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3-Hydroxy-2-[(4-hydroxybenzyl)azaniumyl]-propanoate monohydrate

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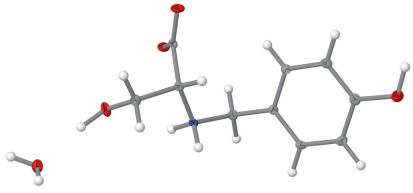
Keywords: crystal structure; hydrate; 3-hydroxy-2-[(4-hydroxybenzyl)azaniumyl]propanoate; C—H···O hydrogen bonding; O—H···O hydrogen bonding; N—H···O hydrogen bonding; synchrotron.

CCDC reference: 1533317

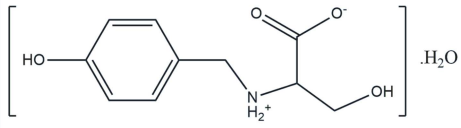
Structural data: full structural data are available from iucrdata.iucr.org

In the title hydrated zwitterion, $C_{10}H_{13}NO_4\cdot H_2O$, the N—C—C—OH side chain shows a *gauche* conformation [torsion angle = −59.05 (18)°]. In the crystal, the components are linked by O—H···O, N—H···O and C—H···O hydrogen bonds to generate a three-dimensional supramolecular network.

3D view



Chemical scheme



Structure description

Recently, a copper(II) complex with a reduced amino acid Schiff base was shown to exhibit inhibiting activity with respect to protein tyrosine phosphatase (Li *et al.*, 2016). In our previous reports, we have shown that VO(II) complexes have very strong inhibitory activity towards protein tyrosine phosphatase (Lu & Zhu, 2014; Han *et al.*, 2012; Li *et al.* 2016). Thus, 3-hydroxy-2-[(4-hydroxybenzyl)amino]propanoic acid was reacted with $VOSO_4$ in alkaline solution to prepare its VO complex in order to test its biological activity. However, no complex of VO(II) was formed; instead crystals of the title compound were obtained.

The molecular structure of the title compound is illustrated in Fig. 1. The asymmetric unit consists of a 3-hydroxy-2-[(4-hydroxybenzyl)azaniumyl]propanoate zwitterion (nominal proton transfer from the carboxylic acid group to the secondary amine group) and a water molecule. The zwitterionic form found agrees with that of many amino acids. The C5—C4—N1—C2 and N1—C1—C2—O3 links both show a *gauche* conformation [torsion angles = 65.0 (2) and −59.05 (18)°, respectively].

In the crystal, molecules are connected through a network of O—H···O, N—H···O and C—H···O hydrogen bonds (Table 1), leading to the formation of a three-dimensional supramolecular network (Fig. 2).

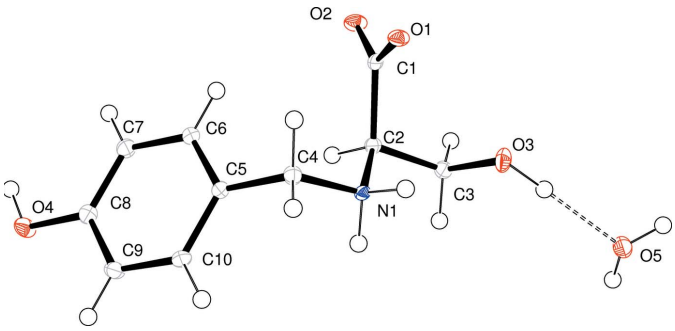


Figure 1
The molecular structure of the title compound, with displacement ellipsoids for non-H atoms drawn at the 50% probability level.

Synthesis and crystallization

A mixture containing $VOSO_4{\cdot}xH_2O$ (0.1 mmol), 3-hydroxy-2-[(4-hydroxybenzyl)amino]propanoic acid (0.1 mmol), KOH (0.2 mmol) and H_2O (6.0 ml) was stirred for 30 min at room temperature. The reaction mixture was filtered and the liquor was kept at room temperature. Light-yellow block-shaped crystals of the title compound appeared after 3 d in 40% yield.

Refinement

Crystal data, data collection and structure refinement details are summarized in Table 2. The DISP instruction was used in the *SHELXL2016* (Sheldrick, 2015) refinements in order to correct anomalous scattering values (f' and f'') of elements for the synchrotron wavelength used.

Funding information

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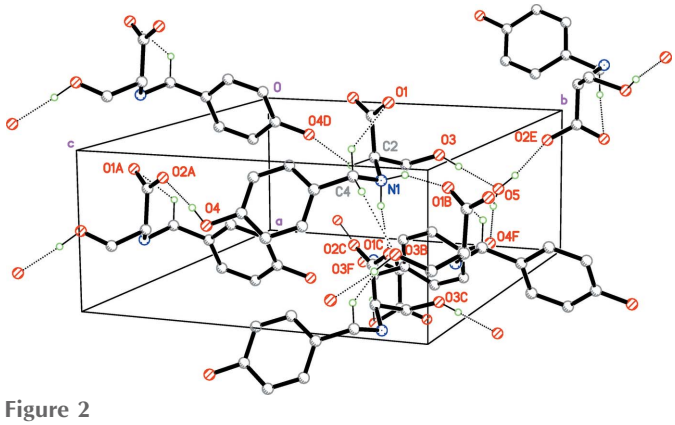

Figure 2
The C—H···O, O—H···O and N—H···O hydrogen-bonded (dotted lines) network. [Symmetry codes: (A) $x+\frac{1}{2}, -y+\frac{1}{2}, 1-z$; (B) $x+\frac{1}{2}, -y+\frac{3}{2}, 1-z$; (C) $x+1, y, z$; (D) $x-\frac{1}{2}, -y+\frac{1}{2}, 1-z$; (E) $-x, y+\frac{1}{2}, -z+\frac{1}{2}$; (F) $-x+\frac{3}{2}, 1-y, z-\frac{1}{2}$.]

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

D—H···A	D—H	H···A	D···A	D—H···A
O3—H3···O5	0.98 (3)	1.79 (3)	2.759 (2)	166 (2)
O4—H4···O2^{i}	0.90 (3)	1.72 (3)	2.624 (2)	174 (3)
N1—H1A···O1^{ii}	0.94 (2)	1.98 (2)	2.8390 (19)	150 (2)
N1—H1B···O1^{iii}	0.96 (3)	1.84 (3)	2.782 (2)	170 (2)
C2—H2···O4^{iv}	1.00	2.54	3.300 (2)	132
C4—H4A···O3^{ii}	0.99	2.55	3.392 (2)	143
O5—H5A···O2^{v}	0.85 (3)	1.99 (3)	2.8380 (19)	172 (3)
O5—H5B···O4^{vi}	0.87 (3)	1.92 (3)	2.785 (2)	171 (3)

Symmetry codes: (i) $x+\frac{1}{2}, -y+\frac{1}{2}, -z+1$; (ii) $x+\frac{1}{2}, -y+\frac{3}{2}, -z+1$; (iii) $x+1, y, z$; (iv) $x-\frac{1}{2}, -y+\frac{1}{2}, -z+1$; (v) $-x, y+\frac{1}{2}, -z+\frac{1}{2}$; (vi) $-x+\frac{3}{2}, -y+1, z-\frac{1}{2}$.

Table 2
Experimental details.

Crystal data	
Chemical formula	$C_{10}H_{13}NO_4{\cdot}H_2O$
M_r	229.23
Crystal system, space group	Orthorhombic, $P2_12_12_1$
Temperature (K)	100
a, b, c (Å)	5.5140 (11), 12.418 (3), 15.372 (3)
V (Å^3)	1052.6 (4)
Z	4
Radiation type	Synchrotron, λ = 0.7200 Å
μ (mm^{-1})	0.12
Crystal size (mm)	0.30 × 0.20 × 0.20
Data collection	
Diffractometer	Mar165
Absorption correction	Multi-scan (*SCALEPACK*; Otwinowski & Minor, 1997)
T_{min}, T_{max}	0.965, 0.977
No. of measured, independent and observed [$I > 2\sigma(I)$] reflections	5039, 2721, 2624
R_{int}	0.025
(sin $\theta/\lambda)_{max}$ (Å^{-1})	0.681
Refinement	
$R[F^2 > 2\sigma(F^2)]$, $wR(F^2)$, S	0.036, 0.097, 1.11
No. of reflections	2721
No. of parameters	164
H-atom treatment	H atoms treated by a mixture of independent and constrained refinement
$\Delta\rho_{max}$, $\Delta\rho_{min}$ (e Å^{-3})	0.19, −0.22
Absolute structure	Refined as an inversion twin
Absolute structure parameter	0.2 (12)

Computer programs: *SCALEPACK* (Otwinowski & Minor, 1997), *HKL-2000* (Otwinowski & Minor, 1997), *SHELXS97* and *SHELXTL* (Sheldrick, 2008), *SHELXL2016* (Sheldrick, 2015) and *publCIF* (Westrip, 2010).

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full crystallographic data

IUCrData (2017). **2**, x170271 [https://doi.org/10.1107/S2414314617002711]

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Crystal data

$C_{10}H_{13}NO_4·H_2O$
M_r = 229.23
Orthorhombic, $P2_12_12_1$
a = 5.5140 (11) Å
b = 12.418 (3) Å
c = 15.372 (3) Å
V = 1052.6 (4) Å^3
Z = 4
F(000) = 488
D_x = 1.447 Mg m^{-3}
Synchrotron radiation, λ = 0.7200 Å
Cell parameters from 5057 reflections
θ = 2.7–29.6°
μ = 0.12 mm^{-1}
T = 100 K
Block, light yelloe
0.30 × 0.20 × 0.20 mm

Data collection

Mar165 diffractometer
Radiation source: synchrotron, 3W1A at BSRF
oscillation mode scans
Absorption correction: multi-scan (SCALEPACK; Otwinowski & Minor, 1997)
T_{min} = 0.965, T_{max} = 0.977
5039 measured reflections
2721 independent reflections
2624 reflections with $I > 2\sigma(I)$
R_{int} = 0.025
θ_{max} = 29.3°, θ_{min} = 2.7°
h = 0→7
k = −16→16
l = −20→20

Refinement

Refinement on F^2
Least-squares matrix: full
$R[F^2 > 2\sigma(F^2)]$ = 0.036
$wR(F^2)$ = 0.097
S = 1.11
2721 reflections
164 parameters
0 restraints
Primary atom site location: structure-invariant direct methods
Hydrogen site location: mixed
H atoms treated by a mixture of independent and constrained refinement
$w = 1/[\sigma^2(F_o^2) + (0.0569P)^2 + 0.2819P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} < 0.001$
$\Delta\rho_{max}$ = 0.19 e Å^{-3}
$\Delta\rho_{min}$ = −0.22 e Å^{-3}
Absolute structure: Refined as an inversion twin
Absolute structure parameter: 0.2 (12)

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.

Refinement. Refined as a 2-component inversion twin.

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

	x	*y*	*z*	U_{iso}*/U_{eq}
O1	−0.1740 (2)	0.64457 (10)	0.46536 (8)	0.0124 (3)
O2	−0.1669 (3)	0.49448 (10)	0.38500 (9)	0.0145 (3)
O3	0.2072 (3)	0.74505 (10)	0.34128 (9)	0.0153 (3)
H3	0.297 (6)	0.788 (2)	0.2981 (18)	0.023*
O4	0.6113 (3)	0.16978 (11)	0.65474 (10)	0.0157 (3)
H4	0.507 (5)	0.116 (2)	0.6406 (18)	0.024*
N1	0.3273 (3)	0.63321 (11)	0.49124 (9)	0.0087 (3)
H1A	0.282 (5)	0.7061 (19)	0.4913 (15)	0.010*
H1B	0.499 (5)	0.6282 (19)	0.4830 (16)	0.010*
C1	−0.0667 (3)	0.57215 (13)	0.42362 (11)	0.0094 (3)
C2	0.2125 (3)	0.57711 (13)	0.41594 (11)	0.0097 (3)
H2	0.276793	0.501896	0.413099	0.012*
C3	0.2880 (4)	0.63678 (14)	0.33320 (11)	0.0118 (3)
H3A	0.466456	0.634670	0.326401	0.014*
H3B	0.213318	0.602571	0.281565	0.014*
C4	0.2672 (3)	0.59029 (14)	0.58072 (11)	0.0113 (3)
H4A	0.338442	0.638592	0.625116	0.014*
H4B	0.088978	0.590776	0.588583	0.014*
C5	0.3605 (3)	0.47754 (14)	0.59503 (11)	0.0104 (3)
C6	0.2266 (4)	0.38722 (13)	0.56981 (11)	0.0120 (3)
H6	0.077310	0.396915	0.539990	0.014*
C7	0.3078 (4)	0.28317 (14)	0.58753 (11)	0.0132 (3)
H7	0.215956	0.222503	0.569270	0.016*
C8	0.5258 (3)	0.26924 (14)	0.63247 (11)	0.0122 (3)
C9	0.6632 (3)	0.35835 (15)	0.65700 (12)	0.0132 (3)
H9	0.812625	0.348687	0.686782	0.016*
C10	0.5809 (4)	0.46163 (13)	0.63769 (12)	0.0121 (3)
H10	0.676154	0.522158	0.653820	0.014*
O5	0.4627 (3)	0.88897 (11)	0.23976 (9)	0.0154 (3)
H5A	0.381 (5)	0.927 (2)	0.2039 (19)	0.023*
H5B	0.595 (6)	0.864 (2)	0.2161 (19)	0.023*

Atomic displacement parameters (Å²)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
O1	0.0082 (6)	0.0102 (5)	0.0188 (6)	0.0008 (5)	0.0009 (5)	−0.0022 (5)
O2	0.0108 (7)	0.0111 (6)	0.0216 (6)	−0.0021 (5)	−0.0026 (5)	−0.0048 (5)
O3	0.0190 (7)	0.0096 (5)	0.0173 (6)	0.0027 (5)	0.0047 (5)	0.0035 (5)
O4	0.0141 (7)	0.0085 (6)	0.0246 (7)	0.0025 (5)	−0.0049 (5)	−0.0007 (5)
N1	0.0074 (7)	0.0066 (6)	0.0122 (6)	−0.0005 (5)	0.0002 (5)	0.0003 (5)
C1	0.0101 (8)	0.0066 (6)	0.0117 (7)	−0.0001 (6)	0.0001 (6)	0.0003 (5)
C2	0.0092 (8)	0.0083 (6)	0.0117 (7)	0.0002 (6)	0.0001 (6)	−0.0008 (5)
C3	0.0105 (8)	0.0111 (7)	0.0137 (7)	0.0016 (6)	0.0014 (6)	0.0007 (6)
C4	0.0112 (9)	0.0114 (7)	0.0113 (7)	0.0008 (6)	0.0007 (6)	0.0003 (6)
C5	0.0098 (8)	0.0110 (7)	0.0106 (7)	−0.0003 (6)	0.0007 (6)	0.0011 (5)

C6	0.0108 (9)	0.0119 (7)	0.0134 (7)	−0.0016 (6)	−0.0012 (6)	0.0019 (6)
C7	0.0142 (9)	0.0106 (7)	0.0149 (7)	−0.0024 (6)	−0.0020 (7)	−0.0002 (6)
C8	0.0126 (9)	0.0112 (7)	0.0127 (7)	0.0009 (6)	0.0015 (6)	−0.0004 (6)
C9	0.0101 (8)	0.0139 (7)	0.0155 (7)	0.0001 (6)	−0.0022 (6)	−0.0001 (6)
C10	0.0106 (8)	0.0115 (7)	0.0141 (7)	−0.0031 (6)	−0.0014 (6)	−0.0006 (6)
O5	0.0146 (8)	0.0158 (6)	0.0157 (6)	0.0026 (5)	0.0016 (5)	0.0035 (5)

Geometric parameters (Å, º)

O1—C1	1.253 (2)	C4—C5	1.508 (2)
O2—C1	1.260 (2)	C4—H4A	0.9900
O3—C3	1.422 (2)	C4—H4B	0.9900
O3—H3	0.98 (3)	C5—C10	1.395 (3)
O4—C8	1.366 (2)	C5—C6	1.398 (2)
O4—H4	0.90 (3)	C6—C7	1.394 (2)
N1—C2	1.492 (2)	C6—H6	0.9500
N1—C4	1.512 (2)	C7—C8	1.397 (3)
N1—H1A	0.94 (2)	C7—H7	0.9500
N1—H1B	0.96 (3)	C8—C9	1.393 (2)
C1—C2	1.545 (3)	C9—C10	1.393 (2)
C2—C3	1.530 (2)	C9—H9	0.9500
C2—H2	1.0000	C10—H10	0.9500
C3—H3A	0.9900	O5—H5A	0.85 (3)
C3—H3B	0.9900	O5—H5B	0.87 (3)
C3—O3—H3	107.0 (16)	C5—C4—H4A	109.1
C8—O4—H4	112.6 (18)	N1—C4—H4A	109.1
C2—N1—C4	116.62 (14)	C5—C4—H4B	109.1
C2—N1—H1A	109.8 (15)	N1—C4—H4B	109.1
C4—N1—H1A	106.3 (15)	H4A—C4—H4B	107.8
C2—N1—H1B	106.7 (14)	C10—C5—C6	118.48 (16)
C4—N1—H1B	108.3 (15)	C10—C5—C4	119.83 (16)
H1A—N1—H1B	109 (2)	C6—C5—C4	121.64 (17)
O1—C1—O2	125.72 (17)	C7—C6—C5	121.34 (17)
O1—C1—C2	118.72 (15)	C7—C6—H6	119.3
O2—C1—C2	115.54 (15)	C5—C6—H6	119.3
N1—C2—C3	107.65 (14)	C6—C7—C8	119.17 (16)
N1—C2—C1	112.46 (14)	C6—C7—H7	120.4
C3—C2—C1	110.73 (14)	C8—C7—H7	120.4
N1—C2—H2	108.6	O4—C8—C9	117.56 (16)
C3—C2—H2	108.6	O4—C8—C7	122.21 (16)
C1—C2—H2	108.6	C9—C8—C7	120.23 (16)
O3—C3—C2	107.47 (13)	C10—C9—C8	119.76 (17)
O3—C3—H3A	110.2	C10—C9—H9	120.1
C2—C3—H3A	110.2	C8—C9—H9	120.1
O3—C3—H3B	110.2	C9—C10—C5	120.98 (16)
C2—C3—H3B	110.2	C9—C10—H10	119.5
H3A—C3—H3B	108.5	C5—C10—H10	119.5

C5—C4—N1	112.66 (14)	H5A—O5—H5B	112 (3)
C4—N1—C2—C3	177.31 (14)	C10—C5—C6—C7	0.9 (3)
C4—N1—C2—C1	55.06 (18)	C4—C5—C6—C7	−176.43 (16)
O1—C1—C2—N1	28.0 (2)	C5—C6—C7—C8	0.9 (3)
O2—C1—C2—N1	−153.20 (14)	C6—C7—C8—O4	177.55 (17)
O1—C1—C2—C3	−92.54 (18)	C6—C7—C8—C9	−1.8 (3)
O2—C1—C2—C3	86.31 (18)	O4—C8—C9—C10	−178.43 (17)
N1—C2—C3—O3	−59.05 (18)	C7—C8—C9—C10	0.9 (3)
C1—C2—C3—O3	64.27 (18)	C8—C9—C10—C5	0.9 (3)
C2—N1—C4—C5	65.0 (2)	C6—C5—C10—C9	−1.7 (3)
N1—C4—C5—C10	96.82 (19)	C4—C5—C10—C9	175.60 (17)
N1—C4—C5—C6	−85.9 (2)		

Hydrogen-bond geometry (Å, º)

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
O3—H3···O5	0.98 (3)	1.79 (3)	2.759 (2)	166 (2)
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Symmetry codes: (i) *x*+1/2, −*y*+1/2, −*z*+1; (ii) *x*+1/2, −*y*+3/2, −*z*+1; (iii) *x*+1, *y*, *z*; (iv) *x*−1/2, −*y*+1/2, −*z*+1; (v) −*x*, *y*+1/2, −*z*+1/2; (vi) −*x*+3/2, −*y*+1, *z*−1/2.