CRYSTALLOGRAPHIC COMMUNICATIONS

OPEN ACCESS

ISSN 2056-9890

Crystal structure of dichloridobis(4-ethylaniline-κN)zinc

J. Govindaraj,[a] S. Thirumurugan,[b] D. Snehalatha Reddy,[b] K. Anbalagan[b] and A. SubbiahPandi[c]*

[a]Department of Physics, Pachaiyappa's College for Men, Kanchipuram 631 501, India, [b]Department of Chemistry, Pondicherry University, Pondicherry 605 014, India, and [c]Department of Physics, Presidency College (Autonomous), Chennai 600 005, India. *Correspondence e-mail: aspandian59@gmail.com

Received 17 November 2014; accepted 21 December 2014

Edited by K. Fejfarova, Institute of Macromolecular Chemistry, AS CR, v.v.i, Czech Republic

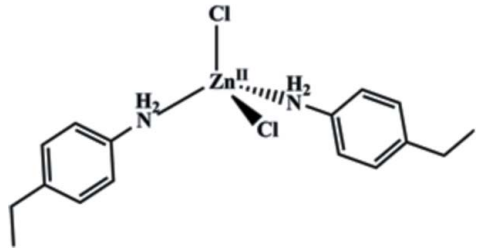
The title compound, $[ZnCl_2(C_8H_{11}N)_2]$, was synthesized by the reaction of zinc dichloride and 4-ethylaniline. The Zn^{2+} cation is coordinated by two Cl^- anions and the N atoms of two 4-ethylaniline ligands, forming a distorted $Zn(N_2Cl_2)$ tetrahedron. The dihedral angle between the two benzene rings is 85.3 (2)° The Zn atom lies on a twofold rotation axis. The ethyl substituents are disordered over two sets of sites in a 0.74 (2):0.26 (2) ratio. In the crystal, N—H···Cl hydrogen bonds link the molecules into sheets perpendicular to the *a* axis. C—H···Cl interactions also occur.

Keywords: crystal structure; zinc complex; tetrahedral coordination; hydrogen bonding.

CCDC reference: 1040586

1. Related literature

For the biological activity and potential applications of mixed-ligand dichloridozinc complexes, see: Tang & Shay (2001); Lynch *et al.* (2001); Coulston & Dandona (1980); May & Contoreggi (1982). For a related structure, see; Ejaz *et al.* (2009).



2. Experimental

2.1. Crystal data

$[ZnCl_2(C_8H_{11}N)_2]$
M_r = 378.63
Monoclinic, $C2/c$
a = 32.7291 (16) Å
b = 4.7499 (4) Å
c = 11.6479 (8) Å
β = 98.016 (7)°
V = 1793.1 (2) Å^3
Z = 4
Mo $K\alpha$ radiation
μ = 1.66 mm^{-1}
T = 293 K
0.35 × 0.30 × 0.25 mm

2.2. Data collection

Oxford diffraction Xcalibur diffractometer with an Eos detector
Absorption correction: multi-scan (*CrysAlis PRO*; Oxford Diffraction, 2009)
T_{min} = 0.564, T_{max} = 0.660
4578 measured reflections
1578 independent reflections
1440 reflections with $I > 2\sigma(I)$
R_{int} = 0.029

2.3. Refinement

$R[F^2 > 2\sigma(F^2)]$ = 0.028
$wR(F^2)$ = 0.077
S = 1.10
1578 reflections
123 parameters
66 restraints
H atoms treated by a mixture of independent and constrained refinement
$\Delta\rho_{max}$ = 0.46 e Å^{-3}
$\Delta\rho_{min}$ = −0.28 e Å^{-3}

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

D—H···A	D—H	H···A	D···A	D—H···A
C2—H2···Cl1^{i}	0.93	2.94	3.630 (2)	132
N1—H1A···Cl1^{ii}	0.88 (2)	2.65 (2)	3.424 (2)	149 (2)
N1—H1B···Cl1^{iii}	0.88 (2)	2.66 (2)	3.5083 (19)	161 (2)

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

Data collection: *CrysAlis CCD* (Oxford Diffraction, 2009); cell refinement: *CrysAlis RED* (Oxford Diffraction, 2009); data reduction: *CrysAlis RED*; program(s) used to solve structure: *SHELXS97* (Sheldrick, 2008); program(s) used to refine structure: *SHELXL97* (Sheldrick, 2008); molecular graphics: *ORTEP-3 for Windows* (Farrugia, 2012) and *DIAMOND* (Brandenburg, 2006); software used to prepare material for publication: *SHELXL97* and *PLATON* (Spek, 2009).

Acknowledgements

KA is thankful to the CSIR, New Delhi [Lr: No. 01 (2570)/12/EMR-II/3.4.2012], for financial support through a major research project. The authors are thankful to the Department of Chemistry, Pondicherry University, for the single-crystal XRD instrumentation facility.

Supporting information for this paper is available from the IUCr electronic archives (Reference: FF2133).

References

Brandenburg, K. (2006). *DIAMOND*. Crystal Impact GbR, Bonn, Germany.
Coulston, L. & Dandona, P. (1980). *Diabetes*, **29**, 665–667.
Ejaz, Sahin, O. & Khan, I. U. (2009). *Acta Cryst.* E**65**, m1457.
Farrugia, L. J. (2012). *J. Appl. Cryst.* **45**, 849–854.
Lynch, C. J., Patson, B. J., Goodman, S. A., Trapolsi, D. & Kimball, S. R. (2001). *Am. J. Physiol. Endocrinol. Metab.* **281**, E25–E34.
May, J. M. & Contoreggi, C. S. (1982). *J. Biol. Chem.* **257**, 4362–4368.
Oxford Diffraction (2009). *CrysAlis CCD*, *CrysAlis RED* and *CrysAlis PRO*. Oxford Diffraction Ltd, Yarnton, England.
Sheldrick, G. M. (2008). *Acta Cryst.* A**64**, 112–122.
Spek, A. L. (2009). *Acta Cryst.* D**65**, 148–155.
Tang, X.-H. & Shay, N. F. (2001). *J. Nutr.* **131**, 1414–1420.

supporting information

Acta Cryst. (2015). E**71**, m21–m22 [doi:10.1107/S2056989014027832]

Crystal structure of dichloridobis(4-ethylaniline-κN)zinc

J. Govindaraj, S. Thirumurugan, D. Snehalatha Reddy, K. Anbalagan and A. SubbiahPandi

S1. Comment

Zinc has many biological functions. It is considered to be an essential nutrient that is required for optimal growth and normal development of vertebrate organisms, as well as being important for maintaining the structure of many proteins. From previous research results, it has been known for many years that zinc mimics the actions of insulin on cells, including promotion of both lipogenesis and glucose transport. Zinc deficiency may indeed affect the optimal functioning of the insulin-signaling pathway (Tang & Shay, 2001; Lynch *et al.*, 2001; Coulston & Dandona, 1980; May & Contoreggi, 1982).

In the title compound (I), (Fig. 1), the Zn^{2+} cation lies on a crystallographic twofold rotation axis, with one half of the molecule connected to the other on by this symmetry operation. The bond distance Zn—Cl and Zn—N are 2.2409 (6) and 2.048 (2) Å, and the bond angles Cl—Zn—Cl and N—Zn—N are 109.41 (3) and 114.80 (11)°. All bond lengths and bond angles in (I) are in the range of expected values. The dihedral angle between the aromatic rings of the 4-ethylaniline ligands is 85.3 (2)°.

N—H···Cl hydrogen bonds serve to link the molecules into sheets perpendicular to the *a* axis.

S2. Experimental

The title compound was synthesized using zinc chloride (0.5 g, 1 mmol) and 4-ethylaniline (0.91 ml, 2 mmol) in 20 ml of ethanol stirring for 2 h. Colorless crystals were obtained and recrystallized from ethanol. The resulting solution was subjected to crystallization by slow evaporation of the solvent resulting in single crystals suitable for X-ray crystallographic studies.

S3. Refinement

N and C-bound H atoms were positioned geometrically (C–H = 0.93–0.97 Å) and allowed to ride on their parent atoms, with U_{iso}(H) = 1.5U_{eq}(C) for methyl H atoms and 1.2U_{eq}(C) for all other H atoms.

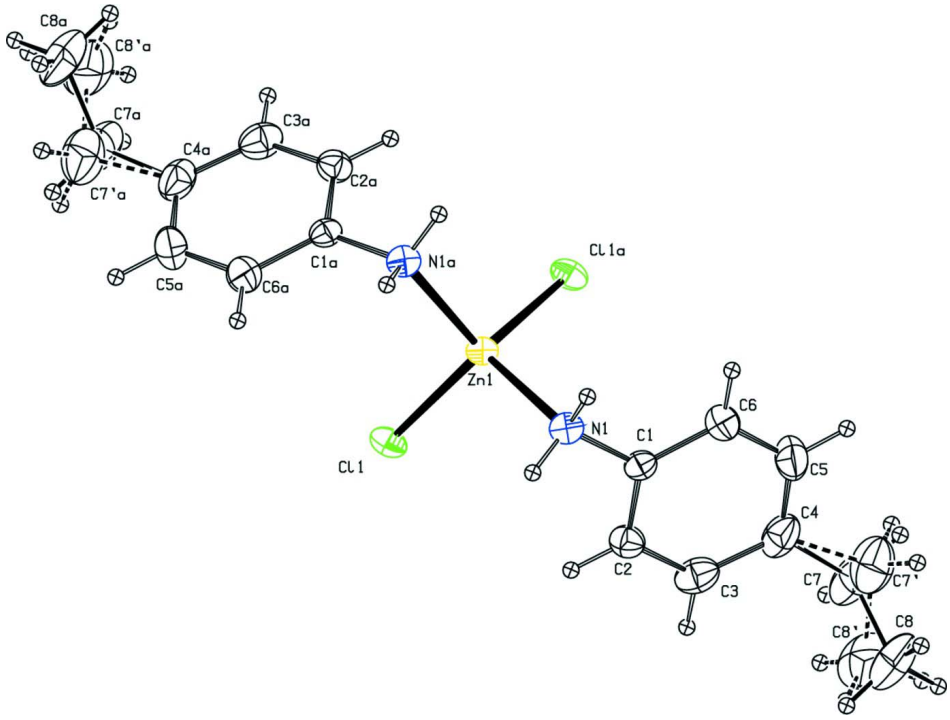


Figure 1

The molecular structure of the title compound with displacement ellipsoids drawn at the 30% probability level.

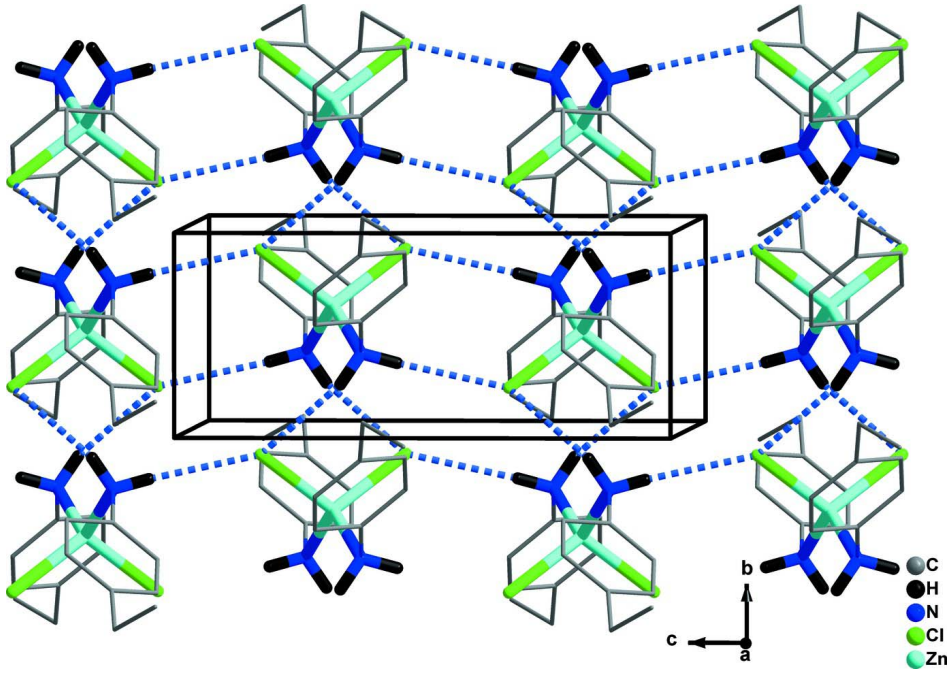


Figure 2

The crystal packing of the title compound viewed down the *a* axis showing the hydrogen bonded sheet. Hydrogen bond are shown as dashed lines. The minor disorder component and hydrogen atoms not participating in N—H···Cl interactions are omitted for clarity.

Dichloridobis(4-ethylaniline-κN)zinc

Crystal data

$[ZnCl_2(C_8H_{11}N)_2]$
$M_r = 378.63$
Monoclinic, $C2/c$
Hall symbol: -C 2yc
$a = 32.7291\ (16)$ Å
$b = 4.7499\ (4)$ Å
$c = 11.6479\ (8)$ Å
$\beta = 98.016\ (7)°$
$V = 1793.1\ (2)$ Å^3
$Z = 4$
$F(000) = 784$
$D_x = 1.403$ Mg m^{-3}
Mo $K\alpha$ radiation, $\lambda = 0.71073$ Å
Cell parameters from 5623 reflections
$\theta = 2.6–24.9°$
$\mu = 1.66$ mm^{-1}
$T = 293$ K
Block, colourless
0.35 × 0.30 × 0.25 mm

Data collection

Oxford diffraction Xcalibur diffractometer with an Eos detector
Radiation source: fine-focus sealed tube
Graphite monochromator
ω and φ scans
Absorption correction: multi-scan (*CrysAlis PRO*; Oxford Diffraction, 2009)
$T_{min} = 0.564$, $T_{max} = 0.660$
4578 measured reflections
1578 independent reflections
1440 reflections with $I > 2\sigma(I)$
$R_{int} = 0.029$
$\theta_{max} = 25.0°$, $\theta_{min} = 4.6°$
$h = -36\rightarrow38$
$k = -5\rightarrow5$
$l = -13\rightarrow13$

Refinement

Refinement on F^2
Least-squares matrix: full
$R[F^2 > 2\sigma(F^2)] = 0.028$
$wR(F^2) = 0.077$
$S = 1.10$
1578 reflections
123 parameters
66 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
$w = 1/[\sigma^2(F_o^2) + (0.0411P)^2]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} = 0.001$
$\Delta\rho_{max} = 0.46$ e Å^{-3}
$\Delta\rho_{min} = -0.28$ 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}	Occ. (<1)
C1	0.41733 (7)	−0.0083 (5)	0.81147 (18)	0.0354 (5)	
C2	0.41424 (8)	0.1721 (5)	0.90189 (19)	0.0434 (6)	
H2	0.4358	0.1863	0.9628	0.052*	

C3	0.37917 (9)	0.3321 (6)	0.9021 (2)	0.0583 (7)	
H3	0.3775	0.4538	0.9638	0.070*	
C4	0.34669 (9)	0.3176 (7)	0.8144 (3)	0.0638 (8)	
C5	0.35114 (10)	0.1405 (8)	0.7234 (3)	0.0727 (9)	
H5	0.3300	0.1306	0.6613	0.087*	
C6	0.38593 (9)	−0.0229 (6)	0.7211 (2)	0.0557 (7)	
H6	0.3880	−0.1417	0.6586	0.067*	
C7	0.3096 (3)	0.505 (3)	0.8230 (13)	0.096 (4)	0.74 (2)
H7A	0.3191	0.6916	0.8480	0.115*	0.74 (2)
H7B	0.2935	0.5229	0.7469	0.115*	0.74 (2)
C8	0.2826 (2)	0.392 (3)	0.9062 (8)	0.108 (3)	0.74 (2)
H8A	0.2596	0.5159	0.9088	0.162*	0.74 (2)
H8B	0.2983	0.3773	0.9821	0.162*	0.74 (2)
H8C	0.2726	0.2085	0.8809	0.162*	0.74 (2)
C7′	0.3026 (5)	0.427 (8)	0.800 (3)	0.090 (7)	0.26 (2)
H7′1	0.2834	0.2723	0.7812	0.109*	0.26 (2)
H7′2	0.2984	0.5626	0.7372	0.109*	0.26 (2)
C8′	0.2950 (11)	0.565 (9)	0.913 (2)	0.119 (8)	0.26 (2)
H8′1	0.2672	0.6353	0.9054	0.178*	0.26 (2)
H8′2	0.3139	0.7185	0.9309	0.178*	0.26 (2)
H8′3	0.2990	0.4293	0.9748	0.178*	0.26 (2)
N1	0.45462 (6)	−0.1686 (4)	0.80964 (16)	0.0371 (4)	
Cl1	0.530459 (19)	0.33620 (12)	0.89413 (4)	0.04150 (19)	
Zn1	0.5000	0.06363 (7)	0.7500	0.03228 (16)	
H1A	0.4494 (7)	−0.320 (4)	0.7676 (17)	0.048 (7)*	
H1B	0.4647 (7)	−0.216 (5)	0.8810 (14)	0.058 (8)*	

Atomic displacement parameters (Å²)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
C1	0.0385 (13)	0.0332 (11)	0.0360 (12)	−0.0035 (11)	0.0103 (10)	0.0059 (9)
C2	0.0492 (15)	0.0410 (13)	0.0398 (12)	0.0043 (12)	0.0059 (10)	0.0019 (10)
C3	0.0674 (19)	0.0497 (16)	0.0624 (17)	0.0114 (16)	0.0253 (15)	0.0013 (13)
C4	0.0467 (16)	0.068 (2)	0.080 (2)	0.0142 (16)	0.0189 (15)	0.0256 (16)
C5	0.0452 (17)	0.104 (3)	0.0649 (19)	−0.0029 (18)	−0.0070 (14)	0.0154 (18)
C6	0.0479 (17)	0.0712 (18)	0.0472 (15)	−0.0081 (15)	0.0034 (13)	−0.0095 (13)
C7	0.061 (4)	0.103 (7)	0.129 (7)	0.030 (4)	0.036 (4)	0.032 (5)
C8	0.060 (4)	0.119 (7)	0.155 (6)	0.017 (4)	0.048 (4)	0.007 (5)
C7′	0.069 (10)	0.075 (11)	0.126 (11)	0.019 (8)	0.010 (9)	0.019 (9)
C8′	0.093 (14)	0.131 (16)	0.136 (13)	0.051 (12)	0.031 (11)	0.011 (13)
N1	0.0438 (12)	0.0288 (10)	0.0396 (11)	0.0005 (9)	0.0087 (9)	0.0011 (8)
Cl1	0.0565 (4)	0.0367 (3)	0.0300 (3)	0.0007 (3)	0.0015 (2)	−0.0050 (2)
Zn1	0.0388 (3)	0.0283 (2)	0.0307 (2)	0.000	0.00804 (16)	0.000

Geometric parameters (Å, º)

C1—C6	1.367 (3)	C8—H8A	0.9600
C1—C2	1.372 (3)	C8—H8B	0.9600

C1—N1	1.441 (3)	C8—H8C	0.9600
C2—C3	1.377 (4)	C7′—C8′	1.527 (19)
C2—H2	0.9300	C7′—H7′1	0.9700
C3—C4	1.370 (4)	C7′—H7′2	0.9700
C3—H3	0.9300	C8′—H8′1	0.9600
C4—C5	1.377 (4)	C8′—H8′2	0.9600
C4—C7	1.521 (6)	C8′—H8′3	0.9600
C4—C7′	1.521 (10)	N1—Zn1	2.0478 (19)
C5—C6	1.381 (4)	N1—H1A	0.875 (16)
C5—H5	0.9300	N1—H1B	0.881 (16)
C6—H6	0.9300	Cl1—Zn1	2.2409 (5)
C7—C8	1.500 (11)	Zn1—N1^{i}	2.0478 (19)
C7—H7A	0.9700	Zn1—Cl1^{i}	2.2409 (6)
C7—H7B	0.9700		

C6—C1—C2	119.7 (2)	H8A—C8—H8B	109.5
C6—C1—N1	120.6 (2)	C7—C8—H8C	109.5
C2—C1—N1	119.6 (2)	H8A—C8—H8C	109.5
C1—C2—C3	119.8 (2)	H8B—C8—H8C	109.5
C1—C2—H2	120.1	C4—C7′—C8′	108.5 (16)
C3—C2—H2	120.1	C4—C7′—H7′1	110.0
C4—C3—C2	122.1 (3)	C8′—C7′—H7′1	110.0
C4—C3—H3	119.0	C4—C7′—H7′2	110.0
C2—C3—H3	119.0	C8′—C7′—H7′2	110.0
C3—C4—C5	116.8 (3)	H7′1—C7′—H7′2	108.4
C3—C4—C7	117.7 (7)	C7′—C8′—H8′1	109.5
C5—C4—C7	125.4 (7)	C7′—C8′—H8′2	109.5
C3—C4—C7′	133.8 (10)	H8′1—C8′—H8′2	109.5
C5—C4—C7′	108.9 (10)	C7′—C8′—H8′3	109.5
C7—C4—C7′	18.8 (14)	H8′1—C8′—H8′3	109.5
C4—C5—C6	122.3 (3)	H8′2—C8′—H8′3	109.5
C4—C5—H5	118.9	C1—N1—Zn1	112.09 (13)
C6—C5—H5	118.9	C1—N1—H1A	110.0 (16)
C1—C6—C5	119.3 (3)	Zn1—N1—H1A	110.4 (16)
C1—C6—H6	120.3	C1—N1—H1B	109.2 (17)
C5—C6—H6	120.3	Zn1—N1—H1B	105.3 (17)
C8—C7—C4	112.3 (8)	H1A—N1—H1B	110 (2)
C8—C7—H7A	109.1	N1^{i}—Zn1—N1	114.80 (11)
C4—C7—H7A	109.1	N1^{i}—Zn1—Cl1^{i}	108.97 (6)
C8—C7—H7B	109.1	N1—Zn1—Cl1^{i}	107.31 (6)
C4—C7—H7B	109.1	N1^{i}—Zn1—Cl1	107.31 (6)
H7A—C7—H7B	107.9	N1—Zn1—Cl1	108.97 (6)
C7—C8—H8A	109.5	Cl1^{i}—Zn1—Cl1	109.41 (3)
C7—C8—H8B	109.5		

C6—C1—C2—C3	1.5 (4)	C3—C4—C7—C8	−77.7 (15)
N1—C1—C2—C3	178.0 (2)	C5—C4—C7—C8	104.8 (14)
C1—C2—C3—C4	0.1 (4)	C7′—C4—C7—C8	74 (5)

C2—C3—C4—C5	−1.8 (4)	C3—C4—C7′—C8′	−4 (5)
C2—C3—C4—C7	−179.6 (5)	C5—C4—C7′—C8′	167 (3)
C2—C3—C4—C7′	168 (2)	C7—C4—C7′—C8′	−39 (4)
C3—C4—C5—C6	2.0 (5)	C6—C1—N1—Zn1	95.6 (2)
C7—C4—C5—C6	179.6 (6)	C2—C1—N1—Zn1	−80.9 (2)
C7′—C4—C5—C6	−170.4 (17)	C1—N1—Zn1—N1^{i}	−159.46 (17)
C2—C1—C6—C5	−1.3 (4)	C1—N1—Zn1—Cl1^{i}	−38.19 (16)
N1—C1—C6—C5	−177.8 (2)	C1—N1—Zn1—Cl1	80.18 (15)
C4—C5—C6—C1	−0.5 (5)		

Symmetry code: (i) −*x*+1, *y*, −*z*+3/2.

Hydrogen-bond geometry (Å, º)

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
C2—H2···Cl1^{ii}	0.93	2.94	3.630 (2)	132
N1—H1*A*···Cl1^{iii}	0.88 (2)	2.65 (2)	3.424 (2)	149 (2)
N1—H1*B*···Cl1^{iv}	0.88 (2)	2.66 (2)	3.5083 (19)	161 (2)

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