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($3R^*$,$4R^*$)-3-Isopropyl-4-phenylazetidin-2-one at 150 K

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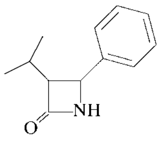
The title compound, $C_{12}H_{15}NO$, crystallized in the centrosymmetric space group *C*2/*c* with one molecule as the asymmetric unit. There is a single conventional N—H···O hydrogen bond, with a donor–acceptor distance of 2.912 (1) Å, which forms an $R_2^2(8)$ cyclic dimer about a center of symmetry. There is a single significant intermolecular C—H···O interaction, which also forms an $R_2^2(8)$ cyclic dimer about a center of symmetry. Taken together, these interactions form chains propagating along [110]. Structural comparisons are made with another β-lactam, (1′*R**,3*R**,4*S**)-3-(1′-hydroxyethyl)-4-phenylazetidin-2-one.

Comment

This report is one of a series on hydrogen bonding and C—H···O interactions in organic solids. The title compound, (I), crystallized in the centrosymmetric space group *C*2/*c* with one molecule as the asymmetric unit. The refined molecule and the labeling scheme are given in Fig. 1. A single hydrogen bond and a single significant intermolecular C—H···O interaction (Taylor & Kennard, 1982; Steiner & Desiraju, 1998) are present in this structure. The geometric parameters of these are given in Table 2. The results of basic first- and second-level graph-set analysis (Bernstein *et al.*, 1995) involving these interactions, labelled *a* and *b* for this purpose in the order of their appearance in Table 2, are given in Table 3. At the first level, each of the two interactions forms a cyclic dimer, $R_2^2(8)$, about a center of symmetry. The second-level chain propagates along [110]. Each molecule is linked by these interactions directly to two others. The resulting chains of molecules fall into two sets, with one comprising molecules of space-group symmetry types 1, 3, 5, and 7, and the other, symmetry types 2, 4, 6, and 8. The packing diagram, Fig. 2, shows a portion of a chain, in which by symmetry all four- and six-membered best-fit ring planes are, respectively, parallel.



(I)

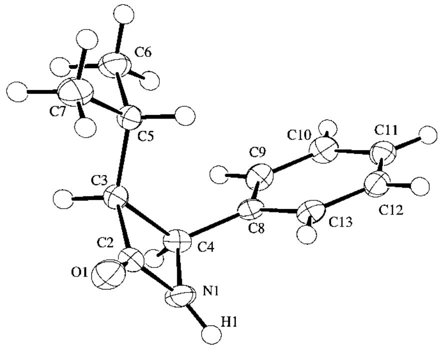


Figure 1
*ORTEP*II (Johnson, 1976) drawing of (I) showing the labeling scheme. Displacement ellipsoids are drawn for 50% probability for all non-H atoms; spheres of arbitrary small radius depict H atoms.

The four-membered ring (N1/C2–C4) of (I) is only roughly planar, the maximum deviation of any of its atoms from the best-fit plane describing them being 0.023 (1) Å, while the average deviation is 0.020 (2) Å. In contrast, the four-membered ring N1/C2–C4 (present labeling) of the quite similar molecule of (1′*R**,3*R**,4*S**)-3-(1′-hydroxyethyl)-4-phenylazetidin-2-one (hereafter, HEPA), determined at room temperature (Burnett *et al.*, 1985), is very nearly planar, with corresponding deviations 0.004 (2) and 0.003 (1) Å, respectively. In (I) and HEPA, the maximum deviations of ring atoms from the best-fit phenyl planes are 0.006 (1) and 0.006 (3) Å. The dihedral angle between the best-fit planes of the four-membered ring and the phenyl ring in (I) is 69.6 (1)°; in HEPA, this angle is 72.7°. Also, in (I), the dihedral angle between phenyl rings not required by symmetry to be parallel is 48.3 (1)°. These features are apparent in the packing diagram, Fig. 2. In both (I) and HEPA, the angular sum at C2 is 360° within less than 1 s.u. in each study, establishing the absence of pyramidal character at C2 in these molecules.

Selected bond distances and angles of (I) are given in Table 1. All distances and angles fall within normal limits. For comparisons with (I), HEPA appears to be quite satisfactory,

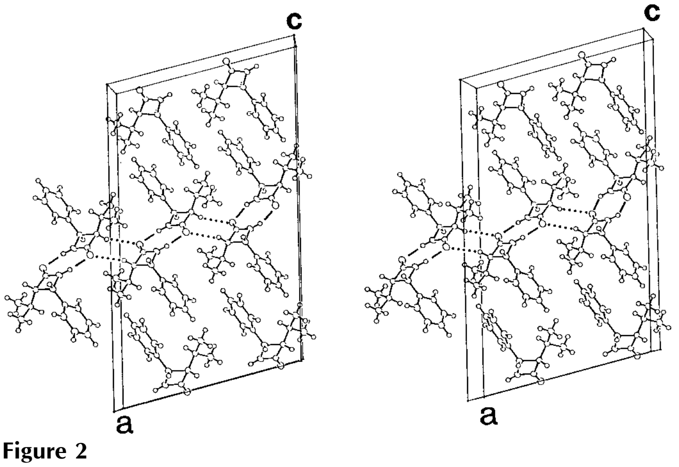


Figure 2
*ORTEP*II (Johnson, 1976) packing diagram of (I) viewed down the *b* axis. Displacement ellipsoids are drawn for 50% probability for all non-H atoms; spheres of arbitrary small radius depict H atoms. Hydrogen bonds are represented by dashes and C—H···O interactions by dotted lines.

though the temperature difference must be noted. For the N1/C2–C4 rings, perhaps the most interesting molecular feature, there is very good agreement in both (intra-ring) distances and angles; upon ordering the two sets of four distances and four angles by magnitude, corresponding distances and angles occur in precisely the same order in (I) and HEPA. Moreover, the mean differences between corresponding distances and angles in the four-membered rings of these two molecules are 0.010 Å and 0.3°, while the s.u.'s are 0.001 Å and 0.1° for (I), and 0.003 Å and 0.2° for HEPA. In (I), the closest intermolecular approaches, excluding pairs of atoms involved in the hydrogen bonding or the tabulated C—H···O interaction, are between C2 and $\mathrm{H1^i}$ [symmetry code: (i) $\frac{3}{2}-x, \frac{1}{2}-y, 1-z$] and fall short of the corresponding Bondi (1964) van der Waals radius sum by 0.06 Å.

Experimental

Compound (I) was obtained as an off-white crystalline powder from a sample in Dr D. J. Hart's chemical collection. A solution of this material in ether produced suitable crystals upon slow evaporation at room temperature. A synthesis is described by Burnett *et al.* (1986).

Crystal data

$C_{12}H_{15}NO$
$M_r = 189.26$
Monoclinic, $C2/c$
$a = 14.7505\ (9)$ Å
$b = 5.7579\ (3)$ Å
$c = 24.941\ (2)$ Å
$\beta = 104.358\ (3)^\circ$
$V = 2052.1\ (2)$ Å^3
$Z = 8$

$D_x = 1.225$ Mg m^{-3}
Mo $K\alpha$ radiation
Cell parameters from 5017 reflections
$\theta = 2.53$–27.46°
$\mu = 0.078$ mm^{-1}
$T = 150$ K
Cut plate, colorless
0.38 × 0.35 × 0.06 mm

Data collection

Nonius KappaCCD diffractometer
ω scans with κ offsets
5017 measured reflections
2274 independent reflections
1896 reflections with $I > 2\sigma_I$

$R_{\mathrm{int}} = 0.026$
$\theta_{\mathrm{max}} = 27.46^\circ$
$h = -18 \rightarrow 18$
$k = -7 \rightarrow 4$
$l = -32 \rightarrow 14$

Refinement

Refinement on F^2
$R(F) = 0.042$
$wR(F^2) = 0.088$
$S = 1.95$
2274 reflections
131 parameters

H atoms treated by a mixture of independent and constrained refinement
$w = 1/[\sigma^2_{\mathrm{cs}} + (0.010I)^2]$
$(\Delta/\sigma)_{\mathrm{max}} < 0.001$
$\Delta\rho_{\mathrm{max}} = 0.28$ e Å^{-3}
$\Delta\rho_{\mathrm{min}} = -0.26$ e Å^{-3}

Table 1
Selected geometric parameters (Å, °).

O1—C2	1.222 (1)	C3—C4	1.583 (1)
N1—C2	1.346 (1)	C3—C5	1.526 (1)
N1—C4	1.468 (1)	C4—C8	1.504 (2)
C2—C3	1.527 (1)		
C2—N1—C4	96.01 (8)	C2—C3—C4	84.60 (7)
O1—C2—N1	131.40 (9)	C2—C3—C5	118.33 (9)
O1—C2—C3	135.63 (9)	N1—C4—C3	86.24 (7)
N1—C2—C3	92.97 (8)	N1—C4—C8	115.04 (8)

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

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
$\mathrm{N1—H1\cdots O1^{i}}$	0.89 (1)	2.06 (1)	2.912 (1)	161
$\mathrm{C3—H3\cdots O1^{ii}}$	0.98	2.71	3.634 (1)	158

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

Table 3
Basic first- and second-level graph-set descriptors involving interactions designated *a*–*b* in the order given in Table 2.

	a	*b*
a	$R_2^2(8)$	$C_2^1(6)$
b		$R_2^2(8)$

Data collection: *COLLECT* (Nonius, 1999); cell refinement: *DENZO-SMN* (Otwinowski & Minor, 1997); data reduction: *DENZO-SMN*; program(s) used to solve structure: *SHELXS*86 (Sheldrick, 1985); program(s) used to refine structure: *TEXSAN* (Molecular Structure Corporation, 1995); molecular graphics: *ORTEP*II (Johnson, 1976); software used to prepare material for publication: *TEXSAN* and *PLATON* (Spek, 1990).

I thank Dr D. J. Hart for providing the sample of (I). I acknowledge with pleasure my use of the departmental X-ray crystallographic facility, which is supervised by staff crystallographer Dr J. C. Gallucci.

Supplementary data for this paper are available from the IUCr electronic archives (Reference: DE1130). Services for accessing these data are described at the back of the journal.

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