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Low-temperature redetermination of 3,4,5,6-tetrahydropyrimidin-2(1*H*)-one

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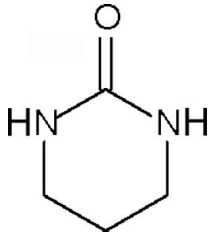
Key indicators: single-crystal X-ray study; T = 100 K; mean σ(C–C) = 0.001 Å; R factor = 0.037; wR factor = 0.111; data-to-parameter ratio = 13.6.

The low-temperature structure of the title compound, $C_4H_8N_2O$, is ordered, whereas the central methylene groups is disordered in the reported room-temperature structure. The molecule lies across a mirror plane; adjacent molecules are linked by an N—H···O hydrogen bond into a chain.

Related literature

For the room-temperature, disordered structure of tetrahydropyrimidin-2(1*H*)-one, see: Calogero *et al.* (1980).



Experimental

Crystal data

$C_4H_8N_2O$
M_r = 100.12
Orthorhombic, *Pnma*
a = 9.9958 (1) Å
b = 7.1327 (1) Å
c = 6.7365 (1) Å
V = 480.29 (1) Å^3
Z = 4
Mo $K\alpha$ radiation
μ = 0.10 mm^{-1}
T = 100 (2) K
0.35 × 0.20 × 0.15 mm

Data collection

Bruker SMART APEX diffractometer
Absorption correction: none
6595 measured reflections
749 independent reflections
719 reflections with $I > 2\sigma(I)$
R_{int} = 0.020

Refinement

$R[F^2 > 2\sigma(F^2)]$ = 0.036
$wR(F^2)$ = 0.110
S = 1.06
749 reflections
55 parameters
All H-atom parameters refined
$\Delta\rho_{max}$ = 0.49 e Å^{-3}
$\Delta\rho_{min}$ = −0.21 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}	0.89 (1)	1.97 (1)	2.864 (1)	178 (1)

Symmetry code: (i) $-x+1, -y+1, -z+1$.

Data collection: *APEX2* (Bruker, 2007); cell refinement: *SAINT* (Bruker, 2007); data reduction: *SAINT*; program(s) used to solve structure: *SHELXS97* (Sheldrick, 2008); program(s) used to refine structure: *SHELXL97* (Sheldrick, 2008); molecular graphics: *X-SEED* (Barbour, 2001); software used to prepare material for publication: *publCIF* (Westrip, 2008).

We thank the University of Malaya for the purchase of the diffractometer.

Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: BV2094).

References

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Calogero, S., Russo, U. & Del Pra, A. (1980). *J. Chem. Soc. Dalton Trans.* pp. 646–653.
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supporting information

Acta Cryst. (2008). E**64**, o914 [doi:10.1107/S160053680801115X]

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S1. Comment

The crystal structure of tetrahydropyrimidin-2(1*H*)-one (Scheme I) was refined as a disorder model, the second of the three methylene carbon atoms being disordered about a mirror plane by a ratio of 0.7:0.3 (Calogero *et al.*, 1980). However, the structure is ordered at low temperature (Fig. 1); adjacent molecules are linked by a N–H···O hydrogen bond into a chain motif.

S2. Experimental

The commercially available compound was crystalline. A large block was cut into a smaller specimen.

S3. Refinement

All hydrogen atoms were located in a difference Fourier map, and were freely refined.

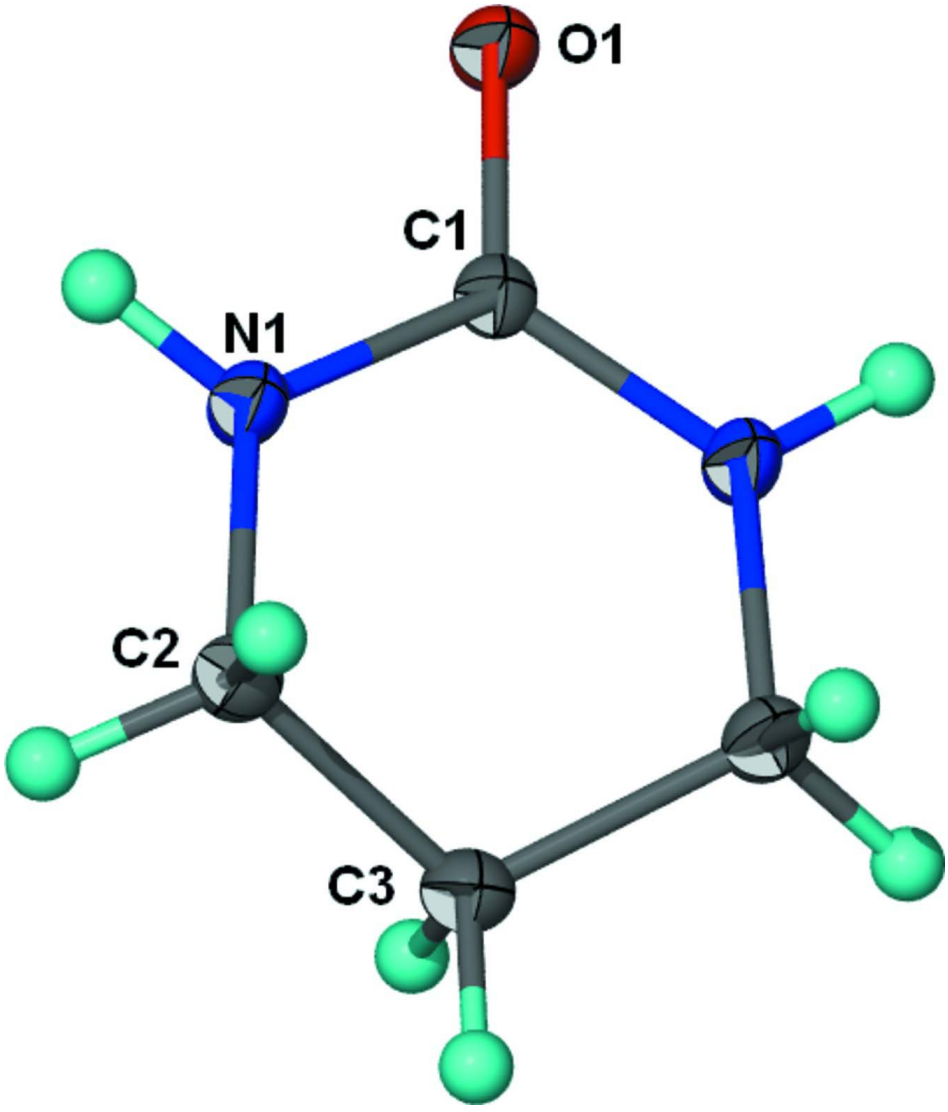

Figure 1

Thermal ellipsoid plot of tetrahydropyrimidin-2(1*H*)-one at the 70% probability level; hydrogen atoms are drawn as spheres of arbitrary radius.

3,4,5,6-tetrahydropyrimidin-2(1*H*)-one

Crystal data

$C_4H_8N_2O$
$M_r = 100.12$
Orthorhombic, *Pnma*
Hall symbol: -P 2ac 2n
$a = 9.9958$ (1) Å
$b = 7.1327$ (1) Å
$c = 6.7365$ (1) Å
$V = 480.29$ (1) Å^3
$Z = 4$
$F(000) = 216$
$D_x = 1.385$ Mg m^{-3}
Mo $K\alpha$ radiation, $\lambda = 0.71073$ Å
Cell parameters from 4972 reflections
$\theta = 3.0$–$30.4°$
$\mu = 0.10$ mm^{-1}
$T = 100$ K
Block, colorless
0.35 × 0.20 × 0.15 mm

Data collection

Bruker SMART APEXII diffractometer
Radiation source: fine-focus sealed tube
Graphite monochromator
φ and ω scans
6595 measured reflections
749 independent reflections
719 reflections with $I > 2\sigma(I)$
$R_{int} = 0.020$
$\theta_{max} = 30.0°$, $\theta_{min} = 3.7°$
$h = -12 \rightarrow 13$
$k = -9 \rightarrow 10$
$l = -9 \rightarrow 9$

Refinement

Refinement on F^2
Least-squares matrix: full
$R[F^2 > 2\sigma(F^2)] = 0.036$
$wR(F^2) = 0.110$
$S = 1.06$
749 reflections
55 parameters
0 restraints
Primary atom site location: structure-invariant direct methods
Secondary atom site location: difference Fourier map
Hydrogen site location: inferred from neighbouring sites
All H-atom parameters refined
$w = 1/[\sigma^2(F_o^2) + (0.0818P)^2 + 0.084P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} = 0.001$
$\Delta\rho_{max} = 0.49$ e Å^{-3}
$\Delta\rho_{min} = -0.22$ e Å^{-3}

Fractional atomic coordinates and isotropic or equivalent isotropic displacement parameters (Å²)

	x	*y*	*z*	U_{iso}*/U_{eq}
O1	0.48291 (8)	0.7500	0.51927 (11)	0.0143 (2)
N1	0.58480 (7)	0.58713 (8)	0.27287 (9)	0.0140 (2)
C1	0.54996 (10)	0.7500	0.36064 (14)	0.0113 (2)
C2	0.67121 (7)	0.57605 (10)	0.09858 (11)	0.0145 (2)
C3	0.65258 (11)	0.7500	−0.02874 (14)	0.0146 (3)
H1	0.5642 (13)	0.4836 (18)	0.3405 (19)	0.023 (3)*
H21	0.6483 (11)	0.4643 (17)	0.0232 (17)	0.017 (3)*
H22	0.7668 (11)	0.5621 (17)	0.1397 (17)	0.017 (2)*
H31	0.5609 (18)	0.7500	−0.089 (3)	0.023 (4)*
H32	0.7163 (18)	0.7500	−0.140 (3)	0.023 (4)*

Atomic displacement parameters (Å²)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
O1	0.0176 (4)	0.0122 (4)	0.0132 (4)	0.000	0.0045 (3)	0.000
N1	0.0188 (4)	0.0099 (3)	0.0133 (3)	0.0003 (2)	0.0047 (2)	0.00012 (19)
C1	0.0109 (4)	0.0111 (5)	0.0118 (4)	0.000	−0.0008 (3)	0.000
C2	0.0173 (4)	0.0128 (4)	0.0133 (4)	0.0010 (2)	0.0039 (2)	−0.0008 (2)
C3	0.0177 (5)	0.0145 (5)	0.0117 (4)	0.000	0.0013 (3)	0.000

Geometric parameters (Å, º)

O1—C1	1.2614 (12)	C2—H21	0.972 (12)
N1—C1	1.3492 (8)	C2—H22	1.000 (11)
N1—C2	1.4597 (9)	C3—C2^{i}	1.5198 (9)
N1—H1	0.892 (13)	C3—H31	1.001 (18)

C1—N1^{i}	1.3492 (8)	C3—H32	0.982 (19)
C2—C3	1.5198 (9)		
C1—N1—C2	123.49 (6)	N1—C2—H22	110.4 (7)
C1—N1—H1	115.4 (8)	C3—C2—H22	110.8 (7)
C2—N1—H1	120.2 (8)	H21—C2—H22	106.8 (10)
O1—C1—N1	120.56 (4)	C2^{i}—C3—C2	109.45 (8)
O1—C1—N1^{i}	120.56 (4)	C2^{i}—C3—H31	109.9 (5)
N1—C1—N1^{i}	118.86 (9)	C2—C3—H31	109.9 (5)
N1—C2—C3	109.71 (6)	C2^{i}—C3—H32	110.5 (5)
N1—C2—H21	109.0 (7)	C2—C3—H32	110.5 (5)
C3—C2—H21	110.2 (7)	H31—C3—H32	106.7 (15)
C2—N1—C1—O1	−174.85 (8)	C1—N1—C2—C3	−30.95 (10)
C2—N1—C1—N1^{i}	7.00 (14)	N1—C2—C3—C2^{i}	52.71 (10)

Symmetry code: (i) x, $-y+3/2$, z.

Hydrogen-bond geometry (Å, º)

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
N1—H1···O1^{ii}	0.89 (1)	1.97 (1)	2.864 (1)	178 (1)

Symmetry code: (ii) $-x+1$, $-y+1$, $-z+1$.