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A monoclinic polymorph of pyrazinic acid

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Key indicators

Single-crystal X-ray study
T = 294 K
Mean σ(C–C) = 0.005 Å
R factor = 0.037
wR factor = 0.079
Data-to-parameter ratio = 6.9

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

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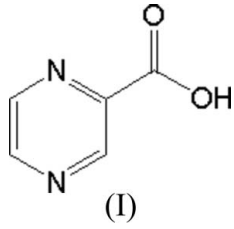
The title compound (pyrazine-2-carboxylic acid), $C_5H_4N_2O_2$, had been previously characterized in space group $Pna2_1$ and has now been obtained as a monoclinic polymorph crystallizing in space group $P2_1$. The molecule is almost planar and is connected to symmetry-related molecules through O—H···N and C—H···O hydrogen bonds, and weak π–π interactions, giving a three-dimensional network.

Comment

Pyrazinic acid (pyrazine-2-carboxylic acid) is one of the most important materials for the preparation of pyrazine derivatives. Some pyrazine derivatives possess bacteriostatic activity. They are widely used in the treatment of tuberculosis and also exhibit fungicidal activity (Kushner *et al.*, 1952). We have designed and synthesized a number of compounds for testing their effective antibacterial activity compared with that of pyrazinic acid. When recrystallizing a commercial impure batch of the title compound, (I), we obtained single crystals of a new monoclinic polymorph for this compound.



(I)

The title compound, $C_5H_4N_2O_2$, crystallizes in space group $P2_1$ with the expected geometry (Table 1). The molecule is almost planar. A weak intramolecular hydrogen bond involves the C3/H3 and hydroxy groups (Fig. 1). Atom N2 of the pyrazine ring is connected to atom H1 of the hydroxy group, while atom H3 is linked to atom O2 of the carbonyl group

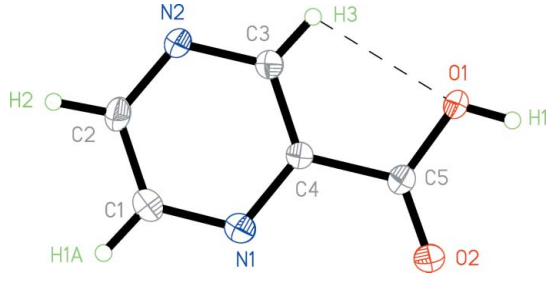


Figure 1
The molecular structure of (I), showing 30% probability displacement ellipsoids. H atoms are drawn as small spheres of arbitrary radii. The dashed line indicates the intramolecular hydrogen bond.

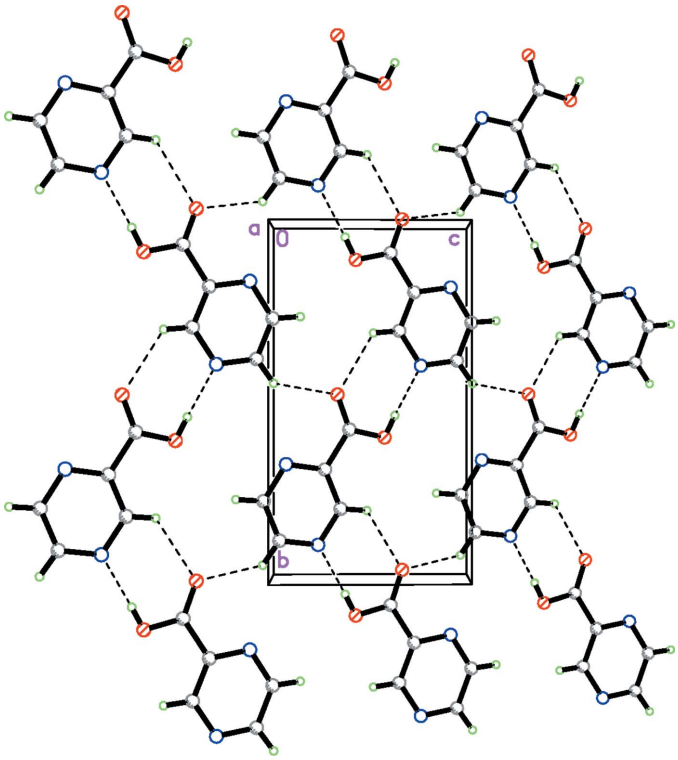


Figure 2
The one-dimensional zigzag chains and two-dimensional structure of compound (I), formed by O—H···N and C—H···O hydrogen bonds (dashed lines) between adjacent molecules.

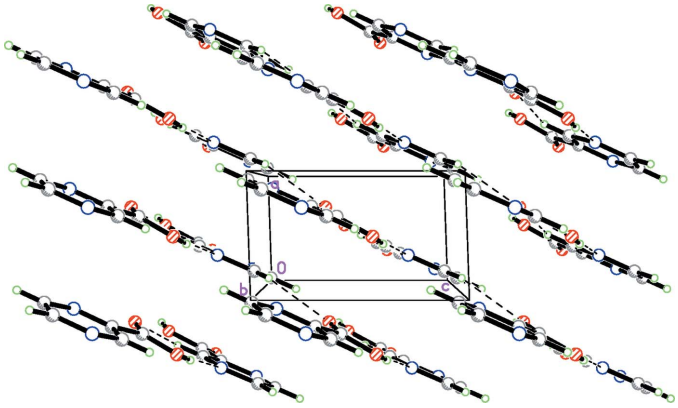


Figure 3
The packing of (I) viewed along the *b* axis, showing the layered structure. Dashed lines indicate hydrogen bonds.

(Table 2). As a result, molecules form zigzag chains along the [010] axis. Between two neighbouring zigzag chains, carboxylic O2 atoms interact with atoms H2 of the pyrazine rings to form C—H···O hydrogen bonds (Table 2). These hydrogen bonds link the one-dimensional zigzag chains, forming a quasi-two-dimensional network, as illustrated in Fig. 2.

In addition, face-to-face π–π stacking interactions are observed in the crystal, with a separation of *ca* 3.36 Å between the centroids of the pyrazine rings (Fig. 3). The complete crystal structure presents a different topology from that of the previously reported orthorhombic polymorph (Tukusagawa *et al.*, 1974).

Experimental

The title compound was obtained by recrystallization of an impure industrial batch of this compound. The crystal used for data collection was obtained by slow evaporation at 298 K of a methanol solution, over a period of one week.

Crystal data

$C_5H_4N_2O_2$
M_r = 124.10
Monoclinic, $P2_1$
a = 3.7249 (14) Å
b = 11.281 (4) Å
c = 6.298 (2) Å
β = 91.936 (7)°
V = 264.48 (18) Å^3
Z = 2
D_x = 1.558 Mg m^{-3}
Mo $K\alpha$ radiation
Cell parameters from 477 reflections
θ = 3.5–23.7°
μ = 0.12 mm^{-1}
T = 294 (2) K
Block, colourless
0.30 × 0.16 × 0.10 mm

Data collection

Bruker SMART CCD area-detector diffractometer
φ and ω scans
Absorption correction: multi-scan (*SADABS*; Sheldrick, 1996)
T_{min} = 0.962, T_{max} = 0.988
1501 measured reflections
571 independent reflections
440 reflections with $I > 2\sigma(I)$
R_{int} = 0.037
θ_{max} = 26.5°
$h = -4 \rightarrow 4$
$k = -14 \rightarrow 6$
$l = -7 \rightarrow 7$

Refinement

Refinement on F^2
$R[F^2 > 2\sigma(F^2)]$ = 0.037
$wR(F^2)$ = 0.079
S = 1.07
571 reflections
83 parameters
H-atom parameters constrained
$w = 1/[\sigma^2(F_o^2) + (0.032P)^2 + 0.0353P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max}$ = 0.002
$\Delta\rho_{max}$ = 0.14 e Å^{-3}
$\Delta\rho_{min}$ = −0.18 e Å^{-3}

Table 1
Selected geometric parameters (Å, °).

O1—C5	1.324 (4)	N1—C1	1.354 (5)
O2—C5	1.201 (4)	N2—C2	1.329 (4)
N1—C4	1.346 (4)	N2—C3	1.346 (4)
C4—N1—C1	115.2 (3)	C2—N2—C3	116.9 (3)

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

D—H···A	D—H	H···A	D···A	D—H···A
O1—H1···N2^{i}	0.82	1.86	2.676 (3)	175
C2—H2···O2^{ii}	0.93	2.46	3.168 (4)	133
C3—H3···O1	0.93	2.40	2.725 (4)	101
C3—H3···O2^{iii}	0.93	2.47	3.152 (4)	131

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

C bound H atoms were placed in calculated positions and allowed to ride on their parent atoms, with C—H distances constrained to 0.93 Å and U_{iso}(H) = 1.2U_{eq}(parent C atom). The H atom of the hydroxy group was found in a difference map, but was constrained with O—H = 0.82 Å and U_{iso}(H) = 1.5U_{eq}(O). In the absence of significant anomalous scattering effects, measured Friedel pairs were merged.

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*.

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