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Dichlorobis(1,3-dimethylthiourea-κ*S*)zinc(**II**)

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Key indicators

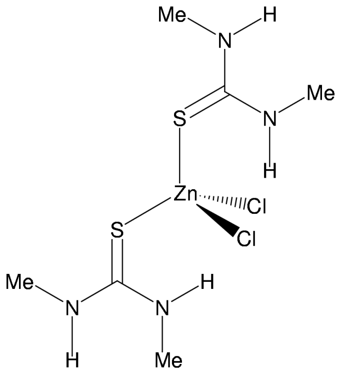
Single-crystal X-ray study
T = 150 K
Mean σ(N–C) = 0.003 Å
R factor = 0.028
wR factor = 0.067
Data-to-parameter ratio = 18.6

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

Determination of the crystal structure of the title compound, [$ZnCl_2(C_3H_6N_2S)_2$], reveals a distorted tetrahedral geometry around the zinc centre which occupies a twofold axis. Both intra- and intermolecular hydrogen bonding is observed between the 1,3-dimethylthiourea NH groups and the coordinated Cl atoms.

Comment

The title compound, (I), was formed as part of our investigations into the formation of bis-thiourea zinc(II) dicarboxylate polymers (Burrows *et al.*, 2000, 2004; Burke *et al.*, 2003).



(I)

The asymmetric unit of (I) (Fig. 1) consists of a zinc(II) centre occupying a twofold symmetry axis, to which is coordinated one 1,3-dimethylthiourea ligand, *via* the S atom, and one Cl^-. The complete molecule is generated by transformation through a twofold rotation axis, inherent in the space group. The geometry around the Zn centre is distorted tetrahedral, with bond angles ranging from 104.35 (3) to

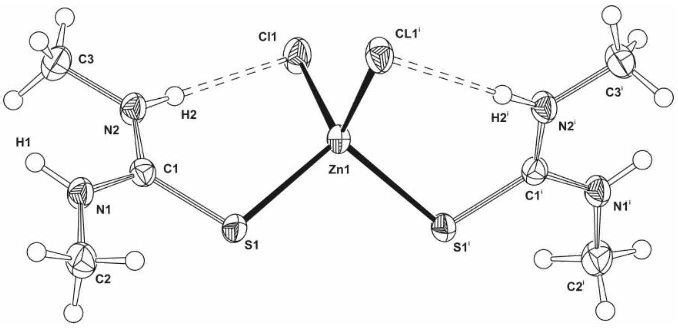


Figure 1
A view of the molecule of (I), showing the atom-labelling scheme. Displacement ellipsoids are drawn at the 30% probability level. H atoms represented by small spheres. [Symmetry code: (i) $1 - x, y, \frac{1}{2} - z$.]

113.300 (19)°. This study confirms previous conclusions on the structure of (I) which emerged on the basis of IR studies (Marcotrigiano, 1975).

The NH groups of the 1,3-dimethylthiourea ligands are arranged such that they facilitate the formation of both intra- and intermolecular hydrogen bonds, involving Cl^- anions as acceptors in both cases; details are given in Table 1. As seen in a number of zinc(II) bis(thiourea) dicarboxylate polymers (Burrows *et al.*, 2000), the intramolecular hydrogen bonds have graph-set notation $S(6)$. The intermolecular hydrogen bonds link the molecules into infinite hydrogen-bonded chains (Fig. 2). These interactions occur pairwise and lead to hydrogen-bonded rings with graph-set notation $R_2^2(12)$. There is no inter-chain hydrogen bonding present.

Experimental

Equimolar aqueous solutions of zinc(II) tetra(1,3-dimethylthiourea) dichloride (Ashcroft, 1970) and sodium salts of succinic, itaconic or mesaconic acids were allowed to evaporate slowly over a period of two weeks, in each case resulting in the formation of colourless crystals. Analysis by single-crystal X-ray diffraction revealed the identity of the products as (I) and confirmed that the dicarboxylate was not incorporated into the structure.

Crystal data

$[ZnCl_2(C_3H_6N_2S)_2]$
$M_r = 344.62$
Monoclinic, $C2/c$
$a = 13.0230$ (4) Å
$b = 8.9470$ (3) Å
$c = 12.4350$ (3) Å
$\beta = 106.967$ (2)°
$V = 1385.82$ (7) Å^3
$Z = 4$
$D_x = 1.652$ Mg m^{-3}
Mo $K\alpha$ radiation
Cell parameters from 1063 reflections
$\theta = 0.2$–26.3°
$\mu = 2.44$ mm^{-1}
$T = 150$ (2) K
Block, colourless
0.18 × 0.15 × 0.15 mm

Data collection

Nonius KappaCCD area-detector diffractometer
φ and ω scans
Absorption correction: multi-scan (Blessing, 1995)
$T_{\min} = 0.655$, $T_{\max} = 0.697$
8273 measured reflections
1580 independent reflections
1439 reflections with $I > 2\sigma(I)$
$R_{\rm int} = 0.034$
$\theta_{\max} = 27.5°$
$h = -16 \rightarrow 16$
$k = -11 \rightarrow 11$
$l = -16 \rightarrow 16$

Refinement

Refinement on F^2
$R[F^2 > 2\sigma(F^2)] = 0.028$
$wR(F^2) = 0.067$
$S = 1.14$
1580 reflections
85 parameters
H atoms treated by a mixture of independent and constrained refinement
$w = 1/[\sigma^2(F_o^2) + (0.0178P)^2 + 2.1477P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{\max} < 0.001$
$\Delta\rho_{\max} = 0.43$ e Å^{-3}
$\Delta\rho_{\min} = -0.44$ e Å^{-3}

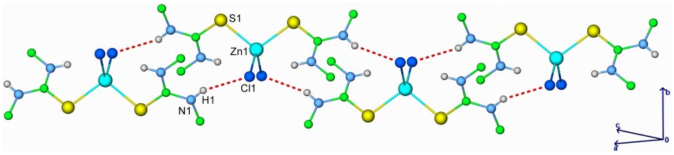


Figure 2
A view of the intermolecular hydrogen-bond interactions in (I), leading to chains along the crystallographic [101] direction.

Table 1
Hydrogen-bonding geometry (Å, °).

D—H···A	D—H	H···A	D···A	D—H···A
N2—H2···Cl1	0.884 (17)	2.337 (18)	3.2110 (19)	170 (3)
N1—H1···Cl1$^{\rm i}$	0.887 (17)	2.47 (2)	3.2737 (19)	152 (2)

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

H atoms were included at calculated positions on all carbon centres, being constrained to an ideal geometry with C—H distances of 0.98 Å and $U_{\rm iso}({\rm H}) = 1.5U_{\rm eq}({\rm C})$. Each group was allowed to rotate freely about its C—N bond. The position of the amino H atoms were located from the difference map and refined isotropically subject to a distance constraint of 0.89 Å.

Data collection: *COLLECT* (Hooft, 1998); cell refinement: *HKL DENZO* (Otwinowski & Minor, 1997); data reduction: *DENZO* and *SCALEPACK* (Otwinowski & Minor, 1997); program(s) used to solve structure: *SHELXS*97 (Sheldrick, 1997); program(s) used to refine structure: *SHELXL*97 (Sheldrick, 1997); molecular graphics: *SHELXTL* (Bruker, 2001); software used to prepare material for publication: *SHELXTL* and local programs.

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