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N,*N*′-Bis(2-hydroxyphenyl)naphthalene-1,4-dicarboxamide dimethyl sulfoxide solvate

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Key indicators

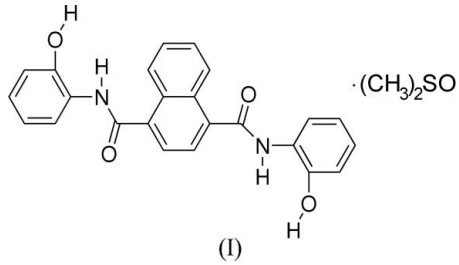
Single-crystal X-ray study
T = 153 K
Mean σ(C–C) = 0.002 Å
Disorder in solvent or counterion
R factor = 0.033
wR factor = 0.096
Data-to-parameter ratio = 14.5

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

The title compound, $C_{24}H_{18}N_2O_4\cdot C_2H_6OS$, adopts an *anti* C═O conformation. The two amide groups are twisted away from the attached central ring by 48.48 (5) and 46.49 (5)°. In the crystal structure, the molecules are connected by O—H···O and N—H···O hydrogen bonds, forming a two-dimensional network parallel to the (110) plane.

Comment

1,4-Naphthalenedicarboxylic acid derivatives are a class of intermediates important for applications as monomers in the preparation of polymers (Fukuzumi *et al.*, 1994; Tsukada *et al.*, 1994). Previously, we have reported the crystal structures of *N*,*N*′-bis(4-nitrophenyl)naphthalene-1,4-dicarboxamide dimethylsulfoxide disolvate (Jing, Qin, Gu, Zhang & Mao, 2006) and *N*,*N*′-bis(2-methoxyphenyl)naphthalene-1,4-dicarboxamide (Jing, Qin, Gu, Zhang & Lei, 2006). We now report the crystal structure of the title compound, (I).



(I)

The bond lengths and angles in (I) are normal. The naphthalene ring system is planar, with a maximum deviation of 0.030 (1) Å for atom C5. The two C═O groups are in an *anti* conformation. As a result of steric effects, the substituent groups at atoms C1 and C4 are twisted away from the plane of the naphthalene ring system (Fig. 1). The O1/N1/C11/C12 and O3/N2/C18/C19 planes form dihedral angles of 48.48 (5) and 46.49 (5)°, respectively, with the C1–C4/C9/C10 plane. The O1/N1/C11/C12 and C12–C17 planes are inclined at an angle of 32.98 (6)°, while the O3/N2/C18/C19 and C19–C24 planes make a dihedral angle of 58.68 (6)°.

The crystal packing is stabilized by N—H···O, O—H···O and C—H···O hydrogen bonds (Table 1). The main molecules are linked into a two-dimensional network parallel to the (110) plane by N—H···O and O—H···O hydrogen bonds.

Experimental

Naphthalene-1,4-dicarboxylic acid (2 mmol) and an excess of thionyl chloride (6 mmol) in dioxane (20 ml) were boiled under reflux for 6 h. The solution was distilled under reduced pressure and a yellow solid

was formed. 2-Aminophenol (4 mmol) in tetrahydrofuran (20 ml) was added to the yellow solid and boiled under reflux for 1 d. The solution was then cooled to ambient temperature and filtered to remove the tetrahydrofuran. The precipitate was dissolved in dimethylsulfoxide and allowed to stand for one month at ambient temperature, after which time colourless single crystals of (I) suitable for X-ray diffraction were obtained.

Crystal data

$C_{24}H_{18}N_2O_4\cdot C_2H_6OS$
$M_r = 476.53$
Triclinic, $P\overline{1}$
$a = 9.5769\ (3)$ Å
$b = 9.6804\ (2)$ Å
$c = 13.0811\ (3)$ Å
$\alpha = 109.796\ (1)^\circ$
$\beta = 98.157\ (1)^\circ$
$\gamma = 91.369\ (1)^\circ$
$V = 1126.14\ (5)$ Å^3
$Z = 2$
$D_x = 1.405$ Mg m^{-3}
Mo $K\alpha$ radiation
$\mu = 0.19$ mm^{-1}
$T = 153\ (2)$ K
Block, colourless
$0.33 \times 0.18 \times 0.16$ mm

Data collection

Rigaku R-AXIS RAPID diffractometer
ω scans
Absorption correction: multi-scan (*ABSCOR*; Higashi, 1995)
$T_{min} = 0.941$, $T_{max} = 0.971$
11181 measured reflections
5129 independent reflections
4544 reflections with $I > 2\sigma(I)$
$R_{int} = 0.013$
$\theta_{max} = 27.5^\circ$

Refinement

Refinement on F^2
$R[F^2 > 2\sigma(F^2)] = 0.033$
$wR(F^2) = 0.096$
$S = 1.01$
5129 reflections
353 parameters
H atoms treated by a mixture of independent and constrained refinement
$w = 1/[\sigma^2(F_o^2) + (0.0566P)^2 + 0.38P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} = 0.001$
$\Delta\rho_{max} = 0.42$ e Å^{-3}
$\Delta\rho_{min} = -0.27$ e Å^{-3}
Extinction correction: *SHELXL97*
Extinction coefficient: 0.0088 (17)

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

D—H···A	D—H	H···A	D···A	D—H···A
N1—H1N···O2^{i}	0.872 (16)	2.170 (17)	2.9796 (12)	154 (1)
N2—H2N···O1^{ii}	0.886 (16)	1.985 (17)	2.8010 (12)	153 (2)
O2—H2O···O5	0.90 (2)	1.70 (2)	2.5972 (15)	176 (2)
O2—H2O···O5′	0.90 (2)	1.80 (2)	2.644 (12)	157 (2)
O4—H4O···O3^{iii}	0.91 (2)	1.89 (2)	2.7978 (12)	174 (2)
O4—H4O···O4^{iii}	0.91 (2)	2.47 (2)	2.8146 (17)	103 (2)
C7—H7···O3^{iv}	0.95	2.57	3.5029 (14)	166

Symmetry codes: (i) $-x, -y+2, -z+1$; (ii) $-x, -y+2, -z$; (iii) $-x+1, -y+1, -z$; (iv) $x, y+1, z$.

The dimethylsulfoxide molecule is disordered over two positions with occupancy factors of 0.9232 (16) and 0.0768 (16). The same anisotropic displacement parameters were used for the major and minor components of the methyl C atoms. The corresponding bond lengths in the major and minor components were restrained to be the same. N-bound and O-bound H atoms were located in a difference Fourier map and refined isotropically [N—H = 0.872 (16) and 0.886 (16) Å, O—H = 0.90 (2) and 0.91 (2) Å]. The C-bound H atoms were placed in calculated positions, with C—H = 0.95 or 0.98 Å, and refined using a riding model, with $U_{iso}(H) = 1.2U_{eq}(C)$. The methyl groups were allowed to rotate but not to tip.

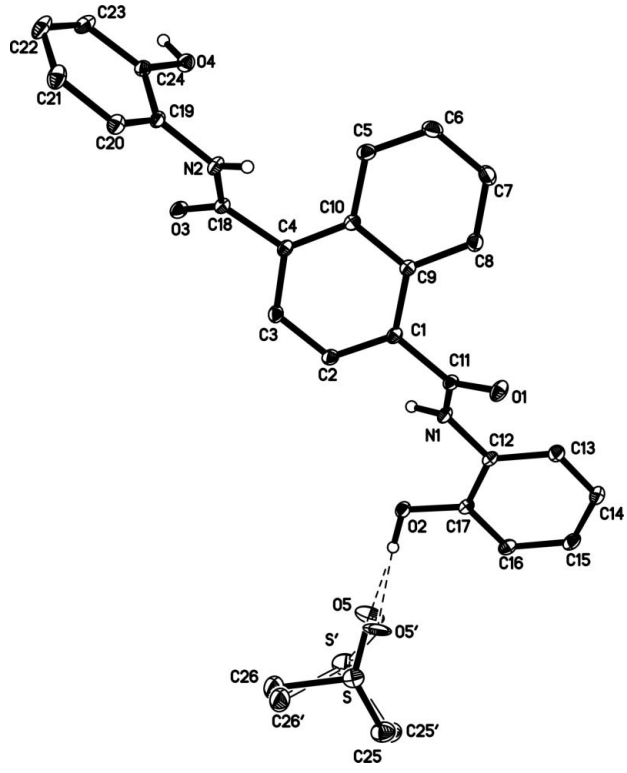


Figure 1
The molecular structure of (I), showing 30% probability displacement ellipsoids and the atomic numbering. Both disordered components of the dimethylsulfoxide molecule are shown. The C-bound H atoms have been omitted for clarity. The hydrogen bonds are shown as dashed lines.

Data collection: *RAPID-AUTO* (Rigaku, 2004); cell refinement: *RAPID-AUTO*; data reduction: *RAPID-AUTO*; program(s) used to solve structure: *SHELXS97* (Sheldrick, 1997); program(s) used to refine structure: *SHELXL97* (Sheldrick, 1997); molecular graphics: *XP* in *SHELXTL* (Bruker, 1997); software used to prepare material for publication: *SHELXL97*.

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