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cis-8-Azabicyclo[5.2.0]nonan-9-one

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Key indicators

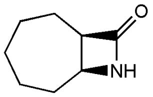
Single-crystal X-ray study
T = 293 K
Mean σ(C–C) = 0.005 Å
R factor = 0.066
wR factor = 0.227
Data-to-parameter ratio = 10.3

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

The title compound, $C_8H_{13}NO$, with two chiral centres in the *cis*-junction between the β-lactam and cycloheptane rings, crystallizes with an orthorhombic unit cell. The strain of the β-lactam moiety forces the cycloheptane ring to assume a chair conformation characterized by C_s symmetry, which is less stable by 1.4 kcal mol^{-1} than the twist-chair form, built up around a twofold axis. The homochiral helices, maintained by N—H···O=C hydrogen bonds, are organized in antiparallel mode around the respective screw axes of the non-centrosymmetric space group $P2_12_12_1$, which means that the enantiomers resolve in crystal conglomerates.

Comment

Similarly to its effect in the cyclohexane-fused homologue (Argay *et al.*, 2004), the narrow and nearly planar β-lactam moiety in the title compound, (3), determines the conformation of the cycloheptane ring.



(3)

The seven-membered ring assumes the uncommon chair conformation (Fig. 1), with an asymmetry parameter ΔC_s = 2.2° (Duax *et al.*, 1976), which means that the endocyclic torsion angles barely differ from the calculated values (Hendrickson, 1967*a*): calc.: 63.8, −83.5, 66.1, 0, −66.1, 83.5, −63.8° exp.: 63.3 (4), −83.5 (4), 66.5 (4), 0 (1), −65.4 (4), 80.4 (4), −61.9 (4)°. In the corresponding β-amino acid (Fábián *et al.*, 2004), the endocyclic torsion angles indicate a distorted twist-chair form, in particular at C1 which bears the carboxylate group. As in the title compound, the strain exerted by the rigid cyclobutane moiety in *cis*-6,7,8,9-hexahydro-5*H*-benzo[3.4]cyclobuta[1.2]cyclohepten-1-ol (Courtois *et al.*, 1985) on the cycloheptane ring stabilizes the chair form with a low asymmetry parameter, ΔC_s = 1.0° (Duax *et al.*, 1976), while the flexible oxazine ring in 2-*p*-chlorophenyl-*cis*-4,5-pentamethylene-4,5-dihydro-1,3-oxazine (Ribár *et al.*, 1977) does not hinder the presence of the most stable (E_0 = 6.0 kcal mol^{-1}) half-chair conformation of the cycloheptane ring Hendrickson (1967*a*). It also has a low asymmetry parameter, ΔC_2 = 3.1°. It is noteworthy that the co-existence of the most favourable twist-chair conformation (82%) and the 'transient' chair form (18%), with an energy maximum on the pseudo-rotation itinerary (Hendrickson, 1961, 1967*b*), was also observed in *cis*-2-hydroxycycloheptanecarboxylic acid (Kálmán *et al.*, 2002). The di-equatorial orientation of the β-lactam moiety to the cycloheptane

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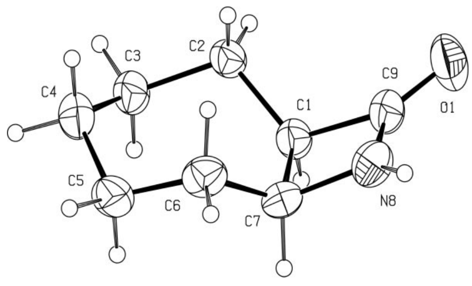

Figure 1
A perspective view of the molecule, with displacement ellipsoids drawn at the 30% probability level. Only non-H atoms are labelled.

ring may account for the different close-packing arrangement from (1) and (2).

In contrast with the cycloheptane (1) (Reck *et al.*, 1990) and cylohexane (2) (Argay *et al.*, 2004) homologues, which are isostructural in space group $P2_1/c$ (Kálmán *et al.*, 1993), the crystals of the title compound (3), with a short *a* axis of 6.683 (1) Å [the monoclinic *b* axis is 6.147 Å in (1), and 6.474 Å in (2)], hallmarked by the antiparallel helices of the homochiral molecules (Fig. 2), held together by N—H···O=C hydrogen bonds (Table 2), reveal spontaneous resolution. Brock & Dunitz (1994) considered that the incidence of spontaneous resolution shown by the title compound relative to its cyclopentane and cyclohexane homologues cannot be predicted. Insufficient observations are available at present to shed light on such phenomena.

Experimental

The preparation of the title compound (m.p. 329 K) was reported by Pihlaja *et al.* (1986). The present work was based on single crystals obtained from diisopropyl ether with m.p. 328–329 K.

Crystal data

$C_8H_{13}NO$
$M_r = 139.19$
Orthorhombic, $P2_12_12_1$
$a = 6.683$ (1) Å
$b = 9.166$ (1) Å
$c = 12.696$ (2) Å
$V = 777.71$ (19) Å^3
$Z = 4$
$D_x = 1.189$ Mg m^{-3}

Cu Kα radiation
Cell parameters from 25 reflections
$\theta = 24.3$–$25.5°$
$\mu = 0.62$ mm^{-1}
$T = 293$ (2) K
Prism, colourless
$0.35 \times 0.20 \times 0.15$ mm

Data collection

Enraf–Nonius CAD-4 diffractometer
ω–θ scans
Absorption correction: ψ scan (North *et al.*, 1968)
$T_{\min} = 0.892$, $T_{\max} = 0.910$
1863 measured reflections
941 independent reflections
842 reflections with $I > 2\sigma(I)$

$R_{\rm int} = 0.017$
$\theta_{\max} = 74.9°$
$h = -8 \rightarrow 8$
$k = -11 \rightarrow 11$
$l = -15 \rightarrow 15$
3 standard reflections
frequency: 60 min
intensity decay: 16%

Refinement

Refinement on F^2
$R[F^2 > 2\sigma(F^2)] = 0.066$
$wR(F^2) = 0.227$
$S = 1.98$
941 reflections
91 parameters

H-atom parameters constrained
$w = 1/[\sigma^2(F_o^2) + (0.1P)^2]$
where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{\max} < 0.001$
$\Delta\rho_{\max} = 0.23$ e Å^{-3}
$\Delta\rho_{\min} = -0.28$ e Å^{-3}

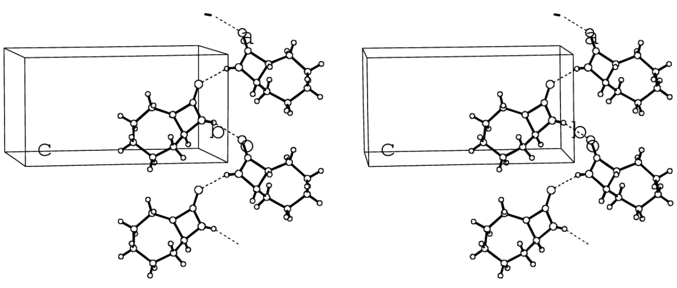

Figure 2
Stereoscopic packing diagram, showing hydrogen bond helices of the homochiral molecules.

Table 1
Selected geometric parameters (Å).

C1—C2	1.513 (4)	C5—C6	1.511 (5)
C1—C7	1.552 (4)	C6—C7	1.505 (5)
C1—C9	1.537 (4)	C7—N8	1.478 (4)
C2—C3	1.521 (4)	N8—C9	1.340 (5)
C3—C4	1.524 (5)	C9—O1	1.210 (4)
C4—C5	1.530 (5)		

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

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
N8—H8···O1$^{\rm i}$	0.86	2.12	2.929 (4)	157

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

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 $U_{\rm iso}$(H) = $1.3U_{\rm eq}$(C/N) for all H atoms. The absolute configuration could not be established by anomalous dispersion effects in the diffraction measurements on the crystal. An arbitrary choice of enantiomer has been made, and Friedel pairs were merged.

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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