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Structure of 4-Phthalimido-*N*-(1,3-thiazol-2-yl)benzenesulfonamide

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Abstract. $C_{17}H_{11}N_3O_4S_2$, $M_r = 385{\cdot}4$, monoclinic, $P2_1/c$, $a = 7{\cdot}955$ (2), $b = 6{\cdot}501$ (1), $c = 35{\cdot}859$ (8) Å, $\beta = 116{\cdot}92$ (2)°, $V = 1653{\cdot}5$ (5) Å^3, $Z = 4$, $D_m = 1{\cdot}560$, $D_x = 1{\cdot}548$ Mg m^{-3}, λ(Cu $K\alpha$) = 1·5418 Å, $\mu = 3{\cdot}13$ mm^{-1}, $F(000) = 792$, $T = 293$ K, final $R = 0{\cdot}061$ for 2116 observed reflections. The molecule exists as an *imido* tautomer with deprotonation of the sulfonamide nitrogen and protonation of the thiazole nitrogen. Centrosymmetrically related pairs of molecules dimerize through hydrogen bonds of the type N–H···O and N–H···N. The thiazole and the phthalimide rings form stacks amongst themselves.

Introduction. Sulfonamides and their derivatives are well known antibacterial drugs. The present compound is a substituted sulfonamide with the substituents a thiazole at the sulfonamide nitrogen, N(1), and a phthalimide ring at the *para* position of the phenyl ring, and is used in the treatment of intestinal infections. The main advantage of this drug is that it does not build up in the blood because of its rapid excretion and poor absorption and hence large doses can be administered without danger of toxic effects. The X-ray structural study of the present compound has been carried out in continuation of our studies (Basak, Mazumdar & Chaudhuri, 1982, 1983, 1984; Basak, Chaudhuri & Mazumdar, 1984) of the substituted sulfonamides in order to obtain detailed structural information and also to study the effects of substituents on the molecular geometry and the change in conformations with the ultimate aim of obtaining a better insight into the activity of these compounds.

Experimental. Transparent, slightly yellowish rectangular crystals from a mixture of acetone and water (1:1), density by flotation (benzene–bromoform), symmetry from oscillation and Weissenberg photographs, $P2_1/c$ (absences: $0k0$, k odd; $h0l$, l odd), crystal size: 0·23 × 0·18 × 0·15 mm, 5051 equivalent reflections collected, Enraf–Nonius CAD-4 diffractometer, moving crystal/moving counter technique, $2 \le 2\theta \le 150°$, −8 to 8 (h), 0 to 7 (k), −40 to 40 (l). Cell parameters from 32 high-angle reflections ($23 \le \theta \le 39°$), $\bar{3}$,0,12 used as intensity standard, average count (= 1361) with σ(calc.) = 106 (7·8%), 2130 reflections were non-zero after merging ($R = 0{\cdot}0909$) with $I \ge 2{\cdot}5\sigma(I)$. No absorption corrections were applied. Structure solved by direct methods using *SHELX*76 (Sheldrick, 1976) with E's ≥ 1·475. Anisotropic full-matrix least-squares refinement based on F with H's (from ΔF synthesis) isotropic gave $R = 0{\cdot}068$.

Further refinement, after omitting 14 reflections with $(F_o - F_c)/\sigma \ge 3{\cdot}0$ (poor agreement probably due to poor quality of the crystals or extinction), gave final $R = 0{\cdot}061$, $wR = 0{\cdot}063$, $R_G = 0{\cdot}079$, $S = 2{\cdot}028$, with $w = 1/\sigma^2(|F_o|) + 0{\cdot}008512(F)^2$. In the final cycle, maximum shift/σ is 0·1, average shift/σ < 0·04; peak heights in final ΔF map from −0·28 to 0·32 e Å^{-3}.

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Scattering factors for the non-H atoms from Cromer & Waber (1965) and for H atoms from Stewart, Davidson & Simpson (1965). All the molecular geometry, intra- and intermolecular calculations were performed with the program *PARST* (Nardelli, 1983).

Discussion. The final atomic parameters are listed in Table 1.* Fig. 1 shows a perspective view of the molecule with atomic numbering scheme. The intramolecular bond lengths and bond angles together with some selected torsion angles are listed in Table 2.

* Lists of structure factors, anisotropic thermal parameters, H-atom parameters, torsion angles and least-squares-planes data have been deposited with the British Library Document Supply Centre as Supplementary Publication No. SUP 43391 (20 pp.). Copies may be obtained through The Executive Secretary, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

Table 1. *Fractional atomic coordinates and equivalent isotropic temperature factors* U_{eq}(Å^2) *for non-hydrogen atoms with e.s.d.'s in parentheses*

$$U_{eq} = \tfrac{1}{3}\sum_i\sum_j U_{ij}a_i^*a_j^*\mathbf{a}_i.\mathbf{a}_j.$$

	x	y	z	U_{eq}
S(1)	0·5938 (2)	0·1645 (2)	0·43526 (3)	0·0477 (5)
S(2)	0·9102 (2)	0·5006 (2)	0·43639 (4)	0·0637 (5)
O(1)	0·5162 (5)	0·0448 (6)	0·4577 (1)	0·065 (1)
O(2)	0·5521 (5)	0·3811 (5)	0·4294 (1)	0·057 (1)
C(1)	0·5086 (5)	0·0520 (6)	0·3849 (1)	0·039 (2)
C(2)	0·4861 (6)	0·1739 (7)	0·3512 (1)	0·043 (2)
C(3)	0·4050 (6)	0·0918 (7)	0·3113 (1)	0·043 (2)
C(4)	0·3508 (5)	−0·1149 (6)	0·3055 (1)	0·036 (2)
C(5)	0·3761 (6)	−0·2363 (7)	0·3393 (1)	0·043 (2)
C(6)	0·4604 (7)	−0·1535 (7)	0·3797 (1)	0·046 (2)
N(1)	0·8168 (6)	0·1218 (6)	0·4590 (1)	0·054 (2)
C(7)	0·9417 (6)	0·2567 (7)	0·4592 (1)	0·047 (2)
C(8)	1·2450 (8)	0·3648 (9)	0·4778 (1)	0·061 (2)
N(2)	1·1277 (6)	0·2179 (7)	0·4795 (1)	0·050 (2)
C(9)	1·1524 (9)	0·5280 (9)	0·4556 (2)	0·070 (3)
N(3)	0·2704 (5)	−0·1988 (5)	0·2647 (1)	0·040 (1)
C(10)	0·3285 (6)	−0·3856 (6)	0·2542 (1)	0·039 (2)
O(3)	0·4618 (4)	−0·4859 (4)	0·2777 (1)	0·052 (1)
C(11)	0·2016 (6)	−0·4200 (7)	0·2091 (1)	0·044 (2)
C(12)	0·1964 (7)	−0·5833 (9)	0·1837 (2)	0·059 (2)
C(13)	0·0645 (8)	−0·5696 (11)	0·1417 (2)	0·072 (2)
C(14)	−0·0516 (8)	−0·4032 (11)	0·1262 (2)	0·068 (2)
C(15)	−0·0472 (7)	−0·2402 (9)	0·1520 (1)	0·061 (2)
C(16)	0·0821 (6)	−0·2538 (7)	0·1940 (1)	0·048 (2)
C(17)	0·1177 (6)	−0·1118 (7)	0·2293 (1)	0·045 (2)
O(4)	0·0397 (5)	0·0460 (6)	0·2294 (1)	0·070 (2)

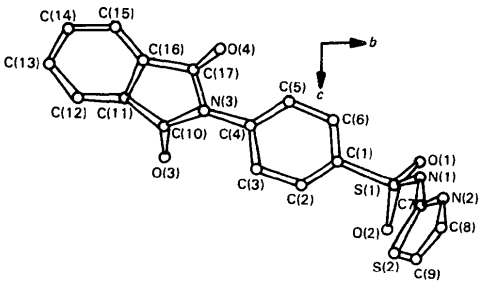

Fig. 1. Perspective view of the molecule down the *a* axis with atomic numbering scheme.

The structure analysis reveals that in the crystalline state the molecule exists as the *imido* tautomer with deprotonation of the sulfonamide nitrogen and protonation of the thiazole nitrogen, N(2). This is indicated by the unequivocal location of the H atom in a ΔF map, successful refinement of its parameters and satisfactory geometry of the hydrogen bonding involving the hydrogen. The preference of a proton for the thiazole nitrogen has also been observed from IR studies (Uno, Machida, Hanai, Ueda & Sasaki, 1963).

The bonding around the sulfur atom S(1) is a distorted tetrahedron similar to that observed in other structures (Basak, Mazumdar & Chaudhuri, 1982, 1983, 1984; Basak, Chaudhuri & Mazumdar, 1984; Cotton & Stokley, 1970; Alléaume, Gulko, Herbstein, Kapon & Marsh, 1976, and references cited therein). The maximum and minimum values of the angle around

Table 2. *Bond distances* (Å), *bond angles* (°) *and some selected torsion angles* (°)

S(1)–O(1)	1·442 (4)	S(1)–O(2)	1·440 (3)
S(1)–C(1)	1·775 (3)	S(1)–N(1)	1·607 (4)
S(2)–C(7)	1·749 (4)	S(2)–C(9)	1·737 (7)
C(1)–C(2)	1·387 (6)	C(1)–C(6)	1·379 (6)
C(2)–C(3)	1·383 (5)	C(3)–C(4)	1·398 (6)
C(4)–C(5)	1·383 (6)	C(4)–N(3)	1·414 (5)
C(5)–C(6)	1·399 (5)	N(1)–C(7)	1·323 (7)
C(7)–N(2)	1·344 (6)	C(8)–N(2)	1·356 (8)
C(8)–C(9)	1·331 (8)	N(3)–C(10)	1·410 (6)
N(3)–C(17)	1·418 (4)	C(10)–O(3)	1·204 (4)
C(10)–C(11)	1·485 (4)	C(11)–C(12)	1·387 (8)
C(11)–C(16)	1·378 (6)	C(12)–C(13)	1·395 (8)
C(14)–C(15)	1·366 (9)	C(14)–C(15)	1·397 (9)
C(15)–C(16)	1·390 (4)	C(16)–C(17)	1·487 (6)
C(17)–O(4)	1·200 (6)		

C(1)–S(1)–N(1)	106·8 (2)	O(2)–S(1)–N(1)	111·9 (2)
O(2)–S(1)–C(1)	107·3 (2)	O(1)–S(1)–N(1)	105·1 (2)
O(1)–S(1)–C(1)	106·4 (2)	O(1)–S(1)–O(2)	118·8 (2)
C(7)–S(2)–C(9)	90·5 (3)	S(1)–C(1)–C(6)	119·4 (3)
S(1)–C(1)–C(2)	119·4 (3)	C(2)–C(1)–C(6)	121·1 (3)
C(1)–C(2)–C(3)	119·8 (4)	C(2)–C(3)–C(4)	119·5 (3)
C(3)–C(4)–N(3)	119·3 (3)	C(3)–C(4)–C(5)	120·4 (3)
C(5)–C(4)–N(3)	120·3 (3)	C(4)–C(5)–C(6)	119·9 (4)
C(1)–C(6)–C(5)	119·1 (4)	S(1)–N(1)–C(7)	122·3 (3)
S(2)–C(7)–N(1)	130·5 (3)	N(1)–C(7)–N(2)	121·2 (4)
S(2)–C(7)–N(2)	108·2 (3)	N(2)–C(8)–C(9)	112·5 (6)
C(7)–N(2)–C(8)	117·0 (4)	S(2)–C(9)–C(8)	111·7 (5)
C(4)–N(3)–C(17)	125·1 (3)	C(4)–N(3)–C(10)	124·0 (3)
C(10)–N(3)–C(17)	110·9 (3)	N(3)–C(10)–C(11)	106·1 (3)
N(3)–C(10)–O(3)	124·8 (3)	O(3)–C(10)–C(11)	129·0 (4)
C(10)–C(11)–C(16)	108·5 (3)	C(10)–C(11)–C(12)	129·3 (4)
C(12)–C(11)–C(16)	122·1 (4)	C(11)–C(12)–C(13)	116·4 (5)
C(12)–C(13)–C(14)	121·9 (6)	C(13)–C(14)–C(15)	121·4 (6)
C(14)–C(15)–C(16)	117·0 (5)	C(11)–C(16)–C(15)	121·1 (4)
C(15)–C(16)–C(17)	130·2 (4)	C(11)–C(16)–C(17)	108·7 (3)
N(3)–C(17)–C(16)	105·7 (3)	C(16)–C(17)–O(4)	129·3 (3)
N(3)–C(17)–O(4)	125·0 (3)		

C(1)–S(1)–N(1)–C(7)	94·5 (4)	O(2)–S(1)–N(1)–C(7)	−22·5 (5)
O(1)–S(1)–N(1)–C(7)	−152·7 (4)	O(2)–S(1)–C(1)–C(2)	23·6 (4)
O(1)–S(1)–C(1)–C(2)	151·7 (4)	O(2)–S(1)–C(1)–C(6)	−154·4 (4)
O(1)–S(1)–C(1)–C(6)	−26·3 (4)	N(1)–S(1)–C(1)–C(6)	85·5 (4)
N(1)–S(1)–C(1)–C(2)	−96·5 (4)	C(9)–S(2)–C(7)–N(1)	−179·6 (5)
C(3)–C(4)–N(3)–C(17)	−50·9 (6)	C(3)–C(4)–N(3)–C(10)	131·4 (4)
C(5)–C(4)–N(3)–C(17)	129·4 (5)	C(5)–C(4)–N(3)–C(10)	−48·4 (6)
S(1)–N(1)–C(7)–S(2)	0·2 (7)	S(1)–N(1)–C(7)–N(2)	179·6 (4)
N(1)–C(7)–N(2)–C(8)	179·7 (5)	C(15)–C(16)–C(17)–O(4)	1·3 (8)
C(11)–C(16)–C(17)–O(4)	−177·6 (5)		

S(1) are 118·8 (2) and 105·1 (2)° respectively. The deviation from the tetrahedral configuration can be attributed to the non-bonded interactions involving the two S—O bonds, resulting in structures with less steric interference (Cotton & Stokley, 1970), and the steric hindrance of dissimilar groups.

The configuration of the phthalimide ring nitrogen, N(3), is almost planar and is at a distance 0·007 (4) Å from the almost planar phenyl ring. The thiazole ring is planar. The thiazole and phenyl rings are folded towards each other, making an angle of 111·3 (1)°. The five-membered ring, N(3), C(17), C(16), C(11), C(10), and the six-membered ring, C(16), C(15), C(14), C(13), C(12), C(11), make an angle of 2·3 (2)°. The dihedral angles between the least-squares planes of the phthalimide ring and the phenyl and thiazole rings are 132·3 (1) and 73·1 (1)° respectively.

The short S(1)—N(1) bond together with marked inequality in the two O—S—N angles indicates a two-coordinated N(1) atom, which provides additional evidence for the *imino* form of the compound (Kálmán, Czugler & Argay, 1981). The N(1)—C(7) bond shows partial double-bond character and the value agrees well with those found in structures (Basak, Chaudhuri & Mazumdar, 1984; Kruger & Gafner, 1971, 1972; Rodier & Masse, 1978; Shefter & Sackman, 1971; Alléaume *et al.*, 1976; Kálmán *et al.*, 1981) where it is in the *imino* form.

The intramolecular dimensions of the thiazole and phthalimide rings agree well with values observed in other compounds containing these moieties.

The conformation of the molecule can be described by the torsion angles about the C(1)—S(1), S(1)—N(1), N(1)—C(7) and C(4)—N(3) central bonds. The torsion angle C(1)—S(1)—N(1)—C(7) lies just outside the clustering range of $|\varepsilon_2| = 60$–$90°$ (Kálmán *et al.*, 1981) but the torsion angles C(x)—C(1)—S(1)—N(1) (where $x = 2$ or 6) lie well inside the clustering range of $|\varepsilon_1| = 70$–$120°$ (Kálmán *et al.*, 1981). The sulfonyl oxygen O(2) which is involved in the larger O—S—N angle is (−)-synperiplanar to C(7) while the other oxygen, O(1), is (−)-antiperiplanar. The rotational flexibility of the thiazole ring about the N(1)—C(7) bond is restricted, as shown by the S(1)—N(1)—C(7)—S(2) and S(1)—N(1)—C(7)—N(2) torsion angles, and this can be explained as due to the S(1)···S(2) [3·320 (2) Å] and N(1)···N(2) [2·324 (6) Å] non-bonded interactions. From the torsion angles about the C(7)—N(2) and C(7)—S(2) central bonds it is evident that the sulfonamide nitrogen, N(1), is antiperiplanar to the thiazole ring. The degree of rotation of the phthalimide ring about the C(4)—N(3) central bonds varies within the range of −48·4 (6) to 131·4 (4)°.

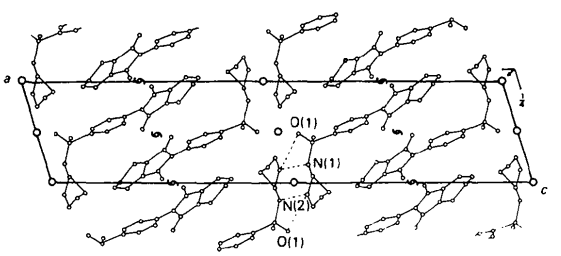

Fig. 2. The packing of the molecules as seen in projection down the *b* axis.

The molecular packing of the crystal viewed down the *b* axis is shown in Fig. 2. The only available proton attached to the thiazole nitrogen, N(2), is the donor in a pair of bifurcated (intermolecular) hydrogen bonds of the type N—H···O (sulfonyl) and N—H···N (*imido*). The angle subtended at the hydrogen, H(1), by the two acceptors is 59 (2)° and is typical for such bifurcated hydrogen bonds (Jeffrey & Maluszynska, 1981). Both hydrogen bonds link molecules related by centres of inversion effectively resulting in dimerization. Two distinct types of ring stacking are observed along the **b** direction; the thiazole rings stack with the thiazole rings while the phthalimide rings stack with the phthalimide rings. The geometrical details of the hydrogen bonding together with the symmetry code are as follows:

D—H···A	D—H (Å)	H···A (Å)	D···A (Å)	∠D—H···A (°)
N(2)—H(1)···O(1^{i})	0·78 (7)	2·62 (6)	3·199 (5)	132 (6)
N(2)—H(1)···N(1^{i})	0·78 (7)	2·25 (7)	3·009 (6)	165 (8)
	∠N(1^{i})···H(1)···O(1^{i}) = 59 (2)°			

Symmetry code: (i) $-x+2, -y, -z+1$.

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