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The Structure of Decussine; A New Indole Alkaloid

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Abstract. $C_{20}H_{19}N_3$, orthorhombic, $P2_12_12_1$, $a = 9{\cdot}391\,(1)$, $b = 9{\cdot}934\,(1)$, $c = 17{\cdot}144\,(2)$ Å, $V = 1599{\cdot}2$ Å^3, $Z = 4$, $D_x = 1{\cdot}251$ Mg m^{-3}, $T = 297$ K, μ(Mo $K\alpha$) = 0·07 mm^{-1}, final $R = 0{\cdot}061$ for 1579 observed reflections. Decussine is a pentacyclic indole alkaloid composed of a tetrahydro-β-carboline skeleton with an azepino ring attached to a pyridine ring. The compound has been isolated from the bark of an African plant (*Strychnos decussata*) and has a pronounced muscle-relaxant effect. The molecules crystallize in a herring-bone pattern with only van der Waals interactions between them.

Introduction. A new indole alkaloid, named decussine, has been isolated from the stem bark of *Strychnos decussata* (Pappe) Gilg. collection No. Lg 10 797 collected in Karawa, Kenya. The alkaloid was shown to have a pronounced muscle-relaxant effect both *in vivo* and *in vitro* and the molecular and crystal structure is reported here. The chemical and pharmacological work has been described by Rolfsen, Olaniyi, Sandberg & Kvick (1980).

A slightly elongated crystal of maximum dimension 0·2 mm crystallized from methanol was used for the data collection on a CAD-4 diffractometer with graphite-monochromatized Mo $K\alpha$ radiation and an $\omega/2\theta$ scan method. A preliminary film investigation indicated orthorhombic symmetry and $P2_12_12_1$ as the space group. The reflections in the octant hkl out to a maximal $\sin\theta/\lambda = 0{\cdot}64$ Å^{-1} were collected. Three reflections were monitored at regular intervals to provide a check on crystal and experimental stability. Only deviations expected from counting statistics were observed. A total of 2011 independent intensities were collected and corrected for background by a profile-analysis method (Lehmann & Larsen, 1974) and for Lorentz and polarization effects, but not for absorption ($\mu = 0{\cdot}07$ mm^{-1}). The structure was solved by direct methods with the program *MULTAN* (Declercq, Germain, Main & Woolfson, 1973) and refined by least squares minimizing the quantity $\sum w(F_o^2 - k^2|F_c|^2)$ with the program *UPALS*. The weights were $w = \sigma^{-2}(F_o^2)$, and $\sigma^2(F_o^2) = \sigma_c^2(F_o^2) + (0{\cdot}05F_o^2)^2$ where $\sigma_c(F_o^2)$ is based on counting statistics. The standard deviation of an observation of unit weight is $S = [\sum w(F_o^2 - k^2|F_c|^2)^2/(m-n)]^{1/2} = 0{\cdot}99$ where m is the total number of observations (1579 reflections with $F_o^2 > 0$) and n is the total number of parameters varied (284).

The 284 parameters included the scale factor, positional parameters for the 42 atoms, anisotropic thermal parameters for the 23 non-H atoms and 19 isotropic thermal parameters for the H atoms. The final agreement factors are $R(F^2) = \sum|F_o^2 - k^2|F_c|^2|/\sum|F_o^2| = 0{\cdot}061$; $R_w(F^2) = [\sum w(F_o^2 - k^2|F_c|^2)^2/\sum wF_o^4]^{1/2} = 0{\cdot}075$; $R(F^2) = 0{\cdot}085$ if the 432 reflections with $F_o^2 < 0$ were included.

The scattering factors for neutral H, C, and N atoms were taken from *International Tables for X-ray Crystallography* (1974). The programs used have been

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Table 1. *Atomic coordinates* ($\times 10^4$, *for* H $\times 10^3$) *and the equivalent isotropic temperature factors* B_{eq} (*Hamilton*, 1959), *with e.s.d.'s in parentheses*

	x	y	z	B_{eq} (Å^2)
N(1)	2271 (2)	367 (2)	3223 (1)	2·09 (5)
C(2)	2127 (3)	−909 (2)	2891 (1)	2·03 (5)
C(3)	1300 (3)	−2018 (2)	3209 (1)	2·22 (6)
N(4)	1436 (3)	−3187 (2)	2784 (1)	2·64 (6)
C(5)	2569 (4)	−3340 (3)	2205 (2)	3·04 (9)
C(6)	2681 (4)	−2154 (3)	1674 (2)	3·02 (8)
C(7)	2718 (3)	−916 (3)	2165 (1)	2·21 (6)
C(8)	3263 (3)	387 (3)	2020 (1)	2·18 (6)
C(9)	3955 (3)	993 (3)	1385 (2)	2·73 (8)
C(10)	4337 (3)	2318 (3)	1431 (2)	3·03 (9)
C(11)	4065 (3)	3068 (3)	2102 (2)	2·90 (8)
C(12)	3408 (3)	2511 (3)	2741 (2)	2·47 (7)
C(13)	2995 (3)	1165 (3)	2693 (1)	2·12 (6)
C(14)	347 (3)	−1916 (3)	3813 (1)	2·55 (7)
C(15)	−114 (3)	−739 (3)	4237 (1)	2·24 (6)
C(16)	−1460 (3)	−804 (4)	4613 (2)	2·91 (8)
C(17)	−1966 (4)	281 (5)	5015 (2)	3·50 (11)
C(18)	3228 (4)	−48 (4)	4544 (2)	2·82 (9)
C(19)	2128 (3)	690 (3)	4054 (1)	2·21 (6)
C(20)	620 (2)	488 (3)	4335 (1)	2·10 (6)
C(21)	−13 (3)	1507 (3)	4754 (1)	2·65 (8)
N(22)	−1295 (3)	1459 (3)	5101 (1)	3·25 (8)
C(23)	728 (4)	−4405 (3)	3042 (2)	3·34 (10)
H1(5)	232 (3)	−412 (3)	193 (1)	5·5 (7)
H2(5)	357 (5)	−351 (4)	248 (2)	8·9 (10)
H1(6)	354 (3)	−222 (2)	133 (2)	5·1 (6)
H2(6)	188 (4)	−214 (4)	127 (2)	9·0 (10)
H(9)	416 (3)	51 (3)	94 (2)	5·3 (7)
H(10)	481 (3)	275 (3)	98 (2)	6·1 (7)
H(11)	441 (3)	399 (3)	212 (2)	6·0 (7)
H(12)	323 (2)	302 (2)	321 (1)	3·0 (5)
H(14)	−19 (3)	−272 (3)	391 (1)	4·9 (6)
H(16)	−202 (3)	−167 (3)	456 (2)	6·1 (7)
H(17)	−285 (4)	19 (3)	525 (2)	6·8 (8)
H1(18)	299 (3)	−104 (3)	456 (1)	4·5 (6)
H2(18)	317 (3)	28 (3)	510 (2)	5·3 (6)
H3(18)	417 (4)	10 (3)	432 (2)	5·3 (7)
H(19)	232 (3)	160 (3)	409 (1)	3·7 (5)
H(21)	48 (3)	239 (3)	479 (1)	3·9 (6)
H1(23)	−35 (5)	−416 (4)	314 (2)	10·4 (11)
H2(23)	95 (5)	−518 (4)	270 (2)	9·1 (10)
H3(23)	98 (4)	−472 (3)	354 (2)	8·4 (10)

described by Lundgren (1979). Atomic coordinates are given in Table 1.*

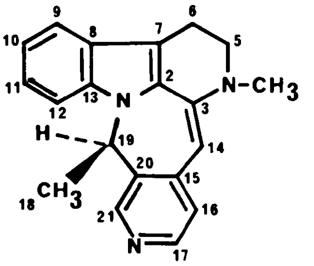

Fig. 1. The atomic numbering in decussine.

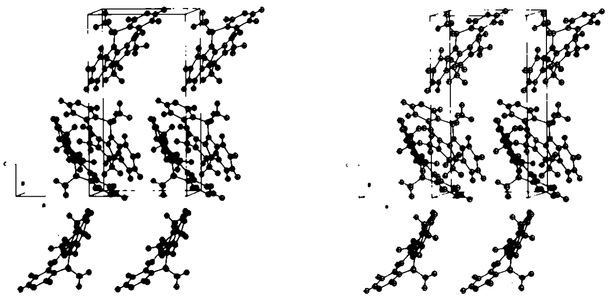

Fig. 2. A stereoscopic illustration of the packing. The N atoms are shown as filled circles.

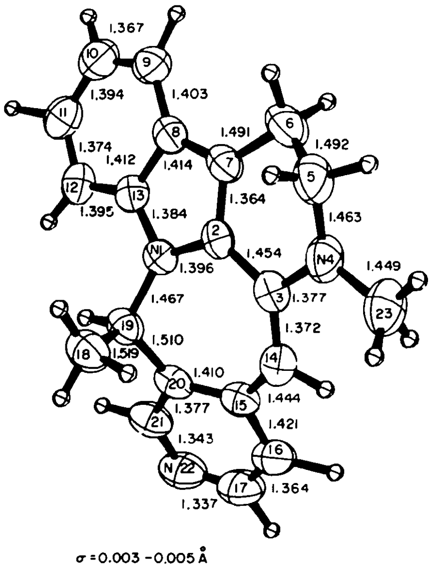

Fig. 3. The molecular bond distances in decussine (Å).

Discussion. The atomic notations and the molecular structure are illustrated in Figs. 1 and 2. The bond distances and torsional angles are given in Figs. 3 and 4. The molecule is numbered according to the biogenetic classification of indole alkaloids (Kompis, Hesse & Schmid, 1971). According to IUPAC and *Chemical Abstracts* rules the compound should be named (*R*)-5,6,7,13-tetrahydro-7,13-dimethyl-7,11,13a-triazabenzo[5,6]cyclohepta[1,2,3-*jk*]fluorene.

The molecules are packed in a herring-bone fashion and held together by van der Waals forces with no intermolecular non-H atom contacts shorter than 3·4 Å. The shortest H–H contact is 2·39 (5) Å [H(17)···H(12)]. The shortest heavy-atom–H contact is found between N(22) and H(19) [2·71 (3) Å].

The β-carboline skeleton is no longer planar as a result of the hydrogenation of its pyridine ring. The

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* Lists of structure factors, anisotropic thermal parameters for non-H atoms and bond distances and angles have been deposited with the British Library Lending Division as Supplementary Publication No. SUP 35911 (19 pp.). Copies may be obtained through The Executive Secretary, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

distances from the least-squares plane through the benzene ring of the β-carboline skeleton are given in Table 2.

The molecular structure shares features with the structure of akagerine (Dupont, Dideberg & Angenot, 1975), which also contains a tetrahydro-β-carboline ring attached to a perhydroazepino ring. Akagerine is, however, a tetracyclic molecule with –OH and –CHO groups added to the perhydroazepino ring. In akagerine the seven-membered ring is completely saturated whereas in decussine C(3)—C(14) [1·372 (4) Å] has a marked double-bond character.

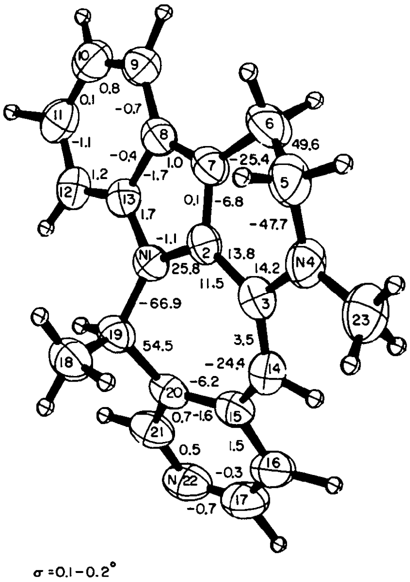

Fig. 4. The ring torsional angles (°).

Table 2. *Deviations of the heavy atoms* (Å) *from the least-squares plane through atoms* C(8), C(9), C(10), C(11), C(12) *and* C(13)

N(1)	−0·054 (4)
C(2)	−0·043 (5)
C(3)	−0·230 (6)
N(4)	−0·073 (6)
C(5)	0·542 (6)
C(6)	−0·023 (6)
C(7)	−0·012 (5)
C(8)	−0·002 (4)
C(9)	0·006 (5)
C(10)	−0·003 (5)
C(11)	−0·004 (5)
C(12)	0·008 (4)
C(13)	−0·005 (4)
C(14)	−0·666 (7)
C(15)	−1·096 (6)
C(16)	−1·960 (7)
C(17)	−2·417 (8)
C(18)	1·699 (6)
C(19)	0·271 (5)
C(20)	−0·752 (5)
C(21)	−1·287 (6)
N(22)	−2·120 (6)
C(23)	−0·171 (9)

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The Structure of 2,2′-Bipyridinium Bis(fluorosulfate)

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Abstract. $C_{10}H_{10}N_2^{2+}.2SO_3F^-$, triclinic, $P\bar{1}$, $a = 7{\cdot}771$ (2), $b = 8{\cdot}955$ (2), $c = 10{\cdot}839$ (3) Å, $\alpha = 96{\cdot}97$ (2), $\beta = 99{\cdot}26$ (2), $\gamma = 107{\cdot}98$ (2)°, $V = 696{\cdot}21$ Å^3, $Z = 2$, D_m (flotation) $= 1{\cdot}68$, $D_c = 1{\cdot}70$ Mg m^{-3}. The structure was solved by direct methods and refined to a final $R(F)$ value of 6·3% for 1485 independent reflections. The molecule consists of a diprotonated bipyridinium cation involved in hydrogen bonding with surrounding fluorosulfate anions [N···O distances are 2·728 (6) and 2·764 (6) Å]. The cation exhibits a *transoid* configuration (distortion of 31·5° from the ideal *trans* configuration). Orientational disorder occurs between the F and O atoms in the fluorosulfate anions.

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