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Reaction of Tetracyanocyclopropyl Ketones with Hydrazine Hydrate

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Abstract—The reaction of 2,2,3,3-tetracyanocyclopropyl ketones with hydrazine involved the addition at the carbonyl and cyano groups and resulted in the formation of previously unknown polycyclic systems whose common feature was the presence in the structure of a pyridazine ring. In the general case 3-substituted 6-amino-8-oxo-4,5,7-tptriazatricyclo[4.3.0.0^{2,9}]-non-3-ene-1,9-dicarbonitriles were obtained. The reaction with 3-pivaloylcyclopropane-1,1,2,2-tetracarbonitrile took another route with the opening of the three-membered ring and the formation of 3-amino-7a-*tert*-butyl-6-oxo-5,6,7,7a-tetrahydro-4a*H*-pyrrolo[2,3-*c*]pyridazine-4,5-dicarbonitrile.

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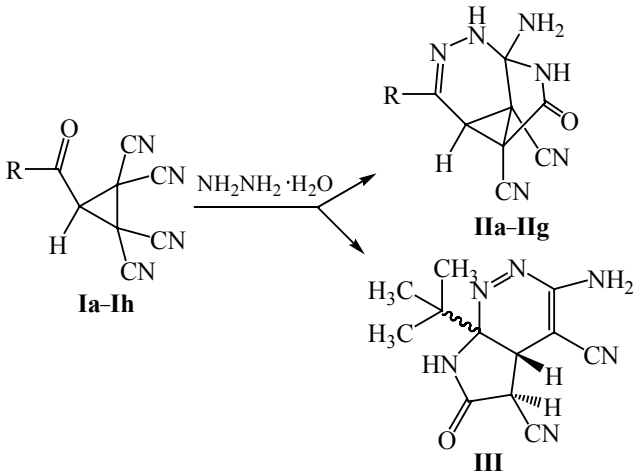
The most important methods of heterocycles preparation containing nitrogen–nitrogen bonds are founded on the reactions of hydrazine with bielectrophilic reagents (1,3- for the synthesis of pyrazole derivatives and 1,4- for pyridazine) [1–3]. The promising for these syntheses bielectrophilic substances are 2,2,3,3-tetracyanocyclopropyl ketones [4, 5] containing five electron-withdrawing groups of various types. These compounds are highly reactive toward nucleophiles [6–8] and can be brought into reactions both like 1,3-bielectrophiles involving the geminal groups and like 1,4-bielectrophiles involving the vicinal groups. Bringing them into the reaction with hydrazine may open the way to preparation of new nitrogen polyfunctional heterocyclic systems as prove the publications on the hydrazine reaction with tetraacetylcyclopropane [9] and on phenylhydrazine with tetracyanocyclopropanes [10]. In both cases the nucleophilic attack is directed on the geminal substituents in the cyclopropane ring followed by its recyclization into pyrazole derivatives.

Here the results are reported on the investigation of the reaction of tetracyanocyclopropyl ketones **Ia–Ih** with hydrazine hydrate. Two possible directions of transformations were discovered. The main pathway characteristic of aryl, heteryl, and alkyl derivatives led to the formation of 3-substituted 6-amino-8-oxo-4,5,7-triazatricyclo[4.3.0.0^{2,9}]non-3-ene-1,9-dicarbonitriles **IIa–IIg** (Scheme 1). Into the reaction a carbonyl and two cyano groups are involved retaining the three-membered

Scheme 1.



R = Ph (**a**), 4-BrC_6H_4 (**b**), 4-$MeOC_6H_4$ (**c**), 3,4-$(MeO)_2C_6H_3$ (**d**), 3-$NO_2C_6H_4$ (**e**), thien-2-yl (**f**), Me (**g**), $(CH_3)_3C$ (**h**).

ring intact. The alternative reaction pathway was observed for *tert*-butyl cyclopropyl ketone **Ih**: The three-membered ring suffered opening, and the process resulted in the formation of diastereomers of 3-amino-7a-*tert*-butyl-6-oxo-5,6,7,7a-tetrahydro-4a*H*-pyrrolo[2,3-*c*]pyridazine-4,5-dicarbonitrile (**III**).

The data of mass spectra and elemental analysis of compounds **IIa–IIg** show that in the course of the reaction one hydrazine molecule has added. The peaks of molecular ions in the mass spectra are of low intensity. The fragmentation proceeds similarly with the splitting of cations $[NH=C=O]^+$, *m/z* 43, and (or) $[NH_2=C=O]^+$, *m/z* 44, having the strongest peaks, and also with the elimination of the nitrogen molecule, therefore in the mass spectra of all compounds **IIa–IIg** a moderately intense peak is present $[M-71]^+$. According to the ^1H and ^{13}C spectra of compounds **IIa–IIg** the cyclopropane ring is not affected by the reaction. It is proved by the signal of the protons of the cyclopropane ring (4.0–4.5 ppm) in the ^1H NMR spectra and of signals of the carbon atoms of the three-membered ring (27.84–29.29 ppm) in the ^{13}C NMR spectra. Besides in the ^1H NMR spectra a signal present at 3.47–3.50 ppm belongs to the protons of a nonconjugated amino group. The assumed assignment of the signals in the ^{13}C NMR spectra of compounds **IIa** and **IIc** is presented in the table [11].

The final structure was suggested proceeding from the 2D HMBC and NOESY spectra of compound **IIa**. In the HMBC spectrum on the line of the chemical shift of the proton of the cyclopropane ring (4.23 ppm) nine correlation peaks were observed (Fig. 1): at the coordinates of atoms C^4, C^5, and C^6 of the cyclopropane ring (27.84, 29.29, 28.55 ppm), C^7 and C^8 of cyano groups (114.04, 113.69 ppm), C^2, C^3 of pyrrole (79.28, 162.63 ppm) and C^1 of pyridazine ring (138.30 ppm), at the coordinates of the atom C^9 of the benzene ring (135.37 ppm).

The chemical shift of the proton at the nitrogen of pyrrole (8.64 ppm) correlates with the shifts of all carbon atoms of the pyrrole ring: C^2 (79.28 ppm), C^3 (162.63 ppm), C^4 (27.84 ppm), C^6 (28.55 ppm). On the line of the chemical shift of the proton at the nitrogen of the pyridazine ring (8.29 ppm) three correlation peaks appear: at the coordinates of atoms C^1 (138.30 ppm), C^2 (79.28 ppm), C^6 (28.55 ppm). The shift of the protons of amino group (3.47 ppm) correlates with the shifts of atoms C^2 (79.28 ppm) and C^6 (28.55 ppm) (Fig. 2).

Besides in the HMBC spectrum correlation peaks were observed at coordinates of chemical shifts of carbon

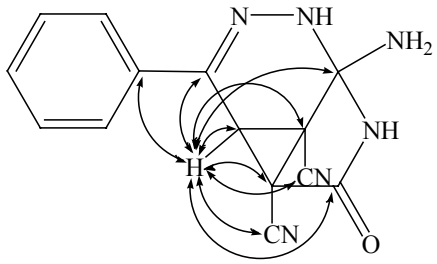


Fig. 1. Correlations of the cyclopropane proton with carbon atoms according to the HMBC NMR spectrum of compound **IIa**.

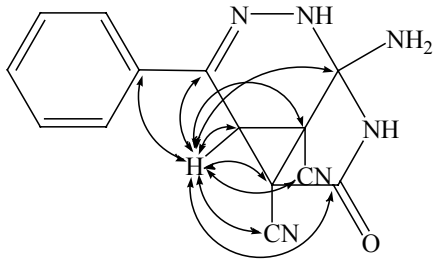
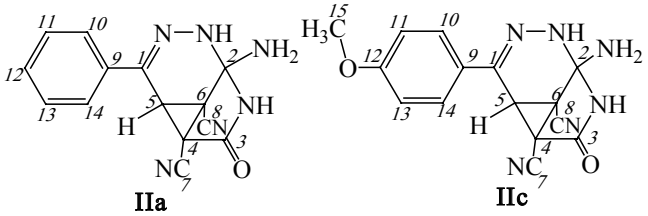


Fig. 2. Correlation of NH and *ortho*-protons of phenyl ring with carbon atoms according to the HMBC NMR spectrum of compound **IIa**.

Data of ^{13}C NMR spectra of compounds **IIa** and **IIc**



Number of carbon atom	Chemical shifts, δ, ppm	
	IIa	**IIc**
C^3	162.63	162.55
C^1	138.30	138.50
C^9	135.37	128.08
C^{10}, C^{11}, C^{12},	129.08, 128.79, 124.08	160.13, 125.63,
C^7, C^8	114.04, 113.69	114.04, 113.69
C^2	79.28	79.31
C^5	29.29	29.24
C^6	28.55	28.84
C^4	27.84	27.93
C^{15}		55.38

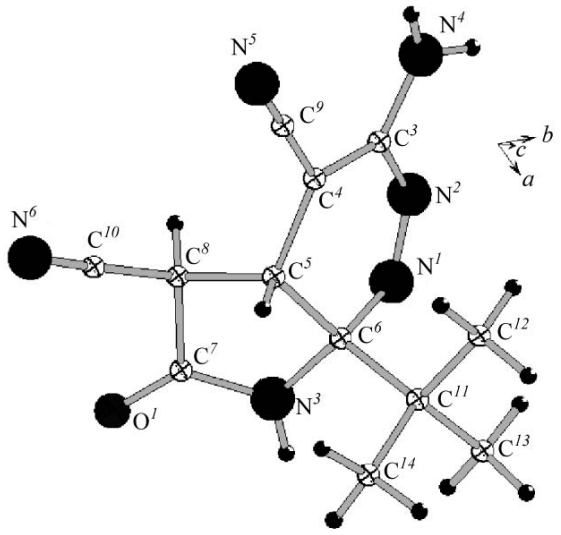


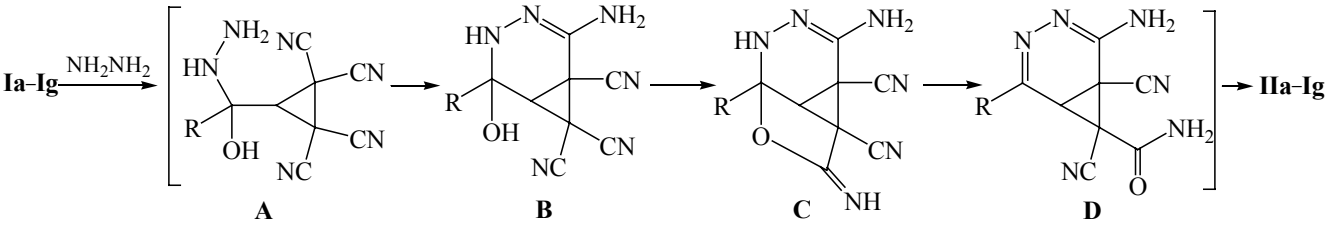
Fig. 3. Molecular structure of (4a*S**,5*S**,7a*R**)-3-amino-7a-*tert*-butyl-6-oxo-5,6,7,7a-tetrahydro-4a*H*-pyrrolo[2,3-*c*]-pyridazine-4,5-dicarbonitrile (**III**) according to XRD analysis.

atoms and protons of the benzene ring. The most informative among them for establishing the final structure was the correlation peak on the line of the chemical shifts of the *ortho*-protons of the benzene ring (8.64 ppm) and atom C^1 (138.30 ppm). The NOESY spectrum contained the correlation peaks at the coordinates of the chemical shifts of the protons at the nitrogen toms of the pyrrole and pyridazine ring and of amino group, and also of the protons of the benzene ring.

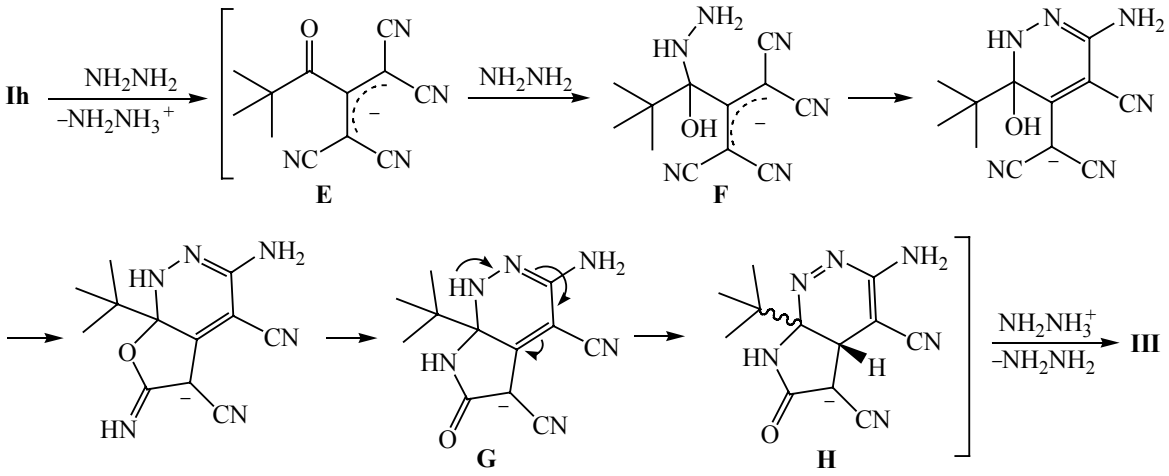
According to ^{1}H NMR spectrum the product of the reaction of cyclopropane **Ih** with hydrazine is not an individual compound but a mixture of two diastereomers in the retio 1:4. The *tert*-butyl group gives rise to a singlet at 0.90 ppm and all other signals are doubled. The structure of one diastereomer was established by XRD method (Fig. 3). The hydrogen atoms of the pyrrole ring appear as doublets with the coupling constant *J* 9.7 Hz for both diastereomers indicating that in both cases the protons are in the *trans*-position with respect to each other, and the isomerism is due to the different position of the *tert*-butyl substituent. IR spectra of compounds **IIa–IIg** and **III** are in agreement with the assumed structures.

As was previously established the cyclopropane ring of compounds **Ia–Ih** suffered opening under the treatment with various bases, in particular, with acetate ion (pK_B 9.3). Hydrazine (pK_B 6.2) is a stronger base and also should effect the opening of the three-membered ring of cyclopropanes **Ia–Ih**. However, as seen from the structure of compounds **IIa–IIg** the majority of initial compounds **Ia–Ig** do not demonstrate the CH-acid properties in the reaction with hydrazine and consequently the cyclopropane ring is not opened. It evidently occurs in this way because the competing addition to the carbonyl group prevails followed by the cyclization with the cyano group and the formation of pyridazine derivative **B** (Scheme 2). The assumption of the primary addition to the cyano group does not explain the regioselectivity of the addition to cyano group in the *cis*-position with respect to the carbonyl. Moreover, when the reaction occurs only at the cyano groups (with ketone oximes), the attack of the nucleophile is in contrast directed to the nitrile group in the trans-position to the carbonyl [7]. Water elimination may occur as well as from intermediate **A** and from **B**, but in any case with the assistance of the cyano group which is thus converted into the carboxamide group. The reaction is completed by the addition of the amide group to the amidine fragment of intermediate **D**. Similar conversions are usual for the polycyano substances where the hydroxy and nitrile groupd are spatially close. It results as a rule in the formation of 2-iminofurane fragment and when therewith the oxygen atom of the furan ring is adjacent to the atom bearing the proton-containing nitrogen heteroatom (as in intermediate **C**) always further recyclization occurs into a pyrrole ring [12-15]. Yet if this recyclization is initiated by a reagent of basic character, in some cases decyclization is observed and the subsequent cyclization at the vicinal cyano group [16, 17], namely, the process is analogous to the path we suggested for the conversion

Scheme 2.



Scheme 3.



of intermediate **C** into final compound **II**.

The formation of bicyclic intermediates and final compound **II** prevents the occurrence of the electrocyclic opening of the cyclopropane ring.

In cyclopropane **Ih** the *tert*-butyl substituent due to the steric hindrance prevents the approach of the nucleophile to the carbonyl group, and thus the reaction proceeds by the competing route starting with the deprotonation followed by the electrocyclic opening of the ring (Scheme 3). At the formation of intermediate anion **E** the initial steric hindrances are partially removed thus providing the possibility for the reaction between the carbonyl group and hydrazine with the formation of intermediate **F**. Further in th course of successive series of heterocyclization arises intermediate **G**. The subsequent 1,5-sigmatropic shift of the proton may occur both in the *cis*- and *trans*-position with respect to the *tert*-butyl group leading to the formation of diastereomeric intermediates **H** whose protonation gives final compound **III**.

Compound **III** can exist as three diastereomers (Fig. 4), and the formation in the reaction of only two may be due to the configurational stability of the proton in the place of joining the pyrrole and pyridazine ring; evidently, the proton conserves the position it acquired in the 1,5-prototropic shift. The hydrogen atom at the cyano group is on the contrary labile and in the course of the reversible deprotonation gets the most feasible *trans*-position.

EXPERIMENTAL

The reaction completeness and purity of compounds synthesized was monitored by TLC on Silufol UV-254 plates, detection of spots by UV irradiation, iodine vapor, or thermal degradation. IR spectra were recorded on a Fourier spectrophotometer FSM-1202 from mulls in mineral oil. ^{1}H and ^{13}C NMR spectra were registered on a spectrometer Bruker DRX-500 at operating frequencies 500.13 and 125 MHz, solvent DMSO-d_6. Mass spectra were taken on an instrument Finnigan MAT INCOS 50 (electron impact, 70 eV). Elemental analysis was carried out on an analyzer Laboratorni Pristroje, Praha. The parameters of the unit cell of molecule **III** and the intensity of the reflections for the XRD analysis were measured on a diffractometer Enraf-Nonius CAD-4. The structure was solved by the direct method using program package SHELX [18]. The positions and thermal parameters of nonhydrogen atoms were refined in the anisotropic approximation, of the hydrogen atoms, in the isotropic approximation. The molecular graphic was performed applying the DIAMOND program [19]. Crystallographic

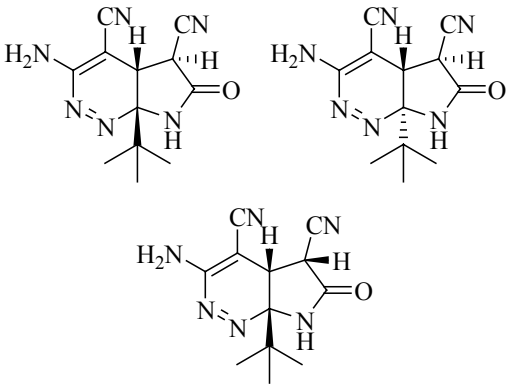


Fig. 4. Diastereomes of compound **III**.

data of compound **III** are deposited into the Cambridge Crystallographic Data Center (CCDC deposition no. 767063) and may be obtained free at CCDC, 12 Union Road, Cambridge, CB2 1EZ, UK (e-mail: deposit@ccdc.cam.ac.uk, http://www.ccdc.cam.ac.uk).

6-Amino-8-oxo-3-phenyl-4,5,7-triazatricyclo[4.3.0.0^{2,9}]non-3-ene-1,9-dicarbonitrile (IIa). In 10 ml of acetonitrile was dissolved 2.46 g (0.01 mol) of cyclopropane **Ia**, and to the solution was added at stirring 0.50 g of 99% water solution of hydrazine hydrate. The precipitate formed in 15 min was filtered off, washed with acetonitrile and water, and recrystallized from acetonitrile. Yield 2.03 g (73%), mp 196–197°C (decomp.). IR spectrum, ν, cm^{-1}: 3437 (NH), 3378 (NH), 3307, 3286 (NH_2), 3028 (C–H), 2257 (C≡N), 1744 (C=O), 1706 (C=N). ^{1}H NMR spectrum, δ, ppm: 3.47 s (2H, NH_2), 4.23 s (1H, C–H), 7.67 d (2H, C_6H_5, ^3J 7.3 Hz), 7.47 t (2H, C_6H_5, ^3J 7.8 Hz), 7.40 t (1H, C_6H_5, ^3J 7.2 Hz), 8.29 s (1H, NH), 8.64 s (1H, NH). Mass spectrum: *m/z* 278. Found, %: C 60.37; H 3.64; N 30.15. $C_{14}H_{10}N_6O$. Calculated, %: C 60.43; H 3.62; N 30.20. *M* 278.09

Compounds **IIb–IIg** were obtained similarly.

6-Amino-3-(4-bromophenyl)-8-oxo-4,5,7-triazatricyclo[4.3.0.0^{2,9}]non-3-ene-1,9-dicarbonitrile (IIb). Yield 2.78 g (78%), mp 127–128°C (decomp.) (acetonitrile). IR spectrum, δ, cm^{-1}: 3414 (NH), 3377 (NH), 3325, 3311 (NH_2), 3024 (CH, cyclopropane), 2254 (C≡N), 1747 (C=O), 1709 (C=N). ^{1}H NMR spectrum, δ, ppm: 3.50 s (2H, NH_2), 4.22 s (1H, CH), 7.58 d (2H, 4-BrC_6H_4, ^3J 8.8 Hz), 7.66 d (2H, 4-BrC_6H_4, ^3J 8.8 Hz), 8.45 c (1H, NH), 8.68 c (1H, NH). Mass spectrum: *m/z* 358, 356. Found, %: C 47.00; H 2.61; N 23.52. $C_{14}H_9BrN_6O$. Calculated, %: C 47.08; H 2.54; N 23.53. *M* 356.00.

6-Amino-3-(4-methoxyphenyl)-8-oxo-4,5,7-triazatricyclo[4.3.0.0^{2,9}]non-3-ene-1,9-dicarbonitrile (IIc). Yield 2.13 g (69%), mp 194–195°C (decomp.) (acetonitrile). IR spectrum, δ, cm^{-1}: 3380 (NH), 3323, 3313 (NH_2), 3028 (CH, cyclopropane), 2258 (C≡N), 1743 (C=O), 1706 (C=N). ^{1}H NMR spectrum, δ, ppm: 3.44 s (2H, NH_2), 3.79 s (3H, CH_3O), 4.20 s (1H, CH), 7.03 d (2H, 4-$CH_3OC_6H_4$, ^3J 8.9 Hz), 7.59 d (2H, 4-$CH_3OC_6H_4$, ^3J 8.9 Hz), 8.03 s (1H, NH), 8.59 s (1H, NH). Mass spectrum: *m/z* 308. Found, %: C 58.34; H 3.91; N 27.29. $C_{15}H_{12}N_6O_2$. Calculated, %: C 58.44; H 3.92; N 27.26. *M* 308.10.

6-Amino-3-(3,4-dimethoxyphenyl)-8-oxo-4,5,7-triazatricyclo[4.3.0.0^{2,9}]non-3-ene-1,9-dicarbonitrile (IId). Yield 2.77 g (82%), mp 190–191°C (decomp.) (acetonitrile). IR spectrum, δ, cm^{-1}: 3422 (NH), 3417 (NH), 3356, 3345 (NH_2), 3024 (CH, cyclopropane), 2259 (C≡N), 1737 (C=O), 1711 (C=N). ^{1}H NMR spectrum, δ, ppm: 3.49 s (2H, NH_2), 3.77 s (3H, OCH_3), 3.80 s (3H, OCH_3), 4.26 s (1H, CH), 7.06 d (1H, C_6H_3, ^3J 8.5 Hz), 7.13 d.d (1H, C_6H_3, ^3J 8.4, ^4J 2.0 Hz), 7.29 d (1H, C_6H_3, ^4J 2.0 Hz), 8.07 s (1H, NH), 8.61 s (1H, NH). Mass spectrum: *m/z* 339 $[M + H]^+$, 163 $[(CH_3O)_2C_6H_4C{=}N]^+$. Found, %: C 56.83; H 4.15; N 24.80. $C_{16}H_{14}N_6O_3$. Calculated, %: C 56.80; H 4.17; N 24.84. *M* 338.11

6-Amino-3-(3-nitrophenyl)-8-oxo-4,5,7-triazatricyclo[4.3.0.0^{2,9}]non-3-ene-1,9-dicarbonitrile (IIe). Yield 2.49 g (77%), mp 192–193°C (decomp.) (ацетонтрил). IR spectrum, δ, cm^{-1}: 3437 (NH), 3404 (NH), 3346, 3327 (NH_2), 3029 (CH, cyclopropane), 2254 (C≡N), 1727 (C=O), 1712 (C=N). ^{1}H NMR spectrum, δ, ppm: 3.57 s (2H, NH_2), 4.42 s (1H, CH), 7.78 t (1H, 3-$NO_2C_6H_4$, ^3J 8.0 Hz), 8.06 d (1H, 3-$NO_2C_6H_4$, ^3J 8.0 Hz), 8.24 d.d (1H, 3-$NO_2C_6H_4$, ^3J 8.3, ^4J 2.1 Hz), 8.46 s (1H, 3-$NO_2C_6H_4$), 8.74 s (1H, NH), 8.77 s (1H, NH). Mass spectrum: *m/z* 223. Found, %: C 51.93; H 2.69; N 30.28. $C_{14}H_9N_7O_3$. Calculated, %: C 52.02; H 2.81; N 30.30. *M* 323.08

6-Amino-8-oxo-3-(2-thienyl)-4,5,7-triazatricyclo[4.3.0.0^{2,9}]non-3-ene-1,9-dicarbonitrile (IIf). Yield 1.99 g (70%), mp 149–150°C (decomp.) (acetonitrile). IR spectrum, δ, cm^{-1}: 3439 (NH), 3360 (NH), 3310, 3304 (NH_2), 3027 (CH, cyclopropane), 2253 (C≡N), 1726 (C=O), 1709 (C=N). ^{1}H NMR spectrum, δ, ppm: 3.48 s (2H, NH_2), 4.32 s (1H, CH), 7.12 d.d (1H, C_4H_3S, ^3J 5.1, ^4J 3.7 Hz), 7.34 d.d (1H, C_4H_3S, ^3J 3.7, ^4J 1.1 Hz), 7.54 d.d (1H, C_4H_3S, ^3J 5.1, ^4J 1.2 Hz), 8.18 s (1H, NH), 8.68 s (1H, NH). Mass spectrum: *m/z* 284. Found, %: C 50.65; H 2.86; N 29.56. $C_{12}H_8N_6OS$. Calculated, %: C 50.70; H 2.84; N 29.56. *M* 284.05

6-Amino-3-methyl-8-oxo-4,5,7-triazatricyclo[4.3.0.0^{2,9}]non-3-ene-1,9-dicarbonitrile (IIg). Yield 1.40 g (65%), mp 218–219°C (decomp.) (acetonitrile). IR spectrum, ν, cm^{-1}: 3440 (NH), 3418 (NH), 3353, 3342 (NH_2), 3024 (CH, cyclopropane), 2251 (C≡N), 1730 (C=O), 1710 (C=N). ^{1}H NMR spectrum, δ, ppm: 1.96 s (3H, CH_3), 3.40 s (2H, NH_2), 3.56 s (1H, CH), 7.41 s (1H, NH), 8.50 s (1H, NH). Mass spectrum: *m/z* 216. Found, %: C 50.05; H 3.76; N 38.89. $C_9H_8N_6O$. Calculated, %: C 50.00; H 3.73; N 38.87. *M* 216.08.

3-Amino-7a-*tert*-butyl-6-oxo-5,6,7,7a-tetrahydro-4a*H*-pyrrolo[2,3-*c*]pyridazine-4,5-dicarbonitrile (III),

diastereomers mixture. In 10 ml of acetonitrile was dissolved 2.26 g (0.01 mol) of cyclopropane **Ih**, and into the solution was added at stirring 0.50 g of 99% water solution of hydrazine hydrate. After 24 h the solvent was evaporated, the residue was diluted with 20 ml of water. The solid compound was ground, filtered off, washed with water and 2-propanol, and recrystallized from a mixture dioxane–2-propanol, 1:1. Yield 1.52 g (59%), mp 236–237°C (decomp.). IR spectrum, ν, cm^{-1}: 3441 (NH), 3338, 3250 (NH_2), 2256 (C≡N), 2197 (C≡N), 1717 (C=O). ^{1}H NMR spectrum, δ, ppm: 0.90 s [9H, $C(CH_3)_3$], 3.36 d [3.48 d] (1H, CH, ^3J 9.7 [^3J 9.7] Hz), 4.20 d [4.26 d], (1H, CH, ^3J 9.7 [^3J 9.7] Hz), 7.93 s [8.06 s] (2H, NH_2), 10.01 s [10.15 s] (1H, NH). Found, %: C 55.86; H 5.57; N 32.41. $C_{12}H_{14}N_6O$. Calculated, %: C 55.80; H 5.46; N 32.54.

Crystallographic parameters of compound **III**: *a* 8.3223(9), *b* 8.6959(5), *c* 10.6443(6) A, α 71.605(5), β 72.104(8), γ 72.104(8)°, *V* 643.96(9) A^3, *Z* 2. Overall number of reflections 2566. Number of reflections with $I > 2\sigma(I)$ 2064. Number of refined parameters 229. Factor of uncertainty *R* 0.0433.

ACKNOWLEDGMENTS

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