The Structure of cis-Bicyclo[3.3.0]octane-3-spiro-5'-hydantoin*

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Abstract. $C_{10}H_{14}N_2O_2$, monoclinic, $C2/m$, $a = 10\cdot171$ (1), $b = 6\cdot600$ (1), $c = 15\cdot421$ (1) Å, $\beta = 100\cdot84$ (1)°, $Z = 4$, $D_x = 1\cdot261$, $D_m = 1\cdot27$ Mg m⁻³, $\mu(Cu\,K\alpha) = 0\cdot738$ mm⁻¹. The two cyclopentane rings have an envelope conformation. The molecules are held together by N–H···O bridges.

Introduction. The present determination continues investigations on the structure of heterocyclic spiro derivatives with pharmacological properties (Gonzalez Trigo, Avendaño & Martínez Moreno, 1974). The title compound was prepared by Dr Gonzalez Trigo and Dr C. Avendaño using the Strecker synthesis on *cis*-bicyclo[3.3.0]octan-3-one. NMR studies revealed that the cyclopentane rings have an envelope conformation and that $C(4')$ of the hydantoin ring is situated at the convex part of the bicyclic system. These facts were confirmed by the present analysis.

Cell parameters were obtained by a least-squares procedure from the settings of 25 reflexions measured on a four-circle diffractometer with Cu $K\alpha$ radiation. Intensities were collected on a Philips PW 1100 computer-controlled four-circle diffractometer operating in the θ–2θ scan mode with graphite-monochromated Cu $K\alpha$ radiation. 941 reflexions up to $\theta = 65°$ were measured. 51 reflexions with $I < 2\sigma(I)$ were omitted as unobserved, leaving 890 unique reflexions which were employed in the analysis. The data were corrected for Lorentz–polarization effects but not for absorption.

The dimensions of the crystal were $0\cdot13 \times 0\cdot20 \times 0\cdot35$ mm.

Systematic absences hkl with $h + k \neq 2n$ indicate space groups $C2/m$, Cm or $C2$. A centrosymmetric structure was suggested by normalized structure factor statistics. As $Z = 4$ the molecules must occupy special positions, which may be at a mirror plane, twofold axis or centre of symmetry. The structure was solved with *MULTAN* 74 (Main, Woolfson, Lessinger, Germain & Declercq, 1974) applied to 200 reflexions with $|E| > 1\cdot90$. All atoms other than H were located from the E map based on the phases obtained. The molecules were found to lie on mirror planes.

The atomic positions were refined by a full-matrix least-squares method with anisotropic temperature factors for non-hydrogen atoms, until R had dropped to $0\cdot10$. A difference synthesis then revealed all the H atoms. Refinement was continued with H atoms included with isotropic temperature factors. The quantity minimized was $\sum w(\Delta F)^2$. For the observed reflexions the final $R = 0\cdot080$ and $R_w = 0\cdot110$ where $R_w = (\sum w\Delta^2/\sum w|F_o|^2)^{1/2}$. Scattering factors were taken from *International Tables for X-ray Crystallography* (1974). The computations were made with XRAY 70 (Stewart, Kundell & Baldwin, 1970). Final parameters for non-hydrogen and H atoms are given in Tables 1 and 2.†

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† Lists of structure factors and anisotropic thermal parameters have been deposited with the British Library Lending Division as Supplementary Publication No. SUP 33920 (6 pp.). Copies may be obtained through The Executive Secretary, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

Table 1. *Final atomic positional parameters* ($\times 10^4$) *with e.s.d.'s in parentheses*

	x	y	z
N(1')	1635 (3)	0	819 (2)
C(2')	1931 (3)	0	12 (2)
N(3')	3320 (2)	0	121 (1)
C(4')	3915 (3)	0	986 (2)
C(3,5')	2803 (3)	0	1526 (2)
O(1)	1175 (2)	0	−701 (1)
O(2)	5113 (2)	0	1268 (1)
C(1)	3762 (4)	1167 (6)	2997 (2)
C(2)	2875 (3)	1832 (4)	2152 (1)
C(4)	3212 (8)	1827 (8)	3837 (2)
C(5)	2516 (13)	0	4059 (5)

Table 2. *Final H atom positional parameters* ($\times 10^3$) *and isotropic thermal parameters* (Å² $\times 10^3$) *with e.s.d.'s in parentheses*

	x	y	z	U_{iso}
H(1')	87 (7)	0	90 (4)	70
H(3')	373 (5)	0	−27 (3)	43
H(1)	466 (7)	211 (12)	280 (4)	143
H(21)	195 (6)	192 (10)	224 (3)	123
H(22)	281 (7)	316 (11)	173 (4)	145
H(41)	284 (6)	311 (11)	370 (4)	122
H(42)	416 (7)	250 (12)	444 (4)	150
H(51)	200 (11)	0	445 (7)	169
H(52)	165 (18)	0	359 (10)	224

Discussion. Bond lengths and angles are shown in Table 3. Fig. 1 displays the structural formula with the atomic labelling.

The molecule has a mirror plane which coincides with the mirror plane in the crystal perpendicular to *b*. In this plane are situated the hydantoin ring and the spiranic C(3,5') (Fig. 1). The hydantoin ring is consequently planar. Bond lengths and angles in this ring are similar to those found in *N*-methyltropane-3-spiro-5'-hydantoin (Smith-Verdier, Florencio & Garcia-Blanco, 1977) and in *N*-methylgranatanine-3-spiro-5'-hydantoin (Florencio, Smith-Verdier & Garcia-Blanco, 1978).

C(1), C(1)', C(2), C(2)' and C(1), C(1)', C(4), C(4)' of the two cyclopentane rings lie in planes perpendicular to the symmetry plane. C(3,5') of the first ring and the C(5) of the second deviate from their respective planes by −0·527 and −0·442 Å respectively; thus the two cyclopentane rings have an undistorted envelope conformation. The angle between the two rings is 119·3°, practically the same as in an idealized system.

C(4') of the hydantoin ring is situated in the convex part of the bicyclic system which results in more steric hindrance, in agreement with the studies carried out by Edward & Jitrangsri (1975) on cyclohexane rings.

Bond distances in the bicyclic system are similar to those found by Ferguson, Phillips & Restivo (1975) in *endo,endo*-2,6-bis(phenylcarbamoyloxy)-*cis*-bicyclo-[3.3.0]octane.
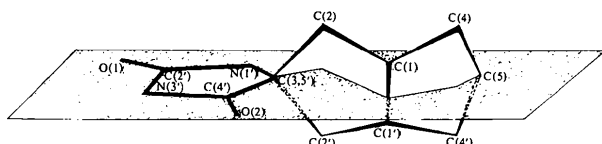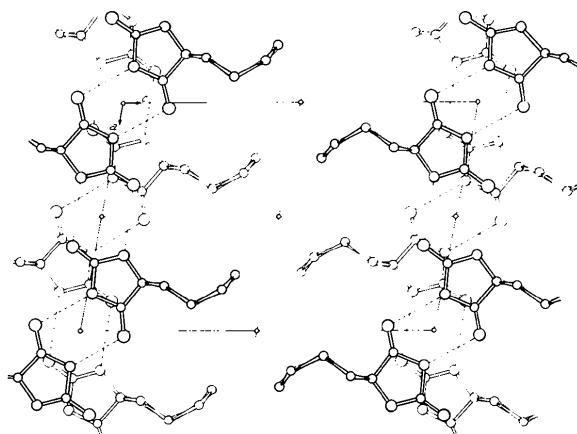

Fig. 2. Projection of the structure down *b*.

Only C(4)—C(5) is considerably shorter than the normal value of 1·539 Å; this effect could be attributed to thermal oscillations of this part of the molecule; the thermal parameters of C(4) and C(5) are very large and the distances between C(5) and the neighbouring molecules are also large, 4·398, 5·333 and 6·340 Å.

Two crystallographically independent intermolecular N—H⋯O bonds occur: N(1')—H(1')⋯O(1) for which N(1')⋯O(1) = 2·829 (4) Å and N(1')—H(1')⋯O(1) (−*x*,−*y*,−*z*) = 163·4°, and N(3')—H(3')⋯O(2) for which N(3')⋯O(2) (−*x* + 1, −*y*, −*z*) = 2·901 (4) Å and N(3')—H(3')⋯O(2) = 177·9°. These hydrogen bonds are indicated by broken lines in Fig. 2 which shows a projection of the crystal structure along *b*. There are no other contacts <4 Å.

Table 3. *Bond lengths (Å) and angles (°) for non-hydrogen atoms with e.s.d.'s in parentheses*

C(1)—C(2)	1·505 (4)	C(2)—C(1)—C(4)	112·7 (4)
C(2)—C(3,5')	1·540 (4)	C(2)—C(1)—C(1)'	107·0 (2)
C(1)—C(1)'	1·540 (4)	C(4)—C(1)—C(1)'	106·2 (3)
C(1)—C(4)	1·567 (7)	C(1)—C(2)—C(3,5')	105·3 (2)
C(4)—C(5)	1·471 (10)	C(2)—C(3,5')—C(2)'	105·3 (2)
C(3,5')—N(1')	1·454 (4)	C(1)—C(4)—C(5)	103·1 (5)
N(1')—C(2')	1·335 (5)	C(4)—C(5)—C(4)'	110·1 (5)
C(2')—N(3')	1·390 (4)	N(1')—C(3,5')—C(4')	100·1 (2)
N(3')—C(4')	1·357 (4)	C(2)—C(3,5')—N(1')	114·0 (2)
C(4')—C(3,5')	1·526 (5)	C(2')—N(1')—C(3,5')	113·8 (3)
C(2')—O(1)	1·218 (4)	N(1')—C(2')—N(3')	106·8 (2)
C(4')—O(2)	1·214 (4)	N(1')—C(2')—O(1)	128·8 (3)
		N(3')—C(2')—O(1)	124·4 (3)
		C(2')—N(3')—C(4')	112·0 (3)
		N(3')—C(4')—C(3,5')	107·3 (2)
		N(3')—C(4')—O(2)	125·7 (3)
		C(3,5')—C(4')—O(2)	127·0 (3)

Fig. 1. Atom-numbering scheme.

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