metal-organic compounds

Dichloridobis(pyrazine-2-carboxamide-κN^4)zinc(II)

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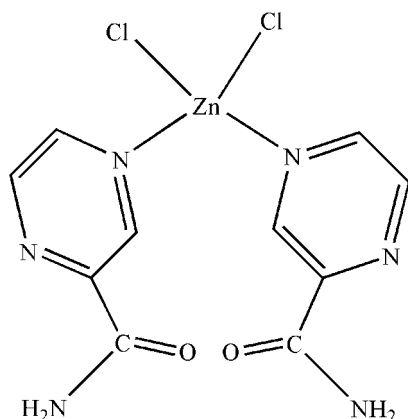Correspondence e-mail: sadif.shirvan1@gmail.com

In the crystal of the title compound, [ZnCl$_2$(C$_5$H$_5$N$_3$O)$_2$], the molecule has *m* symmetry, with the Zn^{II} cation and Cl$^-$ anions located on the mirror plane. The Zn^{II} cation is coordinated by two Cl$^-$ anions and two pyrazine-2-carboxamide ligands in a distorted ZnCl$_2$N$_2$ tetrahedral geometry. The two pyrazine rings are nearly perpendicular to each other [dihedral angle = 86.61 (10)°]. Intermolecular N—H⋯O and N—H⋯N hydrogen bonds and weak C—H⋯O interactions stabilize the crystal packing.

Related literature

For related structures, see: Abu-Youssef *et al.* (2006); Azhdari Tehrani *et al.* (2010); Goher & Mautner (2000); Kristiansson (2002); Mir Mohammad Sadegh *et al.* (2010); Munakata *et al.* (1997); Pacigova *et al.* (2008).

Experimental

Crystal data

[ZnCl$_2$(C$_5$H$_5$N$_3$O)$_2$]	$b = 19.7629$ (14) Å
$M_r = 382.53$	$c = 6.8396$ (5) Å
Monoclinic, $P2_1/m$	$\beta = 105.131$ (7)°
$a = 5.4296$ (5) Å	$V = 708.48$ (10) Å^3

$Z = 2$	$T = 298$ K
Mo $K\alpha$ radiation	$0.40 \times 0.06 \times 0.05$ mm
$\mu = 2.12$ mm^{-1}	

Data collection

Bruker APEXII CCD area-detector' diffractometer	5777 measured reflections
	1441 independent reflections
Absorption correction: multi-scan (*SADABS*; Bruker, 2001)	1064 reflections with $I > 2\sigma(I)$
$T_{min} = 0.881$, $T_{max} = 0.902$	$R_{int} = 0.085$

Refinement

$R[F^2 > 2\sigma(F^2)] = 0.045$	100 parameters
$wR(F^2) = 0.093$	H-atom parameters constrained
$S = 0.97$	$\Delta\rho_{max} = 0.78$ e Å^{-3}
1441 reflections	$\Delta\rho_{min} = -0.65$ e Å^{-3}

Table 1
Selected bond lengths (Å).

Zn1—N1	2.085 (3)	Zn1—Cl2	2.1888 (16)
Zn1—Cl1	2.1945 (16)		

Table 2
Hydrogen-bond geometry (Å, °).

D—H⋯A	D—H	H⋯A	D⋯A	D—H⋯A
N3—H3B⋯O1^i	0.86	2.02	2.875 (5)	175
N3—H3C⋯N2^{ii}	0.86	2.61	3.205 (5)	128
C3—H3⋯O1^{iii}	0.93	2.44	3.357 (5)	170

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

Data collection: *APEX2* (Bruker, 2007); cell refinement: *SAINT* (Bruker, 2007); 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*.

References

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supplementary materials

Dichloridobis(pyrazine-2-carboxamide-*κN*⁴)zinc(II)

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Comment

Pyrazine-2-carboxamide (pzc), is a good ligand, and a few complexes with pzc have been prepared, such as that of mercury (Azhdari Tehrani *et al.*, 2010; Mir Mohammad Sadegh *et al.*, 2010), vanadium (Pacigova *et al.*, 2008), manganese (Abu-Youssef *et al.*, 2006) and copper (Kristiansson, 2002; Munakata *et al.*, 1997; Goher & Mautner, 2000). Here, we report the synthesis and structure of the title compound.

The asymmetric unit of the title compound, (Fig. 1), contains one Zn^{II} atom, two Cl atoms and one pyrazine-2-carboxamide ligand. The Zn^{II} atom is four-coordinated in a distorted tetrahedral configuration by two N atoms from two pyrazine-2-carboxamide ligands and two terminal Cl atoms. The Zn—Cl and Zn—N bond lengths and angles are collected in Table 1.

In the crystal structure, intermolecular N—H⋯O, N—H⋯N and C—H⋯O hydrogen bonds (Table 2, Fig. 2) may stabilize the structure.
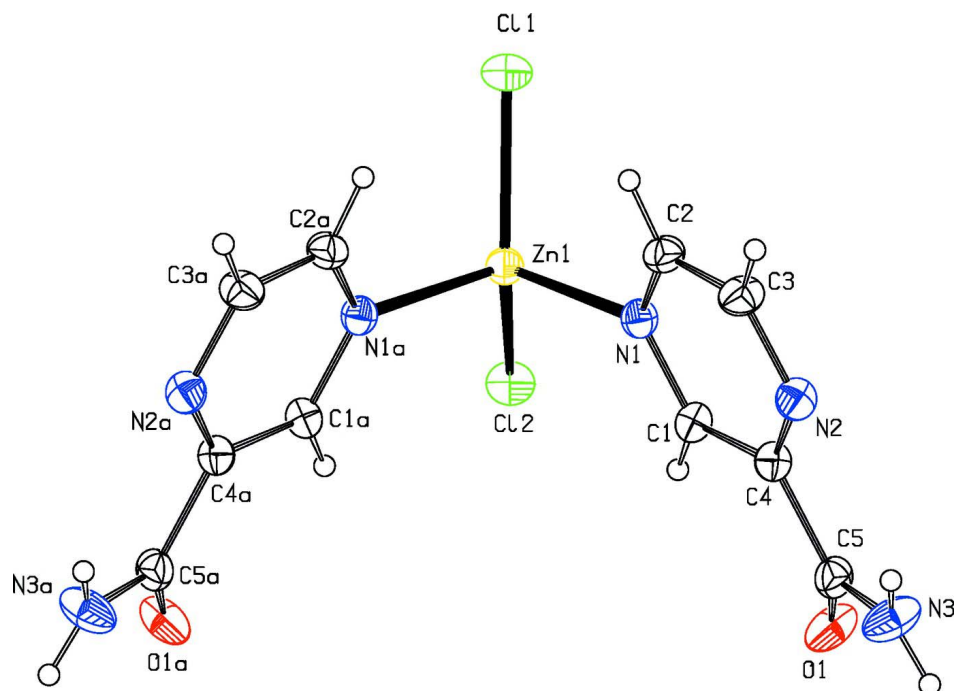
Experimental

A solution of pyrazine-2-carboxamide (0.25 g, 2.0 mmol) in methanol (10 ml) was added to a solution of $ZnCl_2$ (0.13 g, 1.0 mmol) in methanol (10 ml) and the resulting colorless solution was stirred for 15 min at room temperature. This solution was left to evaporate slowly at room temperature. After one week, colorless plate crystals of the title compound were isolated (yield 0.30 g, 78.4%).

Refinement

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

Computing details

Data collection: *APEX2* (Bruker, 2007); cell refinement: *SAINT* (Bruker, 2007); data reduction: *SAINT* (Bruker, 2007); program(s) used to solve structure: *SHELXTL* (Sheldrick, 2008); program(s) used to refine structure: *SHELXTL* (Sheldrick, 2008); molecular graphics: *SHELXTL* (Sheldrick, 2008); software used to prepare material for publication: *SHELXTL* (Sheldrick, 2008).

Figure 1

The molecular structure of the title molecule, with the atom-numbering scheme. Displacement ellipsoids are drawn at the 30% probability level. [Symmetry codes: (*a*) *x*,1/2 - *y*,*z*].

Figure 2

Unit-cell packing diagram for title molecule. Hydrogen bonds are shown as dashed lines

Dichloridobis(pyrazine-2-carboxamide-*κN*⁴)zinc(II)

Crystal data

[ZnCl$_2$(C$_5$H$_5$N$_3$O)$_2$]	Hall symbol: -P 2yb
M_r = 382.53	a = 5.4296 (5) Å
Monoclinic, $P2_1/m$	b = 19.7629 (14) Å

$c = 6.8396$ (5) Å

$\beta = 105.131$ (7)°

$V = 708.48$ (10) Å^3

$Z = 2$

$F(000) = 384$

$D_x = 1.793$ Mg m^{-3}

Mo $K\alpha$ radiation, $\lambda = 0.71073$ Å

Cell parameters from 5777 reflections

$\theta = 2.1–26.0°$

$\mu = 2.12$ mm^{-1}

$T = 298$ K

Plate, colorless

$0.40 \times 0.06 \times 0.05$ mm

Data collection

Bruker APEXII CCD area-detector′ diffractometer

Radiation source: fine-focus sealed tube

Graphite monochromator

ω scans

Absorption correction: multi-scan (*SADABS*; Bruker, 2001)

$T_{min} = 0.881$, $T_{max} = 0.902$

5777 measured reflections

1441 independent reflections

1064 reflections with $I > 2\sigma(I)$

$R_{int} = 0.085$

$\theta_{max} = 26.0°$, $\theta_{min} = 2.1°$

$h = -6 \rightarrow 6$

$k = -21 \rightarrow 24$

$l = -8 \rightarrow 8$

Refinement

Refinement on F^2

Least-squares matrix: full

$R[F^2 > 2\sigma(F^2)] = 0.045$

$wR(F^2) = 0.093$

$S = 0.97$

1441 reflections

100 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

$w = 1/[\sigma^2(F_o^2) + (0.0467P)^2]$ where $P = (F_o^2 + 2F_c^2)/3$

$(\Delta/\sigma)_{max} = 0.003$

$\Delta\rho_{max} = 0.78$ e Å^{-3}

$\Delta\rho_{min} = -0.65$ 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 (Å^2)

	x	y	z	$U_{iso}*/U_{eq}$
C1	0.3963 (7)	0.3616 (2)	0.4810 (6)	0.0307 (9)
H1	0.2583	0.3512	0.3724	0.037*
C2	0.6226 (8)	0.3464 (2)	0.8085 (6)	0.0364 (10)
H2	0.6435	0.3262	0.9347	0.044*
C3	0.8016 (8)	0.3930 (2)	0.7802 (6)	0.0391 (10)
H3	0.9428	0.4022	0.8873	0.047*
C4	0.5722 (7)	0.4094 (2)	0.4563 (6)	0.0310 (8)
C5	0.5370 (8)	0.4445 (2)	0.2579 (6)	0.0366 (9)
N1	0.4213 (6)	0.33021 (16)	0.6574 (5)	0.0312 (7)
N2	0.7776 (6)	0.42473 (18)	0.6048 (5)	0.0366 (8)

N3	0.7336 (7)	0.4801 (2)	0.2331 (6)	0.0535 (11)
H3B	0.7210	0.5022	0.1226	0.064*
H3C	0.8736	0.4812	0.3276	0.064*
O1	0.3309 (6)	0.44069 (17)	0.1293 (4)	0.0506 (8)
Zn1	0.18582 (12)	0.2500	0.69002 (10)	0.0304 (2)
Cl1	0.1855 (3)	0.2500	1.0108 (2)	0.0480 (4)
Cl2	−0.1427 (3)	0.2500	0.4244 (2)	0.0416 (4)

Atomic displacement parameters (Å²)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
C1	0.0264 (19)	0.030 (2)	0.034 (2)	−0.0008 (16)	0.0050 (17)	0.0030 (17)
C2	0.040 (2)	0.038 (2)	0.028 (2)	−0.0004 (18)	0.0041 (18)	0.0030 (18)
C3	0.036 (2)	0.046 (3)	0.030 (2)	−0.0092 (19)	−0.0004 (18)	0.0013 (19)
C4	0.0278 (19)	0.030 (2)	0.034 (2)	−0.0010 (16)	0.0063 (16)	−0.0002 (17)
C5	0.041 (2)	0.031 (2)	0.036 (2)	−0.0065 (18)	0.0050 (19)	0.0002 (18)
N1	0.0300 (16)	0.0295 (18)	0.0341 (19)	−0.0027 (14)	0.0081 (14)	−0.0011 (14)
N2	0.0325 (18)	0.037 (2)	0.037 (2)	−0.0053 (14)	0.0032 (15)	−0.0011 (15)
N3	0.041 (2)	0.073 (3)	0.042 (2)	−0.0229 (19)	0.0026 (17)	0.017 (2)
O1	0.0432 (17)	0.058 (2)	0.0413 (18)	−0.0198 (15)	−0.0053 (15)	0.0173 (16)
Zn1	0.0290 (3)	0.0313 (4)	0.0329 (4)	0.000	0.0114 (3)	0.000
Cl1	0.0570 (10)	0.0575 (11)	0.0328 (8)	0.000	0.0174 (7)	0.000
Cl2	0.0303 (7)	0.0525 (10)	0.0408 (8)	0.000	0.0069 (6)	0.000

Geometric parameters (Å, º)

C1—N1	1.332 (5)	C5—O1	1.232 (5)
C1—C4	1.384 (5)	C5—N3	1.326 (5)
C1—H1	0.9300	N1—Zn1	2.085 (3)
C2—N1	1.333 (5)	N3—H3B	0.8600
C2—C3	1.388 (6)	N3—H3C	0.8600
C2—H2	0.9300	Zn1—N1	2.085 (3)
C3—N2	1.330 (5)	Zn1—N1i	2.085 (3)
C3—H3	0.9300	Zn1—Cl1	2.1945 (16)
C4—N2	1.333 (5)	Zn1—Cl2	2.1888 (16)
C4—C5	1.491 (6)		
N1—C1—C4	121.2 (4)	N3—C5—C4	116.6 (4)
N1—C1—H1	119.4	C1—N1—C2	117.4 (3)
C4—C1—H1	119.4	C1—N1—Zn1	122.1 (3)
N1—C2—C3	120.9 (4)	C2—N1—Zn1	120.1 (3)
N1—C2—H2	119.6	C3—N2—C4	116.4 (3)
C3—C2—H2	119.6	C5—N3—H3B	120.0
N2—C3—C2	122.1 (4)	C5—N3—H3C	120.0
N2—C3—H3	118.9	H3B—N3—H3C	120.0
C2—C3—H3	118.9	N1—Zn1—N1i	99.00 (18)
N2—C4—C1	121.9 (4)	N1—Zn1—Cl2	107.51 (10)
N2—C4—C5	118.1 (3)	N1i—Zn1—Cl2	107.51 (10)
C1—C4—C5	120.0 (3)	N1—Zn1—Cl1	105.53 (9)
O1—C5—N3	123.5 (4)	N1i—Zn1—Cl1	105.53 (9)

O1—C5—C4	119.9 (4)	Cl2—Zn1—Cl1	128.07 (6)
N1—C2—C3—N2	1.9 (7)	C3—C2—N1—Zn1	170.7 (3)
N1—C1—C4—N2	2.1 (6)	C2—C3—N2—C4	−0.2 (6)
N1—C1—C4—C5	−179.0 (4)	C1—C4—N2—C3	−1.7 (6)
N2—C4—C5—O1	−167.5 (4)	C5—C4—N2—C3	179.4 (4)
C1—C4—C5—O1	13.5 (6)	C1—N1—Zn1—N1^i	96.5 (3)
N2—C4—C5—N3	10.8 (6)	C2—N1—Zn1—N1^i	−75.5 (3)
C1—C4—C5—N3	−168.2 (4)	C1—N1—Zn1—Cl2	−15.2 (3)
C4—C1—N1—C2	−0.3 (6)	C2—N1—Zn1—Cl2	172.9 (3)
C4—C1—N1—Zn1	−172.5 (3)	C1—N1—Zn1—Cl1	−154.6 (3)
C3—C2—N1—C1	−1.5 (6)	C2—N1—Zn1—Cl1	33.5 (3)

Symmetry code: (i) x, −y+1/2, z.

Hydrogen-bond geometry (Å, º)

D—H···A	D—H	H···A	D···A	D—H···A
N3—H3B···O1^{ii}	0.86	2.02	2.875 (5)	175
N3—H3C···N2^{iii}	0.86	2.61	3.205 (5)	128
C3—H3···O1^{iv}	0.93	2.44	3.357 (5)	170

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