A 1:1 molecular complex of dicyclohexylamine and cyclohexanone oxime

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The molecules of the title complex, $C_{12}H_{23}N \cdot C_6H_{11}N$, are linked together in chains by O—H$\cdots$N and N—H$\cdots$O hydrogen bonds.

Comment

Both components of the title complex, (I) (Fig. 1), show the expected molecular geometries and both cyclohexane rings of the dicyclohexylamine molecule adopt the chair conformation. The conformation of the oxime six-membered ring is half-chair, very similar to that observed in the structure of cyclohexanone oxime itself (Olivato *et al.*, 2001). The geometrical parameters of the oxime fragment show standard values for oximes (Chertanova *et al.*, 1994). The most interesting feature of this structure is the system of hydrogen bonds. The oxime hydrogen bonds were first classified by Bertolasi *et al.* (1982) and divided into three groups. The structures where the oxime unit is a donor group and forms one hydrogen bond are in group *A*. The structures with an additional hydrogen bond (oxime N atom is a hydrogen-bond acceptor) form group *B* and the structures with one more hydrogen bond, where the oxime O atom is the acceptor, are regarded as group *C*. The later and more elaborate classification introduced by Chertanova *et al.* (1994) shows very few examples of other motifs. In both cases the analyses did not include the directionality of the hydrogen bonds and supposed them to be in the plane of oxime group. In the case of complex (I), one of the two hydrogen bonds is the classical type *A* O—H$\cdots$N bond (Table 1 and Fig. 2). However, the geometry of the second hydrogen bond cannot be regarded as a pure *B*- or *C*-type bond. The N—H vector is not in the plane of the oxime group and is pointed toward the centre of the oxime O—N bond of an adjacent molecule. Therefore, this can be classified as a weak bifurcated hydrogen bond and the overall resulting bonding of the oxime unit in complex (I) is intermediate between *B*- and *C*-types.
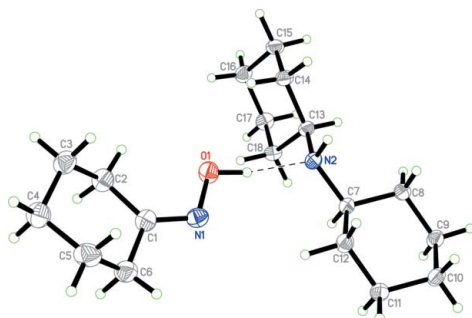

(I)
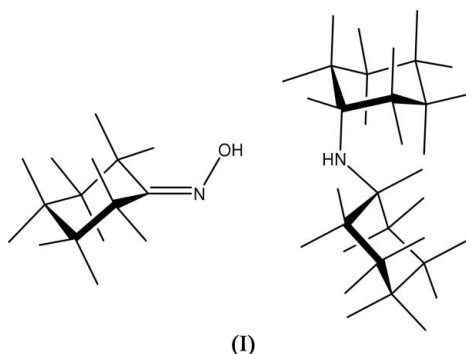
Figure 1
Structure of the molecular complex (I). Displacement ellipsoids are shown at the 50% probability level. The dashed line indicates a hydrogen bond.
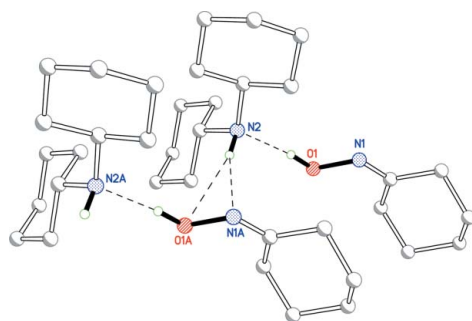

Figure 2
Hydrogen bonds (dashed lines) in the structure of (I). H atoms not involved in the interactions shown have been omitted.

The hydrogen bonds link the molecules into chains, parallel to the *c* axis. The oxime molecules in the chains are also connected by weak C1—H21···O1 (H···O = 2.71 Å) interactions. The chains form loose layers perpendicular to the *a* direction (Fig. 3)

Experimental

A solution of an organic compound (10 mg) in dicyclohexylamine (0.3 ml) was heated at 423 K in an open vessel for 2–3 min. The molecular complex (I) crystallized serendipitously as colourless needles after cooling of the solution, followed by standing at room temperature for several months. Presumably, the compound is a product of some oxidative conversion of the solvent.

Crystal data

$C_{12}H_{23}N \cdot C_6H_{11}NO$	$Z = 4$
$M_r = 294.47$	$D_x = 1.128$ Mg m^{-3}
Orthorhombic, $Pna2_1$	Mo- $K\alpha$ radiation
$a = 29.599$ (6) Å	$\mu = 0.07$ mm^{-1}
$b = 11.359$ (2) Å	$T = 120$ (2) K
$c = 5.1586$ (10) Å	Needle, colourless
$V = 1734.3$ (6) Å^3	$0.70 \times 0.04 \times 0.03$ mm

Data collection

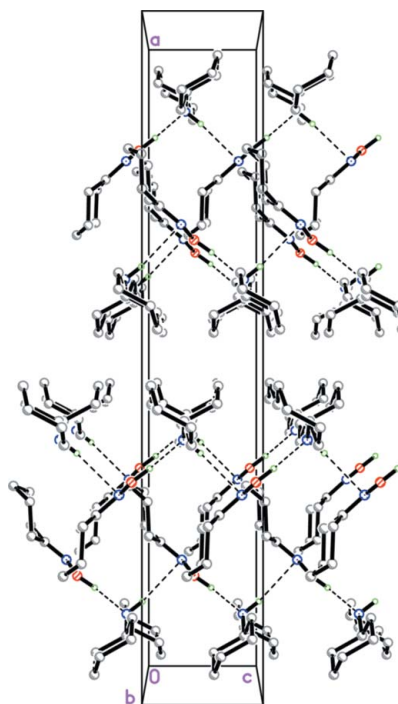|	
Bruker SMART CCD 6000 diffractometer	12334 measured reflections
ω scans	2118 independent reflections
Absorption correction: multi-scan (*SADABS*; Bruker, 2003)	1714 reflections with $I > 2\sigma(I)$
$T_{min} = 0.953$, $T_{max} = 0.998$	$R_{int} = 0.059$
	$\theta_{max} = 27.0°$

Figure 3
Packing of the molecules in the structure of (I), viewed along the *b* axis. H atoms not involved in the hydrogen bonds (dashed lines) shown have been omitted.

Refinement

Refinement on F^2	$w = 1/[\sigma^2(F_o^2) + (0.03P)^2 + 0.6P]$
$R[F^2 > 2\sigma(F^2)] = 0.037$	where $P = (F_o^2 + 2F_c^2)/3$
$wR(F^2) = 0.094$	$(\Delta/\sigma)_{max} < 0.001$
$S = 1.05$	$\Delta\rho_{max} = 0.19$ e Å^{-3}
2118 reflections	$\Delta\rho_{min} = -0.18$ e Å^{-3}
326 parameters	
All H-atom parameters refined	

Table 1
Selected geometric parameters (Å, °).

O1—N1	1.419 (3)	N2—C13	1.477 (3)
N1—C1	1.292 (3)	N2—C7	1.484 (3)
C1—N1—O1	111.7 (2)	N2—C7—C8	113.1 (2)
C13—N2—C7	115.91 (16)	C12—C7—C8	110.0 (2)
N1—C1—C6	116.7 (2)	N2—C13—C18	111.0 (2)
N1—C1—C2	127.0 (2)	N2—C13—C14	108.67 (18)
C6—C1—C2	116.3 (2)	C18—C13—C14	109.7 (2)
N2—C7—C12	109.04 (18)		

Table 2
Hydrogen-bond geometry (Å, °).

$D-H\cdots A$	$D-H$	$H\cdots A$	$D\cdots A$	$D-H\cdots A$
O1—H1O···N2	0.98 (5)	1.85 (5)	2.818 (3)	167 (4)
N2—H2N···O1^i	0.90 (3)	2.62 (3)	3.509 (3)	167 (2)
N2—H2N···N1^i	0.90 (3)	2.59 (3)	3.451 (3)	160 (2)
C2—H21···O1^{ii}	1.04 (3)	2.71 (3)	3.686 (3)	155 (2)

Symmetry codes: (i) $x, y, z + 1$; (ii) $x, y, z - 1$.

All H atoms were located in a difference Fourier map and refined isotropically [C—H = 0.94 (3)–1.05 (4) Å].

Data collection: *SMART* (Bruker, 2003); cell refinement: *SAINT* (Bruker, 2003); data reduction: *SAINT*; program(s) used to solve structure: *SHELXTL* (Bruker, 2003); program(s) used to refine structure: *SHELXTL*; molecular graphics: *SHELXTL*; software used to prepare material for publication: *SHELXTL*.

The manuscript was prepared with the beta test version 1.0.0. of the program *publCIF* to be released by the IUCr and with the program *modiCIFer* to be released by the University of Wisconsin–Madison.

References

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