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Structure of 4-Amino-6,8-dimethylimidazo[1,5-*a*][1,3,5]triazine Monohydrate

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Abstract. $C_7H_9N_3 \cdot H_2O$, $M_r = 181.20$, monoclinic, $P2_1/a$, $a = 8.895$ (1), $b = 13.463$ (2), $c = 7.805$ (1) Å, $\beta = 108.84$ (9)°, $V = 884.6$ (2) Å³, $Z = 4$, $D_m = 1.36$, $D_x = 1.36$ Mg m⁻³, $\lambda(\text{Cu } K\alpha) = 1.54178$ Å, $\mu = 0.725$ mm⁻¹, $F(000) = 384$, $T = 293$ K, $R = 0.041$ for 1040 observed reflexions. The imidazo[1,5-*a*]-[1,3,5]triazine system is planar ($\chi^2 = 38$) within 3σ with average and maximum out-of-mean-plane deviations of 0.004 (6) and 0.009 (3) Å, respectively. Although N(5) can be formally sp^3 hybridized, it has a perfect trigonal configuration: the sum of bond angles around N(5) is 360.0 (4)°. The endocyclic N—C bonds can be divided into two classes: (i) bonds in the range 1.303 (3)–1.326 (3) Å which show strong double-bond character and (ii) bonds in the range 1.365 (3)–1.426 (3) Å which reflect some double-bond character, but to a much lesser extent than the first class. All protons available from the *exo*-amino group and from the water molecule, as well as all acceptors in the ring system and the water O atom, take part in intermolecular hydrogen bonding. The crystal packing is thus governed by hydrogen bonds which form a three-dimensional network.

Experimental. Colourless crystals obtained from chloroform–methanol 95/5. D_m by flotation. Approximate unit-cell dimensions and space group from Weissenberg photographs. Crystal 0.25×0.45

$\times 0.30$ mm. Syntex $P2_1$ diffractometer, graphite-monochromated Cu $K\alpha$ radiation, $\theta/2\theta$ scan mode; precise lattice parameters from 15 reflexions with $6 < \theta < 15^\circ$. No absorption correction. 1181 unique reflexions measured in the range $h -9 \rightarrow 9$, $k 0 \rightarrow 14$, $l 0 \rightarrow 8$, maximum $\sin\theta/\lambda = 0.546$ Å⁻¹ at $T = 293$ K. No significant intensity variation ($\pm 2.5\%$) for two standard reflexions ($\bar{1}\bar{2}\bar{2}$, $\bar{1}12$) checked every 100 reflexions. Peak-profile analysis according to Lehmann & Larsen (1974). Lorentz and polarization correction. 1040 observed reflexions with $I \geq 1.96\sigma(I)$. Structure solved by direct methods using *SHELXS86* (Sheldrick, 1985). All H atoms from $\Delta\rho$ map. Full-matrix least-squares refinement on F , anisotropic thermal parameters for non-H atoms and isotropic for H atoms. F_c values multiplied by $(1 - xF_c^2/\sin 2\theta)$ where x is the empirical isotropic extinction parameter refined to $4.04(3) \times 10^{-6}$. 163 parameters in final cycle. $R = 0.041$, $wR = 0.067$, $w = 1/[\sigma^2(F_o) + 0.00015F_o^2]$, $S = 4.30$, $(\Delta/\sigma)_{\max} = 0.002$, $(\Delta\rho)_{\min} = -0.18$, $(\Delta\rho)_{\max} = 0.15$ e Å⁻³. Programs used: *SHELXS86* (Sheldrick, 1985), *SHELX76* (Sheldrick, 1976) and other local programs (Jaskólski, 1982). Atomic scattering factors from *International Tables for X-ray Crystallography* (1974, Vol. IV). All calculations were performed on an IBM PC computer. The final atomic coordinates are given in Table 1, bond lengths, bond angles and

Table 1. Fractional coordinates of the non-H atoms and equivalent isotropic thermal parameters (Å²) with e.s.d.'s in parentheses

$$U_{eq} = (1/3)\sum_i \sum_k U_{ik} a_i^* a_k^* a_i \cdot a_k$$

	x	y	z	U _{eq}
C(2)	-0.0167 (3)	0.2696 (2)	0.5604 (3)	0.0476 (9)
C(4)	0.0569 (2)	0.3737 (2)	0.3665 (3)	0.0388 (8)
C(6)	0.1545 (2)	0.2732 (2)	0.1459 (3)	0.0405 (7)
C(8)	0.1225 (3)	0.1269 (2)	0.2543 (3)	0.0423 (9)
C(9)	0.0751 (2)	0.1946 (2)	0.3567 (3)	0.0396 (8)
C(11)	0.1945 (3)	0.3507 (2)	0.0319 (3)	0.0512 (11)
C(12)	0.1263 (4)	0.0171 (2)	0.2691 (4)	0.0552 (11)
N(1)	0.0168 (2)	0.1856 (1)	0.4977 (3)	0.0478 (8)
N(3)	0.0014 (2)	0.3637 (1)	0.5041 (2)	0.0445 (8)
N(5)	0.0949 (2)	0.2897 (1)	0.2870 (2)	0.0385 (7)
N(7)	0.1709 (2)	0.1767 (1)	0.1267 (2)	0.0431 (7)
N(10)	0.0732 (2)	0.4631 (2)	0.3063 (3)	0.0489 (9)
O(W)	0.2296 (3)	0.6029 (2)	0.1602 (3)	0.0684 (9)

Table 2. Bond lengths (Å), angles (°) and intermolecular hydrogen bonds (Å, °) with e.s.d.'s in parentheses

N(1)—C(2)	1.303 (3)	N(7)—C(6)	1.321 (3)	
N(1)—C(9)	1.365 (3)	N(7)—C(8)	1.380 (3)	
N(3)—C(2)	1.368 (3)	C(4)—N(10)	1.317 (3)	
N(3)—C(4)	1.326 (3)	C(6)—C(11)	1.487 (3)	
N(5)—C(4)	1.384 (3)	C(8)—C(9)	1.365 (3)	
N(5)—C(6)	1.385 (3)	C(8)—C(12)	1.483 (3)	
N(5)—C(9)	1.425 (3)			
C(2)—N(1)—C(9)	114.7 (2)	N(5)—C(6)—C(11)	126.1 (2)	
N(1)—C(2)—N(3)	128.2 (2)	N(7)—C(6)—C(11)	124.4 (2)	
C(2)—N(3)—C(4)	117.9 (2)	C(6)—N(7)—C(8)	108.9 (2)	
N(3)—C(4)—N(5)	119.3 (2)	N(7)—C(8)—C(9)	109.0 (2)	
N(3)—C(4)—N(10)	119.7 (2)	N(7)—C(8)—C(12)	122.4 (2)	
N(5)—C(4)—C(10)	121.1 (2)	C(9)—C(8)—C(12)	128.5 (2)	
C(4)—N(5)—C(6)	134.4 (2)	N(1)—C(9)—C(8)	133.1 (2)	
C(4)—N(5)—C(9)	119.0 (2)	N(1)—C(9)—N(5)	120.9 (2)	
C(6)—N(5)—C(9)	106.6 (2)	N(5)—C(9)—C(8)	106.0 (2)	
N(5)—C(6)—N(7)	109.4 (2)			
D—H...A	D—H	H...A	D...A	D—H...A
N(10)—H(N1)...O(W)	0.94 (3)	2.11 (3)	2.973 (4)	129 (2)
N(10)—H(N2)...N(3 ⁱ)	0.90 (3)	2.06 (3)	2.950 (3)	172 (2)
O(W)—H(W1)...N(7 ⁱⁱ)	0.90 (4)	1.95 (4)	2.840 (3)	174 (2)
O(W)—H(W2)...N(1 ⁱⁱⁱ)	0.90 (4)	2.27 (3)	3.093 (4)	152 (2)

Symmetry code: (i) $-x, 1-y, 1-z$; (ii) $\frac{1}{2}-x, \frac{1}{2}+y, -z$; (iii) $\frac{1}{2}-x, \frac{1}{2}+y, 1-z$.

hydrogen-bond parameters in Table 2.* A view of the molecule with the atom-numbering scheme is shown in Fig. 1, and of crystal packing in Fig. 2.

Related literature. The imidazo[1,5-*a*][1,3,5]triazine fragment is of interest as an isoelectronic analogue of the adenine ring system. The synthesis, chemistry and structures of analogues of the title compound have been reported (Golankiewicz, Holtwick, Holmes, Duesler & Leonard, 1979; Golankiewicz, Zeidler & De Clercq, 1987).

* Lists of structure factors, anisotropic thermal parameters, torsion angles and H-atom parameters have been deposited with the British Library Document Supply Centre as Supplementary Publication No. SUP 54849 (10 pp.). Copies may be obtained through The Technical Editor, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

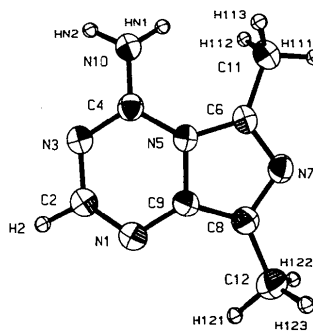


Fig. 1. ORTEP (Johnson, 1976) drawing of the molecule displaying the thermal ellipsoids at 50% probability.

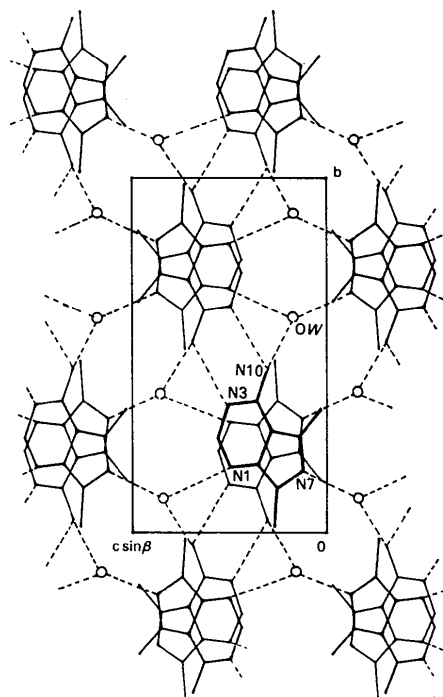


Fig. 2. Projection of the structure down *a* (O atoms as circles, H atoms omitted) (PLUTO; Motherwell & Clegg, 1978). The broken lines indicate hydrogen bonds.

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Structure of Vincamajine

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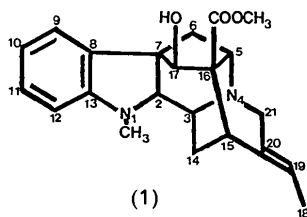
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Abstract. $C_{22}H_{26}N_2O_3$, $M_r = 366.46$, orthorhombic, $P2_12_12_1$, $a = 11.816$ (9), $b = 12.447$ (2), $c = 13.193$ (4) Å, $V = 1940$ (2) Å³, $Z = 4$, $D_x = 1.25$ Mg m⁻³, $\lambda(\text{Mo } K\alpha) = 0.71069$ Å, $\mu(\text{Mo } K\alpha) = 0.0917$ 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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