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Key indicators

Single-crystal X-ray study
 $T = 293\text{ K}$
Mean $\sigma(\text{C}-\text{C}) = 0.006\text{ \AA}$
 R factor = 0.061
 wR factor = 0.207
Data-to-parameter ratio = 14.1For details of how these key indicators were
automatically derived from the article, see
<http://journals.iucr.org/e>.1-*tert*-Butyl-3-(α -hydroxy-4-isopropylbenzyl-
idene)pyrrolidine-2,4-dioneThe title compound, $\text{C}_{18}\text{H}_{23}\text{NO}_3$, is a potent new herbicide containing the pyrrolidine-2,4-dione ring system. In the crystalline state, the molecular skeleton contains a hydrogen-bonded enol group, formed by benzoyl tautomerization.

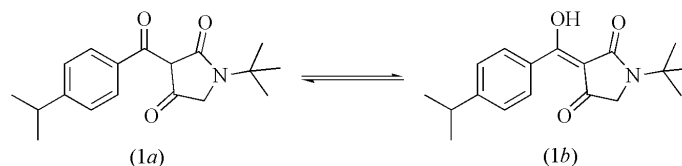
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Comment

Many compounds containing the 3-acylpyrrolidine-2,4-dione moiety are novel heterocyclic compounds with antibiotic activity, such as tenuazonic (Stickings, 1959), streptolydigin (Rinehart *et al.*, 1963), tirandamycin (Rinehart *et al.*, 1971), malonomycin (Bann *et al.*, 1978), α -cyclopiazonic acid (Stickings, 1959; van Rooyen, 1992) and β -cyclopiazonic acid (Holzapfel *et al.*, 1970). All these compounds possess a 3-acyltetramic acid moiety as a tricarbonylmethane structure and the hydrogen chemical shift of the enol hydroxy group is about 11 p.p.m. (Wu *et al.*, 2002). More importantly, most of the excellent inhibitors of *p*-hydroxyphenylpyruvate dioxygenase also possess similar characteristics, which are crucial for their two kinds of bioactivity (Zhu *et al.*, 2004). In this paper, we report the crystal structure of the title compound, (1*b*).



The molecular structure of (1*b*) is shown in Fig. 1. The analysis of crystals grown from a solution of 3-(4-isopropylbenzoyl)-1-*tert*-butylpyrrolidine-2,4-dione, (1*a*), showed that we had obtained crystals of the related tautomeric form, *viz.* 1-*tert*-butyl-3-(α -hydroxybenzylidene)pyrrolidine-2,4-dione, (1*b*). Atom H1, involved in intramolecular hydrogen bonding between O1 and O2, was assigned to O1 rather than to O2, based on bond lengths. The C4–O2 distance is

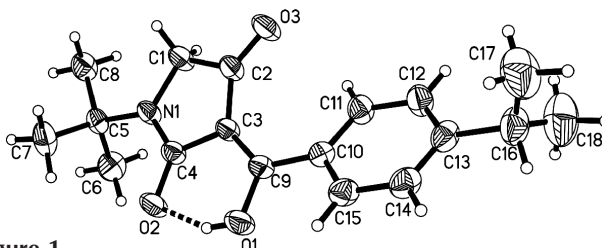


Figure 1

A view of the title compound, with displacement ellipsoids drawn at the 30% probability level. The intramolecular hydrogen bond is shown as a dashed line.

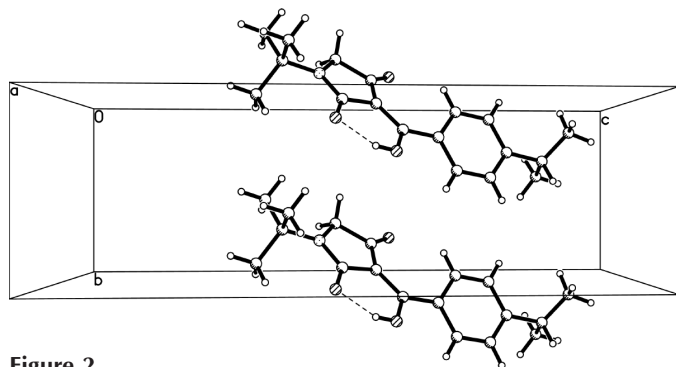


Figure 2
A partial packing diagram of the title compound

1.262 (4) Å. The bond between atoms C2 and O3 is a double bond (C2=O3) of 1.221 (4) Å. In contrast, the C9–O1 distance [1.311 (4) Å] is intermediate between the normal carbonyl C=O bond and the C–O single-bond length (Allen *et al.*, 1987). A similar situation has been reported for the related compounds 3-(1-hydroxyethylidene)-1-phenylpyrrolidine-2,4-dione (Ellis & Spek, 2001), 1-benzyl-3-(α -hydroxybenzylidene)pyrrolidine-2,4-dione (Zhu, Song, Li *et al.*, 2004) and 3-(α -hydroxy-2-methoxybenzylidene)-1-isopropylpyrrolidine-2,4-dione (Zhu, Song, Yao *et al.*, 2004), which contain the same pyrrolidine skeleton. In addition, the X-ray data indicate no hydrogen-bonding interaction between adjacent molecules (Fig. 2), while the compounds mentioned above involve weak intermolecular C–H \cdots O hydrogen bonds.

Experimental

The title compound, (1b), was prepared according to the reported method (Matsuo *et al.*, 1980) and was crystallized from a mixture of petroleum ether and ethyl acetate (*v/v* 3:1).

Crystal data

$C_{18}H_{23}NO_3$	$D_x = 1.198 \text{ Mg m}^{-3}$
$M_r = 301.37$	Mo $K\alpha$ radiation
Monoclinic, $P2_1/n$	Cell parameters from 1698 reflections
$a = 14.896 (3) \text{ \AA}$	$\theta = 2.8\text{--}22.2^\circ$
$b = 5.9680 (11) \text{ \AA}$	$\mu = 0.08 \text{ mm}^{-1}$
$c = 18.894 (4) \text{ \AA}$	$T = 293 (2) \text{ K}$
$\beta = 95.752 (4)^\circ$	Prism, colorless
$V = 1671.2 (6) \text{ \AA}^3$	$0.28 \times 0.16 \times 0.16 \text{ mm}$
$Z = 4$	

Data collection

Bruker SMART CCD area-detector diffractometer	1619 reflections with $I > 2\sigma(I)$
φ and ω scans	$R_{\text{int}} = 0.045$
Absorption correction: none	$\theta_{\text{max}} = 25.0^\circ$
8391 measured reflections	$h = -17 \rightarrow 17$
2938 independent reflections	$k = -5 \rightarrow 7$
	$l = -22 \rightarrow 19$

Refinement

Refinement on F^2	$w = 1/[\sigma^2(F_o^2) + (0.0941P)^2 + 0.9716P]$
$R[F^2 > 2\sigma(F^2)] = 0.061$	where $P = (F_o^2 + 2F_c^2)/3$
$wR(F^2) = 0.207$	$(\Delta/\sigma)_{\text{max}} = 0.004$
$S = 1.02$	$\Delta\rho_{\text{max}} = 0.39 \text{ e \AA}^{-3}$
2938 reflections	$\Delta\rho_{\text{min}} = -0.26 \text{ e \AA}^{-3}$
208 parameters	Extinction correction: <i>SHELXL97</i>
H atoms treated by a mixture of independent and constrained refinement	Extinction coefficient: 0.017 (3)

Table 1

Selected geometric parameters (Å, °).

O1–C9	1.311 (4)	N1–C5	1.487 (4)
O2–C4	1.262 (4)	C1–C2	1.514 (5)
O3–C2	1.221 (4)	C2–C3	1.438 (5)
N1–C4	1.335 (4)	C3–C9	1.388 (5)
N1–C1	1.452 (4)	C3–C4	1.458 (5)
C4–N1–C1	110.2 (3)	O2–C4–C3	123.7 (3)
C4–N1–C5	126.0 (3)	N1–C4–C3	111.5 (3)
C1–N1–C5	123.8 (3)	N1–C5–C8	109.3 (3)
N1–C1–C2	105.1 (3)	N1–C5–C7	109.7 (3)
O3–C2–C3	131.6 (3)	C8–C5–C7	108.9 (3)
O3–C2–C1	121.6 (3)	N1–C5–C6	108.2 (3)
C3–C2–C1	106.8 (3)	C8–C5–C6	109.9 (3)
C9–C3–C2	134.3 (3)	C7–C5–C6	110.9 (3)
C9–C3–C4	119.2 (3)	O1–C9–C3	117.8 (3)
C2–C3–C4	106.3 (3)	O1–C9–C10	113.6 (3)
O2–C4–N1	124.9 (3)	C3–C9–C10	128.5 (3)
C5–N1–C1–C2	178.5 (3)	C2–C3–C9–C10	−10.2 (7)
N1–C1–C2–O3	−174.4 (4)	C4–C3–C9–C10	176.6 (3)
O3–C2–C3–C4	173.0 (4)	O1–C9–C10–C11	158.2 (4)
C5–N1–C4–C3	178.7 (3)	C3–C9–C10–C11	−20.6 (6)
C2–C3–C9–O1	171.0 (4)	O1–C9–C10–C15	−18.4 (5)
C4–C3–C9–O1	−2.2 (5)	C3–C9–C10–C15	162.7 (4)

Table 2

Hydrogen-bonding geometry (Å, °).

$D\cdots H\cdots A$	$D\cdots H$	$H\cdots A$	$D\cdots A$	$D\cdots H\cdots A$
O1–H1 \cdots O2	0.83 (3)	1.71 (3)	2.468 (4)	152 (5)
C11–H11 \cdots O3	0.93	2.25	2.958 (5)	132
C7–H7A \cdots O2	0.96	2.47	3.048 (5)	118

All H atoms were placed in calculated positions, with C–H = 0.93–0.97 Å and O–H = 0.82 Å, and were included in the final cycles of refinement using a riding model, with $U_{\text{iso}}(\text{H}) = 1.2U_{\text{eq}}(\text{C})$ or $1.5U_{\text{eq}}(\text{O})$.

Data collection: *SMART* (Bruker, 1999); cell refinement: *SAINT* (Bruker, 1999); data reduction: *SAINT*; program(s) used to solve structure: *SHELXS97* (Sheldrick, 1997); program(s) used to refine structure: *SHELXL97* (Sheldrick, 1997); molecular graphics: *SHELXTL* (Bruker, 1999); software used to prepare material for publication: *SHELXTL*.

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