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Cyclizations of Monocyclic 5-Nitropyridin-2(1*H*)-ones

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Abstract—Reactions of 5-nitropyridin-2(1*H*)-one and its *N*-methyl derivative with hydrazine hydrate led to the formation of (1*H*-pyrazol-3-yl)acetohydrazide. Under analogous conditions, 1,3-dimethyl-5-nitropyridin-2(1*H*)-one gave rise to 2-(1*H*-pyrazol-3-yl)propionohydrazide, while 6-methyl-5-nitropyridin-2(1*H*)-one was converted into (5-methyl-1*H*-pyrazol-3-yl)acetohydrazide. Hydrazinolysis of 4-methyl-5-nitropyridin-2(1*H*)-one resulted in the formation of 3-methyl-4-nitro-1*H*-pyrazole. The mechanism of recyclization of nitropyridine derivatives by the action of hydrazine hydrate was studied using 5-nitropyridin-2(1*H*)-one and 1-methyl-5-nitropyridin-2(1*H*)-one as examples.

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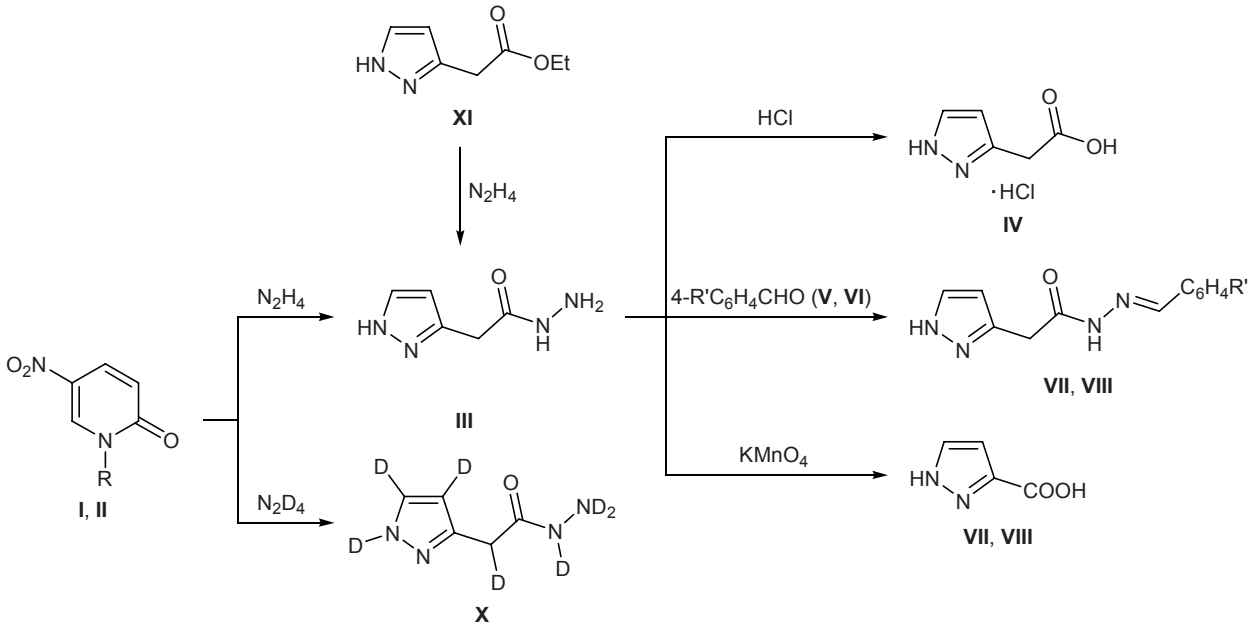
While studying chemical properties of substituted 7-nitro-1,5-dihydro-4*H*-imidazo[4,5-*c*]pyridin-4-ones, we found that they undergo transformation into 7-methyl-1,5-dihydro-4*H*-imidazo[4,5-*d*]pyridazin-4-one derivatives by the action of excess hydrazine hydrate [1]. Using as examples 5-nitropyridin-2-ones fused to a triazole, benzene, pyridine, or thiophene ring, we showed that the observed recyclization is general [2]. Taking into account the above data [1, 2], we believed worthwhile to examine the behavior of monocyclic 5-nitropyridin-2(1*H*)-one (**I**) and its *N*-methyl analog **II** under hydrazinolysis conditions. We have found that compounds **I** and **II** react with hydrazine hydrate to give base **III** whose elemental composition corresponds to the formula $C_5H_8N_4O$ rather than the expected 6-methylpyridazin-3-one which is a structural fragment of the hydrazinolysis products obtained from 5-nitropyridin-2(1*H*)-ones [1, 2]. Qualitative tests with Tollens' reagent and a solution of potassium hypobromite indicated the presence of a hydrazine moiety in molecule **III**. The IR spectrum of **III** contained strong absorption bands at 1660 and 1630 cm^{-1} due to stretching vibrations of C=O and C=N bonds, respectively. Compound **III** displayed in the ^1H NMR spectrum a singlet at δ 4.23 ppm from methylene protons and two doublets at δ 6.85 and 8.12 ppm with a coupling constant *J* of 2.5 Hz. These data suggest the absence of pyridine ring in molecule **III**, for *ortho* protons in a pyridine ring are coupled with a constant *J* of 6–7 Hz. Judging by the ^1H NMR data (chemical shifts and coupling constants), IR spectrum, and elemental composition, compound **III** was presumed to be a pyrazole derivative. Treatment of **III** with concentrated hydrochloric acid on heating gave hydrochloride **IV** and hydrazine hydrochloride. The ^1H NMR spectrum of **IV** resembled that of free base **III**, and the IR spectrum of **IV** contained a carbonyl absorption band at 1725 cm^{-1}; therefore, compound **IV** was assigned the structure of pyrazolylacetic acid hydrochloride, and base **III** was identified as the corresponding hydrazide [3] (Scheme 1). By heating hydrazide **III** with *p*-chloro- and *p*-nitrobenzaldehydes **V** and **VI** in alcoholic solution we obtained hydrazones **VII** and **VIII**, respectively. The oxidation of **III** with potassium permanganate gave 1*H*-pyrazole-3-carboxylic acid (**IX**). The IR and ^1H NMR spectra of compound **IX** were identical to those of an authentic sample of 1*H*-pyrazole-3-carboxylic acid synthesized according to the procedure described in [4]. The spectral parameters of hydrazones **VII** and **VIII** confirmed that the hydrazone fragment is linked to both the carbonyl group and methine group of the benzylidene residue. The structure of hydrazide **III** was finally proved by its independent synthesis from ethyl (1*H*-pyrazol-3-yl)acetate (**XI**) according to the procedure described in [5].

The reaction of 5-nitropyridin-2(1*H*)-one (**I**) with excess tetradeuterohydrazine hydrate on heating led to the formation of (1*H*-pyrazol-3-yl)acetohydrazide (**X**)

[†] Deceased.

Scheme 1.

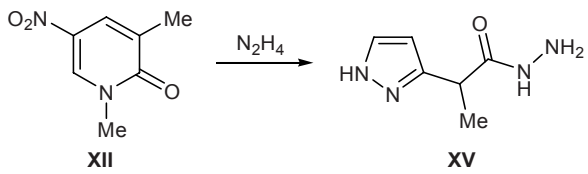


I, R = H; **II**, R = Me; **V**, **VII**, R′ = Cl; **VI**, **VIII**, R′ = NO_2.

having deuterium atoms in positions *1*, *4*, and *5* of the pyrazole ring and one deuterium atom in the side-chain methylene group, as followed from its elemental analysis and ^{1}H NMR spectrum. These findings indicate that the transformation of 5-nitropyridin-2(1*H*)-one (**I**) into pyrazole involves the C^4, C^5, and C^6 atoms of the pyridine ring and that the C^2 and C^3 atoms of the pyridine ring give rise to the acetic acid fragment.

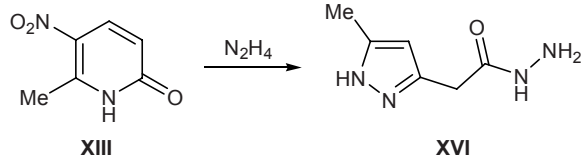
With a view to elucidate the mechanism of recyclization of 5-nitropyridin-2(1*H*)-one derivatives into pyrazoles it was necessary to examine the effect of methyl substitution in positions *3*, *4*, and *6* of the initial compound on the product structure. For this purpose, we performed hydrazinolysis of 1,3-dimethyl-, 6-methyl-, and 4-methyl-5-nitropyridin-2(1*H*)-ones **XII**–**XIV**. The reaction of 1,3-dimethyl-5-nitropyridin-2(1*H*)-one (**XII**) with excess hydrazine hydrate on heating for 5–7 h was accompanied by evolution of ammonia, and the elemental composition of the isolated product (compound **XV**) corresponded to the formula $C_6H_{10}N_4O$. The product showed a positive test for hydrazine fragment. Its ^{1}H NMR spectrum contained two doublets at δ 6.30 and 7.58 ppm with a coupling constant *J* of 2.2 Hz. In addition, signals from methyl protons (δ 1.61 ppm, d) and CH proton (δ 3.81 ppm, q) were present; these signals were assigned to a side-chain ethyl fragment. Taking into account the coupling constants and chemical shifts in the ^{1}H NMR spectra of the initial compound and reaction product and elemental analysis, compound **XV** was assigned the structure of 2-(1*H*-pyrazol-3-yl)propionohydrazide (Scheme 2).

Scheme 2.



Under analogous conditions, from 6-methyl-5-nitropyridin-2(1*H*)-one (**XIII**) and excess hydrazine hydrate we obtained (5-methyl-1*H*-pyrazol-3-yl)acetohydrazide (**XVI**) (Scheme 3).

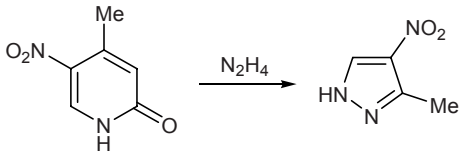
Scheme 3.



Unlike nitropyridinones **I**, **II**, **XII**, and **XIII**, hydrazinolysis of 4-methyl-5-nitropyridin-2(1*H*)-one (**XIV**) gave a product which showed negative tests for amino and hydrazino groups. The reaction was accompanied by evolution of ammonia. Elemental analysis of the

product gave the formula $C_4H_5N_3O_2$, and its molecular weight was determined by mass spectrometry, *M* 126. The IR spectrum contained absorption bands at 1365 and 1515 cm^{-1}, which are typical of symmetric and asymmetric stretching vibrations of a nitro group, respectively. In the ^1H NMR spectrum of the product we observed a signal at δ 2.45 ppm from methyl protons and a downfield signal at δ 8.28 ppm. These data allowed us to identify the product of transformation of compound **XIV** as 3-methyl-4-nitro-1*H*-pyrazole (**XVII**) (Scheme 4). Compound **XVII** was identical to an authentic sample obtained by nitration of 3-methyl-1*H*-pyrazole (**XVIII**) [6] in the melting point and ^1H NMR spectrum.

Scheme 4.

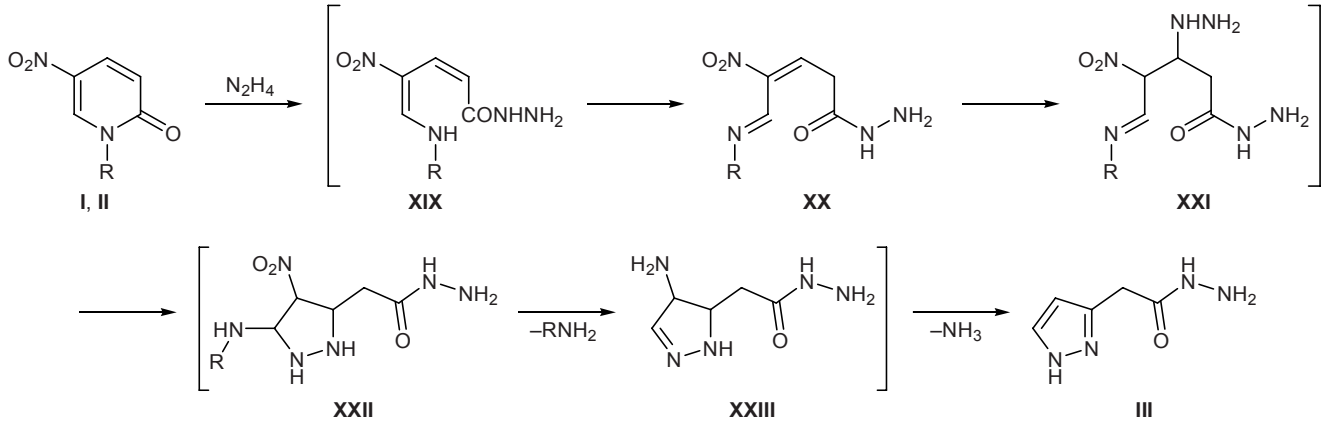


XIV **XVII**

On the basis of our experimental results we proposed a probable mechanism for the transformation of monocyclic 5-nitropyridin-2(1*H*)-ones **I**, **II**, **XII**, and **XIII** into pyrazole derivatives using compounds **I** and **II** as examples (Scheme 5). Strong electron-withdrawing effect of the nitro group on the carbonyl carbon atom (C^2) in 5-nitropyridin-2(1*H*)-one favors nucleophilic attack by hydrazine molecule, followed by opening of the pyridine ring at the C^2–N^1 bond. Hydrazide **XIX** thus formed undergoes aminodiene–enimine isomerization to give structure **XX** which takes up the second hydrazine molecule at the nitroethylene fragment. Next follows intramolecular cyclization of intermediate **XXI** to tetrahydropyrazole derivative **XXII**, and the subsequent elimination of ammonia (or methylamine) molecule, reduction of the nitro group, and elimination of the second ammonia molecule from structure **XXIII** finally yields (1*H*-pyrazol-3-yl)acetohydrazide (**III**). Ammonia and methylamine liberated during the process were detected among the gaseous reaction products by mass spectrometry. An alternative explanation of the mechanistic details is also possible, for example, replacement of the NHR group in structure **XIX** by the hydrazine residue and subsequent intramolecular addition of the terminal amino group at the β-position of intermediate α,β-unsaturated carboxylic acid hydrazide.

It should be noted that intermediate structures outlined in Scheme 5 are purely hypothetical. In order to confirm the proposed scheme it was necessary to use compounds modeling the intermediate structures formed as a result of opening of the pyridine ring under hydrazinolysis conditions. Thus participation of hypothetical structure **XXI** in the process was proved using 4-nitro-5-piperidinopenta-2,4-dienoic acid (**XXIV**) as model compound. Acid **XXIV** was synthesized as shown in Scheme 6. Bromination of furfurol (**XXV**) gave dibromoformylacrylic acid **XXVI** [7] which was treated with sodium nitrite to obtain 2-nitromalonaldehyde sodium salt (**XXVII**) [8]. The reaction of the latter with piperidine afforded α-nitro-β-piperidinoacrolein (**XXVIII**) [9], and condensation of **XXVIII** with malonic acid resulted in the formation of acid **XXIV** [10]. When model compound **XXIV** was heated with hydrazine hydrate under the same conditions as in the transformation of 5-nitropyridin-2(1*H*)-ones **I** and **II**, we observed evolution of ammonia and isolated (1*H*-pyrazol-3-yl)acetohydrazide (**III**).

Scheme 5.

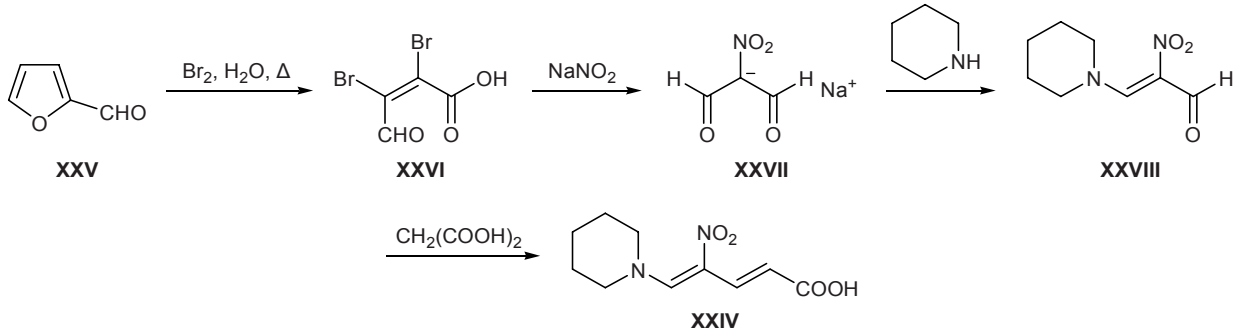


I, **II** **XIX** **XX** **XXI**

XXII **XXIII** **III**

R = H, Me.

Scheme 6.



Thus hydrazinolysis of monocyclic 5-nitropyridin-2(1*H*)-ones **I**, **II**, **XII**, and **XIII** provides a new synthetic route to pyrazole derivatives like **III**, **XV**, and **XVI**.

EXPERIMENTAL

The ^{1}H NMR spectra were recorded on a Tesla BS-467 spectrometer (80 MHz) using CF_3COOH as solvent and on a Varian Gemini-200 spectrometer (200 MHz) from solutions in DMSO-d_6 and $CDCl_3$; the chemical shifts were measured relative to hexamethyldisiloxane as internal reference. The IR spectra were recorded on UR-20 and Specord 75IR instruments from samples prepared as KBr pellets or dispersed in mineral oil. The molecular weight of compound **XVII** was determined by mass spectrometry on a Varian MAT-112 instrument (70 eV) with direct sample admission into the ion source. The purity of the products was checked by TLC on Silufol UV-254 plates using alcohol and chloroform as eluents; spots were visualized under UV light or by treatment with iodine vapor. Compounds **I**, **II**, **XIII**, and **XIV** were prepared according to the procedure described in [11].

(1*H*-Pyrazol-3-yl)acetohydrazide (III). *a*. A mixture of 10 g (71.4 mmol) of 5-nitropyridin-2(1*H*)-one (**I**) and 67 ml (1.4 mol) of 95% hydrazine hydrate was heated for 3–5 h at 100–110°C. The reaction was accompanied by evolution of ammonia which was identified as ammonium chloride (mp 334–336°C [13]) by passing the gaseous products through a solution of hydrogen chloride in ethanol. When the reaction was complete, the mixture was evaporated to dryness in a stream of argon. The residue was ground with ethanol, and the precipitate was filtered off, and recrystallized from ethanol. Yield 7.5 g (75%), colorless crystals, mp 181–183°C. IR spectrum, ν, cm^{-1}: 1630 (C=N), 1660 (C=O). ^{1}H NMR spectrum (CF_3COOH), δ, ppm: 4.25 s (2H, 3-CH_2), 6.85 d (1H, 4-H, J = 2.5 Hz), 8.12 d (1H, 5-H, J = 2.5 Hz). Found, %: C 42.69; H 5.71; N 39.83. $C_5H_8N_4O$. Calculated, %: C 42.85; H 5.75; N 39.98.

b. A mixture of 1.2 g (7.8 mmol) of 1-methyl-5-nitropyridin-2(1*H*)-one (**II**) and 7.3 ml (156.0 mmol) of hydrazine hydrate was heated for 3–5 h at 110–120°C. The reaction was accompanied by evolution of ammonia and methylamine which were identified as the corresponding hydrochlorides by passing the gaseous products through a solution of hydrogen chloride in ethanol. Equimolar amounts of ammonium chloride (mp 332°C [13]) and methylammonium chloride (mp 224°C [13]) were isolated; they were separated by fractional crystallization from anhydrous ethanol [14]. Compound **III** was isolated as described above in *a*. Yield 0.8 g (72%), colorless crystals, mp 181–183°C. IR spectrum, ν, cm^{-1}: 1630 (C=N), 1660 (C=O). ^{1}H NMR spectrum (CF_3COOH), δ, ppm: 4.24 s (2H, 3-CH_2), 6.86 d (1H, 4-H, J = 2.5 Hz), 8.12 d (1H, 5-H, J = 2.5 Hz). Found, %: C 42.72; H 5.70; N 39.85. $C_5H_8N_4O$. Calculated, %: C 42.85; H 5.75; N 39.98.

c. A mixture of 1.5 g (10.0 mmol) of ethyl (1*H*-pyrazol-3-yl)acetate (**XI**) [5], 6.0 ml of propan-2-ol, and 1.5 ml (32.1 mmol) of hydrazine hydrate was heated for 2 h on a water bath. The mixture was evaporated to dryness, the residue was ground with alcohol, and the precipitate was filtered off. Yield 1.3 g (95%), mp 181–183°C (from alcohol). The product showed no depression of the melting point on mixing with samples of **III** obtained as described in *a* and *b*. All samples had identical IR and ^{1}H NMR spectra.

d. A mixture of 1.2 g (5.3 mmol) of 4-nitro-5-piperidinopenta-2,4-dienoic acid (**XXIV**) and 5.0 ml (100 mmol) of hydrazine hydrate was heated for 3 h at 100–110°C. The reaction was accompanied by evolution of ammonia. When the reaction was complete, the mixture was evaporated to dryness in a stream of argon, the residue was ground with ethanol, and the precipitate was filtered off and recrystallized from

alcohol. Yield 0.6 g (86%), mp 181–183°C. ^{1}H NMR spectrum (CF_3COOH), δ, ppm: 4.23 s (2H, 3-CH_2), 6.85 d (1H, 4-N, J = 2.5 Hz), 8.12 d (1H, 5-H, J = 2.5 Hz). Found, %: C 42.74; H 5.68; N 39.80. $C_5H_8N_4O$. Calculated, %: C 42.85; H 5.75; N 39.98.

(1*H*-Pyrazol-3-yl)acetic acid hydrochloride (IV). A solution of 1.0 g (7.14 mmol) of hydrazide **III** in 20 ml of concentrated hydrochloric acid was heated for 2–3 h at 100°C. After cooling, the precipitate of hydrazine hydrochloride was filtered off, the filtrate was evaporated to dryness, and the residue was purified by recrystallization from propan-2-ol with addition of activated charcoal. Yield 0.9 g (78%), mp 138–140°C; published data [5]: mp 138–140°C. IR spectrum: ν(C=O) 1725 cm^{-1}. ^{1}H NMR spectrum (CF_3COOH), δ, ppm: 4.16 s (2H, 3-CH_2), 6.83 d (1H, 4-H, J = 2.5 Hz), 8.13 d (1H, 5-H, J = 2.5 Hz).

***N*′-(4-Chlorobenzylidene)(1*H*-pyrazol-3-yl)-acetohydrazide (VII).** A solution of 0.5 g (3.56 mmol) of 4-chlorobenzaldehyde (**V**) in 10 ml of ethanol was added to a solution of 0.5 g (3.57 mmol) of hydrazide **III** in 15 ml of ethanol. The mixture was heated for 10–15 min under reflux and evaporated to dryness. Yield 0.7 g (75%), colorless crystals, mp 184–185°C (from water). IR spectrum, ν, cm^{-1}: 1620 (C=N); 1670 (C=O). ^{1}H NMR spectrum (CF_3COOH), δ, ppm: 4.18 s (1H, CH=N), 4.38 s (2H, 3-CH_2), 6.80 d (1H, 4-H, J = 2.2 Hz), 7.28 d (2H, 3′-H, 5′-H, J = 7.0 Hz), 7.56 d (2H, 2′-H, 6′-H, J = 7.0 Hz), 8.06 d (1H, 5-H, J = 2.2 Hz). Found, %: C 54.62; H 4.17; N 21.19. $C_{12}H_{11}ClN_4O$. Calculated, %: C 54.81; H 4.22; N.33.

***N*′-(4-Nitrobenzylidene)(1*H*-pyrazol-3-yl)aceto-hydrazide (VIII).** A solution of 0.5 g (3.31 mmol) of 4-nitrobenzaldehyde (**VI**) in 10 ml of ethanol was added to a solution of 0.5 g (3.57 mmol) of hydrazide **III** in 15.0 ml of ethanol. The mixture was heated for 10–15 min under reflux and cooled, and the precipitate was filtered off and dried. Yield 0.9 g (99%), light yellow crystals, mp 242–244°C (from aqueous alcohol, 1:1). ^{1}H NMR spectrum (CF_3COOH), δ, ppm: 4.30 s (1H, CH=N), 4.70 s (2H, 3-CH_2), 6.93 d (1H, 4-H, J = 2.2 Hz), 8.03 d (1H, 5-H, J = 2.2 Hz), 7.93–8.40 m (4H, C_6H_4). IR spectrum, ν, cm^{-1}: 1620 (C=N), 1670 (C=O). Found, %: C 52.60; H 4.00; N 25.48; $C_{12}H_{11}N_5O_3$. Calculated, %: C 52.75; H 4.06; N 25.63.

1*H*-Pyrazol-3-ylcarboxylic acid (IX). Potassium permanganate, 6.0 g (38.0 mmol), was added in portions to a solution of 0.9 g (6.4 mmol) of hydrazide **III** in 45 ml of water, heated to 85–90°C, and the mixture was kept for 2 h at that temperature. The precipitate of manganese(IV) oxide was filtered off and washed with several portions of hot water. The filtrate was combined with the washings, evaporated to 1/3 of the initial volume, cooled, and acidified with concentrated hydrochloric acid, and the precipitate was filtered off and dried. Yield 0.5 g (69%), mp 212–214°C (from ethanol); published data [4]: mp 216–217°C. ^{1}H NMR spectrum (CF_3COOH), δ, ppm: 7.33 s (1H, 4-H), 8.25 s (1H, 5-H).

[(1,4,5-$^2H_3$)-1*H*-Pyrazol-3-yl](2,*N*,*N*′,*N*′-$^2H_4$)-acetohydrazide (X). A mixture of 1.0 g (7.0 mmol) of compound (**I**) and 7.8 ml (140 mmol) of deuterated hydrazine hydrate was heated for 3–5 h at 100–110°C. When the reaction was complete, the mixture was evaporated to dryness in a stream of argon. The residue was ground in ethanol-d_6, and the precipitate was filtered off and recrystallized from ethanol-d_6. Yield 0.8 g (80%), mp 130–132°C. ^{1}H NMR spectrum (DMSO-d_6), δ, ppm: 3.36 s (1H, CHD), 3.44 s (1D, CHD), 7.07 s (1D, 4-D), 7.60 s (1D, 5-D). Found, %: C 40.61; H+D 10.20; N 37.89. $C_5HD_7N_4O$. Calculated, %: C 40.80; H+D 10.27; N 38.06.

1,3-Dimethyl-5-nitropyridin-2(1*H*)-one (XII). A solution of 1.6 ml (38.6 mmol) of concentrated nitric acid (d = 1.5 g/cm^3) in 1.6 ml of concentrated sulfuric acid was added under stirring to a solution of 4.0 g (32.5 mmol) of 1,3-dimethylpyridin-2(1*H*)-one [12] in 16 ml of concentrated sulfuric acid, cooled to 0–5°C. After 1 h, the mixture was allowed to warm up to 10–15°C, stirred for 2–3 h at that temperature, poured onto ice, and neutralized with ammonium hydrogen carbonate. The light yellow precipitate was filtered off and dried. Yield 4.7 g (87%), mp 142–143°C (from propan-2-ol). ^{1}H NMR spectrum (DMSO-d_6), δ, ppm: 2.05 s (3H, 3-CH_3), 3.55 s (3H, 1-CH_3), 8.05 d (1H, 4-H, J = 3.0 Hz), 9.06 d (1H, 6-H, J = 3.0 Hz). Found, %: C 49.83; H 4.75; N 16.51. $C_7H_8N_2O_3$. Calculated, %: C 50.00; H 4.80; N 16.66.

2-(1*H*-Pyrazol-3-yl)propionohydrazide (XV). A solution of 1.7 g (10 mmol) of compound **XII** in 10 ml (200 mmol) of hydrazine hydrate was heated for 3–5 h at 110–120°C. During the process, the originally yellow solution turned dark red, and evolution of ammonia and methylamine was observed. The gaseous products were identified as described above in the synthesis of compound **III** according to method *c*. When the reaction was complete, excess hydrazine hydrate was distilled off to dryness in a stream of argon. The residue was treated with ethanol to isolate product **XV**. Yield 1.2 g (78%), mp 129–131°C (from propan-2-ol). IR spectrum, ν, cm^{-1}: 1630 (C=N), 1660

(C=O). ^{1}H NMR spectrum ($CDCl_3$), δ, ppm: 1.61 d (3H, CH_3), 3.81 q (1H, CH), 6.30 d (1H, 4-H, J = 2.2 Hz), 7.58 d (1H, 5-H, J = 2.2 Hz). Found, %: C 46.58; H 6.49; N 36.20. $C_6H_{10}N_2O$. Calculated, %: C 46.74; H 6.54; N 36.34.

(5-Methyl-1*H*-pyrazol-3-yl)acetohydrazide (XVI) was obtained in a similar way from 1.5 g (10 mmol) of compound **XIII** and 10 ml (200 mmol) of hydrazine hydrate. Yield 1.1 g (72%), mp 192–194°C (from ethanol). IR spectrum, ν, cm^{-1}: 1630 (C=N), 1665 (C=O). ^{1}H NMR spectrum (CF_3COOH), δ, ppm: 2.60 s (3H, CH_3), 4.25 s (2H, CH_2), 6.70 s (1H, 4-H). Found, %: C 46.60; H 6.46; N 36.18. $C_6H_{10}N_4O$. Calculated, %: C 46.74; H 6.54; N 36.34.

3-Methyl-5-nitro-1*H*-pyrazole (XVII) was synthesized in a similar way from 1.5 g (10 mmol) of compound **XIV** and 10 ml (200 mmol) of hydrazine hydrate. Yield 0.8 g (64%), mp 132–134°C (from water). IR spectrum, ν, cm^{-1}: 1365 (NO_2, sym.), 1515 (NO_2, asym.). ^{1}H NMR spectrum (DMSO-d_6), δ, ppm: 2.45 s (3H, CH_3), 8.28 s (1H, 5-H). Found, %: C 37.61; H 3.90; N 32.89. $C_4H_5N_3O_2$. $[M]^+$ 126. Calculated, %: C 37.80; H 3.96; N 33.06.

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