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Structure of an Indole Derivative

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Abstract. Dimethyl 1-acetyl-6-hydroxyindole-4,5-dicarboxylate, $C_{14}H_{13}NO_6$, $M_r = 291{\cdot}26$, orthorhombic, $Pna2_1$ (No. 33), $a = 6{\cdot}939$ (1), $b = 10{\cdot}182$ (3), $c = 19{\cdot}184$ (8) Å, $V = 1355{\cdot}41$ Å^3, $Z = 4$, $D_m = 1{\cdot}42$, $D_x = 1{\cdot}43$ Mg m^{-3}, λ(Cu $K\alpha$) = 1·54178 Å, $\mu = 0{\cdot}97$ mm^{-1}, $F(000) = 608$, $T = 293$ K. Final $R = 0{\cdot}056$ for 882 unique reflections. The C5 and C6 rings are planar. Dihedral angles between benzene ring and C(10)O(3)O(4), C(8)O(1)O(2) groups are 89·0 (2) and 1·3 (2)°, respectively. There are van der Waals forces only between molecules.

Experimental. Colourless prismatic crystal with dimensions 0·15 × 0·15 × 0·75 mm; D_m by flotation in *n*-octane/CCl_4; space group orthorhombic, $Pna2_1$; Syntex $P2_1$ diffractometer, graphite monochromator, $\theta/2\theta$ scan, $2\theta_{\max} = 110°$, time per reflection *ca* 60 s; two standard reflections, variation 3·5%; 25 reflections with $9{\cdot}2 < 2\theta < 33{\cdot}8°$ used for refinement of lattice parameters; absorption correction was not applied; index range $0 \le h \le 7$, $0 \le k \le 10$, $0 \le l \le 20$, 1062 reflections measured, 882 unique, 98 unobserved with $I < 1{\cdot}5\sigma(I)$. Choice of lattice parameters from program *UB* (Sivý, Sivý & Koreň, 1987). Data reduction carried out with program *XP*21 (Pavelčík, 1987). The structure was solved by *MULTAN*80 (Main, Fiske, Hull, Lessinger, Germain, Declercq & Woolfson, 1980). *MULTAN*80 did not give a unique solution. A cluster of peaks forming a hexagonal net was found which gave several choices for the six-membered ring. One selected ring led by Fourier expansion to location of a whole molecule. Two possible positions for one of the methoxycarbonyl groups was found and after isotropic refinement (by full matrix based on *F* values) the correct alternative could be identified. H atoms [found from difference Fourier map; H(2), H(13*A*), H(13*B*), H(13*C*) calculated] were not refined. Final refinement was with all non-H atoms anisotropic [all refinements with *SHELX*76, (Sheldrick, 1976)]. Scattering factors and f', f'' from *International Tables for X-ray Crystallography* (1974). Maximum positive and negative electron density in final difference Fourier synthesis 0·3 and $-0{\cdot}5$ e Å^{-3}. Final $R = 0{\cdot}056$, $wR = 0{\cdot}058$, $w = 1{\cdot}0/[\sigma^2(F_o) + 0{\cdot}005F_o^2]$. $(\Delta/\sigma)_{\max} = 0{\cdot}15$ in final refinement cycle (190 parameters). Calculations performed on an M4030-1 computer, Slovak Technical University, Bratislava, Czechoslovakia.

Table 1. *Fractional coordinates and equivalent isotropic thermal parameters* (Å^2) *with e.s.d.'s in parentheses for non*-H *atoms*

$$B_{\text{eq}} = \tfrac{4}{3}\textstyle\sum_i\sum_j\beta_{ij}\mathbf{a}_i.\mathbf{a}_j.$$

	x	y	z	B_{eq}
N(1)	0·3749 (6)	0·3260 (4)	0·3123 (3)	2·9 (1)
C(2)	0·3924 (7)	0·4040 (6)	0·2514 (4)	3·3 (1)
C(3)	0·4053 (7)	0·3256 (5)	0·1956 (3)	3·0 (1)
C(4')	0·3936 (7)	0·1916 (5)	0·2187 (3)	2·6 (2)
C(7')	0·3750 (7)	0·1939 (6)	0·2916 (3)	2·9 (1)
C(12)	0·3563 (8)	0·3725 (7)	0·3805 (4)	3·9 (2)
O(5)	0·3331 (9)	0·2957 (6)	0·4283 (3)	6·5 (2)
C(13)	0·3630 (9)	0·5187 (7)	0·3910 (4)	4·8 (2)
C(4)	0·3952 (7)	0·0714 (5)	0·1842 (4)	2·7 (2)
C(5)	0·3836 (7)	−0·0472 (6)	0·2227 (4)	3·2 (2)
C(6)	0·3665 (8)	−0·0385 (6)	0·2964 (3)	3·7 (2)
C(7)	0·3603 (9)	0·0794 (5)	0·3314 (4)	3·7 (2)
O(6)	0·3536 (8)	−0·1484 (5)	0·3371 (3)	5·8 (1)
C(10)	0·3979 (9)	0·0770 (5)	0·1048 (4)	3·3 (2)
O(3)	0·2568 (7)	0·0832 (4)	0·0697 (3)	4·8 (1)
O(4)	0·5775 (6)	0·0814 (5)	0·0813 (3)	4·6 (1)
C(11)	0·5988 (13)	0·0925 (10)	0·0066 (5)	7·1 (3)
C(8)	0·3888 (8)	−0·1804 (6)	0·1902 (4)	3·8 (2)
O(1)	0·3736 (7)	−0·2807 (4)	0·2230 (3)	6·2 (2)
O(2)	0·4088 (6)	−0·1787 (4)	0·1220 (3)	4·3 (1)
C(9)	0·4101 (9)	−0·3040 (7)	0·0867 (5)	5·5 (2)

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Table 2. *Bond lengths* (Å) *and angles* (°) *with e.s.d.'s in parentheses for non*-H *atoms*

N(1)—C(2)	1·418 (9)	C(7′)—C(4′)	1·405 (8)
N(1)—C(7′)	1·402 (7)	C(7′)—C(7)	1·397 (9)
N(1)—C(12)	1·397 (9)	C(8)—O(1)	1·204 (8)
C(3)—C(2)	1·338 (9)	C(8)—O(2)	1·316 (10)
C(3)—C(4′)	1·437 (7)	C(8)—C(5)	1·493 (9)
C(4)—C(4′)	1·391 (8)	C(9)—O(2)	1·444 (9)
C(4)—C(5)	1·418 (9)	C(10)—O(3)	1·190 (9)
C(4)—C(10)	1·524 (11)	C(10)—O(4)	1·326 (8)
C(6)—C(5)	1·422 (10)	C(11)—O(4)	1·445 (11)
C(6)—O(6)	1·367 (8)	C(12)—O(5)	1·216 (10)
C(6)—C(7)	1·376 (8)	C(12)—C(13)	1·503 (10)

C(2)—N(1)—C(7′)	107·7 (5)	O(6)—C(6)—C(7)	115·7 (6)
C(2)—N(1)—C(12)	126·1 (5)	C(6)—C(7)—C(7′)	117·3 (6)
C(7′)—N(1)—C(12)	126·2 (5)	C(4′)—C(7′)—N(1)	107·3 (5)
N(1)—C(2)—C(3)	109·3 (5)	C(4′)—C(7′)—C(7)	122·5 (6)
C(2)—C(3)—C(4′)	108·4 (5)	N(1)—C(7′)—C(7)	130·2 (6)
C(3)—C(4′)—C(4)	133·5 (6)	O(1)—C(8)—O(2)	122·7 (6)
C(3)—C(4′)—C(7′)	107·2 (5)	O(1)—C(8)—C(5)	123·4 (7)
C(4)—C(4′)—C(7′)	119·3 (5)	O(2)—C(8)—C(5)	113·9 (6)
C(4′)—C(4)—C(5)	120·1 (7)	C(8)—O(2)—C(9)	117·1 (6)
C(4′)—C(4)—C(10)	116·3 (5)	O(3)—C(10)—O(4)	125·4 (7)
C(5)—C(4)—C(10)	123·6 (5)	O(3)—C(10)—C(4)	123·9 (6)
C(4)—C(5)—C(6)	118·0 (6)	O(4)—C(10)—C(4)	110·6 (6)
C(4)—C(5)—C(8)	123·7 (7)	C(10)—O(4)—C(11)	115·8 (6)
C(6)—C(5)—C(8)	118·3 (6)	O(5)—C(12)—N(1)	120·0 (6)
C(5)—C(6)—C(7)	122·8 (6)	O(5)—C(12)—C(13)	122·7 (7)
C(5)—C(6)—O(6)	121·5 (6)	N(1)—C(12)—C(13)	117·3 (6)

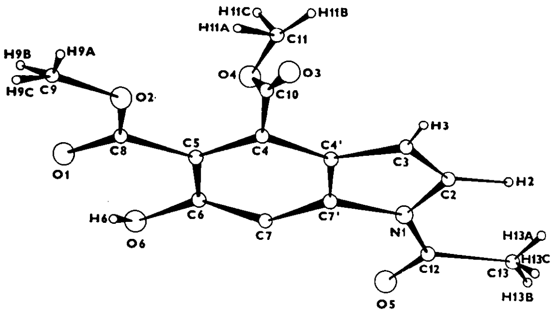


Fig. 1. Structure of the title compound showing the atomic numbering.

The atom coordinates are shown in Table 1.* Bond distances and bond angles for non-H atoms are given in Table 2. The structure is depicted in Fig. 1. Fig. 2 shows the arrangement of the structural units (half only) in the unit cell, van der Waals forces only [minimum H(9*B*)···O(2) intermolecular distance is 2·77 (3) Å].

Related literature. Since it was possible to postulate that the reaction of 4-acetylfuro[3,2-*b*]pyrrole with dimethyl butynedioate proceeded either as a [4+2] cycloaddition or as a [2+2] cycloaddition, the structure of the final product was uncertain (Krutošíková, Dandárová, Alföldi & Kováč, 1988; Dandárová, Krutošikova & Alföldi, 1988). Confirmation of the structure of dimethyl 1-acetyl-6-hydroxyindole-4,5-dicarboxylate enables us to conclude that the reaction proceeds *via* [4+2] cycloaddition (route *A*).

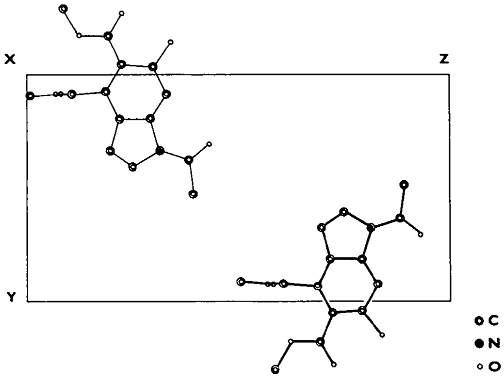
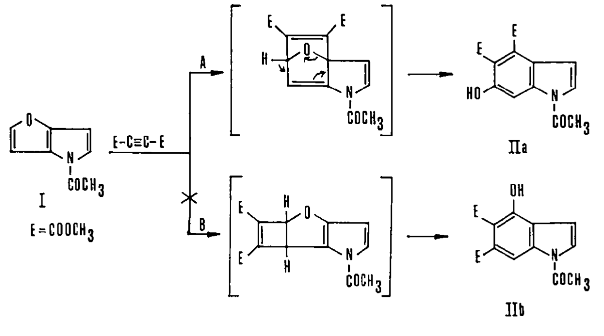


Fig. 2. Projection of the crystal structure along the *x* axis (half of unit cell).



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* Lists of structure amplitudes, anisotropic thermal parameters, H-atom parameters and details of least-squares planes have been deposited with the British Library Document Supply Centre as Supplementary Publication No. SUP 51077 (12 pp.). Copies may be obtained through The Executive Secretary, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.