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1-Methyl-1,3-diazinan-2-one

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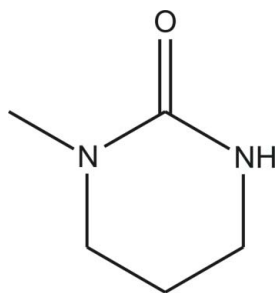
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Key indicators: single-crystal X-ray study; $T = 100$ K; mean $\sigma(\text{C}-\text{C}) = 0.002$ Å; R factor = 0.037; wR factor = 0.092; data-to-parameter ratio = 18.2.

In the crystal structure of the title compound, $\text{C}_5\text{H}_{10}\text{N}_2\text{O}$, molecules are connected *via* pairs of strong $\text{N}-\text{H}\cdots\text{O}$ hydrogen bonds into centrosymmetric dimers, which are stacked along the a axis. The molecule is not planar, the dihedral angle between the $\text{N}/\text{C}/\text{N}$ and $\text{C}/\text{C}/\text{C}$ planes being $42.1(1)^\circ$.

Related literature

For substitution of hexamethylphosphoramide (HMPT) by the cyclic urea 1,3-dimethyl-3,4,5,6-tetrahydropyrimidin-2-one (DMPU), see: Mukhopadhyay & Seebach (1982). For the crystal structure of 3,4,5,6-tetrahydropyrimidin-2-one, see: Rizal *et al.* (2008) and of 1-methyl-imidazolidin-2-one, see: Caudle *et al.* (2005).



Experimental

Crystal data

 $\text{C}_5\text{H}_{10}\text{N}_2\text{O}$ $M_r = 114.15$ Orthorhombic, $Pbca$

$a = 5.8479$ (2) Å
 $b = 13.3438$ (6) Å
 $c = 15.0883$ (8) Å
 $V = 1177.39$ (9) Å³

 $Z = 8$ Mo $K\alpha$ radiation $\mu = 0.09$ mm⁻¹ $T = 100$ K $0.19 \times 0.15 \times 0.11$ mm

Data collection

Bruker–Nonius KappaCCD diffractometer
2628 measured reflections

1434 independent reflections
1190 reflections with $I > 2\sigma(I)$
 $R_{\text{int}} = 0.024$

Refinement

 $R[F^2 > 2\sigma(F^2)] = 0.037$ $wR(F^2) = 0.092$ $S = 1.03$

1434 reflections

79 parameters

H atoms treated by a mixture of independent and constrained refinement

 $\Delta\rho_{\text{max}} = 0.27$ e Å⁻³ $\Delta\rho_{\text{min}} = -0.18$ e Å⁻³

Table 1

Hydrogen-bond geometry (Å, °).

$D-\text{H}\cdots A$	$D-\text{H}$	$\text{H}\cdots A$	$D\cdots A$	$D-\text{H}\cdots A$
$\text{N1}-\text{H1}\cdots\text{O1}^i$	0.88 (2)	2.00 (2)	2.875 (1)	177 (2)

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

Data collection: *COLLECT* (Hooft, 2004); cell refinement: *SCALEPACK* (Otwinowski & Minor, 1997); data reduction: *SCALEPACK*; program(s) used to solve structure: *SHELXS97* (Sheldrick, 2008); program(s) used to refine structure: *SHELXL97* (Sheldrick, 2008); molecular graphics: *DIAMOND* (Brandenburg & Putz, 2005); software used to prepare material for publication: *SHELXL97*.

The authors thank Dr Falk Lissner (Institut für Anorganische Chemie, Universität Stuttgart) for measuring the crystal data.

Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: FK2059).

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1-Methyl-1,3-diazinan-2-one

Ioannis Tiritiris and Willi Kantlehner

S1. Comment

1,3-Dimethyl-3,4,5,6-tetrahydropyrimidin-2-one (DMPU), a liquid at room temperature, is often used in organic synthesis as a polar aprotic solvent, replacing the carcinogenic hexamethylphosphoramide (HMPT) (Mukhopadhyay & Seebach, 1982). In contrast, 3,4,5,6-tetrahydropyrimidin-2-one is a solid with a melting point of 263–267 °C and its ordered crystal structure was quite recently determined (Rizal *et al.*, 2008). The crystal structure of the missing link 1-methyl-3,4,5,6-tetrahydropyrimidin-2-one (I) was previously unknown. Prominent bond parameters for the title molecule are: C1–O1 = 1.248 (1) Å, N1–C1 = 1.357 (1) Å and N2–C1 = 1.362 (1) Å. The bond length between N2 and the terminal C-methyl group (C5) measures 1.453 (1) Å. The C–N2–C angles are: 123.54 (9)° (C4–N2–C1), 120.32 (8)° (C1–N2–C5) and 115.18 (9)° (C5–N2–C4), which indicates a trigonal-planar surrounding of the nitrogen centre by the C atoms. These data are in good agreement with those of the five membered heterocycle 1-methyl-imidazolidin-2-one (Caudle *et al.*, 2005). In contrast to the aforementioned compound, the six membered heterocycle in (I) is non-planar (Fig. 1). The carbon atom C3 is not in the ring plane, the angle between the planes N1/C1/N2 and C2/C3/C4 is 42.1 (1)°. In the packing, each two molecules are linked by strong N–H···O hydrogen bonds, forming centrosymmetric dimers, which are stacked along the *a* axis (Fig.2). The H···O distance is 2.00 (2) Å, with a nearly linear N–H···O angle of 177 (2)° (Tab.1).

S2. Experimental

The title compound was obtained as a byproduct by reaction of 1-Methyl-2-dimethylamino-1,4,5,6-tetrahydropyrimidinium-chloride with excess aqueous sodium hydroxide at room temperature. After distillation of the crude product *in vacuo*, a colourless liquid was obtained. The compound crystallized spontaneously upon standing at room temperature after several days, forming colourless single crystals.

S3. Refinement

The N-bound H atom was located in a difference Fourier map and was refined freely. The hydrogen atoms of the methyl group were allowed to rotate with a fixed angle around the C–N bond to best fit the experimental electron density, with $U(\text{H})$ set to 1.5 $U_{\text{eq}}(\text{C})$ and $d(\text{C}—\text{H}) = 0.98$ Å. The remaining H atoms were placed in calculated positions with $d(\text{C}—\text{H}) = 0.99$ Å and were included in the refinement in the riding model approximation, with $U(\text{H})$ set to 1.2 $U_{\text{eq}}(\text{C})$.

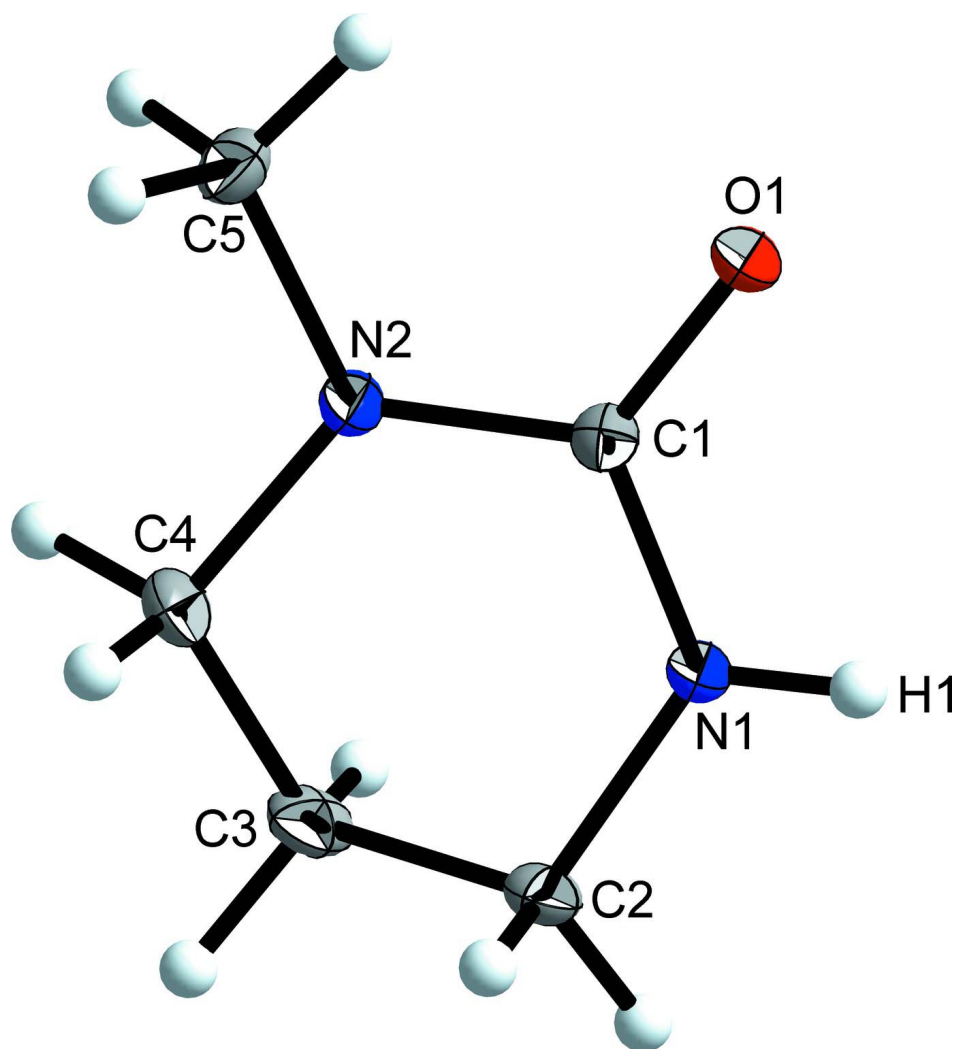


Figure 1

The molecular structure of the title molecule. Anisotropic displacement ellipsoids are shown at the 50% probability level.

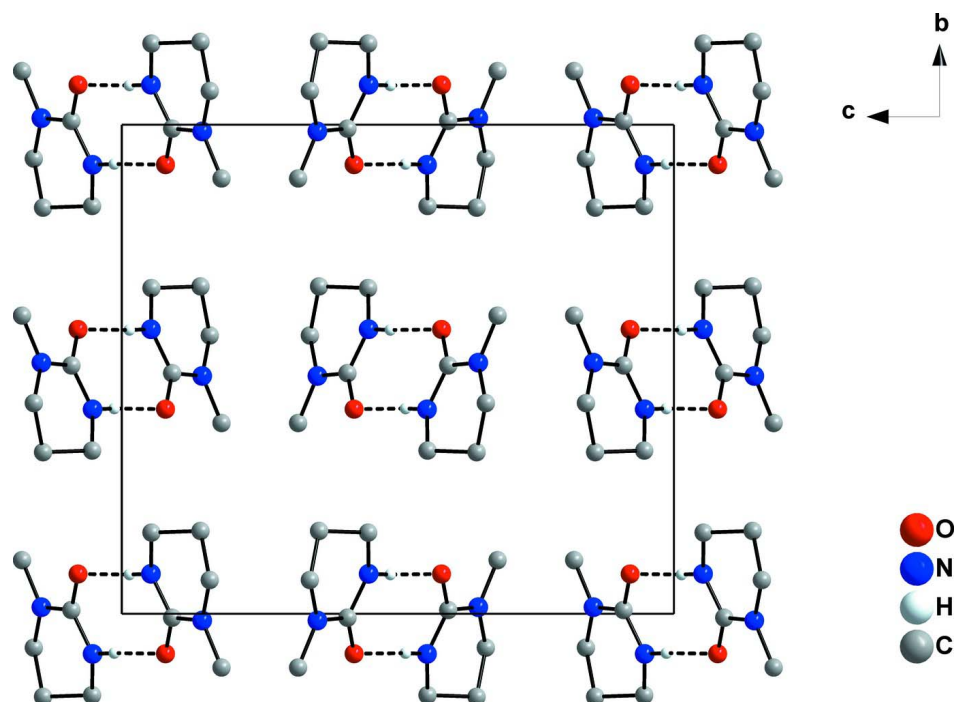


Figure 2

Packing diagram of the title compound, *bc*-view. The N–H···O hydrogen bonds are indicated by dashed lines.

1-Methyl-1,3-diazinan-2-one

Crystal data

$C_5H_{10}N_2O$

$M_r = 114.15$

Orthorhombic, *Pbca*

Hall symbol: $-P\ 2ac\ 2ab$

$a = 5.8479\ (2)\ \text{\AA}$

$b = 13.3438\ (6)\ \text{\AA}$

$c = 15.0883\ (8)\ \text{\AA}$

$V = 1177.39\ (9)\ \text{\AA}^3$

$Z = 8$

$F(000) = 496$

$D_x = 1.288\ \text{Mg m}^{-3}$

Mo $K\alpha$ radiation, $\lambda = 0.71073\ \text{\AA}$

Cell parameters from 1677 reflections

$\theta = 0.4\text{--}28.3^\circ$

$\mu = 0.09\ \text{mm}^{-1}$

$T = 100\ \text{K}$

Lath-shaped, colourless

$0.19 \times 0.15 \times 0.11\ \text{mm}$

Data collection

Bruker–Nonius KappaCCD
diffractometer

Radiation source: sealed tube

Graphite monochromator

φ scans, and ω scans

2628 measured reflections

1434 independent reflections

1190 reflections with $I > 2\sigma(I)$

$R_{\text{int}} = 0.024$

$\theta_{\text{max}} = 28.2^\circ$, $\theta_{\text{min}} = 2.7^\circ$

$h = -7 \rightarrow 7$

$k = -17 \rightarrow 17$

$l = -19 \rightarrow 19$

Refinement

Refinement on F^2

Least-squares matrix: full

$R[F^2 > 2\sigma(F^2)] = 0.037$

$wR(F^2) = 0.092$

$S = 1.03$

1434 reflections

79 parameters

0 restraints

Primary atom site location: structure-invariant
direct methods

Secondary atom site location: difference Fourier map

Hydrogen site location: difference Fourier map

H atoms treated by a mixture of independent and constrained refinement

$$w = 1/[\sigma^2(F_o^2) + (0.0424P)^2 + 0.4078P]$$

$$\text{where } P = (F_o^2 + 2F_c^2)/3$$

$$(\Delta/\sigma)_{\max} < 0.001$$

$$\Delta\rho_{\max} = 0.27 \text{ e } \text{\AA}^{-3}$$

$$\Delta\rho_{\min} = -0.18 \text{ e } \text{\AA}^{-3}$$

Extinction correction: *SHELXL97* (Sheldrick, 2008), $F_c^* = kFc[1 + 0.001x Fc^2 \lambda^3 / \sin(2\theta)]^{-1/4}$

Extinction coefficient: 0.023 (3)

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 $F^2 > \sigma(F^2)$ 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 (\AA^2)

	<i>x</i>	<i>y</i>	<i>z</i>	$U_{\text{iso}}^*/U_{\text{eq}}$
O1	0.13599 (13)	0.58154 (5)	0.07922 (5)	0.0178 (2)
N1	0.21849 (16)	0.41809 (6)	0.05287 (6)	0.0162 (2)
H1	0.108 (3)	0.4198 (11)	0.0134 (10)	0.032 (4)*
N2	0.44922 (16)	0.51361 (6)	0.14565 (6)	0.0147 (2)
C1	0.26381 (18)	0.50789 (7)	0.09125 (6)	0.0127 (2)
C2	0.3761 (2)	0.33384 (7)	0.05379 (7)	0.0170 (2)
H2A	0.4901	0.3415	0.0058	0.020*
H2B	0.2913	0.2706	0.0439	0.020*
C3	0.4956 (2)	0.33051 (8)	0.14262 (7)	0.0203 (3)
H3A	0.6120	0.2766	0.1427	0.024*
H3B	0.3834	0.3162	0.1901	0.024*
C4	0.6091 (2)	0.43089 (8)	0.15938 (8)	0.0209 (3)
H4A	0.6673	0.4330	0.2210	0.025*
H4B	0.7411	0.4387	0.1189	0.025*
C5	0.5230 (2)	0.60977 (8)	0.18066 (7)	0.0182 (3)
H5A	0.4106	0.6613	0.1650	0.027*
H5B	0.6719	0.6274	0.1553	0.027*
H5C	0.5362	0.6055	0.2453	0.027*

Atomic displacement parameters (\AA^2)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
O1	0.0179 (4)	0.0130 (4)	0.0226 (4)	0.0039 (3)	-0.0052 (3)	-0.0015 (3)
N1	0.0153 (5)	0.0118 (4)	0.0213 (5)	0.0022 (4)	-0.0052 (4)	-0.0026 (3)
N2	0.0148 (5)	0.0120 (4)	0.0173 (4)	0.0003 (4)	-0.0034 (3)	0.0002 (3)
C1	0.0131 (5)	0.0122 (4)	0.0128 (4)	-0.0013 (4)	0.0008 (4)	0.0015 (4)
C2	0.0195 (5)	0.0118 (5)	0.0196 (5)	0.0036 (4)	-0.0003 (4)	-0.0011 (4)
C3	0.0260 (6)	0.0142 (5)	0.0206 (5)	0.0071 (5)	-0.0023 (5)	0.0023 (4)
C4	0.0193 (6)	0.0195 (5)	0.0241 (6)	0.0063 (5)	-0.0066 (5)	-0.0012 (4)

C5	0.0196 (5)	0.0160 (5)	0.0190 (5)	-0.0038 (4)	-0.0034 (4)	-0.0010 (4)
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Geometric parameters (Å, °)

O1—C1	1.2480 (13)	C2—H2B	0.9900
N1—C1	1.3570 (13)	C3—C4	1.5162 (16)
N1—C2	1.4537 (13)	C3—H3A	0.9900
N1—H1	0.881 (18)	C3—H3B	0.9900
N2—C1	1.3620 (14)	C4—H4A	0.9900
N2—C5	1.4532 (13)	C4—H4B	0.9900
N2—C4	1.4614 (14)	C5—H5A	0.9800
C2—C3	1.5123 (15)	C5—H5B	0.9800
C2—H2A	0.9900	C5—H5C	0.9800
C1—N1—C2	123.71 (9)	C4—C3—H3A	109.9
C1—N1—H1	114.2 (9)	C2—C3—H3B	109.9
C2—N1—H1	119.6 (10)	C4—C3—H3B	109.9
C1—N2—C5	120.32 (8)	H3A—C3—H3B	108.3
C1—N2—C4	123.54 (9)	N2—C4—C3	111.31 (9)
C5—N2—C4	115.18 (9)	N2—C4—H4A	109.4
O1—C1—N1	121.08 (10)	C3—C4—H4A	109.4
O1—C1—N2	121.36 (9)	N2—C4—H4B	109.4
N1—C1—N2	117.52 (9)	C3—C4—H4B	109.4
N1—C2—C3	108.92 (8)	H4A—C4—H4B	108.0
N1—C2—H2A	109.9	N2—C5—H5A	109.5
C3—C2—H2A	109.9	N2—C5—H5B	109.5
N1—C2—H2B	109.9	H5A—C5—H5B	109.5
C3—C2—H2B	109.9	N2—C5—H5C	109.5
H2A—C2—H2B	108.3	H5A—C5—H5C	109.5
C2—C3—C4	108.92 (8)	H5B—C5—H5C	109.5
C2—C3—H3A	109.9		
C2—N1—C1—O1	170.94 (10)	C1—N1—C2—C3	37.71 (14)
C2—N1—C1—N2	-11.24 (15)	N1—C2—C3—C4	-55.31 (12)
C5—N2—C1—O1	-9.34 (16)	C1—N2—C4—C3	-25.76 (14)
C4—N2—C1—O1	-177.57 (10)	C5—N2—C4—C3	165.46 (9)
C5—N2—C1—N1	172.84 (9)	C2—C3—C4—N2	50.42 (12)
C4—N2—C1—N1	4.62 (15)		

Hydrogen-bond geometry (Å, °)

<i>D</i> —H \cdots <i>A</i>	<i>D</i> —H	H \cdots <i>A</i>	<i>D</i> \cdots <i>A</i>	<i>D</i> —H \cdots <i>A</i>
N1—H1 \cdots O1 ⁱ	0.88 (2)	2.00 (2)	2.875 (1)	177 (2)

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