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Structure of Vincamajine

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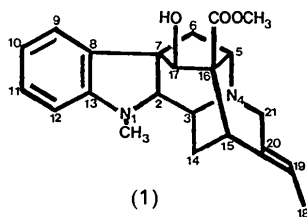
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Abstract. $C_{22}H_{26}N_2O_3$, $M_r = 366.46$, orthorhombic, $P2_12_12_1$, $a = 11.816$ (9), $b = 12.447$ (2), $c = 13.193$ (4) Å, $V = 1940$ (2) Å³, $Z = 4$, $D_x = 1.25$ Mg m⁻³, $\lambda(\text{Mo } K\alpha) = 0.71069$ Å, $\mu(\text{Mo } K\alpha) = 0.0917$ mm⁻¹, $F(000) = 784$, $T = 293$ K, $R = 0.055$ for 1446 observed reflections with $I > 3\sigma(I)$. The *B* ring has an envelope form (with C2 out of the plane defined by the remaining four atoms). The *C* ring is a skew envelope. The *D* ring has a chair form. The *D* and *B* rings are *trans*-fused. The *E* and *F* rings have twist forms. There is an intermolecular hydrogen bond between the hydroxyl H at C17 and N4, with $H \cdots N4 = 1.802$ (5) Å, $O17-H \cdots N4 = 171$ (2)°.

Experimental. Prismatic crystals of vincamajine (1) were grown by slow evaporation from a methanol solution. Preliminary Weissenberg photographs



established symmetry and systematic absences consistent with space group $P2_12_12_1$. A suitable monocrystal, $0.2 \times 0.2 \times 0.8$ mm, was mounted on an Enraf–Nonius CAD-4 diffractometer. Cell parameters were refined from 25 reflections in the range $8 \leq \theta \leq 14^\circ$. The intensities were measured using $\omega/2\theta$ scans up to $2\theta = 50^\circ$ ($0 \leq h \leq 14$, $0 \leq k \leq 14$, $0 \leq l \leq 15$). Three standard reflections monitored every 50

measurements showed a variation of $< 2\%$. Lorentz and polarization corrections, but no correction for absorption. Of 1941 independent reflections measured, 1446 [$I > 3.0\sigma(I)$] were considered observed. $R_{\text{int}} = 0.052$. The structure was solved by direct methods using *MULTAN80* (Main, Fiske, Hull, Lessinger, Germain, Declercq & Woolfson, 1980). Full-matrix least-squares refinement using *SHELX76* (Sheldrick, 1976). The function minimized was $\sum w(\Delta F)^2$. The H atoms were treated as riding on the adjacent atoms, assuming C—H = 1.08 Å and a fixed temperature coefficient of 6.4 Å². The hydroxyl H at O17 was located in a difference Fourier map. Final $R = 0.055$, $wR = 0.059$, $w = 8.47/[\sigma^2(F_o) + 3.7 \times 10^{-4}F_o^2]$, max. shift/e.s.d. = 0.07 (corresponding to the hydroxyl H at C17); max. and min. heights in final difference Fourier map 0.19 and -0.28 e Å⁻³; scattering factors from *International Tables for X-ray Crystallography* (1974, Vol. IV, pp. 99–100, 149). All calculations were performed on a VAX 785.

Final atomic coordinates and derived parameters are presented in Tables 1 and 2.† The molecule is shown with atomic labels in Fig. 1. The packing diagram (Fig. 2) shows no unusual features.

Related literature. The stereochemistry of this alkaloid, as well as that of the closely related compound vincamedine, was initially reported by Gosset–Garnier, Le Men & Janot (1961, 1965). Recently, the structure and absolute configuration of vincamedine

† Lists of structure factors, anisotropic thermal parameters for non-H atoms and H-atom coordinates have been deposited with the British Library Document Supply Centre as Supplementary Publication No. SUP 54765 (10 pp.). Copies may be obtained through The Technical Editor, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

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Table 1. Fractional atomic coordinates ($\times 10^4$) and equivalent isotropic temperature factors for non-H atoms with *e.s.d.*'s in parentheses

$$B_{eq} = (8\pi^2/3)(U_{11} + U_{22} + U_{33}).$$

	<i>x</i>	<i>y</i>	<i>z</i>	$B_{eq}(\text{\AA}^2)$
N1	6364 (3)	-4451 (4)	5630 (3)	3.8 (2)
C2	7172 (4)	-3649 (4)	5232 (4)	3.7 (2)
C3	7345 (4)	-3612 (4)	4066 (4)	3.5 (2)
N4	8088 (3)	-2667 (4)	3882 (3)	3.6 (2)
C5	7592 (4)	-1679 (5)	4349 (4)	3.4 (2)
C6	7642 (4)	-1733 (4)	5503 (4)	3.5 (2)
C7	6739 (4)	-2588 (5)	5686 (4)	3.5 (2)
C8	6385 (4)	-2989 (4)	6710 (4)	3.3 (2)
C9	6254 (5)	-2432 (5)	7630 (4)	4.1 (2)
C10	5846 (5)	-3039 (6)	8457 (4)	4.8 (3)
C11	5544 (5)	-4094 (5)	8358 (5)	4.7 (3)
C12	5675 (4)	-4637 (5)	7438 (4)	4.4 (3)
C13	6123 (4)	-4058 (4)	6622 (4)	3.4 (2)
C14	6253 (4)	-3549 (5)	3433 (4)	3.9 (2)
C15	6086 (4)	-2363 (5)	3144 (4)	3.6 (2)
C16	6285 (4)	-1651 (4)	4101 (4)	3.1 (2)
C17	5731 (4)	-2128 (4)	5075 (3)	3.0 (2)
O17	5156 (3)	-1335 (3)	5646 (3)	4.0 (2)
C18	5824 (6)	-1040 (6)	1103 (5)	6.1 (4)
C19	6880 (5)	-1476 (5)	1554 (4)	4.7 (3)
C20	6999 (4)	-2046 (4)	2395 (4)	3.6 (2)
C21	8133 (4)	-2439 (5)	2774 (4)	4.2 (3)
C22	5798 (4)	-0546 (5)	3900 (4)	3.8 (2)
O22	4907 (3)	-0372 (4)	3499 (3)	5.7 (2)
C23	6065 (7)	1321 (5)	4031 (6)	6.4 (4)
O23	6471 (3)	0269 (3)	4229 (3)	4.8 (2)
C24	6709 (6)	-5568 (5)	5506 (5)	5.3 (3)

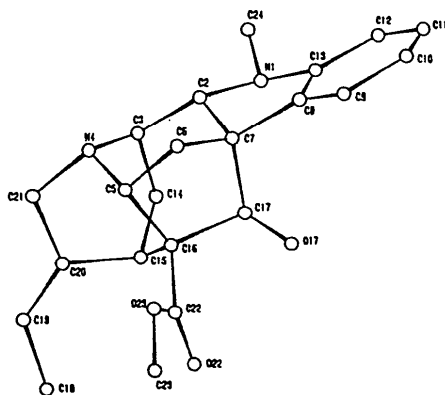


Fig. 1. Atomic numbering and molecular structure of vincamajine.

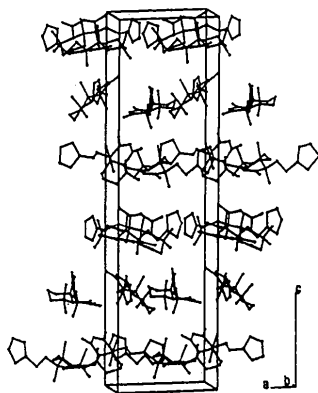


Fig. 2. Packing of the molecules in the crystal structure.

Table 2. Selected bond lengths (\AA) and angles ($^\circ$)

C2—N1	1.478 (7)	C10—C9	1.412 (8)
C13—N1	1.426 (7)	C11—C10	1.366 (9)
C3—C2	1.553 (7)	C12—C11	1.398 (8)
C7—C2	1.537 (8)	C13—C12	1.399 (7)
N4—C3	1.488 (7)	C15—C14	1.538 (8)
C14—C3	1.539 (7)	C16—C15	1.560 (7)
C5—N4	1.495 (7)	C20—C15	1.515 (7)
C21—N4	1.489 (6)	C17—C16	1.559 (7)
C6—C5	1.525 (7)	C22—C16	1.514 (7)
C16—C5	1.580 (6)	C19—C18	1.485 (9)
C7—C6	1.526 (7)	C20—C19	1.325 (8)
C8—C7	1.501 (7)	C21—C20	1.512 (7)
C17—C7	1.548 (7)	O22—C22	1.199 (6)
C9—C8	1.406 (8)	O23—C22	1.360 (6)
C13—C8	1.370 (7)	O23—C23	1.419 (7)
C17—O17	1.416 (6)		
C13—N1—C2	102.9 (4)	C12—C11—C10	121.3 (6)
C3—C2—N1	117.2 (4)	C13—C12—C11	117.4 (5)
C7—C2—N1	103.1 (4)	C8—C13—N1	111.5 (4)
C7—C2—C3	113.8 (5)	C12—C13—N1	127.2 (5)
N4—C3—C2	105.3 (4)	C12—C13—C8	121.3 (5)
C14—C3—C2	115.4 (4)	C15—C14—C3	106.9 (4)
C14—C3—N4	111.4 (4)	C16—C15—C14	109.0 (4)
C5—N4—C3	110.6 (3)	C20—C15—C14	108.7 (4)
C21—N4—C3	109.4 (4)	C20—C15—C16	105.8 (4)
C21—N4—C5	105.2 (4)	C15—C16—C5	107.6 (4)
C6—C5—N4	111.1 (4)	C17—C16—C5	103.4 (4)
C16—C5—N4	108.4 (4)	C17—C16—C15	112.8 (4)
C16—C5—C6	104.2 (4)	C22—C16—C5	115.3 (4)
C7—C6—C5	99.3 (4)	C22—C16—C15	108.5 (4)
C6—C7—C2	107.8 (4)	C22—C16—C17	109.3 (4)
C8—C7—C2	99.0 (4)	C16—C17—C7	104.3 (3)
C8—C7—C6	124.7 (4)	C20—C19—C18	128.3 (6)
C17—C7—C2	111.8 (4)	C19—C20—C15	127.7 (5)
C17—C7—C6	101.4 (4)	C21—C20—C15	109.3 (4)
C17—C7—C8	112.2 (4)	C21—C20—C19	123.0 (5)
C9—C8—C7	130.1 (5)	C20—C21—N4	110.8 (4)
C13—C8—C7	108.0 (5)	O22—C22—C16	125.1 (1)
C13—C8—C9	121.8 (5)	O23—C22—C16	113.6 (4)
C10—C9—C8	116.1 (6)	O23—C22—O22	121.3 (5)
C11—C10—C9	122.0 (6)	C23—O23—C22	115.6 (5)
C16—C17—O17	112.0 (4)	C7—C17—O17	110.5 (4)

have been determined (Solans, Briansó, Mariezcurrena & Gomes, 1987).

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