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N,*N*′-Bis(3-nitrobenzylidene)hydrazine

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Key indicators

Single-crystal X-ray study
T = 294 K
Mean σ(C–C) = 0.003 Å
R factor = 0.038
wR factor = 0.116
Data-to-parameter ratio = 11.9

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

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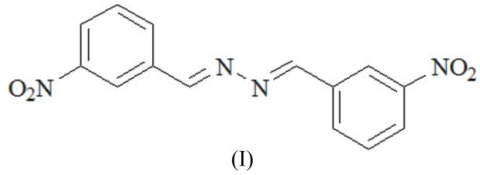
The title compound, $C_{14}H_{10}N_4O_4$, was synthesized by the reaction of 3-nitrobenzaldehyde with hydrazine hydrate. The molecule possesses a crystallographically imposed center of symmetry and is essentially planar; the dihedral angle between the nitro group and the benzene ring is 3.4 (2)°. Intermolecular C—H···O hydrogen bonds link the molecules into an infinite chain.

Comment

Recently, a number of azine compounds containing both a diimine linkage and N—N bonding have been investigated in terms of their crystallography and coordination chemistry (Zheng *et al.*, 2005; Kundu *et al.*, 2005; Kesslen & Euler, 1999; Armstrong *et al.*, 1998; Xu *et al.*, 1997). We report here the crystal structure of the title compound, (I), where two 3-nitrobenzylidene units are directly linked through the imine N atoms.



(I)

The title molecule crystallizes in an *E*,*E* configuration, possessing a crystallographically imposed center of symmetry at the mid-point of the N—N bond (Fig. 1). This configuration agrees with that commonly found in a number of azine compounds (Zheng *et al.*, 2005, Şengül *et al.*, 2004, Liu *et al.*, 2004). The N—N bond length of 1.409 (3) Å (Table 1) is somewhat longer than that observed in related azine compounds (Xu *et al.*, 2005; Şengül *et al.*, 2004). The C═N—N angle [111.9 (2)°] is similar to that in *N*,*N*′-bis(4-chloro-

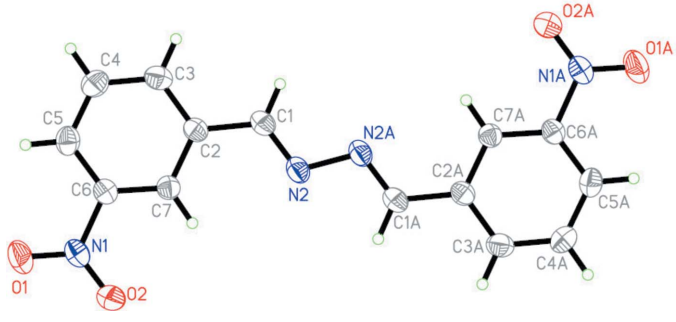


Figure 1
View of the molecule of (I), with displacement ellipsoids drawn at the 30% probability level. The suffix A corresponds to symmetry code (3 − *x*, 1 − *y*, 2 − *z*).

benzylidene)hydrazine (Zheng *et al.*, 2005), but significantly smaller than the ideal sp^2 value of 120°, as a consequence of repulsion between the nitrogen lone pairs and the adjacent C=N bond. The dihedral angle between the nitro group and the benzene ring is 3.4 (2)°. The bis-benzylidenehydrazine backbone assumes a planar structure, with an r.m.s. deviation of 0.012 (2) Å.

In the crystal structure, a weak intermolecular C—H···O hydrogen bond (Table 2) leads to a ten-membered ring, described by the graph-set descriptor $R_2^2(10)$, linking the molecules into an infinite chain (Fig. 2).

Experimental

The title compound was synthesized by the reaction of 3-nitrobenzaldehyde with hydrazine hydrate in refluxing ethanol (Liu *et al.*, 2004). Single crystals suitable for X-ray analysis were obtained by slow evaporation of a tetrahydrofuran solution.

Crystal data

$C_{14}H_{10}N_4O_4$
M_r = 298.26
Monoclinic, $P2_1/n$
a = 6.9694 (19) Å
b = 7.833 (2) Å
c = 12.990 (4) Å
β = 105.175 (4)°
V = 684.4 (3) Å^3
Z = 2
D_x = 1.447 Mg m^{-3}
Mo $K\alpha$ radiation
Cell parameters from 1134 reflections
θ = 3.0–26.1°
μ = 0.11 mm^{-1}
T = 294 (2) K
Block, light yellow
0.34 × 0.30 × 0.14 mm

Data collection

Bruker SMART 1000 CCD area-detector diffractometer
φ and ω scans
Absorption correction: multi-scan (*SADABS*; Sheldrick, 1996)
T_{min} = 0.962, T_{max} = 1.000
3318 measured reflections
1204 independent reflections
814 reflections with $I > 2\sigma(I)$
R_{int} = 0.035
θ_{max} = 25.0°
$h = -8 \rightarrow 8$
$k = -7 \rightarrow 9$
$l = -15 \rightarrow 15$

Refinement

Refinement on F^2
$R[F^2 > 2\sigma(F^2)]$ = 0.038
$wR(F^2)$ = 0.116
S = 1.03
1204 reflections
101 parameters
H-atom parameters constrained
$w = 1/[\sigma^2(F_o^2) + (0.061P)^2 + 0.0625P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} < 0.001$
$\Delta\rho_{max}$ = 0.12 e Å^{-3}
$\Delta\rho_{min}$ = −0.18 e Å^{-3}
Extinction correction: *SHELXL97*
Extinction coefficient: 0.094 (9)

Table 1
Selected geometric parameters (Å, °).

O1—N1	1.223 (2)	N2—C1	1.272 (2)
O2—N1	1.2181 (19)	N2—N2^{i}	1.409 (3)
N1—C6	1.473 (2)		
O2—N1—O1	123.11 (17)	O1—N1—C6	118.20 (17)
O2—N1—C6	118.68 (16)	C1—N2—N2^{i}	111.9 (2)

Symmetry code: (i) $-x+3, -y+1, -z+2$.

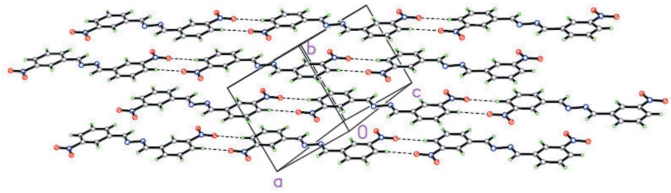


Figure 2
The packing of (I), showing the intermolecular hydrogen-bonded (dashed lines) extended network.

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

D—H···A	D—H	H···A	D···A	D—H···A
C5—H5···O1^{ii}	0.93	2.56	3.487 (3)	176

Symmetry code: (ii) $-x+1, -y+2, -z+2$.

All H atoms were positioned geometrically and refined as riding, with C—H = 0.93 Å and U_{iso}(H) = 1.2U_{eq}(C).

Data collection: *SMART* (Bruker, 1997); cell refinement: *SAINT* (Bruker, 1997); data reduction: *SAINT*; program(s) used to solve structure: *SHELXS97* (Sheldrick, 1997); program(s) used to refine structure: *SHELXL97* (Sheldrick, 1997); molecular graphics: *SHELXTL* (Bruker, 1997); software used to prepare material for publication: *SHELXTL*.

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