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Bis[2-(2-aminoethyl)-1*H*-benzimidazole-κ^2N^2,N^3]zinc(II) bis(perchlorate)

Jine Zhang, Yanping Li, Fangjun Huo and Zhigang Zhang*

Institute of Molecular Science, Chemical Biology and Molecular Engineering Laboratory of the Education Ministry, Shanxi University, Taiyuan, Shanxi 030006, People's Republic of China
Correspondence e-mail: zgzhang@sxu.edu.cn

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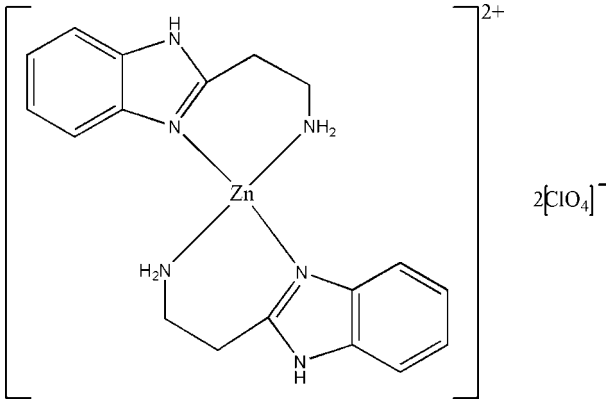
Key indicators: single-crystal X-ray study; T = 293 K; mean σ(C–C) = 0.008 Å; disorder in main residue; R factor = 0.066; wR factor = 0.184; data-to-parameter ratio = 12.4.

In the title compound, $[Zn(C_9H_{11}N_3)_2](ClO_4)_2$, the Zn^{II} atom resides on a crystallographic twofold axis and is coordinated by two benzimidazole N [Zn···N = 1.993 (4) Å] and two amine N atoms [Zn···N = 2.036 (4) Å] in a distorted tetrahedral geometry. The crystal packing is dominated by N—H···O interactions involving the perchlorate anions and π–π stacking interactions with an interplanar separation of 3.42 Å. A weak C—H···O interaction is also present.

Related literature

For related literature, see: Cescon & Day (1961); Maurva *et al.* (2006); Qiu & Tong (2005); Vallee & Auld (1990).



Experimental

Crystal data

$[Zn(C_9H_{11}N_3)_2](ClO_4)_2$
M_r = 586.69
Monoclinic, $C2/c$
a = 11.150 (3) Å
b = 15.343 (4) Å
c = 14.156 (4) Å
β = 93.322 (4)°
V = 2417.7 (12) Å^3
Z = 4
Mo $K\alpha$ radiation
μ = 1.29 mm^{-1}
T = 293 (2) K
0.30 × 0.20 × 0.20 mm

Data collection

Bruker SMART CCD area-detector diffractometer
Absorption correction: multi-scan (*SADABS*; Sheldrick, 1996)
T_{min} = 0.698, T_{max} = 0.782
5777 measured reflections
2131 independent reflections
1880 reflections with $I > 2\sigma(I)$
R_{int} = 0.023

Refinement

$R[F^2 > 2\sigma(F^2)]$ = 0.066
$wR(F^2)$ = 0.184
S = 1.10
2131 reflections
172 parameters
15 restraints
H-atom parameters constrained
$\Delta\rho_{max}$ = 0.81 e Å^{-3}
$\Delta\rho_{min}$ = −0.47 e Å^{-3}

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

D—H···A	D—H	H···A	D···A	D—H···A
N2—H2A···O1	0.86	2.09	2.946 (6)	174
N3—H3A···O4^{i}	0.90	2.12	2.953 (15)	154
C8—H8A···O1^{ii}	0.97	2.42	3.210 (9)	138

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

Data collection: *SMART* (Bruker, 2000); cell refinement: *SAINT* (Bruker, 2000); data reduction: *SAINT*; program(s) used to solve structure: *SHELXS97* (Sheldrick, 1997); program(s) used to refine structure: *SHELXL97* (Sheldrick, 1997); molecular graphics: *SHELXTL* (Bruker, 2000); 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: GG2058).

References

Bruker (2000). *SMART* (Version 5.0), *SAINT* (Version 6.02) and *SHELXTL* (Version 6.1). Bruker AXS Inc., Madison, Wisconsin, USA.
Cescon, L. A. & Day, A. R. (1961). *J. Org. Chem.* **27**, 581–586.
Maurva, M. R., Kumar, A., Ebel, M. & Rehder, D. (2006). *Inorg. Chem.* **45**, 5924–5937.
Qiu, X.-H. & Tong, X.-L. (2005). *Acta Cryst.* E**61**, m2302–m2304.
Sheldrick, G. M. (1990). *Acta Cryst.* A**46**, 467–473.
Sheldrick, G. M. (1996). *SADABS*. University of Göttingen, Germany.
Sheldrick, G. M. (1997). *SHELXL97* and *SHELXS97*. University of Göttingen, Germany.
Vallee, B. L. & Auld, D. S. (1990). *Biochemistry*, **29**, 5647–5659.

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supplementary materials

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Bis[2-(2-aminoethyl)-1*H*-benzimidazole-κ^2N^2,N^3]zinc(II) bis(perchlorate)

J. Zhang, Y. Li, F. Huo and Z. Zhang

Comment

Benzimidazole is of considerable interest as a ligand for transition metal ions. The complexes of transition metal salts with benzimidazole derivatives have been extensively studied as models of some important biological molecules (Maurva *et al.*, 2006). Incorporation of a benzimidazole moiety into alpha-amino acid molecules appeared to be of interest since the benzimidazole group has known biological activity, and its attachment to a carrier group such as an amino acid can facilitate this activity (Cescon & Day, 1961). Zinc complexes may be found in numerous biological systems. They function not only as catalytic centres in enzymes, but also as structural elements supporting three-dimensional protein structures (Vallee & Auld, 1990). In the present paper, we report the synthesis and crystal structure of a new zinc(II) benzimidazole complex, the title compound.

In (I), the Zn(II) ion exhibits a distorted tetrahedral geometry. The coordination sphere of the Zn(II) ion is comprised with two benzimidazole N atoms and two amine N atoms. The Zn—N bond distances (Table 1) are different from those reported in the literature. The Zn—N1 bond distance of 1.993 (4) Å is smaller than that reported in the literature (Qiu & Tong, 2005). The N1—Zn—N3 and N1—Zn—N3A (symmetry code: A -*x* + 2,*y*,-*z* + 1/2) bond angles are 97.02 (16)° and 118.10 (17) °, respectively, indicating a distortion of the tetrahedral coordination.

The ellipsoid plot of the molecule is shown in Fig. 1. The crystal structure of (I) is composed of Bis(1*H*-2-aminoethylbenzimidazole)zinc(II) cations and perchlorate anions. As illustrated in Fig. 2, intramolecular N—H···O hydrogen bonds and π···π stacking interactions between the benzene ring and imidazole ring [*Cg*1···*Cg*2(1/2 - *x*,1/2 - *y*,1 - *z*) = 3.7296 Å; *Cg*1 is the centroid of atoms N1,C1,C6,N2,C7, *Cg*2 is the centroid of atoms C1—C6] play key roles in stabilizing the crystal packing. The detailed hydrogen bonds information are listed in Table 2.

Experimental

All chemicals were of reagent grade and commercially available from the Beijing Chemical Reagents Company of China, and were used without further purification.

The title compound was prepared by adding a methanol solution (5 ml) of $Zn(ClO_4)_2.6H_2O$ (0.5 mmol) to a methanol solution (10 ml) of 2-aminoethybenzimidazole dihydrochloride (0.5 mmol) (Cescon & Day, 1961) neutralized by potassium carbonate. The mixture was stirred for about four hours and then filtered. Afterwards, the filtrate was slowly evaporated at room temperature to yield colorless crystals of (I) suitable for X-ray analysis. Elemental analyses of the title compound found: C 36.86%, H 3.75%, N 14.33%, and the components of the compound are calculated as C 36.85%, H 3.79%, N 14.30%.

Refinement

Disorder is present in the perchlorate anion and aminoethyl bridge and was modelled successfully. H atoms were placed in calculated positions and included in the refinement in the riding-model approximation, with C—H distances in the range 0.93–0.97 Å, N—H distances in the range 0.86–0.90 Å and $U_{iso}(H) = 1.2U_{eq}$ of the carrier atom.

Figures

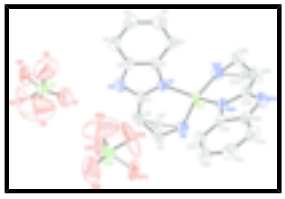

Fig. 1. The structure of the title compound with 30% displacement ellipsoids. H atoms have been omitted for clarity.

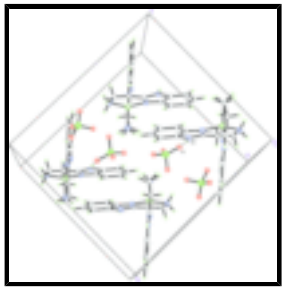

Fig. 2. The packing diagram of (I) with N—H···O hydrogen bonds shown as dashed lines.

Bis[2-(2-aminoethyl)-1*H*-benzimidazole-κ^2*N*^{2},*N*^{3}]zinc(II) bis(perchlorate)

Crystal data

$[Zn(C_9H_{11}N_3)_2](ClO_4)_2$	$F_{000} = 1200$
$M_r = 586.69$	$D_x = 1.612$ Mg m^{-3}
Monoclinic, *C*2/*c*	Mo *K*α radiation λ = 0.71073 Å
Hall symbol: -C 2yc	Cell parameters from 1957 reflections
a = 11.150 (3) Å	θ = 2.3–23.9º
b = 15.343 (4) Å	μ = 1.29 mm^{-1}
c = 14.156 (4) Å	*T* = 293 (2) K
β = 93.322 (4)º	Block, colorless
V = 2417.7 (12) Å^3	0.30 × 0.20 × 0.20 mm
Z = 4	

Data collection

Bruker SMART CCD area-detector diffractometer	2131 independent reflections
Radiation source: fine-focus sealed tube	1880 reflections with $I > 2\sigma(I)$
Monochromator: graphite	$R_{int} = 0.023$
T = 293(2) K	$\theta_{max} = 25.0$º

φ and ω scans

Absorption correction: multi-scan (SADABS; Sheldrick, 1996)

T_{min} = 0.698, T_{max} = 0.782

5777 measured reflections

θ_{min} = 2.3°

h = −13→13

k = −18→18

l = −10→16

Refinement

Refinement on F^2

Least-squares matrix: full

$R[F^2 > 2\sigma(F^2)]$ = 0.066

$wR(F^2)$ = 0.184

S = 1.10

2131 reflections

172 parameters

15 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

$w = 1/[\sigma^2(F_o^2) + (0.1191P)^2 + 1.6906P]$ where $P = (F_o^2 + 2F_c^2)/3$

$(\Delta/\sigma)_{max} < 0.001$

$\Delta\rho_{max}$ = 0.81 e Å^{-3}

$\Delta\rho_{min}$ = −0.47 e Å^{-3}

Extinction correction: none

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}	Occ. (<1)
Zn1	1.0000	0.13946 (4)	0.2500	0.0506 (3)	
N1	0.8741 (3)	0.2124 (2)	0.1829 (3)	0.0534 (9)	
N2	0.6929 (3)	0.2674 (3)	0.1500 (3)	0.0655 (11)	
H2A	0.6165	0.2745	0.1504	0.079*	
C1	0.8843 (4)	0.2811 (3)	0.1191 (3)	0.0538 (11)	
C2	0.9833 (5)	0.3136 (4)	0.0754 (4)	0.0712 (14)	
H2	1.0595	0.2900	0.0872	0.085*	
C3	0.9638 (6)	0.3819 (4)	0.0142 (5)	0.0805 (16)	
H3	1.0287	0.4055	−0.0155	0.097*	
C4	0.8497 (6)	0.4174 (4)	−0.0050 (4)	0.0776 (16)	
H4	0.8413	0.4643	−0.0464	0.093*	
C5	0.7494 (6)	0.3855 (3)	0.0351 (4)	0.0686 (14)	

H5	0.6732	0.4083	0.0211	0.082*	
C6	0.7700 (4)	0.3157 (3)	0.0990 (3)	0.0549 (11)	
C7	0.7577 (4)	0.2068 (3)	0.1993 (4)	0.0624 (12)	
C8	0.7035 (6)	0.1424 (4)	0.2624 (5)	0.0905 (17)	
H8A	0.6298	0.1675	0.2837	0.109*	0.594 (12)
H8B	0.6812	0.0916	0.2247	0.109*	0.594 (12)
H8A'	0.6876	0.1728	0.3205	0.109*	0.406 (12)
H8B'	0.6261	0.1264	0.2327	0.109*	0.406 (12)
Cl1	0.37395 (13)	0.37873 (10)	0.10975 (10)	0.0736 (4)	
O1	0.4321 (5)	0.2999 (4)	0.1393 (6)	0.141 (2)	
N3	0.8879 (4)	0.0694 (3)	0.3295 (3)	0.0673 (11)	
H3A	0.8725	0.0179	0.3008	0.081*	0.594 (12)
H3B	0.9256	0.0584	0.3861	0.081*	0.594 (12)
H3A'	0.9179	0.0152	0.3372	0.081*	0.406 (12)
H3B'	0.8869	0.0940	0.3871	0.081*	0.406 (12)
O2	0.4361 (8)	0.4352 (7)	0.0603 (7)	0.214 (5)	
O3	0.2643 (9)	0.3764 (10)	0.130 (2)	0.235 (14)	0.594 (12)
C9	0.7735 (7)	0.1132 (8)	0.3445 (6)	0.0905 (17)	0.594 (12)
H9A	0.7243	0.0735	0.3790	0.109*	0.594 (12)
H9B	0.7904	0.1633	0.3849	0.109*	0.594 (12)
O3'	0.2863 (12)	0.3531 (10)	0.0330 (13)	0.118 (6)	0.406 (12)
C9'	0.7639 (7)	0.0630 (5)	0.2892 (9)	0.061 (4)	0.406 (12)
H9A'	0.7642	0.0260	0.2337	0.073*	0.406 (12)
H9B'	0.7167	0.0335	0.3350	0.073*	0.406 (12)
O4	0.364 (2)	0.4319 (9)	0.1837 (10)	0.337 (9)	

Atomic displacement parameters (Å^2)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
Zn1	0.0418 (4)	0.0565 (5)	0.0526 (5)	0.000	−0.0053 (3)	0.000
N1	0.0406 (19)	0.056 (2)	0.063 (2)	0.0001 (15)	−0.0033 (16)	0.0050 (17)
N2	0.0411 (19)	0.069 (2)	0.086 (3)	0.0064 (18)	0.0020 (19)	0.003 (2)
C1	0.050 (2)	0.051 (2)	0.060 (3)	−0.0044 (18)	−0.009 (2)	−0.004 (2)
C2	0.057 (3)	0.076 (3)	0.080 (4)	−0.005 (2)	−0.002 (3)	0.015 (3)
C3	0.076 (4)	0.074 (3)	0.092 (4)	−0.009 (3)	0.005 (3)	0.026 (3)
C4	0.094 (4)	0.062 (3)	0.075 (4)	0.001 (3)	−0.010 (3)	0.012 (3)
C5	0.077 (4)	0.053 (2)	0.074 (3)	0.008 (2)	−0.019 (3)	−0.003 (2)
C6	0.054 (3)	0.047 (2)	0.062 (3)	0.0038 (19)	−0.009 (2)	−0.009 (2)
C7	0.044 (2)	0.063 (3)	0.080 (3)	0.001 (2)	−0.001 (2)	0.012 (2)
C8	0.061 (3)	0.099 (3)	0.113 (4)	0.001 (2)	0.019 (3)	0.030 (3)
Cl1	0.0667 (9)	0.0818 (9)	0.0722 (9)	0.0043 (6)	0.0054 (7)	0.0076 (7)
O1	0.095 (4)	0.146 (5)	0.182 (6)	0.024 (4)	0.007 (4)	0.039 (4)
N3	0.064 (2)	0.073 (3)	0.065 (2)	−0.004 (2)	0.005 (2)	0.014 (2)
O2	0.172 (7)	0.230 (9)	0.236 (9)	−0.102 (7)	−0.023 (7)	0.106 (8)
O3	0.056 (6)	0.149 (11)	0.51 (4)	0.036 (6)	0.073 (12)	0.136 (18)
C9	0.061 (3)	0.099 (3)	0.113 (4)	0.001 (2)	0.019 (3)	0.030 (3)
O3'	0.048 (7)	0.149 (13)	0.152 (14)	0.004 (6)	−0.036 (7)	−0.047 (10)
C9'	0.055 (7)	0.056 (7)	0.072 (8)	−0.012 (5)	0.009 (6)	0.012 (6)

O4	0.57 (3)	0.241 (13)	0.207 (10)	−0.006 (16)	0.084 (15)	−0.121 (10)

Geometric parameters (Å, °)

Zn1—N1^{i}	1.993 (4)	C8—H8A	0.9700
Zn1—N1	1.993 (4)	C8—H8B	0.9700
Zn1—N3^{i}	2.036 (4)	C8—H8A'	0.9700
Zn1—N3	2.036 (4)	C8—H8B'	0.9700
N1—C7	1.335 (6)	C3—H3	0.9300
N1—C1	1.397 (6)	Cl1—O3	1.273 (10)
C5—C4	1.373 (9)	Cl1—O2	1.333 (7)
C5—C6	1.412 (7)	Cl1—O4	1.336 (10)
C5—H5	0.9300	Cl1—O1	1.423 (6)
C1—C2	1.389 (7)	Cl1—O3'	1.472 (12)
C1—C6	1.394 (6)	N3—C9'	1.468 (8)
C7—N2	1.347 (6)	N3—C9	1.468 (8)
C7—C8	1.484 (7)	N3—H3A	0.9000
C6—N2	1.372 (6)	N3—H3B	0.9000
N2—H2A	0.8600	N3—H3A'	0.9000
C2—C3	1.369 (8)	N3—H3B'	0.9000
C2—H2	0.9300	C9—H9A	0.9700
C4—C3	1.396 (8)	C9—H9B	0.9700
C4—H4	0.9300	C9'—H9A'	0.9700
C8—C9'	1.433 (7)	C9'—H9B'	0.9700
C8—C9	1.433 (7)		
N1^{i}—Zn1—N1	111.6 (2)	H8A—C8—H8B'	59.1
N1^{i}—Zn1—N3^{i}	97.02 (16)	H8B—C8—H8B'	50.4
N1—Zn1—N3^{i}	118.10 (17)	H8A'—C8—H8B'	106.7
N1^{i}—Zn1—N3	118.10 (17)	C2—C3—C4	122.1 (5)
N1—Zn1—N3	97.02 (16)	C2—C3—H3	119.0
N3^{i}—Zn1—N3	116.3 (3)	C4—C3—H3	119.0
C7—N1—C1	106.1 (4)	O3—Cl1—O2	132.2 (7)
C7—N1—Zn1	123.1 (3)	O3—Cl1—O4	73.4 (14)
C1—N1—Zn1	130.6 (3)	O2—Cl1—O4	94.9 (10)
C4—C5—C6	115.4 (5)	O3—Cl1—O1	109.7 (6)
C4—C5—H5	122.3	O2—Cl1—O1	117.7 (6)
C6—C5—H5	122.3	O4—Cl1—O1	110.3 (9)
C2—C1—C6	120.9 (4)	O3—Cl1—O3'	63.5 (13)
C2—C1—N1	130.9 (4)	O2—Cl1—O3'	97.4 (9)
C6—C1—N1	108.2 (4)	O4—Cl1—O3'	131.1 (11)
N1—C7—N2	111.3 (4)	O1—Cl1—O3'	105.2 (7)
N1—C7—C8	125.6 (4)	C9'—N3—Zn1	114.5 (5)
N2—C7—C8	123.1 (4)	C9—N3—Zn1	113.9 (5)
N2—C6—C1	106.2 (4)	C9'—N3—H3A	67.4
N2—C6—C5	131.6 (5)	C9—N3—H3A	108.8
C1—C6—C5	122.2 (5)	Zn1—N3—H3A	108.8
C7—N2—C6	108.2 (4)	C9'—N3—H3B	135.6

C7—N2—H2A	125.9	C9—N3—H3B	108.8
C6—N2—H2A	125.9	Zn1—N3—H3B	108.8
C3—C2—C1	117.1 (5)	H3A—N3—H3B	107.7
C3—C2—H2	121.5	C9'—N3—H3A'	108.6
C1—C2—H2	121.5	C9—N3—H3A'	136.4
C5—C4—C3	122.4 (5)	Zn1—N3—H3A'	108.6
C5—C4—H4	118.8	H3A—N3—H3A'	45.8
C3—C4—H4	118.8	H3B—N3—H3A'	64.4
C9'—C8—C9	45.1 (7)	C9'—N3—H3B'	108.6
C9'—C8—C7	121.6 (6)	C9—N3—H3B'	67.7
C9—C8—C7	118.3 (6)	Zn1—N3—H3B'	108.6
C9'—C8—H8A	130.4	H3A—N3—H3B'	139.9
C9—C8—H8A	107.7	H3B—N3—H3B'	45.6
C7—C8—H8A	107.7	H3A'—N3—H3B'	107.6
C9'—C8—H8B	64.1	C8—C9—N3	117.6 (6)
C9—C8—H8B	107.7	C8—C9—H9A	107.9
C7—C8—H8B	107.7	N3—C9—H9A	107.9
H8A—C8—H8B	107.1	C8—C9—H9B	107.9
C9'—C8—H8A'	106.9	N3—C9—H9B	107.9
C9—C8—H8A'	65.3	H9A—C9—H9B	107.2
C7—C8—H8A'	106.9	C8—C9'—N3	117.6 (6)
H8A—C8—H8A'	49.3	C8—C9'—H9A'	107.9
H8B—C8—H8A'	143.1	N3—C9'—H9A'	107.9
C9'—C8—H8B'	106.9	C8—C9'—H9B'	107.9
C9—C8—H8B'	134.5	N3—C9'—H9B'	107.9
C7—C8—H8B'	106.9	H9A'—C9'—H9B'	107.2

Symmetry codes: (i) −*x*+2, *y*, −*z*+1/2.

Hydrogen-bond geometry (Å, °)

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
N2—H2A···O1	0.86	2.09	2.946 (6)	174
N3—H3A···O4^{ii}	0.90	2.12	2.953 (15)	154
C8—H8A···O1^{iii}	0.97	2.42	3.210 (9)	138

Symmetry codes: (ii) *x*+1/2, *y*−1/2, *z*; (iii) −*x*+1, *y*, −*z*+1/2.

Fig. 1

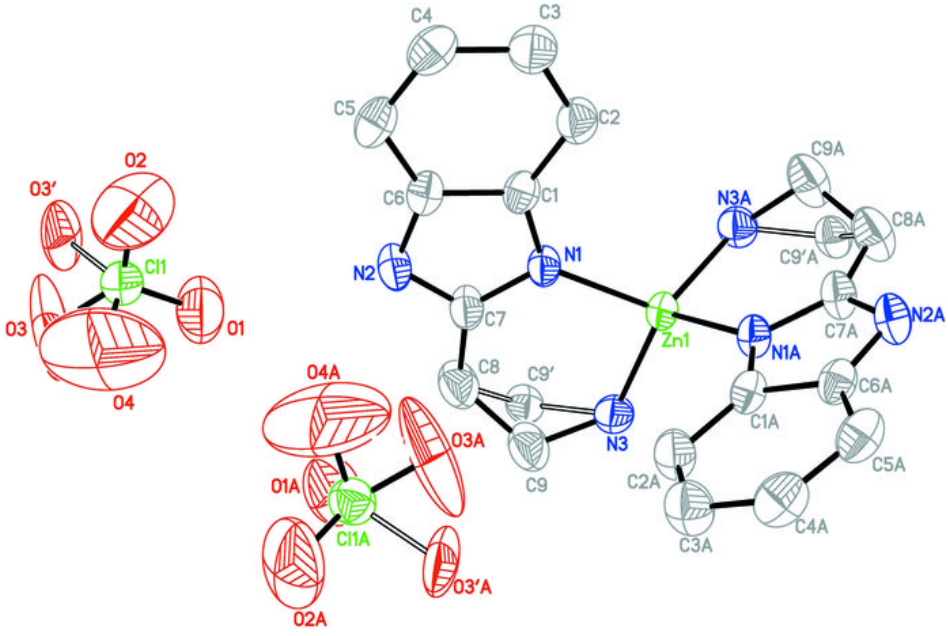
C4
C3
C5
C2
C6
C1
N2
N1
C7
C8
C9′
N3
C9
Zn1
N3A
C9A
C8A
C9′A
N2A
C7A
N1A
C6A
C1A
C5A
C2A
C4A
C3A
O2
O3′
Cl1
O3
O1
O4
O4A
O3A
O1A
Cl1A
O3′A
O2A

Fig. 2