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Structure of 2-Benzoyl-3-phenylindole

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Abstract. $C_{21}H_{15}NO$, $M_r = 297{\cdot}36$, triclinic, $P\bar{1}$, $a = 10{\cdot}702\ (1)$, $b = 9{\cdot}776\ (1)$, $c = 8{\cdot}265\ (1)$ Å, $\alpha = 106{\cdot}43\ (1)$, $\beta = 103{\cdot}63\ (1)$, $\gamma = 99{\cdot}63\ (1)°$, $V = 780{\cdot}2\ (2)$ Å^3, $Z = 2$, $D_x = 1{\cdot}266$ g cm^{-3}, $\lambda(\text{Cu } K\alpha) = 1{\cdot}54184$ Å, $\mu = 5{\cdot}73$ cm^{-1}, $F(000) = 312$, $T = 291$ K, $R = 0{\cdot}0481$, $wR = 0{\cdot}0439$ for 2034 unique observed reflections. The structure has normal bond lengths and angles. The indole group is nearly planar; the maximum deviations from the least-squares plane are 0·003 (2) (benzene ring) and 0·004 (2) Å (pyrrole ring), the dihedral angle between the two ring planes being 2·0 (1)°. The molecule dimerizes around the centre of symmetry by a pair of hydrogen bonds.

Experimental. Crystals by evaporation from CH_3CN, yellow transparent prisms, specimen size 0·52 × 0·25 × 0·15 mm. Philips PW1100 diffractometer, graphite-monochromatized Cu $K\alpha$ radiation. Data collection by $\omega/2\theta$ scan, scan width 1·00°, scan speed 2·10° min^{-1}. 2504 reflections measured, $2\theta_{max}$ 134°, 2034 unique with $I \geq 3\sigma(I)$, $-11 \leq h \leq 11$, $-10 \leq k \leq 10$, $0 \leq l \leq 9$, three standard reflections monitored every 90 min, no significant decay. No absorption correction applied. Lattice parameters from least-squares refinement of 15 reflections, $23{\cdot}67 \leq \theta \leq 59{\cdot}87°$. *MULTAN*80 (Main, Fiske, Hull, Lessinger, Germain, Declercq & Woolfson, 1980) solved the structure. Least-squares refinement with *SHELX*76 (Sheldrick, 1976) minimizing $\sum w(\Delta F)^2$. Non-H atoms anisotropic, H1 isotropically refined, remaining H atoms at calculated positions with isotropic group temperature factors. $R = 0{\cdot}0481$ and $wR = 0{\cdot}0439$, $w = 1/[\sigma^2(F) + 0{\cdot}0001F^2]$, for 2034 observations with $F \geq 6\sigma(F)$ and 257 parameters. Max. $\Delta/\sigma = 0{\cdot}001$ for any parameter, max. and min. values in final $\Delta\rho$ map 0·19 and −0·32 e Å^{-3} respectively. Atomic scattering factors from *International Tables for X-ray Crystallography* (1974). Final atomic parameters are given in Table 1;*

* Lists of structure factors, anisotropic thermal parameters, H-atom parameters, torsion angles and least-squares-planes' data have been deposited with the British Library Document Supply Centre as Supplementary Publication No. SUP 44272 (39 pp.). Copies may be obtained through The Executive Secretary, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

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Table 1. *Fractional atomic coordinates and equivalent isotropic temperature factors with e.s.d.'s in parentheses*

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

	x	y	z	B_{eq}(Å^2)
N1	−0·0703 (2)	0·1379 (2)	0·1796 (2)	4·00 (5)
C2	0·0327 (2)	0·1785 (2)	0·3344 (2)	3·65 (6)
C3	−0·0107 (2)	0·2438 (2)	0·4765 (2)	3·61 (5)
C3a	−0·1467 (2)	0·2417 (2)	0·4033 (2)	3·71 (6)
C4	−0·2450 (2)	0·2857 (2)	0·4779 (3)	4·39 (6)
C5	−0·3707 (2)	0·2622 (2)	0·3671 (3)	4·99 (8)
C6	−0·4009 (2)	0·1951 (2)	0·1827 (3)	5·05 (7)
C7	−0·3074 (2)	0·1510 (2)	0·1063 (3)	4·68 (7)
C7a	−0·1801 (2)	0·1742 (2)	0·2183 (2)	3·82 (6)
C8	0·1547 (2)	0·1330 (2)	0·3230 (2)	3·77 (7)
O9	0·1555 (1)	0·0403 (2)	0·1870 (3)	4·98 (5)
C10	0·2766 (2)	0·1950 (2)	0·4771 (2)	3·62 (6)
C11	0·3217 (1)	0·3449 (2)	0·5717 (2)	4·13 (6)
C12	0·4341 (2)	0·3962 (2)	0·7166 (3)	4·95 (7)
C13	0·4998 (2)	0·2996 (2)	0·7705 (3)	5·29 (8)
C14	0·4555 (2)	0·1503 (2)	0·6755 (3)	5·24 (8)
C15	0·3455 (2)	0·0983 (2)	0·5282 (3)	4·40 (7)
C16	0·0602 (2)	0·2973 (2)	0·6685 (2)	3·72 (6)
C17	0·0752 (2)	0·4430 (2)	0·7704 (2)	4·48 (6)
C18	0·1420 (2)	0·4941 (2)	0·9492 (3)	5·32 (7)
C19	0·1936 (2)	0·4011 (3)	1·0290 (3)	5·64 (7)
C20	0·1787 (2)	0·2565 (2)	0·9302 (3)	5·31 (8)
C21	0·1114 (2)	0·2042 (2)	0·7503 (2)	4·37 (6)

Table 2. *Bond lengths* (Å) *and angles* (°) *for selected atoms and hydrogen-bond data* (Å, °) *with e.s.d.'s in parentheses*

N1–C2	1·382 (2)	C3a–C7a	1·412 (2)
N1–C7a	1·365 (3)	C4–C5	1·378 (3)
C2–C3	1·392 (3)	C5–C6	1·410 (3)
C2–C8	1·462 (3)	C6–C7	1·370 (3)
C3–C3a	1·432 (3)	C7–C7a	1·396 (3)
C3–C16	1·482 (2)	C8–O9	1·232 (2)
C3a–C4	1·407 (3)	C8–C10	1·486 (2)

C2–N1–C7a	109·4 (2)	C3a–C4–C5	118·6 (2)
N1–C2–C8	117·7 (2)	C4–C5–C6	121·0 (2)
N1–C2–C3	109·1 (2)	C5–C6–C7	121·8 (2)
C3–C2–C8	132·8 (2)	C6–C7–C7a	117·4 (2)
C2–C3–C16	129·4 (2)	C3a–C7a–C7	122·2 (2)
C2–C3–C3a	106·3 (2)	N1–C7a–C7	129·8 (2)
C3a–C3–C16	124·2 (2)	N1–C7a–C3a	108·0 (2)
C3–C3a–C7a	107·3 (2)	C2–C8–C10	120·2 (2)
C3–C3a–C4	133·5 (2)	C2–C8–O9	119·9 (2)
C4–C3a–C7a	119·1 (2)	O9–C8–C10	119·8 (2)

N1···O9($-x,-y,-z$)	2·868 (2)	N1–H1···O9	151 (2)
H1···O9	2·02 (2)		

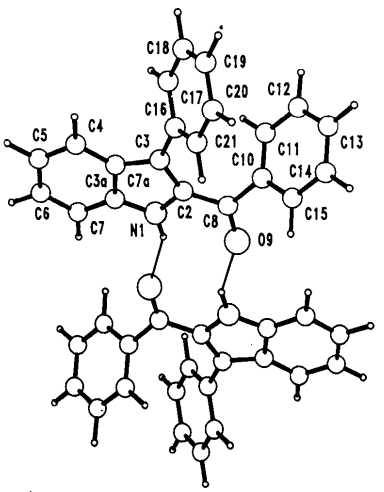

Fig. 1. View of the molecule, with atomic numbering, hydrogen-bonded to its centrosymmetric equivalent.

Table 2 lists bond lengths and angles for selected atoms and hydrogen-bond data. Fig. 1 shows the centrosymmetric arrangement of two hydrogen-bonded molecules, with atomic numbering of the unique molecule. Drawing by *PLUTO* (Motherwell & Clegg, 1978), geometrical calculations by *PARST* (Nardelli, 1983).

Related literature. The structure agrees very well with the X-ray structure of ethyl 3-phenyl-4,5,6,7-tetrahydroindole-2-carboxylate (Law, Lai, Sammes, Katritzky & Mak, 1984) regarding bond lengths and angles as well as hydrogen-bonding properties.

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The Structure of an Intermediate in the Synthesis of Avermectin B_{1a}

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Abstract. (±)-(2a*R**,4a*R**,5a*S**,6a*R**,6b*R**,6c*R**)-Octahydro-2a-methoxy-5a-methyl-4*H*-furo[2,3,4-*cd*]-oxireno[*g*]benzofuran-4-one, $C_{11}H_{14}O_5$, $M_r = 226{\cdot}23$, orthorhombic, $P2_12_12_1$ (spontaneous resolution), $a = 12{\cdot}705$ (2), $b = 5{\cdot}725$ (1), $c = 14{\cdot}617$ (2) Å, $V = 1063{\cdot}2$ (5) Å^3, $Z = 4$, $D_m = 1{\cdot}43$, $D_x = 1{\cdot}41$ Mg m^{-3}, λ(Mo $K\alpha$) = 0·71069 Å, $\mu = 0{\cdot}120$ mm^{-1}, $F(000) = 480$, $T = 293$ K, $R = 0{\cdot}043$ for 1690 observed unique reflections. The absolute configuration is not assigned. The C5—O1—C6 epoxide angle is 60·4 (1)° and the closest intermolecular contact (O1—C11) is 3·142 (3) Å. There are no unusual structural features.

Experimental. Colorless crystal of dimensions 0·2 × 0·5 × 0·6 mm. D_m by flotation in hexane/carbon tetrachloride. Syntex $P\bar{1}$ diffractometer with incident-beam monochromator, 15 centered reflections within $35 \le 2\theta \le 52°$ used for determining lattice parameters. Absorption ignored. $(\sin\theta/\lambda)_{max} = 1{\cdot}275$ Å^{-1}, range of *hkl*: $0 \le h \le 19$, $0 \le k \le 8$, $0 \le l \le 19$. Five standard reflections monitored every 200 reflections with random variation of 4·0% over data collection, θ–2θ scans of 2° min^{-1} in 2θ, 2256 independent reflections collected, 1690 observed [$F_o > 3\sigma(F_o)$]. Structure solved by direct methods with *MITHRIL* (Gilmore, 1983), *DIRDIF* (Beurskens, 1984), and Fourier procedures. All H atoms except H11*B* located in difference maps; constrained to idealized positions with isotropic $B = 1{\cdot}2 \times B$ of bonded atom. $\sum w(F_o - F_c)^2$ minimized where $w = 1/\sigma^2(F_o)$. 145 parameters refined: atom coordinates, anisotropic temperature factors for all non-H atoms, $\Delta/\sigma = 0{\cdot}00$, $R = 0{\cdot}043$, $wR = 0{\cdot}053$, $S = 1{\cdot}64$. Final difference electron density excursions between −0·15 and 0·24 e Å^{-3}. Atomic scattering factors from *International Tables for X-ray Crystallography* (1974) and programs used were those from the Texray Crystallographic Software Package (Molecular Structure Corporation, 1985). Atom num-

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