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Crystallographic report

Dichloro[bis(1-methylimidazole-2)disulfide]zinc(II)

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The Zn center in [ZnCl$_2$(L-S-S-L)], where L-S-S-L = bis(1-methylimidazole-2)disulfide, adopts a tetrahedral configuration defined by two Cl atoms and two N atoms from L-S-S-L, which was obtained by *in situ* oxidation of 1-methylimidazole-2-thione. Copyright © 2004 John Wiley & Sons, Ltd.

KEYWORDS: crystal structure; zinc; disulfide bond; chelate

COMMENT

The [ZnCl$_2$(L-S-S-L)] complex (Fig. 1), where L-S-S-L = bis(1-methylimidazole-2)disulfide, was obtained fortuitously by very slow oxidation of authenticated [ZnCl$_2$(mitH-*S*)$_2$] (mitH-*S* = 1-methylimidazoline-2-thione).[1] The Zn(II) center has a tetrahedral configuration with ZnCl$_2$N$_2$ coordination type, in contrast to the ZnCl$_2$S$_2$ geometry found in [ZnCl$_2$(mitH-*S*)$_2$].[1] The bidentate ligand forms a seven-membered chelate ring. The disulfide bond is twisted from one imidazole ring to the other, and intersects the ZnCl$_2$ plane. The S–S distance of 2.069(1) Å is almost the same as that of the free disulfide cation, which is 2.085(2) Å.[2] The C–S bonds in [ZnCl$_2$(L-S-S-L)] (which are 1.737(3) and 1.754(3) Å, as expected) are almost single bonds and they are longer than those in [ZnCl$_2$(mitH-*S*)$_2$] (1.709(4) and 1.714(5) Å), which contain π-character.[1] Two imidazolyl rings in [ZnCl$_2$(mitH-*S*)$_2$] are splayed somewhat to avoid steric hindrance, whereas in [ZnCl$_2$(L-S-S-L)] the two imidazolyl rings are almost coplanar.

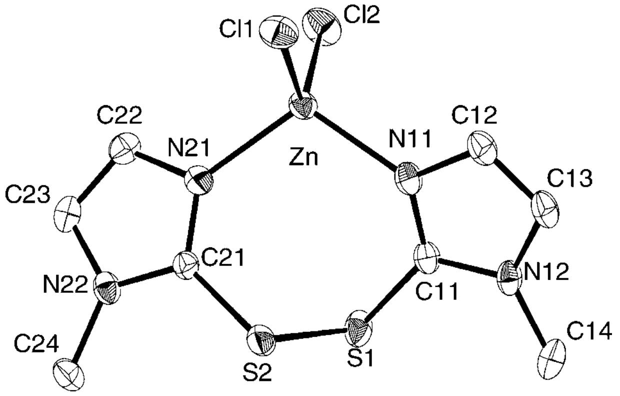

Figure 1. Molecular structure of [ZnCl$_2$(L-S-S-L)]; H atoms removed for clarity. Key geometric parameters: Zn–Cl1 2.259(1), Zn–Cl2 2.238(1), Zn–N11 2.023(3), Zn–N21 2.025(3), S1–S2 2.069(1), S1–C11 1.737(3), S2–C21 1.754(3) Å; Cl1–Zn–Cl2 117.39(4), C11–S1–S2 100.5(1), N11–Zn–N21 111.1(1), C21–S2–S1 101.4(1)°.

EXPERIMENTAL

The [ZnCl$_2$(L-S-S-L)] complex was obtained from the reaction between ZnCl$_2$ (0.69 g, 5.0 mmol) and mitH (1.16 g, 10 mmol) in CH$_3$OH solution (45 cm^3). After filtration of a white powder and crystals of [ZnCl$_2$(mitH-*S*)$_2$][1] (1.26 g, 69% yield), yellow crystals were formed by allowing the filtrate to stand at 0 °C for several months (0.06 g, 4% yield). Data were collected at −80 °C on a Rigaku/MSC Mercury CCD system for crystal dimensions 0.40 × 0.40 × 0.50 mm^3. C$_8$H$_{10}$Cl$_2$N$_4$S$_2$Zn, $M = 362.60$, orthorhombic, *Pbca*, $a = 8.5186(5)$, $b = 13.0905(5)$ and $c = 24.5506(10)$ Å, $V = 2737.7(2)$ Å^3, $Z = 8$, 3117 unique data ($2\theta_{\max}$ 55.0°), 3022 data with $I > 2\sigma(I)$, $R = 0.035$, $R_w = 0.075$. Programs used: SIR92, DIRDIF99, CrystalClear and CrystalStructure. CCDC deposition number: 250494.

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