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Amido Acids of Norbornene Series in Reactions with Sulfonyl Azides

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Abstract—In reaction of amido acids from the norbornene series with arenesunfonyl azides alongside the expected *N,N*-dialkyl-5-oxo-*exo*-2-arylsulfonylamino-4-oxatricyclo[4.2.1.$0^{3,7}$]nonane-*endo*-9-carboxamides (oxabrendanones) the formation of zwitter-ions with a structure of 5-(*N,N*-dialkyliminio)-*exo*-2-arylsulfonylamino-4- oxatricyclo[4.2.1.$0^{3,7}$]nonane-*endo*-9-carboxylates came as a surprise. The structure of compounds obtained was confirmed by the analysis of their IR and ^{1}H NMR spectra. The molecular structure of one among the zwitter-ions was established by XRD analysis.

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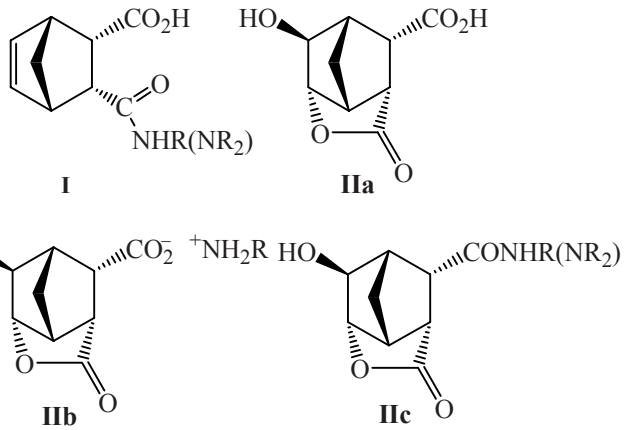
Amido acids **I** are the least understood transformation products of endic anhydride. Virtually no publications exist on the transformations of these compounds involving the strained double bond.

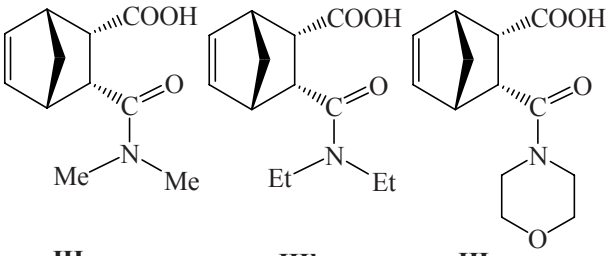
The oxidation of the compounds (**I**, R = Alk, cyclo-Alk, Ar; NR_2 = pyrrolidinyl, morpholino, piperidino, etc.) with peroxy acids depending on the character of substituents resulted in the formation of three types compounds **IIa–IIc** isolated in an individual state as well as like inseparable mixtures [1]. Amidolactones **IIc** are the least studied among these compounds; we formerly have developed a convenient procedure for their synthesis consisting in the aminolysis of the epoxyendic anhydride under mild conditions [2]. The reactions of amido acids **I** with other electrophilic reagents have not been studied before.



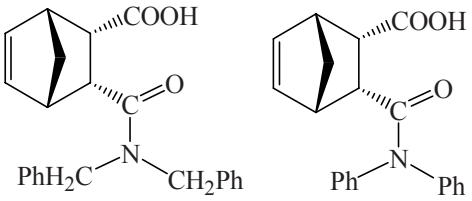
I **IIa** **IIb** **IIc**

Inasmuch as compounds **IIa–IIc** are the products of an intramolecular cyclization of epoxides intermediately formed in the oxidation of the double bond, we believe that the investigation of the reactions between amido acids **I** and other electrophilic reagents would extend the versatility of reaction products and clarify the mechanism of their formation. Among the electrophilic reagents the sulfonyl azides call for special attention because of their high reactivity with respect to double bonds, especially to the strained ones. The reagents are extensively used in the preparation of heterocyclic compounds, first of all, of substituted aziridines [3].

In this study we nave chosen as reagents available tosyl azide and *p*-nitrobenzenesulfonyl azide synthesized by procedure [4] and known amido acids **IIIa–IIIe** obtained by the standard method by the reaction of equimolar amounts of endic anhydride and an appropriate amine in benzene at room temperature [1]. The reactions of amido acids with azides were carried out in anhydrous chloro-form and also in benzene and acetonitrile either at boiling or at UV irradiation. The composition of reaction



IIIa **IIIb** **IIIc**



IIId **IIIe**

products depended on the substrate structure and the reaction conditions.

In reactions of amido acids **IIIa** and **IIIb** with tosyl azide and of amido acid **IIIa** with *p*-nitrobenzenesulfonyl azide in boiling chloroform we obtained mixtures of compounds that were successfully separated because they exhibited a different solubility in organic solvents (Scheme 1). Based on the data of spectral and XRD analyses the compounds obtained were assigned structures of zwitter-ions **IVa–IVc** and amidolactones **Va–Vc**.

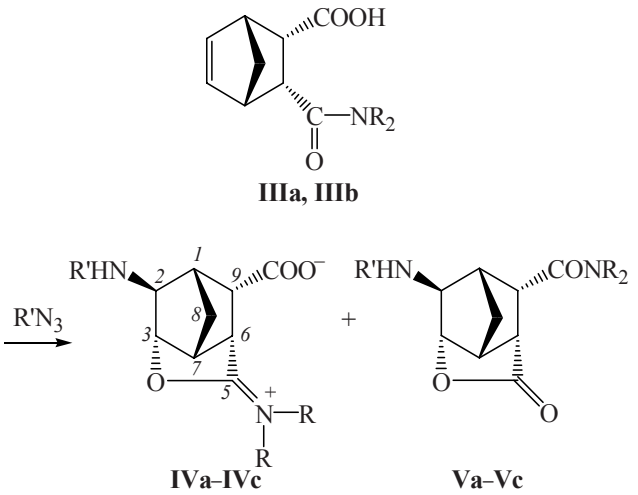
The analysis of IR spectra suggested preliminary conclusions on the character of the formed functional groups. In the spectra of compounds **IVa–IVc** absorption bands were present in the region 1600 cm^{-1} belonging to ionized carboxy groups, whereas in the spectra of amidolactones **Va–Vc** absorption bands appeared corresponding to carbonyl groups both of lactone (1775–1760 cm^{-1}) and amide (1670–1635 cm^{-1}) fragments [5].

The key signals in the ^1H NMR spectra indicating the different structure of compounds obtained are the signals of protons at atoms C^2 and C^3. In the spectra of amidolactones **Va–Vc** these signals appear as a singlet (4.1 ppm) and a doublet (4.3 ppm) respectively; in the spectra of zwitter-ion structures **IVa–IVc** the position of the proton signal at C^3 does not significantly change whereas the signal of the proton at C^2 is observed upfield (2.9–2.8 ppm). The final conclusion on the structure of zwitter-ions **IVa–IVc** was done based on the XRD analysis of compound **IVa**.

The XRD study revealed that compound **IVa** existed in the crystal as a monohydrate (see the figure). The bond distances* O^3–C^9 [1.223(3) Å] and O^2–C^9 [1.234(3) Å] vary insignificantly characteristically of a carboxylate anion (an average bond length C–O in a carboxylate anion is 1.254 Å [6]). The bond distance N^2–C^1 1.296(2) Å is comparable with the mean value [6] for the C_{sp^2}=N^{3+} bond (1.316 Å) suggesting that the positive charge is localized on the atom N^2. Thus in the crystal compound **IVa** exists in a zwitter-ion form.

The five-memebered heterocycle is in the envelope conformation. The deviation of C^7 atom from the mean-square plane of the other atoms of the ring amounts to –0.61 Å. Both five-membered carbocycles included in the framework of the compound are also found to be present in the envelope conformation. The deviation of C^8 atom from the mean-square plane of the other atoms

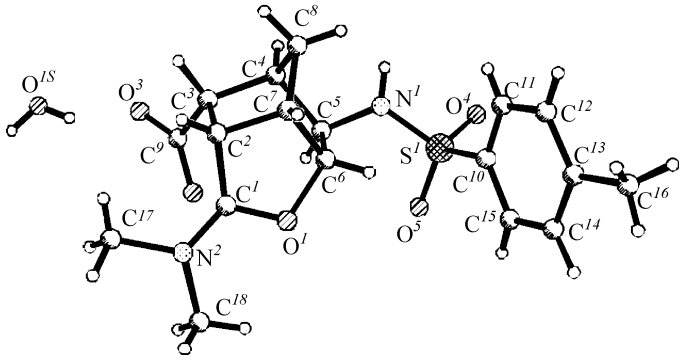
Scheme 1.



IIIa, IIIb

IVa–IVc **Va–Vc**

IV, V, R = CH_3, R' = Ts (**a**), Ns (**b**); R = C_2H_5, R' = Ts (**c**); Ns = *p*-$NO_2C_6H_4SO_2$, Ts = *p*-$CH_3C_6H_4SO_2$.



Molecular structure of compound **IVa** monohydrate according to XRD analysis.

* The numeration of atom is the same as shown in the figure.

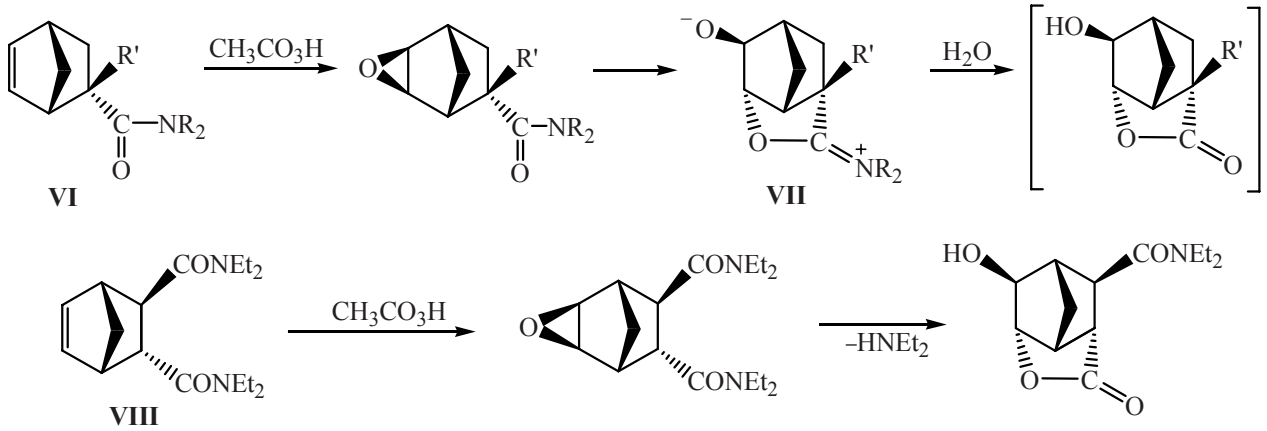
of the ring $C^7C^2C^3C^4C^8$ is 0.84 Å, and for the ring $C^7C^6C^5C^4C^8$, –0.84 Å. The carboxy group has the *endo*-orientation and the substituent at the atom C^5, *exo*-orientation (torsion angles $C^7C^2C^3C^9$ 138.5(2), $N^1C^5C^6C^7$ 108.3(2) deg respectively). This position of substituents results in a shortened intramolecular contact H^5–C^9 2.79 Å (sum of van der Waals radii is 2.87 Å [7]). The carboxy group is turned so that the C^9–O^3 bond is in *ac*-conformation with respect to the C^2–C^3 bond [torsion angle $C^2C^3C^9O^3$ 135.9(2) deg]. The sulfoxide group possesses the +*sc*-orientation with respect to the C^5–C^6 bond [torsion angle $S^1N^1C^5C^6$ 64.6(2) deg] and is located in a position leading to the conformation of the O^5–S^1 bond intermediate between *sp* and +*sc* with respect to the N^1–C^5 bond [torsion angle $O^5S^1N^1C^5$ 31.3(2) deg]. The tolyl substituent is located practically normal to the N^1–C^5 bond and is notably turned with respect to the N^1–S^1 bond [torsion angles $C^{10}S^1N^1C^5$ –84.5(2) deg, $N^1S^1C^{10}C^{11}$–47.3(2) deg respectively]. This position of the substituent results in relatively strong repulsion between the C^{10} atom of the aromatic ring and the bicycloheptane fragment [a shortened intramolecular contact $H^6\cdots C^{10}$ 2.64 Å (2.87 Å)]. A sufficiently strong repulsion was found also in molecule **IVa** between a dimethylamino group and the atoms of the five-membered heterocyle [shortened intramolecular contacts $H^{17A}\cdots C^2$ 2.64 (2.87), $H^{17A}\cdots H^2$ 2.21 (2.34), $H^2\cdots C^{17}$ 2.79 (2.87), $H^{18A}\cdots O^1$ 2.36 E (2.46 Å).

Molecules **IVa** form dimers in the crystal where zwitter-ions are interlinked through bridging water molecules owing to intermolecular hydrogen bonds O^{1S}–$H^{1Sa}\cdots O^2$ ($1-x, -y, 1-z$) (H···O 2.03 Å, O–H···O 160 deg) and O^{1S}–$H^{1Sb}\cdots O^3$ (H···O 1.91 Å, O–H···O 157 deg). The dimers form stacks along the crystallographic direction [*0 1 0*]; the stacks are linked to each other with an intermolecular hydrogen bond N^1–$H^{1N}\cdots O^{1S}$ ($1-x, 0.5+y, 0.5-z$) (H···O 1.93 Å, N–H···O 174 deg). Weaker intermolecular hydrogen bonds were also found in the crystal C^2–$H^2\cdots O^3$ ($1-x, -0.5+y, 0.5-z$) (H···O 2.32 Å, C–H···O 155 deg) and C^7–$H^7\cdots O^{4'}$ ($x, y-1, z$) (H···O 2.41 Å, C–H···O 130 deg).

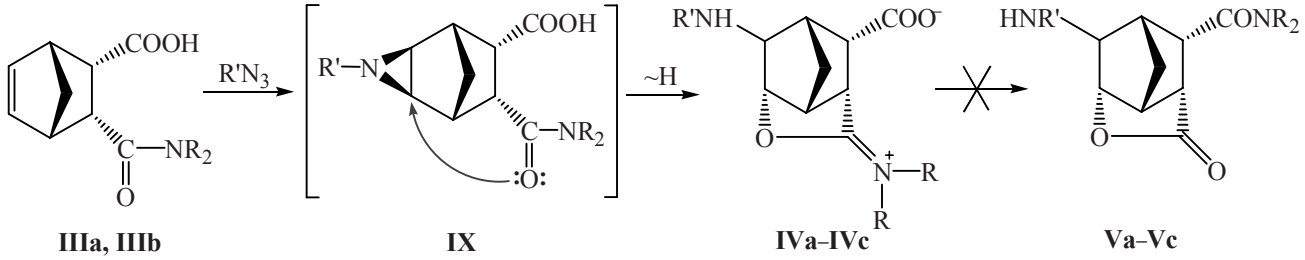
The mixtures of the described compounds formed both in the reactions carried out at room temperature and at UV irradiation, but in the latter case the yield of compounds with a zwitter-ion structure considerably decreased.

The isolation of compounds **IVa–IVc** with the zwitter-ion structure is a sufficiently unusual fact. Compounds of analogous structure **VII** were formerly regarded as

Scheme 2.



Scheme 3.



probable intermediates in the intramolecular cyclization occurring, e.g., during the oxidation with peroxy acids of monocarboxylic acids amides [**VI**, NR_2 = $NHCH_3$, $N(CH_3)_2$, NHPh; R' = H, CH_3] [8] or diamides **VIII** [9].

The zwitter-ions formation is evidently a result of the heterocyclization of the intermediately arising azididines **IX**; therewith compounds **IVa–IVc** should not be regarded as intermediates in the formation of amidolactones **Va–Vc**.

The reactions of amido acids **IIIc–IIIe** with tosyl azide afforded as only reaction products new amidolactones **Vd–Vf** (Scheme 4) whose structure was confirmed by IR and ^{1}H NMR spectra; no zwitter-ion products were detected in these case either by TLC or by measuring the NMR spectra of the reaction mixtures.

The formation of two types compounds from the amido acids of the norbornene series and sulfonyl azides resulted apparently from the competing involvement of two nucleophilic centers (oxygen atoms of the carboxy and amido groups) into the reactions of the intramolecular opening of the aziridine ring. The theoretical concepts on the electron density distribution in these groups suggest the prevalence of the more nucleophilic amide group; however a recent quantum-chemical investigation [10] of the lactonization mechanism of epoxyendic anhydride (**X**) effected by amines in neutral media has shown that in the absence of solvents capable of specific solvation of the substrate the heterocyclization is preceded by an intramolecular proton transfer from the carboxy to amide group. Therefore in the heterocyclization takes part the more nucleophilic ionized carboxy group leading to the formation of amidolactones **IIc** as the only reaction products (Scheme 5).

Presumably, the proton transfer preceded the heterocyclization also in the reaction we studied between amido acids and azides. The formation of compounds mixtures in the presence the molecules of amido acids of bulky dimethyl- and diethylamino groups may result from the additional sterical shielding of the nitrogen atom preventing the proton transfer.

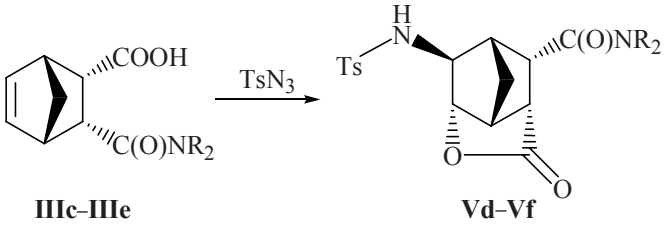
EXPERIMENTAL

IR spectra were measured on a spectrophotometer UR-20 from samples pelletized with KBr. ^{1}H NMR spectra were registered on a spectrometer Varian VXR-Unity at operating frequency 300 MHz from solutions in $CDCl_3$ with TMS as internal reference. The reaction progress was monitored and the purity of compounds synthesized was checked by TLC on Silufol UV-254 plates, eluent ether, develop-ment in iodine vapor. Elemental analyses were carried out on an analyzer Carlo Erba. The melting points were determined in open capillaries and were reported without corrections.

Reaction of amido acids III with arenesulfonyl azides. General procedure. To a solution of 1.61 mmol of amido acid in 5–7 ml of anhydrous chloroform was added 1.61 mmol of arenesulfonyl azide. The reaction mixture was boiled till the completion of the process (TLC monitoring). When zwitter-ion formed in reactions with amido acids **IIIa** and **IIIb** this product was filtered off, washed with chloroform, and the filtrate was evaporated. The residue after evaporation was subjected to column chromatography on silica gel (eluent ethyl ether).

5-(*N*,*N*-Dimethyliminio)-*exo*-2-tosylamino-4-oxatricyclo[4.2.1.0^{3,7}]nonane-*endo*-9-carboxylate (IVa). Yield 17%, mp 238°C (decomp.) (2-propanol–water, 10:1). IR spectrum, ν, cm^{-1}: 3170, 3010, 2985, 1695, 1600, 1398, 1317, 1160, 1110, 950, 900, 824, 760, 665. ^{1}H NMR spectrum, δ, ppm: 7.52 d (2H, H^{Ar}, *J* 3.4 Hz),

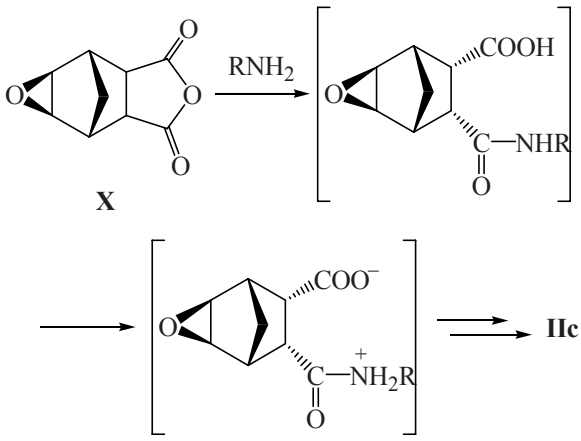
Scheme 4.



IIIc–IIIe **Vd–Vf**

V, NR_2 = —N O (**d**), $NHCH_2Ph$ (**e**), NHPh (**f**).

Scheme 5.



4.33 d (1H, H^3, *J* 4.7 Hz), 3.25 m (1H, H^6), 2.89 m (1H, H^7), 2.87 s (1H, H^2), 2.71 s (1H, H^9), 2.36 s (1H, H^1), 2.38 s (3H, ArMe), 2.27 s (6H, N^+Me_2), 1.98 d (1H, H^{8s}, *J* 10.8 Hz), 1.66 d (1H, H^{8an}, *J* 10.8 Hz). Found, %: N 7.06. $C_{18}H_{22}N_2O_5S$. Calculated, %: N 7.41.

5-(N,N-Dimethyliminio)-*exo*-2-(*p*-nitrophenylsulfonylamino)-4-oxatricyclo-[4.2.1.0^{3,7}]nonane-*endo*-9-carboxylate (IVb). Yield 52%, mp 249°C. IR spectrum, ν, cm^{-1}: 3155, 3010, 2985, 1680, 1620, 1370, 1330, 1185, 1160, 935, 920, 846, 730, 683. ^{1}H NMR spectrum, δ, ppm: 8.12 s (1H, NH), 7.90 d (2H, H^{Ar}, *J* 3.4 Hz), 4.42 d (1H, H^3, *J* 4.8 Hz), 3.32 m (1H, H^6), 2.91 s (1H, H^2), 2.91 m (1H, H^7), 2.89 s (1H, H^9), 2.38 s (1H, H^1), 2.29 s (6H, N^+Me_2), 2.00 d (1H, H^{8s}, *J* 10.9 Hz), 1.72 d (1H, H^{8an}, *J* 10.9 Hz). Found, %: N 10.30. $C_{17}H_{19}N_3O_7S$. Calculated, %: N 10.27.

5-(N,N-Diethyliminio)-*exo*-2-tosylamino-4-oxatricyclo[4.2.1.0^{3,7}]nonane-*endo*-9-carboxylate (IVc). Yield at boiling the reaction mixture 27%, at room temperature 54%, mp 230–231°C (2-propanol–water, 10 : 1). IR spectrum, ν, cm^{-1}: 3165, 3010, 2985, 1675, 1615, 1415, 1325, 1150, 950, 900, 825, 760, 665. ^{1}H NMR spectrum, δ, ppm: 7.86 s (1H, NH), 7.51 d (2H, H^{Ar}, *J* 3.4 Hz), 4.47 d (1H, H^3, *J* 4.6 Hz), 3.24 m (1H, H^6), 2.92 m (4H, CH_2CH_3), 2.89 m (1H, H^7), 2.86 s (1H, H^9), 2.75 s (1H, H^2), 2.41 s (1H, H^1), 2.35 s (3H, ArMe), 2.27 s (6H, N^+Me_2), 1.98 d (1H, H^{8s}, *J* 10.9 Hz), 1.66 d (1H, H^{8an}, *J* 10.9 Hz). Found, %: N 6.69. $C_{20}H_{26}N_2O_5S$. Calculated, %: N 6.90.

***N,N*-Dimethyl-5-oxo-*exo*-2-tosylamino-4-oxatricyclo[4.2.1.0^{3,7}]nonane-*endo*-9-carboxamide (Va)**. Yield 64%, mp 82–83°C. IR spectrum, ν, cm^{-1}: 3270, 3010, 2995, 2145, 1772, 1640, 1600, 1460, 1340, 1169, 1095, 1030, 828, 670. ^{1}H NMR spectrum, δ, ppm: 7.48 d (4H, H^{Ar}, *J* 3.4 Hz), 7.42 s (1H, NH), 4.35 d (1H, H^3, *J* 4.8 Hz), 4.05 s (1H, H^2), 3.31 m (1H, H^6), 2.86 m (1H, H^9), 2.73 m (1H, H^7), 2.41 m (1H, H^1), 2.39 s (3H, ArMe), 2.27 s (6H, NMe_2), 1.88 d (1H, H^{8s}, *J* 10.3 Hz), 1.52 d (1H, H^{8an}, *J* 10.3 Hz). Found, %: N 7.18. $C_{18}H_{22}N_2O_5S$. Calculated, %: N 7.41.

***N,N*-Dimethyl-5-oxo-*exo*-2-(*p*-nitrophenylsulfonylamino)-4-oxatricyclo[4.2.1.0^{3,7}]-nonane-*endo*-9-carboxamide (Vb).** Yield 48%, mp 102–103°C. IR spectrum, ν, cm^{-1}: 3255, 3010, 2995, 1765, 1670, 1625, 1380, 1320, 1095, 1010, 834, 615. ^{1}H NMR spectrum, δ, ppm: 8.42 s (1H, NH), 7.94 d (2H, H^{Ar}, *J* 3.4 Hz), 4.39 d (1H, H^3, *J* 4.7 Hz), 4.15 s (1H, H^2), 3.42 m (1H, H^6), 2.82 s (1H, H^9), 2.74 m (1H, H^7), 2.48 s (1H, H^1), 2.38 s (6H, NMe_2), 1.98 d (1H, H^{8s}, *J* 11.1 Hz), 1.65 d (1H, H^{8an}, *J* 11.1 Hz). Found, %: N 10.19. $C_{17}H_{19}N_3O_7S$. Calculated, %: N 10.27.

***N,N*-Diethyl-5-oxo-*exo*-2-tosylamino-4-oxatricyclo[4.2.1.0^{3,7}]nonane-*endo*-9-carboxamide (Vc)**. Yield 47%, mp 117–118°C. IR spectrum, ν, cm^{-1}: 3230, 3010, 2995, 2158, 1780, 1635, 1585, 1470, 1250, 1195, 1045, 864, 655. ^{1}H NMR spectrum, δ, ppm: 8.34 s (1H, NH), 7.61 d (4H, H^{Ar}, *J* 3.4 Hz), 4.30 d (1H, H^3, *J* 5.1 Hz), 4.02 m (1H, H^2), 3.51 m (1H, H^6), 2.92 m (1H, CH_2CH_3), 2.74 s (1H, H^9), 2.69 m (1H, H^7), 2.51 s (1H, H^1), 2.41 s (3H, ArMe), 2.38 m (1H, CH_2CH_3), 2.27 s (6H, N^+Me_2), 1.85 d (1H, H^{8s}, *J* 10.9 Hz), 1.39 d (1H, H^{8an}, *J* 10.9 Hz). Found, %: N 6.72. $C_{20}H_{26}N_2O_5S$. Calculated, %: N 6.90.

***N*-(Morpholin-4-yl)-*exo*-2-(tosylamino)-4-oxatricyclo[4.2.1.0^{3,7}]nonane-*endo*-9-carboxamide (Vd)**. Yield 96%, mp 86–87°C. IR spectrum, ν, cm^{-1}: 3280, 3010, 2995, 1772, 1640, 1590, 1480, 1347, 1185, 875, 634. ^{1}H NMR spectrum, δ, ppm: 7.49 d (4H, H^{Ar}, *J* 3.4 Hz), 7.16 s (1H, NH), 4.25 d (1H, H^3, *J* 5.1 Hz), 4.12 s (1H, H^2), 3.51 m (1H, H^6), 3.15–2.29 m (4H, morpholyl), 2.72 m (1H, H^7), 2.69 s (1H, H^9), 2.55 s (1H, H^1), 2.33 s (3H, ArMe), 1.83 d (1H, H^{8s}, *J* 11.8 Hz), 1.38 d (1H, H^{8an}, *J* 11.8 Hz). Found, %: N 6.59. $C_{20}H_{24}N_2O_6S$. Calculated, %: N 6.67.

***N,N*-Dibenzyl-5-oxo-*exo*-2-tosylamino-4-oxatricyclo[4.2.1.0^{3,7}]nonane-*endo*-9-carboxamide (Ve)**. Yield 86%, mp 138–139°C. IR spectrum, ν, cm^{-1}: 3255, 3010, 2995, 1760, 1655, 1590, 1477, 1275, 1185, 1083, 1030, 856, 629. ^{1}H NMR spectrum, δ, ppm: 7.58 d (2H, H^{Ar}, *J* 3.4 Hz), 7.28 s (1H, NH), 7.21 m (10H, H^{Ph}), 4.30 d (1H, H^3, *J* 5.1 Hz), 4.08 s (1H, H^2), 3.62 m (1H, H^6), 2.81 m (1H, H^7), 2.77 s (1H, H^9), 2.48 s (1H, H^1), 2.40 s (3H, ArMe), 2.23 s (4H, CH_2Ph), 1.81 d (1HH^{8s}, *J* 10.5 Hz), 1.40 d (1H, H^{8an}, *J* 10.5 Hz). Found, %: N 5.47. $C_{30}H_{30}N_2O_5S$. Calculated, %: N 5.28.

***N,N*-Diphenyl-5-oxo-*exo*-2-tosylamino-4-oxatricyclo[4.2.1.0^{3,7}]nonane-*endo*-9-carboxamide (Vf)**. Yield 72%, mp 115–116°C. IR spectrum, ν, cm^{-1}: 3265, 3010, 2995, 1765, 1640, 1615, 1435, 1335, 1265, 1135, 1019, 855, 665. ^{1}H NMR spectrum, δ, ppm: 7.58 d (2H, H^{Ar}, *J* 3.4 Hz), 7.56 s (1H, NH), 7.21 m (10H, NPh_2), 4.92 m (1H, H^2), 4.30 d (1H, H^3, *J* 5.1 Hz), 3.48 m (1H, H^6), 2.73 m (1H, H^7), 2.72 s (1H, H^9), 2.53 s (1H, H^1), 2.42 s (3H, ArMe), 2.27 s (6H, N^+Me_2), 1.82 d (1H, H^{8s}, *J* 10.8 Hz), 1.42 d (1H, H^{8an}, *J* 10.8 Hz). Found, %: N 5.80. $C_{28}H_{26}N_2O_5S$. Calculated, %: N 5.58.

X-ray diffraction analysis of compound (IVa). Crystals of compound **IVa** monoclimic, $C_{18}H_{24}N_2O_6S$. At 20°C *a* 16.222(1), *b* 7.6506(5), *c* 14.983(1) Å, β 91.048(6)°, *V* 1859.2(2) Å^3, M_r 396.45, *Z* 4, space group $P2_1/c$, d_{calc} 1.416 g/cm^3, μ(MoK_α) 0.213 mm^{-1}, *F*(000) 840. The parameters of unit cell and intensities of 18102 reflections (5421 independent, R_{int} 0.035) were measured on a diffractometer Xcalibur-3 (MoK_α radiatio, CCD-detector, graphite monochromator, ω-scanning, $2\theta_{max}$ 60°). The structure was solved by the direct method with the use of software SHELXTL [11]. The positions of hydrogen atoms were revealed from the difference synthesis of the electron density and refined in the isotropic approximation. The structure was refined for F^2 in the full-matrix mean-squares anisotropic approximation for nonhydrogen atoms till wR_2 0.168 for 5381 reflexions [R_1 0.057 for 3699 reflections with $F > 4\sigma(F)$, *S* 1.071]. The atomic coordinates and complete tables of bond distances and bond angles are deposited to the Cambridge Crystallographic Data Center (CCDC 689325).

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