

Diaquabis(4-oxo-1,4-dihydropyridine-3-sulfonato- κ^2O^3,O^4)zinc(II)

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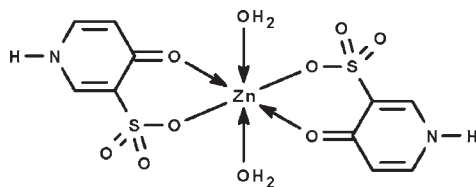
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 Key indicators: single-crystal X-ray study; $T = 293$ K; mean $\sigma(C-C) = 0.003$ Å; R factor = 0.025; wR factor = 0.074; data-to-parameter ratio = 13.7.

In the crystal structure of the title compound, $[Zn(C_5H_4NO_4S)_2(H_2O)_2]$, the 4-oxo-1,4-dihydropyridine-3-sulfonate anion chelates to water-coordinated zinc centres through the carbonyl O atom and through one O atom of the sulfonate group. The Zn^{II} atom lies on a center of inversion, and adjacent molecules are linked by $N-H \cdots O$ and $O-H \cdots O$ hydrogen bonds, forming a three-dimensional network.

Related literature

 For the structure of the 4-oxo-1,4-dihydropyridine-3-sulfonate anion, see: Zhu *et al.* (2009).


Experimental

Crystal data

 $[Zn(C_5H_4NO_4S)_2(H_2O)_2]$
 $M_r = 449.71$
 Monoclinic, $P2_1/c$
 $a = 4.9263$ (1) Å
 $b = 20.9529$ (6) Å

 $c = 7.4437$ (2) Å
 $\beta = 98.9371$ (9)°
 $V = 759.01$ (3) Å³
 $Z = 2$
 Mo $K\alpha$ radiation

 $\mu = 1.95$ mm⁻¹
 $T = 293$ K

 $0.23 \times 0.17 \times 0.14$ mm

Data collection

 Rigaku R-Axis RAPID IP diffractometer
 Absorption correction: multi-scan (ABSCOR; Higashi, 1995)
 $T_{min} = 0.662$, $T_{max} = 0.772$

 7334 measured reflections
 1738 independent reflections
 1629 reflections with $I > 2\sigma(I)$
 $R_{int} = 0.021$

Refinement

 $R[F^2 > 2\sigma(F^2)] = 0.025$
 $wR(F^2) = 0.074$
 $S = 1.10$
 1738 reflections
 127 parameters
 3 restraints

 H atoms treated by a mixture of independent and constrained refinement
 $\Delta\rho_{max} = 0.45$ e Å⁻³
 $\Delta\rho_{min} = -0.46$ e Å⁻³
Table 1

Hydrogen-bond geometry (Å, °).

$D-H \cdots A$	$D-H$	$H \cdots A$	$D \cdots A$	$D-H \cdots A$
$O1w-H1 \cdots O2^i$	0.84 (1)	2.05 (2)	2.797 (2)	147 (2)
$O1w-H2 \cdots O4^{ii}$	0.84 (1)	1.92 (1)	2.744 (2)	171 (3)
$N1-H3 \cdots O3^{iii}$	0.85 (1)	1.91 (1)	2.754 (2)	175 (3)

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

Data collection: *RAPID-AUTO* (Rigaku, 1998); cell refinement: *RAPID-AUTO*; data reduction: *CrystalClear* (Rigaku/MSK, 2002); program(s) used to solve structure: *SHELXS97* (Sheldrick, 2008); program(s) used to refine structure: *SHELXL97* (Sheldrick, 2008); molecular graphics: *X-SEED* (Barbour, 2001); software used to prepare material for publication: *publCIF* (Westrip, 2009).

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Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: BT5120).

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supporting information

Acta Cryst. (2009). E65, m1493 [https://doi.org/10.1107/S1600536809044948]

Diaquabis(4-oxo-1,4-dihydropyridine-3-sulfonato- κ^2O^3,O^4)zinc(II)

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S1. Experimental

Zinc carbonate (0.25 g, 2 mmol) was added to a hot aqueous solution of 4-hydroxypyridine-3-sulfonic acid (0.35 g, 2 mmol); the pH value was adjusted to 6 with 0.1 M sodium hydroxide. The solution was allowed to evaporate slowly at room temperature, and colorless prismatic crystals were isolated after about five days. CH&N elemental analysis. Calc. for $C_{10}H_{12}N_2O_{10}S_2Zn$: C 26.71, H 2.69, N 6.23%; found: C 26.73, H 2.73, N 6.21%.

S2. Refinement

Carbon-bound H-atoms were placed in calculated positions (C—H 0.93 Å) and were included in the refinement in the riding model approximation, with $U(H)$ set to $1.2U(C)$. The amino and water H-atoms were located in a difference Fourier map, and were refined isotropically with a distance restraint of N—H = O—H = 0.85 ± 0.01 Å.

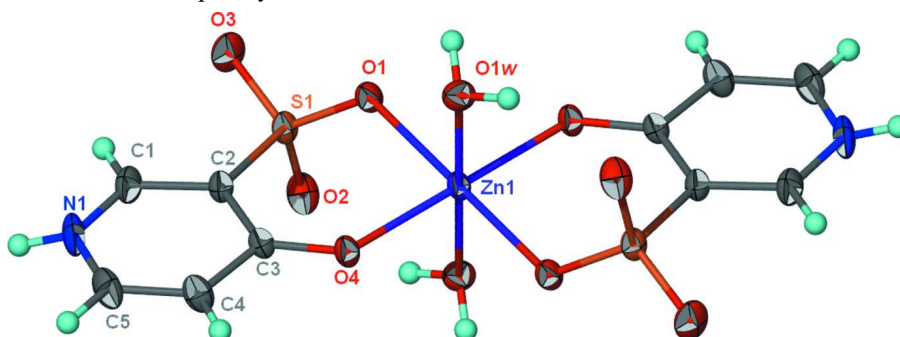


Figure 1

Anisotropic displacement ellipsoid plot (Barbour, 2001) of $Zn(H_2O)_2(C_5H_4NO_4S)_2$ at the 50% probability level; hydrogen atoms are drawn as spheres of arbitrary radius.

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Hall symbol: $-P 2_1/c$

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$b = 20.9529$ (6) Å

$c = 7.4437$ (2) Å

$\beta = 98.9371$ (9)°

$V = 759.01$ (3) Å³

$Z = 2$

$F(000) = 456$

$D_x = 1.968$ Mg m⁻³

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

Cell parameters from 6865 reflections

$\theta = 3.4$ – 27.5 °

$\mu = 1.95$ mm⁻¹

$T = 293$ K

Prism, colorless

$0.23 \times 0.17 \times 0.14$ mm

Data collection

Rigaku R-AXIS RAPID IP
diffractometer
Radiation source: fine-focus sealed tube
Graphite monochromator
 ω scans
Absorption correction: multi-scan
(*ABSCOR*; Higashi, 1995)
 $T_{\min} = 0.662$, $T_{\max} = 0.772$

7334 measured reflections
1738 independent reflections
1629 reflections with $I > 2\sigma(I)$
 $R_{\text{int}} = 0.021$
 $\theta_{\max} = 27.5^\circ$, $\theta_{\min} = 3.4^\circ$
 $h = -6 \rightarrow 5$
 $k = -27 \rightarrow 27$
 $l = -9 \rightarrow 9$

Refinement

Refinement on F^2
Least-squares matrix: full
 $R[F^2 > 2\sigma(F^2)] = 0.025$
 $wR(F^2) = 0.074$
 $S = 1.10$
1738 reflections
127 parameters
3 restraints
Primary atom site location: structure-invariant
direct methods

Secondary atom site location: difference Fourier
map
Hydrogen site location: inferred from
neighbouring sites
H atoms treated by a mixture of independent
and constrained refinement
 $w = 1/[\sigma^2(F_o^2) + (0.0399P)^2 + 0.5207P]$
where $P = (F_o^2 + 2F_c^2)/3$
 $(\Delta/\sigma)_{\max} = 0.001$
 $\Delta\rho_{\max} = 0.45 \text{ e } \text{\AA}^{-3}$
 $\Delta\rho_{\min} = -0.46 \text{ e } \text{\AA}^{-3}$

Fractional atomic coordinates and isotropic or equivalent isotropic displacement parameters (\AA^2)

	<i>x</i>	<i>y</i>	<i>z</i>	$U_{\text{iso}}^*/U_{\text{eq}}$
Zn1	0.5000	0.5000	0.5000	0.01991 (11)
S1	0.54548 (9)	0.35166 (2)	0.43742 (6)	0.02251 (13)
O1	0.6943 (3)	0.41242 (6)	0.46588 (19)	0.0246 (3)
O2	0.4026 (3)	0.33653 (7)	0.5890 (2)	0.0327 (3)
O3	0.7182 (3)	0.30080 (7)	0.3867 (2)	0.0380 (4)
O4	0.1877 (3)	0.47222 (6)	0.29786 (18)	0.0238 (3)
O1W	0.7120 (3)	0.54012 (7)	0.3004 (2)	0.0279 (3)
N1	-0.0061 (4)	0.30854 (10)	0.0136 (2)	0.0361 (5)
C1	0.2078 (4)	0.30795 (10)	0.1490 (3)	0.0303 (4)
H1A	0.3053	0.2703	0.1764	0.036*
C2	0.2849 (4)	0.36189 (8)	0.2474 (3)	0.0221 (4)
C3	0.1359 (4)	0.42039 (8)	0.2098 (2)	0.0209 (4)
C4	-0.0876 (4)	0.41740 (10)	0.0613 (3)	0.0294 (4)
H4	-0.1897	0.4540	0.0274	0.035*
C5	-0.1522 (5)	0.36232 (11)	-0.0300 (3)	0.0353 (5)
H5A	-0.2998	0.3615	-0.1245	0.042*
H1	0.738 (5)	0.5794 (5)	0.318 (4)	0.035 (7)*
H2	0.866 (3)	0.5233 (13)	0.301 (4)	0.040 (7)*
H3	-0.081 (6)	0.2742 (9)	-0.028 (4)	0.051 (8)*

Atomic displacement parameters (\AA^2)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
Zn1	0.01975 (17)	0.01417 (16)	0.02420 (18)	-0.00106 (10)	-0.00165 (12)	-0.00169 (10)
S1	0.0258 (2)	0.0128 (2)	0.0270 (2)	0.00172 (16)	-0.00212 (18)	0.00056 (16)

O1	0.0221 (6)	0.0168 (6)	0.0333 (7)	0.0005 (5)	-0.0006 (5)	-0.0015 (5)
O2	0.0433 (8)	0.0247 (7)	0.0286 (7)	-0.0061 (6)	0.0009 (6)	0.0042 (6)
O3	0.0398 (8)	0.0203 (7)	0.0503 (10)	0.0124 (6)	-0.0037 (7)	-0.0046 (6)
O4	0.0226 (6)	0.0168 (6)	0.0295 (7)	0.0010 (5)	-0.0042 (5)	-0.0031 (5)
O1W	0.0263 (7)	0.0244 (7)	0.0333 (8)	0.0020 (6)	0.0059 (6)	0.0008 (6)
N1	0.0443 (11)	0.0297 (10)	0.0324 (10)	-0.0131 (8)	0.0006 (8)	-0.0141 (7)
C1	0.0366 (11)	0.0203 (9)	0.0337 (10)	-0.0038 (8)	0.0046 (8)	-0.0065 (8)
C2	0.0253 (9)	0.0178 (8)	0.0224 (9)	-0.0022 (7)	0.0008 (7)	-0.0024 (6)
C3	0.0215 (8)	0.0189 (8)	0.0212 (8)	-0.0032 (7)	-0.0005 (7)	0.0008 (6)
C4	0.0300 (10)	0.0295 (10)	0.0256 (9)	-0.0034 (8)	-0.0061 (8)	0.0025 (8)
C5	0.0364 (11)	0.0414 (12)	0.0248 (10)	-0.0108 (9)	-0.0056 (8)	-0.0042 (8)

Geometric parameters (Å, °)

Zn1—O4	2.0618 (12)	O1W—H2	0.835 (10)
Zn1—O4 ⁱ	2.0618 (12)	N1—C1	1.340 (3)
Zn1—O1 ⁱ	2.1031 (13)	N1—C5	1.349 (3)
Zn1—O1	2.1031 (13)	N1—H3	0.846 (10)
Zn1—O1W ⁱ	2.1183 (15)	C1—C2	1.368 (3)
Zn1—O1W	2.1183 (15)	C1—H1A	0.9300
S1—O3	1.4496 (15)	C2—C3	1.434 (2)
S1—O2	1.4547 (17)	C3—C4	1.435 (2)
S1—O1	1.4681 (13)	C4—C5	1.352 (3)
S1—C2	1.7694 (19)	C4—H4	0.9300
O4—C3	1.274 (2)	C5—H5A	0.9300
O1W—H1	0.841 (10)		
O4—Zn1—O4 ⁱ	180.00 (7)	C3—O4—Zn1	133.12 (12)
O4—Zn1—O1 ⁱ	91.86 (5)	Zn1—O1W—H1	111.3 (18)
O4 ⁱ —Zn1—O1 ⁱ	88.14 (5)	Zn1—O1W—H2	112 (2)
O4—Zn1—O1	88.14 (5)	H1—O1W—H2	107 (3)
O4 ⁱ —Zn1—O1	91.86 (5)	C1—N1—C5	121.09 (18)
O1 ⁱ —Zn1—O1	180.0	C1—N1—H3	121 (2)
O4—Zn1—O1W ⁱ	90.38 (6)	C5—N1—H3	116 (2)
O4 ⁱ —Zn1—O1W ⁱ	89.62 (6)	N1—C1—C2	121.0 (2)
O1 ⁱ —Zn1—O1W ⁱ	88.76 (5)	N1—C1—H1A	119.5
O1—Zn1—O1W ⁱ	91.24 (5)	C2—C1—H1A	119.5
O4—Zn1—O1W	89.62 (6)	C1—C2—C3	120.70 (17)
O4 ⁱ —Zn1—O1W	90.38 (6)	C1—C2—S1	115.75 (15)
O1 ⁱ —Zn1—O1W	91.24 (5)	C3—C2—S1	123.06 (13)
O1—Zn1—O1W	88.76 (5)	O4—C3—C2	124.95 (16)
O1W ⁱ —Zn1—O1W	180.00 (7)	O4—C3—C4	120.14 (17)
O3—S1—O2	114.58 (10)	C2—C3—C4	114.91 (16)
O3—S1—O1	112.02 (9)	C5—C4—C3	121.1 (2)
O2—S1—O1	111.59 (9)	C5—C4—H4	119.4
O3—S1—C2	105.27 (9)	C3—C4—H4	119.4
O2—S1—C2	105.55 (9)	C4—C5—N1	121.12 (18)
O1—S1—C2	107.12 (8)	C4—C5—H5A	119.4

S1—O1—Zn1	123.21 (8)	N1—C5—H5A	119.4
O3—S1—O1—Zn1	170.03 (10)	O2—S1—C2—C1	-89.44 (17)
O2—S1—O1—Zn1	-59.98 (12)	O1—S1—C2—C1	151.53 (16)
C2—S1—O1—Zn1	55.08 (12)	O3—S1—C2—C3	-155.77 (16)
O4—Zn1—O1—S1	-40.93 (10)	O2—S1—C2—C3	82.66 (17)
O4 ⁱ —Zn1—O1—S1	139.07 (10)	O1—S1—C2—C3	-36.38 (18)
O1W ⁱ —Zn1—O1—S1	49.40 (10)	Zn1—O4—C3—C2	6.9 (3)
O1W—Zn1—O1—S1	-130.60 (10)	Zn1—O4—C3—C4	-173.69 (14)
O1 ⁱ —Zn1—O4—C3	-173.83 (17)	C1—C2—C3—O4	177.73 (19)
O1—Zn1—O4—C3	6.17 (17)	S1—C2—C3—O4	6.0 (3)
O1W ⁱ —Zn1—O4—C3	-85.06 (18)	C1—C2—C3—C4	-1.7 (3)
O1W—Zn1—O4—C3	94.94 (18)	S1—C2—C3—C4	-173.44 (15)
C5—N1—C1—C2	0.0 (3)	O4—C3—C4—C5	-177.7 (2)
N1—C1—C2—C3	0.9 (3)	C2—C3—C4—C5	1.8 (3)
N1—C1—C2—S1	173.18 (17)	C3—C4—C5—N1	-1.0 (4)
O3—S1—C2—C1	32.14 (19)	C1—N1—C5—C4	0.0 (3)

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

Hydrogen-bond geometry (\AA , $^\circ$)

$D-H\cdots A$	$D-H$	$H\cdots A$	$D\cdots A$	$D-H\cdots A$
O1w—H1 \cdots O2 ⁱ	0.84 (1)	2.05 (2)	2.797 (2)	147 (2)
O1w—H2 \cdots O4 ⁱⁱ	0.84 (1)	1.92 (1)	2.744 (2)	171 (3)
N1—H3 \cdots O3 ⁱⁱⁱ	0.85 (1)	1.91 (1)	2.754 (2)	175 (3)

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