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Nitration of 1,3-Dihydro-2*H*-imidazo[4,5-*b*]pyridin-2-one Derivatives

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Abstract—Nitration of 1,3-dihydro-2*H*-imidazo[4,5-*b*]pyridin-2-one and its *N*-methyl derivatives at 0–5°C and 60°C gives 5-nitro- and 5,6-dinitro-1,3-dihydro-2*H*-imidazo[4,5-*b*]pyridin-2-ones, respectively. The latter can also be obtained by nitration of 5-mononitro derivatives under similar conditions. The nitration of 6-chloro- and 6-bromo-1,3-dihydro-2*H*-imidazo[4,5-*b*]pyridin-2-ones and their *N*-methyl-substituted analogs leads to the formation of the corresponding 6-chloro(bromo)-5-nitro compounds. The same products are formed in the nitration of 5,6-dichloro- and 5,6-dibromo-1,3-dihydro-2*H*-imidazo[4,5-*b*]pyridin-2-ones. In this case, the process involves replacement of the halogen atom in position *5* of the pyridine fragment by nitro group. The nitration of 6-bromo-5-methyl-1,3-dihydro-2*H*-imidazo[4,5-*b*]pyridin-2-one is accompanied by oxidation of the 5-methyl group to carboxy.

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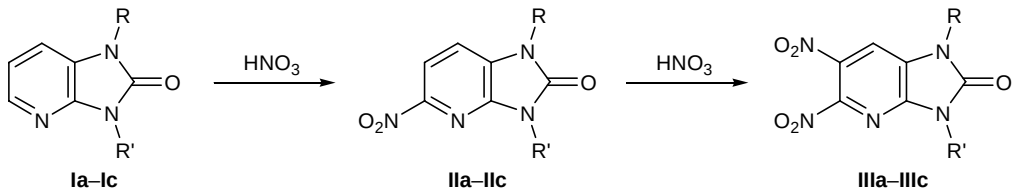
Introduction of a nitro group into the pyridine fragment of imidazo[4,5-*b*]pyridine derivatives and its subsequent transformation into amino or hydrazino group (as in the imidazo[4,5-*c*]pyridine series) open a way to various tricyclic systems which attract interest as potential biologically active compounds [1]. Unsubstituted imidazo[4,5-*b*]pyridine, as well as its 1-methyl derivative do not undergo nitration even on prolonged heating at 200°C with excess potassium nitrate in concentrated sulfuric acid or 5% oleum [2]. Jaine et al. [3] were the first to report on the nitration of imidazo[4,5-*b*]pyridine 4-oxide at the pyridine ring. In this case, the *N*-oxide group exerts *para*-orienting effect, and the nitration occurs at position *7* of the imidazo[4,5-*b*]pyridine system. Unlike 1-methylimidazo[4,5-*b*]pyridine, the nitration of its 3-methyl-substituted analog at 140–160°C afforded 3-methyl-6-nitroimidazo[4,5-*b*]pyridine in 60% yield [4].

In order to facilitate introduction of a nitro group into pyridine ring, 2-methyl- and 2-methoxy-3-methylimidazo[4,5-*b*]pyridines were synthesized. However, contrary to the expectations, nitration of the pyridine ring in these compounds was even more difficult [5]. The nitration of 3-methylimidazo[4,5-*b*]pyridine in concentrated sulfuric acid at room temperature gave the corresponding *N*-nitroamino derivative, and attempts to effect its rearrangement into C-nitro compound (by analogy with 2- and 4-nitroaminopyridines) were unsuccessful. On the other hand, 2-dimethylaminoimidazo[4,5-*b*]pyridine gave rise to 2-dimethylamino-6-nitroimidazo[4,5-*b*]pyridine under mild conditions [6].

Scheme 1.



Ia–Ic **IIa–IIc** **IIIa–IIIc**

R = R' = H (**a**); R = H, R' = Me (**b**); R = Me, R' = H (**c**).

[†] Deceased.

5-Nitro-substituted 1,3-dihydro-2*H*-imidazo[4,5-*b*]-pyridin-2-one and its 1- and 3-methyl derivatives were described in [7]. Treatment of imidazo[4,5-*b*]pyridin-2-one and its *N*-methyl derivatives with nitric acid under mild conditions gave the corresponding nitrates [2] which were converted into 5-nitro-1,3-dihydro-2*H*-imidazo[4,5-*b*]pyridin-2-ones in concentrated sulfuric acid upon raising the temperature from 0 to 20°C.

In continuation of our studies on the nitration of 1,3-dihydro-2*H*-imidazo[4,5-*b*]pyridin-2-one derivatives, in the present work we examined the behavior under nitration conditions of imidazo[4,5-*b*]pyridin-2-ones having nitro, methyl, and halogen substituents in the pyridine fragment. We succeeded in effecting direct nitration of 1,3-dihydro-2*H*-imidazo[4,5-*b*]pyridin-2-one and its 1- and 3-methyl derivatives **Ia**–**Ic**. 5-Nitro compounds **IIa**–**IIc** were obtained by adding substrates **Ia**–**Ic** to a nitrating mixture at 0–5°C, followed by keeping the mixture for 1 h at that temperature and for 2 h at 15°C (Scheme 1). Compounds **IIa**–**IIc** were isolated in 82–85% yield.

The presence of a nitro group in the pyridine fragment of **IIa**–**IIc** does not hamper further substitution. By nitration of compounds **IIa**–**IIc** at 60°C we obtained the corresponding 5,6-dinitro derivatives **IIIa**–**IIIc**. The latter can be synthesized directly by nitration of **Ia**–**Ic** at 60°C.

The structure of 5,6-dinitro-1,3-dihydro-2*H*-imidazo[4,5-*b*]pyridin-2-one (**IIIa**) was also confirmed by independent synthesis, i.e., by nitration of 6-nitro-1,3-dihydro-2*H*-imidazo[4,5-*b*]pyridin-2-one [8]. Dinitro compounds **IIIa**–**IIIc** obtained from **Ia**–**Ic** and **IIa**–**IIc** were identical in the melting points and ^{1}H NMR spectra. Treatment of **IIIa**–**IIIc** with dimethyl sulfate in alkaline medium gave 1,3-dimethyl-5,6-dinitro-1,3-dihydro-2*H*-imidazo[4,5-*b*]pyridin-2-one (**IV**).

The ^{1}H NMR spectra of mononitro compounds **IIa**–**IIc** contained doublet signals at δ 8.13–8.21 and 7.69–7.74 ppm from the vicinal 6-H and 7-H protons in the pyridine fragment and signals from the methyl groups on N^1 and N^3 in the imidazole ring. Dinitro derivatives **IIIa**–**IIIc** showed in the spectra only singlets from 7-H at δ 8.26–8.55 ppm.

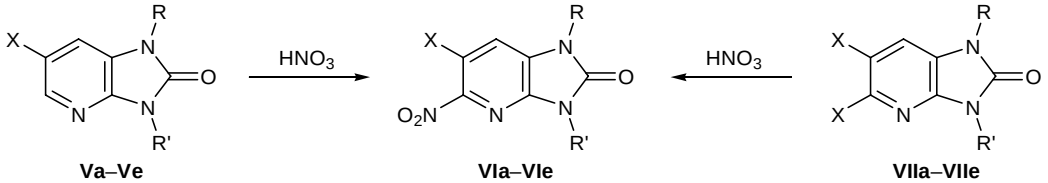
With a view to activate the halogen atoms in the pyridine fragment of 6-chloro(bromo)imidazo[4,5-*b*]-pyridin-2-ones **Va**–**Ve** for nucleophilic replacement, these compounds were subjected to nitration at 60°C. As might be expected, the nitration of **Va**–**Ve** occurred at the 5-position, and the yields of 6-chloro(bromo)-5-nitro-1,3-dihydro-2*H*-imidazo[4,5-*b*]pyridin-2-ones **VIa**–**VIe** thus formed were 31–43% (Scheme 2). 6-Bromo derivative **VId** was also synthesized in a different way, by methylation of 6-bromo-3-methyl-5-nitro-1,3-dihydro-2*H*-imidazo[4,5-*b*]pyridin-2-one (**VIe**) with dimethyl sulfate in alkaline medium.

We also examined the behavior of 5,6-dichloro- and 5,6-dibromo-1,3-dihydro- and 1,3-dimethyl-1,3-dihydro-2*H*-imidazo[4,5-*b*]pyridin-2-ones **VIIa**–**VIId** under the nitration conditions. We anticipated introduction of a nitro group into the 7-position in the pyridine fragment. However, the nitration of **VIIa**–**VIId** was accompanied by replacement of the halogen atom in the α-position with respect to the pyridine nitrogen atom by nitro group. The yields of 6-chloro(bromo) derivatives **VIa**–**VId** were 31–41%. The nitration of 6-bromo-5-methyl-1,3-dihydro-2*H*-imidazo[4,5-*b*]pyridin-2-one (**VIII**) resulted in oxidation of the 5-methyl group to carboxy and formation of 61% of 6-bromo-2-oxo-1,3-dihydro-2*H*-imidazo[4,5-*b*]pyridine-5-carboxylic acid (**IX**).

The structure of nitration products **VIa**–**VIe** was confirmed by the analytical data and ^{1}H NMR spectra. The ^{1}H NMR spectra of **VIa**–**VIe** contained signals from protons on N^1 and N^3 in the imidazole ring and a singlet from 7-H at δ 7.34–8.11 ppm.

Thus the results of our study on the nitration of 1- and 3-methyl-1,3-dihydro-2*H*-imidazo[4,5-*b*]pyridin-2-ones having halogen atoms and a nitro group in the pyridine ring showed that the replacement occurs at the C^5 and C^6 atoms of the pyridine fragment.

Scheme 2.



R = R' = H, X = Cl (**a**), Br (**b**); R = R' = Me, X = Cl (**c**), Br (**d**); R = H, R' = Me, X = Br (**e**).

EXPERIMENTAL

The ^{1}H NMR spectra were recorded on a Varian Gemini-200 spectrometer at 200 MHz using HMDS as internal reference. The purity of the products was checked by TLC on Silufol UV-254 plates using ethanol and chloroform as eluents; spots were visualized under UV light or by treatment with iodine vapor. Compounds **Ia**–**Ic** were synthesized according to the procedures reported in [4, 7, 9]. Compounds **Va**–**Ve**, **VIIa**–**VIIe**, and **VIII** were described in [10].

5-Nitro-1,3-dihydro-2*H*-imidazo[4,5-*b*]pyridin-2-ones IIa–IIc (*general procedure***).** A solution of 10 mmol of compound **Ia**–**Ic** in 9 ml of concentrated sulfuric acid was cooled to 0°C, and 12.5 mmol of nitric acid (d = 1.5 g/cm^3) was added under stirring at such a rate that the temperature did not exceed 5°C. After 1 h, the mixture was allowed to warm up to 15°C, stirred for 2 h at that temperature, and poured onto ice, and the light yellow precipitate was filtered off, washed with cold water until neutral washings, and dried.

5-Nitro-1,3-dihydro-2*H*-imidazo[4,5-*b*]pyridin-2-one (IIa). Yield 82%, mp 350–353°C (from water); published data [7]: mp 338–339°C. ^{1}H NMR spectrum (CD_3COOD), δ, ppm: 7.78 d (1H, 7-H, J = 8.5 Hz), 8.21 d (1H, 6-H, J = 8.5 Hz). Found, %: C 39.78; H 2.19; N 30.86. $C_6H_4N_4O_3$. Calculated, %: C 40.01; H 2.24; N 31.04.

3-Methyl-5-nitro-1,3-dihydro-2*H*-imidazo[4,5-*b*]-pyridin-2-one (IIb). Yield 85%, mp 279–282°C (from alcohol); published data [7]: mp 278–278.5°C. ^{1}H NMR spectrum (CD_3COOD), δ, ppm: 3.54 s (3H, CH_3), 7.74 d (1H, 7-H, J = 8.2 Hz), 8.17 d (1H, 6-H, J = 8.2 Hz). Found, %: C 43.11; H 3.04; N 28.69. $C_7H_6N_4O_3$. Calculated, %: C 43.30; H 3.11; N 28.86.

1-Methyl-5-nitro-1,3-dihydro-2*H*-imidazo[4,5-*b*]-pyridin-2-one (IIc). Yield 84%, mp 356–359°C (from DMF); published data [7]: mp 358–360°C. ^{1}H NMR spectrum (DMSO-d_6), δ, ppm: 3.48 s (3H, CH_3), 7.69 d (1H, 7-H, J = 8.1 Hz), 8.13 d (1H, 6-H, J = 8.1 Hz), 12.21 br.s (1H, 3-H). Found, %: C 43.06; H 3.02; N 28.64. $C_7H_6N_4O_3$. Calculated, %: C 43.30; H 3.11; N 28.86.

5,6-Dinitro-1,3-dihydro-2*H*-imidazo[4,5-*b*]pyridin-2-ones IIIa–IIIc (*general procedure***).** *a*. A solution of 10 mmol of compound **IIa**–**IIc** in 10 ml of concentrated sulfuric acid was cooled to 5°C, 15 mmol of potassium nitrate was added under stirring, and the mixture was heated to 60°C and stirred for 3 h at that temperature. The mixture was cooled and poured onto ice, and the light yellow precipitate was filtered off, washed with cold water, and dried.

b. A solution of 10 mmol of compound **Ia**–**Ic** in 10 ml of concentrated sulfuric acid was cooled to 0°C, 2.5 mmol of nitric acid (d = 1.5 g/cm^3) was added in portions under stirring, maintaining the temperature below 5°C, and the mixture was heated to 60°C and stirred for 2 h at that temperature. The mixture was poured onto ice, and the light yellow precipitate was filtered off, washed with cold water, and dried.

5,6-Dinitro-1,3-dihydro-2*H*-imidazo[4,5-*b*]pyridin-2-one (IIIa). Yield 43% (*a*), 39% (*b*), mp >300°C (from ethanol). ^{1}H NMR spectrum (CD_3COOD), δ, ppm: 8.28 s (1H, 7-H), 11.56 br.s (2H, NH), Found, %: C 31.86; H 1.31; N 30.92. $C_6H_3N_5O_5$. Calculated, %: C 32.01; H 1.34; N 31.11.

3-Methyl-5,6-dinitro-1,3-dihydro-2*H*-imidazo-[4,5-*b*]pyridin-2-one (IIIb). Yield 72% (*a*), 64% (*b*), mp 274–276°C (*a*), 275–278°C (*b*) (from propan-2-ol). ^{1}H NMR spectrum (CD_3COOD), δ, ppm: 3.55 s (3H, CH_3), 8.26 s (1H, 7-H), 11.55 br.s (1H, 1-H). Found, %: C 34.98; H 2.05; N 29.67. $C_7H_5N_5O_5$. Calculated, %: C 35.16; H 2.11; N 29.84.

1-Methyl-5,6-dinitro-1,3-dihydro-2*H*-imidazo-[4,5-*b*]pyridin-2-one (IIIc). Yield 84% (*a*), 70% (*b*), mp >300°C (from propan-2-ol). ^{1}H NMR spectrum (DMSO-d_6), δ, ppm: 3.49 s (3H, CH_3), 8.55 s (1H, 7-H). Found, %: C 35.01; H 2.06; N 29.70. $C_7H_5N_5O_5$. Calculated, %: C 35.15; H 2.11; N 29.84.

Samples of **IIIa**–**IIIc** prepared according to procedures *a* and *b* showed no depression of the melting point on mixing.

Compound **IIIa** was also synthesized by nitration of 10 mmol of 6-nitro-1,3-dihydro-2*H*-imidazo[4,5-*b*]-pyridin-2-one with 15 mmol of nitric acid (d = 1.5 g/cm^3) as described above in *b*. Yield 45%, mp >300°C (from ethanol). ^{1}H NMR spectrum (CD_3COOD), δ, ppm: 8.27 s (1H, 7-H), 11.55 br.s (2H, NH). Found, %: C 31.88; H 1.32; N 30.95. $C_6H_3N_5O_5$. Calculated, %: C 32.01; H 1.34; N 31.11.

1,3-Dimethyl-5,6-dinitro-1,3-dihydro-2*H*-imidazo[4,5-*b*]pyridin-2-one (IV). Compound **IIIb** or **IIIc**, 2 g (8.3 mmol), was dissolved in 20 ml of 6% aqueous sodium hydroxide, and 2.7 ml (29 mmol) of dimethyl sulfate was added under stirring at room temperature. The mixture was stirred for 1.5 h, 4 ml of a 8% solution of NaOH and 1 ml (10.7 mmol) of dimethyl sulfate were added, and the mixture was stirred for 1 h. The light yellow precipitate was filtered

off, washed with water, and recrystallized from propan-2-ol. Yield 1.2 g (57%), mp 183–185°C. ^{1}H NMR spectrum (DMSO-d_6), δ, ppm: 3.49 s (3H, CH_3), 3.56 s (3H, CH_3). 8.55 s (1H, 7-H). Found, %: C 37.81; H 2.73; N 27.58. $C_8H_7N_5O_5$. Calculated, %: C 37.95; H 2.79; N 27.66.

5-Nitro-6-chloro(bromo)-1,3-dihydro-2*H*-imidazo[4,5-*b*]pyridin-2-ones VIa–VIe (*general procedure***).** *a*. A solution of 9 mmol of compound **Va**–**Ve** in 8 ml of concentrated sulfuric acid was cooled to 0°C, and 10 mmol of potassium nitrate or 12 mmol of concentrated nitric acid (d = 1.5 g/cm^3) was added in portions under stirring, maintaining the temperature below 5°C. After 0.5 h, the temperature was raised to 60°C, and the mixture was stirred for 5–7 h at that temperature and poured onto ice. The light yellow precipitate was filtered off, washed with cold water, and dried.

b. A solution of 8 mmol of compound **VIIa**–**VIId** in 12 ml of concentrated sulfuric acid was cooled to 0°C, and 10 mmol of potassium nitrate was added in portions under stirring, maintaining the temperature below 5°C. After 0.5 h, the mixture was heated to 95°C, kept for 3 h at that temperature, cooled, and poured onto ice, and the light yellow precipitate was filtered off, washed with cold water, and dried.

6-Chloro-5-nitro-1,3-dihydro-2*H*-imidazo[4,5-*b*]-pyridin-2-one (VIa). Yield 43% (*a*), 41% (*b*), mp >300°C (from ethanol). ^{1}H NMR spectrum (DMSO-d_6), δ, ppm: 7.34 s (1H, 7-H), 11.12 br.s (1H, 1-H), 11.63 br.s (1H, 3-H). Found, %: C 33.41; H 1.36; N 25.95. $C_6H_3ClN_4O_3$. Calculated, %: C 33.58; H 1.41; N 26.11.

6-Bromo-5-nitro-1,3-dihydro-2*H*-imidazo[4,5-*b*]-pyridin-2-one (VIb). Yield 31% (*a*), 37% (*b*), mp >300°C (from propan-2-ol). ^{1}H NMR spectrum (DMSO-d_6), δ, ppm: 7.66 s (1H, 7-H), 11.61 br.s (1H, 1-H), 12.09 br.s (1H, 3-H). Found, %: C 27.64; H 1.12; N 21.63. $C_6H_3BrN_4O_3$. Calculated, %: C 27.82; H 1.17; N 21.63.

6-Chloro-1,3-dimethyl-5-nitro-1,3-dihydro-2*H*-imidazo[4,5-*b*]pyridin-2-one (VIc). Yield 39% (*a*), 33% (*b*), mp 222–224°C (*a*), 220–222°C (*b*) (from H_2O–AcOH, 1:5). ^{1}H NMR spectrum ($CDCl_3$), δ, ppm: 3.54 s (3H, 1-CH_3), 3.55 s (3H, 3-CH_3), 7.38 s (1H, 7-H). Found, %: C 39.45; H 2.86; N 22.90. $C_8H_7ClN_4O_3$. Calculated, %: C 39.60; H 2.91; N 23.09.

6-Bromo-1,3-dimethyl-5-nitro-1,3-dihydro-2*H*-imidazo[4,5-*b*]pyridin-2-one (VId). Yield 40% (*a*), 35% (*b*), mp 243–245°C (from AcOH). ^{1}H NMR spectrum (DMSO-d_6), δ, ppm: 3.43 s (3H, 1-CH_3), 3.47 s (3H, 3-CH_3), 8.11 s (1H, 7-H). Found, %: C 33.28; H 2.40; N 19.32. $C_8H_7BrN_4O_3$. Calculated, %: C 33.47; H 2.46; N 19.51.

6-Bromo-3-methyl-5-nitro-1,3-dihydro-2*H*-imidazo[4,5-*b*]pyridin-2-one (VIe). Yield 37% (*a*), 31% (*b*), mp 246–248°C (*a*), 247–249°C (*b*) (from propan-2-ol). ^{1}H NMR spectrum (DMSO-d_6), δ, ppm: 3.50 s (3H, 3-CH_3), 7.54 s (1H, 7-H), 11.23 br.s (1H, 1-H). Found, %: C 33.28; H 2.40; N 19.32. $C_8H_7BrN_4O_3$. Calculated, %: C 33.47; H 2.46; N 19.51.

Samples of **VIa**–**VIe** prepared according to procedures *a* and *b* showed no depression of the melting point on mixing

c. *Compound **VId***. Compound **VIe**, 0.6 g (2.4 mmol), was dissolved in 6 ml of a 6% solution of sodium hydroxide, and 0.67 ml (7.2 mmol) of dimethyl sulfate was added under stirring at room temperature. After 3 h, the yellow–green precipitate was filtered off, washed with cold water, and dried. Yield 0.35 g (51%), mp 244–245°C (from AcOH). ^{1}H NMR spectrum (DMSO-d_6), δ, ppm: 3.42 s (3H, 1-CH_3), 3.47 s (3H, 3-CH_3), 8.12 s (1H, 7-H). Found, %: C 33.30; H 2.41; N 19.36. $C_8H_7BrN_4O_3$. Calculated, %: C 33.47; H 2.46; N 19.51.

6-Bromo-2-oxo-1,3-dihydro-2*H*-imidazo[4,5-*b*]-pyridine-5-carboxylic acid (IX). A solution of 0.68 g (3 mmol) of 6-bromo-5-methyl-1,3-dihydro-2*H*-imidazo[4,5-*b*]pyridin-2-one (**VIII**) in 5 ml of concentrated sulfuric acid was cooled to 3°C, and 0.32 g (3.2 mmol) of potassium nitrate was added in portions under stirring, maintaining the temperature below 5°C. After 0.5 h, the mixture was heated to 75°C, stirred for 3 h at that temperature, cooled, and poured onto ice, and the light brown precipitate was filtered off, washed with cold water, and dried. Yield 0.47 g (61%), mp >300°C (from AcOH). ^{1}H NMR spectrum (DMSO-d_6), δ, ppm: 7.52 s (1H, 7-H), 10.08 s (1H, COOH), 11.55 br.s (1H, 1-H), 11.87 br.s (1H, 3-H). Found, %: C 32.41; H 1.50; N 16.13. $C_7H_4BrN_3O_3$. Calculated, %: C 32.58; H 1.56; N.28.

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