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Catalytic Synthesis of 1,3-Propylenediamines

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Abstract—The hydrogenation on Raney nickel of 3-alkenyl-substituted pyrazolines and also of 3-methyl-5-(2-furyl)-1*H*-pyrazoline and 3,3'-bipyrazoline afforded substituted 1,3-diaminobutanes, 1,3-diaminopentanes, 1,3-diaminohexane, and 1,3,4,6-tetraaminohexane. Under the same conditions from 3-acetyl-4-(2-furyl)-1*H*-pyrazoline 3-amino-2-methyl-4-(2-furyl)pyrrolidine was obtained.

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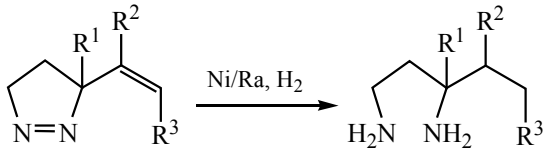
Polymethylene polyamines are found in all living cells, they are involved in many important biological processes and possess a wide range of physiological activity (tuberculocidal, immunodepressant, antitumor etc.) [1–3]. Recent research showed that the polyamine derivatives based on spermidine and spermine exhibit high activity against Alzheimer's disease, cystic fibrosis, and also of various tumors and cancer cells [4–7]. 1,3-Propylenediamine fragment are included into the structure of many drugs used in the treatment of the central nervous system [e.g., antidepressant imipramine (tofranil)] [3]. The modification of the terminal fragments of poly-β-aminoesters with the help of 1,3-diaminopentane improved the biophysical properties of biodegradable polymeric nanoparticles formed as a result of the selfassembling of the DNA plasmids, and of the hydrolytically degradable poly-β-aminoesters favoring the effective function of the nonviral gene transfer to the embryonic human cells (hESCs) [8].

One convenient method of preparation of 1,3-propylenediamines consists in the hydrogenation over Raney Ni under hydrogen pressure of alkyl- and aryl-substituted pyrazolines [9–11]. By this procedure only 1,3-propylenediamines with branched hydrocarbon chain were prepared [9–11]. As known, the hydrogenation direction depends also on the character of the substituent in the initial pyrazoline. For instance, the 1-benzyl-3,5,5-trimethylpyrazoline was converted into toluene, and from methyl 1*H*-pyrazoline-3-carboxylates derivatives of 3- or 1-amino-2-pyrrolidone were obtained [12].

In the present study aiming at the synthesis of new compounds of this series, in particular, of 1,3-propylenediamines of linear structure the hydrogenation was investigated of 3-vinyl- (**Ia**), *cis*-3-[(1*E*)-prop-1-en-1-yl]- (**Ib**), 3-isopropenyl-4,5-dihydro-3*H*-pyrazole (**Ic**), 3-isopropenyl-3-methyl-4,5-dihydro-3*H*-pyrazole (**Id**), 3-methyl-5-(2-furyl)-4,5-dihydro-1*H*-pyrazole (**II**), 3-acetyl-4-(2-furyl)-4,5-dihydro-1*H*-pyrazole (**III**), and 4,4',5,5'-tetrahydro-3*H*,3'*H*-3,3'-bipyrazole (**IV**).

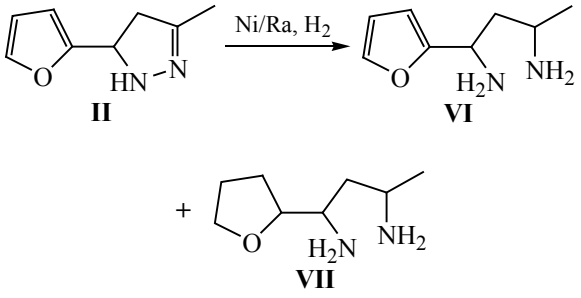
The hydrogenation was carried out in a steel rotating pressure reactor in methanol over Raney nickel at 100°C and the hydrogen pressure 10–13 MPa over 6 h. Under the chosen conditions pyrazolines **Ia–Id** with various vinyl substituents in the position *3* cleanly converted into the corresponding 1,3-propylenediamines **Va–Vd** in 87–99% yields. In none of performed experiments the formation of side products was detected originating from the characteristic of 3-vinylpyrazolines catalytic or thermal dediazotization of the pyrazoline ring [13–15].



Ia–Id **Va–Vd**

$R^1 = R^2 = R^3 = H$ (**a**); $R^1 = R^2 = H$, $R^3 = Me$ (**b**); $R^1 = R^3 = H$, $R^2 = Me$ (**c**); $R^1 = R^2 = Me$, $R^3 = H$ (**d**).

The hydrogenation of 3-methyl-5-(2-furyl)-4,5-dihydro-1*H*-pyrazole (**II**) containing a furan substituent in the position *5* of the heterocycle proceeded both at the pyrazoline fragment and the C=C bonds of furan ring. We isolated from the reaction mixture 1,3-diamino-1-(2-furyl)butane (**VI**) and 1,3-diamino-1-(2-tetrahydrofuryl) butane (**VII**) in 9 and 68% yield respectively. 1,3-Diaminobutane (**VII**) was present as a mixture of diastereomers in the ratio 1 : 1.5 (according to ^{13}C NMR data).



II **VI** **VII**

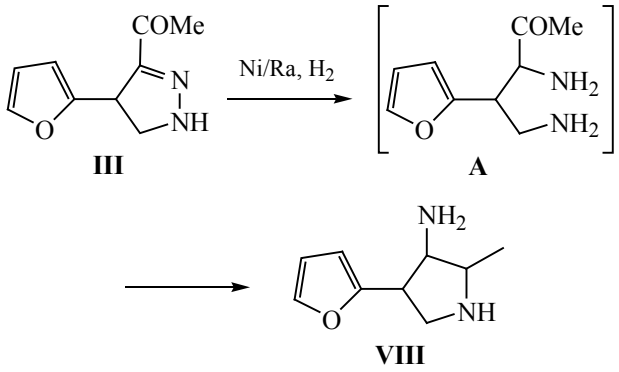
Under the chosen conditions the only product of the catalytic reduction of 3-acetyl-4-(2-furyl)-4,5-dihydro-1*H*-pyrazole (**III**) was 3-amino-2-methyl-4-(2-furyl) pyrrolodine (**VIII**) obtained in 84% yield due to the intramolecular condensation of diamine **A** (Scheme 1).

The structure of compound **VIII** was proved by NMR and mass spectra. In the ^1H NMR spectrum the proton signal of the methyl group gives rise to a doublet at 1.26 ppm, and signals of NH_2 and NH groups appear as a broad singlet at 1.72 ppm. The proton signals of the pyrrolodine ring at the atoms C^2 and C^3 are observed as a multiplet (2.76–2.90 ppm) and a doublet of doublets (3.31 ppm), ^3J 9.9 and ^3J 10.2 Hz. The characteristic signals of the atoms $C^{3'}$, $C^{4'}$, $C^{5'}$, $C^{2'}$ of the furan ring appear in the ^{13}C NMR spectrum at 104.99, 110.00, 141.53, 155.74 ppm respectively.

The mass spectrum of compound **VIII** contained the molecular ion peak, *m/z* 166.1103. The intensity of the molecular ion peaks of all synthesized 1,3-propylenediamines was very low (<0.1%). The interpretation of the mass spectrum of heterocycle **VIII** underlain by the concept of charge and unpaired electron localization showed that the characteristic diagnostic fragments formed by elimination of NH_3 and the decomposition of the nitrogen heterocycle.

The reduction of 4,4',5,5'-tetrahydro-3*H*,3'*H*-3,3'-bipyrazole (**IV**) gave 1,3,4,6-tetraaminohexane (**IX**) in 99% yield as a difficultly separable diastereomeric mixture in the ratio 1 : 1.2 (according to ^{13}C NMR data) (Scheme 2).

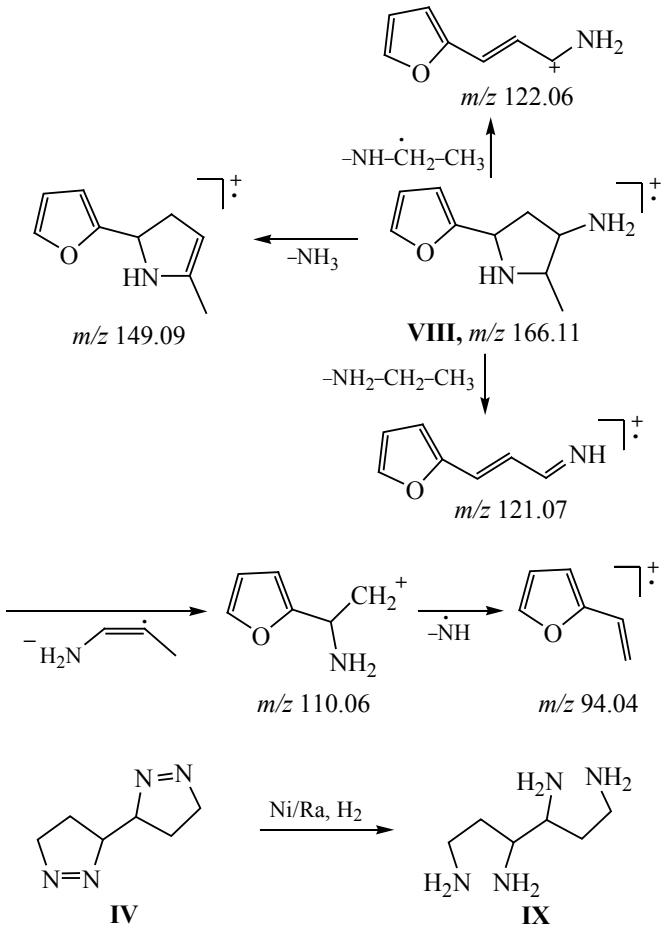
Scheme 1.



III **A** **VIII**

The composition and structure of compounds obtained was proved by mass, IR, ^1H and ^{13}C NMR spectra. For instance, the presence in the IR spectra of the absorption bands of NH_2 groups in the region 3115–3356 cm^{-1} confirmed the formation of the amine fragment. In the ^1H NMR spectra the proton signals of NH_2 and NH groups appear as broadened singlets in the region 1.25–

Scheme 2.



IV **IX**

2.51 ppm. The signals of atoms C^3 (49.40–54.47 ppm) in the ^{13}C NMR spectra of 1,3-propylenediamines are located downfield as compared with the signals of atom C^1 (39.56–42.39 ppm).

Therefore the catalytic hydrogenation of vinyl-substituted pyrazolines with hydrogen in the presence of Raney nickel makes it possible to obtain the corresponding 1,3-diamines which at the presence of a keto group in the molecule immediately transform into pyrrolodines.

EXPERIMENTAL

^1H and ^{13}C NMR spectra were registered on a spectrometer Bruker AM-300 (300.13 and 75.47 MHz respectively), internal reference Me_4Si. IR spectra were recorded on spectrophotometers UR-20 and Specord M-80 from thin films. Mass spectra were obtained on a liquid chromato-mass spectrometer Shimadzu LCMS-2010EV in the chemical ionization mode at the atmospheric pressure, and on a high resolution GC-MS instrument Thermo Finnigan MAT 95 XP at the ionizing electrons energy 70 eV (ionizing chamber temperature 250°C, direct sample admission at 50–270°C, heating rate 10 deg/min).

Initial compounds **Ia, Ic,Id, II–IV** were prepared by procedures [13, 14, 16–18].

Raney nickel. To a mixture of 160 mg of powdery nickel-aluminum alloy (50/50) in 6 ml of water was added within 20–25 min 4.0 g (0.1 mol) of NaOH. The mixture was heated at 40–45°C till total end of gas evolution. The Raney nickel was washed with distilled water till neutral washings, the with MeOH (5×5–6 ml).

3-Isopropenyl-4,5-dihydro-3*H*-pyrazole (Ic). To 78 g (0.48 mol) of isoprene was added 243 ml of ether solution of diazomethane prepared from 24.3 g (0.236 mol) of N-methyl-N-nitrosourea, and the mixture was left for 48 h in the dark at room temperature. The solvent was removed at a reduced pressure. The residue was distilled in a vacuum. Yield 12.43 g (64%), light-yellow oily fluid, bp 40°C (1 kPa). IR spectrum, ν, cm^{-1}: 2974, 1649 (C=C), 1548 (N=N), 1456, 1375, 1278, 898. ^1H NMR spectrum ($CDCl_3$), δ, ppm: 1.19–1.31 m (1H, H^4), 1.65 br.s (3H, Me), 1.65–1.74 m (1H, H^4), 4.10–4.20 m (1H, H^5), 4.47–4.51 m (1H, H^5), 4.74 t (1H, $H^{1'}$, *J* 8.7 Hz), 4.84 br.s (1H, $H^{2'}$), 4.86 br.s (1H, $H^{2'}$). ^{13}C NMR spectrum ($CDCl_3$), δ, ppm: 19.02 (Me), 21.21 (C^4), 75.34 (C^5), 91.46 (C^3), 111.85 ($C^{2'}$), 141.50 ($C^{1'}$). Mass spectrum (Chemical ionization): *m/z* 111 $[M + H]^+$. Mass spectrum, *m/z* (I_{rel}, %): 82 (25) $[M - N_2]^+$, 81 (13), 67 (100), 65 (12), 53 (14), 41 (25). M^+ 110.1466. $C_6H_{10}N_2$. *M* 110.1571.

General procedure of pyrazolines hydrogenation. In a steel rotating pressure reactor (*V* 100 cm^3) was charged the pyrazoline solution in 20 ml of MeOH, Raney nickel prepared from 0.16 g of nickel-aluminum alloy, and the reaction mixture was maintained for 6 h at 100°C and hydrogen pressure 10–13 MPa. The catalyst was filtered off, the solvent was distilled off at a reduced pressure.

1,3-Diaminopentane (Va). From 1 g (0.01 mol) of pyrazoline **Ia** was obtained 0.89 g (87%) of diamine **Va**. IR spectrum, ν, cm^{-1}: 3334–3257, 2958–2873, 1575–1436, 1319 (NH_2). ^1H (NMR spectrum $CDCl_3$), δ, ppm: 0.87 t (3H, Me, *J* 7.3 Hz), 1.18–1.58 m (4H, H^2, H^4), 1.64 br.s (4H, NH_2), 2.68 m (1H, H^3), 2.82 m (2H, H^1). ^{13}C NMR spectrum ($CDCl_3$), δ, ppm: 10.11 (Me), 30.87 (C^4), 39.21 (C^2), 40.06 (C^1), 50.89 (C^3). Mass spectrum (Chemical ionization): *m/z* 103 $[M + H]^+$. Mass spectrum, *m/z* (I_{rel}, %): 85 (54) $[M - NH_3]^+$, 73 (11) $[M - Et]^+$, 72 (69) $[M - NH_2 - CH_2]^+$, 58 (100) $[M - NH_2 - CH_2 - CH_2]^+$, 56 (68), 44 (84) $[NH_2CH_2CH_2]^+$, 41 (27), 32 (99), 29 (83). M^+ 102.0933. $C_5H_{14}N_2$. *M* 102.1781.

The physicochemical constants of 1,3-diaminopentane **Va** coincided with the published data [19].

1,3-Diaminohexane (Vb). From 1 g (0.009 mol) of pyrazoline **Ib** was obtained 1.03 g (99%) of diamine **Vb**. IR spectrum, ν, cm^{-1}: 3356–3196, 2956–2872, 1600, 1450, 1379, 1035 (NH_2). ^1H NMR spectrum ($CDCl_3$), δ, ppm: 0.88 t (3H, Me, *J* 5.7 Hz), 1.18–1.52 m, (6H, $H^{2,4,5}$), 1.71 br.s (4H, NH_2), 2.58–2.86 m (3H, $H^{1,3}$). ^{13}C NMR spectrum ($CDCl_3$), δ, ppm: 13.83 (Me), 18.88 (C^5), 38.84 (C^4), 40.23 (C^2), 40.49 (C^1), 49.40 (C^3). Mass spectrum (Chemical ionization): *m/z* 117 $[M + H]^+$. Mass spectrum: *m/z* (I_{rel}, %): 99 (17) $[M - NH_3]^+$, 86 (9) $[M - NH_2 - CH_2]^+$, 84 (12) $[M - NH_3 - Me]^+$, 73 (10) $[M - Pr]^+$, 72 (42) $[M - NH_2 - CH_2 - CH_2]^+$, 70 (13) $[M - NH_3 - Et]^+$, 56 (36), 44 (98), 42 (13). M^+ 116.0953. $C_6H_{16}N_2$. *M* 116.2047.

1,3-Diamino-4-methylpentane (Vc). From 1 g (0.009 mol) of pyrazoline **Ic** was obtained 0.99 g (95%) of diamine **Vc**. IR spectrum, ν, cm^{-1}: 3358–3197, 2936–2870, 1597, 1465, 1384, 1367, 1041, 848 (NH_2). ^1H NMR spectrum ($CDCl_3$), δ, ppm: 0.77 d (3H, Me, *J* 6.8 Hz), 0.81 d (3H, Me, *J* 6.8 Hz), 1.15–1.35 m (1H, H^4), 1.37–1.57 m (2H, H^2), 1.7 br.s (4H, NH_2), 2.41–2.55 m (1H, H^3), 2.65–2.87 m (2H, H^1). ^{13}C NMR spectrum ($CDCl_3$), δ, ppm: 16.60 (Me), 16.68 (Me), 33.50 (C^4), 37.64 (C^2), 39.56 (C^1), 54.47 (C^3). Mass spectrum (Chemical ionization): *m/z* 117 $[M + H]^+$. Mass spectrum, *m/z* (I_{rel}, %):

99 (17) [M – NH_3]$^+$, 84 (12) [M – NH_3 – Me]$^+$, 73 (10) [M – i-Pr]$^+$, 72 (42) [M – NH_2 – CH_2 – CH_2]$^+$, 56 (24), 44 (100). M^+ 116.0943. $C_6H_{16}N_2$. M 116.2047.

1,3-Diamino-3,4-dimethylpentane (Vd). From 1 g (0.008 mol) of pyrazoline **Id** was obtained 1.04 g (99%) of diamine **Vd**. IR spectrum, ν, cm^{-1}: 3340–3163, 3026–2816, 1598, 1452, 1384, 1375, 1039, 906 (NH_2). ^{1}H NMR spectrum ($CDCl_3$), δ, ppm: 0.83 d (3H, Me, J 6.9 Hz), 0.84 d (3H, Me, J 6.9 Hz), 0.95 s (3H, 5-Me), 1.48 t (2H, H^2, J 7.9 Hz), 1.50 m (1H, H^4), 2.50 br.s (4H, NH_2), 2.72 t (1H, H^1, J 7.5 Hz), 2.73 t (1H, H^1, J 7.9 Hz). ^{13}C NMR spectrum ($CDCl_3$), δ, ppm: 16.46 (Me), 16.89 (Me), 24.03 (C^5), 36.79 (C^4), 36.95 (C^2), 42.39 (C^1), 53.08 (C^3). Mass spectrum (Chemical ionization): *m/z* 131 [M + H]$^+$. Mass spectrum, *m/z* (I_{rel}, %): 113 (2) [M – NH_3]$^+$, 98 (2) [M – NH_3 – Me]$^+$, 87 (25) [M – i-Pr]$^+$, 86 (34) [M – NH_2 – CH_2 – CH_2]$^+$, 70 (17), 58 (100), 44 (12), 43 (13), 41 (9), 29 (29). M^+ 130.1465. $C_7H_{18}N_2$. M 130.2313.

Hydrogenation of 3-methyl-5-(2-furyl)-4,5-dihydro-1*H*-pyrazole (II). From 1 g (0.007 mol) of pyrazoline **II** in the presence of Ni-Ra prepared from 0.16 g of nickel-aluminum alloy was obtained 0.81 g of a mixture of compounds **VI** and **VII** in the yields 9 and 68% respectively.

1,3-Diamino-1-(2-furyl)butane (VI). IR spectrum, ν, cm^{-1}: 3354–3277, 2928–2868, 1589, 1064, 921 (NH_2). ^{1}H NMR spectrum ($CDCl_3$), δ, ppm: 1.19 d (3H, Me, J 6.2 Hz), 1.43 br.s (4H, NH_2), 1.8–2.0 m (2H, H^2), 2.72–2.80 m (1H, H^3), 4.03 t (1H, H^1, J 7.7 Hz), 6.13 m (1H, H$^{3'}$), 6.33 m (1H, H$^{4'}$), 7.35 m (1H, H$^{5'}$). ^{13}C NMR spectrum ($CDCl^3$), δ, ppm: 24.92 (Me), 45.98 (C^2), 45.05 (C^3), 50.66 (C^1), 103.69 (C$^{3'}$), 109.73 (C$^{4'}$) 140.95 (C$^{5'}$), 159.15 (C$^{2'}$). Mass spectrum (Chemical ionization): *m/z* 155 [M + H]$^+$. M^+ 154.2095. $C_8H_{14}N_2O$. M 154.2096.

1,3-Diamino-1-(2-tetrahydrofuryl)butane (VII). A mixture of two diastereomers in the ratio 1:1.5 differing in the position of signals in the ^{13}C NMR spectrum. IR spectrum, ν, cm^{-1}: 3354–3277, 2928–2868, 1589, 1064, 921 (NH_2). ^{1}H NMR spectrum ($CDCl_3$), δ, ppm: 1.19 d (6H, Me, J 6.2 Hz), 1.20–1.80 m (8H, H$^{3',4'}$), 1.43 br.s (8H, NH_2), 1.88 d.d (4H, H^2, J 6.0, J 6.4 Hz), 2.61–2.74 m (1H, H^3, minor diastereomer), 2.91–3.05 m (1H, H^3, main diastereomer), 3.05–3.16 m (2H, H^1), 3.52–3.61 m (1H, H$^{5'}$, minor diastereomer), 3.62–3.91 m (5H, H$^{2',5'}$). ^{13}C NMR spectrum ($CDCl_3$), δ, ppm, minor diastereomer: 24.14 (Me), 25.90 (C$^{3'}$), 28.16 (C$^{4'}$), 43.55 (C^2), 45.26 (C^3), 54.04 (C^1), 67.64 (C$^{5'}$), 83.99 (C$^{2'}$); main diastereomer: 24.54 (Me), 25.62 (C$^{3'}$), 28.16 (C$^{4'}$), 43.41 (C^2), 45.66 (C^3), 52.33 (C^1), 68.02 (C$^{5'}$), 83.21 (C$^{2'}$). Mass spectrum (Chemical ionization): *m/z* 159 [M + H]$^+$. Mass spectrum, *m/z* (I_{rel}, %): 141 (1) [M – NH_3]$^+$, 115 (3) [M – NH_2=$CHCH_3$]$^+$, 100 (3) [M – NH_2=$CHCH_3$ – Me]$^+$, 87 (25) [M – O=$CHCH_2CH(CH_3)NH_2$]$^+$, 70 (7), 44 (100). M^+ 158.2265. $C_8H_{18}N_2O$. M 158.2414.

3-Amino-2-methyl-4-(2-furyl)pyrrolodine (VIII). From 513 mg (0.0028 mol) of pyrazoline **III** in the presence of Ni-Ra prepared from 0.08 g of nickel-aluminum alloy was obtained 389 mg (84%) of pyrrolodine **VIII**. IR spectrum, ν, cm^{-1}: 3354–3115, 2962–2873, 1589, 1509, 1452, 1375, 1010, 734 (NH_2). ^{1}H NMR spectrum ($CDCl_3$), δ, ppm: 1.26 d (3H, Me, J 6.0 Hz), 1.72 br.s (3H, NH_2, NH), 2.76–2.90 m (2H, H^5, H^4), 3.01 m (1H, H^2), 3.16 d.d (1H, H^5, J 7.4, J 11.3 Hz), 3.31 d.d (1H, H^3, J 9.9, J 10.2 Hz), 6.08 d (1H, H$^{3'}$, J 2.7 Hz), 6.29 t (1H, H$^{4'}$, J 1.8, J 2.7 Hz), 7.33 d (1H, H$^{5'}$, J 1.8 Hz). ^{13}C NMR spectrum ($CDCl_3$), δ, ppm: 18.54 (Me), 49.10 (C^5), 50.23 (C^4), 62.13 (C^2), 65.13 (C^3), 104.99 (C$^{3'}$), 110.00 (C$^{4'}$), 141.53 (C$^{5'}$), 155.74 (C$^{2'}$). Mass spectrum (Chemical ionization): *m/z* 167 [M + H]$^+$. Mass spectrum, *m/z* (I_{rel}, %): 166 (16) [M]$^+$, 149 (46) [M – NH_3]$^+$, 121 (11), 110 (24) [C_7H_8NO]$^+$, 94 (70) [C_6H_6O]$^+$, 80 (10), 57 (100) [C_3H_7N]$^+$, 56 (25), 44 (10), 39 (6). M^+ 166.1103. $C_9H_{14}N_2O$. Calculated M 166.2203.

1,3,4,6-Tetraaminohexane (IX). From 1 g (0.007 mol) of pyrazoline **IV** in the presence of Ni-Ra, prepared from 0.22 g of nickel-aluminum alloy was obtained 1.02 g (99%) of tetraamine **IX** as a mixture of two diastereomers in the ratio 1 : 1.2 differing in the position of signals in the ^{13}C NMR spectrum. IR spectrum, ν, cm^{-1}: 3356–3275, 2926–2868, 1585, 1480, 921 (NH_2). ^{1}H NMR spectrum ($CDCl_3$), δ, ppm: 1.25–1.80 m (12H, NH_2, H^{2,5}), 2.70–3.00 m (6H, H^{1,3,4,6}). ^{13}C NMR spectrum ($CDCl_3$), δ, ppm, minor diastereomer: 37.54 (C^2), 39.26 (C^1), 39.41 (C^{1b}) 54.28 (C^3); main diastereomer: 35.48 (C^2), 39.41 (C^1), 53.59 (C^3). Mass spectrum (XAND): *m/z* 147 [M + H]$^+$. $C_6H_{18}N_4$. Calculated M 146.2341.

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able applied properties").

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