

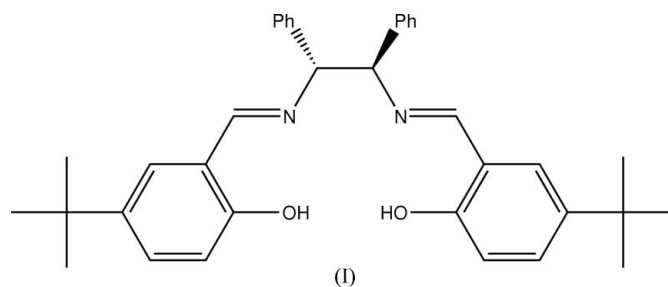
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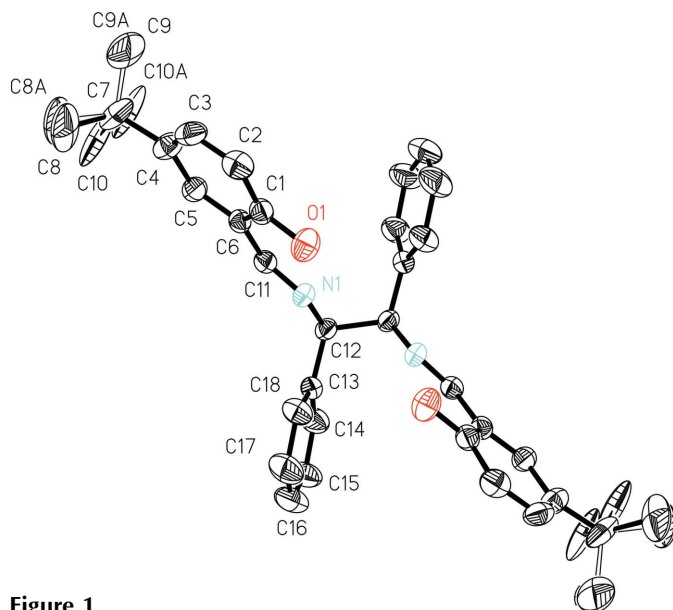
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## Key indicators

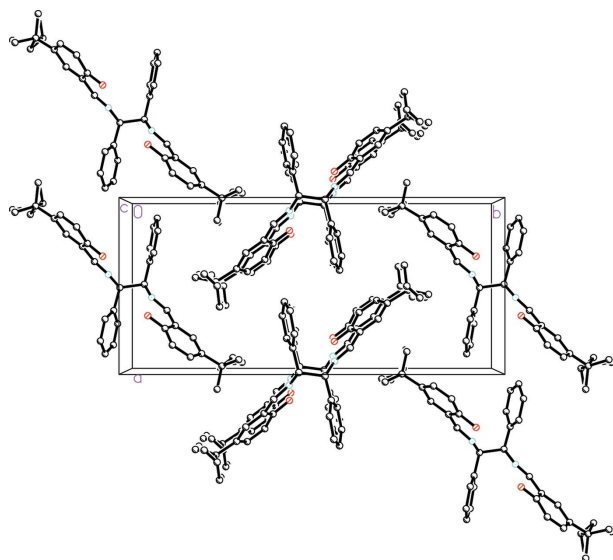
Single-crystal X-ray study  
 $T = 173$  K  
Mean  $\sigma(\text{C}-\text{C}) = 0.005$  Å  
Disorder in main residue  
 $R$  factor = 0.048  
 $wR$  factor = 0.126  
Data-to-parameter ratio = 10.6For details of how these key indicators were  
automatically derived from the article, see  
<http://journals.iucr.org/e>.**(1*R*,2*R*)-(+)-4,4'-Di-*tert*-butyl-2,2'-[1,2-diphenyl-ethane-1,2-diylbis(nitrilomethylidene)]diphenol**The crystal structure of the title compound,  $\text{C}_{36}\text{H}_{40}\text{N}_2\text{O}_2$ , has been determined at 173 (2) K. The molecule has  $C_2$  symmetry and the asymmetric unit contains one half-molecule. An intramolecular  $\text{O}-\text{H}\cdots\text{N}$  hydrogen bond is formed between the phenol OH group and the Schiff base N atom.Received 27 January 2006  
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## Comment

Manganese–salen complexes are used as catalysts due to their ability for highly stereoselective epoxidization of unfunctionalized alkenes (Lane & Burgess, 2003). The title Schiff base, (I), is one of the precursors to the first  $[\text{Mn}(\text{salen})]$  type of asymmetric epoxidation catalysts developed by Jacobsen and co-workers (Zhang *et al.*, 1990). We have previously reported the structures of  $(-)-(1*S*,2*S*)-N,N'$ -bis(salicylidene)-1,2-diphenyl-1,2-ethanediamine (Korendovych & Rybak-Akimova, 2003) and its  $\text{Mn}^{\text{III}}$  complex (Korendovych & Rybak-Akimova, 2004). In this paper, we present the results of our study of another derivative of salen. These results were obtained during a course on 'Diffraction Methods in Structure Determination' at Tufts University.Compound (I) was obtained in the enantiomerically pure  $(+)-(R,R)$  form by a Schiff base condensation of 5-*tert*-butyl-2-hydroxybenzaldehyde and  $(1*R*,2*R*)-(+)-1,2$ -diphenyl-1,2-ethanediamine. It crystallizes in the non-centrosymmetric space group  $P2_12_12$ . The molecule lies on a crystallographic twofold axis. Thus, one half-molecule constitutes the asymmetric unit (Fig. 1). The *tert*-butyl group exhibits orientational disorder, which was modelled with two positions.As in the case of the salicylaldehyde derivative, the structure of (I) is stabilized by the formation of an intramolecular hydrogen bond between the phenol OH group and the Schiff base N atom, forming a six-membered ring. The angle between the planes defined by the rings of the salicylidene fragment and the phenyl substituent in compound (I) is  $65.19(11)^\circ$ , which is very similar to the value of  $63.76(6)^\circ$  observed in the non-substituted salicylaldehyde derivative (Korendovych & Rybak-Akimova, 2003). The angles between the planes of

**Figure 1**

A view of the molecular structure of (I), showing the atom-labelling scheme of the asymmetric unit. Unlabelled atoms are related to labelled atoms by the symmetry operator  $(-x+2, -y+1, z)$ . Displacement ellipsoids are drawn at the 50% probability level. H atoms have been omitted. Both disorder components are shown.

**Figure 2**

The molecular packing of (I), viewed along the  $c$  axis. H atoms have been omitted. Both disorder components are shown.

symmetry-related aromatic rings are  $26.79(9)^\circ$  for the phenyl rings and  $42.10(12)^\circ$  for the salicylidene benzene rings. These values are also fairly close to those observed for the salicylaldehyde derivative, suggesting a minor influence of the *tert*-butyl group in the *para* position on the overall conformation of the molecule. The packing diagram of (I) is presented in Fig. 2.

## Experimental

The title compound was prepared according to the general procedure of Zhang *et al.* (1990). (1*R*,2*R*)-(+)-1,2-Diphenyl-1,2-ethanediamine

(99% purity, 99% e.e.) and 5-*tert*-butyl-2-hydroxybenzaldehyde were purchased from Aldrich and used without further purification. Suitable crystals were obtained by slow evaporation of an ethanol solution of (I). Spectroscopic analysis data were consistent with those reported by Chen *et al.* (2004).

### Crystal data

$C_{36}H_{40}N_2O_2$   
 $M_r = 532.70$   
 Orthorhombic,  $P2_12_12$   
 $a = 10.4213(16) \text{ \AA}$   
 $b = 22.729(4) \text{ \AA}$   
 $c = 6.3769(10) \text{ \AA}$   
 $V = 1510.4(4) \text{ \AA}^3$   
 $Z = 2$   
 $D_x = 1.171 \text{ Mg m}^{-3}$

Mo  $K\alpha$  radiation  
 Cell parameters from 1286 reflections  
 $\theta = 2.7\text{--}26.7^\circ$   
 $\mu = 0.07 \text{ mm}^{-1}$   
 $T = 173(2) \text{ K}$   
 Needle, yellow  
 $0.16 \times 0.06 \times 0.04 \text{ mm}$

### Data collection

Bruker SMART CCD area-detector diffractometer  
 $\varphi$  and  $\omega$  scans  
 Absorption correction: none  
 11737 measured reflections  
 2142 independent reflections

979 reflections with  $I > 2\sigma(I)$   
 $R_{\text{int}} = 0.118$   
 $\theta_{\text{max}} = 28.3^\circ$   
 $h = -13 \rightarrow 13$   
 $k = -29 \rightarrow 30$   
 $l = -8 \rightarrow 8$

### Refinement

Refinement on  $F^2$   
 $R[F^2 > 2\sigma(F^2)] = 0.048$   
 $wR(F^2) = 0.126$   
 $S = 0.88$   
 2142 reflections  
 202 parameters  
 H atoms treated by a mixture of independent and constrained refinement

$w = 1/[\sigma^2(F_o^2) + (0.0572P)^2]$   
 where  $P = (F_o^2 + 2F_c^2)/3$   
 $(\Delta/\sigma)_{\text{max}} < 0.001$   
 $\Delta\rho_{\text{max}} = 0.19 \text{ e \AA}^{-3}$   
 $\Delta\rho_{\text{min}} = -0.16 \text{ e \AA}^{-3}$   
 Extinction correction: SHELXL97 (Sheldrick, 1997)  
 Extinction coefficient: 0.031(3)

**Table 1**

Selected bond lengths ( $\text{\AA}$ ).

N1—C11	1.278(4)	O1—H1	1.05(4)
N1—C12	1.453(4)	C6—C11	1.448(4)
O1—C1	1.348(4)	C12—C12 <sup>i</sup>	1.559(6)

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

**Table 2**

Hydrogen-bond geometry ( $\text{\AA}, ^\circ$ ).

$D-H \cdots A$	$D-H$	$H \cdots A$	$D \cdots A$	$D-H \cdots A$
O1—H1 $\cdots$ N1	1.05(4)	1.64(5)	2.578(4)	147(4)

Carbon-bound H atoms were placed in idealized positions and refined using a riding model, with  $C-H = 0.95\text{--}1.00 \text{ \AA}$  and with  $U_{\text{iso}}(H) = 1.2U_{\text{eq}}(C)$ . The H atom bound to the phenol O atom was found in a difference Fourier map and refined isotropically. In the absence of significant anomalous dispersion effects, Friedel pairs were averaged. The relatively high  $R_{\text{int}}$  value (0.118) was attributed to small size of the crystal, which resulted in weak diffraction. All attempts to obtain better quality crystals failed.

The *tert*-butyl group exhibits orientational disorder, which was modelled with two positions. The disordered C atoms were presumed to have the same anisotropic displacement parameters. Furthermore, C—C(methyl) distances in the *tert*-butyl group were restrained to be

the same for both orientations. The occupancies of the two sites converged at 0.622 (13) and 0.378 (13).

Data collection: *SMART* (Bruker, 1998); cell refinement: *SAINTE* (Bruker, 1998); 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, 2000); software used to prepare material for publication: *XCIF* in *SHELXTL*.

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