

1,4-Bis(3,5-di-*tert*-butyl-2-hydroxybenzylidene-aminomethyl)benzeneDuncan M. Tooke,^{a*} Yufei Song,^b
Gerard A. van Albada,^b Jan
Reedijk^b and Anthony L. Spek^a^aBijvoet Centre for Biomolecular Research,
Department of Crystal and Structural Chemistry,
Utrecht University, Padualaan 8, 3584 CH
Utrecht, The Netherlands, and ^bLeiden Institute
of Chemistry, Gorlaeus Laboratories, Leiden
University, PO Box 9502, 2300 RA Leiden,
The Netherlands

Correspondence e-mail: d.m.tooke@chem.uu.nl

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.

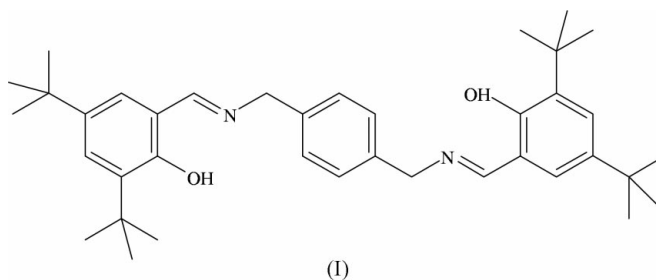
Received 20 September 2004

Accepted 22 September 2004

Online 30 September 2004

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, -CH₂), 7.08 (2H, phenol ring), 7.30 (4H, benzene rings), 7.36 (2H, phenol rings), 8.43 (2H, benzylidenimine).

Crystal data

C₃₈H₅₂N₂O₂
M_r = 568.82
 Monoclinic, *P*2₁/*c*
a = 15.8792 (18) Å
b = 10.4601 (11) Å
c = 10.4395 (15) Å
 β = 106.856 (10)°
V = 1659.5 (4) Å³
Z = 2

D_x = 1.138 Mg m⁻³
 Mo *K*α radiation
 Cell parameters from 46 reflections
 θ = 5.5–20.5°
 μ = 0.07 mm⁻¹
T = 150 K
 Block, yellow
 0.36 × 0.25 × 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* > 2σ(*I*)
R_{int} = 0.057
 θ_{max} = 27.5°
h = −20 → 20
k = −13 → 13
l = −13 → 13

Refinement

Refinement on *F*²
R [*F*² > 2σ(*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 $P = (F_o^2 + 2F_c^2)/3$
 $(\Delta/\sigma)_{max} < 0.001$
 $\Delta\rho_{max} = 0.25 \text{ e \AA}^{-3}$
 $\Delta\rho_{min} = -0.20 \text{ e \AA}^{-3}$

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, °).

<i>D</i> —H... <i>A</i>	<i>D</i> —H	H... <i>A</i>	<i>D</i> ... <i>A</i>	<i>D</i> —H... <i>A</i>
O30—H30...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

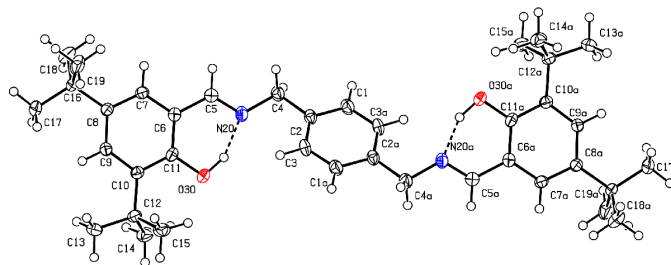


Figure 1

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.

The work described here has been supported by the Leiden University Study group WFMO (Werkgroep Fundamenteel Materialen Onderzoek). The ECOX project is financially supported by the Dutch Economy, Ecology, Technology (EET) programme, a joint programme of the Ministry of Economic Affairs, the Ministry of Education, Culture and Science, and the Ministry of Housing, Spatial Planning and the Environment. ALS and DMT thank the Council for the Chemical Sciences of the Netherlands Organization for Scientific Research (CW-NWO) for their support.

References

Duisenberg, A. J. M. (1992). *J. Appl. Cryst.* **25**, 92–96.
 Duisenberg, A. J. M., Kroon-Batenburg, L. M. J. & Schreurs, A. M. M. (2003). *J. Appl. Cryst.* **36**, 220–229.
 Nonius (2002). COLLECT. Nonius BV, Delft, The Netherlands.
 Sheldrick, G. M. (1986). SHELXS86. University of Göttingen, Germany.
 Sheldrick, G. M. (1997). SHELXL97. University of Göttingen, Germany.
 Sheldrick, G. M. (2004). SADABS. Bruker AXS, Karlsruhe, Germany.
 Song, Y. F., Koval, I. A., Gamez, P., van Albada, G. A., Mutikainen, I., Turpeinen, U. & Reedijk, J. (2004). *Polyhedron*, **23**, 1769–1775.
 Spek, A. L. (2003). *J. Appl. Cryst.* **36**, 7–13.