Acta Crystallographica Section E
Structure Reports Online
ISSN 1600-5368

N′-(4-Hydroxy-3-methoxybenzylidene)-2-methoxybenzohydrazide ethanol hemisolvate

Zuo-Liang Jing,* Yan-Li Zhao, Xin Chen and Ming Yu

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

Correspondence e-mail: jzl74@tust.edu.cn

Key indicators

Single-crystal X-ray study
T = 294 K
Mean σ(C–C) = 0.003 Å
Disorder in solvent or counterion
R factor = 0.047
wR factor = 0.148
Data-to-parameter ratio = 12.1

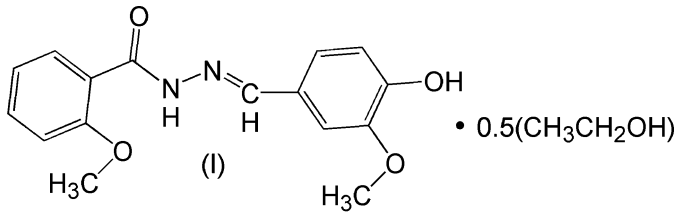
For details of how these key indicators were automatically derived from the article, see http://journals.iucr.org/e.

Received 4 August 2006
Accepted 18 August 2006

In the title compound, $C_{16}H_{16}N_2O_4\cdot0.5CH_3CH_2OH$, the Schiff base is approximately planar. An intramolecular N—H···O hydrogen bond stabilizes the molecular structure. The molecules are linked by O—H···O hydrogen bonds to form a chain along the *b* axis.

Comment

Metal complexes involving Schiff bases have attracted much attention because they can be utilized as model compounds of active centres in various proteins and enzymes (Kahwa *et al.*, 1986; Santos *et al.*, 2001). As part of an investigation of the coordination properties of Schiff bases functioning as ligands, we report here the synthesis and crystal structure of the title compound, (I).



(I)

The asymmetric unit of (I) comprises one 2-methoxybenzoic acid (4-hydroxy-3-methoxy-benzylidene)hydrazide molecule, and an ethanol solvent molecule with an occupancy factor of 0.5 (2) (Fig. 1). The Schiff base is approximately planar; the central chromophore (C8–C15/N1/N2/O3/O4) and the 4-hydroxy-3-methoxybenzaldehyde moiety (C1–C7/O1/O2/C16) are each planar, with r.m.s. deviations of 0.033 and 0.020 Å, respectively. The dihedral angle between these planes is 7.85 (6)°.

An intramolecular N—H···O hydrogen bond stabilizes the molecular structure, while intermolecular O—H···O hydrogen bonds stabilize the crystal structure (Table 1 and Fig. 2). Screw-related Schiff base molecules are linked *via* O—H···O hydrogen bonds involving the carbonyl and hydroxyl groups to form zigzag chains along the *b* axis.

Experimental

An anhydrous ethanol solution (50 ml) of 4-hydroxy-3-methoxybenzaldehyde (1.52 g, 10 mmol) was added to an anhydrous ethanol solution (50 ml) of 2-methoxybenzoic acid hydrazide (1.66 g, 10 mmol), and the mixture was stirred at 330 K for 6 h under N_2, whereupon a yellow precipitate appeared. The product was isolated, recrystallized from anhydrous ethanol and then dried *in vacuo* to give

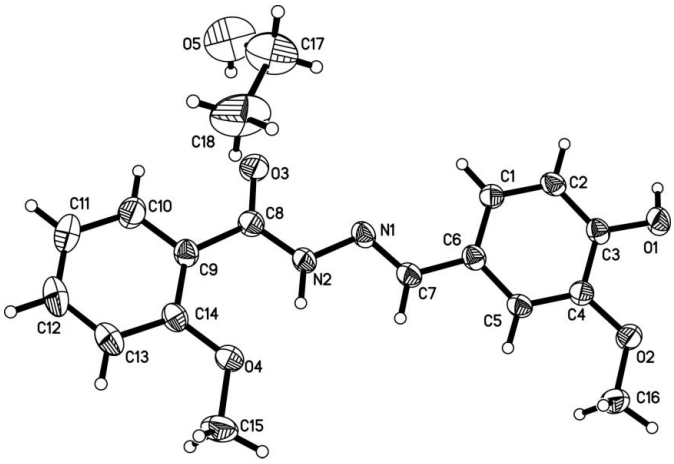


Figure 1
The structure of (I). Displacement ellipsoids are drawn at the 30% probability level. Only one disorder component is shown.

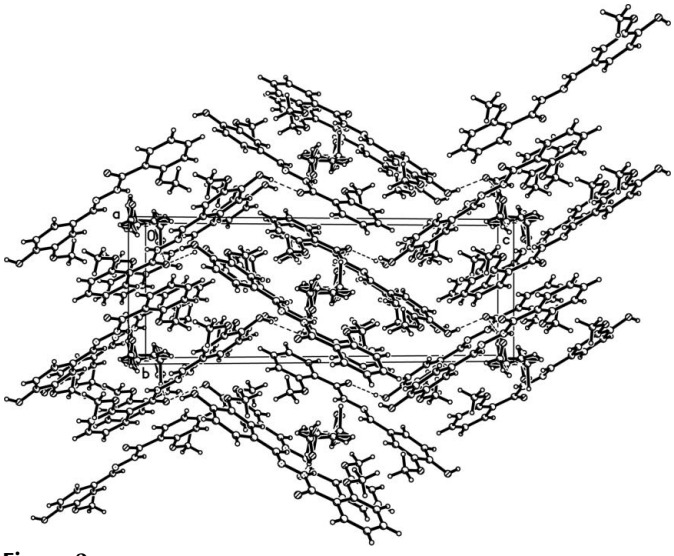


Figure 2
The crystal packing of (I), viewed down the *a* axis. Hydrogen bonds are indicated by dashed lines. Both components of the disordered ethanol molecules are shown.

pure compound (I) in 92% yield. Yellow single crystals of (I) suitable for X-ray analysis were obtained by slow evaporation of an anhydrous ethanol solution.

Crystal data

$C_{16}H_{16}N_2O_4{\cdot}0.5C_2H_6O$
M_r = 323.34
Monoclinic, $P2_1/c$
a = 8.764 (1) Å
b = 8.2705 (9) Å
c = 22.348 (3) Å
β = 99.784 (2)°
V = 1596.3 (3) Å^3
Z = 4
D_x = 1.345 Mg m^{-3}
Mo $K\alpha$ radiation
μ = 0.10 mm^{-1}
T = 294 (2) K
Block, yellow
0.22 × 0.20 × 0.16 mm

Data collection

Bruker SMART CCD area-detector diffractometer
φ and ω scans
Absorption correction: none
8355 measured reflections
2823 independent reflections
2222 reflections with $I > 2\sigma(I)$
R_{int} = 0.014
θ_{max} = 25.0°

Refinement

Refinement on F^2
$R[F^2 > 2\sigma(F^2)]$ = 0.047
$wR(F^2)$ = 0.148
S = 1.07
2823 reflections
234 parameters
H atoms treated by a mixture of independent and constrained refinement
$w = 1/[\sigma^2(F_o^2) + (0.0788P)^2 + 0.4458P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max}$ = 0.001
$\Delta\rho_{max}$ = 0.39 e Å^{-3}
$\Delta\rho_{min}$ = −0.28 e Å^{-3}

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

D—H···A	D—H	H···A	D···A	D—H···A
O1—H1···O3^{i}	0.88 (3)	1.90 (3)	2.704 (2)	152 (3)
N2—H2···O4	0.90 (3)	1.93 (3)	2.635 (2)	134 (2)
O5—H5A···O3	0.85	2.36	3.185 (7)	165

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

The ethanol molecule is disordered across a crystallographic inversion centre and it was refined with an occupancy of 0.5 (2), and with the C17—C18, O5—C17 and O5···C18 distances restrained to 1.54 (2), 2.54 (11) and 2.45 (2) Å, respectively. The U^{ij} components of atoms O5, C17 and C18 were approximated to isotropic behaviour. H atoms attached to N and O atoms of the main molecule were located in a difference Fourier map and refined freely (Table 1). The remaining H atoms were included in calculated positions [O—H = 0.85 Å and C—H = 0.93 (aromatic) or 0.96 Å (methyl)] and refined using a riding-model approximation, with U_{iso}(H) = 1.2U_{eq}(C) or 1.5U_{eq}(methyl C or O).

Data collection: *SMART* (Bruker, 1999); cell refinement: *SAINT* (Bruker, 1999); data reduction: *SAINT*; 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: *SHELXTL*.

References

Bruker (1997). *SHELXTL*. Version 5.10. Bruker AXS Inc., Madison, Wisconsin, USA.
Bruker (1999). *SMART* (Version 5.0) and *SAINT* (Version 4.0). Bruker AXS Inc., Madison, Wisconsin, USA.
Kahwa, I. A., Selbin, J., Hsieh, T. C.-Y. & Laine, R. A. (1986). *Inorg. Chim. Acta*, **118**, 179–185.
Santos, M. L. P., Bagatin, I. A., Pereira, E. M. & Ferreira, A. M. D. C. (2001). *J. Chem. Soc. Dalton Trans.* pp. 838–844.
Sheldrick, G. M. (1997). *SHELXS97* and *SHELXL97*. University of Göttingen, Germany.