Acta Crystallographica Section E
**Structure Reports Online**
ISSN 1600-5368

# Bis(4,4′-methylenedicyclohexylaminium) μ-benzene-1,4-dicarboxylato-bis[trichloridozinc(II)] tetrahydrate

**Chen-Yen Hsu, Chun-Wei Yeh, Chi-Phi Wu, Chia-Her Lin* and Jhy-Der Chen**

Department of Chemistry, Chung-Yuan Christian University, Chung-Li 320, Taiwan
Correspondence e-mail: chiaher@cycu.edu.tw

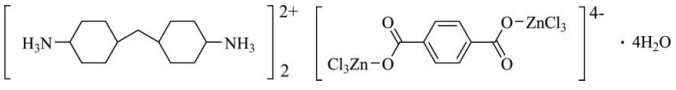
Received 15 September 2008; accepted 12 October 2008

Key indicators: single-crystal X-ray study; $T$ = 295 K; mean $\sigma$(C–C) = 0.009 Å; $R$ factor = 0.057; $wR$ factor = 0.132; data-to-parameter ratio = 16.7.

The title compound, $(C_{13}H_{28}N_2)_2[Zn_2(C_8H_4O_4)Cl_6]\cdot 4H_2O$, was prepared by the reaction of $ZnCl_2\cdot 6H_2O$, benzene-1,4-dicarboxylic acid and 4,4′-diaminodicyclohexylmethane in methanol. The $[Zn_2Cl_6(C_8H_4O_4)]^{4-}$ anions lie on centres of inversion and comprise two $ZnCl_3$ groups bridged by benzene-1,4-dicarboxylate. In addition to N—H···Cl and N—H···O hydrogen bonds between the cations and anions, solvent water molecules form O—H···O and O—H···Cl hydrogen bonds to give a three-dimensional network.

## Related literature

For related structures, see: Clausen *et al.* (2005); Thirumurugan & Rao (2005); Li *et al.* (1998, 1999).



## Experimental

### Crystal data

$(C_{13}H_{28}N_2)_2[Zn_2(C_8H_4O_4)Cl_6]\cdot 4H_2O$
$M_r$ = 1004.36
Monoclinic, $P2_1/c$
$a$ = 14.264 (3) Å
$b$ = 14.202 (2) Å
$c$ = 11.712 (2) Å
$\beta$ = 100.498 (16)°
$V$ = 2333.0 (7) Å$^3$
$Z$ = 2
Mo $K\alpha$ radiation
$\mu$ = 1.42 mm$^{-1}$
$T$ = 295 (2) K
0.70 × 0.40 × 0.10 mm

### Data collection

Bruker $P4$ diffractometer
Absorption correction: $\psi$ scan (*XSCANS*; Siemens, 1995)
$T_{min}$ = 0.694, $T_{max}$ = 0.868
5093 measured reflections
4071 independent reflections
3405 reflections with $I > 2\sigma(I)$
$R_{int}$ = 0.027
3 standard reflections
every 97 reflections
intensity decay: none

### Refinement

$R[F^2 > 2\sigma(F^2)]$ = 0.057
$wR(F^2)$ = 0.132
$S$ = 1.05
4071 reflections
244 parameters
2 restraints
H-atom parameters constrained
$\Delta\rho_{max}$ = 1.10 e Å$^{-3}$
$\Delta\rho_{min}$ = −0.83 e Å$^{-3}$

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

| $D$—H···$A$ | $D$—H | H···$A$ | $D$···$A$ | $D$—H···$A$ |
|---|---|---|---|---|
| N1—H1$A$···Cl3 | 0.89 | 2.41 | 3.252 (4) | 159 |
| N1—H1$B$···Cl2$^{i}$ | 0.89 | 2.51 | 3.290 (4) | 147 |
| N1—H1$C$···O3$^{i}$ | 0.89 | 1.94 | 2.828 (5) | 178 |
| N2—H2$A$···Cl1$^{ii}$ | 0.89 | 2.95 | 3.725 (5) | 146 |
| N2—H2$A$···Cl2$^{ii}$ | 0.89 | 2.67 | 3.321 (4) | 131 |
| N2—H2$B$···O2$^{iii}$ | 0.89 | 2.06 | 2.928 (4) | 166 |
| N2—H2$C$···O4$^{iv}$ | 0.89 | 1.95 | 2.813 (4) | 164 |
| O3—H3$B$···O2 | 1.00 | 1.81 | 2.798 (4) | 167.8 |
| O3—H3$C$···Cl1$^{v}$ | 1.06 | 2.24 | 3.262 (4) | 160.3 |
| O4—H4$B$···Cl3 | 0.98 | 2.21 | 3.172 (4) | 164.9 |
| O4—H4$C$···Cl2$^{vi}$ | 0.94 | 2.38 | 3.258 (3) | 154.9 |

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

Data collection: *XSCANS* (Siemens, 1995); cell refinement: *XSCANS*; data reduction: *SHELXTL* (Sheldrick, 2008); program(s) used to solve structure: *SHELXS97* (Sheldrick, 2008); program(s) used to refine structure: *SHELXL97* (Sheldrick, 2008); molecular graphics: *SHELXTL*; software used to prepare material for publication: *SHELXTL*.

The authors are grateful to the National Science Council of Taiwan for support. This research was also supported by the Project of the Specific Research Fields in Chung Yuan Christian University, Taiwan, under grant No. CYCU-97-CR-CH.

Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: BI2304).

## References

Clausen, H. F., Poulsen, R. D., Bond, A. D., Chevallier, M.-A. S. & Iversen, B. B. (2005). *J. Solid State Chem.* **178**, 3342–3351.
Li, H., Eddaoudi, M., Groy, T. L. & Yaghi, O. M. (1998). *J. Am. Chem. Soc.* **120**, 8571–8572.
Li, H., Eddaoudi, M., O'Keeffe, M. & Yaghi, O. M. (1999). *Nature (London)*, **402**, 276–279.
Sheldrick, G. M. (2008). *Acta Cryst.* A**64**, 112–122.
Siemens (1995). *XSCANS*. Siemens Analytical X-ray Instruments Inc., Madison, Wisconsin, USA.
Thirumurugan, A. & Rao, C. N. R. (2005). *J. Mater. Chem.* **15**, 3852–3858.

# supporting information

*Acta Cryst.* (2008). E**64**, m1412 [doi:10.1107/S1600536808033011]

## Bis(4,4′-methylenedicyclohexylaminium) $\mu$-benzene-1,4-dicarboxylato-bis-[trichloridozinc(II)] tetrahydrate

**Chen-Yen Hsu, Chun-Wei Yeh, Chi-Phi Wu, Chia-Her Lin and Jhy-Der Chen**

### S1. Comment

The dianion of benzene-1,4-dicarboxylic acid is an important linker to bridge metal atoms which show significant chemical and physical properties (Clausen *et al.*, 2005; Thirumurugan & Rao, 2005; Li *et al.*, 1998, 1999). Since the anions contain four O atoms which are good hydrogen-bond acceptors, co-crystallization with organic cations would be expected to result in extensive hydrogen-bond networks. The title compound (Fig. 1) contains N—H···Cl and N—H···O hydrogen bonds between the cations and the anions, as well as O—H···O and O—H···Cl interactions formed by the lattice water molecules.

### S2. Experimental

$ZnCl_2.6H_2O$ (0.49 g, 2.00 mmol) was added to a solution of 4,4′-diaminodicyclohexylmethane (0.21 g,1.00 mmol) and benzene-1,4-dicarboxylic acid (0.17 g, 1.00 mmol) in 30 ml MeOH. The mixture was refluxed for 24 h to yield a colorless solution with some white solid. The solution was filtered and then diethyl ether was added to induce precipitation. The precipitate was filtered and washed by ether (3 × 10 ml), then dried under reduced pressure to give a white powder. Colourless crystals were obtained by slow diffusion of ether into a methanol solution of the white powder over several weeks.

### S3. Refinement

H atoms bound to C and N atoms were placed in idealized positions and constrained to ride on their parent atoms, with C—H = 0.93–0.98 Å and N—H = 0.89 Å, and with $U_{iso}$(H) = 1.2 or 1.5$U_{eq}$(C/N). The H atoms of the water molecules were located in difference Fourier maps, then constrained to ride on their parent O atom with $U_{iso}$(H) = 1.5$U_{eq}$(O). The C8—C11 and C11—C12 bond distances were restrained to be identical with a standard uncertainty of 0.02 Å.

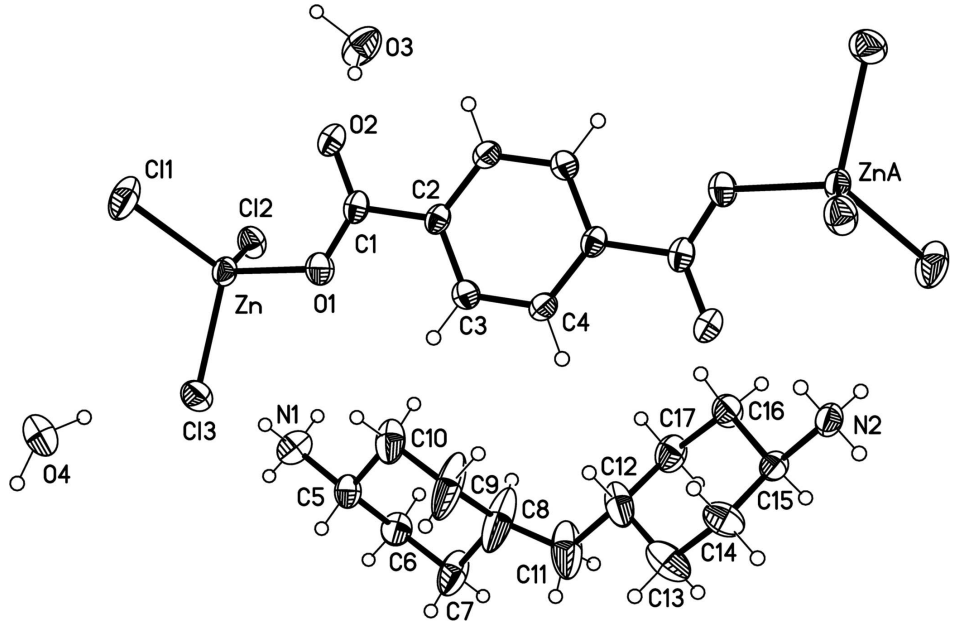



**Figure 1**

Molecular structure of the title compound with displacement ellipsoids drawn at the 30% probability level for non-H atoms. The $[Zn_2Cl_6(C_8H_4O_4)]^{4-}$ anion lies on a centre of inversion. Symmetry code: -*x*, 1 - *y*, -*z*.

**Bis(4,4′-methylenedicyclohexylaminium) *μ*-benzene-1,4-dicarboxylato-bis[trichloridozinc(II)] tetrahydrate**

*Crystal data*

$(C_{13}H_{28}N_2)_2[Zn_2(C_8H_4O_4)Cl_6]\cdot 4H_2O$
$M_r$ = 1004.36
Monoclinic, $P2_1/c$
Hall symbol: -P 2ybc
$a$ = 14.264 (3) Å
$b$ = 14.202 (2) Å
$c$ = 11.712 (2) Å
$\beta$ = 100.498 (16)°
$V$ = 2333.0 (7) Å$^3$
$Z$ = 2
$F(000)$ = 1052
$D_x$ = 1.430 Mg m$^{-3}$
Mo $K\alpha$ radiation, $\lambda$ = 0.71073 Å
Cell parameters from 33 reflections
$\theta$ = 5.7–12.5°
$\mu$ = 1.42 mm$^{-1}$
$T$ = 295 K
Plate, colourless
0.70 × 0.40 × 0.10 mm

*Data collection*

Bruker P4 diffractometer
Radiation source: fine-focus sealed tube
Graphite monochromator
$\omega$ scans
Absorption correction: $\psi$ scan (*XSCANS*; Siemens, 1995)
$T_{min}$ = 0.694, $T_{max}$ = 0.868
5093 measured reflections
4071 independent reflections
3405 reflections with $I > 2\sigma(I)$
$R_{int}$ = 0.027
$\theta_{max}$ = 25.0°, $\theta_{min}$ = 2.0°
$h$ = −16→16
$k$ = −1→16
$l$ = −1→13
3 standard reflections every 97 reflections
intensity decay: none

*Refinement*

Refinement on $F^2$
Least-squares matrix: full
$R[F^2 > 2\sigma(F^2)] = 0.057$
$wR(F^2) = 0.132$
$S = 1.05$
4071 reflections
244 parameters
2 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
$w = 1/[\sigma^2(F_o^2) + (0.0344P)^2 + 8.1458P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} < 0.001$
$\Delta\rho_{max} = 1.10$ e Å$^{-3}$
$\Delta\rho_{min} = -0.83$ 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 (Å²)*

| | *x* | *y* | *z* | $U_{iso}$*/$U_{eq}$ |
|---|---|---|---|---|
| Zn | 0.37495 (3) | 0.50908 (4) | 0.23918 (5) | 0.04083 (17) |
| Cl1 | 0.48147 (11) | 0.41661 (13) | 0.17455 (16) | 0.0807 (5) |
| Cl2 | 0.41895 (9) | 0.66231 (9) | 0.22022 (11) | 0.0522 (3) |
| Cl3 | 0.37246 (11) | 0.48512 (10) | 0.42863 (12) | 0.0611 (4) |
| O1 | 0.2401 (2) | 0.4944 (3) | 0.1703 (3) | 0.0528 (9) |
| O2 | 0.2553 (2) | 0.4968 (3) | −0.0161 (3) | 0.0515 (8) |
| O3 | 0.3060 (3) | 0.6499 (3) | −0.1424 (4) | 0.0829 (14) |
| H3B | 0.2830 | 0.5919 | −0.1077 | 0.124* |
| H3C | 0.3735 | 0.6415 | −0.1668 | 0.124* |
| O4 | 0.4724 (3) | 0.2921 (3) | 0.5161 (3) | 0.0663 (11) |
| H4B | 0.4531 | 0.3555 | 0.4871 | 0.099* |
| H4C | 0.4917 | 0.2894 | 0.5972 | 0.099* |
| N1 | 0.3214 (3) | 0.6947 (3) | 0.5109 (4) | 0.0624 (12) |
| H1A | 0.3470 | 0.6458 | 0.4802 | 0.094* |
| H1B | 0.3575 | 0.7097 | 0.5787 | 0.094* |
| H1C | 0.3179 | 0.7437 | 0.4629 | 0.094* |
| N2 | −0.3906 (3) | 0.6298 (3) | 0.0987 (4) | 0.0495 (10) |
| H2A | −0.4399 | 0.6000 | 0.1193 | 0.074* |
| H2B | −0.3577 | 0.5897 | 0.0629 | 0.074* |
| H2C | −0.4119 | 0.6769 | 0.0508 | 0.074* |
| C1 | 0.2069 (3) | 0.4959 (3) | 0.0619 (4) | 0.0392 (10) |
| C2 | 0.0992 (3) | 0.4974 (3) | 0.0298 (4) | 0.0365 (9) |
| C3 | 0.0434 (3) | 0.4915 (4) | 0.1146 (4) | 0.0447 (11) |
| H3A | 0.0724 | 0.4857 | 0.1922 | 0.054* |
| C4 | −0.0549 (3) | 0.4940 (3) | 0.0853 (4) | 0.0425 (11) |

| | | | | |
|---|---|---|---|---|
| H4A | −0.0915 | 0.4900 | 0.1432 | 0.051* |
| C5 | 0.2238 (3) | 0.6694 (4) | 0.5292 (5) | 0.0494 (12) |
| H5A | 0.2286 | 0.6161 | 0.5831 | 0.059* |
| C6 | 0.1788 (4) | 0.7515 (4) | 0.5812 (5) | 0.0599 (14) |
| H6A | 0.2171 | 0.7677 | 0.6558 | 0.072* |
| H6B | 0.1766 | 0.8058 | 0.5306 | 0.072* |
| C7 | 0.0792 (4) | 0.7268 (6) | 0.5970 (6) | 0.087 (2) |
| H7A | 0.0504 | 0.7796 | 0.6301 | 0.104* |
| H7B | 0.0806 | 0.6733 | 0.6487 | 0.104* |
| C8 | 0.0217 (4) | 0.7026 (8) | 0.4758 (9) | 0.153 (5) |
| H8A | 0.0323 | 0.7544 | 0.4244 | 0.183* |
| C9 | 0.0658 (4) | 0.6144 (7) | 0.4308 (9) | 0.144 (5) |
| H9A | 0.0680 | 0.5633 | 0.4862 | 0.173* |
| H9B | 0.0278 | 0.5943 | 0.3576 | 0.173* |
| C10 | 0.1653 (4) | 0.6397 (5) | 0.4146 (6) | 0.084 (2) |
| H10A | 0.1949 | 0.5858 | 0.3845 | 0.101* |
| H10B | 0.1626 | 0.6907 | 0.3590 | 0.101* |
| C11 | −0.0777 (4) | 0.6972 (7) | 0.4707 (7) | 0.129 (4) |
| H11A | −0.0985 | 0.7584 | 0.4932 | 0.154* |
| H11B | −0.0886 | 0.6529 | 0.5299 | 0.154* |
| C12 | −0.1428 (5) | 0.6698 (5) | 0.3598 (5) | 0.082 (2) |
| H12A | −0.1106 | 0.6224 | 0.3200 | 0.099* |
| C13 | −0.2310 (6) | 0.6253 (5) | 0.3937 (6) | 0.086 (2) |
| H13A | −0.2119 | 0.5736 | 0.4470 | 0.104* |
| H13B | −0.2637 | 0.6716 | 0.4331 | 0.104* |
| C14 | −0.2979 (5) | 0.5892 (4) | 0.2885 (5) | 0.0648 (16) |
| H14A | −0.2665 | 0.5405 | 0.2512 | 0.078* |
| H14B | −0.3538 | 0.5619 | 0.3119 | 0.078* |
| C15 | −0.3274 (3) | 0.6681 (3) | 0.2049 (4) | 0.0448 (11) |
| H15A | −0.3640 | 0.7138 | 0.2416 | 0.054* |
| C16 | −0.2440 (4) | 0.7187 (4) | 0.1693 (5) | 0.0568 (14) |
| H16A | −0.2669 | 0.7726 | 0.1214 | 0.068* |
| H16B | −0.2118 | 0.6767 | 0.1237 | 0.068* |
| C17 | −0.1739 (4) | 0.7515 (4) | 0.2759 (5) | 0.0657 (16) |
| H17A | −0.2035 | 0.8001 | 0.3155 | 0.079* |
| H17B | −0.1182 | 0.7786 | 0.2516 | 0.079* |

*Atomic displacement parameters (Å²)*

| | $U^{11}$ | $U^{22}$ | $U^{33}$ | $U^{12}$ | $U^{13}$ | $U^{23}$ |
|---|---|---|---|---|---|---|
| Zn | 0.0279 (3) | 0.0487 (3) | 0.0448 (3) | 0.0002 (2) | 0.0038 (2) | −0.0039 (2) |
| Cl1 | 0.0513 (8) | 0.0921 (12) | 0.0964 (12) | 0.0202 (8) | 0.0077 (8) | −0.0412 (10) |
| Cl2 | 0.0443 (6) | 0.0524 (7) | 0.0572 (7) | −0.0055 (5) | 0.0023 (5) | 0.0085 (6) |
| Cl3 | 0.0727 (9) | 0.0612 (8) | 0.0493 (7) | 0.0159 (7) | 0.0108 (6) | 0.0109 (6) |
| O1 | 0.0318 (16) | 0.074 (2) | 0.050 (2) | −0.0063 (17) | −0.0002 (14) | −0.0007 (18) |
| O2 | 0.0348 (17) | 0.063 (2) | 0.060 (2) | 0.0050 (16) | 0.0154 (15) | 0.0004 (17) |
| O3 | 0.086 (3) | 0.063 (3) | 0.112 (4) | 0.007 (2) | 0.051 (3) | 0.012 (2) |
| O4 | 0.074 (3) | 0.052 (2) | 0.065 (2) | 0.0020 (19) | −0.010 (2) | 0.0026 (18) |

| | | | | | | |
|---|---|---|---|---|---|---|
| N1 | 0.059 (3) | 0.057 (3) | 0.074 (3) | −0.014 (2) | 0.020 (2) | −0.018 (2) |
| N2 | 0.034 (2) | 0.059 (3) | 0.056 (2) | −0.0009 (19) | 0.0076 (18) | −0.004 (2) |
| C1 | 0.032 (2) | 0.030 (2) | 0.055 (3) | 0.0004 (18) | 0.006 (2) | −0.005 (2) |
| C2 | 0.028 (2) | 0.035 (2) | 0.046 (2) | −0.0022 (18) | 0.0072 (18) | −0.0064 (19) |
| C3 | 0.036 (2) | 0.059 (3) | 0.038 (2) | −0.001 (2) | 0.0023 (19) | −0.002 (2) |
| C4 | 0.035 (2) | 0.054 (3) | 0.040 (2) | 0.000 (2) | 0.0110 (19) | −0.003 (2) |
| C5 | 0.041 (3) | 0.044 (3) | 0.060 (3) | −0.001 (2) | 0.001 (2) | −0.006 (2) |
| C6 | 0.057 (3) | 0.064 (3) | 0.056 (3) | 0.001 (3) | 0.003 (3) | −0.022 (3) |
| C7 | 0.042 (3) | 0.116 (6) | 0.097 (5) | 0.002 (3) | −0.001 (3) | −0.063 (5) |
| C8 | 0.044 (4) | 0.219 (11) | 0.181 (9) | 0.025 (5) | −0.015 (5) | −0.162 (9) |
| C9 | 0.038 (3) | 0.181 (9) | 0.198 (10) | 0.017 (5) | −0.018 (5) | −0.149 (8) |
| C10 | 0.065 (4) | 0.097 (5) | 0.081 (4) | 0.030 (4) | −0.013 (3) | −0.049 (4) |
| C11 | 0.084 (5) | 0.167 (9) | 0.113 (6) | 0.057 (6) | −0.042 (5) | −0.090 (6) |
| C12 | 0.077 (4) | 0.090 (5) | 0.066 (4) | 0.044 (4) | −0.024 (3) | −0.038 (4) |
| C13 | 0.120 (6) | 0.074 (4) | 0.054 (4) | −0.001 (4) | −0.013 (4) | −0.001 (3) |
| C14 | 0.089 (4) | 0.052 (3) | 0.049 (3) | −0.010 (3) | 0.003 (3) | −0.001 (3) |
| C15 | 0.040 (2) | 0.048 (3) | 0.047 (3) | 0.000 (2) | 0.011 (2) | −0.004 (2) |
| C16 | 0.046 (3) | 0.063 (3) | 0.059 (3) | −0.011 (3) | 0.005 (2) | 0.012 (3) |
| C17 | 0.046 (3) | 0.071 (4) | 0.079 (4) | −0.011 (3) | 0.009 (3) | −0.011 (3) |

*Geometric parameters (Å, º)*

| | | | |
|---|---|---|---|
| Zn—O1 | 1.956 (3) | C7—C8 | 1.543 (9) |
| Zn—Cl1 | 2.2418 (15) | C7—H7A | 0.97 |
| Zn—Cl3 | 2.2514 (15) | C7—H7B | 0.97 |
| Zn—Cl2 | 2.2869 (14) | C8—C11 | 1.410 (7) |
| O1—C1 | 1.272 (6) | C8—C9 | 1.537 (10) |
| O2—C1 | 1.243 (6) | C8—H8A | 0.98 |
| O3—H3B | 1.00 | C9—C10 | 1.510 (11) |
| O3—H3C | 1.06 | C9—H9A | 0.97 |
| O4—H4B | 0.98 | C9—H9B | 0.97 |
| O4—H4C | 0.94 | C10—H10A | 0.97 |
| N1—C5 | 1.491 (6) | C10—H10B | 0.97 |
| N1—H1A | 0.89 | C11—C12 | 1.503 (6) |
| N1—H1B | 0.89 | C11—H11A | 0.97 |
| N1—H1C | 0.89 | C11—H11B | 0.97 |
| N2—C15 | 1.500 (6) | C12—C13 | 1.524 (11) |
| N2—H2A | 0.89 | C12—C17 | 1.534 (9) |
| N2—H2B | 0.89 | C12—H12A | 0.98 |
| N2—H2C | 0.89 | C13—C14 | 1.503 (8) |
| C1—C2 | 1.513 (6) | C13—H13A | 0.97 |
| C2—C3 | 1.385 (6) | C13—H13B | 0.97 |
| C2—C4$^{i}$ | 1.386 (6) | C14—C15 | 1.497 (7) |
| C3—C4 | 1.382 (6) | C14—H14A | 0.97 |
| C3—H3A | 0.93 | C14—H14B | 0.97 |
| C4—C2$^{i}$ | 1.386 (6) | C15—C16 | 1.512 (7) |
| C4—H4A | 0.93 | C15—H15A | 0.98 |
| C5—C10 | 1.505 (7) | C16—C17 | 1.523 (8) |

| | | | |
|---|---|---|---|
| C5—C6 | 1.512 (7) | C16—H16A | 0.97 |
| C5—H5A | 0.98 | C16—H16B | 0.97 |
| C6—C7 | 1.506 (8) | C17—H17A | 0.97 |
| C6—H6A | 0.97 | C17—H17B | 0.97 |
| C6—H6B | 0.97 | | |

| | | | |
|---|---|---|---|
| O1—Zn—Cl1 | 118.31 (12) | C7—C8—H8A | 106.4 |
| O1—Zn—Cl3 | 101.50 (11) | C10—C9—C8 | 107.6 (7) |
| Cl1—Zn—Cl3 | 112.19 (7) | C10—C9—H9A | 110.2 |
| O1—Zn—Cl2 | 109.14 (12) | C8—C9—H9A | 110.2 |
| Cl1—Zn—Cl2 | 108.05 (7) | C10—C9—H9B | 110.2 |
| Cl3—Zn—Cl2 | 107.06 (6) | C8—C9—H9B | 110.2 |
| C1—O1—Zn | 124.7 (3) | H9A—C9—H9B | 108.5 |
| H3B—O3—H3C | 113.5 | C5—C10—C9 | 109.6 (6) |
| H4B—O4—H4C | 113.5 | C5—C10—H10A | 109.8 |
| C5—N1—H1A | 109.5 | C9—C10—H10A | 109.8 |
| C5—N1—H1B | 109.5 | C5—C10—H10B | 109.8 |
| H1A—N1—H1B | 109.5 | C9—C10—H10B | 109.8 |
| C5—N1—H1C | 109.5 | H10A—C10—H10B | 108.2 |
| H1A—N1—H1C | 109.5 | C8—C11—C12 | 120.6 (7) |
| H1B—N1—H1C | 109.5 | C8—C11—H11A | 107.2 |
| C15—N2—H2A | 109.5 | C12—C11—H11A | 107.2 |
| C15—N2—H2B | 109.5 | C8—C11—H11B | 107.2 |
| H2A—N2—H2B | 109.5 | C12—C11—H11B | 107.2 |
| C15—N2—H2C | 109.5 | H11A—C11—H11B | 106.8 |
| H2A—N2—H2C | 109.5 | C11—C12—C13 | 107.0 (6) |
| H2B—N2—H2C | 109.5 | C11—C12—C17 | 114.7 (7) |
| O2—C1—O1 | 125.4 (4) | C13—C12—C17 | 109.0 (5) |
| O2—C1—C2 | 119.5 (4) | C11—C12—H12A | 108.6 |
| O1—C1—C2 | 115.2 (4) | C13—C12—H12A | 108.6 |
| C3—C2—C4$^{i}$ | 118.9 (4) | C17—C12—H12A | 108.6 |
| C3—C2—C1 | 120.7 (4) | C14—C13—C12 | 111.0 (6) |
| C4$^{i}$—C2—C1 | 120.4 (4) | C14—C13—H13A | 109.4 |
| C4—C3—C2 | 120.7 (4) | C12—C13—H13A | 109.4 |
| C4—C3—H3A | 119.7 | C14—C13—H13B | 109.4 |
| C2—C3—H3A | 119.7 | C12—C13—H13B | 109.4 |
| C3—C4—C2$^{i}$ | 120.4 (4) | H13A—C13—H13B | 108.0 |
| C3—C4—H4A | 119.8 | C15—C14—C13 | 110.0 (5) |
| C2$^{i}$—C4—H4A | 119.8 | C15—C14—H14A | 109.7 |
| N1—C5—C10 | 108.6 (5) | C13—C14—H14A | 109.7 |
| N1—C5—C6 | 110.4 (4) | C15—C14—H14B | 109.7 |
| C10—C5—C6 | 111.6 (4) | C13—C14—H14B | 109.7 |
| N1—C5—H5A | 108.7 | H14A—C14—H14B | 108.2 |
| C10—C5—H5A | 108.7 | C14—C15—N2 | 109.0 (4) |
| C6—C5—H5A | 108.7 | C14—C15—C16 | 113.2 (4) |
| C7—C6—C5 | 110.4 (5) | N2—C15—C16 | 109.2 (4) |
| C7—C6—H6A | 109.6 | C14—C15—H15A | 108.4 |
| C5—C6—H6A | 109.6 | N2—C15—H15A | 108.4 |

| | | | |
|---|---|---|---|
| C7—C6—H6B | 109.6 | C16—C15—H15A | 108.4 |
| C5—C6—H6B | 109.6 | C15—C16—C17 | 110.6 (5) |
| H6A—C6—H6B | 108.1 | C15—C16—H16A | 109.5 |
| C6—C7—C8 | 107.2 (6) | C17—C16—H16A | 109.5 |
| C6—C7—H7A | 110.3 | C15—C16—H16B | 109.5 |
| C8—C7—H7A | 110.3 | C17—C16—H16B | 109.5 |
| C6—C7—H7B | 110.3 | H16A—C16—H16B | 108.1 |
| C8—C7—H7B | 110.3 | C16—C17—C12 | 111.3 (5) |
| H7A—C7—H7B | 108.5 | C16—C17—H17A | 109.4 |
| C11—C8—C9 | 114.4 (7) | C12—C17—H17A | 109.4 |
| C11—C8—C7 | 114.4 (7) | C16—C17—H17B | 109.4 |
| C9—C8—C7 | 108.3 (7) | C12—C17—H17B | 109.4 |
| C11—C8—H8A | 106.4 | H17A—C17—H17B | 108.0 |
| C9—C8—H8A | 106.4 | | |

Symmetry code: (i) $-x, -y+1, -z$.

*Hydrogen-bond geometry (Å, º)*

| *D*—H···*A* | *D*—H | H···*A* | *D*···*A* | *D*—H···*A* |
|---|---|---|---|---|
| N1—H1*A*···Cl3 | 0.89 | 2.41 | 3.252 (4) | 159 |
| N1—H1*B*···Cl2$^{ii}$ | 0.89 | 2.51 | 3.290 (4) | 147 |
| N1—H1*C*···O3$^{ii}$ | 0.89 | 1.94 | 2.828 (5) | 178 |
| N2—H2*A*···Cl1$^{iii}$ | 0.89 | 2.95 | 3.725 (5) | 146 |
| N2—H2*A*···Cl2$^{iii}$ | 0.89 | 2.67 | 3.321 (4) | 131 |
| N2—H2*B*···O2$^{i}$ | 0.89 | 2.06 | 2.928 (4) | 166 |
| N2—H2*C*···O4$^{iv}$ | 0.89 | 1.95 | 2.813 (4) | 164 |
| O3—H3*B*···O2 | 1.00 | 1.81 | 2.798 (4) | 167.8 |
| O3—H3*C*···Cl1$^{v}$ | 1.06 | 2.24 | 3.262 (4) | 160.3 |
| O4—H4*B*···Cl3 | 0.98 | 2.21 | 3.172 (4) | 164.9 |
| O4—H4*C*···Cl2$^{vi}$ | 0.94 | 2.38 | 3.258 (3) | 154.9 |

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