Synthesis of pyrazole- and thiazole-annulated 3-R-1,5-dinitro-3-azabicyclo[3.3.1]nonanes

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The previously unknown 3-R-1,5-dinitro-3-azabicyclo[3.3.1]nonanes fused to the pyrazole or thiazole rings were synthesized by the reductive cyclization of *m*-dinitroindazoles and benzothiazoles. The method is based on the reduction of carbon—carbon bonds in the benzene ring, which are activated by the *meta*-nitro groups, with $NaBH_4$ followed by the Mannich reaction with formaldehyde and primary amines.

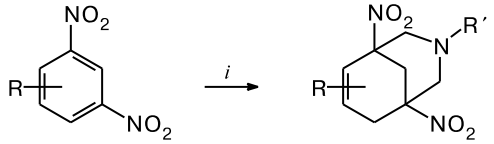
Key words: azabicyclo[3.3.1]nonanes, reduction, Mannich reaction, nitro group, azoles.

The present study is a continuation of our works to develop methods for the synthesis of polycyclic fused heterosystems based on five-membered aromatic 4,6-dinitrobenzannulated heterocycles employing the ability of the starting dinitro compounds to form σ^H-adducts with nucleophilic reagents.[1] The purpose of the present work was to synthesize new 3-R-1,5-dinitro-3-azabicyclo-[3.3.1]nonanes fused to substituted pyrazoles and thiazoles at the C(7)—C(8) bond.

Heteroanalogs of bicyclo[3.3.1]nonanes are structural fragments of terpenoid alkaloids and are used as biologically active compounds.[2] 1,5-Dinitro-3-azabicyclo[3.3.1]-nonane derivatives were found to have high antiarrhythmic activity (see, for example, Ref. 3). Many of these compounds have been synthesized previously[3—6] based on substituted 1,3-dinitrobenzenes (Scheme 1). The method for their synthesis involves the reduction of carbon—carbon bonds in the benzene ring, which are activated by the *meta*-nitro groups, with $NaBH_4$ or KBH_4 followed by the double Mannich reaction with formaldehyde and primary amines. However, similar transformations of *m*-dinitrobenzannulated heterocycles are poorly known. Thus, except for our studies (see below), a few examples of the synthesis of such compounds containing the pyridine ring were reported.[6,7]

According to preliminary calculations for biological activity with the use of the PASS program (see Ref. 8),* some 1,5-dinitro-3-azabicyclo[3.3.1]nonanes containing the pyrazole or thiazole moiety can have, with high probability, certain useful physiological activities (psychotropic, antihypoxic, antineurotoxic, and so on). It would be expected that compounds containing the C_2—C_3 alkyl or ω-methoxyalkyl substituent at the nitrogen atom of the piperidine ring will exhibit the highest activity. Previously,[9] we have shown that such derivatives containing the *N*-carboxyalkyl group can be, in principle, synthesized. Hence, we continued studies aimed at preparing 3-azabicyclo[3.3.1]nonanes fused to the pyrazole ring. These compounds can be synthesized starting from isomeric 4,6-dinitro-1- and -2-phenylindazoles **1** and **2**, which can be prepared based on trinitrotoluene (TNT).[10,11]

Scheme 1



R = OH, Alk, Hal, OAlk, COOH
R′ = Alk, $(CH_2)_nCOOH$

i. 1) $NaBH_4$, THF—EtOH—$HCONH_2$; 2) HCHO, $R'NH_2$, AcOH.

Thus, the reactions of compounds **1** and **2** with $NaBH_4$ afford reduction products **3** and **4**,[12] whose treatment with a formaldehyde solution and primary amines gives tricyclic derivatives, *viz*., 1,5-dinitro-3-azabicyclo[3.3.1]-nonanes **5** or **6** annulated to the pyrazole ring at the C(7)—C(8) bond (Scheme 2).

Then, we performed the reaction under consideration with the use of 4,6-dinitrobenzothiazoles containing the

* The results of the testing of some compounds synthesized in the present study will be published elsewhere.

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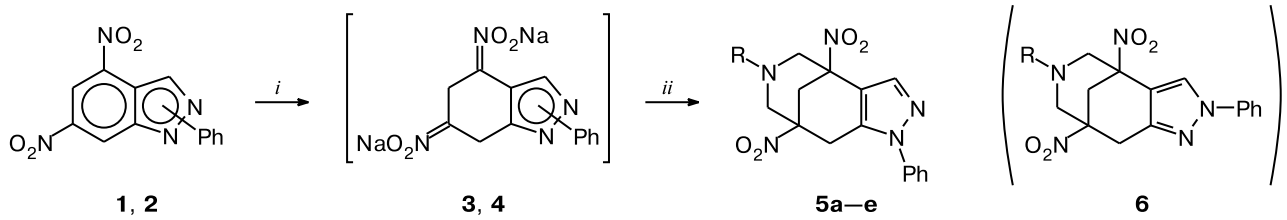
Scheme 2

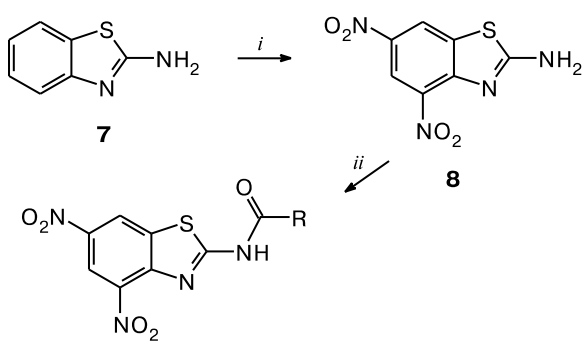


1, **2** **3**, **4** **5a—e** **6**

5: R = CH_2CH_2OH (**a**), CH_2CH_2OMe (**b**), Et (**c**), Pr (**d**), $(CH_2)_3OMe$ (**e**)
6: R = CH_2CH_2OMe

i. $NaBH_4$, EtOH—THF—$HCONH_2$; *ii*. CH_2O, RNH_2, AcOH.

Scheme 3



7 **8** **9** (93%), **10** (91%)

R = Me (**9**), Et (**10**)

i. HNO_3, oleum, 20 °C; *ii*. $(RCO)_2O$, Δ.

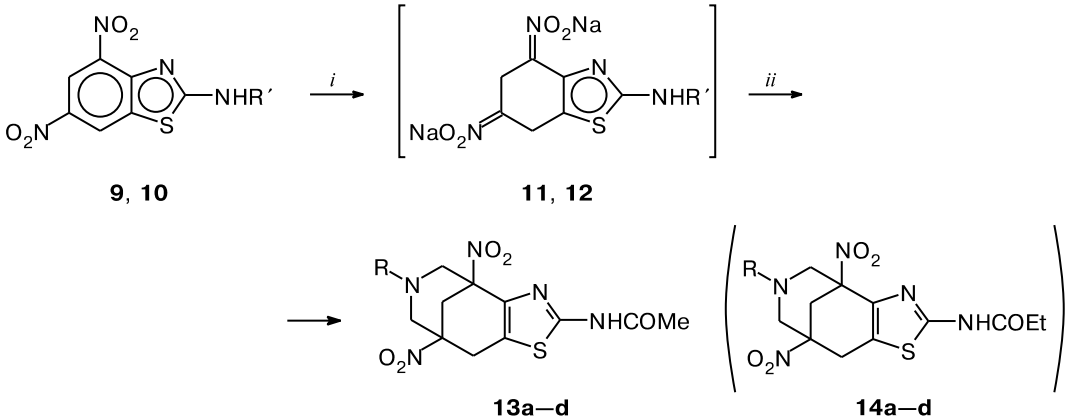
acetylamino or propionylamino substituent at position 2 as the starting compounds. These compounds were synthesized by the nitration of 2-aminobenzothiazole **7** with nitric acid in oleum[13] followed by the acylation of dinitro derivative **8** with acetic or propionic anhydride (Scheme 3).

Dinitrobenzothiazoles **9** and **10** are easily reduced with $NaBH_4$ followed by the Mannich reaction, as was observed for compounds **1** and **2**; in these reactions, a THF—EtOH mixture was used as the solvent. As a result, we synthesized a series of the previously unknown 3-azabicyclo-[3.3.1]nonanes fused to the thiazole ring in good yields (Scheme 4).

The structures of all synthesized compounds were confirmed by ^1H NMR and IR spectroscopy, as well as by elemental analysis.

To summarize, we synthesized a series of new 3-substituted 1,5-dinitro-3-azabicyclo[3.3.1]nonanes fused to

Scheme 4



9, **10** **11**, **12** **13a—d** **14a—d**

Compound	R′	Compound	R	Compound	R	Compound	R
9	COMe	**12**	COEt	**13c**	Et	**14b**	$(CH_2)_3OMe$
10	COEt	**13a**	CH_2COOH	**13d**	$(CH_2)_2OMe$	**14c**	CH_2COOH
11	COMe	**13b**	$(CH_2)_3OMe$	**14a**	Me	**14d**	$(CH_2)_2OMe$

i. $NaBH_4$, EtOH—THF; *ii*. CH_2O, RNH_2, AcOH.

the pyrazole or thiazole ring starting from dinitro derivatives of indazoles and benzothiazoles.

Experimental

The ^{1}H NMR spectra were recorded on a Bruker AM-300 instrument in DMSO-d_6; the chemical shifts are given with respect to Me_4Si. The IR spectra were measured on a Bruker Alpha instrument (KBr pellets). The course of the reactions was monitored and the purity of the compounds was checked by TLC on Silufol UV-254 plates. Compounds **1**,[10] **2**,[11] and **8** (see Ref. 13) were synthesized according to procedures described previously.

Synthesis of compounds 5 and 6 (general procedure). Sodium borohydride (0.43 g, 11.4 mmol) was added portionwise to a solution of compound **1** or **2** (0.57 g, 2 mmol) in a mixture of THF (3 mL), EtOH (8 mL), and formamide (6 mL) at a temperature ≤10 °C for 20 min. After 30 min, water (10 mL), a mixture of a 30% aqueous solution of RNH_2 (2.5 mL) and a 30% formaldehyde solution (2.5 mL), and then glacial AcOH (2.5 mL) were successively added. The reaction mixture was stirred at 20 °C for 30 min and then poured into water (200 mL). The precipitate that formed was filtered off and dried in air.

2-(4,8-Dinitro-1-phenyl-1,4,5,7,8,9-hexahydro-6*H*-4,8-methanopyrazolo[4,3-*d*]azocin-6-yl)ethanol (5a). The yield was 23%, m.p. 103—105 °C. Found (%): C, 54.43; H, 5.27; N, 18.32. $C_{17}H_{19}N_5O_5$. Calculated (%): C, 54.69; H, 5.13; N, 18.76. ^{1}H NMR, δ: 2.44—3.78 (m, 13 H); 7.42—7.61 (m, 6 H, Ph, pyrazole). IR, ν/cm^{-1}: 694, 763, 968, 1066, 1366, 1413, 1455, 1506, 1546, 1598, 1664, 3406.

6-(2-Methoxyethyl)-4,8-dinitro-1-phenyl-4,5,6,7,8,9-hexahydro-1*H*-4,8-methanopyrazolo[4,3-*d*]azocine (5b). The yield was 81%, m.p. 127—129 °C. Found (%): C, 55.96; H, 5.31; N, 17.92. $C_{18}H_{21}N_5O_5$. Calculated (%): C, 55.81; H, 5.46; N, 18.08. ^{1}H NMR, δ: 2.50—3.41 (m, 14 H); 3.79 (d, 1 H, J = 16.3 Hz); 7.42—7.61 (m, 6 H, pyrazole, Ph).

6-Ethyl-4,8-dinitro-1-phenyl-4,5,6,7,8,9-hexahydro-1*H*-4,8-methanopyrazolo[4,3-*d*]azocine (5c). The yield was 49%, m.p. 115—116 °C. Found (%): C, 56.98; H, 5.23; N, 19.43. $C_{17}H_{19}N_5O_4$. Calculated (%): C, 57.14; H, 5.36; N, 19.60. ^{1}H NMR, δ: 0.84 (t, 3 H, Me, J = 7.1 Hz); 2.43—2.59 (m, 3 H); 2.65 (d, 1 H, J = 10.4 Hz); 2.80 (d, 1 H, J = 10.1 Hz); 2.93 (d, 1 H, J = 11.2 Hz); 3.06 (d, 1 H, J = 11.2 Hz); 3.17 (d, 1 H, J = 10 Hz); 3.40 (d, 1 H, J = 16.2 Hz); 3.80 (d, 1 H, J = 16.4 Hz); 7.42—7.62 (m, 6 H, Ph, pyrazole).

4,8-Dinitro-1-phenyl-6-propyl-4,5,6,7,8,9-hexahydro-1*H*-4,8-methanopyrazolo[4,3-*d*]azocine (5d). The yield was 11%, m.p. 103—104 °C. Found (%): C, 58.36; H, 5.58; N, 18.93. $C_{18}H_{21}N_5O_4$. Calculated (%): C, 58.21; H, 5.70; N, 18.86. ^{1}H NMR, δ: 0.50 (t, 3 H, J = 7.2 Hz); 1.21—1.30 (m, 2 H); 2.37 (d, 2 H, J = 2.1 Hz); 2.52 (d, 1 H, J = 11.4 Hz); 2.67 (d, 1 H, J = 10.5 Hz); 2.76 (d, 1 H, J = 10.0 Hz); 2.94 (d, 1 H, J = 11.1 Hz); 3.11 (m, 2 H); 3.33 (d, 1 H, J = 15.3 Hz); 3.80 (d, 1 H, J = 16.3 Hz); 7.43—7.62 (m, 6 H, Ph, pyrazole).

6-(3-Methoxypropyl)-4,8-dinitro-1-phenyl-4,5,6,7,8,9-hexahydro-1*H*-4,8-methanopyrazolo[4,3-*d*]azocine (5e). The yield was 30%, m.p. 134—135 °C. Found (%): C, 57.01; H, 5.82; N, 17.37. $C_{19}H_{23}N_5O_5$. Calculated (%): C, 56.85; H, 5.78; N, 17.45. ^{1}H NMR, δ: 1.41—1.49 (m, 2 H); 2.43—3.34 (m, 14 H); 3.82 (d, 1 H, J = 16.4 Hz); 7.42—7.63 (m, 6 H, Ph, pyrazole).

6-(2-Methoxyethyl)-4,8-dinitro-2-phenyl-4,5,6,7,8,9-hexahydro-2*H*-4,8-methanopyrazolo[4,3-*d*]azocine (6). The yield was 78%, m.p. 109—111 °C. Found (%): C, 55.75; H, 5.15; N, 18.18. $C_{18}H_{21}N_5O_5$. Calculated (%): C, 55.81; H, 5.46; N, 18.08. ^{1}H NMR, δ: 2.50—2.73 (m, 9 H); 2.84—3.52 (m, 6 H); 7.29 (t, 1 H, *p*-Ph, J = 7.3 Hz); 7.47 (t, 2 H, *m*-Ph, J = 7.8 Hz); 7.83 (d, 2 H, *m*-Ph, J = 8.0 Hz); 8.41 (s, 1 H, pyrazole). IR, ν/cm^{-1}: 691, 757, 812, 1012, 1114, 1209, 1460, 1504, 1541, 1598.

Synthesis of compounds 9 and 10 (general procedure). A solution of the starting benzothiazole **8** (6 mmol) in acetic (propionic) anhydride (30 mL) was kept at 50—70 °C for 2 h. Then the reaction mixture was concentrated, EtOH (25 mL) was added to the residue, and the mixture was again concentrated. The precipitate that formed was washed with H_2O, filtered off, and dried in air.

***N*-(4,6-Dinitro-1,3-benzothiazol-2-yl)acetamide (9).** The yield was 93%, m.p. >260 °C. ^{1}H NMR, δ: 2.29 (s, 3 H); 8.89 (s, 1 H); 9.40 (s, 1 H); 13.32 (s, 1 H).

***N*-(4,6-Dinitro-1,3-benzothiazol-2-yl)propanamide (10).** The yield was 91%, m.p. >260 °C. ^{1}H NMR, δ: 1.13 (t, 3 H, J = 7.4 Hz); 2.61 (q, 2 H, J = 7.5 Hz); 8.88 (s, 1 H); 9.40 (s, 1 H); 13.28 (s, 1 H).

Synthesis of compounds 13 and 14 (general procedure). Sodium borohydride (0.23 g, 6 mmol) was added portionwise to a solution of benzothiazole **9** or **10** (1 mmol) in a mixture of THF (4 mL) and EtOH (12 mL) at a temperature ≤10 °C for 15 min. After 1 h, water (9 mL), a mixture of a 30% aqueous solution of RNH_2 (2.5 mL), water (2.5 mL), and a 30% formaldehyde solution, and then glacial AcOH (2.5 mL) were successively added. The reaction mixture was stirred at 20 °C for 30 min and then poured into water (200 mL). The precipitate was filtered off and dried in air.

[2-(Acetylamino)-4,8-dinitro-4,7,8,9-hexahydro-4,8-methano[1,3]thiazolo[4,5-*d*]azocin-6(5*H*)-yl]acetic acid (13a). The yield was 63%, m.p. 281 °C. Found (%): C, 40.43; H, 3.97; N, 18.37. $C_{13}H_{15}N_5O_7S$. Calculated (%): C, 40.52; H, 3.92; N, 18.17. ^{1}H NMR, δ: 2.09 (s, 3 H); 2.90 (d, 1 H, J = 11.0 Hz); 3.04 (d, 1 H, J = 10.3 Hz); 3.19—3.60 (m, 8 H); 12.24 (br.s, 2 H).

***N*-[6-(3-Methoxypropyl)-4,8-dinitro-4,5,6,7,8,9-hexahydro-4,8-methano[1,3]thiazolo[4,5-*d*]azocin-2-yl]acetamide (13b).** The yield was 49%, m.p. 200—201 °C. Found (%): C, 44.93; H, 5.69; N, 17.78. $C_{15}H_{21}N_5O_6S$. Calculated (%): C, 45.11; H, 5.30; N, 17.53. ^{1}H NMR, δ: 1.49 (m, 2 H); 2.09 (s, 3 H); 2.46—2.54 (m, 2 H); 2.64 (d, 1 H, J = 10.4 Hz); 2.75 (d, 1 H, J = 10.5 Hz); 2.85—2.99 (m, 4 H); 3.03 (s, 3 H); 3.12—3.39 (m, 3 H); 3.58 (d, 1 H, J = 16.6 Hz); 12.20 (br.s, 1 H). IR, ν/cm^{-1}: 685, 1077, 1122, 1267, 1343, 1369, 1547, 1693, 2828, 2939, 3163, 3424.

***N*-(6-Ethyl-4,8-dinitro-4,5,6,7,8,9-hexahydro-4,8-methano[1,3]thiazolo[4,5-*d*]azocin-2-yl)acetamide (13c).** The yield was 69%, m.p. 149—150 °C. Found (%): C, 43.66; H, 5.09; N, 19.82. $C_{13}H_{17}N_5O_5S$. Calculated (%): C, 43.94; H, 4.82; N, 19.71. ^{1}H NMR, δ: 0.86 (t, 3 H, J = 6.9 Hz); 2.09 (s, 3 H); 2.44—2.55 (m, 2 H); 2.60 (d, 1 H, J = 10.7 Hz); 2.76 (d, 1 H, J = 10.4 Hz); 2.91 (d, 1 H, J = 11.0 Hz); 3.19 (t, 2 H, J = 10.2 Hz); 3.33—3.39 (m, 2 H); 3.58 (d, 1 H, J = 16.7 Hz); 12.23 (s, 1 H).

***N*-[6-(2-Methoxyethyl)-4,8-dinitro-4,5,6,7,8,9-hexahydro-4,8-methano[1,3]thiazolo[4,5-*d*]azocin-2-yl]acetamide (13d).** The yield was 65%, m.p. 165—165 °C. Found (%): C, 43.55; H, 5.11; N, 18.04. $C_{14}H_{19}N_5O_6S$. Calculated (%): C, 43.63; H, 4.97; N, 18.17. ^{1}H NMR, δ: 2.09 (s, 3 H); 2.57—2.78 (m, 3 H);

2.91 (t, 1 H, J = 10.0 Hz); 3.11 (s, 3 H); 3.18—3.41 (m, 7 H); 3.57 (d, 1 H, J = 16.2 Hz); 12.23 (s, 1 H).

***N*-(6-Methyl-4,8-dinitro-4,5,6,7,8,9-hexahydro-4,8-methano[1,3]thiazolo[4,5-*d*]azocin-2-yl)propanamide (14a).** The yield was 69%, m.p. 248—249 °C. Found (%): C, 44.05; H, 5.02; N, 19.43. $C_{13}H_{17}N_5O_5S$. Calculated (%): C, 43.94; H, 4.82; N, 19.71. ^{1}H NMR, δ: 1.04—1.16 (m, 4 H); 2.27 (s, 2 H); 2.34—2.42 (dd, 2 H, J = 7.4 Hz, J = 14.9 Hz); 2.50—2.54 (m, 1 H); 2.66 (d, 1 H, J = 10.3 Hz); 2.88 (d, 1 H, J = 10.9 Hz); 3.17 (t, 2 H, J = 12.3 Hz); 3.32—3.34 (m, 1 H); 3.40 (d, 1 H, J = 16.8 Hz); 3.58 (d, 1 H, J = 16.6 Hz); 12.12 (s, 1 H).

***N*-[6-(3-Methoxypropyl)-4,8-dinitro-4,5,6,7,8,9-hexahydro-4,8-methano[1,3]thiazolo[4,5-*d*]azocin-2-yl]propanamide (14b).** The yield was 58%, m.p. 134—135 °C. Found (%): C, 46.65; H, 5.41; N, 16.85. $C_{16}H_{23}N_5O_6S$. Calculated (%): C, 46.48; H, 5.61; N, 16.94. ^{1}H NMR, δ: 1.05—1.15 (m, 5 H); 1.49 (d, 2 H, J = 5.7 Hz); 2.38 (dd, 2 H, J_1 = 7.4 Hz, J_2 = 14.9 Hz); 2.48—2.52 (m, 1 H); 2.64 (d, 1 H, J = 10.4 Hz); 2.75 (d, 1 H, J = 10.5 Hz); 2.92 (d, 1 H, J = 11.3 Hz); 2.98 (d, 1 H, J = 6.4 Hz); 3.03 (s, 3 H); 3.14 (d, 1 H, J = 10.2 Hz); 3.23 (d, 1 H, J = 11.1 Hz); 3.31—3.33 (m, 1 H); 3.36 (d, 1 H, J = 17.4 Hz); 3.59 (d, 1 H, J = 16.6 Hz); 12.18 (s, 1 H). IR, ν/cm^{-1}: 1114, 1183, 1264, 1344, 1545, 1694.

[4,8-Dinitro-2-(propionylamino)-4,7,8,9-hexahydro-4,8-methano[1,3]thiazolo[4,5-*d*]azocin-6(5*H*)-yl]acetic acid (14c). The yield was 40%, m.p. 237—238 °C. Found (%): C, 41.99; H, 4.41; N, 17.61. $C_{14}H_{17}N_5O_7S$. Calculated (%): C, 42.10; H, 4.29; N, 17.54. ^{1}H NMR, δ: 1.05 (t, 3 H, J = 7.4 Hz); 2.38 (q, 2 H, J = 7.5 Hz); 2.89 (d, 1 H, J = 11.0 Hz); 3.03 (d, 1 H, J = 10.5 Hz); 3.19—3.46 (m, 7 H); 3.58 (d, 1 H, J = 16.6 Hz); 12.18 (s, 1 H); 12.42 (br.s, 1 H).

***N*-[6-(2-Methoxyethyl)-4,8-dinitro-4,5,6,7,8,9-hexahydro-4,8-methano[1,3]thiazolo[4,5-*d*]azocin-2-yl]propanamide (14d).** The yield was 50%, m.p. 130—132 °C. Found (%): C, 45.33; H, 5.41; N, 17.88. $C_{15}H_{21}N_5O_6S$. Calculated (%): C, 45.11; H, 5.30; N, 17.53. ^{1}H NMR, δ: 1.05 (s, 3 H); 2.38 (d, 2 H, J = 6.9 Hz); 2.50—2.78 (m, 3 H); 2.90 (t, 1 H, J = 10.0 Hz); 3.11 (s, 3 H); 3.21—3.41 (m, 7 H); 3.57 (d, 1 H, J = 16.6 Hz); 12.18 (s, 1 H).

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