organic compounds

4,4′-Dimethyl-2,2′-[1,1′-(ethane-1,2-diyldinitrilo)diethylidyne]diphenol

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The title compound, $C_{20}H_{24}N_2O_2$, is a Schiff base compound derived from the condensation of 2-hydroxy-5-methyl-acetophenone and 1,2-diaminoethane in ethanol. The molecule has crystallographic twofold rotation symmetry. The molecular structure is stabilized by weak intramolecular O—H···N interactions and the crystal packing is stabilized by weak intermolecular C—H···O and C—H···π interactions.

Related literature

For related literature, see: Cozzi (2004); Sun *et al.* (2004); Xiao & Wang (2006). A similar Schiff base compound has been reported (Zhang & Li, 2006).
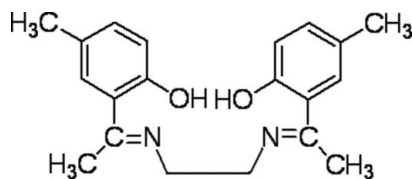
Experimental

Crystal data

$C_{20}H_{24}N_2O_2$	Orthorhombic, $Pbcn$
M_r = 324.41	a = 18.9118 (8) Å
b = 6.9370 (3) Å	Mo $K\alpha$ radiation
c = 13.4509 (5) Å	μ = 0.08 mm^{-1}
V = 1764.64 (13) Å^3	T = 295 (2) K
Z = 4	0.30 × 0.22 × 0.20 mm

Data collection

Bruker Kappa APEXII diffractometer	10871 measured reflections
Absorption correction: multi-scan (*SADABS*; Sheldrick, 1996) T_{min} = 0.895, T_{max} = 0.984	2193 independent reflections 1556 reflections with $I > 2\sigma(I)$ R_{int} = 0.024

Refinement

$R[F^2 > 2\sigma(F^2)]$ = 0.049	112 parameters
$wR(F^2)$ = 0.157	H-atom parameters constrained
S = 1.05	$\Delta\rho_{max}$ = 0.24 e Å^{-3}
2193 reflections	$\Delta\rho_{min}$ = −0.22 e Å^{-3}

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

$Cg1$ is the centroid of the C1–C6 ring.

D—H···A	D—H	H···A	D···A	D—H···A
O1—H1···N1	0.82	1.78	2.5070 (18)	147
C6—H6···O1[i]	0.93	2.58	3.490 (2)	166
C2—H2···$Cg1$[ii]	0.93	2.84	3.700	154

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

Data collection: *APEX2* (Bruker, 2004); cell refinement: *APEX2*; data reduction: *APEX2*; program(s) used to solve structure: *SHELXS97* (Sheldrick, 1997); program(s) used to refine structure: *SHELXL97* (Sheldrick, 1997); molecular graphics: *PLATON* (Spek, 2003); software used to prepare material for publication: *SHELXL97*.

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supplementary materials

4,4'-Dimethyl-2,2'-[1,1'-(ethane-1,2-diyldinitrilo)diethylidyne]diphenol

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Comment

Schiff base compounds constitute an important class of ligands which have been extensively studied in coordination chemistry mainly due to their facile synthesis and easily tunable steric, electronic and catalytic properties (Cozzi, 2004).

The geometric parameters in the molecule of title compound (I) (Fig. 1) agree with the reported values of similar structures (Sun *et al.*, 2004; Xiao & Wang, 2006; Zhang & Li, 2006). In the crystal, molecule placed on a twofold rotation axis passed through the middle of C10–C10*a* bond.

The N1/C8/C9 moiety is coplanar with the benzene ring (C1—C6) in each half of the molecule, with the dihedral angle of 2.23 (13)°. Atom C7 deviates by 0.032Å from the plane of benzene ring (C1—C6).

The molecular structure is stabilized by intramolecular O–H⋯N interaction. The crystal structure of (I) (Fig. 2) is stabilized by weak intermolecular C–H⋯O and C–H⋯π interactions involving the C1—C6 (centroid *Cg1*) ring. The details of these interactions are given in Table. A similiar Schiff base compound has been reported (Zhang & Li, 2006).

Experimental

1,2-Diaminoethane (8.17 mmol, 0.50 ml) in 20 ml of dry ethanol was added dropwise to a stirred solution of 2-hydroxy-5-methylacetophenone (16.24 mmol, 2.44 g) in 75 ml of dry ethanol. After the addition was over, the reaction mixture was stirred for another 12 h. The product was formed as a yellow colored solid and filtered. The product was washed with cold dry methanol followed by diethyl ether to remove the uncondensed amine. The single crystals were formed by slow evaporation of chloroform. Yield: 4.95 g (65%).

Refinement

H atoms were positioned geometrically and refined as riding, with C–H = 0.93Å and $U_{iso}(H) = 1.2U_{eq}(C)$ for aromatic C–H, with C–H = 0.97Å and $U_{iso}(H) = 1.2U_{eq}(C)$ for CH_2, with C–H = 0.96Å and $U_{iso}(H) = 1.5U_{eq}(C)$ for methyl C and with O–H = 0.82Å and $U_{iso}(H) = 1.2U_{eq}(O)$ for OH.
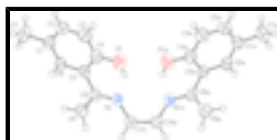
Figures

Fig. 1. The molecular structure of (I), with atom labeling scheme. Displacement ellipsoids are drawn at 50% probability level. H atoms are presented as spheres of arbitrary radius.
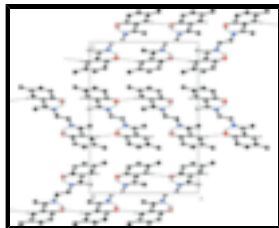
Fig. 2. The crystal packing of (I), viewed down the *b* axis. Hydrogen bonds are shown as dashed lines. H atoms not involved in hydrogen bonding have been omitted for crarity.

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Crystal data

$C_{20}H_{24}N_2O_2$	$F_{000} = 696$
$M_r = 324.41$	$D_x = 1.221$ Mg m^{-3}
Orthorhombic, *Pbcn*	Mo *K*α radiation $\lambda = 0.71073$ Å
Hall symbol: -P 2n 2ab	Cell parameters from 3689 reflections
$a = 18.9118$ (8) Å	$\theta = 3.0$–27.6º
$b = 6.9370$ (3) Å	$\mu = 0.08$ mm^{-1}
$c = 13.4509$ (5) Å	$T = 295$ (2) K
$V = 1764.64$ (13) Å^3	Needle, yellow
$Z = 4$	$0.30 \times 0.22 \times 0.20$ mm

Data collection

Bruker Kappa APEXII diffractometer	2193 independent reflections
Radiation source: Fine-focus sealed tube	1556 reflections with $I > 2\sigma(I)$
Monochromator: Graphite	$R_{int} = 0.024$
$T = 295(2)$ K	$\theta_{max} = 28.3$º
ω and φ scans	$\theta_{min} = 3.0$º
Absorption correction: multi-scan (SADABS; Sheldrick, 1996)	$h = -25 \rightarrow 25$
$T_{min} = 0.895$, $T_{max} = 0.984$	$k = -9 \rightarrow 8$
10871 measured reflections	$l = -17 \rightarrow 8$

Refinement

Refinement on F^2	Secondary atom site location: Difmap
Least-squares matrix: Full	Hydrogen site location: Geom
$R[F^2 > 2\sigma(F^2)] = 0.049$	H-atom parameters constrained
$wR(F^2) = 0.157$	$w = 1/[\sigma^2(F_o^2) + (0.079P)^2 + 0.4202P]$ where $P = (F_o^2 + 2F_c^2)/3$
$S = 1.05$	$(\Delta/\sigma)_{max} < 0.001$
2193 reflections	$\Delta\rho_{max} = 0.24$ e Å^{-3}
112 parameters	$\Delta\rho_{min} = -0.22$ e Å^{-3}
Primary atom site location: Direct	Extinction correction: None

Fractional atomic coordinates and isotropic or equivalent isotropic displacement parameters (Å²)

	x	*y*	*z*	U_{iso}*/U_{eq}
C1	0.67691 (8)	0.2634 (3)	−0.03521 (12)	0.0472 (4)
C2	0.69390 (9)	0.3718 (3)	0.04826 (14)	0.0532 (4)
H2	0.7209	0.4829	0.0410	0.064*
C3	0.67168 (10)	0.3180 (3)	0.14096 (13)	0.0568 (5)
H3	0.6841	0.3929	0.1956	0.068*
C4	0.63097 (9)	0.1540 (2)	0.15501 (12)	0.0467 (4)
C5	0.61416 (7)	0.0376 (2)	0.07231 (10)	0.0369 (3)
C6	0.63775 (8)	0.0983 (2)	−0.02103 (11)	0.0416 (4)
H6	0.6265	0.0237	−0.0762	0.050*
C7	0.70060 (12)	0.3257 (3)	−0.13669 (15)	0.0720 (6)
H7A	0.6847	0.2339	−0.1852	0.108*
H7B	0.7513	0.3329	−0.1383	0.108*
H7C	0.6810	0.4501	−0.1516	0.108*
C8	0.57283 (7)	−0.1400 (2)	0.08506 (11)	0.0378 (3)
C9	0.55678 (10)	−0.2657 (3)	−0.00308 (13)	0.0530 (4)
H9A	0.5568	−0.3984	0.0171	0.080*
H9B	0.5922	−0.2464	−0.0533	0.080*
H9C	0.5112	−0.2326	−0.0294	0.080*
C10	0.50985 (9)	−0.3514 (2)	0.19560 (13)	0.0488 (4)
H10A	0.5370	−0.4662	0.1801	0.059*
H10B	0.4674	−0.3523	0.1552	0.059*
N1	0.55154 (7)	−0.18039 (19)	0.17332 (10)	0.0445 (3)
O1	0.60886 (9)	0.1126 (2)	0.24693 (9)	0.0703 (4)
H1	0.5850	0.0139	0.2457	0.105*

Atomic displacement parameters (Å²)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
C1	0.0434 (8)	0.0526 (10)	0.0456 (9)	0.0041 (7)	0.0027 (6)	0.0115 (7)
C2	0.0528 (9)	0.0477 (9)	0.0592 (11)	−0.0067 (7)	−0.0001 (8)	0.0048 (8)
C3	0.0683 (11)	0.0549 (11)	0.0471 (11)	−0.0109 (8)	−0.0046 (8)	−0.0091 (8)
C4	0.0557 (9)	0.0497 (9)	0.0346 (8)	−0.0006 (7)	−0.0011 (6)	−0.0028 (6)
C5	0.0374 (7)	0.0418 (8)	0.0315 (7)	0.0051 (6)	0.0004 (5)	−0.0006 (6)
C6	0.0429 (8)	0.0498 (9)	0.0323 (8)	0.0038 (6)	0.0003 (6)	0.0010 (6)
C7	0.0805 (13)	0.0825 (15)	0.0530 (12)	−0.0141 (11)	0.0104 (9)	0.0185 (10)
C8	0.0385 (7)	0.0411 (8)	0.0340 (8)	0.0062 (6)	0.0021 (5)	−0.0032 (6)
C9	0.0672 (10)	0.0501 (10)	0.0416 (9)	−0.0042 (8)	0.0049 (7)	−0.0082 (7)
C10	0.0561 (9)	0.0418 (8)	0.0487 (10)	−0.0028 (7)	0.0141 (7)	−0.0043 (7)
N1	0.0510 (7)	0.0455 (7)	0.0370 (7)	−0.0029 (6)	0.0091 (5)	−0.0023 (5)
O1	0.1047 (11)	0.0748 (10)	0.0314 (7)	−0.0278 (8)	0.0082 (6)	−0.0093 (6)

Geometric parameters (Å, °)

C1—C6	1.377 (2)	C7—H7B	0.9600
C1—C2	1.389 (3)	C7—H7C	0.9600

C1—C7	1.500 (2)	C8—N1	1.2845 (19)
C2—C3	1.368 (3)	C8—C9	1.502 (2)
C2—H2	0.9300	C9—H9A	0.9600
C3—C4	1.387 (2)	C9—H9B	0.9600
C3—H3	0.9300	C9—H9C	0.9600
C4—O1	1.336 (2)	C10—N1	1.456 (2)
C4—C5	1.411 (2)	C10—C10^i	1.510 (3)
C5—C6	1.397 (2)	C10—H10A	0.9700
C5—C8	1.469 (2)	C10—H10B	0.9700
C6—H6	0.9300	O1—H1	0.8200
C7—H7A	0.9600		
C6—C1—C2	117.55 (15)	C1—C7—H7C	109.5
C6—C1—C7	121.74 (17)	H7A—C7—H7C	109.5
C2—C1—C7	120.71 (17)	H7B—C7—H7C	109.5
C3—C2—C1	121.24 (16)	N1—C8—C5	117.22 (13)
C3—C2—H2	119.4	N1—C8—C9	122.65 (14)
C1—C2—H2	119.4	C5—C8—C9	120.13 (13)
C2—C3—C4	121.23 (16)	C8—C9—H9A	109.5
C2—C3—H3	119.4	C8—C9—H9B	109.5
C4—C3—H3	119.4	H9A—C9—H9B	109.5
O1—C4—C3	118.44 (15)	C8—C9—H9C	109.5
O1—C4—C5	122.40 (15)	H9A—C9—H9C	109.5
C3—C4—C5	119.16 (15)	H9B—C9—H9C	109.5
C6—C5—C4	117.65 (14)	N1—C10—C10^i	109.45 (12)
C6—C5—C8	121.83 (13)	N1—C10—H10A	109.8
C4—C5—C8	120.52 (13)	C10^i—C10—H10A	109.8
C1—C6—C5	123.14 (14)	N1—C10—H10B	109.8
C1—C6—H6	118.4	C10^i—C10—H10B	109.8
C5—C6—H6	118.4	H10A—C10—H10B	108.2
C1—C7—H7A	109.5	C8—N1—C10	122.52 (13)
C1—C7—H7B	109.5	C4—O1—H1	109.5
H7A—C7—H7B	109.5		
C6—C1—C2—C3	−0.9 (3)	C7—C1—C6—C5	−179.22 (16)
C7—C1—C2—C3	178.99 (17)	C4—C5—C6—C1	0.8 (2)
C1—C2—C3—C4	−0.4 (3)	C8—C5—C6—C1	−179.60 (13)
C2—C3—C4—O1	−177.76 (17)	C6—C5—C8—N1	−177.73 (13)
C2—C3—C4—C5	2.0 (3)	C4—C5—C8—N1	1.8 (2)
O1—C4—C5—C6	177.59 (15)	C6—C5—C8—C9	2.0 (2)
C3—C4—C5—C6	−2.2 (2)	C4—C5—C8—C9	−178.43 (14)
O1—C4—C5—C8	−2.0 (2)	C5—C8—N1—C10	179.63 (13)
C3—C4—C5—C8	178.26 (15)	C9—C8—N1—C10	−0.1 (2)
C2—C1—C6—C5	0.7 (2)	C10^i—C10—N1—C8	−177.01 (16)

Symmetry codes: (i) −x+1, y, −z+1/2.

Hydrogen-bond geometry (Å, °)

D—H···A	D—H	H···A	D···A	D—H···A
O1—H1···N1	0.82	1.78	2.5070 (18)	147

C6—H6···O1[ii]	0.93	2.58	3.490 (2)	166
C2—H2···Cg1[iii]	0.93	2.84	3.700	154

Symmetry codes: (ii) x, $-y$, $z-1/2$; (iii) $-x-1/2$, $y-3/2$, z.

Fig. 1

Fig. 2