Acta Crystallographica Section E
Structure Reports Online
ISSN 1600-5368

Tetragonal polymorph of the 1:1 adduct of sulfathiazole with pyridine

Tatiana N. Drebushchak,[a]* Mikhail A. Mikhailenko,[b] Elena V. Boldyreva[a] and Tatyana P. Shakhtshneider[a]

[a]Institute of Solid State Chemistry and Mechanochemistry, SD Russian Academy of Sciences, Kutateladze 18, Novosibirsk 128, 630128 Russian Federation, and [b]Novosibirsk State University, REC-008, Pirogova Street 2, Novosibirsk 90, 630090 Russian Federation

Correspondence e-mail: tanya@xray.nsu.ru

Key indicators

Single-crystal X-ray study
T = 295 K
Mean σ(C–C) = 0.013 Å
R factor = 0.064
wR factor = 0.132
Data-to-parameter ratio = 12.3

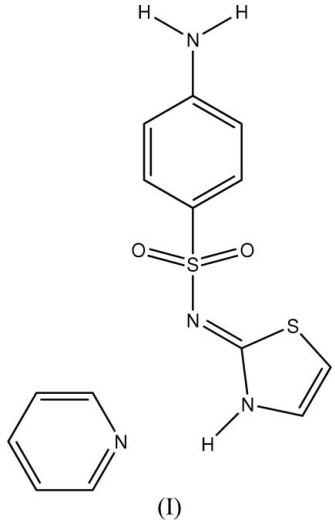
For details of how these key indicators were automatically derived from the article, see http://journals.iucr.org/e.

Received 18 May 2006
Accepted 29 May 2006

The title tetragonal polymorph is one of the two sulfathiazole–pyridine adducts, $C_9H_9N_3O_2S_2{\cdot}C_5H_5N$, that can be formed either by crystallization from an *n*-propanol–pyridine solution, or by exposure of solid sulfathiazole to pyridine vapour. The asymmetric unit consists of a hydrogen-bonded sulfathiazole–pyridine pair. Hydrogen bonds of the form $N_{aniline}$—$H\cdots O_{sulfonyl}$ form a three-dimensional network. Pyridine molecules linked to sulfathiazole molecules by N_{amino}—$H\cdots N_{pyridine}$ hydrogen bonds are located in the channels of the sulfathiazole framework which extend along the 4_1 axis. The angle between neighbouring pyridine rings in the channels is 55.2 (3)°. The adduct is stable in pyridine vapour, but decomposes in air under ambient conditions, giving the metastable polymorph I of sulfathiazole [Kruger & Gafner (1972). *Acta Cryst.* B**28**, 272–283].

Comment

Sulfathiazole gives over 100 solvates, for only a few of which crystal structures have been reported (Bingham *et al.*, 2001). Two polymorphs of the adduct of sulfathiazole with pyridine (1:1) could be crystallized from propanol–pyridine solution (Mikhailenko *et al.*, 2005). The structure of the pyramidal crystals of the tetragonal polymorph, (I), is described in the present contribution.



(I)

The pyramidal crystals of (I) have tetragonal symmetry (space group $P4_1$). The space group $P4_1$ is very rare for molecular crystals (Cambridge Structural Database; Version V5.27; Allen, 2002). Only one of the previously known solvates of sulfathiazole (Bingham *et al.*, 2001) can be

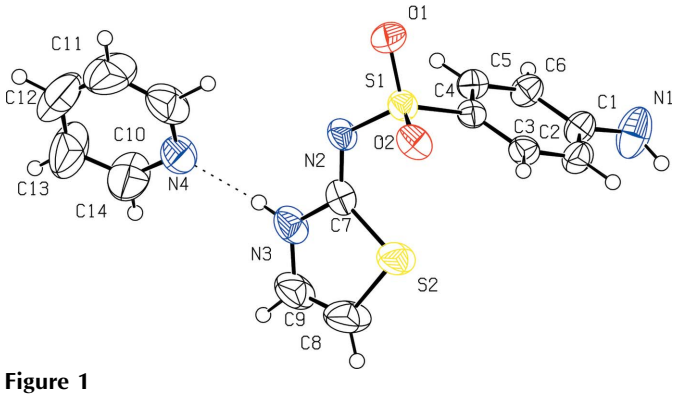


Figure 1
The molecular structure of the 1:1 sulfathiazole adduct with pyridine in the tetragonal polymorph. Displacement ellipsoids are plotted at the 50% probability level. The dashed line indicates a hydrogen bond.

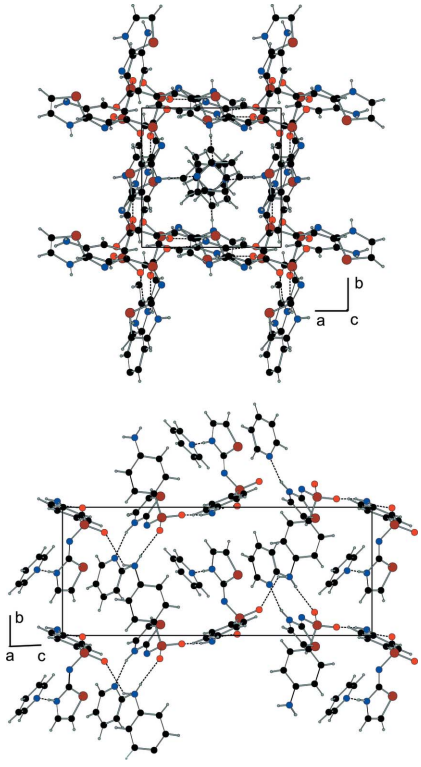


Figure 2
Parts of the structure of the tetragonal 1:1 sulfathiazole adduct with pyridine, shown in two projections. Dashed lines indicate hydrogen bonds.

supposed to be tetragonal, judging from the cell parameters.

The sulfathiazole molecules of (I) exist as imide tautomers (Fig. 1), similar to those found in five polymorphs of pure sulfathiazole (Kruger & Gafner, 1971, 1972; Babilev *et al.*, 1987; Hughes *et al.*, 1999), and in the monoclinic polymorph of the title adduct (Drebushchak *et al.*, 2006). The sulfathiazole–pyridine pairs are linked *via* an $N_{amino}—H\cdots N_{pyridine}$ hydrogen bond in both tetragonal and monoclinic polymorphs. In the tetragonal polymorph, the hydrogen-bonded sulfathiazole molecules (with thiazole and benzene rings being almost normal to each other) form a three-dimensional framework, in which pyridine guests fill the channels, which extend along the 4_1 axis. The angle between neighbouring pyridine rings in the channels is 55.2 (3)°. The parameters characterizing the hydrogen bonds are summarized in Table 1. The components of the crystal structure in two projections are shown in Fig. 2. The adduct can be classified as an appendage inclusion compound (Herbstein, 2005), with the host structure stabilized by the presence of the guest.

The tetragonal polymorph of the adduct is stable in pyridine vapour, but decomposes in air under ambient conditions, giving the metastable polymorph I of sulfathiazole [Kruger & Gafner, 1972; m.p. 474.8 K (Mikhailenko *et al.*, 2005)]. It is interesting that the decomposition of all other sulfathiazole adducts has always been reported to give polymorphs II (Kruger & Gafner,1971), III (Kruger & Gafner, 1972) and, particularly often, IV (Babilev *et al.*, 1987), but never polymorph I (Bingham *et al.*, 2001) of the sulfathiazole.

Experimental

The tetragonal polymorph, (I), of the 1:1 adduct of sulfathiazole with pyridine was obtained by slow evaporation of the solvent from a solution containing sulfathiazole (1.5 g), propanol (40 ml) and pyridine (20 ml). The synthesis of the polymorphs has been described in more detail by Mikhailenko *et al.* (2005).

Crystal data

$C_9H_9N_3O_2S_2\cdot C_5H_5N$
M_r = 334.43
Tetragonal, $P4_1$
a = 8.670 (2) Å
c = 20.927 (10) Å
V = 1573.2 (9) Å^3
Z = 4
D_x = 1.412 Mg m^{-3}
Mo $K\alpha$ radiation
μ = 0.35 mm^{-1}
T = 295 (2) K
Pyramid, colourless
0.46 × 0.41 × 0.38 mm

Data collection

Stoe STADI-4 four-circle diffractometer
ω/θ scans
Absorption correction: ψ scan (*X-RED*; Stoe & Cie, 1997)
T_{min} = 0.771, T_{max} = 0.874
2518 measured reflections
2518 independent reflections
1726 reflections with $I > 2\sigma(I)$
R_{int} = 0.021
θ_{max} = 25.5°
3 standard reflections
frequency: 180 min
intensity decay: 38.0%

Refinement

Refinement on F^2
$R[F^2 > 2\sigma(F^2)]$ = 0.064
$wR(F^2)$ = 0.132
S = 1.17
2518 reflections
205 parameters
H atoms treated by a mixture of independent and constrained refinement
$w = 1/[\sigma^2(F_o^2) + (0.03P)^2 + 1.3187P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} < 0.001$
$\Delta\rho_{max}$ = 0.23 e Å^{-3}
$\Delta\rho_{min}$ = −0.28 e Å^{-3}
Absolute structure: Flack (1983), with 1006 Friedel pairs
Flack parameter: 0.31 (17)

Table 1
Hydrogen-bond geometry (Å, °).

D—H···A	D—H	H···A	D···A	D—H···A
N3—H7···N4	0.86	1.93	2.773 (9)	165
N1—H1···O1^{i}	0.86	2.12	2.976 (9)	176
N1—H2···O2^{ii}	0.86	2.49	3.040 (8)	123

Symmetry codes: (i) $x+1, y, z$; (ii) $y+1, -x+2, z-\frac{1}{4}$.

The tetragonal polymorph is not stable under ambient conditions and loses pyridine easily. Due to this instability, problems were encountered during data collection. One unique set of data was collected completely up to $\theta = 25.5^\circ$, but for the second equivalent set we were forced to stop data collection at $\theta = 22.5^\circ$, because of a strong decay in intensities due to crystal decomposition. For atom N4, U_{eq} is low compared with its neighbours, and this can be interpreted by the fact that N4 is involved in hydrogen-bond formation, in contrast with its neighbours, and therefore a slight disorder or high mobility of pyridine does not affect atom N4 very much – the molecule of pyridine is 'pinned' by atom N4 to the NH group of sulfathiazole. The amino atoms H1 and H2 were located in difference maps. The remaining H atoms were positioned geometrically at C—H = 0.93 and N—H = 0.86 Å. Atom H10 was refined freely but all other H atoms were refined using a riding model, with $U_{iso}(H) = 1.2U_{eq}$ of the carrier atom.

Data collection: *STADI4* (Stoe & Cie, 1997); cell refinement: *STADI4*; data reduction: *X-RED* (Stoe & Cie, 1997); program(s) used to solve structure: *SHELXS97* (Sheldrick, 1990); program(s) used to refine structure: *SHELXL97* (Sheldrick, 1997); molecular graphics: *PLATON* (Spek, 2003) and *PowderCell* (Kraus & Nolze, 1999); software used to prepare material for publication: *SHELXL97*.

This work was supported by grants from BRHE (NO-008-XI, Y3-C-08–01), grants from the Russian Ministry of Education and Science (8156, 2.2.2.3.2007), and grants from RAS (Programs 'Supramolecular Chemistry' and 'Fundamental research for Medicine').

References

Allen, F. H. (2002). *Acta Cryst.* B**58**, 380–388.
Babilev, F. V., Bel'skii, V. K., Simonov, A. & Arzamastsev, A. P. (1987). *Khim. Farm. Zh.* **21**, 1275–1280. (In Russian).
Bingham, A. L., Hughes, D. S., Hursthouse, M. B., Lancaster, R. W., Tavener, S. & Threlfall, T. L. (2001). *Chem. Commun.* pp. 603–604.
Drebushchak, T. N., Mikhailenko, M. A., Boldyreva, E. V. & Shakhtshneider, T. P. (2006). *Acta Cryst.* E. In the press.
Flack, H. D. (1983). *Acta Cryst.* A**39**, 876–881.
Herbstein, F. H. (2005). *Crystalline Molecular Complexes and Compounds. Structures and Principles*, p. 1274. Oxford University Press.
Hughes, D. S., Hursthouse, M. B., Threlfall, T. & Tavener, S. (1999). *Acta Cryst.* C**55**, 1831–1833.
Kraus, W. & Nolze, G. (1999). *PowderCell Software.* Version 2.4. Federal Institute for Materials Research and Testing, Berlin, Germany.
Kruger, G. J. & Gafner, G. (1971). *Acta Cryst.* B**27**, 326–333.
Kruger, G. J. & Gafner, G. (1972). *Acta Cryst.* B**28**, 272–283.
Mikhailenko, M. A., Drebushchak, T. N., Drebushchak, V. A., Boldyreva, E. V. & Boldyrev, V. V. (2005). *J. Cryst. Growth*, **274**, 569–572.
Sheldrick, G. M. (1990). *Acta Cryst.* A**46**, 467–473.
Sheldrick, G. M. (1997). *SHELXL97*. University of Göttingen, Germany.
Spek, A. L. (2003). *J. Appl. Cryst.* **36**, 7–13.
Stoe & Cie (1997). *STADI4* and *X-RED*. Stoe & Cie GmbH, Darmstadt, Germany.