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[(*E*)-But-2-enoato-κO]chlorido(2,2′-diamino-4,4′-bi-1,3-thiazole-$\kappa^2N^3,N^{3'}$)-zinc(II) monohydrate

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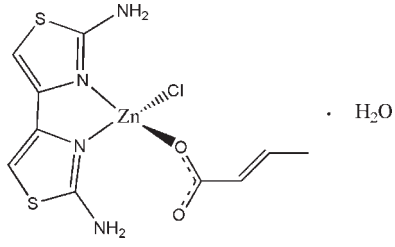
Key indicators: single-crystal X-ray study; T = 294 K; mean σ(C–C) = 0.005 Å; R factor = 0.032; wR factor = 0.080; data-to-parameter ratio = 14.3.

In the title compound, $[Zn(C_4H_5O_2)Cl(C_6H_6N_4S_2)]\cdot H_2O$, the Zn^{II} cation is coordinated by a bidentate diaminobithiazole (DABT) ligand, a but-2-enoate anion and a Cl^- anion in a distorted tetrahedral geometry. Within the DABT ligand, the two thiazole rings are twisted to each other at a dihedral angle of 4.38 (10)°. An intramolecular N—H···O interaction occurs. The centroid–centroid distance of 3.6650 (17) Å and partially overlapped arrangement between nearly parallel thiazole rings of adjacent complexes indicate the existence of π–π stacking in the crystal structure. Extensive O—H···Cl, O—H···O, N—H···Cl and N—H···O hydrogen bonding helps to stabilize the crystal structure.

Related literature

For the potential applications of metal complexes of diaminobithiazole in the biological field, see: Waring (1981); Fisher *et al.* (1985). For dihedral angles between thiazole rings in diaminobithiazole complexes, see: Du *et al.* (2010); Zhang *et al.* (2006).



Experimental

Crystal data

$[Zn(C_4H_5O_2)Cl(C_6H_6N_4S_2)]\cdot H_2O$
M_r = 402.18
Monoclinic, $P2_1/n$
a = 7.2782 (13) Å
b = 16.2846 (16) Å
c = 13.237 (2) Å
β = 99.252 (16)°
V = 1548.5 (4) Å^3
Z = 4
Mo $K\alpha$ radiation
μ = 2.04 mm^{-1}
T = 294 K
0.36 × 0.30 × 0.24 mm

Data collection

Rigaku R-AXIS RAPID IP diffractometer
Absorption correction: multi-scan (*ABSCOR*; Higashi, 1995)
T_{min} = 0.75, T_{max} = 0.88
7862 measured reflections
2735 independent reflections
2293 reflections with $I > 2\sigma(I)$
R_{int} = 0.026

Refinement

$R[F^2 > 2\sigma(F^2)]$ = 0.032
$wR(F^2)$ = 0.080
S = 1.05
2735 reflections
191 parameters
H-atom parameters constrained
$\Delta\rho_{max}$ = 0.71 e Å^{-3}
$\Delta\rho_{min}$ = −0.45 e Å^{-3}

Table 1
Selected bond lengths (Å).

Zn—O1	1.961 (2)	Zn—N3	2.060 (2)
Zn—N1	2.029 (2)	Zn—Cl1	2.2223 (9)

Table 2
Hydrogen-bond geometry (Å, °).

D—H···A	D—H	H···A	D···A	D—H···A
O1W—H1A···Cl1^{i}	0.86	2.56	3.345 (3)	152
O1W—H1B···O1	0.86	2.04	2.859 (4)	160
N2—H2A···O2	0.86	2.22	2.959 (4)	144
N2—H2B···O1W^{ii}	0.86	2.23	3.032 (4)	154
N4—H4A···O1W	0.86	2.30	3.043 (4)	145
N4—H4B···Cl1^{iii}	0.86	2.66	3.393 (3)	144

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

Data collection: *PROCESS-AUTO* (Rigaku, 1998); cell refinement: *PROCESS-AUTO*; data reduction: *CrystalStructure* (Rigaku/MSC, 2002); program(s) used to solve structure: *SIR92* (Altomare *et al.*, 1993); program(s) used to refine structure: *SHELXL97* (Sheldrick, 2008); molecular graphics: *ORTEP-3 for Windows* (Farrugia, 1997); software used to prepare material for publication: *WinGX* (Farrugia, 1999).

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Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: NG2750).

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supporting information

Acta Cryst. (2010). E**66**, m466 [doi:10.1107/S160053681001113X]

[(*E*)-But-2-enoato-κO]chlorido(2,2′-diamino-4,4′-bi-1,3-thiazole-$\kappa^2N^3,N^{3'}$)zinc(II) monohydrate

Mei Du, Yan-Li Wang, Bing-Xin Liu and Duan-Jun Xu

S1. Comment

Some metal complexes with 2,2′-diamino-4,4′-bi-1,3-thiazole (DABT) have shown the potential application in the biological field (Waring, 1981; Fisher *et al.*, 1985). As a part of serial structural investigation of metal complexes with DABT, the title Zn^{II} complex was prepared in the laboratory and its X-ray structure is presented here.

The molecular structure of the title compound is shown in Fig. 1. The Zn^{II} cation is coordinated by a diaminobithiazole (DABT) ligand, a but-2-enoate anion and a Cl^- anion in a distorted tetrahedral geometry (Table 1). Within the DABT ligand the two thiazole rings are twisted to each other at a dihedral angle of 4.38 (10)°, which agrees with 9.51 (17)° found in a Pb^{II} complex of DABT (Du *et al.*, 2010) and 9.5 (2)° found in a Cd^{II} complex of DABT (Zhang *et al.*, 2006). The partially overlapped arrangement of centroids distance of 3.6650 (17) Å between nearly parallel thiazole rings of the adjacent complexes indicate the existence of π-π stacking in the crystal structure (Fig. 2). The extensive hydrogen bonding help to stabilize the crystal structure (Table 2).

S2. Experimental

A water-ethanol solution (20 ml, 1:1) of DABT (0.10 g, 0.5 mmol) and $ZnCl_2$ (0.07 g, 0.5 mmol) was refluxed for 10 min, then an aqueous solution (20 ml) of (E)-but-2-enoatic acid (0.09 g, 1 mmol) and NaOH (0.04 g, 1 mmol) was mixed with the above solution. The mixture was refluxed for 6 h and then filtered. The single crystals of the title compound were obtained from the filtrate after a week.

S3. Refinement

H atoms of water molecule were located in a difference Fourier map and were refined as riding in as-found relative positions with $U_{iso}(H) = 1.2U_{eq}(O)$. Other H atoms were placed in calculated positions with C—H = 0.96 Å (methyl), 0.93 Å (aromatic) and N—H = 0.86 Å, and refined in the riding model with $U_{iso}(H) = 1.5U_{eq}(C)$ for methyl and $1.2U_{eq}(C,N)$ for the others.

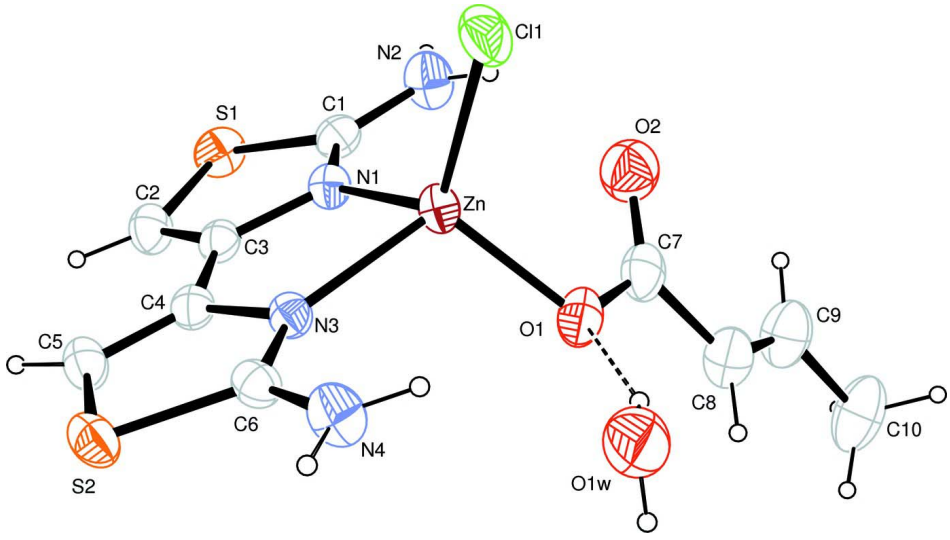

Figure 1

The molecular structure of the title compound with 40% probability displacement ellipsoids. Dashed lines indicate the hydrogen bonding.

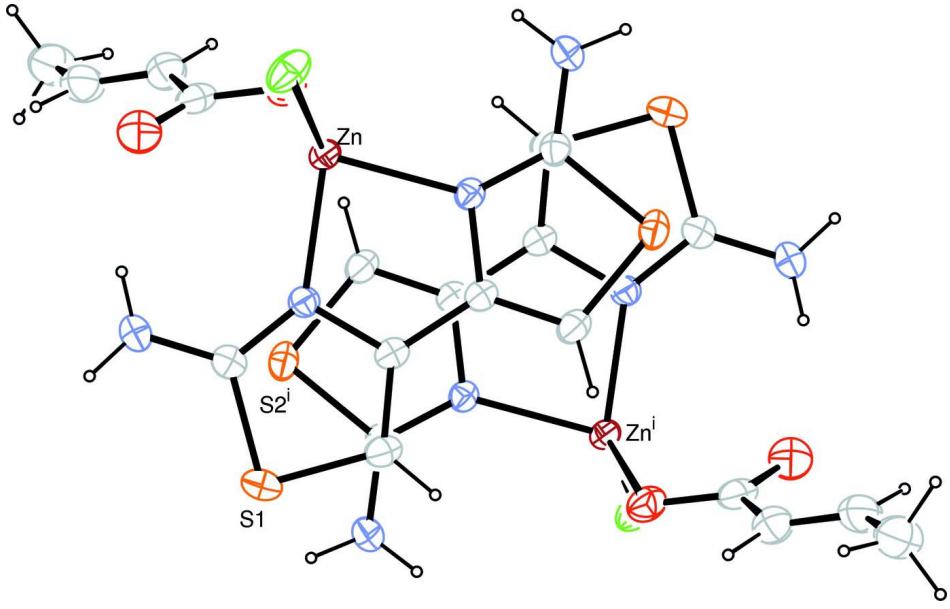

Figure 2

The partially overlapped arrangement between thiazole rings showing π-π stacking [symmetry code: (i) 1-x, -y, 1-z].

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Crystal data

$[Zn(C_4H_5O_2)Cl(C_6H_6N_4S_2)]\cdot H_2O$
M_r = 402.18
Monoclinic, $P2_1/n$
Hall symbol: -P 2yn
a = 7.2782 (13) Å
b = 16.2846 (16) Å
c = 13.237 (2) Å
β = 99.252 (16)°
V = 1548.5 (4) Å^3
Z = 4
$F(000)$ = 816
D_x = 1.725 Mg m^{-3}

Mo $K\alpha$ radiation, λ = 0.71073 Å
Cell parameters from 3446 reflections
θ = 2.0–24.6°
μ = 2.04 mm^{-1}
T = 294 K
Block, yellow
0.36 × 0.30 × 0.24 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} = 0.75, T_{max} = 0.88
7862 measured reflections
2735 independent reflections
2293 reflections with $I > 2\sigma(I)$
R_{int} = 0.026
θ_{max} = 25.0°, θ_{min} = 2.0°
h = −8→8
k = −12→19
l = −15→15

Refinement

Refinement on F^2
Least-squares matrix: full
$R[F^2 > 2\sigma(F^2)]$ = 0.032
$wR(F^2)$ = 0.080
S = 1.05
2735 reflections
191 parameters
0 restraints
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
$w = 1/[\sigma^2(F_o^2) + (0.0353P)^2 + 1.093P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max}$ = 0.002
$\Delta\rho_{max}$ = 0.71 e Å^{-3}
$\Delta\rho_{min}$ = −0.45 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.

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 (Å²)

	x	y	z	U_{iso}*/U_{eq}
Zn	0.77894 (5)	0.12724 (2)	0.64617 (2)	0.03260 (13)
Cl1	1.01129 (13)	0.21725 (6)	0.67179 (7)	0.0541 (3)
S1	0.93933 (12)	−0.14538 (5)	0.65212 (7)	0.0433 (2)
S2	0.56191 (12)	0.09881 (5)	0.30033 (6)	0.0418 (2)
N1	0.8456 (3)	0.00649 (15)	0.64140 (17)	0.0314 (6)
N2	0.9835 (4)	−0.03495 (18)	0.8057 (2)	0.0483 (7)
H2A	0.9737	0.0134	0.8305	0.058*
H2B	1.0326	−0.0740	0.8447	0.058*
N3	0.6716 (3)	0.10813 (15)	0.49432 (17)	0.0299 (5)
N4	0.5365 (4)	0.22969 (16)	0.4215 (2)	0.0444 (7)
H4A	0.5508	0.2545	0.4796	0.053*
H4B	0.4859	0.2549	0.3670	0.053*

O1	0.5737 (3)	0.15367 (14)	0.72007 (17)	0.0471 (6)
O2	0.7729 (4)	0.11162 (17)	0.8535 (2)	0.0630 (7)
O1W	0.4071 (4)	0.29467 (17)	0.6138 (2)	0.0726 (8)
H1A	0.2909	0.2835	0.6079	0.087*
H1B	0.4522	0.2592	0.6587	0.087*
C1	0.9224 (4)	−0.04933 (19)	0.7066 (2)	0.0351 (7)
C2	0.8373 (4)	−0.10659 (19)	0.5354 (2)	0.0396 (8)
H2	0.8137	−0.1368	0.4751	0.047*
C3	0.7965 (4)	−0.02684 (18)	0.5437 (2)	0.0316 (7)
C4	0.7091 (4)	0.02855 (18)	0.4637 (2)	0.0309 (7)
C5	0.6607 (4)	0.0136 (2)	0.3637 (2)	0.0386 (7)
H5	0.6782	−0.0363	0.3325	0.046*
C6	0.5924 (4)	0.15275 (19)	0.4159 (2)	0.0333 (7)
C7	0.6148 (5)	0.1325 (2)	0.8138 (3)	0.0448 (8)
C8	0.4511 (6)	0.1316 (2)	0.8698 (3)	0.0541 (9)
H8	0.3377	0.1509	0.8357	0.065*
C9	0.4583 (6)	0.1061 (2)	0.9613 (3)	0.0623 (11)
H9	0.5745	0.0909	0.9963	0.075*
C10	0.2972 (7)	0.0984 (3)	1.0172 (3)	0.0724 (13)
H10A	0.3061	0.1397	1.0696	0.109*
H10B	0.2981	0.0450	1.0479	0.109*
H10C	0.1834	0.1057	0.9702	0.109*

Atomic displacement parameters (Å²)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
Zn	0.0384 (2)	0.0298 (2)	0.0298 (2)	−0.00060 (15)	0.00584 (14)	−0.00417 (15)
Cl1	0.0597 (6)	0.0545 (6)	0.0507 (5)	−0.0236 (4)	0.0161 (4)	−0.0186 (4)
S1	0.0460 (5)	0.0295 (4)	0.0551 (5)	0.0035 (4)	0.0103 (4)	0.0042 (4)
S2	0.0460 (5)	0.0489 (5)	0.0286 (4)	−0.0057 (4)	0.0004 (3)	−0.0015 (4)
N1	0.0353 (14)	0.0292 (13)	0.0305 (13)	−0.0011 (11)	0.0076 (10)	0.0008 (11)
N2	0.0629 (19)	0.0439 (17)	0.0357 (15)	0.0110 (14)	0.0010 (13)	0.0064 (13)
N3	0.0316 (13)	0.0310 (13)	0.0279 (12)	−0.0041 (11)	0.0069 (10)	−0.0008 (10)
N4	0.0572 (18)	0.0363 (16)	0.0371 (15)	0.0029 (13)	0.0001 (13)	0.0059 (12)
O1	0.0528 (15)	0.0469 (14)	0.0449 (14)	0.0028 (11)	0.0173 (11)	−0.0083 (11)
O2	0.0653 (19)	0.0609 (17)	0.0627 (17)	0.0061 (14)	0.0103 (14)	−0.0015 (14)
O1W	0.0642 (18)	0.072 (2)	0.084 (2)	−0.0139 (15)	0.0188 (15)	−0.0228 (16)
C1	0.0323 (17)	0.0348 (17)	0.0397 (18)	−0.0008 (14)	0.0100 (13)	0.0035 (14)
C2	0.046 (2)	0.0319 (18)	0.0428 (18)	−0.0028 (14)	0.0115 (15)	−0.0064 (14)
C3	0.0298 (16)	0.0315 (17)	0.0353 (16)	−0.0052 (13)	0.0110 (12)	−0.0043 (13)
C4	0.0310 (16)	0.0304 (16)	0.0329 (16)	−0.0055 (13)	0.0098 (12)	−0.0025 (13)
C5	0.0449 (19)	0.0372 (18)	0.0343 (17)	−0.0060 (15)	0.0081 (14)	−0.0069 (14)
C6	0.0337 (17)	0.0347 (17)	0.0309 (16)	−0.0053 (13)	0.0038 (13)	0.0006 (13)
C7	0.056 (2)	0.0315 (18)	0.050 (2)	−0.0028 (16)	0.0166 (17)	−0.0093 (16)
C8	0.062 (2)	0.051 (2)	0.051 (2)	0.0083 (18)	0.0151 (18)	−0.0034 (18)
C9	0.079 (3)	0.050 (2)	0.062 (3)	0.014 (2)	0.022 (2)	0.002 (2)
C10	0.097 (3)	0.063 (3)	0.069 (3)	0.017 (2)	0.048 (3)	0.011 (2)

Geometric parameters (Å, º)

Zn—O1	1.961 (2)	O1—C7	1.275 (4)
Zn—N1	2.029 (2)	O2—C7	1.234 (4)
Zn—N3	2.060 (2)	O1W—H1A	0.8564
Zn—Cl1	2.2223 (9)	O1W—H1B	0.8550
S1—C2	1.723 (3)	C2—C3	1.340 (4)
S1—C1	1.735 (3)	C2—H2	0.9300
S2—C5	1.718 (3)	C3—C4	1.457 (4)
S2—C6	1.747 (3)	C4—C5	1.336 (4)
N1—C1	1.315 (4)	C5—H5	0.9300
N1—C3	1.395 (4)	C7—C8	1.502 (5)
N2—C1	1.336 (4)	C8—C9	1.273 (5)
N2—H2A	0.8600	C8—H8	0.9300
N2—H2B	0.8600	C9—C10	1.490 (5)
N3—C6	1.321 (4)	C9—H9	0.9300
N3—C4	1.398 (4)	C10—H10A	0.9600
N4—C6	1.323 (4)	C10—H10B	0.9600
N4—H4A	0.8600	C10—H10C	0.9600
N4—H4B	0.8600		
O1—Zn—N1	115.69 (10)	C2—C3—N1	115.2 (3)
O1—Zn—N3	108.68 (10)	C2—C3—C4	128.1 (3)
N1—Zn—N3	83.01 (9)	N1—C3—C4	116.7 (2)
O1—Zn—Cl1	113.65 (7)	C5—C4—N3	115.0 (3)
N1—Zn—Cl1	117.66 (7)	C5—C4—C3	128.5 (3)
N3—Zn—Cl1	114.11 (7)	N3—C4—C3	116.5 (2)
C2—S1—C1	89.60 (15)	C4—C5—S2	111.1 (2)
C5—S2—C6	89.66 (15)	C4—C5—H5	124.5
C1—N1—C3	111.0 (2)	S2—C5—H5	124.5
C1—N1—Zn	136.7 (2)	N3—C6—N4	125.2 (3)
C3—N1—Zn	112.28 (18)	N3—C6—S2	112.9 (2)
C1—N2—H2A	120.0	N4—C6—S2	121.9 (2)
C1—N2—H2B	120.0	O2—C7—O1	123.1 (3)
H2A—N2—H2B	120.0	O2—C7—C8	123.1 (3)
C6—N3—C4	111.3 (2)	O1—C7—C8	113.7 (3)
C6—N3—Zn	137.1 (2)	C9—C8—C7	124.0 (4)
C4—N3—Zn	111.23 (18)	C9—C8—H8	118.0
C6—N4—H4A	120.0	C7—C8—H8	118.0
C6—N4—H4B	120.0	C8—C9—C10	125.8 (4)
H4A—N4—H4B	120.0	C8—C9—H9	117.1
C7—O1—Zn	110.3 (2)	C10—C9—H9	117.1
H1A—O1W—H1B	100.6	C9—C10—H10A	109.5
N1—C1—N2	124.2 (3)	C9—C10—H10B	109.5
N1—C1—S1	113.6 (2)	H10A—C10—H10B	109.5
N2—C1—S1	122.1 (2)	C9—C10—H10C	109.5
C3—C2—S1	110.6 (2)	H10A—C10—H10C	109.5
C3—C2—H2	124.7	H10B—C10—H10C	109.5

S1—C2—H2	124.7

Hydrogen-bond geometry (Å, º)

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
O1*W*—H1*A*···Cl1^{i}	0.86	2.56	3.345 (3)	152
O1*W*—H1*B*···O1	0.86	2.04	2.859 (4)	160
N2—H2*A*···O2	0.86	2.22	2.959 (4)	144
N2—H2*B*···O1*W*^{ii}	0.86	2.23	3.032 (4)	154
N4—H4*A*···O1*W*	0.86	2.30	3.043 (4)	145
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