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3-Methoxysalicylaldehyde 4-methoxybenzoylhydrazone monohydrate

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Key indicators

Single-crystal X-ray study
T = 295 K
Mean σ(C–C) = 0.004 Å
R factor = 0.041
wR factor = 0.102
Data-to-parameter ratio = 9.2

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

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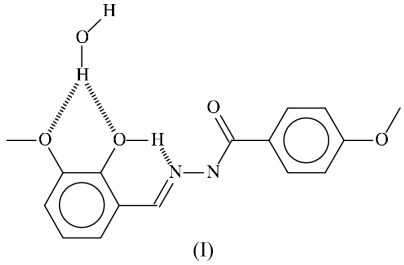
The title compound, $C_{16}H_{16}N_2O_4\cdot H_2O$, crystallizes as a monohydrate in which the water molecule forms bifurcated hydrogen bonds to the hydroxyl and methoxy O atoms of the methoxysalicyldehyde portion of the Schiff base. The molecule is approximately planar; hydrogen-bonding interactions involving the amino group and the water molecule give rise to a layer structure.

Comment

Hydroxy-substituted benzaldehyde reagents used for condensation with benzoylhydrazine afford Schiff base hydrazones which can function as terdentate ligands towards a number of metal cations (Gao *et al.*, 1998; Liu & Gao, 1998; Chen *et al.*, 1999). In our studies on the title hydrazone, (I), we have isolated V^{5+} and Fe^{3+} complexes (Huo, Gao, Liu, Li & Ng, 2004; Huo, Gao, Liu, Li, Zhao & Zhao *et al.*, 2004; Huo, Gao, Liu, Zhao & Ng, 2004) in which the deprotonated 3-methoxysalicylaldehyde 4-methoxybenzoylhydrazone entity chelates in this manner. This hydrazone crystallizes as a monohydrate (Fig. 1).



(I)

The hydrazone is essentially flat; the aromatic ring (C10–C15) makes a dihedral angle of 5.1 (5)° with the C9/N2/O3 fragment. In the unsubstituted compound, *viz*. benzaldehyde benzoylhydrazone, the molecule is twisted with the dihedral angle between the corresponding ring and fragment being

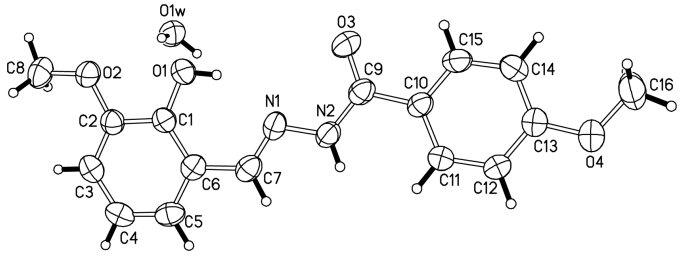


Figure 1
*ORTEP*II (Johnson, 1976) plot of the title compound. Displacement ellipsoids for non-H atoms are drawn at the 50% probability level. H atoms are drawn as spheres of arbitrary radii.

23.6° (Litvinov *et al.*, 1991). This feature is also noted in salicyldehydyde benzoylhydrazone (dihedral angle = 21.5°; Lyubchova *et al.*, 1995). As geometry optimizations on (I) also implicate a twisted conformation, the observed planarity can be attributed to water molecules of crystallization, whose presence ensures that the molecules can be packed efficiently in a layer motif (Fig. 2). One of its H atoms engages in binding to two O atoms simultaneously (Table 2).

Experimental

An ethanol solution (25 ml) of 3-methoxysalicylaldehyde (6.04 g, 0.04 mol) was added dropwise to an ethanol solution (100 ml) of 4-methoxybenzoylhydrazine (6.44 g, 0.04 mol). The mixture was refluxed for a hour to complete the condensation. Yellow crystals (m.p. 442–444 K) were isolated from the filtered solution after a few days. Analysis calculated for $C_{16}H_{18}N_2O_5$: C 60.37, H 5.70, N 8.80%; found: C 60.54, H 5.95, N 8.61%.

Crystal data

$C_{16}H_{16}N_2O_4\cdot H_2O$
M_r = 318.32
Orthorhombic, $P2_12_12_1$
a = 5.013 (1) Å
b = 12.733 (3) Å
c = 24.096 (5) Å
V = 1537.9 (5) Å^3
Z = 4
D_x = 1.375 Mg m^{-3}
Mo $K\alpha$ radiation
Cell parameters from 14318 reflections
θ = 3.0–27.5°
μ = 0.10 mm^{-1}
T = 295 (2) K
Prism, yellow
0.39 × 0.26 × 0.21 mm

Data collection

Rigaku R-AXIS RAPID diffractometer
ω scans
Absorption correction: multi-scan (*ABSCOR*; Higashi, 1995)
T_{min} = 0.830, T_{max} = 0.979
14735 measured reflections
2073 independent reflections
1784 reflections with $I > 2\sigma(I)$
R_{int} = 0.038
θ_{max} = 27.5°
$h = -6 \rightarrow 5$
$k = -16 \rightarrow 16$
$l = -31 \rightarrow 31$

Refinement

Refinement on F^2
$R[F^2 > 2\sigma(F^2)]$ = 0.041
$wR(F^2)$ = 0.102
S = 1.04
2073 reflections
226 parameters
H atoms treated by a mixture of independent and constrained refinement
$w = 1/[\sigma^2(F_o^2) + (0.0563P)^2 + 0.2354P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max}$ = 0.001
$\Delta\rho_{max}$ = 0.24 e Å^{-3}
$\Delta\rho_{min}$ = −0.12 e Å^{-3}

Table 1
Selected geometric parameters (Å, °).

O1—C1	1.348 (3)	N1—N2	1.382 (3)
O2—C2	1.373 (3)	N1—C7	1.284 (3)
O2—C8	1.426 (3)	N2—C9	1.353 (3)
O3—C9	1.228 (3)	C6—C7	1.458 (3)
O4—C13	1.367 (3)	C9—C10	1.490 (3)
O4—C16	1.417 (3)		
C2—O2—C8	117.2 (2)	C5—C6—C7	118.6 (2)
C13—O4—C16	118.1 (2)	N1—C7—C6	122.6 (2)
C7—N1—N2	114.5 (2)	O3—C9—N2	122.1 (2)
C9—N2—N1	120.3 (2)	O3—C9—C10	121.2 (2)
O1—C1—C2	116.8 (2)	N2—C9—C10	116.7 (2)
O1—C1—C6	123.9 (2)	C15—C10—C9	117.6 (2)
O2—C2—C1	114.3 (2)	C11—C10—C9	124.9 (2)
O2—C2—C3	125.4 (2)	O4—C13—C12	116.0 (2)
C1—C6—C7	122.2 (2)	O4—C13—C14	124.7 (2)

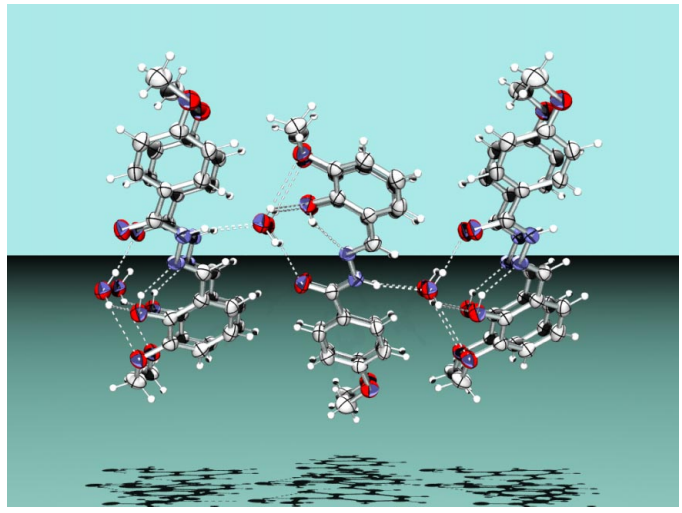


Figure 2
POV-Ray (Cason, 2002)/*ORTEP*II (Johnson, 1976) plot of the hydrogen-bonded layer structure, viewed along the *a* axis.

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

D—H···A	D—H	H···A	D···A	D—H···A
O1—H1*o*···N1	0.85 (1)	1.96 (2)	2.700 (2)	145 (3)
O1*w*—H1*w*1···O1	0.86 (1)	2.33 (2)	3.067 (3)	145 (3)
O1*w*—H1*w*1···O2	0.86 (1)	2.40 (3)	3.104 (3)	140 (4)
O1*w*—H1*w*2···O3^{i}	0.87 (1)	1.84 (1)	2.701 (2)	171 (3)
N2—H2*n*···O1*w*^{ii}	0.85 (1)	2.08 (1)	2.904 (3)	165 (2)

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

The H atoms were placed in calculated positions [aromatic C—H = 0.93 Å and U_{iso}(H) = 1.2U_{eq}(C)]. Those of the methoxy groups were rotated to fit the electron density [C—H = 0.96 Å and U_{iso}(H) = 1.5U_{eq}(C)]. The H atoms were included in the refinements in the riding-model approximation. The water and amino H atoms were located and refined with distance restraints of O—H = N—H = 0.85 (1) Å and H···H = 1.39 (1) Å. In the absence of significant anomalous dispersion effects, Friedel pair reflections were merged before the final refinement.

Data collection: *RAPID-AUTO* (Rigaku, 1998); cell refinement: *RAPID-AUTO*; data reduction: *CrystalStructure* (Rigaku/MSC, 2002); program(s) used to solve structure: *SHELXS*97 (Sheldrick, 1997); program(s) used to refine structure: *SHELXL*97 (Sheldrick, 1997); molecular graphics: *ORTEP*II (Johnson, 1976) and *POV-Ray* (Cason, 2002); software used to prepare material for publication: *SHELXL*97.

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