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2,6-Di-*tert*-butyl-4-(isopropylaminomethyl)phenol

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Key indicators

Single-crystal X-ray study
T = 294 K
Mean σ(C–C) = 0.005 Å
R factor = 0.056
wR factor = 0.166
Data-to-parameter ratio = 16.6

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

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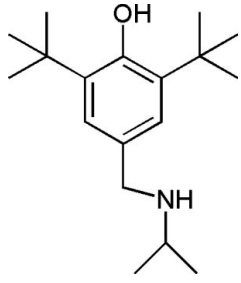
The title compound, $C_{18}H_{31}NO$, was prepared from 3,5-di-*tert*-butyl-4-hydroxybenzaldehyde through an indirect reductive amination process. There are two molecules in the asymmetric unit which are linked by an O—H···N hydrogen bond [O···N = 2.784 (4) Å]. This molecule pair is, in turn, linked into a four-molecule centrosymmetric cluster by a second intermolecular O—H···N hydrogen bond [O···N = 2.852 (3) Å].

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). In our research, we aim to synthesize secondary benzyl amine derivatives with a sterically hindered phenol group, as intermediates. Initially, we tried to prepare this type of intermediate through amination of the hindered phenol benzyl halide using a primary amine, but, as we have reported previously, the main product was the tertiary amine, even when excess primary amine was used (Zeng *et al.*, 2005). However, when we attempted an indirect reductive amination synthetic procedure, the desired title compound, (I), was produced.



(I)

Selected bond lengths and angles for (I) are shown in Table 1. There are two independent molecules in the asymmetric unit, in which the methylisopropylamine substituents have different conformations. These differences are described by the torsion angles C16—N1—C15—C4 and C34—N2—C33—C22 (Table 1). Both types of molecules are linked into centrosymmetric clusters *via* intermolecular O—H···N hydrogen bonds (Table 2 and Fig. 2)

Experimental

3,5-Di-*tert*-butyl-4-hydroxybenzaldehyde (11.71 g, 0.05 mol) and isopropylamine (2.95 g, 0.05 mol) were mixed in toluene (150 ml) with stirring at 303 K for 0.5 h and then heated to reflux for 4 h. The toluene was removed by vacuum evaporation and the product was

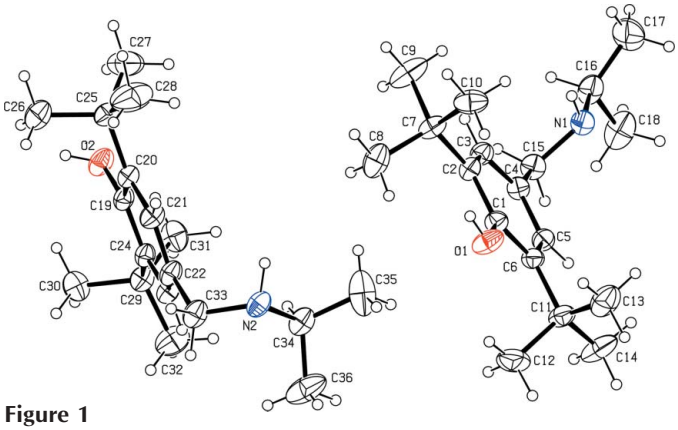


Figure 1
A view of the asymmetric unit of (I), with displacement ellipsoids drawn at the 30% probability level and H atoms shown as small spheres of arbitrary radii.

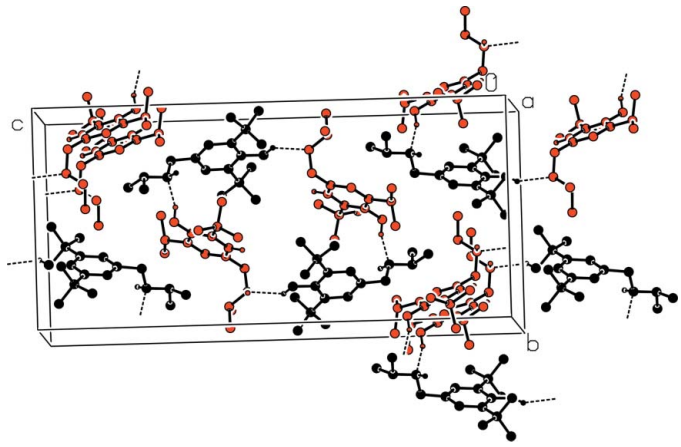


Figure 2
A packing diagram (*PLATON*; Spek, 2003) for (I). Dashed lines indicate the hydrogen bonds. Red and black colouring distinguishes the two different independent molecules in the crystal structure.

dried. The imine 2,6-di-*tert*-butyl-4-[(*E*)-butyliminomethyl]phenol (13.75 g) was obtained. This imine was pure enough for further reaction. The imine (5.5 g) was added to dry ethanol (50 ml) in an ice bath, and $NaBH_4$ (0.026 mol, 1.0 g) was added at 268 K and stirred for 0.5 h. The mixture was treated with 10% hydrochloric acid until pH 2 was reached. The ethanol was removed by vacuum evaporation at 318 K and then 20% Na_2CO_3 was added until the pH was 10. The product was extracted with diethyl ether and separated by column chromatography (ethyl acetate–petroleum ether, 1:10 *v*/*v*). The title compound (3.13 g) was obtained in a yield of 56.5%. Suitable crystals (m.p. 325–326 K) were obtained from a solution of (I) in petroleum ether in a vacuum station at a temperature of approximately 278 K. Spectroscopic analysis: ^{1}H NMR ($CDCl_3$, δ, p.p.m.): 1.11 (*s*, 3H), 1.13 (*s*, 3H), 1.45 (*s*, 18H), 6.09 (*s*, 1H), 2.89 (*m*, 1H), 3.69 (*s*, 2H), 5.12 (*s*, 1H) 7.11 (*s*, 2H); ^{13}C NMR ($CDCl_3$, δ, p.p.m.): 152.9 (1C), 136.0 (2C), 131.0 (1C), 125.1 (2C), 52.3 (1C), 48.7 (1C), 34.5 (2C), 30.6 (6C), 23.1 (2C), 12.2 (1C).

Crystal data

$C_{18}H_{31}NO$
M_r = 277.44
Monoclinic, $P2_1/n$
a = 9.5296 (17) Å
b = 13.411 (3) Å
c = 28.434 (5) Å
β = 94.443 (4)°
V = 3623.0 (12) Å^3
Z = 8
D_x = 1.017 Mg m^{-3}
Mo $K\alpha$ radiation
Cell parameters from 2027 reflections
θ = 2.6–20.3°
μ = 0.06 mm^{-1}
T = 294 (2) K
Block, colourless
0.24 × 0.16 × 0.14 mm

Data collection

Bruker SMART CCD area-detector diffractometer
φ and ω scans
Absorption correction: multi-scan (*SADABS*; Sheldrick, 1996)
T_{min} = 0.970, T_{max} = 0.991
18150 measured reflections
6384 independent reflections
2553 reflections with $I > 2\sigma(I)$
R_{int} = 0.096
θ_{max} = 25.0°
$h = -6 \rightarrow 11$
$k = -15 \rightarrow 15$
$l = -33 \rightarrow 33$

Refinement

Refinement on F^2
$R[F^2 > 2\sigma(F^2)]$ = 0.056
$wR(F^2)$ = 0.166
S = 0.99
6384 reflections
385 parameters
H atoms treated by a mixture of independent and constrained refinement
$w = 1/[\sigma^2(F_o^2) + (0.0599P)^2 + 0.2582P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max}$ = 0.001
$\Delta\rho_{max}$ = 0.16 e Å^{-3}
$\Delta\rho_{min}$ = −0.16 e Å^{-3}

Table 1
Selected geometric parameters (Å, °).

N1—C15	1.479 (4)	N2—C34	1.476 (4)
N1—C16	1.489 (4)	O1—C1	1.374 (3)
N2—C33	1.470 (4)	O2—C19	1.391 (3)
C15—N1—C16	113.6 (3)	C33—N2—C34	114.3 (3)
O1—C1—C2—C3	−179.5 (3)	O2—C19—C20—C21	−179.4 (3)
C7—C2—C3—C4	176.5 (3)	C25—C20—C21—C22	177.5 (3)
C15—C4—C5—C6	176.5 (3)	C22—C23—C24—C19	−0.9 (5)
C16—N1—C15—C4	−167.2 (3)	C34—N2—C33—C22	−68.9 (4)
C15—N1—C16—C17	163.6 (3)	C33—N2—C34—C35	163.5 (3)

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

D—H···A	D—H	H···A	D···A	D—H···A
O2—H2···N1^{i}	0.82	2.06	2.784 (4)	148
O1—H1···N2^{ii}	0.82	2.34	2.852 (3)	121

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

H atoms bonded to N atoms were refined independently, with U_{iso}(H) = 1.2U_{eq}(N). All other H atoms were placed in calculated positions, with C—H distances in the range 0.93–0.98 Å and O—H = 0.82 Å. They were included in the refinement with U_{iso}(H) = 1.2U_{eq}(C) or 1.5U_{eq}(C_{methyl} and O).

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: *PLATON* (Spek, 2003); software used to prepare material for publication: *SHELXTL*.

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