organic papers

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(E)-1-[3-(Benzyloxy)benzylidene]-2-(2,4-dinitro-phenyl)hydrazine

The title compound, $C_{20}H_{16}N_4O_5$, is non-planar. The central benzene ring makes dihedral angles of 58.14 (6) and 5.00 (9)° with the terminal phenyl ring and the nitrophenylhydrazine mean plane, respectively. A bifurcated intra/intermolecular N—H···(O,O) hydrogen bond helps to establish the molecular conformation and consolidate the crystal packing.

Comment

Schiff bases have been studied extensively because of their potential biological activity (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). As part of an investigation of the coordination properties of Schiff bases functioning as ligands, we report the synthesis and structure of the title compound, (I) (Fig. 1).

(I)
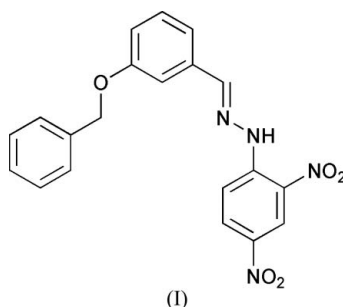
In (I), the nitrophenylhydrazine system (C15–C20/O2/O3/N1/N2/N3) is planar, with an r.m.s. deviation for fitted atoms of 0.0060 Å. This plane makes dihedral angles of 62.13 (5) and
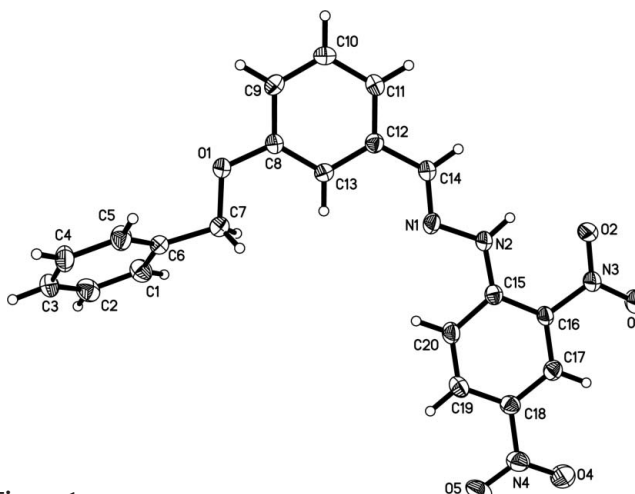
Figure 1
The structure of (I), with displacement ellipsoids for non-H atoms drawn at the 30% probability level.
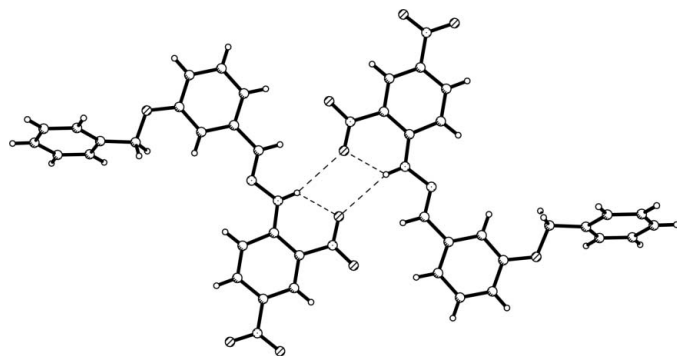
Figure 2
Intermolecular and intramolecular hydrogen-bonding interactions (dashed lines).

5.00 (9) with the terminal phenyl ring (C1–C6) and the central benzene ring (C8–C13), respectively. The dihedral angle between the terminal phenyl and the central benzene rings is 58.14 (6). The O2—N3—C16—C17 and O3—N3—C16—C15 torsion angles are 179.3 (2) and 179.9 (2), respectively, confirming the coplanarity of the nitro group (O2/N3/O3) and its attached aromatic ring, while the other nitro group (O4/N4/O5) makes a dihedral angle of 4.29 (4).

A bifurcated N—H···(O,O) intramolecular/intermolecular N—H···O hydrogen bond system is found in (I) (Table 2). The intramolecular bond stabilizes the conformation of the molecule, while the intermolecular bond to an O atom of an adjacent nitro group, forms centrosymmetric dimers (Fig. 2) and consolidates the crystal packing.

Experimental

An anhydrous ethanol solution of 3-(benzyloxy)benzaldehyde (2.12 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 89% yield. Red single crystals of (I) suitable for X-ray analysis were obtained by slow evaporation of a dimethylformamide solution.

Crystal data

$C_{20}H_{16}N_4O_5$	$Z = 2$
$M_r = 392.37$	$D_x = 1.443$ Mg m^{-3}
Triclinic, $P\bar{1}$	Mo $K\alpha$ radiation
$a = 5.9288$ (18) Å	Cell parameters from 1729
$b = 7.651$ (2) Å	reflections
$c = 20.087$ (6) Å	$\theta = 2.7$–$26.3°$
$\alpha = 96.711$ (5)°	$\mu = 0.11$ mm^{-1}
$\beta = 93.768$ (5)°	$T = 294$ (2) K
$\gamma = 90.115$ (5)°	Block, red
$V = 902.9$ (5) Å^3	0.26 × 0.20 × 0.18 mm

Data collection

Bruker SMART APEX CCD area-detector diffractometer	3137 independent reflections
φ and ω scans	2118 reflections with $I > 2\sigma(I)$
Absorption correction: multi-scan (SADABS; Sheldrick, 1996)	$R_{int} = 0.017$
$T_{min} = 0.960$, $T_{max} = 0.981$	$\theta_{max} = 25.0°$
4495 measured reflections	$h = -7 \rightarrow 7$
	$k = -8 \rightarrow 9$
	$l = -23 \rightarrow 14$

Refinement

Refinement on F^2	$w = 1/[\sigma^2(F_o^2) + (0.053P)^2$
$R[F^2 > 2\sigma(F^2)] = 0.042$	$+ 0.193P]$
$wR(F^2) = 0.117$	where $P = (F_o^2 + 2F_c^2)/3$
$S = 1.02$	$(\Delta/\sigma)_{max} = 0.003$
3137 reflections	$\Delta\rho_{max} = 0.17$ e Å^{-3}
262 parameters	$\Delta\rho_{min} = -0.15$ e Å^{-3}
H-atom parameters constrained	

Table 1
Selected geometric parameters (Å, °).

O1—C8	1.376 (2)	O5—N4	1.223 (2)
O1—C7	1.431 (2)	N1—C14	1.284 (3)
O2—N3	1.237 (2)	N1—N2	1.388 (2)
O3—N3	1.222 (2)	N3—C16	1.453 (3)
O4—N4	1.218 (2)	N4—C18	1.465 (3)
C8—O1—C7	117.32 (15)	O1—C7—C6	109.34 (16)
C14—N1—N2	114.42 (16)	O1—C8—C13	124.91 (18)
C15—N2—N1	120.11 (16)	O1—C8—C9	114.77 (17)
O3—N3—O2	121.55 (18)	N2—C15—C20	119.94 (18)
O3—N3—C16	119.34 (18)	N2—C15—C16	123.99 (18)
O2—N3—C16	119.10 (16)	C17—C16—N3	116.02 (17)
O4—N4—O5	123.4 (2)	C15—C16—N3	121.86 (18)
O4—N4—C18	118.55 (19)	C17—C18—N4	119.20 (18)
O5—N4—C18	118.04 (19)	C19—C18—N4	119.58 (18)

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

D—H···A	D—H	H···A	D···A	D—H···A
N2—H2A···O2^i	0.86	2.64	3.460 (2)	160
N2—H2A···O2	0.86	2.01	2.630 (2)	128

Symmetry code: (i) $-x + 2, -y, -z$.

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: 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, 1997a); molecular graphics: SHELXTL (Sheldrick, 1997b); software used to prepare material for publication: SHELXTL.

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