Acta Crystallographica Section E
Structure Reports Online
ISSN 1600-5368

Tris(ethylenediamine-κ^2N,N')zinc(II) bis(1,2-dicyanoethylenedithiolato-κ^2S,S')nickelate(II)

Ai-Yun Fu,[a,b]* Da-Qi Wang[a] and Tao Yu[a]

[a]Department of Chemistry, Dezhou University, Shandong Dezhou 253023, People's Republic of China, and [b]Department of Chemistry, Liaocheng University, Shandong Liaocheng 252059, People's Republic of China

Correspondence e-mail: aiyunfu@yahoo.com.cn

Key indicators

Single-crystal X-ray study
T = 295 K
Mean σ(C–C) = 0.009 Å
R factor = 0.049
wR factor = 0.070
Data-to-parameter ratio = 15.6

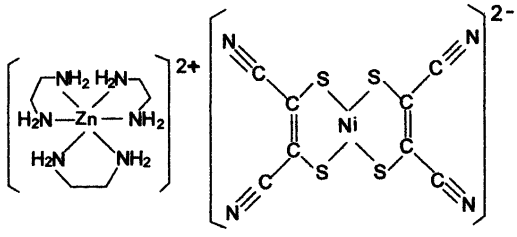
For details of how these key indicators were automatically derived from the article, see http://journals.iucr.org/e.

Received 8 October 2004
Accepted 15 November 2004
Online 27 November 2004

The title complex, $[Zn(C_2H_8N_2)_3][Ni(C_4N_2S_2)_2]$, contains a $[Zn(C_2H_8N_2)_3]^{2+}$ complex cation with a distorted octahedral coordination of the Zn atom and an $[Ni(C_4N_2S_2)_2]^{2-}$ anion with a slightly distorted square-planar geometry for the Ni^{II} atom. The cation occupies a special position on a twofold axis, whereas the anion lies about a crystallographic inversion centre.

Comment

The crystal structure of the title compound, (I), is built of $[Zn(en)_3]^{2+}$ complex cations and $[Ni(mnt)_2]^{2-}$ complex anions (en is ethylenediamine, $H_2NCH_2CH_2NH_2$, and mnt = deprotonated 1,2-dicyanoethylenedithiolate, $[S_2C_2(CN)_2]^{2-}$). The cation occupies a special position on a twofold axis, whereas the anion is located on a crystallographic inversion centre. The structures of cation and anion are shown in Fig. 1.



(I)

The central Zn^{II} atom in the $[Zn(en)_3]^{2+}$ cation has a distorted octahedral geometry, formed by six N atoms of the three bidentate en ligands. The two symmetry-independent *trans* angles for the Zn1 octahedron are 169.4 (2) and 172.59 (18)°, chelate bite N—Zn1—N angles within the en ligands are 79.6 (3) and 80.66 (17)°, and the remaining angles in the Zn1 octahedron span the range 92.0 (2)–95.4 (2)°. The average Zn—N bond length of 2.156 Å is comparable to the value of 2.181 Å observed earlier in another $[Zn(en)_3]^{2+}$ cationic complex (Fu *et al.*, 2004).

Atom Ni1 in the $[Ni(mnt)_2]^{2-}$ anion has a slightly distorted square planar environment, with an endocylic S1—Ni1—S2 chelate bite angle of 87.70 (6)° and an exocyclic angle S1—Ni1—$S2^{iv}$ of 92.30 (5)° [symmetry code: (iv) $\frac{3}{2}-x$, $\frac{3}{2}-y$, $1-z$]; the Ni1—S1 and Ni1—S2 bonds are almost identical [2.1693 (14) and 2.1722 (15) Å, respectively].

There are no particularly short interionic contacts in the structure, the shortest Ni···N distance being 3.437 (5) Å [Ni1···$N4^{v}$; symmetry code: (v) $1+x$, $1-y$, $z-\frac{1}{2}$]. The interionic N—H···N and N—H···S contacts may be regarded as rather weak hydrogen bonds (Table 1 and Fig. 2)

Experimental

H_2mnt (1.00 mmol) and NaOH (2.00 mmol) were dissolved in ethanol (20 ml). 1.5 mmol of en and an ethanol solution (30 ml) of $ZnSO_4$ (0.5 mmol) and $NiSO_4$ (0.5 mmol) were added dropwise to this solution at 313 K. The mixture was stirred for 6 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 13 d. Large red block-shaped crystals of (I) were obtained. Elemental analysis found: C 28.58, H 4.06, N 23.79, S 21.81%; calculated for $C_{14}H_{24}N_{10}NiS_4Zn$: C 28.76, H 4.14, N 23.95, S 21.93%.

Crystal data

$[Zn(C_2H_8N_2)_3][Ni(C_4N_2S_2)_2]$	D_x = 1.621 Mg m^{-3}
M_r = 584.75	Cell parameters from 843 reflections
Monoclinic, $C2/c$	θ = 2.7–18.0°
a = 8.7430 (10) Å	μ = 2.16 mm^{-1}
b = 17.0087 (19) Å	T = 295 (2) K
c = 16.3138 (17) Å	Block, red
β = 98.982 (3)°	0.30 × 0.23 × 0.12 mm
V = 2396.2 (5) Å^3	
Z = 4	

Data collection

Bruker SMART CCD area-detector diffractometer	2120 independent reflections
φ and ω scans	1039 reflections with $I > 2\sigma(I)$
Absorption correction: multi-scan (*SADABS*; Bruker, 1997)	R_{int} = 0.071
T_{min} = 0.558, T_{max} = 0.774	θ_{max} = 25.0°
6159 measured reflections	$h = -9 \rightarrow 10$
	$k = -13 \rightarrow 20$
	$l = -19 \rightarrow 17$

Refinement

Refinement on F^2	H-atom parameters constrained
$R[F^2 > 2\sigma(F^2)]$ = 0.049	$w = 1/[\sigma^2(F_o^2) + (0.0064P)^2]$ where $P = (F_o^2 + 2F_c^2)/3$
$wR(F^2)$ = 0.070	$(\Delta/\sigma)_{max} < 0.001$
S = 1.00	$\Delta\rho_{max}$ = 0.49 e Å^{-3}
2120 reflections	$\Delta\rho_{min}$ = −0.48 e Å^{-3}
136 parameters	

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

D—H···A	D—H	H···A	D···A	D—H···A
N3—H3B···N2^{i}	0.90	2.70	3.590 (7)	171
N4—H4B···N1^{ii}	0.90	2.31	3.208 (6)	175
N5—H5A···S2^{iii}	0.90	2.81	3.696 (5)	169
N5—H5B···N1^{ii}	0.90	2.57	3.331 (6)	142

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

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), and 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, 1990); 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*.

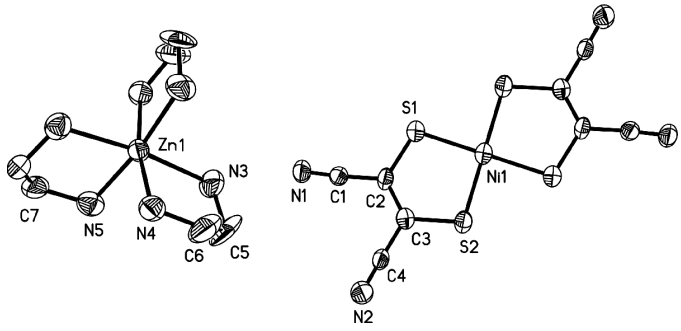


Figure 1
The cation and anion in the structure of (I), showing 50% probability displacement ellipsoids and the atom-numbering scheme. H atoms have been omitted. Unlabelled atoms in the cation are related by the symmetry code $-x, y, \frac{3}{2}-z$. Unlabelled atoms in the anion are related by the symmetry code $\frac{3}{2}-x, \frac{3}{2}-y, 1-z$.

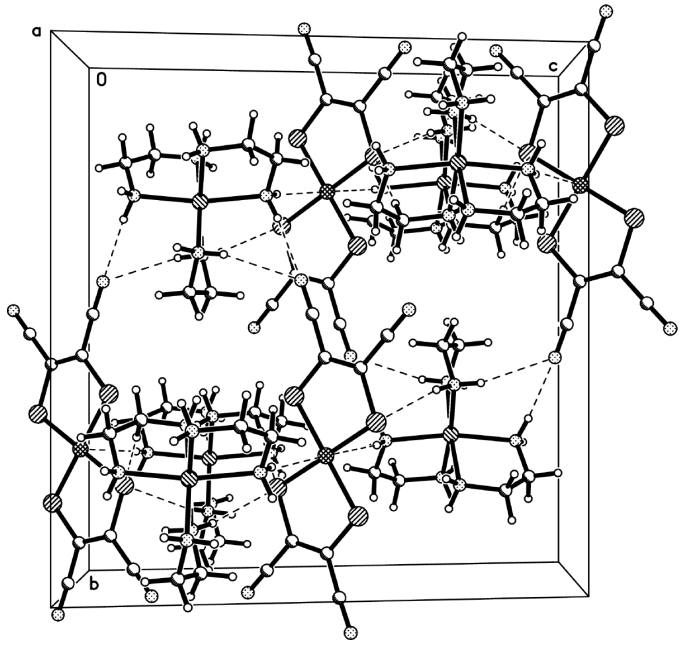


Figure 2
The crystal packing of (I), showing the Ni···N, N—H···S and N—H···N interactions as dashed lines; the view is along the *a* axis.

The authors thank the Science and Technology Office of Dezhou City, Shandong Province, People's Republic of China, for research grant No. 030701.

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