Acta Crystallographica Section E
Structure Reports Online
ISSN 1600-5368

N,*N*′-Bis(2-methylphenyl)oxamide

Yong-Hong Wen, Li-Li Xu, Xue-Mei Li and Shu-Sheng Zhang*

College of Chemistry and Molecular Engineering, Qingdao University of Science and Technology, 266042 Qingdao, Shandong, People's Republic of China

Correspondence e-mail: shushzhang@126.com

Key indicators

Single-crystal X-ray study
T = 293 K
Mean σ(C–C) = 0.003 Å
R factor = 0.053
wR factor = 0.155
Data-to-parameter ratio = 14.3

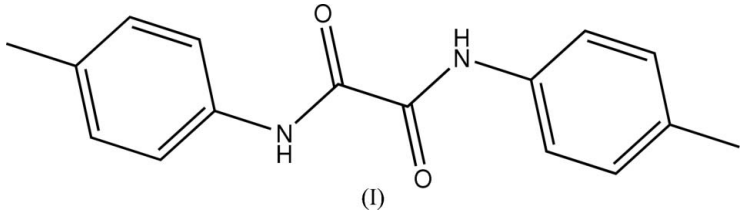
For details of how these key indicators were automatically derived from the article, see http://journals.iucr.org/e.

Received 5 June 2006
Accepted 5 July 2006

The molecule of the title compound, $C_{16}H_{16}N_2O_2$, lies on a crystallographically imposed center of symmetry at the midpoint of the C—C bond of the oxamide unit. An intramolecular C—H···O hydrogen bond forms a six-membered ring, and molecules are linked into ribbons along the *a* axis by N—H···O hydrogen bonds.

Comment

We have recently reported the synthesis and crystal structure of *N*,*N*′-diphenyloxalamide, (II) (Wen *et al.*, 2006). In our ongoing studies of oxalamide derivatives, the title compound, (I), was obtained.



(I)

The title compound has a crystallographically imposed center of symmetry at the mid-point of the C—C bond of the oxamide unit. Bond lengths and angles in (I) are within normal ranges (Allen *et al.*, 1987) and comparable to those found in (II). The dihedral angle between the benzene ring and the N1/C7/O1/N1^{i}/C7^{i}/O1^{i} plane [symmetry code: (i) $-x$, $1-y$, $-z$] is 24.5 (1)°. An intramolecular C5—H5*A*···O1 hydrogen bond forms a six-membered ring, and molecules are linked into ribbons along the *a* axis (Fig. 2) by N—H···O hydrogen bonds (Table 1).

Experimental

To a solution of 2-ethoxyaniline (21.8 g, 0.2 mol) in benzene (70 ml) was added dropwise a solution of oxalyl chloride (6.4 g, 0.05 mol) in benzene (30 ml), and the mixture was stirred at 343 K for 7 h. After

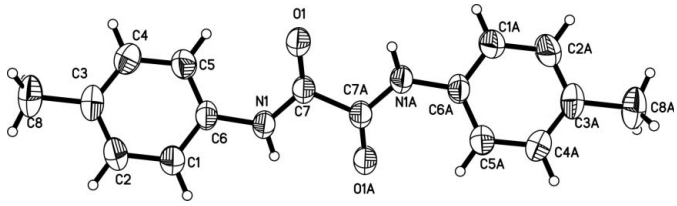


Figure 1
The structure of compound (I), showing 50% probability displacement ellipsoids and the atom-numbering scheme. [Symmetry code: (A) $-x$, $1-y$, $-z$.]

© 2006 International Union of Crystallography
All rights reserved

cooling to room temperature, water (50 ml) was added to the reaction and the organic phase was washed 3 times with water to give (I) as a solid. The title compound was obtained after drying at room temperature for 3 d. Colorless single crystals suitable for X-ray diffraction study were obtained by slow evaporation of an *N*,*N*-dimethylformamide (DMF) solution over a period of 9 h.

Crystal data

$C_{16}H_{16}N_2O_2$
M_r = 268.31
Triclinic, $P\overline{1}$
a = 5.3504 (13) Å
b = 6.7371 (17) Å
c = 10.228 (3) Å
α = 99.582 (4)°
β = 96.152 (4)°
γ = 106.527 (4)°
V = 343.83 (15) Å^3
Z = 1
D_x = 1.296 Mg m^{-3}
Mo $K\alpha$ radiation
μ = 0.09 mm^{-1}
T = 293 (2) K
Plate, colorless
0.26 × 0.18 × 0.07 mm

Data collection

Siemens SMART 1000 CCD area-detector diffractometer
ω scans
Absorption correction: multi-scan (*SADABS*; Sheldrick, 1996)
T_{min} = 0.978, T_{max} = 0.994
1934 measured reflections
1311 independent reflections
1111 reflections with $I > 2\sigma(I)$
R_{int} = 0.014
θ_{max} = 26.0°

Refinement

Refinement on F^2
$R[F^2 > 2\sigma(F^2)]$ = 0.053
$wR(F^2)$ = 0.156
S = 1.03
1311 reflections
92 parameters
H-atom parameters constrained
$w = 1/[\sigma^2(F_o^2) + (0.0866P)^2 + 0.0923P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} < 0.001$
$\Delta\rho_{max}$ = 0.23 e Å^{-3}
$\Delta\rho_{min}$ = −0.17 e Å^{-3}
Extinction correction: *SHELXL97*
Extinction coefficient: 0.14 (3)

Table 1
Hydrogen-bond geometry (Å, °).

D—H···A	D—H	H···A	D···A	D—H···A
N1—H1B···O1^{ii}	0.86	2.39	3.171 (3)	151
N1—H1B···O1^{i}	0.86	2.26	2.676 (2)	110
C5—H5A···O1	0.93	2.42	2.953 (3)	116

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

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

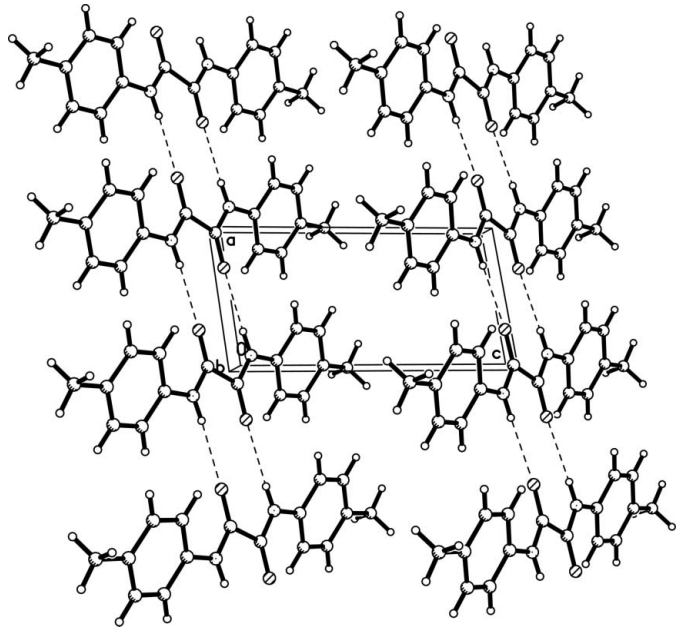


Figure 2
A view down the *b* axis, showing the ribbons generated by hydrogen bonds (indicated by dashed lines).

This project was supported by the Special Project of Qingdao for Leadership 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 *SAINT*. Siemens Analytical X-ray Instruments Inc., Madison, Wisconsin, USA.
Spek, A. L. (2003). *J. Appl. Cryst.* **36**, 7–13.
Wen, Y.-H., Li, X.-M., Wang, L. & Zhang, S.-S. (2006). *Acta Cryst.* E**62**, o2185–o2186.