organic papers

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Key indicators

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

The molecule of the title compound, $C_{20}H_{18}N_4O_6 \cdot C_3H_3N$, is non-planar. The central benzene ring makes dihedral angles of 85.73 (10) and 2.70 (12)° with the terminal phenyl ring and the nitrophenylhydrazine mean plane, respectively. An intramolecular N—H···O hydrogen bond helps to establish the molecular conformation.

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 in investigating the coordination properties of Schiff bases functioning as ligands. We report here the synthesis and structure of the title Schiff base compound, (I) (Fig. 1).


(I)
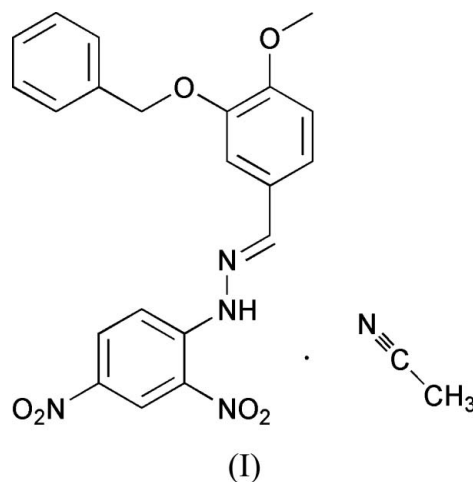
The nitrophenylhydrazine residue (C16–C21/O5/O6/N1/N2/N3) is planar, with an r.m.s. deviation for fitted atoms of 0.0200 Å. This plane makes dihedral angles of 85.10 (9) and 2.70 (12)° with the terminal phenyl ring (C1–C6) and the central benzene ring (C8–C13), respectively. The central benzene ring is almost perpendicular to the terminal phenyl ring, with a dihedral angle of 85.73 (10)°. The O5—N4—C19—C18 and O6—N4—C19—C20 torsion angles are −179.2 (3) and −179.4 (3)°, respectively, confirming the coplanarity of the nitro group (O5/N4/O6) and its attached aromatic ring, while the other nitro group (O3/N3/O4) makes a dihedral angle of 6.75 (3)°.

An intramolecular N—H···O hydrogen bond is found in (I) (Table 2). which helps to stabilize the conformation of the molecule (Fig. 2).
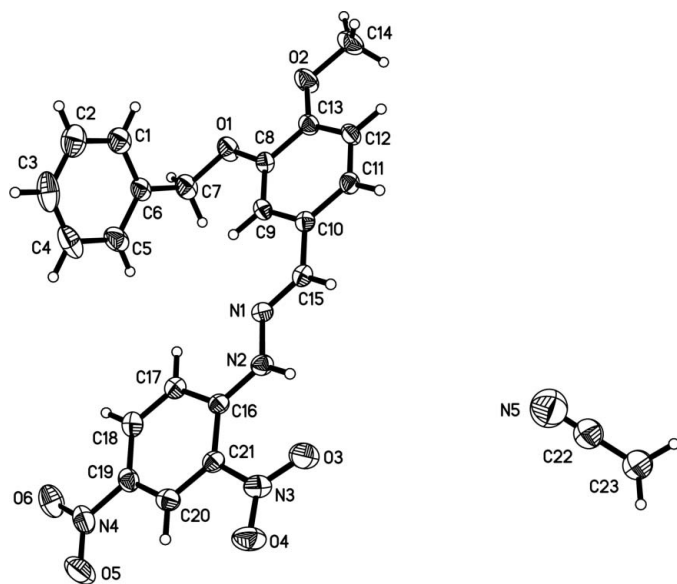
Figure 1
The structure of (I), with displacement ellipsoids for non-H atoms drawn at the 30% probability level.
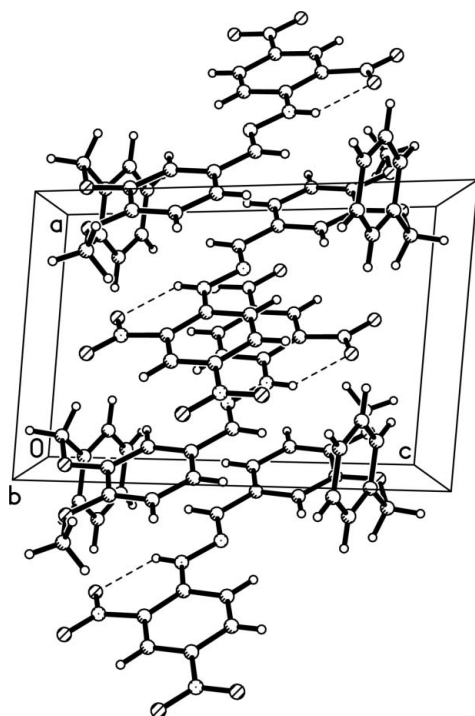
Figure 2
Packing diagram for (I), with hydrogen bonds drawn as dashed lines.

Experimental

An anhydrous ethanol solution of 3-(benzyloxy)-4-methoxy-benzaldehyde (2.42 g, 10 mmol) was added to an anhydrous ethanol solution of 1-(2,4-dinitrophenyl)hydrazine (1.98 g, 10 mmol) and the mixture stirred at 350 K for 5 h under nitrogen, whereupon a red precipitate appeared. The product was isolated, recrystallized from acetonitrile and dried in a vacuum to give the pure compound in 88% yield. Red single crystals of (I) suitable for X-ray analysis were obtained by slow evaporation of an acetonitrile solution.

Crystal data

$C_{21}H_{18}N_4O_6 \cdot C_2H_3N$	$Z = 2$
$M_r = 463.45$	$D_x = 1.368$ Mg m^{-3}
Triclinic, $P\bar{1}$	Mo $K\alpha$ radiation
$a = 7.945$ (3) Å	Cell parameters from 1295
$b = 12.253$ (5) Å	reflections
$c = 12.549$ (5) Å	$\theta = 2.6$–$25.5°$
$\alpha = 67.618$ (7)°	$\mu = 0.10$ mm^{-1}
$\beta = 84.794$ (7)°	$T = 293$ (2) K
$\gamma = 87.729$ (8)°	Block, red
$V = 1124.9$ (8) Å^3	$0.20 \times 0.20 \times 0.08$ mm

Data collection

Bruker SMART APEX CCD area-detector diffractometer	3947 independent reflections
	1985 reflections with $I > 2\sigma(I)$
φ and ω scans	$R_{int} = 0.026$
Absorption correction: multi-scan (*SADABS*; Sheldrick, 1996)	$\theta_{max} = 25.0°$
	$h = -8 \rightarrow 9$
$T_{min} = 0.968$, $T_{max} = 0.992$	$k = -11 \rightarrow 14$
5736 measured reflections	$l = -14 \rightarrow 14$

Refinement

Refinement on F^2	H-atom parameters constrained
$R[F^2 > 2\sigma(F^2)] = 0.051$	$w = 1/[\sigma^2(F_o^2) + (0.0615P)^2$
$wR(F^2) = 0.153$	$\quad + 0.2043P]$
$S = 1.03$	$\quad$ where $P = (F_o^2 + 2F_c^2)/3$
3947 reflections	$(\Delta/\sigma)_{max} = 0.001$
309 parameters	$\Delta\rho_{max} = 0.33$ e Å^{-3}
	$\Delta\rho_{min} = -0.28$ e Å^{-3}

Table 1
Selected geometric parameters (Å, °).

O1—C8	1.368 (3)	O6—N4	1.222 (3)
O1—C7	1.434 (3)	N1—C15	1.277 (3)
O2—C13	1.365 (3)	N1—N2	1.373 (3)
O2—C14	1.421 (3)	N2—C16	1.349 (3)
O3—N3	1.233 (3)	N3—C21	1.446 (4)
O4—N3	1.227 (3)	N4—C19	1.452 (4)
O5—N4	1.229 (3)	N5—C22	1.126 (5)
C8—O1—C7	117.5 (2)	O1—C8—C9	125.3 (3)
C13—O2—C14	118.6 (2)	O1—C8—C13	115.3 (2)
C15—N1—N2	117.1 (2)	O2—C13—C12	124.9 (3)
C16—N2—N1	119.2 (2)	O2—C13—C8	115.3 (3)
O4—N3—O3	122.0 (3)	N1—C15—C10	119.9 (3)
O4—N3—C21	119.0 (3)	N2—C16—C21	124.3 (3)
O3—N3—C21	119.0 (2)	N2—C16—C17	119.6 (2)
O6—N4—O5	123.2 (3)	C20—C19—N4	119.0 (3)
O6—N4—C19	117.8 (3)	C18—C19—N4	120.0 (3)
O5—N4—C19	119.0 (3)	C16—C21—N3	121.9 (3)
O1—C7—C6	113.5 (3)	N5—C22—C23	179.3 (5)

Table 2
Hydrogen-bond geometry (Å, °).

D—H$\cdots A$	D—H	H$\cdots A$	$D\cdots A$	D—H$\cdots A$
N2—H2$\cdots$O3	0.86	2.02	2.625 (3)	127

The 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: 0.93 Å and $U_{iso}(H) = 1.2U_{eq}(C)$ for aromatic CH; 0.97 Å and $U_{iso}(H) = 1.2U_{eq}(C)$ for methylene CH$_2$; 0.96 Å and $U_{iso}(H) = 1.5U_{eq}(C)$ for methyl CH$_3$; 0.86 Å and $U_{iso}(H) = 1.2U_{eq}(N)$ for NH.

Data collection: *SMART* (Bruker, 1999); cell refinement: *SAINT* (Bruker, 1999); data reduction: *SAINT*; program(s) used to solve structure: *SHELXS97* (Sheldrick, 1997a); 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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