conventional R value was 7%.* The atomic coordinates are listed in Table 1, bond lengths and angles in Tables 2 and 3. Deviations from the principal mean planes are listed in Table 4.

Discussion. The result of the structure determination is illustrated in Fig. 2. Torsional angles are shown in Fig. 1. Ring C has a half-chair conformation, where C(6), C(7), C(2) and C(3) are slightly displaced from their mean plane (Table 4). Ring D is chair shaped. For ring E we calculate a pseudorotation phase angle φ (Altona,

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* Lists of structure factors and anisotropic thermal parameters have been deposited with the British Library Lending Division as Supplementary Publication No. SUP 32492 (16 pp.). Copies may be obtained through The Executive Secretary, International Union of Crystallography, 13 White Friars, Chester CH1 1NZ, England.
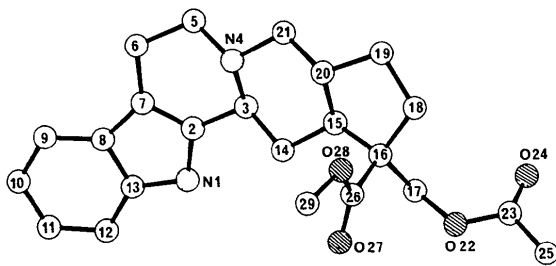

Fig. 2. A perspective view of the molecule.

Geise & Romers, 1968) of 6·3° and a maximum torsional angle φ_m of 46·7°, corresponding to a $C_2(18)$ half-chair form. The six- and five-membered rings A and B are planar within ±0·01 and ±0·06 Å respectively. The angle between the normals to these planes is 2·1°. The C/D ring junction is *trans*. C(17) is slightly out of the mean plane of the acetate group (0·10 Å) and the torsional angle C(16)—C(17)—O(22)—C(22) is −114°.

The methanol molecule is involved in two hydrogen bonds linking two different molecules (translated along **a**): N(1)H···O(30) (3·03 Å) and O(30)H···N(4) (2·90 Å).

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Pandine

By ARNAUD DUCRUIX AND CLAUDINE PASCARD

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Abstract. $C_{21}H_{24}O_3N_2$, $M_r = 352$, triclinic, $P1$, $a = 6·918$ (4), $b = 12·634$ (5), $c = 13·008$ (5) Å, $\alpha = 94·00$ (2), $\beta = 93·73$ (2), $\gamma = 100·61$ (2)°, $Z = 2$. The unit cell contains two independent molecules of pandine, one molecule of methanol and two molecules of water. The two molecules of the alkaloid are in the same conformation, and the configuration at C(20) is R.

Introduction. 4031 intensities were collected on a Philips PW 1100 diffractometer (with Cu $K\alpha$ radiation and a graphite monochromator) up to $2\theta = 68°$. 3403 were above the background (2σ), and were corrected for the Lp factor, but not for absorption.

The structure was solved with *MULTAN* (Germain, Main & Woolfson, 1971), and refined by least-squares methods with isotropic temperature factors to a final R

Table 1. *Final positional and thermal parameters* ($\times 10^4$) *with standard deviations in parentheses*

O(2) coordinates were kept fixed during the refinement.

	x	y	z	$B(\text{Å}^2)$
Molecule 1				
O(1)	2611 (15)	651 (8)	6902 (8)	5·23 (15)
O(2)	4428	3865	8774	5·04 (15)
O(3)	5546 (15)	5474 (8)	8269 (8)	5·34 (16)
N(1)	6599 (15)	5766 (8)	6332 (8)	3·97 (15)
N(4)	2392 (16)	2839 (9)	5120 (8)	4·32 (16)
C(2)	5431 (16)	4757 (9)	6266 (9)	3·43 (16)
C(3)	4566 (17)	3180 (9)	4956 (9)	3·71 (17)
C(5)	1527 (20)	3840 (11)	5117 (10)	5·13 (22)
C(6)	3053 (19)	4697 (10)	4716 (9)	4·55 (20)
C(7)	5064 (17)	4397 (9)	5120 (8)	3·41 (16)
C(8)	6751 (17)	5106 (10)	4667 (9)	3·78 (17)
C(9)	7490 (19)	5060 (11)	3695 (10)	4·77 (21)
C(10)	9066 (20)	5888 (11)	3488 (10)	5·49 (24)
C(11)	9769 (19)	6673 (11)	4183 (10)	4·85 (21)
C(12)	9091 (20)	6722 (10)	5197 (10)	4·78 (21)
C(13)	7554 (18)	5927 (10)	5370 (9)	4·30 (19)
C(14)	5493 (18)	2597 (10)	5813 (9)	3·94 (18)
C(15)	4832 (21)	1366 (12)	5620 (10)	5·67 (25)
C(16)	4810 (17)	4077 (9)	7000 (9)	3·43 (16)
C(17)	4287 (16)	2887 (9)	6710 (9)	3·28 (16)
C(18)	−579 (36)	322 (19)	5275 (18)	11·03 (54)
C(19)	1285 (30)	461 (15)	5131 (15)	8·58 (40)
C(20)	2640 (19)	1188 (10)	5952 (10)	4·54 (20)
C(21)	2320 (17)	2357 (9)	6130 (9)	3·70 (17)
C(22)	4888 (18)	4444 (10)	8081 (9)	3·99 (18)
C(23)	5708 (23)	5926 (13)	9352 (12)	6·60 (29)
Molecule 2				
O(101)	−873 (14)	10512 (8)	7858 (8)	4·60 (14)
O(102)	−405 (14)	7132 (8)	7773 (8)	4·80 (15)
O(103)	646 (15)	6102 (8)	8962 (8)	4·96 (15)
N(101)	1644 (16)	6966 (9)	10944 (8)	4·24 (16)
N(104)	−1269 (15)	9703 (8)	10571 (8)	3·59 (14)
C(102)	926 (17)	9737 (9)	10487 (9)	3·44 (16)
C(103)	895 (17)	9737 (9)	10846 (9)	3·41 (16)
C(105)	−2474 (18)	8817 (10)	11062 (9)	4·19 (19)
C(106)	−1082 (17)	8458 (9)	11813 (9)	3·80 (18)
C(107)	932 (17)	8636 (9)	11300 (9)	3·51 (16)
C(108)	2510 (17)	8432 (10)	12087 (9)	3·87 (18)
C(109)	3471 (19)	9067 (10)	12950 (10)	4·52 (20)
C(101)	4698 (20)	8555 (11)	13550 (10)	4·86 (22)
C(111)	4904 (21)	7519 (12)	13336 (11)	5·73 (25)
C(112)	4016 (21)	6915 (11)	12472 (10)	5·04 (22)
C(113)	2794 (17)	7392 (9)	11859 (9)	3·65 (17)
C(114)	1812 (16)	9882 (9)	9832 (9)	3·29 (16)
C(115)	1392 (18)	10931 (12)	9393 (9)	4·23 (19)
C(116)	397 (17)	7896 (9)	9477 (9)	3·60 (17)
C(107)	322 (17)	9000 (9)	9159 (8)	3·13 (15)
C(118)	−1385 (24)	12550 (13)	8791 (12)	6·92 (31)
C(119)	−2043 (21)	11504 (11)	9275 (11)	5·64 (25)
C(120)	−783 (17)	10670 (10)	9006 (9)	4·00 (18)
C(121)	−1448 (17)	9568 (9)	9406 (9)	3·55 (17)
C(122)	159 (18)	7054 (10)	8633 (9)	4·05 (18)
C(123)	554 (22)	5279 (12)	8134 (11)	5·96 (26)
O(*M*)	5284 (17)	9398 (9)	7362 (9)	7·13 (22)
C(*M*)	4846 (29)	8353 (16)	7006 (15)	9·11 (42)
OH(21)	1302 (25)	2159 (13)	3038 (12)	11·79 (38)
OH(22)	7903 (28)	1293 (15)	2075 (14)	13·21 (44)

value of 0·12. H atoms which could be calculated in the theoretical position (C—H: 1·0 Å, $\widehat{\text{C—C—H}}$: 109°) were recalculated at each cycle but not refined.

The scattering factors were those of Doyle & Turner (1968) for the heavy atoms, and those of Stewart, Davidson & Simpson (1965) for H.

Table 2. *Intramolecular bond distances* (Å) *with standard deviations in parentheses*

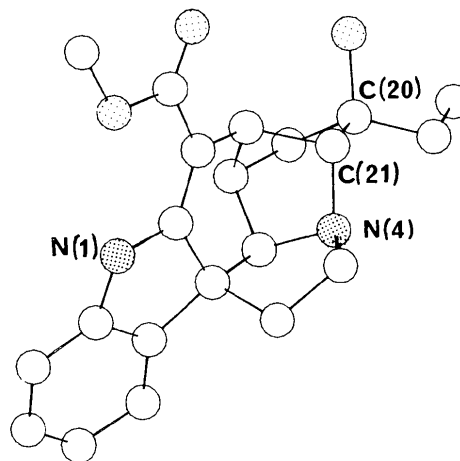| Molecule 1		Molecule 2	
O(1)—C(20)	1·45 (2)	O(101)—C(120)	1·49 (2)
O(2)—C(22)	1·22 (–)	O(102)—C(122)	1·18 (2)
O(3)—C(22)	1·30 (2)	O(103)—C(122)	1·39 (2)
O(3)—C(23)	1·47 (2)	O(103)—C(123)	1·43 (2)
N(1)—C(2)	1·37 (2)	N(101)—C(102)	1·37 (2)
N(1)—C(13)	1·46 (2)	N(101)—C(113)	1·40 (2)
N(4)—C(3)	1·52 (2)	N(104)—C(103)	1·51 (2)
N(4)—C(5)	1·50 (2)	N(104)—C(105)	1·48 (2)
N(4)—C(21)	1·49 (2)	N(104)—C(121)	1·51 (2)
C(2)—C(7)	1·52 (2)	C(102)—C(107)	1·46 (2)
C(2)—C(16)	1·37 (2)	C(102)—C(116)	1·37 (2)
C(3)—C(7)	1·51 (2)	C(103)—C(107)	1·55 (2)
C(3)—C(14)	1·55 (2)	C(103)—C(114)	1·51 (2)
C(5)—C(6)	1·51 (2)	C(105)—C(106)	1·48 (2)
C(6)—C(7)	1·58 (2)	C(106)—C(107)	1·57 (2)
C(7)—C(8)	1·51 (2)	C(107)—C(108)	1·52 (2)
C(8)—C(9)	1·40 (2)	C(108)—C(109)	1·39 (2)
C(8)—C(13)	1·34 (2)	C(108)—C(113)	1·38 (2)
C(9)—C(10)	1·42 (2)	C(109)—C(110)	1·39 (2)
C(10)—C(11)	1·29 (2)	C(110)—C(111)	1·35 (2)
C(11)—C(12)	1·43 (2)	C(111)—C(112)	1·36 (2)
C(12)—C(13)	1·36 (2)	C(112)—C(113)	1·37 (2)
C(14)—C(15)	1·54 (2)	C(114)—C(115)	1·55 (2)
C(14)—C(17)	1·54 (2)	C(114)—C(117)	1·55 (2)
C(15)—C(20)	1·58 (2)	C(115)—C(120)	1·52 (2)
C(16)—C(17)	1·50 (2)	C(116)—C(117)	1·49 (2)
C(16)—C(22)	1·44 (2)	C(116)—C(122)	1·45 (2)
C(17)—C(21)	1·53 (2)	C(117)—C(121)	1·57 (2)
C(18)—C(19)	1·30 (3)	C(118)—C(119)	1·51 (2)
C(19)—C(20)	1·51 (2)	C(119)—C(120)	1·52 (2)
C(20)—C(21)	1·54 (2)	C(120)—C(121)	1·52 (2)

Fig. 1. The molecular structure of pandine.

Table 3. Bond angles (°) with standard deviations in parentheses

Molecule 1			Molecule 2	
C(22)–O(3)–C(23)	117 (1)		C(122)–O(103)–C(123)	113 (1)
C(2)–N(1)–C(13)	110 (1)		C(102)–N(101)–C(113)	110 (1)
C(3)–N(4)–C(5)	106 (1)		C(103)–N(104)–C(105)	110 (1)
C(3)–N(4)–C(21)	106 (1)		C(103)–N(104)–C(121)	103 (1)
C(5)–N(4)–C(21)	113 (1)		C(105)–N(104)–C(121)	114 (1)
N(1)–C(2)–C(7)	106 (1)		N(101)–C(102)–C(107)	107 (1)
N(1)–C(2)–C(16)	132 (1)		N(101)–C(102)–C(116)	131 (1)
C(7)–C(2)–C(16)	122 (1)		C(107)–C(102)–C(116)	122 (1)
N(4)–C(3)–C(7)	107 (1)		N(104)–C(103)–C(107)	104 (1)
N(4)–C(3)–C(14)	100 (1)		N(104)–C(103)–C(114)	104 (1)
C(7)–C(3)–C(14)	114 (1)		C(107)–C(103)–C(114)	116 (1)
N(4)–C(5)–C(6)	106 (1)		N(104)–C(105)–C(106)	105 (1)
C(5)–C(6)–C(7)	103 (1)		C(105)–C(106)–C(107)	104 (1)
C(2)–C(7)–C(3)	110 (1)		C(102)–C(107)–C(103)	111 (1)
C(2)–C(7)–C(6)	108 (1)		C(102)–C(107)–C(106)	110 (1)
C(2)–C(7)–C(8)	102 (1)		C(102)–C(107)–C(108)	102 (1)
C(3)–C(7)–C(6)	101 (1)		C(103)–C(107)–C(106)	100 (1)
C(3)–C(7)–C(8)	125 (1)		C(103)–C(107)–C(108)	125 (1)
C(6)–C(7)–C(8)	109 (1)		C(106)–C(107)–C(108)	108 (1)
C(7)–C(8)–C(9)	132 (1)		C(107)–C(108)–C(109)	131 (1)
C(7)–C(8)–C(13)	109 (1)		C(107)–C(108)–C(113)	107 (1)
C(9)–C(8)–C(13)	119 (1)		C(109)–C(108)–C(113)	122 (1)
C(8)–C(9)–C(10)	118 (1)		C(108)–C(109)–C(110)	114 (1)
C(9)–C(10)–C(11)	120 (1)		C(109)–C(110)–C(111)	123 (1)
C(10)–C(11)–C(12)	123 (1)		C(110)–C(111)–C(112)	122 (1)
C(11)–C(12)–C(13)	116 (1)		C(111)–C(112)–C(113)	116 (1)
N(1)–C(13)–C(8)	109 (1)		N(101)–C(113)–C(108)	108 (1)
N(1)–C(13)–C(12)	128 (1)		N(101)–C(113)–C(112)	129 (1)
C(8)–C(13)–C(12)	124 (1)		C(108)–C(113)–C(112)	122 (1)
C(3)–C(14)–C(15)	111 (1)		C(103)–C(114)–C(115)	110 (1)
C(3)–C(14)–C(17)	99 (1)		C(103)–C(114)–C(117)	98 (1)
C(15)–C(14)–C(17)	103 (1)		C(115)–C(114)–C(117)	102 (1)
C(14)–C(15)–C(20)	102 (1)		C(114)–C(115)–C(120)	105 (1)
C(2)–C(16)–C(17)	119 (1)		C(102)–C(116)–C(117)	119 (1)
C(2)–C(16)–C(22)	122 (1)		C(102)–C(116)–C(122)	125 (1)
C(17)–C(16)–C(22)	118 (1)		C(117)–C(116)–C(122)	115 (1)
C(14)–C(17)–C(16)	109 (1)		C(114)–C(117)–C(116)	112 (1)
C(14)–C(17)–C(21)	93 (1)		C(114)–C(117)–C(121)	92 (1)
C(16)–C(17)–C(21)	122 (1)		C(116)–C(117)–C(121)	121 (1)
C(18)–C(19)–C(20)	116 (2)		C(118)–C(119)–C(120)	112 (1)
O(1)–C(20)–C(15)	107 (1)		O(101)–C(120)–C(115)	107 (1)
O(1)–C(20)–C(19)	109 (1)		O(101)–C(120)–C(119)	107 (1)
O(1)–C(20)–C(21)	112 (1)		O(101)–C(120)–C(121)	108 (1)
C(15)–C(20)–C(19)	110 (1)		C(115)–C(120)–C(119)	118 (1)
C(15)–C(20)–C(21)	102 (1)		C(115)–C(120)–C(121)	102 (1)
C(19)–C(20)–C(21)	117 (1)		C(119)–C(120)–C(121)	115 (1)
N(4)–C(21)–C(17)	104 (1)		N(104)–C(121)–C(117)	103 (1)
N(4)–C(21)–C(20)	108 (1)		N(104)–C(121)–C(120)	108 (1)
C(17)–C(21)–C(20)	100 (1)		C(117)–C(121)–C(120)	101 (1)
O(2)–C(22)–O(3)	122 (–)		O(102)–C(122)–C(103)	123 (1)
O(2)–C(22)–C(16)	125 (–)		O(102)–C(122)–C(116)	126 (1)
O(3)–C(22)–C(16)	113 (1)		O(103)–C(122)–C(116)	112 (1)

Fractional coordinates and thermal parameters are given in Table 1.*

Discussion. Pandine is a hexacyclic alkaloid of the ψ-vincadifformine type, isolated from *Pandaca* (Hoizey, Debray, Le Men-Olivier & Le Men, 1974).
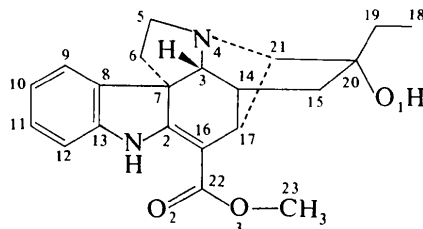
The structure proposed by Le Men, Hoizey, Lukacs, Le Men-Olivier & Levy (1974) is confirmed, and the stereochemistry at C(20), which was unknown, is found to be 20(*R*) with the absolute configuration adopted by Le Men *et al.* (1974) (Fig. 1). Distances and angles are given in Tables 2 and 3. There is no significant difference between the two molecules of the asymmetric unit, which are in the same conformation.

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