Acta Crystallographica Section E
Structure Reports Online
ISSN 1600-5368

6-[(5-*tert*-Butyl-2-hydroxyanilino)methylene]cyclohexa-2,4-dienone

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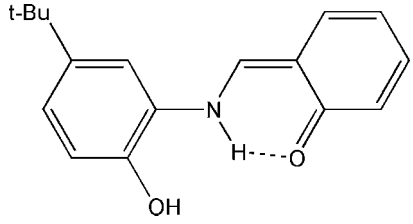
Received 20 November 2007; accepted 20 November 2007

Key indicators: single-crystal X-ray study; T = 153 K; mean σ(C–C) = 0.002 Å; R factor = 0.039; wR factor = 0.108; data-to-parameter ratio = 18.0.

In the title compound, $C_{17}H_{19}NO_2$, the dihedral angle between the two aromatic rings is 26.02 (5)°. One phenol O atom is deprotonated and the N atom of the azomethine unit carries the H atom, forming an intramolecular hydrogen bond. The packing is stabilized by an O—H···O hydrogen bond.

Related literature

Aromatic Schiff bases with *ortho*-hydroxy groups are useful as acyclic polydentate ligands for the preparation of chelate complexes with a wide variety of metal ions (Freeman & White, 1956; Calligaris & Randaccio, 1987; Pettinari *et al.*, 2001; Hernández-Molina & Mederos, 2004). For related literature, see: Böhme & Günther (2006, 2007); Böhme, Wiesner & Günther (2006); Dubs *et al.* (2000); Hopfl *et al.* (1998); Nazir *et al.* (2000); Pradeep (2005).



Experimental

Crystal data

$C_{17}H_{19}NO_2$
M_r = 269.33
Monoclinic, $P2_1/c$
a = 10.3600 (4) Å
b = 9.5756 (3) Å
c = 14.7335 (6) Å
β = 99.664 (2)°
V = 1440.87 (9) Å^3
Z = 4
Mo $K\alpha$ radiation
μ = 0.08 mm^{-1}
T = 153 (2) K
0.5 × 0.37 × 0.25 mm

Data collection

Bruker SMART CCD area-detector diffractometer
Absorption correction: none
14317 measured reflections
3478 independent reflections
2819 reflections with $I > 2\sigma(I)$
R_{int} = 0.025

Refinement

$R[F^2 > 2\sigma(F^2)]$ = 0.039
$wR(F^2)$ = 0.108
S = 1.08
3478 reflections
193 parameters
H atoms treated by a mixture of independent and constrained refinement
$\Delta\rho_{max}$ = 0.33 e Å^{-3}
$\Delta\rho_{min}$ = −0.22 e Å^{-3}

Table 1
Hydrogen-bond geometry (Å, °).

D—H···A	D—H	H···A	D···A	D—H···A
N1—H3···O1	0.939 (16)	1.83 (2)	2.601 (1)	137.8 (13)
O2—H2···O1^{i}	0.84	1.75	2.583 (1)	174 (1)

Symmetry code: (i) $-x+1, y+\frac{1}{2}, -z+\frac{5}{2}$.

Data collection: *SMART* (Bruker, 2004); cell refinement: *SAINT* (Bruker, 2004); data reduction: *SAINT*; program(s) used to solve structure: *SHELXS97* (Sheldrick, 1997); program(s) used to refine structure: *SHELXL97* (Sheldrick, 1997); molecular graphics: *ORTEP-3* (Farrugia, 1997); software used to prepare material for publication: *SHELXL97*.

Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: BT2630).

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  doi:10.1107/S1600536807061211

supplementary materials

Acta Cryst. (2008). E**64**, o14 [**doi:10.1107/S1600536807061211**]

6-[(5-*tert*-Butyl-2-hydroxyanilino)methylene]cyclohexa-2,4-dienone

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Comment

Recently, we are working on silicon and titanium complexes with tridentate O,*N*,*O*-ligands (Böhme & Günther, 2006; Böhme, Wiesner & Günther, 2006; Böhme & Günther, 2007). The title compound, $C_{17}H_{19}NO_2$, was prepared in order to extend the series of available ligands. The preparation of the title compound was performed according to methods described in the literature for the parent compound salicyclidene-*o*-aminophenol ("salopH_2") (Freeman and White, 1956; Pettinari *et al.*, 2001) by reaction of salicyclaldehyde and 2-amino-4-*tert*-butylphenol in ethanol. The molecule is non-planar with a dihedral angle between the two aromatic rings of 26.02 (5)°. The atom H3 forms an intramolecular hydrogen bond between the phenolic oxygen atom O1 and N1 of the azomethine unit. The hydrogen atom H3 is localized at a distance of 0.94 (2) Å from N1. This hints to the presence of the keto-amine form. The presence of a quinoidal structure is further supported by the shortening of the bond O1—C3 to 1.296 (1) Å and the lengthening of the adjacent C—C bonds in the phenyl ring [C2—C3 1.437 (2), C3—C4 1.426 (2) Å] (Nazir *et al.*, 2000). There are few structure reports of Schiff-bases with oxygen in *ortho*-position where the intramolecular bridging hydrogen atom is localized at the nitrogen atom (*e.g.* Pradeep, 2005; Dubs *et al.*, 2000; Hopfl *et al.*, 1998). The crystal packing is stabilized by a hydrogen bond O2—H2···O1 forming a helix along the crystallographic 2_1 axis.

Experimental

2-Amino-4-*tert*-butylphenol (3.07 g, 18.6 mmol) was dissolved in ethanol (100 ml). This solution was heated slowly to 313 K and after a few minutes salicylaldehyde (2.27 g, 1.96 ml, 18.6 mmol) was added with a syringe. The reaction mixture was boiled at reflux temperature for 1.5 h. After that time a red solution was formed. The solution was concentrated in a vacuum to a small volume (30 ml) until a red crystalline precipitate deposited. The precipitate was filtered off and washed with ethanol. After drying, the product was purified by recrystallization with ethanol. Red prisms (4.38 g, 87.6%, m.p. 415 K). NMR ($CDCl_3$, 300 K, TMS): ^{1}H: δ=12,37 (s, OH), 8.64 (s, CH—N), 7.41–6.92 (m, $CH_{aromatic}$), 1.33 (s, $C(CH_3)_3$); ^{13}C: 163.5 (C1), 160.5 (C3), 147.4 (C9), 144.0 (C12), 135.0, 133.4, 132.5, 125.6, 119.4, 119.3, 117.2, 115.4, 115.4 (9 signals for aromatic C), 34.3 (C14), 31.5 (C15—C17).

Refinement

Hydrogen atoms bonded to C were positioned geometrically and allowed to ride on their parent atoms, with C—H = 0.95 Å for Csp^2 and 0.98 for methyl. U_{iso}(H) = xU_{eq}(C), where x = 1.2 for Csp^2 and 1.5 for methyl. The amino H atom was located by difference Fourier synthesis and freely refined.

Figures

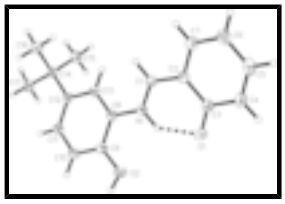

Fig. 1. The molecular structure of the title compound, drawn with 50% probability displacement ellipsoids.

6-[(5-*tert*-Butyl-2-hydroxyanilino)methylene]cyclohexa-2,4-dienone

Crystal data

$C_{17}H_{19}NO_2$	F_{000} = 576
M_r = 269.33	D_x = 1.242 Mg m^{-3}
Monoclinic, $P2_1/c$	Mo $K\alpha$ radiation λ = 0.71073 Å
Hall symbol: -P 2ybc	Cell parameters from 5301 reflections
a = 10.3600 (4) Å	θ = 2.8–30.5º
b = 9.5756 (3) Å	μ = 0.08 mm^{-1}
c = 14.7335 (6) Å	T = 153 (2) K
β = 99.664 (2)º	Block, orange
V = 1440.87 (9) Å^3	0.5 × 0.37 × 0.25 mm
Z = 4	

Data collection

Bruker SMART CCD area-detector diffractometer	2819 reflections with $I > 2\sigma(I)$
Radiation source: sealed tube	R_{int} = 0.025
Monochromator: graphite	θ_{max} = 28.0º
T = 153(2) K	θ_{min} = 2.6º
phi and ω scans	h = −13→13
Absorption correction: none	k = −9→12
14317 measured reflections	l = −19→16
3478 independent reflections	

Refinement

Refinement on F^2	Secondary atom site location: difference Fourier map
Least-squares matrix: full	Hydrogen site location: inferred from neighbouring sites
$R[F^2 > 2\sigma(F^2)]$ = 0.039	H atoms treated by a mixture of independent and constrained refinement
$wR(F^2)$ = 0.108	$w = 1/[\sigma^2(F_o^2) + (0.0528P)^2 + 0.3328P]$ where $P = (F_o^2 + 2F_c^2)/3$
S = 1.08	$(\Delta/\sigma)_{max}$ = 0.001

3478 reflections	$\Delta\rho_{max} = 0.33$ e Å^{-3}
193 parameters	$\Delta\rho_{min} = -0.22$ e Å^{-3}
Primary atom site location: structure-invariant direct methods	Extinction correction: none

Special details

Geometry. All e.s.d.'s (except the e.s.d. in the dihedral angle between two l.s. planes) are estimated using the full covariance matrix. The cell e.s.d.'s are taken into account individually in the estimation of e.s.d.'s in distances, angles and torsion angles; correlations between e.s.d.'s in cell parameters are only used when they are defined by crystal symmetry. An approximate (isotropic) treatment of cell e.s.d.'s is used for estimating e.s.d.'s involving l.s. planes.

Refinement. Refinement of F^2 against ALL reflections. The weighted *R*-factor *wR* and goodness of fit S are based on F^2, conventional *R*-factors *R* are based on F, with F set to zero for negative F^2. The threshold expression of $F^2 > 2\text{sigma}(F^2)$ is used only for calculating *R*-factors(gt) *etc*. and is not relevant to the choice of reflections for refinement. *R*-factors based on F^2 are statistically about twice as large as those based on F, and R– factors based on ALL data will be even larger.

Fractional atomic coordinates and isotropic or equivalent isotropic displacement parameters (Å^2)

	x	*y*	*z*	U_{iso}*/U_{eq}
O1	0.59234 (8)	0.06870 (8)	1.16198 (5)	0.0251 (2)
O2	0.43203 (9)	0.39390 (9)	1.20845 (6)	0.0278 (2)
H2	0.4195	0.4471	1.2514	0.042*
N1	0.47127 (9)	0.26094 (9)	1.05548 (6)	0.0194 (2)
H3	0.4953 (14)	0.2185 (16)	1.1133 (11)	0.039 (4)*
C1	0.53484 (11)	0.21410 (11)	0.99239 (8)	0.0199 (2)
H1	0.5173 (12)	0.2582 (14)	0.9312 (9)	0.022 (3)*
C2	0.62986 (11)	0.10555 (11)	1.00874 (8)	0.0198 (2)
C3	0.65831 (11)	0.03867 (11)	1.09717 (8)	0.0207 (2)
C4	0.76261 (12)	−0.06030 (12)	1.10942 (9)	0.0269 (3)
H4	0.7860	−0.1056	1.1672	0.032*
C5	0.82989 (12)	−0.09136 (13)	1.03933 (10)	0.0308 (3)
H5	0.8999	−0.1566	1.0501	0.037*
C6	0.79790 (13)	−0.02904 (13)	0.95184 (10)	0.0313 (3)
H6	0.8441	−0.0535	0.9036	0.038*
C7	0.69883 (12)	0.06762 (12)	0.93748 (8)	0.0252 (3)
H7	0.6762	0.1098	0.8786	0.030*
C8	0.37998 (10)	0.37285 (11)	1.04619 (7)	0.0187 (2)
C9	0.36246 (11)	0.44028 (11)	1.12805 (8)	0.0203 (2)
C10	0.27440 (11)	0.55129 (12)	1.12145 (8)	0.0227 (2)
H10	0.2626	0.6006	1.1754	0.027*
C11	0.20359 (11)	0.59050 (11)	1.03643 (8)	0.0213 (2)
H11	0.1435	0.6659	1.0339	0.026*
C12	0.21797 (10)	0.52265 (11)	0.95479 (7)	0.0187 (2)
C13	0.30923 (11)	0.41376 (11)	0.96159 (8)	0.0195 (2)
H13	0.3232	0.3668	0.9073	0.023*
C14	0.13591 (11)	0.56085 (11)	0.86144 (8)	0.0211 (2)
C15	0.06649 (12)	0.42959 (12)	0.81692 (9)	0.0273 (3)

H15A	0.0097	0.4553	0.7593	0.041*
H15B	0.0135	0.3879	0.8591	0.041*
H15C	0.1320	0.3619	0.8040	0.041*
C16	0.22489 (13)	0.61849 (14)	0.79661 (9)	0.0313 (3)
H16A	0.2679	0.7039	0.8230	0.047*
H16B	0.1721	0.6396	0.7366	0.047*
H16C	0.2914	0.5487	0.7889	0.047*
C17	0.03131 (12)	0.67090 (13)	0.87157 (9)	0.0296 (3)
H17A	0.0741	0.7574	0.8962	0.044*
H17B	−0.0250	0.6361	0.9138	0.044*
H17C	−0.0218	0.6894	0.8112	0.044*

Atomic displacement parameters (Å^2)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
O1	0.0310 (4)	0.0266 (4)	0.0172 (4)	0.0025 (3)	0.0029 (3)	0.0025 (3)
O2	0.0329 (5)	0.0336 (5)	0.0152 (4)	0.0096 (4)	−0.0004 (3)	−0.0037 (3)
N1	0.0224 (5)	0.0196 (4)	0.0155 (5)	0.0022 (3)	0.0013 (4)	0.0000 (3)
C1	0.0222 (5)	0.0197 (5)	0.0175 (5)	−0.0008 (4)	0.0031 (4)	0.0008 (4)
C2	0.0208 (5)	0.0184 (5)	0.0202 (6)	−0.0012 (4)	0.0034 (4)	−0.0004 (4)
C3	0.0226 (5)	0.0180 (5)	0.0206 (6)	−0.0027 (4)	0.0006 (4)	−0.0005 (4)
C4	0.0282 (6)	0.0210 (5)	0.0289 (6)	0.0014 (4)	−0.0021 (5)	0.0034 (4)
C5	0.0265 (6)	0.0232 (6)	0.0423 (8)	0.0058 (5)	0.0051 (5)	0.0021 (5)
C6	0.0324 (7)	0.0272 (6)	0.0379 (7)	0.0041 (5)	0.0164 (6)	−0.0002 (5)
C7	0.0285 (6)	0.0243 (5)	0.0244 (6)	0.0009 (4)	0.0086 (5)	0.0016 (5)
C8	0.0191 (5)	0.0186 (5)	0.0184 (5)	0.0005 (4)	0.0035 (4)	−0.0010 (4)
C9	0.0208 (5)	0.0232 (5)	0.0165 (5)	−0.0007 (4)	0.0019 (4)	−0.0012 (4)
C10	0.0255 (6)	0.0241 (5)	0.0187 (6)	0.0016 (4)	0.0045 (4)	−0.0045 (4)
C11	0.0219 (5)	0.0198 (5)	0.0227 (6)	0.0019 (4)	0.0046 (4)	−0.0007 (4)
C12	0.0195 (5)	0.0183 (5)	0.0181 (5)	−0.0023 (4)	0.0024 (4)	0.0018 (4)
C13	0.0228 (5)	0.0199 (5)	0.0160 (5)	−0.0011 (4)	0.0033 (4)	−0.0022 (4)
C14	0.0231 (5)	0.0198 (5)	0.0191 (6)	−0.0005 (4)	0.0004 (4)	0.0014 (4)
C15	0.0278 (6)	0.0233 (5)	0.0277 (6)	−0.0006 (5)	−0.0045 (5)	−0.0011 (5)
C16	0.0345 (7)	0.0371 (7)	0.0216 (6)	−0.0056 (5)	0.0026 (5)	0.0061 (5)
C17	0.0339 (6)	0.0242 (6)	0.0281 (7)	0.0071 (5)	−0.0022 (5)	0.0029 (5)

Geometric parameters (Å, °)

O1—C3	1.296 (1)	C9—C10	1.3935 (15)
O2—C9	1.354 (1)	C10—C11	1.3928 (16)
O2—H2	0.8400	C10—H10	0.9500
N1—C1	1.3055 (14)	C11—C12	1.3969 (16)
N1—C8	1.4206 (13)	C11—H11	0.9500
N1—H3	0.94 (2)	C12—C13	1.3998 (15)
C1—C2	1.4240 (15)	C12—C14	1.5348 (15)
C1—H1	0.985 (13)	C13—H13	0.9500
C2—C7	1.4131 (16)	C14—C17	1.5367 (16)
C2—C3	1.437 (2)	C14—C16	1.5369 (17)
C3—C4	1.426 (2)	C14—C15	1.5392 (15)

C4—C5	1.3720 (19)	C15—H15A	0.9800
C4—H4	0.9500	C15—H15B	0.9800
C5—C6	1.4087 (19)	C15—H15C	0.9800
C5—H5	0.9500	C16—H16A	0.9800
C6—C7	1.3718 (17)	C16—H16B	0.9800
C6—H6	0.9500	C16—H16C	0.9800
C7—H7	0.9500	C17—H17A	0.9800
C8—C13	1.3920 (15)	C17—H17B	0.9800
C8—C9	1.4065 (15)	C17—H17C	0.9800
C9—O2—H2	109.5	C10—C11—C12	122.20 (10)
C1—N1—C8	126.71 (10)	C10—C11—H11	118.9
C1—N1—H3	114.1 (9)	C12—C11—H11	118.9
C8—N1—H3	119.0 (9)	C11—C12—C13	116.95 (10)
N1—C1—C2	123.19 (10)	C11—C12—C14	122.52 (10)
N1—C1—H1	117.9 (7)	C13—C12—C14	120.50 (10)
C2—C1—H1	118.9 (7)	C8—C13—C12	121.43 (10)
C7—C2—C1	118.90 (10)	C8—C13—H13	119.3
C7—C2—C3	120.45 (10)	C12—C13—H13	119.3
C1—C2—C3	120.62 (10)	C12—C14—C17	111.72 (9)
O1—C3—C4	122.43 (10)	C12—C14—C16	110.04 (9)
O1—C3—C2	121.04 (10)	C17—C14—C16	108.69 (10)
C4—C3—C2	116.53 (11)	C12—C14—C15	109.64 (9)
C5—C4—C3	121.25 (11)	C17—C14—C15	108.35 (9)
C5—C4—H4	119.4	C16—C14—C15	108.33 (10)
C3—C4—H4	119.4	C14—C15—H15A	109.5
C4—C5—C6	121.72 (11)	C14—C15—H15B	109.5
C4—C5—H5	119.1	H15A—C15—H15B	109.5
C6—C5—H5	119.1	C14—C15—H15C	109.5
C7—C6—C5	118.81 (12)	H15A—C15—H15C	109.5
C7—C6—H6	120.6	H15B—C15—H15C	109.5
C5—C6—H6	120.6	C14—C16—H16A	109.5
C6—C7—C2	121.16 (11)	C14—C16—H16B	109.5
C6—C7—H7	119.4	H16A—C16—H16B	109.5
C2—C7—H7	119.4	C14—C16—H16C	109.5
C13—C8—C9	120.95 (10)	H16A—C16—H16C	109.5
C13—C8—N1	122.72 (10)	H16B—C16—H16C	109.5
C9—C8—N1	116.32 (9)	C14—C17—H17A	109.5
O2—C9—C10	123.87 (10)	C14—C17—H17B	109.5
O2—C9—C8	118.25 (10)	H17A—C17—H17B	109.5
C10—C9—C8	117.88 (10)	C14—C17—H17C	109.5
C11—C10—C9	120.55 (10)	H17A—C17—H17C	109.5
C11—C10—H10	119.7	H17B—C17—H17C	109.5
C9—C10—H10	119.7		
C8—N1—C1—C2	176.77 (10)	C13—C8—C9—C10	−1.48 (16)
N1—C1—C2—C7	−176.40 (11)	N1—C8—C9—C10	179.50 (10)
N1—C1—C2—C3	1.59 (17)	O2—C9—C10—C11	−178.18 (11)
C7—C2—C3—O1	−177.34 (10)	C8—C9—C10—C11	1.92 (17)
C1—C2—C3—O1	4.69 (16)	C9—C10—C11—C12	−0.65 (18)

C7—C2—C3—C4	3.18 (16)	C10—C11—C12—C13	−1.09 (16)
C1—C2—C3—C4	−174.79 (10)	C10—C11—C12—C14	177.18 (10)
O1—C3—C4—C5	179.24 (11)	C9—C8—C13—C12	−0.27 (16)
C2—C3—C4—C5	−1.29 (16)	N1—C8—C13—C12	178.69 (10)
C3—C4—C5—C6	−1.08 (19)	C11—C12—C13—C8	1.54 (16)
C4—C5—C6—C7	1.58 (19)	C14—C12—C13—C8	−176.77 (10)
C5—C6—C7—C2	0.38 (18)	C11—C12—C14—C17	−4.95 (15)
C1—C2—C7—C6	175.20 (11)	C13—C12—C14—C17	173.26 (10)
C3—C2—C7—C6	−2.80 (17)	C11—C12—C14—C16	115.87 (12)
C1—N1—C8—C13	25.00 (17)	C13—C12—C14—C16	−65.91 (13)
C1—N1—C8—C9	−156.00 (11)	C11—C12—C14—C15	−125.08 (11)
C13—C8—C9—O2	178.62 (10)	C13—C12—C14—C15	53.14 (14)
N1—C8—C9—O2	−0.40 (15)		

Hydrogen-bond geometry (Å, °)

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
N1—H3···O1	0.939 (16)	1.83 (2)	2.601 (1)	137.8 (13)
O2—H2···O1^{i}	0.84	1.75	2.583 (1)	174 (1)

Symmetry codes: (i) −*x*+1, *y*+1/2, −*z*+5/2.

Fig. 1