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Structure of Senecionine, a Pyrrolizidine Alkaloid

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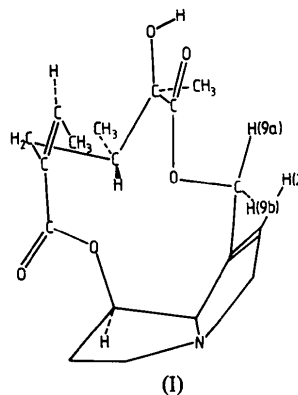
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Abstract. $C_{18}H_{25}NO_5$, $M_r = 335.4$, orthorhombic, space group $P2_12_12_1$, $a = 9.072$ (1), $b = 17.236$ (1), $c = 11.224$ (1) Å, $U = 1755.0$ (2) Å³, $Z = 4$, $D_m = 1.266$ (3), $D_c = 1.269$ Mg m⁻³, $F(000) = 720$, $\mu(Cu K\alpha) = 0.67$ mm⁻¹. The structure was solved by direct methods with diffractometer data measured with Cu $K\alpha$ radiation, and full-matrix least-squares refinement converged at $R = 0.046$ for 1694 observed reflections. With respect to the pyrrolizidine nucleus and the primary-ester system, senecionine adopts a conformation similar to the one proposed on NMR grounds, but differs at the α,β -unsaturated ester grouping; the pucker of the pyrrolizidine nucleus is *exo* and the ester carbonyl bonds are antiparallel. Inter-molecular hydrogen bonds involving the hydroxyl substituent and the N atoms, $N \cdots O$ 2.827 (4) Å, link the molecules into helices extending along the c axis.

Introduction. Senecionine (I) is a typical hepatotoxic macrocyclic pyrrolizidine alkaloid. It is one of the longest known and most commonly encountered alkaloids in the *Senecio* species, its chemical structure being established by Kropman & Warren (1949). A conformational structure (I) has been proposed for senecionine (Culvenor, 1966; Bull, Culvenor & Dick, 1968), based largely on inferences from NMR measurements. The present analysis, which is part of an investigation of stereochemical aspects of pyrrolizidine alkaloids, was performed in order to compare the proposed conformation with the crystal structure.



Experimental. Weissenberg photographs showed that the prismatic crystals grown from acetone were orthorhombic and systematic extinctions indicated the space group $P2_12_12_1$. Cell parameters were determined by least squares from 2θ values measured for 25 strong reflections with Cu $K\alpha$ radiation ($\lambda = 1.5418$ Å) on a diffractometer. The crystal density was determined by flotation.

Integrated intensities were measured with Cu $K\alpha$ radiation (graphite-crystal monochromator, $\lambda = 1.5418$ Å) from a crystal ca $0.20 \times 0.25 \times 0.45$ mm aligned on a Rigaku – AFC four-circle diffractometer with the longest crystal axis c approximately parallel to the diffractometer ϕ axis. The intensities were recorded by an $\omega - 2\theta$ scan, 2θ scan rate 2° min^{-1} , scan range ($\Delta\omega$) $1.2^\circ + 0.5^\circ \tan\theta$ and 10s stationary background

counts. Three reference reflections monitored every 50 reflections showed no significant variation in intensity during the data-collection period. Of the 1718 non-equivalent terms measured to a 2θ maximum of 130° ,

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

	x	y	z	$B_{eq}(\text{\AA}^2)$
C(1)	-2938 (4)	41 (2)	4982 (3)	2.65 (13)
C(2)	-3930 (4)	324 (2)	5690 (3)	3.41 (16)
C(3)	-3965 (5)	1195 (2)	5676 (4)	3.64 (17)
N(4)	-2920 (3)	1397 (1)	4711 (3)	2.47 (11)
C(5)	-1821 (4)	1998 (2)	5015 (4)	3.16 (16)
C(6)	-393 (4)	1715 (2)	4455 (4)	2.86 (15)
C(7)	-485 (4)	838 (2)	4611 (3)	2.37 (12)
C(8)	-2105 (4)	678 (2)	4355 (3)	2.38 (13)
C(9)	-2734 (5)	-799 (2)	4674 (4)	3.59 (16)
O(10)	-1419 (3)	-1135 (1)	5215 (2)	3.29 (10)
C(11)	-1608 (4)	-1481 (2)	6285 (3)	2.70 (14)
C(12)	-157 (4)	-1795 (2)	6766 (3)	2.78 (14)
C(13)	1024 (4)	-1138 (2)	6793 (3)	2.83 (14)
C(14)	497 (4)	-470 (2)	7616 (3)	2.79 (13)
C(15)	1370 (4)	266 (2)	7409 (3)	2.57 (13)
C(16)	1228 (3)	599 (2)	6186 (3)	2.36 (13)
O(17)	-197 (2)	616 (1)	5849 (2)	2.36 (9)
O(20)	-2772 (3)	-1530 (2)	6763 (2)	3.99 (12)
C(21)	330 (5)	-2475 (2)	5968 (4)	3.94 (14)
O(22)	-374 (3)	-2115 (3)	7915 (1)	3.49 (12)
C(23)	2538 (5)	-1431 (3)	7177 (5)	4.64 (12)
C(25)	2243 (4)	584 (2)	8223 (3)	3.27 (15)
C(26)	3172 (5)	1303 (3)	8082 (5)	4.51 (21)
O(27)	2225 (3)	804 (2)	5561 (2)	3.38 (11)

* Calculated from the refined anisotropic thermal parameters (deposited), $B_{eq} = 8\pi^2 U_{eq}$.

Table 2. H-atom coordinates ($\times 10^3$) and isotropic temperature factors with e.s.d.'s in parentheses

The atoms are given the same numbering as the atom to which they are bonded.

	x	y	z	$B_{iso}(\text{\AA}^2)$
H(2)	-480 (6)	1 (3)	610 (4)	7.1 (12)
H(3a)	-365 (5)	145 (3)	645 (5)	5.6 (11)
H(3b)	-498 (5)	138 (2)	551 (4)	4.1 (9)
H(5a)	-186 (6)	211 (3)	594 (5)	6.7 (13)
H(5b)	-203 (5)	251 (3)	472 (4)	5.3 (10)
H(6a)	29 (5)	195 (2)	477 (4)	3.9 (10)
H(6b)	-42 (4)	179 (2)	347 (4)	3.5 (8)
H(7)	21 (3)	55 (2)	414 (3)	1.6 (6)
H(8)	-197 (5)	60 (2)	344 (4)	3.9 (9)
H(9a)	-253 (6)	-85 (3)	370 (5)	7.8 (14)
H(9b)	-367 (5)	-110 (3)	497 (4)	4.7 (10)
H(13)	112 (4)	-93 (2)	604 (3)	2.6 (7)
H(14a)	64 (4)	-65 (2)	846 (4)	3.5 (8)
H(14b)	-65 (5)	-36 (2)	748 (4)	4.2 (9)
H(21a)	123 (7)	-273 (3)	637 (5)	7.4 (5)
H(21b)	55 (6)	-235 (3)	514 (6)	7.4 (5)
H(21c)	-56 (6)	-287 (3)	586 (5)	7.4 (5)
H(22)	-75 (6)	-185 (3)	839 (5)	5.8 (14)
H(23a)	313 (6)	-179 (4)	663 (5)	7.4 (5)
H(23b)	327 (7)	-108 (4)	725 (5)	7.4 (5)
H(23c)*	209	-169	797	
H(25)	235 (5)	31 (3)	902 (4)	5.4 (11)
H(26a)	447 (6)	109 (3)	833 (5)	7.4 (5)
H(26b)	310 (7)	159 (4)	865 (6)	7.4 (5)
H(26c)	303 (7)	167 (4)	749 (5)	7.4 (5)

* Coordinates were calculated for this atom.

the 1694 for which $|F_o| > 2\sigma|F_o|$ were used for the structure refinement. The intensities were not corrected for absorption. The scattering factors for O, N and C were from Cromer & Mann (1968), for H from Stewart, Davidson & Simpson (1965). Anomalous-dispersion corrections were made for the non-H atoms with the values of Cromer & Liberman (1970).

The structure was solved by direct methods with *SHELX 76* (Sheldrick, 1976). After full-matrix least-squares refinement with anisotropic temperature factors, the sites of 24 H atoms were located on the subsequent difference map. Final refinement, in which positional coordinates and isotropic temperature factors of the H atoms were allowed to vary, converged at $R = 0.046$ and $R_w = 0.054$. The function minimized was $\sum w(|F_o| - |F_c|)^2$ with weights $(\sigma^2|F_o| + 5 \times 10^{-5}|F_o|^2)^{-1}$. The mean parameter shift-to-error ratios at convergence were 0.08:1 for the H atoms and 0.03:1 for all other parameters. The largest peaks on the final difference map were of heights 0.22 and -0.28 e \AA^{-3} . Final atom coordinates are given in Tables 1 and 2,* Fig. 1 which contains the atom numbering and Fig. 2 which shows the molecular packing have been prepared from the output of *ORTEP* (Johnson, 1965).

* Lists of structure amplitudes, anisotropic thermal parameters, and intermolecular separations have been deposited with the British Library Lending Division as Supplementary Publication No. SUP 36891 (20 pp.). Copies may be obtained through The Executive Secretary, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

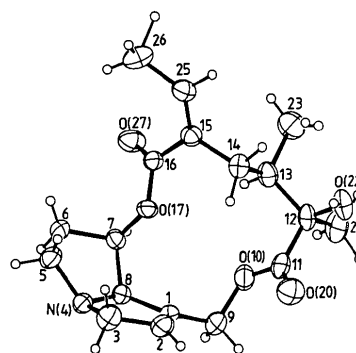


Fig. 1. A perspective view of the molecule with thermal ellipsoids scaled to 40% probability. The C atoms are denoted by numerals only.

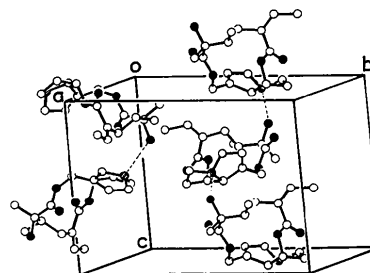


Fig. 2. The molecular packing in the crystal.

Discussion. The molecular conformation of senecionine is illustrated in Fig. 1, bond lengths and angles are given in Table 3 and torsional angles in Table 4. The crystal structure is isostructural with that of retrorsine* (Coleman, Coucourakis & Pretorius, 1980) which differs from senecionine in having a CH₂-OH grouping at C(12) in place of the CH₃ substituent.

In general, there is a fairly close resemblance to the conformation (I) proposed earlier in regard to the pyrrolizidine nucleus and the section around the allylic ester grouping, but the secondary-ester group is twisted and the adjacent C=C bond is more nearly *cis* than *trans* to the carbonyl group.

The pyrrolizidine ring is *exo*-buckled at an angle of 35.3 (4)°. *Exo*-buckling has also been observed in other retronecine alkaloids, and the puckering angles in crystals of retrorsine and jacobine (Pérez-Salazar, Cano & García-Blanco, 1978) have the respective values of 36.8 (8) and 36.9 (4)°. These values are in accord with the conclusion from NMR data that retronecine esters in solution are *exo*-buckled to about

* Transformations of the atomic coordinates given in Tables 1 and 2 are according to the expressions $x = -\frac{1}{2} + z'$, $y = -x'$, $z = 1 - y'$, where x' , y' , z' refer to the atomic coordinates derived for retrorsine.

Table 3. Bond lengths (Å) and angles (°)

The e.s.d.'s for the angles are 0.3° and for the lengths are given in parentheses.

C(1)-C(2)	1.296 (5)	C(11)-C(12)	1.522 (5)
C(1)-C(8)	1.507 (5)	C(11)-O(20)	1.187 (4)
C(1)-C(9)	1.500 (5)	C(12)-C(13)	1.559 (5)
C(2)-C(3)	1.502 (5)	C(12)-C(21)	1.540 (5)
C(3)-N(4)	1.481 (5)	C(12)-O(22)	1.416 (4)
N(4)-C(5)	1.478 (4)	C(13)-C(14)	1.552 (5)
N(4)-C(8)	1.497 (4)	C(13)-C(23)	1.526 (6)
C(5)-C(6)	1.520 (5)	C(14)-C(15)	1.513 (5)
C(6)-C(7)	1.524 (5)	C(15)-C(16)	1.493 (5)
C(7)-C(8)	1.523 (5)	C(15)-C(25)	1.328 (5)
C(7)-O(17)	1.465 (4)	C(16)-O(17)	1.347 (3)
C(9)-O(10)	1.459 (5)	C(16)-O(27)	1.198 (4)
O(10)-C(11)	1.352 (4)	C(25)-C(26)	1.507 (6)
C(2)-C(1)-C(8)	111.1	C(12)-C(11)-O(20)	125.7
C(2)-C(1)-C(9)	126.2	C(11)-C(12)-C(13)	110.0
C(8)-C(1)-C(9)	122.2	C(11)-C(12)-C(21)	108.2
C(1)-C(2)-C(3)	122.6	C(11)-C(12)-O(22)	110.0
C(2)-C(3)-N(4)	103.2	C(13)-C(12)-C(21)	111.5
C(3)-N(4)-C(5)	115.3	C(13)-C(12)-O(22)	111.1
C(3)-N(4)-C(8)	108.5	C(21)-C(12)-O(22)	105.9
C(5)-N(4)-C(8)	108.0	C(12)-C(13)-C(14)	109.8
N(4)-C(5)-C(6)	104.7	C(12)-C(13)-C(23)	112.6
C(5)-C(6)-C(7)	102.9	C(14)-C(13)-C(23)	110.8
C(6)-C(7)-C(8)	102.2	C(13)-C(14)-C(15)	111.7
C(6)-C(7)-O(17)	111.0	C(14)-C(15)-C(16)	114.7
C(8)-C(7)-O(17)	107.7	C(14)-C(15)-C(25)	123.6
C(1)-C(8)-N(4)	103.3	C(16)-C(15)-C(25)	121.7
C(1)-C(8)-C(7)	121.9	C(15)-C(15)-O(17)	110.4
N(4)-C(8)-C(7)	106.0	C(15)-C(16)-O(27)	125.9
C(1)-C(9)-O(10)	112.8	O(17)-C(16)-O(27)	123.6
C(9)-O(10)-C(11)	116.2	C(7)-O(17)-C(16)	116.3
O(10)-C(11)-C(12)	111.2	C(15)-C(25)-C(26)	126.9
O(10)-C(11)-O(20)	123.1		

Table 4. Torsional angles (°); e.s.d.'s are about 0.4°

Atoms are represented by their identification number. Corresponding values for retrorsine and jacobine are included.

	Senecionine	Retrorsine	Jacobine
Pyrrolizidine nucleus			
1-2-3-4	6.5	6.4	7.1
1-8-4-3	10.7	9.4	8.2
1-8-4-5	136.4	135.2	133.3
1-8-7-6	-146.5	-145.1	-145.7
1-8-7-17	-29.5	-28.2	-29.3
2-3-4-5	-131.8	-131.7	-129.8
2-3-4-8	-10.6	-9.8	-9.2
2-1-8-4	-6.8	-5.5	-4.0
2-1-8-7	112.0	112.4	114.7
3-4-5-6	139.4	141.1	140.4
3-4-8-7	-118.5	-119.0	-119.5
3-2-1-8	0.2	0.5	-2.0
3-2-1-9	-172.2	-172.5	-171.4
4-5-6-7	-36.0	-37.0	-37.4
4-8-1-9	165.9	166.7	166.1
4-8-7-6	-29.0	-28.6	-28.1
4-8-7-17	88.0	88.3	88.2
5-4-8-7	7.1	6.7	5.6
5-6-7-8	39.6	40.9	39.7
5-6-7-17	-74.9	-73.7	-75.6
6-5-4-8	17.9	18.7	19.6
7-8-1-9	-75.3	-75.4	-75.3
Esterifying acid moiety			
1-9-10-11	92.4	91.4	88.4
2-1-9-10	-108.3	-107.8	-106.2
6-7-17-16	-79.1	-81.5	-82.4
7-17-16-15	-172.6	-173.9	-175.8
7-17-16-27	5.3	5.9	4.5
8-1-9-10	80.1	81.0	85.6
8-7-17-16	169.8	168.0	167.2
9-10-11-12	-179.0	-179.7	179.2
9-10-11-20	1.5	2.4	0.4
10-11-12-13	54.0	54.3	51.8
10-11-12-21	-68.1	-65.5	-71.1
10-11-12-22	176.8	178.9	174.3
11-12-13-14	60.5	60.0	63.3
11-12-13-23	-175.6	-178.0	-172.4
12-13-14-15	-163.9	-164.1	-159.3
13-12-11-20	-126.5	-127.7	-129.5
13-14-15-16	62.5	64.0	59.8
13-14-15-25	-115.1	-105.3	
14-13-12-21	-179.4	178.3	-177.2
14-13-12-22	-61.5	-62.0	-58.8
14-15-16-17	46.4	46.2	50.7
14-15-16-27	-131.5	-133.6	-129.6
14-15-25-26	178.6	175.8	
15-14-13-23	71.1	73.8	75.9
16-15-25-26	1.2	2.9	
17-16-15-25	-136.0	-140.3	
20-11-12-21	111.4	112.5	107.6
20-11-12-22	-3.8	-3.2	-7.0
21-12-13-23	-55.5	-59.7	-52.9
22-12-13-23	62.4	60.0	65.5
25-15-16-27	46.2	39.9	

this degree, whereas heliotridine esters in solution exhibit averaging between *exo*- and *endo*-buckling (Culvenor & Woods, 1965). The angle between the mean planes defined by the atoms C(1), C(2), C(3), N(4), C(8) and C(5), N(4), C(8), C(7) is 127.9 (4)°; cf. 127.8 (8)° in retrorsine and 127.3 (5)° in jacobine. In the unsaturated ring the atoms are only coplanar to within ±0.068 (4) Å with C(9) lying 0.252 (5) Å from the plane. This is similar to the situation in retrorsine in

which the atoms are coplanar within ± 0.061 (10) Å with C(9) lying 0.238 (11) Å out-of-plane, and jacobine where C(9) lies 0.247 (6) Å from its associated ring plane [atoms coplanar to within ± 0.055 (5) Å]. It is interesting to note that in crystals of otosenine (Pérez-Salazar, Cano, Fayos, Martínez-Carrera & García-Blanco, 1977) and senkirkine (Birnbaum, 1974), in which there is no ring fusion at C(8) and N(4), C(9) lies 0.250 (7) and 0.261 (3) Å respectively from the plane of the atoms C(1), C(2), C(3), N(4), C(8) which are coplanar within ± 0.05 (1) Å in each molecule.

The 12-membered macrocyclic system has a conformation which closely resembles the ones in crystals of retrorsine* and jacobine (see Table 4), and is similar to those observed in crystals of swazine methiodide (Laing & Sommerville, 1972), senkirkine, otosenine, and yamataimine hydrobromide (Hikichi, Furuya & Iitaka, 1978). The carbonyl bonds of the ester functions are antiparallel as observed to date in all the 12-membered-ring alkaloids. The angle between the bonds is 16.8 (3)° compared with values 17.2 (7)° in retrorsine, 18.8 (3)° in jacobine, 13.4 (5)° in otosenine and 28.3 (3)° in senkirkine. The intramolecular O(17) ··· O(10) distance of 3.293 (3) Å is similar to the values 3.277 (10) Å in retrorsine, 3.255 (4) Å in jacobine, 3.32 (1) Å in otosenine and 3.348 (3) Å in senkirkine.

The conformation about the primary-ester grouping at C(9) shows a remarkably good agreement with the postulated conformation (I). The torsional angles C(2)–C(1)–C(9)–O(10) -108.3 (3), C(1)–C(9)–O(10)–C(11) 92.4 (3) and C(9)–O(10)–C(11)–O(20) 1.5 (3)° define a system in which one H atom, H(9*b*), that at low field in the NMR spectrum, lies close to the plane of the unsaturated ring [torsional angle, H(9*b*)–C(9)–C(1)–C(2) 13 (3)°] and is also close to the plane of the ester carbonyl group [torsional angle, H(9*b*)–C(9)–O(10)–C(11) -28 (3)°]. Furthermore, the α -OH at C(12) also is close to the plane of the carbonyl group [torsional angle, O(20)–C(11)–C(12)–O(22) -3.8 (3)°] as was previously proposed on the basis of hydrogen bonding evident in the IR spectrum of a solution of senecionine in carbon tetrachloride. The allylic ester carbonyl is approximately parallel to the C(2)–H(2) bond, and atoms in the ester group C(9), O(10), C(11), O(20), C(12) are coplanar within ± 0.011 (4) Å. The relative positions of the H atoms at C(9), the primary-ester carbonyl and the ring double bond are closely similar to those in jacobine, as

discussed by Stoeckli-Evans & Crout (1976). These authors also discussed the correlation between these positions and the degree of magnetic non-equivalence of the C(9) H atoms as exhibited in NMR spectra. They reported an insensitivity of the magnetic non-equivalence to changes of solvent for three macrocyclic alkaloids which strongly suggested that their conformation is determined largely by intramolecular forces. This reasoning leads also to an expectation that the alkaloids will have the same conformation in the crystal as in solution.

The secondary-ester system at C(7) shows a substantial twist imposed on the preferred conformation of a secondary ester (Culvenor, 1966) in which the atoms C(7), H(7), O(17), C(16), O(27), C(15) would be coplanar and H(7) *cis* to O(27). In the crystal structure, the atoms C(7), O(17), C(16), O(27), C(15) are coplanar within ± 0.058 (3) Å [torsional angle C(7)–O(17)–C(16)–C(15) -172.6 (3)°] but there is an angle of 68 (1)° between the planes defined by the atoms H(7), C(7), O(17) and O(17), C(16), O(27), C(15). The unsaturated side-chain C(15)=C(25)–C(26) also is not in the position suggested in (I). The double bond is not *trans*-coplanar with the carbonyl group but in a near-*cis* arrangement with the torsional angle C(25)–C(15)–C(16)–O(27) 46.2 (3)°; *cf.* 41.2 (6)° in retrorsine. This situation may be due to crystal packing but the impression is gained from models that it is more likely that the constraints of the 12-membered ring and the minimizing of non-bonded interactions between substituents outweigh the energies involved in a less-than-ideal conformation of the α,β -unsaturated secondary ester (see Fig. 1). The similar orientation of the C=C–C=O grouping observed in crystals of swazine methiodide and retrorsine hydrobromide ethanol solvate (Stoeckli-Evans, 1979) supports this interpretation. In the two latter, the C=C–C=O torsional angle has the respective values 54.1 and 43.7 (9)°.

The bond lengths and angles are in good agreement with those reported for other pyrrolizidine alkaloids. The ring-fusion distance, N(4)–C(8), of 1.497 (4) Å is similar to the values 1.51 (1) Å in retrorsine and 1.486 (5) Å in jacobine while the angles subtended at N(4) have a mean value 110.6 (3)° with C(3)–N(4)–C(5) 115.3 (3)°; the corresponding values in retrorsine are 110.7 (6) and 114.8 (6)° and in jacobine are 110.4 (4) and 115.2 (4)°. The central C–C bond in the C=C–C=O grouping is 1.493 (5) Å which compares well with the values 1.50 (1) Å in retrorsine and 1.497 (4) Å in senkirkine. Apart from C(6)–H(6*b*) of 0.82 (4) Å, C(26)–H(26*b*) of 0.81 (7) Å and C(26)–H(26*a*) of 1.27 (6) Å, the C–H bond lengths range between 0.90 (6) and 1.12 (4) Å with mean value 1.01 Å. There is no indication of intramolecular hydrogen bonding in the molecule [the O(20) ··· H(22) distance is 2.65 (6) Å].

* Differences between the conformations of the retrorsine molecules in crystals of the free alkaloid and in crystals of retrorsine hydrobromide ethanol solvate (Stoeckli-Evans, 1979) are reflected in the torsional angles C(2)–C(1)–C(9)–O(10), C(1)–C(9)–O(10)–C(11), C(13)–C(14)–C(15)–C(16), C(14)–C(15)–C(16)–O(17) and C(8)–C(1)–C(9)–O(10) which have the respective values of -87.0 (5), 79.5 (9), 78.6 (1), 36.3 (5) and 93.6 (5)° in the latter.

An intermolecular hydrogen bond involving the hydroxyl substituent at C(12) and the N atom of an adjacent molecule related by a twofold screw axis ($x = \frac{1}{2}, y = \frac{1}{2}$) links the molecules into helices along *c*. The O(22)··N(4), O(22)—H(22) and H(22)··N(4) distances are 2.827 (4), 0.77 (5) and 2.07 (5) Å respectively [*cf.* distances of 2.85 (1), 0.98 (7) and 1.90 (7) Å in retrorsine] and the N(4)··H(22)—O(22) angle is 167 (3)° (162° in retrorsine). Similar interactions are observed in the jacobine crystal in which the comparable dimensions are 2.928 (5), 0.71 (5), 2.23 (5) Å and 167 (7)°. In senkirikine, H(22) is donated intramolecularly to O(20) in an asymmetric bifurcated hydrogen bond; the H(22)··O(20) and O(22)··O(2) distances are 2.22 (5) and 2.680 (3) Å respectively and the O—H··O angle 118 (2)°. Although the O(22)··O(20) distances in crystals of retrorsine hydrobromide ethanol solvate of 2.63 (2) Å and swazine methiodide of 2.69 Å are indicative of intramolecular hydrogen bonding, this could not be substantiated as the H(22) coordinates are not available. However, this distance is lengthened to 2.724 (4) Å in crystals of senecionine, 2.705 (10) Å in retrorsine and 2.711 (5) Å in jacobine where the hydroxyl substituent at C(12) is involved only in intermolecular hydrogen bonding.

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Methyl 1,2-Bis(dimethylamino)-4-methyl-1*H*-pyrrolo[1,2-*a*]indole-3-carboxylate

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Abstract. C₁₈H₂₃N₃O₂, *M_r* = 313.4, orthorhombic, *Pbca*, *a* = 8.349 (2), *b* = 23.465 (5), *c* = 17.387 (3) Å, *Z* = 8, *D_c* = 1.22 g cm⁻³, *F*(000) = 1344, Mo *K*α radiation, λ = 0.71069 Å, μ(Mo *K*α) = 0.5 cm⁻¹. Space group determined uniquely from systematic

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absences. The structure was solved by direct methods and refined by full-matrix least-squares calculations; *R* = 0.059 for 786 reflections with *I* > 3σ(*I*). The analysis establishes the structure of this product from the reaction between the methyl ester of 3-methyl-

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