APPLIED ORGANOMETALLIC CHEMISTRY
Appl. Organometal. Chem. 2003; **17**: 319–320
Main Group Metal Compounds
Published online in Wiley InterScience (www.interscience.wiley.com). DOI:10.1002/aoc.438

Crystallographic report

Crystal structure of tetra(4-methyl-5-imidazole-carboxyaldehyde)zinc(II) diperchlorate

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Received 6 January 2003; Revised 16 January 2003; Accepted 17 January 2003

The central zinc(II) atom in the title complex is tetrahedrally coordinated by four nitrogen atoms derived from 4-methyl-5-imidazolecarboxyaldehyde ligands with Zn–N in the range 2.007(3) to 2.026(4) Å.

KEYWORDS: 4-methyl-5-imidazolecarboxyaldehyde; X-ray crystallography; zinc; hydrogen bond

COMMENT

Imidazole and its derivatives frequently coordinate metal ions in the active sites of metalloenzymes and, are therefore, the study of metal complexes is of some current interest so as to establish a relationship between structure and/or function in metalloenzymes.[1,2] In this work, the preparation and structure of a new imidazole-containing metal complex, namely $[Zn(IMA)_4](ClO_4)_2$ (IMA = 4-methyl-5-imidazolecarboxyaldehyde), is described. The zinc(II) atom is situated on a crystallographic two-fold axis and exists within a distorted tetrahedral geometry defined by four nitrogen atoms from four IMA ligands; Fig. 1. The Zn–N bond lengths are in the range 2.007(3) to 2.026(4) Å, *i.e.* typical Zn–N(imidazole) bond lengths reported in four-coordinated imidazole-containing zinc(II) complexes.[1,2] The slight difference in the Zn–N bond lengths is mainly attributed to the different strength of hydrogen bonds involving the imidazole rings, as summarized in the figure caption.

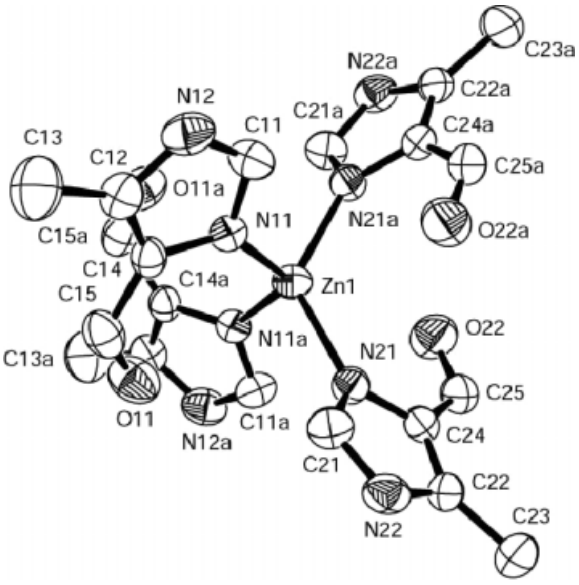

Figure 1. The dication of $[Zn(IMA)_4]^{2+}$ in the title complex; hydrogen atoms have been removed for clarity. Key geometric parameters: Zn1–N11 2.007(4), Zn1–N21 2.026(4) Å; N11–Zn1–N21 102.65(14), N11–Zn1–N11a 121.5(2), N11-Zn1-N21a 102.91(14), N21–Zn1–N21a 126.2(2)°; N12···O4b 2.935(6), N12···O1c 2.882(6), N22···O3d 2.845(5) Å. Symmetry operations: (a) $-x$, y, $-z+1/2$; (b) $-x+1/2$, $y-1/2$, $-z+1/2$; (c) x, $-y+2$, $z-1/2$; (d) $-x+1/2$, $-y+3/2$, $-z+1$.

EXPERIMENTAL

Synthesis

An aqueous solution of IMA (0.44 g, 4.0 mmol) was added to an ethanol solution of $Zn(NO_3)\cdot 6H_2O$ (0.29 g, 1.0 mmol) and $NaClO_4$ (0.28 g, 2.0 mmol), and the pH value of the solution was adjusted to 6–7 with an aqueous solution of NaOH. The resulting mixture was

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Contract/grant sponsor: NSFC; Contract/grant numbers: 20271044, 20273052.
Contract/grant sponsor: NSFC of Fujian Province of China; Contract/grant number: E0110001.

allowed to stand in air and colorless crystals of the title complex were obtained within a week (0.65 g, 93%).

Crystallography

Intensity data for the title compound were collected at 298 K on a Siemens *R*3 diffractometer for a colorless crystal $0.40 \times 0.42 \times 0.50$ mm^3. $C_{20}H_{24}Cl_2N_8O_{12}Zn$, $M = 704.74$, monoclinic, $C2/c$, $a = 18.396(4)$, $b = 8.0670(16)$, $c = 18.649(4)$ Å, $\beta = 90.008(3)^\circ$, $V = 2768(1)$ Å^3, $Z = 4$; 2163 unique data ($\theta = 26.0^\circ$), 1610 data with $I > 2\sigma(I)$. $R_1 = 0.049$, $wR_2 = 0.124$; $\rho_{max} = 0.34$ e$^-$ Å^{-3}. Programs used: SHELXL and ORTEP. The unit cell appears to be metrically orthorhombic and, indeed, the systematic absences are consistent with the space groups $Cmc2_1$ and $Cmcm$. However, merging the data both in *mm2* and *mmm* gave R_{int} values of 0.502, thereby precluding the higher symmetry space groups. CCDC deposition number: 200314.

Acknowledgements

This work was supported by NSFC (grant nos. 20271044 and 20273052) and NSFC of Fujian Province of China (grant no. E0110001).

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