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(*E*)-1-(2,4-Dinitrophenyl)-2-[4-methoxy-3-(4-methylbenzenesulfonyloxy)benzylidene]hydrazine

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In the title compound, $C_{21}H_{18}N_4O_8S$, the isovanillin group makes dihedral angles of 31.69 (9) and 3.67 (9)° with the terminal 4-methylbenzene ring and the phenylhydrazine mean plane, respectively. The crystal structure is stabilized by a bifurcated intramolecular/intermolecular N—H···O hydrogen-bond system and a weak non-classical intermolecular C—H···(O,O) hydrogen-bond contact.

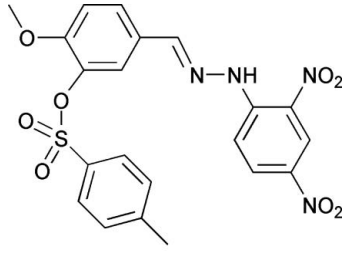
Key indicators

Single-crystal X-ray study
T = 294 K
Mean σ(C–C) = 0.005 Å
R factor = 0.049
wR factor = 0.131
Data-to-parameter ratio = 12.5

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). Consequently, a large number of 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). We report here the synthesis and molecular structure of the title Schiff base compound, (I) (Fig. 1)



(I)

All bond lengths and angles are within normal ranges (Allen *et al.*, 1987). The phenylhydrazine residue (C16–C21/N1/N2) is essentially planar, with an r.m.s. deviation for fitted atoms of 0.0126 Å. This plane makes dihedral angles of 28.67 (9) and 3.67 (9)° with the benzene ring (C1–C6) and the isovanillin group (C8–C13/C15/O1/O4), respectively. In addition, the dihedral angle between the benzene ring (C1–C6) and the isovanillin group (C8–C13/C15/O1/O4) is 31.69 (9)°. The two nitro groups (O5/N3/O6) and (O7/N4/O8) and their attached aromatic ring (C16–C21) are not coplanar. The dihedral angles are 5.28 (3)° for O5/N3/O6 and 7.36 (5)° for O7/N4/O8.

A bifurcated intramolecular/intermolecular N—H···(O,O) hydrogen-bond system is found in the crystal structure of (I) (Table 1). The intramolecular bond stabilizes the conformation of the molecule, while the intermolecular bond forms centrosymmetric dimers (Fig. 2). An O5···O5($-x+3, -y+1, -z$) short contact of 2.860 (3) Å is observed in the dimer. There is also a weak C—H···O interaction, linking adjacent molecules into a one-dimensional extended chain.

Experimental

An anhydrous ethanol solution (50 ml) of 5-formyl-2-methoxyphenyl 4-methylbenzenesulfonate (3.06 g, 10 mmol) was added to an anhydrous ethanol solution (100 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 a red precipitate. The product was isolated, recrystallized from acetonitrile, and then dried in a vacuum to give the pure compound in 87% yield. Red single crystals of (I) suitable for X-ray analysis were obtained by slow evaporation of a dimethylformamide solution.

Crystal data

$C_{21}H_{18}N_4O_8S$
M_r = 486.46
Triclinic, $P\overline{1}$
a = 7.365 (2) Å
b = 12.978 (4) Å
c = 13.547 (4) Å
α = 62.937 (4)°
β = 88.904 (5)°
γ = 74.845 (5)°
V = 1105.5 (6) Å^3
Z = 2
D_x = 1.461 Mg m^{-3}
Mo $K\alpha$ radiation
μ = 0.20 mm^{-1}
T = 294 (2) K
Block, red
0.28 × 0.24 × 0.20 mm

Data collection

Bruker SMART APEX CCD area-detector diffractometer
φ and ω scans
Absorption correction: multi-scan (*SADABS*; Sheldrick, 1996)
T_{min} = 0.928, T_{max} = 0.960
5658 measured reflections
3876 independent reflections
2342 reflections with $I > 2\sigma(I)$
R_{int} = 0.030
θ_{max} = 25.0°

Refinement

Refinement on F^2
$R[F^2 > 2\sigma(F^2)]$ = 0.049
$wR(F^2)$ = 0.131
S = 1.03
3876 reflections
309 parameters
H-atom parameters constrained
$w = 1/[\sigma^2(F_o^2) + (0.0602P)^2]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max}$ = 0.002
$\Delta\rho_{max}$ = 0.21 e Å^{-3}
$\Delta\rho_{min}$ = −0.28 e Å^{-3}

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

D—H···A	D—H	H···A	D···A	D—H···A
N2—H2···O5	0.86	2.04	2.644 (3)	126
N2—H2···O5^{i}	0.86	2.59	3.315 (3)	142
C7—H7A···O3^{ii}	0.96	2.53	3.396 (4)	151

Symmetry codes: (i) $-x+3, -y+1, -z$; (ii) $-x+1, -y+1, -z+1$.

H atoms were included in calculated positions and refined using a riding-model approximation: C—H = 0.93 Å and U_{iso}(H) = 1.2U_{eq}(C) for Csp^2; C—H = 0.96 Å and U_{iso}(H) = 1.5U_{eq}(C) for methyl; N—H = 0.86 Å and U_{iso}(H) = 1.2U_{eq}(N) for amino H.

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*.

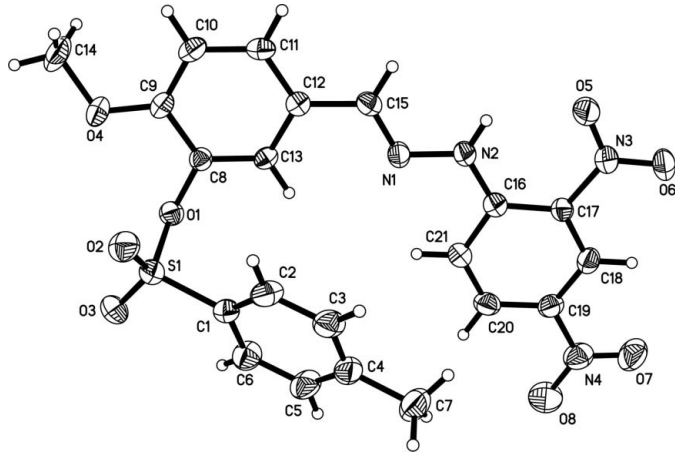


Figure 1
The molecular structure of (I), with displacement ellipsoids for non-H atoms drawn at the 30% probability level.

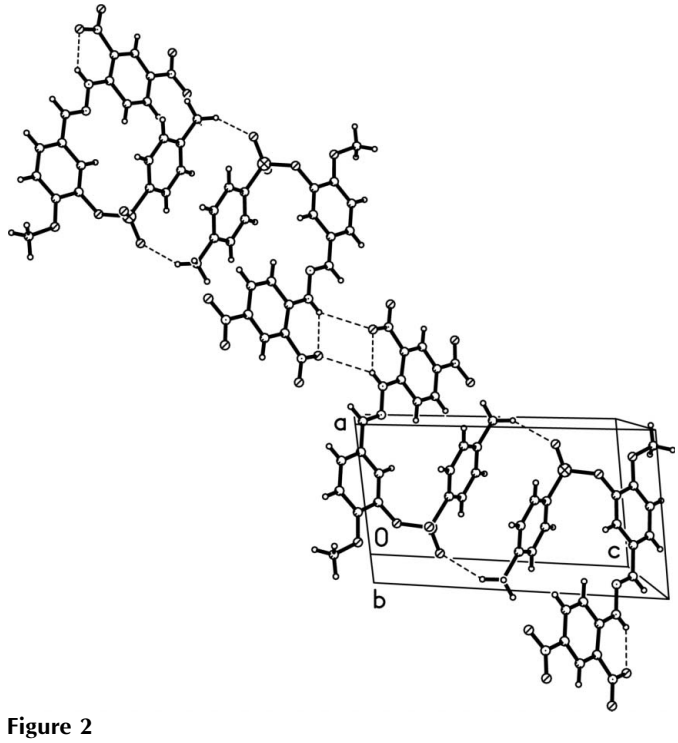


Figure 2
A packing diagram for (I), with hydrogen bonds shown as dashed lines.

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