

## 1,6-Bis(2-aminophenoxy)hexane

Jie Cai, Xin-Chen Zhu,\* Yun-Qian Zhang and Bi-Xue Zhu

Department of Chemistry, Guizhou University, Guiyang 550025, People's Republic of China

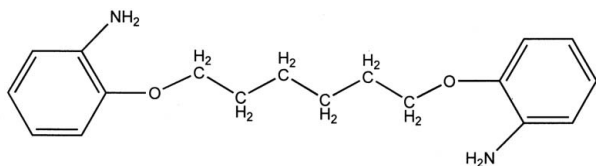
Correspondence e-mail: sci.bxzhu@gzu.edu.cn

Received 22 July 2007; accepted 30 July 2007

 Key indicators: single-crystal X-ray study;  $T = 293$  K; mean  $\sigma(\text{C}-\text{C}) = 0.002$  Å;  $R$  factor = 0.036;  $wR$  factor = 0.109; data-to-parameter ratio = 14.7.

In the structure of the title compound,  $\text{C}_{14}\text{H}_{24}\text{N}_2\text{O}_2$ , the two benzene rings are linked by a 1,6-dioxyhexyl chain to form a centrosymmetric nonplanar structure, the dihedral angle between the two rings being  $0.00$  ( $9$ )°. The structure displays an intramolecular  $\text{N}-\text{H}\cdots\text{O}$  hydrogen bond and the molecular packing is controlled by  $\text{N}-\text{H}\cdots\pi$  and  $\text{C}-\text{H}\cdots\pi$  interactions.

### Related literature

 For related literature, see: Lacroix (2001); Sabater *et al.* (2001).


### Experimental

#### Crystal data

$\text{C}_{14}\text{H}_{24}\text{N}_2\text{O}_2$   
 $M_r = 300.39$   
 Monoclinic,  $P2_1/c$   
 $a = 8.9200$  (13) Å  
 $b = 6.4935$  (9) Å  
 $c = 14.464$  (2) Å  
 $\beta = 95.378$  (6)°

$V = 834.1$  (2) Å<sup>3</sup>  
 $Z = 2$   
 Mo  $K\alpha$  radiation  
 $\mu = 0.08$  mm<sup>-1</sup>  
 $T = 293$  (2) K  
 $0.17 \times 0.13 \times 0.09$  mm

#### Data collection

Bruker APEXII CCD area-detector diffractometer  
 Absorption correction: multi-scan (*SADABS*; Bruker, 2002)  
 $T_{\min} = 0.987$ ,  $T_{\max} = 0.993$   
 6624 measured reflections  
 1488 independent reflections  
 1260 reflections with  $I > 2\sigma(I)$   
 $R_{\text{int}} = 0.021$

#### Refinement

$R[F^2 > 2\sigma(F^2)] = 0.036$   
 $wR(F^2) = 0.109$   
 $S = 1.09$   
 1488 reflections  
 101 parameters  
 H-atom parameters constrained  
 $\Delta\rho_{\max} = 0.20$  e Å<sup>-3</sup>  
 $\Delta\rho_{\min} = -0.17$  e Å<sup>-3</sup>

**Table 1**

Hydrogen-bond geometry (Å, °).

Cg1 is the centroid of the C1–C6 benzene ring.

$D-\text{H}\cdots A$	$D-\text{H}$	$\text{H}\cdots A$	$D\cdots A$	$D-\text{H}\cdots A$
$\text{N1}-\text{H1B}\cdots\text{O1}$	0.86	2.27	2.6097 (15)	104
$\text{N1}-\text{H1A}\cdots\text{Cg1}^{\text{ii}}$	0.86	2.64	3.3641 (15)	142
$\text{C9}-\text{H9A}\cdots\text{Cg1}^{\text{iii}}$	0.97	3.01	3.7718 (15)	137

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

Data collection: *APEX2* (Bruker, 2005); cell refinement: *SAINT* (Bruker, 2002); data reduction: *SAINT*; program(s) used to solve structure: *SHELXS97* (Sheldrick, 1997); program(s) used to refine structure: *SHELXL97* (Sheldrick, 1997); molecular graphics: *ORTEP-3 for Windows* (Farrugia, 1997); software used to prepare material for publication: *WinGX* (Farrugia, 1999).

The authors acknowledge the support of the Natural Science Foundation of Guizhou Province (grant No.20052011).

Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: RK2030).

### References

- Bruker (2002). *SAINT* and *SADABS*. Bruker AXS, Inc., Madison, Wisconsin, USA.  
 Bruker (2005). *APEX2*. Version 1.22. Bruker AXS, Inc., Madison, Wisconsin, USA.  
 Farrugia, L. J. (1997). *J. Appl. Cryst.* **30**, 565.  
 Farrugia, L. J. (1999). *J. Appl. Cryst.* **32**, 837–838.  
 Lacroix, P. G. (2001). *Eur. J. Inorg. Chem.* pp. 339–348.  
 Sabater, M. J., Alvaro, M., Garcia, H., Palomares, E. & Scaiano, J. C. (2001). *J. Am. Chem. Soc.* **123**, 7074–7080.  
 Sheldrick, G. M. (1997). *SHELXS97* and *SHELXL97*. University of Göttingen, Germany.

**supplementary materials**

*Acta Cryst.* (2007). E63, o3710 [ doi:10.1107/S1600536807037324 ]

## 1,6-Bis(2-aminophenoxy)hexane

J. Cai, X.-C. Zhu, Y.-Q. Zhang and B.-X. Zhu

### Comment

Diamine compounds not only are the materials of preparing dyes, paints, oil dope, but also are the important intermediate of synthesizing Schiff base compounds. Recently, Schiff base metal complexes have been widely investigated for their properties and applications in different fields, such as catalysis (Sabater *et al.*, 2001) and materials chemistry (Lacroix, 2001).

In the crystal structure of the title compound (I), the two phenyl rings are linked by 1,6-dioxyhexyl chain with form a non-coplanar structure (Fig. 1). Both of the benzene rings in the same molecules are parallel one another. The molecule placed in the center of symmetry position in the middle of C9—C9<sup>i</sup> bond [symmetry codes: (i)  $1 - x, -y, 1 - z$ ]. In the crystal, strong N—H $\cdots$  $\pi$  and C—H $\cdots$  $\pi$  interactions occur between adjacent molecules, with an N1—H1A $\cdots$ Cg(1)<sup>ii</sup> angle of 142.07°, H1A $\cdots$ Cg(1)<sup>ii</sup> distance of 2.6439 Å, N1 $\cdots$ Cg(1)<sup>ii</sup> distances of 3.3641 (15)Å and C9—H9A $\cdots$ Cg(1)<sup>iii</sup> angle of 136.68°, H9A $\cdots$ Cg(1)<sup>iii</sup> distance of 3.0068 Å, C9 $\cdots$ Cg(1)<sup>iii</sup> distances of 3.7718 (15) Å, respectively. Cg(1)<sup>ii</sup> and Cg(1)<sup>iii</sup> are the centroid of the C1/C6-benzene rings [symmetry codes: (ii)  $-x, 1/2 + y, 1/2 - z$ ; (iii)  $1 - x, -1/2 + y, 1/2 - z$ ]. Simultaneously, the structure displays intramolecular N1—H1B $\cdots$ O1 hydrogen bonding with parameters: N1—H1B = 0.86 Å, H1B $\cdots$ O1 = 2.27 Å, N $\cdots$ O = 2.609 (15) Å, and N—H $\cdots$ O = 103.69°.

### Experimental

Here, the title compound (I) [1,6-bis(2-aminophenoxy)-hexane] was prepared from 1,6-hexanediol, *p*-toluenesulfonyl chloride and *o*-hydroxyaniline: *p*-toluenesulfonyl chloride (7.62 g, 40 mmol) was added slowly, whilst stirring, to a pyridine solution (50 ml) containing 1,6-hexanediol (2.36 g, 20 mmol). The mixture was stirred for about 4 h in the range of 268–278 K. Water (40 ml) was added to the resulting solution, the precipitate was collected by filtration, the solid product was crystallized using ethanol. The solid product (0.852 g, 2 mmol) dissolved in DMF (100 ml) containing K<sub>2</sub>CO<sub>3</sub> (2 g), *o*-hydroxyaniline(0.38 g, 4 mmol) was added slowly, to the DMF solution, and the mixture was heated at 353 K for 10 h, and then the solvent was removed under reduced pressure. The crude product was purified by column chromatography over silica gel using 80% dichloromethane–hexane to afford pure crystals (I), 0.413 g, in a yield of 82%. Single crystals suitable for X-ray diffraction were obtained from an ethanol–CH<sub>2</sub>Cl<sub>2</sub> mixture by slow evaporation at room temperature.

### Refinement

All H atoms were placed in calculated positions and refined as riding, with C—H = 0.93–0.97 Å, N—H = 0.86 Å, and with  $U_{\text{iso}}(\text{H}) = 1.2 U_{\text{eq}}(\text{C}, \text{N})$ .

## Figures

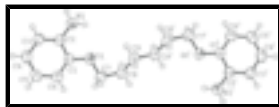


Fig. 1. The molecular structure of (I), shown with 50% probability displacement ellipsoids. H atoms are shown as spheres of arbitrary radius.

## 1,6-Bis(2-aminophenoxy)hexane

### Crystal data

$C_{18}H_{24}N_2O_2$

$M_r = 300.39$

Monoclinic,  $P2_1/c$

Hall symbol: -P 2ybc

$a = 8.9200$  (13) Å

$b = 6.4935$  (9) Å

$c = 14.464$  (2) Å

$\beta = 95.378$  (6)°

$V = 834.1$  (2) Å<sup>3</sup>

$Z = 2$

$F_{000} = 324$

$D_x = 1.196$  Mg m<sup>-3</sup>

Mo  $K\alpha$  radiation

$\lambda = 0.71073$  Å

Cell parameters from 1488 reflections

$\theta = 1.0$ – $25.1$ °

$\mu = 0.08$  mm<sup>-1</sup>

$T = 293$  (2) K

Prism, colourless

$0.17 \times 0.13 \times 0.09$  mm

### Data collection

Bruker APEXII CCD area-detector diffractometer

Radiation source: fine-focus sealed tube

Monochromator: graphite

$T = 293$ (2) K

$\varphi$  and  $\omega$  scans

Absorption correction: multi-scan (SADABS; Bruker, 2002)

$T_{\min} = 0.987$ ,  $T_{\max} = 0.993$

6624 measured reflections

1488 independent reflections

1260 reflections with  $I > 2\sigma(I)$

$R_{\text{int}} = 0.021$

$\theta_{\max} = 25.1$ °

$\theta_{\min} = 2.3$ °

$h = -10 \rightarrow 10$

$k = -7 \rightarrow 7$

$l = -17 \rightarrow 17$

### Refinement

Refinement on  $F^2$

Least-squares matrix: full

$R[F^2 > 2\sigma(F^2)] = 0.036$

$wR(F^2) = 0.109$

$S = 1.09$

1488 reflections

101 parameters

Hydrogen site location: inferred from neighbouring sites

H-atom parameters constrained

$$w = 1/[\sigma^2(F_o^2) + (0.059P)^2 + 0.0859P]$$

where  $P = (F_o^2 + 2F_c^2)/3$

$(\Delta/\sigma)_{\max} < 0.001$

$\Delta\rho_{\max} = 0.20$  e Å<sup>-3</sup>

$\Delta\rho_{\min} = -0.17$  e Å<sup>-3</sup>

Extinction correction: SHELXL97 (Sheldrick, 1997),

$$F_c^* = kFc[1 + 0.001x Fc^2 \lambda^3 / \sin(2\theta)]^{-1/4}$$

Primary atom site location: structure-invariant direct methods Extinction coefficient: 0.030 (5)  
 Secondary atom site location: difference Fourier map

*Special details*

**Geometry.** All e.s.d.'s (except the e.s.d. in the dihedral angle between two l.s. planes) are estimated using the full covariance matrix. The cell e.s.d.'s are taken into account individually in the estimation of e.s.d.'s in distances, angles and torsion angles; correlations between e.s.d.'s in cell parameters are only used when they are defined by crystal symmetry. An approximate (isotropic) treatment of cell e.s.d.'s is used for estimating e.s.d.'s involving l.s. planes.

**Refinement.** Refinement of  $F^2$  against ALL reflections. The weighted  $R$ -factor  $wR$  and goodness of fit  $S$  are based on  $F^2$ , conventional  $R$ -factors  $R$  are based on  $F$ , with  $F$  set to zero for negative  $F^2$ . The threshold expression of  $F^2 > 2\sigma(F^2)$  is used only for calculating  $R$ -factors(gt) *etc.* and is not relevant to the choice of reflections for refinement.  $R$ -factors based on  $F^2$  are statistically about twice as large as those based on  $F$ , and  $R$ -factors based on ALL data will be even larger.

*Fractional atomic coordinates and isotropic or equivalent isotropic displacement parameters ( $\text{\AA}^2$ )*

	$x$	$y$	$z$	$U_{\text{iso}}^*/U_{\text{eq}}$
C3	0.91315 (15)	0.3758 (2)	0.14192 (9)	0.0571 (4)
H3	0.9703	0.4957	0.1438	0.069*
C2	0.84506 (13)	0.31610 (19)	0.21986 (8)	0.0467 (3)
C1	0.76042 (13)	0.13443 (19)	0.21573 (8)	0.0467 (3)
C6	0.74420 (16)	0.0200 (2)	0.13524 (10)	0.0611 (4)
H6	0.6869	-0.0998	0.1326	0.073*
C5	0.81346 (18)	0.0835 (3)	0.05801 (9)	0.0701 (4)
H5	0.8027	0.0059	0.0038	0.084*
C4	0.89747 (17)	0.2600 (2)	0.06157 (9)	0.0667 (4)
H4	0.9440	0.3021	0.0098	0.080*
C7	0.62464 (16)	-0.1047 (2)	0.30452 (10)	0.0633 (4)
H7A	0.6828	-0.2159	0.2809	0.076*
H7B	0.5271	-0.1002	0.2688	0.076*
C8	0.60592 (16)	-0.1384 (2)	0.40572 (10)	0.0650 (4)
H8A	0.7047	-0.1372	0.4400	0.078*
H8B	0.5630	-0.2741	0.4130	0.078*
C9	0.50761 (14)	0.0188 (2)	0.44852 (9)	0.0597 (4)
H9A	0.4082	0.0166	0.4151	0.072*
H9B	0.5498	0.1548	0.4411	0.072*
N1	0.85419 (15)	0.43023 (19)	0.30098 (8)	0.0683 (4)
H1A	0.9039	0.5439	0.3046	0.082*
H1B	0.8102	0.3875	0.3478	0.082*
O1	0.70143 (10)	0.08709 (14)	0.29728 (6)	0.0582 (3)

*Atomic displacement parameters ( $\text{\AA}^2$ )*

	$U^{11}$	$U^{22}$	$U^{33}$	$U^{12}$	$U^{13}$	$U^{23}$
C3	0.0590 (8)	0.0561 (8)	0.0569 (8)	-0.0084 (6)	0.0092 (6)	0.0084 (6)
C2	0.0455 (6)	0.0477 (7)	0.0470 (7)	0.0010 (5)	0.0049 (5)	0.0012 (5)
C1	0.0449 (6)	0.0505 (7)	0.0453 (7)	-0.0008 (5)	0.0082 (5)	0.0019 (5)

## supplementary materials

---

C6	0.0674 (8)	0.0588 (9)	0.0572 (8)	-0.0141 (7)	0.0066 (6)	-0.0049 (6)
C5	0.0882 (10)	0.0784 (11)	0.0442 (7)	-0.0108 (8)	0.0090 (7)	-0.0077 (7)
C4	0.0778 (9)	0.0782 (10)	0.0460 (7)	-0.0066 (8)	0.0150 (6)	0.0084 (7)
C7	0.0674 (9)	0.0529 (8)	0.0723 (9)	-0.0095 (6)	0.0202 (7)	0.0051 (7)
C8	0.0628 (8)	0.0605 (9)	0.0741 (9)	-0.0016 (6)	0.0191 (7)	0.0182 (7)
C9	0.0514 (7)	0.0659 (9)	0.0635 (8)	-0.0007 (6)	0.0135 (6)	0.0191 (7)
N1	0.0852 (8)	0.0604 (8)	0.0620 (7)	-0.0191 (6)	0.0209 (6)	-0.0143 (6)
O1	0.0653 (6)	0.0570 (6)	0.0556 (6)	-0.0130 (4)	0.0225 (4)	-0.0005 (4)

### Geometric parameters (Å, °)

C3—C4	1.3800 (19)	C7—O1	1.4300 (16)
C3—C2	1.3850 (17)	C7—C8	1.505 (2)
C3—H3	0.9300	C7—H7A	0.9700
C2—N1	1.3836 (16)	C7—H7B	0.9700
C2—C1	1.3988 (17)	C8—C9	1.515 (2)
C1—O1	1.3707 (14)	C8—H8A	0.9700
C1—C6	1.3773 (18)	C8—H8B	0.9700
C6—C5	1.3888 (19)	C9—C9 <sup>i</sup>	1.528 (2)
C6—H6	0.9300	C9—H9A	0.9700
C5—C4	1.368 (2)	C9—H9B	0.9700
C5—H5	0.9300	N1—H1A	0.8600
C4—H4	0.9300	N1—H1B	0.8600
C4—C3—C2	121.04 (12)	C8—C7—H7A	110.3
C4—C3—H3	119.5	O1—C7—H7B	110.3
C2—C3—H3	119.5	C8—C7—H7B	110.3
N1—C2—C3	123.02 (12)	H7A—C7—H7B	108.5
N1—C2—C1	118.46 (11)	C7—C8—C9	114.87 (12)
C3—C2—C1	118.50 (11)	C7—C8—H8A	108.5
O1—C1—C6	126.01 (12)	C9—C8—H8A	108.5
O1—C1—C2	113.68 (10)	C7—C8—H8B	108.5
C6—C1—C2	120.31 (11)	C9—C8—H8B	108.5
C1—C6—C5	120.02 (13)	H8A—C8—H8B	107.5
C1—C6—H6	120.0	C8—C9—C9 <sup>i</sup>	113.49 (14)
C5—C6—H6	120.0	C8—C9—H9A	108.9
C4—C5—C6	120.14 (13)	C9 <sup>i</sup> —C9—H9A	108.9
C4—C5—H5	119.9	C8—C9—H9B	108.9
C6—C5—H5	119.9	C9 <sup>i</sup> —C9—H9B	108.9
C5—C4—C3	119.99 (12)	H9A—C9—H9B	107.7
C5—C4—H4	120.0	C2—N1—H1A	120.0
C3—C4—H4	120.0	C2—N1—H1B	120.0
O1—C7—C8	107.19 (12)	H1A—N1—H1B	120.0
O1—C7—H7A	110.3	C1—O1—C7	118.92 (10)
C4—C3—C2—N1	-178.13 (13)	C1—C6—C5—C4	-0.2 (2)
C4—C3—C2—C1	0.4 (2)	C6—C5—C4—C3	-0.2 (2)
N1—C2—C1—O1	-3.00 (16)	C2—C3—C4—C5	0.1 (2)
C3—C2—C1—O1	178.37 (10)	O1—C7—C8—C9	63.87 (16)
N1—C2—C1—C6	177.78 (12)	C7—C8—C9—C9 <sup>i</sup>	-179.39 (14)

C3—C2—C1—C6	-0.85 (19)	C6—C1—O1—C7	4.97 (19)
O1—C1—C6—C5	-178.37 (12)	C2—C1—O1—C7	-174.19 (11)
C2—C1—C6—C5	0.7 (2)	C8—C7—O1—C1	166.55 (11)

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

*Hydrogen-bond geometry* ( $\text{\AA}, ^\circ$ )

$D-H\cdots A$	$D-H$	$H\cdots A$	$D\cdots A$	$D-H\cdots A$
N1—H1B $\cdots$ O1	0.86	2.27	2.6097 (15)	104

*Selected  $\pi$ -(phenyl) interaction parameters* ( $\text{\AA}, ^\circ$ )

H $\cdots$ Cg <sup>ii</sup>	2.6439	N $\cdots$ Cg <sup>ii</sup>	3.3641 (15)	N—H $\cdots$ Cg <sup>ii</sup>	142.07
H $\cdots$ Cg <sup>iii</sup>	3.0068	C $\cdots$ Cg <sup>iii</sup>	3.7718 (15)	C—H $\cdots$ Cg <sup>iii</sup>	136.68

Symmetry codes: (ii)  $-x, 1/2 + y, 1/2 - z$ ; (iii)  $1 - x, -1/2 + y, 1/2 - z$ .

Fig. 1

