Selective Reduction of 2,4-Dinitro- and 4,5-Dinitroimidazole Derivatives Using Iron Dust

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A series of N-substituted 2,4-dinitroimidazoles, 4,5-dinitroimidazoles, and 2-methyl-4,5-dinitroimidazoles have been selectively reduced to the corresponding aminonitroimidazole derivatives, using iron dust in glacial acetic acid at room temperature. 2,4-Dinitroimidazoles have been reduced to the 2-amino-4-nitro-derivatives only but 4,5-dinitroimidazoles have given 4-amino-5-nitro- or 5-amino-4-nitro-derivatives depended on the structure of the N-substituent.

\[
\begin{align*}
\text{O}_2\text{N} & \quad \text{NO}_2 \\
\text{N} & \quad \text{CH}_2\text{R} \\
\text{Fe/CH}_3\text{COOH} & \quad \text{O}_2\text{N} & \quad \text{NH}_2 \\
\text{N} & \quad \text{CH}_2\text{R} \\
\text{Fe/CH}_3\text{COOH} & \quad \text{O}_2\text{N} & \quad \text{H}_2\text{N} & \quad \text{O}_2\text{N} \\
\text{N} & \quad \text{CH}_2\text{R} & \quad \text{H}_2\text{N} & \quad \text{O}_2\text{N} \\
\text{N} & \quad \text{CH}_2\text{R} & \quad \text{H}_2\text{N} & \quad \text{O}_2\text{N} \\
\end{align*}
\]

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INTRODUCTION

Imidazoles and their derivatives are very important group of compounds for their pharmacological properties [1,2]. Particularly, the nitroimidazole class of medicines mainly shows activity against bacteria [3,4]. Also, these drugs have become the important agents for treatment of serious infections caused by protozoa. Some of them have been tested against HIV [5]. 2-Nitroimidazoles played a major role as bioreductive markers for tumour hypoxia [6] and as radiosensitizers [7,8]. Our earlier investigations have been devoted to synthesis and antifungal as well as antibacterial properties of N-phenacyl-4,5-dinitroimidazole and 4-substituted amino-5-nitroimidazole derivatives, which have been prepared by nucleophilic displacement of the nitro group in 4,5-dinitroimidazole derivatives by primary or secondary amines [9,10].

Reduction of the nitro compounds has been one of the most important reactions in organic chemistry used as a routine method for the preparation of various nitrogen derivatives such as amines, nitrosocompounds, or hydroxylamines. The reported reduction methods of aromatic nitro compounds to prepare amino derivatives are very numerous. The reduction of the nitroazoles is readily achieved by using of one of many possible reagents like for instance iron in an acidic medium [11,12], hydrogen in the presence of palladium [13,14], Raney nickel [15] sodium borohydride [16]. Only limited number of all reduction methods have described the selective reduction of one nitro group in dinitro-compounds with remain unchanged of other functional groups. For example, reduction of 2,4-dinitrophenol using sodium sulfide has led to 2-amino-4-nitrophenol [17], but 4-amino-2-nitro-carboxamide mustard have been obtained by selective 4-nitro group reduction of 2,4-dinitrobenzamide derivative with SnCl₂ in concentrated HCl [18]. Lin and Sun [17] have found that using either Zn in HCOONH₄ or tin (II) chloride dihydrate can deliver traceless synthesis of 2-quinoxalinone analogues, an o-nitroaniline intermediate without further reduction of another nitro group under microwave irradiation or by conventional heating.

Products of reductions of nitroimidazole derivatives exhibit potential biological significance and are intermediates in syntheses of a variety of biologically active imidazoles. The compounds containing amino group in azoles ring can show good antimicrobial activity. In particular, the introduction of a bromine or two chlorine atoms or one phenyl group to the phenyl ring, except for the amino group in 2-amino-4(5)-arylimidazoles
leads to compounds provided some antimicrobial activity [19]. Moreover, compound with the amino and the nitro groups have played important role as potent inhibitor of Coxackie virus B3 replication [20]. Synthetic 2-aminoimidazole derivatives including 2-aminohistamine have shown to have H1 and H2 receptor agonist and antagonist activity. Other 2-aminoimidazole derivatives are selective 5-HT3 receptor antagonists, which may be potentially useful in the treatment of chemotherapy induced emesis [21]. Imidazole alkaloids containing the amino group at C-2 position in the heterocyclic ring also show interesting biological property such as anthelminthic activity (dorimidazole A, preclathridine A) [1].

RESULTS AND DISCUSSION

The reduction of N-substituted 2,4-dinitroimidazole, 4,5-dinitroimidazole, and 2-methyl-4,5-dinitroimidazole to the corresponding aminonitroderivatives by use of iron dust in glacial acetic acid at room temperature exhibits high selectivity. It is very surprising that iron dust in an acidic medium is capable of reducing one nitro group without further reduction of second nitro group. It is known that iron in the presence of acid is not selective agent [11]. In our experiments, treatment of dinitroimidazole derivatives with iron dust afforded, after purification, the crystalline aminonitro-compounds. Stability of these compounds depended on the position of the amino group, decreased in a series of 2-amino, 5-amino and 4-amino compounds.

The starting 4,5-dinitroimidazole (1), 2-methyl-4,5-dinitroimidazole (2), and 2,4-dinitroimidazole (3) were prepared according to the methods described in the literature [22,23]. The N-substituted derivatives of 4,5-dinitroimidazoles (4–17) were obtained in the reaction of 1 or 2 with (CH3)2SO4, epoxypropane, epichlorohydrin, or phenacyl bromides in accordance with the method described in the literature [9,23]. The N-(2-hydroxypropyl) and N-(3-chloro-2-hydroxypropyl) compounds (6–8) were oxidized by Jones reagent to the desired carbonyl derivatives (10–12) [24]. Additionally, 2-methyl-4,5-dinitroimidazole was alkylated with epibromohydrin according to the method described for prepared epichlorohydrin derivatives [9]. The treatment of 2 with an excess of epibromohydrin (1:2) under reflux without solvent for about 3 h led to new 1-(3-bromo-2-hydroxypropyl)-2-methyl-4,5-dinitroimidazole (9). This new derivative was oxidized by Jones reagent to the 1-(3-bromo-2-oxopropyl)-2-methyl-4,5-dinitroimidazole (13) in accordance with the method described earlier [24]. Synthesis of N-substituted 4,5-dinitroimidazole derivatives 4–17 is shown in the Scheme 1.

Also, the 2,4-dinitroimidazole (3) was put on the reactions with (CH3)2SO4 and phenacyl bromides according to the methods described in the literature [9,23,25]. The reactions of 3 with appropriate reagents resulted in the formation of N-substituted derivatives of 2,4-dinitroimidazole (18–20), as shown in the Scheme 2.

The N-methyl (18) and N-phenacyl derivatives of 2,4-dinitroimidazole (19,20) in the reduction gave only respective 2-amino-4-nitroimidazole with yield 54–82% (Scheme 3). When the reaction was complete, the excess of iron and its oxidation products were filtered off and the reaction mixture was diluted with water. The 2-amino-1-methyl-4-nitroimidazole (21) was isolated by extraction. After removal of the solvent, the crude product was crystallized. The precipitated crude reduction products (22, 23) were filtered off. A large, lipophilic phenacyl group facilitated obtaining the aminonitroderivatives. Reduction
N-substituted 2,4-dinitroimidazole derivatives 18–20 is shown in the Scheme 3.

The structures of 21–23 were confirmed by full spectral data. The infrared spectra showed absorptions at about 3400 and 3265 cm \(^{-1}\) and also 1560 and 1300 cm \(^{-1}\) indicative for the N—H and NO\(_2\) resonances, respectively. The mass spectra exhibit strong molecular ions at 142, 246, and 280, respectively. In addition to the molecular ions, in spectra of compounds 22 and 23 strong signal (rel. int. 100%) corresponding to ion from phenacyl (m/z 105) or p-chlorophenacyl group (m/z 139) was observed. In the \(^1\)H NMR spectra, the signals of the amino groups are as singlets at about 6.30 ppm, CH\(_2\) protons of the phenacyl groups resonated as singlet at 5.56 and 5.54 ppm. The aromatic protons at C-5 position of the imidazole ring were observed at about 7.84 ppm.

Reduction of 4,5-dinitroimidazole alkyl derivatives led to N-alkylaminonitroimidazoles, as well. The iron dust in glacial acetic acid reduced with facility one nitro group but the second remained unreactive. In the same conditions, mixture of two isomers: 4-amino-5-nitro- and 5-amino-4-nitro- with predominance of the latter mentioned were formed (Scheme 4). The 4,5-diaminomidazoles were not observed in the reaction mixtures. Formation of the aminonitroimidazoles depended on the position of the new formed amino group and yielding of 4-amino-compounds was the poorest. The low efficiency in reduction reactions of 4,5-dinitroimidazole alkyl derivatives probably is connected with structures and stability of compounds obtained. Moreover, the isolation of pure, definite, aminonitroderivatives was very inconvenient. Some of them were obtained after complex extraction, then purification by column chromatography and additional crystallization.

Reduction of N-substituted 4,5-dinitroimidazole derivatives 4, 5, 7–17 is shown in the Scheme 4.

Reaction of the compounds containing N-methyl group (4, 5) with iron dust afforded 4-amino-5-nitroimidazoles (24, 25) only that were separated after extraction with chloroform. In the reduction of the N-halohydroxypropyl derivatives (7–9) were formed 5-amino-4-nitro-compounds (26–28). These substances were obtained as solid products. Other 4,5-dinitroimidazoles (10–17) containing the carbonyl group in the chain.
at \(N-1\) position of the imidazole ring provided mainly 4-amino-5-nitro- derivatives, after extraction (29–34, 36, 37). Only in the reduction of 2-methyl-4,5-dinitro-1-phenacylimidazole (15) and (17), the mixtures of two products were obtained. After reduction of 15, isomers: 4-amino-2-methyl-5-nitro-1-phenacylimidazole (34) and 5-amino-2-methyl-4-nitro-1-phenacylimidazole (35) were obtained. Compound 35 was separated by the filtration and purified by crystallization. Dominating product, 34, was obtained after extraction and was purified by column chromatography. Similarly, the 4-amino-1-(p-chlorophenacyl)-2-methyl-5-nitroimidazole (37) and 5-amino-1-(p-chlorophenacyl)-2-methyl-4-nitroimidazole (38) were obtained as products of the reduction of 1-(p-chlorophenacyl)-2-methyl-4,5-dinitroimidazole (17). These derivatives were separated by column chromatography. In all cases, 4,5-diamino derivatives were not observed.

The infrared spectra of 24–38 showed absorptions at about 3400 and 3260 cm\(^{-1}\) and also 1560 and 1300 cm\(^{-1}\) indicative for the \(\text{N}^{\equiv}\text{H}\) and \(\text{NO}_2\) resonances, respectively. The mass spectra confirmed that only one nitro group in the dinitroimidazoles was reduced. The \(^1\text{H}\) NMR spectra of new products provided evidence for the presence of the amino group. The \(\text{NH}_2\) protons were observed as a singlet at about 7.40 ppm (24, 25) or within the range of 7.57–7.71 ppm (26–38). In the \(^{13}\text{C}\) NMR spectra of 26–28, signal for the carbon jointed with hydroxyl group was near 67 ppm. Moreover, the signal corresponded to the C=O group of 29–38 resonated in the range of 190.88–201.22 ppm. The position of the amino group in 26–38 was connected with the place of a signal of a methylene group at \(N-1\) position of the heterocyclic ring. In the \(^{13}\text{C}\) NMR of 26–28, the signal in the range 46.29–47.09 ppm was assigned to the \(\text{CH}_2\) group. It is also observed that in the \(^{13}\text{C}\) NMR of 29–32, the resonances due to \(\text{CH}_2\) occurred in the range 52.21–54.40 ppm. It is very interesting that the signals of the methylene groups of the pairs 34, 35 and 37, 38 occurred at different values: 52.01 and 50.27 ppm or 51.96 and 49.65 ppm, respectively. In the spectra of 5-amino-4-nitro-products, the carbons of the signals in the imidazole ring appeared in lower values, too. Additionally, the X-ray structures determination of the pair of isomers (34,35) facilitated the interpretation of NMR data and to determine the position of the amino group. The geometry of the molecules confirmed that the compound 35 is 5-amino-4-nitro- derivative but the second is 4-amino-5-nitro-isomer (34). The results obtained were in agreement with our interpretation of NMR spectra. The signals of the methylene group and carbon atoms of the imidazole ring of the all 5-amino-4-nitro compounds were shifted toward the lower field.

In conclusion, we have demonstrated very selective nitro group reduction method in the dinitroimidazole derivatives using iron dust in the acetic acid solution at room temperature. This method provided the products with the amino and the nitro group simultaneously. The presence of an electron-donating amino group at a position neighbouring to the nitro group can have influence on the many biological properties, which depended on the kind of substituents at positions \(N-1\) and \(C-2\) in the heterocyclic ring, as well.

To recapitulate, the \(N\)-derivatives of 2,4-dinitroimidazoles were reduced only to the 2-amino-4-nitro compounds. It was found that the reduction process among the \(N\)-derivatives of 4,5-dinitroimidazoles is more complicated, but it concerns mainly the 4-NO2 group. Nitro group at \(C-5\) position of imidazole ring is susceptible to reduction when there is a hydroxy group in the \(N-1\) alkyl chain connected with tetrahedral carbon atom. Probably, some specific trigonal arrangements have the influence on the direction of nitro group reduction process.

**EXPERIMENTAL**

Melting points were determined on a Boetius apparatus and were uncorrected. The \(^1\text{H}\) and \(^{13}\text{C}\) NMR spectra were recorded on Varian Gemini 300 VT spectrometer (300 and 75 MHz respectively). Chemical shifts (\(\delta\)) are expressed in ppm, relative to tetramethylsilane (TMS) as an internal standard, using DMSO-\(d_6\) as solvents. Coupling constants (\(J\) values) are expressed in Hertz (Hz). Splitting patterns are designated as follows: s, singlet; d, doublet; t, triplet; m, multiplet. MS spectra were recorded on a 402 AMD INTECTRA apparatus by the electron impact technique, operating at 75 eV. The infrared (IR) spectra were recorded in KBr tablets using a Specord 75–IR spectrophotometer and were expressed in cm\(^{-1}\) scale. Elemental analysis was performed on a Vario EL III model of elemental analyzer and data of C, H, and N were within \(\pm 0.4\%\) of calculated values. The progress of reactions and purity of products were controlled with thin-layer chromatography method (TLC) on silica gel plates (60 F254 from Merck) in a CHCl3/MeOH (9:1, v/v) as a developing system. The spots on the plates were observed in the UV light (\(\lambda = 254\text{nm}\)). Solid products of amino-nitro-derivatives were purified in the crystallization process using acetonitrile. Crude, oily products were purified by column chromatography on silica gel using the mixture of chloroform and methanol (50:0 → 50:5) as eluent. Among substances, which were used as substrates, the epichlorohydrin, epoxypropane, epibromohydrin, phenacyl bromides, and iron dust were commercial products. Compounds 4–8, 10–12, and 14–18 were obtained according to the literature method [9,23–25].

1-(3-Bromo-2-hydroxypropyl)-2-methyl-4,5-dinitroimidazole (9). The 2-methyl-4,5-dinitroimidazole (3.44 g, 20 mmol) was added to epibromohydrin (3.42 mL, 40 mmol). The mixture was heated under reflux for about 3 h. Then, cooled and poured into the water. The precipitate was filtered off, washed with water, air-dried and crystallized from 40% EtOH as yellow needles; 5.40 g (87.5%); mp 103–105\(^\circ\)C; \(R_f = 0.64; \text{IR: 3380, 1520, 1340; }^{1}\text{H NMR (300 MHz, DMSO-}\delta_6\text{): }\delta = 5.92\text{ (m, 1H, OH), 4.49 (m, 1H, CH), 4.21 and 4.00 (2 m, 2H,}$$
CH₂Br), 3.59 (m, 2H, N-CH₂), 2.49 (s, 3H, CH₃); ¹³C NMR (75 MHz, DMSO-d₆): δ = 145.42, 139.29, 130.68, 67.97, 50.09, 35.81, 13.72; ms: m/z 308 (2) and 310 (2) (M⁺), 182 (100.0); Anal. calc. for C₁₀H₈N₂O₂Br: C, 33.62, 134.06, 128.92, 128.11, 119.67, 51.83; ms: m/z 246 (14) (M⁺), 156 (100); Rf ¼ 0.43; IR: 3375, 3265, 1675, 1300, 1290, 1190, 1010; ¹H NMR (300 MHz, DMSO-d₆-d₂): δ = 193.95, 146.10, 140.06, 129.42, 53.28, 33.62, 13.31; ms: m/z 308 (15) and 306 (16) (M⁺), 156 (100); Anal. calc. for C₁₀H₈N₂O₂: C, 33.82; H, 4.25; N, 39.44; found: C, 33.86; H, 4.20; N, 39.42.

2-Amino-1-methyl-4-nitroimidazole (21). This compound was obtained as yellow needles (MeCN), 0.40 g (81.3%); mp 243–245°C; Rₚ = 0.41; IR: 3400, 3270, 1680, 1627, 1560; ¹H NMR (300 MHz, DMSO-d₆): δ = 8.05 (m, 2H, 2, 6-Ph), 7.82 (s, 1H, 5-Im), 7.70 (m, 2H, 3, 5-Ph), 6.33 (s, 2H, NH₂), 5.56 (s, 2H, CH₂); ¹³C NMR (75 MHz, DMSO-d₆): δ = 191.33, 149.48, 143.64, 138.87, 133.03, 130.00, 129.05, 119.54, 51.83; ms: m/z 246 (14) (M⁺), 105 (100); Anal. calc. for C₁₁H₇N₄O₃: C, 53.68; H, 4.09; N, 32.76; found: C, 53.73; H, 4.11; N, 22.81.

2-Amino-1-(p-chlorophenacyl)-4-nitroimidazole (23). This compound was obtained as yellow needles (MeCN), 0.42 g (75.0%); mp 260–262°C; Rₚ = 0.43; IR: 3375, 3265, 1675, 1634, 1565, 1285; ¹H NMR (300 MHz, DMSO-d₆): δ = 8.19 (s, 1H, 5-Im), 8.09 (m, 2H, 2, 6-Ph), 7.76 (m, 1H, 4-Ph), 7.66 (m, 2H, 3, 5-Ph), 6.27 (s, 2H, CH₂); ¹³C NMR (75 MHz, DMSO-d₆): δ = 190.84, 144.30, 141.69, 134.87, 133.34, 129.27, 128.27, 126.86, 57.24; ms: m/z 276 (28) (M⁺), 105 (100); Anal. calc. for C₁₁H₇N₂O₂Cl: C, 47.86; H, 2.92; N, 20.29; found: C, 47.90; H, 2.94; N, 20.32.

Aminonitroimidazoles (21–28). Appropriate N-substituted 2,4-dinitro-, 4,5-dinitro-, or 2-methyl-4,5-dinitroimidazole derivatives (2 mmol) were dissolved in glacial AcOH (25 mL), and the excess of iron dust (0.37 g, 6.60 mmol) was added. The resulting mixtures were then left for about 3 days at room temperature shaking them from time to time. Upon completion reaction, the excess of iron and its oxidation products were filtered off, and the reaction mixtures were diluted with water (75 mL). The precipitated crude products were filtered off. If products not solidified, the filtrate was extracted with CHCl₃ (4 × 5 mL) and the combined organic phases were dried (MgSO₄), filtered off. After removal of the solvents, the crude product was purified by column chromatography on silica gel. All new products were crystallized from MeCN.
CH3), 3.78 (m, 2H, CH2Cl), 3.75 (m, 2H, CH2), 2.23 (s, 3H, CH3); 13C NMR (75 MHz, DMSO–d6): δ = 7.65 (1H, CH), 7.63 (2H, NH2), 4.68 (s, 2H, CH2Br); 2.24 (s, 3H, CH3); 13C NMR (75 MHz, DMSO–d6): δ = 195.73, 153.44, 153.12, 120.52, 21.49, 13.62; ms: m/z 234 (18), 232 (50 (M+)), 67 (100); Anal. calc. for C10H9N5O3Cl: C, 36.23; H, 3.90; N, 24.14; found: C, 36.20; H, 3.89; N, 24.15.

4-Amino-3-(chloro-2-oxopropyl)-2-methyl-5-nitroimidazole (32). This compound was obtained as yellow crystals (McCN), 0.21 g (43.5%); mp 186–189°C; Rf = 0.29; IR: 3395, 3260, 1725, 1625, 1540, 1360; 1H NMR (300 MHz, DMSO–d6): δ = 7.65 (s, 2H, NH2), 5.23 (s, 2H, N–CH3), 4.68 (s, 2H, CH2Br), 2.24 (s, 3H, CH3); 13C NMR (75 MHz, DMSO–d6): δ = 195.72, 153.44, 153.11, 120.52, 21.49, 13.61; ms: m/z 278 (7), 276 (7) (M+), 67 (100); Anal. calc. for C10H9N5O3Br: C, 30.46; H, 3.28; N, 20.30; found: C, 30.42; H, 3.26; N, 20.27.

4-Amino-5-nitro-1-phenacylimidazolide (33). This compound was obtained as dark yellow pales (McCN), 0.10 g (20.4%); mp 219–221°C; Rf = 0.30; IR: 3400, 3250, 1670, 1625, 1510; 1H NMR (300 MHz, DMSO–d6): δ = 8.01 (m, 2H, 2,6-Ph), 7.65 (m, 3H, 3,4,5-Ph), 7.69 (s, 2H, NH2), 7.23 (s, 1H, 2-Im), 5.64 (s, 2H, CH2), 13C NMR (75 MHz, DMSO–d6): δ = 191.49, 153.65, 144.06, 134.18, 133.75, 129.01, 128.32, 120.42, 50.55; ms: m/z 246 (6 (M+)), 105 (100); Anal. calc. for C12H11N5O3: C, 53.69; H, 4.09; N, 22.77; found: C, 53.72; H, 4.10; N, 22.74.

4-Amino-2-methyl-5-nitro-1-phenacylimidazolide (34). This compound was obtained as light yellow pales (McCN), 0.16 g (30.1%); mp 227–229°C; Rf = 0.45; IR: 3400, 3250, 1680, 1620, 1500, 1340; 1H NMR (300 MHz, DMSO–d6): δ = 8.06 (m, 2H, 2,6-Ph), 7.68 (m, 5H, 3,4,5-Ph, NH2), 5.84 (s, 2H, CH2), 2.28 (s, 3H, CH3); 13C NMR (75 MHz, DMSO–d6): δ = 192.52, 153.66, 153.28, 134.06, 133.94, 128.80, 128.07, 120.42, 52.01, 13.70; ms: m/z 260 (27 (M+)), 105 (100); Anal. calc. for C12H12N5O3C: C, 55.41; H, 4.65; N, 21.54; found: C, 55.42; H, 4.62; N, 21.54.

5-Amino-2-methyl-4-nitro-1-phenacylimidazolide (35). This compound was obtained as yellow needles (McCN), 0.03 g (5.8%); mp 265–267°C; Rf = 0.28; IR: 3495, 3265, 1665, 1625, 1555, 1355; 1H NMR (300 MHz, DMSO–d6): δ = 8.06 (m, 2H, 2,6-Ph), 7.69 (m, 5H, 3,4,5-Ph, NH2), 5.63 (s, 2H, CH2), 2.10 (s, 3H, CH3); 13C NMR (75 MHz, DMSO–d6): δ = 192.80, 146.08, 142.11, 135.40, 134.40, 129.83, 129.06, 121.77, 50.27, 13.44; ms: m/z 260 (53 (M+)), 105 (100); Anal. calc. for C12H12N5O3C: C, 55.41; H, 4.65; N, 21.54; found: C, 55.40; H, 4.67; N, 21.50.

REFERENCES AND NOTES

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