metal-organic papers

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Key indicators

Single-crystal X-ray study
T = 293 K
Mean σ(C–C) = 0.007 Å
R factor = 0.057
wR factor = 0.155
Data-to-parameter ratio = 16.7

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

Bis(3-amino-5-methylisoxazole-κN)dichlorozinc(II)

The title compound, $[\text{Zn}^{\text{II}}\text{Cl}_2(\text{C}_4\text{H}_6\text{N}_2\text{O})_2]$, is built up from two isoxazole rings and two Cl atoms in a tetrahedral arrangement around the Zn atom. There are intramolecular N—H⋯Cl hydrogen bonds. Infinite supramolecular chains, formed by N—H⋯Cl intermolecular hydrogen bonds, are associated further with each other by π–π interactions between the anti-parallel isoxazole rings.

Comment

3-Amino-5-methylisoxazole is a key intermediate of sulfamethoxazole, a well known antibiotic. As part of our research interest in isoxazole derivatives, the title compound, (I), has been prepared and its structure determined.

(I)
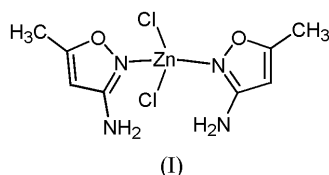
The molecule of (I), with the atom-numbering scheme, is shown in Fig. 1. Two isoxazole rings are arranged in a *cis* conformation with respect to the N1—Zn—N3 plane. Such an arrangement is certainly induced by the intramolecular N2—H2A⋯Cl2 and N4—H4E⋯Cl2 hydrogen-bond interactions. The Zn atom is coordinated by two N atoms and two Cl atoms. The two Zn—N distances are identical within experimental error, as are the Zn—Cl distances (Table 1). The bond angles around Zn range from 104.55 (11) to 120.56 (6)° and so the coordination around the Zn atom may be regarded as distorted tetrahedral. The bond lengths and angles within the isoxazole rings agree with those in previously reported structures (Meyer & Pritzkow, 1999; García-Raso *et al.*, 2000; Takasuka & Nakai, 2001).
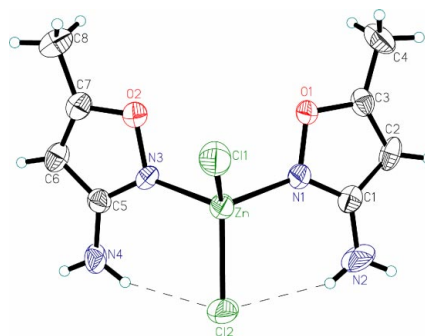

Figure 1
View of the molecular complex, (I), with the atom-numbering scheme. Displacement ellipsoids are drawn at the 40% probability level. Intramolecular N—H⋯Cl hydrogen bonds are illustrated by dashed lines.

π–π interaction may arise from the uneven charge distribution across the system due to electrostatic attraction (Muehldorf *et al.*, 1988). In (I), π–π interactions arise from the uneven charge distribution between the anti-parallel isoxazole rings C1—C2—C3 and their symmetry-related counterparts at $(-x, 2 - y, -z)$, with a centroid–centroid separation of 3.452 Å.

In the crystal structure, infinite supramolecular chains of (I) running along the [001] direction, as shown in Fig. 2, are formed by the N4—H4D···Cl1[ii] hydrogen bond. The chains are then associated further with each other by π–π interactions, forming layers running parallel to the (010) plane, and are connected to neighboring layers by N2—H2B···Cl1[i] hydrogen bonds (symmetry codes as in Table 2).

Experimental

3-Amino-5-methylisoxazole and an aqueous solution of $ZnCl_2$ (20%) in a molar ratio of 1:1 were mixed together and heated, with stirring, at a temperature of 353 K for 30 min. Crystals of (I) were obtained by slow evaporation of the excess of water for two weeks at room temperature; yield approximately 25%.

Crystal data

$[ZnCl_2(C_4H_6N_2O)_2]$	Mo $K\alpha$ radiation
$M_r = 332.49$	Cell parameters from 596
Orthorhombic, $Pccn$	reflections
$a = 13.105 (3)$ Å	$\theta = 2.6$–$22.4°$
$b = 14.054 (3)$ Å	$\mu = 2.26$ mm^{-1}
$c = 14.344 (3)$ Å	$T = 293 (2)$ K
$V = 2641.8 (10)$ Å^3	Prism, colorless
$Z = 8$	$0.25 \times 0.15 \times 0.15$ mm
$D_x = 1.672$ Mg m^{-3}	

Data collection

Bruker SMART CCD area-detector diffractometer	2602 independent reflections
	1744 reflections with $>2\sigma(I)$
φ and ω scans	$R_{int} = 0.029$
Absorption correction: multi-scan (*SADABS*; Bruker, 2000)	$\theta_{max} = 26.0°$
	$h = -13 \rightarrow 16$
$T_{min} = 0.67$, $T_{max} = 0.71$	$k = -16 \rightarrow 17$
13278 measured reflections	$l = -17 \rightarrow 17$

Refinement

Refinement on F^2	H-atom parameters constrained
$R[F^2 > 2\sigma(F^2)] = 0.057$	$w = 1/[\sigma^2(F_o^2) + (0.0752P)^2]$
$wR(F^2) = 0.155$	where $P = (F_o^2 + 2F_c^2)/3$
$S = 1.24$	$(\Delta/\sigma)_{max} < 0.001$
2602 reflections	$\Delta\rho_{max} = 0.49$ e Å^{-3}
156 parameters	$\Delta\rho_{min} = -0.53$ e Å^{-3}

Table 1
Selected bond lengths (Å).

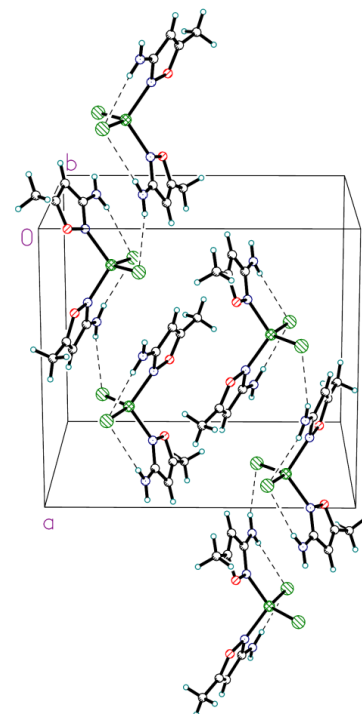|			
Zn—N3	2.002 (4)	C3—C4	1.485 (7)
Zn—N1	2.010 (4)	C5—N3	1.306 (6)
Zn—Cl2	2.2344 (13)	C5—N4	1.348 (6)
Zn—Cl1	2.2372 (14)	C5—C6	1.397 (7)
C1—N1	1.314 (6)	C6—C7	1.325 (6)
C1—N2	1.335 (6)	C7—O2	1.348 (5)
C1—C2	1.399 (7)	C7—C8	1.484 (6)
C2—C3	1.332 (7)	N1—O1	1.417 (4)
C3—O1	1.341 (5)	N3—O2	1.412 (4)

Figure 2
View of the molecular packing, showing the N—H···Cl interactions as dashed lines and the π–π stacking of parallel rings.

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

D—H···A	D—H	H···A	D···A	D—H···A
N2—H2A···Cl2	0.86	2.61	3.394 (5)	152
N4—H4E···Cl2	0.86	2.67	3.409 (4)	145
N2—H2B···Cl1[i]	0.86	2.90	3.341 (4)	114
N4—H4D···Cl1[ii]	0.86	2.72	3.462 (4)	146

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

All H atoms were positioned geometrically and allowed to ride on their parent atoms at distances of 0.86–0.96 Å, with $U_{iso} = 1.2$ or $1.5U_{eq}$ of the parent atom.

Data collection: *SMART* (Bruker, 2000); cell refinement: *SMART*; data reduction: *SAINT* (Bruker, 2000); program(s) used to solve structure: *SHELXTL* (Bruker, 2000); program(s) used to refine structure: *SHELXTL*; molecular graphics: *SHELXTL*; software used to prepare material for publication: *SHELXTL*.

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