Acta Crystallographica Section E
Structure Reports Online
ISSN 1600-5368

3,3′-Dimethyl-2,2′-dinitro-*N*,*N*′-(*o*-phenylene)-dibenzamide

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Received 8 May 2006
Accepted 23 May 2006

Key indicators

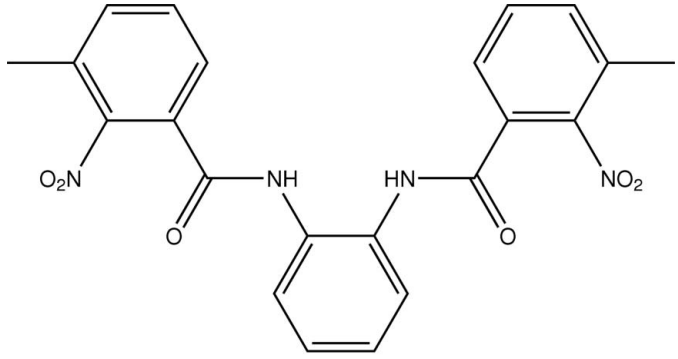
Single-crystal X-ray study
T = 293 K
Mean σ(C–C) = 0.004 Å
R factor = 0.063
wR factor = 0.186
Data-to-parameter ratio = 13.6

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

In the crystal structure of the title compound, $C_{22}H_{18}N_4O_6$, molecules are linked into ribbons along the *a* axis by N—H···O hydrogen bonds. The packing is further stabilized by a π–π interaction.

Comment

We have previously reported the structure of an amide-type acyclic polyether with 1,3-dihydroxybenzene as the backbone (Wen *et al.*, 2004). In our ongoing studies of polyamide-type compounds, the title compound, (I), has been synthesized.



(I)

The bond lengths and angles in (I) show normal values (Allen *et al.*, 1987). The dihedral angles between the central benzene ring (C8–C13) and the two outer benzene rings (C1–C6 and C15–C20) are 19.7 (1) and 8.9 (1)°, respectively, while the two outer benzene rings make a dihedral angle of 13.1 (1)°. Both methyl groups are involved in intramolecular C—H···O interactions with the nitro O atoms (Table 1). In the crystal structure, molecules are linked into ribbons along the *a* axis (Fig. 2) *via* N—H···O hydrogen bonds (Table 1). The packing is further stabilized by a π–π interaction between the C15–C20 benzene rings; the distance between the centroids, $Cg\cdots Cg^{iii}$, and the interplanar distance are 3.806 (2) and 3.422 (2) Å, respectively [symmetry code: (iii) $2-x, -y, 1-z$].

Experimental

A solution of 3-methyl-2-nitrobenzoyl chloride (3.71 g, 30 mmol) in CH_2Cl_2 (40 ml) was added dropwise over a period of 2 h to a solution of *o*-diaminobenzene (3.24 g, 30 mmol) and Et_3N (8.3 ml) in CH_2Cl_2 (50 ml). After the addition was completed, the reaction mixture was stirred at 273 K for 1 h and at room temperature for 3 h. The volatiles were removed *in vacuo* to give an off-white solid. Single crystals were obtained by slow evaporation of a glacial AcOH solution at room temperature over a period of one week.

Crystal data

$C_{22}H_{18}N_4O_6$
$M_r = 434.40$
Triclinic, $P\overline{1}$
$a = 9.607$ (2) Å
$b = 10.542$ (2) Å
$c = 11.408$ (3) Å
$\alpha = 86.301$ (3)°
$\beta = 65.394$ (3)°
$\gamma = 77.029$ (3)°
$V = 1023.1$ (4) Å^3
$Z = 2$
$D_x = 1.410$ Mg m^{-3}
Mo $K\alpha$ radiation
$\mu = 0.11$ mm^{-1}
$T = 293$ (2) K
Column, pale yellow
0.34 × 0.26 × 0.23 mm

Data collection

Siemens SMART 1000 CCD area-detector diffractometer
ω scans
Absorption correction: multi-scan (*SADABS*; Sheldrick, 1996)
$T_{min} = 0.965$, $T_{max} = 0.976$
5827 measured reflections
3943 independent reflections
3019 reflections with $I > 2\sigma(I)$
$R_{int} = 0.013$
$\theta_{max} = 26.1°$

Refinement

Refinement on F^2
$R[F^2 > 2\sigma(F^2)] = 0.063$
$wR(F^2) = 0.186$
$S = 1.03$
3943 reflections
289 parameters
H-atom parameters constrained
$w = 1/[\sigma^2(F_o^2) + (0.0944P)^2 + 0.5433P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} < 0.001$
$\Delta\rho_{max} = 0.73$ e Å^{-3}
$\Delta\rho_{min} = -0.31$ e Å^{-3}

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

D—H···A	D—H	H···A	D···A	D—H···A
N2—H2A···O4^{i}	0.86	2.19	3.044 (3)	169
N3—H3A···O3^{ii}	0.86	2.17	2.950 (3)	151
C21—H21C···O1	0.96	2.44	2.914 (5)	110
C22—H22A···O5	0.96	2.26	2.934 (4)	126

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

All H atoms were located in difference Fourier maps and constrained to ride on their parent atoms, with C—H = 0.93–0.96 Å, N—H = 0.86 Å and U_{iso}(H) = 1.2U_{eq}(C,N) or 1.5U_{eq}(methyl C).

Data collection: *SMART* (Siemens, 1996); cell refinement: *SAINT* (Siemens, 1996); data reduction: *SAINT*; program(s) used to solve structure: *SHELXTL* (Sheldrick, 1997); program(s) used to refine structure: *SHELXTL*; molecular graphics: *SHELXTL*; software used to prepare material for publication: *SHELXTL*, *PARST* (Nardelli, 1995) and *PLATON* (Spek, 2003).

This project was supported by the Special Project of Qingdao for Leadership of Science and Technology (No. 05-2-JC-80) and the Outstanding Adult-Young Scientific Research Encouraging Foundation of Shandong Province (No. 2005BS04007).

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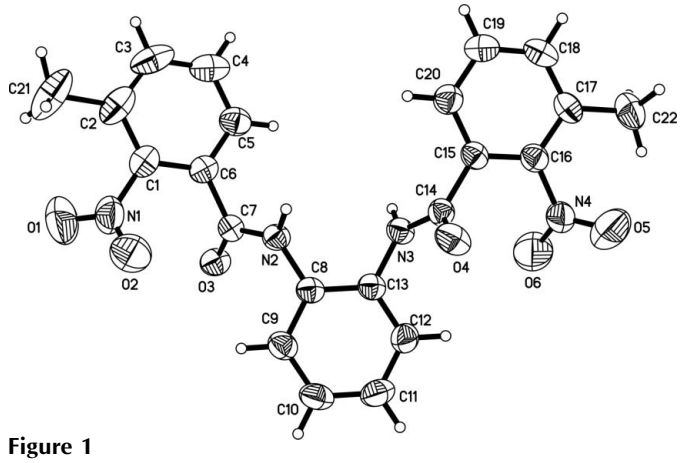

Figure 1
The molecular structure of (I), showing 50% probability displacement ellipsoids and the atom-numbering scheme.

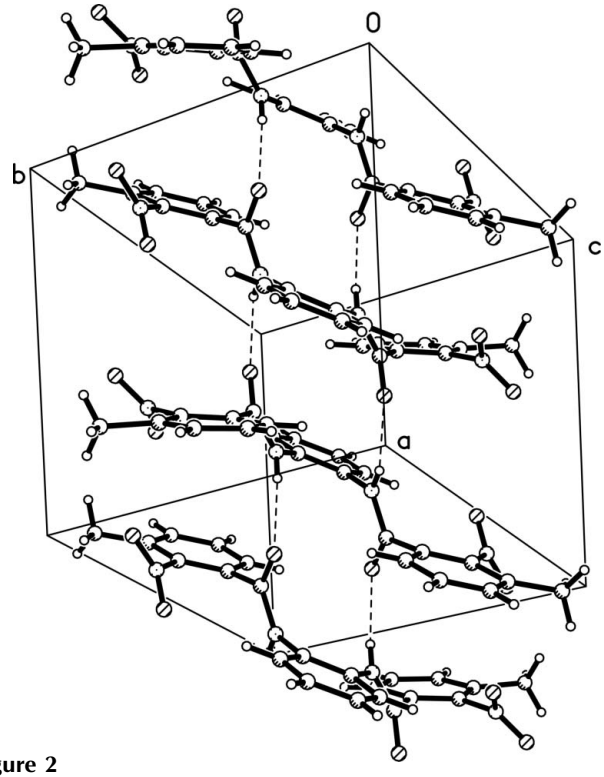

Figure 2
The molecular ribbon of (I) along the *a* axis. Hydrogen bonds are indicated by dashed lines.

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