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N,*N*′-Bis(4-nitrophenyl)oxamide

Yong-Hong Wen, Xin Shen, Sai Bi 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 = 0.048
wR factor = 0.120
Data-to-parameter ratio = 12.0

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

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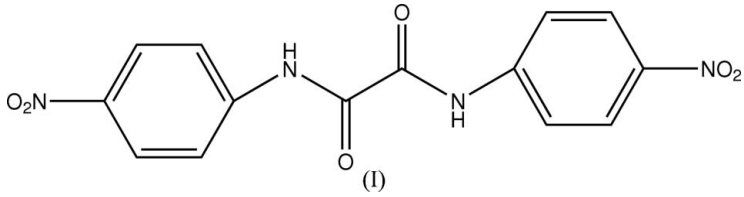
The molecule of the title compound, $C_{14}H_{10}N_4O_6$, lies on a crystallographically imposed center of symmetry at the mid-point of the C—C bond of the oxamide unit. Molecules are linked into zigzag chains by N—H···O hydrogen bonds. The packing is further stabilized by van der Waals forces.

Comment

N,*N*′-Diphenyloxamide and its derivatives have been widely applied in a number of materials as anti-oxidants, ultraviolet absorbents and/or metal ion passivators (Feng *et al.*, 1997). We report here the synthesis and crystal structure of *N*,*N*′-bis(4-nitrophenyl)oxamide, (I).



(I)

In the molecule of (I), the bond lengths and angles are within normal ranges (Allen *et al.*, 1987). These is a crystallographically imposed center of symmetry at the mid-point of the C7—C7^{i} bond [symmetry code: (i) $1-x, -y, -z$] and an intramolecular C5—H5*A*···O3 hydrogen bond forms a six-membered ring (Fig. 1).

In the crystal structure, molecules of (I) are linked into zigzag chains by intermolecular N2—H2*A*···O2^{ii} hydrogen bonds [symmetry code: (ii) $1+x, -\frac{1}{2}-y, \frac{1}{2}+z$] (Fig. 2 and Table 2). The packing is further stabilized by van der Waals forces.

Experimental

To a solution of 4-nitrophenylamine (27.6 g, 0.2 mol) in benzene (60 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

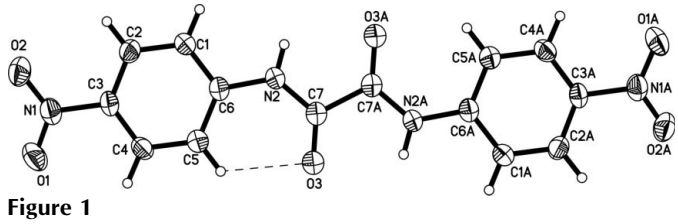


Figure 1
The structure of compound (I), showing 50% probability displacement ellipsoids and the atom-numbering scheme. The suffix A corresponds to symmetry code $(1-x, -y, -z)$. The dashed line indicates a hydrogen bond.

6 h. After cooling to room temperature, 50 ml water was added to the reaction and the organic phase was washed three times with water to give a solid. The title compound was obtained after drying at room temperature for 3 d. Colourless single crystals suitable for X-ray diffraction study were obtained by slow evaporation of a *N*,*N*-dimethylformamide (DMF) solution over a period of 6 d.

Crystal data

$C_{14}H_{10}N_4O_6$
M_r = 330.26
Monoclinic, $P2_1/c$
a = 3.7185 (8) Å
b = 13.942 (3) Å
c = 13.571 (3) Å
β = 99.059 (6)°
V = 694.8 (3) Å^3
Z = 2
D_x = 1.579 Mg m^{-3}
Mo $K\alpha$ radiation
μ = 0.13 mm^{-1}
T = 293 (2) K
Needle, colorless
0.50 × 0.08 × 0.06 mm

Data collection

Siemens SMART 1000 CCD area-detector diffractometer
ω scans
Absorption correction: multi-scan (*SADABS*; Sheldrick, 1996)
T_{min} = 0.939, T_{max} = 0.992
3825 measured reflections
1370 independent reflections
1001 reflections with $I > 2\sigma(I)$
R_{int} = 0.031
θ_{max} = 26.0°

Refinement

Refinement on F^2
$R[F^2 > 2\sigma(F^2)]$ = 0.048
$wR(F^2)$ = 0.120
S = 1.07
1370 reflections
114 parameters
H-atom parameters constrained
$w = 1/[\sigma^2(F_o^2) + (0.0562P)^2 + 0.0313P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} < 0.001$
$\Delta\rho_{max}$ = 0.24 e Å^{-3}
$\Delta\rho_{min}$ = −0.19 e Å^{-3}

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

D—H···A	D—H	H···A	D···A	D—H···A
N2—H2A···O3^{i}	0.86	2.25	2.677 (2)	110
N2—H2A···O2^{ii}	0.86	2.32	3.148 (2)	161
C5—H5A···O3	0.93	2.29	2.898 (3)	122

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

All H atoms were located in difference Fourier maps and constrained to ride on their parent atoms, with C—H = 0.93 Å and U_{iso}(H) = 1.2U_{eq}(C).

Data collection: *SMART* (Siemens, 1996); cell refinement: *SAINT* (Siemens, 1996); data reduction: *SAINT*; program(s) used to solve structure: *SHELXS97* (Sheldrick, 1997*a*); program(s) used to refine structure: *SHELXL97* (Sheldrick, 1997*a*); molecular graphics: *SHELXTL* (Sheldrick, 1997*b*); software used to prepare material for publication: *SHELXTL*, *PARST* (Nardelli, 1995) and *PLATON* (Spek, 2003).

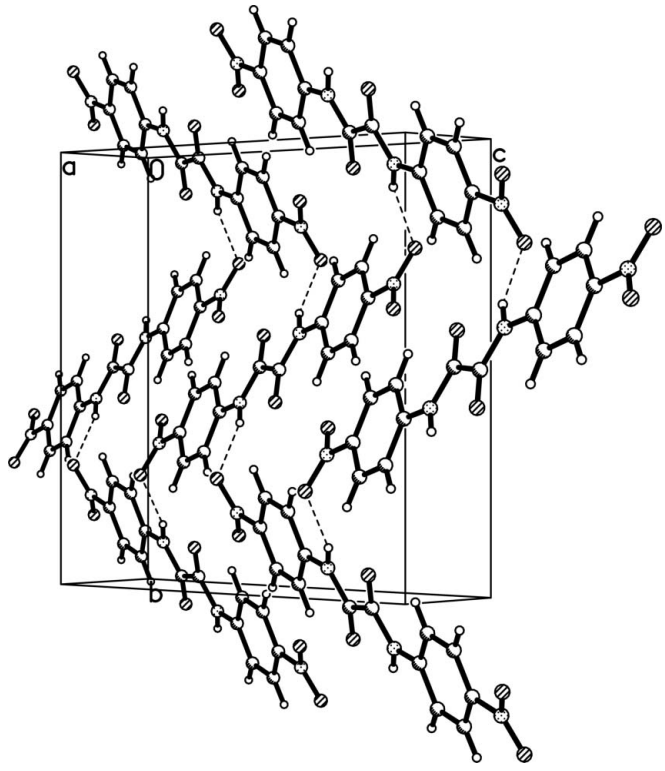


Figure 2
Packing diagram of (I), showing the hydrogen-bonded (dashed lines) zigzag chain.

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