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# 1-Benzyl-3-(α-hydroxybenzylidene)-pyrrolidine-2,4-dione

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**Key indicators**

Single-crystal X-ray study
*T* = 293 K
Mean σ(C–C) = 0.006 Å
*R* factor = 0.059
*wR* factor = 0.131
Data-to-parameter ratio = 9.1

For details of how these key indicators were automatically derived from the article, see http://journals.iucr.org/e.

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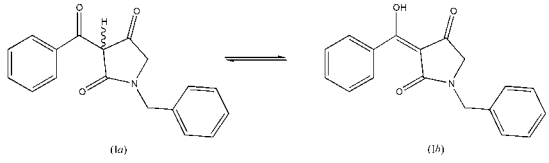
The title compound, $C_{18}H_{15}NO_3$, is a potent new herbicide containing the pyrrolidine-2,4-dione ring system. In the crystalline state, the molecular skeleton contains one enol hydrogen-bonded moiety, formed from benzoyl C=O isomerization.

## Comment

Many compounds containing the 3-acylpyrrolidine-2,4-dione moiety are novel heterocyclic compounds with antibiotic activity, such as tenuazonic (Sticking, 1959), streptolydigin (Rinehart *et al.*, 1963), tirandamycin (Mackellar *et al.*, 1971), malonomycin (Bann *et al.*, 1978), α-cyclopiazonic acid (Sticking, 1959) and β-cyclopiazonic acid (Holzapfel *et al.*, 1970). All these compounds possess a 3-acyltetramic acid moiety as a tricarbonylmethane structure and their hydrogen chemical shift of the enol hydroxy is about 11 p.p.m. (Wu *et al.*, 2002). On the other hand, 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). Up to now, we have synthesized a series of 3-(un)substituted benzoyl-1-benzylpyrrolidine-2,4-dione compounds and some of them have high herbicidal activity. The structure reported here, (I*b*), helps us to investigate the relationship between structure and herbicidal activity. To the best of our knowledge, this is the first reported crystal structure determination of a molecule with a 3-benzoylpyrrolidine-2,4-dione ring system.



The molecular structure of (I*b*) is shown in Fig. 1. The analysis of crystals grown from a solution of 3-benzoyl-1-benzylpyrrolidine-2,4-dione, (I*a*), showed that we had obtained crystals of the related tautomeric form 1-benzyl-3-(α-hydroxybenzylidene)pyrrolidine-2,4-dione, (I*b*). Atom H1,

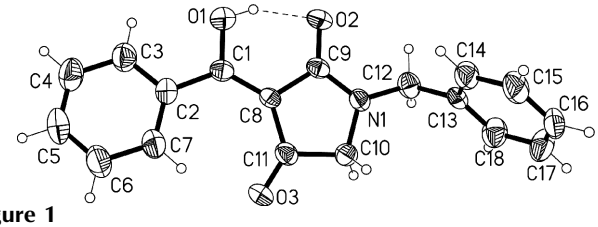



**Figure 1**
View of the title compound, drawn with 40% probability ellipsoids.

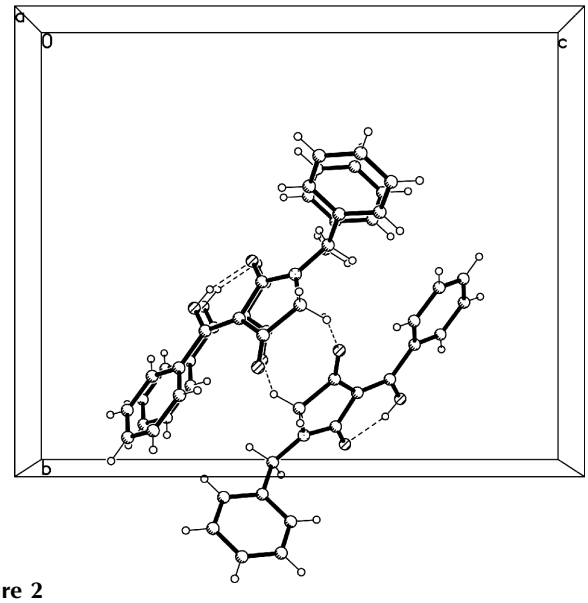



**Figure 2**
Packing diagram showing the intra- and intermolecular hydrogen bonds.

involved in intramolecular hydrogen bonding between O1 and O2, was assigned to O1 rather than to O2, based on bond lengths. The C9—O2 distance is 1.268 (4) Å, which is longer than the normal carbonyl bond length (C11—O3) of 1.219 (5) Å. In contrast, the C1—O1 distance [1.326 (4) Å] is intermediate between the normal carbonyl bond and the C—O single bond length (Ibers & Hamilton, 1974). A similar situation has been found in 3-(1-hydroxyethylidene)-1-phenylpyrrolidine-2,4-dione, which contains the same pyrrolidine skeleton (Ellis & Spek, 2001). In addition, the X-ray data also indicates a weak hydrogen-bonding interaction between two adjacent molecules (see Fig. 2), with an O···C distance of 3.109 (6) Å.

## Experimental

The title compound was obtained according to the similar reported procedure of Matsuo *et al.* (1980). Colorless single crystals were obtained by recrystallization of 3-benzoyl-1-benzylpyrrolidine-2,4-dione from petroleum ether and ethyl acetate.

*Crystal data*

$C_{18}H_{15}NO_3$
$M_r$ = 293.31
Orthorhombic, $P2_12_12_1$
$a$ = 5.569 (3) Å
$b$ = 15.062 (7) Å
$c$ = 18.245 (9) Å
$V$ = 1530.3 (12) Å$^3$
$Z$ = 4
$D_x$ = 1.273 Mg m$^{-3}$
Mo $K\alpha$ radiation
Cell parameters from 765 reflections
$\theta$ = 2.6–19.6°
$\mu$ = 0.09 mm$^{-1}$
$T$ = 293 (2) K
Prism, colourless
0.20 × 0.18 × 0.16 mm

*Data collection*

Bruker SMART CCD area-detector diffractometer
$\varphi$ and $\omega$ scans
Absorption correction: none
8033 measured reflections
1830 independent reflections
1101 reflections with $I > 2\sigma(I)$
$R_{int}$ = 0.060
$\theta_{max}$ = 26.4°
$h = -6 \rightarrow 5$
$k = -14 \rightarrow 18$
$l = -22 \rightarrow 21$

*Refinement*

Refinement on $F^2$
$R[F^2 > 2\sigma(F^2)]$ = 0.059
$wR(F^2)$ = 0.131
$S$ = 1.07
1830 reflections
200 parameters
H-atom parameters constrained
$w = 1/[\sigma^2(F_o^2) + (0.0612P)^2]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} < 0.001$
$\Delta\rho_{max}$ = 0.17 e Å$^{-3}$
$\Delta\rho_{min}$ = −0.12 e Å$^{-3}$

**Table 1**
Selected geometric parameters (Å, °).

| | | | |
|---|---|---|---|
| N1—C9 | 1.345 (4) | C2—C3 | 1.388 (5) |
| N1—C10 | 1.454 (5) | C2—C7 | 1.392 (6) |
| N1—C12 | 1.475 (4) | C8—C11 | 1.469 (5) |
| O1—C1 | 1.326 (4) | C8—C9 | 1.479 (5) |
| O2—C9 | 1.268 (4) | C10—C11 | 1.535 (5) |
| O3—C11 | 1.219 (5) | C12—C13 | 1.514 (6) |
| C1—C8 | 1.399 (5) | C13—C14 | 1.379 (6) |
| C1—C2 | 1.487 (5) | | |
| C9—N1—C10 | 111.9 (3) | C1—C8—C9 | 118.5 (3) |
| C9—N1—C12 | 124.7 (3) | C11—C8—C9 | 105.8 (3) |
| C10—N1—C12 | 122.2 (3) | O2—C9—N1 | 124.1 (4) |
| C1—O1—H1 | 109.5 | O2—C9—C8 | 125.0 (3) |
| O1—C1—C8 | 118.0 (3) | N1—C9—C8 | 110.8 (3) |
| O1—C1—C2 | 112.2 (3) | N1—C10—C11 | 104.4 (3) |
| C8—C1—C2 | 129.8 (4) | N1—C10—H10*A* | 110.9 |
| C3—C2—C7 | 117.3 (4) | O3—C11—C8 | 131.3 (4) |
| C1—C8—C11 | 135.5 (4) | O3—C11—C10 | 121.8 (4) |
| O1—C1—C2—C3 | 8.6 (5) | C1—C8—C9—O2 | 1.2 (5) |
| C8—C1—C2—C3 | −169.4 (4) | C11—C8—C9—O2 | 177.0 (3) |
| O1—C1—C2—C7 | −169.8 (4) | C1—C8—C9—N1 | −177.9 (3) |
| C8—C1—C2—C7 | 12.1 (6) | C11—C8—C9—N1 | −2.1 (4) |
| C1—C2—C3—C4 | 179.7 (4) | C9—N1—C12—C13 | 129.4 (4) |
| C10—N1—C9—O2 | −178.4 (3) | C10—N1—C12—C13 | −63.9 (4) |
| C12—N1—C9—O2 | −10.5 (5) | N1—C12—C13—C14 | −60.2 (5) |
| C10—N1—C9—C8 | 0.6 (4) | N1—C12—C13—C18 | 118.9 (4) |
| C12—N1—C9—C8 | 168.5 (3) | | |

**Table 2**
Hydrogen-bonding geometry (Å, °).

| *D*—H···*A* | *D*—H | H···*A* | *D*···*A* | *D*—H···*A* |
|---|---|---|---|---|
| C10—H10*B*···O3$^{i}$ | 0.97 | 2.46 | 3.019 (5) | 116 |
| O1—H1···O2 | 0.82 | 1.74 | 2.507 (4) | 155 |
| C3—H3···O1 | 0.93 | 2.32 | 2.669 (6) | 102 |
| C7—H7···O3 | 0.93 | 2.14 | 2.969 (6) | 148 |
| C12—H12*A*···O2 | 0.97 | 2.52 | 2.908 (5) | 104 |

Symmetry code: (i) $x - \frac{1}{2}, \frac{3}{2} - y, 1 - z$.

All H atoms were placed in calculated positions, with C–H = 0.93 or 0.97 Å and O—H = 0.82 Å, and included in the final cycles of refinement using a riding model, with $U_{iso}$(H) = 1.2$U_{eq}$(C) or 1.5$U_{eq}$(O). Friedel pairs were not merged.

Data collection: *SMART* (Bruker, 1999); cell refinement: *SMART*; data reduction: *SAINT* (Bruker, 1999); program(s) used to solve structure: *SHELXS*97 (Sheldrick, 1997); program(s) used to refine structure: *SHELXL*97 (Sheldrick, 1997); molecular graphics: *SHELXTL* (Bruker, 1999); software used to prepare material for publication: *SHELXTL*.

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