complex). The following base catalyzed ionic ring opening with C—N bond cleavage leads to open form **B**, which undergoes intramolecular aldol-crotonic condensation involving methyl and carbonyl groups. Recyclization of pyridinium salt (**3b**) leads to isomeric *ortho*-phenylenediamine (**4b**) and *para*-phenylenediamine (**5**) through intermediate pseudo-bases **A** and **C** (Scheme 2).

Benzimidazoles (**6a-c**) are the final compounds of domino anionic reaction of 3-benzoylamino-1,2-dimethyl-5-nitropyridinium salts (**3a-c**) with NaOH water-alcohol solution. Intramolecular heterocyclization of rearrangement products [*N*-benzoyl-*o*-phenylenediamines (**4a-c**)] leads to benzimidazoles (**6a-c**) in the presence of strong base. Imidazole ring of nitrobenzimidazoles (**6a-c**) are formed via interaction of anion of arylmethylamino group of dianion with carbonyl of amide group (Scheme 3).

CONCLUSION

In conclusion, we have demonstrated possibility of the formation of imidazole ring of benzimidazoles from *N*-benzoyl-*o*-phenylenediamines in the presence of base.



EXPERIMENTAL

The IR spectra were obtained on a Simex FT 801 instrument in the solid phase with an attachment for a single broken internal reflection. The ¹H-NMR spectra were recorded on a Bruker Avance DRX-250 in CDCl₃ and DMSO-*d*₆, internal standard was the residual protons of the solvent (CDCl₃ δ 7.25 and DMSO-*d*₆ δ 2.50 ppm). ¹³C-NMR spectra were recorded on a Bruker DRX-250 spectrometer with DMSO-*d*₆ as internal standard (δ C 40.1 ppm). Elemental analysis was carried out on a Perkin-Elmer 2400 series II CHN Analyzer. Column chromatography was performed using Merck 60A silica gel, 0.060–0.200 mm. Monitoring of the course of reactions and purity of the compounds obtained was by TLC on Silufol UV-254 plates. 3-Aminopyridines (**1a-c**) was synthesized according to the procedures described in [9].

General procedure for the synthesis of 3-benzoylamino-5-nitropyridines (2a-c). Benzoyl chloride 1.08 g (7.7 mmol) was added dropwise to a solution of 3-aminopyridine (**1a-c**) [9] (7 mmol) in absolute pyridine (5 mL) at 0°C. The mixture was stirred 10 min at 0°C and then 2 h at RT. After that, absolute ethanol (1.5 mL) was added, and the mixture was stirred 10 min. The reaction mixture was diluted with chilled water, and the precipitate was filtered. Pyridines (**2a-c**) were recrystallized from 95% ethanol.

***N*-(2-Methyl-5-nitropyridin-3-yl)benzamide (2a).** Yield 85%; white crystals, mp 183–184°C; IR: NH 3450, CO 1690, NO₂ 1520, 1350 cm⁻¹; ¹H-NMR (250 MHz, DMSO-*d*₆): δ_H 2.75 (s, 3H, 2-CH₃), 7.51–7.68 (m, 3H, Ph), 8.01–8.10 (m, 2H, Ph), 9.00 (d, ⁴*J* = 2.5 Hz, 1H, 6-H), 9.11 (d, ⁴*J* = 2.5 Hz, 1H, 4-H), 9.45 (s, 1H, NH); *Anal.* Calcd for C₁₃H₁₁N₃O₃: C, 60.70; H, 4.31; N, 16.33. Found: C, 60.64; H, 4.28; N, 15.95.

***N*-(2,6-Dimethyl-5-nitropyridin-3-yl)benzamide (2b).** Yield 77%, white crystals, mp 176–177°C; IR: NH 3470, CO 1700, NO₂

1520, 1340 cm⁻¹; ¹H-NMR (250 MHz, DMSO-*d*₆): δ_H 2.56 (s, 3H, 2-CH₃), 2.75 (s, 3H, 6-CH₃), 7.52–7.65 (m, 3H, Ph), 7.98–8.04 (m, 2H, Ph), 8.53 (s, 1H, 4-H), 10.30 (s, 1H, NH); *Anal.* Calcd for C₁₄H₁₃N₃O₃: C, 61.99; H, 4.83; N, 15.49. Found: C, 61.89; H, 4.81; N, 15.18.

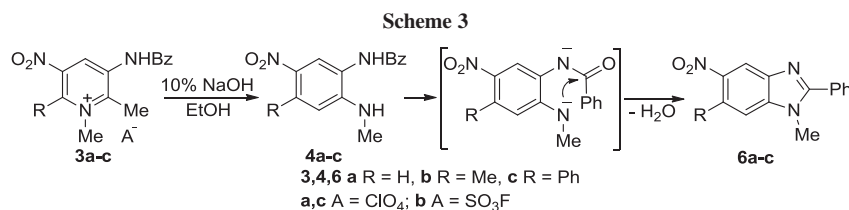
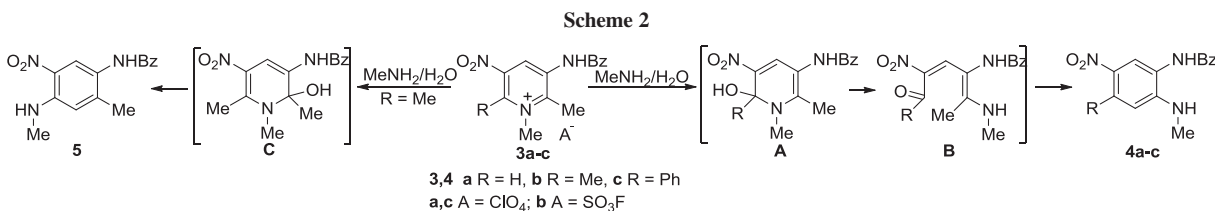
***N*-(2-Methyl-5-nitro-6-phenylpyridin-3-yl)benzamide (2c).** Yield 80%, white crystals, mp 195–196°C; IR: NH 3440, CO 1700, NO₂ 1520, 1320; ¹H-NMR (250 MHz, DMSO-*d*₆): δ_H 2.64 (s, 3H, 2-CH₃), 7.46–7.62 (m, 8H, Ph), 7.99–8.08 (m, 2H, Ph), 8.59 (s, 1H, 4-H), 10.35 (s, 1H, NH); *Anal.* Calcd for C₁₉H₁₅N₃O₃: C, 68.46; H, 4.54; N, 12.61. Found: C, 68.92; H, 4.28; N, 12.28.

General procedure for the synthesis of 3-benzoylamino-5-nitropyridinium salts (3a, c). The mixture of pyridine (**2a, c**) (5 mmol) and Me₂SO₄ (15 mmol) was heated. Then, mixture was chilled and washed with dry ether (3 × 10 mL). The ether was removed by decantation. The residue was dissolved in H₂O (5 mL), and saturated aqueous solution of NaClO₄ (5.3 mol) was added. Pyridinium perchlorates (**3a, c**) were filtered, dried, and recrystallized from ethanol.

3-(Benzoylamino)-1,2-dimethyl-5-nitropyridinium perchlorate (3a). Conditions 4 h and 70°C, yield 71%, colorless crystals, mp 223–224°C. ¹H-NMR (250 MHz, DMSO-*d*₆): δ_H 2.82 (s, 3H, 2-CH₃), 4.97 (s, 3H, N—CH₃), 7.55–7.74 (m, 3H, Ph), 8.01–8.08 (m, 2H, Ph), 9.39 (d, ⁴*J* = 2.2 Hz, 1H, 6-H), 10.05 (d, ⁴*J* = 2.2 Hz, 1H, 4-H), 11.04 (s, 1H, NH); *Anal.* Calcd for C₁₄H₁₄ClN₃O₇: C, 45.23; H, 3.80; N, 17.30. Found: C, 45.39; H, 3.65; N, 11.15.

3-(Benzoylamino)-1,2-dimethyl-5-nitro-6-phenylpyridinium perchlorate (3c). Conditions 12 h and 90°C, yield 77%, colorless crystals, mp 128–129°C. ¹H-NMR (250 MHz, DMSO-*d*₆): δ_H 2.84 (s, 3H, 2-CH₃), 3.92 (s, 3H, N—CH₃), 7.56–7.77 (m, 8H, Ph), 8.01–8.11 (m, 2H, Ph), 9.33 (s, 1H, 4-H), 11.10 (s, 1H, NH); *Anal.* Calcd for C₂₀H₁₈ClN₃O₇: C, 53.64; H, 4.05; N, 9.38. Found: C, 53.89; H, 3.89; N, 9.64.

3-(Benzoylamino)-1,2,6-trimethyl-5-nitropyridinium fluoridosulfate (3b). The solution of MeSO₃F (15 mol) in chlorobenzene (3 mL) was added dropwise to a solution of pyridine (**2b**) (5 mmol) in chlorobenzene (15 mL) at 0°C with stirring. The mixture was stirred 30 min at 0°C and 5 days at RT. Then, the mixture was diluted with diethyl ether. The precipitate was filtered and recrystallized from ethanol. The colorless crystals were obtained in 94% yield, mp 212–213°C. ¹H-NMR (250 MHz, DMSO-*d*₆): δ_H 2.82 (s, 3H, 2-CH₃), 2.92 (s, 3H, 6-CH₃), 4.29 (s, 3H,



N—CH₃), 7.52–7.66 (m, 3H, Ph), 8.06–8.11 (m, 2H, Ph), 9.09 (s, 1H, 4-H), 11.04 (s, 1H, NH). *Anal.* Calcd for C₁₅H₁₆FN₃O₆S: C, 46.75; H, 4.18; N, 10.90. Found: C, 46.40; H, 4.39; N, 10.62.

General procedure for the reaction of pyridinium salts (3a–c) with aqueous solution MeNH₂. The 41% aqueous solution of MeNH₂ (40 mL) was added to solution of salt (3a–c) (2 mmol) in DMF (4 mL). The mixture was stirred 2 h at RT. The reaction mixture was diluted with water twice, and the precipitate was filtered. The precipitate was purified by column chromatography (eluent chloroform–ethyl acetate, 9:1), recrystallized from ethanol.

***N*-[2-(Methylamino)-5-nitrophenyl]benzamide (4a).** Yield 71%, light yellow crystals, mp 204–205°C (lit. mp 195°C [10]); IR: NH 3380, CO 1650, NO₂ 1550, 1310 cm^{−1}. ¹H-NMR (250 MHz, DMSO-*d*₆): δ_H 2.84 (d, *J*=4.6 Hz, 3H, NH—CH₃), 6.71 (d, ³*J*=8.8 Hz, 1H, 3-H), 6.86 (br. s, 1H, NH—CH₃), 7.50–7.59 (m, 3H, Ph, 4-H), 7.98–8.14 (m, 4H, Ph, 6-H), 9.82 (s, 1H, NH). *Anal.* Calcd for C₁₄H₁₃N₃O₃: C, 61.99; H, 4.83; N, 15.49. Found: C, 62.05; H, 4.71; N, 15.35.

***N*-[5-(Methylamino)-2-nitrobiphenyl-4-yl]benzamide (4c).** Yield 80%, light yellow crystals, mp 200–201°C; IR: NH 3410, CO 1655, NO₂ 1520, 1340 cm^{−1}. ¹H-NMR (250 MHz, CDCl₃): δ_H 2.90 (br. s, 3H, NH—CH₃), 4.95 (br. s, 1H, NH—CH₃), 6.52 (s, 1H, 6-H), 7.25–7.31 (m, 2H, Ph), 7.35–7.43 (m, 3H, Ph), 7.45–7.61 (m, 3H, Ph), 7.81 (s, 1H, NH), 7.87–7.95 (m, 2H, Ph), 8.00 (s, 1H, 3-H). *Anal.* Calcd for C₂₀H₁₇N₃O₃: C, 69.15; H, 4.93; N, 12.10. Found: C, 69.19; H, 5.06; N, 11.94.

***N*-[4-Methyl-2-(methylamino)-5-nitrophenyl]benzamide (4b) and *N*-[2-methyl-4-(methylamino)-5-nitrophenyl]benzamide (5).** Were separated by column chromatography (eluent chloroform–ethyl acetate, 9:1), recrystallized from ethanol. For **4b**: yield 40%, light yellow crystals, mp 211–212°C. IR: NH 3390, CO 1630, NO₂ 1540, 1320 cm^{−1}. ¹H-NMR (250 MHz, DMSO-*d*₆): δ_H 2.61 (s, 3H, 4-CH₃), 2.84 (d, *J*=4.9 Hz, 3H, NH—CH₃), 6.56 (s, 1H, 3-H), 6.61 (q, *J*=4.9 Hz, 1H, NH—CH₃), 7.50–7.62 (m, 3H, Ph), 8.00 (s, 1H, 6-H), 8.01–8.04 (m, 2H, Ph), 9.71 (s, 1H, NH). ¹³C-NMR (100 MHz, DMSO-*d*₆): δ_C 22.01, 29.51, 111.80, 120.52, 125.24, 127.98, 128.18, 131.59, 134.15, 134.97, 135.85, 149.64, 166.17. *Anal.* Calcd for C₁₅H₁₅N₃O₃: C, 63.15; H, 5.30; N, 14.73. Found: C, 63.08; H, 5.29; N, 14.72. For **5**: yield 24%, orange crystals, mp 220–221°C. IR: NH 3277, CO 1651, NO₂ 1543, 1321 cm^{−1}. ¹H-NMR (250 MHz, DMSO-*d*₆): δ_H 2.29 (s, 3H, 2-CH₃), 2.99 (d, *J*=5.0 Hz, 3H, NH—CH₃), 6.92 (s, 1H, 3-H), 7.50–7.63 (m, 3H, Ph), 7.94–8.00 (m, 2H, Ph), 8.05 (s, 1H, 6-H), 8.18 (q, *J*=5.0 Hz, 1H, NH—CH₃), 9.87 (s, 1H, NH). ¹³C-NMR (100 MHz, DMSO-*d*₆): δ_C 18.61, 29.77, 114.75, 123.31, 124.80, 127.61, 128.40, 128.42, 131.62, 134.22, 144.40, 145.33, 165.91. *Anal.* Calcd for C₁₅H₁₅N₃O₃: C, 63.15; H, 5.30; N, 14.73. Found: C, 63.11; H, 5.31; N, 14.80.

General procedure for the reaction of pyridinium salts (3a–c) with aqueous solution of NaOH. The 10% aqueous solution of NaOH (2 mL) was added to suspension of salt (3a–c) (1 mmol) in ethanol (4 mL). The reaction mixture was stirred 24 h at RT, diluted with water twice, and neutralized with 50% acetic acid. The precipitate was filtered and purified by column chromatography (eluent chloroform–ethyl acetate, 9:1), recrystallized from ethanol.

***1*-Methyl-5-nitro-2-phenyl-1*H*-benzimidazole (6a).** Yield 65%, white crystals, mp 181–182°C (lit. mp 189°C [10]); IR: NO₂ 1520, 1350 cm^{−1}. ¹H-NMR (250 MHz, CDCl₃): δ_H 3.91 (s, 3H, N—CH₃), 7.45 (d, ³*J*=8.7 Hz, 1H, 7-H), 7.53–7.77 (m, 5H, Ph),

8.23 (dd, ³*J*=8.7 Hz, ⁴*J*=0.7 Hz, 1H, 6-H), 8.69 (d, ⁴*J*=0.7 Hz, 1H, 4-H). *Anal.* Calcd for C₁₄H₁₁N₃O₂: C, 66.40; H, 4.38; N, 16.59. Found: C, 66.31; H, 4.43; N, 16.64.

***1,6*-Dimethyl-5-nitro-2-phenyl-1*H*-benzimidazole (6b).** Yield 67%, white crystals, mp 184–185°C. IR: NO₂ 1513, 1310 cm^{−1}. ¹H-NMR (250 MHz, CDCl₃): δ_H 2.64 (s, 3H, 6-CH₃), 2.81 (s, 3H, N—CH₃), 7.47–7.63 (m, 3H, Ph, 7-H), 7.82–7.91 (m, 3H, Ph), 9.02 (s, 1H, 4-H). *Anal.* Calcd for C₁₅H₁₃N₃O₂: C, 67.40; H, 4.90; N, 15.72. Found: C, 67.52; H, 5.00; N, 15.61.

***1*-Methyl-5-nitro-2,6-diphenyl-1*H*-benzimidazole (6c).** Yield 60%, white crystals, mp 201–202°C. IR: NO₂ 1540, 1355 cm^{−1}. ¹H-NMR (250 MHz, DMSO-*d*₆): δ_H 3.93 (s, 3H, N—CH₃), 7.34–7.63 (m, 10H, Ph), 7.78 (s, 1H, 7-H), 8.33 (s, 1H, 4-H). *Anal.* Calcd for C₂₀H₁₅N₃O₂: C, 72.94; H, 4.59; N, 12.76. Found: C, 73.07; H, 4.33; N, 12.79.

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