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

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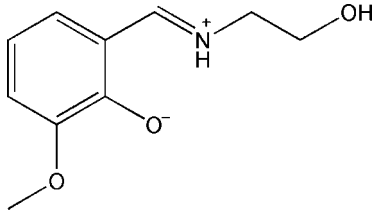
Key indicators: single-crystal X-ray study; T = 295 K; mean σ(C–C) = 0.004 Å; R factor = 0.034; wR factor = 0.084; data-to-parameter ratio = 8.2.

The title Schiff base compound, $C_{10}H_{13}NO_3$, 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 C=N bond. An intramolecular N—H···O hydrogen bond occurs. In the crystal structure, molecules are linked into chains by intermolecular O—H···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

$C_{10}H_{13}NO_3$
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^3
Z = 4
Mo $K\alpha$ radiation
μ = 0.10 mm^{-1}
T = 295 K
0.30 × 0.30 × 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_{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
$\Delta\rho_{max}$ = 0.09 e Å^{-3}
$\Delta\rho_{min}$ = −0.15 e Å^{-3}

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

D—H···A	D—H	H···A	D···A	D—H···A
N1—H1···O1	0.86	1.95	2.617 (2)	134
O3—H3···O1^{i}	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*.

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

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supplementary materials

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

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Comment

The title compound, (I), derived from 3-methoxy-2-hydroxybenzaldehyde and 2-aminoethanol, is a potential NO_3 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^+ and the phenolate O^-, 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^{i} (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.

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 temprature and filtered. After keeping the filtrate in air for 3 d, yellow blocks of (I) (yield 66%; mp 338–339 K) were obtained.

Refinement

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

Figures

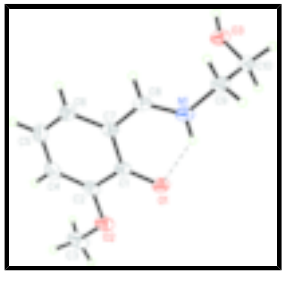

Fig. 1. The structure of (I) with displacement ellipsoids drawn at the 50% probability level. The N—H···O hydrogen bond is shown as a dashed line.

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

Crystal data

$C_{10}H_{13}NO_3$	F_{000} = 416
M_r = 195.21	D_x = 1.293 Mg m^{-3}
Orthorhombic, *Pca*2$_1$	Mo *K*α radiation λ = 0.71073 Å
Hall symbol: P 2c -2ac	Cell parameters from 1828 reflections
a = 14.148 (6) Å	θ = 2.9–20.4º
b = 6.587 (3) Å	μ = 0.10 mm^{-1}
c = 10.760 (4) Å	*T* = 295 K
V = 1002.8 (7) Å^3	Block, yellow
Z = 4	0.30 × 0.30 × 0.12 mm

Data collection

Bruker APEX CCD diffractometer	1041 independent reflections
Radiation source: fine-focus sealed tube	923 reflections with $I > 2\sigma(I)$
Monochromator: graphite	R_{int} = 0.037
T = 295 K	θ_{max} = 26.0º
φ and ω scans	θ_{min} = 2.9º
Absorption correction: multi-scan (SADABS; Bruker, 2002)	*h* = −17→17
T_{min} = 0.974, T_{max} = 0.991	*k* = −8→8
7345 measured reflections	*l* = −12→13

Refinement

Refinement on F^2	Secondary atom site location: difference Fourier map
Least-squares matrix: full	Hydrogen site location: inferred from neighbouring sites
$R[F^2 > 2\sigma(F^2)]$ = 0.034	H-atom parameters constrained
$wR(F^2)$ = 0.084	$w = 1/[\sigma^2(F_o^2) + (0.0473P)^2 + 0.0337P]$ where $P = (F_o^2 + 2F_c^2)/3$
S = 1.07	$(\Delta/\sigma)_{max} < 0.001$
1041 reflections	$\Delta\rho_{max}$ = 0.09 e Å^{-3}
127 parameters	$\Delta\rho_{min}$ = −0.15 e Å^{-3}
1 restraint	Extinction correction: none
Primary atom site location: structure-invariant direct methods	

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 (Å^2)

	x	*y*	*z*	U_{iso}*/U_{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^2)

	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, °)

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
N1—H1···O1	0.86	1.95	2.617 (2)	134
O3—H3···O1^{i}	0.82	1.95	2.741 (3)	161

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

Fig. 1