

Poly[bis(μ_2 -5-*n*-butyltetrazolato- $\kappa^2N^1:N^4$)zinc(II)]

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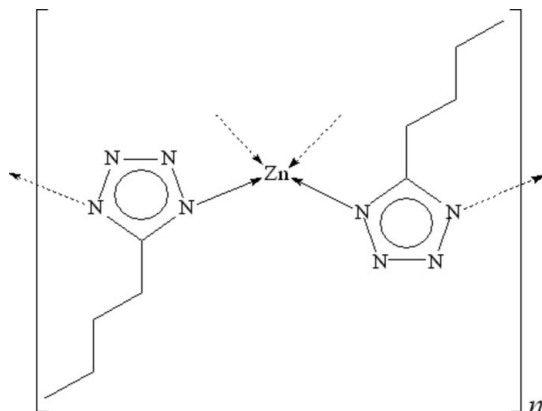
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 Key indicators: single-crystal X-ray study; $T = 113$ K; mean $\sigma(\text{C}-\text{C}) = 0.003$ Å; R factor = 0.022; wR factor = 0.050; data-to-parameter ratio = 19.3.

In the title complex, $[\text{Zn}(\text{C}_5\text{H}_9\text{N}_4)_2]_n$, the Zn^{II} center is coordinated by four N atoms of different tetrazolate ligands with a slightly distorted tetrahedral geometry [Zn–N distances and N–Zn–N angles are in the ranges 1.991 (2)–2.007 (2) Å and 104.22 (8)–116.13 (8)°, respectively]. Each ligand links two Zn^{II} atoms through its 1- and 4-position tetrazole N atoms, forming a single, fully connected three-dimensional framework with a diamond-like topology. In the crystal structure, the $\text{Zn} \cdots \text{Zn}$ separations across each tetrazole unit are 6.115 (2) and 6.134 (2) Å and the $\text{Zn} \cdots \text{Zn} \cdots \text{Zn}$ angles are in the range 107.77 (8)–116.83 (8)°.

Related literature

For related literature, see: Li *et al.* (2007) and references therein; Wang *et al.* (2005); Ye *et al.* (2005).



Experimental

Crystal data

$[\text{Zn}(\text{C}_5\text{H}_9\text{N}_4)_2]$	$V = 1399.4 (5) \text{ \AA}^3$
$M_r = 315.69$	$Z = 4$
Orthorhombic, $P2_12_12_1$	Mo $K\alpha$ radiation
$a = 9.6534 (19) \text{ \AA}$	$\mu = 1.76 \text{ mm}^{-1}$
$b = 10.096 (2) \text{ \AA}$	$T = 113 (2) \text{ K}$
$c = 14.359 (3) \text{ \AA}$	$0.24 \times 0.22 \times 0.22 \text{ mm}$

Data collection

Bruker SMART 1000 CCD area-detector diffractometer	17371 measured reflections
Absorption correction: multi-scan (SADABS; Bruker, 1998)	3325 independent reflections
$T_{\text{min}} = 0.978$, $T_{\text{max}} = 1.000$	3068 reflections with $I > 2\sigma(I)$
(expected range = 0.665–0.680)	$R_{\text{int}} = 0.032$

Refinement

$R[F^2 > 2\sigma(F^2)] = 0.022$	$\Delta\rho_{\text{max}} = 0.50 \text{ e \AA}^{-3}$
$wR(F^2) = 0.050$	$\Delta\rho_{\text{min}} = -0.35 \text{ e \AA}^{-3}$
$S = 1.02$	Absolute structure: Flack (1983),
3325 reflections	1405 Friedel pairs
172 parameters	Flack parameter: $-0.012 (9)$
H-atom parameters constrained	

Data collection: SMART (Bruker, 1998); cell refinement: SAINT (Bruker, 1998); data reduction: SHELXTL (Bruker, 1998); program(s) used to solve structure: SHELXS97 (Sheldrick, 1997); program(s) used to refine structure: SHELXL97 (Sheldrick, 1997); molecular graphics: SHELXTL; software used to prepare material for publication: SHELXTL.

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Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: SF2013).

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supplementary materials

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Poly[bis(μ_2 -5-*n*-butyltetrazolato- $\kappa^2 N^1:N^4$)zinc(II)]

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Comment

As the analogue of carboxylic acid, it has been realised that 5-substituted tetrazolate-based organic ligands have great potential in generating coordination polymers with novel network topologies and interesting properties [Li *et al.*, 2007 and references therein]. Recently, three Zn^{II} tetrazolate-based complexes having similar diamondoid structure to the title complex, [Zn(C₅H₉N₄)₂]_n: *catena*-[bis(μ_2 -5-phenyltetrazolato-N,N'')-zinc(II)], *catena*-[bis(μ_2 -5-(4'-amino-6'-pyridyl)tetrazolato-N,N'')-zinc(II)] (Ye *et al.*, 2005) and *catena*-[bis(μ_2 tetrazolato-N,N'')-zinc(II)] (Wang *et al.*, 2005) were reported.

As shown in Fig. 1, in the title complex the Zn^{II} center locates at the crystallographically general position and is coordinated by four N atoms of distinct tetrazolate ligands. The coordination geometry is of slightly distorted tetrahedron with Zn—N distances and N—Zn—N angles being in the range of 1.991 (2)–2.007 (2) Å and 104.22 (8)–116.13 (8) °, respectively. Each ligand links two Zn^{II} atoms through its 1- and 4-position tetrazole N atoms to form a single three-dimensional diamond-like framework (Fig. 2). In the structure, the Zn—Zn separations across each tetrazole moiety are 6.115 (2) and 6.134 (2) Å and the Zn—Zn—Zn angles are revealed to range from 107.77 (8) to 116.83 (8) °.

Experimental

A mixture of ZnCl₂ (27 mg, 0.2 mmol), NaN₃ (33 mg, 0.5 mmol) and valeronitrile (33 mg, 0.4 mmol) in 10 ml of water was sealed in a Teflon-lined stainless-steel Parr bomb that was heated at 373 K for 48 h. Colorless crystals of the title compound were collected after the bomb was allowed to cool to room temperature spontaneously. Yield, 30% with respect to Zn^{II}. *Caution:* Metal azides may be explosive. Although we have met no problems in this work, only a small amount of them should be prepared and handled with great caution.

Refinement

H atoms were included in calculated positions and treated in the subsequent refinement as riding atoms, with C—H = 0.97 and 0.96 Å and $U_{\text{iso}}(\text{H}) = 1.2 U_{\text{eq}}(\text{C}, \text{N})$.

Figures

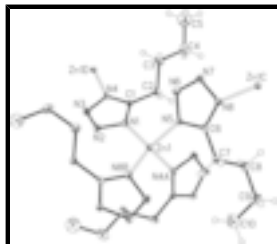


Fig. 1. View of coordination environments of Zn^{II} and ligand in the title complex with 40% displacement probability. [Herein, labelled atoms A—D correspond to symmetry operations i—iv, respectively. (i) = $1/2 + x, -3/2 - y, -z$; (ii) = $-x, 1/2 + y, -1/2 - z$; (iii) = $-x, -1/2 + y, -1/2 - z$ and (iv) = $-1/2 + x, -3/2 - y, -z$]

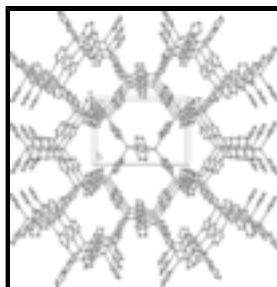


Fig. 2. three-dimensional structure of the title compound with butyl groups omitted for clarity.

Poly[bis(μ_2 -5-*n*-butyltetrazolato- $\kappa^2\text{N}^1:\text{N}^4$)zinc(II)]

Crystal data

$[\text{Zn}(\text{C}_5\text{H}_9\text{N}_4)_2]$

$M_r = 315.69$

Orthorhombic, $P2_12_12_1$

Hall symbol: P 2ac 2ab

$a = 9.6534$ (19) Å

$b = 10.096$ (2) Å

$c = 14.359$ (3) Å

$V = 1399.4$ (5) Å³

$Z = 4$

$F_{000} = 656$

$D_x = 1.498$ Mg m⁻³

Mo $K\alpha$ radiation

$\lambda = 0.71073$ Å

Cell parameters from 5045 reflections

$\theta = 2.8$ – 27.9°

$\mu = 1.76$ mm⁻¹

$T = 113$ (2) K

Block, colorless

$0.24 \times 0.22 \times 0.22$ mm

Data collection

Bruker Smart 1000 CCD area-detector diffractometer

Radiation source: fine-focus sealed tube

Monochromator: graphite

$T = 113$ (2) K

ϕ and ω scan

Absorption correction: multi-scan

(SADABS; Bruker, 1998)

$T_{\text{min}} = 0.978$, $T_{\text{max}} = 1.000$

17371 measured reflections

3325 independent reflections

3068 reflections with $I > 2\sigma(I)$

$R_{\text{int}} = 0.032$

$\theta_{\text{max}} = 27.9^\circ$

$\theta_{\text{min}} = 2.8^\circ$

$h = -11 \rightarrow 12$

$k = -13 \rightarrow 13$

$l = -18 \rightarrow 18$

Refinement

Refinement on F^2	Hydrogen site location: inferred from neighbouring sites
Least-squares matrix: full	H-atom parameters constrained
$R[F^2 > 2\sigma(F^2)] = 0.022$	$w = 1/[\sigma^2(F_o^2) + (0.0274P)^2]$
$wR(F^2) = 0.050$	where $P = (F_o^2 + 2F_c^2)/3$
$S = 1.02$	$(\Delta/\sigma)_{\max} = 0.001$
3325 reflections	$\Delta\rho_{\max} = 0.50 \text{ e } \text{\AA}^{-3}$
172 parameters	$\Delta\rho_{\min} = -0.35 \text{ e } \text{\AA}^{-3}$
Primary atom site location: structure-invariant direct methods	Extinction correction: none
Secondary atom site location: difference Fourier map	Absolute structure: Flack (1983), 1405 Friedel pairs
	Flack parameter: $-0.012 (9)$

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 (\AA^2)

	x	y	z	$U_{\text{iso}}^*/U_{\text{eq}}$
Zn1	-0.029407 (19)	-0.736890 (17)	-0.131493 (12)	0.01072 (6)
C1	-0.26406 (18)	-0.79890 (17)	0.00726 (12)	0.0128 (4)
C2	-0.2305 (2)	-0.94018 (19)	0.02652 (15)	0.0211 (4)
H2A	-0.2072	-0.9498	0.0919	0.025*
H2B	-0.1497	-0.9653	-0.0096	0.025*
C3	-0.3495 (2)	-1.03381 (19)	0.00312 (17)	0.0294 (5)
H3A	-0.3606	-1.0382	-0.0639	0.035*
H3B	-0.4347	-0.9993	0.0294	0.035*
C4	-0.3233 (3)	-1.1747 (2)	0.0416 (2)	0.0495 (7)
H4A	-0.2441	-1.2133	0.0101	0.059*
H4B	-0.3018	-1.1693	0.1075	0.059*
C5	-0.4459 (3)	-1.2612 (2)	0.0281 (2)	0.0636 (8)
H5A	-0.4274	-1.3472	0.0538	0.085*
H5B	-0.4652	-1.2695	-0.0373	0.085*
H5C	-0.5245	-1.2230	0.0590	0.085*
C6	0.04569 (18)	-0.97174 (16)	-0.25457 (11)	0.0131 (4)
C7	0.19069 (19)	-0.92793 (18)	-0.27100 (14)	0.0168 (4)

supplementary materials

H7A	0.2021	-0.8392	-0.2463	0.020*
H7B	0.2073	-0.9240	-0.3376	0.020*
C8	0.29893 (19)	-1.01919 (19)	-0.22630 (15)	0.0226 (4)
H8A	0.2883	-1.1079	-0.2512	0.027*
H8B	0.2828	-1.0232	-0.1597	0.027*
C9	0.4475 (2)	-0.9711 (2)	-0.24428 (16)	0.0313 (5)
H9A	0.5121	-1.0369	-0.2210	0.038*
H9B	0.4619	-0.9629	-0.3109	0.038*
C10	0.4783 (2)	-0.8388 (2)	-0.1982 (2)	0.0468 (7)
H10A	0.5718	-0.8128	-0.2120	0.070*
H10B	0.4673	-0.8470	-0.1320	0.070*
H10C	0.4154	-0.7729	-0.2214	0.070*
N1	-0.19539 (15)	-0.71806 (14)	-0.05010 (10)	0.0135 (3)
N2	-0.25827 (17)	-0.59748 (15)	-0.04447 (12)	0.0188 (4)
N3	-0.36058 (17)	-0.60558 (15)	0.01261 (11)	0.0177 (3)
N4	-0.36690 (14)	-0.73163 (15)	0.04700 (10)	0.0137 (3)
N5	-0.04611 (16)	-0.90468 (13)	-0.20430 (10)	0.0131 (3)
N6	-0.16579 (16)	-0.97651 (15)	-0.20460 (11)	0.0162 (3)
N7	-0.14673 (16)	-1.08254 (16)	-0.25271 (11)	0.0162 (3)
N8	-0.01317 (16)	-1.08258 (14)	-0.28558 (10)	0.0136 (3)

Atomic displacement parameters (\AA^2)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
Zn1	0.01061 (9)	0.01059 (9)	0.01096 (9)	-0.00005 (7)	0.00044 (8)	0.00010 (7)
C1	0.0115 (8)	0.0141 (9)	0.0126 (8)	0.0008 (6)	-0.0007 (7)	-0.0011 (7)
C2	0.0235 (11)	0.0146 (10)	0.0250 (10)	0.0049 (8)	0.0080 (9)	0.0031 (8)
C3	0.0306 (12)	0.0158 (11)	0.0418 (14)	-0.0035 (9)	0.0143 (10)	-0.0064 (9)
C4	0.0491 (16)	0.0252 (13)	0.074 (2)	0.0053 (12)	0.0047 (16)	-0.0033 (13)
C5	0.066 (2)	0.0236 (13)	0.071 (2)	-0.0005 (14)	0.0079 (17)	0.0005 (14)
C6	0.0151 (9)	0.0116 (8)	0.0126 (8)	0.0030 (7)	-0.0003 (8)	0.0003 (6)
C7	0.0139 (9)	0.0140 (9)	0.0223 (10)	-0.0017 (7)	0.0028 (8)	-0.0055 (8)
C8	0.0185 (10)	0.0204 (10)	0.0288 (11)	0.0016 (8)	-0.0013 (9)	-0.0045 (8)
C9	0.0156 (11)	0.0356 (12)	0.0425 (13)	0.0034 (9)	-0.0011 (10)	-0.0062 (10)
C10	0.0216 (12)	0.0350 (13)	0.074 (2)	-0.0046 (11)	-0.0066 (15)	-0.0078 (13)
N1	0.0142 (7)	0.0127 (8)	0.0136 (7)	0.0004 (6)	0.0014 (6)	-0.0001 (6)
N2	0.0200 (8)	0.0135 (8)	0.0229 (9)	0.0030 (6)	0.0073 (7)	0.0021 (7)
N3	0.0213 (9)	0.0119 (8)	0.0200 (8)	0.0004 (6)	0.0064 (7)	0.0032 (6)
N4	0.0166 (7)	0.0105 (7)	0.0140 (7)	0.0007 (6)	0.0007 (6)	0.0014 (6)
N5	0.0123 (8)	0.0124 (7)	0.0146 (7)	-0.0003 (6)	0.0014 (6)	-0.0006 (6)
N6	0.0132 (8)	0.0161 (8)	0.0195 (8)	-0.0045 (6)	0.0017 (7)	-0.0047 (7)
N7	0.0129 (8)	0.0194 (8)	0.0162 (8)	-0.0011 (6)	0.0027 (7)	-0.0027 (6)
N8	0.0124 (8)	0.0141 (7)	0.0143 (7)	-0.0016 (6)	0.0015 (6)	-0.0024 (6)

Geometric parameters (\AA , $^\circ$)

Zn1—N1	1.9923 (15)	C6—C7	1.487 (2)
Zn1—N5	1.9971 (14)	C7—C8	1.534 (3)

Zn1—N8 ⁱ	2.0035 (14)	C7—H7A	0.9700
Zn1—N4 ⁱⁱ	2.0085 (14)	C7—H7B	0.9700
C1—N4	1.331 (2)	C8—C9	1.536 (3)
C1—N1	1.336 (2)	C8—H8A	0.9700
C1—C2	1.489 (3)	C8—H8B	0.9700
C2—C3	1.525 (3)	C9—C10	1.521 (3)
C2—H2A	0.9700	C9—H9A	0.9700
C2—H2B	0.9700	C9—H9B	0.9700
C3—C4	1.547 (3)	C10—H10A	0.9600
C3—H3A	0.9700	C10—H10B	0.9600
C3—H3B	0.9700	C10—H10C	0.9600
C4—C5	1.484 (3)	N1—N2	1.363 (2)
C4—H4A	0.9700	N2—N3	1.286 (2)
C4—H4B	0.9700	N3—N4	1.366 (2)
C5—H5A	0.9600	N4—Zn1 ⁱⁱⁱ	2.0085 (14)
C5—H5B	0.9600	N5—N6	1.364 (2)
C5—H5C	0.9600	N6—N7	1.287 (2)
C6—N5	1.329 (2)	N7—N8	1.373 (2)
C6—N8	1.332 (2)	N8—Zn1 ^{iv}	2.0035 (14)
N1—Zn1—N5	108.85 (6)	C8—C7—H7A	108.9
N1—Zn1—N8 ⁱ	116.07 (6)	C6—C7—H7B	108.9
N5—Zn1—N8 ⁱ	111.41 (6)	C8—C7—H7B	108.9
N1—Zn1—N4 ⁱⁱ	106.79 (6)	H7A—C7—H7B	107.7
N5—Zn1—N4 ⁱⁱ	104.18 (6)	C7—C8—C9	112.09 (17)
N8 ⁱ —Zn1—N4 ⁱⁱ	108.77 (6)	C7—C8—H8A	109.2
N4—C1—N1	108.82 (16)	C9—C8—H8A	109.2
N4—C1—C2	124.86 (16)	C7—C8—H8B	109.2
N1—C1—C2	126.30 (16)	C9—C8—H8B	109.2
C1—C2—C3	112.90 (17)	H8A—C8—H8B	107.9
C1—C2—H2A	109.0	C10—C9—C8	112.78 (18)
C3—C2—H2A	109.0	C10—C9—H9A	109.0
C1—C2—H2B	109.0	C8—C9—H9A	109.0
C3—C2—H2B	109.0	C10—C9—H9B	109.0
H2A—C2—H2B	107.8	C8—C9—H9B	109.0
C2—C3—C4	111.6 (2)	H9A—C9—H9B	107.8
C2—C3—H3A	109.3	C9—C10—H10A	109.5
C4—C3—H3A	109.3	C9—C10—H10B	109.5
C2—C3—H3B	109.3	H10A—C10—H10B	109.5
C4—C3—H3B	109.3	C9—C10—H10C	109.5
H3A—C3—H3B	108.0	H10A—C10—H10C	109.5
C5—C4—C3	111.4 (2)	H10B—C10—H10C	109.5
C5—C4—H4A	109.4	C1—N1—N2	106.75 (14)
C3—C4—H4A	109.4	C1—N1—Zn1	134.64 (12)
C5—C4—H4B	109.4	N2—N1—Zn1	118.57 (11)
C3—C4—H4B	109.4	N3—N2—N1	108.85 (14)
H4A—C4—H4B	108.0	N2—N3—N4	108.89 (14)
C4—C5—H5A	109.5	C1—N4—N3	106.68 (14)

supplementary materials

C4—C5—H5B	109.5	C1—N4—Zn1 ⁱⁱⁱ	139.52 (13)
H5A—C5—H5B	109.5	N3—N4—Zn1 ⁱⁱⁱ	113.61 (11)
C4—C5—H5C	109.5	C6—N5—N6	106.99 (14)
H5A—C5—H5C	109.5	C6—N5—Zn1	131.52 (12)
H5B—C5—H5C	109.5	N6—N5—Zn1	121.40 (11)
N5—C6—N8	108.99 (15)	N7—N6—N5	108.83 (14)
N5—C6—C7	124.24 (15)	N6—N7—N8	108.60 (14)
N8—C6—C7	126.77 (16)	C6—N8—N7	106.59 (14)
C6—C7—C8	113.35 (16)	C6—N8—Zn1 ^{iv}	139.87 (13)
C6—C7—H7A	108.9	N7—N8—Zn1 ^{iv}	113.40 (11)
N4—C1—C2—C3	60.4 (3)	N1—C1—N4—Zn1 ⁱⁱⁱ	174.28 (14)
N1—C1—C2—C3	-121.4 (2)	C2—C1—N4—Zn1 ⁱⁱⁱ	-7.2 (3)
C1—C2—C3—C4	-168.7 (2)	N2—N3—N4—C1	-0.44 (19)
C2—C3—C4—C5	173.6 (2)	N2—N3—N4—Zn1 ⁱⁱⁱ	-176.38 (12)
N5—C6—C7—C8	114.1 (2)	N8—C6—N5—N6	-0.20 (19)
N8—C6—C7—C8	-64.9 (2)	C7—C6—N5—N6	-179.41 (16)
C6—C7—C8—C9	-179.85 (16)	N8—C6—N5—Zn1	176.40 (12)
C7—C8—C9—C10	65.2 (2)	C7—C6—N5—Zn1	-2.8 (3)
N4—C1—N1—N2	0.4 (2)	N1—Zn1—N5—C6	-166.75 (15)
C2—C1—N1—N2	-178.09 (17)	N8 ⁱ —Zn1—N5—C6	64.01 (17)
N4—C1—N1—Zn1	177.93 (12)	N4 ⁱⁱ —Zn1—N5—C6	-53.10 (16)
C2—C1—N1—Zn1	-0.5 (3)	N1—Zn1—N5—N6	9.43 (14)
N5—Zn1—N1—C1	46.63 (19)	N8 ⁱ —Zn1—N5—N6	-119.81 (13)
N8 ⁱ —Zn1—N1—C1	173.24 (16)	N4 ⁱⁱ —Zn1—N5—N6	123.08 (13)
N4 ⁱⁱ —Zn1—N1—C1	-65.30 (18)	C6—N5—N6—N7	0.2 (2)
N5—Zn1—N1—N2	-136.05 (13)	Zn1—N5—N6—N7	-176.78 (11)
N8 ⁱ —Zn1—N1—N2	-9.44 (15)	N5—N6—N7—N8	-0.18 (19)
N4 ⁱⁱ —Zn1—N1—N2	112.02 (13)	N5—C6—N8—N7	0.09 (18)
C1—N1—N2—N3	-0.7 (2)	C7—C6—N8—N7	179.28 (17)
Zn1—N1—N2—N3	-178.68 (12)	N5—C6—N8—Zn1 ^{iv}	175.30 (14)
N1—N2—N3—N4	0.7 (2)	C7—C6—N8—Zn1 ^{iv}	-5.5 (3)
N1—C1—N4—N3	0.01 (19)	N6—N7—N8—C6	0.06 (18)
C2—C1—N4—N3	178.52 (17)	N6—N7—N8—Zn1 ^{iv}	-176.58 (12)

Symmetry codes: (i) $-x, y+1/2, -z-1/2$; (ii) $x+1/2, -y-3/2, -z$; (iii) $x-1/2, -y-3/2, -z$; (iv) $-x, y-1/2, -z-1/2$.

Fig. 1

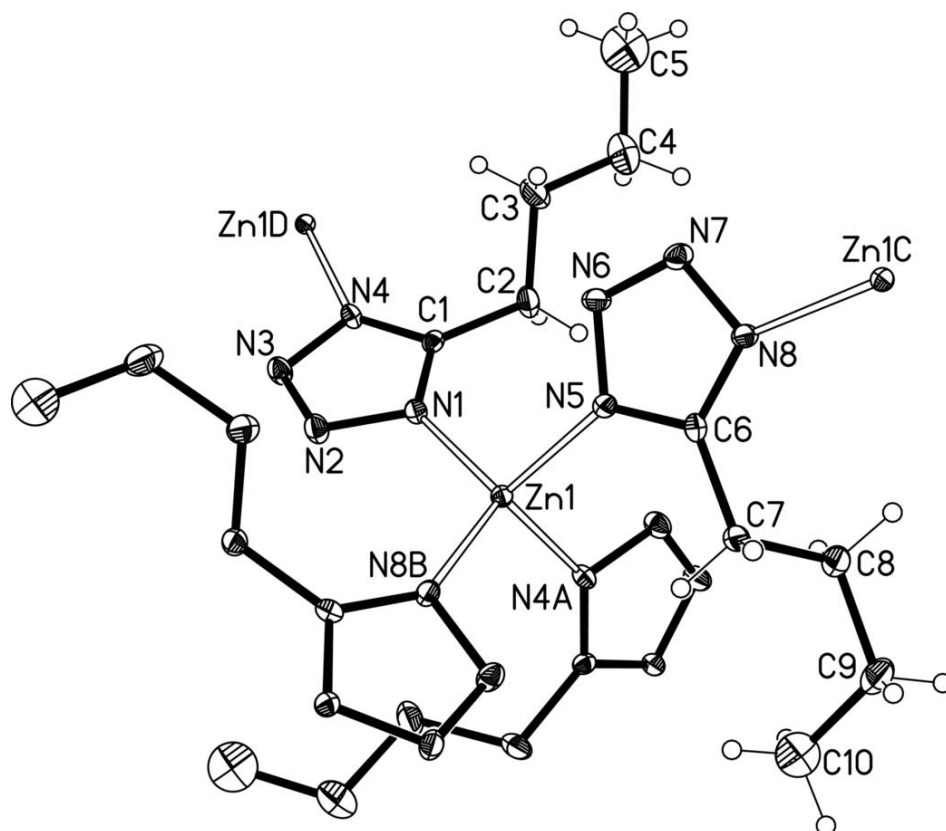


Fig. 2

