Acta Crystallographica Section E
Structure Reports
Online

ISSN 1600-5368

N,N′-Bis(6-*tert*-butoxycarbonylamino-2-pyridyl)-terephthalamide dimethyl sulfoxide disolvate

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Key indicators

Single-crystal X-ray study
T = 295 K
Mean σ(C–C) = 0.016 Å
R factor = 0.061
wR factor = 0.207
Data-to-parameter ratio = 11.0

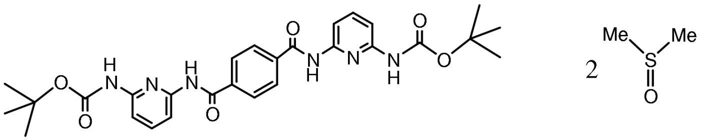
For details of how these key indicators were automatically derived from the article, see http://journals.iucr.org/e.

In the title compound, $C_{28}H_{32}N_6O_6{\cdot}2C_2H_6OS$, the host molecules are linked by N—H···N hydrogen bonds at both of the terminal amide groups, forming zigzag chains along the $[01\bar{1}]$ direction. Two dimethyl sulfoxide molecules are connected to a host molecule by N—H···O hydrogen bonds.

Received 2 December 2004
Accepted 16 December 2004
Online 24 December 2004

Comment

Oligocarboxamide molecular strands self-organize into helical structures (Berl, Huc *et al.*, 2000; Berl, Krische *et al.*, 2000; Berl *et al.*, 2001*a*,*b*). Recently, we found that oligocarboxamides form inclusion compounds with various solvent molecules. The structure of the title compound, (I), has been determined to reveal the site of the host molecule to which the guest solvent molecules are connected.



(I)

The host molecule shows an extended and slightly curved structure, having a local non-crystallographic center of symmetry (Fig. 1). The dihedral angles between each amide group and the aromatic rings to which is it bonded are

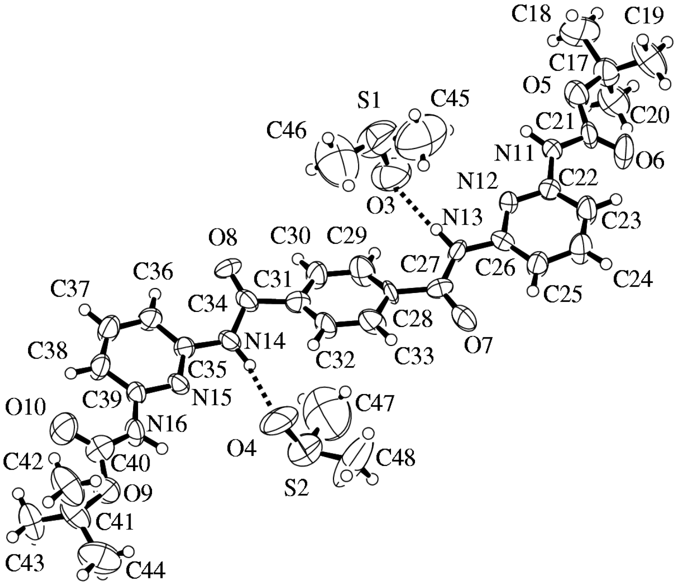


Figure 1
The molecular structure of (I), showing displacement ellipsoids at the 50% probability level. Dashed lines indicate hydrogen bonds.

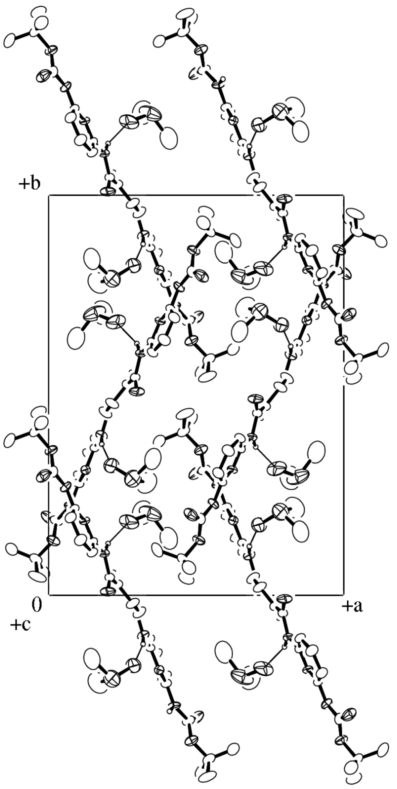


Figure 2
The crystal structure of (I), projected along the *c* axis. Thin lines indicate hydrogen bonds.

14.0 (2)–30.7 (3)°, except for that of 7.8 (2)° between the amide O7/C27/N13 group and the N12/C22–C26 pyridine ring. The dihedral angles between the central benzene (C28–C33) and the pyridine rings (N12/C22–C26 and N15/C35–C39) are 15.5 (4) and 20.2 (4)°, respectively, and that between the two pyridine rings is 12.1 (3)°. The guest dimethyl sulfoxide molecules are connected to the host molecule by N13—H13···O3 and N14—H14···O4 hydrogen bonds (Table 2). In the crystal structure, the host molecules related by the *n*-glide perpendicular to the *a* axis are connected by N11—H11···N15^{i} [symmetry code: (i) $\frac{3}{2} - x, \frac{1}{2} + y, z - \frac{1}{2}$] and N12···H16^{i}—N16^{i} paired hydrogen bonds, forming zigzag chains along the $[01\overline{1}]$ direction (Fig. 2).

Experimental

Compound (I) was prepared according to a procedure described previously for a similar compound (Berl *et al.*, 2001*a*). The crystals were grown from a dimethyl sulfoxide solution. A crystal specimen was sealed in a capillary to avoid efflorescence.

Crystal data

$C_{28}H_{32}N_6O_6{\cdot}2C_2H_6OS$
$M_r = 704.85$
Orthorhombic, $Pna2_1$
$a = 16.597$ (3) Å
$b = 22.522$ (3) Å
$c = 9.871$ (2) Å
$V = 3689.8$ (11) Å^3
$Z = 4$
$D_x = 1.269$ Mg m^{-3}

Mo Kα radiation
Cell parameters from 30 reflections
$\theta = 10.2$–12.9°
$\mu = 0.20$ mm^{-1}
$T = 295$ K
Prism, colorless
0.5 × 0.3 × 0.2 mm

Data collection

Rigaku AFC-7*R* diffractometer
ω scans
Absorption correction: by integration (*ABSCOR*; Higashi, 1999)
$T_{\min} = 0.921$, $T_{\max} = 0.949$
5747 measured reflections
4775 independent reflections
1742 reflections with $I > 2\sigma(I)$

$R_{\rm int} = 0.037$
$\theta_{\max} = 27.5°$
$h = -9 \rightarrow 21$
$k = 0 \rightarrow 29$
$l = -12 \rightarrow 5$
3 standard reflections
every 150 reflections
intensity decay: 7.4%

Refinement

Refinement on F^2
$R[F^2 > 2\sigma(F^2)] = 0.061$
$wR(F^2) = 0.207$
$S = 0.95$
4775 reflections
433 parameters

H-atom parameters constrained
$w = 1/[\sigma^2(F_o^2) + (0.0925P)^2]$
where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{\max} = 0.001$
$\Delta\rho_{\max} = 0.29$ e Å^{-3}
$\Delta\rho_{\min} = -0.23$ e Å^{-3}

Table 1
Selected torsion angles (°).

O5—C21—N11—C22	−179.3 (8)	N14—C34—C31—C30	152.4 (9)
O9—C40—N16—C39	169.0 (8)	N15—C35—N14—C34	−164.4 (8)
N11—C21—O5—C17	−170.6 (8)	N15—C39—N16—C40	174.2 (9)
N12—C22—N11—C21	−160.9 (9)	N16—C40—O9—C41	170.7 (9)
N12—C26—N13—C27	170.8 (8)	C26—N13—C27—C28	−179.1 (8)
N13—C27—C28—C33	−159.2 (9)	C31—C34—N14—C35	170.9 (8)

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

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
N11—H11···N15^{i}	0.95	2.02	2.968 (11)	176
N13—H13···O3	0.95	2.13	3.055 (12)	165
N14—H14···O4	0.95	1.98	2.864 (13)	153
N16—H16···N12^{ii}	0.95	2.15	3.083 (11)	167

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

All the H atoms were positioned geometrically and refined as riding, with C—H and N—H distances of 0.95 Å, and with $U_{\rm iso}$(H) = $1.2U_{\rm eq}$(parent atom). Methyl groups C18, C19, C20, C42, C43 and C44 show relatively large displacement parameters, suggesting rotational disorder of the *tert*-butyl groups.

Data collection: *WinAFC Diffractometer Control Software* (Rigaku, 1999); cell refinement: *WinAFC Diffractometer Control Software;* data reduction: *TEXSAN* (Molecular Structure Corporation, 2001); program(s) used to solve structure: *SIR*92 (Altomare *et al.*, 1994); program(s) used to refine structure: *SHELXL*97 (Sheldrick, 1997); molecular graphics: *ORTEP*II (Johnson, 1976); software used to prepare material for publication: *TEXSAN*.

HM thanks the Yamada Science Foundation for financial assistance.

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Acta Cryst. (2005). E**61**, o182–o184 Shigeru Ohba *et al.* • $C_{28}H_{32}N_6O_6{\cdot}2C_2H_6OS$ **o183**

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