

Acta Crystallographica Section E

## Structure Reports

Online

ISSN 1600-5368

## (E)-2-[(2-Hydroxyethyl)iminomethyl]-6-methoxyphenolate

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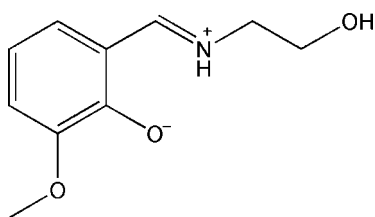
Received 13 February 2009; accepted 13 February 2009

 Key indicators: single-crystal X-ray study;  $T = 295$  K; mean  $\sigma(\text{C}-\text{C}) = 0.004$  Å;  $R$  factor = 0.034;  $wR$  factor = 0.084; data-to-parameter ratio = 8.2.

The title Schiff base compound,  $\text{C}_{10}\text{H}_{13}\text{NO}_3$ , obtained by the reaction of 2-hydroxy-3-methoxybenzaldehyde and 2-aminoethanol in methanol solution, crystallizes in a zwitterionic form, in which the molecule adopts a *trans* configuration about the central  $\text{C}=\text{N}$  bond. An intramolecular  $\text{N}-\text{H}\cdots\text{O}$  hydrogen bond occurs. In the crystal structure, molecules are linked into chains by intermolecular  $\text{O}-\text{H}\cdots\text{O}$  hydrogen bonding.

### Related literature

For related structures, see: Cui *et al.* (1999); Dong *et al.* (2007); Li *et al.* (2005); Ng (2008); Oshio *et al.* (2003); Sun *et al.* (2006). For reference structural data, see: Allen *et al.* (1987).



### Experimental

#### Crystal data

 $\text{C}_{10}\text{H}_{13}\text{NO}_3$ 
 $M_r = 195.21$ 

 Orthorhombic,  $Pca2_1$ 
 $a = 14.148$  (6) Å

 $b = 6.587$  (3) Å

 $c = 10.760$  (4) Å

 $V = 1002.8$  (7) Å<sup>3</sup>
 $Z = 4$ 

 Mo  $K\alpha$  radiation

 $\mu = 0.10$  mm<sup>-1</sup>
 $T = 295$  K

 $0.30 \times 0.30 \times 0.12$  mm

#### Data collection

Bruker SMART APEX CCD diffractometer

Absorption correction: multi-scan (SADABS; Bruker, 2002)

 $T_{\min} = 0.974$ ,  $T_{\max} = 0.991$ 

7345 measured reflections

1041 independent reflections

 923 reflections with  $I > 2\sigma(I)$ 
 $R_{\text{int}} = 0.037$ 

#### Refinement

 $R[F^2 > 2\sigma(F^2)] = 0.034$ 
 $wR(F^2) = 0.084$ 
 $S = 1.07$ 

1041 reflections

127 parameters

1 restraint

H-atom parameters constrained

 $\Delta\rho_{\text{max}} = 0.09$  e Å<sup>-3</sup>
 $\Delta\rho_{\text{min}} = -0.15$  e Å<sup>-3</sup>
**Table 1**

Hydrogen-bond geometry (Å, °).

$D-H\cdots A$	$D-H$	$H\cdots A$	$D\cdots A$	$D-H\cdots A$
$\text{N1}-\text{H1}\cdots\text{O1}$	0.86	1.95	2.617 (2)	134
$\text{O3}-\text{H3}\cdots\text{O1}^{\dagger}$	0.82	1.95	2.741 (3)	161

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

Data collection: SMART (Bruker, 2002); cell refinement: SAINT (Bruker, 2002); data reduction: SAINT; program(s) used to solve structure: SHELXS97 (Sheldrick, 2008); program(s) used to refine structure: SHELXL97 (Sheldrick, 2008); molecular graphics: ORTEP-3 for Windows (Farrugia, 1997); software used to prepare material for publication: SHELXL97.

This work was supported by the Key Laboratory of Colloid Interface Chemistry of the Ministry of Education (200707).

Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: HB2911).

### References

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## supporting information

*Acta Cryst.* (2009). E65, o559 [doi:10.1107/S1600536809005297]

**(E)-2-[(2-Hydroxyethyl)iminiomethyl]-6-methoxyphenolate****Guo-Xia Tan and Xi-Cheng Liu****S1. Comment**

The title compound, (I), derived from 3-methoxy-2-hydroxybenzaldehyde and 2-aminoethanol, is a potential NO<sub>3</sub> tetradentate Schiff base ligand and its complexes with Cd(II), Cu(II), Zn(II) and Fe(III) have been reported (Cui *et al.*, 1999; Dong *et al.*, 2007; Li *et al.*, 2005; Oshio *et al.*, 2003). Here, the structure of (I) is described.

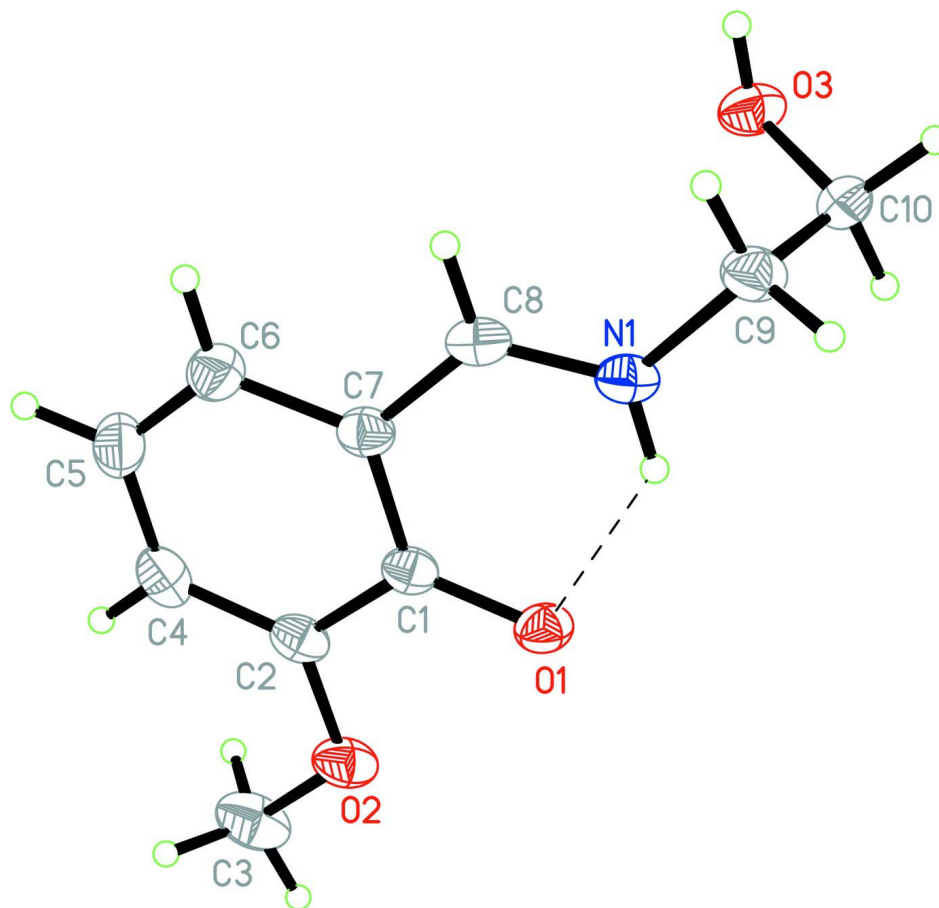
The title molecule exists in a zwitterionic form with a strong intramolecular N—H···O hydrogen bond (Table 1) between the NH<sup>+</sup> and the phenolate O<sup>-</sup>, as shown in Fig. 1. The bond lengths and angles are within normal ranges (Allen *et al.*, 1987). The N1=C8 [1.294 (3) Å] and N1—C8 [1.453 (3) Å] bond distances are comparable to these found in similar Schiff base compounds, such as 2,4-Dibromo-6-(2-hydroxyethyliminiomethyl)-phenolate [1.277 (5) and 1.451 (4) Å] (Sun *et al.*, 2006) and 4-chloro-2-[tris(hydroxymethyl)methyliminiomethyl]phenolate [1.288 (2) and 1.467 (2) Å] (Ng, 2008). As expected, the molecule adopts a *trans* configuration about the central C=N bond. In the crystal structure, O3—H3···O1<sup>i</sup> (symmetry code as given in Table 1) intermolecular hydrogen bonds formed between the hydroxy and oxygen of phenolate link the molecules into a one-dimension supramolecular chain.

**S2. Experimental**

3-Methoxy-2-hydroxybenzaldehyde (0.152 g, 1 mmol) and equimolar 2-aminoethanol (0.061 g, 1 mmol) were refluxed for 30 min in methanol solution (15 ml). The reaction mixtures were cooled to room temperature and filtered. After keeping the filtrate in air for 3 d, yellow blocks of (I) (yield 66%; mp 338–339 K) were obtained.

**S3. Refinement**

H atoms were placed at calculated positions and refined in the riding-model approximation, with C—H = 0.93 Å and  $U_{\text{iso}}(\text{H}) = 1.2U_{\text{eq}}(\text{C})$  for *sp*<sup>2</sup> H atoms, C—H = 0.96 Å and  $U_{\text{iso}}(\text{H}) = 1.5U_{\text{eq}}(\text{C})$  for methyl H atoms, C—H = 0.97 Å and  $U_{\text{iso}}(\text{H}) = 1.2U_{\text{eq}}(\text{C})$  for methylene H atoms, N—H = 0.86 Å and  $U_{\text{iso}}(\text{H}) = 1.2U_{\text{eq}}(\text{C})$  for imino group, and O—H = 0.82 Å and  $U_{\text{iso}}(\text{H}) = 1.5U_{\text{eq}}(\text{C})$  for hydroxy. Friedel pairs were merged.

**Figure 1**

The structure of (I) with displacement ellipsoids drawn at the 50% probability level. The N—H...O hydrogen bond is shown as a dashed line.

### (E)-2-[(2-Hydroxyethyl)iminiomethyl]-6-methoxyphenolate

#### Crystal data

$C_{10}H_{13}NO_3$   
 $M_r = 195.21$   
 Orthorhombic,  $Pca2_1$   
 Hall symbol: P 2c -2ac  
 $a = 14.148 (6) \text{ \AA}$   
 $b = 6.587 (3) \text{ \AA}$   
 $c = 10.760 (4) \text{ \AA}$   
 $V = 1002.8 (7) \text{ \AA}^3$   
 $Z = 4$

$F(000) = 416$   
 $D_x = 1.293 \text{ Mg m}^{-3}$   
 Mo  $K\alpha$  radiation,  $\lambda = 0.71073 \text{ \AA}$   
 Cell parameters from 1828 reflections  
 $\theta = 2.9\text{--}20.4^\circ$   
 $\mu = 0.10 \text{ mm}^{-1}$   
 $T = 295 \text{ K}$   
 Block, yellow  
 $0.30 \times 0.30 \times 0.12 \text{ mm}$

#### Data collection

Bruker APEX CCD  
 diffractometer  
 Radiation source: fine-focus sealed tube  
 Graphite monochromator  
 $\varphi$  and  $\omega$  scans

Absorption correction: multi-scan  
 (SADABS; Bruker, 2002)  
 $T_{\min} = 0.974$ ,  $T_{\max} = 0.991$   
 7345 measured reflections  
 1041 independent reflections  
 923 reflections with  $I > 2\sigma(I)$

$R_{\text{int}} = 0.037$   
 $\theta_{\text{max}} = 26.0^\circ$ ,  $\theta_{\text{min}} = 2.9^\circ$   
 $h = -17 \rightarrow 17$

$k = -8 \rightarrow 8$   
 $l = -12 \rightarrow 13$

*Refinement*

Refinement on  $F^2$   
 Least-squares matrix: full  
 $R[F^2 > 2\sigma(F^2)] = 0.034$   
 $wR(F^2) = 0.084$   
 $S = 1.07$   
 1041 reflections  
 127 parameters  
 1 restraint  
 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  
 $w = 1/[\sigma^2(F_o^2) + (0.0473P)^2 + 0.0337P]$   
 where  $P = (F_o^2 + 2F_c^2)/3$   
 $(\Delta/\sigma)_{\text{max}} < 0.001$   
 $\Delta\rho_{\text{max}} = 0.09 \text{ e } \text{\AA}^{-3}$   
 $\Delta\rho_{\text{min}} = -0.15 \text{ e } \text{\AA}^{-3}$

*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 ( $\text{\AA}^2$ )*

	$x$	$y$	$z$	$U_{\text{iso}}^*/U_{\text{eq}}$
C1	0.53278 (16)	0.7444 (3)	0.0860 (2)	0.0408 (5)
C2	0.57651 (16)	0.9338 (3)	0.0535 (2)	0.0451 (6)
C3	0.69788 (18)	1.1717 (4)	0.1022 (3)	0.0718 (9)
H3A	0.6519	1.2763	0.1164	0.108*
H3B	0.7207	1.1807	0.0183	0.108*
H3C	0.7497	1.1885	0.1588	0.108*
C4	0.5396 (2)	1.0535 (4)	-0.0390 (3)	0.0557 (7)
H4	0.5680	1.1776	-0.0566	0.067*
C5	0.4599 (2)	0.9927 (5)	-0.1078 (3)	0.0625 (8)
H5	0.4366	1.0750	-0.1710	0.075*
C6	0.41744 (18)	0.8135 (4)	-0.0814 (3)	0.0570 (7)
H6	0.3655	0.7718	-0.1278	0.068*
C7	0.45145 (15)	0.6891 (4)	0.0160 (2)	0.0436 (5)
C8	0.39971 (16)	0.5130 (4)	0.0485 (2)	0.0460 (6)
H8	0.3479	0.4790	-0.0006	0.055*
C9	0.36073 (17)	0.2262 (4)	0.1804 (3)	0.0535 (6)
H9A	0.4005	0.1091	0.1961	0.064*
H9B	0.3173	0.1915	0.1140	0.064*
C10	0.30532 (16)	0.2758 (4)	0.2962 (3)	0.0529 (6)
H10A	0.2717	0.1553	0.3232	0.064*
H10B	0.3490	0.3137	0.3617	0.064*
N1	0.41934 (14)	0.3958 (3)	0.1415 (2)	0.0478 (5)

H1	0.4701	0.4199	0.1829	0.057*
O1	0.56592 (11)	0.6322 (2)	0.17429 (18)	0.0486 (4)
O2	0.65545 (11)	0.9784 (3)	0.1213 (2)	0.0564 (5)
O3	0.23996 (12)	0.4345 (2)	0.2785 (2)	0.0584 (5)
H3	0.1946	0.3924	0.2387	0.088*

*Atomic displacement parameters (Å<sup>2</sup>)*

	$U^{11}$	$U^{22}$	$U^{33}$	$U^{12}$	$U^{13}$	$U^{23}$
C1	0.0358 (11)	0.0387 (12)	0.0478 (13)	0.0046 (9)	0.0063 (10)	-0.0074 (11)
C2	0.0393 (12)	0.0407 (13)	0.0553 (16)	0.0030 (10)	0.0127 (11)	-0.0106 (12)
C3	0.0623 (17)	0.0421 (15)	0.111 (3)	-0.0143 (14)	0.0175 (19)	-0.0154 (16)
C4	0.0608 (17)	0.0435 (14)	0.0629 (17)	0.0038 (13)	0.0206 (15)	0.0050 (13)
C5	0.0670 (18)	0.0703 (19)	0.0502 (16)	0.0090 (15)	0.0064 (14)	0.0132 (15)
C6	0.0507 (14)	0.0726 (18)	0.0477 (15)	0.0022 (13)	-0.0017 (12)	0.0014 (14)
C7	0.0378 (12)	0.0484 (13)	0.0445 (13)	0.0014 (11)	0.0028 (10)	-0.0071 (12)
C8	0.0341 (12)	0.0521 (14)	0.0520 (14)	0.0004 (10)	-0.0026 (11)	-0.0117 (12)
C9	0.0420 (13)	0.0424 (13)	0.0760 (18)	-0.0020 (10)	-0.0002 (14)	0.0002 (13)
C10	0.0422 (12)	0.0533 (15)	0.0633 (16)	0.0033 (11)	-0.0071 (12)	0.0138 (13)
N1	0.0354 (10)	0.0481 (12)	0.0600 (14)	-0.0036 (9)	-0.0025 (9)	-0.0050 (11)
O1	0.0400 (8)	0.0440 (9)	0.0619 (11)	-0.0040 (7)	-0.0082 (8)	0.0003 (9)
O2	0.0436 (9)	0.0433 (9)	0.0822 (14)	-0.0083 (8)	0.0020 (9)	-0.0062 (9)
O3	0.0410 (9)	0.0591 (10)	0.0750 (13)	0.0088 (9)	-0.0069 (9)	-0.0038 (10)

*Geometric parameters (Å, °)*

C1—O1	1.291 (3)	C6—H6	0.9300
C1—C7	1.423 (3)	C7—C8	1.416 (3)
C1—C2	1.436 (3)	C8—N1	1.294 (3)
C2—O2	1.366 (3)	C8—H8	0.9300
C2—C4	1.373 (4)	C9—N1	1.453 (3)
C3—O2	1.422 (3)	C9—C10	1.508 (4)
C3—H3A	0.9600	C9—H9A	0.9700
C3—H3B	0.9600	C9—H9B	0.9700
C3—H3C	0.9600	C10—O3	1.409 (3)
C4—C5	1.407 (4)	C10—H10A	0.9700
C4—H4	0.9300	C10—H10B	0.9700
C5—C6	1.355 (4)	N1—H1	0.8600
C5—H5	0.9300	O3—H3	0.8200
C6—C7	1.415 (4)		
O1—C1—C7	122.5 (2)	C6—C7—C1	121.3 (2)
O1—C1—C2	121.3 (2)	C8—C7—C1	119.8 (2)
C7—C1—C2	116.2 (2)	N1—C8—C7	124.6 (2)
O2—C2—C4	125.1 (2)	N1—C8—H8	117.7
O2—C2—C1	114.1 (2)	C7—C8—H8	117.7
C4—C2—C1	120.8 (2)	N1—C9—C10	111.6 (2)
O2—C3—H3A	109.5	N1—C9—H9A	109.3

O2—C3—H3B	109.5	C10—C9—H9A	109.3
H3A—C3—H3B	109.5	N1—C9—H9B	109.3
O2—C3—H3C	109.5	C10—C9—H9B	109.3
H3A—C3—H3C	109.5	H9A—C9—H9B	108.0
H3B—C3—H3C	109.5	O3—C10—C9	113.0 (2)
C2—C4—C5	121.5 (2)	O3—C10—H10A	109.0
C2—C4—H4	119.2	C9—C10—H10A	109.0
C5—C4—H4	119.2	O3—C10—H10B	109.0
C6—C5—C4	119.5 (3)	C9—C10—H10B	109.0
C6—C5—H5	120.2	H10A—C10—H10B	107.8
C4—C5—H5	120.2	C8—N1—C9	124.0 (2)
C5—C6—C7	120.6 (3)	C8—N1—H1	118.0
C5—C6—H6	119.7	C9—N1—H1	118.0
C7—C6—H6	119.7	C2—O2—C3	117.4 (2)
C6—C7—C8	118.8 (2)	C10—O3—H3	109.5
O1—C1—C2—O2	1.6 (3)	C2—C1—C7—C6	1.1 (3)
C7—C1—C2—O2	-178.65 (19)	O1—C1—C7—C8	4.8 (3)
O1—C1—C2—C4	-178.8 (2)	C2—C1—C7—C8	-175.0 (2)
C7—C1—C2—C4	1.0 (3)	C6—C7—C8—N1	-174.5 (2)
O2—C2—C4—C5	177.5 (2)	C1—C7—C8—N1	1.8 (3)
C1—C2—C4—C5	-2.1 (3)	N1—C9—C10—O3	63.7 (3)
C2—C4—C5—C6	1.0 (4)	C7—C8—N1—C9	174.2 (2)
C4—C5—C6—C7	1.2 (4)	C10—C9—N1—C8	-104.5 (3)
C5—C6—C7—C8	174.0 (2)	C4—C2—O2—C3	5.8 (3)
C5—C6—C7—C1	-2.2 (4)	C1—C2—O2—C3	-174.6 (2)
O1—C1—C7—C6	-179.1 (2)		

Hydrogen-bond geometry (Å, °)

<i>D</i> —H... <i>A</i>	<i>D</i> —H	H... <i>A</i>	<i>D</i> ... <i>A</i>	<i>D</i> —H... <i>A</i>
N1—H1...O1	0.86	1.95	2.617 (2)	134
O3—H3...O1 <sup>i</sup>	0.82	1.95	2.741 (3)	161

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