C10	0.4024 (2)	0.2193 (2)	0.03446 (7)	0.0590 (7)
C11	0.3484 (3)	0.1142 (2)	0.01876 (9)	0.0704 (8)
C12	0.2450 (3)	0.0404 (3)	0.03236 (10)	0.0810 (9)
C13	0.1952 (3)	0.0699 (3)	0.06187 (11)	0.0825 (9)
C14	0.2491 (2)	0.1753 (2)	0.07741 (9)	0.0681 (7)
F1†	0.5770 (6)	0.3274 (8)	0.1594 (3)	0.236 (6)
F1‡	0.5684 (6)	0.3173 (5)	0.1372 (3)	0.145 (5)
F2†	2/3	1/3	0.1069 (3)	0.145 (4)
F2‡	2/3	1/3	0.1905 (4)	0.228 (9)
B1	2/3	1/3	0.1498 (2)	0.083 (2)

† Site occupancy = 0.580 (12). ‡ Site occupancy = 0.420 (12).

Table 2. *Selected geometric parameters (Å, °)*

N1—C6	1.362 (3)	C2—C3	1.412 (3)
N1—C2	1.374 (3)	C3—C4	1.345 (4)
N1—C7	1.455 (3)	C4—C5	1.392 (4)
O2—C2	1.272 (2)	C5—C6	1.349 (4)
C6—N1—C2	122.4 (2)	N1—C2—C3	116.3 (2)
C6—N1—C7	119.4 (2)	C4—C3—C2	121.0 (3)
C2—N1—C7	118.1 (2)	C3—C4—C5	120.7 (3)
O2—C2—N1	117.8 (2)	C6—C5—C4	118.9 (3)
O2—C2—C3	125.8 (2)	C5—C6—N1	120.6 (3)
C6—N1—C7—C8	101.1 (2)	N1—C7—C8—C9	177.7 (2)
C2—N1—C7—C8	−75.6 (2)	O3—C8—C9—C14	1.1 (3)
N1—C7—C8—O3	−2.2 (3)	O3—C8—C9—C10	179.8 (2)

Data collection: *CAD-4 Software* (Enraf–Nonius, 1989). Cell refinement: *CAD-4 Software*. Data reduction: *SDP-Plus* (Frenz, 1985). Program(s) used to solve structure: *SHELXS96* (Sheldrick, 1996). Program(s) used to refine structure: *SHELXL93* (Sheldrick, 1993). Molecular graphics: *ORTEX* (McArdle, 1995). Software used to prepare material for publication: *SHELXL93*.

This work has been supported by The Netherlands Organization for Fundamental Research (NWO). The authors are also indebted to the Russian Foundation for Basic Research for covering the licence fee for the use of the Cambridge Structural Database (project 96-07-89187).

Supplementary data for this paper are available from the IUCr electronic archives (Reference: NA1311). Services for accessing these data are described at the back of the journal. A packing diagram has also been deposited.

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Acta Cryst. (1997). C**53**, 1911–1912

(−)-2-Azabicyclo[2.2.1]hept-5-en-3-one (Lactam)

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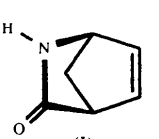
(*Received 22 April 1997; accepted 20 August 1997*)

Abstract

In the lactam structure, C_6H_7NO, molecules are linked by hydrogen bonds between N—H and C=O [N···O 2.885 (2) Å] to form ribbons parallel to the [100] 2_1 axis. The shortest intermolecular distances outside the ribbons are relatively large [C···O 3.535 (2) and C···C 3.600 (2) Å].

Comment

The (+) and (−) enantiomers of the title bicyclic lactam are key synthons for the preparation of carbocyclic nucleosides used as chemotherapeutic agents. It was discovered recently (Potter *et al.*, 1996) that the (±)-lactam crystallizes as a conglomerate; this finding induced us to focus on the possibility of resolving this compound by the entrainment method (Jacques, Collet & Wilen, 1981). An amazing observation was made during this entrainment process: under certain conditions, the crystallization of supersaturated solutions proceeds *via* a series of oscillations of their enantiomeric composition from (+) to (−), and conversely, until the final racemic equilibrium composition is achieved. To understand this behaviour, it seemed useful to elucidate the crystallographic structure of the enantiomer, (I).



(I)

The structure of (I) is organized in ribbons of molecules linked by hydrogen bonds between C=O and N—H [N···O 2.885 (2) Å]. These ribbons are parallel to [100] and the molecules are related by a 2_1 axis. In the plane perpendicular to the ribbons, their section has a herring-bone disposition. Outside the ribbons, the structure is relatively loose and the shortest intermolecular distances are large [C4···C5($-x$, $\frac{1}{2}+y$, $\frac{1}{2}-z$) 3.600 (2) and C7···O8($-x$, $y-\frac{1}{2}$, $\frac{1}{2}-z$) 3.535 (2) Å].

Studies of the possibility of layered crystal growth during the oscillating crystallization [alternate (+) and (−) layers containing the hydrogen-bonded ribbons] are in progress. Similar behaviour has been observed for helicene (Green & Knossow, 1981).

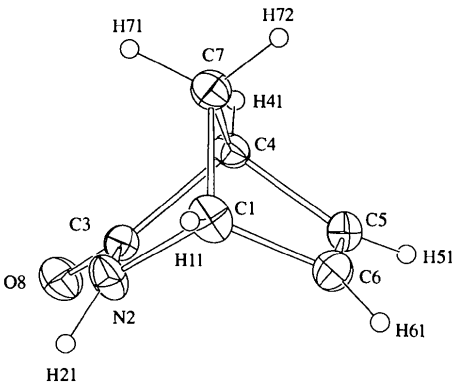


Fig. 1. The molecular structure of the title compound showing 50% probability displacement ellipsoids. H atoms are shown as circles of arbitrary size.

Experimental

Crystals of the lactam were obtained from a solution of the compound in methyl *tert*-butyl ether.

Crystal data

C_6H_7NO
M_r = 109.13
Orthorhombic
$P2_12_12_1$
a = 6.1050 (6) Å
b = 6.2520 (6) Å
c = 14.547 (1) Å
V = 555.2 (1) Å^3
Z = 4
D_x = 1.305 Mg m^{-3}
D_m not measured
Mo $K\alpha$ radiation
λ = 0.71073 Å
Cell parameters from 25 reflections
θ = 10–12°
μ = 0.09 mm^{-1}
T = 293 K
Parallelepiped
0.4 × 0.3 × 0.3 mm
Translucent white

Data collection

Enraf–Nonius CAD-4 diffractometer
ω scans
Absorption correction: none
1009 measured reflections
970 independent reflections
970 reflections with $I > 0$
θ_{max} = 29.96°
$h = 0 \rightarrow 8$
$k = 0 \rightarrow 8$
$l = 0 \rightarrow 20$
2 standard reflections every 100 reflections
intensity decay: 0.5%

Refinement

Refinement on F
R = 0.057
wR = 0.028
S = 3.274
970 reflections
95 parameters
$\Delta\rho_{max}$ = 0.26 e Å^{-3}
$\Delta\rho_{min}$ = −0.18 e Å^{-3}
Extinction correction: Zachariasen (1967)
Extinction coefficient: 5.3 (3) × 10^{-5}
Only coordinates of H atoms refined (U_{iso} = 0.06 Å^2)
$w = 1/\sigma^2(F)$
$(\Delta/\sigma)_{max}$ = 0.012
Scattering factors from *International Tables for X-ray Crystallography* (Vol. IV)
Absolute configuration: chosen arbitrarily

Table 1. *Fractional atomic coordinates and equivalent isotropic displacement parameters (Å^2)*

$U_{eq} = (1/3)\Sigma_i\Sigma_j U^{ij} a_i^* a_j^* \mathbf{a}_i.\mathbf{a}_j$.

	x	y	z	U_{eq}
C1	0.4162 (3)	0.9309 (3)	0.3692 (1)	0.053 (1)
N2	0.2880 (3)	1.0987 (3)	0.4161 (1)	0.0499 (9)
C3	0.0793 (3)	1.0797 (3)	0.39029 (9)	0.0376 (9)
C4	0.0794 (3)	0.9011 (3)	0.3190 (1)	0.0381 (8)
C5	0.1305 (4)	0.7004 (3)	0.3732 (1)	0.053 (1)
C6	0.3314 (4)	0.7171 (3)	0.4023 (1)	0.060 (1)
C7	0.3040 (3)	0.9406 (3)	0.2754 (1)	0.045 (1)
O8	−0.0804 (2)	1.1800 (2)	0.41885 (8)	0.0520 (7)

Table 2. *Selected geometric parameters (Å, °)*

C1—N2	1.476 (3)	C3—O8	1.232 (2)
C1—C6	1.513 (3)	C4—C5	1.514 (2)
C1—C7	1.529 (3)	C4—C7	1.532 (3)
N2—C3	1.333 (2)	C5—C6	1.302 (3)
C3—C4	1.523 (2)		
N2—C1—C6	107.4 (2)	C3—C4—C5	104.7 (1)
N2—C1—C7	98.5 (1)	C3—C4—C7	99.5 (1)
C6—C1—C7	99.6 (2)	C5—C4—C7	99.5 (1)
C1—N2—C3	108.3 (1)	C4—C5—C6	107.3 (2)
N2—C3—C4	104.9 (1)	C1—C6—C5	106.8 (2)
N2—C3—O8	128.0 (1)	C1—C7—C4	91.4 (1)
C4—C3—O8	127.1 (2)		

Data collection: *CAD-4 Software* (Enraf–Nonius, 1989). Cell refinement: *CAD-4 Software*. Data reduction: *DIFDAT*, *SORTRF* and *ADDREF* in *Xtal*3.0 (Hall & Stewart, 1990). Program(s) used to solve structure: *Xtal*3.0. Program(s) used to refine structure: *CRYLSQ* in *Xtal*3.0. Molecular graphics: *Xtal*3.0. Software used to prepare material for publication: *BONDLA* and *CIFIO* in *Xtal*3.0.

We thank Dr G. A. Potter for supplying the lactam sample.

Supplementary data for this paper are available from the IUCr electronic archives (Reference: FR1062). Services for accessing these data are described at the back of the journal.

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