organic papers

N,N'-Bis(p-methylphenyl)-2,2'-(p-phenylene-dioxy)diacetamide

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Key indicators

Single-crystal X-ray study

$T = 293$ K

Mean σ(C–C) = 0.003 Å

R factor = 0.062

wR factor = 0.195

Data-to-parameter ratio = 10.7

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

In the title centrosymmetric compound, $C_{24}H_{24}N_2O_4$, the molecules are linked into a three-dimensional network through intermolecular N—H···O interactions. The crystal packing is further stabilized by C—H···π interactions.

Comment

Much attention has been paid to the amide-type acyclic polyethers with versatile molecular structures because they offer many advantages over the use of traditional crown ethers in the extraction of metal ions (Bünzli, 1987) and as active materials for ion-selective electrodes (Guggi *et al.*, 1977). For example, 2,2'-[1,2-phenylenebis(oxy)]-bis[*N*-methyl-*N*-phenyl(acetamide)] is a useful ionophore in electrode membranes and sensors (Farrell *et al.*, 1996). In order to investigate the effect of the skeleton of the acyclic polyether on the coordination selectivity and extractability of metal ions, and to find a novel and more selective ligand for the extraction of metal ions, we have designed and synthesized a series of acyclic polyethers. In the present paper, we report the synthesis and X-ray crystal structure of the title compound, (I) (Fig. 1).

(I)
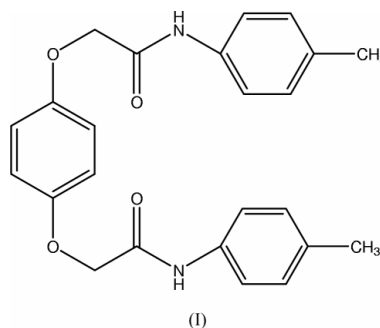
The asymmetric unit of (I) contains half the molecule, the other half being related by a crystallographic centre of symmetry at the centroid of the benzene ring (C10–C12/C10[i]–C12[i]) [symmetry code: (i) $1 - x$, $-y$, $1 - z$]. The bond lengths (Table 1) and angles in (I) are normal (Allen *et al.*, 1987).

The molecule of (I) is essentially planar, with a dihedral angle of 9.69 (12)° between the C10–C12/C10[i]–C12[i] ring and each of the two outer benzene rings. The O2···N1 separation of 2.621 (3) Å, which is less than the sum of the corresponding van der Waals radii (2.85 Å), suggests a possible intramolecular hydrogen bond (Table 2). There is another intramolecular interaction, C6—H6···O1, forming a six-membered ring. Molecules of (I) are linked into a three-dimensional network *via* intermolecular N1—H1N1···O1 interactions. The packing is further stabilized by C—H···π interactions (Table 2).
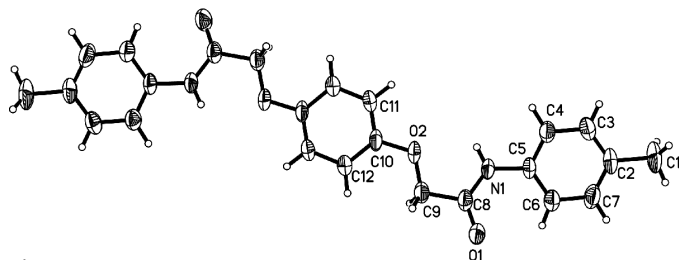
Figure 1
View of (I), showing 50% probability displacement ellipsoids and the atom-numbering scheme of the asymmetric unit.

Experimental

N-p-Methylphenyl chloroacetamide was prepared by the reaction of *p*-methylaniline and chloroacetyl chloride in the presence of triethylamine according to a literature method (Li, 2001). To a solution of *N-p*-methylphenyl chloroacetamide (1.84 g, 10 mmol) in acetone (35 ml) was added 1,4-dihydroxybenzene (0.55 g, 5.0 mmol), K_2CO_3 (1.52 g, 11 mmol) and KI (0.5 g). The mixture was stirred at 328 K for 5 h. After cooling to room temperature, the mixture was washed three times with water, and then filtered. The residue was recrystallized from ethanol and water. The title compound was obtained after drying of the resulting yellow powder at room temperature for 48 h. Light brown single crystals suitable for X-ray diffraction study were obtained by slow evaporation of a DMF–CH_3CH_2OH (1:20, *v/v*) solution over a period of one month.

Crystal data

$C_{24}H_{24}N_2O_4$	$D_x = 1.264$ Mg m^{-3}
$M_r = 404.46$	Mo $K\alpha$ radiation
Monoclinic, $P2_1/c$	Cell parameters from 2022
$a = 6.8881$ (14) Å	reflections
$b = 10.081$ (2) Å	$\theta = 3.3$–$25.0°$
$c = 15.312$ (3) Å	$\mu = 0.09$ mm^{-1}
$\beta = 91.68$ (3)°	$T = 293$ (2) K
$V = 1062.8$ (4) Å^3	Block, light brown
$Z = 2$	$0.32 \times 0.24 \times 0.22$ mm

Data collection

Bruker SMART 1000 CCD area detector diffractometer	1859 independent reflections
	1556 reflections with $I > 2\sigma(I)$
ω scans	$R_{int} = 0.057$
Absorption correction: multi-scan (*SADABS*; Sheldrick, 1996)	$\theta_{max} = 25.0°$
	$h = -8 \rightarrow 0$
$T_{min} = 0.973$, $T_{max} = 0.981$	$k = -11 \rightarrow 0$
2048 measured reflections	$l = -18 \rightarrow 18$

Refinement

Refinement on F^2	$w = 1/[\sigma^2(F_o^2) + (0.1185P)^2$
$R[F^2 > 2\sigma(F^2)] = 0.062$	$+ 0.4468P]$
$wR(F^2) = 0.195$	where $P = (F_o^2 + 2F_c^2)/3$
$S = 1.05$	$(\Delta/\sigma)_{max} < 0.001$
1859 reflections	$\Delta\rho_{max} = 0.36$ e Å^{-3}
173 parameters	$\Delta\rho_{min} = -0.28$ e Å^{-3}
H atoms treated by a mixture of independent and constrained refinement	Extinction correction: *SHELXL*
	Extinction coefficient: 0.038 (8)

Table 1
Selected bond distances (Å).

O1—C8	1.220 (3)	N1—C8	1.343 (3)
O2—C10	1.392 (2)	N1—C5	1.434 (2)
O2—C9	1.411 (3)		

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

D—H$\cdots A$	D—H	H$\cdots A$	$D\cdots A$	D—H$\cdots A$
N1—H1N1$\cdots$O2	0.86 (3)	2.22 (3)	2.621 (3)	108 (2)
N1—H1N1$\cdots$O1^{ii}	0.86 (3)	2.43 (3)	3.112 (3)	137 (2)
C6—H6$\cdots$O1	0.93 (3)	2.32 (3)	2.885 (3)	119 (2)
C9—H9$A\cdots$Cg1^{iii}	1.02 (3)	2.74	3.68	154

Symmetry codes: (ii) $-x, y - \frac{1}{2}, \frac{1}{2} - z$; (iii) $1 + x, y, z$. Note: Cg1 denotes the centroid of the C2–C7 ring.

All H atoms were located in difference Fourier maps and refined isotropically except the H atoms on C1, which were positioned geometrically and treated as riding, with C—H = 0.96 Å and $U_{iso}(H) = 1.5U_{eq}(C)$.

Data collection: *SMART* (Siemens, 1996); cell refinement: *SAINT* (Siemens, 1996); data reduction: *SAINT*; program(s) used to solve structure: *SHELXTL* (Sheldrick, 1997); program(s) used to refine structure: *SHELXTL*; molecular graphics: *SHELXTL*; software used to prepare material for publication: *SHELXTL, PARST* (Nardelli, 1995) and *PLATON* (Spek, 2003).

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