Acta Crystallographica Section E
Structure Reports Online
ISSN 1600-5368

N,*N*′-Bis[(3,4-methylenedioxyphenyl)methylene]phenylene-1,4-diamine

Shu-Ping Yang,[a]* Han Li-Jun,[b] Wang Da-Qi[c] and Ding Tie-Zhu[d]

[a]Department of Chemical Engineering, Huaihai Institute of Technology, Lianyungang 222005, People's Republic of China, [b]Department of Mathematics and Science, Huaihai Institute of Technology, Lianyungang 222005, People's Republic of China, [c]College of Chemistry and Chemical Engineering, Liaocheng University, Shandong 252059, People's Republic of China, and [d]Department of Physics, Inner Mongolia University, Hohhot 010021, People's Republic of China

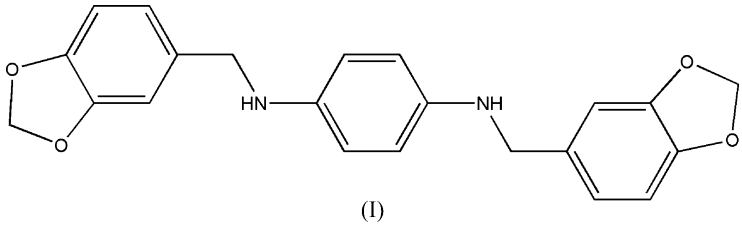
Correspondence e-mail: yangshuping@hhit.edu.cn

Received 28 November 2006
Accepted 6 December 2006

In the title compound, $C_{22}H_{20}N_2O_4$, the molecule is centrosymmetric. The molecules are linked by one C—H···O and one C—H···π interaction into an [001] chain of $R_2^2(30)$ rings and an [010] chain of $R_2^2(22)$ rings.

Comment

As part of our study of diamine compounds, in this paper we describe the crystal structure of the title compound, (I).



(I)

The molecule of (I) (Fig. 1), is disposed about a centre of symmetry at ($\frac{1}{2}$, $\frac{1}{2}$, $\frac{1}{2}$). The angle N1—C4—C5 is 113.8 (2)°, and the dihedral angle between the 3,4-methylenedioxyphenyl ring and the benzene ring C1–C3/C1*A*–C3*A* is 78.73 (7)°.

In the crystal structure of (I), C—H···O interactions link the molecules into a chain of $R_2^2(30)$ rings (Bernstein *et al.*, 1995) running parallel to the [001] direction (Fig. 2, Table 1). The molecules are further linked by C—H···π interactions [C4···*Cg*1^{ii} = 3.94 Å and C4—H4···*Cg*1^{ii} = 136°, where *Cg*1 is the centroid of ring C5–C10; symmetry code: (ii) *x*, 1 + *y*, *z*] (Fig. 3), and the propagation by inversion of this hydrogen bond generates a chain of $R_2^2(22)$ rings (García-Báez *et al.*, 2002) running parallel to the [010] direction (Fig. 3). The combination of the [001] chain and the [010] chain generates a (100) sheet. Neighbouring sheets are connected by van der Waals forces, resulting in a three-dimensional network structure.

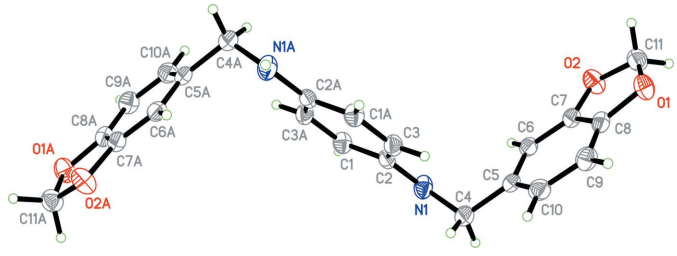


Figure 1
The molecular structure of (I), showing the atom-labelling scheme. Displacement ellipsoids are drawn at the 30% probability level. [Symmetry code: (A) 1 − *x*, 1 − *y*, 1 − *z*.]

Key indicators

Single-crystal X-ray study
T = 298 K
Mean σ(C–C) = 0.003 Å
R factor = 0.047
wR factor = 0.136
Data-to-parameter ratio = 12.1

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

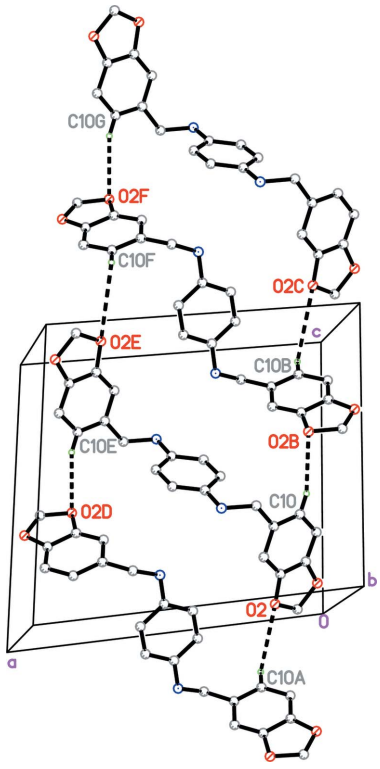


Figure 2
A view of part of the crystal structure of (I), showing the formation of a chain of $R_2^2(30)$ rings along the [001] direction. Displacement ellipsoids are drawn at the 30% probability level. For clarity, H atoms not involved in the motif shown have been omitted [symmetry codes: (A) $x, \frac{3}{2}-y, -\frac{1}{2}+z$; (B) $x, \frac{3}{2}-y, \frac{1}{2}+z$; (C) $x, y, 1+z$; (D) $1+x, -\frac{3}{2}+y, \frac{1}{2}-z$; (E) $1-x, 1-y, 1-z$; (F) $1+x, -\frac{3}{2}+y, \frac{3}{2}-z$; (G) $1-x, 1-y, 2-z$]. Dashed lines indicate hydrogen bonds.

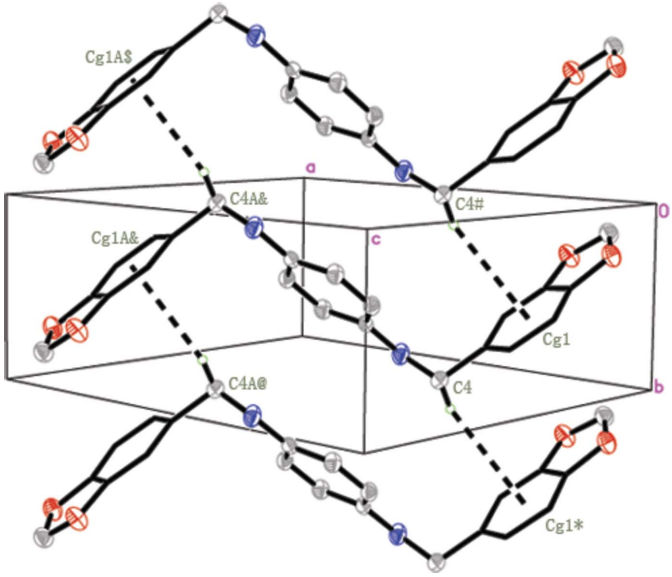


Figure 3
A view of part of the crystal structure of (I), showing the formation of the the [010] chain of $R_2^2(22)$ rings linked by C—H$\cdots\pi$ interactions. Displacement ellipsoids are drawn at the 30% probability level. For clarity, bonds on ring C5–C10 are shown as thin lines and H atoms not involved in the motif shown have been omitted [symmetry codes: (*) $x, 1+y, z$; (#) $x, -1+y, z$; (&) $1-x, 1-y, 1-z$; ($) $1-x, -y, 1-z$; (@) $1-x, -1-y, 1-z$]. Dashed lines indicate hydrogen bonds.

Experimental

A mixture of *N*,*N*′-bis(3,4-methylenedioxybenzyl)phenylene-1,4-diamine (3.72 g, 10 mmol) and sodium borohydride (1.52 g, 40 mmol) was refluxed for about 2 h in ethanol (50 ml). The mixture was cooled, and the product filtered off, washed with ethanol and dried. Yellow crystals of (I) suitable for X-ray structure analysis were obtained by recrystallizing the crude product from ethanol (m.p. 453–455 K).

Crystal data

$C_{22}H_{20}N_2O_4$
M_r = 376.40
Monoclinic, $P2_1/c$
a = 14.2255 (14) Å
b = 4.9257 (16) Å
c = 12.7837 (18) Å
β = 101.624 (3)°
V = 877.4 (3) Å^3
Z = 2
D_x = 1.425 Mg m^{-3}
Mo $K\alpha$ radiation
μ = 0.10 mm^{-1}
T = 298 (2) K
Plate, yellow
0.50 × 0.41 × 0.13 mm

Data collection

Siemens SMART 1000 CCD area-detector diffractometer
φ and ω scans
Absorption correction: multi-scan (*SADABS*; Sheldrick, 1996)
T_{min} = 0.952, T_{max} = 0.987
4222 measured reflections
1532 independent reflections
1037 reflections with $I > 2\sigma(I)$
R_{int} = 0.041
θ_{max} = 25.0°

Refinement

Refinement on F^2
$R[F^2 > 2\sigma(F^2)]$ = 0.047
$wR(F^2)$ = 0.136
S = 1.02
1532 reflections
127 parameters
H-atom parameters constrained
$w = 1/[\sigma^2(F_o^2) + (0.067P)^2 + 0.1742P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} < 0.001$
$\Delta\rho_{max}$ = 0.30 e Å^{-3}
$\Delta\rho_{min}$ = −0.24 e Å^{-3}

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

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
C10—H10···O2^{i}	0.93	2.76	3.657 (3)	162

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

All H atoms were positioned geometrically and refined as riding on their parent atoms, with N—H = 0.86 Å and U_{iso}(H) = 1.2U_{eq}(N) for amino H atoms, and C—H = 0.93–0.97 Å and U_{iso}(H) = 1.2U_{eq}(C) for all other H atoms.

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*.

We acknowledge the financial support of the Huaihai Institute of Technology Science Foundation.

References

Bernstein, J., Davis, R. E., Shimoni, L. & Chang, N.-L. (1995). *Angew. Chem. Int. Ed. Engl.* **34**, 1555–1573.
García-Báez, E. V., Martínez-Martínez, F. J., Höpfl, H. & Padilla-Martínez, I. I. (2002). *Cryst. Growth Des.* **3**, 34–45.

Sheldrick, G. M. (1996). *SADABS*. University of Göttingen, Germany.
Sheldrick, G. M. (1997*a*). *SHELXS97* and *SHELXL97*. University of Göttingen, Germany.
Sheldrick, G. M. (1997*b*). *SHELXTL*. Version 5.1. Bruker AXS Inc., Madison, Wisconsin, USA.
Siemens (1996). *SMART* and *SAINT*. Siemens Analytical X-ray Instruments Inc., Madison, Wisconsin, USA.