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1,2-Epoxy-3-hydroxy-α-lycoran-7-one

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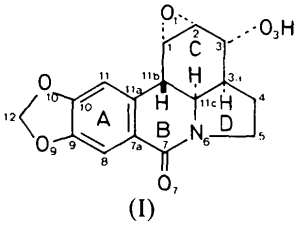
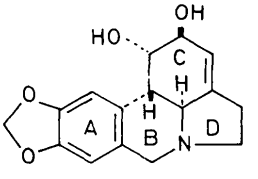
Abstract. $C_{16}H_{15}NO_5$. Monoclinic, $P2_1/c$, $a = 17{\cdot}06$ (2), $b = 9{\cdot}33$ (1), $c = 8{\cdot}25$ (1) Å, $\beta = 98{\cdot}4$ (2)°, $D_c = 1{\cdot}54$ g cm^{-3}, $Z = 4$. Full-matrix least-squares refinement gave $R = 0{\cdot}08$ for 199 parameters and 1453 significant reflexions [$I > 2\sigma(I)$]. The compound is an intermediate in the synthesis of lycorine.

Introduction. The title compound (I) is an intermediate in the total synthesis (Møller, Steinberg & Torssell, 1978) of lycorine (II). The structure was solved to determine the configuration of the epoxy and hydroxy groups relative to the junction of rings *B* and *D*.



(I)



(II)

Single crystals were obtained from an aqueous solution by controlled cooling (10°C d^{-1}). A crystal, 0·4 × 0·6 × 0·7 mm, was mounted on a Picker FACS-1 diffractometer and intensities were measured out to $2\theta = 45°$ with monochromated Mo $K\alpha$ radiation. Data were collected with the ω–2θ step-scanning technique, with a step length of 0·04° and a scan width of (3·92 + 0·692 tan θ)°. 1712 independent reflexions were obtained of which 1453 has $I > 2\sigma(I)$ according to counting statistics. No corrections were made for absorption.

The structure was determined with *MULTAN* (Germain, Main, & Woolfson, 1971). Least-squares

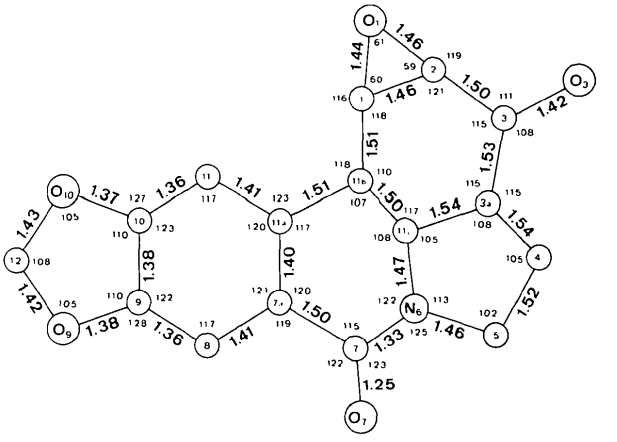


Fig. 1. Bond distances (Å) and angles (°). The mean estimated standard deviations are 0·006 Å for bond lengths and 0·4° for angles.

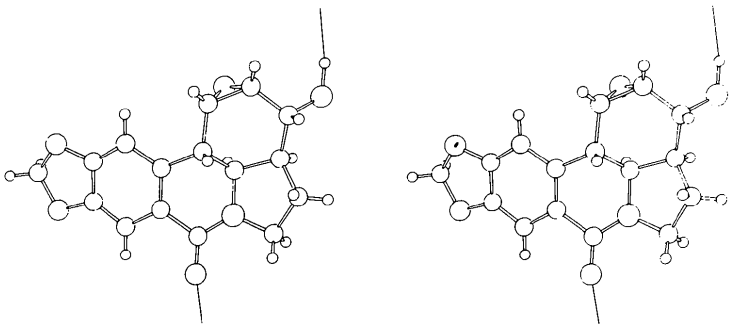


Fig. 2. A stereoview of one molecule. The single lines join the H atom of the hydrogen bond to the O atom of the neighbouring molecule.

Table 1. *Fractional atomic coordinates* ($\times 10^4$)

	x	*y*	*z*
O(1)	2814 (2)	2980 (3)	7216 (4)
O(3)	1022 (2)	1868 (3)	6443 (4)
O(7)	1688 (2)	9246 (3)	7579 (4)
O(9)	4804 (2)	9488 (3)	8244 (4)
O(10)	5238 (2)	7284 (3)	9312 (4)
N(6)	1560 (2)	6864 (3)	7038 (4)
C(1)	2777 (2)	3723 (4)	8734 (5)
C(2)	2204 (2)	2590 (5)	8202 (6)
C(3)	1357 (2)	2931 (4)	7562 (5)
C(3a)	1218 (2)	4392 (4)	6732 (5)
C(4)	532 (2)	5270 (4)	7254 (6)
C(5)	693 (2)	6804 (5)	6766 (6)
C(7)	1982 (3)	8023 (4)	7525 (5)
C(7a)	2848 (2)	7761 (4)	8023 (5)
C(8)	3383 (2)	8896 (5)	7896 (5)
C(9)	4168 (3)	8598 (5)	8321 (5)
C(10)	4427 (2)	7273 (5)	8913 (5)
C(11)	3926 (2)	6169 (5)	9098 (5)
C(11a)	3113 (2)	6409 (4)	8586 (5)
C(11b)	2490 (2)	5260 (4)	8585 (5)
C(11c)	1926 (2)	5441 (4)	7021 (5)
C(12)	5482 (3)	8592 (5)	8621 (7)
H(1)	2982	3554	9896
H(2)	2188	1961	9151
H(3)	1062	2909	8467
H(3a)	1100	4230	5570
H(41)	543	5213	8437
H(42)	25	4935	6757
H(51)	462	7513	7382
H(52)	490	6972	5611
H(8)	3189	9841	7518
H(11)	4114	5254	9570
H(11b)	2210	5420	9500
H(11c)	2220	5340	6110
H(121)	5699	8399	7632
H(122)	5876	9071	9368
H(13)	1218	813	6785

refinement (*LINUS*, Coppens & Hamilton; 1970) of atomic coordinates, anisotropic thermal parameters and a scale factor gave a final R of 0·08 and R_w = 0·10. The H atoms, other than that of the hydrogen bond, were included in the final cycle but their parameters were not refined. A final difference map showed the position of H(13) of the hydrogen bond. The weighting scheme was $w = \{[\sigma(F_o)^2 + 1{\cdot}03F_o^2]^{1/2} - |F_o|\}^{-2}$. The scattering factors were those of Cromer & Mann (1968) for C, N and O and of Stewart, Davidson & Simpson (1965) for H.

Atomic coordinates are given in Table 1,* bond lengths and angles in Fig. 1.

Discussion. A stereodrawing of the molecule is shown in Fig. 2. Ring *B* has a distorted boat form, as does *C*. *D* is in the envelope form with C(4) out of the plane. The epoxy group forces *C* into the boat form. The junction between *B* and *C* is *trans*, and between *C* and *D* *cis*, as is the case in lycorine (Roques, Piquion, Fourme & André, 1974; Gopalakrishna, Watson, Pacheco & Silva, 1976), lycorine hydrobromide (Roques & Cotrait, 1974), and dihydrolycorine hydrobromide (Shiro, Sato & Koyama, 1968).

Bond lengths and angles have the expected values. N(6)–C(7)–O(7) is partially delocalized. There is hydrogen bonding between molecules related by a unit-cell translation along **b**, O(3)–O(7) = 2·80 Å, so that there are infinite chains of molecules parallel to **b**.

* Lists of structure factors and thermal parameters have been deposited with the British Library Lending Division as Supplementary Publication No. SUP 33189 (8 pp.). Copies may be obtained through The Executive Secretary, International Union of Crystallography, 13 White Friars, Chester CH1 1NZ, England.

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