

1,4-Bis(3,5-di-*tert*-butyl-2-hydroxybenzylidene-aminomethyl)benzeneDuncan M. Tooke,^{a*} Yufei Song,^b
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Key indicators

Single-crystal X-ray study

T = 150 K

Mean $\sigma(\text{C}-\text{C}) = 0.002 \text{ \AA}$

R factor = 0.047

wR factor = 0.115

Data-to-parameter ratio = 19.0

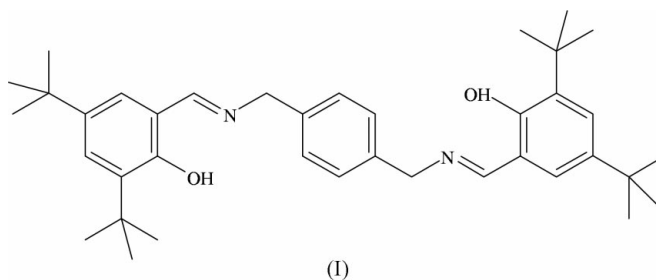
For details of how these key indicators were
automatically derived from the article, see
<http://journals.iucr.org/e>.The title compound, $\text{C}_{38}\text{H}_{52}\text{N}_2\text{O}_2$, has been obtained by a Schiff base reaction of 3,5-di-*tert*-butylsalicylaldehyde and *p*-xylenediamine in methanol. The molecule is located on a crystallographic inversion centre, and the hydrogen bonding is exclusively intramolecular.

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Comment

In the course of research into ligands that are suitable for use in supramolecular chemistry, we have synthesized a number of imine-base ligand systems through which a variety of supramolecular architectures can be assembled (Song *et al.*, 2004). The ease of synthesis of these ligand systems has allowed us to probe systematically the effects of modifications to the ligand backbone, by which it is possible to control the topology or micro-architecture of the arrays. In the present study, bis(3,5-di-*tert*-butyl-2-hydroxybenzyl)-*p*-xylenediamine (H_2L), (I), has been obtained by a Schiff base reaction.The molecule crystallizes in the monoclinic space group $P2_1/c$, with the two halves of the molecule related by a centre of symmetry. The $\text{O}-\text{H} \cdots \text{N}$ hydrogen bonding is exclusively intramolecular.

Experimental

Bis(3,5-di-*tert*-butyl-2-hydroxybenzyl)-*p*-xylenediamine was synthesized in 88% yield by mixing two equivalents of 3,5-di-*tert*-butylsalicylaldehyde and one equivalent of *p*-xylenediamine in methanol. Recrystallization of the compound from chloroform led to the formation of yellow crystals suitable for X-ray measurement. Yield 88%. Elemental analysis calculated for $\text{C}_{38}\text{H}_{52}\text{N}_2\text{O}_2$: C 80.24, H 9.21, N 4.92%; found C 80.56, H 9.23, N 4.89%. IR (solid, cm^{-1}): 2950.2 (s), 1630.4 (s), 1470.6 (s), 1359.1 (s), 1274.1 (m), 1249.7 (s), 1205.1 (m), 1170.9 (s), 870.5 (s), 795.7 (s), 771.0 (s), 731.6 (m), 698.1 (s), 477.8 (m). ^1H NMR (300 MHz, p.p.m.): 1.29 (18H, *tert*-butyl), 1.42 (18H, *tert*-butyl), 4.76 (4H, $-\text{CH}_2$), 7.08 (2H, phenol ring), 7.30 (4H, benzene rings), 7.36 (2H, phenol rings), 8.43 (2H, benzylideneimine).

Crystal data

$C_{38}H_{52}N_2O_2$
 $M_r = 568.82$
 Monoclinic, $P2_1/c$
 $a = 15.8792$ (18) Å
 $b = 10.4601$ (11) Å
 $c = 10.4395$ (15) Å
 $\beta = 106.856$ (10)°
 $V = 1659.5$ (4) Å³
 $Z = 2$

$D_x = 1.138$ Mg m⁻³
 Mo $K\alpha$ radiation
 Cell parameters from 46 reflections
 $\theta = 5.5$ – 20.5 °
 $\mu = 0.07$ mm⁻¹
 $T = 150$ K
 Block, yellow
 $0.36 \times 0.25 \times 0.08$ mm

Data collection

Nonius KappaCCD diffractometer
 Wide-angle φ and ω scans
 Absorption correction: multi-scan
 (SADABS; Sheldrick, 2004)
 $T_{min} = 0.850$, $T_{max} = 1.000$
 43 559 measured reflections
 3791 independent reflections

2661 reflections with $I > 2\sigma(I)$
 $R_{int} = 0.057$
 $\theta_{max} = 27.5$ °
 $h = -20 \rightarrow 20$
 $k = -13 \rightarrow 13$
 $l = -13 \rightarrow 13$

Refinement

Refinement on F^2
 $R[F^2 > 2\sigma(F^2)] = 0.047$
 $wR(F^2) = 0.115$
 $S = 1.03$
 3791 reflections
 200 parameters
 H atoms treated by a mixture of independent and constrained refinement

$w = 1/[\sigma^2(F_o^2) + (0.0486P)^2 + 0.5767P]$
 where $P = (F_o^2 + 2F_c^2)/3$
 $(\Delta/\sigma)_{max} < 0.001$
 $\Delta\rho_{max} = 0.25$ e Å⁻³
 $\Delta\rho_{min} = -0.20$ e Å⁻³

Table 1

Selected geometric parameters (Å, °).

O30–C11	1.3555 (18)	N20–C4	1.4578 (18)
N20–C5	1.2741 (19)		
C4–N20–C5	119.43 (13)	O30–C11–C10	119.90 (13)
N20–C4–C2	111.72 (12)	O30–C11–C6	119.92 (12)
N20–C5–C6	121.81 (13)		

Table 2

Hydrogen-bonding geometry (Å, °).

$D-H \cdots A$	$D-H$	$H \cdots A$	$D \cdots A$	$D-H \cdots A$
O30–H30 \cdots N20	0.98 (2)	1.65 (2)	2.5573 (16)	152.9 (19)

All H atoms bonded to C were placed in idealized positions and constrained to ride on their parent atoms, with $U_{iso}(H) = 1.2 U_{eq}(C)$ and $C-H = 0.95$ Å for aromatic H atoms, and $U_{iso}(H) = 1.5 U_{eq}(C)$ and $C-H = 0.98$ and 0.99 Å for all other H atoms. In addition, the

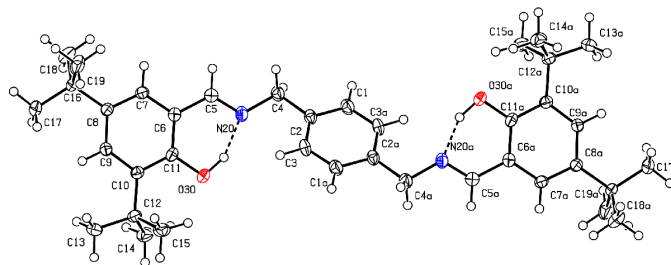


Figure 1

A view of the title compound, with the atom-numbering scheme. Displacement ellipsoids for non-H atoms are drawn at the 50% probability level. The dashed lines indicate hydrogen bonds. [Symmetry code: (a) $-x, -y, -z$.]

methyl groups were allowed to rotate but not to tip. The hydroxy H atom was refined isotropically.

Data collection: COLLECT (Nonius, 2002); cell refinement: DIRAX (Duisenberg, 1992); data reduction: EVALCCD (Duisenberg *et al.*, 2003); program(s) used to solve structure: SHELXS86 (Sheldrick, 1986); program(s) used to refine structure: SHELXL97 (Sheldrick, 1997); molecular graphics: PLATON (Spek, 2003); software used to prepare material for publication: PLATON.

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