Acta Crystallographica Section E
Structure Reports Online
ISSN 1600-5368

(*E*)-1-(4-Ethoxy-3-methoxybenzylidene)-2-(4-nitrophenyl)hydrazine

Jun Shi‡

Department of Basic Course, Tianjin Agricultural College, Tianjin 300384, People's Republic of China

‡ Current address: School of Chemical Engineering and Technology Tianjin University Tianjin 300072 People's Republic of China

Correspondence e-mail: shi_jun99@163.com

Received 16 November 2005
Accepted 24 November 2005
Online 14 December 2005

Key indicators

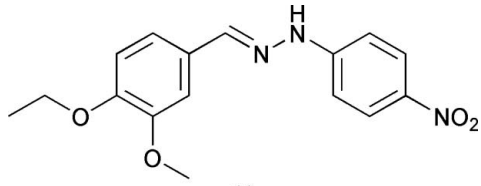
Single-crystal X-ray study
T = 294 K
Mean σ(C–C) = 0.008 Å
R factor = 0.051
wR factor = 0.146
Data-to-parameter ratio = 6.7

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

The title compound, $C_{16}H_{17}N_3O_4$, crystallizes with two molecules in the asymmetric unit. Each independent molecule is nearly planar. Two bifurcated intermolecular N—H···(O,O) hydrogen-bond systems help to consolidate the crystal packing.

Comment

Metal complexes based on Schiff bases have attracted much attention because of their biological activity (Kahwa *et al.*, 1986). Consequently, a large number of Schiff base derivatives have been synthesized and employed to develop protein and enzyme mimics, such as models to mimic hydrolase in the hydrolysis of *p*-nitrophenyl picolinate (Li *et al.*, 2005).



(I)

As part of an investigation of their crystal structures, which will provide useful information for the coordination properties of Schiff bases functioning as ligands, in the present study we report the synthesis and molecular structure of the nitrophenylhydrazine Schiff base derivative (I) (Fig. 1).

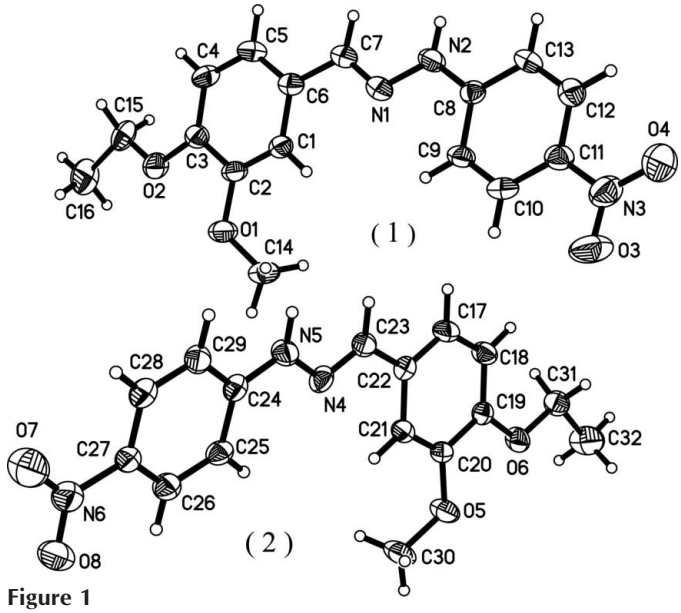


Figure 1
The structure of the asymmetric unit of (I), with displacement ellipsoids for non-H atoms drawn at the 30% probability level.

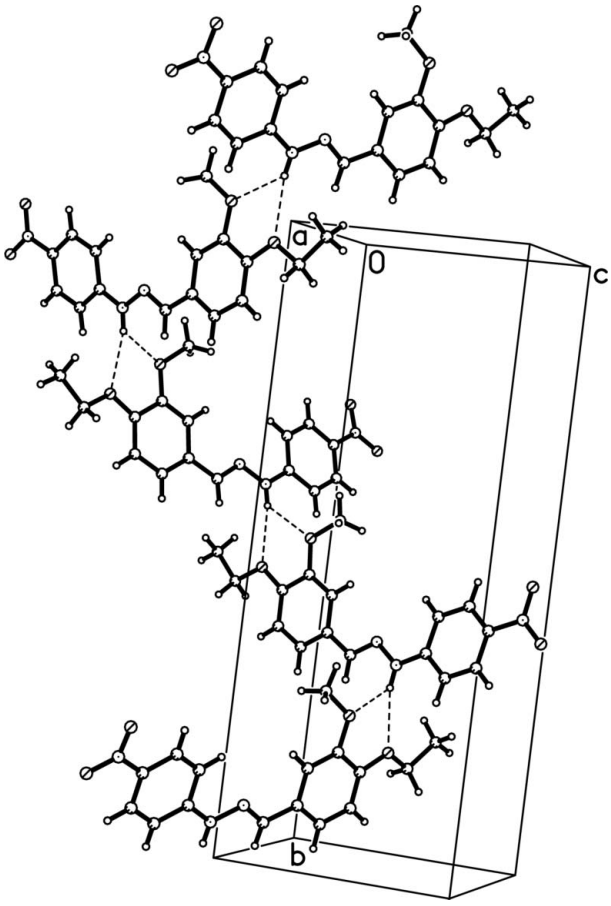


Figure 2
Packing diagram for (I), with H bonds drawn as dashed lines.

The asymmetric unit of (I) consists of two independent molecules, which are quite similar to each other. In molecule 1, the vanillin group (C1–C7/O1/O2) is planar, with an r.m.s. deviation, δ, from the mean plane of 0.029Å, and it makes a dihedral angle of 11.29 (18)° with the phenylhydrazine residue (C8–C13/N1/N2). In molecule 2, the vanillin group (C17–C23/O5/O6) is also planar, with δ = 0.027Å, and it makes a dihedral angle of 10.87 (13)° with the phenylhydrazine residue (C24–C29/N4/N5). The nitro group and its attached aromatic ring are not coplanar, with dihedral angles of 7.23 (9) (for O3/N3/O4) and 4.61 (6)° (for O7/N6/O8). Furthermore, the phenylhydrazine residues of the two independent molecules are almost perpendicular to each other, with a dihedral angle of 86.36 (9)°, while the dihedral angle between the two vanillin groups is 77.10 (9)°. All bond lengths and angles are within normal ranges (Allen *et al.*, 1987).

Two bifurcated intermolecular N—H···(O,O) hydrogen-bond systems are found in (I) (Table 1 and Fig. 2), which help to consolidate the crystal packing. The hydrogen bonds link adjacent molecules, forming infinite chains.

Experimental

An anhydrous ethanol solution of 4-ethoxy-3-methoxybenzaldehyde (1.80 g, 10 mmol) was added to an anhydrous ethanol solution of 1-(4-nitrophenyl)hydrazine (1.53 g, 10 mmol) and the mixture stirred at 350 K for 5 h under nitrogen. A yellow precipitate appeared which was isolated, recrystallized from acetonitrile, and then dried in a vacuum to give the pure compound in 83% yield. Yellow single crystals of (I) suitable for X-ray analysis were obtained by slow evaporation of an acetonitrile solution.

Crystal data

$C_{16}H_{17}N_3O_4$
M_r = 315.33
Monoclinic, $P2_1$
a = 7.589 (2) Å
b = 23.491 (7) Å
c = 8.929 (2) Å
β = 96.287 (5)°
V = 1582.2 (7) Å^3
Z = 4
D_x = 1.324 Mg m^{-3}
Mo $K\alpha$ radiation
Cell parameters from 1909 reflections
θ = 2.8–22.1°
μ = 0.10 mm^{-1}
T = 294 (2) K
Block, yellow
0.22 × 0.20 × 0.18 mm

Data collection

Bruker SMART APEX CCD area-detector diffractometer
φ and ω scans
Absorption correction: multi-scan (*SADABS*; Sheldrick, 1996)
T_{min} = 0.962, T_{max} = 0.983
7919 measured reflections
2820 independent reflections
1741 reflections with $I > 2\sigma(I)$
R_{int} = 0.044
θ_{max} = 25.0°
$h = -8 \rightarrow 6$
$k = -27 \rightarrow 27$
$l = -10 \rightarrow 10$

Refinement

Refinement on F^2
$R[F^2 > 2\sigma(F^2)]$ = 0.051
$wR(F^2)$ = 0.146
S = 1.14
2820 reflections
419 parameters
H-atom parameters constrained
$w = 1/[\sigma^2(F_o^2) + (0.072P)^2]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max}$ = 0.021
$\Delta\rho_{max}$ = 0.31 e Å^{-3}
$\Delta\rho_{min}$ = −0.21 e Å^{-3}

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

D—H···A	D—H	H···A	D···A	D—H···A
N2—H2···O5^{i}	0.86	2.39	3.042 (6)	133
N2—H2···O6^{i}	0.86	2.36	3.179 (6)	160
N5—H5···O1^{ii}	0.86	2.37	2.949 (6)	125
N5—H5···O2^{ii}	0.86	2.36	3.198 (7)	166

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

The H atoms were included in calculated positions and refined using a riding-model approximation. Constrained C—H and N—H bond lengths and U_{iso}(H) parameters: 0.93Å and U_{iso}(H) = 1.2U_{eq}(C) for aromatic CH; 0.97Å and U_{iso}(H) = 1.2U_{eq}(C) for methylene CH_2; 0.96Å and U_{iso}(H) = 1.5U_{eq}(C) for methyl CH_3; 0.86Å and U_{iso}(H) = 1.2U_{eq}(N) for imino NH. In the absence of significant anomalous dispersion effects, Friedel pairs were averaged.

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*.

The author is very much indebted to Professor Dr Jian-Rong Han of Hebei University of Science & Technology for helpful discussions on the crystal structure.

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.
Li, J.-Z., Xu, B., Li, S.-X., Zeng, W. & Qin, S.-Y. (2005). *Transition Met. Chem.* **30**, 669–676.
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*). *SHELXTL97*. Version 5.10 for Windows NT. Bruker AXS Inc., Madison, Wisconsin, USA.