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4-Hydroxy-3,5-dimethoxybenzaldehyde 3,4,5-trimethoxybenzoylhydrazone monohydrate

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Key indicators

Single-crystal X-ray study
T = 273 K
Mean σ(C–C) = 0.003 Å
R factor = 0.053
wR factor = 0.151
Data-to-parameter ratio = 17.6

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

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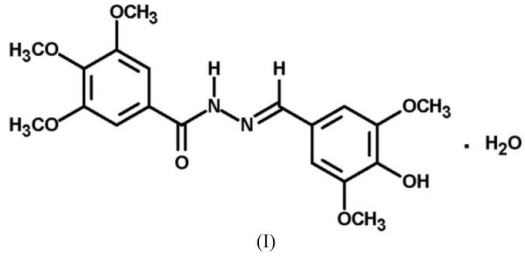
The title compound, $C_{19}H_{22}N_2O_7\cdot H_2O$, was synthesized as part of a continuing project involving the structures of hydrazone derivatives. The molecules pack in a three-dimensional framework structure by a combination of O—H···O, O—H···N and N—H···O hydrogen bonds.

Comment

Molecules containing hydrazone units are of interest to us (Sun *et al.*, 2006) owing to their broad spectrum of activities and applications (Gup & Kirkan, 2005; Ganjali *et al.*, 2006; Getautis *et al.*, 2006). Moreover, many hydrazones have also been used as ligands for complexation of metal ions (Kuriakose *et al.*, 2007).



(I)

Compound (I) possesses normal geometric parameters (Fig. 1) (Allen *et al.*, 1987). The central hydrazone component is effectively planar with an all-*trans* extended conformation (Table 1). On the other hand, the two benzene rings form an angle of 19.1 (1)° to one another and the C11 benzene ring is slightly twisted out of the plane of the central hydrazone unit. The existence of (I) in the expected keto form is evident from the C10—O4 bond length of 1.231 (2) Å. Of the five methoxy groups only O2/C7 is coplanar with one of the benzene rings, with the others showing out-of-plane twists ranging from ∼15 to 85° (Table 1). Similar geometry has been observed in related hydrazone analogues (Peralta *et al.*, 2007; Raj & Kurup, 2007).

The molecules are linked into a three-dimensional framework structure by a combination of six independent hydrogen bonds (Table 2, Fig. 2). The water molecule plays a significant role in the crystal packing as it is the acceptor in one hydrogen bond and a donor in three others, including one bifurcated bond (Table 2).

Experimental

A mixture of 4-hydroxy-3,5-dimethoxybenzaldehyde (1 mmol) and 3,4,5-trimethoxybenzoylhydrazide (1 mmol) in anhydrous ethanol (30 ml) was refluxed for 5 h and then cooled to room temperature.

The precipitate was filtered off and dried. The crude product was recrystallized from ethanol. Colorless crystals were obtained in 76% yield. Analysis calculated for $C_{19}H_{24}N_2O_8$: C 55.88, H 5.92, N 6.86%; found: C 55.83, H 5.95, N 6.92%. A single crystal suitable for an X-ray structural analysis was obtained by slowly evaporating an ethanol solution at room temperature.

Crystal data

$C_{19}H_{22}N_2O_7\cdot H_2O$
M_r = 408.40
Monoclinic, $P2_1/c$
a = 10.1299 (4) Å
b = 13.8108 (6) Å
c = 14.8758 (6) Å
β = 109.598 (2)°
V = 1960.59 (14) Å^3
Z = 4
Mo $K\alpha$ radiation
μ = 0.11 mm^{-1}
T = 273 (2) K
0.35 × 0.24 × 0.12 mm

Data collection

Bruker SMART CCD area-detector diffractometer
Absorption correction: multi-scan (*SADABS*; Sheldrick, 1996)
T_{min} = 0.963, T_{max} = 0.987
26378 measured reflections
4755 independent reflections
2463 reflections with $I > 2\sigma(I)$
R_{int} = 0.071

Refinement

$R[F^2 > 2\sigma(F^2)]$ = 0.053
$wR(F^2)$ = 0.151
S = 1.03
4755 reflections
270 parameters
H-atom parameters constrained
$\Delta\rho_{max}$ = 0.26 e Å^{-3}
$\Delta\rho_{min}$ = −0.22 e Å^{-3}

Table 1
Selected torsion angles (°).

C9—N1—N2—C10	170.63 (18)	C1—C2—O2—C7	−175.5 (2)
N1—N2—C10—C11	−173.93 (16)	N2—C10—C11—C16	−15.4 (3)
C8—O3—C6—C5	33.3 (3)	C16—C15—O7—C19	15.0 (4)
N2—N1—C9—C4	−177.10 (18)	C14—C13—O5—C17	95.6 (3)
C5—C4—C9—N1	−176.0 (2)	C13—C14—O6—C18	84.6 (3)

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

D—H···A	D—H	H···A	D···A	D—H···A
O1—H1···O8	0.85	1.94	2.727 (2)	154
O1—H1···O3	0.85	2.26	2.704 (2)	113
N2—H2···O1^{i}	0.86	2.49	3.313 (2)	160
O8—H17···O4^{ii}	0.85	2.19	3.033 (2)	171
O8—H18···O4^{iii}	0.85	2.17	2.956 (2)	154
O8—H18···N1^{iii}	0.85	2.50	3.143 (2)	133

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

All H atoms were initially located in a difference Fourier map and were treated as riding atoms, with C—H = 0.93 (Csp^2) and 0.96 Å(methyl), N—H = 0.86 Å, and O—H = 0.85 Å, and with U_{iso}(H) = kU_{eq}(C,N,O), where k = 1.5 for the methyl and the hydroxyl groups and k = 1.2 for all other H atoms.

Data collection: *SMART* (Siemens, 1996); cell refinement: *SAINT* (Siemens, 1996); data reduction: *SAINT* (Siemens, 1996); program(s) used to solve structure: *SHELXS97* (Sheldrick, 1997*a*); program(s) used to refine structure: *SHELXL97* (Sheldrick, 1997*a*); molecular graphics: *SHELXTL* (Sheldrick, 1997*b*); software used to prepare material for publication: *SHELXTL*.

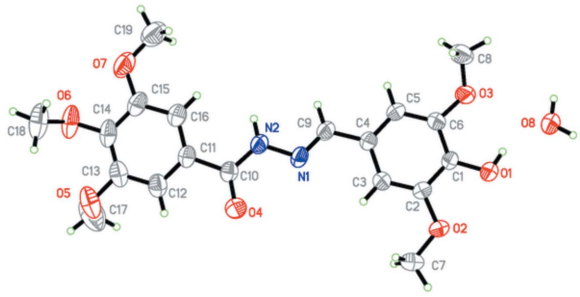


Figure 1
The molecular structure of (I), showing the atom-labeling scheme. Displacement ellipsoids are drawn at the 50% probability level. H atoms are shown as small spheres of arbitrary radius.

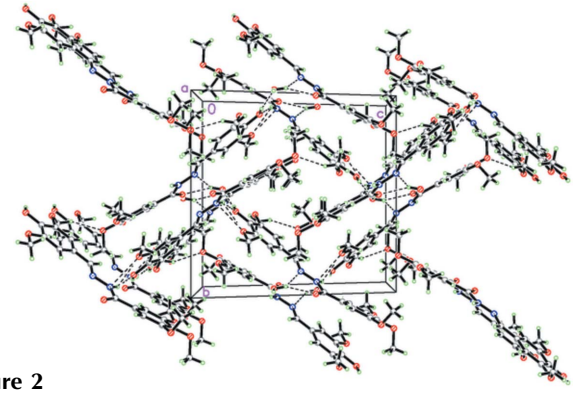


Figure 2
A packing diagram for (I), viewed down the *a* axis. Dashed lines indicate hydrogen bonds.

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