Supplementary data for this paper are available from the IUCr electronic archives (Reference: AB1537). Services for accessing these data are described at the back of the journal.

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Diethyl *N*,*N*′-(Naphthalene-1,8-diyl)-dioxamate

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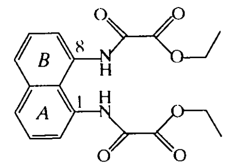
Abstract

The reaction of ethyloxalyl chloride and naphthalene-1,8-diamine in refluxing tetrahydrofuran yielded the title compound, $C_{18}H_{18}N_2O_6$. The crystal structure analysis reveals a molecule with one oxamate substituent nearly coplanar with the aromatic naphthalene system, while the second is strongly twisted with respect to the naphthalene system. Three strong intramolecular hydrogen bonds are observed, one in each oxamic acid ethyl ester group and one linking the two substituents. Symmetry-related molecules are connected by a fourth intermolecular hydrogen bond.

Comment

In recent years, special attention has been devoted to transition metal compounds with oxamate ligands. In particular, bimetallic chain compounds with Mn^{II} and Cu^{II} have been studied in order to understand their magnetic properties (Stumpf, Pei, Kahn *et al.*, 1993; Stumpf, Pei, Ouahab *et al.*, 1993). In addition, the presence of strong electron-donor atoms, such as the amide N atom, stabilizes high oxidation states for transition metals. Some electrochemical studies have been carried out with copper(II)–oxamate complexes (see, for example, Cervera *et al.*, 1998). In the present paper, we report the crystal structure of the diethyl ester, (I), of *N*,*N*′-(naphthalene-1,8-diyl)bis(oxamic acid), an oxamate ligand which has not been described before.



(I)

The molecular structure of the title compound together with the atom-numbering scheme is shown in Fig. 1. Two oxamic acid ethyl ester groups are connected to the naphthalene system in positions 1 and 8. A planar arrangement of the two substituents with respect to the naphthalene system is hindered due to the steric interactions between them. This results in one substituent (described by the best plane through atoms N2, O4, O5 and O6) being strongly twisted [57.5 (1)°] around the C8—N2 bond with respect to the plane described by aromatic ring *B* (C5–C10), while the other substituent (atoms N1, O1, O2 and O3) is more coplanar [14.0 (1)°] with aromatic ring *A* (C1–C4, C9, C10). The naphthalene system is also affected and exhibits a small distortion [dihedral angle *A*/*B* = 5.8 (1)°] and a large exocyclic

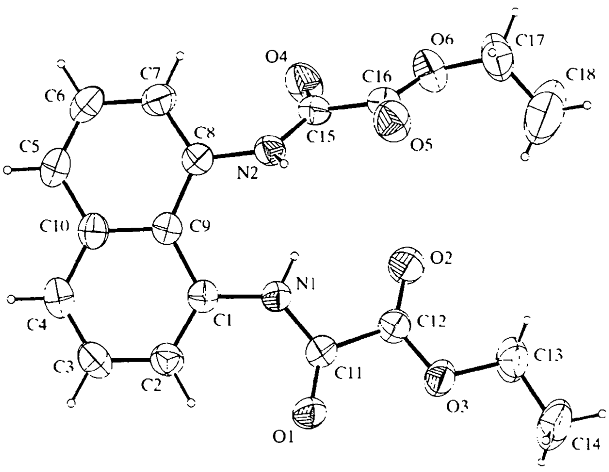


Fig. 1. *PLATON* (Spek, 1990) representation of the title compound showing 50% probability displacement ellipsoids.

angle [C1—C9—C8 126.8 (2)°]. The nearly planar arrangement of the substituents (atoms N1, O1, O2, O3, C11, C12 and C13 for the group connected to ring *A*, and atoms N2, O4, O5, O6, C15, C16 and C17 for the group connected to ring *B*) is stabilized by N—H···O intramolecular hydrogen bonds [N1—H···O2 2.659 (2) and N2—H···O5 2.701 (2) Å]. This formation of formal five-membered rings, including the H atoms, results in smaller than normal N—C—C bond angles [N1—C11—C12 110.0 (2) and N2—C15—C16 111.4 (2)°]. In the crystal, two molecules related by a centre of symmetry are linked by the formation of a hydrogen bond between the N2—H group and atom O1 of a neighbouring molecule.

Experimental

The title compound was prepared according to Cervera *et al.* (1998). The resulting precipitate was recrystallized from a dichloromethane–acetonitrile mixture (1:1) by slow evaporation of the solvent at room temperature.

Crystal data

$C_{18}H_{18}N_2O_6$
$M_r = 358.34$
Triclinic
$P\bar{1}$
$a = 8.4670\,(7)$ Å
$b = 9.7895\,(6)$ Å
$c = 11.0973\,(6)$ Å
$\alpha = 73.717\,(6)^\circ$
$\beta = 81.592\,(6)^\circ$
$\gamma = 87.474\,(9)^\circ$
$V = 873.44\,(10)$ Å^3
$Z = 2$
$D_x = 1.363$ Mg m^{-3}
D_m not measured

Mo $K\alpha$ radiation
$\lambda = 0.71073$ Å
Cell parameters from 24 reflections
$\theta = 12.5$–20.0°
$\mu = 0.104$ mm^{-1}
$T = 293\,(2)$ K
Block
0.76 × 0.68 × 0.68 mm
Transparent

Data collection

Stoe AED-4 diffractometer
$\omega/2\theta$ scans
Absorption correction: none
3242 measured reflections
3242 independent reflections
2842 reflections with $I > 2\sigma(I)$

$\theta_{\max} = 25.48^\circ$
$h = -10 \rightarrow 10$
$k = -11 \rightarrow 11$
$l = 0 \rightarrow 13$
2 standard reflections
frequency: 60 min
intensity decay: <2%

Refinement

Refinement on F^2
$R[F^2 > 2\sigma(F^2)] = 0.050$
$wR(F^2) = 0.199$
$S = 1.256$
3242 reflections
245 parameters
H atoms: see below
$w = 1/[\sigma^2(F_o^2) + (0.1133P)^2 + 0.1884P]$ where $P = (F_o^2 + 2F_c^2)/3$

$(\Delta/\sigma)_{\max} = 0.001$
$\Delta\rho_{\max} = 0.596$ e Å^{-3}
$\Delta\rho_{\min} = -0.571$ e Å^{-3}
Extinction correction: none
Scattering factors from *International Tables for Crystallography* (Vol. C)

Table 1. *Hydrogen-bonding geometry (Å, °)*

D—H···A	D—H	H···A	D···A	D—H···A
N1—H1N···N2	0.87 (3)	2.13 (3)	2.759 (2)	129 (2)
N1—H1N···O2	0.87 (3)	2.21 (3)	2.659 (2)	112 (2)
N2—H2N···O1^{i}	0.86 (3)	2.20 (3)	2.929 (2)	144 (2)
N2—H2N···O5	0.86 (3)	2.28 (3)	2.701 (2)	111 (2)

Symmetry code: (i) $1 - x, -y, 1 - z$.

The title structure was solved by direct methods. The positions of the amino H atoms were located from difference Fourier maps and refined freely. The remaining H atoms were included in calculated positions and treated as riding atoms using *SHELXL*97 (Sheldrick, 1997*a*) default parameters. Atoms C14 and C18, at the extremities of the side chains, undergo relatively strong thermal motion.

Data collection: *STADI*4 (Stoe & Cie, 1996*a*). Cell refinement: *STADI*4. Data reduction: *X-RED* (Stoe & Cie, 1996*b*). Program(s) used to solve structure: *SHELXS*97 (Sheldrick, 1997*b*). Program(s) used to refine structure: *SHELXL*97 (Sheldrick, 1997*a*). Molecular graphics: *PLATON* (Spek, 1990). Software used to prepare material for publication: *SHELXL*97.

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