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## Structure Reports

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## 2-[(2-[(2-Hydroxy-5-methoxybenzylidene)amino]ethyl]imino)methyl]-4-methoxyphenol

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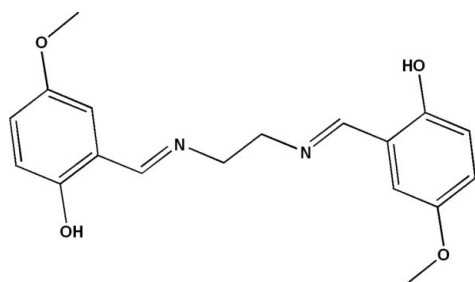
Received 23 April 2012; accepted 24 April 2012

Key indicators: single-crystal X-ray study;  $T = 295$  K; mean  $\sigma(\text{C}-\text{C}) = 0.002$  Å;  $R$  factor = 0.054;  $wR$  factor = 0.167; data-to-parameter ratio = 15.0.

The asymmetric unit of the title compound,  $\text{C}_{18}\text{H}_{20}\text{N}_2\text{O}_4$ , contains one-half molecule with an inversion center located at the centroid of the molecule. In the crystal, molecules are linked by  $\text{C}-\text{H}\cdots\pi$  interactions, forming layers parallel to (101). An intramolecular  $\text{O}-\text{H}\cdots\text{N}$  hydrogen bond also occurs.

### Related literature

For the synthesis of similar compounds see: Srinivasan *et al.* (1986); Moutet & Ourari (1997); Ourari *et al.* (2008).



### Experimental

#### Crystal data

$\text{C}_{18}\text{H}_{20}\text{N}_2\text{O}_4$   
 $M_r = 328.36$   
Monoclinic,  $P2_1/c$   
 $a = 15.0040$  (12) Å  
 $b = 5.9722$  (3) Å  
 $c = 9.3128$  (8) Å  
 $\beta = 92.001$  (3)°

$V = 833.98$  (11) Å<sup>3</sup>  
 $Z = 2$   
Mo  $K\alpha$  radiation  
 $\mu = 0.09$  mm<sup>-1</sup>  
 $T = 295$  K  
 $0.50 \times 0.23 \times 0.19$  mm

#### Data collection

Nonius KappaCCD diffractometer  
3001 measured reflections  
1664 independent reflections  
1097 reflections with  $I > 2\sigma(I)$   
 $R_{\text{int}} = 0.021$

#### Refinement

$R[F^2 > 2\sigma(F^2)] = 0.054$   
 $wR(F^2) = 0.167$   
 $S = 1.05$   
1664 reflections  
111 parameters  
H-atom parameters constrained  
 $\Delta\rho_{\text{max}} = 0.23$  e Å<sup>-3</sup>  
 $\Delta\rho_{\text{min}} = -0.16$  e Å<sup>-3</sup>

Table 1

Hydrogen-bond geometry (Å, °).

$C_g$  is the centroid of the C4–C9 ring.

$D-H\cdots A$	$D-H$	$H\cdots A$	$D\cdots A$	$D-H\cdots A$
$O5-H5\cdots N2$	0.82	1.85	2.5844 (18)	148
$C10-H10C\cdots C_g^i$	0.96	2.64	3.521 (2)	152

Symmetry code: (i)  $x, -y + \frac{1}{2}, z - \frac{3}{2}$ .

Data collection: *COLLECT* (Nonius, 1998); cell refinement: *SCALEPACK* (Otwinowski & Minor, 1997); data reduction: *DENZO* (Otwinowski & Minor 1997) and *SCALEPACK*; program(s) used to solve structure: *SIR2002* (Burla *et al.*, 2005); program(s) used to refine structure: *SHELXL97* (Sheldrick, 2008); molecular graphics: *ORTEP-3 for Windows* (Farrugia, 1997) and *DIAMOND* (Brandenburg & Berndt, 2001); software used to prepare material for publication: *WinGX* (Farrugia, 1999).

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Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: BQ2353).

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## supplementary materials

*Acta Cryst.* (2012). E68, o1760 [doi:10.1107/S1600536812018405]

## 2-[(2-[(2-Hydroxy-5-methoxybenzylidene)amino]ethyl)imino)methyl]-4-methoxyphenol

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### Comment

The tetradentate Schiff base ligands derived from salicylaldehyde derivatives and diamino compounds have been found to be excellent chelating agents for most applications in coordination chemistry such as in catalysis (Srinivasan *et al.*, 1986) and electrocatalysis (Moutet & Ourari, 1997; Ourari *et al.*, 2008). Here, we report the synthesis of the title compound and its crystal structure.

The molecular structure of (I), and the atomic numbering used, is illustrated in Fig. 1. The asymmetric unit of the title compound, consists of one-half of the molecule, with the other half generated by a crystallographic inversion center. The crystal packing in the title structure can be described by a zigzag layers parallel to (101) plane (Fig. 2). There is one intramolecular O—H $\cdots$ N hydrogen bonding in this packing (Table 1, Fig. 2), which it is stabilized C—H $\cdots$  $\pi$  and Van der Waals interactions (table 1) All these interactions link the molecules within the layers and also link the layers together and reinforcing the cohesion of the structure.

### Experimental

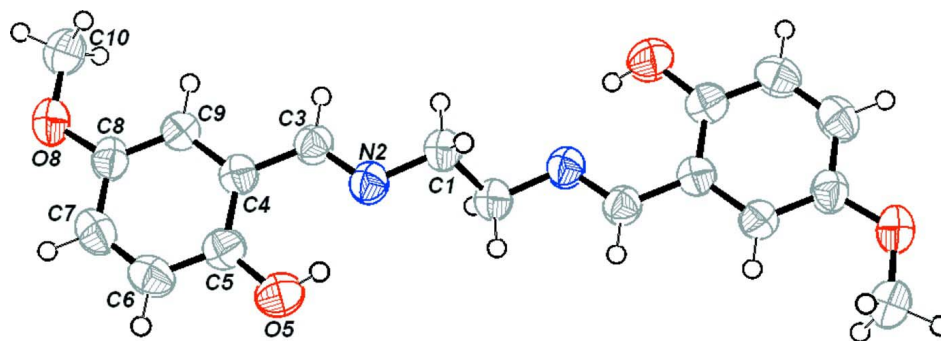
60 mg of 1,2-diaminoethane (1 mmol) were dissolved in 10 ml of absolute ethanol. This solution was drop wise added, under stirring, to an ethanolic solution (10 ml) containing 304 mg of 5-methoxysalicylaldehyde (2 mmol). This mixture was refluxed for 1 h after which a yellow precipitate is formed, recovered by filtration, washed several times with diethyl oxide and dried to yield 282 mg (86%) of the title compound. The suitable crystals for X-ray analysis were obtained by slow evaporation from a mixture of solvents ethanol/dichloromethane (8/2, v/v).

### Refinement

The H atoms were localized on Fourier maps but introduced in calculated positions and treated as riding on their parent atoms (C and O) with C—H = 0.93 Å (methine, aromatic), 0.96 Å (methyl), 0.97 Å (methylene) and O—H = 0.82 Å (hydroxyl) with  $U_{\text{iso}}(\text{H}) = 1.2U_{\text{eq}}(\text{C})$  or  $U_{\text{iso}}(\text{H}) = 1.5U_{\text{eq}}(\text{O})$ .

### Computing details

Data collection: *COLLECT* (Nonius, 1998); cell refinement: *SCALEPACK* (Otwinowski & Minor, 1997); data reduction: *DENZO* and *SCALEPACK* (Otwinowski & Minor 1997); program(s) used to solve structure: *SIR2002* (Burla *et al.*, 2005); program(s) used to refine structure: *SHELXL97* (Sheldrick, 2008); molecular graphics: *ORTEP-3 for Windows* (Farrugia, 1997) and *DIAMOND* (Brandenburg & Berndt, 2001); software used to prepare material for publication: *WinGX* (Farrugia, 1999).


**Figure 1**

The molecular geometry of (I) with the atom-labeling scheme. Displacement ellipsoids are drawn at the 50% probability level. Only the contents of the asymmetric unit are numbered.

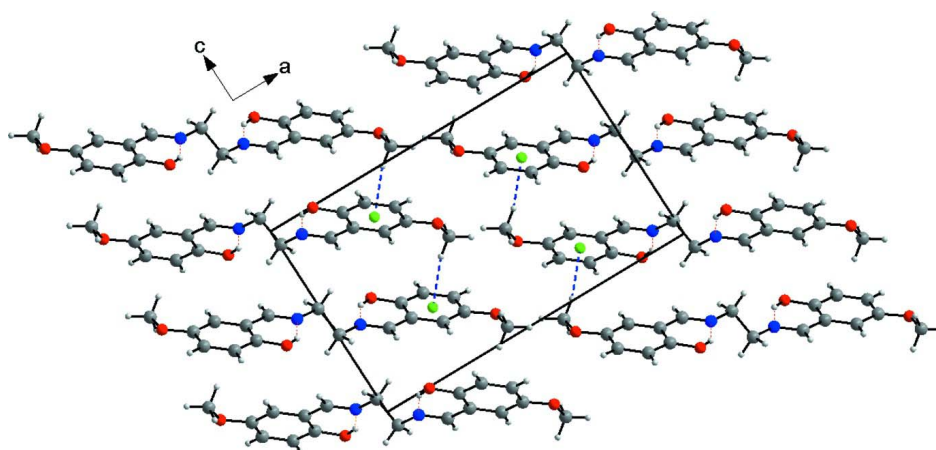

**Figure 2**

Diagram of layered packing parallel to (101) plane and showing O—H...N and C—H... $\pi$  interactions.

### 2-[(2-[(2-Hydroxy-5-methoxybenzylidene)amino]ethyl)imino)methyl]- 4-methoxyphenol

#### Crystal data

$C_{18}H_{20}N_2O_4$

$M_r = 328.36$

Monoclinic,  $P2_1/c$

Hall symbol:  $-P\ 2_1/c$

$a = 15.0040$  (12) Å

$b = 5.9722$  (3) Å

$c = 9.3128$  (8) Å

$\beta = 92.001$  (3)°

$V = 833.98$  (11) Å<sup>3</sup>

$Z = 2$

$F(000) = 348$

$D_x = 1.308$  Mg m<sup>-3</sup>

Mo  $K\alpha$  radiation,  $\lambda = 0.71073$  Å

Cell parameters from 1761 reflections

$\theta = 1.0$ – $26.4$ °

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

$T = 295$  K

Prism, colorless

$0.50 \times 0.23 \times 0.19$  mm

#### Data collection

Nonius KappaCCD  
diffractometer

Radiation source: Enraf–Nonius FR590

Graphite monochromator

Detector resolution: 9 pixels mm<sup>-1</sup>

CCD rotation images, thick slices scans

3001 measured reflections

1664 independent reflections

1097 reflections with  $I > 2\sigma(I)$

$R_{int} = 0.021$

$\theta_{\max} = 26.4^\circ$ ,  $\theta_{\min} = 2.7^\circ$   
 $h = -18 \rightarrow 18$

$k = -6 \rightarrow 7$   
 $l = -11 \rightarrow 11$

*Refinement*

Refinement on  $F^2$   
 Least-squares matrix: full  
 $R[F^2 > 2\sigma(F^2)] = 0.054$   
 $wR(F^2) = 0.167$   
 $S = 1.05$   
 1664 reflections  
 111 parameters  
 0 restraints  
 Primary atom site location: structure-invariant  
 direct methods  
 Secondary atom site location: difference Fourier  
 map

Hydrogen site location: inferred from  
 neighbouring sites  
 H-atom parameters constrained  
 $w = 1/[\sigma^2(F_o^2) + (0.101P)^2 + 0.0006P]$   
 where  $P = (F_o^2 + 2F_c^2)/3$   
 $(\Delta/\sigma)_{\max} < 0.001$   
 $\Delta\rho_{\max} = 0.23 \text{ e } \text{\AA}^{-3}$   
 $\Delta\rho_{\min} = -0.16 \text{ e } \text{\AA}^{-3}$   
 Extinction correction: *SHELXL97* (Sheldrick,  
 2008),  $F_c^* = kFc[1 + 0.001x \text{Fc}^2 \lambda^3 / \sin(2\theta)]^{-1/4}$   
 Extinction coefficient: 0.08 (2)

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

	<i>x</i>	<i>y</i>	<i>z</i>	$U_{\text{iso}}^*/U_{\text{eq}}$
C1	0.00544 (12)	0.5332 (3)	0.42227 (18)	0.0554 (5)
H1A	0.0237	0.404	0.3674	0.066*
H1B	-0.0511	0.586	0.3816	0.066*
C3	0.13689 (11)	0.6835 (3)	0.32973 (18)	0.0500 (5)
H3	0.1399	0.5537	0.2749	0.06*
C4	0.20642 (11)	0.8520 (3)	0.31789 (17)	0.0488 (5)
C5	0.20521 (13)	1.0483 (3)	0.40154 (19)	0.0545 (5)
C6	0.27360 (14)	1.2037 (3)	0.3894 (2)	0.0620 (5)
H6	0.2731	1.3341	0.4439	0.074*
C7	0.34154 (14)	1.1672 (3)	0.2983 (2)	0.0623 (5)
H7	0.3863	1.2739	0.2909	0.075*
C8	0.34469 (12)	0.9719 (3)	0.21605 (18)	0.0542 (5)
C9	0.27715 (11)	0.8166 (3)	0.22553 (18)	0.0515 (5)
H9	0.2784	0.687	0.1702	0.062*
C10	0.42592 (14)	0.7497 (3)	0.0532 (3)	0.0764 (7)
H10A	0.4313	0.6263	0.119	0.115*
H10B	0.4781	0.7572	-0.0033	0.115*
H10C	0.3742	0.7287	-0.0089	0.115*
N2	0.07208 (9)	0.7091 (2)	0.41315 (16)	0.0544 (5)
O5	0.13958 (10)	1.0889 (2)	0.49361 (15)	0.0712 (5)
H5	0.1037	0.9853	0.4903	0.107*
O8	0.41724 (10)	0.9514 (2)	0.13122 (15)	0.0727 (5)

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

	$U^{11}$	$U^{22}$	$U^{33}$	$U^{12}$	$U^{13}$	$U^{23}$
C1	0.0480 (10)	0.0591 (10)	0.0594 (11)	-0.0045 (7)	0.0065 (8)	-0.0027 (8)
C3	0.0467 (10)	0.0539 (9)	0.0494 (10)	0.0010 (7)	0.0029 (8)	-0.0004 (7)
C4	0.0494 (10)	0.0483 (9)	0.0486 (10)	0.0007 (7)	0.0001 (8)	0.0034 (6)
C5	0.0598 (12)	0.0495 (10)	0.0541 (10)	0.0057 (8)	0.0025 (9)	0.0020 (7)
C6	0.0718 (13)	0.0466 (9)	0.0672 (12)	-0.0003 (8)	-0.0038 (10)	-0.0033 (8)
C7	0.0652 (12)	0.0518 (10)	0.0696 (12)	-0.0104 (8)	-0.0025 (10)	0.0063 (9)
C8	0.0517 (11)	0.0589 (10)	0.0521 (10)	-0.0062 (7)	0.0040 (8)	0.0086 (7)
C9	0.0546 (11)	0.0514 (9)	0.0488 (10)	-0.0030 (7)	0.0044 (9)	-0.0013 (7)
C10	0.0628 (13)	0.0883 (14)	0.0793 (15)	-0.0105 (10)	0.0209 (11)	-0.0118 (11)
N2	0.0486 (9)	0.0571 (9)	0.0578 (9)	-0.0012 (6)	0.0068 (7)	-0.0004 (6)
O5	0.0726 (10)	0.0631 (8)	0.0789 (10)	0.0062 (6)	0.0182 (8)	-0.0140 (6)
O8	0.0643 (9)	0.0764 (9)	0.0786 (10)	-0.0194 (7)	0.0215 (7)	-0.0033 (7)

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

C1—N2	1.455 (2)	C6—H6	0.93
C1—C1 <sup>i</sup>	1.515 (3)	C7—C8	1.397 (2)
C1—H1A	0.97	C7—H7	0.93
C1—H1B	0.97	C8—O8	1.373 (2)
C3—N2	1.275 (2)	C8—C9	1.379 (2)
C3—C4	1.456 (2)	C9—H9	0.93
C3—H3	0.93	C10—O8	1.415 (2)
C4—C9	1.405 (2)	C10—H10A	0.96
C4—C5	1.408 (2)	C10—H10B	0.96
C5—O5	1.350 (2)	C10—H10C	0.96
C5—C6	1.391 (3)	O5—H5	0.82
C6—C7	1.366 (3)		
N2—C1—C1 <sup>i</sup>	109.99 (18)	C6—C7—C8	120.91 (16)
N2—C1—H1A	109.7	C6—C7—H7	119.5
C1 <sup>i</sup> —C1—H1A	109.7	C8—C7—H7	119.5
N2—C1—H1B	109.7	O8—C8—C9	125.19 (16)
C1 <sup>i</sup> —C1—H1B	109.7	O8—C8—C7	115.64 (15)
H1A—C1—H1B	108.2	C9—C8—C7	119.18 (17)
N2—C3—C4	121.80 (15)	C8—C9—C4	120.69 (16)
N2—C3—H3	119.1	C8—C9—H9	119.7
C4—C3—H3	119.1	C4—C9—H9	119.7
C9—C4—C5	119.26 (16)	O8—C10—H10A	109.5
C9—C4—C3	120.03 (15)	O8—C10—H10B	109.5
C5—C4—C3	120.68 (16)	H10A—C10—H10B	109.5
O5—C5—C6	119.26 (16)	O8—C10—H10C	109.5
O5—C5—C4	121.64 (16)	H10A—C10—H10C	109.5
C6—C5—C4	119.10 (17)	H10B—C10—H10C	109.5
C7—C6—C5	120.86 (16)	C3—N2—C1	119.31 (15)
C7—C6—H6	119.6	C5—O5—H5	109.5
C5—C6—H6	119.6	C8—O8—C10	117.41 (14)

N2—C3—C4—C9	179.47 (16)	C6—C7—C8—C9	1.2 (3)
N2—C3—C4—C5	1.4 (3)	O8—C8—C9—C4	178.87 (17)
C9—C4—C5—O5	-178.94 (15)	C7—C8—C9—C4	-0.8 (3)
C3—C4—C5—O5	-0.9 (3)	C5—C4—C9—C8	-0.2 (2)
C9—C4—C5—C6	0.8 (2)	C3—C4—C9—C8	-178.27 (15)
C3—C4—C5—C6	178.82 (15)	C4—C3—N2—C1	-178.95 (15)
O5—C5—C6—C7	179.38 (17)	C1 <sup>i</sup> —C1—N2—C3	127.0 (2)
C4—C5—C6—C7	-0.3 (3)	C9—C8—O8—C10	-3.6 (3)
C5—C6—C7—C8	-0.7 (3)	C7—C8—O8—C10	176.06 (17)
C6—C7—C8—O8	-178.45 (17)		

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

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

C<sub>g</sub> is the centroid of the C4—C9 ring.

<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>
O5—H5 $\cdots$ N2	0.82	1.85	2.5844 (18)	148
C10—H10C $\cdots$ C <sub>g</sub> <sup>ii</sup>	0.96	2.64	3.521 (2)	152

Symmetry code: (ii)  $x, -y+1/2, z-3/2$ .