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(*E*)-1-[3-Methoxy-4-(*p*-tolylsulfonyloxy)-benzylidene]-2-(4-nitrophenyl)hydrazine

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Key indicators

Single-crystal X-ray study
T = 294 K
Mean σ(C–C) = 0.007 Å
R factor = 0.051
wR factor = 0.086
Data-to-parameter ratio = 14.9

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

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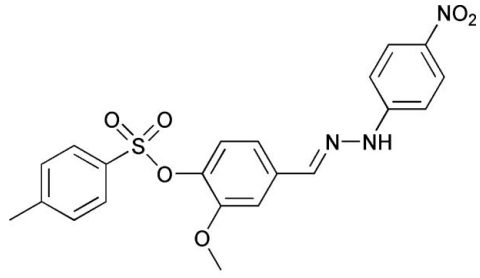
The title compound, $C_{21}H_{19}N_3O_6S$, crystallizes with two molecules in the asymmetric unit. The conformations of these non-planar molecules are similar. Intermolecular N—H···O and C—H···O hydrogen bonds help to consolidate the crystal structure.

Comment

There has been a steady growth of interest in the synthesis, structure and reactivity of Schiff bases due to their potential uses as antibacterial and antitumor agents (Klayman *et al.*, 1979). Among the large number of these compounds, 1-(4-nitrophenyl)hydrazine forms a variety of Schiff bases with aldehydes, and the synthesis and crystal structures of some of them, such as (*E*)-1-(4-methoxy-3-propoxybenzylidene)-2-(4-nitrophenyl)hydrazine (Shi, 2005) and (*E*)-1-[4-(benzyloxy)-benzylidene]-2-(4-nitrophenyl)hydrazine (Jun, 2005) have been reported. In the present study, we report the synthesis and crystal structure of the nitrophenylhydrazine Schiff base derivative (I) (Fig. 1).



(I)

The asymmetric unit of (I) consists of two independent molecules, which are quite similar to each other. All the bond lengths and angles in (I) are within their normal ranges (Allen *et al.*, 1987). In molecule 1, the vanillin group (atoms C7–C13/O3/O4) is planar, with an r.m.s. deviation, δ, from the mean plane of 0.0319 Å. It makes dihedral angles of 10.71 (3) and 57.10 (15)° with the nitrophenylhydrazine residue (C1–C6/N1–N3/O1/O2) and the terminal benzene ring (C15–C20), respectively. In molecule 2, the vanillin group (atoms C28–C33/O9/O10) is also planar, with δ = 0.0328 Å, and it makes dihedral angles of 6.08 (2) and 57.01 (14)° with the nitrophenylhydrazine residue (C24–C29/N4/N5) and the terminal benzene ring (C36–C41), respectively. In addition, the vanillin groups of molecules 1 and 2 are close to coplanar, making a dihedral angle of 7.26 (3)°, while the dihedral angle between the nitrophenylhydrazine residues is 9.84 (2)°.

Two intermolecular N—H···O hydrogen bonds and two weak non-classical intermolecular C—H···O hydrogen bonds

are found in (I) (Table 1 and Fig. 2), which help to consolidate the crystal structure. These hydrogen bonds link adjacent molecules, forming an infinite network.

Experimental

An anhydrous ethanol solution (50 ml) of 4-formyl-2-methoxyphenyl 4-methylbenzenesulfonate (3.06 g, 10 mmol) was added to an anhydrous ethanol solution (50 ml) 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 ethanol 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 ethanol solution.

Crystal data

$C_{21}H_{19}N_3O_6S$
M_r = 441.45
Orthorhombic, $Pna2_1$
a = 20.272 (3) Å
b = 16.568 (3) Å
c = 12.348 (2) Å
V = 4147.3 (12) Å^3
Z = 8
D_x = 1.414 Mg m^{-3}
Mo $K\alpha$ radiation
μ = 0.20 mm^{-1}
T = 294 (2) K
Block, yellow
0.22 × 0.22 × 0.20 mm

Data collection

Bruker SMART APEX CCD diffractometer
ω scans
Absorption correction: multi-scan (*SADABS*; Sheldrick, 1996)
T_{min} = 0.945, T_{max} = 0.961
22718 measured reflections
8350 independent reflections
3462 reflections with $I > 2\sigma(I)$
R_{int} = 0.074
θ_{max} = 26.5°

Refinement

Refinement on F^2
$R[F^2 > 2\sigma(F^2)]$ = 0.051
$wR(F^2)$ = 0.086
S = 1.08
8350 reflections
562 parameters
H-atom parameters constrained
$w = 1/[\sigma^2(F_o^2) + (0.0156P)^2]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max}$ = 0.001
$\Delta\rho_{max}$ = 0.33 e Å^{-3}
$\Delta\rho_{min}$ = −0.21 e Å^{-3}
Absolute structure: Flack (1983), 3859 Friedel pairs
Flack parameter: −0.05 (8)

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

D—H···A	D—H	H···A	D···A	D—H···A
N2—H2···O6^{i}	0.86	2.13	2.981 (5)	168
N5—H5···O12^{ii}	0.86	2.25	3.107 (5)	172
C14—H14B···O1^{iii}	0.96	2.58	3.476 (6)	156
C35—H35B···O8^{iii}	0.96	2.56	3.482 (6)	161

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

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*.

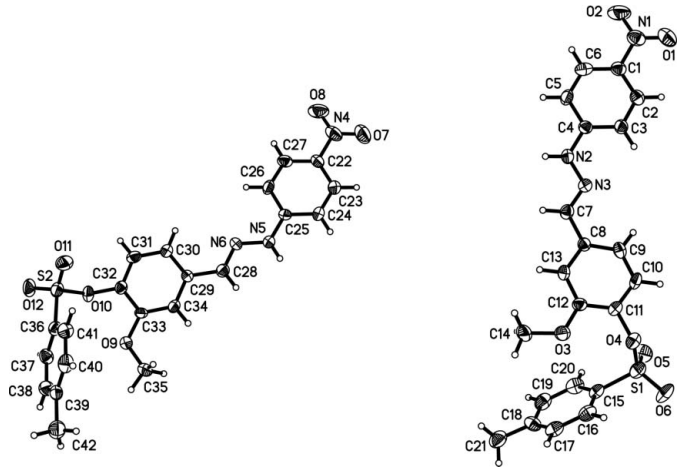


Figure 1
The asymmetric unit of (I), with displacement ellipsoids for non-H atoms drawn at the 30% probability level.

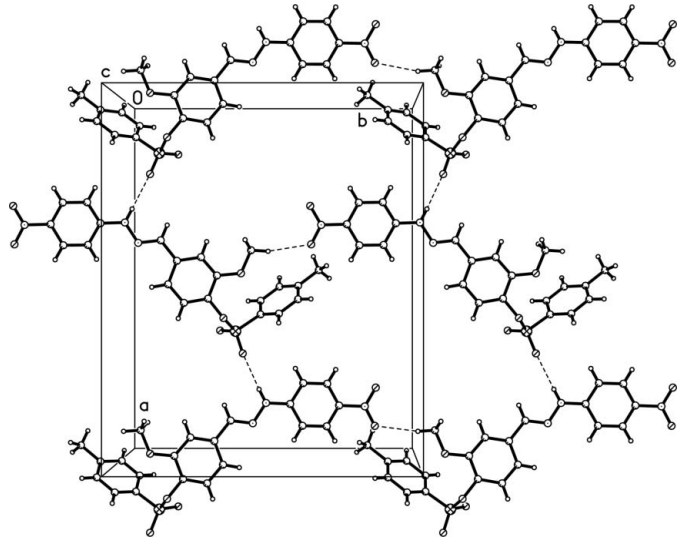


Figure 2
Packing diagram for (I), with hydrogen bonds shown as dashed lines.

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