

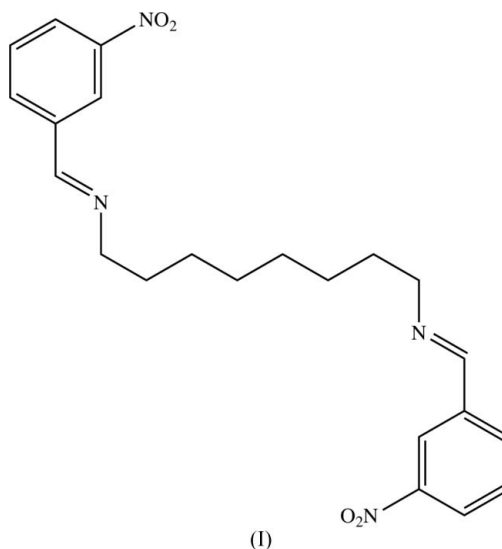
Sheets of π -stacked centrosymmetric molecules in
N,N'-bis(3-nitrobenzylidene)octane-1,8-diamineChristopher Glidewell,^{a*} John N. Low,^b Janet M. S. Skakle^b and James L. Wardell^c^aSchool of Chemistry, University of St Andrews, Fife KY16 9ST, Scotland, ^bDepartment of Chemistry, University of Aberdeen, Meston Walk, Old Aberdeen AB24 3UE, Scotland, and ^cInstituto de Química, Departamento de Química Inorgânica, Universidade Federal do Rio de Janeiro, 21945-970 Rio de Janeiro, RJ, Brazil

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Key indicators

Single-crystal X-ray study
 $T = 120$ K
Mean $\sigma(\text{C}-\text{C}) = 0.003$ Å
 R factor = 0.058
 wR factor = 0.158
Data-to-parameter ratio = 17.7For details of how these key indicators were automatically derived from the article, see <http://journals.iucr.org/e>.In molecules of the title compound, $\text{C}_{22}\text{H}_{26}\text{N}_4\text{O}_4$, which lie across centres of inversion, the central octane fragment adopts a nearly planar all-*trans* conformation. There are no hydrogen bonds in the crystal structure but the molecules are linked into sheets by a single π - π stacking interaction.

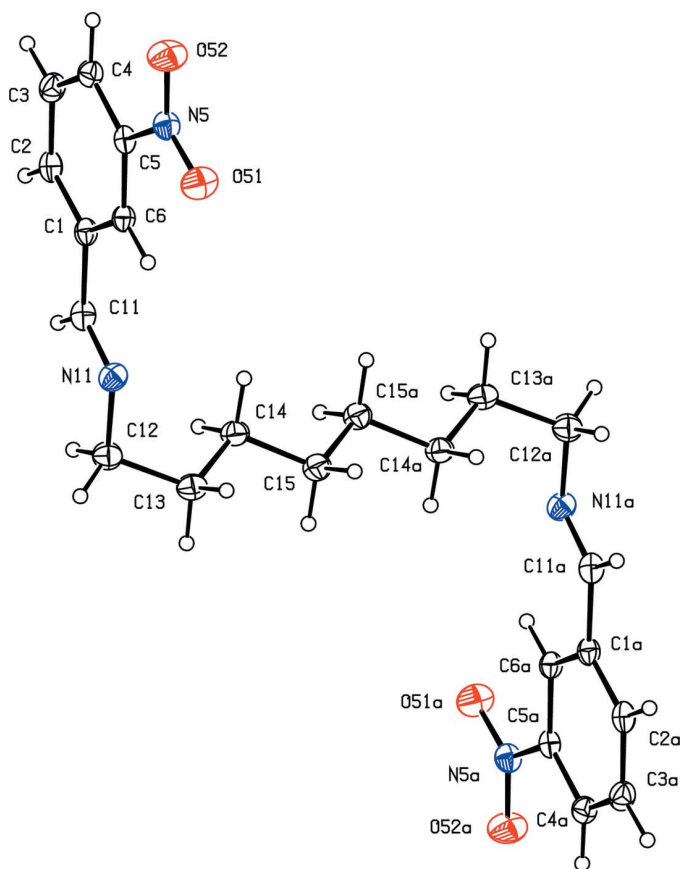
Comment

As part of our continuing studies of the supramolecular arrangements in imines, and especially in compounds of the type $\text{O}_2\text{NC}_6\text{H}_4\text{CH}=\text{N}-(R)-\text{N}=\text{CHC}_6\text{H}_4\text{NO}_2$, where $R = 1,2\text{-cyclo-C}_6\text{H}_{10}$ (Glidewell, Low, Skakle & Wardell, 2005; Glidewell, Low & Wardell, 2005) or $R = (\text{CH}_2)_n$ (Bomfim *et al.*, 2005), we now report the molecular and supramolecular structure of the title compound, (I).The molecules of (I) lie across centres of inversion in the space group $P2_1/c$, with the reference molecule selected as that lying across $(\frac{1}{2}, \frac{1}{2}, \frac{1}{2})$ (Fig. 1). The bond distances (Table 1) clearly show the presence of the double bond $\text{C11}=\text{N11}$, with typical skeletal bond angles at C11 and N11. The nitroaryl $-\text{CH}=\text{N}-\text{C}$ fragment is almost planar, as is the eight-carbon fragment of the central spacer, as shown by the leading torsional angles (Table 1); however, the overall molecular conformation is very far from being planar, as shown by the torsion angles around the $\text{N11}-\text{C11}$ and $\text{C12}-\text{C13}$ bonds, where the non-H substituents are mutually anticlinal and synclinal respectively (Table 1 and Fig. 1).The supramolecular aggregation is very simple; there are no hydrogen bonds of any kind, but a single aromatic π - π stacking interaction links the molecules into sheets. The aryl

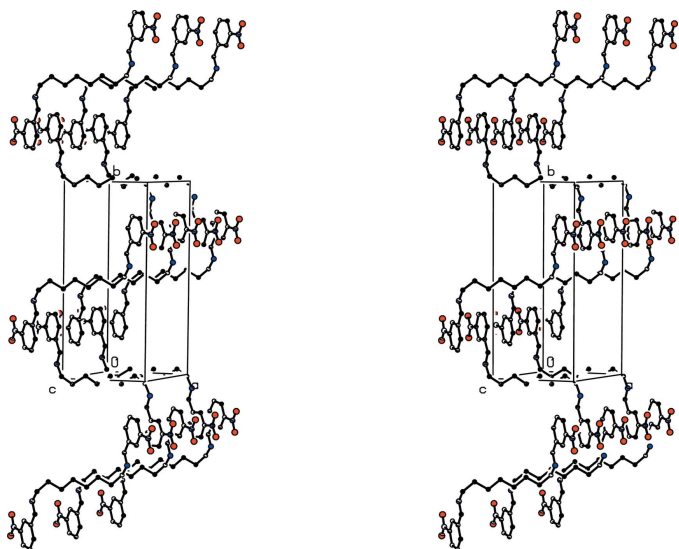
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Figure 1

The molecule of compound (I), showing the atom-labelling scheme. The atoms marked 'a' are at the symmetry position $(1-x, 1-y, 1-z)$ and displacement ellipsoids are drawn at the 30% probability level.


Figure 2

Stereoview of part of the crystal structure of compound (I), showing the formation of a π -stacked (100) sheet. For the sake of clarity, the H atoms have been omitted.

ring at (x, y, z) , which is part of the molecule centred across $(\frac{1}{2}, \frac{1}{2}, \frac{1}{2})$, is almost parallel with the aryl rings at $(x, \frac{1}{2} - y, \frac{1}{2} + z)$ and $(x, \frac{1}{2} - y, -\frac{1}{2} + z)$, which form parts of the molecules centred

across $(\frac{1}{2}, 0, 1)$ and $(\frac{1}{2}, 0, 0)$, respectively. The dihedral angle between adjacent rings is only $2.5(2)^\circ$, with the ring-centroid separations both $3.762(2) \text{ \AA}$; the interplanar spacings are *ca* 3.39 \AA and the ring-centroid offsets are *ca* 1.63 \AA . Propagation of this single interaction by the space group symmetry then generates a (100) sheet in which each molecule is linked to four others (Fig. 2); there are, however, no direction-specific interactions between adjacent sheets.

Experimental

A solution of 3-nitrobenzaldehyde (0.4 mmol) and 1,8-diaminooctane (0.2 mmol) in methanol (20 ml) was heated under reflux for 1 h; the mixture was cooled and the solvent was removed under reduced pressure. The solid residue was recrystallized from 1,2-dichloroethane to yield crystals of compound (I) suitable for single-crystal X-ray diffraction (m.p. 359–361 K). IR (KBr): 3086, 2933–2832, 1646, 1610, 1580, 1536, 1468, 1439, 1343, 1270, 1155, 1078, 1027, 971, 983, 931, 828, 805, 734, 684, 675, 629, 508 cm^{-1} .

Crystal data

$\text{C}_{22}\text{H}_{26}\text{N}_4\text{O}_4$
 $M_r = 410.47$
 Monoclinic, $P2_1/c$
 $a = 8.1297(5) \text{ \AA}$
 $b = 19.2452(15) \text{ \AA}$
 $c = 7.4113(4) \text{ \AA}$
 $\beta = 115.321(4)^\circ$
 $V = 1048.15(12) \text{ \AA}^3$
 $Z = 2$

$D_x = 1.301 \text{ Mg m}^{-3}$
 Mo $K\alpha$ radiation
 Cell parameters from 2407 reflections
 $\theta = 3.2\text{--}27.6^\circ$
 $\mu = 0.09 \text{ mm}^{-1}$
 $T = 120(2) \text{ K}$
 Plate, yellow
 $0.35 \times 0.24 \times 0.06 \text{ mm}$

Data collection

Bruker–Nonius KappaCCD diffractometer
 φ and ω scans
 Absorption correction: multi-scan (SADABS; Sheldrick, 2003)
 $T_{\min} = 0.978$, $T_{\max} = 0.995$
 11275 measured reflections

2407 independent reflections
 1284 reflections with $I > 2\sigma(I)$
 $R_{\text{int}} = 0.070$
 $\theta_{\max} = 27.6^\circ$
 $h = -10 \rightarrow 10$
 $k = -24 \rightarrow 24$
 $l = -9 \rightarrow 9$

Refinement

Refinement on F^2
 $R[F^2 > 2\sigma(F^2)] = 0.058$
 $wR(F^2) = 0.158$
 $S = 1.01$
 2407 reflections
 136 parameters

H-atom parameters constrained
 $w = 1/[\sigma^2(F_o^2) + (0.0804P)^2]$
 where $P = (F_o^2 + 2F_c^2)/3$
 $(\Delta/\sigma)_{\max} < 0.001$
 $\Delta\rho_{\max} = 0.30 \text{ e \AA}^{-3}$
 $\Delta\rho_{\min} = -0.30 \text{ e \AA}^{-3}$

Table 1

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

C1–C11	1.476 (3)	N11–C12	1.455 (2)
C11–N11	1.268 (2)		
C1–C11–N11	122.55 (18)	C11–N11–C12	117.28 (17)
C2–C1–C11–N11	−178.68 (19)	C12–C13–C14–C15	−175.69 (16)
C1–C11–N11–C12	−179.34 (18)	C13–C14–C15–C15 ⁱ	176.9 (2)
C11–N11–C12–C13	113.8 (2)	C4–C5–N5–O51	−177.60 (19)
N11–C12–C13–C14	−64.9 (2)		

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

All H atoms were located in difference maps and then treated as riding atoms, with C–H distances of 0.95 (aromatic) or 0.99 \AA (aliphatic), and with $U_{\text{iso}}(\text{H}) = 1.2U_{\text{eq}}(\text{C})$.

Data collection: *COLLECT* (Hooft, 1999); cell refinement: *DENZO* (Otwinowski & Minor, 1997) and *COLLECT*; data reduction: *DENZO* and *COLLECT*; program(s) used to solve structure: *OSCAIL* (McArdle, 2003) and *SHELXS97* (Sheldrick, 1997); program(s) used to refine structure: *OSCAIL* 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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