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p-Methoxybenzaldehyde benzoylhydrazone monohydrate

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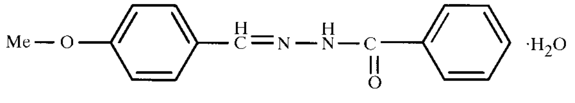
The crystal structure of the title compound, $C_{15}H_{14}N_2O_2{\cdot}H_2O$, is in the keto tautomeric form and the configuration at the azomethine C=N double bond is *E*. The molecule is non-planar, with a dihedral angle of 27.3 (1)° between the aromatic rings. The crystal structure is stabilized by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



(I)

The molecule is non-planar with a dihedral angle of 27.3 (1)° 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) Å and C6—C5—C8 = 120.0 (2)°], while that at C2 is larger [C3—C2—O1 = 114.7 (2) Å and C7—C2—O1 = 125.0 (2)°], 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)° and C9—C10—C15 = 122.0 (2)°], probably caused by the contacts H11···O2 of 2.72 Å and H15···N2 of 2.74 Å. The torsion angles C4—C5—C8—N1 of 11.7 (4)° and N2—C9—C10—C15 of −44.7 (3)° 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)° with the hydrazone bridge and the phenyl ring is twisted by an angle of 43.1 (1)° with respect to the plane of the central hydrazone linkage.

The water and hydrazone group are involved in N—H···O and O—H···O hydrogen bonds forming a two-dimensional network. The H1*W* atom is involved in a three-center hydrogen bond.

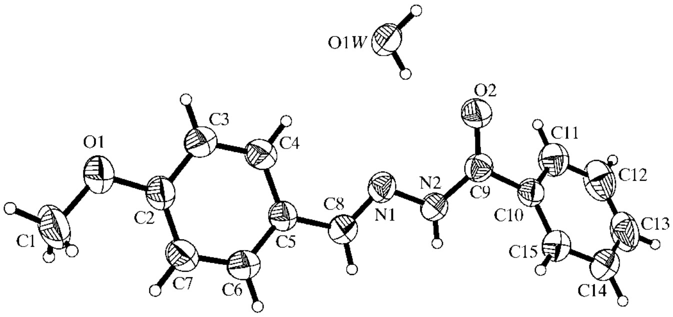


Figure 1
The structure of (I) showing 50% probability displacement ellipsoids and the atom-numbering scheme.

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)°
$V = 1445.68$ (13) Å^3
$Z = 4$
$D_x = 1.251$ Mg m^{-3}
Mo $K\alpha$ radiation
Cell parameters from 3248 reflections
$\theta = 1.90$–29.37°
$\mu = 0.088$ mm^{-1}
$T = 293$ (2) K
Slab, colorless
0.40 × 0.24 × 0.16 mm

Data collection

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

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

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

Table 1
Selected geometric parameters (Å, °).

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 (Å, °).

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
O1*W*—H1*W*···N1	0.90 (3)	2.53 (2)	3.257 (2)	138 (3)
O1*W*—H1*W*···O2	0.90 (3)	2.05 (4)	2.858 (3)	150 (3)
N2—H2···O1*W*^{i}	0.86	1.99	2.829 (2)	165
O1*W*—H2*W*···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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