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(Acetato-κO){2-[(2-aminoethyl-κN)disulfanyl]-ethanaminium}dichloridozinc(II)

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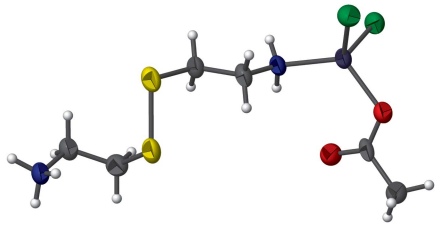
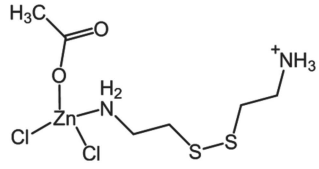
Structural data: full structural data are available from iucrdata.iucr.org

In the title compound, $[Zn(C_4H_{13}N_2S_2)(CH_3COO)Cl_2]$, the Zn^{II} ion is in a tetrahedral coordination geometry, coordinated by one acetate, two chloride and one 2-[(2-aminoethyl)disulfanyl]ethanaminium ligand, with a Zn—O distance of 1.977 (3) Å, a Zn—N distance of 2.015 (3) Å and Zn—Cl distances of 2.2673 (18) and 2.2688 (15) Å. In the crystal, molecules are self-assembled by N—H···Cl hydrogen bonds, leading to a one-dimensional chain structure. The chains interact with each other through N—H···O, N—H···S, C—H···Cl and C—H···S hydrogen bonding, completing a three-dimensional hydrogen-bonding network structure.

3D view



Chemical scheme



Structure description

Lead(II), bismuth(III), and copper(I) compounds with 2,2′-dithiobis(ethylamine) can show a variety of extended structural arrangements and are used as ionic conductive and non-linear optical materials (Louvain *et al.*, 2007, 2014; Tamura *et al.*, 2007; Bi *et al.*, 2008).

In the title compound, $[Zn(C_4H_{13}N_2S_2)(CH_3COO)Cl_2]$, the Zn(II) ion is in a tetrahedral coordination geometry, Fig. 1, coordinated by one acetate, two chloride and one 2-[(2-aminoethyl)disulfanyl]ethanaminium ligands, with a Zn1—O1 distance of 1.977 (3) Å, a Zn1—N1 distance of 2.015 (3) Å and Zn1—Cl distances of 2.2673 (18) and 2.2688 (15) Å. In the crystal, molecules are self-assembled by N—H···Cl hydrogen bonds, Table 1, leading to a one-dimensional chain structure. The chains interact with each other through N—H···O, N—H···S, C—H···Cl and C—H···S hydrogen bonding, completing a three-dimensional hydrogen-bonding network structure, Fig. 2.

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

D—H···A	D—H	H···A	D···A	D—H···A
N1—H1A···Cl2^{i}	0.91	2.53	3.436 (4)	172
N1—H1B···Cl2^{ii}	0.91	2.70	3.522 (4)	151
N1—H1B···O2	0.91	2.62	3.075 (4)	112
N2—H2A···O2^{iii}	0.91	1.84	2.730 (4)	164
N2—H2B···Cl1^{iv}	0.91	2.32	3.190 (4)	159
N2—H2C···S2	0.91	2.81	3.261 (3)	112
N2—H2C···O1^{v}	0.91	2.06	2.922 (4)	157
C2—H3···Cl2^{ii}	0.99	2.85	3.705 (4)	145
C3—H5···Cl1^{vi}	0.99	2.92	3.690 (5)	135
C4—H7···S1	0.99	2.88	3.412 (4)	115
C6—H9···S1^{v}	0.98	2.97	3.765 (5)	139

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

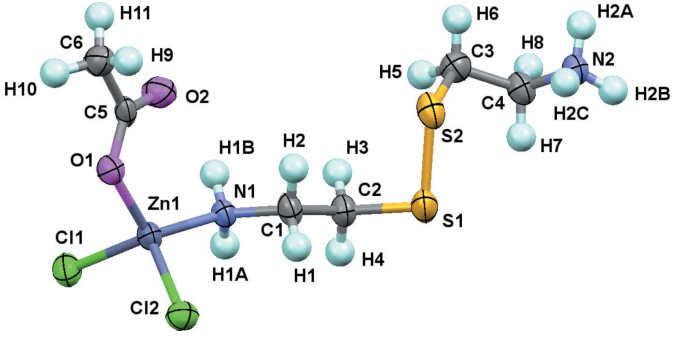

Figure 1
The molecular structure of the title compound, showing the atom-numbering scheme. Displacement ellipsoids are drawn at the 50% probability level and H atoms are drawn as spheres of arbitrary radii.

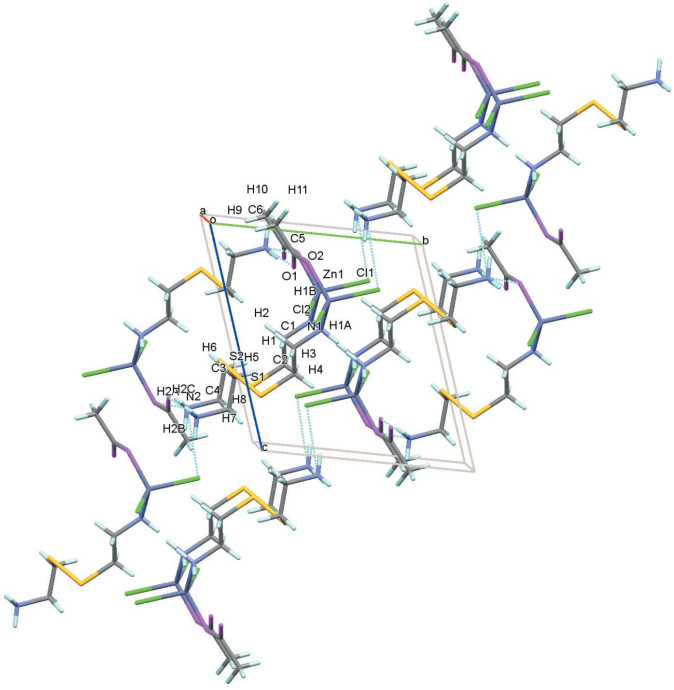

Figure 2
The crystal packing of the title compound viewed along a axis. Hydrogen bonds are shown as dashed lines.

Table 2
Experimental details.

Crystal data	
Chemical formula	$[Zn(C_4H_{13}N_2S_2)(C_2H_3O_2)Cl_2]$
M_r	348.60
Crystal system, space group	Triclinic, $P\overline{1}$
Temperature (K)	200
a, b, c (Å)	6.907 (4), 10.320 (7), 10.707 (7)
α, β, γ (°)	68.331 (14), 81.936 (15), 71.917 (12)
V (Å^3)	674.0 (8)
Z	2
Radiation type	Mo $K\alpha$
μ (mm^{-1})	2.51
Crystal size (mm)	0.20 × 0.10 × 0.10
Data collection	
Diffractometer	Rigaku R-AXIS 7
Absorption correction	Multi-scan (*ABSCOR*; Higashi, 1995)
T_{min}, T_{max}	0.466, 0.806
No. of measured, independent and observed [$I > 2\sigma(I)$] reflections	5387, 3039, 2471
R_{int}	0.039
(sin $\theta/\lambda)_{max}$ (Å^{-1})	0.649
Refinement	
$R[F^2 > 2\sigma(F^2)]$, $wR(F^2)$, S	0.049, 0.127, 1.10
No. of reflections	3039
No. of parameters	138
H-atom treatment	H-atom parameters constrained
$\Delta\rho_{max}$, $\Delta\rho_{min}$ (e Å^{-3})	1.20, −0.52

Computer programs: *PROCESS-AUTO* (Rigaku, 2000), *SHELXS2014* (Sheldrick, 2008), *SHELXL2014* (Sheldrick, 2015), *Yadokari-XG* (Kabuto *et al.*, 2009).

Synthesis and crystallization

To a solution of aminoethanethiol hydrochloride (19.0 mg, 0.167 mmol) in 1.5 ml of methanol was added 2-benzoylpyridine (30.1 mg, 0.164 mmol). The mixture was stirred at room temperature for 1 h. To the resulting solution was added a solution of $Zn(CH_3COO)_2$ (36.0 mg, 0.164 mmol) in 5 ml of methanol. The mixture was stirred at room temperature for 1 h. To the colorless solution was added $Ni(NO_3)_2{\cdot}6H_2O$ (7.1 mg, 0.024 mmol). The red solution was stirred at room temperature for 1.5 h to give a red solution, which was allowed to stand overnight. A small amount of colorless block-shaped crystals of $[Zn(C_4H_{13}N_2S_2)(C_2H_3O_2)Cl_2]$ was obtained.

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**, x160105 [doi:10.1107/S241431461600105X]

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(Acetato-κO){2-[(2-aminoethyl-κN)disulfanyl]ethan-1-aminium}dichloridozinc(II)

Crystal data

$[Zn(C_4H_{13}N_2S_2)(C_2H_3O_2)Cl_2]$
M_r = 348.60
Triclinic, $P\overline{1}$
a = 6.907 (4) Å
b = 10.320 (7) Å
c = 10.707 (7) Å
α = 68.331 (14)°
β = 81.936 (15)°
γ = 71.917 (12)°
V = 674.0 (8) Å^3
Z = 2
$F(000)$ = 356
D_x = 1.718 Mg m^{-3}
Mo $K\alpha$ radiation, λ = 0.71075 Å
Cell parameters from 1503 reflections
θ = 3.1–27.7°
μ = 2.51 mm^{-1}
T = 200 K
Block, colorless
0.20 × 0.10 × 0.10 mm

Data collection

Rigaku R-AXIS 7 diffractometer
Detector resolution: 10.00 pixels mm^{-1}
/w scans
Absorption correction: multi-scan (*ABSCOR*; Higashi, 1995)
T_{min} = 0.466, T_{max} = 0.806
5387 measured reflections
3039 independent reflections
2471 reflections with $I > 2\sigma(I)$
R_{int} = 0.039
θ_{max} = 27.5°, θ_{min} = 3.1°
h = −8→8
k = −13→13
l = −13→13

Refinement

Refinement on F^2
Least-squares matrix: full
$R[F^2 > 2\sigma(F^2)]$ = 0.049
$wR(F^2)$ = 0.127
S = 1.10
3039 reflections
138 parameters
0 restraints
Hydrogen site location: inferred from neighbouring sites
H-atom parameters constrained
$w = 1/[\sigma^2(F_o^2) + (0.0573P)^2 + 0.149P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max}$ = 0.001
$\Delta\rho_{max}$ = 1.20 e Å^{-3}
$\Delta\rho_{min}$ = −0.52 e Å^{-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.

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

	x	*y*	*z*	U_{iso}*/U_{eq}
Zn1	0.37629 (6)	0.52770 (4)	0.71825 (4)	0.03651 (17)
Cl1	0.46685 (15)	0.28147 (10)	0.79271 (10)	0.0476 (3)
Cl2	0.64255 (14)	0.61874 (10)	0.62265 (10)	0.0440 (3)
N1	0.1676 (4)	0.5888 (3)	0.5784 (3)	0.0355 (7)
H1A	0.2094	0.5278	0.5303	0.043*
H1B	0.0485	0.5755	0.6218	0.043*
N2	−0.5827 (5)	1.2558 (3)	0.1010 (3)	0.0407 (7)
H2A	−0.7085	1.2940	0.1325	0.061*
H2B	−0.5815	1.2885	0.0095	0.061*
H2C	−0.4884	1.2836	0.1288	0.061*
S1	−0.03739 (16)	0.94437 (11)	0.24546 (10)	0.0488 (3)
S2	−0.26116 (16)	1.05857 (11)	0.34154 (11)	0.0472 (3)
C1	0.1253 (6)	0.7383 (4)	0.4832 (4)	0.0395 (9)
H1	0.2556	0.7629	0.4521	0.047*
H2	0.0433	0.8054	0.5297	0.047*
C2	0.0112 (6)	0.7605 (4)	0.3617 (4)	0.0397 (9)
H3	−0.1198	0.7370	0.3924	0.048*
H4	0.0926	0.6931	0.3153	0.048*
O1	0.3115 (4)	0.5845 (3)	0.8799 (2)	0.0426 (6)
C3	−0.4926 (6)	1.0338 (4)	0.3006 (4)	0.0474 (10)
H5	−0.4811	0.9287	0.3338	0.057*
H6	−0.6103	1.0806	0.3486	0.057*
C5	0.1261 (5)	0.6340 (4)	0.9035 (4)	0.0365 (8)
O2	−0.0136 (4)	0.6437 (3)	0.8396 (3)	0.0526 (7)
C4	−0.5334 (6)	1.0955 (4)	0.1531 (4)	0.0432 (9)
H7	−0.4119	1.0551	0.1037	0.052*
H8	−0.6486	1.0662	0.1372	0.052*
C6	0.0799 (6)	0.6893 (5)	1.0234 (4)	0.0483 (10)
H9	0.0993	0.7864	0.9946	0.072*
H10	0.1723	0.6226	1.0963	0.072*
H11	−0.0614	0.6939	1.0554	0.072*

Atomic displacement parameters (Å²)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
Zn1	0.0315 (3)	0.0388 (3)	0.0309 (3)	−0.00430 (19)	−0.00581 (19)	−0.0056 (2)
Cl1	0.0531 (6)	0.0391 (5)	0.0416 (5)	−0.0057 (5)	−0.0049 (5)	−0.0087 (4)
Cl2	0.0361 (5)	0.0510 (6)	0.0440 (6)	−0.0130 (4)	0.0023 (4)	−0.0161 (5)
N1	0.0299 (15)	0.0360 (16)	0.0321 (16)	−0.0050 (13)	−0.0049 (13)	−0.0041 (13)
N2	0.0350 (16)	0.0451 (18)	0.0339 (17)	−0.0058 (14)	−0.0067 (14)	−0.0071 (14)
S1	0.0419 (5)	0.0464 (6)	0.0409 (6)	−0.0078 (5)	−0.0062 (5)	0.0021 (5)
S2	0.0519 (6)	0.0395 (5)	0.0438 (6)	−0.0048 (5)	−0.0164 (5)	−0.0087 (4)
C1	0.0365 (19)	0.0343 (19)	0.040 (2)	−0.0060 (16)	−0.0127 (17)	−0.0031 (17)
C2	0.040 (2)	0.039 (2)	0.034 (2)	−0.0057 (17)	−0.0078 (16)	−0.0068 (16)
O1	0.0351 (14)	0.0494 (16)	0.0365 (15)	−0.0056 (12)	−0.0047 (12)	−0.0111 (13)

C3	0.043 (2)	0.046 (2)	0.046 (2)	−0.0115 (18)	0.0021 (19)	−0.0107 (19)
C5	0.0272 (18)	0.0367 (19)	0.0322 (19)	−0.0080 (15)	−0.0094 (16)	0.0053 (15)
O2	0.0388 (15)	0.0707 (19)	0.0471 (17)	−0.0110 (14)	−0.0017 (13)	−0.0225 (15)
C4	0.041 (2)	0.042 (2)	0.043 (2)	−0.0066 (17)	−0.0096 (18)	−0.0110 (18)
C6	0.040 (2)	0.059 (2)	0.041 (2)	−0.0099 (19)	−0.0010 (18)	−0.0154 (19)

Geometric parameters (Å, º)

Zn1—O1	1.977 (3)	C1—H1	0.9900
Zn1—N1	2.015 (3)	C1—H2	0.9900
Zn1—Cl1	2.2673 (18)	C2—H3	0.9900
Zn1—Cl2	2.2688 (15)	C2—H4	0.9900
N1—C1	1.465 (4)	O1—C5	1.251 (4)
N1—H1A	0.9100	C3—C4	1.500 (5)
N1—H1B	0.9100	C3—H5	0.9900
N2—C4	1.478 (5)	C3—H6	0.9900
N2—H2A	0.9100	C5—O2	1.218 (4)
N2—H2B	0.9100	C5—C6	1.545 (5)
N2—H2C	0.9100	C4—H7	0.9900
S1—C2	1.800 (4)	C4—H8	0.9900
S1—S2	2.0384 (17)	C6—H9	0.9800
S2—C3	1.829 (4)	C6—H10	0.9800
C1—C2	1.524 (5)	C6—H11	0.9800
O1—Zn1—N1	121.89 (12)	C1—C2—H3	109.3
O1—Zn1—Cl1	106.49 (8)	S1—C2—H3	109.3
N1—Zn1—Cl1	104.45 (9)	C1—C2—H4	109.3
O1—Zn1—Cl2	102.22 (8)	S1—C2—H4	109.3
N1—Zn1—Cl2	109.45 (10)	H3—C2—H4	108.0
Cl1—Zn1—Cl2	112.50 (5)	C5—O1—Zn1	115.4 (2)
C1—N1—Zn1	117.2 (2)	C4—C3—S2	113.7 (3)
C1—N1—H1A	108.0	C4—C3—H5	108.8
Zn1—N1—H1A	108.0	S2—C3—H5	108.8
C1—N1—H1B	108.0	C4—C3—H6	108.8
Zn1—N1—H1B	108.0	S2—C3—H6	108.8
H1A—N1—H1B	107.2	H5—C3—H6	107.7
C4—N2—H2A	109.5	O2—C5—O1	125.7 (4)
C4—N2—H2B	109.5	O2—C5—C6	119.8 (3)
H2A—N2—H2B	109.5	O1—C5—C6	114.5 (3)
C4—N2—H2C	109.5	N2—C4—C3	111.7 (3)
H2A—N2—H2C	109.5	N2—C4—H7	109.3
H2B—N2—H2C	109.5	C3—C4—H7	109.3
C2—S1—S2	103.27 (14)	N2—C4—H8	109.3
C3—S2—S1	102.93 (14)	C3—C4—H8	109.3
N1—C1—C2	112.4 (3)	H7—C4—H8	107.9
N1—C1—H1	109.1	C5—C6—H9	109.5
C2—C1—H1	109.1	C5—C6—H10	109.5
N1—C1—H2	109.1	H9—C6—H10	109.5

C2—C1—H2	109.1	C5—C6—H11	109.5
H1—C1—H2	107.9	H9—C6—H11	109.5
C1—C2—S1	111.6 (2)	H10—C6—H11	109.5
Zn1—N1—C1—C2	164.8 (2)	Zn1—O1—C5—O2	3.3 (5)
N1—C1—C2—S1	−179.5 (3)	Zn1—O1—C5—C6	−175.2 (2)
S2—S1—C2—C1	−73.0 (3)	S2—C3—C4—N2	−67.0 (4)
S1—S2—C3—C4	−62.3 (3)		

Hydrogen-bond geometry (Å, º)

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
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