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Cyclohexanecarboxamide

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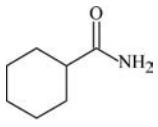
The title compound, $C_7H_{13}NO$, forms $R_2^2(8)$ N—H···O hydrogen-bonded dimers and *C*4 N—H···O-linked chains, which are further stabilized by a C—H···O interaction. The combination of these interactions results in a hydrogen-bonded network parallel to (100), with a motif that can be described by the secondary graph set $R_6^4(16)$. The existence of the same hydrogen-bonding motif in 1-phenylcyclopentanecarboxamide and 1-(2-bromophenyl)cyclohexanecarboxamide [Lemmerer & Michael (2008). *CrystEngComm*, **10**, 95–102 indicates that replacing the H atom on position 1 with a more bulky group does not necessarily disrupt the observed hydrogen-bonding pattern. The presence of a C—H···O interaction to stabilize the $R_6^4(16)$ network does, however, seem to be required. In addition, the title compound is isomorphous with a previously published structure of cyclopentanecarboxamide [Winter *et al.* (1981). *Acta Cryst.* B**37**, 2183–2185].

Comment

The hydrogen-bonding capabilities of amides have been extensively researched (Taylor *et al.*, 1984; Leiserowitz & Schmidt, 1969), and they have been exploited directly, or as precursors, in crystal engineering and the pharmaceutical industry (Reddy *et al.*, 2006), to name but a few. In addition, a systematic study of the effect of different 2,6-disubstitution on the structure of phenylamides and their consequent thermal behaviour has been published (Omondi *et al.*, 2005), as well as a similar structural study of a series of slightly more complex 1-arylcycloalkanecarboxamides (Lemmerer & Michael, 2008). In a study of symmetric and asymmetric imides and their polymorphs, the title compound, (I), was synthesized as a precursor for imide synthesis.



(I)

Compound (I) crystallizes in the space group *C*2/*c* with one molecule in the asymmetric unit (Fig. 1). The molecule adopts a chair conformation in which the amide group is rotated to be almost perpendicular to the cyclohexyl ring; atom N1 is orientated such that it lies almost eclipsed relative to atom H2, the N1—C1—C2—H2 torsion angle being −2.2°.

The structure of (I) contains two distinctive types of N—H···O hydrogen bonds. One of these [N1—H1*A*···O1^{ii}; symmetry code: (ii) $-x+\frac{1}{2}, -y+\frac{3}{2}, -z+1$; Fig. 2 and Table 1] is a hydrogen bond between two amide molecules to form an $R_2^2(8)$ dimer (Etter *et al.*, 1990; Bernstein *et al.*, 1995), with the molecules related to each other through a centre of inversion, while the other [N1—H1*B*···O1^{iv}; symmetry code: (iv) $x, -y+1, z+\frac{1}{2}$; Fig. 2 and Table 1] is an interaction along the *c* axis to form a *C*4 hydrogen-bonded chain, in which the molecules are related to each other by a *c*-glide plane. This *C*4 chain is further stabilized by a C—H···O interaction (C2—H2···O1^{iv}; Fig. 2 and Table 1). The combination of the two

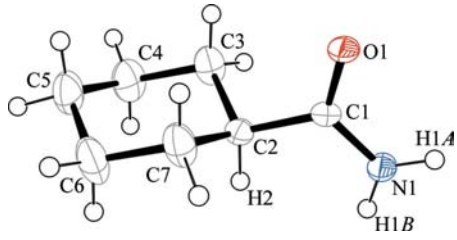


Figure 1
The molecular structure of (I), showing the atomic numbering scheme. Displacement ellipsoids are drawn at the 50% probability level.

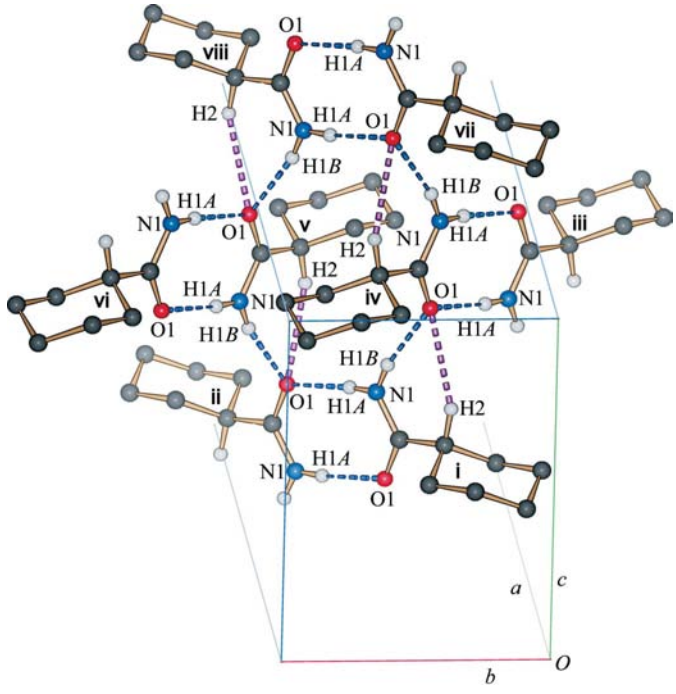


Figure 2
The intermolecular N—H···O and C—H···O hydrogen-bonding network (dashed lines) in the structure of (I), where molecules are connected to each other to form dimers and chains. The combination of these results in a hydrogen-bonded sheet running parallel to (100). H atoms not involved in these interactions have been omitted for clarity. [Symmetry codes: (i) x, y, z; (ii) $-x+\frac{1}{2}, -y+\frac{3}{2}, -z+1$; (iii) $-x+\frac{1}{2}, y-\frac{1}{2}, -z+\frac{3}{2}$; (iv) $x, -y+1, z+\frac{1}{2}$; (v) $-x+\frac{1}{2}, y+\frac{1}{2}, -z+\frac{3}{2}$; (vi) $x, -y+2, z+\frac{1}{2}$; (vii) $x, y, z+1$; (viii) $-x+\frac{1}{2}, -y+\frac{3}{2}, -z+2$.]

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N—H···O interactions results in the hydrogen-bonded dimers being almost perpendicular to each other [C1—C2···C2^{iv}—C1^{iv} = −90.99 (16)°] and in the formation of a hydrogen-bonded network parallel to (100) and centred at x = 0.25. The combination of the two hydrogen bonds also results in a motif that can be described by the secondary graph set $R_6^4(16)$.

A search of the Cambridge Structural Database (CSD; Version 5.30, November 2008 release; Allen, 2002) led to the discovery of the isomorphous structure of cyclopentanecarboxamide (CSD refcode BARFEF; Winter *et al.*, 1981), which has an identical hydrogen-bonding pattern despite the presence of disorder in the five-membered ring. The $R_6^4(16)$ motif can also be found in the structures of 1-phenylcyclopentanecarboxamide and 1-(2-bromophenyl)cyclohexanecarboxamide, indicating that replacing atom H2 with a more bulky group does not necessarily disrupt this hydrogen-bond pattern (Lemmerer & Michael, 2008). However, a C—H···O interaction does seem to be required as it is present in all four structures. In the case of 1-phenylcyclopentanecarboxamide and 1-(2-bromophenyl)cyclohexanecarboxamide, the C—H···O interaction occurs between the amide O atom and one of the ring CH_2 groups.

Experimental

The title compound was prepared as described by Lumsden (1905). The product was recrystallized from ethanol using a slow evaporation technique at room temperature, giving a 71% yield of colourless plate-like crystals [m.p. 458–460 K (literature value 459–460.5 K; McElvain & Starn, 1955)]. ^{1}H NMR (300 MHz, $CDCl_3$, Me_4Si): δ 5.69 (1H, *s*, N—H), 5.52 (1H, *s*, N—H), 2.15 (1H, *tt*, J = 3.5 and 11.6 Hz, H1), 1.93–1.69 (5H, *m*, H2, H3, H4, H5, H6), 1.48–1.30 (5H, *m*, H2, H3, H4, H5, H6); ^{13}C NMR (75 MHz, $CDCl_3$, Me_4Si): δ 178.7 (C6), 44.8 (C1), 297 (C2, C6), 25.7 (C3, C5), 25.6 (C4); ATR–IR: ν_{max} 3337 (*m*, *b*, N—H), 3154 (*m*, *b*, N—H), 2927 (*m*, *sh*, C—H), 2851 (*m*, *sh*, C—H), 1635 (*s*, C═O), 1428 (*s*, C—N), 1344 (*w*, *sh*), 1285 (*m*, *sh*), 1230 (*m*, *sh*), 1154 (*m*, *sh*), 666 (*s*) cm^{-1}.

Crystal data

$C_7H_{13}NO$
M_r = 127.18
Monoclinic, $C2/c$
a = 24.624 (3) Å
b = 6.6934 (9) Å
c = 9.4030 (13) Å
β = 102.088 (3)°
V = 1515.4 (3) Å^3
Z = 8
Mo $K\alpha$ radiation
μ = 0.07 mm^{-1}
T = 173 K
0.48 × 0.37 × 0.10 mm

Data collection

Bruker APEXII CCD area-detector diffractometer
9664 measured reflections
1828 independent reflections
1440 reflections with $I > 2\sigma(I)$
R_{int} = 0.068

Refinement

$R[F^2 > 2\sigma(F^2)]$ = 0.056
$wR(F^2)$ = 0.143
S = 1.05
1828 reflections
82 parameters
H-atom parameters constrained
$\Delta\rho_{max}$ = 0.28 e Å^{-3}
$\Delta\rho_{min}$ = −0.16 e Å^{-3}

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

D—H···A	D—H	H···A	D···A	D—H···A
N1—H1A···O1^{ii}	0.88	2.06	2.9394 (16)	175
N1—H1B···O1^{iv}	0.88	1.99	2.8549 (15)	166
C2—H2···O1^{iv}	1.00	2.56	3.4283 (17)	145

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

All H atoms were positioned geometrically and allowed to ride on their parent atoms [C—H = 1.00 (CH) or 0.99 Å (CH_2), N—H = 0.88 Å (NH_2) and U_{iso}(H) = 1.2U_{eq}(C,N)].

Data collection: *APEX2* (Bruker, 2005); cell refinement: *APEX2*; data reduction: *SAINT* (Bruker, 2005); program(s) used to solve structure: *SHELXS97* (Sheldrick, 2008); program(s) used to refine structure: *SHELXL97* (Sheldrick, 2008); molecular graphics: *ORTEP-3 for Windows* (Farrugia, 1997) and *SCHAKAL99* (Keller, 1999); software used to prepare material for publication: *WinGX* (Farrugia, 1999) and *PLATON* (Spek, 2009).

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Supplementary data for this paper are available from the IUCr electronic archives (Reference: FG3132). Services for accessing these data are described at the back of the journal.

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