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Key indicators

Single-crystal X-ray study

T = 150 K

Mean $\sigma(C-C)$ = 0.003 Å

Disorder in main residue

R factor = 0.058

wR factor = 0.174

Data-to-parameter ratio = 12.6

For details of how these key indicators were
automatically derived from the article, see
<http://journals.iucr.org/e>.**(E)-2-[1,3-Dihydroxy-2-(hydroxymethyl)propan-2-ylaminomethylene]-4-[(4-ethylphenyl)diazenyl]-6-methoxycyclohexanone**

The title compound, $C_{20}H_{25}N_3O_5$, adopts the keto–amine tautomeric form and displays the characteristic features of azobenzene derivatives. Intramolecular $N-H \cdots O$ and $O-H \cdots O$ and intermolecular $O-H \cdots O$ interactions influence the conformation of the molecules and the crystal packing.

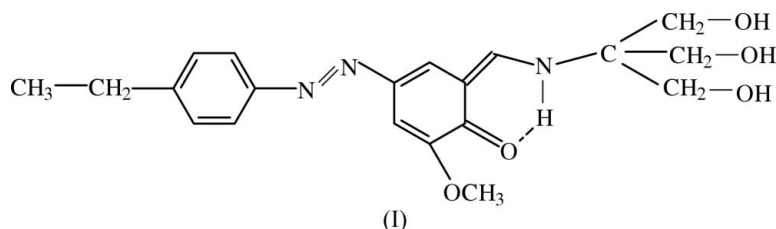
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Comment

Azo compounds are the most widely used class of dyes, owing to their versatile application in various fields, such as the dyeing of textiles and fibres, the colouring of different materials, and high-technology areas, such as electro-optical devices and ink-jet printers (Peters & Freeman, 1991). There is interest in Schiff base ligands and their complexes with regard to their impressive antitumour activities (Zhou *et al.*, 2000). Schiff base compounds can be classified by their photochromic and thermochromic characteristics (Cohen *et al.*, 1964; Moustakali *et al.*, 1978; Hadjoudis *et al.*, 1987). On the basis of studies of some thermochromic and photochromic Schiff base compounds, it has been proposed that molecules exhibiting thermochromism are planar, while those exhibiting photochromism are non-planar (Moustakali *et al.*, 1978).



o-Hydroxyarylidene Schiff bases display two possible tautomeric forms, namely, phenol–imine and keto–amine. In the solid state, the keto–amine tautomer has been found in naphthaldimine (Hökelek *et al.*, 2000; Ünver *et al.*, 2002), while the phenol–imine tautomer is found in salicylaldimine Schiff bases (Yıldız *et al.*, 1998; Elmalı & Elerman, 1998; Dey *et al.*, 2001; Yang & Vittal, 2003; Karadayı *et al.*, 2003).

The molecular structure of (I), with the atom-labelling scheme, is shown in Fig. 1. The $N=N$ double bond length [1.232 (3) Å] is comparable to that [1.223 (7) Å] in the azo compound 2-hydroxy-5-[[4-(2-pyridinylamino)sulfonyl]phenyl]azobenzoic acid (van der Sluis *et al.*, 1990). In the molecule of (I), the $C12=O2$, $N2-C9$ and $C11-C16$ bond lengths (Table 1) agree with the corresponding distances in 4-[(3-chlorophenyl)diazenyl]-2-[[tris(hydroxymethyl)methyl]aminomethylene]cyclohexa-3,5-dien-1(2*H*)-one [1.286 (2), 1.414 (2) and 1.411 (3) Å, respectively; Odabaşoğlu *et al.*, 2003].

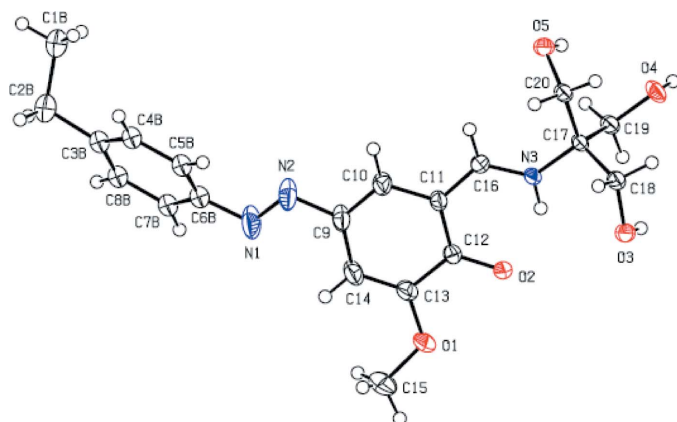


Figure 1
A view of (I), with the atom-numbering scheme and 50% probability displacement ellipsoids. Only the major disorder component is shown.

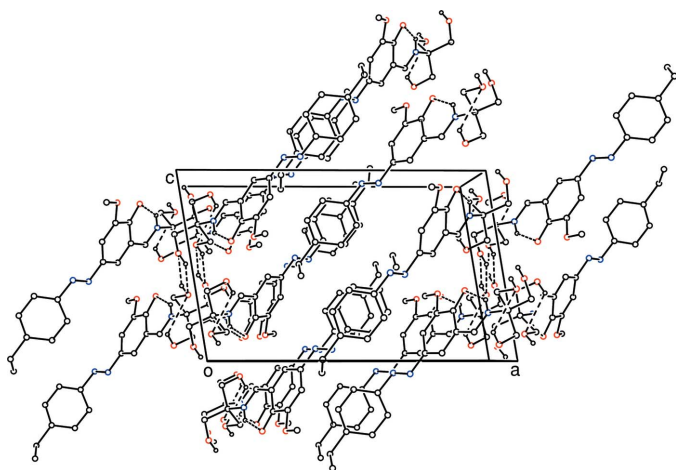


Figure 2
A packing diagram for (I), showing hydrogen bonds as dashed lines, with the major disorder component. Non-hydrogen-bonding H atoms have been omitted for clarity.

The intra- and intermolecular hydrogen bonding in (I) is shown in Fig. 2 and the geometric values are given in Table 2. Atom H3 bonded to N3 forms a strong intramolecular hydrogen bond with atom O2, with an N...O distance of 2.603 (2) Å, as in our previous work (Odabaşoğlu, *et al.*, 2003).

Experimental

The title compound, (I), was prepared as described by Odabaşoğlu *et al.* (2003), using *o*-vanillin, 4-ethylaniline and tris(hydroxymethyl)aminomethane as starting materials. The product was recrystallized from ethanol and well shaped crystals of (I) were obtained by slow evaporation of an ethanol solution (yield 80%, m.p. 453–455 K).

Crystal data

$C_{20}H_{25}N_3O_5$	$D_x = 1.345 \text{ Mg m}^{-3}$
$M_r = 387.43$	Mo $K\alpha$ radiation
Monoclinic, $P2_1/c$	Cell parameters from 14646 reflections
$a = 17.0925 (16) \text{ \AA}$	$\theta = 1.9\text{--}28.8^\circ$
$b = 10.6353 (6) \text{ \AA}$	$\mu = 0.10 \text{ mm}^{-1}$
$c = 10.6634 (9) \text{ \AA}$	$T = 150 \text{ K}$
$\beta = 99.263 (7)^\circ$	Prism, yellow
$V = 1913.2 (3) \text{ \AA}^3$	$0.42 \times 0.29 \times 0.15 \text{ mm}$
$Z = 4$	

Data collection

Stoe IPDS-II diffractometer
 ω scans
 Absorption correction: none
 16869 measured reflections
 3769 independent reflections
 2603 reflections with $I > 2\sigma(I)$

$R_{\text{int}} = 0.052$
 $\theta_{\text{max}} = 26.0^\circ$
 $h = -21 \rightarrow 21$
 $k = -13 \rightarrow 13$
 $l = -12 \rightarrow 13$

Refinement

Refinement on F^2
 $R[F^2 > 2\sigma(F^2)] = 0.058$
 $wR(F^2) = 0.174$
 $S = 1.05$
 3769 reflections
 299 parameters
 H-atom parameters constrained

$w = 1/[\sigma^2(F_o^2) + (0.1061P)^2 + 0.2543P]$
 where $P = (F_o^2 + 2F_c^2)/3$
 $(\Delta/\sigma)_{\text{max}} < 0.001$
 $\Delta\rho_{\text{max}} = 0.58 \text{ e \AA}^{-3}$
 $\Delta\rho_{\text{min}} = -0.61 \text{ e \AA}^{-3}$

Table 1

Selected geometric parameters (Å, °).

C15—O1	1.422 (3)	C13—O1	1.355 (3)
C9—N2	1.410 (3)	C16—N3	1.304 (3)
C11—C16	1.413 (3)	C18—O3	1.424 (3)
C11—C12	1.424 (3)	C19—O4	1.420 (3)
C12—O2	1.283 (3)	C20—O5	1.428 (3)
C12—C13	1.439 (3)	N1—N2	1.232 (3)
O2—C12—C11	122.5 (2)	N1—N2—C9	116.5 (2)
O2—C12—C13	120.8 (2)	C16—N3—C17	128.61 (19)
N3—C16—C11	122.7 (2)		

Table 2

Hydrogen-bond geometry (Å, °).

$D\text{—}H\cdots A$	$D\text{—}H$	$H\cdots A$	$D\cdots A$	$D\text{—}H\cdots A$
N3—H3...O2	0.86	1.90	2.603 (2)	138
O5—H5...O4	0.82	2.55	3.062 (2)	121
O3—H3A...O4 ⁱ	0.82	1.97	2.733 (2)	153
O4—H4...O3 ⁱ	0.82	1.99	2.733 (2)	150

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

The large s.u. values and displacement parameters of some atoms in the molecule are likely caused by some disorder of the 4-ethylphenyl group. This disorder was modelled as three different orientations (C1A—C8A, C1B—C8B and C1C—C8C) with occupancy factors of 0.329 (3), 0.351 (3) and 0.320 (4), respectively. An H atom is located on N3 rather than on O2, thus confirming a preference for the keto-amine tautomer in the solid state. All H atoms were refined using a riding model, with C—H = 0.93–0.97 Å, N—H = 0.86 Å and O—H = 0.82 Å. The constraint $U_{\text{iso}}(\text{H}) = 1.2U_{\text{eq}}(\text{C})$ or $1.5U_{\text{eq}}(\text{methyl C and hydroxyl O})$ was applied.

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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