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3,5-Di-*tert*-butyl-*N*-[2-(methylsulfanyl)phenyl]-salicylaldimine

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Key indicators

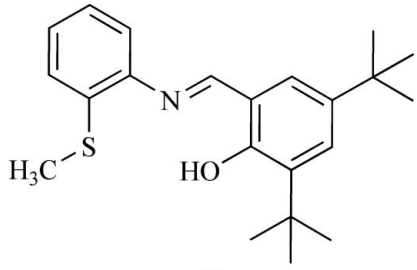
Single-crystal X-ray study
T = 173 K
Mean σ(C–C) = 0.003 Å
R factor = 0.047
wR factor = 0.147
Data-to-parameter ratio = 20.0

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

The molecule of the title compound, $C_{22}H_{29}NOS$, is non-planar, with a dihedral angle of 32.06 (9)° between the two aromatic rings.

Comment

Schiff base ligands are routinely used in coordination chemistry. The choice of ligand for a metal complex is crucial in determining its chemical and physical properties. Our group is interested in the synthesis and preparation of novel sulfur-containing Schiff base ligands for use in coordination chemistry (Halbach & Hamaker 2006; Hamaker & Oberts, 2006). As part of our ongoing studies, we report the synthesis, properties and crystal structure of the title compound, (I).



(I)

Unlike the 3-methoxy derivative (Hamaker & Corgliano, 2006), the molecule of (I) is non-planar (Fig. 1), with a dihedral angle between the two aromatic rings of 32.06 (9)°. The bond lengths and angles (Table 1) are similar to those of related molecules (Hamaker & Corgliano, 2006; Hamaker *et al.*, 2006). Similar to the 3-methoxy derivative, there is an intramolecular O1—H1···N1 hydrogen bond (Table 2).

Experimental

A solution of 2-(methylsulfanyl)aniline (1.84 g, 13.2 mmol) in 30 ml ethanol was placed into a 150 ml flask equipped with a reflux condenser. To this solution, 3,5-di-*tert*-butyl-2-hydroxybenzaldehyde (3.01 g, 12.9 mmol) was added. The mixture was refluxed for 1 h and then cooled to room temperature, during which time a light-yellow precipitate formed. The mixture was then placed in a freezer at 233 K for 3 d. The solid was collected by suction filtration, washed with ice-cold ethanol and air-dried, resulting in shiny yellow microcrystals (yield: 3.648 g, 86%). Single crystals of (I) were grown by slow evaporation of a saturated ethanol solution. IR (Nujol, ν, cm^{-1}): 3577 (OH); 1612 (*s*, N=C). Analysis calculated (found) (%) for $C_{22}H_{29}NOS$: C 74.32 (74.37), H 8.22 (8.26), N 3.94 (4.10).

Crystal data

$C_{22}H_{29}NOS$
M_r = 355.52
Monoclinic, $P2_1/c$
a = 17.876 (3) Å
b = 8.990 (2) Å
c = 12.6963 (12) Å
β = 98.161 (8)°
V = 2019.7 (6) Å^3
Z = 4
D_x = 1.169 Mg m^{-3}
Mo $K\alpha$ radiation
μ = 0.17 mm^{-1}
T = 173 (2) K
Prism, yellow
0.48 × 0.37 × 0.37 mm

Data collection

Enraf–Nonius CAD-4 diffractometer
$\omega/2\theta$ scans
Absorption correction: none
4796 measured reflections
4589 independent reflections
3291 reflections with $I > 2\sigma(I)$
R_{int} = 0.041
θ_{max} = 27.5°
3 standard reflections
frequency: 120 min
intensity decay: 4%

Refinement

Refinement on F^2
$R[F^2 > 2\sigma(F^2)]$ = 0.047
$wR(F^2)$ = 0.147
S = 1.04
4589 reflections
230 parameters
H atoms treated by a mixture of independent and constrained refinement
$w = 1/[\sigma^2(F_o^2) + (0.0762P)^2 + 1.1485P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} < 0.001$
$\Delta\rho_{max}$ = 0.41 e Å^{-3}
$\Delta\rho_{min}$ = −0.32 e Å^{-3}

Table 1
Selected geometric parameters (Å, °).

C1—S1	1.792 (2)	C7—N1	1.413 (2)
C2—S1	1.760 (2)	C10—N1	1.281 (2)
N1—C10—C11	121.88 (18)	C2—S1—C1	102.46 (11)
C10—N1—C7	122.34 (17)		

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

D—H···A	D—H	H···A	D···A	D—H···A
O1—H1···N1	0.80 (4)	1.90 (4)	2.571 (2)	142 (3)

The H atom of the hydroxy group was found in a difference Fourier map and refined isotropically. The other H atoms were treated as riding atoms, with C—H distances of 0.95 (aromatic) or 0.98 Å (aliphatic), and with U_{iso}(H) = 1.2U_{eq}(C) or 1.5U_{eq}(methyl C). The displacement ellipsoids for the methyl carbons of the C30–C33 *tert*-butyl group are elongated, suggesting possible disorder. Attempts to model the *tert*-butyl group as disordered over two sites did not give satisfactory results. The elongated displacement ellipsoids reflect partial free rotation of the *tert*-butyl group.

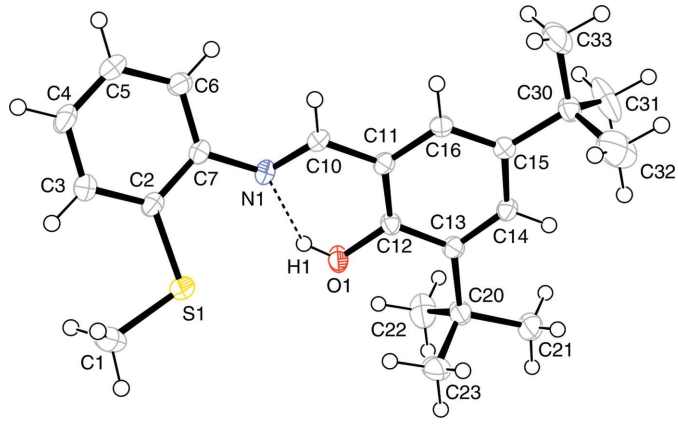


Figure 1
The molecular structure of (I) with 50% probability displacement ellipsoids. The dashed line indicates the hydrogen bond.

Data collection: *CAD-4 EXPRESS* (Enraf–Nonius, 1994); cell refinement: *CAD-4 EXPRESS*; data reduction: *XCAD4* (Harms & Wocadlo, 1995); program(s) used to solve structure: *DIRDIF99* (Beurskens *et al.*, 1999); program(s) used to refine structure: *SHELXL97* (Sheldrick, 1997); molecular graphics: *ORTEP-3 for Windows* (Farrugia, 1997); software used to prepare material for publication: *WinGX* (Farrugia, 1999).

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