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Structure of 1,2,3,9b-Tetrahydro-9bβ-hydroxy-2β-methoxy-1α-phenyl-5*H*-pyrrolo-[2,1-*a*]isoindol-5-one

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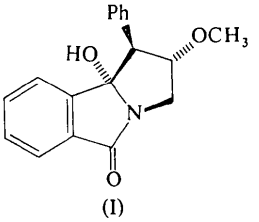
Abstract. $C_{18}H_{17}NO_3$, $M_r = 295{\cdot}3$, orthorhombic, $P2_12_12_1$, $a = 9{\cdot}141\ (3)$, $b = 8{\cdot}670\ (3)$, $c = 18{\cdot}538\ (5)$ Å, $V = 1469{\cdot}2$ Å^3, $Z = 4$, $D_c = 1{\cdot}335$ Mg m^{-3}. The structure was refined to $R = 0{\cdot}054$ for 1281 reflections. The marginal five-membered ring takes an envelope conformation.

Introduction. It has been reported that *N*-2- and *N*-3-alkenylphthalimides undergo photochemical cyclization in methanol (Maruyama, Kubo, Machida, Oda, Kanaoka & Fukuyama, 1978). In order to confirm the molecular structure of a product (I) obtained by the photolysis of *N*-(3-phenyl-2-propenyl)phthalimide in methanol, a single-crystal X-ray analysis has been undertaken.



(I)

A preliminary photographic investigation showed the crystal to be orthorhombic with systematic absences uniquely characterizing space group $P2_12_12_1$. The unit-cell constants were obtained from the least-squares treatment of the angular settings of 13 reflections measured on a Rigaku computer-controlled four-circle diffractometer with Ni-filtered Cu $K\alpha$ radiation. The intensities were measured by the θ–2θ scan technique with a scan speed of 4° min^{-1} in 2θ. The backgrounds were counted for 8 s at each end of the scan range. The intensities of 1281 reflections were measured in the range $0 < \sin\theta/\lambda < 0{\cdot}56$ Å^{-1}, and corrected for the Lorentz and polarization factors. The structure was solved by application of *MULTAN* using the 250 largest $|E|$'s (Germain, Main & Woolfson, 1971). The *E* map computed from the phase set with the largest figure of merit revealed all 22 non-hydrogen atoms. The structure was refined by the block-diagonal least-squares method (Ashida, 1973) with anisotropic temperature factors for non-hydrogen atoms. All H atoms were located in the difference Fourier synthesis, and included in the refinement with isotropic temperature factors. The weighting scheme used in the final cycle of the refinement was: $w = 0{\cdot}3$ for $F_o = 0$ and $w = [\sigma^2(F_o) + 0{\cdot}022\ F_o + 0{\cdot}00037\ F_o^2]^{1/2}$ for $F_o > 0$, where the values of $\sigma(F_o)$ were calculated from counting statistics. The final R value was 0·054 for 1281 reflections (0·041 for 1160 non-zero reflections). The atomic scattering factors were taken from *International Tables for X-ray Crystallography* (1974). The final atomic coordinates are given in Table 1.*

* Lists of structure factors and thermal parameters have been deposited with the British Library Lending Division as Supplementary Publication No. SUP 35206 (8 pp.). Copies may be obtained through The Executive Secretary, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

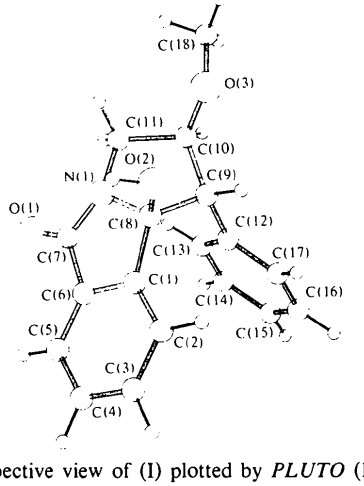


Fig. 1. A perspective view of (I) plotted by *PLUTO* (Motherwell, 1976).

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Table 1. *Atomic parameters with e.s.d.'s in parentheses*

	x	y	z	B_{eq} or B (Å^2)
C(1)	0·2621 (4)	0·7659 (4)	0·3464 (2)	3·1
C(2)	0·1491 (4)	0·8337 (4)	0·3843 (2)	3·7
C(3)	0·0187 (5)	0·8617 (5)	0·3482 (2)	4·4
C(4)	0·0014 (5)	0·8199 (5)	0·2760 (2)	4·7
C(5)	0·1137 (5)	0·7502 (5)	0·2387 (2)	4·2
C(6)	0·2441 (4)	0·7248 (4)	0·2742 (2)	3·1
C(7)	0·3834 (4)	0·6568 (4)	0·2481 (2)	3·5
C(8)	0·4177 (4)	0·7335 (4)	0·3688 (2)	2·8
C(9)	0·4553 (4)	0·6011 (4)	0·4222 (2)	3·0
C(10)	0·6052 (4)	0·5416 (4)	0·3948 (2)	3·3
C(11)	0·6143 (4)	0·5873 (5)	0·3139 (2)	4·1
C(12)	0·3408 (4)	0·4758 (4)	0·4276 (2)	2·8
C(13)	0·3284 (4)	0·3582 (4)	0·3774 (2)	3·5
C(14)	0·2269 (5)	0·2407 (5)	0·3860 (2)	4·1
C(15)	0·1348 (5)	0·2399 (5)	0·4450 (2)	4·2
C(16)	0·1451 (4)	0·3568 (5)	0·4954 (2)	3·7
C(17)	0·2455 (4)	0·4760 (5)	0·4866 (2)	3·4
C(18)	0·8528 (5)	0·5295 (6)	0·4314 (2)	4·9
N(1)	0·4830 (3)	0·6788 (4)	0·3010 (1)	3·1
O(1)	0·4049 (3)	0·5910 (4)	0·1903 (1)	4·9
O(2)	0·4901 (3)	0·8647 (3)	0·3963 (1)	3·6
O(3)	0·7170 (3)	0·6120 (3)	0·4356 (1)	4·0
H(2)	0·165 (4)	0·865 (5)	0·438 (2)	2·5
H(3)	−0·073 (5)	0·921 (6)	0·376 (2)	4·7
H(4)	−0·098 (4)	0·834 (5)	0·250 (2)	3·0
H(5)	0·105 (4)	0·723 (5)	0·188 (2)	3·3
H(9)	0·458 (4)	0·648 (4)	0·472 (2)	1·6
H(10)	0·617 (4)	0·417 (5)	0·399 (2)	1·8
H(11*A*)	0·621 (4)	0·501 (5)	0·282 (2)	3·2
H(11*B*)	0·709 (4)	0·649 (5)	0·304 (2)	3·4
H(13)	0·391 (5)	0·362 (5)	0·334 (2)	3·8
H(14)	0·219 (4)	0·153 (5)	0·347 (2)	2·4
H(15)	0·066 (4)	0·157 (5)	0·453 (2)	3·7
H(16)	0·080 (4)	0·357 (5)	0·538 (2)	3·4
H(17)	0·248 (4)	0·568 (5)	0·520 (2)	2·3
H(18*A*)	0·900 (4)	0·536 (5)	0·381 (2)	2·6
H(18*B*)	0·928 (4)	0·593 (6)	0·461 (2)	3·7
H(18*C*)	0·843 (5)	0·428 (5)	0·433 (2)	3·2
H(O2)	0·509 (5)	0·944 (5)	0·360 (2)	3·1

Table 2. *Bond lengths* (Å) *and angles* (°) *with e.s.d.'s in parentheses*

C(1)–C(2)	1·381 (5)	C(1)–C(6)	1·395 (5)
C(1)–C(8)	1·508 (5)	C(2)–C(3)	1·389 (6)
C(3)–C(4)	1·394 (6)	C(4)–C(5)	1·378 (6)
C(5)–C(6)	1·379 (6)	C(6)–C(7)	1·484 (6)
C(7)–O(1)	1·230 (5)	C(7)–N(1)	1·351 (5)
C(8)–C(9)	1·554 (5)	C(8)–O(2)	1·411 (4)
C(8)–N(1)	1·469 (5)	C(9)–C(10)	1·550 (5)
C(9)–C(12)	1·512 (5)	C(10)–C(11)	1·552 (6)
C(10)–O(3)	1·411 (5)	C(11)–N(1)	1·459 (5)
C(12)–C(13)	1·386 (5)	C(12)–C(17)	1·398 (5)
C(13)–C(14)	1·386 (6)	C(14)–C(15)	1·380 (6)
C(15)–C(16)	1·382 (6)	C(16)–C(17)	1·392 (6)
C(18)–O(3)	1·435 (6)		

C(2)–C(1)–C(6)	120·6 (4)	C(2)–C(1)–C(8)	130·2 (3)
C(6)–C(1)–C(8)	109·1 (3)	C(1)–C(2)–C(3)	118·1 (4)
C(2)–C(3)–C(4)	121·0 (4)	C(3)–C(4)–C(5)	120·7 (4)
C(4)–C(5)–C(6)	118·3 (4)	C(1)–C(6)–C(5)	121·3 (4)
C(1)–C(6)–C(7)	108·2 (3)	C(5)–C(6)–C(7)	130·5 (4)
C(6)–C(7)–O(1)	127·3 (4)	C(6)–C(7)–N(1)	106·6 (3)
O(1)–C(7)–N(1)	126·1 (4)	C(1)–C(8)–C(9)	121·4 (3)
C(1)–C(8)–O(2)	113·0 (3)	C(1)–C(8)–N(1)	102·0 (3)
C(9)–C(8)–O(2)	105·1 (3)	C(9)–C(8)–N(1)	102·5 (3)
O(2)–C(8)–N(1)	112·3 (3)	C(8)–C(9)–C(10)	103·4 (3)
C(8)–C(9)–C(12)	114·8 (3)	C(10)–C(9)–C(12)	113·3 (3)
C(9)–C(10)–C(11)	106·2 (3)	C(9)–C(10)–O(3)	108·7 (3)
C(11)–C(10)–O(3)	111·7 (3)	C(10)–C(11)–N(1)	104·7 (3)
C(9)–C(12)–C(13)	122·7 (3)	C(9)–C(12)–C(17)	118·9 (3)
C(13)–C(12)–C(17)	118·4 (3)	C(12)–C(13)–C(14)	121·2 (4)
C(13)–C(14)–C(15)	120·2 (4)	C(14)–C(15)–C(16)	119·4 (2)
C(15)–C(16)–C(17)	120·7 (4)	C(12)–C(17)–C(16)	120·1 (4)
C(10)–O(3)–C(18)	112·4 (3)	C(7)–N(1)–C(8)	113·1 (3)
C(7)–N(1)–C(11)	126·7 (3)	C(8)–N(1)–C(11)	111·6 (3)

Discussion. The molecular structure has been established as (I) by the present analysis. A perspective view of the molecule is shown in Fig. 1, together with the atom-numbering scheme. Bond lengths and angles for the non-hydrogen atoms are listed in Table 2. The five-membered ring comprising C(8), C(9), C(10), C(11), and N(1) takes an envelope conformation with C(8) deviating by 0·51 (1) Å from the plane through the remaining four atoms. The phenyl and hydroxy groups are *trans* to each other and occupy quasi-axial positions in the ring. N(1) deviates by 0·13 (1) Å from the plane through C(1), C(6), C(7), and C(8). An intermolecular hydrogen bond between the hydroxy group and the carbonyl oxygen connects molecules related by the screw axis parallel to **b**. The distance from O(2) to O(1) of the molecule at $(1-x, \frac{1}{2}+y, \frac{1}{2}-z)$ is 2·710 (4) Å, and H(O2)···O(1) is 1·76 (4) Å. The angles O(2)–H(O2)···O(1) and H(O2)···O(1)–C(7) are 162 (4) and 150 (1)° respectively. There is no unusually short contact in this crystal.

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