

N,N'-[Methylenedi(3,5-dimethyl-*o*-phenylene)]dipthalimide

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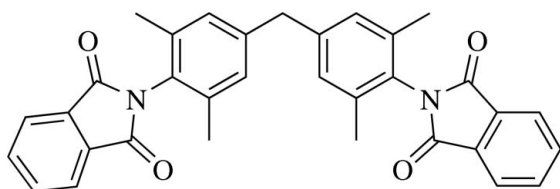
Received 10 September 2007; accepted 21 September 2007

Key indicators: single-crystal X-ray study; *T* = 292 K; mean $\sigma(\text{C}-\text{C})$ = 0.004 Å; *R* factor = 0.049; *wR* factor = 0.135; data-to-parameter ratio = 9.8.

In the structure of the title compound, $\text{C}_{33}\text{H}_{26}\text{N}_2\text{O}_4$, two phthalimide units are symmetrically linked by a bis(3,5-dimethylphenyl)methane bridge. The methylene C atom of this bridge lies on a twofold rotation axis. The dihedral angle between the planes of the two central benzene rings is 61.4 (4)°. The terminal isoindole group is approximately planar, with an r.m.s. deviation of atoms from the mean plane of 0.012 Å and a dihedral angle of 75.3 (14)° with the attached benzene ring. An extensive network of C—H...O hydrogen bonds stabilizes the crystal structure.

Related literature

For details of the biological activity and uses of bis(imide) derivatives, see: Rich *et al.* (1975); Degenhardt *et al.* (2002); Mallakpour & Kowsari (2004); Zhang *et al.* (1999); Langhals & Kirner (2000); Yakimov & Forrest (2002). For a related structure, see: Li *et al.* (2007).



Experimental

Crystal data

$\text{C}_{33}\text{H}_{26}\text{N}_2\text{O}_4$
M_r = 514.56
 Orthorhombic, *P*2₁2₁2
a = 16.221 (3) Å
b = 8.4722 (18) Å
c = 10.136 (2) Å
V = 1392.9 (5) Å³
Z = 2
 Mo *K*α radiation
 μ = 0.08 mm⁻¹
T = 292 (2) K
 0.20 × 0.20 × 0.10 mm

Data collection

Bruker SMART 4K CCD area-detector diffractometer
 Absorption correction: none
 9315 measured reflections
 1762 independent reflections
 1223 reflections with *I* > 2σ(*I*)
R_{int} = 0.035

Refinement

$R[F^2 > 2\sigma(F^2)]$ = 0.049
 $wR(F^2)$ = 0.135
S = 1.03
 1762 reflections
 179 parameters
 H-atom parameters constrained
 $\Delta\rho_{\text{max}}$ = 0.13 e Å⁻³
 $\Delta\rho_{\text{min}}$ = -0.14 e Å⁻³

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>
C4—H4...O1 ⁱ	0.93	2.48	3.304 (4)	148
C16—H16...O2 ⁱⁱ	0.93	2.33	3.213 (4)	159
C9—H9A...O2 ⁱⁱⁱ	0.96	2.67	3.545 (4)	151

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

Data collection: *SMART* (Bruker, 2001); cell refinement: *SAINTE* (Bruker, 2001); data reduction: *SAINTE* (Bruker, 2001); program(s) used to solve structure: *SHELXS97* (Sheldrick, 1997); program(s) used to refine structure: *SHELXL97* (Sheldrick, 1997); molecular graphics: *SHELXTL* (Bruker, 1997); software used to prepare material for publication: *SHELXTL*.

The authors thank Dr Meng Xiang-Gao for the X-ray data collection.

Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: SJ2357).

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supplementary materials

Acta Cryst. (2007). E63, o4176 [doi:10.1107/S1600536807046624]

N,N'-[Methylenedi(3,5-dimethyl-*o*-phenylene)]diphthalimide

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Comment

Bisimides are heterocyclic compounds, some of which have biological activity (Rich *et al.*, 1975). Moreover, they are synthetic precursors with applications in organic synthesis (Degenhardt *et al.*, 2002), polymer synthesis (Mallakpour & Kowsari, 2004), supramolecular chemistry (Zhang *et al.*, 1999), and for the development of new materials (Langhals & Kirner, 2000) and molecular electronic devices (Yakimov & Forrest, 2002).

Following our studies on the synthesis of bisimides derivatives, (Li *et al.*, 2007) we report here the structure of the title compound (I), Fig. 1. In the compound, two phthalimide units are symmetrically linked by a bis(3,5-dimethylphenyl)methane bridge. The methylene C atom of this bridge lies on a twofold rotation axis. The dihedral angle between the planes of the two central benzene rings is 61.4 (4)°. The terminal isoindole group is approximately planar with 0.012 Å r.m.s. deviation of atoms from the best fit plane and makes a dihedral angle of 75.3 (14)° with the attached benzene ring. Compared to a similar structure (Li *et al.*, 2007), the packing pattern is different which may result from the methyl groups on the two central benzene rings. This is because the hydrogen atoms of the methyl groups form weak intermolecular C—H···O hydrogen bonds which contribute to the stability of the structure (Table 1).

Experimental

A solution of phthaloyl dichloride (420, 2 mmol) was added slowly over a period of 10 min to a solution of 4-(4-amino-3,5-dimethylbenzyl)-2,6-dimethylbenzenamine (510 mg, 2 mmol) in dichloromethane (25 ml) at 273 K to yield a light yellow precipitate. Triethylamine (5 ml) was then added to dissolve the precipitate which became a yellow suspension after stirring for 12 h. The compound was filtered and dried to give (I), (yield 350 mg, 68%). Single crystals of (I) were obtained by recrystallization from DMF at room temperature.

Refinement

In the absence of significant anomalous dispersion effects, Friedel pairs were merged. All H atoms were initially located in a difference Fourier map and then included with constrained bond lengths and isotropic displacement parameters: C—H = 0.93 Å and $U_{\text{iso}}(\text{H}) = 1.2U_{\text{eq}}(\text{C})$ for aromatic H atoms, C—H = 0.96 Å and $U_{\text{iso}}(\text{H}) = 1.5U_{\text{eq}}(\text{C})$ for methyl H atoms, C—H = 0.97 Å and $U_{\text{iso}}(\text{H}) = 1.2U_{\text{eq}}(\text{C})$ for methylene H atoms.

Figures

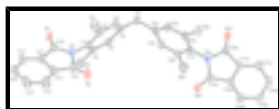


Fig. 1. The molecular structure of (I) with 50% probability displacement ellipsoids (arbitrary spheres for H atoms). Atoms labelled a are related to other atoms by the symmetry operation $x + 1, -y + 1, z$

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Crystal data

$C_{33}H_{26}N_2O_4$	$F_{000} = 540$
$M_r = 514.56$	$D_x = 1.227 \text{ Mg m}^{-3}$
Orthorhombic, $P2_12_12$	Mo $K\alpha$ radiation
Hall symbol: P 2 2ab	$\lambda = 0.71073 \text{ \AA}$
$a = 16.221 (3) \text{ \AA}$	Cell parameters from 1971 reflections
$b = 8.4722 (18) \text{ \AA}$	$\theta = 2.4\text{--}20.7^\circ$
$c = 10.136 (2) \text{ \AA}$	$\mu = 0.08 \text{ mm}^{-1}$
$V = 1392.9 (5) \text{ \AA}^3$	$T = 292 (2) \text{ K}$
$Z = 2$	Block, colourless
	$0.20 \times 0.20 \times 0.10 \text{ mm}$

Data collection

Bruker SMART 4K CCD area-detector diffractometer	1223 reflections with $I > 2\sigma(I)$
Radiation source: fine-focus sealed tube	$R_{\text{int}} = 0.035$
Monochromator: graphite	$\theta_{\text{max}} = 27.0^\circ$
$T = 292(2) \text{ K}$	$\theta_{\text{min}} = 2.0^\circ$
φ and ω scans	$h = -19 \rightarrow 20$
Absorption correction: none	$k = -10 \rightarrow 10$
9315 measured reflections	$l = -12 \rightarrow 10$
1762 independent reflections	

Refinement

Refinement on F^2	H-atom parameters constrained
Least-squares matrix: full	$w = 1/[\sigma^2(F_o^2) + (0.0778P)^2 + 0.0282P]$
$R[F^2 > 2\sigma(F^2)] = 0.049$	where $P = (F_o^2 + 2F_c^2)/3$
$wR(F^2) = 0.135$	$(\Delta/\sigma)_{\text{max}} < 0.001$
$S = 1.04$	$\Delta\rho_{\text{max}} = 0.13 \text{ e \AA}^{-3}$
1762 reflections	$\Delta\rho_{\text{min}} = -0.14 \text{ e \AA}^{-3}$
179 parameters	Extinction correction: none
Primary atom site location: structure-invariant direct methods	
Secondary atom site location: difference Fourier map	
Hydrogen site location: inferred from neighbouring sites	

Special details

Geometry. All e.s.d.'s (except the e.s.d. in the dihedral angle between two l.s. planes) are estimated using the full covariance matrix. The cell e.s.d.'s are taken into account individually in the estimation of e.s.d.'s in distances, angles and torsion angles; correlations between e.s.d.'s in cell parameters are only used when they are defined by crystal symmetry. An approximate (isotropic) treatment of cell e.s.d.'s is used for estimating e.s.d.'s involving l.s. planes.

Refinement. Refinement of F^2 against ALL reflections. The weighted R -factor wR and goodness of fit S are based on F^2 , conventional R -factors R are based on F , with F set to zero for negative F^2 . The threshold expression of $F^2 > 2\sigma(F^2)$ is used only for calculating R -factors(gt) *etc.* and is not relevant to the choice of reflections for refinement. R -factors based on F^2 are statistically about twice as large as those based on F , and R -factors based on ALL data will be even larger.

Fractional atomic coordinates and isotropic or equivalent isotropic displacement parameters (\AA^2)

	<i>x</i>	<i>y</i>	<i>z</i>	$U_{\text{iso}}^*/U_{\text{eq}}$	Occ. (<1)
O1	0.11270 (12)	0.3874 (3)	0.8973 (2)	0.0822 (7)	
C4	0.36944 (16)	0.5785 (4)	0.9058 (3)	0.0676 (8)	
H4	0.3816	0.6821	0.9291	0.081*	
C6	0.29729 (15)	0.5483 (3)	0.8366 (3)	0.0638 (7)	
C7	0.28176 (15)	0.3923 (4)	0.8016 (3)	0.0613 (7)	
C2	0.42398 (14)	0.4605 (4)	0.9415 (3)	0.0654 (8)	
C10	0.12854 (15)	0.3540 (3)	0.7854 (3)	0.0650 (7)	
N1	0.20752 (12)	0.3573 (3)	0.7307 (2)	0.0687 (7)	
C1	0.5000	0.5000	1.0209 (4)	0.0820 (13)	
H1A	0.4874	0.5891	1.0776	0.098*	0.50
H1B	0.5126	0.4109	1.0776	0.098*	0.50
C5	0.33479 (17)	0.2697 (4)	0.8357 (3)	0.0705 (8)	
C3	0.40569 (16)	0.3082 (4)	0.9057 (3)	0.0718 (8)	
H3	0.4420	0.2280	0.9290	0.086*	
C11	0.07252 (16)	0.3054 (4)	0.6778 (3)	0.0690 (8)	
O2	0.26424 (14)	0.3205 (6)	0.5260 (3)	0.1490 (16)	
C17	-0.01132 (17)	0.2809 (4)	0.6786 (3)	0.0855 (10)	
H17	-0.0421	0.2949	0.7552	0.103*	
C13	0.20528 (17)	0.3221 (5)	0.5960 (3)	0.0971 (12)	
C12	0.11783 (17)	0.2860 (5)	0.5650 (3)	0.0870 (10)	
C16	-0.0480 (2)	0.2354 (5)	0.5634 (5)	0.1075 (12)	
H16	-0.1046	0.2184	0.5612	0.129*	
C8	0.3160 (2)	0.1013 (5)	0.8045 (5)	0.1101 (13)	
H8A	0.3557	0.0344	0.8471	0.165*	
H8B	0.3187	0.0856	0.7108	0.165*	
H8C	0.2618	0.0758	0.8356	0.165*	
C9	0.23849 (19)	0.6788 (4)	0.8031 (4)	0.0868 (10)	
H9A	0.2287	0.6794	0.7097	0.130*	
H9B	0.2618	0.7781	0.8293	0.130*	
H9C	0.1874	0.6624	0.8488	0.130*	
C14	0.0814 (2)	0.2415 (7)	0.4501 (4)	0.1268 (17)	
H14	0.1120	0.2298	0.3732	0.152*	
C15	-0.0023 (3)	0.2143 (6)	0.4510 (4)	0.1265 (16)	

supplementary materials

H15 -0.0283 0.1811 0.3741 0.152*

Atomic displacement parameters (\AA^2)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
O1	0.0570 (11)	0.1194 (17)	0.0702 (14)	-0.0088 (11)	0.0105 (10)	-0.0066 (13)
C4	0.0482 (14)	0.0855 (18)	0.0692 (18)	-0.0171 (13)	0.0017 (13)	0.0010 (15)
C6	0.0462 (14)	0.0812 (19)	0.0640 (16)	-0.0096 (13)	0.0021 (13)	0.0086 (14)
C7	0.0359 (12)	0.089 (2)	0.0591 (16)	-0.0094 (12)	0.0029 (11)	0.0000 (14)
C2	0.0359 (12)	0.108 (2)	0.0526 (14)	-0.0085 (14)	0.0054 (11)	0.0045 (16)
C10	0.0437 (13)	0.0829 (18)	0.0685 (18)	-0.0066 (13)	0.0054 (13)	-0.0023 (15)
N1	0.0407 (11)	0.1025 (17)	0.0629 (14)	-0.0096 (12)	0.0018 (10)	-0.0099 (13)
C1	0.0447 (19)	0.145 (4)	0.056 (2)	-0.013 (2)	0.000	0.000
C5	0.0526 (15)	0.083 (2)	0.0753 (19)	-0.0060 (14)	0.0059 (15)	-0.0026 (16)
C3	0.0451 (14)	0.097 (2)	0.0737 (19)	0.0052 (14)	0.0008 (13)	0.0075 (18)
C11	0.0431 (13)	0.0869 (19)	0.0768 (19)	-0.0027 (13)	-0.0025 (14)	-0.0072 (17)
O2	0.0531 (13)	0.310 (5)	0.0842 (17)	-0.017 (2)	0.0157 (13)	-0.054 (2)
C17	0.0418 (15)	0.111 (2)	0.104 (2)	-0.0021 (16)	-0.0051 (17)	0.000 (2)
C13	0.0441 (15)	0.171 (3)	0.076 (2)	-0.007 (2)	0.0060 (15)	-0.032 (2)
C12	0.0462 (15)	0.143 (3)	0.072 (2)	-0.0026 (17)	-0.0051 (15)	-0.017 (2)
C16	0.0493 (17)	0.144 (3)	0.129 (3)	-0.0055 (19)	-0.022 (2)	-0.016 (3)
C8	0.087 (2)	0.092 (2)	0.151 (4)	-0.0020 (19)	-0.008 (3)	-0.019 (3)
C9	0.0641 (18)	0.089 (2)	0.107 (3)	-0.0053 (16)	-0.0040 (18)	0.017 (2)
C14	0.072 (2)	0.223 (5)	0.085 (2)	-0.004 (3)	-0.009 (2)	-0.043 (3)
C15	0.074 (2)	0.194 (4)	0.111 (3)	0.000 (3)	-0.037 (3)	-0.051 (3)

Geometric parameters (\AA , $^\circ$)

O1—C10	1.197 (3)	C11—C12	1.369 (4)
C4—C2	1.383 (4)	C11—C17	1.376 (4)
C4—C6	1.388 (4)	O2—C13	1.191 (4)
C4—H4	0.9300	C17—C16	1.366 (5)
C6—C7	1.392 (4)	C17—H17	0.9300
C6—C9	1.499 (4)	C13—C12	1.485 (4)
C7—C5	1.392 (4)	C12—C14	1.360 (5)
C7—N1	1.434 (3)	C16—C15	1.370 (6)
C2—C3	1.373 (4)	C16—H16	0.9300
C2—C1	1.510 (3)	C8—H8A	0.9600
C10—N1	1.396 (3)	C8—H8B	0.9600
C10—C11	1.478 (4)	C8—H8C	0.9600
N1—C13	1.397 (4)	C9—H9A	0.9600
C1—C2 ⁱ	1.510 (3)	C9—H9B	0.9600
C1—H1A	0.9700	C9—H9C	0.9600
C1—H1B	0.9700	C14—C15	1.377 (6)
C5—C3	1.390 (4)	C14—H14	0.9300
C5—C8	1.492 (5)	C15—H15	0.9300
C3—H3	0.9300		
C2—C4—C6	122.6 (3)	C17—C11—C10	130.2 (3)

C2—C4—H4	118.7	C16—C17—C11	117.9 (3)
C6—C4—H4	118.7	C16—C17—H17	121.1
C4—C6—C7	117.1 (3)	C11—C17—H17	121.1
C4—C6—C9	121.0 (3)	O2—C13—N1	124.3 (3)
C7—C6—C9	121.8 (2)	O2—C13—C12	129.7 (3)
C6—C7—C5	122.2 (2)	N1—C13—C12	106.0 (2)
C6—C7—N1	118.5 (3)	C14—C12—C11	121.0 (3)
C5—C7—N1	119.3 (3)	C14—C12—C13	130.8 (3)
C3—C2—C4	118.1 (2)	C11—C12—C13	108.1 (3)
C3—C2—C1	121.7 (2)	C17—C16—C15	120.8 (3)
C4—C2—C1	120.1 (3)	C17—C16—H16	119.6
O1—C10—N1	124.7 (3)	C15—C16—H16	119.6
O1—C10—C11	129.3 (2)	C5—C8—H8A	109.5
N1—C10—C11	106.0 (2)	C5—C8—H8B	109.5
C10—N1—C13	111.1 (2)	H8A—C8—H8B	109.5
C10—N1—C7	125.1 (2)	C5—C8—H8C	109.5
C13—N1—C7	123.8 (2)	H8A—C8—H8C	109.5
C2 ⁱ —C1—C2	115.6 (3)	H8B—C8—H8C	109.5
C2 ⁱ —C1—H1A	108.4	C6—C9—H9A	109.5
C2—C1—H1A	108.4	C6—C9—H9B	109.5
C2 ⁱ —C1—H1B	108.4	H9A—C9—H9B	109.5
C2—C1—H1B	108.4	C6—C9—H9C	109.5
H1A—C1—H1B	107.4	H9A—C9—H9C	109.5
C3—C5—C7	117.6 (3)	H9B—C9—H9C	109.5
C3—C5—C8	120.1 (3)	C12—C14—C15	118.0 (4)
C7—C5—C8	122.3 (3)	C12—C14—H14	121.0
C2—C3—C5	122.3 (3)	C15—C14—H14	121.0
C2—C3—H3	118.8	C16—C15—C14	121.1 (4)
C5—C3—H3	118.8	C16—C15—H15	119.5
C12—C11—C17	121.2 (3)	C14—C15—H15	119.5
C12—C11—C10	108.6 (2)		
C2—C4—C6—C7	-1.0 (4)	C8—C5—C3—C2	-177.2 (3)
C2—C4—C6—C9	178.2 (3)	O1—C10—C11—C12	177.3 (3)
C4—C6—C7—C5	1.1 (4)	N1—C10—C11—C12	-1.6 (4)
C9—C6—C7—C5	-178.1 (3)	O1—C10—C11—C17	-2.4 (6)
C4—C6—C7—N1	179.7 (2)	N1—C10—C11—C17	178.7 (3)
C9—C6—C7—N1	0.5 (4)	C12—C11—C17—C16	0.5 (5)
C6—C4—C2—C3	0.5 (4)	C10—C11—C17—C16	-179.8 (3)
C6—C4—C2—C1	-177.9 (3)	C10—N1—C13—O2	178.3 (4)
O1—C10—N1—C13	-176.1 (3)	C7—N1—C13—O2	-1.5 (7)
C11—C10—N1—C13	2.8 (4)	C10—N1—C13—C12	-2.9 (4)
O1—C10—N1—C7	3.7 (5)	C7—N1—C13—C12	177.2 (3)
C11—C10—N1—C7	-177.3 (3)	C17—C11—C12—C14	-0.1 (6)
C6—C7—N1—C10	-74.6 (4)	C10—C11—C12—C14	-179.9 (4)
C5—C7—N1—C10	103.9 (3)	C17—C11—C12—C13	179.6 (3)
C6—C7—N1—C13	105.2 (4)	C10—C11—C12—C13	-0.2 (4)
C5—C7—N1—C13	-76.2 (4)	O2—C13—C12—C14	0.1 (8)
C3—C2—C1—C2 ⁱ	91.2 (3)	N1—C13—C12—C14	-178.5 (4)

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C4—C2—C1—C2 ⁱ	-90.4 (3)	O2—C13—C12—C11	-179.5 (5)
C6—C7—C5—C3	-0.8 (4)	N1—C13—C12—C11	1.9 (5)
N1—C7—C5—C3	-179.4 (2)	C11—C17—C16—C15	0.3 (6)
C6—C7—C5—C8	176.7 (3)	C11—C12—C14—C15	-0.9 (7)
N1—C7—C5—C8	-1.9 (5)	C13—C12—C14—C15	179.4 (5)
C4—C2—C3—C5	-0.2 (4)	C17—C16—C15—C14	-1.4 (8)
C1—C2—C3—C5	178.2 (3)	C12—C14—C15—C16	1.7 (8)
C7—C5—C3—C2	0.3 (4)		

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

Hydrogen-bond geometry (\AA , $^\circ$)

$D-H\cdots A$	$D-H$	$H\cdots A$	$D\cdots A$	$D-H\cdots A$
C4—H4 \cdots O1 ⁱⁱ	0.93	2.48	3.304 (4)	148
C16—H16 \cdots O2 ⁱⁱⁱ	0.93	2.33	3.213 (4)	159
C9—H9A \cdots O2 ^{iv}	0.96	2.67	3.545 (4)	151

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

Fig. 1

