Reactions of *o*-tosylaminobenzaldehyde with *p*-aminobenzenesulfonamides. Synthesis and structures of 13-(4-aminosulfonylphenyl)-6,12-epimino-5,11-ditosyl-5,6,11,12-tetrahydrodibenzo[*b*,*f*]-1,5-diazocine and its *N*-substituted derivatives

L. Yu. Ukhin,[a] Zh. I. Orlova,[a] and V. N. Khrustalev[b]*

[a]*Institute of Physical and Organic Chemistry, Rostov State University, 194/2 prosp. Stachki, 344090 Rostov on Don, Russian Federation. Fax: 007 (863 2) 28 5667*

[b]*A. N. Nesmeyanov Institute of Organoelement Compounds, Russian Academy of Sciences, 28 ul. Vavilova, 117813 Moscow, Russian Federation. Fax: 007 (095) 135 5085*

The reactions of *o*-tosylaminobenzaldehyde (**1**) with *p*-aminobenzenesulfonamides (**2a—c**) yielded 13-(*p*-$RNHSO_2C_6H_4$)-6,12-epimino-5,11-ditosyl-5,6,11,12-tetrahydrodibenzo[*b*,*f*]-1,5-diazocines (**3a—c**) (R = H (**a**); 2-thiazolyl (**b**); or 2,6-dimethoxy-4-pyrimidinyl 2,6-dimethoxy-4-pyrimidinyl (**c**). The structure of compound **3c** was confirmed by X-ray diffraction study.

Key words: *o*-tosylaminobenzaldehyde, *p*-aminobenzenesulfonamides, 13-(*p*-$RNHSO_2C_6H_4$)-6,12-epimino-5,11-ditosyl-5,6,11,12-tetrahydrodibenzo[*b*,*f*]-1,5-diazocines.

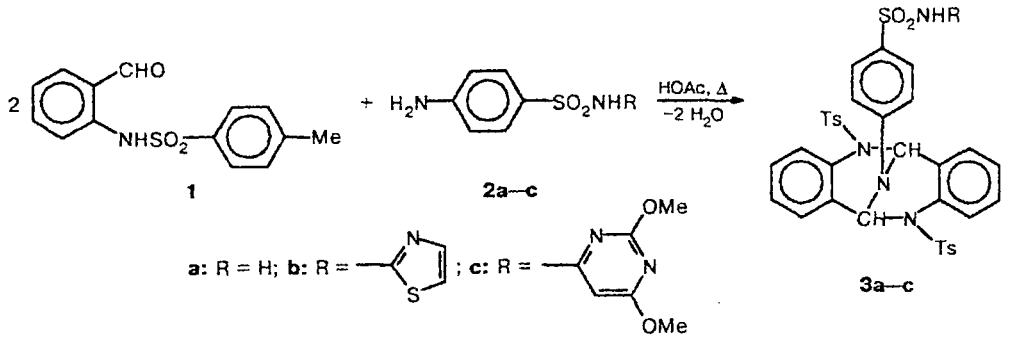
The important role of sulfanilamide compounds in medicine is well known. Many of these compounds exhibit antimicrobial activity and are used in the treatment of many infectious diseases. Some of them are capable of stimulating pancreatic β-cells, which produce insulin, and find use in therapy of diabetes. For this reason, it was of interest to introduce bridging sulfanilamide groups into the rigid cage structures of the azabicyclo[3.3.1]nonane derivatives, which we have described previously.[1—3]

In this work, we studied the reactions of *o*-tosylaminobenzaldehyde **1** with sulfanilamide (streptocid) (**2a**), 2-sulfanilamidothiazole (norsulfazole) (**2b**), 4-sulfanilamido-2,6-dimethoxypyrimidine (sulfadimethoxine) (**2c**), and 2-sulfanilamido-4,6-dimethylpyrimidine (sulfadimesine) (**2d**). The reactions with **2a—c** afforded the corresponding derivatives of azabicyclo[3.3.1]nonane **3a—c** (Scheme 1).

However, we failed to obtain product **3** with R = 4,6-dimethyl-2-pyrimidinyl (**3d**) because in this case, the reaction ceased at the stage of formation of the corresponding Schiff base, which did no enter into further condensation.

In the ^{1}H NMR spectra of the azabicyclo[3.3.1]nonane derivatives containing alkyl substituents at the bridging nitrogen atom, which were prepared previously,[1—3] the signal of the methine protons is observed at about δ 6. In the ^{1}H NMR spectra of compounds **3a—c**, this signal shifts substantially to the region typical of aromatic protons. The proposed structure

Scheme 1



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was confirmed by X-ray diffraction study of compound **3a**. Based on the results of X-ray diffraction study of compound **3a**, it is reasonable to attribute the downfield shift of the signal of the methine protons in compounds **3a—c** to the fact that their orientations are restricted within the cone of the shielding of the benzene rings of the sulfonamide substituents.

X-ray diffraction study of compound **3a** revealed the disorder of the sulfanilamide group (between three orientations) and the solvate water molecule (between two orientations) (Fig. 1). This disorder is, apparently, determined by the low barrier to rotation of the sulfonamide group about the S—C bond due to the absence of steric hindrance and the possibility of the involvement of this group in intermolecular hydrogen bonds with

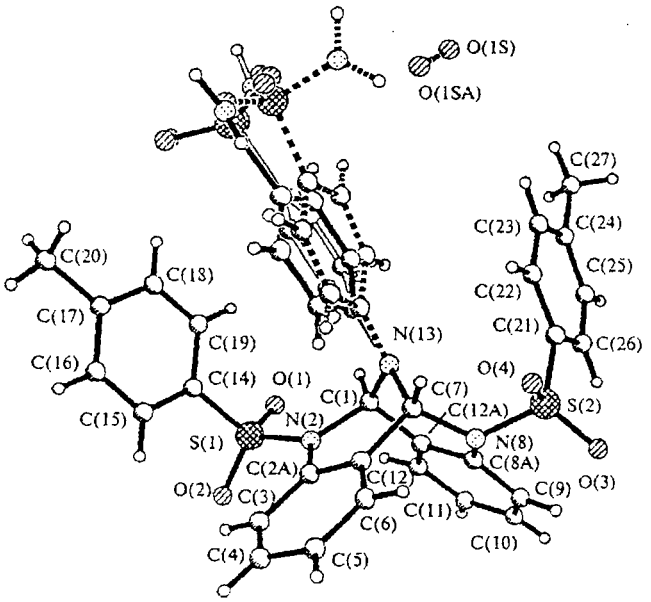

Fig. 1. Overall view of molecule **3a**.

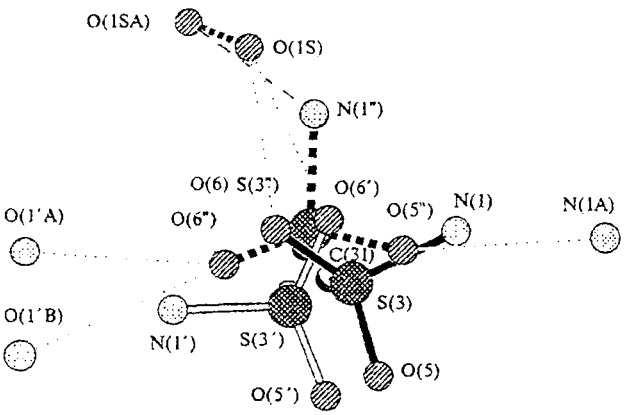

Fig. 2. Disordered fragment of the structure of **3a**; the probable hydrogen bonds are indicated by dashed and dotted lines.

different centers (Fig. 2). Apparently, all possible types of hydrogen bonds occur in the crystal (N(1")...O(1SA), 2.54(2); O(6)...O(1S), 3.03(2); O(6')...O(1S), 3.07(2); O(6")...N(1') ($-x$, 0.5 $-$ y, z), 2.99(2); O(6")...N(1') (-0.25 + y, 0.25 $-$ x, -2.75 $-$ z), 3.00(2); and O(5")...N(1) (0.5 $-$ x, 0.5 $-$ y, -2.5 $-$ z), 2.81(2) Å). Formation of these bonds causes changes in the positions of the sulfonamide substituents with respect to the core of molecule **3a** and as well of the solvate water molecule.

The hydropyrimidine fragments adopt an asymmetrical half-chair conformation (the deviations of the C(1) and N(13) atoms from the mean plane through the remaining atoms of the 1,2,3,4-tetrahydroquinazoline N(2)—C(2A)—C(3)—C(4)—C(5)—C(6)—C(6A)—C(7) system are 0.29 and -0.51 Å, respectively; the deviations of the C(7) and N(13) atoms from the mean plane of the remaining atoms of the 1,2,3,4-tetrahydroquinazoline N(8)-C(8a)-C(9)-C(10)-C(11)-C(12)-C(12A)-C(1) system are 0.31 and -0.53 Å, respectively). The N(8) and N(13) atoms have a pyramidal configuration (the sums of the van der Waals radii are 349.5 and 344.0; 348.7; 347.6° for three positions, respectively). The N(2) atom has a planar-trigonal configuration (the sum of the bond angles is 359.6°), apparently, due to steric hindrances (see Fig. 1). The coordination polyhedra about the sulfur atoms are distorted tetrahedra (the O—S—O bond angles (Table 1) are larger than the ideal tetrahedral value (109.5°)). It is interesting to note that the S(2)—N(8) bond (1.672(4) Å) is substantially longer than the S(1)—N(2) bond (1.647(4) Å) (Table 2). The lengths of the latter bond agrees with the average value of the $S(sp^3)$—$N(sp^2)$ bond (1.64(2) Å).[4]

Experimental

The IR spectra were recorded on a Specord IR-75 instrument as Nujol mulls. The ^1H NMR spectra were measured on a UNITY-300 spectrometer. The synthesis of compound **3b** was carried out with the use of both 2-sulfanilamidothiazole **2b** and its 1 : 1 complex with propylene carbonate, which was formed after recrystallization of **2b** from propylene carbonate.

13-(4-Aminosulfonylphenyl)-6,12-epimino-5,11-ditosyl-5,6,11,12-tetrahydrodibenzo[*b*,*f*]-1,5-diazocine (3a). A solution of *o*-tosylaminobenzaldehyde (0.27 g, 1 mmol) and sulfanilamide (0.15 g, 0.87 mmol) in glacial acetic acid (4 mL) was boiled for 4 h and then cooled. Addition of water afforded a precipitate, which was filtered off and recrystallized from Pr^iH and then from CH_3CN. The yield was 0.2 g (59%). Colorless crystals, m.p. 275—277 °C. Found (%): C, 58.46; H, 4.81; N, 7.95; S, 13.71. $C_{34}H_{30}N_4S_3O_6 \cdot 0.5\ H_2O$. Calculated (%): C, 58.07; H, 4.46; N, 8.06; S, 13.81. IR, ν/cm^{-1}: 3387, 3233 (NH), 1605, 1587 (arom.), 1354, 1341, 1161 (SO_2). ^1H NMR (DMF-d_7) δ: 2.29 (s, 6 H, CH_3); 6.95—8.05 (m, 2 OH, arom.; 2 H, CH, 2 H, NH_2).

13-(4-Thiazol-2-ylaminosulfonylphenyl)-6,12-epimino-5,11-ditosyl-5,6,11,12-tetrahydrodibenzo[*b*,*f*]-1,5-diazocine (3b). *A.* A solution of *o*-tosylaminobenzaldehyde (1.1 g, 4 mmol) and 2-sulfanilamidothiazole (0.51 g, 2 mmol) in glacial acetic acid (15 mL) was boiled for 6 h and filtered without cooling. Then the reaction mixture was cooled. When rubbing slightly with a

Table 1. Bond angles (ω/deg) in the structure of **3a**

Angle	ω/deg	Angle	ω/deg	Angle	ω/deg
O(1)—S(1)—O(2)	119.1(3)	N(8)—C(7)—C(6A)	110.2(4)	C(28)—C(33)—C(32)	121(3)
O(1)—S(1)—N(2)	105.2(2)	C(12A)—C(8A)—C(9)	119.5(5)	O(5)—S(3)—O(6)	130(2)
O(2)—S(1)—N(2)	108.7(2)	C(12A)—C(8A)—N(8)	118.7(4)	O(5)—S(3)—N(1)	98(1)
O(1)—S(1)—C(14)	108.0(3)	C(9)—C(8A)—N(8)	121.7(5)	O(6)—S(3)—N(1)	112(2)
O(2)—S(1)—C(14)	108.1(3)	C(10)—C(9)—C(8A)	119.2(5)	O(5)—S(3)—C(31)	108(1)
N(2)—S(1)—C(14)	107.3(2)	C(9)—C(10)—C(11)	121.9(5)	O(6)—S(3)—C(31)	100(2)
O(4)—S(2)—O(3)	119.5(2)	C(12)—C(11)—C(10)	118.3(5)	N(1)—S(3)—C(31)	108(1)
O(4)—S(2)—N(8)	105.6(2)	C(11)—C(12)—C(12A)	121.8(5)	C(33′)—C(28′)—C(29′)	113(2)
O(3)—S(2)—N(8)	107.8(2)	C(12)—C(12A)—C(8A)	119.1(5)	C(33′)—C(28′)—N(13)	127(2)
O(4)—S(2)—C(21)	109.6(3)	C(12)—C(12A)—C(1)	119.0(5)	C(29′)—C(28′)—N(13)	120(2)
O(3)—S(2)—C(21)	108.2(2)	C(8A)—C(12A)—C(1)	121.8(4)	C(28′)—C(29′)—C(30′)	121(2)
N(8)—S(2)—C(21)	105.2(2)	C(15)—C(14)—C(19)	120.2(5)	C(31′)—C(30′)—C(29′)	118(2)
C(2A)—N(2)—C(1)	115.9(4)	C(15)—C(14)—S(1)	120.9(4)	C(32′)—C(31′)—C(30′)	120(1)
C(2A)—N(2)—S(1)	124.1(3)	C(19)—C(14)—S(1)	119.0(4)	C(32′)—C(31′)—S(3′)	124(2)
C(1)—N(2)—S(1)	119.6(3)	C(16)—C(15)—C(14)	119.2(5)	C(30′)—C(31′)—S(3′)	116(2)
C(8A)—N(8)—C(7)	114.8(4)	C(15)—C(16)—C(17)	121.8(6)	C(31′)—C(32′)—C(33′)	123(2)
C(8A)—N(8)—S(2)	119.6(3)	C(18)—C(17)—C(16)	117.8(6)	C(32′)—C(33′)—C(28′)	125(2)
C(7)—N(8)—S(2)	115.1(3)	C(18)—C(17)—C(20)	121.6(6)	O(5′)—S(3′)—O(6′)	125(2)
C(28′)—N(13)—C(1)	113.4(6)	C(16)—C(17)—C(20)	120.5(6)	O(5′)—S(3′)—N(1′)	106(1)
C(28″)—N(13)—C(1)	128.7(8)	C(19)—C(18)—C(17)	122.1(6)	O(6′)—S(3′)—N(1′)	108(2)
C(1)—N(13)—C(28)	121.4(5)	C(18)—C(19)—C(14)	118.9(6)	O(5′)—S(3′)—C(31′)	108.7(9)
C(28′)—N(13)—C(7)	126.4(8)	C(26)—C(21)—C(22)	121.4(6)	O(6′)—S(3′)—C(31′)	103(2)
C(28″)—N(13)—C(7)	110(1)	C(26)—C(21)—S(2)	119.4(4)	N(1′)—S(3′)—C(31′)	104(1)
C(1)—N(13)—C(7)	108.9(4)	C(22)—C(21)—S(2)	119.1(5)	C(29″)—C(28″)—N(13)	133(4)
C(28)—N(13)—C(7)	115(1)	C(21)—C(22)—C(23)	117.3(7)	C(29″)—C(28″)—C(33″)	120(4)
N(13)—C(1)—N(2)	110.6(4)	C(25)—C(26)—C(21)	119.5(7)	N(13)—C(28″)—C(33″)	107(3)
N(13)—C(1)—C(12A)	109.6(4)	C(24)—C(23)—C(22)	121.3(7)	C(28″)—C(29″)—C(30″)	122(3)
N(2)—C(1)—C(12A)	110.7(4)	C(25)—C(24)—C(23)	119.1(7)	C(29″)—C(30″)—C(31″)	121(2)
C(6A)—C(2A)—C(3)	118.7(4)	C(25)—C(24)—C(27)	120(1)	C(30″)—C(31″)—C(32″)	122(1)
C(6A)—C(2A)—N(2)	117.8(4)	C(23)—C(24)—C(27)	121(1)	C(30″)—C(31″)—S(3″)	118(1)
C(3)—C(2A)—N(2)	123.5(4)	C(24)—C(25)—C(26)	121.4(8)	C(32″)—C(31″)—S(3″)	120(1)
C(4)—C(3)—C(2A)	120.5(5)	C(33)—C(28)—C(29)	118(3)	C(33″)—C(32″)—C(31″)	118(2)
C(3)—C(4)—C(5)	121.2(5)	C(33)—C(28)—N(13)	119(3)	C(32″)—C(33″)—C(28″)	117(2)
C(4)—C(5)—C(6)	118.5(5)	C(29)—C(28)—N(13)	123(2)	O(5″)—S(3″)—O(6″)	127(1)
C(5)—C(6)—C(6A)	121.6(5)	C(30)—C(29)—C(28)	123(2)	O(5″)—S(3″)—N(1″)	100(1)
C(2A)—C(6A)—C(6)	119.5(4)	C(29)—C(30)—C(31)	118(2)	O(6″)—S(3″)—N(1″)	110(1)
C(2A)—C(6A)—C(7)	122.5(4)	C(32)—C(31)—C(30)	120(1)	O(5″)—S(3″)—C(31″)	108(1)
C(6)—C(6A)—C(7)	117.9(4)	C(32)—C(31)—S(3)	122(2)	O(6″)—S(3″)—C(31″)	108.1(9)
N(13)—C(7)—N(8)	108.8(4)	C(30)—C(31)—S(2)	117(1)	N(1″)—S(3″)—C(31″)	101.3(9)
N(13)—C(7)—C(6A)	111.6(4)	C(31)—C(32)—C(33)	121(2)		

rod, a crystalline precipitate formed. The precipitate was filtered off and washed with cold HOAc, Pr^iOH, and hexane. The yield of the crude product was 1.12 g (73%).

B. A solution of *o*-tosylaminobenzaldehyde (1.1 g, 4 mmol) and the complex of 2-sulfanilamidothiazole with propylene carbonate (0.8 g, 2.2 mmol) in glacial HOAc (15 mL) was boiled for 5 h, filtered, and cooled. The crystalline precipitate formed was filtered off, washed with MeOH, and dried. The yield of the crude product was 1.06 g (69%).

The product was twice recrystallized from nitromethane to give the analytically pure compound. Colorless crystals, m.p. 295—300 °C (decomp.). Found (%): C, 57.76; H, 3.97; N, 8.96; S, 16.68. $C_{37}H_{31}N_5S_4O_6$. Calculated (%): C, 57.73; H, 4.03; N, 9.10; S, 16.64. IR, ν/cm^{-1}: 1594; 1581, 1560, 1541 (arom.), 1354, 1341, 1161, 1150 (SO_2). ^1H NMR (DMSO-d_6), δ: 2.15 (s, 6 H, CH_3), 6.75—7.60 (m, 22 H, arom.; 2 H, CH).

13-[(2,6-Dimethoxypyrimidin-4-ylaminosulfonyl)phenyl]-6,12-epimino-5,11-ditosyl-5,6,11,12-tetrahydrodibenzo[*b*,*f*]-1,5-diazocine (3c). A solution of *o*-tosylaminobenzaldehyde (1 g, 1.8 mmol) and 4-sulfanilamido-2,6-dimethoxypyrimidine (0.5 g, 1.6 mmol) in glacial HOAc (15 mL) was boiled for 6 h, filtered, and cooled. The crystalline precipitate formed was filtered off and washed with cold HOAc and hexane. The yield of the crude product was 0.76 g (50%). Recrystallization from nitromethane gave colorless crystals, m. p. 258—260 °C. Found (%): C, 57.88; H, 4.68; N, 10.66; S, 11.95. $C_{40}H_{36}N_6S_3O_8$. Calculated (%): C, 58.25; H, 4.36; N, 10.19; S, 11.65. IR, ν/cm^{-1}: 3233 (NH), 1594, 1587 (arom.), 1354, 1161 (SO_2). ^1H NMR (DMSO-d_6), δ: 2.09 (s, 6 H, CH_3); 3.76 (s, 3 H, OCH_3); 3.78 (s, 3 H, OCH_3); 6.02 (s, 1 H, pyrimidine); 7.00 (s, 2 H, CH); 6.69—7.73 (m, 2 OH, arom.); 11.62 (s, 1 H, NH).

X-ray diffraction study of compound 3a. Crystals of **3a** ($C_{34}H_{30}N_4O_6S_3 \cdot 0.5\ H_2O$, M = 695.81) are tetragonal, space group $I4_1/a$; at 20 °C, a = 21.401(2), c = 27.959(3) Å, V = 12806(2) Å^3, Z = 16, d_{calc} = 1.425 g cm^{-3}. The unit cell parameters and intensities of 6607 reflections were measured on an automated four-circle Siemens P3/PC diffractometer

Table 2. Bond lengths (d) in the structure of **3a**

Bond	d/Å	Bond	d/Å
S(1)—O(1)	1.428(4)	C(21)—C(26)	1.380(8)
S(1)—O(2)	1.433(4)	C(21)—C(22)	1.388(8)
S(1)—N(2)	1.647(4)	C(22)—C(23)	1.41(1)
S(1)—C(14)	1.755(5)	C(26)—C(25)	1.374(9)
S(2)—O(4)	1.423(4)	C(23)—C(24)	1.38(1)
S(2)—O(3)	1.424(4)	C(24)—C(25)	1.37(1)
S(2)—N(8)	1.672(4)	C(24)—C(27)	1.53(1)
S(2)—C(21)	1.752(5)	C(28)—C(33)	1.30(4)
N(2)—C(2A)	1.431(6)	C(28)—C(29)	1.38(4)
N(2)—C(1)	1.503(6)	C(29)—C(30)	1.35(2)
N(8)—C(8A)	1.437(6)	C(30)—C(31)	1.45(2)
N(8)—C(7)	1.483(6)	C(31)—C(32)	1.24(3)
N(13)—C(28′)	1.39(2)	C(31)—S(3)	1.759(8)
N(13)—C(28″)	1.43(4)	C(32)—C(33)	1.47(4)
N(13)—C(1)	1.432(6)	N(1)—S(3)	1.65(1)
N(13)—C(28)	1.45(3)	O(5)—S(3)	1.372(7)
N(13)—C(7)	1.458(6)	O(6)—S(3)	1.372(7)
C(1)—C(12A)	1.504(7)	C(28′)—C(33′)	1.35(3)
C(2A)—C(6A)	1.393(6)	C(28′)—C(29′)	1.39(3)

Bond	d/Å	Bond	d/Å
C(2A)—C(3)	1.405(7)	C(29′)—C(30′)	1.42(3)
C(3)—C(4)	1.378(7)	C(30′)—C(31′)	1.32(3)
C(4)—C(5)	1.380(7)	C(31′)—C(32′)	1.29(3)
C(5)—C(6)	1.383(7)	C(31′)—S(3′)	1.795(8)
C(6)—C(6A)	1.394(7)	C(32′)—C(33′)	1.30(2)
C(6A)—C(7)	1.503(6)	N(1′)—S(3′)	1.65(1)
C(8A)—C(12A)	1.399(7)	O(5′)—S(3′)	1.372(7)
C(8A)—C(9)	1.404(7)	O(6′)—S(3′)	1.372(7)
C(9)—C(10)	1.382(8)	C(28″)—C(29″)	1.33(6)
C(10)—C(11)	1.383(9)	C(28″)—C(33″)	1.58(5)
C(11)—C(12)	1.380(8)	C(29″)—C(30″)	1.39(4)
C(12)—C(12A)	1.390(7)	C(30″)—C(31″)	1.41(2)
C(14)—C(15)	1.379(8)	C(31″)—C(32″)	1.46(2)
C(14)—C(19)	1.390(8)	C(31″)—S(3″)	1.795(8)
C(15)—C(16)	1.372(8)	C(32″)—C(33″)	1.39(3)
C(16)—C(17)	1.378(8)	N(1″)—S(3″)	1.65(1)
C(17)—C(18)	1.377(9)	O(5″)—S(3″)	1.372(7)
C(17)—C(20)	1.507(9)	O(6″)—S(3″)	1.372(7)
C(18)—C(19)	1.367(9)		

Table 3. Coordinates ($\times 10^4$) and equivalent isotropic thermal parameters ($\times 10^3$/Å^2) of nonhydrogen atoms in the structure of **3a**

Atom	x	y	z	U_{eq}
S(1)	984(1)	5943(1)	−13810(1)	43(1)
S(2)	−7(1)	5831(1)	−11488(1)	34(1)
O(1)	381(2)	5781(2)	−13989(1)	55(1)
O(2)	1280(2)	6501(2)	−13979(1)	53(1)
O(3)	−303(2)	6339(2)	−11249(1)	43(1)
O(4)	491(2)	5508(2)	−11259(1)	47(1)
N(2)	896(2)	5996(2)	−13226(1)	33(1)
N(8)	290(2)	6102(2)	−12000(1)	30(1)
N(13)	453(2)	5338(2)	−12612(1)	33(1)
C(1)	308(2)	5755(2)	−12997(2)	33(1)
C(2A)	1378(2)	6190(2)	−12904(2)	29(1)
C(3)	1918(2)	6510(2)	−13051(2)	37(1)
C(4)	2358(2)	6697(2)	−12721(2)	40(1)
C(5)	2272(2)	6593(2)	−12238(2)	40(1)
C(6)	1738(2)	6282(2)	−12092(2)	35(1)
C(6A)	1295(2)	6071(2)	−12419(2)	28(1)
C(7)	758(2)	5690(2)	−12231(2)	29(1)
C(8A)	−105(2)	6439(2)	−12327(2)	33(1)
C(9)	−491(2)	6929(2)	−12172(2)	42(1)
C(10)	−850(3)	7246(2)	−12503(2)	47(2)
C(11)	−825(3)	7110(3)	−12986(2)	50(2)
C(12)	−430(3)	6639(3)	−13135(2)	44(1)
C(12A)	−84(2)	6287(2)	−12813(2)	34(1)
C(14)	1489(3)	5313(3)	−13919(2)	42(1)
C(15)	2125(3)	5404(3)	−13955(2)	48(1)
C(16)	2505(3)	4900(3)	−14044(2)	58(2)
C(17)	2268(3)	4305(3)	−14094(3)	63(2)
C(18)	1633(3)	4225(3)	−14045(3)	75(2)
C(19)	1239(3)	4715(3)	−13958(2)	62(2)
C(20)	2698(4)	3761(3)	−14191(4)	104(3)
C(21)	−586(2)	5296(2)	−11658(2)	39(1)
C(22)	−433(3)	4668(3)	−11698(2)	62(2)
C(26)	−1173(3)	5512(3)	−11779(2)	52(2)
C(23)	−905(5)	4258(3)	−11858(3)	91(3)

Atom	x	y	z	U_{eq}
C(24)	−1490(4)	4476(5)	−11984(3)	101(3)
C(25)	−1614(4)	5100(4)	−11945(3)	83(2)
C(27)	−1999(6)	4024(6)	−12154(5)	182(7)
C(28)	716(13)	4724(12)	−12700(5)	11(6)
C(29)	1319(9)	4568(8)	−12579(5)	21(4)
C(30)	1565(9)	3995(8)	−12658(5)	40(4)
C(31)	1169(9)	3535(5)	−12884(4)	36(4)
C(32)	626(11)	3668(9)	−12996(5)	51(5)
C(33)	373(13)	4297(14)	−12904(6)	61(8)
N(1)	2029(11)	2644(16)	−12579(9)	144(11)
O(5)	1898(7)	2827(7)	−13379(5)	77(4)
O(6)	978(14)	2415(21)	−12938(22)	295(30)
S(3)	1501(3)	2778(3)	−12995(2)	62(1)
C(28′)	605(8)	4737(8)	−12764(4)	14(6)
C(29′)	1145(9)	4450(9)	−12591(5)	23(5)
C(30′)	1313(8)	3837(8)	−12737(6)	27(4)
C(31′)	944(9)	3543(4)	−13044(5)	43(5)
C(32′)	444(8)	3813(8)	−13202(5)	42(4)
C(33′)	283(7)	4375(7)	−13074(5)	32(4)
N(1′)	560(7)	2484(9)	−13484(7)	87(6)
O(5′)	1630(7)	2821(9)	−13566(6)	93(6)
O(6′)	1257(17)	2469(14)	−12788(6)	135(12)
S(3′)	1183(3)	2772(3)	−13217(2)	67(1)
C(28″)	704(21)	4720(20)	−12635(7)	68(10)
C(29″)	1244(15)	4474(16)	−12500(7)	53(9)
C(30″)	1376(8)	3845(8)	−12567(5)	27(4)
C(31″)	935(8)	3447(4)	−12781(4)	38(4)
C(32″)	324(9)	3670(8)	−12941(5)	43(4)
C(33″)	186(11)	4297(11)	−12876(5)	45(6)
N(1″)	900(11)	2362(11)	−12314(6)	103(7)
O(5″)	1767(5)	2578(10)	−12780(8)	96(5)
O(6″)	777(7)	2384(7)	−13197(5)	74(4)
S(3″)	1132(3)	2634(3)	−12836(2)	64(2)
O(1S)	−222(12)	2445(10)	−12029(10)	244(13)

(293 K, λ Mo-Kα radiation, graphite monochromator, θ/2θ scanning technique, θ_{max} = 26°). The structure was solved by the direct method and refined by the full-matrix least-squares method with anisotropic thermal parameters for nonhydrogen atoms. It was found that the sulfanilamide fragment was disordered between three orientations with equal occupancies. This fragment was refined isotropically. X-ray diffraction study, which was then carried out at low temperature (−120 °C), confirmed the statistical disorder of the structure. The solvate water molecule was revealed from the difference Fourier map. This molecule occupies a special position, namely, the twofold axis, and is disordered at two sites about this axis. The hydrogen atoms (except for the hydrogen atoms of the solvate water molecule whose positions were not revealed) were placed in calculated positions and included in the isotropic refinement with fixed positional (the riding model) and thermal parameters (for the methyl groups, U_{iso} of the H atoms were equal to 1.5 U_{iso} of the C atom; for the remaining groups, U_{iso} of the H atoms were equal to 1.2 U_{iso} of the C atom). The coordinates and equivalent isotropic thermal parameters of nonhydrogen atoms are given in Table 3. The final values of the R factors were as follows: R_1 = 0.086 using 6224 independent reflections with $I > 2\sigma(I)$, and wR_2 = 0.254 based on a total of 6260 independent reflections. All calculations were carried out on an IBM PC/AT-486 computer using the SHELXTL PLUS program package.[5] The final refinement was performed using the SHELXL-93 program.

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