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

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In the title compound, $C_{21}H_{18}N_4O_8S$, the vanillin group makes dihedral angles of 59.15 (9) and 5.36 (8)° with the terminal 4-methylbenzene ring and the phenylhydrazine mean plane, respectively. An intramolecular hydrogen bond links the NH group to an O atom of the nearest nitro group. The crystal structure contains three very weak intermolecular hydrogen bonds, *viz*. one N—H···O and two C—H···O.

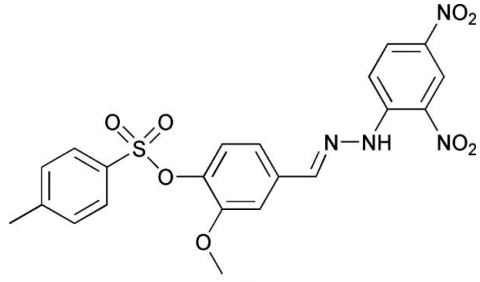
Key indicators

Single-crystal X-ray study
T = 294 K
Mean σ(C–C) = 0.004 Å
R factor = 0.048
wR factor = 0.134
Data-to-parameter ratio = 12.3

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

Comment

The synthesis and structure of Schiff bases have attracted much attention in biology and chemistry (Kahwa *et al.*, 1986). One of the aims of investigating the structural chemistry of Schiff bases is to develop protein and enzyme mimics (Santos *et al.*, 2001). Structural information is useful when investigating the coordination properties of Schiff bases functioning as ligands. 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 (atoms C16–C21/N1/N2) is planar, with a r.m.s. deviation for fitted atoms of 0.0113 Å. This plane makes dihedral angles of 62.98 (10)° and 5.36 (8)° with the terminal benzene ring (C1–C6) and the vanillin group (C8–C13/C15/O3/O4), respectively. In addition, the dihedral angle between the terminal benzene ring and the vanillin group is 59.15 (9)°.

An intramolecular hydrogen bond links the NH group to O5, thereby influencing the molecular conformation. The crystal structure contains three very weak intermolecular hydrogen bonds, *viz*. one N—H···O and two C—H···O (Table 1). These C—H···O hydrogen bonds link molecules into one-dimensional extended chains (Fig. 2).

Experimental

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

drous 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. An orange precipitate appeared which was isolated, recrystallized from acetonitrile, and then dried in a vacuum to give the pure compound in 87% yield. Orange single crystals of (I) suitable for X-ray analysis were obtained by slow evaporation of an *N*,*N*-dimethylformamide solution.

Crystal data

$C_{21}H_{18}N_4O_8S$
M_r = 486.45
Triclinic, $P\overline{1}$
a = 7.6419 (19) Å
b = 9.533 (2) Å
c = 15.727 (4) Å
α = 105.317 (4)°
β = 96.560 (4)°
γ = 95.347 (4)°
V = 1088.7 (5) Å^3
Z = 2
D_x = 1.484 Mg m^{-3}
Mo $K\alpha$ radiation
μ = 0.21 mm^{-1}
T = 294 (2) K
Block, orange
0.30 × 0.26 × 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} = 0.926, T_{max} = 0.960
5565 measured reflections
3816 independent reflections
2310 reflections with $I > 2\sigma(I)$
R_{int} = 0.027
θ_{max} = 25.0°

Refinement

Refinement on F^2
$R[F^2 > 2\sigma(F^2)]$ = 0.048
$wR(F^2)$ = 0.134
S = 1.04
3816 reflections
309 parameters
H-atom parameters constrained
$w = 1/[\sigma^2(F_o^2) + (0.0643P)^2 + 0.0431P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max}$ = 0.001
$\Delta\rho_{max}$ = 0.19 e Å^{-3}
$\Delta\rho_{min}$ = −0.38 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	2.00	2.617 (3)	128
N2—H2···O2^{i}	0.86	2.58	3.348 (3)	149
C15—H15···O2^{i}	0.93	2.33	3.222 (3)	160
C13—H13···O5^{ii}	0.93	2.65	3.303 (4)	128

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

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 = 0.96 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*.

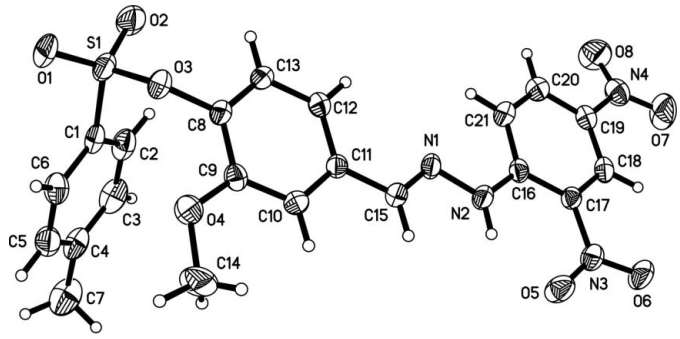


Figure 1
The molecular structure of (I), with displacement ellipsoids for non-H atoms drawn at the 30% probability level.

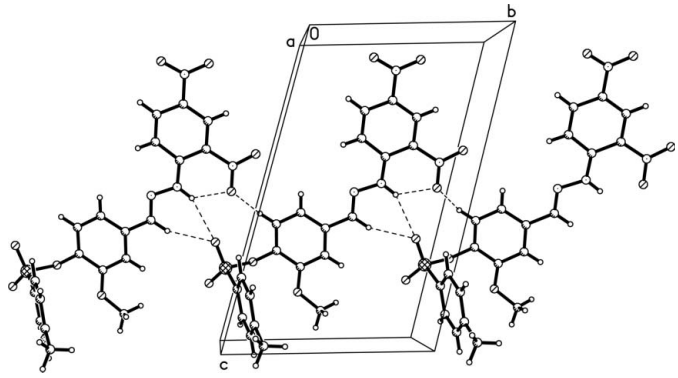


Figure 2
A packing diagram for (I), with hydrogen bonds drawn as dashed lines.

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