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2-(Acetylsulfanyl)pyridine *N*-oxide

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Key indicators

Single-crystal X-ray study
T = 303 K
Mean σ(C–C) = 0.006 Å
R factor = 0.052
wR factor = 0.150
Data-to-parameter ratio = 12.6

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

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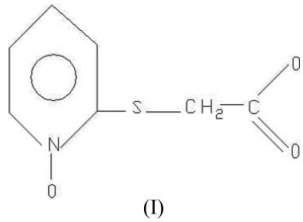
The title compound, $C_7H_7NO_3S$, is a simple new pyridylthio-*N*-oxide of pharmacological interest. There are two independent molecules in the asymmetric unit. Strong O—H···O hydrogen-bond interactions link the molecules in ribbons lying in the (101) plane.

Comment

N-Oxides and their derivatives show a broad spectrum of biological activity, such as antifungal, antibacterial, antimicrobial and antibiotic activities (Lobana & Bhatia, 1989). These compounds are also found to be involved in the DNA strand scission under physiological conditions (Katsuyuki *et al.*, 1991).In view of the importance of *N*-oxide derivatives, the title compound, (I), has been synthesized and the crystal structure determined.



(I)

The asymmetric unit of (I) contains two independent molecules with similar geometry. Both molecules are essentially planar, the maximum deviations from planarity being 0.093 (4) and 0.102 (3) Å for atoms C2 and O11, respectively.

The C—C, C—O and C—S bond lengths and angles are normal (Table 1). The N—O bond lengths are in good

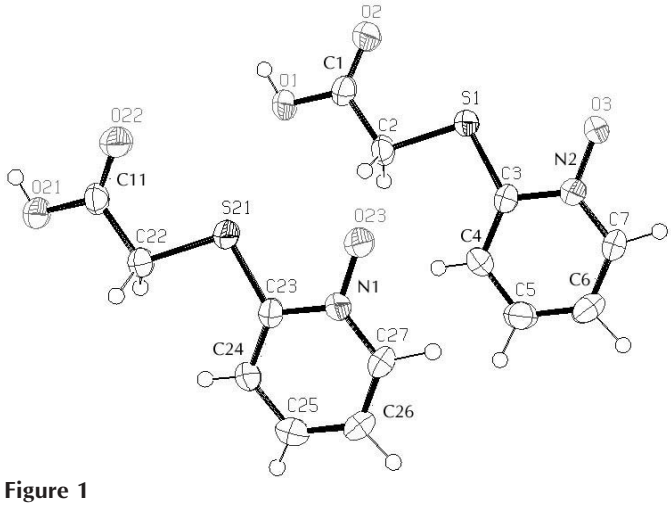


Figure 1
View of the asymmetric unit of the title compound (50% probability displacement ellipsoids).

agreement with the mean value of 1.335 Å reported in the literature for pyridine *N*-oxides (Allen *et al.*, 1987).

In the crystal structure of (I), the molecules are linked by strong O—H···O hydrogen-bond interactions to form ribbons running parallel to the (101) plane (Table 2). In addition, weak C—H···O interactions involving the ribbons are observed.

Experimental

The title compound was prepared by heating a mixture of 1-hydroxy-2-pyridinethione sodium salt (0.447 g, 3 mmol) and chloroacetic acid (0.292 g, 3.1 mmol) in methanol at 343 K with magnetic stirring for 1 h. Fine crystals were obtained after a week by slow cooling of the solution (yield 87%).

Crystal data

$C_7H_7NO_3S$
M_r = 185.20
Monoclinic, $P2_1/c$
a = 9.826 (4) Å
b = 13.596 (2) Å
c = 11.990 (5) Å
β = 102.06 (4)°
V = 1566.6 (10) Å^3
Z = 8
D_x = 1.562 Mg m^{-3}
Mo $K\alpha$ radiation
Cell parameters from 25 reflections
θ = 2–25°
μ = 0.37 mm^{-1}
T = 303 K
Prism, white
0.24 × 0.20 × 0.18 mm

Data collection

Nonius MACH3 four-circle diffractometer
ω–2θ scans
Absorption correction: ψ scan (North *et al.*, 1968)
T_{min} = 0.914, T_{max} = 0.935
3186 measured reflections
2751 independent reflections
1546 reflections with $I > 2\sigma(I)$
R_{int} = 0.060
θ_{max} = 25.0°
$h = 0 \rightarrow 11$
$k = -1 \rightarrow 16$
$l = -14 \rightarrow 13$
3 standard reflections
frequency: 60 min
intensity decay: none

Refinement

Refinement on F^2
$R[F^2 > 2\sigma(F^2)]$ = 0.052
$wR(F^2)$ = 0.150
S = 1.02
2751 reflections
219 parameters
All H-atom parameters refined
$w = 1/[\sigma^2(F_o^2) + (0.075P)^2]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} < 0.001$
$\Delta\rho_{max}$ = 0.34 e Å^{-3}
$\Delta\rho_{min}$ = −0.33 e Å^{-3}

Table 1
Selected geometric parameters (Å, °).

C2—S8	1.806 (4)	C13—N11	1.364 (5)
C3—N1	1.359 (5)	C13—S20	1.736 (4)
C3—S8	1.739 (4)	C17—N11	1.340 (5)
C7—N1	1.346 (5)	N1—O3	1.324 (4)
C12—S20	1.802 (4)	N11—O13	1.323 (4)
O3—N1—C7	122.0 (3)	O13—N11—C13	116.7 (3)
O3—N1—C3	116.4 (3)	C17—N11—C13	121.9 (3)
C7—N1—C3	121.7 (4)	C3—S8—C2	101.01 (19)
O13—N11—C17	121.4 (3)	C13—S20—C12	100.62 (19)

Table 2
Hydrogen-bond geometry (Å, °).

D—H···A	D—H	H···A	D···A	D—H···A
O2—H2···O13^{i}	0.82	1.76	2.568 (5)	167
O12—H12···O3^{ii}	0.82	1.74	2.541 (5)	163
C4—H4···O12^{iii}	0.93	2.51	3.325 (6)	147
C14—H14···O2^{iii}	0.93	2.55	3.344 (6)	144
C6—H6···O1^{iv}	0.93	2.51	3.420 (6)	168
C5—H5···O3^{iv}	0.93	2.45	3.250 (5)	144
C7—H7···O11^{v}	0.93	2.41	3.286 (6)	156
C15—H15···O13^{vi}	0.93	2.51	3.347 (5)	149
C16—H16···O11^{vi}	0.93	2.57	3.477 (6)	164
C17—H17···O1^{vii}	0.93	2.40	3.279 (6)	157

Symmetry codes: (i) $x+1, -y+\frac{3}{2}, z-\frac{1}{2}$; (ii) $x, -y+\frac{3}{2}, z-\frac{1}{2}$; (iii) $-x+1, -y+1, -z$; (iv) $-x+1, y-\frac{1}{2}, -z+\frac{1}{2}$; (v) $x, -y+\frac{3}{2}, z+\frac{1}{2}$; (vi) $-x, y-\frac{1}{2}, -z+\frac{1}{2}$; (vii) $x-1, -y+\frac{3}{2}, z+\frac{1}{2}$.

The H atoms were placed in calculated positions, with C—H = 0.93 or 0.97 Å and O—H = 0.82 Å, and refined using a riding model, with U_{iso} (H) = 1.2U_{eq}(C) or 1.5U_{eq}(O).

Data collection: *CAD-4 EXPRESS* (Enraf–Nonius, 1994); cell refinement: *CAD-4 EXPRESS*; data reduction: *XCAD4* (Harms & Wocadlo, 1995); program(s) used to solve structure: *SHELXS97* (Sheldrick, 1997); program(s) used to refine structure: *SHELXL97* (Sheldrick, 1997); molecular graphics: *PLATON* (Spek, 2003); software used to prepare material for publication: *SHELXL97*.

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