Acta Crystallographica Section E
Structure Reports Online
ISSN 1600-5368

N,*N*′-(2-Hydroxy-3-methoxybenzyl)benzene-1,4-diamine

H.-T. Xia,[a]* Y.-F. Liu,[a] S.-P. Yang[a] and D.-Q. Wang[b]

[a]Department of Chemical Engineering, Huaihai Institute of Technology, Lianyungang 222005, People's Republic of China, and [b]College of Chemistry and Chemical Engineering, Liaocheng University, Shandong 252059, People's Republic of China

Correspondence e-mail: xht161006@hhit.edu.cn

Received 5 November 2006
Accepted 17 November 2006

The title compound, $C_{22}H_{24}N_2O_4$, crystallizes with an inversion centre at the mid-point of the central benzene ring. Molecules are linked into sheets by N—H···O and O—H···N hydrogen bonds, leading to fused $R_2^2(4)$ rings, which form sheets parallel to the (010) plane.

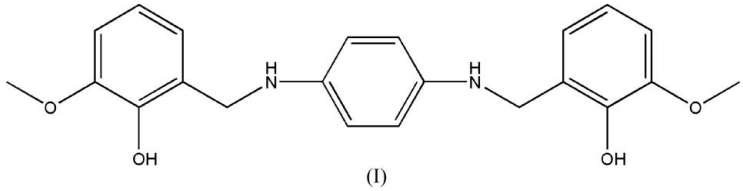
Key indicators

Single-crystal X-ray study
T = 298 K
Mean σ(C–C) = 0.007 Å
R factor = 0.059
wR factor = 0.201
Data-to-parameter ratio = 13.4

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

Comment

As part of our investigation of the crystal structures of diamine derivatives, we report here the crystal structure of a new diamine, *N*,*N*′-(2-hydroxy-3-methoxybenzyl)benzene-1,4-diamine, (I) (Fig. 1).



(I)

The molecule of (I) has an inversion centre at the mid-point of the central benzene ring. Molecules are linked into sheets involving $R_2^2(4)$ rings (Bernstein *et al.*, 1995) through N—H···O and O—H···N hydrogen bonds (Fig. 2); neighbouring sheets are connected by van der Waals forces (Fig. 3), thereby linking the molecules into a three-dimensional network structure.

Experimental

Solutions of *N*,*N*′-bis(2-hydroxy-3-methoxybenzylene)benzene-1,4-diamine (10 mmol) in methanol–chloroform (1:1 *v*/*v*) (20 ml) and $NaBH_4$ (40 mmol) were mixed. A solution of *N*,*N*′-bis(2-hydroxy-3-methoxybenzylene)benzene-1,4-diamine (10 mmol) in methanol–chloroform (1:1 *v*/*v*) (20 ml) and $NaBH_4$ (40 mmol, solid) was added.The resulting solution was stirred at room temperature for 30 h and then filtered. The solution was allowed to stand to produce crystals of (I) slowly.

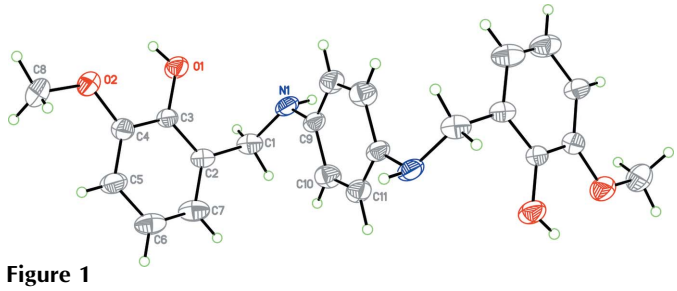


Figure 1
The molecular structure of (I), showing the atom-labelling scheme. Displacement ellipsoids are at the 30% probability level. Unlabelled atoms are related to labelled atoms by $1-x, 1-y, 1-z$.

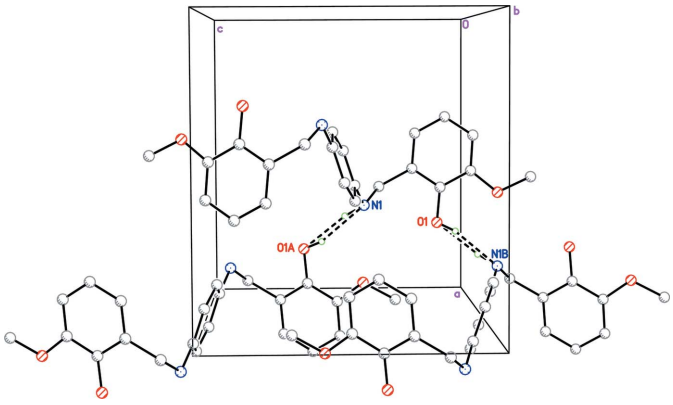


Figure 2
A portion of the crystal structure of (I), showing the formation of a hydrogen-bonded sheet built from C—H···O and O—H···N hydrogen bonds. For clarity, H atoms not involved in the hydrogen bonding have been omitted. Dashed lines indicate hydrogen bonds. [Symmetry codes: (A) $\frac{3}{2} - x$, y, 1/2+z; (B) $\frac{3}{2} - x$, y, $-\frac{1}{2} + z$.]

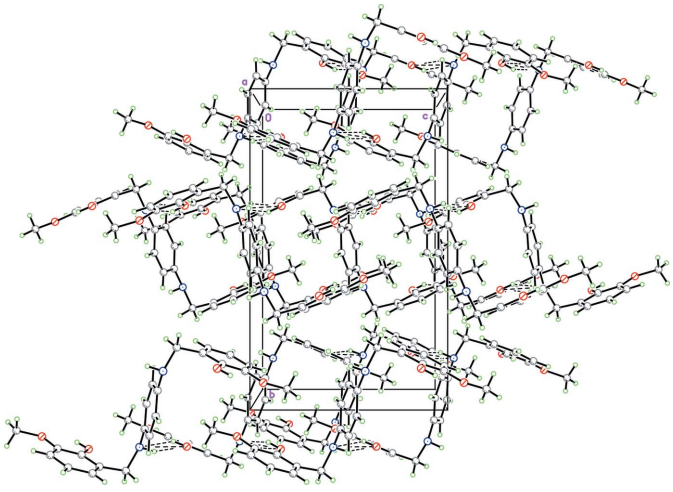


Figure 3
A packing diagram of (I).

Crystal data

$C_{22}H_{24}N_2O_4$
M_r = 380.43
Orthorhombic, *Pccn*
a = 11.308 (2) Å
b = 16.736 (3) Å
c = 10.325 (2) Å
V = 1954.0 (6) Å^3
Z = 4
D_x = 1.293 Mg m^{-3}
Mo $K\alpha$ radiation
μ = 0.09 mm^{-1}
T = 298 (2) K
Block, yellow
0.19 × 0.18 × 0.15 mm

Data collection

Bruker SMART CCD area-detector diffractometer
φ and ω scans
Absorption correction: multi-scan (*SADABS*; Sheldrick, 1996)
T_{min} = 0.983, T_{max} = 0.987
9222 measured reflections
1702 independent reflections
753 reflections with $I > 2\sigma(I)$
R_{int} = 0.094
θ_{max} = 25.0°

Refinement

Refinement on F^2
$R[F^2 > 2\sigma(F^2)]$ = 0.059
$wR(F^2)$ = 0.201
S = 1.03
1702 reflections
127 parameters
H-atom parameters constrained
$w = 1/[\sigma^2(F_o^2) + (0.0746P)^2 + 0.9056P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} < 0.001$
$\Delta\rho_{max}$ = 0.34 e Å^{-3}
$\Delta\rho_{min}$ = −0.37 e Å^{-3}

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

D—H···A	D—H	H···A	D···A	D—H···A
O1—H1···N1^{i}	0.82	2.15	2.890 (7)	149
N1—H1A···O1^{ii}	0.90	2.04	2.890 (7)	157

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

All H atoms were located in difference Fourier maps and were treated as riding atoms, with C—H = 0.93 (aryl), 0.96 (methyl) or 0.97 Å (methylene), O—H = 0.82 Å (hydroxy) and N—H = 0.90 Å (amine), and with U_{iso}(H) = 1.2U_{eq}(C,N) for aryl, methylene and NH H atoms or 1.5U_{eq}(C,O) for methyl and hydroxy H atoms.

Data collection: *SMART* (Siemens, 1996); cell refinement: *SMART*; data reduction: *SAINT* (Siemens, 1996); 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 financial support from the Liaocheng University Science Foundation.

References

Bernstein, J., Davis, R. E., Shimoni, L. & Chang, N.-L. (1995). *Angew. Chem. Int. Ed. Engl.* **34**, 1555–1573.
Sheldrick, G. M. (1996). *SADABS*. University of Göttingen, Germany.
Sheldrick, G. M. (1997*a*). *SHELXL97* and *SHELXS97*. University of Göttingen, Germany.
Sheldrick, G. M. (1997*b*). *SHELXTL*. Version 5.10. Bruker AXS Inc., Madison, Wisconsin, USA.
Siemens. (1996). *SMART* and *SAINT*. Siemens Analytical X-ray Systems, Inc., Madison, Wisconsin, USA.