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cis-7-Azabicyclo[4.2.0]octan-8-one

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Key indicators

Single-crystal X-ray study
T = 293 K
Mean σ(C–C) = 0.002 Å
Disorder in main residue
R factor = 0.052
wR factor = 0.185
Data-to-parameter ratio = 13.3

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

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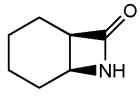
Under the strain of the quasi-planar β-lactam moiety, the rigid cyclohexane ring in the title compound, $C_7H_{11}NO$, can assume either a flexible boat form (77%) or a flexible half-chair form (23%). These two forms can be present simultaneously. The racemic crystals, isostructural with the *cis*-6-azabicyclo[3.2.0]-heptan-7-one homologue [Reck *et al.* (1990). *Acta Cryst.* C**46**, 720–722], are characterized by N—H···O═C hydrogen bonds that are formed along the screw axes of the similar monoclinic unit cell.

Comment

The alicyclic β-amino acids and their β-lactam forms are important intermediates in the syntheses of saturated and partly saturated heterocyclic compounds that have been studied from a pharmaceutical aspect (Fülöp *et al.*, 1998). They occur in antibiotics and they have been introduced into modified peptides in order to increase their stability, *i.e.* biological activity (Fülöp, 2001). The racemic crystals of alicyclic (cyclopentane, cyclohexane, cycloheptane and cyclo-octane) β-amino acids with the common space group $P\overline{1}$ are isostructural (Kálmán *et al.*, 1993). This phenomenon can be attributed to the robustness of their close packing due to three hydrogen bonds of the N—H···O═C type formed between the zwitterionic molecules (Fábián *et al.*, 2004). The corresponding β-lactams contain only one hydrogen bond, which invariably develops homochiral helices in space group $P2_1/c$ or $P2_12_12_1$.



(2)

The crystals of the cyclopentane (1) (Reck *et al.*, 1990) and cyclohexane (2) homologues are isostructural (Fig. 1), but their packing is different from that of the cycloheptane homologue (3). In (1) and (2), the space group is $P2_1/c$, and in (3) it is $P2_12_12_1$ (Argay *et al.*, 2004). In the racemic crystals of (1) and (2), the enantiomers are organized in homochiral helices with antiparallel orientation. A similar close packing is exhibited by 4-methyl-10-aza-*trans*-bi-cyclo[7.2.0]undeca-2,5,7-trien-11-one, (4) (Paquette *et al.*, 1973). In these crystal structures, hallmarked by the common β-lactam ring, antiparallel helices are formed along the similarly short *b* axes: 6.147 (1), 6.474 (2) and 5.706 (4) Å; the differences in the cycloalkane (five-, six- and nine-membered) rings account for the different *a* and *c* axes. The parameters of the N—H···O bond in (2) are $d(D\cdots A)$ = 2.878 (2) Å, $d(H\cdots A)$ = 2.03 Å, $<(D—H\cdots A)$ = 171°. The crystals of (1) and (2) display a high degree of isostructurality (Π = 0.045 and

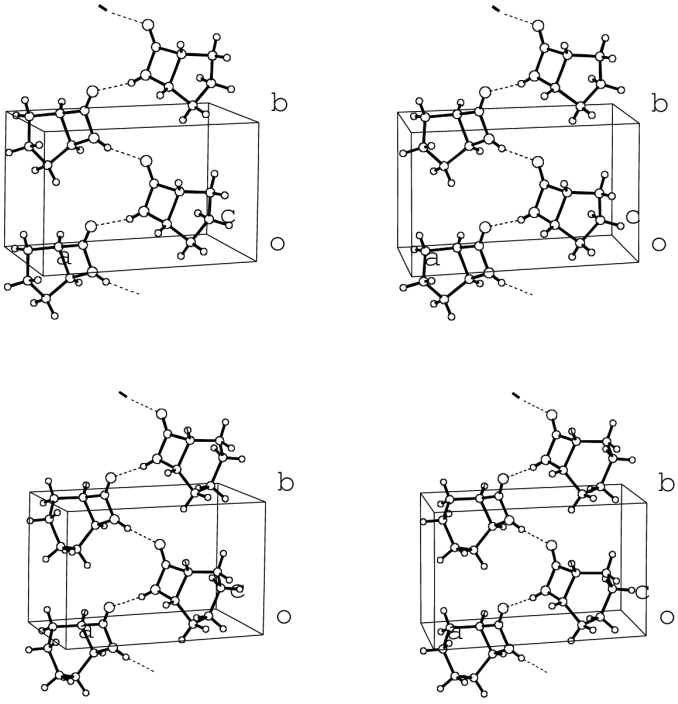


Figure 1
Stereoscopic views of the crystal structures of the cyclopentane homologue (Reck *et al.*, 1990) and the dominant conformer of the title compound, viewed along the *c* axis, showing their isostructurality.

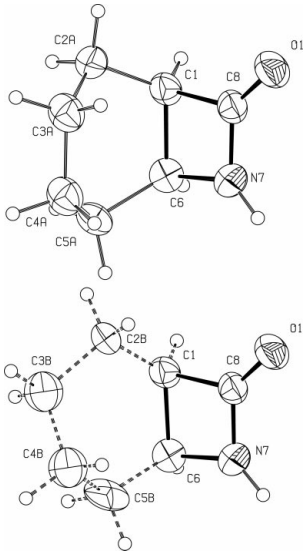


Figure 2
A perspective view of the disordered molecules, with displacement ellipsoids drawn at the 30% probability level. Only non-H atoms are labelled.

I_v = 82%; Fábián & Kálmán, 1999), while (4), with its much larger cycloalkane ring, is merely homostructural (Kálmán & Párkányi, 1997) with them. The rigid β-lactam moiety *cis*-fused to the cycloalkane ring in (1) causes the flexible cyclopentane ring to assume an envelope shape (pseudorotation; Altona *et al.*, 1968); the molecule is almost perfectly bisected by a non-centrosymmetric mirror plane (C_s).

In the title compound, (2), the effect of the β-lactam moiety twists the cyclohexane ring from the rigid chair conformation into an almost perfect boat shape (puckering parameters: Q = 0.65 Å, φ = 243° and θ = 92°; Cremer & Pople, 1975), (Fig. 2*a*). However, as revealed by the positional disorder of atoms C2, C3, C4 and C5, 23% of the molecules assume another canonical form, a likewise flexible half-chair (Fig. 2*b*) with puckering parameters Q = 0.55 Å, φ = 157° and θ = 53°. The predominant boat conformer is characterized by a mirror plane which passes though C2*A* and C5*A*, while the half-chair form has a twofold (C_2) axis which bisects the C1—C6 and C3*B*—C4*B* bonds.

Experimental

Bestian *et al.* (1968) reported the synthesis of the title compound, (2), under the name *cis*-3,4-tetramethylene-azetidinone, with m.p. 330–331 K. The product that we prepared and crystallized from ethyl acetate melts at 329–330 K.

Crystal data

$C_7H_{11}NO$
M_r = 125.17
Monoclinic, $P2_1/c$
a = 11.333 (1) Å
b = 6.474 (1) Å
c = 10.181 (1) Å
β = 112.00 (1)°
V = 692.59 (14) Å^3
Z = 4
D_x = 1.200 Mg m^{-3}
Cu $K\alpha$ radiation
Cell parameters from 25 reflections
θ = 29.3–32.3°
μ = 0.64 mm^{-1}
T = 293 (2) K
Prism, colourless
0.50 × 0.35 × 0.30 mm

Data collection

Enraf–Nonius CAD-4 diffractometer
ω–θ scans
Absorption correction: ψ scan (North *et al.*, 1968)
T_{min} = 0.766, T_{max} = 0.822
1554 measured reflections
1433 independent reflections
1256 reflections with $I > 2\sigma(I)$
R_{int} = 0.010
θ_{max} = 75.6°
$h = -14 \rightarrow 7$
$k = -1 \rightarrow 8$
$l = -12 \rightarrow 12$
3 standard reflections
frequency: 60 min
intensity decay: 8%

Refinement

Refinement on F^2
$R[F^2 > 2\sigma(F^2)]$ = 0.052
$wR(F^2)$ = 0.185
S = 1.30
1433 reflections
108 parameters
H-atom parameters constrained
$w = 1/[\sigma^2(F_o^2) + (0.101P)^2 + 0.05P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} < 0.001$
$\Delta\rho_{max}$ = 0.23 e Å^{-3}
$\Delta\rho_{min}$ = −0.15 e Å^{-3}
Extinction correction: *SHELXL*97
Extinction coefficient: 0.012 (3)

Table 1
Selected geometric parameters (Å).

C1—C2*A*	1.519 (4)	C4*A*—C5*A*	1.519 (4)
C1—C8	1.524 (2)	C5A—C6	1.521 (4)
C1—C6	1.555 (2)	C6—N7	1.4710 (19)
C2*A*—C3*A*	1.516 (5)	N7—C8	1.334 (2)
C3*A*—C4*A*	1.519 (4)	C8—O1	1.2232 (19)

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

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
N7—H7···O1^{i}	0.86	2.03	2.8781 (17)	171

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

All H atoms were positioned geometrically and allowed to ride on their parent atoms, with C—H = 0.97–0.98 Å and N—H = 0.86 Å, and U_{iso}(H) = 1.3U_{eq}(C/N) for all H atoms.

Data collection: *CAD*-4 *EXPRESS* (Enraf–Nonius, 1992); cell refinement: *CAD*-4 *EXPRESS*; data reduction: *XCAD*4 (Harms, 1996); program(s) used to solve structure: *SHELXS*97 (Sheldrick, 1997); program(s) used to refine structure: *SHELXL*97 (Sheldrick, 1997); molecular graphics: *ORTEP*-3 (Farrugia, 1997) and *RPluto* in CSD (Allen, 2002); software used to prepare material for publication: *SHELXL*97.

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