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1,2-Bis(*p*-nitrophenoxy)ethane

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Key indicators

Single-crystal X-ray study
T = 173 K
Mean σ(C–C) = 0.002 Å
R factor = 0.036
wR factor = 0.102
Data-to-parameter ratio = 14.6

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

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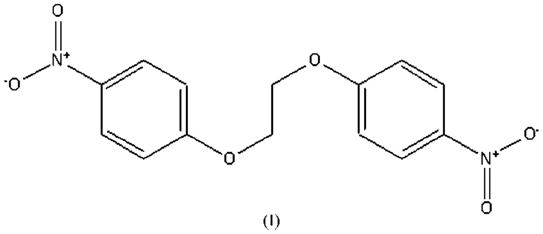
Molecules of the title compound, $C_{14}H_{12}N_2O_6$, are located on centres of inversion. The molecule is essentially planar, with the exception of the nitro group, which is twisted slightly out of the plane of the remaining atoms.

Comment

Polyimides form a very interesting group of mechanically, chemically and heat-resistant polymers (Lee & Jung, 1998). In many cases, polyimides are insoluble and do not react below their decomposition temperatures. On the other hand, their resistivity complicates their processing for technical applications. For these reasons their application as engineering materials is restricted (Im & Jung, 2000). Therefore, efforts have been made to improve their processability while maintaining their excellent 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 processability. Incorporation of flexible segments such as –O–, $-SO_2-$, $-CH_2-$ and $-C(CF_3)_2-$, 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. This is concomitant with reduction of crystallinity and enhancement of solubility. Unfortunately, while the solubility of non-coplanar structures with pendant aliphatic segments is enhanced, the thermal properties of such substances are usually worsened.



(I)

Recently, Hsio *et al.* (1997) reported some soluble thermoplastic polyimides derived from spiro-linked diamine structures in an effort to overcome this problem. The latter polymers had glass transition temperatures between 509 and 529 K and withstood temperatures up to 723 K (in N_2). The presence of flexible groups on the polyimide backbone causes a decrease in the rigidity of the polymer chain. Therefore,

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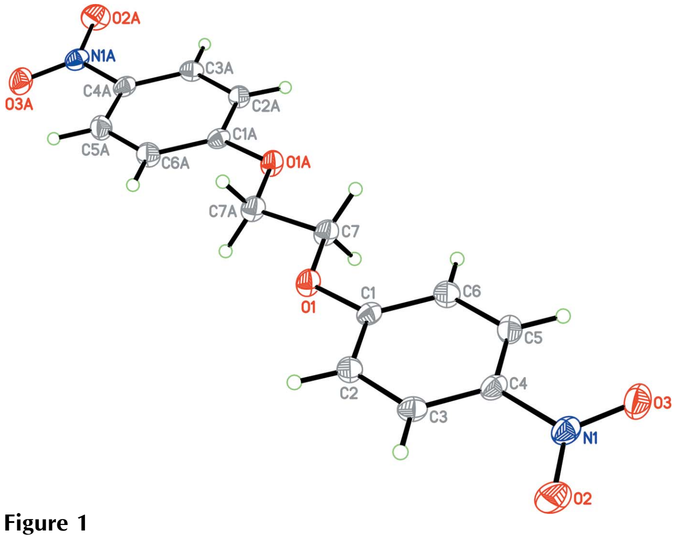

Figure 1
Molecular structure of (I). Displacement ellipsoids are drawn at the 50% probability level. [Symmetry code: (A) $-x + 2, -y + 1, -z + 1$.]

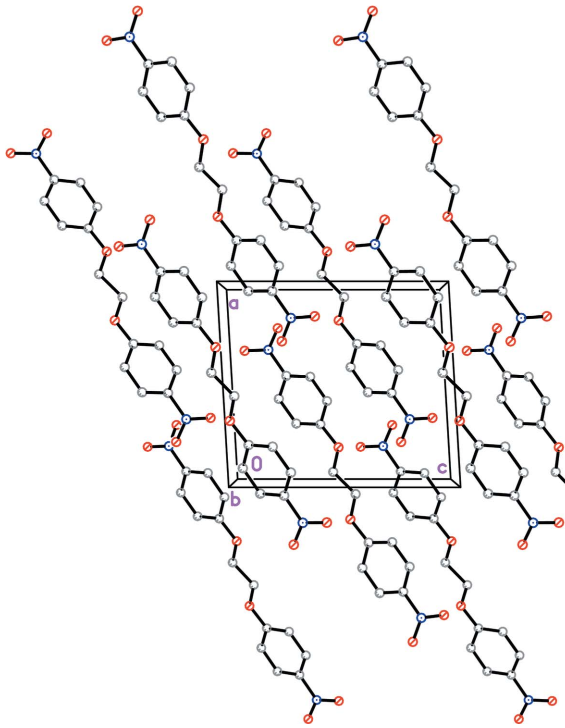

Figure 2
Packing diagram of (I), viewed along the b axis. H atoms have been omitted for clarity.

enhanced solubility of the polymer is expected to result (Hedrich, 1992).

The molecules of the title compound, (I), are located on centres of inversion. Geometric parameters are normal. All the non-H atoms apart from the nitro groups lie in a common plane with an r.m.s. deviation of 0.0128 Å (Khawar Rauf *et al.*, 2006). The NO_2 group makes a dihedral angle of 12.15 (13)° with the mean plane through the benzene ring (see also Table 1). The crystal structure is stabilized by a C—H···O hydrogen bond (Table 2).

Experimental

p-Nitrophenol (8.0 g, 0.057 mol) and anhydrous potassium carbonate (7.86 g, 0.057 mol) were suspended in a mixture of *N*,*N*′-dimethylformamide (50 ml) and toluene (30 ml). The mixture was refluxed at 473 K with a Dean–Stark trap for azeotropic removal of water. When most of the toluene had been removed, 1,2-dichloroethane (2.35 ml, 0.0285 mol) was added to the mixture, which was then refluxed for 12 h. The resulting solution was allowed to cool to room temperature and then it was poured into 300 ml of methanol–water (1:1) to give a yellow precipitate. After repeated washing with water, the product was separated by filtration and recrystallized from ethanol in 83% yield (m.p. 432 K).

Crystal data

$C_{14}H_{12}N_2O_6$
$M_r = 304.26$
Monoclinic, $P2_1/n$
$a = 9.1465$ (12) Å
$b = 7.3137$ (7) Å
$c = 10.2852$ (13) Å
$\beta = 93.353$ (10)°
$V = 686.85$ (14) Å^3
$Z = 2$
$D_x = 1.471$ Mg m^{-3}
Mo $K\alpha$ radiation
$\mu = 0.12$ mm^{-1}
$T = 173$ (2) K
Block, light yellow
0.49 × 0.46 × 0.44 mm

Data collection

Stoe IPDS II two-circle diffractometer
ω scans
Absorption correction: none
8567 measured reflections
1477 independent reflections
1380 reflections with $I > 2\sigma(I)$
$R_{\rm int} = 0.037$
$\theta_{\rm max} = 27.0°$

Refinement

Refinement on F^2
$R[F^2 > 2\sigma(F^2)] = 0.036$
$wR(F^2) = 0.103$
$S = 1.08$
1477 reflections
101 parameters
H-atom parameters constrained
$w = 1/[\sigma^2(F_o^2) + (0.0586P)^2 + 0.1845P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{\rm max} < 0.001$
$\Delta\rho_{\rm max} = 0.37$ e Å^{-3}
$\Delta\rho_{\rm min} = -0.20$ e Å^{-3}
Extinction correction: *SHELXL97*
Extinction coefficient: 0.11 (2)

Table 1
Selected torsion angles (°).

O3—N1—C4—C3	−168.86 (10)	O2—N1—C4—C3	11.59 (15)

Table 2
Hydrogen-bond geometry (Å, °).

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
C3—H3···O3$^{\rm i}$	0.95	2.48	3.4166 (14)	169

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

All the H atoms were located in a difference map. However, they were refined using a riding model, with C—H = 0.95 and 0.99 Å for aryl and methylene H atoms, respectively. $U_{\rm iso}$(H) values were set to $1.2U_{\rm eq}$(C).

Data collection: *X-AREA* (Stoe & Cie, 2001); cell refinement: *X-AREA*; data reduction: *X-AREA*; program(s) used to solve structure: *SHELXS97* (Sheldrick, 1997); 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*.

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