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# *N*,*N*′-Bis(*p*-methoxyphenyl)-2,2′-(*p*-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 = 0.068
*wR* factor = 0.185
Data-to-parameter ratio = 16.9

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

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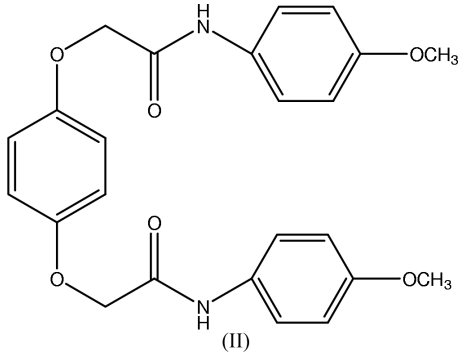
The title compound, $C_{24}H_{24}N_2O_6$, crystallizes in the triclinic space group $P\overline{1}$ with two half molecules in the asymmetric unit. The two independent molecules differ in the orientation of the carbonyl group. In the crystal packing, the molecules are linked into chains *via* intermolecular N—H···O interactions. The packing is further stabilized by C—H···π interactions.

## Comment

Recently, we have reported the crystal structure of an amide-type acyclic polyether with 1,4-dihydroxybenzene as a skeleton, *N*,*N*′-bis(*p*-methylphenyl)-2,2′-(*p*-phenylenedioxy)-diacetamide, (I) (Wen *et al.*, 2004). We report here the crystal structure of *N*,*N*′-bis(*p*-methoxyphenyl)-2,2′-(*p*-phenylenedioxy)diacetamide, (II).




(II)

The asymmetric unit of (II) contains two half molecules, the other halves being related by crystallographic inversion centres (Fig. 1). The corresponding bond lengths and angles in the two independent molecules *A* (containing O3) and *B*

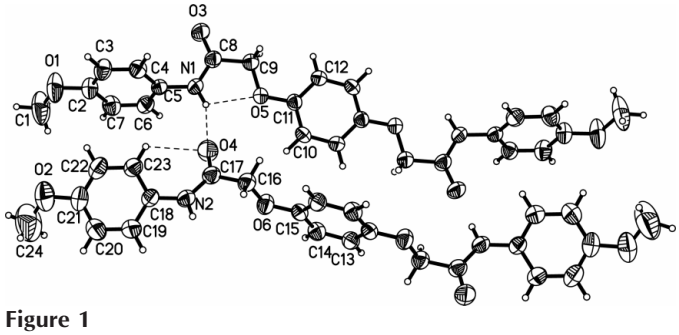



**Figure 1**
The structure of the two molecules of the asymmetric unit of (II), showing 50% probability displacement ellipsoids and the atom-numbering scheme. Unlabelled atoms in molecule *A* are related to labelled atoms by $(1-x, 1-y, -1-z)$. Unlabelled atoms in molecule *B* are related to labelled atoms by $(2-x, 1-y, -1-z)$.

(containing O4) agree with each other, and are comparable to those in (I) (Wen *et al.*, 2004). However, the N—C—C—O and O—C—C—O torsion angles in the acetamide linkage of *A* and *B* deviate significantly from one another (Table 1). The dihedral angle between the central and each of the outer benzene rings is 66.6 (1)° in *A* and 69.9 (1)° in *B*. In the centrosymmetric compound, (I), this dihedral angle is 9.69 (12)°.

In the asymmetric unit of (II), the two independent molecules are linked by N1—H1···O4 hydrogen bonds (Table 2). There is an intramolecular hydrogen bond in both *A* and *B*, forming five- and six-membered rings, respectively. Molecules of (II) are linked into chains *via* intermolecular N2—H2···O3(1 + *x*, *y*, *z*) interactions. The packing is further stabilized by C—H···$\pi$ interactions (Table 2)

## Experimental

To a solution of *N*-*p*-methoxyphenyl chloroacetamide (2.00 g, 10 mmol) in acetone (35 ml) were added 1,4-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 ethanol and water. The title compound was obtained after drying the resulting yellow powder at room temperature for 48 h. Colourless single crystals of (II) suitable for X-ray diffraction analysis were obtained by slow evaporation of a solution in dimethylformamide–ethanol (1:20, *v*/*v*) over a period of one month.

*Crystal data*

$C_{24}H_{24}N_2O_6$
$M_r$ = 436.45
Triclinic, $P\overline{1}$
$a$ = 9.8370 (7) Å
$b$ = 10.1922 (8) Å
$c$ = 10.9181 (8) Å
$\alpha$ = 94.327 (1)°
$\beta$ = 92.633 (1)°
$\gamma$ = 97.719 (1)°
$V$ = 1079.90 (14) Å$^3$
$Z$ = 2
$D_x$ = 1.342 Mg m$^{-3}$
Mo $K\alpha$ radiation
Cell parameters from 6061 reflections
$\theta$ = 1.9–28.0°
$\mu$ = 0.10 mm$^{-1}$
$T$ = 293 (2) K
Block, colourless
0.34 × 0.22 × 0.16 mm

*Data collection*

Siemens SMART 1000 CCD area-detector diffractometer
$\omega$ scans
Absorption correction: multi-scan, (*SADABS*; Sheldrick, 1996)
$T_{min}$ = 0.968, $T_{max}$ = 0.985
9528 measured reflections
4932 independent reflections
3671 reflections with $I > 2\sigma(I)$
$R_{int}$ = 0.019
$\theta_{max}$ = 28.0°
$h = -12 \rightarrow 12$
$k = -13 \rightarrow 13$
$l = -14 \rightarrow 14$

*Refinement*

Refinement on $F^2$
$R[F^2 > 2\sigma(F^2)]$ = 0.068
$wR(F^2)$ = 0.185
$S$ = 1.04
4932 reflections
291 parameters
H-atom parameters constrained
$w = 1/[\sigma^2(F_o^2) + (0.0865P)^2 + 0.3355P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} < 0.001$
$\Delta\rho_{max}$ = 0.39 e Å$^{-3}$
$\Delta\rho_{min}$ = −0.27 e Å$^{-3}$

**Table 1**
Selected geometric parameters (Å, °).

| | | | |
|---|---|---|---|
| O1—C2 | 1.365 (3) | O5—C9 | 1.406 (2) |
| O1—C1 | 1.413 (4) | O6—C15 | 1.380 (2) |
| O2—C24 | 1.284 (5) | O6—C16 | 1.419 (3) |
| O2—C21 | 1.365 (3) | N1—C8 | 1.324 (3) |
| O3—C8 | 1.225 (2) | N1—C5 | 1.432 (2) |
| O4—C17 | 1.217 (3) | N2—C17 | 1.339 (3) |
| O5—C11 | 1.375 (2) | N2—C18 | 1.409 (3) |
| | | | |
| C1—O1—C2—C7 | 5.8 (5) | C16—O6—C15—C14 | −176.51 (19) |
| C8—N1—C5—C4 | −70.5 (3) | C15—O6—C16—C17 | 175.41 (17) |
| C5—N1—C8—O3 | 5.1 (3) | C18—N2—C17—O4 | −6.0 (4) |
| C11—O5—C9—C8 | −174.49 (17) | O6—C16—C17—O4 | −89.5 (3) |
| O3—C8—C9—O5 | −171.9 (2) | O6—C16—C17—N2 | 90.5 (2) |
| N1—C8—C9—O5 | 8.9 (3) | C17—N2—C18—C23 | −22.7 (3) |
| C9—O5—C11—C10 | 171.12 (19) | C24—O2—C21—C20 | −25.1 (6) |

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

*Cg*1 and *Cg*2 are the centroids of the central benzene rings of molecules *A* and *B*, respectively, and *Cg*3 is the centroid of the outer ring of molecule *B*.

| *D*—H···*A* | *D*—H | H···*A* | *D*···*A* | *D*—H···*A* |
|---|---|---|---|---|
| N1—H1···O4$^{i}$ | 0.86 | 2.19 | 2.965 (2) | 149 |
| N1—H1···O5$^{i}$ | 0.86 | 2.24 | 2.624 (2) | 107 |
| N2—H2···O3$^{ii}$ | 0.86 | 1.94 | 2.793 (2) | 172 |
| C23—H23···O4$^{i}$ | 0.93 | 2.40 | 2.941 (3) | 117 |
| C3—H3···*Cg*1$^{iii}$ | 0.93 | 2.75 | 3.57 | 147 |
| C12—H12···*Cg*2$^{iv}$ | 0.93 | 2.89 | 3.67 | 142 |
| C13—H13···*Cg*3$^{v}$ | 0.93 | 2.96 | 3.72 | 140 |

Symmetry codes: (i) $x, y, z$; (ii) $x+1, y, z$; (iii) $x, y, z+1$; (iv) $x-1, y, z$; (v) $x, y, z-1$.

All H atoms were positioned geometrically, with C—H = 0.93–0.97 Å and N—H = 0.86 Å, and were treated as riding, with $U_{iso}$(H) = 1.2 or 1.5 times $U_{eq}$ of the parent 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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