

**(E)-1-[2-(2-Chlorobenzoyloxy)benzylidene]-
2-(2,4-dinitrophenyl)hydrazine N,N-dimethyl-
formamide solvate****Jun Shi†**Department of Basic Course, Tianjin Agricultural
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Key indicatorsSingle-crystal X-ray study
 $T = 294$ K
Mean $\sigma(\text{C}-\text{C}) = 0.004$ Å
Disorder in solvent or counterion
 R factor = 0.043
 wR factor = 0.146
Data-to-parameter ratio = 11.3For details of how these key indicators were
automatically derived from the article, see
<http://journals.iucr.org/e>.

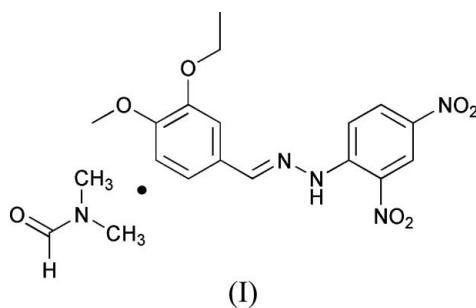
In the title compound, $\text{C}_{16}\text{H}_{16}\text{N}_4\text{O}_6 \cdot \text{C}_3\text{H}_7\text{NO}$, the isovanillin group makes a dihedral angle of $2.18(13)^\circ$ with the phenylhydrazine residue. The crystal packing is stabilized by bifurcated $\text{N}-\text{H} \cdots (\text{O},\text{O})$ intra- and intermolecular hydrogen bonds, which form inversion-related dimers, together with intermolecular $\text{C}-\text{H} \cdots \text{O}$ hydrogen bonds.

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Comment

Metal complexes based on Schiff bases have attracted much attention in biology and chemistry (Kahwa *et al.*, 1986). Research in this area has been stimulated by catalysis, design of molecular magnets, materials chemistry, and biological modelling applications such as protein and enzyme mimics (Larson & Pecoraro, 1991; Santos *et al.*, 2001). As part of an investigation of the crystal structures of such compounds, which will provide useful information on the coordination properties of Schiff bases functioning as ligands, in the present study we report the synthesis and crystal structure of the title dinitrophenylhydrazine Schiff base derivative, (I) (Fig. 1).



In the molecule of (I), the phenylhydrazine residue (C11–C16/N1/N2) is almost planar, with an r.m.s. deviation for the fitted atoms of 0.0284 Å. The isovanillin group (C1–C6/C10/O1/O2) is also planar, the r.m.s. deviation being 0.0075 Å. The dihedral angle between these two planes is $2.18(13)^\circ$. The nitro groups and their attached aromatic ring are not coplanar; the C11–C16 plane makes dihedral angles of $5.0(5)$ and $5.2(5)^\circ$ with the O3/N3/O4 and O5/N4/O6 planes, respectively. All bond lengths and angles are within normal ranges (Allen *et al.*, 1987).

A bifurcated $\text{N}-\text{H}(\text{O},\text{O})$ intra- and intermolecular $\text{N}-\text{H} \cdots \text{O}$ hydrogen-bond system is found in (I) (Table 1). The intramolecular bond stabilizes the conformation of the molecule, while the intermolecular bond forms centrosymmetric dimers (Fig. 2). An $\text{O}3 \cdots \text{O}3(1-x, 2-y, -z)$ short contact of $2.630(3)$ Å is observed in the dimer. The main molecule and the solvent molecule are linked by $\text{C}-\text{H} \cdots \text{O}$ hydrogen bonds.

Experimental

An anhydrous ethanol solution (50 ml) of 3-ethoxy-4-methoxybenzaldehyde (1.80 g, 10 mmol) was added to an anhydrous ethanol solution (50 ml) of 1-(2,4-dinitrophenyl)hydrazine (1.98 g, 10 mmol), and the mixture was stirred at 350 K for 5 h under nitrogen. A red precipitate appeared, and this was isolated, recrystallized from acetonitrile and then dried in a vacuum, to give the pure compound in 85% yield. Red single crystals of (I) suitable for X-ray analysis were obtained by slow evaporation of an *N,N*-dimethylformamide solution.

Crystal data

$C_{16}H_{16}N_4O_6 \cdot C_3H_7NO$	$Z = 4$
$M_r = 433.42$	$D_x = 1.374 \text{ Mg m}^{-3}$
Monoclinic, $P2_1/c$	Mo $K\alpha$ radiation
$a = 10.306 (5) \text{ \AA}$	$\mu = 0.11 \text{ mm}^{-1}$
$b = 9.612 (4) \text{ \AA}$	$T = 294 (2) \text{ K}$
$c = 21.152 (10) \text{ \AA}$	Block, red
$\beta = 90.377 (9)^\circ$	$0.28 \times 0.20 \times 0.12 \text{ mm}$
$V = 2095.3 (17) \text{ \AA}^3$	

Data collection

Bruker SMART APEX CCD area-detector diffractometer	10354 measured reflections
φ and ω scans	3690 independent reflections
Absorption correction: multi-scan (SADABS; Sheldrick, 1996)	1991 reflections with $I > 2\sigma(I)$
$T_{\min} = 0.948, T_{\max} = 0.987$	$R_{\text{int}} = 0.048$
	$\theta_{\text{max}} = 25.0^\circ$

Refinement

Refinement on F^2	$w = 1/[\sigma^2(F_o^2) + (0.0639P)^2 + 1.3187P]$
$R[F^2 > 2\sigma(F^2)] = 0.043$	where $P = (F_o^2 + 2F_c^2)/3$
$wR(F^2) = 0.146$	$(\Delta/\sigma)_{\text{max}} = 0.002$
$S = 0.89$	$\Delta\rho_{\text{max}} = 0.18 \text{ e \AA}^{-3}$
3690 reflections	$\Delta\rho_{\text{min}} = -0.16 \text{ e \AA}^{-3}$
327 parameters	Extinction correction: SHELXL97 (Sheldrick, 1997a)
H-atom parameters constrained	Extinction coefficient: 0.0111 (14)

Table 1

Hydrogen-bond geometry ($\text{\AA}, ^\circ$).

$D-H \cdots A$	$D-H$	$H \cdots A$	$D \cdots A$	$D-H \cdots A$
$N2-H2A \cdots O3^i$	0.86	2.01	2.613 (3)	127
$N2-H2A \cdots O3^i$	0.86	2.40	3.226 (3)	160
$C19-H19C \cdots O4^{ii}$	0.96	2.39	3.332 (6)	167
$C15-H15 \cdots O5^{iii}$	0.93	2.43	3.311 (4)	158

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

The solvent molecule is disordered over two positions, with occupancy factors of 0.815 (7) and 0.185 (7). The $C=O, N-C_{sp^2}$ and $N-C_{sp^3}$ distances involving the disordered atoms were restrained to 1.33 (1), 1.45 (1) and 1.22 (1) \AA , respectively, and the U^{ij} components were restrained to an approximate isotropic behaviour. H atoms were included in calculated positions and refined using a riding-model approximation, with $C-H = 0.93 \text{ \AA}$ and $U_{\text{iso}}(H) = 1.2U_{\text{eq}}(C)$ for aromatic CH, $C-H = 0.97 \text{ \AA}$ and $U_{\text{iso}}(H) = 1.2U_{\text{eq}}(C)$ for methylene CH_2 , $C-H = 0.96 \text{ \AA}$ and $U_{\text{iso}}(H) = 1.5U_{\text{eq}}(C)$ for methyl CH_3 , and $N-H = 0.86 \text{ \AA}$ and $U_{\text{iso}}(H) = 1.2U_{\text{eq}}(N)$ for imino NH.

Data collection: SMART (Bruker, 1999); cell refinement: SAINT (Bruker, 1999); data reduction: SAINT; program(s) used to solve structure: SHELXS97 (Sheldrick, 1997a); program(s) used to refine

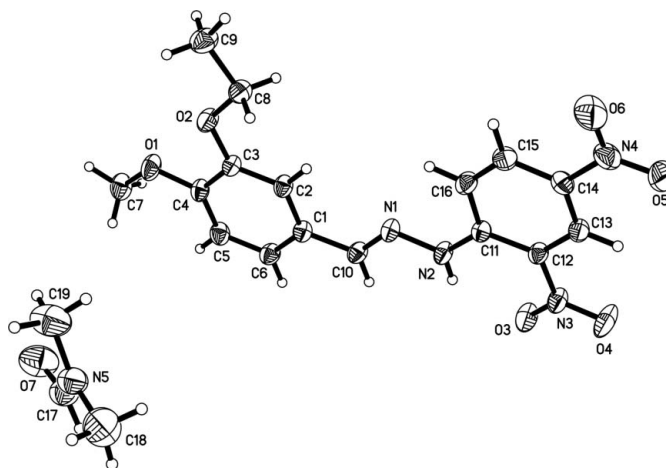


Figure 1

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

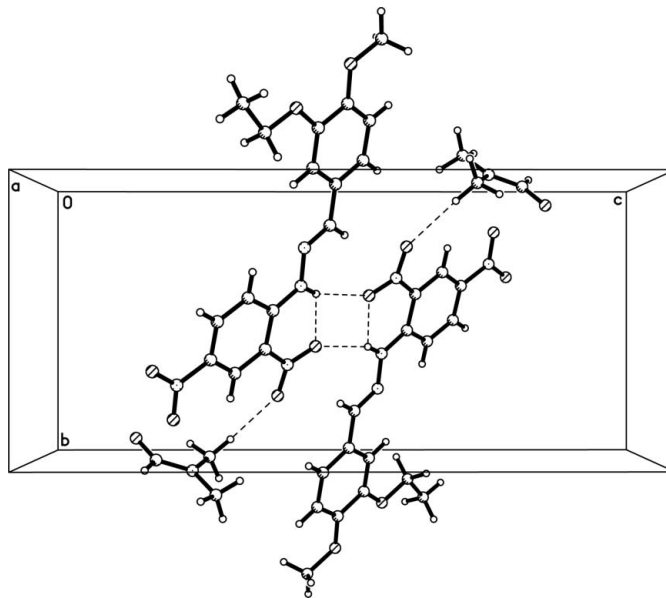


Figure 2

A partial packing diagram for (I), with hydrogen bonds drawn as dashed lines.

structure: SHELXL97 (Sheldrick, 1997a); molecular graphics: SHELXTL (Sheldrick, 1997b); software used to prepare material for publication: SHELXTL.

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