Acta Crystallographica Section E
Structure Reports Online
ISSN 1600-5368

Diaquabis(4,5-diazafluoren-9-one-κ^2N,N')-zinc(II) diperchlorate

Rong-Lan Zhang,[a] Jian-She Zhao,[a] Shi-Yao Yang[b] and Seik Weng Ng[c]*

[a]Department of Chemistry, Shaanxi Key Laboratory for Physico-Inorganic Chemistry, Northwest University, Xi'an 710069, People's Republic of China, [b]Department of Chemistry, Xiamen University, Xiamen 361005, People's Republic of China, and [c]Department of Chemistry, University of Malaya, 50603 Kuala Lumpur, Malaysia

Correspondence e-mail: seikweng@um.edu.my

Key indicators

Single-crystal X-ray study
T = 298 K
Mean σ(C–C) = 0.007 Å
Disorder in solvent or counterion
R factor = 0.068
wR factor = 0.185
Data-to-parameter ratio = 12.4

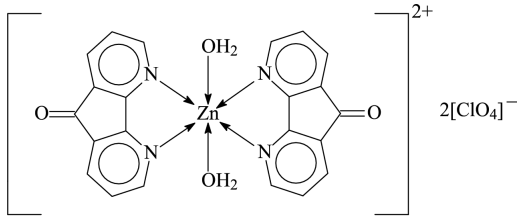
For details of how these key indicators were automatically derived from the article, see http://journals.iucr.org/e.

Received 12 January 2004
Accepted 26 January 2004
Online 7 February 2004

The Zn atom in the title compound, $[Zn(C_{11}H_6N_2O)_2(H_2O)_2](ClO_4)_2$, lies on a center of symmetry in the crystal structure, and it is chelated by the heterocyclic ligands [Zn—O = 2.151 (4) and 2.276 (4) Å]. The coordinated water molecule interacts with the perchlorate ions to form a hydrogen-bonded layer structure. The compound is isomorphous with the Cu analog, whose structure has been detailed by Menon & Rajasekharan [*Polyhedron* (1998), **17**, 2463–2476].

Comment

Although 4,5-diazafluoren-9-one sometimes does not exhibit its chelating behavior, it is known to bind copper(II) if the anionic group is the perchlorate group. The crystal structure of diaquabis(4,5-diazafluoren-9-one)copper diperchlorate has been described in detail (Gu *et al.*, 2002; Menon & Rajasekharan, 1998; Zhao *et al.*, 2003; Zhang, Zhao *et al.*, 2003). The present centrosymmetric Zn analog, (I) (Fig. 1), is isomorphous with the Cu compound. With chloride as the counterion, the ligand does not behave as a chelate; the ligand is instead protonated in $(C_6H_{12}N_2O)_2(ZnCl_4)\cdot 2H_2O$ (Zhang, Liu *et al.*, 2003).



(I)

Experimental

One drop of tris(2-aminoethyl)amine and three drops of perchloric acid were added to an ethanol solution of zinc dichloride (0.14 g, 1 mmol). 4,5-Diazafluoren-9-one (0.18 g, 1 mmol) dissolved in ethanol was added, and the mixture heated for 1 h. Yellow crystals of (I) separated from the solution after several days.

Crystal data

$[Zn(C_{11}H_6N_2O)_2(H_2O)_2](ClO_4)_2$
M_r = 664.66
Monoclinic, $P2_1/n$
a = 8.1493 (4) Å
b = 11.5615 (5) Å
c = 13.2055 (6) Å
β = 90.662 (1)°
V = 1244.1 (1) Å^3
Z = 2
D_x = 1.774 Mg m^{-3}
Mo $K\alpha$ radiation
Cell parameters from 6381 reflections
θ = 2.3–28.1°
μ = 1.28 mm^{-1}
T = 298 (2) K
Irregular block, yellow
0.31 × 0.27 × 0.25 mm

Data collection

Bruker SMART APEX area-detector diffractometer
φ and ω scans
Absorption correction: multi-scan (*SADABS*; Sheldrick, 1996)
T_{min} = 0.440, T_{max} = 0.724
14 093 measured reflections
2848 independent reflections
2431 reflections with $I > 2\sigma(I)$
R_{int} = 0.040
θ_{max} = 27.5°
$h = -10 \rightarrow 10$
$k = -15 \rightarrow 14$
$l = -17 \rightarrow 17$

Refinement

Refinement on F^2
$R[F^2 > 2\sigma(F^2)]$ = 0.068
$wR(F^2)$ = 0.185
S = 1.10
2848 reflections
230 parameters
H atoms treated by a mixture of independent and constrained refinement
$w = 1/[\sigma^2(F_o^2) + (0.1043P)^2 + 0.991P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max}$ = 0.001
$\Delta\rho_{max}$ = 0.60 e Å^{-3}
$\Delta\rho_{min}$ = −0.44 e Å^{-3}

Table 1
Selected geometric parameters (Å, °).

Zn1—O1*w*	2.073 (3)	Zn1—N2	2.151 (4)
Zn1—N1	2.276 (4)		
O1*w*—Zn1—O1*w*^{i}	180.0	N1—Zn1—N1^{i}	180.0
O1*w*—Zn1—N1	88.9 (2)	N1—Zn1—N2	80.8 (1)
O1*w*—Zn1—N1^{i}	91.1 (2)	N1—Zn1—N2^{i}	99.2 (1)
O1*w*—Zn1—N2	90.6 (1)	N2—Zn1—N2^{i}	180.0
O1*w*—Zn1—N2^{i}	89.4 (1)		

Symmetry code: (i) $1-x, 1-y, 1-z$.

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

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
O1*w*—H1*w*1···O2	0.84 (1)	1.96 (4)	2.667 (7)	140 (6)
O1*w*—H1*w*2···O4^{ii}	0.84 (1)	2.03 (2)	2.80 (1)	152 (4)
O1*w*—H1*w*2···O4′^{ii}	0.84 (1)	1.84 (4)	2.54 (1)	138 (6)

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

The aromatic H atoms were positioned geometrically [C—H = 0.93 Å and U_{iso}(H) = 1.2U_{eq}(C)] and were allowed to ride on their parent atoms in the riding-model approximation. The water H atoms were located and refined with distance restraints O—H = 0.85 (1) and H···H = 1.39 (1) Å. The perchlorate ion is disordered over two sites sharing a common Cl atom, and it was refined as two ions, with distance restraints Cl—O = 1.44 (1) and O···O = 2.35 (1) Å. The displacement parameters of the O atoms were restrained to be approximately isotropic. The perchlorate group refined to a ratio of 0.62 (1)/0.38 (1).

Data collection: *SMART* (Bruker, 2001); cell refinement: *SMART*; data reduction: *SAINT* (Bruker, 2001); program(s) used to solve structure: *SHELXS*97 (Sheldrick, 1997); program(s) used to refine structure: *SHELXL*97 (Sheldrick, 1997); molecular graphics: *ORTEP*II (Johnson, 1976); software used to prepare material for publication: *SHELXL*97.

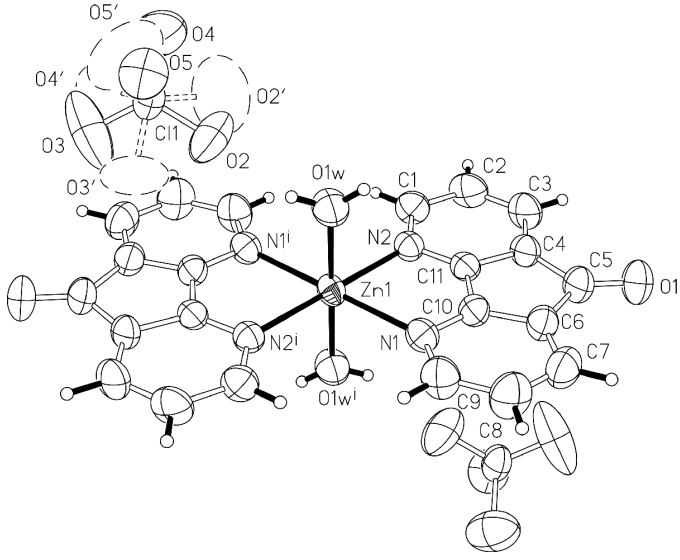


Figure 1
*ORTEP*II (Johnson, 1976) plot of (I); displacement ellipsoids are drawn at the 50% probability level. H atoms are drawn as spheres of arbitrary radii. Both disorder components of the perchlorate anion are shown. [Symmetry code: (i) $1-x, 1-y, 1-z$.]

The authors thank the National Science Foundation of China (No. 20071027), the National Science Foundation of Shaanxi Province (No. OOJK130), Xiamen University and the University of Malaya for supporting this work.

References

Bruker (2001). *SAINT* (Version 6.28) and *SMART* (Version 6.28). Bruker AXS Inc., Madison, Wisconsin, USA.
Gu, A.-P., Zhang, R.-L., Zhang, G., Zhao, J.-S., He, S.-Y., Du, J.-M. & Wang, D.-Q. (2002). *Chin. J. Chem.* **20**, 1616–1620.
Johnson, C. K. (1976). *ORTEP*II. Report ORNL-5138. Oak Ridge National Laboratory, Tennessee, USA.
Menon, S. & Rajasekharan, M. V. (1998). *Polyhedron*, **17**, 2463–2476.
Sheldrick, G. M. (1996). *SADABS*. University of Göttingen, Germany.
Sheldrick, G. M. (1997). *SHELXS*97 and *SHELXL*97. University of Göttingen, Germany.
Zhang, R.-L., Liu, H.-M., Zhao, J.-S., Gu, A.-P., He, S.-Y., Liu, J.-N. & Shi, Q.-Z. (2003). *Chin. J. Org. Chem.* **19**, 722–726.
Zhang, R.-L., Zhao, J.-S., Gu, A.-P. & He, S.-Y. (2003). *Acta Chim. Sin.* **61**, 262–266.
Zhao, J.-S., Zhang, R.-L., He, S.-Y., Xue, G.-L., Dou, J.-M. & Wang, D.-Q. (2003). *Chin. J. Struct. Chem.* **22**, 477–480.