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Key indicators

Single-crystal X-ray study
 $T = 296$ K
Mean $\sigma(\text{C}-\text{C}) = 0.006$ Å
 R factor = 0.068
 wR factor = 0.214
Data-to-parameter ratio = 14.1

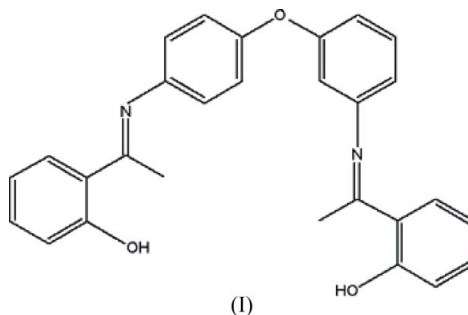
For details of how these key indicators were automatically derived from the article, see <http://journals.iucr.org/e>.

Bis{4-[2-(2-hydroxyphenyl)ethyleneamino]-phenyl} ether

In the title compound, $\text{C}_{28}\text{H}_{24}\text{N}_2\text{O}_3$, the two molecules in the asymmetric unit have different conformations about the ether bonds. The bond lengths and angles of the two molecules are nearly identical. The crystal structure is stabilized by intramolecular $\text{O}-\text{H}\cdots\text{N}$ and intermolecular $\text{C}-\text{H}\cdots\text{O}$ hydrogen bonding.

Comment

Schiff base compounds with dinuclear systems are of increasing interest. They can serve as paramagnetic building blocks for multidimensional expanded structures. Their important roles in biological systems, *e.g.* in many metalloenzymes, in redox and non-redox proteins, and as a catalyst in olefin epoxidation (Karmakar *et al.*, 2004), are documented. Schiff bases derived from 2-hydroxyacetophenone are important ligands for transition metal complexes, especially for Ni and Cu (Dietz *et al.*, 2000). They are also interesting in hydrogen-bonding studies (Abilgaard *et al.*, 2004). Ardakani *et al.* (2004) have studied the selective nitrate PVC membrane electrode from Schiff bases derived from 2-hydroxyacetophenone. A steric squeezing in *ortho*-hydroxy ketimines brings considerable shortening of the hydrogen bond (Filarowski *et al.*, 2002). Pronounced dynamics of a methyl group and its influence on the hydrogen bonding have been found in NMR spectra of deuterio derivatives of *o*-hydroxy ketones (Hansen *et al.*, 1994). *o*-Hydroxyacetophenones have been used for syntheses of compounds with antimycobacterial activity; these compounds were screened against *Mycobacterium tuberculosis* H37Rv using the Alamar Blue susceptibility test (Sriram *et al.*, 2005). Hydroxy Schiff bases have been synthesized from *o*-hydroxyacetophenone; their thermal decomposition and mass fragmentation at different temperatures were studied and compared. *o*-Hydroxyacetophenone Schiff bases decompose through benzimidazole derivative intermediates (Saeed *et al.*, 1986).



The two independent molecules, (IA) and (IB) of the title compound, (I), are shown in Fig. 1. The two molecules of the

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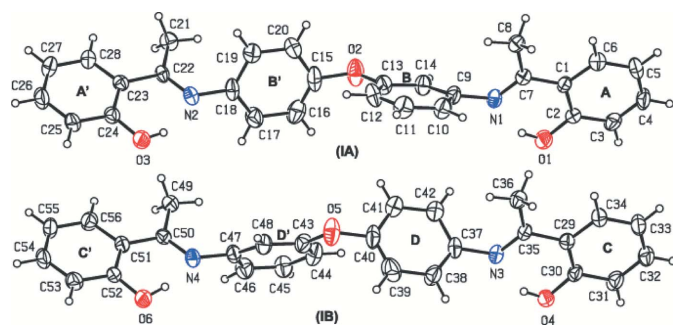


Figure 1

An ORTEP-3 view of the asymmetric unit of (I) with the atom-numbering scheme and 30% probability displacement ellipsoids.

asymmetric unit have nearly identical bond lengths and angles; the chemically analogous groups within each molecule also reveal close values of geometrical parameters (Table 1). In the solid state, molecules of (I) exist in a non-planar conformation. In the two molecules, the conformations about the ether bonds (involving O2 and O5) are different [for (IA), C15–O2–C13–C12 = $-12.9(7)^\circ$, and for (IB), C43–O5–C40–C39 = $-96.4(6)^\circ$]. In molecule (IA), the dihedral angle between planes A (C1–C6) and B (C9–C14) is $67.9(2)^\circ$, and the dihedral angle between the analogous planes A' (C23–C28) and B' (C15–C20) is $63.7(2)^\circ$. In molecule (IB), the corresponding angles are in good agreement with those of (IA) [the dihedral angle between the planes C (C1–C6) and D (C9–C14) is $64.5(2)^\circ$; that between the planes C' (C9–C14) and D' (C23–C28) is $67.7(2)^\circ$].

In the crystal structure, there are intramolecular O–H...N and intermolecular C–H...O hydrogen bonds connecting molecules along the *b* axis. (Table 2).

Experimental

2-Hydroxyacetophenone (0.272 g, 0.24 ml, 2 mmol) and 3,4'-diaminodiphenylether (0.33 g, 1 mmol) were dissolved in warm ethanol (10 ml). The reaction mixture was refluxed for 10 h and allowed to stand aside. Yellow crystals were filtered off and washed with ethanol. The pure Schiff base was recrystallized as plate-like colourless crystals from ethanol (yield 78%). M.p. 455–457 K. IR (KBr, ν , cm^{-1}): 3244 (OH), 1620 (C=N). ^1H NMR (250 MHz, CDCl_3): 1.65 (6H, *s*, CH_3), 6.21 (2H, *d*, Ar), 6.24 (2H, *d*, Ar), 6.88–7.64 (5H, *m*, Ar), 7.94 (2H, *d*, Ar), 14.57 (2H, *s*, OH). ^{13}C NMR (62.9 MHz, CDCl_3): 17.11, 17.22, 30.91, 111.26, 114.56, 115.99, 118.13, 118.23, 119.94, 122.82, 128.93, 130.25, 133.07, 133.20, 142.73, 145.60, 146.60, 148.63, 153.73, 158.29, 161.98, 171.60 (C=N).

Crystal data

$\text{C}_{28}\text{H}_{24}\text{N}_2\text{O}_3$
 $M_r = 436.49$
 Triclinic, $P\bar{1}$
 $a = 9.4897(8) \text{ \AA}$
 $b = 10.4644(8) \text{ \AA}$
 $c = 22.8605(19) \text{ \AA}$
 $\alpha = 96.588(6)^\circ$
 $\beta = 98.781(7)^\circ$
 $\gamma = 89.957(6)^\circ$

$V = 2228.4(3) \text{ \AA}^3$
 $Z = 4$
 $D_x = 1.301 \text{ Mg m}^{-3}$
 Mo $K\alpha$ radiation
 $\mu = 0.09 \text{ mm}^{-1}$
 $T = 296 \text{ K}$
 Plate, colourless
 $0.35 \times 0.26 \times 0.08 \text{ mm}$

Data collection

Stoe IPDS-2 diffractometer
 ω scans
 Absorption correction: integration
 (*X-RED32*; Stoe & Cie, 2002)
 $T_{\text{min}} = 0.971$, $T_{\text{max}} = 0.993$

27310 measured reflections
 8425 independent reflections
 3164 reflections with $I > 2\sigma(I)$
 $R_{\text{int}} = 0.118$
 $\theta_{\text{max}} = 26.0^\circ$

Refinement

Refinement on F^2
 $R[F^2 > 2\sigma(F^2)] = 0.068$
 $wR(F^2) = 0.214$
 $S = 0.89$
 8425 reflections
 596 parameters

H-atom parameters constrained
 $w = 1/[\sigma^2(F_o^2) + (0.0985P)^2]$
 where $P = (F_o^2 + 2F_c^2)/3$
 $(\Delta/\sigma)_{\text{max}} < 0.001$
 $\Delta\rho_{\text{max}} = 0.51 \text{ e \AA}^{-3}$
 $\Delta\rho_{\text{min}} = -0.23 \text{ e \AA}^{-3}$

Table 1

Selected geometric parameters (\AA , $^\circ$).

O1–C2	1.345 (5)	N1–C7	1.291 (5)
O2–C13	1.390 (6)	N1–C9	1.426 (5)
O2–C15	1.393 (5)	N2–C18	1.405 (5)
O3–C24	1.349 (5)	N2–C22	1.295 (5)
O4–C30	1.360 (5)	N3–C37	1.412 (5)
O5–C40	1.407 (5)	N3–C35	1.293 (5)
O5–C43	1.396 (6)	N4–C50	1.288 (5)
O6–C52	1.347 (5)	N4–C47	1.427 (5)
C13–O2–C15	118.5 (4)	N2–C22–C23	117.3 (4)
C40–O5–C43	118.9 (4)	O3–C24–C23	121.3 (3)
C7–N1–C9	122.8 (3)	O3–C24–C25	118.5 (4)
C18–N2–C22	122.7 (3)	O4–C30–C31	116.7 (4)
C35–N3–C37	122.1 (3)	O4–C30–C29	122.8 (3)
C47–N4–C50	122.6 (3)	N3–C35–C36	124.5 (3)
O1–C2–C3	119.1 (4)	N3–C35–C29	116.6 (3)
O1–C2–C1	121.0 (3)	N3–C37–C42	119.3 (4)
N1–C7–C8	123.2 (3)	N3–C37–C38	122.2 (4)
N1–C7–C1	117.9 (4)	O5–C40–C41	119.6 (5)
N1–C9–C10	119.4 (4)	O5–C40–C39	118.3 (5)
N1–C9–C14	122.3 (4)	O5–C43–C44	122.6 (4)
O2–C13–C14	115.3 (4)	O5–C43–C48	115.6 (4)
O2–C13–C12	123.7 (4)	N4–C47–C48	122.3 (4)
O2–C15–C20	118.2 (5)	N4–C47–C46	118.8 (4)
O2–C15–C16	120.6 (5)	N4–C50–C51	118.5 (4)
N2–C18–C17	121.7 (4)	N4–C50–C49	122.9 (3)
N2–C18–C19	119.6 (4)	O6–C52–C51	122.4 (3)
N2–C22–C21	123.7 (3)	O6–C52–C53	117.7 (4)

Table 2

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

<i>D</i> –H... <i>A</i>	<i>D</i> –H	H... <i>A</i>	<i>D</i> ... <i>A</i>	<i>D</i> –H... <i>A</i>
O1–H1...N1	0.82	1.82	2.546 (4)	148
O3–H3A...N2	0.82	1.80	2.531 (4)	148
O4–H4A...N3	0.82	1.81	2.536 (4)	146
O6–H6A...N4	0.82	1.93	2.556 (4)	133
C8–H8B...O3 ⁱ	0.96	2.63	3.313 (5)	129
C21–H21B...O1 ⁱⁱ	0.96	2.59	3.254 (5)	126
C36–H36C...O6 ⁱⁱⁱ	0.96	2.58	3.241 (5)	126
C49–H49B...O4 ^{iv}	0.96	2.61	3.301 (5)	129

Symmetry codes: (i) $-x, -y + 1, -z + 1$; (ii) $-x + 1, -y + 1, -z + 1$; (iii) $-x, -y + 2, -z + 1$; (iv) $-x + 1, -y + 2, -z + 1$.

All H atoms were placed in calculated positions (C–H = 0.93–0.96 \AA and O–H = 0.82 \AA) and included in the refinement in the riding mode, with U_{iso} constrained to be $1.2U_{\text{eq}}$ of the carrier C atom ($1.5U_{\text{eq}}$ for methyl and hydroxy H atoms).

Data collection: *X-AREA* (Stoe & Cie, 2002); cell refinement: *X-AREA*; data reduction: *X-RED32* (Stoe & Cie, 2002); program(s) used to solve structure: *SHELXS97* (Sheldrick, 1997); 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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