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Synthesis of N^3-Substituted Methyl *exo*-3,4-Diazatricyclo[5.2.1.02,6]dec-4-ene-5-carboxylates

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Abstract—Reactions of methyl *exo*-3,4-diazatricyclo[5.2.1.$0^{2,6}$]dec-4-ene-5-carboxylate with methyl diazoacetate, methyl iodide, ethylene oxide, acetic acid, nitrosonium cation, and sulfur led to the formation of a series of N^3-substituted *exo*-3,4-diazatricyclo[5.2.1.$0^{2,6}$]dec-4-enes.

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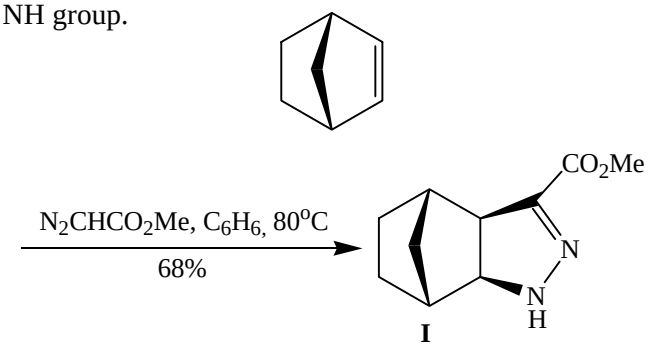
The high biological activity (antiviral, neurotropic, antiphlogistic, antiglycemic [1, 2]) of amines from the norbornane series calls for development for them of new preparation methods and ways of their chemical modification. The procedure of 1,3-dipolar cycloaddition of diazo compounds to norbornene and its derivatives is an efficient approach to the synthesis of diamines from the norbornane series [3, 4]. Among compounds obtained a substance was found endowed with a high antiarrhythmic effect [4]. Moreover, it is known that pyrazoles with a fused norbornane fragment possessed antiviral activity [5].

Acylation, alkylation, and some other chemical reactions of monocyclic pyrazolines are sufficiently well documented [6–10], but these reactions are not studied with pyrazolines of the norbornane series.

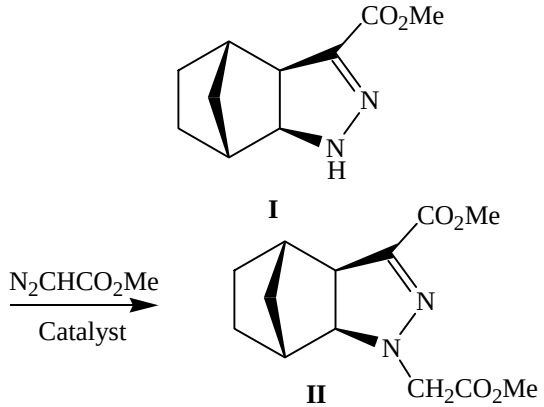
In this study with the goal of preparation of new 3,4-diazatricyclo[5.2.1.$0^{2,6}$]dec-4-ene derivatives we carried out reactions of methyl *exo*-3,4-diazatricyclo-[5.2.1.$0^{2,6}$]-dec-4-ene-5-carboxylate (**I**) with methyl diazoacetate, methyl iodide, ethylene oxide, acetic acid, nitrosonium cation, and sulfur.

Initial compound **I** was obtained in 68% yield by *exo*-stereoselective 1,3-dipolar cycloaddition of methyl diazoacetate to norbornene at 80°C in benzene solution. Spin-spin coupling of protons at C^2 and C^6 ($^3J_{1,2} = {}^3J_{6,7} = 0$ Hz) and the chemical shift of C^{10} (δ 32.94 ppm) [11] prove the *exo*-position of the pyrazoline fragment of the molecule. In the ^{1}H NMR spectrum appeared a signal from the proton of NH group as a broadened singlat at 6.34 ppm, and in the IR spectrum the absorption band at 3280 cm^{-1} corresponded to thestretching vibrations of the NH group.



I

Catalyzed reaction of compound **I** with methyl diazoacetate at 40°C in CH_2Cl_2 occurred by regioselective



Catalyst	$Rh_2(OAc)_4$	CuCN	CuCl	$(TfOCu)_2 \cdot C_6H_6$
Yield of compound **II**, %	92	97	87	87

insertion of methoxycarbonylcarbene into N–H bond. Among various catalysts [CuCl, CuCN, $(TfOCu)_2 \cdot C_6H_6$, $Rh_2(OAc)_4$] the most efficient under the chosen conditions was CuCN in whose presence the yield of methyl 3-methoxycarbonylmethyl-*exo*-3,4-diazatricyclo-[5.2.1.$0^{2,6}$]dec-4-ene-5-carboxylate (**II**) reached 97%. On performing the reaction at 20°C in CH_2Cl_2 or at 35°C in Et_2O the yield of heterocycle **II** considerably reduced.

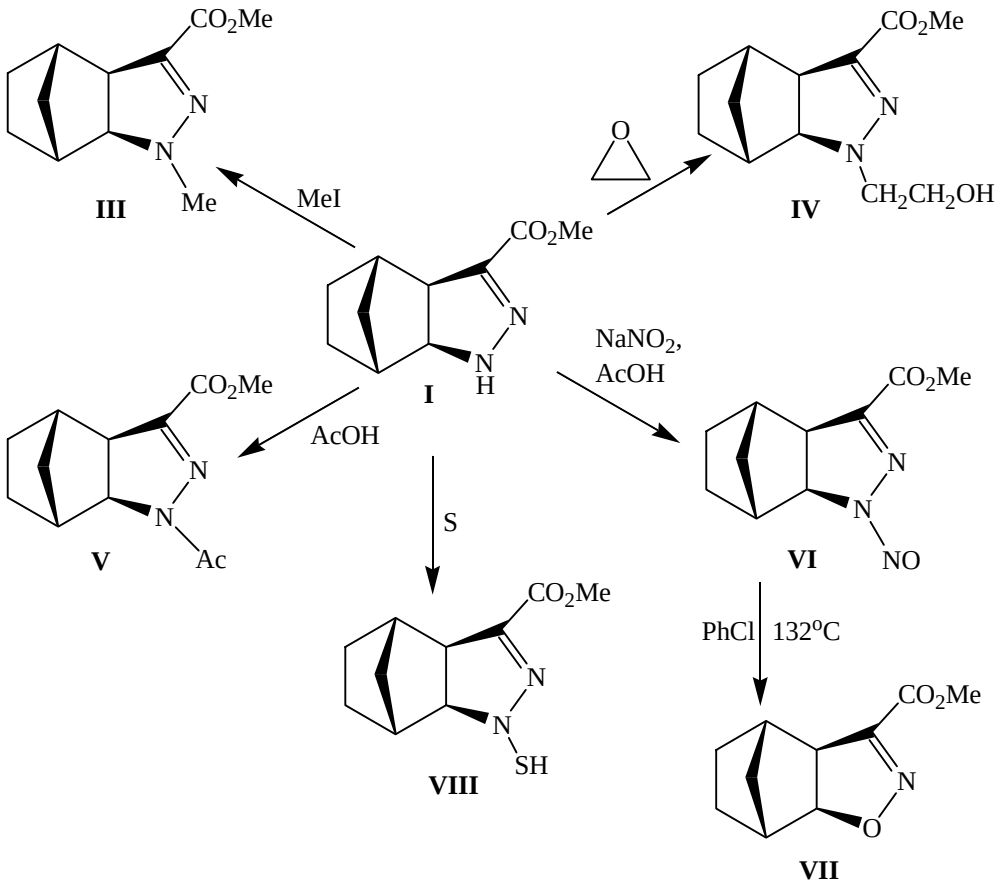
As with monocyclic pyrazolines [7] in the reaction of compound **I** with equimolar amount of MeI in boiling ethanol a regioselective alkylation at the N^3 atom was observed giving methyl 3-methyl-exo-3,4-diazatricyclo-[5.2.1.$0^{2,6}$]dec-4-ene-5-carboxylate (**III**) in 78% yield.

Compound **I** reluctantly reacted with ethylene oxide. After passing a 50-fold molar excess of gaseous ethylene oxide through water-methanol (1:1) suspension of compound **I** for 3 h at 60°C methyl 3-(2-hydroxyethyl)-*exo*-3,4-diazatricyclo[5.2.1.$0^{2,6}$]dec-4-ene-5-carboxylate (**IV**) was obtained in 49% yield.

Compound **I** is easily acetylated at boiling with excess AcOH giving in quantitative yield acetylpyrazoline (**V**); the reaction with $NaNO_2$ in AcOH at 0°C occurs with a complete conversion of initial compound within 15 min leading to the formation of N-nitrosopyrazoline **VI** in 70% yield. On heating compound **VI** in PhCl for 2 h the pyrazoline fragment of the molecule suffered nitrogen elimination leading to dihydroxyisoxazole **VII** in 92% yield. No products of norbornane skeleton isomerization were found in the reaction mixture.

Pyrazolines are capable to convert into pyrazoles by dehydrogenation effected with sulfur [12]. However in reaction of compound **I** with sulfur at 160–180°C instead of expected dehydrogenation the sulfur atom inserted into the N–H bond giving N-thiol **VIII** in 92% yield. Reaction carried out at 200–220°C resulted in a complex mixture of substances. Thiol **VIII** was isolated in individual state by sublimation at 150–160°C.

Composition and structure of compounds **I–VIII** prepared were confirmed by elemental analysis and by ^1H and ^{13}C NMR spectroscopy with the use of procedures H–H COSY and {C,H}-correlation. The *exo*-position of the pyrazoline fragment in compounds **I–VIII** is proved



by the coupling constants $^3J_{1,2} = {}^3J_{6,7} = 0$ Hz and the chemical shift of signal from C^{10} atoms in the region 32.30–33.54 ppm [11]. In the ^{1}H NMR spectrum of compound **II** the methylene protons signals from NCH_2CO_2Me group are nonequivalent and appear as well resolved doublets (^2J 17.9 Hz). The introduction into the position *3* of pyrazoline molecule of acetyl or nitroso group resulted in the downfield shift of the signal from H^2 proton and in decrease in the coupling constant between protons attached to C^2 and C^6 ($^3J_{2,6}$) to 8–9.1 Hz.

Thus we developed convenient synthetic routes to a number of N^3-substituted methyl 3,4-diazatricyclo-[5.2.1.0^{2,6}]dec-4-ene-5-carboxylates which might by applied to the fine organic synthesis and preparation of biologically active substances.

EXPERIMENTAL

IR spectra were obtained on a spectrophotometer Specord M-80 from thin films or mulls in mineral oil. ^{1}H and ^{13}C NMR spectra were registered on a spectrometer Bruker AM-300 (at 300.13 and 75.47 MHz respectively) in $CDCl_3$, internal reference Me_4Si. Melting points were measured on a Bötius heating microblock. GLC analyses were performed on a chromatograph Chrom-5 equipped with a flame-ionization detector, column 1200×5 mm, stationary phase 5% SE-30 on Inerton N-AW DMCS (0.125–0.160 mm), carrier gas helium. TLC was done on TCX Silufol plates (Kavalier), preparative separation was carried out by column chromatography on SiO_2 (Lancaster, 70–230 mesh).

Methyl *exo*-3,4-diazatricyclo[5.2.1.0^{2,6}]dec-4-ene-5-carboxylate (I). To a solution of 5.0 g (5.3 mmol) of norbornene in 80 ml of benzene was added at stirring 5.9 g (5.9 mmol) of methyl diazoacetate, and the reaction mixture was boiled for 6 h. The solvent was removed at a reduced pressure. Yield 6.9 g (67%). Yellowish crystals, mp 110–111°C (*i*-PrOH–petroleum ether). IR spectrum, cm^{-1}: 844, 1108, 1210, 1258, 1354, 1528, 1690, 2854, 2950, 3280. ^{1}H NMR spectrum, δ, ppm: 1.16–1.20 m (1H, H^{9endo}), 1.21 d (1H, H^{10anti}, $^2J_{10anti,10syn}$ 10.4 Hz), 1.30–1.34 m (1H, H^{8endo}), 1.42 d (1H, H^{10syn}, $^2J_{10anti,10syn}$ 10.4 Hz), 1.47–1.51 m (1H, H^{9exo}), 1.53–1.57 m (1H, H^{8exo}), 2.29 d (1H, H^7, $^3J_{7,8exo}$ 3.8 Hz), 2.52 d (1H, H^1, $^3J_{9exo,1}$ 3.2 Hz), 3.16 d (1H, H^6, $^3J_{6,2}$ 10.2 Hz), 3.74 s (3H, Me), 3.90 d (1H, H^2, $^3J_{6,2}$ 10.2 Hz), 6.34 br.s (1H, NH). ^{13}C NMR spectrum, δ, ppm: 24.37 (C^9), 27.86 (C^8), 32.94 (C^{10}), 40.30 (C^7), 44.36 (C^1), 51.90 (OMe), 53.39 (C^6), 68.11 (C^2), 141.51 (C^5), 163.34 (CO_2). Found, %: C 61.74; H 7.32; N 14.37. $C_{10}H_{14}N_2O_2$. Calculated, %: C 61.84; H 7.27; N 14.42.

Methyl 3-methoxycarbonylmethyl-*exo*-3,4-diazatricyclo[5.2.1.0^{2,6}]dec-4-ene-5-carboxylate (II). To a solution of 0.5 g (2.6 mmol) of compound **I** and 0.052 mmol of an appropriate catalyst in 15 ml of CH_2Cl_2 at stirring under an argon atmosphere was added by portions within 3 h 0.44 g (4.4 mmol) of methyl diazoacetate in 15 ml of CH_2Cl_2, and the reaction mixture was stirred for 2 h more. The solvent was removed at a reduced pressure,the residue was dissolved in 5 ml of Et_2O and passed through a thin bed of Al_2O_3. On removing the solvent at a reduced pressure the residue was subjected to chromatography on SiO_2. We obtained compound **II** as an oily fluid, R_f 0.47 (*i*-PrOH–petroleum ether, 1:7). IR spectrum, cm^{-1}: 1062, 1134, 1420, 1500, 1508, 1514, 1540, 1596, 1720, 1736, 2810, 3020. ^{1}H NMR spectrum, δ, ppm: 1.07–1.12 m (1H, H^{9endo}), 1.20 d (1H, H^{10anti}, $^2J_{10anti,10syn}$ 10.5 Hz), 1.25–1.31 m (1H, H^{8endo}), 1.41 d (1H, H^{10syn}, $^2J_{10anti,10syn}$ 10.5 Hz), 1.48–1.51 m (2H, H^{8exo}, H^{9exo}), 2.36 br.s (1H, H^7), 2.57 br.s (1H, H^1), 3.32 d (1H, H^6, $^3J_{6,2}$ 10.3 Hz), 3.69 s (3H, Me), 3.77 s (3H, Me), 3.85 d (1H, H^2, $^3J_{6,2}$ 10.3 Hz), 3.99 d (1H, CH_2CO_2, ^2J 17.9 Hz), 4.32 d (1H, CH_2CO_2, ^2J 17.9 Hz). ^{13}C NMR spectrum, δ, ppm: 24.06 (C^9), 27.80 (C^8), 33.44 (C^{10}), 40.68 (C^1), 41.82 (C^7), 51.70 (O<u>Me</u>), 51.90 (O<u>Me</u>), 51.96 (<u>C</u>H_2CO_2), 54.93 (C^6), 72.09 (C^2), 139.85 (C^5), 163.10 (CO_2), 170.08 (CO_2). Found, %: C 59.29; H 6.95; N 10.28. $C_{13}H_{18}N_2O_4$. Calculated, %: C 59.33; H 6.85; N 10.32.

Methyl 3-methyl-*exo*-3,4-diazatricyclo[5.2.1.0^{2,6}]-dec-4-ene-5-carboxylate (III). A solution of 1 g (0.5 mmol) of pyrazoline **I** and 0.8 g (0.56 mmol) of MeI in 15 ml of EtOH was boiled for 24 h. The reaction mixture was cooled to room temperature, and thereto was added in succession 20 ml of saturated water solution of $NaHCO_3$, 5 ml of saturated water solution of Na_2SO_3, and 10 ml of EtOH; the separated precipitate was filtered off. At a reduced pressure EtOH was removed, and from the water solution the products were extracted into CH_2Cl_2 (3×15 ml), the extract was dried with Na_2SO_4. CH_2Cl_2 was removed in a vacuum, and the residue was subjected to chromatography on SiO_2. Yield 0.83 g (78%). Oily fluid, R_f 0.58 (*i*-PrOH–petroleum ether, 1:3). IR spectrum, cm^{-1}: 1160, 1168, 1188, 1240, 1328, 1356, 1424, 1728, 2800, 3060. ^{1}H NMR spectrum, δ, ppm: 1.16–1.20 m (1H, H^{9endo}), 1.21 d (1H, H^{10anti}, $^2J_{10anti,10syn}$ 10.4 Hz), 1.30–1.34 m (1H, H^{8endo}), 1.42 d (1H, H^{10syn}, $^2J_{10anti,10syn}$ 10.4 Hz), 1.47–1.51 m (1H, H^{9exo}), 1.53–

1.57 m (1H, H^{8exo}), 2.47 br.s (1H, H^{7}), 2.57 br.s (1H, H^{1}), 3.10 s (3H, NMe), 3.24 d (1H, H^{6}, $^{3}J_{6,2}$ 10.7 Hz), 3.63 d (1H, H^{2}, $^{3}J_{6,2}$ 10.7 Hz), 3.78 s (3H, OMe). ^{13}C NMR spectrum, δ, ppm: 24.31 (C^{9}), 27.69 (C^{8}), 33.33 (C^{10}), 38.30 (NMe), 40.72 (C^{7}), 41.47 (C^{1}), 51.55 (OMe), 54.01 (C^{6}), 73.76 (C^{2}), 141.51 (C^{5}), 163.39 (CO_2). Found, %: C 64.82; H 7.96; N 13.73. $C_{11}H_{16}N_2O_2$. Calculated, %: C 64.74; H 7.84; N 13.45.

Methyl 3-(2-hydroxyethyl)-*exo*-3,4-diazatricyclo-[5.2.1.0^{2,6}]dec-4-ene-5-carboxylate (IV). Through a suspension of 0.5 g (2.5 mmol) of compound **I** in 20 ml of a mixture of MeOH and H_2O, 1:1, at 60°C was passed 5.5 g (130 mmol) of ethylene oxide for 3 h. The solvent was removed at a reduced pressure, and the residue was subjected to chromatography on SiO_2. Yield 0.3 g (49%). Oily fluid, R_f 0.36 (*i*-PrOH– petroleum ether, 1:5). IR spectrum, cm^{-1}: 1056, 1120, 1160, 1216, 1256, 1688, 1708, 2800, 3050, 3100, 3670. ^{1}H NMR spectrum (DMF-d_7), δ, ppm: 1.17 m (1H, H^{9endo}), 1.17 d (1H, H^{10anti}, $^{2}J_{10anti,10syn}$ 9.9 Hz), 1.22-1.29 m (2H, H^{10syn}, H^{8endo}), 1.45–1.48 m (2H, H^{9exo}, H^{8exo}), 2.42 br.s (1H, H^{7}), 2.51 br.s (1H, H^{1}), 3.19 d (1H, H^{6}, $^{3}J_{6,2}$ 10.5 Hz), 3.41–3.50 m (4H, CH_2CH_2O), 3.70 s (3H, Me), 3.93 d (1H, H^{2}, $^{3}J_{6,2}$ 10.5 Hz). ^{13}C NMR spectrum (DMF-d_7), δ, ppm: 24.66 (C^{9}), 28.12 (C^{8}), 33.54 (C^{10}), 41.53 (C^{7}), 42.89 (C^{1}), 51.21 (Me), 54.28 (NCH_2), 54.35 (C^{6}), 60.61 (CH_2O), 73.24 (C^{2}), 136.67 (C^{5}), 163.90 (CO_2). Found, %: C 60.47; H 7.50; N 11.57. $C_{12}H_{18}N_2O_3$. Calculated, %: C 60.49; H 7.61; N 11.76.

Methyl 3-acetyl-*exo*-3,4-diazatricyclo-[5.2.1.0^{2,6}]-dec-4-ene-5-carboxylate (V). A solution of 0.5 g (2.6 mmol) of compound **I** in 10 ml of AcOH was boiled for 3 h. AcOH was removed at a reduced pressure, and the residue was subjected to chromatography on SiO_2. Yield 0.6 g (98%). Oily fluid, R_f 0.55 (petroleum ether–*i*-PrOH, 5:1). IR spectrum, cm^{-1}: 1114, 1258, 1318, 1372, 1456, 1690, 2800, 3100. ^{1}H NMR spectrum, δ, ppm: 1.18 s (2H, H^{10anti}, H^{10syn}), 1.23–1.34 m (2H, H^{8endo}, H^{9endo}), 1.47–1.61 m (1H, H^{8exo}, H^{9exo}), 2.31 s (3H, COMe), 2.59 br.s (1H, H^{7}), 2.79 br.s (1H, H^{1}), 3.24 d (1H, H^{6}, $^{3}J_{6,2}$ 9.1 Hz), 3.85 s (3H, OMe), 4.25 d (1H, H^{2}, $^{3}J_{6,2}$ 9.1 Hz). ^{13}C NMR spectrum, δ, ppm: 21.69 (CO<u>Me</u>), 24.40 (C^{9}), 27.32 (C^{8}), 32.49 (C^{10}), 40.35 (C^{7}), 41.30 (C^{1}), 52.51 (O<u>Me</u>), 54.18 (C^{6}), 65.88 (C^{2}), 148.70 (C^{5}), 162.12 (CO_2), 169.95 (<u>C</u>OMe). Found, %: C 61.10; H 6.79; N 11.90. $C_{12}H_{16}N_2O_3$. Calculated, %: C 61.00; H 6.83; N 11.86.

Methyl 3-nitroso-*exo*-3,4-diazatricyclo-[5.2.1.0^{2,6}]dec-4-ene-5-carboxylate (VI). To a solution of 0.5 g (2.6 mmol) of compound **I** in 20 ml of AcOH was added by portions at 0–10°C while stirring 1.3 g (18.84 mmol) of $NaNO_2$, and the stirring was continued for 15 min at room temperature. The reaction mixture was poured into 25 ml of H_2O at 0°C, the separated precipitate was filtered off and washed with cold H_2O. Yield 0.4 g (70%). Yellow crystals, mp 67–68°C (AcOH–H_2O). IR spectrum, cm^{-1}: 1102, 1114, 1156, 1222, 1252, 1432, 1726, 2750, 3020. ^{1}H NMR spectrum, δ, ppm: 1.11 d (1H, H^{10anti}, $^{2}J_{10anti,10syn}$ 11.2 Hz), 1.20 d (1H, H^{10syn}, $^{2}J_{10anti,10syn}$ 11.2 Hz), 1.30–1.37 m (2H, H^{8endo} , H^{9endo}), 1.53–1.68 m (2H, H^{8exo}, H^{9exo}), 2.70 br.s (1H, H^{7}), 2.75 br.s (1H, H^{1}), 3.31 d (1H, H^{6}, $^{3}J_{6,2}$ 8.0 Hz), 3.94 s (3H, OMe), 4.35 d (1H, H^{2}, $^{3}J_{6,2}$ 8.0 Hz). ^{13}C NMR spectrum, δ, ppm: 24.31 (C^{9}), 27.06 (C^{8}), 32.50 (C^{10}), 39.57 (C^{7}), 39.94 (C^{1}), 53.06 (OMe), 54.24 (C^{6}), 66.40 (C^{2}), 155.26 (C^{5}), 161.51 (CO_2). Found, %: C 53.72; H 5.84; N 18.77. $C_{10}H_{13}N_3O_3$. Calculated, %: C 53.80; H 5.87; N 18.82.

Methyl *exo*-3-oxa-4-*exo*-azatricylo[5.2.1.0^{2,6}]-dec-4-ene-5-carboxylate (VII). A solution of 0.5 g (2.24 mmol) of compound **VI** in 20 ml of PhCl was boiled under an argon atmosphere for 2 h. The solvent was removed at a reduced pressure, and the residue was subjected to chromatography on SiO_2. Yield 0.4 g (92%). Yellowish fluid, R_f 0.49 (*i*-PrOH–petroleum ether, 1:5). IR spectrum, cm^{-1}: 820, 874, 1030, 1064, 1496, 1508, 1618, 1702, 2328, 2360. ^{1}H NMR spectrum, δ, ppm: 1.12–1.29 m (2H, H^{8endo}, H^{9endo}), 1.23 d (1H, H^{10anti}, $^{2}J_{10anti,10syn}$ 10.8 Hz), 1.39 d (1H, H^{10syn}, $^{2}J_{10anti,10syn}$ 10.8 Hz), 1.51–1.62 m (2H, H^{8exo}, H^{9exo}), 2.57 br.s (1H, H^{7}), 2.61 br.s (1H, H^{1}), 3.30 d (1H, H^{6}, $^{3}J_{6,2}$ 8.4 Hz), 3.86 s (3H, OMe), 4.68 d (1H, H^{2}, $^{3}J_{6,2}$ 8.4 Hz). ^{13}C NMR spectrum, δ, ppm: 22.55 (C^{9}), 27.09 (C^{8}), 32.20 (C^{10}), 39.28 (C^{7}), 42.83 (C^{1}), 52.54 (OMe), 55.48 (C^{6}), 90.31 (C^{2}), 152.01 (C^{5}), 161.20 (CO_2). Found, %: C 61.51; H 6.66; N 7.16. $C_{10}H_{13}NO_3$. Calculated, %: C 61.53; H 6.71; N 7.18.

Methyl 3-sulfanyl-*exo*-3,4-diazatricyclo-[5.2.1.0^{2,6}]dec-4-ene-5-carboxylate (VIII). In a reactor for sublimation was melted 1 g (5.15 mmol) of compound **I** and 0.17 g (5.3 mmol) of sulfur for 2 h at 160–180°C and residual pressure 2 mm Hg. Yield 1.07 g (92%). Yellow crystals, mp 141–142°C (sublimation). IR spectrum, cm^{-1}: 1096, 1120, 1152, 1248, 1280, 1344, 1432, 1520, 1644, 1680, 2800, 3050, 3200, 3400. ^{1}H NMR spectrum, δ, ppm: 1.16–1.20 m (1H, H^{9endo}), 1.21 d (1H,

H^{10anti}, $^2J_{10anti,10syn}$ 10.8 Hz), 1.30–1.34 m (1H, H^{8endo}), 1.41 d (1H, H^{10syn}, $^2J_{10anti,10syn}$ 10.8 Hz), 1.47–1.51 m (1H, H^{9exo}), 1.53–1.57 m (1H, H^{8exo}), 2.33 br.s (1H, H^7), 2.57 br.s (1H, H^1), 3.20 d (1H, H^6, $^3J_{6,2}$ 10.1 Hz), 3.80 s (3H, OMe), 3.94 d (1H, H^2, $^3J_{6,2}$ 10.2 Hz). ^{13}C NMR spectrum, δ, ppm: 24.46 (C^9), 27.95 (C^8), 33.04 (C^{10}), 40.38 (C^7), 44.42 (C^1), 51.76 (OMe), 53.57 (C^6), 68.19 (C^2), 142.00 (C^5), 163.39 (CO_2). Found, %: C 52.15; H 6.50; N 12.22; S 14.11. $C_{10}H_{14}N_2O_2S$. Calculated, %: C 53.08; H 6.24; N 12.38; S 14.17.

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