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2′-(3,4-Dimethoxybenzylidene)-2-hydroxybenzohydrazide

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Key indicators

Single-crystal X-ray study
T = 293 K
Mean σ(C–C) = 0.003 Å
R factor = 0.042
wR factor = 0.093
Data-to-parameter ratio = 13.8

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

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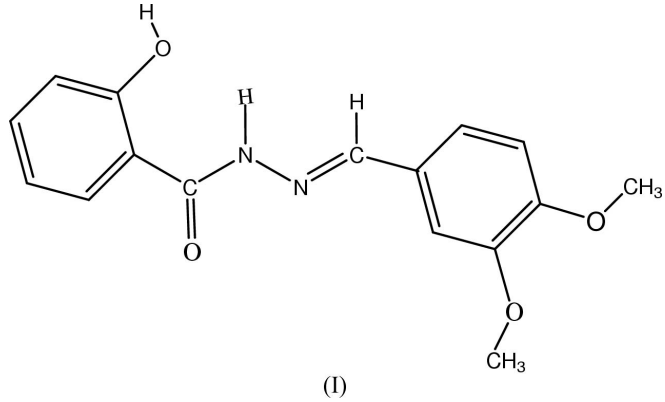
The title compound, $C_{16}H_{16}N_2O_4$, was synthesized by the reaction of 2-hydroxybenzoylhydrazine with 3,4-dimethoxybenzaldehyde in ethanol. The molecule is non-planar and the dihedral angle between the two aromatic rings is 32.76 (7)°. O—H···O and C—H···O hydrogen bonds link the glide-related molecules into a chain along [101].

Comment

Some benzoylhydrazone compounds possess bacteriostatic activity. This type of compound has wide application in tuberculosis treatment and also exhibits fungicidal activity (Edwards *et al.*, 1975). Some hydrazonocarbonyl compounds also show bioactivity (Zhi *et al.*, 2003; Yang & Pan, 2004). In order to explore more effective antibacterial medicines, we have synthesized the title compound, (I).



(I)

The title molecule (Fig. 1) is non-planar; the dihedral angle between the two aromatic rings is 32.76 (7)°. As a result of conjugation, the C═O distance [1.2236 (17) Å] is longer than the normal value of 1.20 Å, and the C1—N1 bond distance [1.337 (2) Å] is longer than the C═N double-bond distance (1.32 Å; John, 1998) and shorter than the C—N single-bond distance (1.475 Å; John, 1998). The two methoxy groups are slightly twisted away from the planes of the attached benzene rings [C15—O3—C12—C11 = −5.6 (3)° and C16—O4—C13—C14 = 11.9 (2)°].

An intramolecular N1—H1···O2 hydrogen bond forming a six-membered ring is observed between the NH group and the hydroxy O atom (Table 1). The O2—H2···N2^{i} and O2—H2···O1^{i} [symmetry code: (i) $\frac{1}{2}+x$, $\frac{1}{2}-y$, $\frac{1}{2}+z$] hydrogen bonds form a five-membered ring. These hydrogen bonds link the glide-related molecules into a chain along [101] (Fig. 2). The chain structure is further stabilized by a C8—H8···O1^{i} hydrogen bond.

Experimental

2-Hydroxybenzoylhydrazine (0.02 mol, 3.04 g) was dissolved in anhydrous ethanol (50 ml) and 3,4-dimethoxybenzaldehyde (0.02 mol, 3.32 g) was added. The mixture was refluxed for 2 h, and the precipitate which formed was collected by filtration and washed with ethanol. The product was recrystallized from ethanol and dried under reduced pressure to give the title compound. The compound (2.0 mmol, 0.55 g) was dissolved in dimethylformamide (30 ml) and kept at room temperature for 30 d to obtain colourless single crystals, which were collected and washed with distilled water.

Crystal data

$C_{16}H_{16}N_2O_4$
M_r = 300.31
Monoclinic, $P2_1/n$
a = 6.2877 (6) Å
b = 21.724 (2) Å
c = 10.9742 (11) Å
β = 90.390 (2)°
V = 1499.0 (3) Å^3
Z = 4
D_x = 1.331 Mg m^{-3}
Mo $K\alpha$ radiation
Cell parameters from 1585 reflections
θ = 5.3–48.1°
μ = 0.10 mm^{-1}
T = 293 (2) K
Block, colourless
0.42 × 0.35 × 0.32 mm

Data collection

Bruker SMART APEX area-detector diffractometer
φ and ω scans
Absorption correction: multi-scan (*SADABS*; Bruker, 2002)
T_{min} = 0.960, T_{max} = 0.970
8757 measured reflections
3226 independent reflections
1797 reflections with $I > 2\sigma(I)$
R_{int} = 0.037
θ_{max} = 27.0°
$h = -8 \rightarrow 6$
$k = -27 \rightarrow 27$
$l = -12 \rightarrow 14$

Refinement

Refinement on F^2
$R[F^2 > 2\sigma(F^2)]$ = 0.042
$wR(F^2)$ = 0.093
S = 0.87
3226 reflections
234 parameters
H atoms treated by a mixture of independent and constrained refinement
$w = 1/[\sigma^2(F_o^2) + (0.036P)^2]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max}$ = 0.001
$\Delta\rho_{max}$ = 0.13 e Å^{-3}
$\Delta\rho_{min}$ = −0.11 e Å^{-3}
Extinction correction: *SHELXL97*
Extinction coefficient: 0.0041 (10)

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

D—H···A	D—H	H···A	D···A	D—H···A
O2—H2···N2^{i}	0.81 (2)	2.39 (2)	3.098 (2)	147 (2)
O2—H2···O1^{i}	0.81 (2)	2.11 (2)	2.791 (2)	142 (2)
N1—H1···O2	0.84 (3)	2.01 (2)	2.662 (2)	133 (1)
C3—H3···O1	0.96 (2)	2.36 (2)	2.742 (2)	103 (1)
C8—H8···O1^{ii}	0.93 (3)	2.39 (2)	3.281 (2)	162 (1)

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

Atoms H1, H2, H3, H5, H8, H10, H11 and H14 were located in a difference map and their parameters were refined. The remaining H atoms were positioned geometrically and allowed to ride on their parent atoms, with C—H = 0.93 or 0.96 Å and U_{iso}(H) = 1.2 or 1.5U_{eq}(parent atom). For the refined H atoms, N—H = 0.845 (17) Å, O—H = 0.81 (2) Å and C—H = 0.925 (15)–0.961 (17) Å.

Data collection: *SMART* (Bruker, 2002); cell refinement: *SAINT* (Bruker, 2002); data reduction: *SAINT*; program(s) used to solve structure: *SHELXTL* (Bruker, 2002); program(s) used to refine structure: *SHELXTL*; molecular graphics: *SHELXTL*; software used to prepare material for publication: *SHELXTL*.

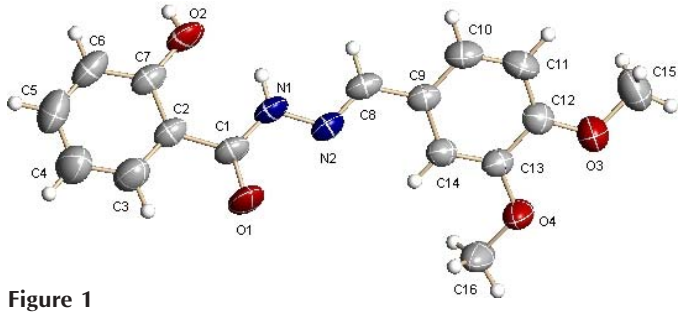


Figure 1
The structure of (I), showing the atomic numbering. Displacement ellipsoids are drawn at the 30% probability level.

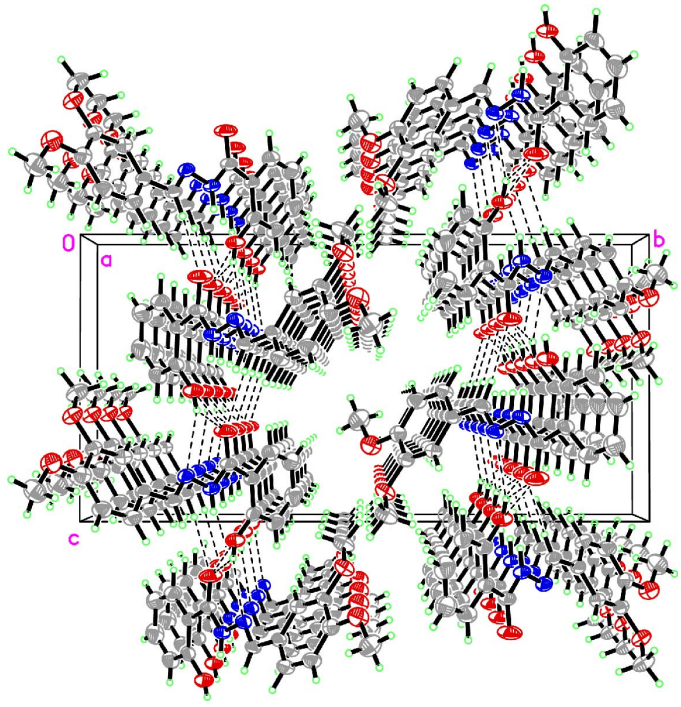


Figure 2
The packing of (I), viewed down the *a* axis, showing the hydrogen-bonded chains. Hydrogen bonds are indicated by dashed lines.

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