

2,6-Di-*tert*-butyl-4-(phenyliminomethyl)phenolXi-Long Yan, Li-Gong Chen and
Tao Zeng*School of Chemical Engineering and
Technology, Tianjin University, Tianjin 300072,
People's Republic of China

Correspondence e-mail: zengtaotj@126.com

Key indicators

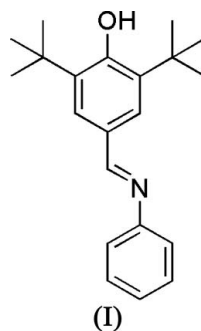
Single-crystal X-ray study
 $T = 294$ K
Mean $\sigma(\text{C}-\text{C}) = 0.003$ Å
 R factor = 0.051
 wR factor = 0.154
Data-to-parameter ratio = 21.1For details of how these key indicators were
automatically derived from the article, see
<http://journals.iucr.org/e>.

In the title molecular structure, $\text{C}_{21}\text{H}_{27}\text{NO}$, the dihedral angle between the phenyl ring and the benzene ring planes is $63.28(9)^\circ$. It appears that the hydroxy group is prevented from forming a hydrogen bond because of steric hindrance by the *tert*-butyl groups.

Received 23 February 2006
Accepted 10 March 2006

Comment

Hindered phenol antioxidants are widely used in polymers and lubricants (Yamazaki & Seguchi, 1997). As reported previously (Shu *et al.*, 2005), we have synthesized a series of secondary benzylamines with hindered phenol groups *via* an indirect reductive amination procedure. Thus, a series of imines with hindered phenol groups has been prepared as intermediates. We synthesized (I) from 3,5-di-*tert*-butyl-4-hydroxybenzaldehyde and aniline.



Selected bond lengths and angles are given in Table 1. The hydroxy group is not involved in hydrogen bonding; the closest intermolecular distance between O1 and the most likely acceptor atom N1ⁱ [symmetry code: (i) $x, \frac{1}{2} - y, \frac{1}{2} + z$] is $3.582(2)$ Å.

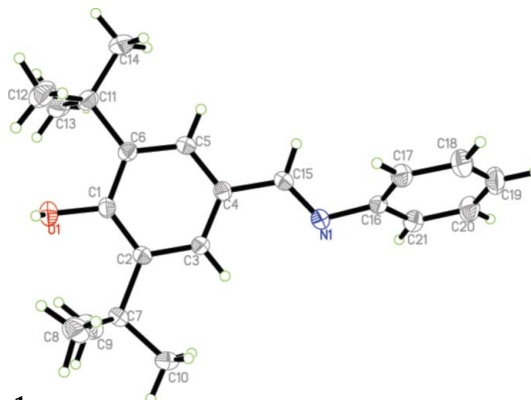


Figure 1

The molecular structure of (I), with displacement ellipsoids drawn at the 30% probability level and H atoms as small spheres of arbitrary radii.

Experimental

A mixture of 3,5-di-*tert*-butyl-4-hydroxybenzaldehyde (11.7 g, 0.05 mol) and aniline (15.4 g, 0.05 mol) was stirred in refluxing toluene (100 ml) and the imine (15.4 g) was obtained in nearly quantitative yield (99.5%). Suitable crystals (m.p. 372–373 K) were obtained by slow evaporation of a solution of (I) in dichloromethane and ethanol (5:1 v/v).

Crystal data

C₂₁H₂₇NO
M_r = 309.44
 Monoclinic, *P*₂₁/*c*
a = 14.592 (2) Å
b = 10.9764 (17) Å
c = 11.925 (2) Å
 β = 101.209 (3)°
V = 1873.7 (5) Å³
Z = 4
D_x = 1.097 Mg m⁻³
 Mo *K*α radiation
 Cell parameters from 3107 reflections
 θ = 2.3–27.2°
 μ = 0.07 mm⁻¹
T = 294 (2) K
 Block, colourless
 0.24 × 0.20 × 0.16 mm

Data collection

Bruker SMART CCD diffractometer
 φ and ω scans
 Absorption correction: multi-scan (*SADABS*; Sheldrick, 1996)
T_{min} = 0.984, *T_{max}* = 0.990
 11349 measured reflections
 4521 independent reflections
 2586 reflections with *I* > 2σ(*I*)
R_{int} = 0.033
 θ_{max} = 28.3°
h = -17 → 19
k = -12 → 14
l = -12 → 15

Refinement

Refinement on *F*²
R [*F*² > 2σ(*F*²)] = 0.051
wR (*F*²) = 0.154
S = 1.03
 4521 reflections
 214 parameters
 H-atom parameters constrained
 $w = 1/[\sigma^2(F_o^2) + (0.0673P)^2 + 0.3616P]$
 where $P = (F_o^2 + 2F_c^2)/3$
 $(\Delta/\sigma)_{\text{max}} < 0.001$
 $\Delta\rho_{\text{max}} = 0.35 \text{ e } \text{Å}^{-3}$
 $\Delta\rho_{\text{min}} = -0.36 \text{ e } \text{Å}^{-3}$

Table 1

Selected geometric parameters (Å, °).

N1–C15	1.267 (2)	C4–C15	1.463 (2)
N1–C16	1.420 (2)	C6–C11	1.541 (2)
C15–N1–C16	118.21 (14)	N1–C15–C4	124.10 (15)
O1–C1–C6	116.24 (14)	N1–C15–H15	117.9
C14–C11–C6	111.50 (15)		
O1–C1–C2–C3	177.95 (15)	C5–C4–C15–N1	-173.62 (17)
C7–C2–C3–C4	-177.59 (15)	N1–C16–C21–C20	-178.35 (17)
C16–N1–C15–C4	-172.48 (15)		

The H atom of the hydroxy group was located in a difference Fourier map and was refined as riding with O–H = 0.85 Å, and *U_{iso}*(H) = 1.2*U_{eq}*(O). All other H atoms were positioned geometrically, with C–H = 0.93–0.96 Å, and refined using a riding-model approximation, with *U_{iso}*(H) = 1.2*U_{eq}*(C) or 1.5*U_{eq}*(methyl C).

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

References

Bruker (1997). *SMART*, *SAINTE* 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. A Polym. Chem.* **35**, 2431–2439.