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1-Oxo-6-(2-phenylacetylamino)-1-penicillanic acid

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The crystal structure of the title compound (CAS: 4052–54-4), $C_{16}H_{18}N_2O_5S$, contains O—H···O and N—H···O hydrogen bonds. The four-membered ring is folded and the five-membered ring has an envelope conformation.

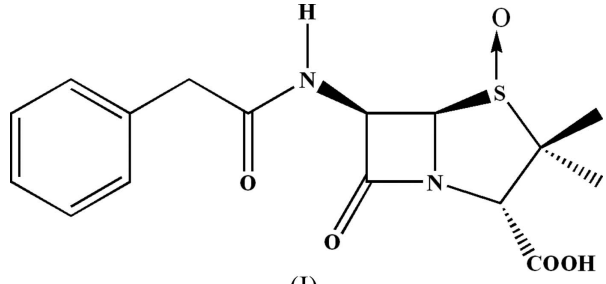
Key indicators

Single-crystal X-ray study
T = 294 K
Mean σ(C–C) = 0.006 Å
R factor = 0.038
wR factor = 0.082
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.

Comment

The title compound, (I), is an early derivative of penicillin which was prepared by Rogers & Folkers (1946). Penicillin G sulfoxide, which is the common name of 1-oxo-6-(2-phenylacetylamino)-1-penicillanic acid, decomposes at higher temperatures (>333 K), because it has an unsaturated S=O bond. It is an important intermediate in the synthesis of 7-aminodeacetoxy-cefalosporanic acid (7-ADCA) (Pan *et al.* 2001).



(I)

Selected bond lengths and angles are listed in Table 1. The four-membered ring (C9–C11,N2) is folded and the five-membered ring (C10,N2,C12,S1,C14) has an envelope conformation. The crystal structure contains intermolecular O—H···O and N—H···O hydrogen bonds (Table 2), giving an infinite three-dimensional network. An intramolecular N—H···O interaction forms a six-membered ring, which also has an envelope conformation, with atom N2 as the flap.

Experimental

1-Oxo-6-(2-phenylacetylamino)-1-penicillanic acid was synthesized according to the method described by Chow *et al.* (1962). Colorless single crystals of (I) were grown by slow evaporation of a methanol solution.

Crystal data

$C_{16}H_{18}N_2O_5S$
M_r = 350.38
Trigonal, $P3_2$
a = 11.6285 (12) Å
c = 10.894 (2) Å
V = 1275.7 (3) Å^3
Z = 3
D_x = 1.368 Mg m^{-3}
Mo $K\alpha$ radiation
μ = 0.22 mm^{-1}
T = 294 (2) K
Block, colourless
0.22 × 0.16 × 0.12 mm

Data collection

Bruker SMART CCD area-detector diffractometer
φ and ω scans
Absorption correction: multi-scan (*SADABS*; Bruker, 1997)
T_{min} = 0.943, T_{max} = 0.974
6825 measured reflections
2703 independent reflections
1994 reflections with $I > 2\sigma(I)$
R_{int} = 0.043
θ_{max} = 25.5°

Refinement

Refinement on F^2
$R[F^2 > 2\sigma(F^2)]$ = 0.039
$wR(F^2)$ = 0.082
S = 1.01
2703 reflections
220 parameters
H-atom parameters constrained
$w = 1/[\sigma^2(F_o^2) + (0.0359P)^2]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max}$ = 0.003
$\Delta\rho_{max}$ = 0.18 e Å^{-3}
$\Delta\rho_{min}$ = −0.16 e Å^{-3}
Absolute structure: Flack (1983), 1116 Freidel pairs
Flack parameter: −0.04 (8)

Table 1
Selected geometric parameters (Å, °).

O1—C8	1.223 (4)	C7—C8	1.504 (4)
O4—C13	1.182 (4)	C12—C13	1.512 (4)
C1—C2	1.371 (5)	C14—C16	1.520 (5)
O3—S1—C10	103.47 (13)	C11—N2—C12	124.2 (2)
O3—S1—C14	105.21 (15)	C10—N2—C12	116.6 (2)
C10—S1—C14	89.03 (14)	C3—C2—C1	119.1 (5)
C4—C5—C6—C7	179.3 (4)	C6—C7—C8—O1	36.7 (5)
C1—C6—C7—C8	99.7 (4)	C8—N1—C9—C10	105.6 (3)
C9—N1—C8—O1	2.7 (4)	C10—N2—C12—C14	3.1 (3)

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

D—H···A	D—H	H···A	D···A	D—H···A
O5—H5···O1^{i}	0.82	1.79	2.597 (3)	166
N1—H1···O2^{ii}	0.86	2.33	3.142 (3)	157
N1—H1···O3	0.86	2.40	2.844 (3)	113

Symmetry codes: (i) $-x+y+1, -x+1, z+\frac{1}{3}$; (ii) $-y+1, x-y+1, z-\frac{1}{3}$.

The H atom of the OH group was initially located in a difference Fourier map, but subsequently the O—H distance was constrained to 0.82 Å and the U_{iso}(H) value set equal to 1.2U_{eq}(O). All other H atoms were positioned geometrically (N—H = 0.86, C—H = 0.93–0.98 Å) and refined as riding, with U_{iso}(H) = 1.2 or 1.5 times U_{eq}(parent atom).

Data collection: *SMART* (Bruker, 1997); cell refinement: *SAINT* (Bruker, 1997); data reduction: *SAINT*; program(s) used to solve structure: *SHELXS97* (Sheldrick, 1997); program(s) used to refine structure: *SHELXL97* (Sheldrick, 1997); molecular graphics: *SHELXTL* (Bruker, 1997); software used to prepare material for publication: *SHELXTL*.

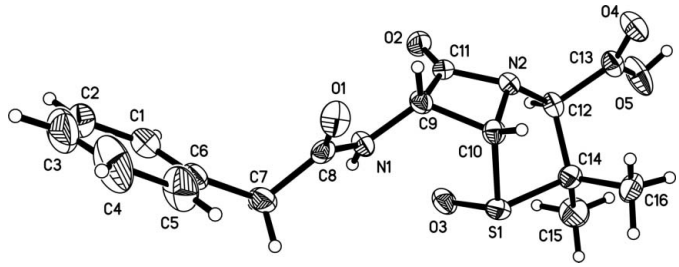


Figure 1
The molecular structure of (I), showing the atom-numbering scheme and 30% probability displacement ellipsoids.

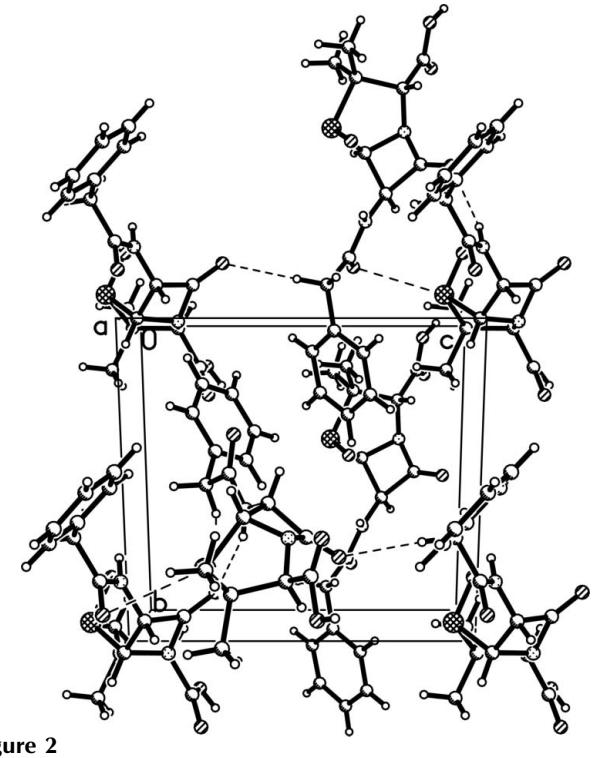


Figure 2
Packing diagram for (I), with hydrogen bonds shown as dashed lines.

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