Synthesis of substituted 1,2-di(alkylsulfonyl)indolizines. Molecular and crystal structure of 3-(4-fluorobenzoyl)-6-methyl-1,2-di(propylsulfonyl)indolizine*

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The reactions of substituted pyridinium salts with *E*-1,2-di(alkylsulfonyl)-1,2-dichloroethenes proceed regiospecifically. Heating of these reagents in chloroform in the presence of a threefold excess of Et_3N affords substituted 1,2-di(alkylsulfonyl)indolizines in high yields. The structures of the reaction products were confirmed by physicochemical methods, including X-ray diffraction.

Key words: 1,2-di(alkylsulfonyl)indolizines, 3-(4-fluorobenzoyl)-6-methyl-1,2-di(propylsulfonyl)indolizine, pyridinium salts, *E*-1,2-di(alkylsulfonyl)-1,2-dichloroethene, pyridinium ylides, X-ray diffraction study.

The indolizine ring is present in many practically important compounds, for example, in alkaloids and pharmaceuticals.[1] Generally, these compounds are synthesized by the Chichibabin reaction of α-picolines with phenacyl halides[2] or by 1,3-dipolar cycloaddition of polarized ethylenes to pyridinium, quinolinium, and isoquinolinium ylides.[3,4] The former reaction affords aromatic heterocycles, whereas the latter reaction produces hydrogenated indolizines. Earlier,[5] we have found that the reaction of *N*-phenacylpyridinium ylide with *E*-1,2-dichloro-1,2-di(ethylsulfonyl)ethene yielded 3-benzoyl-1,2-di(ethylsulfonyl)indolizine, whose structure has been established by various physicochemical methods, including X-ray diffraction.

With the aim of improving this method for the synthesis of polyfunctionalized indolizines (which are potentially biologically active compounds), we studied the reaction of substituted pyridinium ylides with *E*-1,2-di(alkylsulfonyl)-1,2-dichloroethenes and examined the influence of the structure of pyridinium ylide on the regioselectivity of this reaction.

Pyridinium ylides **1** were generated by treating pyridinium salt **2** with a threefold excess of Et_3N in $CHCl_3$ followed by the addition of *E*-1,2-di(alkylsulfonyl)-1,2-dichloroethene **3**. The reaction mixture was refluxed for 40 min. Substituted 1,2-di(alkylsulfonyl)indolizines **4** were isolated in high yields (63—88%) (Table 1).

Apparently, these reactions occur by the 1,3-dipolar cycloaddition mechanism through the formation of hydrogenated indolizines **5**. Double dehydrochlorination of the latter affords substituted 1,2-di(alkylsulfonyl)indolizines **4** (Scheme 1).

Intermediates **5** were not isolated. However, their *trans-trans-trans* structures were confirmed, with a certain probability, by the general features of concerted reactions and the available experimental data. It is known[6] that 1,3-dipolar cycloaddition occurs with retention of the starting symmetry of substituted ethenes, *i.e.*, the *E* configuration of the starting ethenes is retained in intermediate **5**.

Earlier,[3,4,7—9] the reactions of pyridinium, quinolinium, and isoquinolinium ylides with unsaturated nitriles have been found to proceed with high selectivity *via* 1,3-dipolar cycloaddition to form *trans-trans-trans* isomers with respect to the substituted tetrahydropyrrole fragment. The H(3) and H(8a) atoms lie in a single plane. Besides, the *trans* arrangement of the hydrogen and chlorine atoms in intermediate **5** is favorable for *trans*-elimination of HCl, which is confirmed by the high yields of indolizines **4** (see Table 1).

The structures of compounds **4** were established by elemental analysis, ^1H NMR and IR spectroscopy, and mass spectrometry (Tables 1 and 2). The IR spectra of

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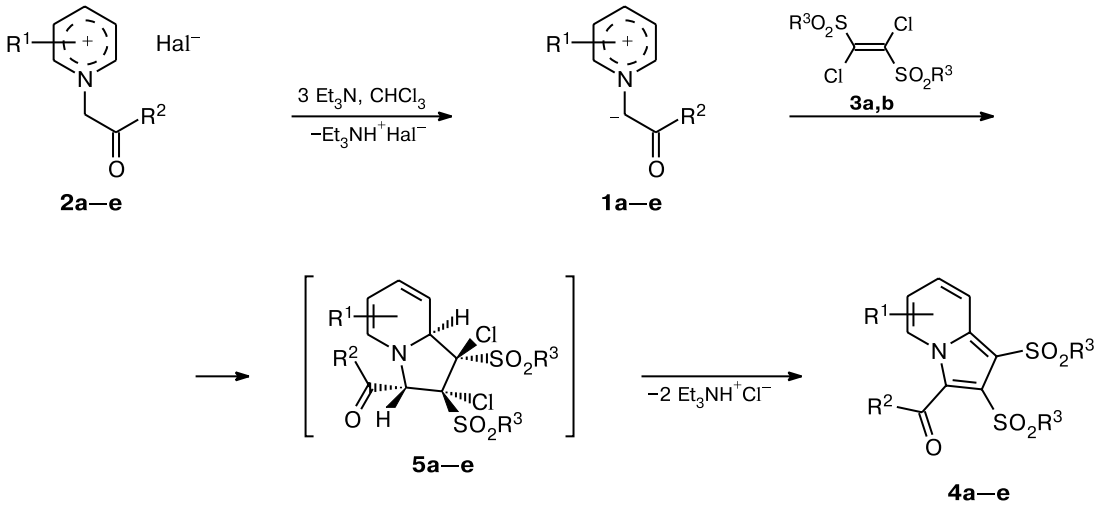
Table 1. Physicochemical characteristics of substituted 1,2-di(alkylsulfonyl)indolizines **4**

Compound	M.p./°C (Solvent)	Yield (%)	Found / Calculated (%)				Molecular formula
			C	H	N	S	
4a	229* (Acetone)	63	45.76 / 45.34	4.81 / 4.68	8.31 / 8.13	18.53 / 18.62	$C_{13}H_{16}N_2O_5S_2$
4b	106—107 ($Et_2O/CHCl_3$)	73	51.87 / 52.03	6.01 / 6.08	3.29 / 3.37	15.18 / 15.43	$C_{18}H_{25}NO_6S_2$
4c	157—158 ($CHCl_3$)	71	62.11 / 62.00	6.71 / 6.60	2.92 / 2.78	12.94 / 12.73	$C_{26}H_{33}NO_5S_2$
4d	166—167 ($Et_2O/CHCl_3$)	69	48.06 / 48.24	5.04 / 5.13	3.53 / 3.75	17.29 / 17.17	$C_{15}H_{19}NO_6S_2$
4e	181—182 (Acetone)	88	56.01 / 56.23	5.35 / 5.39	6.33 / 6.25	14.72 / 14.29	$C_{21}H_{24}N_2O_5S_2$

* Compound **4a** melts with decomposition.

Scheme 1



1, 2	R^1	R^2	**4, 5**	R^1	R^2	R^3
a	H	NH_2	**a**	H	NH_2	Et
b	4-Et	OMe	**b**	7-Et	OMe	Pr^n
c	4-Bu^t	4-MeC_6H_4	**c**	7-Bu^t	4-MeC_6H_4	Pr^n
d	3-Me	Me	**d**	8-Me	OMe	Et
e	3-NH_2	Ph	**e**	8-NH_2	Ph	Pr^n

3: R^3 = Et (**a**), Pr^n (**b**)

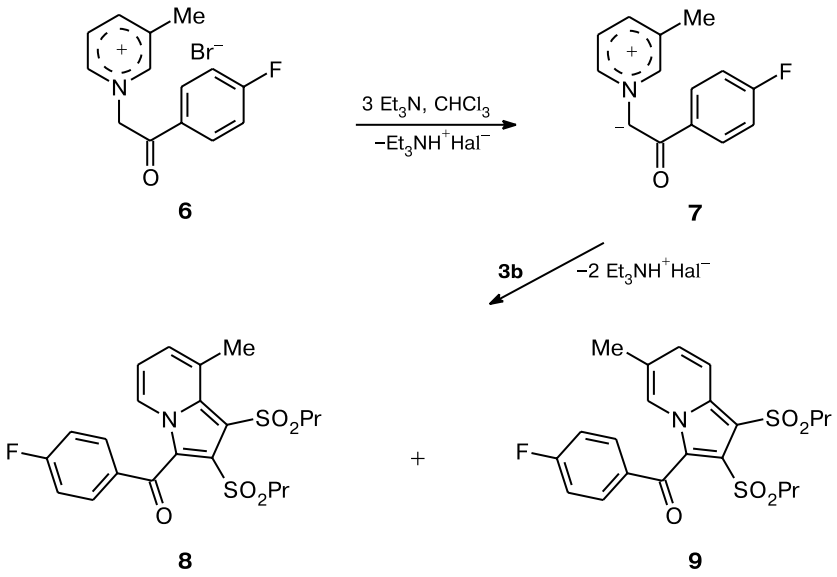
compounds **4** show absorption bands corresponding to stretching vibrations of the carbonyl (1712—1688 cm^{-1}) and sulfonyl groups[12] (1324—1308 and 1145—1140 cm^{-1}). In addition, the spectra of compounds **4a,e** show stretching and bending vibrations in the 1636—1624 and 3444—3168 cm^{-1} regions, respectively, characteristic of the amide and amino groups. The mass spectra of all compounds **4** have the molecular ion peak $M^{+\bullet}$. Analysis of the ^1H NMR spectra led to the conclusion that the reactions of 3-substituted pyridinium ylides **1d,e** with ethylenes **3a,b** proceed regioselectively. Although there are two possible reaction pathways, *i.e.*, the reaction can occur at the C(2) or C(6) atom of pyridinium ylides **1d,e**, annelation does occur at position 2. As a result, the Me or NH_2 substituent is present at position 8 of indolizines **4d,e**. The signals for the protons of the pyridine fragment of molecules **4d,e** appear as two doublets at δ 6.68—7.14 and 7.72—8.29 for the H(7) and H(5) protons, respectively, and as one triplet at δ 6.75—6.92 for H(6).

We found that the reaction of ylide **7**, which was generated from the corresponding salt **6**, with ethene **3b** proceeded nonregioselectively, resulting in the formation of

Table 2. Spectroscopic characteristics of substituted 1,2-di(alkylsulfonyl)indolizines **4**

Compound	IR, ν/cm^{-1}			^{1}H NMR (DMSO-d$_6$, δ, *J*/Hz)	*m/z* (*I*$_{rel}$ (%))
	C=O	SO_2	NH_2		
4a	1688	1308, 1140	3296, 3168, 1636 (δ)	1.16, 1.28 (both t, 3 H each, Me, *J* = 7.8); 3.50, 3.65 (both q, 2 H each, CH_2, *J* = 7.8); 7.12 (t, 1 H, C(6)H, *J* = 8.1, *J* = 6.8); 7.43 (t, 1 H, C(7)H, *J* = 6.8, *J* = 7.2); 8.04 (s, 1 H, NH); 8.22 (m, 3 H, C(5)H, C(8)H, NH)	344 $[M]^+$ (28)
4b	1700	1324, 1141	—	1.05 (t, 3 H, Me, *J* = 7.7); 1.17, 1.28 (both t, 3 H each, Me, *J* = 8.1); 1.76, 1.94 (both m, 2 H each, CH_2); 2.76 (q, 2 H, CH_2, *J* = 7.7); 3.48, 3.59 (both q, 2 H each, CH_2, *J* = 8.1); 3.97 (s, 3 H, MeO); 7.05 (d, 1 H, C(6)H, *J* = 8.0); 8.07 (s, 1 H, C(8)H); 8.44 (d, 1 H, C(5)H, *J* = 8.0)	415 $[M]^+$ (32)
4c	1710	1316, 1145	—	0.97, 1.08 (both t, 3 H each, Me, *J* = 7.9); 1.26 (s, 9 H, Bu^t); 1.64, 1.77 (both m, 2 H each, CH_2); 2.45 (s, 3 H, Me); 3.50 (m, 4 H, $(CH_2)_2$); 7.05 (d, 1 H, C(6)H, *J* = 8.8); 7.26, 7.62 (both d, 2 H each, C_6H_4, *J* = 8.1); 7.76 (d, 1 H, C(5)H, *J* = 8.8); 8.22 (s, 1 H, C(8)H)	503 $[M]^+$ (51)
4d	1705	1324, 1140	—	1.51 (m, 6 H, 2 Me); 2.88 (s, 3 H, Me); 3.70, 3.85 (both q, 2 H each, CH_2, *J* = 7.9); 4.03 (s, 3 H, MeO); 6.92 (t, 1 H, C(6)H, *J* = 8.0, *J* = 6.4); 7.14 (d, 1 H, C(7)H, *J* = 8.0); 8.29 (d, 1 H, C(5)H, *J* = 6.4)	373 $[M]^+$ (24)
4e	1712	1312, 1144	3444, 3424, 3344, 1624 (δ)	0.95, 1.11 (both t, 3 H each, Me, *J* = 7.9); 1.68, 1.95, 3.51, 3.62 (all m, 2 H each, CH_2); 6.50 (s, 2 H, NH_2); 6.68 (d, 1 H, C(7)H, *J* = 8.1); 6.75 (m, 1 H, C(5)H); 7.15, 7.53, 7.68 (all m, 5 H, Ph); 7.72 (d, 1 H, C(5)H, *J* = 6.3)	448 $[M]^+$ (20)

Scheme 2



a mixture of isomeric indolizines **8** and **9** (Scheme 2). A comparison of the ^{1}H NMR spectra of indolizines **4d,e** with the spectrum of a mixture of compounds **8** and **9** demonstrates that, in the latter case, annelation also occurs predominantly at position 2. The ratio of isomeric indolizines **8** : **9** = 3 : 1. This difference in reactivity is, apparently, associated with stabilization of invertomers of pyridinium ylides (*syn* $\rightleftharpoons$ *anti*) and the fact that the reactivity depends on the nature of the substituent in the benzene ring (4-MeC_6H_4 or 4-FC_6H_4). Earlier,[3,4,10,11] this effect has been observed in the reactions of α- and β-picolinium ylides with unsaturated thioamides.

Attempts to separate a mixture of indolizines **8** and **9** failed. However, a crystal of isomer **9** was mechanically isolated from the reaction mixture and studied by X-ray diffraction.

The molecular structure of indolizine **9** is shown in Fig. 1. The bond lengths and bond angles are given in

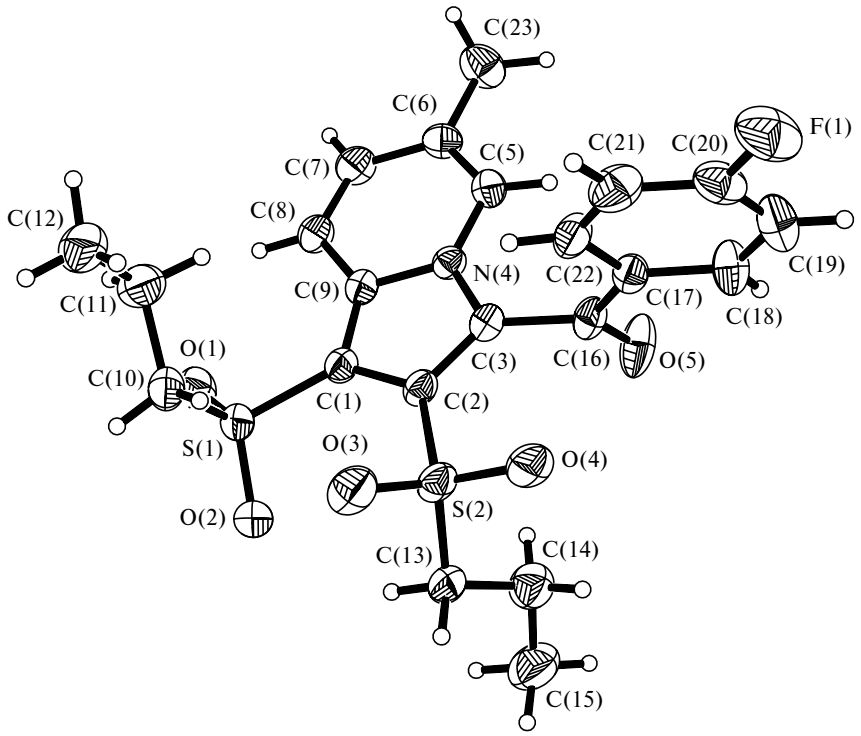


Fig. 1. Molecular structure of indolizine **9**.

Tables 3 and 4, respectively. The geometric parameters of molecule **9** have standard values[13] and are consistent with the proposed structure.

The bicyclic fragment in compound **9** is planar (within 0.008 Å). The steric hindrance associated with the presence of shortened intramolecular nonbonded contacts not only causes rotation of the aryl substituent relative to the plane of the indolizine ring by 98.5° and influences the geometry of the propylsulfonyl substituents, but also excludes conjugation of the C=O group with the indolizine

Table 3. Bond lengths (*d*) in molecule **9**

Bond	*d*/Å	Bond	*d*/Å
F(1)—C(20)	1.353(4)	C(5)—C(6)	1.355(5)
S(1)—O(1)	1.431(3)	C(6)—C(7)	1.414(5)
S(1)—O(2)	1.440(3)	C(6)—C(23)	1.499(5)
S(1)—C(1)	1.745(3)	C(7)—C(8)	1.335(5)
S(1)—C(10)	1.770(4)	C(8)—C(9)	1.417(4)
S(2)—O(3)	1.429(3)	C(10)—C(11)	1.499(6)
S(2)—O(4)	1.435(3)	C(11)—C(12)	1.509(7)
S(2)—C(13)	1.752(4)	C(13)—C(14)	1.505(6)
S(2)—C(2)	1.770(3)	C(14)—C(15)	1.503(7)
O(5)—C(16)	1.213(4)	C(16)—C(17)	1.466(5)
N(4)—C(3)	1.381(4)	C(17)—C(22)	1.381(5)
N(4)—C(9)	1.385(4)	C(17)—C(18)	1.389(5)
N(4)—C(5)	1.393(4)	C(18)—C(19)	1.369(6)
C(1)—C(9)	1.389(4)	C(19)—C(20)	1.353(6)
C(1)—C(2)	1.427(4)	C(20)—C(21)	1.360(6)
C(2)—C(3)	1.368(4)	C(21)—C(22)	1.379(6)
C(3)—C(16)	1.509(4)		

Table 4. Bond angles (ω) in molecule **9**

Angle	ω/deg	Angle	ω/deg
O(1)—S(1)—O(2)	117.2(2)	C(5)—C(6)—C(7)	118.6(3)
O(1)—S(1)—C(1)	108.0(2)	C(5)—C(6)—C(23)	120.9(4)
O(2)—S(1)—C(1)	108.7(2)	C(7)—C(6)—C(23)	120.6(3)
O(1)—S(1)—C(10)	108.3(2)	C(8)—C(7)—C(6)	122.6(3)
O(2)—S(1)—C(10)	108.6(2)	C(7)—C(8)—C(9)	119.4(3)
C(1)—S(1)—C(10)	105.4(2)	N(4)—C(9)—C(1)	107.0(2)
O(3)—S(2)—O(4)	118.3(2)	N(4)—C(9)—C(8)	117.9(3)
O(3)—S(2)—C(13)	108.8(2)	C(1)—C(9)—C(8)	135.1(3)
O(4)—S(2)—C(13)	107.7(2)	C(11)—C(10)—S(1)	112.8(3)
O(3)—S(2)—C(2)	108.3(2)	C(10)—C(11)—C(12)	114.1(4)
O(4)—S(2)—C(2)	105.8(2)	C(14)—C(13)—S(2)	114.1(3)
C(13)—S(2)—C(2)	107.5(2)	C(15)—C(14)—C(13)	113.0(5)
C(3)—N(4)—C(9)	110.2(2)	O(5)—C(16)—C(17)	122.0(3)
C(3)—N(4)—C(5)	128.0(3)	O(5)—C(16)—C(3)	116.4(3)
C(9)—N(4)—C(5)	121.8(3)	C(17)—C(16)—C(3)	121.4(3)
C(9)—C(1)—C(2)	107.1(3)	C(22)—C(17)—C(18)	118.4(3)
C(9)—C(1)—S(1)	123.4(2)	C(22)—C(17)—C(16)	122.3(3)
C(2)—C(1)—S(1)	129.5(2)	C(18)—C(17)—C(16)	119.3(3)
C(3)—C(2)—C(1)	108.4(3)	C(19)—C(18)—C(17)	121.2(4)
C(3)—C(2)—S(2)	122.4(2)	C(20)—C(19)—C(18)	118.4(4)
C(1)—C(2)—S(2)	129.1(2)	C(19)—C(20)—F(1)	118.8(4)
C(2)—C(3)—N(4)	107.3(3)	C(19)—C(20)—C(21)	122.9(4)
C(2)—C(3)—C(16)	132.4(3)	F(1)—C(20)—C(21)	118.3(4)
N(4)—C(3)—C(16)	119.3(3)	C(20)—C(21)—C(22)	118.5(4)
C(6)—C(5)—N(4)	119.7(3)	C(21)—C(22)—C(17)	120.5(4)

and aryl rings, which is evident from the corresponding bond lengths (see Table 3).

The observed distortion of the tetrahedral configuration of the sulfur atoms (the bond angles at the S(1) and S(2) atoms vary in ranges of 105.4—117.2(2) and 105.8—118.3(2)°, respectively) is typical of arylsulfonyl-substituted derivatives.[14] An increase in the O(1)—S(1)—O(2) and O(3)—S(2)—O(4) bond angles (117.2(2) and 118.3(2)°, respectively) is typical of sulfonyl compounds and is consistent with the concepts of the valence shell electron pair repulsion (VSEPR) theory.[15]

In molecule **9**, the C_{sp^2}—S bonds differ in length. The C(2)—S(2) bond length (1.770(3) Å) is typical of C_{sp^2}—S single bonds (1.763 Å),[13] whereas the C(1)—S(1) bond is slightly shortened (1.745(3) Å). This difference in the C_{sp^2}—S bond lengths can be attributed to the difference in the degree of involvement of the sulfonyl groups in conjugation with the π system of the indolizine ring, which apparently depends on the deviation of the sulfur atoms from this plane. Actually, the S(1) atom lies exactly in the plane of the heterocycle (the deviation from the plane is −0.021 Å), whereas the S(2) atom deviates from this plane by 0.096 Å. The more noticeable deviation of the S(2) atom is, apparently, caused by the steric effects of both the adjacent propylsulfonyl group and the 4-fluorobenzoyl substituent. Earlier, we have found an analogous structure and bond length distribution for the substituted 1,2-di(ethylsulfonyl)indolizine molecule.[5] As can be seen from Fig. 1, the propyl substituents deviate from the plane of the bicyclic fragment in opposite directions.

Noteworthy is the intramolecular O(1)...H(8) nonbonded contact (2.39(5) Å), which can be considered[16] as an O...H—C hydrogen bond with the usual geometric parameters (O(1)...C(8), 2.972(3) Å; C(8)—H(8), 0.96(5) Å; C(8)—H(8)...O(1), 119(3)°).

Analysis of the molecular packing in the crystal structure of **9** demonstrated that there are no intermolecular nonbonded contacts, whose lengths are smaller than the sums of the corresponding van der Waals radii.[17]

Experimental

The IR spectra were measured on Perkin—Elmer-577 and Specord M-82 instruments in KBr pellets at a concentration of 0.01 mol L^{-1}. The ^1H NMR spectra were recorded on Bruker DRX-500 (500 MHz) and Bruker WM-250 (250 MHz) instruments for 5—12% solutions in DMSO-d_6 with Me_4Si as the internal standard and in $CDCl_3$. The mass spectra were obtained on a Finnigan MAT INCOS 50 quadrupole mass spectrometer; the ionization energy was 70 eV. The reactions were monitored and the purity of the reaction products was analyzed by thin-layer chromatography on Silufol UV-254 plates with the use of a 2 : 1 hexane—acetone mixture as the eluent; visualization was carried out with iodine vapor.

X-ray diffraction study of 3-(4-fluorobenzoyl)-6-methyl-1,2-di(propylsulfonyl)indolizine (9). Colorless crystals of indolizine **9** were prepared by slow evaporation of a solution of **9** in ethanol for 3 days. The crystals of compound **9** are monoclinic, at 25 °C a = 9.168(7), b = 18.991(13), c = 13.031(9) Å, β = 93.74(6)°, V = 2264(3) $Å^3$, d_{calc} = 1.366 g cm^{-3}, Z = 4, space group $P2_1/n$. The unit cell parameters and intensities of 5262 independent reflections were measured on a four-circle automated Siemens P3/PC diffractometer (λ(Mo-Kα), graphite monochromator, θ/2θ scanning technique, θ_{max} = 28°). The structure was solved by direct methods and refined by the full-matrix least-squares method with anisotropic displacement parameters for non-hydrogen atoms. All hydrogen atoms were revealed from the difference Fourier synthesis and refined isotropically. The final R factors were as follows: R = 0.062 based on 2805 independent reflections with $I > 2\sigma(I)$ and R_w = 0.157 based on all 4927 reflections. All calculations were carried out using the SHELXTL PLUS program package (PC version). The atomic coordinates were deposited with the Cambridge Structural Database.

***E*-1,2-Di(alkylsulfonyl)-1,2-dichloroethenes 3a,b** were synthesized according to a procedure described earlier.[12]

Substituted 1,2-di(alkylsulfonyl)indolizines 4a—e (general procedure). Triethylamine (0.003 mol) was added with stirring to a solution of compound **2** (0.001 mol) in $CHCl_3$, and then a solution of sulfones **3a,b** was added dropwise. The reaction mixture was refluxed for 40 min (chromatographic control), diluted with $CHCl_3$, washed with water, and dried with $MgSO_4$. Then the solution was concentrated and the solid residue was crystallized. The characteristics of compounds **4a—e** are given in Tables 1 and 2.

Synthesis of 8- and 3-(4-fluorobenzoyl)-6-methyl-1,2-di(propylsulfonyl)indolizines (8 and 9). The reactions of compounds **6** and **3b** were carried out according to the procedure described for compound **4**. The solid residue was crystallized from ethanol, and a mixture of isomeric indolizines **8** and **9** was isolated in a ratio of 3 : 1 in 68% yield. Found (%): C, 56.54; H, 5.11; N, 3.08; S, 13.89. $C_{22}H_{24}FNO_5S_2$. Calculated (%): C, 56.75; H, 5.20; N, 3.02; S, 13.77. IR, ν/cm^{-1}: 1708 (CO); 1322 and 1140 (SO_2). ^1H NMR (250 MHz, $CDCl_3$), δ: 1.05 and 1.16 (both m, 3 H each, Me); 1.78 and 2.07 (both m, 2 H each, CH_2); 2.24 (s, Me, isomer **9**); 2.82 (s, Me, isomer **8**); 3.64 (m, 4 H, $(CH_2)_2$); 6.78 (t, C(6)H, isomer **8**, J = 7.9 Hz, J = 6.3 Hz); 7.16 and 7.83 (both m, C_6H_4, C(8)H (isomer **9**), C(5)H (isomer **8**)); 7.53 (s, C(5)H, isomer **9**); 7.62 (d, C(7)H, isomer **8**, J = 6.9 Hz); 8.33 (d, C(7)H, isomer **9**, J = 6.8 Hz).

References

1. *The Merck Index,* Ed. V. J. O'Neil, Merck and Co., Inc., Whitehouse Station, New York, 2001, 951.
2. A. E. Chichibabin, *Ber. Dtsch.Chem. Ges.*, 1925, **58**, 1706.
3. A. M. Shestopalov, Dr. Sc. (Chem.) Thesis, N. D. Zelinsky Institute of Organic Chemistry of the Russian Academy of Sciences, Moscow, 1991, 377 pp. (in Russian).
4. V. P. Litvinov, *Zh. Org. Khim.*, 1995, **30**, 1441 [*Russ. J. Org. Chem.*, 1995, **30** (Engl. Transl.).
5. V. N. Nesterov, N. E. Dontsova, A. M. Shestopalov, Yu. T. Struchkov, and V. P. Litvinov, *Khim. Geterotsikl. Soedin.*, 1996, **7**, 950 [*Chem. Heterocycl. Compd.*, 1996, **7** (Engl. Transl.)].

6. *The Chemistry of Heterocyclic Compounds. Vol. 59. Synthetic Applications of 1,3-Dipolar Cycloaddition Chemistry Toward Heterocycles and Natural Products*, Eds A. Padwa and W. H. Pearson, John Wiley and Sons, Inc., New York, 2002, 940 pp.
7. A. M. Shestopalov, V. P. Litvinov, Yu. A. Sharanin, and G. E. Khoroshilov, *Dokl. Akad. Nauk SSSR,* 1990, **312**, 1156 [*Dokl. Chem.*, 1990 (Engl. Transl.)].
8. A. M. Shestopalov, V. P. Litvinov, L. A. Rodinovskaya, and Yu. A. Sharanin, *Izv. Akad. Nauk SSSR, Ser. Khim.*, 1991, 146 [*Bull. Acad. Sci. USSR, Div. Chem. Sci.*, 1991, **40**, 129 (Engl. Transl.)].
9. A. M. Shestopalov, L. A. Rodinovskaya, Yu. A. Sharanin, and V. P. Litvinov, *Izv. Akad. Nauk SSSR, Ser. Khim.*, 1991, 1630 [*Bull. Acad. Sci. USSR, Div. Chem. Sci.*, 1991, **40**, 1446 (Engl. Transl.)].
10. A. M. Shestopalov, L. A. Rodinovskaya, V. P. Litvinov, B. Buinitskii, and M. Mikolaichik, *Dokl. Akad. Nauk,* 1992, **323**, 1116 [*Dokl. Chem.*, 1992 (Engl. Transl.)].
11. A. M. Shestopalov, O. P. Bogomolova, L. A. Rodinovskaya, V. P. Litvinov, B. Bujnicki, M. Mikolajczyk, V. N. Nesterov, and Yu. T. Struchkov, *Heteroatom. Chem.*, 1993, **4**, 593.
12. E. N. Prilezhaeva, N. E. Dontsova, N. P. Petukhova, and V. S. Bogdanov, *Gazz. Chim. Ital.*, 1990, **120**, 235.
13. F. N. Allen, O. Kennard, D. J. Watson, L. Brammer, A. G. Orpen, and R. Taylor, *J. Chem. Soc., Perkin Trans. 2*, 1987, S1.
14. A. M. O'Connell and E. N. Maslen, *Acta Crystallogr.*, 1967, **22**, 134.
15. R. J. Gillespie and I. Hargittai, *The VSEPR Model of Molecular Geometry*, Allyn and Bacon, 1991.
16. Z. Berkovitch-Yellin and L. Leiserowitz, *Acta Crystallogr.*, 1984, **B40**, 159.
17. R. S. Rowland and R. Taylor, *J. Phys. Chem.*, 1996, **100**, 7384.

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