

**(Z)-4-[(E)-(4-Butylphenyl)diazenyl]-6-[[1,3-dihydroxy-2-(hydroxymethyl)propan-2-ylamino]methylene]-2-methoxycyclohexa-2,4-dienone****Cem Cüneyt Ersanlı,<sup>a\*</sup> Çiğdem Albayrak,<sup>b</sup> Mustafa Odabaşoğlu<sup>b</sup> and Orhan Büyükgüngör<sup>a</sup>**<sup>a</sup>Department of Physics, Faculty of Arts and Sciences, Ondokuz Mayıs University, 55139 Kurupelit Samsun, Turkey, and <sup>b</sup>Department of Chemistry, Faculty of Arts and Sciences, Ondokuz Mayıs University, 55139 Kurupelit Samsun, Turkey

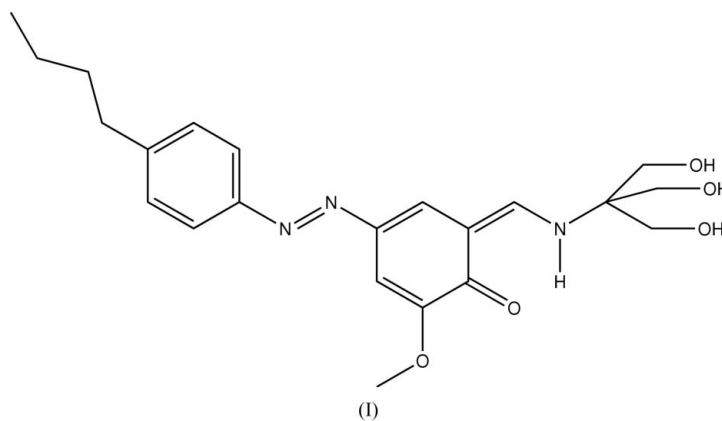
Correspondence e-mail: ccersan@omu.edu.tr

**Key indicators**Single-crystal X-ray study  
 $T = 296$  K  
Mean  $\sigma(\text{C}-\text{C}) = 0.007$  Å  
Disorder in main residue  
 $R$  factor = 0.083  
 $wR$  factor = 0.250  
Data-to-parameter ratio = 16.6For details of how these key indicators were automatically derived from the article, see <http://journals.iucr.org/e>.

The title compound,  $\text{C}_{22}\text{H}_{29}\text{N}_3\text{O}_5$ , is approximately planar with the aromatic rings in a *trans* configuration with respect to the azo double bond. The enamine portion of the molecule exists as the keto–amine tautomer, stabilized by a strong intramolecular  $\text{N}-\text{H}\cdots\text{O}$  hydrogen bond. Intramolecular  $\text{N}-\text{H}\cdots\text{O}$  and  $\text{O}-\text{H}\cdots\text{O}$  and intermolecular  $\text{O}-\text{H}\cdots\text{O}$  and  $\text{C}-\text{H}\cdots\text{O}$  interactions influence the conformation of the molecules and the crystal packing.

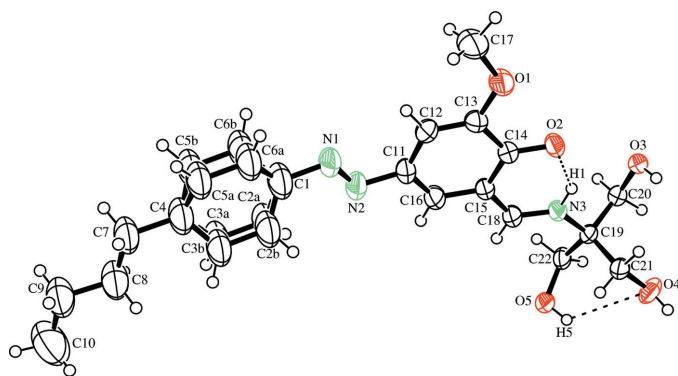
**Comment**

Azo compounds are the most widely used class of dyes, owing to their versatile application in various fields, such as dyeing and colouring, 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).

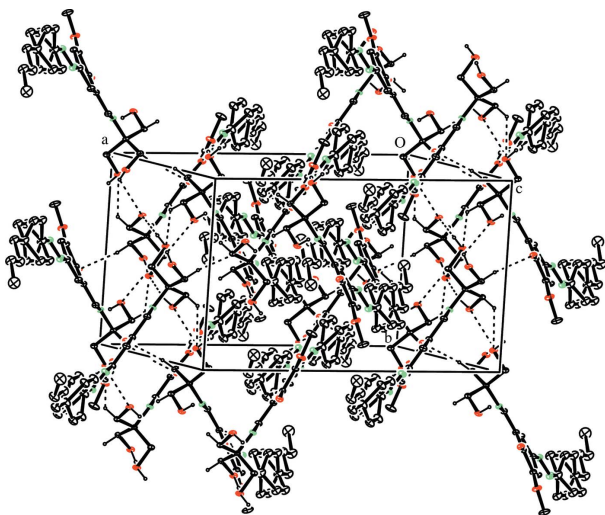


We report here the structure of the Schiff base compound, (I) (Fig. 1 and Table 1). There are two possible tautomers that this molecule could adopt, *viz.* the keto–amine and enol–imine tautomeric forms. The structural data clearly show that (I) adopts the keto–amine form, stabilized by the formation of an  $\text{N}-\text{H}\cdots\text{O}$  hydrogen bond. Similar *o*-hydroxy Schiff bases have been found to adopt the keto (Ersanlı *et al.*, 2003; Ersanlı, Albayrak, Odabaşoğlu, Thöne & Erdönmez, 2004; Ersanlı *et al.*, 2005), enol (Ersanlı, Albayrak, Odabaşoğlu & Erdönmez, 2004; Ersanlı, Odabaşoğlu *et al.*, 2004) or enol/keto

Received 1 November 2005  
Accepted 11 November 2005  
Online 16 November 2005



**Figure 1**  
A view of (I), with the atom-numbering scheme and 50% probability displacement ellipsoids. There is orientational disorder in the C1–C6 benzene ring; occupancy factors for atoms C2a/C3a/C5a/C6a and C2b/C3b/C5b/C6b are 0.617 (6) and 0.383 (6), respectively. Hydrogen bonds are shown as dashed lines.



**Figure 2**  
Packing diagram for (I), with hydrogen bonds drawn as dashed lines. For the sake of clarity, all H atoms, except for H3, H4, H5, H20B and H22B, have been omitted.

forms (Nazır *et al.*, 2000). The C14–O2 and C18–N3 bond distances (Table 1) are reasonably similar, a characteristic of systems in the keto–amine form, and comparable to those found in 4-[(2-chlorophenyl)diazenyl]-6-methoxy-2-[[tris-(hydroxymethyl)methyl]aminomethylene]cyclohexa-3,5-dien-1(2H)-one [1.287 (2) and 1.294 (2) Å; Ersanlı, Albayrak, Odabaşoğlu, Thöne & Erdönmez, 2004]. In contrast, the corresponding distances are significantly different in systems known to adopt the enol-imine tautomeric form, with a much longer C–O bond, for example, 1.354 (2) and 1.275 (2) Å in 2-[2-(hydroxymethyl)phenyliminomethyl]phenol (Ersanlı, Odabaşoğlu *et al.*, 2004), and 1.355 (2) and 1.278 (2) Å in 4-(2-hydroxyphenyliminomethyl)phenol (Ersanlı, Albayrak, Odabaşoğlu & Erdönmez, 2004).

Intramolecular N3–H1···O2 and C22–O5–H5···O4 hydrogen bonds influence the conformation of the molecule (Table 2). The packing is also stabilized by a number of intermolecular O–H···O and C–H···O interactions (Fig. 2).

## Experimental

The title compound, (I), was prepared as previously described (Ersanlı, Albayrak, Odabaşoğlu, Thöne & Erdönmez, 2004). The product was recrystallized from ethanol and well shaped crystals were obtained by slow evaporation of an acetonitrile solution (m.p. 461–463 K).

### Crystal data

C<sub>22</sub>H<sub>29</sub>N<sub>3</sub>O<sub>5</sub>  
*M<sub>r</sub>* = 415.48  
 Monoclinic, *P*<sub>2</sub><sub>1</sub>/*c*  
*a* = 18.3934 (13) Å  
*b* = 10.6960 (7) Å  
*c* = 10.8516 (8) Å  
 $\beta$  = 95.488 (6)°  
*V* = 2125.1 (3) Å<sup>3</sup>  
*Z* = 4

*D<sub>x</sub>* = 1.299 Mg m<sup>−3</sup>  
 Mo *K*α radiation  
 Cell parameters from 17039 reflections  
 $\theta$  = 1.9–27.9°  
 $\mu$  = 0.09 mm<sup>−1</sup>  
*T* = 296 (2) K  
 Plate, orange  
 0.30 × 0.20 × 0.05 mm

### Data collection

Stoe IPDS-II diffractometer  
 $\omega$  scans  
 Absorption correction: integration  
 (*X-RED32*; Stoe & Cie, 2002)  
*T<sub>min</sub>* = 0.975, *T<sub>max</sub>* = 0.996  
 23762 measured reflections  
 4173 independent reflections

2194 reflections with *I* > 2σ(*I*)  
*R<sub>int</sub>* = 0.097  
 $\theta_{\max}$  = 26.0°  
*h* = −22 → 22  
*k* = −13 → 13  
*l* = −13 → 13

### Refinement

Refinement on *F*<sup>2</sup>  
*R* [*F*<sup>2</sup> > 2σ(*F*<sup>2</sup>)] = 0.083  
*wR* (*F*<sup>2</sup>) = 0.250  
*S* = 1.02  
 4173 reflections  
 252 parameters

H atoms treated by a mixture of independent and constrained refinement  
 $w = 1/[\sigma^2(F_o^2) + (0.1495P)^2]$   
 where  $P = (F_o^2 + 2F_c^2)/3$   
 $(\Delta/\sigma)_{\max} = 0.001$   
 $\Delta\rho_{\max} = 0.52 \text{ e } \text{Å}^{-3}$   
 $\Delta\rho_{\min} = -0.52 \text{ e } \text{Å}^{-3}$

**Table 1**

Selected geometric parameters (Å, °).

C1–N1	1.427 (7)	C18–N3	1.289 (5)
C4–C7	1.525 (8)	C20–O3	1.422 (5)
C11–N2	1.406 (6)	C21–O4	1.416 (5)
C13–O1	1.363 (5)	C22–O5	1.416 (5)
C14–O2	1.276 (5)	N1–N2	1.238 (5)
C17–O1	1.423 (6)		
C16–C11–N2	116.6 (4)	O2–C14–C13	120.3 (3)
N2–C11–C12	123.9 (4)	N3–C18–C15	122.7 (4)
C12–C13–O1	125.9 (4)	N1–N2–C11	115.5 (4)
O2–C14–C15	123.2 (3)	C18–N3–C19	129.2 (3)
C1–N1–N2–C11	−175.6 (5)		

**Table 2**

Hydrogen-bond geometry (Å, °).

<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>
O4–H4···O3 <sup>i</sup>	0.82	2.03	2.769 (4)	150
O3–H3···O4 <sup>i</sup>	0.82	2.00	2.769 (4)	155
O5–H5···O3 <sup>ii</sup>	0.82	2.37	2.771 (3)	111
C20–H20B···O4 <sup>iii</sup>	0.97	2.55	3.310 (5)	135
C22–H22B···O2 <sup>ii</sup>	0.97	2.42	3.375 (5)	169
N3–H1···O2	0.88 (5)	1.88 (5)	2.603 (4)	138 (4)
O5–H5···O4	0.82	2.56	3.088 (4)	124

Symmetry codes: (i)  $-x + 2, -y, -z + 2$ ; (ii)  $-x + 2, y - \frac{1}{2}, -z + \frac{3}{2}$ ; (iii)  $-x + 2, y + \frac{1}{2}, -z + \frac{3}{2}$ .

The crystals were weakly diffracting but attempts to find one of better quality were not successful. Furthermore, high displacement parameters for atoms in the benzene ring suggested possible disorder. This was resolved by refining alternative positions for the unsubstituted atoms C2, C3, C5 and C6 of the benzene ring. Their occupancies refined to 0.617 (6) and 0.383 (6), respectively. All H atoms bound to C atoms were refined using a riding model [C–H = 0.93 Å and  $U_{\text{iso}}(\text{H}) = 1.2U_{\text{eq}}(\text{C})$  for aromatic C atoms, C–H = 0.97 Å and  $U_{\text{iso}}(\text{H}) = 1.2U_{\text{eq}}(\text{C})$  for methylene C atoms, and C–H = 0.96 Å and  $U_{\text{iso}}(\text{H}) = 1.5U_{\text{eq}}(\text{C})$  for methyl C atoms]. The H atoms of the hydroxy O atoms were refined with O–H constrained to 0.82 Å and with  $U_{\text{iso}}(\text{H}) = 1.5U_{\text{eq}}(\text{O})$ . The H atom bonded to N was refined freely, with  $U_{\text{iso}}(\text{H}) = 1.2U_{\text{eq}}(\text{N})$ .

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).

The authors acknowledge the Faculty of Arts and Sciences, Ondokuz Mayıs University, Turkey, for the use of the Stoe IPDS-II diffractometer (purchased under grant No. F279 of the University Research Fund).

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