

Acta Crystallographica Section E

## Structure Reports

Online

ISSN 1600-5368

## 4,4'-Dimethoxy-2,2'-[1,1'-(propane-1,3-diyldinitrilo)diethylidyne]diphenol

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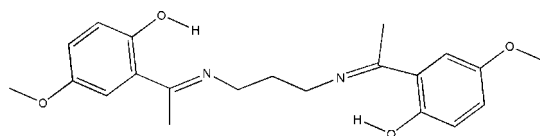
Received 21 July 2008; accepted 27 July 2008

Key indicators: single-crystal X-ray study;  $T = 100$  K; mean  $\sigma(\text{C}-\text{C}) = 0.001$  Å;  $R$  factor = 0.058;  $wR$  factor = 0.149; data-to-parameter ratio = 41.8.

In the crystal structure, the title Schiff base compound,  $\text{C}_{21}\text{H}_{26}\text{N}_2\text{O}_4$ , has twofold rotation symmetry. The imino group is coplanar with the aromatic ring. An intramolecular  $\text{O}-\text{H}\cdots\text{N}$  hydrogen bond forms a six-membered ring, producing an  $S(6)$  ring motif. The two benzene rings are almost perpendicular to each other, making a dihedral angle of  $85.00(2)^\circ$ . The methoxy group is approximately coplanar with the benzene ring, with a  $\text{C}-\text{O}-\text{C}-\text{C}$  torsion angle of  $2.34(12)^\circ$ . Neighbouring molecules are linked together by weak intermolecular  $\text{C}-\text{H}\cdots\text{O}$  hydrogen bonds and a  $\text{C}-\text{H}\cdots\pi$  interaction, forming a sheet parallel to the  $ab$  plane. The molecules also adopt a zigzag arrangement along the  $c$  axis.

## Related literature

For bond-length data, see: Allen *et al.* (1987). For hydrogen-bond motifs, see: Bernstein *et al.* (1995). For information on Schiff base ligands and complexes, and their applications, see, for example: Fun, Kargar & Kia (2008); Fun, Kia & Kargar (2008); Fun, Mirkhani *et al.* (2008a,b); Calligaris & Randaccio (1987); Casellato & Vigato (1977); Kia, Mirkhani, Kalman & Deak (2007); Kia, Mirkhani, Harkema & van Hummel (2007); Pal *et al.* (2005); Reglinski *et al.* (2004); Hou *et al.* (2001); Ren *et al.* (2002).



## Experimental

## Crystal data

 $\text{C}_{21}\text{H}_{26}\text{N}_2\text{O}_4$  $M_r = 370.44$ 

Monoclinic,  $C2/c$   
 $a = 12.8042(2)$  Å  
 $b = 5.0508(1)$  Å  
 $c = 28.6019(6)$  Å  
 $\beta = 93.109(2)^\circ$   
 $V = 1847.00(6)$  Å<sup>3</sup>

$Z = 4$   
 Mo  $K\alpha$  radiation  
 $\mu = 0.09$  mm<sup>-1</sup>  
 $T = 100.0(1)$  K  
 $0.47 \times 0.44 \times 0.29$  mm

## Data collection

Bruker SMART APEXII CCD  
 area-detector diffractometer  
 Absorption correction: multi-scan  
 (SADABS; Bruker, 2005)  
 $T_{\min} = 0.884$ ,  $T_{\max} = 0.974$

34801 measured reflections  
 5229 independent reflections  
 4304 reflections with  $I > 2\sigma(I)$   
 $R_{\text{int}} = 0.031$

## Refinement

$R[F^2 > 2\sigma(F^2)] = 0.057$   
 $wR(F^2) = 0.148$   
 $S = 1.11$   
 5229 reflections

125 parameters  
 H-atom parameters constrained  
 $\Delta\rho_{\text{max}} = 0.45$  e Å<sup>-3</sup>  
 $\Delta\rho_{\text{min}} = -0.28$  e Å<sup>-3</sup>

Table 1

Hydrogen-bond geometry (Å, °).

$D-H\cdots A$	$D-H$	$H\cdots A$	$D\cdots A$	$D-H\cdots A$
$\text{O1}-\text{H1O1}\cdots\text{N1}$	0.97	1.62	2.5241 (10)	153
$\text{C11}-\text{H11A}\cdots\text{O1}^{\text{i}}$	0.96	2.53	3.4448 (12)	160
$\text{C11}-\text{H11B}\cdots\text{O1}^{\text{ii}}$	0.96	2.53	3.4360 (12)	157
$\text{C10}-\text{H10C}\cdots\text{Cg1}^{\text{iii}}$	0.96	2.68	3.5224 (10)	147

Symmetry codes: (i)  $x - \frac{1}{2}, y - \frac{1}{2}, z$ ; (ii)  $x - \frac{1}{2}, y + \frac{1}{2}, z$ ; (iii)  $x, y + 1, z$ . Cg1 is the centroid of the C1-C6 benzene ring.

Data collection: APEX2 (Bruker, 2005); cell refinement: APEX2; data reduction: SAINT (Bruker, 2005); program(s) used to solve structure: SHELXTL (Sheldrick, 2008); program(s) used to refine structure: SHELXTL; molecular graphics: SHELXTL; software used to prepare material for publication: SHELXTL, PLATON (Spek, 2003) and PARST95 (Nardelli, 1995).

HKF and RK thank the Malaysian Government and Universiti Sains Malaysia for the Science Fund (grant No. 305/PFIZIK/613312). RK thanks Universiti Sains Malaysia for the award of a post-doctoral research fellowship.

Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: IS2318).

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**supplementary materials**

*Acta Cryst.* (2008). E64, o1657-o1658 [ doi:10.1107/S1600536808023738 ]

## 4,4'-Dimethoxy-2,2'-[1,1'-(propane-1,3-diylidinitrilo)diethyldiyne]diphenol

H.-K. Fun and R. Kia

### Comment

The condensation of primary amines with carbonyl compounds yields Schiff base (Casellato & Vigato, 1977) that are still now regarded as one of the most potential group of chelators for facile preparations of metallo-organic hybrid materials. In the past two decades, the synthesis, structure and properties of Schiff base complexes have stimulated much interest for their noteworthy contributions in single molecule-based magnetism, materials science, catalysis of many reactions like carbonylation, hydroformylation, reduction, oxidation, epoxidation and hydrolysis, etc (Kia, Mirkhani, Kalman & Deak, 2007; Kia, Mirkhani, Harkema & van Hummel, 2007; Pal et al., 2005; Reglinski et al., 2004; Hou et al., 2001; Ren et al., 2002). This is due to the fact that Schiff bases offer opportunities for inducing substrate chirality, tuning the metal-centered electronic factor and enhancing the solubility and stability of either homogeneous or heterogeneous catalysts. Only a relatively small number of free Schiff base ligands have been characterized (Calligaris & Randaccio, 1987). As an extension of our work (Fun, Kargar & Kia, 2008; Fun, Kia & Kargar, 2008; Fun et al., 2008a,b) on the structural characterization of Schiff base compounds, the title compound (I), is reported here.

The molecule of the title compound, (I), has a crystallographic twofold rotation symmetry (Fig. 1). The bond lengths and angles are within normal ranges (Allen et al., 1987). The asymmetric unit of the compound is composed of one-half of the molecule. An intramolecular O—H $\cdots$ N hydrogen bond forms a six-membered ring, producing an S(6) ring motif (Bernstein et al. 1995). The two benzene rings are almost perpendicular to each other with a dihedral angle of 85.00 (2) $^{\circ}$ . The methoxy group is coplanar with the benzene ring, with the C10—O2—C4—C3 torsion angle of 2.34 (12) $^{\circ}$ . In the crystal structure neighbouring molecules are linked together by weak intermolecular C—H $\cdots$ O hydrogen bonds and a C—H $\cdots$  $\pi$  interaction to form a sheet parallel to the *ab* plane (Fig. 2). These molecules also adopt a zigzag arrangement along the *c* axis (Fig. 3).

### Experimental

The synthetic method has been described earlier (Reglinski *et al.*, 2004). Single crystals suitable for *X*-ray diffraction were obtained by evaporation of an ethanol solution at room temperature.

### Refinement

H atom bound to O1 was located from a difference Fourier map and refined as riding, with  $U_{\text{iso}}(\text{H}) = 1.5U_{\text{eq}}(\text{O})$ . Other H atoms were positioned geometrically (C—H = 0.93 – 0.96 Å) and refined using a riding model. A rotating-group model was applied for the methyl groups.

### Figures

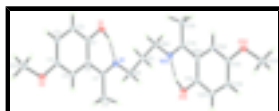


Fig. 1. The molecular structure of (I) with atom labels and 50% probability ellipsoids for non-H atoms. The suffix A corresponds to symmetry code  $(-x + 1, y, -z + 1/2)$ .

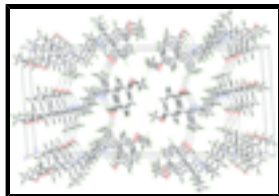


Fig. 2. The crystal packing, showing stacking of molecules down the *b* axis. Intramolecular and intermolecular interactions are shown as dashed lines.

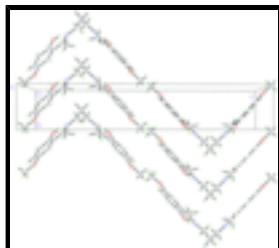


Fig. 3. The crystal packing, showing zigzag arrangement of molecules along the *c* axis.

**4,4'-Dimethoxy-2,2'-[1,1'-(propane-1,3-diylidinitrilo)diethylidyne]diphenol**

*Crystal data*

$C_{21}H_{26}N_2O_4$

$M_r = 370.44$

Monoclinic, *C2/c*

Hall symbol: -C 2yc

$a = 12.8042$  (2) Å

$b = 5.0508$  (1) Å

$c = 28.6019$  (6) Å

$\beta = 93.109$  (2)°

$V = 1847.00$  (6) Å<sup>3</sup>

$Z = 4$

$F_{000} = 792$

$D_x = 1.332$  Mg m<sup>-3</sup>

Mo  $K\alpha$  radiation

$\lambda = 0.71073$  Å

Cell parameters from 9900 reflections

$\theta = 2.9$ – $38.4$ °

$\mu = 0.09$  mm<sup>-1</sup>

$T = 100.0$  (1) K

Block, yellow

$0.47 \times 0.44 \times 0.29$  mm

*Data collection*

Bruker SMART APEXII CCD area-detector diffractometer

Radiation source: fine-focus sealed tube

Monochromator: graphite

$T = 100.0$ (1) K

$\varphi$  and  $\omega$  scans

Absorption correction: multi-scan (*SADABS*; Bruker, 2005)

$T_{\min} = 0.884$ ,  $T_{\max} = 0.974$

34801 measured reflections

5229 independent reflections

4304 reflections with  $I > 2\sigma(I)$

$R_{\text{int}} = 0.031$

$\theta_{\text{max}} = 38.8$ °

$\theta_{\text{min}} = 2.9$ °

$h = -22 \rightarrow 18$

$k = -8 \rightarrow 8$

$l = -48 \rightarrow 50$

*Refinement*

Refinement on  $F^2$

Least-squares matrix: full

Secondary atom site location: difference Fourier map

Hydrogen site location: inferred from neighbouring sites

$$R[F^2 > 2\sigma(F^2)] = 0.057$$

$$wR(F^2) = 0.148$$

$$S = 1.11$$

5229 reflections

125 parameters

Primary atom site location: structure-invariant direct methods

H-atom parameters constrained

$$w = 1/[\sigma^2(F_o^2) + (0.0571P)^2 + 1.5152P]$$

$$\text{where } P = (F_o^2 + 2F_c^2)/3$$

$$(\Delta/\sigma)_{\max} < 0.001$$

$$\Delta\rho_{\max} = 0.45 \text{ e } \text{\AA}^{-3}$$

$$\Delta\rho_{\min} = -0.28 \text{ e } \text{\AA}^{-3}$$

Extinction correction: none

### Special details

**Experimental.** The low-temperature data was collected with the Oxford Cyrosystem Cobra low-temperature attachment.

**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  $F^2 > \sigma(F^2)$  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 ( $\text{\AA}^2$ )

	$x$	$y$	$z$	$U_{\text{iso}}^*/U_{\text{eq}}$
O1	0.59948 (5)	0.57780 (14)	0.14698 (2)	0.01790 (13)
H1O1	0.5565	0.4795	0.1680	0.027*
O2	0.32409 (5)	1.27531 (14)	0.05669 (2)	0.01928 (13)
N1	0.44774 (6)	0.41178 (14)	0.19154 (2)	0.01406 (12)
C1	0.53083 (6)	0.74582 (16)	0.12471 (3)	0.01383 (13)
C2	0.56814 (6)	0.91899 (18)	0.09166 (3)	0.01619 (14)
H2A	0.6385	0.9140	0.0851	0.019*
C3	0.50202 (7)	1.09968 (17)	0.06815 (3)	0.01633 (14)
H3A	0.5281	1.2150	0.0463	0.020*
C4	0.39649 (6)	1.10628 (16)	0.07764 (3)	0.01411 (13)
C5	0.35797 (6)	0.93301 (16)	0.11033 (3)	0.01378 (13)
H5A	0.2873	0.9380	0.1162	0.017*
C6	0.42331 (6)	0.75110 (15)	0.13460 (3)	0.01242 (13)
C7	0.38248 (6)	0.57157 (16)	0.17015 (3)	0.01281 (13)
C8	0.41119 (7)	0.23770 (17)	0.22797 (3)	0.01519 (14)
H8A	0.3572	0.1216	0.2145	0.018*
H8B	0.3807	0.3436	0.2520	0.018*
C9	0.5000	0.0715 (2)	0.2500	0.01590 (19)
H9	0.4651	-0.0665	0.2746	0.019*
C10	0.35993 (8)	1.44822 (19)	0.02170 (3)	0.01964 (16)
H10A	0.3025	1.5528	0.0090	0.029*
H10B	0.3883	1.3457	-0.0029	0.029*

## supplementary materials

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H10C	0.4131	1.5628	0.0353	0.029*
C11	0.26922 (7)	0.57899 (19)	0.18136 (4)	0.01984 (16)
H11A	0.2370	0.4123	0.1733	0.030*
H11B	0.2343	0.7180	0.1638	0.030*
H11C	0.2639	0.6114	0.2142	0.030*

### Atomic displacement parameters ( $\text{\AA}^2$ )

	$U^{11}$	$U^{22}$	$U^{33}$	$U^{12}$	$U^{13}$	$U^{23}$
O1	0.0124 (2)	0.0187 (3)	0.0228 (3)	0.0024 (2)	0.0023 (2)	0.0063 (2)
O2	0.0159 (3)	0.0187 (3)	0.0233 (3)	0.0031 (2)	0.0012 (2)	0.0076 (2)
N1	0.0143 (3)	0.0140 (3)	0.0139 (3)	-0.0006 (2)	0.0013 (2)	0.0014 (2)
C1	0.0119 (3)	0.0142 (3)	0.0155 (3)	0.0007 (2)	0.0015 (2)	0.0006 (2)
C2	0.0130 (3)	0.0177 (3)	0.0181 (3)	0.0002 (3)	0.0032 (2)	0.0024 (3)
C3	0.0160 (3)	0.0162 (3)	0.0170 (3)	-0.0008 (3)	0.0028 (3)	0.0027 (3)
C4	0.0137 (3)	0.0132 (3)	0.0154 (3)	0.0011 (2)	0.0008 (2)	0.0010 (2)
C5	0.0120 (3)	0.0139 (3)	0.0155 (3)	0.0005 (2)	0.0015 (2)	0.0005 (2)
C6	0.0114 (3)	0.0124 (3)	0.0135 (3)	0.0001 (2)	0.0018 (2)	-0.0001 (2)
C7	0.0122 (3)	0.0125 (3)	0.0137 (3)	-0.0008 (2)	0.0013 (2)	-0.0009 (2)
C8	0.0156 (3)	0.0152 (3)	0.0147 (3)	-0.0021 (2)	0.0011 (2)	0.0011 (3)
C9	0.0191 (5)	0.0125 (4)	0.0161 (5)	0.000	0.0000 (4)	0.000
C10	0.0229 (4)	0.0173 (3)	0.0186 (4)	0.0016 (3)	0.0005 (3)	0.0046 (3)
C11	0.0134 (3)	0.0215 (4)	0.0250 (4)	0.0007 (3)	0.0045 (3)	0.0063 (3)

### Geometric parameters ( $\text{\AA}$ , $^\circ$ )

O1—C1	1.3556 (10)	C5—H5A	0.9300
O1—H1O1	0.9728	C6—C7	1.4787 (11)
O2—C4	1.3736 (10)	C7—C11	1.5025 (11)
O2—C10	1.4230 (11)	C8—C9	1.5221 (11)
N1—C7	1.2917 (11)	C8—H8A	0.9700
N1—C8	1.4597 (11)	C8—H8B	0.9700
C1—C2	1.3915 (12)	C9—C8 <sup>i</sup>	1.5221 (11)
C1—C6	1.4208 (11)	C9—H9	1.1014
C2—C3	1.3928 (12)	C10—H10A	0.9600
C2—H2A	0.9300	C10—H10B	0.9600
C3—C4	1.3929 (12)	C10—H10C	0.9600
C3—H3A	0.9300	C11—H11A	0.9600
C4—C5	1.3906 (12)	C11—H11B	0.9600
C5—C6	1.4010 (11)	C11—H11C	0.9600
C1—O1—H1O1	103.6	C6—C7—C11	120.84 (7)
C4—O2—C10	116.90 (7)	N1—C8—C9	111.49 (6)
C7—N1—C8	119.36 (7)	N1—C8—H8A	109.3
O1—C1—C2	118.35 (7)	C9—C8—H8A	109.3
O1—C1—C6	121.93 (7)	N1—C8—H8B	109.3
C2—C1—C6	119.71 (7)	C9—C8—H8B	109.3
C1—C2—C3	121.18 (8)	H8A—C8—H8B	108.0
C1—C2—H2A	119.4	C8—C9—C8 <sup>i</sup>	113.08 (10)

C3—C2—H2A	119.4	C8—C9—H9	107.1
C2—C3—C4	119.50 (8)	C8 <sup>i</sup> —C9—H9	113.8
C2—C3—H3A	120.3	O2—C10—H10A	109.5
C4—C3—H3A	120.3	O2—C10—H10B	109.5
O2—C4—C5	115.40 (7)	H10A—C10—H10B	109.5
O2—C4—C3	124.67 (7)	O2—C10—H10C	109.5
C5—C4—C3	119.94 (7)	H10A—C10—H10C	109.5
C4—C5—C6	121.46 (7)	H10B—C10—H10C	109.5
C4—C5—H5A	119.3	C7—C11—H11A	109.5
C6—C5—H5A	119.3	C7—C11—H11B	109.5
C5—C6—C1	118.21 (7)	H11A—C11—H11B	109.5
C5—C6—C7	121.26 (7)	C7—C11—H11C	109.5
C1—C6—C7	120.52 (7)	H11A—C11—H11C	109.5
N1—C7—C6	117.71 (7)	H11B—C11—H11C	109.5
N1—C7—C11	121.44 (7)		
O1—C1—C2—C3	-178.88 (8)	C2—C1—C6—C5	0.02 (12)
C6—C1—C2—C3	0.44 (13)	O1—C1—C6—C7	0.25 (12)
C1—C2—C3—C4	-0.42 (13)	C2—C1—C6—C7	-179.04 (8)
C10—O2—C4—C5	-177.58 (8)	C8—N1—C7—C6	177.96 (7)
C10—O2—C4—C3	2.34 (13)	C8—N1—C7—C11	-1.07 (12)
C2—C3—C4—O2	-179.99 (8)	C5—C6—C7—N1	-179.06 (8)
C2—C3—C4—C5	-0.07 (13)	C1—C6—C7—N1	-0.02 (11)
O2—C4—C5—C6	-179.53 (7)	C5—C6—C7—C11	-0.02 (12)
C3—C4—C5—C6	0.54 (13)	C1—C6—C7—C11	179.01 (8)
C4—C5—C6—C1	-0.51 (12)	C7—N1—C8—C9	-178.18 (7)
C4—C5—C6—C7	178.54 (7)	N1—C8—C9—C8 <sup>i</sup>	58.96 (5)
O1—C1—C6—C5	179.32 (8)		

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

*Hydrogen-bond geometry* ( $\text{\AA}, ^\circ$ )

<i>D</i> —H $\cdots$ <i>A</i>	<i>D</i> —H	H $\cdots$ <i>A</i>	<i>D</i> $\cdots$ <i>A</i>	<i>D</i> —H $\cdots$ <i>A</i>
O1—H1O1 $\cdots$ N1	0.97	1.62	2.5241 (10)	153
C11—H11A $\cdots$ O1 <sup>ii</sup>	0.96	2.53	3.4448 (12)	160
C11—H11B $\cdots$ O1 <sup>iii</sup>	0.96	2.53	3.4360 (12)	157
C10—H10C $\cdots$ Cg1 <sup>iv</sup>	0.96	2.68	3.5224 (10)	147

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





Fig. 2

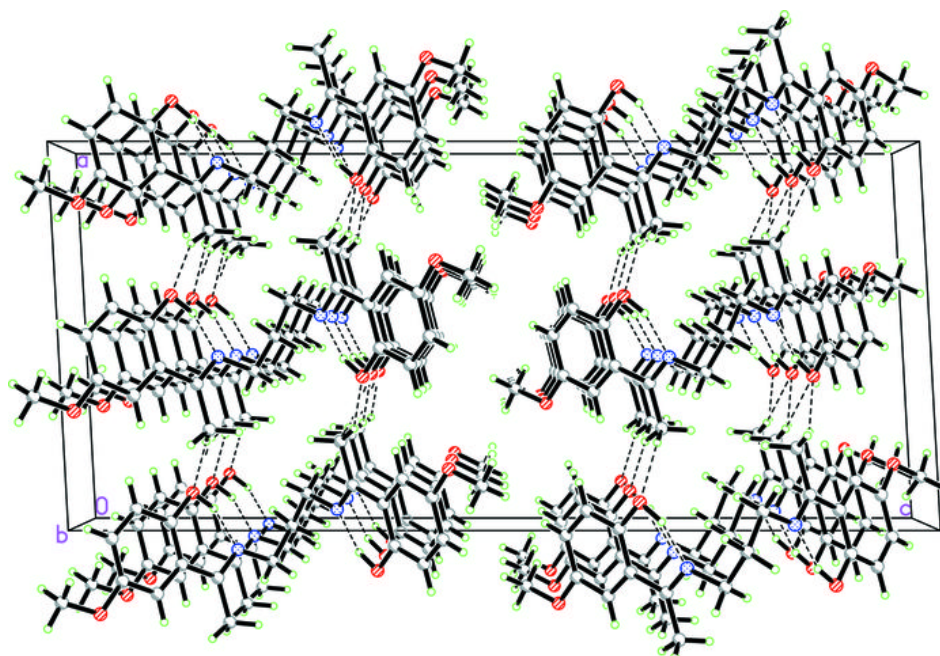


Fig. 3

