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Tris(ethylenediamine-κ^2N,N')zinc(II) bis[2,3-dimercaptobutenedinitrile(2–)-κ^2S,S']zincate(II)

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Key indicators

Single-crystal X-ray study
T = 296 K
Mean σ(C–C) = 0.011 Å
R factor = 0.054
wR factor = 0.138
Data-to-parameter ratio = 16.6

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

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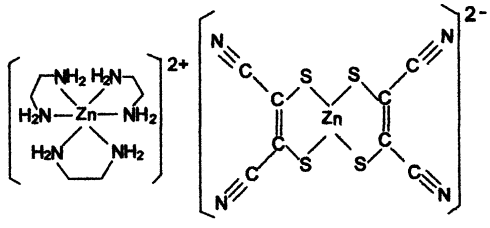
The title complex, $[Zn(C_2H_8N_2)_3][Zn(C_4N_2S_2)_2]$, exists as discrete ions, both of which lie on twofold rotation axes. The $[Zn(C_2H_8N_2)_3]^{2+}$ cation exhibits a slightly distorted octahedral geometry. In the $[Zn(C_4N_2S_2)_2]^{2-}$ anion, the Zn^{II} atom is surrounded by two chelating ligands in a distorted tetrahedral geometry. The crystal structure is stabilized by hydrogen bonds of the types N—H···N and N—H···S.

Comment

The title compound, (I), consists of discrete $[Zn(en)_3]^{2+}$ cations and $[Zn(mnt)_2]^{2-}$ anions (where en is ethylenediamine and mnt is 2,3-dimercaptobutenedinitrile). As shown in Fig. 1, Zn2 in the cation is coordinated by six N atoms from three en molecules. A crystallographic twofold rotation axis in the cation passes through Zn2 and the centre of the C7—C7^{i} bond [symmetry code: (i) $x-\frac{1}{2}, \frac{3}{2}-y, -z$]. The average Zn—N bond length of 2.181 (6) Å is in the normal range for zinc(II) complexes with amine N atoms. The *trans* angles of the ZnN_6 octahedron are 165.5 (4), 167.0 (2) and 167.0 (2)°. The other angles are in the range 79.5 (2)–99.8 (3)°, indicating a distorted octahedral geometry. Atom Zn1 in the anion is four-coordinated by two mnt ligands *via* four S atoms, with Zn1—S distances of 2.3229 (17) and 2.3311 (18) Å. The angles around atom Zn1 range from 93.65 (6) to 123.30 (6)°, indicating a distorted tetrahedral environment. The amine N atoms in en and the nitrile N and mercapto S atoms of mnt participate in intermolecular N—H···N and N—H···S hydrogen bonds, forming a three-dimensional hydrogen-bond network (Fig. 2 and Table 1).



(I)

Experimental

H_2mnt (1.00 mmol) and NaOH (2.00 mmol) were dissolved in 20 ml ethanol. To this solution, en (1.5 mmol) and an ethanol solution (30 ml) of $ZnSO_4$ (1.0 mmol) were added dropwise at 313 K. The mixture was stirred for 4 h and part of the solvent was evaporated in a rotary vacuum evaporator. The resulting solution was filtered and left in the air for about 8 d. Large colourless block-like crystals of (I) were obtained. Elemental analysis found: C 28.36, H 4.02, N 23.55, S 21.61%; calculated for $C_{14}H_{24}N_{10}S_4Zn_2$: C 28.43, H 4.09, N 23.68, S 21.69%.

Crystal data

$[Zn(C_2H_8N_2)_3][Zn(C_4N_2S_2)_2]$
M_r = 591.41
Orthorhombic, *Pbcn*
a = 12.0231 (12) Å
b = 14.269 (3) Å
c = 14.559 (3) Å
V = 2497.8 (8) Å^3
Z = 4
D_x = 1.573 Mg m^{-3}
Mo *Kα* radiation
Cell parameters from 1781 reflections
θ = 2.6–20.6°
μ = 2.28 mm^{-1}
T = 296 (2) K
Block, colourless
0.25 × 0.20 × 0.10 mm

Data collection

Bruker SMART CCD area-detector diffractometer
φ and ω scans
Absorption correction: multi-scan (*SADABS*; Sheldrick, 1996)
T_{min} = 0.600, T_{max} = 0.804
12094 measured reflections
2158 independent reflections
1150 reflections with $I > 2\sigma(I)$
R_{int} = 0.060
θ_{max} = 25.0°
h = −14 → 13
k = −16 → 16
l = −16 → 17

Refinement

Refinement on F^2
$R[F^2 > 2\sigma(F^2)]$ = 0.054
$wR(F^2)$ = 0.138
S = 1.00
2158 reflections
130 parameters
H-atom parameters constrained
$w = 1/[\sigma^2(F_o^2) + (0.0649P)^2]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} < 0.001$
$\Delta\rho_{max}$ = 0.75 e Å^{-3}
$\Delta\rho_{min}$ = −0.75 e Å^{-3}

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

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
N5—H5*B*···N1^{i}	0.90	2.40	3.265 (10)	162
N5—H5*A*···N1^{ii}	0.90	2.56	3.348 (9)	146
N4—H4*A*···N2^{iii}	0.90	2.46	3.056 (9)	124
N3—H3*B*···N1^{iv}	0.90	2.53	3.255 (9)	138
N3—H3*A*···S1^{ii}	0.90	2.64	3.505 (8)	162

Symmetry codes: (i) $x-\frac{1}{2},\frac{3}{2}-y,-z$; (ii) $\frac{1}{2}-x,\frac{1}{2}+y,z$; (iii) $x,1-y,z-\frac{1}{2}$; (iv) $\frac{1}{2}-x,\frac{3}{2}-y,\frac{1}{2}+z$.

All H atoms were placed in idealized positions and constrained to ride on their parent atoms, with C—H = 0.97 Å and U_{iso}(H) = 1.2U_{eq}(C), or with N—H = 0.90 Å and U_{iso}(H) = 1.2U_{eq}(N).

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

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References

Bruker (1997). *SMART* and *SAINT*. Bruker AXS Inc., Madison, Wisconsin, USA.
Sheldrick, G. M. (1996). *SADABS*. University of Göttingen, Germany.

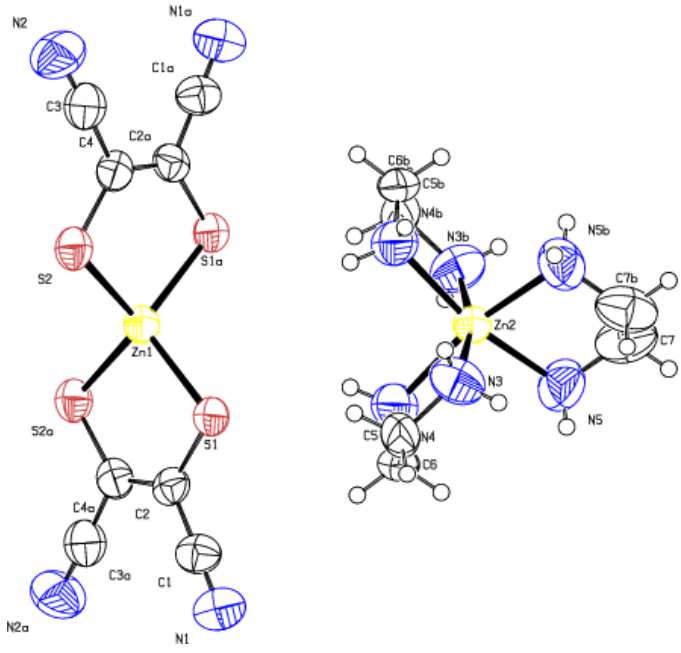

Figure 1
The ions of the title compound, showing 50% probability displacement ellipsoids and the atom-numbering scheme. [Symmetry code: $-x, y, \frac{1}{2}-z$, for both *a* and *b* in the anion and cation, respectively.]

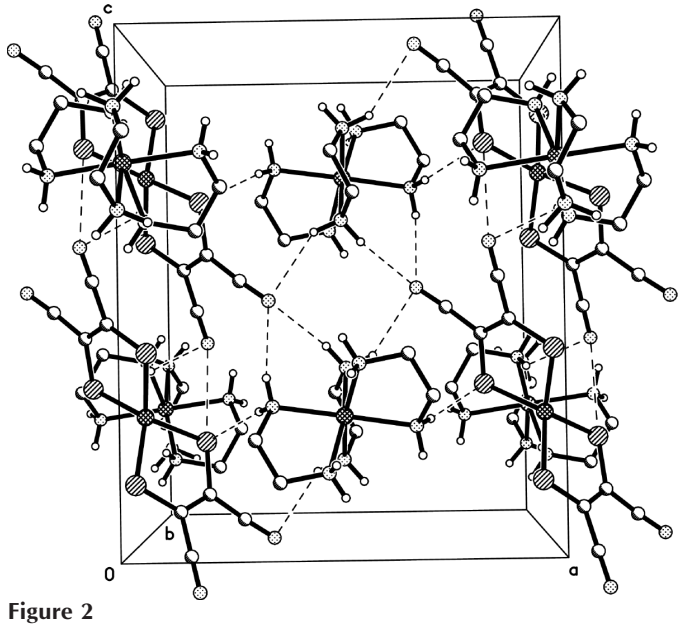

Figure 2
Crystal packing of (I), showing the N—H···N and N—H···S hydrogen-bonding interactions as dashed lines. H atoms bonded to C atoms have been omitted.

Sheldrick, G. M. (1997*a*). *SHELXS*97 and *SHELXL*97. University of Göttingen, Germany.
Sheldrick, G. M. (1997*b*). *SHELXTL*. Version 5.1. Bruker AXS Inc., Madison, Wisconsin, USA.