

Compound (II)*Crystal data*

C₁₇H₁₆N₂O₅
M_r = 328.32
 Monoclinic
Cc
a = 17.9115 (13) Å
b = 11.6279 (13) Å
c = 7.7010 (7) Å
 β = 95.842 (3)°
V = 1595.6 (3) Å³
Z = 4
D_s = 1.367 Mg m⁻³
D_m not measured

Data collection

Siemens SMART area-detector diffractometer
 ω scans
 Absorption correction: multi-scan (SADABS; Sheldrick, 1996)
T_{min} = 0.96, *T_{max}* = 0.968
 4693 measured reflections
 2388 independent reflections

Refinement

Refinement on *F*²
R[*F*² > 2 σ (*F*²)] = 0.043
wR(*F*²) = 0.098
S = 0.912
 2388 reflections
 218 parameters
 H-atom parameters constrained

Mo *K* α radiation
 λ = 0.71073 Å
 Cell parameters from 2974 reflections
 θ = 2.09–28.54°
 μ = 0.102 mm⁻¹
T = 180 (2) K
 Block
 0.40 × 0.38 × 0.32 mm
 Pale yellow

1714 reflections with *I* > 2 σ (*I*)
R_{int} = 0.055
 θ_{\max} = 28.54°
h = -12 → 23
k = -15 → 15
l = -9 → 10
 Intensity decay: none

$w = 1/[\sigma^2(F_o^2) + (0.0442P)^2]$
 where $P = (F_o^2 + 2F_c^2)/3$
 $(\Delta/\sigma)_{\max} < 0.001$
 $\Delta\rho_{\max} = 0.16 \text{ e \AA}^{-3}$
 $\Delta\rho_{\min} = -0.19 \text{ e \AA}^{-3}$
 Extinction correction: none
 Scattering factors from *International Tables for Crystallography* (Vol. C)

Table 2. Selected geometric parameters (Å, °) for (II)

| | | | |
|-------------|-------------|-------------|-----------|
| O1—C5 | 1.397 (3) | N2—C3 | 1.491 (3) |
| O1—N2 | 1.453 (3) | C3—C4 | 1.541 (4) |
| O4—N3 | 1.225 (4) | C4—C5 | 1.506 (4) |
| O5—N3 | 1.233 (4) | | |
| C5—O1—N2 | 104.9 (2) | N2—C3—C4 | 103.3 (2) |
| O1—N2—C3 | 105.68 (19) | C5—C4—C3 | 104.5 (2) |
| O4—N3—O5 | 124.2 (3) | O1—C5—C4 | 106.1 (2) |
| C5—O1—N2—C3 | -39.5 (2) | N2—O1—C5—C4 | 38.0 (2) |
| O1—N2—C3—C4 | 24.2 (3) | C3—C4—C5—O1 | -22.0 (3) |
| N2—C3—C4—C5 | -1.9 (3) | | |

The crystal structure of (II) is non-centrosymmetric, but the Flack parameter [1.9 (13); Flack, 1983] was indeterminate and hence the absolute structure could not be determined.

For both compounds, data collection: SMART (Siemens, 1994); cell refinement: SAINT (Siemens, 1995); data reduction: SAINT; program(s) used to solve structures: SHELXTL/PC (Sheldrick, 1994); program(s) used to refine structures: SHELXL97 (Sheldrick, 1997); molecular graphics: SHELXTL/PC; software used to prepare material for publication: SHELXTL/PC.

We wish to acknowledge the use of the EPSRC's Chemical Database Service at Daresbury Laboratory (Fletcher *et al.*, 1996) for access to the Cambridge Structural Database (Allen & Kennard, 1993).

Supplementary data for this paper are available from the IUCr electronic archives (Reference: CF1330). Services for accessing these data are described at the back of the journal.

References

- Allen, F. H. & Kennard, O. (1993). *Chem. Des. Autom. News*, **8**, 31–37.
 Bonnet-Delpon, D., Bégué, J.-P., Lequeux, T. & Ourevitch, M. (1996). *Tetrahedron*, **52**, 59–70.
 Bravo, P., Bruche, L., Farina, A., Fronza, G., Meille, S. V. & Merli, A. (1993). *Tetrahedron Asymmetry*, **4**, 2131–2134.
 Cremer, D. & Pople, J. A. (1975). *J. Am. Chem. Soc.* **97**, 1354–1358.
 Cum, G., Aversa, M. C. & Uccella, N. (1968). *Gazz. Chim. Ital.* **98**, 782–794.
 Flack, H. D. (1983). *Acta Cryst.* **A39**, 876–881.
 Fletcher, D. A., McMeeking, R. F. & Parkin, D. (1996). *J. Chem. Inf. Comput. Sci.* **36**, 746–749.
 Hammer, J. & Macaluso, A. (1964). *Chem. Rev.* **64**, 473–495.
 Huisgen, R. (1968). *Angew. Chem. Int. Ed. Engl.* **7**, 321–328.
 Sheldrick, G. M. (1994). *SHELXTL/PC Reference Manual*. Version 5.0. Siemens Analytical X-ray Instruments Inc., Madison, Wisconsin, USA.
 Sheldrick, G. M. (1996). *SADABS. Program for Empirical Absorption Correction of Area Detector Data*. University of Göttingen, Germany.
 Sheldrick, G. M. (1997). *SHELXL97. Program for the Refinement of Crystal Structures*. University of Göttingen, Germany.
 Siemens (1994). *SMART*. Version 4.021. *Data Collection Software*. Siemens Analytical X-ray Instruments Inc., Madison, Wisconsin, USA.
 Siemens (1995). *SAINT*. Version 4.021. *Data Integration Software*. Siemens Analytical X-ray Instruments Inc., Madison, Wisconsin, USA.
 Spek, A. L. (1998). *PLATON for Windows*. University of Utrecht, The Netherlands.

Acta Cryst. (1999). **C55**, 1831–1833

A new polymorph of sulfathiazole

DAVID S. HUGHES,^a MICHAEL B. HURSTHOUSE,^b TERRY THRELFALL^c AND STEWART TAVENER^c

^aDepartment of Chemistry, University of Wales Cardiff, PO Box 912, Park Place, Cardiff CF1 3TB, Wales, ^bDepartment of Chemistry, University of Southampton, Highfield, Southampton SO17 1BJ, England, and ^cDepartment of Chemistry, University of York, Heslington, York YO10 5DD, England. E-mail: mbh@soton.ac.uk

(Received 3 June 1999; accepted 26 July 1999)

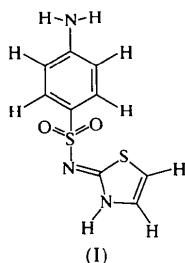
Abstract

Crystals of sulfathiazole, 4-amino-*N*-thiazol-2-ylidenebenzenesulfonamide, C₉H₉N₃O₂S₂, formed from boiling water are shown to be a fifth polymorph. The crystal structure contains two independent molecules, which associate through hydrogen bonds and van der Waals

interactions to produce a two-dimensional sheet structure.

Comment

Sulfathiazole (4-amino-*N*-thiazol-2-ylidenebenzenesulfonamide), (I), first prepared in 1938, was one of the earliest examples of the potent antimicrobial drugs derived from sulfanilamide, commonly called the sulfonamides. The polymorphism of sulfathiazole has been extensively investigated and Burger & Dialer (1983) have described it as the 'classic' polymorphic compound. Four polymorphs have previously been structurally identified (Kruger & Gafner, 1971, 1972; Babilev *et al.*, 1987), and their hydrogen-bonding patterns discussed (Blagden *et al.*, 1998). As part of our own extensive study of the polymorphism of this compound, of its solvates and adducts, and of its potential use as a supramolecular linker, we have shown that the form from boiling water which has long been termed polymorph II in the pharmaceutical literature and which has been assumed to have one of the previously known structures, does in fact have a new structure which therefore identifies a fifth polymorph. A preliminary report of this finding has been published (Hughes *et al.*, 1997), and a structure determination based on powder XRD analysis has appeared (Chan *et al.*, 1999). We present here the single-crystal structure of this new polymorph.



The crystal structure contains two independent molecules which we term *A* and *B*. These associate into two infinite (*AB*) sheets, parallel to the *ab* face of the unit cell and centred approximately at $z = \frac{1}{4}$ and $\frac{3}{4}$. A view of the two independent molecules showing the atom numbering is given in Fig. 1, and the packing diagram, viewed along *a*, shows the layer structure (Fig. 2).

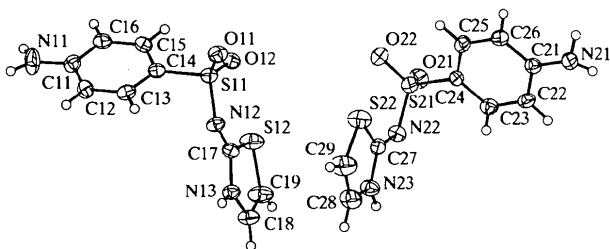


Fig. 1. A general view of the molecular structures of the two independent molecules of sulfathiazole, polymorph V. The ellipsoids are drawn at the 35% probability level and H atoms are shown as spheres of arbitrary radii.

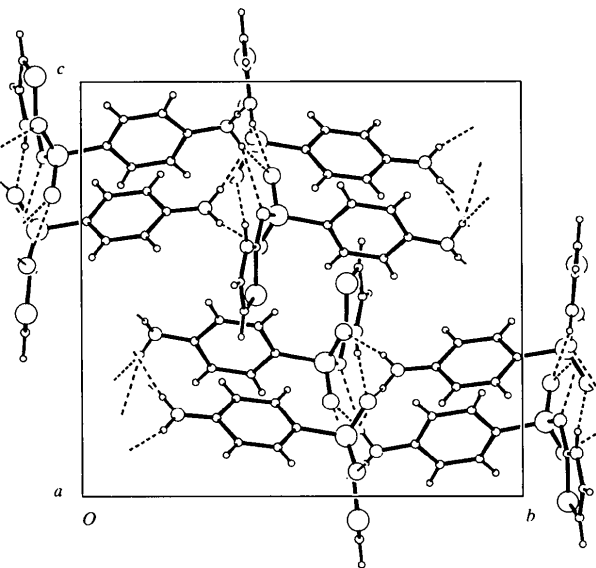


Fig. 2. The unit-cell contents for sulfathiazole, polymorph V, viewed along *a*.

The detailed discussion of the structures of the four previously crystallographically recognized polymorphs concentrated on the networks of molecules connected via 'classical' hydrogen bonds, *i.e.* N—H...N and N—H...O. Not unnaturally, the final descriptions depend critically on the cut-off values for the N...N and N...O contacts. Using the fairly large *D*...*A* value of 3.3 Å (Table 1), we find that, of the hydrogen-bonding interactions in the present structure, all but two are weak and occur only within the layers.

The sheets mentioned above are formed by juxtaposition of alternating rows of *A* and *B* molecules created by 2_1 screw-axis relationships. The two strongest hydrogen bonds, with *D*...*A* distances of 2.794 (4) and 2.865 (4) Å, occur between thiazole N—H groups and sulfonyl O atoms, between *A* and *B* rows. The next set, with *D*...*A* distances in the range 3.010 (4)–3.061 (4) Å, involve the NH₂ H atoms and sulfonyl O atoms, one within an *A* row, one within a *B* row and one between *AB* rows. The remaining contacts identified involve two further amine–sulfonyl-O interactions of 3.116 (4) and 3.211 (4) Å, and one amine–amine interaction of 3.267 (4) Å. Two of the hydrogen bonds, however, involve small *D*—H...*A* angles of 132 (3) and 141 (3)°, and are likely to be very weak. All other intermolecular contacts are greater than the van der Waals radii sums.

Experimental

Crystals of the new polymorph of sulfathiazole were obtained by evaporation to dryness of a boiling aqueous solution, followed by drying above 373 K (Anwar *et al.*, 1989).

Crystal dataC₉H₉N₃O₂S₂ $M_r = 255.31$

Monoclinic

 $P2_1/n$ $a = 10.399 (2) \text{ \AA}$ $b = 15.132 (3) \text{ \AA}$ $c = 14.280 (3) \text{ \AA}$ $\beta = 91.21 (3)^\circ$ $V = 2246.6 (8) \text{ \AA}^3$ $Z = 8$ $D_x = 1.51 \text{ Mg m}^{-3}$ D_m not measuredMo $K\alpha$ radiation $\lambda = 0.71073 \text{ \AA}$

Cell parameters from 2749

reflections

 $\theta = 3.05\text{--}25.00^\circ$ $\mu = 0.462 \text{ mm}^{-1}$ $T = 150 (2) \text{ K}$

Block

 $0.15 \times 0.10 \times 0.08 \text{ mm}$

Colourless

Data collection

Nonius KappaCCD area-detector diffractometer

 $\varphi + \omega$ scans to fill the Ewald sphere

Absorption correction:

multi-scan (SORTAV;

Blessing, 1995)

 $T_{\min} = 0.868, T_{\max} = 0.990$

28 369 measured reflections

3936 independent reflections

2749 reflections with

 $I > 2\sigma(I)$ $R_{\text{int}} = 0.098$ $\theta_{\max} = 25^\circ$ $h = -12 \rightarrow 12$ $k = -17 \rightarrow 17$ $l = -16 \rightarrow 16$

Intensity decay: none

RefinementRefinement on F^2 $R(F) = 0.046$ $wR(F^2) = 0.079$ $S = 0.858$

3936 reflections

319 parameters

H atoms: see below

 $w = 1/[\sigma^2(F_o^2) + (0.1P)^2]$ where $P = (F_o^2 + 2F_c^2)/3$ $(\Delta/\sigma)_{\max} = -0.038$ $\Delta\rho_{\max} = 0.256 \text{ e \AA}^{-3}$ $\Delta\rho_{\min} = -0.373 \text{ e \AA}^{-3}$

Extinction correction: none

Scattering factors from

International Tables for Crystallography (Vol. C)Table 1. *Hydrogen-bonding geometry* ($\text{\AA}, ^\circ$)

| D—H...A | D—H | H...A | D...A | D—H...A |
|------------------------------|----------|----------|-----------|---------|
| N11—H10...N12 ⁱ | 0.90 (2) | 2.14 (2) | 3.016 (4) | 165 (3) |
| N11—H10...O11 ⁱ | 0.90 (2) | 2.54 (3) | 3.211 (4) | 132 (3) |
| N11—H11...O22 ⁱⁱ | 0.93 (2) | 2.10 (2) | 3.010 (4) | 167 (5) |
| N13—H17...O21 ⁱⁱⁱ | 0.86 | 2.04 | 2.865 (4) | 161 |
| N21—H20...O12 ^{iv} | 0.90 (2) | 2.24 (2) | 3.061 (4) | 152 (3) |
| N21—H21...O21 ^v | 0.90 (2) | 2.37 (3) | 3.116 (4) | 141 (3) |
| N21—H21...N22 ^v | 0.90 (2) | 2.44 (2) | 3.267 (4) | 154 (3) |
| N23—H27...O11 ^{vi} | 0.86 | 1.94 | 2.794 (4) | 173 |

Symmetry codes: (i) $\frac{1}{2} - x, \frac{1}{2} + y, -\frac{1}{2} - z$; (ii) $-x, 1 - y, -z$; (iii) $\frac{1}{2} + x, \frac{1}{2} - y, z - \frac{1}{2}$; (iv) $-x, -y, -z$; (v) $\frac{1}{2} - x, y - \frac{1}{2}, \frac{1}{2} - z$; (vi) $\frac{1}{2} + x, \frac{1}{2} - y, \frac{1}{2} + z$.

The H atoms on N11 and N21 were refined isotropically using *DFIX* restraints (Sheldrick, 1997); all other H atoms were treated as riding atoms.

Data collection: *DENZO* (Otwinowski & Minor, 1997) and *COLLECT* (Hoof, 1998). Cell refinement: *DENZO* and *COLLECT*. Data reduction: *DENZO* and *COLLECT*. Program(s) used to solve structure: *SHELXS97* (Sheldrick, 1990). Program(s) used to refine structure: *SHELXL97* (Sheldrick, 1997). Molecular graphics: *CAMERON* (Chemical Crystallography Laboratory, 1993). Software used to prepare material for publication: *SHELXL97*.

We thank the EPSRC for support of the crystallography facilities at Southampton University.

Supplementary data for this paper are available from the IUCr electronic archives (Reference: FG1566). Services for accessing these data are described at the back of the journal.

References

- Anwar, J., Tarling, S. E. & Barnes, P. (1989). *J. Pharm. Sci.* **78**, 337–342.
- Babilev, F. V., Bel'skii, V. K., Simonov, A. & Arzamastsev, A. P. (1987). *Khim. Farm. Zh.* **21**, 1275–1280.
- Blagden, N., Davey, R. J., Lieberman, H. F., Williams, L., Payne, R., Roberts, R., Rowe, R. & Docherty, R. (1998). *J. Chem. Soc. Faraday Trans.* **94**, 1035–1044.
- Blessing, R. H. (1995). *Acta Cryst.* **A51**, 33–38.
- Burger, A. & Dialer, D. (1983). *Pharm. Acta Helv.* **58**, 72–78.
- Chan, F. C., Anwar, J., Cernik, R., Barnes, P. & Wilson, R. M. (1999). *J. Appl. Cryst.* **32**, 436–441.
- Chemical Crystallography Laboratory (1993). *CAMERON. A Molecular Graphics Package*. Chemical Crystallography Laboratory, University of Oxford, England.
- Hoof, R. (1998). *COLLECT Software*. Nonius BV, Delft, The Netherlands.
- Hughes, D. S., Hursthouse, M. B., Lancaster, R. W., Tavener, S., Threlfall, T. L. & Turner, P. (1997). *J. Pharm. Pharmacol.* **49**, S4, 20.
- Kruger, G. J. & Gafner, G. (1971). *Acta Cryst.* **B27**, 326–333.
- Kruger, G. J. & Gafner, G. (1972). *Acta Cryst.* **B28**, 272–283.
- Otwinowski, Z. & Minor, W. (1997). *Methods Enzymol.* **276**, 307–326.
- Sheldrick, G. M. (1990). *Acta Cryst.* **A46**, 467–473.
- Sheldrick, G. M. (1997). *SHELX97. Release 97-2. Program for the Refinement of Crystal Structures*. University of Göttingen, Germany.

Acta Cryst. (1999). **C55**, 1833–1835

***N*-(4-Chlorophenyl)-*N*-methylcyanamide**

IAN D. CUNNINGHAM,^a MARK E. LIGHT^b AND MICHAEL B. HURSTHOUSE^b

^a*Department of Chemistry, School of Physical Sciences, University of Surrey, Guilford GU2 5XH, England, and*

^b*University of Southampton, Department of Chemistry, Highfield, Southampton SO17 1BJ, England. E-mail: light@soton.ac.uk*

(Received 7 July 1999; accepted 28 July 1999)

Abstract

An X-ray structural analysis of the title compound, C₈H₇ClN₂, shows an essentially planar molecule, but with an unusually long aryl-C to planar-N bond.

Comment

In the course of on-going investigations of *N*-arylcyanoguanidines, the title compound, (1), was prepared and isolated. In simple *N*-arylcyanoguanidines such as *N*-cyano-*N'*-(4-methoxyphenyl)guanidine, (2), the aryl-bearing amino lone pair conjugates predominantly with