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1,2-Bis(4-aminophenoxy)ethane

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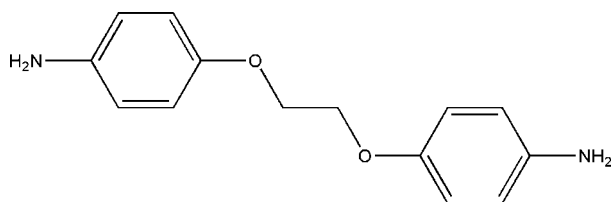
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Key indicators: single-crystal X-ray study; $T = 173$ K; mean $\sigma(\text{C}-\text{C}) = 0.002$ Å; disorder in main residue; R factor = 0.049; wR factor = 0.119; data-to-parameter ratio = 17.9.

The molecule of the title compound, $\text{C}_{14}\text{H}_{16}\text{N}_2\text{O}_2$, is located on a crystallographic twofold rotation axis. The central O—C—C—O bridge adopts a *gauche* conformation. One of the amine H atoms is disordered over two equally occupied positions. The crystal structure is stabilized by N—H···O and N—H···N hydrogen bonds.

Related literature

For related literature, see: Barikani & Mehdipour-Ataei (2000); Eastmond & Paprotny (1999); Hsio *et al.* (1997); Liaw & Liaw (2001); Yang & Chen (1993); Hergenrother *et al.* (2002).



Experimental

Crystal data

$\text{C}_{14}\text{H}_{16}\text{N}_2\text{O}_2$
 $M_r = 244.29$
Orthorhombic, *Pbcn*
 $a = 14.2157$ (9) Å

$b = 10.4608$ (8) Å
 $c = 8.1817$ (5) Å
 $V = 1216.68$ (14) Å³
 $Z = 4$

Mo $K\alpha$ radiation
 $\mu = 0.09$ mm⁻¹

$T = 173$ (2) K
 $0.37 \times 0.35 \times 0.23$ mm

Data collection

Stoe IPDSII two-circle diffractometer
Absorption correction: none
15103 measured reflections

1700 independent reflections
1549 reflections with $I > 2\sigma(I)$
 $R_{\text{int}} = 0.049$

Refinement

$R[F^2 > 2\sigma(F^2)] = 0.048$
 $wR(F^2) = 0.119$
 $S = 1.20$
1700 reflections
95 parameters

H atoms treated by a mixture of independent and constrained refinement
 $\Delta\rho_{\text{max}} = 0.29$ e Å⁻³
 $\Delta\rho_{\text{min}} = -0.19$ e Å⁻³

Table 1

Hydrogen-bond geometry (Å, °).

<i>D</i> —H··· <i>A</i>	<i>D</i> —H	H··· <i>A</i>	<i>D</i> ··· <i>A</i>	<i>D</i> —H··· <i>A</i>
N1—H1A···O1 ⁱ	0.93 (2)	2.53 (2)	3.4082 (18)	158.6 (18)
N1—H1B···N1 ⁱⁱ	0.94 (4)	2.48 (4)	3.360 (3)	157 (4)
N1—H1C···N1 ⁱⁱⁱ	0.96 (5)	2.61 (5)	3.468 (3)	148 (3)

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

Data collection: *X-AREA* (Stoe & Cie, 2001); cell refinement: *X-AREA*; data reduction: *X-AREA*; program(s) used to solve structure: *SHELXS97* (Sheldrick, 2008); program(s) used to refine structure: *SHELXL97* (Sheldrick, 2008); molecular graphics: *XP* in *SHELXTL-Plus* (Sheldrick, 2008); software used to prepare material for publication: *PLATON* (Spek, 2003).

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Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: ZL2110).

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supplementary materials

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1,2-Bis(4-aminophenoxy)ethane

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Comment

Aromatic polyimides are well accepted as high performance and heat resistant materials (Hergenrother *et al.*, 2000). They exhibit a favorable balance of physical and chemical properties, show excellent thermal, mechanical and electrical properties and are thus widely used in microelectronics and aerospace engineering (Eastmond & Paprotny, 1999). However, the technological and industrial application of rigid polyimides are limited by processing difficulties due to their high melting or glass transition temperatures and their lack of solubility in most organic solvents (Hsio *et al.*, 1997). Strong interactions between polyimide chains and their rigid structures are the main reason for these behaviors. To overcome such a drawback, different methods have been introduced to modify their structures. Many efforts have been made in designing and synthesizing new dianhydrides (Eastmond & Paprotny, 1999) and diamines (Yang & Chen, 1993), and therefore producing a great variety of more soluble and processable polyimides for various purposes and applications. Incorporation of flexible units such as $-\text{NHCO}-$, $-\text{O}-$, (Barikani & Mehdipour-Ataei, 2000), $-\text{CO}-$ and $-\text{SO}_2-$ is one of the most important approaches to overcome these processing problems (Liaw & Liaw, 2001). The title compound is such a new starting material for the synthesis of high performance polyimides.

Molecules of the title compound, $\text{C}_{14}\text{H}_{16}\text{N}_2\text{O}_4$, are located on a crystallographic twofold rotation axis. The central $\text{O}-\text{C}-\text{C}-\text{O}$ bridge adopts a *gauche* conformation. One of the amino H atoms is disordered over two equally occupied positions. As a result of that, neighbouring molecules are connected by alternating hydrogen bonds, either $\text{N1}-\text{H1B}\cdots\text{N1}^{\text{ii}}$ or $\text{N1}-\text{H1C}\cdots\text{N1}^{\text{iii}}$, because H1B and H1C and their symmetry equivalents would be too close to each other and would be mutually exclusive (symmetry codes: see Table 1). In addition, the crystal structure is stabilized by $\text{N}-\text{H}\cdots\text{O}$ hydrogen bonds (Table 1).

Experimental

A two neck 250 ml round bottom flask was charged with 1 g of 1,2-di(*p*-nitrophenyloxy) ethylene (3.28 mmoles), 10 ml of hydrazine monohydrate, 80 ml of ethanol and 0.06 g of 5% palladium on carbon (Pd/C). The mixture was heated to reflux for 16 h and then filtered to remove Pd/C and the crude solid was recrystallized from ethanol to yield 92.2% of the diamine, m.p. 352K.

Refinement

All H atoms could be located by difference Fourier synthesis but were ultimately placed in calculated positions using a riding model with $\text{C}-\text{H}(\text{aromatic}) = 0.95 \text{ \AA}$ or $\text{C}-\text{H}(\text{methylene}) = 0.99 \text{ \AA}$ with fixed individual displacement parameters [$U_{\text{iso}}(\text{H}) = 1.2 U_{\text{eq}}(\text{C})$]. The amino H atoms were freely refined. One of the amino H atoms is disordered over two equally occupied positions.

Figures

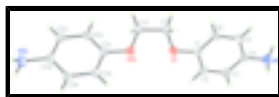


Fig. 1. The structure of the title compound showing the atomic numbering scheme. Displacement ellipsoids are drawn at the 50% probability level and only one of the two alternative types of N-H hydrogen atoms is shown. Symmetry code for generating equivalent atoms: (A) $-x+2, y, -z+3/2$.

1,2-Bis(4-aminophenoxy)ethane

Crystal data

$C_{14}H_{16}N_2O_2$

$M_r = 244.29$

Orthorhombic, *Pbcn*

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

$a = 14.2157\ (9)\ \text{\AA}$

$b = 10.4608\ (8)\ \text{\AA}$

$c = 8.1817\ (5)\ \text{\AA}$

$V = 1216.68\ (14)\ \text{\AA}^3$

$Z = 4$

$F_{000} = 520$

$D_x = 1.334\ \text{Mg m}^{-3}$

Melting point: 179 K

Mo $K\alpha$ radiation

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

Cell parameters from 14232 reflections

$\theta = 3.5\text{--}29.7^\circ$

$\mu = 0.09\ \text{mm}^{-1}$

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

Block, dark red

$0.37 \times 0.35 \times 0.23\ \text{mm}$

Data collection

Stoe IPDSII two-circle diffractometer

Radiation source: fine-focus sealed tube

Monochromator: graphite

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

ω scans

Absorption correction: none

15103 measured reflections

1700 independent reflections

1549 reflections with $I > 2\sigma(I)$

$R_{\text{int}} = 0.049$

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

$\theta_{\text{min}} = 3.5^\circ$

$h = -19 \rightarrow 19$

$k = -14 \rightarrow 11$

$l = -11 \rightarrow 11$

Refinement

Refinement on F^2

Least-squares matrix: full

$R[F^2 > 2\sigma(F^2)] = 0.048$

$wR(F^2) = 0.119$

$S = 1.20$

1700 reflections

Hydrogen site location: inferred from neighbouring sites

H atoms treated by a mixture of independent and constrained refinement

$w = 1/[\sigma^2(F_o^2) + (0.0299P)^2 + 0.7945P]$

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

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

$\Delta\rho_{\text{max}} = 0.29\ \text{e \AA}^{-3}$

$\Delta\rho_{\text{min}} = -0.19\ \text{e \AA}^{-3}$

95 parameters

Extinction correction: SHELXL97 (Sheldrick, 2008),

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

Primary atom site location: structure-invariant direct methods

Extinction coefficient: 0.024 (2)

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 $F^2 > \sigma(F^2)$ 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 (\AA^2)

	x	y	z	$U_{\text{iso}}^*/U_{\text{eq}}$	Occ. (<1)
O1	0.91391 (6)	0.60429 (10)	0.65677 (12)	0.0243 (3)	
N1	0.52550 (9)	0.65323 (15)	0.5428 (2)	0.0332 (3)	
H1A	0.5093 (15)	0.718 (2)	0.470 (3)	0.048 (6)*	
H1B	0.496 (3)	0.573 (4)	0.537 (5)	0.042 (11)*	0.50
H1C	0.486 (3)	0.650 (4)	0.638 (6)	0.048 (12)*	0.50
C1	0.81695 (9)	0.61092 (12)	0.63296 (15)	0.0198 (3)	
C2	0.78489 (9)	0.71034 (13)	0.53437 (17)	0.0232 (3)	
H2	0.8285	0.7684	0.4872	0.028*	
C3	0.68899 (10)	0.72479 (13)	0.50483 (17)	0.0239 (3)	
H3	0.6678	0.7931	0.4377	0.029*	
C4	0.62345 (9)	0.64020 (13)	0.57255 (17)	0.0234 (3)	
C5	0.65662 (10)	0.54140 (14)	0.67195 (19)	0.0268 (3)	
H5	0.6131	0.4835	0.7196	0.032*	
C6	0.75275 (10)	0.52624 (13)	0.70255 (17)	0.0240 (3)	
H6	0.7742	0.4586	0.7704	0.029*	
C7	0.94691 (9)	0.49765 (13)	0.75075 (18)	0.0244 (3)	
H7A	0.9230	0.4169	0.7033	0.029*	
H7B	0.9237	0.5043	0.8645	0.029*	

Atomic displacement parameters (\AA^2)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
O1	0.0192 (4)	0.0272 (5)	0.0264 (5)	-0.0010 (4)	-0.0026 (4)	0.0063 (4)
N1	0.0205 (6)	0.0377 (7)	0.0413 (8)	0.0012 (5)	-0.0023 (5)	0.0043 (6)
C1	0.0202 (6)	0.0214 (6)	0.0178 (5)	-0.0003 (5)	-0.0017 (4)	-0.0017 (5)
C2	0.0231 (6)	0.0230 (6)	0.0235 (6)	-0.0010 (5)	0.0019 (5)	0.0039 (5)
C3	0.0246 (6)	0.0239 (6)	0.0233 (6)	0.0036 (5)	-0.0001 (5)	0.0028 (5)
C4	0.0205 (6)	0.0254 (6)	0.0244 (6)	0.0005 (5)	-0.0010 (5)	-0.0028 (5)

supplementary materials

C5	0.0228 (6)	0.0267 (6)	0.0310 (7)	-0.0048 (5)	-0.0004 (5)	0.0043 (6)
C6	0.0247 (6)	0.0223 (6)	0.0250 (6)	-0.0023 (5)	-0.0033 (5)	0.0050 (5)
C7	0.0237 (6)	0.0233 (6)	0.0261 (6)	-0.0007 (5)	-0.0050 (5)	0.0024 (5)

Geometric parameters (\AA , $^\circ$)

O1—C1	1.3938 (15)	C3—C4	1.3992 (19)
O1—C7	1.4338 (16)	C3—H3	0.9500
N1—C4	1.4202 (18)	C4—C5	1.397 (2)
N1—H1A	0.93 (2)	C5—C6	1.3983 (19)
N1—H1B	0.94 (4)	C5—H5	0.9500
N1—H1C	0.96 (5)	C6—H6	0.9500
C1—C2	1.3928 (18)	C7—C7 ⁱ	1.510 (2)
C1—C6	1.3935 (18)	C7—H7A	0.9900
C2—C3	1.3927 (18)	C7—H7B	0.9900
C2—H2	0.9500		
C1—O1—C7	115.93 (10)	C5—C4—C3	118.27 (12)
C4—N1—H1A	115.0 (14)	C5—C4—N1	120.12 (13)
C4—N1—H1B	112 (3)	C3—C4—N1	121.61 (13)
H1A—N1—H1B	120 (3)	C4—C5—C6	121.20 (12)
C4—N1—H1C	115 (3)	C4—C5—H5	119.4
H1A—N1—H1C	114 (3)	C6—C5—H5	119.4
H1B—N1—H1C	75 (3)	C1—C6—C5	119.65 (12)
C2—C1—C6	119.80 (12)	C1—C6—H6	120.2
C2—C1—O1	116.21 (11)	C5—C6—H6	120.2
C6—C1—O1	123.98 (12)	O1—C7—C7 ⁱ	108.83 (10)
C3—C2—C1	120.12 (12)	O1—C7—H7A	109.9
C3—C2—H2	119.9	C7 ⁱ —C7—H7A	109.9
C1—C2—H2	119.9	O1—C7—H7B	109.9
C2—C3—C4	120.96 (12)	C7 ⁱ —C7—H7B	109.9
C2—C3—H3	119.5	H7A—C7—H7B	108.3
C4—C3—H3	119.5		
C7—O1—C1—C2	-176.63 (12)	C3—C4—C5—C6	-0.5 (2)
C7—O1—C1—C6	4.11 (19)	N1—C4—C5—C6	179.72 (14)
C6—C1—C2—C3	-0.3 (2)	C2—C1—C6—C5	0.4 (2)
O1—C1—C2—C3	-179.57 (12)	O1—C1—C6—C5	179.62 (13)
C1—C2—C3—C4	-0.2 (2)	C4—C5—C6—C1	0.0 (2)
C2—C3—C4—C5	0.6 (2)	C1—O1—C7—C7 ⁱ	174.05 (12)
C2—C3—C4—N1	-179.62 (14)		

Symmetry codes: (i) $-x+2, y, -z+3/2$.

Hydrogen-bond geometry (\AA , $^\circ$)

$D-H\cdots A$	$D-H$	$H\cdots A$	$D\cdots A$	$D-H\cdots A$
N1—H1A \cdots O1 ⁱⁱ	0.93 (2)	2.53 (2)	3.4082 (18)	158.6 (18)
N1—H1B \cdots N1 ⁱⁱⁱ	0.94 (4)	2.48 (4)	3.360 (3)	157 (4)
N1—H1C \cdots N1 ^{iv}	0.96 (5)	2.61 (5)	3.468 (3)	148 (3)

Symmetry codes: (ii) $x-1/2, -y+3/2, -z+1$; (iii) $-x+1, -y+1, -z+1$; (iv) $-x+1, y, -z+3/2$.

Fig. 1

