further significance when the physical properties of the heterocycle in solution are examined.

The X-ray structure of 3,4-diphenyl-1,2,5-oxadiazole (Mellini & Merlino, 1976*b*) as well as those of 3,4-diphenyl-1,2,5-thiadiazole and 3,4-diphenyl-1,2,5-selenadiazole (Mellini & Merlino, 1976*a*) show that intermolecular bonds between nitrogen and chalcogen atoms are present only in the selenium derivative. Nevertheless, they are weaker than those observed in the 1,2,5-telluradiazole crystals and do not give rise to a ribbon polymeric structure.

A comparison of the interatomic N—C distances in the four isologous heterocycles shows an increasing localization of the double bond N=C on going from oxygen [1·330 (7) and 1·364 (7) Å] to sulphur [1·327 (5) and 1·342 (6) Å], selenium [1·298 (7) and 1·303 (7) Å] and tellurium [1·273 (10) Å]. The last value agrees well with the expected length (1·275 Å) for a double N=C bond; besides, the observed C—C distance [1·421 (11) Å] is not far from the value of 1·48 Å characteristic of a single $C_{sp^2}$—$C_{sp^2}$ bond. The aromaticity in the 1,2,5-telluradiazole is therefore almost completely lost.

We wish to thank the ISSECC of Florence for collection of the intensity data and for computer time.

### References

Bertini, V. (1967). *Gazz. Chim. Ital.* **97**, 1870–1884.

Bertini, V., Lucchesini, F. & De Munno, A. (1982). *Synthesis*, pp. 681–683.

Hamilton, W. C. (1959). *Acta Cryst.* **12**, 609–610.

*International Tables for X-ray Crystallography* (1974). Vol. IV. Birmingham: Kynoch Press.

Mellini, M. & Merlino, S. (1976*a*). *Acta Cryst.* B**32**, 1074–1078.

Mellini, M. & Merlino, S. (1976*b*). *Acta Cryst.* B**32**, 1079–1082.

Sheldrick, G. M. (1976). *SHELX76*. Program for crystal structure determination. Univ. of Cambridge, England.

Stewart, J. M., Kundell, F. A. & Baldwin, J. C. (1970). The *XRAY70* system. Univ. of Maryland, College Park, Maryland.

Stewart, R. F., Davidson, E. R. & Simpson, W. T. (1965). *J. Chem. Phys.* **42**, 3175–3187.

---

*Acta Cryst.* (1984). C**40**, 655–657

# Structure of 4-Amino-*N*-(4,6-dimethyl-2-pyrimidinyl)benzenesulphonamide (Sulfadimidine), $C_{12}H_{14}N_4O_2S$

By R. K. Tiwari, M. Haridas and T. P. Singh

*Department of Physics, Sardar Patel University, Vallabh Vidyanagar* 388 120, *Gujarat, India*

(*Received* 21 *February* 1983; *accepted* 6 *December* 1983)

**Abstract.** $M_r = 278·3$, monoclinic, $P2_1/c$, $a = 9·27\ (1)$, $b = 18·94\ (2)$, $c = 7·46\ (1)$ Å, $\beta = 97·3\ (3)°$, $V = 1299\ (3)$ Å$^3$, $Z = 4$, $D_m = 1·465\ (1)$, $D_x = 1·423\ (3)$ Mg m$^{-3}$, $\lambda(\mathrm{Cu}\ K\alpha) = 1·5418$ Å, $\mu(\mathrm{Cu}\ K\alpha) = 2·28$ mm$^{-1}$, $F(000) = 584$, room temperature. The structure was determined by direct methods and refined to an $R$ value of 0·065 for 1681 observed reflexions. The mean planes of the benzene and pyrimidine rings are inclined to each other at 78·1 (6)°. The geometry around the S atom is considerably distorted from the ideal tetrahedral geometry. The structure is stabilized by a network of three-dimensional hydrogen bonds and van der Waals forces.

**Introduction.** Sulphonamides are widely used as antibacterial agents in medical practice. As a part of our programme on the X-ray structure determination of sulphonamides (Joshi, Tiwari, Patel & Singh, 1983), we report here the crystal structure of sulfadimidine.

**Experimental.** Thin platy crystals of sulfadimidine from the slow evaporation of a solution in acetonitrile at 283 K, crystal used: 0·81 × 0·32 × 0·72 mm. Unit-cell dimensions and space group from oscillation and Weissenberg photographs. Density by flotation in benzene/carbon tetrachloride. 1681 intensity data recorded photographically ($h$ −11–7, $k$ 0–24, $l$ 0–6), 924 unobserved. Corrections for Lorentz and polarization factors and spot-shape distortions. Structure determined by *MULTAN* (Germain, Main & Woolfson, 1971) and by difference Fourier calculations. Refinement by block-diagonal least squares minimizing $\sum w(\Delta F)^2$. H atoms located by $\Delta F$ synthesis. Final $R$ for 1681 observed reflexions = 0·065, $R_w = 0·069$, $S = 1·84$; $(\Delta/\sigma)_{\max} = 0·60$, final $\Delta\rho$ excursions −0·12–0·15 e Å$^{-3}$. The Hughes (1941) weighting function was used in the final cycle of the refinement with $F_{\min} = 8·0$. Atomic scattering factors from Cromer & Waber (1965) for non-hydrogen atoms and Stewart, Davidson & Simpson (1965) for H atoms.

0108-2701/84/040655-03$01.50

Table 1. *Fractional coordinates* ($\times 10^4$) *and equivalent isotropic thermal parameters* ($\times 10^3$) *of non-hydrogen atoms*

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

| | $x$ | $y$ | $z$ | $U_{eq}$(Å$^2$) |
|---|---|---|---|---|
| C(1) | 6190 (9) | 3924 (4) | 3059 (14) | 7·8 (8) |
| C(2) | 6456 (10) | 3848 (5) | 1316 (14) | 9·1 (9) |
| C(3) | 6725 (10) | 4436 (5) | 314 (15) | 9·6 (10) |
| C(4) | 6777 (9) | 5103 (5) | 1122 (14) | 8·0 (9) |
| C(5) | 6564 (10) | 5172 (4) | 2901 (14) | 8·3 (9) |
| C(6) | 6257 (10) | 4583 (5) | 3912 (17) | 10·3 (10) |
| N(7) | 7065 (9) | 5685 (4) | 165 (14) | 11·1 (9) |
| S(8) | 5908 (2) | 3185 (1) | 4414 (4) | 7·1 (2) |
| O(9) | 7127 (6) | 2720 (3) | 4447 (11) | 9·8 (7) |
| O(10) | 5524 (7) | 3428 (3) | 6099 (10) | 10·5 (7) |
| N(11) | 4585 (7) | 2726 (4) | 3329 (10) | 7·5 (7) |
| C(12) | 3084 (9) | 2843 (5) | 3364 (13) | 7·7 (8) |
| N(13) | 2314 (8) | 2241 (4) | 3179 (13) | 9·9 (8) |
| C(14) | 852 (10) | 2328 (5) | 2987 (17) | 10·6 (10) |
| C(15) | 295 (11) | 2993 (6) | 3080 (21) | 11·8 (14) |
| C(16) | 1166 (10) | 3570 (5) | 3221 (17) | 10·3 (11) |
| N(17) | 2633 (8) | 3489 (4) | 3393 (12) | 8·8 (8) |
| C(18) | −52 (11) | 1672 (6) | 2849 (19) | 12·2 (13) |
| C(19) | 601 (12) | 4304 (6) | 3246 (22) | 14·5 (15) |

**Discussion.** The final positional parameters and equivalent isotropic temperature factors of non-hydrogen atoms are given in Table 1.*

The numbering scheme and the geometry of the molecule (*ORTEP*; Johnson, 1965) are shown in Fig. 1. The bond lengths and angles of sulfadimidine along with those obtained by Basak, Mazumdar & Chaudhuri (1983) are listed in Table 2.† As can be seen from Table 2, there are some significant differences in the molecular dimensions obtained from these independent investigations, but these might be due to the usual fluctuations in data. The endocyclic angle at C(12) of 129·5 (9)° [129·1 (1)° in Basak, Mazumdar & Chaudhuri (1983)] is considerably larger than the value usually observed for a pyrimidine ring. A careful examination of this molecular dimension reveals an interesting aspect of the substitution site and the magnitude of the substituent *versus* N—C—N angle in the pyrimidine ring of sulphonamides. If the substituent is merely a methyl group at atom C(12), as in sulfisomidine (Reinhardt, Tiwari & Singh, 1980), the value of the N—C—N angle is normal [122·1 (3)°]. However, if a more bulky group is attached to this atom, as in sulfadimidine (present study) and sulfadiazine (Patel, Tiwari, Patel & Singh, 1983), the value of the N—C—N angle is rather high (≃ 129°). This is the major molecular change introduced by the substitution and is possibly due to the following factors:

(1) The inherent nature of the pyrimidinyl N atoms to participate in the hydrogen-bonding scheme. This can cause opening of the N(13)—C(12)—N(17) angle.

(2) The presence of a bulky group at C(12) will introduce an additional steric hindrance on the ring system.

The tetrahedral geometry around the S atom is distorted from the ideal fourfold configuration. This is evident from the deviations in the values of the bond angles around S from 109·5° and is further supported by the non-bonded contact distances in this moiety, *i.e.* C(1)···O(9) = 2·61 (1), C(1)···O(10) = 2·60 (1), N(11)···O(9) = 2·398 (9), N(11)···O(10) = 2·52 (1) Å. The planes of the benzene and pyrimidine rings are inclined to each other at 78·1° (6) [75·5° in Basak, Mazumdar & Chaudhuri (1983)]. The molecule shows a *gauche* conformation about the S(8)—N(11) bond with a torsional angle of 84·2 (6)°.

The molecular packing of sulfadimidine as viewed down the *c* axis is shown in Fig. 2. The crystal structure is stabilized by a network of hydrogen bonds and van der Waals forces. The amino nitrogen N($7^{i}$) participates in two unequal hydrogen bonds with the symmetry-related pyrimidinyl N atoms N($13^{v}$) and N($17^{iii}$). The imino N($11^{i}$) forms a hydrogen bond with O($10^{iii}$) of the sulphonyl group.

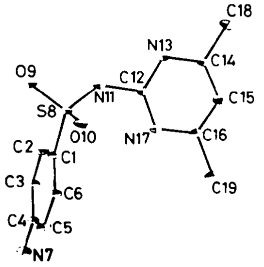

Fig. 1. An *ORTEP* drawing (Johnson, 1965) of sulfadimidine with thermal ellipsoids at the 50% probability level.

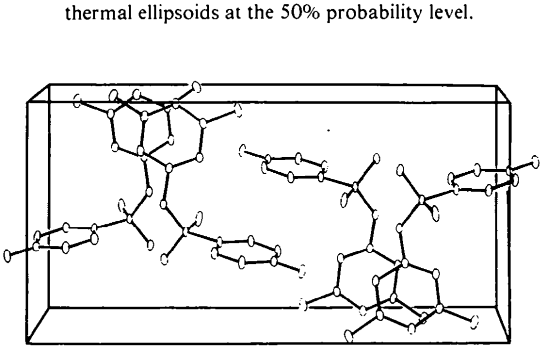

Fig. 2. A projection of the crystal structure along the *c* axis.

---

* Lists of structure factors, anisotropic thermal parameters, H-atom positions, distances and angles involving H atoms, parameters of hydrogen bonds and equations of least-squares planes have been deposited with the British Library Lending Division as Supplementary Publication No. SUP 39064 (17 pp.). Copies may be obtained through The Executive Secretary, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

† *Note:* After this paper had been submitted for publication the work of Basak, Mazumdar & Chaudhuri (1983) on sulfamethazine (sulfadimidine) was brought to our attention. We therefore report only the differences between the two structure determinations and emphasize the points which have not been considered by Basak, Majumdar & Chaudhuri (1983). [An *Abstract* on this structure was communicated to the 14th National Conference on Crystallography held in India (Tiwari & Singh, 1982*b*).]

Table 2. *Comparison of bond lengths* (Å) *and bond angles* (°) *in the two studies, with e.s.d.'s in parentheses*

| | This work | Basak, Mazumdar & Chaudhuri (1983) | | This work | Basak, Mazumdar & Chaudhuri (1983) | | This work | Basak, Mazumdar & Chaudhuri (1983) |
|---|---|---|---|---|---|---|---|---|
| C(1)–C(2) | 1·36 (2) | 1·391 (4) | S(8)–C(1) | 1·765 (9) | 1·746 (3) | C(14)–C(15) | 1·37 (1) | 1·385 (5) |
| C(2)–C(3) | 1·38 (1) | 1·374 (4) | S(8)–O(9) | 1·430 (6) | 1·431 (2) | C(14)–C(18) | 1·50 (1) | 1·499 (5) |
| C(3)–C(4) | 1·40 (1) | 1·400 (4) | S(8)–O(10) | 1·426 (8) | 1·435 (2) | C(15)–C(16) | 1·36 (1) | 1·367 (5) |
| C(4)–C(5) | 1·37 (1) | 1·398 (4) | S(8)–N(11) | 1·632 (7) | 1·632 (2) | C(16)–N(17) | 1·36 (1) | 1·353 (3) |
| C(4)–N(7) | 1·36 (1) | 1·367 (3) | N(11)–C(12) | 1·41 (1) | 1·406 (3) | C(16)–C(19) | 1·49 (2) | 1·497 (5) |
| C(5)–C(6) | 1·40 (1) | 1·371 (3) | C(12)–N(13) | 1·34 (1) | 1·333 (3) | N(17)–C(12) | 1·29 (1) | 1·324 (3) |
| C(6)–C(1) | 1·40 (1) | 1·383 (3) | N(13)–C(14) | 1·36 (1) | 1·347 (3) | | | |
| C(6)–C(1)–C(2) | 121·7 (9) | 119·7 (2) | O(9)–S(8)–C(1) | 108·9 (4) | 109·0 (1) | C(12)–N(13)–C(14) | 114·8 (8) | 115·2 (2) |
| S(8)–C(1)–C(6) | 116·6 (7) | 119·7 (2) | O(9)–S(8)–O(10) | 118·0 (4) | 118·9 (1) | C(15)–C(14)–N(13) | 119·0 (10) | 120·4 (3) |
| S(8)–C(1)–C(2) | 121·5 (7) | 120·4 (2) | O(9)–S(8)–N(11) | 102·9 (4) | 103·1 (1) | C(15)–C(14)–C(18) | 123·0 (10) | 122·0 (3) |
| C(1)–C(2)–C(3) | 120·0 (9) | 119·8 (2) | O(10)–S(8)–C(1) | 108·7 (4) | 108·1 (1) | C(18)–C(14)–N(13) | 116·8 (9) | 117·6 (3) |
| C(2)–C(3)–C(4) | 119·6 (9) | 120·9 (3) | O(10)–S(8)–N(11) | 110·7 (4) | 109·0 (1) | C(14)–C(15)–C(16) | 121·0 (11) | 119·5 (3) |
| N(7)–C(4)–C(5) | 119·6 (9) | 120·4 (2) | N(11)–S(8)–C(1) | 107·1 (4) | 108·2 (1) | C(15)–C(16)–N(17) | 120·0 (10) | 121·1 (3) |
| N(7)–C(4)–C(3) | 120·4 (9) | 121·3 (2) | S(8)–N(11)–C(12) | 126·0 (6) | 128·0 (2) | N(17)–C(16)–C(19) | 117·1 (9) | 116·9 (3) |
| C(3)–C(4)–C(5) | 120·0 (9) | 118·3 (2) | N(13)–C(12)–N(11) | 112·2 (8) | 113·8 (2) | C(19)–C(16)–C(15) | 123·0 (10) | 122·1 (3) |
| C(4)–C(5)–C(6) | 120·8 (9) | 120·6 (2) | N(17)–C(12)–N(13) | 129·5 (9) | 129·1 (2) | C(12)–N(17)–C(16) | 115·3 (8) | 114·8 (2) |
| C(5)–C(6)–C(1) | 117·9 (9) | 120·5 (2) | N(17)–C(12)–N(11) | 118·0 (8) | 117·1 (2) | | | |

Table 3. *Angle of inclination between the planes of the two rings in sulphonamides*

| Name | Angle | Reference |
|---|---|---|
| Sulfisoxazole | 68·1° | Chatterjee, Dattagupta & Saha (1979) |
| Sulfamoxole | 55·2 | Haridas, Kulkarni, Tiwari & Singh (1982) |
| Sulfadimidine | 78·1 | Present study |
| Sulfisomidine | 78·6 | Reinhardt, Tiwari & Singh (1980) |
| Phthalyl sulfacetamide | 61·7 | Patel (1983) |
| Sulfadimethoxine | 77·6 | Patel, Tiwari, Patel & Singh (1983) |
| Sulfadiazine | 75·7 | Joshi, Tiwari, Patel & Singh (1983) |
| Sulfathiazole I | 80·2 | Kruger & Gafner (1972) |
| Sulfathiazole II | 90·4 | Kruger & Gafner (1971) |
| Sulfathiazole III | 91·9 | Kruger & Gafner (1972) |
| Silver sulfadiazine | 68·0 | Cook & Turner (1974) |
| Succinyl sulfathiazole | 83·4 | Rodier, Chauvet & Masse (1978) |
| *N*-(1-Pyridinio)-*p*-chlorobenzene-sulphonamidate | 62·4 | Cameron & Duncanson (1976) |

There is yet another interesting observation which pertains to the mutual orientation of the rings in sulphones and sulphonamides. The two planar rings are generally orthogonal to each other (Table 3). This is mainly due to the steric hindrance caused by the sulphonyl moiety and its participation in hydrogen bonding through proton-acceptor O atoms and proton-donor N atom. In addition, the intramolecular repulsions of the two ring systems and sulphonyl moiety will prefer an orthogonal arrangement of these rings. There is a remarkable example of such a situation in dapsone (Tiwari & Singh, 1982*a*) and 2,4-dinitrobenzyl *p*-tolyl sulphone (Harlow, Simonsen, Pfluger & Sammes, 1974). In dapsone where the two *p*-aminobenzene rings are connected *via* an $-SO_2-$ group, their mutual orientation is 90°. In 2,4-dinitrobenzyl *p*-tolyl sulphone, the distance between the rings is elongated by an additional $-CH_2-$ group thus reducing the influence of repulsive interactions between these ring systems. Under these conditions, the orientation of these rings will be mainly due to crystal forces and packing requirements as found in 2,4-dinitrobenzyl *p*-tolyl sulphone in which the rings are almost coplanar (16·2°).

The authors thank DST (India) for financial support.

### References

BASAK, A. K., MAZUMDAR, S. K. & CHAUDHURI, S. (1983). *Acta Cryst.* C**39**, 492–494.

CAMERON, A. F. & DUNCANSON, F. D. (1976). *Acta Cryst.* B**32**, 1991–1994.

CHATTERJEE, C., DATTAGUPTA, J. K. & SAHA, N. N. (1979). *Indian J. Phys.* **53**A, 648–651.

COOK, D. S. & TURNER, M. F. (1974). *J. Chem. Soc. Perkin Trans.* 2, pp. 1021–1025.

CROMER, D. T. & WABER, J. T. (1965). *Acta Cryst.* **18**, 104–109.

GERMAIN, G., MAIN, P. & WOOLFSON, M. M. (1971). *Acta Cryst.* A**27**, 368–376.

HARIDAS, M., KULKARNI, N. R., TIWARI, R. K. & SINGH, T. P. (1982). *Curr. Sci.* **51**(23), 1111–1112.

HARLOW, R. L., SIMONSEN, S. H., PFLUGER, C. E. & SAMMES, M. P. (1974). *Acta Cryst.* B**30**, 2264–2267.

HUGHES, E. W. (1941). *J. Am. Chem. Soc.* **63**, 1737–1752.

JOHNSON, C. K. (1965). *ORTEP*. Report ORNL-3794. Oak Ridge National Laboratory, Tennessee.

JOSHI, V. V., TIWARI, R. K., PATEL, T. C. & SINGH, T. P. (1983). *Indian J. Phys.* **57**A, 79–89.

KRUGER, G. J. & GAFNER, G. (1971). *Acta Cryst.* B**27**, 326–333.

KRUGER, G. J. & GAFNER, G. (1972). *Acta Cryst.* B**28**, 272–283.

PATEL, U. (1983). Personal communication.

PATEL, U., TIWARI, R. K., PATEL, T. C. & SINGH, T. P. (1983). *Indian J. Phys.* **57**A, 90–99.

REINHARDT, R., TIWARI, R. K. & SINGH, T. P. (1980). *Curr. Sci.* **49**, 586–587.

RODIER, N., CHAUVET, A. & MASSE, J. (1978). *Acta Cryst.* B**34**, 218–221.

STEWART, R. F., DAVIDSON, E. R. & SIMPSON, W. T. (1965). *J. Chem. Phys.* **42**, 3175–3187.

TIWARI, R. K. & SINGH, T. P. (1982*a*). *Indian J. Phys.* **56**A, 420–426.

TIWARI, R. K. & SINGH, T. P. (1982*b*). 14th NSC, IIT Kharagpur, India. Dec. 20–23, Abstract No. II/15.