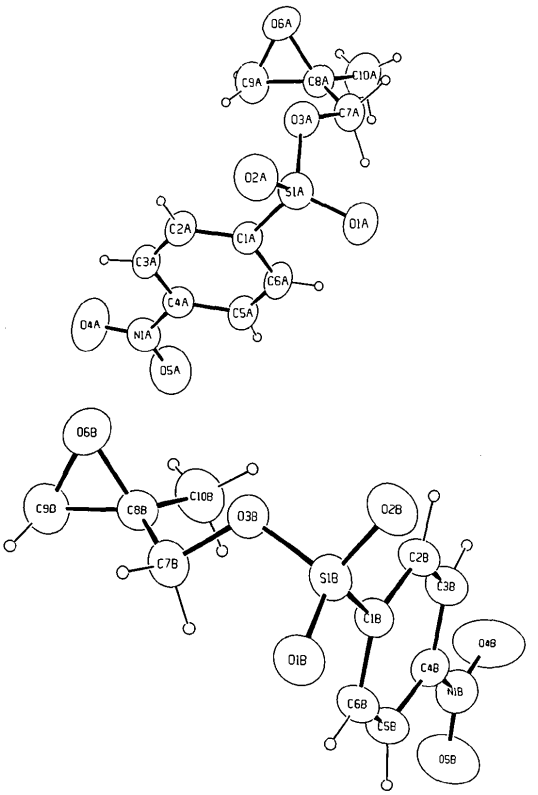

Fig. 1. Numbering scheme and thermal ellipsoids drawn at the 40% probability level for the two independent molecules. H atoms are drawn as circles of arbitrary radius.

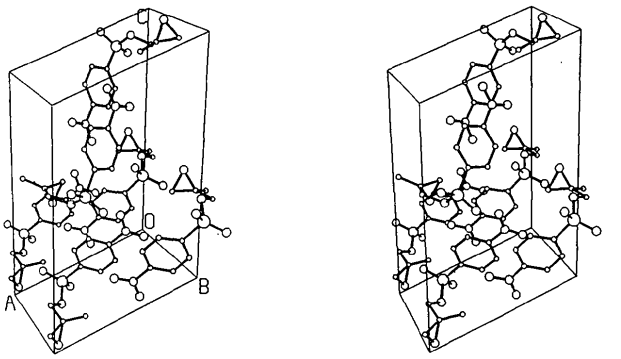

Fig. 2. Stereoview of the unit cell, illustrating the pseudoracemic nature of the packing.

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Structure of 1-Acetylindole-2,3-dione

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Abstract. $C_{10}H_7NO_3$, $M_r = 189{\cdot}17$, monoclinic, $P2_1/n$, $a = 8{\cdot}892\ (2)$, $b = 5{\cdot}108\ (2)$, $c = 18{\cdot}573\ (3)$ Å, $\beta = 98{\cdot}22\ (2)°$, $V = 834{\cdot}8\ (7)$ Å^3, $Z = 4$, $D_x = 1{\cdot}505$ g cm^{-3}, λ(Mo $K\alpha$) $= 0{\cdot}71073$ Å, $\mu = 1{\cdot}06$ cm^{-1}, $F(000) = 392$, $T = 298$ K, $R = 0{\cdot}038$ for 1488 observed reflections. The heterocyclic ring is essentially planar making a dihedral angle of 7·9 (6)° with the acetyl moiety. The C(2)—C(3) distance is 1·538 (2) Å.

Experimental. Crystals of the title compound were obtained from acetic anhydride at 298 K. The data collection and refinement parameters are summarized in Table 1.

The structure was solved using standard direct methods and difference Fourier techniques. In final cycles of full-matrix least-squares refinement all non-H atoms were treated anisotropically, the H atoms were refined isotropically.

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Table 1. *Crystallographic summary*

Data collection[i]	
Crystal dimensions (mm)	0·62 × 0·30 × 0·15
Mode	ω–2θ
Scan rate (° min^{-1})	2·8–10·0
θ range (°)	0–27
Range of *hkl*	$-11 \le h \le 11$, $0 \le k \le 6$, $0 \le l \le 23$
Standard reflections (h^{-1})	1
Variation	No significant variation
Absorption correction	None
Total reflections measured	1751
Unique reflections	1696
R_{int}	0·017
Diffractometer	Enraf–Nonius CAD-4, with graphite monochromator
Structure determination[ii]	
Reflections used [$I > 3\sigma(I)$]	1488
Number of variables	155
R, wR	0·038, 0·040
w	$1/[\sigma^2(F_o) + 0{\cdot}002F_o^2]$
Max. shift/e.s.d.	0·002
Max., min., density in final difference map (e Å^{-3})	0·16, −0·25
S	2·16

Notes: (i) Unit-cell parameters by least-squares refinement of the setting angles of 25 reflections with $10 < \theta < 26°$. No absorption correction. (ii) Function minimized was $\sum w(|F_o| - |F_c|)^2$.

Table 2. *Final atomic coordinates and isotropic temperature factors* (Å^2)

$B_{eq} = (4/3)\sum_i\sum_j \beta_{ij}\mathbf{a}_i.\mathbf{a}_j$.

	x	y	z	B_{eq}
N	0·6834 (1)	0·4010 (2)	0·6231 (1)	2·66 (3)
O(1)	0·7871 (1)	0·0503 (2)	0·6947 (1)	3·62 (3)
O(2)	0·5066 (1)	0·0857 (2)	0·7563 (1)	3·76 (3)
O(3)	0·7876 (1)	0·6536 (2)	0·5436 (1)	4·27 (4)
C(2)	0·6885 (2)	0·2063 (3)	0·6769 (1)	2·69 (4)
C(3)	0·5388 (2)	0·2285 (3)	0·7090 (1)	2·74 (4)
C(4)	0·3133 (2)	0·5411 (3)	0·6758 (1)	3·37 (5)
C(5)	0·2585 (2)	0·7457 (3)	0·6310 (1)	3·72 (5)
C(6)	0·3452 (2)	0·8446 (3)	0·5813 (1)	3·62 (5)
C(7)	0·4883 (2)	0·7467 (3)	0·5746 (1)	3·25 (4)
C(8)	0·5430 (1)	0·5416 (3)	0·6198 (1)	2·56 (4)
C(9)	0·4558 (2)	0·4420 (3)	0·6699 (1)	2·73 (4)
C(10)	0·8001 (2)	0·4616 (3)	0·5817 (1)	2·91 (4)
C(11)	0·9310 (2)	0·2792 (4)	0·5863 (1)	3·70 (5)

Table 3. *Interatomic distances* (Å) *and angles* (°)

N—C(2)	1·406 (2)	N—C(8)	1·434 (2)
N—C(10)	1·411 (2)	O(1)—C(2)	1·196 (2)
O(2)—C(3)	1·207 (2)	O(3)—C(10)	1·205 (2)
C(2)—C(3)	1·538 (2)	C(3)—C(9)	1·452 (2)
C(4)—C(5)	1·381 (2)	C(4)—C(9)	1·383 (2)
C(5)—C(6)	1·380 (2)	C(6)—C(7)	1·389 (2)
C(7)—C(8)	1·386 (2)	C(8)—C(9)	1·390 (2)
C(10)—C(11)	1·484 (2)		
C(2)—N—C(8)	108·9 (1)	C(2)—N—C(10)	126·2 (1)
C(8)—N—C(10)	124·8 (1)	N—C(2)—O(1)	128·0 (1)
N—C(2)—C(3)	106·4 (1)	O(1)—C(2)—C(3)	125·6 (1)
O(2)—C(3)—C(2)	123·1 (1)	O(2)—C(3)—C(9)	131·4 (1)
C(2)—C(3)—C(9)	105·5 (1)	C(5)—C(4)—C(9)	118·5 (1)
C(4)—C(5)—C(6)	119·8 (2)	C(5)—C(6)—C(7)	122·3 (2)
C(6)—C(7)—C(8)	117·7 (1)	N—C(8)—C(7)	129·2 (1)
N—C(8)—C(9)	110·7 (1)	C(7)—C(8)—C(9)	120·1 (1)
C(3)—C(9)—C(4)	129·9 (1)	C(3)—C(9)—C(8)	108·5 (1)
C(4)—C(9)—C(8)	121·6 (1)	N—C(10)—O(3)	119·0 (1)
N—C(10)—C(11)	117·6 (1)	O(3)—C(10)—C(11)	123·4 (1)

Scattering factors for non-H atoms were taken from Cromer & Mann (1968) with corrections for anomalous dispersion taken from Cromer & Liberman (1970), and for H atoms were taken from Stewart, Davidson & Simpson (1965). Programs used: *SHELX*76 (Sheldrick, 1976) and *ORTEP* (Johnson, 1965). Most of the calculations were performed on a VAX 4620 computer at the Instituto de Física e Química de São Carlos.

Atomic coordinates are listed in Table 2,* bond lengths and bond angles are listed in Table 3. Fig. 1 is a projection of the molecule showing the atom numbering and Fig. 2 is a stereoscopic drawing of the unit-cell contents.

Related literature. The heterocyclic ring is essentially planar [σ_{av}, defined as $(\sum_i d_i^2/N-3)^{1/2}$, is 0·013 Å],

* Lists of structure factors, anisotropic thermal parameters and H-atom parameters have been deposited with the British Library Document Supply Centre as Supplementary Publication No. SUP 54340 (12 pp.). Copies may be obtained through The Technical Editor, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England. [CIF reference: LI0085]

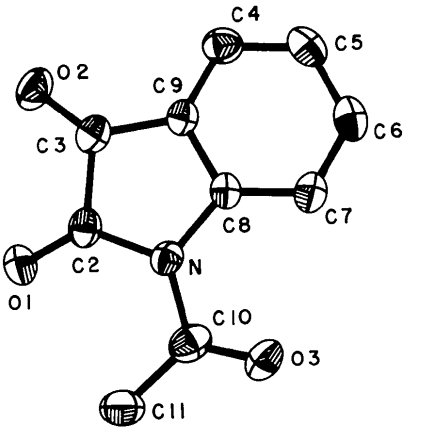

Fig. 1. Perspective view of the molecule showing the atom numbering. Thermal ellipsoids are drawn at the 50% probability level.

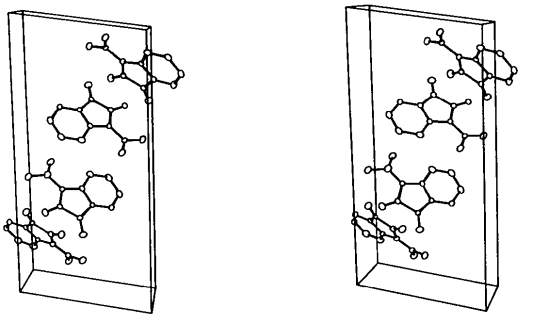

Fig. 2. Stereoview of the unit-cell packing.

making a dihedral angle of 7·9 (6)° with the acetyl moiety. The C(2)—C(3) bond of 1·583 (2) Å is significantly longer than the value of 1·48 Å expected for a C(sp^2)—C(sp^2) single bond. As suggested by Palenik, Koziol, Katritzky & Fan (1990), this lengthening often observed in *cis*-diketones can be ascribed to non-bonded lone pair–lone pair repulsions.

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Structure of a Glycyrrhetinic Acid Derivative

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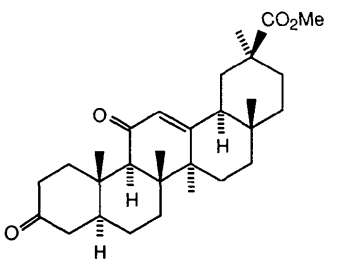
Abstract. Methyl 4,4-desmethyl-3,11-dioxo-18α-olean-12-en-30-oate, $C_{29}H_{42}O_4$, $M_r = 454.65$, orthorhombic, $P2_12_12_1$, $a = 12.521$ (1), $b = 29.271$ (3), $c = 6.779$ (1) Å, $V = 2484.3$ (3) Å^3, $Z = 4$, $D_x = 1.215$ Mg m^{-3}, λ(Cu $K\alpha$) = 1.54178 Å, $\mu = 0.63$ mm^{-1}, $F(000) = 992$, $T = 295$ K, $R = 0.044$ for 2390 observed reflections. The ring junctions *A*/*B*, *B*/*C* and *D*/*E* are all *trans*. The conformations of rings *A*, *B*, *D* and *E* are all chair.

Experimental. Colorless plates (I) obtained from ethyl acetate. Crystal of dimensions 0.4 × 0.4 × 0.1 mm. Rigaku AFC-5R diffractometer, graphite-monochromatized Cu $K\alpha$ radiation. Cell dimensions determined from 2θ angles for 25 reflections in the range $30 < 2\theta < 45°$. Intensities measured up to $\theta = 70°$ in h 0/15, k 0/34 and l −8/0, ω–2θ scans, ω-scan width $(1 + 0.2\tan\theta)°$, three standard reflections monitored every 100 measurements showed no significant change. 2580 unique reflections measured, 2390 intensities observed [$F_o > 3\sigma(F_o)$] and two very strong reflections rejected], no absorption corrections.



(I)

Structure solved by *MULTAN*87 (Debaerdemaeker, Germain, Main, Tate & Woolfson, 1987). H atoms located on a difference density map. Positional and thermal parameters refined by block-diagonal least squares, isotropic for H and anisotropic for other atoms, 467 parameters. $\sum(w|\Delta F|^2)$ minimized, $w = 1/[\sigma^2(F_o) + 0.0022|F_o|^2]$, $w = 0$ for 59 reflections with $w^{1/2}|\Delta F| > 3$. The final $R = 0.044$, $wR = 0.057$, $S = 1.1171$. The maximum Δ/σ in the final cycle was 0.2. The highest and lowest peaks in the final difference density map were 0.6 and −0.3 e Å^{-3}. Atomic scattering factors calculated by $\sum[a_i\exp(-b_i\lambda^{-2}\sin^2\theta)] + c$ ($i = 1,...,4$) (*International Tables for X-ray Crystallography*, 1974, Vol. IV). Calculations performed on a VAX station 3100 computer. The final atomic coordinates and equivalent isotropic temperature factors are given in Table 1.* A perspective view of the molecule with the atom labeling is presented in Fig. 1.

Related literature. Structure–activity relationships of the title compound have been referred to by Terasawa, Okada, Hara & Itoh (1991).

The author thanks Drs Terasawa and Okada for supplying the crystals.

* Lists of H-atom coordinates, anisotropic temperature factors, bond lengths, bond angles and structure factors have been deposited with the British Library Document Supply Centre as Supplementary Publication No. SUP 54608 (20 pp.). Copies may be obtained through The Technical Editor, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England. [CIF reference: AS0539]

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