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

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Key indicators

Single-crystal X-ray study
T = 295 K
Mean σ(C–C) = 0.005 Å
R factor = 0.043
wR factor = 0.105
Data-to-parameter ratio = 11.1

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

The asymmetric unit of the title compound, $C_9H_9N_3O_2S_2$·C_5H_5N, contains a sulfathiazole and a pyridine molecule linked *via* N—H⋯N hydrogen bonds, as in the tetragonal polymorph. The monoclinic polymorph is unstable in air at ambient conditions and loses pyridine giving sulfathiazole. The monoclinic polymorph belongs to the class of disappearing polymorphs; it was the first polymorph to be obtained, but could not be obtained again, after the tetragonal polymorph was crystallized.

Comment
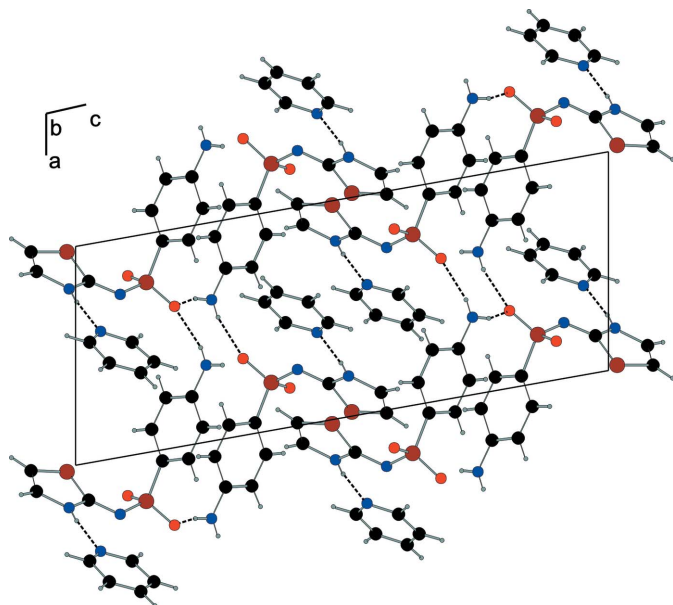
The asymmetric unit of the title compound contains a sulfathiazole and a pyridine molecule linked *via* N—H⋯N hydrogen bonds, as in the tetragonal polymorph (Drebushchak *et al.*, 2006), but comparison of the two structures shows that the two polymorphs differ in the length of the N—H⋯N bond [the N⋯N distance is 2.768 (10) Å in the tetragonal polymorph and 2.822 (4) Å in the monoclinic polymorph], in the conformation of the sulfathiazole molecules, in the mutual orientation of the pyridine and the thiazole rings of sulfathiazole, and by the contacts between neighbouring molecules in the crystal structure. In the monoclinic polymorph, $N_{aniline}$—H⋯$O_{sulfonyl}$ hydrogen bonds link sulfathiazole molecules. The packing pattern is shown in Figs. 2 and 3. Parameters characterizing the geometry of the hydrogen bonds are summarized in Table 1.

(I)
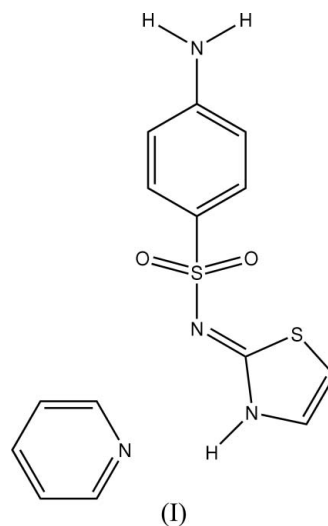
The monoclinic adduct is unstable in air at ambient conditions and loses pyridine molecules (decay 50.3% in this experiment), giving sulfathiazole (Kruger & Gafner, 1972).
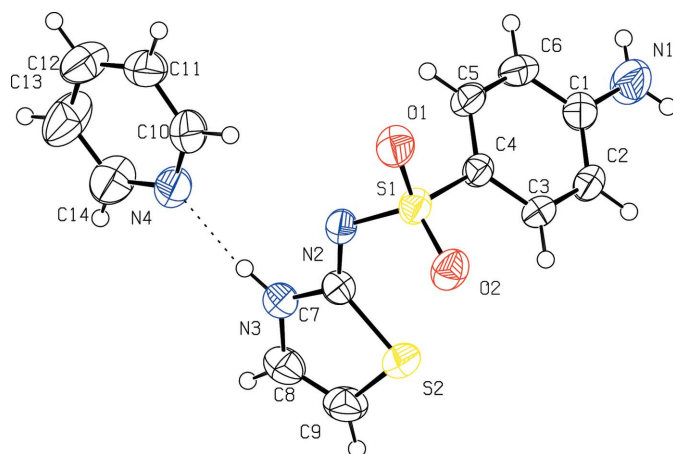
Figure 1
The molecular structure of the sulfathiazole adduct with pyridine in the monoclinic polymorph; displacement ellipsoids are plotted at the 50% probability level. The hydrogen bond is shown as a dashed line.

Figure 3
Packing diagram of the title compound, projected on to the *bc* plane. Dashed lines indicate hydrogen bonds.
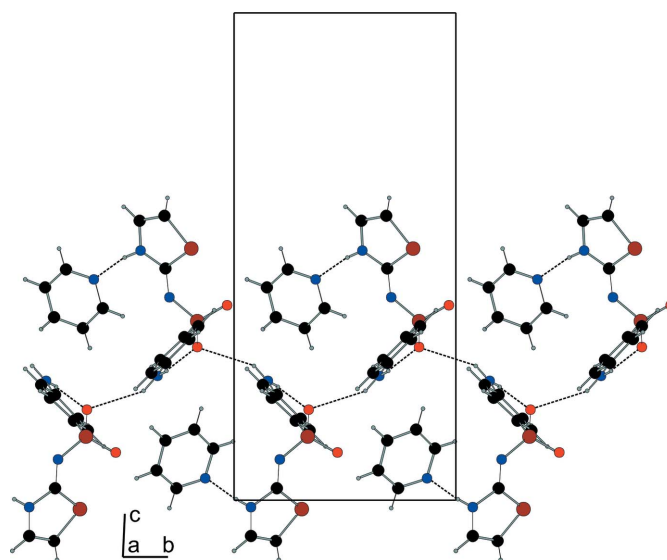

Figure 2
Packing diagram of the title compound, projected on to the *ac* plane. Dashed lines indicate hydrogen bonds.

The monoclinic polymorph belongs to the class of disappearing polymorphs (Dunitz & Bernstein, 1995); it was the first polymorph to be obtained, but could not be obtained again, after the tetragonal polymorph of the same adduct (Drebushchak *et al.*, 2006) was crystallized. This suggests that the monoclinic polymorph is thermodynamically less stable than the tetragonal one.

Experimental

The monoclinic polymorph of the sulfathiazole–pyridine adduct was obtained by slow evaporation of solvent from a solution containing sulfathiazole (1.5 g), propanol (20 ml) and pyridine (40 ml). The crystallization was observed after about 50% of the solvent had evaporated. The synthesis and crystallization of both polymorphs was described by Mikhailenko *et al.* (2005).

Crystal data

$C_9H_9N_3O_2S_2 \cdot C_5H_5N$
$M_r = 334.43$
Monoclinic, $P2_1/c$
$a = 8.385\ (3)$ Å
$b = 9.272\ (2)$ Å
$c = 20.704\ (8)$ Å
$\beta = 100.10\ (3)^\circ$
$V = 1584.7\ (9)$ Å^3

$Z = 4$
$D_x = 1.402$ Mg m^{-3}
Mo $K\alpha$ radiation
$\mu = 0.35$ mm^{-1}
$T = 295\ (2)$ K
Prism, colourless
$0.46 \times 0.19 \times 0.10$ mm

Data collection

Stoe STADI4 four-circle
  diffractometer
ω scans
Absorption correction: none
2335 measured reflections
2335 independent reflections

1764 reflections with $I > 2\sigma(I)$
$\theta_{max} = 24.0^\circ$
3 standard reflections
  frequency: 60 min
  intensity decay: 50.3%

Refinement

Refinement on F^2
$R[F^2 > 2\sigma(F^2)] = 0.043$
$wR(F^2) = 0.105$
$S = 1.10$
2335 reflections
211 parameters
H atoms treated by a mixture of
  independent and constrained
  refinement

$w = 1/[\sigma^2(F_o^2) + (0.0398P)^2 + 0.7805P]$
  where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} < 0.001$
$\Delta\rho_{max} = 0.17$ e Å^{-3}
$\Delta\rho_{min} = -0.26$ e Å^{-3}

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

$D-H\cdots A$	$D-H$	$H\cdots A$	$D\cdots A$	$D-H\cdots A$
N3—H7$\cdots$N4	0.87 (3)	1.96 (3)	2.821 (4)	172 (3)
N1—H1$\cdots$O1^i	0.82 (4)	2.25 (4)	3.059 (5)	168 (3)
N1—H2$\cdots$O1^{ii}	0.84 (4)	2.52 (4)	3.273 (5)	150 (4)

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

All C-bound H atoms were positioned geometrically and refined with a riding model, fixing the bond lengths at 0.93 Å. U_{iso}(H) values were set at $1.2U_{eq}$ of the carrier atom. The H atoms bonded to N were refined isotropically.

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*.

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