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Tris(ethylenediamine)zinc(II) dichloride monohydrate

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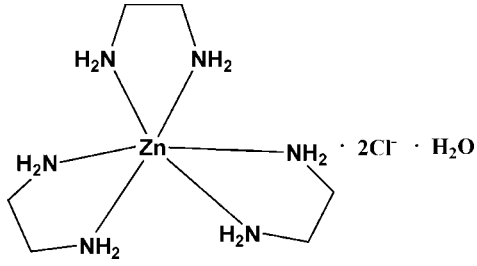
Key indicators: single-crystal X-ray study; T = 293 K; mean σ(C–C) = 0.005 Å; R factor = 0.036; wR factor = 0.086; data-to-parameter ratio = 20.5.

The asymmetric unit of the title compound, $[Zn(C_2H_8N_2)_3]Cl_2\cdot H_2O$, contains a discrete $[Zn(C_2H_8N_2)_3]^{2+}$ cation with a distorted octahedral geometry around Zn, two uncoordinated chloride ions and one water molecule. The crystal structure exhibits N—H···O, N—H···Cl and O—H···O hydrogen bonds.

Related literature

For related structures, see: Bernhardt & Riley (2003); Cernak *et al.* (1984); Emsley *et al.* (1989); Muralikrishna *et al.* (1983); Nesterova *et al.* (2006); Wu *et al.* (2001).



Experimental

Crystal data

$[Zn(C_2H_8N_2)_3]Cl_2\cdot H_2O$
M_r = 334.60
Monoclinic, $P2_1/c$
a = 8.8165 (10) Å
b = 11.9379 (14) Å
c = 14.4043 (17) Å
β = 92.804 (2)°
V = 1514.2 (3) Å^3
Z = 4
Mo $K\alpha$ radiation
μ = 1.97 mm^{-1}
T = 293 (2) K
0.25 × 0.22 × 0.16 mm

Data collection

Bruker APEX CCD diffractometer
Absorption correction: multi-scan (*SADABS*; Sheldrick, 2000)
T_{min} = 0.639, T_{max} = 0.744
11550 measured reflections
2975 independent reflections
2511 reflections with $I > 2\sigma(I)$
R_{int} = 0.030

Refinement

$R[F^2 > 2\sigma(F^2)]$ = 0.035
$wR(F^2)$ = 0.085
S = 1.10
2975 reflections
145 parameters
H-atom parameters constrained
$\Delta\rho_{max}$ = 0.39 e Å^{-3}
$\Delta\rho_{min}$ = −0.27 e Å^{-3}

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

D—H···A	D—H	H···A	D···A	D—H···A
N1—H1D···Cl2^{i}	0.90	2.86	3.739 (3)	165
N2—H2C···Cl1	0.90	2.50	3.363 (3)	162
N2—H2D···Cl2^{ii}	0.90	2.48	3.332 (2)	158
N3—H3C···O1W^{iii}	0.90	2.27	3.159 (3)	169
N3—H3D···Cl2	0.90	2.73	3.605 (3)	165
N4—H4C···O1W^{iv}	0.90	2.39	3.260 (3)	164
N4—H4D···Cl1	0.90	2.52	3.375 (3)	159
N5—H5D···Cl2^{i}	0.90	2.57	3.420 (3)	158
N6—H6C···Cl1^{iv}	0.90	2.44	3.309 (3)	161
N6—H6D···Cl2	0.90	2.58	3.471 (3)	172
O1W—H1WA···Cl1	0.85	2.25	3.097 (3)	171
O1W—H1WB···Cl2^{v}	0.85	2.34	3.187 (3)	180

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

Data collection: *SMART* (Bruker, 2000); cell refinement: *SAINT* (Bruker, 2000); data reduction: *SAINT*; program(s) used to solve structure: *SHELXS97* (Sheldrick, 2008); program(s) used to refine structure: *SHELXL97* (Sheldrick, 2008); molecular graphics: *SHELXTL* (Sheldrick, 2008); 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: BT2779).

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Tris(ethylenediamine)zinc(II) dichloride monohydrate

Lin Cheng, Yan-Yan Sun, Ya-Wen Zhang and Gang Xu

S1. Comment

The preparation of complexes including different stereoisomers is a fascinating and promising means. There are many complexes including $[Zn(en)_3]^{2+}$ cation (en = ethylenediamine), which have been reported, due that $[Zn(en)_3]^{2+}$ cation has two simple and intuitive stereoisomers (Bernhardt *et al.*, 2003; Cernak *et al.*, 1984; Emsley *et al.*, 1989; Muralikrishna *et al.*, 1983; Nesterova *et al.*, 2006; Wu *et al.*, 2001). Different from the similar compound $[Zn(en)_3]Cl_2.2H_2O$ (Muralikrishna *et al.*, 1983; Wu *et al.*, 2001), here, we report a salt $[Zn(en)_3]Cl_2.H_2O$. In the asymmetric unit of the salt, there are only one crystal water molecule.

The asymmetric unit of the title salt, $[Zn(en)_3]Cl_2.H_2O$, contains a discrete $[Zn(en)_3]^{2+}$ cation, two uncoordinated chloride ions and one water molecule. The Zn(II) ion displays a distorted octahedral geometry, being surrounded by three en ligands. The Zn···N distances are between 2.159 (2) and 2.220 (2) Å. Each en acts as a chelating bidentate ligand. In crystal, the $[Zn(en)_3]^{2+}$ cations, chloride ions and the crystal water are linked together by N—H···O, N—H···Cl and O—H···Cl hydrogen bonds (Table 2).

S2. Experimental

To a solution of $ZnCl_2.2H_2O$ (0.172 g, 1 mmol) in CH_3OH (5 ml), an aqueous solution (5 ml) of bib (bib = 1,3-bis(4,5-Dihydro-1*H*-imidazol-2-yl)benzene) (0.214 g, 1 mmol) was added. After the mixture was stirred for half an hour, a white precipitate formed. 3 ml en was added to the mixture and the precipitate disappeared. Then the mixture was stirred for an hour and filtered. The filtrate was allowed to evaporate slowly at room temperature. After 3 weeks, colorless block shaped crystals were obtained in 40% yield (0.034 g) based on Zn(II).

S3. Refinement

H atoms were located in a difference map, but refined using a riding model with N—H = 0.90, O—H = 0.85 Å and C—H = 0.97 Å and with U_{iso}(H) = 1.2 U_{iso}(C,N,O).

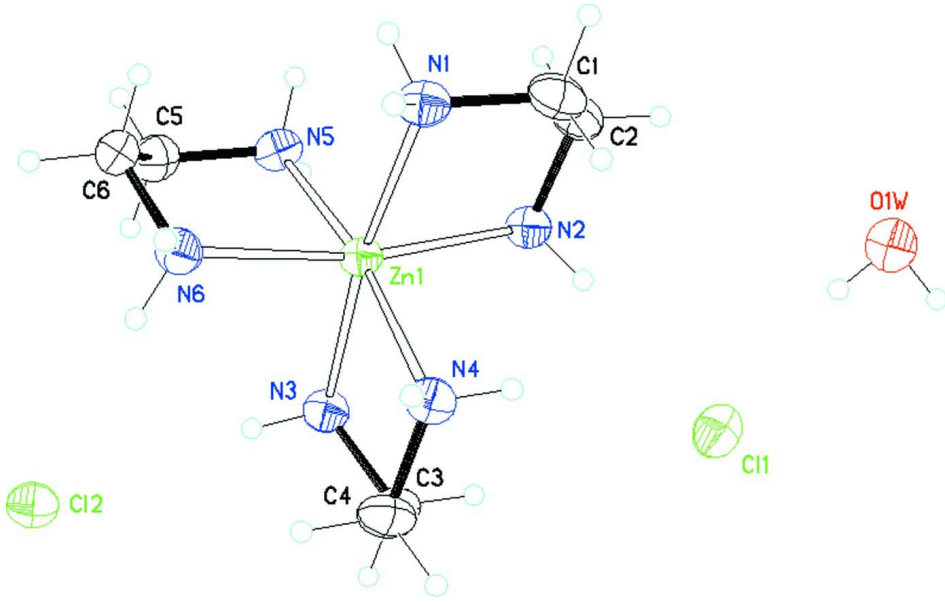

Figure 1

The asymmetric unit of the title compound with 30% displacement ellipsoids.

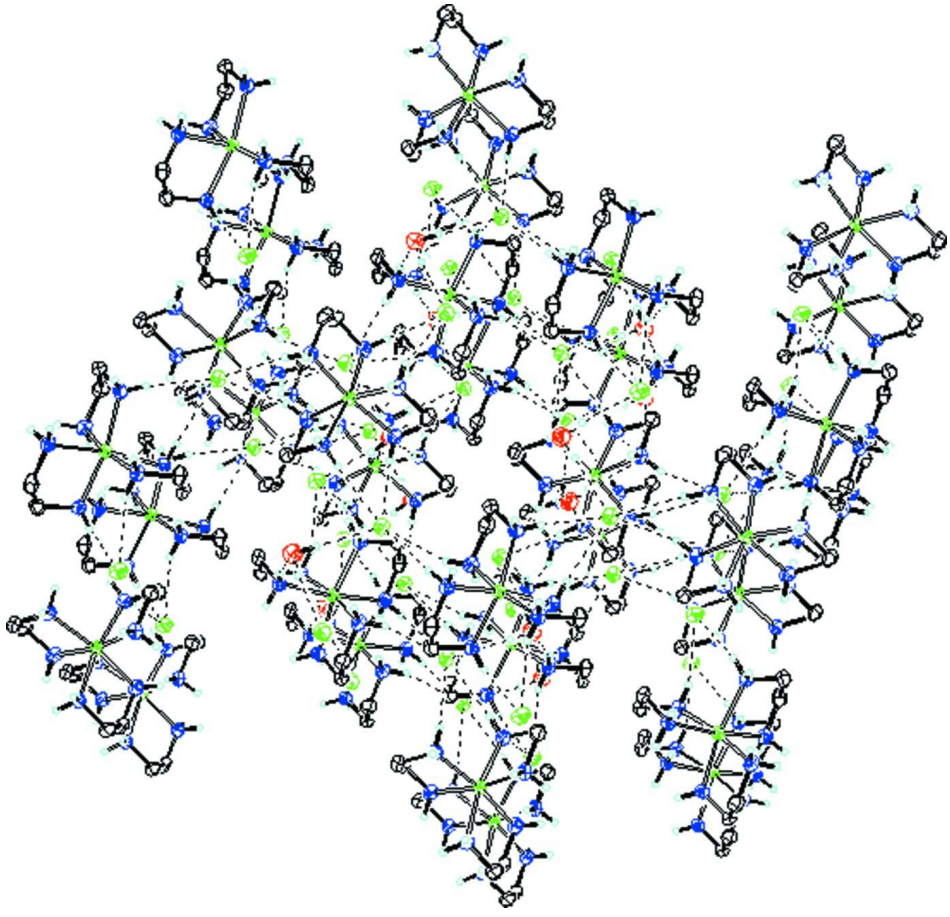

Figure 2

Partial packing diagram. The H atoms bonded to C atoms are omitted for clarity.

Tris(ethylenediamine)zinc(II) dichloride monohydrate

Crystal data

$[Zn(C_2H_8N_2)_3]Cl_2·H_2O$
M_r = 334.60
Monoclinic, $P2_1/c$
Hall symbol: -P 2ybc
a = 8.8165 (10) Å
b = 11.9379 (14) Å
c = 14.4043 (17) Å
β = 92.804 (2)°
V = 1514.2 (3) Å^3
Z = 4
$F(000)$ = 704
D_x = 1.468 Mg m^{-3}
Mo $K\alpha$ radiation, λ = 0.71073 Å
Cell parameters from 783 reflections
θ = 2.5–28.0°
μ = 1.97 mm^{-1}
T = 293 K
Block, colorless
0.25 × 0.22 × 0.16 mm

Data collection

Bruker APEX CCD diffractometer
Radiation source: fine-focus sealed tube
Graphite monochromator
φ and ω scan
Absorption correction: multi-scan (*SADABS*; Sheldrick, 2000)
T_{min} = 0.639, T_{max} = 0.744
11550 measured reflections
2975 independent reflections
2511 reflections with $I > 2\sigma(I)$
R_{int} = 0.030
θ_{max} = 26.0°, θ_{min} = 2.2°
h = −10→10
k = −14→14
l = −17→17

Refinement

Refinement on F^2
Least-squares matrix: full
$R[F^2 > 2\sigma(F^2)]$ = 0.035
$wR(F^2)$ = 0.085
S = 1.10
2975 reflections
145 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
$w = 1/[\sigma^2(F_o^2) + (0.0384P)^2 + 0.5868P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} < 0.001$
$\Delta\rho_{max}$ = 0.39 e Å^{-3}
$\Delta\rho_{min}$ = −0.27 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	0.24154 (3)	0.55616 (3)	0.20878 (2)	0.03553 (12)

Cl1	0.31636 (9)	0.23340 (7)	0.36125 (6)	0.0567 (2)
Cl2	0.20561 (8)	0.87644 (7)	0.38972 (5)	0.0505 (2)
C1	0.3870 (3)	0.3760 (3)	0.1009 (2)	0.0555 (8)
H1A	0.4452	0.3450	0.1538	0.067*
H1B	0.4297	0.3480	0.0446	0.067*
C2	0.2244 (4)	0.3402 (3)	0.1041 (2)	0.0540 (8)
H2A	0.1681	0.3659	0.0486	0.065*
H2B	0.2186	0.2591	0.1058	0.065*
C3	0.1673 (4)	0.5327 (3)	0.4086 (2)	0.0507 (8)
H3A	0.1431	0.4536	0.4034	0.061*
H3B	0.1264	0.5610	0.4653	0.061*
C4	0.3359 (4)	0.5484 (3)	0.4120 (2)	0.0557 (9)
H4A	0.3601	0.6273	0.4192	0.067*
H4B	0.3816	0.5085	0.4649	0.067*
C5	0.0887 (4)	0.7490 (3)	0.1124 (2)	0.0565 (9)
H5A	0.0391	0.7783	0.1659	0.068*
H5B	0.0411	0.7824	0.0569	0.068*
C6	0.2557 (4)	0.7787 (3)	0.1193 (2)	0.0508 (8)
H6A	0.3040	0.7535	0.0640	0.061*
H6B	0.2676	0.8594	0.1238	0.061*
N1	0.3960 (3)	0.4984 (2)	0.10299 (17)	0.0465 (6)
H1C	0.4885	0.5300	0.1105	0.070*
H1D	0.3684	0.5262	0.0466	0.070*
N2	0.1568 (3)	0.3873 (2)	0.18697 (16)	0.0419 (6)
H2C	0.1778	0.3383	0.2333	0.063*
H2D	0.0546	0.3854	0.1839	0.063*
N3	0.0993 (3)	0.5935 (2)	0.32796 (16)	0.0443 (6)
H3C	−0.0010	0.5790	0.3232	0.067*
H3D	0.1079	0.6675	0.3389	0.067*
N4	0.3974 (3)	0.5059 (2)	0.32603 (16)	0.0445 (6)
H4C	0.4947	0.5276	0.3223	0.067*
H4D	0.3872	0.4312	0.3204	0.067*
N5	0.0720 (3)	0.6272 (2)	0.10862 (16)	0.0456 (6)
H5C	−0.0218	0.6039	0.1214	0.068*
H5D	0.0933	0.6075	0.0504	0.068*
N6	0.3279 (3)	0.7250 (2)	0.20173 (16)	0.0444 (6)
H6C	0.4288	0.7342	0.1982	0.067*
H6D	0.3054	0.7678	0.2506	0.067*
O1W	0.2475 (3)	0.0426 (2)	0.21973 (17)	0.0692 (7)
H1WA	0.2630	0.1002	0.2536	0.104*
H1WB	0.2361	−0.0020	0.2649	0.104*

Atomic displacement parameters (Å²)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
Zn1	0.03116 (18)	0.0416 (2)	0.03389 (18)	−0.00316 (13)	0.00251 (12)	0.00012 (13)
Cl1	0.0520 (5)	0.0563 (5)	0.0622 (5)	0.0042 (4)	0.0079 (4)	0.0108 (4)
Cl2	0.0449 (4)	0.0597 (5)	0.0467 (4)	0.0018 (3)	0.0009 (3)	−0.0014 (4)

C1	0.0461 (18)	0.073 (2)	0.0480 (18)	0.0078 (16)	0.0089 (14)	−0.0111 (17)
C2	0.0550 (19)	0.056 (2)	0.0503 (19)	−0.0051 (16)	0.0010 (15)	−0.0111 (16)
C3	0.0565 (19)	0.061 (2)	0.0348 (15)	0.0002 (16)	0.0076 (14)	−0.0003 (14)
C4	0.0529 (19)	0.073 (2)	0.0403 (17)	0.0025 (16)	−0.0075 (14)	−0.0069 (16)
C5	0.056 (2)	0.060 (2)	0.0534 (19)	0.0148 (16)	0.0015 (15)	0.0061 (16)
C6	0.067 (2)	0.0420 (18)	0.0440 (17)	−0.0034 (15)	0.0052 (15)	0.0025 (14)
N1	0.0359 (13)	0.0597 (17)	0.0444 (14)	−0.0059 (12)	0.0072 (10)	−0.0020 (12)
N2	0.0349 (12)	0.0484 (15)	0.0425 (13)	−0.0093 (11)	0.0032 (10)	−0.0006 (11)
N3	0.0368 (13)	0.0514 (15)	0.0453 (14)	−0.0006 (11)	0.0072 (10)	−0.0015 (12)
N4	0.0354 (13)	0.0527 (16)	0.0451 (14)	−0.0013 (11)	−0.0023 (10)	−0.0031 (12)
N5	0.0357 (13)	0.0582 (17)	0.0426 (13)	−0.0029 (11)	−0.0004 (10)	0.0052 (12)
N6	0.0407 (13)	0.0467 (15)	0.0461 (14)	−0.0083 (11)	0.0042 (11)	−0.0033 (12)
O1W	0.0747 (18)	0.0651 (17)	0.0678 (16)	0.0051 (12)	0.0032 (14)	0.0022 (12)

Geometric parameters (Å, º)

Zn1—N6	2.158 (2)	C5—C6	1.513 (5)
Zn1—N2	2.167 (2)	C5—H5A	0.9700
Zn1—N5	2.196 (2)	C5—H5B	0.9700
Zn1—N1	2.203 (2)	C6—N6	1.467 (4)
Zn1—N4	2.208 (2)	C6—H6A	0.9700
Zn1—N3	2.220 (2)	C6—H6B	0.9700
C1—N1	1.464 (4)	N1—H1C	0.9000
C1—C2	1.498 (4)	N1—H1D	0.9000
C1—H1A	0.9700	N2—H2C	0.9000
C1—H1B	0.9700	N2—H2D	0.9000
C2—N2	1.472 (4)	N3—H3C	0.9000
C2—H2A	0.9700	N3—H3D	0.9000
C2—H2B	0.9700	N4—H4C	0.9000
C3—N3	1.472 (4)	N4—H4D	0.9000
C3—C4	1.496 (4)	N5—H5C	0.9000
C3—H3A	0.9700	N5—H5D	0.9000
C3—H3B	0.9700	N6—H6C	0.9000
C4—N4	1.466 (4)	N6—H6D	0.9000
C4—H4A	0.9700	O1W—H1WA	0.8500
C4—H4B	0.9700	O1W—H1WB	0.8503
C5—N5	1.463 (4)		
N6—Zn1—N2	168.95 (9)	H5A—C5—H5B	108.3
N6—Zn1—N5	80.73 (9)	N6—C6—C5	109.4 (2)
N2—Zn1—N5	92.59 (9)	N6—C6—H6A	109.8
N6—Zn1—N1	91.62 (9)	C5—C6—H6A	109.8
N2—Zn1—N1	80.16 (9)	N6—C6—H6B	109.8
N5—Zn1—N1	95.18 (9)	C5—C6—H6B	109.8
N6—Zn1—N4	94.68 (9)	H6A—C6—H6B	108.2
N2—Zn1—N4	93.19 (9)	C1—N1—Zn1	106.97 (18)
N5—Zn1—N4	170.26 (9)	C1—N1—H1C	117.9
N1—Zn1—N4	93.52 (9)	Zn1—N1—H1C	111.9

N6—Zn1—N3	93.60 (9)	C1—N1—H1D	109.7
N2—Zn1—N3	95.45 (9)	Zn1—N1—H1D	111.1
N5—Zn1—N3	92.20 (9)	H1C—N1—H1D	99.1
N1—Zn1—N3	171.56 (9)	C2—N2—Zn1	108.81 (18)
N4—Zn1—N3	79.46 (9)	C2—N2—H2C	106.0
N1—C1—C2	109.6 (3)	Zn1—N2—H2C	116.1
N1—C1—H1A	109.8	C2—N2—H2D	113.2
C2—C1—H1A	109.8	Zn1—N2—H2D	111.6
N1—C1—H1B	109.8	H2C—N2—H2D	100.9
C2—C1—H1B	109.8	C3—N3—Zn1	106.68 (18)
H1A—C1—H1B	108.2	C3—N3—H3C	109.1
N2—C2—C1	110.0 (2)	Zn1—N3—H3C	119.5
N2—C2—H2A	109.7	C3—N3—H3D	108.6
C1—C2—H2A	109.7	Zn1—N3—H3D	106.6
N2—C2—H2B	109.7	H3C—N3—H3D	106.0
C1—C2—H2B	109.7	C4—N4—Zn1	108.07 (17)
H2A—C2—H2B	108.2	C4—N4—H4C	110.2
N3—C3—C4	109.3 (3)	Zn1—N4—H4C	115.8
N3—C3—H3A	109.8	C4—N4—H4D	112.3
C4—C3—H3A	109.8	Zn1—N4—H4D	98.3
N3—C3—H3B	109.8	H4C—N4—H4D	111.7
C4—C3—H3B	109.8	C5—N5—Zn1	107.18 (17)
H3A—C3—H3B	108.3	C5—N5—H5C	113.0
N4—C4—C3	109.7 (2)	Zn1—N5—H5C	110.4
N4—C4—H4A	109.7	C5—N5—H5D	105.6
C3—C4—H4A	109.7	Zn1—N5—H5D	110.2
N4—C4—H4B	109.7	H5C—N5—H5D	110.2
C3—C4—H4B	109.7	C6—N6—Zn1	107.84 (18)
H4A—C4—H4B	108.2	C6—N6—H6C	107.0
N5—C5—C6	109.4 (2)	Zn1—N6—H6C	118.0
N5—C5—H5A	109.8	C6—N6—H6D	106.3
C6—C5—H5A	109.8	Zn1—N6—H6D	113.7
N5—C5—H5B	109.8	H6C—N6—H6D	103.4
C6—C5—H5B	109.8	H1WA—O1W—H1WB	95.1

Hydrogen-bond geometry (Å, º)

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
N1—H1*D*···Cl2^{i}	0.90	2.86	3.739 (3)	165
N2—H2*C*···Cl1	0.90	2.50	3.363 (3)	162
N2—H2*D*···Cl2^{ii}	0.90	2.48	3.332 (2)	158
N3—H3*C*···O1*W*^{iii}	0.90	2.27	3.159 (3)	169
N3—H3*D*···Cl2	0.90	2.73	3.605 (3)	165
N4—H4*C*···O1*W*^{iv}	0.90	2.39	3.260 (3)	164
N4—H4*D*···Cl1	0.90	2.52	3.375 (3)	159
N5—H5*D*···Cl2^{i}	0.90	2.57	3.420 (3)	158
N6—H6*C*···Cl1^{iv}	0.90	2.44	3.309 (3)	161
N6—H6*D*···Cl2	0.90	2.58	3.471 (3)	172

O1*W*—H1*WA*···Cl1	0.85	2.25	3.097 (3)	171
O1*W*—H1*WB*···Cl2^{v}	0.85	2.34	3.187 (3)	180

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