N-(3,5-Di-*tert*-butyl-4-hydroxybenzyl)phthalimide

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Key indicators

Single-crystal X-ray study
$T = 294$ K
Mean σ(C–C) = 0.004 Å
R factor = 0.038
wR factor = 0.106
Data-to-parameter ratio = 9.7

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

In the title compound, $C_{23}H_{27}NO_3$, the molecules are held together by intermolecular C—H⋯O hydrogen bonds and π–π stacking interactions.

Comment

3,5-Di-*tert*-butyl-4-hydroxybenzylamine is an important building block in the synthesis of sterically hindered phenols and their derivatives (Silin *et al.*, 1999). Its precursor, N-(3,5-di-*tert*-butyl-4-hydroxybenzyl)phthalimide, (I), was synthesized from phthalimide and benzyl chloride in high yield. The crystal structures of related compounds have been reported by Zeng *et al.* (2005) and Shu *et al.* (2005).

(I)
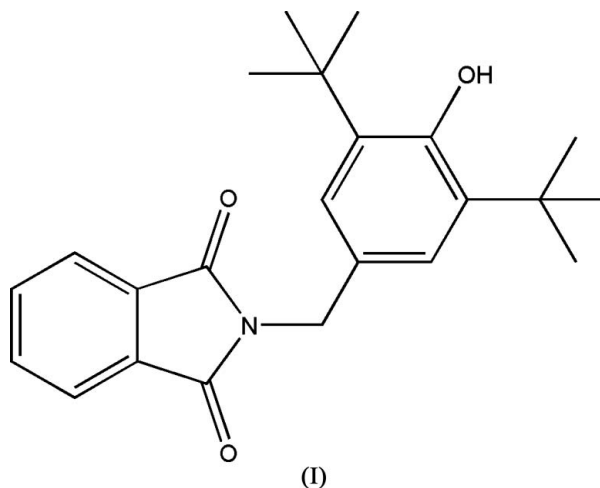
The molecular structure of (I) (Fig. 1) shows that the phthalimide ring system is essentially planar, with a mean deviation of 0.011 (0) Å. The dihedral angle between the N1/C1/C2/C7/C8 and C2–C7 rings is 1.44 (1)°. Selected bond lengths and angles for (I) are given in Table 1.

The molecular structure is stabilized by intramolecular C—H⋯O hydrogen bonds (Table 2). In the crystal structure (Fig. 2), intermolecular C—H⋯O hydrogen bonds are also observed. Strong π–π interactions exist between the aryl rings. The centroid–centroid separations of ring pairs are as follows: five-membered phthalimide rings, 3.9705 (18) ($\frac{1}{2} - y, \frac{1}{2} + x, -\frac{1}{4} + z$) and 3.9708 (18) Å ($-\frac{1}{2} + y, \frac{1}{2} - x, \frac{1}{4} + z$); six- and five-membered phthalimide rings, 3.5750 (17) ($\frac{1}{2} - y, \frac{1}{2} + x, -\frac{1}{4} + z$) and 4.2920 (18) Å ($-\frac{1}{2} + y, \frac{1}{2} - x, \frac{1}{4} + z$), respectively; six-membered phthalimide rings 3.9257 (17) Å ($\frac{1}{2} - y, \frac{1}{2} + x, -\frac{1}{4} + z$) and ($-\frac{1}{2} + y, \frac{1}{2} - x, \frac{1}{4} + z$); phenol rings 4.5885 (17) Å ($y, x, 1 - z$).

Experimental

The title compound was obtained by the reaction of phthalimide (7.36 g, 0.05 mol) with 3,5-di-*tert*-butyl-4-hydroxybenzyl chloride (12.7 g, 0.05 mol) in the presence of potassium carbonate (6.90 g, aqueous) in dimethylformamide (50 ml) for 2 h at ambient temperature. It was recrystallized from ethanol as a colourless solid (yield 18.15g, 99%, m.p. 439–440 K).

Crystal data

$C_{23}H_{27}NO_3$	Mo $K\alpha$ radiation
$M_r = 365.46$	Cell parameters from 5012
Tetragonal, $P4_32_12$	reflections
$a = 17.1925$ (16) Å	$\theta = 2.2–24.4°$
$c = 13.7715$ (19) Å	$\mu = 0.08$ mm^{-1}
$V = 4070.6$ (8) Å^3	$T = 294$ (2) K
$Z = 8$	Block, colourless
$D_x = 1.193$ Mg m^{-3}	$0.26 \times 0.22 \times 0.20$ mm

Data collection

Bruker SMART CCD area-detector diffractometer	2428 independent reflections
	1640 reflections with $I > 2\sigma(I)$
φ and ω scans	$R_{int} = 0.057$
Absorption correction: multi-scan (*SADABS*; Sheldrick, 1996)	$\theta_{max} = 26.5°$
	$h = -21 \rightarrow 17$
$T_{min} = 0.971$, $T_{max} = 0.985$	$k = -17 \rightarrow 21$
23105 measured reflections	$l = -17 \rightarrow 16$

Refinement

Refinement on F^2	$w = 1/[\sigma^2(F_o^2) + (0.0391P)^2$
$R[F^2 > 2\sigma(F^2)] = 0.038$	$+ 1.0321P]$
$wR(F^2) = 0.106$	where $P = (F_o^2 + 2F_c^2)/3$
$S = 1.06$	$(\Delta/\sigma)_{max} < 0.001$
2428 reflections	$\Delta\rho_{max} = 0.14$ e Å^{-3}
251 parameters	$\Delta\rho_{min} = -0.18$ e Å^{-3}
H-atom parameters constrained	Extinction correction: *SHELXL97*
	Extinction coefficient: 0.0033 (5)

Table 1
Selected geometric parameters (Å, °).

O1—C1	1.206 (3)	N1—C1	1.393 (4)
O2—C8	1.203 (3)	N1—C8	1.403 (3)
O3—C13	1.387 (3)	N1—C9	1.461 (4)
C1—N1—C8	111.4 (2)	O1—C1—N1	125.3 (3)
C1—N1—C9	124.2 (3)	O1—C1—C2	128.5 (3)
C8—N1—C9	123.9 (2)	N1—C1—C2	106.2 (2)
C8—N1—C1—O1	177.8 (3)	C1—N1—C8—O2	−178.6 (3)
C9—N1—C1—O1	4.9 (5)	C9—N1—C8—O2	−5.7 (5)

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

D—H$\cdots A$	D—H	H$\cdots A$	$D\cdots A$	D—H$\cdots A$
O3—H3$\cdots$O1^i	0.88	2.51	3.364 (3)	164
C9—H9$B$$\cdots$O1	0.97	2.57	2.921 (4)	101
C15—H15$\cdots$O2	0.93	2.54	3.219 (4)	130
C17—H17$A$$\cdots$O2^{ii}	0.96	2.57	3.490 (4)	161
C22—H22$A$$\cdots$O1^i	0.96	2.55	3.223 (4)	127

Symmetry codes: (i) $x + \frac{1}{2}, -y + \frac{3}{2}, -z + \frac{1}{4}$; (ii) $y - \frac{1}{2}, -x + \frac{3}{2}, z + \frac{1}{4}$.
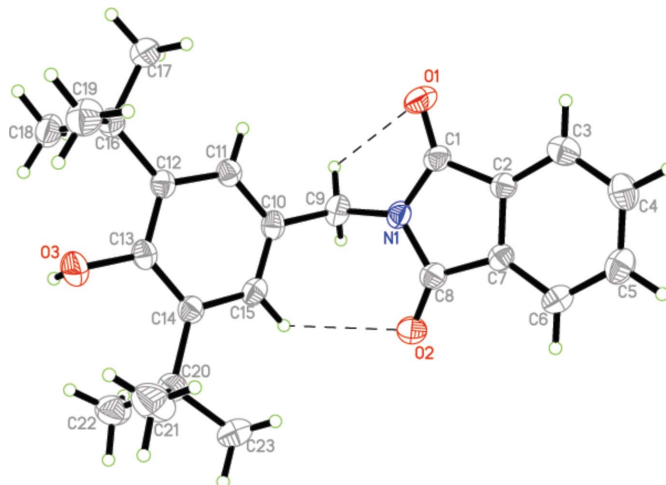
Figure 1
View of the molecular structure of (I), showing the atom-labelling scheme. Displacement ellipsoids are drawn at the 30% probability level and H atoms are shown as small spheres of arbitrary radii. Dashed lines indicate intramolecular hydrogen bonds.
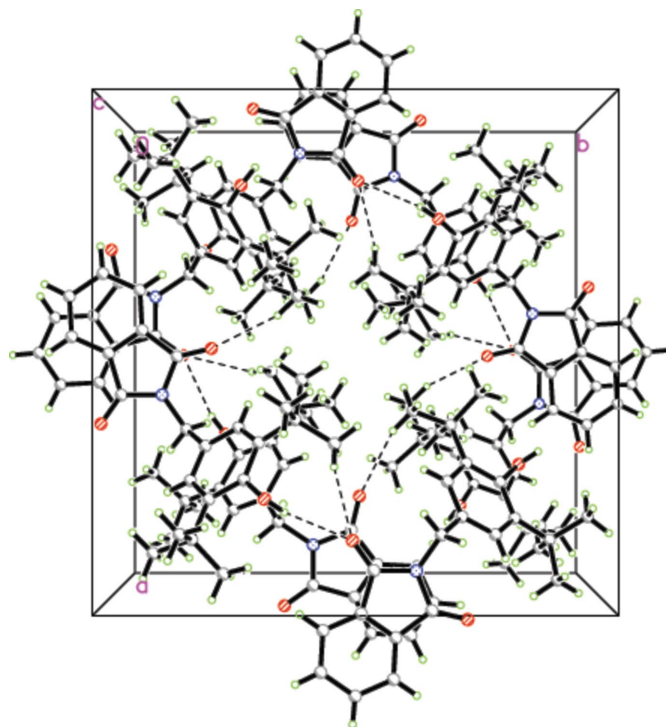
Figure 2
A packing diagram for (I). The molecules are held together by π–π stacking interactions. Dashed lines indicate intermolecular hydrogen bonds.

H atoms were positioned geometrically and constrained to ride on their parent atoms [C—H = 0.93 Å for Csp^2—H and 0.97 Å for CH$_2$ groups, with U_{iso}(H) = 1.2U_{eq}(C); C—H = 0.96 Å for methyl groups and 0.88 Å for O—H, with U_{iso}(H) = 1.5U_{eq}(C,O)]. In the absence of significant anomalous dispersion effects, Friedel pairs were averaged.

Data collection: *SMART* (Bruker, 1997); cell refinement: *SAINT* (Bruker, 1997); data reduction: *SAINT*; 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*.

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