

1-(2,5-Dimethylphenyl)piperazine-1,4-dium tetrachloridozincate monohydrate

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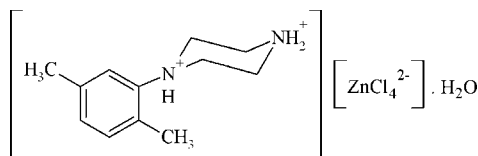
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Key indicators: single-crystal X-ray study; $T = 100$ K; mean $\sigma(\text{C}-\text{C}) = 0.002$ Å; R factor = 0.027; wR factor = 0.064; data-to-parameter ratio = 25.7.

In the title compound, $(\text{C}_{12}\text{H}_{20}\text{N}_2)[\text{ZnCl}_4]\cdot\text{H}_2\text{O}$, the two piperazine N atoms are protonated and the $[\text{ZnCl}_4]^{2-}$ anions adopt a slightly distorted tetrahedral configuration. In the crystal, $\text{O}-\text{H}\cdots\text{Cl}$ hydrogen bonds link the tetrachloridozincate anions and the water molecules into corrugated inorganic chains parallel to $[010]$. The crystal structure is stabilized by $\text{N}-\text{H}\cdots\text{Cl}$, $\text{N}-\text{H}\cdots\text{O}$ and $\text{O}-\text{H}\cdots\text{Cl}$ hydrogen bonds, with the $\text{N}-\text{H}$ hydrogen bond originating from one of the two N atoms being trifurcated.

Related literature

For common applications of organic–inorganic hybrid materials, see: Dai *et al.* (2002); Tao *et al.* (2003). For a related structure and discussion of geometrical features, see: Ben Gharbia *et al.* (2007). For the geometry around the zinc atom, see: Harrison (2005).



Experimental

Crystal data

$(\text{C}_{12}\text{H}_{20}\text{N}_2)[\text{ZnCl}_4]\cdot\text{H}_2\text{O}$

$M_r = 417.49$

Monoclinic, $P2_1/c$
 $a = 7.0999$ (8) Å
 $b = 8.0679$ (8) Å
 $c = 29.933$ (3) Å
 $\beta = 95.314$ (2)°
 $V = 1707.2$ (3) Å³

$Z = 4$
 Mo $K\alpha$ radiation
 $\mu = 2.06$ mm⁻¹
 $T = 100$ K
 $0.45 \times 0.39 \times 0.31$ mm

Data collection

Bruker SMART APEX CCD diffractometer
 Absorption correction: multi-scan (SADABS; Bruker, 2009)
 $T_{\min} = 0.589$, $T_{\max} = 0.746$

10440 measured reflections
 4995 independent reflections
 4738 reflections with $I > 2\sigma(I)$
 $R_{\text{int}} = 0.016$

Refinement

$R[F^2 > 2\sigma(F^2)] = 0.027$
 $wR(F^2) = 0.064$
 $S = 1.13$
 4995 reflections
 194 parameters
 3 restraints

H atoms treated by a mixture of independent and constrained refinement
 $\Delta\rho_{\text{max}} = 0.56$ e Å⁻³
 $\Delta\rho_{\text{min}} = -0.44$ e Å⁻³

Table 1

Hydrogen-bond geometry (Å, °).

$D-\text{H}\cdots A$	$D-\text{H}$	$\text{H}\cdots A$	$D\cdots A$	$D-\text{H}\cdots A$
$\text{N1}-\text{H1A}\cdots\text{Cl3}^{\text{i}}$	0.90 (2)	2.46 (2)	3.2300 (14)	144 (2)
$\text{N2}-\text{H2A}\cdots\text{O1}$	0.92	1.92	2.7925 (17)	157
$\text{N2}-\text{H2B}\cdots\text{O1}^{\text{ii}}$	0.92	2.19	2.9145 (17)	135
$\text{N2}-\text{H2B}\cdots\text{Cl4}^{\text{ii}}$	0.92	2.77	3.3965 (14)	127
$\text{N2}-\text{H2B}\cdots\text{Cl1}^{\text{iii}}$	0.92	2.85	3.4036 (14)	120
$\text{O1}-\text{H1C}\cdots\text{Cl2}$	0.83 (2)	2.43 (2)	3.2361 (12)	164 (2)
$\text{O1}-\text{H1B}\cdots\text{Cl4}^{\text{iii}}$	0.84 (2)	2.31 (2)	3.1518 (13)	178 (3)

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

Data collection: *APEX2* (Bruker, 2009); cell refinement: *SAINT* (Bruker, 2009); data reduction: *SAINT*; program(s) used to solve structure: *SHELXTL* (Sheldrick, 2008); program(s) used to refine structure: *SHELXTL*; molecular graphics: *SHELXTL*; 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: VN2004).

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supplementary materials

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1-(2,5-Dimethylphenyl)piperazine-1,4-dium tetrachloridozincate monohydrate

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Comment

Organic-inorganic hybrid materials continue to attract much attention due to their potential applications in various fields (Dai *et al.*, 2002; Tao *et al.*, 2003). In this work, we report the crystal structure of one such compound, [1-(2,5-(CH₃)₂C₆H₃)C₄H₁₁N₂]ZnCl₄.H₂O. As shown in Fig. 1, the asymmetric unit consists of one 1-(2,5-dimethylphenyl)piperazine-1,4-dium dication doubly protonated at the N1 and N2 nitrogen atoms, one water molecule and one [ZnCl₄]²⁻ anion. The atomic arrangement of [1-(2,5-(CH₃)₂C₆H₃)C₄H₁₁N₂]ZnCl₄.H₂O can be described as built up by corrugated inorganic chains of [ZnCl₄]²⁻ tetrahedra and water molecules that extend along the *b* axis, held together by O—H···Cl hydrogen bonds (Fig. 2, Table 1). Two such chains cross the unit cell at $z = (2n + 1)/4$ and $x = 1/2$. The organic groups are located between these chains and connect to them through N—H···Cl and N—H···O hydrogen bonds to form a three dimensional infinite network (Fig. 3, Table 1). Among all the hydrogen bonds, one is trifurcated: N2—H2B···(Cl1, Cl4, O1). Within the network, the 1-(2,5-dimethylphenyl)piperazine-1,4-dium dications are arranged into antiparallel dimers. No π - π stacking interactions between the phenylene rings or C—H··· π interactions towards them are observed. In the organic entity, the piperazine-1,4-dium ring adopts a typical chair conformation and all the geometrical features agree with those found in 1-(2,3-dimethylphenyl)piperazinium tetrachlorozincate(II) monohydrate (Ben Gharbia *et al.*, 2007). It is worth noticing that in the [ZnCl₄]²⁻ anion, the Zn—Cl bond lengths and Cl—Zn—Cl bond angles are not equal to one another, but vary with the environment around the Cl atom with Zn—Cl bond lengths between 2.2619 (4) and 2.2857 (4) Å. In the title compound, all the chloride ions are involved in hydrogen bonding. However, only the Cl4 chloride atom participates in two N—H···Cl bonds, and the Zn1—Cl4 bond distance is with 2.2857 (4) Å the longest (Table 1). The Cl—Zn—Cl angles range between 105.803 (16) and 112.737 (17)°. These values indicate that the coordination geometry of the Zn atom can be regarded as being a slightly distorted tetrahedron (Harrison, 2005).

Experimental

A mixture of an aqueous solution of 1-(2,5-dimethylphenyl)piperazine (2 mmol, 0.380 g), zinc chloride (2 mmol, 0.396 g) and HCl (10 ml, 0.4 M) in a Petri dish was slowly evaporated at room temperature. Single crystals of the title compound, suitable for X-ray diffraction analysis, were obtained after several days by slow evaporation at room temperature (yield 68%).

Refinement

Reflection (0 0 1) was obscured by the beamstop and was omitted from the refinement. C—H hydrogen atoms were placed in calculated positions with C—H distances in the range 0.93–0.97 Å. The water hydrogen atom positions were refined with O—H distance restraints of 0.84 (2) Å, and the N—H distance of N1 to 0.91 (2) Å. The $U_{\text{iso}}(\text{H})$ values of all H atoms were constrained to 1.2 or 1.5 times U_{eq} of the respective parent atom.

Figures

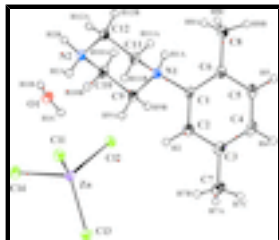


Fig. 1. A view of the title compound, showing 50% probability displacement ellipsoids, arbitrary spheres for the H atoms, and the atom numbering scheme.

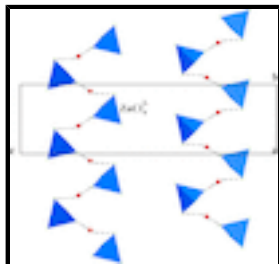


Fig. 2. Projection along the *a* axis of the inorganic corrugated chain in the structure of the title compound. Hydrogen bonds are denoted as dashed lines.

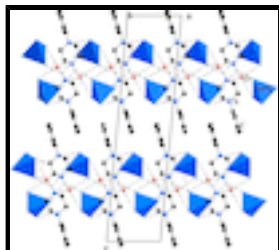


Fig. 3. The packing of [1-(2,5-(CH₃)₂C₆H₃)C₄H₁₁N₂]ZnCl₄.H₂O, viewed down the *b* axis. Hydrogen bonds are denoted by dashed lines.

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Crystal data

(C₁₂H₂₀N₂)[ZnCl₄]·H₂O

M_r = 417.49

Monoclinic, *P*2₁/*c*

Hall symbol: -*P* 2ybc

a = 7.0999 (8) Å

b = 8.0679 (8) Å

c = 29.933 (3) Å

β = 95.314 (2)°

V = 1707.2 (3) Å³

Z = 4

F(000) = 856

D_x = 1.624 Mg m⁻³

Mo *K*α radiation, λ = 0.71073 Å

Cell parameters from 6435 reflections

θ = 2.6–31.0°

μ = 2.06 mm⁻¹

T = 100 K

Block, colourless

0.45 × 0.39 × 0.31 mm

Data collection

Bruker SMART APEX CCD
diffractometer

Radiation source: fine-focus sealed tube
graphite

ω scans

4995 independent reflections

4738 reflections with *I* > 2σ(*I*)

*R*_{int} = 0.016

θ_{max} = 31.0°, θ_{min} = 2.6°

Absorption correction: multi-scan
(*SADABS*; Bruker, 2009) $h = -7 \rightarrow 10$
 $T_{\min} = 0.589$, $T_{\max} = 0.746$ $k = -11 \rightarrow 9$
 10440 measured reflections $l = -43 \rightarrow 35$

Refinement

Refinement on F^2 Primary atom site location: structure-invariant direct methods
 Least-squares matrix: full Secondary atom site location: difference Fourier map
 $R[F^2 > 2\sigma(F^2)] = 0.027$ Hydrogen site location: inferred from neighbouring sites
 $wR(F^2) = 0.064$ H atoms treated by a mixture of independent and constrained refinement
 $S = 1.13$ $w = 1/[\sigma^2(F_o^2) + (0.0258P)^2 + 1.2487P]$
 4995 reflections where $P = (F_o^2 + 2F_c^2)/3$
 194 parameters $(\Delta/\sigma)_{\max} = 0.001$
 3 restraints $\Delta\rho_{\max} = 0.56 \text{ e } \text{\AA}^{-3}$
 $\Delta\rho_{\min} = -0.44 \text{ e } \text{\AA}^{-3}$

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 (\AA^2)

	<i>x</i>	<i>y</i>	<i>z</i>	$U_{\text{iso}}^*/U_{\text{eq}}$
C1	0.8731 (2)	0.27945 (19)	0.07313 (5)	0.0113 (3)
C2	0.8347 (2)	0.4334 (2)	0.05374 (5)	0.0130 (3)
H2	0.8573	0.5314	0.0710	0.016*
C3	0.7627 (2)	0.4442 (2)	0.00884 (5)	0.0141 (3)
C4	0.7332 (2)	0.2967 (2)	-0.01512 (5)	0.0165 (3)
H4	0.6897	0.3009	-0.0461	0.020*
C5	0.7664 (2)	0.1440 (2)	0.00543 (5)	0.0167 (3)
H5	0.7415	0.0460	-0.0117	0.020*
C6	0.8351 (2)	0.1302 (2)	0.05048 (5)	0.0132 (3)
C7	0.7193 (2)	0.6104 (2)	-0.01244 (6)	0.0181 (3)
H7A	0.8379	0.6673	-0.0171	0.027*
H7B	0.6471	0.6773	0.0073	0.027*
H7C	0.6448	0.5950	-0.0414	0.027*

supplementary materials

C8	0.8579 (2)	-0.0383 (2)	0.07188 (6)	0.0171 (3)
H8A	0.9591	-0.0345	0.0965	0.026*
H8B	0.8903	-0.1193	0.0494	0.026*
H8C	0.7391	-0.0709	0.0837	0.026*
C9	1.0911 (2)	0.4117 (2)	0.13428 (5)	0.0138 (3)
H9A	1.0178	0.5160	0.1349	0.017*
H9B	1.1859	0.4248	0.1123	0.017*
C10	1.1903 (2)	0.3783 (2)	0.18030 (5)	0.0134 (3)
H10A	1.2682	0.2768	0.1793	0.016*
H10B	1.2755	0.4719	0.1894	0.016*
C11	0.8135 (2)	0.2553 (2)	0.15357 (5)	0.0123 (3)
H11A	0.7264	0.1626	0.1448	0.015*
H11B	0.7382	0.3586	0.1535	0.015*
C12	0.9092 (2)	0.2244 (2)	0.20022 (5)	0.0136 (3)
H12A	0.8123	0.2206	0.2220	0.016*
H12B	0.9739	0.1156	0.2009	0.016*
C11	0.09333 (5)	0.80876 (5)	0.181490 (13)	0.01515 (8)
C12	0.52791 (5)	0.64529 (5)	0.132584 (12)	0.01449 (8)
C13	0.34586 (5)	1.07346 (5)	0.105858 (12)	0.01456 (8)
C14	0.55737 (5)	0.97161 (5)	0.219958 (12)	0.01489 (8)
N1	0.95988 (18)	0.27010 (16)	0.12030 (4)	0.0100 (2)
H1A	1.034 (3)	0.180 (2)	0.1220 (7)	0.012*
N2	1.04987 (18)	0.35667 (16)	0.21386 (4)	0.0115 (2)
H2A	0.9874	0.4552	0.2171	0.017*
H2B	1.1124	0.3297	0.2412	0.017*
O1	0.77955 (16)	0.59834 (15)	0.22753 (4)	0.0141 (2)
H1C	0.721 (3)	0.632 (3)	0.2039 (7)	0.037 (7)*
H1B	0.691 (3)	0.564 (3)	0.2421 (8)	0.033 (7)*
Zn1	0.38078 (2)	0.86992 (2)	0.158284 (6)	0.01076 (5)

Atomic displacement parameters (\AA^2)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
C1	0.0112 (6)	0.0153 (7)	0.0071 (6)	-0.0005 (5)	0.0002 (5)	-0.0015 (5)
C2	0.0120 (6)	0.0152 (7)	0.0118 (7)	-0.0001 (5)	0.0007 (5)	-0.0004 (5)
C3	0.0101 (6)	0.0200 (8)	0.0125 (7)	0.0021 (5)	0.0017 (5)	0.0024 (6)
C4	0.0137 (7)	0.0264 (8)	0.0094 (7)	0.0002 (6)	0.0001 (5)	-0.0008 (6)
C5	0.0168 (7)	0.0205 (8)	0.0123 (7)	-0.0009 (6)	-0.0010 (5)	-0.0051 (6)
C6	0.0122 (7)	0.0153 (7)	0.0119 (7)	-0.0006 (5)	0.0006 (5)	-0.0023 (5)
C7	0.0152 (7)	0.0236 (8)	0.0156 (7)	0.0042 (6)	0.0011 (6)	0.0058 (6)
C8	0.0211 (8)	0.0133 (7)	0.0166 (7)	-0.0024 (6)	-0.0002 (6)	-0.0032 (6)
C9	0.0160 (7)	0.0142 (7)	0.0106 (6)	-0.0057 (5)	-0.0021 (5)	-0.0001 (5)
C10	0.0121 (6)	0.0174 (7)	0.0104 (6)	-0.0028 (5)	-0.0003 (5)	-0.0005 (5)
C11	0.0114 (6)	0.0163 (7)	0.0092 (6)	-0.0015 (5)	0.0013 (5)	-0.0012 (5)
C12	0.0167 (7)	0.0137 (7)	0.0101 (6)	-0.0043 (5)	0.0005 (5)	0.0002 (5)
C11	0.01373 (16)	0.01711 (17)	0.01506 (17)	-0.00208 (13)	0.00366 (12)	-0.00155 (13)
C12	0.01657 (17)	0.01327 (16)	0.01348 (16)	0.00368 (13)	0.00053 (12)	-0.00140 (12)
C13	0.01723 (17)	0.01358 (16)	0.01277 (16)	0.00171 (13)	0.00079 (12)	0.00279 (12)

C14	0.01381 (16)	0.01768 (17)	0.01260 (16)	-0.00089 (13)	-0.00196 (12)	-0.00211 (13)
N1	0.0111 (5)	0.0108 (6)	0.0078 (5)	0.0000 (4)	-0.0004 (4)	-0.0009 (4)
N2	0.0139 (6)	0.0122 (6)	0.0083 (5)	0.0003 (4)	0.0003 (4)	-0.0007 (4)
O1	0.0130 (5)	0.0165 (5)	0.0126 (5)	0.0005 (4)	0.0006 (4)	0.0012 (4)
Zn1	0.01151 (9)	0.01048 (9)	0.01017 (9)	0.00058 (6)	0.00039 (6)	-0.00003 (6)

Geometric parameters (Å, °)

C1—C2	1.387 (2)	C9—H9B	0.9900
C1—C6	1.396 (2)	C10—N2	1.490 (2)
C1—N1	1.4892 (18)	C10—H10A	0.9900
C2—C3	1.396 (2)	C10—H10B	0.9900
C2—H2	0.9500	C11—N1	1.5096 (19)
C3—C4	1.395 (2)	C11—C12	1.516 (2)
C3—C7	1.504 (2)	C11—H11A	0.9900
C4—C5	1.387 (2)	C11—H11B	0.9900
C4—H4	0.9500	C12—N2	1.4923 (19)
C5—C6	1.395 (2)	C12—H12A	0.9900
C5—H5	0.9500	C12—H12B	0.9900
C6—C8	1.506 (2)	C11—Zn1	2.2702 (5)
C7—H7A	0.9800	C12—Zn1	2.2619 (4)
C7—H7B	0.9800	C13—Zn1	2.2690 (4)
C7—H7C	0.9800	C14—Zn1	2.2857 (4)
C8—H8A	0.9800	N1—H1A	0.901 (15)
C8—H8B	0.9800	N2—H2A	0.9200
C8—H8C	0.9800	N2—H2B	0.9200
C9—N1	1.5086 (19)	O1—H1C	0.832 (17)
C9—C10	1.512 (2)	O1—H1B	0.842 (16)
C9—H9A	0.9900		
C2—C1—C6	123.19 (13)	N2—C10—H10A	109.5
C2—C1—N1	119.34 (13)	C9—C10—H10A	109.5
C6—C1—N1	117.47 (13)	N2—C10—H10B	109.5
C1—C2—C3	119.92 (15)	C9—C10—H10B	109.5
C1—C2—H2	120.0	H10A—C10—H10B	108.1
C3—C2—H2	120.0	N1—C11—C12	110.09 (12)
C4—C3—C2	117.78 (15)	N1—C11—H11A	109.6
C4—C3—C7	121.85 (14)	C12—C11—H11A	109.6
C2—C3—C7	120.37 (15)	N1—C11—H11B	109.6
C5—C4—C3	121.21 (14)	C12—C11—H11B	109.6
C5—C4—H4	119.4	H11A—C11—H11B	108.2
C3—C4—H4	119.4	N2—C12—C11	111.49 (12)
C4—C5—C6	121.95 (15)	N2—C12—H12A	109.3
C4—C5—H5	119.0	C11—C12—H12A	109.3
C6—C5—H5	119.0	N2—C12—H12B	109.3
C5—C6—C1	115.80 (14)	C11—C12—H12B	109.3
C5—C6—C8	119.83 (14)	H12A—C12—H12B	108.0
C1—C6—C8	124.34 (14)	C1—N1—C9	114.53 (12)
C3—C7—H7A	109.5	C1—N1—C11	112.33 (11)
C3—C7—H7B	109.5	C9—N1—C11	108.80 (11)

supplementary materials

H7A—C7—H7B	109.5	C1—N1—H1A	106.4 (13)
C3—C7—H7C	109.5	C9—N1—H1A	104.7 (13)
H7A—C7—H7C	109.5	C11—N1—H1A	109.6 (13)
H7B—C7—H7C	109.5	C10—N2—C12	111.83 (12)
C6—C8—H8A	109.5	C10—N2—H2A	109.3
C6—C8—H8B	109.5	C12—N2—H2A	109.3
H8A—C8—H8B	109.5	C10—N2—H2B	109.3
C6—C8—H8C	109.5	C12—N2—H2B	109.3
H8A—C8—H8C	109.5	H2A—N2—H2B	107.9
H8B—C8—H8C	109.5	H1C—O1—H1B	102 (2)
N1—C9—C10	109.95 (12)	C12—Zn1—C13	111.651 (16)
N1—C9—H9A	109.7	C12—Zn1—C11	112.737 (17)
C10—C9—H9A	109.7	C13—Zn1—C11	108.974 (16)
N1—C9—H9B	109.7	C12—Zn1—C14	109.040 (16)
C10—C9—H9B	109.7	C13—Zn1—C14	108.388 (17)
H9A—C9—H9B	108.2	C11—Zn1—C14	105.803 (16)
N2—C10—C9	110.52 (12)		
C6—C1—C2—C3	-3.1 (2)	N1—C9—C10—N2	-58.83 (17)
N1—C1—C2—C3	176.62 (13)	N1—C11—C12—N2	56.18 (17)
C1—C2—C3—C4	-0.4 (2)	C2—C1—N1—C9	-31.94 (19)
C1—C2—C3—C7	179.62 (14)	C6—C1—N1—C9	147.82 (14)
C2—C3—C4—C5	2.8 (2)	C2—C1—N1—C11	92.83 (16)
C7—C3—C4—C5	-177.28 (15)	C6—C1—N1—C11	-87.40 (16)
C3—C4—C5—C6	-1.7 (3)	C10—C9—N1—C1	-172.56 (12)
C4—C5—C6—C1	-1.7 (2)	C10—C9—N1—C11	60.82 (16)
C4—C5—C6—C8	176.45 (15)	C12—C11—N1—C1	172.87 (12)
C2—C1—C6—C5	4.1 (2)	C12—C11—N1—C9	-59.25 (16)
N1—C1—C6—C5	-175.63 (13)	C9—C10—N2—C12	55.24 (17)
C2—C1—C6—C8	-173.91 (15)	C11—C12—N2—C10	-54.17 (17)
N1—C1—C6—C8	6.3 (2)		

Hydrogen-bond geometry (Å, °)

<i>D</i> —H \cdots <i>A</i>	<i>D</i> —H	H \cdots <i>A</i>	<i>D</i> \cdots <i>A</i>	<i>D</i> —H \cdots <i>A</i>
N1—H1A \cdots C13 ⁱ	0.90 (2)	2.46 (2)	3.2300 (14)	144.(2)
N2—H2A \cdots O1	0.92	1.92	2.7925 (17)	157
N2—H2B \cdots O1 ⁱⁱ	0.92	2.19	2.9145 (17)	135
N2—H2B \cdots C14 ⁱⁱ	0.92	2.77	3.3965 (14)	127
N2—H2B \cdots C11 ⁱⁱⁱ	0.92	2.85	3.4036 (14)	120
O1—H1C \cdots C12	0.83 (2)	2.43 (2)	3.2361 (12)	164 (2)
O1—H1B \cdots C14 ⁱⁱⁱ	0.84 (2)	2.31 (2)	3.1518 (13)	178 (3)

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

Fig. 1

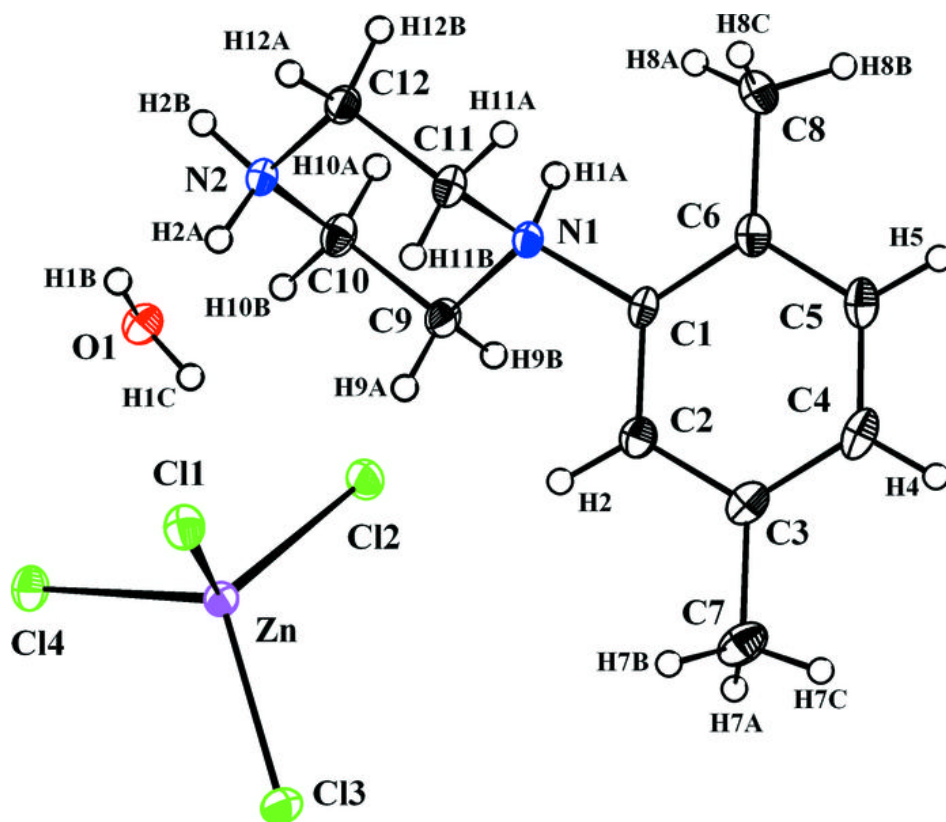


Fig. 2

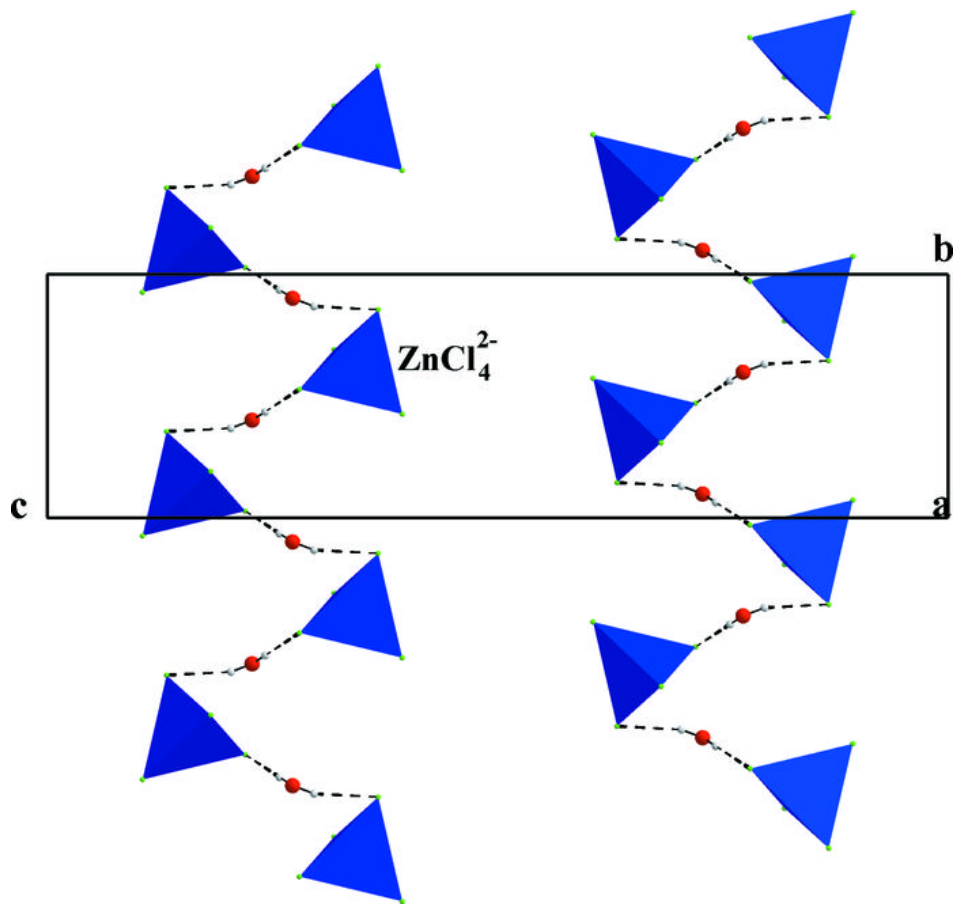


Fig. 3

