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Cyclobutylamine hemihydrate

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Key indicators

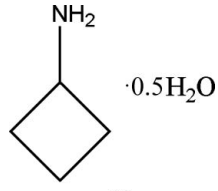
Single-crystal synchrotron study
T = 205 K
Mean σ(C–C) = 0.003 Å
R factor = 0.063
wR factor = 0.072
Data-to-parameter ratio = 12.0

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

The asymmetric unit of cyclobutylamine hemihydrate, $C_4H_9N{\cdot}0.5H_2O$, consists of two cyclobutylamine molecules bridged by a water molecule *via* N···H—O hydrogen bonds. This molecular arrangement is further connected by significantly weaker N—H···O contacts to form columns parallel to the *b* axis.

Comment

The crystal structure of cyclobutylamine hemihydrate ($C_4H_7NH_2{\cdot}0.5H_2O$), (I), was determined at 205 K (just below the ~210 K melting point) as part of our low-temperature and high-pressure structural studies of prototypical hydrogen-bonded molecular systems. It crystallizes in the monoclinic space group $P2_1/n$ with two cyclobutylamine molecules and one water molecule in the asymmetric unit (Fig. 1). Pairs of cyclobutylamine molecules are bridged by a single water molecule through N···H—O hydrogen bonds, which have N···O distances of 2.880 (3) and 2.895 (2) Å (Fig. 2 and Table 1). Significantly weaker N—H···O contacts link this molecular assembly to form columns parallel to the *b* axis, with N···O distances ranging in length from 3.176 (3) and 3.281 (3) Å to a more marginal distance of 3.604 (3) Å. As the N···O distances increase, there is a concomitant decrease in the N—H···O angles from 173.0 (19) to 160.1 (19)° as the interaction weakens. The remaining N—H···O interaction (N11—H111···O1) would appear to link the columns into slabs parallel to ($\bar{1}$01). However, as this interaction has a very long N···O contact distance of 3.833 (3) Å, and the N—H···O angle is 134.3 (15)°, it is unlikely to offer any significant contribution to the intermolecular bonding.



(I)

Experimental

The sample of cyclobutylamine hemihydrate 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, by successive translations of the capillary through the gas stream, so that the sample was

partially remelted and the number of crystallites reduced, until a single crystal was obtained at 205 K.

Crystal data

$C_4H_9N{\cdot}0.5H_2O$
M_r = 80.13
Monoclinic, $P2_1/n$
a = 14.048 (6) Å
b = 5.209 (2) Å
c = 14.489 (6) Å
β = 97.369 (4)°
V = 1051.5 (7) Å^3
Z = 8
D_x = 1.012 Mg m^{-3}
Synchrotron radiation
λ = 0.6813 Å
Cell parameters from 2051 reflections
θ = 8–46°
μ = 0.07 mm^{-1}
T = 205 K
Cylinder, colourless
0.20 × 0.20 (radius) mm

Data collection

Bruker SMART diffractometer
ω scans
Absorption correction: multi-scan (*SADABS*; Sheldrick, 2004)
T_{min} = 0.55, T_{max} = 0.99
8565 measured reflections
2525 independent reflections
1411 reflections with $I > 2\sigma(I)$
R_{int} = 0.071
θ_{max} = 27.5°
$h = -18 \rightarrow 19$
$k = -6 \rightarrow 6$
$l = -19 \rightarrow 18$

Refinement

Refinement on F
$R[F^2 > 2\sigma(F^2)]$ = 0.063
$wR(F^2)$ = 0.072
S = 1.14
1411 reflections
118 parameters
H atoms treated by a mixture of independent and constrained refinement
$w = [1 - (F_o - F_c)^2/36\sigma^2(F)]^2/[2.28T_o(x) + 0.243T_1(x) + 1.74T_2(x)]$ where T_i are Chebychev polynomials and $x = F_c/F_{max}$ (Prince, 1982; Watkin, 1994)
$(\Delta/\sigma)_{max} < 0.001$
$\Delta\rho_{max}$ = 0.17 e Å^{-3}
$\Delta\rho_{min}$ = −0.18 e Å^{-3}

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

D—H···A	D—H	H···A	D···A	D—H···A
O1—H1···N11	0.82 (1)	2.08 (1)	2.895 (2)	174 (3)
O1—H2···N21	0.82 (1)	2.07 (1)	2.880 (3)	174 (3)

H atoms attached to C atoms were placed in idealized positions (C—H = 0.94–1.00 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) was 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).

We thank Dr T. Prior of Daresbury Laboratory for his help during the experiment on station 9.8 at SRS. We also thank the EPSRC for funding both this project and DRA's Advanced Research Fellowship.

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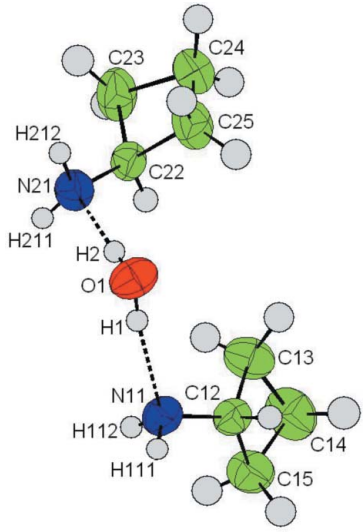

Figure 1
The asymmetric unit of (I), showing 30% probability displacement ellipsoids. The dashed lines indicate the O—H···N hydrogen bonds.

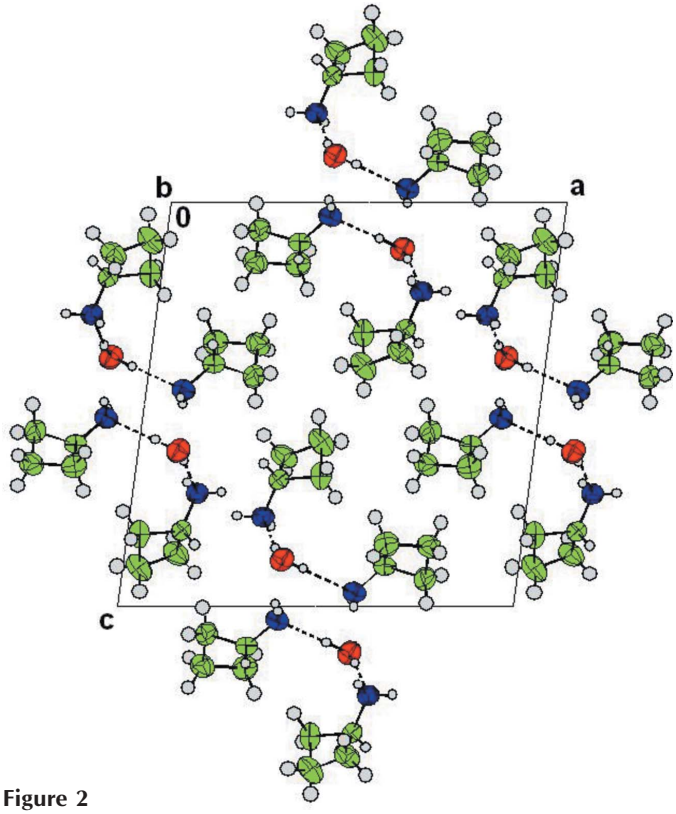

Figure 2
The packing of (I), viewed along the b axis. The O—H···N hydrogen bonds are shown as dashed lines.

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$R_{int} = 0.071$
$\theta_{max} = 27.5°$, $\theta_{min} = 4.0°$
$h = -18\rightarrow19$
$k = -6\rightarrow6$
$l = -19\rightarrow18$

Refinement

Refinement on F
Least-squares matrix: full
$R[F^2 > 2\sigma(F^2)] = 0.063$
$wR(F^2) = 0.072$
$S = 1.14$
1411 reflections
118 parameters
7 restraints
Primary atom site location: structure-invariant direct methods
Hydrogen site location: inferred from neighbouring sites
H atoms treated by a mixture of independent and constrained refinement
$w = [1-(F_o-F_c)^2/36\sigma^2(F)]^2/[2.28T_o(x) + 0.243T_1(x) + 1.74T_2(x)]$
where T_i are the Chebychev polynomials and x = F_c/F_{max} (*P*rince, 1982; Watkin, 1994)
$(\Delta/\sigma)_{max} = 0.000218$
$\Delta\rho_{max} = 0.17$ e Å^{-3}
$\Delta\rho_{min} = -0.18$ e Å^{-3}

Special details

Refinement. ABSTM02_ALERT_3_B The ratio of expected to reported Tmax/Tmin(*RR*′) is < 0.75 T min and Tmax reported: 0.550 0.990 T min(prime) and Tmax expected: 0.987 0.987 *RR*(prime) = 0.556
SADABS was also used to correct for the decay of the synchrotron X-ray beam. The overall sample absorption, especially at the relatively short wavelength, is extremely low.
PLAT241_ALERT_2_C Check High U_{eq} as Compared to Neighbors for C23 PLAT242_ALERT_2_C Check Low U_{eq} as Compared to Neighbors for C12 PLAT242_ALERT_2_C Check Low U_{eq} as Compared to Neighbors for C22
The data were collected very close to the sample melting temperature and, consequently, the temperature factors are relatively large.
PLAT420_ALERT_2_C D—H Without Acceptor N11 - H111 ··· ? PLAT420_ALERT_2_C D—H Without Acceptor N21 - H211 ··· ?
Although the relevant N—H···O angles suggest that the oxygen atom acts as an acceptor for both N11—H111 and N21—H211, the H···A distances are relatively long and suggest that these interactions are at best extremely weak. Details of the various distances are mentioned in the comments section.

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

	x	*y*	*z*	U_{iso}*/U_{eq}
N11	0.66281 (12)	0.6261 (3)	0.22065 (10)	0.0590
C12	0.63480 (12)	0.6188 (3)	0.31320 (12)	0.0524
C13	0.52996 (14)	0.5731 (4)	0.32271 (17)	0.0742
C14	0.54031 (19)	0.7577 (5)	0.40528 (19)	0.0882
C15	0.62917 (17)	0.8626 (4)	0.37068 (15)	0.0751
O1	0.60087 (11)	0.1702 (3)	0.11547 (10)	0.0705
N21	0.40677 (12)	0.2763 (3)	0.03446 (11)	0.0623
C22	0.33839 (13)	0.2419 (4)	0.09937 (11)	0.0534
C25	0.34180 (17)	−0.0039 (5)	0.15398 (16)	0.0773
C24	0.23267 (16)	0.0020 (5)	0.14836 (15)	0.0752
C23	0.23237 (15)	0.1925 (6)	0.06886 (15)	0.0846
H121	0.6732	0.4839	0.3504	0.0625*
H131	0.5138	0.3963	0.3372	0.0889*
H132	0.4884	0.6336	0.2675	0.0894*
H141	0.5514	0.6690	0.4633	0.1089*
H142	0.4882	0.8755	0.4054	0.1088*
H151	0.6820	0.8860	0.4183	0.0917*
H152	0.6191	1.0139	0.3349	0.0923*
H221	0.3435	0.3886	0.1421	0.0646*
H251	0.3778	0.0052	0.2178	0.0935*
H252	0.3650	−0.1418	0.1190	0.0938*
H241	0.2105	0.0777	0.2026	0.0910*
H242	0.1995	−0.1578	0.1341	0.0915*
H231	0.1894	0.3458	0.0706	0.1014*
H232	0.2216	0.1046	0.0084	0.1013*
H211	0.4070 (17)	0.435 (2)	0.0116 (15)	0.0744*
H1	0.6198 (15)	0.303 (3)	0.1417 (16)	0.1011*
H2	0.5447 (9)	0.202 (5)	0.0966 (18)	0.1017*
H212	0.4029 (16)	0.142 (3)	−0.0039 (13)	0.0737*
H111	0.7268 (7)	0.634 (4)	0.2227 (14)	0.0715*
H112	0.6347 (14)	0.767 (3)	0.1950 (14)	0.0719*

Atomic displacement parameters (Å²)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
N11	0.0650 (9)	0.0589 (9)	0.0525 (8)	−0.0002 (7)	0.0051 (7)	−0.0081 (7)
C12	0.0524 (9)	0.0517 (9)	0.0517 (9)	0.0010 (7)	0.0018 (7)	−0.0005 (7)
C13	0.0556 (11)	0.0703 (13)	0.0975 (15)	−0.0014 (9)	0.0131 (10)	−0.0004 (12)
C14	0.0908 (16)	0.0859 (16)	0.0964 (16)	0.0068 (13)	0.0448 (13)	−0.0057 (14)
C15	0.0918 (15)	0.0652 (12)	0.0728 (13)	−0.0111 (10)	0.0278 (11)	−0.0215 (10)
O1	0.0712 (9)	0.0643 (9)	0.0741 (9)	0.0055 (7)	0.0025 (7)	−0.0171 (7)
N21	0.0700 (10)	0.0620 (10)	0.0558 (8)	−0.0048 (8)	0.0120 (7)	0.0000 (8)
C22	0.0667 (10)	0.0482 (9)	0.0447 (8)	0.0021 (8)	0.0054 (7)	−0.0016 (7)
C25	0.0811 (14)	0.0738 (14)	0.0764 (13)	0.0088 (11)	0.0073 (10)	0.0255 (11)
C24	0.0802 (14)	0.0782 (15)	0.0694 (13)	−0.0122 (11)	0.0181 (10)	0.0092 (11)
C23	0.0596 (11)	0.122 (2)	0.0709 (12)	0.0029 (12)	0.0025 (9)	0.0293 (13)

Geometric parameters (Å, º)

N11—C12	1.446 (2)	O1—H2	0.819 (10)
N11—H111	0.896 (9)	N21—C22	1.439 (2)
N11—H112	0.892 (9)	N21—H211	0.892 (9)
C12—C13	1.516 (3)	N21—H212	0.892 (9)
C12—C15	1.526 (3)	C22—C25	1.502 (3)
C12—H121	1.001	C22—C23	1.520 (3)
C13—C14	1.527 (4)	C22—H221	0.980
C13—H131	0.977	C25—C24	1.525 (3)
C13—H132	0.980	C25—H251	0.996
C14—C15	1.506 (3)	C25—H252	0.960
C14—H141	0.955	C24—C23	1.520 (3)
C14—H142	0.955	C24—H241	0.966
C15—H151	0.954	C24—H242	0.963
C15—H152	0.944	C23—H231	1.004
O1—H1	0.817 (10)	C23—H232	0.983
C12—N11—H111	111.2 (14)	C22—N21—H211	113.2 (15)
C12—N11—H112	104.5 (14)	C22—N21—H212	108.5 (14)
H111—N11—H112	111.5 (19)	H211—N21—H212	120 (2)
N11—C12—C13	118.15 (16)	N21—C22—C25	118.13 (17)
N11—C12—C15	121.58 (16)	N21—C22—C23	122.83 (15)
C13—C12—C15	87.84 (15)	C25—C22—C23	88.44 (17)
N11—C12—H121	109.1	N21—C22—H221	108.3
C13—C12—H121	107.7	C25—C22—H221	109.7
C15—C12—H121	110.6	C23—C22—H221	107.8
C12—C13—C14	88.64 (17)	C22—C25—C24	89.49 (16)
C12—C13—H131	115.0	C22—C25—H251	115.2
C14—C13—H131	115.2	C24—C25—H251	115.9
C12—C13—H132	111.1	C22—C25—H252	110.5
C14—C13—H132	115.1	C24—C25—H252	113.2
H131—C13—H132	110.4	H251—C25—H252	111.0

C13—C14—C15	88.14 (16)	C25—C24—C23	87.61 (15)
C13—C14—H141	111.9	C25—C24—H241	113.1
C15—C14—H141	114.9	C23—C24—H241	112.4
C13—C14—H142	114.2	C25—C24—H242	116.7
C15—C14—H142	115.8	C23—C24—H242	116.6
H141—C14—H142	110.3	H241—C24—H242	109.2
C12—C15—C14	89.01 (17)	C22—C23—C24	89.02 (15)
C12—C15—H151	114.3	C22—C23—H231	115.4
C14—C15—H151	114.0	C24—C23—H231	116.3
C12—C15—H152	114.2	C22—C23—H232	111.8
C14—C15—H152	114.5	C24—C23—H232	110.9
H151—C15—H152	109.8	H231—C23—H232	111.6
H1—O1—H2	103 (2)		

Hydrogen-bond geometry (Å, º)

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
O1—H1···N11	0.82 (1)	2.08 (1)	2.895 (2)	174 (3)
O1—H2···N21	0.82 (1)	2.07 (1)	2.880 (3)	174 (3)