Acta Crystallographica Section E
Structure Reports Online
ISSN 1600-5368

(*E*)-2-Methoxy-*N*′-[4-methoxy-3-(4-methylphenylsulfonyloxy)benzylidene]benzohydrazide ethanol solvate hemihydrate

Xin Chen* and Ming Yu

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

Correspondence e-mail: chen_xin9999@163.com

Received 26 October 2006
Accepted 6 November 2006

In the title compound, $C_{23}H_{22}N_2O_6S\cdot C_2H_6O\cdot 0.5H_2O$, the isovanillin group makes dihedral angles of 50.57 (8) and 8.89 (11)°, respectively, with the methyl- and methoxy-substituted benzene rings. Intramolecular N—H···O hydrogen bonds help to stabilize the molecular conformation, while intermolecular O—H···O and C—H···O hydrogen bonds link adjacent molecules, forming an infinite network.

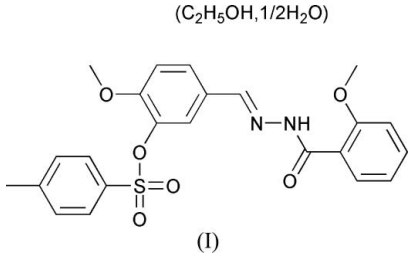
Key indicators

Single-crystal X-ray study
T = 294 K
Mean σ(C–C) = 0.004 Å
Disorder in solvent or counterion
R factor = 0.060
wR factor = 0.189
Data-to-parameter ratio = 13.7

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

Comment

Metal complexes based on Schiff bases have attracted much attention because of their biological activity (Kahwa *et al.*, 1986; Klayman *et al.*, 1979). Many Schiff base derivatives have been synthesized and employed to develop protein and enzyme mimics (Santos *et al.*, 2001), such as models to mimic hydrolase in the hydrolysis of *p*-nitrophenyl picolinate (Li *et al.*, 2005). Structural information is useful when investigating the coordination properties of Schiff bases functioning as ligands. We report here the synthesis and molecular structure of the title Schiff base compound, (I) (Fig. 1).



(I)

In (I), bond lengths and angles are within normal ranges (Allen *et al.*, 1987). The isovanillin group (C8–C13/C15/O3/O5) is nearly planar, with an r.m.s. deviation for fitted atoms of 0.0378 Å. This plane makes dihedral angles of 50.57 (8) and 8.89 (11)°, respectively, with the C1–C6 and C16–C21 benzene rings. The dihedral angle between these two benzene rings is 53.47 (10)°.

An intramolecular hydrogen bond links the NH group to O6, thereby influencing the molecular conformation. There are two O—H···O intermolecular hydrogen bonds and one weak non-classical intermolecular C—H···O hydrogen bond linking the main molecule and the solvent molecules (Table 1 and Fig. 2). These hydrogen bonds link molecules into an infinite network.

Experimental

An anhydrous ethanol solution (50 ml) of 5-formyl-2-methoxyphenyl 4-methylbenzenesulfonate (3.06 g, 10 mmol) was added to an anhy-

drous ethanol solution (50 ml) of 2-methoxybenzohydrazide (1.66 g, 10 mmol) and the mixture stirred at 350 K for 3 h under N_2, giving a white precipitate. The product was isolated, recrystallized from ethanol, and then dried in a vacuum to give pure compound (I) in 81% yield. Colourless single crystals of (I) suitable for X-ray analysis were obtained by slow evaporation of a 95% ethanol–water solution.

Crystal data

$C_{23}H_{22}N_2O_6S\cdot C_2H_6O\cdot 0.5H_2O$
$M_r = 509.57$
Triclinic, $P\overline{1}$
$a = 7.9106\ (16)$ Å
$b = 8.6824\ (17)$ Å
$c = 19.266\ (4)$ Å
$\alpha = 87.61\ (3)^\circ$
$\beta = 80.12\ (3)^\circ$
$\gamma = 81.39\ (3)^\circ$
$V = 1288.8\ (5)$ Å^3
$Z = 2$
$D_x = 1.313$ Mg m^{-3}
Mo $K\alpha$ radiation
$\mu = 0.17$ mm^{-1}
$T = 294\ (2)$ K
Block, colourless
0.14 × 0.12 × 0.10 mm

Data collection

Bruker SMART APEX CCD area-detector diffractometer
φ and ω scans
Absorption correction: multi-scan (*SADABS*; Sheldrick, 1996)
$T_{min} = 0.954$, $T_{max} = 0.983$
7839 measured reflections
4492 independent reflections
3294 reflections with $I > 2\sigma(I)$
$R_{int} = 0.027$
$\theta_{max} = 25.0^\circ$

Refinement

Refinement on F^2
$R[F^2 > 2\sigma(F^2)] = 0.060$
$wR(F^2) = 0.189$
$S = 1.07$
4492 reflections
329 parameters
H-atom parameters constrained
$w = 1/[\sigma^2(F_o^2) + (0.1233P)^2 + 0.0157P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} = 0.020$
$\Delta\rho_{max} = 0.40$ e Å^{-3}
$\Delta\rho_{min} = -0.30$ e Å^{-3}

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

D—H···A	D—H	H···A	D···A	D—H···A
N2—H2···O6	0.86	1.97	2.641 (3)	134
O8—H8B···O4^{i}	0.87	2.37	2.938 (11)	124
O7—H7···O4^{i}	0.82	2.39	2.814 (4)	113
C15—H15···O8^{ii}	0.93	2.49	3.414 (8)	172
C14—H14C···O1^{iii}	0.96	2.42	3.223 (4)	141

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

The H atoms were included in calculated positions and refined using a riding-model approximation. Constrained C—H and N—H bond lengths and isotropic U parameters: 0.93 Å and U_{iso}(H) = $1.2U_{eq}$(C) for Csp^2, 0.97 Å and U_{iso}(H) = $1.2U_{eq}$(C) for methylene, 0.96 Å and U_{iso}(H) = $1.5U_{eq}$(C) for methyl, 0.85 Å and U_{iso}(H) = $1.2U_{eq}$(O) for water, 0.82 Å and U_{iso}(H) = $1.5U_{eq}$(O) for hydroxy, and 0.86 Å and U_{iso}(H) = $1.2U_{eq}$(N) for imino H atoms. The water molecule (O8, H8*A* and H8*B*) was refined using a disorder model, with occupancy factors constrained to be 0.50.

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

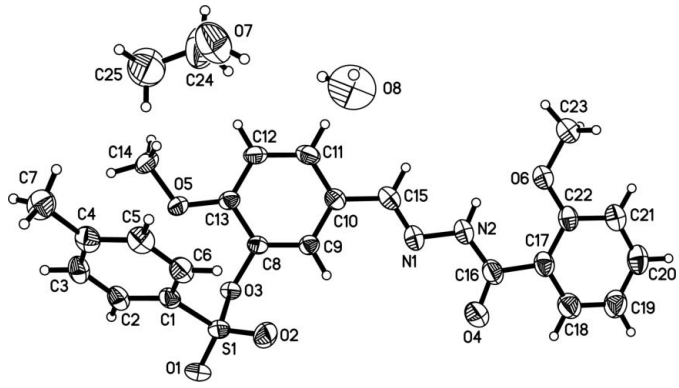


Figure 1
The asymmetric unit of (I), with displacement ellipsoids for non-H atoms drawn at the 30% probability level.

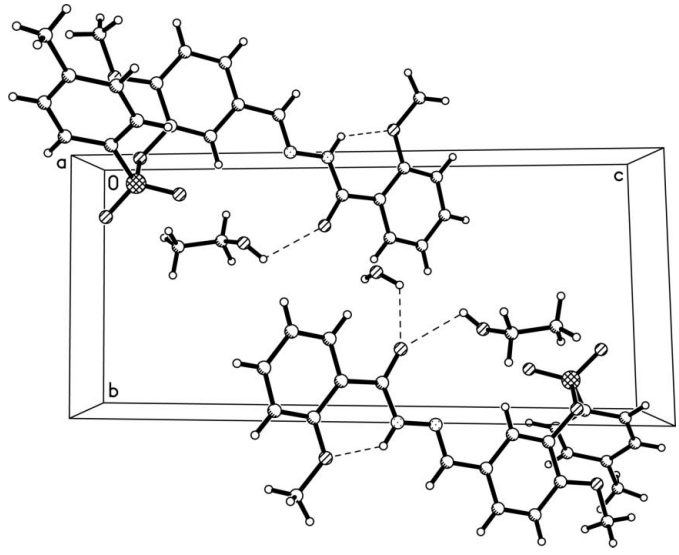


Figure 2
A packing diagram for (I), with hydrogen bonds shown as dashed lines.

References

Allen, F. H., Kennard, O., Watson, D. G., Brammer, L., Orpen, A. G. & Taylor, R. (1987). *J. Chem. Soc. Perkin Trans. 2*, pp. S1–19.
Bruker (1999). *SMART* (Version 5.0) and *SAINT* (Version 4.0) for Windows NT. 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.
Klayman, D. L., Bartosevich, J. F., Griffin, T. S., Mason, C. J. & Scovill, J. P. (1979). *J. Med. Chem.* **22**, 855–862.
Li, J.-Z., Xu, B., Li, S.-X., Zeng, W. & Qin, S.-Y. (2005). *Transition Met. Chem.* **30**, 669–676.
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. (1996). *SADABS*. University of Göttingen, Germany.
Sheldrick, G. M. (1997*a*). *SHELXS97* and *SHELXL97*. University of Göttingen, Germany.
Sheldrick, G. M. (1997*b*). *SHELXTL*. Version 5.10. Bruker AXS Inc., Madison, Wisconsin, USA.