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1,1′-(Butane-1,4-diyl)diimidazole-3,3′-diium tetrachloridozincate(II) dihydrate

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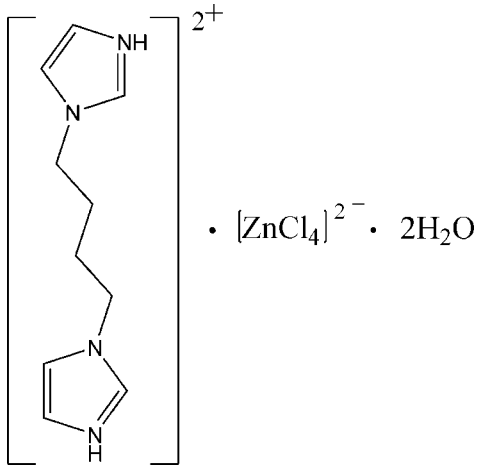
Key indicators: single-crystal X-ray study; T = 291 K; mean σ(C–C) = 0.003 Å; R factor = 0.033; wR factor = 0.081; data-to-parameter ratio = 20.2.

In the title compound, $(C_{10}H_{16}N_4)[ZnCl_4]\cdot 2H_2O$, the cation lies abouton a center of inversion and the anion about a twofold rotation axis. The Zn^{II} atom is four-coordinate in a tetrahedral environment. The cations, anions and water molecules are linked by N—H···O, N—H···Cl and O—H···Cl hydrogen bonds into a two-dimensional network.

Related literature

For background and the synthesis of 1,1′-(1,4-butanediyl)diimidazole, see: Ma *et al.* (2003)



Experimental

Crystal data

$(C_{10}H_{16}N_4)[ZnCl_4]\cdot 2H_2O$
M_r = 435.47
Monoclinic, $P2/n$
a = 7.4010 (15) Å
b = 10.927 (2) Å
c = 11.058 (2) Å
β = 95.23 (3)°
V = 890.6 (3) Å^3
Z = 2
Mo $K\alpha$ radiation
μ = 1.99 mm^{-1}
T = 291 (2) K
0.18 × 0.17 × 0.15 mm

Data collection

Rigaku R-AXIS RAPID diffractometer
Absorption correction: multi-scan (*ABSCOR*; Higashi, 1995)
T_{min} = 0.713, T_{max} = 0.751
8575 measured reflections
2042 independent reflections
1760 reflections with $I > 2\sigma(I)$
R_{int} = 0.042

Refinement

$R[F^2 > 2\sigma(F^2)]$ = 0.032
$wR(F^2)$ = 0.081
S = 1.07
2042 reflections
101 parameters
H atoms treated by a mixture of independent and constrained refinement
$\Delta\rho_{max}$ = 0.47 e Å^{-3}
$\Delta\rho_{min}$ = −0.37 e Å^{-3}

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

D—H···A	D—H	H···A	D···A	D—H···A
O1—H10···Cl3^{i}	0.85	2.43	3.275 (2)	177
O1—H9···Cl2^{ii}	0.85	2.52	3.337 (3)	161
N2—H3···Cl2^{i}	0.85 (3)	2.82 (3)	3.350 (2)	122 (3)
N2—H3···O1	0.85 (3)	2.15 (3)	2.890 (3)	145 (3)

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

Data collection: *RAPID-AUTO* (Rigaku, 1998); cell refinement: *RAPID-AUTO*; data reduction: *CrystalStructure* (Rigaku/MSC, 2002); 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: *SHELXL97*.

The authors thank Heilongjiang University for supporting this study.

Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: NG2438).

References

Higashi, T. (1995). *ABSCOR*. Rigaku Corporation, Tokyo, Japan.
Ma, J.-F., Yang, J., Zheng, G.-L. & Liu, J.-F. (2003). *Inorg. Chem.* **42**, 7531–7534.
Rigaku (1998). *RAPID-AUTO*. Rigaku Corporation, Tokyo, Japan.
Rigaku/MSC (2002). *CrystalStructure*. Rigaku/MSC Inc., The Woodlands, Texas, USA.
Sheldrick, G. M. (2008). *Acta Cryst.* A**64**, 112–122.

supporting information

Acta Cryst. (2008). E**64**, m628 [doi:10.1107/S160053680800874X]

1,1′-(Butane-1,4-diyl)diimidazole-3,3′-diium tetrachloridozincate(II) dihydrate

Ying-Hui Yu, Ai-E Shi, Yu Su, Guang-Feng Hou and Jin-Sheng Gao

S1. Comment

The 1,1′-(1,4-butanediyl)diimidazole can be used as a flexible ligand to construct coordination polymer materials(Ma *et al.*., 2003). In our attempt to synthesize the zinc complex with the 1,1′-(1,4-butanediyl)diimidazole, we unexpectedly obtained the title compound (I). Herein, we report its crystal structure.

The Zn^{II} atom lies on an inversion center and is coordinated by four chlorine anions in an tetrahedronal environment(Figure 1). The 1,1′-(1,4-butanediyl)diimidazole molecule also lies on an inversion center and its N atom is protonated.

In the crystal structure, the cations and anions are linked by N—H···Cl hydrogen bonds. In addition, the water molecules are both as acceptor and donor of hydrogen bond link these molecule into a two-dimensional supramolecular network *via* N—H···O, O—H···Cl hydrogen bonds (Table 1; Figure 2).

S2. Experimental

1,1′-(1,4-Butanediyl)diimidazole was prepared of imidazole and 1,4-dibromobutane in dimethylsulfoxide solution (Ma *et al.*., 2003). $ZnCl_2$ (0.272 g, 2 mmol) and 1,1′-(1,4-butanediyl)diimidazole (0.380 g, 2 mmol) were dissolved in hot methanol solution (15 ml) and added two drops hydrochloric acid then a clear solution was obtained. The resulting solution was allowed to stand in a desiccator at room temperature for several days. Colroless crystals of (I) were obtained. Unexpectedly, the salt-type adducts of this ligands was crystallized from solution.

S3. Refinement

H atoms bound to C atoms were placed in calculated positions and treated as riding on their parent atoms, with C—H = 0.93 Å (Caromatic) and with U_{iso}(H) = 1.2U_{eq}(C). The N-bound H atoms were located in a difference Fourier map and free refined, Water H atoms were initially located in a difference Fourier map but they were treated as riding on their parent atoms with O—H = 0.85 Å, H···H = 1.39 and with U_{iso}(H) = 1.5U_{eq}(O).

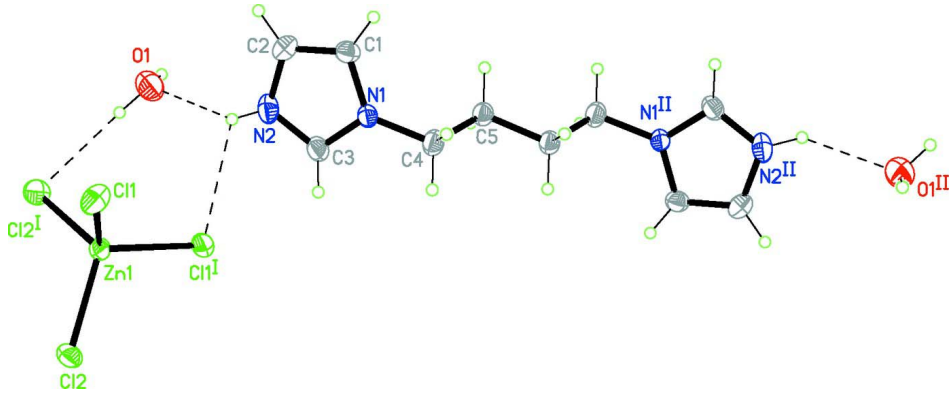


Figure 1

The molecular structure of (I), showing displacement ellipsoids at the 30% probability level for non-H atoms. Dashed lines indicate the intramolecular hydrogen bonding interactions.

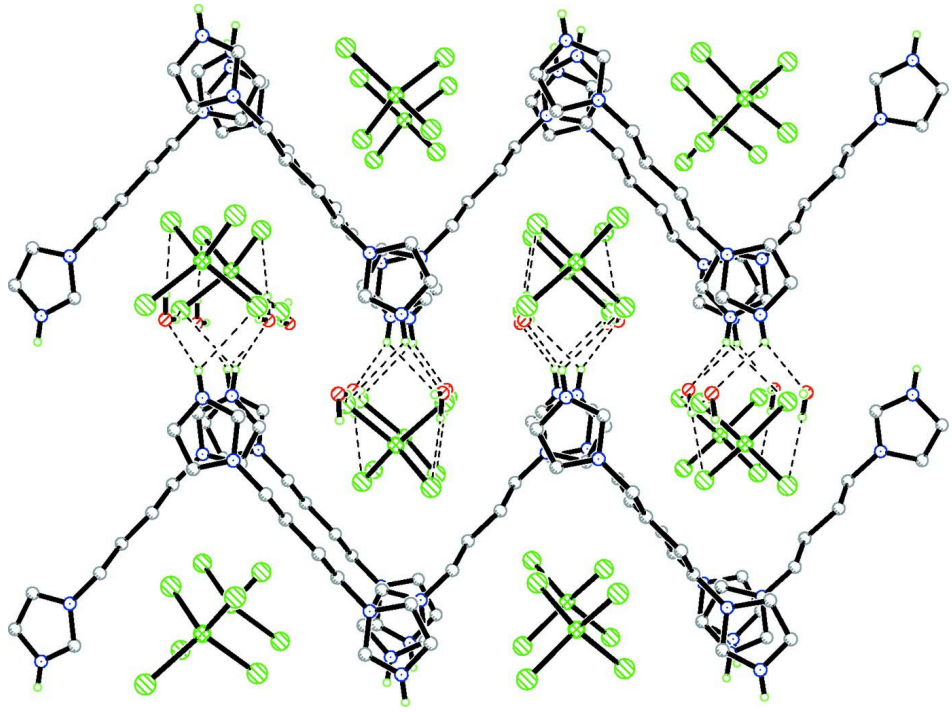


Figure 2

A partial packing view, showing the two-dimensional hydrogen-bonding network. Dashed lines indicate the hydrogen-bonding interactions. H atoms not involved in hydrogen bonds have been omitted for clarity.

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$c = 11.058\ (2)$ Å
$\beta = 95.23\ (3)°$
$V = 890.6\ (3)$ Å^3
$Z = 2$
$F(000) = 444$
$D_x = 1.624$ Mg m^{-3}

Mo $K\alpha$ radiation, λ = 0.71073 Å
Cell parameters from 6883 reflections
θ = 3.2–27.5°
μ = 1.99 mm^{-1}
T = 291 K
Block, colorless
0.18 × 0.17 × 0.15 mm

Data collection

Rigaku R-AXIS RAPID diffractometer
Radiation source: fine-focus sealed tube
Graphite monochromator
ω scans
Absorption correction: multi-scan (*ABSCOR*; Higashi, 1995)
T_{min} = 0.713, T_{max} = 0.751
8575 measured reflections
2042 independent reflections
1760 reflections with $I > 2\sigma(I)$
R_{int} = 0.042
θ_{max} = 27.5°, θ_{min} = 3.2°
h = −9→9
k = −14→14
l = −14→14

Refinement

Refinement on F^2
Least-squares matrix: full
$R[F^2 > 2\sigma(F^2)]$ = 0.032
$wR(F^2)$ = 0.081
S = 1.07
2042 reflections
101 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 atoms treated by a mixture of independent and constrained refinement
$w = 1/[\sigma^2(F_o^2) + (0.0301P)^2 + 0.5173P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max}$ = 0.001
$\Delta\rho_{max}$ = 0.47 e Å^{-3}
$\Delta\rho_{min}$ = −0.37 e Å^{-3}
Extinction correction: *SHELXL97* (Sheldrick, 2008), $Fc^*=kFc[1+0.001xFc^2\lambda^3/\sin(2\theta)]^{-1/4}$
Extinction coefficient: 0.038 (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	0.7500	0.75494 (3)	0.2500	0.03710 (15)
C1	0.4713 (3)	0.1878 (2)	0.2017 (2)	0.0405 (5)
H1	0.4499	0.1104	0.1684	0.049*
C2	0.4320 (4)	0.2946 (2)	0.1464 (2)	0.0469 (6)
H2	0.3790	0.3056	0.0676	0.056*
C3	0.5555 (4)	0.3335 (2)	0.3300 (2)	0.0448 (6)
H4	0.6019	0.3752	0.3993	0.054*
C4	0.6136 (3)	0.1221 (2)	0.4085 (2)	0.0450 (6)
H5	0.6771	0.1641	0.4770	0.054*
H6	0.6995	0.0680	0.3742	0.054*
C5	0.4626 (3)	0.0464 (2)	0.4529 (2)	0.0376 (5)

H7	0.3985	0.0040	0.3849	0.045*
H8	0.3770	0.0997	0.4885	0.045*
Cl2	0.87983 (10)	0.63431 (6)	0.11389 (5)	0.0543 (2)
Cl3	0.96871 (9)	0.86758 (5)	0.35304 (6)	0.0529 (2)
H3	0.475 (4)	0.461 (3)	0.216 (3)	0.066 (9)*
N1	0.5489 (2)	0.21294 (16)	0.31649 (16)	0.0347 (4)
N2	0.4846 (3)	0.3841 (2)	0.2278 (2)	0.0499 (5)
O1	0.3280 (3)	0.6019 (2)	0.1132 (2)	0.0775 (7)
H9	0.2192	0.6042	0.1319	0.116*
H10	0.3814	0.6708	0.1191	0.116*

Atomic displacement parameters (Å²)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
Zn1	0.0465 (2)	0.0277 (2)	0.0365 (2)	0.000	0.00102 (15)	0.000
C1	0.0413 (12)	0.0380 (12)	0.0412 (12)	0.0001 (10)	−0.0009 (10)	−0.0065 (10)
C2	0.0495 (14)	0.0545 (14)	0.0359 (12)	0.0036 (12)	0.0001 (10)	0.0056 (11)
C3	0.0631 (16)	0.0326 (11)	0.0393 (12)	−0.0061 (10)	0.0085 (11)	−0.0020 (10)
C4	0.0403 (13)	0.0434 (13)	0.0499 (13)	−0.0007 (10)	−0.0029 (10)	0.0146 (11)
C5	0.0362 (11)	0.0354 (11)	0.0408 (12)	0.0017 (9)	0.0019 (9)	0.0055 (10)
Cl2	0.0739 (5)	0.0480 (4)	0.0405 (3)	0.0215 (3)	0.0029 (3)	−0.0024 (3)
Cl3	0.0563 (4)	0.0355 (3)	0.0639 (4)	−0.0094 (3)	−0.0101 (3)	0.0011 (3)
N1	0.0374 (10)	0.0297 (8)	0.0371 (9)	−0.0009 (7)	0.0030 (8)	0.0036 (7)
N2	0.0659 (15)	0.0330 (10)	0.0522 (12)	0.0040 (10)	0.0135 (10)	0.0086 (9)
O1	0.0740 (15)	0.0611 (13)	0.0940 (17)	−0.0160 (11)	−0.0113 (12)	0.0114 (12)

Geometric parameters (Å, º)

Zn1—Cl3	2.2577 (8)	C3—H4	0.9300
Zn1—Cl3^{i}	2.2577 (8)	C4—N1	1.470 (3)
Zn1—Cl2	2.2782 (8)	C4—C5	1.508 (3)
Zn1—Cl2^{i}	2.2782 (7)	C4—H5	0.9700
C1—C2	1.337 (3)	C4—H6	0.9700
C1—N1	1.372 (3)	C5—C5^{ii}	1.521 (4)
C1—H1	0.9300	C5—H7	0.9700
C2—N2	1.362 (3)	C5—H8	0.9700
C2—H2	0.9300	N2—H3	0.85 (3)
C3—N2	1.322 (3)	O1—H9	0.8500
C3—N1	1.326 (3)	O1—H10	0.8501
Cl3—Zn1—Cl3^{i}	113.93 (4)	C5—C4—H5	109.0
Cl3—Zn1—Cl2	108.83 (3)	N1—C4—H6	109.0
Cl3^{i}—Zn1—Cl2	107.95 (3)	C5—C4—H6	109.0
Cl3—Zn1—Cl2^{i}	107.95 (3)	H5—C4—H6	107.8
Cl3^{i}—Zn1—Cl2^{i}	108.83 (3)	C4—C5—C5^{ii}	110.7 (2)
Cl2—Zn1—Cl2^{i}	109.29 (4)	C4—C5—H7	109.5
C2—C1—N1	107.7 (2)	C5^{ii}—C5—H7	109.5
C2—C1—H1	126.2	C4—C5—H8	109.5

N1—C1—H1	126.2	C5^{ii}—C5—H8	109.5
C1—C2—N2	106.7 (2)	H7—C5—H8	108.1
C1—C2—H2	126.7	C3—N1—C1	108.14 (19)
N2—C2—H2	126.7	C3—N1—C4	125.9 (2)
N2—C3—N1	108.1 (2)	C1—N1—C4	125.95 (19)
N2—C3—H4	125.9	C3—N2—C2	109.4 (2)
N1—C3—H4	125.9	C3—N2—H3	125 (2)
N1—C4—C5	113.02 (19)	C2—N2—H3	126 (2)
N1—C4—H5	109.0	H9—O1—H10	113.5

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

Hydrogen-bond geometry (Å, º)

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
O1—H10···Cl3^{i}	0.85	2.43	3.275 (2)	177
O1—H9···Cl2^{iii}	0.85	2.52	3.337 (3)	161
N2—H3···Cl2^{i}	0.85 (3)	2.82 (3)	3.350 (2)	122 (3)
N2—H3···O1	0.85 (3)	2.15 (3)	2.890 (3)	145 (3)

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