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(*E*,*E*)-4,4′-Dimethyl-2,2′-(1,1′-dibenzylazino)diphenol

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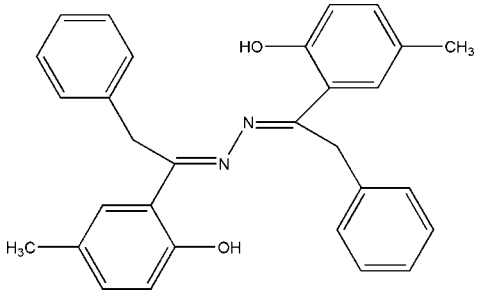
Key indicators: single-crystal X-ray study; T = 273 K; mean σ(C–C) = 0.003 Å; R factor = 0.045; wR factor = 0.163; data-to-parameter ratio = 13.3.

The title molecule, $C_{30}H_{28}N_2O_2$, (I), resides on a crystallographic inversion centre located at the midpoint of the N—N bond. It contains an intramolecular O—H···N interaction [O···N = 2.572 (2) Å] and a weak C—H···N contact [C···N = 2.752 (2) Å]. There are no intermolecular interactions of note in the crystal structure.

Related literature

For related literature, see: Glaser *et al.* (1995); Hunig *et al.* (2000); Kesslen *et al.* (1999); Kundu *et al.* (2005).



Experimental

Crystal data

$C_{30}H_{28}N_2O_2$
M_r = 448.54
Monoclinic, $P2_1/c$
a = 7.7881 (3) Å
b = 9.3199 (4) Å
c = 16.5419 (6) Å
β = 98.388 (2)°
V = 1187.84 (8) Å^3
Z = 2
Mo $K\alpha$ radiation
μ = 0.08 mm^{-1}
T = 273 (2) K
0.32 × 0.23 × 0.12 mm

Data collection

Bruker APEXII CCD area-detector diffractometer
Absorption correction: multi-scan (*SADABS*; Sheldrick, 2003)
T_{min} = 0.989, T_{max} = 0.992
13182 measured reflections
2089 independent reflections
1435 reflections with $I > 2\sigma(I)$
R_{int} = 0.049

Refinement

$R[F^2 > 2\sigma(F^2)]$ = 0.045
$wR(F^2)$ = 0.163
S = 1.00
2089 reflections
157 parameters
24 restraints
H-atom parameters constrained
$\Delta\rho_{max}$ = 0.16 e Å^{-3}
$\Delta\rho_{min}$ = −0.14 e Å^{-3}

Table 1
Hydrogen-bond geometry (Å, °).

D—H···A	D—H	H···A	D···A	D—H···A
O1—H1···N1	0.82	1.85	2.572 (2)	146
C9—H9A···N1^{i}	0.97	2.37	2.752 (2)	103

Symmetry code: (i) $-x+2, -y+1, -z+1$.

Data collection: *APEX2* (Bruker, 2005); cell refinement: *SAINT* (Bruker, 2005); data reduction: *SAINT*; program(s) used to solve structure: *SHELXS97* (Sheldrick, 1997*a*); program(s) used to refine structure: *SHELXL97* (Sheldrick, 1997*a*); molecular graphics: *SHELXTL* (Sheldrick, 1997*b*); software used to prepare material for publication: *SHELXTL*.

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Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: GG2038).

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supplementary materials

Acta Cryst. (2007). E**63**, o4061 [**doi:10.1107/S1600536807044182**]

(*E*,*E*)-4,4'-Dimethyl-2,2'-(1,1'-dibenzylazino)diphenol

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Comment

Recently, a number of azine compounds containing both a diimine linkage and N—N bond have been investigated in terms of their crystallography and coordination chemistry (Kundu *et al.*, 2005; Kesslen *et al.*, 1999). As an extension of work on the structural characterization of azine derivatives, the title compound, (I),was synthesized and its crystal structure is reported here.

In the title compound (I), there is a crystallographic centre of symmetry at the midpoint of the N—N bond (Fig. 1). The molecule displays the (E,*E*) conformation with respect to the C8═N1 and its symmetry related C8a═N1a double bond (Fig. 1). This configuration agrees with those commonly found in similar compounds (Glaser *et al.*, 1995; Hunig *et al.*, 2000). The C_6 aromatic rings in the asymmetric unit, C1–C6 (A), C10–C15 (B) have dihedral angles of 81.36 (7)°. The crystal structure is stabilized by intramolecular O—H···N hydrogen bonds and C—H···N contacts (Table 1).

Experimental

An ethanol solution (50 ml) of hydrazine (0.02 mol) and 1-(2-hydroxy-5-methylphenyl)-2-phenylethanone (0.04 mol) was refluxed and stirred for 5 h; the mixture was cooled and the resulting solid product, (I), was collected by filtration. Crystals suitable for single-crystal X-ray diffraction were grown by slow evaporation of a solution in acetone.

Refinement

All H atoms were positioned geometrically and treated as riding on their parent atoms, with *C*—$H_{(methyl)}$ = 0.96 Å, *C*—$H_{(methylene)}$ = 0.97 Å, C—$H_{(aromatic)}$ = 0.93 Å, O—H = 0.82 Å and with U_{iso}(H) =1.5U_{eq}(C_{methyl},*O*) and 1.2U_{eq}($C_{aromatic}$,$C_{methylene}$).

Figures

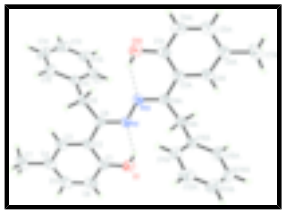


Fig. 1. The molecular structure of compound (I), showing the atom-numbering scheme. Displacement ellipsoids are drawn at the 30% probability level. Dashed lines show intramolecular hydrogen bonds.

(*E*,*E*)-4,4'-Dimethyl-2,2'-(1,1'-dibenzylazino)diphenol

Crystal data

$C_{30}H_{28}N_2O_2$	F_{000} = 476
M_r = 448.54	D_x = 1.254 Mg m^{-3}
Monoclinic, $P2_1/c$	Mo $K\alpha$ radiation λ = 0.71073 Å
Hall symbol: -P 2ybc	Cell parameters from 1572 reflections
a = 7.7881 (3) Å	θ = 2.5–19.9º
b = 9.3199 (4) Å	μ = 0.08 mm^{-1}
c = 16.5419 (6) Å	T = 273 (2) K
β = 98.388 (2)º	Plate, yellow
V = 1187.84 (8) Å^3	0.32 × 0.23 × 0.12 mm
Z = 2	

Data collection

Bruker APEXII CCD area-detector diffractometer	2089 independent reflections
Radiation source: fine-focus sealed tube	1435 reflections with $I > 2\sigma(I)$
Monochromator: graphite	R_{int} = 0.049
T = 273(2) K	θ_{max} = 25.0º
φ and ω scans	θ_{min} = 2.5º
Absorption correction: multi-scan (SADABS; Sheldrick, 2003)	h = −9→9
T_{min} = 0.989, T_{max} = 0.992	k = −11→11
13182 measured reflections	l = −19→19

Refinement

Refinement on F^2	Secondary atom site location: difference Fourier map
Least-squares matrix: full	Hydrogen site location: inferred from neighbouring sites
$R[F^2 > 2\sigma(F^2)]$ = 0.045	H-atom parameters constrained
$wR(F^2)$ = 0.163	$w = 1/[\sigma^2(F_o^2) + (0.106P)^2]$ where $P = (F_o^2 + 2F_c^2)/3$
S = 1.01	$(\Delta/\sigma)_{max} < 0.001$
2089 reflections	$\Delta\rho_{max}$ = 0.16 e Å^{-3}
157 parameters	$\Delta\rho_{min}$ = −0.14 e Å^{-3}
24 restraints	Extinction correction: SHELXL97 (Sheldrick, 1997a), $Fc^*=kFc[1+0.001xFc^2\lambda^3/\sin(2\theta)]^{-1/4}$
Primary atom site location: structure-invariant direct methods	Extinction coefficient: 0.026 (7)

Special details

Geometry. All e.s.d.'s (except the e.s.d. in the dihedral angle between two l.s. planes) are estimated using the full covariance matrix. The cell e.s.d.'s are taken into account individually in the estimation of e.s.d.'s in distances, angles and torsion angles; correlations between e.s.d.'s in cell parameters are only used when they are defined by crystal symmetry. An approximate (isotropic) treatment of cell e.s.d.'s is used for estimating e.s.d.'s involving l.s. planes.

Refinement. Refinement of F^2 against ALL reflections. The weighted *R*-factor *wR* and goodness of fit S are based on F^2, conventional *R*-factors *R* are based on F, with F set to zero for negative F^2. The threshold expression of $F^2 > 2\text{sigma}(F^2)$ is used only for calculating *R*-factors(gt) *etc*. and is not relevant to the choice of reflections for refinement. *R*-factors based on F^2 are statistically about twice as large as those based on F, and R– factors based on ALL data will be even larger.

Fractional atomic coordinates and isotropic or equivalent isotropic displacement parameters (Å^2)

	x	*y*	*z*	U_{iso}*/U_{eq}
O1	1.0013 (2)	0.69688 (17)	0.65896 (9)	0.0737 (5)
H1	1.0204	0.6307	0.6288	0.111*
N1	0.9839 (2)	0.56036 (16)	0.52287 (10)	0.0502 (5)
C1	0.9186 (3)	0.8041 (2)	0.61378 (14)	0.0568 (6)
C2	0.8747 (2)	0.7964 (2)	0.52817 (12)	0.0492 (5)
C3	0.7900 (2)	0.9149 (2)	0.48862 (14)	0.0543 (6)
H3	0.7609	0.9113	0.4321	0.065*
C4	0.7470 (3)	1.0370 (2)	0.52864 (15)	0.0599 (6)
C5	0.7929 (3)	1.0392 (3)	0.61281 (17)	0.0715 (7)
H5	0.7660	1.1197	0.6417	0.086*
C6	0.8768 (3)	0.9260 (3)	0.65486 (15)	0.0697 (7)
H6	0.9057	0.9312	0.7113	0.084*
C7	0.6570 (3)	1.1611 (2)	0.48306 (18)	0.0810 (8)
H7A	0.6090	1.2226	0.5206	0.121*
H7B	0.5656	1.1261	0.4426	0.121*
H7C	0.7389	1.2142	0.4567	0.121*
C8	0.9139 (2)	0.66973 (19)	0.48152 (12)	0.0463 (5)
C9	0.8695 (2)	0.6714 (2)	0.38985 (12)	0.0508 (6)
H9A	0.9417	0.6019	0.3670	0.061*
H9B	0.8963	0.7654	0.3698	0.061*
C10	0.6811 (3)	0.63753 (19)	0.36001 (12)	0.0477 (5)
C11	0.5936 (3)	0.5321 (2)	0.39669 (13)	0.0589 (6)
H11	0.6507	0.4824	0.4415	0.071*
C12	0.4225 (3)	0.4998 (3)	0.36756 (17)	0.0778 (8)
H12	0.3655	0.4280	0.3923	0.093*
C13	0.3372 (3)	0.5736 (4)	0.30232 (19)	0.0886 (9)
H13	0.2222	0.5517	0.2826	0.106*
C14	0.4203 (4)	0.6795 (3)	0.26598 (17)	0.0897 (9)
H14	0.3616	0.7300	0.2219	0.108*
C15	0.5920 (3)	0.7115 (3)	0.29474 (14)	0.0694 (7)
H15	0.6479	0.7837	0.2698	0.083*

Atomic displacement parameters (Å^2)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
O1	0.0905 (12)	0.0692 (11)	0.0575 (10)	−0.0016 (9)	−0.0020 (9)	−0.0031 (8)
N1	0.0501 (9)	0.0438 (9)	0.0543 (11)	−0.0025 (7)	−0.0004 (8)	−0.0038 (7)
C1	0.0570 (12)	0.0534 (12)	0.0603 (14)	−0.0109 (10)	0.0097 (10)	−0.0054 (11)
C2	0.0435 (10)	0.0463 (11)	0.0585 (14)	−0.0085 (8)	0.0093 (9)	−0.0027 (9)
C3	0.0500 (10)	0.0498 (11)	0.0644 (13)	−0.0069 (9)	0.0123 (9)	−0.0007 (10)
C4	0.0519 (11)	0.0489 (12)	0.0824 (16)	−0.0081 (9)	0.0213 (10)	−0.0055 (10)
C5	0.0699 (13)	0.0583 (13)	0.0920 (17)	−0.0088 (10)	0.0316 (12)	−0.0157 (12)
C6	0.0760 (14)	0.0721 (15)	0.0638 (14)	−0.0137 (12)	0.0191 (11)	−0.0151 (12)
C7	0.0686 (15)	0.0512 (13)	0.127 (2)	0.0038 (11)	0.0285 (15)	0.0020 (14)
C8	0.0405 (10)	0.0443 (11)	0.0537 (13)	−0.0059 (8)	0.0055 (9)	−0.0004 (9)
C9	0.0560 (11)	0.0472 (11)	0.0505 (13)	0.0003 (9)	0.0116 (9)	0.0043 (9)
C10	0.0577 (11)	0.0407 (10)	0.0437 (11)	0.0066 (9)	0.0037 (9)	−0.0046 (9)
C11	0.0582 (12)	0.0552 (13)	0.0608 (14)	−0.0041 (10)	0.0001 (11)	−0.0001 (10)
C12	0.0643 (15)	0.0831 (17)	0.0839 (19)	−0.0162 (13)	0.0043 (14)	−0.0184 (15)
C13	0.0556 (14)	0.116 (2)	0.088 (2)	0.0092 (15)	−0.0097 (15)	−0.0383 (19)
C14	0.0828 (19)	0.116 (2)	0.0616 (17)	0.0375 (17)	−0.0189 (15)	−0.0110 (16)
C15	0.0849 (17)	0.0682 (15)	0.0530 (14)	0.0157 (12)	0.0027 (12)	0.0064 (11)

Geometric parameters (Å, °)

O1—C1	1.353 (3)	C7—H7C	0.9600
O1—H1	0.8200	C8—C9	1.505 (3)
N1—C8	1.302 (2)	C9—C10	1.512 (3)
N1—N1^{i}	1.399 (3)	C9—H9A	0.9700
C1—C6	1.387 (3)	C9—H9B	0.9700
C1—C2	1.409 (3)	C10—C15	1.380 (3)
C2—C3	1.398 (3)	C10—C11	1.385 (3)
C2—C8	1.467 (3)	C11—C12	1.383 (3)
C3—C4	1.382 (3)	C11—H11	0.9300
C3—H3	0.9300	C12—C13	1.368 (4)
C4—C5	1.386 (3)	C12—H12	0.9300
C4—C7	1.498 (3)	C13—C14	1.367 (4)
C5—C6	1.375 (3)	C13—H13	0.9300
C5—H5	0.9300	C14—C15	1.385 (4)
C6—H6	0.9300	C14—H14	0.9300
C7—H7A	0.9600	C15—H15	0.9300
C7—H7B	0.9600		
C1—O1—H1	109.5	N1—C8—C9	123.66 (17)
C8—N1—N1^{i}	116.10 (19)	C2—C8—C9	119.12 (17)
O1—C1—C6	117.5 (2)	C8—C9—C10	113.35 (15)
O1—C1—C2	122.94 (18)	C8—C9—H9A	108.9
C6—C1—C2	119.6 (2)	C10—C9—H9A	108.9
C3—C2—C1	117.32 (19)	C8—C9—H9B	108.9
C3—C2—C8	120.63 (19)	C10—C9—H9B	108.9

C1—C2—C8	122.05 (18)	H9A—C9—H9B	107.7
C4—C3—C2	123.8 (2)	C15—C10—C11	118.3 (2)
C4—C3—H3	118.1	C15—C10—C9	120.29 (19)
C2—C3—H3	118.1	C11—C10—C9	121.39 (17)
C3—C4—C5	116.7 (2)	C12—C11—C10	120.8 (2)
C3—C4—C7	121.5 (2)	C12—C11—H11	119.6
C5—C4—C7	121.8 (2)	C10—C11—H11	119.6
C6—C5—C4	122.0 (2)	C13—C12—C11	119.9 (3)
C6—C5—H5	119.0	C13—C12—H12	120.1
C4—C5—H5	119.0	C11—C12—H12	120.1
C5—C6—C1	120.6 (2)	C14—C13—C12	120.2 (2)
C5—C6—H6	119.7	C14—C13—H13	119.9
C1—C6—H6	119.7	C12—C13—H13	119.9
C4—C7—H7A	109.5	C13—C14—C15	120.0 (2)
C4—C7—H7B	109.5	C13—C14—H14	120.0
H7A—C7—H7B	109.5	C15—C14—H14	120.0
C4—C7—H7C	109.5	C10—C15—C14	120.7 (3)
H7A—C7—H7C	109.5	C10—C15—H15	119.6
H7B—C7—H7C	109.5	C14—C15—H15	119.6
N1—C8—C2	117.21 (18)		
O1—C1—C2—C3	−179.72 (18)	C1—C2—C8—N1	3.4 (3)
C6—C1—C2—C3	0.2 (3)	C3—C2—C8—C9	2.9 (3)
O1—C1—C2—C8	0.7 (3)	C1—C2—C8—C9	−177.61 (16)
C6—C1—C2—C8	−179.32 (18)	N1—C8—C9—C10	97.6 (2)
C1—C2—C3—C4	−0.4 (3)	C2—C8—C9—C10	−81.4 (2)
C8—C2—C3—C4	179.19 (16)	C8—C9—C10—C15	140.17 (19)
C2—C3—C4—C5	0.3 (3)	C8—C9—C10—C11	−40.1 (2)
C2—C3—C4—C7	179.69 (19)	C15—C10—C11—C12	1.3 (3)
C3—C4—C5—C6	−0.1 (3)	C9—C10—C11—C12	−178.43 (19)
C7—C4—C5—C6	−179.5 (2)	C10—C11—C12—C13	−0.8 (4)
C4—C5—C6—C1	−0.1 (3)	C11—C12—C13—C14	−0.2 (4)
O1—C1—C6—C5	179.9 (2)	C12—C13—C14—C15	0.6 (4)
C2—C1—C6—C5	0.0 (3)	C11—C10—C15—C14	−1.0 (3)
N1^{i}—N1—C8—C2	178.47 (17)	C9—C10—C15—C14	178.8 (2)
N1^{i}—N1—C8—C9	−0.5 (3)	C13—C14—C15—C10	0.0 (4)
C3—C2—C8—N1	−176.16 (16)		

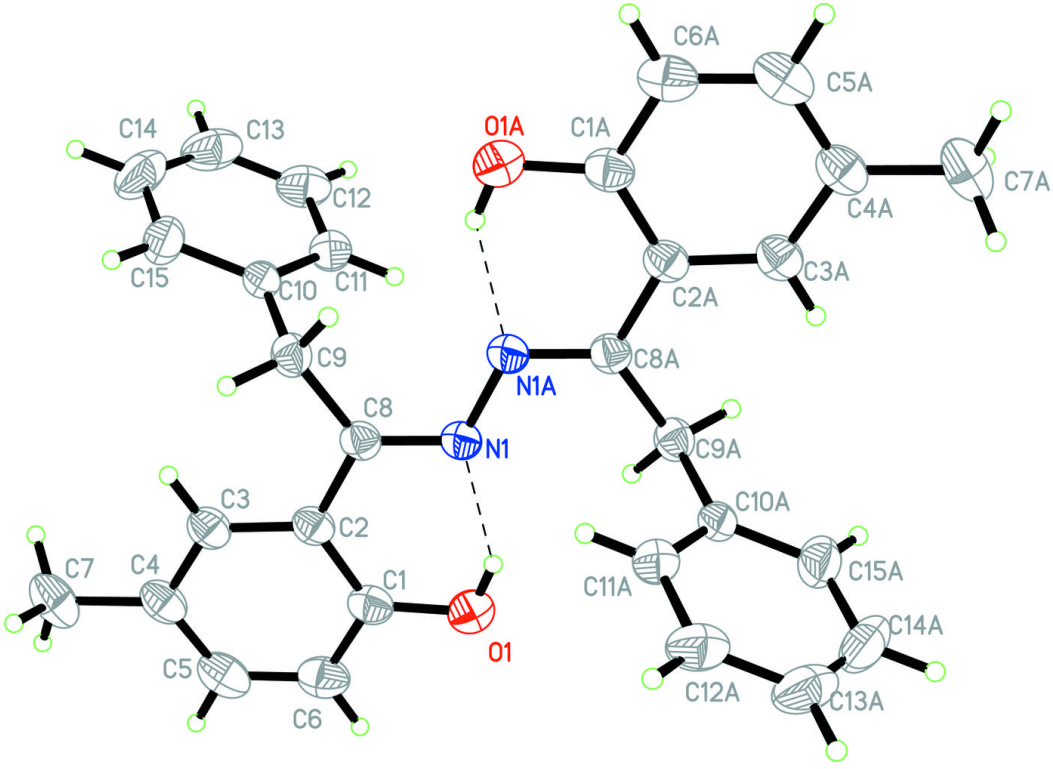
Symmetry codes: (i) −*x*+2, −*y*+1, −*z*+1.

Hydrogen-bond geometry (Å, °)

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
O1—H1···N1	0.82	1.85	2.572 (2)	146
C9—H9A···N1^{i}	0.97	2.37	2.752 (2)	103

Symmetry codes: (i) −*x*+2, −*y*+1, −*z*+1.

Fig. 1


C6A
C5A
O1A
C1A
C14
C13
C12
C4A
C7A
C15
C10
C11
C3A
C2A
C9
C8A
N1A
C8
N1
C9A
C3
C10A
C2
C7
C4
C1
C15A
C11A
C14A
O1
C5
C12A
C13A
C6