8,9,10,11,11a,11b,12,13-Octahydro-6*H*,7a*H*-benzo[5,6][1,3]oxazino[3,4-*a*]quinoline

By A. Griffiths

Physics Department, Portsmouth Polytechnic, King Henry I Street, Portsmouth, PO1 2DZ, England

Abstract. $C_{16}H_{21}NO$, $M_r = 243 \cdot 15$, monoclinic, $P2_1/n$, $a = 5 \cdot 161$ (2), $b = 15 \cdot 576$ (10), $c = 16 \cdot 362$ (10) Å, $\beta = 89 \cdot 52$ (3)°, $V = 1315 \cdot 2$ Å^3, $Z = 4$, $D_c = 1 \cdot 227$ g cm^{-3}. The structure was solved by direct methods and refined by block-diagonal least-squares methods to a residual $R = 0 \cdot 064$ for 815 reflexions.

Introduction. Crystals of the title compound were provided by Dr J. Mitchell (Chemistry Department, Portsmouth Polytechnic). 2733 independent reflexions

Table 1. *Final fractional coordinates* ($\times 10^4$)

	x	y	z
C(1)	7324 (10)	5865 (4)	2607 (4)
C(2)	6870 (12)	5672 (4)	3417 (4)
C(3)	5208 (11)	5006 (4)	3593 (3)
C(4)	3947 (9)	4542 (3)	3005 (3)
C(5)	2110 (10)	3837 (3)	3236 (3)
C(6)	2036 (9)	3171 (3)	2559 (3)
C(7)	1331 (8)	3621 (3)	1768 (3)
C(8)	941 (8)	3012 (3)	1046 (3)
C(9)	3218 (9)	2409 (3)	882 (3)
C(10)	2861 (9)	1880 (3)	109 (3)
C(11)	2377 (9)	2441 (3)	−633 (3)
C(12)	−2 (9)	3022 (3)	−469 (3)
C(13)	302 (8)	3549 (3)	298 (3)
O(14)	2394 (6)	4148 (2)	152 (2)
C(15)	2631 (9)	4714 (3)	813 (3)
N(16)	3344 (7)	4250 (2)	1554 (2)
C(17)	4447 (9)	4738 (3)	2185 (3)
C(18)	6136 (10)	5419 (3)	2001 (3)
H(1)	8573	6343	2469
H(2)	7752	5996	3853
H(3)	4888	4863	4185
H(5*A*)	2640	3566	3758
H(5*B*)	315	4088	3307
H(6*A*)	3785	2887	2500
H(6*B*)	728	2707	2693
H(7)	−327	3940	1867
H(8)	−604	2638	1166
H(9*A*)	4833	2767	804
H(9*B*)	3465	2016	1354
H(10*A*)	4410	1512	9
H(10*B*)	1302	1490	195
H(11*A*)	3937	2806	−735
H(11*B*)	2058	2069	−1120
H(12*A*)	−193	3409	−950
H(12*B*)	−1545	2642	−414
H(13)	−1347	3878	412
H(15*A*)	4015	5161	676
H(15*B*)	957	5030	903
H(18)	6483	5567	1414

(1360 non-zero) were measured with filtered Mo $K\alpha$ radiation on a Stoe STADI-4, four-circle computer-controlled diffractometer. A θ–2θ scan was adopted, and the background measured for 30 s at each end of the scanning range. The intensities were corrected for Lorentz and polarization effects and these reflexions for which $I < 3\sigma(I)$ were given zero weight. The unit-cell parameters were refined by least-squares methods using the 2θ angles measured on the diffractometer for 13 reflexions.

The structure was solved by direct methods using *MULTAN* (Germain, Main & Woolfson, 1971). All the non-hydrogen atoms appeared clearly in the E map based on the set of signs with the highest figure of merit. Refinement was carried out using a block-diagonal least-squares program which minimized the function $\Sigma (w\Delta^2) = \Sigma [w(K|F_o| - |F_c|)^2]$. Atomic scattering factors for all atoms were obtained from an expression of the form $f = A \exp(-ax^2) + B \exp(-bx^2) + C$ (Forsyth & Wells, 1959). The non-hydrogen atoms were refined with anisotropic thermal parameters, while the H atoms were positioned, but not refined, at calculated coordinates. The R value for all non-zero reflexions settled around $0 \cdot 15$ even though repeated difference syntheses showed no features in excess of $\pm 0 \cdot 5$ e Å^{-3}. Examination of the structure factors showed that approximately 40% had very low amplitudes [barely over the $I < 3\sigma(I)$ limit] showing poor agreement with F_c, and when these were given zero weight the R value for 815 reflexions was $0 \cdot 064$. (Since this was not an accurate structural study, but rather a determination of stereochemistry, it was felt that the reduction to 815 reflexions was justified.) The final positional parameters are listed in Table 1.*

Discussion. The structure determination of the title compound was undertaken in support of an ongoing IR and NMR spectroscopic study of the stereochemistry of nitrogen bridgehead compounds (Mitchell, 1975).
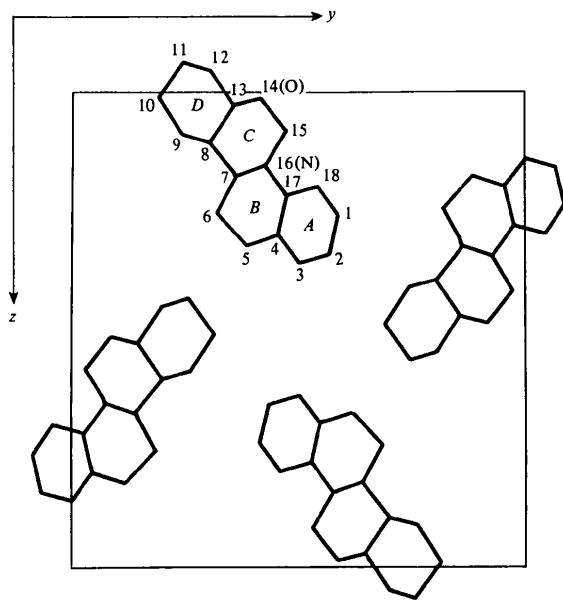

Fig. 1. The (100) projection showing molecular packing and atom numbering.

Table 2. *Bond lengths* (Å) *and angles* (°)

C(1)–C(2)	1·376 (9)	C(1)–C(2)–C(3)	117·7 (5)
C(2)–C(3)	1·376 (8)	C(2)–C(3)–C(4)	123·3 (5)
C(3)–C(4)	1·374 (7)	C(3)–C(4)–C(17)	118·3 (4)
C(4)–C(5)	1·496 (7)	C(4)–C(17)–C(18)	118·6 (4)
C(4)–C(17)	1·398 (7)	C(7)–C(18)–C(1)	120·8 (5)
C(5)–C(6)	1·517 (7)	C(18)–C(1)–C(2)	121·2 (5)
C(6)–C(7)	1·520 (7)	C(17)–C(4)–C(5)	120·9 (4)
C(7)–C(8)	1·529 (7)	C(4)–C(5)–C(6)	109·6 (4)
C(7)–N(16)	1·468 (6)	C(5)–C(6)–C(7)	108·4 (4)
C(8)–C(9)	1·527 (6)	C(6)–C(7)–N(16)	109·6 (4)
C(8)–C(13)	1·521 (7)	C(7)–N(16)–C(17)	118·4 (4)
C(9)–C(10)	1·522 (7)	N(16)–C(17)–C(4)	120·9 (4)
C(10)–C(11)	1·518 (7)	N(16)–C(7)–C(8)	109·0 (4)
C(11)–C(12)	1·547 (7)	C(7)–C(8)–C(13)	108·3 (4)
C(12)–C(13)	1·511 (7)	C(8)–C(13)–O(14)	108·6 (3)
C(13)–O(14)	1·445 (5)	C(13)–O(14)–C(15)	110·4 (3)
O(14)–C(15)	1·402 (6)	O(14)–C(15)–N(16)	110·8 (4)
C(15)–N(16)	1·461 (6)	C(15)–N(16)–C(7)	110·0 (3)
N(16)–C(17)	1·407 (6)	C(8)–C(9)–C(10)	112·2 (4)
C(17)–C(18)	1·402 (7)	C(9)–C(10)–C(11)	112·2 (4)
C(18)–C(1)	1·362 (8)	C(10)–C(11)–C(12)	109·5 (4)
		C(11)–C(12)–C(13)	111·8 (4)
		C(12)–C(13)–C(8)	113·4 (4)
		C(13)–C(8)–C(9)	111·7 (4)

Table 3. *Torsion angles determined using calculated hydrogen positions*

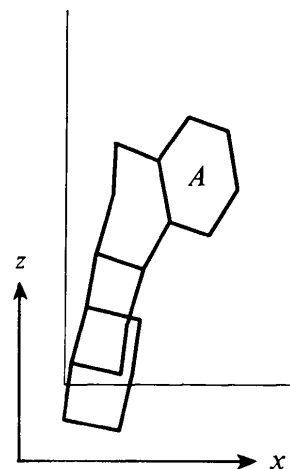| Bond direction	Torsion angle
C(5)–C(6)	−57·9°
C(6)–C(7)	66·5
C(7)–C(8)	−54·7
C(8)–C(13)	56·0
C(13)–C(12)	−55·5
C(12)–C(11)	55·3
C(11)–C(10)	−57·7
C(10)–C(9)	58·5
C(9)–C(8)	−56·2

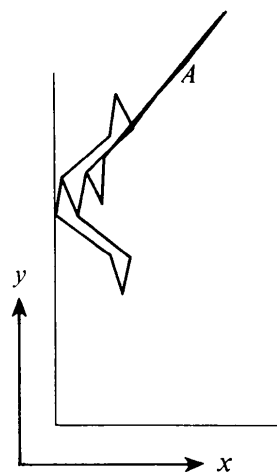
Fig. 2. The (010) projection of a single molecule.

Fig. 3. The (001) projection of a single molecule.

Figs. 1, 2 and 3 give a clear indication of the molecular conformation and confirm the spectroscopic data conclusions (Mitchell, 1975). The least-squares best-fit planes through rings C and D are given by: $0.2352x - 0.1865y - 0.0600z + 1.00 = 0$ and $-0.2341x - 0.1922y + 0.0414z + 1.00 = 0$ respectively. These planes are inclined at an angle of 76·6°. Table 3 lists the torsion angles within and around ring D and these are typical of normal cyclohexane rings. The molecular geometry is given in Table 2 and examination shows that no bond lengths or angles depart significantly from accepted values.

References

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