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Key indicators

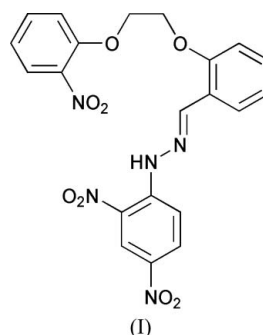
Single-crystal X-ray study
 $T = 294\text{ K}$
Mean $\sigma(\text{C}-\text{C}) = 0.005\text{ \AA}$
Disorder in main residue
 R factor = 0.051
 wR factor = 0.158
Data-to-parameter ratio = 11.6For details of how these key indicators were
automatically derived from the article, see
<http://journals.iucr.org/e>.**(E)-1-(2,4-Dinitrophenyl)-2-{2-[2-(2-nitro-
phenoxy)ethoxy]benzylidene}hydrazine**

The molecule of the title compound, $\text{C}_{21}\text{H}_{17}\text{N}_5\text{O}_8$, is non-planar. The central benzene ring makes dihedral angles of $39.57(10)$ and $2.93(17)^\circ$ with the two terminal benzene rings. An intramolecular $\text{N}-\text{H}\cdots\text{O}$ hydrogen bond helps to establish the molecular conformation.

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Comment

The synthesis and structure of Schiff bases have attracted 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) in order to investigate their crystal structures, which will provide useful information for the coordination properties of Schiff bases functioning as ligands. We report here the synthesis and crystal structure of the title compound, (I).



The molecular structure of compound (I) is illustrated in Fig. 1 and selected bond distances and angles are given in Table 1. The central benzene ring (C9–C14) makes dihedral angles of $39.57(10)^\circ$ and $2.93(17)^\circ$ with the terminal benzene rings of the 1-(2,4-dinitrophenyl)hydrazine group, (C1–C6) and (C16–C21), respectively. The two nitro groups of the dinitrophenylhydrazine group are not coplanar with the benzene ring, the dihedral angles between them being $5.82(59)^\circ$ and $7.89(42)^\circ$ for (O5/N4/O6) and (O7/N5/O8), respectively. One of the O atoms of the other nitro group (O1/N3/O2) is disordered over two positions, O2 and O2' (occupancies 0.5/0.5). Otherwise, the bond lengths and angles for (I) are within normal limits.

An intramolecular $\text{N}-\text{H}\cdots\text{O}$ hydrogen bond is found in compound (I) (Table 2), and this helps to stabilize the conformation of the molecule (Fig. 2). In the crystal structure, the packing is further stabilized by van der Waals forces.

Experimental

To a solution of 2-hydroxybenzaldehyde (6.1 g, 50 mmol) and potassium carbonate (13.8 g, 100 mmol) in acetonitrile (500 ml),

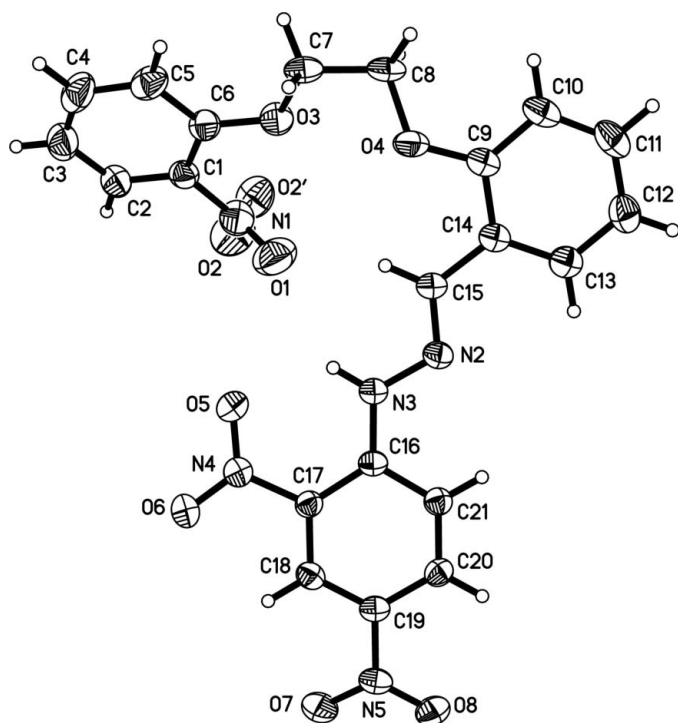


Figure 1
The molecular structure of (I), showing the atom labelling and displacement ellipsoids drawn at the 30% probability level.

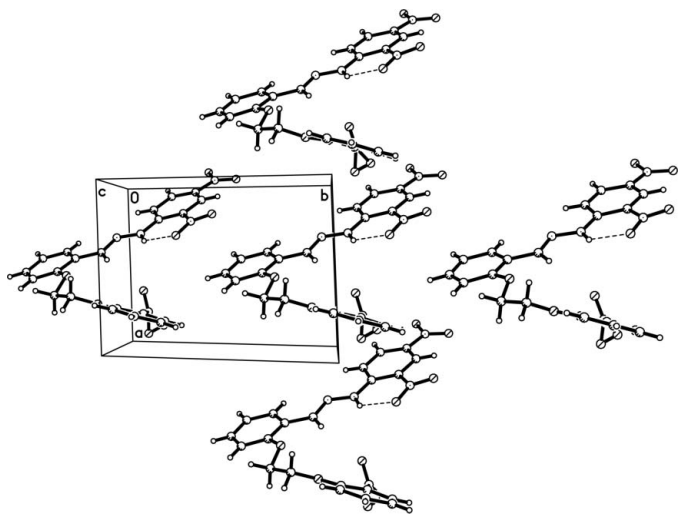


Figure 2
A view, along *c*, of the crystal packing of compound (I), showing the intramolecular hydrogen-bonding interactions (dashed lines).

1-bromo-2-chloroethane (7.1 g, 50 mmol) was added dropwise over 30 minutes, and the mixture refluxed for 24 h under nitrogen. 2-nitrophenol (7.0 g, 50 mmol), dissolved in acetonitrile (100 ml), was then added dropwise over 30 minutes. This mixture was refluxed for an additional 48 h under nitrogen. The solvent was then removed and the resultant mixture poured into ice-water (500 ml). A white precipitate was isolated and recrystallized from ethanol to give the pure compound, 2-(2-(2-nitrophenoxy)ethoxy)benzaldehyde, in 28% yield. An anhydrous ethanol solution of 2-(2-(2-nitrophenoxy)ethoxy)benzaldehyde (2.87 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 then dried in a vacuum to give pure compound (I): 88% yield. Red single crystals of (I), suitable for X-ray analysis, were obtained by slow evaporation of an acetonitrile solution.

Crystal data

$C_{21}H_{17}N_5O_8$
 $M_r = 467.40$
Triclinic, $P\bar{1}$
 $a = 7.953 (3) \text{ \AA}$
 $b = 11.686 (5) \text{ \AA}$
 $c = 12.524 (5) \text{ \AA}$
 $\alpha = 65.107 (6)^\circ$
 $\beta = 87.463 (7)^\circ$
 $\gamma = 86.980 (8)^\circ$
 $V = 1054.0 (7) \text{ \AA}^3$

$Z = 2$
 $D_x = 1.473 \text{ Mg m}^{-3}$
Mo $K\alpha$ radiation
Cell parameters from 1432 reflections
 $\theta = 1.8\text{--}25.0^\circ$
 $\mu = 0.12 \text{ mm}^{-1}$
 $T = 294 (2) \text{ K}$
Block, red
 $0.24 \times 0.16 \times 0.12 \text{ mm}$

Data collection

Bruker SMART CCD area-detector diffractometer
 φ and ω scans
Absorption correction: multi-scan (SADABS; Sheldrick, 1996)
 $T_{\min} = 0.961$, $T_{\max} = 0.986$
5343 measured reflections

3677 independent reflections
2069 reflections with $I > 2\sigma(I)$
 $R_{\text{int}} = 0.020$
 $\theta_{\max} = 25.0^\circ$
 $h = -9 \rightarrow 6$
 $k = -13 \rightarrow 11$
 $l = -14 \rightarrow 14$

Refinement

Refinement on F^2
 $R[F^2 > 2\sigma(F^2)] = 0.051$
 $wR(F^2) = 0.158$
 $S = 1.00$
3677 reflections
316 parameters
H-atom parameters constrained

$w = 1/[\sigma^2(F_o^2) + (0.0713P)^2 + 0.3108P]$
where $P = (F_o^2 + 2F_c^2)/3$
 $(\Delta/\sigma)_{\max} = 0.001$
 $\Delta\rho_{\max} = 0.39 \text{ e \AA}^{-3}$
 $\Delta\rho_{\min} = -0.21 \text{ e \AA}^{-3}$

Table 1

Selected geometric parameters (\AA , $^\circ$).

O1—N1	1.237 (4)	O7—N5	1.218 (3)
O2—N1	1.159 (6)	O8—N5	1.222 (3)
O2'—N1	1.243 (6)	N1—C1	1.450 (4)
O3—C6	1.348 (4)	N2—C15	1.275 (3)
O3—C7	1.427 (4)	N2—N3	1.367 (3)
O4—C9	1.363 (4)	N3—C16	1.347 (3)
O4—C8	1.416 (4)	N4—C17	1.447 (4)
O5—N4	1.226 (3)	N5—C19	1.452 (4)
O6—N4	1.211 (3)		
C6—O3—C7	119.2 (3)	C2—C1—N1	118.2 (3)
C9—O4—C8	120.6 (2)	C6—C1—N1	118.6 (3)
O2—N1—O1	114.3 (6)	O3—C6—C1	117.2 (3)
O2—N1—C1	122.8 (5)	O3—C6—C5	125.5 (3)
O1—N1—C1	119.0 (4)	O4—C8—C7	106.9 (2)
C15—N2—N3	114.4 (3)	O4—C9—C10	124.4 (3)
C16—N3—N2	120.2 (2)	O4—C9—C14	114.3 (3)
O6—N4—O5	121.6 (3)	N3—C16—C21	120.5 (3)
O6—N4—C17	118.6 (3)	N3—C16—C17	123.2 (3)
O5—N4—C17	119.8 (3)	C18—C17—N4	116.4 (3)
O7—N5—O8	123.6 (3)	C16—C17—N4	121.7 (3)
O7—N5—C19	117.9 (3)	C18—C19—N5	119.3 (3)
O8—N5—C19	118.4 (3)	C20—C19—N5	119.8 (3)

Table 2

Hydrogen-bond geometry (\AA , $^\circ$).

$D\text{—}H\cdots A$	$D\text{—}H$	$H\cdots A$	$D\cdots A$	$D\text{—}H\cdots A$
N3—H3 \cdots O5	0.86	1.99	2.614 (3)	129

The H atoms were included in calculated positions and refined using a riding model approximation. C—H distances 0.93–0.96 Å and $U_{\text{iso}}(\text{H}) = 1.2U_{\text{eq}}(\text{C}_{\text{aromatic}})$ or $1.5U_{\text{eq}}(\text{C}_{\text{methyl}})$. The N—H distance is 0.86 Å and $U_{\text{iso}}(\text{H}) = 1.2U_{\text{eq}}(\text{N})$.

Data collection: *SMART* (Bruker, 1999); cell refinement: *SAINTE* (Bruker, 1999); data reduction: *SAINTE*; 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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