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3-[(2-Hydroxybenzyl)azaniumyl]propanoate monohydrate

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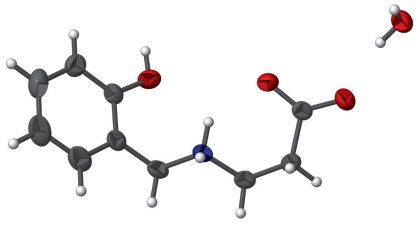
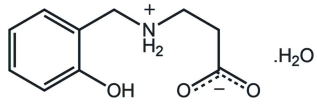
Structural data: full structural data are available from iucrdata.iucr.org

The title compound, $C_{10}H_{13}NO_3\cdot H_2O$, is a zwitterion hydrate with the zwitterion comprising a central ammonium group and a carboxylate residue. In the zwitterion, the hydroxybenzene and carboxylate groups are directed to the same side of the molecule and each orientated to place an O atom in a position to form an intramolecular ammonium-N—H···O hydrogen bond, each closing an *S*(6) loop. The three-dimensional architecture is stabilized by hydroxy-O—H···O(carboxylate), water-O—H···O(carboxylate) and ammonium-N—H···O(water) hydrogen bonds.

3D view



Chemical scheme



Structure description

Reduced Schiff bases such as the title compound were prepared during an on-going study of the coordination chemistry of organotin carboxylates of Schiff bases derived from amino acids (Basu Baul *et al.*, 2013).

The title compound, Fig. 1, features a 2-$(OH)C_6H_4CH_2NH_2^+CH_2CH_2CO_2^-$ zwitterion and a water molecule of crystallization. The assignment is confirmed by the equivalence of the C···O bond lengths, *i.e.* C10—O2, O3 are 1.2527 (16) and 1.2496 (16) Å, respectively, and the pattern of hydrogen bonding involving the ammonium cation, as discussed below. The C2—C7—N1—C8—C9 backbone of the zwitterion is planar with a r.m.s. deviation = 0.0121 Å. The hydroxybenzene ring is twisted out of this plane [dihedral angle = 70.07 (8)°] as is the carboxylate group [dihedral angle = 48.26 (12)°]. The terminal residues lie approximately to the same side of the molecule and form a dihedral angle of 34.16 (15)°. The somewhat flattened U-shaped conformation places both the hydroxy-O atom and one carboxylate-O atom in proximity to one of the ammonium-N—H atoms

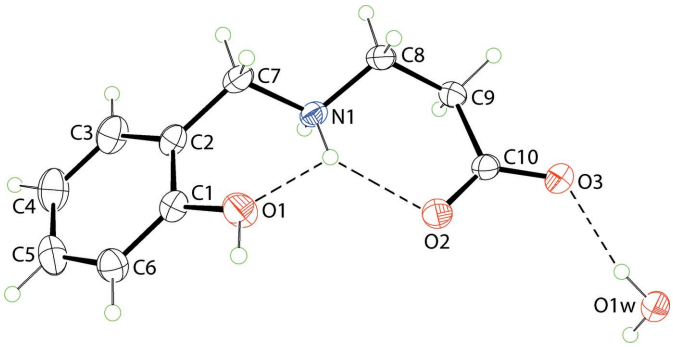


Figure 1
The molecular structure of the title compound showing the atom-labelling scheme and displacement ellipsoids at the 35% probability level. Hydrogen bonds are shown as dashed lines.

leading to the formation of intramolecular N—H···O hydrogen bonds and a pair of *S*(6) loops, Table 1.

In the crystal, O—H···O and N—H···O hydrogen bonds, Table 1, assemble the components into a three-dimensional architecture. The water molecule participates in two donor hydrogen bonds, bridging symmetry-related carboxylate-O3 atoms along the *c* axis, and an acceptor, *i.e.* ammonium-N—H···O(water), hydrogen bond along the *b* axis. Finally, hydroxy-O—H···O2(carboxylate) hydrogen bonds provide links along the *a* axis, Fig. 2.

The most closely related structure available in the literature is that of the 5-bromo zwitterion derivative (Yin *et al.*, 2006). A different conformation is found so that while both terminal residues remain directed to one side of the planar backbone,

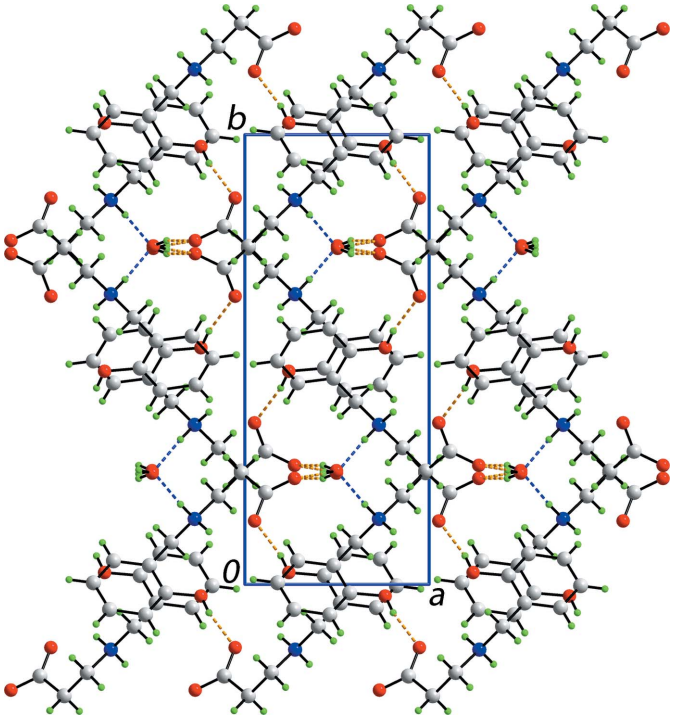


Figure 2
A view of the unit-cell contents of the title compound shown in projection down the *c* axis. The O—H···O and N—H···O hydrogen bonds are shown as orange and blue dashed lines, respectively.

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

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
N1—H2*N*···O1	0.91 (1)	2.45 (1)	2.9862 (16)	118 (1)
N1—H2*N*···O2	0.91 (1)	2.02 (1)	2.7174 (15)	133 (1)
O1—H1*O*···O2^{i}	0.85 (2)	1.83 (2)	2.6607 (16)	167 (2)
N1—H1*N*···O1*W*^{ii}	0.92 (1)	1.83 (1)	2.7460 (15)	171 (1)
O1*W*—H1*W*···O3	0.85 (1)	1.89 (1)	2.7274 (15)	166 (2)
O1*W*—H2*W*···O3^{iii}	0.85 (2)	1.92 (2)	2.7710 (15)	176 (1)

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

the hydroxy group is orientated away from the central ammonium group precluding the formation of an intramolecular hydrogen bond. Finally, the title zwitterion has been reported to complex copper(I) *via* a carboxylate-O atom in the [Cu($^{-}$O$_2$CCH$_2$CH$_2$NH$_2^{+}$CH$_2$C$_6$H$_4$OH-2)(1,10-phenanthroline)]ClO$_4$ salt (Yang *et al.*, 2001).

Synthesis and crystallization

Salicylaldehyde (1.37 g, 11.22 mmol) in ethanol (2 ml) was added dropwise to a previously ice-cooled solution of *β*-alanine (1 g, 11.22 mmol) in water (6 ml) containing KOH (0.62 g, 11.22 mmol). The resulting yellow reaction mixture was allowed to stir for 2 h at room temperature and then the reaction mixture was again placed in an ice-bath. An aqueous solution of sodium borohydride (0.59 g, 15.59 mmol) in water

Table 2
Experimental details.

Crystal data	
Chemical formula	C$_{10}$H$_{13}$NO$_3$·H$_2$O
M_r	213.23
Crystal system, space group	Monoclinic, $P2_1/c$
Temperature (K)	293
a, *b*, *c* (Å)	7.3280 (4), 17.0138 (7), 8.9223 (4)
β (°)	107.818 (5)
V (Å^3)	1059.05 (9)
Z	4
Radiation type	Mo *Kα*
μ (mm^{-1})	0.10
Crystal size (mm)	0.35 × 0.25 × 0.15
Data collection	
Diffractometer	Agilent Xcalibur Eos Gemini diffractometer
Absorption correction	Multi-scan (*CrysAlis PRO*; Agilent, 2013)
T_{min}, T_{max}	0.971, 1.000
No. of measured, independent and observed [$I > 2\sigma(I)$] reflections	5004, 2344, 1962
R_{int}	0.015
(sin *θ*/*λ*)$_{max}$ (Å^{-1})	0.650
Refinement	
$R[F^2 > 2\sigma(F^2)]$, $wR(F^2)$, *S*	0.041, 0.109, 1.05
No. of reflections	2344
No. of parameters	151
No. of restraints	6
$\Delta\rho_{max}$, $\Delta\rho_{min}$ (e Å^{-3})	0.16, −0.20

Computer programs: *CrysAlis PRO* (Agilent, 2013), *SHELXS97* (Sheldrick, 2008), *SHELXL2014* (Sheldrick, 2015), *ORTEP-3 for Windows* (Farrugia, 2012), *DIAMOND* (Brandenburg, 2006), *publCIF* (Westrip, 2010).

(2 ml) was added dropwise with stirring to the cooled solution. The yellow colour slowly discharged and stirring continued for another 3 h. The reaction mixture was then acidified with dilute acetic acid to maintain a pH of 5. The resulting colourless solid was filtered off, washed successively with water, ethanol and diethyl ether, and dried *in vacuo*. The dried product was recrystallized from a water/ethanol (1:1) mixture. Yield: 70%. M.p. 128–130°C. Crystals were grown from an aqueous solution of the compound by slow evaporation at room temperature.

Refinement

Crystal data, data collection and structure refinement details are summarized in Table 2.

Acknowledgements

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full crystallographic data

IUCrData (2016). **1**, x152453 [doi:10.1107/S2414314615024530]

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3-[(2-Hydroxybenzyl)azaniumyl]propanoate monohydrate

Crystal data

$C_{10}H_{13}NO_3·H_2O$
M_r = 213.23
Monoclinic, $P2_1/c$
a = 7.3280 (4) Å
b = 17.0138 (7) Å
c = 8.9223 (4) Å
β = 107.818 (5)°
V = 1059.05 (9) Å^3
Z = 4
$F(000)$ = 456
D_x = 1.337 Mg m^{-3}
Mo $K\alpha$ radiation, λ = 0.71073 Å
Cell parameters from 1703 reflections
θ = 3.8–28.6°
μ = 0.10 mm^{-1}
T = 293 K
Prism, colourless
0.35 × 0.25 × 0.15 mm

Data collection

Agilent Xcalibur Eos Gemini diffractometer
Radiation source: Enhance (Mo) X-ray Source
Detector resolution: 16.1279 pixels mm^{-1}
ω scans
Absorption correction: multi-scan (*CrysAlis PRO*; Agilent, 2013)
T_{min} = 0.971, T_{max} = 1.000
5004 measured reflections
2344 independent reflections
1962 reflections with $I > 2\sigma(I)$
R_{int} = 0.015
θ_{max} = 27.5°, θ_{min} = 3.4°
h = −9→4
k = −22→20
l = −9→11

Refinement

Refinement on F^2
Least-squares matrix: full
$R[F^2 > 2\sigma(F^2)]$ = 0.041
$wR(F^2)$ = 0.109
S = 1.05
2344 reflections
151 parameters
6 restraints
Hydrogen site location: mixed
$w = 1/[\sigma^2(F_o^2) + (0.052P)^2 + 0.2051P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} < 0.001$
$\Delta\rho_{max}$ = 0.16 e Å^{-3}
$\Delta\rho_{min}$ = −0.20 e Å^{-3}

Special details

Geometry. All esds (except the esd in the dihedral angle between two l.s. planes) are estimated using the full covariance matrix. The cell esds are taken into account individually in the estimation of esds in distances, angles and torsion angles; correlations between esds in cell parameters are only used when they are defined by crystal symmetry. An approximate (isotropic) treatment of cell esds is used for estimating esds involving l.s. planes.

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

	x	y	z	U_{iso}*/U_{eq}
O1	0.76143 (15)	0.52616 (7)	0.81882 (14)	0.0502 (3)

H1O	0.805 (3)	0.5632 (9)	0.884 (2)	0.075*
O2	1.05270 (14)	0.35918 (6)	0.99304 (12)	0.0456 (3)
O3	1.26960 (15)	0.26454 (7)	1.02418 (12)	0.0486 (3)
N1	0.72736 (16)	0.35468 (6)	0.74403 (12)	0.0314 (3)
H1N	0.6506 (18)	0.3222 (8)	0.7816 (17)	0.038*
H2N	0.8050 (19)	0.3831 (8)	0.8240 (14)	0.038*
C1	0.56867 (19)	0.51930 (8)	0.78695 (16)	0.0354 (3)
C2	0.48179 (19)	0.45826 (7)	0.68570 (15)	0.0348 (3)
C3	0.2865 (2)	0.44795 (9)	0.6492 (2)	0.0487 (4)
H3	0.2273	0.4075	0.5817	0.058*
C4	0.1777 (2)	0.49673 (10)	0.7112 (2)	0.0564 (4)
H4	0.0458	0.4896	0.6848	0.068*
C5	0.2656 (2)	0.55610 (9)	0.8125 (2)	0.0499 (4)
H5	0.1926	0.5886	0.8555	0.060*
C6	0.4608 (2)	0.56783 (8)	0.85086 (18)	0.0428 (3)
H6	0.5193	0.6081	0.9192	0.051*
C7	0.6016 (2)	0.40636 (8)	0.61911 (15)	0.0376 (3)
H7A	0.6803	0.4384	0.5734	0.045*
H7B	0.5190	0.3741	0.5362	0.045*
C8	0.85599 (19)	0.30361 (9)	0.68568 (15)	0.0371 (3)
H8A	0.9369	0.3363	0.6434	0.045*
H8B	0.7790	0.2708	0.6010	0.045*
C9	0.9807 (2)	0.25196 (8)	0.81421 (16)	0.0360 (3)
H9A	1.0585	0.2190	0.7692	0.043*
H9B	0.8985	0.2176	0.8519	0.043*
C10	1.11237 (19)	0.29581 (8)	0.95388 (14)	0.0338 (3)
O1W	1.49712 (14)	0.25271 (6)	1.32892 (12)	0.0436 (3)
H2W	1.424 (2)	0.2457 (11)	1.3856 (19)	0.065*
H1W	1.424 (2)	0.2644 (11)	1.2376 (13)	0.065*

Atomic displacement parameters (Å²)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
O1	0.0322 (6)	0.0544 (6)	0.0607 (7)	−0.0073 (5)	0.0095 (5)	−0.0260 (5)
O2	0.0373 (5)	0.0473 (6)	0.0457 (6)	0.0019 (5)	0.0029 (4)	−0.0185 (5)
O3	0.0423 (6)	0.0615 (7)	0.0355 (5)	0.0138 (5)	0.0021 (4)	−0.0033 (5)
N1	0.0301 (6)	0.0357 (6)	0.0259 (5)	−0.0023 (5)	0.0048 (4)	−0.0023 (4)
C1	0.0318 (7)	0.0359 (7)	0.0357 (7)	−0.0005 (6)	0.0060 (5)	0.0029 (5)
C2	0.0335 (7)	0.0323 (6)	0.0345 (6)	−0.0002 (5)	0.0044 (5)	0.0056 (5)
C3	0.0375 (8)	0.0439 (8)	0.0574 (9)	−0.0081 (7)	0.0039 (7)	0.0035 (7)
C4	0.0318 (8)	0.0585 (10)	0.0771 (12)	−0.0009 (7)	0.0140 (8)	0.0110 (9)
C5	0.0454 (9)	0.0471 (8)	0.0621 (10)	0.0136 (7)	0.0238 (8)	0.0138 (7)
C6	0.0447 (8)	0.0371 (7)	0.0465 (8)	0.0030 (6)	0.0139 (7)	0.0006 (6)
C7	0.0417 (8)	0.0372 (7)	0.0275 (6)	−0.0015 (6)	0.0013 (5)	0.0000 (5)
C8	0.0357 (7)	0.0475 (8)	0.0279 (6)	−0.0004 (6)	0.0094 (5)	−0.0070 (5)
C9	0.0354 (7)	0.0377 (7)	0.0348 (7)	0.0020 (6)	0.0105 (6)	−0.0081 (5)
C10	0.0319 (7)	0.0426 (7)	0.0275 (6)	−0.0005 (6)	0.0096 (5)	−0.0017 (5)
O1W	0.0345 (5)	0.0573 (6)	0.0369 (5)	0.0042 (5)	0.0079 (4)	−0.0007 (5)

Geometric parameters (Å, º)

O1—C1	1.3579 (17)	C4—H4	0.9300
O1—H1O	0.849 (9)	C5—C6	1.380 (2)
O2—C10	1.2527 (16)	C5—H5	0.9300
O3—C10	1.2496 (16)	C6—H6	0.9300
N1—C8	1.4882 (17)	C7—H7A	0.9700
N1—C7	1.4955 (16)	C7—H7B	0.9700
N1—H1N	0.922 (9)	C8—C9	1.5104 (19)
N1—H2N	0.904 (9)	C8—H8A	0.9700
C1—C6	1.381 (2)	C8—H8B	0.9700
C1—C2	1.3956 (19)	C9—C10	1.5180 (18)
C2—C3	1.378 (2)	C9—H9A	0.9700
C2—C7	1.491 (2)	C9—H9B	0.9700
C3—C4	1.379 (2)	O1W—H2W	0.850 (9)
C3—H3	0.9300	O1W—H1W	0.851 (9)
C4—C5	1.377 (3)		
C1—O1—H1O	110.9 (14)	C1—C6—H6	120.3
C8—N1—C7	113.24 (10)	C2—C7—N1	110.80 (10)
C8—N1—H1N	107.4 (9)	C2—C7—H7A	109.5
C7—N1—H1N	108.5 (9)	N1—C7—H7A	109.5
C8—N1—H2N	106.0 (10)	C2—C7—H7B	109.5
C7—N1—H2N	111.7 (9)	N1—C7—H7B	109.5
H1N—N1—H2N	109.9 (13)	H7A—C7—H7B	108.1
O1—C1—C6	123.51 (13)	N1—C8—C9	112.02 (10)
O1—C1—C2	116.03 (12)	N1—C8—H8A	109.2
C6—C1—C2	120.45 (13)	C9—C8—H8A	109.2
C3—C2—C1	118.87 (14)	N1—C8—H8B	109.2
C3—C2—C7	121.71 (13)	C9—C8—H8B	109.2
C1—C2—C7	119.42 (12)	H8A—C8—H8B	107.9
C2—C3—C4	120.98 (15)	C8—C9—C10	114.98 (11)
C2—C3—H3	119.5	C8—C9—H9A	108.5
C4—C3—H3	119.5	C10—C9—H9A	108.5
C5—C4—C3	119.54 (15)	C8—C9—H9B	108.5
C5—C4—H4	120.2	C10—C9—H9B	108.5
C3—C4—H4	120.2	H9A—C9—H9B	107.5
C4—C5—C6	120.70 (15)	O3—C10—O2	124.98 (12)
C4—C5—H5	119.7	O3—C10—C9	117.36 (12)
C6—C5—H5	119.7	O2—C10—C9	117.62 (11)
C5—C6—C1	119.46 (14)	H2W—O1W—H1W	106.0 (15)
C5—C6—H6	120.3		
O1—C1—C2—C3	179.74 (13)	O1—C1—C6—C5	−179.56 (14)
C6—C1—C2—C3	0.8 (2)	C2—C1—C6—C5	−0.7 (2)
O1—C1—C2—C7	−0.67 (18)	C3—C2—C7—N1	−110.11 (14)
C6—C1—C2—C7	−179.59 (12)	C1—C2—C7—N1	70.31 (16)
C1—C2—C3—C4	−0.1 (2)	C8—N1—C7—C2	−177.77 (11)

C7—C2—C3—C4	−179.67 (14)	C7—N1—C8—C9	−179.82 (11)
C2—C3—C4—C5	−0.7 (3)	N1—C8—C9—C10	−59.93 (15)
C3—C4—C5—C6	0.8 (2)	C8—C9—C10—O3	−148.37 (13)
C4—C5—C6—C1	−0.1 (2)	C8—C9—C10—O2	33.91 (17)

Hydrogen-bond geometry (Å, º)

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
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N1—H2*N*···O2	0.91 (1)	2.02 (1)	2.7174 (15)	133 (1)
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N1—H1*N*···O1*W*^{ii}	0.92 (1)	1.83 (1)	2.7460 (15)	171 (1)
O1*W*—H1*W*···O3	0.85 (1)	1.89 (1)	2.7274 (15)	166 (2)
O1*W*—H2*W*···O3^{iii}	0.85 (2)	1.92 (2)	2.7710 (15)	176 (1)

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