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Asymmetrical Oxidation of Menthone Dithiolane

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Abstract—Asymmetrical oxidation was performed of menthone dithiolane obtained in 95–98% yield by condensation of menthone with 1,2-ethanedithiol in the presence of boron trifluoride etherate. 6-Isopropyl-9-methyl-1,4-dithiaspiro[4,5]decane-1,4-dioxide (yield 5–55%) and 6-isopropyl-9-methyl-1,4-dithiaspiro[4,5]-decane-1,1,4-trioxide (yield 65–70%) were synthesized. Chemical structures of compounds obtained were proved by XRD analysis, NMR and IR spectroscopy.

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Nowadays an urgent problem exists of asymmetrical oxidation for preparation of optically active sulfoxides required in various chemical transformations in asymmetrical synthesis and in production of physiologically active substances. Several main approaches are known to the synthesis of chiral sulfoxides of high enantiomeric purity: optical separation of achiral sulfoxides, asymmetrical synthesis where a sulfo group is introduced into an optically active compound, and an asymmetrical oxidation of the corresponding sulfides [1–3]. One of the methods of chiral sulfoxides preparation consists in the oxidation of a chiral sulfide. Chiral sulfoxides can be prepared from dithiolanes. Published examples of asymmetrical dithiolanes oxidation describe the use of Sharpless reagent [4], catalysts based on vanadium(IV) and other metals, enzyme preparations and purified enzymes [1–4].

Terpene dithiolanes (ketals) and their oxidation products, sulfoxides and sulfones, are of practical interest as presumably physiologically active compounds that can find application for production of pharmaceuticals [5–7], pesticides, perfumes [5], flotators [8], plasticizers, detergents [5], extraction reagents [9]. Dithiolane and oxathiolane rings are present in the structure of nucleosides with antiviral action [10]. These nucleoside analogs containing in the ring more than one heteroatom are of great interest because of their powerful anti-HIV and anti-HBV action [11]. This class of compounds is also known to possess fungistatic activity toward fungi of genera *Rodotorula Rubra, Penicillium chrisogenum, Aspergillus fumigatus,* and also toward nonpathogenic fungi of the genus *Candida Parapsilosis* [12].

In this study we carried out the asymmetrical oxidation of optically active menthone dithiolate in order to prepare a chiral disulfoxide and a chiral sulfenyl-sulfonyl derivative.

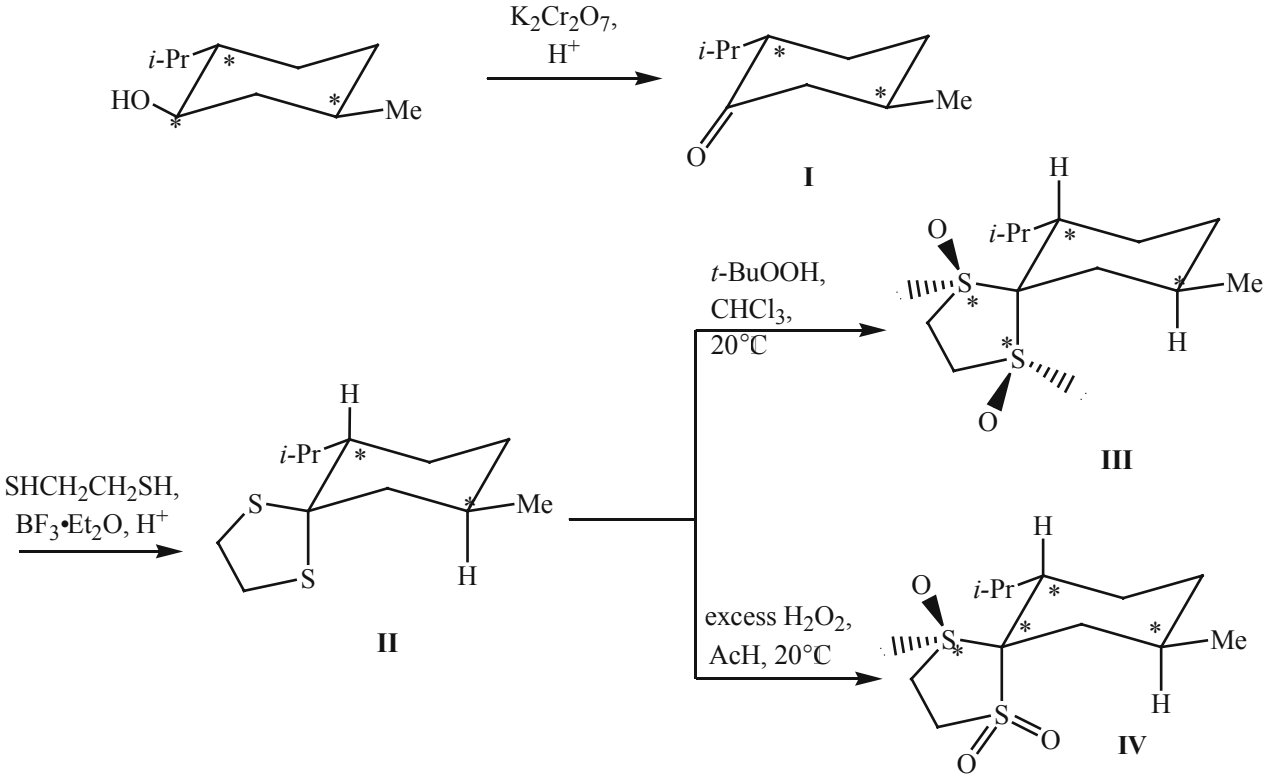
The most convenient method of thiolanes preparation consists in the reaction of a ketone with 1,2-ethanedithiol in the presence of the boron trifluoride etherate [13–16].

These reactions of dithiolane preparation are used in the preparative organic chemistry for temporary blocking a carbonyl group. The keto group is easily recovered from the ketal by hydrolysis with dilute mineral acid [13, 17].

The applied initial terpene ketone was *dl*(1*R*,4*S* and 1*S*,4*R*)- (**Ia**) or *l*(1*R*,4*S*)-menthone (**Ib**) that was obtained by an oxidation of commercial *dl*- or *l*-menthol, $[\alpha]_D$ +50° (*C* 10.0, EtOH), with potassium bichromate in acid medium by procedure [18]. Yields of compounds **Ia** and **Ib** were 94 and 96% respectively.

The reaction of equimolar quantities of *dl*- or *l*-menthone **Ia** or **Ib** and 2-ethanedithiol in acetic acid in the presence of Lewis catalyst $BF_3 \cdot Et_2O$ led to the formation of *dl*-menthone dithiolane (**IIa**) or *l*-menthone dithiolane (**IIb**) in 95 and 98% yield respectively (see

Scheme.



the scheme). In the IR spectrum of compounds **IIa** and **IIb** the disappearance was observed of the carbonyl group absorption in the region 1720–1760 cm^{-1} and the appearance of a characteristic absorption band at 880 cm^{-1} corresponding to the thioether bond of the dithiolane ring. The formation of compounds **IIa** and **IIb** was proved by GC-MS method. The ^{1}H and ^{13}C NMR spectra of compounds **IIa** and **IIb** are similar to those described in [19].

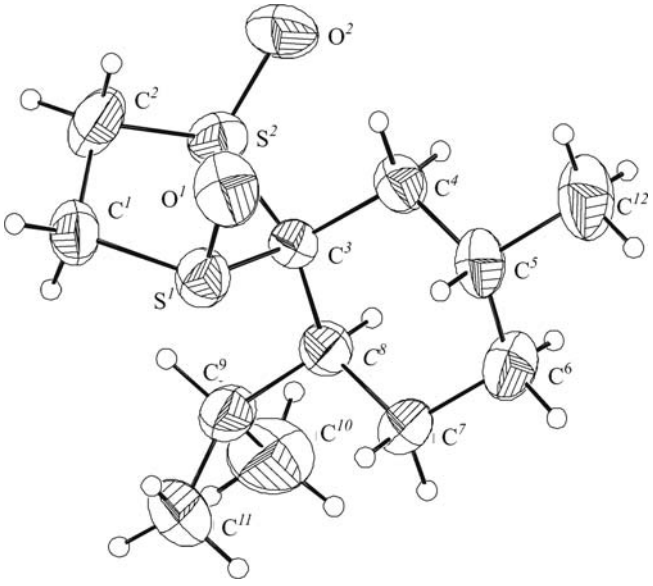


Fig. 1. General view of molecule (1*S*,4*R*,5*S*,6*S*,9*R*)-6-isopropyl-9-methyl-1,4-dithiaspiro[4,5]decane-1,4-dioxide (**IIIb**) according to XRD data.

In the synthesis of sulfoxides and sulfones from the menthone dithiolane hydrogen peroxide or *tert*-butyl hydroperoxide were used. By oxidation of racemic menthone dithiolane (**IIa**) with H_2O_2 at the ratio substrate–oxidant 1:2 we obtained a racenic sulfinyl-sulfonyl derivative **IVa**, $[\alpha]_D$ 0° (*C* 0.52, EtOH), in 52% yield. The structure of compound **IVa** was established by XRD method.

The oxidation of *l*-menthone dithiolane (**IIb**) with *t*-BuOOH at the ratio substrate–oxidant 1:1 provided disulfoxide (**IIIb**), $[\alpha]_D$ –37.6° (*C* 0.4, EtOH), in 50–55% yield (*d.e.* >95%) after crystallization from EtOH. In the IR spectrum of compound **IIIb** a characteristic absorption band of the stretching vibrations of the sulfoxide group ν(SO) was observed at 1054 cm^{-1}. The final proof of the structure of the reaction product was obtained by XRD analysis on a single crystal. The obtained crystals of compound **IIIb** belong to monoclinic

crystal system and chiral space group of $P2_1$ symmetry. Methyl and isopropyl groups of the cyclohexane ring in the molecule of compound **IIIb** are located equatorially. Oxygen atoms are placed in pseudoaxial positions with respect to the dithiolane ring and are turned in the direction of the isopropyl group (Fig. 1). The molecular packing of the compound occurs by formation of chains of molecules **IIIb** in the crystal; therewith the oxygen atoms of the SO groups formed short inter-molecular contacts with the methylene groups of the spiroring (Fig. 2).

The oxidation of compound **IIb** at the ratio substrate–oxidant 1:0.5 (H_2O_2, *t*-BuOOH) and 1:1 (H_2O_2) led to the formation of disulfoxide **IIIb** that was identified by TLC (R_f 0.05, C_6H_{14}–Et_2O, 1:2) and IR spectrum [ν(SO) 1054 cm^{-1}) compared with an authentic sample of compound **IIIb**.

Further oxidation of S^1 atom located in the axial position with respect to the cyclohexane ring is hampered by the steric effect of the neighboring isopropyl group. This steric effect should be especially important for *t*-BuOOH since the oxidant proper also contains a bulky *tert*-butyl group.

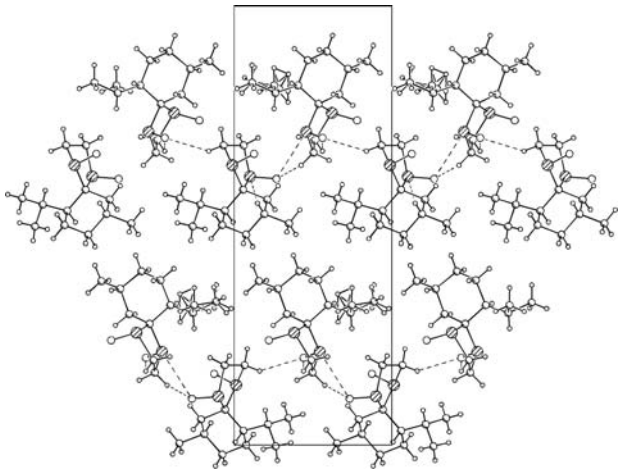


Fig. 2. Crystal packing of (1S,4R,5S,6S,9R)-6-isopropyl-9-methyl-1,4-dithiaspiro[4,5]decane-1,4-dioxide (**IIIb**).

The oxidation of dithiolane **IIb** with excess H_2O_2 (the ratio substrate–oxidant 1:2, 1:4) resulted in sulfinyl-

Main parameters of XRD experiments

Compound	**IVb**	**IVa**	**IIIb**
Empirical formula	$C_{12}H_{22}O_3S_2$	$C_{12}H_{22}O_3S_2$	$C_{12}H_{22}O_2S_2$
Crystal system	Orthorhombic	Monoclinic	Monoclinic
Space group	$P2_12_12_1$	$P2_1/c$	$P2_1$
a, Å	9.2717(3)	8.4576(2)	6.6283(2)
b, Å	10.4891(4)	9.3466(2)	24.0984(7)
c, Å	14.2145(5)	17.8332(4)	8.6954(2)
α, deg	90	90	90
β, deg	90	99.246(2)	95.026(2)
γ, deg	90	90	90
V, Å^3 *Z*	1382.39(8)4	1391.40(5)4	1383.59(7)4
D_{calc}, g/cm^3	1.338	1.329	1.260
μ, mm^{-1}	0.380	0.378	0.370
F(000)	600	600	568
Measured reflexions	6491	12167	9584
independent	2782 (R_{int} 0.0212)	2814 (R_{int} 0.0232)	5497 (R_{int} 0.0295)
Among them with $I>2\sigma(I)$	2175	2378	4266
S by F^2	1.001	1.000	1.003
R_1	0.0313	0.0313	0.0363
wR_2	0.0657	0.0961	0.0745
Maximum and minimum peaks of residual electron density, e/Å^3	0.235 and –0.159	0.290 and –0.285	0.306 and –0.224

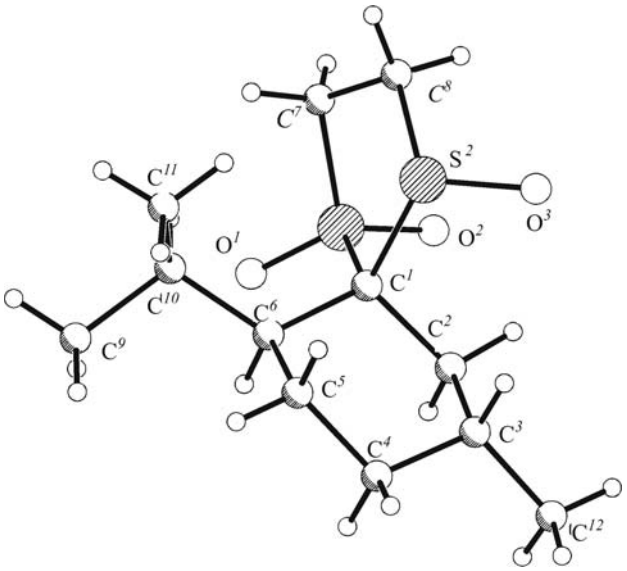


Fig. 3. General view of molecule (4*R*,5*S*,6*S*,9*R*)-6-isopropyl-9-methyl-1,4-dithiaspiro[4,5]decane-1,1,4-trioxide (**IVb**) according to XRD data.

sulfonyl derivative **IVb** $[\alpha]_D$ –23.3° (*C* 0.41, EtOH), in 65–70% yield (*d.e.*>98%). In the IR spectrum of the latter characteristic absorption bands were present of sulfonyl and sulfenyl groups at 1314 [$\nu_{as}(SO_2)$], 1136 [$\nu_s(SO_2)$], 1056 [ν(SO)] cm^{-1}. The structure of compound **IVb** was unambiguously established by XRD analysis. As expected, the oxidation to sulfone group occurred with S^1 atom located in the equatorial position with respect to the cyclohexane ring (Fig. 3).

The IR spectrum of compound **IVa** contained characteristic bands of stretching vibrations of sulfoxide group 1046 [ν(SO)] cm^{-1} and sulfone group 1314 [$\nu_{as}(SO_2)$], 1128 [$\nu_s(SO_2)$] cm^{-1}.

The oxidation product of racemic dithiolane **IVa** formed monoclinic crystals belonging to the centrosymmetrical space group $P2_1/c$, whereas the oxidation product of the enantiomerically pure menthone dithiolane **IVb** possessed crystals of orthorhombic crystal system with the chiral space group of $P2_12_12_1$ symmetry.

EXPERIMENTAL

IR spectra were recorded on a spectrophotometer Specord M80 in cells with a thickness of the absorbing film 0.2 mm in the range of wave numbers 400–4000 cm^{-1} (solutions in CCl_4 for liquids and pellets with KBr for solid substances). Melting points were measured on a Gallenkamp-Sanyo instrument. ^1H and ^{13}C NMR spectra were registered on a spectrometer Bruker DRX 400 (400.13 and 100.62MHz respectively) in $CDCl_3$ using TMS as internal reference. The complete assignment of ^1H and ^{13}C signals was performed applying 2D homo- (^1H–^1H COSY, ^1H–^1H NOESY) and heteronuclear experiments (^1H–^{13}C HSQC, ^1H–^{13}C HMBC).

GLC was carried out on a chromatograph Chrom-5 equipped with a flame-ionization detector, column 2000×4 mm, stationary phase 5% Carbowax-20 on Chromaton-N-AW-DMCS, carrier gas helium, ramp from 50 to 250°C at a rate 6 deg/min.

The optical rotation was measured on an automatic digital polarimeter Kruss P3002RS. Mass spectra were registered on a GC-MS instrument Finnigan Trace DSQ, column TR 5MS (30 m), ramp 50–220°C at a rate 4 deg/min. TLC was carried out on Silufol and Sorbfil plates using solvent system C_6H_{14}–Et_2O, spots were visualized with alcoholic vanillin solution and 5% $KMnO_4$ solution. Elemental analysis was performed on an automatic analyzer EA 1110 CHNS-O.

X-ray diffraction analysis was performed on an automatic diffractometer Xcalibur-3 with a CCD-detector (Oxford-Diffraction). Sampling was carried out by the standard procedure using the program CrysAlis Pro, the structures were solved by the direct method by SHELXS-97 software and refined by the program SHELXL-97, by F^2. The experiments were carried out at 295(2) K, ω-scanning, scanning step 1°, wave length of radiation used λ 0.71073 Å (MoK_α), no correction for extinction was done. The data of XRD studies are deposited into the Cambridge Structural Database (CCDC) under numbers 653258–653260 and are freely available (www.ccdc.cam.ac.uk/data_request/cif). The main experimental parameters are compiled in the table.

(1*R*,4*S*)-1-Methyl-4-isopropylcyclohexanone (Ib) was obtained by oxidation of *l*-menthol, $[\alpha]_D$ +50° (*C* 10.0, EtOH), by procedure [18]. Yield 96%, R_f 0.60 (C_6H_{14}– Et_2O, 1:2), $[\alpha]_D$ –28.6° (*C* 1.1, EtOH) ($[\alpha]_D$ –28 ± 1°, liquid [20]). IR spectrum, ν, cm^{-1}: 1715 (C=O). ^1H and ^{13}C NMR spectra of compound **Ib** are similar to the published spectra of *l*-menthone [20].

***dl*-1-Methyl-4-isopropylcyclohexanone (Ia)** was obtained by oxidation of *dl*-menthol by procedure [18]. Yield 94%, R_f 0.60 (C_6H_{14}–Et_2O, 1:2). IR spectrum, ν, cm^{-1}: 1715 (C=O). ^1H and ^{13}C NMR spectra are analogous to the spectra of compound **Ib**.

(6*S*,9*R*)-6-Isopropyl-9-methyl-1,4-dithiaspiro-[4.5]decane (IIb). To 1 g (6.48 mmol) of *l*-menthone

(**Ib**) dissolved in 50 ml of acetic acid was added at room temperature 0.54 ml (6.48 mmol) of 1,2-ethanedithiol and 0.82 ml (6.48 mmol) of boron trifluoride etherate. The mixture was stirred for 24 h at room temperature. Then the mixture was poured into water, the reaction products were extracted into ether, and the organic extracts were washed with $NaHCO_3$ solution, H_2O, and a saturated NaCl solution, dried with anhydrous Na_2SO_4, ether was distilled off in a vacuum, and the reaction product was subjected to column chromatography (eluent heptane–ether, 50:1). Yield 1.46 g (98%), oily substance, R_f 0.72 (C_6H_{14}–Et_2O, 1:2), $[\alpha]_D$ –13.4° (*C* 0.97, EtOH). IR spectrum, ν, cm^{-1}: 880 (C–S). ^{1}H NMR spectrum, δ, ppm: 0.85 m (1H, H$^{8''}$), 0.89 d (3H, C^{14}H$_3$, *J* 6.2 Hz), 0.93 d (3H, C^{13}H$_3$, *J* 7.0 Hz), 0.96 d (3H, C^{12}H$_3$, *J* 7.0 Hz), 1.23 t.d.d (1H, H$^{7''}$, *J* 13.3, 12.4, 3.3 Hz), 1.47 d.d.d (1H, H^6, *J* 12.4, 3.2, 1.3 Hz), 1.60 d.d (1H, H$^{10''}$, *J* 12.8, 11.7 Hz), 1.65–1.78 m (3H, H$^{7'}$, H$^{8'}$, H^9), 2.16 d.t (1H, H$^{10'}$, *J* 12.8, 2.6 Hz), 2.38 septet d (1H, H^{11}, *J* 7.0, 1.3 Hz), 3.18–3.32 m (4H, C^{2}H$_2$, C^{3}H$_2$). ^{13}C NMR spectrum, δ, ppm: 18.38, 21.82, 24.71 (C^{12}, C^{13}, C^{14}), 26.57 (C^7), 27.70 (C^{11}), 32.22 (C^9), 34.70 (C^8), 38.60 (C^3), 39.11 (C^2), 51.84 (C^6), 55.72 (C^{10}), 74.87 (C^5). Mass spectrum (70 эВ), *m/z* (I_{rel}, %): 230 [*M*] (32), 202 (36), 145 (100), 105 (9), 95 (18), 55 (15). Found, %: C 60.50; H 9.09; S 27.78. $C_{12}H_{22}S_2$. Calculated, %: C 62.61; H 9.57; S 27.83.

In the same way compound **IIa** was obtained. Yield 95%, oily substance, R_f 0.72 (C_6H_{14}–Et_2O, 1:2).

(1S,4R,5S,6*S*,9*R*)-6-Isopropyl-9-methyl-1,4-dithiaspiro[4,5]decane-1,4-dioxide (IIIb). Into a solution of 1 g (4.35 mmol) of compound **IIb** in 50 ml of chloroform was added to equimolar ratio while stirring a solution of *tert*-butyl hydroperoxide in chloroform (*tert*-butyl hydroperoxide was extracted into chloroform from its commercial water solution, and the concentration of resulting solution was determined iodometrically). After 4 h of stirring at room temperature into the reaction mixture was added a water solution of $FeSO_4 \cdot 7H_2O$ and citric acid, and the reaction product was extracted into ethyl ether. The extract was washed with saturated solutions of $NaHCO_3$ and NaCl, and dried with anhydrous Na_2SO_4. On distilling off the solvent the oxidation product was purified by crystallization from a mixture heptane–ether, 2:1. Yield 50–55% (*d.e.*>95%), colorless crystals, mp 123–124°C, R_f 0.05 (C_6H_{14}–Et_2O, 1:2), $[\alpha]_D$ –37.6° (*C* 0.4, EtOH). IR spectrum, ν, cm^{-1}: 1054 (SO). ^{1}H NMR spectrum, δ, ppm: 0.81 d (3H, C^{13}H$_3$, *J* 6.8 Hz), 0.99 d (3H, C^{12}H$_3$, *J* 6.8 Hz), 1.04 d (3H, C^{14}H$_3$, *J* 6.4 Hz), 1.03–1.07 m (1H, H$^{8''}$), 1.35 d.d (1H, H$^{10''}$, *J* 14.3, 12.2 Hz), 1.53 t.d.d (1H, H$^{7''}$, *J* 13.5, 12.5, 3.2 Hz), 1.73 septet (1H, H^{11}, *J* 6.8 Hz), 1.78–1.95 m (3H, H^9, H$^{8'}$, H$^{7'}$), 2.01 d.d.d (1H, H^6, *J* 12.5, 3.2, 1.0 Hz), 2.25 d.d.d (1H, H$^{10'}$, *J* 14.3, 3.3, 2.0 Hz), 2.89 d.d.d (1H, H$^{3''}$, *J* 14.5, 14.3, 5.0 Hz), 3.44 d.d.d (1H, H$^{3'}$, *J* 14.5, 5.0, 2.9 Hz), 3.77 d.d.d (1H, H$^{2''}$, *J* 14.3, 12.9, 5.0 Hz), 3.86 d.d.d (1H, H$^{2'}$, *J* 12.9, 5.0, 2.9 Hz). ^{13}C NMR spectrum, δ, ppm: 18.97 (C^{13}), 22.49 (C^{14}), 23.98 (C^{12}), 25.66 (C^7), 27.87 (C^{11}), 30.64 (C^9), 33.01 (C^{10}), 33.85 (C^8), 45.39 (C^3), 48.24 (C^2), 48.74 (C^6), 88.01 (C^5). Found, %: C 54.30; H 8.48; S 24.43. $C_{12}H_{22}S_2O_2$. Calculated, %: C 54.96; H 8.40; S 24.43.

(4R,5S,6*S*,9*R*)-6-Isopropyl-8-methyl-1,4-dithiaspiro[4,5]decane-1,1,4-trioxide (IVb). To 1 g of menthone dithiolane (**IIb**), dissolved in 20 ml of acetic acid was added at stirring 30% solution of hydrogen peroxide to a molar ratio substrate–oxidant 1:5. After 4 h of stirring at room temperature the reaction mixture was poured into water, and the reaction product was extracted into ethyl ether. The extract was washed with a saturated solution of $NaHCO_3$, H_2O, and dried with anhydrous Na_2SO_4. Ether was distilled off in a vacuum, and the residue was crystallized from ethanol. Yield 65–70%, colorless crystals, mp 144°C, R_f 0.12 (C_6H_{14}–Et_2O, 1:2), $[\alpha]_D$ –23.3° (*C* 0.41, EtOH). IR spectrum, ν, cm^{-1}: 1056 (SO), 1314 [$\nu_{as}(SO_2)$], 1136 [$\nu_s(SO_2)$]. ^{1}H NMR spectrum, δ, ppm: 0.85 d (3H, C^{13}H$_3$, *J* 7.1 Hz), 1.00 d and 1.01d (6H, C^{12}H$_3$, C^{14}H$_3$, *J* 6.7 Hz), 1.01 m (1H, H$^{8''}$), 1.49 d.d (1H, H$^{10''}$, *J* 14.3, 12.3 Hz), 1.55 d.d.d.d (1H, H$^{7''}$, *J* 13.6, 13.0, 12.6, 3.6 Hz), 1.68 m (1H, H^9), 1.86 d.d.d.d (1H, H$^{8'}$, *J* 12.7, 6.0, 3.6, 3.3 Hz), 1.92 d.d.d.d (1H, H$^{7'}$, *J* 13.6, 3.6, 3.4, 3.3 Hz), 2.02 d.d.d (1H, H^6, *J* 12.6, 3.6, 0.8 Hz), 2.21 septet (1H, H^{11}, *J* 7.1 Hz), 2.34 d.d.d (1H, H$^{10'}$, *J* 14.3, 3.1, 2.2 Hz), 3.33 d.d.d (1H, H$^{3''}$, *J* 14.1, 11.2, 6.3 Hz), 3.39 d.d.d (1H, H$^{3'}$, *J* 14.1, 7.2, 4.0 Hz), 3.57 d.d.d (1H, H$^{2''}$, *J* 13.6, 6.3, 4.0 Hz), 3.89 d.d.d (1H, H$^{2'}$, *J* 13.6, 11.2, 7.2 Hz). ^{13}C NMR spectrum, δ, ppm: 19.12 (C^{13}), 22.23 and 23.73 (C^{14} and C^{12}), 25.36 (C^7), 27.63 (C^{11}), 30.76 (C^9), 33.40 (C^8), 35.94 (C^{10}), 42.84 (C^3), 45.30 (C^6), 47.41 (C^2), 85.26 (C^5). Found, %: C 52.20; H 7.99; S 22.17. $C_{12}H_{22}S_2O_3$. Calculated, %: C 51.80; H 7.91; S 23.02.

***dl*-6-Isopropyl-9-methyl-1,4-dithiaspiro[4,5]-decane-1,1,4-trioxide (IVa)** was obtained similarly to **IVb** enantiomer at the molar ratio substrate–oxidant (H_2O_2) 1:2, 1:4. Yield 52%, colorless crystals, mp 141–142°C, R_f 0.12 (C_6H_{14}–Et_2O, 1:2). IR spectrum, ν, cm^{-1}: 1046 (SO), 1314 [$\nu_{as}(SO_2)$], 1128 [$\nu_s(SO_2)$]. Found, %: C 52.00; H 7.72; S 22.38. $C_{12}H_{22}S_2O_3$. Calculated, %: C 51.80; H 7.91; S 23.02. ^{1}H and ^{13}C NMR spectra are analogous to those of compound **IVb**.

The study was carried out under a financial support of the project "Development of semisynthetic and application of natural physiologically active biomolecules for creation of preparations possessing antitumor, antioxidant, and adaptogenic activity" by the Federal target program "Research and development of priority development directions of the scientific and engineering complex of Russia in 2007–2012" (State Contract no. 02.512.11.2025), and also of the program of the President of the Russian Federation on the support of the leading scientific schools (grant NSh-4028.2008.3).

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