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(Z)-3-[Hydroxy(2,4-dimethoxyphenyl)methylene]-1-isopropylpyrrolidine-2,4-dione

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Key indicators

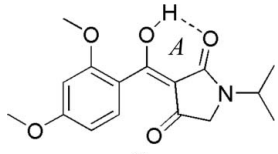
Single-crystal X-ray study
T = 294 K
Mean σ(C–C) = 0.003 Å
R factor = 0.041
wR factor = 0.112
Data-to-parameter ratio = 15.0

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

The title compound, $C_{16}H_{19}NO_5$, is a potent new herbicide containing the pyrrolidine-2,4-dione ring system. In the crystalline state, the molecular skeleton contains one enol grouping, which is intramolecularly hydrogen bonded to a neighbouring keto O atom. The dihedral angle between the six-membered ring formed by the enol group and the benzene ring is 62.02 (6)°.

Comment

Many compounds containing the 3-acylpyrrolidine-2,4-dione system are novel heterocyclic compounds with antibiotic activity; these include tenuazonic acid (Stickings, 1959), streptolydigin (Rinehart *et al.*, 1963), tirandamycin (Rinehart *et al.*, 1971), malonomycin (Van Der Baan *et al.*, 1978), α-cyclopiazonic acid (Stickings, 1959; van Rooyen *et al.*, 1992) and β-cyclopiazonic acid (Holzapfel *et al.*, 1970). All these compounds possess a 3-acyltetramic acid grouping as a tricarbonylmethane fragment, and the ^{1}H chemical shift of the enol hydroxy group is about 11 p.p.m. (Wu *et al.*, 2002). Most of the excellent inhibitors of *p*-hydroxyphenylpyruvate dioxygenase also possess similar characteristics, which are crucial for their two kinds of bioactivity (Zhu Hu & Yang, 2004). Previously, we have synthesized a series of 3-(un)substituted benzoyl-1-alkylpyrrolidine-2,4-dione compounds and found that the title compound, (I), possesses high herbicidal activity. The structure of (I) reported here helps us to investigate the relationship between structure and herbicidal activity.



(I)

The molecular structure of (I) is shown in Fig. 1. Atom H3*A*, involved in intramolecular hydrogen bonding between atoms O2 and O3, was assigned to O3 rather than to O2, on the basis of the bond lengths. The C13—O2 distance is 1.256 (2) Å, which is longer than the normal carbonyl bond length (C13═O1) of 1.216 (2) Å. In contrast, the C9—O3 distance [1.320 (2) Å] is intermediate between a normal carbonyl C═O double bond and a C—O single-bond length (Allen *et al.*, 1987). A similar situation was reported for 3-(1-hydroxyethylidene)-1-phenylpyrrolidine-2,4-dione (Ellis & Spek, 2001), 1-benzyl-3-(α-hydroxybenzylidene)pyrrolidine-2,4-dione, (II) (Zhu, Song, Li *et al.*, 2004), 1-*tert*-butyl-3-(α-hydroxy-4-isopropylbenzylidene)pyrrolidine-2,4-dione, (III)

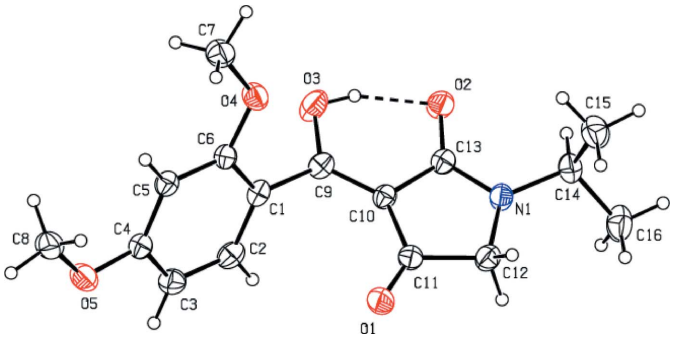


Figure 1
A view of compound (I), showing the atom-numbering scheme. Displacement ellipsoids are drawn at the 30% probability level. The intramolecular O—H···O hydrogen bond is indicated by a dashed line.

(Xu, 2005), and 3-(α-hydroxyl-2-methoxylbenzylidene)-1-isopropylpyrrolidine-2,4-dione, (IV) (Zhu, Song, Yao *et al.*, 2004). The dihedral angle formed by the enol ring *A* with the benzene ring is 62.02 (6)°, which is larger than the dihedral angles for (II), (III) and (IV) (10, 21 and 53°, respectively).

The crystal structure of (I) also involves one weak intramolecular and three intermolecular C—H···O hydrogen-bonding interactions (Fig. 2 and Table 2).

Experimental

Compound (I) was obtained according to the procedure reported by Matsuo *et al.* (1980). Colourless single crystals of (I) were obtained by recrystallization of 1-isopropyl-3-(α-hydroxy-2,4-dimethoxylbenzylidene)pyrrolidine-2,4-dione from petroleum ether and ethyl acetate (1:3).

Crystal data

$C_{16}H_{19}NO_5$
M_r = 305.32
Monoclinic, $C2/c$
a = 21.288 (4) Å
b = 12.132 (2) Å
c = 13.767 (3) Å
β = 121.790 (3)°
V = 3022.0 (10) Å^3
Z = 8
D_x = 1.342 Mg m^{-3}
Mo $K\alpha$ radiation
μ = 0.10 mm^{-1}
T = 294 (2) K
Prism, colourless
0.34 × 0.24 × 0.16 mm

Data collection

Bruker SMART CCD area-detector diffractometer
φ and ω scans
Absorption correction: multi-scan (*SADABS*; Sheldrick, 1996)
T_{min} = 0.972, T_{max} = 0.984
8407 measured reflections
3120 independent reflections
1894 reflections with $I > 2\sigma(I)$
R_{int} = 0.040
θ_{max} = 26.5°

Refinement

Refinement on F^2
$R[F^2 > 2\sigma(F^2)]$ = 0.041
$wR(F^2)$ = 0.112
S = 1.00
3120 reflections
208 parameters
H atoms treated by a mixture of independent and constrained refinement
$w = 1/[\sigma^2(F_o^2) + (0.0453P)^2 + 1.206P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max}$ = 0.003
$\Delta\rho_{max}$ = 0.18 e Å^{-3}
$\Delta\rho_{min}$ = −0.14 e Å^{-3}
Extinction correction: *SHELXL97* (Sheldrick, 1997)
Extinction coefficient: 0.0025 (3)

Table 1
Selected bond lengths (Å).

O1—C11	1.216 (2)	C9—C10	1.373 (2)
O2—C13	1.256 (2)	C10—C13	1.452 (2)
O3—C9	1.320 (2)		

Table 2
Hydrogen-bond geometry (Å, °).

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
O3—H3*A*···O2	0.862 (10)	1.706 (13)	2.531 (2)	159 (3)
C14—H14···O2	0.98	2.52	2.873 (2)	101
C2—H2···O1	0.93	2.47	3.3286	153
C8—H8*B*···O2	0.96	2.58	3.4585	152
C8—H8*C*···O3	0.96	2.42	3.3767	172

The hydroxyl H atom (O3)H3*A* was found in a difference Fourier map and refined freely. The other H atoms were placed in calculated positions, with C—H = 0.93–0.97 Å, and included in the final cycles of refinement using a riding model, with U_{iso}(H) = 1.2U_{eq}(C).

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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