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Dichlorido{2-[3-(2-pyridyl)pyrazol-1-ylmethyl]pyridine}zinc(II)

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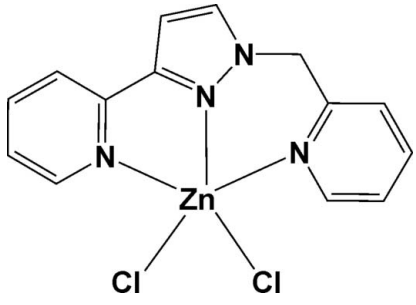
Key indicators: single-crystal X-ray study; T = 294 K; mean σ(C–C) = 0.004 Å; R factor = 0.032; wR factor = 0.073; data-to-parameter ratio = 16.0.

In the title complex, $[ZnCl_2(C_{14}H_{12}N_4)]$, Zn^{II} is pentacoordinated by three N-atom donors from one 2-[3-(2-pyridyl)pyrazol-1-ylmethyl]pyridine (L) ligand and by two Cl^- anions in a distorted trigonal–bipyramidal geometry. Adjacent mononuclear Zn^{II} complexes form π–π stacking interactions between pyridine and pyrazole rings of neighbouring L ligands, with centroid–centroid separations of 3.671 (1) and 3.681 (1) Å, generating one-dimensional chains.

Related literature

For related literature, see: Bell *et al.* (2003); Janiak (2000); Khlobystov *et al.* (2001); Paul *et al.* (2004); Singh *et al.* (2003); Song *et al.* (2005); Steel (2005); Ward *et al.* (2001); Zou *et al.* (2004).



Experimental

Crystal data

$[ZnCl_2(C_{14}H_{12}N_4)]$
M_r = 372.55
Monoclinic, $P2_1/n$
a = 7.2813 (10) Å
b = 15.248 (2) Å
c = 13.5034 (19) Å
β = 90.967 (2)°
V = 1499.0 (4) Å^3
Z = 4
Mo $K\alpha$ radiation
μ = 1.99 mm^{-1}
T = 294 (2) K
0.20 × 0.18 × 0.14 mm

Data collection

Bruker SMART CCD diffractometer
Absorption correction: multi-scan (*SADABS*; Bruker, 1998)
T_{min} = 0.692, T_{max} = 0.768
8317 measured reflections
3037 independent reflections
2174 reflections with $I > 2\sigma(I)$
R_{int} = 0.040

Refinement

$R[F^2 > 2\sigma(F^2)]$ = 0.033
$wR(F^2)$ = 0.073
S = 1.01
3037 reflections
190 parameters
H-atom parameters constrained
$\Delta\rho_{max}$ = 0.27 e Å^{-3}
$\Delta\rho_{min}$ = −0.35 e Å^{-3}

Table 1
Selected geometric parameters (Å, °).

Zn1—Cl1	2.2596 (9)	Zn1—N3	2.304 (2)
Zn1—Cl2	2.2640 (8)	Zn1—N4	2.302 (2)
Zn1—N2	2.051 (2)		
Cl1—Zn1—Cl2	111.34 (3)	Cl2—Zn1—N3	89.36 (6)
Cl1—Zn1—N2	104.04 (7)	Cl2—Zn1—N4	95.75 (6)
Cl1—Zn1—N3	109.88 (6)	N2—Zn1—N3	72.73 (8)
Cl1—Zn1—N4	102.14 (7)	N2—Zn1—N4	82.34 (8)
Cl2—Zn1—N2	144.09 (7)	N3—Zn1—N4	143.13 (8)

Data collection: *SMART* (Bruker, 1998); cell refinement: *SAINT* (Bruker, 1998); data reduction: *SAINT*; program(s) used to solve structure: *SHELXS97* (Sheldrick, 1997); program(s) used to refine structure: *SHELXL97* (Sheldrick, 1997); molecular graphics: *SHELXTL* (Bruker, 1998); software used to prepare material for publication: *SHELXTL* and *PLATON* (Spek, 2003).

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Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: BI2215).

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supplementary materials

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Dichlorido{2-[3-(2-pyridyl)pyrazol-1-ylmethyl]pyridine}zinc(II)

C.-S. Liu and J.-N. Zhou

Comment

Nowadays, much attention is focused on the synthetic approach and the structural control of metal-organic coordination architectures with such ligands based on pyrazolyl-pyridine chelating units (Steel, 2005; Ward *et al.*, 2001). In this field, Ward *et al.* have reported many novel functional complexes through the use of 3-(2-pyridyl)pyrazole and/or 3-(2-pyridyl)pyrazole-based ligands (Bell *et al.*, 2003; Paul *et al.*, 2004; Singh *et al.*, 2003; Ward *et al.*, 2001; Zou *et al.*, 2004). In this paper, we present a zinc chloride complex of the ligand 2-[3-(2-pyridyl)pyrazol-1-ylmethyl]pyridine (denoted *L*).

In the title complex, Zn^{II} is five-coordinated by three N donors from one *L* ligand and two Cl^{-} anions (Figure 1). The coordination geometry around each Zn^{II} center can be described as distorted trigonal bipyramidal. The coordinated tridentate *L* ligand gives rise to one five-membered chelate ring [Zn1/N2/C6/C5/N3], with the N2—Zn1—N3 angle equal to 72.73 (3)°, and one six-membered chelate ring [Zn1/N2/N1/C9/C10/N4], with the N2—Zn1—N4 angle equal to 82.34 (8)°. Distortions from ideal trigonal bipyramidal geometry are evident from the bond lengths and bond angles (Table 1).

The mononuclear Zn^{II} complexes form π···π stacking interactions between adjacent pyridine and pyrazole rings of the *L* ligands, with centroid-centroid separations of 3.671 (1) Å (dashed open lines in Figure 2) and 3.681 (1) Å (dashed solid lines in Figure 2), and with a dihedral angle of 4.0° between the planes of the rings (Janiak, 2000; Khlobystov *et al.*, 2001). These interactions generate one-dimensional chains along the *a* axis.

Experimental

The ligand 2-[3-(2-pyridyl)pyrazol-1-ylmethyl]pyridine (*L*) was synthesized according to a general literature procedure (Singh *et al.*, 2003; Song *et al.*, 2005). Reaction of *L* (0.1 mmol) with $ZnCl_2$ (0.1 mmol) in the mixed solution of MeOH (15 ml) and CH_3CN (5 ml) for a few minutes afforded the yellow solid, which was filtered. The resulting solution was kept at room temperature and yellow single crystals suitable for X-ray analysis were obtained by slow evaporation of the solvent after several days. Yield: 30%. Elemental analysis calculated: C, 45.13; H, 3.25; N, 15.04%; found: C, 45.26; H, 3.14; N, 15.11%.

Refinement

H atoms were included in calculated positions and treated in subsequent refinement as riding atoms, with C—H = 0.93 (aromatic) or 0.97 Å (methylene), and with U_{iso}(H) = 1.2 or 1.5 U_{eq}(C).

Figures

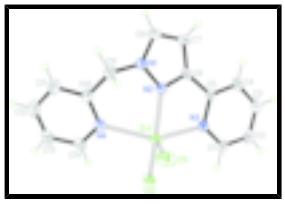


Fig. 1. The molecular structure of the title complex. Displacement ellipsoids are drawn at the 30% probability level for non-H atoms.

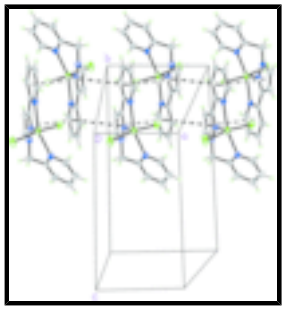


Fig. 2. Part of the crystal packing in the title complex, showing a chain based on π···π stacking (dashed solid and open lines).

Dichlorido{2-[3-(2-pyridyl)pyrazol-1-ylmethyl]pyridine}zinc(II)

Crystal data

$[ZnCl_2(C_{14}H_{12}N_4)]$	F_{000} = 752
M_r = 372.55	D_x = 1.651 Mg m^{-3}
Monoclinic, $P2_1/n$	Mo $K\alpha$ radiation λ = 0.71073 Å
Hall symbol: -P 2yn	Cell parameters from 2602 reflections
a = 7.2813 (10) Å	θ = 2.7–24.8º
b = 15.248 (2) Å	μ = 1.99 mm^{-1}
c = 13.5034 (19) Å	T = 294 (2) K
β = 90.967 (2)º	Block, yellow
V = 1499.0 (4) Å^3	0.20 × 0.18 × 0.14 mm
Z = 4	

Data collection

Bruker SMART CCD diffractometer	3037 independent reflections
Radiation source: fine-focus sealed tube	2174 reflections with $I > 2\sigma(I)$
Monochromator: graphite	R_{int} = 0.040
T = 294(2) K	θ_{max} = 26.4º
φ and ω scans	θ_{min} = 2.0º
Absorption correction: multi-scan (SADABS; Bruker, 1998)	h = −9→5
T_{min} = 0.692, T_{max} = 0.768	k = −19→16
8317 measured reflections	l = −16→16

Refinement

Refinement on F^2	Secondary atom site location: difference Fourier map
Least-squares matrix: full	Hydrogen site location: inferred from neighbouring sites
$R[F^2 > 2\sigma(F^2)] = 0.033$	H-atom parameters constrained
$wR(F^2) = 0.073$	$w = 1/[\sigma^2(F_o^2) + (0.0275P)^2 + 0.3375P]$ where $P = (F_o^2 + 2F_c^2)/3$
$S = 1.01$	$(\Delta/\sigma)_{max} < 0.001$
3037 reflections	$\Delta\rho_{max} = 0.27$ e Å^{-3}
190 parameters	$\Delta\rho_{min} = -0.35$ e Å^{-3}
Primary atom site location: structure-invariant direct methods	Extinction correction: none

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 $F^2 > 2$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	0.16259 (4)	1.08055 (2)	0.29567 (3)	0.03662 (12)
C1	0.1757 (4)	1.1774 (2)	0.5136 (2)	0.0449 (8)
H1A	0.1377	1.2270	0.4786	0.054*
C2	0.2070 (4)	1.1849 (2)	0.6138 (2)	0.0480 (8)
H2A	0.1902	1.2381	0.6460	0.058*
C3	0.2636 (4)	1.1118 (2)	0.6654 (2)	0.0452 (8)
H3A	0.2894	1.1155	0.7330	0.054*
C4	0.2820 (4)	1.0326 (2)	0.6165 (2)	0.0391 (7)
H4A	0.3173	0.9822	0.6506	0.047*
C5	0.2467 (3)	1.03049 (17)	0.51566 (19)	0.0306 (6)
C6	0.2617 (3)	0.95185 (18)	0.4549 (2)	0.0318 (6)
C7	0.3003 (4)	0.86392 (19)	0.4753 (2)	0.0417 (7)
H7A	0.3237	0.8387	0.5369	0.050*
C8	0.2963 (4)	0.82293 (19)	0.3856 (2)	0.0432 (7)
H8A	0.3184	0.7637	0.3745	0.052*
C9	0.2510 (4)	0.8755 (2)	0.2084 (2)	0.0445 (8)
H9A	0.2684	0.8145	0.1905	0.053*
H9B	0.3528	0.9087	0.1820	0.053*

C10	0.0750 (4)	0.90799 (19)	0.1612 (2)	0.0404 (7)
C11	−0.0245 (5)	0.8540 (2)	0.0987 (3)	0.0576 (9)
H11A	0.0123	0.7962	0.0891	0.069*
C12	−0.1790 (5)	0.8857 (3)	0.0502 (3)	0.0659 (10)
H12A	−0.2462	0.8502	0.0068	0.079*
C13	−0.2310 (5)	0.9702 (3)	0.0671 (2)	0.0580 (9)
H13A	−0.3342	0.9935	0.0353	0.070*
C14	−0.1280 (4)	1.0205 (2)	0.1322 (2)	0.0477 (8)
H14A	−0.1660	1.0776	0.1442	0.057*
N1	0.2550 (3)	0.88307 (15)	0.31618 (18)	0.0361 (5)
N2	0.2340 (3)	0.96230 (14)	0.35770 (16)	0.0337 (5)
N3	0.1972 (3)	1.10258 (14)	0.46378 (17)	0.0353 (6)
N4	0.0236 (3)	0.99150 (15)	0.17893 (16)	0.0379 (6)
Cl1	0.41903 (11)	1.12397 (6)	0.21749 (7)	0.0648 (3)
Cl2	−0.05580 (10)	1.18623 (5)	0.29099 (6)	0.0433 (2)

Atomic displacement parameters (Å^2)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
Zn1	0.0335 (2)	0.03112 (19)	0.0450 (2)	0.00138 (15)	−0.00478 (14)	0.00854 (16)
C1	0.0465 (19)	0.0372 (17)	0.051 (2)	0.0021 (14)	−0.0064 (15)	−0.0050 (15)
C2	0.0441 (19)	0.0449 (19)	0.055 (2)	−0.0030 (15)	−0.0004 (15)	−0.0192 (16)
C3	0.0386 (18)	0.062 (2)	0.0355 (18)	−0.0093 (15)	−0.0015 (14)	−0.0085 (16)
C4	0.0308 (16)	0.0507 (19)	0.0356 (17)	−0.0045 (14)	−0.0016 (12)	0.0059 (14)
C5	0.0225 (14)	0.0349 (15)	0.0344 (15)	−0.0004 (12)	−0.0024 (11)	0.0026 (12)
C6	0.0222 (14)	0.0364 (15)	0.0367 (16)	−0.0003 (12)	−0.0006 (12)	0.0046 (12)
C7	0.0471 (19)	0.0393 (17)	0.0389 (18)	0.0061 (14)	0.0026 (14)	0.0126 (14)
C8	0.0466 (19)	0.0278 (15)	0.055 (2)	0.0045 (13)	0.0026 (15)	0.0056 (14)
C9	0.0493 (19)	0.0441 (18)	0.0400 (18)	0.0106 (15)	−0.0011 (14)	−0.0105 (14)
C10	0.0410 (18)	0.0443 (19)	0.0360 (16)	−0.0022 (14)	0.0014 (13)	−0.0047 (14)
C11	0.062 (2)	0.053 (2)	0.057 (2)	−0.0007 (17)	−0.0059 (18)	−0.0208 (17)
C12	0.064 (3)	0.077 (3)	0.056 (2)	−0.004 (2)	−0.0168 (19)	−0.023 (2)
C13	0.049 (2)	0.076 (3)	0.049 (2)	0.0038 (19)	−0.0168 (16)	−0.0018 (19)
C14	0.0442 (19)	0.0478 (19)	0.051 (2)	0.0022 (15)	−0.0100 (15)	0.0033 (16)
N1	0.0386 (14)	0.0296 (12)	0.0400 (14)	0.0050 (11)	−0.0006 (11)	−0.0008 (11)
N2	0.0390 (14)	0.0282 (13)	0.0339 (13)	0.0030 (10)	−0.0045 (10)	0.0008 (10)
N3	0.0363 (14)	0.0324 (13)	0.0371 (14)	0.0032 (10)	−0.0065 (10)	−0.0021 (10)
N4	0.0403 (15)	0.0385 (14)	0.0346 (13)	0.0011 (11)	−0.0060 (11)	0.0008 (11)
Cl1	0.0379 (5)	0.0673 (6)	0.0895 (7)	−0.0021 (4)	0.0071 (4)	0.0308 (5)
Cl2	0.0409 (4)	0.0365 (4)	0.0521 (5)	0.0065 (3)	−0.0077 (3)	0.0077 (3)

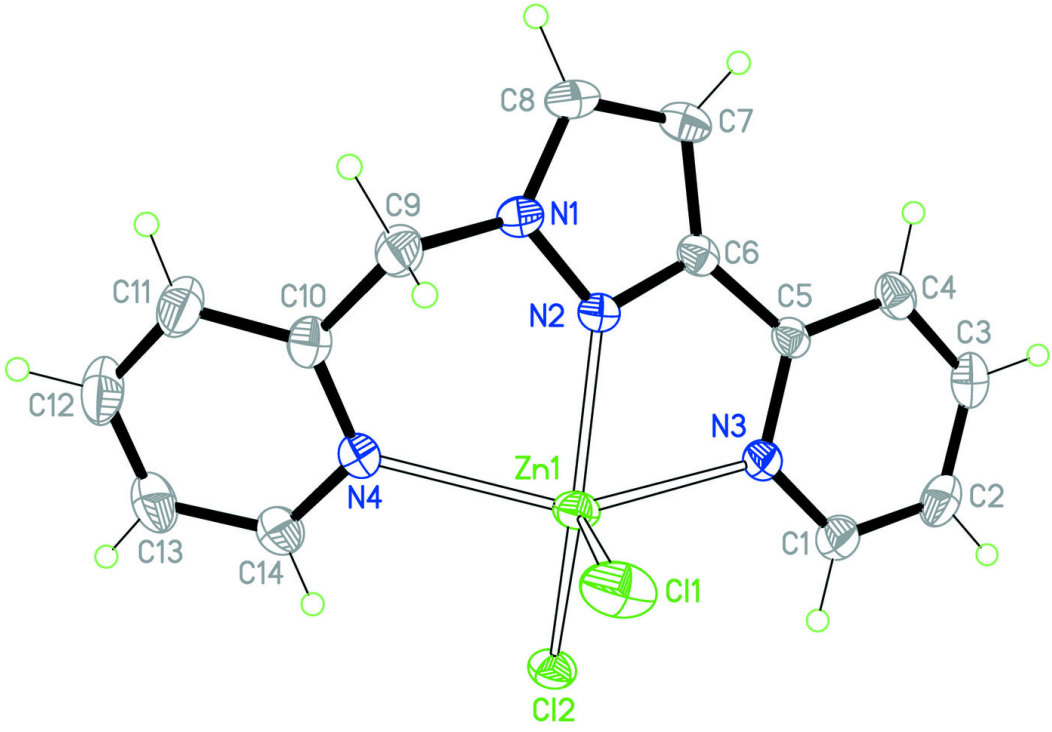
Geometric parameters (Å, °)

Zn1—Cl1	2.2596 (9)	C7—H7A	0.930
Zn1—Cl2	2.2640 (8)	C8—N1	1.342 (4)
Zn1—N2	2.051 (2)	C8—H8A	0.930
Zn1—N3	2.304 (2)	C9—N1	1.459 (4)
Zn1—N4	2.302 (2)	C9—C10	1.505 (4)
C1—N3	1.335 (3)	C9—H9A	0.970

C1—C2	1.374 (4)	C9—H9B	0.970
C1—H1A	0.930	C10—N4	1.350 (4)
C2—C3	1.374 (4)	C10—C11	1.377 (4)
C2—H2A	0.930	C11—C12	1.379 (5)
C3—C4	1.384 (4)	C11—H11A	0.930
C3—H3A	0.930	C12—C13	1.363 (5)
C4—C5	1.382 (4)	C12—H12A	0.930
C4—H4A	0.930	C13—C14	1.378 (4)
C5—N3	1.349 (3)	C13—H13A	0.930
C5—C6	1.458 (4)	C14—N4	1.337 (3)
C6—N2	1.334 (3)	C14—H14A	0.930
C6—C7	1.396 (4)	N1—N2	1.342 (3)
C7—C8	1.363 (4)		
Cl1—Zn1—Cl2	111.34 (3)	N1—C9—C10	113.5 (2)
Cl1—Zn1—N2	104.04 (7)	N1—C9—H9A	108.9
Cl1—Zn1—N3	109.88 (6)	C10—C9—H9A	108.9
Cl1—Zn1—N4	102.14 (7)	N1—C9—H9B	108.9
Cl2—Zn1—N2	144.09 (7)	C10—C9—H9B	108.9
Cl2—Zn1—N3	89.36 (6)	H9A—C9—H9B	107.7
Cl2—Zn1—N4	95.75 (6)	N4—C10—C11	121.9 (3)
N2—Zn1—N3	72.73 (8)	N4—C10—C9	118.2 (3)
N2—Zn1—N4	82.34 (8)	C11—C10—C9	119.9 (3)
N3—Zn1—N4	143.13 (8)	C10—C11—C12	119.9 (3)
N3—C1—C2	123.2 (3)	C10—C11—H11A	120.1
N3—C1—H1A	118.4	C12—C11—H11A	120.1
C2—C1—H1A	118.4	C13—C12—C11	118.6 (3)
C1—C2—C3	118.5 (3)	C13—C12—H12A	120.7
C1—C2—H2A	120.8	C11—C12—H12A	120.7
C3—C2—H2A	120.8	C12—C13—C14	118.9 (3)
C2—C3—C4	119.7 (3)	C12—C13—H13A	120.6
C2—C3—H3A	120.1	C14—C13—H13A	120.6
C4—C3—H3A	120.1	N4—C14—C13	123.6 (3)
C5—C4—C3	118.3 (3)	N4—C14—H14A	118.2
C5—C4—H4A	120.9	C13—C14—H14A	118.2
C3—C4—H4A	120.9	N2—N1—C8	110.4 (2)
N3—C5—C4	122.4 (3)	N2—N1—C9	119.1 (2)
N3—C5—C6	113.6 (2)	C8—N1—C9	130.0 (3)
C4—C5—C6	124.0 (3)	C6—N2—N1	106.7 (2)
N2—C6—C7	109.6 (3)	C6—N2—Zn1	122.67 (18)
N2—C6—C5	116.4 (2)	N1—N2—Zn1	130.60 (17)
C7—C6—C5	134.1 (3)	C1—N3—C5	117.9 (2)
C8—C7—C6	105.3 (3)	C1—N3—Zn1	127.5 (2)
C8—C7—H7A	127.3	C5—N3—Zn1	114.62 (17)
C6—C7—H7A	127.3	C14—N4—C10	117.2 (3)
N1—C8—C7	108.0 (3)	C14—N4—Zn1	118.6 (2)
N1—C8—H8A	126.0	C10—N4—Zn1	123.89 (19)
C7—C8—H8A	126.0		
N3—C1—C2—C3	0.2 (5)	N3—Zn1—N2—C6	1.1 (2)

C1—C2—C3—C4	−2.0 (5)	Cl1—Zn1—N2—N1	−75.0 (2)
C2—C3—C4—C5	1.6 (4)	Cl2—Zn1—N2—N1	114.9 (2)
C3—C4—C5—N3	0.7 (4)	N4—Zn1—N2—N1	25.6 (2)
C3—C4—C5—C6	179.7 (2)	N3—Zn1—N2—N1	178.2 (2)
N3—C5—C6—N2	3.1 (3)	C2—C1—N3—C5	2.1 (4)
C4—C5—C6—N2	−175.9 (2)	C2—C1—N3—Zn1	−177.2 (2)
N3—C5—C6—C7	−177.0 (3)	C4—C5—N3—C1	−2.5 (4)
C4—C5—C6—C7	4.0 (5)	C6—C5—N3—C1	178.5 (2)
N2—C6—C7—C8	0.7 (3)	C4—C5—N3—Zn1	176.8 (2)
C5—C6—C7—C8	−179.2 (3)	C6—C5—N3—Zn1	−2.2 (3)
C6—C7—C8—N1	−0.9 (3)	N2—Zn1—N3—C1	−180.0 (3)
N1—C9—C10—N4	57.2 (4)	Cl1—Zn1—N3—C1	80.9 (2)
N1—C9—C10—C11	−125.4 (3)	Cl2—Zn1—N3—C1	−31.6 (2)
N4—C10—C11—C12	1.7 (5)	N4—Zn1—N3—C1	−130.3 (2)
C9—C10—C11—C12	−175.6 (3)	N2—Zn1—N3—C5	0.73 (18)
C10—C11—C12—C13	−1.1 (6)	Cl1—Zn1—N3—C5	−98.34 (18)
C11—C12—C13—C14	−0.3 (6)	Cl2—Zn1—N3—C5	149.14 (18)
C12—C13—C14—N4	1.2 (5)	N4—Zn1—N3—C5	50.4 (2)
C7—C8—N1—N2	0.7 (3)	C13—C14—N4—C10	−0.7 (5)
C7—C8—N1—C9	173.4 (3)	C13—C14—N4—Zn1	−174.5 (3)
C10—C9—N1—N2	−58.7 (4)	C11—C10—N4—C14	−0.8 (4)
C10—C9—N1—C8	129.1 (3)	C9—C10—N4—C14	176.6 (3)
C7—C6—N2—N1	−0.3 (3)	C11—C10—N4—Zn1	172.6 (2)
C5—C6—N2—N1	179.6 (2)	C9—C10—N4—Zn1	−10.0 (4)
C7—C6—N2—Zn1	177.39 (18)	N2—Zn1—N4—C14	149.3 (2)
C5—C6—N2—Zn1	−2.7 (3)	Cl1—Zn1—N4—C14	−107.9 (2)
C8—N1—N2—C6	−0.3 (3)	Cl2—Zn1—N4—C14	5.4 (2)
C9—N1—N2—C6	−173.9 (2)	N3—Zn1—N4—C14	102.0 (2)
C8—N1—N2—Zn1	−177.7 (2)	N2—Zn1—N4—C10	−24.0 (2)
C9—N1—N2—Zn1	8.7 (4)	Cl1—Zn1—N4—C10	78.8 (2)
Cl1—Zn1—N2—C6	107.9 (2)	Cl2—Zn1—N4—C10	−167.9 (2)
Cl2—Zn1—N2—C6	−62.2 (3)	N3—Zn1—N4—C10	−71.3 (3)
N4—Zn1—N2—C6	−151.4 (2)		

Fig. 1


C8
C7
C9
N1
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C11
C10
N2
C5
C4
C3
C12
N3
Zn1
N4
C2
C13
C1
C14
Cl1
Cl2

Fig. 2