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N,*N*′-Bis(4-methoxyphenyl)-3-oxapentanediamide

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Key indicators

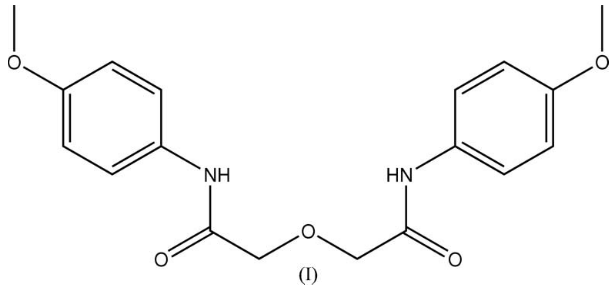
Single-crystal X-ray study
T = 293 K
Mean σ(C–C) = 0.003 Å
R factor = 0.058
wR factor = 0.163
Data-to-parameter ratio = 15.0

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

The molecule of the title compound, $C_{18}H_{20}N_2O_5$, shows a wing-like conformation, with a dihedral angle of 12.7 (1)° between the two benzene rings. In the crystal structure, intermolecular N—H···O hydrogen bonds link the molecules into chains extending along the *b* axis.

Comment

Amide-type acyclic polyethers have been widely studied in the complexation and extraction of metal ions (Li *et al.*, 2003). We have previously reported the structure of an amide-type acyclic polyether with a dihydroxybenzene skeleton, namely *N*,*N*′-bis(*p*-methoxyphenyl)-2,2′-(*p*-phenylenedioxy)-diacetamide (Wen *et al.*, 2005). We report here the crystal structure of the title compound, (I).



(I)

The molecule of (I) has a wing-like conformation, with a dihedral angle of 12.7 (1)° between the two benzene rings. In the molecule, which exhibits a pseudo-twofold axis, all bond lengths and angles are within normal ranges (Allen *et al.*, 1987), and the corresponding values of the two wings are comparable to one another. Each benzene ring and its attached methoxy group are coplanar. The sum of the bond angles arround each N atom is about 360°, implying a planar configuration. In the crystal structure, intermolecular N—H···O nydrogen bonds (Table 2) link the molecules into chains extending along the *b* axis (Fig. 2).

Experimental

$SOCl_2$ (5.0 ml, 0.08 mol) was added slowly to a solution of oxydiacetic acid (2.68 g, 0.02 mol) in benzene. After 2 h of stirring at 343 K, the mixture turned clear, and stirring was continued for a further 2 h. Benzene and an excess of $SOCl_2$ were then removed under reduced pressure to give oxydiacetic acid dichloride. This compound (1.71 g, 0.01 mol) in benzene (20 ml) was added dropwise to a solution of *p*-methoxyaniline (2.46 g, 0.02 mol) and pyridine (2 ml) in benzene (40 ml), and the mixture was stirred at 343 K for 10 h. After cooling, the mixture was washed three times with water, and then filtered. The title compound was recrystallized from benzene as a light-brown

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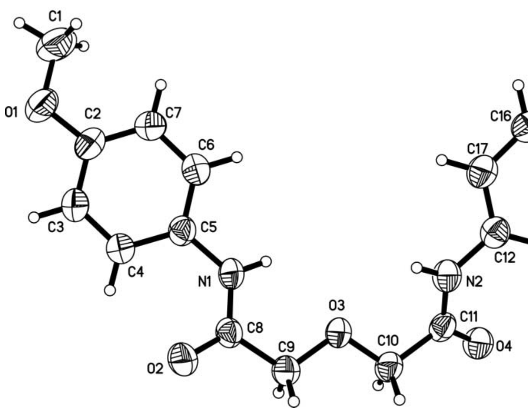

Figure 1
View of (I), showing the atom-numbering scheme and displacement ellipsoids drawn at the 50% probability level.

powder. Dark-brown single crystals suitable for X-ray diffraction study were obtained by slow evaporation of an ethanol–ethyl acetate (1:20 *v*/*v*) solution over a period of one month.

Crystal data

$C_{18}H_{20}N_2O_5$
$M_r = 344.36$
Triclinic, $P\overline{1}$
$a = 8.1399$ (10) Å
$b = 8.4654$ (11) Å
$c = 13.3901$ (17) Å
$\alpha = 103.201$ (2)°
$\beta = 101.383$ (2)°
$\gamma = 92.577$ (2)°
$V = 876.70$ (19) Å^3

$Z = 2$
$D_x = 1.304$ Mg m^{-3}
Mo $K\alpha$ radiation
Cell parameters from 1756 reflections
$\theta = 2.5$–25.4°
$\mu = 0.10$ mm^{-1}
$T = 293$ (2) K
Plate, dark brown
0.42 × 0.31 × 0.09 mm

Data collection

Siemens SMART 1000 CCD area-detector diffractometer
ω scans
Absorption correction: multi-scan (*SADABS*; Sheldrick, 1996)
$T_{\min} = 0.961$, $T_{\max} = 0.991$
5013 measured reflections

3379 independent reflections
2691 reflections with $I > 2\sigma(I)$
$R_{\rm int} = 0.012$
$\theta_{\max} = 26.0°$
$h = -10 \rightarrow 10$
$k = -10 \rightarrow 8$
$l = -16 \rightarrow 16$

Refinement

Refinement on F^2
$R[F^2 > 2\sigma(F^2)] = 0.058$
$wR(F^2) = 0.163$
$S = 1.04$
3379 reflections
226 parameters
H-atom parameters constrained

$w = 1/[\sigma^2(F_o^2) + (0.0837P)^2 + 0.2739P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{\max} < 0.001$
$\Delta\rho_{\max} = 0.90$ e Å^{-3}
$\Delta\rho_{\min} = -0.22$ e Å^{-3}

Table 1
Selected geometric parameters (Å, °).

O1—C2	1.369 (2)	O5—C15	1.367 (3)
O1—C1	1.422 (3)	O5—C18	1.432 (3)
O2—C8	1.228 (2)	N1—C8	1.335 (3)
O3—C10	1.414 (2)	N1—C5	1.425 (2)
O3—C9	1.418 (2)	N2—C11	1.335 (3)
O4—C11	1.231 (2)	N2—C12	1.423 (3)
C8—N1—C5	127.12 (16)	C11—N2—C12	127.27 (17)

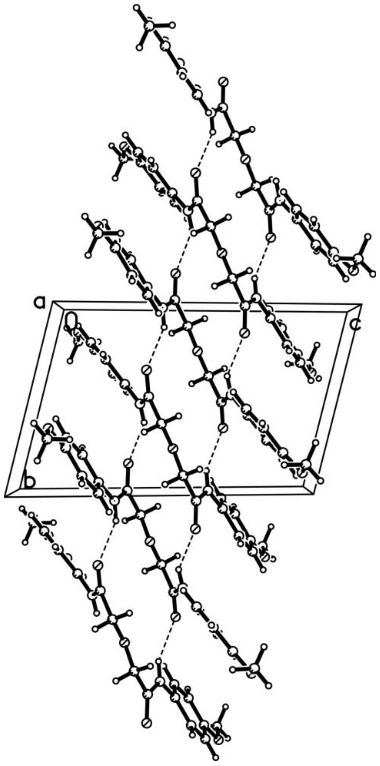

Figure 2
The crystal packing, viewed down the *a* axis, showing the hydrogen-bonded chains of molecules. Hydrogen bonds are indicated by dashed lines.

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

D—H···A	D—H	H···A	D···A	D—H···A
N1—H1A···O4$^{\rm i}$	0.86	2.19	2.951 (2)	147
N2—H2A···O2$^{\rm ii}$	0.86	2.16	2.952 (2)	152

Symmetry codes: (i) $-x, -y+2, -z+1$; (ii) $-x, -y+1, -z+1$.

All H atoms were located in difference Fourier maps and constrained to ride on their parent atoms, with C—H = 0.93–0.97 Å and N—H = 0.86 Å, and $U_{\rm iso}$(H) = $1.2U_{\rm eq}$(C,N) or $1.5U_{\rm eq}$(methyl 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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