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Crystal structure of 2,2′-bipyridine-1,1′-diium tetrachloridozincate

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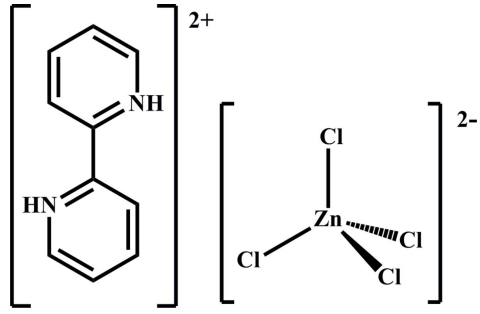
In the crystal structure of the title salt, $(C_{10}H_{10}N_2)[ZnCl_4]$, the bipyridinediium dication is not planar, with a dihedral angle of 37.21 (9)° between the planes of the two pyridine rings. In the crystal, the slightly distorted $[ZnCl_4]^{2-}$ anions are packed into rods parallel to [001], with the organic cations arranged in corrugated layers parallel to (100). Cations and anions are linked through N—H···Cl hydrogen bonds, forming chains parallel to $[20\bar{1}]$. Additional C—H···Cl interactions consolidate the crystal packing.

Keywords: crystal structure; 2,2′-bipyridine-1,1′-diium; tetrachloridozincate; hydrogen bonding.

CCDC reference: 1049571

1. Related literature

For the crystal structure of 4,4′-bipyridine-1,1′-diium tetrachloridozincate, see: Gillon *et al.* (2000). For other bipyridine derivatives with a $[ZnCl_4]^{2-}$ counter-anion, see: Rice *et al.* (2002).



2. Experimental

2.1. Crystal data

$(C_{10}H_{10}N_2)[ZnCl_4]$
M_r = 365.39
Monoclinic, $P2_1/c$
a = 7.1059 (4) Å
b = 13.6075 (6) Å
c = 14.2631 (7) Å
β = 100.816 (5)°
V = 1354.65 (12) Å^3
Z = 4
Mo $K\alpha$ radiation
μ = 2.58 mm^{-1}
T = 293 K
0.25 × 0.20 × 0.18 mm

2.2. Data collection

Oxford Diffraction Xcalibur diffractometer with an Eos detector
Absorption correction: multi-scan (*CrysAlis PRO*; Oxford Diffraction, 2009)
T_{min} = 0.565, T_{max} = 0.654
7435 measured reflections
3115 independent reflections
2717 reflections with $I > 2\sigma(I)$
R_{int} = 0.024

2.3. Refinement

$R[F^2 > 2\sigma(F^2)]$ = 0.025
$wR(F^2)$ = 0.056
S = 1.05
3115 reflections
154 parameters
H-atom parameters constrained
$\Delta\rho_{max}$ = 0.49 e Å^{-3}
$\Delta\rho_{min}$ = −0.51 e Å^{-3}

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

D—H···A	D—H	H···A	D···A	D—H···A
N1—H1A···Cl4^{i}	0.86	2.33	3.1058 (15)	150
N2—H2A···Cl1^{ii}	0.86	2.26	3.0693 (15)	157
C1—H1···Cl2^{iii}	0.93	2.74	3.4842 (19)	137
C3—H3···Cl4^{iv}	0.93	2.83	3.664 (2)	150
C10—H10···Cl2^{v}	0.93	2.67	3.570 (2)	162

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

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); software used to prepare material for publication: *SHELXL97* and *PLATON* (Spek, 2009).

Acknowledgements

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Supporting information for this paper is available from the IUCr electronic archives (Reference: WM5125).

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Acta Cryst. (2015). E**71**, m67–m68 [doi:10.1107/S2056989015003175]

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S1. Experimental

Zinc chloride (136 mg, 1 mmol) was dissolved in 20 ml of water. To this solution was added dropwise 2,2′-bipyridine (156 mg, 1 mmol) in 20 ml of an EtOH/HCl mixture (1:9 *v*/*v*). The mixture was heated to 333 K for 2–3 hrs and allowed to stand until colorless crystals separated. The crystals were filtered and repeatedly recrystallized by using acidified water.

S2. Refinement

N and C-bound H atoms were positioned geometrically (N—H = 0.86; C—H = 0.93 Å) and allowed to ride on their parent atoms, with U_{iso}(H) = 1.2U_{eq}(N, C).

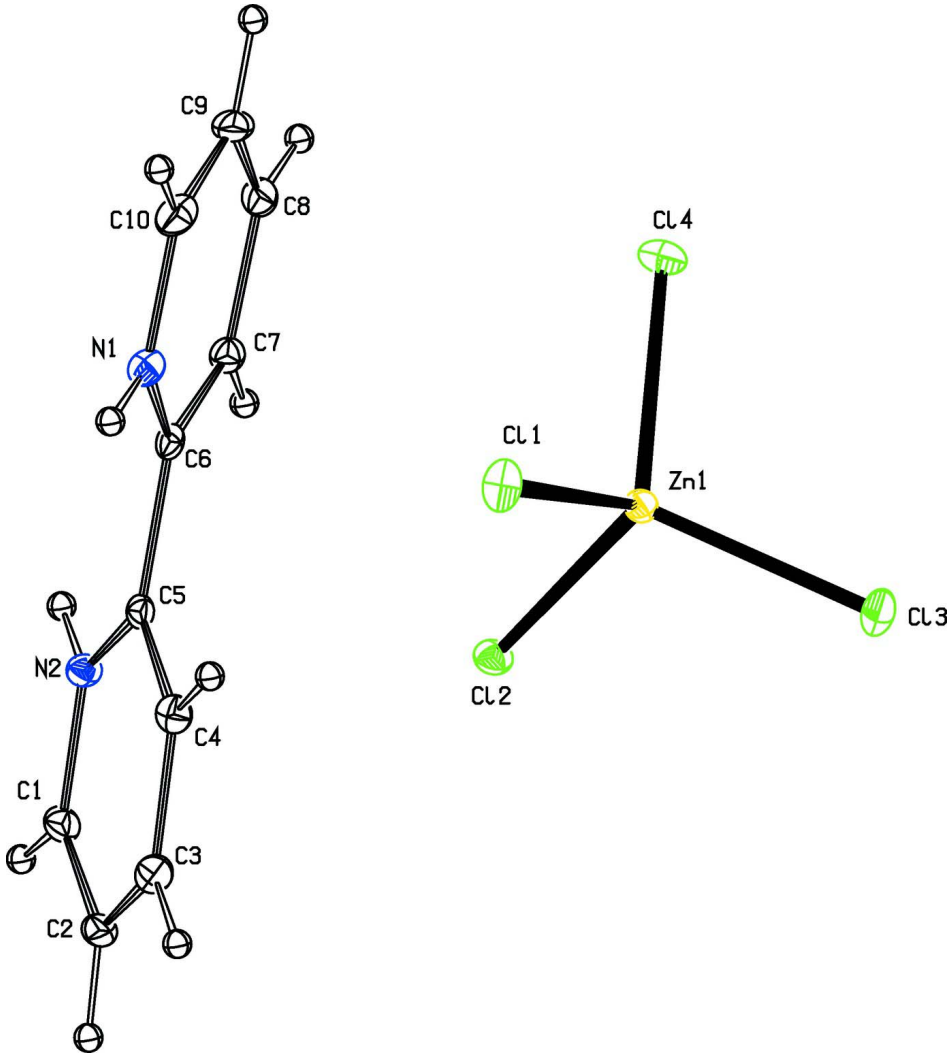

Figure 1

The molecular components of the title salt with displacement ellipsoids drawn at the 30% probability level.

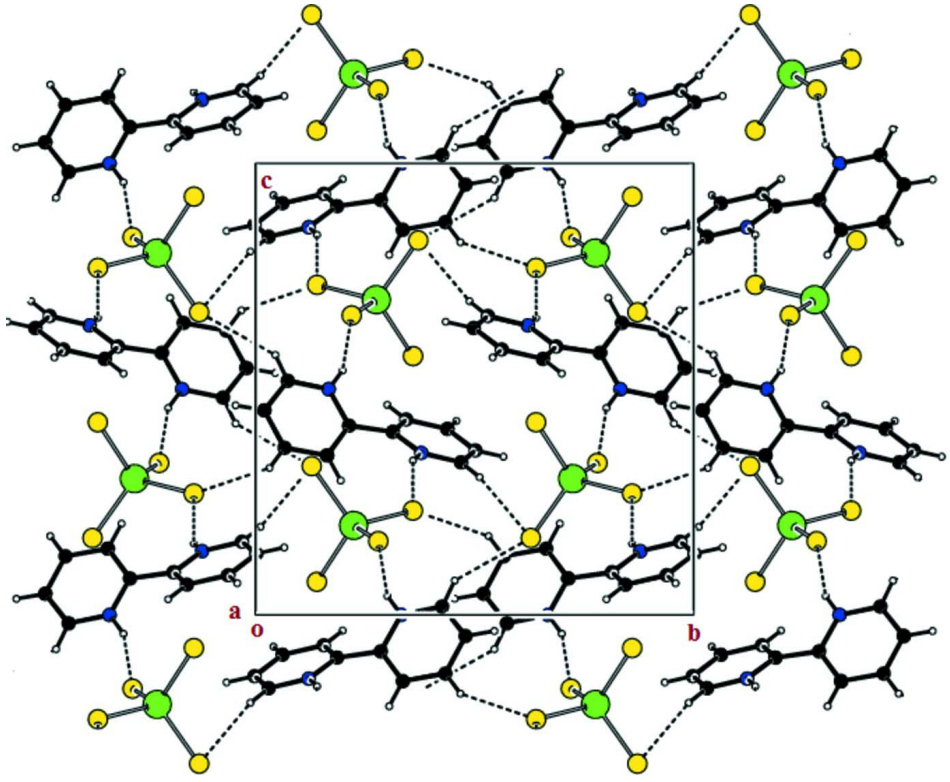

Figure 2

The crystal packing of the title compound viewed along [100].

2,2′-Bipyridine-1,1′-diium tetrachloridozincate

Crystal data

$(C_{10}H_{10}N_2)[ZnCl_4]$
$M_r = 365.39$
Monoclinic, $P2_1/c$
Hall symbol: -p 2ybc
$a = 7.1059$ (4) Å
$b = 13.6075$ (6) Å
$c = 14.2631$ (7) Å
$\beta = 100.816$ (5)°
$V = 1354.65$ (12) Å^3
$Z = 4$
$F(000) = 728$
$D_x = 1.791$ Mg m^{-3}
Mo $K\alpha$ radiation, $\lambda = 0.71073$ Å
Cell parameters from 2717 reflections
$\theta = 3.7$–29.2°
$\mu = 2.58$ mm^{-1}
$T = 293$ K
Block, colourless
0.25 × 0.20 × 0.18 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)
$T_{min} = 0.565$, $T_{max} = 0.654$
7435 measured reflections
3115 independent reflections
2717 reflections with $I > 2\sigma(I)$
$R_{int} = 0.024$
$\theta_{max} = 29.2°$, $\theta_{min} = 3.7°$
$h = -9 \rightarrow 9$
$k = -17 \rightarrow 17$
$l = -18 \rightarrow 17$

Refinement

Refinement on F^2
Least-squares matrix: full
$R[F^2 > 2\sigma(F^2)] = 0.025$
$wR(F^2) = 0.056$
$S = 1.05$
3115 reflections
154 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
$w = 1/[\sigma^2(F_o^2) + (0.0235P)^2 + 0.1003P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} < 0.001$
$\Delta\rho_{max} = 0.49$ e Å^{-3}
$\Delta\rho_{min} = -0.51$ 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}
C1	0.6920 (3)	0.07397 (14)	0.51855 (13)	0.0181 (4)
H1	0.6776	0.0453	0.5759	0.022*
C2	0.7677 (3)	0.02053 (14)	0.45317 (14)	0.0200 (4)
H2	0.8001	−0.0452	0.4643	0.024*
C3	0.7947 (3)	0.06655 (14)	0.37010 (14)	0.0192 (4)
H3	0.8492	0.0321	0.3256	0.023*
C4	0.7409 (3)	0.16360 (14)	0.35321 (13)	0.0164 (4)
H4	0.7598	0.1947	0.2977	0.020*
C5	0.6590 (3)	0.21376 (13)	0.41919 (12)	0.0130 (4)
C6	0.5805 (3)	0.31365 (14)	0.40546 (12)	0.0132 (4)
C7	0.4136 (3)	0.34390 (14)	0.43280 (12)	0.0159 (4)
H7	0.3465	0.3010	0.4652	0.019*
C8	0.3461 (3)	0.43815 (15)	0.41188 (13)	0.0205 (4)
H8	0.2330	0.4586	0.4297	0.025*
C9	0.4473 (3)	0.50196 (14)	0.36437 (13)	0.0234 (4)
H9	0.4041	0.5658	0.3506	0.028*
C10	0.6122 (3)	0.46959 (14)	0.33786 (13)	0.0225 (4)
H10	0.6811	0.5114	0.3052	0.027*
N1	0.6743 (2)	0.37817 (11)	0.35883 (10)	0.0163 (3)
H1A	0.7786	0.3595	0.3419	0.020*
N2	0.6388 (2)	0.16722 (11)	0.50012 (10)	0.0147 (3)
H2A	0.5896	0.1990	0.5417	0.018*
Zn1	0.14622 (3)	0.224086 (15)	0.198218 (14)	0.01454 (7)
Cl1	0.43041 (7)	0.28035 (4)	0.16341 (3)	0.02205 (12)
Cl2	0.22538 (7)	0.12838 (3)	0.32961 (3)	0.01744 (10)

Cl3	−0.01698 (8)	0.14035 (4)	0.07333 (4)	0.03026 (13)
Cl4	−0.01829 (7)	0.35933 (3)	0.23116 (3)	0.02098 (11)

Atomic displacement parameters (Å²)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
C1	0.0177 (10)	0.0151 (9)	0.0199 (10)	−0.0006 (8)	−0.0003 (8)	0.0045 (7)
C2	0.0127 (10)	0.0140 (9)	0.0310 (11)	0.0012 (8)	−0.0017 (8)	−0.0010 (8)
C3	0.0121 (10)	0.0210 (10)	0.0250 (10)	0.0014 (8)	0.0050 (8)	−0.0068 (8)
C4	0.0136 (10)	0.0189 (10)	0.0174 (9)	−0.0007 (8)	0.0047 (7)	−0.0003 (7)
C5	0.0104 (9)	0.0139 (9)	0.0138 (9)	−0.0023 (7)	−0.0002 (7)	0.0003 (7)
C6	0.0160 (10)	0.0146 (9)	0.0081 (8)	−0.0026 (7)	0.0002 (7)	−0.0004 (7)
C7	0.0175 (10)	0.0158 (9)	0.0141 (9)	−0.0002 (8)	0.0027 (7)	−0.0009 (7)
C8	0.0213 (11)	0.0214 (10)	0.0172 (10)	0.0039 (8)	−0.0007 (8)	−0.0045 (8)
C9	0.0370 (13)	0.0130 (9)	0.0170 (10)	0.0050 (9)	−0.0032 (8)	0.0000 (8)
C10	0.0350 (12)	0.0164 (10)	0.0148 (10)	−0.0064 (9)	0.0015 (8)	0.0023 (7)
N1	0.0186 (9)	0.0158 (8)	0.0151 (8)	−0.0026 (7)	0.0044 (6)	−0.0005 (6)
N2	0.0171 (8)	0.0141 (8)	0.0132 (8)	0.0017 (6)	0.0035 (6)	−0.0009 (6)
Zn1	0.01358 (12)	0.01320 (12)	0.01649 (12)	−0.00066 (8)	0.00192 (8)	0.00128 (8)
Cl1	0.0188 (3)	0.0326 (3)	0.0162 (2)	−0.0072 (2)	0.00697 (18)	−0.00060 (19)
Cl2	0.0193 (2)	0.0146 (2)	0.0184 (2)	0.00016 (18)	0.00339 (17)	0.00387 (17)
Cl3	0.0305 (3)	0.0245 (3)	0.0295 (3)	−0.0056 (2)	−0.0106 (2)	−0.0036 (2)
Cl4	0.0207 (3)	0.0164 (2)	0.0278 (3)	0.00419 (19)	0.0097 (2)	0.00458 (19)

Geometric parameters (Å, º)

C1—N2	1.336 (2)	C7—H7	0.9300
C1—C2	1.370 (3)	C8—C9	1.383 (3)
C1—H1	0.9300	C8—H8	0.9300
C2—C3	1.385 (3)	C9—C10	1.370 (3)
C2—H2	0.9300	C9—H9	0.9300
C3—C4	1.383 (3)	C10—N1	1.335 (2)
C3—H3	0.9300	C10—H10	0.9300
C4—C5	1.377 (2)	N1—H1A	0.8600
C4—H4	0.9300	N2—H2A	0.8600
C5—N2	1.348 (2)	Zn1—Cl3	2.2452 (5)
C5—C6	1.468 (2)	Zn1—Cl2	2.2647 (5)
C6—N1	1.350 (2)	Zn1—Cl4	2.2760 (5)
C6—C7	1.379 (2)	Zn1—Cl1	2.2994 (5)
C7—C8	1.382 (3)		
N2—C1—C2	120.20 (17)	C7—C8—C9	119.83 (18)
N2—C1—H1	119.9	C7—C8—H8	120.1
C2—C1—H1	119.9	C9—C8—H8	120.1
C1—C2—C3	118.53 (18)	C10—C9—C8	118.92 (18)
C1—C2—H2	120.7	C10—C9—H9	120.5
C3—C2—H2	120.7	C8—C9—H9	120.5
C4—C3—C2	120.15 (17)	N1—C10—C9	120.09 (18)

C4—C3—H3	119.9	N1—C10—H10	120.0
C2—C3—H3	119.9	C9—C10—H10	120.0
C5—C4—C3	119.53 (17)	C10—N1—C6	122.94 (17)
C5—C4—H4	120.2	C10—N1—H1A	118.5
C3—C4—H4	120.2	C6—N1—H1A	118.5
N2—C5—C4	118.61 (16)	C1—N2—C5	122.91 (16)
N2—C5—C6	116.77 (15)	C1—N2—H2A	118.5
C4—C5—C6	124.51 (16)	C5—N2—H2A	118.5
N1—C6—C7	118.37 (17)	Cl3—Zn1—Cl2	112.08 (2)
N1—C6—C5	117.22 (16)	Cl3—Zn1—Cl4	111.43 (2)
C7—C6—C5	124.33 (16)	Cl2—Zn1—Cl4	110.585 (18)
C6—C7—C8	119.84 (18)	Cl3—Zn1—Cl1	109.97 (2)
C6—C7—H7	120.1	Cl2—Zn1—Cl1	106.16 (2)
C8—C7—H7	120.1	Cl4—Zn1—Cl1	106.33 (2)

N2—C1—C2—C3	2.6 (3)	C5—C6—C7—C8	−176.39 (17)
C1—C2—C3—C4	−1.8 (3)	C6—C7—C8—C9	−0.6 (3)
C2—C3—C4—C5	−0.5 (3)	C7—C8—C9—C10	0.8 (3)
C3—C4—C5—N2	1.9 (3)	C8—C9—C10—N1	−0.7 (3)
C3—C4—C5—C6	−174.26 (18)	C9—C10—N1—C6	0.5 (3)
N2—C5—C6—N1	146.62 (17)	C7—C6—N1—C10	−0.2 (3)
C4—C5—C6—N1	−37.1 (3)	C5—C6—N1—C10	176.69 (16)
N2—C5—C6—C7	−36.7 (3)	C2—C1—N2—C5	−1.2 (3)
C4—C5—C6—C7	139.56 (19)	C4—C5—N2—C1	−1.1 (3)
N1—C6—C7—C8	0.3 (3)	C6—C5—N2—C1	175.37 (17)

Hydrogen-bond geometry (Å, º)

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
N1—H1*A*···Cl4^{i}	0.86	2.33	3.1058 (15)	150
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