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Orthorhombic polymorph of 1,2-bis(2-aminophenoxy)ethane

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The orthorhombic polymorph of the title compound, $C_{14}H_{16}N_2O_2$, is devoid of crystallographically imposed molecular symmetry. The crystal structure is stabilized by N—H···O hydrogen-bonding interactions.

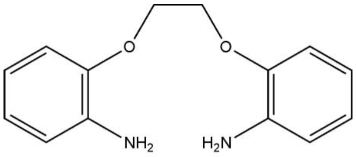
Key indicators

Single-crystal X-ray study
T = 100 K
Mean σ(C–C) = 0.003 Å
R factor = 0.033
wR factor = 0.084
Data-to-parameter ratio = 9.7

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

Comment

Amines are commonly used as starting materials in solution-phase combinatorial synthesis and are used as reagents to cleave desired compounds from resins in solid-phase combinatorial synthesis. Also, several biologically active compounds, possessing analgesic activity, are known to contain diamine units (Chang *et al.*, 1994). In transition metal chemistry, amines containing at least two N atoms are desirable since they produce quite stable complexes. Indeed, many amine-containing complexes have been synthesized and their properties (*e.g.* carcinogenic, optical, biological activities *etc.*) established (Jain *et al.*, 1994). In this study, 1,2-bis(*o*-aminophenoxy)ethane, (I), was synthesized from bis(*o*-nitrophenoxy)ethane, Pd/C and hydrazine hydrate in the presence of methanol.



(I)

Compound (I) (Fig. 1) is an orthorhombic polymorph, a monoclinic ($C2/c$) polymorph having been reported recently (Rademeyer *et al.*, 2005). The key difference between the structures is found in the molecular symmetry, there being none in the orthorhombic polymorph and crystallographically imposed twofold rotation symmetry in the monoclinic form. The geometric parameters in the two forms are in agreement. The angle between the planes of the two aromatic rings is 84.82 (8)° and the central torsion angle, O1—C7—C8—O2, has a value of −75.68 (14)°. The torsion angles about the O—CH_2 bonds are 178.74 (12)° (C9—O2—C8—C7) and −174.15 (12)° (C1—O1—C7—C8), and the torsion angles about the C_{ar}—O bonds are −8.7 (2)° (C7—O1—C1—C2) and −1.4 (2)° (C8—O2—C9—C10). In the crystal structure of (I), a three-dimensional network is formed *via* N—H···O hydrogen bonds as detailed in Table 1 and illustrated in Fig. 2.

Experimental

Bis(*o*-nitrophenoxy)ethane (0.76 g, 0.025 mol) was dissolved in methanol (100 ml) and Pd/C (0.75 g) was added. The reaction mixture was stirred on a water bath for 0.5 h. To the mixture, hydrazine hydrate (15 ml, $d = 1.03$ Mg m^{-3}, 0.3 mol) was added dropwise and it was refluxed for 2.5 h. The solution was filtered, and the solid was kept in a refrigerator overnight and recrystallized from dioxane/methanol (1:1 *v*/*v*), with evaporation under vacuum. Yield 90%, m.p. 440 K. IR (cm^{-1}): 3445 (aromatic NH$_2$), 3059 (aromatic C—H), 2884 (aliphatic C—H), 1275 (C—N), 1504 (aromatic C=C). ^{1}H NMR: δ 4.0 (*s*, CH$_2$—O), 4.1–3.4 (*s*, Ar—NH$_2$), 7.2–6.5 (*s*, aromatic protons).

Crystal data

$C_{14}H_{16}N_2O_2$
$M_r = 244.29$
Orthorhombic, $Pbn2_1$
$a = 7.0644$ (6) Å
$b = 7.5770$ (7) Å
$c = 23.8053$ (10) Å
$V = 1274.23$ (17) Å^3
$Z = 4$
$D_x = 1.273$ Mg m^{-3}
Mo $K\alpha$ radiation
$\mu = 0.09$ mm^{-1}
$T = 100$ K
Prism, colourless
0.33 × 0.30 × 0.28 mm

Data collection

Bruker–Nonius KappaCCD diffractometer
ω scans
Absorption correction: Gaussian integration (Coppens, 1970)
$T_{\min} = 0.972$, $T_{\max} = 0.976$
13155 measured reflections
1594 independent reflections
1530 reflections with $I > 2\sigma(I)$
$R_{\rm int} = 0.024$
$\theta_{\max} = 28.5°$

Refinement

Refinement on F^2
$R[F^2 > 2\sigma(F^2)] = 0.033$
$wR(F^2) = 0.084$
$S = 1.04$
1594 reflections
164 parameters
H-atom parameters constrained
$w = 1/[\sigma^2(F_o^2) + (0.0554P)^2 + 0.2427P]$
where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{\max} < 0.001$
$\Delta\rho_{\max} = 0.26$ e Å^{-3}
$\Delta\rho_{\min} = -0.19$ e Å^{-3}

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

D—H···A	D—H	H···A	D···A	D—H···A
N1—H1A···O2$^{\rm i}$	0.86	2.45	3.2512 (19)	155
N1—H1B···O2	0.86	2.30	2.6405 (19)	104
N2—H2A···O1$^{\rm ii}$	0.86	2.37	3.1602 (19)	154
N2—H2B···O1	0.86	2.31	2.6470 (19)	104

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

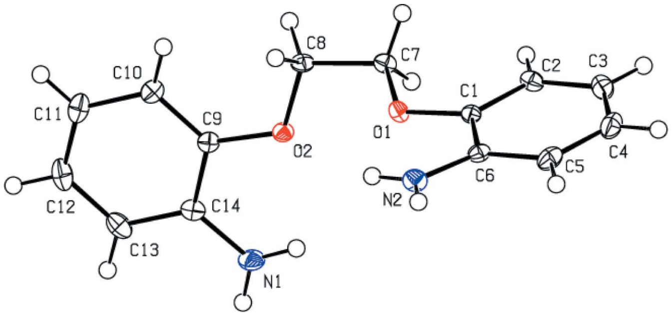


Figure 1
Molecular structure of (I) showing the atom labelling scheme and displacement ellipsoids at the 50% probability level.

H atoms were placed in geometrically idealized positions and constrained to ride on their parents atoms, with N—H = 0.86 Å and C—H = 0.93–0.97 Å, and with $U_{\rm iso}$(H) = 1.2$U_{\rm eq}$(parent atom). In the absence of significant anomalous scattering effects, Friedel pairs were averaged in the final refinement.

Data collection: *COLLECT* (Nonius, 1999); cell refinement: *EVALCCD* (Duisenberg *et al.*, 2003); data reduction: *SADABS* (Sheldrick, 2002); 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).

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