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N,*N*-Bis(3,5-di-*tert*-butyl-4-hydroxybenzyl)-*N*-propylamine

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Key indicators

Single-crystal X-ray study
T = 294 K
Mean σ(C–C) = 0.007 Å
R factor = 0.054
wR factor = 0.164
Data-to-parameter ratio = 11.1

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

In the title compound, $C_{33}H_{53}NO_2$, the phenolic hydroxyl groups are sterically hindered by the adjacent tert-butyl groups.

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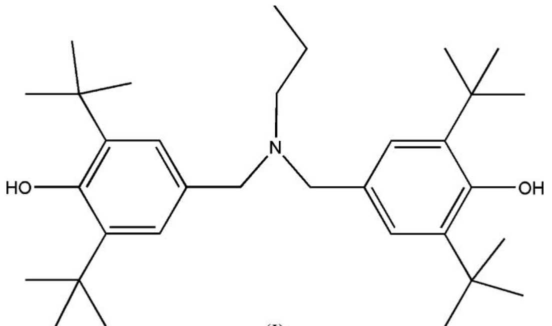
Comment

Sterically hindered phenol anti-oxidants are widely used in polymers and lubricants. They can protect polymers by increasing both their process stability and their long-term stability against oxidative degradation (Yamazaki & Seguchi, 1997). The title compound, $C_{33}H_{53}NO_2$, (I), was synthesized and its structure is reported here. Selected bond lengths and angles are listed in Table 1. The phenolic hydroxyl groups are sterically hindered by the adjacent *tert*-butyl groups.



(I)

Experimental

4-Bromomethyl-2,6-di-*tert*-butyl-phenol was synthesized according to the method described by Rieker *et al.* (1968). Propylamine (2.0 g,

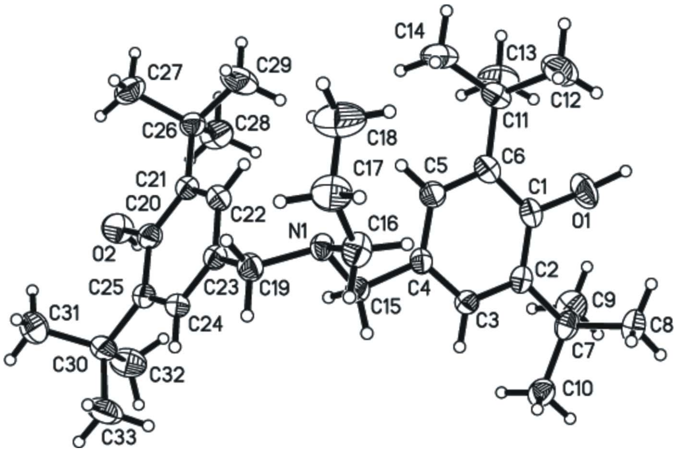


Figure 1
A view of the molecular structure of (I). Displacement ellipsoids are drawn at the 30% probability level and H atoms are shown as small spheres of arbitrary radii.

0.034 mol) and 4-bromomethyl-2,6-di-*tert*-butyl-phenol (9.0 g, 0.03 mol) were added, with stirring to THF (40 ml) at 273 K. The reaction mixture was stirred at 273–278 K for a further 3 h; a solution of Na_2CO_3 (1.60 g, 0.015 mol) in water (10 ml) was then added. The solvent THF was evaporated under reduced pressure and the product was extracted with diethyl ether. The product (6.93 g) was obtained in a yield of 93.2%. Suitable crystals (m.p. 418–420 K) were obtained by slow evaporation of a solution in dichloromethane and ethanol. ^{1}H NMR ($CDCl_3$, δ, p.p.m.): 0.91 (*t*, 3H), 1.43 (*s*, 36H), 1.54 (*m*, 2H), 2.35 (*t*, 2H), 3.44 (*s*, 4H), 5.04 (*s*, 2H), 7.16(*s*, 4H) ^{13}C NMR ($CDCl_3$, δ, p.p.m.): 152.6 (2C), 135.6 (4C), 131.1 (2C), 125.3 (4C), 58.6 (2C), 55.5 (1C), 34.5 (4C), 30.7 (12C), 20.6 (1C), 12.2 (1C).

Crystal data

$C_{33}H_{53}NO_2$
$M_r = 495.76$
Tetragonal, $P4_3$
$a = 15.822$ (4) Å
$c = 12.844$ (4) Å
$V = 3215.4$ (15) Å^3
$Z = 4$
$D_x = 1.024$ Mg m^{-3}
Mo $K\alpha$ radiation
Cell parameters from 3052 reflections
$\theta = 2.4$–19.6°
$\mu = 0.06$ mm^{-1}
$T = 294$ (2) K
Block, colourless
0.24 × 0.22 × 0.18 mm

Data collection

Bruker SMART CCD area-detector diffractometer
φ and ω scans
Absorption correction: multi-scan (*SADABS*; Sheldrick, 1996)
$T_{min} = 0.972$, $T_{max} = 0.989$
18244 measured reflections
3601 independent reflections
1888 reflections with $I > 2\sigma(I)$
$R_{int} = 0.128$
$\theta_{max} = 27.0°$
$h = -19 \rightarrow 18$
$k = -20 \rightarrow 15$
$l = -16 \rightarrow 15$

Refinement

Refinement on F^2
$R[F^2 > 2\sigma(F^2)] = 0.054$
$wR(F^2) = 0.164$
$S = 1.02$
3601 reflections
325 parameters
H-atom parameters constrained
$w = 1/[\sigma^2(F_o^2) + (0.0563P)^2 + 0.9914P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} < 0.001$
$\Delta\rho_{max} = 0.21$ e Å^{-3}
$\Delta\rho_{min} = -0.15$ e Å^{-3}

Table 1
Selected geometric parameters (Å, °).

N1—C19	1.465 (5)	C4—C15	1.516 (6)
N1—C15	1.466 (5)	C6—C11	1.549 (7)
O1—C1	1.381 (5)	C11—C12	1.547 (7)
C1—C6	1.404 (7)	C17—C18	1.474 (10)
C19—N1—C15	110.2 (3)	C2—C3—H3*A*	118.7
C19—N1—C16	111.4 (3)	C4—C15—H15*A*	108.7
C15—N1—C16	110.0 (3)	N1—C15—H15*B*	108.7
O1—C1—C6	119.8 (5)	C18—C17—C16	113.0 (5)
C1—C2—C7	121.9 (4)		
O1—C1—C2—C3	177.7 (4)	C3—C2—C7—C10	5.9 (6)
O1—C1—C2—C7	−2.8 (6)	N1—C16—C17—C18	−57.6 (8)
C7—C2—C3—C4	−178.1 (4)		

O-bound H-atoms were located in a difference Fourier map and refined using a riding model, with $U_{iso}(H) = 1.2U_{eq}(O)$ and O—H = 0.98 and 1.08 Å. All other H atoms were positioned geometrically (C—H = 0.96–0.97 Å) and refined using a riding model, with $U_{iso}(H) = 1.2U_{eq}(C)$. Refinement was attempted in space groups $P4_1$ and $P4_3$ and identical results were obtained with respect to the *R* factors, goodness-of-fit and convergence. However, in the absence of significant anomalous dispersion atoms, the space group assignment cannot be determined simply from the X-ray diffraction data, and thus space group $P4_3$ was chosen arbitrarily. In the absence of significant anomalous scattering effects, Friedel pairs were merged.

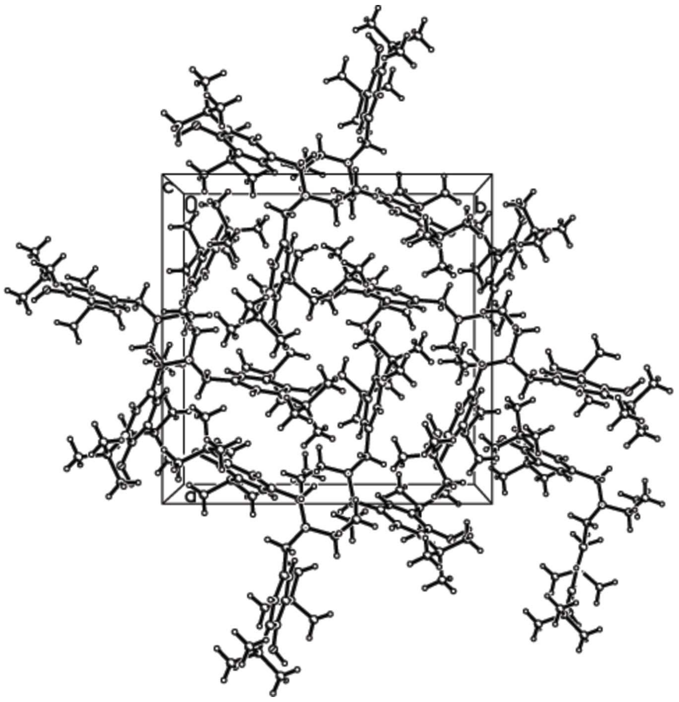


Figure 2
The packing of (I).

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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