The bond lengths and angles are similar to those reported for related structures. The ring-fusion distance N(4)—C(8) of 1·505 (4) Å and the exocyclic angle C(3)—N(4)—C(5) with value 114·4 (2)° are similar to the respective values 1·486 (5) Å and 115·2 (4)° in jacobine and 1·491 (7) Å and 114·4 (2)° in monocrotaline. The oxetane ring angles are similar in value to those reported in comparable structures by Castellano & Hodder (1973) and Büldt, Debaerdemaeker & Friedrichsen (1980). The angle subtended at the C atom opposite to the O is significantly smaller in value than the angle at the O. In grantaline the difference is 6·9 (2)° while in the structures referred to above the difference is on average 9·5°. As expected, a number of the exocyclic angles associated with the oxetane ring also are severely distorted from the regular tetrahedral value. The largest distortion is at C(14) where the angles C(15)—C(14)—C(19) and C(15)—C(14)—C(13) have the respective values of 122·9 (3) and 117·5 (2)°.

In the crystal the molecules are linked into helices along the *b* axis by intermolecular hydrogen bonds involving the α-OH substituent and the N atom of an adjacent molecule related by the twofold screw axis. The O(23)···N(4), O(23)—H(23) and H(23)···N(4) distances are 2·853 (4), 0·83 (5) and 2·05 (5) Å respectively and the N(4)···H(23)—O(23) angle is 163°. Similar interactions have been observed in crystals of other pyrrolizidine alkaloids; for example, in jacobine and senecionine the comparable dimensions are 2·827 (4), 0·77 (5), 2·07 (5) Å, 167 (3)° and 2·928 (5), 0·71 (5), 2·23 (5) Å and 167 (7)° respectively.

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Dehydrosenecionine, $C_{18}H_{23}NO_5$: Active Metabolite of the Pyrrolizidine Alkaloid Senecionine

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Abstract. M_r = 333·4, orthorhombic, $P2_12_12_1$, a = 6·796 (1), b = 14·842 (2), c = 16·917 (1) Å from diffractometer measurements ($\bar{\lambda}$ = 1·5418 Å), U = 1706·4 (4) Å^3, D_x = 1·298 Mg m^{-3}, Z = 4, $F(000)$ = 712, μ(Cu $K\alpha$) = 0·69 mm^{-1}, T = 288 K. 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 = 0·047 for 1540 observed reflections. Apart from the pyrrolizidine nucleus, the molecule has a conformation which closely resembles that of the parent alkaloid.

Introduction. The present analysis forms part of a structural study of hepatotoxic pyrrolizidine alkaloids. It is well established that the nucleotoxic properties of the alkaloids are due to metabolic activation to the

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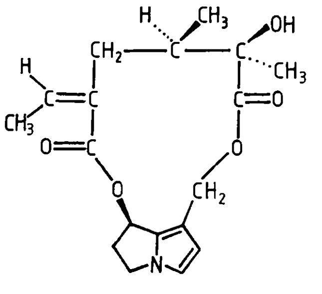
corresponding pyrrolic derivatives or dehydroalkaloids (McLean, 1970; Huxtable, 1979, 1980). The conformation of the hepatotoxic alkaloid senecionine, $C_{18}N_{25}NO_5$, which contains a 12-membered macroring, has been accurately defined (Mackay & Culvenor, 1982). To assess the conformational change which occurs when the alkaloid is converted into its toxic metabolite, X-ray analysis of crystals of dehydrosenecionine (I) (Culvenor, Edgar, Smith & Tweeddale, 1970) was undertaken.



(I)

Experimental. As the tabular crystals grown from ethanol proved to be unstable in air, a crystal of dimensions *ca* 0·45 × 0·18 × 0·16 mm was sealed in a thin-walled Lindeman-glass tube for the data collection. Intensities measured at 288 K with Cu *Kα* radiation (graphite-crystal monochromator) on a Rigaku–AFC four-circle diffractometer; of 1652 non-equivalent terms ($2\theta_{\max} = 130°$) recorded by an ω–2θ scan of 4° min^{-1} and with 10s stationary background counts, 1540 for which $|F_o| > 2\sigma|F_c|$ were used for the structure refinement; during the data-collection period the three reference reflections monitored every 50 reflections showed a gradual 4% decrease in intensity and the data were scaled accordingly; intensities not corrected for absorption or extinction; scattering factors for O, N and C from Cromer & Mann (1968), for H from Stewart, Davidson & Simpson (1965); anomalous-dispersion corrections made for the non-hydrogen atoms with the values of Cromer & Liberman (1970).

Structure solved by direct methods with *SHELX76* (Sheldrick, 1976); H atoms located on a difference map after full-matrix least-squares refinement of the O, N and C atoms with anisotropic temperature factors; H-atom coordinates and isotropic temperature factors refined; final refinement converged at $R = R_w = 0{\cdot}047$; the function minimized was $\sum w(|F_o| - |F_c|)^2$ with the terms weighted according to $(\sigma^2|F_o| + 0{\cdot}0005|F_o|^2)^{-1}$; largest peaks on the final difference map were of heights 0·21 and −0·29 e Å^{-3}, and the mean parameter shift-to-error ratios at convergence were 0·14:1 for H atoms and 0·10:1 for all other parameters.

Discussion. Final atomic coordinates are given in Table 1.* Figs. 1 and 2 have been prepared with *ORTEP* (Johnson, 1965). The molecular conformation is illustrated in Fig. 1, bond lengths and angles are given in Table 2 and torsional angles in Table 3.

Apart from the pyrrolizidine nucleus, there is a fairly close resemblance to the conformation of the parent alkaloid (see Table 3). As expected, the additional unsaturation in the pyrrolizidine nucleus has resulted in considerable flattening so that the angle between the

Table 1. *Final atomic coordinates* (× 10^4, *for* H × 10^3) *and isotropic temperature factors with e.s.d.'s in parentheses*

For non-hydrogen atoms $B_{eq} = 8\pi^2 U_{eq} = \frac{8}{3}\pi^2\sum_i\sum_j U_{ij}a_i^*a_j^*\mathbf{a}_i.\mathbf{a}_j$.

	x	y	z	B_{eq} or B_{iso} (Å^2)
C(1)	−1406 (5)	−1939 (2)	6103 (2)	3·0 (1)
C(2)	−3198 (6)	−2428 (2)	6144 (2)	3·5 (1)
C(3)	−4484 (6)	−2072 (3)	5605 (2)	3·8 (2)
N(4)	−3542 (4)	−1378 (2)	5231 (2)	3·3 (1)
C(5)	−4047 (5)	−682 (3)	4655 (2)	3·9 (2)
C(6)	−2007 (6)	−365 (3)	4390 (2)	3·7 (1)
C(7)	−610 (5)	−586 (2)	5074 (2)	2·8 (1)
C(8)	−1668 (5)	−1297 (2)	5523 (2)	2·8 (1)
C(9)	389 (7)	−2144 (2)	6567 (2)	4·0 (1)
O(10)	1618 (4)	−1358 (1)	6711 (1)	3·5 (1)
C(11)	1439 (5)	−933 (2)	7410 (2)	3·4 (1)
C(12)	3195 (5)	−305 (2)	7553 (2)	2·9 (1)
C(13)	3428 (5)	366 (2)	6869 (2)	2·9 (1)
C(14)	1653 (6)	1017 (2)	6836 (2)	3·0 (1)
C(15)	1418 (5)	1450 (2)	6031 (2)	2·7 (1)
C(16)	1003 (4)	804 (2)	5377 (2)	2·6 (1)
O(17)	−313 (3)	179 (1)	5606 (1)	2·8 (1)
C(18)	4989 (7)	−910 (3)	7669 (2)	4·0 (2)
C(19)	5367 (6)	896 (4)	6905 (3)	4·6 (2)
C(20)	1560 (5)	2332 (2)	5908 (2)	3·3 (1)
C(21)	1289 (7)	2829 (3)	5145 (3)	3·9 (2)
O(22)	150 (5)	−1076 (2)	7877 (2)	5·1 (1)
O(23)	2832 (4)	212 (2)	8249 (1)	3·4 (1)
O(24)	1768 (4)	806 (1)	4732 (1)	3·3 (1)
H(2)†	−354 (6)	−295 (2)	652 (2)	4·7 (8)
H(3)	−582 (7)	−217 (3)	551 (3)	7·5 (13)
H(5*a*)	−484 (6)	−22 (3)	493 (2)	4·7 (8)
H(5*b*)	−474 (6)	−96 (2)	422 (2)	4·3 (8)
H(6*a*)	−209 (6)	27 (3)	426 (2)	5·0 (9)
H(6*b*)	−153 (6)	−74 (3)	390 (2)	5·2 (9)
H(7)	63 (6)	−79 (2)	487 (2)	3·9 (8)
H(9*a*)	116 (6)	−258 (3)	628 (2)	4·7 (9)
H(9*b*)	−1 (7)	−242 (3)	711 (3)	6·2 (10)
H(13)	345 (4)	−1 (2)	637 (2)	2·4 (6)
H(14*a*)	48 (5)	67 (2)	699 (2)	2·3 (6)
H(14*b*)	188 (6)	151 (3)	725 (2)	4·6 (8)
H(18*a*)	612 (6)	−52 (3)	783 (2)	5·0 (9)
H(18*b*)	468 (6)	−138 (3)	808 (2)	5·5 (9)
H(18*c*)	523 (7)	−123 (3)	722 (2)	5·5 (10)
H(19*a*)	656 (6)	53 (3)	678 (2)	4·7 (9)
H(19*b*)	545 (7)	120 (3)	739 (3)	5·5 (11)
H(19*c*)	530 (7)	141 (3)	649 (3)	6·2 (10)
H(20)	183 (6)	270 (2)	638 (2)	4·3 (8)
H(21*a*)	107 (7)	243 (3)	465 (3)	6·4 (11)
H(21*b*)	242 (7)	321 (3)	498 (3)	7·0 (12)
H(21*c*)	30 (7)	326 (3)	523 (2)	5·6 (10)
H(23)	270 (7)	−19 (3)	866 (3)	6·2 (10)

† H atoms have the same numbering as the atoms to which they are bonded.

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* Lists of structure factors, anisotropic temperature factors and intermolecular contacts have been deposited with the British Library Lending Division as Supplementary Publication No. SUP 38451 (19 pp.). Copies may be obtained through The Executive Secretary, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

Table 2. *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)	1·419 (5)	C(11)–C(12)	1·533 (5)
C(1)–C(8)	1·379 (5)	C(11)–O(22)	1·199 (5)
C(1)–C(9)	1·482 (6)	C(12)–C(13)	1·535 (5)
C(2)–C(3)	1·369 (5)	C(12)–C(18)	1·527 (6)
C(3)–N(4)	1·368 (5)	C(12)–O(23)	1·427 (4)
N(4)–C(5)	1·461 (5)	C(13)–C(14)	1·547 (5)
N(4)–C(8)	1·371 (4)	C(13)–C(19)	1·536 (6)
C(5)–C(6)	1·531 (5)	C(14)–C(15)	1·514 (5)
C(6)–C(7)	1·532 (5)	C(15)–C(16)	1·491 (5)
C(7)–C(8)	1·486 (5)	C(15)–C(20)	1·329 (4)
C(7)–O(17)	1·463 (4)	C(16)–O(17)	1·346 (3)
C(9)–O(10)	1·455 (10)	C(16)–O(24)	1·209 (4)
O(10)–C(11)	1·346 (4)	C(20)–C(21)	1·498 (6)

C(2)–C(1)–C(8)	106·1	C(12)–C(11)–O(22)	124·9
C(2)–C(1)–C(9)	125·1	C(11)–C(12)–C(13)	110·8
C(8)–C(1)–C(9)	128·7	C(11)–C(12)–C(18)	106·5
C(1)–C(2)–C(3)	108·5	C(11)–C(12)–O(23)	108·8
C(2)–C(3)–N(4)	107·5	C(13)–C(12)–C(18)	113·3
C(3)–N(4)–C(5)	137·0	C(13)–C(12)–O(23)	106·9
C(3)–N(4)–C(8)	109·5	C(18)–C(12)–O(23)	110·4
C(5)–N(4)–C(8)	113·4	C(12)–C(13)–C(14)	110·6
N(4)–C(5)–C(6)	101·5	C(12)–C(13)–C(19)	113·0
C(5)–C(6)–C(7)	105·9	C(14)–C(13)–C(19)	110·5
C(6)–C(7)–C(8)	103·8	C(13)–C(14)–C(15)	112·3
C(6)–C(7)–O(17)	112·6	C(14)–C(15)–C(16)	114·5
C(8)–C(7)–O(17)	107·7	C(14)–C(15)–C(20)	123·5
C(1)–C(8)–N(4)	108·4	C(16)–C(15)–C(20)	122·1
C(1)–C(8)–C(7)	142·4	C(15)–C(16)–O(17)	110·8
N(4)–C(8)–C(7)	109·1	C(15)–C(16)–O(24)	125·9
C(1)–C(9)–O(10)	113·4	O(17)–C(16)–O(24)	123·2
C(9)–O(10)–C(11)	118·1	C(7)–O(17)–C(16)	116·7
O(10)–C(11)–C(12)	110·7	C(15)–C(20)–C(21)	127·7
O(10)–C(11)–O(22)	124·2		

Table 3. *Torsional angles* (°) (*e.s.d.'s are about* 0·3°)

Atoms are represented by their identification number. Corresponding values for senecionine (Mackay & Culvenor, 1982) are included.

Pyrrolizidine nucleus	Dehydrosenecionine	Senecionine	Esterifying acid moiety	Dehydrosenecionine	Senecionine
1–2–3–4	0·0	6·5	1–9–10–11	97·7	92·4
1–8–4–3	−0·9	10·7	2–1–9–10	−152·5	−108·3
1–8–4–5	175·3	136·4	6–7–17–16	−81·2	−79·1
1–8–7–6	166·4	−146·5	7–17–16–15	−169·9	−172·6
1–8–7–17	−74·1	−29·5	7–17–16–24	7·9	5·3
2–3–4–5	−174·4	−131·8	8–1–9–10	31·9	80·1
2–3–4–8	0·5	−10·6	8–7–17–16	165·0	169·8
2–1–8–4	0·9	−6·8	9–10–11–12	164·2	−179·0
2–1–8–7	−174·3	112·0	9–10–11–22	−11·1	1·5
3–4–5–6	−164·6	139·4	10–11–12–13	55·7	54·0
3–4–8–7	176·0	−118·5	10–11–12–18	−68·0	−68·1
3–2–1–8	−0·6	0·2	10–11–12–23	172·9	176·8
3–2–1–9	−177·0	−172·2	11–12–13–14	65·8	60·5
4–5–6–7	−24·8	−36·0	11–12–13–19	−169·6	−175·6
4–8–1–9	177·2	165·9	12–13–14–15	−159·1	−163·9
4–8–7–6	−8·8	−29·0	13–12–11–22	−129·1	−126·5
4–8–7–17	110·7	88·0	13–14–15–16	62·8	62·5
5–4–8–7	−7·8	7·1	13–14–15–20	−117·4	−115·1
5–6–7–8	21·1	39·6	14–13–12–18	−174·5	−179·4
5–6–7–17	−95·0	−74·9	14–13–12–23	−52·6	−61·5
6–5–4–8	20·7	17·9	14–15–16–17	42·3	46·4
7–8–1–9	2·0	−75·3	14–15–16–24	−135·4	−131·5
			14–15–20–21	−177·8	178·6
			15–14–13–19	75·0	71·1
			16–15–20–21	2·0	1·2
			17–16–15–20	−137·5	−136·0
			18–12–13–19	−49·9	−55·5
			18–12–11–22	107·2	111·4
			19–13–12–23	71·9	62·4
			22–11–12–23	−11·8	−3·8
			20–15–16–24	44·8	46·2

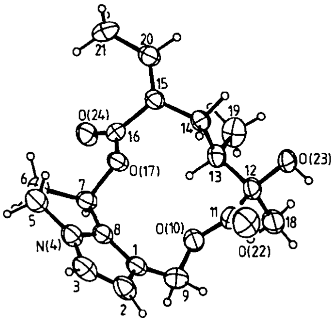

Fig. 1. A perspective view of the molecule with thermal ellipsoids scaled to 50% probability.

mean planes defined by the atoms C(1), C(2), C(3), N(4), C(8) and N(4), C(8), C(7) is 176·3 (4)° compared with the value 127·9 (4)° in senecionine. As in the latter, the pyrrolizidine ring is *exo*-buckled with a puckering angle of 28·5 (4)° [*cf.* 35·3 (4)° in senecionine]. The atoms in the unsaturated ring are coplanar to within ± 0·005 (4) Å with C(9) lying only 0·057 (3) Å from the plane. This contrasts with the situation in senecionine and the closely related alkaloid retrorsine (Coleman, Coucourakis & Pretorius, 1980), in which C(9) lies 0·252 (5) and 0·238 (11) Å respectively from the ring plane.

In the macrocyclic system the carbonyl bonds of the ester functions are *anti*-parallel as observed in all the 12-membered-ring alkaloids. The angle between the bonds is 24·5 (4)° [*cf.* 16·8 (3)° in senecionine] and the intramolecular O(17)···O(10) distance of 3·228 (3) Å is similar to the value 3·293 (3) Å in the parent alkaloid.

There is an appreciable twist in the primary-ester system in which the torsional angles C(9)–O(10)–C(11)–C(12) and C(9)–O(10)–C(11)–O(22) have the respective values 164·2 (3) and −11·1 (3)°. In senecionine, the comparable angles are −179·0 (3) and 1·5 (3)° which illustrate that the ester group in the parent alkaloid is planar to within experimental error. Moreover, in dehydrosenecionine H(9*b*) lies further from the plane of the unsaturated ring [torsional angle H(9*b*)–C(9)–C(1)–C(2) −31 (3)°, *cf.* 13 (3)° in senecionine] and the α-OH substituent at C(12) lies further from the C(12), C(11), O(10) plane [torsional angle O(22)–C(11)–C(12)–O(23) −11·8 (3), −3·8 (3)° in senecionine] than in the parent alkaloid.

The conformation of the secondary-ester system at C(7) is very similar to that in the parent alkaloid; the torsional angle C(7)–O(17)–C(16)–C(15) −169·9 (3)° compares well with the value −172·6 (3)° in senecionine; however, the angle between the planes defined by atoms H(7), C(7), O(17) and O(17), C(16), O(24), C(15) of 46 (1)° is much smaller than the value 68 (1)° in the latter. The near-*cis* arrangement of the double bond in the side chain, C(20)—C(21), with the carbonyl group is defined by the torsional angle C(20)—C(15)—C(16)—O(24) 44·8 (3)° [*cf.* value 46·2 (3)° in senecionine].

The bond lengths and angles are in good agreement with those reported for comparable structures. The bonds in the unsaturated ring have a mean length of 1·381 (5) Å with the two C—N bonds having a mean length of 1·370 (5) Å. These values are indicative of extensive electron delocalization around the ring as observed in other pyrrole moieties (English, McGillivray & Smal, 1980; Acheson, Ferris, Critchley & Watkin, 1980). The considerable distortion of the exocyclic angles from standard trigonal values, C(3)—N(4)—C(5) 137·0 (3)° and C(1)—C(8)—C(7) 142·4 (3)°, is a consequence of the flattening of the pyrrolizidine nucleus. The central C—C bond in the C=C—C=O grouping of 1·491 (5) Å is similar to the value 1·493 (5) Å in the parent alkaloid, and the C—H lengths in the molecule range in value from 0·91 (4) Å [C(18)—H(18*c*)] to 1·05 (4) Å [C(9)—H(9*b*)]. As in senecionine, there is no indication of intramolecular hydrogen bonding.

The mode of packing in the crystal (Fig. 2) is quite different from that observed in senecionine. In the latter the α-OH substituent is involved in intermolecular H-bonding interactions with the N atom. In dehydrosenecionine, the α-OH group forms hydrogen bonds with the carbonyl O of the secondary-ester system, O(24), of a molecule related by the twofold screw axis ($x = \frac{1}{4}, y = 0$). These interactions link the molecules into helices along the *c* axis. The O(23)···O(24), O(23)—H(23) and H(23)···O(24) distances are 2·941 (3), 0·91 (4) and 2·07 (4) Å respectively and the O(24)···H(23)—O(23) angle is 160 (3)°.

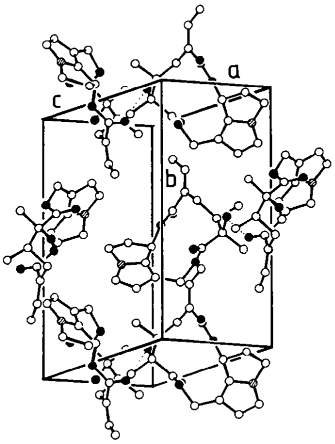

Fig. 2. The molecular packing in the crystal.

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Structure of Perylene–Chloranil* Molecular Complex (1:1), $C_{20}H_{12}.C_6Cl_4O_2$

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Abstract. $M_r = 498·19$, triclinic, $P\bar{1}$, $a = 8·602$ (1), $b = 9·635$ (2), $c = 7·234$ (2) Å, $\alpha = 102·67$ (2), $\beta =$ 111·17 (2), $\gamma = 64·38$ (1)°, $V = 502·7$ Å^3, $Z = 1$, $D_m = 1·64$, $D_x = 1·645$ Mg m^{-3}, λ(Mo $K\alpha$) = 0·7107 Å, μ(Mo $K\alpha$) = 6·13 cm^{-1}, $F(000) = 252$, $T =$ 294 ± 1 K, $R = 0·046$ for 1803 observed reflections.

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* Chloranil is 2,3,5,6-tetrachloro-2,5-cyclohexadiene-1,4-dione.

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