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N,*N*′-Bis(*p*-methoxyphenyl)-2,2′-(*m*-phenylenedioxy)diacetamide

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Key indicators

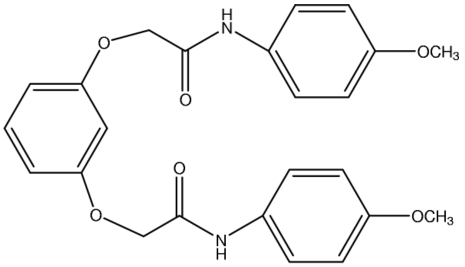
Single-crystal X-ray study
T = 293 K
Mean σ(C–C) = 0.003 Å
R factor = 0.049
wR factor = 0.120
Data-to-parameter ratio = 13.1

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

In the title compound, $C_{24}H_{24}N_2O_6$, the molecules are linked into a three-dimensional network by intermolecular N—H⋯O and C—H⋯O hydrogen bonds. The packing is further stabilized by C—H⋯π interactions.

Comment

Previously, we have reported the structure of an amide-type acyclic polyether with 1,3-dihydroxybenzene as skeleton, namely *N*,*N*′-bis(*p*-tolyl)-2,2′-(*m*-phenylenedioxy)diacetamide, (II) (Zhang *et al.*, 2004). In order to study the effect of the terminal groups of the acyclic polyethers on the coordination selectivity and extractability for metal ions, we have synthesized and carried out the structure determination of the title compound, (I) (Fig. 1).



(I)

The bond lengths (Table 1) and angles in (I) are in good agreement with those in (II). In (I), the dihedral angles between the central benzene ring (C10–C15) and the two outer benzene rings (C2–C7 and C18–C23) are 80.2 (1) and 52.7 (1)°, respectively. Both the outer benzene rings are involved in intramolecular C—H⋯O interactions with the carbonyl O atoms (Table 2). Molecules of (I) are linked into a three-dimensional network *via* intermolecular N—H⋯O and C—H⋯O hydrogen bonds. The packing is further stabilized by C—H⋯π interactions (Table 2).

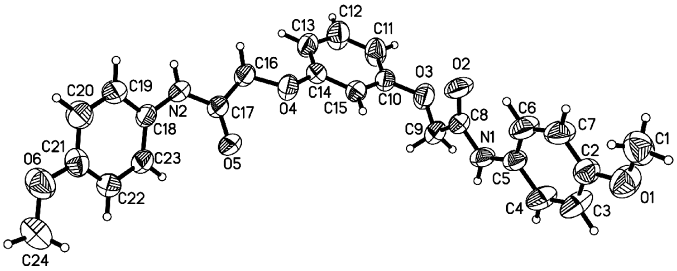


Figure 1
The structure of (I), showing 50% probability displacement ellipsoids and the atom-numbering scheme.

Experimental

N-(*p*-Methoxyphenyl)chloroacetamide was prepared by the reaction of *p*-methoxyaniline and chloroacetyl chloride in the presence of triethylamine, according to the literature method of Li (2001). To a solution of *N*-(*p*-methoxyphenyl)chloroacetamide (2.0 g, 10 mmol) in acetone (35 ml) was added 1,3-dihydroxybenzene (0.55 g, 5.0 mmol), K_2CO_3 (1.52 g, 11 mmol) and KI (0.5 g), and 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 filtered solid was recrystallized from alcohol and water. The title compound was obtained after drying the light-grey powder at room temperature for 48 h. Colourless single crystals suitable for X-ray diffraction analysis were obtained by slow evaporation of a DMF–ethanol (1:20 *v*/*v*) solution over a period of one month.

Crystal data

$C_{24}H_{24}N_2O_6$
$M_r = 436.45$
Monoclinic, $P2_1/c$
$a = 11.2370\ (13)$ Å
$b = 18.256\ (2)$ Å
$c = 10.4779\ (12)$ Å
$\beta = 93.532\ (2)^\circ$
$V = 2145.4\ (4)$ Å^3
$Z = 4$
$D_x = 1.351$ Mg m^{-3}
Mo $K\alpha$ radiation
Cell parameters from 1683 reflections
$\theta = 2.2$–25.0°
$\mu = 0.10$ mm^{-1}
$T = 293\ (2)$ K
Needle, colourless
0.40 × 0.11 × 0.08 mm

Data collection

Bruker SMART 1000 CCD area-detector diffractometer
ω scans
Absorption correction: multi-scan (*SADABS*; Sheldrick, 1996)
$T_{\min} = 0.962$, $T_{\max} = 0.992$
11 048 measured reflections
3792 independent reflections
2349 reflections with $I > 2\sigma(I)$
$R_{\rm int} = 0.036$
$\theta_{\max} = 25.0^\circ$
$h = -12 \rightarrow 13$
$k = -17 \rightarrow 21$
$l = -12 \rightarrow 12$

Refinement

Refinement on F^2
$R[F^2 > 2\sigma(F^2)] = 0.049$
$wR(F^2) = 0.120$
$S = 1.00$
3792 reflections
289 parameters
H-atom parameters constrained
$w = 1/[\sigma^2(F_o^2) + (0.0509P)^2 + 0.1396P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{\max} < 0.001$
$\Delta\rho_{\max} = 0.15$ e Å^{-3}
$\Delta\rho_{\min} = -0.12$ e Å^{-3}

Table 1

Selected bond distances (Å).

O2—C8	1.222 (2)	O5—C17	1.225 (2)
O3—C10	1.371 (2)	N1—C8	1.340 (3)
O3—C9	1.413 (3)	N1—C5	1.409 (3)
O4—C14	1.376 (2)	N2—C17	1.341 (3)
O4—C16	1.418 (2)	N2—C18	1.417 (3)

Table 2

Hydrogen-bonding geometry (Å, °).

D—H···A	D—H	H···A	D···A	D—H···A
N1—H1···O5$^{\rm i}$	0.86	2.02	2.875 (2)	172
N2—H2···O2$^{\rm ii}$	0.86	2.04	2.897 (2)	172
C6—H6···O2	0.93	2.35	2.936 (4)	121
C11—H11···O1$^{\rm iii}$	0.93	2.53	3.448 (3)	170
C23—H23···O5	0.93	2.32	2.853 (3)	116
C1—H1C···$Cg1^{\rm i}$	0.96	2.72	3.608 (3)	155
C16—H16A···$Cg2^{\rm iv}$	0.97	2.78	3.729 (2)	165
C24—H24B···$Cg2^{\rm v}$	0.96	2.70	3.590 (3)	155

Symmetry codes: (i) $x, \frac{1}{2}-y, z-\frac{1}{2}$; (ii) $-x, -y, 1-z$; (iii) $1+x, \frac{1}{2}-y, \frac{1}{2}+z$; (iv) $-x, -y, 2-z$; (v) $-1-x, -y, 2-z$. *Cg*1 and *Cg*2 denote the centroids of the C2–C7 and C18–C23 benzene rings, respectively

After location in a difference map, all the H atoms were positioned geometrically and refined as riding, with C—H distances in the range 0.93–0.97 Å and $U_{\rm iso}({\rm H}) = 1.2U_{\rm eq}({\rm C})$, or $1.5U_{\rm eq}({\rm C})$ for methyl H atoms.

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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