

p-Methoxybenzaldehyde benzoyl-hydrazone monohydrate

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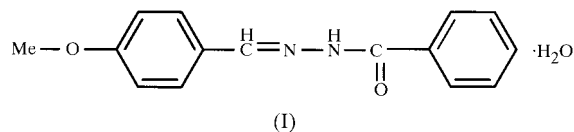
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The crystal structure of the title compound, $C_{15}H_{14}N_2O_2 \cdot H_2O$, is in the keto tautomeric form and the configuration at the azomethine $C=N$ double bond is *E*. The molecule is non-planar, with a dihedral angle of $27.3(1)^\circ$ between the aromatic rings. The crystal structure is stabilized by extensive hydrogen bonding involving the water molecule and hydrazone moiety.

Comment

Aroylhydrazone compounds are being studied extensively because of the strong coordinating hydrazone group which is capable of forming polynuclear complexes. As a continuation of our work on the synthesis and characterization of aroylhydrazone compounds (Fun *et al.*, 1996, 1997, 1999; Lu *et al.*, 1999; Shanmuga Sundara Raj *et al.*, 1999), we report the crystal structure of the title compound, (I).



The molecule is non-planar with a dihedral angle of $27.3(1)^\circ$ between the aromatic rings. The $N1-N2$ and $C9-O2$ bond distances, which are consistent with those in the related compounds *p*-methoxybenzaldehyde isonicotinoylhydrazone monohydrate (Shanmuga Sundara Raj *et al.*, 1999) and its dihydrate derivative (Fun *et al.*, 1996), indicate that these bonds correspond to single and double bonds. Thus, the molecule is in the keto tautomeric form. Also, the configuration at the azomethine $N1-C8$ double bond is *E* (Fig. 1). The $C1-O1$ and $O1-C2$ bond lengths in the hydroxyphenyl moiety are consistent with those [$1.432(2)$ and $1.370(1)$, and $1.436(2)$ and $1.367(2)$ Å, respectively] in the above related

compounds. The keto group is in the plane of the central hydrazone bridge.

The asymmetry of the exocyclic angles at $C5$ is small [$C4-C5-C8 = 122.3(2)^\circ$ and $C6-C5-C8 = 120.0(2)^\circ$], while that at $C2$ is larger [$C3-C2-O1 = 114.7(2)^\circ$ and $C7-C2-O1 = 125.0(2)^\circ$], is similar to that usually found in anisoles, and is caused by the tendency the methoxy group has to be coplanar with the phenyl ring. Conjugation of oxygen with phenyl, which is responsible for this coplanarity (Domiano *et al.*, 1979), also causes some shortening of the $C2-O1$ bond. Some asymmetry is observed also for the exocyclic angles at $C10$ [$C9-C10-C11 = 118.2(2)^\circ$ and $C9-C10-C15 = 122.0(2)^\circ$], probably caused by the contacts $H11 \cdots O2$ of 2.72 Å and $H15 \cdots N2$ of 2.74 Å. The torsion angles $C4-C5-C8-N1$ of $11.7(4)^\circ$ and $N2-C9-C10-C15$ of $-44.7(3)^\circ$ indicate that the methoxyphenyl and phenyl substituents are in synperiplanar and synclinal orientations with respect to the central hydrazone plane. The methoxyphenyl ring makes a dihedral angle of $16.4(1)^\circ$ with the hydrazone bridge and the phenyl ring is twisted by an angle of $43.1(1)^\circ$ with respect to the plane of the central hydrazone linkage.

The water and hydrazone group are involved in $N-H \cdots O$ and $O-H \cdots O$ hydrogen bonds forming a two-dimensional network. The $H1W$ atom is involved in a three-center hydrogen bond.

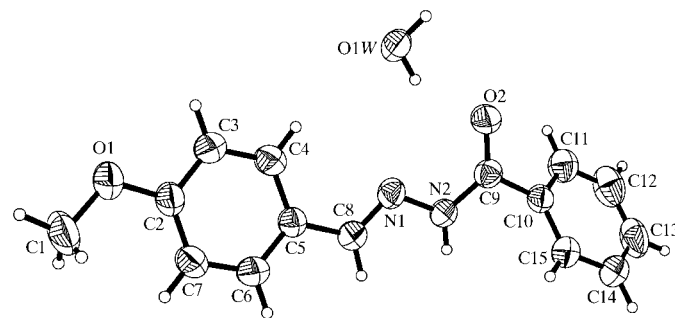


Figure 1
The structure of (I) showing 50% probability displacement ellipsoids and the atom-numbering scheme.

Experimental

The synthesis of the title compound was carried out by reaction of *p*-methoxybenzaldehyde and benzoylhydrazine in ethanol solution under reflux for 3 h. Single crystals were obtained by recrystallization from ethanol.

Crystal data

$C_{15}H_{14}N_2O_2 \cdot H_2O$
 $M_r = 272.30$
Monoclinic, $P2_1/c$
 $a = 11.9398(6)$ Å
 $b = 11.8595(6)$ Å
 $c = 11.3972(6)$ Å
 $\beta = 116.389(1)^\circ$
 $V = 1445.68(13)$ Å³
 $Z = 4$

$D_x = 1.251$ Mg m⁻³
Mo $K\alpha$ radiation
Cell parameters from 3248 reflections
 $\theta = 1.90-29.37^\circ$
 $\mu = 0.088$ mm⁻¹
 $T = 293(2)$ K
Slab, colorless
 $0.40 \times 0.24 \times 0.16$ mm

Data collection

Siemens SMART CCD area-detector diffractometer
 ω scans
 10036 measured reflections
 3650 independent reflections
 1704 reflections with $I > 2\sigma(I)$

$R_{\text{int}} = 0.067$
 $\theta_{\text{max}} = 29.34^\circ$
 $h = -16 \rightarrow 14$
 $k = -12 \rightarrow 16$
 $l = -14 \rightarrow 15$

Refinement

Refinement on F^2
 $R[F^2 > 2\sigma(F^2)] = 0.053$
 $wR(F^2) = 0.120$
 $S = 0.925$
 2833 reflections
 190 parameters
 H atoms: see below

$w = 1/[\sigma^2(F_o^2) + (0.0764P)^2]$
 where $P = (F_o^2 + 2F_c^2)/3$
 $(\Delta/\sigma)_{\text{max}} < 0.001$
 $\Delta\rho_{\text{max}} = 0.23 \text{ e } \text{\AA}^{-3}$
 $\Delta\rho_{\text{min}} = -0.21 \text{ e } \text{\AA}^{-3}$
 Extinction correction: *SHELXTL*
 (Sheldrick, 1997)
 Extinction coefficient: 0.013 (3)

Table 1

Selected geometric parameters (\AA , $^\circ$).

O1—C2	1.371 (2)	N1—N2	1.392 (2)
O1—C1	1.434 (3)	N2—C9	1.346 (3)
O2—C9	1.236 (2)	C5—C8	1.459 (3)
N1—C8	1.274 (3)	C10—C15	1.368 (3)
C8—N1—N2	115.3 (2)	O2—C9—C10	121.6 (2)
C9—N2—N1	119.1 (2)	N2—C9—C10	115.7 (2)
O2—C9—N2	122.7 (2)		
C8—N1—N2—C9	−173.6 (2)	N1—N2—C9—C10	178.7 (2)
N2—N1—C8—C5	−179.9 (2)		

Table 2

Hydrogen-bonding geometry (\AA , $^\circ$).

$D-H\cdots A$	$D-H$	$H\cdots A$	$D\cdots A$	$D-H\cdots A$
O1W—H1W \cdots N1	0.90 (3)	2.53 (2)	3.257 (2)	138 (3)
O1W—H1W \cdots O2	0.90 (3)	2.05 (4)	2.858 (3)	150 (3)
N2—H2 \cdots O1W ⁱ	0.86	1.99	2.829 (2)	165
O1W—H2W \cdots O2 ⁱⁱ	0.89 (3)	1.95 (3)	2.825 (2)	169 (3)

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

The collection of intensity data was as described by Shanmuga Sundara Raj *et al.* (1999). The H atoms of the water molecule were refined isotropically while all the others were fixed geometrically and allowed to ride on their parent atoms.

Data collection: *SMART* (Siemens, 1996); cell refinement: *SAINT* (Siemens, 1996); data reduction: *SAINT*; program(s) used to solve structure: *SHELXTL* (Sheldrick, 1997); program(s) used to refine structure: *SHELXTL*; molecular graphics: *SHELXTL*; software used to prepare material for publication: *SHELXTL*, *PARST* (Nardelli, 1995) and *PLATON* (Spek, 1990).

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Supplementary data for this paper are available from the IUCr electronic archives (Reference: NA1476). Services for accessing these data are described at the back of the journal.

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