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2-(2,4-Dihydroxyphenyl)-*N*,*N*-dimethyl-2-oxoacetamide

Wei Huang, Peiliang Zhao, Zhongzhen Zhou, Qiong Chen, Deyou Teng and Guangfu Yang*

Key Laboratory of Pesticide and Chemical Biology of the Ministry of Education, College of Chemistry, Central China Normal University, Wuhan 430079, China

Correspondence e-mail:
gfyang@mail.ccnu.edu.cn

Key indicators

Single-crystal X-ray study
T = 283 K
Mean σ(C–C) = 0.002 Å
R factor = 0.049
wR factor = 0.148
Data-to-parameter ratio = 16.8

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

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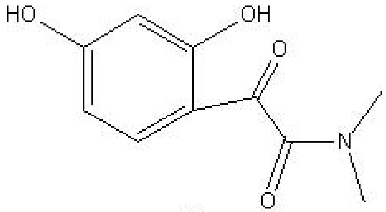
In the crystal structure of the title compound, $C_{10}H_{11}NO_4$, O—H···O inter- and intramolecular hydrogen bonds are present, as well as intermolecular C—H···O interactions.

Comment

α-Ketoamides have potential applications for the synthesis of various useful products, including α-amino acids, α-hydroxy-acids and heterocyclic compounds (Zhou & Chen, 2001). Furthermore, α-ketoamide derivatives have been reported to exhibit a range of biological properties, including anti-microbial and antipsychotic activity (Yang *et al.*, 2002). For these reasons, we are interested in exploring and developing novel strategies for the synthesis of α-ketoamides.



(I)

In this paper, we present the X-ray crystallographic analysis of the title compound, (I). The bond lengths and angles are unremarkable. The dihedral angle between the plane through C9/C10/N1/C8/O4/C7 and the plane of the benzene ring is 89.5 (2)° (Fig. 1). As shown in Fig. 2, the molecules are linked by both intra- and intermolecular hydrogen bonds. Geometric details of these are listed in Table 1. No π–π stacking interactions are observed in the crystal structure.

Experimental

2,4-Dihydroxyacetophenone (10 mmol) and dimethyl oxalate (1.28 mmol) were dissolved in dimethylformamide (5 ml). This was

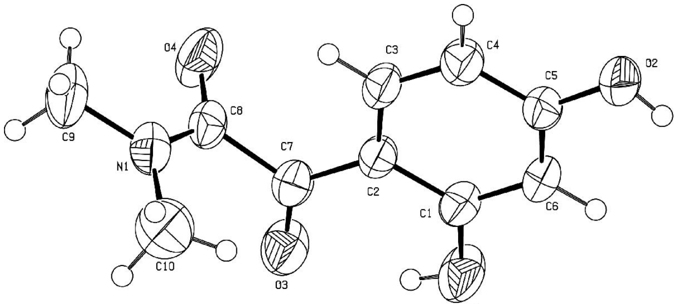


Figure 1
A view of the molecule of (I), showing the atom-labelling scheme. Displacement ellipsoids are drawn at the 50% probability level and H atoms are represented by circles of arbitrary size.

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added to a solution of sodium (0.8 g) in methanol (20 ml) and the mixture was stirred at room temperature for 48 h. Acetic acid (36 ml) was then added and the mixture was heated to 373 K for 5 h. The resulting mixture was purified by column chromatography on silica gel using acetone/diethyl ether (9:1 *v*/*v*) as eluent to afford (I) (yield 40%, m.p. 464 K). ^{1}H NMR (CDCl$_3$, 400 MHz): 11.10 (*s*, 1H, 2′OH), 10.79 (*s*, 1H, 4′OH), 7.45 (*d*, 1H, 6′-H), 6.43 (*d*, 1H, 5′-H), 6.32 (*d*, 1H, 3′-H), 2.93 (*s*, 3H, N-CH$_3$), 2.85 (*s*, 3H, N-CH$_3$); MS (EI 70 eV) *m*/*z* (%): 209 (4), 191 (4), 136 (66), 107 (28), 70 (100), 52 (84). Crystals suitable for an X-ray diffraction study were grown from acetone at 277 K.

Crystal data

$C_{10}H_{11}NO_4$
$M_r = 209.20$
Monoclinic, $P2_1/c$
$a = 9.3158$ (13) Å
$b = 6.4393$ (9) Å
$c = 17.399$ (2) Å
$\beta = 92.022$ (2)°
$V = 1043.1$ (2) Å^3
$Z = 4$
$D_x = 1.332$ Mg m^{-3}
Mo $K\alpha$ radiation
Cell parameters from 1822 reflections
$\theta = 2.5$–26.5°
$\mu = 0.10$ mm^{-1}
$T = 283$ (2) K
Block, yellow
0.40 × 0.20 × 0.10 mm

Data collection

Bruker SMART 4K CCD area-detector diffractometer
φ and ω scans
Absorption correction: none
5991 measured reflections
2373 independent reflections
1740 reflections with $I > 2\sigma(I)$
$R_{\rm int} = 0.025$
$\theta_{\rm max} = 27.5°$
$h = -12 \rightarrow 12$
$k = -8 \rightarrow 7$
$l = -22 \rightarrow 17$

Refinement

Refinement on F^2
$R[F^2 > 2\sigma(F^2)] = 0.049$
$wR(F^2) = 0.148$
$S = 1.05$
2373 reflections
141 parameters
H-atom parameters constrained
$w = 1/[\sigma^2(F_o^2) + (0.0777P)^2 + 0.1291P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{\rm max} = 0.001$
$\Delta\rho_{\rm max} = 0.22$ e Å^{-3}
$\Delta\rho_{\rm min} = -0.17$ e Å^{-3}

Table 1
Hydrogen-bonding geometry (Å, °).

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
O1—H1···O3	0.82	1.90	2.6158 (18)	146
O2—H2···O4$^{\rm i}$	0.82	1.82	2.6333 (17)	174
C3—H3···O2$^{\rm ii}$	0.93	2.60	3.422 (2)	148

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

H atoms were positioned geometrically and treated as riding, with O—H = 0.82 Å and C—H = 0.93 or 0.96 Å. $U_{\rm iso}$(H) values were set equal to $xU_{\rm eq}$(carrier atom), where $x = 1.2$ for Csp^2 and $x = 1.5$ for O and methyl C.

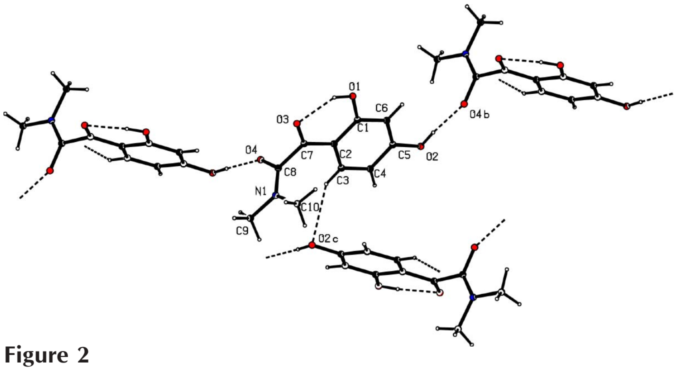

Figure 2
Hydrogen bonding in the crystal structure of (I). Hydrogen bonds are shown as dashed lines. [Symmetry codes: (*b*) $x, -y + \frac{1}{2}, z - \frac{1}{2}$; (*c*) $-x + 1, y - \frac{1}{2}, -z + \frac{3}{2}$]

Data collection: *SMART* (Bruker, 1997); 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, 1997); software used to prepare material for publication: *SHELXTL*.

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