

Table 3. *Hydrogen-bond distances (Å) and angles (°)*

X—H...Y	X...Y	H...Y	∠X—H...Y
O(4A)—H(4A)...O(2A ⁱⁱ)	2.471 (2)	1.58 (3)	173 (3)
O(4A ⁱ)—H(4A ⁱ)...O(2A ⁱⁱ)	2.514 (2)	1.62 (3)	172 (3)
O(5B)—H(5B)...O(1A ⁱⁱⁱ)	2.591 (2)	1.73 (3)	163 (3)
O(5B ⁱ)—H(5B ⁱ)...O(3A ^{iv})	2.604 (2)	1.86 (3)	161 (3)
N(2B)—H(2B)...O(1A ^{iv})	2.732 (2)	1.93 (3)	159 (3)
N(2B)—H(2B)...O(3A ^{iv})	3.087 (2)	2.50 (3)	127 (2)
N(2B ⁱ)—H(2B ⁱ)...O(2A ⁱ)	2.729 (2)	1.96 (3)	158 (3)
N(2B ⁱ)—H(2B ⁱ)...O(4A ⁱ)	2.919 (2)	2.35 (3)	127 (2)
N(1B)—H(1B)...O(2A ⁱ)	2.789 (3)	2.26 (3)	119 (2)
N(1B)—H(1B)...O(5B ⁱ)	2.679 (2)	2.41 (3)	99 (2)
N(1B)—H(1B)...O(4A ⁱ)	3.358 (2)	2.53 (3)	159 (3)
N(1B)—H(12B)...O(3A ^{iv})	2.965 (2)	2.17 (3)	158 (3)
N(1B ⁱ)—H(11B ⁱ)...O(3A ^{iv})	2.948 (2)	2.06 (3)	163 (3)
N(1B ⁱ)—H(11B ⁱ)...O(5B ⁱ)	2.674 (2)	2.42 (3)	96 (2)
N(1B ⁱ)—H(11B ⁱ)...O(1A ^{iv})	2.838 (3)	2.59 (3)	96 (2)
N(1B ⁱ)—H(12B ⁱ)...O(5B ⁱⁱⁱ)	3.051 (2)	2.24 (3)	162 (3)
N(1B ⁱ)—H(12B ⁱ)...O(1A ^{iv})	2.838 (3)	2.52 (3)	103 (2)

Other short distances

O(5B)—H(5B)...O(1A ^{iv})	2.957 (2)	3.06 (3)	75 (2)
C(3B)—H(3B)...O(5B ⁱ)	3.031 (2)	2.45 (3)	118 (2)
C(3B ⁱ)—H(3B ⁱ)...O(1A ^{iv})	3.101 (2)	2.35 (3)	146 (3)

Symmetry code

(i) $x - 1, y, z$	(vi) $x + 1, y, z$
(ii) $x, y + 1, z$	(vii) $x + 1, 1 - y, z + 0.5$
(iii) $x, y - 1, z$	(viii) $x, 2 - y, z + 0.5$
(iv) $x - 1, 2 - y, z - 0.5$	(ix) $x + 1, y + 1, z$
(v) x, y, z	(x) $x, 1 - y, z - 0.5$

Upon storage at room temperature for about a year the crystals of the hydrogen oxalate of formamide oxime undergo solid-state transformation into a new crystalline form. Several crystal modifications, including the above-mentioned, have been observed during the crystallization work, all with a short needle axis of about 3.5 Å.

The author wishes to thank Mr Flemming Hansen for collecting the data for the structure determination. The diffractometer and an X-ray generator were

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Structure of N^1 -(2,6-Dimethyl-4-pyrimidinyl)sulphanilamide (Sulfisomidine),* $C_{12}H_{14}N_4O_2S$

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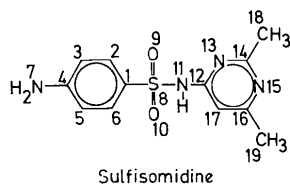
Abstract. $M_r = 278.3$, orthorhombic, $Pca2_1$, $a = 12.614$ (5), $b = 11.278$ (4), $c = 9.374$ (8) Å, $V = 1333.6$ (2) Å³, $Z = 4$, $D_m = 1.378$ (5), $D_x = 1.386$ (5) Mg m⁻³, $\lambda(\text{Mo } K\alpha) = 0.71069$ Å, $\mu =$

0.238 mm⁻¹, $F(000) = 584$, $T = 293$ K. Final $R = 0.037$ for 2006 observed reflections. The angle of twist between the planes of the two rings is 78.6 (2)°. The benzene ring is planar while the pyrimidine ring is slightly folded about the C(12)–N(15) axis. The geometry around the sulphur atom is distorted from the

*4-Amino- N -(2,6-dimethyl-4-pyrimidinyl)benzenesulphonamide.

ideal tetrahedral symmetry. The molecule shows a *gauche* conformation about the S—N bond with a torsion angle of $59.6(2)^\circ$. The crystal structure is stabilized by a network of hydrogen bonds and van der Waals forces. The molecules are stacked together along the diagonals of the *ab* plane.

Introduction. The sulphonamides are well known antibacterial drugs in some common bacterial diseases. They exert their antibacterial action by inhibiting competitively with *para*-aminobenzoic acid (PABA) the enzyme dihydropteroate synthase (DHPS). The inhibition of this enzyme leads to bacteriostasis. As part of our programme on systematic studies on sulphonamides and the enzyme DHPS (Singh, Patel & Haridas, 1984), we report here the crystal structure of sulfisomidine. A brief report on the structure solution was made earlier (Reinhardt, Tiwari & Singh, 1980).



Experimental. Sulfisomidine samples synthesized by Nordmark Werke GmbH, Hamburg (Germany) and obtained on the open market in the form of tablets, crystallized from solution in acetone. Thin needle-like crystals: one $0.70 \times 0.30 \times 0.29$ mm used for data collection. Unit-cell dimensions and space group from oscillation and Weissenberg photographs and diffractometry. Unit-cell dimensions refined using 24 high-angle reflections (θ range: 25 to 43°). Density by flotation in benzene/carbon tetrachloride. Intensity data collected on a computer-controlled Siemens four-circle diffractometer using Nb-filtered Mo $K\alpha$ radiation. Total number of reflections measured 3478, total number of independent reflections 2434, observed reflections ($I \geq 2\sigma$) 2006, for $\sin\theta/\lambda \leq 0.75 \text{ \AA}^{-1}$ with $h = \pm 18$, $k = \pm 16$, $l = 13$. Corrections for Lorentz and polarization effects but not for absorption ($\mu_r = 0.071$), R_{int} (for merged data) = 0.027 . Intensity measurements of standard reflections 002 and 600 repeated after every 50 reflections, variations in intensity throughout experiment $\leq 2.8\%$. Partial structure solved by *MULTAN* (Germain, Main & Woolfson, 1971) and remaining atoms from $\Delta\rho$ maps. The z coordinate of the sulphur atom was held fixed to specify the origin. Refinement by full-matrix least-squares procedure. Positions of hydrogen atoms by successive $\Delta\rho$ maps. Parameters refined using anisotropic temperature factors for non-hydrogen atoms and isotropic temperature factors for hydrogen atoms. Final R for 2006 reflections = 0.037 , $wR = 0.036$, $S = 3.60$. All observed reflections given unit

weights which gave a minimum variation of R as a function of 2θ . $(\Delta/\sigma)_{\text{max}} = 0.058$, final excursions = -0.14 to 0.16 e \AA^{-3} . $|F_o|$ used in full-matrix least-squares refinement using *XRAY76* (Stewart, Machin, Dickinson, Ammon, Heck & Flack, 1976). Atomic scattering factors of Cromer & Mann (1968) for C, N, O and S and of Stewart, Davidson & Simpson (1965) for H. Calculations on the Cyber computer at FU Berlin and HP-1000 system at AIIMS, New Delhi.

Discussion. The final positional and equivalent isotropic temperature factors of non-hydrogen atoms are given in Table 1.* The bond lengths and angles involving non-hydrogen atoms are listed in Table 2. The stereoscopic view with thermal ellipsoids is illustrated in Fig. 1. The crystal structure, hydrogen-bonding scheme and the numbering system are shown in Fig. 2. The average C—H and N—H distances are $0.89(5)$ and $0.78(6) \text{ \AA}$ respectively. The bond lengths and angles in the benzene ring are normal. The bond lengths in the pyrimidine ring range from $1.323(4)$ to $1.414(4) \text{ \AA}$ and valency angles from $118.0(3)$ to $122.1(3)^\circ$. These values are, in general, in good agreement with those found in a standard pyrimidine ring. The value of the endocyclic angle N(13)—C(14)—N(15) is $122.1(3)^\circ$. The corresponding angle is $129.5(9)^\circ$ in sulfadimidine (Tiwari, Haridas & Singh, 1984), $129(1)^\circ$ in sulfadimethoxine (Patel, Tiwari, Patel & Singh, 1983), $128.0(10)^\circ$ in sulfadiazine (Joshi, Tiwari, Patel & Singh, 1983), and $127.1(7)^\circ$ in sulfamerazine (Ravindra Acharya, Kuchela & Kartha, 1982). The large difference is, perhaps, due to the difference in the size of the substituent at the C(14) atom. In the title compound, the substituent at C(14) is a methyl group whereas in the others the substituents are more bulky.

The bonding around the sulphur atom is distorted from the ideal tetrahedral geometry. The maximum and minimum values of angles around the sulphur atom are $115.5(3)$ and $103.9(3)^\circ$ respectively. The deviation from the tetrahedral configuration of 109.5° probably results from steric hindrance of dissimilar groups and from the type of non-bonded interactions described by Bartell (1962). The bond distances around the sulphur atom are S(8)—C(1) = $1.755(3)$, S(8)—O(9) = $1.430(2)$, S(8)—O(10) = $1.451(2)$ and S(8)—N(11) = $1.630(3) \text{ \AA}$. Abrahams (1955) has calculated the S—C single-bond distance to be 1.82 \AA which is close to the sum of the Pauling (1960) covalent radii for sulphur and carbon atoms. On this evidence, the

* Lists of structure factors, anisotropic thermal parameters, hydrogen-atom positions, distances and angles involving hydrogen atoms, parameters of hydrogen bonds and van der Waals distances and the equations of least-squares planes have been deposited with the British Library Lending Division as Supplementary Publication No. SUP 39993 (21 pp.). Copies may be obtained through The Executive Secretary, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

Table 1. Final positional parameters (for C, N, O: $\times 10^4$ and for S: $\times 10^5$) and equivalent isotropic thermal parameters (\AA^2) with standard deviations in parentheses

	<i>x</i>	<i>y</i>	<i>z</i>	<i>U</i> _{eq} [*]
C(1)	10571 (2)	1495 (3)	6410 (4)	3.4 (1)
C(2)	10162 (3)	884 (3)	7555 (4)	4.9 (2)
C(3)	10574 (4)	-202 (4)	7923 (6)	6.4 (3)
C(4)	11410 (3)	-700 (3)	7179 (5)	5.3 (2)
C(5)	11821 (3)	-80 (3)	6026 (5)	4.9 (2)
C(6)	11415 (3)	1015 (3)	5650 (4)	4.4 (2)
N(7)	11805 (3)	-1786 (3)	7560 (6)	8.2 (3)
S(8)	100193 (5)	28648 (6)	59230	3.2 (3)
O(9)	10321 (2)	3125 (2)	4486 (3)	4.9 (1)
O(10)	8889 (1)	2772 (2)	6186 (3)	4.1 (1)
N(11)	10409 (2)	3883 (2)	7040 (3)	3.2 (1)
C(12)	11445 (2)	4111 (2)	7211 (3)	2.9 (1)
N(13)	12205 (2)	3496 (2)	6489 (3)	3.4 (1)
C(14)	13208 (2)	3792 (3)	6689 (4)	3.4 (1)
N(15)	13491 (2)	4683 (2)	7546 (3)	3.3 (1)
C(16)	12775 (2)	5277 (2)	8352 (4)	3.2 (1)
C(17)	11740 (2)	5003 (3)	8193 (3)	3.3 (2)
C(18)	14062 (3)	3106 (3)	5960 (6)	5.1 (2)
C(19)	13213 (3)	6160 (3)	9372 (4)	4.2 (2)

$$* U_{eq} = (U_{11}U_{22}U_{33})^{1/3}.$$

Table 2. Interatomic distances (\AA) and bond angles ($^\circ$)

C(1)–C(2)	1.377 (5)	S(8)–N(11)	1.630 (3)
C(2)–C(3)	1.374 (6)	N(11)–C(12)	1.342 (3)
C(3)–C(4)	1.383 (7)	C(12)–N(13)	1.363 (4)
C(4)–C(5)	1.389 (6)	N(13)–C(14)	1.323 (4)
C(5)–C(6)	1.382 (5)	C(14)–N(15)	1.335 (4)
C(6)–C(1)	1.390 (5)	N(15)–C(16)	1.355 (4)
N(7)–C(4)	1.370 (5)	C(16)–C(17)	1.349 (4)
S(8)–C(1)	1.755 (3)	C(17)–C(12)	1.414 (4)
S(8)–O(9)	1.430 (3)	C(18)–C(14)	1.492 (5)
S(8)–O(10)	1.451 (2)	C(19)–C(16)	1.487 (5)
C(6)–C(1)–C(2)	119.5 (3)	N(11)–S(8)–C(1)	109.5 (1)
S(8)–C(1)–C(6)	120.9 (3)	S(8)–N(11)–C(12)	120.4 (2)
S(8)–C(1)–C(2)	119.7 (2)	N(13)–C(12)–N(11)	121.9 (3)
C(1)–C(2)–C(3)	120.0 (4)	C(17)–C(12)–N(13)	120.0 (2)
C(2)–C(3)–C(4)	121.6 (4)	C(17)–C(12)–N(11)	118.1 (3)
C(3)–C(4)–C(5)	118.1 (4)	C(12)–N(13)–C(14)	118.3 (3)
N(7)–C(4)–C(5)	121.2 (4)	N(15)–C(14)–N(13)	122.1 (3)
N(7)–C(4)–C(3)	120.7 (4)	N(15)–C(14)–C(18)	118.3 (3)
C(4)–C(5)–C(6)	120.7 (4)	C(18)–C(14)–N(13)	122.1 (3)
C(5)–C(6)–C(1)	120.0 (3)	C(14)–C(15)–C(16)	118.3 (3)
O(9)–S(8)–C(1)	108.7 (2)	N(15)–C(16)–C(17)	119.7 (3)
O(9)–S(8)–O(10)	115.9 (2)	C(17)–C(16)–C(19)	122.0 (2)
O(9)–S(8)–N(11)	112.3 (2)	C(19)–C(16)–N(15)	116.3 (3)
O(10)–S(8)–C(1)	106.4 (1)	C(12)–C(17)–C(16)	119.3 (3)
O(10)–S(8)–N(11)	103.8 (1)		

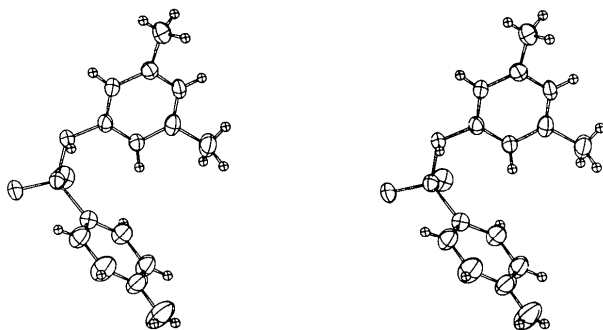


Fig. 1. Molecular structure viewed along [001].

S(8)–C(1) bond distance of 1.755 (3) \AA appears to be considerably shorter. Schomaker & Stevenson (1941) have estimated the S–N single bond to be 1.74 \AA , while in sulphamic acid (Sass, 1960) where the nitrogen atom has no orbital available for π -bonding, the S–N link is 1.76 (2) \AA . Both of the above values are significantly greater than the S(8)–N(11) distance of 1.630 (3) \AA in sulphisomidine indicating a considerable double-bond character for this bond. Cruickshank (1961) has assigned the bond orders of about 0.66 for S–O bonds in the region of 1.43 \AA . The two S–O bonds in the present structure [1.430 (3), 1.451 (2) \AA] therefore possess considerable double-bond character. They also differ from each other by 0.021 \AA which is significant. This may be related to the hydrogen-bond characteristics because O(10) is a proton acceptor in an intermolecular hydrogen bond N(7)–H(71)···O(10) while O(9) does not participate in such an interaction. Recent studies on the behaviour of bonding around the sulphur atom for a large number of structures containing the *N*-substituted-arylsulphonamide moiety have been reported by Kálmán, Czugler & Argay (1981), the results of which generally conform to the above observations.

The benzene ring is planar whereas the pyrimidine ring is slightly folded along the line C(12)–N(15). Atoms C(12), N(13), C(14) and N(15) are coplanar while the atoms N(15), C(16), C(17) and C(12) are almost coplanar. The dihedral angle between the best least-squares planes of the halves of the pyrimidine ring is 4.0 (2)°. This distortion is similar to that observed in the pyrimidine ring of barbital (Patel & Singh, 1984). The planes of the two rings are rotated with respect to each other at 78.8 (2)°. The molecule maintains a *gauche* conformation about the S(8)–N(11) bond with a torsion angle of 59.6 (2)°. The dihedral angles between the planes C(1)–S(8)–O(9) and C(1)–S(8)–O(10), and N(11)–S(8)–O(9) and N(11)–S(8)–O(10) are 69.1 (2) and 54.1 (2)° respectively.

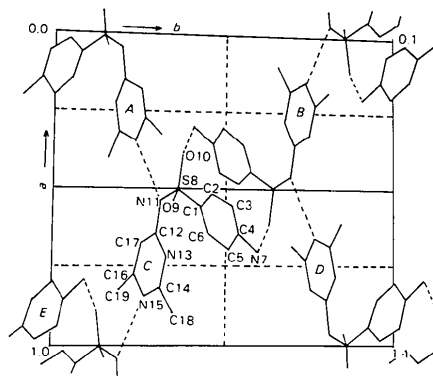


Fig. 2. The molecular packing in the crystal structure as viewed along the *c* axis. The hydrogen bonds are shown by broken lines.

The molecular packing of sulfisomidine as seen down the *c* axis is shown in Fig. 2. The dotted lines indicate the hydrogen bonds. The amino nitrogen atom N(7) is a proton donor in a hydrogen bond with O(10) of the symmetry-related molecule [N(7)—H(71)⋯O(10) = 3.131 (5) Å, 165.5 (5)°] and the imino nitrogen atom N(11) forms a hydrogen bond with N(15) of the symmetry-related molecule [N(11)—H(11)⋯N(15) = 2.948 (3) Å, 149.3 (4)°]. There is one significantly short intermolecular contact distance of van der Waals type between O(9) and N(15) [O(9)⋯N(15) = 2.939 (4) Å]. The molecules are stacked together along the diagonals of the *ab* plane and cross-link each other at the centre of the cell.

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Structure of (7*S*)-*N*-Acetyldemecolcine, C₂₃H₂₇NO₆

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Abstract. $M_r = 413.47$, monoclinic, $P2_1$, $a = 11.629$ (1), $b = 11.748$ (1), $c = 15.782$ (2) Å, $\beta = 97.23$ (1)°, $V = 2138.9$ Å³, $D_m = 1.27$ (1), $D_x = 1.284$ g cm⁻³, $Z = 4$, $\lambda(\text{Cu K}\alpha) = 1.5418$ Å, $\mu = 6.12$ cm⁻¹, $F(000) = 880$, $T = 293$ K, $R = 0.044$ for 2998 reflections. The asymmetric unit of this relatively active colchinoid compound contains two independent molecules which do not differ significantly in bond lengths or angles and the molecular conformations are very similar to each other and to those of previously reported compounds of this type.

Introduction. Colchicine and its analogs are thought to inhibit biological processes by blocking the polymerization of tubulin. While chemical manipulation of colchicine has indicated that aromatic substitution, proper chirality of the acetimido function and a carbonyl group at C(9) are necessary for effective tubulin binding (Capraro & Brossi, 1983), definite conclusions as to the role of these functionalities are lacking. The present structure represents the first of a series of investigations of the stereochemical characteristics of both relatively active and relatively inactive colchinoids.

The general formula of the colchinoids is given as (I) and *N*-acetyldemecolcine ($R_1 = R_2 = R_3 = R_4 = \text{methyl}$, $R_5 = \text{acetyl}$) is a member of the demecolcine

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