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Bis[μ-5-(5-carboxylato-3-pyridyl)tetrazolato-$\kappa^3N^1,N^5:N^2$]bis[triaquazinc(II)]

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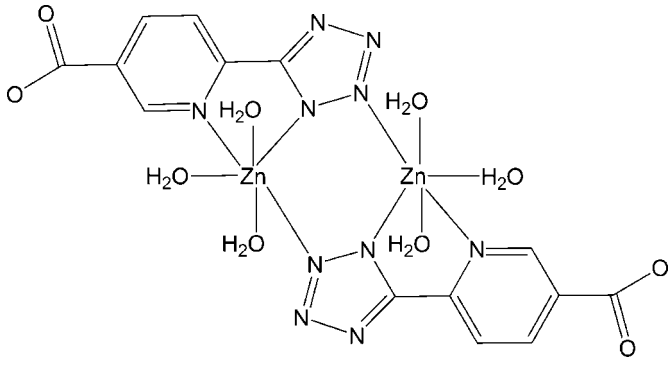
Key indicators: single-crystal X-ray study; T = 295 K; mean σ(C–C) = 0.003 Å; R factor = 0.032; wR factor = 0.064; data-to-parameter ratio = 13.4.

In the title complex, $[Zn_2(C_7H_3N_5O_2)_2(H_2O)_6]$, the 5-(5-carboxylato-3-pyridyl)tetrazolate ligand chelates the Zn^{II} center through one pyridyl N and one tetrazolate N atom, and uses another N atom to bridge to the second Zn atom, forming a centrosymmetric dinuclear unit. Three coordinated water molecules complete the distorted octahedral geometry of the Zn^{II} atom. O—H···O and O—H···N hydrogen bonds involving the coordinated water molecules, tetrazolate N atoms and the carboxylate group result in a three-dimensional structure.

Related literature

For background, see: Li *et al.* (2005); Sun *et al.* (2009).



Experimental

Crystal data

$[Zn_2(C_7H_3N_5O_2)_2(H_2O)_6]$
M_r = 617.12
Monoclinic, $P2_1/c$
a = 12.751 (5) Å
b = 12.685 (4) Å
c = 6.992 (3) Å
β = 104.914 (4)°
V = 1092.9 (7) Å^3
Z = 2
Mo $K\alpha$ radiation
μ = 2.27 mm^{-1}
T = 295 K
0.12 × 0.08 × 0.08 mm

Data collection

Rigaku Mercury CCD diffractometer
Absorption correction: multi-scan (*CrystalClear*; Rigaku, 2000)
T_{min} = 0.880, T_{max} = 1.000
(expected range = 0.734–0.834)
8378 measured reflections
2502 independent reflections
2146 reflections with $I > 2\sigma(I)$
R_{int} = 0.031

Refinement

$R[F^2 > 2\sigma(F^2)]$ = 0.032
$wR(F^2)$ = 0.064
S = 1.10
2502 reflections
187 parameters
6 restraints
H atoms treated by a mixture of independent and constrained refinement
$\Delta\rho_{max}$ = 0.44 e Å^{-3}
$\Delta\rho_{min}$ = −0.31 e Å^{-3}

Table 1
Selected geometric parameters (Å, °).

Zn1—O3	2.0547 (18)	Zn1—N5^{i}	2.1333 (19)
Zn1—O5	2.0587 (19)	Zn1—N2	2.1470 (18)
Zn1—O4	2.1317 (18)	Zn1—N1	2.2114 (19)
O3—Zn1—O5	92.12 (8)	O5—Zn1—O4	177.71 (8)
O3—Zn1—O4	85.92 (7)		

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

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

D—H···A	D—H	H···A	D···A	D—H···A
O4—H4B···O2^{ii}	0.846 (10)	1.942 (11)	2.782 (3)	172 (3)
O4—H4A···N3^{iii}	0.841 (10)	2.110 (11)	2.940 (3)	169 (3)
O3—H3B···O1^{ii}	0.849 (10)	1.875 (12)	2.712 (3)	169 (3)
O3—H3A···O1^{iv}	0.846 (10)	1.880 (11)	2.719 (2)	171 (3)
O5—H5B···O1^{v}	0.844 (10)	1.922 (13)	2.740 (3)	163 (3)
O5—H5A···N4^{vi}	0.841 (10)	1.960 (10)	2.800 (3)	177 (3)

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

Data collection: *CrystalClear* (Rigaku, 2000); cell refinement: *CrystalClear*; data reduction: *CrystalClear*; 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: *SHELXL97*.

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Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: NG2558).

References

Barbour, L. J. (2001). *J. Supramol. Chem.* **1**, 189–191.
Li, J.-T., Tao, J., Huang, R.-B. & Zhang, L.-S. (2005). *Acta Cryst.* E**61**, m984–m985.
Rigaku (2000). *CrystalClear*. Rigaku Corporation, Tokyo, Japan.
Sheldrick, G. M. (2008). *Acta Cryst.* A**64**, 112–122.
Sun, Z.-H., Meng, L.-B. & Lin, H. (2009). *Acta Cryst.* E**65**, m280.

supporting information

Acta Cryst. (2009). E**65**, m425 [doi:10.1107/S1600536809009660]

Bis[μ-5-(5-carboxylato-3-pyridyl)tetrazolato-$\kappa^3N^1,N^5:N^2$]bis[triaquazinc(II)]

Haoyong Yin, Ling Wang and Qiulin Nie

S1. Comment

Metal complexes based on tetrazol ligands have attracted great interests (Li *et al.* 2005; Sun *et al.* 2009). In the contribution, we report the title binuclear complex (I) based on tetrazol ligand obtained by *in situ* ligand synthesis.

In the structure of (I), 3-carboxylatopyridyl-6-tetrazolato ligand chelates Zn^{II} center through one pyridyl N and one tetrazolato N and another bridging tetrazolato N atom results in a centrosymmetrical binuclear unit. Three coordinated water molecules complete the distorted octahedral geometry of Zn^{II} center (Fig.1). There exist various hydrogen-bonding interactions between coordinated water molecules and tetrazol N, carboxylate group of the ligand (Table. 2). The hydrogen bonds connect binuclear complex into a three-dimensional structure (Fig.2).

S2. Experimental

A mixture of $Zn(NO_3)_2.6H_2O$ (149 mg, 0.5 mmol), sodium azide(33 mg, 0.5 mmol) and 6-cyanopyridine-3-carboxylic acid (74 mg, 0.5 mmol) was suspended in water (10 ml) and heated in a teflon-lined steel bomb at 160 ° C for 3 days. The colorless crystals were obtained.

S3. Refinement

H atoms bonded to C were located geometrically (C—H = 0.95 Å) with U_{iso}(H) = 1.2 U_{eq}(C). H atoms bonded to O were located by difference maps and refined with a distance restraint of O—H = 0.85 (1) Å. The displacement factors were freely refined.

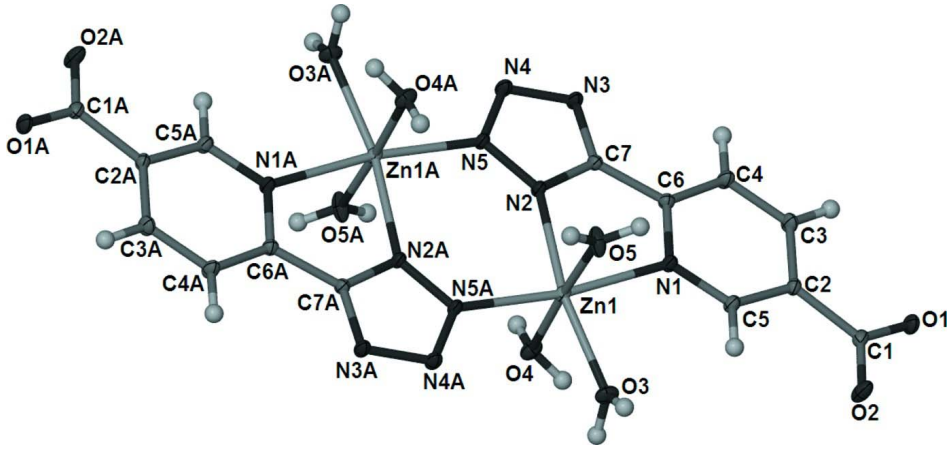

Figure 1

ORTEP of complex (I) with 30% thermal ellipsoids. [Symmetry code: (A) -*x*, 1-*y*, -*z*.]

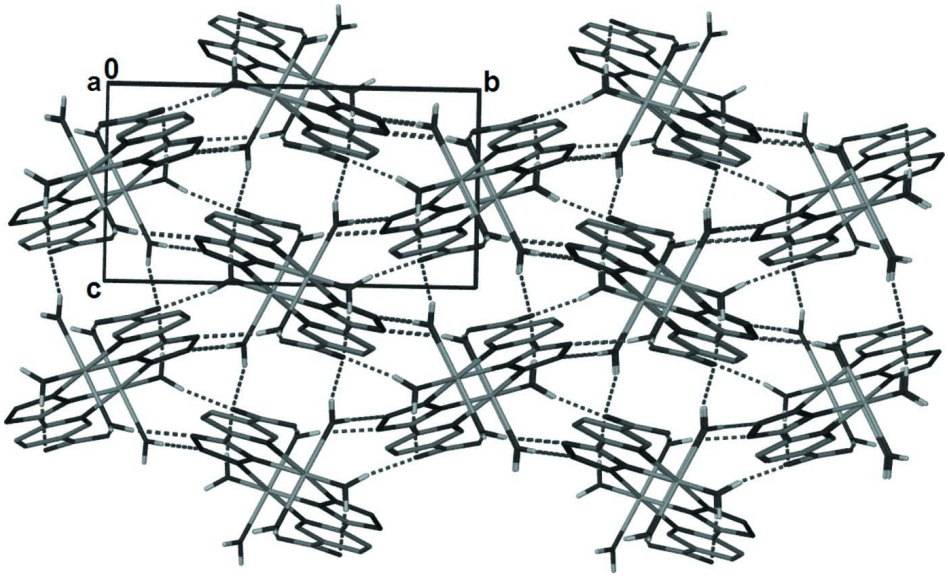

Figure 2

The packing structure viewed along *a* axis.

Bis[μ-5-(5-carboxylato-3-pyridyl)tetrazolato- $\kappa^3N^1,N^5:N^2$]bis[triaquazinc(II)]

Crystal data

$[Zn_2(C_7H_3N_5O_2)_2(H_2O)_6]$
$M_r = 617.12$
Monoclinic, $P2_1/c$
Hall symbol: -P 2ybc
$a = 12.751$ (5) Å
$b = 12.685$ (4) Å
$c = 6.992$ (3) Å
$\beta = 104.914$ (4)°
$V = 1092.9$ (7) Å^3
$Z = 2$
$F(000) = 624$
$D_x = 1.875$ Mg m^{-3}
Mo $K\alpha$ radiation, $\lambda = 0.71073$ Å
Cell parameters from 2535 reflections
$\theta = 3.2$–$27.5°$
$\mu = 2.27$ mm^{-1}
$T = 295$ K
Prism, colorless
0.12 × 0.08 × 0.08 mm

Data collection

Rigaku Mercury CCD diffractometer
Radiation source: fine-focus sealed tube
Graphite monochromator
Detector resolution: 14.6306 pixels mm^{-1}
CCD_Profile_fitting scans
Absorption correction: multi-scan (*CrystalClear*; Rigaku, 2000)
$T_{min} = 0.880$, $T_{max} = 1.000$
8378 measured reflections
2502 independent reflections
2146 reflections with $I > 2\sigma(I)$
$R_{int} = 0.031$
$\theta_{max} = 27.5°$, $\theta_{min} = 2.3°$
$h = -13\rightarrow16$
$k = -16\rightarrow13$
$l = -9\rightarrow8$

Refinement

Refinement on F^2
Least-squares matrix: full
$R[F^2 > 2\sigma(F^2)] = 0.032$
$wR(F^2) = 0.064$
$S = 1.10$
2502 reflections
187 parameters
6 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
$w = 1/[\sigma^2(F_o^2) + (0.0236P)^2 + 0.4632P]$ where $P = (F_o^2 + 2F_c^2)/3$

$(\Delta/\sigma)_{max} = 0.001$
$\Delta\rho_{max} = 0.44$ e Å^{-3}
$\Delta\rho_{min} = -0.31$ 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.

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}
Zn1	0.16114 (2)	0.451195 (18)	0.04261 (4)	0.01891 (9)
N1	0.29843 (15)	0.55236 (13)	0.1954 (3)	0.0199 (4)
O2	0.62383 (14)	0.48266 (13)	0.2838 (3)	0.0345 (4)
O1	0.67185 (13)	0.64838 (12)	0.3616 (2)	0.0273 (4)
N2	0.08352 (15)	0.59050 (14)	0.1144 (3)	0.0184 (4)
C1	0.60194 (19)	0.57469 (18)	0.3163 (3)	0.0219 (5)
C4	0.3458 (2)	0.72742 (18)	0.3169 (4)	0.0258 (5)
H4	0.3239	0.7953	0.3491	0.031*
C6	0.26966 (19)	0.65036 (16)	0.2412 (3)	0.0196 (5)
C3	0.4544 (2)	0.70372 (18)	0.3447 (4)	0.0260 (5)
H3	0.5079	0.7557	0.3958	0.031*
C2	0.48558 (18)	0.60427 (17)	0.2980 (3)	0.0193 (5)
C5	0.40336 (19)	0.53119 (17)	0.2249 (3)	0.0217 (5)
H5	0.4236	0.4624	0.1944	0.026*
O3	0.26729 (15)	0.34205 (13)	−0.0178 (3)	0.0278 (4)
O4	0.16780 (16)	0.52386 (13)	−0.2289 (3)	0.0277 (4)
C7	0.15272 (19)	0.66661 (16)	0.1971 (3)	0.0184 (5)
O5	0.16098 (16)	0.37901 (15)	0.3062 (3)	0.0353 (5)
N5	−0.01525 (15)	0.63182 (14)	0.0916 (3)	0.0195 (4)
N4	−0.00558 (16)	0.72963 (15)	0.1574 (3)	0.0250 (4)
N3	0.09973 (16)	0.75412 (15)	0.2242 (3)	0.0245 (4)
H4A	0.141 (2)	0.5845 (12)	−0.254 (4)	0.041 (9)*
H3A	0.289 (2)	0.2851 (13)	0.042 (4)	0.040 (8)*
H4B	0.2293 (14)	0.527 (2)	−0.254 (5)	0.047 (9)*
H3B	0.279 (2)	0.339 (2)	−0.132 (2)	0.045 (9)*
H5A	0.114 (2)	0.336 (2)	0.320 (5)	0.058 (10)*
H5B	0.2086 (18)	0.383 (2)	0.415 (2)	0.040 (8)*

Atomic displacement parameters (Å^2)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
Zn1	0.01593 (16)	0.01373 (14)	0.02694 (15)	−0.00018 (10)	0.00528 (11)	−0.00126 (10)

N1	0.0160 (10)	0.0159 (9)	0.0274 (10)	−0.0006 (7)	0.0051 (8)	−0.0027 (8)
O2	0.0210 (10)	0.0243 (9)	0.0606 (12)	0.0007 (7)	0.0146 (9)	−0.0065 (8)
O1	0.0170 (9)	0.0271 (9)	0.0372 (10)	−0.0048 (7)	0.0059 (8)	−0.0073 (7)
N2	0.0140 (10)	0.0172 (9)	0.0240 (10)	0.0005 (7)	0.0051 (8)	−0.0017 (7)
C1	0.0166 (13)	0.0276 (13)	0.0222 (12)	−0.0001 (9)	0.0061 (10)	−0.0008 (9)
C4	0.0196 (13)	0.0194 (11)	0.0382 (14)	0.0004 (9)	0.0071 (11)	−0.0072 (10)
C6	0.0188 (13)	0.0170 (11)	0.0228 (11)	0.0007 (9)	0.0051 (10)	−0.0022 (9)
C3	0.0206 (13)	0.0215 (12)	0.0344 (14)	−0.0052 (10)	0.0042 (11)	−0.0072 (10)
C2	0.0146 (12)	0.0207 (11)	0.0222 (12)	−0.0003 (9)	0.0039 (9)	−0.0007 (9)
C5	0.0173 (13)	0.0192 (11)	0.0288 (12)	0.0011 (9)	0.0063 (10)	−0.0016 (9)
O3	0.0332 (11)	0.0185 (9)	0.0347 (10)	0.0079 (7)	0.0141 (9)	0.0029 (8)
O4	0.0258 (11)	0.0238 (9)	0.0367 (10)	0.0053 (7)	0.0139 (9)	0.0075 (8)
C7	0.0168 (12)	0.0167 (10)	0.0218 (11)	−0.0013 (8)	0.0050 (9)	−0.0036 (9)
O5	0.0301 (12)	0.0394 (11)	0.0302 (10)	−0.0183 (9)	−0.0033 (9)	0.0129 (8)
N5	0.0135 (10)	0.0175 (9)	0.0273 (10)	0.0009 (7)	0.0050 (8)	−0.0028 (8)
N4	0.0185 (11)	0.0199 (10)	0.0363 (12)	0.0006 (8)	0.0067 (9)	−0.0077 (8)
N3	0.0170 (11)	0.0202 (10)	0.0353 (11)	0.0011 (8)	0.0048 (9)	−0.0089 (8)

Geometric parameters (Å, º)

Zn1—O3	2.0547 (18)	C6—C7	1.457 (3)
Zn1—O5	2.0587 (19)	C3—C2	1.386 (3)
Zn1—O4	2.1317 (18)	C3—H3	0.9500
Zn1—N5^{i}	2.1333 (19)	C2—C5	1.394 (3)
Zn1—N2	2.1470 (18)	C5—H5	0.9500
Zn1—N1	2.2114 (19)	O3—H3A	0.846 (10)
N1—C5	1.328 (3)	O3—H3B	0.849 (10)
N1—C6	1.358 (3)	O4—H4A	0.841 (10)
O2—C1	1.235 (3)	O4—H4B	0.846 (10)
O1—C1	1.274 (3)	C7—N3	1.338 (3)
N2—C7	1.335 (3)	O5—H5A	0.841 (10)
N2—N5	1.335 (2)	O5—H5B	0.844 (10)
C1—C2	1.504 (3)	N5—N4	1.318 (3)
C4—C3	1.381 (3)	N5—Zn1^{i}	2.1333 (19)
C4—C6	1.384 (3)	N4—N3	1.340 (3)
C4—H4	0.9500		
O3—Zn1—O5	92.12 (8)	N1—C6—C7	113.79 (19)
O3—Zn1—O4	85.92 (7)	C4—C6—C7	124.1 (2)
O5—Zn1—O4	177.71 (8)	C4—C3—C2	120.2 (2)
O3—Zn1—N5^{i}	97.06 (7)	C4—C3—H3	119.9
O5—Zn1—N5^{i}	88.43 (7)	C2—C3—H3	119.9
O4—Zn1—N5^{i}	92.97 (7)	C3—C2—C5	117.2 (2)
O3—Zn1—N2	166.01 (7)	C3—C2—C1	122.9 (2)
O5—Zn1—N2	92.86 (8)	C5—C2—C1	119.8 (2)
O4—Zn1—N2	88.79 (7)	N1—C5—C2	123.7 (2)
N5^{i}—Zn1—N2	96.13 (7)	N1—C5—H5	118.1
O3—Zn1—N1	90.51 (7)	C2—C5—H5	118.1

O5—Zn1—N1	90.49 (7)	Zn1—O3—H3A	128.6 (19)
O4—Zn1—N1	88.35 (7)	Zn1—O3—H3B	121 (2)
N5^{i}—Zn1—N1	172.39 (7)	H3A—O3—H3B	108 (3)
N2—Zn1—N1	76.39 (7)	Zn1—O4—H4A	118.2 (19)
C5—N1—C6	118.12 (19)	Zn1—O4—H4B	117 (2)
C5—N1—Zn1	126.76 (14)	H4A—O4—H4B	105 (3)
C6—N1—Zn1	114.64 (15)	N2—C7—N3	111.1 (2)
C7—N2—N5	105.42 (17)	N2—C7—C6	121.04 (19)
C7—N2—Zn1	113.79 (15)	N3—C7—C6	127.8 (2)
N5—N2—Zn1	140.58 (14)	Zn1—O5—H5A	124 (2)
O2—C1—O1	124.2 (2)	Zn1—O5—H5B	128 (2)
O2—C1—C2	119.0 (2)	H5A—O5—H5B	108 (3)
O1—C1—C2	116.8 (2)	N4—N5—N2	109.10 (17)
C3—C4—C6	118.7 (2)	N4—N5—Zn1^{i}	127.65 (14)
C3—C4—H4	120.7	N2—N5—Zn1^{i}	123.21 (13)
C6—C4—H4	120.7	N5—N4—N3	109.59 (17)
N1—C6—C4	122.1 (2)	C7—N3—N4	104.80 (18)

Symmetry code: (i) −*x*, −*y*+1, −*z*.

Hydrogen-bond geometry (Å, º)

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
O4—H4*B*···O2^{ii}	0.85 (1)	1.94 (1)	2.782 (3)	172 (3)
O4—H4*A*···N3^{iii}	0.84 (1)	2.11 (1)	2.940 (3)	169 (3)
O3—H3*B*···O1^{ii}	0.85 (1)	1.88 (1)	2.712 (3)	169 (3)
O3—H3*A*···O1^{iv}	0.85 (1)	1.88 (1)	2.719 (2)	171 (3)
O5—H5*B*···O1^{v}	0.84 (1)	1.92 (1)	2.740 (3)	163 (3)
O5—H5*A*···N4^{vi}	0.84 (1)	1.96 (1)	2.800 (3)	177 (3)

Symmetry codes: (ii) −*x*+1, −*y*+1, −*z*; (iii) *x*, −*y*+3/2, *z*−1/2; (iv) −*x*+1, *y*−1/2, −*z*+1/2; (v) −*x*+1, −*y*+1, −*z*+1; (vi) −*x*, *y*−1/2, −*z*+1/2.