C109—O11—C102	122.4 (3)	O14—C112—O13	122.7 (4)
C112—O13—C107	117.3 (3)	O22—C202—O21	117.2 (3)
C203—C1—C108	116.6 (3)	O21—C202—C203	117.5 (3)
O12—C102—O11	116.6 (4)	C204—C203—C1	128.2 (3)
C107—C108—C1	122.5 (3)	C202—C203—C1	111.4 (3)
C109—C108—C1	121.0 (3)	O23—C205—C210	115.9 (3)
O11—C109—C108	115.6 (3)		

O12—C102—C103—C104	176.1 (4)
C112—O13—C107—C108	−108.8 (4)
O13—C107—C108—C1	−0.2 (5)
C203—C1—C108—C107	107.0 (4)
C203—C1—C108—C109	−78.5 (4)
C103—C104—C110—C105	177.7 (4)
O22—C202—C203—C204	−177.8 (4)
C108—C1—C203—C204	−2.3 (5)
C108—C1—C203—C202	177.0 (3)
O24—C211—C212—C213	−177.7 (4)

The temperature of the crystal was controlled using an Oxford Cryosystem Cryostream Cooler (Cosier & Glazer, 1986). H atoms were added at calculated positions and refined using a riding model including free rotation of methyl groups about C—C bonds. Anisotropic displacement parameters were used for all non-H atoms; H atoms were given isotropic displacement parameters equal to 1.2 (or 1.5 for methyl H atoms) times the equivalent isotropic displacement parameter of the atom to which they are attached.

Data collection: Siemens *P3R3* system. Cell refinement: Siemens *P3R3* system. Data reduction: *SHELXTL-Plus* (Sheldrick, 1990). Program(s) used to solve structure: *SHELXTL-Plus*. Program(s) used to refine structure: *SHELXL93* (Sheldrick, 1993). Molecular graphics: *SHELXTL-Plus*. Software used to prepare material for publication: *SHELXL93*.

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Lists of structure factors, anisotropic displacement parameters, H-atom coordinates and complete geometry have been deposited with the IUCr (Reference: CF1115). Copies may be obtained through The Managing Editor, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

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2-{1-[(2-Amino-4,5-dimethylphenyl)imino]ethyl}phenol

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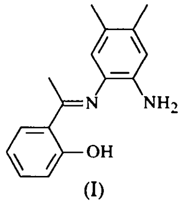
Abstract

In the title compound, $C_{16}H_{18}N_2O$, the imine $N\cdots O$ separation is 2.521 (2) Å, indicative of intramolecular hydrogen bonding within the salicylideneimine unit. The two aromatic rings are inclined at an angle of 50.04 (6)° with respect to one another, which results in a conformation unsuitable for meridional tridentate ligand complexation to a metal.

Comment

The X-ray structures of various metal complexes with tridentate Schiff base ligands have been reported (Elias, Hilms & Paulus, 1982; Roper, Paulus & Elias, 1989). Both square-planar and octahedral geometries are known, and in the latter case, the two ligands occupy meridional positions (Sim, Sinn, Petty, Merrill & Wilson, 1981). However, no X-ray structures of uncoordinated tridentate ligands of this type have yet been reported. We have therefore determined the crystal structure of the new NNO-donor tridentate ligand, (I), in order to assess the structural changes which occur upon coordination to a metal species and to compare these changes with those of the related ONNO-donor tetradentate ligands (Corden, Errington, Moore & Wallbridge, 1996; Cannadine, Corden, Errington, Moore & Wallbridge, 1996).



(I)

The molecular structure of the title compound displays a C9—C7—N2—C6 backbone which is not exactly planar, but has a torsion angle of 174.18 (15)° about the C7—N2 bond; the two aromatic rings are inclined at an angle of 50.04 (6)° with respect to one another (Fig. 1). The enolimine tautomer is observed rather than the ketamine form, as is apparent from the O1—C14 and C7—N2 bond distances of 1.349 (2) and 1.298 (2) Å, respectively, which are consistent with the presence of O—C single and C=N double bonds, respectively. There is evidence of intramolecular hydrogen bonding between the hydroxy and imine groups since the O1···N2 distance of 2.521 (2) Å is significantly shorter than the sum of the corresponding van der Waals radii of 3.07 Å (Bondi, 1964). A shortening of the C1—N1 bond to 1.385 (3) Å, compared with a value of 1.407 (5) Å in 1,2-diaminobenzene (Stalhandske, 1981), and the C6—N2 bond length of 1.421 (2) Å, indicate some degree of overlap between the N1 lone pair and the π system of the aromatic ring, and accordingly the NH_2 group has had its H atoms inserted at positions calculated for a planar configuration. There are no significant intermolecular interactions involving this group.

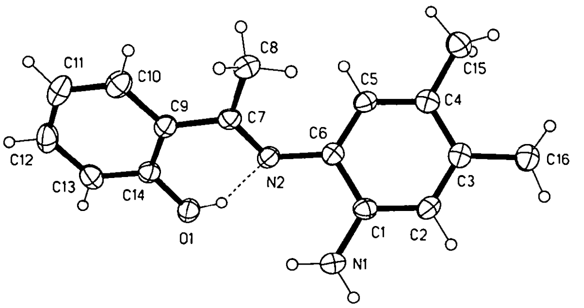


Fig. 1. View of the title molecule showing the atomic numbering scheme. Displacement ellipsoids are drawn at the 50% probability level.

The conformation of the molecule is obviously not appropriate for meridional coordination to a metal centre and rearrangements similar to those necessary for the tetradentate Schiff base ligands (Corden, Errington, Moore & Wallbridge, 1996) are required. No metal complexes of compound (I) are yet known, however, and more structural determinations on metal complexes containing this type of ligand are required before firm conclusions on the mode of coordination can be drawn.

Experimental

To a stirred solution of 2-hydroxyacetophenone (41.6 mmol) in methanol (100 ml) was added 4,5-dimethyl-1,2-phenyldiamine (41.6 mmol) in methanol (80 ml). The resultant solution was heated to reflux and then cooled to room temperature during which time a yellow crystalline solid precipitated.

Crystal data

$C_{16}H_{18}N_2O$
$M_r = 254.32$
Orthorhombic
$P2_12_12_1$
$a = 6.728$ (2) Å
$b = 7.940$ (7) Å
$c = 25.085$ (9) Å
$V = 1340.0$ (13) Å^3
$Z = 4$
$D_x = 1.261$ Mg m^{-3}
D_m not measured

Mo $K\alpha$ radiation
$\lambda = 0.71073$ Å
Cell parameters from 250 reflections
$\theta = 2.69–24.98°$
$\mu = 0.080$ mm^{-1}
$T = 150$ (2) K
Block
0.24 × 0.22 × 0.22 mm
Yellow

Data collection

Delft Instruments FAST TV area detector diffractometer
ω scans
Absorption correction: none
5958 measured reflections
2108 independent reflections

1822 observed reflections $[I > 2\sigma(I)]$
$R_{\rm int} = 0.0667$
$\theta_{\rm max} = 24.98°$
$h = -7 \rightarrow 7$
$k = -6 \rightarrow 8$
$l = -28 \rightarrow 27$
No standard reflections

Refinement

Refinement on F^2
$R(F) = 0.0382$
$wR(F^2) = 0.1122$
$S = 1.058$
2105 reflections
176 parameters
H atoms: see below
$w = 1/[\sigma^2(F_o^2) + (0.0371P)^2]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{\rm max} = -0.003$

$\Delta\rho_{\rm max} = 0.206$ e Å^{-3}
$\Delta\rho_{\rm min} = -0.164$ e Å^{-3}
Extinction correction: none
Atomic scattering factors from *International Tables for Crystallography* (1992, Vol. C, Tables 4.2.6.8 and 6.1.1.4)
Absolute configuration: Flack (1983)
Flack parameter = −0.4 (17)

Table 1. *Fractional atomic coordinates and equivalent isotropic displacement parameters (Å^2)*

$U_{\rm eq} = (1/3)\Sigma_i\Sigma_jU_{ij}a_i^*a_j^*\mathbf{a}_i.\mathbf{a}_j$.

	x	y	z	$U_{\rm eq}$
O1	0.3193 (2)	0.0326 (2)	0.33213 (5)	0.0337 (4)
N1	0.3484 (3)	0.2858 (2)	0.20153 (7)	0.0446 (5)
N2	0.0654 (2)	0.1399 (2)	0.26647 (6)	0.0248 (4)
C1	0.1780 (3)	0.2197 (2)	0.17864 (8)	0.0256 (5)
C2	0.1523 (3)	0.2140 (2)	0.12357 (7)	0.0270 (5)
C3	−0.0140 (3)	0.1425 (2)	0.09980 (7)	0.0259 (5)
C4	−0.1648 (3)	0.0780 (2)	0.13214 (8)	0.0273 (5)
C5	−0.1416 (3)	0.0847 (2)	0.18707 (8)	0.0255 (5)
C6	0.0260 (3)	0.1534 (2)	0.21100 (7)	0.0241 (5)
C7	−0.0590 (3)	0.1808 (2)	0.30385 (7)	0.0234 (4)
C8	−0.2532 (3)	0.2678 (2)	0.29427 (8)	0.0284 (5)
C9	0.0007 (3)	0.1424 (2)	0.35908 (7)	0.0245 (5)
C10	−0.1283 (3)	0.1740 (2)	0.40204 (8)	0.0320 (5)
C11	−0.0760 (3)	0.1368 (2)	0.45379 (8)	0.0363 (5)
C12	0.1083 (3)	0.0670 (2)	0.46441 (8)	0.0356 (6)

C13	0.2377 (3)	0.0333 (2)	0.42326 (8)	0.0324 (5)
C14	0.1868 (3)	0.0686 (2)	0.37086 (8)	0.0263 (5)
C15	−0.3485 (3)	−0.0004 (3)	0.10785 (8)	0.0367 (5)
C16	−0.0282 (3)	0.1318 (2)	0.04014 (7)	0.0336 (5)

Table 2. *Selected geometric parameters (Å, °)*

O1—C14	1.349 (2)	N2—C7	1.298 (2)
N1—C1	1.385 (3)	N2—C6	1.421 (2)
C7—N2—C6	124.6 (2)	N2—C7—C9	116.8 (2)
N1—C1—C2	121.7 (2)	N2—C7—C8	124.2 (2)
N1—C1—C6	120.2 (2)	O1—C14—C13	118.4 (2)
C5—C6—N2	123.0 (2)	O1—C14—C9	121.5 (2)
C1—C6—N2	117.2 (2)		
C6—N2—C7—C9	174.18 (15)	C8—C7—C9—C10	5.1 (2)
C6—N2—C7—C8	−7.7 (3)		

Data were collected from 0.2° ω-rotation exposures of 10 s each, with a crystal–detector distance of 49.37 (8) mm. Crystal decay was found to be negligible by comparison of intensities of repeated reflections. H atoms were added at calculated positions and refined using a riding model, including free rotation of methyl and hydroxy groups. Anisotropic displacement parameters were used for all non-H atoms and H atoms were given isotropic displacement parameters equal to 1.2 (or 1.5 for methyl H atoms) times the equivalent isotropic displacement parameters of their attached atoms.

Data collection: *MADNES* (Pflugrath & Messerschmidt, 1992). Cell refinement: *MADNES*. Data reduction: *SHELXTL-Plus* (Sheldrick, 1990). Program(s) used to solve structure: *SHELXTL-Plus*. Program(s) used to refine structure: *SHELXL*93 (Sheldrick, 1993). Molecular graphics: *SHELXTL-Plus*. Software used to prepare material for publication: *SHELXL*93.

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Lists of structure factors, anisotropic displacement parameters, H-atom coordinates and complete geometry have been deposited with the IUCr (Reference: CF1102). Copies may be obtained through The Managing Editor, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

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A Sulfonyl-Substituted Aziridine

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Abstract

In the title compound, 1-*p*-toluenesulfonylaziridine-(2*S*)-carboxylic acid *tert*-butyl ester, $C_{14}H_{19}NO_4S$, the *p*-toluenesulfonyl substituent on the N atom and the ester substituent on one C atom of the aziridine ring are mutually *trans*, minimizing steric interactions. The two N—C ring bond lengths [1.479 (2) and 1.484 (2) Å] are insignificantly different, consistent with the lack of regioselectivity in reactions with carbon nucleophiles.

Comment

The title compound (I) was originally prepared from the corresponding carboxylic acid, the esterification being achieved by treatment with *tert*-butyl 2,2,2-trichloroacetimidate in the presence of catalytic amounts of boron trifluoride–diethyl ether (Armstrong, Brackenridge, Jackson & Kirk, 1988). It was planned to explore its reactions with organometallic nucleophiles as a potential route to optically active α-amino acids but the results of such studies have been published recently by other researchers, together with details of the preparation and spectroscopic characterization of the compound (Baldwin, Spivey, Schofield & Sweeney, 1993). The compound has also been prepared by dicyclohexylcarbodiimide-mediated esterification of the carboxylic acid using *tert*-butanol (Church & Young, 1994). We report here the crystallographic confirmation of the structure (Fig. 1).