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Synthesis and Some Reactions of New *N*-[Aryl(Benzyl, Cyclohexyl, Propyl)sulfonyl]-4-azatricyclo[5.2.1.0^{2,6}]dec-8-enes

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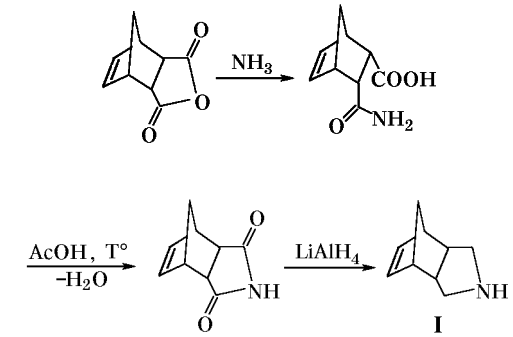
Abstract—A synthesis was accomplished of 4-azatricyclo[5.2.1.0^{2,6}]dec-8-ene by aminolysis of bicyclo-[2.2.1]hept-2-ene-endo,endo-5,6-dicarboxylic acid anhydride followed by transformation of amidoacid into imide that was subsequently reduced by lithium aluminum hydride. The reaction of the key tricyclic amine with sulfonyl chlorides afforded *N*-[aryl(benzyl, cyclohexyl, propyl)sulfonyl]-4-azatricyclo[5.2.1.0^{2,6}]dec-8-enes.The sulfonamides were subjected to epoxidation with perphthalic acid. By reaction of sulfonamides with p-nitrophenyl azide triazolines were obtained. The structure of compounds synthesized was confirmed by IR, ^{1}H and ^{13}C NMR spectra.

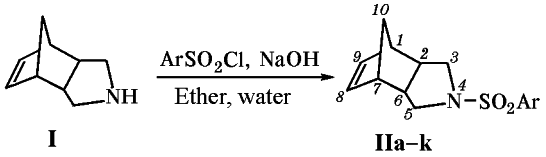
Already about 50 years have passed since the first publications on the synthesis of 4-azatricyclo-[5.2.1.0^{2,6}]dec-8-ene (**I**). However among its derivatives mainly alkylation products and salts were investigated; the latter showed considerable biological activity (hypotensive, tranquilizing, and ganglioplegic) [1]. A number of sulfonylureas was prepared from the key amine **I** that showed promise in diabetes treatment[2, 3]. Among the amine derivatives were also found substances active against gram-positive and gram-negative bacteria [4], and also morphine antagonists [5].

The only described sulfonamide, *N*-phenylsulfonyl-4-azatricyclo[5.2.1.0^{2,6}]dec-8-ene, is insufficiently investigated, and the range of its biological activity is not outlined [6, 7]. Incidentally, the related arylsulfonamides, derivatives of 5-aminomethylbicyclo-[2.2.1]hept-2-enes, possess a considerable neurotropic (analgetic, anticonvulsant, tranquilizing) and also antiphlogistic activity [8]. In that connection the goal of the present research consisted in preparation of sulfonamides from 4-azatricyclo[5.2.1.0^{2,6}]dec-8-ene and investigation of their reactions at the strained double bond [9].

Amine (**I**) was prepared along the previously described protocol from the bicyclo[2.2.1]hept-2-ene-*endo,endo*-5,6-dicarboxylic acid anhydride.

The reaction of amine **I** with sulfonyl chlorides was carried out in a two-phase system (ether–water) at equimolar ratio of reagents (amine, sulfonyl chloride, sodium hydroxide) [10]. In keeping with the previously obtained data on the dependence on the structure of the neurotropic activity of sulfonamides from the other cage-like amines [8, 11] we applied predominantly aromatic sulfonyl chlorides **IIa–k**.





Ar = C_6H_4F-*p* (**IIa**), C_6H_4Cl-*p* (**IIb**), C_6H_4Br-*p* (**IIc**), $C_6H_4NO_2$-*p* (**IId**), $C_6H_4NO_2$-*m* (**IIe**), $C_6H_3(NO_2)_2$-*o,p* (**IIf**), $C_6H_3OCH_3$-*o*), NO_2-*m′* (**IIg**), $C_6H_3CH_3$-*o*), NO_2-*p* (**IIh**), C_6d3NO_2-*o*), CH_3-*p* (**IIi**), $C_6H_2(i\text{-Pr})_3$-*o,o′,p* (**IIj**), $C_6H_4OCH_3$-*p* (**IIk**).

From amine **I** and toluene-3,4-disulfonyl dichloride sulfonamide **IIl** was obtained.

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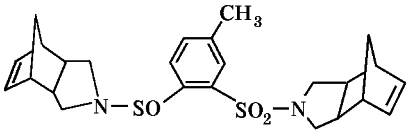
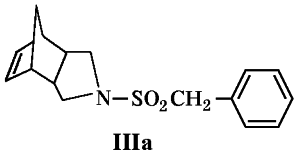
Table 1. Yields, melting points, IR spectra, and elemental analyses of compounds **IIa–l, IIIa–c**

Compd. no.	Yield, %	mp, °C	IR spectrum, cm^{-1}	Found,%			Formula	Calculated,%		
				C	H	N		C	H	N
IIa	80	104–105	3066, 3050, 1591, 1494, 1346, 1324, 1170, 1153, 713				$H_{16}FNO_2S$	61.43	5.46	4.78
IIb	81	101–102					$C_{15}H_{16}ClNO_2S$	58.16	5.17	4.52
IIc	80	103–104	3070, 1569, 1469, 1340, 1168, 1143, 1011, 793, 748				$C_{15}H_{16}BrNO_2S$	50.85	4.52	3.95
IId	93	178–179	3065, 1527, 1463, 1348, 1167, 738	56.38	4.94	8.85	$C_{15}H_{16}N_2O_4S$	56.25	5.00	8.75
IIe	85	115–116					$C_{15}H_{16}N_2O_4S$	56.25	5.00	8.75
IIf	79	88–90	1563, 1478, 1380, 1359, 1183, 1074, 730	49.41	4.21	11.59	$C_{15}H_{15}N_3O_6S$	49.32	4.11	11.51
IIg	78	155–156	3085, 1530, 1492, 1352, 1340, 1172, 1157, 1076, 723	54.98	5.22	8.09	$C_{16}H_{18}N_2O_5S$	54.86	5.14	8.00
IIh	82	107–108	3092, 1535, 1488, 1357, 1334, 1170, 1148, 1089, 758	57.57	5.49	8.47	$C_{16}H_{18}N_2O_4S$	57.49	5.39	8.38
IIi	77	111–112		57.54	5.47	8.50	$C_{16}H_{18}N_2O_4S$	57.49	5.39	8.38
IIj	74	157–158	3078, 1610, 1481, 1370, 1335, 1170, 1079, 721	71.74	8.84	3.57	$C_{24}H_{35}NO_2S$	71.82	8.73	3.49
IIk	79	67–68					$C_{16}H_{19}NO_3S$	62.95	6.23	4.59
III	83	184–186	3060, 1483, 1356, 1179, 1153, 1088, 722	61.68	6.26	5.69	$C_{25}H_{30}N_2O_4S_2$	61.73	6.17	5.76
IIIa	76	133–135	3069, 1598, 1491, 1330, 1150, 1060, 705				$C_{16}H_{19}NO_2S$	66.44	6.57	4.84
IIIb	57	90–91	3083, 1478, 1325, 1155, 1142, 1095, 720	64.16	8.09	5.05	$C_{15}H_{23}NO_2S$	64.06	8.18	4.98
IIIc	69	Oily substance	3065, 1325, 1158, 1057, 723				$C_{12}H_{19}NO_2S$	59.75	7.88	5.81

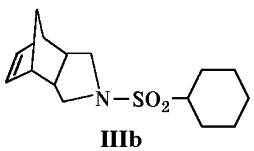
For the sake of comparison we carried out also reactions with benzyl-, cyclohexyl-, and propylsulfonyl chlorides which furnished compounds **IIIa–c**.



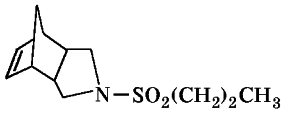
III



IIIa



IIIb



IIIc

The characteristics of sulfonamides obtained and the data of their IR spectra are compiled in Table 1. All compounds synthesized contain a strained double bond appearing in the IR spectra as absorption bands in the regions 1575–1550, 3065–3040 and 735–700 cm^{-1} (ν C=C, ν =C–H, δ =C–H respectively) [9]. The unusual position of the first band is due to the strain in the double bond leading to decrease in the factor of the kinematic interaction of C=C and

Table 2. ^{1}H NMR spectra δ, ppm, coupling constants, Hz

Compd. no.	H^{8}, H^{9}	H^{1}, H^{7}	H^{2}, H^{6}	H^{3A}, H^{3B}	H^{5A}, H^{5B}	H^{10S}, H^{10A}	H arom
I	6.20	2.82	2.70	2.68, 2.57, ^{2}J 11.7	2.68, 2.57	1.46, 1.42, ^{2}J 7.8	–
IIa	5.93	2.72	2.76	3.07, 2.78	3.07, 2.78	1.46, 1.31, ^{2}J 8.5	7.70, 7.13
IIb	5.95	2.72	2.82	3.05, 2.83	3.05, 2.83	1.45, 1.35	7.68, 7.56
IIc	5.98	2.75	2.80	3.07, 2.82	3.07, 2.82	1.48, 1.35	7.70, 7.55
IId	5.94	2.79	2.79	3.10, 2.85	3.10, 2.85	1.48, 1.32, ^{2}J 8.5	8.30, 7.86
IIe	5.97	2.79	2.80	3.13, 2.82	3.13, 2.82	1.47, 1.34	8.45–7.88
IIg	6.08	2.82	2.85	3.29, 2.97	3.27, 3.95	1.50, 1.36	8.49–7.38
IIh	6.17	2.84	2.86	3.28, 3.00	3.28, 3.00	1.55, 1.45	8.46–7.62
IIj	6.12	2.68	2.84	3.19, 2.92	3.19, 2.92	1.57, 1.47	7.07
IIk	5.99	2.65	2.80	3.03, 2.82	2.97, 2.82	1.50, 1.38	7.55, 6.98
IIIb	6.34	2.88	2.88	3.25, 2.96	3.25, 2.96	1.50, 1.42	–

Table 3. ^{13}C NMR spectra of N-substituted 4-azatricyclo[5.2.1.$0^{2,6}$]dec-8-enes and their epoxy derivatives, δ, ppm.

Compd. no.	C^{8}, C^{9}	C^{1}, C^{7}	C^{2}, C^{6}	C^{3}, C^{5}	C^{10}	C arom
IIa	135.3	45.9	46.3	50.5	52.6	166.5, 63.9, 30.4, 16.4
IId	135.8	45.9	46.4	50.6	52.6	150.2, 142.6, 128.8, 124.3
IIg	135.8	46.2	46.5	50.5	52.5	161.6, 140.7, 128.9, 129.7, 127.4, 112.5
IVa	49.6	40.8	44.1	48.2	29.6	166.7, 164.1, 130.7, 116.6
IVc	49.6	40.8	44.1	48.3	29.6	140.4, 134.1, 132.5, 129.4
IVd	49.4	40.8	44.1	48.3	29.6	150.4, 141.2, 129.1, 124.5
IVf	49.7	40.5	44.2	48.1	29.9	153.4, 151.6, 136.2, 124.1
IVg	49.3	40.7	44.7	48.1	20.8	130.7, 129.3, 128.9
IVh	49.4	40.9	44.8	48.2	29.8	–

C–C bonds that is proportional to the cosine of the angle between them. This band is of low intensity because of the high degree of symmetry in the fragment. Both the first and the second of the mentioned bands are overlapped by the absorption bands of the benzene ring. The characteristic absorption band of the norbornene double bond is observed in the region 730–700 cm^{-1} in contrast to the other unsaturated systems. In the spectra of sulfonamides the bands of N–H groups are lacking, and are present the absorption bands of sulfonyl group 1350–1320 and 1170–1140 cm^{-1}) and of substituents in the benzene ring: nitro group (1560–1520 and 1375–1335 cm^{-1}) and methoxy group (2850 cm^{-1}) [12].

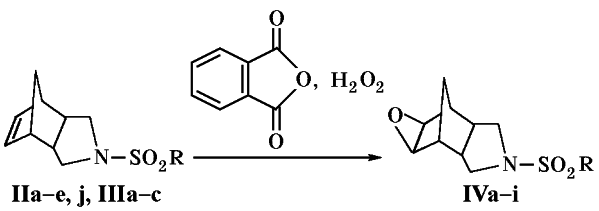
To confirm the structure of compounds we measured the ^{1}H and ^{13}C NMR spectra. In Table 2 are listed the parameters of proton spectra of compounds **IIa–k, IIIb**, and for comparison also that of amine **I**.

Molecules of all compounds are in a symmetrical or nearly symmetrical conformation where the chemical shifts are equal in the pairs of protons H^{8} and H^{9}, H^{1} and H^{7}, H^{2} and H^{6}, H^{3} and H^{5}. In the molecule of amine **I** and its derivatives the protons in the methylene groups at C^{3} and C^{5} carbons (H^{3A} and H^{3B}, H^{5A} and H^{5B}) are nonequivalent. The bridging protons H^{10s} and H^{10a} appear in an AB-system pattern, and their nonequivalence in the sulfonamide molecules is considerably more pronounced than in amine **I**. The comparison with the spectrum of amine **I** showed that the introduction of an electron-withdrawing sulfonyl group and electron-withdrawing substituents into the benzene ring first of all affected the proton signals in the neighboring methylene groups at C^{3} and C^{5} carbons, and also signals of protons attached to C^{2} and C^{6} carbons further on along the carbon–carbon chain.

Table 4. Yields, melting points, IR spectra, and elemental analyses of epoxy derivatives **IVa**–**h**

Compd. no.	Yield, %	mp, °C (2-propanol)	IR spectrum, cm^{-1}	Found,%			Formula	Calculated,%		
				C	H	N		C	H	N
IVa	71	172.5–173.5	3065, 3034, 1588, 1492, 1346, 1152, 853	58.19	5.08	4.55	$C_{15}H_{16}FNO_3S$	58.25	5.18	4.53
IVb	87	162–163				4.27	$C_{15}H_{16}ClNO_3S$	55.30	4.92	4.30
IVc	72	157–158	3050, 3037, 1573, 1345, 1328, 1167, 1025, 852	48.77	4.42	3.85	$C_{15}H_{16}BrNO_3S$	48.65	4.32	3.78
IVd	85	216–218	3066, 3026, 1528, 1349, 1302, 1172, 1152, 853	53.52	4.70	8.28	$C_{15}H_{16}N_2O_5S$	53.57	4.76	8.33
IVe	73	176–177				8.29	$C_{15}H_{16}N_2O_5S$	53.57	4.76	8.33
IVf	74	190–191	3040, 1603, 1363, 1329, 1164, 1037, 855			3.42	$C_{24}H_{35}NO_3S$	69.06	8.39	3.36
IVg	95	164–165	3032, 1329, 1168, 1144, 1040, 848			4.57	$C_{16}H_{19}NO_3S$	62.95	6.23	4.59
IVh	71	159–160	3045, 1318, 1135, 1049, 855	60.63	7.61	4.77	$C_{15}H_{23}NO_3S$	60.61	7.74	4.71

Parameters of ^{13}C NMR spectra of sulfonamides **IIa, d, g** are listed in Table 3; the assignment was performed by Rabenstein and Nakashima procedure. The olefin carbons give rise to signals at 135.8 ppm; as in the proton spectra, all "twin" nuclei have signals with equal chemical shifts; the most downfield signals correspond to bridging carbons (52.5–52.6 ppm) and to C^3 and C^5 (50.5 ppm). The obtained sulfonamides containing a strained double bond [9] were subjected to reactions resulting in this bond functionalization, with peracids and aryl azides. As epoxidizing agent we chose the perphthalic acid *in statu nascendi* prepared from phthalic anhydride and 30% water solution of hydrogen peroxide. The reaction was carried out in ethyl acetate in the presence of carbamide used to adjust the proton-donor and proton-acceptor quality of the medium [13]. The reaction progress was monitored by TLC.



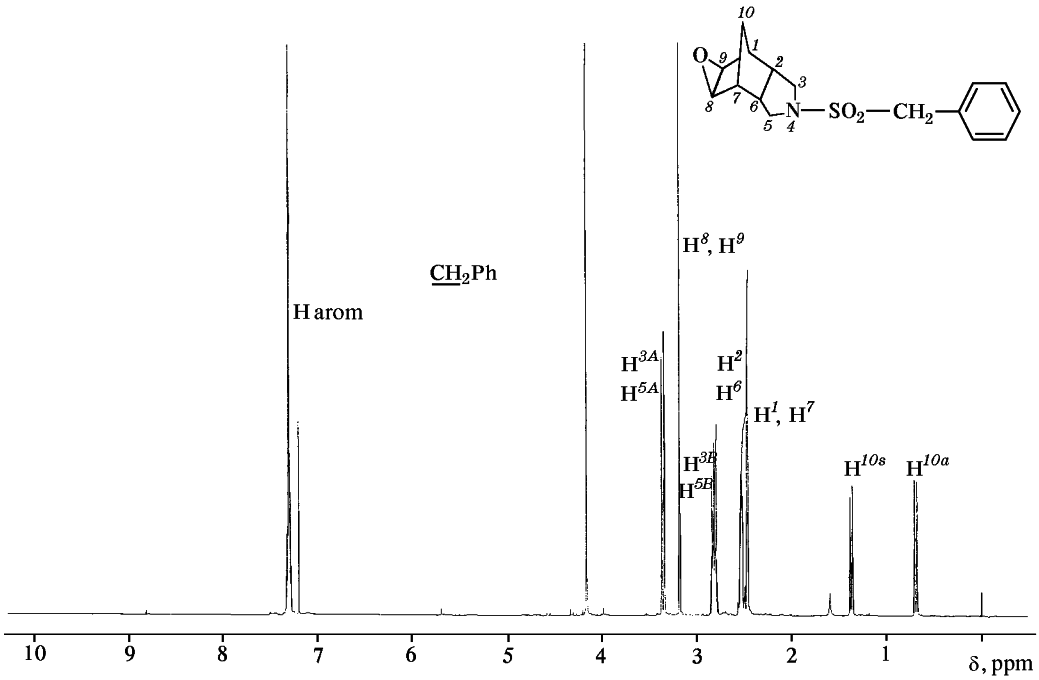
IIa–e, j, IIIa–c **IVa–i**

R = C_6H_4F-*p* (**IVa**), C_6H_4Cl-*p* (**IVb**), C_6H_4Br-*p* (**IVc**), $C_6H_4NO_2$–*p* (**IVd**), $C_6H_4NO_2$-*m* (**IVe**), $C_6H_2(i\text{-}Pr)_3$-*o*,*o'p* (**IVf**), $CH_2C_6H_5$ (**IVg**), C_6H_{11}-*cyclo* (**IVh**), C_3H_7 (**IVi**).

The characteristics of the epoxides obtained are given in Table 4. All compounds prepared except derivative **IVi** are crystalline. The IR spectra contain absorption bands in the region 855–848 and 3045–3020 cm^{-1} (ν C–O and νC–H in epoxynorbornanes [10, 14]). No absorption was observed in the range 3400–3200 cm^{-1} (ν OH, ν NH), and appeared bands characteristic of SO_2 group (1350–1320 and 1175–1135 cm^{-1}) and of bonds in the benzene ring and its substituents, in particular, of nitro group (1560–1520 and 1340–1305 cm^{-1}) [12].

The structure of epoxides was unambiguously confirmed by their ^1H and ^{13}C NMR spectra (Tables 3, 5, figure). The signals were assigned with the help of two-dimensional spectra COSY and HECTOR. The ^1H NMR spectra of epoxides **IVa, c, d, f–h** lack signals at 6 ppm, and at 3.14–3.38 ppm appear signals of the protons of the epoxy ring sensitive to a certain extent to the character of the solvent. The shift of the signal belonging to one of the bridging hydrogens (H^{10A}) to 0.7–0.8 ppm confirms theexo-orientation of the epoxy ring [15].

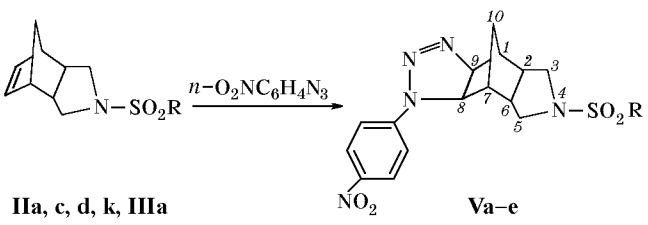
In the ^{13}C NMR spectra (Table 3) appear the signals of carbon atoms from the epoxy ring (C^8, C^9) in the region 49.3–49.7 ppm. In keeping with [16] the signals of the bridging carbons (C^{10}) are shifted upfield (29.6–29.9 ppm) compared to the unsaturated compounds. This shift is an additional proof of the *exo*-orientation of the epoxy ring. In the ^{13}C NMR spectra are also observed the resonances of carbon



^{1}H NMR spectrum of N-benzylsulfonyl-exo-8,9-epoxy-4-azatricyclo-[5.2.1.0^{2,6}]decane.

atoms belonging to the substituents (substituted benzene rings and a cyclohexyl moiety).

Reactions of compound **I** with azides were not studied before in contrast to the first member of the series, norbornene [17]. Due to the high strain in the double bond the latter readily reacted with various azides, and depending on the type of azide the reactions afforded substituted triazolines, aziridines, or products of their subsequent transformations. Among these substances biologically active compounds were revealed [18]. The reaction mechanism of norbornene and its derivatives involved formation of a five-membered activated complex, and these processes could be classed among [3+2]cycloadditions. Unlike norbornene the disubstituted norbornenes were seldom brought into reactions with azides [18, 19].



IIa, c, d, k, IIIa **Va–e**

Ar = C_6H_4F-*p* (**Va**), C_6H_5Br-*p* (**Vb**), $C_6H_4NO_2$-*p* (**Vc**), $C_6H_2(i\text{-}Pr)_3$-*o,o′,p* (**Vd**), $CH_2C_6H_5F$ (**Ve**).

The sulfonamides synthesized were brought into reaction with equimolar amount of *p*-nitrophenyl azide at reflux in chloroform (TLC monitoring).

The characteristics of triazolines Va–e are listed in Table 6. In the IR spectra of the compounds alongside the absorption bands originating from the structure of the initial tricyclic alkenes appeared new bands in the regions 1520–1500 and 1350–1320 cm^{-1} belonging to nitro groups [12], and also medium bands at 1595 cm^{-1} presumably corresponding to stretching vibrations of unsymmetrically substituted N=N band of the triazoline moiety [20]. The structure of compounds **Va, c–e** was supported by the ^{1}H NMR spectra (Table 5) containing doublets in the region 4.90–4.70 and 4.80–4.05 ppm with a vicinal coupling constant 9 Hz corresponding to protons H^8, H^9 of the triazoline moiety [21]. The shift of one of the bridging protons (H^{10A}) signal to 0.95–0.85 ppm evidences *exo*-orientation of the triazoline moiety.

EXPERIMENTAL

IR spectra were recorded on spectrometer Specord IR75 from samples pelletized with KBr. ^{1}H NMR spectra were registered on spectrometers Varian VXR-300 and Inova-400 at operating frequencies 300 and 400 MHz respectively from solutions in deutero-

Table 5. ^{1}H NMR spectra of compounds **IVa, c, d, f–h, Va, c–e,** δ, ppm, coupling constants, Hz

Compd. no.	H^8, H^9	H^1, H^7	H^2, H^6	H^{3A}, H^{3B}	H^{3A}, H^{3B}	H^{10S}, H^{10A}	H arom
IVa	3.25	2.52	2.52	3.43, 2.64	3.41, 2.62	1.40, 0.70, ^2J 10.2	7.72, 7.15
IVc	3.25	2.52	2.52	3.44, 2.63	3.42, 2.60	1.40, 0.70, ^2J 10.1	7.71, 7.56
IVd	3.21	2.50	2.59	3.39, 2.78	3.39, 2.76	1.21, 0.74, ^2J 9.6	8.44, 8.00
IVf	3.14	2.50	2.66	3.27, 2.89	3.25, 2.89	1.40, 0.76, ^2J 10.1	7.09
IVg	3.18	2.46	2.53	3.36, 2.83	3.34, 2.80	1.37, 0.69, ^2J 9.9	7.30
IVh	3.38	2.62	2.72	3.59, 3.23	3.59, 3.23	1.49, 0.82, ^2J 9.8	–
Va	4.90, 4.04, $^3J_{8,9}$ 9.0	2.46	2.58	3.76, 2.77, $^2J_{3,5}$ 10.3	3.56, 2.75	1.46, 0.93, ^2J 9.9	8.20–7.50
Vc	4.92, 4.05, $^3J_{8,9}$ 9.0	2.32	2.60	3.80, 2.98	3.63, 2.98	1.44, 0.94, ^2J 10.0	8.40–8.00
Vd	4.70, 3.97, $^3J_{8,9}$ 8.7	2.68	2.80	3.64, 3.02, $^2J_{3,5}$ 10.1	3.45, 2.99	1.32, 0.94, ^2J 11.0	8.10, 7.39, 7.19
Ve	4.72, 3.77, $^3J_{8,9}$ 8.8	2.46	2.66	3.61, 3.04, $^2J_{3,5}$ 10.5	3.49, 2.97	1.27, 0.84, ^2J 10.8	8.18, 7.42, 7.24

Table 6. Yields, melting points, IR spectra, and elemental analyses of triazolines **Va–e**

Compd. no.	Yield, %	mp, °C	IR spectrum, cm^{-1}	Found, N, %	Formula	Calculated, N, %
Va	79	235–237 (decomp.)	1595, 1505, 1354, 1320, 1176, 1085, 838	15.36	$C_{21}H_{20}FN_5O_4S$	15.32
Vb	78	234–236 (decomp.)		13.45	$C_{21}H_{20}BrN_5O_4S$	13.51
Vc	77	240–242 (decomp.)	1584, 1520, 1500, 1348, 1318, 1180, 1089, 850	17.48	$C_{21}H_{20}N_6O_6S$	17.36
Vd	76	207–209 (decomp.)	1595, 1501, 1375, 1324, 1165, 1077, 844	12.46	$C_{30}H_{39}N_5O_4S$	12.39
Ve	85	193–195 (decomp.)	1596, 1501, 1378, 1335, 1172, 1147, 844	15.34	$C_{22}H_{23}N_5O_4S$	15.45

chloroform, internal reference HMDS. ^{13}C NMR spectra were measured on spectrometer Inova-400 at operating frequency 100.6 MHz. Some spectra were registered by COSY and HECTOR procedures. The reaction progress was monitored and the purity of compounds synthesized was checked by TLC on Silufol UV-254 plates, eluent ether, development in iodine vapor. Elemental analysis was carried out on Karlo Erba analyzer.

4-Azatricyclo[5.2.1.0^{2,6}]dec-8-ene (**I**) was prepared by procedures [3, 6]; the characteristics of compound obtained were in agreement with the published data.

N-[Aryl(benzyl, cyclohexyl, propyl)sulfonyl]-4-azatricyclo[5.2.1.0^{2,6}]dec-8-enes (**IIa–k, IIIa–c**). To a stirred mixture of 0.3 g (2.2 mmol) of amine **I** and 0.4 ml (2.2 mmol) of 20% water solution of sodium hydroxide in 15 ml of ether was added dropwise a solution of 2.2 mmol of an appropriate aryl(cyclohexyl, benzyl, propyl)sulfonyl chloride in 10 ml of ether. The reaction mixture was stirred at room temperature till completion of the reaction (TLC monitoring). The solvent was removed, the residue was dissolved in 20 ml of chloroform–water mixture (1 : 1), the organic layer was separated, dried with calcined magnesium sulfate, the solvent was removed, and the residue was recrystallized from 2-propanol. The physical properties and spectral parameters of compounds **IIa–l, IIIa–c** are given in Tables 1–3.

***N*-[Aryl(benzyl, cyclohexyl, propyl)sulfonyl]-*exo*-8,9-epoxy-4-azatricyclo[5.2.1.0^{2,6}]decanes (IVa–i).** To a mixture of 0.6 mmol of an appropriate sulfonamide, 0.18 g (1.2 mmol) of phthalic anhydride,

and 0.02 g (0.3 mmol) of urea in 15 ml of ethyl acetate was added at room temperature under stirring 0.1 ml (1.2 mmol) of 35% water solution of hydrogen peroxide. The stirring was continued till completion of the reaction (TLC monitoring). On neutralizing the reaction mixture with saturated solution of sodium hydrogen carbonate the organic layer was separated, dried with calcined magnesium sulfate, the solvent was removed, and the residue was recrystallized from 2-propanol. The physical properties and spectral parameters of compounds **IVa–i** are given in Tables 4, 5.

Products of reaction between sulfonamides and aryl azides Va–e. A mixture of 1 mmol of an appropriate sulfonamide and 0.16 g (1 mmol) of *p*-nitrophenyl azide was heated to reflux in 10 ml of chloroform till the completion of reaction (TLC monitoring). The separated precipitate was filtered off and recrystallized from aqueous acetone. The physical properties and spectral parameters of compounds **Va–e** are presented in Tables 5, 6.

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