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Structure of Nanteine

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Abstract. Nanteine is an aporphine alkaloid, $C_{20}H_{21}NO_4$, $M_r = 339\cdot39$, orthorhombic, $P2_12_12_1$, $a = 5\cdot010\,(3)$, $b = 16\cdot940\,(5)$, $c = 19\cdot446\,(6)$ Å, $V = 1650\,(1)$ Å^3, $Z = 4$, $D_x = 1\cdot366$ g cm^{-3}, λ(Mo $K\alpha$) = 0·7107 Å, $\mu = 0\cdot89$ cm^{-1}, $F(000) = 720$, $T = 293\,(1)$ K, $R = 0\cdot050$, $wR = 0\cdot061$ for 1334 independent observed reflections. The structure is composed of isolated molecules which are held together only by van der Waals interactions. Ring *B* adopts a screw-boat conformation, whereas the nearest form of the *C* ring is that of a half-chair. The interplanar angle between the two aromatic rings *A* and *D* is 24·9 (1)°.

Table 1. *Fractional atomic coordinates* (× 10^4) *and equivalent isotropic temperature factors* (Å^2 × 10^3)

$U_{eq} = 1/3(U_{11} + U_{22} + U_{33})$.

	x	y	z	U_{eq}
C1	314 (11)	4449 (3)	4297 (2)	46 (1)
C2	789 (11)	5253 (3)	4158 (2)	44 (1)
C3	−482 (11)	5876 (3)	4621 (2)	51 (1)
C4	971 (12)	6642 (3)	4559 (2)	52 (1)
C5	2878 (10)	6340 (2)	3434 (2)	37 (1)
C6	2505 (9)	6433 (2)	2670 (2)	39 (1)
C7	4461 (8)	5933 (2)	2310 (2)	36 (1)
C8	5698 (11)	6205 (2)	1709 (2)	42 (1)
C9	7475 (9)	5726 (2)	1392 (2)	40 (1)
C10	8121 (9)	4992 (3)	1653 (2)	42 (1)
C11	7012 (9)	4704 (3)	2238 (3)	44 (1)
C12	5074 (9)	5181 (2)	2572 (2)	37 (1)
C13	3563 (9)	4909 (2)	3181 (2)	38 (1)
C14	3121 (9)	4114 (3)	3338 (2)	42 (1)
C15	1483 (10)	3885 (2)	3884 (2)	46 (1)
C16	2389 (9)	5480 (2)	3613 (2)	38 (1)
C17	−924 (14)	2849 (3)	4405 (3)	68 (2)
C18	2414 (11)	3187 (3)	2470 (3)	56 (1)
C19	2133 (14)	7699 (3)	3785 (3)	59 (1)
C20	10658 (11)	5193 (3)	729 (3)	54 (1)
N	1137 (9)	6879 (2)	3838 (2)	45 (1)
O1	4265 (7)	3508 (2)	2951 (2)	44 (1)
O2	1207 (9)	3090 (2)	3985 (2)	59 (1)
O3	8896 (9)	5859 (2)	796 (2)	58 (1)
O4	9971 (8)	4628 (2)	1233 (2)	62 (1)

Experimental. Nanteine was isolated from the aerial part of *Corydalis cava* (L.) as described by Preininger, Thakur, & Šantavy (1976) and Preininger, Vesely, Gašić, Šimanek & Dolejš (1975). A single crystal of size 0·36 × 0·22 × 0·18 mm was mounted on a CAD-4 diffractometer (Berne) using graphite-monochromated Mo $K\alpha$ radiation. Cell constants were refined by least-squares fit for 19 centred reflections with θ range 4·1–9·9°. Intensity data were collected with ω–2θ scan in range $2\theta < 50°$; h 0 to 5, k 0 to 19, l 0 to 22. The systematic absences ($h00$: $h = 2n + 1$, $0k0$; $k = 2n + 1$, $00l$: $l = 2n + 1$) correspond to those of orthorhombic space group $P2_12_12_1$. One standard reflection (041) was measured every 200 min, but no intensity variations were recorded. Of 1709 measured reflections, 1335 with $I > 3\cdot5\sigma(I)$ were considered observed. Data were corrected for Lorentz and polarization effects. Structure was solved using *SHELXS*86 (Sheldrick, 1986) which located all non-H atoms ($R = 0\cdot175$). Full-matrix least-squares refinement (on F) with program *SHELXS*76 (Sheldrick, 1976) minimized $\sum w(\Delta F)^2$ for 227 parameters with $w = [\sigma^2(F_o) + 0\cdot1F_o^2]^{-1}$. Final $R = 0\cdot050$, $wR = 0\cdot061$, $(\Delta/\sigma)_{max} = 0\cdot16$. A final difference Fourier map showed excursion of density from $-0\cdot19$ to $+0\cdot13$ e Å^{-3}. Positions of H atoms were generated from assumed geometries and their positions were taken into account without refinement in structure-factor calculations, with common isotropic temperature factor $U = 0\cdot074\,(4)$ Å^2. Scattering factors were taken from *SHELX*76.

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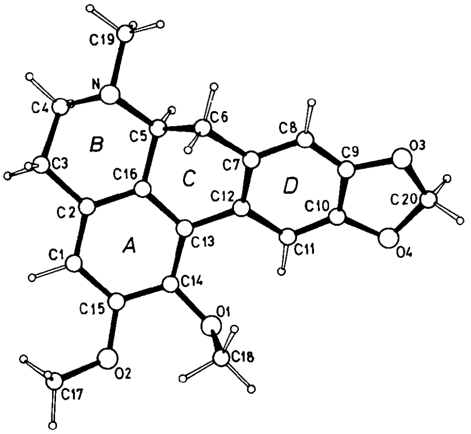

Fig. 1. Perspective view of the molecule.

Calculations were performed on a PC/AT computer. The structure is defined in Table 1 and Fig. 1.*

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* Lists of structure factors, anisotropic thermal parameters and H-atom parameters have been deposited with the British Library Document Supply Centre as Supplementary Publication No. SUP 54261 (12 pp.). Copies may be obtained through The Technical Editor, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

Related literature. Structure of leucoxine hydrobromide and isoboldine hydrobromide: Brown & Hall (1977); structure of bulbocapnine methiodide: Wei, Basu, Einstein & Hingerty (1984); structure of bulbocapnine: Ribár, Mészáros, Gašić, Kanyó & Engel (1991).

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Structure of 1*H*-3,4-Dihydro-2,3-benzothiazine 2,2-Dioxide

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Abstract. $C_8H_9NO_2S$, $M_r = 183{\cdot}23$, monoclinic, $P2_1/c$, $a = 9{\cdot}492\,(3)$, $b = 10{\cdot}443\,(2)$, $c = 9{\cdot}348\,(3)$ Å, $\beta = 119{\cdot}04\,(5)^\circ$, $V = 810\,(1)$ Å^3, $Z = 4$, $D_x = 1{\cdot}502$ Mg m^{-3}, $\lambda(\text{Mo } K\alpha) = 0{\cdot}71073$ Å, $\mu = 0{\cdot}337$ mm^{-1}, $F(000) = 384$, $T = 293$ K, $R = 0{\cdot}036$ for 894 observed reflections. A distorted tetrahedral geometry is observed about sulfur, with an O—S—O angle of 118·9 (1)° and a dihedral angle between planes N—S—C and O—S—O of 90·4 (1)°. The S—N distances [1·603 (4) Å] are shorter than those previously found in related compounds. The N atom is in a distorted pyramidal conformation, with the H atom located almost perpendicular to the heterocyclic ring. Molecules related by $\bar{x} + 1$, $\bar{y} + 1$, $\bar{z}$ form hydrogen-bonded dimers through a pair of N—H···O type hydrogen bonds [N···O 3·033 (5), H···O

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