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Structure of (+)-Epilupinine

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Abstract. $C_{10}H_{19}NO$, M_r = 169·3, orthorhombic, $P2_12_12_1$, a = 9·069 (2), b = 9·660 (2), c = 11·431 (2) Å, Z = 4, D_x = 1·12 Mg m^{-3}, μ(Cu $K\alpha$) = 0·56 mm^{-1}. The final R value is 0·060 for 613 independent reflexions. The hydroxymethyl group is equatorially bonded to the *trans*-quinolizidine nucleus. The molecules are linked by intermolecular N···H′–O′ hydrogen bonds.

Introduction. The investigation of the (+)-epilupinine structure has been undertaken as part of systematic studies on lupinine and its derivatives. (+)-Epilupinine (m.p. 351 K) isolated from *Lupinus pilosus* was recrystallized from *n*-heptane. Precession photographs indicated univocally the space group $P2_12_12_1$. A single crystal 0·15 × 0·17 × 0·40 mm was sealed in a glass capillary. Intensity data were measured on a Syntex $P2_1$ automatic diffractometer with the θ–2θ scan technique, using graphite-monochromated Cu $K\alpha$ radiation. Lorentz and polarization corrections were applied, but no absorption correction was made. 613 reflexions with $I \geq 1{\cdot}96\sigma(I)$ were used in the calculations. The structure was solved by direct methods using *MULTAN* (Germain, Main & Woolfson, 1971) and refined by full-matrix least squares with isotropic and then anisotropic thermal parameters to R = 0·15. All H atoms were found from a difference Fourier synthesis and were given isotropic temperature factors of 5·0 Å^2. H atoms were included in the refinement, but their parameters were kept fixed. Weighted full-matrix least-squares refinement gave final R = 0·060 and R_w = 0·070 $\{R = \sum(||F_o| - |F_c||)/\sum|F_o|$, $R_w = [\sum w(\Delta F)^2/\sum wF_o^2]^{1/2}\}$. The weighting scheme used was: $w = (F_o/F_l)^2$ if $F_o < F_l$, $w = 1$ if $F_l \leq F_o \leq F_h$, $w = (F_h/F_o)^2$ if $F_o > F_h$, with F_l = 4·1 and F_h = 11·2. Positional parameters are given in Table 1.* The configuration of the molecule has been chosen as (1*S*,5*R*,10*R*), *i.e.* in agreement with that established for lupinine (Koziol, Gdaniec & Kosturkiewicz, 1980).

* Lists of structure factors and anisotropic thermal parameters of the heavy atoms have been deposited with the British Library Lending Division as Supplementary Publication No. SUP 34919 (10 pp.). Copies may be obtained through The Executive Secretary, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

Table 1. *Positional atomic parameters* (×10^4 *for non-hydrogen atoms*, ×10^3 *for* H *atoms*) *and isotropic thermal parameters for non-hydrogen atoms* (B_{iso} = 5·0 Å^2 *for all* H *atoms*)

	x	y	z	B (Å^2)
C(1)	678 (6)	8025 (5)	3799 (5)	2·8 (4)
C(2)	1119 (7)	6821 (6)	2987 (5)	3·7 (5)
C(3)	2662 (7)	6362 (7)	3215 (6)	3·9 (5)
C(4)	3707 (7)	7585 (7)	3139 (5)	3·8 (5)
N(5)	3293 (4)	8698 (5)	3942 (4)	2·9 (3)
C(6)	4401 (6)	9808 (7)	3846 (6)	4·1 (5)
C(7)	4090 (7)	10986 (7)	4681 (6)	4·1 (5)
C(8)	2559 (8)	11551 (7)	4471 (6)	4·4 (5)
C(9)	1433 (7)	10404 (6)	4550 (5)	4·3 (5)
C(10)	1781 (6)	9218 (6)	3699 (5)	2·8 (4)
C(11)	−916 (6)	8469 (6)	3536 (5)	3·2 (4)
O(1)	−1933 (4)	7367 (4)	3691 (3)	3·5 (3)
H(1)	−187	688	437	
H(21)	101	724	205	
H(22)	43	596	339	
H(31)	276	586	404	
H(32)	299	559	257	
H(41)	371	798	240	
H(42)	476	716	333	
H(61)	448	1011	308	
H(62)	551	948	397	
H(71)	413	1063	561	
H(72)	500	1170	472	
H(81)	255	1210	377	
H(82)	250	1207	497	
H(91)	156	1000	531	
H(92)	30	1072	457	
H(101)	180	956	287	
H(11)	81	748	467	
H(111)	−123	945	411	
H(112)	−99	875	271	

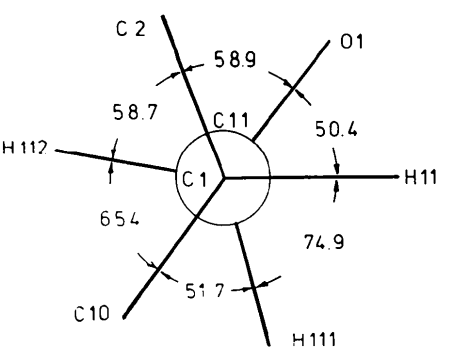

Fig. 1. A Newman projection along the C(1)–C(11) bond.

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Table 2. *Bond lengths* (Å)

C(1)–C(2)	1·541 (8)	C(7)–C(8)	1·511 (9)
C(2)–C(3)	1·491 (9)	C(8)–C(9)	1·509 (9)
C(3)–C(4)	1·517 (9)	C(9)–C(10)	1·536 (8)
C(4)–N(5)	1·463 (8)	C(10)–C(1)	1·530 (7)
N(5)–C(6)	1·473 (8)	C(1)–C(11)	1·537 (8)
N(5)–C(10)	1·486 (7)	C(11)–O(1)	1·419 (7)
C(6)–C(7)	1·512 (9)		

Table 3. *Bond angles* (°)

C(10)–C(1)–C(2)	110·7 (5)	C(7)–C(8)–C(9)	110·3 (6)
C(1)–C(2)–C(3)	111·3 (5)	C(8)–C(9)–C(10)	111·8 (5)
C(2)–C(3)–C(4)	110·1 (5)	C(9)–C(10)–C(1)	112·3 (5)
C(3)–C(4)–N(5)	112·1 (5)	C(9)–C(10)–N(5)	108·8 (4)
C(4)–N(5)–C(10)	111·6 (4)	C(1)–C(10)–N(5)	109·6 (4)
C(4)–N(5)–C(6)	108·2 (4)	C(10)–C(1)–C(11)	113·0 (5)
C(6)–N(5)–C(10)	111·7 (4)	C(2)–C(1)–C(11)	109·7 (5)
N(5)–C(6)–C(7)	111·9 (5)	C(1)–C(11)–O(1)	112·2 (5)
C(6)–C(7)–C(8)	110·1 (6)		

Table 4. *Torsion angles* (°)

C(10)–C(1)–C(2)–C(3)	54·6 (6)
C(1)–C(2)–C(3)–C(4)	−53·8 (7)
C(2)–C(3)–C(4)–N(5)	56·5 (6)
C(3)–C(4)–N(5)–C(10)	−59·2 (6)
C(4)–N(5)–C(10)–C(1)	58·2 (6)
N(5)–C(10)–C(1)–C(2)	−55·4 (6)
N(5)–C(6)–C(7)–C(8)	−56·6 (7)
C(6)–C(7)–C(8)–C(9)	54·8 (7)
C(7)–C(8)–C(9)–C(10)	−56·0 (7)
C(8)–C(9)–C(10)–N(5)	56·5 (6)
C(9)–C(10)–N(5)–C(6)	−57·3 (6)
C(10)–N(5)–C(6)–C(7)	58·8 (6)
N(5)–C(10)–C(1)–C(11)	−179·0 (5)
C(3)–C(2)–C(1)–C(11)	179·9 (6)
O(1)–C(11)–C(1)–C(2)	58·9 (7)
O(1)–C(11)–C(1)–C(10)	−177·0 (5)

Discussion. Bond lengths, bond angles and torsion angles for the epilupinine molecule are given in Tables 2, 3 and 4, respectively. The average C–H bond length is 1·02 Å. The bond lengths and angles are not significantly different from those found in lupinine (Kozioł, Kosturkiewicz & Podkowińska, 1978). Both rings in the *trans*-quinolizidine system are in chair conformations. The values of the torsion angles C(3)–C(2)–C(1)–C(11) [179·9°] and N(5)–C(10)–C(1)–C(11) [−179·0°] indicate equatorial orientation of the $-CH_2OH$ group. This result is in agreement with the conclusions reached by Thomas, Vipond & Marion (1955). The orientation of the hydroxyl group is shown in Fig. 1. Fig. 2 shows the conformation of the molecule.

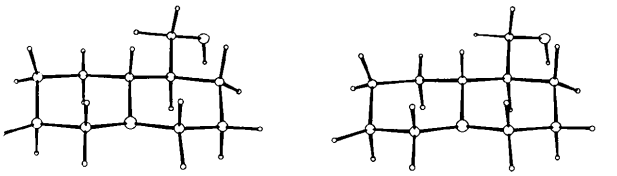

Fig. 2. Stereoscopic view of the (+)-epilupinine molecule.

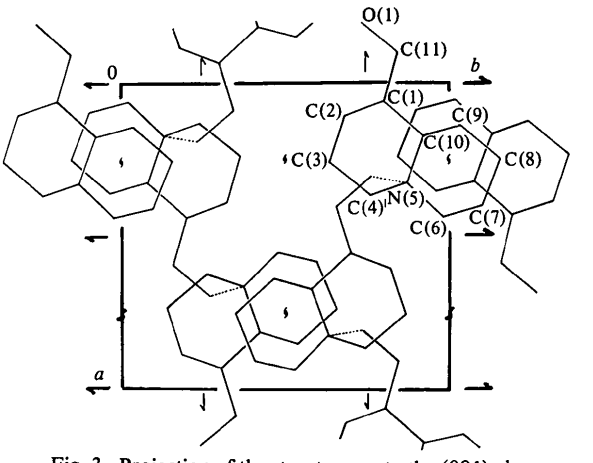

Fig. 3. Projection of the structure on to the (001) plane.

The molecules in the crystal (Fig. 3) are linked by intermolecular N(5)···H(1′)–O(1′) hydrogen bonds, forming infinite chains along the *a* axis. The hydrogen-bond parameters are: N(5)···O(1′) 2·902 (6), H(1′)–O(1′) 0·91, N(5)···H(1′) 2·01 Å, ∠N(5)···H(1′)–O(1′) 164·9°. The geometry of this hydrogen bond is similar to that found in lupinine.

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