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(*E*)-1-(5-Bromo-2-ethoxy-3-methoxybenzylidene)-2-(2,4-dinitrophenyl)hydrazine dimethylformamide solvate

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Key indicators

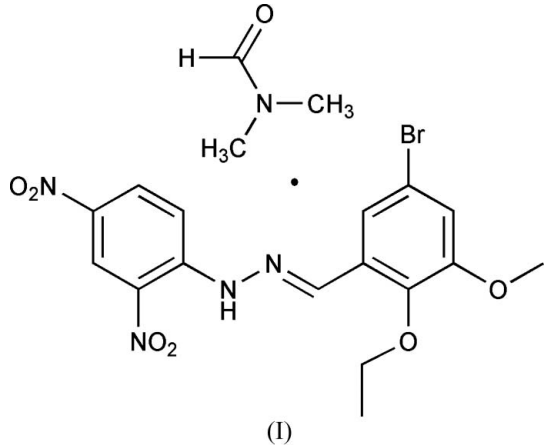
Single-crystal X-ray study
T = 294 K
Mean σ(C–C) = 0.005 Å
R factor = 0.040
wR factor = 0.104
Data-to-parameter ratio = 13.4

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

In the title compound, $C_{16}H_{15}BrN_4O_6\cdot C_3H_7NO$, the substituted vanillin group makes a dihedral angle of 7.58 (10)° with the nitrophenylhydrazine mean plane. A bifurcated intra/intermolecular N—H···(O,O) hydrogen bond and two C—H···O interactions help to establish the molecular conformation and consolidate the crystal packing.

Comment

Schiff base ligands have played an important role in the development of coordination chemistry since the late 19th century. Consequently, a large number of these species are reported to be superior reagents in biological, pharmacological, clinical and analytical applications (Santos *et al.*, 2001; Wang *et al.*, 2005; Yang *et al.*, 2005). As a continuation of these studies, we report here the synthesis and structure of the title compound, (I) (Fig. 1).



(I)

The bond lengths and angles for (I) are within their normal ranges (Allen *et al.*, 1987). The substituted vanillin group (C1–C6/C10/O1/O2/Br1) is essentially planar, with an r.m.s. deviation for fitted atoms of 0.0157 Å, The nitrophenylhydrazine system (C11–C16/O3/O4/N2/N3) is also planar (r.m.s. deviations for fitted atoms = 0.0299 Å). The dihedral angle between these two planes is 7.58 (10)°.

The N3/O3/O4 nitro group is almost coplanar with its attached benzene ring [torsion angles O3—N3—C12—C13 and O4—N3—C12—C11 of 176.5 (3) and −179.9 (3)°, respectively], while the other nitro group (N4/O5/O6) makes a dihedral angle of 6.79 (5)° with the ring.

A bifurcated N—H···(O,O) intra/intermolecular N—H···O hydrogen bond is found in (I) (Table 1). The intramolecular bond may stabilize the orientation of the N3/O3/O4 nitro group, while the intermolecular bond consolidates the

crystal structure. The crystal structure for (I) is also stabilized by weak non-classical intermolecular C—H···O═C and C—H···O═N bonds linking the main molecule and the solvent molecule.

Experimental

An anhydrous ethanol solution (50 ml) of 5-bromo-2-ethoxy-3-methoxybenzaldehyde (2.59 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 stirred at 350 K for 5 h under nitrogen, giving an orange precipitate. The product was then isolated, recrystallized from ethanol and then dried *in vacuo* to give the pure compound in 81% yield. Orange single crystals of (I) suitable for X-ray analysis were obtained by slow evaporation of a dimethylformamide solution.

Crystal data

$C_{16}H_{15}BrN_4O_6\cdot C_3H_7NO$
$M_r = 512.33$
Triclinic, $P\overline{1}$
$a = 8.521$ (2) Å
$b = 11.698$ (3) Å
$c = 13.135$ (4) Å
$\alpha = 64.889$ (4)°
$\beta = 73.484$ (4)°
$\gamma = 80.844$ (5)°
$V = 1135.5$ (5) Å^3
$Z = 2$
$D_x = 1.498$ Mg m^{-3}
Mo $K\alpha$ radiation
$\mu = 1.86$ mm^{-1}
$T = 294$ (2) K
Block, orange
0.30 × 0.20 × 0.16 mm

Data collection

Bruker SMART APEX CCD area-detector diffractometer
φ and ω scans
Absorption correction: multi-scan (*SADABS*; Sheldrick, 1996)
$T_{min} = 0.605$, $T_{max} = 0.743$
5768 measured reflections
3952 independent reflections
2660 reflections with $I > 2\sigma(I)$
$R_{int} = 0.024$
$\theta_{max} = 25.0°$

Refinement

Refinement on F^2
$R[F^2 > 2\sigma(F^2)] = 0.040$
$wR(F^2) = 0.104$
$S = 1.03$
3952 reflections
294 parameters
H-atom parameters constrained
$w = 1/[\sigma^2(F_o^2) + (0.0472P)^2 + 0.1537P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} < 0.001$
$\Delta\rho_{max} = 0.31$ e Å^{-3}
$\Delta\rho_{min} = -0.30$ e Å^{-3}
Extinction correction: *SHELXL97*
Extinction coefficient: 0.0242 (18)

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

D—H···A	D—H	H···A	D···A	D—H···A
N2—H2···O8^{i}	0.86	2.31	3.014 (4)	139
N2—H2···O3	0.86	2.05	2.643 (3)	125
C10—H10···O8^{i}	0.93	2.42	3.170 (4)	137
C17—H17···O3^{ii}	0.93	2.48	3.118 (6)	126

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

H atoms were included in calculated positions (N—H = 0.86 Å and C—H = 0.93–0.96 Å) and refined as riding, with U_{iso}(H) = $1.2U_{eq}$(C,*N*) or $1.5U_{eq}$(methyl C).

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*.

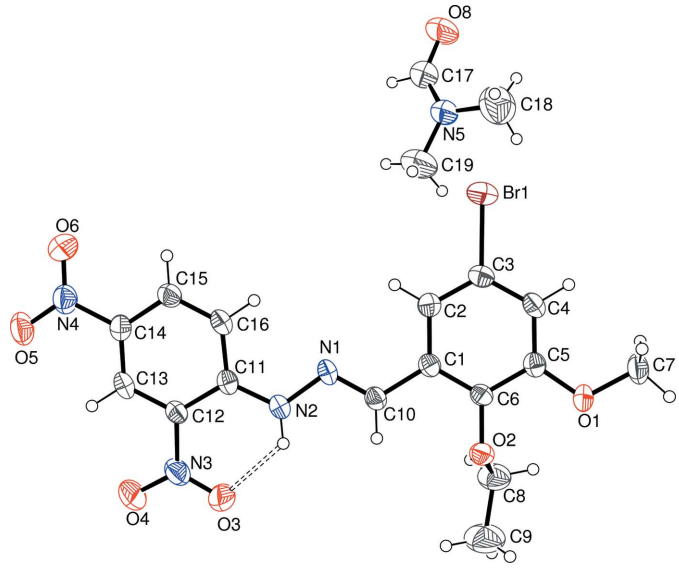


Figure 1
The structure of (I), with displacement ellipsoids for non-H atoms drawn at the 40% probability level. The intramolecular N—H···O hydrogen bond is shown as dashed lines.

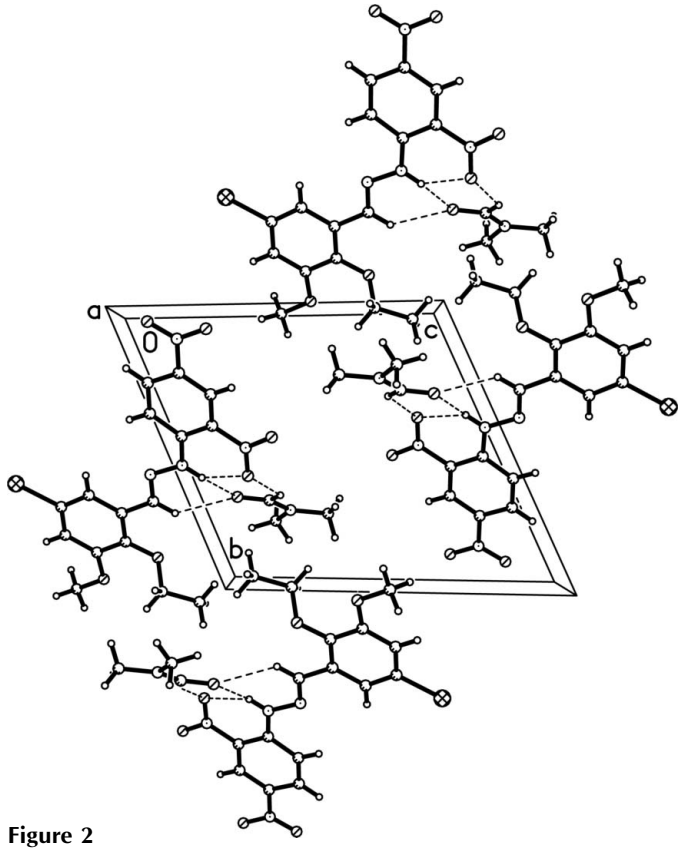


Figure 2
Packing diagram for (I), with hydrogen bonds shown as dashed lines.

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