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(*E*)-1-[2-(Benzyloxy)-3-methoxybenzylidene]-2-(2,4-dinitrophenyl)hydrazine dimethylformamide solvate

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Key indicators

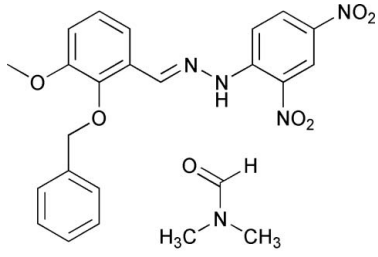
Single-crystal X-ray study
T = 294 K
Mean σ(C–C) = 0.005 Å
R factor = 0.051
wR factor = 0.140
Data-to-parameter ratio = 13.1

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

In the title compound, $C_{21}H_{18}N_4O_6\cdot C_3H_7NO$, the substituted *o*-vanillin group makes dihedral angles of 37.17 (11) and 5.68 (8)°, respectively, with the phenyl ring and the phenylhydrazine mean plane. The packing is stabilized by an intramolecular N—H···O hydrogen bond and a weak non-classical intermolecular C—H···O hydrogen bond.

Comment

Schiff base ligands have played an important role in the development of coordination chemistry since the late 19th century (Santos *et al.*, 2001). Consequently, a large number of this species have been reported to be excellent reagents in biological, pharmacological, clinical and analytical applications (Wang *et al.*, 2005; Yang *et al.*, 2005). As part of an investigation of their crystal structures, which will provide useful information for the coordination properties of Schiff bases functioning as ligands, we report here the synthesis and molecular structure of the title compound, (I).



(I)

All bond lengths and angles are within normal ranges (Allen *et al.*, 1987). The substituted *o*-vanillin group (atoms C8–C13/C15/O1/O2) is planar, with an r.m.s. deviation for fitted atoms of 0.0284 Å. The phenylhydrazine residue (C16–C21/N1/N2) is also planar, with an r.m.s. deviation for fitted atoms of 0.0292 Å. The dihedral angle between them is 5.68 (8)°. The phenyl ring (C1–C6) makes dihedral angles of 42.75 (11) and 37.17 (11)°, respectively, with the phenylhydrazine residue (C16–C21/N1/N2) and the substituted *o*-vanillin group (C8–C13/C15/O1/O2).

An intramolecular N2—H2···O6 hydrogen bond is found in (I) (Table 1), which helps to stabilize the conformation of the molecule. There is also a weak C15—H15···O7 hydrogen bond linking the main molecule and the solvent molecule (Fig. 2).

Experimental

An anhydrous ethanol solution (50 ml) of 2-benzyloxy-3-methoxybenzaldehyde (2.42 g, 10 mmol) was added to an anhydrous ethanol

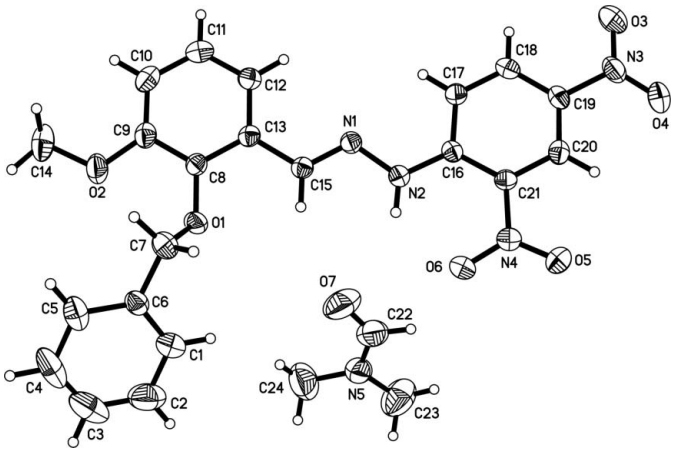


Figure 1
The molecular structure of (I), with displacement ellipsoids for non-H atoms drawn at the 30% probability level.

solution (100 ml) of 1-(2,4-dinitrophenyl)hydrazine (1.98 g, 10 mmol) and the mixture refluxed for 5 h under nitrogen, giving a red precipitate. The product was isolated, recrystallized from acetonitrile, and then dried in a vacuum to give the pure compound in 81% yield. Red single crystals of (I) suitable for X-ray analysis were obtained by slow evaporation of a dimethylformamide solution.

Crystal data

$C_{21}H_{18}N_4O_6\cdot C_3H_7NO$
M_r = 495.49
Triclinic, $P\overline{1}$
a = 8.244 (3) Å
b = 11.792 (4) Å
c = 12.789 (4) Å
α = 86.261 (7)°
β = 87.312 (6)°
γ = 86.856 (6)°
V = 1237.6 (7) Å^3
Z = 2
D_x = 1.330 Mg m^{-3}
Mo $K\alpha$ radiation
μ = 0.10 mm^{-1}
T = 294 (2) K
Block, red
0.25 × 0.20 × 0.12 mm

Data collection

Bruker SMART APEX CCD area-detector diffractometer
φ and ω scans
Absorption correction: multi-scan (*SADABS*; Sheldrick, 1996)
T_{min} = 0.949, T_{max} = 0.988
6206 measured reflections
4290 independent reflections
2133 reflections with $I > 2\sigma(I)$
R_{int} = 0.025
θ_{max} = 25.0°

Refinement

Refinement on F^2
$R[F^2 > 2\sigma(F^2)]$ = 0.051
$wR(F^2)$ = 0.140
S = 1.03
4290 reflections
328 parameters
H-atom parameters constrained
$w = 1/[\sigma^2(F_o^2) + (0.0526P)^2 + 0.1421P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max}$ = 0.003
$\Delta\rho_{max}$ = 0.15 e Å^{-3}
$\Delta\rho_{min}$ = −0.17 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	2.05	2.653 (3)	126
C15—H15···O7	0.93	2.58	3.369 (4)	143

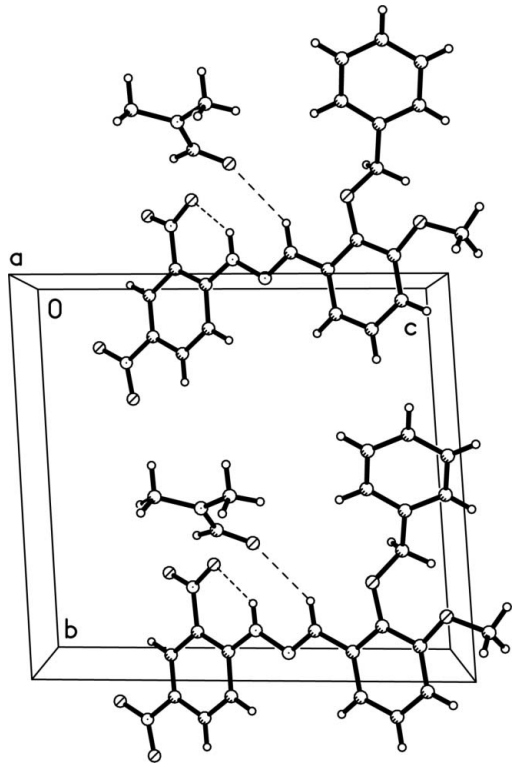


Figure 2
A packing diagram for (I), with hydrogen bonds drawn as dashed lines.

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: C—H = 0.93 Å and U_{iso}(H) = 1.2U_{eq}(C) for Csp^2, C—H = 0.97 Å and U_{iso}(H) = 1.2U_{eq}(C) for methylene, C—H = 0.96 Å and U_{iso}(H) = 1.5U_{eq}(C) for methyl, and N—H = 0.86 Å and U_{iso}(H) = 1.2U_{eq}(N) for NH H atoms.

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*.

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