199 parameters
H atoms: riding and rotating model with $U_{\rm iso}$ refined for groups
$w = 1/[\sigma^2(F_o^2) + (0.0791P)^2 + 1.5737P]$ where $P = (F_o^2 + 2F_c^2)/3$
Extinction correction: none
Atomic scattering factors from *International Tables for Crystallography* (1992, Vol. C, Tables 4.2.6.8 and 6.1.1.4)

Table 3. *Fractional atomic coordinates and equivalent isotropic displacement parameters* (Å^2) *for* (2)

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

	x	y	z	$U_{\rm eq}$
Te1	0.04660 (4)	0.03535 (3)	0.76279 (3)	0.04207 (10)
I1	0.2362 (3)	0.1281 (3)	0.8664 (3)	0.0528 (5)
Br1	0.2023 (7)	0.1392 (7)	0.8806 (7)	0.0484 (10)
I2	−0.2112 (4)	0.2143 (3)	0.6845 (4)	0.0488 (3)
Br2	−0.2032 (10)	0.2339 (7)	0.6974 (9)	0.0464 (8)
S1	0.2269 (2)	−0.18068 (12)	0.7989 (2)	0.0497 (3)
S2	−0.02116 (15)	−0.14714 (13)	0.70432 (15)	0.0486 (3)
O1	0.4367 (5)	0.3439 (5)	0.1442 (4)	0.0676 (11)
N1	0.1525 (5)	−0.3857 (4)	0.7581 (4)	0.0426 (8)
C1	0.1226 (5)	−0.2552 (5)	0.7561 (5)	0.0399 (8)
C2	0.0623 (6)	−0.4507 (5)	0.7243 (6)	0.0492 (11)
C3	−0.0651 (7)	−0.5157 (7)	0.8526 (7)	0.0620 (14)
C4	0.2794 (6)	−0.4749 (5)	0.7929 (6)	0.0524 (11)
C5	0.4078 (8)	−0.4848 (10)	0.6640 (9)	0.083 (2)
C6	0.1879 (6)	0.1311 (5)	0.5539 (5)	0.0435 (9)
C7	0.1887 (6)	0.2761 (5)	0.5045 (5)	0.0469 (10)
C8	0.2744 (6)	0.3428 (5)	0.3689 (6)	0.0527 (11)
C9	0.3605 (6)	0.2656 (6)	0.2786 (6)	0.0506 (11)
C10	0.3622 (6)	0.1227 (6)	0.3279 (6)	0.0516 (11)
C11	0.2754 (6)	0.0544 (5)	0.4655 (6)	0.0487 (10)
C12	0.4960 (8)	0.2801 (9)	0.0375 (7)	0.077 (2)

Table 4. *Selected geometric parameters* (Å, °) *for* (2)

Te1—C6	2.123 (5)	Te1—I1	3.049 (3)
Te1—S1	2.5233 (15)	Te1—Br1$^{\rm i}$	3.676 (6)
Te1—S2	2.5349 (14)	Te1—I1$^{\rm i}$	3.872 (3)
Te1—Br1	2.967 (8)	S1—C1	1.726 (4)
Te1—Br2	2.962 (8)	S2—C1	1.737 (5)
Te1—I2	3.003 (4)		
C6—Te1—S1	92.63 (14)	S2—Te1—I2	84.78 (5)
C6—Te1—S2	94.87 (13)	C6—Te1—I1	87.84 (14)
S1—Te1—S2	71.27 (4)	S1—Te1—I1	80.04 (7)
C6—Te1—Br1	91.4 (2)	S2—Te1—I1	151.26 (7)
S1—Te1—Br1	84.62 (13)	I2—Te1—I1	123.92 (7)
S2—Te1—Br1	155.30 (13)	C6—Te1—Br1$^{\rm i}$	177.7 (2)
C6—Te1—Br2	89.0 (2)	C6—Te1—I1$^{\rm i}$	175.64 (14)
S1—Te1—Br2	162.24 (9)	Te1—I1—Te1$^{\rm i}$	84.52 (7)
S2—Te1—Br2	90.98 (9)	Te1—Br1—Te1$^{\rm i}$	89.2 (2)
Br1—Te1—Br2	113.03 (15)	C1—S1—Te1	86.2 (2)
C6—Te1—I2	88.7 (2)	C1—S2—Te1	85.6 (2)
S1—Te1—I2	156.04 (5)	S1—C1—S2	116.6 (3)
S2—Te1—S1—C1	−3.0 (2)	Br2—Te1—C6—C11	−126.5 (4)
S1—Te1—S2—C1	3.0 (2)	I2—Te1—C6—C11	−120.2 (4)
C4—N1—C1—S1	−1.9 (7)	I1—Te1—C6—C11	115.8 (4)
C2—N1—C1—S2	−3.3 (7)	S1—Te1—C6—C7	−145.8 (4)
Te1—S1—C1—S2	4.6 (3)	S2—Te1—C6—C7	142.8 (4)
Te1—S2—C1—S1	−4.6 (3)	Br1—Te1—C6—C7	−61.1 (4)
S1—Te1—C6—C11	35.9 (4)	Br2—Te1—C6—C7	51.9 (4)
S2—Te1—C6—C11	−35.6 (4)	I2—Te1—C6—C7	58.2 (4)
Br1—Te1—C6—C11	120.5 (4)	I1—Te1—C6—C7	−65.8 (4)

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

Both ethyl groups in one of the three independent molecules of (1) were disordered. For one of the ethyl groups the disorder was successfully resolved [C(4)/C(4′) and C(5)/C(5′) were refined isotropically with occupancy factors of 0.5 for each position], but for the other, C(2), C(3), it failed (anisotropic refinement gave very large amplitudes of the thermal motion of the atoms). An absorption correction for (2) was made assuming that the crystal contains an equimolar ratio of Br and I atoms. The positions of the non-stoichiometrically replaced Br atoms were located in a difference Fourier synthesis. The I/Br occupancy factors were refined at an early stage (isotropic for all non-H atoms) by a least-squares procedure using the restrictions $U_{\rm iso}({\rm Br}_j) = U_{\rm iso}({\rm I}_j)$, $g({\rm Br}_j) + g({\rm I}_j) = 1$ and $g({\rm Br1}) = g({\rm Br2})$, and then fixed.

For both compounds, program(s) used to solve structures: *SHELXS*86 (Sheldrick, 1985); program(s) used to refine structures: *SHELXL*93 (Sheldrick, 1993).

Lists of structure factors, anisotropic displacement parameters, H-atom coordinates and complete geometry have been deposited with the IUCr (Reference: FG1060). Copies may be obtained through The Managing Editor, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

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N-*p*-Tolylvanillaldimine, $C_{15}H_{15}NO_2$

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Abstract

The title compound, 2-methoxy-4-(*p*-tolyliminomethyl)-phenol, contains two phenyl rings bridged by a C=N imino moiety, the planes of which are inclined at an

angle of 40.26 (9)°, showing significant deviation of the molecule from planarity. The characteristic of aromatic Schiff bases with *N*-aryl substituents is that the terminal phenyl rings are twisted relative to the plane in which the C═N double bond lies. The hydroxy group is involved simultaneously in intramolecular and intermolecular three-centre hydrogen bonding with two acceptor atoms. Adjacent molecules are linked *via* an intermolecular O—H···N hydrogen bond [O1···N1 2.839 (3) Å, O1—H1···N1 154.9°] forming infinite chains along the *y* axis.

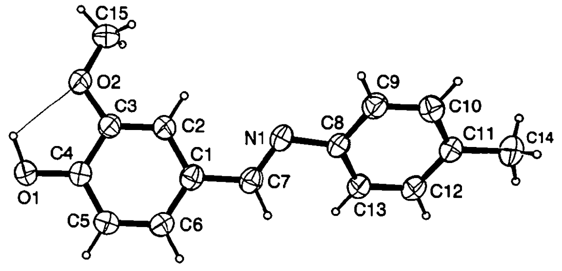


Fig. 1. *ORTEP*II (Johnson, 1976) drawing of the title compound with displacement ellipsoids shown at the 30% probability level. H atoms are drawn as unlabelled spheres of arbitrary radii.

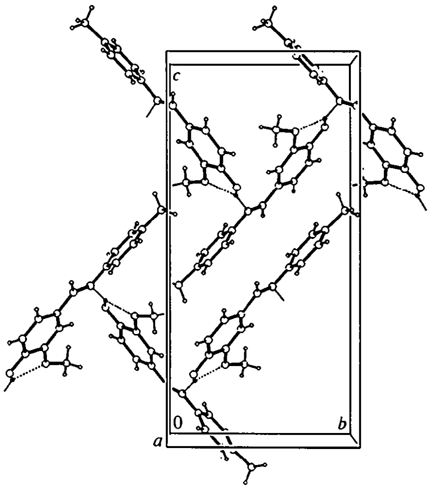
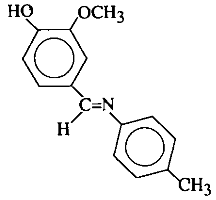


Fig. 2. Packing diagram (*PLUTON*92; Spek, 1992) of the title compound viewed down [100].

Comment

Preparation and structural investigation of N-substituted Schiff bases that can act as ligands with varying degrees of denticity forms part of our research on transition metal complexes. A search of the August 1994 release of the Cambridge Structural Database (Allen *et al.*, 1987) revealed that there are no reported structures of either a Schiff base derived from vanillin (4-hydroxy-3-methoxybenzaldehyde) or a metal complex with that monodentate base. Here we report the structure of the Schiff base *N*-*p*-tolylvanillaldimine, (I).



(I)

A perspective view of the molecule with the crystallographic atom-numbering scheme is displayed in Fig. 1. The packing arrangement showing the molecules linked by intermolecular hydrogen bonding is presented in Fig. 2.

The molecule may be described in terms of three planar subunits, namely two terminal benzene rings and their substituents [planar to within 0.05 Å, except for the methoxy atom C15 which is 0.392 (5) Å out of the C1–C6 plane] bridged by a central part consisting of N1, C1, C7 and C8, with the 4-hydroxy-3-methoxyphenyl and *p*-tolyl moieties *trans* to each other. The benzene rings of this aromatic Schiff base are buckled out of the plane in which the double bond of the N═C—H subunit lies (Bürgi & Dunitz, 1970). Such a conformation is confirmed by the torsion angles C2—C1—C7—N1 [2.4 (1)°] and C7—N1—C8—C13 [38.0 (2)°]. The twisting angles of the *p*-tolyl [35.4 (2)°] and the vanillidene [5.5 (2)°] rings with respect to the plane defined by the C—N═C—C subunit exhibit the general trend observed previously of aniline rings being more twisted than benzylidene rings (Nakai, Shiro, Ezumi, Sakata & Kubota, 1976; Nakai, Ezumi & Shiro, 1981; Bar & Bernstein, 1982; Baumeister, Hartung & Gdaniec, 1987; Ondráček, Kovářová, Maixner & Jursik, 1993).

The C1—C7 [1.463 (3) Å] and N1—C8 [1.424 (3) Å] distances confirm π-electron delocalization between the benzene rings, and the molecule can be regarded as a partially delocalized π-electron system. In benzylideneaniline, which is a non-substituted structural fragment of (I), the N—C_{ar}, C(sp^2)—C_{ar} and N═C(sp^2) bond lengths of the azomethine portion are 1.460 (3), 1.496 (3) and 1.237 (3) Å, respectively (Bürgi & Dunitz, 1970). If the terminal phenyl rings of benzylideneaniline are substituted by different substituents, the general pattern of two long and one short bond distance is not preserved. On the contrary, the shortening of N—C_{ar} and C(sp^2)—C_{ar} to 1.424 (3) and 1.463 (3) Å, respectively, and the lengthening of N═C(sp^2) to 1.277 (3) Å is observed in (I) and in similar structures (Nakai *et al.*, 1976; Nakai *et al.*, 1981; Bar & Bernstein, 1982; Baumeister *et al.*, 1987; Ondráček *et al.*, 1993). The C11—C14 bond distance of 1.506 (4) Å is in good agreement with C_{ar}—$C(sp^3)$ bond lengths. Using a 3σ criterion, the lengths of O1—C4 [1.367 (3) Å] and O2—C3 [1.360 (3) Å] are the same and fall into the range for the O—C_{ar} bond type. All other bond lengths and angles are within the expected ranges (Allen *et al.*, 1987).

The crystal structure is stabilized by intramolecular and intermolecular O—H···O and O—H···N hydrogen bonding and intermolecular C—H···O close contacts. An intramolecular O—H···O hydrogen bond is found between the hydroxy O1 and methoxy O2 atoms and there is an intermolecular O—H···N hydrogen bond linking the neighbouring molecules into infinite chains along the *y* axis (Table 3). A close C2—H···O1 contact of 3.390 (3) Å can be regarded as a weak electrostatic attraction (Steiner & Saenger, 1993).

Experimental

The title compound was prepared by the condensation reaction of vanillin (0.152 g) in 5 ml of MeOH and *p*-toluidine (0.107 g) in 5 ml of MeOH (1 eq. vanillin to 1 eq. *p*-toluidine). The reaction mixture, with a few drops of triethylamine added, was refluxed and stirred for 3 h. The excess of methanol was evaporated under reduced pressure, and from the dark red residue layered by ether, the diffraction-quality pale yellow crystals were obtained.

Crystal data

$C_{15}H_{15}NO_2$
$M_r = 241.29$
Monoclinic
$P2_1/c$
$a = 6.453$ (2) Å
$b = 9.997$ (2) Å
$c = 20.114$ (4) Å
$\beta = 90.38$ (1)°
$V = 1297.5$ (5) Å^3
$Z = 4$
$D_x = 1.235$ Mg m^{-3}
$D_m = 1.25$ Mg m^{-3}
D_m measured by flotation

Mo $K\alpha$ radiation
$\lambda = 0.7107$ Å
Cell parameters from 24 reflections
$\theta = 10.00$–19.00°
$\mu = 0.08$ mm^{-1}
$T = 293$ K
Crystal block
0.75 × 0.74 × 0.51 mm
Pale yellow

Data collection

Phillips PW1100 diffractometer
ω scans
Absorption correction: none
2919 measured reflections
2838 independent reflections
1596 observed reflections [$I > 2.0\sigma(I)$]

$R_{int} = 0.012$
$\theta_{max} = 26.96°$
$h = -8 \rightarrow 8$
$k = 0 \rightarrow 12$
$l = 0 \rightarrow 25$
3 standard reflections
frequency: 90 min
intensity decay: 2%

Refinement

Refinement on F
$R = 0.048$
$wR = 0.085$
$S = 1.27$
1596 reflections
163 parameters
H-atom parameters not refined
$w = 1/[\sigma^2(F) + 0.0040F^2]$

$(\Delta/\sigma)_{max} < 0.001$
$\Delta\rho_{max} = 0.25$ e Å^{-3}
$\Delta\rho_{min} = -0.17$ e Å^{-3}
Extinction correction: none
Atomic scattering factors from *International Tables for X-ray Crystallography* (1974, Vol. IV, Table 2.2B)

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

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

	x	y	z	U_{eq}
O1	0.2323 (2)	0.1312 (2)	0.1634 (1)	0.063 (1)
O2	−0.0717 (3)	0.3059 (2)	0.1833 (1)	0.084 (1)
N1	0.1520 (3)	0.5647 (2)	0.3977 (1)	0.055 (1)
C1	0.2714 (4)	0.3895 (2)	0.3247 (1)	0.056 (1)
C2	0.0991 (4)	0.3923 (2)	0.2825 (1)	0.060 (1)
C3	0.0857 (4)	0.3083 (2)	0.2286 (1)	0.057 (1)
C4	0.2437 (3)	0.2168 (2)	0.2162 (1)	0.052 (1)
C5	0.4146 (4)	0.2134 (3)	0.2574 (1)	0.066 (1)
C6	0.4285 (4)	0.2998 (3)	0.3111 (1)	0.065 (1)
C7	0.2913 (4)	0.4804 (2)	0.3814 (1)	0.058 (1)
C8	0.1930 (3)	0.6555 (2)	0.4507 (1)	0.053 (1)
C9	0.0276 (4)	0.6946 (3)	0.4898 (1)	0.067 (1)
C10	0.0552 (4)	0.7894 (3)	0.5391 (1)	0.072 (2)
C11	0.2440 (4)	0.8499 (3)	0.5500 (1)	0.064 (1)
C12	0.4086 (4)	0.8096 (3)	0.5110 (1)	0.070 (1)
C13	0.3844 (4)	0.7145 (3)	0.4617 (1)	0.063 (1)
C14	0.2692 (5)	0.9571 (3)	0.6020 (2)	0.091 (2)
C15	−0.2118 (4)	0.4140 (3)	0.1818 (2)	0.085 (2)

Table 2. *Selected bond lengths* (Å) *and angles* (°)

O1—C4	1.367 (3)	N1—C8	1.424 (3)
O2—C3	1.360 (3)	C1—C7	1.463 (3)
O2—C15	1.409 (3)	C11—C14	1.506 (4)
N1—C7	1.277 (3)	C(Ph)—$C(Ph)_{av}$	1.383 (4)
C3—O2—C15	118.5 (2)	O1—C4—C5	119.5 (2)
C7—N1—C8	119.1 (2)	N1—C7—C1	123.5 (2)
C2—C1—C7	121.8 (2)	N1—C8—C9	117.7 (2)
C6—C1—C7	119.8 (2)	N1—C8—C13	123.5 (2)
O2—C3—C2	125.7 (2)	C10—C11—C14	120.9 (2)
O2—C3—C4	114.4 (2)	C12—C11—C14	121.4 (3)
O1—C4—C3	120.9 (2)	C(Ph)—C(Ph)—$C(Ph)_{av}$	120.0 (2)

Table 3. *Hydrogen-bonding geometry* (Å, °)

D—H···A	D—H	H···A	D···A	D—H···A
O1—H1···O2	0.97	2.17	2.659 (3)	109.7
O1—H1···N1^{i}	0.97	1.93	2.839 (3)	154.9
C2—H2···O1^{ii}	0.95	2.46	3.390 (3)	165.0

Symmetry codes: (i) $-x, y - \frac{1}{2}, \frac{1}{2} - z$; (ii) $-x, \frac{1}{2} + y, \frac{1}{2} - z$.

The structure was solved by direct methods and refined by full-matrix least-squares methods. H atoms were positioned with appropriate orientations on geometric grounds (C—H 0.95 Å) and included in the structure-factor calculations. The hydroxyl H atom was found in the $\Delta\rho$ map calculated at the end of the refinement process as a small positive electron density. All calculations were performed on a personal computer.

Data collection: *DIF*4 (Stoe & Cie, 1992*a*). Cell refinement: *DIF*4. Data reduction: *REDU*4 (Stoe & Cie, 1992*b*). Program(s) used to solve structure: *NRCVAX SOLVER* (Gabe, Le Page, Charland, Lee & White, 1989). Program(s) used to refine structure: *NRCVAX LSTSQ*. Molecular graphics: *ORTEP*II (Johnson, 1976) and *PLUTON*92 (Spek, 1992). Software used to prepare material for publication: *NRCVAX TABLES*.

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Lists of structure factors, anisotropic displacement parameters, H-atom coordinates and complete geometry have been deposited with the IUCr (Reference: KA1112). Copies may be obtained through The Managing Editor, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

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N,*N'*-[Dithiobis(*o*-phenylene)]bis(2-hydroxy-1-naphthaldimine)

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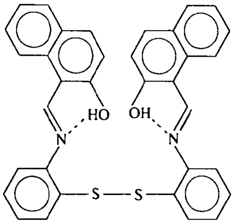
Abstract

The crystal structure of the title compound, 1,1'-[2,2'-dithiobis(phenyliminomethyl)]di-2-naphthol, $C_{34}H_{24}N_2O_2S_2$, has been determined. Two 2-hydroxy-2-mercaptophenyl-1-naphthaldimine moieties, which can act as tridentate ligands, are joined by the S atoms. The molecule is not planar and contains intramolecular O···N hydrogen bonds.

Comment

Schiff bases have been used extensively as ligands in the field of coordination chemistry. Some of the reasons are that the intramolecular hydrogen bond between the O and N atoms plays an important role in the formation of metal complexes, and that Schiff base compounds show photochromism and thermochromism in the solid state by proton transfer from the hydroxyl O atom to the imine N atom. Proton transfer may also be a basis for the development of molecular switches. The charge transport occurs through overlapping intermolecular π orbitals with proton transfer. Both mononuclear and binuclear complexes of Mn^{II} with the Schiff base disulfide ligand *N*,*N'*-[dithiobis(*o*-phenylene)]bis(salicylideneaminato) have been synthesized and characterized by electrochemical, magnetic, spectral and diffraction methods (Kessissoglou, Butler & Pecoraro, 1987). This paper reports the structure of the Schiff base disulfide ligand (I).



(I)

Two 2-hydroxy-2-mercaptophenyl-1-naphthaldimine moieties, *A* and *B* (Fig. 1), which can act as tridentate ligands, are bonded by the S atoms, forming a disulfide bridge. The S(1)—S(2) distance is 2.041 (2) Å; this is in good agreement with disulfide bonds reported for analogous structures (Bonds & Ibers, 1972; Riley & Seff, 1972; Kessissoglou, Butler & Pecoraro, 1987; Sellmann, Prechtel, Knoch & Moll, 1993). The most interesting feature of this structure is the significant twist of *A* relative to *B*. The orientation of *A* with respect to *B* is defined by the torsion angle between them [85.0 (2)°]. The planarity of *A* and *B* differs; in *A* the angle between the naphthalene and benzene moieties is 3.63°, whereas in *B* it is 28.16°.

Intramolecular hydrogen bonds occur between O(1) and N(1) [2.603 (7) Å] and O(2) and N(2) [2.512 (7) Å], the H atoms essentially being bonded to the O atoms. The O(1)—C(1) and O(2)—C(34) bond lengths [1.352 (7) and 1.373 (7) Å, respectively] are in the range observed for similar structures (Inabe, Hoshino, Mitani