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1,2-Bis(2-aminophenoxy)ethane

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The crystal structure of 1,2-bis(2-aminophenoxy)ethane, $C_{14}H_{16}N_2O_2$, exhibits intermolecular close contacts between the amine groups and O atoms of neighbouring molecules. In addition, weak C—H···O hydrogen-bonding interactions are present. A crystallographic twofold axis bisects the central C—C bond.

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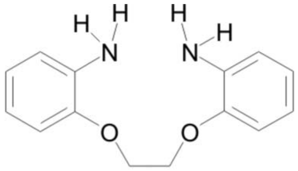
Key indicators

Single-crystal X-ray study
T = 120 K
Mean σ(C–C) = 0.003 Å
R factor = 0.061
wR factor = 0.177
Data-to-parameter ratio = 21.8

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

Comment

1,2-Bis(2-aminophenoxy)ethane, (I), is a precursor in the synthesis of bisaminophenol tetraacetic acid (BAPTA), a ligand employed in the development of molecular sensors for calcium (Tsien, 1980).



(I)

We thought that the crystal structure of (I) would be of interest as molecules containing two aromatic amine groups and two O atoms are likely to exhibit classical hydrogen bonding. In theory, a number of intramolecular and intermolecular hydrogen-bonding interactions involving the two amine groups as hydrogen-bond donors or acceptors, and the O atoms as hydrogen-bond acceptors are possible. A fundamental requirement in the field of crystal engineering, is an understanding of the non-covalent interactions present in crystal structures. In this study, an analysis of intermolecular interactions occurring in the solid state was performed.

The asymmetric unit of (I) contains one-half molecule with a twofold axis at ($\frac{1}{2}$, y, $\frac{1}{4}$) bisecting the C7—C7′ [symmetery code ('): $1 - x, y, \frac{1}{2} - z$] bond. The molecular geometry and labelling scheme are illustrated in Fig. 1. In the molecule, the atoms constituting the aromatic ring and atoms O1, N1, C7 and C7′ are coplanar with an s.u. value of 0.003 Å. The angle

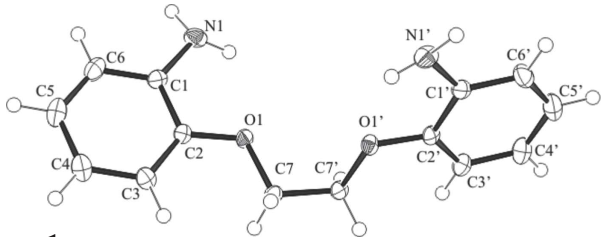


Figure 1
The molecular structure of (I), showing the atomic numbering scheme and ellipsoids at the 50% probability level for non-H atoms (*ORTEP3*; Farrugia, 1997). [Symmetery code ('): $1 - x, y, \frac{1}{2} - z$.]

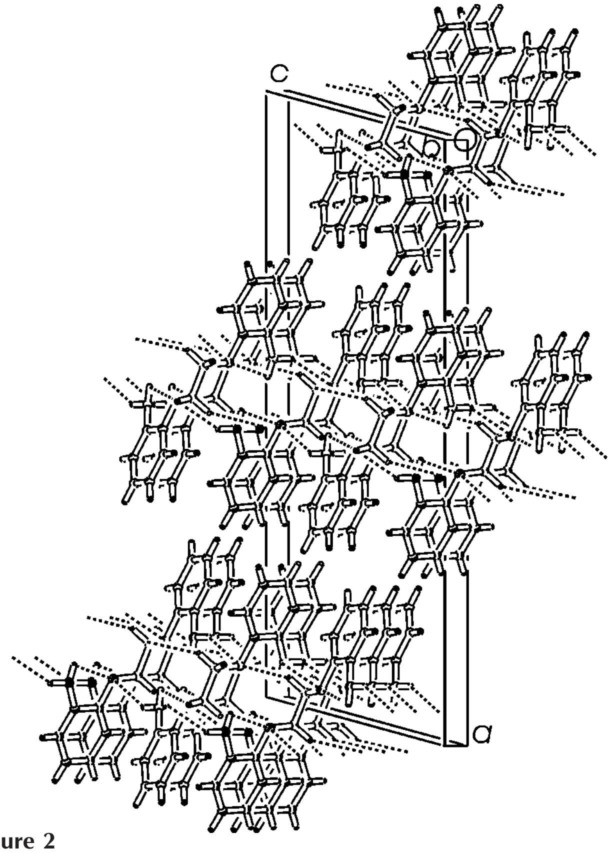


Figure 2
Packing diagram of (I), illustrating the layered structure and interactions (dashed lines) (*PLATON*; Spek, 2003).

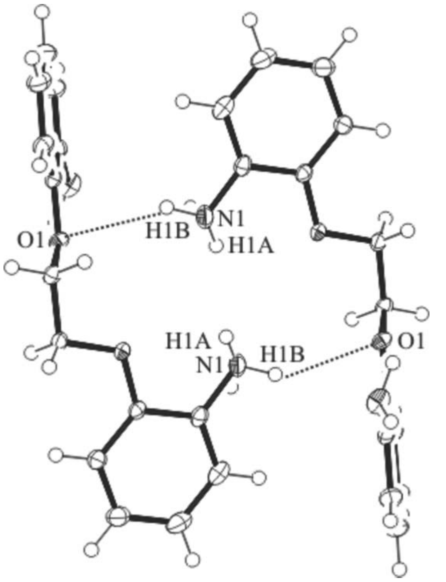


Figure 3
The zero-dimensional dimer motif (*ORTEP3*; Farrugia, 1997).

between the two aromatic ring planes is 70.3 (1)°, and the central torsion angle, O1—C7—C7′—O1′, has a value of 67.98 (14)°.

The molecular packing is illustrated in Fig. 2. A layered structure is formed, with the aromatic rings packing in two neighbouring layers, separated by a layer containing the hydrogen-bonding O and N atoms, as well as the C7 and C7' atoms.

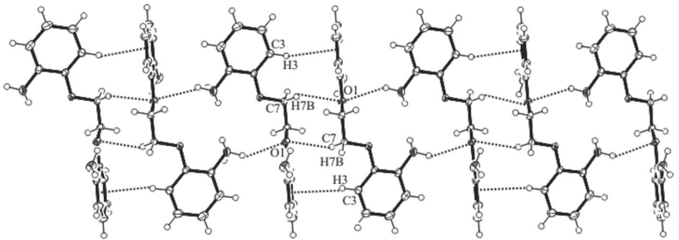


Figure 4
The one-dimensional chain motif (*ORTEP3*; Farrugia, 1997).

In a molecule, the distance between N1 and O1 of 2.641 (4) Å may suggest an intramolecular hydrogen-bonding interaction occurring through H1*A*; however, the small N1—H1*A*···O1 angle of 103.6 (17)° indicates that this is not a true classical hydrogen bond. Instead, atom N1 engages in a weak interaction through atom H1*B* to atom O1 (symmetry code: x, $-y + 1$, $z - \frac{1}{2}$). For this interaction, the angle of the O1—H1*A* bond to the plane formed by atom O1 atom and neighbouring C atoms is 77°, illustrating that the O-atom lone pair is not aligned favourably to allow a strong classical hydrogen bond. Interaction parameters are listed in Table 1.

Atom C7 interacts with atom O1 (symmetry code: x, $1 - y$, $-\frac{1}{2} + z$) through atom H7*B* [C7···O1 = 3.409 (5) Å] through a weak non-classical hydrogen-bonding interaction. In the aromatic layer, one weak edge-on C—H···π interaction is observed involving atoms C3—H3···C1—C6 (symmetry code: x, $1 - y$, $-\frac{1}{2} + z$) with a donor-to-centroid distance of 2.970 Å.

One way of describing the crystal structure of (I) is to consider a dimer consisting of two molecules interacting *via* weak interactions as the basic structural unit (Fig. 3). Only one amine group per molecule is involved in the formation of the dimer, which can be viewed as a zero-dimensional synthon for this structure. In the dimer, the two N—H···O interactions form an $R_2^2(16)$ supramolecular ring (using the notation of Etter & MacDonald, 1990). Neighbouring dimers are connected by weaker C7—H7*B*···O1 and C3—H3···C1—C6 contacts, resulting in a one-dimensional chain of dimers (Fig. 3). Adjacent chains are connected by interactions involving the second amine functional group on a molecule. Weak N—H···O interactions connect chains to form a two-dimensional sheet parallel to the crystallographic *bc* plane, as illustrated in Fig. 2. Stacking of these sheets in the *a*-axis direction produces the three-dimensional crystal structure.

Experimental

1,2-Bis(2-aminophenoxy)ethane was synthesized as previously reported (Tsien, 1980). 1,2-Bis(2-nitrophenoxy)ethane (4 g, 13 mmol) and 0.40 g of 10% palladium on carbon were stirred in ethanol (400 ml) at room temperature while hydrogen gas was bubbled through very slowly. After 6 h, the solution was filtered through celite and sodium borohydride (0.20 g, 5.3 mmol) was added to the filtrate as a decolourizing agent. The solvent was evaporated to 80 ml and the solution was allowed to cool, affording 2.35 g of *white* crystals (74%, m.p. 341–342 K). ^{1}H NMR (300 MHz, $CDCl_3$): δ 6.6–6.9 (*m*, ArH), 4.35 (*s*, OCH_2CH_2O), 3.40 (broad *s*, NH_2).

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Crystal data

$C_{14}H_{16}N_2O_2$
$M_r = 244.29$
Monoclinic, $C2/c$
$a = 26.33$ (2) Å
$b = 5.979$ (8) Å
$c = 8.163$ (10) Å
$\beta = 105.14$ (10)°
$V = 1241$ (3) Å^3
$Z = 4$
$D_x = 1.308$ Mg m^{-3}
Mo $K\alpha$ radiation
Cell parameters from 170 reflections
$\theta = 2$–32°
$\mu = 0.09$ mm^{-1}
$T = 120$ (2) K
Block, white
0.4 × 0.4 × 0.3 mm

Data collection

Oxford Excalibur2 diffractometer
ω–2θ scans
Absorption correction: none
5831 measured reflections
1962 independent reflections
1660 reflections with $I > 2\sigma(I)$
$R_{\rm int} = 0.074$
$\theta_{\rm max} = 31.7$°
$h = -38 \rightarrow 37$
$k = -8 \rightarrow 8$
$l = -11 \rightarrow 7$

Refinement

Refinement on F^2
$R[F^2 > 2\sigma(F^2)] = 0.061$
$wR(F^2) = 0.177$
$S = 1.09$
1962 reflections
90 parameters
H atoms treated by a mixture of independent and constrained refinement
$w = 1/[\sigma^2(F_o^2) + (0.1046P)^2 + 0.6144P]$
where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{\rm max} = 0.005$
$\Delta\rho_{\rm max} = 0.55$ e Å^{-3}
$\Delta\rho_{\rm min} = -0.28$ e Å^{-3}

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

D—H···A	D—H	H···A	D···A	D—H···A
N1—H1B···O1$^{\rm i}$	0.86 (2)	2.48 (3)	3.236 (3)	148 (2)

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

Both H atoms on the amine N atom were located in difference Fourier maps and refined isotropically, providing accurate atomic positions for discussion of the interactions present in the structure. All other H atoms were placed in calculated positions, with methylene C—H = 0.97 Å and aromatic C—H = 0.93 Å, and refined using a riding model, with $U_{\rm iso}({\rm H}) = 1.5U_{\rm eq}({\rm C})$.

Data collection: *CrysAlis CCD* (Oxford Diffraction, 2003); cell refinement: *CrysAlis CCD*; data reduction: *CrysAlis RED* (Oxford Diffraction, 2003); program(s) used to solve structure: *SHELXS97* (Sheldrick, 1997); program(s) used to refine structure: *SHELXL97* (Sheldrick, 1997); molecular graphics: *ORTEP3 for Windows* (Farrugia, 1997); software used to prepare material for publication: *WinGX* (Farrugia, 1999) and *PLATON* (Spek, 2003).

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