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catena-Poly[[tetraaquazinc(II)]-μ-1,3,4-thiadiazol-2,5-diyldithiodiacetato-κ^2O:O']

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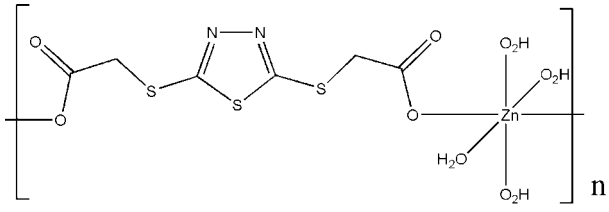
Key indicators: single-crystal X-ray study; T = 291 K; mean σ(C–C) = 0.003 Å; R factor = 0.021; wR factor = 0.047; data-to-parameter ratio = 15.3.

In the title linear coordination polymer, $[Zn(C_6H_4N_2O_4S_3)(H_2O)_4]_n$, the Zn^{II} atom is coordinated by four O atoms from four water molecules and two O atoms from two [5-(carboxylatomethylsulfanyl)-1,3,4-thiadiazol-2-ylsulfanyl]-acetate units in an octahedral coordination environment. The chains are linked into a three-dimensional supramolecular network *via* O—H···O and O—H···N hydrogen bonds.

Related literature

For the structure of other metal 1,3,4-thiadiazolyl-2,5-dithioacetates, see Gao *et al.* (2005, 2006); Zhang *et al.* (2006).



Experimental

Crystal data

$[Zn(C_6H_4N_2O_4S_3)(H_2O)_4]$
M_r = 401.73
Monoclinic, $P2_1$
a = 5.1554 (10) Å
b = 9.5043 (19) Å
c = 13.627 (3) Å
β = 94.82 (3)°
V = 665.3 (2) Å^3
Z = 2
Mo $K\alpha$ radiation
μ = 2.35 mm^{-1}
T = 291 (2) K
0.42 × 0.18 × 0.18 mm

Data collection

Rigaku R-AXIS RAPID diffractometer
Absorption correction: multi-scan (*ABSCOR*; Higashi, 1995)
T_{min} = 0.439, T_{max} = 0.675
6408 measured reflections
2774 independent reflections
2624 reflections with $I > 2\sigma(I)$
R_{int} = 0.022

Refinement

$R[F^2 > 2\sigma(F^2)]$ = 0.021
$wR(F^2)$ = 0.046
S = 1.06
2774 reflections
181 parameters
1 restraint
H-atom parameters constrained
$\Delta\rho_{max}$ = 0.26 e Å^{-3}
$\Delta\rho_{min}$ = −0.33 e Å^{-3}
Absolute structure: Flack (1983), 1151 Friedel pairs
Flack parameter: 0.014 (8)

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

D—H···A	D—H	H···A	D···A	D—H···A
O5—H6···O3	0.85	2.53	2.971 (3)	113
O5—H6···O6^{i}	0.85	2.23	3.053 (3)	165
O5—H5···N2^{i}	0.85	2.05	2.897 (3)	172
O6—H8···O4^{ii}	0.85	2.02	2.762 (2)	145
O6—H7···O7^{ii}	0.85	2.36	3.116 (3)	148
O7—H10···O3^{iii}	0.85	1.94	2.770 (3)	166
O7—H9···O1^{iv}	0.85	2.61	2.992 (2)	109
O7—H9···O2^{iv}	0.85	1.85	2.680 (3)	166
O8—H12···N1^{i}	0.85	1.98	2.819 (3)	172
O8—H11···O1^{v}	0.85	1.87	2.713 (2)	175

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

Data collection: *RAPID-AUTO* (Rigaku, 1998); cell refinement: *RAPID-AUTO*; data reduction: *CrystalStructure* (Rigaku/MSC, 2002); program(s) used to solve structure: *SHELXS97* (Sheldrick, 2008); program(s) used to refine structure: *SHELXL97* (Sheldrick, 2008); molecular graphics: *SHELXTL* (Sheldrick, 2008); software used to prepare material for publication: *SHELXL97*.

The authors thank Heilongjiang University for supporting this study.

Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: NG2446).

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supporting information

Acta Cryst. (2008). E**64**, m794 [doi:10.1107/S1600536808013251]

catena-Poly[[tetraaquazinc(II)]-μ-1,3,4-thiadiazol-2,5-diyldithiodiacetato-κ^2O:O']

Ying-Hui Yu, Chuan He, Guang-Feng Hou, Jin-Sheng Gao and Hong-Kun Zhang

S1. Comment

1,3,4-Thiadiazolyl-2,5-dithioacetic acid is a multidentate flexible aromatic carboxylic acid having two —S—CH_2CO_2H arms, and its N atoms can be considered as a potential coordinate candidate which also would coordinate with metal atoms fomed a supramolecular complexes. The structure of 1,3,4-Thiadiazolyl-2,5-dithioacetic acid was reported by Gao *et al.* (2005) and Zhang *et al.*, (2006). In this paper, we reporte a new one-dimensional title compound crystal structure, synthesized by the reaction of 1,3,4-Thiadiazolyl-2,5-dithioacetic acid and zinc dichloride in aqueous solution.

Complex (I) consists of molecules of tetraaquazinc(II)-1,3,4-thiadiazol-2,5-diyldithiodiacetato. The zinc atom is six-coordinated in an octahedron environment (Figure 1), each zinc atom connect with two 1,3,4-Thiadiazolyl-2,5-dithio-acetic acid ligand and four water molecules formed a one-dimensional chain structure along *c* axis (Figure 2).

There are eight symmetry-independent 'active′ H atoms in the crystal structure; all of them participate in hydrogen bonds, which link the one-dimensional chain structure into an infinite three-dimensional network (Table 1, Figure 3).

S2. Experimental

1,3,4-Thiadiazolyl-2,5-dithioacetic acid was prepared from 2,5-dimercapto-1,3,4-thiadiazole, using the method for synthesis of benzene-1,2-dioxyacetic acid reported by us (Gao *et al.*, 2006). The colorless zinc complex was obtained from the reaction of zinc dichloride hexahydrate (0.244 g, 1 mmol) and 1,3,4-Thiadiazolyl-2,5-dithioacetic acid (0.532 g, 2 mmol) in hot water (20 ml), and then the pH was adjusted to about 6 with 0.2 *M* sodium hydroxide. The resulting solution was filtered and allowed to stand in a desiccator at room temperature for several days.

S3. Refinement

H atoms bound to C atoms were placed in calculated positions and treated as riding on their parent atoms, with C—H = 0.97 Å (methylene) and with U_{iso}(H) = 1.2U_{eq}(C). Water H atoms were initially located in a difference Fourier map but they were treated as riding on their parent atoms with O—H = 0.85 Å, and with U_{iso}(H) = 1.5U_{eq}(O).

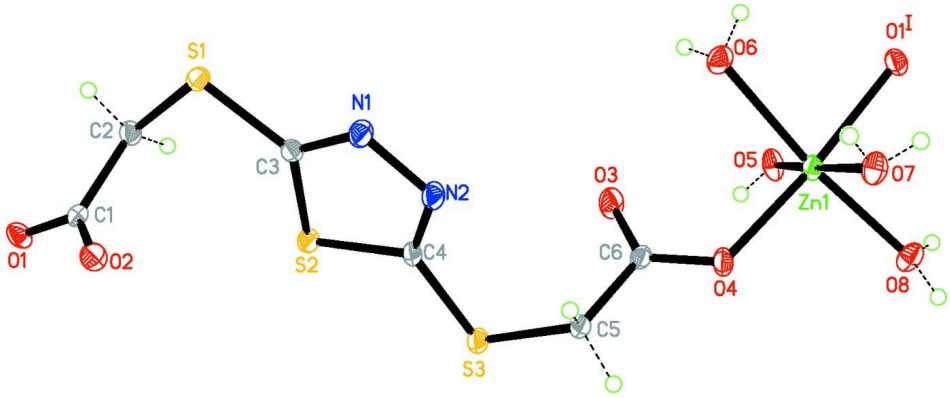

Figure 1

The molecular structure of (I), showing displacement ellipsoids at the 30% probability level for non-H atoms.

Figure 2

One-dimensional chain structure of the title complex. H atoms have been omitted for clarity.

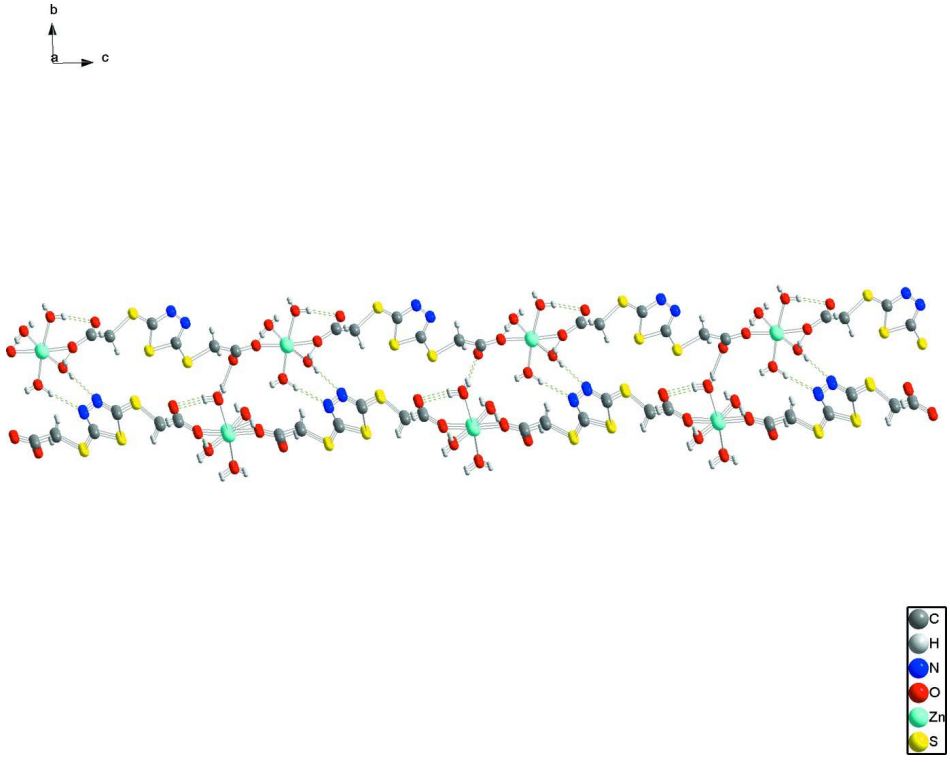

Figure 3

A partial packing view, showing the three-dimensional hydrogen-bonding network. Dashed lines indicate the hydrogen-bonding interactions. H atoms not involved in hydrogen bonds have been omitted for clarity.

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Hall symbol: P 2yb
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$b = 9.5043$ (19) Å
$c = 13.627$ (3) Å
$\beta = 94.82$ (3)°
$V = 665.3$ (2) Å^3
$Z = 2$
$F(000) = 408$
$D_x = 2.005$ Mg m^{-3}
Mo $K\alpha$ radiation, $\lambda = 0.71073$ Å
Cell parameters from 6179 reflections
$\theta = 3.0$–$27.5°$
$\mu = 2.35$ mm^{-1}
$T = 291$ K
Block, colorless
0.42 × 0.18 × 0.18 mm

Data collection

Rigaku R-AXIS RAPID diffractometer
Radiation source: fine-focus sealed tube
Graphite monochromator
ω scans
Absorption correction: multi-scan (*ABSCOR*; Higashi, 1995)
$T_{min} = 0.439$, $T_{max} = 0.675$
6408 measured reflections
2774 independent reflections
2624 reflections with $I > 2\sigma(I)$
$R_{int} = 0.022$
$\theta_{max} = 27.5°$, $\theta_{min} = 3.0°$
$h = -6 \rightarrow 6$
$k = -11 \rightarrow 12$
$l = -17 \rightarrow 17$

Refinement

Refinement on F^2
Least-squares matrix: full
$R[F^2 > 2\sigma(F^2)] = 0.021$
$wR(F^2) = 0.046$
$S = 1.06$
2774 reflections
181 parameters
1 restraint
Primary atom site location: structure-invariant direct methods
Secondary atom site location: difference Fourier map
Hydrogen site location: inferred from neighbouring sites
H-atom parameters constrained
$w = 1/[\sigma^2(F_o^2) + (0.0189P)^2]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} = 0.001$
$\Delta\rho_{max} = 0.26$ e Å^{-3}
$\Delta\rho_{min} = -0.33$ e Å^{-3}
Absolute structure: Flack (1983), 1151 Friedel pairs
Absolute structure parameter: 0.014 (8)

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	−0.1447 (4)	0.4252 (3)	1.17156 (18)	0.0201 (5)
C2	−0.3790 (4)	0.4114 (3)	1.09577 (18)	0.0218 (5)
H1	−0.5359	0.4172	1.1301	0.026*

H2	−0.3749	0.3184	1.0665	0.026*
C3	−0.1709 (4)	0.4813 (2)	0.92174 (18)	0.0199 (5)
C4	0.1357 (4)	0.3689 (2)	0.82727 (18)	0.0213 (5)
C5	0.4372 (4)	0.3491 (3)	0.67199 (16)	0.0211 (4)
H3	0.4337	0.4499	0.6833	0.025*
H4	0.6113	0.3248	0.6558	0.025*
C6	0.2476 (4)	0.3147 (2)	0.58475 (18)	0.0207 (5)
N1	−0.1311 (4)	0.5509 (2)	0.84268 (16)	0.0278 (5)
N2	0.0486 (4)	0.4848 (2)	0.78684 (16)	0.0280 (5)
O1	−0.1686 (3)	0.3552 (2)	1.24944 (12)	0.0262 (4)
O2	0.0419 (3)	0.5006 (2)	1.15424 (13)	0.0272 (4)
O3	0.0336 (3)	0.2622 (2)	0.59581 (14)	0.0328 (4)
O4	0.3282 (3)	0.3463 (3)	0.50187 (11)	0.0275 (3)
O5	−0.0626 (3)	0.14243 (19)	0.39464 (14)	0.0290 (4)
H5	−0.0598	0.0891	0.3447	0.044*
H6	0.0049	0.1028	0.4466	0.044*
O6	−0.2103 (3)	0.45593 (19)	0.44603 (14)	0.0279 (4)
H7	−0.3137	0.5040	0.4074	0.042*
H8	−0.3032	0.3988	0.4759	0.042*
O7	0.2564 (3)	0.54165 (19)	0.33802 (14)	0.0274 (4)
H9	0.2034	0.5402	0.2772	0.041*
H10	0.1894	0.6112	0.3658	0.041*
O8	0.3690 (3)	0.2453 (2)	0.29289 (14)	0.0291 (4)
H11	0.5185	0.2761	0.2817	0.044*
H12	0.3081	0.1896	0.2479	0.044*
S1	−0.40220 (11)	0.53897 (6)	0.99806 (5)	0.02380 (13)
S2	0.00595 (10)	0.32704 (7)	0.93689 (4)	0.02359 (12)
S3	0.37405 (12)	0.26074 (7)	0.78411 (5)	0.02603 (14)
Zn1	0.09545 (4)	0.34105 (3)	0.374965 (19)	0.02112 (7)

Atomic displacement parameters (Å²)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
C1	0.0183 (10)	0.0232 (12)	0.0194 (14)	0.0027 (9)	0.0046 (9)	−0.0024 (10)
C2	0.0213 (10)	0.0259 (12)	0.0183 (13)	−0.0012 (9)	0.0025 (9)	0.0012 (10)
C3	0.0216 (11)	0.0202 (12)	0.0177 (13)	0.0025 (9)	0.0008 (8)	−0.0002 (9)
C4	0.0243 (10)	0.0253 (14)	0.0143 (12)	0.0034 (9)	0.0026 (8)	0.0016 (9)
C5	0.0212 (9)	0.0246 (11)	0.0180 (11)	0.0037 (11)	0.0039 (8)	0.0002 (12)
C6	0.0219 (10)	0.0218 (13)	0.0187 (12)	0.0012 (9)	0.0043 (8)	−0.0012 (9)
N1	0.0349 (11)	0.0273 (11)	0.0222 (12)	0.0108 (9)	0.0086 (9)	0.0074 (9)
N2	0.0352 (11)	0.0306 (12)	0.0193 (12)	0.0100 (9)	0.0089 (9)	0.0079 (9)
O1	0.0229 (7)	0.0355 (10)	0.0199 (9)	−0.0069 (8)	−0.0007 (6)	0.0066 (9)
O2	0.0226 (8)	0.0360 (10)	0.0233 (10)	−0.0072 (7)	0.0047 (7)	0.0047 (8)
O3	0.0296 (9)	0.0432 (11)	0.0261 (11)	−0.0132 (8)	0.0053 (8)	0.0036 (9)
O4	0.0249 (7)	0.0422 (9)	0.0159 (8)	−0.0072 (9)	0.0044 (6)	−0.0006 (10)
O5	0.0394 (10)	0.0293 (9)	0.0182 (10)	−0.0110 (8)	0.0016 (7)	−0.0012 (8)
O6	0.0249 (8)	0.0308 (10)	0.0291 (11)	−0.0037 (7)	0.0088 (7)	0.0034 (8)
O7	0.0337 (9)	0.0244 (9)	0.0250 (10)	−0.0053 (7)	0.0079 (7)	−0.0032 (8)

O8	0.0223 (8)	0.0364 (11)	0.0296 (11)	−0.0093 (7)	0.0075 (7)	−0.0132 (8)
S1	0.0246 (3)	0.0267 (3)	0.0207 (3)	0.0070 (2)	0.0053 (2)	0.0032 (2)
S2	0.0284 (2)	0.0245 (3)	0.0185 (3)	0.0081 (3)	0.0058 (2)	0.0065 (3)
S3	0.0302 (3)	0.0303 (3)	0.0183 (3)	0.0106 (3)	0.0058 (2)	0.0043 (3)
Zn1	0.02055 (11)	0.02492 (14)	0.01799 (14)	−0.00523 (11)	0.00214 (9)	−0.00268 (13)

Geometric parameters (Å, º)

C1—O2	1.238 (3)	C6—O4	1.272 (3)
C1—O1	1.267 (3)	N1—N2	1.397 (3)
C1—C2	1.527 (3)	O1—Zn1^{i}	2.0989 (17)
C2—S1	1.797 (2)	O4—Zn1	2.0209 (17)
C2—H1	0.9700	O5—Zn1	2.0824 (18)
C2—H2	0.9700	O5—H5	0.8500
C3—N1	1.295 (3)	O5—H6	0.8500
C3—S2	1.730 (2)	O6—Zn1	2.2072 (17)
C3—S1	1.736 (2)	O6—H7	0.8500
C4—N2	1.295 (3)	O6—H8	0.8500
C4—S2	1.734 (2)	O7—Zn1	2.1557 (18)
C4—S3	1.742 (2)	O7—H9	0.8501
C5—C6	1.510 (3)	O7—H10	0.8500
C5—S3	1.797 (2)	O8—Zn1	2.0815 (17)
C5—H3	0.9700	O8—H11	0.8500
C5—H4	0.9700	O8—H12	0.8500
C6—O3	1.231 (3)	Zn1—O1^{ii}	2.0989 (17)
O2—C1—O1	126.5 (2)	Zn1—O5—H6	111.8
O2—C1—C2	120.2 (2)	H5—O5—H6	111.7
O1—C1—C2	113.2 (2)	Zn1—O6—H7	115.4
C1—C2—S1	116.27 (17)	Zn1—O6—H8	110.2
C1—C2—H1	108.2	H7—O6—H8	106.9
S1—C2—H1	108.2	Zn1—O7—H9	96.6
C1—C2—H2	108.2	Zn1—O7—H10	113.9
S1—C2—H2	108.2	H9—O7—H10	109.7
H1—C2—H2	107.4	Zn1—O8—H11	127.7
N1—C3—S2	114.44 (18)	Zn1—O8—H12	115.7
N1—C3—S1	120.11 (17)	H11—O8—H12	111.7
S2—C3—S1	125.31 (14)	C3—S1—C2	102.97 (11)
N2—C4—S2	114.60 (17)	C3—S2—C4	86.59 (11)
N2—C4—S3	125.98 (19)	C4—S3—C5	101.23 (12)
S2—C4—S3	119.35 (13)	O4—Zn1—O8	95.17 (7)
C6—C5—S3	114.60 (17)	O4—Zn1—O5	97.06 (8)
C6—C5—H3	108.6	O8—Zn1—O5	87.88 (7)
S3—C5—H3	108.6	O4—Zn1—O1^{ii}	173.48 (9)
C6—C5—H4	108.6	O8—Zn1—O1^{ii}	90.71 (7)
S3—C5—H4	108.6	O5—Zn1—O1^{ii}	85.94 (7)
H3—C5—H4	107.6	O4—Zn1—O7	88.02 (8)
O3—C6—O4	124.6 (2)	O8—Zn1—O7	88.26 (7)

O3—C6—C5	121.2 (2)	O5—Zn1—O7	173.88 (8)
O4—C6—C5	114.17 (19)	O1^{ii}—Zn1—O7	89.36 (7)
C3—N1—N2	112.4 (2)	O4—Zn1—O6	90.40 (7)
C4—N2—N1	111.9 (2)	O8—Zn1—O6	173.27 (8)
C1—O1—Zn1^{i}	127.90 (15)	O5—Zn1—O6	95.18 (7)
C6—O4—Zn1	122.62 (14)	O1^{ii}—Zn1—O6	83.55 (7)
Zn1—O5—H5	113.8	O7—Zn1—O6	88.17 (6)

Symmetry codes: (i) x, y, $z+1$; (ii) x, y, $z-1$.

Hydrogen-bond geometry (Å, º)

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
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O8—H12···N1^{iii}	0.85	1.98	2.819 (3)	172
O8—H11···O1^{vi}	0.85	1.87	2.713 (2)	175

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