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Cylopentylamine monohydrate

D. R. Allan

School of Chemistry, The University of
Edinburgh, King's Buildings, West Mains Road,
Edinburgh EH9 3JJ, Scotland

Correspondence e-mail: d.r.allan@ed.ac.uk

Key indicators

Single-crystal synchrotron study
$T = 205$ K
Mean σ(C–C) = 0.003 Å
R factor = 0.056
wR factor = 0.054
Data-to-parameter ratio = 11.5

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

The crystal structure of cylopentylamine monohydrate, $C_5H_{11}N \cdot H_2O$, is composed of molecular chains of alternating cyclopentylamine and water molecules which are linked by O—H$\cdots$O and O—H$\cdots$N hydrogen bonds. These chains are parallel to the monoclinic b axis and they are bridged by weaker O$\cdots$H—N contacts, forming hydrogen-bonded layers of molecules parallel to (100).

Comment

The crystal structure of cyclopentylamine monohydrate, (I), was determined at 205 K (just below the $\sim$215 K melting point) as part of a series of studies on the structural behaviour of prototypical hydrogen-bonded molecular systems at conditions of either non-ambient temperature or pressure. It crystallizes in the monoclinic space group $P2_1/c$ with one cyclopentylamine molecule and a single water molecule in the asymmetric unit (Fig. 1).
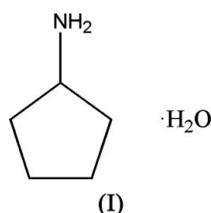
The water molecules are linked by O—H$\cdots$O hydrogen bonds, forming the backbone of molecular chains which run parallel to the b axis, while O—H$\cdots$N hydrogen bonds link the cyclopentylamine molecules to this backbone in an alternating sequence (Fig. 2 and Table 1). The lengths of these hydrogen bonds are fairly similar, while the weaker O—H$\cdots$N hydrogen bond is correspondingly less linear. Significantly weaker O$\cdots$H—N contacts bridge neighbouring molecular chains, forming slabs of molecules parallel to (100) (Fig. 3). One of these O$\cdots$N distances is marginal.

Experimental

The sample of cyclopentylamine monohydrate was prepared from anhydrous starting material (of 99% purity, as received from Aldrich) and placed in a sealed glass capillary tube with an internal diameter of ca 0.2 mm. The sample was cooled using an Oxford Cryosystems low-temperature device (Cosier & Glazer, 1986) until crystallization was observed. The temperature was then cycled between 180 and 215 K, and the capillary successively translated through the gas stream, so that the sample was partially remelted and the number of crystallites reduced. The final sample, at 205 K, was composed of a small number
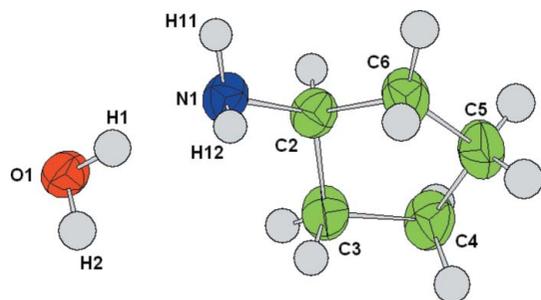
Figure 1
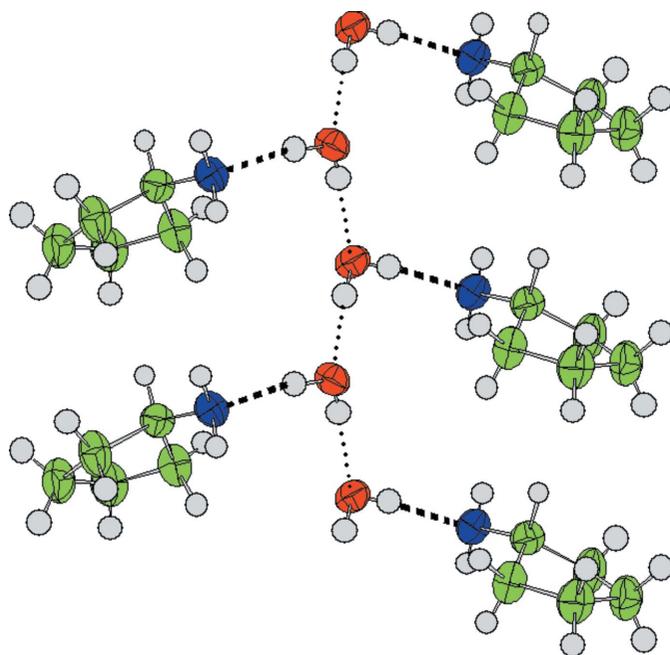The asymmetric unit of (I), showing 30% probability displacement ellipsoids.

of crystals and the reflections from the largest of these were indexed and their intensities subsequently used for structure solution.

Crystal data

$C_5H_{11}N\cdot H_2O$
$M_r = 103.16$
Monoclinic, $P2_1/c$
$a = 12.969\,(4)$ Å
$b = 4.7125\,(13)$ Å
$c = 11.005\,(3)$ Å
$\beta = 102.614\,(17)°$
$V = 656.4\,(3)$ Å^3
$Z = 4$
$D_x = 1.044$ Mg m^{-3}

Synchrotron radiation
$\lambda = 0.6813$ Å
Cell parameters from 445 reflections
$\theta = 8–43°$
$\mu = 0.07$ mm^{-1}
$T = 205$ K
Cylinder, colourless
0.20×0.10 (radius) mm

Data collection

Bruker SMART diffractometer
φ and ω scans
Absorption correction: multi-scan (*SADABS*; Sheldrick, 2004)
$T_{min} = 0.35$, $T_{max} = 0.99$
5211 measured reflections
1573 independent reflections

875 reflections with $I > 2\sigma(I)$
$R_{int} = 0.051$
$\theta_{max} = 27.5°$
$h = -17 \rightarrow 17$
$k = -6 \rightarrow 6$
$l = -14 \rightarrow 14$

Refinement

Refinement on F
$R[F^2 > 2\sigma(F^2)] = 0.056$
$wR(F^2) = 0.054$
$S = 1.13$
875 reflections
76 parameters
H atoms treated by a mixture of independent and constrained refinement

Modified Chebychev polynomial (Watkin, 1994; Prince, 1982) with the coefficients 2.88, −1.06, 1.90
$(\Delta/\sigma)_{max} = 0.001$
$\Delta\rho_{max} = 0.25$ e Å^{-3}
$\Delta\rho_{min} = -0.16$ e Å^{-3}

Figure 2
The hydrogen-bonded molecular chains of (I), viewed perpendicular to (101). The O—H···O and O—H···N hydrogen bonds are shown as light dotted lines and heavy dashed lines, respectively.
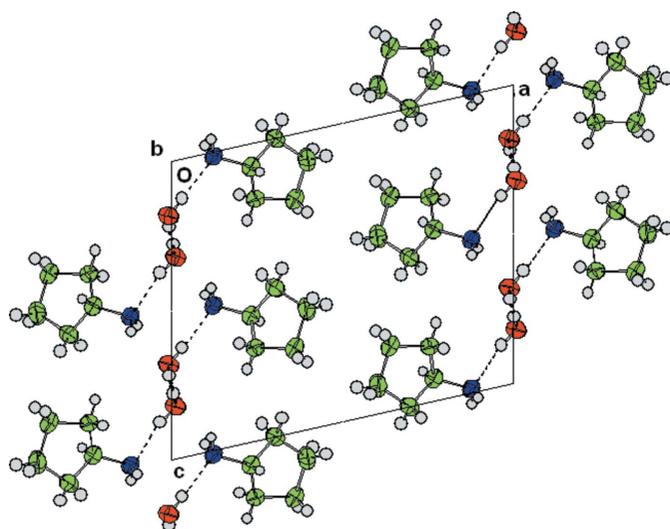
Table 1
Hydrogen-bond geometry (Å, °).

D—H···A	D—H	H···A	D···A	D—H···A
O1—H1···N1	0.82 (1)	2.01 (1)	2.821 (2)	172 (2)
O1—H2···O1^i	0.82 (1)	2.00 (1)	2.820 (1)	178 (3)
N1—H11···O1^{ii}	0.89 (1)	2.35 (1)	3.137 (2)	148 (2)
N1—H12···O1^{iii}	0.89 (1)	2.55 (1)	3.426 (2)	166 (2)

Symmetry codes: (i) $-x+2, y+\frac{1}{2}, -z+\frac{3}{2}$; (ii) $-x+2, -y, -z+2$; (iii) $-x+2, -y+1, -z+2$.

H atoms attached to C atoms were placed in idealized positions (C—H = 0.96–1.00 Å) and allowed to ride on their parent atoms. H atoms attached to N and O atoms were located in a difference map and restrained to idealized distances and angles [N—H = 0.90 (1) Å, O—H = 0.82 (1) Å and O—H—O = 104 (1)°]. All H atoms were constrained so that U_{iso}(H) values were equal to $1.2U_{eq}$ of their respective parent atoms.

Data collection: *SMART* (Bruker, 2001); cell refinement: *SAINT*; data reduction: *SAINT* (Bruker, 2003); program(s) used to solve structure: *SIR92* (Altomare *et al.*, 1994); program(s) used to refine structure: *CRYSTALS* (Betteridge *et al.*, 2003); molecular graphics: *CAMERON* (Watkin *et al.*, 1996); software used to prepare material for publication: *CRYSTALS* and *PLATON* (Spek, 2003).

Figure 3
The packing of (I), viewed along the *b* axis. The O—H···O and O—H···N hydrogen bonds are shown as dashed lines.

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supporting information

Cylopentylamine monohydrate

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Monoclinic, $P2_1/c$	Synchrotron radiation, $\lambda = 0.68130$ Å
Hall symbol: -P 2ybc	Cell parameters from 445 reflections
$a = 12.969$ (4) Å	$\theta = 8$–43°
$b = 4.7125$ (13) Å	$\mu = 0.07$ mm^{-1}
$c = 11.005$ (3) Å	$T = 205$ K
$\beta = 102.614$ (17)°	Cylinder, colourless
$V = 656.4$ (3) Å^3	$0.20 \times 0.10 \times 0.10 \times 0.10$ (radius) mm
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Data collection

Bruker SMART diffractometer	1573 independent reflections
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φ and ω scans	$R_{int} = 0.051$
Absorption correction: multi-scan (SADABS; Sheldrick, 2004)	$\theta_{max} = 27.5°$, $\theta_{min} = 4.3°$
$T_{min} = 0.35$, $T_{max} = 0.99$	$h = -17 \rightarrow 17$
5211 measured reflections	$k = -6 \rightarrow 6$
	$l = -14 \rightarrow 14$

Refinement

Refinement on F	Hydrogen site location: inferred from neighbouring sites
Least-squares matrix: full	H atoms treated by a mixture of independent and constrained refinement
$R[F^2 > 2\sigma(F^2)] = 0.056$	
$wR(F^2) = 0.054$	Modified Chebychev polynomial (Watkin, 1994; *P*rince, 1982) *w*ith the coefficients 2.88, -1.06, 1.90
$S = 1.13$	
875 reflections	
76 parameters	$(\Delta/\sigma)_{max} = 0.001$
5 restraints	$\Delta\rho_{max} = 0.25$ e Å^{-3}
Primary atom site location: structure-invariant direct methods	$\Delta\rho_{min} = -0.16$ e Å^{-3}

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

	x	y	z	U_{iso}*/U_{eq}
O1	1.01039 (11)	0.1644 (3)	0.82203 (12)	0.0497
N1	0.87797 (12)	0.2930 (4)	0.98670 (14)	0.0473
C2	0.76853 (14)	0.3412 (4)	0.92430 (17)	0.0473
C3	0.75593 (15)	0.5310 (5)	0.81296 (17)	0.0552

C6	0.69733 (15)	0.4823 (6)	1.00014 (19)	0.0638
C4	0.63976 (15)	0.6122 (6)	0.78280 (19)	0.0630
C5	0.60449 (16)	0.5968 (6)	0.9053 (2)	0.0678
H21	0.7390	0.1534	0.8947	0.0574*
H32	0.7998	0.7044	0.8354	0.0677*
H31	0.7771	0.4371	0.7433	0.0675*
H62	0.7337	0.6375	1.0504	0.0798*
H61	0.6744	0.3489	1.0547	0.0796*
H42	0.6295	0.8019	0.7461	0.0731*
H41	0.5999	0.4774	0.7238	0.0730*
H52	0.5840	0.7785	0.9321	0.0824*
H51	0.5454	0.4708	0.9009	0.0827*
H1	0.9678 (15)	0.188 (5)	0.8667 (18)	0.0777*
H12	0.9016 (16)	0.454 (3)	1.0256 (19)	0.0630*
H2	1.0062 (19)	0.311 (3)	0.7810 (19)	0.0780*
H11	0.8823 (17)	0.155 (4)	1.0422 (16)	0.0627*

Atomic displacement parameters (Å²)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
O1	0.0579 (8)	0.0455 (8)	0.0482 (8)	0.0092 (7)	0.0172 (6)	0.0043 (6)
N1	0.0446 (9)	0.0530 (11)	0.0420 (8)	0.0072 (8)	0.0044 (7)	0.0023 (8)
C2	0.0442 (10)	0.0479 (11)	0.0474 (10)	0.0012 (9)	0.0046 (8)	−0.0010 (9)
C3	0.0486 (10)	0.0762 (15)	0.0403 (10)	0.0097 (11)	0.0089 (8)	0.0055 (10)
C6	0.0542 (12)	0.0909 (17)	0.0500 (11)	0.0145 (12)	0.0194 (9)	0.0110 (12)
C4	0.0465 (11)	0.0831 (17)	0.0536 (12)	0.0070 (11)	−0.0021 (9)	0.0056 (11)
C5	0.0438 (11)	0.0847 (17)	0.0756 (15)	0.0073 (11)	0.0148 (10)	0.0102 (13)

Geometric parameters (Å, °)

O1—H1	0.823 (9)	C3—H31	0.975
O1—H2	0.822 (9)	C6—C5	1.510 (3)
N1—C2	1.453 (2)	C6—H62	0.975
N1—H12	0.894 (9)	C6—H61	0.960
N1—H11	0.887 (9)	C4—C5	1.517 (3)
C2—C3	1.497 (3)	C4—H42	0.978
C2—C6	1.526 (3)	C4—H41	0.973
C2—H21	0.991	C5—H52	0.961
C3—C4	1.519 (3)	C5—H51	0.962
C3—H32	0.995		
H1—O1—H2	103.9 (9)	C2—C6—H62	111.3
C2—N1—H12	106.9 (14)	C5—C6—H62	109.7
C2—N1—H11	110.4 (14)	C2—C6—H61	111.4
H12—N1—H11	109.4 (19)	C5—C6—H61	110.9
N1—C2—C3	113.65 (16)	H62—C6—H61	108.3
N1—C2—C6	117.06 (16)	C3—C4—C5	105.72 (16)
C3—C2—C6	102.50 (17)	C3—C4—H42	111.0

N1—C2—H21	106.4	C5—C4—H42	111.8
C3—C2—H21	107.4	C3—C4—H41	109.8
C6—C2—H21	109.4	C5—C4—H41	110.0
C2—C3—C4	104.77 (17)	H42—C4—H41	108.4
C2—C3—H32	109.5	C4—C5—C6	106.34 (16)
C4—C3—H32	109.5	C4—C5—H52	112.7
C2—C3—H31	111.9	C6—C5—H52	109.8
C4—C3—H31	112.3	C4—C5—H51	112.1
H32—C3—H31	108.9	C6—C5—H51	108.7
C2—C6—C5	105.32 (17)	H52—C5—H51	107.2

Hydrogen-bond geometry (Å, º)

D—H···A	D—H	H···A	D···A	D—H···A
O1—H1···N1	0.82 (1)	2.01 (1)	2.821 (2)	172 (2)
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