Reactions of acceptor thiophene 1,1-dioxides with dienes. Synthesis of bisadducts*

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Cycloaddition of dienes to thiophene dioxides affords mono- and bisadducts, depending on the reaction conditions. Regio- and stereochemical specific features of the cycloaddition were revealed. The thermal rearrangement of the monoadduct was found.

Key words: Diels—Alder reaction, regioselectivity, stereoselectivity, acceptor thiophene 1,1-dioxides, dienes, dienophile, bisadduct.

In recent time, thiophene 1,1-dioxides find increasing use in the synthetic practice. For instance, cycloaddition reactions involving thiophene dioxides as acceptor dienes and different dienophiles provide synthetic routes for diverse carbon- and heterocyclic systems.[1—4]

Earlier studied[3,4] halogen- and alkyl-containing thiophene dioxides have the properties of cyclic dienes and enter in reaction with dienophiles followed by the elimination of sulfur dioxide and formation of cyclohexadiene derivatives. Just this remarkable property of the diene system makes these compounds synthetically attractive. However, similar transformations often require high temperatures and dienophiles capable of participating in reactions with inverse electron demands. These conditions are not always easy to obey for known halogen- and alkyl-containing thiophene dioxides.

Results and Discussion

We have previously developed the methods for syntheses of thiophene 1,1-dioxides containing electron-withdrawing substituents.[5] We also showed that in cycloaddition these substances are active dienophiles contrary to earlier described halogen- and alkyl-containing thiophene dioxides.[6] Thus, depending on the nature of substituents, thiophene dioxides can manifest the properties of both acceptor dienes and dienophiles. For instance, the reactions of 2,5-bis(methylsulfonyl)thiophene 1,1-dioxide with 1,3-dienes complete in 1—2 h at −10 °C and afford the Diels—Alder adducts in high yields. The adduct that formed contains a double bond activated by a sulfonyl group. Therefore, we proposed the secondary addition of dienes to the activated double bond of the formed adduct. This transformation is especially interesting from the viewpoint of synthesis, because this is a method for the preparation of bisadducts with two different dienes. A high difference in temperature of the mono- and biscycloadditions enables one to vary the diene in the second step of cycloaddition and obtain bisadducts with different substituents.

The synthesis of bisadducts on the basis of one dienophile is a rather rare transformation. One of the most known reactions of this type is diene addition to 1,4-benzoquinone.[7,8] Some thiophene dioxides can react with two dienophile molecules with SO_2 elimination to form bisadducts. However, this transformation is restricted by only several examples and does not allow one to vary the diene component.[3,9]

We studied the reactions of isoprene, piperilene, 2,3-dimethylbutadiene, cyclopentadiene, and furan with monoadducts of thiophene dioxides **2a—c** synthesized according to an earlier described procedure[6] (Scheme 1).

It turned out that only adduct **2a** can react with these dienes. The heating of adduct **2a** in a miniautoclave for 8—10 h with dienes in acetonitrile, tetrachloroethylene, toluene, and *o*-dichlorobenzene with temperature variation from 70 to 150 °C gave the target products in yields not higher than 25%. The reaction mixtures containing diene and monoadducts **2b,c** underwent no changes for 12 h at 150 °C using the same solvents, and we isolated only the starting reactants. We elucidated that the best solvent was acetic anhydride, being the efficient medium in the Diels—Alder reactions involving insoluble acceptor dienophiles.[10—12] It turned out that its use in the reactions of **2a** with dienes made it possible to substantially increase the yield of the target bisadducts at 120 °C (Table 1). At

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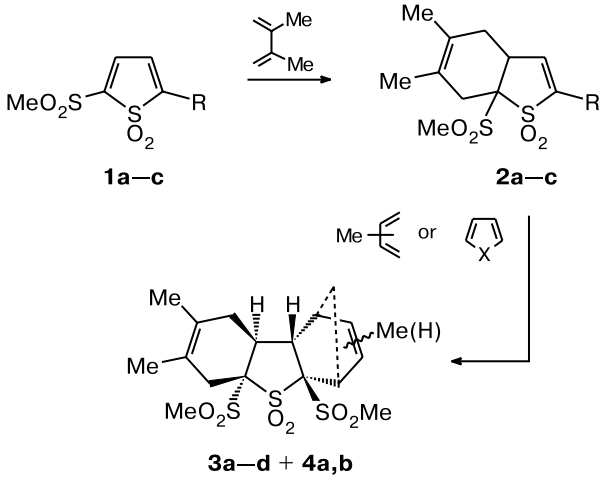
Scheme 1

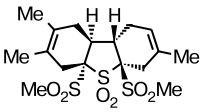
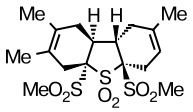
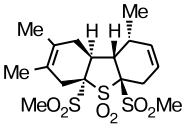
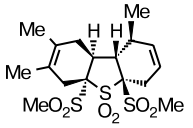
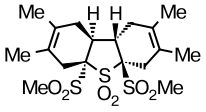
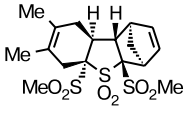


1, 2: R = $MeSO_2$ (**a**), Cl (**b**), Me (**c**)

X = CH_2, O

the same time, the reactions of **2b,c** with dienes in acetic anhydride gave no noticeable amounts of bisadducts even on heating the reaction mixture to 160 °C for 16 h.

Table 1. Yield and ratio of isomers (in parentheses) for bisadducts **3** and **4**

Diene	Bisadducts		Yield (%)
	 **3a**	 **4a**	34 (3 : 2)
	 **3b**	 **4b**	35 (2.5 : 1)
	 **3c**		55
	 **3d**		51

We have previously shown[6] that the Diels—Alder reactions with acceptor thiophene dioxides are chemo-, regio-, and stereoselective. The reaction of **2a** with isoprene afforded a mixture of regioisomers **3a** and **4a**, and that with piperilene gave a mixture of stereoisomers **3b** and **4b**. The ^{1}H NMR spectra of the reaction products show that the reactions of **2a** with 2,3-dimethylbutadiene and cyclopentadiene are stereoselective and afford only bisadducts **3c** and **3d** in 55 and 56%, respectively.

The structure of adduct **3c** was unambiguously determined by X-ray diffraction analysis. The molecular structure of bisadduct **3c** is shown in Fig. 1. In bisadduct **3c**, the six-membered rings are in the *trans*-orientation relative to each other, which agrees well with the concepts that the *trans*-arrangement of the rings is preferential because of lower steric hindrance in a molecule. The *cis*-conjunction of the formed cyclohexene rings in the tricyclic adduct corresponds to the general regularities of the Diels—Alder reaction.[11—13]

The data of X-ray diffraction and ^{1}H NMR spectroscopy indicate the mutual *trans*-orientation of the six-membered rings in all compounds obtained. The relative orientation of the six-membered rings in adducts **3a—d** and **4a,b** was determined from the proton spin-spin coupling constant in the five-membered ring at the C(9a) and C(9b) atoms, which was 5.3 Hz for all bisadducts (Scheme 2). The structure of adduct **3d** was confirmed by NOESY NMR experiment. The characteristic cross-peaks are shown in Scheme 2.

para-Adduct **3a** prevails in the reactions with isoprene, which is well consistent with the known facts.[11—13] The mixture also contains *meta*-regioisomer **4a**, and the *meta*- to *para*-isomer ratio in the resulting mixture is 2 : 3. We determined the isomer ratio and assigned the signals in the spectrum by comparison of the spectrum of monoadduct **2a** with isoprene. We have previously shown[6]

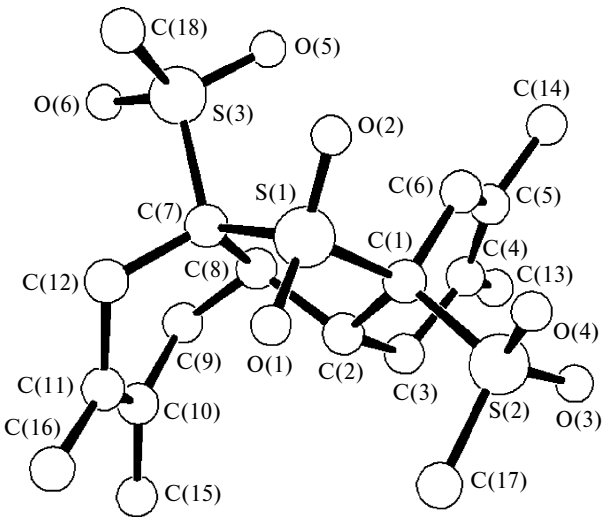
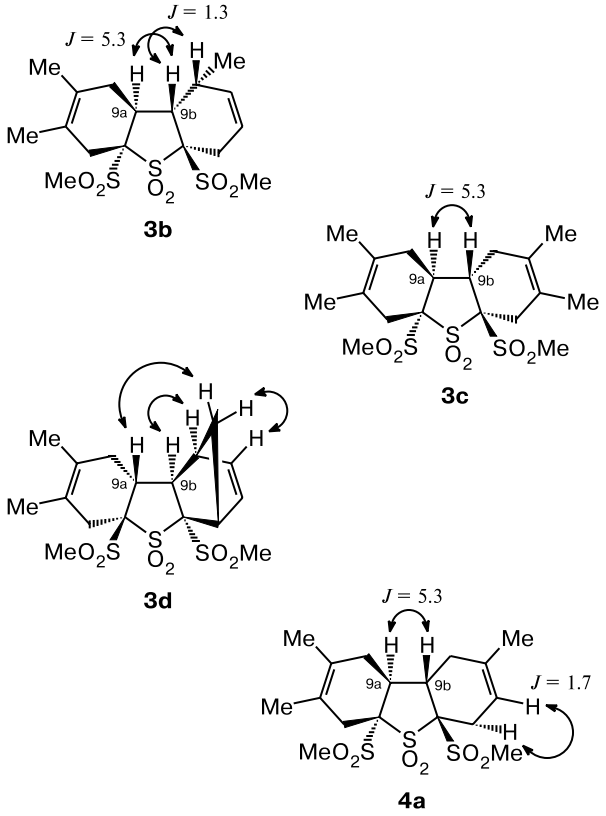


Fig. 1. Molecular structure of bisadduct **3c**.

Scheme 2



that this reaction is regioselective and the monoadduct is formed exclusively as the *para*-isomer.

The ^{1}H NMR spectrum of the adduct with piperilene contains signals belonging to both possible stereoisomers: *endo*- and *exo*-adducts **3b** and **4b** of the Diels—Alder reaction in the ratio *endo* : *exo* = 2.5 : 1. As in the case of the reaction with isoprene, the chemical shifts in the earlier studied monoadduct with piperilene (whose structure was unambiguously confirmed by X-ray diffraction[6]) are in good agreement with the observed chemical shifts in the ^{1}H and ^{13}C NMR spectra of the bisadduct. It should be mentioned that the *meta*-addition of piperilene to monoadduct **2a** is observed as in the case of the reaction of piperilene with the corresponding thiophene dioxide.[6] The predomination of the *ortho*-isomer in the reaction products should be expected, which would correspond to the experimental facts well known for piperilene cycloaddition.[10—13] The predomination of a *meta*-isomer is an unusual fact, which we explained as addition to the least sterically hindered side of a dienophile. In the case of the monoadduct with piperilene, the calculated activation energies of the transition state confirm that the formation of a *meta-endo*-adduct is preferential.[6] These data suggest that the same regularities take place for the formation of the transition complex of piperilene and monoadduct **2a** and, hence, the stereochemistry of the reaction is similar.

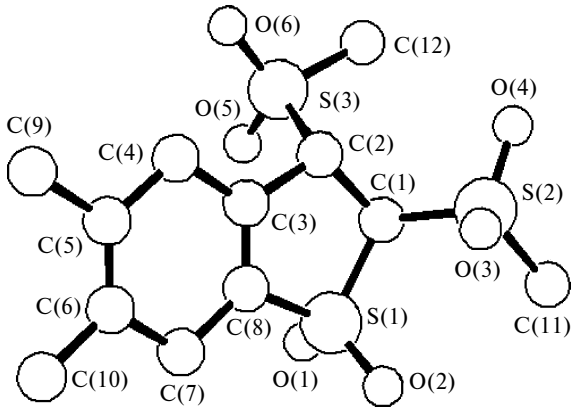


Fig. 2. Molecular structure of compound **5**.

We obtained unexpected results for the reaction of monoadduct **2a** with furans. It turned out that the heating of substance **2a** with furan and sylvan affords (according to the data of the NMR spectra and melting point) the same product. The X-ray structural studies of this substance showed that tetrahydrobenzothiophene dioxide derivative **5** was formed in these cases (Fig. 2).

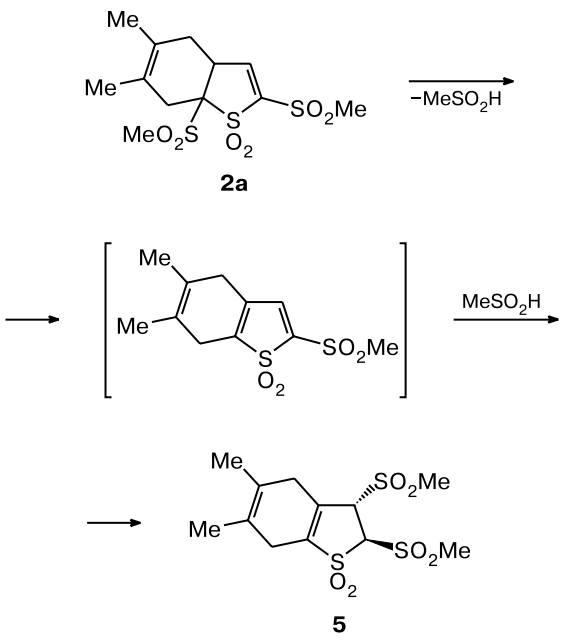
Based on the obtained results, we can assume the thermal transformation of **2a** into product **5**. The reference experiment (reflux of **2a** in an acetonitrile solution) showed that the considered rearrangement involved no furans, and the product was isolated in ~100% yield.

The proposed scheme of this transformation includes the elimination of methylsulfinic acid from the site of cycle conjunction and the subsequent Michael addition to the double bond activated by two acceptors to form a more stable *trans*-isomer (Scheme 3). The driving force of this transformation is a decrease in the steric strain of the molecule.

The elimination of a sulfonyl group is a rather well described transformation.[14] The removal of a sulfonyl moiety is a popular transformation widely used in synthetic practice for the formation of a new double carbon—carbon bond in a molecule and occurs, as a rule, under the action of bases.[14,15] Since monoadduct **2a** contains the double bond activated by two acceptors, the $MeSO_2$ group can easily be eliminated to form a favorable system of conjugated bonds. The same factors strongly facilitate the Michael addition of methylsulfinic acid. Further we are planning to prove that this reaction is general for the addition of different nucleophiles to monoadduct **2a**.

Thus, we studied the addition of the second diene molecule to monoadducts of acceptor thiophene dioxides with dienes under more rigid conditions and affording tricyclic bisadducts. This controlled assembling of six-membered fragment on the basis of one "core" (thiophene dioxide) can be useful for the creation of various carbocyclic and heterocyclic systems.

Scheme 3



Experimental

^{1}H and ^{13}C NMR spectra were recorded on a Varian VXR-400 spectrometer (working frequencies 400 and 100 MHz), using tetramethylsilane as the internal standard. Mass spectra were obtained on a Finnigan SSQ 7000 instrument. TLC was carried out on Merck $60F_{254}$ plates, and silica gel (63—200 mesh, Merck) was used for column chromatography.

Synthesis of 2,3-dimethyl-4a,5a-bis(methylsulfonyl)-1,4,4a,5a,6,9,9a,9b-octahydrodibenzo[*b*,*d*]thiophene 5,5-dioxides 3a—d and 4a,b (general procedure). Freshly distilled diene (1 mL, 12—15 mmol) was added to monoadduct **2a** (71 mg, 0.2 mmol) in acetic anhydride (1 mL), and the mixture was heated for 3 h at 120 °C. Then all volatile components were removed *in vacuo*, and the residue was purified by chromatography on silica gel (hexane—ethyl acetate (1 : 1) as eluent) to obtain bisadducts.

2,3,7-Trimethyl-4a,5a-bis(methylsulfonyl)-1,4,4a,5a,6,9,9a,9b-octahydrodibenzo[*b*,*d*]thiophene 5,5-dioxide (3a) and 2,3,8-trimethyl-4a,5a-bis(methylsulfonyl)-1,4,4a,5a,6,9,9a,9b-octahydrodibenzo[*b*,*d*]thiophene 5,5-dioxide (4a). The yield was 34% (mixture **3a**+**4a**). Found (%): C, 47.99; H, 6.13. $C_{17}H_{26}O_6S_3$. Calculated (%): C, 48.32; H, 6.20. ^{1}H NMR of **3a** ($CDCl_3$), δ: 1.73 (br.s, 6 H, C(2)Me, C(3)Me); 1.75 (s, 3 H, C(7)Me); 1.85 (m, 2 H, H(1), H(9)); 2.14, 2.18 (both d, 1 H each, H(1), H(9), J = 7.6 Hz, J = 6.2 Hz); 2.40 (m, 2 H, H(4), H(6)); 2.68—2.72 (ddd, 1 H, H(4), H(6), J = 1.8 Hz, J = 4.4 Hz, J = 6.5 Hz); 2.75 (m, 1 H, H(4), H(6)); 3.08 (s, 3 H, SO_2Me); 3.10 (s, 3 H, SO_2Me); 3.39, 3.44 (both dd, 1 H each, H(9a), H(9b), J = 5.3 Hz, J = 1.7 Hz); 5.49 (t, 1 H, H(8), J = 3.5 Hz); **4a**: 1.73 (br.s, 6 H, C(2)Me, C(3)Me); 1.75 (s, 3 H, C(8)Me); 1.85 (m, 2 H, H(1), H(9)); 2.18 (d, 1 H, H(1) or H(9), J = 7.6 Hz, J = 6.2 Hz); 2.40 (m, 2 H, H(4), H(6)); 2.75 (m, 2 H, H(1) or H(9)); 2.79—2.83 (ddd, 1 H, H(4), H(6), J = 1.8 Hz, J = 4.4 Hz, J = 6.5 Hz); 3.01 (s, 3 H, SO_2Me); 3.05 (s, 3 H, SO_2Me); 3.64, 3.75 (both dd, 1 H each, H(9a), H(9b), J = 5.3 Hz, J = 2.3 Hz); 5.65 (t, 1 H, H(7), J = 1.7 Hz).

9-*endo*- (3b) and 9-*exo*-2,3,9-Trimethyl-4a,5a-bis(methylsulfonyl)-1,4,4a,5a,6,9,9a,9b-octahydrodibenzo[*b*,*d*]thiophene 5,5-dioxide (4b). The yield was 35% (mixture **3b**+**4b**). Found (%): C, 48.38; H, 6.16. $C_{17}H_{26}S_3O_6$. Calculated (%): C, 48.32; H, 6.20. ^{1}H NMR of **3b** ($CDCl_3$), δ: 1.70 (br.s, 6 H, C(2)Me, C(3)Me); 1.75 (br.s, 3 H, C(1)Me); 2.12 (dd, 1 H, H(1), J = 1.3 Hz, J = 9.5 Hz); 2.40—2.48 (ddd, 2 H, H(4) or H(6), J = 1.5 Hz, J = 5.4 Hz, J = 7.6 Hz); 2.49—2.51 (m, 2 H, H(9)); 3.08 (s, 3 H, SO_2Me); 3.17—3.20 (m, 2 H, H(6) or H(4)); 3.43 (s, 3 H, SO_2Me); 3.56, 3.61 (both dd, 1 H each, H(9a), H(9b), J = 5.3 Hz, J = 1.3 Hz); 5.84—5.88 (dt, 1 H, H(8), J = 4.7 Hz, J = 5.6 Hz, J = 9.5 Hz); 6.05—6.11 (м, 1 H, H(7)); **4b**: 1.73 (br.s, 6 H, C(2)Me, C(3)Me); 1.77 (br.s, 3 H, C(1)Me); 2.19 (dd, 1 H, H(1), J = 2.8 Hz, J = 7.9 Hz); 2.51—2.63 (m, 2 H, H(4) or H(6)); 2.74—2.78 (ddd, 2 H, H(9), J = 1.7 Hz, J = 5.3 Hz, J = 7.9 Hz); 3.13 (s, 3 H, SO_2Me); 3.24—3.29 (m, 2 H, H(4) or H(6)); 3.37 (s, 3 H, SO_2Me); 3.58, 3.63 (both dd, 1 H each, H(9a), H(9b), J = 5.3 Hz, J = 7.9 Hz); 5.84—5.88 (dt, 1 H, H(2), J = 2.8 Hz, J = 5.6 Hz, J = 9.1 Hz); 6.05—6.11 (m, 1 H, H(3)).

2,3,7,8-Tetramethyl-4a,5a-bis(methylsulfonyl)-1,4,4a,5a,6,9,9a,9b-octahydrodibenzo[*b*,*d*]thiophene 5,5-dioxide (3c). The yield was 55%, m.p. 265 °C. Found (%): C, 49.74; H, 6.55. $C_{18}H_{28}O_6S_3$. Calculated (%): C, 49.52; H, 6.46. ^{1}H NMR ($CDCl_3$), δ: 1.72, 1.75 (both br.s, 6 H each, C(2)Me, C(3)Me, C(7)Me, C(8)Me); 2.13—2.17, 2.40—2.44 (both br.d, 2 H each, H(4), H(6), J = 16.7 Hz); 2.68—2.73 (m, 4 H, H(1), H(9)); 3.10 (s, 6 H, C(4a)—SO_2Me, C(5a)—SO_2Me); 3.14, 3.20 (both dd, 1 H each, H(9a), H(9b), J = 5.3 Hz, J = 3.8 Hz). ^{13}C NMR (CD_3CN), δ: 18.5 (C(2)Me, C(3)Me); 19.7 (C(7)Me, C(8)Me); 29.9 (C(1), C(9)); 31.1 (C(4), C(6)); 37.6 (SO_2Me); 39.0 (SO_2Me); 86.0 (C(9a), C(9b)); 122.1 (C(4a), C(5b)); 123.3 (C(3), C(7)); 125.2 (C(2), C(8)).

2,3-Dimethyl-6,9-(*endo*-methylene)-4a,5a-bis(methylsulfonyl)-1,4,4a,5a,6,9,9a,9b-octahydrodibenzo[*b*,*d*]thiophene 5,5-dioxide (3d). The yield was 51%, m.p. 291 °C. Found (%): C, 48.38; H, 5.81. $C_{17}H_{24}S_3O_6$. Calculated (%): C, 48.55; H, 5.75. ^{1}H NMR ($CDCl_3$), δ: 1.69 (br.s, 6 H, C(2)Me, C(3)Me); 1.84—1.87 (dd, 1 H, H(10), J = 1.3 Hz, J = 9.9 Hz); 2.02—2.07 (m, 1 H, H(10)); 2.42—2.49, 2.57—2.62 (both m, 2 H each, H(6), H(9)); 2.88 (t, 1 H, H(4), J = 8.3 Hz); 3.00 (m, 1 H, H(1)); 3.11, 3.15 (both s, 3 H each, SO_2Me); 3.50 (m, 1 H, H(9a)); 3.90—3.95 (dd, 1 H, H(9b), J = 5.3 Hz, J = 1.3 Hz); 6.15, 6.39 (both dd, 1 H each, C<u>H</u>=C<u>H</u>, J = 3.3 Hz, J = 8.3 Hz, J = 3.3 Hz, J = 9.9 Hz). ^{13}C NMR ($CDCl_3$), δ: 18.8 (C(7)Me); 19.8 (C(8)Me); 28.5; 33.1; 36.8; 39.3 (SO_2Me); 40.8 (SO_2Me); 48.9; 50.4 (C(9a), C(9b)); 89.1; 95.9; 100.2; 120.7; 123.2; 134.7 (C(5a)); 139.7 (C(4a)).

Thermal rearrangement of monoadduct 2a. Monoadduct **2a** (350 mg, 1 mmol) was refluxed for 8 h in a solution of MeCN (20 mL). The solvent was evaporated *in vacuo*, and the residue was recrystallized from toluene to obtain **5,6-dimethyl-2,3-bis(methylsulfonyl)-2,3,4,7-tetrahydro-1-benzothiophene 1,1-dioxide (5)**. The yield was 350 mg (98%), m.p. 227 °C. Found (%): C, 40.74; H, 5.15. $C_{12}H_{18}O_6S_3$. Calculated (%): C, 40.66; H, 5.12. ^{1}H NMR (CD_3CN), δ: 1.70 (br.s, 6 H, C(5)Me, C(6)Me); 2.96—3.14 (m, 4 H, H(4), H(7)); 3.14, 3.19 (both s, 3 H each, SO_2Me); 4.96 (d, 1 H, H(3), J = 2.0 Hz); 5.47 (d, 1 H,

Table 2. Selected crystallographic parameters of compounds **3c** and **5**

Parameter	**3c**	**5**
Molecular formula	$C_{18}H_{28}O_6S_3$	$C_{12}H_{18}O_6S_3$
Molecular weight	436.58	354.47
Crystal system	Monoclinic	Triclinic
Space group	$C2/c$	$P\bar{1}$
a/Å	21.587(4)	8.530(2)
b/Å	15.118(3)	10.226(2)
c/Å	15.241(3)	12.400(2)
β/deg	124.19(3)	103.80(3)
γ/deg	90	105.70(3)
V/Å^3	4114.3(14)	926.4(3)
Z	8	2
d/g cm^{-1}	1.410	1.407
μ/mm^{-1}	0.392	0.428
θ_{max}/deg	24.97	24.97
Scan range	$1.76° \le \theta \le 24.97°$	$1.85° \le \theta \le 24.97°$
Number of independent reflections	2246	2501
Number of reflections with $I \ge 2\sigma(I)$	2304	2027
Number of refined parameters	357	283
R_1 ($I \ge 2\sigma(I)$)	0.0265	0.0381
wR_2 (against all reflections)	0.0727	0.1110

H(2), J = 2.0 Hz). ^{13}C NMR (CD_3CN), δ: 18.2 (C(5)); 28.3 (C(6)); 27.0; 35.2; 40.8 (SO_2Me); 41.5 (SO_2Me); 64.9; 75.2; 121.1; 129.8; 136.8; 140.4. Mass spectrum (EI, 70 eV), m/z (I_{rel} (%)): 354 $[M]^+$ (5), 274 $[M - MeSO_2]^+$ (10), 259 $[M - MeSO_2 - Me]^+$ (15), 195 $[M - MeSO_2 - Me - SO_2]^+$ (30), 130 (45), 115 (50), 106 $[M - MeSO_2 - 2\ Me - 2\ SO_2]^+$ (20), 91 $[M - MeSO_2 - 3\ Me - 2\ SO_2]^+$ (100), 77 $[M - MeSO_2 - 4\ Me - 2\ SO_2]^+$ (20), 65 (60), 39 (35).

X-ray diffraction study of compounds 3c and 5. Single crystals of compound **3c** were obtained by recrystallization from chloroform, and those of **5** were recrystallized from toluene. X-ray diffraction study was carried out on an Enraf-Nonius CAD-4 diffractometer (β-filter, Mo-Kα radiation, λ = 0.71073 Å, at 293 K, θ/2θ scan mode). The selected experimental parameters and crystallographic data for compounds **3c** and **5** are given in Table 2. The structures were solved by a combination of the direct method and Fourier transform. Positions of non-hydrogen atoms were refined by the full-matrix least-squares method in the anisotropic approximation, and positions of hydrogen atoms were calculated in the isotropic approximation. The calculations were performed using the SHELX-97 program package. The complete X-ray diffraction data were deposited at the Cambridge Structure Database.

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