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## Structure Reports

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## 1,4-Bis(4-aminophenoxy)butane

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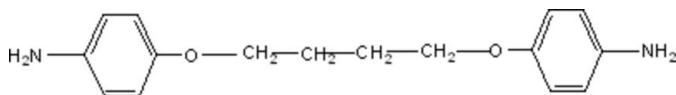
Received 7 August 2007; accepted 7 August 2007

Key indicators: single-crystal X-ray study;  $T = 173$  K; mean  $\sigma(\text{C}-\text{C}) = 0.001$  Å;  $R$  factor = 0.036;  $wR$  factor = 0.099; data-to-parameter ratio = 20.5.

Molecules of the title compound,  $\text{C}_{16}\text{H}_{20}\text{N}_2\text{O}_2$ , reside across crystallographic centres of inversion with one half-molecule in the asymmetric unit. The bond lengths and angles are normal and the crystal packing is stabilized by  $\text{N}-\text{H}\cdots\text{N}$  and  $\text{N}-\text{H}\cdots\text{O}$  hydrogen bonds.

## Related literature

For related literature, see: Butt *et al.* (2005); Chol *et al.* (2001); Eastmond *et al.* (1996); Jung & Park (1996); Liaw *et al.* (1998); Sroog (1991); Yang *et al.* (2000).



## Experimental

## Crystal data

$\text{C}_{16}\text{H}_{20}\text{N}_2\text{O}_2$   
 $M_r = 272.34$   
 Orthorhombic, *Pbca*  
 $a = 5.2638$  (4) Å  
 $b = 13.6385$  (8) Å  
 $c = 20.4415$  (11) Å

$V = 1467.50$  (16) Å<sup>3</sup>  
 $Z = 4$   
 Mo  $K\alpha$  radiation  
 $\mu = 0.08$  mm<sup>-1</sup>  
 $T = 173$  (2) K  
 $0.42 \times 0.39 \times 0.27$  mm

## Data collection

Stoe IPDS II two-circle diffractometer  
 Absorption correction: none  
 18475 measured reflections

2051 independent reflections  
 1847 reflections with  $I > 2\sigma(I)$   
 $R_{\text{int}} = 0.043$

## Refinement

$R[F^2 > 2\sigma(F^2)] = 0.036$   
 $wR(F^2) = 0.099$   
 $S = 1.04$   
 2051 reflections  
 100 parameters

H atoms treated by a mixture of independent and constrained refinement  
 $\Delta\rho_{\text{max}} = 0.27$  e Å<sup>-3</sup>  
 $\Delta\rho_{\text{min}} = -0.18$  e Å<sup>-3</sup>

Table 1

Hydrogen-bond geometry (Å, °).

$D-H\cdots A$	$D-H$	$H\cdots A$	$D\cdots A$	$D-H\cdots A$
$\text{N1}-\text{H1A}\cdots\text{N1}^{\text{i}}$	0.911 (14)	2.302 (14)	3.1895 (9)	164.6 (11)
$\text{N1}-\text{H1B}\cdots\text{O1}^{\text{ii}}$	0.886 (14)	2.334 (14)	3.1790 (11)	159.3 (11)

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

Data collection: *X-AREA* (Stoe & Cie, 2001); cell refinement: *X-AREA*; data reduction: *X-AREA*; program(s) used to solve structure: *SHELXS97* (Sheldrick, 1990); program(s) used to refine structure: *SHELXL97* (Sheldrick, 1997); molecular graphics: *XP* in *SHELXTL-Plus* (Sheldrick, 1991); software used to prepare material for publication: *SHELXL97*.

The authors are grateful to the Department of Chemistry, Quaid-i-Azam University, Islamabad, Pakistan.

Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: GG2035).

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**supplementary materials**

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## 1,4-Bis(4-aminophenoxy)butane

T. Naz, Z. Akhter, M. Bolte and H. M. Siddiqi

### Comment

Aromatic polyamides are considered as one of the most important classes of polymers because they have excellent thermal, mechanical and electrical properties (Chol *et al.*, 2001), as well as outstanding chemical resistance (Sroog, 1991). However, their applications are often limited due to poor solubility, partially due to strong interchain interactions (Butt *et al.*, 2005). To overcome these limitations, many efforts have been made to improve the processability of the polyamides while maintaining their thermal and mechanical properties (Chol *et al.*, 2001). For example, bulky lateral substituents (Yang *et al.*, 2000), flexible alkyl side chains (Jung & Park, 1996), non-coplanar biphenyl groups, and flexible alkyl or aryl ether spacers (Liaw *et al.*, 1998) have been used to enhance solubility and thus process ability. Incorporation of flexible segments such as –O–, –SO<sub>2</sub>–, –CH<sub>2</sub>– and –C(CF<sub>3</sub>)<sub>2</sub>–, and of bulky pendant groups such as *tert*-butyl and adamantyl, were found to be successful in altering crystallinity and intermolecular interactions to increase solubility (Eastmond *et al.*, 1996). Bulky pendant groups increase the disorder in chains and hinder dense chain packing which enhance the solubility. Many efforts have been made in the design and synthesis of new diamines. The title compound (I) is the result of an attempt to prepare soluble and processable organic based aromatic polyamides and ferrocene containing polyamides.

### Experimental

Synthesis of the organic diamine TN4 consists of two steps. Step-1: (N4) A mixture of 1.40 ml (0.015 mol) of 1,4-butanediol, 4.38 g (0.0318 moles) anhydrous K<sub>2</sub>CO<sub>3</sub> and 5 g (0.0318 moles) *p*-nitrochlorobenzene in 80 ml of DMF was heated at 120°C for 24 h under a N<sub>2</sub> atmosphere. When the reaction was over the mixture was poured into 500 ml distilled water to form light yellow precipitat, which was collected by filtration and washed several times thoroughly with water. The crude product was recrystallized from ethanol. *M.p.* 132°C, Yield = 80%.

Step-2: A two neck flask was charged with (N4) 1 g, 10 mL of hydrazine monohydrate, 80 mL ethanol and 0.1 g of 5% palladium on carbon (Pd—C). The mixture was refluxed for 24 h and then filtered to remove the (Pd—C). The filtrate was concentrated on rotary evaporator to remove the solvent (ethanol). The white colored precipitates were then recrystallized from ethanol. *m.p.* = 134°C, yield 70%.

### Refinement

H atoms were found in a difference map, but those bonded to C were refined using a riding model with C—H = 0.95Å and  $U_{\text{iso}}(\text{H}) = 1.2U_{\text{eq}}(\text{C})$  [C—H = 0.99Å for the methylene groups]. The H atoms bonded to N were freely refined.

## Figures

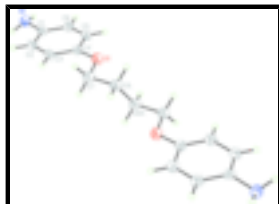


Fig. 1. Perspective view of the title compound (I) with the atom numbering; displacement ellipsoids are at the 50% probability level. Symmetry operator (A):  $-x + 1, -y + 2, -z$ .

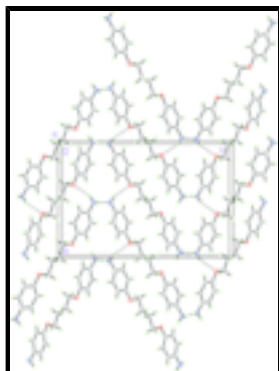


Fig. 2. Packing diagram of the title compound (I) with a view onto the  $bc$  plane. Hydrogen bonds are drawn as dashed lines.

## 1,4-Bis(4-aminophenoxy)butane

### Crystal data

$C_{16}H_{20}N_2O_2$

$M_r = 272.34$

Orthorhombic,  $Pbca$

Hall symbol:  $-P\ 2ac\ 2ab$

$a = 5.2638\ (4)\ \text{\AA}$

$b = 13.6385\ (8)\ \text{\AA}$

$c = 20.4415\ (11)\ \text{\AA}$

$V = 1467.50\ (16)\ \text{\AA}^3$

$Z = 4$

$F_{000} = 584$

$D_x = 1.233\ \text{Mg m}^{-3}$

Melting point: 407 K

Mo  $K\alpha$  radiation

$\lambda = 0.71073\ \text{\AA}$

Cell parameters from 18762 reflections

$\theta = 3.6\text{--}29.7^\circ$

$\mu = 0.08\ \text{mm}^{-1}$

$T = 173\ (2)\ \text{K}$

Block, colourless

$0.42 \times 0.39 \times 0.27\ \text{mm}$

### Data collection

Stoe IPDS II two-circle diffractometer

Radiation source: fine-focus sealed tube

Monochromator: graphite

$T = 173\ (2)\ \text{K}$

$\omega$  scans

Absorption correction: none

18475 measured reflections

2051 independent reflections

1847 reflections with  $I > 2\sigma(I)$

$R_{\text{int}} = 0.043$

$\theta_{\text{max}} = 29.6^\circ$

$\theta_{\text{min}} = 3.6^\circ$

$h = -7 \rightarrow 7$

$k = -18 \rightarrow 18$

$l = -28 \rightarrow 28$

Refinement

Refinement on $F^2$	Hydrogen site location: inferred from neighbouring sites
Least-squares matrix: full	H atoms treated by a mixture of independent and constrained refinement
$R[F^2 > 2\sigma(F^2)] = 0.036$	$w = 1/[\sigma^2(F_o^2) + (0.0511P)^2 + 0.3016P]$ where $P = (F_o^2 + 2F_c^2)/3$
$wR(F^2) = 0.099$	$(\Delta/\sigma)_{\max} < 0.001$
$S = 1.04$	$\Delta\rho_{\max} = 0.27 \text{ e } \text{\AA}^{-3}$
2051 reflections	$\Delta\rho_{\min} = -0.17 \text{ e } \text{\AA}^{-3}$
100 parameters	Extinction correction: SHELXL97, $F_c^* = kFc[1 + 0.001 \times Fc^2 \lambda^3 / \sin(2\theta)]^{-1/4}$
Primary atom site location: structure-invariant direct methods	Extinction coefficient: 0.024 (3)
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 ( $\text{\AA}^2$ )

	$x$	$y$	$z$	$U_{\text{iso}}^*/U_{\text{eq}}$
N1	0.04075 (16)	0.52438 (6)	0.20593 (4)	0.03056 (18)
H1A	-0.118 (3)	0.5273 (9)	0.2238 (6)	0.041 (3)*
H1B	0.057 (3)	0.4711 (10)	0.1814 (7)	0.042 (3)*
O1	0.36952 (14)	0.87454 (5)	0.08907 (3)	0.03265 (18)
C1	0.11977 (16)	0.61127 (6)	0.17419 (4)	0.02505 (18)
C2	0.33000 (17)	0.61024 (6)	0.13248 (4)	0.02799 (19)
H2	0.4146	0.5500	0.1242	0.034*
C3	0.41864 (17)	0.69611 (6)	0.10265 (4)	0.02789 (19)
H3	0.5601	0.6937	0.0738	0.033*
C4	0.29826 (16)	0.78512 (6)	0.11548 (4)	0.02537 (18)
C5	0.09139 (17)	0.78744 (6)	0.15830 (4)	0.02801 (19)
H5	0.0111	0.8481	0.1679	0.034*
C6	0.00208 (16)	0.70152 (6)	0.18700 (4)	0.02793 (19)
H6	-0.1401	0.7040	0.2156	0.034*
C7	0.57284 (18)	0.87461 (6)	0.04201 (4)	0.0301 (2)

## supplementary materials

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H7A	0.5284	0.8327	0.0042	0.036*
H7B	0.7305	0.8489	0.0621	0.036*
C8	0.61124 (16)	0.98013 (6)	0.01998 (4)	0.02869 (19)
H8A	0.7679	0.9841	-0.0067	0.034*
H8B	0.6355	1.0221	0.0590	0.034*

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

	$U^{11}$	$U^{22}$	$U^{33}$	$U^{12}$	$U^{13}$	$U^{23}$
N1	0.0364 (4)	0.0263 (4)	0.0290 (4)	-0.0052 (3)	0.0038 (3)	0.0005 (3)
O1	0.0411 (4)	0.0251 (3)	0.0318 (3)	-0.0013 (2)	0.0120 (3)	0.0033 (2)
C1	0.0284 (4)	0.0263 (4)	0.0205 (3)	-0.0045 (3)	-0.0032 (3)	0.0002 (3)
C2	0.0311 (4)	0.0252 (4)	0.0278 (4)	0.0007 (3)	0.0011 (3)	-0.0013 (3)
C3	0.0291 (4)	0.0278 (4)	0.0268 (4)	-0.0006 (3)	0.0043 (3)	-0.0006 (3)
C4	0.0292 (4)	0.0242 (4)	0.0227 (4)	-0.0025 (3)	-0.0006 (3)	0.0011 (3)
C5	0.0304 (4)	0.0270 (4)	0.0266 (4)	0.0028 (3)	0.0024 (3)	0.0009 (3)
C6	0.0269 (4)	0.0314 (4)	0.0254 (4)	-0.0006 (3)	0.0030 (3)	0.0015 (3)
C7	0.0312 (4)	0.0303 (4)	0.0288 (4)	-0.0011 (3)	0.0047 (3)	0.0033 (3)
C8	0.0282 (4)	0.0312 (4)	0.0266 (4)	-0.0064 (3)	0.0000 (3)	0.0032 (3)

### Geometric parameters ( $\text{\AA}$ , $^\circ$ )

N1—C1	1.4136 (11)	C4—C5	1.3975 (12)
N1—H1A	0.911 (14)	C5—C6	1.3923 (12)
N1—H1B	0.886 (14)	C5—H5	0.9500
O1—C4	1.3855 (10)	C6—H6	0.9500
O1—C7	1.4389 (10)	C7—C8	1.5214 (12)
C1—C2	1.3971 (12)	C7—H7A	0.9900
C1—C6	1.4026 (12)	C7—H7B	0.9900
C2—C3	1.4003 (12)	C8—C8 <sup>i</sup>	1.5273 (17)
C2—H2	0.9500	C8—H8A	0.9900
C3—C4	1.3942 (12)	C8—H8B	0.9900
C3—H3	0.9500		
C1—N1—H1A	114.6 (8)	C6—C5—H5	119.7
C1—N1—H1B	113.5 (8)	C4—C5—H5	119.7
H1A—N1—H1B	110.7 (12)	C5—C6—C1	120.72 (8)
C4—O1—C7	117.55 (7)	C5—C6—H6	119.6
C2—C1—C6	118.20 (7)	C1—C6—H6	119.6
C2—C1—N1	120.32 (8)	O1—C7—C8	107.30 (7)
C6—C1—N1	121.33 (8)	O1—C7—H7A	110.3
C1—C2—C3	121.42 (8)	C8—C7—H7A	110.3
C1—C2—H2	119.3	O1—C7—H7B	110.3
C3—C2—H2	119.3	C8—C7—H7B	110.3
C4—C3—C2	119.66 (8)	H7A—C7—H7B	108.5
C4—C3—H3	120.2	C7—C8—C8 <sup>i</sup>	113.08 (9)
C2—C3—H3	120.2	C7—C8—H8A	109.0
O1—C4—C3	124.76 (8)	C8 <sup>i</sup> —C8—H8A	109.0
O1—C4—C5	115.79 (7)	C7—C8—H8B	109.0

C3—C4—C5	119.45 (7)	C8 <sup>i</sup> —C8—H8B	109.0
C6—C5—C4	120.52 (8)	H8A—C8—H8B	107.8
C6—C1—C2—C3	-1.61 (12)	O1—C4—C5—C6	179.24 (8)
N1—C1—C2—C3	-177.24 (8)	C3—C4—C5—C6	-1.34 (13)
C1—C2—C3—C4	1.11 (13)	C4—C5—C6—C1	0.83 (13)
C7—O1—C4—C3	3.89 (12)	C2—C1—C6—C5	0.64 (12)
C7—O1—C4—C5	-176.71 (7)	N1—C1—C6—C5	176.22 (8)
C2—C3—C4—O1	179.75 (8)	C4—O1—C7—C8	179.14 (7)
C2—C3—C4—C5	0.38 (13)	O1—C7—C8—C8 <sup>i</sup>	-68.00 (11)

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

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

$D-H\cdots A$	$D-H$	$H\cdots A$	$D\cdots A$	$D-H\cdots A$
N1—H1A $\cdots$ N1 <sup>ii</sup>	0.911 (14)	2.302 (14)	3.1895 (9)	164.6 (11)
N1—H1B $\cdots$ O1 <sup>iii</sup>	0.886 (14)	2.334 (14)	3.1790 (11)	159.3 (11)

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

Fig. 1

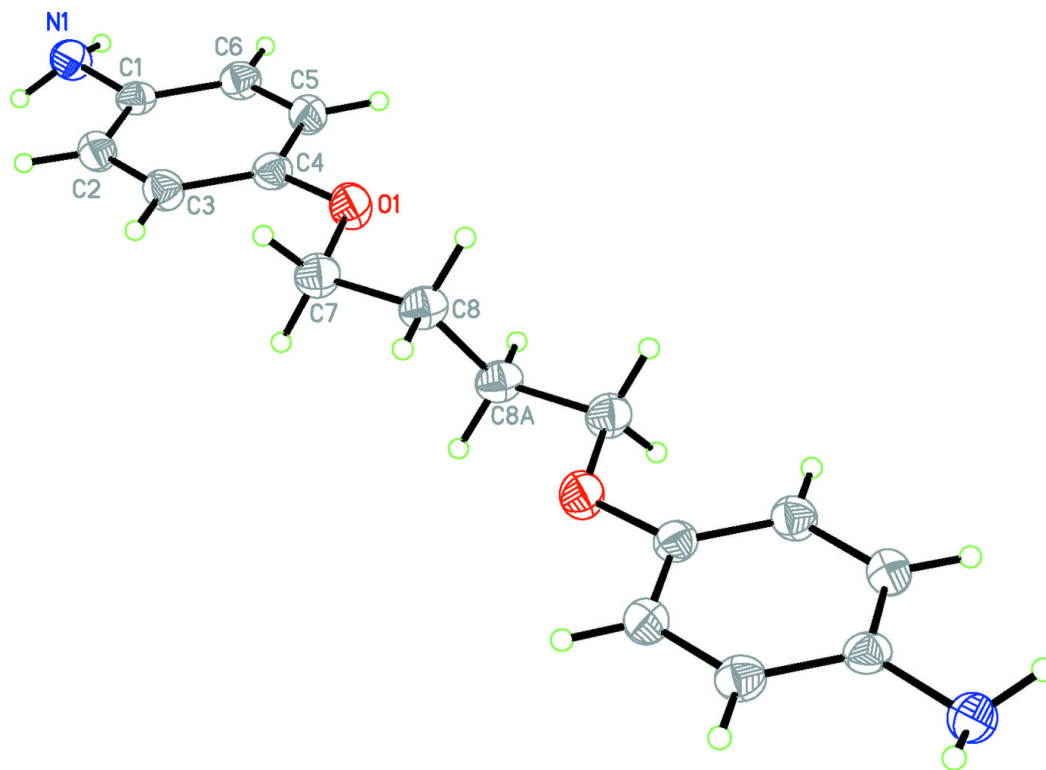




Fig. 2

