# organic papers

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Key indicators

Single-crystal X-ray study
$T = 294$ K
Mean σ(C–C) = 0.003 Å
R factor = 0.045
wR factor = 0.135
Data-to-parameter ratio = 17.6

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

2,6-Di-tert-butyl-4-[(4-methoxyphenylimino)methyl]phenol

The title compound, $C_{22}H_{29}NO_2$, was prepared from 3,5-di-tert-butyl-4-hydroxybenzaldehyde and 4-methoxyaniline by an indirect reductive amination process. Intermolecular O—H···N hydrogen bonds seem to be effective in the stabilization of the crystal structure.

Comment

Hindered phenol anti-oxidants are widely used in polymers and lubricants (Yamazaki & Seguchi, 1997). As we have reported previously, we have tried to synthesize a series of secondary benzylamine derivatives with a hindered phenol group, as intermediates (Shu *et al.*, 2005).

However, when we attempted an indirect reductive amination synthetic procedure, the title compound, (I), was produced from 3,5-di-tert-butyl-4-hydroxybenzaldehyde and 4-methoxyaniline.

(I)
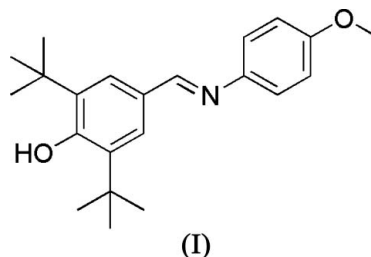
The bond lengths and angles (Table 1) are in normal ranges (Allen *et al.*, 1987). In the crystal structure, the molecules are linked by intermolecular O—H···N hydrogen bonds (Table 2), forming a one-dimensional chain parallel to the *b* axis (Fig. 2).

Experimental

A mixture of 3,5-di-tert-butyl-4-hydroxybenzaldehyde (11.7 g, 0.05 mol) and 4-methoxyaniline (6.15 g, 0.05 mol) was stirred in toluene (100 ml), affording (I) (yield 16.8 g, 99.5%; m.p. 388–389 K). Suitable crystals were obtained by slow evaporation of a solution in a mixture of dichloromethane/ethanol (1:1).

Crystal data

$C_{22}H_{29}NO_2$	Mo $K\alpha$ radiation
$M_r = 339.46$	Cell parameters from 3392
Orthorhombic, *Pbca*	reflections
$a = 12.281$ (3) Å	$\theta = 2.4$–24.0°
$b = 13.945$ (3) Å	$\mu = 0.07$ mm^{-1}
$c = 23.527$ (5) Å	$T = 294$ (2) K
$V = 4029.3$ (15) Å^3	Block, colorless
$Z = 8$	$0.26 \times 0.22 \times 0.20$ mm
$D_x = 1.119$ Mg m^{-3}	

Data collection

Bruker SMART CCD area-detector diffractometer	4142 independent reflections
	2131 reflections with $I > 2\sigma(I)$
φ and ω scans	$R_{int} = 0.067$
Absorption correction: multi-scan (*SADABS*; Sheldrick, 1996)	$\theta_{max} = 26.5°$
	$h = -7 \rightarrow 15$
$T_{min} = 0.982$, $T_{max} = 0.986$	$k = -17 \rightarrow 17$
21536 measured reflections	$l = -29 \rightarrow 29$

Refinement

Refinement on F^2	$w = 1/[\sigma^2(F_o^2) + (0.0513P)^2 + 0.9192P]$
$R[F^2 > 2\sigma(F^2)] = 0.045$	
$wR(F^2) = 0.135$	where $P = (F_o^2 + 2F_c^2)/3$
$S = 1.00$	$(\Delta/\sigma)_{max} = 0.001$
4142 reflections	$\Delta\rho_{max} = 0.15$ e Å^{-3}
235 parameters	$\Delta\rho_{min} = -0.17$ e Å^{-3}
H-atom parameters constrained	Extinction correction: *SHELXL97*
	Extinction coefficient: 0.0022 (4)

Figure 1
A drawing of the title molecule with the atom-numbering scheme. Displacement ellipsoids are drawn at the 30% probability level.

Table 1
Selected geometric parameters (Å, °).

O1—C4	1.380 (2)	N1—C15	1.273 (2)
O2—C19	1.370 (2)	N1—C16	1.427 (2)
O2—C22	1.402 (3)		
C15—N1—C16	118.21 (18)	N1—C15—C1	125.5 (2)
O1—C4—C5	118.94 (17)	O2—C19—C18	125.3 (2)
O1—C4—C3	118.40 (18)	O2—C19—C20	115.4 (2)
C16—N1—C15—C1	175.77 (19)	C15—N1—C16—C17	−43.6 (3)
C2—C1—C15—N1	−7.6 (3)	C15—N1—C16—C21	140.1 (2)
C6—C1—C15—N1	175.7 (2)		

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

D—H$\cdots A$	D—H	H$\cdots A$	$D\cdots A$	D—H$\cdots A$
O1—H1$\cdots$N1^i	0.82	2.08	2.897 (2)	172

Symmetry code: (i) $-x + \frac{1}{2}, y - \frac{1}{2}, z$.

H atoms were positioned geometrically (O—H = 0.82 Å, and C—H = 0.93 Å for aromatic and methine H atoms and 0.96 Å for methyl H atoms) and constrained to ride on their parent atoms, with $U_{iso}(H) = xU_{eq}(C,O)$, where $x = 1.2$ for aromatic and methine H atoms and $x = 1.5$ for all other H atoms.

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*.
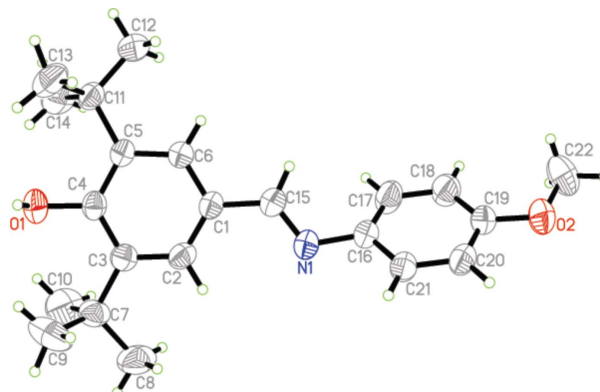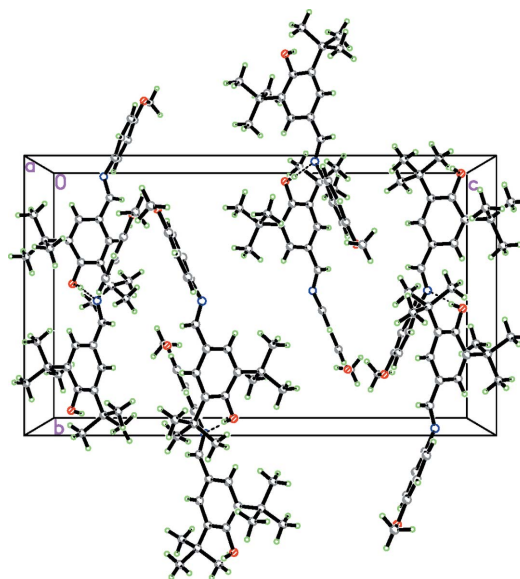

Figure 2
A packing diagram of (I). Hydrogen bonds are shown as dashed lines.

References

Allen, F. H., Kennard, O., Watson, D. G., Brammer, L., Orpen, A. G. & Taylor, R. (1987). *J. Chem. Soc. Perkin Trans 2*, pp. S1–19.
Bruker (1997). *SMART*, *SAINT* and *SHELXTL*. Bruker AXS Inc., Madison, Wisconsin, USA.
Sheldrick, G. M. (1996). *SADABS*. University of Göttingen, Germany.
Sheldrick, G. M. (1997). *SHELXS97* and *SHELXL97*. University of Göttingen, Germany.
Shu, X.-G., Zeng, T., Chen, L.-G., Yan, F.-Y. & Zhang, Y.-C. (2005). *Acta Cryst.* E**61**, o4192–o4194.
Yamazaki, T. & Seguchi, T. (1997). *J. Polym. Sci. Part A Polym. Chem.* **35**, 2431–2439.