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N,*N*′-Bis(3-nitrobenzylidene)-*trans*-1,2-cyclohexanediamine

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Key indicators

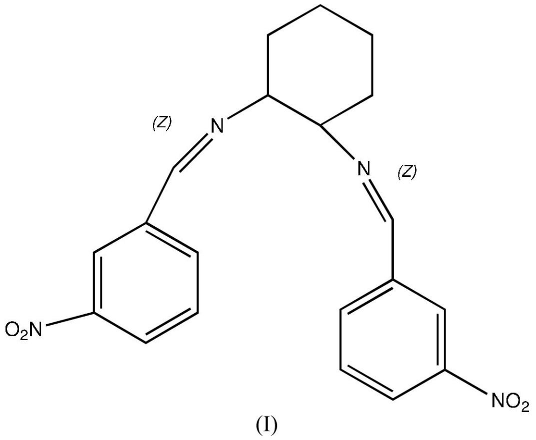
Single-crystal X-ray study
T = 298 K
Mean σ(C–C) = 0.006 Å
R factor = 0.093
wR factor = 0.199
Data-to-parameter ratio = 16.4

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

The title compound, $C_{20}H_{20}N_4O_4$, is a potentially *N*,*N*′-bidentate donor ligand with two chiral C atoms. In the crystal structure, the molecules form centrosymmetric dimers *via* C—H···N hydrogen bonds.

Comment

Chiral Schiff base complexes based on optically active 1,2-diamines (mainly 1,2-cyclohexanediamine) have been used in the epoxidation of *cis*-disubstituted olefins (Bernardo *et al.*, 1996; Jacobsen, 1993), cyclic dienes (Chang *et al.*, 1994) or polyenes (Chang *et al.*, 1993). As we are interested in chiral Schiff base ligands, we report here the synthesis and crystal structure analysis of a new potentially bidentate chiral ligand, the title compound, (I).



(I)

The molecular structure of (I) is illustrated in Fig. 1. The bond lengths and angles are within normal ranges (Allen *et al.*,

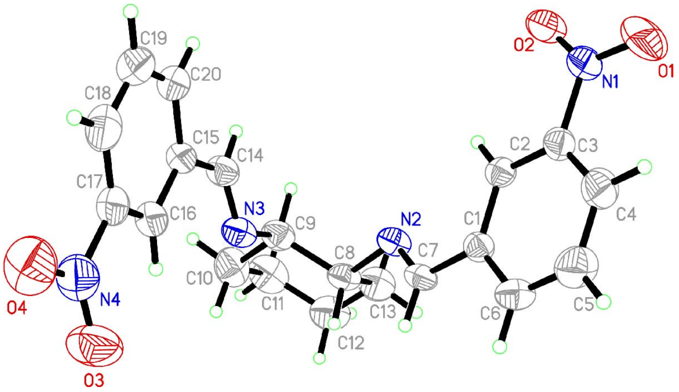


Figure 1
The molecuar structure of (I), showing 30% probability displacement ellipsoids and the atom-numbering scheme.

1987). The C15—C14—N3 bond angle of 124.0 (4)° is almost the same as the C1—C7—N2 angle [123.6 (3)°]. The dihedral angle between the two benzene rings is 62.8 (2)°.

In the crystal structure of (I), molecules related by a centre of symmetry are linked to form dimers *via* C—H···N hydrogen bonds (Table 1 and Fig. 2).

Experimental

1,2-Diaminocyclohexane (57.1 mg, 0.5 mmol) and 3-nitrobenzaldehyde (151.1 mg, 1 mmol) were combined in 10 ml of ethanol as solvent. After stirring for 2 h, the resulting solution was allowed to stand in air for 10 d. White crystals of (I) formed on slow evaporation of the solvent. The crystals were isolated, washed with ethanol and dried. Elemental analysis, found: C 63.18, H 5.32, N 14.69%; calculated for $C_{20}H_{20}N_4O_4$: C 63.15, H 5.30, N 14.73%.

Crystal data

$C_{20}H_{20}N_4O_4$
$M_r = 380.40$
Monoclinic, $C2/c$
$a = 19.288$ (4) Å
$b = 15.840$ (3) Å
$c = 15.485$ (3) Å
$\beta = 124.297$ (3)°
$V = 3908.3$ (13) Å^3
$Z = 8$

$D_x = 1.293$ Mg m^{-3}
Mo $K\alpha$ radiation
Cell parameters from 1852 reflections
$\theta = 4.3$–28.4°
$\mu = 0.09$ mm^{-1}
$T = 298$ (2) K
Block, white
0.26 × 0.21 × 0.08 mm

Data collection

Bruker SMART CCD area-detector diffractometer
φ and ω scans
Absorption correction: multi-scan (*SADABS*; Sheldrick, 1996)
$T_{\rm min} = 0.976$, $T_{\rm max} = 0.993$
13 932 measured reflections

4260 independent reflections
1899 reflections with $I > 2\sigma(I)$
$R_{\rm int} = 0.071$
$\theta_{\rm max} = 27.0°$
$h = -17 \rightarrow 24$
$k = -19 \rightarrow 20$
$l = -19 \rightarrow 18$

Refinement

Refinement on F^2
$R[F^2 > 2\sigma(F^2)] = 0.093$
$wR(F^2) = 0.199$
$S = 1.06$
4260 reflections
259 parameters
H atoms treated by a mixture of independent and constrained refinement

$w = 1/[\sigma^2(F_o^2) + (0.0612P)^2 + 1.3941P]$
where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{\rm max} < 0.001$
$\Delta\rho_{\rm max} = 0.23$ e Å^{-3}
$\Delta\rho_{\rm min} = -0.14$ e Å^{-3}

Table 1
Hydrogen-bond geometry (Å, °).

D—H···A	D—H	H···A	D···A	D—H···A
C6—H6···N3$^{\rm i}$	0.93	2.61	3.520 (5)	166

Symmetry code: (i) $-x + \frac{1}{2}, -y + \frac{1}{2}, -z + 2$.

The H atoms bonded to atoms C8 and C9 were located in a Fourier difference map; their positional parameters were refined, with $U_{\rm iso}({\rm H}) = 1.2U_{\rm eq}({\rm C})$. All other H atoms were placed in geometrically idealized positions and constrained to ride on their parent atoms, with C—H distances in the range 0.93–0.97 Å and with $U_{\rm iso}({\rm H}) = 1.2U_{\rm eq}({\rm C})$.

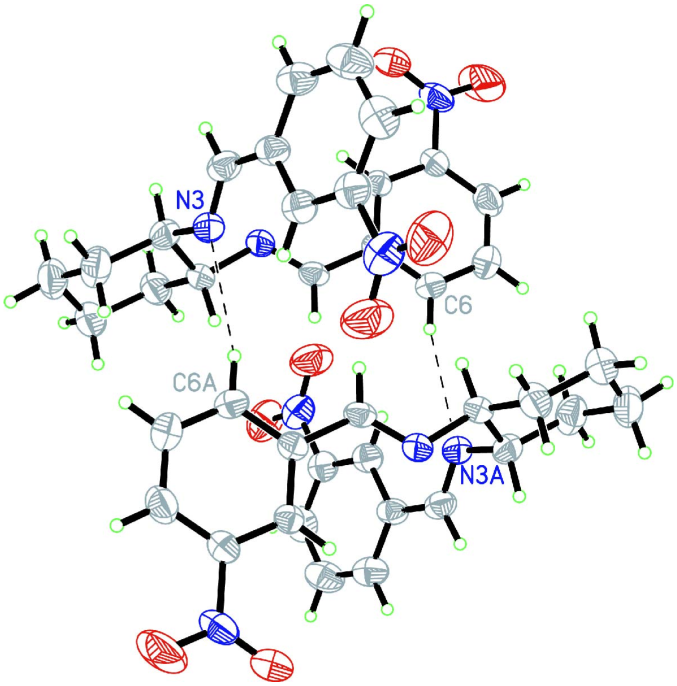


Figure 2
A view of the hydrogen-bonded dimer of (I). Dashed lines indicate hydrogen bonds. [Symmetry code: (A) $\frac{1}{2} - x, \frac{1}{2} - y, 2 - z$.]

Data collection: *SMART* (Siemens, 1996); cell refinement: *SAINT* (Siemens, 1996); 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*.

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