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2′-(2-Hydroxy-4-methoxybenzylidene)-nicotinohydrazide monohydrate

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In the crystal structure of the title compound, $C_{14}H_{15}N_3O_4$, the methoxyphenyl ring makes a dihedral angle of 2.27 (6)° with the hydrazone group. The crystal packing is stabilized by N—H···O and O—H···O hydrogen bonds.

Key indicators

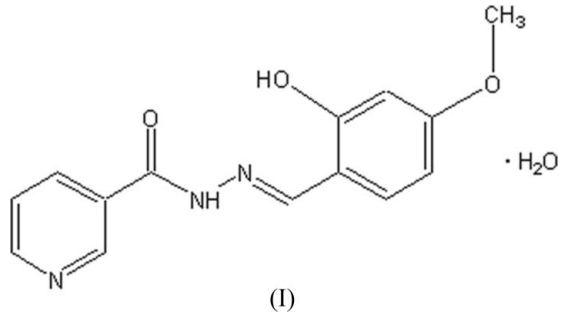
Single-crystal X-ray study
T = 150 K
Mean σ(C–C) = 0.002 Å
R factor = 0.046
wR factor = 0.148
Data-to-parameter ratio = 11.4

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

Comment

The coordination chemistry of hydrazones containing N and O donors and their metal complexes has gained considerable attraction because of their biological activity and their ability to act as potential inhibitors for many enzymes (Song *et al.*, 1994; Lu *et al.*, 1996). The presence of heterocyclic rings in the hydrazones is important for their pharmacological properties (Dilworth, 1976). Hydrazone ligands can coordinate to metal ions to produce stable metal complexes owing to their facile keto–enol tautomerism.

The molecular structure of the title compound, (I), is shown in Fig. 1. The dihedral angle between the aromatic rings is 1.78 (8)°. The C═N double bond is *trans* configured. The N1—N2 and C9—O3 bond distances are in agreement with those in *p*-methoxybenzaldehyde benzoylhydrazone monohydrate (Shanmuga Sundara Raj *et al.*, 2000) and isonicotinoyl hydrazone monohydrate (Shanmuga Sundara Raj *et al.*, 1999).



(I)

In the crystal structure, which is stabilized by N—H···O and O—H···O hydrogen bonds, the molecules are arranged in layers.

Experimental

Nicotinic acid hydrazide (1 mmol) dissolved in ethanol (15 ml) was refluxed with 2-hydroxy-4-methoxybenzaldehyde (1 mmol) in ethanol (15 ml) in the presence of a few drops of glacial acetic acid for 5 h. Single crystals suitable for X-ray analysis were obtained by recrystallization from a methanol – water (1:1) mixture.

Crystal data

$C_{14}H_{13}N_3O_3\cdot H_2O$
M_r = 289.29
Triclinic, $P\overline{1}$
a = 6.452 (2) Å
b = 7.884 (3) Å
c = 14.128 (4) Å
α = 80.11 (3)°
β = 77.24 (3)°
γ = 75.90 (3)°
V = 674.5 (4) Å^3
Z = 2
Mo $K\alpha$ radiation
μ = 0.11 mm^{-1}
T = 150 (2) K
0.33 × 0.26 × 0.21 mm

Data collection

Oxford Diffraction Xcalibur-S diffractometer
Absorption correction: multi-scan (*CrysAlis RED*; Oxford Diffraction, 2006)
T_{min} = 0.966, T_{max} = 0.978
7088 measured reflections
2370 independent reflections
1606 reflections with $I > 2\sigma(I)$
R_{int} = 0.022

Refinement

$R[F^2 > 2\sigma(F^2)]$ = 0.046
$wR(F^2)$ = 0.148
S = 1.06
2370 reflections
207 parameters
H atoms treated by a mixture of independent and constrained refinement
$\Delta\rho_{max}$ = 0.53 e Å^{-3}
$\Delta\rho_{min}$ = −0.27 e Å^{-3}

Table 1
Selected geometric parameters (Å, °).

O1—C2	1.360 (2)	N1—N2	1.386 (2)
O1—C1	1.430 (2)	N2—C9	1.352 (2)
O3—C9	1.227 (2)	C5—C8	1.446 (3)
N1—C8	1.283 (2)		
C2—O1—C1	118.13 (14)	O3—C9—N2	122.41 (17)
C8—N1—N2	116.23 (16)	O3—C9—C10	120.84 (17)
C9—N2—N1	118.30 (16)		

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

D—H···A	D—H	H···A	D···A	D—H···A
N2—H2N···O4^{i}	0.93 (3)	1.99 (3)	2.902 (3)	170 (2)
O4—H111···O3	0.95 (3)	1.83 (3)	2.749 (2)	161 (3)
O2—H102···N1	0.93 (3)	1.84 (3)	2.657 (2)	145 (3)
O4—H112···N3^{ii}	0.83 (3)	2.12 (3)	2.950 (3)	173 (3)

Symmetry codes: (i) $x+1, y, z$; (ii) $x, y-1, z$.

C–bonded H atoms were placed in calculated positions and refined with C—H bond lengths constrained to 0.93 Å (aromatic CH) and 0.96 Å (methyl CH_3) and U_{iso}(H) = 1.2U_{eq}(C) or 1.5 U_{eq}(C_{methyl}). The methyl group was allowed to rotate but not to tip. H atoms bonded to N and O were freely refined; distances are given in Table 2.

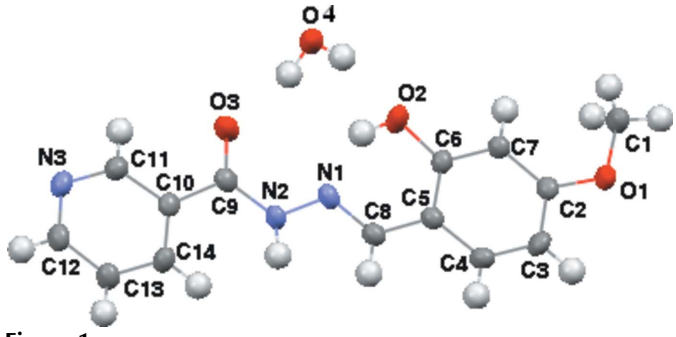


Figure 1
The molecular structure of compound (I), with 50% probability displacement ellipsoids and the atom numbering scheme.

Data collection: *CrysAlis CCD* (Oxford Diffraction, 2006); cell refinement: *CrysAlis RED* (Oxford Diffraction, 2006); data reduction: *CrysAlis RED*; 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: *SHELXL97*.

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