IUCrData

ISSN 2414-3146

(−)-Sparteinium tetrachloridozincate monohydrate

Sylvain Bernès,[a]* René Gutiérrez,[b] Guadalupe Hernández-Téllez[b] and Gloria E. Moreno[b]

[a]Instituto de Física, Universidad Autónoma de Puebla, Av. San Claudio y 18 Sur, 72570 Puebla, Pue., Mexico, and [b]Laboratorio de Síntesis de Complejos, Facultad de Ciencias Químicas, Universidad Autónoma de Puebla, A.P. 1067, 72001 Puebla, Pue., Mexico. *Correspondence e-mail: sylvain_bernes@hotmail.com

Received 14 July 2016
Accepted 31 July 2016

Edited by M. Weil, Vienna University of Technology, Austria

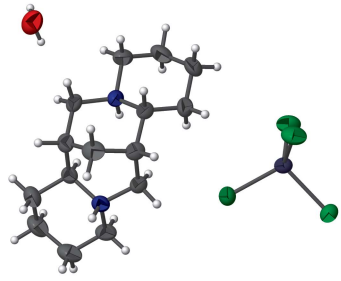
Keywords: crystal structure; sparteine; zinc; hydrate.

CCDC reference: 1497008

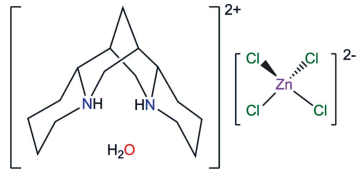
Structural data: full structural data are available from iucrdata.iucr.org

The title ionic compound, $(C_{15}H_{28}N_2)[ZnCl_4]\cdot H_2O$, is isostructural with the Cu^{II} analogue published previously [Lee *et al.* (2004). *Bull. Korean Chem. Soc.* **25**, 823–828; Jasiewicz *et al.* (2006). *J. Mol. Struct.* **794**, 311–319]. The $[ZnCl_4]^{2-}$ anion is, however, much more close to the tetrahedral conformation than the $[CuCl_4]^{2-}$ ion. In the tetrachloridozincate anion, the Cl—Zn—Cl angles are in the range 103.57 (3)–116.81 (3)°.

3D view



Chemical scheme



Structure description

The orthorhombic unit cell for the title compound $(esp)^{2+}(ZnCl_4)^{2-}\cdot H_2O$ (Fig. 1) has parameters close to those reported for the copper analogue, $(esp)^{2+}(CuCl_4)^{2-}\cdot H_2O$, and both complexes crystallize in the same chiral space group, $P2_12_12_1$ [Lee *et al.*, 2004; Jasiewicz *et al.*, 2006; CSD (Groom *et al.*, 2016) refcodes CANVOD and CANVOD01, respectively]. The ligand *esp* is sparteine, also known as lupinidine, an alkaloid having four chiral centres and two protonable N sites. For the structure reported here, (–)-sparteine was used, which has the *RSSS* configuration (Hoppe & Hense, 1997). The same arrangement of ions and lattice water in the crystal is observed with Zn^{II} and Cu^{II}. However, the tetrachloridozincate ion is almost tetrahedral, while the Cu analogue, $(CuCl_4)^{2-}$, with one electron less, is strongly distorted. For the structure reported here, the Cl—Zn—Cl angles are in the range 103.57 (3)–116.81 (3)°, while the Cl—Cu—Cl angles are in the range 97.9–135.3° (Lee *et al.*, 2004) or 97.8–135.3° (Jasiewicz *et al.*, 2006). This difference may be quantitatively estimated using the τ_4' parameter defined for four-coordinate atoms (Okuniewski *et al.*, 2015; extreme values for τ_4' are 0 for a square planar and 1 for a tetrahedral conformation). In the title complex, $\tau_4' = 0.92$ for $(ZnCl_4)^{2-}$, while in the case of CANVOD and CANVOD01, $\tau_4' = 0.69$ for $(CuCl_4)^{2-}$.

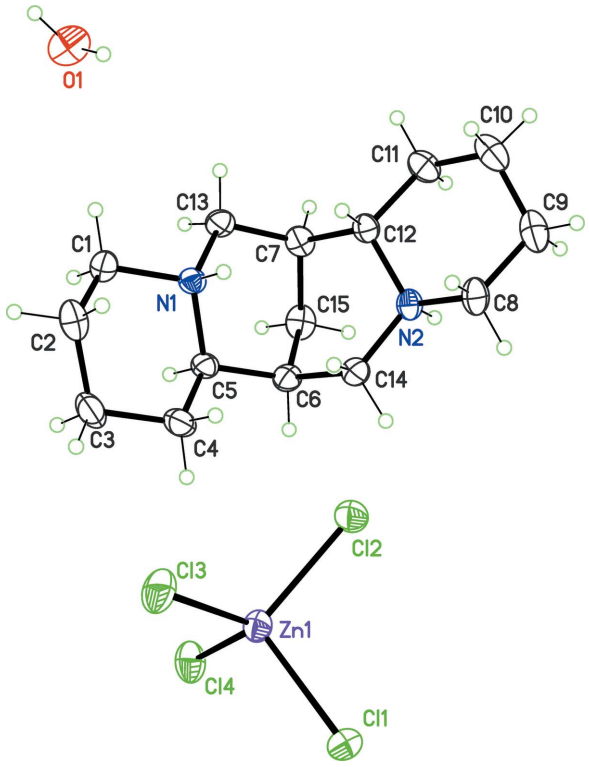


Figure 1
The structures of the molecular entities of the title compound, with displacement ellipsoids for non-H atoms drawn at the 30% probability level. The choice for the asymmetric unit, as well as the labelling scheme (including H atoms), are the same as for CANVOD (Lee *et al.*, 2004).

With Cu^{II}, the isotypic complex was synthesized with $(CuBr_4)^{2-}$ (Lee *et al.*, 2004). The last (–)-sparteinium salt for which a crystal structure has been reported was also obtained as an hydrate, with a complex heteropolyoxidometalate anion (Streb *et al.*, 2007). The dication of the α-isomer of sparteine has also been characterized, with $(CuCl_4)^{2-}$ or $(CuBr_4)^{2-}$, both crystallized as monohydrate species (Jasiewicz *et al.*, 2006).

Regardless of the $(MX_4)^{2-}$ dianion used with $(esp)^{2+}$, the inclusion of a water molecule in the lattice seems to be a stabilizing factor for the compound. For the title compound, this molecule behaves as acceptor and donor for hydrogen bonding (Table 1, entries 2–5). The crystal cohesion is completed with an N—H···Cl contact linking cations and anions (Table 1, entry 1; Fig. 2).

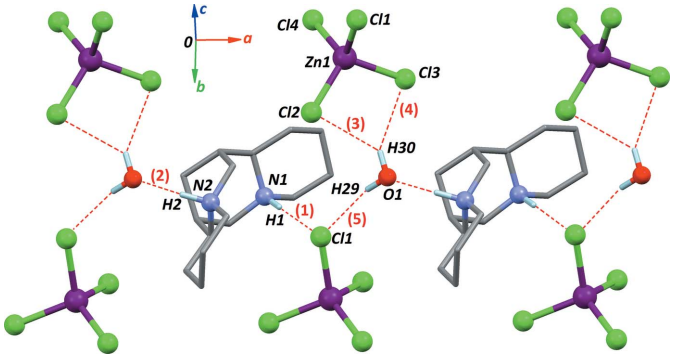


Figure 2
Part of the crystal structure, showing hydrogen bonds (dashed lines) and omitting H atoms not involved in hydrogen bonding. Hydrogen bonds are labelled (1)–(5), which correspond to entries 1–5 in Table 1.

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

D—H···A	D—H	H···A	D···A	D—H···A
N1—H1···Cl1^{i}	0.98	2.31	3.279 (2)	170
N2—H2···O1^{ii}	0.98	1.83	2.798 (3)	168
O1—H30···Cl2^{i}	0.85 (1)	2.71 (4)	3.355 (3)	133 (4)
O1—H30···Cl3^{i}	0.85 (1)	2.76 (3)	3.473 (3)	143 (4)
O1—H29···Cl1^{iii}	0.86 (1)	2.42 (1)	3.257 (3)	167 (4)

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

Table 2
Experimental details.

Crystal data	
Chemical formula	$(C_{15}H_{28}N_2)[ZnCl_4]\cdot H_2O$
M_r	461.58
Crystal system, space group	Orthorhombic, $P2_12_12_1$
Temperature (K)	297
a, b, c (Å)	8.4549 (6), 14.7691 (10), 16.3607 (9)
V (Å^3)	2043.0 (2)
Z	4
Radiation type	Mo $K\alpha$
μ (mm^{-1})	1.73
Crystal size (mm)	0.5 × 0.5 × 0.5
Data collection	
Diffractometer	Bruker P4
Absorption correction	ψ scan (*XSCANS*; Bruker, 1997)
T_{min}, T_{max}	0.210, 0.277
No. of measured, independent and observed [$I > 2\sigma(I)$] reflections	4207, 3989, 3673
R_{int}	0.017
(sin $\theta/\lambda)_{max}$ (Å^{-1})	0.703
Refinement	
$R[F^2 > 2\sigma(F^2)]$, $wR(F^2)$, S	0.027, 0.068, 1.05
No. of reflections	3989
No. of parameters	215
No. of restraints	3
H-atom treatment	H atoms treated by a mixture of independent and constrained refinement
$\Delta\rho_{max}$, $\Delta\rho_{min}$ (e Å^{-3})	0.26, −0.27
Absolute structure	Flack x determined using 593 quotients $[(I^+)-(I^-)]/[(I^+)+(I^-)]$ (Parsons *et al.*, 2013)
Absolute structure parameter	−0.004 (11)

Computer programs: *XSCANS* (Bruker, 1997), *SHELXL2014* (Sheldrick, 2015), *XP* in *SHELXTL* (Sheldrick, 2008) and *Mercury* (Macrae *et al.*, 2008).

Synthesis and crystallization

The compound was obtained as a low-yield by-product during the direct synthesis of the Zn^{II} coordination complex [Zn(esp)Cl(PhCOO)], for which the crystal structure has been reported (Alcántara-Flores *et al.*, 2009). The synthesis of this complex was carried out using equimolar amounts of zinc powder and (–)-sparteine, an excess of benzoyl chloride, and DMSO. The mixture was stirred at 338 K for 8 h, cooled, and filtered. In this kind of reaction, the oxidative dissolution of

zerovalent metals M^0 in presence of an acyl halide has been shown to afford small quantities of $(MX_4)^{2-}$, and water is provided by DMSO (Garnovskii *et al.*, 1995).

Refinement

Crystal data, data collection and structure refinement details are summarized in Table 2. The structure was refined starting from the atomic coordinates reported for CANVOD (Lee *et al.*, 2004), after substituting the Cu site by a Zn site.

Acknowledgements

SB acknowledges support by Instituto de Física Luis Rivera Terrazas (Puebla, Mexico).

References

Alcántara-Flores, J. L., Arias-López, N., Bernès, S., Gutiérrez, R. & Reyes Ortega, Y. (2009). *Acta Cryst.* E**65**, m1142–m1143.

Bruker (1997). *XSCANS*. Bruker AXS Inc., Madison, Wisconsin, USA.

Garnovskii, A. D., Kharisov, B. I., Gojon-Zorrilla, G. & Garnovskii, D. A. (1995). *Russ. Chem. Rev.* **64**, 201–221.

Groom, C. R., Bruno, I. J., Lightfoot, M. P. & Ward, S. C. (2016). *Acta Cryst.* B**72**, 171–179.

Hoppe, D. & Hense, T. (1997). *Angew. Chem. Int. Ed. Engl.* **36**, 2282–2316.

Jasiewicz, B., Boczoń, W., Muth, D., Warżajtis, B., Rychlewska, U., Andrzejewski, B. & Toliński, T. (2006). *J. Mol. Struct.* **794**, 311–319.

Lee, Y.-M., Park, S.-M., Kang, S. K., Kim, Y.-I. & Choi, S.-N. (2004). *Bull. Korean Chem. Soc.* **25**, 823–828.

Macrae, C. F., Bruno, I. J., Chisholm, J. A., Edgington, P. R., McCabe, P., Pidcock, E., Rodriguez-Monge, L., Taylor, R., van de Streek, J. & Wood, P. A. (2008). *J. Appl. Cryst.* **41**, 466–470.

Okuniewski, A., Rosiak, D., Chojnacki, J. & Becker, B. (2015). *Polyhedron*, **90**, 47–57.

Parsons, S., Flack, H. D. & Wagner, T. (2013). *Acta Cryst.* B**69**, 249–259.

Sheldrick, G. M. (2008). *Acta Cryst.* A**64**, 112–122.

Sheldrick, G. M. (2015). *Acta Cryst.* C**71**, 3–8.

Streb, C., Long, D.-L. & Cronin, L. (2007). *Chem. Commun.* pp. 471–473.

full crystallographic data

IUCrData (2016). **1**, x161237 [doi:10.1107/S2414314616012372]

(−)-Sparteinium tetrachloridozincate monohydrate

Sylvain Bernès, René Gutiérrez, Guadalupe Hernández-Téllez and Gloria E. Moreno

(-)-7,15-Diazatetracyclo[7.7.1.0^{2,7}.0^{10,15}]heptadecane-7,15-diium tetrachloridozincate monohydrate

Crystal data

$(C_{15}H_{28}N_2)[ZnCl_4]\cdot H_2O$
M_r = 461.58
Orthorhombic, $P2_12_12_1$
a = 8.4549 (6) Å
b = 14.7691 (10) Å
c = 16.3607 (9) Å
V = 2043.0 (2) Å^3
Z = 4
F(000) = 960
D_x = 1.501 Mg m^{-3}
Mo $K\alpha$ radiation, λ = 0.71073 Å
Cell parameters from 65 reflections
θ = 4.6–14.0°
μ = 1.73 mm^{-1}
T = 297 K
Prism, pale_yellow
0.5 × 0.5 × 0.5 mm

Data collection

Bruker P4 diffractometer
Radiation source: fine-focus sealed tube
Graphite monochromator
$2\theta/\omega$ scans
Absorption correction: ψ scan (XSCANS; Bruker, 1997)
T_{min} = 0.210, T_{max} = 0.277
4207 measured reflections
3989 independent reflections
3673 reflections with $I > 2\sigma(I)$
R_{int} = 0.017
θ_{max} = 30.0°, θ_{min} = 1.9°
h = −11→1
k = −20→1
l = −1→23
3 standard reflections every 97 reflections
intensity decay: 1.5%

Refinement

Refinement on F^2
Least-squares matrix: full
$R[F^2 > 2\sigma(F^2)]$ = 0.027
$wR(F^2)$ = 0.068
S = 1.05
3989 reflections
215 parameters
3 restraints
0 constraints
Primary atom site location: isomorphous structure methods
Secondary atom site location: difference Fourier map
Hydrogen site location: mixed
H atoms treated by a mixture of independent and constrained refinement
$w = 1/[\sigma^2(F_o^2) + (0.0309P)^2 + 0.3509P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} < 0.001$
$\Delta\rho_{max}$ = 0.26 e Å^{-3}
$\Delta\rho_{min}$ = −0.27 e Å^{-3}
Extinction correction: SHELXL2014 (Sheldrick, 2015), $Fc^* = kFc[1+0.001xFc^2\lambda^3/\sin(2\theta)]^{-1/4}$
Extinction coefficient: 0.0095 (6)
Absolute structure: Flack x determined using 593 quotients $[(I^+)-(I^-)]/[(I^+)+(I^-)]$ (Parsons *et al.*, 2013)
Absolute structure parameter: −0.004 (11)

Fractional atomic coordinates and isotropic or equivalent isotropic displacement parameters (Å²)

	x	*y*	*z*	U_{iso}*/U_{eq}
Zn1	0.47336 (4)	0.00375 (2)	0.13525 (2)	0.03925 (8)
Cl1	0.43251 (8)	−0.14733 (4)	0.17192 (4)	0.04092 (14)
Cl2	0.60888 (9)	0.06123 (4)	0.24235 (4)	0.04872 (16)
Cl3	0.23318 (10)	0.07286 (6)	0.13219 (6)	0.0611 (2)
Cl4	0.59037 (10)	0.01131 (6)	0.01301 (4)	0.0581 (2)
N1	0.7771 (3)	0.39420 (14)	0.16364 (12)	0.0352 (4)
H1	0.7235	0.3756	0.2140	0.042*
N2	1.0034 (3)	0.25994 (14)	0.31415 (12)	0.0378 (4)
H2	1.1143	0.2420	0.3078	0.045*
C1	0.6597 (4)	0.44897 (19)	0.11347 (17)	0.0485 (7)
H3	0.6328	0.5041	0.1426	0.058*
H4	0.7076	0.4658	0.0618	0.058*
C2	0.5127 (4)	0.3951 (3)	0.0979 (2)	0.0611 (8)
H5	0.4415	0.4299	0.0636	0.073*
H6	0.4595	0.3832	0.1493	0.073*
C3	0.5513 (5)	0.3052 (2)	0.05581 (19)	0.0605 (9)
H7	0.4561	0.2689	0.0519	0.073*
H8	0.5890	0.3169	0.0008	0.073*
C4	0.6767 (4)	0.25307 (19)	0.10303 (17)	0.0514 (7)
H9	0.6331	0.2341	0.1551	0.062*
H10	0.7050	0.1991	0.0726	0.062*
C5	0.8241 (4)	0.30901 (16)	0.11801 (14)	0.0388 (5)
H11	0.8644	0.3278	0.0645	0.047*
C6	0.9576 (4)	0.25900 (17)	0.16147 (15)	0.0403 (5)
H12	0.9900	0.2087	0.1262	0.048*
C7	1.0478 (3)	0.39856 (18)	0.22780 (15)	0.0418 (5)
H13	1.1381	0.4392	0.2355	0.050*
C8	0.9460 (4)	0.2225 (2)	0.39410 (16)	0.0497 (7)
H14	0.9533	0.1570	0.3931	0.060*
H15	0.8358	0.2388	0.4018	0.060*
C9	1.0428 (5)	0.2589 (3)	0.46439 (18)	0.0661 (9)
H16	1.1502	0.2363	0.4602	0.079*
H17	0.9986	0.2373	0.5155	0.079*
C10	1.0455 (5)	0.3613 (3)	0.46488 (18)	0.0653 (9)
H18	1.1169	0.3824	0.5072	0.078*
H19	0.9405	0.3840	0.4773	0.078*
C11	1.0988 (4)	0.3981 (2)	0.38265 (17)	0.0531 (7)
H20	1.0929	0.4637	0.3834	0.064*
H21	1.2081	0.3812	0.3733	0.064*
C12	0.9973 (3)	0.36208 (16)	0.31331 (15)	0.0374 (5)
H22	0.8878	0.3807	0.3233	0.045*
C13	0.9161 (3)	0.45208 (16)	0.18746 (15)	0.0415 (6)
H23	0.8806	0.4989	0.2247	0.050*
H24	0.9575	0.4816	0.1390	0.050*
C14	0.9123 (3)	0.21852 (17)	0.24551 (15)	0.0411 (5)

H25	0.8002	0.2278	0.2549	0.049*
H26	0.9316	0.1538	0.2447	0.049*
C15	1.0995 (3)	0.3218 (2)	0.17079 (18)	0.0481 (6)
H27	1.1309	0.3458	0.1180	0.058*
H28	1.1885	0.2893	0.1941	0.058*
O1	0.6895 (3)	0.68939 (18)	0.1878 (2)	0.0692 (7)
H29	0.615 (4)	0.728 (3)	0.191 (3)	0.104*
H30	0.663 (6)	0.652 (2)	0.225 (2)	0.104*

Atomic displacement parameters (Å²)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
Zn1	0.04187 (15)	0.03891 (14)	0.03698 (13)	0.00235 (14)	−0.00004 (12)	0.00472 (12)
Cl1	0.0427 (3)	0.0356 (2)	0.0444 (3)	0.0003 (2)	0.0092 (3)	0.0001 (2)
Cl2	0.0510 (4)	0.0450 (3)	0.0501 (3)	−0.0082 (3)	−0.0069 (3)	0.0003 (3)
Cl3	0.0533 (4)	0.0619 (4)	0.0682 (4)	0.0224 (4)	−0.0027 (4)	0.0087 (4)
Cl4	0.0641 (4)	0.0739 (5)	0.0364 (3)	0.0058 (4)	0.0041 (3)	0.0145 (3)
N1	0.0422 (10)	0.0339 (9)	0.0295 (8)	−0.0015 (9)	0.0028 (9)	−0.0006 (7)
N2	0.0337 (11)	0.0444 (10)	0.0353 (9)	−0.0008 (9)	0.0005 (9)	0.0061 (8)
C1	0.0596 (17)	0.0436 (13)	0.0423 (13)	0.0089 (14)	−0.0058 (13)	0.0032 (11)
C2	0.0538 (18)	0.077 (2)	0.0529 (16)	0.0031 (18)	−0.0186 (16)	0.0067 (15)
C3	0.072 (2)	0.0652 (18)	0.0442 (13)	−0.0149 (18)	−0.0240 (16)	0.0035 (13)
C4	0.070 (2)	0.0438 (13)	0.0408 (12)	−0.0132 (14)	−0.0098 (14)	−0.0037 (11)
C5	0.0534 (15)	0.0347 (11)	0.0284 (10)	−0.0007 (11)	0.0024 (10)	−0.0019 (8)
C6	0.0469 (14)	0.0394 (11)	0.0346 (10)	0.0054 (12)	0.0081 (11)	−0.0020 (9)
C7	0.0362 (12)	0.0465 (12)	0.0429 (12)	−0.0138 (11)	0.0015 (11)	0.0050 (10)
C8	0.0524 (17)	0.0590 (15)	0.0376 (11)	−0.0026 (15)	0.0018 (13)	0.0153 (11)
C9	0.070 (2)	0.087 (2)	0.0411 (14)	0.006 (2)	−0.0077 (16)	0.0119 (15)
C10	0.069 (2)	0.086 (2)	0.0410 (14)	−0.002 (2)	−0.0115 (16)	−0.0094 (14)
C11	0.0483 (16)	0.0615 (16)	0.0497 (15)	−0.0074 (15)	−0.0099 (13)	−0.0073 (13)
C12	0.0326 (12)	0.0416 (11)	0.0379 (11)	−0.0026 (10)	−0.0022 (10)	0.0013 (9)
C13	0.0505 (14)	0.0336 (10)	0.0405 (12)	−0.0108 (11)	0.0020 (12)	0.0034 (9)
C14	0.0476 (14)	0.0364 (11)	0.0392 (12)	−0.0018 (11)	−0.0011 (12)	0.0014 (9)
C15	0.0380 (13)	0.0614 (16)	0.0448 (13)	0.0011 (13)	0.0136 (12)	0.0105 (13)
O1	0.0514 (13)	0.0620 (14)	0.0942 (19)	−0.0091 (11)	0.0106 (15)	0.0043 (14)

Geometric parameters (Å, º)

Zn1—Cl4	2.2341 (7)	C6—C14	1.547 (3)
Zn1—Cl2	2.2592 (7)	C6—H12	0.9800
Zn1—Cl3	2.2734 (8)	C7—C13	1.516 (4)
Zn1—Cl1	2.3362 (7)	C7—C15	1.532 (4)
N1—C13	1.505 (3)	C7—C12	1.559 (3)
N1—C5	1.516 (3)	C7—H13	0.9800
N1—C1	1.521 (3)	C8—C9	1.511 (5)
N1—H1	0.9800	C8—H14	0.9700
N2—C14	1.493 (3)	C8—H15	0.9700
N2—C8	1.501 (3)	C9—C10	1.513 (5)

N2—C12	1.509 (3)	C9—H16	0.9700
N2—H2	0.9800	C9—H17	0.9700
C1—C2	1.498 (5)	C10—C11	1.519 (4)
C1—H3	0.9700	C10—H18	0.9700
C1—H4	0.9700	C10—H19	0.9700
C2—C3	1.530 (5)	C11—C12	1.518 (4)
C2—H5	0.9700	C11—H20	0.9700
C2—H6	0.9700	C11—H21	0.9700
C3—C4	1.521 (5)	C12—H22	0.9800
C3—H7	0.9700	C13—H23	0.9700
C3—H8	0.9700	C13—H24	0.9700
C4—C5	1.515 (4)	C14—H25	0.9700
C4—H9	0.9700	C14—H26	0.9700
C4—H10	0.9700	C15—H27	0.9700
C5—C6	1.525 (4)	C15—H28	0.9700
C5—H11	0.9800	O1—H29	0.855 (11)
C6—C15	1.524 (4)	O1—H30	0.851 (11)

Cl4—Zn1—Cl2	116.81 (3)	C13—C7—C12	111.7 (2)
Cl4—Zn1—Cl3	110.69 (3)	C15—C7—C12	111.6 (2)
Cl2—Zn1—Cl3	107.54 (3)	C13—C7—H13	108.0
Cl4—Zn1—Cl1	110.06 (3)	C15—C7—H13	108.0
Cl2—Zn1—Cl1	103.57 (3)	C12—C7—H13	108.0
Cl3—Zn1—Cl1	107.60 (3)	N2—C8—C9	110.9 (3)
C13—N1—C5	113.2 (2)	N2—C8—H14	109.5
C13—N1—C1	110.3 (2)	C9—C8—H14	109.5
C5—N1—C1	110.28 (19)	N2—C8—H15	109.5
C13—N1—H1	107.6	C9—C8—H15	109.5
C5—N1—H1	107.6	H14—C8—H15	108.0
C1—N1—H1	107.6	C8—C9—C10	111.6 (3)
C14—N2—C8	109.7 (2)	C8—C9—H16	109.3
C14—N2—C12	112.65 (19)	C10—C9—H16	109.3
C8—N2—C12	111.4 (2)	C8—C9—H17	109.3
C14—N2—H2	107.6	C10—C9—H17	109.3
C8—N2—H2	107.6	H16—C9—H17	108.0
C12—N2—H2	107.6	C9—C10—C11	110.9 (3)
C2—C1—N1	110.5 (2)	C9—C10—H18	109.5
C2—C1—H3	109.5	C11—C10—H18	109.5
N1—C1—H3	109.5	C9—C10—H19	109.5
C2—C1—H4	109.5	C11—C10—H19	109.5
N1—C1—H4	109.5	H18—C10—H19	108.0
H3—C1—H4	108.1	C12—C11—C10	111.7 (3)
C1—C2—C3	111.1 (3)	C12—C11—H20	109.3
C1—C2—H5	109.4	C10—C11—H20	109.3
C3—C2—H5	109.4	C12—C11—H21	109.3
C1—C2—H6	109.4	C10—C11—H21	109.3
C3—C2—H6	109.4	H20—C11—H21	107.9
H5—C2—H6	108.0	N2—C12—C11	108.9 (2)

C4—C3—C2	111.1 (2)	N2—C12—C7	110.1 (2)
C4—C3—H7	109.4	C11—C12—C7	113.3 (2)
C2—C3—H7	109.4	N2—C12—H22	108.1
C4—C3—H8	109.4	C11—C12—H22	108.1
C2—C3—H8	109.4	C7—C12—H22	108.1
H7—C3—H8	108.0	N1—C13—C7	112.95 (19)
C5—C4—C3	112.3 (2)	N1—C13—H23	109.0
C5—C4—H9	109.1	C7—C13—H23	109.0
C3—C4—H9	109.1	N1—C13—H24	109.0
C5—C4—H10	109.1	C7—C13—H24	109.0
C3—C4—H10	109.1	H23—C13—H24	107.8
H9—C4—H10	107.9	N2—C14—C6	112.5 (2)
C4—C5—N1	108.5 (2)	N2—C14—H25	109.1
C4—C5—C6	114.8 (2)	C6—C14—H25	109.1
N1—C5—C6	111.49 (19)	N2—C14—H26	109.1
C4—C5—H11	107.2	C6—C14—H26	109.1
N1—C5—H11	107.2	H25—C14—H26	107.8
C6—C5—H11	107.2	C6—C15—C7	106.6 (2)
C15—C6—C5	109.6 (2)	C6—C15—H27	110.4
C15—C6—C14	109.9 (2)	C7—C15—H27	110.4
C5—C6—C14	114.7 (2)	C6—C15—H28	110.4
C15—C6—H12	107.4	C7—C15—H28	110.4
C5—C6—H12	107.4	H27—C15—H28	108.6
C14—C6—H12	107.4	H29—O1—H30	102 (2)
C13—C7—C15	109.3 (2)		

C13—N1—C1—C2	−173.4 (2)	C8—N2—C12—C11	58.7 (3)
C5—N1—C1—C2	60.8 (3)	C14—N2—C12—C7	−52.7 (3)
N1—C1—C2—C3	−56.3 (3)	C8—N2—C12—C7	−176.5 (2)
C1—C2—C3—C4	52.9 (4)	C10—C11—C12—N2	−57.3 (3)
C2—C3—C4—C5	−54.1 (4)	C10—C11—C12—C7	179.8 (3)
C3—C4—C5—N1	57.4 (3)	C13—C7—C12—N2	119.0 (2)
C3—C4—C5—C6	−177.1 (2)	C15—C7—C12—N2	−3.8 (3)
C13—N1—C5—C4	175.8 (2)	C13—C7—C12—C11	−118.8 (3)
C1—N1—C5—C4	−60.1 (3)	C15—C7—C12—C11	118.5 (3)
C13—N1—C5—C6	48.4 (3)	C5—N1—C13—C7	−48.2 (3)
C1—N1—C5—C6	172.5 (2)	C1—N1—C13—C7	−172.3 (2)
C4—C5—C6—C15	178.6 (2)	C15—C7—C13—N1	56.2 (3)
N1—C5—C6—C15	−57.6 (3)	C12—C7—C13—N1	−67.9 (3)
C4—C5—C6—C14	−57.2 (3)	C8—N2—C14—C6	176.1 (2)
N1—C5—C6—C14	66.6 (3)	C12—N2—C14—C6	51.3 (3)
C14—N2—C8—C9	176.5 (3)	C15—C6—C14—N2	7.5 (3)
C12—N2—C8—C9	−58.0 (3)	C5—C6—C14—N2	−116.5 (3)
N2—C8—C9—C10	54.9 (4)	C5—C6—C15—C7	65.0 (3)
C8—C9—C10—C11	−53.4 (5)	C14—C6—C15—C7	−62.0 (3)
C9—C10—C11—C12	55.3 (4)	C13—C7—C15—C6	−63.8 (3)
C14—N2—C12—C11	−177.5 (2)	C12—C7—C15—C6	60.3 (3)

Hydrogen-bond geometry (Å, º)

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
N1—H1···Cl1^{i}	0.98	2.31	3.279 (2)	170
N2—H2···O1^{ii}	0.98	1.83	2.798 (3)	168
O1—H30···Cl2^{i}	0.85 (1)	2.71 (4)	3.355 (3)	133 (4)
O1—H30···Cl3^{i}	0.85 (1)	2.76 (3)	3.473 (3)	143 (4)
O1—H29···Cl1^{iii}	0.86 (1)	2.42 (1)	3.257 (3)	167 (4)

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