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(1*R*,2*R*)-*N*,*N*′-Bis(4-nitrophenylmethylene)-cyclohexane-1,2-diamine

Christopher Glidewell,[a]* John N. Low,[b] Janet M. S. Skakle[b] and James L. Wardell[c]

[a]School of Chemistry, University of St Andrews, Fife KY16 9ST, Scotland, [b]Department of Chemistry, University of Aberdeen, Meston Walk, Old Aberdeen AB24 3UE, Scotland, and [c]Instituto de Química, Departamento de Química Inorgânica, Universidade Federal do Rio de Janeiro, 21945-970 Rio de Janeiro, RJ, Brazil

Correspondence e-mail: cg@st-andrews.ac.uk

Key indicators

Single-crystal X-ray study
T = 120 K
Mean σ(C–C) = 0.006 Å
R factor = 0.060
wR factor = 0.124
Data-to-parameter ratio = 9.5

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

The title compound, $C_{20}H_{20}N_4O_4$, crystallizes with two molecules in the asymmetric unit. Each independent molecule exhibits approximate twofold rotation symmetry, but conformational differences between the molecules preclude any higher symmetry.

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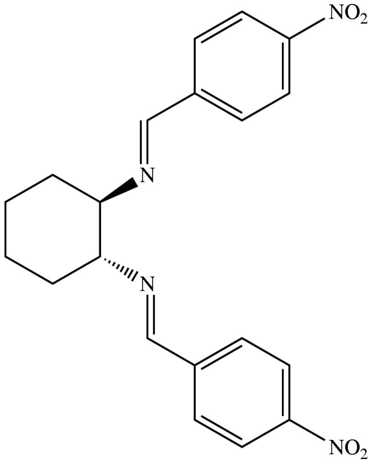
Comment

Following on from our study of the crystal structures of the diimines, *N*,*N*′-bis(4-nitrobenzylidene)ethane-1,2-diamine and -propane-1,3-diamine, obtained from 4-nitrobenzaldehyde and the appropriate α,ω-diaminoalkanes (Bomfim *et al.*, 2005), we have now studied the structure of the title compound, (I), in which the functional groups are on adjacent carbon sites in a carbocyclic ring.



(I)

In the two independent molecules of compound (I) (Fig. 1), the corresponding bond distances and interbond angles are very similar, showing no unusual values. There are clear differences between the single C—N and double C═N bonds (Table 1). The four independent chains which are pendent from the cyclohexane rings all occupy equatorial sites (Fig. 1) and all have fairly similar conformations (Table 1), such that each molecule has approximate, although not exact, twofold rotational symmetry. The biggest difference between the two independent molecules arises from the torsion angles between the nitro groups on the benzene rings, 9.8 (2) and 17.6 (2)° in molecule *A*, but only 0.8 (2) and 1.3 (2)° in molecule *B*. These differences suffice to preclude the possibility of any additional crystallographic symmetry. There is only one direction-specific interaction between the molecules (Table 2) which might be of significance. Otherwise, the structure consists of isolated pseudosymmetric molecules.

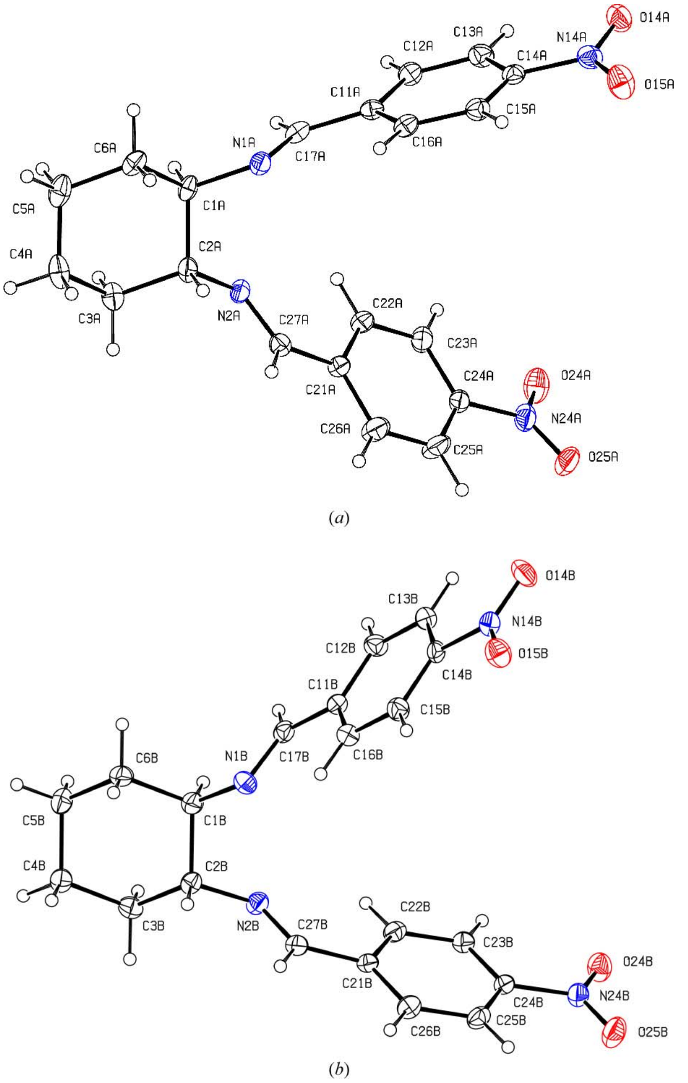


Figure 1
The two independent molecules of compound (I), showing the atom-labelling scheme for (*a*) molecule *A* and (*b*) molecule *B*. Displacement ellipsoids are drawn at the 30% probability level.

Experimental

A mixture of (1*R*,2*R*)-cyclohexane-1,2-diamine (0.228 g, 2 mmol) and 4-nitrobenzaldehyde (0.604 g, 4 mmol) in MeOH (30 ml) was heated under reflux for 30 min. The solution was then cooled to ambient temperature and the solvent was removed. The resulting solid was recrystallized from aqueous ethanol (m.p. 381–382 K). IR (KBr disk) (ν, cm^{-1}): 2924, 2853, 1644, 1602, 1521, 1347, 1140, 1107, 936, 860, 829, 747, 690, 584, 484, 444.

Crystal data

$C_{20}H_{20}N_4O_4$
$M_r = 380.40$
Monoclinic, $C2$
$a = 52.275$ (3) Å
$b = 5.3605$ (2) Å
$c = 14.0209$ (8) Å
$\beta = 104.952$ (2)°
$V = 3795.9$ (3) Å^3
$Z = 8$
$D_x = 1.331$ Mg m^{-3}
Mo $K\alpha$ radiation
Cell parameters from 4774 reflections
$\theta = 3.0$–27.7°
$\mu = 0.10$ mm^{-1}
$T = 120$ (2) K
Needle, yellow
0.38 × 0.06 × 0.04 mm

Data collection

Bruker Nonius KappaCCD area-detector diffractometer
φ and ω scans
Absorption correction: multi-scan (*SADABS*; Sheldrick, 2003)
$T_{\min} = 0.956$, $T_{\max} = 0.996$
19 060 measured reflections
4774 independent reflections
2943 reflections with $I > 2\sigma(I)$
$R_{\rm int} = 0.086$
$\theta_{\max} = 27.7°$
$h = -67 \rightarrow 67$
$k = -6 \rightarrow 6$
$l = -18 \rightarrow 18$

Refinement

Refinement on F^2
$R[F^2 > 2\sigma(F^2)] = 0.060$
$wR(F^2) = 0.124$
$S = 1.02$
4774 reflections
505 parameters
H-atom parameters constrained
$w = 1/[\sigma^2(F_o^2) + (0.0388P)^2 + 2.286P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{\max} < 0.001$
$\Delta\rho_{\max} = 0.23$ e Å^{-3}
$\Delta\rho_{\min} = -0.26$ e Å^{-3}
Absolute structure: Friedel pairs merged

Table 1
Selected geometric parameters (Å, °).

C1*A*—N1*A*	1.468 (5)	C1*B*—N1*B*	1.448 (5)
C2*A*—N2*A*	1.467 (5)	C2*B*—N2*B*	1.460 (5)
N1*A*—C17*A*	1.254 (5)	N1*B*—C17*B*	1.262 (5)
N2*A*—C27*A*	1.277 (5)	N2*B*—C27*B*	1.269 (5)
C2*A*—C1*A*—N1*A*—C17*A*	108.4 (4)	C2*B*—C1*B*—N1*B*—C17*B*	136.7 (4)
C1*A*—N1*A*—C17*A*—C11*A*	−176.0 (4)	C1*B*—N1*B*—C17*B*—C11*B*	177.1 (4)
N1*A*—C17*A*—C11*A*—C12*A*	−178.8 (4)	N1*B*—C17*B*—C11*B*—C12*B*	−176.5 (4)
C13*A*—C14*A*—N14*A*—O14*A*	10.2 (6)	C13*B*—C14*B*—N14*B*—O14*B*	−2.0 (6)
C1*A*—C2*A*—N2*A*—C27*A*	137.6 (4)	C1*B*—C2*B*—N2*B*—C27*B*	122.2 (4)
C2*A*—N2*A*—C27*A*—C21*A*	−173.2 (3)	C2*B*—N2*B*—C27*B*—C21*B*	−173.6 (3)
N2*A*—C27*A*—C21*A*—C26*A*	−172.5 (4)	N2*B*—C27*B*—C21*B*—C26*B*	177.6 (4)
C23*A*—C24*A*—N24*A*—O24*A*	−15.9 (6)	C23*B*—C24*B*—N24*B*—O24*B*	−2.5 (5)

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

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
C15*B*—H15*B*···O25*A*$^{\rm i}$	0.95	2.48	3.392 (6)	161

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

All H atoms were located in difference maps and then treated as riding atoms with C—H distances of 0.95 (aromatic and CH—), 0.99 (CH$_2$) or 1.00 Å (aliphatic CH), and with $U_{\rm iso}$(H) = 1.2$U_{\rm eq}$(C). In the absence of significant anomalous scattering, Friedel pairs were merged prior to the final refinement, and the absolute configuration was set according to the known absolute configuration of the (1*R*,2*R*)-1,2-diaminocyclohexane employed in the synthesis.

Data collection: *COLLECT* (Hooft, 1999); cell refinement: *DENZO* (Otwinowski & Minor, 1997) and *COLLECT*; data reduction: *DENZO* and *COLLECT*; program(s) used to solve structure: *SHELXS97* (Sheldrick, 1997); program(s) used to refine structure: *OSCAIL* (McArdle, 2003) and *SHELXL97* (Sheldrick, 1997); molecular graphics: *PLATON* (Spek, 2003); software used to prepare material for publication: *SHELXL97* and *PRPKAPPA* (Ferguson, 1999).

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