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2-Hydroxy-3-methoxybenzaldehyde 2,4-dinitrophenylhydrazone dimethylformamide solvate

Zuo-Liang Jing, Ming Yu, Xin Chen, Chun-Hua Diao, Qi-Liang Deng and Zhi Fan*

College of Sciences, Tianjin University of Science and Technology, Tianjin 300222, People's Republic of China

Correspondence e-mail: zhifan@public.tpt.tj.cn

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Key indicators

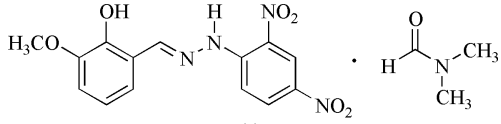
Single-crystal X-ray study
T = 293 K
Mean σ(C–C) = 0.004 Å
R factor = 0.056
wR factor = 0.165
Data-to-parameter ratio = 14.0

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

The title compound, $C_{14}H_{12}N_4O_6\cdot C_3H_7NO$, was prepared by the reaction of 2-hydroxy-3-methoxybenzaldehyde and 2,4-dinitrophenylhydrazine in *N*,*N*-dimethylformamide. In the crystal structure, the molecule of 2-hydroxy-3-methoxybenzaldehyde-2,4-dinitrophenylhydrazone is approximately planar, with a mean deviation of non-H atoms from the plane of 0.109 Å. The crystal packing is stabilized by O—H···O and N—H···O hydrogen bonds and aromatic packing interactions.

Comment

Schiff base ligands derived from 2-hydroxy-3-methoxybenzaldehyde, such as semicarbazide 2,6-pyridinediamine, have been reported previously (Li *et al.*, 1995; Galić *et al.*, 2000). As part of an investigation of the coordination properties of Schiff base compounds, we report the synthesis and molecular structure of the title compound, (I) (Fig. 1).



(I)

The bond lengths of C6—C8 [1.460 (4) Å], C8—N1 [1.274 (3) Å] and N1—N2 [1.374 (3) Å] correspond to those in analogous compounds (Li *et al.* 1995; He *et al.* 2002). In the crystal structure, the molecule of 2-hydroxy-3-methoxybenzaldehyde-2,4-dinitrophenylhydrazine is approximately planar, with a mean deviation of non-H atoms from the plane of 0.109 Å.

O—H···O and N—H···O hydrogen bonds (Table 1) are observed in the crystal structure of (I). The relatively short distance of 3.404 Å between adjacent parallel aromatic rings indicates the presence of π–π stacking interactions, which

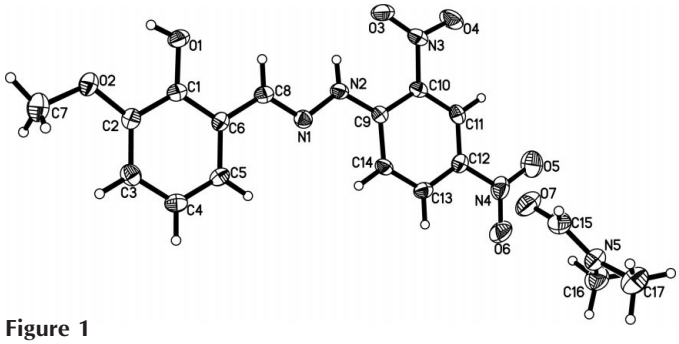


Figure 1
A view of (I), showing the atom-numbering scheme. Displacement ellipsoids are drawn at the 30% probability level and H atoms are shown as small spheres of arbitrary radii.

stabilize the crystal packing (Fig. 2), together with the hydrogen bonds.

Experimental

An anhydrous ethanol solution of 2-hydroxy-3-methoxybenzaldehyde (1.52 g, 10 mmol) was added to an anhydrous ethanol solution of 2,4-dinitrophenylhydrazine (1.98 g, 10 mmol), and the mixture was stirred at 343 K for 5 h under nitrogen. A yellow precipitate was formed. The product was isolated and was recrystallized from ethanol, and then dried *in vacuo* to give pure compound (I) in 78% yield. Bright yellow single crystals of (I) suitable for X-ray analysis were obtained by slow evaporation of a solution in *N,N*-dimethyl formamide.

Crystal data

$C_{14}H_{12}N_4O_6\cdot C_3H_7NO$
$M_r = 405.37$
Triclinic, $P\overline{1}$
$a = 7.015\ (4)$ Å
$b = 7.759\ (4)$ Å
$c = 18.638\ (9)$ Å
$\alpha = 89.456\ (9)^\circ$
$\beta = 84.369\ (9)^\circ$
$\gamma = 68.346\ (8)^\circ$
$V = 938.0\ (8)$ Å^3
$Z = 2$
$D_x = 1.435$ Mg m^{-3}
Mo $K\alpha$ radiation
Cell parameters from 640 reflections
$\theta = 3.1$–25.9°
$\mu = 0.11$ mm^{-1}
$T = 293\ (2)$ K
Block, yellow
$0.28 \times 0.18 \times 0.16$ mm

Data collection

Bruker SMART CCD area-detector diffractometer
φ and ω scans
Absorption correction: multi-scan (*SADABS*; Bruker, 1999)
$T_{min} = 0.966$, $T_{max} = 0.982$
5446 measured reflections
3785 independent reflections
2059 reflections with $I > 2\sigma(I)$
$R_{int} = 0.026$
$\theta_{max} = 26.4^\circ$
$h = -8 \rightarrow 8$
$k = -9 \rightarrow 9$
$l = -23 \rightarrow 13$

Refinement

Refinement on F^2
$R[F^2 > 2\sigma(F^2)] = 0.056$
$wR(F^2) = 0.165$
$S = 1.03$
3785 reflections
270 parameters
H atoms treated by a mixture of independent and constrained refinement
$w = 1/[\sigma^2(F_o^2) + (0.0785P)^2]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} < 0.001$
$\Delta\rho_{max} = 0.20$ e Å^{-3}
$\Delta\rho_{min} = -0.22$ e Å^{-3}

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

D—H···A	D—H	H···A	D···A	D—H···A
O1—H1···O2	0.82	2.21	2.664 (3)	115
O1—H1···O7^{i}	0.82	2.01	2.745 (3)	149
N2—H2···O3	0.86 (3)	1.99 (3)	2.634 (3)	131 (3)

Symmetry code: (i) $-x+1, -y, -z+1$.

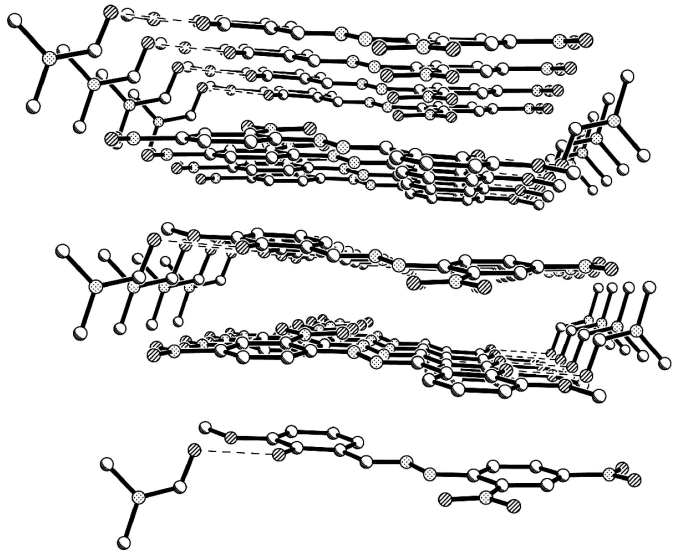


Figure 2
The crystal packing of (I), showing its layered structure. H atoms have been omitted for clarity. Dashed lines indicate hydrogen bonds.

Atom H2 (attached to N2) was located in a difference Fourier map and refined freely. All other H atoms were also located in a difference Fourier map and were subsequently refined in a riding model, with C—H distances in the range 0.93–0.96 Å and an O—H distance of 0.82 Å, and with $U_{iso}(H) = 1.2U_{eq}(C)$ or $1.5U_{eq}(O)$.

Data collection: *SMART* (Bruker, 1999); cell refinement: *SMART*; data reduction: *SAINT* (Bruker, 1999); program(s) used to solve structure: *SHELXS97* (Sheldrick, 1997); program(s) used to refine structure: *SHELXL97* (Sheldrick, 1997); molecular graphics: *SHELXTL* (Bruker, 1999); software used to prepare material for publication: *WinGX* (Farrugia, 1999).

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