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N,N′-Bis(2-pyridylethyl)oxamide

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Key indicators

Single-crystal X-ray study
T = 293 K
Mean σ(C–C) = 0.002 Å
R factor = 0.040
wR factor = 0.181
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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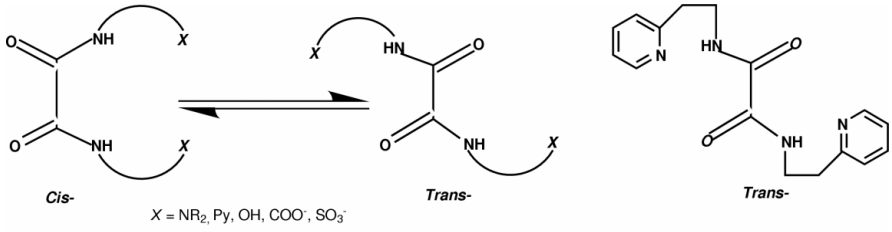
The molecular structure of the title compound, $C_{16}H_{18}N_4O_2$, adopts the *trans* conformation, and has an inversion center at the midpoint of the C—C bond in the oxamidate plane. The two phenyl planes are parallel, while the dihedral angle between the oxamidate plane and the phenyl ring is 2.5 (2)°.

Comment

N,N′-Disubstituted oxamidates are versatile ligands in coordination chemistry. They can adopt either the *cis*-(I) or *trans*-(II) conformation to form metal complexes, which are excellent building blocks for more complicated structures, such as metallosupramolecular frameworks (Chen, Qiu *et al.*, 1994). As part of our systematic study on the coordination chemistry of *N,N′*-disubstituted oxamidate ligands, we recently synthesized the ligand *N,N′*-bis(2-pyridylethyl)oxamide, (III), and analysed its crystal structure. An inversion center is located at the midpoint of the bond C8—C8^{i} (Table 1). Atoms C7 and C7^{i} are in the oxamidate plane, which is composed of atoms C8, C8^{i}, N2, N2^{i}, O1 and O1^{i}. The dihedral angle between the oxamidate plane and each of the phenyl planes is only 2.5 (2)°,



(I) (II) (III)

and the two phenyl rings are parallel to each other. The two C═O groups lie on opposite sides of the bond C8—C8^{i} and the molecule adopts the *trans* conformation. The torsion angle N2—C7—C6—C5 is 178.15 (12)°. Though atoms C8 and C8^{i} are sp^2 hybridized, the bond C8—C8^{i} is mainly a single bond and the bond length is 1.537 (3) Å, which is longer than that of a C═C double bond. Similar situations were found in the analogue *N,N′*-bis(2-pyridylmethyl)oxamide, in which the C—C bond lengths are 1.536 (7) and 1.513 (7) Å (Lloret *et al.*, 1989), as well as in other oxamide compounds (Sanada *et al.*, 1998; Ruiz *et al.*, 1997; Chen, Tang *et al*, 1994; Zhang *et al.*, 1996). In the crystal structure, symmetry-related molecules are linked by two types of intermolecular hydrogen bonds, *viz.* N—H···N and C—H···O. As indicated in Table 2, the hydrogen bond between N2 and N1^{ii} is much stronger than that between C1 and O1^{iii}.

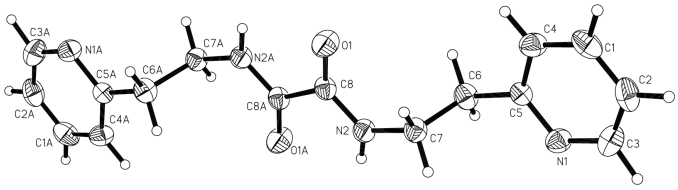


Figure 1
View of the molecule of (III), showing the atom-labelling scheme; the suffix A here corresponds to symmetry code i in the text and Table 1. Displacement ellipsoids are drawn at the 30% probability level.

Experimental

N,N′-Bis(2-pyridylethyl)oxamide was prepared according to a procedure similar to that reported by Ojima & Yamada (1970). An ethanol solution (50 ml) of diethyl oxalate (14.62 g, 0.10 mol) was added dropwise to a 100 ml ethanol solution of 2-(2-aminoethyl)-pyridine (24.24 g, 0.20 mol) with stirring. The mixture was then stirred and refluxed at 353 K for 1 h. The resulting solution was cooled to room temperature and a white precipitation appeared. The precipitate was filtered off and washed with water and diethyl ether. The yield was approximately 93%. Colourless single crystals were obtained by recrystallization from ethanol.

Crystal data

$C_{16}H_{18}N_4O_2$
M_r = 298.34
Monoclinic, $P2_1/c$
a = 9.489 (6) Å
b = 6.248 (4) Å
c = 13.062 (8) Å
β = 100.28 (1)°
V = 762.0 (8) Å^3
Z = 2
D_x = 1.300 Mg m^{-3}
Mo $K\alpha$ radiation
Cell parameters from 2131 reflections
θ = 2.2–26.8°
μ = 0.09 mm^{-1}
T = 293 (2) K
Prism, colourless
0.47 × 0.33 × 0.20 mm

Data collection

Bruker SMART CCD area-detector diffractometer
φ and ω scans
Absorption correction: multi-scan (*SADABS*; Sheldrick, 1996; Blessing, 1995)
T_{min} = 0.959, T_{max} = 0.982
4245 measured reflections
1642 independent reflections
1274 reflections with $I > 2\sigma(I)$
R_{int} = 0.018
θ_{max} = 27.1°
$h = -11 \rightarrow 12$
$k = -7 \rightarrow 7$
$l = -14 \rightarrow 16$

Refinement

Refinement on F^2
$R[F^2 > 2\sigma(F^2)]$ = 0.040
$wR(F^2)$ = 0.181
S = 1.06
1642 reflections
137 parameters
All H-atom parameters refined
$w = 1/[\sigma^2(F_o^2) + (0.1359P)^2]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max}$ = 0.001
$\Delta\rho_{max}$ = 0.22 e Å^{-3}
$\Delta\rho_{min}$ = −0.13 e Å^{-3}
Extinction correction: *SHELXL*97
Extinction coefficient: 0.07 (2)

Table 1
Selected geometric parameters (Å, °).

N2—C8	1.324 (2)	C8—C8^{i}	1.537 (3)
N2—C7	1.451 (2)	C7—C6	1.520 (2)
O1—C8	1.2224 (19)	C5—C6	1.502 (2)
C8—N2—C7	122.37 (13)	C5—C6—C7	111.21 (13)
N2—C7—C6	112.13 (13)		

Symmetry code: (i) $-x, -y, 2-z$.

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

D—H···A	D—H	H···A	D···A	D—H···A
N2—H2A···N1^{ii}	0.878 (19)	2.204 (19)	3.008 (3)	152.1 (17)
C1—H1···O1^{iii}	0.94 (2)	2.452 (19)	3.308 (3)	151.4 (17)

Symmetry codes: (ii) $-x, y-\frac{1}{2}, \frac{3}{2}-z$; (iii) $1-x, 1-y, 2-z$.

Data collection: *SMART* (Bruker, 1998); cell refinement: *SMART*; data reduction: *SAINT* (Bruker, 1998); program(s) used to solve structure: *SHELXS*97 (Sheldrick, 1997); program(s) used to refine structure: *SHELXL*97 (Sheldrick, 1997); molecular graphics: *SHELXTL* (Bruker, 1998); software used to prepare material for publication: *SHELXTL*.

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