

N,N'-Bis(4-methylphenyl)-3-oxapentanediamideYong-Hong Wen, Hui-Ling Wen,
Xue-Mei Li and Shu-Sheng
Zhang*College of Chemistry and Molecular Engi-
neering, Qingdao University of Science and
Technology, 266042 Qingdao, Shandong,
People's Republic of China

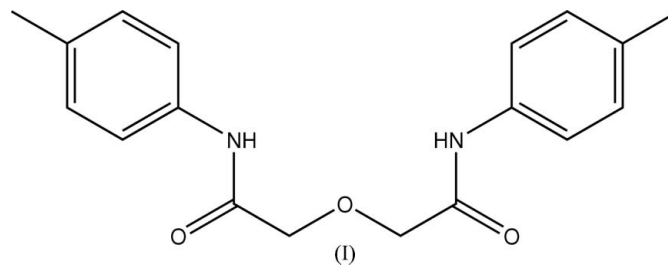
Correspondence e-mail: shushzhang@126.com

The title compound, $C_{18}H_{20}N_2O_3$, has twofold rotation symmetry. Molecules are linked into ribbons along the *c* axis by $N-H \cdots O$ and $C-H \cdots O$ hydrogen bonds.Received 4 April 2006
Accepted 8 April 2006

Comment

We have recently reported the structure of *N,N'*-bis(4-methoxyphenyl)-3-oxapentanediamide, (II), (Wen *et al.*, 2006). In our ongoing studies of amide-type acyclic polyethers, the title compound, (I), has been prepared.

Key indicators

Single-crystal X-ray study
 $T = 293$ K
Mean $\sigma(C-C) = 0.002$ Å
 R factor = 0.045
 wR factor = 0.133
Data-to-parameter ratio = 15.3For details of how these key indicators were
automatically derived from the article, see
<http://journals.iucr.org/e>.

In the molecule of (I), a crystallographic twofold rotation axis passes through O2. All bond lengths and angles are within normal ranges (Allen *et al.*, 1987), and comparable to those in the compound (II). There exists one intramolecular hydrogen bond, $C6-H6A \cdots O1$ (Table 2), forming a six-membered ring. In the crystal structure, molecules are linked into ribbons along the *c* axis (Fig. 2) by $N1-H1A \cdots O1^i$ and $C4-H4A \cdots O1^i$ intermolecular hydrogen bonds (symmetry code as in Table 2).

Experimental

$SOCl_2$ (5.0 ml, 0.08 mol) was slowly added to a solution of oxydiacetic acid (2.68 g, 0.02 mol) in benzene (20 ml). After stirring for 3 h at

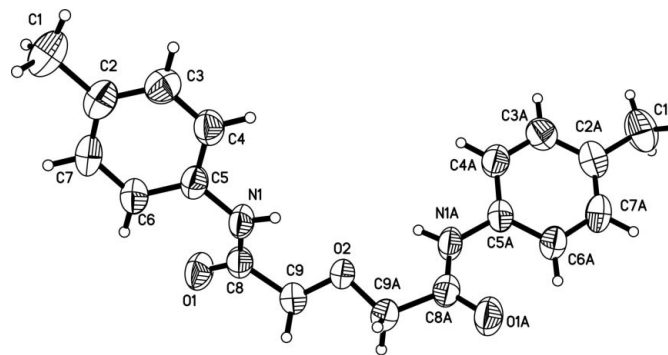


Figure 1

The molecular structure of (I), showing 50% probability displacement ellipsoids and the atom numbering scheme. The suffix A corresponds to symmetry code $(1 - x, y, \frac{1}{2} - z)$.

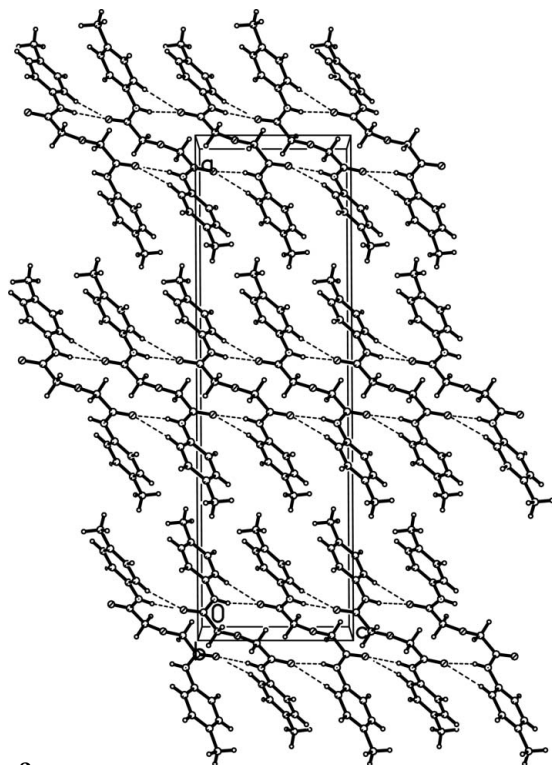


Figure 2
A view down the *b* axis, showing the ribbons. Hydrogen bonds are indicated by dashed lines.

343 K, the mixture turned clear and was stirred for a further 2 h. Benzene and excess 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*-methylaniline (2.14 g, 0.02 mol) and pyridine (2 ml) in benzene (40 ml), and the mixture was stirred at 343 K for 12 h. After cooling to room temperature, the mixture was washed three times with water and then filtered. The title compound was recrystallized from benzene as a light-brown powder. Dark-brown single crystals suitable for X-ray diffraction study were obtained by slow evaporation of an ethanol–ethyl acetate (1:16, *v/v*) solution over a period of one month.

Crystal data

$\text{C}_{18}\text{H}_{20}\text{N}_2\text{O}_3$	$Z = 4$
$M_r = 312.36$	$D_x = 1.274 \text{ Mg m}^{-3}$
Monoclinic, $C2/c$	Mo $K\alpha$ radiation
$a = 29.059 (3) \text{ \AA}$	$\mu = 0.09 \text{ mm}^{-1}$
$b = 6.2798 (7) \text{ \AA}$	$T = 293 (2) \text{ K}$
$c = 8.9209 (10) \text{ \AA}$	Column, dark-brown
$\beta = 90.299 (2)^\circ$	$0.33 \times 0.24 \times 0.11 \text{ mm}$
$V = 1627.9 (3) \text{ \AA}^3$	

Data collection

Siemens SMART 1000 CCD area-detector diffractometer	4509 measured reflections
ω scans	1606 independent reflections
Absorption correction: multi-scan (SADABS; Sheldrick, 1996)	1278 reflections with $I > 2\sigma(I)$
$T_{\min} = 0.972$, $T_{\max} = 0.990$	$R_{\text{int}} = 0.018$
	$\theta_{\text{max}} = 26.0^\circ$

Refinement

Refinement on F^2	$w = 1/[\sigma^2(F_o^2) + (0.0709P)^2 + 0.5191P]$
$R[F^2 > 2\sigma(F^2)] = 0.045$	where $P = (F_o^2 + 2F_c^2)/3$
$wR(F^2) = 0.133$	$(\Delta/\sigma)_{\text{max}} < 0.001$
$S = 1.04$	$\Delta\rho_{\text{max}} = 0.17 \text{ e \AA}^{-3}$
1606 reflections	$\Delta\rho_{\text{min}} = -0.15 \text{ e \AA}^{-3}$
105 parameters	
H-atom parameters constrained	

Table 1

Selected bond lengths (\AA).

O1–C8	1.2216 (18)	N1–C8	1.346 (2)
O2–C9	1.4059 (16)	N1–C5	1.4166 (19)

Table 2

Hydrogen-bond geometry (\AA , $^\circ$).

$D\text{---}H\cdots A$	$D\text{---}H$	$H\cdots A$	$D\cdots A$	$D\text{---}H\cdots A$
N1–H1A \cdots O1 ⁱ	0.86	2.32	3.131 (2)	158
C4–H4A \cdots O1 ⁱ	0.93	2.55	3.298 (2)	138
C6–H6A \cdots O1	0.93	2.40	2.920 (2)	115

Symmetry code: (i) $x, -y, z - \frac{1}{2}$.

All H atoms were located in a difference Fourier map and constrained to ride on their parent atoms, with $\text{C–H} = 0.93\text{--}0.97 \text{ \AA}$ and $\text{N–H} = 0.86 \text{ \AA}$, and with $U_{\text{iso}}(\text{H}) = 1.2U_{\text{eq}}(\text{C}, \text{N})$ [$U_{\text{iso}}(\text{H}) = 1.5U_{\text{eq}}(\text{C})$ 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).

This project was supported by the Special Project of Qingdao for Generalship of Science and Technology (No. 05-2-JC-80) and the Outstanding Adult-Young Scientific Research Encouraging Foundation of Shandong Province (No. 2005BS04007).

References

- Allen, F. H., Kennard, O., Watson, D. G., Brammer, L., Orpen, A. G. & Taylor, R. (1987). *J. Chem. Soc. Perkin Trans. 2*, pp. S1–19.
- Nardelli, M. (1995). *J. Appl. Cryst.* **28**, 659.
- Sheldrick, G. M. (1996). *SADABS*. University of Göttingen, Germany.
- Sheldrick, G. M. (1997). *SHELXTL* (Version 5.1). Bruker AXS, Inc., Madison, Wisconsin, USA.
- Siemens (1996). *SMART* and *SAINTE*. Siemens Analytical X-ray Instruments Inc., Madison, Wisconsin, USA.
- Spek, A. L. (2003). *J. Appl. Cryst.* **36**, 7–13.
- Wen, Y.-H., Wen, H.-L., Zhang, S.-S., Li, X.-M. & Zhang, K. (2006). *Acta Cryst.* **E62**, o682–o684.