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(*Z*)-6-{[1,3-Dihydroxy-2-(hydroxymethyl)propan-2-ylamino]methylene}-4-[(*E*)-(4-ethylphenyl)diazenyl]-cyclohexa-2,4-dienone

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The title compound, $C_{19}H_{23}N_3O_4$, adopts the keto-amine tautomeric form, with the H atom located on N rather than on O. This H atom is involved in a strong intramolecular hydrogen bond. The configuration around the azo N═N double bond is *trans* and the dihedral angle between the two aromatic rings is 8.94 (2)°. The molecules, with intramolecular N—H···O hydrogen bonding, are linked by intermolecular O—H···O hydrogen bonds, forming a three-dimensional network.

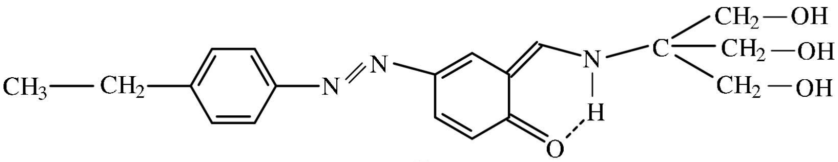
Key indicators

Single-crystal X-ray study
T = 296 K
Mean *σ*(C–C) = 0.010 Å
Disorder in main residue
R factor = 0.107
wR factor = 0.313
Data-to-parameter ratio = 15.5

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

Comment

Optically active azobenzene polymers are very important functional materials because of their photoresponsive properties. The pendant azobenzene groups in these materials behave as both photoresponsive chromophores and mesogens (Labarthet *et al.*, 1999). Recently, the formation of holographic gratings with polymeric azobenzene liquid crystals (LC) containing only azobenzene groups, where each group is mesogenic and photoresponsive, was reported (Yamamoto *et al.*, 2001). We present here the crystal structure of the title compound, (I).



(I)

In the azo group of (I), the N1—C6 and N2—C9 bond lengths [1.428 (9) and 1.428 (8) Å, respectively] indicate significant single-bond character, whereas the –N═N– bond length [1.253 (7) Å] is indicative of significant double-bond character. Similar values have been observed in other *trans*-azo compounds (Odabaşoğlu *et al.*, 2003; Koşar *et al.*, 2004). The 4-ethyl group shows disorder and was studied in two different orientations as groups C*a* and C*b* (C*a* is the C1*a*–C2*a* group and C*b* is the C1*b*–C2*b* group), with occupancy factors of 0.396 (14) and 0.604 (14), respectively (Fig. 1). In the molecule of (I), the C12═O1 and C11—C15 bond lengths are 1.292 (7) and 1.416 (8) Å, respectively (Table 1), and these are in agreement with the corresponding distances in 4-[(4-methylphenyl)diazenyl]-6-methoxy-2-{[tris(hydroxymethyl)-methyl]aminomethylene}-3,5-dien-1(2*H*)-one [1.279 (5) and 1.406 (6) Å, respectively; Koşar *et al.*, 2004].

In the molecule of (I), the dihedral angle, θ_1, between the mean planes of the C3–C8 benzene ring and the C6—N1═N2—C9 azo bridge is 12.14 (3)°, and the angle, θ_2, between the planes of the C6—N1═N2—C9 azo group and the substituted C9–C14 phenyl ring is 8.55 (2)°. The angle, θ_3, between the planes of the two rings is 8.94 (2)°.

In the crystal structure, compound (I) has strong intramolecular N—H···O [N3···O1 = 2.652 (6) and N3···O2 = 2.712 (7) Å] and weak intermolecular O—H···O [O3···O1 = 2.685 (6), O2···O3 = 2.718 (6) and O4···O1 = 2.726 (6) Å] hydrogen bonds (see Table 2 for details).

Experimental

Compound (I) was prepared as described in the literature (Odabaşoğlu *et al.*, 2003) using salicylaldehyde, 4-ethylaniline and tris(hydroxymethyl)aminomethane as starting materials. The product was recrystallized from ethanol and well shaped crystals were obtained by slow evaporation of the same solution (yield 85%; m.p. 463–465 K).

Crystal data

$C_{19}H_{23}N_3O_4$
$M_r = 357.40$
Monoclinic, $P2_1/c$
$a = 18.441$ (4) Å
$b = 7.8992$ (10) Å
$c = 13.014$ (3) Å
$\beta = 95.516$ (16)°
$V = 1887.0$ (6) Å^3
$Z = 4$
$D_x = 1.258$ Mg m^{-3}
Mo $K\alpha$ radiation
Cell parameters from 7373 reflections
$\theta = 1.6$–23.5°
$\mu = 0.09$ mm^{-1}
$T = 296$ K
Plate, brown
0.50 × 0.40 × 0.12 mm

Data collection

Stoe IPDS 2 diffractometer
ω scans
Absorption correction: none
9340 measured reflections
3690 independent reflections
1336 reflections with $I > 2\sigma(I)$
$R_{\rm int} = 0.129$
$\theta_{\rm max} = 26.0°$
$h = -22 \rightarrow 22$
$k = -9 \rightarrow 9$
$l = -13 \rightarrow 16$

Refinement

Refinement on F^2
$R[F^2 > 2\sigma(F^2)] = 0.107$
$wR(F^2) = 0.313$
$S = 0.93$
3690 reflections
238 parameters
H atoms treated by a mixture of independent and constrained refinement
$w = 1/[\sigma^2(F_o^2) + (0.157P)^2]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{\rm max} < 0.001$
$\Delta\rho_{\rm max} = 0.47$ e Å^{-3}
$\Delta\rho_{\rm min} = -0.31$ e Å^{-3}

Table 1
Selected geometric parameters (Å, °).

C6—N1	1.428 (9)	C15—N3	1.295 (7)
C9—N2	1.428 (8)	C16—N3	1.482 (7)
C11—C15	1.416 (8)	N1—N2	1.253 (7)
C12—O1	1.292 (7)		
C6—N1—N2—C9	−175.9 (5)		

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

D—H···A	D—H	H···A	D···A	D—H···A
O2—H2···O3$^{\rm i}$	0.83 (2)	1.93 (4)	2.718 (6)	158 (8)
O3—H3···O1$^{\rm ii}$	0.84 (2)	1.85 (2)	2.685 (6)	177 (8)
N3—H3A···O1	0.93 (8)	2.00 (7)	2.652 (6)	125 (6)
N3—H3A···O2	0.93 (8)	2.20 (7)	2.712 (7)	114 (5)
O4—H4···O1$^{\rm ii}$	0.82 (2)	1.96 (3)	2.762 (6)	163 (8)

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

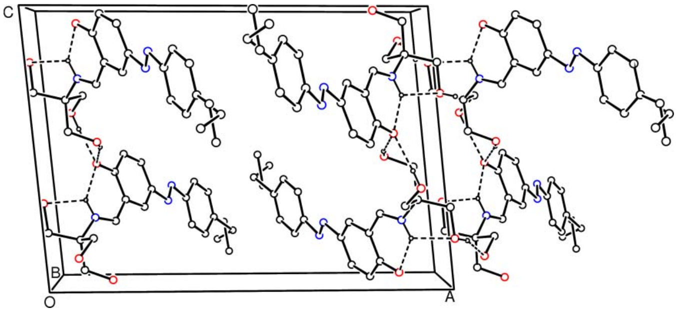


Figure 2
A packing diagram for (I), showing the hydrogen bonds (dashed lines). Both disorder components are shown. H atoms not involved in hydrogen bonding have been omitted for clarity.

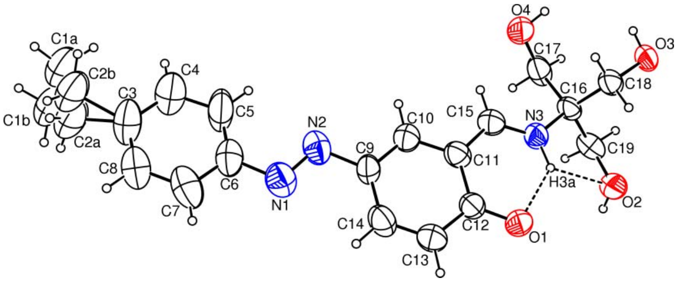


Figure 1
A view of the molecule of (I), with the atom-numbering scheme and 50% probability displacement ellipsoids. Dashed lines indicate the intramolecular hydrogen bonds. Both disorder components are shown.

The crystal of (I) used for data collection was not of good quality and was twinned. Accordingly, the quality of the crystallographic data was not as desired and this is the reason for the low quality of some crystallographic parameters. All attempts to find a better crystal failed. All H atoms bound to C atoms were refined using a riding model, with C—H = 0.93 Å and $U_{\rm iso}({\rm H}) = 1.2U_{\rm eq}({\rm C})$ for aromatic C atoms, C—H = 0.97 Å and $U_{\rm iso}({\rm H}) = 1.2U_{\rm eq}({\rm C})$ for methylene C atoms, and C—H = 0.96 Å and $U_{\rm iso}({\rm H}) = 1.5U_{\rm eq}({\rm C})$ for methyl C atoms. The H atoms of the hydroxyl O atoms were refined with O—H restrained to 0.83 (2) Å and with $U_{\rm iso}({\rm H}) = 1.5U_{\rm eq}({\rm O})$. The H atom bonded to N was refined freely, with $U_{\rm iso}({\rm H}) = 1.2U_{\rm eq}({\rm N})$.

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: *ORTEP3* for Windows (Farrugia, 1997); software used to prepare material for publication: *WinGX* (Farrugia, 1999).

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