

N,N'*-Diphenyl-naphthalene-1,4-dicarboxamide dimethylacetamide disolvate*Ming-Qi Li and Lin-Hai Jing***

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The title compound, $C_{24}H_{18}N_2O_2 \cdot 2C_4H_9NO$, crystallizes in an anti C=O orientation. The two amide groups are twisted away from the attached ring by 71.75 (4) and 88.13 (5)°. The crystal packing is stabilized by N—H···O and C—H···O hydrogen bonds.

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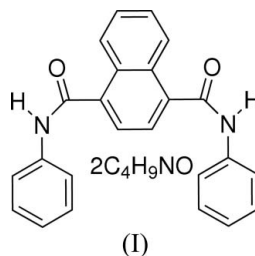
Key indicators

Single-crystal X-ray study
 $T = 153$ K
 Mean $\sigma(C-C) = 0.002$ Å
 Disorder in solvent or counterion
 R factor = 0.044
 wR factor = 0.130
 Data-to-parameter ratio = 13.6

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

Comment

Naphthalene-1,4-dicarboxylic 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 dimethyl naphthalene-1,4-dicarboxylate (Jing *et al.*, 2005), diphenyl naphthalene-1,4-dicarboxylate (Jing *et al.*, 2006a), *N,N*-bis(4-nitrophenyl)naphthalene-1,4-dicarboxamide bis-(dimethylsulfoxide) solvate (Jing *et al.*, 2006b), di-*n*-propyl naphthalene-1,4-dicarboxylate (Jing, Qin, Zhang, Gu & Lei, 2006) and diethyl naphthalene-1,4-dicarboxylate (Jing, Qin, Zhang, Gu & Mao, 2006). We now report the crystal structure of the title compound, (I).



The bond lengths and angles in (I) are normal. The naphthalene ring system is planar, with a maximum deviation of 0.020 (1) Å for atom C4. The two C=O groups possess an *anti* orientation. 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 O2/N2/C18/C19 planes form dihedral angles of 71.75 (4) and 88.13 (5)°, respectively, with the plane formed by atoms C1–C4/C9/C10. The O1/N1/C11/C12 and C12–C17 planes are inclined at an angle of 10.23 (7)°, while the O2/N2/C18/C19 and C19–C24 planes make a dihedral angle of 9.17 (12)°. The crystal packing is stabilized by N—H···O and C—H···O hydrogen bonds (Table 1).

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

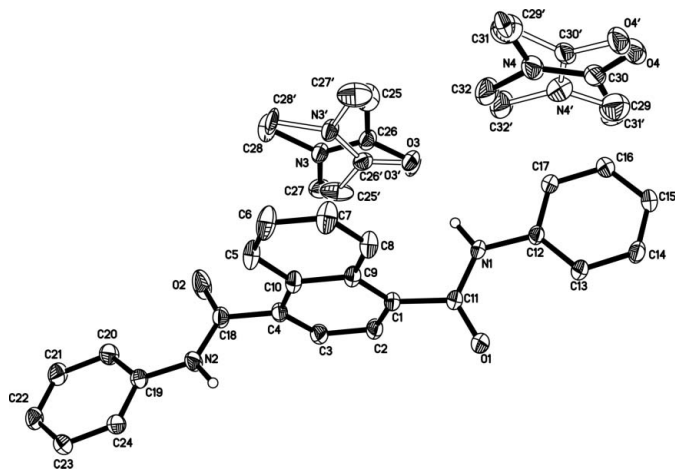


Figure 1
The structure of (I), showing 30% probability displacement ellipsoids and the atomic numbering. All the disordered components are shown. H atoms have been omitted for clarity.

was obtained. Aniline (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 dimethylacetamide 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

$C_{24}H_{18}N_2O_2 \cdot 2C_4H_9NO$
 $M_r = 540.65$
 Monoclinic, $P2_1/c$
 $a = 12.3070$ (3) Å
 $b = 20.1236$ (6) Å
 $c = 12.1210$ (4) Å
 $\beta = 108.173$ (1)°
 $V = 2852.16$ (14) Å³
 $Z = 4$
 $D_x = 1.259$ Mg m⁻³
 Mo $K\alpha$ radiation
 $\mu = 0.08$ mm⁻¹
 $T = 153$ (2) K
 Block, colourless
 $0.57 \times 0.32 \times 0.28$ mm

Data collection

Rigaku R-AXIS RAPID diffractometer
 ω scans
 Absorption correction: none
 27820 measured reflections
 6539 independent reflections
 5513 reflections with $I > 2\sigma(I)$
 $R_{int} = 0.016$
 $\theta_{max} = 27.5^\circ$

Refinement

Refinement on F^2
 $R[F^2 > 2\sigma(F^2)] = 0.044$
 $wR(F^2) = 0.130$
 $S = 1.00$
 6539 reflections
 480 parameters
 H atoms treated by a mixture of independent and constrained refinement
 $w = 1/[\sigma^2(F_o^2) + (0.076P)^2 + 0.58P]$
 where $P = (F_o^2 + 2F_c^2)/3$
 $(\Delta/\sigma)_{max} = 0.001$
 $\Delta\rho_{max} = 0.43$ e Å⁻³
 $\Delta\rho_{min} = -0.43$ e Å⁻³
 Extinction correction: *SHELXL97*
 Extinction coefficient: 0.0086 (11)

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

<i>D</i> —H··· <i>A</i>	<i>D</i> —H	H··· <i>A</i>	<i>D</i> ··· <i>A</i>	<i>D</i> —H··· <i>A</i>
N1—H1N···O3	0.912 (16)	1.975 (17)	2.883 (5)	174 (1)
N1—H1N···O3 ⁱ	0.912 (16)	1.91 (2)	2.818 (16)	174 (1)
N2—H2N···O4 ⁱ	0.905 (17)	1.937 (17)	2.829 (3)	169 (1)
N2—H2N···O4 ⁱⁱ	0.905 (17)	1.936 (18)	2.836 (6)	172 (1)
C13—H13···O1	0.95	2.29	2.8866 (16)	120
C14—H14···O2 ⁱⁱ	0.95	2.43	3.2491 (19)	144
C16—H16···O4 ⁱⁱⁱ	0.95	2.42	3.306 (4)	155
C20—H20···O2	0.95	2.28	2.878 (2)	120

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

Both dimethylacetamide molecules are disordered over two positions. The site-occupation factors for the disordered atoms were refined to 0.705 (3) and 0.295 (3), respectively, for the major and minor components of one of the dimethylacetamide molecules (with O3), and to 0.704 (3) and 0.296 (3), respectively, for the major and minor components of the other molecule. The corresponding bond lengths in the major and minor components were restrained to be equal. N-bound H atoms were located in a difference Fourier map and refined isotropically [$N-H = 0.912$ (16) and 0.905 (17) 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 $U_{iso}(H) = 1.2U_{eq}(C)$ or $1.0U_{eq}(\text{methyl } C)$. The methyl groups were allowed to rotate but not to tip.

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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