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4-[(4-Methylphenyl)diazenyl]-6-methoxy-2-{[tris(hydroxymethyl)methyl]amino-methylene}cyclohexa-3,5-dien-1(2*H*)-one

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The title compound, $C_{19}H_{23}N_3O_5$, adopts the keto–amine tautomeric form and the configuration around the —N═N— double bond is *trans*. There is an intramolecular N—H···O hydrogen bond and intermolecular O—H···O interactions, forming a three-dimensional network.

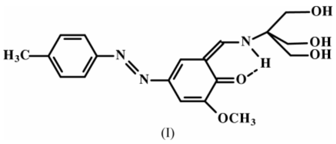
Key indicators

Single-crystal X-ray study
T = 293 K
Mean σ(C–C) = 0.007 Å
Disorder in main residue
R factor = 0.071
wR factor = 0.173
Data-to-parameter ratio = 12.1

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

Comment

The azo compound class accounts for 60–70% of all dyes. Azo dyes give bright strong colours. They are widely used in textile, printing, paper manufacturing, pharmaceutical and food industries. All of them contain at least one azo group, —N═N—, which links two sp^2-hybridized C atoms. In our ongoing work, these C atoms are part of aromatic systems. There is considerable interest in Schiff base ligands and their complexes in respect of their striking antitumour activities (Zhou *et al.*, 2000). *N*-Substituted *o*-hydroxyimines have been reported to display thermochromism and photochromism in the solid state by proton transfer from the hydroxy O atom to the N atom (Hadjoudis *et al.*, 1987; Xu *et al.*, 1994).



(I)

An *ORTEP*-3 (Farrugia, 1997) view of the molecule is shown in Fig. 1. The —N═N— double-bond length is 1.247 (5) Å, which is longer than in the azo compound 2-hydroxy-5-([4-(2-pyridinylamino)sulfonyl]phenyl)azobenzoic acid (van der Sluis & Spek, 1990) [1.223 (7) Å], which has no intramolecular hydrogen bond. The high s.u. values and high displacement parameters of some atoms in the molecule are likely caused by some disorder. Atom C18 of the methyl group shows positional disorder, with occupancy factors of 0.550 (5) and 0.450 (5). The phenyl rings show also orientational disorder with the same occupancy factors (Fig. 1). The molecule exists as the keto–amine tautomer. Some special bond lengths are C10—O1 = 1.279 (5) Å and C11—C12 = 1.340 (6) Å. The corresponding bond lengths in *N*-(2-fluoro-3-methoxy)salicylaldimine, which exists in the phenol–imine tautomeric form, are 1.347 (3) and 1.374 (3) Å, respectively (Ünver *et al.*, 2002). An H atom was located on N3 rather than on O1, thus confirming a preference for the keto–amine tautomer in the solid state. There is a strong intramolecular N3—H1···O1 hydrogen bond, which is a common feature of *o*-hydroxysalicylidene systems (Odabaşoğlu, Albayrak, Büyükgüngör & Goesmann, 2003; Odabaşoğlu, Albayrak, Büyükgüngör & Lönnecke, 2003; Filarowski *et al.*, 2003; Nazır *et al.*, 2000; Yıldız *et al.*, 1998).

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Experimental

The title compound, (I), was prepared as described in the literature (Odabaşoğlu, Albayrak, Büyükgüngör & Lönnecke, 2003) using *o*-vanillin, 4-methylaniline and tris(hydroxymethyl)aminomethane as starting materials. The product was recrystallized from ethanol and well shaped crystals were obtained by slow evaporation of a methanol solution (yield: 83%, m.p. 368–370 K).

Crystal data

$C_{19}H_{23}N_3O_5$
$M_r = 373.40$
Monoclinic, $P2_1/c$
$a = 17.4603$ (17) Å
$b = 10.7156$ (12) Å
$c = 10.6495$ (9) Å
$\beta = 94.830$ (7)°
$V = 1985.4$ (3) Å^3
$Z = 4$
$D_x = 1.249$ Mg m^{-3}
Mo $K\alpha$ radiation
Cell parameters from 6249 reflections
$\theta = 25.8$–1.9°
$\mu = 0.09$ mm^{-1}
$T = 293$ (2) K
Prism, orange
0.25 × 0.15 × 0.09 mm

Data collection

Stoe IPDS-2 diffractometer
ω scans
Absorption correction: by integration (*X-RED*32; Stoe & Cie, 2002)
$T_{\min} = 0.983$, $T_{\max} = 0.992$
15669 measured reflections
3500 independent reflections
1490 reflections with $I > 2\sigma(I)$
$R_{\rm int} = 0.137$
$\theta_{\max} = 25.0°$
$h = -20 \rightarrow 20$
$k = -12 \rightarrow 12$
$l = -12 \rightarrow 12$

Refinement

Refinement on F^2
$R[F^2 > 2\sigma(F^2)] = 0.071$
$wR(F^2) = 0.173$
$S = 0.94$
3500 reflections
289 parameters
H atoms treated by a mixture of independent and constrained refinement
$w = 1/[\sigma^2(F_o^2) + (0.066P)^2]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{\max} = 0.029$
$\Delta\rho_{\max} = 0.19$ e Å^{-3}
$\Delta\rho_{\min} = -0.26$ e Å^{-3}

Table 1
Selected geometric parameters (Å, °).

C1—N1	1.429 (6)	C9—C10	1.424 (6)
C4—C19	1.516 (8)	C10—O1	1.279 (5)
C7—C8	1.363 (6)	C11—C12	1.340 (6)
C7—N2	1.417 (5)	C13—N3	1.296 (5)
C7—C12	1.417 (6)	C14—N3	1.460 (5)
C8—C9	1.402 (6)	N1—N2	1.247 (5)
C9—C13	1.406 (6)		
C8—C7—N2	115.2 (4)	C12—C11—O5	127.1 (4)
C8—C7—C12	119.4 (4)	N3—C13—C9	123.3 (4)
N2—C7—C12	125.3 (4)	C15—C14—C17	106.9 (4)
O1—C10—C9	122.7 (4)	C13—N3—C14	128.4 (4)
C10—C9—C13—N3	1.0 (7)	C1—N1—N2—C7	177.2 (4)

Table 2
Hydrogen-bonding geometry (Å, °).

D—H···A	D—H	H···A	D···A	D—H···A
N3—H1···O1	0.87 (4)	1.91 (4)	2.611 (4)	136 (4)
O2—H22···O4$^{\rm i}$	0.87 (5)	1.92 (5)	2.774 (4)	171 (4)
O3—H33···O2$^{\rm ii}$	0.80 (6)	1.92 (6)	2.726 (5)	179 (6)
O4—H44···O1$^{\rm iii}$	0.90 (5)	1.98 (5)	2.780 (4)	148 (4)
O4—H44···O5$^{\rm iii}$	0.90 (5)	2.26 (5)	2.973 (5)	136 (4)

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

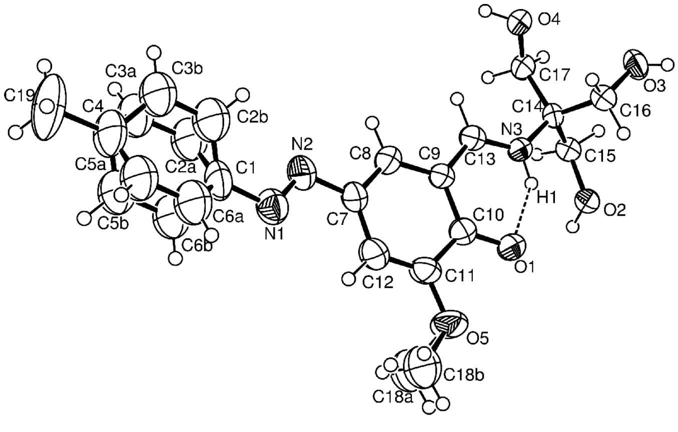

Figure 1
An *ORTEP*-3 (Farrugia, 1997) view of (I), with the atom-numbering scheme and 50% probability displacement ellipsoids. There is an orientational disorder, and the occupancy factors of atoms C2*a*/C3*a*/C5*a*/C6*a*/C18*a* and C2*b*/C3*b*/C5*b*/C6*b*/C18*b* are 55.0 (5) and 45.0 (5)%, respectively.

The H atoms attached to atoms C18 and C19 and the disordered C atoms were refined using a riding model, with C—H (aromatic CH) = 0.93 Å and C—H (CH_3) = 0.96 Å. The $U_{\rm iso}$ values were assigned as $1.5U_{\rm eq}(X)$ for C18 and $1.2U_{\rm eq}(X)$ for the others. The other H atoms were refined isotropically. These C—H bond lengths are in the range 0.95 (5)–1.04 (5) Å.

Data collection: *X-AREA* (Stoe & Cie, 2002); cell refinement: *X-AREA*; data reduction: *X-RED*32 (Stoe & Cie, 2002); program(s) used to solve structure: *SHELXS*97 (Sheldrick, 1997); program(s) used to refine structure: *SHELXL*97 (Sheldrick, 1997); molecular graphics: *ORTEP*-3 *for Windows* (Farrugia, 1997); software used to prepare material for publication: *WinGX* (Farrugia, 1999).

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