This plane intersects the *B* ring plane, comprised of atoms C(10), C(1), C(6), C(7) and C(8), at an angle of 72·70°. The latter angle is substantially smaller than that observed in the 1-hydroxy precursor, where the pendant aryl ring was nearly perpendicular (88·0°) to the dihydronaphthalene ring plane. These pronounced structural changes of the title compound in comparison to its 1-hydroxy precursor are most easily explained by the disruption of the strong hydrogen bond between O atoms O(3) and O(5) in the latter.

An analysis of the closest intermolecular contact distances reveals that van der Waals forces are likely to be the dominant stabilizing force in the crystal lattice of the title compound. Several such interactions were noted to occur between neighboring molecules and the shortest of these have been tabulated with their symmetry relationships in Table 2.

In conclusion, several topographical alterations are found between the title compound and its 1-hydroxy precursor as a result of simple methyl substitution for an H atom. The capacity of a podophyllotoxin analogue to bind to tubulin or to trap a DNA–topoisomerase II complex is dramatically affected by what often appear to be minor chemical modifications (Chow *et al.*, 1988; Kohn, 1987; Jardine, 1980; Loike & Horwitz, 1976). Extensive chemical, structural and biological investigations of these and other podophyllotoxin derivatives are now in progress in our laboratories to ascertain the molecular requirements associated with these biological modes of action.

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Structure of Methyl 5-Phenyl-2-propionyl-3-pyrrolecarboxylate

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Abstract. $C_{15}H_{15}NO_3$, $M_r = 257\cdot4$, monoclinic, $P2_1/n$, $a = 10\cdot070$ (2), $b = 5\cdot335$ (3), $c = 24\cdot23$ (1) Å, $\beta = 91\cdot60$ (3)°, $V = 1301$ (2) Å^3, $Z = 4$, D_m (flotation) = $1\cdot312$ (3), $D_x = 1\cdot314$ g cm^{-3}, Cu $K\alpha(\lambda = 1\cdot5418$ Å), $\mu = 6\cdot64$ cm^{-1}, $F(000) = 544$, $T = 295$ K, final $R(F) = 0\cdot037$, $wR = 0\cdot035$ for 850 significant reflections, $I \geq 2\cdot5\sigma(I)$. The phenyl ring and the pyrrole ring are

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planar, and the dihedral angle between them is 18·6°. The nitrogen of the pyrrole ring exhibits N—H tautomerism and forms N—H···O type hydrogen bonds with the screw related carbonyl oxygen. In the unit cell the molecules form dimers across the centres of inversion and infinite spirals across the screw axis.

Introduction. The title compound is an intermediate of a reaction in the synthesis of bispyrroles, which are potential anticancer agents. The title compound is reported to exist in solution in the pyrolenin (azafulvene) form (Iyer, Ratnam, Kulkarni & Sonaseth, 1986). The structure solution has been undertaken to confirm the chemical structure proposed by the chemists.
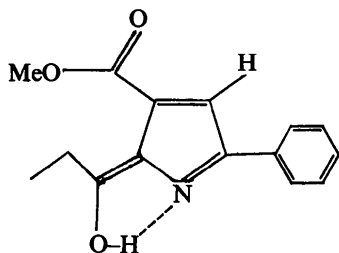
The structure obtained by crystallographic study is
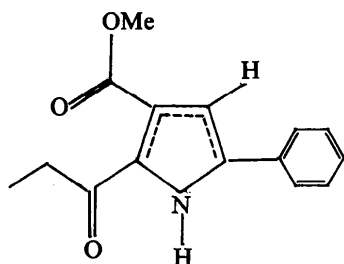
Experimental. Thin needles, transparent crystals, 0·15 × 0·20 × 0·4 mm, from aqueous methanol, three-dimensional intensity data collected on an Enraf–Nonius CAD-4 automated diffractometer in the scan mode $\omega/2\theta$ with $4 \le 2\theta \le 120°$ with graphite-monochromated Cu $K\alpha$ radiation ($\lambda = 1.5418$ Å), data corrected for direct beam polarization and Lorentz effects, not for absorption. Unit-cell parameters from least-squares refinement for 25 reflections in the range $26 \le 2\theta \le 72°$, 1908 unique reflections in the limits $-11 \le h \le 11$, $0 \le k \le 5$, $0 \le l \le 27$, 850 reflections observed with $I \ge 2.5\sigma(I)$. Three standard reflections monitored every 100 reflections showed no significant variation. Structure solution by direct methods using *SHELXS*86 (Sheldrick, 1986), full-matrix least-squares refinement using *SHELX*76 (Sheldrick, 1976), H atom positions from ΔF syntheses, anisotropic refinement for non-H atoms and isotropic for hydrogens to a final $R(F) = 0.037$, wR

$= 0.035$ with individual weighting scheme based on counting statistics $w = 1/[\sigma^2(F) + 0.0555(F_o)^2]$, final cycle of refinement had (shift/e.s.d.)$_{max} = 0.194$, (shift/e.s.d.)$_{mean} = 0.869$ and goodness of fit, $S = 0.39$ for 171 parameters, final electron density map had no peaks greater than 0·15 e Å^{-3}. Scattering factors are as in *SHELX*76.

Discussion. Atomic positions and equivalent temperature factors for non-H atoms are given in Table 1.* Fig. 1 shows a stereoview of the molecule. Fig. 2 shows the bond lengths and bond angles involving non-H atoms. The average e.s.d. in bond lengths and in angles is 0·006 Å and 0·4° respectively. The average distance 1·383 (7) Å around the phenyl ring and the distances around the pyrrole ring are comparable with the standard values. The widening of the angle C(3)—C(12)—O(13) 127·0 (4) and C(3)—C(2)—C(16) 135·7 (3)° reduces the steric interaction between the atoms HC(18)2—O(13) 2·34 (4) Å, which is less than the van der Waals radii sum of 2·61 Å. The important torsion angles are given in Table 2.

The phenyl and the pyrrole rings are planar with $\chi^2 = 3.6$ and 0·0 respectively. The phenyl ring is rotated by 18·6° with respect to the pyrrole ring and the dihedral angle between the carbomethoxy group and the pyrrole ring is 131·32°. The crystal structure is stabilized by N—H···O and C—H···O types of hydrogen bonds. These hydrogen bonds help in forming dimers across the centres of inversion, and infinite spirals about the screw axes. Such dimer formation has been observed in other related compounds *viz* 3-ethyl-6-phenyl-4-carbomethoxy-1*H*-2-pyridone (Nethaji, Pattabhi & Gabe, 1989) and 3,6-diphenyl-4-carboethoxy-1*H*-2-pyridone (Nethaji & Pattabhi, 1989). The oxygen O(17) is involved in bifurcated hydrogen bonds with N(1) and C(11), where one acceptor is hydrogen bonded to two donors and this is commonly observed in urea complexes (Vaughan & Donohue, 1952), in *N,N'*-diphenylurea (Dannecker, Kopf & Rust, 1979), and in symmetric ureas (Deshpande, Meredith & Pasternak, 1968). N(1)···O(17) 2·951 (5), N(1)—HN(1) 0·95 (5) Å and N(1)—HN(1)···O(17) 154 (4)°, and C(11)···O(17) 3·338 (6), C(11)—HC(11) 0·99 (5) Å and C(11)—HC(11)···O(17) 134 (3)° at the symmetry position $\frac{1}{2}-x$, $\frac{1}{2}+y$, $\frac{1}{2}-z$. Dimers are also formed having C(15)—HC(15)···O(13) 3·424 (8),

Table 1. *Fractional positional parameters* (× 10⁴) *and* B_{eq} *values* (Å²) *for* non-H *atoms with e.s.d.'s in parentheses*

$$B_{eq} = 8\pi^2/3[U_{22} + 1/\sin^2\beta(U_{11} + U_{33} + 2U_{13}\cos\beta)].$$

	x	y	z	B_{eq}
N(1)	1840 (3)	1224 (7)	3395 (1)	3·29 (4)
C(2)	2733 (4)	-441 (8)	3622 (1)	3·08 (3)
C(3)	2698 (4)	-118 (8)	4193 (2)	3·32 (4)
C(4)	1773 (4)	1758 (9)	4295 (2)	3·66 (4)
C(5)	1244 (4)	2580 (8)	3797 (2)	3·18 (3)
C(6)	216 (4)	4453 (8)	3678 (2)	3·36 (3)
C(7)	-96 (5)	6188 (9)	4083 (2)	4·21 (4)
C(8)	-1089 (5)	7953 (10)	3986 (2)	5·11 (5)
C(9)	-1785 (5)	7979 (11)	3489 (2)	4·95 (4)
C(10)	-1479 (5)	6287 (11)	3086 (2)	4·70 (3)
C(11)	-496 (5)	4518 (10)	3179 (2)	4·37 (4)
C(12)	3469 (5)	-1460 (9)	4627 (2)	3·82 (4)
O(13)	4589 (3)	-2191 (7)	4603 (1)	5·05 (3)
O(14)	2733 (3)	-1727 (6)	5082 (1)	4·70 (3)
C(15)	3405 (7)	-2933 (15)	5546 (2)	5·81 (5)
C(16)	3422 (4)	-2137 (8)	3245 (2)	3·13 (3)
O(17)	3212 (3)	-1848 (6)	2747 (1)	4·05 (4)
C(18)	4342 (5)	-4122 (10)	3466 (2)	3·60 (4)
C(19)	4888 (6)	-5833 (12)	3026 (2)	4·38 (4)

Table 2. *Important torsion angles* (°)

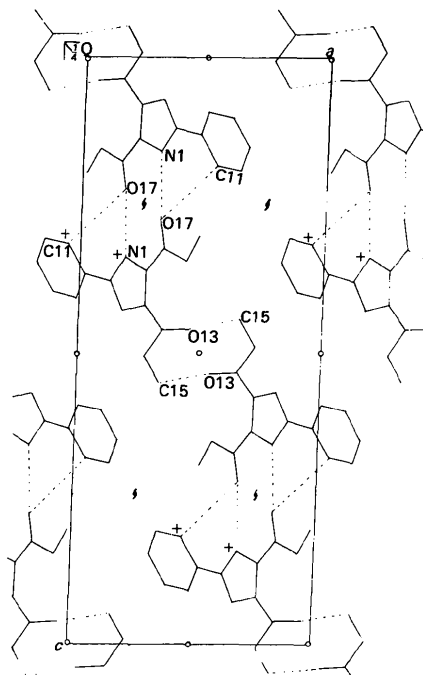| N(1)—C(2)—C(16)—O(17)	4·3 (6)	C(3)—C(2)—C(16)—C(18)	0·5 (7)
N(1)—C(2)—C(16)—C(18)	-175·8 (4)	C(4)—C(3)—C(12)—O(13)	-144·8 (5)
C(2)—C(3)—C(12)—O(13)	35·4 (8)	C(4)—C(3)—C(12)—O(14)	33·7 (6)
C(2)—C(3)—C(12)—O(14)	-146·1 (4)	C(2)—C(16)—C(18)—C(19)	175·6 (4)
C(3)—C(2)—C(16)—O(17)	-179·4 (4)	C(3)—C(12)—O(14)—C(15)	-177·6 (4)
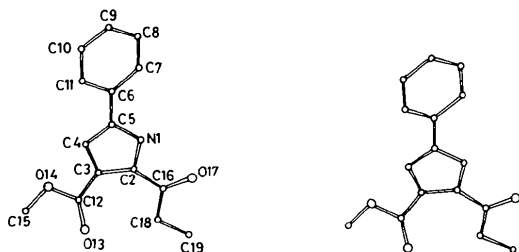
Fig. 1. Stereoview of the molecule with atom numbering.

Fig. 3. Packing of the molecules in the unit cell projected down the *b* axis. Hydrogen bonds are shown as dotted lines.

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

C(15)—HC(15) 1·04 (7) Å and C(15)—HC(15)··· O(13) 138 (5)° at the symmetry position $1 - x$, $-y$, $1 - z$. The packing of the molecules in the unit cell projected down the *b* axis is shown in Fig. 3.

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