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Structures of the Natural Alkaloids Vincadifformine and 8-Oxotabersonine

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Abstract

The structures of two indole alkaloids, vincadifformine [methyl (5′*R*,12a*R*,19′*R*)-2,3-didehydroaspidospermidine-3-carboxylate] and 8-oxotabersonine [methyl (5′*R*,12a*R*,19′*R*)-2,3,6,7-tetradehydro-8-oxoaspidospermidine-3-carboxylate] have been established and are compared. The main difference between the two molecules is in the conformation of the six-membered heterocyclic ring which exhibits a chair conformation in vincadifformine but presents a more flattened sofa conformation in 8-oxotabersonine as a result of the presence of a double bond and an oxo group.

Comment

The *Apocynaceae* family has been known for some time now to be a rich source of complex and intriguing indole alkaloids. In particular, the widespread occurrence of aspidospermine alkaloids has attracted considerable attention over the past several decades (Saxton, 1983). By far the most common structural feature that occurs throughout this subtype of indole alkaloids is that found in vincadifformine (1), a crystalline base existing in nature in both enantiomers, which is a member of a distinct group of aspidospermine-like alkaloids bearing an extra C atom in the form of a CO_2Me group (Plat, Le Men, Janot, Budzikiewicz, Wilson, Durham & Djerassi, 1962).

The crystal structures of naturally occurring and synthetic indoles embodying the same chromophore, for example ervafoline, epiervafoline and 19′-hydroxyervafolidine (Henriques, Kan, Chiaroni, Riche, Husson, Kan & Lounasmaa, 1982), dibromovobtusine (Lefebvre-Soubeyran, 1973) and cathovalinine (Chiaroni, Riche, Diatta, Andriamialisoa, Langlois & Potier, 1976), have been well established. As vincadifformine is the most abundant of all these alkaloids, it is surprising to discover that no X-ray investigations have been recorded to date.

Here we report the structures of vincadifformine and 8-oxotabersonine (2), a related alkaloid first isolated from *Amsonia elliptica* (Aimi, Asada, Sakai & Haginiwa, 1978) and subsequently from *Hazunta modesta* (Bui, Das & Potier, 1980). Table 1 gives the atomic coordinates of both structures while Fig. 1 shows the two molecules with the numbering scheme. Selected bond lengths, bond angles and torsion angles are listed in Table 2. The bond distances of (1) and (2) do not display any particular features, all being comparable with values in the literature (see for example Allen, Kennard, Watson, Brammer, Orpen & Taylor, 1987). Table 3 gives the conformational parameters of the non-aromatic rings of the two compounds.

The skeletons of vincadifformine and 8-oxotabersonine are quite similar, the main difference being found in

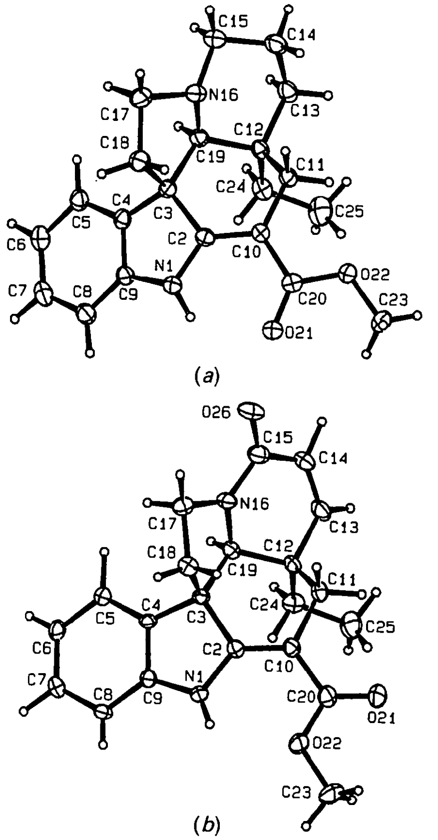

Fig. 1. (*a*) Vincadifformine and (*b*) 8-oxotabersonine; probability level 20%, H atoms not to scale.

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the six-membered heterocyclic ring *A*. In (2), the ring is flattened strongly (*cf.* Q_T in Table 3) by the presence of a conjugated system involving the double bond C13=C14, the C15—O26 carbonyl and the lone pair of N16, and assumes a $^5S_6$ conformation, while the same ring in (1) exhibits a chair conformation. At the same time, all the bonds are shortened and the bond angles enlarged in the C12, C13, C14, C15, N16 chain in (2) (see Table 2). In both structures, the indole ring (N1, C2, C3, C4, C9) is twisted $^2T_3$ and the C2, C3, C19, C12, C11, C10 ring has a $^5S_4$ conformation but the C3, C19, N16, C17, C18 ring is $^4T_3$ in (1) and $^4T_5$ in (2). In both structures, the H atom bonded to N1 makes an intermolecular hydrogen bond with one of the O atoms of the carbomethoxy group [N1···O 2.708-2.711 Å, N1—H1···O 122-127°] but whereas H1 is bonded to the carboxylic O atom in (1), it is the second O atom in (2) which is involved. The crystal packing of both structures is determined by residual forces and weak hydrogen bonds.

Experimental

Vincadifformine (1)

Crystal data

$C_{21}H_{26}N_2O_2$
$M_r = 338.45$
Orthorhombic
$P2_12_12_1$
$a = 10.090$ (2) Å
$b = 13.406$ (2) Å
$c = 13.625$ (2) Å
$V = 1843.0$ (5) Å^3
$Z = 4$
$D_x = 1.220$ Mg m^{-3}

Mo $K\alpha$ radiation
$\lambda = 0.71069$ Å
Cell parameters from 25 reflections
$\theta = 13\text{-}17°$
$\mu = 0.0734$ mm^{-1}
Room temperature
Prism
0.30 × 0.28 × 0.28 mm
Colourless

Data collection

Enraf-Nonius CAD-4 diffractometer
$\omega/2\theta$ scans
Absorption correction: none
2393 measured reflections
2393 independent reflections
1400 observed reflections [$I > 2\sigma(I)$]

$\theta_{max} = 17°$
$h = 0 \rightarrow 13$
$k = 0 \rightarrow 17$
$l = 0 \rightarrow 17$
1 standard reflection frequency: 180 min intensity variation: none

Refinement

Refinement on F
Final $R = 0.035$
$wR = 0.039$
$S = 1.31$
1400 reflections
331 parameters
All H-atom parameters refined
$w = 2F_o\text{Lp}/[\sigma(I_o)^2+(0.03I_o)^2]^{1/2}$
$(\Delta/\sigma)_{max} = 0.01$
$\Delta\rho_{max} = 0.15$ e Å^{-3}

Extinction correction: Stout & Jensen (1968)
Extinction coefficient: $g = 8.2$ (8) × 10^{-6}
Atomic scattering factors from *International Tables for X-ray Crystallography* [1974, Vol. IV, Tables 2.2A, 2.3.1 (O, N, C) and 2.2C (H)]

8-Oxotabersonine (2)

Crystal data

$C_{21}H_{22}N_2O_3.\frac{1}{2}C_4H_8O_2$
$M_r = 394.47$
Tetragonal
$P4_32_12$
$a = 10.677$ (1) Å
$c = 35.268$ (3) Å
$V = 4020.5$ (6) Å^3
$Z = 8$
$D_x = 1.303$ Mg m^{-3}

Mo $K\alpha$ radiation
$\lambda = 0.71069$ Å
Cell parameters from 25 reflections
$\theta = 12\text{-}13°$
$\mu = 0.0836$ mm^{-1}
Room temperature
Prism
0.28 × 0.28 × 0.28 mm
Colourless

Data collection

Enraf-Nonius CAD-4 diffractometer
$\omega/2\theta$ scans
Absorption correction: none
4316 measured reflections
2740 independent reflections
1469 observed reflections [$I > \sigma(I)$]

$R_{int} = 0.027$
$\theta_{max} = 27.5°$
$h = 0 \rightarrow 13$
$k = 0 \rightarrow 13$
$l = -45 \rightarrow 45$
1 standard reflection frequency: 180 min intensity variation: none

Refinement

Refinement on F
Final $R = 0.060$
$wR = 0.056$
$S = 1.78$
1469 reflections
328 parameters
$w = 2F_o\text{Lp}/[\sigma(I_o)^2+(0.03I_o)^2]^{1/2}$
$(\Delta/\sigma)_{max} = 0.03$
$\Delta\rho_{max} = 0.30$ e Å^{-3}

Extinction correction: Stout & Jensen (1968)
Extinction coefficient: $g = 3.3$ (3) × 10^{-6}
Atomic scattering factors from *International Tables for X-ray Crystallography* [1974, Vol. IV, Tables 2.2A, 2.3.1 (O, N, C) and 2.2C (H)]

The positions of seven H atoms were calculated but the H atoms of the disordered ethyl acetate group were not introduced.

Table 1. *Fractional atomic coordinates and equivalent isotropic thermal parameters* (Å^2)

$$U_{eq} = \tfrac{1}{3}\Sigma_i\Sigma_j U_{ij}a_i^*a_j^*\mathbf{a}_i.\mathbf{a}_j.$$

	x	y	z	U_{eq}
(1)				
O21	0.2210 (2)	0.4495 (1)	0.4570 (1)	0.0586 (7)
O22	0.4095 (2)	0.3858 (1)	0.5174 (1)	0.0549 (6)
N1	0.1150 (2)	0.3637 (2)	0.2951 (2)	0.0522 (7)
N16	0.4306 (2)	0.1530 (2)	0.1738 (2)	0.0579 (8)
C2	0.2331 (3)	0.3115 (2)	0.3019 (2)	0.0442 (8)
C3	0.2333 (3)	0.2357 (2)	0.2194 (2)	0.0440 (8)
C4	0.0862 (3)	0.2251 (2)	0.2010 (2)	0.0472 (8)
C5	0.0129 (3)	0.1567 (2)	0.1464 (2)	0.0560 (10)
C6	−0.1221 (3)	0.1716 (2)	0.1358 (2)	0.0652 (12)
C7	−0.1828 (3)	0.2530 (3)	0.1763 (2)	0.0678 (12)
C8	−0.1116 (3)	0.3232 (2)	0.2298 (2)	0.0587 (10)
C9	0.0226 (3)	0.3063 (2)	0.2422 (2)	0.0481 (9)
C10	0.3270 (3)	0.3171 (2)	0.3721 (2)	0.0413 (8)
C11	0.4317 (3)	0.2366 (2)	0.3747 (2)	0.0458 (8)
C12	0.3698 (3)	0.1339 (2)	0.3469 (2)	0.0454 (8)
C13	0.4749 (3)	0.0500 (2)	0.3501 (2)	0.0591 (10)
C14	0.5781 (3)	0.0546 (2)	0.2692 (3)	0.0687 (12)
C15	0.5151 (3)	0.0657 (2)	0.1697 (3)	0.0707 (11)
C17	0.3783 (3)	0.1932 (2)	0.0831 (2)	0.0630 (10)
C18	0.2985 (3)	0.2794 (2)	0.1231 (2)	0.0568 (10)

C19	0.3199 (3)	0.1430 (2)	0.2407 (2)	0.0455 (7)
C20	0.3114 (3)	0.3896 (2)	0.4503 (2)	0.0451 (9)
C23	0.4010 (3)	0.4556 (3)	0.5976 (2)	0.0711 (12)
C24	0.2542 (3)	0.1068 (2)	0.4144 (2)	0.0576 (10)
C25	0.2843 (4)	0.1102 (3)	0.5247 (2)	0.0893 (14)
(2)				
O21	0.1561 (4)	−0.6929 (3)	0.0607 (1)	0.0747 (16)
O22	0.1093 (4)	−0.5029 (4)	0.0388 (1)	0.0692 (14)
O26	0.2595 (4)	−0.5078 (4)	0.26117 (8)	0.0764 (15)
N1	0.1689 (4)	−0.2968 (4)	0.0799 (1)	0.0444 (14)
N16	0.2717 (4)	−0.4217 (4)	0.2024 (1)	0.0498 (15)
C2	0.1912 (4)	−0.3933 (5)	0.1054 (1)	0.0380 (15)
C3	0.2330 (4)	−0.3324 (4)	0.1418 (1)	0.0361 (16)
C4	0.2715 (5)	−0.2030 (4)	0.1291 (1)	0.0366 (15)
C5	0.3334 (5)	−0.1071 (5)	0.1473 (1)	0.0438 (18)
C6	0.3433 (5)	0.0075 (5)	0.1288 (2)	0.0504 (20)
C7	0.2919 (5)	0.0261 (5)	0.0935 (1)	0.0527 (18)
C8	0.2328 (5)	−0.0717 (5)	0.0747 (1)	0.0473 (19)
C9	0.2227 (5)	−0.1858 (5)	0.0929 (1)	0.0386 (17)
C10	0.1842 (5)	−0.5179 (5)	0.1006 (1)	0.0428 (16)
C11	0.2368 (5)	−0.5969 (5)	0.1328 (1)	0.0468 (18)
C12	0.3590 (5)	−0.5396 (5)	0.1477 (1)	0.0448 (16)
C13	0.4134 (6)	−0.6196 (5)	0.1792 (2)	0.0608 (20)
C14	0.3929 (6)	−0.6018 (5)	0.2153 (2)	0.0655 (22)
C15	0.3055 (5)	−0.5073 (6)	0.2290 (1)	0.0617 (21)
C17	0.1765 (5)	−0.3281 (5)	0.2081 (1)	0.0545 (20)
C18	0.1191 (5)	−0.3214 (5)	0.1692 (1)	0.0501 (16)
C19	0.3293 (5)	−0.4111 (5)	0.1653 (1)	0.0410 (16)
C20	0.1498 (5)	−0.5811 (5)	0.0660 (2)	0.0527 (19)
C23	0.0841 (7)	−0.5594 (6)	0.0024 (2)	0.0920 (28)
C24	0.4585 (5)	−0.5226 (6)	0.1169 (1)	0.0522 (19)
C25	0.4891 (6)	−0.6414 (6)	0.0945 (2)	0.0812 (27)
O1*S*	0.5456 (6)	0.54561	0.00000	0.1998 (43)
O2*S*	0.3830 (10)	0.5010 (20)	0.0110 (5)	0.396 (12)
C3*S*	0.4279 (8)	0.5880 (10)	0.0051 (3)	0.2602 (61)
C4*S*	0.3950 (10)	0.7000 (10)	−0.0081 (3)	0.1953 (57)

Table 2. *Selected bond lengths* (Å), *bond angles* (°) *and torsion angles* (°)

	(1)	(2)
O26—C15		1.210 (6)
O21—C20	1.219 (3)	1.343 (7)
O22—C20	1.348 (3)	1.444 (7)
O22—C23	1.441 (3)	1.236 (5)
N1—C2	1.385 (3)	1.388 (6)
N1—C9	1.407 (3)	1.395 (6)
N16—C15	1.449 (3)	1.359 (6)
N16—C17	1.448 (3)	1.440 (6)
N16—C19	1.448 (3)	1.450 (5)
C2—C3	1.515 (3)	1.507 (5)
C2—C10	1.348 (4)	1.343 (7)
C3—C4	1.512 (4)	1.509 (6)
C3—C18	1.580 (3)	1.558 (6)
C3—C19	1.547 (4)	1.565 (6)
C10—C11	1.511 (4)	1.522 (6)
C10—C20	1.451 (3)	1.442 (7)
C11—C12	1.559 (3)	1.534 (7)
C12—C13	1.546 (4)	1.517 (7)
C12—C19	1.537 (3)	1.539 (7)
C12—C24	1.529 (4)	1.530 (6)
C13—C14	1.518 (4)	1.306 (9)
C14—C15	1.505 (5)	1.457 (8)
C17—C18	1.510 (3)	1.504 (5)
C24—C25	1.534 (3)	1.530 (8)
C20—O22—C23	116.5 (2)	115.8 (5)
C2—N1—C9	109.1 (2)	110.3 (4)
C17—N16—C19	106.9 (2)	111.8 (4)
C15—N16—C19	113.8 (2)	124.3 (4)
C15—N16—C17	118.8 (3)	123.9 (4)
N1—C2—C10	128.6 (3)	130.1 (4)
N1—C2—C3	106.9 (2)	106.4 (4)
C3—C2—C10	124.2 (3)	123.5 (4)
C2—C3—C19	113.6 (2)	114.5 (4)
C2—C3—C18	111.6 (2)	109.3 (3)
C2—C3—C4	100.6 (2)	102.9 (3)
C18—C3—C19	102.6 (2)	103.0 (3)
C4—C3—C19	120.7 (2)	118.0 (4)
C4—C3—C18	107.8 (2)	109.1 (4)
C3—C4—C9	108.3 (2)	106.9 (4)
C5—C4—C9	119.2 (3)	120.6 (4)
N1—C9—C8	128.4 (3)	129.0 (4)
N1—C9—C4	109.3 (3)	110.1 (4)
C2—C10—C20	118.8 (3)	125.8 (5)
C2—C10—C11	117.9 (3)	115.7 (4)
C11—C10—C20	122.5 (3)	117.8 (4)
C10—C11—C12	110.2 (2)	110.4 (4)
C11—C12—C24	111.7 (2)	113.2 (3)
C11—C12—C19	106.9 (2)	108.6 (4)
C11—C12—C13	111.2 (2)	110.6 (4)
C19—C12—C24	109.6 (2)	108.9 (4)
C13—C12—C24	109.5 (2)	108.7 (4)
C13—C12—C19	108.0 (2)	106.6 (4)
C12—C13—C14	114.9 (2)	124.6 (5)
C13—C14—C15	111.6 (3)	122.1 (6)
N16—C15—C14	107.1 (2)	114.0 (4)
N16—C17—C18	99.8 (2)	101.2 (4)
C3—C18—C17	103.8 (2)	104.1 (4)
C3—C19—C12	115.2 (2)	113.6 (3)
N16—C19—C12	110.3 (2)	112.4 (4)
N16—C19—C3	104.1 (2)	103.9 (3)
O22—C20—C10	113.1 (2)	113.3 (5)
O21—C20—C10	125.3 (3)	125.3 (5)
O21—C20—O22	121.6 (2)	121.4 (5)
C12—C24—C25	115.5 (3)	114.6 (4)
C9—N1—C2—C3	21.5 (3)	15.5 (5)
N1—C2—C3—C4	−23.6 (3)	−18.3 (4)
C10—C2—C3—C19	20.3 (4)	30.2 (6)
C3—C2—C10—C11	−6.3 (4)	−7.3 (7)
C18—C3—C19—N16	10.3 (3)	−6.8 (4)
C2—C3—C19—C12	10.6 (3)	−2.7 (5)
C19—C3—C18—C17	18.2 (3)	28.4 (4)
C2—C10—C11—C12	−36.7 (4)	−41.0 (6)
C10—C11—C12—C19	63.0 (3)	64.7 (5)
C10—C11—C12—C24	−56.8 (3)	−56.4 (5)
C11—C12—C19—C3	−49.6 (3)	−41.2 (5)
C13—C12—C19—N16	−51.8 (3)	−42.7 (5)
C19—C12—C13—C14	46.7 (3)	24.2 (8)
C12—C13—C14—C15	−50.0 (4)	5.2 (10)
C13—C14—C15—N16	55.2 (3)	−15.0 (8)
C14—C15—N16—C19	−65.1 (3)	−8.8 (7)
N16—C17—C18—C3	−39.8 (3)	−39.2 (4)
C17—N16—C19—C3	−37.5 (3)	−19.0 (5)
C15—N16—C19—C12	65.1 (3)	39.3 (6)
C19—N16—C17—C18	49.1 (3)	37.2 (5)

Table 3. *Conformation of the non-aromatic rings* (Å^2, °) *according to Cremer & Pople* (1975)

Ring		(1)	(2)
A (N16,C15,C14,C13,C12,C19)	Q_T	0.562 (3)	0.405 (5)
	φ	9.6 (19)	−79.3 (9)
	θ	9.0 (3)	66.1 (8)
B (C2,C3,C19,C12,C11,C10)	Q_T	0.574 (3)	0.597 (5)
	φ	−160.9 (3)	−150.9 (5)
	θ	65.2 (3)	71.1 (4)
C (C3,C19,N16,C17,C18)	Q_T	0.444 (3)	0.379 (5)
	φ	−83.9 (4)	−60.4 (7)
D (N1,C2,C3,C4,C9)	Q_T	0.226 (3)	0.174 (4)
	φ	−129.1 (7)	−123.7 (16)

The title componds were extracted from *Apocynacae* plants (1) and *Amsonia elliptica* and *Hazunta modesta* (2) and crystals obtained from *n*-hexane and ethyl acetate, respectively. 8-Oxotabersonine co-crystallizes with ethyl acetate in the ratio

2:1, with a disordered solvate molecule at the $(x,x,0)$ position on the twofold axis. The refinement of the disordered ethyl acetate molecule gave some trouble; several attempts were made to interpret the confused difference map and to refine the six independent heavy atoms with population factors of 0.5, but there were always very high correlations between various parameters and an unacceptable molecular geometry. The best interpretation was to place the bridge O atom O1*S* on the twofold axis and to consider the C atoms of the acetyl and ethyl groups to be equivalent; in this interpretation, the carboxyl O atom O2*S* is the only atom with a population factor of 0.5. At the end of the refinement, the geometry of the group was quite acceptable but nevertheless, we considered the inclusion of the H atoms of the ethyl acetate molecule in the model to be of little significance.

Data collection and cell refinement: Enraf-Nonius software. Data reduction and structure refinement: *SDP-Plus* (Frenz, 1983). Structure solution: *MULTAN*11/82 (Main, Fiske, Hull, Lessinger, Germain, Declercq & Woolfson, 1982). Software used to prepare material for publication: *PARST* (Nardelli, 1983) and *ORTEP*II (Johnson, 1976).

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Lists of structure factors, anisotropic thermal parameters, H-atom coordinates and complete geometry have been deposited with the British Library Document Supply Centre as Supplementary Publication No. SUP 71227 (36 pp.). Copies may be obtained through The Technical Editor, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England. [CIF reference: NA1036]

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Structure of (±)-Tetrahydropalmatine

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Abstract

In 5,8,13,13a-tetrahydro-2,3,9,10-tetramethoxy-6*H*-dibenzo[*a*,*g*]quinolizine, according to the puckering parameters [Cremer & Pople (1975). *J. Am. Chem. Soc.* **97**, 1354–1558] the *trans* fused ring *B* [Q = 0.518 (5) Å, φ = 145.4 (7)°, θ = 51.6 (6)°] and ring *C* [Q = 0.529 (4) Å, φ = 328.2 (5)°, θ = 48.2 (4)°] of the quinolizine moiety both have almost perfect half-chair conformations. The mean value of the three C—N—C angles is 110.0 (3)° indicating sp^3 hybridization of the N atom. Molecules are held together by van der Waals interactions.

Comment

Tetrahydropalmatine is a tetrahydroprotoberberine alkaloid previously isolated from *Corydalis aurea*, *C. lutea* and *C. ochroleuca* (Glasby, 1975). We have isolated the same alkaloid from the roots of *C. cava*, as well as from the overground parts and roots of *C. solida* collected at the Fruška Gora mountain (Vojvodina). Isolation was performed as described by Gašić, Popović & Dragutinović (1985). The dihedral angle between the phenyl rings *A* and *D* is 25.8 (1)° as compared to 33.8 (1)° in isocorypalmine (Ribár, Radivojević, Gašić, Kanyó & Golič, 1992) and 36.7 (1)° in corydaline (Ribár, Lazar, Radivojević, Engel, Gašić & Kanyó, 1992). The O3—C19 bond is perpendicular to the plane of phenyl ring *D*, while the O4—C20 bond is in the plane of the same ring. The O1—C21 and O2—C18 bonds are in the plane of phenyl ring *A*, as indicated