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Cyclobutylamine hemihydrate

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The asymmetric unit of cyclobutylamine hemihydrate, $C_4H_9N\cdot0.5H_2O$, 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.

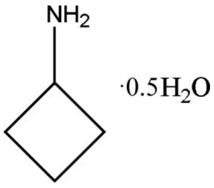
Key indicators

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.

Comment

The crystal structure of cyclobutylamine hemihydrate ($C_4H_7NH_2\cdot0.5H_2O$), (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
$\beta = 97.369\ (4)^\circ$
$V = 1051.5\ (7)$ Å^3
$Z = 8$
$D_x = 1.012$ Mg m^{-3}
Synchrotron radiation
$\lambda = 0.6813$ Å
Cell parameters from 2051 reflections
$\theta = 8$–46°
$\mu = 0.07$ 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 $I > 2\sigma(I)$
$R_{\rm int} = 0.071$
$\theta_{\max} = 27.5^\circ$
$h = -18 \rightarrow 19$
$k = -6 \rightarrow 6$
$l = -19 \rightarrow 18$

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 $U_{\rm iso}$(H) was equal to $1.2U_{\rm eq}$ 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).

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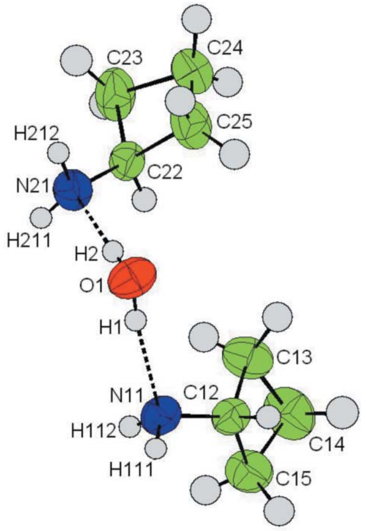

Figure 1
The asymmetric unit of (I), showing 30% probability displacement ellipsoids. The dashed lines indicate the O—H⋯N hydrogen bonds.

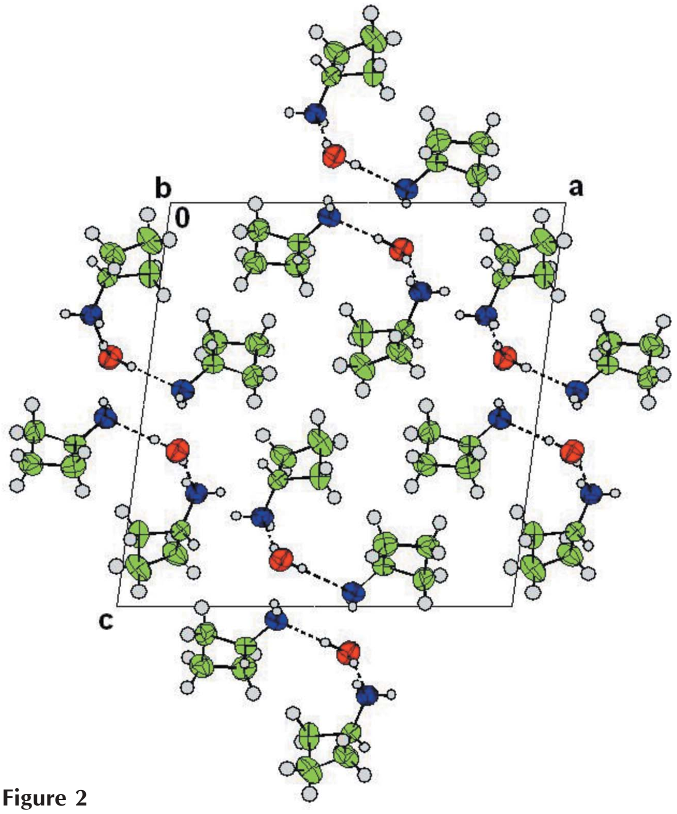

Figure 2
The packing of (I), viewed along the *b* axis. The O—H⋯N hydrogen bonds are shown as dashed lines.

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