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Key indicators

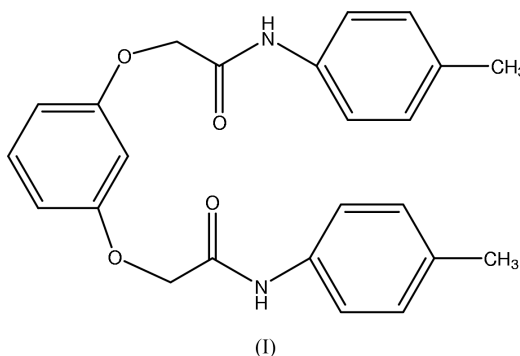
Single-crystal X-ray study
 $T = 293$ K
Mean $\sigma(\text{C}-\text{C}) = 0.006$ Å
 R factor = 0.085
 wR factor = 0.257
Data-to-parameter ratio = 16.1For details of how these key indicators were automatically derived from the article, see <http://journals.iucr.org/e>.*N,N'*-Bis(*p*-tolyl)-2,2'-(*m*-phenylenedioxy)-diacetamideIn the title compound, $\text{C}_{24}\text{H}_{24}\text{N}_2\text{O}_4$, the molecules are linked into a three-dimensional network by intermolecular $\text{N}-\text{H}\cdots\text{O}$ hydrogen bonds. The packing is further stabilized by $\text{C}-\text{H}\cdots\pi$ interactions.

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Comment

We have reported a novel amide-type acyclic polyether with 1,4-dihydroxybenzene as a skeleton, namely *N,N'*-bis(*p*-tolyl)-2,2'-(*p*-phenylenedioxy)diacetamide, (II) (Wen *et al.*, 2004), which is a possible active material for ion-selective electrodes or a fine extractor of metal ions. In our ongoing study of the effect of the skeleton of the acyclic polyether on the coordination selectivity and extractability of metal ions, we have carried out a similar reaction for *N*-(*p*-tolyl)-2-chloroacetamide with 1,3-dihydroxybenzene, and obtained the title compound, (I) (Fig. 1).The bond lengths (Table 1) and angles in (I) are comparable with those in (II). The molecule in (II) is essentially planar, while the molecule in (I) has a wing-like conformation. In (I), the dihedral angles between the central benzene ring (C10–C15) and the two outer benzene rings (C2–C7 and C18–C23) are 28.2 (2) and 86.9 (3)°, respectively. Both the outer benzene rings are involved in intramolecular $\text{C}-\text{H}\cdots\text{O}$ interactions, with the two carbonyl O atoms as acceptors (Table 2). Molecules of (I) are linked into a three-dimensional network *via* intermolecular $\text{N}-\text{H}\cdots\text{O}$ hydrogen bonds (Table 2). The packing is further stabilized by $\text{C}-\text{H}\cdots\pi$ interactions (Table 2).

Experimental

N-(*p*-Tolyl)-2-chloroacetamide was prepared by the reaction of *p*-methylaniline and chloroacetyl chloride in the presence of triethyl-

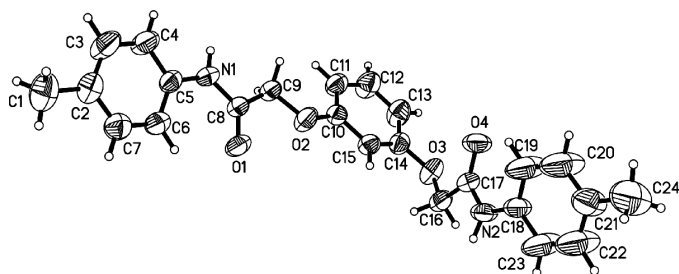


Figure 1
The structure of (I), showing 50% probability displacement ellipsoids and the atom-numbering scheme.

amine, according to the literature method of Li (2001). To a solution of *N*-(*p*-tolyl)-2-chloroacetamide (1.84 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 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 yellow powder at room temperature for 48 h. Light-brown 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

$C_{24}H_{24}N_2O_4$
 $M_r = 404.45$
 Orthorhombic, *Pbca*
 $a = 18.5029$ (11) Å
 $b = 10.6914$ (7) Å
 $c = 21.3741$ (13) Å
 $V = 4228.3$ (5) Å³
 $Z = 8$
 $D_x = 1.271$ Mg m⁻³
 Mo $K\alpha$ radiation
 Cell parameters from 3272 reflections
 $\theta = 2.9$ – 26.0°
 $\mu = 0.09$ mm⁻¹
 $T = 293$ (2) K
 Block, brown
 $0.30 \times 0.26 \times 0.24$ mm

Data collection

Bruker SMART 1000 CCD area-detector diffractometer
 ω scans
 Absorption correction: multi-scan (*SADABS*; Sheldrick, 1996)
 $T_{min} = 0.974$, $T_{max} = 0.979$
 21758 measured reflections
 4172 independent reflections
 2699 reflections with $I > 2\sigma(I)$
 $R_{int} = 0.040$
 $\theta_{max} = 26.0^\circ$
 $h = -17 \rightarrow 22$
 $k = -13 \rightarrow 13$
 $l = -24 \rightarrow 26$

Refinement

Refinement on F^2
 $R[F^2 > 2\sigma(F^2)] = 0.085$
 $wR(F^2) = 0.257$
 $S = 1.02$
 4172 reflections
 259 parameters
 H-atom parameters constrained
 $w = 1/[\sigma^2(F_o^2) + (0.1182P)^2 + 4.5457P]$
 where $P = (F_o^2 + 2F_c^2)/3$
 $(\Delta/\sigma)_{max} < 0.001$
 $\Delta\rho_{max} = 0.63$ e Å⁻³
 $\Delta\rho_{min} = -0.62$ e Å⁻³

Table 1
Selected distances (Å).

| | | | |
|--------|-----------|--------|-----------|
| O1—C8 | 1.225 (4) | O4—C17 | 1.229 (4) |
| O2—C10 | 1.368 (4) | N1—C8 | 1.347 (4) |
| O2—C9 | 1.413 (4) | N1—C5 | 1.411 (4) |
| O3—C14 | 1.375 (4) | N2—C17 | 1.330 (4) |
| O3—C16 | 1.413 (4) | N2—C18 | 1.410 (5) |

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

| <i>D</i> —H... <i>A</i> | <i>D</i> —H | H... <i>A</i> | <i>D</i> ... <i>A</i> | <i>D</i> —H... <i>A</i> |
|-----------------------------|-------------|---------------|-----------------------|-------------------------|
| N1—H1A...O4 ⁱ | 0.86 | 2.09 | 2.946 (4) | 176 |
| N2—H2A...O1 ⁱⁱ | 0.86 | 2.06 | 2.912 (4) | 172 |
| C6—H6A...O1 | 0.93 | 2.24 | 2.844 (5) | 122 |
| C19—H19A...O4 | 0.93 | 2.41 | 2.926 (7) | 115 |
| C9—H9A...Cg1 ⁱⁱⁱ | 0.97 | 2.80 | 3.716 | 158 |
| C9—H9B...Cg2 ⁱ | 0.97 | 2.88 | 3.639 | 135 |

Symmetry codes: (i) $-x, 1 - y, -z$; (ii) $-\frac{1}{2} - x, y - \frac{1}{2}, z$; (iii) $-x, 2 - y, -z$. Cg1 and Cg2 denote the centroids of the C2–C7 and C10–C15 benzene rings

All H atoms were positioned geometrically, with C–H = 0.93–0.96 Å and N–H = 0.86 Å, and treated as riding, with $U_{iso}(H) = 1.2$ or 1.5 times U_{eq} of the carrier atoms.

Data collection: *SMART* (Siemens, 1996); cell refinement: *SAINTE* (Siemens, 1996); data reduction: *SAINTE*; 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, 1990).

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