

Table 4. *Important intermolecular interactions less than 3.5 Å*

No.	From†	To	Distance
1	O(1)	O(3 ⁱ)	2.868 (6)*
2	O(4)	O(5 ⁱ)	2.886 (6)*
3	O(4)	O(6 ⁱ)	2.802 (7)*
4	O(3)	O(2 ^{iv})	2.710 (6)*
5	C(2)	O(6 ⁱ)	3.427 (7)
6	O(2)	N	2.981 (6)*
7	O(2)	C(3 ⁱ)	3.489 (7)
8	O(2)	O(6 ⁱ)	2.768 (6)*
9	O(5)	O(6 ⁱ)	2.762 (7)*
10	N'	O(2 ⁱⁱⁱ)	3.237 (7)*
11	N'	O(4 ^{iv})	3.232 (6)*
12	N'	O(6 ^{iv})	3.449 (6)
13	C(2)	O(6 ⁱⁱ)	3.352 (6)
14	O(3)	O(5 ^v)	2.845 (6)*
15	O(4)	O(5 ^{vi})	2.901 (6)*

* Hydrogen-bond suggested.

† Atom at (i).

Key to the symmetry operation: (i) x, y, z ; (ii) $-x, 1-y, -z$; (iii) $1+x, y, z$; (iv) $1+x, y, 1+z$; (v) $-x, 1-y, 1-z$; (vi) $1-x, 1-y, -z$.

O(5)···O(6) and O(6)···O(4) (Nos. 2, 9 and 3 in Table 4, respectively) to form a triangular pillar infinitely extending along the c axis. The two triangular pillars related by a centre of symmetry at $(\frac{1}{2}, 0, \frac{1}{2})$ are connected by hydrogen bonds O(4)···O(5) (No. 15 in Table 4) to form a pair of pillars. The pair of pillars is connected to the complex ions by the two strong and one weak hydrogen bonds O(5)···O(3), O(6)···O(2)

and O(4)···N' (Nos. 14, 8 and 11 in Table 4, respectively). Between the pairs of pillars, however, there is no direct interaction which might result in relatively large temperature factors associated with atoms O(4), O(5) and O(6). The hydrogen atoms attached to these have not appeared clearly on the difference Fourier map.

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The Molecular Structure of a Compound with the Lycocotinine Skeleton, C₂₁H₃₁O₂N*

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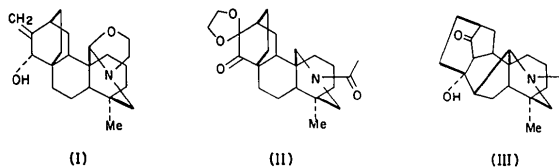
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The compound C₂₁H₃₁O₂N was synthesized from an aconite alkaloid, atisine. It has the lycocotinine skeleton. The crystals are orthorhombic, space group $P2_12_12_1$ with $a = 10.404$, $b = 22.040$ and $c = 7.666$ Å. Data were collected with an automatic Picker diffractometer. The structure was solved by direct phasing methods and the refinement was carried out by least-squares procedure to the final R value of 0.049.

Introduction

The structure of the first C₂₀ aconite alkaloid, atisine (I), was deduced chemically by Wiesner, Armstrong, Bartlett & Edwards (1954) and the structure of lycocotinine, the C₁₉ aconite alkaloid, was determined by X-ray analysis by Przybylska & Marion (1956). Since then a possible biosynthetic transformation of atisine type starting materials into lycocotinine type

alkaloids was postulated. Recently T.Y.R. Tsai and K. Wiesner succeeded in transforming the compound (II), which was prepared from atisine by Johnston & Overton (1972), into the compound (III).



* I issued as N.R.C.C. No. 15106.

The crystals of (III) were obtained from Professor K. Wiesner, who suggested the X-ray determination to confirm the structure. The chemical and the X-ray results were reported in a previous communication (Przybylska, Tsai & Wiesner, 1975).

Experimental

The colourless prisms, grown from ether-pentane, were elongated along the *c* axis. The preliminary examination of the crystals was carried out with precession photographs. The crystal was cut with a chip of razor blade into a 0.24 mm diameter sphere and mounted along the *b* axis. The data were collected by a Picker card-programmed automatic diffractometer with Ni-filtered Cu radiation. The unit-cell dimensions were obtained from the 2θ values of the high-order axial reflexions, measured at a take-off angle of 2.0° . The crystal data are presented in Table 1. The $\theta/2\theta$ scanning method was used at a take-off angle of 2.6° . The scan ranges were 1.6° in 2θ for reflexions of 2θ up to 100° and 2.0° for those with larger 2θ values. The speed was 2° per min and since the diffractometer was set up for protein data collection, only one background measurement was taken for 10 s at the lower side of the peak. High intensities were remeasured with lower

current settings and, if necessary, attenuated with an additional thickness of Ni foil. One standard reflexion, measured after every 40–50 reflexions, was used for scaling.

The threshold was taken as a net count of 50, or 10% of the background, whichever was higher (Ahmed, Hall, Pippy & Huber, 1973). 1673 independent reflexions were measured within $2\theta=127^\circ$ ($\sin^2 \theta_{\max}=0.8009$) and out of these 1526 were observed above threshold (91%). Reflexions with intensities below threshold were excluded from the least-squares refinement. There was no evidence of crystal deterioration during the collection of data.

The intensities were corrected for Lorentz and polarization factors but the absorption corrections were considered unnecessary.

Structure determination

The structure was solved by the direct phasing method (Karle & Hauptman, 1956). The scale factor and the overall temperature factor ($B=3.7 \text{ \AA}^2$) were evaluated by Wilson's method. The examination of the high normalized structure factors revealed that there was a preponderance of *E*'s with $l=0, 3$ and 6 , and a deficiency of $h0l$ reflexions.

A number of preliminary tangent refinement cycles had failed until eight *E* values with assigned phases were used. The *E* map carried out with 187 phased reflexions out of 194 with *E*'s > 1.5 revealed all the atoms of the molecule. The structure was then confirmed by Fourier methods.

Two least-squares cycles brought the *R* value from 0.20 to 0.14. At this point the anisotropic refinement was undertaken and after three more cycles, when *R*

Table 1. *Crystal data*

$C_{21}H_{31}O_2N$, F. W. 329.49; m.p. $157-9^\circ\text{C}$
 Space group: $P2_12_12_1$, $Z=4$
 $a=10.404$ (3), $b=22.040$ (4), $c=7.666$ (3) \AA
 $V=1757.84 \text{ \AA}^3$
 $D_x=1.245 \text{ g cm}^{-3}$
 $D_m=1.242 \text{ g cm}^{-3}$ (by flotation in KI solution at 23°C)
 $F(000)=720$, $\mu(\text{Cu})=6.22 \text{ cm}^{-1}$

Table 2. *Final fractional coordinates and vibration tensor components for the expression*
 $T = \exp \{ -2\pi^2(U_{11}a^{*2}h^2 + \dots + 2U_{12}a^*b^*hk) \}$ and their *e.s.d.*'s (all $\times 10^4$)

	<i>x</i>	<i>y</i>	<i>z</i>	U_{11}	U_{22}	U_{33}	$2U_{23}$	$2U_{13}$	$2U_{12}$
N(1)	1986 (3)	3506 (1)	7341 (4)	301 (15)	399 (15)	426 (18)	176 (33)	-152 (32)	-114 (27)
O(1)	5790 (3)	4078 (1)	3926 (4)	752 (21)	787 (20)	478 (17)	-299 (34)	592 (35)	-712 (36)
O(2)	7655 (3)	4967 (2)	7186 (5)	490 (17)	879 (20)	910 (24)	-452 (45)	288 (40)	-704 (33)
C(1)	3542 (4)	3608 (2)	10592 (5)	600 (27)	699 (27)	301 (22)	116 (45)	-50 (44)	-285 (49)
C(2)	2518 (4)	3124 (2)	10943 (5)	592 (26)	748 (30)	353 (22)	262 (48)	82 (46)	-317 (49)
C(3)	2934 (4)	2528 (2)	10131 (7)	535 (26)	586 (25)	674 (29)	602 (47)	-128 (52)	-141 (46)
C(4)	3260 (4)	2583 (2)	8183 (6)	398 (21)	327 (20)	684 (30)	81 (42)	48 (46)	30 (35)
C(5)	4428 (3)	3004 (2)	7955 (6)	288 (18)	381 (20)	602 (25)	39 (42)	-57 (42)	67 (34)
C(6)	4685 (4)	3136 (2)	5998 (6)	495 (24)	416 (22)	648 (29)	-420 (45)	402 (49)	-287 (39)
C(7)	4039 (4)	3752 (2)	5634 (5)	429 (21)	485 (22)	305 (20)	-38 (38)	3 (39)	-281 (38)
C(8)	5082 (3)	4246 (2)	5465 (5)	394 (22)	532 (22)	376 (21)	-90 (42)	135 (40)	-273 (39)
C(9)	5990 (3)	4197 (1)	7065 (5)	293 (18)	342 (20)	493 (23)	-169 (39)	79 (39)	-8 (31)
C(10)	6569 (4)	4811 (2)	7417 (6)	481 (22)	549 (25)	481 (26)	-200 (45)	21 (45)	-359 (41)
C(11)	5507 (4)	5180 (2)	8208 (6)	583 (26)	426 (22)	628 (29)	-345 (46)	47 (51)	-136 (43)
C(12)	4893 (4)	4728 (2)	9484 (6)	511 (25)	544 (25)	480 (25)	-405 (45)	39 (46)	-240 (43)
C(13)	5257 (3)	4077 (2)	8798 (5)	315 (19)	450 (20)	391 (21)	-33 (39)	-166 (38)	-48 (35)
C(14)	4088 (3)	3635 (2)	8720 (4)	313 (18)	401 (20)	292 (19)	27 (36)	-68 (35)	-62 (35)
C(15)	3189 (3)	3853 (1)	7287 (5)	327 (18)	298 (17)	325 (19)	14 (34)	-29 (36)	-20 (30)
C(16)	2127 (4)	2850 (2)	7133 (5)	407 (21)	458 (22)	474 (24)	-59 (42)	1 (43)	-251 (37)
C(17)	4539 (4)	4889 (2)	5211 (6)	588 (27)	524 (25)	603 (28)	271 (49)	-122 (53)	-213 (45)
C(18)	4519 (6)	5327 (2)	6713 (7)	1254 (47)	500 (27)	904 (41)	21 (58)	-192 (83)	440 (64)
C(19)	3541 (4)	1942 (2)	7495 (9)	708 (31)	369 (25)	1381 (52)	-82 (67)	616 (78)	-194 (44)
C(20)	979 (4)	3757 (2)	6234 (6)	372 (22)	716 (30)	559 (26)	562 (51)	-262 (45)	-197 (44)
C(21)	-349 (4)	3623 (2)	6938 (6)	372 (21)	866 (32)	649 (30)	478 (58)	-118 (48)	84 (48)

had decreased to 0.10, the difference map was calculated. It showed the positions of all the hydrogen atoms which were then refined isotropically. Eight additional cycles completed the refinement, which was carried out by the block-diagonal approximation, with only the observed reflexions, minimizing the expression $\sum w(|F_o| - |F_c|)^2$ where $w = 1/\{1 + [(|F_o| - 40)/35]^2\}$ and $2.0 \leq |F_o| \leq 109.5$. The final R value $[= \sum |AF| / \sum |F_o|]$ for the observed reflexions was 0.049 and R_w , defined as $[\sum w(AF)^2 / \sum wF_o^2]^{1/2}$, was 0.041.

The final cycle gave the mean shift/error value for the non-hydrogen atoms of 0.15 and the maximum of 0.52. For hydrogen atoms all shifts were below 0.67 of the corresponding e.s.d.'s, except one which was equal to the e.s.d. value. The final difference map showed two peaks of $\sim 0.3 \text{ e } \text{Å}^{-3}$, one negative and one positive.

The atomic parameters and their estimated standard deviations as derived from the least-squares refinement, for C, N and O, are presented in Table 2 and those for H atoms in Table 3.* Two reflexions showed high discrepancies between F_o and F_c values: 130 appeared to be affected by extinction and the high F_o for 270 was probably due to multiple diffraction. Both reflexions were excluded from the final stages of refinement.

All calculations were carried out with the NRC crystallographic computer programs of Ahmed, Hall, Pippy & Huber (1973). The scattering factor curves were those of Hanson, Herman, Lea & Skillman (1964) except for the hydrogen atoms, for which the values of Stewart, Davidson & Simpson (1965) were used.

* A list of structure factors has been deposited with the British Library Lending Division as Supplementary Publication No. SUP 31439 (11 pp., 1 microfiche). Copies may be obtained through The Executive Secretary, International Union of Crystallography, 13 White Friars, Chester CH1 1NZ, England.

Discussion

A stereoscopic view of the molecule drawn by ORTEP program (Johnson, 1965) is shown in Fig. 1. The structure has the lycoctonine skeleton and the

Table 3. Fractional coordinates ($\times 10^3$) with e.s.d.'s and temperature factors for the hydrogen atoms

The number of the parent atom is obtained by omitting the last digit. H(1) is attached to O(1).

	<i>x</i>	<i>y</i>	<i>z</i>	<i>B</i> (Å ²)
H(1)	628 (4)	437 (2)	367 (6)	8.1 (1.4)
H(11)	315 (4)	407 (2)	1086 (5)	5.3 (1.0)
H(12)	417 (4)	357 (2)	1140 (6)	7.0 (1.2)
H(21)	160 (3)	325 (1)	1046 (5)	3.0 (0.8)
H(22)	232 (4)	313 (2)	1224 (6)	6.9 (1.2)
H(31)	221 (3)	219 (1)	1023 (5)	4.5 (0.9)
H(32)	373 (4)	240 (2)	1090 (5)	6.1 (1.1)
H(51)	525 (3)	283 (1)	856 (5)	3.8 (0.9)
H(61)	569 (3)	313 (1)	572 (5)	3.7 (0.8)
H(62)	434 (3)	282 (1)	524 (5)	3.6 (0.8)
H(71)	349 (3)	375 (1)	452 (5)	3.4 (0.8)
H(91)	671 (3)	386 (1)	680 (4)	2.7 (0.9)
H(111)	583 (4)	559 (2)	887 (5)	5.2 (1.0)
H(121)	388 (3)	481 (2)	957 (5)	4.7 (0.9)
H(122)	530 (4)	478 (2)	1074 (5)	5.4 (1.0)
H(131)	593 (4)	388 (2)	966 (5)	5.0 (1.0)
H(151)	287 (3)	431 (1)	745 (4)	2.4 (0.7)
H(161)	129 (3)	266 (1)	758 (4)	3.0 (0.8)
H(162)	225 (3)	273 (2)	584 (5)	4.3 (0.9)
H(171)	358 (5)	490 (2)	450 (7)	9.3 (1.5)
H(172)	511 (4)	513 (2)	423 (6)	7.9 (1.3)
H(181)	473 (4)	574 (2)	627 (6)	7.4 (1.2)
H(182)	357 (6)	535 (3)	729 (9)	14.0 (2.1)
H(191)	281 (4)	171 (2)	756 (6)	7.8 (1.3)
H(192)	442 (4)	179 (2)	828 (6)	8.5 (1.4)
H(193)	390 (4)	196 (2)	628 (6)	7.4 (1.3)
H(201)	112 (3)	419 (2)	601 (5)	4.6 (0.9)
H(202)	106 (4)	360 (2)	491 (5)	5.2 (1.0)
H(211)	-55 (4)	374 (2)	824 (5)	6.4 (1.1)
H(212)	-102 (4)	383 (2)	627 (6)	7.4 (1.3)
H(213)	-43 (5)	317 (2)	696 (6)	8.9 (1.4)

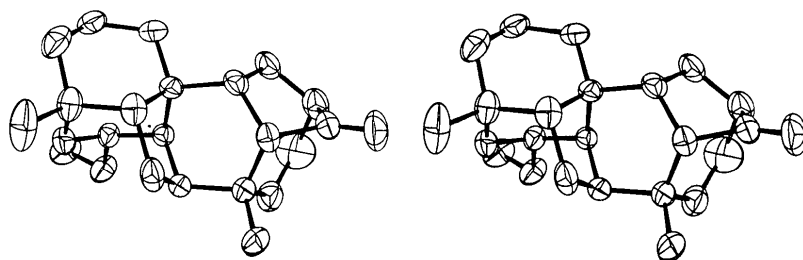


Fig. 1. Stereoscopic view. The thermal ellipsoids enclose 50% probability.

Table 4. Mean planes: $lX + mY + nZ = p$

(a) Deviations from the mean planes (Å). * Identifies atoms excluded from the calculation.

Ring A	Ring B	Ring C	Ring D	Ring E	Ring F	Carbonyl							
C(1)	0.063	C(14)	-0.088	C(14)	-0.059	C(13)	0.034	C(8)	0.003	C(11)	-0.018	C(9)	-0.003
C(14)	-0.042	C(15)	0.082	C(5)	0.109	C(14)	-0.034	C(9)	-0.003	C(12)	0.026	C(10)	0.015
C(3)	-0.081	C(16)	-0.111	C(7)	0.070	C(8)	-0.037	C(11)	0.004	C(13)	-0.020	C(11)	-0.004
C(4)	0.055	C(4)	0.111	C(6)	-0.132	C(7)	0.040	C(18)	-0.007	C(9)	0.012	O(2)	-0.004
C(2)*	0.587	N(1)*	-0.585	C(15)*	0.739	C(9)*	0.507	C(10)*	0.831	C(10)*	-0.647		
C(5)*	-0.677	C(5)*	0.830	C(15)*	-0.867	C(17)*	0.250						

Table 4 (cont.)

(b) Parameters referred to Cartesian coordinates in Å

	<i>l</i>	<i>m</i>	<i>n</i>	<i>p</i>	χ^2
Ring <i>A</i>	-0.9012	0.3401	-0.2688	-2.8617	924.9
Ring <i>B</i>	0.6202	-0.1811	-0.7633	-3.8268	2706.7
Ring <i>C</i>	-0.9410	-0.3310	-0.0704	-7.0653	2721.2
Ring <i>D</i>	0.6575	-0.7427	-0.1267	-3.9667	403.5
Ring <i>E</i>	0.7417	0.3951	-0.5420	5.3447	4.6
Ring <i>F</i>	-0.8560	-0.0038	-0.5170	-8.1821	103.3
Carbonyl	-0.2559	0.3104	-0.9155	-3.6793	16.4

CH₃CH₂, CH₃, OH and CO groups were found in the positions as predicted by Wiesner.

The values of bond lengths in Fig. 2(a) are not corrected for thermal vibration. The e.s.d.'s did not exceed 0.007 Å and they should be considered as underestimated. The C-C bond lengths cover a range of 1.502 to 1.573 Å, giving an average of 1.541 Å. The C-C bonds close to CO group are shortened as expected and the values for C-O (1.440 Å), C=O (1.195 Å) and C-N bonds (1.468 and 1.461 Å) also agree well with the theoretical values (*Molecular*

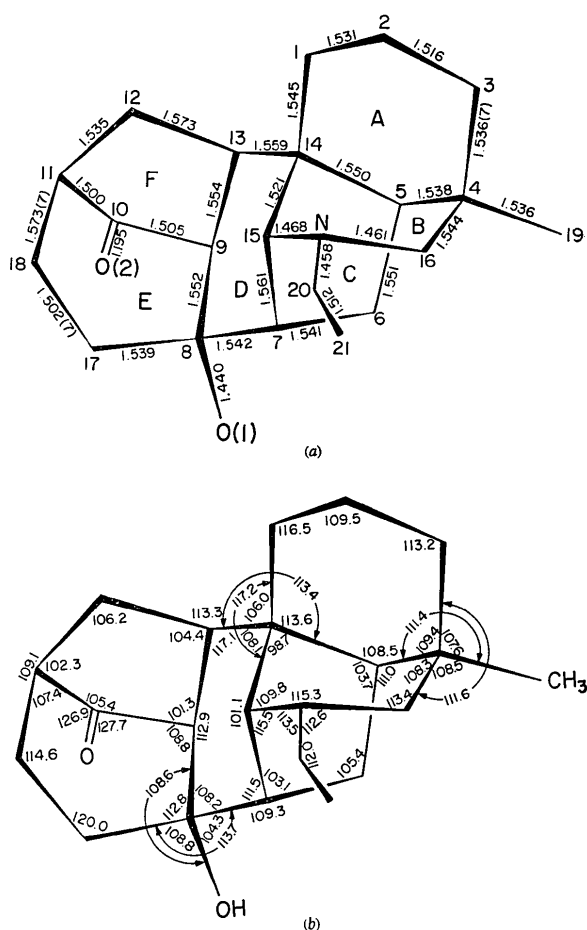


Fig. 2. (a) Bond lengths (Å). Unless otherwise indicated the e.s.d.'s are 0.006 Å. (b) Valency angles (°). The e.s.d.'s are 0.3°.

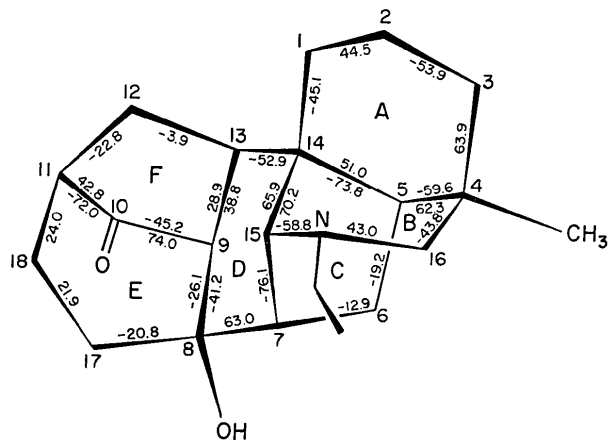


Fig. 3. Torsional angles (°). Additional values for ring *C*: C(15)-C(14)-C(5)-C(6) = 44.3°, C(6)-C(7)-C(15)-C(14) = 41.0° and C(7)-C(15)-C(14)-C(5) = -52.3°.

Structures & Dimensions, 1972). C-H bonds give an average of 1.04 Å and they range from 0.85 to 1.14 Å.

The angles are presented in Fig. 2(b). The five-membered rings give an average of 103.2° for their angles and they range from 98.7 to 106.2°. The mean for C-C-H and H-C-H angles is 107.9° and they vary from 90.8 to 129.5°.

The data regarding the mean planes for the different rings and the carbonyl group are given in Table 4. The C(10) atom of the carbonyl group shows a large deviation from the mean plane and only the four atoms of the ring *E* can be considered as planar.

The torsional angles are shown in Fig. 3. Their e.s.d.'s were calculated according to Huber (1961) and they vary from 0.3 to 0.5°. The signs of the angles are in agreement with the convention of the IUPAC-IUB Commission on Biological Nomenclature (1970).

Ring *A* is of somewhat distorted chair form. Rings *B* and *D* also have chair conformations but are appreciably flattened at N(1) and C(9). Ring *E* is of boat form with C(10) and C(17) at 0.83 and 0.25 Å from the plane of the other four atoms. Ring *C* has a distorted half-chair form and ring *F* is close to an envelope (Brutcher & Bauer, 1962.)

The molecular structures of a number of alkaloids with the lycotonine skeleton were determined by X-ray methods (Przybylska, 1961*a, b*, 1965). The conformational analysis of this type of skeleton in lappaconine hydrobromide (Birnbbaum, 1970) was described in full detail and with the exception of the difference in the conformation of the ring *A*, which is of a boat form in lappaconine, the two skeletons are identical. The ring *A* in the above mentioned structures assumes either the flattened-chair or the flattened-boat conformation depending on the position of the substituent groups.

The absolute stereochemistry of atisine was determined by optical rotatory dispersion methods (Dvornik & Edwards, 1958; ApSimon & Edwards, 1962;

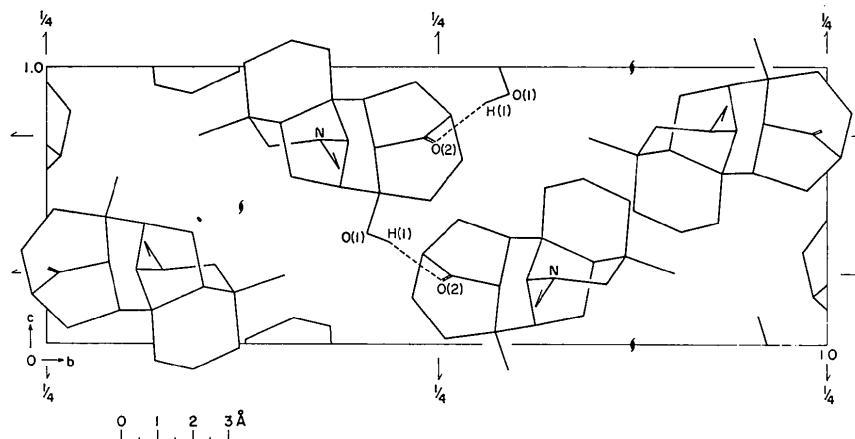


Fig. 4. Projection of the structure down the a axis. The hydrogen bonds are drawn with broken lines.

Vorbrueggen & Djerassi, 1962) and of lycoctonine by X-ray anomalous scattering (Przybylska & Marion, 1959). The configuration given in this publication being in agreement with these investigations can therefore be taken as absolute.

The packing of the molecules is shown in Fig. 4. The hydrogen bonds between O(1) and O(2) of the neighbouring molecules, shown by dashed lines, give rise to chains of H-bonded molecules parallel to the c axis. The pertinent data are: $O(1)\cdots O(2)=2.97$, $H(1)\cdots O(2)=2.15$ Å, $O(1)-H(1)\cdots O(2)=160.9^\circ$. It is interesting that the atom H(1) is also directed towards O(2) within the molecule, giving the intramolecular distance $O(1)\cdots O(2)$ of 3.72 Å and the angle $O(1)-H(1)\cdots O(2)$ of 112.3° . There are no other intermolecular distances that are significantly shorter than the sum of the van der Waals radii.

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