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A monoclinic polymorph of hypoxanthine

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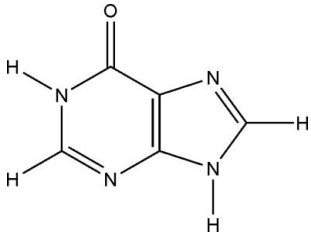
Key indicators: single-crystal X-ray study; T = 292 K; mean σ(C–C) = 0.002 Å; R factor = 0.036; wR factor = 0.109; data-to-parameter ratio = 12.1.

A monoclinic polymorph of hypoxanthine (systematic name: 1,7-dihydro-6*H*-purin-6-one), $C_5H_4N_4O$, is reported. The hydrogen-bonding motifs involve a layered structure of N—H···O and N—H···N hydrogen-bonded molecules. The mean stacking distance between adjacent layers is 3.672 Å.

Related literature

The triclinic polymorph of hypoxanthine was described by Schmalle *et al.* (1988). There are weak intermolecular C—H···N and C—H···O hydrogen bonds (Taylor & Kennard, 1982).



Experimental

Crystal data

$C_5H_4N_4O$
M_r = 136.12
Monoclinic, $P2_1/c$
a = 3.6725 (19) Å
b = 17.960 (9) Å
c = 9.010 (5) Å
β = 107.469 (19)°
V = 566.9 (5) Å^3
Z = 4
Mo $K\alpha$ radiation
μ = 0.12 mm^{-1}
T = 292 (2) K
0.48 × 0.13 × 0.11 mm

Data collection

Bruker SMART APEXII CCD area-detector diffractometer
Absorption correction: multi-scan (*SADABS*; Sheldrick, 1996)
T_{min} = 0.979, T_{max} = 0.988
2981 measured reflections
1113 independent reflections
964 reflections with $I > 2\sigma(I)$
R_{int} = 0.016

Refinement

$R[F^2 > 2\sigma(F^2)]$ = 0.036
$wR(F^2)$ = 0.109
S = 1.04
1113 reflections
92 parameters
H-atom parameters constrained
$\Delta\rho_{max}$ = 0.19 e Å^{-3}
$\Delta\rho_{min}$ = −0.19 e Å^{-3}

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

D—H···A	D—H	H···A	D···A	D—H···A
N1—H1···O1^{i}	0.88	1.91	2.7846 (18)	172
N4—H4···N3^{ii}	0.88	1.95	2.8208 (19)	168
C2—H2···N2^{iii}	0.95	2.60	3.376 (2)	139
C5—H5···O1^{ii}	0.95	2.48	3.1933 (19)	132

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

Data collection: *APRX2* (Bruker, 2004); cell refinement: *SAINT* (Bruker, 2004); 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, 2004); software used to prepare material for publication: *SHELXTL*.

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Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: SG2177).

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Schmalle, H. W., Hänggi, G. & Dubler, E. (1988). *Acta Cryst.* C**44**, 732–736.
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Sheldrick, G. M. (1997). *SHELXS97* and *SHELXL97*. University of Göttingen, Germany.
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supplementary materials

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A monoclinic polymorph of hypoxanthine

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Comment

A triclinic polymorph of hypoxanthine (I) has been reported (Schmalle *et al.*, 1988). Here we describe a monoclinic polymorph (Figure1). There are two hydrogen bonds of the type N1—H1···O1^{i} with N1—O1^{i} donor-acceptor distance of 2.7846 (18) Å and N4—H4···N3^{ii} with N4—N3^{ii} donor-acceptor distance of 2.8208 (19) Å (Figure 2). In addition, there are different weak intermolecular contacts (Taylor & Kennard,1982) of the form C2—H2···N2^{iii} with C2—N2^{iii} distance of 3.376 (2) Å and C5—H5···O1^{ii} with C5—O1^{ii} distance of 3.1933 (19) Å (Table 1). Thus, the hypoxanthine molecules form sheets approximately parallel to the (10$\bar{2}$) plane. The separations between parallel hypoxanthine molecules stacked along the *a* axis is 3.672 Å (Figure 3).

Experimental

Hydrothermal treatment of $Ba(ClO_4)_2{\cdot}6H_2O$(0.1332 g, 0.3 mmol), Inosine(0.1610 g, 0.6 mmol), And 95% ethanol solution(4 ml) over three days at 70 ^{o}C yielded colorless plate crystals of hypoxanthine(I). The product was isolated, washed three times with 70% ethanol solution, and dried in a vacuum desiccator using $CaCl_2$. Yield: 15%. CH&N analysis: Calculated for $C_5H_4N_4O$: C 44.12, H 2.96, N 41.16%; found: C 44.05, H 2.93, N 41.04%.

Refinement

H atoms bonded to C or N atoms were placed in calculated positions, with C—H = 0.95 Å and N—H= 0.88 Å, and were included in the refinement in the riding-model approximation, and U_{iso}(H) = 1.2U_{eq}(C or N).

Figures

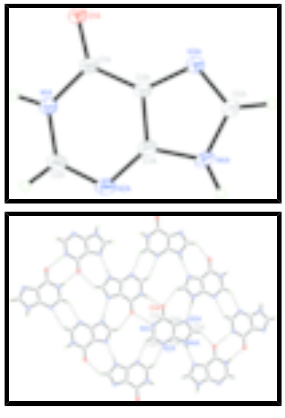

Figure 1 The asymmetric unit of compound (I), with displacement ellipsoids drawn at the 30% probability level. Figure 2 The hydrogen-bonding motif in (I). Dashed lines indicate the hydrogen bonds. Figure 3. The packing of (I) in the crystal.

1,7-dihydro-6*H*-purin-6-one

Crystal data

$C_5H_4N_4O$	$Z = 4$
$M_r = 136.12$	$F_{000} = 280$
Monoclinic, $P2_1/c$	$D_x = 1.595$ Mg m^{-3}
$a = 3.6725$ (19) Å	Mo $K\alpha$ radiation $\lambda = 0.71073$ Å
$b = 17.960$ (9) Å	$\mu = 0.12$ mm^{-1}
$c = 9.010$ (5) Å	$T = 292$ (2) K
$\beta = 107.469$ (19)º	Plate, colorless
$V = 566.9$ (5) Å^3	0.48 × 0.13 × 0.11 mm

Data collection

Bruker SMART APEXII CCD area-detector diffractometer	1113 independent reflections
Radiation source: fine-focus sealed tube	964 reflections with $I > 2\sigma(I)$
Monochromator: graphite	$R_{int} = 0.016$
$T = 292(2)$ K	$\theta_{max} = 26.0$º
φ and ω scans	$\theta_{min} = 2.3$º
Absorption correction: multi-scan (SADABS; Sheldrick, 1996)	$h = -4\rightarrow4$
$T_{min} = 0.979$, $T_{max} = 0.988$	$k = -22\rightarrow21$
2981 measured reflections	$l = -6\rightarrow11$

Refinement

Refinement on F^2	Hydrogen site location: inferred from neighbouring sites
Least-squares matrix: full	H-atom parameters constrained
$R[F^2 > 2\sigma(F^2)] = 0.036$	$w = 1/[\sigma^2(F_o^2) + (0.0758P)^2 + 0.069P]$ where $P = (F_o^2 + 2F_c^2)/3$
$wR(F^2) = 0.109$	$(\Delta/\sigma)_{max} < 0.001$
$S = 1.04$	$\Delta\rho_{max} = 0.19$ e Å^{-3}
1113 reflections	$\Delta\rho_{min} = -0.19$ e Å^{-3}
92 parameters	Extinction correction: SHELXL97, $Fc^*=kFc[1+0.001xFc^2\lambda^3/\sin(2\theta)]^{-1/4}$
Primary atom site location: structure-invariant direct methods	Extinction coefficient: 0.067 (11)
Secondary atom site location: difference Fourier map	

Special details

Geometry. All e.s.d.'s (except the e.s.d. in the dihedral angle between two l.s. planes) are estimated using the full covariance matrix. The cell e.s.d.'s are taken into account individually in the estimation of e.s.d.'s in distances, angles and torsion angles; correlations between e.s.d.'s in cell parameters are only used when they are defined by crystal symmetry. An approximate (isotropic) treatment of cell e.s.d.'s is used for estimating e.s.d.'s involving l.s. planes.

Refinement. Refinement of F^2 against ALL reflections. The weighted *R*-factor *wR* and goodness of fit S are based on F^2, conventional *R*-factors *R* are based on F, with F set to zero for negative F^2. The threshold expression of $F^2 > 2\mathrm{sigma}(F^2)$ is used only for calculating *R*-factors(gt) *etc*. and is not relevant to the choice of reflections for refinement. *R*-factors based on F^2 are statistically about twice as large as those based on F, and R– factors based on ALL data will be even larger.

Fractional atomic coordinates and isotropic or equivalent isotropic displacement parameters (Å^2)

	x	*y*	*z*	U_{iso}*/U_{eq}
O1	0.5544 (3)	0.40375 (5)	0.51583 (12)	0.0469 (3)
N1	0.1515 (3)	0.46886 (6)	0.31417 (13)	0.0374 (3)
H1	0.2213	0.5107	0.3658	0.045*
N2	−0.2451 (3)	0.41739 (6)	0.08071 (13)	0.0388 (3)
N3	0.2692 (3)	0.26683 (6)	0.31153 (13)	0.0367 (3)
N4	−0.1664 (3)	0.28307 (6)	0.08049 (13)	0.0377 (3)
H4	−0.3275	0.2721	−0.0109	0.045*
C1	0.3174 (4)	0.40330 (7)	0.38534 (16)	0.0339 (3)
C2	−0.1094 (4)	0.47339 (8)	0.17154 (16)	0.0395 (4)
H2	−0.2006	0.5216	0.1350	0.047*
C3	−0.0906 (4)	0.35170 (7)	0.14506 (15)	0.0329 (3)
C4	0.1784 (3)	0.34128 (7)	0.28815 (14)	0.0317 (3)
C5	0.0550 (4)	0.23473 (8)	0.18446 (16)	0.0394 (4)
H5	0.0552	0.1826	0.1670	0.047*

Atomic displacement parameters (Å^2)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
O1	0.0569 (7)	0.0343 (6)	0.0312 (6)	−0.0004 (4)	−0.0145 (5)	−0.0024 (4)
N1	0.0449 (7)	0.0273 (6)	0.0308 (7)	0.0004 (4)	−0.0026 (5)	0.0000 (4)
N2	0.0406 (6)	0.0380 (7)	0.0284 (6)	0.0014 (5)	−0.0038 (5)	0.0046 (5)
N3	0.0422 (6)	0.0294 (6)	0.0289 (6)	0.0001 (4)	−0.0039 (5)	0.0000 (4)
N4	0.0407 (6)	0.0380 (7)	0.0233 (6)	−0.0042 (4)	−0.0075 (5)	−0.0041 (4)
C1	0.0358 (6)	0.0322 (7)	0.0261 (7)	−0.0012 (5)	−0.0023 (5)	0.0016 (5)
C2	0.0429 (7)	0.0355 (7)	0.0318 (8)	0.0036 (5)	−0.0013 (6)	0.0062 (5)
C3	0.0340 (6)	0.0348 (7)	0.0244 (7)	−0.0014 (5)	0.0004 (5)	0.0012 (5)
C4	0.0338 (7)	0.0299 (7)	0.0247 (7)	−0.0005 (5)	−0.0014 (5)	0.0018 (5)
C5	0.0446 (8)	0.0320 (7)	0.0317 (8)	−0.0016 (5)	−0.0035 (6)	−0.0029 (5)

Geometric parameters (Å, °)

O1—C1	1.2352 (17)	N4—C5	1.3546 (17)
N1—C2	1.3553 (18)	N4—C3	1.3557 (17)

N1—C1	1.3908 (17)	N4—H4	0.8800
N1—H1	0.8800	C1—C4	1.4141 (18)
N2—C2	1.2980 (18)	C2—H2	0.9500
N2—C3	1.3612 (17)	C3—C4	1.3815 (19)
N3—C5	1.3118 (17)	C5—H5	0.9500
N3—C4	1.3788 (18)		
C2—N1—C1	125.10 (11)	N2—C2—H2	117.3
C2—N1—H1	117.4	N1—C2—H2	117.3
C1—N1—H1	117.4	N4—C3—N2	126.84 (12)
C2—N2—C3	111.78 (12)	N4—C3—C4	105.93 (11)
C5—N3—C4	103.81 (11)	N2—C3—C4	127.22 (12)
C5—N4—C3	106.41 (11)	N3—C4—C3	110.26 (11)
C5—N4—H4	126.8	N3—C4—C1	130.06 (12)
C3—N4—H4	126.8	C3—C4—C1	119.69 (12)
O1—C1—N1	121.29 (11)	N3—C5—N4	113.58 (13)
O1—C1—C4	127.93 (11)	N3—C5—H5	123.2
N1—C1—C4	110.78 (12)	N4—C5—H5	123.2
N2—C2—N1	125.43 (13)		

Hydrogen-bond geometry (Å, °)

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
N1—H1···O1^{i}	0.88	1.91	2.7846 (18)	172
N4—H4···N3^{ii}	0.88	1.95	2.8208 (19)	168
C2—H2···N2^{iii}	0.95	2.60	3.376 (2)	139
C5—H5···O1^{ii}	0.95	2.48	3.1933 (19)	132

Symmetry codes: (i) −*x*+1, −*y*+1, −*z*+1; (ii) *x*−1, −*y*+1/2, *z*−1/2; (iii) −*x*−1, −*y*+1, −*z*.

Fig. 1

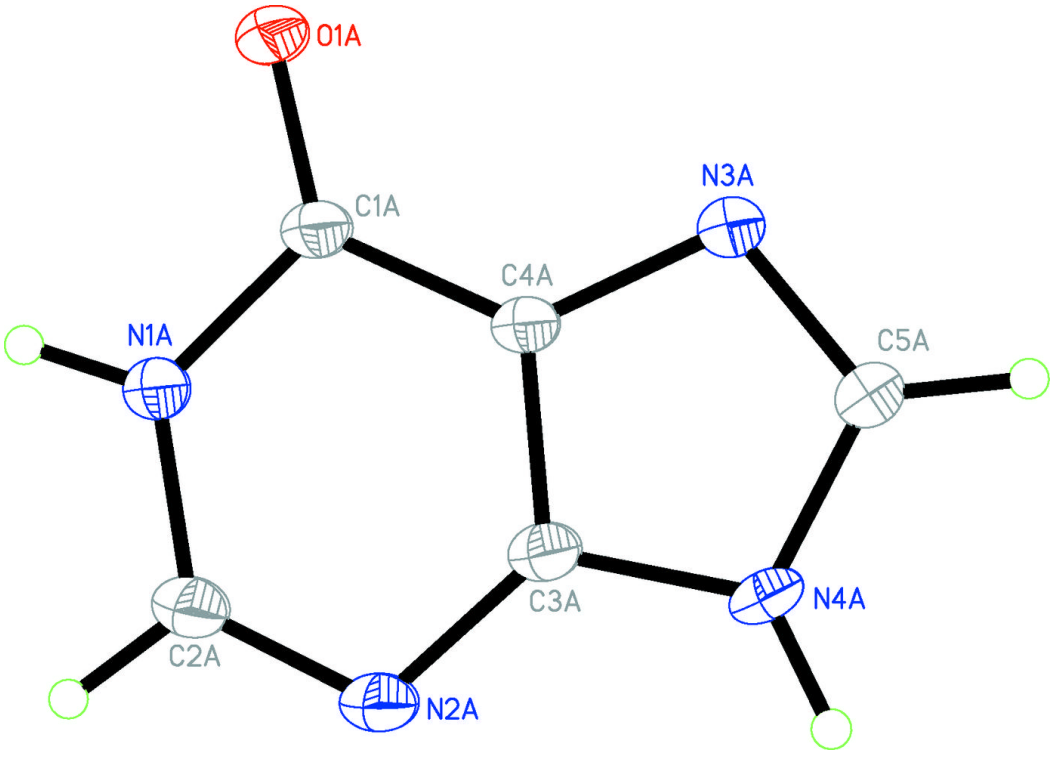

Fig. 2

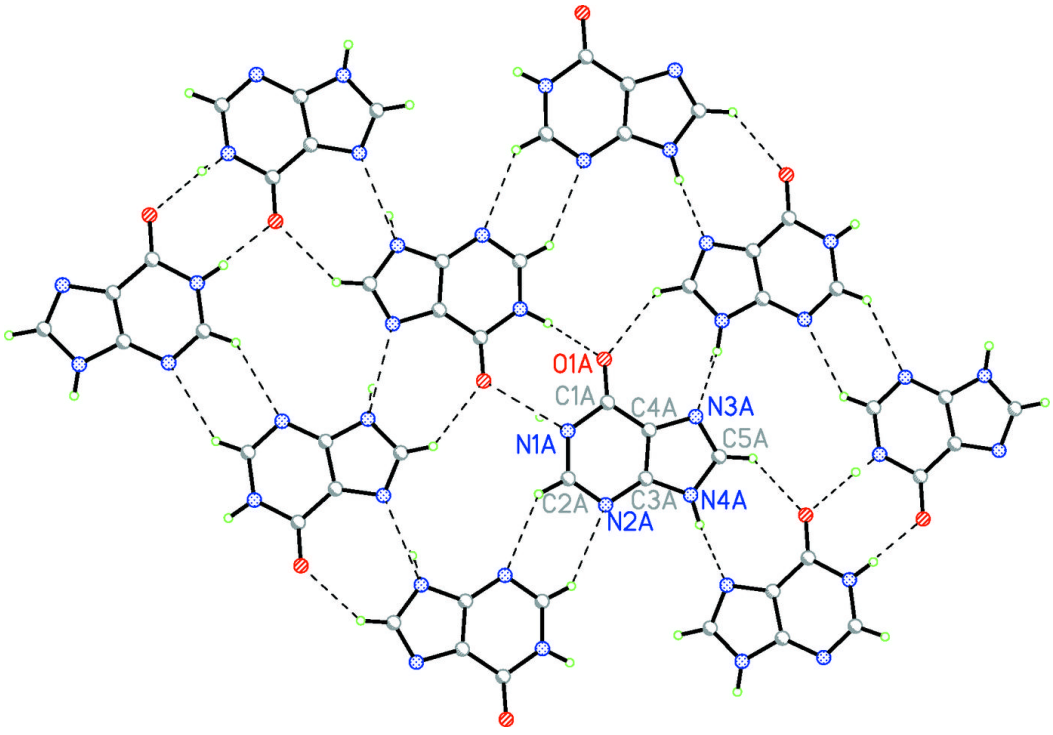

Fig. 3