

*trans* arrangement for the lone pair of the N atom and the  $\pi$  bond of the C=O group. Data in Table 3 indicate that the geometry of the four-membered SNCO ring closed by an S...O interaction is nearly the same in all three sulphilimines. Nevertheless, the shortest S...O distance (2.708 Å) in DMBES is accompanied by a long C=O bond (1.246 Å) and by a relatively wide C(alkyl)-S...O angle (155.2°), while the longest S...O distance (2.838 Å) in DEDAS is associated with an unusually wide N-C-O bond angle (130.3°) and with the less collinear C(alkyl)-S...O angle (143.0°). Although the C=O bond in DEDAS is not so markedly elongated (1.224 Å), the low  $\nu(\text{CO})$  data (1622 and 1624  $\text{cm}^{-1}$  in KBr and  $\text{CHCl}_3$ , respectively), which are not a consequence of a coupled vibration of the SNCO moiety as they exhibit no shifting on  $^{15}\text{N}$  substitution (Kucsman, Ruff & Tanács, 1967), also point out a significant S...O interaction (*cf.* Schmidt & Tulinsky, 1967; Pinel, Mollier, Llaguno & Paul, 1971).

The authors thank Mr Cs. Kertész for technical aid.

*Acta Cryst.* (1982). **B38**, 1845–1847

### $N^1$ -(3,4-Dimethyl-5-isoxazolyl)sulfanilamide (Sulfisoxazole)

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(Received 26 October 1981; accepted 22 December 1981)

**Abstract.**  $\text{C}_{11}\text{H}_{13}\text{N}_3\text{O}_3\text{S}$ ,  $M_r = 267.3$ , orthorhombic, *Pbca*,  $Z = 8$ ,  $a = 14.917(4)$ ,  $b = 14.506(4)$ ,  $c = 11.570(2)$  Å,  $V = 2503.6$  Å<sup>3</sup>,  $D_c = 1.418$ ,  $D_m$  (floatation) = 1.402  $\text{Mg m}^{-3}$ ,  $F(000) = 1120$ ,  $\mu(\text{Cu K}\alpha) = 2.24$   $\text{mm}^{-1}$ . Refinement with 1765 diffractometer data gave a final  $R = 0.069$ . Estimated standard deviations for bond lengths and angles involving nonhydrogen atoms average 0.005 Å and 0.3° respectively. The crystal structure is stabilized by hydrogen bonds of the types  $\text{NH}\cdots\text{OS}$  and  $\text{NH}\cdots\text{N}$ .

**Introduction.** Sulfisoxazole [ $N^1$ -(3,4-dimethyl-5-isoxazolyl)sulfanilamide] is a sulfonamide drug. Its chemical structure is given in Fig. 1. Though less active than sulfadiazine, it is better tolerated because of its high solubility and rapid excretion. Its significant activity against *Proteus vulgaris* and *E. coli* makes it useful in urinary-tract infections. Preliminary results of the crystal structure of this drug have been reported earlier (Chatterjee, Dattagupta & Saha, 1979) and the structure refinement is reported here.

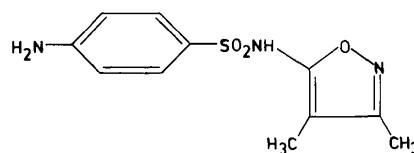


Fig. 1. Sulfisoxazole molecule.

Needle-shaped single crystals of sulfisoxazole were grown by slow evaporation of a solution of the compound in 1:1 water-alcohol mixture at room temperature, and a crystal of dimensions 0.38 × 0.36 × 0.40 mm was used for collecting intensity data.

The structure was initially solved by the heavy-atom method (Chatterjee, Dattagupta & Saha, 1979) using visual data. For further refinement of the structure, diffractometric data were collected on an automatic Stoe diffractometer and 1769 reflections were measured using Ni-filtered  $\text{Cu K}\alpha$  radiation. The  $\theta/2\theta$  scan technique was employed and three standard reflections were monitored after each group of 100 measure-

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Table 1. Positional ( $\times 10^4$ ) and isotropic temperature parameters with *e.s.d.*'s in parenthesesFor non-hydrogen atoms  $B_{eq} = \frac{1}{3} \sum_i \sum_j \beta_{ij} (a_i \cdot a_j)$ .

	x	y	z	$B_{eq}$ or $B$ ( $\text{\AA}^2$ )
S	921 (1)	1659 (1)	703 (1)	3.6
C(1)	-676 (3)	4296 (3)	995 (4)	4.0
C(2)	-129 (3)	4007 (3)	1900 (4)	3.9
C(3)	363 (3)	3206 (3)	1811 (4)	3.7
C(4)	316 (3)	2684 (3)	804 (3)	3.1
C(5)	-218 (3)	2969 (3)	-114 (4)	4.0
C(6)	-723 (3)	3766 (3)	-10 (4)	4.5
C(7)	2328 (3)	2533 (3)	-161 (3)	3.1
C(8)	2977 (3)	3555 (3)	-1250 (3)	3.5
C(9)	2770 (3)	3347 (3)	-86 (3)	3.2
C(10)	2986 (4)	3908 (4)	958 (4)	5.3
C(11)	3465 (4)	4375 (3)	-1710 (4)	5.0
N(1)	-1188 (3)	5068 (3)	1092 (4)	5.7
N(2)	1995 (2)	1937 (2)	667 (3)	3.4
N(3)	2675 (3)	2921 (3)	-1952 (3)	4.3
O(1)	703 (2)	1224 (2)	-368 (3)	5.0
O(2)	831 (2)	1157 (2)	1763 (3)	5.5
O(3)	2256 (2)	2245 (2)	-1261 (2)	4.2
H(N1)	-1435 (3)	5350 (3)	437 (4)	5.3 (2)
H'(N1)	-1066 (3)	5480 (3)	1762 (4)	5.3 (2)
H(N2)	2242 (2)	2023 (2)	1470 (3)	3.3 (1)
H(C2)	-90 (3)	4415 (3)	2680 (4)	3.9 (2)
H(C3)	783 (3)	2984 (3)	2519 (4)	3.6 (2)
H(C5)	-238 (3)	2571 (3)	-903 (4)	3.8 (2)
H(C6)	-1156 (3)	3978 (3)	-710 (4)	4.4 (2)
H(C10)	3347 (4)	4525 (4)	722 (4)	5.0 (2)
H'(C10)	3398 (4)	3491 (4)	1520 (4)	5.0 (2)
H''(C10)	2375 (4)	4093 (4)	1402 (4)	5.0 (2)
H(C11)	3648 (4)	4866 (3)	-1050 (4)	4.8 (2)
H'(C11)	3022 (4)	4700 (3)	-2326 (4)	4.8 (2)
H''(C11)	4062 (4)	4139 (3)	-2148 (4)	4.8 (2)

ments; they did not show any noticeable change in intensity.

The structure was redetermined using direct methods (*MULTAN* program of Main, Germain & Woolfson, 1970) to confirm that previously obtained by the heavy-atom method. Refinement was by the method of least squares, minimizing the function  $\sum w(|F_o| - |F_c|)^2$  where  $w = 1/\sigma^2(F)$ . H atoms were located from a three-dimensional difference Fourier map, and by stereochemical considerations. They were assigned the temperature factors of the heavier atoms to which they are attached. Four strong reflections (002, 020, 321, 420) which exhibited an extinction effect were omitted from the last cycle of least-squares refinement, which converged to  $R = \sum ||F_o| - |F_c|| / \sum F_o = 0.069$  for 1765 reflections. The scattering factors were taken from *International Tables for X-ray Crystallography* (1974). Positional parameters of the atoms together with their standard deviations are given in Table 1.\*

\* Lists of structure factors and anisotropic thermal parameters have been deposited with the British Library Lending Division as Supplementary Publication No. SUP 36686 (15 pp.). Copies may be obtained through The Executive Secretary, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

**Discussion.** The bond lengths and angles are given in Table 2. The average C-H and N-H distances in the structure are 1.08 and 0.98 Å respectively.

The bond distances around the S atom in the sulfoxazole molecule (Table 2) agree well with the corresponding distances in other sulfonamide drugs. Of all the angles around S the angle O(1)-S-O(2) ( $119.8^\circ$ ) is much larger than the standard tetrahedral value, but conforms to a particular nontetrahedral arrangement commonly observed in sulfonamides (Chatterjee, Dattagupta & Saha, 1981).

The isoxazole ring in the structure exhibits a resonance effect. The bond lengths and angles within the ring are comparable to those observed in 3,4'-biisoxazole (Biagini, Cannas & Marongiu, 1969).

Earlier studies (Kálmán, Czugler & Argay, 1981) of compounds containing (aryl)SO<sub>2</sub>NX.X' (X, X' = H, C, N, S, P) groups, as in the present compound, show that for the majority of the compounds the torsion angles about the S<sup>VI</sup>-C(ring) bond fall in the range

Table 2. Bond lengths (Å) and bond angles ( $^\circ$ )

N(1)-C(1)	1.360 (6)	S-N(2)	1.653 (3)
C(1)-C(2)	1.392 (6)	N(2)-C(7)	1.383 (5)
C(2)-C(3)	1.378 (6)	C(7)-O(3)	1.344 (4)
C(3)-C(4)	1.391 (6)	O(3)-N(3)	1.411 (5)
C(4)-C(5)	1.391 (6)	N(3)-C(8)	1.307 (6)
C(5)-C(6)	1.385 (6)	C(8)-C(9)	1.414 (5)
C(6)-C(1)	1.396 (6)	C(8)-C(11)	1.493 (6)
C(4)-S	1.743 (5)	C(9)-C(7)	1.355 (6)
S-O(1)	1.428 (4)	C(9)-C(10)	1.492 (6)
S-O(2)	1.433 (4)		
N(1)-C(1)-C(2)	121.0 (4)	O(1)-S-O(2)	119.8 (2)
N(1)-C(1)-C(6)	119.6 (4)	N(2)-S-C(4)	107.2 (2)
C(6)-C(1)-C(2)	119.3 (4)	S-N(2)-C(7)	121.2 (3)
C(1)-C(2)-C(3)	120.6 (4)	N(2)-C(7)-O(3)	115.6 (3)
C(2)-C(3)-C(4)	119.6 (4)	N(2)-C(7)-C(9)	132.5 (4)
C(3)-C(4)-C(5)	120.4 (4)	C(7)-O(3)-N(3)	106.5 (3)
C(4)-C(5)-C(6)	119.5 (4)	O(3)-N(3)-C(8)	106.8 (4)
C(5)-C(6)-C(1)	120.3 (4)	N(3)-C(8)-C(9)	111.5 (4)
C(3)-C(4)-S	119.6 (3)	N(3)-C(8)-C(11)	120.5 (4)
C(5)-C(4)-S	119.9 (3)	C(11)-C(8)-C(9)	128.0 (4)
C(4)-S-O(1)	108.5 (2)	C(8)-C(9)-C(7)	103.3 (4)
C(4)-S-O(2)	109.1 (2)	C(8)-C(9)-C(10)	127.4 (4)
O(1)-S-N(2)	107.8 (2)	C(10)-C(9)-C(7)	129.2 (4)
O(2)-S-N(2)	103.6 (2)	C(9)-C(7)-O(3)	111.7 (4)

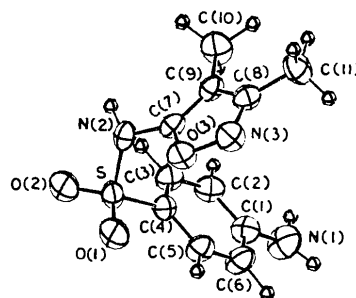


Fig. 2. Perspective (ORTEP) view of the sulfoxazole molecule.

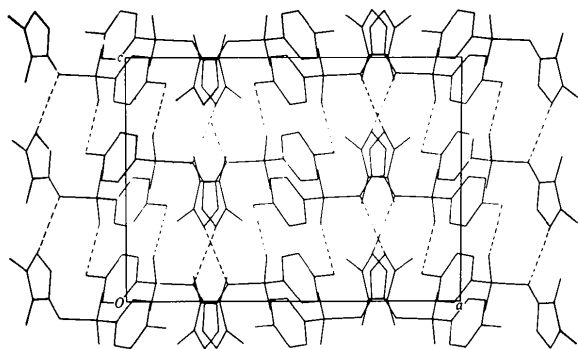


Fig. 3. Projection of the crystal structure down *b*.

Table 3. *Least-squares planes*

Equations of the least-squares planes are in the form  $AX + BY + CZ = D$ , where  $X$ ,  $Y$  and  $Z$  are coordinates in Å. Deviations (Å) of relevant atoms from the planes are given in square brackets.

	<i>A</i>	<i>B</i>	<i>C</i>	<i>D</i>
Plane 1: C(1), C(2), C(3), C(4), C(5), C(6)				
	0.7628	0.5211	-0.3828	2.0368
[C(1) 0.001 (5), C(2) 0.004 (5), C(3) 0.002 (5), C(4) -0.004 (4), C(5) 0.010 (5), C(6) -0.008 (5), S -0.046 (1), N(1) -0.041 (5)]				
Plane 2: C(7), O(3), N(3), C(8), C(9)				
	-0.8674	0.4903	-0.0851	-1.1965
[C(7) 0.002 (4), O(3) -0.002 (3), N(3) 0.005 (4), C(8) -0.004 (4), C(9) 0.001 (4), N(2) -0.073 (3), C(10) 0.018 (6), C(11) -0.007 (6)]				

Dihedral angle (°) between planes 1 and 2: 111.9

70–120° and those about the  $S^{VI}-N$  bond lie in the range 60–90°, irrespective of signs. It may be noted that the torsion angles as obtained by us for the sulfisoxazole molecule are  $C(5)-C(4)-S-N(2) = 112.5^\circ$  and  $C(4)-S-N(2)-C(7) = -56.2^\circ$  respectively.

An ORTEP drawing (Johnson, 1965) of the molecule with the atomic numbering is shown in Fig. 2.

The projection of the structure down *b* is shown in Fig. 3, in which the hydrogen bonds are indicated by broken lines. Of the three available H atoms, two take part in the formation of two intermolecular hydrogen bonds,  $N(1)-H'(N1)\cdots O(2^i)$  [(i):  $-x, \frac{1}{2} + y, \frac{1}{2} - z$ ] and  $N(2)-H(N2)\cdots N(3^{ii})$  [(ii):  $x, \frac{1}{2} - y, \frac{1}{2} + z$ ] with distances 2.990 (6) and 2.943 (5) Å respectively. The relevant hydrogen-bond angles are:  $N(1)-H'(N1)\cdots O(2^i) = 172.4 (1)$ ,  $H'(N1)-N(1)\cdots O(2^i) = 5.1 (3)$  and  $N(2)-H(N2)\cdots N(3^{ii}) = 174.8 (4)$ ,  $H(N2)-N(2)\cdots N(3^{ii}) = 3.4 (2)^\circ$ . The atom H(N1) at N(1) does not take part in hydrogen bonding. A similar situation was also observed in  $\beta$ -sulfanilamide (O'Connell & Maslen, 1967). An overlap of the isoxazole rings is observed along the *y* direction (Fig. 3).

Deviations from least-squares planes are shown in Table 3.

We thank Professor W. Saenger, Max-Planck-Institut für Experimentelle Medizin, Göttingen, West Germany, for his very valuable suggestions and help.

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